© SABS. This standard may reside on internal network or intranet on a Single Site only and is exclusively available to The Cosmetic, Toiletry and Fragrance Association in accordance with copyright exploitation agreement no. 014/009/26-295, valid until 2027-07-31. The single-site licence allows authorised staff member of The Cosmetic, Toiletry and Fragrance Association to make five (5) printouts of the standard and will expire after one year unless renewed. Unauthorised copying, sharing, distribution, or access outside approved networks is prohibited.

ISBN 978-0-626-21439-5

SANS 1302:2008

Edition 1.1
Any reference to SABS 1302 is deemed
to be a reference to this standard
(Government Notice No. 1373 of 8 November 2002)

SOUTH AFRICAN NATIONAL STANDARD

Toothpaste

Published by Standards South Africa
1 dr lategan road groenkloof ✉ private bag x191 pretoria 0001
tel: 012 428 7911 fax: 012 344 1568 international code + 27 12
www.stansa.co.za
© Standards South Africa

standards SouthAfrica
(a division of SABS)

This product is copyrighted by SABS, 1 Dr Lategan Road, Groenkloof, 0181, South Africa. All rights reserved.

© SABS. This standard may reside on internal network or intranet on a Single Site only and is exclusively available to The Cosmetic, Toiletry and Fragrance Association in accordance with copyright exploitation agreement no. 014/009/26-295, valid until 2027-07-31. The single-site licence allows authorised staff member of The Cosmetic, Toiletry and Fragrance Association to make five (5) printouts of the standard and will expire after one year unless renewed. Unauthorised copying, sharing, distribution, or access outside approved networks is prohibited.

SANS 1302:2008
Edition 1.1

Table of changes

Change No.	Date	Scope
Amdt 1	2008	Amended to change the designation of SABS standards to SANS standards, to update the definition of "acceptable," and to delete reference to national legislation.

Foreword

This South African standard was approved by National Committee StanSA SC 5140.21B, *Cosmetics – Toothpaste*, in accordance with procedures of Standards South Africa, in compliance with annex 3 of the WTO/TBT agreement.

This document was published in June 2008. This document supersedes SABS 1302:1980 (first edition).

A reference is made in 3.1.2 and 3.1.8 to the use of colourants and the determination of fluoride ion content in accordance with "the current relevant national legislation". In South Africa this means the Foodstuffs, Cosmetics and Disinfectants Act, 1972 (Act No. 54 of 1972).

A reference is made in 4.1.1 to the allowed nominal volume of toothpaste contained in a tube in terms of the regulations framed under "the current relevant national legislation." In South Africa this means the Trade Metrology Act, 1973 (Act No. 77 of 1973).

A vertical line in the margin shows where the text has been technically modified by amendment No.1.

This product is copyrighted by SABS, 1 Dr Lategan Road, Groenkloof, 0181, South Africa. All rights reserved.

© SABS. This standard may reside on internal network or intranet on a Single Site only and is exclusively available to The Cosmetic, Toiletry and Fragrance Association in accordance with copyright exploitation agreement no. 014/009/26-295, valid until 2027-07-31. The single-site licence allows authorised staff member of The Cosmetic, Toiletry and Fragrance Association to make five (5) printouts of the standard and will expire after one year unless renewed. Unauthorised copying, sharing, distribution, or access outside approved networks is prohibited.

Contents

Page

Foreword

1 Scope 3

2 Definitions 3

3 Requirements 4

4 Packing and marking 5

5 Sampling and compliance with the specification 6

6 Inspection and methods of test 6

6.1 Inspection 6
6.2 Test samples 6
6.3 Methods of tests 7
6.3.1 Consistency 7
6.3.2 Lead content 7
6.3.3 pH value 9
6.3.4 Abrasiveness 9
6.3.5 Extrusion test 12
6.3.6 Stability 13
6.3.7 Determination of fluoride ion content 13
6.3.8 Test for contamination and attack on tube and cap 15

Figures 1-5 15-18

Appendix A Applicable standards 19

Appendix B Notes to purchasers 19

This product is copyrighted by SABS, 1 Dr Lategan Road, Groenkloof, 0181, South Africa. All rights reserved.

© SABS. This standard may reside on internal network or intranet on a Single Site only and is exclusively available to The Cosmetic, Toiletry and Fragrance Association in accordance with copyright exploitation agreement no. 014/009/26-295, valid until 2027-07-31. The single-site licence allows authorised staff member of The Cosmetic, Toiletry and Fragrance Association to make five (5) printouts of the standard and will expire after one year unless renewed. Unauthorised copying, sharing, distribution, or access outside approved networks is prohibited.

This page is intentionally left blank

This product is copyrighted by SABS, 1 Dr Lategan Road, Groenkloof, 0181, South Africa. All rights reserved.

© SABS. This standard may reside on internal network or intranet on a Single Site only and is exclusively available to The Cosmetic, Toiletry and Fragrance Association in accordance with copyright exploitation agreement no. 014/009/26-295, valid until 2027-07-31. The single-site licence allows authorised staff member of The Cosmetic, Toiletry and Fragrance Association to make five (5) printouts of the standard and will expire after one year unless renewed. Unauthorised copying, sharing, distribution, or access outside approved networks is prohibited.

Toothpaste

1 Scope

1.1 This specification covers the requirements for toothpastes (fluoridated and non-fluoridated) intended for use with a brush in the cleaning of natural teeth.

NOTE

a) The standard referred to in the specification is noted in appendix A.

b) Requirements that must be specified by the purchaser are listed in appendix B.

2 Definitions

2.1 For the purposes of this specification the following definitions shall apply:

acceptable
acceptable to the authority administering this standard, or to the parties concluding the purchase contract, as relevant **Amdt 1**

batch
that quantity of toothpaste made from the same materials and filled into tubes in one continuous cycle of operations

composite pack
a pack containing two or more tubes of toothpaste of the same nominal volume or of different nominal volumes, and in which the tubes are separated from one another

current statutory formulary
one of the formularies official in the Republic of South Africa at the date of manufacture of the product

dentine
the calcified tissue, tubular in structure, that forms the bulk of a tooth

enamel
the highly calcified tissue, prismatic in structure, that covers the crown of a tooth

lot
not less than 100 tubes of toothpaste of the same nominal volume, bearing the same batch identification, and packed in individual containers, from one manufacturer, or not less than 100 identical composite packs from one manufacturer, submitted at any one time for inspection and testing

This product is copyrighted by SABS, 1 Dr Lategan Road, Groenkloof, 0181, South Africa. All rights reserved.

