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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) d'adhésifs multicomposants

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Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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

ISO 10364 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 11, *Products*.

This second edition cancels and replaces the first edition (ISO 10364:1993), which has been technically revised. The revision is based on EN 14022, prepared by the European Committee for Standardization (CEN), Technical Committee CEN/TC 193, *Adhesives*.

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

SAFETY STATEMENT — Persons using this document should be familiar with normal laboratory practice, if applicable. This document does not purport to address all of the safety concerns, 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 requirements.

1 Scope

This International Standard specifies methods for determining the pot life of multi-component adhesives in order to be able to determine whether the pot life conforms to the minimum specified working life required of an adhesive.

NOTE 1 For the purposes of simplification, the term “pot life” is deemed to have the same meaning as “working life” and will be used to represent both throughout this International Standard.

The test methods described are suitable for assessing all multi-component adhesives, and especially epoxy-based and polyurethane-based adhesives, but they are not suitable for some acrylic-based adhesives.

NOTE 2 This International Standard can also be used for assessing non-structural adhesives.

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.

ISO 472, *Plastics — Vocabulary*

ISO 2555, *Plastics — Resins in the liquid state or as emulsions or dispersions — Determination of apparent viscosity by the Brookfield Test method*

ISO 3219, *Plastics — Polymers/resins in the liquid state or as emulsions or dispersions — Determination of viscosity using a rotational viscometer with defined shear rate*

ISO 15605, *Adhesives — Sampling*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 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

4 Principle

This International Standard specifies six methods for the determination of the pot life of multi-component adhesives.

In method 1, the pot life is determined from the increase in viscosity of the adhesive as it reacts. This method is not suitable for the determination of pot lives that are shorter than 5 min.

In method 2, the pot life is determined from the decrease in the mass of mixed adhesive which is extruded in unit time under standard conditions. This method is not suitable for the determination of pot lives that are shorter than 5 min.

In method 3, the pot life is determined as the time taken to reach a point when the mixed adhesive can no longer be spread by hand.

In method 4, the pot life is determined as the time taken to reach a point when the surface of the mixed adhesive is no longer tacky, i.e. it is dry to the touch.

In method 5, the pot life is determined as the time taken by the mixed adhesive to reach a defined temperature, the so-called critical temperature. This method is applicable to all multi-component systems.

In method 6, the pot life is determined as the time taken for the bond strength of the adhesive to drop to a predetermined value or to decrease by a predetermined percentage. This method is not suitable for the determination of pot lives that are shorter than 5 min.

5 Apparatus

5.1 Balance, capable of weighing up to (500 ± 0,2) g for methods 1, 2, 5 and 6 and up to (100 ± 0,2) g for methods 3, 4 and 5.

5.2 Beaker, squat form, made of a material which does not react with the adhesive under test, with a wall thickness which does not exceed 1 mm and with a capacity of 400 ml for methods 1 and 6, 250 ml for method 3, 50 ml for methods 4 and 5 and 200 ml for method 5.

5.3 Spatula, made of a material which does not react with the adhesive under test and with a square, not rounded, end.

5.4 Rotational viscometer, as specified in ISO 2555 or ISO 3219.

5.5 Water bath, capable of being maintained at constant temperature to within ± 0,1 °C for methods 2, 3 and 4 and within ± 0,2 °C for method 5 throughout the temperature range 15 °C to 30 °C.

5.6 Stopwatch, accurate to within ± 1 s.

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

5.8 Disposable plastic cartridges, internal diameter 47 mm, length 210 mm and fitted with a threaded end fitting and a piston, both cartridge and piston being made of a material which does not react with the adhesive under test.

5.9 Stirrer, with a rigid, helical stirrer blade made of a material which does not react with the adhesive under test.

5.10 Stirrer motor, electrically or pneumatically powered, whose speed can be regulated between 0 rpm and 1 000 rpm.

5.11 Extrusion nozzle, made of material which does not react with the adhesive under test, capable of being screwed onto the end fitting of the cartridge (5.8). 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.

