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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Dentistry — Resin-based filling materials

Art dentaire — Produits d'obturation à base de résines synthétiques

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Reference number
ISO 4049 : 1988 (E)

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 approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 4049 was prepared by Technical Committee ISO/TC 106, *Dentistry*.

This second edition cancels and replaces the first edition (ISO 4049 : 1978), of which it constitutes a technical revision (see the Introduction).

Introduction

This second edition of ISO 4049 takes account of the considerable volume of technical information which has accumulated since the first edition was published in 1978. Some of the tests in the first edition have been omitted and others added for the reasons given below.

This International Standard does not cover requirements for materials intended for the restoration of occlusal surfaces or those intended to prevent caries. In order to make this clear, a classification system has been introduced (see clause 3). This International Standard therefore covers class B materials, i.e. materials other than those intended for occlusal surfaces, and manufacturers are now required to classify their materials accordingly. Furthermore, in order to assist the purchaser, manufacturers are now also required (see clause 8) to describe the filler particle size range and the principal component of the resin base.

The possibility was considered that materials might be classified by filler loading or its corollary, water uptake, and solubility of the resin phase. However collaborative testing revealed considerable overlapping of these properties in "conventional" and "microfine" materials and such a classification was not adopted.

Resin-based restorative materials activated by external energy are now well established and requirements for these materials are therefore included. As the materials do not have an unlimited working time in the dental surgery, a test for sensitivity to ambient light has been included (see 7.6).

Working and setting times of chemically cured materials cannot be determined accurately because of their rapid setting and varying viscosities after mixing. The test in the first edition of this International Standard, using an oscillating rheometer, had poor sensitivity and gave results that could not be correlated with "clinical" working time. In this second edition the test has been replaced by one which is simple and widely applicable.

The flexural strength test (see 7.8) has been aligned with the test used for denture-base polymers by requiring that the specimen be immersed in water during testing. A requirement relating to modulus-dependent flexural strength has been included with the limiting value set to reveal conventional composites with poor filler/resin bonding.

Requirements have been included for materials claimed to be radio-opaque (see 4.5).

Although tests are not included in this second edition for determining non-mandatory or optional properties, such as polymerization shrinkage, it is hoped to do so in a later edition. At present more than one test may be used to determine a single such property which makes true comparisons impossible and confuses the purchaser.

The test for depth of cure of external-energy-activated materials will be reviewed and revised, if necessary, when more data become available.

Specific qualitative and quantitative requirements for freedom from biological hazard are not included in this International Standard, but it is recommended that reference should be made to ISO/TR 7405 when assessing possible biological or toxicological hazards.

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Dentistry — Resin-based filling materials

1 Scope

This International Standard specifies requirements for dental resin-based restorative materials supplied in a form suitable for mechanical mixing, hand-mixing, or external energy activation, and intended for use primarily for the direct restoration of class III, IV and V cavities in the teeth, i.e. class B materials (see clause 3).

This International Standard does not cover requirements for materials intended for the restoration of occlusal surfaces, i.e. class A materials (see clause 3), or materials intended to prevent caries.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3665 : 1976, *Photography — Intra-oral dental radiographic film — Specification*.

ISO/TR 7405 : 1984, *Biological evaluation of dental materials*.

ISO 7491 : 1985, *Dental materials — Determination of colour stability of dental polymeric materials*.

ISO 8601 : 1988, *Data elements and interchange formats — Information interchange — Representation of dates and times*.

3 Classification

For the purposes of this International Standard, dental resin-based restorative materials are classified as follows :

Class A : Materials claimed by the manufacturer as suitable for the restoration of cavities involving occlusal surfaces

Class B : All other materials

Type 1 : Chemically-cured materials, i.e. those materials where setting is effected by mixing an initiator and activator

Type 2 : External-energy-activated materials, i.e. those materials where setting is effected by the application of energy, such as blue light

4 Requirements

4.1 Biocompatibility

See the Introduction for guidance on biocompatibility.

4.2 Physical and mechanical properties

4.2.1 General

If the material is supplied by the manufacturer in pre-coloured standard shades, each shade shall be capable of satisfying the

requirements specified in 4.3 appropriate to the material type. If the material is supplied for "tinting" or "blending" to the user's prescription, the material shall comply with the requirements both when used alone and when used with the maximum recommended proportion of tinter or blender [see 8.3 g)].

