INTERNATIONAL STANDARD

ISO 21563

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Dentistry — Hydrocolloid impression materials

Médecine bucco-dentaire — Produits pour empreintes à base d'hydrocolloïdes

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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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 21563 was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 2, *Prosthodontic materials*.

This first edition of ISO 21563 constitutes a consolidation of the three standards listed below and, as such, cancels and replaces, in whole, all three of the standards listed.

— ISO 1563:1990, *Dentistry — Alginate impression materials*

— ISO 1564:1995, *Dental aqueous impression materials based on agar*

— ISO 13716:1999, *Dentistry — Reversible/irreversible hydrocolloid impression materials systems*

Re-evaluations of all the provisions stated in the three ISO standards to be included in the consolidation led to the significant technical changes listed as follows.

— The alginate hydrocolloid impression materials (ISO 1563) are now required to be subject to the same tear strength test that has been in effect for the agar hydrocolloid impression materials (ISO 1564 and ISO 13716) instead of being subject to a compressive strength test.

— The requirement for the alginate impression material powder materials to be “free from foreign materials”, as stated in ISO 1563, has not been carried forward into the consolidation because no objective test has been specified for determining compliance with the requirement.

— The “gelation temperature” requirements in ISO 1564 and ISO 13716 have not been carried forward for the agar impression materials because results of the elastic recovery test (7.5), if conducted following the required manufacturer’s instructions for use (8.2.1 and/or 8.2.2), will indicate whether adequate gelation will take place during clinical use of the materials.

Introduction

Parties seeking clarification of any provisions of this International Standard, or desiring to recommend improvements for the next edition, are encouraged to do so by contacting ISO/TC 106, Dentistry, whose address can be obtained through inquiry to the national standards body representing the interests of the inquiring parties.

Dentistry — Hydrocolloid impression materials

1 Scope

This International Standard specifies the requirements and tests for helping determine whether the elastic aqueous agar and alginate hydrocolloid dental impression materials, as prepared for retail marketing, are of the quality needed for their intended purposes. It also specifies requirements for labelling and instructions for use.

NOTE This International Standard specifies no requirements or tests for freedom from unacceptable biological hazards. However, it is recommended that, to address possible biological hazards associated with the use of hydrocolloid impression materials, interested parties should refer to ISO 7405 and ISO 10993.

2 Normative references

The following referenced documents are indispensable for application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1942, *Dentistry — Vocabulary*

ISO 6873, *Dentistry — Gypsum products*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1942 and the following apply.

3.1
bonding
adherence of the reversible and non-reversible impression material components constituting a single impression after each of the separate but interfacing materials has reached the level of elasticity and effective setting required for successful removal from the mouth

3.2
bulk container
labelled consumer packaging or primary packaging container holding a greater amount of otherwise unpackaged granular, liquid, powder, or other loose substance than is usually needed for a single dental clinical or laboratory procedure

3.3
combined reversible/non-reversible impression material system
system of impression making in which a light bodied agar material is first syringed around selected teeth so that it can bond with the non-reversible alginate material that will be forced over it later during the formation of an impression

3.4
consumer packaging
retail packaging
sales packaging
packaging constituting, with its contents, a sales unit to the final user or consumer at the point of retail

[SOURCE: ISO 21067:2007, definition 2.2.5]

3.5
elastic recovery test
method of determining whether an elastic impression material will possess the elastic properties required to recover optimally from deformation occurring during removal of impressions from contact with the impressed oral or craniofacial tissues

3.6
extrusion temperature
temperature at which a liquefied Type 3 or Type 3A agar impression material is extruded from the containing cartridge or syringe onto any oral cavity tissue

3.7
impression
negative copy of oral or craniofacial tissue surfaces obtained by impressing a mouldable impression material, usually contained in an impression tray, or injected into contact with the tissue surfaces, and allowing it to harden, or to become elastic, such that the entire impression material/tray assembly can be removed from the contact without significant harm to the tissues or to the assembly

Note 1 to entry: A properly formed impression is capable of having a relatively fluid model (cast) forming material poured against the intaglio surface so that, when the modelling material sets, a positive copy of the impressed surfaces is formed.

3.8
initial setting time
time, measured from commencement of mixing components of a material, or otherwise activating the chemistry involved, and ending at a time when results of a prescribed test show that the activated material has begun to set at a rate indicating that the effective setting time will be reached at some predictable time thereafter

Note 1 to entry: Initial setting times stated in the manufacturer's instructions are useful to test operators, users and standards developers because they can be helpful:

— in determining whether quality of a product has deteriorated before or after opening of the packaging; for example, if the initial setting time found by the test operator or user corresponds closely to that stated in the manufacturer's instructions, it can be assumed that the product is of a quality suitable for testing or use;

— in the development of standards for certain materials when there is a need for a standard to identify a reference point in time that can be used as a basis for specifying a later point in time at which a subsequently specified procedure can safely begin.

EXAMPLE It is reasonable to expect that the effective setting times for certain types of gypsum product mixtures will have been reached within 45 min after the initial setting times previously recorded for the mixes.

3.9
liquefaction
process of heating an agar impression or duplicating material to change it from the elastic gel state to the mouldable or pourable sol state

3.10
non-reversible impression material
any impression material which, having been brought to the effective setting stage as required for removal from the mouth, cannot be returned to the mouldable state required for forming impressions

3.11
primary packaging
primary container
immediate container (deprecated)
packaging designed to be in direct contact with the product

Note 1 to entry: Adapted from ISO 21067:2007, definition 2.2.2.

3.12
reversible impression material
impression material such as an agar hydrocolloid which, after having been brought to the gel state for marketing purposes, can be heated so as to bring it to the relatively fluid colloid or paste-like state required for making an impression

Note 1 to entry: Whereas in past years the "gel to sol" and "sol to gel" reversibility capacities of such impression materials has allowed them to be recycled for repeated uses, modern infection control practices now discourage user recycling of the reversible impression materials for repeated uses in the mouth.

3.13
secondary packaging
over packaging (deprecated)
packaging designed to contain one or more primary packagings together with any protective materials, accessory devices that may have to be provided for use with the product

Note 1 to entry: Adapted from ISO 21067:2007, definition 2.2.3.

3.14
storing
process of holding increments of liquefied reversible agar hydrocolloid impression material at a reduced temperature pending time they will be injected or tempered for impression making purposes

3.15
strain-in-compression test
standardized test method for determining whether elastic impression materials, when formed as impressions, will have the

— flexibility required to permit removal of the impressions from the mouth without significant injury to the impressed oral tissues, and

— the stiffness to resist the deforming forces possibly occurring during the pouring of a model-forming gypsum into the impressions, or to make the material more effective for transferring implant components and securing them in desired positions in an impression

3.16
tempering
process of holding a heavy or medium bodied agar impression material in a slightly higher than mouth temperature water bath, after the material has been placed into an impression tray, so as to further reduce the sol state temperature as necessary for safe and effective seating in the mouth

3.17
unit packet
packaging containing only the amount of product usually needed for a single dental clinical or laboratory application

4 Classification of agar hydrocolloid impression materials

The agar impression materials are classified according to the consistencies they exhibit while they are ready for impressing against the oral or craniofacial tissue surfaces, and when tested according to 5.2.

Type 1 Heavy bodied, for making impressions of complete or partial dental arches, with or without the use of companion increments of lighter bodied Type 2 or Type 3 agar impression materials.

Type 2 Medium bodied, for making impressions of complete and partial dental arches, with or without the use of companion syringe-extruded increments of Type 3 agar materials.

Type 3 Light bodied, for syringe use with either the Type 1 or Type 2 agar materials.

Type 3A Light bodied, material formulated for syringe use in a reversible/non-reversible impression material system, and that has been claimed to be capable of bonding to a companion alginate impression material that will make up the greater part of an agar/alginate impression material system.

5 Requirements — Characteristics and properties

The requirements applicable to only one category of hydrocolloid impression materials (agar or alginate) are stated immediately below in 5.1 to 5.5. The requirements applicable to both categories are displayed in Table 1 which constitutes a part of Clause 5.

5.1 **Consistency** (agar impression materials of all Types, in the sol state only)

After being exposed to the recommended storing temperature treatment recommended in the manufacturer's instructions, the material shall have a consistency that will allow the entire content of the tube or syringes to be extruded within 30 s. No specimens need to be made but material shall be tested to see if all can be extruded within 30s.

5.2 **Working time** (alginate materials only)

When tested in accordance with 7.1, the thickness of the layer of material remaining between the tip of the test penetrator and the test base plate shall not exceed 0,25 mm.

5.3 **Initial setting time** (alginate materials only)

When tested in accordance with 7.2, the initial setting time shall be within 20 % of that stated in the manufacturer's instructions [8.2.3 h)]

5.4 **Linear dimensional change** (Type 3A agar materials only)

When tested in accordance with 7.8, the dimensional change shall not exceed 1,0 %

5.5 **Tensile bond strength** (Type 3A agar materials only)

When tested in accordance with 7.9, the minimum tensile bond strength shall not be less than 50 kPa

Table 1 — Other requirements for properties — Agar and alginate materials

Test subclause number	Test procedure	Agar materials		Alginate powder and paste/paste materials
		Type 1 and Type 2	Type 3 and Type 3A	
7.3	**Detail reproduction before and after disinfection** Line width reproduced (μm)	20	20	20
7.4	**Compatibility with gypsum** Line width reproduced (μm)	50	50	50
7.5	**Elastic recovery** % (min.)	96,5	96,5	95,0
7.6	**Strain-in-compression** % Range: min. to max.	4,0 to 15,0	4,0 to 15,0	5,0 to 20,0
7.7	**Tear strength** N/mm (min.)	0,75	0,50	0,38

6 Pre-test planning approaches

The information included in this clause is provided to help test operators avoid losses of time due to trial and error efforts occurring when such information is not taken into account before test procedures such as those described in Clause 7 are begun.

6.1 Sampling

Observe the following guidelines when procuring samples of materials for testing.

a) Procure only samples that have been packaged for retail marketing and that have labelling **use by** dates that have not expired

b) Wherever possible select only those samples that have the same lot (batch) number [see 8.1c)]

c) Procure samples in minimal amounts shown below when conducting certification testing that will require production of the several specimens needed for complete evaluation of the material.

   — For agar materials, Type 1 and Type 2 — at least 30 large tubes or the equivalent.

   — For agar materials, Type 3 and Type 3A – at least 150 sticks, cartridges or capsules.

   — For alginate impression materials – at least 900 g.

   — For alginate paste/paste materials – 5 l.

   — Gypsum materials for the compatibility with gypsum test — at least 1 000 g.

NOTE The sample sizes specified in this subclause have been justified by taking into account the probable amount to be consumed in testing for compliance with all stated requirements, and also the additional amounts often needed for pre-test specimen preparation and testing practice.

6.2 Pre-test product examinations

These evaluations are helpful in determining whether the sample procured (6.1) is fit for objective testing.

