International Standard ISO 5833/1

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Implants for surgery — Acrylic resin cements — Part 1 : Orthopaedic applications

Implants chirurgicaux — Ciments à base de résine acrylique — Partie 1 : Applications orthopédiques

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FOREWORD

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 5833/1 was developed by Technical Committee ISO/TC 150, *Implants for surgery*, and was circulated to the member bodies in November 1977.

It has been approved by the member bodies of the following countries :

Australia	Germany, F. R.	Switzerland
Belgium	India	Turkey
Brazil	Italy	United Kingdom
Canada	New Zealand	USA
Czechoslovakia	South Africa, Rep. of	USSR
France	Spain	

No member body expressed disapproval of the document.

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Implants for surgery — Acrylic resin cements — Part 1 : Orthopaedic applications

1 SCOPE

This International Standard specifies composition, physical performance and packaging requirements for self-curing resins based on poly(methyl methacrylate), and known as "acrylic bone cements", primarily used for the fixation of internal orthopaedic endoprostheses.

This International Standard does not deal with the toxicity or biocompatibility aspects of the cements.

2 FIELD OF APPLICATION

This International Standard is intended to be used only for the purpose of maintaining the uniformity of the product. The results obtained from the tests prescribed herein may not be directly correlatable with the performance of the product when placed in the human body.

3 DEFINITIONS

3.1 unit pack : One package or vial of pre-weighed powder component and one package or vial of pre-measured liquid component, together with any packages of radiopaque additives where such additives are packaged separately.

3.2 doughing time : The time after commencement of mixing at which the mixture ceases to adhere to a standard probe (see 7.4).

3.3 setting time : The time after commencement of mixing at which the rate of increase of temperature due to exothermic reaction is maximum (see 7.6).

3.4 exothermic temperature : The maximum temperature of the mixture due to self-curing in a standard mould (see 7.5).

3.5 intrusion : The distance of flow of the mixture into a standard mould under load (see 7.7).

3.6 indentation : The penetration value in a one day old specimen derived from dial gauge readings on a Rockwell hardness tester (see 7.9).

3.7 recovery : The per cent change in penetration upon removal of the indentation load (see 7.9).

4 COMPOSITION

While a variety of co-polymers and co-monomers may be incorporated, the set cement shall contain poly(methyl methacrylate) as its main ingredient.

5 PHYSICAL REQUIREMENTS

5.1 Liquid component

5.1.1 *Appearance*

The liquid shall be clear and free of any particulate matter or sediment, when viewed against an illuminated screen (see 7.2.1).

5.1.2 *Stability*

The liquid shall show no turbidity or deposit when subjected to the procedure described in 7.3.

5.1.3 *Sterility*

The liquid shall be sterile.

5.2 Powder component

5.2.1 *Appearance*

The powder shall be pourable and free from extraneous materials such as dirt or lint (see 7.2.2).

5.2.2 *Sterility*

The powder shall be sterile.

5.3 Powder-liquid mixture

The properties of the powder-liquid mixture determined by the relevant method under clause 7 shall be in accordance with the values specified in table 1.

TABLE 1 — **Properties of powder-liquid mixture**

Doughing time	Setting time	Exothermic temperature	Intrusion
maximum	range	maximum	minimum
5,0 minutes	4 to 15 minutes	90 °C	2,0 mm

5.4 Cured polymer

The properties of the cured polymer determined by the relevant method under clause 7 shall conform to the values prescribed in table 2.

TABLE 2 — **Properties of cured polymer**

Compressive strength	Recovery	Indentation
minimum	minimum	maximum
70 MPa	60 %	0,2 mm

6 TEST SAMPLES

Nine unit packs of the cement shall be procured to provide sufficient material for all the tests prescribed in this International Standard.

7 METHODS OF TEST

7.1 Test conditions

Unless otherwise specified,

a) all equipment, mixing surfaces and materials shall have been conditioned at a temperature of 23 ± 2 °C and 50 ± 10 % relative humidity; and

b) tests shall be performed under these conditions.

7.2 Inspection

Visual inspection by the naked eye shall be used in determining compliance with the requirements specified in 5.1.1, 5.2.1, 7.3, 7.4, 10.1 and 10.2.

