BRITISH STANDARD

BS 5136:1981

Incorporating
Amendment No. 1

Specification for

Toothpastes

UDC 665.583.44

Cooperating organizations

The Dental Standards Committee, under whose direction this British Standard was prepared, consists of representatives from the following:

Association of Dental Hospitals of the United Kingdom
British Dental Association*
British Dental Trade Association
British Society for the Study of Prosthetic Dentistry
Dental Materials Panel
Department of Health and Social Security*
Department of Industry (Laboratory of the Government Chemist)*
Department of Industry (National Engineering Laboratory)*
Department of Industry (National Physical Laboratory)
Institute of Dental Surgery
Ministry of Defence*
Royal Society of Medicine
Scottish Health Services

The organizations marked with an asterisk in the above list, together with the following, were directly represented on the Technical Committee entrusted with the preparation of this British Standard:

British Paedodontic Society
Cosmetic, Toiletry and Perfumery Association Limited
London Chamber of Commerce Incorporated
London Dental Study Club

This British Standard, having been prepared under the direction of the Dental Standards Committee, was published under the authority of the Executive Board and comes into effect on 30 January 1981

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3781	October 1981	Indicated by a sideline in the margin

Contents

Foreword

This British Standard, originally published in 1974, has been revised under the direction of the Dental Standards Committee primarily to incorporate an alternative method of abrasivity measurement, the surface profile method, which does not require access to a means of irradiation. The radio-tracer method remains as the primary method of abrasivity measurement and is used as the reference method in any cases of dispute or if the results achieved by the surface profile method fall between the reduced limits for that method and the normal limits, as specified for the radio-tracer method.

This British Standard is primarily concerned with excessive abrasion of toothpastes on the dental tissues, since this could conceivably represent a harmful property which cannot easily be recognized by the user. Relatively serious wear of exposed dentine can occur in practice owing to the considerably lower wear resistance of dentine as compared with enamel. It should be noted, however, that other factors, such as poor brushing technique or too acid a diet, play a more active role orally than the abrasive in damaging the teeth and their associated tissues.

The primary test method for abrasivity, which was developed from basic research undertaken at the National Engineering Laboratory, involves a radio-tracer technique of wear resistance in which extracted human teeth are irradiated, subjected to simulated brushing, and the abrasivity value calculated on the basis of the measurement of radio-activity transferred to the toothpaste. The surface profile method employs an identical brushing programme, but relies for its measurement of abrasivity on a highly accurate measurement of the depth of the abraded groove. Both methods produce results expressed as a ratio of the abrasivity of the test paste to that of a standard reference paste, in order to minimize the effects of the variability of tooth substance between one tooth and another. Alternative substrates to human dentine and enamel have been examined, but have been found unsuitable.

The limits for abrasivity given in this standard are based on wide experience of toothpastes currently available for general use. These limits are given as pass or fail requirements. Numerical values derived from abrasivity tests for toothpastes complying with the requirements of this standard are not intended to be treated as an index of the abrasivity of different toothpastes, as the clinical significance of such differences has not fully been established. A testing exercise was undertaken by the major United Kingdom dentifrice manufacturers in order to provide the data used by the responsible committee when deciding whether to include the surface profile method in this standard.

The designations "RDA (BSI)" and "REA (BSI)" are now generally being used by researchers in the field of toothpastes to indicate radio-active dentine abrasivity and radio-active enamel abrasivity respectively. The values are expressed as the ratio of the abrasivity of the test paste to that of the reference paste, and the suffix (BSI) is used to differentiate results from those obtained using the American (ADA) method.

The inclusion of a requirement and test method for lead content should not necessarily be taken as an indication that the lead content of toothpastes constitutes a significant hazard. Additionally, other trace elements such as cadmium, arsenic and antimomy have deliberately been omitted from this standard because their significance as a potential hazard is even lower than that of lead.

Attention is drawn to the EEC Cosmetics Directive, 76/768/EEC, adopted as United Kingdom Statutory Instrument 1978 No. 1354 "The Cosmetics Products Regulations 1978", which lists prohibited substances in Schedule 1 and provides controls concerning marking of containers. In addition, the Weights and Measures (Packaged Goods) Regulations provide controls for the declaration of the volume of the contents.

Summary of pages

This document comprises a front cover, an inside front cover, pages i to iv, pages 1 to 16, an inside back cover and a back cover.

This standard has been updated (see copyright date) and may have had amendments incorporated. This will be indicated in the amendment table on the inside front cover.

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1 Scope

This British Standard specifies requirements and test methods for the abrasivity, lead content and general properties of toothpastes, for use with a brush in the manual cleaning of natural teeth.

2 References

The titles of the publications referred to in this standard are listed on the inside back cover.

3 Requirements

3.1 Composition. The toothpaste shall not contain sucrose or other readily fermentable carbohydrates.

3.2 Toxicity. The toothpaste shall not contain ingredients in sufficient concentration to cause a toxic or irritant reaction when used in the oral cavity, nor shall it be otherwise harmful in normal use.

3.3 Lead content. The lead content of the toothpaste, when tested in accordance with **5.2**, shall not exceed 5 mg/kg (5 p.p.m.).

3.4 pH value. The pH value of the toothpaste, determined at a temperature of 23 ± 1 °C in accordance with **5.3**, shall be neither below 4.5 nor above 10.5.

3.5 Abrasivity

3.5.1 *Radio-tracer method*. When tested in accordance with **5.4**, the abrasivity of the toothpaste, calculated in accordance with the statistical treatment given in Appendix C, shall not exceed the following limits:

for dentine: 2 × the abrasivity of the standard reference paste;

for enamel: 4 × the abrasivity of the standard reference paste.

The standard reference paste shall be formulated as detailed in **5.4.1**.

3.5.2 *Surface profile method*. When tested in accordance with Appendix E, the abrasivity of the toothpaste, calculated in accordance with the statistical treatment given in Appendix C, shall not exceed the following limits:

for dentine: 2 × the abrasivity of the standard reference paste;

for enamel: 4 × the abrasivity of the standard reference paste.

The standard reference paste shall be formulated as detailed in **5.4.1**.

In any cases of dispute, or if the results exceed the following more stringent limits for the surface profile method, the abrasivity of the toothpaste shall be determined using the radio-tracer method:

for dentine: 1.5 × the abrasivity of the standard reference paste;

for enamel: 3 × the abrasivity of the standard reference paste.

If the results obtained by the two methods are different, those obtained using the radio-tracer method shall be accepted.

3.6 Consistency. The toothpaste shall be free of lumps or particles which are palpable in the mouth as separate or discrete particles.

3.7 Extrusion from tube. The bulk of the toothpaste shall extrude from the container in a continuous mass at a temperature of 10 °C, without the application of excessive force.

