BRITISH STANDARD

BS 5666-7:
1991

Methods of analysis of

Wood preservatives and treated timber —

Part 7: Quantitative analysis of preservatives containing bis(tri-*n*-butyltin)oxide: determination of total tin

Confirmed
November 2008

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

Committees responsible for this British Standard

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

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

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Contents

Foreword

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

This Part of BS 5666 was first published in 1980. This edition introduces technical changes but it does not reflect a full review or revision of this Part of the standard, which will be undertaken in due course. BS 5666-7:1980 is withdrawn.

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

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

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

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

Summary of pages

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

0 Introduction

Wood preservative solutions containing bis(tri-*n*-butyltin)oxide are usually formulated with technical grade material containing at least 95 % bis(tri-*n*-butyltin)oxide, e.g. material complying with the requirements of BS 4630. It is known that this compound can be degraded to give dibutyltin and monobutyltin compounds that will result in a decrease in fungistatic activity. It may, therefore, be necessary to determine the organotins present, but in many cases the determination of the total tin content gives adequate information. A further Part of this standard will be published dealing with the determination of the different organotins.

1 Scope

This Part of this British Standard details procedures for the determination of the total tin content present in solutions and in treated wood containing bis(tri-*n*-butyltin)oxide. The methods have been found suitable for the determination of tin in the presence of the following:

copper naphthenate;
dieldrin (HEOD);
γ-hexachlorocyclohexane (γ-HCH, lindane);
pentachlorophenyl laurate;
pentachlorophenol and other chlorophenols;
2-phenylphenol;
zinc naphthenate;
water-repellent waxes and resins.

It is assumed that all the tin determined by these methods has been derived from bis(tri-*n*-butyltin)oxide and hence the tin contents are expressed in terms of bis(tri-*n*-butyltin)oxide.

With aged samples of treated wood, it is occasionally difficult to acid extract all the tin, particularly if iron or some other cations are present. The method described in **2.4.4** has been shown to ensure that, in all cases, the proportion of tin not extracted is unlikely to affect the validity of the determination. The wet ashing technique used in the colorimetric method (see clause **3**) ensures that tin in all types of samples is brought into solution.

The recommendations made as to the size of sample to be taken for the analysis of preservative solutions assume that the bis(tri-*n*-butyltin)oxide content is about 1 % by mass. For samples of other compositions, adjustments may have to be made in the quantity taken for analysis. It is difficult to make recommendations as to the mass of the wood sample to be analysed since the predominant factor is the quantity of preservative within the wood rather than the mass of the wood itself. It may be necessary to make some adjustment to the values quoted in the standard, either to the mass of the treated wood sample taken for analysis or to the level of dilution of the tin solution resulting from extraction. Furthermore, it should be noted that the range of concentrations suggested for the calibration solutions used in these methods may have to be adjusted to suit the sensitivity of the instruments being used.

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

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

2 Method 1. Atomic absorption spectrometric method

2.1 Principle

The tin contents of suitably diluted preservative solutions or wood samples quantitatively extracted with hydrochloric acid in ethanol are analysed using atomic absorption spectrometry.

In this method, any variation in response due to the presence of other butyltin compounds is considered to be within the limits of experimental error of the procedure[1] [2].

It has been found with some atomic absorption spectrometers that the presence of easily ionized metals in the solution, e.g. the alkali metals, enhance the tin absorbance signal. Of these metals, lithium was found to have the greatest effect, and hence lithium is added to all test and calibration solutions. The presence of lithium also minimizes differences in the responses of other organotins.

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[1] The responses of monobutyltin and dibutyltin compounds differ slightly from that of tributyltin compounds. With equivalent tin contents, dibutyltin compounds give an absorbance signal of 3 % to 5 % more than tributylin compounds. As the dibutyltin contents of fresh solutions or recently treated wood are low, this difference is unlikely to be significant.

[2] For reference, see "The determination of bis(tri-*n*-butyltin)oxide and di-*n*-butyltin oxide in preserved softwood by atomic absorption spectrometry and polarography" Williams, A.I., *Analyst*, 1973, 98, 233.

2.2 Reagents

Reagents shall be of recognized analytical reagent grade.

