## **CCA TIMBER TREATMENT**

## MANUAL FOR TIMBER TREATERS AND TESTING

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## Introduction

An important aspect of timber preservation is the means of checking to ensure that timber has been adequately treated to the required retention. This means that the operator must ensure that firstly, the chemicals have been mixed to the correct strength, and secondly, that the preservative has been impregnated appropriately into the wood.

TPC 'Woodmark' licensees are required to sample both solutions and timber as per a regime set out in the TPC Quality Manual.

## 8.1 Solution Strength Testing

## 8.1.1 CCA Preservatives

Method 1 - Specific Gravity (Hydrometer) Test

Specific gravity hydrometers are required to cover with readable accuracy the full range of solution concentrations encountered within a plant's hazard class treatment approvals. Preservative concentrates may be diluted with measured amounts of distilled water to bring them within a measurable hydrometer range.

A thermometer (0°C to 50°C) shall be available to determine the temperature of the solution.

**NOTE:** The specific gravity of the solution is affected by temperature. As the temperature rises, specific gravity falls. Specific gravity determinations by hydrometer must therefore be related to temperature before solution concentration can be determined. It is desirable if practicable to take SG readings on solutions at 15°C which is the temperature that most hydrometers are calibrated. This may require warming or cooling the solution as required.

The following points should be observed when taking hydrometer readings:

- 1. Use only a clean, dry hydrometer jar and hydrometer
- 2. Fill the jar with test solution, slowly do not aerate solution. Note that some sample taps on work tanks are inclined to aerate solution. Place on a level, secure surface
- 3. Watch for sludge!! may need to filter the solution first.
- 4. Immerse hydrometer carefully to determine approximate floating level. Wipe off any adherent air bubbles
- 5. Insert hydrometer to approximate floating level and leave for 2-3 minutes while it achieves temperature equality with solution.
- 6. Gently spin the stem and read where the solution level at the bottom of the meniscus passes through the scale.
- 7. Take temperature of solution in jar.

8. Calculate the solution strength applicable to the sample by crossreferencing the SG and temperature on the appropriate chart.

Method 2 - CCA by Titration

#### Purpose

Titration is one method used by timber treaters to determine CCA concentrations. This method is used mostly by treaters using the Alternating Pressure Method because the solution becomes contaminated by soluble and insoluble material from the lumber. This does not affect the efficacy of the preservative, but makes solution strengths by hydrometer inaccurate.

#### Scope

This titration is used in determining the Hexavalant chromium concentration in treating solutions.

#### References

This titration is based on:

- 10.100 'Standardisation of Potassium Dichromate Solution Against Iron', pp 376-377 "A Textbook of Quantitative Inorganic Analysis", Vogel 5th Edition.
- 10.101 'Determination of Chromium in a Chromium (III) Salt', pp 377-378, "A Textbook of Quantitative Inorganic Analysis", Vogel 5th Edition.

#### Apparatus

- 1 x 50ml Burette with Bottle
- 2 x 250ml Conical Flasks
- 2 x 10ml pipettes (marked differently to tell apart)
- 1 x plastic graduated Pasteur Pipette
- 2 x Rubber Pipette Fillers

#### Reagents

Potassium Dichromate Solution Ferrous Ammonium Sulphate Solution Sodium Diphenylamine Sulphonate Indicator

## Procedure

## Preliminary

- 1. Take one conical flask and add approximately 100mls of tap water.
- 2. Add 2ml of indicator solution to the flask.
- 3. Pipette 10mls of Ferrous Ammonium Sulphate solution into the flask.
- 4. Pipette 10mls of treating solution using ANOTHER<sup>\*</sup> pipette into the flask.
- 5. Titrate with 0.167 M Potassium Dichromate. The pale green colour will slowly darken. As it does add the Dichromate drop by drop until the addition of the final drop will turn the solution a clear purple colour.

This is the endpoint. This is **A** mls.

**Note:** If the solution has already turned purple then another 10 mls of Ferrous Ammonium Sulphate must be added to the preliminary and standardisation titrations.

