TECHNICAL REPORT

ISO TR 11405

First edition
1994-12-15

Dental materials — Guidance on testing of adhesion to tooth structure

Produits dentaires — Lignes directrices pour l'essai d'adhésion à la structure de la dent

Reference number
ISO/TR 11405:1994(E)

Contents

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

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.

The main task of technical committees is to prepare International Standards, but in exceptional circumstances a technical committee may propose the publication of a Technical Report of one of the following types:

— type 1, when the required support cannot be obtained for the publication of an International Standard, despite repeated efforts;

— type 2, when the subject is still under technical development or where for any other reason there is the future but not immediate possibility of an agreement on an International Standard;

— type 3, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example).

Technical Reports of types 1 and 2 are subject to review within three years of publication, to decide whether they can be transformed into International Standards. Technical Reports of type 3 do not necessarily have to be reviewed until the data they provide are considered to be no longer valid or useful.

ISO/TR 11405, which is a Technical Report of type 2, was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 1, *Filling and restorative materials*.

This document is being issued in the type 2 Technical Report series of publications (according to subclause G.4.2.2 of part 1 of the ISO/IEC Directives, 1992) as a "prospective standard for provisional application" in the field of adhesion to tooth structure in dentistry because there is an urgent need for guidance on how standards in this field should be used to meet an identified need.

This document is not to be refarded as "International Standard". It is proposed for provisional application so that information and experience of its use in practice may be gathered. Comments on the content of this document should be sent to the ISO Central Secretariat.

A review of this type 2 Technical Report will be carried out not later than two years after its publication with the options of: extension for another two years; conversion into an International Standard; or withdrawal.

Annex A of this Technical Report is for information only.

Introduction

The increasing importance of adhesion in restorative dentistry has made it evident that information is needed on the relative performance of materials which are claimed to bond to tooth structure. In the absence of comparative clinical trials, much emphasis has been placed on laboratory assessment of bond strength. While bond strengths cannot predict clinical behaviour, they are valuable for screening.

Adhesive materials are used in many types of restorative and prophylactic work. Even if the stress on the bond in most circumstances can be defined as either tensile, shear or a combination of these, there are no specific laboratory or clinical tests which can be valid for all the various clinical applications of adhesive materials.

It is, therefore, the intention of this Technical Report to standardize as far as possible different procedures whereby the effect or quality of a bond between a dental material and the tooth structure may be substantiated. By gaining experience with a specific testing system, a correlation between laboratory and clinical performance of the materials should be sought. Data from such correlations may then form the basis for revision of the document and simplification of appropriate testing.

This page intentionally left blank

Dental materials — Guidance on testing of adhesion to tooth structure

1 Scope

This Technical Report specifies test methods for evaluation of the adhesive bond between dental materials and tooth structure, i.e. enamel and dentine. It describes two bond strength measurement tests, tensile and shear, a test for measurement of marginal gaps around fillings, a microleakage test, and gives guidance on clinical usage tests for such materials.

2 Normative references

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

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 3823-1:1986, *Dental rotary instruments — Part 1: Steel and carbide burs.*

DIN 69176:1985, *Körnungen aus Elektrokorund und Siliciumkarbid für Schleifmittel auf Unterlagen — Teil 1: Bezeichnung und Korngrössenverteilung.*

3 Definitions

For the purposes of this Technical Report, the following definitions apply. [1], [2]

3.1 adhere: Be in a state of adherence.

3.2 adherence: State in which two surfaces are held together by interfacial forces.

3.3 adherend: Body that is held, or is intended to be held, to another body by an adhesive.

3.4 adhesion: State in which two surface are held together by chemical or physical forces or both with the aid of an adhesive.

3.5 adhesive: Substance capable of holding materials together by adhesion.

3.6 bond strength: Force per unit area required to break a bonded assembly with failure occurring in or near the adhesive/adherend interface.

3.7 substrate: Material upon the surface of which an adhesive-containing substance is spread for any purpose, such as bonding or coating.

4 Requirements

This Technical Report contains no requirements for dental materials.

5 Sampling

The amount of test material shall be sufficient for all planned tests and have the same lot or batch number.

6 Test methods

This Technical Report describes various types of tests:

a) screening tests;

b) bond strength measurements;

c) gap measurement test for adhesion to dentine;

d) microleakage test;

e) clinical usage tests.

For some types, specific tests are described in detail. For other types, guidelines are given. It is not the intention to recommend testing each material by every test; some tests will not be appropriate. However, the quality and sophistication of a laboratory test cannot compensate for the fact that the final evidence of adhesive properties has to be a clinical usage test.

6.1 Screening tests

6.1.1 Introduction

Many screening tests may be necessary in the development of new adhesive materials or for production control. Such tests may be performed on bovine teeth.

However, the chemistry and especially the structure of bovine teeth are not identical to those of human teeth, and results from bovine teeth cannot replace those from human teeth. The results of such tests should not be used for advertising or promotion of the material.

6.1.2 Tooth substrate, storage and preparation

The condition, storage and preparation of teeth, either human or bovine, should be as described in 6.2 to 6.4.

6.1.3 Test methods

6.1.3.1 Type of test

Many types of screening tests are available, for example bond strength measurements, microleakage tests, marginal gap measurements. If bond strength measurements are used, the method adopted should be either

a) tensile (bond broken by a force perpendicular to the tooth surface); or

b) shear (bond broken by a force parallel to the tooth surface).

Methods which introduce "peel" stresses are not acceptable. Many current test methods fail to exert purely tensile or shear forces on the bond. A special problem is the alignment in the tensile test and the correct and reproducible loading in the shear test. It is recommended that similar tests for screening purposes to those described for bond strength measurement on human teeth (6.2.2 and 6.2.3) should be used.

All forces exerted on a bond can be resolved into shear and tensile components and, since there is no direct relation between the results obtained from the two measurements, it is preferable to measure both.

