# INTERNATIONAL STANDARD

ISO 5833

Second edition 2002-05-01

# Implants for surgery — Acrylic resin cements

Implants chirurgicaux — Ciments à base de résine acrylique

Reference number ISO 5833:2002(E)

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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.

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

International Standard ISO 5833 was prepared by Technical Committee ISO/TC 150, *Implants for surgery*, Subcommittee SC 1, *Materials*.

This second edition cancels and replaces the first edition (ISO 5833:1992), which has been technically revised.

Annexes A, B, C, D, E and F form a normative part of this International Standard.

# Implants for surgery — Acrylic resin cements

# 1 Scope

This International Standard specifies the physical, mechanical, packaging and labelling requirements for curing polymerizing radio-opaque and non-radio-opaque resin cements based on poly(methacrylic acid esters). It applies to two types of cement, intended respectively for use with a syringe or in the dough state, for the fixation of internal orthopaedic prostheses and supplied as units containing premeasured amounts of sterile powder and of sterile liquid in forms suitable for mixing at the time of implantation.

This International Standard does not cover the hazards associated with the use of the cement in respect of either the patient or the user of the cement.

All requirements apply to, and all tests are intended to be performed on, the sterile product.

### 2 Term and definition

For the purposes of this International Standard, the following term and definition apply.

#### 2.1

#### unit of cement

one package or vial of sterile premeasured powder component and one package or vial of sterile premeasured liquid component

NOTE For cements in which the radio-opaque agent is supplied separately, the unit of cement includes the vial or package of premeasured radio-opaque powder component.

# 3 Liquid component

### 3.1 Appearance

When inspected by normal or corrected vision, the liquid component shall be free from particles and other contaminants.

### 3.2 Stability

When tested as described in annex A, the flow time of the samples of liquid component shall not increase by more than 10 %.

### 3.3 Accuracy of contents

When measured to an accuracy of  $\pm$  0,1 ml, the volume of the liquid component of each of five units shall be within 5 % of that stated on the package [see 9.1 b)].

# 4 Powder component

### 4.1 General

The powder component includes the polymer particles, initiator agents, and, if applicable, the radio-opaque agent. In some cases, the radio-opaque agent is supplied separately.

## 4.2 Appearance

When inspected by normal or corrected vision, the powder shall be free from agglomerates and extraneous material.

# 4.3 Accuracy of contents

When weighed to an accuracy of  $\pm$  0,1 g, the mass of the powder component of each of five units shall be within 5 % of that stated on the package [see 9.1 b)].

The components used for the determinations specified in 3.3 and 4.3 may be used subsequently for other tests described in this International Standard, with the condition that no mass and/or volume loss for each cement component is produced, and all the requirements of clauses 3 and 4 are satisfied.

# 5 Liquid-powder mixture intended for syringe usage

When determined by the methods given in Tables 1 and 2, the setting properties and the properties of the set cement shall comply with the values given in Tables 1 and 2.

Table 1 — Requirements and test methods for setting properties of liquid-powder mixtures

	Doughing time			Setting time		Maximum temperature		
Mixture	Average	Maximum deviation from average	Test method	Average	Test method	Average	Maximum deviation from average	Test method
	min	min		min		°C	°C	
Syringe usage (see clause 5)	_	_	_	6,5 to 15	Annex C	90	± 5	Annex C
Dough state usage (see 6.1)	≤ 5	1,5	Annex B	3 to 15	Annex C	90	± 5	Annex C

Table 2 — Requirements and test methods for set and polymerized cement

Average comp	ressive strength	Bending	modulus	Bending strength		
	Test method		Test method		Test method	
MPa		MPa		MPa		
≥ 70	Annex E	≥ 1 800	Annex F	≥ 50	Annex F	

# 6 Liquid-powder mixture intended for use in dough state

### 6.1 Setting properties, test methods and requirements

When determined by the methods given in annexes B, C, D, E and F, the setting properties and the properties of the set cement shall comply with the values given in Tables 1 and 2.

