

This draft Guidance Document was developed by the Task Force on Biocides that approved it at its 6th meeting held on 11-12 September 2008 and agreed that it be transmitted to the WNT for comment.

ACTION REQUIRED: *The WNT is invited to comment on the draft Guidance Document by 25 November 2008*

OECD Guidance

**on the Estimation of Emissions from Wood Preservative –
Treated Wood to the Environment: for Wood held in Storage
after Treatment and for Wooden Commodities that are not
covered and are not in Contact with Ground**

**Guidance and Criteria for Industry for the Preparation and Presentation of
Emission Rate Data from Wood Treated with Preservatives in Support of
Regulatory Decisions in OECD Countries**

FOREWORD

This document is intended to provide guidance to applicants wishing to have particular active substances approved or wood preservative products registered in OECD countries. It provides guidance with respect to the collection, preparation, quality, and reporting of emission data to be submitted to enable an environmental risk assessment of emissions of wood preservatives from treated wood to be performed. The recording forms for emission data and information that are included in the Appendix 1 of this guideline are intended to be illustrative of the approach to be taken in the preparation of the required test report. The appendix has not been critically examined for their technical content.

This guidance was developed with the aim of facilitating the compilation of emission data from preservative treated wood to submissions to OECD countries by providing a common methodology, quality criteria and reporting format.

Where on particular points of detail, additional or more detailed guidance is required, applicants and other interested parties are advised to contact the relevant authority of the country to which the documentation is to be submitted.

The OECD Taskforce on Biocides (OECD TFB) decided in March 2007 that the draft OECD guideline for the testing of treated wood that was not covered and not in contact with the ground, often referred to as Test Guideline 1 (TG1)¹, did not provide the flexibility of approach demanded by the OECD member countries in order to determine the required emission data. The OECD TFB agreed that a guidance document was to be developed from TG1 that encompassed the required flexibility. This document provides the requisite guidance and now replaces TG1.

This guidance document is based on and is consistent with the OECD Guidelines for testing chemicals and was prepared with the benefit of the comments provided by the delegations of countries participating in the OECD Taskforce on Biocides and industry representatives.

Note:

This document will be periodically revised, as some sections will be added or updated. Please consult the OECD Web site at <http://www.oecd.org> or contact the OECD Secretariat to make sure that you have the latest version.

Health and Safety Division
Environment Directorate
Organisation for Economic Co-operation and Development
2, rue André Pascal
75775 Paris Cedex 16
France

¹ 'OECD Guideline For The Testing of Chemicals - Estimation of Emissions from Preservative – Treated Wood to the Environment: Laboratory Method for Wood held in Storage after Treatment and for Wooden Commodities that are not covered and are not in Contact with Ground'

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1 GENERAL INTRODUCTION

- 1.1 The guidance provided and criteria specified in this document, apply to the collection, preparation and presentation of emission data from wood treated with preservatives submitted in support of applications for the approval, or renewal of approval, for active substances and the authorisation of wood preservative products.
- 1.2 This document is intended to provide guidance to industry to enable applicants to perform the most appropriate leaching test methodology in the laboratory according to the application method of the wood preservative, use class and use pattern of treated wood or wooden commodities in order to obtain as realistic an emissions rate as possible. This document also provides guidance to regulators and Competent Authorities to perform the risk assessment.
- 1.3 Use Classes (1-5) are used internationally and categorise the biological hazard to which the treated commodity will be subjected (1,2).
- 1.4 Use Classes also define the situation in which the treated commodity is used and determine the environmental compartments (air, water, soil) which are potentially at risk from emission of components from the preservative treated wood. Rain falling on treated wood could produce emissions that run off into surface water and/or soil.
- 1.5 The emissions from preservative treated wood to the environment need to be quantified to enable an environmental risk assessment of the treated wood. This guidance document describes a laboratory method for the estimation of emissions from preservative treated wood in two situations where emissions could enter the environment:
 - 1.5.1 Emissions from preservative-treated wood ‘in storage’, i.e., stored outside at the preservative treatment site. Weather conditions (e.g. rain falling on the treated wood) could produce emissions which run off into surface water and/or soil. This wood could be treated to a retention that would be appropriate to Use Classes 1 – 5.
 - 1.5.2 Emissions from preservative-treated wood ‘in service’, i.e., used in commodities where the wood or wood-based product is not covered and not in contact with the ground. It is either continuously exposed to the weather or is protected from the weather but subject to frequent wetting [i.e. as in Use Class 3 (1), e.g. exterior joinery, cladding and decking timbers].
- 1.6 Emissions from wood or wooden commodities in service that are not covered, permanently exposed to wetting and are in contact with the ground [i.e. as in Use Class 4 (1), e.g. fence posts] were initially considered by the Task Force on Biocides but no progress could be made.
- 1.7 For wood or wooden commodities that are not covered, permanently exposed to wetting and in contact with fresh water [i.e. as in Use Class 4 (1), e.g. freshwater jetties] or seawater [i.e. as in Use Class 5 (1), e.g. marine piles and jetties], please refer to the OECD Test Guideline n°313: ‘Estimation of Emissions from Preservative - Treated Wood to the Environment: Laboratory Method for Wooden Commodities that are not Covered and are in Contact with Fresh Water or Seawater’.
- 1.8 The objective is to achieve standardization to the extent that is practical and feasible, of the emission data from treated wood or wooden commodities that are not covered and are not in contact with soil (use class 3) with a view to:
 - 1.8.1 Ensure the quality, consistency and reproducibility of the emissions results;