3.8 Stability. The toothpaste shall not segregate, ferment or deteriorate during normal conditions of storage and use.

3.9 Container. The toothpaste shall be packed in a container that shall not corrode, deteriorate, or cause contamination of the toothpaste during normal conditions of storage and use.

4 Sampling

A representative sample shall be taken from each batch or consignment.

5 Test methods

5.1 General. Water used in the test methods in this standard shall comply with the requirements of BS 3978. When the term “test paste” is used, this refers to the toothpaste being tested for compliance with this British Standard. The term “standard reference paste” refers to the paste prepared in accordance with **5.4.1** and Appendix A.

5.2 Determination of lead content

5.2.1 *Principle*. Lead in toothpaste is extracted with acid after low temperature ashing. The ammonium pyrrolidine dithiocarbamate (APDC) complex is formed, extracted into 4-methylpentan-2-one and the lead is determined by atomic absorption spectroscopy.

5.2.2 *Precautions*. All apparatus and reagents shall be lead free. Contamination, particularly from atmospheric dust, shall be prevented. To avoid volatilization of lead, it is essential that ashing be carried out at a low temperature; 450 °C is recommended, 500 °C maximum.

5.2.3 *Apparatus*. The following apparatus is required.

5.2.3.1 *Borosilicate glass beakers*, 100 ml capacity, or silica evaporating dishes.

5.2.3.2 *Conical flasks*, 100 ml capacity.

5.2.3.3 *Separating funnels*, 100 ml capacity.

5.2.3.4 *Muffle furnace*

5.2.3.5 *Atomic absorption spectrophotometer and lead hollow cathode lamp*

5.2.3.6 *Pipette*, Pasteur type.

5.2.3.7 *Pipette*, 10 ml.

5.2.4 *Reagents*. The following reagents shall be of analytical grade.

5.2.4.1 *Acetone*

5.2.4.2 *Ethanol*

5.2.4.3 *4-methylpentan-2-one* (*iso*butyl methyl ketone).

5.2.4.4 *Nitric acid*, concentrated.

5.2.4.5 *Nitric acid*, 5N.

5.2.4.6 *Ammonia solution*, 5N.

5.2.4.7 *APDC* 1 % *solution*. Wash 1.5 g of ammonium pyrrolidine dithiocarbamate (APDC) with 20 ml of acetone in a P4 sintered glass crucible. Dry, weigh 1 g and dilute to 100 ml with water.

5.2.4.8 *Standard lead solution* 1 000 mg/kg (1 000 p.p.m.) of lead.

5.2.5 ***Procedure***

5.2.5.1 *Destruction of organic matter*. Accurately weigh about 2 g of paste into a 100 ml beaker. Slurry with 10 ml to 15 ml of ethanol and evaporate to dryness on a water bath. Place in the muffle furnace at 100 °C and slowly raise the temperature by 50 °C steps to 450 °C to avoid ignition. Complete the ashing at 450 °C. If the ash is dark coloured, wet it with a few drops of the concentrated nitric acid and reheat in the furnace at 450 °C for between 30 min and 1 h.

5.2.5.2 *Acid extraction of the ash*. Add 5 ml of water and 10 ml of 5N nitric acid to the cooled ash and boil gently for 5 min. Filter the solution into a 100 ml conical flask, rinsing with two 5 ml portions of water and retaining as much as possible of any insoluble matter in the beaker or dish. Add 10 ml of 5N nitric acid to the beaker and boil gently for 5 min. Filter into the same conical flask, rinsing with two 5 ml portions of water.

5.2.5.3 *Determination of lead.* Adjust the pH value of the acid solution to between 3 and 4, using the ammonia solution, transfer to a separating funnel and cool to room temperature.

NOTE The effect of the pH value on extraction of lead is shown in Table 1.

Table 1 — Effect of pH value on extraction of lead

pH	Peak height (mm)	
	0.5 µg/ml	1.0 µg/ml
1	55	100
2	55	122
3	72	142
4	68	136
5	66	108
6	54	115
7	44	92
8	50	90

Add 2 ml of APDC solution, mix and set aside for 5 min. Add 10 ml of 4-methylpentan-2-one, measured accurately using a pipette, and shake vigorously for 1 min. Allow to separate and remove the clear organic layer with a Pasteur pipette. Aspirate the organic layer into the atomic absorption spectrophotometer fitted with a lead hollow cathode lamp set at 283.3 nm.

5.2.5.4 *Calibration curve*. Submit aliquot portions of standard lead solution containing 0 to 20 μg of lead to the procedure in **5.2.5.3**. Construct a new curve for each set of measurements.

5.2.6 *Calculation*. Relate the test result to the calibration curve and calculate the lead content in mg/kg (p.p.m.).

5.3 Determination of pH value. Dilute the test paste with 4 parts of water to 1 part of test paste.

Determine the pH value of this slurry at a temperature of 23 ± 1 °C using a pH meter with a glass and calomel electrode assembly.

NOTE The following British Standards relate to pH and to performance requirements for apparatus used for determining the pH value:

BS 1647, BS 2586, BS 3145.

5.4 Determination of abrasivity by the radio-tracer method

5.4.1 *Standard reference paste*. The standard reference paste shall be formulated as follows.

Constituent	**% by mass**
Precipitated calcium carbonate[a]	40.00
Glycerol (see BS 2625)	23.00
Sodium carboxymethylcellulose (CMC)[b]	1.40
Dodecyl sodium sulphate[b]	1.00
Sodium silicate (80° TW of approximately pH7) (see BS 3984)	0.50
Saccharin sodium	0.15
Formalin (40 % (*m*/*m*) formaldehyde) (see BS 2942)	0.10
Peppermint flavouring	0.80
Water	33.05

[a] See Appendix A and Appendix D.
[b] See Appendix D.

The constituents shall comply with the requirements of the British Standards given in parentheses above. For details of mixing and further requirements concerning the precipitated calcium carbonate, see Appendix A.

5.4.2 *Apparatus*. The following apparatus is required.