### 6.2 Intrusion

When determined as described in annex D, the average intrusion of at least one sample shall be not less than 2 mm.

# 7 Set and polymerized cement

Table 2 presents the requirements and test methods for set and polymerized cement.

# 8 Packaging

**8.1** Each component of the cement shall be packaged and sterilized separately using a suitable method. The liquid component shall be sterilized by ultrafiltration prior to being filled into an aseptic container. Each component shall be packaged in a double-layer sealed container. The components of a single unit of cement shall be further packaged in a container that shall contain the accompanying documentation as described in 9.2 and shall present the information as described in 9.1.

Each component of the cement shall be packaged and sterilized in a double-layer sealed container and then packaged in an outer container which shall contain the accompanying documentation.

- **8.2** In the case of cement being sold with two cement units per container, the requirements of 8.1 shall apply.
- **8.3** The materials of the package should not contaminate or permit contamination of the contents. The packaging should prevent damage to, or leakage of, the contents during transit and storage and should be designed so that it is easy to open and facilitates aseptic presentation of the contents.

### 9 Labelling

### 9.1 Unit package

At least the following information shall appear on the unit package of each cement unit:

- a) a reference to this International Standard (ISO 5833);
- a description of the contents, including the mass of the powder component and the mass or volume of the liquid component, and the generic names of the constituents;
- the name and address of the manufacturer, and the supplier if different from the manufacturer;
- d) a warning that the package contains flammable liquid;
- e) a statement that the contents are sterile and a warning against the use of an opened or damaged package;
- f) an instruction to store the package at a temperature below 25 °C, and away from strong light;
- g) the batch or lot numbers of the liquid and the powder component and the expiry date of the material.

NOTE Legal requirements for labelling may apply in some countries.

## 9.2 Accompanying documentation

At least the following information shall appear on the accompanying documentation (see clause 8):

- a) instructions for handling the components and preparing the cement for use, including details of the equipment needed and an instruction to mix the entire contents of the cement component packages. The instructions shall emphasise the importance of minimizing the entrapment of air;
- b) instructions and recommendations for using the cement, including necessary precautions, and drawing attention to the expiry date marked on the package;
- a statement drawing attention to the toxic, hazardous and irritant properties associated with the handling and use
  of the components and the cement;
- d) a statement that high ambient or component temperatures will decrease, and low ambient or component temperatures will increase, the doughing, working and setting times of the cement;
- e) whether the cement is intended for use with a syringe or in the dough state;
- f) the relative proportions of the powder and liquid components, expressed as a percent mass or volume fraction;
- g) a warning against re-sterilization of either the powder or liquid components;
- h) a statement that, once opened, any pack should be completely used or discarded and not retained for use on a later occasion.

NOTE It is helpful to provide a graphical representation of the effect of temperature on the length of the phases in cement curing, prepared from experimental data on the particular brand of cement.

# Annex A

(normative)

# **Determination of stability of liquid component**

# A.1 Principle

The flow time (viscosity) of the liquid component is determined before and after accelerated ageing by heating, and the increase in flow time after heating is calculated. Two units of the liquid component are tested.

# A.2 Apparatus

- A.2.1 Clean glass U-tube viscometer.
- **A.2.2** Timing device, with an accuracy of  $\pm$  0,1 s.
- A.2.3 Means of heating test specimens.

# A.3 Test conditions

Condition the viscometer and the test specimens at (23  $\pm$  1)  $^{\circ}$ C for at least 1 h before beginning the test. Perform the viscosity measurements at (23  $\pm$  1)  $^{\circ}$ C.

# A.4 Procedure

- **A.4.1** Fill the viscometer in the usual way with the liquid component.
- **A.4.2** Record the flow time taken for the meniscus to fall to the equilibrium level (time  $t_a$ ).
- **A.4.3** Heat an aliquot of the liquid component at  $(60 \pm 2)$  °C for  $(48 \pm 2)$  h in the dark in a closed container, allow it to cool to  $(23 \pm 1)$  °C and to remain at this temperature for at least 1 h.
- **A.4.4** Repeat A.4.1 and A.4.2 and record the flow time (time  $t_{\rm b}$ ).
- **A.4.5** Repeat A.4.1 to A.4.4 on a second unit of liquid component.

