PD CEN/TS 16663:2016

BSI Standards Publication

Durability of wood and wood-based products — Determination of emissions from preservative treated wood to the environment — Wooden commodities exposed in Use Class 3 (Not covered, not in contact with the ground) — Semi-field method

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National foreword

This Published Document is the UK implementation of CEN/TS 16663:2016. It supersedes PD CEN/TR 16663:2014 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee B/515, Wood preservation.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Durability of wood and wood-based products - Determination of emissions from preservative treated wood to the environment - Wooden commodities exposed in Use Class 3 (Not covered, not in contact with the ground) - Semi-field method

Durabilité du bois et des matériaux dérivés du bois - Détermination des émissions dans l'environnement du bois traité avec des produits de préservation - Produits de base en bois exposés à la classe d'emploi n° 3 (dans un endroit abrité, n'étant pas en contact avec le sol) - Méthode semi-terrain

Dauerhaftigkeit von Holz und Holzprodukten - Abschätzung von Emissionen von mit Holzschutzmitteln behandeltem Holz an die Umwelt - Holzprodukte in Gebrauchsklasse 3 (nicht abgedeckt, ohne Erdkontakt) - Semi-Feldverfahren

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European foreword

This document (CEN/TS 16663:2016) has been prepared by Technical Committee CEN/TC 38 "Durability of wood and wood-based products", the secretariat of which is held by AFNOR.

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Introduction

The leaching from preservative treated wood into the environment needs to be quantified to enable an environmental risk assessment to be made of the treated wood, e.g. according to the Biocidal Products Regulation, 528/2012. This document describes a semi-field method for the determination of leachate from preservative treated wood where the preservative treated wood is not covered and not in contact with the ground or water (use class 3 according to EN 335).

The method is a semi-field procedure for obtaining water samples (leachate) from treated wood exposed out of ground contact, during natural exposure. The quantities of emissions in the leachate are related to the surface area of the wood and can be used in scenarios for the environmental risk assessment of the treated wood.

NOTE The leachate can also be tested for eco-toxicological effects (example: OECD 202 testing on *Daphnia* sp.).

1 Scope

This Technical Specification specifies a method for determining the leaching of active ingredients or other compounds from treated wood by a semi-field method for use class 3 (outdoor above ground). The preservative treated wood can be tested with or without subsequently surface coating or other water-repellent treatment. The method is applicable to the testing of commercial or experimental preservatives or paint systems applied to timber by methods appropriate to commercial practice.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN ISO 5667-3, *Water quality - Sampling - Part 3: Preservation and handling of water samples (ISO 5667-3)*

3 Description of the test method

3.1 Principle

Panels are treated, assembled and placed outdoors, out of ground contact and exposed to the normal environmental and ecological factors which affect preservative treated wood so exposed in practice. The rainwater is retained and the leachate is monitored by chemical analyses of the active ingredients and/or other compounds.

3.2 Quality criteria

The validity of the analytical method for the substances in question should be determined before conducting the test:

a) accuracy;

b) specificity;

c) limit of detection (LOD);

d) limit of quantification (LOQ);

e) precision.

NOTE 1 EN ISO 5667-3 may give guidance on the preservation and handling of water samples.

NOTE 2 This may include CAS no or chemical formulation.

3.3 Wood preservative

The test report shall state the name and other designation of the tested product, and the trade or common name of the active ingredient(s) as defined in the regulation n°528/2012.

Use of a coating is possible (identity and amount of coating used shall be stated in the test report).

3.4 Apparatus

3.4.1 Vessel for receiving water

Laboratory brown glass flask or plastic jars with no impurities that can influence the active ingredient.

NOTE EN ISO 5667-3 gives the good practices to preserve water samples.

The brown glass flask or plastic jars shall be protected from heat and sunlight.

The capacity of the vessels depends on the exposure scenario. If the expected annual precipitation is approximately 700 mm and the test set-up is placed horizontal 25 l containers are recommended. If the test set-ups are exposed vertically 5 l containers are recommended.

