SLOVENSKI STANDARD
SIST EN 46:1995

01-november-1995

Zaščitna sredstva za les - Določanje preventivnega delovanja proti jajčnim ličinkam hišnega kozlička Hylotrupes bajulus (Linnaeus) - Laboratorijska metoda

Wood preservatives - Determination of the preventive action against recently hatched larvae of Hylotrupes bajulus (Linnaeus) (Laboratory method)

Holzschutzmittel - Bestimmung der vorbeugenden Wirkung gegenüber Eilarven von Hylotrupes bajulus (Linnaeus) (Laboratoriumsverfahren)

Produits de préservation des bois - Détermination de l'efficacité préventive contre les larves récemment écloses d'Hylotrupes bajulus (Linnaeus) (Méthode de laboratoire)

Ta slovenski standard je istoveten z: **EN 46:1988/AC:1992**

ICS:

71.100.50 Kemikalije za zaščito lesa Wood-protecting chemicals

SIST EN 46:1995 **en**

EUROPEAN STANDARD
NORME EUROPÉENNE
EUROPÄISCHE NORM

EN 46

November 1988

Supersedes EN 46, August 1976

UDC 674.048.4:620.193.87

Key words : Wood, Wood preservatives, Pesticides, Insecticides, Protection against the pest, Laboratory tests, Determination, Effectiveness, Prevention, Lyctus.

English version

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

Central Secretariat : Rue Bréderode 2, B-1000 Brussels

Ref.No. EN 46:1988 E

B R I E F H I S T O R Y

This European Standard was drawn up by the Technical Committee CEN/TC 38 "Methods of test for wood preservatives", the Secretariat of which is held by AFNOR.

According to the Common CEN/CENELEC Rules, following countries are bound to implement this European Standard :

Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxemburg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

Contents

0 Introduction

This European Standard specifies a laboratory method of test which gives a basis for the assessment of the preventive action of a wood preservative, when applied as a surface treatment, against *Hylotrupes bajulus*, whereas the method for determining the toxic values against *Hylotrupes bajulus* (EN 47) provides a means of checking whether a preservative prevents attack by these larvae and prevents their survival within totally impregnated wood.

This method makes it possible to determine whether recently hatched larvae are capable of boring through the treated surface of a susceptible wood species and of surviving in the untreated part of the test specimen. For this purpose, the procedure seeks to reproduce normal egg-laying conditions existing in cracks in wood, which provide the principal egg-laying sites. It takes account of the fact that, if larvae pass through the treated surface, they will then tunnel in the direction of the least protected regions of the wood.

This laboratory method provides one criterion by which the value of a preservative can be assessed. In making this assessment, the methods by which the preservative may be applied should be taken into account. This test is of particular interest when applied to test specimens which have been subjected to an ageing procedure. It is further recommended that results from this test should be supplemented by those from other appropriate tests and, above all, by practical experience.

When using products which are very active at very low concentration, it is of great importance that suitable precautions be taken to isolate and separate as far as possible operations involving chemical products, other products, treated wood, all clothing and laboratory apparatus. Suitable precautions shall include the use of separate rooms, areas within the rooms, extraction facilities, conditioning chambers and special personnel training.

1 Object

This European Standard specifies a method for the determination of the preventive action of a wood preservative against recently hatched larvae of *Hylotrupes bajulus* (Linnaeus) when the preservative is applied as a surface treatment to wood.

2 Field of application

This method is applicable to:

- water-insoluble chemicals which are being studied as active insecticides; or
- organic water-insoluble formulations, as supplied or as prepared in the laboratory by dilution of concentrates; or
- organic water-dispersible formulations as supplied or as prepared in the laboratory by dilution of concentrates; or
- water-soluble materials, for example salts.

The method is applicable whether or not the test specimens have been subjected to appropriate ageing procedures.

3 Principle

Depending on the test being carried out, surface treatment of one or more sets of test specimens of a susceptible wood species with the preservative or, if a concentrate is being used or toxic values sought, with known dilutions of the preservative.

Exposure of the treated test specimens to recently hatched larvae of *Hylotrupes bajulus* and comparison of the resulting attack with that in untreated control specimens and also with that in solvent- or diluent-treated control specimens if the preservative is a laboratory solution or dilution.

4 Test materials

4.1 Biological material

Hylotrupes bajulus (Linnaeus) larvae, within three days of hatching.

4.1.1 *Source of larvae.* Obtain the larvae from cultures reared as described in annex B.

4.1.2 *Provision of larvae.* Collect larvae from eggs laid by different females.

4.1.3 *Choice of larvae.* Use a mixed batch of these larvae for the test. Use 10 larvae per treated specimen or control specimen.

4.2 Products and reagents

4.2.1 *Pure paraffin wax*, setting point 52 °C to 53 °C, for fixing the glass plate in all cases and for sealing the end surfaces of test specimens to be treated with solutions in which water is the continuous phase.

