BRITISH STANDARD

BS 5666-5: 1986

Methods of analysis of

Wood preservatives and treated timber —

Part 5: Determination of zinc naphthenate in preservative solutions and treated timber

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

Confirmed November 2008

UDC 674.048:620.1

Committees responsible for this British Standard

The preparation of this British Standard was entrusted by the Wood Preservation Standards 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 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 6, an inside back cover and a back cover.

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

This British Standard, having been prepared under the direction of the Wood Preservation Standards Committee, was published under the authority of the Board of BSI and comes into effect on
28 February 1986

The following BSI references relate to the work on this standard:
Committee reference WPC/11
Draft for comment 84/55183 DC

ISBN 0 580 15022 4

Amendments issued since publication

Amd. No.	Date of issue	Comments

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1 Scope and field of application

This Part of BS 5666 describes procedures for the determination of zinc naphthenate, expressed as zinc, in preservative solutions and in treated timber. Two methods are described: an atomic absorption spectrophotometric method and a colorimetric method.

Both methods have been found suitable for the determination of zinc naphthenate in the presence of tributyltin oxide, pentachlorophenol, o-phenylphenol, monochloronaphthalene, polychloronaphthalene, gamma-HCH (gamma-BHC)[1)], dieldrin[1)], copper naphthenate, water-repellent waxes and resins.

The procedures described have been primarily designed for the analysis of preservative formulations complying with BS 5707-1. For samples of other compositions it may be necessary to make adjustments in the quantities taken for analysis.

The timber sample for analysis may be in the form of sawdust, wood flour or thin sections.

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 BS 5666-1:1978.

2 Method I. Atomic absorption spectrophotometric method

2.1 Principle

The zinc in the zinc naphthenate present is extracted quantitatively from preservative solutions or treated timber with hydrochloric acid solution. The resulting zinc solution is analysed using an atomic absorption spectrophotometer. The determination is carried out in the presence of excess potassium ions because the presence of sodium or potassium ions enhances the zinc absorbance signal.

2.2 Reagents

Except where otherwise specified, all reagents shall be of recognized analytical grade and water complying with BS 3978 shall be used throughout.

2.2.1 *White spirit*, complying with BS 245.

2.2.2 *Hydrochloric acid solution*, c(HCl) = 5 mol/L approximately. Dilute 500 mL of concentrated hydrochloric acid (ρ_{20} = 1.18 g/mL) to 1 L with water.

2.2.3 *Hydrochloric acid solution*, c(HCl) = 1 mol/L approximately. Dilute 100 mL of concentrated hydrochloric acid (ρ_{20} = 1.18 g/mL) to 1 L with water.

2.2.4 *Hydrochloric acid/potassium chloride solution*. Dissolve 4.8 g of potassium chloride in 100 mL of water. Add this to 100 mL of concentrated hydrochloric acid (ρ_{20} = 1.18 g/mL) and dilute to 1 L with water.

2.2.5 *Standard zinc solution*. 1 mL ≡ 100 μg of zinc. Dissolve 0.100 g of zinc metal in 100 mL of concentrated hydrochloric acid solution (ρ_{20} = 1.18 g/mL). Transfer the solution to a 1 L one-mark standard volumetric flask (see **2.3.1**) and dilute to the mark with water.

2.3 Apparatus

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

2.3.2 *Atomic absorption spectrophotometer*, together with a suitable source of resonance radiation for zinc, e.g. a hollow cathode lamp.

2.4 Procedure

2.4.1 *Instrument settings and operation*. The instrument settings and operating conditions for the determination of zinc shall be as recommended by the instrument manufacturer. Zinc is determined in an air/acetylene flame at a wavelength of 213.9 nm.

2.4.2 *Preparation of calibration solutions*, Into a series of 100 mL one-mark volumetric flasks, transfer 0, 0.50, 1.00, 2.00, 3.00, 4.00, 5.00, and 10.00 mL of the standard zinc solution (**2.2.5**). Add 20 mL of the hydrochloric acid/potassium chloride solution (**2.2.4**) and dilute to the mark with the hydrochloric acid solution (**2.2.3**).