4.2.2 Minimum working time, type 1 materials

The working time for type 1 materials, determined in accordance with 7.4, shall be not less than 90 s.

4.2.3 Setting time, type 1 materials

The setting time for type 1 materials, determined in accordance with 7.5, shall be not more than 5 min.

4.2.4 Sensitivity to ambient light, type 2 materials

When tested in accordance with 7.6, there shall be no detectable change in the consistency of any of the three samples of type 2 materials after being exposed to the test light for 60 s.

4.2.5 Depth of cure, type 2 materials

When determined in accordance with 7.7, the depth of cure of type 2 materials shall be not less than 2 mm, and, in any event, no more than 0,5 mm below the value stated by the manufacturer.

NOTE — This test is considered to represent about twice the optimal conversion of monomer to polymer.

4.2.6 Flexural strength

The flexural strength of type 1 and type 2 materials, determined in accordance with 7.8, shall be not lower than the value of $N = [(flexural\ modulus \times 0,002\ 5) + 40]$ MPa, and, in any event, not lower than 50 MPa.

4.2.7 Water absorption and solubility, types 1 and 2 materials

When determined in accordance with 7.9, the water absorption of type 1 and type 2 materials shall not be greater than $50\ \mu\text{g}/\text{mm}^3$ and the solubility shall not be greater than $5\ \mu\text{g}/\text{mm}^3$.

4.3 Shade

When the material is assessed in accordance with 7.10 by three observers, the shade of the set material shall match closely that of the manufacturer's shade guide. If a shade guide is not supplied by the manufacturer, samples from two further batches shall be taken for comparative purposes; all three samples shall show no more than a slight change in colour.

4.4 Colour stability

When the material is assessed in accordance with 7.10, none of the three observers shall observe more than a slight change in colour.

4.5 Radio-opacity

If the manufacturer claims that the material is radio-opaque [see 7.2.3.2 b)], the radio-opacity, determined in accordance with 7.11, shall be greater than that of the same thickness of aluminium.

5 Sampling

The test sample shall consist of retail packages from the same batch containing enough material to carry out the specified tests, plus an allowance for repeat tests, if necessary.

NOTE — 50 g should be sufficient, but two further samples of different batches may be required for the shade test (see 4.3).

6 Preparation of test specimens

NOTE — For the preparation of type 2 materials, reference should be made to the manufacturer's instructions [see 8.3 e)] which will state the external energy source or sources recommended for the materials to be tested. Care should be taken to ensure that the source is in a satisfactory operating condition.

Mix or otherwise prepare the material in accordance with the manufacturer's instructions and the test conditions specified in 7.2.

7 Test methods

7.1 General reagent and apparatus

7.1.1 Water

Water prepared by

- multiple distillation, or
- distillation followed by de-ionization, or
- distillation followed by reverse osmosis.

7.1.2 Glass slides/plates

Quartz glass plates, 2 mm thick, are required for use with type 2 materials being cured by ultraviolet light only. For type 1 materials and type 2 cured by blue light, standard glass microscope slides may be used.

7.2 Test conditions

Unless specified otherwise by the manufacturer, prepare and test all specimens at $(23 \pm 1)^\circ\text{C}$. Control the relative humidity to ensure that it remains greater than 30 % at all times. If the material was refrigerated for storage, allow sufficient time for it to attain $(23 \pm 1)^\circ\text{C}$.

7.3 Inspection

Visually inspect to check that requirements specified in clause 8 have been met.

7.4 Working time, type 1 materials

Dimensions in millimetres

7.4.1 Apparatus

Thermocouple apparatus, as shown in figure 1.

The apparatus consists of a piece of polyethylene tubing, (A), located on a block of polyamide or similar material, (B), which has a hole into which is inserted a stainless steel tube, (C), containing a stabilized thermocouple (D).

The tube (A) is 8 mm long, 4 mm in internal diameter and has a wall thickness of 1 mm. The locating part of block (B) is 4 mm in diameter and 2 mm high. When assembled the two components form a specimen well 6 mm high $\times$ 4 mm in diameter. In order to facilitate removal of the specimen after testing, the thermocouple (D) has a conical tip which protrudes 1 mm into the base of the specimen well. The tolerances on the above-mentioned dimensions are $\pm 0,2$ mm.