3.4.2 Gutter, screws, hooks

All materials used to fix and support the panels and collect the rainwater shall be made of inert materials. Stainless steel has been found to be suitable (see Annex A).

3.4.3 Weather station

A weather station capable of monitoring the quantity of rainfall, the wind direction and wind speed at the test site shall be used.

3.4.4 Wood working equipment

Equipment capable of producing the desired finish of the surface; e.g. fine sawn or planed.

3.4.5 Condition chamber

Conditioning chamber, well ventilated, controlled at (20 ± 2) °C and (65 ± 5) % relative humidity.

3.4.6 Mixing vessel

3.4.7 Preservative treatment

Equipment suitable for carrying out vacuum, vacuum-pressure, immersion or surface treatment of specimens.

3.4.8 Balance

A balance capable of weighing to the nearest 0,1 g.

3.4.9 Kiln

A kiln suitable to dry the preservative treated timber after treatment and post treatment conditioning, if required. The drying procedure shall follow recommendations from the supplier of the product under test.

3.4.10 Safety equipment and protection clothing

Appropriate for the test product, to ensure the safety of the operator.

3.4.11 Refrigerator/Freezer

A refrigerator or freezer (minimum - 18°C) to store the leachate at low temperature to reduce degradation of the substances to be analysed and the growth of mircoorganisms in the leachate. The storage procedure shall follow recommendations from the supplier of the product under test or EN ISO 5667-3.

3.4.12 Chemical analysis equipment

Analytical equipment appropriate to determine the compounds of interest to be analysed in the leachate at the desired concentration.

3.4.13 Exposure site

An open area, free from tall vegetation and not excessively influenced by industrial or agricultural pollution and no obstacle to the rain. Distance to building should be specified in the test report.

3.4.14 Exposure weathering racks

The exposure racks, to which the panels are attached, should be robust to maintain the panels in the required orientation for the duration of the test. Racks that have been found to be suitable are illustrated in Annex A.

3.5 Test specimens

3.5.1 General

The test specimens that make up the test panel are representative or typical of commercial timber, and exposed to mimic service exposure (e.g. vertical spruce shiplap cladding. Alternatively the test specimens may be selected to be a 'realistic worst case' estimation of the emission for a wood preservative in Use Class 3.

3.5.2 Species of wood

The wood species shall be typical of the wood species used for the efficacy testing of wood preservatives, e.g. sapwood of *Pinus sylvestris* (L) (Scots pine).

Additional tests may be made using other species; this should be stated in the test report.

3.5.3 Quality of wood and wood moisture content

The quality of wood and wood moisture content shall be typical of the wood used commercially or use sound straight-grained wood. Material of resinous appearance shall be avoided. Use wood with between 2 annual growth rings per 10 mm and 10 annual growth rings per 10 mm in case of Scots pine. The proportion of latewood in the annual rings shall not exceed 30 % of the whole cross-section for Scots pine sapwood.

When penetrating processes are being used for the treatment the specimens shall consist of sapwood only when using Scots pine. For superficial treatments some heartwood is permitted on the back of the specimens. An example is given in Annex C.

In a batch of specimens to be treated, the density of an individual is permitted to differ from the mean value of the batch by ± 15 %. The mean density of the treated specimens used for the test shall be recorded in the test report.

Knots are permitted to a limited extent. One knot less than 2 cm in diameter and maximum 3 knots of less than 1 cm are permitted in each board.

If other wood species are used the number of annual rings and density shall be stated in the report.

The density of the panels for each test setup is distributed evenly. Knots and other defects are evenly distributed over the 3 replicas. Annex C gives guidance to the preferred orientation of the growth rings.

3.5.4 Preparation of test specimens

The timber is conditioned to 12 ± 2 % mass fraction moisture content.

Prepare test specimens of (100 ± 2) mm × (25 ± 2) mm cross section of sawn timber. The length of the panel is (760 ± 5) mm. Panels of other sizes can be prepared and used in this test.