Ethanol (C_2H_5OH), 99 % (*V*/*V*) complying with BS 507 or 99 % (*V*/*V*) industrial methylated spirits complying with BS 3591 shall be used to make up the reagents where ethanol is specified.

NOTE The use of industrial methylated spirits is governed by the Methylated Spirits Regulations 1987 (SI 1987, No. 2009). 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 spirit is an acceptable alternative to ethanol.

2.2.1 *Hydrochloric acid solution A, c* (HCl) about 21 g/L. Quantitatively transfer 25.0 mL of concentrated hydrochloric acid solution (ρ_{20} = 1.18 g/mL) into a 500 mL one-mark volumetric flask and make up to the mark with ethanol.

2.2.2 *Hydrochloric acid solution B, c* (HCl) about 2.1 g/L. Quantitatively transfer 100.0 mL of the hydrochloric acid solution A (**2.2.1**) into a 1 000 mL one-mark volumetric flask and make up to the mark with ethanol.

2.2.3 *Lithium chloride solution* (1 mL = 5 000 μg of lithium). Dissolve 15.30 g of anhydrous lithium chloride (LiCl) in 350 mL of ethanol. Transfer quantitatively to a 500 mL one-mark volumetric flask, add 50.0 mL of the hydrochloric acid solution A (**2.2.1**) and make up to the mark with ethanol.

2.2.4 *Standard bis(tri-n-butyltin)oxide solution*. (1 mL = 200 μg of bis(tri-*n*-butyltin)oxide) $[(C_4H_9)_3Sn]_2O$. Dissolve 0.1000 g of pure bis(tri-*n*-butyltin)oxide[3] in 100 mL of ethanol. Transfer quantitatively to a 500 mL one-mark volumetric flask, add 50.0 mL of the hydrochloric acid solution A (**2.2.1**) and make up to the mark with ethanol.

CAUTION. Bis(tri-*n*-butyltin)oxide is toxic; it is essential that care is taken in handling it.

2.3 Apparatus

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

2.3.2 *Atomic absorption spectrometer*, together with a suitable source of the resonance radiation for tin, 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 tin shall be as recommended by the manufacturer of the spectrometer.

NOTE 1 The solvent, ethanol, will contribute to the fuel in the flame and hence the acetylene flow given for aqueous solutions should be reduced. In order to minimize interference effects from neighbouring resonance lines of copper and zinc, the band pass accepted by the monochromator should be as small as possible, bearing in mind that the additional amplification required for low light levels could result in an unacceptable noise level.

NOTE 2 The tin absorbance signal is enhanced by the presence of easily ionized metals (e.g. the alkali metals) in the solution. Of these metals, lithium has been found to have the greatest effect and hence lithium chloride is added to all test and calibration solutions. The addition of lithium chloride however will necessitate frequent cleaning of the burner head either by scraping or by flushing through with water and ethanol.

The preferred fuel and oxidant are acetylene and nitrous oxide respectively, because interference effects are minimized and the sensitivity is improved.

CAUTION. It is essential that the flow rates of the gases exceed the flame velocity, otherwise a flashback will occur with consequent explosion. It is essential that the operator follow the instructions given for the instrument.

2.4.2 ***Preparation of calibration solutions***

Into a series of 100 mL one-mark volumetric flasks transfer 0, 1.0, 2.0, 3.0, 5.0, 10.0, 15.0, 20.0, 25.0 and 50.0 mL of the bis(tri-*n*-butyltin)oxide standard solution (**2.2.4**), add 20.0 mL of the lithium chloride solution (**2.2.3**) and dilute to the mark with the hydrochloric acid solution B (**2.2.2**). This will provide solutions containing 0, 2, 4, 6, 10, 20, 30, 40, 50 and 100 μg/mL respectively of bis(tri-*n*-butyltin)oxide.

2.4.3 ***Analysis of preservative solutions***

Weigh out accurately 0.4000 g[4] of the preservative into a tared 100 mL one-mark volumetric flask. Add 20.0 mL of the lithium chloride solution (**2.2.3**) and dilute to the mark with the hydrochloric acid solution B (**2.2.2**).

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[3] Bis(tri-*n*-butyltin)oxide readily absorbs carbon dioxide from the atmosphere; precautions should be taken to prevent this during storage.

