

Australian/New Zealand Standard™

**Methods for sampling and analysing
timber preservatives and preservative-
treated timber**

**Part 3: Analysis methods for
determination of preservative retention**



AS/NZS 1605.3:2018

This Joint Australian/New Zealand Standard was prepared by Joint Technical Committee TM-012, Timber Grading and Preservation. It was approved on behalf of the Council of Standards Australia on 22 December 2017 and by the New Zealand Standards Approval Board on 5 March 2018.
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The following are represented on Committee TM-012:

Australian and New Zealand Timber Preservative Manufacturers Association
Australian Forest Products Association
Australian Pesticides and Veterinary Medicines Authority
Australian Timber Flooring Association
Australian Timber Importers Federation
Building Research Association of New Zealand
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Australian/New Zealand Standard™

Methods for sampling and analysing timber preservatives and preservative- treated timber

Part 3: Analysis methods for determination of preservative retention

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PREFACE

This Standard was prepared by the Joint Standards Australia/Standards New Zealand Committee TM-012, Timber Grading and Preservation, to supersede AS/NZS 1605.3:2006.

The objective of this revision is to add analytical procedures to determine—

- (a) triadimefon and cyproconazole in glueline-treated and surface-treated timber;
- (b) quaternary ammonium compound in wood treated with ACQ preservatives;
- (c) didecyldimethyl ammonium chloride in wood treated with ACQ preservatives;
- (d) zinc borate in reconstituted wood products;
- (e) thiacloprid in glueline-treated plywood and laminated veneer lumber (LVL); and
- (f) deltamethrin in glueline-treated engineered wood products.

The objective of this Standard is to set out methods for the sampling and analysing of preservative treated timber.

AS/NZS

- 1605 Methods for sampling and analysing timber preservatives and preservative-treated timber
- 1605.1 Part 1: General requirements, sampling, and determination of sapwood and heartwood presence
- 1605.2 Part 2: Determination of preservative penetration by spot tests
- 1605.3 Part 3: Analysis methods for determination of preservative retention (this Standard)
- 1605.4 Part 4: Analysis methods for determination of preservative solution concentration

This Standard is intended to be read in conjunction with AS/NZS 1605.1 and the AS/NZS 1604 series, NZS 3640 or the relevant Standard to which timber is claimed to have been treated. The preservatives incorporated in the AS/NZS 1604 series are required to be accompanied by analytical methods for their determination, which are incorporated in the AS/NZS 1605 series.

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STANDARDS AUSTRALIA/STANDARDS NEW ZEALAND

Australian/New Zealand Standard**Methods for sampling and analysing timber preservatives and preservative-treated timber****Part 3: Analysis methods for determination of preservative retention**

SECTION 1 SCOPE AND GENERAL

1.1 SCOPE

This Standard specifies analysis methods for determining preservative retention in preservative-treated wood products. The calculations necessary for determining compliance with the requirements specified in the AS/NZS 1604 series and NZS 3640 are outlined.

1.2 APPLICATION

This Standard should be read in conjunction with—

- (a) the AS/NZS 1604 series, NZS 3640 or the relevant Standard to which timber is claimed to have been treated; and
- (b) AS/NZS 1605.1.

Methods for sampling and analysing timber preservatives and preservative-treated timber are categorized into the following parts of the AS/NZS 1605 series, according to the general nature of the tests:

- (i) Sampling preservative liquids and preservative-treated timber Part 1.
- (ii) Determination of presence of sapwood and heartwood Part 1.
- (iii) Determination of preservative penetration by spot tests Part 2.
- (iv) Analysis of preservative retention Part 3.
- (v) Analysis of preservative solution concentration Part 4.

1.3 REFERENCED DOCUMENTS

The following documents are referred to in this Standard:

AS/NZS

- 1604 Specification for preservative treatment (all parts)
- 1605 Methods for sampling and analysing timber preservatives and preservative-treated timber (all parts)
- 1605.1 Part 1: General requirements, sampling, and determination of sapwood and heartwood presence

AS/NZS

- 4491 Timber—Glossary of terms in timber-related Standards

NZS

- 3640 Chemical preservation of round and sawn timber

1.4 DEFINITIONS

Refer to AS/NZS 1605.1 for applicable definitions.

