INTERNATIONAL STANDARD

ISO 13716

First edition
1999-05-01

Dentistry — Reversible-irreversible hydrocolloid impression material systems

Art dentaire — Systèmes de produits pour empreintes à base d'hydrocolloïdes réversibles-irréversibles

Reference number
ISO 13716:1999(E)

Contents

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Printed in Switzerland

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 13716 was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 2, *Prosthodontic materials*.

Introduction

Specific qualitative and quantitative requirements for freedom from biological hazards are not included in this ISO Standard. But it is recommended that, in assessing possible biological or toxicological hazards, reference be made to ISO 7405 and ISO 10993-1.

Dentistry — Reversible-irreversible hydrocolloid impression material systems

1 Scope

This international Standard specifies requirements and test methods for tensile bond strength and linear dimensional change of reversible-irreversible hydrocolloid impression materials used in dentistry, as well as requirements for their labelling and manufacturer's instructions.

This International Standard is applicable to those alginate and syringeable agar dental impression materials which have been formulated such that they will bond to each other, when used in combination, to provide elastic impressions of oral tissues.

NOTE Requirements for other characteristics and properties of these impression materials are given in ISO 1563 and ISO 1564.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 1563:1990, *Dental alginate impression material.*

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

ISO 1942 (all parts), *Dental vocabulary.*

3 Terms and definitions

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

3.1
bond
⟨between dental reversible and irreversible impression materials⟩ adherence of the materials to each other after both materials have set

3.2
storing
⟨of agar impression material⟩ conditioning of the material, immediately after liquefaction, to reduce and maintain the temperature required for use in a succeeding step

3.3
tensile bond strength
⟨of reversible-irreversible impression material specimen⟩ force per unit area required to create a rupture in a specimen tested in the tensile mode

4 Requirements

4.1 Biocompatibility

See Introduction.

4.2 Requirements given in ISO 1563 and ISO 1564 for characteristics and properties

When a manufacturer identifies any alginate material as being acceptable for use in combination with a particular agar material, each alginate material so identified shall be subject to all applicable requirements and tests stated in this International Standard and in ISO 1563 for alginate materials. Likewise, any agar material identified for use with a particular alginate material shall be subject to all applicable requirements and tests stated in this International Standard and to those in ISO 1564, except for the gelation temperature requirement (see clause 2).

4.3 Requirements given in this International Standard for characteristics and properties

4.3.1 Tensile bond strength

When tested in accordance with clause 7, the tensile bond strength between the agar and alginate shall not be less than 50 kPa.

4.3.2 Linear dimensional change

When tested in accordance with clause 8, the negative dimensional change of the combined impression material system specimens shall not be more than 1,0 % when measured at 20 min after commencement of mixing of the alginate component.

4.4 Requirements for instructions for use, packaging and labelling

The materials shall be subject to applicable requirements specified in clauses 9 and 10 of this International Standard and to requirements specified for instructions, packaging and labelling in pertinent clauses of ISO 1563 and ISO 1564.

5 Sampling

Samples shall be procured from a single manufacturing batch as packaged for retail marketing, to include the manufacturer's instructions.

6 Test methods — General

6.1 Laboratory conditions

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

6.2 Apparatus function verification

Before using any accessories, instruments or other items of equipment, calibrate or otherwise determine whether they comply with specifications given in this International Standard or in a related supporting standard.

6.3 Material manipulation and specimen preparation

Unless otherwise specified in this International Standard, prepare and manipulate the materials used for forming the test specimens using the equipment and procedures recommended in the manufacturer's instructions (see clause 9 of this International Standard and corresponding pertinent clauses in ISO 1563 and ISO 1564).

Prepare a mix consisting of 15 g of alginate powder with the specified amount of distilled or deionized water for making each tensile bond strength and linear dimensional change test specimen.

One stick (approximately 2,5 ml to 3,5 ml) of the agar material is needed for each specimen.

