BRITISH STANDARD

BS EN 14022:2010

Structural adhesives — Determination of the pot life (working life) of multicomponent adhesives

ICS 83.180

National foreword

This British Standard is the UK implementation of EN 14022:2010. It supersedes BS EN 14022:2003 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/52, Adhesives.

A list of organizations represented on this committee can be obtained on request to its secretary.

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

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

Strukturklebstoffe - Bestimmung der Topfzeit (Verarbeitungszeit) von Mehrkomponentenklebstoffen

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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of an identical text or by endorsement, at the latest by August 2010, and conflicting national standards shall be withdrawn at the latest by August 2010.

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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.

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.

As quickly as practical, remove the seal from the threaded end fitting of the cartridge, screw on the calibrated nozzle (see 6.3.2.5), insert the piston and fix the cartridge in the gun (see 6.3.2.6).

As quickly as practical, set the required extrusion pressure.

Rapidly extrude, into one of the foil dishes (see 6.3.2.9), a sufficient quantity of adhesive to ensure the removal of any air trapped in the cartridge together with any unmixed material that can have been retained in the end fitting during stirring.

Note the time and then extrude the freshly, and thoroughly, mixed adhesive at the required pressure for the required period of time. Weigh and note the amount extruded.

The pot life is determined by repeating this procedure, at appropriate intervals, until the quantity of adhesive extruded under standard conditions has fallen to an agreed level. The time that has lapsed, to the moment at which this occurs, is the pot life.

6.4 Method 3: Determination by means of manual application

6.4.1 Principle

A multi-component adhesive is mixed and its pot life found by measuring the length of time after which can no longer be spread by hand.

6.4.2 Apparatus

6.4.2.1 Balance, capable of weighing up to (100 ± 0,1) g.

6.4.2.2 Beaker, squat form, 250 ml, beaker, made of an unreactive material, whose wall thickness does not exceed 1 mm.

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

6.4.2.4 Aluminium plate, clean, degreased (dimensions 400 mm × 200 mm × 1 mm).

6.4.2.5 Spatula, made of an unreactive material.

6.4.2.6 Stopwatch, accurate to ± 1 s.

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

6.4.3 Procedure

By using the bath (see 6.4.2.3) and enclosure (see 6.4.2.7), ensure that all the components are maintained at an appropriate temperature; (23 ± 2) °C is commonly used.

Using the beaker (see 6.4.2.2), mix a sample of the adhesive in accordance with the manufacturer's instructions. Start to measure the time immediately after the mixing procedure begins, and record it.

The preferred amount of adhesive is (50 ± 10) g.

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.

The pot life is determined by noting the time lapse between the moment the mixing begins and the point at which a small quantity of adhesive, taken from the beaker, can no longer be manually spread on the aluminium plate with the spatula.

6.5 Method 4: Determination by means of exothermic reaction temperature

6.5.1 Principle

A multi-component adhesive is homogenized and the pot life is determined as the time by which the batch reaches a defined temperature, the critical temperature (e.g. 40 °C), measured from the beginning of mixing. For products producing less than 40 °C exothermic reaction heat in the defined batch, the maximum temperature is taken as criteria. The pot life depends of the mass and the temperature of the batch as well as that of the room.

NOTE This method is applicable for all multi-component systems.

6.5.2 Apparatus

6.5.2.1 Pot life measuring device, thermocouple with appropriate recording device with an accuracy of ± 1 °C.

6.5.2.2 Beaker, squat form, 200 ml made of an unreactive material, whose wall thickness does not exceed 1 mm.

6.5.2.3 Bath, capable of being maintained within ± 0,2 °C throughout a temperature range between 15 °C and 30 °C.

6.5.2.4 Spatula, made of non-reactive material with adhesive with an angular, not circular end.

6.5.3 Procedure

Before the measurement starts, the critical temperature shall be defined, taking into account the exothermal and processing behaviour of the adhesive system considered. The components of the product shall be maintained at (23 ± 1) °C. The time necessary to maintain the products at this temperature depends on the kind and mass of the batch and shall be determined for each batch before the test starts. The single components shall be weighed into the beaker (see 6.5.2.2) according to the mixing ratio specific for the product. Start recording the time and mix the batch with an angular not circular end of a spatula (see 6.5.2.4) 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.

Dip the temperature probe (see 6.5.2.1) after 1 min into the middle of the batch.

For pot lives of more than 10 min the preferred mass of the batch of the products is 100 g. For products with pot life less than 10 min the preferred mass is (50 ± 10) g. Deviating masses should be given in the test report.

Record the whole time from the beginning of the mixing as well as the temperature starting from the termination of the batch ready to use. The measurement is finished when the critical temperature or the maximum temperature respectively is reached.

The time between the beginning of mixing and the reaching of the critical temperature is taken as pot life. For products which do not reach the critical temperature, the time until the maximum temperature is reached is taken as pot life.

