

Instruction No. 03/2024-Customs

F.No.401/09/2024-Cus.III
Government of India
Ministry of Finance, Department of Revenue
(Central Board of Indirect Taxes & Customs)

Room No. 229A, North Block, New Delhi
Dated : 19.02.2024

To

All Principal Chief Commissioners/ Chief Commissioners,
(Customs/ Customs (Preventive) /Customs and Central Taxes),
All Principal Commissioners/ Commissioners (Customs/ Customs (Preventive),
All Principal Director Generals/Director Generals under CBIC.

Madam/Sir,

Subject: - Compliance of imported consignments of Boric Acid (Technical Grade) with notified Bureau of India Standards (Standards for Boric Acid) Order, 2019-reg

Reference is invited to the Ministry of Agriculture & Farmers Welfare Order S.O. No. 1766 (E) dated 16.05.2019, the Bureau of Indian Standards (Standards for Boric Acid) Order, 2019 vide which Boric Acid has been brought under BIS Standard IS 10116:2015 (copy enclosed). Copy of IS 10116:2015 (Reaffirmed in 2020) for Boric Acid is also enclosed.

2. The salient feature of the Bureau of Indian Standard IS 10116:2015 (Reaffirmed in 2020) for Boric Acid (Technical Grade) are as under:

- a. Presence of Polishing Compounds comprising of Sulphate and Chloride in Boric Acid (Technical Grade) within the specified range.
- b. Packing of Boric Acid (Technical Grade) in jute bags with suitable liner inside.

3. It is requested that necessary action may be taken to sensitize officers under your jurisdiction to undertake checking of all imported consignments of Boric Acid to ensure strict compliance with the specifications prescribed in IS 10116:2015 (Reaffirmed in 2020) for Boric Acid (Technical Grade).

4. The difficulties, if any, may be brought to the notice of the Board.

5. Hindi version follows.

Encls: as stated above.

Yours faithfully,


(Radhe Krishna)
Under Secretary (Cus-III)
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Copy to, The Agriculture Commissioner, Department of Agriculture and Farmers Welfare, Krishi Bhawan, New Delhi-110001.

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MINISTRY OF AGRICULTURE AND FARMERS WELFARE
(Department of Agriculture, Co-operation and Farmers Welfare)

ORDER

New Delhi, the 16th May, 2019

S.O. 1766 (E).— In exercise of the powers conferred by sub-section (1) and (2) of section 16 of the Bureau of Indian Standards Act, 2016 (11 of 2016), the Central Government, after consulting the Bureau of Indian Standards, is of the opinion that it is necessary and expedient so to do in the public interest hereby makes the following Order, namely: -

1. Short title and commencement. - (1) This Order may be called the Bureau of Indian Standards (Standards for Boric Acid) Order, 2019.

(2) This Order shall come into force on the date of its publication in the Official Gazette.

2. Compulsory use of Standard Mark. - Goods or articles specified in column (1) of the Schedule shall conform to the corresponding Indian Standard given in column (2) thereof and shall bear the Standard Mark under a licence issued by the Bureau of Indian Standards in accordance with the Bureau of Indian Standards (Conformity Assessment) Regulation, 2018:

Provided that nothing in this Order shall apply in relation to goods or articles specified in this clause which is meant for export and which conforms to the specification required by the country of its export.

3. Certification and enforcement. - The Bureau of Indian Standards shall be the certifying and enforcing authority for the enforcement of the standards specified under clause 2, and may designate an officer not below the rank of the General Manager of the District Industries Center in the Department of Industries of the State Government to be the enforcing authority for the purpose of this Order.

SCHEDULE

Standards for Boric Acid

Goods or articles	Indian Standard	Title of Indian Standard
(1)	(2)	(3)
Boric Acid	IS 10116:2015	Boric Acid -Specification

[F. No. 13028/02/2018-PP-I]

ATISH CHANDRA, Jt. Secy.

Explanation. - For the purposes of this Schedule, the latest version of the Indian Standards, including the amendments thereof, as notified by the Bureau of Indian Standards from time to time, shall apply from the date as notified by the Bureau of Indian Standards.

बोरिक एसिड — विशिष्टि
(पहला पुनरीक्षण)

Boric Acid — Specification
(First Revision)

ICS 71.060.30

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BUREAU OF INDIAN STANDARDS
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April 2015

Price Group 5

FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Inorganic Chemicals Sectional Committee had been approved by Chemical Division Council.

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 which is covered under a separate standard, IS 263 : 1990 'Boric acid for cosmetic industry — Specification'.

This standard was first published in 1982. In this revision, requirement for moisture has been modified for technical grade boric acid. Also, a requirement for polishing compounds has been incorporated for the technical grade boric acid prescribing the limits for chloride and sulphate. Packing clause has been also appropriately modified to include packing of technical grade boric acid in jute bags with liners.

The composition of the Committee responsible for formulation of this standard is given in Annex C.

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 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off values should be the same as that of the specified value in this standard.

Indian Standard

BORIC ACID — SPECIFICATION

(First Revision)

1 SCOPE

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, enamel industries, carom board powder, talc powder and ramming mass.

