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Indian Standard

SPECIFICATION FOR COPPER PHOSPHATE — ZINC PHOSPHATE DENTAL CEMENT

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INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 1

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0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 7 December 1970, after the draft finalized by the Dental Materials Sectional Committee had been approved by the Chemical Division Council.

0.2 In the preparation of this standard, assistance has been obtained from US Federal Specification U-C-198 a ' Copper and zinc phosphate dental cement ', of 1957, published by the General Services Administration, USA.

0.3 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 analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for copper phosphate — zinc phosphate dental cement.

2. REQUIREMENTS

2.1 Description — The material shall consist essentially of a powder and liquid which, when mixed as directed, shall possess the working qualities specified in 2.4.2.

2.2 **Liquid Component** — The liquid shall be essentially a phosphoric acid solution and shall be free from cloudiness, precipitates, deposits, or sediments. The liquid shall be furnished in bottles in an amount 20 percent in excess of that necessary to combine with the total amount of powder in a bottle of full portion size, when mixed to standard testing consistency.

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*Rules for rounding off numerical values (*revised*).

2.3 Powder — The powder shall be composed basically of zinc oxide, to which has been added not less than 25 parts of cuprous oxide, and shall be free from lumps or granules. Unless otherwise specified, the powder shall be furnished in bottles, each containing 25 g.

2.4 **Cement**

2.4.1 The cement shall not be irritating to oral tissues or cause objectionable discoloration of tooth structure.

2.4.2 The cement, when mixed as directed and spatulated in the usual dental manner, shall:

a) harden or set to a condition satisfactory for dental use,

b) be free from poisonous and foreign materials,

c) not form lumps or granules, and

d) not evolve gas.

2.4.3 *Time of Setting* — The time of setting shall be not less than 4 minutes, nor more than 10 minutes, when tested as prescribed in A-3.

2.4.4 *Ultimate Compressive Strength* — It shall be not less than 1 000 kg/cm^2 for specimens crushed 7 days after mixing, when tested as prescribed in A-4.

2.4.5 *Film Thickness* — The film thickness, tested as prescribed in **A-S,** shall be not more than 0·04 mm.

2.4.6 *Disintegration* — The disintegration of the cement shall not exceed 0·20 percent by weight after immersion for 7 days in distilled water when tested as prescribed in A-5.

2.4.7 Arsenic -The cement shall contain not more than 2 parts per million of arsenic when tested as prescribed in A-6.

2.5 **Instructions for Use** — Adequate and accurate instructions for proportioning and manipulating the material shall accompany each package of the material. These instructions shall include the powder-liquid ratio, the temperature of the mixing slab, the rate of powder incorporation, and the time of mixing.

3. **PACKING AND MARKING**

3.1 Packing

3.1.1 The solid and the liquid component shall be individually packed in well made glass bottles, which shall be securely stoppered with a screw cap properly cushioned and having an impervious liner. Each bottle shall be further sealed by a gel or similar viscous cap covering the stopper externally and extending well down the neck of the bottle,

3.1.2 ***Unit Package*** — This shall consist of one bottle each of powder and liquid; one measuring pipette made of good quality glass, fitted with a rubber bulb, and suitable in style and design for dispensing the liquid drop by drop; and instructions for use (see 2.5), contained in a cardboard box, or as agreed to between the purchaser and the supplier.

3.2 Marking

3.2.1 Each individual bottle shall carry a label indicating the name of the material, quantity contained, the manufacturer's name and recognized trade-mark, if any, and the lot number.

3.2.2 Each unit package and bulk package shall be suitably marked with the name of the material, quantity of contents and name of the manufacturer.

3.2.3 The bulk package and unit package may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act, and the Rules and Regulations made thereunder. Presence of this mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard, under a well-defined system of inspection, testing and quality control during production. This system, which is devised and supervised by ISI and operated by the producer, has the further safeguard that the products as actually marketed are continuously checked by ISI for conformity to the standard. Details of conditions, under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 The method of preparing samples of the material and the criteria for conformity shall be as given in Appendix B or as agreed to between the purchaser and the supplier.

APPENDIX A

METHODS OF TEST FOR COPPER PHOSPHATE — ZINC PHOSPHATE DENTAL CEMENT

A-1. DETERMINATION OF TESTING CONSISTENCY

A-1.1 Apparatus — The type of apparatus shall be essentially that shown in Fig. 1. This apparatus consists of two flat glass plates, a weight, a gauge plug and a glass tube of inside diameter approximately 6·5 mm which will deliver 0·50 ± 0·02 ml of mixed cement. The combined weight of the top plate and the weight shall be 120 g.

