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Buildings and civil engineering works — Sealants — Determination of changes in cohesion and appearance of elastic weatherproofing sealants after exposure of statically cured specimens to artificial weathering and mechanical cycling

Bâtiments et ouvrages de génie civil — Mastics — Détermination des variations de cohésion et apparence des mastics élastiques résistants aux intempéries après exposition d'éprouvettes statiquement polymérisées à un cycle mécanique et de vieillissement artificiel

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Foreword

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The committee responsible for this document is ISO/TC 59, *Buildings and civil engineering works*, Subcommittee SC 8, *Sealants*.

Buildings and civil engineering works — Sealants — Determination of changes in cohesion and appearance of elastic weatherproofing sealants after exposure of statically cured specimens to artificial weathering and mechanical cycling

1 Scope

This International Standard specifies laboratory exposure procedures for determining the effects of cyclic movement and artificial weathering on cured, elastic weatherproofing joint sealants (one- or multi-component).

2 Normative reference

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

ISO 4628-4:2003, *Paints and varnishes — Evaluation of degradation of coatings — Designation of quantity and size of defects, and of intensity of uniform changes in appearance — Part 4: Assessment of degree of cracking*

ISO 4892-1:1999, *Plastics — Methods of exposure to laboratory light sources — Part 1: General guidance*

ISO 4892-2:2013, *Plastics — Methods of exposure to laboratory light sources — Part 2: Xenon-arc lamps*

ISO 4892-3:2013, *Plastics —Methods of exposure to laboratory light sources — Part 3: Fluorescent UV lamps*

ISO 4892-4:2013, *Plastics — Methods of exposure to laboratory light sources — Part 4: Open-flame carbon-arc lamps*

ISO 6927:2012, *Buildings and civil engineering works — Sealants — Vocabulary*

ISO 8339:2005, *Building construction — Sealants — Determination of tensile properties (Extension to break)*

ISO 11431:2002, *Building construction — Jointing products — Determination of adhesion/cohesion properties of sealants after exposure to heat, water and artificial light through glass*

ISO 11600:2002, *Building construction — Jointing products — Classification and requirements for sealants*

ISO 13640:1999, *Building construction — Jointing products — Specifications for test substrates*

CIE Publication No. 20-1972, *Recommendations for the integrated spectral irradiance and the spectral distribution of simulated solar radiation for testing purposes*

CIE Publication No. 85-1989, *Technical report — Solar spectral irradiance*, ISBN 3 900 734 22 4

3 Definitions

For the purposes of this document, the definitions given in ISO 6927 apply.

Any notation in this standard shown as 'target set value x ± operational fluctuation y' shall be interpreted as follows: set the experimental parameter at the target value x and maintain the experimental parameter during the test procedure at ± y from the specified setting x. If the operational fluctuations exceed the maximum allowable value after the equipment has stabilized, discontinue the test and correct the cause of the problem before continuing.

4 Principle

Test specimens are prepared in which the sealant to be tested adheres to two parallel support surfaces (substrates). The specimens are conditioned statically (no movement) in a controlled climate. The conditioned specimens are then exposed to repetitive degradation cycles of artificial weathering (light, heat, and moisture) and cyclic movement under controlled environmental conditions. Within each cycle, weathering is carried out for six weeks in an artificial weathering machine. Simultaneously, with the weathering, mechanical cycling is carried out by changing the position of the extension/compression once a week. After completion of each degradation cycle (each lasting six weeks), the specimens (in their extended/compressed state) are visually examined for changes in appearance, cohesion, and adhesion of the sealant beads. The rating for quantity, width, and depth of cohesive cracks for a specific extension/compression value achieved along the length of the specimen as well as the depth, length, and range of any very significant loss of cohesion or adhesion (defined as >3 mm crack depth) is determined and the general condition of the sealant is reported. The weathering and mechanical cycling exposure and the examination for failures constitute a degradation cycle and the degradation cycle is repeated as often as desired to achieve a certain exposure.

