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Trajnost lesa in lesnih proizvodov ï Terenski preskusi in preskusi s pospeġenim kondicioniranjem (FACT) zaġļitnih sredstvev za les brez stika z zemljo

Durability of wood and wood-based products - Field and accelerated conditioning tests (FACT) for wood preservative out of ground contact

Dauerhauftigkeit von Holz und Holzwerkstoffen - Freiland- und beschleunigte Alterungsprüfverfahren (FACT)

Durabilité du bois et des matériaux dérivés du bois - Essais de champ et de conditionnement accéléré (FACT) pour les produits de préservation du bois hors contact du sol

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Dauerhauftigkeit von Holz und Holzwerkstoffen – Freiland- und beschleunigte Alterungsprüfverfahren (FACT)

This Technical Report was approved by CEN on 26 November 2003. It has been drawn up by the Technical Committee CEN/TC 38.

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Contents

Foreword

This document (CEN/TR 14723:2003) has been prepared by Technical Committee CEN/TC 38 "Durability of wood and wood-based products", the secretariat of which is held by AFNOR.

This status of this document as Technical Report has been chosen because its content is the result of the co-normative research as field tests for wood preservatives out of ground contact in conjunction with methods for preconditioning test specimens prior to test.

This co-normative research has been undertaken in the framework of the SMT Project: F.A.C.T SMT4 CT96 2135. (Co-ordinators: Dr D.J. DICKINSON and Dr S. MOLNAR from Imperial College of Science, Technology and Medicine (London)).

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

1 Scope

This Technical Report presents a summary of the research of the S.M.T. Project: F.A.C.T, SMT4 CT96 2135. As such it details the supporting information for the proposals for a new conditioning standard to replace EN 73 and EN 84 and for modification and validation of ENV 12037. The full report is presented and referenced to Technical annexes, which are available on request.

The FACT project involved the study of the fate of chosen wood preservatives in field and laboratory tests and the affect on biological performance. The work consisted of four core tasks and associated sub-tasks designed to provide the scientific background to the proposals made.

— **Task 1**

This consisted of a study of artificial weathering techniques for the conditioning of treated test blocks prior to biological test. It was originally hoped that it might be possible to relate different weathering cycles to the different climatic conditions found within Europe. However this did not prove possible but it is possible to recommend a single weathering cycle to replace the use of EN 73 and EN 84. A draft experimental standard has been prepared based on the findings of this work.

All the supporting data and associated studies are summarised in this Technical report in clauses 5 and 6.

— **Task 2**

This task consisted of an extensive field test based on ENV 12037 and associated sub-tasks. The method proved very robust but significant modifications are recommended and the ENV is to be redrafted in the light of results contained in this report. This field test should be considered as 'simulated service trial' rather than an accelerated field test; a concept that needs careful consideration within the EN 599 framework.

Sub-tasks 2.3 and 2.4 examined the suitability of the method for composites and the testing of naturally durable timbers. At this stage it is too early to make recommendation for their inclusion in the standard but the results look promising and the method will probably be modified at a later revision.

— **Tasks 3 and 4**

These tasks examined the role of bio-conditioning of test samples in 'use class' 3 and 4. The results are presented in clauses 8 and 9 and clearly indicate the importance of this subject. It is recommended that urgent attention is given to taking this work further particularly for the testing of organic based preservatives. Although not yet ready for standardisation, the results presented here will prove invaluable to industry in designing development programmes for the new generation of organic based preservatives.

In summary:

— a new experimental standard will be produced to replace EN 73 and EN 84 for the physical conditioning of test blocks;

— ENV 12037 will be modified to take into account the extensive findings of the project;

— the use of ENV 12037 for natural durability and composite testing will be considered when more results are available;

— further work on biological conditions is urgently recommended.