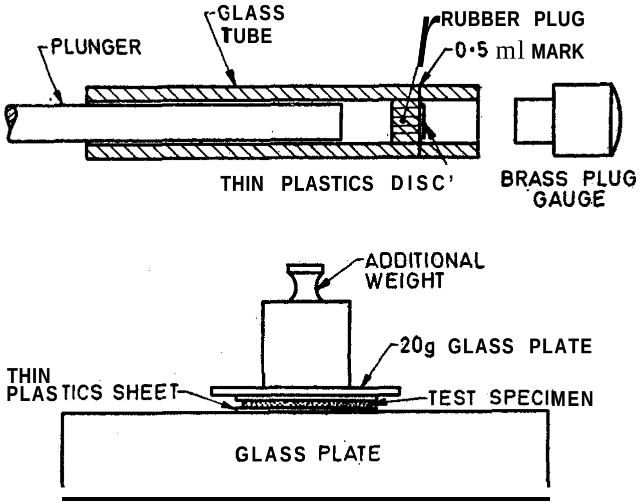


FIG. 1 APPARATUS FOR MEASURING CONSISTENCY

A-1.2 Procedure — Mix trial amounts of the powder with 0·50 ml of the liquid. Then deliver from the glass tube 0·50 ± 0·02 ml of each mix on to a flat glass plate. Three minutes after the mixing is started, carefully lower the top glass plate weighing approximately 20 g and the necessary additional weight required to total 120 g on to the soft cement. Make trials until the average of the major and minor diameters of the slumped mass of cement is 30 ± 1 mm ten minutes after starting the mix. Carry out 3 such determinations.

A-1.3 The average weight of powder used in three determinations, to the nearest 0·05 g, shall be taken as the standard testing consistency.

A-2. PREPARATION OF TEST SPECIMENS

A-2.1 Conduct the preparation of test specimens at 27 ± 2°C and at a relative humidity between 55 and 75 percent.

A-2.2 The powder/liquid ratio shall be determined by the consistency test (A-1).

A-2.3 The mixing technique employed shall be according to manufacturer's instructions (2.5). All apparatus and instruments shall be clean, dry and free from particles of hardened cement.

A-3. DETERMINATION OF TIME OF SETTING

A-3.1 Apparatus — The type of apparatus required is shown in Fig. 2.

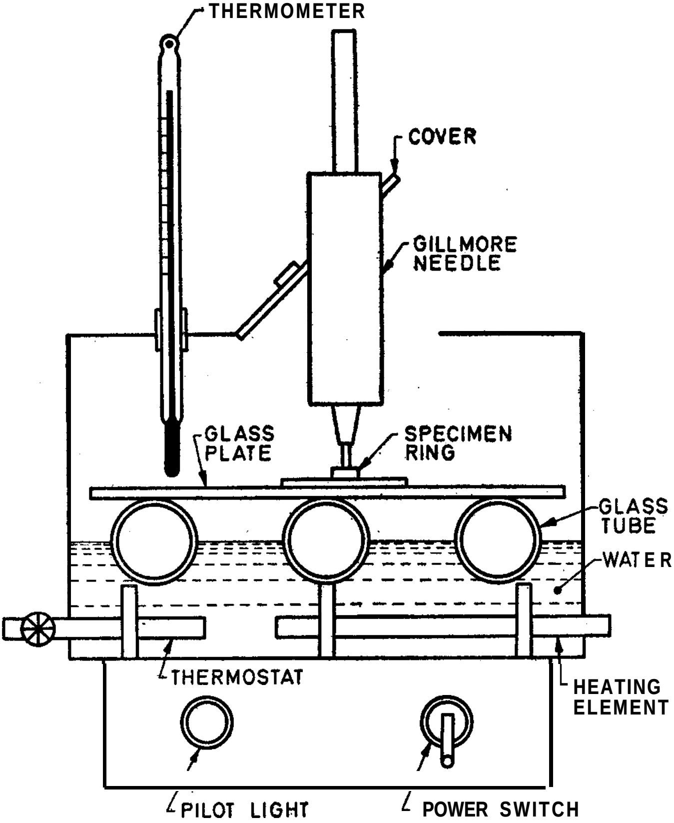


FIG. 2 APPARATUS FOR DETERMINATION OF SETTING TIME AT CONTROLLED TEMPERATURE AND HUMIDITY

A-3.1.1 *Metal Ring* — cylindrical, 5 mm high and 10 mm inside diameter.