© SABS. This standard may reside on internal network or intranet on a Single Site only and is exclusively available to The Cosmetic, Toiletry and Fragrance Association in accordance with copyright exploitation agreement no. 014/009/26-295, valid until 2027-07-31. The single-site licence allows authorised staff member of The Cosmetic, Toiletry and Fragrance Association to make five (5) printouts of the standard and will expire after one year unless renewed. Unauthorised copying, sharing, distribution, or access outside approved networks is prohibited.

3 Requirements

3.1 Toothpaste

3.1.1 Type

Toothpaste shall be of the fluoridated type or the non-fluoridated type, as specified by the purchaser.

3.1.2 Composition

Any colourants used shall be those permitted for use in terms of the regulations framed under the current relevant national legislation (see foreword). A toothpaste shall not contain mono- or di-saccharides, except those through which acceptable clinical trials have been proved to be non-cariogenic. Ingredients that are listed in a current statutory formulary shall be of the purity laid down in that publication, all ingredients shall be of a quality suitable for the intended use, and shall comply with the relevant legal requirements. **Amdt 1**

3.1.3 Consistency

A toothpaste shall have an acceptable consistency and, when tested in accordance with 6.3.1, shall be free from lumps and particles that are palpable in the mouth as separate entities.

3.1.4 Freedom from toxicity

a) **General**. A toothpaste shall not contain any substance which would be harmful if ingested during normal brushing and shall not, in normal use, cause a toxic or irritant reaction when used in the oral cavity, or be otherwise harmful.

b) **Lead content**. The lead content of a toothpaste, determined in accordance with 6.3.2, shall not exceed 5 µg/g.

3.1.5 pH value

The pH value of a toothpaste, determined in accordance with 6.3.3, shall be not less than 4,5 and not more than 10,5.

3.1.6 Abrasive properties

When tested in accordance with 6.3.4, the abrasiveness of a toothpaste shall not exceed the following limits:

a) To dentine: Twice that of the reference paste.

b) To enamel: Four times that of the reference paste.

3.1.7 Stability

When tested in accordance with 6.3.6, a toothpaste shall not ferment, segregate, or otherwise deteriorate.

3.1.8 Fluoride ion content

The fluoride ion content of a fluoridated toothpaste, determined in accordance with 6.3.7, shall be as laid down in the current relevant national legislation (see foreword). **Amdt 1**

This product is copyrighted by SABS, 1 Dr Lategan Road, Groenkloof, 0181, South Africa. All rights reserved.

© SABS. This standard may reside on internal network or intranet on a Single Site only and is exclusively available to The Cosmetic, Toiletry and Fragrance Association in accordance with copyright exploitation agreement no. 014/009/26-295, valid until 2027-07-31. The single-site licence allows authorised staff member of The Cosmetic, Toiletry and Fragrance Association to make five (5) printouts of the standard and will expire after one year unless renewed. Unauthorised copying, sharing, distribution, or access outside approved networks is prohibited.

3.2 Tubes

3.2.1 Paint

The lead content, determined in accordance with 6.3.2, of the paints on a toothpaste tube shall not exceed 250 µg/g.

3.2.2 Extrusion from the tube

When a tube is tested in accordance with 6.3.5, at least 95 % of the nominal volume of the toothpaste shall be extruded from the tube.

3.2.3 Contamination and attack on tube and cap

When a tube of toothpaste is tested in accordance with 6.3.8, neither the tube nor its cap shall show any visible sign of corrosion or other deterioration and the toothpaste shall show no sign of contamination.

4 Packing and marking

NOTE The packing and marking shall comply with all the relevant statutory requirements.

4.1 Packing

4.1.1 Tubes

Toothpaste shall be packed in acceptably sealed collapsible tubes. The tubes shall have closures that are leak-proof to the product. Except as otherwise allowed in terms of the regulations framed under the current relevant national legislation (see foreword) the nominal volume (in the case of a composite pack, the total nominal volume) of the toothpaste contained in the tube(s) in a pack shall be one of the following, as specified by the purchaser: **Amdt 1**

Any volume up to and including 15 mL, and thereafter 25; 50; 75; 100; 125; 150; 175; or 200 mL.

4.1.2 Packaging

Both individually packed tubes and composite packs shall be packed in bulk packages that are capable of protecting the contents from damage during normal transportation and storage.

Only individually packed tubes of toothpaste of the same nominal volume and bearing the same batch identification or only identical composite packs (i.e. packs of tubes containing the same nominal volume(s) and bearing the same batch identification(s)) shall be packed together in a bulk package.

4.2 Marking

4.2.1 Tubes

The following information shall appear in legible and durable marking on each tube:

a) The name of the toothpaste;

b) the name and business address of the manufacturer or importer or seller or person on whose behalf the toothpaste was packed;

c) a description of the contents;

This product is copyrighted by SABS, 1 Dr Lategan Road, Groenkloof, 0181, South Africa. All rights reserved.

© SABS. This standard may reside on internal network or intranet on a Single Site only and is exclusively available to The Cosmetic, Toiletry and Fragrance Association in accordance with copyright exploitation agreement no. 014/009/26-295, valid until 2027-07-31. The single-site licence allows authorised staff member of The Cosmetic, Toiletry and Fragrance Association to make five (5) printouts of the standard and will expire after one year unless renewed. Unauthorised copying, sharing, distribution, or access outside approved networks is prohibited.

d) in the case of fluoridated toothpaste, the words "FLUORIDE TOOTHPASTE" or "FLUORIDE DENTAL CRÈME" given in letters of height at least 3 mm in a colour which affords a distinct contrast with the background;

e) the batch identification.

4.2.2 Containers and composite packs

The information required in terms of 4.2.1(a)-(d) (inclusive) shall appear on

a) each individual container, together with the nominal volume, in millilitres, of the contents of the tube;

b) each composite pack, together with the total nominal volume, in millilitres, of the contents of the tubes in the pack.

4.2.3 Bulk packages

The information required in terms of 4.2.1 and 4.2.2 and the quantity of containers shall be indelibly marked on each bulk package.

5 Sampling and compliance with the specification

NOTE This section applies to the sampling for inspection and testing before acceptance or rejection of single lots (consignments) in cases where no information about the implementation of quality control or testing during manufacture is available to help in assessing the quality of the lot. It is also used as the procedure for adjudicating in cases of dispute.

5.1 Sampling

The following sampling procedure shall be applied (after, when relevant, checking for compliance with the requirements of 4.1.2 and 4.2.3) in determining whether a lot complies with the specification. The samples so taken shall be deemed to represent the lot.

From the lot take at random 20 individual containers, or composite packs, as relevant.

5.2 Compliance with the specification

The lot shall be deemed to comply with the requirements of the specification if, on inspection and testing, the sample taken in accordance with 5.1 is found to comply with all the relevant requirements of the specification.

6 Inspection and methods of test

6.1 Inspection

Inspect the samples taken in accordance with 5.1 for compliance with the relevant requirements of 4.1.1, 4.2.1, and 4.2.2.

