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IS 10116 (1982): Boric acid [CHD 1: Inorganic Chemicals]



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“Knowledge is such a treasure which cannot be stolen”



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IS : 10116 - 1982

*Indian Standard*  
SPECIFICATION FOR  
BORIC ACID

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**BUREAU OF INDIAN STANDARDS**  
MANAK BHAVAN, 9 BHADUR SHAH ZAFAR MARG  
NEW DELHI 110002

Gr 4

May 1982

# Indian Standard

## SPECIFICATION FOR BORIC ACID

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*Indian Standard*  
SPECIFICATION FOR  
BORIC ACID

0. FOREWORD

**0.1** This Indian Standard was adopted by the Indian Standards Institution on 24 February 1982, after the draft finalized by the Inorganic Chemicals ( Misc ) Sectional Committee had been approved by the Chemical Division Council.

**0.2** Boric Acid is used in the manufacture of glass, ceramics, abrasives, metal alloys, electrolytic condensers, explosives, nuclear energy, cosmetics and other industries. This standard covers specification for all these uses, except cosmetics for which IS : 263-1977\* will be revised by the Cosmetics Sectional Committee of the Petroleum, Coal and Related Products Division Council. The revised standard will include only the cosmetic grade of the material.

**0.3** This standard only includes limit tests on various characteristics, as these have been found to be practical and are in use by all concerned. However, for more accurate determinations, attention is drawn to the following ISO Standards, issued by the International Organization for Standardization.

- a) ISO 3119-1976 Boric acid, boric oxide and disodium tetraborates for industrial use — Determination of chromium content-diphenyl carbazide photometric method;
- b) ISO 3121-1976 Boric acid, boric oxide and disodium tetraborates, for industrial use — Determination of chloride content — Mercurimetric method; and
- c) ISO 3123-1976 Boric acid, boric oxide, disodium tetraborates sodium perborates and crude borates for industrial use — Determination of iron content — 2, 2' — Bipyridyl photometric method.

**0.4** 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.

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\*Specification for boric acid ( *third revision* ).

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

**IS : 10116 - 1982**

**1. SCOPE**

**1.1** This standard prescribes the requirements and the methods of sampling and test for boric acid used in the manufacture of capacitors, electronics, nuclear energy, foundries, fire proofing of workman's apparel glass and ceramics, explosives, refractories, paints, chemicals, dye intermediates, electroplating, leather, adhesives and enamel industries.

**2. GRADES**

**2.1** There shall be two grades of the material, namely:

- (a) Special Quality (SQ) — for use in capacitors, electronics, nuclear energy, foundries, and fire-proofing of workman's apparel; and
- (b) Technical (TECH) — for use in glass, ceramics, explosives, refractories, paints, chemicals, dye intermediates, electroplating, leather, adhesives and enamel industries.

**3. REQUIREMENTS**

**3.1 Description** — Special quality grade (SQ) material shall be in the form of granules only. The technical grade (TECH) material shall be in the form of granules, powder, crystals or flakes. Both the grades shall be free from odour, dirt, foreign matter and visible impurities.

**3.2** The material, when tested according to the methods prescribed in Appendix A, shall comply with the requirements given in Table 1. Reference to the relevant clauses of Appendix A is given in col 5 of the Table 1.

**3.3 Boric Acid for use in Explosives** — This shall meet the requirement of technical grade of the material ( see col 4, Table 1 ) and shall conform to the following additional requirements:

- a) Iron ( as Fe )  
percent by mass, *Max*                      0.05
- b) Chloride ( as Cl ),  
percent by mass, *Max*                      0.03
- c) Sulphate ( as SO<sub>4</sub> ),  
percent by mass, *Max*                      0.10



TABLE 1 REQUIREMENTS FOR BORIC ACID

( Clauses 3.2, 3.3, A-6 3.1, A-11.3.3, B-5.2 and B-5.3 )

