Adopted:
16 October 2007

OECD GUIDELINE FOR THE TESTING OF CHEMICALS

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

INTRODUCTION

1. The emissions from preservative treated wood to the environment need to be quantified to enable an environmental risk assessment of the treated wood. This Guideline describes a laboratory method for the estimation of emissions from preservative treated wood in two situations where emissions could enter the environment:

- Emissions from treated wood in contact with fresh water. Emissions from the surface of the treated wood could enter the water.

- Emissions from treated wood in contact with seawater. Emissions from the surface of the treated wood could enter the seawater.

2. This Guideline is intended for testing the emissions from wood and wooden commodities that are not covered and are in contact with fresh water or seawater. Use Classes are used internationally and categorise the biological hazard to which the treated commodity will be subjected. 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 the preservative treated wood.

3. The method is a laboratory procedure for obtaining samples (emissate) from water used to immerse treated wood, at increasing time intervals after exposure. The quantity of emissions in the emissate is related to the surface area of the wood and the length of exposure, to estimate a flux in $mg/m^2/day$. The flux (leaching rate) after increasing periods of exposure can thus be estimated.

4. The quantity of emissions can be used in an environmental risk assessment of the treated wood.

INITIAL CONSIDERATIONS

5. The mechanism of leaching at the wood surface by fresh water is not assumed to be identical in nature and severity to leaching from a wood surface by seawater. Thus, for wood preservative products or formulations used to treat wood used in seawater environs, a wood leaching study for seawater is necessary.

6. The wood, in the case of wood treated with a wood preservative, should be representative of commercially used wood. It should be treated in accordance with the preservative manufacturer's instructions and in compliance with appropriate standards and specifications. The parameters for the post treatment conditioning of the wood prior to the commencement of the test should be stated.

© OECD, (2007)
You are free to use this material for personal, non-commercial purposes without seeking prior consent from the OECD, provided the source is duly mentioned. Any commercial use of this material is subject to written permission from the OECD.

7. The wood samples used should be representative of the commodities used (e.g., with regard to species, density and other characteristics).

8. The test can be applied to wood using a penetrating process or superficial application or to treated wood which has an additional mandatory surface treatment (e.g., paint that is applied as a requirement for commercial use).

9. The composition, amount, pH and the physical form of water is important in determining the quantity, content and nature of emissions from wood.

PRINCIPLE OF THE METHOD

10. Preservative treated wood test specimens are immersed in water. The water (emissate) is collected and chemically analyzed multiple times over the exposure period sufficient to perform statistical calculations. Emission rates in $mg/m^2/day$ are calculated from analytical results. The sampling periods should be recorded. Tests with untreated samples can be discontinued if there is no background detected in the first three data points.

11. The inclusion of untreated wood specimens allows for the determination of background levels for emissates from wood other than the preservative used.

QUALITY CRITERIA

Accuracy

12. The accuracy of the method to estimate emission depends upon the test specimens being representative of commercially treated wood, how representative the water is of real water and how the exposure regime is representative of natural conditions.

13. The accuracy, precision and repeatability of the analytical method should be determined before conducting the test.

Reproducibility

14. Three water samples are collected and analyzed and the mean value is taken as the emission value. The reproducibility of the results within one laboratory and between different laboratories depends upon the immersion regime and the wood used as test specimens.

Acceptable Range of Results

15. A range of results from this test where the upper and lower values differ by less than one order of magnitude is acceptable.

TEST CONDITIONS

Water

16. Freshwater leaching scenarios: Deionised water (e.g., ASTM D 1193 Type II) is recommended for use in the leaching test when wood exposed to freshwater is to be evaluated. The water temperature shall be 20 °C +/- 2 °C and the measured pH and water temperature included in the test report. Analysis of

samples of the water used taken before immersion of the treated specimens allows the estimation of the analyzed substances in the water. This is a control to determine background levels of substances which are then chemically analyzed.