## A.5 Calculation and expression of results

Calculate the percentage change  $\Delta t$  in flow time for each unit of liquid component using the expression:

$$\Delta t = rac{t_{\mathsf{b}} - t_{\mathsf{a}}}{t_{\mathsf{a}}} imes exttt{100 } \%$$

# A.6 Test report

- a) a reference to this International Standard (ISO 5833);
- b) identity (including batch or lot number) of the liquid component;
- c) flow times before and after heating;
- d) percentage change in flow time for each unit of cement.

# **Annex B**

(normative)

# Determination of doughing time of liquid-powder mixture of cement intended for dough usage

# **B.1** Principle

The cement is mixed and the time recorded from the beginning of mixing until the mixture is able to separate cleanly from a gloved finger. Either two or four units of cement are tested.

# **B.2** Apparatus

- **B.2.1** Timing device, with an accuracy of  $\pm$  1 s.
- **B.2.2** Unpowdered latex surgical gloves.
- **B.2.3** Equipment, as recommended by the cement manufacturer, for mixing cement.

### **B.3** Test conditions

Condition the mixing equipment and the contents of the cement units at  $(23 \pm 1)$  °C and at a relative humidity (RH) of not less than 40 % for at least 2 h before beginning the test. Perform the test at  $(23 \pm 1)$  °C and a RH of not less than 40 %.

### **B.4** Procedure

- **B.4.1** Mix all the components of a single unit of cement following the manufacturer's instructions. Start the timing device when the liquid is first added to the powder.
- **B.4.2** After approximately 1 min, gently touch the surface of the mixture with a finger gloved with an unpowdered non-water-rinsed latex surgical glove, and observe if fibres form between the cement and the glove as the finger leaves the surface. Clean the glove of all adherent material.
- **B.4.3** Repeat the probing process at maximum intervals of 15 s, gently mixing the cement so as to expose a fresh surface for each probing, ensuring that testing is performed on a fresh surface that has not previously been probed. Record the time at which the gloved finger first separates cleanly from the cement as the doughing time of that mixture.
- **B.4.4** Repeat the process described in B.4.1 through B.4.3 for a second unit of cement.
- **B.4.5** If the two doughing times differ by more than 30 s, repeat B.4.1 to B.4.3 for a further two units of cement.

# **B.5** Calculation and expression of results

Calculate the average doughing time of the two or four determinations made. Round the result to the nearest 15 s and express this as the average doughing time.

# **B.6** Test report

- a) a reference to this International Standard (ISO 5833);
- b) identification (including batch or lot number) of the cement;
- c) average doughing time;
- d) minimum and maximum doughing times.

# **Annex C**

(normative)

# Determination of maximum temperature and setting time of liquid-powder mixture

# C.1 Principle

The exothermic reaction occurring when the powder and liquid components are mixed is monitored and the maximum temperature attained by the bulk is recorded. The setting time is defined as the time taken to reach a temperature midway between ambient and maximum. Either two or four units of cement are tested.

# C.2 Apparatus

- **C.2.1 Mould and plunger**, of dimensions shown in Figure C.1, made of polytetrafluoroethylene, poly(ethylene terephthalate), polyoxymethylene, or high density polyethylene, equipped with a thermocouple of wire diameter approximately 0,5 mm, positioned with its junction (3  $\pm$  0,5) mm above the internal surface of the mould base.
- **C.2.2 Device** capable of converting the thermocouple output signal into temperature readings and making a continuous record of temperature, the thermocouple and converting device having an accuracy of  $\pm$  0,5 °C.
- **C.2.3 C-clamp** or other device, for clamping the plunger and mould together.
- **C.2.4** Timing device, with an accuracy of  $\pm$  0,1 s.
- C.2.5 Equipment, as recommended by the cement manufacturer, for mixing the cement.