6.6 Method 5: Determination by means of a drying recorder

6.6.1 Principle

The method is appropriate both for two component adhesives and especially for one component adhesives reactive to air humidity (e.g. PUR prepolymers). A thin weight-loaded pin ("needle") is drawn through a thin adhesive layer with an uniform throughput speed. As long as the adhesive is flowable, the trace being drawn by the pin disappears again. After the film formation has begun, a clearly visible trace remains. When the film has dried, the pin lifts out of the trace and continues gliding on the film surface without leaving any marks.

6.6.2 Apparatus

6.6.2.1 Drying recorder, with several (e.g. six or ten) parallel arranged sample holders and just as many motor driven linear moving needle holders.

6.6.2.2 Steel pins, 1 mm diameter fitting into the needle holders and rounded at the front face.

6.6.2.3 Drilled weight stones, which can be attached on the needles, with a mass of 10 g.

6.6.2.4 A gauge, with a length of 0,03 m minimum, with a centimetres and millimetres grading.

6.6.2.5 Beaker, of 50 ml and made of non-reactive and non soluble material, squat form, with a thickness of 1 mm maximum.

6.6.2.6 Spatula, made of non-reactive material.

6.6.2.7 Stopwatch, accurate to ± 1 s.

6.6.2.8 Flat glass ledge with the dimensions 300 mm × 25 mm × 3 mm.

6.6.2.9 Doctor blade, for 250 µm film thickness, with a width of 20 mm.

6.6.2.10 Conditioning chamber, capable to maintain a temperature of (23 ± 2) °C and a relative humidity of (50 ± 5) %, at least category 2.

6.6.2.11 Balance, with a measuring range of 100 g and a maximum uncertainty of ± 0,1 g.

6.6.3 Procedure

The throughput speed of the drying recorder (see 6.6.2.1) is set up using a gauge (see 6.6.2.4) and a stopwatch (see 6.6.2.7), measured and recorded. The throughput speed shall be chosen according to the adhesive's reactivity, so that at the end of the test procedure the film can be expected to be dry. For comparison measurements the same throughput speed should be applied.

The glass ledge (see 6.6.2.8) shall be provided with a starting line (e.g. by using a permanent overhead marker), situated 1 cm to 3 cm apart from one end.

Before beginning the measurement, the adhesive shall be adjusted to (23 ± 1) °C, e.g. by keeping it long enough in the conditioning chamber (see 6.6.2.10).

Two component adhesives are weighed into a beaker (see 6.6.2.5) according to the mixing ratio given by the manufacturer. The preferred total mass of the sample is 25 g. Any other quantity shall be recorded and given in the test report.

Immediately after completion of the mixing the adhesive is put into the doctor blade (see 6.6.2.9) using the spatula (see 6.6.2.6). By means of the doctor blade the adhesive is then spread in a uniform 250 µm thick layer across the whole length of the glass ledge (see 6.6.2.8).

In case the adhesive is supplied in a double cartridge, the adhesive may be squeezed directly into the doctor blade according to the instructions for use.

Also a one component adhesive reactive to humidity can directly be applied from the batch into the doctor blade.

The glass ledge (see 6.6.2.8) is immediately put into the sample receiver of the drying recorder (see 6.6.2.1), the needle is positioned on the starting line by moving the needle holder. The needle shall be positioned at right height such as it only touches the surface of the adhesive. The 10 g weight stone is placed on the top of the needle by means of the drilling and the feed is started immediately.

After completion of the test, the starting point and the end point of the film formation time can be easily determined by means of the trace caused by the needle. Both starting point and end point of the trace are measured from the starting line using the gauge. The film formation time and the film drying time are calculated using the following formulas and the results shall be given in the test report.

$$\text{film formation time, in min} = \frac{\text{Covered distance until formation point, in mm}}{\text{throughput speed, in mm/min}} \quad (1)$$

$$\text{film drying time, in min} = \frac{\text{Covered distance until film drying point, in mm}}{\text{throughput speed, in mm/min}} \quad (2)$$

For highly viscous or thixtropic adhesives the pin can leave a consistent trace yet at the beginning of the test. Thus, there is no need to report the film formation time; this shall be stated in the test report.

7 Expression of results

Express the pot life or working life of the adhesive in hours (h) and/or minutes (min) as the mean of three assessments.

8 Test report

The test report shall include, at least, the following information:

a) a reference to this European Standard and the method reference number;

b) all details necessary for the complete identification of the adhesive, including type, source, manufacturer's code number, and date of manufacture;

c) the quantity of adhesive used after mixing the adhesive for use;

d) the proportions taken when mixing the adhesive;

e) the material and dimensions of the adhesive container;

f) the description of all details necessary for the complete identification of the apparatus used;

g) the temperature of components prior to mixing;

h) where appropriate, the intermediate results and the time intervals between tests;

i) the pot life or the working life of the adhesive;

j) pertinent observations, such as setting, discoloration, separation, caking, or gelling which might have influenced the usability of the adhesive;

k) room temperature (only for drying recorder method, see 4.5);

l) relative humidity (only for drying recorder method, see 4.5);

m) film thickness (only for drying recorder method, see 4.5);

n) throughput speed (only for drying recorder method, see 4.5);

o) any other factors which might have influenced the result

p) the date of the test (and the time of the test if the drying recorder method is used).

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