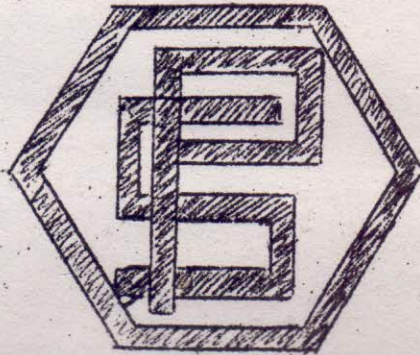


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PAKISTAN STANDARD 3973 1997.

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SPECIFICATION FOR *Skin powder*
SKIN POWDER



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PAKISTAN STANDARDS INSTITUTION
39, Garden Road, Saddar
KARACHI- 74400

AF-64/94

Price Rs.

PAKISTAN STANDARD SPECIFICATION
FOR

SKIN POWDER.

Cosmetic and Toilet Goods Sectional Committee

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PAKISTAN STANDARDS INSTITUTION

F O R
SKIN POWDER^S

0. FOREWORD

0.1 This Pakistan Standard was adopted by the Pakistan Standards Institution on 13th May 1997 after the draft finalized by the cosmetic & Toilet Goods Sectional Committee had been approved by the Chemical Divisional Council.

0.2 Two groups of Powders namely body powders and face powders are covered in this specification medicated powders for which therapeutic claims are made (for example, prickly heat powders) are not included in this specification.

0.3 This Standard is intended chiefly to cover the technical provisions relating to the supply of material and it does not include all necessary provisions of a contract.

0.4 For the purpose of deciding whether and particular requirements of this standard is complied with the final value, observed or calculated expressing the result of a test or analysis, shall be rounded off in accordance with PS: 103-1985 Rules rounding off numerical values. The number of significant places retained in the rounded off values should be same as that of the specified value in this standard.

1. SCOPE

1.1 This specification prescribes the requirements and methods of sampling and test for skin powders. It does not cover skin powder for infants.

2. TYPES

Skin powders shall be classified into two types as follows

a) Body powders:

These shall include talcum powders, toilet powders, and dusting powders;

b) Face powders:

These shall include loose powders and compressed powders.

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3. REQUIREMENTS

3.1 General Requirements

3.1.1 Body Powders

These shall consist principally of a finely powdered, free flowing, absorbent, innocuous material such as natural talc (hydrous silicate of magnesium with the formula $Mg_3 Si_4 O_{11} \cdot H_2O$); they shall be completely free from grit and may contain small amounts of perfume and coloring matter and other raw materials consistent with the accepted practice in the cosmetic industry. The letter may include materials having antiperspirant and deodorant properties.

3.1.2 Face Powders

These shall essentially be similar to body powder described under 3.1.1 except that they shall be of finer particle size.

3.1.3 The raw materials used shall be in such concentrations that they are free from harmful or toxic effects. Interaction of raw materials used in the formulation should not produce harmful or toxic effects.

3.1.4 It shall be the responsibility of the manufacturers of skin powders to satisfy themselves of the dermatological safety of a new formulation, or of a new material used in an old formulation before releasing the product for sale.

3.1.5 The material should not be the cause of bacterial and fungal contamination; this possibility may be obviated by, for instance, a process of sterilization.

3.2 Raw Materials

Unless specified otherwise, all the raw materials used in the manufacture of skin powder shall comply with the requirements prescribed in the ^{relevant} Pakistan Standards and where such standards do not exist, shall be dermatologically safe.

3.3 Freedom from Boric Acid

The material shall be free from boric acid when tested by the method prescribed in Appendix A.

3.4 Other Requirements

The material shall also comply with the requirements given in Table 1 when tested according to the relevant methods prescribed in Column 4 of the Table.