2 Acronyms

a.i	Active ingredient (biocide)
EUC	European Use Class
HDO	N-Cyclohexyldiazeniumdioxid
UC	use class
ICP	induction coupled plasma atomic emission spectroscopy
m.c or MC	moisture content
m/m	mass/mass
nr of reps	number of replicates
OSB	oriented strand board
QUV	UV artificial weathering machine manufactured by Q-panel Co. Cleveland, Ohio, USA
r.h	relative humidity
SD	standard deviation
TM	trademark
TBTO/TnBTO	tri n-butyltin oxide
UVS-Cabinet	cabinet with UV-light and spray option for artificial weathering of treated wood
WHC	water holding capacity
WMC	wood moisture content in %

3 Description of the project, background and objectives

CEN TC38 WG25 (ex WG 5) has clearly identified important gaps in the test methods necessary for testing wood preservatives. There is an urgent need for new wood preservatives, particularly in the above ground, exposed situation, (Use Class 3). No suitable field test exists for this use class for unpainted timber. Also current pre-conditioning systems are inadequate and in some cases are not relevant to certain use classes or industry's needs. There exists therefore, a need to establish a suitable field test and a single progressive, preconditioning system suitable for each of the use classes in which construction timbers are exposed. The development of new, environmentally safe wood preservatives is urgently needed as the pressure on the older systems increases and the use of durable, tropical timbers become less acceptable.

In summary the objectives consist of:

a) to identify and quantify the role of leaching, light, hydrolysis and non-decay, biological factors in the long term performance of wood preservatives. These factors are to be related directly to the specific European Use Classes i.e.:

   — EUC 1 (Dry timber in building, subjected to insect attack);

   — EUC 2 (Timber in building subjected to occasional wetting and at risk from insects and brown rot fungi);

— EUC 3 (Timber exposed to the weather but out of ground contact and subject to insect and fungal decay from both white and brown rot fungi);

— EUC 4 (Timber in ground contact or fresh water and subject to all biological agencies of attack including soft-rot fungi);

The important factor studied will be related directly to the fate of the preservative in the field and to the existing biological test relevant to each use class.

b) to study accelerated ageing procedures and biological preconditioning procedures capable of reproducing the ageing effects occurring in practice. This will be done by studying the losses and changes of chosen preservatives in comparison to field data. This will then enable schedules and procedures to be defined in order to simulate natural long term exposure of preservative treated wood;

c) to prepare a technical case to recommend specific ageing and preconditioning cycles suitable to be used as standard laboratory procedures for accelerated ageing of treated wood samples, prior to biological test.

4 Project contributors and roles

P1.Imperial College of Science, Technology and Medicine (IMPCOL) UK

Partner 1 (IMPCOL) was responsible for co-ordinating the project. They acted as a site for the field test in order to provide identical conditions for their work in Task 3 concerned with bio-preconditioning. They were also responsible for studying bio-conditioning in EUC 4. Both these tasks were long term and fed directly from the work of the other participants. (Core tasks 2, 3 and 4; Sub-task 2.5).

P2. Building Research Establishment (BRE) UK

Partner 2 (BRE) was responsible for carrying out the fundamental laboratory work on the stability of insecticides in EUC 1 and 2. This work helped identify the true role of associated factors in the loss of efficacy and underpin the laboratory weathering trials. (Sub-tasks 1.1 and 2.5).

P3. TNO. Building Construction Research (T.N.O) Netherlands

Partner 3 (TNO) took responsibility for the laboratory weathering trials with TnBTO and also for the chemical analysis of this preservative in the field trials. They exposed superficially treated samples in the lap-joint trial and also studied natural durability of imported timbers in the lap-joint trial. They also co-operated with P15 on M/C studies.(Core tasks 1 (b) and 2; Sub-tasks 2.1, 2.4 and 2.6).

P4. Bundesanstalt für Materialforschung und -prüfung (BAM) Germany

Partner 4 (BAM) was a key partner in laboratory weathering trials. They assumed responsibility for the organo-metallic preservative in both the laboratory and field trials in co-operation with their industrial partner (Wolman). They also exposed superficially treated lap-joints and conducted ecological studies on these joints. They also compared the laboratory ageing procedures to the German RAL tests in an associated trial funded nationally. They also carried out bio-preconditioning trials with superficially treated timber. Partner 4 co-ordinated the field trials.(Core tasks 1 (b), 1.2 and 2; Sub-tasks 2.1, 2.2 and 3.1).

P5. Universiteit Ghent (RUG) Belgium

Partner 5 (RUG) was responsible for the laboratory trials and field trials with regard to the triazole preservative. This was in co-operation with their industrial partner, Janssen.(Core tasks 1 (b) and 2; Sub-task 2.1).

P6. Centre Technique du Bois et de l'Ameublement (CTBA) France

Partner 6 (CTBA) exposed field trials in two sites with impregnated joints. They will also carried out mild leaching procedures for EUC 2. (Core task 2; Sub-task 2.7).

