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BSI Standards Publication

Durability of wood and wood-based products — Preservative-treated solid wood — Determination of the penetration and retention of creosote in treated wood

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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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Dauerhaftigkeit von Holz und Holzprodukten - Mit Holzschutzmitteln behandeltes Vollholz - Bestimmung der Eindringtiefe und der Aufnahme von Kreosot (Teerimprägnieröl) in behandeltem Holz

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Foreword

This document (EN 12490:2010) 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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1 Scope

This European Standard specifies the reference methods for determining the penetration and retention of creosote in timber freshly treated with creosote, principally in order to ascertain whether the treated timber conforms to specifications written in terms of EN 351-1. It also provides guidance on the acquisition of test samples and their handling between sampling and analysis.

NOTE In the day-to-day practice at the plant, other methods (e.g. weighing the charge before and after treatment) can be used for determining the retention, provided that a significant relationship can be established with this method.

2 Normative references

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

EN 351-1, *Durability of wood and wood-based products — Preservative-treated solid wood — Part 1: Classification of preservative penetration and retention*

EN 351-2, *Durability of wood and wood-based products — Preservative-treated solid wood — Part 2: Guidance on sampling for the analysis of preservative-treated wood*

EN ISO 3696:1995, *Water for analytical laboratory use — Specification and test methods (ISO 3696:1987)*

ISO 3131, *Wood — Determination of density for physical and mechanical tests*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
analytical zone
part of the treated wood which is analysed for assessing compliance with the retention requirement

[Adapted from EN 1001-2:2005, 4.03]

NOTE For definition of "retention requirement", see 3.6.

3.2
batch
clearly identifiable collection of units of preservative-treated wood manufactured to conform to the same defined penetration and retention requirements

[EN 1001-2:2005, 4.04]

3.3
charge
all the wood treated together in a single operation

[EN 1001-2:2005, 4.13]

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3.4
composite sample
collection of all test samples derived from the sampling units taken from the batch in accordance with the chosen sampling plan for the determination of retention

[EN 1001-2:2005, 4.15]

3.5
penetration requirement
minimum depth to which the creosote is required to penetrate the wood

[Adapted from EN 1001-2.2005, 4.59]

3.6
retention requirement
loading of the creosote that is required in the analytical zone

[Adapted from EN 1001-2:2005, 4.73]

NOTE 1 For definition of "analytical zone" see 3.1.

NOTE 2 The retention requirement is expressed in kilograms of creosote per cubic metre of treated wood.

3.7
sampling unit
unit (for example a pole, a sleeper, a board or a fence post) of preservative treated wood taken from a batch or charge of preservative treated wood

[Adapted from EN 1001-2:2005, 4.75]

NOTE For definition of "batch" see 3.2. For definition of "charge" see 3.3.

3.8
test sample
portion of preservative treated wood taken from a sampling unit, in accordance with the recommendations of EN 351-2

3.9
transition wood
wood in a zone between the true sapwood and the true heartwood

[Adapted from EN 1001-2:2005, 1.45]

NOTE This is distinguishable only in a very few wood species. In general its treatability is similar to that of heartwood.

4 Sampling of creosote-treated wood for the determination of penetration and retention

4.1 General requirements

The acquisition of sampling units and test samples shall follow the procedures established in EN 351-1 and EN 351-2. Additionally, when sampling from a freshly treated batch, the wood shall be allowed to cool to ambient temperature before taking test samples.

4.2 Specific requirements for test samples for the determination of penetration

The penetration of creosote in each of the test samples shall be determined immediately after sampling, according to Clause 5, in order to avoid creosote migration from the treated to the untreated area.

4.3 Specific requirements for test samples for the determination of retention

A batch to be sampled for the determination of retention shall be sampled at a time less than 30 days after the treatment.

NOTE 1 The retention requirements defined by the specifier are only applicable to treated wood as produced, not to the treated wood in service.

The composite sample for a batch or charge shall comprise test samples taken in accordance with the chosen sampling plan. Sufficient test sample material shall be taken to ensure that at least 1 g of creosote is contained in the composite sample.