- 1.8.2 Provide as realistic an emissions rate as possible under laboratory conditions given the application method of the wood preservative, use class and intended use pattern of treated wood or wooden commodities;
- 1.8.3 Assist the evaluation of emissions data for active substances and wood preservative products to enable a consistent approach across OECD countries.
- 1.9 The recommended methods provided in this Guidance can be utilized to generate water samples (emissate), which then will be analyzed for the chemical(s) of interest. The water samples are to be obtained from the water that is used to immerse treated wood or from water that is collected as a result of simulated artificial rainfall onto treated wood.
- A degree of flexibility is provided within this guidance document regarding the immersion duration of the test to enable applicants to select the most appropriate testing regime. The Applicant is strongly advised to consult with the Authority with which registration is being sought. This will allow for the Applicant to verify that the selected immersion regime is the most appropriate and up to date with the regulating Agency's current standards.
 - Provision is made in this guidance document to also allow the use of artificial rainfall onto treated wood, subject to the reproducibility, robustness, accuracy and reliability of the data.
 - In ALL cases the Applicant must justify the testing regime used according to the application method of the wood preservative, use class and intended use pattern of the treated wood or wooden commodities.
 - The quantities of emissions in the emissate are related to the surface area of the wood and the length of exposure, to estimate a flux in mg / m² / day. The flux (emission rate) after increasing periods of exposure can thus be estimated.
 - The quantity of emissions may be used in an environmental risk assessment, which also requires scenarios and other parameters. This risk assessment will be updated and refined as research findings and scientific methods are developed.
- 1.10 Applicants should be aware that different regulatory authorities of the OECD countries may have different requirements. Therefore, Applicants are advised to consult the regulatory authority in the country to which the data will be submitted prior to commencing testing. This will ensure there is agreement regarding the most appropriate testing regime (i.e., artificial rainfall vs. immersion, immersion durations and frequencies, etc.). The selection of the testing method will consider the application method of the wood preservative, the use class, and the use pattern of treated wood or wooden commodities.
- 1.11 In European countries, Use Class 3 treated wood or wooden commodities are typically used in a vertical position and are primarily used in construction, with less exposure to rain. Whereas in North America, preserved wood is used in both vertical and horizontal positions and in the absence of data, 'realistic worst case' scenarios are used to determine the risk to the environment
- 1.12 Applicants should also be aware that depending on the testing regime selected, some regulatory authorities may apply uncertainty factors to the endpoint determined for the treated wood. This is to reduce concerns regarding the extrapolation of the emissions results, statistical analyses, variability of the determined emissions rates (both laboratory and field) and the applicability of using laboratory tests to predict realistic worst-case emissions rates in the environment. This approach was agreed at the European Union (EU) Leaching Workshop in June 2005 and adopted at the EU Biocides

Technical Meeting (TM) in October 2005 (TMIII05) (3). Care should be taken when carrying out the extrapolation using uncertainty factors as this may result in a cumulative leaching being higher than the amount originally applied or the calculated retention. For superficial treatment processes, the probability that this will occur is higher than for penetration treatment processes. In this case, it was recommended in the EU Leaching Workshop to divide the total amount of wood preservative that is initially applied to the wood (based on the prescribed application rate or the calculated retention) by the (default) service life for obtaining a daily leaching value. If uncertainty factors are not applied, the applicant should explain the inapplicability of such assessment factors. This approach was also adopted at the EU Biocides TM in October 2005.

2 INITIAL CONSIDERATIONS

- 2.1 The principal agent for causing emissions from wood during open-air storage and use is rainfall. Wood exposed in above ground situations is subjected to intermittent wetting by rainfall and drying of the wood surface between the rainfall events. This wetting and drying cycle is simulated in the method outlined below. It is assumed that emissions obtained by exposure to rainfall can be represented by intermittent immersion in water followed by subsequent drying events.
- 2.2 The wood, in the case of wood treated with a wood preservative, should be representative of commercially used wood. It should be treated and dried in accordance with the preservative manufacturer's instructions and in compliance with appropriate standards and specifications. The parameters for the treatment and post treatment conditioning (drying) of the wood prior to the commencement of the test should be stated.
- 2.3 The wood samples used should be representative of the commodities used (e.g., with regard to species, density and other characteristics).
- 2.4 The test can be applied to wood treated using a penetrating process or superficial application (brush, spray or dipping), or to treated wood which has an additional surface treatment (e.g., paint that is applied as a requirement for commercial use).
- 2.5 In real exposure situations the composition, amount, pH and physical form (i.e., 0.5 to 3 mm raindrop diameter, large vs. mist-like) of rain affects the amount of emission. The spacing between rainfall/drying events, intensity of rainfall, and frequency of rainfall influence emission rate. With this level of complexity, emission in real exposure will be variable. Laboratory studies improve the reproducibility, accuracy, precision and reliability of emission values which are obtained. The immersion methods identified below are relatively simple to conduct and can produce reproducible results. Artificial rainfall methods are more complex, but they can simulate real rainfall.