P7. Swedish University of Agricultural Sciences. Department of Wood Science (Formerly: Department of Forest Products). (SLU)Sweden

Partner 7 (SLU) exposed impregnated lap-joints and carried out ecological studies of the joints. They also exposed natural durability trials in parallel. SLU also co-operated with P1 in the bio-preconditioning work in EUC 4, where they provided the essential field data from their field sites. (Core tasks 2 and 4; Sub -tasks 2.2 and 2.4).

P8. Janssen Pharmaceutica (Janssen) Belgium

Partner 8 (Janssen) provided the technical knowledge on the triazole preservative and co-operated with P5 (RUG) in this respect. (Core tasks 1 (b), 2 and 2.1)

P9. Borax Europe (Borax) UK

Partner 9 (BORAX) was responsible for carrying out a lap-joint test with borates in order to provide the data on early losses of soluble preservatives intended for EUC 2, but exposed to weathering during construction. (Sub-task 2.7).

P10. Holzforschung Austria (formerly: Austrian Forest Products Laboratory) (HFA) Austria

Partner 10 (AHF1) exposed superficially treated lap-joints and ran a parallel trial with Spruce joints which they funded locally. (Core task 2).

P11. Technical Research Centre of Finland (VTT) Finland

Partner 11 (VTT) will provided the Northern exposure site for the impregnated lap-joint trial.

P12. Dr. Wolman GmbH (Wolman) Germany

Partner 12 (Wolman) provided the technical expertise on the organo-metallic preservative in co-operation with Partner 4. (BAM).(Core task 1 (b); Sub-task 2.1).

P13. Eidgenössische Materialprüfungs- und Forschungsanstalt (EMPA) Switzerland

Partner 13 (EMPA) carried out most of the laboratory work on the conditioning test with insecticides and superficially treated fungicides. This major part of the project was funded by the Swiss Government. Partner 13 also helped to co-ordinate the laboratory work for the consortium. (Core tasks 1 (a), 1(b) and 1.2).

P14. Bundesforschungsanstalt für Forst-und Holzwirtschaft (BFH) Germany

Partner 14 (BFH) was solely responsible for the work on the lap-joint and weathering tests with regard to composites. (Sub-task 2.3).

P15. SHR Hout Research (SHR) Netherlands

Partner 15 (SHR) was responsible for carrying out critical moisture content studies of the lap-joints and relating these to actual profiles in use. (Sub-task 2.6).

5 Core task 1 part A: weathering cycles (EUC 1, 2 and 3), superficial treatments

5.1 Introduction

The aim within Core Task 1 was to find accelerated artificial ageing procedures for EUC 1 to 3, which can replace the long natural weathering time prior to the biological test.

The results of Core Task 1 will be described in three parts, entitled "Model Compounds" (P2: Sub-task 1.1.1), "Superficial Treated Wood" (P13: Task 1 A, Sub-Task 1.1 and 1.2) and "Pressure Treated Wood" (P5: Task 1 B). This new classification of Core Task 1 has been chosen to allow the logical assessment and interpretation of the different treatments and ageing procedures.

Both reports by P13 and P5 will refer to the Sub-tasks, previously known as 1A, 1B, and to Sub-task 1.2.

For the development of an artificial ageing method it is expected that it is only a model procedure for homologation and approval of wood preservatives in Europe, analogous to EN 73 and EN 84. It can never be a representative pattern of the different climatic zones in Europe. It needs to be validated and calibrated subsequently in every single climatic zone. If a factorial difference exists between the biological data after artificial and natural ageing in defined climatic zones, the factor, which determines the application level of the wood preservative or of the content of active ingredients (*a.i*) should be taken into consideration.

In the following project two terms are distinguished: "Artificial ageing" is a generic term for all the procedures, which aim at accelerated ageing of the wood treatment. It includes all the methods for climatic simulations in the different hazard classes (EUC) 1 to 5. "Artificial weathering" includes only the methods, which aim at accelerated ageing combined with sprinkling. They are destined to simulate the weathering conditions in EUC 3.

5.2 Aims and objectives

The aim within Core Task 1 was to find model methods which allow the accelerated ageing of superficial and pressure treated wood prior to the biological test and to simulate outdoors conditions in EUC 1 to 3.

