

Specification for Toothpastes

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Cosmetic, Toiletry and Perfumery Association Limited
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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 antimony 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.

A British Standard does not purport to include all the necessary provisions of a contract. Users of British Standards are responsible for their correct application.

Compliance with a British Standard does not of itself confer immunity from legal obligations.

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.

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: $2 \times$ the abrasivity of the standard reference paste;

for enamel: $4 \times$ 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: $2 \times$ the abrasivity of the standard reference paste;

for enamel: $4 \times$ 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: $1.5 \times$ the abrasivity of the standard reference paste;

for enamel: $3 \times$ 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 (isobutyl 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