INTERNATIONAL STANDARD

ISO 5833

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Implants for surgery — Acrylic resin cements

Implants chirurgicaux — Ciments à base de résine acrylique

Reference number
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Contents

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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.

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.

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

This first edition of ISO 5833 cancels and replaces the first edition of ISO 5833-1, published in 1979 and the planned ISO 5833-2, of which it constitutes a minor revision.

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

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Implants for surgery — Acrylic resin cements

1 Scope

This International Standard applies to radio-opaque and non-radio-opaque cements and specifies physical, mechanical, packaging and labelling requirements for self-curing resin cement based on poly(methacrylic acid esters), of two types intended respectively for use with a syringe or in the dough state for the fixation of internal orthopaedic prostheses and supplied as units containing pre-measured 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 to be performed on, the sterile product.

2 Definition

For the purposes of this International Standard, the following definition applies.

2.1 unit of cement: One package or vial of sterile pre-measured powder component and one package or vial of sterile pre-measured liquid component.

3 Liquid component

3.1 Appearance

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

3.2 Stability

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

3.3 Accuracy of contents

When measured to an accuracy of ± 0,1 ml, the volume of the liquid component of each and every one of five units shall be within 5 % of that stated on the package [see 9.1 a)].

4 Powder component

4.1 Appearance

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

4.2 Accuracy of contents

When weighed to an accuracy of ± 0,1 g, the mass of the powder component of each and every one of five units shall be within 5 % of that stated on the package [see 9.1 a)].

NOTE 1 The components used for the determinations specified in 3.3 and 4.2 may be used subsequently for other tests described in this International Standard.

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.

6 Liquid-powder mixture intended for use in dough state

6.1 Setting properties

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.

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 cured cement

Table 2 sets out the requirements and test methods for set and cured cement.

8 Packaging

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.

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 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;

b) the relative proportions of the powder and liquid components expressed as a mass or volume percent;

c) the name and address of the manufacturer, and the supplier if different from the manufacturer;

d) a statement that the contents are sterile, and a warning against the use of an opened or damaged package;

e) a warning that the package contains flammable liquid;

f) an instruction to store the package in the dark at below 25 °C;

g) the batch or lot numbers of the liquid and the powder component and the expiry date of the material;

h) the number and date of this International Standard (i.e. ISO 5833:1992).

NOTE 2 Legal requirements for labelling may apply in some countries.

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

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

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

Average compressive strength		Bending modulus		Bending strength	
min. MPa	Test method	min. MPa	Test method	min. MPa	Test method
70	Annex E	1 800	Annex F	50	Annex F

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 package. The instructions shall emphasize the importance of minimizing the entrapment of air;

b) instructions and recommendations for using the cement, including necessary precautions;

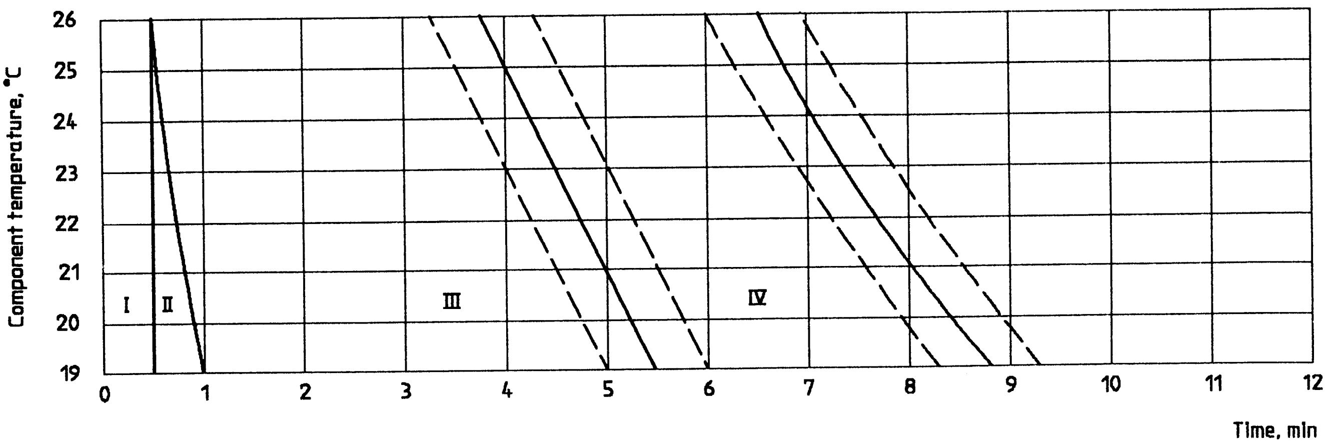
c) a statement drawing attention to the toxic, hazardous and irritant properties associated with the handling and use of the components and the cement;

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) a graphical representation of effect of temperature on the length of the phases in cement curing, prepared from experimental data on the particular brand of cement;

NOTE 3 Examples of graphs are shown in figures 1 and 2.