2 REFERENCES

The standards listed below contain provisions which, through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	Title
265 : 1993	Specification for hydrochloric acid <i>fourth revision</i>)
266 : 1993	Specification for sulphuric acid (<i>third revision</i>)
323 : 2009	Rectified spirit for industrial uses — Specification (<i>second revision</i>)
1070 : 1992	Reagent grade water — Specification (<i>third revision</i>)
2088 : 1983	Methods for determination of arsenic (<i>second revision</i>)
4161 : 1967	Specification for Nessler cylinders
4905 : 1968	Methods for random sampling

3 GRADES

There shall be three grades of the material depending on the use, namely,

- a) *Special quality (SQ)*, for use in capacitors, electronics nuclear energy, foundries and fire proofing of workman's apparel;
- b) *Technical (TECH)*, for use in glass, ceramics, explosives, refractories, paints, chemicals, dye intermediates, electroplating, leather, adhesives and enamel industries, talc powder, ramming mass and as carom board powder; and

- c) Explosive.

4 REQUIREMENTS

4.1 Description

4.2 Special quality grade (SQ) material shall be in the form of granules only. The technical grade (TECH) and explosive grade material shall be in the form of granules, powder, crystals or flakes. Material of all grades shall be free from odour, dirt, foreign matter and visible impurities.

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

4.4 Additional Requirement for Technical Grade Boric Acid

4.5 The technical grade boric acid shall also comply with the following additional requirements of polishing compounds when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col 5 of the Table 1.

- a) Chloride (as Cl), : 0.05 – 0.15
percent by mass
- b) Sulphate (SO₄), : 0.15 – 0.25
percent by mass

4.6 Boric Acid for Use in Explosives

The material shall meet the requirements for boric acid and moisture for technical grade (*see*, Table 1, col 4) and shall also conform to the following additional requirements when tested according to the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col 5 of the Table 1.

- a) Iron (as Fe) : 0.05
percent by mass, *Max*
- b) Chloride (as Cl), : 0.03
percent by mass, *Max*
- c) Sulphate (SO₄), : 0.10
percent by mass, *Max*

5 PACKING AND MARKING

5.1 Packing

The special quality and explosive grade material shall

Table 1 Requirements for Boric Acid
(Clauses 4.2, 4.3 and 4.4)

Sl No.	Characteristic	Requirement		Method of Test, Ref to Clause No.
		Special Quality (SQ)	Technical (TECH)	
(1)	(2)	(3)	(4)	(5)
i)	Boric acid (as H_3BO_3), percentage by mass, <i>Min</i>	99.9	99.5	A-2
ii)	Moisture percent by mass, <i>Max</i>	—	0.2	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), percentage 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 (Cl), percent by mass, <i>Max</i>	0.000 04	—	A-10
x)	Sulphate (as SO_4), percent by mass, <i>Max</i>	0.000 2	—	A-11
xi)	Phosphate (PO_4), percent by mass, <i>Max</i>	0.001	—	A-12

be packed as agreed to between the purchaser and the supplier. The technical grade boric acid shall be packed in jute bags with suitable liner inside, as agreed to between the purchaser and the supplier.

5.2 Marking

The containers shall bear legibly and indelibly the following information:

- Name and grade of the material;
- Name of the manufacturer and his recognized trade-mark, if any;
- Net mass;
- Date of manufacture; and
- Batch Number.

5.3 BIS Certification Marking

The product may also be marked with Standard Mark.

5.3.1 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.

6 SAMPLING

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

ANNEX A

(Clauses 4.2, 4.3, 4.4 and Table 1)

METHODS OF TEST FOR BORIC ACID

A-1 QUALITY OF REAGENTS

Unless specified otherwise, pure chemicals and distilled water (see IS 1070) 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 gram 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 an indicator.

A-2.1.2 Standard Sodium Hydroxide Solution — 1 N.

A-2.1.3 Phenolphthalein Indicator Solution — Dissolve 0.1 g of the phenolphthalein in 60 ml of rectified spirit (see IS 323) 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 of standard sodium hydroxide solution used in the titration, in ml; and

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

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 h. 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 1 h. Disconnect the desiccator from the vacuum line and allow to stand under vacuum at room temperature for 12 h. 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

m = loss in mass on drying, in g; and

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

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 1 h. 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

$$\text{Matter insoluble in water, percent by mass} = \frac{100 M_1}{M}$$

where

M_1 = mass of the dried residue, in g; and

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

A-5 TEST FOR LEAD

A-5.0 Outline of the Method

Heavy metal is 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).

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 [$\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O}$] 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 this solution and make it up to 1 000 ml. One millilitre of this solution contains 0.001 mg of lead (as Pb).

A-5.3 Procedure

Dissolve 1 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 min 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).

A-6.2 Reagents

A-6.2.1 *Concentrated Hydrochloric Acid* — (see IS 265).

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 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 [$\text{FeSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$] 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 explosive 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 s and allow the liquids to separate. Carry out a control test in another Nessler cylinder with 1 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.

A-6.3.1 The limits prescribed in Table 1 for SQ grade and 4.4 for explosive grade 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.

