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SPECIFICATION FOR KAOLIN FOR COSMETIC INDUSTRY (METRIC UNITS)

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BUREAU OF CEYLON STANDARDS

SPECIFICATION FOR KAOLIN FOR COSMETIC INDUSTRY (METRIC UNITS)

S.L.S. 264 : 1974

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BUREAU OF CEYLON STANDARDS
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COLOMBO 3.

Sri Lanka Standards are subject to periodical revision in order to accommodate the progress made by industry. Suggestions for improvement will be recorded and brought to the notice of the Committees to which the revisions are entrusted.

This Standard does not purport to include all the necessary provisions of a contract.

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SRI LANKA STANDARD SPECIFICATION FOR KAOLIN FOR COSMETIC INDUSTRY (METRIC UNITS)

FOREWORD

This Sri Lanka Standard Specification was prepared by the Drafting Committee for Kaolin for Cosmetic Industry. It was approved by the Agricultural and Chemicals Divisional Committee of the Bureau of Ceylon Standards and was authorised for adoption and publication by the Council of the Bureau on 21st May, 1974.

There are large deposits of kaolin in this country. The crude kaolin refined is now used in large quantities as a raw material for several products. Refined kaolin conforming to certain quality standards could be used in varying proportions in cosmetic preparations including talcum powder meant for infants. This standard does not specify the proportions in which it should be used in the different cosmetic industries. Products containing kaolin should be clinically tested before they are introduced for general use.

This standard is subject to the restrictions imposed under the Ceylon Food and Drugs Act and the regulations framed thereunder wherever applicable.

In cosmetics prepared from kaolin, freedom from grit is an important attribute. Till such time as a reproducible method of test for this characteristic is developed, the method given in Appendix A shall be used for testing freedom from grit.

Dimensions and other characteristics of this specification are given in metric units. For the purpose of deciding whether a particular requirement of this standard is complied with, the final value observed or calculated expressing the result of a test or observation, shall be rounded off in accordance with C.S. 102 : Ceylon Standard on Presentation of Numerical Values. The number of figures to be retained in the rounded off values shall be the same as that of the specified value in this standard.

The assistance obtained from the publications of the Indian Standards Institution in the preparation of this standard is acknowledged.

1. SCOPE

This standard prescribes the requirements and methods of sampling and test for kaolin (china-clay) for use in cosmetic industry.

2. REQUIREMENTS

2.1 Description—The material shall be finely powdered, natural mineral kaolinite consisting essentially of hydrated aluminium silicate. The material shall be essentially insoluble in water and mineral acids.

2.2 The material shall also comply with the requirements laid down in Table 1.

3. METHODS OF TEST

The material shall be tested in accordance with the methods prescribed in Appendix A.

4. PACKING AND MARKING

4.1 The material shall be packed in well-closed containers as agreed to between the purchaser and the supplier.

4.2 The packages shall be securely closed and legibly marked with the following:

(*a*) Name of the material;

(*b*) Manufacturer's or supplier's name;

(*c*) Mass of the material in the package;

(*d*) Recognized trade-mark, if any; and

(*e*) Batch number or code number;

5. SAMPLING

5.1 The method for preparing representative test samples of the material and the criterion for conformity shall be as given in Appendix B.

TABLE 1—REQUIREMENTS FOR KAOLIN

Sl No.	Characteristic	Requirement	Method of Test (Ref. to Cl No. in Appendix A)
(1)	(2)	(3)	(4)
(*i*)	Bulk density	As agreed to between the purchaser and the supplier	A–2
(*ii*)	Fineness:		
	(*a*) Percent by mass, max., retained on 106 micron sieve*	0.1	A–3
	(*b*) Percent by mass, max., retained on 53 micron sieve*	2.0	
(*iii*)	Grit	Nil	A–4
(*iv*)	Loss on drying, percent by mass, max.	1.0	A–5
(*v*)	Loss on ignition, percent by mass, max.	15.0	A–6
(*vi*)	Carbonates	To pass the test	A–7
(*vii*)	Matter soluble in dilute hydrochloric acid, percent by mass, max.	1.0	A–8
(*viii*)	Iron	To pass the test	A–9
(*ix*)	Heavy metals (as Pb), mg/kg, max.	5	C.S. **
(*x*)	Arsenic (as As), mg/kg, max.	2	C.S. §
(*xi*)	Brightness	75	A–10

* C.S. 124 : Test Sieves

**Sri Lanka Standard Method for the Determination of Lead (under preparation)

§ Sri Lanka Standard Method for the Determination of Arsenic (under preparation).