## Standardisation

- 1. Take one conical flask and add approximately 100mls of tap water.
- 2. Add one indicator tablet or 2ml of indicator solution to the flask.
- 3. Pipette 10mls of Ferrous Ammonium sulphate solution into the flask.
- 4. Titrate with 0.167 M Potassium Dichromate. The pale green colour will slowly darken. As it does add the Dichromate drop by drop until the addition of the final drop will turn the solution a clear purple colour.

This is the endpoint. This is **B** mls.

<sup>&</sup>lt;sup>\*</sup> Separate pipettes must be used and even though washed after use, must not be interchanged. Mark the pipettes and keep

them separate.

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## Calculations

(B-A) x F = % CCA in solution

F (Factor) for Impretect C Salt is 0.110 F (Factor) for Impretect C Oxide is 0.063

Example

A = 22.3 mls B = 38.9 mls

Solution Strength (Impretect C Salt)

- = (38.9 22.3) x 0.110%
- = 16.6 x 0.110%
- = 1.83%

## Records

Results must be written on to charge sheets.

## Method 3 - Refractometer

Check a sample of water which has been left to stand for a period in the room (the water temperature should be the same as the room temperature) regardless of the measuring temperature. Set the boundary line to zero by using the adjusting screw for calibration. Then measure the sample, and note the scale reading which can be taken as the true value.

Variations in room temperature may cause an error, so it is recommended that the zero-setting be checked at intervals of 20 minutes using the test water mentioned above.

#### **Precautions:**

- 1. Hold the refractometer between the thumb and finger tips of the left hand and use the right hand for adjusting the eyepiece and manipulating the sample. Do not hold the refractometer by enveloping the entire tube with the palm of the hand.
- 2. When the sample is severely turbid or coloured, the field of vision darkens and the boundary line may become unclear or completely disappear. In such cases, try reading in direct sunlight or bright light.
- 3. Since the refractometer is an optical instrument, do not drop it or handle it roughly.
- 4. Since the prism has a relatively soft surface, be careful not to scratch it.

- 5. After use, clean the prism surface and daylight plate with a soft cloth soaked in water and wipe off the moisture with a dry cloth.
- 6. If the prism surface is smeared with oil or similar liquids it will repel the sample and obstruct the measurement. Wipe off the oil smear or contaminant with warm water.

## NEVER SPLASH WATER ON THE UNIT

Confirm with your Technical Advisor the procedure for determining concentration:

- > direct
- using conversion chart.

## 8.1.2 Boron Preservatives

Method 1 - F-Bor by Titration

## Purpose

This titration is used for analysis of F-Bor liquid deliveries.

This titration is used by timber treaters to analyse their boron baths and is normally used when their solutions are cloudy or there has been contamination. This method is not useful for treaters who use a dye in their bath as it is difficult to observe the end point colour change.

## Scope

This titration is used in determining the Boric Acid equivalent in boron treating solutions.

## References

10.34 'Determination of Boric Acid' Vogel 5th Edition, pages 299-300.

## Apparatus

- 1 x 500 Volumetric Flask
- 1 x 25ml pipette
- 1 x 250ml conical flask
- 1 x dropper bottle
- 1 x spatula
- 1 x 25ml burette

## Reagents

0.2 N Hydrochloric Acid

0.2 N Sodium Hydroxide Mannitol Boron Indicator

## Procedure

- 1. Pipette 25ml of sample into a 500ml volumetric flask, make up to the mark with water and mix well.
- 2. Pipette 25ml of the solution (make sure pipette has been rinsed out thoroughly in water) into a 250ml conical flask.
- 3. Add 100mls of water and 5 drops of indicator solution.
- 4. Add slowly, drop by drop, 0.2 N HCL from the dropper bottle until the colour of the solution goes a red-orange.
- 5. Add 1 teaspoon of Mannitol and swirl the flask, dissolving the Mannitol.
- 6. Titrate with 0.2 N Sodium Hydroxide. The end point occurs when the colour changes to a red-purple.