SL No.	CHARACTERISTIC	REQUIREMENT		METHOD OF TEST ( REF TO CL No. IN APPENDIX A )
		Special Quality ( SQ )	Technical ( TECH )	
(1)	(2)	(3)	(4)	(5)
i)	Boric acid ( as $H_2BO_3$ ), percent by mass, <i>Min</i>	99.9	99.5	A-2
ii)	Moisture percent by mass, <i>Max</i>	—	0.5	A-3
iii)	Water insoluble matter, percent by mass, <i>Max</i>	0.005	—	A-4
iv)	Heavy metals ( as Pb ), percent by mass, <i>Max</i>	0.000 2	—	A-5
v)	Iron ( as Fe ), percent by mass, <i>Max</i>	0.000 2	—	A-6
vi)	Calcium ( as Ca ), percent by mass, <i>Max</i>	0.002 5	—	A-7
vii)	Arsenic ( as As ), percent by mass, <i>Max</i>	0.000 1	—	A-8
viii)	Sodium ( as Na ), percent by mass, <i>Max</i>	0.001	—	A-9
ix)	Chloride ( as Cl ), percent by mass, <i>Max</i>	0.000 04	—	A-10
x)	Sulphate ( as $SO_4$ ), per- cent by mass, <i>Max</i>	0.000 2	—	A-11
xi)	Phosphate ( as $PO_4$ ), per- cent by mass, <i>Max</i>	0.001	—	A-12

#### 4. PACKING AND MARKING

**4.1 Packing** — The material shall be packed as agreed to between the purchaser and the supplier.

**4.2 Marking** — The containers shall bear legibly and indelibly the following information:

- a) Name and grade of the material;
- b) Name of the manufacturer and his recognized trade-mark, if any;

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- c) Net mass,
- d) Date of manufacture, and
- e) Batch Number.

### **4.3 BIS Certification Marking**

The product may also be marked with Standard Mark

**4.3.1** The use of the Standard Mark is governed by the provision of 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 Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

## **5. SAMPLING**

**5.1** The method of drawing representative samples of the material and the criteria for conformity shall be as prescribed in Appendix B.

# **A P P E N D I X A**

( *Clause 3.2* )

## **METHODS OF TEST FOR BORIC ACID**

### **A-1. QUALITY OF REAGENTS**

**A-1.1** Unless specified otherwise, pure chemicals and distilled water ( *see* IS : 1070-1977\* ) shall be used in tests.

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

### **A-2. BORIC ACID**

#### **A-2.1 Reagents**

**A-2.1.1** *Mannitol ( or Sorbitol )* — neutral. The reagent shall satisfy the following test:

Five grams of the reagent dissolved in 50 ml of carbon dioxide-free water shall require for neutralization not more than 0.3 ml of 0.02 N sodium hydroxide solution using phenolphthalein solution as indicator.

**A-2.1.2** *Standard Sodium Hydroxide Solution* — 1 N.

\*Specification for water for general laboratory use ( *second revision* ).

**A-2.1.3 Phenolphthalein Indicator Solution** — Dissolve 0.1 g of the phenolphthalein in 60 ml of rectified spirit ( *see* 323-1959\* ) and dilute with water to 100 ml.

**A-2.2 Procedure** — Weigh accurately about 2 g of the dried material ( *see* A-3 ) and dissolve it in about 120 ml of water by heating, avoid boiling. Cool to room temperature, add approximately 15 g of mannitol (or sorbitol) and 0.4 ml of phenolphthalein indicator solution. Titrate the solution with standard sodium hydroxide solution to a distinct pink colour.

NOTE — To ensure that the correct titration end point is obtained, the following standard colours matching solution may be used for comparison with the solution being titrated:

Mix 50 ml of 3.81 g/l solution of disodium tetraborate (  $\text{Na}_2\text{B}_4\text{O}_7 \cdot 10\text{H}_2\text{O}$  ), 100 ml of water, 2.0 ml of hydrochloric acid ( 0.25 N ) and 0.4 ml of phenolphthalein indicator solution.

Equal volume of this solution and of the titrated liquid shall be compared in similar beakers.

### A-2.3 Calculation

$$\text{Boric acid, percent by mass} = \frac{6.183 V}{M}$$

where

$V$  = volume in ml of standard sodium hydroxide solution used in the titration, and

$M$  = mass in g of the dried material taken for the test.

## A-3. MOISTURE

### A-3.1 Apparatus

**A-3.1.1 Dish** — Circular, made of glass or aluminium, 100 mm in diameter and 100 mm deep, with tight-fitting cover or lid.