6.2.1 Examinations for compliance with labelling requirements

Examine the consumer packaging components for labelling compliance with the provision of 8.1 before any attempt to open a packaging component has defaced or obliterated any labelling entry information needed for storage or use of the product. Record the name, Type, Lot number and Use by date as may be applicable for each primary container of the material to be tested.

6.2.2 Examinations for effectiveness of the packaging

Before opening any primary container, examine it for possibilities that the quality of the content may have been compromised since its manufacture; for example, evidence such as:

— loose tube caps or canister lids, or leakage;

— container rupture or punctures;

— shrinkage of the agar content of a container such as can be detected by sight, sound or touch.

Immediately after opening an alginate container, examine the content for lumps or granules that may be due to faulty or compromised packaging.

Caution: Do not use any compromised materials for preparing specimens.

6.2.3 Examinations for compliance with requirements for instructions for use

Before opening any primary container:

— examine the labels to determine whether they include any of the instructions for use information specified in 8.2;

— locate and retain any instruction sheet that may have been provided outside the primary container.

Immediately after the first opening of a primary container for powder alginate, examine the content for any instruction sheet that may have been placed inside the container.

6.3 Essential pre-test preparatory practices

6.3.1 Laboratory conditions

Unless otherwise specified in this document, conduct all specimen preparation and testing under ambient laboratory conditions of (23 ± 2) °C and (50 ± 10) % relative humidity. And, unless otherwise specified, bring all equipment and materials to be used in the tests to the ambient temperature before beginning specimen preparation

6.3.2 Apparatus function verification steps

a) Examine all accessories, instruments and equipment for functional effectiveness before they are used in a test.

b) Perform whatever calibration steps necessary to ensure that the items comply with specifications stated for them in this document, or in the normative supporting reference ISO 6873

c) Clear all instrumentation or equipment surfaces that will come in contact with the specimen material of any contaminants that might influence the test result.

6.3.3 Test material handling and use

6.3.3.1 Identification of separately-packaged samples

When the sample procured for testing (6.1) includes two or more separate packages, assign an identifying numeric or alphabetical/numeric symbol to each separate primary container for the purpose of maintaining a record of the particular container from which the materials used to form a particular specimen was taken.

6.3.3.2 Storage and manipulation

Unless otherwise specified in this International Standard, store, prepare and manipulate the materials used for forming the test specimens employing the equipment and procedures recommended in the manufacturer's instructions (8.2). When mixing the alginate materials, record the time required for each specimen preparation mix.

6.3.3.3 Mixing water for the alginate and gypsum products

The quality and temperature of the water used for making the specimens shall be as specified below:

— water quality: Grade 3 in accordance with ISO 3696, obtainable by distillation, deionization or reverse osmosis;

— water temperature: as recommended by the manufacturer [8.2.3 c)].

6.3.3.4 Amount of material to be prepared for each specimen

a) **For agar hydrocolloid material mould assemblies**

Type 1 and Type 2 agar materials – one tube for each specimen.

Type 3 and Type 3A agar materials, when used to make part of a specimen, such as for detail reproduction, gypsum compatibility, dimensional change or tensile bond strength test — one stick or one cartridge.

For Type 3 and 3A agar materials when used to make up the entire volume of the elastic recovery, strain-in-compression or tear strength specimen — a volume greater than that contained in one syringe will usually be needed.

b) **For alginate materials**

Powder or paste materials supplied in bulk containers — a mixture having a volume of about 40 ml (enough for making a medium-sized impression).

Powder materials supplied in unit packets — whatever volume results from mixing the powder provided in one packet with the recommended volume of water.

6.3.4 Simulated oral time/temperature treatment of specimens formed in completely closed moulds

After the specimen forming material has been completely enclosed in the specimen forming assembly, the entire assembly shall be conditioned for the time and at the temperature [8.2.1 c)] simulating that to which the material should be exposed after the impression has been seated in the mouth; for example:

— an assembly containing alginate alone, or containing agar/alginate combinations, shall be immersed in a cooling water bath set at (35 ± 2) °C and shall remain so immersed for the time recommended in the instructions for the impression material/tray assembly to remain seated in the mouth;

— assemblies containing agar material alone shall be immersed in the cooling a water bath for the time and at the cooling water temperature recommended in the instructions for obtaining the desired degree of gelation of the material after it has been seated in the mouth.

6.3.5 Order of conducting tests

When testing the alginate impression materials always conduct the working time test (7.1) and the initial setting time test (7.2) first in order because, when the results obtained in these tests differ significantly from manufacturer claims, [8.2.3g)] and [8.2.3 h)], it is likely that the quality of the sample procured for testing has somehow been compromised and that the manufacturer should be contacted relative to the difference noted.

6.3.6 Test schedules timing

Time the schedules for specimen preparation and testing using a timing device such as a stopwatch accurate within 1 s over a period of 30 s

6.3.7 Pass/fail determinations

The minimum number of specimens to be tested for pass/fail determinations shall be either three or five, as indicated in the first Specimen preparation subclause for each related test in Clause 7.

a) **For a three-specimen minimum**, make a series of three specimens initially. If at least two of the three specimens comply with the related requirement, the material passes. If none comply, the material fails. If only one specimen complies, make three additional specimens. If all three of the additional specimens comply, the material passes; otherwise the material fails.

b) **For a five-specimen minimum**, make and test a series of five specimens initially. If at least four of the five specimens comply with the related requirement, the material passes. If only one or two

specimens comply, the material fails. If only three specimens comply, make a series of five additional specimens. If four of the second series of specimens comply, the material passes; otherwise the material fails.

6.3.8 Expression of test results

Record the number of specimens tested and whether the material passes or fails.

7 Test methods

Test operators are advised to become familiar with the content of Clause 6 before beginning any specimen preparation

7.1 **Working time test** (alginate materials only)

7.1.1 Apparatus and materials (the array of devices or materials used or available for an undertaking).

a) **Rigid ring mould**, height (16,0 ± 0,1) mm, internal diameter (30,0 ± 0,2) mm.

b) **High vacuum grease**, such as silicone grease which will not be reactive with the ring mould or the material being tested.

c) **Flat glass plate**, approximately 50 mm × 50 mm and at least 3 mm thick.

d) **Working time test instrument** (Figure A.1). The combined mass of the penetrator shaft and penetrator tip shall be (50 ± 0,5) g.

7.1.2 Specimen preparation

7.1.2.1 Prepare a minimum of three specimens

7.1.2.2 Advance preparation steps

a) Mark or modify the test instrument base [7.1.1d)], the ring mould [7.1.1a)], and the flat plate [7.1.1c)] such that these parts can be related to each other in the same position for each test.

b) Coat surfaces of the ring mould with a thin film of the high vacuum grease [7.1.1b)].

c) Centre and fix the ring mould onto the flat plate in position for the test

d) Elevate and lock the penetrator shaft and the dial indicator spindle of the test instrument (Figure A.1) so as to allow the centre of the ring mould/flat plate assembly to be positioned directly below the penetrator tip.

e) Unlock the penetrator shaft so as to allow the tip to descend into contact with the centre of the glass base plate surface below the ring mould. Lower the dial indicator spindle contact point to rest on the top of the penetrator shaft and record the resulting dial indicator reading as the fiducial Reading **a**.

f) Elevate and lock the penetrator shaft, and the dial indicator spindle, so that the penetrator tip is positioned far enough above the top of the ring mould to allow removal of the ring mould/flat plate assembly in preparation for filling the mould assembly with impression material.

7.1.2.3 Specimen formation and pre-test positioning

Immediately after completion of mixing the alginate, slightly overfill the ring mould and strike off the excess level with the top surfaces of the mould; centre the test specimen assembly beneath the penetrator tip. Then unlock the penetrator shaft and allow it to descend until the tip is barely in contact with the top surface of the material to be tested and lock the shaft with the tip in this position.

7.1.3 Test procedure

At 5 s before the end of the working time stated in the instructions, loosen the penetrator shaft locking screw so as to allow the penetrator tip to descend into the material under the combined weight of the shaft/tip complex [7.1.1 d)].

Ten seconds thereafter, lower the dial indicator contact point to rest on the top of the penetrator shaft. Immediately thereafter record the resulting dial indicator reading as Reading **b**. Then calculate the difference between Readings **a** and **b** to the nearest 0,01 mm and record whether the thickness of the material remaining between the penetrator tip and the glass plate surface complies with the allowance stated in 5.3.

NOTE Since the material in the working time test assembly will not have reached the initial setting stage by the time descent of the penetrator has been completed it will therefore be possible and time saving, to begin testing the same specimen for initial setting time according to 7.2.3 shortly thereafter.

7.1.4 Pass/fail determinations/and expression of results

See 6.3.7 and 6.3.8.

7.2 Initial setting time test (alginate impression materials only)

7.2.1 Apparatus

a) **Rigid ring mould** [7.1.1a)], coated with the high vacuum grease [7.1.1b)].

b) **Cylindrical polymethylmethacrylate test rod**, approximately 100 mm long and 6 mm in diameter, having both ends polished to a high lustre (scratch free as can be determined visually without magnification).

7.2.2 Specimen preparation

7.2.2.1 Prepare a minimum of three specimens

As indicated in the NOTE under 7.1.3, the specimens prepared for the working time test can also be used for the initial setting time test. Otherwise complete the first three steps, a), b) and c) described in 7.1.2.2 for forming the specimens according to the introductory sentence of 7.1.2.3 and conduct the test as described in 7.2.3 below.

7.2.3 Test procedure

At 5 s after the working time stated in the instructions [8.2.3 g)] begin the test by placing an end of the test rod [7.2.1b)] into momentary contact with the unset specimen material. Clear any material left on the rod from the contact. Then repeat the contact/withdrawal rod clearing steps at 10 s intervals until the test rod separates cleanly from the material. Record the time of this occurrence as the initial setting time and compare it to the requirement stated in 5.4.

7.2.4 Pass/fail determinations and expression of results

See 6.3.7 and 6.3.8.

7.3 Detail reproduction test before and after specimen disinfection

7.3.1 Apparatus and materials

a) **Lined detail reproduction specimen forming test block** (Figure A.2).

b) **Perforated ring mould and ring mould retainer specimen forming accessories** (Figure A.3).

c) **Soft mouldable clay-like, putty-like or wax**, to partially fill perforations in the ring mould [7.3.1 b)] so as to help keep the light bodied agar materials confined in the mould.

d) **Flat glass specimen cover plate**, approximately 50 mm x 50 mm and at least 3 mm thick having one side covered with a polyethylene sheet approximately 0,035 mm thick.

