SLOVENSKI STANDARD
SIST-TS ENV 807:2004

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Zaġļitna sredstva za les ï Ugotavljanje uļinkovitosti zaġļitnega sredstva proti glivam razkrojevalkam lesa

Wood preservatives - Determination of the effectiveness against soft rotting micro-fungi and other soil inhabiting micro-organisms

Holzschutzmittel - Prüfverfahren für die Bestimmung der Grenze der Wirksamkeit gegen Moderfäule und andere erdbewohnende Mikroorganismen

Produits de préservation du bois - Détermination de l'efficacité vis-a-vis des micro-organismes de pourriture molle et d'autres micro-organismes du sol

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71.100.50 Kemikalije za zaġļito lesa Wood-protecting chemicals

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Wood preservatives - Determination of the effectiveness against soft rotting micro-fungi and other soil inhabiting micro-organisms

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This European Prestandard (ENV) was approved by CEN on 1 March 2001 as a prospective standard for provisional application.

The period of validity of this ENV is limited initially to three years. After two years the members of CEN will be requested to submit their comments, particularly on the question whether the ENV can be converted into a European Standard.

CEN members are required to announce the existence of this ENV in the same way as for an EN and to make the ENV available promptly at national level in an appropriate form. It is permissible to keep conflicting national standards in force (in parallel to the ENV) until the final decision about the possible conversion of the ENV into an EN is reached.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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Contents

Foreword

This European Prestandard has been prepared by Technical Committee CEN/TC 38, "Durability of wood and derived materials", the secretariat of which is held by AFNOR.

This European Prestandard supersedes ENV 807:1993.

The annexes A, B, C, D, E and F are informative.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to announce this European Prestandard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Introduction

This European Prestandard specifies a laboratory method of test which gives a basis for assessing the effectiveness of a wood preservative against micro-fungi (ascomycetes and fungi imperfecti) which cause soft rot of wood in service. The infection source is the natural micro-flora of the soil which may also contain other micro-organisms, such as bacteria and other fungi, such as moulds and basidiomycetes. This laboratory method provides one criterion by which the value of a wood preservative product can be assessed. This information has to be supplemented by data from other relevant tests and from practical experience.

1 Scope

This European Prestandard specifies a method of test for determining the toxic effectiveness of a wood preservative, applied to wood by full impregnation, against the micro-fungi which cause soft rot of wood.

The method is applicable to testing of formulated products or of their active ingredients.

NOTE A method suitable for undertaking screening tests of potential active ingredients is given in annex A.

2 Normative references

This European Prestandard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Prestandard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies (including amendments).

EN 84, *Wood preservatives — Accelerated ageing of treated wood prior to biological testing — Leaching procedure*

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

3 Terms and definitions

For the purposes of this European Prestandard, the following terms and definitions apply:

3.1
representative sample
sample having its physical or chemical characteristics identical to the volumetric average characteristics of the total volume being sampled

3.2
supplier
sponsor of the test

4 Principle

A number of small test specimens (as small stakes) are impregnated with the preservative under test at a minimum of three concentrations ranged about the retention expected to provide protection throughout the test period. The test specimens are exposed to leaching according to EN 84. The specimens are partly buried vertically in a microbially active soil. Sets of test specimens are assessed after 8, 16, 24 and 32 weeks of exposure. The performance of the test preservative is evaluated by comparison with the performance of a reference preservative.

5 Test materials

5.1 Biological materials

5.1.1 Soil

Natural top soil or a fertile loam-based horticultural soil [1)] of pH 6 to pH 8 and not containing added agro-chemicals. It shall have a waterholding capacity (WHC) of between 25 % (*m/m)* and 60 % (*m/m*).

NOTE 1 A suitable method for determining WHC is described in annex B.

[1)] A horticultural soil of the John Innes No.2 type and with the following composition has been found to be suitable; seven parts by volume loam, three parts by volume sphagnum peat, two parts by volume sharp sand plus 0,6 g chalk and 6,0 g slow release fertilizer per litre of soil mixture. If the WHC is too high, it can be lowered by modifying the soil with the addition of sand.

If a natural soil is used, it shall have the turf or top 50 mm removed and shall not be taken from a depth below 200 mm from the original surface. It shall be passed through a sieve of nominal aperture size 12,5 mm. If it is necessary to store the soil prior to use, it shall be stored in closed moisture-proof containers. Before use, thoroughly mix the sample of soil.

NOTE 2 The soil should only be collected in a moist condition.

