Analytical cosmetics

BIS specification and analytical methods for skincream and toothpaste.

IS 6608: 2004 *Indian standards Skin Cream Specifications*

This standard prescribes the requirements and the methods of Sampling and test for skin creams

IS No.	*Title*
265 : 1993	Hydrochloric acid (*fourth revision*)
1070 : 1992	Water reagent grade (*third revision*)
2088 : 1983	Methods for determination of arsenic (*second revision*)
3958 : 1984	Methods of sampling cosmetics (*first revision*)
4011 : 1997	Methods of test for safety evaluation of cosmetics (*second revision*)
4707	Classification of cosmetic raw materials and adjuncts:
(Part 1) : 2001	Dyes, colours and pigments (*second revision*)
(Part 2) : 2001	List of raw materials generally not recognized as safe for use in cosmetics (*second revision*)
14648 : 1999	Methods of test for microbiological examinations of cosmetics

3. REQUIREMENTS

3.1 Description

The skin cream shall be in the form of a thick emulsion or unctuous mass with a pleasant odour. It shall be white or pigmented or of uniform colour.

3.2 Ingredients

Unless specified otherwise, all the raw materials used in the manufacture of skin creams shall conform to the requirements prescribed in the relevant Indian Standards where such standards exist.

3.2.1 The dyes, colours (pigments, lakes etc) if used in the manufacture of skin creams shall comply with 1S 4707 (Part 1) subject to the provision of schedule Q of *Drugs and Cosmetic Act,* issued by the Government of India.

3.2.2 Other ingredients shall comply with the provisions of IS 4707 (Part 2).

3.3 The material shall also comply with the requirements given in Table 1 when tested as prescribed in COI4 of the Table 1.

Requirements for Skin Creams

Table 1 Requirements for Skin Creams

(*Clause* 3.3)

Sl No.	Characteristics	Requirement	Method of Test, Ref to	
			Annex	IS No.
(1)	(2)	(3)	(4)	(5)
i)	Thermal stability	To pass the test	A	
ii)	*p*H[1]	4.0 to 9.0	B	
iii)	Total Fatty substance content, percent by mass, *Min*	5.0	C	
iv)	Total residue, percent by mass, *Min*	10	D	
v)	Heavy metals[2] (as Pb), parts per million, *Max*	20	E	
vi)	Arsenic[2] (as As_2O_3), parts per million, *Max*	2	F	
vii)	Microbial content/limit			
	a) Total viable count cfu/g	Not more than 1 000		14648
	b) Gram Negative pathogens	Less than 10		14648

[1] For creams based on beeswax and borax, the *p*H shall be between 5.0-10.0

[2] If all the raw materials requiring test for heavy metals and arsenic have been so tested and comply with the requirements, then the manufacturer may not test the finished cosmetic for heavy metals and arsenic.

4.2 Specific Requirements

4. ADDITIONAL REQUIREMENTS FOR ECO MARK (OPTIONAL)

4.1 Requirements for quality, safety and performance prescribed under 4.1.1 to 4.1.4.

4.1.1 All the ingredients that go into formulation of cosmetics shall comply with the provisions of IS 4707 (Part 1) and 1S 4707 (Part 2). The product shall also meet specific requirements as given in the standard.

4.1.2 The product package shall display a list of key ingredients in descending order of quantity present.

4.1.3 The product shall not be manufactured from any carcinogenic ingredients.

4.1.4 The manufacturer shall produce to BIS environmental consent clearance from the concerned State Pollution Control Board as per the provisions of the *Water (Prevention and Control of Pollution) Cess Act 1977* and the *Air (Prevention and Control Pollution) Act, 1981* along with the authorization, if required under the *Environment (Protection) Act, 1986* and the Rules made there under, while applying for ECO Mark. Additionally, provisions of the *Drugs and Cosmetics Act, 1940* and the Rules thereunder shall also be complied with.

4.2 Specific Requirements

4.2.1 Product shall be dermatologically safe when tested as per IS4011,

4.2.2 Heavy metals calculated as lead (Pb) and arsenic (as AS20J shall not exceed 20 and 2 ppm, respectively when tested by the respective method prescribed in Indian Standards.

5 PACKING AND MARKING

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in suitable well-closed containers.