1.5 PREPARATION OF TEST PIECES FROM TEST SPECIMEN

Test pieces shall be prepared from test specimens cut according to AS/NZS 1605.1, from samples taken according to AS/NZS 1605.1.

The material for analysis shall be evenly cut from across the full cross-section of the penetration zone for the appropriate hazard class specified in the AS/NZS 1604 series or the relevant Standard.

1.6 USE OF ANALYTICAL METHODS FOR EXAMINATION OF PRESERVATIVE PENETRATION

Analytical methods may be used where no effective spot test is available. In such cases, the sample shall be taken from the central ninth of the sapwood penetration zone.

Where heartwood penetration or an envelope is specified, additional samples shall be taken adjacent to the boundary of the required penetration zone. Each sample taken shall be analysed individually for the presence of preservative. For example, in the case of an H3 sample, the central ninth of the penetration zone is required to be analysed as one sample, and samples taken from the heartwood analysed separately.

1.7 RELATED METHODS OF ANALYSIS

The analytical methods given in this Standard are intended as referee methods for the determination of timber preservative retentions. Alternative methods should be compared with methods presented in this Standard before being routinely used, provided that they are of comparable accuracy, e.g. copper chromium arsenic (CCA) treated timber may be analysed by atomic absorption spectrophotometry (AAS), X-ray diffraction (XRF), or inductively-coupled plasma (ICP) methods, and boron-treated timber by alternative water extracted titration method.

SECTION 2 DETERMINATION OF COPPER, CHROMIUM AND ARSENIC IN PRESERVATIVE-TREATED TIMBER

2.1 PRINCIPLE

Copper, chromium and arsenic compounds are leached quantitatively from the timber sample with a mixture of diluted sulphuric acid and hydrogen peroxide solution. After adding sodium sulphate to the resulting extracts, the elemental concentrations are determined by atomic absorption spectrophotometry.

NOTE: The method may be used for copper alone in determining the copper content in timber treated with copper-based preservatives.

2.2 REAGENTS

All reagents shall be of analytical reagent quality. The water used for preparing the reagents shall be distilled.

The following reagents shall be used:

- (a) *Sulphuric acid, 2.5 M* Cautiously add, with stirring and cooling, 280 mL sulphuric acid (sg = 1.84 g/mL) to 1500 mL water. Cool and dilute with water to 2000 mL.
- (b) *Hydrogen peroxide solution, 30% (100 volume)*.
- (c) *Sodium sulphate, 3.0% solution* Dissolve 30 g anhydrous sodium sulphate in water and dilute with water to 1000 mL.
- (d) *Sulphuric acid, 0.5 M/sodium sulphate 3.0% diluent solution* Dilute 200 mL sulphuric acid solution [Clause 2.2(a)] plus 100 mL of 3.0% sodium sulphate solution to 1000 mL with water and mix.
- (e) *Standard solution (1 mL = 500 µg of arsenic)*

The standard solution shall be prepared as follows:

- (i) Dissolve 0.9825 g copper sulphate pentahydrate, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, in a little water and transfer the solution to a 500 mL one-marked volumetric flask.
- (ii) Dissolve 1.4135 g potassium dichromate, $\text{K}_2\text{Cr}_2\text{O}_7$, in a little water, add 50 mL sulphuric acid solution [Clause 2.2(a)] and 10 mL hydrogen peroxide solution [Clause 2.2(b)]. Boil, cool and transfer the mixture to the one-marked volumetric flask containing the copper sulphate solution.
- (iii) Dissolve 0.6600 g arsenic trioxide, As_2O_3 , by boiling it in a solution containing 50 mL sulphuric acid solution [Clause 2.2(a)] 10 mL hydrogen peroxide solution [Clause 2.2(b)] and 75 mL water. Cool and transfer it to the one-marked volumetric flask. Add 50 mL sodium sulphate solution [Clause 2.2(c)] to the one-marked volumetric flask. Dilute to the mark with water and mix.

2.3 PROCEDURE

2.3.1 Instrument settings and operation

The instrument settings and operating conditions for determining copper, chromium and arsenic shall be as recommended in the instrument user's manual.