NOTE A very thin film of the high vacuum grease [7.1.1 b)] spread over the underside of the glass plate will keep the polyethylene sheet smoothly adapted to the plate.

e) **Mould release solution** (for the alginate containing specimens only), such as a freshly made 1 % solution of tetradecylamine in acetone, for preventing adherence of the alginate specimen materials to the lined test block surface.

f) **Oven**, held at (35 ± 2) °C, for simulated intra-oral temperature conditioning of the test block.

g) Equipment recommended for **liquefaction, storing and tempering** the agar materials and the recommended syringes and needles as may be applicable [8.2.2 a)] and the items needed for proportioning and mixing the alginates [8.2.3 c)].

h) **Two water baths**, for use depending upon the material being tested

— one held within whatever temperature range the manufacturer instructions may recommend for cooling agar impressions in the mouth, or

— one held at simulated intra oral temperature of (35 ± 2) °C, for use with the alginate materials that are exposed to that approximate temperature while setting in the mouth.

i) **C-clamp**, having a minimum screw opening of 40 mm and a minimum throat depth of at least 30 mm.

j) **Disinfectant** identified by the manufacturer [8.2.1 f) 1 second subdivision].

k) **Microscope**, equipped for x 4 to x 12 magnification and low angle illumination.

7.3.2 Examination and conditioning of equipment and accessories

a) Use the microscope [7.3.1 k)] to determine whether the lines on the test block have been cleared of contaminants.

NOTE An ultrasonic cleaning device containing a concentrated solution of sodium bicarbonate in water has been found useful for removing alginate contaminants from impression trays and test accessories.

b) Place the test block [7.3.1a)] in the oven [7.3.1 f)] for conditioning at least 15 min.

7.3.3 Specimen preparation — Agar materials

7.3.3.1 Prepare a minimum of three specimens

7.3.3.2 Advance preparation steps — Agar materials

a) Bring each type of material to be used for forming a specimen to the recommended liquefaction temperature [8.2.2 b)] and immediately thereafter transfer the tube or syringe to the recommended storing temperature water bath [8.2.2 b)], there to be held until the extrusion for the next step

b) As time for specimen formation nears, transfer the tubes containing the Type 1 and Type 2 materials to the recommended tempering treatment bath [8.2.2 b)].

7.3.3.3 Specimen formation steps — Agar materials

7.3.3.3.1 Specimen formation and post-formation steps — Type 1 and Type 2 agar materials

After the materials have been brought to the recommended tempering temperature complete the following steps within 30 s.

a) Remove the test block from the oven and seat the retainer containing the perforated ring mould [7.3.1b)] to rest on the shoulder of the test block, thus creating the specimen forming mould cavity.

b) Immediately thereafter, begin extruding the tempered material directly into the mould cavity so that the first increments will first enter the Lines **a**, **b** and **c** on one side of the test block surface, and then be caused to flow into the lines for their full lengths.

c) Then extrude enough material to slightly over-fill the mould cavity immediately thereafter and slowly force the polyethylene covered glass plate against the excess material until the plate is in near contact to the top surfaces of the prepared ring mould forming sides of the mould cavity.

d) Complete formation of the specimen by using the C-clamp [7.3.1 i)] to force the covered glass plate into contact with the top surfaces of the mould cavity ring mould.

e) Immediately thereafter, immerse the specimen forming assembly in the water bath [7.3.1 h)] at the temperature and for the time recommended for cooling the impression in the mouth [8.2.1 c)].

f) After the simulated oral time-temperature (6.3.4) treatment, complete the final post-formation specimen preparation steps as follows:

   — separate the impression material/ring mould assembly from the test block and mould retainer components;

   — rinse the assembly with water;

   — use a gentle air stream to expel gross amounts of water from the lined surface.

Caution: Do not dry the surface.

7.3.3.3.2 Specimen formation and post-formation steps — Type 3 and Type 3A Agar materials

Form the specimen in much the same way as for the **Type 1** and **Type 2** materials, with the following two exceptions:

a) Extrude the material from a filled syringe taken directly from the storing temperature treatment water bath (7.3.3.2)

b) Extrude only enough of the Type 3 or Type 3A material into the mould cavity to cover the lines of the test block surface. The additional amount of material required to force the first increments of the Type 3 materials into the lines and to complete the necessary overfilling of the mould cavity, shall consist of either a Type 1 or Type 2 agar material. The additional amount of material required to force the increment of Type 3A materials into the lines shall consist of a companion alginate [Clause 4 d)].

7.3.3.4 Advance preparation steps — Alginate materials

For specimens that will be formed wholly, or in part of alginate materials, paint a thin film of mould release solution [7.3.1 e)] to cover the surface of the test block.

Proportion the alginate ingredients for mixing.

7.3.3.5 Specimen formation and post-formation steps — Alginate materials

Follow essentially the same order of steps specified for the agar materials, with the following exceptions:

— At 30 s prior to the stated working time, deliver approximately 6,5 ml of the mixed material onto the approximate centre of the test block.

— At 15 s prior to the stated working time begin pressing the polyethylene covered glass plate [7.3.1 d)] against the overfilled amount of alginate extending above the ring mould cavity, slowly and without any twisting motion, so as to expel most of the excess.

— Then use the C-clamp [7.3.1 i)] to force the plate into contact with the top surface of the perforated ring mould so as to give final form to the specimen.

— Expose the specimen forming assembly to the simulated oral temperature treatment (see 6.3.4)

7.3.4 Test procedure steps

Immediately after separation of the specimen from the mould, use the microscope [7.3.1 k)] to examine the lined specimen surface for compliance with the detail reproduction requirement specified for the undisinfected specimen (See the requirement in Table 1). If the specimen complies, proceed with the following steps. If not, the specimen fails.

NOTE When viewing detail reproduction and gypsum compatibility specimens for test purposes, colour differences of the materials may make it necessary to use different lighting intensities (overhead and/or microscope lighting), or differently coloured microscope lighting filters, or both, in order to determine whether the required lines have been clearly reproduced in surfaces of the impression material specimens and in gypsum specimens made for the gypsum compatibility test.

Complete the test immediately thereafter by first disinfecting the specimen according to the manufacturer instructions [8.2.1 f)], and then by re-examining the treated specimen for continued compliance with the detail reproduction required for the pre-disinfected specimen.

7.3.5 Pass/fail determination and expression of results

See 6.3.7 and 6.3.8.

7.4 Compatibility with gypsum test

7.4.1 Apparatus and materials

a) **Agar, alginate or agar/alginate impression material test specimen assemblies** formed and treated according to 7.3.3 or 7.3.4 and which have been found to be in compliance with the detail reproduction requirement before and after being disinfected.

b) **Post disinfecting treatment agent** that may be recommended according to [8.2.1 f) 3)].

c) **Slit mould** (Figure A.4) having a rabetted recess machined into the top surfaces, along with a companion clamping mechanism such as a worm gear hose clamp for closing the slit.

NOTE Use of the slit mould requires that the mould be clamped such that the slit will be closed during formation of the gypsum specimen. Later, after the gypsum specimen has set, the clamping force is released to allow the slit to open for easy removal of the specimen. The brass alloy used for the mould should therefore have a strain-at-elastic limit sufficiently high to permit the slit to be closed and opened repeatedly without significant permanent reduction in width.

d) **High vacuum grease,** such as silicone grease, that will be non-reactive with the slit mould [7.4.1 c)] and the gypsum products [7.4.1 f)].

e) **Microscope** [7.3.1 k)].

f) **Two dental gypsum products** [see 8.2.1 f) 4)]:

— one Type 3 Dental stone, model and,

— one Type 4 or one Type 5 Dental stone, high strength.

7.4.2 Specimen preparation

7.4.2.1 Prepare a minimum of three specimens for each gypsum product tested [7.4.1 f)]

7.4.2.2 Advance preparation steps

a) Determine whether the gypsum products [7.4.1 f)] comply with the initial setting time and detail reproduction requirements of ISO 6873. Do not use any gypsum that does not comply with these requirements.

b) Treat the inner surface of the slit mould [7.4.1 c)], including the slit surfaces, with a thin film of the high vacuum grease [7.4.1 d)], and then use the clamping mechanism to close the slit.

c) Use any post-disinfecting agent [7.4.1 b)], that may be recommended in the instructions for treating the impression before pouring the gypsum.

d) Position and temporarily fix the ring mould/impression material specimen assembly, lined surface down, in the rabetted recess of the slit mould and invert the assembly in preparation for pouring of the gypsum product.

e) Pre-proportion the water/gypsum mixing components using 120 g of gypsum powder along with the volume of water required according to the water/powder ratio specified in the gypsum manufacturer's instructions for use [8.2.3 c)].

7.4.2.3 Specimen formation steps

Begin mixing the gypsum at one minute before the end of the time lapse permitted between removal of the impression from the mouth and the time recommended for commencement of pouring the model [See 8.2.1 e)].

Upon completion of the mix, direct first increments of the gypsum mixture, via mechanical vibration, so that they will flow first down along an internal side surface of the mould, and then flow over the ends of the raised Lines **a**, **b** and **c** on one side of the impression material specimen, and then be directed to gradually flow along and over the lines to their opposite ends. Continue adding increments thereafter until the mould cavity is slightly underfilled.

Unless otherwise specified by the gypsum or impression material manufacturer's instructions, allow the poured impression gypsum assembly to set in the laboratory environment until 45 min after the initial setting time previously determined for the gypsum product in accordance with [7.4.2.2 a)]. Then separate the gypsum specimen from the assembly and begin the test procedure immediately thereafter.

7.4.3 Test procedure

Use the microscope [7.4.1 e)] to examine the surface of the gypsum specimens for compliance with the requirement to copy Line **a, b,** or Line **c,** as specified in Table 1.

7.4.4 Pass/fail determination and expression of results

See 6.3.7 and 6.3.8.

7.5 Elastic recovery test

7.5.1 Apparatus and materials

a) **Specimen forming split mould** with **Fixation ring** (Figure A.5).

b) **High vacuum grease**, such as silicone grease [7.1.1 b)].

c) **Two flat glass plates**, approximately 50 mm by 50 mm at least 3 mm thick each having one flat surface covered with a thin polyethylene sheet approximately 0,035 mm thick.

d) **C-clamp**, [7.3.1 i].

e) **two water baths** [7.3.1 h)].

f) **Oven** held at a temperature of (35 ± 2) °C, for simulating intra-oral temperature conditioning of the split mould halves.

g) **Flat glass or metal test plate**, approximately 15 mm × 15 mm and 2 mm thick.

h) **Test instrument** such as the one shown in Figure A.6 equipped with dial indicator having graduations of 0,01 mm. The dial indicator spindle shall have the capacity for contributing, along with the mass of the test plate [7.5.1 g)], to application of an initial force of (0,6 ± 0,1) N on the specimen. The stop on the test instrument shall be set to limit compression of the test specimen to (4,0 ± 0,1) mm.

7.5.2 Specimen preparation

7.5.2.1 Prepare a minimum of five specimens

7.5.2.2 Advance preparation steps

a) Apply a very thin film of the high vacuum grease [7.5.1 b)] to the internal surfaces of the fixation ring and to all surfaces of the split mould halves [7.5.1 a)].

b) Place the specimen forming mould halves and one of the polyethylene covered glass plates [7.5.1 c)] in the oven [7.5.1 f)] for simulated intra-oral temperature conditioning for at least 15 minutes.

c) Bring the agar materials to the storing temperature, or tempered, condition recommended for their use in impression making [8.2.2 b)] and hold them in readiness for specimen formation.

d) Proportion the components to be mixed for the alginate material.