[4] See clause **1**.

Using the operating conditions suitable for the instrument being used, aspirate successively the hydrochloric acid solution B (**2.2.2**) to obtain the blank absorbance and a suitable range of calibration solutions, aspirating the hydrochloric acid solution B (**2.2.2**) between each calibration solution. Then aspirate the test solution(s) and check the calibration solutions after the last solution has been run.

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

Plot a calibration graph of the concentration of bis(tri-*n*-butyltin)oxide in μg/mL against absorbance. Determine the bis(tri-*n*-butyltin)oxide content of the sample solution by comparing the absorbance reading with the calibration graph and express the result in μg/mL of bis(tri-*n*-butyltin)oxide in the test solution.

2.4.4 *Analysis of treated wood*

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 clause **5** of BS 5666-1:1987.

Divide the prepared sample into two portions each of about 0.5 g. Determine the moisture content on one portion according to the procedure described in clause **6** of BS 5666-1:1987. Reserve the other portion for analysis (**2.4.4.2**).

2.4.4.2 *Extraction and preparation of test solution*

Weigh about 0.5 g of the prepared test sample (**2.4.4.1**) to the nearest 0.01 g into a suitable flask. Add 25.0 mL of the hydrochloric acid solution B (**2.2.2**) and fit a reflux condenser to the flask. Reflux for 10 min and allow to cool. Filter the flask contents through a filter paper[5] into a 50 mL one-mark volumetric flask, wash the contents of the flask into the paper with the hydrochloric acid solution B (**2.2.2**), wash the paper and contents with this acid and dilute to the mark with these washings. Transfer 2.00 mL of the lithium chloride solution (**2.2.3**) into a 10 mL one-mark volumetric flask, make up to the mark with the extract solution and mix.

2.4.4.3 *Measurement*

Using the operating conditions recommended for the instrument being used, aspirate successively the hydrochloric acid solution B (**2.2.2**) to obtain the blank absorbance and a suitable range of calibration solutions, aspirating the hydrochloric acid solution B (**2.2.2**) between each calibration solution. Then aspirate the test solution(s) and check the calibration solutions after the last solution has been run.

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

2.4.5 *Calculations*

2.4.5.1 *The percentage of bis(tri-n-butyltin)oxide in solution*

The percentage by mass of bis(tri-*n*-butyltin)oxide in the solution under test is given by:

$$\frac{C}{100 m_1}$$

where

C is the concentration of bis(tri-*n*-butyltin)oxide, in the test solution (in μg/mL);

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

2.4.5.2 *The percentage of bis(tri-n-butyltin)oxide in treated wood*

The percentage by mass of bis(tri-*n*-butyltin)oxide in the dry wood is given by:

$$\frac{C\ (100 + h)}{16000\ m_2}$$

where

C is the concentration of bis(tri-*n*-butyltin)oxide, in the test solution (in μg/mL);

h is the percentage moisture content by mass of the prepared timber sample (**2.4.4.1**);

m_2 is the mass of the prepared sample of timber, (in g).

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[5] A Whatman No. 42 filter paper has been found to be suitable.

3 Method 2. Colorimetric method

3.1 Principle

After destruction of organic matter, tin is converted into tin (IV) iodide and extracted into toluene from a 4.5 mol/L sulphuric acid solution. It is then extracted into aqueous sodium hydroxide solution and, after acidification and removal of liberated iodine, the tin is determined spectrometrically as its complex with catechol violet[6].

The range of the colorimetric procedure is 1 µg to 30 µg of tin, which is equivalent to 2.5 µg to 75 µg of bis(tri-*n*-butyltin)oxide. Appropriate dilution of the solution before extraction of tin(IV) iodide may be required.

3.2 Reagents

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

Ethanol (C_2H_5OH), 99 % (*V*/*V*) complying with BS 507 or 99 % (*V*/*V*) industrial methylated spirits complying with the requirements of BS 3591 shall be used to make up the reagents where ethanol is specified.

NOTE The use of industrial methylated spirits is governed by the Methylated Spirits Regulations 1987 (SI 1987, No. 2009). 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 spirit is an acceptable alternative to ethanol.

