
**Adhesives — Methods of preparing bulk
specimens —**

**Part 2:
Elevated-temperature-curing one-part
systems**

Adhésifs — Méthodes de préparation d'éprouvettes massiques —

Partie 2: Systèmes monocomposants durcissant à température élevée



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Contents

	Page
1 Scope	1
2 Normative references	1
3 Mould description	1
4 Dispensing the adhesive	2
5 Moulding	4
6 Curing	4
7 Specimen preparation report	4

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 3.

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 part of ISO 15166 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 15166-2 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 11, *Products*.

ISO 15166 consists of the following parts, under the general title *Adhesives — Methods of preparing bulk specimens*:

- *Part 1: Two-part systems*
- *Part 2: Elevated-temperature-curing one-part systems*

Introduction

Accurate values for the mechanical properties of structural adhesives are needed in the design of bonded joints to enable calculations of the stress distribution in the loaded joint and to determine failure criteria for the adhesive material. The use of test methods that employ specimens in the form of a bonded joint for the measurement of these properties is not ideal. This is because the thickness dimension of the bond constitutes the gauge length of the specimen for the measurement of strain and, since the thickness is small (typically < 1 mm), strains cannot be routinely determined with high accuracy. Furthermore, the failure of a joint specimen usually arises under a multiaxial stress state, the complexity of which depends upon many factors such as the geometry of the joint, the dimensions and properties of the adherends and the geometry of any fillets. The failure data derived from these specimens may be informative for design purposes but additional data are needed corresponding to deformation and failure under more simple stress states such as shear and uniaxial tension.

The availability of bulk specimens with suitable dimensions will enable mechanical properties under different loading conditions and environments to be measured using methods developed and standardized for engineering plastics. These are both accurate and relatively inexpensive.

Procedures are described that will enable specimens to be obtained that have structures and properties comparable with those for the adhesive in a bonded joint. Additional tests on joint specimens will be necessary to give information relating to the performance of the bond between the adhesive and the adherend.

Adhesives — Methods of preparing bulk specimens —

Part 2:

Elevated-temperature-curing one-part systems

1 Scope

This part of ISO 15166 describes methods for the preparation of bulk specimens of adhesives.

The procedures described are suitable for elevated-temperature, one-part adhesives such as epoxies. They are not suitable for adhesives where the cure requires the evaporation of solvents or liberation of gas.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 15166. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 15166 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 2818:1994, *Plastics — Preparation of test specimens by machining*.

ISO 3167:—¹⁾, *Plastics — Multipurpose test specimens*.

3 Mould description

3.1 General

The mould shall consist of two plates separated by a frame. For non-free-flowing adhesives, a U-shaped or a closed frame shall be used (see Figure 1).

3.2 Plates

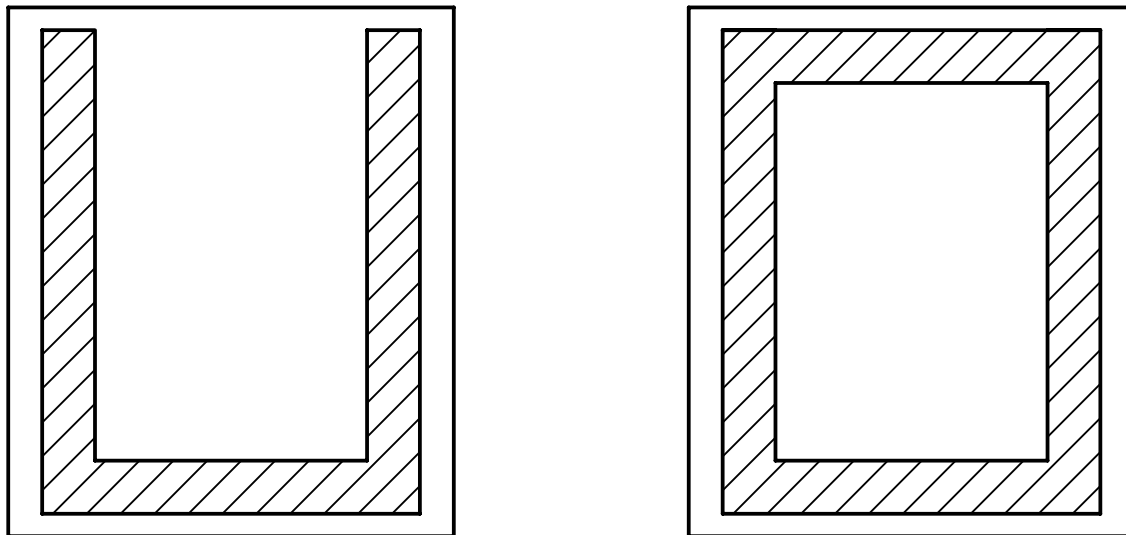
The two plates shall be made of metal and be at least 5 mm thick. This is both to provide a heat sink while curing and to avoid significant bowing of the plates under the forces experienced during moulding.