The thermocouple consists of wires ($0,2 \pm 0,05$) mm in diameter, made of a material (e.g. copper/constantan) capable of registering temperature changes in a specimen of setting material to an accuracy of $0,1$ °C. The thermocouple is connected to an instrument (e.g. voltmeter or chart recorder) capable of recording the temperature to that accuracy.

7.4.2 Procedure

Prepare the test material in accordance with the manufacturer's instructions (see 8.3) and start timing from the moment mixing is begun. Maintain the mould at (23 ± 1) °C and, 30 s after the start of mixing, place the mixed material in the mould and record the temperature, t_1 , of the material. Maintain the apparatus at (23 ± 1) °C and continuously record the temperature of the material until the peak temperature is passed.

NOTE — A typical recording trace is shown in figure 2. As soon as the material is inserted into the mould, the temperature falls slightly until it becomes steady at t_0 and then starts to increase. The point at which the temperature begins to increase denotes the start of the setting reaction and, therefore, the end of the working time. This point should be determined by drawing a proof line at $t_0 \pm 0,01$ °C and recording T_w at the point of intersection with the trace. The results are extremely temperature-dependent and slight variations within the permitted temperature range will cause variations of several seconds.

Record the time, T_w , from the start of mixing until the temperature starts to increase.

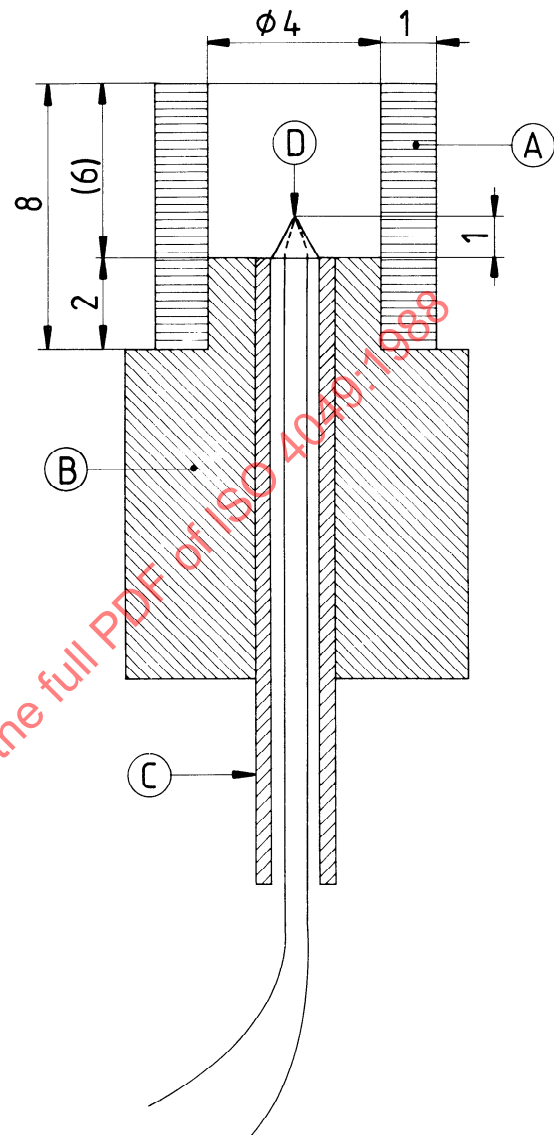
Carry out five determinations.

7.4.3 Interpretation of results

If at least four of the times obtained are longer than 90 s, the material is deemed to have complied with the requirement of 4.2.2.

If three or more of the times are shorter than 90 s, the material is deemed to have failed.

If only three times are longer than 90 s, repeat the whole test. If three or fewer times are longer than 90 s on the second occasion, the material is deemed to have failed the whole test.