17. Seawater leaching scenarios: Synthetic seawater (e.g., ASTM D 1141 Substitute Ocean Water, without Heavy Metals) is recommended for use in the leaching test when wood exposed to seawater is to be evaluated. The water temperature shall be 20 °C +/- 2 °C and the measured pH and water temperature included in the test report. Analysis of samples of the water used taken before immersion of the treated specimens allows the estimation of the analyzed substances in the water. This is a control for the analysis of background levels for substances of importance.

Wood Test Specimens

18. 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). Additional tests may be made using other species.

19. 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. The source, density and number of annual rings per 10 mm should be recorded.

20. Wood test specimens are recommended to be sets of five according to EN 113 size blocks (25 mm x 50 mm x 15 mm dimensions) with the longitudinal faces parallel to the grain of the wood, although other dimensions such as 50 mm, by 150 mm, by 10 mm may be used. The test specimen should be completely immersed into the water. Test specimens shall consist of 100% sapwood. Each specimen is uniquely marked so that it can be identified throughout the test.

21. All test specimens should be planed or plane sawn and the surfaces should not be sanded.

22. The number of sets of wood test specimens used for analysing is at least five: three sets of specimens are treated with preservative, one set of specimens is untreated and one set of specimens 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 specimens which are within 5% of the mean value of the preservative retentions of the pool of test specimens.

23. All test specimens are end-sealed with a substance which prevents penetration of preservative into the end grain of the specimens or prevents leaching from the specimens via the end grain. 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.

24. 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.

Immersion Container

25. The container is made of an inert material and is large enough to contain 5 EN113 wood specimens in 500 ml of water resulting in a surface area to water volume ratio of 0.4 cm^2/ml.

© OECD, (2007)

Specimen Test Assembly

26. The test specimens are supported on an assembly which allows all exposed surfaces of the specimen to be in contact with water.

PROCEDURE FOR PRESERVATIVE TREATMENT

Preparation of the Treated Test Specimens

27. The wood test specimen to be treated with the preservative under test is treated by the method specified for the preservative, which may be by a penetrating treatment process or a superficial application process, which may be with a dip, spray or brush.

Preservatives to be applied by penetrating treatment process

28. 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 for use in Use Class 4 or 5. The specimen is again weighed after treatment and the retention of the preservative (kg/m^3) is calculated from the equation:

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

29. 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.

Preservatives to be applied by superficial application processes

30. The superficial application process includes dipping, spraying or brushing of the wood test specimens. The process and application rate (e.g. $litres/m^2$) should be as specified for the superficial application of the preservative.

31. Also note in this case, 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.

Conditioning of the Test Specimens after Treatment

32. After treatment, the treated test specimens should be conditioned in accordance with the recommendations made by the supplier of the test preservative according to the preservative label requirements or as in accordance with commercial treatment practices or in accordance with EN 252 Standard.

Preparation and Selection of Test Specimens

33. After post treatment conditioning, the mean retention of the group of test specimens is calculated and three representative sets of specimens with a retention within 5% of the mean for the group are randomly selected for leaching measurements.

© OECD, (2007)

PROCEDURE FOR PRESERVATIVE EMISSION MEASUREMENTS

Immersion Method

34. The test specimens are weighed and subsequently totally immersed in the water and the date and time recorded. The container is covered to reduce evaporation.

35. The water is replaced at the following intervals: 6 hours, 1 day, 2 days, 4 days, 8 days, 15 days, 22 days, 29 days (note: these are total times not interval times). The time and date of the water change and the mass of water recovered from the container should be recorded.

36. After each water exchange, a sample of water in which the set of test specimens has been immersed is retained for subsequent chemical analysis.

37. The sampling procedure allows the calculation of the profile of the quantity of emissions against time. Samples should be stored under conditions that preserve the analyte e.g., in a refrigerator in the dark to reduce microbial growth in the sample before analysis.