3.2.1 *Acidic potassium iodide solution*, 75 g/L. Mix 2.5 mL of the potassium iodide solution (**3.2.6**) and 25 mL of the sulphuric acid solution (**3.2.10**).

3.2.2 *Ammonia solution, diluted c* (NH_3) about 4 mol/L. Mix 1 volume of concentrated ammonia solution, (ρ_{20} = 0.880 g/mL) with 3.6 volumes of water.

3.2.3 *L-ascorbic acid solution*, 50 g/L. Dissolve 5 g of L-ascorbic acid $[\underbrace{\mathrm{O.CO.C(OH){:}\ C(OH).C}}\mathrm{H.CH(OH)CH_2OH}]$ in 100 mL of water. Prepare freshly as required.

3.2.4 *Catechol violet (pyrocatechol sulphonphthalein) solution*, 0.5 g/L. Dissolve 0.05 g of catechol violet $[[C_6H_3(OH_2)]\ [C_6H_4SO_3H]\ C{:}\ C_6H_3O(OH)]$ in 100 mL of ethanol. Keep in a cool dark place. The solution is stable for one week.

3.2.5 *Hydrochloric acid solution, c* (HCl), 5.5 mol/L. Mix equal volumes of concentrated hydrochloric acid solution (ρ_{20} = 1.18 g/mL) and water.

3.2.6 *Potassium iodide solution*, 830 g/L. Dissolve 83 g of potassium iodide (KI) in 100 mL of water. Prepare freshly as required.

3.2.7 *Sodium acetate solution*, 200 gL. Dissolve 20 g of sodium acetate trihydrate ($CH_3COONa.3H_2O$) in 100 mL of water.

3.2.8 *Sodium hydroxide solution A*, 200 g/L. Dissolve 20 g of sodium hydroxide (NaOH) in 100 mL of water.

3.2.9 *Sodium hydroxide solution B*, 4 g/L. Dissolve 0.4 g of sodium hydroxide (NaOH) in 100 mL of water.

3.2.10 *Sulphuric acid solution c* (H_2SO_4), 4.5 mol/L. Cautiously add 25 mL of concentrated sulphuric acid (ρ_{20} = 1.84 g/mL) to 50 mL of water; cool and dilute to 100 mL with water.

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

3.2.11 *Standard tin solution* (1 mL = 10 µg of tin). Dissolve 0.1000 g of pure granulated tin in 20 mL of concentrated sulphuric acid (**3.2.12**) by heating until fumes appear. Cool, dilute cautiously with 150 mL of water and cool. Cautiously add 65 mL of concentrated sulphuric acid (**3.2.12**), cool, transfer to a 500 mL one-mark volumetric flask and dilute to the mark with water.

As required, prepare the standard tin solution by transferring 5.00 mL of this solution to a 100 mL one-mark volumetric flask and diluting to the mark with water.

3.2.12 *Concentrated sulphuric acid* (H_2SO_4), (ρ_{20} = 1.84 g/mL)

3.2.13 *Nitric acid* (HNO_3), (ρ_{20} = 1.42 g/mL).

3.2.14 *Perchloric acid* ($HClO_4$), (ρ_{20} = 1.68 g/mL).

3.2.15 *Toluene* ($C_6H_5.CH_3$)

3.3 Apparatus

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

3.3.2 *Spectrometer or photoelectric absorptometer*, suitable for the measurement of absorbance at a wavelength of 552 nm and fitted with appropriate cells.

3.3.3 *pH meter*, complying with BS 3145, capable of reading to 0.1 pH unit.

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[6] For reference, see "Reports of the Metallic Impurities in Organic Matter Subcommittee to the Analytical Methods Committee", *Analyst*, 1967, 92, 320, and *Analyst*, 1968, 93, 414.

3.4 Procedure

3.4.1 *Preparation of the calibration graph*

Transfer to a series of 50 mL beakers suitable volumes of standard tin solution (**3.2.11**) to cover the range 0 μg to 30 μg of tin, and dilute the contents of each beaker to 7 mL with water. Treat each solution as follows[7].