5 Apparatus

5.1 Support

Anodized aluminium support (as shown in Figure 1) for the preparation of test specimens, consisting of two pivoting, L-shaped anodized aluminium support elements of dimensions 120 mm × 18 mm × 18 mm (length × width × height) and 2 mm thickness riveted onto an anodized aluminium base-plate of 2 mm thickness such that a cavity of dimensions 120 mm × 20 mm × 18 mm (length × width × height) is formed. Riveting of the support elements on the base-plate shall be such that they can be turned freely with minimal friction on the pivot (fulcrum). The base plate holds five equally spaced holes of 5 mm diameter (for improved ventilation of the back face of the sealant such as to ensure better cure or drying of the sealant) and two 3 mm holes for fixation of the specimen (see Figure 1 and Annex A). For the specification of the anodized aluminium, refer to ISO 13640. All surfaces of the anodized aluminium support to be later in contact with the sealant shall be cleaned according to the sealant manufacturer's recommendation.

NOTE Achieving optimum adhesion on the support substrate is important in order to obtain reproducible ratings for surface and bulk degradation (cracking, crazing, cohesive failure, etc.) that is induced or influenced by mechanical cycling. Even a partial loss of adhesion will cause a section of the test specimen to be exposed to no or a lower degree of mechanical cycling than intended for a given movement amplitude of the sealant and invalidate the results obtained for this movement exposure (as assessed along the extended leg of the test specimen). Currently, no cleaning procedure and cleaning agent(s) have been identified that provide optimum adhesion on the support substrate for all sealant products. Therefore, no cleaning procedure is specified in this International Standard. If the manufacturer does not provide a recommendation for the cleaning procedure, the following method is suggested for consideration by the experimenter: Clean all surfaces of the anodized aluminium support to be later in contact with the sealant with high purity acetone (purity, by gas chromatography: 99,8 %) as follows: a) saturate a clean, lint-free paper tissue or cloth with the solvent; b) clean the substrate with the solvent-saturated cloth or tissue by wiping a minimum of three times so that visible contamination cannot be observed; c) wipe the substrate with solvent-saturated cloth or tissue and immediately afterwards dry wipe the substrate surface thoroughly using a dry, clean, lint-free paper tissue or cloth before the solvent completely evaporates. Repeat step c) at least once. In each wipe of the substrate surface during step c), a new, uncontaminated cloth or tissue should be used. For severely contaminated substrates, additional pre-cleaning steps can be required.

If other support materials are to be used, they shall be characterized and shall be described in the test report. If other support dimensions are used, they shall be described in the test report.