7.2.1 The liquid component shall comply with the requirements of 5.1, 9.1, 9.2, 10.1 and 10.2.

7.2.2 The powder component shall comply with the requirements of 5.2, 9.1, 9.2, 10.1 and 10.2.

7.3 Effect of storage at 60 °C on liquid component

A 1 ml specimen of liquid shall be taken from a package or vial of liquid which has been held for a period of 24 h in a covered vessel at a temperature of 60 ± 2 °C.

After the sample has been allowed to cool to 23 °C, it shall be poured into 20 ml of analytical grade methanol. No turbidity or white deposit shall be observed (see 7.2).

7.4 Doughing time

All the powder and liquid of a single unit pack shall be mixed together as directed by the manufacturer's instuctions [see 10.2 b)]. A stopwatch shall be started at the commencement of mixing and all subsequent times shall be read from this stopwatch.

Approximately 1,5 min after the onset of mixing, the mixture shall be probed gently to a depth of approximately 10 mm with a clean glass rod of approximately 10 mm diameter and having a rounded probing end. The rod shall be wiped clean between determinations. As it leaves the surface, the rod shall be examined (see 7.2) for the formation of fibres between it and the surface of the mix. This process of probing shall be repeated from that time on, at 15 s intervals, until the rod separates cleanly without any material adhering. The time at which this is first observed is denoted as the doughing time. The mixture shall be mixed between determinations to expose fresh material for each probing.

7.4.1 Two separate determinations of the doughing time shall be made.

7.4.2 The two values shall agree within 30 s of each other, otherwise the test shall be repeated on a third unit. If the third determination does not agree within 30 s of one of the other determinations, the material shall have failed the requirements for doughing time.

7.4.3 The doughing time shall be deemed to be the average of all determinations reported to the nearest 15 s (see 5.3.1 and 10.2).

7.5 Exothermic temperature

Within 1 min after expiry of the doughing time, approximately 25 g of the dough made as described in 7.4 shall be gently packed into the plunger cavity of the mould as shown in figure 1. This mould shall be made of polytetrafluoroethylene (PTFE) or other suitable material and be equipped with a thermocouple (wire diameter approximately 0,5 mm) or similar device positioned with its junction in the centre of the mould at a height of 3,0 mm in the internal cavity. The plunger shall be immediately seated with a C-clamp or suitable press to produce the 6,0 ± 0,1 mm specimen height.

After the plunger has been seated, the excess material and the C-clamp or press shall be removed for the remainder of the procedure. The temperature shall be continuously recorded.

Time shall continue to be measured from the onset of mixing the powder with the liquid. Temperatures shall be recorded until cooling is observed. The maximum temperature recorded shall be reported to the nearest 1 °C.

7.5.1 The maximum temperature shall be the average of two separate determinations reported to the nearest 1 °C.

7.5.2 If the difference between the maximum temperatures of the two determinations is greater than 5 °C, the test shall be repeated on a third unit. If the third determination does not agree within 5 °C of one of the other determinations, the material shall have failed the requirements for determination of the exothermic temperature.

7.5.3 The maximum temperature shall be deemed to be the average of all determinations to the nearest 1 °C and shall be reported as the exothermic temperature (see 5.3.1).

7.6 Setting time

From the continuous time versus temperature recording of 7.5, the setting time shall be defined as that time measured from the beginning of mixing when the temperature of the polymerizing mass reaches

$$\frac{T_{max} + T_{ambient}}{2}$$

where

T_{max} is the maximum temperature;

$T_{ambient}$ is the ambient temperature (see figure 2).

The setting time shall be reported to the nearest 15 s.

7.6.1 Two separate determinations of the setting time shall be made.

7.6.2 The two values should agree within 1 min of each other, otherwise the test shall be repeated on a third unit. If the third does not agree within 1 min of one of the other determinations, the material shall have failed the requirement for the setting time.

7.6.3 The setting time shall be deemed to be the average of all determinations, reported to the nearest 15 s [see 5.3.1 and 10.2 a)].

7.7 Intrusion

The mould necessary for this test shall be made of polytetrafluoroethylene (PTFE) or other suitable material and is shown in figure 3. One complete unit pack shall be mixed in accordance with the manufacturer's instructions [see 10.2 b)]. When doughing is achieved, the entire mixture shall be gently packed flat into the mould and the plunger inserted. At 1 min after the doughing time a force of 49 N shall be applied to the top of the plunger for 1 min. The force shall then be removed and the mixture allowed to set.