### C.3 Test conditions

Condition the mixing and test equipment and the contents of the cement unit at  $(23 \pm 1)$  °C and at a relative humidity (RH) of not less than 40 % for at least 2 h before beginning the test. Perform the test at  $(23 \pm 1)$  °C and at a RH of not less than 40 %.

### C.4 Procedure

- **C.4.1** Record the ambient temperature from the thermocouple in the mould.
- **C.4.2** Mix all the components of a single unit of cement following the manufacturer's instructions.
- **C.4.3** Start the timing device as soon as the powder and liquid come into contact.
- **C.4.4** Fill the mould with approximately 25 g of cement immediately after the mixing is completed. Seat the plunger in the mould and clamp the plunger with the C-clamp to ensure a constant volume. A reinforcing polymer plate may be used along the bottom of the die to prevent warping of the die during polymerization and expansion of the cement. Trim off any cement expelled from the mould.
- **C.4.5** Continue the temperature measurement until shortly after the temperature begins to fall.
- C.4.6 Repeat C.4.2 to C.4.5 for a second unit of cement.
- **C.4.7** If the two maximum temperatures (see C.5.1) differ by more than 10  $^{\circ}$ C, or the setting times (see C.5.2) differ by more than 1 min, repeat C.4.1 to C.4.5 for a further two units of cement.

# C.5 Calculation and expression of results

### C.5.1 Maximum temperature

**C.5.1.1** For each unit of cement, plot the recorded temperatures against time, and record the highest temperature attained to the nearest 1 °C as the maximum temperature for the sample.

An example of a plot is shown in Figure C.2.

**C.5.1.2** Calculate the average value for the two or four determinations. Round the result to the nearest 1  $^{\circ}$ C (rounding values of 0,5  $^{\circ}$ C upwards) and record this as the maximum temperature.

### C.5.2 Setting time

**C.5.2.1** For each unit of cement, determine from the plot made in C.5.1 the setting time,  $t_{\rm set}$ , measured from the beginning of mixing until the temperature of the polymerizing mass reaches the setting temperature  $T_{\rm set}$  defined as:

$$T_{
m set} = rac{T_{
m max} + T_{
m amb}}{2}$$

where

 $T_{\text{amb}}$  is the recorded ambient temperature (see C.4.1);

 $T_{\rm max}$  is the highest temperature attained.

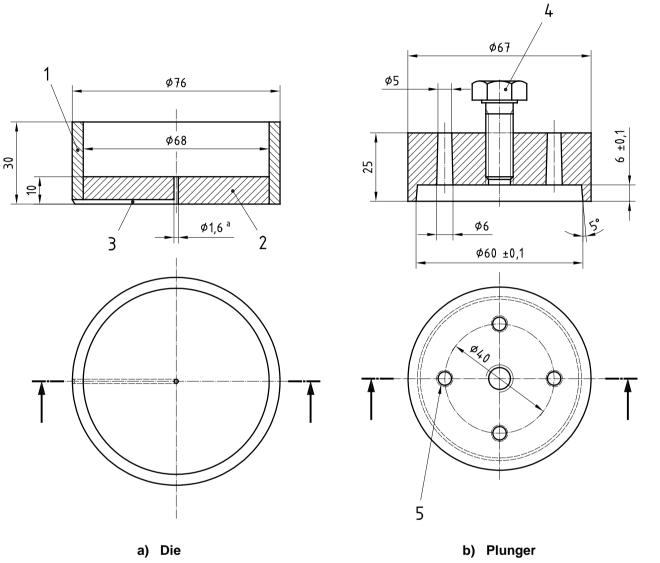
**C.5.2.2** Record the value of  $t_{\text{set}}$  to the nearest 5 s. Calculate the average value of  $t_{\text{set}}$  for the two or four determinations. Round the result to the nearest 15 s, and express this as the setting time.

# C.6 Test report

- a) a reference to this International Standard (ISO 5833);
- b) identification (including batch or lot numbers) of the cement;
- c) average maximum temperature;
- d) individual maximum temperatures;
- e) average setting time;
- f) individual setting times.