5.2 Marking

The containers shall be legibly marked with the following information:

a) **Name of the material;**

b) **Manufacturer's name and/or his recognized trade-mark, if arty;**

c) **Net mass of the material;**

d) **Month and year of manufacturing packing;**

e) **Batch or lot number, in code or otherwise;**

f) **Expiry date or "Best use before. . ." (month and year to be declared by the manufacturer);**

g) **List of key ingredients; and NOTE — This is exempted in case of pack sizes of 30 g/60 ml or less.**

h) **Any other information required by statutory authorities.**

6. SAMPLING

6.1 Representative samples of the material shall be drawn as prescribed in IS 3958.

6.2 Tests for all the characteristics shall be carried out on the composite sample as per methods referred under CO14 and 5 of Table 1.

6.3 The material shall be taken to have conformed to the standard if the composite sample passes all the tests.

7. QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (see IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

Annex	Test	Principle
ANNEX A	**TEST FOR THERMAL STABILITY**	A humidity chamber/incubator controlled at 60 to 70 percent relative humidity and 45 + 1degreeCelcius. The sample shall be taken to have passed the test, if on removal from the incubator shows **no oil separation or any other phase separation.**
ANNEX B	**DETERMINATION OF pH**	B-1 **APPARATUS** A pH meter, preferably equipped with a glass electrode.
ANNEX C	**DETERMINATION OF TOTAL FATTY SUBSTANCE CONTENT**	**C-O PRINCIPLE OF THE METHOD** The emulsion is broken with dilute mineral acid and the fatty matter is extracted with petroleum ether. It is weighed after removal of the solvent.

Annex	Test	Principle
ANNEX D	**DETERMINATION OF RESIDUE**	Weigh accurately about 5 g of the material in a weighed, clean and dry squat form weighing bottle and dry to constant mass at 105 + 1“C. Cool in a desiccator and weigh.
ANNEX E	**TEST FOR HEAVY METALS**	**E-1 OUTLINE OF THE METHOD** The colour produced with hydrogen sulphide solution is matched against that obtained with standard lead solution.
ANNEX F	**DETERMINATION OF ARSENIC**	**F-1 OUTLINE OF THE METHOD** Arsenic present in a solution of the material is reduced to arsine, which is made to react with mercuric bromide paper. The stain produced is compared with a standard stain.

BIS Specification for Toothpaste

TOOTHPASTE-SPECIFICATION

IS 6356:2001

This standard prescribes the requirements and the methods of sampling and test for toothpaste.

3.1 Dentifrice

A dentifrice is any substance or combination of substances specially prepared for the public for cleaning the accessible surfaces of teeth.

3.2 Toothpaste

A toothpaste is defined as a dentifrice in the form of a smooth, semisolid, homogeneous mass containing acceptable ingredients such as abrasives/polishing agents, surface active agents, humectants, binding agent, and other appropriate substances for oral health maintenance. The product can be opaque, transparent, or combination thereof, colored or white, packed in a suitable container from which it can be extruded in the form of a continuous mass.

TOOTHPASTE-SPECIFICATION

4. TYPES

The toothpaste shall be of either Type 1 or Type 2:

Type 1— Non-Fluoridated

Type 2 — Fluoridated

TOOTHPASTE SPECIFICATION

5.1 Composition

A toothpaste shall not contain mono or disaccharides, for example, sucrose or other readily fermentable carbohydrates. All the raw materials used shall conform to respective Indian Standards wherever they exist.

5.2 Dispensing

The paste shall extrude from the collapsible tube or any other suitable container in which it is packed, at 27+ 2°C in the form of **continuous mass with the application of normal force, without the application of excessive force which would cause injury to the tube or the container. It shall be possible to extrude bulk of the contents from the container or the tube starting from the crimped end of the tube by rolling the tube gradually.**

5.3 Stability

The toothpaste shall not show any physical sign of deterioration during normal conditions of storage and use. When subjected to a temperature of 45 ±2°C for a period of 28 days the toothpaste shall meet the requirements of the standard.

5.4 **Packaging Material Inertness**

The collapsible tubes or any other suitable container used for packaging of toothpaste shall not corrode, deteriorate or cause contamination of the toothpaste during normal condition of storage and use. There should be no sign of corrosion, chemical attack or other damage

TOOTHPASTE SPECIFICATION

5.5 Acceptance Test

The toothpaste shall also comply with the requirements given in Table 1, when tested according to the methods given in Annex B to Annex G, as per reference made in Contents of Table 1.

5.6 Shelf Life

Shelf life shall be declared by the manufacturer for all types of toothpaste. **Manufacturing date (month and year) should be mentioned on tube and carton. The expiry date or 'Best use before' shall be mentioned on the tube and carton**. During the shelf life the product will meet the requirement of the standard.