TABLE 1 - REQUIREMENTS FOR SKIN POWDERS

Sl. No.	CHARACTERISTIC	REQUIREMENTS FOR		Method of test Reference to Appendix
		Body Powder	Face Powder	
(1)	(2)	(3)	(4)	(5)
i.	Matter insoluble in boiling water, per cent by mass, min.	95.0	95.0	B
ii.	Fineness			
	a) Residue on 75-um sieve, per cent by mass, max.	1.5	0.5	C
	b) Residue on 150 um sieve per cent by mass, max.	0.1	0.1	
iii.	Moisture and volatile matter, per cent by mass, max.	2.0	3.0	D
iv.	pH of aqueous suspension	6.5-8	6.5-8	E
v.	Lead (as Pb), mg/kg, max	25	25	F
vi.	Arsenic (as As), mg/kg, max	0.5	0.5	G

4. PACKING

The material shall be packed in suitable, well closed containers. A number of these containers shall be enclosed in a package.

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5. MARKING

5.1 The containers shall be marked legibly and indelibly with the following information;

- a) Name and type of material;
- b) Name and address of the manufacturer;
- c) Registered trade mark if any;
- d) Net mass, in gram, of the material; and
- e) Batch or code number.

f) List of ingredients on the label of immediate container
g) Precautionary measures

6. SAMPLING

6.1 Representative samples of the material shall be drawn according to the relevant clauses

6.2 Number of tests

6.2.1 Each container selected shall be examined for packaging and marking requirements as prescribed in 1720 - 85

6.2.2 Test for detection of boric acid shall be carried out on each individual sample obtained as prescribed in 1720-85

6.2.3 Tests for requirements shall be conducted on the composite sample obtained as prescribed in 1720 - 85.

7. METHODS OF TEST

7.1 Tests shall be carried out as prescribed in Appendix A and the relevant appendices indicated in Column 5 of Table 1.

7.2 All weighings shall be carried out in closed vessels.

7.3 Unless specified otherwise, chemicals of analytical grade and distilled water of accepted purity shall be employed in tests.

8 CONFORMITY TO STANDARD

A lot shall be considered as conforming to the requirements of this specification if the following conditions are satisfied.

Contd.....P/5

- 8.1 Each container tested as in 6.2.1 satisfies the packaging and marking requirements.
- 8.2 Each individual sample tested as in 6.2.2 satisfies the relevant requirements.
- 8.3 The test results on the composite sample tested as in 6.2.3 satisfy the relevant requirements.

APPENDIX A

DETECTION OF BORIC ACID

A.1 PROCEDURE

Weigh to the nearest milligram about 1 g of the material and place it in a boiling tube (20 mm X 3 mm). Add about 2 ml of concentrated sulphuric acid and about 5 ml of methyl alcohol. Stopper the tube with a cork carrying two bent tubes as shown in Figure 1. Boil the contents in the test tube. Blow in air through one tube and light the vapours that come off the other bent tube (outlet tube). A green flame is obtained if boric acid is present.

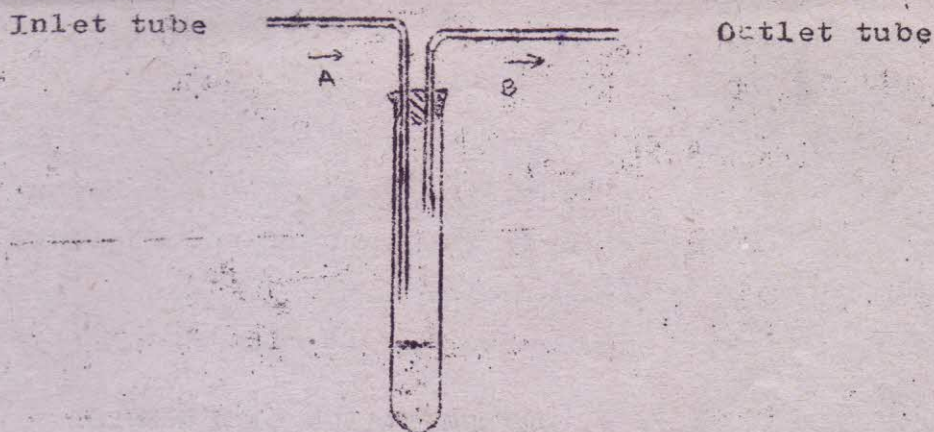


FIGURE 1 - Boiling tube arrangement for detection of boric acid.

