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

Adhésifs structuraux — Détermination de la vie en pot (durée d'utilisation) des adhésifs multi-composants

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO document should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 11, *Products,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 193, *Adhesives,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This fourth edition cancels and replaces the third edition (ISO 10364:2015), which has been technically revised.

The main changes are as follows:

— subclause 6.6, Method 5: Determination by control of “snap time”, has been added.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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 determine any regulatory requirements prior to use.

1 Scope

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

The different methods described in this document to measure the property do not necessarily provide identical results.

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

NOTE 1 Some of the methods described in this document can also be suitable for determination of working life of one-part adhesives that react to humidity (e.g. PUR prepolymers).

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

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 using a single cylinder type rotational viscometer method*

ISO 3219-2, *Rheology — Part 2: General principles of rotational and oscillatory rheometry*

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.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
pot life
working life
maximum period of time during which a multi-part adhesive can be used after mixing the components

4 Principle

This document specifies five methods for the determination of the pot life of multi-part 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 by the mixed adhesive to reach a specified temperature, the so-called critical temperature. This method is applicable to all multi-part systems.

In method 4, the working live of low viscose, self-levelling two-part adhesives, or one-part moisture curing adhesives is determined by means of the film formation point and the drying point. A thin weight-loaded pin ("needle") is drawn at a constant speed through a thin layer of the respective adhesive. The time at which the trace, generated by the needle, is no longer levelled out by the still flowing adhesive is defined as film formation time while the point at which the needle lifts out of the adhesive and continues gliding on the film surface without leaving any marks is defined as film drying time.

In method 5, the pot life is determined as the change in string building, called snap time.

5 Apparatus

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

5.2 Beaker, squat shape, plain bottom of appropriate size, made of a material which does not react with the adhesive under test, with a wall thickness which does not exceed 1 mm.

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

5.4 Rotational viscometer, as specified in ISO 2555 or ISO 3219-2.

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

5.6 Stopwatch, uncertainty of measurement to ±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 should be regulated between 0 min^{-1} and 1 000 min^{-1}.

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 3 mm shall be used.

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 uncertainty of measurement of ±10 kPa.

5.14 Tared aluminium-foil dishes, of suitable capacity.

5.15 Surgical blade, with 250 µm film thickness and a length of 20 mm.

5.16 Spreader, capable of spreading a layer of adhesive with a thickness of 1,0 ± 0,2 mm.

5.17 Thermocouple, uncertainty of measurement of ±1 °C, with a suitable recording device.

5.18 Gauge, with centimetre and millimetre grading and a minimum length of 30 mm.

5.19 Drying recorder, with several (e.g. six or 10) sample holders arranged in parallel and accompanying motor driven linear moving needle holders.

5.20 Steel pins, 1 mm in diameter, which fit into the needle holders and are rounded at the front face.

5.21 Drilled weight stone, with a mass of 10 g that shall be attached to the needle.

5.22 Flat glass ledge, 300 mm × 25 mm × 3 mm.

5.23 Conditioning chamber, that is able to maintain a temperature of (23 ± 2) °C and a relative humidity of (50 ± 5) %.

6 Procedure

6.1 Sampling

Each component of the adhesive shall be sampled, prepared and examined in accordance with ISO 15605. For each of the five methods given in 6.2 to 6.6, take at least three samples for testing.

6.2 Method 1: Determination from the change in apparent viscosity

In principle, each rotational viscometer, equipped either with a cylindrical, a cone-plate, or a plate-plate measuring system, capable of handling the expected viscosities can be used. It shall be differentiated between cylindrical measuring systems with a narrow gap between the two coaxial surfaces of which one is rotating and the other remains static and those, like Brookfield having a large, so to say, infinite gap. While the first require typically with app. 10 ml a small amount of adhesive such systems are together with cone-plate and plate-plate measuring systems suitable for adhesives showing a relative short pot life. The latter, like Brookfield, requires, with typically 300 ml, a significantly larger amount and are therefore not suitable for fast-reacting adhesives or adhesives showing a high exotherm. In case the adhesive contains mineral fillers, either cylindrical or a plate-plate system should be used. The use of cone-plate systems should be limited to un-filled adhesives.

The use of a disposable measuring system, especially a disposable static measuring chamber, is recommended to avoid time-consuming cleaning operation.

Condition the components of the adhesive separately using the water bath (5.5) and bring each of the components to a specified uniform temperature. Then, weigh the individual components into a beaker (5.2) of appropriate size in the proportions specified for the particular adhesive under test.

NOTE (23 ± 2) °C is commonly used.

The amount of mixture depends on the amount required by the specific viscometer used. In all cases, a sufficient amount to allow a fast, easy transfer into the measuring system shall be prepared.