6.4 Pass/fail determinations

Prepare a series of five specimens initially for each of the tests. If four of the five specimens comply with the specified requirement, the material passes. If only one or two specimens comply, the material fails. If only three specimens comply, test an additional series of five specimens. If eight of the ten specimens tested in the two series comply, the material passes; otherwise the material fails.

6.5 Expression of test results

Report the number of specimens tested, the number of specimens complying with the specified requirement, and whether the material passes or fails.

6.6 Pass/fail determinations and expression of results for tests specified in ISO 1563 and ISO 1564

Use criteria specified in ISO 1563 and ISO 1564.

7 Tensile bond strength test

7.1 Apparatus

7.1.1 Apparatus for preparing the agar

7.1.1.1 Two water baths, one for liquefying the agar and one for storing the liquefied material.

7.1.1.2 Temperature-measuring device, such as 76 mm immersion calibrated thermometer having graduations of 0,1 °C.

7.1.1.3 Syringes and needles, for use in dispensing the agar in clinical use, and wire probes for cleaning syringe needle barrels. The inside diameters of the syringe needles shall be the same as those recommended by the manufacturer for making crown and fixed partial denture impressions [9.2.1 a)].

Before using the syringes, verify their ability to prevent aspiration of water during the storing cycle.

7.1.2 Apparatus for preparing the alginate

7.1.2.1 Container, capable of being sealed, for storage of the alginate to protect it from moisture contamination after initial opening of the package.

7.1.2.2 Weighing boat.

7.1.2.3 Balance, accurate to 0,1 g.

7.1.2.4 Scoop for removing alginate powder from the container.

7.1.2.5 Graduated cylinder or syringe for measuring water.

7.1.2.6 Distilled or deionized water, at the temperature required for mixing the alginate according to the manufacturer's instructions.

7.1.2.7 Temperature-measuring device (7.1.1.2) for measuring water temperature.

7.1.2.8 Mixing equipment or utensils recommended by the manufacturer. When hand mixing is recommended, the bowl shall have a depth of approximately 90 mm and an opening of approximately 120 mm in diameter.

7.1.2.9 Timing device, such as a stopwatch.

7.1.3 Apparatus for forming and testing specimens

7.1.3.1 Mated set(s) of **specimen-forming component halves** (Figure 1) with each mated set marked to indicate which half is to be designated as the top and which is to be the bottom. The bottom half for each mated set shall be the half for which the lesser orifice diameter has been recorded. The recorded diameter for the orifice of each half shall be determined by calculating the average of two internal diameter measurements, made at right angles to each other, for each orifice.

7.1.3.2 **Tubular support** for the top specimen-forming half (Figure 3).

7.1.3.3 **Metal or plastic disc**, approximately (25 ± 0,5) mm in diameter and (1,30 ± 0,5) mm thick, to be used for shaping the alginate cavity into which the agar will be injected.

7.1.3.4 **V-trough** (Figure 2 and Figure 3).

7.1.3.5 **Weight** (Figure 3).

7.1.3.6 **Humidor**, capable of maintaining a temperature of (35 ± 1) °C and (95 ± 5) % relative humidity, to simulate the temperature and humidity of the oral environment.

7.1.3.7 **Tensile-testing instrument**, capable of applying a test load at a rate of 500 mm/min and accurate to 1 N.

7.2 Advance preparation steps

Use the temperature-measuring device (7.1.1.2) to verify the temperature of the oral-environment-simulating humidor (7.1.3.6).

Assemble each of the specimen-forming halves as illustrated in Figure 1.

Position the V-trough (7.1.3.4) with its long axis slightly off vertical (about 20°), with the specimen assembly base support at the lower end.

Use the water baths (7.1.1.1) to liquefy the agar and to store the agar-filled syringes according to the manufacturer's instructions.

Measure the amounts of alginate powder and water needed for a mixture of the alginate component.

When the agar material in the syringe has reached the storing temperature, remove the needle cap from the syringe and return the syringe to the storing bath.

7.3 Specimen preparation (five specimens)

Prepare a mix of the alginate impression material (6.3) and within 45 s after completion of mixing, accomplish the following steps.