6.1.3.2 Condition of adhesive

Bonding materials may be used in a thin film or in bulk.

As far as possible, materials should be applied in a manner which duplicates their clinical use.

6.1.3.3 Storage of test specimens

Test specimens shall be prepared at (23 ± 2) °C and stored in water at (37 ± 2) °C prior to testing at (23 ± 2) °C. Storage in water for 24 h is normally sufficient to discriminate between those materials which cannot and those which can withstand a wet environment. Thermal cycling between 5 °C and 55 °C may be used as an accelerated ageing test. Longer periods of water storage may be necessary to show durability of bond.

The recommended procedure is as follows:

Test type 1: Short term test after 24 h in water at 37 °C.

Test type 2: Thermocycling test comprising 500 cycles in water between 5 °C and 55 °C, starting after 20 h to 24 h storage in water at 37 °C. The exposure to each bath should be at least 20 s, and the transfer time between baths 5 s to 10 s.

Test type 3: Long term test after six-month storage in water at 37 °C.

6.1.3.4 Strain rate for bond breakage

The standard rate of loading a bonded specimen is recommended to be (0,75 ± 0,30) mm/min cross-head speed, or more accurately (50 ± 2) N/min. (The stiffness in the various testing machines and bond assemblies varies widely and hence 50 N/min is more meaningful than 0,75 mm/min.)

6.1.3.5 Treatment of results

The bond strength values obtained by tensile or shear testing show large coefficients of variation, i.e. 20 % to 50 %. If the variation is above 50 %, a thorough inspection of the overall procedure is recommended. For screening purposes, six to ten specimens are re-

quired to give a meaningful average. Of greater importance at this stage may be inspection of fracture surfaces visually at × 10 magnification and to determine whether the fracture pattern is of an adhesive, cohesive or mixed nature.

6.2 Bond strength measurements

6.2.1 Introduction

The measurement of bond strength may be important when evaluating an adhesive material. However, the technique-sensitivity of such tests and the scatter of results due to variations in the substrate, adhesive and handling limit the importance and use of the results.

Adhesive materials are used for many different purposes in the mouth. The choice of test shall be considered according to the intended use of the material.

This Technical Report describes two types of tests, tensile and shear. In addition, several variations such as application in thin film and bulk, and short or long exposure time to a wet environment are described. A set of tests may be necessary properly to evaluate the bond strength of a material.

When bond strength is to be measured, the raw data are in units of force (newtons). It is desirable to convert this into stress units, i.e., force per unit area (megapascals). Hence, control of the surface for application of the adhesive material is paramount.

6.2.2 Tooth substrate and storage

6.2.2.1 Substrate

Human permanent teeth are required for the measurement of clinically relevant bond strength. When measuring bond strength to dentine, this Technical Report recommends that the superficial dentine, i.e. as close to enamel as possible, at the buccal aspect of the third permanent molar from patients in the age group 18 to 25 years be used to reduce variations. However, if this restriction is difficult to fulfil, the other molars or premolars may be used, provided the teeth used are listed in the report of the investigation.

6.2.2.2 Time after extraction

There is increasing evidence that changes in dentine occur after extraction which may influence bond strength measurements. The effect may vary with different types of bonding materials. Ideally, bond strengths should be measured immediately post-extraction, but clearly this is not generally feasible. It appears that most changes occur in the initial days or weeks after extraction, and therefore teeth one month, but not more than six months, after extraction should be used. Do not use root-filled teeth. Teeth which have been extracted for longer than six months may undergo degenerative changes in dentinal protein.

6.2.2.3 Condition of teeth

The teeth used for bond strength measurement should be caries-free and preferably unrestored. However, small and superficial restorations not in the adhesion test area may be present. There is some evidence to suggest that different areas of the dentition may give different results with bonding to dentine and enamel. Bearing in mind the number of measurements required, it is not possible to control variables such as age of the donating patient, cultural and dietary history, state of health, or to standardize the composition and structure of the teeth.

6.2.2.4 Storage of teeth

Prior to storage, the teeth should be thoroughly washed in running water and all blood and adherent tissue removed, preferably by the clinician. Teeth should be placed immediately after extraction in distilled water (grade 3, ISO 3696) or in a 0,5 % chloramine bacteriostatic/bactericidal solution for a maximum of 1 week and thereafter stored in distilled water in a refrigerator at a nominal 4 °C. To minimize deterioration, the storage medium should be replaced periodically. It is essential that no other chemical agents be used. Such agents may be absorbed by, and alter, tooth substance.

6.2.2.5 Tooth surface preparation

A standard, reproducible, flat surface is required. Tooth surfaces shall be kept wet at all times. Exposure of a tooth surface to the air for more than 15 minutes may cause irreversible changes in bonding character. Dentine is especially sensitive to dehydration. The use of high-speed handpieces to prepare tooth surfaces for bonding studies is to be avoided. A standard surface is recommended such as that produced against a silicon carbide abrasive paper with a mean grit size of approximately 18 µm (Grade 1000, DIN 69176) under running water. To control the planing and the angle of the surface during surface preparation, the tooth should be mounted in a holder by means of a dental die stone or a cold-curing resin. The absorption of resin and the heat of polymerization may adversely affect the tooth. Avoid smearing of embedding material on the tooth surface during final preparation against the 1000 grade silicon carbide, e.g. by

mounting the tooth above the surface of the embedding material.

6.2.2.6 Re-use of teeth

Fresh teeth should preferably be used. If it is not possible to use a fresh tooth for every measurement, it is essential that "control" measurements using a standard material be undertaken frequently to check for irreversible changes caused by previous treatments.