Add 1.0 mL of sodium hydroxide solution A (**3.2.8**) and mix; add 2.5 mL of the hydrochloric acid solution 5.5 mol/L (**3.2.5**) and mix; add 2.0 mL of the catechol violet solution (**3.2.4**), mix and add 5 mL of the sodium acetate solution (**3.2.7**).

Adjust the pH of each solution with the diluted ammonia solution (**3.2.2**) to 3.8 ± 0.2 using the pH meter (**3.3.3**), transfer to a series of 25 mL one-mark volumetric flasks, dilute to the mark with water, mix well and set aside. Measure the absorbance of each solution after 30 min at a wavelength of 552 nm. Prepare the calibration graph by plotting the absorbance values against micrograms of tin.

3.4.2 *Analysis of preservative solutions*

Take an accurately known mass in the range of 0.25 g to 0.5 g of preservative solution in a suitable beaker (100 mL or 250 mL capacity) and proceed as described in **3.4.4**.

3.4.3 *Analysis of treated wood*

3.4.3.1 *Preparation of timber sample for analysis*

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

Divide the prepared sample into two unequal portions, (0.5 g and 0.1 g respectively). Determine the moisture content on the 0.5 g portion according to the procedure described in clause **6** of BS 5666-1:1987. Reserve the 0.1 g sample for the analysis (**3.4.3.2**).

3.4.3.2 *Extraction and preparation of test solution*

Weigh about 0.1 g of the prepared test sample (**3.4.3.1**) to the nearest 0.001 g into a suitable beaker (100 mL to 250 mL capacity) and proceed as described in **3.4.4**.

3.4.4 *Wet ashing procedure*

Add 5 mL of the concentrated sulphuric acid (**3.2.12**) and approximately 2 mL to 3 mL of the nitric acid (**3.2.13**) to the sample in the beaker. Cover and allow any reaction to take place before placing on a hot plate. Heat and allow to char, ensuring that an excess of oxidant is maintained (denoted by the evolution of brown fumes) by the addition of the nitric acid (**3.2.13**) drop by drop. Continue heating with the cover partly removed until all the organic matter has been destroyed and the solution is transparent but coloured when heated to evolution of sulphur trioxide fumes[8]. Maintain at this temperature and add nitric acid (**3.2.13**) drop by drop to give a pale yellow to colourless solution. Cool, add 5 mL to 10 mL of water and heat to boiling, allow to evaporate until sulphur trioxide fumes are evolved; if the solution is still pale yellow and the colour persists on the addition of more nitric acid (**3.2.13**), cool, add approximately 0.5 mL of the perchloric acid (**3.2.14**) and 0.5 mL of the nitric acid (**3.2.13**), and heat until sulphur trioxide fumes are evolved. The solution should be water white. Cool, add approximately 10 mL water[9], cool, transfer quantitatively to a 25 mL one-mark volumetric flask with the sulphuric acid solution (**3.2.10**) and make up to the mark with the sulphuric acid solution (**3.2.10**).

3.4.5 *Adjustment of the tin content*

As necessary, dilute the solution prepared as described in **3.4.4** so that the concentration of tin is suitable for the method, i.e. within the range of 1 μg to 30 μg in 25 mL of solution. Record the dilution factor.

For example, if the tin content of the solution is expected to be in the range of 100 μg to 3 mg of tin (i.e. equivalent to 250 μg to 7.5 mg of bis(tri-*n*-butyltin)oxide) proceed as follows.

Quantitatively transfer 10.00 mL of the solution (from **3.4.4**) to a 100 mL one-mark volumetric flask, dilute to the mark with the sulphuric acid solution (**3.2.10**) and mix. Quantitatively transfer 10.00 mL of this solution to another 100 mL one-mark volumetric flask, dilute to the mark with the sulphuric acid solution (**3.2.10**) and mix.

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[7] The order of addition of the reagents is important and it is essential that the stated order is strictly followed.

[8] Care should be taken to ensure that losses of sulphur trioxide are small, otherwise the final solution will not be 4.5 mol/L with respect to the sulphuric acid.

[9] This volume of water should be adjusted to allow for the amount of sulphuric acid left at this stage. After addition of water, the concentration of acid should be 4.5 mol/L.