**A-3.2 Procedure** — Clean, dry and weigh the dish and cover. Weigh accurately about 10 g of the material as quickly as possible, the final mass being taken with the dish covered by the lid. Spread the boric acid evenly in the dish, by gentle tapping and shaking. Keep the dish with the lid off, in an oven at  $40 \pm 2^\circ\text{C}$  for 4 hours. At the end of the period transfer the dish to a vacuum desiccator and connect the desiccator to the pump. The final pressure inside the desiccator, when steady conditions have been attained, should be not more than 200 mm of mercury. After the minimum pressure has been reached, leave the desiccator connected to the vacuum for one hour. Disconnect the desiccator from the vacuum line and allow to stand under vacuum at room temperature for 12 hours.

\*Specification for rectified spirit ( *revised* ).

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Break the vacuum by admitting dry air through a guard tube containing fused calcium chloride. Weigh the dish with the cover on. Use the dried material for test in A-2.

### A-3.3 Calculation

$$\text{Moisture, percent by mass} = \frac{100 m}{M}$$

where

$$\begin{aligned} m &= \text{loss in mass in g on drying, and} \\ M &= \text{mass in g of the material taken for the test.} \end{aligned}$$

### A-4. WATER INSOLUBLE MATTER

**A-4.1 Procedure** — Dissolve 10 g of the material in 300 ml of hot water and heat on a steam-bath for one hour. Filter any undissolved residue through a Gooch crucible or a sintered glass crucible No. G4, wash it with hot water and dry at 105°C. Cool and weigh the residue till constant mass is obtained.

### A-4.2 Calculation

$$\text{Matter insoluble in water} = 100 \times \frac{M_1}{M}$$

where

$$\begin{aligned} M_1 &= \text{mass in g of the dried residue, and} \\ M &= \text{mass in g of the material taken for the test.} \end{aligned}$$

### A-5. TEST FOR HEAVY METALS

**A-5.0 Outline of the Method** — Heavy metals are determined by comparing the colour produced by the material with hydrogen sulphide solution against that produced by a standard lead solution.

#### A-5.1 Apparatus

**A-5.1.1 Nessler Cylinders** — 50 ml capacity ( see IS : 4161-1967\* ).

#### A-5.2 Reagents

**A-5.2.1 Dilute Hydrochloric Acid** — approximately 1 N.

**A-5.2.2 Hydrogen Sulphide Solution** — Saturated and freshly prepared.

**A-5.2.3 Standard Lead Solution** — Dissolve 0.1831 g of lead acetate [ Pb ( C<sub>2</sub>H<sub>3</sub>O<sub>2</sub> )<sub>2</sub> · 3H<sub>2</sub>O ] in 100 ml of water and clear any cloudiness that might appear with a few drops of acetic acid. Dilute the solution to 1 000 ml. Take 10 ml of this solution and make it up to 1 000 ml. One millilitre of this solution contains 0.001 mg of lead ( as Pb ).

\*Specification for Nessler cylinders

**A-5.3 Procedure** — Dissolve 1.000 g of the material in 25 ml of hot water, add 0.5 ml of dilute hydrochloric acid, heat to 80°C, transfer to a Nessler cylinder and add 10 ml of hydrogen sulphide solution. Make up the solution to 50 ml. Carry out a control test using 2 ml of standard lead solution and the same quantity of reagents in the same total volume of the reaction mixture. Compare the colour of the solutions to 10 minutes after the addition of hydrogen sulphide solution.

**A-5.3.1** The prescribed limit shall be taken as not having been exceeded if the intensity of the colour produced in the test with the material is not greater than that produced in the control test.

## A-6. DETERMINATION OF IRON

**A-6.0 Outline of the Method** — Iron is determined colorimetrically by visual comparison using potassium thiocyanate.

### A-6.1 Apparatus

**A-6.1.1 Nessler Cylinders** — 100 ml capacity ( see IS : 4161-1967\* ).

### A-6.2 Reagents

**A-6.2.1 Concentrated Hydrochloric Acid** — see IS : 265-1976†

**A-6.2.2 Ammonium Persulphate** — solid.

**A-6.2.3 Butanolic Potassium Thiocyanate** — Dissolve 10 g of potassium thiocyanate in 10 ml of water. Add sufficient *n*-butanol to make up the the volume to 100 ml and shake vigorously until the solution is clear.

**A-6.2.4 Standard Iron Solution** — Dissolve 0.702 g of ferrous ammonium sulphate [  $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$  ] in water and add 10 ml of dilute sulphuric acid ( 10 percent *v/v* ). Dilute the solution to one litre. Take 10 ml of this solution and dilute to 100 ml before use. One millilitre of this solution contains 0.01 mg of iron ( as Fe ).