A-3.1.2 *Gillmore Needle* — weighing 450 ± 5 g and having an end 1·06 mm in diameter.

A-3.2 Procedure -Place the metal ring on a flat plate and fill it with the cement of standard consistency (**A-l**). Three minutes after starting the mix, transfer the specimen to an atmosphere of 100 percent relative humidity at 37°C. Three and a half minutes after starting the mix, lower the Gillmore needle vertically on to the test specimen until the surface of the cement is touched. Repeat this at 30 second intervals.

A-3.2.1 The time of setting shall be the number of minutes elapsed from the starting of the mix to the time when the needle fails to make a perceptible circle on the surface of the specimen. Report the result to the nearest minute.

A-4. DETERMINATION OF ULTIMATE COMPRESSIVE STRENGTH

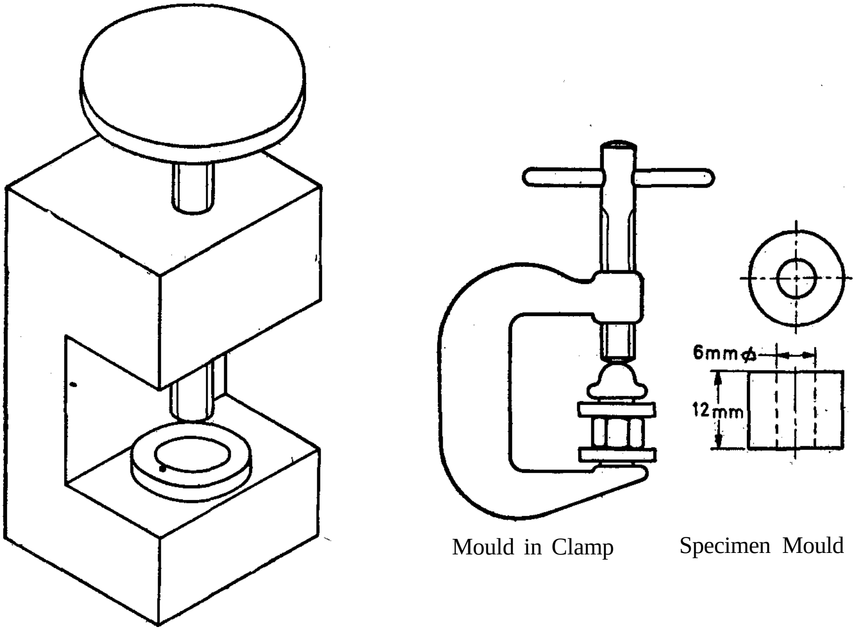
A-4.1 Preparation of Test Specimens -The test specimen shall be cylinders 12 mm in height and 6 mm in diameter. The ends of the specimen should be flat, smooth, parallel to each other and at right angles to the long axis of the cylinder. An apparatus found convenient for forming these test cylinders is shown in Fig. 3. Place a cylindrical mould (made of hard rubber, glass, stainless steel or any other substance which will not react with cement), 12 mm high and 6 mm in diameter on a flat glass plate and slightly overfill with cement of standard consistency within three minutes after commencing the mixing. Press on top of the mould a second flat glass plate. Hold the mould and the plates firmly together with a small G-clamp. All apparatus should be at room temperature. The moulds may be coated with a 3 percent solution of a microcrystalline wax (melting point 91 to 86°C) in benzene. Three minutes after starting the mix, transfer the mould and clamp to an atmosphere of 100 percent relative humidity at 37°C. Thirty minutes later, remove the specimens from the, conditioned atmosphere and immerse them in distilled water at room temperature. Surface the ends of the cylinder plane at right angles to the axis, by drawing the moulds containing the specimens back and forth across a glass plate coated with an abrasive such as 75 micron silicon carbide powder and water. Rotate them about one-fourth turn every few strokes. Keep the test specimens wet during the grinding. After surfacing, remove the specimens from the mould by a screw jack (see Fig. 3) and immerse in distilled water till taken up for test. Test after 7 days.

A-4.2 **Procedure —** Insert the specimen, prepared according to A-4.1, between the platens of the testing machine with a small piece of wet blotting paper approximately 0·5 mm thick at each end, Operate the machine at a speed which will move the crushing head 0·25 mm per minute.