Copper shall be determined in a fuel-lean air/acetylene flame at 327.4 nm, chromium in a fuel-lean nitrous oxide/acetylene flame at 357.9 nm, and arsenic in a nitrous oxide/acetylene flame at 193.7 nm, with background correction for non-atomic absorption.

2.3.2 Preparation of calibration solutions

The procedure for the preparation of the calibration solutions shall be as follows:

- (a) Transfer aliquot portions of 2, 4, 8 and 16 mL of the standard solution [Clause 2.2(e)] to a series of 200 mL one-marked volumetric flasks.
- (b) Dilute to the mark with sulphuric acid/sodium sulphate solution [Clause 2.2(d)] and mix. When made up this way, 1 mL aliquot portions of this solution contain 5, 10, 20 and 40 µg, respectively, of copper and 10, 20, 40, and 80 µg, respectively, of chromium and of arsenic.

It is necessary to adjust the number of calibration solutions and the range of concentrations covered to suit the sensitivity of the instrument used.

2.3.3 Analysis of treated timber

The procedure for the analysis of treated timber shall be as follows:

- (a) Comminute the dry timber sample so that it will pass through a 0.1 cm mesh. Oven-dry at 103°C before weighing a representative portion for analysis. Accurately weigh—
 - (i) ≈2 g for H1, H2, or H3;
or
 - (ii) ≈1 g for all other hazard class treatments.
- (b) Transfer the timber sample to a 250 mL conical flask, add 40 mL sulphuric acid solution [Clause 2.2(a)] and, with caution, 8 mL hydrogen peroxide solution [Clause 2.2(b)].
- (c) Heat at 75°C in a water bath for 30 min with occasional swirling to mix the contents of the flask.
- (d) Remove the flask from the water bath, add 80 mL water and 20 mL sodium sulphate solution [Clause 2.2(c)] and cool the flask and contents to room temperature.
- (e) Transfer the contents quantitatively to a 200 mL one-marked volumetric flask, dilute to the mark with water and mix.
- (f) Filter a portion of this solution through a Whatman No. 541 filter paper, discarding the first 50 mL, to give the test solution.
- (g) Using the operating conditions suitable for the instrument, aspirate the sulphuric acid/sodium sulphate solution [Clause 2.2(d)] to obtain the blank absorbance, followed by a suitable range of calibration solutions and the test solution(s).
- (h) Check the calibration solutions after the last test solution has been run.
- (i) Plot the calibration curves of copper, chromium and arsenic (in µg/mL) against absorbance.
- (j) Determine the contents of copper, chromium and arsenic in the test solutions by comparing the absorbance readings with the calibration curves.

If a number of samples are to be analysed, it is necessary to check the instrument stability by bracketing samples with appropriate standard solutions.

2.4 CALCULATIONS OF THE PERCENTAGES OF METALS IN TREATED TIMBER

The percentage by mass of copper or chromium or arsenic in the dry timber shall be calculated from the following equation:

$$\text{Percent metal} = \frac{X}{50 m} \quad \dots 2.4$$

where

X = the concentration of the appropriate metal in $\mu\text{g/mL}$ of the test solution
(Clause 2.3.3)

m = the oven-dried mass of the timber sample taken, in grams

Total active element concentration is determined by adding the values for the percent metal for each component.

2.5 COMPOSITION OF COPPER CHROMIUM ARSENIC (CCA)

The composition of copper chromium arsenic (CCA) shall be calculated to determine whether it conforms to the element ratios as shown in the AS/NZS 1604 series.

SECTION 3 DETERMINATION OF BORON IN PRESERVATIVE-TREATED TIMBER

3.1 PRINCIPLE

After dry-ashing the timber sample in lime-eschka mixture, boron is determined titrimetrically by using mannitol.

3.2 REAGENTS

All reagents shall be of analytical reagent quality. The water used for preparing the reagents shall be distilled.