**A-6.3 Procedure** — Dissolve 5 g of the material for SQ grade and 0.02 g in case of TECH grade in 25 ml of water and transfer to a Nessler cylinder. Add 2 ml of hydrochloric acid, 30 mg of ammonium persulphate and 15 ml of butanolic potassium thiocyanate solution. Make up to 100 ml, shake vigorously for 30 seconds and allow the liquids to separate. Carry out a control test in another Nessler cylinder with one ml of standard iron solution and the same quantities of other reagents in the same total volume of the reaction mixture. Compare the colour of the butanol layer in the two sets.

\*Specification for Nessler cylinders.

†Specification for hydrochloric acid ( *second revision* ).

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**A-6.3.1** The limits prescribed in Table 1 shall be taken as not having been exceeded if the colour of the butanol layer in the test with the material is not darker than the colour produced in the control test.

### A-7. CALCIUM

#### A-7.1 Apparatus

**A-7.1.1** *Platinum Basin*

#### A-7.2 Reagents

**A-7.2.1** *Methanol*

**A-7.2.2** *Hydrochloric Acid* — see IS : 265-1976\*.

**A-7.2.3** *Sulphuric Acid* — see IS : 266-1977†.

**A-7.2.4** *Strong Ammonia Solution*

**A-7.2.5** *Ammonia-Ammonium Chloride Buffer Solution*

**A-7.2.6** *Sodium Sulphide Solution*

**A-7.2.7** *EDTA Solution* — 0.01 M.

**A-7.2.8** *Methyl Thymol Blue Indicator Solution*

**A-7.3** Dissolve 2.5 g of the powdered sample in 25 ml of methanol in a platinum basin, add 5 drops of hydrochloric acid and evaporate to dryness. To the residue add 15 ml of methanol and 3 drops of hydrochloric acid and again evaporate to dryness. Moisten the residue with sulphuric acid and ignite. Dissolve the residue in 50 ml of water, add 10 ml of ammonia-ammonium chloride buffer solution, 25 ml of strong ammonia solution and 5 drops of sodium sulphide solution. Titrate with EDTA solution using methyl thymol blue as indicator, until the blue solution becomes colourless or grey. Not more than 0.15 ml of EDTA is required to pass the test.

### A-8. ARSENIC

**A-8.1 Procedure** — Dissolve 2.5 g of the material in 10 ml of the water. Carry out the test for arsenic as prescribed in IS : 2088-1971‡ using for comparison a stain obtained with 0.0025 mg of arsenic trioxide.

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\*Specification for hydrochloric acid (*second revision*).

†Specification for sulphuric acid (*second revision*).

‡Methods for determination of arsenic (*first revision*).

**A-8.1.1** The material shall be taken to have satisfied the requirement of the test if the length and intensity of the stain is not greater than that produced in the control test.

## **A-9. SODIUM**

**A-9.1** Determine the sodium by flame photometer at 589 nm according to the directions of the manufacturer of apparatus.

## **A-10. CHLORIDE**

**A-10.0 Outline of the Method** — Excess silver nitrate in nitric acid solution produces a white suspension of silver chloride on addition to a solution containing chloride iron.

### **A-10.1 Apparatus**

**A-10.1.1 Nessler cylinders** — see IS : 4161-1967\*.

### **A-10.2 Reagents**

**A-10.2.1 Silver Nitrate Solution** — 1 N. Mix equal volume of silver nitrate solution and concentrated nitric acid.

**A-10.2.2 Standard Chloride Solution** — Dissolve 1.6484 g of pure dry sodium chloride in water and dilute to 1 litre. Dilute 10 ml of this solution to one litre so that one ml contains 0.01 mg of chloride ( as Cl ).

### **A-10.3 Procedure**

**A-10.3.1** Boil about 100 ml of water in 250-ml pyrex beaker covered with a watch glass for 10 minutes, reject contents and rinse beaker with water. Weigh into the beaker 80 g of boric acid sample and add 200 ml of water. Cover, stand on a hot plate bringing just to boil to dissolve the crystals, then cool to room temperature.