6.2.3 Tensile bond strength

6.2.3.1 Apparatus

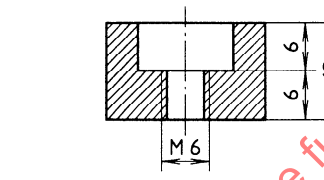
The various parts of the tensile test specimen are shown in figure 1: a) is a holder for mounting of teeth (tooth cup); b) is the counterpart either as a holder for the adhesive materials applied in bulk, i.e. the material cup or as the second substrate, i.e. a small rod, when applying the adhesive in thin film. For light-curing systems, the material cup may be made totally of a transparent material, or alternatively it may be split into two parts with a smaller cup in a transparent resin and the connection to c) in metal, where c) is the coupling to the alignment rod.

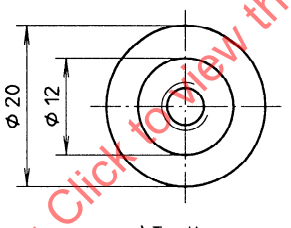
Figure 2 shows the bonding alignment apparatus with alignment rods, a), and the bonding alignment block, b).

Figure 3 shows the measurement alignment block, b), and the necessary alignment rods, a).

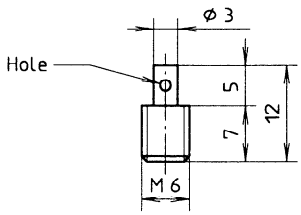
Figure 4 shows the polishing block.

Dimensions in millimetres





a) Tooth cup



b) Material cup/rod

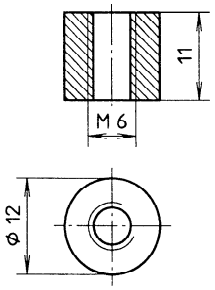


Figure 1 — Tensile specimen

Dimensions in millimetres

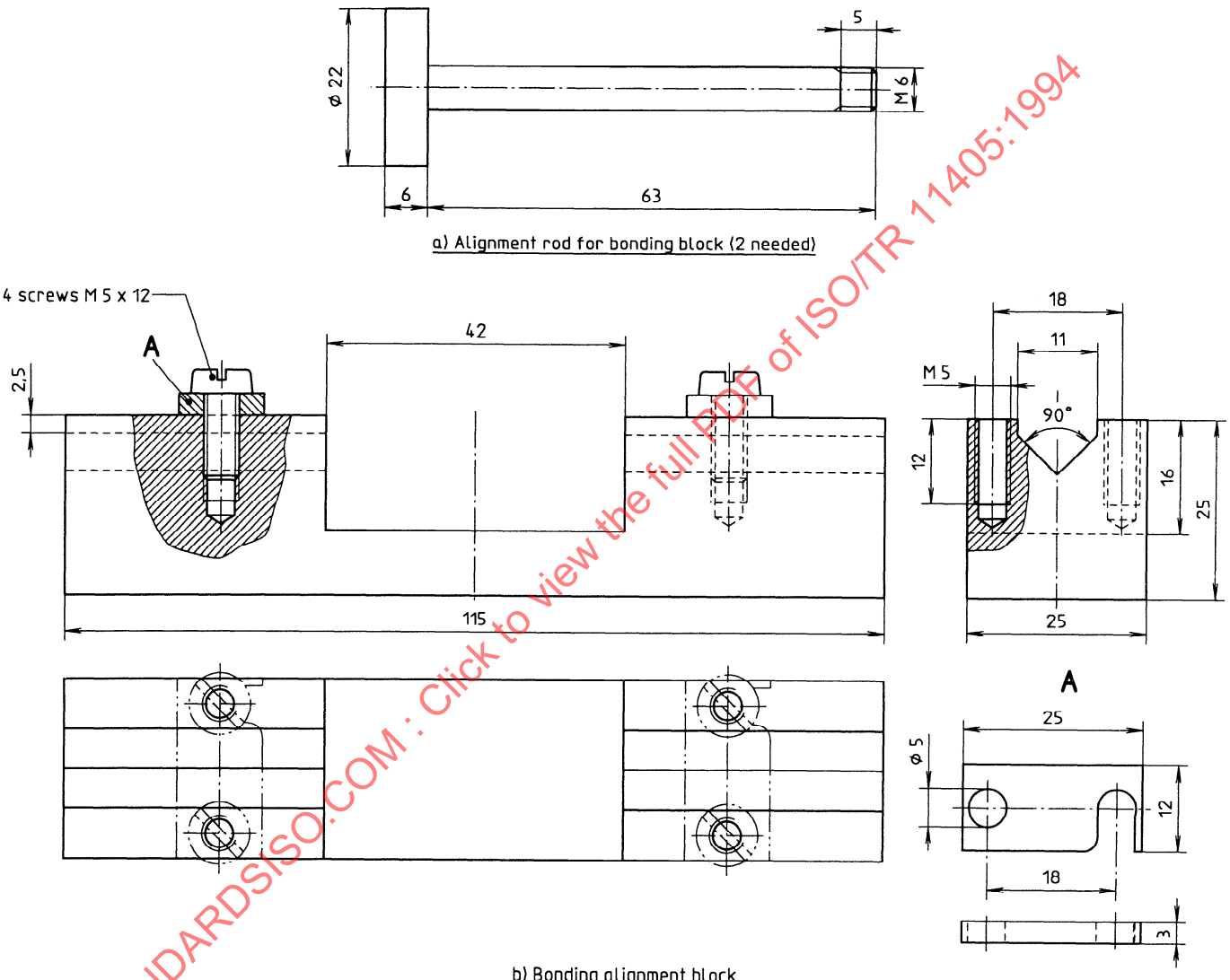
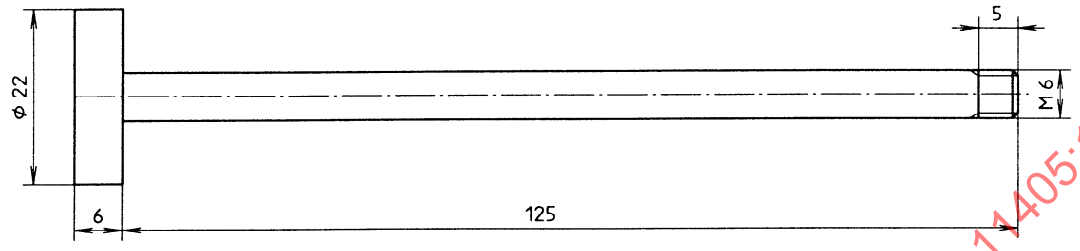
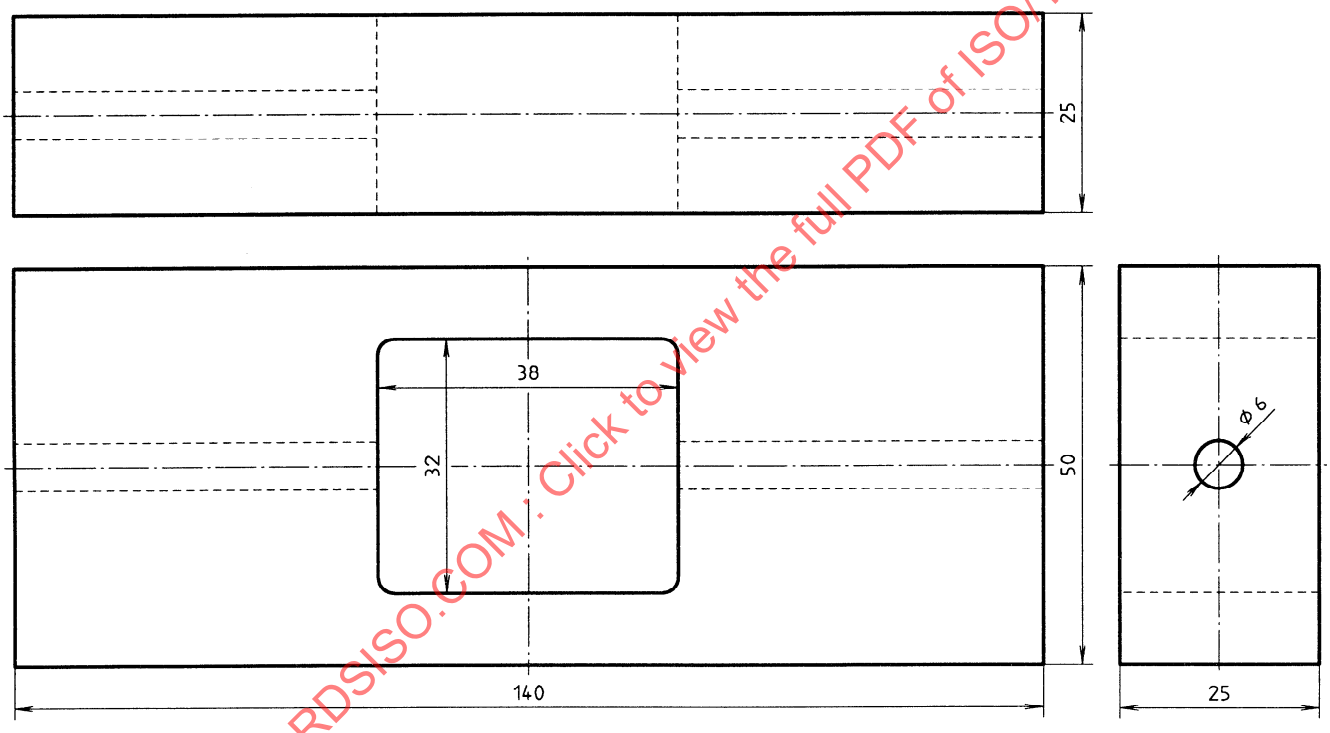