APPENDIX B

DETERMINATION OF MATTER INSOLUBLE IN BOILING WATER

B.1 PROCEDURE

Weigh to the nearest milligram about 1 g of the material and transfer to a 500-ml beaker. If necessary, wet the material with a little rectified spirit. Add to the beaker about 200 ml of water and boil. Allow to settle and filter the supernatant liquid through a Gooch crucible. Wash the residue in the beaker with water and transfer completely to the filter. Dry the residue in the crucible at $105 \pm 2^\circ\text{C}$ cool in a desiccator and weigh. Repeat the heating, cooling and weighing operations until the difference in mass between two successive weighings does not exceed 5 mg.

B.2 CALCULATION

Matter insoluble in boiling water, per cent by mass

$$= \frac{m_1}{m_0} \times 100$$

where,

m_0 = mass, in gram, of the material taken for the test and
 m_1 = mass, in gram, of the residue.

APPENDIX C

DETERMINATION OF FINENESS

C.1 PROCEDURE

Place about 10 g of the material, weighed to the nearest 0.01 g, in a (a) 75-um sieve, (b) 150 um sieve, both sieve conforming to BS: _____ and wash by means of a slow stream of running tap water and finally with a fine stream from a wash bottle until all the material that can pass through the sieve has passed. In case the material is not easily

wetted by water, the washing could be started, with a slow stream of filtered denatured spirit.

Let the water drain from the sieve and then dry the sieve containing the residue on a steam bath. Carefully transfer the residue on to a tared watch glass and dry it at $105 \pm 2^\circ\text{C}$. Cool in a desiccator and weigh. Repeat the heating, cooling and weighing operations until the difference in mass between two successive weighings does not exceed 10 mg.

C.2 CALCULATION

Material retained on the sieve, per cent by mass = $\frac{m_1}{m_0} \times 100$

where,

m_0 = mass, in gram, of the material taken for the test; and

m_1 = mass, in gram of the residue retained on the sieve.

APPENDIX D

DETERMINATION OF MOISTURE AND VOLATILE MATTER

D.1 PROCEDURE

Weigh to the nearest milligram, about 5 g. of the material in a porcelain or glass dish, 60 mm to 80 mm in diameter and 20 mm to 40 mm in depth. Dry in an air oven at a temperature of $105 \pm 2^\circ\text{C}$. Cool in a desiccator and weigh. Repeat the heating, cooling and weighing operations until the difference in mass between two successive weighings does not exceed 5 mg.

D.2 CALCULATION

Moisture and volatile matter, per cent by mass = $\frac{m_1}{m_0} \times 100$

where,

m_0 = mass, in grams, of the material taken for the test;

m_1 = loss in mass, in grams, on drying.

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APPENDIX E

DETERMINATION OF pH OF AQUEOUS SUSPENSION

Take 10.0 ± 0.1 g of the material in a 150 ml beaker and add 90 ml of freshly boiled and cooled water. Stir well to make a thorough suspension. Using a pH meter, determine the pH of the suspension after 60 ± 5 s of making the suspension at ambient temperature.

APPENDIX F

DETERMINATION OF LEAD

F.1 APPARATUS

Nessler cylinders, 50 ml capacity.

F.2 REAGENTS

All reagents used shall be free from lead.

F.2.1 Ammonia solution, dilute

Dilute 375 ml of ammonia solution, containing not less than 27.0 per cent m/m and not more than 30.6 per cent m/m of ammonia, to 1 000 ml with water.

F.2.2 Ammonium chloride, containing not less than 99.5 per cent of ammonium chloride (calculated with reference to the dried substance).

F.2.3 Citric Acid

F.2.4 Hydrochloric Acid, 32 per cent m/m.

F.2.5 Lead solution, strong

Dissolve 0.16 g of lead nitrate in 5 ml of nitric acid and sufficient water to produce 100 ml of solution.

F.2.6 Lead solution, dilute

Dilute 1.0 ml strong lead solution (see F.2.5) with sufficient water to produce 100 ml. 1 ml contains 0.01 mg of lead. The solution must be freshly prepared.