APPENDIX A

METHODS OF TEST FOR KAOLIN FOR COSMETIC INDUSTRY

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water shall be employed in tests.

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

A-2 DETERMINATION OF BULK DENSITY

A-2.1 Apparatus

A-2.1.1 Assemble the apparatus as shown in Fig. 1. The base of the measuring cylinder A shall be ground flat and the empty measuring cylinder A together with the rubber bung shall weigh 250 ± 5 g. It shall be accurately calibrated to 250 ml with an error, if any, of less than one millilitre. The distance between 0- and 250-ml graduation on the measuring cylinder A shall be not less than 220 mm and not more

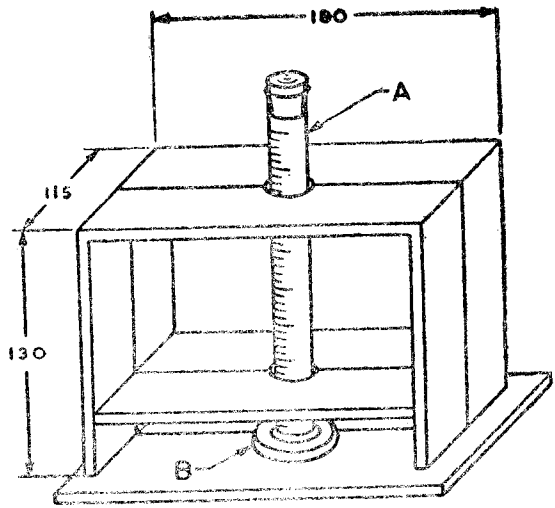

Fig. 1—Apparatus for Determination of Bulk Density.

than 240 mm. The distance between the flat-ground part of the base of measuring cylinder A and the rubber base pad B, when the measuring cylinder A is raised to the full height, shall be 25 $\pm$ 2 mm.

A-2.1.2 The rubber base pad B shall have a shore hardness of 42 to 50.

A-2.1.3 Pans of the balance shall be at least 100 mm in diameter. The balance shall be sensitive to less than 0.1 g.

A-2.2 Procedure—Sieve about 40 g of the material through 250-micron sieve (see C.S. 124*) on to a tared glazed paper and weigh it accurately. Slip the powder gently and smoothly into the measuring cylinder, which should be held at 45° to the vertical, without knocking or squeeing. Assemble the apparatus as shown in Fig. 1. With the thumb and four fingers of one hand, gently grasp the upper part of the cylinder and, within one second, lift it about 25 mm (taking care not to jerk the cylinder by knocking it against the upper stop), then let it drop. Note the volume after dropping it once. Continue lifting and dropping until 50 complete drops have been given to the cylinder. During the operation, give a gentle turn of about 10° in clockwise direction to the cylinder after every two drops. As soon as 50 drops are completed, raise the cylinder to eye level and read the volume of the material.

A-2.3 Calculation

Bulk density in g per ml:

(*a*) After one drop $= \frac{m}{V_1}$

(*b*) After 50 drops $= \frac{m}{V_{50}}$

where,

m = mass in g of the material taken for the test,

V_1 = volume in ml of the material after one drop, and

V_{50} = volume in ml of the material after 50 drops.

* C.S. 124 : Ceylon Standard on Test Sieves.

A–3 DETERMINATION OF FINENESS

A–3.1 Procedure for Material Retained on 106-micron sieve—Place about 10 g of the material weighed to the nearest mg, in 90-micron sieve (see C.S. 124*) and wash, first by means of a slow stream of running tap water and then with a fine stream from a wash bottle until all the material that can pass through the sieve has passed. Let the water drain from the sieve, then dry the sieve containing the residue on a stream-bath. If there is any residue, carefully transfer it on to a tared watch-glass and dry it to constant mass at 105 ± 2°C.