6.2 Test samples

6.2.1 For tests on tubes

Use 10 tubes (of, in the case of composite packs, each nominal size) for the extrusion test (see 6.3.5) and retain the emptied tubes for the determination of the lead content of the paint (see 6.3.2.3(b)), and the toothpaste for the preparation of the composite sample(s) (see 6.2.2). Use

This product is copyrighted by SABS, 1 Dr Lategan Road, Groenkloof, 0181, South Africa. All rights reserved.

© SABS. This standard may reside on internal network or intranet on a Single Site only and is exclusively available to The Cosmetic, Toiletry and Fragrance Association in accordance with copyright exploitation agreement no. 014/009/26-295, valid until 2027-07-31. The single-site licence allows authorised staff member of The Cosmetic, Toiletry and Fragrance Association to make five (5) printouts of the standard and will expire after one year unless renewed. Unauthorised copying, sharing, distribution, or access outside approved networks is prohibited.

the other 10 tubes (of, when relevant, each nominal size) for the test for contamination and attack on tube and cap (see 6.3.8).

6.2.2 For tests on toothpaste

From the toothpaste extruded from each set of tubes bearing the same batch identification, prepare a composite sample of sufficient size for all the remaining tests by taking an approximately equal volume of paste from each of the 10 appropriate measuring cylinders used in the extrusion test (see 6.2.1 and 6.3.5). Mix thoroughly and store in a plastics container (tightly closed). Submit each composite sample to all the relevant tests on toothpaste given in 6.3.

6.3 Methods of test

6.3.1 Consistency

Test a portion of the composite sample for compliance with the requirements of 3.1.3.

6.3.2 Lead content

6.3.2.1 Apparatus

a) **Spectrophotometer**

An atomic absorption spectrophotometer with a lead hollow cathode lamp (of wavelength 283,3 nm) and an air/ acetylene flame.

b) **Sand bath**.

6.3.2.2 Reagents

NOTE Unless inconsistent with the text, use only reagents of analytical reagent grade (or if this is unobtainable, of the purest grade available) and distilled or demineralized water.

a) **Acetone**.

b) **iso-Butyl methyl ketone** (4-methylpentan-2-one), saturated with water.

c) **Ethanol**, lead free, 99 % (V/V).

d) **Nitric acid**, concentrated (density = 1,42 g/mL).

e) **Nitric acid**, dilute, c(HNO_3) = 5 mol/L.

f) **Ammonia solution**, dilute, c(NH_3) = 5 mol/L.

g) **Ammonium pyrrolidin-1-yl-dithioformate*** **solution** prepared as follows: Wash about 1,5 g of ammonium pyrrolidin-1-yl-dithioformate with 20 mL of the acetone in a sintered glass crucible. Dry with suction. Dissolve 1 g in water, dilute to 100 mL with water and mix well.

h) **Standard lead solutions**

1) **Stock solution**. Dissolve 159,9 mg of lead nitrate ($Pb(NO_3)_2$) in 1 % (V/V) nitric acid in a 100 mL volumetric flask. Dilute to the mark with the nitric acid and mix. This solution has a lead content of 1 mg/mL.

* Ammonium pyrrolidin-dithiocarbamate.

This product is copyrighted by SABS, 1 Dr Lategan Road, Groenkloof, 0181, South Africa. All rights reserved.

© SABS. This standard may reside on internal network or intranet on a Single Site only and is exclusively available to The Cosmetic, Toiletry and Fragrance Association in accordance with copyright exploitation agreement no. 014/009/26-295, valid until 2027-07-31. The single-site licence allows authorised staff member of The Cosmetic, Toiletry and Fragrance Association to make five (5) printouts of the standard and will expire after one year unless renewed. Unauthorised copying, sharing, distribution, or access outside approved networks is prohibited.

2) **Work solutions**. To 5 mL of the stock solution in a 100 mL volumetric flask, add 0,5 mL of the concentrated nitric acid, dilute to the mark with water, and mix. Into each of six 50 mL volumetric flasks pipette 1 mL of the concentrated nitric acid, then, by adding 0, 1, 2, 4, 6, 8 mL of the diluted stock solution, diluting to the mark with water and mixing, obtain solutions that contain respectively 0, 1, 2, 4, 6, 8 µg of lead/mL.

6.3.2.3 Preparation of sample solutions

a) **Toothpaste**. Weigh out accurately about 5 g of the composite sample into a silica evaporating dish. So add 10-15 mL of the ethanol to the paste as to form a slurry, and then evaporate to dryness in a water bath. Place in a muffle furnace at 100 °C and slowly raise the temperature (by 50 °C steps to avoid ignition) to 450 °C, and complete the ashing at 450 °C. If the ash is dark coloured, moisten it with a few drops of the concentrated nitric acid and reheat in the furnace at 450 °C for 1 h. Add 5 mL of water and 10 mL of the dilute nitric acid to the cooled ash and boil gently for 5 min. Filter the cooled solution into a 100 mL volumetric flask, rinsing with two successive 5 mL volumes of water and retaining as much as possible of the insoluble matter in the dish. Add 10 mL of the dilute nitric acid to the insoluble matter in the dish and boil for 5 min. Cool and filter into the volumetric flask, rinsing with water as before. Dilute to the mark with water and mix. Prepare a blank by repeating the above procedure, omitting the toothpaste sample.

b) **Paint**. Remove the paint from the appropriate tubes from the extrusion test (see 6.2.1 and 6.3.5), using a suitable lead-free organic solvent. Evaporate the solvent and then dry to constant mass. Mix the residue thoroughly, and weigh out accurately about 0,5 g into each of two test tubes. Add 5 mL of the concentrated nitric acid to each test tube and to one empty test tube (blank), and add two boiling chips to each tube. So place the tubes in the sand bath at 90-110 °C that the liquid surface is just above the sand level. After the initial fuming has subsided, increase the temperature until the vapours condense in the upper 10-20 mm of the tubes (bath temperature 160-170 °C) and maintain at this temperature for 3 h. Cool the tubes and filter the contents of each tube into a separate 25 mL volumetric flask, rinsing each tube and residue with four 4 mL volumes of water. Dilute the contents of each flask to the mark with water and mix.

6.3.2.4 Preparation of standard curves

a) **For toothpaste**

1) Pipette 5 mL of each of the working solutions (see 6.3.2.2(h)(2)) into individual separating funnels.