These calibration solutions contain 0, 0.5, 1.0, 2.0, 3.0, 4.0, 5.0, and 10.0 μg/mL respectively of zinc.

NOTE A usable calibration graph should be obtained over the range of 0 μg/mL to 5 μg/mL zinc. Above this value, the curve becomes progressively non-linear. It is recommended that this part of the graph should not be used for the determination, but it is useful for obtaining approximate values.

2.4.3 *Analysis of preservative solutions*

2.4.3.1 *Preparation of test solution*, Accurately weigh approximately 1 g (see note) of the preservative solution into a tared 50 mL one-mark volumetric flask. Dilute to the mark with the white spirit (**2.2.1**) and mix thoroughly.

NOTE This mass may be adjusted to obtain a zinc content within the range of the procedure if the preservative has not been formulated to comply with BS 5707-1.

[1)] See BS 1831.

Pipette 1 mL of this solution into a 50 mL separating funnel, add approximately 10 mL of white spirit and swirl to mix. Extract successively with three 20 mL portions of the hydrochloric acid solution (**2.2.3**). For each extraction, shake vigorously for at least 90 s, allow to settle, and run off the aqueous layer into a 100 mL one-mark volumetric flask. Add 20 mL of the hydrochloric acid/potassium chloride solution (**2.2.4**) to the flask, dilute to the mark with the hydrochloric acid solution (**2.2.3**) and mix to provide the test solution.

2.4.3.2 *Measurement*. Using the operating conditions suitable for the instrument used, aspirate successively an appropriate range of calibration solutions (see **2.4.2**), the test solution or solutions, and again the calibration solutions to check the stability of the instrument readings, and measure the absorbance of each solution.

NOTE If a number of samples are to be analysed, it may be advisable to check the instrument stability by bracketing each test solution between appropriate calibration solutions.

Plot a calibration curve of the concentration of zinc against absorbance for the range of calibration solutions. If the absorbance of the test solution is above the range for the calibration solutions, adjust the concentration of the test solution by diluting it with a mixture of 20 mL of the hydrochloric acid solution (**2.2.4**) and 80 mL of the hydrochloric acid solution (**2.2.3**) so that the absorbance falls within this range, and note the dilution factor. Then repeat the aspiration procedure with both calibration solutions and test solution to produce revised values. Determine the zinc content of the test solution by reference to the calibration graph and express the concentrations as micrograms of zinc per millilitre of the test solution.

2.4.4 *Analysis of treated timber*

2.4.4.1 *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 BS 5666-1.

Divide the prepared sample into two portions, each of about 0.5 g. On one portion determine the moisture content according to the procedure described in **7.2** of BS 5666-1:1978.

NOTE For the purposes of this clause, in line 2 of **7.2** of BS 5666-1:1978 the word "extraction" should be read as "preparation".

2.4.4.2 *Extraction and preparation of test solution*, Accurately weigh about 0.5 g (see note) of the prepared timber sample (**2.4.4.1**) into a tared 50 mL conical flask.

NOTE For treated timber it is difficult to recommend quantities to be taken for analysis because the predominant factor is the amount of preservative within the timber rather than the mass of the wood itself. It may therefore be necessary to make some adjustments to the values quoted in this standard either in respect of the mass of treated timber sample taken for analysis or the level of dilution of the zinc solution resulting from the extraction.

Add 10 mL of the hydrochloric acid solution (**2.2.2**) and stand the flask in a water bath, controlled at 55 °C to 60 °C, for 30 min with occasional swirling. Remove the flask from the water bath, cool to room temperature and filter the contents of the flask through a filter paper[2)] into a 50 mL one-mark volumetric flask. Wash the residue and the filter paper thoroughly with water and add the washings to the volumetric flask. Dilute to the mark with water and mix.