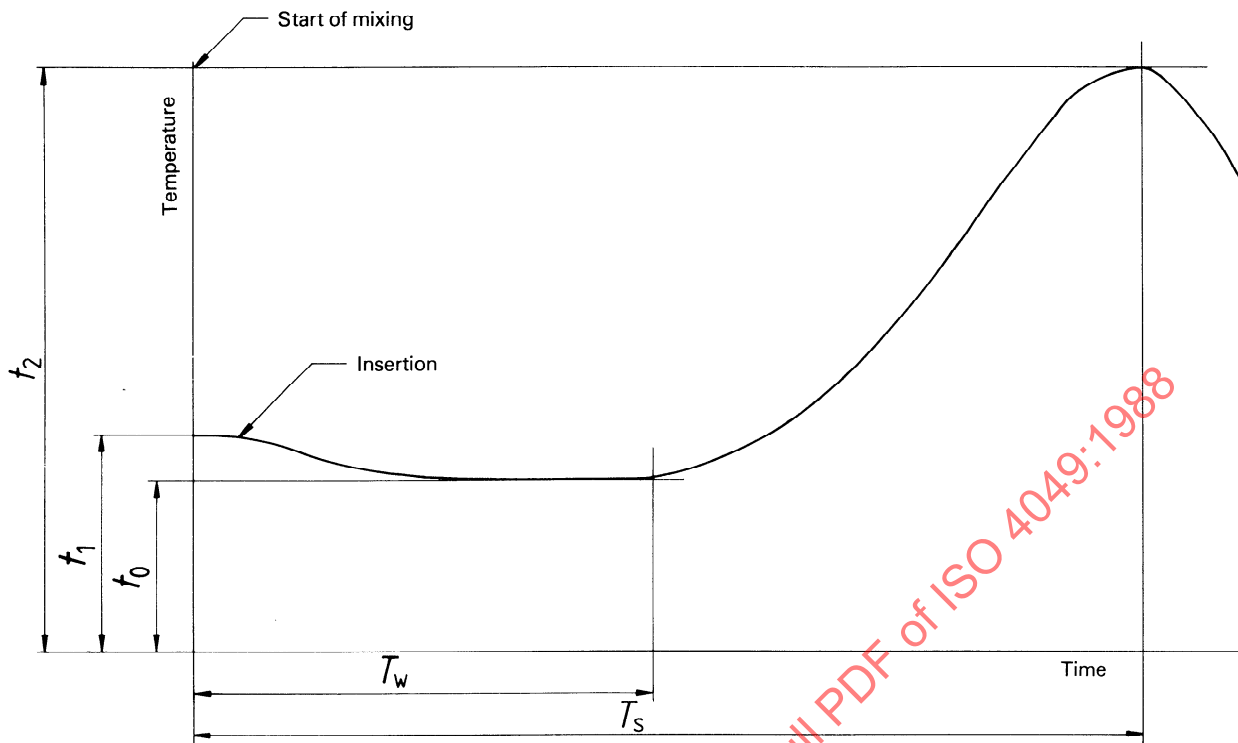


Key (see also 7.4.1)

- (A) Polyethylene tubing
- (B) Polyamide block
- (C) Stainless steel tube
- (D) Thermocouple-cone of solder

NOTE — Dimensional tolerances shall be $\pm 0,2$ mm.

Figure 1 — Apparatus for determination of working and setting times



NOTE — The typical recording trace illustrated shows the temperature at the time of insertion, t_1 , the slight temperature drop immediately after insertion, t_0 , and the initial time of temperature increase, T_w , which denotes the start of the setting reaction and, therefore, the end of the working time. At $(37 \pm 1)^\circ\text{C}$, the peak temperature t_2 is noted to measure T_s , the setting time.

Figure 2 — Typical recording trace showing temperature changes with time for determination of working and setting times

7.5 Setting time, type 1 materials

7.5.1 Apparatus

Thermocouple apparatus, as specified in 7.4.1.

7.5.2 Procedure

Repeat the procedure specified in 7.4.2, but maintain the apparatus at $(37 \pm 1)^\circ\text{C}$.

Measure the time from the start of mixing until the maximum temperature is reached. Record this time, T_s , as the setting time (see figure 2).

7.5.3 Interpretation of results

If at least four of the times obtained are shorter than 5 min, the material is deemed to have complied with the requirement of 4.2.3.

If three or more of the times are longer than 5 min, the material is deemed to have failed.

If only three of the times are shorter than 5 min, repeat the whole test. If one or more times are longer than 5 min on the second occasion, the material is deemed to have failed the whole test.

7.6 Sensitivity to ambient light, type 2 materials

7.6.1 Apparatus

7.6.1.1 Xenon lamp or radiation source of equivalent performance, with colour conversion and ultraviolet filters inserted.

The colour conversion filter shall be 3 mm thick hardened glass and shall have an internal transmittance which matches within $\pm 10\%$ that shown in figure 3.¹⁾

1) A suitable filter which corresponds to the internal transmittance shown in figure 3 and which is available commercially is the FG 15, hardened, rough-polished, 3 mm thick (supplied by Schott Glaswerke, Postbox 2480, D-6500, Mainz 1, Germany, F.R.). This information is given for the convenience of the users of this International Standard and does not constitute an endorsement of this product by ISO.