Alternatively the test specimens can be planed or prepared in a way typical of commercial practice. The actual dimensions of the test specimens shall be stated in the report.

Individual test specimens shall be assigned unique identification number in such a way that these numbers are retained trough out all preparation operations.

3.5.5 Number of test specimens

Prepare at least 21 panels for each combination of test parameters. Seven panels are used for one test set-up. Three replicas for each combination of test parameters are prepared.

3.5.6 End seal

The wood panels are end-sealed after preservative treatment by a material, which prevents water entry as well as microbial infection. The end-sealer shall remain effective during long-term exposure to the weather and may not contain biocides.

3.6 Procedure

3.6.1 Product under test

The sample of preservative shall be representative of the product to be tested. The product shall be analysed for active ingredient content prior to use.

If a particular coating or water-repelling system is specified by the preservative supplier as an integral part of the protection system, it shall be included in the test.

Samples shall be stored and handled in accordance with any written requirements from the supplier.

3.6.2 Treatment and handling of test specimens

Determine and record the volume of each panel at 12 % moisture content. Prior to product treatment determine the mass to the nearest 0,1 g.

Apply the test preservative to the test specimen panels using the process specified by the supplier of the preservative/protection system.

A list of model treatments is given in Annex B.

After treatment, excess solution on the surface of the test specimens shall be allowed to drip off or be removed with a cloth. Reweigh each test specimen immediately and record the mass after treatment to the nearest 0,1 g. Calculate the uptake of preservative solution and active ingredients for each test specimen and express the result in kilograms of preservative/ active ingredients per cubic metre of wood for penetration processes and in grams of preservative per square metre of wood for superficial application methods.

Reject any test specimen with retention deviating by more than 15 % from the average for that type of test specimen and substitute an appropriate alternative which falls within this range.

Determine the average preservative uptake of 7 test specimens for one panel. The average preservative uptake between the 3 panels shall be minimized and may not exceed 15 %.

The preservative supplier's instructions for the drying and post treatment conditioning process should be followed. A process which has been found to be suitable is to dry the treated panels in a well-ventilated area protected from rain and frost and a temperature of max 30°C. Ensure free ventilation when storing the panels. Use an inert material such as glass or stainless steel to support the panels. The panels may not touch each other. Alternative a kiln can be used (see 3.4.9). The process shall be reported in the test report.

Follow the supplier's instructions for:

— Drying of the treated panels after treatment;

— Period between treatment and exposure (the time shall be noted in the report);

— Application of surface coating or water-repellent treatment (if relevant).

Before exposure:

— Apply the weatherproof end-seal to the top end of the panels (see 3.5.6);

— Ensure permanent labels are fixed to the back of the panel. Alternatively the identification may be written with a water-proof marker at the end of the panel prior to applying a water-proof transparent end-seal.

3.6.3 Untreated controls (optional)

An untreated control panel is optional, to measure the background levels of the analysed substances in rainfall, and emitted from untreated wood. The control test specimens and panels shall be prepared, stored and handled in the same way as treated test specimens and panels before installation.

3.6.4 Exposure test site

The test is valid using one test site; however, it may be advantageous to select more than one test site representing markedly different climatic conditions.

Place the test panels on the weather rack (see Annex A).

The test set up can be exposed horizontally or vertically depending on the data required. The vertically exposed test setups can be orientated to reflect the most severe weather conditions concerning wind and rain.

3.6.5 Duration of the test

The duration of the assessment period depends on the data required. The aim is to have sufficient data points to allow extrapolation in order to estimate a long term leaching.

For the assessment of long term emission according to Biocidal Products Regulation, study duration of 2 years has been found to be suitable. It is recommended to continue the test until the accumulated leaching in mg/m^2 has reached a plateau.

3.6.6 Collection of the leachates

The leachate shall be sampled after each significant rain event or after the required period of exposure. The time and the date are recorded.