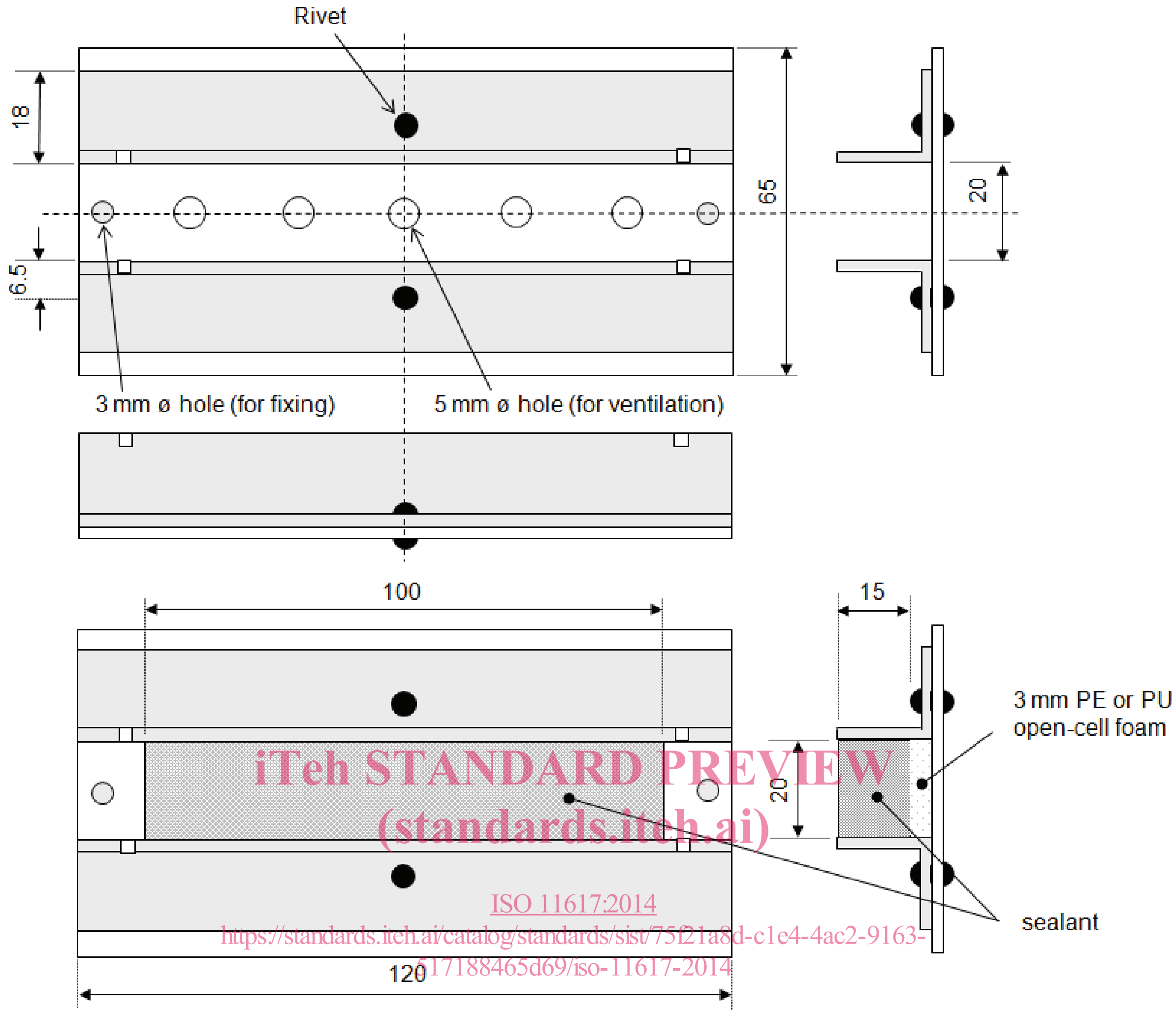

Figure 1 — Schematic drawing of test specimen — Sealant in anodized aluminium support used for cyclic mechanical movement of sealant (all units in mm)

5.2 Spacers

Spacers for the preparation of the specimens, of dimensions 20 mm × 18 mm × 10 mm, with anti-adherent surface (see Figure 1) shall be used. If the spacers are made of material to which the sealant adheres, their surface shall be made anti-adherent, e.g. by a thin wax coating.

5.3 Backing material (bond breaker)

Open-cell foam backing material [polyethylene (PE) or polyurethane (PU) foam] of 3 mm thickness for the preparation of test specimens shall be used. The foam backing material shall not restrict the movement of the L-shaped pivoted support elements.

5.4 Separators

Separators of appropriate dimensions shall be used to hold the test specimens in extension up to the maximum specified movement amplitude of the sealant.

5.5 Container

Container filled with demineralised or distilled water shall be used for conditioning according to method B.

5.6 Ventilated convection-type oven

Ventilated convection-type oven, capable of being maintained at (70 ± 2) °C, shall be used for conditioning according to method B.