5.7 Additional Requirements for ECO-Mark (Optional)

5.7.1 ***General Requirements***

5.7.1.1 The product shall conform to the requirements for quality, safety and performance prescribed under 5.7.1.2 to 5.7.1.5.

5.7.1.2 All the ingredients that go into formulation of cosmetics shall comply with the provisions of IS 4707 (Part 1) and IS 4707 (Part 2). The product shall also meet specific requirements as given in the standard.

5.7.1.3 The product package shall display a list of key ingredients in descending order of quantity present.

5.7.1.4 The product shall not be manufactured from any carcinogenic ingredients.

5.7.1.5 The manufacturer shall produce to BIS environmental consent clearance from the concerned State Pollution Control Board as per the provisions of the ***Water (Prevention and Control of Pollution) Cess Act, 1977*** and the ***Air (Prevention and Control Pollution) Act,*** 1981 along with the authorization, if required under the ***Environment (Protection) Act,*** 1986 and the Rules made thereunder, while applying for ECO-Mark. Additionally, provisions oftbe ***Drugs and Cosmetics Act,*** 1940 and the Rules thereunder shall also be complied with.

5.7.2 ***Specific Requirements***

Heavy metals calculated as lead (Pb) and arsenic (As20J) shall not exceed 20 and 2 ppm, respectively when tested by the respective method prescribed in Indian Standards.

6. ABRASIVITY (TYPE TEST)

The toothpaste shall not exceed the limits of dentin abrasivity that of 2.5 times when tested as per the procedure given in Annex H.

It may be noted that type test is recommended to be done on the formulation only once to pass the above criterion. This test need not be done for each and every batch. However, the type test is a must again if the abrasive system is changed in the formulation. It is not required for the new formulation as long as abrasive components are not changed.

7 PACKING AND MARKING

7.1 Packing

Toothpaste shall be packed in collapsible tubes or in any other suitable containers like sachets, pumps or other suitable dispensing systems. When packed in containers, the containers shall be properly sealed and have a leak-proof cap or closure. The containers, if necessary, may further be packed in cartons or any other suitable packaging material.

7.1.1 The material for product packaging shall meet the parameters evolved under the scheme of labelling environment friendly packaging/packaging materials.

7.2 Marking

The tubes and the cartons shall be legibly marked with the following information:

a) Name and type of toothpaste;

b) Name and address of the manufacturer;

c) Net mass or volume of the material in the tube;

d) Batch number, in code or otherwise;

e) Month and year of manufacture;

f) Fluoride ion content in ppm for Type 2 toothpaste;

g) Expiry date or 'Best use before. . ..' (month and year to be declared by the manufacturer);

h) Foaming/non-foaming; and

j) List of key ingredients.

NOTE — This is exempted in case of pack sizes of 30g/60 ml or less

7.2.1 *BIS Certification Marking*

7.2.1.1 The use of the Standard Mark is governed by the provisions of the ***Bureau of Indian Standards Act,*** 1986 and the Rules and Regulations made thereunder. The details of conditions under which the licence for the use of the Standard Mark may be granted to manufacturers or producers maybe obtained from the Bureau of Indian Standards.

7.2.1.2 If the product is covered under ECO-Mark (optional), it shall be suitably marked with ECO-Mark logo besides Standard Mark. The label may clearly specify that ECO-Mark is applicable to the contents or the package or both, as the case may be. If the product package is not separately covered under ECOMark scheme, it shall be clearly mentioned on the product that ECO-Mark label is applicable to contents only.

8 SAMPLING

8.1 Representative test samples of the material shall be drawn as prescribed in IS 3958.

8.2 Number of Tests and Criteria for Conformity

The tests for abrasivity, stability and container's inertness shall be type tests and shall be performed for product approval whereas tests for dispensing, fineness, pH, heavy metals, arsenic, foaming power, fluoride content and microbial counts shall be carried out on each batch for acceptance of the product.

9. QUALITY OF REAGENT

Unless specified otherwise, pure chemicals and distilled water (see IS 1070) shall be employed in tests.

NOTE — ‘Pure chemicals’ shall mean chemicats that do not contain impurities, which affect the results of anatysis.