A–3.2 Calculation

Material retained on the specified sieve, percent by mass $= 100 \frac{m_1}{m}$

where,

m_1 = mass in g of the residue retained on the sieve, and

m = mass in g of the material taken for the test

A–3.3 Procedure for Material Retained on 53-micron sieve*—Report the procedure prescribed in A-3.1 using a fresh quantity of the material and 45-micron sieve*. Calculate as given in A–3.2.

A–4 TEST FOR GRIT—Take a 20 g sample of the material in a beaker and wet it with a little rectified spirit. Remove by overflow in a carefully controlled steady stream of water, a larger portion of the material. The grit being heavier will remain in the beaker along with some kaolin. Test the residue by rubbing between the finger and the thumb, for the presence of grit.

A–5 DETERMINATION OF LOSS ON DRYING

A–5.1 Procedure—Weigh about 4 g of the material to the nearest mg, in a crucible and heat at 105 ± 2°C in an oven to constant mass. Preserve the dried material for test in A-6.

A–5.2 Calculation

Loss on drying, percent by mass $= \frac{(m - m_1)}{m} \times 100$

* C.S. 124 : Ceylon Standard on Test Sieves.

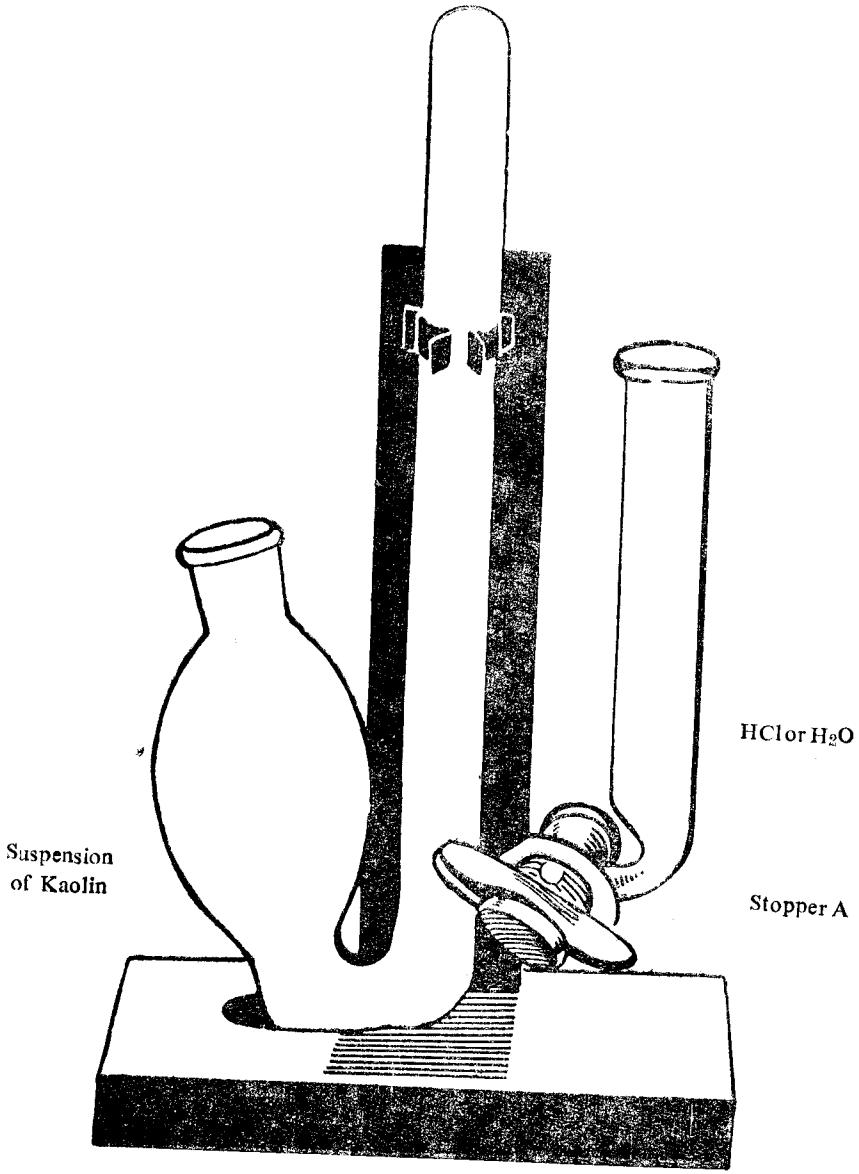

Fig. 2—Ureometer

where,

m = mass in g of the material taken for the test, and

m_1 = mass in g of the material after drying.

A-6 DETERMINATION OF LOSS ON IGNITION

A-6.1 **Procedure**—Heat the dish with the dried material, as preserved in A-5.1, at red heat to constant mass. Cool in a desiccator and weigh.

A-6.2 **Calculation**

Loss on ignition, percent by mass = $\frac{m - m_1}{m} \times 100$

where,

m = mass in g of the material taken for the test in A-5.1 and

m_1 = mass in g of the ignited material.

A-7 TEST FOR CARBONATES

A-7.1 **Apparatus**—Ureometer (Fig. 2).

A-7.2 **Procedure**—Set up 2 ureometer tubes, each containing about 30 ml of a 10% suspension of kaolin in water. Through the open side arm of one add 2 ml of 20 N hydrochloric acid, while to the other 2.0 ml of water. Close the stopper (A), fill open side arm with water and allow to stand for 30 minutes. If no collection of gas (CO_2) takes place in the tube containing kaolin and hydrochloric acid, the material shall be taken as having passed the test.