If a horticultural soil is used which is sterilized during its preparation, then 20 % (*m/m*) of a natural soil, prepared as above, shall be added and the soils thoroughly mixed prior to the start of the test.

The soil shall be used only once.

NOTE 3 If assurance of the virulence of the soil is required, the test procedure using cotton cloth described in annex C, or a similar standardized procedure, may be used.

5.2 Products and reagents

5.2.1 Solvents and diluents

Water to grade 3 of EN ISO 3696 and, if appropriate, volatile organic liquids leaving in the wood no residue which would have a toxic effect on the soil inhabiting micro-organisms at the end of the post-treatment conditioning period.

NOTE Information on appropriate solvents and diluents should be provided by the supplier.

5.2.2 Reference preservative

A copper/chromium preservative with a composition equivalent to the following :

$CuSO_4 \cdot 5H_20$	50,0 % (*m/m*)
$K_2Cr_20_7$	48,0 % (*m/m*)
CrO_3	2,0 % (*m/m*)

The preservative shall be prepared from ingredients of at least 95 % (*m/m*) purity.

5.2.3 Xylene

($C_6H_4(CH_3)_2$) mixed isomers, technical grade.

5.3 Apparatus

5.3.1 Conditioning chamber, well ventilated and maintained at (20 ± 2) °C and (65 ± 5) % r.h.

5.3.2 Ventilated drying oven, capable of being maintained at (103 ± 2) °C.

5.3.3 Desiccators, with efficient desiccant (silica gel for example).

5.3.4 Treatment vessels, of a material that does not react with either the preservative or solvents or diluents, for example of glass for organic products and plastics materials for salts containing fluorine.

5.3.5 Weights, of a material that does not react with the preservative solutions under test, to provide ballast for the test specimens.

5.3.6 Plastics mesh, of a material that does not react with the preservative solutions under test, for retaining test specimens during impregnation.

5.3.7 Vacuum vessels, fitted with stopcocks.

5.3.8 Vacuum pump, fitted with a pressure gauge and capable of maintaining a pressure of 0,7 kPa.

5.3.9 Drying vessels, provided with a cover and containing supports which will give a minimum of contact with the treated test specimens which are to be placed on them. The vessels and supports shall be of a material that does not react with the test solvent or test preservative, for example glass for organic products or of plastic material for salts containing fluorine.

5.3.10 Culture chamber (incubator or room), dark and maintained at (27 ± 2) °C and (70 ± 5) % r.h.

5.3.11 Vacuum filtration apparatus, comprising vacuum flask, 146 mm diameter Buchner funnel and fitting coarse grade filter papers.

5.3.12 Test containers, made of material which does not have a toxic effect on the soil inhabiting micro-organisms and provided with a ventilated lid. The depth shall be at least 150 mm, so as to provide at least 30 mm below the test specimens when inserted in the soil to a depth of 80 mm and adequate clearance above the top of the protruding parts of the test specimens.

NOTE The exact dimensions are not critical but they determine the number of test specimens in each vessel (which should not be less than 10). An example of a suitable test container is described in annex D.

5.3.13 Safety equipment and protective clothing, appropriate for the test product, test solvent and reference preservative, to ensure the safety of the operator.

5.3.14 Ordinary laboratory equipment, including a balance accurate to 0,001 g.

6 Sample of the preservative

The sample of preservative shall be representative of the product to be tested. Samples shall be stored and handled in accordance with any written recommendations from the supplier.

NOTE For the sampling of preservatives from bulk supplies, the procedure given in EN 212 should be used.

7 Test specimens

7.1 Species of wood

The following species shall be used for the test :

— Scots pine (*Pinus sylvestris Linnaeus*) for products intended to be used on softwoods ;

— beech (*Fagus sylvatica Linnaeus*) for products intended to be used on hardwoods.

NOTE Additional tests may be carried out using other species but, if so, this should be stated in the test report.

7.2 Wood quality

The wood shall be free from cracks, stain, decay, insect damage and other defects. The wood shall not have been water-stored, floated, chemically treated or steamed.

NOTE Wood that has been kiln dried at temperatures below 60 °C may be used.

The Scots pine shall be exclusively sapwood containing little resin and having between 2,5 annual growth rings per 10 mm and eight annual growth rings per 10 mm. The proportion of latewood in the annual rings shall not exceed 30 % of the whole.

The beech shall be even-grained, free from tyloses and discolouration. It shall have between two annual growth rings per 10 mm and six annual growth rings per 10 mm.

