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Dentistry — Determination of the strength of dental amalgam by the Hertzian indentation strength (HIT) method

Médicine bucco-dentaire — Détermination de la résistance des amalgames dentaire par l'indentation hertzienne (HIT) méthode

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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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

The committee responsible for this document is ISO/TC 106, *Dentistry*, Subcommittee SC 1, *Filling and restorative materials*.

Introduction

This document gives the practical details of the test method for the measurement of the strength of a dental amalgam by the Hertzian indentation (HIT) method. In this test, the specimen geometry and the localized application of force leads to radial crack formation at the surface opposite the one to which the force is applied and under the point at which it is applied. This protocol produces a loading condition similar to that encountered during normal oral function.

When a requirement is agreed, it is the intention of ISO/TC 106/SC 1 to consider the inclusion of this test procedure in the ISO standards for dental amalgam.

This method for measuring the strength of a brittle material (and materials that have very low plasticity) can be applied to other dental restorative materials.

Dentistry — Determination of the strength of dental amalgam by the Hertzian indentation strength (HIT) method

1 Scope

This document gives the practical details of the test method for the measurement of the strength of a dental amalgam by the Hertzian indentation strength test (HIT) method.

It is applicable to dental amalgam formed from products that are within the scope of ISO 24234 and ISO 20749.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 6344-1, *Coated abrasives — Grain size analysis — Part 1: Grain size distribution test*

ISO 7488, *Dental amalgamators*

ISO 13565-2, *Geometrical Product Specifications (GPS) — Surface texture: Profile method; Surfaces having stratified functional properties — Part 2: Height characterization using the linear material ratio curve*

ISO 13897, *Dentistry — Amalgam capsules*

ISO 24234, *Dentistry — Dental amalgam*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1
dental amalgam alloy
alloy in fine particles, composed mainly of silver, tin and copper, which, when mixed with *dental mercury* (3.2), produces a dental amalgam

3.2
dental mercury
mercury supplied for use in the preparation of dental amalgam

3.3
pre-capsulated dental amalgam product
dental amalgam product supplied in a sealed capsule that contains measured amounts of *dental amalgam alloy* (3.1) powder and *dental mercury* (3.2) with masses that are appropriate for the production of a mass of dental amalgam that is considered to be suitable for a single small or medium size restoration in a single tooth

Note 1 to entry: The dental amalgam alloy powder and dental mercury are separated by a barrier that is broken immediately prior to mixing to allow their contact. The capsule remains sealed until mixing has been completed.

3.4
dental amalgam alloy tablet
dental amalgam alloy (3.1) powder that has been compressed to form a single entity for the purpose of providing a pre-dosed quantity of the alloy that, when mixed with an appropriate mass of *dental mercury* (3.2), produces a mass of dental amalgam that is considered to be suitable for a single small or medium size restoration in a single tooth

Note 1 to entry: During mixing, the tablet is intended to break apart, forming a fine powder.

3.5
dental mercury sachet
measured quantity of *dental mercury* (3.2) supplied in a sachet (for use in a reusable mixing capsule) in a mass that, when mixed with an appropriate mass of *dental amalgam alloy* (3.1), produces a mass of dental amalgam that is considered to be suitable for a single small or medium size restoration in a single tooth

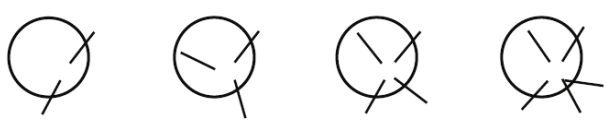
Note 1 to entry: The sachet is intended to rupture during mixing to allow the dental mercury to come into contact with the dental amalgam alloy.

3.6
top surface of the test-piece
surface of the disc shaped test-piece that has been produced by carving back unset amalgam that is above the level of the mould until the surface of the test-piece is flat and level with that mould surface

3.7
test surface of the test-piece
surface of the disc-shaped test-piece that has been produced by contact with the polished glass plate when the mixed amalgam is packed into the mould

3.8
radial cracking
fracture pattern of Hertzian indentation test-piece disc in which (more or less) planar cracks form along radii, normal to the face of the disc, thus dissecting it into two or more sectors

EXAMPLE Some radial fracture patterns in disc shaped test-pieces are illustrated here.



Note 1 to entry: Such radial cracks initiate on the test surface of the test-piece and propagate through the disc to produce approximately equiangular dissection in most cases.