5.12 Extrusion gun, powered by compressed air, suitable for use with the cartridge (5.8).

5.13 Pressure gauge, capable of measuring air pressures up to 500 kPa with an accuracy of ± 10 kPa.

5.14 Tared aluminium-foil dishes, of suitable capacity.

5.15 Clean, degreased aluminium plate, dimensions 400 mm × 200 mm × 1 mm for method 3 and 100 mm × 100 mm × 1 mm for method 4.

5.16 Spreader, capable of spreading a layer of adhesive approximately 1 mm thick.

5.17 Thermocouple, accurate to within ± 1 °C, with a suitable recording device.

5.18 Bond strength measuring instrument, suitable for shear specimens or peel specimens (see Note 2 to 6.7).

6 Procedure

6.1 Sampling

The adhesive shall be sampled, prepared and examined in accordance with ISO 15605. For each of the six methods, take at least three samples for testing.

6.2 Method 1: Determination from the change in apparent viscosity

Condition the components of the adhesive separately at (23 ± 2) °C. Then weigh the individual components into a beaker (5.2) in the proportions specified for the particular adhesive under test.

NOTE 1 The preferred amount of mixture is 200 g; however, other quantities can also be used.

Start the stopwatch (5.6) and mix the test sample with the square (not rounded) end of the spatula (5.3) for (60 ± 10) s. Take care that the areas in the angle between the side and bottom of the beaker are well mixed.

On completion of mixing, immediately start measuring the viscosity of the adhesive using the viscometer (5.4).

Take the first viscosity measurement after mixing as the starting point. This measurement is considered as representing the start of the chemical reaction for which the change in apparent viscosity is monitored. Continue making measurements at intervals dependent on the expected pot life.

NOTE 2 The number of measurements, as well as the degree of shear during mixing and during the measurement itself, can have an influence on the viscosity and hence the pot life. Therefore, it is recommended that the measurement interval, as well as the mixing speed and the rotational speed of the viscometer, be selected to suit the adhesive under test.

The pot life of the adhesive is the difference between the 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 from the change in extrusion rate

Using the water bath (5.5) in the test enclosure (5.7), bring each of the components to an agreed, uniform temperature.

NOTE 1 (23 ± 2) °C is commonly used.

Prepare a sample of the adhesive in accordance with the manufacturer's instructions. Weigh the components directly into a cartridge (5.8) and mix them thoroughly *in situ* using a suitable stirrer (5.9) operating at a speed of (600 ± 100) rpm for (60 ± 10) s.

NOTE 2 The preferred amount of mixture is 200 g; however, other quantities can also be used.

As quickly as is practical, remove the seal from the threaded end fitting of the cartridge, screw on the extrusion nozzle (5.11), insert the piston and fix the cartridge in the extrusion gun (5.12).

As quickly as is practical, establish the required extrusion pressure.

Extrude rapidly, through a static mixer into a weighed aluminium-foil dish (5.14), a quantity of adhesive sufficient to remove any air trapped in the cartridge and any unmixed material in the end fitting.

Start the stopwatch (5.6) and extrude the freshly mixed adhesive at the specified pressure for the specified length of time. Reweigh the dish and record the amount of adhesive extruded.

Repeat this procedure at appropriate intervals until the quantity of adhesive extruded under the specified conditions has fallen to an agreed level. The time that has elapsed up to the moment when this occurs is the pot life.

6.4 Method 3: Determination from the ease of manual application

Using the water bath (5.5) in the test enclosure (5.7), bring the components of the adhesive to a suitable temperature.

NOTE 1 (23 ± 2) °C is commonly used.

In a beaker (5.2), mix a test sample of the adhesive in accordance with the manufacturer's instructions.

NOTE 2 The preferred amount of mixture is 50 g; however, other quantities can also be used.