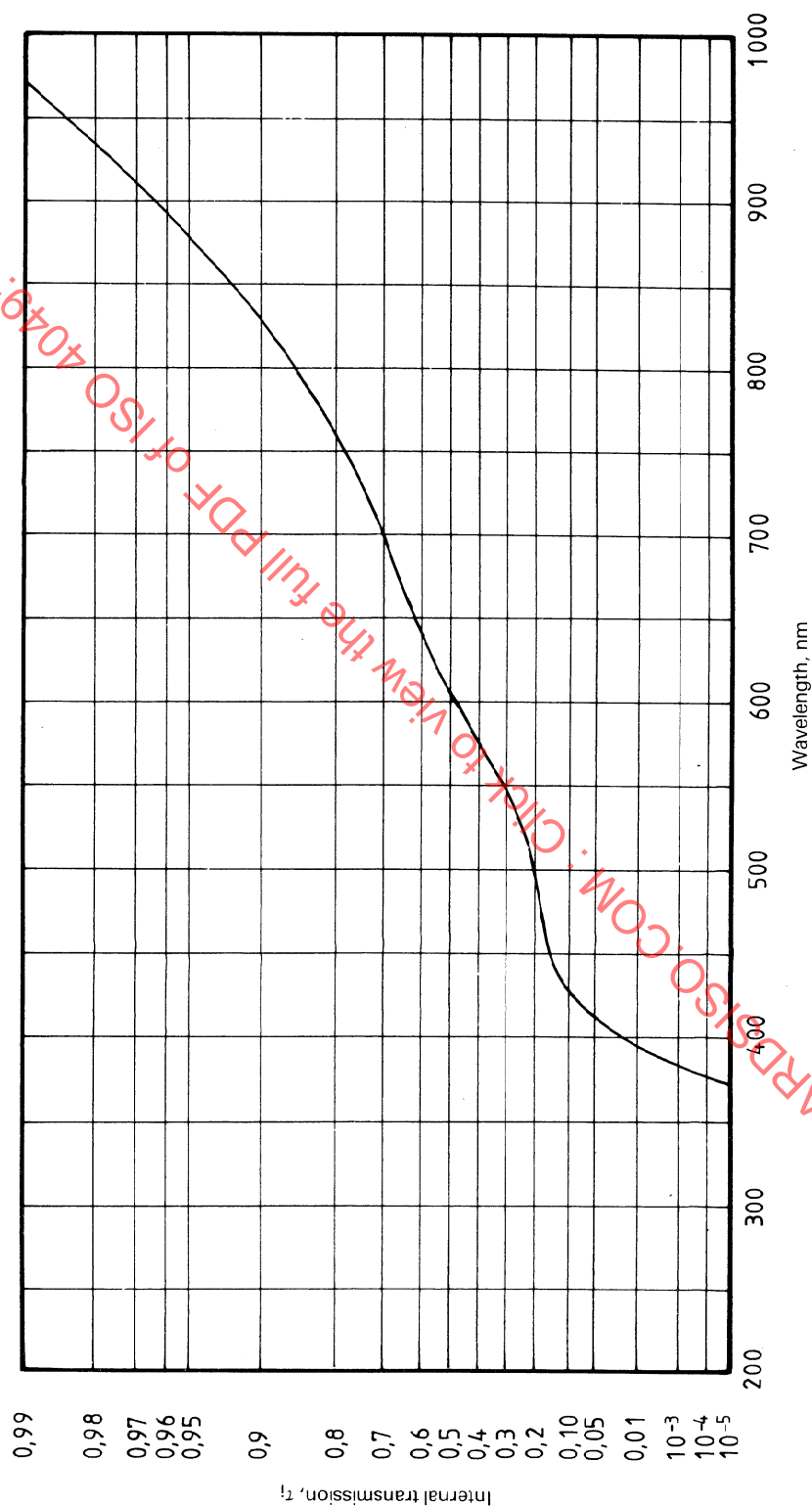


Figure 3 — Internal transmittance for colour conversion filter (see 7.6.1.1)

The ultraviolet filter shall be made of borosilicate glass with a transmittance of less than 1 % below 300 nm and greater than 90 % above 370 nm.

NOTE — The purpose of the filter is to convert the xenon, or equivalent, spectrum to that approximating to a dental operating light.