2) Using pH paper, check that the pH values of the solutions lie between 3 and 4, adjusting if necessary with the dilute ammonia solution. Add 2 mL of the ammonium pyrrolidin-1-yl-dithioformate solution to each funnel, mix, and allow to stand for 5 min. Add 10 mL of the iso-butyl methyl ketone to each funnel and shake vigorously for 5 min. Allow the layers to separate and remove the organic extract from each funnel using a Pasteur pipette. Aspirate the organic extracts in turn into the atomic absorption spectrophotometer, aspirating the iso-butyl methyl ketone between each determination.

b) **For paint**

Aspirate the working solutions (see 6.3.2.2(h)(2)) into the atomic absorption spectrophotometer, aspirating water between each determination.

c) In each case draw a standard curve of absorbance against lead content.

This product is copyrighted by SABS, 1 Dr Lategan Road, Groenkloof, 0181, South Africa. All rights reserved.

© SABS. This standard may reside on internal network or intranet on a Single Site only and is exclusively available to The Cosmetic, Toiletry and Fragrance Association in accordance with copyright exploitation agreement no. 014/009/26-295, valid until 2027-07-31. The single-site licence allows authorised staff member of The Cosmetic, Toiletry and Fragrance Association to make five (5) printouts of the standard and will expire after one year unless renewed. Unauthorised copying, sharing, distribution, or access outside approved networks is prohibited.

6.3.2.5 Procedure

a) **Toothpaste**. Pipette 50 mL each of the sample and the blank solutions (6.3.2.3(a)) into individual separating funnels and then proceed as in 6.3.2.4(a)(2).

b) **Paint**. Aspirate the sample and blank solutions (in turn) into the atomic absorption spectrophotometer, aspirating water between each determination.

6.3.2.6 Calculations

a) Determine the lead contents of the sample and blank solution by reference to the relevant standard curve.

b) Calculate the lead content of the sample as follows:

$$\text{Lead content, } \mu g/g = \frac{(A - B) \times D}{m}$$

where

A = concentration of lead in the sample solution (from the standard curve), µg/mL

B = concentration of lead in the blank solution, µg/mL

D = dilution of sample, mL

m = mass of sample taken, g

6.3.3 pH value

Use SANS 5011 to determine the pH value of a slurry consisting of one part by mass of the composite sample and four parts by mass of water.

6.3.4 Abrasiveness

6.3.4.1 Apparatus

a) **Brushing machine**. A brushing machine similar to that shown in figure 1 and figure 2 and having a 38 mm reciprocating stroke. It is so constructed that the tooth specimen can be withdrawn for irradiation.

b) **Brush**. A "hard" grade brush with nylon filament tufts of minimum tuft spacing, and constructed as follows:

Filament:	Diameter	approximately 0,30 mm
	Length	11-12 mm
	Trim	flat or serrated
Brush head:	Number of longitudinal rows of tufts	3 or 4
	Width	7,5-10,0 mm
	Length	31-38 mm

c) **Counter**. Counting equipment suitable for a source of β-radiation.

This product is copyrighted by SABS, 1 Dr Lategan Road, Groenkloof, 0181, South Africa. All rights reserved.

© SABS. This standard may reside on internal network or intranet on a Single Site only and is exclusively available to The Cosmetic, Toiletry and Fragrance Association in accordance with copyright exploitation agreement no. 014/009/26-295, valid until 2027-07-31. The single-site licence allows authorised staff member of The Cosmetic, Toiletry and Fragrance Association to make five (5) printouts of the standard and will expire after one year unless renewed. Unauthorised copying, sharing, distribution, or access outside approved networks is prohibited.

d) **Dental handpiece**. A straight dental handpiece that is capable of holding, in turn, a rotating brush and a diamond disc.

e) **Neutron flux source**. A source of neutron flux of 10^{12} neutrons/cm^2.s.

6.3.4.2 Reagents

a) **Bacteriostatic solution**. An aqueous solution containing 10 g of chlorhexidine gluconate per litre.

b) **Diluent**. An aqueous solution containing 5 g of sodium carboxymethyl cellulose per litre.

c) **Mounting medium**. A suitable epoxy resin which, after neutron irradiation, does not lead to a level of radio-activity that may interfere with the test.

d) **Reference paste**

1) **Composition**. As given in table 1.

Table 1 — Composition of reference paste

1	2
Constituent*	**g/kg**
Calcium carbonate, precipitated+	400,0
Glycerol	230,0
Sodium carboxymethyl cellulose	14,0
Dodecyl sodium sulphate	10,0
Sodium silicate	5,0
Saccharin sodium	1,5
Formalin (400 g/L formaldehyde)	1,0
Peppermint flavouring	8,0
Water	330,5

*Ensure that the constituents are suitable for dental use.

+That complies with the requirements in the current statutory formulary, and with the following additional requirements:

Apparent density (fluffed), g/cm^3	0,78-0,89
Flow point, mL water/15 g	13 ± 1
Fineness: % passing through a sieve of aperture size 75 µm, min.	99,7
Mean particle size, µm	12,5 ± 2,0

2) **Preparation**. Transfer the glycerol to a mixing bowl and, with slow stirring using a mechanical stirrer equipped with an anchorshaped blade, add the sodium carboxymethyl cellulose and then the saccharin sodium. To 50 mL of the required quantity of water in a beaker, add the sodium silicate and stir until dissolved. Add this to the mixture in the bowl, and then add 205,5 mL of the water. Transfer to a vacuum mixing vessel and stir at the slowest speed until the gel is smooth (about 15 min). Slowly add the calcium carbonate to the gel. When all the powder is mixed, stop the stirrer and scrape the blade and the inside of the mixing vessel. Restart the stirrer and stir at the slowest speed, under reduced pressure, until the mixture is smooth (about 10 min).

This product is copyrighted by SABS, 1 Dr Lategan Road, Groenkloof, 0181, South Africa. All rights reserved.

© SABS. This standard may reside on internal network or intranet on a Single Site only and is exclusively available to The Cosmetic, Toiletry and Fragrance Association in accordance with copyright exploitation agreement no. 014/009/26-295, valid until 2027-07-31. The single-site licence allows authorised staff member of The Cosmetic, Toiletry and Fragrance Association to make five (5) printouts of the standard and will expire after one year unless renewed. Unauthorised copying, sharing, distribution, or access outside approved networks is prohibited.

Place the dodecyl sodium sulphate in a beaker, add the remaining 75 mL of water (warmed to about 45 °C) and stir until dissolved. Add this to the mixture in the vessel, with slow stirring for 3 min under reduced pressure.

Add the formalin and then the peppermint flavouring and stir for 2 min under reduced pressure.

6.3.4.3 Tooth specimens

a) **Type**

The specimens shall be cut from permanent teeth of humans, preferably in the age group 25-40 years. The teeth shall have been stored since extraction in the bacteriostatic solution. The teeth preferred for testing the enamel and dentine are incisors and single-rooted teeth respectively. An alternative source of dentine tissue is the root of a large maxillary incisor.

b) **Preparation**

1) **General**. Thoroughly clean the teeth with a dental polishing paste on a rotating brush held in a straight dental handpiece. Remove all traces of the paste by washing the teeth thoroughly, and cut each specimen under a jet of water, using a diamond disc rotating in the dental handpiece.