The following reagents shall be used:

- (a) *Lime eschka* Mix 250 g anhydrous sodium carbonate and 750 g anhydrous powdered calcium oxide.
- (b) *Mannitol solution* Dissolve 15 g mannitol in 100 mL water. Prepare freshly before use because of biodegradation.
- (c) *Phenolphthalein indicator* Dissolve 0.25 g phenolphthalein in 50 mL ethanol, then add 50 mL water. Adjust pH by adding dilute sodium hydroxide solution until the faint pink colouration persists.
- (d) *Methyl red indicator* Filtered, saturated solution of methyl red in ethanol.
- (e) *Standard sodium hydroxide solutions, 0.05M* Dissolve 2 g sodium hydroxide in 1000 mL water, and standardize as follows:
 - (i) Accurately weigh three 0.1 g portions of potassium hydrogen phthalate (KHP) and dissolve in 25 mL water. After adding 0.5 mL phenolphthalein indicator, titrate with the sodium hydroxide solution.
 - (ii) The molarity (M) is given by the following equation:

$$M = \frac{(\text{Weight of KHP}) \times 1000}{(\text{Titre}) \times 204.22} \quad \dots 3.2$$

3.3 PROCEDURE FOR ANALYSIS OF TREATED TIMBER

The procedure for the analysis of treated timber shall be as follows:

- (a) Commminute the timber sample so that it will pass through a 0.1 cm mesh. Oven-dry at 70°C before taking a representative portion for analysis.
- (b) Weigh 1.5 g lime eschka into a numbered porcelain crucible. Accurately weigh a portion (≈ 2.5 g) of sample into the crucible and mix immediately with the lime eschka. Ash at 550°C for 1 h and allow to cool.
- (c) Transfer the ash to a beaker and rinse out the crucible with small portions of dilute (10%) hydrochloric acid and water.
- (d) Transfer the washings to the beaker, making sure the sides of the crucible are rubbed down with a glass rod fitted with a short length (about 15 mm to 20 mm) of rubber tubing.
- (e) Carefully add 10 mL concentrated hydrochloric acid to the beaker, covering with a watch-glass to prevent spattering.

NOTE: More acid may be required if the solid contents are not completely dissolved.

- (f) Wash down the watch-glass and beaker sides with distilled water and add 1 mL phenolphthalein indicator.
- (g) Turn the solution just alkaline with cool, freshly prepared 15% sodium hydroxide solution. Stir vigorously and add drop-wise to achieve the pink-grey precipitate colour required.
- (h) Quantitatively transfer the contents of the beaker to a 100 mL volumetric flask, and make up to the mark when cool. (The precipitate is transferred to the volumetric flask.)
- (i) Filter a portion of the solution through a Whatman No. 542 filter paper.
- (j) Pipette 25 mL of the filtrate into a 250 mL Erlenmeyer flask, dilute to 50 mL with water and add 1 mL phenolphthalein indicator.
- (k) Add dilute hydrochloric acid drop-wise until phenolphthalein turns colourless.
- (l) Add 0.5 mL methyl red indicator, which should turn yellow.
- (m) Add dilute hydrochloric acid drop-wise until methyl red turns red. If the solution stands for 4 h or more, the acid aliquot shall be refluxed to eliminate carbon dioxide, and the solution shall be cooled.
- (n) Add the standard sodium hydroxide solution drop-wise until the red colour is just discharged, leaving the solution straw yellow.
- (o) Add 10 mL 15% freshly prepared mannitol solution, whereupon the solution should again turn to the red colour of methyl red.
- (p) Titrate the solution with the standard sodium hydroxide. During the course of titration the colour will change from *red* to the *yellow* of methyl red, and then to the *pink* of phenolphthalein. The end-point is the first permanent appearance of this pink colour.
- (q) Run a blank determination in conjunction with the above procedure, using 2.50 g ground, untreated sapwood in the initial stages.
- (r) Obtain the titre for this blank determination in exactly the same way as for the sample determination.

3.4 CALCULATIONS

The percentage by mass of boron in the dry timber shall be calculated from the following equation:

$$\text{Percent boron} = 1.7288 (T - B) (M) \quad \dots 3.4$$

where

T and B = titre and blank volumes of M standard sodium hydroxide solution
[Clause 3.2(e)(ii)]

M = molarity [see Clause 3.2(e)(ii)]

SECTION 4 DETERMINATION OF FLUORIDE IN PRESERVATIVE-TREATED TIMBER

4.1 PRINCIPLE

The timber sample is fused with sodium hydroxide. The melt is dissolved in water, and fluoride level is determined by ion-selective electrode.