Table 1 Requirements for Toothpaste

(*Clause* 5.5)

Sl No.	Characteristic	Requirement for		Method of Test, Ref to Annex
		Non-fluoridated	Fluoridated	
(1)	(2)	(3)	(4)	(5)
i)	Fineness:			
	a) Particles retained on 150 micron IS Sieve, percent by mass, *Max*	10	1.0	B
	b) Particles retained on 75 micron IS Sieve, percent by mass, *Max*	2.5	2.5	—
ii)	*p*H of aqueous suspension	5.5 to 10.5	5.5 to 10.5	C
iii)	Heavy metals (as lead), parts per million, *Max*	20	20	D
iv)	Arsenic (as As_2O_3), parts per million, *Max*	2	2	E
v)	Foaming power, ml, *Min*[1)]	50	50	F
vi)	Available Fluoride ion, parts per million, *Max*	50	1 000	G
vii)	Microbial counts:			
	a) Total viable counts per gram, *Max*	1 000	1 000	IS 14648
	b) Gram negative pathogens per gram, *Max*	Absent	Absent	IS 14648

NOTE — If all the raw materials used in the toothpaste formulation have been tested for heavy metals and arsenic and comply with the requirement, then manufacturer may not test the finished cosmetic for heavy metals and arsenic.

[1)] Applicable to foaming toothpaste only.

Annexure	Annexure	Guidelines
Annexure A	**ANNEX A** **INGREDIENTS CONVENTIONALLY USED IN THE MANUFACTURE OF TOOTHPASTE**	**INGREDIENTS CONVENTIONALLY USED IN THE MANUFACTURE OF TOOTHPASTE** Raw material used in toothpaste formulation falls into the following categories: 1. Polishing agents 2. Surface active agents 3. Humectant 4. Binding agent 5. Others as per IS 4707 (Part 1) and IS 4707 (Part 2)
Annexure B	**ANNEX B** DETERMINATION OF FINENESS	B-1 OUTLINE OF THE METHOD Squeeze the toothpaste and feel the presence of the particles/agglomerates/granules. Subject the toothpaste suspension to an ultrasonic treatment and pass through fineness test. Ultrasonification loosens out the l agglomerates into the constituent materials.

Annexure	Annexure	Guidelines
Annexure C	**ANNEX C DETERMINATION OF pH**	pH is determined using pH meter.
Annexure D	**ANNEX D**	[Clause 5.5 and *Table 1, S1No. (iii)]* DETERMINATION OF HEAVY METALS D-1 OUTLINE OF THE METHOD The colour produced with thioacetamide reagent in test solution is matched against that obtained with standard lead solution
ANNEX E	DETERMINATION OF ARSENIC	
ANNEX F	DETERMINATION OF FOAMING POWER	**F-1 GENERAL** Strict attention shall be paid to all details of the procedure in order to ensure concordant results. Particular care should be taken to shake the cylinder exactly as described. **F-2 OUTLINE OF THE METHOD** A suspension of the material in water is taken in a graduated cylinder and given 12 shakes under prescribed conditions. The volume of the foam formed is observed after keeping the cylinder for 5 minutes.
ANNEX G		DETERMINATION G-1.1 Principle Water soluble species are converted to fluoride ion by acid hydrolysis. The fluoride ion activity is then determined potentiometrically with the help of fluoride ion sensitive electrode.

Annexure	Annexure	Guidelines
ANNEX H	**ANNEX H** *(Foreword and Clause 6)* **ABRASIVITY (RDA) MEASUREMENT TEST (HEFFERREN**	**H-1 SCOPE** This annex identifies the specific procedures for determination of dentifrice abrasivity using the ADA laboratory method
ANNEX J	**ANNEX J**	**(Clause 2)** **LIST OF ADJUNCT INDIAN STANDARDS**

BIS requirements of Toothpastes

SRIP EDU PHARMA

Characteristics	Requirements Non Fluoridated	Requirements Fluoridated
Fineness: • Particles retained on 150 micron IS sieve, percent by mass, Max • Particles retained on 75 micron IS sieve, percent by mass, Max	10 2.5	1.0 2.5
The pH of aqueous suspension should be	5.5 to 10.5	5.5 to 10.5

BIS requirements of Toothpastes

Characteristics	Requirements Non Fluoridated	Requirements Fluoridated
The maximum heavy metals (lead) permitted	20 ppm	20 ppm
The maximum arsenic (As_2O_3) permitted	2 ppm	2 ppm
Foaming power, ml, Min	50	50
Available fluoride ion, parts per million, Max	50	1000
Microbial counts: • Total viable counts per gram, Max • Gram negative pathogens per gram, Max	 1000 Absent	 1000 Absent