Figure 2 — Bonding alignment apparatus

Dimensions in millimetres



a) Alignment rod for measurement block (2 needed)



b) Measurement alignment block

Figure 3 — Measurement alignment

Dimensions in millimetres

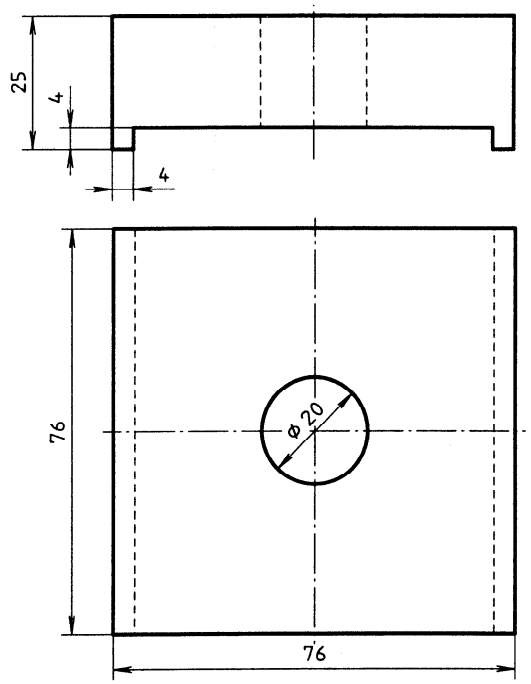


Figure 4 — Polishing block

6.2.3.2 Mounting of teeth

Make sure that the teeth, when mounted in the holder, can be mounted in the tensile test alignment block together with the adhesive material. (An alternative measurement alignment block for longer specimens is shown in figure 5.)

Make sure that the tooth has form, undercuts, holes or retentive pins that will secure retention in the holder. Mount the tooth with a cold-curing, slow-setting resin or dental die stone. The part of the tooth of interest for planing, polishing and bonding shall be positioned above the edge of the tooth cup so that polishing can be performed without contaminating the tooth surface with metal or mounting material. Place the mounted tooth in water at (23 ± 2) °C as soon as possible.

6.2.3.3 Polishing of teeth

Plane and polish the exposed surface of the tooth by means of the polishing block (figure 4) on wet carborundum paper No. 1000, fixed to a hard, plane surface. Control movements of the polishing block to avoid contamination with metallic particles. Polish until the surface is even and smooth when inspected by low magnification (× 2). Teeth with perforations to the pulp chamber shall be discarded.