According to the Technical Annex of project CT96 2135. The detailed aims of Core Task 1 are:

a) establish a suitable dry weather cycle for insecticides in EUC 1 and 2. This tasks involves the establishment of dry accelerated cycles with specific regard to the loss of insecticides in EUC 1 and 2, prior to biological testing according to EN 46 (*Hylotrupes bajulus*). The loss of insecticides will be related to loss under natural conditions and the existing preconditioning procedure EN 73 (Task 1A);

b) establish the importance of hydrolysis and photo-degradation of insecticides and fungicides in EUC 1 and 2 in service and related it to what happens in artificial procedures. (Sub-Task 1.1);

c) establish the fate of model compounds subjected to light, heat and moisture under conditions relevant to EUC 1 and 2. (Sub-task 1.1.1 Model compounds study);

d) develop "wet" artificial weathering cycles for fungicides and insecticides for EUC 3. The task involved the use of wet artificial weathering cycles with respect of EUC 3 prior to both insect (EN 47) and fungal tests (EN 113) with pressure treated wood. After weathering the appropriate biological tests will be performed and if necessary chemical analysis of the active ingredients. The chemical changes and losses of fungicide will be directly related to studies of the lap-joint field test (Task 2). Different partners will study different active ingredients (Task 1(B));

e) establish the justification to apply the same approach to superficially treated wood in EUC 3 in an additional national programme (Sub-task 1.2). Since superficial treatments are used in certain parts of Europe and extremely sensitive to the various weather influences, the preservatives will be applied to the wood by brushing or by means of a short-time dipping process. The fungicides and insecticides will be studied on wood blocks treated superficially prior to decay testing and using the newly established weathering cycles. The influence of the ageing on the active ingredients will be studied on the basis of the mass loss of the wood samples (fungi) and the mortality of the insects, if necessary, on selective analysis of the active ingredients.

5.3 Deliverables

On planning the project CEN/TC 38 expected the following deliverables from Core Task 1:

a) data on the role of heat, light and moisture on the longevity of active ingredients in wood will be presented. A statement on the suitability of the proposed EUC 2 artificial weathering cycle for simulating conditions and losses in real exposures, such as the construction phase;

b) artificial weathering cycles (dry) suitable to EUC 1 and 2;

c) artificial weathering cycles with additional leaching and photochemical degradation for EUC 2 exposed during the construction phase to the natural climate conditions;

d) artificial weathering cycles with leaching and photochemical degradation for EUC 3 (long term weather exposition).

5.4 Material and methods

a) Model compounds (Sub-task 1.1):

The suitability of the EMPA standard cycle for artificial conditioning (Table 1) at recreating the conditions in EUC 2 and allowing degradation processes to occur has been assessed.

Wood preservative ingredients that are susceptible to losses by evaporation, hydrolysis and photolysis were selected as the model compounds. Treated timber samples were exposed to light of known intensity and wavelength, and to known relative humidity, temperature and hydrolysing conditions. The laboratory tests, which simulated conditions in the EUC 2 artificial weathering cycle, were also compared with replicate samples that were exposed for 2 months in a 'natural' weathering exposure.

Table 1 — EMPA Standard cycle for artificial conditioning EUC 2

Apparatus	QUV accelerated weathering apparatus, with spray option, manufactured by the Q-Panel Company, Cleveland, Ohio, USA.
UV light installation	UVA-340 (12 to 88) tubes (λ ≈ 340 nm)
Temperature of chamber	40 °C during light exposure
Wetting water	Deionised water, with an electrical conductivity of < 5 µS/cm (hydraulic pressure at entering the QUV device:+ 0,3 MPa (3,0 bar))
Water flow rate	3 l/min
Cycle	15 min spraying (dark) and 30 min light (without spraying)
Duration of the weathering test	7 days ≈ 2 months natural weathering

In all experiments Scots pine sapwood (*Pinus sylvestris*) panels were treated with model compound containing wood preservative formulations and the analytical zone was 0,0 mm to 0,5 mm from the surface. Full details of these exposures are recorded in Technical Annex 2.

1) Photolysis

The potential for photolysis to occur in treated timber was monitored by the extent of conversion of tri-butyl tinoxide (TnBTO) to di-butyl (DBTO) and mono-butyl (MBTO) tinoxide. Panels were treated with a 1 % TnBTO solution in white spirit and exposed to accelerated weathering in QUV chambers. The zone 0,0 mm to 0,5 mm from the surface were analysed for TnBTO and its breakdown products (DBTO and MBTO) using a polarographic method.