2) **Enamel specimens** (see figure 3). Cut off the root and mount (see (c)(1) below) the crown (or a slice of the crown) in the mounting medium with the labial or buccal surface uppermost and in an orientation that will give crosswise brushing in the brushing machine. Ensure that each specimen protrudes above the surface of the resin by about 2 mm, and that the exposed surface has an area of about 50 mm^2 and is intact and undamaged.

3) **Dentine specimens** (see figure 4). Cut off the crown and root (or, when relevant, the lower end of the root), remove the enamel, and mount in the mounting medium. Ensure that each specimen protrudes above the surface of the resin by 1-2 mm, and that the exposed surface has an area of at least 50 mm^2 and is intact and undamaged.

c) **Mounting**

1) Dry each specimen, without heating, before mounting it. Cast the tapered holder and mount the specimen in one operation using a suitable moulding block (see figure 5). Use polyvinyl alcohol as the release reagent.

2) After about 12 h remove the mounted specimen from the mould and store in the bacteriostatic solution in a suitable container. Immediately before irradiation thoroughly brush the specimen with toothpaste, wash in water, place in a polyethylene capsule filled with freshly prepared bacteriostatic solution and seal the capsule. Ensure that a specimen is at no time allowed to dry out except for the short period required immediately before the mounting process. Avoid handling the specimens and do not use detergents for cleaning to limit the formation of ^{24}Na during irradiation.

6.3.4.4 Procedure

a) **Irradiation of tooth specimen**. Irradiate the capsule containing the specimen and bacteriostatic solution for 3-5 h in a neutron flux not exceeding 10^{12} neutrons/cm^2.s so that the final specific activity is about $3{,}7 \times 10^7$ Bq. 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 higher than 65 °C.

After irradiation, wait 4 d before testing to allow any highly radioactive elements to decay.

This product is copyrighted by SABS, 1 Dr Lategan Road, Groenkloof, 0181, South Africa. All rights reserved.

© SABS. This standard may reside on internal network or intranet on a Single Site only and is exclusively available to The Cosmetic, Toiletry and Fragrance Association in accordance with copyright exploitation agreement no. 014/009/26-295, valid until 2027-07-31. The single-site licence allows authorised staff member of The Cosmetic, Toiletry and Fragrance Association to make five (5) printouts of the standard and will expire after one year unless renewed. Unauthorised copying, sharing, distribution, or access outside approved networks is prohibited.

Handle the irradiated specimen in accordance with normal practice for a source of the above strength and type. Use tongs (of length exceeding 250 mm) to handle the specimen, and wear suitable goggles.

b) **Conditioning of tooth specimen**. Transfer the specimen to the trough of the brushing machine and condition the surface by brushing, for 1 000 double brush-strokes in the case of a dentine specimen, and 10 000 double brush-strokes in the case of an enamel specimen, in a slurry of the reference paste (20 g of reference paste plus 70 g of the diluent). Determine the volume of this slurry before adding it to the trough as this is required later for correction purposes. At the end of the conditioning period pour the slurry into a container and thoroughly wash the brush and specimen in water.

c) **Testing**

1) Dry the trough containing the tooth specimen with a paper tissue, add 30 g of a reference paste slurry containing 20 g of reference paste and 10 g of the diluent. Ensure that the specimen is adequately covered, and brush the specimen for 500 double strokes in the case of a dentine specimen, and 2 000 double strokes in the case of an enamel specimen. Add 15 mL of the diluent to the trough, mix thoroughly with a rubber-capped glass rod, and brush for a further 500 or 2 000 double strokes as relevant. Repeat this procedure for a further three additions of 15 mL of the diluent. (At the completion of the above procedure the dentine and enamel specimens will have been brushed for totals of 2 500 and 10 000 double strokes respectively.)

2) Pour the active slurry from the trough and break any foam present with a drop of diethyl ether. After gentle stirring, withdraw two 1 mL samples of the slurry using a pipette fitted with a micro-filter and transfer each sample to a suitable container. Dry the contents under an infra-red lamp or in an oven.

3) After carrying out the conditioning procedure given in (b) above and the test procedure given in (c)(1) and (2) above using the reference paste, test the composite sample in the same way, and then carry out another control test with the reference paste. Do not use any dentine specimen for more than 50 000 double brush-strokes; an enamel specimen may be used for as long as it remains free from exposure of dentine.

d) **Counting**. Determine the radioactivity of the slurry samples with a counter suitable for β-rays of 1,7 meV. Correct the count rate for a total of 5 000 counts for "dead time" and background errors and calculate the mean. If their individual values differ by more than 5 % from the mean, repeat the test. Determine the levels of radioactivity of the samples within a short period of one another so that corrections for activity decay are reduced to low values. Make any corrections that are necessary on the basis of a half life of 14,3 d(^{32}p).

Corrections for β-absorption and back-scatter can be neglected when the above counter geometry is used.

A small correction is necessary for the slightly different ratios of sample to total volumes of the paste slurry used in the different tests. The correction factor can be calculated from the measured volumes of dentifrice slurry used in the conditioning runs.

e) **Calculation**. The ratio of the abrasiveness of the test paste to the reference paste is given by the corrected ratio of the count rates.

6.3.5 Extrusion test

Maintain the tubes of toothpaste (6.2.1) at 10 ± 1 °C for at least 2 h and then test each tube as follows:

This product is copyrighted by SABS, 1 Dr Lategan Road, Groenkloof, 0181, South Africa. All rights reserved.

© SABS. This standard may reside on internal network or intranet on a Single Site only and is exclusively available to The Cosmetic, Toiletry and Fragrance Association in accordance with copyright exploitation agreement no. 014/009/26-295, valid until 2027-07-31. The single-site licence allows authorised staff member of The Cosmetic, Toiletry and Fragrance Association to make five (5) printouts of the standard and will expire after one year unless renewed. Unauthorised copying, sharing, distribution, or access outside approved networks is prohibited.

Remove the cap and invert the tube over a plastics measuring cylinder of suitable volume. By gradually rolling the base of the tube tightly downwards, extrude the contents into the measuring cylinder. Measure the volume of toothpaste extruded, and record the result as a percentage of the nominal volume of the contents.

6.3.6 Stability

Transfer a suitable quantity of the composite sample into each of four glass test tubes, stopper them, and proceed as follows:

a) Heat two of the test tubes for 72 h in an oven maintained at 45 ± 1 °C. Allow to cool and examine the contents visually for homogeneity and for signs of fermentation or other deterioration (or both).

b) Cool the other two test tubes to -5 ± 1 °C, maintain at this temperature for 1 h, allow to attain room temperature, and examine the contents visually for homogeneity.