6.2.3.4 Preparation of test specimen

The tooth surface prepared for application of adhesive material shall be preconditioned according to the manufacturer's instructions.

If no instructions are given, rinse with running water for 10 s and remove visible water on the surface with filter paper or by a light or short stream of oil-free compressed air immediately before application of the adhesive material. Mix and apply the adhesive material according to the instructions given by the manufacturer in a room at (23 ± 2) °C and (50 ± 5) % relative humidity.

6.2.3.4.1 Thin film

A preformed adherend is fixed to the alignment rods by the same coupling as the material cup.

Mount the bonding alignment block vertically and fix the tooth cup with alignment rod to the lower part of the block.

Apply a thin layer of the adhesive material on the tooth surface and lower the adherend to contact the adhesive material. Place a load of 10 N on top of the upper alignment rod. The upper rod is fixed to the block sufficiently to prevent distortion, but to allow sliding of the rod in the V-shaped groove. After 10 s, fix the upper rod completely to the alignment block.

The total procedure from application of the material to the fixation of the upper rod shall be performed within the working time given by the manufacturer.

6.2.3.4.2 Bulk

When using the material cups for multiple use, coat the inner part of the material cup and the fixation pin with a thin layer of silicone grease or a PTFE dry film lubricant. Avoid coating the edge of the holder.

Apply a thin layer of the adhesive material to the tooth surface. Fill the material cup to a slight excess and place it firmly in the correct position on the tooth. Fix the alignment rod immediately to the block. Ensure that the material cup maintains contact with the tooth surface during fixation. The fixation of the alignment rod shall be finished within the working time given by the manufacturer of the adhesive material.

6.2.3.4.3 Prestorage

Immediately after fixation, transfer the entire bonding alignment block to a water-saturated chamber at (37 ± 2) °C. Remove the block after 1 h storage. Carefully remove the surplus material around the

Dimensions in millimetres

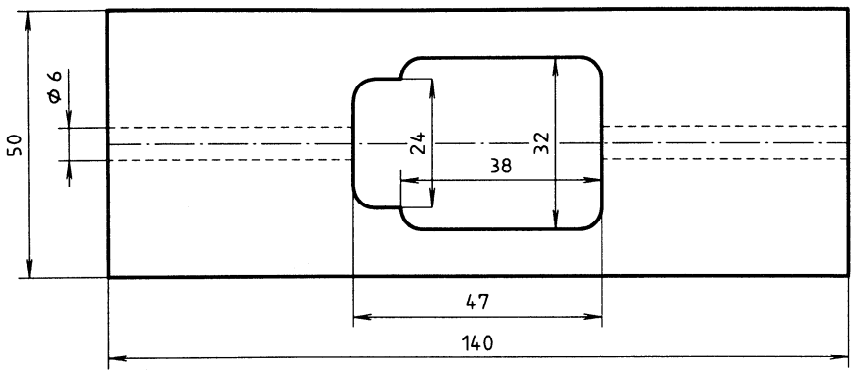


Figure 5 — Alternative measurement alignment block

edge of the material cup with a scalpel while the specimen is still fixed to the block. Unscrew the specimen carefully from the rods without exerting rotational or bending forces on the bond. Transfer the specimen to a water bath at (37 ± 2) °C for further storage.

NOTE 1 See 6.1.3.3 and 6.2.5 for information regarding alternative storage conditions.

6.2.3.5 Tensile testing

The test shall be performed at (23 ± 2) °C and (50 ± 5) % relative humidity. Mount the tensile test specimen in the measurement alignment block.

Do not apply any bending or rotational forces on the adhesive material during mounting.

Mount the entire assembly in a testing machine. The connection between the material cup alignment rod and the machine should be by a flexible connection, e.g. a chain or string.

Apply the tensile load as described in 6.1.3.4.

6.2.4 Shear bond strength

6.2.4.1 Apparatus

The various parts of the shear test specimen and testing rig are shown in figures 6 and 7.

Figure 6 is a schematic drawing of the specimen mounted in the rig ready for loading.

Figure 7 a) is the fixation part of the rig with holes for fixation of the specimen and guidance slots (keyways) for the loading plate. Figure 7 b) shows the loading plate with three holes and a flat (blunt) 1 mm broad shearing edge at a 90° angle to the direction of the load or the back of the loading plate. The plate has three holes with different diameters, 3 mm, 5 mm and 10 mm, for various specimen sizes.

All contacting metal surfaces shall be plane and polished.