Dimensions in millimetres

Tolerance  $\pm$  0,2 mm unless otherwise indicated



# Key

- 1 outer ring
- 2 bottom
- 3 channel for thermocouple
- 4 optional polymer screw of any suitable size to aid removal of test specimen
- 5 four tapered holes for extrusion of excess material
- <sup>a</sup> Diameter of hole for thermocouple.

Figure C.1 — Mould for determination of maximum temperature and setting time

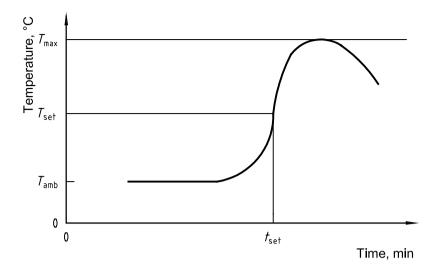


Figure C.2 — Typical curve for determination of maximum temperature and setting time

# Annex D

(normative)

# Determination of intrusion of liquid-powder mixture of cement intended for dough usage

# **D.1** Principle

The cement is mixed and compressed in a mould having a perforated bottom face. After the cement has set, the extent of intrusion of the cement into the perforations is measured. Either one or two units of cement are tested.

# **D.2** Apparatus

- **D.2.1 Mould and plunger**, of dimensions shown in Figure D.1, made of polytetrafluoroethylene, poly(ethylene terephthalate), polyoxymethylene or high density polyethylene.
- **D.2.2** Means of applying a compressive force to the mould.
- **D.2.3** Means of measuring the extent of intrusion to an accuracy of  $\pm$  0,5 mm.
- **D.2.4** Equipment, as recommended by the cement manufacturer, for mixing the cement.

#### D.3 Test conditions

Condition the mixing and test equipment and the contents of the cement units at (23  $\pm$  1)  $^{\circ}$ C for at least 2 h before beginning the test. Perform the test at (23  $\pm$  1)  $^{\circ}$ C.

### D.4 Procedure

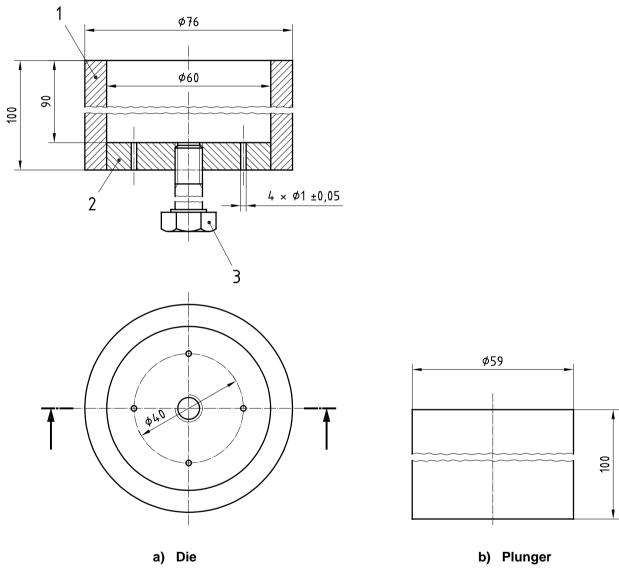
- D.4.1 Mix all the components of a single unit of cement, following the manufacturer's instructions.
- **D.4.2** By means of the procedure given in B.4.2 and B.4.3, determine when the doughing time of the mixture has been reached. Immediately pack the mixture gently into the mould and insert the plunger.
- **D.4.3** 1 min  $\pm$  10 s after doughing time is reached, apply a force of (49  $\pm$  1) N to the plunger for a period of 1 min  $\pm$  2 s. Remove the force and allow the cement to set.
- **D.4.4** Remove the set cement from the mould and measure the extent of intrusion of the cement into each of the four holes in the mould. This may be determined by measuring the depth of the holes not filled with cement, and subtracting this from the initial hole depth. Calculate the average of the four values to the nearest 0,5 mm.
- **D.4.5** If the average intrusion is less than 2 mm, repeat D.4.1 to D.4.4 for a second unit of cement.