7.6.1.2 Two glass slides/plates (see 7.1.2).

7.6.1.3 Illuminance-measuring device e.g. luxmeter, capable of measuring illuminance of $(10\,000 \pm 2\,000)$ lx.

7.6.1.4 Adjustable table.

7.6.2 Procedure

In a dark room, position the illuminance-measuring device cell (7.6.1.3) under the xenon lamp with colour conversion and ultraviolet filters inserted (7.6.1.1) at such a height as to provide an illuminance of $(10\,000 \pm 2\,000)$ lx. [The adjustable table (7.6.1.4) is required to do this efficiently.] Place approximately 30 mg of material on a glass slide (7.6.1.2), position the slide on top of the cell and expose it to the light for (60 ± 5) s. Remove the slide with the sample from the irradiated area and press the second microscope slide against the material to produce a thin layer.

Visually inspect the material to see whether it is physically homogeneous.

NOTE — During this test, if the material has begun to set, clefts and voids will appear in the specimen when the thin layer is being produced.

Repeat the entire procedure twice, using a new sample of material for each test. Record the results of all three tests.

7.6.3 Interpretation of results

If, on visual inspection, the material of all three samples remains physically homogeneous, the material is deemed to have complied with the requirement of 4.2.4.

7.7 Depth of cure, type 2 materials

7.7.1 Apparatus

7.7.1.1 Metal mould, for the preparation of a cylindrical specimen, 6 mm long and 4 mm in diameter unless the manufacturer claims a depth of cure in excess of 5 mm; in this event, the mould shall be at least 2 mm longer than the claimed depth of cure.

7.7.1.2 Two glass slides/plates (see 7.1.2), each of sufficient area to cover one end of the mould.

7.7.1.3 White filter paper.

7.7.1.4 Film, transparent to the activating radiation, (50 ± 30) µm thick, e.g. polyester.

7.7.1.5 External energy source, as recommended by the manufacturer for use with the test material [see 8.3 e)].

7.7.1.6 Micrometer, accurate to 0,01 mm.

7.7.1.7 Plastics spatula.

7.7.2 Procedure

Place the mould (7.7.1.1) on a strip of the transparent film (7.7.1.4) covering the filter paper (7.7.1.3) and fill it with the test material, prepared in accordance with the manufacturer's instructions. Take care to exclude air bubbles. Slightly overfill the mould and put a second strip of the transparent film on top. Press the mould and strip of film between the glass slides (7.7.1.2) to exude excess material. Remove the microscope slide covering the strips of film and gently place the exit window of the external energy source (7.7.1.5) against the strip of film. Irradiate the material for the time recommended by the manufacturer to achieve a depth of cure of at least 2 mm.

Remove the specimen from the mould (180 ± 20) s after completion of exposure and gently remove the uncured material with the plastics spatula (7.7.1.7). Measure the height of the cylinder of cured material with the micrometer (7.7.1.6) to an accuracy of $\pm 0,1$ mm.

Record this value as the depth of cure.

Repeat the test twice.

7.7.3 Interpretation of results

If all three values are greater than 2 mm, the material has complied with the first requirement of 4.2.5.

In order to comply with the second requirement of 4.2.5, all three values shall be no more than 0,5 mm below the value stated by the manufacturer.

7.8 Flexural properties

7.8.1 Apparatus

7.8.1.1 Mould, lightly coated with separating medium¹⁾, for preparation of a test specimen (25 ± 2) mm $\times$ $(2 \pm 0,1)$ mm $\times$ $(2 \pm 0,1)$ mm. A suitable mould is illustrated in figure 4.

7.8.1.2 Two glass slides/plates (see 7.1.2), each of sufficient area to cover the mould.

7.8.1.3 Small clamp.

1) Spectroscopic grease is suitable.