7.5.2.3 Specimen formation and positioning for testing steps

Accomplish the first five of the following six steps after the agar materials have been brought to the recommended storing temperature or tempering readiness [8.2.2 b)], or within 60 s after completion of an alginate mix.

a) Centre the fixation ring on the unconditioned polyethylene glass plate and half fill the resultant mould cavity with the mixed alginate impression material or, with an agar material having been held at the recommended storing or tempering temperature.

b) Remove the two split mould halves from the conditioning oven, align them together and press them down through the impression material in the fixation ring simultaneously until their bottom surfaces are in near contact with the underlying polyethylene covered glass plate, so as to force excess material above the top of the split mould halves.

c) Remove the conditioned top covering glass plate from the oven and press it, polyethylene covered surface down, against the material protruding above the top surface of the mould so as to expel almost all of the excess.

d) Then use the C-clamp [7.5.1 d)] to force the plates into contact with the top and bottom surfaces of the mould halves so as to give final form to the specimen.

NOTE Rigid metal or polymer back-up plates may be used between glass plate surfaces and the C-clamp parts to minimize scratching and breakage of the glass plates.

e) Place this specimen forming assembly in the appropriate simulated oral temperature or cooling water bath [7.5.1 e)] for the time specified for leaving the impression in the mouth [8.2.1 c)].

f) Within 40 s after completion of the appropriate water bath treatment, separate the specimen from the assembly, place the test plate [7.5.1 g)] to rest on the top surface of the specimen and then centre this assembly on the base of the test instrument [7.5.1 h)] so that the centre of the specimen is in axial alignment with the dial indicator spindle as depicted in Figure A.6.

7.5.3 Test procedure

Conduct the test in accordance with the following time schedule, where t is the time the specimen is removed from the water bath.

t + 45 s: Gently lower the dial indicator spindle contact point to seat on the test plate resting on top of the specimen.

t + 55 s: Read the dial indicator and record the reading as h_1.

t + 60 s: Deform the specimen (4,0 ± 0,1) mm, as limited by the stop on the test instrument within 1 s, and immediately thereafter release the deforming force such that it will be completed within 5s.

t + 66 s: Complete release of the deforming force over a period of 5 s and immediately thereafter lift and fix the dial indicator contact point above and out of contact with the plate resting on the specimen.

NOTE Possibilities for lateral displacement of the specimen during application of the deforming force can be reduced by cementing an abrasive paper sheet (about ISO grit number P1200) to cover the surfaces of the instrument base and the test plate that will contact the top and bottom surfaces of the specimens during the test.

t + 96 s: Gently return the dial indicator contact point to rest on the test plate.

t + 106 s: Record the dial indicator reading as h_2.

7.5.4 Calculation of results

Use the following equation to calculate the elastic recovery (K) for each specimen to the nearest 0,1 %:

$$K = \left[100 - \left(100 \frac{h_1 - h_2}{h_0} \right) \right]$$

where

h_0 is the measured height of the split mould,

h_1 is the dial indicator reading at t + 55 s (immediately before the specimen is deformed), and

h_2 is the dial indicator reading at t + 106 s (40 s after the deforming force has been completely removed from the specimen).

If a failing value is recorded for a specimen, section the specimen axially, or longitudinally, into eight approximately equal sized segments and examine each segment for internal defects such as air inclusions that might have contributed to the failure of the specimen. Do not use values reported for defective specimens in pass/fail determinations.

7.5.5 Pass/fail determinations and expression of results

See 6.3.7 and 6.3.8.

7.6 Strain-in-compression test

7.6.1 Apparatus and materials

a) **Items** listed in [7.5.1 a)] to [7.5.1 f)] for preparing the specimens used in 7.5

b) **Test instrument**, such as the one shown in Figure A.7 equipped with dial a indicator having graduations of 0,01 mm.

7.6.2 Specimen preparation

7.6.2.1 Prepare a minimum of five specimens

Form and otherwise prepare the specimens as for the elastic recovery test, except that the test plate [7.5.1 g)] shall not be placed on the specimen.

7.6.3 Test procedure

Immediately after separation of the specimen from the forming assembly, place it on the base of the test instrument [7.6.1 b)] so that its centre is in axial alignment with the centre of the loading shaft foot. Conduct the test in accordance with the following time schedule, where t is the time the specimen is removed from the appropriate simulated oral temperature or cooling water bath.

a) t + 45 s Slowly lower the loading shaft foot of the instrument into contact with the top of the specimen so as to apply the required initial load of (1,2 ± 0,1) N on the specimen.

b) t + 80 s Begin and complete the following five steps in rapid succession.

   — Lock the loading shaft in place.

   — Lower the dial indicator contact point into contact with the top of the loading shaft.

   — Note the dial indicator reading to the nearest 0,01 mm record the reading later as h_1.

   — Lift and lock the dial indicator contact point from contact with the loading shaft.

   — Place the weight (Figure A.7, key item 2) required to help provide for a total force of (12,2 ± 0,1) N to rest on the weight support collar of the loading shaft.

c) t + 90 s Unlock the shaft and allow it to descend slowly so as to complete loading of the total required force over a period of 10 s. Allow the force to remain until

d) t + 120s Then lock the shaft in place, return the dial indicator contact point into contact with the top of the loading shaft and immediately thereafter record the dial indicator reading as h_2.

7.6.4 Calculation of results

Use the following equation to calculate the strain-in compression (E) for each specimen to the nearest 0,1 %:

$$E = 100\left(\frac{h_1 - h_2}{h_0}\right)$$

where

E is the percentage of strain-in compression,

h_0 is the height of the split mould,

h_1 is the dial indicator reading 30 s after application of the initial load, and

h_2 is the dial indicator reading at 30 s after application of the total load.

Examine any failing specimens according to the procedure described in the last paragraph of 7.5.4.

7.6.5 Pass/fail determinations and expression of results

See 6.3.7 and 6.3.8.

7.7 Tear strength test

7.7.1 Apparatus and materials

a) **Specimen sheet forming mould** (Figure A.9) having a depth that will provide for a specimen thickness of (4,0 ± 0,5) mm.

NOTE 1 The specimen thickness may vary within the stated tolerance depending upon the capacity of the gripping mechanism available for the test. Use of the optional tear test specimen preparation procedure described in Annex A for fitting the test specimen for gripping in the test instrument will allow accommodation of any specimen thickness within the specified tolerance.

b) **Polyethylene sheets**, approximately 0,035 mm thick and having length/width dimensions approximating those for the mould cavity floor and the mould cover (Figure A.9).

c) **High vacuum grease**, such as a silicone grease [7.1.1 b)].

d) **Oven**, held at (35±2) °C.

e) **Two water baths** [7.3.1 h)].

f) **Specimen sheet support pad,** on which to place the formed specimen sheet during use of the die [7.7.1g)] for precision cut-out of the specimen. Length/width dimensions of the pad shall approximate those of the specimen forming sheet.

NOTE 2 The pad, which may consist of layers of water proof paper, or polymer or wax sheets, may need to vary in thickness depending upon resistance to cutting exhibited by the specimen sheet and pad forming materials.

g) ASTM D624, Die C, for cutting specimens to dimensions shown in Figure A.8.

NOTE 3 Satisfactory tear strength test specimens, such as depicted in Figure A.8, can also be produced using a machined or moulded polymeric specimen forming mould plate.

h) **Specimen thickness measuring instrument,** such as a dial indicator mounted on a conventional dial indicator support stand. The dial indicator shall have graduations of 0,01 mm, a measuring range in excess of 10 mm, a disk-like contact point about 10 mm in diameter, and a spindle travel distance controlled such that the measuring stress applied by the contact point does not exceed 22 kPa.

i) **Test instrument,** capable of measuring a tensile force of at least 50 N at a rate of 500 mm/min.

7.7.2 Specimen preparation

7.7.2.1 Prepare a minimum of five specimens

7.7.2.2 Advance preparation steps

7.7.2.2.1 For all specimens

— Apply a thin film of the high vacuum grease [7.7.1 c)] to the under surface of the mould cavity cover for the specimen sheet forming mould (Figure A.9, key item 4).

— Adapt a wrinkle free polyethylene sheet [7.7.1 b)] to the grease covered surface cavity cover.

— Condition the specimen sheet forming mould, without the cover, in the oven [7.7.1 d)] for at least 15 min.

7.7.2.2.2 For the agar specimens only

Conduct the liquefaction, storing and tempering procedures required to bring the materials to consistency required for forming an impression.

7.7.2.2.3 For the alginate specimens only

Proportion the water/powder or paste/paste components for mixing.

7.7.2.3 Specimen sheet forming steps

a) Verify the readiness of the agar impression material, or complete a mixture of the alginate ingredients.

b) Remove the specimen sheet forming mould from the oven.

c) Within 60 s after removing the mould from the oven complete the following three steps:

— slightly overfill the specimen sheet forming mould cavity with impression material;

— press the polyethylene covered surface of the specimen sheet mould cover into contact with the mould cavity borders so as to expel the excess and give the specimen sheet its final form;

— condition the specimen sheet forming assembly in the appropriate cooling or mouth temperature water bath [7.7.1 e)] for the period of time recommended in the instructions for leaving the impressions in the mouth [8.2.1 c)].

7.7.2.4 Specimen shaping and further preparatory steps

Accomplish the following steps within 90 s after removal of the specimen forming assembly from the water bath conditioning.

a) Separate the formed specimen sheet from the mould and place it on the specimen sheet support pad [7.7.1 f)].

b) Use the die [7.7.1 g)] to cut the specimen to the desired shape (Figure A.8).

NOTE 1 If a specimen forming mould plate such as the one described in the Note appearing under 7.7.1.g) is used to form the specimen, the resulting specimen will be ready for the thickness measurement and testing immediately after separation from the mould plate

CAUTION — Handle specimens very carefully during subsequent steps to avoid stressing the notched area of the specimen before the test load is applied.

c) Use the instrument [7.7.1 h)] to measure thickness of the specimen at a point centred and just inside the apex of the 90° angle notch and record the measurement.

NOTE 2 To avoid pre-test stress of the specimen during the thickness measurement it may be necessary to increase dimensions of the measuring instrument base so as to provide complete support for the underside of the specimen.

d) Align and secure the specimen in the gripping mechanism of the test instrument [7.7.1 i)] for testing taking the following factors into account:

— experience seems to indicate that the optimum air pressure for use in pneumatic gripping of the hydrocolloid impression material specimens is about 83 kPa (12 PSI);

— depending upon the type of grip face surfacing, it may be necessary to cover gripping surfaces of the test instrument with adhesive backed abrasive paper (about ISO Grit number P280) in order to achieve effective gripping without significant pre-test stressing of the specimen.