After setting has occurred, the specimen shall be removed and the length of the intrusion into all four of the 1,0 mm diameter holes of the die shall be measured to the nearest 0,5 mm.

This test shall be performed once. If the material fails to comply with the requirement of 5.3 on the first test, a second test shall be performed and compliance determined on the result of the second test.

7.8 Compressive strength

The test specimens shall be cylinders 12 mm high and 6 mm in diameter. The ends of the specimens shall be flat and smooth and shall be parallel to each other and at right angles to the long axis of the cylinder. An apparatus found convenient for forming these test cylinders is shown in figure 4.

The stainless steel perforated plate shown in figure 4 containing cavities for five specimens shall be placed on the bottom plate. With a spatula the five cavities shall be slightly overfilled from a single unit pack of mixed cement of standard proportions at the commencement of dough time. The top plate shall be positioned and seated firmly with a C-clamp or suitable press. After 1 h the clamp or press shall be removed. At this time the ends of the specimens may be ground flat perpendicular to the axis by the use of a small amount of 240 mesh silicon carbide powder and water. The moulds containing the specimens shall be drawn back and forth across a flat glass plate coated with the abrasive and water. Following this surface treatment, the specimens may be removed from the mould by tapping with the removal rod.

After a time lapse of 24 ± 2 h, the specimen shall be subjected to compressive strength testing.

All specimens shall be tested on any universal testing machine equipped to record load versus deformation. A deformation cross-head speed and a chart speed of 20 or 25,4 mm/min shall be employed. Specimens should be tested without the use of any type of padding material between the specimen and the platens of the machine. The failure load shall be the load at the 2,0 % offset (2,0 % proof stress), upper yield point or at fracture, whichever occurs first.

7.8.1 The compressive strength shall be calculated as the failure load divided by the measured cross-sectional area (approximately 28 mm^2).

7.8.2 The compressive strength shall be deemed to be the average of the results of the five specimens of a given group, reported to the nearest megapascal (see 5.4.1).

7.9 Indentation and recovery

Indentation shall be determined with a Rockwell superficial hardness tester at 24 ± 2 h after start of mixing. The disc made as described in 7.5 shall be utilized. Any flash or other irregularity which would prevent the disc from seating firmly on the hardness tester shall be removed.

At 24 ± 2 h after the start of mixing the specimens shall be subjected, in three separate areas, to a minor static load of 3 kg employing a steel ball of 12,7 mm diameter, and the dial gauge then adjusted to zero. Then a major static load of 30 kg which includes the minor load of 3 kg shall be applied for 10 min, at which time the depth of the indentation shall be read from the dial gauge and shall be recorded to the nearest 0,001 mm as reading *A*. The major load shall then be removed leaving the minor load on the specimen. Ten minutes later the dial gauge shall be read and the value recorded as reading *B*. The averages of reading *A* and reading *B* from the three areas shall be rounded to the nearest 0,01 mm.

7.9.1 The indentation shall be recorded as *A* (avg) and shall comply with the requirements of 5.4.1.

7.9.2 Per cent recovery shall be calculated from the formula

$$\frac{A\ (\text{avg}) - B\ (\text{avg})}{A\ (\text{avg})} \times 100$$

8 PERMISSIBLE VARIATIONS IN QUANTITIES PACKAGED (CONTENTS)

The mass and/or volume of the powder and liquid components shall not deviate by more than 5 % from those stated on the label of the pack in accordance with 10.1 b).

8.1 Mass and volume measurements shall be made on the respective powder and liquid components of five unit packs. These unit packs may be subsequently utilized in any of the non-sterile tests of this International Standard.

8.2 No powder component of a given unit pack shall deviate by more than 2 % from the average mass for the powder of the five unit packs.

8.3 No liquid component of a given unit pack shall deviate by more than 5 % from the average mass or volume for the liquid of the five unit packs.

8.4 Where a radiopaque material is supplied for addition to the powder at the discretion of the surgeon, the mass of the radiopaque material shall not deviate by more than 15 % from the value stated on the label of the pack in accordance with 10.1 b).

9 PACKAGING

9.1 The product shall be supplied in properly sealed containers made of materials that will not contaminate or permit contamination of the contents. The containers shall be packaged so as to prevent damage or leakage during shipping and storage. The product shall be packaged so as to permit the sterile transfer of contents to the sterile field.