NOTE 2 Wood treated with creosote by vacuum/high pressure processes can normally be expected to contain more than 10 % by mass of creosote. Therefore, it will require approximately 10 g to 12 g of creosoted wood to obtain at least 1 g of creosote in the composite sample.

NOTE 3 The larger the number of test samples included in the composite sample, the more accurate the resultant retention determination should be.

As soon as possible after the test samples have been obtained, and the penetration measured the wood not included in the specified analytical zone of the test samples shall be removed. The remaining wood constitutes the composite sample for the determination of retention.

NOTE 4 If the penetration and retention are determined from the same test samples, the penetration should be determined before any wood is removed from the test samples.

The composite sample shall be stored in a stoppered glass bottle to prevent any free creosote being lost before the analysis.

5 Determination of the penetration of creosote in treated wood

For each of the test samples, measure and record the penetration of creosote in the treated wood, as the distance, in millimetres (mm), of the furthest point from the surface to which creosote can be seen to be present in the wood, in accordance with the general recommendations in EN 351-2.

NOTE 1 Penetration should be assessed visually from a freshly cut test sample, the colour of the creosote clearly indicating its extent in the treated timber.

NOTE 2 In some cases the creosote may not penetrate continuously through the early and late wood of the treated timber.

NOTE 3 If the boundary between the sapwood and the heartwood cannot be distinguished visually, in some cases a chemical method can be used to distinguish between them. Some examples are given in Annex A. Where no distinction is possible, all the wood is regarded as sapwood.

NOTE 4 Occasionally small zones of sapwood adjacent to the heartwood cannot be treated, for example transition wood. These should be ignored for the purposes of assessing sapwood penetration.

NOTE 5 Creosote treated zones can be subsurfaced by migrating creosote during sampling. To fully visualize creosote treated zones within taken voluminous drilling cores from sampling units, a final cut through the centre of the core, direction from the untreated to the treated zone and therefore surface, should be made. The inner untreated zone cannot be subsurfaced.

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6 Determination of the retention of creosote in treated wood

6.1 Reagents

6.1.1 Water of grade 3, according to EN ISO 3696:1995.

6.1.2 Chromic acid saturated solution in concentrated sulphuric acid.

6.1.3 Toluene, $C_6H_5CH_3$, analytical grade.

6.1.4 Suitable detergent solution in water.

6.2 Apparatus

Ordinary laboratory apparatus and the following (see Figure 1).

6.2.1 Glass round-bottomed flask, capacity 500 ml or 1 000 ml.

6.2.2 Heating mantle for the glass round-bottomed flask (6.2.1).

6.2.3 Glass Soxhlet-apparatus, capacity 60 ml.

6.2.4 Cellulose extraction thimble (28 mm diameter and 80 mm height).

6.2.5 Glass water trap, Dean and Stark type, with a capacity of 10 ml, fitted with a stopcock and marked in graduations of 0,1 ml.