Pipette 25 mL of this solution into a 100 mL one-mark volumetric flask, add 20 mL of the hydrochloric acid/potassium chloride solution (**2.2.4**) and dilute to the mark with the hydrochloric acid solution (**2.2.3**). Mix thoroughly to give the test solution.

2.4.4.3 *Measurement*. Determine the zinc content of the test solution as described in **2.4.3.2**.

2.4.5 *Calculations*

2.4.5.1 *Preservative solutions*, The percentage by mass of zinc in the preservative is given by the formula:

$$\frac{C \times D}{2m_1}$$

where

C is the concentration of zinc in the test solution (**2.4.3.1**) as determined from the calibration graph (in μg/mL);

m_1 is the mass of the preservative solution taken (in g);

D is the dilution factor arising from the adjustment of the concentration of the test solution (no adjustment, $D = 1$) (see **2.4.3.2**).

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

2.4.5.2 *Treated timber*. The percentage by mass of zinc in the dry test sample is given by the formula:

$$\frac{C \times D}{50 m_2}$$

where

C is the concentration of zinc in the test solution (**2.4.4.2**) as determined from the calibration graph (in μg/mL);

m_2 is the oven dry equivalent mass of the test portion of the prepared timber sample, i.e. the mass of the test portion corrected for the moisture content (see **2.4.4.1**) (in g);

D is the dilution factor arising from the adjustment of the concentration of the test solution (no adjustment, $D = 1$) (see **2.4.3.2**).

3 Method II. Colorimetric method

3.1 Principle

The zinc in the zinc naphthenate present is extracted quantitatively from preservative solutions or treated timber with a hydrochloric acid solution. The zinc content of the resulting solution is separated from other metals by a partitioning sequence between toluene and buffered aqueous systems. The zinc is then determined colorimetrically as the dithizone complex.

3.2 Reagents

All reagents shall be of recognized analytical grade and water complying with BS 3978 shall be used throughout.

3.2.1 *Toluene*, complying with grade 2 of BS 805.

3.2.2 *White spirit*, complying with BS 245.

3.2.3 *Ammonia solution*, concentrated (ρ_{20} = 0.880 g/mL).

3.2.4 *Sodium sulphate*, anhydrous and granular.

3.2.5 *Acetic acid/sodium acetate solution*. Dissolve 27.2 g of sodium acetate trihydrate in 100 mL of water, add 12 mL of glacial acetic acid and dilute to 200 mL with water.

3.2.6 *Acetate buffer solution*. Prepare this solution daily as required. Transfer 50 mL of the acetic acid/sodium acetate solution (**3.2.5**) and 10 mL of the sodium thiosulphate solution (**3.2.16**) to a 100 mL separating funnel and extract this mixture successively with 5 mL portions of the dithizone solution (**3.2.10**) until the green colour persists. Extract once more with 5 mL of the dithizone solution (**3.2.10**) and then with 5 mL of the toluene (**3.2.1**). Discard the toluene extract and washings and store the extracted acetate buffer solution in a well-stoppered bottle.

3.2.7 *Citric acid solution*. Dissolve 100 g of citric acid in 100 mL of water and dilute to 200 mL with water.

3.2.8 *Citrate buffer solution*. Prepare this solution daily as required. Transfer 20 mL of the citric acid solution (**3.2.7**) to a 100 mL separating funnel. Using litmus paper as an indicator, neutralize this solution with the ammonia solution (**3.2.3**), add a further 1.5 mL of the ammonia solution and dilute to a total volume of 30 mL with water. Extract successively with 5 mL portions of the dithizone solution (**3.2.10**) until the green colour persists.

NOTE The aqueous phase should be pale yellow. If it is not, add further ammonia solution drop by drop, shaking after each addition, until the aqueous phase becomes pale yellow.

Extract once more with 5 mL of the dithizone solution (**3.2.10**) and then wash the aqueous phase with successive 5 mL portions of the toluene (**3.2.1**) until the aqueous phase is colourless. Discard the toluene extract and washings and store the extracted citrate buffer solution in a well-stoppered bottle.