Copper and brass are suggested as a plate material as they are good thermal conductors. However, when using certain components which are aggressive to copper, alternative metallic materials shall be used.

Make sure that the adhesive does not adhere to the metal plates after cure.

NOTE This can be achieved by applying on the plates a low-surface-energy material such as a polytetrafluoroethylene (PTFE) film, or coating or spraying a release material on the surface of the plates (PTFE sprays need caution so as not to contaminate any other surfaces in the vicinity).

1) To be published. (Revision of ISO 3167:1993)



a) U-shaped frame for non-free-flowing adhesives b) Continuous frame for free-flowing adhesive

Figure 1 — Examples of suitable moulds

For preparing bulk specimens of adhesives that cure very rapidly at ambient temperature and where the evolution of large quantities of heat on curing is likely to lead to different properties of the adhesive, the plates in the mould shall be constructed to allow cooling water to pass through them.

3.3 Frame

In the case of low-viscosity materials which would flow out of the open frame within the time taken to close the mould, it is necessary to use a continuous frame to avoid leakage [see Figure 1b)].

Make sure that the adhesive does not adhere to the frame after cure.

NOTE 1 This can be achieved by applying on the frame a low-surface-energy material such as a PTFE film, or coating or spraying a release material on the surface of the frame. PTFE sprays need caution so as not to contaminate any other surfaces in the vicinity. Alternatively the frame could be constructed from a low-surface-energy plastic such as PTFE.

The height of the frame determines the specimen thickness and shall be uniform to within $\pm 0,5\%$.

NOTE 2 A height of 2 mm to 3 mm is suitable for most tests.

The dimensions of the mould shall be chosen to suit the type of test specimen required.

When bulk specimens are to be prepared having the standard multipurpose test specimen geometry specified in ISO 3167, the minimum length of the cast material shall be greater than 150 mm.

Mechanical test specimens shall be machined in accordance with ISO 2818, stamped or cut from the cast materials.

Alternatively, pre-formed or shaped frames can be used to manufacture specimens of the desired geometry.

4 Dispensing the adhesive

4.1 Preparation of the adhesive

Precautions shall be taken to minimize the volume of any entrapped air in the adhesive since air bubbles present in the material will lead to voids in the final bulk specimen. Entrapped air can be removed from the material by stirring

the adhesive under vacuum. When there is a risk of losing volatile substances in this process, the operation shall be carried out at sufficiently low temperatures to avoid the loss of these substances.

NOTE The adhesive manufacturer should be consulted on whether vacuum degassing is feasible.