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 and avoid mixing-in of air.

Upon completion of mixing, immediately transfer the mixed adhesive into the measuring system avoiding incorporation of air bubbles and start measuring the viscosity of the adhesive using the viscometer (5.4).

As an alternative to manually metering and mixing the individual components, the adhesive can be dispensed directly into the measuring system from a two-part cartridge through a static mixer following the procedure prescribed by the adhesive supplier. Prior to dispensing, the adhesive shall be conditioned in a conditioning chamber. Start the stopwatch at the time when dispense is started.

Depending on the viscometer used, readings should be taken at appropriate intervals or the viscosity-time graph is recorded. Typically, the pot life of the adhesive is specified as the time difference between start of mixing and the time when a specific viscosity, e.g. 100 000 mPas, is reached.

It is also possible to specify the end point as a fixed agreed multiple, e.g. the double of the starting viscosity. In this case, the first viscosity measurement after mixing is taken as the starting point. Differences in the time required for mixing and transferring the adhesive into the measuring system will have, depending on the kinetics of the crosslinking reaction, a more or less effect on the test result.

The number of measurements, as well as the degree of shear during mixing and during the measurement itself, might 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, shall be selected to suit the adhesive under test.

The test report according to Clause 8 shall express the following:

— pot life result expressed in hours/minutes/seconds;

— measuring system used;

— shear conditions;

— either shear rate or shear speed, in mm^{-1};

— continuous or interrupted shear;

— if interrupted, shear time intervals between and duration of shear periods;

— time intervals between the individual readings;

— conditioning temperature;

— mix ratio;

— total amount of mixed adhesive;

— amount of adhesive transferred into the measuring system.

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) min^{-1} for (60 ± 10) s. Take care that the areas in the angle between the side and bottom of the cartridge are well mixed and avoid mixing-in of air.

As an alternative to manually metering and mixing the individual components, the adhesive can be dispensed directly into the measuring system from a two-part cartridge through a static mixer following the procedure prescribed by the adhesive supplier. Avoid entrapping air during the filling process by keeping the orifice of the static mixer always below the surface level of the adhesive.

NOTE 2 The preferred amount of mixture is 200 g; however, other quantities can also be used. Alternatively, it is possible to mix the components outside the cartridge and transfer the mixed material into a cartridge.

As quickly as is practical, remove the stirrer from the cartridge, insert a plunger, and press it down to the adhesive's level allowing air to escape, remove the seal from the threaded end fitting of the cartridge, screw on the extrusion nozzle (5.11), and fix the cartridge in the extrusion gun (5.12).

As quickly as is practical, establish the agreed upon extrusion pressure.

Extrude rapidly, through the nozzle, into a weighed aluminium-foil dish (5.14), about 50 ml of adhesive in order to remove any air trapped and potentially present unmixed material that might be retained in the end fitting during stirring.

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 reaction temperature

Before the measurement starts, the critical temperature (e.g. 40 °C) shall be specified, 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.

6.5 Method 4: Determination by means of a drying recorder

The throughput speed of the drying recorder (5.19) is set using a gauge (5.18) and a stopwatch (5.6), measured and recorded. The throughput speed shall be chosen according to the adhesive's reactivity, such that at the end of the test procedure, the film is dry. For comparison measurements, the same throughput speed shall be applied.

The flat glass ledge (5.22) shall be marked at the starting line (e.g. a permanent marker), situated 1 cm to 3 cm apart from one end.

Before beginning the measurement, the adhesive shall be adjusted to (23 ± 2) °C, e.g. by keeping it long enough in the conditioning chamber (5.23).

Two-part adhesives shall be weighed into a beaker (5.2) 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 onto the surgical blade (5.15) using the spatula (5.3). The surgical blade shall be used to spread the adhesive to a uniform thickness of 250 µm the whole length of the glass ledge (5.22).

Where the adhesive is supplied in a double cartridge, the adhesive can be squeezed directly into the surgical blade according to the manufacturer's instructions.

Likewise, a one-part adhesive, which is reactive to humidity, can be applied directly onto the surgical blade.

The glass ledge (5.22) is put immediately into the sample receiver of the drying recorder (5.19) and the needle is positioned on the starting line by moving the needle holder. The needle shall be positioned at the correct height, such that it just touches the surface of the adhesive. The 10 g weight stone (5.21) is placed on the top of the needle by means of the drilled hole and the feed is started immediately.

After completion of the test, the starting point and the end point of the film formation time shall be determined through the trace of the needle. Both the 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 Formula (1) and Formula (2) and the results shall be given in the test report.

$$t_f = \frac{s_f}{V} \quad (1)$$

$$t_d = \frac{s_d}{V} \quad (2)$$

where

t_f is the film formation time, in min;

t_d is the film drying time, in min;

s_f is the covered distance until formation point, in mm;

s_d is the covered distance until film drying point, in mm;

V is the throughput speed, in mm/min.