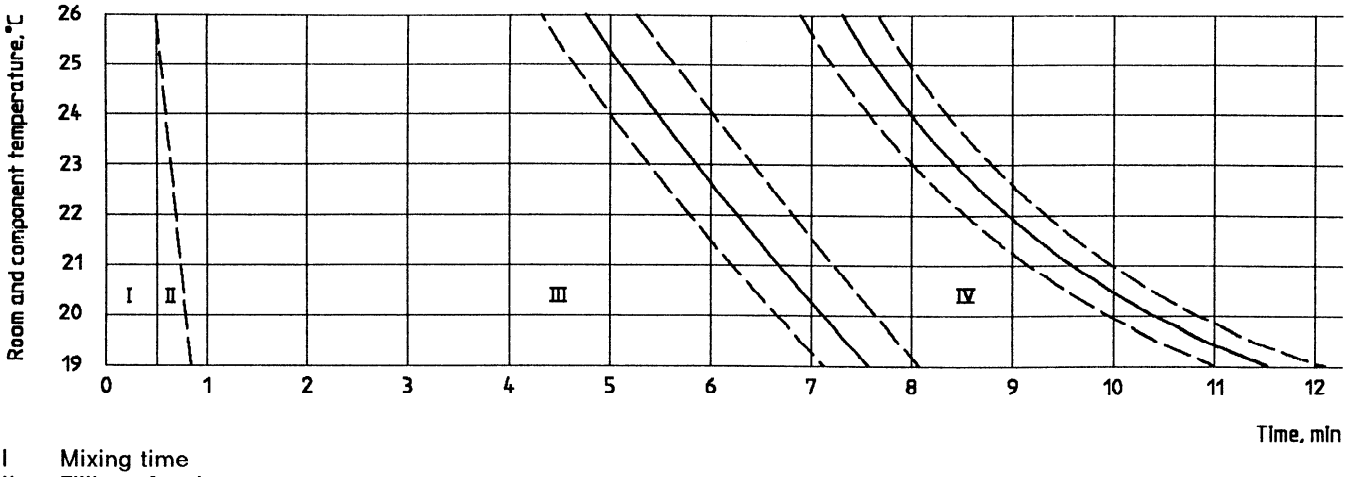
f) whether the cement is intended for use with a syringe or in the dough state.



I Mixing time
II Sticky phase
III Working time
IV Hardening time

A deviation of ± 30 s on working and hardening times may occur. The optimum working temperature has been determined to be 23 °C ± x °C.

Figure 1 — Example of graph showing working data for cement intended for dough usage



I Mixing time
II Filling of syringe
III Working time
IV Hardening time

A deviation of ± 30 s on working and hardening times may occur. The optimum working temperature has been determined to be around 23 °C.

Figure 2 — Example of graph showing working data for cement intended for syringe usage

Annex A
(normative)

Method for determination of stability of liquid component

NOTE 4 Other methods of equivalent accuracy and precision may be used, but the method given in this annex is the referee method in cases of dispute.

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.

A.2 Apparatus

A.2.1 Clean glass U-tube viscometer.

A.2.2 Timing device, of accuracy ± 0,2 s.

A.2.3 Means of heating test specimens.

A.3 Test conditions

Maintain the viscometer and the test specimens at 23 °C ± 1 °C for at least 16 h before beginning the test. Perform the viscosity measurements at 23 °C ± 1 °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_b).

A.4.3 Heat an aliquot of the liquid component at 60 °C ± 2 °C for 48 h ± 2 h in the dark in a closed container; allow it to cool to 23 °C ± 1 °C and to remain at this temperature for at least 16 h.

A.4.4 Repeat A.4.1 and A.4.2 and record the flow time (time t_a).

A.4.5 Repeat A.4.1 to A.4.4 on the liquid component of a second unit of cement.

A.5 Calculations and expression of results

Calculate the percentage change in flow time for each unit of cement using the expression:

$$\text{percentage change} = \frac{t_a - t_b}{t_b} \times 100$$

A.6 Test report

The test report shall include at least the following information:

a) the identity (including batch or lot number) of the liquid component;

b) the flow times before and after heating;

c) the percentage change in flow time for each unit of cement.

Annex B
(normative)

Method for 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.

B.2 Apparatus

B.2.1 Timing device of accuracy ± 0,2 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

Maintain the mixing equipment and the contents of the cement units at 23 °C ± 1 °C and at a relative humidity (R.H.) of not less than 40 % for at least 16 h before beginning the test. Perform the test at 23 °C ± 1 °C and a R.H. 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 probe 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 intervals of 15 s, gently mixing the cement so as to expose a fresh surface for each probing, until the gloved finger separates cleanly from the cement. Record the time at which this first occurs as the doughing time of that mixture.

B.4.4 Repeat B.4.1 to 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

The test report shall include at least the following information:

a) the identity (including batch or lot number) of the cement;

b) the average doughing time;

c) the minimum and maximum doughing times.

Annex C
(normative)

Method for determination of maximum temperature and setting time of liquid-power 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 taken as the time taken to reach a temperature midway between ambient and maximum.

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 mm ± 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 ± 0,5 °C.

C.2.3 C-clamp or other device for clamping the plunger and mould together.

C.2.4 Timing device of accuracy ± 0,2 s.

C.2.5 Equipment as recommended by the cement manufacturer, for mixing the cement.

C.2.6 Thermometer.

C.3 Test conditions

Maintain the mixing and test equipment and the contents of the cement unit at 23 °C ± 1 °C and at a R.H. of not less than 40 % for at least 16 h before beginning the test. Perform the test at 23 °C ± 1 °C and at a R.H. of not less than 40 %.

C.4 Procedure

C.4.1 Record the ambient temperature.

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 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 approximately 25 g of cement into the mould, seat the plunger and trim off any cement expelled from the mould. For cements intended for syringe usage, fill the mould from the syringe and proceed as for dough usage cements.

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 °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.

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

NOTE 5 An example of a plot is shown in figure C.2.