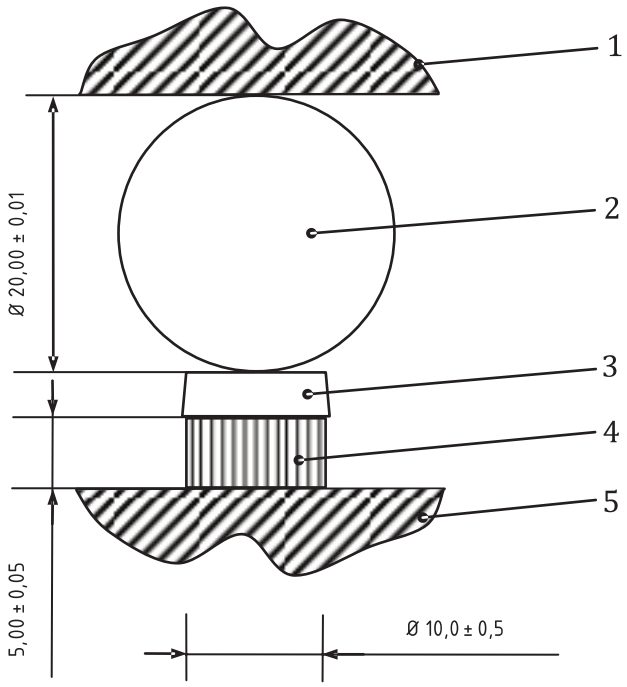
4 Hertzian indentation strength test (HIT) applied to dental amalgam

This test utilizes a disc-shaped test-piece to which a linearly increasing force is applied through a hard steel ball sited at the centre of one of the flat faces of the disc. The other face of the disc is in contact with a smooth substrate that is also disc shaped and thicker than the test-piece. The substrate has a Young's modulus value that is of the same order as that of dentine. Fracture is produced by radial cracks initiating at the surface in contact with the substrate. By having close control of the test-piece, the

resistance to fracture is quantified by the fracture force. Dental amalgam products are compared using the values of the fracture force.

A vertical section through the apparatus required for testing is shown in Figure 1.

Dimensions in millimetres



Key

1 universal mechanical testing machine upper compression platen
2 chrome steel bearing ball
3 dental amalgam test-piece (see Figure 2 for the dimensions and dimensional tolerances for this)
4 30 % glass-filled polyamide disc
5 universal mechanical testing machine lower compression platen

Figure 1 — Loading arrangement for the Hertzian indentation strength test (HIT)

5 Apparatus for the production of test-pieces

5.1 Equipment

5.1.1 Mould, as shown in Figure 2.

5.1.2 Flat square scratch-free polished **glass plate** with an edge length greater than 30 mm.

5.1.3 Glass **microscope slide** to provide a straight edge to carve back the dental amalgam.

5.1.4 Hand-instrument for dental amalgam packing.

5.1.5 Dental amalgam mechanical mixer.

5.2 Specifications for the mould

The mould shall be made of hardened tool steel or hardened stainless steel. The upper and lower surfaces shall be flat and parallel and have a core roughness depth (R_k) not greater than 6,3 µm when tested in accordance with ISO 13565-2. The hole shall have a taper of (7 ± 2)° to allow the amalgam disc to be ejected without undue force when this is applied to the face that has the smaller diameter for the hole. The tapered surface shall be smooth enough not to impede the ejection of the test-piece. For example, it may be honed to a core roughness depth (R_k) of 6,3 µm (when tested in accordance with ISO 13565-2).

NOTE 1 For convenience, to distinguish between the two surfaces during test-piece production, a small engraved mark (set away from the hole) can be made on one of the mould faces.

NOTE 2 The angle of the taper, (7 ± 2)°, is the included angle. The wall of the mould is at an angle of (3,5 ± 1,0)° with the centre line.