For highly viscous or thixotropic adhesives, the pin may leave a consistent trace 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.

6.6 Method 5: Determination by control of "snap time"

Snap time is suitable for two-part silicones. Other two-part adhesives might show similar flow behaviour and can be tested by snap time as well.

The adhesive and all parts shall be adjusted to constant temperature (23 ± 2) °C in advance.

As the snap time strongly depends on material temperature, intensive machine mixing may heat the fluids and shorten snap time, compared to static mixer or stirring by hand. Always use the same dosing and mixing system to compare snap time.

Extrude 30 ml to 75 ml freshly homogeneous mixed adhesive (purge static mixer sufficiently in advance) into a small cup, e.g. made of polyethylene.

Start the timer.

Stir briefly and vigorously with a wooden spatula, after half of the expected pot life. Remove the spatula quickly and focus on the adhesive string building. Leave the spatula standing in the mixed material.

Repeat this operation every 5 min (or less, for fast curing products to get an adequate time scale). If the vigorous stirring is repeated too long and too often, especially at the beginning of the test, the build-up of mechanical strength is disturbed and simulates a longer pot life.

The pot life or snap time is the time from extrusion of the adhesive until the point at which it no longer forms long strings (see Figure 1) when the spatula is removed quickly, but breaks off in short lengths (see Figure 2).

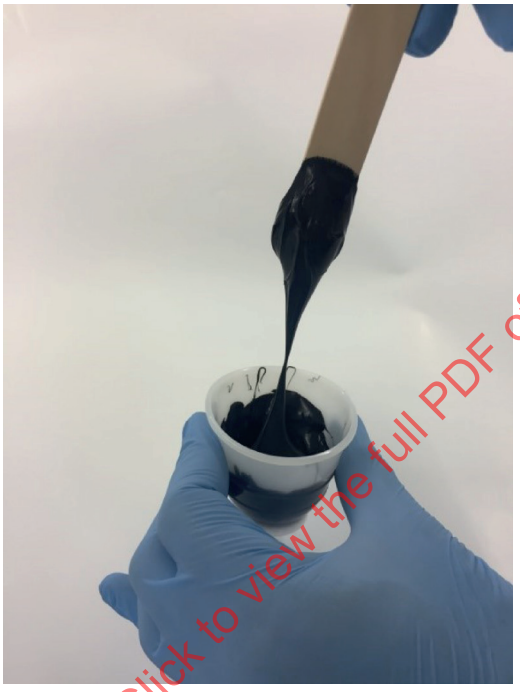

Figure 1 — Material shows paste-like behaviour — Snap time not yet reached

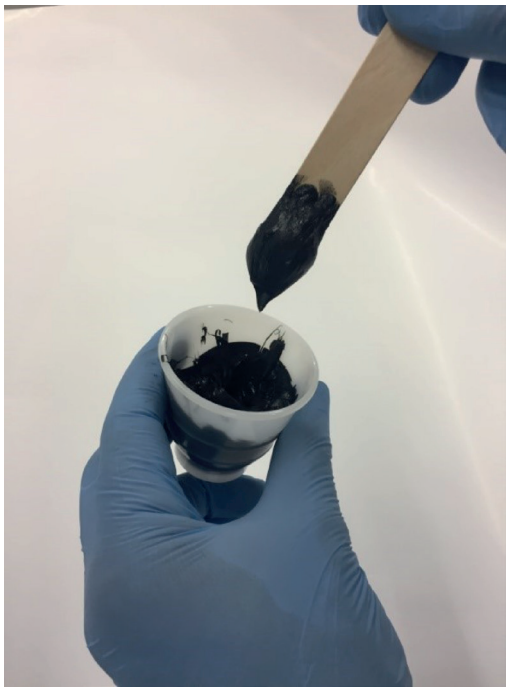

Figure 2 — Material shows rubber-like behaviour — Snap time reached

7 Expression of results

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

8 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 10364, and the reference number of the method used;

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

c) quantity of adhesive mixed for the test;

d) details of the mixing process (i.e. manual mix, mix by electrical stirrer, static mixer tip);

e) proportions taken when mixing the adhesive;

f) capacity of the beaker used to mix the adhesive and the material from which it was made;

g) complete description of the other apparatus used;

h) temperature of the components prior to mixing and any other key temperatures (e.g. the temperature at which the viscosity was determined in the case of method 1);

i) where appropriate, the intermediate results and the time intervals between measurements;

j) pot life of the adhesive;

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

l) any other factors which might have influenced the result;