3.4.6 *Separation of tin*

Quantitatively transfer 25.00 mL of the solution (prepared as described in **3.4.4** or diluted as described in **3.4.5**) to a separating funnel. Add 2.5 mL of the potassium iodide solution (**3.2.6**) to the solution in the separating funnel and mix. Add 10 mL of toluene (**3.2.15**), shake the mixture vigorously for 2 min, allow the layers to separate, and discard the lower aqueous phase. Wash the toluene layer with 5 mL of the acidic potassium iodide solution (**3.2.1**), swirling the mixture gently but not shaking it, and discard the washings[10]. Add 5 mL of water to the washed toluene solution, followed by the sodium hydroxide solution A (**3.2.8**) drop by drop, shaking the mixture after the addition of each drop, until the toluene solution is colourless and then add 0.1 mL (2 drops) of the sodium hydroxide solution A (**3.2.8**) in excess[11]. Shake the mixture for 30 s, allow the layers to separate, and transfer the lower aqueous layer to a 50 mL beaker. Wash the toluene layer with 3 mL of the sodium hydroxide solution B (**3.2.9**), shaking the mixture for 30 s, and add the washings to the aqueous alkaline solution in the beaker; reserve the toluene solution.

3.4.7 *Determination of tin*

Acidify the combined alkaline extract and washings in the beaker obtained as described in **3.4.6** with 2.5 mL of the hydrochloric acid solution (**3.2.5**) and decolorize the liberated iodine by the addition of the L-ascorbic acid solution (**3.2.3**) drop by drop. Add 2.0 mL of the catechol violet solution (**3.2.4**) and mix.

Wash the reserved toluene with 5 mL of the sodium acetate solution (**3.2.7**), swirling the mixture but not shaking it, add the aqueous washings to the solution in the beaker and mix. Adjust the pH value of the solution with the diluted ammonia solution (**3.2.2**) to 3.8 ± 0.2 using the pH meter (**3.3.3**). Transfer the solution to a 25 mL one-mark volumetric flask, dilute to the mark with water, mix well and set aside. Measure the absorbance of the test solution after 30 min at a wavelength of 552 nm.

To determine the tin content, compare the absorbance value with the calibration graph prepared as described in **3.4.1**.

3.4.8 *Calculations*

3.4.8.1 *Percentage of bis(tri-n-butyltin)oxide in solution*

The percentage, by mass, of bis(tri-*n*-butyltin)oxide in the solutions under test is given by:

$$\frac{2.5 m_3 n}{M_1} \times 10^{-4}$$

where

m_3 is the amount of tin found (in μg);

n is the dilution factor (see **3.4.5**);

m_1 is the mass of preservative solution (in g).

NOTE The factor of 2.5 is for the conversion of tin content to bis(tri-*n*-butyltin)oxide content.

3.4.8.2 *Percentage of bis(tri-n-butyltin)oxide in treated wood*

The percentage, by mass, of bis(tri-*n*-butyltin)oxide in the dry wood is given by:

$$\frac{2.5 m_4 n \; (100 + h)}{10^6 M_2}$$

where

m_4 is the amount of tin found (in μg);

n is the dilution factor (see **3.4.5**);

h is the percentage moisture content by mass of the prepared timber sample (**3.4.3.1**);

M_2 is the mass of the prepared sample of timber (in g).

4 Test report

The test report shall include the following particulars:

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

b) a reference to this British Standard and the method used, e.g. BS 5666-7, method 2;

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

d) the results of the analysis and the method of expression used, e.g. 1.0 % by mass of bis(tri-*n*-butyltin)oxide;

e) any unusual features noted during the analysis.

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[10] The toluene layer will be coloured pink with extracted iodine.

[11] Usually a total of 8 to 10 drops of sodium hydroxide solution is required.

Publication(s) referred to

BS 507, *Specification for ethanol for industrial use.*
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 3145, *Specification for laboratory pH meters.*
BS 3591, *Specification for industrial methylated spirits.*
BS 3978, *Specification for water for laboratory use.*
BS 4630, *Specification for tributyltin oxide [di(tributyltin)oxide].*
BS 5666, *Methods of analysis of wood preservatives and treated timber.*
BS 5666-1, *Guide to sampling and preparation of wood preservatives and treated timber for analysis.*

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