3 PRINCIPLE OF THE RECOMMENDED METHODS

- 3.1 The schedule of the immersion days in the lab tests are not intended to simulate the frequency of days with rainfall. The aim of the intervals between the immersion days is to ensure that the specimens are subjected to a simulation of wet dry cycles which occur in service. The guidance document does not include methods of further extrapolation of the data which can be performed (e.g., on the basis of a fictitious schedule of rainy days or the duration of water contact).
- 3.2 Three main immersion regimes are recommended as follows:

- 3.2.1 The applicant must provide adequate discussion of the conditions of these regimes (e.g., purpose is to estimate emissions from wood that is treated and then exposed vertically or horizontally) for data to be acceptable when using these regimes.
- 3.2.2 The 3 x 1 minute immersion regime: A 1 minute immersion in water of the treated test specimens to simulate exposure to a rainfall event. There are three immersions or rainfall events per day, with test specimens allowed to dry for 3 hours between immersions simulating the wetting and drying cycle of natural exposure situations. The days of rainfall are set for 1, 3, 5, 8, 10, 12, 15, 17 and 19 days.
- 3.2.3 It should be noted, the OECD Emission Scenarios for Wood Preservatives that are applied for the authorization procedure of the European BPD are based on the assumptions, that the total amount of rainfall per year is 720 mm and that there is a rain event every third day.
- 3.2.4 The daily regime is intended to produce the same moisture content on the surface of the wood, and the same moisture content profile in the wood, and thus the same emission, as wood in a real exposure situation on a day during which rain occurs. It is intended that a 3 x 1 minute dip, three times per day, every third day, will give the same moisture content cycle in the wood, and the same emission from the wood, as exposure under real conditions. If the dipping and drying regime is continued for one year, the emission quantity and rate would be the same as the emission obtained from wood after exposure in a real situation in a region of Europe where there is 720 mm of rain per year, with rain events occurring on 260 days of the year. The test is likely to produce results which are below the limit of detection, which mimics results from studies carried out in the field.
- 3.2.5 The 2 x 1 hour immersion regime: A 1 hour immersion in water of the treated test specimens to simulate exposure to a rainfall event. There are two immersions or rainfall events per day, with test specimens allowed to dry for 4 hours between immersions simulating the wetting and drying cycle of vertical natural exposure situations. The days of rainfall are set for 1, 3, 5, 8, 10, 12, 15, 17 and 19 days.
- 3.2.6 The 1 x 2 hours immersion regime: A 2 hour immersion in water of the treated test specimens to simulate exposure to a rainfall event. There is only 1 immersion or rainfall event per day, with test specimens allowed to dry between immersions simulating the wetting and drying cycle of horizontal natural exposure situations. The days of rainfall are set for 1, 3, 5, 8, 10, 12, 15, 17 and 19 days.
- 3.3 Other immersion regimes, other than those outlined above, may also be used if the Applicant considers that they are more appropriate. It is strongly recommended that if a particular immersion regime is considered by an Applicant, that the appropriate regulatory Agency is consulted prior to initiating the study to determine whether or not it is acceptable. The appropriateness of the experimental methodology is ultimately determined by the regulatory Agency, not the Applicant.
- 3.4 Artificial rainfall regimes may be used in lieu of immersion regimes. The inclusion of this type of methodology for collecting leachates from treated wood allows Applicants the opportunity to generate and submit data that may be more representative of what will be occurring once wood specimens are treated. However, it is important to recognize, that despite the practicality of the data, there are several variables that need to be considered and addressed. This will ensure the generation of robust, accurate, reproducible and reliable data from the artificial rainfall testing. Initially, upon deciding to utilize such a method, the Applicant is recommended to consult with the Agency at which registration is sought to create a collaborative effort for the generation of optimum results.

- 3.5 The Applicant must always be able to scientifically justify the selected testing regime according to the application method of the preservative, the use class and intended use pattern of the treated wood. In addition, it is recommended that the Applicant consult the regulatory authority in the country to which the data will be submitted prior to commencing the test to ensure that there is agreement that the most appropriate method has been selected.
- 3.6 The water (emissate) collected for each replicate sample on each sampling day is analysed. Tests with untreated samples can be discontinued if there is no background detected in the first three data points. Emission rates in mg / m² / day are calculated from analytical results.
- 3.7 The inclusion of untreated wood specimens allows for the determination of background levels for emissates from wood other than the preservatives used.

4 QUALITY CRITERIA

4.1 Accuracy

- 4.1.1 The accuracy, precision and repeatability of the analytical method should be determined before conducting the test (e.g., ASTM D 1193 Type II).
- 4.1.2 In addition to this, it is recommended that there is a discussion of method validation. This will allow for the lab technician as well as the reviewer to feel confident in the accuracy and reliability of the results. The method validation will allow conclusions to be made regarding the analytical method in terms of being the most accurate and scientifically appropriate for a specific chemical and wood type.