EMISSION MEASUREMENTS

Treated Samples

38. Collected water is chemically analyzed for the active ingredient and/or relevant degradation/transformation products, if appropriate.

Untreated Samples

39. Collection of the water (emissate) in this system and subsequent analysis of substances that had leached from the untreated wood samples allow the estimation of the possible emission rate of the preservative from untreated wood. Collection and analysis of the emissate after increasing time periods of exposure allow 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 to confirm that the wood used as a source of samples had not been previously treated with the preservative.

DATA AND REPORTING

Chemical Analyzes

40. The collected water is chemically analyzed and the water analysis result is expressed in appropriate units, e.g., µg/L.

Reporting of Data

41. All results are recorded. The Annex shows an example of a suggested recording form for one set of treated test specimens, and the summary table for calculating the mean emission values over each sampling interval.

42. The daily emission flux in $mg/m^2/day$ is calculated by taking the mean of the three measurements from the three replicates and dividing by the number of days of immersion.

© OECD, (2007)

Test Report

43. 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/m^3 or L/m^2, respectively) specified for wood used in contact with water;
- The species of wood used, with its density, and 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 L/m^2 or kg/m^3;
- 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;
- 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 time and date of each immersion event, 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;
- Any variation from the described method and any factors that may have influenced the results.

© OECD, (2007)

LITERATURE

1) European Standard, EN 84 – 1997. Wood preservatives. Accelerated ageing of treated wood prior to biological testing. Leaching procedure.

2) European Standard, EN 113/A1 – 2004. Wood preservatives. Test method for determining the protective effectiveness against wood destroying basidiomycetes. Determination of the toxic values.

3) European Standard, EN 252 – 1989. Field test method for testing the relative protective effectiveness of a wood preservative in ground contact.

4) European Standard, EN 335 – Part 1: 2006. Durability of wood and wood-based products – Definition of use classes – Part1: General.

5) American Society for Testing and Materials Standards, ASTM D 1141 – 1998. Standard Practice for the Preparation of Substitute Ocean Water, Without Heavy Metals. *Annual Book of ASTM Standards*, Volume 11.02.

6) American Society for Testing and Materials Standards, ASTM D 1193-77 Type II – 1983. Specifications for Reagent Water. *Annual Book of ASTM Standards,* Volume 11.01.

© OECD, (2007)

ANNEX

Recording Form for Guideline

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

Test house	
Wood preservative	
Supplier of the preservative	
Specific and unique name or code of the preservative	
Trade or common name of the preservative	
Coformulants	
Relevant retention for wood used in contact with water	
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	
Variation of guideline parameters	e.g. water quality, dimension of test specimens etc.

* x, y, z represent the three replicate samples

Time	Water exchange	Specimen mass		Water uptake		Water sample				
		Treated (mean)	Untreated	Treated (mean)	Untreated		Test water	x	y	z
	Date	g	g	g	g	no.	pH	pH	pH	pH
start										
6h						1				
24h						2				
2 d						3				
4 d						4				
8 d						5				
15 d						6				
22 d						7				
29 d						8				

Please prepare separate tables for each active ingredient

Time	Water exchange	Analytical Results															
		Untreated specimens			Treated specimens												
		Concen-tration a.i. in water	Quantity emitted	Emission rate	Concentration a.i. in water				Quantity emitted				Emission rate				
					x	y	z	Mean	x	y	z	Mean	x	y	z	Mean	
	Date	mg/L	mg/m²	mg/m²/d	mg/L	mg/L	mg/L	mg/L	mg/m²	mg/m²	mg/m²	mg/m²	mg/m²/d	mg/m²/d	mg/m²/d	mg/m²/d	
6h																	
24h																	
2 d																	
4 d																	
8 d																	
15 d																	
22 d																	
29 d																	

Note: Since results from untreated may have to be used to correct emission rates from treated samples, the untreated results should come first and all values for treated samples would be "corrected values". There may also be a correction for the initial water analysis.

© OECD, (2007)