**A-10.3.2** Filter all samples and standards through No. 4 sintered glass funnel using suction. Dilute 50 ml sample solution to 100 ml in a Nessler cylinder. Add 5 ml of silver nitrate reagent, at once stir thoroughly, stand in the dark for 10 minutes. Carry out test in another Nessler cylinder with 0.8 ml of standard chloride solution for SQ grade and 4 ml of standard chloride solution for TECH grade. Compare turbidity produced 5 minutes after the addition of silver nitrate.

**A-10.3.3** The relevant limit prescribed in Table 1 shall be taken as not having been exceeded if the turbidity produced in test with the material is not greater than the turbidity produced in the control test.

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\*Specification for Nessler cylinders.

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## **A-11. SULPHATE**

**A-11.0 Outline of the Method** — In acetic acid solution, sulphate ion reacts with excess barium chloride precipitating barium sulphate. The turbidity produced compared with standard solution.

### **A-11.1 Apparatus**

**A-11.1.1 Nessler cylinder** — see IS : 4161-1967\*.

### **A-11.2 Reagents**

**A-11.2.1 Acetic Acid** — 30 percent (*v/v*).

**A-11.2.2 Standard Sulphate Solution A** — Dissolve 0.1814 g of pure potassium sulphate in water and make up to 1 000 ml. One millilitre of the solution contains 0.1 mg of sulphate ( as  $\text{SO}_4$  ).

**A-11.2.3 Standard Sulphate Solution B** — Prepared by diluting 50 ml of standard sulphate solution A to 1 000 ml. One millilitre of the solution contains 0.005 mg of sulphate ( as  $\text{SO}_4$  ).

**A-11.2.4 Barium Sulphate Reagents** — Mix 15 ml of 0.5 percent (*m/v*) solution, 55 ml of water, 20 ml of sulphate free alcohol ( 95 percent ), add 5 ml of 0.0181 percent solution (*m/v*) of potassium sulphate and dilute to 100 ml with water and mix.

**A-11.2.5 Barium Chloride Solution** — 0.5 M. ( 122.1 g of barium chloride dissolved in water and made up to 1 000 ml ).

### **A-11.3 Procedure**

**A-11.3.1** Boil about 100 ml of water in a 500-ml conical beaker covered with watch glass for 10 minutes. Reject contents and rinse the beaker with water.

**A-11.3.2** Weigh 20 g of boric acid sample and add 10 ml of water. Cover and heat bringing just to boil to dissolve the crystals; then cool to room temperature. Filter 50 ml of the supernatant clear liquid to a Nessler cylinder. Add 2 ml of acetic acid and 5 ml of barium sulphate reagent, dilute to the mark. Carry out a control test in another Nessler cylinder with 4 ml of standard sulphate solution (*B*) and the same quantities of the reagents in the same total volume of the reaction mixture. Compare the turbidity produced in the two cylinders.

**A-11.3.3** The limit prescribed in Table 1 shall be taken as not having been exceeded if the turbidity produced in the test with the material is not greater than the turbidity produced in the control test.

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\*Specification for Nessler cylinders.



**A-12. PHOSPHATES**

**A-12.0 Outline of the Method** — Phosphates are determined by comparing the colour produced with ammonium molybdate reagent against a standard phosphate solution.

**A-12.1 Reagents**

**A-12.1.1 Dilute Sulphuric Acid** — approximately 5 N and 1 N.

**A-12.1.2 Phosphate Reagent No 1** — Dissolve without heating 5 g of ammonium molybdate in 100 ml of 1 N sulphuric acid.

**A-12.1.3 Phosphate Reagent No 2** — Dissolve without heating 0.2 g of *n*-methyl-*p*-aminophenol sulphate (metol) and 20 g of potassium metabisulphate in 100 ml of water.

**A-12.1.4 Standard Phosphate Solution** — Dissolve 1.43 g of potassium dihydrogen orthophosphate in 1 000 ml of water and store in a stoppered polythene bottle. Dilute 1 ml of this solution to 100 ml with water immediately before use. One millilitre of this diluted solution is equivalent to 0.01 mg of phosphate (as  $\text{PO}_4$ ).

**A-12.1.5 Standard Colour for Phosphate Test** — Mix one ml of standard phosphate solution with 20 ml of water, 3 ml of dilute sulphuric acid (1 N), one ml of phosphate reagent No. 1 and one ml of phosphate reagent No. 2 and place on water-bath at 60°C for 10 minutes.