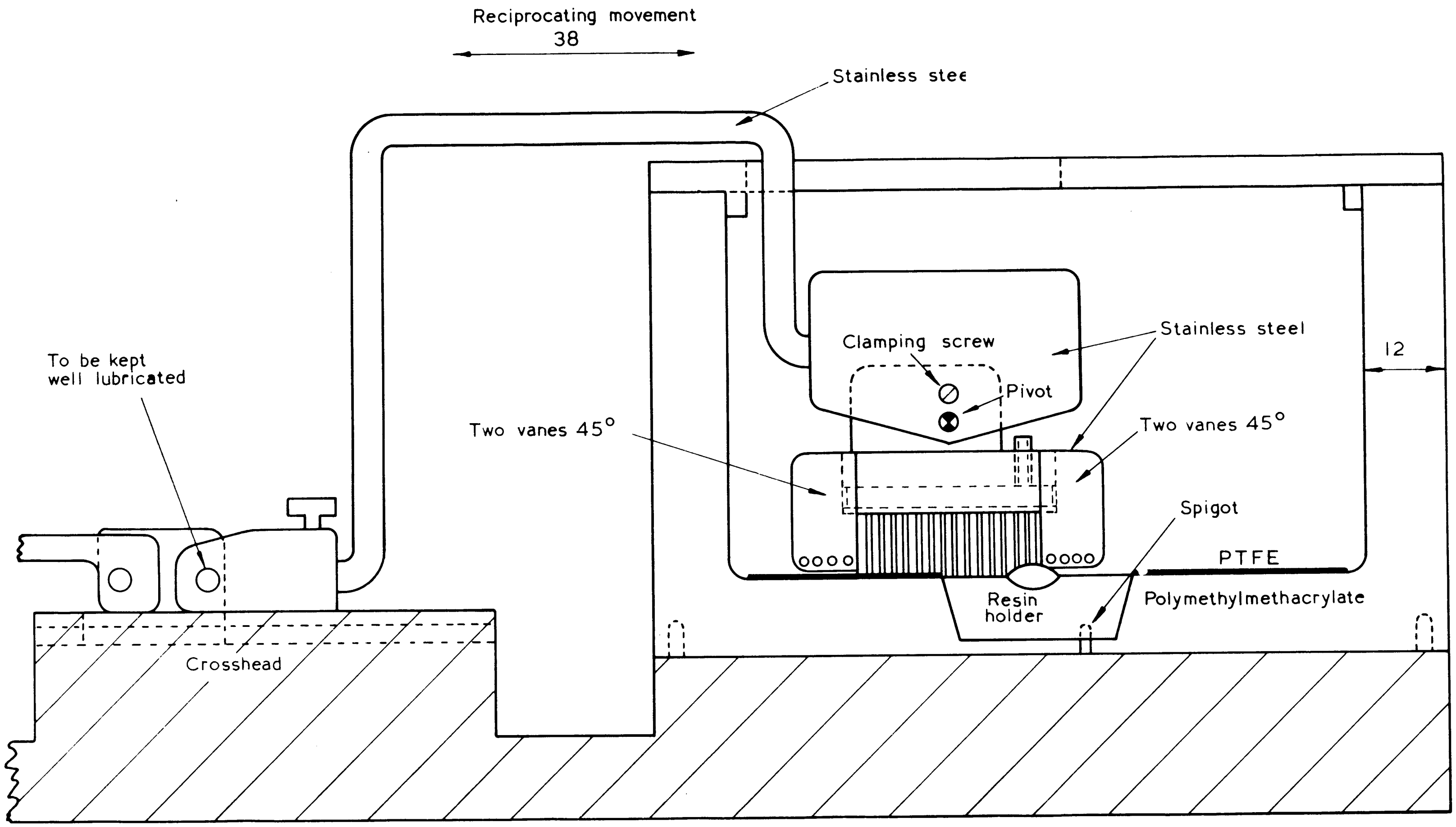
5.4.2.1 *Brushing machine*[1)], generally conforming to the design given in Figure 1 and so constructed that the tooth specimen can be withdrawn for neutron irradiation. The brushing movement shall be a 38 mm reciprocating stroke derived from a drive of 150 r/min.

NOTE It is important that the brushing machine be constructed on sound engineering principles, especially with regard to the brush movement. It is essential that the reciprocating movement of the brush is parallel both with the floor of the brushing box in a horizontal plane and with the walls of the box in a vertical plane and that the specimen (see **5.4.3**) is mounted so that the brushing movement is at right angles to its long axis. (See also Appendix B regarding brush head angulation.)

5.4.2.2 *Brush*, complying with the relevant details given in Appendix B. The mass of the brush and associated parts shall be such that a force of 2 N is exerted on the tooth specimens.

5.4.2.3 *Source of neutron flux*, of 10^{12} neutrons/(cm^2 s).

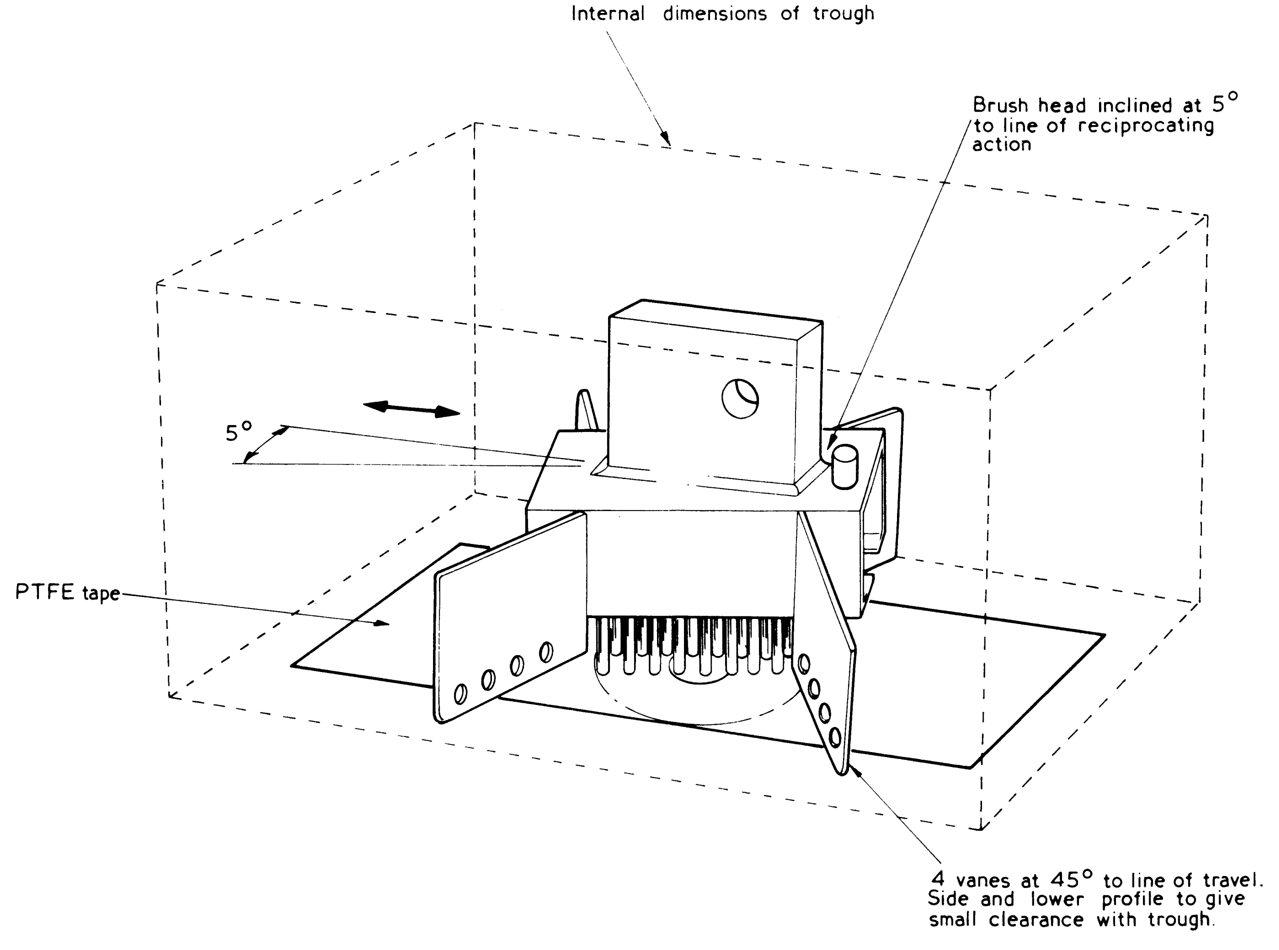
[1)] Information on the availability of a suitable machine may be obtained on application to the Secretary of DNS/6, British Standards Institution, 2 Park Street, London W1A 2BS.