**Note:** If there are crystals in the sample received, these must be dissolved by warming and stirring before analysis proceeds.

#### Calculations

% Boric equivalent = ml 0.2 N Sodium Hydroxide

#### Records

Results should be written on to the relevant reconciliation statement.

F-Bor By Specific Gravity

## Purpose

This method is used in determining the Boric Acid equivalent in F-Bor treating solution.

## Scope

This method is used by timber treaters to analyse their F-Bor baths.

## Apparatus

1 x Thermometer - checked or calibrated

- 1 x 250ml measuring cylinder
- 1 x Hydrometer s.g. of 1.000 1.200 checked or calibrated
- 1 x F-Bor concentration chart Refer Appendix F or G

## Procedure

- Please Note: the following points are important to maintain accuracy.
- 1. All glassware must be kept in spotless condition. Surface deposits on hydrometers or unclean measuring cylinders lead to inaccuracy.
- 2. Fill the measuring cylinder slowly do not aerate the solution. Ensure that the measuring cylinder is level. Fill to approximately 220ml.
- 3. Insert the hydrometer carefully and leave for 2-3 minutes while it achieves temperature equality with the solution.
- 4. Gently spin the hydrometer. Ensure there are no air bubbles sticking to the surface of the hydrometer as this will result in incorrect readings.

Hydrometer readings should be taken at the bottom of the meniscus at eye level. (The meniscus is the concave skin formed on the surface of this liquid through the forces of tension).

5. Take temperature of solution.

## Calculations

Read solution strength off charts.

#### Records

Results should be written on to charge sheet.

## 8.1.3 MoistureGuard Solution Strength Determination

There is no current test available which can be applied at plant level. However, an accurate test can be provided by the Osmose Laboratory in Auckland, as required.

## 8.1.4 ACQ

Refer to your Osmose Technical Representative.

## 9.0 TESTING OF TREATED TIMBER

## 9.1 Introduction

As an ongoing part of a treatment plants quality assurance plan, regular testing of treated timber is required. While a charge sheet will provide details as to a overall net retention of a charge, only regular sampling will confirm that the required penetration has been achieved on a piece by piece basis.

## 9.2 Sampling Frequency

The Timber Preservation Council requires all licensees to sample their treated throughput to clearly define schedules, the frequency of which depends on the relevant hazard class.

These schedules are included in this manual in this Section.

## 9.3 Penetration and Retention Sampling Requirements

Samples should be taken in clear, straight-grained wood away from knots, splits, checks or other defects and at a minimum distance from the end or edge of pieces as indicated in the table below. Samples shall be representative of the charge, but where mixed charges occur, sampling shall be directed at the produce considered to be the most difficult to treat.

Hazard Class	Preservative Type	Minimum distance from piece end (mm)
H1 Group A	Boron compounds CCA	300 450
H1 Group B	Boron compounds CCA	150 from one edge
H3 Group A	CCA, LOSP	450
H3 Group B	CCA, LOSP	150 from one edge
H4 Group A	CCA	600
H4 Group B	CCA	150 from one edge
H5 Groups A,	CCA	600: sawn timber
B and C		Poles: ground-line
H5 Group D	CCA	150 from one edge
H6	CCA	Mid-point

## 9.4 Penetration and Retention Tolerance

For most Hazard Classes the penetration and retention requirements must be met in a minimum of 90% of the samples in any set. Exceptions are in the heartwood penetration requirements in Hazard Class H4 where the requirement must be met in a minimum of 60% of the samples in any set.

## 9.5 Substandard Treatments

Timber charges represented by samples not meeting the requirements of this specification shall be classed as substandard and shall either be redried and retreated, rebranded in a lower Hazard Class whose requirements it meets, or its brands removed and the timber sold as untreated.

## 9.6 Penetration Tests

Sampling requirements for each Hazard Class are given later in this Section. Penetration requirements for each Hazard Class are given in the TPC Specification Manual MP3640.