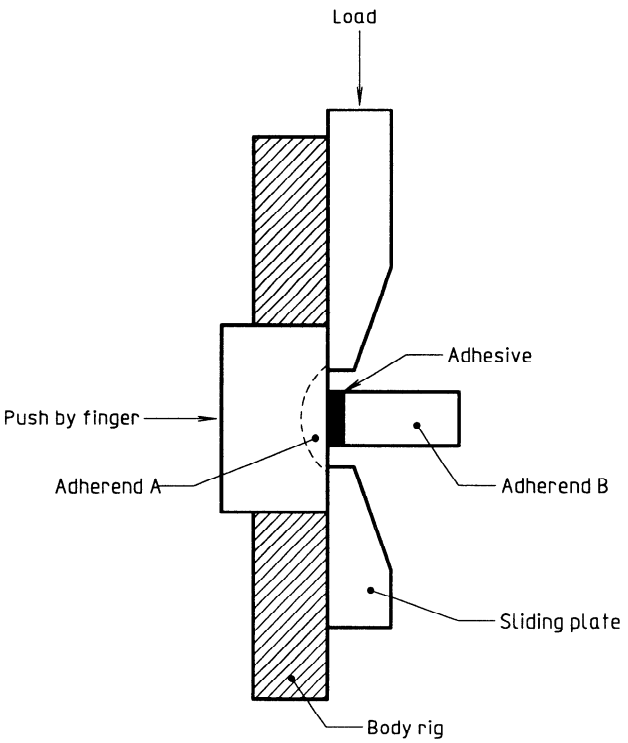


Figure 6 — Schematic diagram of testing jig for shear bond strength

Dimensions in millimetres

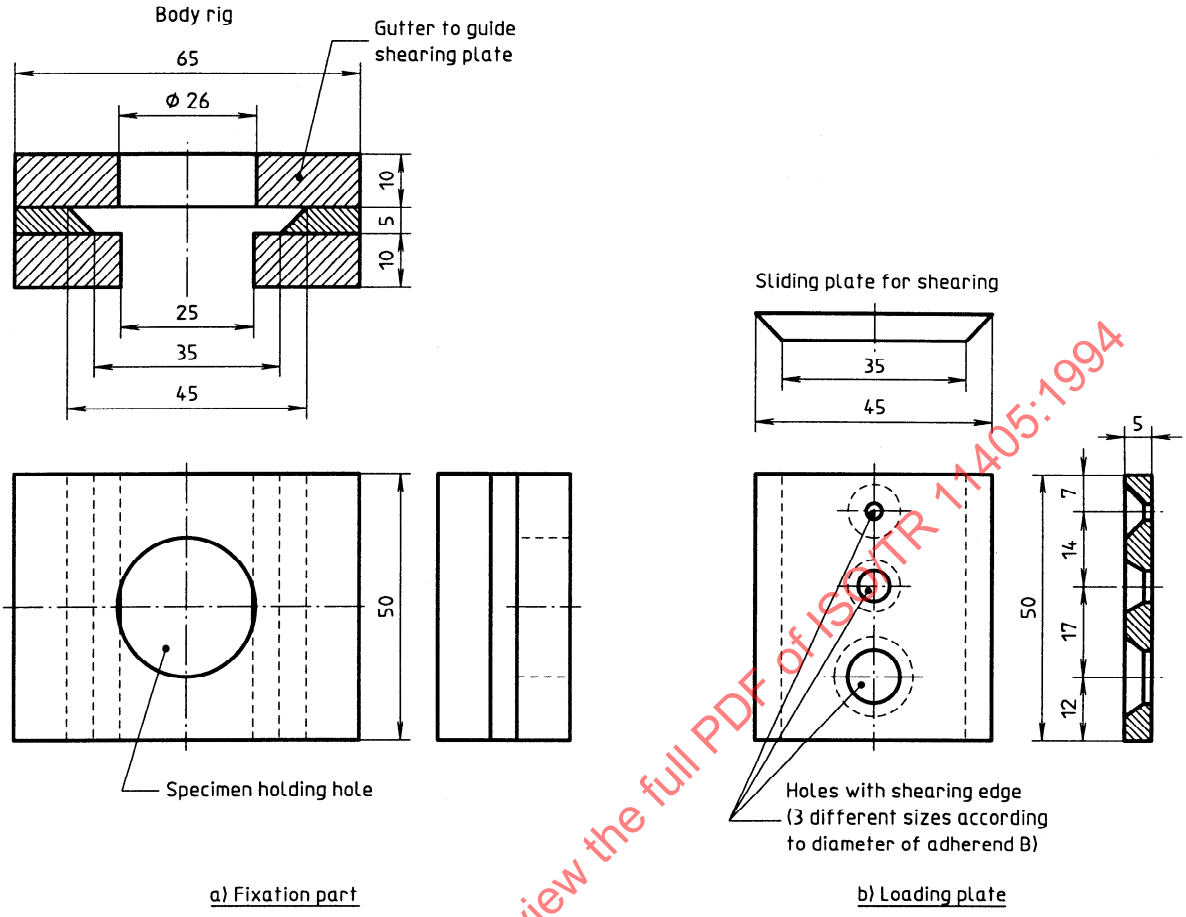


Figure 7 — Constructional figure of rig for bond strength measuring by shear

6.2.4.2 Mounting of teeth

See 6.2.3.2.

6.2.4.3 Polishing of teeth

See 6.2.3.3.

By the shear bond strength test, the distance between the prepared tooth surface and the top surface of the mounting resin shall be a maximum of 1 mm.

6.2.4.4 Preparation of test specimen

See 6.2.3.4 for general information.

6.2.4.4.1 Thin film

An adhesive used as a thin film should be confined to the area of application of the subsequent adherend. This can be achieved by firmly clamping a split non-stick polytetrafluoroethylene mould (figure 8) such as Teflon[1)] to the tooth specimen using a suitable device (e.g., figure 9). Alternatively, adhesive tape with a 3 mm diameter hole may be applied to the tooth surface. However, ensure that the tape is not affected by the adhesive. Following the manufacturer's instructions, apply the adhesive to the tooth surface in the hole. If adhesive tape is used, position the split mould over the hole in the adhesive tape and clamp into place.

Fill the split mould with the adherend material using a method involving minimal risk of entrapping air along the contact area of the adhesive. After curing, remove the specimen from the apparatus by releasing fixation screw E (figure 9) without applying any force on the specimen, i.e., shear, bend or rotation.

1) Teflon is a tradename.

This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

Dimensions in millimetres

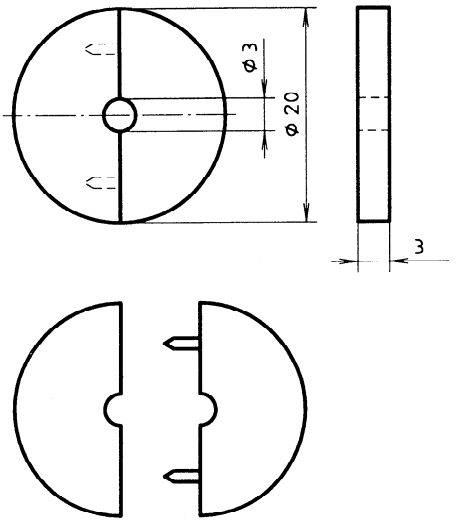


Figure 8 — Split mould

6.2.4.4.2 Pre-formed adherend rod

Use a PVC tape with a circular hole as above.