# D.5 Test report

The test report shall include at least the following information:

- a) a reference to this International Standard (ISO 5833);
- b) identification (including batch or lot numbers) of the cement;
- c) average intrusion for each sample tested.

 $\label{eq:decomposition} \mbox{Dimensions in millimetres}$   $\mbox{Tolerance} \pm \mbox{0,2 mm unless otherwise indicated}$ 



### Key

- 1 outer ring
- 2 bottom
- 3 optional polymer screw of any suitable size to aid removal of test specimen

Figure D.1 — Mould for determination of intrusion

# Annex E

(normative)

# Determination of compressive strength of polymerized cement

# **E.1** Principle

The cement is mixed and cylinders of cement are cast. The compressive strength of the cylinders is then determined. Five specimens obtained from one unit of cement are tested.

# **E.2** Apparatus

- **E.2.1 Mould**, **end plates** and **removal rod** of dimensions shown in Figure E.1, made of stainless steel, or other device for producing cylinders of cement of appropriate dimensions.
- **E.2.2 C-clamp** or other device for clamping the mould and end plates together.
- E.2.3 24-mesh silicon carbide grinding abrasive and a flat plate.
- E.2.4 Mould release agent (optional).
- **E.2.5** Equipment as recommended by the cement manufacturer, for mixing the cement.
- **E.2.6** Test machine capable of applying and measuring a compressive force of at least 4 kN, equipped to record load versus crosshead displacement.

#### E.3 Test conditions

Maintain the mixing and test equipment at (23  $\pm$  1)  $^{\circ}$ C, for at least 2 h before beginning the test. Perform the test at (23  $\pm$  1)  $^{\circ}$ C.

### E.4 Procedure

- **E.4.1** If required, lightly coat the interior faces of the mould and the inward faces of the end plates with mould release agent.
- **E.4.2** Place the mould on one end plate.
- **E.4.3** Mix all the components of a single unit of cement, following the manufacturer's instructions.
- **E.4.4** For cements intended for dough usage, determine when the doughing time of the mixture has been reached by means of the procedure given in B.4.2 and B.4.3. Within 1 min after this time, slightly overfill each of the cavities of the mould with mixture and place the second end plate on top of the mould. For cements intended for syringe usage, fill the cavities from the syringe and proceed as for dough usage cements. Fill the cavities of the mould with cement immediately after the mixing is completed.
- **E.4.5** Clamp the end plates and the mould together and allow the cement to set. After approximately 1 h remove the clamp and end plates.
- **E.4.6** If using the mould shown in Figure E.1, grind both of the ends of the cement cylinders plane with the faces of the mould by drawing the mould back and forth across a plate coated with silicon carbide abrasive and water. Remove the cement cylinders from the mould by means of the removal rod.

- **E.4.7** If using another type of mould, grind the cement cylinders so as to produce right cylinders of length  $(12 \pm 0.1)$  mm and diameter  $(6 \pm 0.1)$  mm.
- **E.4.8** Condition the cylinders at  $(23 \pm 1)$  °C.
- **E.4.9** At  $(24 \pm 2)$  h after mixing of the cement was begun, measure the average diameter of each test piece, taking the measurements in two mutually perpendicular directions for at least two sections. Place a cylinder in the test machine, without any type of pad between the cylinder and the platen of the test machine. Operate the test machine to produce a curve of displacement against load, using a constant cross-head speed in the range 19,8 mm/min to 25,6 mm/min. Stop the machine when the cylinder fractures or when the upper yield point has been passed.

An example of an idealized load displacement curve is shown in Figure E.2.