5.7 Fully automated test chamber with an artificial light source

Fully automated test chamber with an artificial light source (5.8), shall be used, capable of exposing the test specimens to radiation under controlled conditions of temperature, relative humidity, and water, complying with the requirements of ISO 4892, Parts 1, 2, 3, and 4. The radiation is always directed towards the same surface of the sealant specimen. Standard practices for operating such accelerated weathering chambers are described in ISO 4892-1.

The level of irradiance and water exposure at the specimen surface as described in 5.8 and 8.2 cannot be altered.

In fully automated test equipment, exposure to water for this test method is accomplished by water spraying the specimen surface or immersing the test specimens in water.[1),2)] Contamination of the water is to be avoided. The purity of the water to be used is described in ISO 4892-1. The water temperatures are typically (21 ± 5) °C for the spray water and typically (40 ± 5) °C for the re-circulated immersion water.[3)]

Suitable equipment and test procedures for cyclic exposures to water are described in ISO 4892, Parts 1, 2, 3, and 4. Water is a key factor contributing to the ageing of sealants, especially in combination with exposure to light. In xenon arc devices that use water spray for wetting, relative humidity during the light period shall be maintained at (50 ± 10)% r.h. (see ISO 4892-2, Table 3, Method A, Cycle Number 1).[4)]

1) Adequate heat transfer between the test specimen and the environment is essential during the lower temperature period in the fluorescent UV/condensation device in order for condensation on the sealant to occur. This places restrictions on the thermal mass and, consequently, on the dimensions of a specimen. No experimental data have been generated on the time-of-wetness of sealant test specimens of the kind specified in this International Standard when placed in fluorescent UV/condensation device operating at conditions specified in this International Standard. However, testing conducted by ASTM C24 on ISO 8339 specimens appears to suggest that the condensation process provided in the fluorescent UV/condensation apparatus is generally not applicable to the type of sealant specimens tested. Therefore, wetting in this International Standard is carried out by water spray on the exposed specimen surface (default method). However, the front surface water spray accessory was not designed for this purpose and requires an unreasonable amount of pure water for the wet period specified. Therefore, often the equipment is modified to allow re-circulation of the water during the exposure period. Some fluorescent UV equipment has adaptable spray manifolds, which allow installation of lower flow type nozzles, thus reducing the amount of pure water used.

2) Data generated with these two methods of water exposure (spray or immersion) in a round robin test on a set of sealants for revision of ISO 11431 showed acceptable correlation, although contributions to the various degradation mechanisms acting in the specimens (e.g. hydrolysis, thermal shock, leaching of formulation components, etc.) can differ between these exposures. The degree of correlation between these two methods thus can vary depending on the specific sealant tested.

3) Spray water can be fresh or re-circulated from a holding tank. Immersion water is generally in a holding tank for re-circulation. The temperature of the spray water is uncontrolled and for fresh water is typically (21 ± 5) °C. Re-circulated spray water can be at a higher temperature. The uncontrolled temperature of the re-circulated immersion water during operation of the weathering device is typically (40 ± 5) °C. It can be controlled by heating the water to a higher temperature. However, heating is not desirable because the water immersion temperature would then differ to a larger extent from the spray water temperature.

4) Generally, automated-weathering equipment based on xenon-arc light with water immersion exposure and fluorescent UV lamp type equipment do not allow control of humidity during the light period.

In the immersion technique, the test specimens are placed in a chamber that is periodically flooded with re-circulated water. During immersion, the specimens are completely covered by water. The water temperature is measured below the water surface with the black standard thermometer. The immersion system shall be made from corrosion resistant materials that do not contaminate the water employed.

5.8 Artificial light source

Light sources for the simulation of the global radiation at the surface of the earth are subject to development. The degree of approximation to the spectral power distribution according to CIE publication No. 85 (Table 4) depends on the type of lamp. Xenon-arc lamps with suitable filters are preferred and are considered the default for the purpose of this International Standard.

Several factors can change the intensity and the spectral power distribution of the artificial light source during service. Comply with the manufacturer's recommendations and the requirements of ISO 4892 to maintain constant irradiation conditions.