— Move the entire mix to one side of the mixing bowl so that the orifices of the specimen-forming halves (7.1.3.1) can be forced into the mixture repeatedly to slightly overfill each half.

— Overfill the specimen-forming top half with alginate, strike off the excess, and place that half to rest temporarily, with the orifice upward, on the tubular support [Figure 3 b)].

— Overfill the bottom half with alginate, strike off the excess and use the disc (7.1.3.3) to scoop out a uniform concavity in the alginate at the orifice of the half.

— Inject the agar to overfill the concavity in the alginate.

— Seat the bottom half in the V-trough (7.1.3.4) with the flat surface of the cap in contact with the specimen assembly base support, with lateral surfaces of the cap in contact with the internal walls of the V-trough, and with the cap-retaining pin in contact with the bottom of the V-trough.

  At this stage, keep the trough in a vertical, or near vertical, position to prevent the agar from flowing out of the cavity.

— Carefully force the borders of the orifice of the top half into alignment and contact with the borders of the orifice of the bottom half seated in the trough.

— Seat the open end of the weight (7.1.3.5) against the flat surface of the top half to hold the two halves in alignment.

NOTE It is possible for one experienced person to conduct the specimen-forming procedures described above, but it will be easier for two persons to prepare the specimens according to the test schedule in much the same manner that dentists and their assistants cooperate in clinical practice.

Immediately after completing the preceding steps, transfer the trough/specimen/weight assembly to rest horizontally on the floor of the humidor (7.1.3.6). During the transfer, keep the assembly, with the specimen assembly base support at the lower end, slightly tilted from the horizontal so as to avoid any motion that will disturb the agar/alginate interface.

Allow the assembly to remain in the humidor for the time the manufacturer recommends for leaving the impression in the mouth (9.2.3).

7.4 Tensile test procedure

After removal of the assembly from the humidor, separate the specimen from the assembly, clear excess impression material from around the junction of the two halves and quickly examine the junction for alignment. (Do not test misaligned specimens.)

Carefully mount the specimen in the tensile-testing instrument (7.1.3.7) within 30 s after removal from the humidor. Immediately thereafter, apply the test load at a rate of 500 mm/min until rupture occurs.

Examine the ruptured specimen for defects which may have contributed to failure. Do not report values for defective specimens.

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

7.5 Calculation of results

Calculate the tensile bond strength using the formula:

$$B = \frac{F \times 981}{A}$$

where

B is the tensile bond strength, in kilopascals,

F is the tensile force, in newtons, required to rupture the specimen;

A is the surface area, in square millimetres, of the orifice of the bottom specimen-forming half (7.1.3.1).

Report the tensile bond strength values for each specimen to the nearest 10 kPa.

7.6 Pass/fail determinations and expression of results

See 6.4 and 6.5.

8 Linear dimensional change test

This test shall be conducted for each agar/alginate combination recommended by the manufacture (see 4.2 and 9.1).

8.1 Apparatus

8.1.1 Detail reproduction lined test block, illustrated in ISO 1563 and ISO 1564.

8.1.2 Impression-material specimen tray (Figure 4). When the manufacturer of the alginate material recommends the use of a tray adhesive, the surface of the impression tray which will be in contact with the alginate shall be treated with the recommended adhesive.

8.1.3 Syringes (7.1.1.3).

8.1.4 Mould-release agent, such as a fresh 1 % solution of tetradecylamine in acetone, to prevent the alginate from adhering to the lined test block.

8.1.5 Timing device, such as a stopwatch.

8.1.6 Measuring microscope, accurate to 0,01 mm, equipped for ×4 to ×12 magnification, low-angle illumination, and a measuring travel of at least 27 mm.

8.1.7 First humidor (7.1.3.6) (oral environment simulation).

8.1.8 Second humidor, maintained at ambient laboratory conditions of (23 ± 2) °C and (50 ± 10) % relative humidity.

8.2 Advance preparation steps

— Clean the test block (8.1.1) ultrasonically and inspect the lines via magnification to assure that they have been cleared of debris.