4.2 Reproducibility

- 4.2.1 Three water samples are collected and analysed and the mean value is taken as the emission value. The reproducibility of the results within one laboratory and between different laboratories depend upon various factors which include but are not limited to; the testing regime used, the type of wood species used, and the treatment level of the wood.
- 4.2.2 Standard curves are necessary for supporting the reproducibility of the quantification method applied. This would ensure that the technician establishes the consistency of the analytical method as well as verifies the instrumental performance. In addition, such a curve may enable the technician to analyze, interpret and extrapolate data in a variety of ways. This standard curve is also commonly referred to as calibration of the instrument.
- 4.2.3 It is important to recognize this general requirement of a standard curve is not the same as the validation of the analytical method. The standard curve serves the purpose to ensure consistency of the instrumentation which in turn will confirm that the experimental results are reliable.
- 4.2.4 A calibration curve (also referred to as a working curve or analytical curve) can be generated using several standards that contain the exact known concentration of the chemical. Plotting the instrumental output via this method will allow for a linear response to be observed; and even an extrapolation of data once collection commences. Such an output will confirm that the instrumental performance is satisfactory and consistent.
- 4.2.5 A standard curve can be generated by adding increments of a standard solution to sample aliquots of the same size (also referred to as spiking). As a result, the instrumental response

will provide an output that is proportional to the concentration and support the reliability of the instrumentation.

4.3 *Acceptable Range of Results*

- 4.3.1 As the test involves the use of samples cut from commercially available treated wood, a range of emission rates can be expected.
- 4.3.2 Data on the quality of the analytical method does not consider variation between repeated experiments. Therefore variation between repeated experiments is higher than the uncertainty of the test procedure itself.

5 TEST CONDITIONS

5.1 *Water*

- 5.1.1 Deionised water is recommended (e.g., ASTM D 1193 Type II) for consistency and reproducibility of the results. It is recommended that the pH of the water at the beginning of either the immersion or artificial rainfall study be confirmed to be in the range of 5-7. Upon collection of the water samples, the pH values will also need to be documented. The water temperature shall be 20 °C +/- 2 °C, relative air humidity of 65% ± 5% and the measured pH and water temperature included in the test report. Analysis of samples of the water used taken before the immersion of the treated specimens or artificial rainfall onto treated wood (as appropriate) allows the estimation of the analysed substances in the water. The water that is used for purposes of immersion or artificial rainfall is suggested to be analyzed prior to initiation of the study. This analysis will serve as a control to determine, if any, background levels of the chemical or other interfering components are in the water.

5.2 *Environmental Conditions*

- 5.2.1 It is recommended that standard laboratory conditions are used including performing the test at a room temperature of 20 °C ± 2 °C, relative air humidity of 65 % ± 5 % and that the specimens are kept in the dark for the duration of the experiment to avoid any interferences resulting from photolysis (i.e., prevent exposure to ultraviolet light because ultraviolet light has been scientifically determined to potentially influence the amount of chemical that is leached off the wood). All test conditions should be recorded and included in the report.

5.3 *Wood Test Specimens*

- 5.3.1 The wood species should be typical of the wood species used for the efficacy testing of wood preservatives. The recommended species are *Pinus sylvestris L.* (Scots pine), *Pinus resinosa Ait.* (red pine), or *Pinus spp* (Southern pine). A pine species is required to be utilized, with the option to utilize additional species as necessary; preferably one that is likely to be treated with the chemical under consideration. Some wood species are less permeable and may not take-up water or leach preservatives as readily as pine sapwood. Additional tests may be made using other species.

- 5.3.2 Straight grained wood without knots should be used. Material of a resinous appearance should be avoided. The wood should be typical of wood which is available commercially. Emissions from commercially treated timbers may differ from those using laboratory treated samples because of a number of factors and some treatment processes cannot be replicated in the laboratory. The dimensions of the samples, the origin, density and number of annual rings per 10 mm should be recorded.
- 5.3.3 Wood test specimens are recommended to be sets of five EN 113 size blocks (25 mm x 50 mm x 15 mm dimensions) with the longitudinal faces parallel to the grain of the wood. Other dimensions such as 50 mm x 150 mm x 10 mm may be used if the Applicant considers that they are more appropriate to the use class and intended use pattern of the treated wood or wooden commodities. For artificial rainfall, it is recommended that boards measuring 5cm (nominal thickness) x 30cm (nominal width) with an arbitrary length (with sealed ends) are used. However, the Applicant should always justify the selected dimensions of the test specimens. Test specimens shall consist of 100 % sapwood. Each specimen is uniquely marked so that it can be identified throughout the test.
- 5.3.4 All test specimens should be planed or plane sawn and the surfaces should not be sanded.
- 5.3.5 The number of sets of wood test specimens used for analysis is at least five: three sets of specimens are treated with preservative, one set of specimens is untreated and one set of specimens is used for the estimation of the oven dry moisture content of the test specimens before treatment. Sufficient test specimens are prepared to allow selection of three (sets of five specimens if five EN 113 size blocks are used in each test assembly) which are within 5 % of the mean value of the preservative retentions of the pool of test specimens. This sample size is the minimum required, whilst the typical number of samples to be collected is in triplicate. As always, the greater sample size will enhance the quality of the data reported.
- 5.3.6 All test specimens are end-sealed with a substance which prevents penetration of preservative into the specimens during superficial treatments and out of the end-grain during the leaching tests. It is necessary to distinguish between specimens used for superficial application and penetration processes for the application of the end-sealant. The application of the end-sealant has to be applied prior to treatment only in case of superficial application. The end-grain has to be open for treatments by penetration processes. Therefore, the specimens have to be end-sealed at the end of the conditioning period. The emission has to be estimated for the longitudinal surface area only. Sealants should be inspected and reapplied if necessary prior to initiating leaching and should not be reapplied after leaching has been initiated.