5.8.1 Xenon-arc light source (default)

Xenon-arc light source with daylight filters shall be used for the simulation of terrestrial daylight as defined in the CIE publication No. 85. The spectral power distribution of the radiation shall comply with the requirements outlined in ISO 4892-2, method A. Irradiance at the surface of the test specimens between the wavelengths of 300 nm and 800 nm shall be set at 550 W/m^2 and maintained at ±75 W/m^2. The equivalent irradiance setting for 300 nm to 400 nm shall be 60 W/m^2 maintained at ±2 W/m^2 and the setting for 340 nm shall be 0,51 W/(m^2 nm) maintained at ±0,02 W/(m^2 nm). If, exceptionally, other intensities will be used, these shall be stated in the test report. Irradiance below 300 nm shall not exceed 1 W/m^2. The irradiance shall not vary by more than ±10 % over the whole specimen exposure area.

5.8.2 Fluorescent ultraviolet source (option)

Fluorescent UVA-340 lamp(s) shall be used. The radiation of UVA-340 lamp(s) is mainly in the ultraviolet region between 300 nm and 360 nm with negligible visible and infrared radiation. The spectral power distribution of the radiation shall comply with the requirements outlined in ISO 4892-3 for a lamp with 343 nm peak emission. Irradiance below 300 nm shall not exceed 1 W/m^2. The irradiance shall not vary by more than ±10 % over the whole specimen exposure area.

5.8.3 Open-flame carbon arc source (option)

Open-flame carbon arc light sources typically use carbon rods, which contain a mixture of metal salts. An electric current is passed between the carbon rods, which burn and give off ultraviolet, visible, and infrared radiation. Use carbon rods recommended by the device manufacturer. The spectral power distribution of the radiation shall comply with the requirements outlined in ISO 4892-4, open-flame carbon arc light source with daylight type filter (type 1 filter).[5)]

5.9 Black standard (insulated) and black panel (uninsulated) temperature sensors

Black standard (default) and black panel thermometer (option) temperature sensors shall comply with the requirements outlined in ISO 4892-1, 5.2. The default thermometer is the black standard thermometer.

NOTE Under given operation conditions, black panel (uninsulated) thermometers tend to indicate lower temperatures than the black standard (insulated) thermometers. The temperature difference between the two types ranges between 3 °C and 12 °C, being smaller at lower irradiance levels.

The thermometer shall be mounted on the specimen rack so that its surface is in the same relative position and subjected to the same influences as the test specimens. Readings shall only be taken after sufficient time has elapsed for the temperature to become constant.

5) The chemical composition of the carbon rods can affect the spectral power distribution of open flame carbon arc light sources.

6 Preparation of test specimens

Three test specimens for each sealant and each aging method shall be prepared.

Bring the sealant to (23 ± 2) °C before preparation of the specimens (this is generally achieved by conditioning the packaged sealant for 24 h at this temperature). Prepare three specimens. For each specimen, assemble one support (5.1), see Figure 1, by inserting two spacers (5.2) at the ends of the joint. Apply the open-cell backing foam (5.3) to the bottom of the joint.

Follow the instructions of the sealant manufacturer concerning the sealant application, for instance, whether a primer is to be used on the contact surface of the L-shaped support elements.

Fill the hollow volume (dimensions: width × depth × length = 20 mm × 15 mm × 100 mm) formed by the support, the backing foam and spacers with the sealant, while taking the following precautions:

a) Avoid the formation of air bubbles.

b) Press the sealant to the inner surfaces of the pivoted support elements.

c) Trim the sealant surface so that it is flushed with the faces of the support elements and spacers.

After preparation, the sealant test specimens shall be examined for defects. Any test specimens deemed unsuitable for testing shall be rejected.

7 Conditioning

7.1 General

Condition the specimens at rest (static conditioning) in accordance with method A (default) or method B (option), as agreed between the parties concerned.