2) Hydrolysis

The potential for hydrolysis to occur in treated timber was monitored by the extent of conversion of trihexylene glycol diborate (THGBB) to boric acid. To recreate the conditions of EUC 2 panels treated with a 5 % THGBB solution in white spirit were exposed in alternating cold cabinets and conditioning rooms at 90 % r.h , to create condensation. The analytical method includes extraction of the THGBB from the wood samples in hexane followed by a thorough washing of the hexane with water to hydrolyse the glycol to boric acid, transferring it to the aqueous layer. The aqueous layer was then analysed quantitatively for boron.

3) Evaporation

The potential for evaporative loss to occur in treated timber was monitored by the extent of loss of lindane during exposure. Extensive research has been conducted on the loss of preservative active ingredients due to evaporative loss and the connection with the actives vapour pressure. A review of the literature is presented in Technical Annex 2. A summary of findings highlighted that evaporation of lindane, our chosen model compound, in dry and wet conditions needed investigating. Panels treated with a 0,5 % lindane solution in white spirit were exposed in two vacuum ovens set at 40 °C (the operating temperature in the EUC 2 weathering cycle), one at a relative humidity of less than 60 %, and one at a relative humidity greater than 90 %. Panels treated with lindane were exposed in both conditions for one week as well as being cold stored for reference material. The samples were extracted by methanol reflux and analysed by gas chromatography.

4) Natural 'weathering trials'

Samples of treated timber prepared with TnBTO, trihexylene glycol diborate and lindane and were exposed inside the window joinery tests facility at BRE to simulate a 2 month exposure during the construction phase. Conditions are warm but variable (5 °C to 22 °C), moist (70 % r.h to 95 % r.h and weekly misting with de-ionised water) and light (exposed at 45° behind south facing glass). These conditions would allow losses to occur. The information from these 'natural' weathered panels has been compared with the model compound studies to assess the suitability of the artificial weathering cycle at simulating losses during the construction phase.

b) Surface treated wood

The wood preservatives, ageing methods, organisms and biological tests used within the project are described in the Technical annex of Core Task 1. The biological tests have been carried out according to EN standards and to an institution method of P13.

With the extensive tests, carried out within the frame of Core Task 1, it has been shown that the concentrations of active ingredients (*a.i*) in wood preservatives, determined at the beginning, were not chosen within the most suitable range. Due to the long test periods of ageing procedures and biological tests, the concentrations of the active ingredients could, however, not be changed in the course of the project, as the different ageing methods would have been even less comparable. As a consequence, no draft of a method for artificial ageing according to EUC 1 to 3 can be presented to CEN/TC 38.

5.5 Results and discussion

a) Model compounds (Sub-Task 1.1):

A comparison of losses in the simulated artificial weathering conditions and the 'natural' weathering conditions appears in Table 2. This is a synthesis of the data that appears in Technical Annex 2.

Results indicate rapid losses of TnBTO and the appearance of the degradation products DBTO and MBTO within a day of exposure to photolytic conditions. Rapid hydrolysis of glycol diborate under artificial weathering was observed and significant losses of lindane due to evaporation occur during the 7 days exposure.