Penetration spot-tests are to be performed on-site using a method as detailed in this section. All plants are required to hold functional stop-test equipment and viable chemicals appropriate to the preservative used.

## 9.7 Retention Tests

Sampling requirements for each Hazard Class are given later in this Section. Retention tests are to be conducted by an approved laboratory.

## 9.8 Timber Samples

It is optional whether a plant takes sawn samples or increment borings, except that sawn samples shall be taken from H1 timbers which require a minimum core loading.

All charges containing sawn timber treated under Hazard Classes 3, 4, 5 and 6 which require heartwood penetration, shall have samples taken from exposed heartwood surfaces.

Sawn Samples shall permit examination of the full cross-section. Only a 5mm biscuit from each sample need be held after the required end off-cut has been discarded.

- Increment Borings shall be taken at right angles to the annual rings directed toward the pith of round produce or towards the centre of sawn timbers.
  - All holes created as a result of sampling shall be tightly plugged with dowels treated with a suitable preservative at a retention loading equivalent to or greater than the produce sampled.

## 9.9 Minimum Sampling

All charges shall have a minimum of ten samples taken unless the number of pieces in a charge is less than ten, in which case all pieces will be sampled. All details relating to penetration tests conducted shall be recorded on the relevant charge sheet. All samples shall be indelibly labelled with he charge number and shall be held for audit purposes for no less than three months.

# <u>Note</u>: Samples that are to be analysed for retention shall not be spot-tested prior to despatch to the laboratory as this can make selection of the correct analytical zone difficult.

## 9.9.1 Sampling Intensity

The standard sampling procedure is documented in MP3460 1992 and the following pages of this section. Additional requirements are:

- a) All charges undergoing re-treatment, having previously failed on retention shall be resampled after treatment for retention testing
- b) All charges showing an undercharge of 10% or greater shall be regarded as suspect and sampled for penetration but only after consultation with your Osmose representative
- c) Low absorptions can be indicative of poor treatment. Charges of timber which achieve absorptions lower than expected seasonal uptakes or absorptions unaccountably lower than those of previous charges of similar material shall be tested for penetration and retention
- d) If a "new" treatment process is introduced, the plant shall return to the "Initial Sampling" point of the sampling schedule. Refer to your Osmose Technical Representative for advice and assistance.

## 9.10 Chemical Tests for Presence of Preservative

## 9.10.1 Boron

9.10.1.1 Pyrocatechol Violet Test "PCV Test" - Reagents

Reagents

- (1) 0.2% solution of pyrocatechol violet in water
- (2) Buffer solution pH 9-8.2

#### Pyrocatechol Violet "PCV" Solution

Make a solution of 2g per litre of the dyestuff in distilled water. Add a few mls of chloroform to stop mould growth. Store in a brown glass bottle out of direct sunlight and out of contact with any metals : under these conditions storage life is virtually indefinite.

#### Phosphate Buffer for "PCV" Solution

0.68g of anhydrous potassium dihydrogen phosphate A.R. previously dried at 110°C and 13.49g of disodium hydrogen phosphate A.R. dried at 130°C and dissolved in 1 litre of distilled water.

Alternatively, make a 2% solution of sodium dihydrogen phosphate A.R. and using a pH meter, add 20% sodium hydroxide solution until the pH of the solution is between 8.0 and 8.2.

A brown glass bottle is preferable for the storage of this buffer solution as there is a tendency for algae to grow in it.

- Spray the freshly cut sample with solution (1), allow to soak in then spray with solution (2).
- Samples containing sufficient boric acid to comply with TPC requirements will show a distinct pinkish-red colour, borderline cases at about 0.2% boric acid will show a muddy brownish green colour and inadequately treated areas will appear yellow. Very heavily loaded areas may appear whitish. Care should be exercised in spraying the reagents, particularly on denser timbers, as the spray may run and give an incorrect interpretation of the result.