3.2.9 *Dithizone stock solution*. Dissolve 0.1 g of diphenylthiocarbazone (dithizone) in 100 mL of the toluene (**3.2.1**) and dilute to 250 mL with the toluene (**3.2.1**). Store this solution in a refrigerator.

3.2.10 *Dilute dithizone solution*. Prepare this solution daily as required. Extract 100 mL of the dithizone stock solution (**3.2.9**) with two successive 200 mL portions of water each containing 4 mL of the ammonia solution (**3.2.3**) using a 500 mL separating funnel[3]. Discard the toluene layer.

Transfer the combined aqueous extracts to a 1 L separating funnel, acidify with the hydrochloric acid solution (**3.2.13**) and extract the precipitated dithizone with 400 mL of the toluene (**3.2.1**). Wash the toluene extract with two successive 200 mL portions of water and filter the separated toluene solution through a dry filter "ashless" paper[4]. Keep the solution cool and away from direct light.

3.2.11 *Hydrochloric acid solution*, c(HCl) = 5 mol/L approximately. Dilute 500 mL of concentrated hydrochloric acid solution (ρ_{20} = 1.18 g/mL) to 1 L with water.

3.2.12 *Hydrochloric acid solution*, c(HCl) = 1 mol/L approximately. Dilute 100 mL of concentrated hydrochloric acid solution (ρ = 1.18 g/mL) to 1 L with water.

3.2.13 *Hydrochloric acid solution*, c(HCl) = 0.1 mol/L approximately. Dilute 50 mL of the hydrochloric acid solution (**3.2.12**) to 500 mL with water.

[3] It may be necessary to filter the combined aqueous extracts. A Whatman No. 42 paper has been found suitable.
[4] A Whatman No. 43 filter paper has been found to be suitable.

3.2.14 *Hydrochloric acid solution*, c(HCl) = 0.02 mol/L approximately. Dilute 20 mL of the hydrochloric acid solution (**3.2.12**) to 1 L with water.

3.2.15 *Sodium sulphide solution*. Dissolve 0.1 g of sodium sulphide in 100 mL of water and dilute to 250 mL with water.

3.2.16 *Sodium thiosulphate solution*. Dissolve 50 g of sodium thiosulphate pentahydrate ($Na_2S_2O_3.5H_2O$) in 100 mL of water and dilute to 200 mL with water.

3.2.17 *Standard zinc solution*, 1 mL ≡ 1 μg of zinc. Dissolve 0.1000 g of zinc metal in 50 mL of concentrated hydrochloric acid solution (ρ_{20} = 1.18 g/mL). Transfer to a 500 mL one-mark volumetric flask and dilute to the mark with water. Pipette 5 mL of this solution into a 1 L one-mark volumetric flask and dilute to the mark with the hydrochloric acid solution (**3.2.12**).

3.3 Apparatus

3.3.1 *Volumetric glassware*, of class A quality in accordance with BS 700-2 (type II), BS 846, BS 1583, or BS 1792 as appropriate.

3.3.2 *Spectrophotometer or photoelectric absorptiometer*, suitable for the measurement of absorbance at a wavelength of 532 nm and fitted with appropriate cells.

3.4 Procedure

3.4.1 *Analysis of preservative solutions*

3.4.1.1 *Extraction*. Accurately weigh approximately 1 g (see note to **2.4.3.1**) of the preservative solution into a tared 100 mL one-mark volumetric flask, dilute to the mark with the white spirit (**3.2.2**) and mix thoroughly. Pipette 1 mL of this solution into a 50 mL separating funnel, add approximately 10 mL of white spirit, and swirl to mix.

Extract successively with three 20 mL portions of the hydrochloric acid solution (**3.2.12**). For each extraction, shake vigorously for at least 90 s, allow the layers to separate, and run off the aqueous layer into a 100 mL volumetric flask. Dilute to the mark with the hydrochloric acid solution (**3.2.12**) and mix.

