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Konstrukcijska lepila - Ugotavljanje roka uporabnosti večkomponentnih lepil

Structural Adhesives - Determination of the pot life (working life) of multicomponent adhesives

Strukturklebstoffe - Bestimmung der Topfzeit (Verarbeitungszeit) von Mehrkomponentenklebstoffen

Adhésifs structuraux - Détermination de la durée de vie en pot (délai d'utilisation) des adhésifs multicomposants

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Structural Adhesives - Determination of the pot life (working life) of multi-component adhesives

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Foreword

This document (EN 14022:2010) has been prepared by Technical Committee CEN/TC 193 “Adhesives”, the secretariat of which is held by AENOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by August 2010, and conflicting national standards shall be withdrawn at the latest by August 2010.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN 14022:2003.

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According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and the United Kingdom.

1 Scope

This European Standard specifies means of determining in appropriate ways the variable property known alternatively as useable working life and pot life.

This European Standard specifies five methods for the determination of the time available for use, each of which is related to specific circumstances; particularly important being the rheology of the adhesive concerned and its rate of reaction.

This European Standard can also be used for assessing non-structural adhesives.

NOTE EN 302-7 could also be used for the determination of working life of adhesives for load-bearing timber structures.

Because of the different properties of the individual multi-component systems, like rheology or viscosity, respectively velocity of hardening, etc., not all methods can be applied to each multi-component system with the same suitability.

SAFETY STATEMENT— Persons using this document should be familiar with the normal laboratory practice, if applicable. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any regulatory conditions.

ENVIRONMENTAL STATEMENT — It is understood that some of the material permitted in this standard may have negative environmental impact. As technological advantages lead to acceptable alternatives for these materials, they will be eliminated from this standard to the extent possible.

At the end of the test, the user of the standard should take care to carry out an appropriate disposal of the wastes, according to local regulation.

2 Normative references

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

EN 923:2005, *Adhesives — Terms and definitions*

EN 1067, *Adhesives — Examination and preparation of samples for testing*

EN 12092:2001, *Adhesives — Determination of viscosity*

EN ISO 15605, *Adhesives — Sampling (ISO 15605:2000)*

3 Terms and definitions

For the purposes of this document, the terms and the definitions given in EN 923:2005 and the following apply.

3.1
pot life
working life
period of time during which a multi-component adhesive can be used after its component parts have been mixed

NOTE 1 The pot life of any reacting adhesives is affected by the rate at which the heat energy generated by the reaction is dissipated. The rate of dissipation depends significantly upon the volume and temperature of the mixed adhesive and the ambient temperature. The term "pot life" is also used to describe the period during which hot-melt adhesives remain fit for use when kept at their normal operating temperatures.

NOTE 2 While in some of the measuring methods described in this standard the pot life is measured as the period of time which starts when the mixing ends, in some other methods described in this standard, the pot life is measured as the period of time which starts when the mixing starts.

4 Typical purposes for the test methods

4.1 Method 1: Determination by means of change in apparent viscosity (rotating viscometer or oscillating rheometer)

This test method provides a means of measuring a pot life greater than 5 min; where pot life is quantified by means of a specified increase in the viscosity of the reacting adhesive.

4.2 Method 2: Determination by means of a change in extrusion rate

This test method provides a means of measuring a pot life (for paste-like adhesives) greater than 5 min; where pot life is quantified by means of a specified decrease in the weight of reacting adhesives extruded, in unit time, under standard conditions.

4.3 Method 3: Determination by means of manual application

This generally applicable method provides a means of measuring a pot life of any duration; where pot life is quantified as the time by which a reacting adhesive can no longer be spread by hand.

4.4 Method 4: Determination by means of exothermic reaction temperature

This test method provides a means of measuring a pot life which can be used for any reactive multi-component system; where pot life is quantified as the time by which a batch of the reaction product reaches a defined temperature, the so-called critical temperature (e.g. 40 °C). For products producing less than 40 °C exothermic reaction heat in the defined batch, the maximum temperature is taken as criteria.

4.5 Method 5: Determination by means of a drying recorder

This test method provides a means of measuring the pot life, for two component adhesives and specially for one component adhesives which easily react with air humidity (e.g. PUR prepolymers).

5 Limits of the test methods

While the described test methods are suitable for assessing multi-component epoxide or polyurethane-based adhesives, they are not suitable for some acrylic based-adhesives.

6 Determination

6.1 General

The adhesive to be tested, by any one the five given test methods, shall be sampled, prepared and examined according to EN ISO 15605 and EN 1067. For any method chosen, at least three samples shall be evaluated and the mean determined.