F.2.7 Nitric Acid, containing not less than 69.0 per cent m/m and not more than 71.0 per cent m/m of nitric acid.

F.2.8 Potassium cyanide, solution, 10 per cent m/v.

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G-

F-2.8 Potassium Cyanide, solution, 10 per cent m/v.

Dissolve 10 gm of Potassium Cyanide in 90 ml of water add 2 ml of hydrogen peroxide solution allow to stand for twenty four hours and make up to 100 ml with water.

F-3.1 Preparation of primary solution.

Dissolve 2.5 gm material in 10 ml HCl and 20 ml of water add 0.5 ml HNO_3 boil to remove CO_2 if any is generated and filter to the cool filtrate add 2 gms of ammonium chloride and 2 gm of ammonium thiocyanide and extract with two successive quantities each of 10 ml of a mixture of equal parts of amyl alcohol and solvent ether. To the aqueous layer add 2 gm of citric acid.

F-3.2 Preparation of auxiliary solution.

Mix 5 ml of HCl and 30 ml of water and add 2 gm of ammonium chloride and 2 gm of ammonium thiocyanate and extract with two successive quantities each of 10 ml of mixture of equal parts of amyl alcohol and solvent ether. To the aqueous layer add 2 gm citric acid.

F-3.3 Add 10 ml of dilute lead solution to the auxiliary solution (see F.3.2). Make each solution alkaline, if necessary, with ammonia solution and add 1 ml of potassium cyanide solution. The solution should not now be more than faintly opalescent (see Note).

NOTE - If the colours of the solution differ, equalise them by the addition of a few drops of a highly diluted solution of burnt sugar or other non-reaction substance.

F-3.4 Dilute each solution to 50 ml with water and add 0.1 ml of sodium sulphide solution to each and mix thoroughly.

F-3.5 Compare the colours by a suitable method, such as by light reflected from a white tile through the Nessler cylinders.

F-3.6 The material shall satisfy the requirement for lead prescribed in Table 1, if the colour in the primary solution is not greater than that in the auxiliary solution.

APPENDIX G

DETERMINATION OF ARSENIC

G.1

APPARATUS

A wide-mouthed bottle capable of holding about 120 ml, fitted with a rubber bung through which passes a glass tube; the latter, made from ordinary glass tubing and having a total length of 200 mm and an internal diameter of exactly 6.5 mm (external diameter about 8 mm). The tube is drawn out at one end to a diameter of about 1 mm and a hole not less than 2 mm in diameter is blown in the side of the tube, near the constricted part. When the bung is inserted in the bottle containing 70 ml of liquid, the constricted end of the tube is above the surface of the liquid and the hole in the side is below the bottom of the bung. The upper end of the tube is out of square, and is either slightly rounded or ground or ground smooth.

Two rubber bungs (about 25 mm X 25 mm), each with a hole bored centrally and true, exactly 6.5 mm in diameter, are fitted with a rubber band or spring clip for holding them together.

G.2

REAGENTS

All together used shall be free from arsenic.

G.2.1

Arsenic solution, strong.

Dissolve 0.132 g of arsenic trioxide in 50 ml of hydrochloric acid and sufficient water to produce 100 ml.

G.2.2

Arsenic Solution, Dilute

Dilute 1 ml of strong arsenic solution (see G.2.1) with sufficient water to produce 100 ml. 1 ml contains 0.01 mg of arsenic. The solution must be freshly prepared.

G.2.3

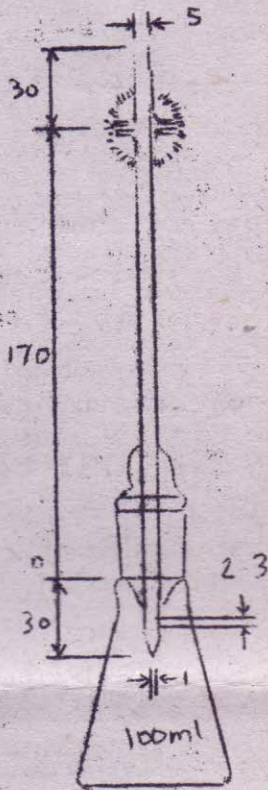
Hydrochloric acid, 32 per cent m/m.