4.2 REAGENTS

All reagents shall be of analytical reagent quality. The water used for preparing the reagents shall be distilled.

The following reagents shall be used:

- (a) *Sodium hydroxide concentrate, 60%* Dissolve 600 g sodium hydroxide in 1000 mL water. Cool and store in a polyethylene container.
- (b) *SSA-EDTA buffer* Dissolve 204 g sulphasalicylic acid (SSA) and 74 g disodium dehydrate EDTA in 700 mL water and adjust the pH to 9.5 with concentrated (s.g.:0.880) ammonia solution before dilution to 1000 mL. This buffer is 0.8M SSA and 0.2M EDTA.
- (c) *Fluoride standard, 1000 µg/mL* Dissolve 2.210 g sodium fluoride in water and dilute to 1000 mL. Store in a polyethylene container.

4.3 PROCEDURE

4.3.1 Instrument operation

An ion-selective meter shall be used to determine fluoride level.

Several different types of ion-selective meters are available, and each has different operating characteristics. The operating manual shall be followed.

Particular attention shall be given to measuring electrode slope and setting the meter to the current slope value; solutions shall be gently stirred during electrode equilibration.

4.3.2 Preparation of calibration solutions

The procedure for the preparation of the calibration solutions shall be as follows:

- (a) Transfer a 200 mL aliquot of the 1000 µg/mL fluoride standard [Clause 4.2(c)] to a 1000 mL one-marked volumetric flask, dilute to the mark and mix well.
- (b) Transfer aliquot of 1 and 10 mL of this 200 µg/mL standard solution to 200 mL standard solution to 200 mL one-marked volumetric flasks.
- (c) Transfer a 20 mL aliquot of the original 1000 µg/mL fluoride standard [Clause 4.2(c)] to a third 200 mL one-marked volumetric flask.
- (d) To each of the three volumetric flasks, add 10 mL sodium hydroxide concentrate [Clause 4.2(a)] and 100 mL SSA-EDTA buffer [Clause 4.2(b)], dilute to the mark and mix well. These calibration solutions contain 1, 10 and 100 µg fluoride/mL, respectively.
- (e) Store the calibration solutions in polyethylene containers.

4.3.3 Analysis of treated timber

The procedure for the analysis of treated timber shall be as follows:

- (a) Comminute the timber sample so that it will pass through a 0.1 cm mesh. Oven-dry at 70°C before taking a representative portion for analysis.
- (b) Weigh 2 g ground timber into a 70 mL nickel crucible.
- (c) Add 10 mL sodium hydroxide concentrate, and stir to wet all the material in the crucible.
- (d) Evaporate to dryness in an oven at 80°C.
- (e) Place in a muffle furnace and raise the temperature to 550°C, holding that temperature for 1 h.
- (f) Cool the crucible in a desiccator, add 30 mL water, and warm until the contents dissolve.
- (g) Transfer the solution to a 100 mL volumetric flask and dilute to the mark with water.
- (h) Filter the solution (or allow the solids to settle first) and pipette 25 mL into a small beaker, together with 25 mL SSA-EDTA buffer.
- (i) Using suitable operating procedures for the electrode/meter combination, use calibration solutions (Clause 4.3.2) and set up the instrument calibration at 1, 10 and 100 µg/mL fluoride, ensuring that sufficient equilibration time is allowed, especially at 1 µg/mL.
- (j) Read the fluoride content of the solutions on the calibrated instrument, together with a reagent blank solution.
- (k) If the fluoride content of the timber is expected to be below 0.2% sodium fluoride, reduce the dilution to 100 mL of the crucible contents to 50 mL, and introduce an appropriate factor into the calculations.

4.4 CALCULATIONS

The percentage by mass of fluoride (NaF) in the dry timber, expressed in terms of equivalent sodium fluoride, shall be calculated from the following equation:

$$\text{Percent NaF} = 0.02210 X \quad \dots 4.4$$

where

X = the fluoride concentration of the test solution, in µg/mL

SECTION 5 DETERMINATION OF PENTACHLOROPHENOL (PCP) IN PRESERVATIVE-TREATED TIMBER

5.1 PRINCIPLE

Pentachlorophenol is leached quantitatively from the timber sample with methanol, using ultrasonic agitation. The resulting solution is determined by a high-performance liquid-chromatography system.