4.2.2 *Pure gelatin*, for sealing the end surfaces of specimens to be treated with solutions in which an organic solvent is the continuous phase.

4.2.3 *Solvents and diluents* (if toxic values are being sought)

For water-soluble preservatives:

distilled or deionized water.

For preservatives to be diluted with or dissolved in an organic solvent:

suitable volatile liquids which leave no residue which would have a toxic effect on the insect[1] in the wood at the end of the post-treatment conditioning period.

4.3 Apparatus

4.3.1 *Culturing chamber*, with air circulation, and controlled at between 27 °C and 29 °C with a tolerance of ± 1 °C and at 85 ± 5 % r.h.

4.3.2 *Conditioning chamber*, well ventilated and controlled at 20 ± 2 °C and at 65 ± 5 % r.h.[2].

4.3.3 *Laboratory work area*, well ventilated, where treatment of the test specimens is carried out[3].

4.3.4 *Testing chamber*, ventilated and air conditioned, controlled at between 21 °C and 23 °C with a tolerance of ± 1 °C and at between 70 % and 75 % r.h. with a tolerance of ± 5 % r.h.

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[1] Do not use benzene as a solvent as it poses a health risk for those conducting the test.

[2] The conditioning of test specimens after treatment may be carried out in the laboratory work area (4.3.3) provided that this meets the conditions specified for the chamber (4.3.2).

[3] Proper safety measures for handling flammable or toxic materials shall be followed. Avoid excessive exposure to solvents or their vapours.

4.3.5 *Treatment vessels*, of a material that does not react with the preservative under test, for example of glass for organic products and of plastics materials for salts containing fluorine.

4.3.6 *Weights*, chemically inert, for ballasting the test specimens.

4.3.7 *Protective gloves.*

4.3.8 *Glass plates*, 48 mm long and 25 mm wide, intended to provide a lateral slit on the test specimens.

4.3.9 *Ordinary laboratory equipment*, including an analytical balance.

5 Sample of the preservative

The sample shall be representative of the product under test.

6 Test specimens

6.1 Species of wood

The reference species is Scots pine, *Pinus sylvestris* Linnaeus[4].

Additional tests may be made using other species but, if so, this shall be stated in the test report.

6.2 Quality of wood

Use only sound sapwood, straight-grained, without knots and with a low resin content.

Cut the test specimens from wood of average growth rate (2.5 to 8 annual rings per centimetre).

The proportion of summer wood in the annual rings shall not exceed 30 % of the whole.

The wood shall neither have been floated nor subjected to any chemical or heat treatment[5]. It shall be air-dried and shall not have been stored for more than five years.

6.3 Provision of test specimens[6]

Cut the test specimens from planed strips having a cross section 25 mm x 15 mm. The annual rings shall lie in a plane which is essentially parallel to the narrow faces measuring 50 mm x 15 mm and shall not deviate by more than 30° (see figure 1).

The transverse cross sections shall be cut cleanly and have sharp edges.

Avoid using test specimens from the butt or crown of the tree.

Take the specimens required for one test from three lots, each from a different tree, and at random from within each of these lots.

6.4 Dimensions of test specimens

The nominal dimensions of each specimen measured at 12 % (*m/m*) moisture content shall be 50 mm x 25 mm x 15 mm.

The total surface area of the longitudinal faces is theoretically 40 cm^2.

Check the size of each specimen to determine the actual area treated. Allow for any possible encroachment of the end sealing compound on to the faces of the specimen.

6.5 Number of test specimens[7]

Use:

(a) for each preservative, each concentration and each duration of treatment, six treated test specimens (two from each lot) (see **6.3**);

(b) for a complete test of any given preservative, three untreated control specimens (one from each lot) (see **6.3**);

(c) if a solvent or diluent (including water) is used, three control specimens treated with the solvent or the diluent (one from each lot) (see **6.3**).

7 Procedure

7.1 Preparation of the test specimens

7.1.1 *Conditioning of the test specimens prior to sealing.* Allow the test specimens to reach equilibrium in the conditioning chamber (**4.3.2**).

7.1.2 *Sealing of the transverse surfaces.* Seal the transverse surfaces as follows.

7.1.2.1 *For tests with solutions in which water is the continuous phase.* Use three coats of the pure paraffin wax (**4.2.1**) at about 100 °C, so that the first coat adheres closely to the wood and successive layers bond closely to one another.

7.1.2.2 *For tests with solutions in which an organic solvent is the continuous phase.* For tests with organic solvent-based preservatives, which would dissolve paraffin wax, use the pure gelatin (**4.2.2**). Apply the first coat with a 200 g/L aqueous solution at 40 °C, then, after a short period of drying, apply two further coats of 300 g/L gelatin in water at 50 °C. Condition once more in the conditioning chamber (**4.3.2**).