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Apparatus of Determination of Arsenic

G-2.4 Potassium Iodide, containing not less than 99.0 per cent of potassium iodide (calculated with reference to the dried subst

G-2.5 Lead acetate, solution, 10.0 per cent m/v.

G-2.6 Mercuric chloride, containing not less than 99.5 per cent of mercuric chloride.

G-2.7 Mercuric chloride paper

Smooth white filter paper, not less than 25 mm in width, soaked in a saturated solution of mercuric chloride, pressed to remove superfluous solution, and dried at about 60°C in the dark. The grade of the filter paper is such that the mass is between 65 g/m² and 130 g/m², and the thickness in mg of 400 papers is approximately equal numerically, to the mass in g/m².

NOTE - Mercuric chloride paper should be stored in a stoppered bottle in the dark. Paper which has been exposed to sunlight or to the vapour of ammonia affords a lighter stain or no stain at all when employed in the limit test for arsenic.

G-2.8 Standard hydrochloric Acid

Hydrochloric acid diluted with water to contain about 32 % w/w of HCl and complying with following additional tests.

(i) Dilute 10 ml with sufficient water to produce 30 ml add 5 ml of ammonium thiocyanate solution and stir immediately no colour is produced.

(ii) To 50 ml add 0.2 ml of bromine solution evaporate on a water bath until reduced to 16 ml adding more bromine solution if necessary in order that an excess as indicated by the colour may be present throughout the evaporation add 50 ml of water and 5 drops of stannous chloride and apply the general test, the stain produced is not deeper than a 0.2 ml standard stain prepared with the same acid, showing that the proportion of arsenic present does not exceed 0.05 part per million.

G-2.9 Stannous chloride solution

Prepared from Stannous chloride solution by adding an equal volume of hydrochloric acid boiling down to the original volume and filtering through a fine grain filter paper. It complies with the following tests. To

To 10 ml add 6 ml of water and 10 ml hydrochloric acid distil and collect 16 ml. To the distillate add 50 ml of water and 2 drops of stannous chloride solution and apply the general test. The stain produced is not deeper than 1 ml standard stain showing that the proportion of arsenic present does not exceed 1 part per

~~million~~
million

G-3. PROCEDURE

- G-3.1 Lightly pack a glass tube with cotton wool, previously moistened with lead acetate solution and dried, so that the upper surface of the cotton wool is not less than 25 mm below the top of the tubes. The upper end of the tube is then inserted into the narrow end of one of the pair of rubber bungs, either to a depth of about 10 mm when the tube has a rounded-off end, or so that the frond end of the tube is flush with the larger end of the bung. A piece of mercuric chloride paper is placed flat on the top of the bung and the other bung placed over it and secured by means of the rubber band or spring clip in such a manner that the borings of the 2 bungs (or the upper bungs and the glass tube) meet to form a true tube 6.5 mm in diameter interrupted by a diaphragm of mercuric chloride paper.
- G-3.2 Disperse 10 g of the material in 50 ml of water and add 10 ml of stannated hydrochloric acid. Place the solution in a wide mouthed bottle, add 1 g of potassium iodide and 10 g of zinc and place the prepared glass tube quickly in position. Allow the action to proceed for 40 minutes.
- G-3.3 Compare in daylight, the yellow stain which is produced on the mercuric chloride paper if arsenic is present, with the standard stain (see Note) produced by operating in a similar manner with a solution containing 50 ml of water, 10 ml of stannated hydrochloric acid and quantities dilute arsenic solution. Make the comparison of the stains immediately at the completion of the test.
- NOTE - The standard stain used for comparison must be freshly prepared since the stain fades on keeping.
- G-3.4 The material shall satisfy the requirement for arsenic prescribed in Table 1 if the depth of colour of the stain produced in the test is not deeper than that of the 1 ml standard stain.

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