6.3.7 Determination of fluoride ion content

6.3.7.1 Apparatus

a) **Gas chromatograph**. A suitable gas chromatograph equipped with a flame ionization detector, and having a column of length about 1,5 m and nominal outside diameter 6 mm packed with 10 % methyl silicone (100 %) on 80-100 mesh flux calcined diatomite, acid washed.

b) **Gas chromatograph operating conditions**

1) **Temperatures**

Detector	100 °C
Injection	80 °C
Oven	50 °C

2) **Carrier gas (nitrogen) flow rate**, 20 mL/min

c) **Microsyringe**, 1 µL.

6.3.7.2 Reagents

NOTE Unless inconsistent with the text, use only reagents of analytical reagent grade and distilled water.

a) **Hydrochloric acid**, concentrated.

b) **Nitrogen**. A source of pure nitrogen gas.

c) **n-Pentane solution in benzene**. 1,6 g of n-pentane made up to 1 L with benzene.

d) **Standard fluoride solution**. An aqueous solution of sodium fluoride (NaF) containing 1,0 mg of F^-/mL.

e) **Trimethylchlorosilane**.

6.3.7.3 Determination of the response factor of the apparatus

a) Pipette 5 mL of the standard fluoride solution into a 50 mL plastics flask. Using a measuring cylinder, add 2 mL of the hydrochloric acid, stopper the flask, and mix the contents. Add from a burette 2,0 mL of the trimethylchlorosilane.

This product is copyrighted by SABS, 1 Dr Lategan Road, Groenkloof, 0181, South Africa. All rights reserved.

© SABS. This standard may reside on internal network or intranet on a Single Site only and is exclusively available to The Cosmetic, Toiletry and Fragrance Association in accordance with copyright exploitation agreement no. 014/009/26-295, valid until 2027-07-31. The single-site licence allows authorised staff member of The Cosmetic, Toiletry and Fragrance Association to make five (5) printouts of the standard and will expire after one year unless renewed. Unauthorised copying, sharing, distribution, or access outside approved networks is prohibited.

b) Securely stopper the flask, mix well by shaking and allow to stand for 15 min to cool. Using a safety pipette, pipette 10 mL of the benzene solution of n-pentane into the flask, and stopper. Mix thoroughly by shaking vigorously for 2 min. Transfer the contents of the flask to a centrifuge tube, stopper, and centrifuge at 2 500 r.p.m. for 5 min. Inject 1,0 µL of the upper layer onto the column and allow the chromatogram to develop.

c) Draw tangent base lines to the peaks of interest, and measure the heights of the trimethylfluorosilane (hf) and n-pentane (hp) peaks. (Alternatively determine the areas of the relevant peaks.)

d) Calculate the response factor (k) as follows:

$$k = 3{,}2 \times \frac{hf}{hp}$$

6.3.7.4 Procedure

Weigh out accurately about 3,5 g of the composite sample into a centrifuge tube. Add 10 mL of water and 4-6 small glass beads. Stopper and shake vigorously until the paste is dispersed and removed from the sides of the tube. Centrifuge at 2 000 r.p.m. for 10 min.

Decant the upper aqueous layer into a 50 mL plastics flask. Redisperse the residue in the tube with 5 mL of water and centrifuge. Decant the aqueous layer into the plastics flask.

Cautiously add 5 mL of the hydrochloric acid to the residue in the centrifuge tube. When any effervescence has subsided, stopper and shake vigorously to disperse. Centrifuge and decant the solution into the plastics flask. Redisperse the residue (if any) and wash the tube with 3 mL of the hydrochloric acid. Centrifuge and decant into the plastics flask. Wash the tube and residue (if any) with 5 mL water, centrifuge, and decant into the plastics flask. Wash the tube and residue (if any) with a further 5 mL of water, centrifuge if necessary, and add the washings to the plastics flask.

Stopper the plastics flask, shake to mix, then heat in a boiling water bath for 1 min. Cool to room temperature in a cold water bath.

Add to the contents of the flask from a burette 2,0 mL of the trimethylchlorosilane. Stopper, shake well, and allow to stand for 15 min. Using a safety pipette, pipette 10 mL of the benzene solution of n-pentane into the flask. Shake vigorously for at least 2 min.

Pour the contents of the flask into a centrifuge tube, stopper, and centrifuge at 2 500 r.p.m. for 10 min.

NOTE Some pastes develop emulsions that require at least one decanting and a second centrifuging in order to separate the organic layer.

Inject 1 µL of the organic phase onto the column and allow the chromatogram to develop, then proceed as in 6.3.7.3(c).

6.3.7.5 Calculations

Calculate the total fluoride content as follows:

$$\text{Total fluoride content, as } F^-, \% \text{ (by mass)} = \frac{1{,}6 \times h'f}{m \times h'p \times k}$$

where

This product is copyrighted by SABS, 1 Dr Lategan Road, Groenkloof, 0181, South Africa. All rights reserved.

© SABS. This standard may reside on internal network or intranet on a Single Site only and is exclusively available to The Cosmetic, Toiletry and Fragrance Association in accordance with copyright exploitation agreement no. 014/009/26-295, valid until 2027-07-31. The single-site licence allows authorised staff member of The Cosmetic, Toiletry and Fragrance Association to make five (5) printouts of the standard and will expire after one year unless renewed. Unauthorised copying, sharing, distribution, or access outside approved networks is prohibited.

h'f = height of trimethylfluorosilane peak

h'p = height of n-pentane peak

m = mass of sample used, g

k = response factor (from 6.3.7.3(d))

6.3.8 Test for contamination and attack on tube and cap

Heat the tubes of toothpaste (6.2.1) at 45 ± 1 °C for 10 d. Extrude a portion of the paste and examine it for visible contamination. Slit the tube, remove the paste, and examine the surfaces of the tube and cap for signs of corrosion, chemical attack, or other damage. Disregard surface staining by colouring agents in the toothpaste.