A-4.3 Expression of Results -- Report the value for compressive strength as the average of three or more from a lot of five specimens and round off to the nearest 10 kg/cm^2. If the values for individual specimens fall more than 15 percent below the average of the five, discard them and report the average of the remaining specimens. If more than two of the specimens are discarded, repeat the test.

A-5; DETERMINATION OF FILM THICKNESS

A-5.1 Procedure-Place a portion of a mix of standard consistency (see A-l) between two flat square or round glass plates of uniform thickness.



Mould in Clamp

Specimen Mould

Screw Jack for Ejecting Specimen from Moulds

FIG. 3 APPARATUS USED IN FORMING ULTIMATE COMPRESSIVE STRENGTH SPECIMENS

The surface areas of the plates between which the cement is spread shall be approximately 2 square centimetres. Three minutes after the mix is started apply a load of 15 kg vertically on the top plate. Ten minutes after the mix is started, determine the thickness of the two plates with the cement film between them.

A-5.2 Record the difference in thickness of the plates with and without the cement film as the film thickness. Calculate and report the average of three tests to the nearest 5 microns.

A-6. TEST FOR DISINTEGRATION

A-6.1 Preparation of Test Specimens — Place 0·5 ml of cement of standard testing consistency (see **A-1**) in a split stainless steel ring, 20 mm inside diameter and 1·5 mm thick, which is placed on a flat plate and separated from it by a thin polyethylene sheet. Use another flat plate faced with a sheet of thin polyethylene to press the cement into the ring. Place a tared piece of fine platinum or corrosion-resistant wire in the soft cement as the specimens are formed to provide a convenient means of holding the specimens. Three minutes after the mix is started, place the plates and cement for one hour in an atmosphere having a relative humidity of 100 percent at 37°C. Two such specimens shall be used for each determination.

A-6.2 **Procedure** — Place two specimens, prepared and conditioned in accordance with A-6.1, in each of two tared weighing bottles and weigh (*see* Fig. 4). Take the combined weight of the specimen and the weighing bottle, less the weight of weighing bottle and the platinum wire, as the weight of the specimens of cement. Immediately submerge the specimens by pouring 50 ml of distilled water into the weighing bottle which shall be stored for 7 days at 37°C. Then remove the specimens from the water. There shall be no evidence of crystal growth on the surface of the specimens. Evaporate the water from the weighing bottle at a temperature just below 100°C, and then dry the bottle at 150°C. Cool to room temperature in a desiccator containing thoroughly dry anhydrous calcium sulphate ($CaSO_4$) or silica gel, freshly dried at 130°C. Weigh the weighing bottle and contents with a precision of 0·2 mg. Repeat this cycle of heating the weighing bottle to 150°C, cooling over a desiccant and reweighing until the weight loss of each bottle is not more than 0·5 mg.

A-6.3 Expression of Results-Record difference between the final weight of the weighing bottle and its initial weight as the amount of disintegration. Divide the gain in weight by the weight of the specimens, times 100, to obtain the percentage of disintegration. Calculate and report the average of the two tests to the nearest 0·1 percent.

A-7. DETERMINATION OF ARSENIC

A-7.1 Apparatus — A suitable apparatus is shown in Fig. 5. The generator consists of a 50 ml capacity wide-mouth bottle C which is fitted with a perforated rubber bung. Through the perforation is inserted a vertical exit tube about 12 cm in overall length, about 1 cm in diameter on its upper portion and constricted at its lower extremity to a tube about 4 cm in length and about 5 mm in diameter. The small portion of the tube shall extend to just slightly below the bung. This tube shall be packed with glass wool as shown at *B*. Into the upper end of this tube shall be placed a second glass tube 12 cm in length with an internal diameter of 2·5 to 3·0 mm, by means of a rubber bung.

A-7.2 Reagents — The reagents shall be of recognized analytical reagent quality and shall be free from arsenic impurities. Distilled water shall be used throughout.