All dimensions are in millimetres

(a) General view

Figure 1 — Toothpaste testing rig



(b) Brushing head with stirring vanes

Figure 1 — Toothpaste testing rig (*concluded*)

5.4.2.4 *Counting equipment*, suitable for a source of β radiation.

5.4.3 ***Preparation of tooth specimens***

5.4.3.1 *Enamel.* Prepare a specimen using human permanent teeth which have been stored, since extraction, in water with a suitable bacteriostatic agent such as chlorhexidine gluconate[2)] (1 % concentration). Cut off the root and mount the crown or slice of the crown, in a mounting medium such as epoxy resin[2)] with the labial or buccal surface uppermost and in an orientation to give crosswise brushing in the machine [see Figure 2(a) and Figure 2(b)]. The specimen shall stand proud of the surface by about 2 mm and shall have an area of about 50 mm^2 exposed, which shall be intact and undamaged.

NOTE Take particular care to ensure that no dentine or cementum is exposed. Such tissues wear many times faster than enamel and even a 1 % exposure could double the effective wear rate of the specimen.

5.4.3.2 *Dentine.* Prepare a specimen using single rooted human permanent teeth which have been stored, since extraction, in water with a suitable bacteriostatic agent such as chlorhexidine gluconate[2)] (1 % concentration). Cut off the crown, remove enamel and root, and mount in a suitable medium such as epoxy resin[2)] as shown in Figure 3(a) and Figure 3(b). A small amount of exposed enamel will not seriously affect the overall wear rate of the specimen. The specimen shall stand proud of the surface by 1 mm to 2 mm and have an area of at least 50 mm^2 exposed, which shall be intact and undamaged.

An alternative source of dentine tissue is the root of a large maxillary incisor; see Figure 3(c) and Figure 3(d).

[2)] See Appendix D

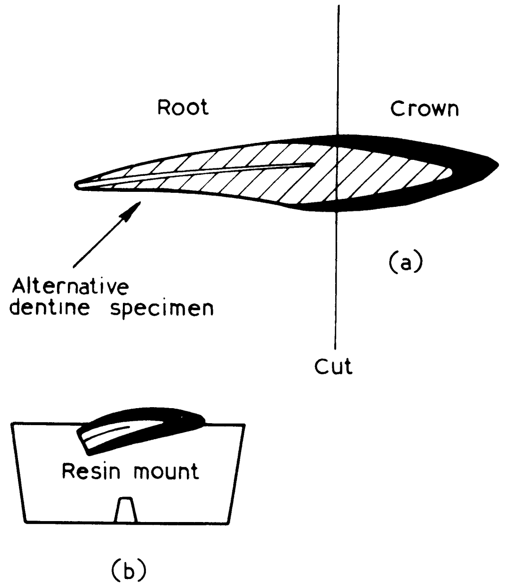


Figure 2 — Preparation of enamel specimen

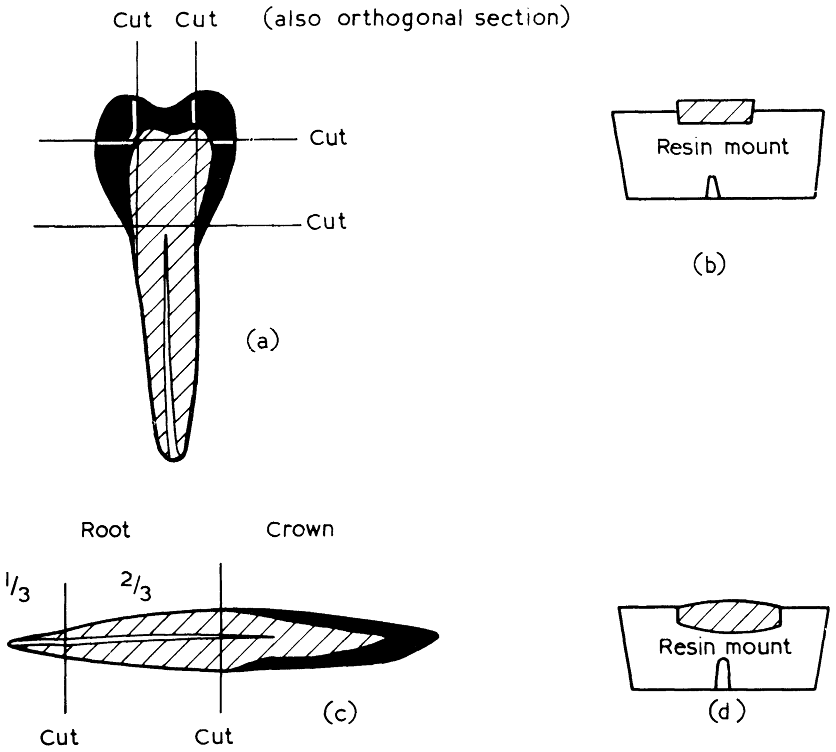


Figure 3 — Preparation of dentine specimen (radio-tracer method)

5.4.3.3 *Mounting technique and storage.* Thoroughly clean the teeth selected for test purposes using a dental polishing paste on a rotating brush held in a straight dental handpiece, and then carefully wash to remove any abrasive matter. Cut the specimen under a jet of water using a diamond disc rotating in a straight dental handpiece. The specimen may be held in a stick of impression compound[3)], the end of which is softened by heating in a flame.