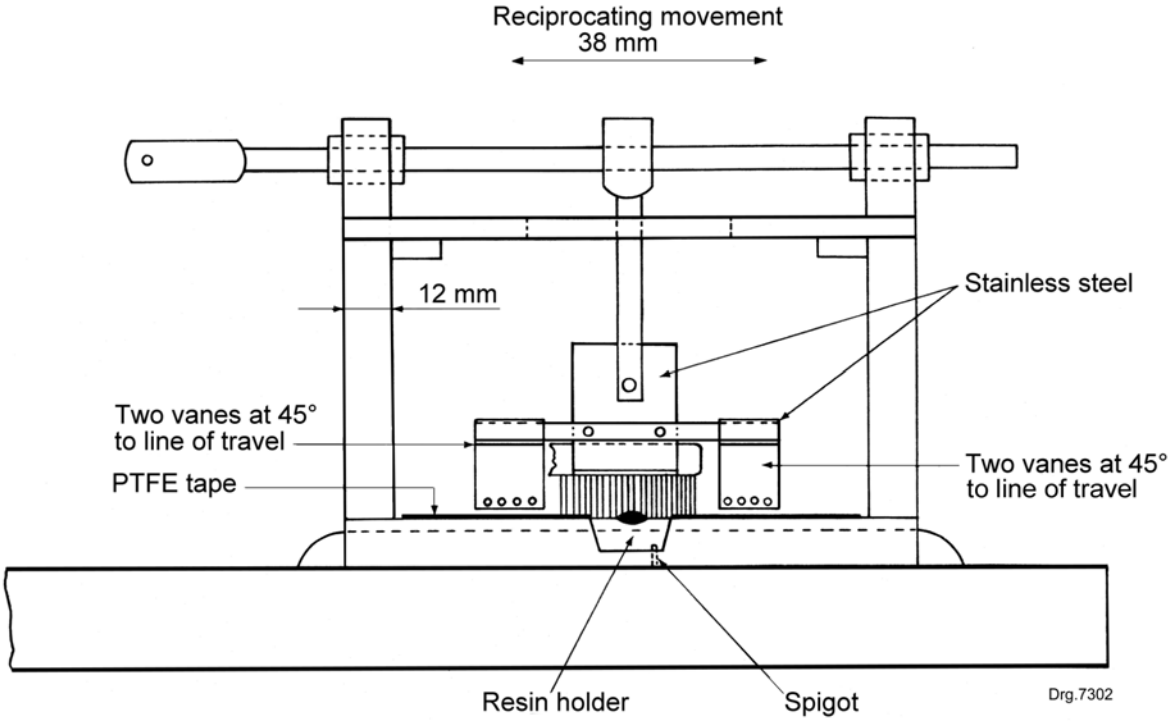


Figure 1 — Toothpaste testing rig

This product is copyrighted by SABS, 1 Dr Lategan Road, Groenkloof, 0181, South Africa. All rights reserved.

© SABS. This standard may reside on internal network or intranet on a Single Site only and is exclusively available to The Cosmetic, Toiletry and Fragrance Association in accordance with copyright exploitation agreement no. 014/009/26-295, valid until 2027-07-31. The single-site licence allows authorised staff member of The Cosmetic, Toiletry and Fragrance Association to make five (5) printouts of the standard and will expire after one year unless renewed. Unauthorised copying, sharing, distribution, or access outside approved networks is prohibited.

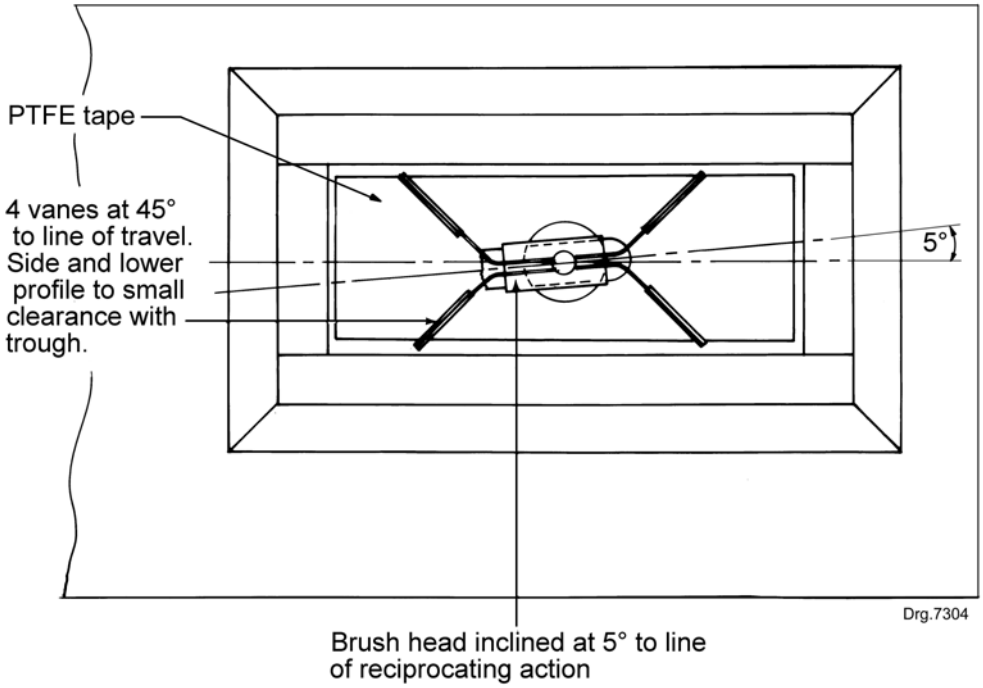


Figure 2 — Brushing head with stirring vanes

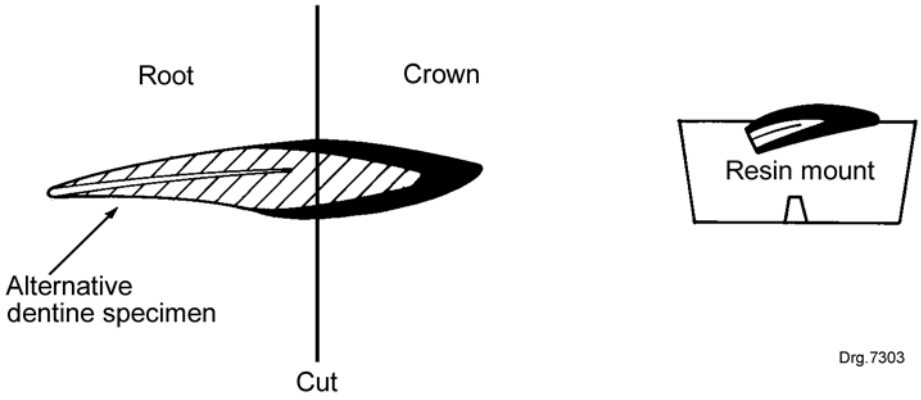


Figure 3 — Preparation of enamel specimen

This product is copyrighted by SABS, 1 Dr Lategan Road, Groenkloof, 0181, South Africa. All rights reserved.

© SABS. This standard may reside on internal network or intranet on a Single Site only and is exclusively available to The Cosmetic, Toiletry and Fragrance Association in accordance with copyright exploitation agreement no. 014/009/26-295, valid until 2027-07-31. The single-site licence allows authorised staff member of The Cosmetic, Toiletry and Fragrance Association to make five (5) printouts of the standard and will expire after one year unless renewed. Unauthorised copying, sharing, distribution, or access outside approved networks is prohibited.