7.3 Provision of test specimens

Condition the wood to (12 ± 2) % (*m/m*) moisture content. Prepare planed strips having a cross-section of (10 ± 0,1) mm × (5 ± 0,1) mm. The longitudinal faces shall be parallel to the direction of the grain. The annual rings

shall have a contact angle of (90 ± 15) ° to the broad faces. Make transverse cuts, neatly to give sharp edges and a fine-sawn finish to the end-grain surfaces, to give test specimens (100 ± 1) mm long.

The specimens shall originate from a minimum of three trees or shall be taken from a stock of more than 500 specimens and originating from at least five planks.

NOTE A moisture meter of the two pronged electrical conductivity type is suitable for assessing moisture content.

7.4 Dimensions and density of specimens

The dimensions of each test specimen at (12 ± 2) % (*m/m*) moisture content shall be (100 ± 1) mm × (10 ± 0,1) mm × (5 ± 0,1) mm.

For the purposes of calculating the density of the specimens (8.1.1) and the mass of preservative retained per unit volume of wood (8.1.3), the nominal volume of each test specimen shall be taken as 5,0 cm^3.

In any batch of specimens, the mass of an individual is permitted to differ from the mean value of the batch by ± 10(*m/m*).

7.5 Number and distribution of test specimens

The test specimens are divided into :

s_1 treated test specimens.

$s_{1.1}$ test specimens treated with the test preservative: these are impregnated with the solutions of the test preservative (clause 6) and subjected to attack by the micro-organisms in the soil. Use at least six test specimens for each combination of test preservative concentration, species of wood and exposure period (8.3.1).

$s_{1.2}$ test specimens treated with the reference preservative: these specimens are impregnated with the solutions of the reference preservative (5.2.2) and subjected to attack by the micro-organisms in the soil. Use at least six test specimens for each combination of reference preservative concentration, species of wood and exposure period.

s_2 untreated test specimens.

$s_{2.1}$ virulence control specimens : these specimens are not treated, they are of the same species of wood as the treated test specimens, and are subjected to attack by the micro-organisms in the soil. They are used to provide a measure of comparability between tests. Use three virulence control specimens for each test container.

NOTE 1 The virulence control specimens are assessed after 16 weeks exposure.

$s_{2.2}$ moisture monitoring specimens : these specimens are not treated, they are of the same wood species as the treated test specimens and are planted in the soil to assess that the moisture content level established in the test specimens is adequate to support active fungal attack. Use three moisture monitoring specimens for each test container.

NOTE 2 If the test is to be carried out in eight test containers of the type described in annex D (four containers for Scots pine specimens and four containers for beech specimens, (see 8.2.2), each of these containers would require three replicates of the virulence control specimens and three replicates of the moisture monitoring specimens of the appropriate species of wood; this gives a total of 12 virulence control specimens and 12 moisture monitoring specimens per species of wood. With smaller test containers, lower numbers of replicates are acceptable but each test container should contain at least one replicate virulence control specimen and one moisture monitoring specimen.

s_3 treated check test specimens for calculation of the correction values.

$s_{3.1}$ check test specimens treated with the test preservative: these are test specimens treated in exactly the same way as the $s_{1.1}$ test specimens, except that after drying, conditioning and leaching, they are allowed to dry fully and are not planted in the soil. Use at least four specimens for each combination

of tests preservative concentration and species of wood. Variations in the mass of these specimens make it possible to determine the correction value (C_1) for the variations in mass of the treated test specimens $s_{1.1}$, resulting from factors other than attack by the soil inhabiting micro-organisms. At a given treating solution concentration, the correction value C_1 is the mean percentage change in mass of the $s_{3.1}$ test specimens.

$s_{3.2}$ check test specimens treated with the reference preservative: these are test specimens treated in exactly the same way as the $s_{1.2}$ test specimens, except that after drying, conditioning and leaching, they are allowed to dry fully and are not planted in the soil. Use at least four specimens for each combination of reference preservative concentration and species of wood. Variations in the mass of these specimens make it possible to determine the correction value (C_2) for the variations in mass of the reference preservative treated test specimens $s_{1.2}$ resulting from factors other than attack by the soil inhabiting micro-organisms. At a given treating solution concentration, the correction value C_2 is the mean percentage change in mass of the $s_{3.2}$ test specimens.

Mark each test specimen so that it can be identified throughout the test.

NOTE 3 It is advisable to treat more specimens than the minimum number required to allow the rejection of specimens having more than the permitted variation in the quantity of product absorbed (8.1.3).