5.2 REAGENTS

All reagents shall be of chromatographic reagent quality. The water used for preparing the reagents shall be distilled.

The following reagents shall be used:

- (a) *Methanol*.
- (b) *Acetonitrile:water:acetic acid (75:25:0.1)* Add 1 mL acetic acid to a mixture of 750 mL acetonitrile and 250 mL water. Degas before use.
- (c) *Acetonitrile:acetic acid (100:1)* Add 1 mL acetic acid into acetonitrile. Make up to 1000 mL. Degas before use.
- (d) *Pentachlorophenol* Purified for use as a standard.
- (e) *Standard solutions* Working standards should contain 0.002% m/v and 0.020% m/v pure pentachlorophenol in methanol.

5.3 PROCEDURE

5.3.1 Instruments, settings and operation

A high-performance liquid-chromatography system shall be used. The system shall be capable of step-gradient in solvent composition at a specified run time, with UV detection at 220 nm and peak area integration. Ultrasonic treatment shall be carried out using a thermally insulated ultrasonic bath with a power rating of approximately 50 W/L.

The high-performance liquid-chromatography system consists of the following:

- (a) *Detector settings* 0.30 AUFS for Waters 450 at 220 nm.
- (b) *Column* Waters Associates microbondapak C-18, 30 cm × 4 mm steel.

5.3.2 Preparation of samples

The procedure for the preparation of the samples shall be as follows:

- (a) Comminute the dry timber sample so that it will pass through a 0.2 cm mesh.
- (b) Take 0.5 g to 1 g mass from the specified zone such that its maximum thickness in the longitudinal direction is 4 mm.

NOTE: Larger samples may be taken provided the thickness is not increased and the total extracting volume is adjusted accordingly.
- (c) Place the sample in a 50 mL beaker with 25 mL methanol. Cover and place in an ultrasonic bath at 50°C to 55°C for 1 h.
- (d) Decant extract into a 50 mL volumetric flask and re-extract sample with second portion of methanol. Combine extracts, cool, and make up to the mark with methanol.

- (e) Dry the extracted timber sample at 100°C for 4 h before weighing to 0.01 g.
- (f) Submit the extract to the high-performance liquid-chromatography system.

5.3.3 Analysis of treated timber

The procedure for the analysis of the treated timber shall be as follows:

- (a) Filter extracts or standards through a 0.5 µm PTFE filter in an 8 mm Swinnex filter holder.
- (b) Inject 20 µL of extract of standards into the high-performance liquid-chromatography system with an initial solvent flow rate of 2.5 mL/min of 75:25:0.1 acetonitrile: water: acetic acid until 2 min after injection.
- (c) Step to 100:0.1 acetonitrile: acetic acid for 2 min, return to 75:25:0.1 acetonitrile:water:acetic acid for 4 min.
- (d) Inject 2 or 3 times per sample or standard.
- (e) Compare peak areas or percent mass/volume pentachlorophenol in sample extracts.

5.4 CALCULATIONS

The percent mass/mass of pentachlorophenol (PCP) on an oven-dried timber basis shall be calculated from the following equation:

$$PCP (\% m/m) = \frac{50 \times E}{m} \quad \dots 5.4$$

where

E = percent PCP mass/volume in the final extract

m = oven-dried mass of timber, in grams

SECTION 6 DETERMINATION OF CREOSOTE OR PIGMENT-EMULSIFIED CREOSOTE IN PRESERVATIVE-TREATED TIMBER

6.1 PRINCIPLE

The retention of creosote, pigment-emulsified creosote, mineral oil and pentachlorophenol, or combinations of these, is measured by extracting these from a weighed timber sample using toluene as a solvent and expressing the mass loss as a percentage of the oven-dried extracted timber mass. The moisture content of the timber shall lie between 8% to 18% of the oven-dried extracted timber mass, or an appropriate volume correction shall be made.

6.2 REAGENT—TOLUENE

NOTE: Laboratory reagent-grade toluene may be used if it has been distilled to remove water.