6.2 Method 1: Determination by means of a change in apparent viscosity (rotating viscometer or oscillating rheometer)

6.2.1 Principle

A multi-component adhesive is mixed and its pot life established by measuring the time taken for its viscosity to change by a specified amount.

This method is not suitable for the determination of pot lives that are shorter than 5 min in duration.

6.2.2 Apparatus

6.2.2.1 **Balance**, capable of weighing up to (500 ± 0,2) g.

6.2.2.2 **Beaker**, squat form, capacity 400 ml, made of an unreactive material with adhesive, whose wall thickness does not exceed 1 mm.

6.2.2.3 **Spatula** made of a non-reactive material with adhesive and with an angular, not circular end.

6.2.2.4 **Viscometer**, any suitable means of measuring the viscosity of the adhesive (rotating viscometer or oscillating rheometer) may be selected. See EN 12092:2001, Method 1, for the viscosity measurement with rotating viscometer.

6.2.2.5 **Stopwatch**, accurate to ± 1 s.

6.2.2.6 **Test enclosure,** capable of being maintained at the test temperature and if necessary at a relative humidity of (50 ± 5) %.

6.2.3 Procedure

Both components of the product shall be maintained separately at (23 ± 2) °C. The single components shall be weighed into the beaker (see 6.2.2.2) according to the mixing ratio specific for the product, with the preferred mass of the batch of the products being (200 ± 20) g.

Start the stopwatch (see 6.2.2.5) and mix the batch with an angular, not circular end of a spatula (see 6.2.2.3) for (60 ± 10) s. Take care that also the areas within the angle between side and bottom of the beaker are well mixed.

It is also allowed to determine a pot life of a batch which was produced with the help of a static or dynamic mixer, which has to be defined specifically.

Immediately after mixing measure the viscosity with a viscometer (see 6.2.2.4) of the freshly prepared adhesive.

Record the first data after having finished mixing as the starting viscosity point. Continue the measurement in intervals dependent on the pot life expected.

NOTE The number of measurements as well as the extent of shear at mixing of the components and the measurement itself can have an influence on viscosity and pot life. Therefore, it is recommended to fix the intervals of measurements as well as mixing, shear speed and rotational speed specific to the adhesive.

The pot life of the adhesive is the difference between time at the end of mixing and the time when a fixed agreed viscosity is reached. Usually the fixed agreed viscosity is double the starting viscosity.

6.3 Method 2: Determination by means of a change in extrusion rate

6.3.1 Principle

A multi-component adhesive is mixed and its pot life established by measuring the time taken for there to be a specified decrease in the quantity of adhesive extruded through a calibrated orifice, in unit time, under standard conditions.

This method is not suitable for the measurement of pot lives that are shorter than 5 min in duration.

6.3.2 Apparatus

6.3.2.1 Cartridge, plastic disposable cartridge of 47 mm internal diameter and 210 mm length, fitted with an appropriate piston; both components being made from a non-reactive material.

6.3.2.2 Balance, capable of weighing up to (500 ± 0,2) g.

6.3.2.3 Stirrer, rigid, helicoidal steering spindle made from non-reactive material suitable for use in conjunction with the adhesive being assessed.

6.3.2.4 Motor, an electrically or pneumatically powered stirrer motor whose speed can be regulated between 0 r/min and 1 000 r/min.

6.3.2.5 Nozzle, calibrated made from non-reactive material, capable of being screwed onto the end fitting of the cartridge (see 6.3.2.1). The diameter of the nozzle's extrusion orifice shall be suitable for dispensing the mixed adhesive. An orifice diameter of 2 mm to 4 mm is recommended for evaluation.

6.3.2.6 Extrusion gun, air pressurised extrusion gun suitable for use with the cartridge described in 6.3.2.1.

6.3.2.7 Pressure gauge, air pressure gauge capable of measuring air pressure up to 500 kPa with a precision of ± 10 kPa.

6.3.2.8 Stopwatch, accurate to ± 1 s.

6.3.2.9 Dishes, of suitable capacity, pre-weighed, aluminium foil dishes.

6.3.2.10 Bath, capable of being maintained within ± 0,1 °C throughout a temperature range between 15 °C and 30 °C.

6.3.2.11 Test enclosure, capable of being maintained at the test temperature and if necessary at a relative humidity of (50 ± 5) %.

6.3.3 Procedure

By using the bath (see 6.3.2.10) and the enclosure (see 6.3.2.11), ensure that all the components are maintained at an agreed, uniform temperature; (23 ± 2) °C is commonly used.

Prepare a sample of the adhesive in accordance with the manufacturer instructions, by weighing the components directly into the cartridge (see 6.3.2.1) prior to mixing them, in situ, at a speed of (600 ± 100) r/min for (60 ± 10) s.

The preferred amount of adhesive is (200 ± 20) g.