NOTE 3 Fitting the specimens for gripping according to the option described in Annex B helps reduce possibilities for pre-test stressing of the specimen.

7.7.3 Test procedure

Immediately after securing the specimen in the gripping mechanism, apply a tensile test load at a speed of 500 mm/min until rupture of the specimen. Record the force required to achieve rupture.

7.7.4 Calculation of results

Use the following equation to calculate the tear strength for each specimen to the nearest 0,01 N/mm:

$$Ts = \frac{F}{d}$$

where

Ts is the tear strength (N/mm),

F is the maximum force, in Newtons, applied to cause rupture of the specimen,

d is the specimen thickness (mm).

7.7.5 Pass/fail determinations and expression of results

See 6.3.7 and 6.3.8.

7.8 Linear dimensional change test (Type 3A agar materials with companion alginate only)

7.8.1 Apparatus and materials

a) **All of the apparatus and materials** required for preparing and testing Type 3A materials for the detail reproduction test (7.3.1).

b) **Measuring microscope** accurate to 0,01 mm, and capable of x 4 to x 12 magnification and of measuring straight-line distances of at least 30 mm.

7.8.2 Specimen preparation

7.8.2.1 Prepare a minimum of five specimens

7.8.2.2 Advance preparation steps

7.8.2.2.1 Test block preparation and positioning for measurement verification

a) Clean the test block ultrasonically to remove any contamination from the lines (see the NOTE immediately below 7.3.2 a).

b) Position the test block on the microscope base or stage [7.8.1 b)] with Line d_1 to the right and with Line **c** appearing as the lower Line depicted in Figure A.10, key item 1.

c) Position the x-axis of the microscope cross hair parallel to, and approximately 0,03 mm below Line **c** as depicted in Figure A.10, key items 5 and 6.

d) Adjust the microscope slide or stage to bring the y-axis of the cross hair to a position at least 0,1 mm outside and to the right of Line d_1 on the test block.

7.8.2.2.2 Test block line distance measurement verification steps

Proceed with the following steps taking into account that, after positioning the test block according to the last step in 7.8.2.2.1 d) the direction of travel of the microscope slide or stage should not be reversed at any point during the subsequent travels until after the final measurement between Lines d_1 and d_2 has been recorded.

a) Adjust the microscope slide or stage so that the left edge of the y-axis of the cross hair is in precise alignment with the inner edge of Line d_1 [see Figure A.10 c), key item 5]. Stop the travel motion at this point and record this position as the **initial fiducial measurement**.

b) Then adjust the travel of the cross hair to bring the same left edge of the y-axis cross hair into precise alignment with the inner edge of Line d_2 (see Figure A.10, key item 6). Stop the travel motion at this point and record the reading for this position as the **final fiducial measurement.**

c) Calculate and record the difference between the **initial and final fiducial reading.** Make two additional such measurements of the distance between Lines d_1 and d_2. Then calculate and record the mean for the three distance measurements and record the result as L_1, the **fiducial distance** of Line **c** between Lines d_1 and d_2 on the test block.

7.8.2.3 Specimen formation and pre test measurement

Form, treat and test each specimen according to the procedures specified in 7.3 for the Type 3A/Companion alginate Detail reproduction **test** specimens.

7.8.3 Test procedure

After each specimen has been found to be in compliance with the Detail reproduction requirement (see Table 1), position it on the microscope base or stage for essentially the same line distance measurements as those conducted for the test block (7.8.2.2.1 and 7.8.2.2.2), with the following exceptions:

a) position the specimen on the test instrument base or stage with Line d_2 to the right so that the distance measurement can begin with the left edge of the y-axis of the microscope cross hair in precise alignment with the inner edge of Line d_2, as illustrated in Figure A.10, key item 4;

b) record only one measurement of the distance between Lines d_2 and d_1 as L_2.

7.8.4 Calculation of results

Use the following equation to calculate the dimensional change for each specimen to the nearest 0,1 %:

$$\Delta L = 100\left(\frac{L_2 - L_1}{L_1}\right)$$

where

L_1 is the mean of the series of three measurements made between Lines d_1 and d_2, along Line c, on the test block [7.8.2.2.2 c)],

L_2 is the result of only one measurement made between Lines d_2 and d_1, along Line c on the impression material specimen.

7.8.5 Pass and fail determinations and expression of results

See 6.3.7 and 6.3.8.

7.9 Tensile bond strength test (Type 3A agar/companion alginate material specimen only)

7.9.1 Apparatus

a) **Mated set(s) of specimen forming component halves** [Figures A.11 and A.13 b)].

b) **Specimen assembly aligning and supporting trough** [Figure A.12 a)].

c) **Separate tubular specimen support ring** (not illustrated) having diameter and length dimensions approximating those of the **tubular specimen assembly base support ring** fixed to the specimen aligning trough, and of the open ended tubular space provided below the lead shot compartment of the specimen stabilizing weight (see Figure A.12, a, and b).

d) **Metal or plastic disc,** approximately 25 mm in diameter and 1,5 mm thick, to be used for scooping out the alginate cavity (Figure A.13 b) and key item 8) into which the agar will be injected.

e) **Stabilizing weight**, partially tubular [Figure A.12 b)] and Figure A.13 a)].

f) **One humidity chamber** held at the simulated oral environment temperature of (35±2) °C and at a relative humidity of (95 ± 5)%.

g) **Tensile-testing instrument,** capable of measuring a tensile force of at least 50 N at a rate of 500 mm/min.

7.9.2 Specimen preparation

7.9.2.1 Prepare a minimum of five specimens

7.9.2.2 Advance preparation steps

a) Measure the orifices of each mated set of specimen forming component halves and record the diameter for each orifice. Then designate the half having the lesser orifice diameter as being the lower half. Mark each half to clearly indicate which is to be the bottom or top half.

b) Bring increments of the Type 3A agar impression material to liquefaction and then reduce them to the recommended storing temperature [8.2.2 b)] in preparation for syringe use in forming part of the specimen.

c) Proportion the components for the companion alginate mixture.

d) Assemble each of the specimen-forming half components as illustrated in Figure A.11 d).

e) Fix the specimen aligning trough [7.9.1 b)] in position with its long axis slightly off vertical (about 20°), and with the specimen assembly base support ring at the lower end.

7.9.3 Specimen preparation steps

Prepare a mix of the alginate impression material and within 45 s after completion of mixing, accomplish the following 8 steps.

a) Move the entire mix to one side of the mixing bowl so that the orifices of the specimen forming mould halves [7.9.1 a)] can be forced into the mixture repeatedly so as to slightly overfill each specimen forming half.

b) Overfill the top half, strike off the excess protruding above the orifice, invert the mould half and place it to rest temporarily with the top flat surface seated against the top surfaces of the separate tubular support ring [7.9.1 c)].

c) Overfill the lower half with alginate, strike off the excess protruding above the orifice of the half and use the disc [7.9.1 d)] to scoop out a uniform concavity in the alginate at the orifice of the half. (Figure A.13, key item 8).

d) Invert the lower half and place it to rest, with the flat surface of the cap seated on the top surfaces of the specimen support ring fixed to the aluminium trough liner [Figure A.13 a)] with the lateral surfaces of the cap in contact with the walls of the liner, and with the cap retaining pin pointed toward the angle of the liner [Figure A.13a), key item 6].

e) Syringe the liquefied agar to slightly overfill the alginate concavity prepared in the lower half without having the syringe tip contacting surfaces of the concavity.

f) Complete formation of the specimen by carefully forcing the borders of the top half orifice into alignment and contact with the borders of the orifice of the lower half seated in the trough.

g) Seat the tubular end of the stabilizing weight (Figure A.13, key item 7) against the cap of the top half of the specimen forming assembly so as to help stabilize alignment of the joined halves.

NOTE It is possible for one experienced person to conduct the specimen forming procedures described above, but it is easier for two persons to prepare the specimens according to a test schedule much the same as is used when dentists and their assistants cooperate in using the materials in clinical practice.

— Immediately after completing the preceding steps, begin transferring the specimen/trough/stabilizing weight assembly to the relative humidity chamber [7.9.1f)] held at the simulated oral environment temperature and humidity. During the transfer and during the humidity chamber treatment, keep the assembly positioned so that the end of the trough opposite the assembly base support remains slightly elevated (about 20° off horizontal), thereby minimizing possibilities for stressing the agar/alginate interface of the specimen.

— Allow the assembly to remain in the relative humidity chamber for the time recommended in the instructions for leaving an alginate impression in the mouth [8.2.1 c)].

— Immediately thereafter remove the assembly, separate the specimen assembly from the trough, clear excess impression material from around the junction of the two halves, and quickly examine the junction alignment. Do not test misaligned specimens.

7.9.4 Test procedure steps

Within 30 s after removal of the specimen from the humidity chamber, carefully mount the specimen in the tensile-testing instrument [7.9.1 g)] and, immediately thereafter, apply the test load at a rate of 500 mm/min until rupture occurs.

Record the load at rupture for each of the five specimens to the nearest 1 N.

7.9.5 Calculation of results

Use the following equation to calculate the tensile bond strength for each specimen to the nearest 10 kPa.

$$B = \frac{Fx1000}{A}$$

where

B is the tensile bond strength, in kilopascals;

F is the force, in Newtons, required to rupture the specimen;

A is the surface area, in square millimetres, of the inside diameter of the orifice border of the lower specimen-forming half [7.9.2.2a)].

7.9.6 Pass/fail determination and expression of results

See 6.3.7 and 6.3.8.

8 Requirements — Labelling and instructions for use

8.1 Labelling

Labelling of the consumer packages including primary bulk packaging containers not covered by secondary packaging (bottles, bags, canisters, cartons, etc.) shall bear the following information as may be applicable.

a) Trade name (brand name).

b) Name of the manufacturer, or the name of another company authorized by the manufacturer to market the material under a different brand name.

c) **Use by date** (expiry date) as expressed in one of the two examples shown below, each of which requires the numerical part to be presented in six digits according to the provision stated in ISO 8601 for *Calendar date representations with reduced precision*.

EXAMPLE 1 Consisting of words and numerical digits

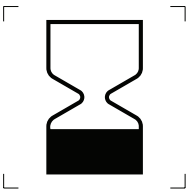
Use by 2012-11

where

the words **Use by** are to indicate that a product cannot be expected to provide for optimum results if used after the expiration of some identified time period, and

the first four of the six numerical digits represent the year 2012, and the last two digits represent the month of November, the month after which the product should not be used.

EXAMPLE 2 Consisting of the standardized hour glass symbol depicted in ISO 15223-1 followed by six numerical digits as shown immediately below



2012 - 11

where the hour glass symbol represents the words **Use by** and the six numerical digits represent the same calendar date explained for EXAMPLE 1.

d) Temperature range and/or other conditions recommended for storage of the unopened package.

e) Additionally for the agar impression materials: The Type identification: either as Type 1, Type 2, Type 3 or Type 3A or, by the word type description, Heavy-bodied, Medium-bodied, Light-bodied or, Light-bodied for reversible-non-reversible systems.

f) Minimum net mass content (g or kg) or net volume (ml) or, where applicable, the number of individual unit packet containers furnished in a consumer package and the minimum net mass content of each such packet.

g) Lot (batch) number(s).