— Paint the top-most surfaces of the test block, including the lined surface, the sides and the shoulder, with the mould-release agent (8.1.4).

— Place the test block in the first humidor (8.1.7) for conditioning at (35 ± 1) °C for at least 15 min.

8.3 Test block line length measurement procedures

8.3.1 Initial test block positioning

— Position the test block on the microscope (8.1.6) stage with Line d_1 to the right as shown in Figure 5.

— Relate the x-axis of the microscope cross-hair parallel to, and approximately 0,03 mm below, Line c as shown in Figure 5 c). This will place the y-axis of the cross-hair parallel to Lines d_1 and d_2.

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

8.3.2 Test block line measurement steps

To avoid errors in measurements made after positioning the test block according to the last step in 8.3.1, do not reverse the direction of travel for the microscope slide or stage at any point until after the final measurement between Lines d_1 and d_2 has been recorded.

— Move the inner edge of the y-axis of the cross-hair into alignment with the inner edge of Line d_1, stop the travel motion, and record the reading for this position as the initial measurement.

— Then move the inner edge of the y-axis of the cross-hair into alignment with the inner edge of Line d_2, stop the travel motion, and record the reading for this position as the final measurement.

— Calculate and record the difference between the initial and final readings. Make two additional measurements of the distance between Lines d_1 and d_2, average the three values, and record the result as L_1.

8.3.3 Specimen preparation (five specimens)

Proceed with the following specimen preparation steps in rapid succession.

— Make an alginate mix and use an increment to slightly overfill the specimen tray (8.1.2) and strike off the excess.

— Remove the test block from the humidor and dispense 1 ml to 2 ml of the agar material from a syringe to cover the lines on the block.

— Press the alginate-filled specimen tray over the agar material with sufficient force to seat the tray on the test block and extrude the excess material.

— Place the specimen-forming assembly in the first humidor (8.1.7) and allow it to remain 5,5 min from the commencement of mixing the alginate.

— Then remove the assembly from the first humidor, separate the specimen in the tray from the assembly, and place the specimen in the tray in the second humidor (8.1.8), and allow it to remain until 19 min after commencement of mixing the alginate.

8.3.4 Test specimen measurement

Upon completion of the humidor storage period, transfer the specimen in the tray to the microscope stage so that Line d_a is to the right, as shown in Figure 5 b), then proceed with for measurement of the distance between Lines d_1 and d_2, along Line c, following the procedure specified in 8.3.2, with the following exception: the three measurements required for each specimen shall be completed within 90 s after placement of the specimen on the microscope stage.

Record the average of the three measurements made for each specimen as L_2.

8.3.5 Calculation of results

Calculate the dimensional change, ΔL, as a percentage, using the formula:

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

where

L_1 is the average of the three measurements made between Lines d_1 and d_2, along Line c, on the test block.

L_2 is the average of the three measurements made between Lines d_2 and d_1, along Line c on the impression material specimen.

Report the percentage dimensional change for each specimen.

8.3.6 Pass/fail determinations and expression of results

See 6.4 and 6.5.

9 Information required in manufacturer's instructions

9.1 Requirements in ISO 1563 and ISO 1564

When an agar or alginate material is claimed to be satisfactory for use, either in a combined reversible-irreversible system or in a single-material impression procedure, each package shall be accompanied by instructions specifying how the material should be used to obtain optimum performance with each method. The instructions, therefore, shall include the directions for use specified in ISO 1563 and ISO 1564 (as applicable) as well as those given in 9.2.

9.2 Additional instructions required for combined systems

9.2.1 For agar materials

a) Brand name of the syringe and diameter of the lumen of the needle (7.1.1.3) used for syringing the material.

b) Maximum time allowed between completion of syringing the agar and seating the impression tray containing the alginate material.

c) Name of the brand or brands of alginate material recommended for use with the agar in the combined material impression system.