**E.4.10** Repeat E.4.9 for each of the cylinders.

# E.5 Calculation and expression of results

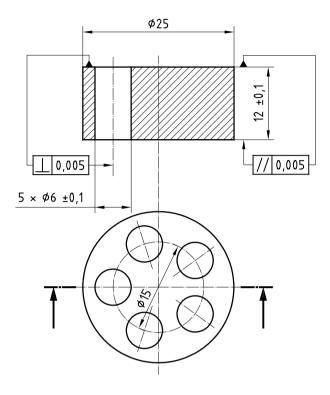
For each cylinder, record the force applied to cause fracture, or the 2 % offset load or the upper yield-point load, whichever occurred first. Divide this force by the original cross-sectional area, in square millimetres, of the cylinder and express the quotient as the compressive strength, in megapascals. Calculate the average compressive strength of the five cylinders.

# E.6 Test report

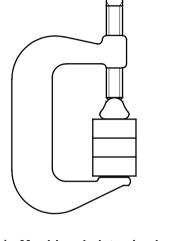
- a) a reference to this International Standard (ISO 5833);
- b) identification (including batch or lot numbers) of the cement;
- c) average compressive strength, in megapascals, and the standard deviation.

Dimensions in millimetres

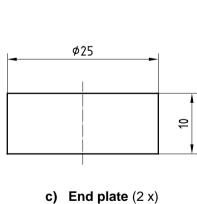
Tolerance  $\pm$  0,2 mm unless otherwise indicated



Perforated plate

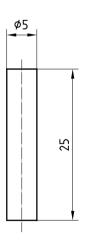


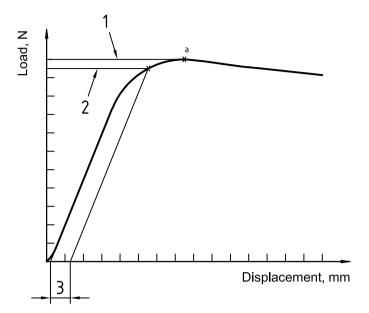
b) Mould and plates in clamp



d) Removal rod

Figure E.1 — Mould components for preparing compressive strength test specimens





# Key

- 1 Ultimate load
- 2 Yield load
- 3 2 % offset
- <sup>a</sup> Upper yield point

Figure E.2 — Idealized curve of load vs. displacement for cement

# **Annex F**

(normative)

# Determination of bending modulus and bending strength of polymerized cement

# F.1 Principle

The cement is mixed and rectangular test specimens are prepared. The bending modulus and bending strength of the specimens are determined by means of a four-point bend test. Five specimens from one unit of cement are tested.

# F.2 Apparatus

- **F.2.1** Bend test machine having a cross-head speed of (5  $\pm$  1) mm/min, equipped with a device for measuring and recording the deflection of the centre of the specimen to an accuracy of  $\pm$  0,05 mm.
- **F.2.2** Four-point bend test rig having the dimensions shown in Figure F.1, with means to prevent misalignment of the test specimen on the supports. The loading points should be of the rolling type and have a suitable diameter to minimize the localized deformation of the specimen during loading. The test rig should be such that equal loads are applied to the loading points.
- **F.2.3** Moulds made of a suitable material to produce five specimens of approximately 75 mm length, 10 mm width and 3,3 mm depth. If preferred, a single mould of 90 mm width may be used and the specimen subsequently cut.
- NOTE Polytetrafluorethylene, poly(ethylene tetrephthalate), polyoxymethylene, high density polyethylene and aluminium alloys have been found to be suitable materials.
- **F.2.4** Flat, smooth plates (two for each mould) of a suitable material and size to cover the upper and lower surfaces of the moulds (see F.2.3 and F.2.3 Note).
- F.2.5 Polyester film.
- **F.2.6** C-clamp(s) or other device(s) for clamping the mould(s) between the top and bottom plates.
- **F.2.7** Equipment as recommended by the cement manufacturer, for mixing cement.

### F.3 Test conditions

Condition the mould(s), plates, mixing equipment and the contents of the cement unit at (23  $\pm$  1)  $^{\circ}$ C for at least 2 h before casting the test specimen(s). Cast the test specimen(s) at (23  $\pm$  1)  $^{\circ}$ C.