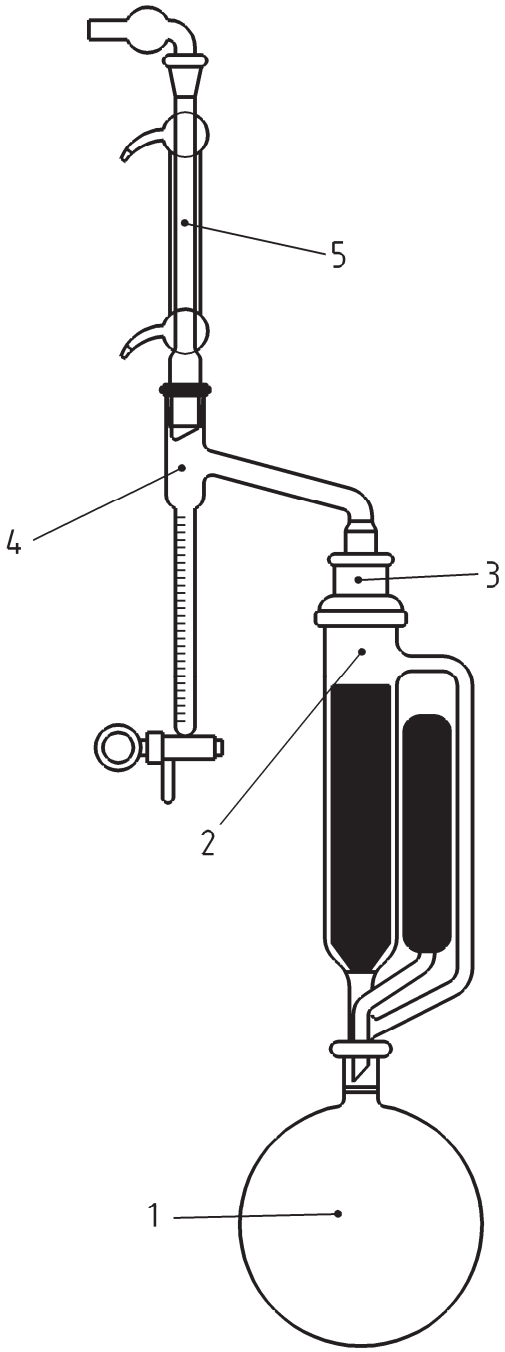
6.2.6 Glass reflux condenser of the "Liebig-West" type.

6.2.7 Balance with an accuracy of 0,01 g.

6.2.8 Glass rod, or rod made of another inert material, with a diameter of about 3 mm and as long as the condenser (6.2.6) and the water trap (6.2.5) together.

6.2.9 Vented drying oven, that can be maintained at (115 ± 5) °C and operated in a fume cupboard.

6.2.10 Desiccator with a drying agent of the indicating type (e.g. silica gel).



Key

1 Round bottom flask (6.2.1)
2 Soxhlet-apparatus (6.2.3) and extraction thimble (6.2.4)
3 Adaptor that fits between the Soxhlet (6.2.3) and the water trap (6.2.5)
4 Water trap (6.2.5)
5 Reflux condenser (6.2.6)

Figure 1 — Apparatus

6.3 Preparation of the water trap and the condenser before the extraction

Thoroughly clean the inner surface of the water trap (6.2.5) of the apparatus and the inside surface of the condenser (6.2.6) with a warm solution (50 °C to 60 °C) of detergent (6.1.4). Rinse thoroughly to remove all trace of detergent with water (6.1.1), and allow to drain for a few minutes. Dry the outer surface of the water trap. Inspect carefully the inner surface of the water trap and condenser. If droplets of water remain on these surfaces, repeat the cleaning operation several times with the detergent solution (6.1.4).

If this fails to produce a droplet-free inner surfaces, rinse the equipment several times with a saturated solution of chromic acid (6.1.2).

CAUTION — Care should be taken to prevent contact of the chromic acid solution (6.1.2) with skin or clothing. If contact does occur, wash immediately with copious quantities of water.

After treatment with this acid solution, rinse the apparatus thoroughly with water (6.1.1), drain and inspect as before. Repeat the cleaning operation, if necessary.

6.4 Procedure for the extraction of creosote from the composite sample

Heat an empty extraction thimble (6.2.4) in the oven (6.2.9) at (115 ± 5) °C for a minimum of 90 min. Transfer it to the desiccator (6.2.10) and allow to it to cool to room temperature.

CAUTION — Do the following operations in a fume cupboard.

Transfer about 200 ml of toluene (6.1.3) to the round-bottomed flask (6.2.1) and add approximately 2 ml of water (6.1.1). Assemble the apparatus as in Figure 1. Place the flask (6.2.1) in the heating mantle (6.2.2), apply heat and reflux for about 30 min. Discontinue the heating and allow the contents of the water trap to cool to room temperature. Free any water adhering to the walls of the condenser or water trap so that it drops to the water layer in the trap, using the rod (6.2.8). Using the stopcock, drain the water from the trap until approximately 1 ml remains and record the water level to the nearest 0,1 ml.