6.3 PROCEDURE

6.3.1 Determination of moisture content

The moisture content of the test piece is the mass of water recovered during the extraction of preservative and is expressed as a percentage of the mass of extracted timber after oven-drying in a well-ventilated oven at $105 \pm 2^\circ\text{C}$ to constant mass.

6.3.2 Apparatus

The following apparatus shall be used for the extraction of creosote or mineral oil, or both (see Figure 6.3.2):

- (a) *Extraction flask, Soxhlet extractor* The apparatus shall be of glass with standard ground-glass joints open to the atmosphere at the top.
- (b) *Water trap, condenser* The apparatus shall be a Dean and Stark side-arm attachment made of glass with standard ground-glass joints, fitted with a water-cooled condenser at the top and open to the atmosphere. The size of the apparatus shall be appropriate to collect within the graduation range of the water trap. The trap shall be chemically clean so that the shape of the meniscus at the end of the extraction is the same as at the beginning. Coating the trap from time to time with silicone resin gives a uniform meniscus.

NOTE: A thin rod of some material to which water does not adhere, such as polytetrafluoroethylene, may be used to transfer water condensed elsewhere to the water layer of the trap.

- (c) *Heating apparatus* An electric heating mantle shall be used.

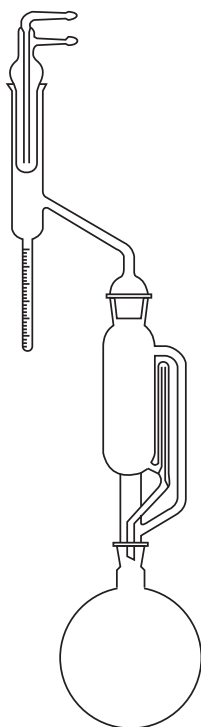


FIGURE 6.3.2 EXTRACTION FLASK, SOXHLET EXTRACTOR, WATER TRAP AND COLDFINGER (OR CONDENSER) FOR EXTRACTION OF CREOSOTE AND/OR MINERAL OIL FROM TREATED TIMBER

6.3.3 Extraction of timber

The procedure for the extraction of timber shall be as follows:

- (a) Take a test piece of 5 g to 20 g treated timber. If the test piece to be analysed has been broken into fragments, use the larger of these fragments to make a composite sample of aggregate mass in excess of 10 g. Weigh the test piece in air to an accuracy of 0.02 g.
- (b) Place in the extraction flask sufficient toluene to fill the water trap and Soxhlet extractor and to maintain a reservoir in the extraction flask. Add approximately 0.5 mL distilled water to the toluene. Assemble the apparatus, apply heat and reflux for 30 min. Allow the contents of the water trap to cool to room temperature then, using the polytetrafluoroethylene rod, transfer any water adhering to the walls of the condenser and water trap to the water layer in the trap. Note the volume of water in the trap to the nearest 0.01 mL.
- (c) Transfer the test piece to the Soxhlet extractor in the assembled apparatus.
- (d) Apply sufficient heat to the extraction flask to reflux the toluene at the base of the condenser and to operate the Soxhlet siphon at least once every 12 min. Continue the refluxing for 16 h or until no more colour is extracted from the sample, whichever is greater. Allow the contents of the trap to cool to room temperature before transferring any water adhering to the walls of the condenser or water trap, or both, to the water layer in the trap. Read and record the volume of water in the trap to the nearest 0.01 mL.

The difference between this reading and the first reading represents the mass of water in the test piece.

6.4 CALCULATIONS

The moisture content, mass and percentage of preservative in a test piece shall be calculated from the following equations:

$$(a) \quad \text{Moisture content of test piece} = \frac{m_3}{m_2} \times 100 \quad \dots 6.4(1)$$

$$(b) \quad \text{Mass of preservative (creosote or mineral oil, or both)} = m_4 = m_1 - m_2 - m_3 \quad \dots 6.4(2)$$

$$(c) \quad \text{Percent creosote (or mineral, or both) oil} = \frac{m_4}{m_2} \times 100 \quad \dots 6.4(3)$$