7.1.3 *Treatment of the test specimens*[8]

7.1.3.1 *Preparation of treatment solutions*

7.1.3.1.1 *Solid preservatives*

water-soluble preservatives: dissolve these in the distilled or deionized water (**4.2.3**) to the predetermined

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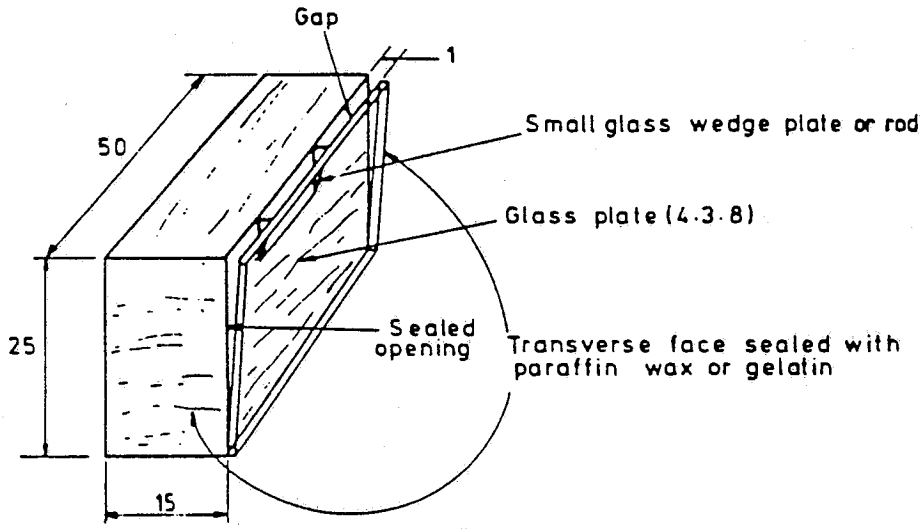
[4] In southern European countries the pine species most frequently infested by *Hylotrupes bajulus* may be used as an alternative, provided that the suitability of the species for use in the tests specified in this standard has been demonstrated in all aspects (development of larvae, resistance to impregnation, etc.).

[5] Gentle artificial drying at below 60 °C is, however, permissible.

[6] For special tests, specimens may be obtained according to a given series. As a result, it may be preferable to take specimens from pretreated strips.

[7] It is advisable to treat more than the specified number of test specimens so that any specimens with abnormally high or low retentions can be rejected from the batch.

[8] Apply, if necessary, treatment by pipette (see Annex D).



All dimensions in millimetres.

Figure 1. Test specimen fitted with its glass plate

concentration or to a series of concentrations if toxic values are being sought;

water-insoluble preservatives: dissolve these in an appropriate solvent as described in **4.2.3** to the intended concentration or to a series of concentrations if toxic values are being sought.

7.1.3.1.2 *Liquid preservatives.* If appropriate, use the preservative without further preparation other than any necessary stirring.

Otherwise, i.e. if it is a concentrate or if toxic values are being sought, dilute the preservative. Dilute any concentrate with the diluent to the required working concentration, as specified by the manufacturer.

If toxic values are being sought, prepare a series of at least five concentrations by mass, distributed about the expected toxic values. A solvent- or diluent-treated control specimen, i.e. treatment at concentration 0, shall also be used. If the approximate toxic values are unknown, the concentrations shall form a widely spaced geometric progression for a first test and a more closely spaced geometric or arithmetic progression for subsequent tests.

Treatment solutions shall be freshly prepared.

7.1.3.2 *Dipping.* Weigh each end-sealed specimen to the nearest 0.01 g to obtain its initial mass.

Immerse successively each test specimen in the solution, moving it in the solution during dipping.

The dipping times to be used shall be one of the following, agreed beforehand according to the purpose of the test:

either one 10 s period and/or two periods of 10 s at an interval of 24 h[9];

or a period sufficient for a determined quantity to be retained by the test specimen[10].

Using forceps, remove each test specimen from the liquid and, if necessary, sponge off any droplets remaining on the transverse sections. Then, still holding it with the forceps, turn the block in the air, bringing each surface uppermost until the preservative is completely absorbed by the wood.

Then weigh the specimen to the nearest 0.01 g. In the case of water-soluble preservatives, for example salts, and water-insoluble chemicals which are being studied as active substances, calculate the mass of preservative retained by each specimen from the mass of solution absorbed and its concentration.

In the case of organic water-insoluble formulations, the retention is expressed for each specimen in terms of the corresponding mass of formulation ready for use but, if a concentrate is supplied, the retention is expressed in terms of the solution prepared ready for use as specified by the manufacturer.

Calculate the mass of preservative retained per unit area of wood surface.