9.2 The contents shall be easily accessible, easy to open and convenient to mix in the operating room. The entire contents of the unit pack (both powder and liquid) must be mixed to achieve recommended proportions.

10 LABELLING

10.1 On the label

The following minimal information shall appear on the label of each unit pack :

a) Identification of manufacturer and distributor.

b) Mass and/or volume of both the liquid and the powder, and the mass of any radiopaque material supplied separately.

c) Constituents of the powder and liquid in terms of mass or volume per cent. This shall include generic names of co-polymers, chemical activators, plasticizers, monomers, chemical promoters, stabilizers, cross-linking agents and any other ingredients such as radiopacifiers, gels, fillers or antibiotics.

d) A statement that the contents are sterile and that sterility is guaranteed only if the containers are undamaged.

e) The following, or equivalent, warnings :

1) Storage of the liquid component above 30 °C or in direct sunlight should be avoided to prevent premature polymerization.

2) The liquid is flammable.

f) The words "Sterilization must only be performed by the manufacturer. Re-sterilization of any of the components should not be attempted under any circumstances".

g) Clear identification as to the batch or lot number of each component of each unit pack.

10.2 On the product identification insert

The following information shall appear on the product identification insert :

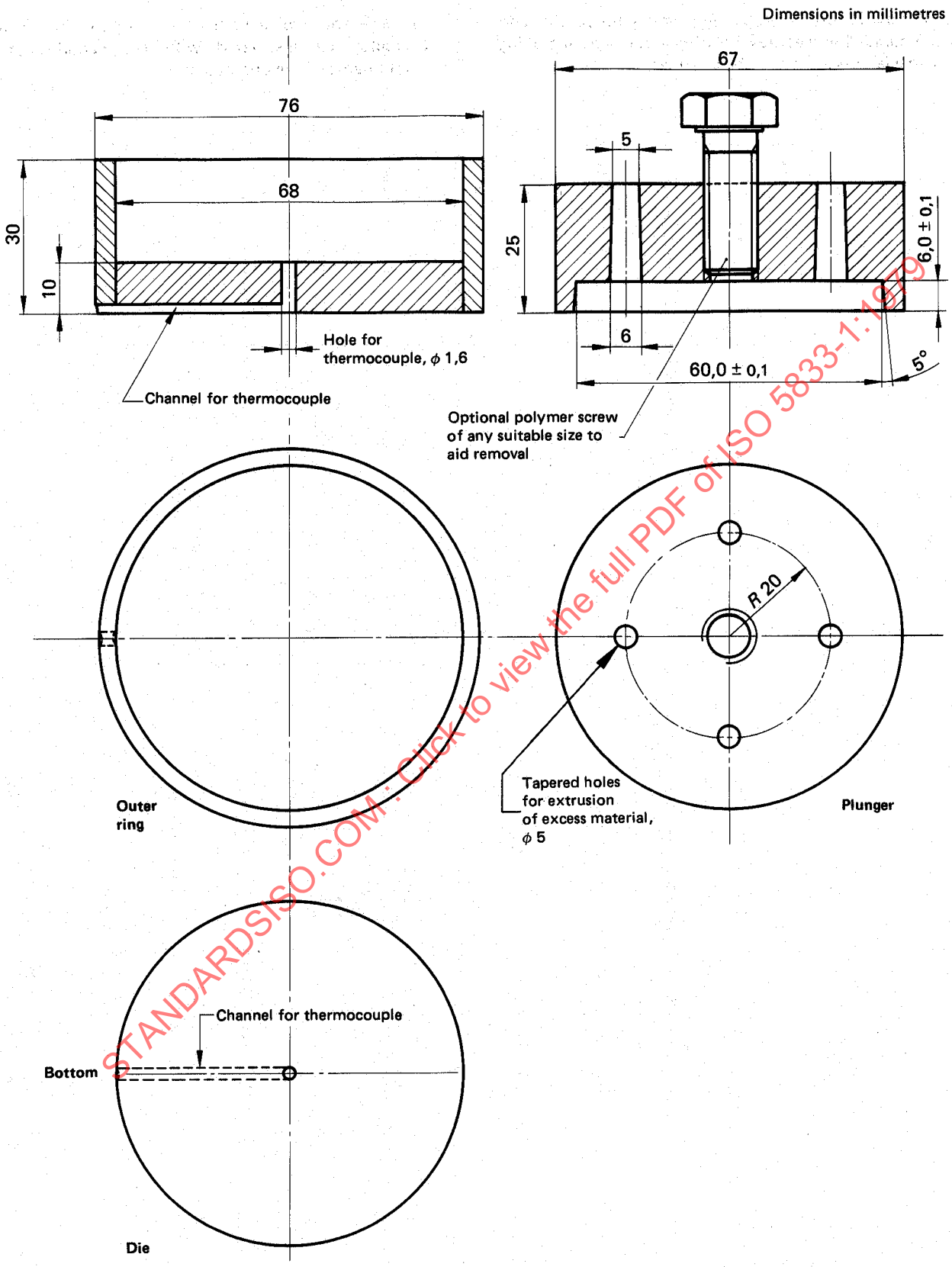
a) The approximate doughing and setting time at 23 °C, together with a statement that any temperature above 23 °C will cause shorter doughing, working and setting times. Also, any lowering below 23 °C of either the ambient temperature or that of the material will increase doughing, working and setting times.