Dimensions in millimetres

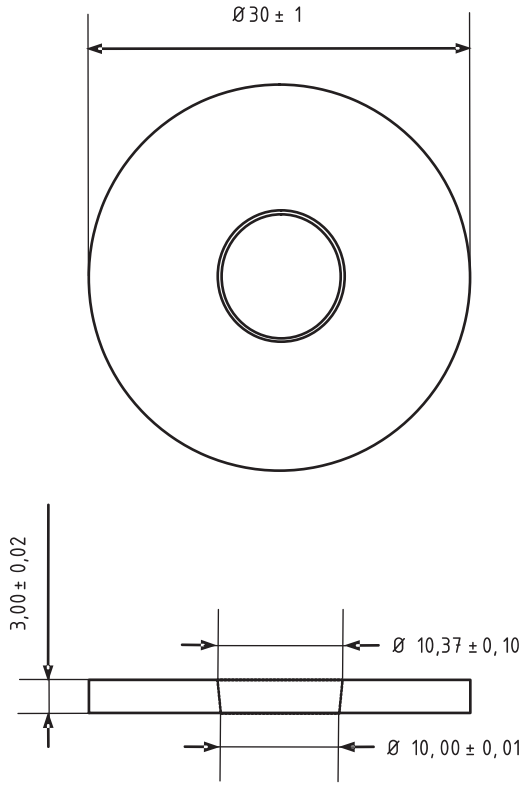


Figure 2 — Mould to produce test-pieces for the Hertzian indentation strength test (HIT)

6 Sampling

Procure sufficient dental amalgam alloy and dental mercury from single lots that have been produced for retail. The required mass of dental amalgam alloy will depend upon the dental amalgam alloy: dental mercury mixing ratio. Depending on the product, a total of the order 3,2 g of mixed amalgam will be required for each test-piece.

7 Preparation of dental amalgam test-pieces

7.1 Mixing the dental amalgam

Prepare test-pieces at (23 ± 2) °C.

For a dental amalgam alloy product supplied either as tablets or as a free-flowing powder in bulk, the ratio by mass of the dental amalgam alloy to that of the dental mercury shall be the ratio recommended by the manufacturer. Use dental mercury sachets that comply with ISO 24234. Use a capsule (with a pestle, if needed) that complies with Type 2 of ISO 13897. Use any other mixing accessory that is required, as recommended by the manufacturer. If more than one mix is required to make the test-piece, produce these mixes simultaneously using equipment of the same type for each mix. However, if the last mix can be produced within the working time of the first mix, mixing these masses sequentially on a single piece of equipment is allowed.

For pre-capsulated products, use as many capsules as needed. Mix the contents of the capsules either simultaneously using the same number of pieces of equipment of the same type, or sequentially on a single piece of equipment. (The latter is allowed, provided the mixing of the last capsule is completed before the end of the working time of the first.) If necessary, use only a portion of the dental amalgam mix from one of these capsules.

Use a mechanical mixing machine for dental amalgam that complies with ISO 7488 and that is recommended for mixing the dental amalgam alloy product with dental mercury or mixing the pre-capsulated product. Use the dental amalgam mechanical mixer setting and mixing time that is recommended by the manufacturer of the dental amalgam alloy or pre-capsulated product (for the mass of dental amalgam alloy that is being mixed).

Mix a mass of the dental amalgam sufficient to make a disc-shaped test-piece that is 10 mm in diameter and 3 mm high after packing into the mould shown in Figure 2.

7.2 Test-piece production

7.2.1 Packing the mould, removal of the test-piece and inspection for surface defects

Place the steel mould on the glass plate with the side that has the greater diameter for the tapered hole in contact with the plate.

NOTE The surface of the glass plate acts as a matrix for the test surface of the test-piece.

Pack the dental amalgam by hand, overfilling slightly. Carve back using the edge of the microscope slide to produce a flat surface (on the dental amalgam) that is level with that of the mould surface.

Allow the dental amalgam to set for 10 min. Carefully eject the test-piece from the mould by applying light finger-pressure to the surface of the test-piece that had been carved back (i.e. the “top surface”), while holding the mould in the other hand. Check visually that the “test surface” is defect-free everywhere, other than possibly at the margin. Use visual inspection without magnification. Carry out this inspection at an illuminance of at least 1 000 lx and at a distance not exceeding 250 mm. A person making the inspection shall have nominally normal visual acuity. Corrective (non-magnifying) untinted lenses may be worn. If a defect is detected, reject that test-piece and make a replacement.

To prevent any damage to the test surface during ejection, placing a thick soft pad (such as dental napkins) under the mould to “catch” the ejected test-piece is recommended.

After ejection, do not grind or polish the surfaces of the test-piece.

7.3 Measurement of the test-piece and storage prior to testing

7.3.1 Equipment

7.3.1.1 Micrometre screw gauge (with flat anvils that are at least 6 mm in diameter) or a similar measuring instrument, to measure the test-piece thickness to an accuracy of 0,01 mm.

NOTE Flat anvils with such a diameter are required to minimize the possibility of surface marking.