A-7.2.3 *Sulphuric Acid* — see IS 266.

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 sulphate 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 using for comparison a stain obtained with 0.002 5 mg of arsenic trioxide.

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

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 ion.

A-10.1 Apparatus

A-10.1.1 Nessler Cylinders — see IS 4161.

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 A — Dissolve 1.648 4 g of pure dry sodium chloride in water and dilute to 1 litre. Dilute 100 ml of this solution to one litre so that one ml contains 0.1 mg of chloride (as Cl).

A-10.2.3 Standard Chloride Solution B — Dissolve 1.648 4 g of pure dry sodium chloride in water and dilute to 1 litre. Dilute 10 ml of this solution to one litre so that 1 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 min, reject contents and rinse beaker with water. Weigh into the beaker 80 g of boric acid sample and add 200 ml of water. Cover and place it on a hot plate bringing just to boil to dissolve the crystals and then cool to room temperature.

A-10.3.2 For SQ Grade

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 min. Carry out test in another Nessler cylinder with 0.8 ml of standard chloride solution B for SQ grade. Compare turbidity produced 5 min after the addition of silver nitrate.

A-10.3.3 For Technical Grade and Explosive Grade

Filter all samples and standards through No. 4 sintered glass funnel using suction. Dilute 2.5 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 min. Carry out test in another Nessler cylinder with 3 ml of standard chloride solution A for explosive grade. For technical grade, carry out test with 5 ml and 15 ml of standard chloride solution A in two separate Nessler cylinders. Compare turbidity produced 5 min after the addition of silver nitrate.

A-10.3.4 For SQ and Explosive Grade

The relevant limit prescribed in Table 1 for SQ grade (see 4.4) for explosive grade 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.

A-10.3.5 For Technical Grade

The relevant limit prescribed in 4.3 shall be taken as within the range, if the turbidity produced in test with the material lies in between the turbidity produced in the two control test carried out with 5 ml and 15 ml of standard chloride solution A.

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 is compared with the standard solution.

A-11.1 APPARATUS

A-11.1.1 Nessler Cylinder — see IS 4161.

A-11.2 Reagents

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

A-11.2.2 Standard Sulphate Solution A — Dissolve 0.181 4 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 SO_4).

A-11.2.3 Standard Sulphate Solution B — It is 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 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 0.018 1 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 min. Reject contents and rinse the beaker with water.

A-11.3.2 For SQ Grade

Weigh 20 g of boric acid sample and add 100 ml of water. Cover and heat bringing just to boil to dissolve the crystals; then cool to room temperature. Filter the solution and take 50 ml of the 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 For Technical Grade and Explosive Grade

Weigh 20 g of boric acid sample and add 100 ml of water. Cover and heat bringing just to boil to dissolve the crystals; then cool to room temperature. Filter 5 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 10 ml of standard sulphate solution (A) and the same quantities of the reagents for explosive grade. For technical grade, carry out a control test with 15 ml and 25 ml of standard sulphate solution (A) in two separate Nessler cylinders. In the same total volume of the reaction mixture compare the turbidity produced in the control and test Nessler cylinders.

A-11.3.4 For SQ and Explosive Grade

The relevant limit prescribed in Table 1 for SQ grade and 4.4 for explosive grade 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.

A-11.3.5 For Technical Grade

The relevant limit prescribed in 4.3 shall be taken as within the range, if the turbidity produced in test with the material lies in between the turbidity produced in the two control test carried out with 15 ml and 25 ml of standard sulphate solution A.

A-12 8PHOSPHATES

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 dilute solution is equivalent to 0.01 mg of phosphate (as PO_4).

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

A-12.2 Procedure

Dissolve 1 g of the material in 15 ml of water and neutralize with sulphuric acid (1N). Add 3 ml of sulphuric acid (5 N), 1 ml of phosphate reagent No. 1 and 1 ml of phosphate reagent No. 2 and place on a water bath at 60°C for 10 min. 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.

ANNEX B

(Clause 6.1)

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 Sample 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 shall be mixed thoroughly by suitable means.

B-1.5 The 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 1.

Table 2 Number of Containers to be Selected for Sampling
(Clause B-2.2)

Sl No.	Lot Size N	No. of Containers to be Selected n
(1)	(2)	(3)
i)	5 to 50	3
ii)	51 - 200	4
iii)	210 - 400	5
iv)	401 - 650	6
v)	651 - 1 000	7
vi)	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) 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 (*see* 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 4 and Table 1 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 and 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 labeled 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 (*see* **B-3.1.2**) and a set of individual samples (*see* **B-3.1.3**) 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 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

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requirement for boric acid if the value of the expression ($-0.6 R$) is equal to or greater than the value specified in Table 1.

B-5.2 For Composite Sample

The test result 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 4 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.

ANNEX C

(Foreword)

COMMITTEE COMPOSITION

Inorganic Chemical Sectional Committee, CHD 01

<i>Organization</i>	<i>Representative(S)</i>
Central Salt and Marine Chemicals Research Institute, Bhavnagar, Gujarat	SHRI M. R. GANDHI (<i>Chairman</i>) DR R. S. SHUKLA (<i>Alternate</i>)
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