where

m_1 = mass of test piece before extraction, in grams

m_2 = oven-dried mass of extracted test piece, in grams

m_3 = mL of water collected from test piece, in grams

m_4 = mass of toluene-soluble extract, in grams

6.5 CORRECTION

Correction for toluene soluble extract should be applied when the toluene soluble extract from untreated timber exceeds 5% of its oven-dried mass. The extract content of untreated timber may be determined by Soxhlet extraction in the same manner as preservative-treated timber. The calculation for this shall be as follows:

$$(a) \quad m_4 = m_1 - m_2 - m_3 \quad \dots 6.5(1)$$

$$(b) \quad \text{Percent extract} = \frac{m_4}{m_2} \times 100 \quad \dots 6.5(2)$$

where

m_1 = mass of test piece before extraction, in grams

m_2 = oven-dried mass of extracted test piece, in grams

m_3 = mL of water collected from test piece, in grams

m_4 = mass of toluene-soluble extract, in grams

SECTION 7 DETERMINATION OF ORGANO TIN OXIDE (TBTO) IN PRESERVATIVE - TREATED TIMBER

7.1 PRINCIPLE

Tri-(*n*-butyl) tin oxide (TBTO) is extracted quantitatively from the timber sample with a mixture of hydrochloric acid and ethanol. The resultant solution is determined by atomic absorption spectrophotometry.

7.2 REAGENTS

All reagents shall be of analytical reagent quality.

The following reagents shall be used:

- (a) *2% hydrochloric acid in ethanol* Add 20 mL of 32% hydrochloric acid to approximately 600 mL ethanol. Mix well and make up to 1000 mL with ethanol.
- (b) *TBTO standard solution* Weigh exactly 0.2511 g TBTO into a 1000 mL volumetric flask. Make up to the mark with 2% hydrochloric acid in ethanol. This is a 1000 ppm standard solution. Working standards should contain 0, 10, 20, 50 and 100 ppm TBTO made by pipetting 0, 2, 4, 10 and 20 mL of 1000 ppm standard solution to 200 mL volumetrics, making up to the mark with 2% hydrochloric acid in ethanol. Mix well.

7.3 PROCEDURE

7.3.1 Instrument settings and operation

An atomic absorption spectrophotometer shall be used in the determination of tin.

The instrument settings and operating conditions for the atomic absorption spectrophotometer used in the determination of tin shall be as recommended in the instrument user's manual.

Tin shall be determined in a fuel-lean reducing red cone nitrous oxide/acetylene flame at 224.6 nm with deuterium background corrector on.

7.3.2 Sample preparation

The procedure for the preparation of the samples shall be as follows:

- (a) Comminute the timber sample so that it will pass through a 0.5 cm mesh.
- (b) Weigh a portion (≈ 1 g) out on a 50 mL beaker (m_1).
- (c) Put the beaker in a well-ventilated 105°C oven for 24 h. Reweigh when cool (m_2).
- (d) Accurately weigh another portion of timber sample (≈ 1 g) into a 100 mL round-bottomed quick-fit flask.
- (e) Add 12 mL of 2% hydrochloric acid in ethanol solvent and connect the flask to a water-cooled condenser. Place in an electrically operated heating mantle.
- (f) Allow to reflux for 10 min. Cool and remove the solvent to a 25 mL volumetric flask.
- (g) Add an additional 12 mL of solvent and reflux for another 10 min.
- (h) Cool and transfer solvent to the 25 mL volumetric flask.
- (i) Make up to the mark with solvent.

Submit sample to analysis by atomic absorption spectrophotometry.

7.4 CALCULATIONS

Calculations shall be as follows:

- (a) Moisture content (MC) shall be calculated from the following equation:

$$MC = \frac{m_1 - m_2}{m_2} \times 100 \quad \dots 7.4(1)$$

where

m_1 = mass of initial portion of timber [Clause 7.3.2(b)], in grams

m_2 = mass of oven-dried timber [Clause 7.3.2(c)], in grams

- (b) Percent tin in oven-dried timber shall be calculated from the following equation:

$$\text{Percent tin} = \frac{\text{ppm tin} \times 25 \times (MC + 100)}{10^6 \times m} \quad \dots 7.4(2)$$

where

m = mass of timber, in grams