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PROVISIONAL SPECIFICATION FOR AMORPHOUS BORON

(Second Revision)

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6.2 **Sampling**

6.2.1 The representative sample of 50 g shall be taken from each batch / lot. The number of samples to be drawn from the batch / lot shall be decided mutually by Quality Assurance Authority and the manufacture/suppliers.

6.2 2 The samples shall be placed in a clean, dry and air tight container which has no action on the material.

6.3 **Test Requirement**

Samples taken from the lot / batch shall comply with Clause 3 and 4 above and in addition shall satisfy the test requirements as given in Table No.1

TABLE –1

Test requirements for Boron, amorphous

Sr. No.	Characteristics	Passing Standard			Test Method
		Grade II		Grade I	
		(A)	(B)		
1.	Volatile matter (to constant weight at 105 ± 2^0C), percent by mass	0.5 max	0.5 max	0.5 max	As per IND/ME/980 (prov.)
2.	Total boron content, Percent by mass	85 - 88	85 - 88	91 (minimum)	Appendix 'A'
3.	Total Magnesium content, percent by mass	7-11	7-11	5 (maximum)	Appendix 'A'
4.	Bulk density, g/cc	0.6 min	0.2 min	0.6 min	Appendix 'B'
5.	Particle size (surface weighted mean), µm	0.1 – 2.0	2.0 - 10.0	0.1 – 2.0	By LASER diffraction method on Malvern's Master Sizer 2000. Appendix 'C'.

APPENDIX 'A'

DETERMINATION OF TOTAL BORON AND MAGNESIUM CONTENT OF AMORPHOUS BORON

1. **Apparatus / Equipment**
   a) Platinum crucible.
   b) Muffle furnace.
   c) 250 ml volumetric flasks and beakers.

2. **Reagent:** All reagents are of A.R. grade except mentioned otherwise. Distilled water is used for preparation of solution and during titration.
   a) Sodium Carbonate
   b) 1:1 HCl
   c) 6 N NaOH
   d) 0.1 N HCl
   e) 0.1 N NaOH
   f) 0.05 N NaOH
   g) Mannitol
   h) Ammonia buffer solution (Reagent B) Qualigen make.
   i) 0.01 M EDTA.
   j) Indicator: Methyl Red, Phenolphthalein and Eriochrome Black T.

3. **Preparation of sample solution**

Weigh accurately about 0.150 g of the boron powder (volatile matter free) and place it, layer by layer, with 8 g of dry sodium carbonate in platinum crucible (five to six boron powder layers are sandwiched between sodium carbonate layers). Transfer the crucible with lid into muffle furnace. Start heating and maintain the temperature at 850^0C for half an hour. After cooling, take out the crucible and dissolve the fused mass in 50 ml of HCl (1:1). Transfer the solution to 250 ml volumetric flask and make up to the mark with distilled water. Use this stock solution for determination of total boron and magnesium content.

4. **Determination of total boron content**

Out line of the method

The stock solution mainly contains hydrochloric acid and boric acid. HCl is neutralised with NaOH solution and then mannitol is added. It forms a complex with boric acid liberating equivalent amount of protons. This solution is titrated against NaOH solution using an acid-base indicator.

Procedure

Pipette out 25 ml of the stock solution (as prepared in clause 3 above) into a 500 ml conical flask and add few drops of methyl red indicator. Neutralise the excess acid by slow addition of ~ 6 N NaOH with the indication of change of colour from pink to yellow. Add 0.1 N HCl drop wise until pink colour reappears and then add excess 5 ml of ~ 0.1 N HCl to this solution. Place the flask on hot plate. Just heat to boil to remove dissolved carbon dioxide and allow to cool. Neutralise the solution using 0.1 N NaOH till colour changes from pink to yellow and then just acidify the solution with one or two drops of 0.1N HCl. The colour changes from yellow to pink. Again neutralise it with 0.05N NaOH solution till the colour changes to a clear lemon yellow. Add 20 to 25 drops of phenolphthalein indicator and 5 g of mannitol. Ensure the dissolution of mannitol and then titrate against 0.05 N NaOH solution till a permanent pink colour persists. Note the burette reading R1. Carry out the blank experiment and note the burette reading R2.

Calculation

$$\text{Total Boron percent by mass} = \frac{(R1-R2) \times N \times F \times 10}{W}$$

Where R1 - ml of titrant (NaOH) required for neutralising protons (H+) liberated on the addition of mannitol.

R2 - ml of titrant (NaOH) required for 'Blank Titration'.

N - Normality of standardised titrant (NaOH).

F - 1.082 (conversion factor for Boron)

W - Weight of the sample in g.

Run the titration in duplicate and report total boron content, percent by mass, as the mean of the two values.

5. **Determination of Magnesium Content**

Procedure

Pipette out 25 ml of the stock solution (as prepared in clause 3 above) into a conical flask. Neutralise it with ~ 6N NaOH using methyl red as indicator until colour changes from pink to yellow. Add 0.1 N HCl dropwise till the solution becomes pink. Then neutralise with 0.1 N NaOH until the yellow colour reappears. Add 2 ml of ammonia buffer solution (Reagent B). Titrate the solution against 0.01 M EDTA solution using Eriochrome Black-T as an indicator till colour changes from wine red to ink blue. Note the burette reading (R1). Carry out a 'Blank titration' and note the burette reading (R2).

Calculation

$$\text{Magnesium, percent by mass} = \frac{(R1 - R2) \times F \times M \times 10}{W}$$

Where R1 - ml of EDTA required for Mg

R2 - ml of EDTA required for Blank titration

F - 2.432 (conversion factor for Magnesium)

M - Molarity of EDTA (0.01 M)

W - Weight of sample in g

Run the titration in duplicate and report the Magnesium, percent by mass, as the mean of the two values.

APPENDIX 'B'

DETERMINATION OF BULK DENSITY (B.D.)

Bulk density of the boron powder is determined using "Densitometer" (Make – Indian Equipment Corporation, Mumbai).

Procedure: Weigh accurately clean, dry and empty 50 ml graduated glass measuring cylinder on analytical balance (W1). Fill it with the boron powder up to 50 ml mark gently and without tapping and reweigh (W2). Clamp the cylinder tightly in the Densitometer. Close the mouth of the cylinder with aluminium foil or glossy paper to avoid spillage. Set the tapping-timer switch to nine minutes. (The apparatus offers 32 taps per minute with the height of fall from 8 cm). Switch on the Densitometer. Remove the cylinder after the instrument is switched off automatically. Note the volume (V) in cubic centimetre (cc) of the boron powder. Calculate the Bulk Density (B.D.) as follows.

$$\text{B.D., g/cc} = \frac{W2 - W1}{V}$$

Where

W1 - Weight of the empty cylinder in g.

W2 - Weight of the cylinder filled with boron powder in g

V - Volume of boron powder in cubic centimetre.

Run the experiment in the duplicate. Report the B.D. as the mean of the two values.

APPENDIX 'C'

DETERMINATION OF PARTICLE SIZE

Particle size of boron powder is determined by the instrument "Master Sizer 2000" of Malvern Instruments Ltd., U.K. The procedure followed is as per the instruction manual provided by the firm.

Test conditions are as follows:

- Dispersion medium: distilled water
- Rate of stirring : 2000 rpm
- Dispersion aid : Nonidet P-40 (Merck)

"Surface weighted mean D [3,2]" is reported as particle size of the sample.