7.3.1.2 Air oven or incubator maintained at (37 ± 2) °C.

7.3.2 General

Measure the thickness of the test-piece to an accuracy of 0,01 mm. Take care not to mark the surfaces when the test-piece is placed between the anvils of the micrometre and they are closed onto the surfaces. If the thickness is outside the range (3,00 ± 0,01) mm, reject this test-piece and replace it with another.

After removing the test-piece from the micrometre, inspect the test surface to determine whether it has been marked during measurement. Carry out this inspection at an illuminance of at least 1 000 lx and at a distance not exceeding 250 mm. A person making the inspection shall have nominally normal visual acuity. Corrective (non-magnifying) untinted lenses may be worn. If marking is seen, reject and replace the test-piece.

Immediately after this inspection, place the test-piece in an air oven or incubator maintained at (37 ± 2) °C.

Make 10 test-pieces. Store these test-pieces for the specified time period.

NOTE The storage time is not specified in this document. The decision on this will be made when the test is adopted in the ISO standards on dental amalgam. For any other application, the user can select a time appropriate for the investigation being undertaken.

After the storage period, remove the test-pieces from the oven, taking care not to touch the test surface.

Place the test-pieces with the test surface upwards on a clean and flat surface. Leave to allow them to equilibrate with the ambient temperature.

8 Test procedure

8.1 Apparatus

8.1.1 Bearing ball, chrome steel, minimum Vickers' hardness 7,0 GPa, polished surface, 20 mm in diameter.

8.1.2 Substrate disc, dimensions: 10 mm in diameter and 5,00 mm thick. The Young's modulus of this disc is (10 ± 1) GPa. 30 % glass-filled polyamide rod[1)] is a suitable material. Lathe-cutting the disc from such a rod is recommended. If necessary, wet-grind the end faces under light pressure on coated abrasives (that comply with micro-grit size P1200 in accordance with ISO 6344-1) to remove any lathe turning marks.

8.1.3 Universal mechanical testing machine with compression testing facility. Load cell capacity 5 kN. Minimum data acquisition rate: 100 data points/s.

8.2 Mechanical testing

(20 ± 5) min after a test-piece has been removed from the oven (or incubator), place it centrally with its test surface downward on the glass-filled polyamide disc (which is placed centrally on the lower compression platen of the universal mechanical testing machine), i.e. the test surface is to be in contact with the filled polyamide disc. The steel loading ball is positioned centrally on the upper surface of the dental amalgam test-piece (see Figure 1).

The designs of mechanical testing machines are various. For this reason, the means by which the ball is accurately located and fixed to the upper platen (and centred on the test-piece) is not prescribed. Adopt an appropriate solution. However, the arrangement should have a high axial stiffness.

1) Polyamide – Nylon 6,6 – 30 % glass fibre reinforced — Rod. (PA 6,6 30 %GFR) is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.

Apply force to the test-piece using a cross-head speed of 0,2 mm/min, collecting the force *vs.* displacement values digitally at an acquisition rate of at least 100 data points/s. Record the force at which fracture occurs.

NOTE In the absence of explosive fracture, the continued presence of material under the ball maintains a finite force. In such a circumstance, there are several methods available to determine the fracture force: acoustic emission, or load-cell ringing, or a clear force drop, or a discontinuity in the force *vs.* displacement output in the case of a "silent" fracture.

It is possible that incomplete separation will exist in a failed test-piece. Under such a circumstance, use gentle finger-pressure to complete the separation (after the test-piece is removed from the testing machine).

Collect and piece together the test-piece fragments. Determine whether radial dissection has occurred by ascertaining, using visual inspection without magnification, whether bottom-initiated radial fracture has taken place. Carry out this inspection at an illuminance of at least 1 000 lx and at a distance not exceeding 250 mm. A person making the inspection shall have nominally normal visual acuity. Corrective (non-magnifying) untinted lenses may be worn. If it has not so fractured, the result is to be rejected.

Fracture of a test-piece can produce between two and five fragments. The result is still valid if more than two fragments are produced, provided that bottom-initiated radial fracture has occurred.

Load all 10 test-pieces to fracture.

For each test-piece, record the force (in newtons) at which fracture occurs and whether failure is valid or not.

9 Treatment of data and reporting of results

Report all test results. Include any result that has not been accepted with the reason for this rejection.

Calculate the mean value for the accepted results for the test-pieces. Report this.

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