4.2 Dispensing

4.2.1 Non-free-flowing adhesives

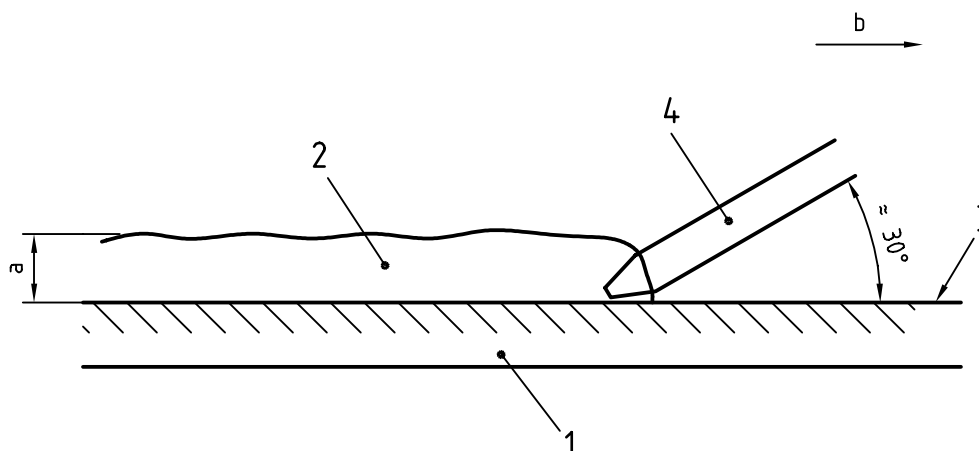
These adhesives shall be dispensed from cartridges with a nozzle. A continuous, steady flow of adhesive is required while dispensing.

The nozzle shall remain within the cast of adhesive at all times to prevent the introduction of air (see Figure 2).

The nozzle shall be drawn slowly down the centre of the mould in a continuous action without lifting from the bottom of the mould or stopping the flow of adhesive (see Figure 2).

NOTE It is recommended that the nozzle be kept at an angle of about 30° to the plate (see Figure 2).

The thickness of the dispensed adhesive shall be greater than the height of the frame.



Key

- 1 Metal plate
- 2 Adhesive cast
- 3 Release agent
- 4 Nozzle

^a Larger than the frame height

^b Direction of nozzle travel

Figure 2 — Casting of the adhesive

4.2.2 Free-flowing adhesives

Where the adhesive is not supplied in a cartridge, the adhesive shall be degassed in a container with a lip to aid the pouring of the adhesive into the mould.

Dispense the adhesive carefully into the centre of the mould so as to avoid introducing air by turbulent flow. Make sure that the volume of the adhesive is less than the volume of the mould cavity.

If the free flowing adhesive is supplied in a cartridge, use the procedure described in 4.2.1 with a continuous frame and make sure that the volume of the adhesive is less than the volume of the mould cavity.

5 Moulding

Close the mould by placing the plate which constitutes the upper part of the mould over the bottom of the U-shaped frame, then slowly press down onto the adhesive using hand pressure to spread the adhesive in the mould until the plate rests on the frame. Apply sufficient clamping pressure between the upper and lower plates of the mould to prevent any excessive leakage of the adhesive during the curing process.

Place the mould in the curing oven such that it is inclined at an angle between 45° and 90° to the horizontal.

6 Curing

Cure the adhesive in accordance with the manufacturer's instructions.

When curing adhesives that undergo a rapid exothermic reaction, it is important to ensure that the temperature rise does not exceed the maximum cure temperature specified by the adhesive manufacturer. When the maximum temperature is exceeded, use the lower temperature of the cure temperature range specified by the manufacturer.

It is unlikely that excessive temperatures will be experienced in mouldings with thicknesses ≤ 2 mm. Where there is a need for bulk specimens with a thickness greater than 3 mm, it is recommended that trial experiments be carried out using a mould in which calibrated temperature sensors are located in the mould cavity so as to measure the temperature in the centre of the moulding. If excessive temperatures are recorded, explore the use of lower cure temperatures and extended cure times. Specimens prepared this way shall be post-cured under recommended cure conditions.

7 Specimen preparation report

The specimen preparation report shall include the following:

- a) a reference to this part of ISO 15166;
- b) identification of the adhesive, including batch number, manufacturer, etc.;
- c) the date and time of casting;
- d) the moulding parameters, including curing temperatures and time, mould details, use of release agents, etc.;
- e) details of any post-cure;
- f) the storage conditions after cure.

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