8.2 Requirements — Instructions for use

Each package of impression material, as prepared for retail marketing, shall include the complete instructions for use presented in the order of their application, and without interpolation of promotional information between the individual instructions.

8.2.1 For all hydrocolloid impression materials covered by this International Standard — Agar and alginate

a) Trade name (when the instructions are furnished on a sheet separate from the package labelling).

b) Procedures for users to follow in avoiding degrading effects on the materials such as moisture contamination or moisture loss.

c) Minimum time for the impression to remain in the mouth between completion of seating and removal.

d) Any treatment of the impression required after it has been removed from the mouth and before commencement of disinfection.

e) Maximum time lapse permitted between removal of the impression from the mouth and commencement of pouring the gypsum model.

f) Regular mailing address, telephone number, or electronic mail address of the manufacturer, or of another company authorized by the manufacturer to market the material under a different trade name, through which users can communicate to:

— inquire about behavioural characteristics of the material;

— obtain identification of at least one disinfecting product, by generic active ingredient (glutaraldehyde, hypochlorite, iodophor, etc.) that has been found to be effective for disinfecting impressions made of the material being tested without compromising the surface quality or dimensions;

— obtain detailed instructions, pertinent to disinfection of impressions made of the materials, to include any post disinfectant treatment required before pouring gypsum into the impression;

— identification of at least two dental gypsum products by trade name, both complying with the requirements of ISO 6873, and both of which have been found by the impression material

manufacturer to be compatible with the impression material being tested: one Type 3 product (dental stone, model) and, either one Type 4 or one Type 5 product (dental stone, high strength).

8.2.2 Additional instructions for agar hydrocolloid impression materials only

a) Equipment required for liquefaction, storing and tempering of the materials as may be applicable and the kind of syringe and needle recommended for use with the materials to be syringed.

b) Times, temperatures and procedures recommended for the liquefaction, storing, extrusion and/or tempering the material.

c) Maximum time allowed for the material to remain at the storing temperature before being extruded from the syringe or tube for use in the subsequent step.

d) A **caution** advising users that pain or injury to oral tissues might result if Type 3 and Type 3A materials are extruded onto them at temperatures above 52 °C.

e) Procedures recommended to preventing dilution or excessive water loss of the materials during the liquefaction/storing cycles.

f) Recommended circulating cooling water temperature range along with the time for cooling the impression in the mouth.

g) Number of times the material in an unopened primary container tube can undergo liquefaction for use after it has been exposed to the first liquefaction/gelation cycle.

h) Identification of the companion alginate impression material to be used with a Type 3A agar material formulated for use in a reversible/non-reversible impression material system.

8.2.3 Additional instructions for alginate hydrocolloid materials only

a) A caution against breathing dusts generated during use of the powder material.

b) Any treatment of the powder required prior to mixing to ensure uniform powder particle distribution.

c) For material supplied in powder form, the recommended:

— grade of water for mixing with the powder,

— mixing water temperature,

— powder/water ratio (g/ml).

d) For the paste/paste materials — Mass/mass proportions.

e) Recommended mixing apparatus and procedures for their use.

f) Mixing time range within which a homogeneous mixture of the material can be obtained by a person having adequate practical experience.

g) Working time.

h) Initial setting time.

i) Handling factors that can significantly affect the working time and initial setting time of water/powder and paste/paste alginate mixtures:

— ionic content of the mixing water used with the powder materials;

— water/powder or paste/paste ratios;

— room or mixing water temperature;

— pre-use moisture contamination of the powder or materials;

— contamination of the mixing apparatus;

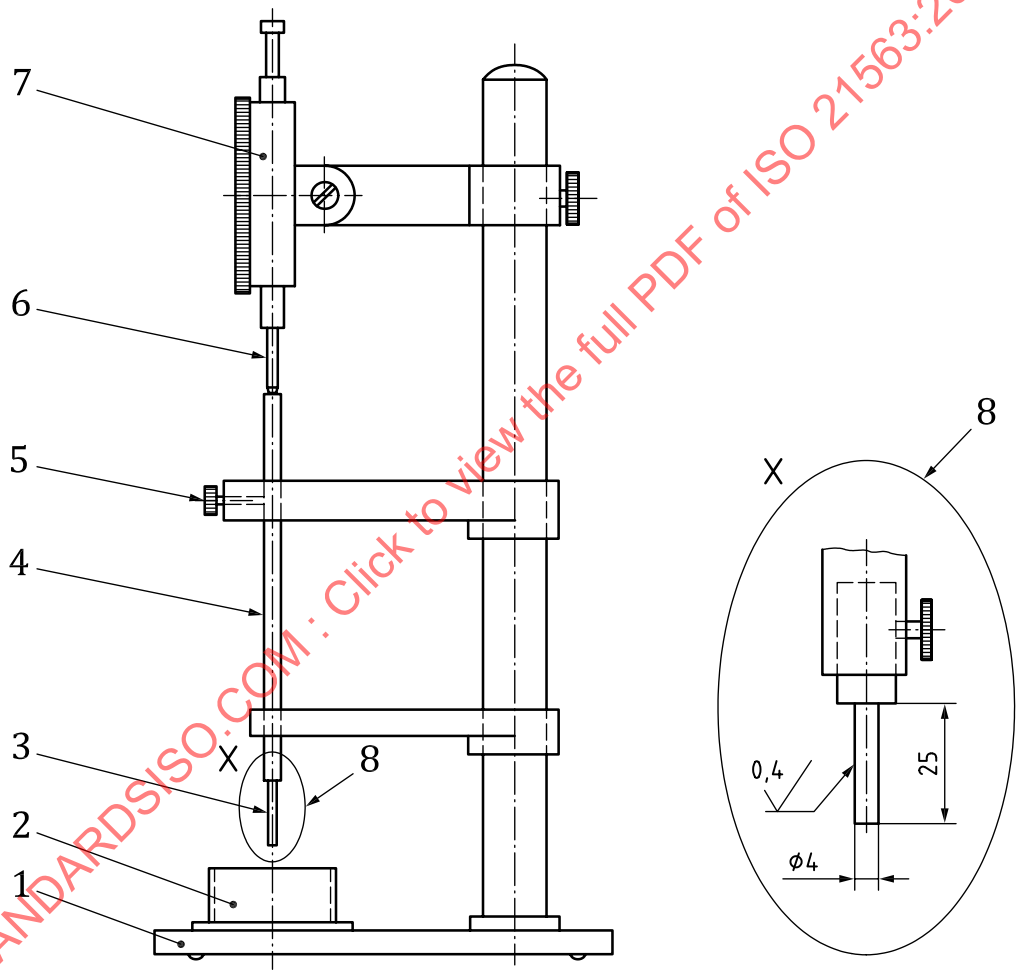
— speed and friction of mixing.

Annex A
(normative)

Figures illustrating instruments and accessories used in tests

All of the figures in this annex, except Figures A.6 and A.13, specify dimensions for the instrument parts and accessories depicted. Other helpful related information is provided in the key sections of the figures, in 6.3.2 and in the apparatus and materials subclauses of the main body of the standard.

Dimensions in millimetres

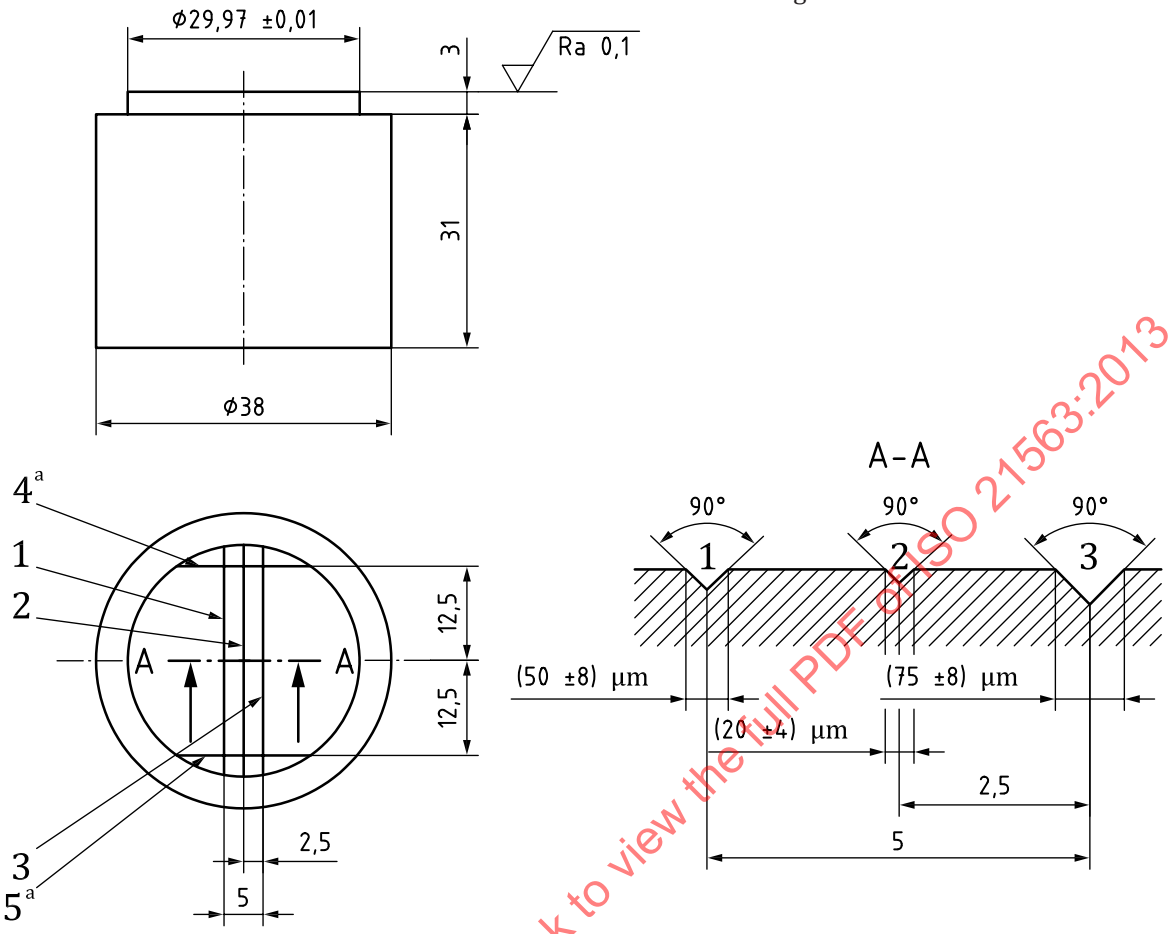


Key

1 instrument base
2 specimen containing ring mould/glass plate assembly
3 test instrument penetrator tip
4 penetrator shaft with tip attached: total mass of 50 g
5 shaft locking knob and screw
6 dial indicator spindle
7 dial indicator having divisions of 0,01 mm
8 method of attaching the penetrator tip to the shaft and related dimensions

Figure A.1 — Working time test instrument — Alginate impression material

Dimensions in millimetres and, unless otherwise specified, the tolerance for the dimensions, shall be + 0,1 mm.
The test block shall be made of cast or wrought corrosion resistant austenitic steel.