3.4.1.2 *Separation*. Pipette 5 mL of the solution from **3.4.1.1** into a 50 mL separating funnel (designated funnel A), add 5 mL of the hydrochloric acid solution (**3.2.12**) and mix. Add the ammonia solution (**3.2.3**) drop by drop until the mixture is alkaline to litmus paper and then add 3 mL of the citrate buffer solution (**3.2.8**).

Add 5 mL of the dithizone solution (**3.2.10**), shake vigorously, and allow the layers to separate. By means of a teat pipette, remove the upper organic layer and transfer it to a second 50 mL separating funnel (designated funnel B). Continue the extraction of the aqueous layer with successive 5 mL portions of the dithizone solution (**3.2.10**) until the organic layer remains green, each time transferring the organic layer to funnel B. Extract once more with the dithizone solution (**3.2.10**) and add the organic layer to funnel B. Discard the aqueous layer and rinse funnel A several times with water.

To the combined extracts in funnel B add 5 mL of the hydrochloric acid solution (**3.2.14**), shake vigorously and allow the layers to separate. Run off the aqueous layer into the clean funnel A. Continue the extraction of the organic solvent layer with successive 2 mL portions of the hydrochloric acid solution (**3.2.14**) until the organic layer remains green, then extract once more adding each 2 mL extract to funnel A. Wash the combined aqueous extracts by shaking with 10 mL of the toluene (**3.2.1**). Discard both the organic solvent in funnel B and the toluene washings. Rinse funnel B several times with the toluene (**3.2.1**). Add 6 mL of the acetate buffer solution (**3.2.6**) to funnel A and mix.

NOTE The pH should be between 4.0 and 4.5. Use narrow range indicator paper to determine the pH and make adjustments if necessary.

Add 5 mL of the dithizone solution (**3.2.10**) to the mixture in funnel A, shake vigorously for at least 1 min, allow the layers to separate, and then transfer the organic layer in funnel A to funnel B by means of a teat pipette. Repeat this extraction and transfer process with successive 2 mL portions of the dithizone solution (**3.2.10**) until the upper layer remains green, then extract once more. Wash the aqueous residue in funnel A with 5 mL of the toluene (**3.2.1**) and add the washing to the combined organic extracts in funnel B. Discard the contents of funnel A.

Wash the combined organic extracts in funnel B with two successive 15 mL portions of water and discard the washings. Add 10 mL of the sodium sulphide solution (**3.2.15**), shake vigorously for at least 20 s and allow the layers to separate. Discard the aqueous layer. Repeat this extraction with successive 10 mL portions of the sodium sulphide solution (**3.2.15**) until the lower layer no longer turns yellow.

3.4.1.3 *Test solution*. Add 1 g of the sodium sulphate (**3.2.4**) to the contents of funnel B, shake for several seconds and filter[5)] the liquid into a 25 mL one-mark volumetric flask. Wash the funnel, sodium sulphate and filter paper with small quantities of the toluene (**3.2.1**), add the washings to the volumetric flask and then dilute to the mark with toluene to give the test solution.

3.4.1.4 *Reagent blank*. Add 10 mL of the hydrochloric acid solution (**3.2.12**) to a 50 mL separating funnel and carry out the procedures described in **3.4.1.2** and **3.4.1.3**, starting from the addition of ammonia solution.

3.4.1.5 *Measurement*. Measure the absorbance of the test solution from **3.4.1.3** at a wavelength of 532 nm against the reagent blank (see **3.4.1.4**). If the absorbance of the test solution is above the range for the calibration solutions, adjust the concentration of the test solution by diluting it with toluene so that the absorbance falls within the range of absorbances used for the calibration curve (see **3.4.3**) and note the dilution factor. Determine the zinc content by reference to the calibration curve.

3.4.2 *Analysis of treated timber*

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 BS 5666-1.

Divide the prepared sample into two portions each of about 0.5 g. On one portion determine the moisture content according to the procedure described in **7.2** of BS 5666-1:1978.

NOTE Fr the purposes of this clause, in line 2 of **7.2** of BS 5666-1:1978, the word "extraction" should be read as "preparation".