5.4 Specimen Test Assemblies

- 5.4.1 The container is made of an inert material and is large enough to allow the full immersion of the test specimens used and result in the desired wood surface area (i.e., treated wood that is exposed to water) to water volume ratio of $0.4 \text{ cm}^2/\text{cm}^3$. If the dimensions are changed and the change is justified, it should be ensured to retain the surface to water volume ratio ($0.4 \text{ cm}^2/\text{cm}^3$). Only the area of treated wood that is exposed to the water has to be used for the calculation.
- 5.4.2 The container size will need to be of a sufficient size so that all of the surfaces of the wood which are to be exposed to water have complete and maximized contact with the water during immersion.

- 5.4.3 The test specimens in artificial rainfall regimes will be supported on an assembly with the appropriate orientation relative to the position that the wood will be expected to be when it is stored at a facility or placed in use (e.g., horizontal). As for the container size, it will need to be fairly close to the dimensions of the wood under consideration. This will help to capture water coming into contact with the wood with the least amount of excess rain water. Excess rainwater can potentially result in dilution of the emissate and skew the experimental results.

6 PROCEDURE FOR PRESERVATIVE TREATMENT

6.1 *Preparation of the Treated Test Specimens*

- 6.1.1 The preservative is applied to the test specimens by the method specified for the preservative, which may be by a penetrating treatment process or a superficial application process (e.g., dip, spray or brush).

6.2 *Preservatives to be applied by penetrating treatment process*

- 6.2.1 A solution of the preservative should be prepared that will achieve the specified uptake or retention when applied using the penetrating treatment process. The wood test specimen is weighed and its dimensions are measured. The penetrating treatment process should be as specified for the application of the preservative to wood that is not covered nor in contact with the ground. The specimen is again weighed after treatment and the retention of the preservative (kg/m³) is calculated from the equation:

$$\frac{\text{Mass after treatment}(kg) - \text{Mass before treatment}(kg)}{\text{Test specimen volume}(m^3)} \times \frac{\text{Solution Concentration} (\% \text{ mass / mass})}{100}$$

- 6.2.2 Note that timber treated in an industrial treatment plant (e.g. by vacuum pressure impregnation) may be used in this test. The procedures used should be recorded and the retention of material treated in this way must be analyzed and recorded.

- 6.2.3 All raw data should be recorded such as the initial and final weights, and then submitted to the appropriate Agencies.

6.3 *Preservatives to be applied by superficial application processes*

- 6.3.1 The superficial application process includes dipping, spraying or brushing of the wood test specimens. The process and application rate (e.g. litres/m²) should be as specified for the superficial application of the preservative.

- 6.3.2 In addition, timber treated in an industrial treatment plant may be used in this test. The procedures used should be recorded and the retention of material treated in this way must be analyzed and recorded. However, where this differs from the application method specified for the wood preservative (e.g., treatment processes such as oil treatment, steam fixation, flow coating, drying and kiln drying) (see points 6.1.1 and 6.3.1 above), the Applicant must be able to scientifically justify their selection and should consult the regulatory authority in the country to which the data will be submitted prior to conducting the test.

6.4 *Conditioning (drying) of the Test Specimens After Treatment*

6.4.1 After treatment, the treated test specimens should be conditioned (dried) in accordance with the recommendations made by the supplier of the test preservative and in accordance with any preservative label requirements and/or in accordance with commercial treatment practices and/or in accordance with EN 252 Standard.

6.4.2 Also the conditioning (drying) process and environment should be monitored and recorded in the raw data.

6.5 *Preparation and Selection of Test Specimens*

6.5.1 After post treatment conditioning (drying), the mean retention of the group of test specimens is calculated and three sets of five representative test specimens with a retention within +/- 5% of the mean of the group are randomly selected for leaching measurements.

7 PROCEDURE FOR PRESERVATIVE EMISSION MEASUREMENTS

7.1 *3 x 1 minute Immersion Regime*

7.1.1 The test specimens are weighed and the mass, date and time recorded. The surfaces of the test specimens are immersed in water for 1 minute, removed from the water and allowed to drain for 10 seconds, allowing run-off to return to the water. The test specimens are weighed again and then allowed to dry between immersions at room temperature in the laboratory.