8 Procedures

8.1 Preparation of test specimens

8.1.1 Conditioning of specimens before treatment

Place the numbered test specimens in the oven (5.3.2) and leave them there for 18 h to 24 h [2]. Cool to room temperature in a desiccator (5.3.3) and weigh to the nearest 0,001 g to determine the initial dry mass (m_0). Replace the test specimens in the desiccator and store them there in order to keep them dry until impregnation. Calculate the mean density of the specimens of each species using the mean mass and the nominal volume (see 7.4).

8.1.2 Preparation of treatment solutions

Prepare a series of solutions of at least three concentrations (expressed as % (m/m)) of the test preservative (clause 6) in the appropriate solvent or diluent (5.2.1). A solvent or diluent control, that is treatment at concentration 0, shall also be included.

NOTE 1 It is preferable to use at least five concentrations of the test preservative except when there is prior experience of the performance of the test preservative in the test. It is normal for the treating solution concentrations to be arranged in a geometric or arithmetic progression.

NOTE 2 The selection of the treating solution concentrations (and therefore retentions) of the test preservative should be made giving consideration to the performance of the reference preservative in the test and the method of calculation of the results (see annex E). It is also necessary to include a retention of the test preservative which fails in the test before the reference preservative fails, to be able to calculate accurately the nominal effective retention of the test preservative (see clause 10). With beech test specimens, the retentions in the test specimens will normally be ranged about the likely retention to be effective in practice. However, with Scots pine sapwood test specimens, the retentions in the test specimens should be over a range which is much lower than the likely retention to be effective in practice. This is because of the good performance of wood preservatives in this species of wood in laboratory tests and is exemplified by the selection of the treating solution concentrations for the reference preservative.

Prepare the reference preservative (5.2.2) at the following concentrations in water :

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[2] In the case of supplementary tests (7.1) using species of wood other than Scots pine sapwood or beech, this drying time may need to be longer than 18 h to 24 h; the drying time should be such that the test specimens achieve constant mass. This can be established by selecting at random from the batch being dried 10 test specimens; after drying and cooling as directed, determine the total mass, return the specimens to the oven and repeat the operation at intervals of not less than 4 h ; constant mass is achieved when the total mass of the selected specimens does not lose more than 0,05 g between weighings.

— Scots pine: 0,1 % - 0,16 % - 0,25 % - 0,4 % (m/m) ;

— beech: 1,0 % - 1,6 % - 2,5 % - 4,0 % (m/m).

All treatment solutions shall be freshly prepared.

8.1.3 Impregnation

Carry out impregnation of the sets of test specimens with the test preservative solutions in ascending order of concentration starting with the solvent control (concentration = 0). Using clean equipment, impregnate the appropriate sets of test specimens with the reference preservative, again in ascending order of concentration.

The following procedure ensures the required complete impregnation of test specimens by the test solutions.

For each solution place the test specimens, kept dry as described in 8.1.1 and of known mass m_0, in one of the treatment vessels (5.3.4) so that as much of their surface as possible is exposed (for example, by stacking them crosswise). Ballast the stack of specimens with the weights (5.3.5) using the plastics mesh (5.3.6) if necessary, to prevent them floating when the liquid is admitted.

Place each treatment vessel in one of the vacuum vessels (5.3.7) and after reducing the pressure to 0,7 kPa, using the vacuum pump (5.3.8), hold it at this pressure for 15 min. After this period, close the stopcock to the vacuum pump and open the stopcock to allow the solution of preservative to be drawn into the treatment vessel within the vacuum vessel until it completely covers the test specimens. Keep the specimens covered completely by the solution throughout the remainder of the impregnation process.

Next, admit air slowly to bring the vacuum vessel back to atmospheric pressure, remove the treatment vessel with its submerged specimens from the vacuum vessel. Cover the top of the treatment vessel and leave it for 2 h, adding further solution if necessary to keep the specimens fully covered by the liquid.

After impregnation, remove the test specimens one by one from the treatment vessel and remove excess liquid from them by lightly blotting with absorbent paper. Immediately weigh each to the nearest 0,001 g to ascertain the mass after impregnation (m_1).

In the case of preservatives which are being studied as active ingredients, calculate the mass of preservative retained for each test specimen, from the mass of solution absorbed (m_1 - m_0) and its concentration [3)].

In the case of formulated wood preservatives, express the retention for each test specimen in terms of the ready-to-use product, and, for products supplied in the form of a concentrate, in terms of the product as supplied.