Remove the test samples from the stoppered glass bottle (4.3). Cut the test samples into small pieces, so that they will fit into the extraction thimble (6.2.4). Weigh the composite sample to the nearest 0,01 g in a weighing bottle and transfer it to the extraction thimble (6.2.4), taken from the desiccator (6.2.10). Place the thimble in the Soxhlet (6.2.3). Weigh the empty weighing bottle to the nearest 0,01 g and record the mass of the composite sample (m_1), calculated by difference.

NOTE 1 The procedures involving the composite sample should be carried out quickly and efficiently to avoid loss of creosote during handling.

Apply heat and reflux the toluene at a rate where at least one drop per second falls from the tip of the condenser. The extraction time shall be not less than 2 h and not more than 6 h and, within this 6 h, the extraction shall continue until the toluene in the Soxhlet apparatus becomes colourless.

After this extraction, discontinue the heating and allow the contents of the water trap to cool to room temperature. Free any water adhering to the walls of the condenser or the water trap so that it drops to the water layer in the trap, using the glass rod (6.2.8). Read the water content of the trap to the nearest 0,1 ml. Record the difference in the amount of water before and after the reflux, as the mass of water (m_2) in the sample (1,0 ml = 1,0 g).

NOTE 2 It is very important to recover all the water droplets adhering to the condenser tube, the condenser tip and the sides of the water trap, as the calculated retention will depend very much on the mass of water (m_2).

Remove the thimble (6.2.4) from the Soxhlet apparatus and allow it to stand in a fume cupboard until the sample appears dry. Transfer the contents of the thimble quantitatively to a watch glass. Place the watch glass in the oven (6.2.9) for 24 h at 115 °C. Transfer it to the desiccator (6.2.10) and allow to cool to room temperature. Weigh the watch glass with the extracted wood samples to an accuracy of 0,01 g. Remove the wood sample and weigh the watch glass to a similar accuracy. Record the net mass of the wood (m_3), calculated by difference.

6.5 Expression of results

Calculate the fraction of the original sample which was creosoted, using the following equation:

$$K = \frac{m_1 - m_2 - m_3}{m_3} \quad (1)$$

where

K is the fraction of creosote;

m_1 is the mass, expressed in grams (g), of the composite sample before extraction;

m_2 is the mass, expressed in grams (g), of the water in the sample, calculated as the difference of the amount of water in the water trap before and after extraction;

m_3 is the mass, expressed in grams (g), of the oven dry extracted material.

Calculate the retention of creosote (R) in the analytical zone of the charge or batch (expressed in kilograms per cubic metre (kg/m³)), using the equation:

$$R = K \times \rho \times 1{,}2 \quad (2)$$

where

K is the fraction calculated above;

ρ is the density, expressed in kilograms per cubic metre (kg/m³), of the untreated wood determined, prior to treatment, according to ISO 3131 in the absolute dry condition (0 % (m/m) moisture content) in the analytical zone. Alternatively, if agreed with the purchaser, the average density can be taken from the table in Annex B.

NOTE 1 When timber for treatment is sourced from a single forest area, it is sufficient to establish the average density for that source.

NOTE 2 1,2 is a correction factor applied because it has been found that toluene does not extract all of the creosote present in the treated timber, that some creosote does evaporate from the test samples, that creosote is lost on the tool when cutting the samples or on the surface of the stoppered glass bottle during storage of the samples.

7 Test report

The test report shall include at least the following information:

a) the number and date of this European Standard;

b) a description of the batch or charge of creosoted wood including its approximate size in cubic metres (m³) and the date of treatment;

c) the date of sampling for both penetration and retention requirements;

d) the sampling procedure followed based on EN 351-2;

e) the full identification of the test samples and details of the preparation for analysis;

f) the results of the determination of the penetration in millimetres (mm) and, if available, the pass/fail result for the penetration requirements (see Clause 5);

g) the results of the determination of the retention of creosote in the analytical zone (see Clause 6) expressed to the nearest 5 kg/m^3 and, if available, the retention requirement;

h) any particular points observed in the course of the sampling procedure or of the test;

i) any operations not specified in the method or regarded as optional which might have affected the procedure.