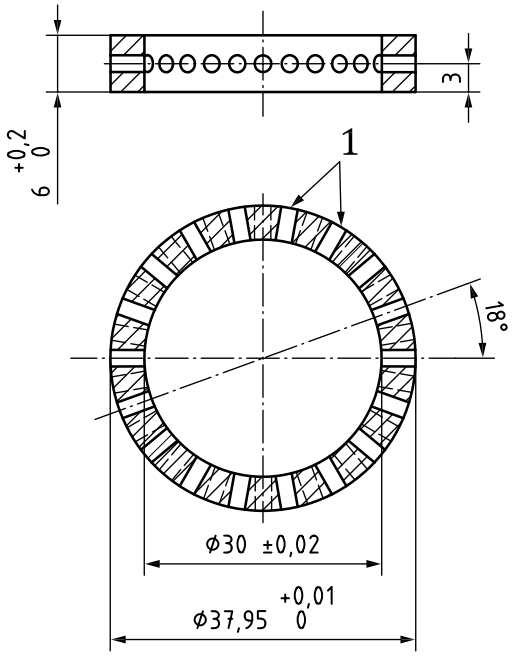


Key

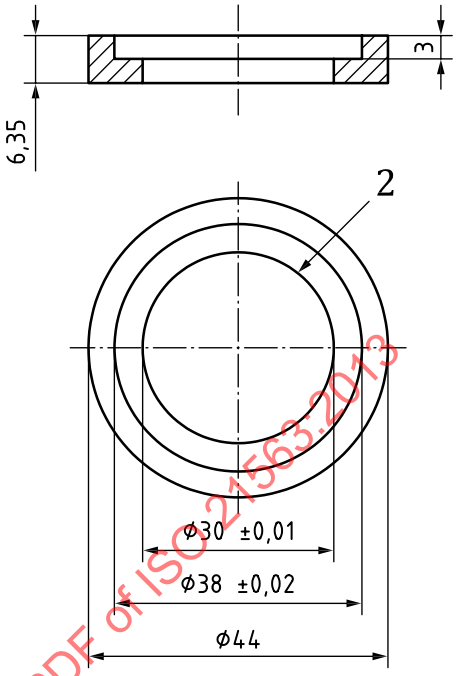
1 line a
2 line b
3 line c
4 line d_1
5 line d_2
a Lines d_1 and d_2 shall be the same width as line c.

Figure A.2 — Detail reproduction test — Lined test block

Dimensions in millimetres



a) Specimen forming ring mould [a]



b) Ring mould retainer [a]

Key

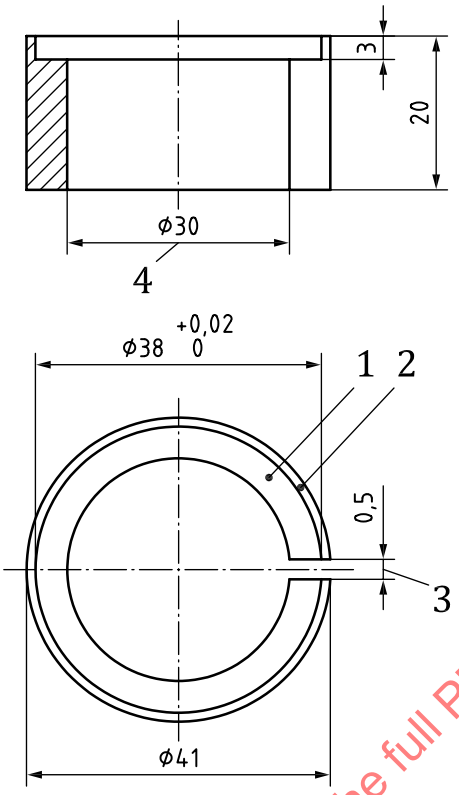
1 specimen material retention holes: 18 holes in each of two rows with the holes having nominal diameters of 2 mm

2 floor of the specimen forming ring mould retainer

[a] Specimen forming ring mould and retainer made of anodized aluminium, brass or corrsoion-resistant steel.

Figure A.3 — Detail reproduction test — Specimen forming accessories

Dimensions in millimetres

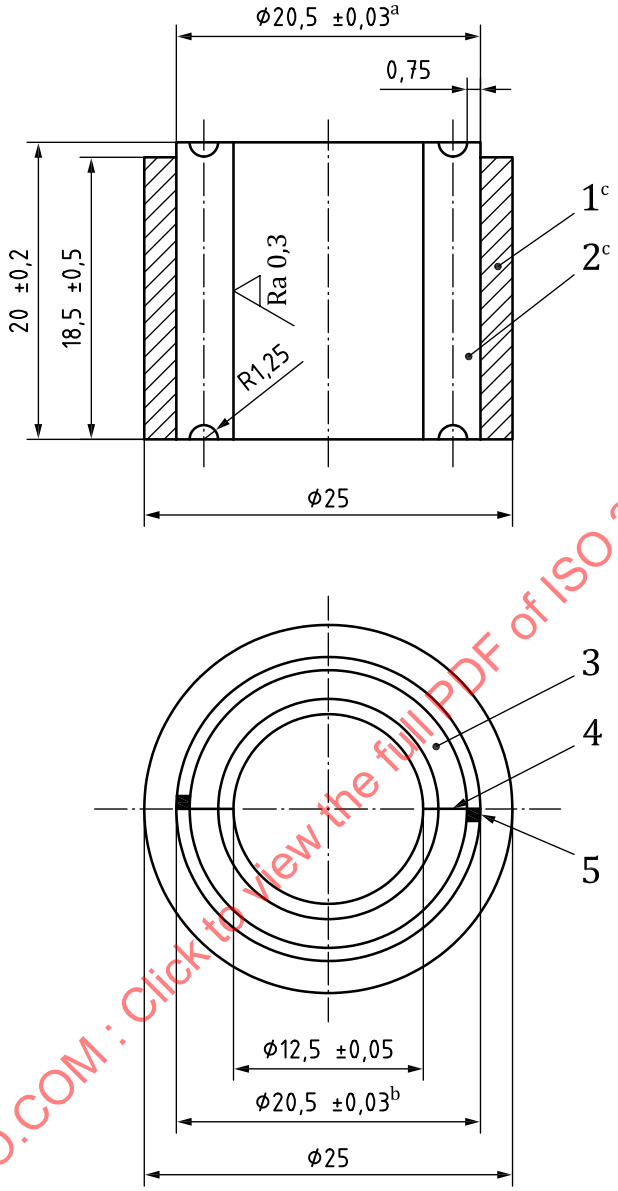


Key

1 floor of specimen forming assembly retaining recess
2 recess rim
3 width of slit in wall of mould before the slit has been closed
4 internal diameter of slit mould after the slit has been closed

Figure A.4 — Compatibility with gypsum test — Specimen forming slit mould

Dimensions in millimetres. Surface texture is 3,2 micrometres unless otherwise specified



Key

1 fixation ring

2 split mould half, no bell mouth in bore of the assembly

3 grooved top and bottom surfaces of the split mould halves

4 split mould half interface

5 spillways (1 mm wide x 1 mm deep) cut through the external rims of the split mould halves adjacent to their interfacing end surfaces

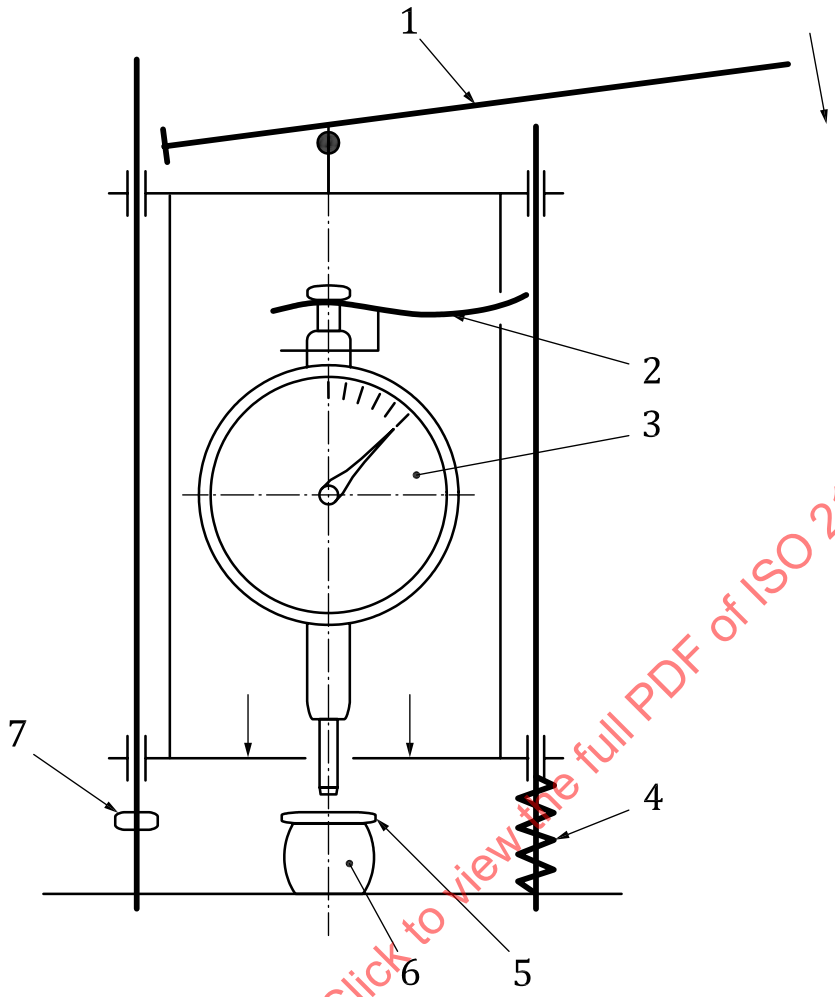
a Outside diameter of the split mould halves assembly.

b Inside diameter of the fixation ring.

c Corrosion-resistant steel.