7.1.2 There are three immersion events of 1 minute duration each on an 'immersion day' with a 3 hour conditioning (drying) period between immersions (e.g. at 10.00, 13.00 and 16.00). After an immersion day, the test specimens are allowed to dry for the prescribed period between wetting and drying of immersion events. The three, 1 minute immersion events occur at the following day intervals from initiation of the test: 1, 3, 5, 8, 10, 12, 15, 17 and 19 days. The immersion regime and sampling regime are recorded.

7.1.3 It is recommended that emittance samples taken on each immersion day to be pooled and retained for subsequent analysis to give a profile of the quantity of emissions against time. The pooled water samples may need to be concentrated by an appropriate technique before analysis. Samples should be stored under conditions that preserve the analyte (i.e., refrigerate in the dark to reduce microbial growth in the sample before analysis).

7.1.4 If samples are not analysed immediately on the day of extraction, the analyte should be frozen to reduce the potential for residual degradation. Frozen samples would require a storage stability analysis to ensure the results are scientifically valid and that there was no degradation or alteration of the chemical during the storage time.

7.1.5 It is important to change the water between immersion events. This is something that may potentially impact the leaching amounts of the chemical if there is not fresh water for each sampling event.

7.2 2 x 1 hour Immersion Regime

- 7.2.1 The test specimens are weighed and the mass, date and time recorded. The surfaces of the test specimens are completely immersed in the water for 60 minutes, removed from the water and allowed to drain for 10 seconds, allowing run-off to return to the water. The test specimens are weighed again and then allowed to dry between immersions at room temperature in the laboratory.
- 7.2.2 There are two immersion events on an ‘immersion day’ with a 4 hour conditioning (drying) period between immersions (e.g. at 08:00 and 13.00). After an immersion day, the test specimens are allowed to dry for the prescribed period between wetting and drying of immersion events. Two, one hour immersion events occur at the following day intervals from initiation of the test: 1, 3, 5, 8, 10, 12, 15, 17 and 19. The immersion regime and sampling regime are recorded.
- 7.2.3 The method allows the individual emissate samples taken on one day to be analysed or tested to give a profile of the quantity of emissions against time. If samples are not analysed immediately on the day of extraction, the analyte should be frozen to reduce the potential for residual degradation. Frozen samples would require a storage stability analysis to ensure the results are scientifically valid and that there was no degradation or alteration of the chemical during the storage time.
- 7.2.4 It is important to change the water between immersion events. This is something that may potentially impact the leaching amounts of the chemical if there is not fresh water for each sampling event.

7.3 1 x 2 hours Immersion Regime

- 7.3.1 The test specimens are weighed and the mass, date and time recorded. The surfaces of the test specimens are completely immersed in the water for 120 minutes, removed from the water and allowed to drain for 10 seconds, allowing run-off to return to the water. The test specimens are weighed again and then allowed to dry between immersions at room temperature in the laboratory.
- 7.3.2 There is one immersion event on an ‘immersion day’. After an immersion day, the test specimens are allowed to dry for the prescribed period between wetting and drying of immersion events. One, two hours immersion event occurs at the following day intervals from initiation of the test: 1, 3, 5, 8, 10, 12, 15, 17 and 19 days. The immersion regime and sampling regime are recorded.
- 7.3.3 The method allows the individual emissate samples taken on one day to be analysed or tested to give a profile of the quantity of emissions against time. If samples are not analysed immediately on the day of extraction, the analyte should be frozen to reduce the potential for residual degradation. Frozen samples would require a storage stability analysis to ensure the results are scientifically valid and that there was no degradation or alteration of the chemical during the storage time.
- 7.3.4 It is important to change the water between immersion events with fresh water. This is something that may potentially impact the leaching amounts of the chemical if there is not fresh water for each sampling event.

7.4 Artificial Rainfall

- 7.4.1 For the development of the artificial rainfall methodology, the following factors were considered. The artificial rainfall studies are required to be conducted entirely in a laboratory setting which will remove the potential for environmental variations (e.g. UV radiation impacting the leaching of various chemicals (Lebow 2003)). However, if the Applicant wishes to extrapolate and invest in the time and analysis of the impact of external factors, this is open for consideration when discussed with the regulatory Agency. In the lab, the treated specimens will be exposed to cyclic rainfall events. The purpose of this is to allow for the natural wetting and drying of wood to occur. Rainfall regimes are recommended to be over the course of one or two months, depending on resources available to the Applicants.
- 7.4.2 There are a variety of climates in which the wood types considered by this Guidance document will be found. The conditions in the Pacific Northwest tend to include misty rain and clouds which result in the wood being fairly saturated with seldom drying events. Such conditions allow the preservative to diffuse to the surface before the next rain event. This region can also experience occasional dry spells in which the wood cracks and as a result a fresh supply of preservative can move to the surface and be readily available for leaching during the following rainfall event. This is an example of the type of environment that is favourable for maximizing the leaching potential of preservatives.
- 7.4.3 The uptake of water as well as the length of drying time needed for checking is a function of the moisture content of the boards. The moisture content is a quality that can be measured by weighing the boards before, during and after rainfall. Sufficient drying and wetting will be desired, so a moisture content range is provided in the protocol which will allow for saturation as well as any potential checking (cracking) of the wood specimens. In addition, this cyclic process between wetting and drying is recommended to occur over the duration of one to two months. In obtaining leaching data on a monthly basis, the data will be able to be extrapolated to represent yearly amounts.
- 7.4.4 The rate of rainfall is also important to consider. This can possibly affect the length of time it takes to reach a desired moisture content (e.g. hard falling rain will not be as readily absorbed by the wood). One of the goals of this Guidance is to create as many uniform wetting and drying intervals as possible throughout the course of one to two months. Based on the technologies available and scientific experience (Lebow 2003), air atomizing nozzles are the most practical for producing artificial rainfall. This equipment produces a fine mist quite similar to the Pacific Northwest. Additional technologies that may be available to produce slow rainfall rates (i.e. 1-10 mm/hr) are open for consideration for this Guidance document.