Annex A
(informative)

Sapwood/heartwood boundary determination

A.1 Scope

This annex gives procedures for determining the sapwood/heartwood boundaries for pines and for Douglas fir.

A.2 Pines

A.2.1 Reagents

A.2.1.1 2-methoxyaniline solution.

Weigh 8,5 g concentrated hydrochloric acid (37 % (m/m)). Dilute with water to make 495 g of solution. Add 5 g of 2-methoxyaniline ($CH_3OC_6H_4NH_2$), and stir until dissolved completely.

A.2.1.2 Sodium nitrite solution. Dissolve 50 g sodium nitrite ($NaNO_2$), in 450 ml of water.

NOTE For maximum shelf life, both solutions A.2.1.1 and A.2.1.2 should be stored in a refrigerator or other cool, dark location. Under such conditions the storage life exceeds one month.

CAUTION — Given the concerns about the health effect of aromatic amines, it is essential that the operations are carried out in a fume cupboard.

A.2.2 Procedure

Mix the solutions of 2-methoxyaniline (A.2.1.1) and of sodium nitrite (A.2.1.2) in equal volumes.

NOTE 1 The mix of the two solutions can be used over a period of several days, although stored solutions should be filtered before use.

Apply the mix by dipping, brushing or spraying to the surface of the wood.

CAUTION — Given the concerns about the health effects of aromatic amines, if spraying is selected, it is essential that it is carried out in a fume cupboard.

Generally after several minutes, the heartwood will develop a bright red or reddish-orange colour whilst the sapwood remains a pale yellow orange colour. In some instances, however, the intensity of the red and reddish-orange colour can vary and the colour development can take longer than several minutes.

NOTE 2 Smooth surfaces should give better results than rough surfaces.

A.3 Douglas fir

A.3.1 Reagents

A.3.1.1 Methyl orange (4-[4-dimethylamino)phenylazo] benzene sulfonic acid, sodium salt, CI 13025), $(CH_3)_2\ NC_6H_4N = NC_6H_4SO_3Na$, 1 g/l solution in water.

A.3.1.2 Alizarine red S (sodium alizarinsulfonate, 3,4-dihydroxy-9, 10-dioxo-2-anthracene-sulfonic acid, sodium salt, CI 58005), $C_{14}H_6Na0_7S$, 7,5 g/l solution in water.

A.3.2 Procedure

A.3.2.1 Apply the methyl orange solution (A.3.1.1) to the wood surface. Generally after a few minutes the heartwood becomes reddish and the sapwood yellowish. These colours are comparatively permanent.

A.3.2.2 Apply the alizarine red S solution (A.3.1.2) to the wood surface. Generally after a few minutes the heartwood and one or two adjacent annual rings in the sapwood become yellow, the sapwood pink, or some other shade of red.

NOTE 1 Both tests work best on freshly exposed surfaces.

NOTE 2 Smooth surfaces should give better results than rough surfaces.

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Annex B
(normative)

Density of some selected timber species that are commonly treated with creosote

Scientific name	Common name	Density at 0 % (m/m) moisture content kg/m^3
Pinus sylvestris	Scots pine - Redwood	460
Pseudotsuga menziesii	Douglas fir, cultivated in Europe	450
Picea abies	Norway spruce	400
Fagus sylvatica	European beech	625
Quercus robur	European oak	625

NOTE These values are derived by taking the mean values for the density, given in EN 350-2, corrected for 0 % (m/m) moisture content, in kilograms per cubic metre (kg/m³).

Bibliography

[1] EN 350-2, *Durability of wood and wood-based products — Natural durability of solid wood — Part 2: Guide to natural durability and treatability of selected wood species of importance in Europe*

[2] EN 1001-2:2005, *Durability of wood and wood-based products — Terminology — Part 2: Vocabulary*

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