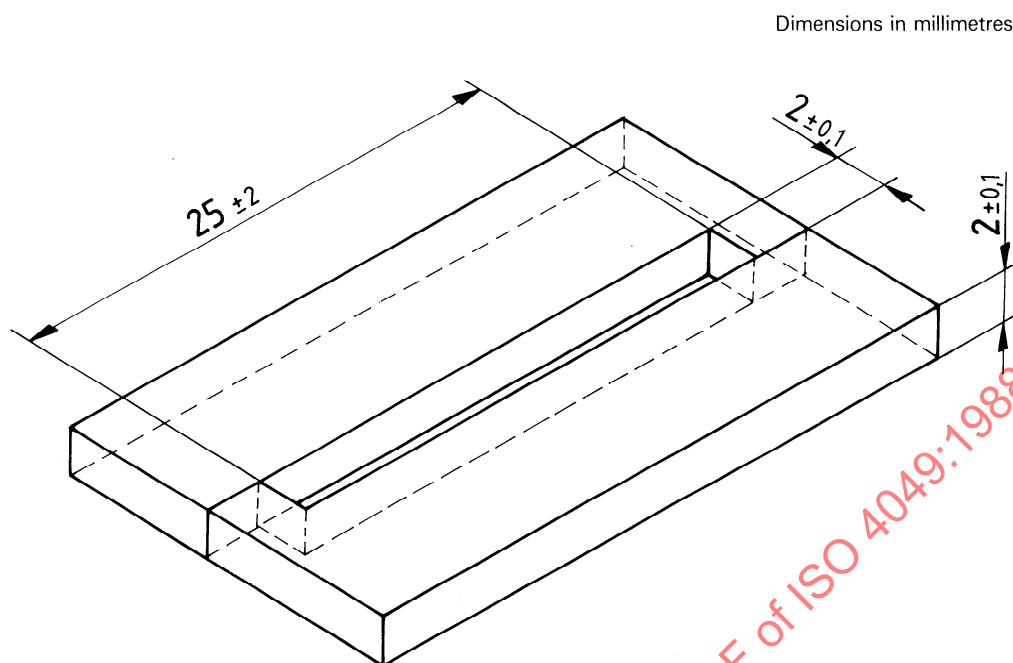


Figure 4 — Mould (7.8.1.1) for flexural strength testing

7.8.1.4 Water bath, capable of maintaining a temperature of $(37 \pm 1) ^\circ\text{C}$.

7.8.1.5 External energy source (for type 2 materials), as recommended by the manufacturer for use with the test material [see 8.3 e)].

7.8.1.6 Micrometer, accurate to 0,01 mm.

7.8.1.7 Test equipment and apparatus for application of load as specified in 7.8.3.

The apparatus consists essentially of two rods (2 mm in diameter), mounted parallel with 20 mm between centres, and a third rod (2 mm in diameter) centred between, and parallel to, the other two, so that the three rods in combination can be used to give a three-point loading to the specimen. The equipment shall allow the apparatus to be immersed in water at $(37 \pm 1) ^\circ\text{C}$ during testing.

7.8.2 Preparation of test specimens

7.8.2.1 Type 1 materials

Prepare the material in accordance with the manufacturer's instructions and immediately place it in the mould (7.8.1.1) positioned on one of the glass slides (7.8.1.2). Place the second glass slide on top of the mould and gently apply pressure by means of the clamp (7.8.1.3), thus exuding excess material from the mould. Place the assembly in the water bath (7.8.1.4), maintained at $(37 \pm 1) ^\circ\text{C}$, 3 min after the start of mixing. Remove the clamp, remove the flash and separate the mould

15 min after the start of mixing. Store the specimen in distilled water at $(37 \pm 1) ^\circ\text{C}$ for the time specified in 7.8.3. Prepare five specimens.

7.8.2.2 Type 2 materials

Prepare the material in accordance with the manufacturer's instructions and fill the mould with the material as described in 7.8.2.1. Place the exit window of the external energy source (7.8.1.5) at the centre of the specimen and against the quartz glass plate. Irradiate that section of the specimen for the recommended exposure time. Move the exit window to the section next to the centre overlapping the previous section and irradiate for the appropriate time. Then irradiate the section on the other side of the centre in the same way. Continue this procedure until the entire length of the specimen has been irradiated for the recommended exposure time. Repeat the irradiation procedure on the other side of the specimen. Place the assembly in the water bath maintained at $(37 \pm 1) ^\circ\text{C}$ for 15 min. Then remove the specimen from the mould and store in distilled water at $(37 \pm 1) ^\circ\text{C}$.

7.8.3 Procedure

Measure the dimensions of the specimen to an accuracy of $\pm 0,01$ mm 23 h and 45 min after the start of mixing, in the case of type 1 materials, or irradiation, in the case of type 2 materials. Transfer the specimen to the flexural strength testing apparatus in the testing equipment (7.8.1.7). Maintain the water surrounding the apparatus and specimen at $(37 \pm 1) ^\circ\text{C}$. Allow the specimen to stabilize for 10 min at $(37 \pm 1) ^\circ\text{C}$ prior to testing.