The sampling period should be increased to larger intervals as the test progresses, but a short sampling period in the beginning is known to be suitable. It can be increased according to rainfall, e.g. 0 mm-50 mm, 50 mm-100 mm, 100 mm-200 mm, 200 mm-400 mm of rainfall).

Any signs of microbiological growth in the leachate or on the wood shall be recorded in the report.

The leachate from different rain events can be bulked. A sample of the leachate from a study period may be stored (e.g. 1 l). The amount of leachate can be large and it can be an advantage to store only a few percent of the leachate. The same percentage of leachate shall be sampled and stored after each rain event.

Samples should be stored under conditions that preserve the compounds of interest, e.g. in a refrigerator or in a freezer in the dark to reduce microbial growth in the sample before analysis (see Annex D).

NOTE Guidance on the preservation and handling of water samples according to EN ISO 5667-3.

These conditions have to be convenient with the nature (organic, inorganic compound) of the active ingredient. A maximum time of storage before the analysis should be defined.

The leachate is stored in a freezer at a temperature below −18 °C after for example, acidification for inorganic compound or extraction for organics.

3.6.7 Chemical analysis

Chemical analysis is conducted after a planned time schedule. Chemical analysis is carried out on the leachate collected (or a sample) after increasing periods of exposure (e.g. 0 d-30 d, 30 d-100 d).

NOTE This is an example of a time schedule: 0 mm-50 mm of rain, 50 mm-100 mm of rain, 100 mm-200 mm of rain, 200 mm-400 mm of rain, 400 mm-700 mm of rain, 700 mm-1 100 mm of rain, and so on.

3.7 Expression of results

The leachate is analysed for active ingredients and the results are expressed in µg/mL or mg/mL.

The leaching is then calculated as mg active ingredient per square metre exposed wood, mg/m^2, for the chosen period, e.g. 0 mm-50 mm, 50 mm-100 mm, etc.

The area of exposed wood for one test set-up is defined as the front, 2 small sides of the test specimens and the bottom end grain. For specimens with the cross section of 100 × 25 mm the exposed area for each test set-up is:

$$7 \times (0{,}76\ \text{m} \times 0{,}1\ \text{m} + 2\ (0{,}76\ \text{m} \times 0{,}025\ \text{m}) + 0{,}025\ \text{m} \times 0{,}1\ \text{m}) = 0{,}815\ 5\ \text{m}^2$$

Additional assessment shall be done, for example the percentage of loss of an ingredient after a given exposure period.

4 Test report

The test report shall include:

a) number of this document (CEN/TS 16663:2016);

b) the specific and unique name or code of the product tested;

c) the name of the organization responsible for the report and the date of issue;

d) the name(s) and signature(s) of the persons responsible;

e) the date of order, the name and address of the sponsor of the test;

f) chemical analysis of the biocides of the wood preservative used;

g) any solvent or diluent used to dilute the wood preservative tested;

h) if an additional surface treatment is used, the specific and unique name or code of the product tested;

i) the wood species and the density of the selected panels;

j) the surface texture of the wood (e.g. sawn or planed);

k) the number of replicates;

l) the method and date of treatment with the test preservative;

m) the method and length of conditioning of the wood specimens;

n) the individual and mean retention of product in kilograms per cubic metre or grams or ml per square metre as appropriate;

o) the method and drying time after treatment with the test preservative and details of post treatment conditioning;

p) the method and date of treatment with the additional surface treatment (if relevant);

q) the individual and mean retention of the additional surface treatment in grams per square metre or ml per square metre as appropriate;

r) the method and drying time after treatment with the additional surface treatment and details of post treatment conditioning;

s) the location (latitude, longitude and elevation) and detailed description of the exposure site(s) including information on temperature, rainfall (precipitation) and orientation;

t) the date and installation of the test set-ups;

u) the date of each collection leachate and the corresponding mm rainfall for each chemical analysis period;

v) the conditions of storage of the leachate prior to chemical analysis;

w) Analytical methods;

x) the duration of the exposure period(s);