Carefully dry the surface of the specimen without heating, prior to mounting in epoxy resin. The casting of the tapered holder and mounting of the tissue may be performed in one operation using the moulding block shown in Figure 4. Use polyvinyl alcohol as the release agent. Do not use any mounting medium that might lead to a level of radioactivity after neutron irradiation, which would interfere with the test.

After about 12 h, remove the mounted specimen from the mould and transfer it to a vessel containing water and a suitable bacteriostatic agent such as chlorhexidine gluconate[3)] (1 % concentration). Immediately prior to irradiation, thoroughly brush the tooth using the reference paste, wash in water and place in a sealed polyethylene capsule filled with fresh water and a bacteriostat. At no time shall the specimens be allowed to dry out except for the short period required for mounting purposes. Avoid handling the specimens or using detergents for cleaning, to limit formation of ^{24}Na during irradiation.

5.4.4 *Irradiation of tooth specimen.* Irradiate the polyethylene capsule containing the specimens, water and bacteriostat to an integrated thermal neutron flux of 10^{16} neutrons/(cm^{2}s) (dentine) or 3×10^{16} neutrons/(cm^2 s) (enamel). (For instance, 3 h for dentine and 10 h for enamel in a thermal neutron flux of 10^{12} neutrons/(cm^2 s) would be suitable. Avoid using flux levels in excess of 10^{12} neutrons/(cm^2 s).)

Place the specimens at a position in the thermal column where the cadmium ratio (gold) is 7 or higher and the temperature less than 65 °C. The irradiation should be carried out with 20 cm of lead shielding to reduce the gamma ray dose received by the specimens.

The total absorbed dose received by the tissues from all sources of radiation should not exceed 3 Mrad (dentine) and 10 Mrad (enamel). The final specific activity will be of the order of 1 MBq.

Carry out the irradiation using a low temperature reactor designed to keep the temperature of the specimen as low as possible, but in no case above 65 °C.

After removing the capsule from the reactor, wait 4 days before carrying out the test procedure to allow any highly radioactive elements, such as ^{24}Na, to decay.

Handle the mounted, irradiated specimen in accordance with normal practice for a source of the above strength and type. 10 mm of polymethyl methacrylate or water provides sufficient shielding from the β particles. Under no circumstances handle the specimen without tongs (length greater than 0.25 m) or view directly without suitable goggles. Detailed advice on the handling of radioactive tracer elements should be sought before carrying out the experiments.

5.4.5 *Conditioning of tooth specimen.* Transfer the radioactive specimens of dentine and enamel to the trough of the brushing machine and condition their surfaces, at room temperature, by brushing for 1 000 and 10 000 double brush strokes respectively in a slurry of the standard reference paste (20 g of standard reference paste plus 70 g of diluent, 0.5 % of aqueous solution of sodium carboxymethylcellulose[3)]). Determine the volume of this slurry before adding it to the trough as its magnitude is required for correction purposes later. At the end of the conditioning period, pour away the slurry into a suitable container and thoroughly wash the brush and specimen in water.

[3)] See Appendix D.

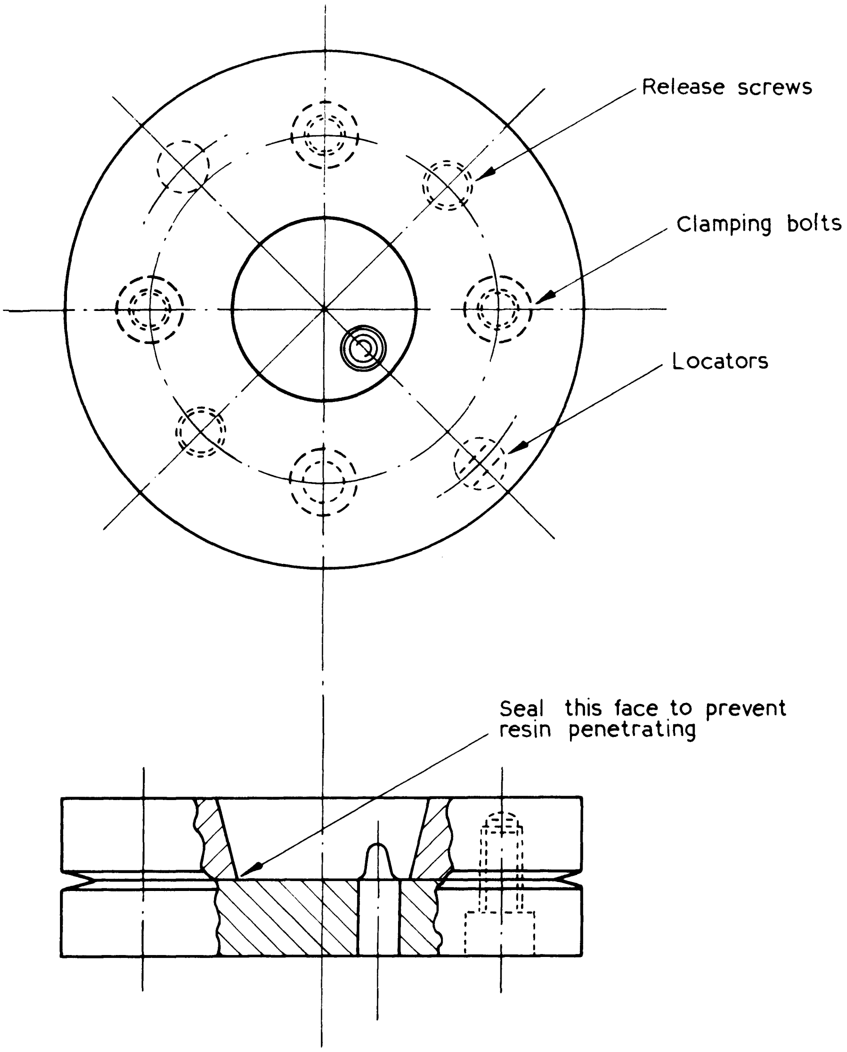


Figure 4 — General arrangement of mould for tooth mount

5.4.6 *Procedure.* Dry the trough containing the specimen with a paper tissue, weigh and add 30 g of the standard reference paste slurry, made up to a concentration of 67 % (20 g of standard reference paste plus 10 g of diluent, 0.5 % of aqueous solution of sodium carboxymethylcellulose[4]). After ensuring that it is adequately covered, brush the specimen for 250 or 1 000 double strokes, depending upon whether the specimen is dentine or enamel. At the end of this run, add 15 ml of diluent to the trough and after thorough mixing with a glass rod fitted with a rubber cap, brush for a further 250 or 1 000 double strokes. Repeat this procedure for a further three additions of 15 ml of diluent.