A-8 DETERMINATION OF MATTER SOLUBLE IN DILUTE HYDROCHLORIC ACID

A-8.1 **Reagent**—Dilute hydrochloric acid—approximately 5N.

A-8.2 **Procedure**—Mix about 5 g of the material, weighed to the nearest mg, with 50 ml of dilute hydrochloric acid in a 250 ml beaker. Cover the beaker with a watch-glass and warm the contents on a steam-bath. Digest

the contents of the beaker for about half an hour and filter. Wash the residue with warm dilute hydrochloric acid. Evaporate the filtrate to dryness and dry at 105 ± 2°C to constant mass.

A-8.3 Calculation

Matter soluble in dilute hydrochloric acid, percent by mass, $= \frac{m_1}{m} \times 100$

where,

m_1 = mass in g of the dry residue in the dish, and

m = mass in g of the material taken for test

A-9 TEST FOR IRON

A-9.1 Outline of the Method—Ferric ions give a rose wine colour with ammonium thiocyanate. To an acidified solution of iron in the ferric state, ammonium thiocyanate is added.

A-9.2 Reagents

A-9.2.1 Ammonium Thiocyanate Solution—20% solution of ammonium thiocyanate.

A-9.2.2 Standard Iron Solution—Dissolve 0.216 g, of ferric ammonium sulphate [Fe $(NH_4)(SO_4)_2 12H_2O$] in water, add 5 ml, of dilute nitric acid and make up to 500 ml with water. This solution contains 50μg/ml.

A-9.2.3 Working Standard—Dilute 10 ml of the standard iron solution to 50 ml with water. This solution contains 10μg Fe/ml.

A-9.2.4 Concentrated Nitric Acid.

A-9.2.5 Dilute Ammonia Solution.

A-9.3 Procedure—Weigh about 2.0 g of the sample to the nearest mg and boil gently with 3 ml. of conc. nitric acid and 7 ml of water for 2-3 minutes. Filter and wash the residue with water. Collect the filtrate and washings and make up to 100 ml with water (Sample solution).

Measure 5 ml of this solution, add about 10 ml of water and neutralise to litmus with dilute ammonia solution. Add 5 ml of dilute nitric acid, 1 ml of ammonium thiocyanate solution and make up to 100 ml with water. The colour produced is not deeper than that produced by treating 4 ml of the working standard in the same manner, commencing at the words 'add about 10 ml of water....'