b) Adequate and accurate instructions for storing. Instructions shall include a directive to mix all of the powder with all of the liquid of a single unit pack. Temperatures and procedure required to mix the material along with recommended suitable mixing vessels and mixing tools shall be given. The manufacturer should indicate approximate working times of the material.

c) A description of proper techniques for placing and recommended procedures for using the cement, including any special precautions to be observed.

d) Reference to any toxic, hazardous or irritating characteristics associated with the handling of the components of the uncured mix.

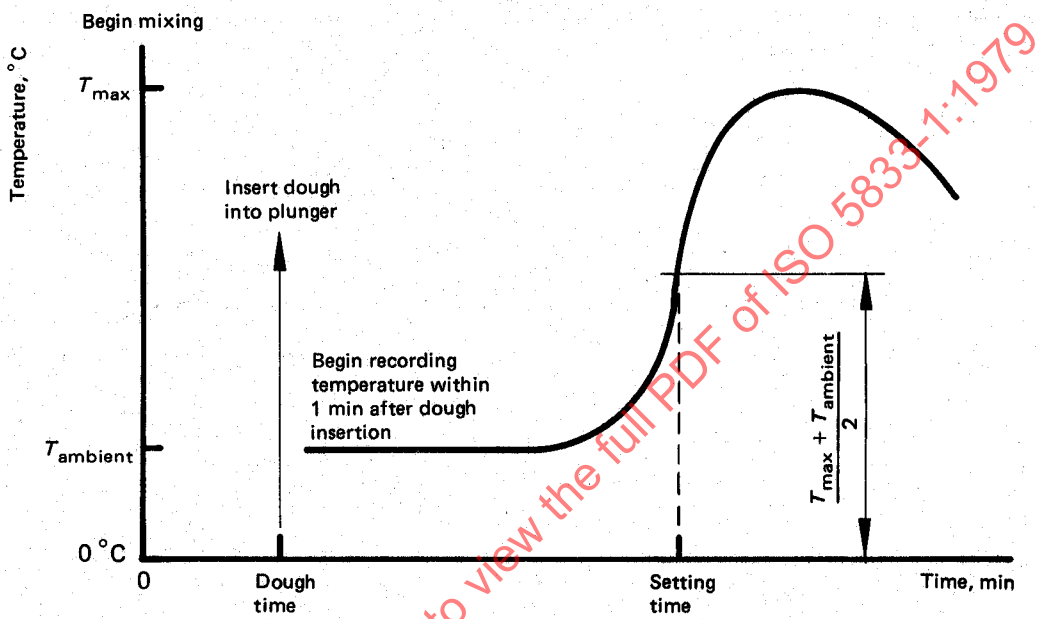
Dimensions in millimetres



Material for all components : Polytetrafluoroethylene, poly(ethylene terephthalate), polyoxymethylene, or high density polyethylene.

All dimensions ± 0,2 unless otherwise specified.

FIGURE 1 — **Exothermic heat mould**



NOTE : $\frac{T_{max} + T_{ambient}}{2} = T_{ambient} + \frac{T_{max} - T_{ambient}}{2}$

FIGURE 2 – **Schematic diagram of exothermic temperature determination**