Pour the active slurry from the trough and break the foam, if present, with a drop of diethyl ether. After gentle stirring, withdraw two 1 ml samples of the slurry with a pipette and transfer to aluminium planchets of 25 mm diameter. Dry the planchets under an infra-red lamp or in an oven.

An alternative, gravimetric, method of sampling may be used, as follows.

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[4] See Appendix D.

Pour the active slurry from the trough into a 250 ml beaker and break the foam, if present, with a drop of diethyl ether or acetone. After gentle stirring, weigh out two 1 g samples onto aluminium planchets of 25 cm diameter, using a top-loading balance accurate to 10 mg. Dry the planchets under an infra-red lamp or in an oven. When using this method, do not correct the activities for the different densities of the slurries as stated in **5.4.7**.

Carry out the above conditioning and test procedure at room temperature, first with the standard reference paste, and then with the test paste followed by a repeat run with the standard reference paste. No dentine specimen shall be used for more than 50 000 brush strokes, but this may be extended for enamel so long as there is no evidence of dentine exposure.

5.4.7 *Test results*. Determine the radioactivity of the slurry samples with a 50 mm diameter end-window Geiger Müller counter suitable for β rays of 1.7 meV. Place the planchets close to the window. Correct the count rate for "dead time" and background errors for a total of 5 000 counts. Calculate the mean. If their individual values differ by more than 5 % from the mean, the test shall be discarded.

Determine the levels of radioactivity of the planchets within a short period of one another so that corrections for decay in activity are reduced to low values. Make any corrections that are necessary on the basis of a half-life of 14.3 days (^{32} P). Corrections for β absorption and backscatter can be neglected when the above counter geometry is used. If other counter geometries are used, activities have to be corrected using appropriate radio-tracer techniques.

As a constant mass of dentifrice and diluent is added to the trough, whilst a constant volume is withdrawn for sampling, it is necessary to make a small correction for the slightly different ratios of sample to total volumes of paste slurry used in the different tests. This correction factor can be ascertained from the measured volumes of dentifrice slurry employed for the conditioning runs, since these are also performed at the same concentration, namely 22 % (*m*/*m*).

The ratio of the abrasivity of the test paste to the standard reference paste is given by the corrected ratio of the count rates. For details of the recommended statistical procedure, see Appendix C.

5.4.8 *Alternative procedure for measurement of radioactivity of slurry*. Immediately after the brushing programme has been completed, pour the active slurry from the trough into a beaker and break the foam, if present, with a drop of diethyl ether. After gentle stirring, transfer a proportion of the slurry to a suitable β particle counter having a nominal capacity of 10 ml.

Carefully wipe away any surplus slurry emerging from the overflow and immediately determine the β activity of the sample by counting for a maximum period of 3 min, in the case of dentine specimens, and 5 min for enamel specimens. Within these periods record at least 1 000 counts with the standard reference paste slurry. If the number of counts is less than this, then it is advisable to use the dry counting procedure. Count both the standard reference paste slurry and the test paste slurry for the same period of time (the tests again following the sequence: standard; test; standard). The ratio of the number of counts recorded is a measure of the relative abrasivity after corrections for counter dead time and background radiation have been applied. No corrections are necessary for absorption, or, if the measurements are made within a short period of one another, for normal activity decay. Small corrections may have to be applied to allow for the fact that the sampling volumes from the two paste slurries will not always be the same fraction of the total final volume of slurry used for the test. This correction is similar to that applied with the dry counting procedure.

6 Marking

6.1 Information. The immediate container (i.e. tube), or any outer container, shall be clearly marked with the following information.

a) The name or trade mark of the manufacturer or the supplier.

b) Nominal volume of contents in ml at the time of packing[5].

c) The number of this British Standard, i.e. BS 5136.

NOTE Marking BS 5136 on or in relation to a product is a claim by the manufacturer that the product has been manufactured in accordance with the requirements of the standard. The accuracy of such a claim is therefore solely the manufacturer's responsibility. Enquiries as to the availability of third party certification to support such claims should be addressed to the Director, British Standards Institution, Maylands Avenue, Hemel Hempstead, Herts HP2 4SQ in the case of certification marks administered by BSI or to the appropriate authority for other certification marks.

Details of the volume contained in the tube, as referred to in b), shall be readily visible to the purchaser without the need to remove any outer container used for retail packaging of the tube.

6.2 Decorative paints. Paints used in decorating and marking toothpaste tubes shall contain not more than 250 mg/kg (250 p.p.m.) of lead when a sample, prepared in accordance with Appendix B of BS 3443:1968, is tested using a recognized method of chemical analysis.

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[5] The contents of toothpastes packed in tubes has to be expressed as a volume, in ml, although the contents of toothpastes packed in the containers may be expressed either as a volume, in ml, or as a mass, in g, (see foreword).

Appendix A Details of the standard reference paste

A.1 Abrasive

Dense precipitated calcium carbonate (see Appendix D), which has been calibrated against the primary standard material held by the Laboratory of the Government Chemist, Cornwall House, Stamford Street, London SE1.

Physical and chemical characteristics of the calcium carbonate are as follows

% purity as $CaCO_3$	98 % min. (dry)
Acid insoluble matter	0.2 % max.
pH (20 % (*m*/*m*) aqueous slurry measured after 10 min)	9.7 max.
Total calcium as Ca	38.5 % to 40.0 %
Loss on heating to 105 °C	2.0 % max.
Apparent density (fluffed)	0.78 g/cm^3 to 0.89 g/cm^3
Flow point	13 ± 1
Fineness through a 75 µm aperture[a] BS 410 test sieve	99.7 % min.
Mean particle size (Coulter)	12.5 ± 2.0 µm

[a] This corresponds to 200 mesh.

NOTE Because of the extreme sensitivity of the methods of abrasivity measurement, even two apparently identical batches of the same reference abrasive material have been found to produce significantly different test results; because of this it is most important that the reference abrasive material be calibrated against a primary standard.

For the same reason also, it is strongly recommended that the calcium carbonate listed in Appendix D be used. This particular product has been thoroughly investigated as regards its suitability for the test procedures and, subject to calibration, is known to be satisfactory.

A.2 A recommended method of manufacture

A.2.1 *Constituents*. The constituents and the order in which they are used in the mixing procedure are listed below (see **5.4.1** for further details of constituents).