## F.4 Procedure

- **F.4.1** Cover the bottom plate(s) of the mould(s) with polyester film. Place the mould(s) on top of the plate(s).
- F.4.2 Mix all the components of a single unit of cement, following the manufacturer's instructions.
- **F.4.3** For cements intended for dough usage, determine when the doughing time of the mixture has been reached by means of the procedure given in B.4.2 and B.4.3. Within 1 min after this time, gently pack the mixture into the mould(s). Fill the mould with cement immediately after the mixing is completed, add a layer of polyester film, add the top plate(s), and clamp the top and bottom plates to the mould(s). For cements intended for syringe use, fill the moulds from the syringe and proceed as for dough-usage cements.

- **F.4.4** After approximately 1 h remove the clamp, the top and bottom plates and the polyester film.
- **F.4.5** Take care to avoid overheating the test specimens. Wet-grind the edges and top faces of the specimens with 400 grade emery paper to the required breadth and thickness. Denote the unground bottom face, as it shall be used as the tensile face during bending.

If single test specimens are prepared in individual mould cavities, remove the specimens from their moulds.

The rectangular test specimens shall have a length of (75  $\pm$  0,1) mm, width of (10  $\pm$  0,1) mm, and thickness of (3,3  $\pm$  0,1) mm.

Condition the specimens at (23  $\pm$  1)  $^{\circ}$ C for (24  $\pm$  2) h prior to testing.

- **F.4.6** Measure the specimen thickness and width to an accuracy of  $\pm$  0,1 mm, taking readings for at least three cross-sections of the specimen, and place it symmetrically in the four-point bend test rig.
- **F.4.7** By means of the bend test machine, increase the force on the central loading points from zero using a crosshead speed of 5 mm/min  $\pm$  1 mm/min, recording the deflection of the specimen as a function of the applied force. Continue to increase the force until the test specimen breaks.
- **F.4.8** Record the deflection occurring at applied forces of 15 N and 50 N, to the nearest 0,05 mm. Record the force at break to the nearest 0,5 N.
- **F.4.9** Repeat F.4.6 to F.4.8 for each of the four remaining test specimens.

# F.5 Calculation and expression of results

### F.5.1 Bending modulus

For each test specimen, calculate the bending modulus, E, in megapascals, from the expression:

$$E = \frac{\Delta Fa}{4fbh^3} \cdot (3l^2 - 4a^2)$$

where

- f is the difference between the deflections under the loads of 15 N and 50 N, in millimetres;
- b is the average measured width of specimen, in millimetres;
- h is the average measured thickness of specimen, in millimetres;
- l is the distance between the outer loading points (60 mm);
- $\Delta F$  is the load range (50 N 15 N = 35 N);
- a is the distance between the inner and outer loading points (20 mm).

Calculate the average value of the bending modulus for the five test specimens, expressed in megapascals, and the standard deviation.

# F.5.2 Bending strength

For each test specimen, calculate the bending strength, B, in megapascals, from the expression:

$$B = \frac{3Fa}{bh^2}$$

where

F is the force at break, in newtons;

b is the average measured width of specimen, in millimetres;

h is the average measured thickness of specimen, in millimetres;

a is the distance between the inner and outer loading points (20 mm).

Calculate the average value of the bending strength for the five test specimens, expressed in megapascals, and the standard deviation.

# F.6 Test report

- a) a reference to this International Standard (ISO 5833);
- b) identification (including batch or lot numbers) of the cement;
- the average and the standard deviation of the values of bending modulus for the five test specimens, expressed in megapascals;
- d) the average and the standard deviation of the values of bending strength for the five test specimens, expressed in megapascals.

## Key

- 1 central loading plunger
- 2 inner loading points
- 3 test specimen
- 4 device for measuring deflection (dial gauge or other device)
- 5 outer loading points
- F force
- $l_1$  distance between outer loading points [(60  $\pm$  1) mm]
- $l_2$  distance between outer and inner loading points [(20  $\pm$  1) mm]
- a All loading points.
- <sup>b</sup> Between any two loading points.

Figure F.1 — Four-point bend test rig

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