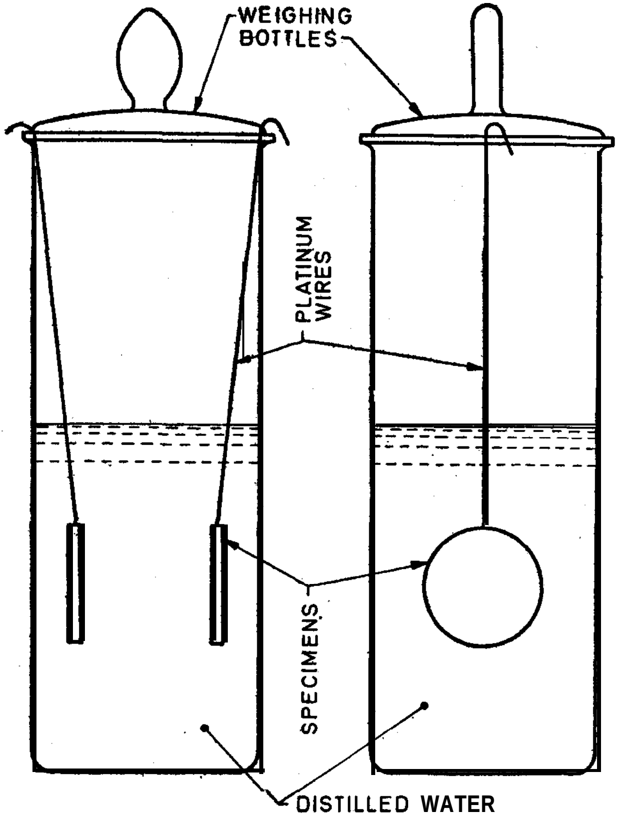


FIG. 4 WEIGHING BOTTLE CONTAINING SOLUBILITY SPECIMENS

A-7.2.1 *Standard Arsenic Solution* — Dissolve 0·132 of arsenic trioxide (As_2O_3) in 10 ml of 10 percent sodium hydroxide solution. Neutralize the alkaline arsenic solution with dilute sulphuric acid (one volume of concentrated acid plus nine volumes of distilled water). Add 10 ml more of the diluted acid and dilute with water to 1 litre. One millilitre of the solution contains 0·1 mg of arsenic. To 10 ml of this solution add 10 ml of dilute sulphuric acid and add water to make 1 litre of solution. This final solution contains 0·001 mg of arsenic per millilitre.

A-7.2.2 *Potassium Iodide Solution* — Dissolve 10 g of potassium iodide in water and dilute to 100 ml.

A-7.2.3 *Stannous Chloride Solution -Dissolve* 40 g of stannous chloride dihydrate in concentrated hydrochloric acid and dilute to 100 ml with concentrated hydrochloric acid.

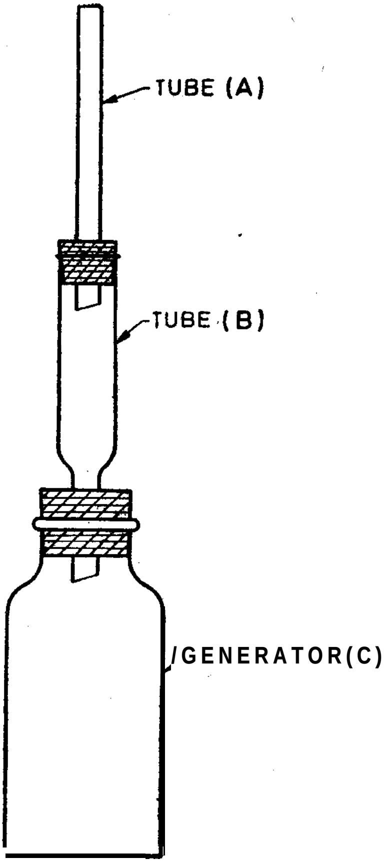


FIG. 5 APPARATUS FOR ARSENIC DETERMINATION

A-7.2.4 *Concentrated Sulphuric Acid*

A-7.2.5 *Granulated Zinc* — arsenic-free.

A-7.2.6 *Lead Acetate Solution* -- Dissolve 10 g of lead acetate in water and add enough acetic acid to clear the solution. Dilute with water to 100 ml.

A-7.2.7 *Mercuric Bromide Paper Strips* — *Use* filter paper strips which are 2·5 mm wide and cut to a length of 12 cm. Soak the strips in mercuric bromide solution (5 percent) for one hour and dry in air. Prepare the mercuric bromide solution by dissolving 5 g of mercuric bromide in 95 percent ethanol and diluting with ethanol to 100 ml.

A-7.3 Preparation of Test Specimen-Powder 1 g of hardened cement, obtained from specimen 24-hour old, that has been stored in a dry air-tight container, to pass a 75-micron sieve. Digest the powdered sample in 50 ml of distilled water on a steam-bath for one hour. Use the filtrate in the test for arsenic.