3.4.2.2 *Extraction*. Accurately weigh approximately 0.5 g (see note to **2.4.4.2**) of the prepared timber sample (see **3.4.2.1**) into a tared 50 mL conical flask. Add 10 mL of the hydrochloric acid solution (**3.2.11**) and stand the flask in a water-bath, controlled at 55 °C to 60 °C, for 30 min with occasional swirling. Remove the flask from the water bath, cool to room temperature, and filter the contents of the flask through a filter paper[5)] into a 100 mL one-mark volumetric flask. Wash the residue and the filter paper thoroughly with water and add the washings to the filtrate in the volumetric flask. Add 50 mL of the hydrochloric acid solution (**3.2.12**), dilute to the mark with water and mix. Pipette 25 mL of this solution into a 100 mL one-mark volumetric flask, dilute to the mark with the hydrochloric acid solution (**3.2.12**) and mix.

3.4.2.3 *Separation and preparation of test solution*. Pipette 5 mL of the extract solution from **3.4.2.2** into a 50 mL separating funnel (funnel A), add 5 mL of the hydrochloric acid solution (**3.2.12**) and mix. Carry out the procedures described in **3.4.1.2** and **3.4.1.3**, starting from the addition of ammonia solution.

3.4.2.4 *Measurement*. Measure the absorbance of the test solution from **3.4.2.3** at a wavelength of 532 nm against the reagent blank (see **3.4.1.4**). If the absorbance of the test solution is above the range for the calibration solutions, adjust the concentration of the test solution by diluting it with toluene so that the absorbance falls within the range of absorbances used for the calibration curve (see **3.4.3**) and note the dilution factor. Determine the zinc content by reference to the calibration curve.

3.4.3 *Preparation of calibration curve*. Into a series of 50 mL separating funnels, pipette suitable volumes of the standard zinc solution (**3.2.17**) to cover the required range, which should be within the range 0 μg Zn to 10 μg Zn. To each funnel (which will now be designated funnel A) add enough of the hydrochloric acid solution (**3.2.12**) to bring the total volume up to 10 mL and mix. Carry out the procedures described in **3.4.1.2** and **3.4.1.3** starting from the addition of ammonia solution.

Measure the absorbance of each solution at 532 nm against the reagent blank (**3.4.1.4**). Plot the absorbance values against the corresponding zinc content in micrograms.

3.4.4 *Calculations*

3.4.4.1 *Preservative solutions*. The percentage by mass of zinc in the preservative is given by the formula:

$$\frac{N \times D}{5 m_1}$$

where

N is the zinc content of the test solution as determined from the calibration curve (in μg);

m_1 is the mass of the preservative solution taken (in g);

D is the dilution factor arising from adjustment of the concentration of the test solution (no adjustment $D = 1$) (see **3.4.1.5**).

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

3.4.4.2 *Treated timber*. The percentage by mass of zinc in the dry test sample is given by the formula:

$$\frac{N \times D}{125 m_2}$$

where

N is the zinc content of the test solution as determined from the calibration curve (in µg);

m_2 is the oven dry equivalent mass of the test portion of the prepared timber sample, i.e. the mass of the test portion corrected for the moisture content (see **3.4.2.1**) (in g);

D is the dilution factor arising from the adjustment of the concentration of the test solution (no adjustment, $D = 1$) (see **3.4.2.4**).

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 method used, i.e. method I or II of BS 5666-5;

c) for treated timber, the mass of prepared timber sample taken and its moisture content;

d) any deviation from the method described;

e) the results of the analysis;

f) any unusual features noted during the analysis.

Publications referred to

BS 135, BS 458, BS 805, *Specifications for benzene, xylenes and toluenes.*

BS 245, *Specification for mineral solvents (white spirit and related hydrocarbon solvents) for paints and other purposes.*

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 1831, *Recommended common names for pesticides.*

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.*

BS 5707, *Solutions of wood preservatives in organic solvents.*

BS 5707-1, *Specification for solutions for general purpose applications, including timber that is to be painted.*

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