
**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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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.

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 documents 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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#).

The committee responsible for this document is ISO/TC 61, *Plastics*, Subcommittee SC 11, *Products*.

This third edition cancels and replaces the ~~second edition~~ (ISO 10364:2007), which has been technically revised.

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

SAFETY STATEMENT — Persons using this International Standard should be familiar with normal laboratory practice, if applicable. This International Standard 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-part adhesives in order to be able to determine whether the pot life conforms to the minimum specified working life required of an adhesive.

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. Methods described to measure the property provide different answers. So the results shall be specified with respect to the method used.

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 International Standard can also be suitable for determination of working life of one-part adhesives that react to humidity (e.g. PUR prepolymers).

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

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2 Normative references

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 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

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

4 Principle

This International Standard specifies four 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 defined 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.

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.

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, accurate 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 can 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 accuracy 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 approximately 1 mm thick.
- 5.17 Thermocouple**, accurate to ± 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 can be attached to the needle.
- 5.22 Flat glass ledge**, 300 mm \times 25 mm \times 3 mm.
- 5.23 Conditioning chamber**, of at least category 2 that is able to maintain a temperature of (23 ± 2) °C and a relative humidity of (50 ± 5) %.

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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 four methods given in 6.2 to 6.5, 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, require, 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 the time consuming cleaning operation.

Condition the components of the adhesive separately using the water bath (5.5) and bring each of the components to an agreed, 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 might need to be extended).

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 defined as the time difference between start of mixing and the time when a fixed agreed viscosity, e.g. 100 000 mPas, is reached.

It is also possible to define 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, 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, shall be selected to suit the adhesive under test.

The test report according to [Clause 8](#) should 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 adhesive mixed;
- 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 $(600 \pm 100) \text{ min}^{-1}$ for $(60 \pm 10) \text{ s}$. 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 can 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.

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6.4 Method 3: Determination from the reaction temperature

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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 $(23 \pm 1)^\circ\text{C}$. 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 20g. 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 $(60 \pm 10) \text{ s}$. 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 should be applied.