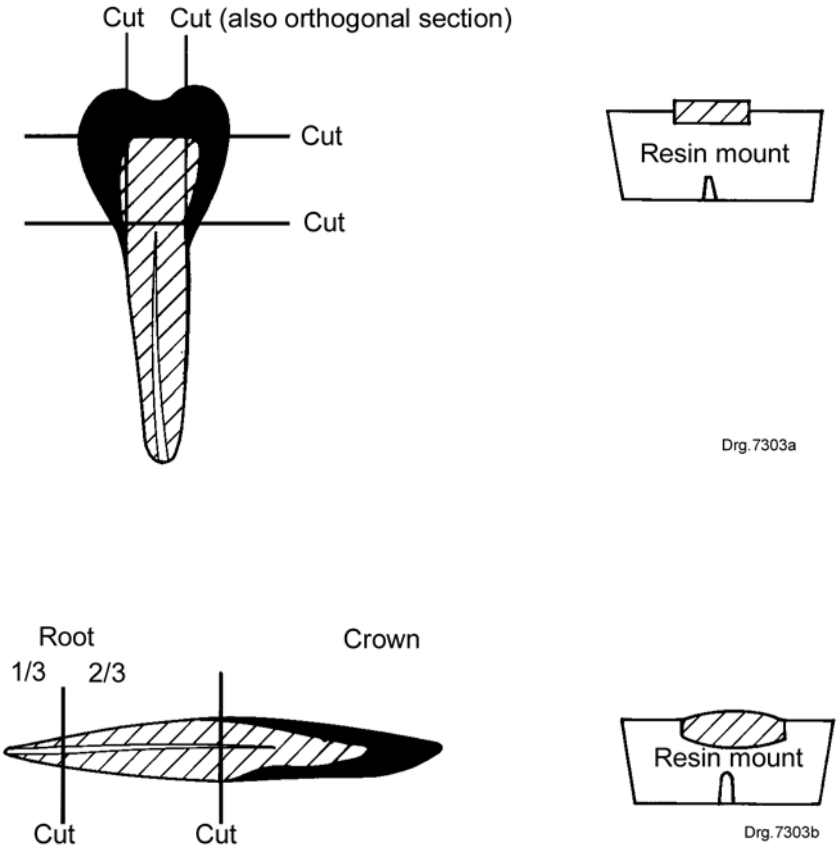


Figure 4 — Preparation of dentine specimen

This product is copyrighted by SABS, 1 Dr Lategan Road, Groenkloof, 0181, South Africa. All rights reserved.

© SABS. This standard may reside on internal network or intranet on a Single Site only and is exclusively available to The Cosmetic, Toiletry and Fragrance Association in accordance with copyright exploitation agreement no. 014/009/26-295, valid until 2027-07-31. The single-site licence allows authorised staff member of The Cosmetic, Toiletry and Fragrance Association to make five (5) printouts of the standard and will expire after one year unless renewed. Unauthorised copying, sharing, distribution, or access outside approved networks is prohibited.

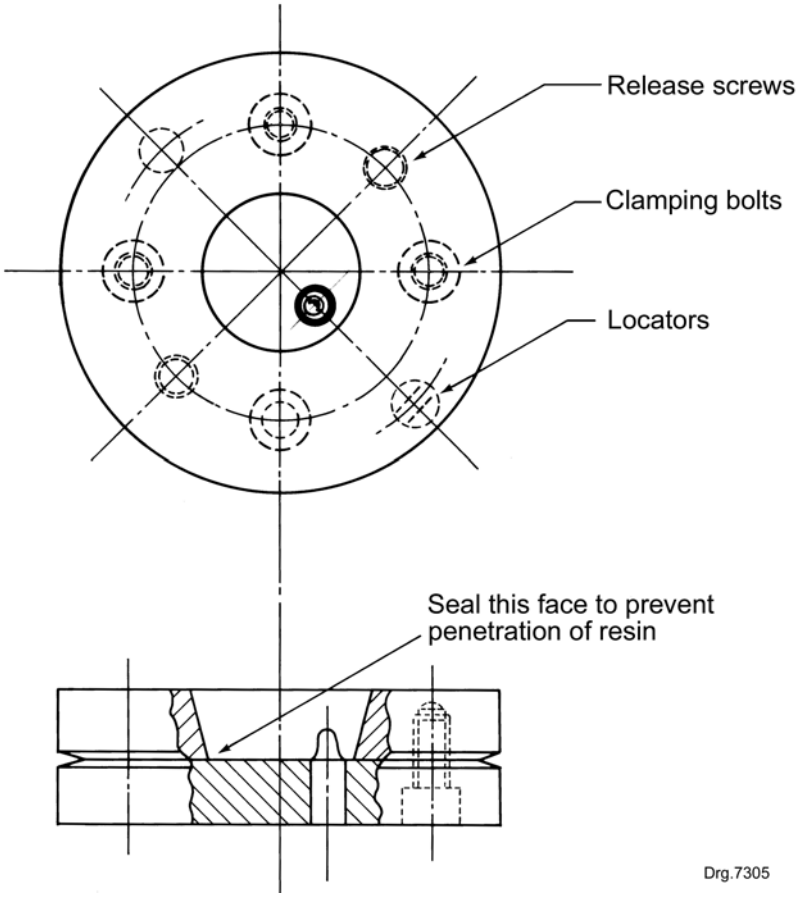


Figure 5 — General arrangement of mould for tooth mount

This product is copyrighted by SABS, 1 Dr Lategan Road, Groenkloof, 0181, South Africa. All rights reserved.

© SABS. This standard may reside on internal network or intranet on a Single Site only and is exclusively available to The Cosmetic, Toiletry and Fragrance Association in accordance with copyright exploitation agreement no. 014/009/26-295, valid until 2027-07-31. The single-site licence allows authorised staff member of The Cosmetic, Toiletry and Fragrance Association to make five (5) printouts of the standard and will expire after one year unless renewed. Unauthorised copying, sharing, distribution, or access outside approved networks is prohibited.

Appendix A[1)]

Applicable standards

Reference is made to the latest issue of the following standard:

SANS 5011/ISO 10523, *Water quality — Determination of pH*.

Appendix B

Notes to purchasers

The following requirements must be specified by the purchaser in tender invitations and in each order or contract:

a) The type (see 3.1.1)

b) The nominal volume or total nominal volume (as relevant) of the contents (see 4.1.1)

1) The information in the appendices is additional to that in the specification and has been included purely for information and guidance.

© Standards South Africa

This product is copyrighted by SABS, 1 Dr Lategan Road, Groenkloof, 0181, South Africa. All rights reserved.