NOTE If the product has been supplied as a concentrate for dilution prior to use, the nominal effective concentration established (see clause 10) will need to be equivalent to the product as marketed, to provide the data in the correct form for use in EN 599-1.

Calculate the mass of active ingredient or formulation retained per unit volume of wood, in kilograms per cubic metre, for each test specimen from the retention of product and volume of the test specimens (7.4) and the mean value for each simultaneously impregnated group of test specimens.

Reject those test specimens in which the quantity of product absorbed varies by more than 15 % from the mean absorption of the group. Replace them with supplementary specimens and calculate a new mean.

8.1.4 Drying and conditioning of specimens after treatment

NOTE The procedures described below are usually applicable, but if the nature of the test preservative is such that alternative procedures are required, details of the procedure used should be included in the test report.

Keep the test specimens for four weeks in the conditioning chamber (5.3.1). Arrange the test specimens in the drying vessels (5.3.9), resting on their narrow faces on the supports, and placing only specimens treated with the

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[3)] When dealing with preservative formulations whose constituents are absorbed selectively by the wood, it may be necessary to carry out chemical analysis of the solution before and after impregnation. Similarly, analysis is recommended when very dilute solutions are used.

same concentration of the test or reference preservative in the same drying vessel; avoid contact between specimens. Invert the test specimens twice a week at intervals of three or four days.

In the case of test specimens impregnated using water as the solvent or diluent, keep the vessels covered for two weeks. To prevent mould growth, also place in each vessel a small dish containing xylene (5.2.3). During the third week, uncover each vessel progressively each day to allow the specimens to dry steadily. From the beginning of the fourth week, leave the vessels completely open.

In the case of test specimens impregnated using a volatile organic liquid as the solvent or diluent, keep each vessel covered for one week. Open each vessel gradually during the second week and finally leave them open during the third and fourth weeks.

8.1.5 Leaching procedure

Subject all treated test specimens and treated check test specimens ($s_{1.1}$, $s_{1.2}$, $s_{3.1}$ and $s_{3.2}$) to the procedure described in EN 84.

Stop the drying stage of EN 84 when all the test specimens have reached a moisture content of (50 ± 5) % (*m/m*). If the test specimens become too dry, they shall be rewetted by a short soak in water.

NOTE The moisture content should be checked by periodic weighing of a minimum of 10 specimens taken at random during the drying period and comparing their mass with their initial dry mass (m_0), making an allowance for the mass of preservative retained. Drying to 50 % moisture content is likely to occur within 24 h.

8.1.6 Wetting of untreated specimens

Impregnate the virulence control specimens ($s_{2.1}$) and the moisture monitoring specimens ($s_{2.2}$) with water, using the method described in EN 84. Allow to soak for 2 h, then lay to dry. Continue with the drying stage of EN 84, stopping this when the test specimens have reached a moisture content (50 ± 5) % (*m/m*).

NOTE This impregnation procedure should be timed to coincide with the end of the leaching period of the treated specimens (8.1.5) in order that drying of all the test specimens is undertaken at the same time.

8.2 Exposure of the test specimens to soil inhabiting micro-organisms

8.2.1 Preparation of test containers

Determine the mass of soil required to provide at least 120 mm depth of soil in a selected test container. Determine the moisture content and water holding capacity (WHC) of the soil (5.1.1). Calculate the amount of water required to bring the soil in the fully charged container to 95 % of its WHC.

NOTE A suitable procedure for determining WHC and the quantity of water required to wet up the soil is described in annex B.

Add the required volume of soil to each test container and add the calculated amount of water slowly whilst thoroughly mixing to ensure an even distribution of moisture.

8.2.2 Planting the test specimens

Plant the Scots pine and beech test specimens in different containers. Plant the treated and untreated test specimens s_1 and s_2 vertically with 20 mm of their length protruding above the surface of the soil and with a minimum of 20 mm between adjacent specimens and from the sides of the container. Assign the correct number of virulence control specimens $s_{2.1}$ and moisture monitoring specimens $s_{2.2}$ (7.5.2) to each test container and distribute them at random. Assign the positions of the test specimens treated with the test preservative $s_{1.1}$ and the reference preservative $s_{1.2}$ at random among all the test vessels being used for the appropriate species of wood. During planting and subsequent handling ensure the exact location of each specimen is recorded to guard against loss of identity if the numbering is obscured. Apply a ventilated lid to each charged test container.

NOTE The test specimens of each species of wood are separated to reduce the number of untreated specimens (s_2) that are required and to allow separate adjustment to the moisture content of the test specimens (8.2.4) which may vary between wood species.