**A-12.2 Procedure** — Dissolve one g of the material in 15 ml of water and neutralize with sulphuric acid (1 N). Add 3 ml of sulphuric acid (5 N), 1 ml of phosphate reagent No. 1 and one ml of phosphate reagent No 2 and place on a water bath at 60°C for 10 minutes. The material shall conform to the requirement of the test if any colour produced is not deeper than the standard colour prepared in **A-12.1.5**.

**A P P E N D I X B**

( Clause 5.1 )

**SAMPLING OF BORIC ACID****B-1. GENERAL REQUIREMENTS OF SAMPLING**

**B-1.0** In drawing 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.

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**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 shall be mixed thoroughly by suitable means.

**B-1.5** This sample shall be placed in suitable, clean and dry glass containers on which the material has no action

**B-1.6** The sample containers shall be of such a size that they are almost completely filled with the sample

**B-1.7** Each sample container shall be sealed airtight after filling and marked with full details of sampling, date of sampling and year of manufacture of the material

**B-2. SCALE OF SAMPLING**

**B-2.1 Lot** — All the containers of the same grade in a single consignment of the material, drawn from the same batch of manufacture shall constitute a lot. If a consignment is declared or known to consist of different batches of manufacture or of different types of containers, the containers of the same type belonging to the same batch shall be grouped together and each such group shall constitute a 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 chosen from the lot shall depend on the size of the lot ( $N$ ) and shall be as specified in Table 2.

**TABLE 2 NUMBER OF CONTAINERS TO BE SELECTED FOR SAMPLING**

LOT SIZE	NO. OF CONTAINERS TO BE SELECTED
$N$	$n$
(1)	(2)
5 to 50	3
51 to 200	4
201 to 400	5
401 to 650	6
651 to 1 000	7
1 000 and above	8

**B-2.3** The containers shall be chosen at random from the lot and in order to ensure randomness of selection, a random number table ( see IS : 4905-1968\* ) as agreed to between the purchaser and the supplier shall be used. In case such a random number table is not available, the following procedure shall be adopted:

‘Arrange all the containers in the lot in a systematic manner and starting from any container, count them as 1, 2, 3..... etc., up to  $r$  and so on, where  $r$  is equal to the integral part of  $N/n$ . Every  $r$ th container thus counted shall be taken out to give a sample for test.

### **B-3 TEST SAMPLES AND REFEREE SAMPLE**

#### **B-3.1 Preparation of Test Samples**

**B-3.1.1** Draw with an appropriate sampling instrument a small portion of the material from different parts of each container selected ( Table 2 ). The total quantity of the material drawn from each container shall be sufficient to conduct the tests for all the characteristics given under **3** and shall be not less than 0.5 kg.

**B-3.1.2** 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 well mixed up 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-3.1.3** The remaining portion of the material from each container ( after the quantity needed for the formation of composite sample has been taken ) shall be divided into three equal parts, each part weighing not less than 50 g. These parts shall be immediately transferred to thoroughly dried bottles which are then sealed airtight with stoppers and labelled with all the particulars of sampling given under **B-1.7**. The material in each such sealed bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets of samples in such a way that each set has an individual test sample representing each container selected. One of these three sets shall be sent to the purchaser, another to the supplier and the third shall be used as referee sample.

**B-3.2 Referee Sample** — The referee sample shall consist of the composite sample ( **B-3.1.2** ) and a set of individual samples ( **B-3.1.3** ) marked for this purpose and shall bear the seals of the purchaser and

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\*Methods for random sampling

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the supplier. These 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 the determination of boric acid shall be conducted on each of the individual samples.

**B-4.2** Tests for the remaining characteristics shall be conducted on the composite sample.

**B-5. CRITERIA FOR CONFORMITY**

**B-5.1 For Individual Samples** — The mean ( $\bar{X}$ ) and range ( $R$ ) of the test results for boric acid shall be calculated (range being the difference between the maximum and minimum values of the test results).

**B-5.1.1** The lot shall be declared to have satisfied the requirement for boric acid if the value of the expression ( $\bar{X} - 0.6 R$ ) is equal to or greater than the value specified in Table 1.

**B-5.2 For Composite Sample** — The test results on the composite sample shall meet the corresponding requirements specified in Table 1.

**B-5.3** A lot shall be declared as conforming to this specification if it satisfies the relevant requirements for each of the characteristics listed in 3 and in Table 1. 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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