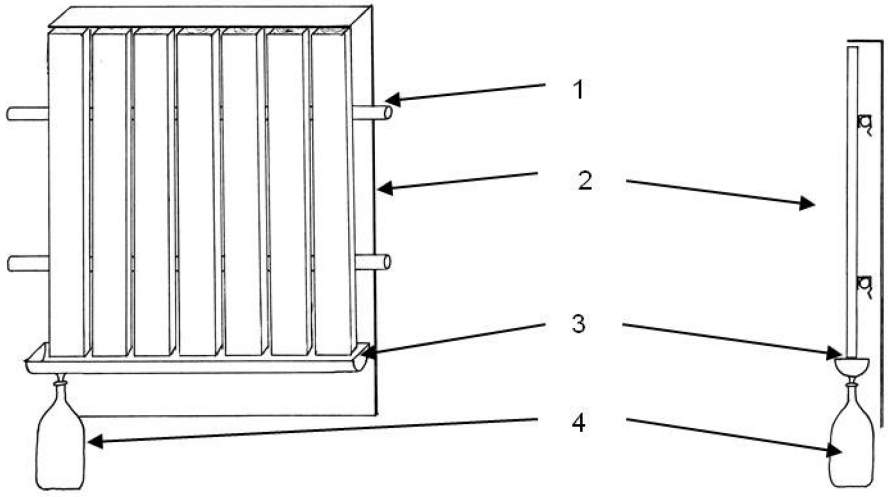
y) the leaching in mg/m^2 exposed wood for the assessment periods;

z) any deviations from this standard and any special factors which may have influenced the results.

Annex A
(informative)

Test set-up and weather rack

A.1 Vertical exposure



a) Panels exposed vertically **b) Sectional view**

Key

1 stainless steel tubes

2 backing to prevent rain from behind

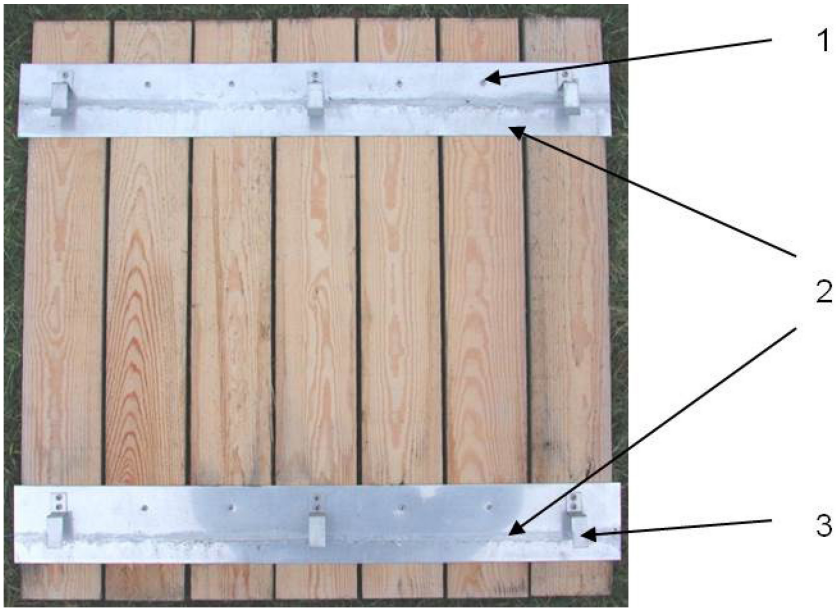
3 gutter made of stainless steel

4 laboratory glass flask to sample rain

Figure A.1 — Test set-up for vertical exposure

The following pictures show examples of test set-ups and weather rack at the field test area:

Figure A.2 — Vertically oriented test series



Key

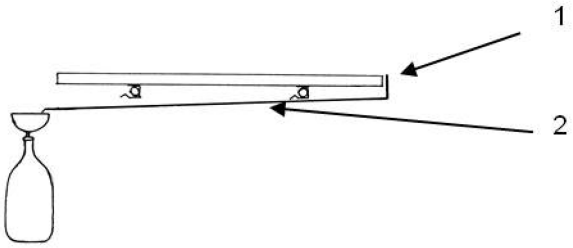
1 stainless steel screws

2 stainless steel boards

3 stainless steel hooks

Figure A.3 — Attachment of the panels for the test set-up

A.2 Horizontal exposure



Key

1 end grain sealed

2 stainless steel tray

Figure A.4 — Sectional view panels exposed horizontally

Annex B
(informative)

Model treatments

B.1 For preservative intended for superficial treatment

— Dipping process: Total immersion for approximately 3 min.

— Brushing: (150 ± 30) g/m^2.

B.2 For preservative intended for penetration treatment

— Double vacuum process:

  — initial pressure: 0,7 bar for 3 min;

  — atmospheric pressure for 3 min;

  — final pressure: 0,35 bar for 20 min.;

— Vacuum pressure process:

  — initial pressure: 0,1 bar for 60 min;

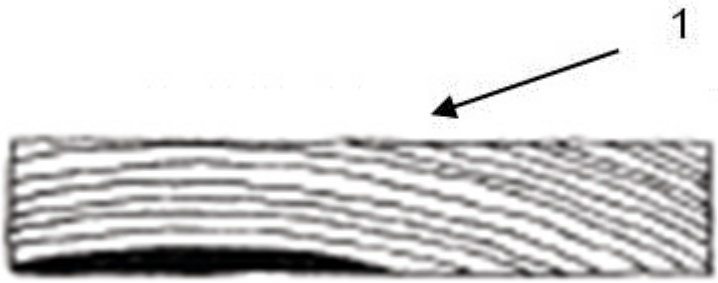
  — final pressure at 12 bar for 120 min.

Annex C
(informative)

Orientation of annual growth rings and accepted heartwood

The following figure gives an example on the orientation of annual growth rings.

For superficial treatments the amount and location of accepted heartwood is also shown. There should be minimum 15 mm of sapwood from the surface.



Key
1 Exposed side

Figure C.1 — Example on the orientation of annual growth rings

Annex D
(informative)

Stability study

When an experiment is initiated which includes new or unknown substances for the laboratory it is necessary to ensure that these substances can be handled, stored an analysed during the test.

Information concerning potential breakdown of the substances during the test and possible metabolites can be available from the manufacturer or literature. Also information concerning storage and analytical procedures can be available.

In some cases it is necessary to conduct a stability study to ensure that the substances can be detected during the test and to establish limits of quantification. The following procedure is suggested:

— 10 l x 1 l demineralized water is spiked with the substances in question so the concentration is in the range expected in the test.

— 1 bottle is analysed immediately.

— 4 bottles are placed at 4 °C in the dark.

— 4 bottles are placed at room temperature and exposed to sunlight.

— 1 bottle is placed in the freezer at - 21 °C.

Concentration of the substances is analysed after 0 d, 4 d, 9 d and 14 d exposure.

The sample in the freezer is analysed after 14 d.

At the time defined above each sample is extracted with an appropriate organic solvent. The solvent is stored at −18 °C until the end of the stability study. At the end of the stability study all samples are analysed using appropriate equipment. The extracted samples are stable in organic medium as long as the samples are protected from sun light.

— **Limit of detection/quantification**

The analytical method is developed and different calibration solutions (at different concentrations) are analysed. The quantification limit is chosen to be the concentration of the most diluted calibration solution which gives a good correlation.

— **Recovery**

A triplicate recovery experiment is performed at two levels: just above the quantification limit and at a concentration, which is expected to be close to maximum concentrations that will be found during the study.

Based on the results from the stability study the following conditions can be defined:

— stability of the substances at the test site;

— optimal storage procedure of the leachate;

— LOQ.

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