Table 2 — Summary of losses due to degradation processes

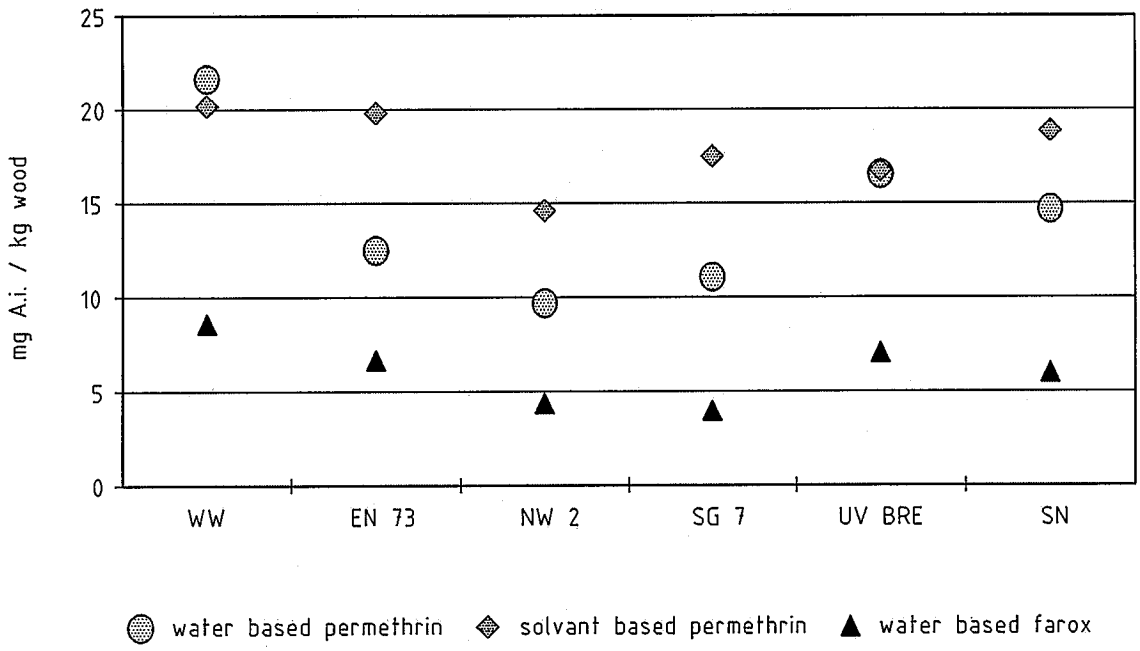
Type of degradation	Model compound	Losses in artificial weathering simulation in laboratory (1 week)	Losses in 'natural' weathering test (2 months)	EMPA weathering cycle for EUC 2
Photolysis	TnBTO	40 % conversion of TnBTO	10 % conversion of TnBTO	suitable
Hydrolysis	Trihexylene glycol diborate (THGBB)	Total loss	50 %	suitable (severe)
Evaporation	Lindane	30 %	50 %	probably suitable

b) Surface treated wood:

In general it is to be stated that natural weathering and also the different artificial ageing methods can have a varying influence on the biocidal protection of the treated wood, depending on the active ingredient (*a.i*), the formulation of the wood preservative (water - or solvent-based) and on the wood species.

The influence of the different ageing methods varies according to the wood species. In comparative tests with insecticides on Pine and Spruce, the chosen *a.i* concentrations on Spruce allowed a better differentiation of the ageing methods. That can be due to the lesser penetration of the *a.i* into the wood and, consequently, to a more intensive ageing exposition.

EN 73: it is suitable for the artificial ageing of treated wood, used in practice under dry conditions (EUC 1) and not exposed to weathering during the construction phase respectively protected by that time with a top-coat. If the *a.i* degradation is not a consequence of evaporation only but also of hydrolysis and/or photochemical processes, EN 73 cannot simulate the real conditions in EUC 1 and 2 (Figure 1).