A-7.4 **Procedure** — Transfer the filtrate, prepared in A-7.3, to the wide mouth bottle (Fig. 5). For the comparison standard place in a bottle of a second generator (Fig. 5) 48 ml of water and 2·0 ml of standard arsenic solution. Add to each bottle 5 ml of concentrated sulphuric acid, 7·5 ml of potassium iodide solution and 0·20 ml of stannous chloride solution. Mix and allow the bottle to stand for 20 minutes in a water-bath at 27·0 ± 2·0°C. During this 20-minute period moisten the glass wool in lower tube *B* with 10 percent lead acetate' solution (Note), and carefully centre a dry mercuric bromide paper strip in the top tube A. Crimp the upper end of the paper strip so that 10 cm will be in position in the tube. At the end of the 20-minute period add 5 g of granulated zinc to the solution in each generator bottle, and put tubes *A* and *B* in place as shown in Fig. 5. Return both generators to the water-bath for one and one-half hours before comparing the stains. Remove the strips and average the length of the stains on both sides of the strip.

NOTE — **The character of the stain is affected by the amount of lead acetate solution used to moisten the glass wool. If the wool is too wet, the stain, which appears on the mercuric bromide paper soon after the zinc is added, will be partly washed out at the end of the one and one-half hours. For this reason, all tubes in a set of generators shall be charged with equal amounts of lead acetate, and any excess shall be drawn off by suction.**

A-7.4.1 *Expression of Results* — If the yellow-to-brown stain is shorter for the sample than for the'standard, the amount of water-soluble arsenic in the cement is less than the prescribed limit.

APPENDIX B

(*Clause* 4.1)

SAMPLING OF COPPER PHOSPHATE — ZINC PHOSPHATE CEMENT

B-1. GENERAL REQUIREMENTS OF SAMPLING

B-1.0 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.1 Samples shall not be taken in an exposed place.

B-1.2 The sampling instrument shall be clean and dry.

B-1.3 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.4 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.5 The samples shall be placed in clean, dry, air-tight glass or other suitable containers.

B-1.6 The sample containers shall be of such size that they are almost completely filled by the sample.

B-1.7 Each sample cantainer shall be sealed air-tight with a suitable stopper after filling, and marked with full details of sampling, the date of sampling and the year of manufacture of the material.

B-2. SCALE 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 or known to consist of different batches of manufacture, the containers belonging to the same batch shall be grouped together and each such group shall constitute a separate lot.

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

B-2.2 The number of containers (n) to be selected from the lot shall depend on the size of the lot (N) and shall be as given in Table 1, subject to the provision that if n containers do not provide sufficient material for carrying out all the tests specified in 2, then at least as many containers as will provide sufficient material shall be taken out.

TABLE 1 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING

LOT SIZE (N) (1)	NUMBER OF CONTAINERS TO BE SELECTED (n) (2)
3 to 50	3
51 „ 200	4
201 „ 400	5
401 „ 650	6
651 „ 1000	7

B-3. TEST SAMPLES AND REFEREE SAMPLE

B-3.1 Preparation of Test Samples

B-3.1.1 *Liquid Component-Empty* the contents of all the sample containers selected into a clean glass-stoppered bottle. Thoroughly mix the contents and divide the composite sample into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

B-3.1.2 *Solid Component* — Empty the contents of all the sample containers selected into a square-sided jar having a capacity of 2 litres and a self-sealing cap. Rotate the jar on its minor axis for two hours at the rate of 25 rev/min. Divide the composite sample 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 one composite sample each of the solid component and the liquid component, marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in a case of dispute.

B-4. NUMBER OF TESTS

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

B-5. CRITERIA FOR CONFORMITY

B-5.1 A, lot shall be declared as conforming to this specification if the composite sample satisfies the requirements for each of the characteristics listed in 2. If the requirements for any of the characteristics are not met, the lot shall be declared to have not satisfied the requirements of the specification.

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- **Chemical**
- **Civil Engineering**
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- **Electrotechnical**
- **Marine, Cargo Movement and Packaging**
- **Mechanical Engineering**
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- **Textile**

Of these, the standards belonging to the Chemical Group fall under the following categories:

- Acids
- Adhesives
- Alcohols and Allied Products
- Alkalis
- Brushware
- Ceramicware, Enamelware and Laboratory Porcelain
- Chemical Hazards
- Chemicals, Inorganic (Miscellaneous)
- Chemicals, Organic (Miscellaneous)
- Coal and Coke
- Coal Carbonization Products
- Cosmetics and Toilet Goods
- Dental Materials
- Dye Intermediates
- Electroplating Chemicals
- Explosive and Pyrotechnic Materials
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- Footwear
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