## 9.10.2 Copper (as present in CCA and ACQ treated wood)

9.10.2.1 Rubeanic Acid Test

Reagents:

- (1) 5% ammonia solution
- (2) 0.5% rubeanic acid (dithio oxamide) in alcohol

#### Ammonia Buffer for Rubeanic Acid Test

Dilute 1 part of 0.880SG ammonia with 6 parts of distilled water or 1 part of 0.91 solution with four parts of distilled water.

#### Rubeanic Acid (Dithio oxamide) Solution

5g of rubeanic acid dissolved in a litre of a mixture of 90 parts ethanol and 10 parts acetone. Iso-propyl alcohol may be substituted for ethanol if required.

- Spray specimen with (1) followed by (2). Coppercontaining areas show a greenish black to almost black colouration, untreated areas retain natural wood colour. The colouration is substantially permanent and samples may be kept for reference for a period of years if needed.
- 9.10.2.2 Chrome Azurol S Test

#### Reagent

(1) 0.5% Chrome azurol S dye and 5% sodium acetate in distilled water

#### Chrome Azurol S Solution

5g of Chrome azurol S dyestuff and 50g of sodium acetate are dissolved in 1 litre of water.

• Spray the specimen with the solution, or dip it into the solution. Copper gives a strong royal blue colour, untreated areas are orange. The colour persists for some weeks.

#### 9.10.3 LOSP actives

There is no field test currently available. Samples must be forwarded to a laboratory to perform this analysis.

## 9.11 Sampling Schedule for Testing Hazard Classes H1, H2, H3 and H4 Rounds and Part Rounds

## 9.11.1 Penetration

9.11.1.1 Diffusion

All charges are to be stop-tested for penetration using the PCV spot test. Refer section 9.10.1

9.11.1.2 Pressure

Every 10<sup>th</sup> charge is to be stop tested for penetration using a suitable spot test.

## <u>Note</u>: Boron-treated timber that is to be kiln-dried must be spottested for penetration <u>prior</u> to drying.

## 9.11.2 Retention

Sample as per the flowchart below. Send 10 samples per charge to an approved laboratory (for H1 these must be cross-sections)



retention after drying.

#### 9.11.3 Failure

If any sample set fails to comply with Hazard Class requirements for penetration or retention, initiate Corrective Action and if necessary obtain advice from your Osmose Technical Advisor.

## 9.12 Sampling Schedule for Testing Hazard Class H4 and Sawn Timber and H5 (excluding Sawn Piles)

## 9.12.1 Penetration

All charges are to be spot-tested for penetration using a suitable spot test.

## 9.12.2 Retention

Sample as per flowchart below. Send 10 samples per charge to an approved laboratory.



## 9.12.3 Failure

If any sample set fails to comply with Hazard Class requirements for penetration or retention, initiate Corrective Action and if necessary obtain advice from your Osmose Technical Advisor.

## 9.13 Sampling Schedule for Testing Hazard Class H5 Sawn Piles

Obtain approval from the Timber Preservation Council to produce sawn piles.

Obtain advice from your Osmose Technical Advisor regarding Code of Practice - Quality Control of Preservative Treatment for H5 Sawn Piles.

## 9.13.1 Penetration

All charges are to be spot tested for penetration using Rubeanic Acid or Chrome Azurol. *Refer Section 9.10.1.* 

## 9.13.2 Retention

Sample as per flow chart below. Send 10 samples per charge to an approved laboratory.



**Note:** Obtain advice from Technical Advisor re Code of Practice - Quality Control of Preservative Treatment for H5 Sawn Piles

## 9.13.3 Failure

If any sample set fails to comply with Hazard Class requirements for penetration or retention, initiate Corrective Action and if necessary obtain advice from the Technical Advisor.

## 9.14 Sampling Schedule for Testing Hazard Class H6

## 9.14.1 Penetration

All charges are to be stop-tested for penetration using a recommended spot test.

## 9.14.2 Retention

All charges are to be tested for retention. Send 10 samples per charge to an approved laboratory.

#### 9.14.3 Failure

If any sample set fails to comply with Hazard Class requirements for penetration or retention, initiate Corrective Action and if necessary obtain advice from the Technical Advisor.