A-9.4 Calculation—This test corresponds to a limit of 0.04% of iron calculated as Fe, in the following manner:—

4 ml of the working standard contains 40μg of Fe. If the colour obtained from the sample solution is identical with that of the standard 0.1 g of the sample (5 ml of the solution) contain the equivalent of 40μg of Fe.

$$\text{Therefore the percentage of iron} = \frac{0.00004 \times 100}{0.1}$$
$$= 0.04\%$$

A-10 DETERMINATION OF BRIGHTNESS

A-10.1 Apparatus—Photovolt Photoelectric reflection meter Model No. 610, with search unit No. 6105, Green tristimulus filter, calibrated white enamel working standard plaque, having a standard brightness of 73.0 and an external mirror type Galvanometer.

A-10.2 Procedure—The search unit is first placed on the calibrated enamel plaque which serves as the working standard and the needle is set at 73.0 for the green tristimulus filter. Then the search unit is placed on the sample and the pointer will indicate the reflection value directly.

APPENDIX B
SAMPLING OF KAOLIN FOR COSMETIC INDUSTRY

B-1 GENERAL REQUIREMENTS

B-1.1 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.2 Samples shall not be taken in an exposed place.

B-1.3 The sampling instrument shall be clean and dry.

B-1.4 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.5 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.6 The samples shall be placed in clean, dry, air-tight, glass or other suitable containers.

B-1.7 Each sample container shall be of such a size that it is almost completely filled by the sample.

B-1.8 Each sample container shall be sealed air-tight with a stopper after filling and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

B-2 SIZE OF SAMPLING

B-2.1 Lot—All the containers in a single consignment of the material drawn from a single batch of manufacture shall constitute a lot. If a consignment is declared to consist of different batches of manufacture, the batches shall be marked separately and the groups of containers in each batch shall constitute separate lots.

B-2.1.1 Samples shall be tested from each lot for ascertaining conformity of the material to the requirements of the specification.

B-2.2 The number of containers (n) to be chosen from the lot shall depend on the size of the lot (N) and shall be in accordance with Table 2.

TABLE 2

NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

Lot Size N (1)	Sample Size n (2)
3 to 50	3
51 to 200	4
201 to 400	5
401 to 650	6
651 to 1000	7

B-2.3 The containers to be selected for sampling shall be chosen at random from the lot and for this purpose random number tables shall be used. In case such tables are not available, the following procedure may be adopted.

Starting from any container, count them as 1, 2, 3 .....r, and so on, in a systematic manner, where r is the integral part of N/n. Every rth container thus counted shall be withdrawn from the lot.

B-3 TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Samples—Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected (see Table 2). The total quantity of the material drawn from each container shall be sufficient to conduct the test for all the characteristics given in Clause 2 and shall be not less than 0.2 kg.

B-3.1.1 Thoroughly mix all portions of the material drawn from the same container. Out of these portions, equal quantity shall be taken from each selected container and shall be mixed well together so as to form a composite sample weighing not less than 0.5 kg. This composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

B-3.2 Referee Sample—The referee sample shall consist of the composite sample marked for this purpose and shall bear the seals of the purchaser and the supplier. It shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of dispute between the two.

B-4 NUMBER OF TESTS

B-4.1 Tests for all the requirements given in Clause 2 shall be conducted on the composite sample.

B-5 CRITERION FOR CONFORMITY

B-5.1 A lot shall be declared as conforming to ths specification if the composite sample satisfies all the requirements given in Clause 2.

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The Sri Lanka Standards Institution is the owner of the registered certification mark shown below. Beneath the mark, the number of the Sri Lanka Standard relevant to the product is indicated. This mark may be used only by those who have obtained permits under the SLS certification marks scheme. The presence of this mark on or in relation to a product conveys the assurance that they have been produced to comply with the requirements of the relevant Sri Lanka Standard under a well designed system of quality control inspection and testing operated by the manufacturer and supervised by the SLSI *which includes surveillance inspection of the factory, testing of both factory and market samples.*

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