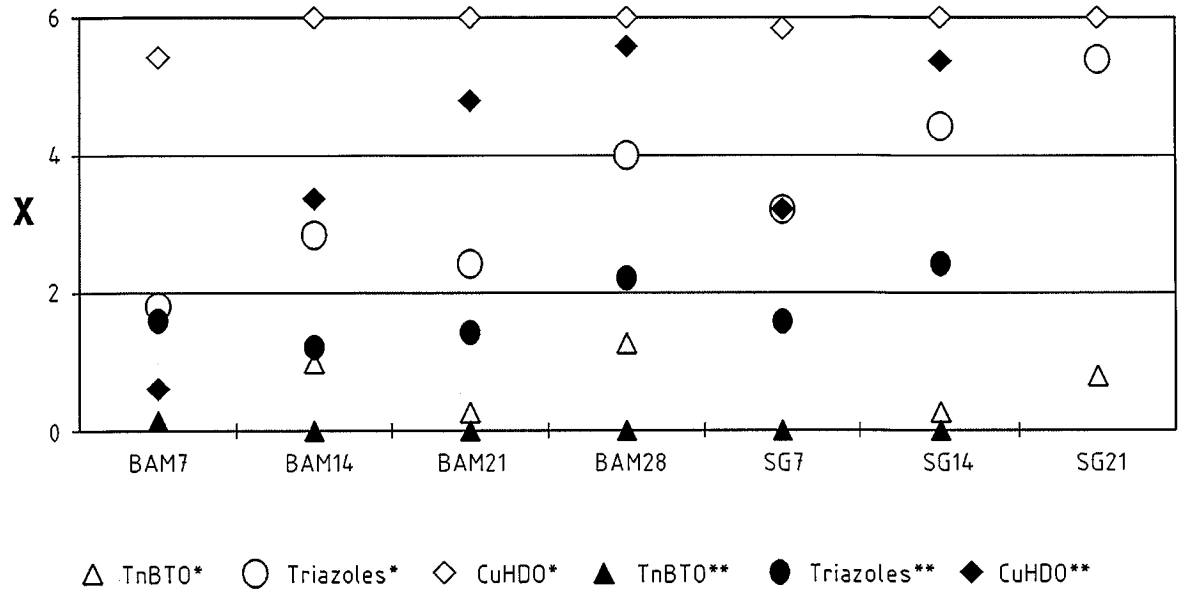
Key

1	water based permethrin
2	solvent based permethrin
3	water based farox
x	mg A.i. / kg wood
WW	without weathering
NW2	Two months natural weathering
SG 7	7 days artificial weathering according to the EMPA-cycle
UV BRE	Ageing 7 days under BRE condensation conditions
SN	SN 198890: 14 days ageing by 70 °C / 50 % relative air humidity

Figure 1 — Influence of the ageing method on the insecticide degradation on surface treated pine wood

EN 84: it is suitable for simulation of the environmental conditions in EUC 3 in which the *a.i* can be leached by water only. It is not suitable in situations in which the *a.i* are also degraded by light, hydrolysis and evaporation (see situations in the field). A method for artificial weathering should include all these parameters.

The artificial weathering procedures of BAM or EMPA SG, tested within the project can be taken as a basis for the further development of the method (Figure 2). In opposition to the BAM method, the EMPA SG method shows an acceleration of the ageing around factor 2. Independently of the method to be chosen, the different test parameters need to be optimized. The same weathering cycle can be used for both simulations, the construction phase previous to the exposure in EUC 1 or 2 and the climatic conditions in EUC 3. The difference consists merely in the number of weathering cycles, which should be carried out to simulate real conditions in the different EUC's. The factor between the two ageing times respectively number of ageing cycles in EUC 2 and EUC 3 should amount to 3 at least.



Key

Treatment

*	Brushing
**	Steeping
BAM 7	BAM cycle, 7 days
SG 7	EMPA SG cycle, 7 days

Figure 2 — Relative influence of different ageing on the loss of efficacy of the fungicidal wood preservation after treatment by brushing respectively steeping (criterion: superficial attack by basidiomycetes, rating 1 to 6)

To evaluate the suitability of the chosen artificial weathering method in EUC 3, the weathering method with 6 months exposure time and a 45° inclination of the treated wood samples is appropriate, as described in EN 152-1, subclause 7.2.1. A 90° inclination would prolong the required ageing time by a factor of approximately 2.

It should be taken into account that different types of active ingredients and also different wood preservative formulations (e.g. water and solvent based preservatives) have to be used for the further development by CEN/TC 38 of the EMPA SG or the BAM artificial weathering method. They show varying behaviour under different weathering influences. In tests combining ageing and biological test it is important that the individual *a.i* are tested in three concentrations at least, as for the individual ageing methods – especially in tests with fungi – mass losses caused by fungi respectively mortality rates of insects can be reached only within a very close range of concentration, which is required for the differentiation of the ageing methods.

As a rule, a statement with the biological test can only be made about "effective or ineffective" – without any closer differentiation. With the chemical analysis the influence of the different ageing methods on the degradation of the *a.i* can be studied continuously, from the initial active substance content up to the concentration of the detection limit. In opposition to the biological test one *a.i* concentration respectively application rate is in general sufficient. The data of the chemical analyses are available within shorter time than those of the biological tests. The evaluation of the ageing methods based on chemical analyses implies that a validated analytical method is available and that a sufficient number of random samples per test are analyzed. Subsequently it is possible to analyze mixed samples of the different random samples.

If an artificial weathering method is developed by means of chemical analyses, it should be validated in the end with a ringtest combined with a biological test, before it can be submitted to the final vote of the different countries as a European Standard. Tests within the project have revealed that the data of the chemical analyses and those of the biological tests do not always correspond.