Immediately after mixing, start the stopwatch (5.6).

The pot life is taken as the time which elapses between mixing and the point when a small quantity of adhesive taken from the beaker can no longer be spread manually on an aluminium plate (5.15) with the square (not rounded) end of the spatula (5.3).

6.5 Method 4: Determination from the change in surface tackiness

Using the water bath (5.5) in the test enclosure (5.7), bring the components of the adhesive to a suitable temperature.

NOTE 1 (23 ± 2) °C is commonly used.

In a beaker (5.2), mix a test sample of the adhesive in accordance with the manufacturer's instructions (see, however, Note 3).

NOTE 2 The preferred amount of mixture is 25 g; however, other quantities can also be used.

Immediately after mixing, start the stopwatch (5.6).

Immediately take a small quantity of the mixed adhesive and spread it, by means of the square (not rounded) end of the spatula (5.3) or the spreader (5.16), approximately 1 mm thick on an aluminium plate (5.15).

NOTE 3 If the adhesive is supplied in a twin cartridge, then the adhesive can be extruded directly onto the aluminium plate in accordance with the manufacturer's instructions.

The pot life is taken as the time which elapses between mixing and the point when the surface of the adhesive layer is no longer tacky.

6.6 Method 5: Determination from the reaction temperature

Before the measurement starts, the critical temperature (e.g. 40 °C) shall be defined, taking into account the heat generated by the chemical reaction between the components and the processing behaviour of the adhesive system under test. Then bring the components of the adhesive to (23 ± 1) °C. The time necessary to do this will depend on the type and mass of adhesive concerned, and shall be determined for each adhesive before starting the test. Weigh the individual components into a beaker (5.2) in the proportions specified for the particular adhesive under test.

NOTE For adhesives with pot lives of more than 10 min, the preferred amount of mixture is 100 g. For adhesives with pot lives less than 10 min, the preferred amount of mixture is 20 g. However, other quantities can also be used.

Start the stopwatch (5.6) and mix the test sample with the square (not rounded) end of the spatula (5.3) for (60 ± 10) s. Take care that the areas in the angle between the side and bottom of the beaker are well mixed.

Record the time and, using the thermocouple (5.17), the temperature in the middle of the mixture from the beginning of mixing, which represents the start of the chemical reaction for which the change in temperature is being monitored. Stop taking measurements when the critical temperature (or the maximum temperature — see below) is reached.

Take the time between the beginning of mixing and the point when the critical temperature is reached as the pot life. For products that do not reach the critical temperature, take the time until the maximum temperature is reached as the pot life.

6.7 Method 6: Determination from the bond strength

Condition the components of the adhesive separately at (23 ± 2) °C. Then weigh the individual components into a beaker (5.2) in the proportions specified for the particular adhesive under test.

NOTE 1 The preferred amount of mixture is 200 g; however, other quantities can also be used.

Start the stopwatch (5.6) and mix the test sample with the square (not rounded) end of the spatula (5.3) for (60 ± 10) s. Take care that the areas in the angle between the side and bottom of the beaker are well mixed.

Prepare specimens for determining the bond strength of the adhesive in accordance with any suitable ISO test method.

NOTE 2 For example, the shear specimens and peel specimens described in the following International Standards may be used: ISO 4587; ISO 6237; ISO 8510-2 and ISO 9653.

When preparing the test specimens, apply the adhesive in accordance with the manufacturer's instructions.

Determine the bond strength of the freshly prepared adhesive and also after time intervals appropriate to the pot life of the adhesive as stated by the adhesive manufacturer. The time intervals chosen shall be recorded in the test report.

Take the pot life as the total time which elapses between the time when the adhesive was prepared and the time when the bond strength has dropped to a predetermined value or has decreased by a predetermined percentage.

7 Expression of results

Express the pot life of the adhesive in hours and/or minutes as the mean of the at least three determinations.