Constituent	% by mass	Procedure
Glycerol	23.00	a)
Sodium CMC	1.40	
Saccharin sodium	0.15	b)
Sodium silicate	0.50	c)
Water	5.00	
Water	20.55	d)
Calcium carbonate	40.00	e)
Water	7.50	f)
Dodecyl sodium sulphate	1.00	
Formalin	0.10	g)
Peppermint flavour	0.80	

A.2.2 *Procedure*. The procedure shall be performed as follows.

a) Use a vertical bowl mixer with an anchor-shaped blade. Weigh the glycerol into a tared mixing bowl and, with slow stirring, add the weighed amount of sodium CMC.

b) Add the saccharin sodium.

c) Weigh the 5 % quantity of water into a beaker, add the sodium silicate and stir until dissolved.

d) Add this to the dispersion in the mixing bowl followed by the 20.55 % quantity of water. Transfer to a vacuum mixing vessel and stir for 15 min on slowest speed until the gel is smooth.

e) Weigh the calcium carbonate and add it slowly to the gel. When all the powder is mixed, stop the stirrer and scrape the blade and side of the mixing bowl. Restart the stirrer and mix, under vacuum, for 10 min at the slowest speed, until smooth.

f) Weigh the dodecyl sodium sulphate into a beaker, add the remaining 4.5 % water (warmed to approximately 45 °C), and stir until dissolved.

Add this to the mix in the mixing bowl with slow stirring for 3 min under vacuum.

g) Add the formalin, followed by the peppermint flavouring and stir for a further 2 min under vacuum.

NOTE No heating is necessary other than for step f), where the use of warm water assists in dissolving the dodecyl sodium sulphate.

Appendix B Brush head mounting, conditioning, replacement and design

B.1 Mounting

The brush head shall be attached to the driving arm of the brushing machine, with the long axis of the brush head at an angle of between 4° and 5° (in the horizontal plane) to the direction of the reciprocating movement.

B.2 Conditioning

The brush head shall be immersed in water for a minimum of 3 h before commencing the test procedure.

B.3 Replacement

Replace the brush head after it has been used for approximately 50 000 brush strokes.

B.4 Design

Although the design of brush head[6] does not appear to affect the results of the abrasion test, it is advisable, in the interests of sensitivity, to use a brush of low compliance. (The term "compliance" may be defined as the property of a brush to conform to a given surface.) This may be achieved in practice by selecting a brush with nylon 6.10 filaments of approximately 0.30 mm diameter[7], set out with minimum tuft spacing to give a high density of filaments. A flat or serrated trim is recommended and the filaments should be between 11 mm and 12 mm in length. A three or four row brush is desirable with a width of between 7.5 mm and 10 mm. A brush length of 31 mm to 38 mm fits in well with the stroke displacement of the brushing machine.

Appendix C Recommended statistical analysis

C.1 Test the toothpaste in the sequence: standard; test; standard.

C.2 Calculate the arithmetic mean (R) of the two standard reference readings. (If they differ by more than 40 % of this mean value, the complete test should be repeated).

C.3 Take logarithms of the test (T) and the mean standard reference (R) paste wear values.

C.4 Subtract log T from log R for each pair of variates and calculate the standard error $s/\sqrt{n}$ of their mean difference, D_{m}, where s is the standard deviation.

C.5 Compare the ratio

a) *for the radio-tracer method*

$$\frac{D_{\mathrm{m}} + \log 2 \text{ (or } \log 4)}{s/\sqrt{n}}$$

where

log 2 (or log 4) is the abrasivity limit (see **3.5.1**)

n is the number of test sequences performed (see **C.1**)

b) *for the surface profile method*

$$\frac{D_{\mathrm{m}} + \log 1.5 \text{ (or } \log 3)}{s/\sqrt{n}}$$

where

log 1.5 (or log 3) is the abrasivity limit (see **3.5.2**)

n is the number of test sequences performed (see **C.1**)

with the value of the random variate of Students' t distribution for $n - 1$ degrees of freedom and at a significance level of 10 % (corresponding to 5 % level single sided test). If the ratio is positive and greater than t, then the toothpaste shall be accepted.

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[6] Information on a brush which fits the details given may be obtained on application to the Secretary of DNS/6, British Standards Institution, 2 Park Street, London W1A 2BS.

[7] This diameter filament is usually found in "hard" grade brushes.

C.6 The required number of test sequences, *n*, will depend upon the expected level of abrasivity of the test sample relative to that of the reference paste. If the ratio is small, there will be no need to carry out a large number of repeat measurements to meet the specification. Alternatively, if the ratio of the *t* test: reference paste abrasivity is close to the relevant limit, then more measurements may be necessary in order to establish a satisfactory comparison. In general, a minimum value of $n = 4$ should be aimed at and the tests should be carried out on at least two dental tissue specimens of the same type.

Appendix D Reagents

Experience has shown that any sensitive method of abrasivity measurement, such as that specified in this British Standard, can be significantly affected by undetectable characteristics in the reagents used. It is for this reason that reference is made to the particular commercial products listed below, all of which have been used in the investigations associated with development of the specified test, and not by way of any implication as regards quality. Each product is listed under the clause number where it is mentioned.

Appendix E Determination of abrasivity by the surface profile method

E.1 Introduction

This method may be used as an alternative to the radio-tracer method, subject to the conditions stated in **3.5**

E.2 Apparatus

The following apparatus is required.

E.2.1 *Surface profile measuring instrument*[8], fitted with a stylus of tip radius approximately 0.25 µm, and being capable of measuring the cross-sectional areas of grooves 0.5 mm to 2.5 mm wide and 0.1 µm to 20 µm deep.

E.2.2 *Planimeter*, for measuring the cross-sectional area of the abraded groove. Alternatively, an integrator attachment may be used to measure the area automatically.

E.2.3 *Waterproof silicon carbide papers*, grades 220, 320, 400 and 600.

E.2.4 *Lapping machine*, with an unslotted cast iron wheel. The abrading surface is impregnated with 3 µm powdered diamond for the final polishing of enamel samples.

E.2.5 *Brushing machine*, as detailed in **5.4.2.1**.

E.2.6 *Brush*, as detailed in **5.4.2.2**.

E.3 Preparation of enamel and dentine specimens

Prepare specimens from human permanent teeth which have been stored, since extraction, in water with a suitable bacteriostatic agent e.g. chlorhexidine gluconate[9] (1 % concentration). The specimens should be cut as shown in Figure 2 and Figure 3.

E.4 Mounting technique and storage

Thoroughly clean the teeth selected for test purposes with a dental polishing paste on a rotating brush held in a straight dental handpiece, and then carefully wash to remove any abrasive matter. Cut the specimen under a jet of water using a diamond disc rotating in a straight dental handpiece. The specimen may be held in a stick of impression compound[9], the end of which is softened by heating in a flame. Carefully dry the surface of the specimen without heating, prior to mounting in epoxy resin. The casting of the tapered holder and mounting of the specimen may be performed in one operation using the moulding block shown in Figure 4, Use polyvinyl alcohol as the release agent.