Apply a thin layer of the adhesive material and place the adherend rod in the hole. Make sure that the long axis of the rod remains as close as possible to a 90° angle to the tooth surface during the necessary curing time.

Remove the tape before loading.

6.2.4.4.3 Bulk

Use a split mould as described in 6.2.4.4.1.

6.2.4.5 Shear loading

Position the specimen in the loading rig. Apply a slight finger pressure on the back (bottom) of the specimen to prevent displacement.

Mount the rig in a testing machine and apply the load as described in 6.1.3.4.

6.2.5 Storage of specimens

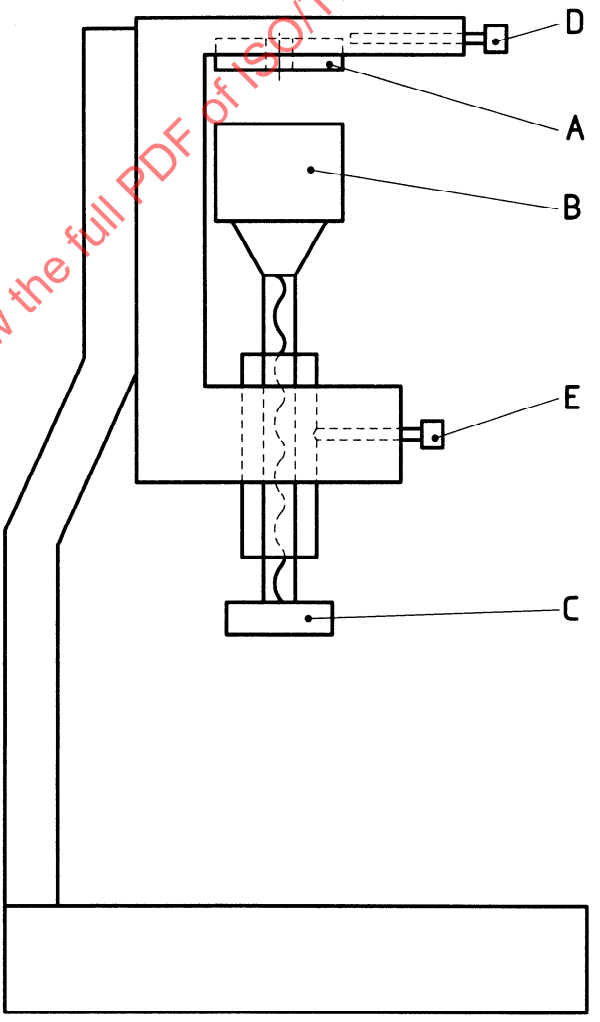
See also 6.1.3.3.

6.2.5.1 Short-term test

After removal from the bonding alignment apparatus, store the specimen in distilled water at (37 ± 2) °C for 24 h.

6.2.5.2 Thermocycling test

Store for 20 h to 24 h at 37 °C. The specimens shall thereafter be placed in a thermocycling apparatus with one bath of distilled water at (5 ± 2) °C and another at (55 ± 2) °C. The specimens shall be cycled with 500 exposures to each bath, i.e. at least 20 s in each bath. The transfer time from one bath to the other should be within 5 s to 10 s.



Key
A: Split mould
B: Tooth cup
C: Fixation screw
D: Fixation screw
E: Fixation screw

Figure 9 — Specimen preparation device

6.2.5.3 Long-term test

Store for six months in distilled water at (37 ± 2) °C.

6.2.6 Strain rate

See 6.1.3.4.

6.2.7 Treatment of results

For in-house screening tests, see 6.1.3.5.

Bond strength results for use in advertising or certification purposes for a material should be based on sound statistical methods and a sufficient number of specimens. If the data are normally distributed, a mean, standard deviation and coefficient of variation can be calculated. Means can be compared by analysis of variance (ANOVA).

However, very often results from adhesion testing are not normally distributed. Therefore, the use of probability of failure, calculated from the Weibull distribution function, provides a suitable means of comparing many materials. The stress to give 10 % failure ($P_{f\,10}$) and that to give 90 % failure ($P_{f\,90}$) are convenient ways of characterizing the strength of a bond.

A minimum of 15 specimens is recommended.

6.3 Gap measurement test for adhesion to dentin

6.3.1 Introduction

Another approach to test the efficacy of an adhesive material with an intended use as a dentine bonding agent in the cavity filling technique is the gap measurement test [3]. The test is technique-sensitive and requires good training in handling and application of all the materials used in the procedure. The test should be performed at (23 ± 2) °C to limit influences from thermal variables. The effect of adhesives for bonding of resin-based filling materials to dentine in prevention of gaps between the filling and the cavity wall is largely dependent upon the physical properties of the composite. Quality testing of dentin adhesives needs, therefore, to be performed in combination with a composite recommended by the manufacturer.

6.3.2 Tooth substrate and storage

6.3.2.1 Substrate

See 6.2.2.1.

6.3.2.2 Time after extraction

See 6.2.2.2.

6.3.2.3 Condition of teeth

See 6.2.2.3.

6.3.2.4 Storage of teeth

See 6.2.2.4.

6.3.3 Cavity preparation

Plane the buccal surface of the tooth on wet silicon carbide paper (for grade, see 6.2.2.5), fixed to a hard, plane surface, to expose a dentine area of at least 4 mm diameter. Prepare a dentine cavity (3,0 ± 0,1) mm diameter, approximately 1,5 mm deep, with a cavosurface angle of 90°. Use a carbide bur with a straight fissure head without cross-cuts (ISO 3823-1:1986, subclause 6.4) at 4 000 r/min and extensive water cooling. The specimens shall be assessed microscopically at × 5 magnification to ensure that the entire cavosurface margin is surrounded by dentine.