7.2 Method A (default)

Place the specimens, such that air can freely circulate to the back-face of the base-plate. Condition the specimens, with the spacers in place, for 28 d at (23 ± 2) °C and (50 ± 5) % relative humidity. After the conditioning, remove the spacers at both sides of the sealant joint.

7.3 Method B (option)

Condition the specimens first according to method A. Then subject them three times to the following conditioning cycle:

a) 3 d in the oven (5.6) at (70 ± 2) °C;

b) 1 d in distilled water at (23 ± 2) °C;

c) 2 d in the oven (5.6) at (70 ± 2) °C;

d) 1 d in distilled water at (23 ± 2) °C.

This cycle can be carried out alternatively in the sequence c), d), a), and b). After the conditioning, remove the spacers at both sides of the sealant joint.

NOTE Conditioning B is a normal conditioning method using the influence of water and heat to accelerate the cure of the sealant. It is not intended to give information on the durability of the sealant.

8 Test procedure

8.1 General

After conditioning and removal of the spacers, expose the specimens to the artificial weathering cycles and mechanical movement (fatigue) cycles, as agreed by the parties concerned. The choice of the type of accelerated weathering exposure shall be by mutual agreement among the interested parties. The default degradation cycle is six weeks of exposure in Xenon-arc type automatic weathering equipment (8.2.1) with the test specimen simultaneously being exposed to mechanical cycling (8.3). The default value for the total number of degradation cycles is three.

NOTE Because the different types of exposures can produce different test results, they cannot be used interchangeably without supporting data that demonstrates equivalency of the procedures for the materials tested.

8.2 Accelerated weathering exposure conditions (default period: six weeks)

During the artificial weathering cycle, expose the test specimens to radiation by the artificial light source such that the specimen test surface faces the lamp. During repeated exposure periods, i.e. when the specimens are exposed to several degradation cycles, direct the radiation towards the exposed surface of the sealant. Mount the test specimens so that the plane of the test surface is at a distance from the lamp(s) consistent with the method for operating the apparatus (ISO 4892-2 or ISO 4892-3 or ISO 4892-4). Control the test temperatures with a black standard thermometer (default) or black panel thermometer (option), as specified in 5.9, mounted on the specimen rack so that the face of the temperature sensor is in the same relative position and is subjected to the same influences as the test specimens.

8.2.1 Exposure in automatic weathering equipment — Xenon-arc type (default)

The light source shall be one or more xenon-arc lamps with daylight filters installed to simulate terrestrial daylight. The spectral power distribution, operating practices, and irradiance-uniformity and short wavelength limit shall be as defined in 5.8.1.

The standard conditions of test (default) are repeated cycles of exposure that consist of six weeks of exposure in the xenon-arc machine, the irradiance level being set as specified in 5.8.1, with alternating periods of dry and wet:

a) A dry period of 102 min, in which the specimens are exposed to radiation and heat. From the start of the dry period the temperature is allowed to rise, until it reaches a steady temperature of (65 ± 3) °C, as measured on the black standard thermometer (5.9). Relative humidity during the dry period is (50 ± 10) % using the xenon-arc device with water spray. The chamber air temperature in machines that allow for its control shall be (38 ± 3) °C.

b) A wet period of 18 min, in which the specimens are exposed to radiation and wetting either by water spray on the exposed surface or immersion in water. The water temperatures are typically (21 ± 5) °C for the spray water and typically (40 ± 5) °C for the re-circulated immersion water.

The cycles of dry and wet exposures are repeated 504 times (default) (six weeks total duration in the weathering machine).

NOTE Alternative exposure cycles as appropriate and agreed amongst the parties concerned can be used. However, if the experimenter deviates from the default values specified, both the default values as well as the actual conditions used shall be reported.

Filtered xenon arcs provide a good representation of the spectral power distribution as defined in the CIE publication No. 85 and therefore, are considered as the reference for the purpose of this International Standard.