Figure A.5 — Elastic recovery test — Specimen forming split mould assembly

Dimensions in millimetres

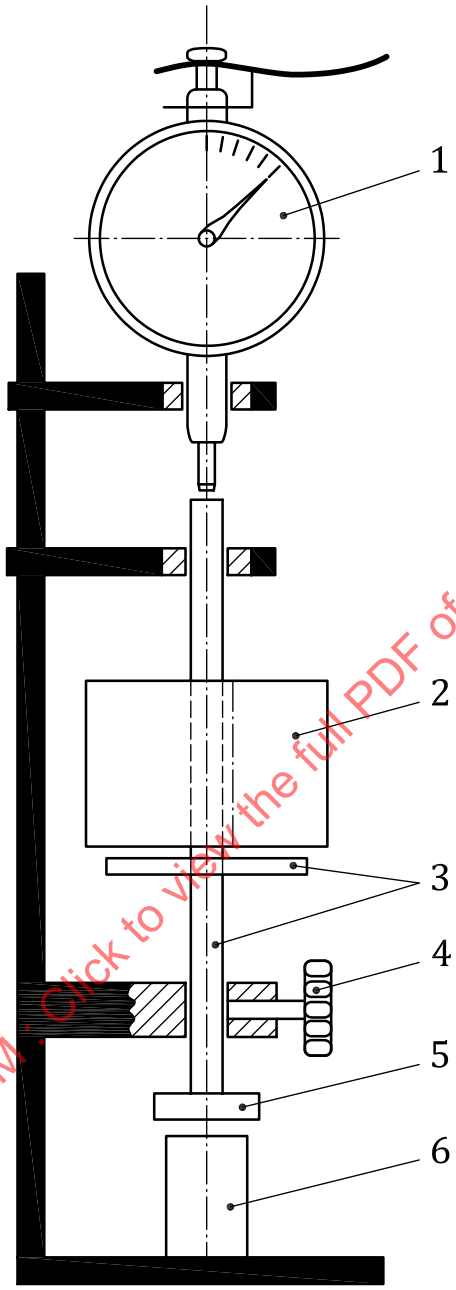


Key

1 level for activating the required compressive force
2 dial indicator spindle position control lever
3 dial indicator having divisions of 0,01 mm
4 spring (optional)
5 test plate
6 specimen compressed to limit
7 compressive force stop set to limit compression of the specimen to (4,0 ± 0,1) mm

Figure A.6 — Elastic recovery test instrument

Dimensions in millimetres



Key

1 dial indicator having divisions of 0,01 mm

2 slotted weight having a mass which, when added to the mass of the loading shaft (item 3), will provide for the total force of (12,2 ± 0,1) N needed to complete the strain-in compression test

3 loading shaft which, along with its weight support collar, has a total mass that provides for the initial force of (1,2 ± 0,1) N needed for the test

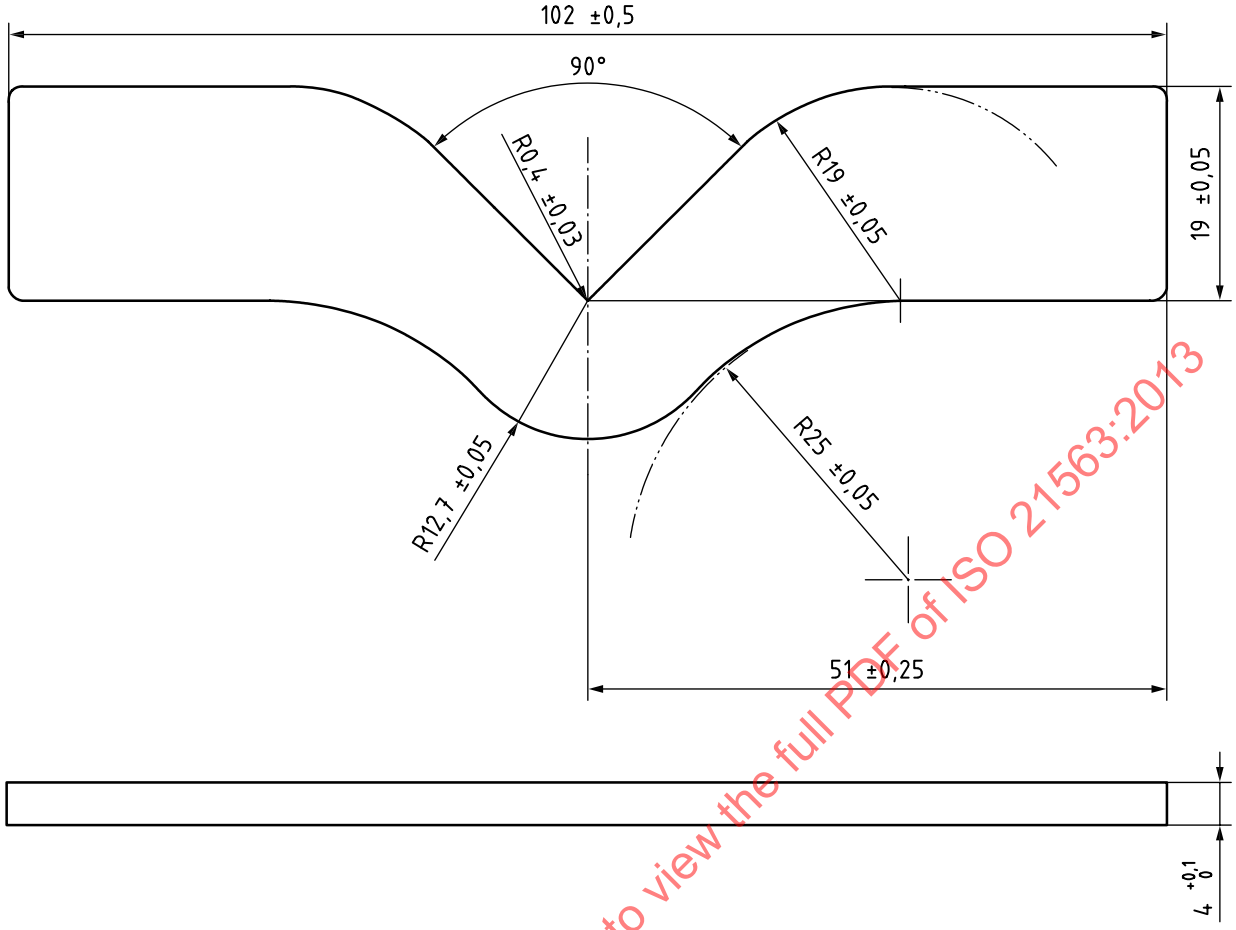
4 shaft locking knob and screw

5 shaft foot, 12 mm Ø

6 specimen

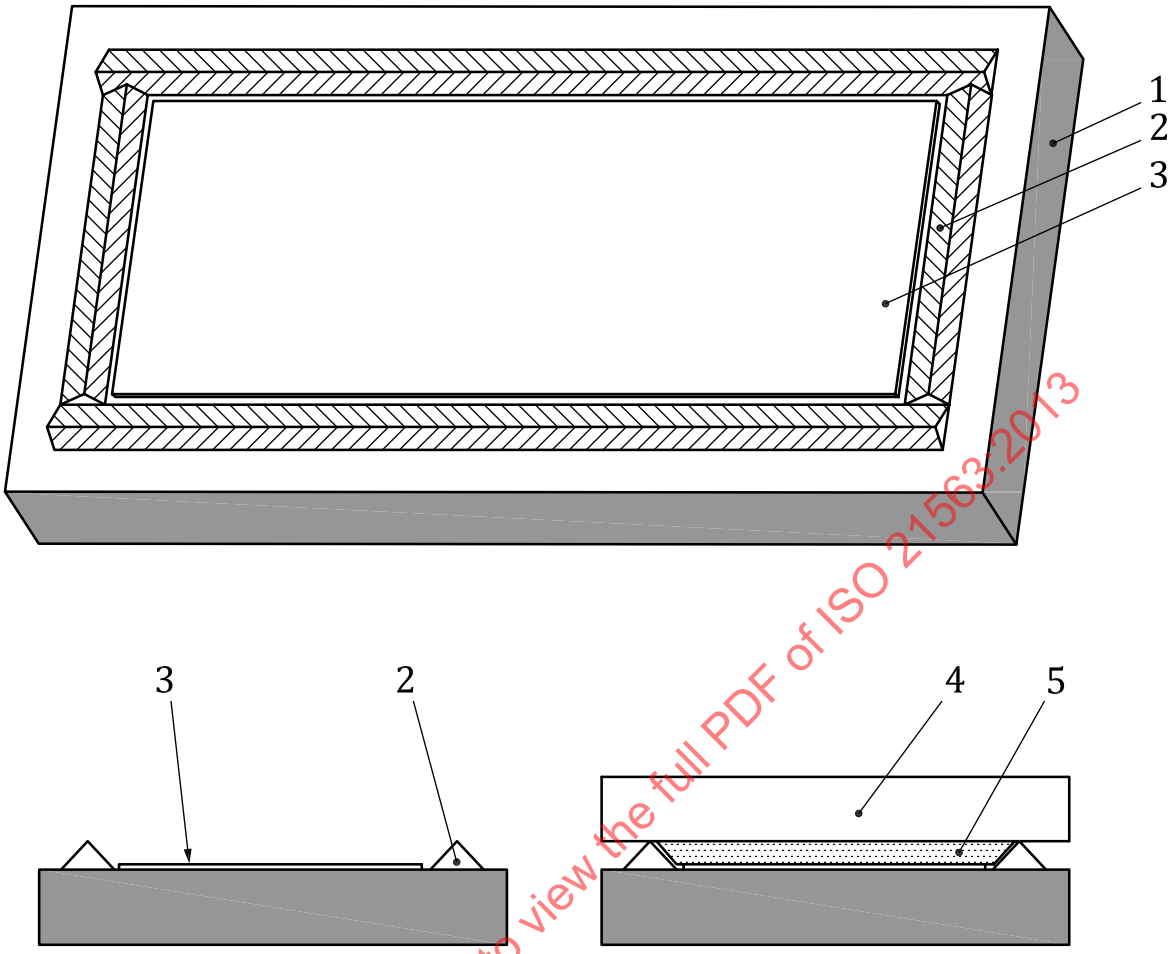
Figure A.7 — Strain-in-compression test instrument

Dimensions in millimetres



NOTE Radii of the curved surfaces are tangential to the straight surfaces.

Figure A.8 — Tear test specimen shape dimensions



Key

1 mould cavity base: for example, a glass dental cement mixing slab of approximate dimensions (154 × 75 × 12) mm; or, thinner sheets of glass having approximately the same length/width dimensions, which are cemented in layers to produce a base having essentially the same dimensions

2 mould cavity border: consisting of four triangular rod stock pieces, adjusted in height and cemented to the base so as to form a mould cavity (4,0 + 0,1) mm deep (see specimen thickness, Figure A.8) and a mould cavity floor 120 mm long and 45 mm wide; the wall surfaces of the moulds cavity border shall have no undercut areas that will cause stress to the specimens during their separation from the mould

3 mould cavity floor: sometimes adjusted to height by a thin sheet wax or polymer sheet additions, in order to provide for the desired mould cavity depth

4 mould cavity cover: glass slab having essentially the same dimensions as the mould cavity base

5 specimen material

Figure A.9 — Tear test — Specimen sheet forming mould