6.3.4 Filling procedure

The teeth should be conditioned in distilled water at (23 ± 2) °C for a minimum of 12 h prior to the filling procedure. Follow the manufacturer's instructions closely, including the choice of other necessary materials to complete the total filling procedure. Syringing materials of high viscosity into the cavity reduces the risk of voids along the cavity walls.

6.3.5 Storage of specimen

After completion of the restoration, store the specimen in water (grade 3 in ISO 3696) at (23 ± 2) °C. The test can be used to control the efficacy of the adhesive at various times after completion of the restoration. To test the initial effect of an adhesive to prevent gaps due to contraction stresses in the restorative material, specimens should be inspected after 10 min when the main part of contraction has occurred and the maximum gap will be visible.

6.3.6 Gap measurement

Remove approximately 0,1 mm of the surface of the filling and dentine by gentle, wet grinding on silicon carbide paper (for grade, see 6.2.2.5). Proceed with polishing on linen cloth with an aqueous slurry of 0,3 µm abrasive particles. The surface of the speci-

men should be kept continuously wet and at a temperature of (23 ± 2) °C.

Before measuring the gaps between the filling and the cavity wall, rinse the specimen surface thoroughly with a water spray to remove debris in the gaps. Measure at (23 ± 2) °C the maximum width of the largest gap observed along the cavity wall in a measuring microscope or in a universal microscope with a measuring eyepiece. The measurement shall be performed without dehydration of the tooth/filling surface, e.g., in a water-saturated chamber. A minimum of 10 cavities should be examined.

6.4 Microleakage test

6.4.1 Introduction

A microleakage test is another way to test the efficacy of a material or a combination of materials to establish bonds both to enamel and dentine. A variety of methods has been described with some variations in results. Standardization of such methods is therefore necessary in order to get comparable results from different laboratories. In this respect it seems important to standardize quality of teeth, type of cavity and the quantification of leakage. The type of tracer substance does not seem to be of major importance apart from radioactive tracers which will show diffusion of water through closed interfaces in addition to leakage along patent interfaces.

6.4.2 Tooth substrate and storage

See 6.2.2.1 to 6.2.2.4.

6.4.3 Cavity preparation

Several cavity types are of interest when studying leakage. When testing the quality of a particular material or combination of materials to prevent leakage, a standard cavity 3 mm in diameter, and with a depth of at least 1 mm into the dentine in the mid-part of the buccal surface of a third molar should be used.

Start cavity preparation in enamel with a high-speed handpiece using a small cylindrical diamond. Finish cavity walls to an exact diameter with a carbide bur with a straight fissure head without cross-cut (ISO 3823-1:1986, subclause 6.4) at 4 000 r/min and water cooling. If a cavity solely surrounded by dentine is of interest, follow the procedure described in 6.3.3. A minimum of 10 cavities should be examined.

6.4.4 Filling procedure

Teeth should be conditioned in distilled water at (23 ± 2) °C before starting the filling procedure. Follow the manufacturer's instructions closely. Syringing high viscosity materials into the cavity reduces the number of voids along the cavity walls.

6.4.5 Storage of specimen

Immediately after completion of the filling procedure, immerse the specimen in the tracer solution and store at (23 ± 2) °C for 24 h.

If the effect of thermocycling is part of the test, start the thermocycling procedure according to 6.2.5.2 after 1 h storage at (23 ± 2) °C with the tooth immersed in tracer solution.

6.4.6 Measurement of microleakage

Cut the tooth longitudinally twice to either side of the midline of the cavity with a slow speed diamond saw under water cooling. Score all four surfaces, if possible, for quantification of microleakage. Inspect under a microscope at × 10 magnification for penetration of tracer along the cavity walls. Use the following quantification:

no penetration = 0;

penetration into the enamel part of the cavity wall = 1;

penetration into the dentine part of the cavity wall but not including the cavity pulpal floor = 2;

penetration including the cavity pulpal floor = 3.

If using a dentine cavity:

no penetration = 0;

penetration into the dentine/composite interface but not including the cavity pulpal floor = 1;

penetration including the cavity pulpal floor = 2.

6.4.7 Treatment of results

Count the number of observations and use non-parametric statistics when comparing products or procedures.

6.5 Clinical usage tests

6.5.1 Introduction

A clinical usage test is so far the only real basis for judgement of clinical efficacy and lifetime of an adhesive material. Such tests should be designed and performed according to accepted clinical procedures and the intended use of the material.

6.5.2 Guidelines

Clinical usage tests should follow International Standards published by ISO or CEN [4], or appropriate protocols such as those that may be available from the International Dental Federation (FDI) or American Dental Association (ADA) [5], or as described below.

6.5.3 Restorations

The type of restoration used should be decided by the manufacturer according to the intended use of the material. If cavities are prepared, a cavity type with limited variations in form and size is preferred.

6.5.4 Observation time

A total observation time of three years is recommended.

Restorations must be observed at baseline and at least once a year for the following three years.

6.5.5 Sample size

Sample size will depend upon recall rate. In general, the number of restorations at the end of three years should not be less than 25. The reasons for loss of patients and/or failure of restorations shall be identified.

6.5.6 Clinical procedures

A detailed description of the clinical procedures comprising design, instruments used, pretreatment of surfaces, mixing and placement of material, finishing, etc. shall be given.

6.5.7 Evaluation

The evaluation should include both direct and, if possible, indirect clinical methods.

The direct clinical methods should be based on the USPHS system [6], [7] or on the "modified Ryge-criteria" [8].

Indirect methods should be used to quantify gaps, loss of material, etc.

6.5.8 Treatment of results

Consideration should be given to the use of a life-table analysis in order to take into account the loss of patients or the loss of restorations due to unrelated causes. [9], [10].