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[8] Talysurf 10, manufactured by Rank Taylor Hobson is known to be suitable for this purpose.

[9] See Appendix D.

Clause reference	Reagent	Commercial product and manufacturer
5.4.1	Precipitated calcium carbonate	Sturcal L (J. and E. Sturge Ltd.)
	Sodium carboxymethylcellulose (CMC)	7MF (Hercules Powder Co. Ltd.)
	Dodecyl sodium sulphate	Dental grade (Cyclo Chemicals Ltd.)
5.4.3	Epoxy resin	Araldite MY 753 (Ciba)
	Impression compound	Green or brown stick impression compound (Dental Fillings Ltd.)
	Chlorhexidine gluconate	Hibitane (Imperial Chemical Industries Ltd.)
5.4.4	Polymethyl methacrylate	Perspex (Imperial Chemical Industries Ltd.)
5.4.5 and **5.4.6**	Sodium carboxymethylcellulose (CMC)	7MF (Hercules Powder Co. Ltd.)

Mount the specimen horizontally in the epoxy resin such that it is just below the surface as shown in Figure 5(a). The orientation of the specimen shall be perpendicular to the direction of brushing.

After 12 h, remove the mounted specimen from the mould and transfer it to a vessel containing water and a suitable bacteriostatic agent e.g. chlorhexidine gluconate[10] (1 % concentration). At no time shall the specimens be allowed to dry out, except for the short period required for mounting purposes.

E.5 Conditioning

Grind the upper face using the grades of waterproof silicon carbide paper, water lubricated, to expose an area of the specimen 2 mm by 6 mm. Continue the grinding operation using progressively finer silicon carbide papers, water lubricated, finishing with 600 grade paper. Grind the upper face so that it is parallel to the lower face. To assist in achieving the degree of parallelism, a tilted, water-lubricated grinding table[11] may be used. Finish the grinding process for enamel specimens using the lapping machine.

The tolerance of flatness of the upper face, as measured by the surface profile measuring instrument, shall be as follows.

a) *For dentine*, the surface shall not deviate by more than 1.0 µm from absolute flatness, measured across the test area in a direction perpendicular to the direction of brushing;

b) *For enamel*, the surface shall not deviate by more than 0.1 µm from absolute flatness, measured across the test area in a direction perpendicular to the direction of brushing.

Prior to each test run, repeat the final polishing operation (using 600 grade silicon carbide paper for dentine and the lapping machine for enamel) to remove the minimum amount of tissue to achieve flatness within the tolerances defined above.

E.6 Procedure

Mask the boundaries of the test area with two strips of unplasticized PVC self-adhesive tape (maximum thickness of 50 µm) to expose a 2 mm wide tract parallel with the direction of brushing. The object of the masking operation is to create an unabraded reference surface either side of the test area. The position of the masking tape with respect to the specimen is illustrated in Figure 5(b).

Set the specimen in the trough with the exposed face standing approximately 2 mm proud of the surface of the trough. Carry out the brushing operation as in **5.4.6** with respect to sequential dilution, but use only 200 strokes per dilution for dentine and 2 000 for enamel.

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[10] See Appendix D.

[11] The table manufactured by Metaserve Ltd. is known to be suitable for this purpose.

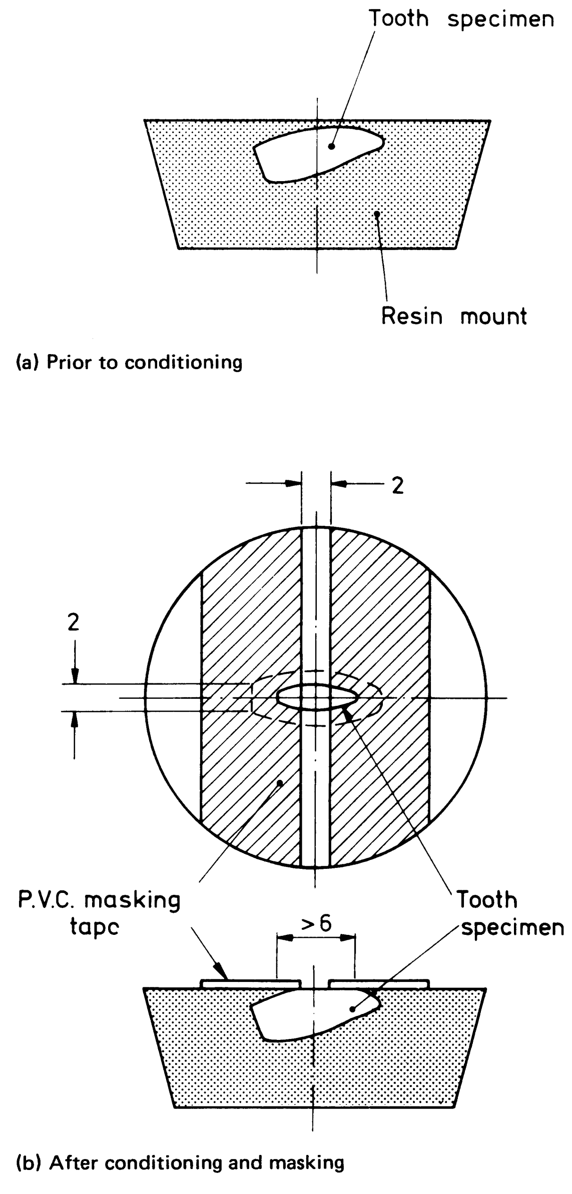

Figure 5 — Mounted tooth specimen (surface profile method)

The testing sequence shall be: reference paste; test paste; reference paste.

The reference paste shall be formulated as in **5.4.1**. After the brushing has been completed, remove the mounted specimen, wash clean, and dry using paper tissue. Measure the contour of the abraded groove using the surface profile measuring instrument, and measure the area between the base line and the magnified trace using the planimeter. Divide the cross-sectional area of the abraded groove by the width of the groove, in order to obtain the average groove depth.

E.7 Treatment of results

The results obtained are treated as described in Appendix C to assess compliance with the requirements of **3.5.2**.

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Publications referred to

BS 410, *Specification for test sieves.*

BS 1647, *pH scale.*

BS 2586, *Glass electrodes for measurement of pH.*

BS 2625, *Chemically pure glycerol*[12)].

BS 2942, *Formaldehyde solution.*

BS 3145, *Specification for laboratory pH meters.*

BS 3443, *Code of safety requirements for children's toys and playthings.*

BS 3978, *Water for laboratory use.*

BS 3984, *Sodium silicates.*

[12)] Included in BS 2621-5 "*Glycerol (glycerine)*".

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