Twenty-four hours after the start of mixing, apply a load to the specimen at a cross-head speed of $(0,75 \pm 0,25)$ mm/min or at a rate of loading (50 ± 16) N/min until the specimen fractures. Record the maximum load exerted on the specimen.

Repeat the test on the four other specimens.

7.8.4 Calculation and expression of results

7.8.4.1 Flexural strength

Calculate the flexural strength, σ , in megapascals, from the following equation :

$$\sigma = \frac{3 Fl}{2 bh^2}$$

where

F is the maximum load, in newtons, exerted on the specimen;

l is the distance, in millimetres, between the supports, accurate to $\pm 0,01$ mm;

b is the width, in millimetres, of the specimen measured immediately prior to testing;

h is the height, in millimetres, of the specimen measured immediately prior to testing.

7.8.4.2 Flexural modulus

Calculate the flexural modulus, E , in megapascals, from the following equation :

$$E = \frac{F_1 l^3}{4 bh^3 d}$$

where

F_1 is the load, in newtons, at a convenient point in the straight line portion of the trace;

NOTE — For greater accuracy, the straight line may be extended.

d is the deflection, in millimetres, at load F_1 ;

l , b and h are as defined in 7.8.4.1.

7.8.4.3 Interpretation of results

7.8.4.3.1 Calculate the five flexural strength results.

If four or five of the results are above 50 MPa, the material is deemed to have complied with one of the requirements of 4.2.6.

If four or five of the results are below 50 MPa, the material is deemed to have failed absolutely.

If only three of the results are greater than 50 MPa, repeat the whole test. If only four results are above 50 MPa on the second occasion, the material is deemed to have failed the whole test.

7.8.4.3.2 If four or five out of five specimens or, where a second series was necessary, eight or nine out of ten specimens were above 50 MPa, discard the failed results and calculate the mean flexural strength, $\bar{\sigma}$, of the successful specimens to three significant figures.

Calculate the flexural modulus, in megapascals, of each of the successful specimens and determine the mean value, $\bar{E}$; using this mean value, calculate the value of N , in megapascals, using the following formula :

$$N = (\bar{E} \times 0,0025) + 40$$

The material is deemed to comply with the second requirement of 4.2.6 if $\bar{\sigma} > N$.

7.9 Water sorption and solubility

7.9.1 Apparatus

7.9.1.1 Mould, for the preparation of specimen discs (15 ± 1) mm in diameter and $(0,5 \pm 0,1)$ mm thick. A suitable mould is illustrated in figure 5.

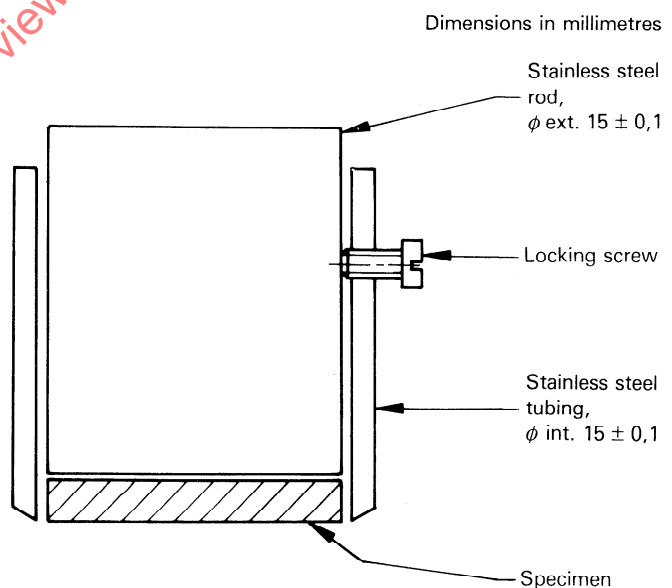


Figure 5 — Mould (7.9.1.1) for water sorption and solubility test (see 7.9)

7.9.1.2 Film, transparent to the activating radiation, (50 ± 30) μ m thick, e.g. polyester.

7.9.1.3 Glass slide/plate (see 7.1.2).

7.9.1.4 Two desiccators, containing anhydrous calcium chloride or silica gel freshly dried for 5 h at 130 °C. Replace the silica gel with freshly dried gel after each weighing sequence.