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[9] If the rate of solidification of some constituents of a preservative formulation would have the effect of retarding its penetration during the second dipping, this interval has to be reduced. The interval employed shall be mentioned in the test report.

[10] The dipping time depends upon the type of preservative and may extend to several hours for water-soluble preservatives. The progress of absorption is monitored by successive weighings of the treated specimens. For this long period dipping the treated specimens are immersed together and kept submerged by the weights (**4.3.6**).

7.1.4 *Drying and conditioning of the test specimens after treatment*[1]. If the end-sealing has been damaged before or during treatment, eliminate the test specimens concerned from the tests.

After treatment, condition the specimens for four weeks in the environment specified for the conditioning chamber (4.3.2). Arrange the specimens on their narrow faces, resting on two glass rods, not touching one another. Invert the specimens twice a week.

7.2 Exposure of the test specimens to the insects

Place one of the glass plates (4.3.8) against one of the wide faces of each test specimen.

At one of the 50 mm edges, insert a spacer 0.35 mm thick between the glass plate and a specimen so as to leave a gap 0.35 mm wide (see figure 1).

Fix the plate by dipping the transverse faces and the narrow longitudinal face opposite the wedge in the paraffin wax (4.2.1) kept close to melting point so as to seal the openings at the edge of these faces. After cooling remove the wedge.

Next place the 10 larvae in the gap thus provided in the middle part of the specimen.

7.3 Conditions and duration of the test

Place all the test specimens in the testing chamber (4.3.4), keeping the treated specimens separate from the untreated control specimens and the solvent- or diluent-treated control specimens, and also, if toxic values are being sought, keeping the different concentrations separate.

The total duration of the test, during which examinations and observations are carried out as described in 7.4.1, is 12 weeks.

7.4 Examination of the test specimens

7.4.1 *Examination.* Four weeks after the start of the test, carefully remove the glass plate and ascertain the tunnelling and mortality rate of the larvae. Those larvae which have tunnelled leave a small quantity of wood dust at the tunnel entrance. Any dead newly hatched larvae are completely dried up at the end of four weeks and are dark in colour.

At least 70 % of the larvae in contact with the control specimens shall have tunnelled, otherwise, stop the test and begin again.

If, after four weeks:

all the larvae are already dead on the surface of the treated test specimens and if the control specimens have been sufficiently attacked (see above), consider the test completed and determine the number of live larvae in the control specimens by cutting up the wood;

larvae have tunnelled in the treated test specimens and the control specimens have also been sufficiently attacked (see above), continue the test for a further eight weeks and then carry out a final examination of the test specimens by cutting them up at the end of the total of 12 weeks.

For tests with several concentrations:

the test at any concentration may be considered complete when, at the end of four weeks, all the larvae are dead on the surface of the set of treated test specimens for that concentration;

continue the test for a further eight weeks on those concentrations for which the treated test specimens contain larvae that have tunnelled as well as on the control specimens. Carry out a final examination by cutting up the wood at the end of the total of 12 weeks.

7.4.2 *Validity of the test.* The test shall be considered valid if at least 70 % of the larvae exposed to all of the untreated control specimens survive and, if applicable, at least 70 % of those exposed to all of the control specimens treated with the solvent or the diluent alone, survive. Otherwise, repeat the test.

8 Expression of results

8.1 Evaluation of attack

The extent of attack shall be evaluated in terms of:

the number of dead larvae which have not tunnelled;

the number of dead larvae which have tunnelled;

the number of live larvae and the state of these larvae;

the number of larvae not retrieved.

8.2 Toxic values

The toxic values of a preservative are expressed as the following two concentrations:

the lowest concentration at which, at the end of the test, all the larvae are dead;

the next, lower, concentration in the series at which live larvae are found.

Express these values as grams of preservative per square metre of treated wood surface and also state the corresponding concentrations of the preservative in the solvent or diluent.

9 Test report

The test report shall give the following.

(a) the number of this European Standard;

(b) the name of applicant;

(c) the name and type (see clause 2) of product test and whether the formulation has been disclosed;

(d) any solvent or diluent that may have been used;

(e) the species of wood used;

(f) if applicable, the concentration(s), as percentages by mass, of preservative tested;

(g) the method, the date and the conditions of treatment;

(h) for each test specimen (or each strip when the specimens are taken from pretreated strips):

if dipping
- the mass, in grams, of solution absorbed;
- the corresponding mass of test product per unit surface area, in grams per square metre;

if treating by pipette
- the volume of solution applied to the treated surface of the specimen (or, where necessary, the volume of solvent or diluent in millilitres;
- the corresponding amount of the product under test in grams per square metre or millilitres per square metre;

[1] Drying and conditioning of the specimens depend on the nature of the preservative under test and on the solvent or diluent used. It may be necessary to modify the conditions specified but, if so, this should be stated in the test report.