7.5 Artificial Rainfall Regime

- 7.5.1 The test specimens are recommended to be 5 cm (nominal thickness) x 30 cm (nominal width) with an arbitrary length. In addition, the ends will need to be sealed. Upon selection of the boards, they will need to be weighed and the mass, date and time are all to be recorded prior to beginning the artificial rainfall. The surfaces of the test specimens should be positioned in such a way that they will be in maximum contact with the rainfall as it is applied. The rainfall is recommended to be applied with air atomizing nozzles but this can be discussed further with the regulatory Agency if another technology is considered to be more appropriate. At least three treated boards will need to be utilized as

well as three untreated boards. This will allow for a minimum of three water collections per sampling increment. This is required in order to have a sufficient sample size for data analysis.

- 7.5.2 The emissate collection basins will need to be placed underneath the board with the least amount of space to reduce dilution of the emissate for analysis. Deionised water will be used for the artificial rainfall, and the pH and temperatures should be recorded prior to initiating the study. The pH can also be collected post rainfall to study the potential impacts of the wood preservative on the pH of waters found in the environment. In addition, blank water samples need to be obtained and stored for analysis. This will allow the technician to identify, if any, interfering components that may impact the analytical results.
- 7.5.3 Each rainfall event should involve water being applied uniformly over the individual wood specimens and continued until the specimens achieve a representative moisture content regime (including wetting and drying). Since the moisture content will be driving the duration of the study, there is not a schedule to follow for the artificial rainfall; rather, the study should occur over the course of at least one month to allow for sufficient data collection. It is recommended to have at least three cycles of drying and wetting before terminating the study. With a significant amount of data, extrapolations can be considered more accurate.
- 7.5.4 If a different apparatus is utilized, the intensity of the rainfall (mm/hour) as well as the total amount of rainfall will need to be documented. The intensity of the rainfall will be in the range of 1mm/hour to 10 mm/hour. Refer to Section 3.3 for a complete discussion and examples of sampling regimes.
- 7.5.5 Between each rainfall event, the water will be removed and stored for analysis or analyzed immediately as appropriate. No water should be used for more than one rainfall event. Storage conditions will need to be documented and verified in terms of being favourable to the stability of the chemical being researched.

8 EMISSION MEASUREMENTS

Collected water is to be chemically analyzed for the active ingredient along with relevant by-products and degradates if appropriate. In addition, collection of the water (emissate) in subsequent analysis of untreated wood samples allow for an estimation of the interfering components. This second analysis serves as a control procedure to determine background levels of the test substance in untreated wood and to confirm that the wood used had not been previously treated with the preservative. Collection of these data will allow for the emission rate to be experimentally calculated.

8.1 *Treated Samples*

- 8.1.1 Collected water is chemically analyzed for the active ingredient and/or relevant transformation products, if appropriate. Collection of the water (emissate) in this system, and subsequent analysis of substances that have been emitted from the untreated wood samples, allow the estimation of the emission rate of the preservatives from the treated wood. Collection and analysis of the emissate after increasing time periods of exposure allows the rate of change of the emission rate to be estimated.

8.2 *Untreated Specimens*

8.2.1 Collection of the water (emissate) in this system and subsequent analysis of substances that have been emitted from the untreated wood samples allow the estimation of the active ingredient and /or relevant transformation products, or other substances from untreated wood. Collection and analysis of the emissate after increasing time periods of exposure allows the rate of change of the emission rate with time to be estimated. This analysis is a control procedure to determine background levels of the test substance in untreated wood, and to confirm that the wood used as a source of the samples had not been previously treated with the preservative.

9 **DATA AND REPORTING**

9.1 *Chemical Analyses*

9.1.1 The analytical method will need to be validated prior to study initiation. This will be accomplished through establishing a Limit of Quantisation (LOQ) as well as a Limit of Detection (LOD). Usually, the LOD is less than the LOQ, but both values justify that the analytical method is designed to obtain and detect at even the lowest levels. This will need to be done through an analysis of at least seven replicates of each fortification level to support the accuracy and precision of the method. The fortification levels include the LOQ, an intermediate concentration level, the maximum concentration level of the validation range, and a blank or control. The analytical method will be considered accurate and precise if a 70-120% recovery value is established and the coefficient of variation is equal to or less than 20%.

9.1.2 The collected water is chemically analyzed and the water analysis result is expressed in appropriate units, e.g., $\mu\text{g/l}$. The analytical result is converted to the absolute quantity over surface unit in the emissate in mg/m^2 . The emission flux for a day is calculated by taking the mean (where appropriate) of the measurement(s) taken on that day expressed in mg/m^2 to obtain the $\text{mg/m}^2/\text{day}$ or flux per day.

9.1.3 If the analysis of the samples from the untreated specimens shows detectable levels of the preservative used in the test, implying a background level obtained from untreated wood, it should be subtracted from the analysed results for the treated specimens.

9.1.4 If the analysis of the samples from the water shows detectable levels of the preservative used in the test, implying a background level in the water, it should be subtracted from the analysed results for the treated specimens and the untreated specimens.

9.2 *Reporting of Data*

9.2.1 All results are recorded (preferably using Excel, so that the data report can be easily extracted and reviewed by the appropriate Regulatory Agencies). Annex 1 shows an example of a suggested recording form for sets of treated test specimens, and the summary table for calculating the mean daily emission values over each sampling interval.

9.2.2 It is recommended that the pH of the test water is recorded for each specimen and sample time.

9.2.3 The mean of the results obtained from the three replicate sets is the daily emission rate for that product in mg/m²/day.

9.3 *Test Report*

9.3.1 At least the following information shall be provided in the test report:

- The name of the supplier of the preservative under test;
- The specific and unique name or code of the preservative tested;
- The trade or common name of the active ingredient(s) with a generic description of the coformulants (e.g. co-solvent, resin), and the composition in % m/m of the ingredients;
- The relevant retention or loading (in kg/m³ or litres/m² respectively) specified for wood used out of ground contact;
- A Batch Code and associated Certificate of Conformity or Certificate of Analysis for the active ingredients for product;
- Estimated moisture content of the specimens before the emission test;
- The species of wood used, with its density, growth rate in rings per 10 mm;
- The loading or retention of the preservative tested and the formula used to calculate the retention, expressed as litres/m² or kg/m³;
- Conditioning procedures used, specifying the type, conditions and duration;
- Specification of the end sealant used and the number of times applied;
- Specification of any subsequent treatment of the wood, e.g. specification of the supplier, type, characteristics and loading of a paint;
- The nominal surface area of a test specimen exposed to water, the nominal volume of water used for each immersion event and the ratio of the surface area to volume of water in cm²/cm³ (or cm²/ml);
- For specimens cut from commercially treated wood:
 - The date of application of the preservative, and the parameters of the treatment schedule used for a penetration process, or the method of application if a superficial treatment was used;
 - The retention or loading (in kg/m³ or liters/m² respectively) of the preservative under test, determined by chemical analysis
- For specimens treated in the laboratory:
 - The source and species of wood used, with its density, growth rate in rings per 10 mm, surface appearance;
 - Estimated moisture content of the specimens before treatment
 - The retention or loading (in kg/m³ or liters/m² respectively)
 - The method of application of the preservative, specifying the treatment schedule used for a penetrating process, and the method of application if a superficial treatment was used;
 - The date of application of the preservative, and an estimate of the moisture content of the test specimens, expressed as a percentage;
 - Conditioning procedures used, specifying the type, conditions and duration;
- For each test:
 - The time and date of each simulated rainfall event (immersion or artificial rainfall), the amount of water used for the immersion of the test specimens at each event, and the amount of water absorbed by the wood during immersion;
 - Mean daily emission values over each sampling interval;

- It is recommended that the pH of the test water is recorded for each specimen and sample time
- The mean of the results obtained from the tree replicate sets is the daily emission rate for that product in mg/m²/day;
- Re-sealing of a specimen
- Any variation from the described method and any factors that may have influenced the results.
- GLP-certificate

10 LITERATURE

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APPENDIX 1

Example Recording Form – page 1

Estimation of Emissions from Preservative-Treated Wood to the Environment: Laboratory Method for Wood held in Storage after Treatment and for Wooden Commodities that are not Covered, and are not in Contact with Ground

Test house	
Wood preservative	
Supplier of the preservative	
Specific and unique name or code of the preservative	
Trade or common name of the preservative	
Name and content (% m/m) of active ingredient(s)	
Name of co-formulants and composition (% m/m) of ingredients	
Relevant retention for wood out of ground contact	
Application	
Application method	
Date of application	
Formula used to calculate the retention:	
Conditioning procedure	
Duration of conditioning	
End sealant / number of times applied	
Subsequent treatment	if relevant
Test specimens	
Wood species	
Density of the wood	(minimum ... mean value ... maximum)
Growth rate (rings per 10 mm)	(minimum ... mean value ... maximum)
Moisture content	
Test assemblies*	Retention (e.g. kg/m³)
Treated ,x'	Mean value and standard deviation or range for 5 specimens
Treated ,y'	Mean value and standard deviation or range for 5 specimens
Treated ,z'	Mean value and standard deviation or range for 5 specimens
Untreated	
Test method parameters	e.g. testing regime used, water quality, dimension of test specimens, test conditions, surface area to water volume ratio, etc.

* x, y, z represent the three replicate samples

