CONTROLLED COPY No. 1

DC-3431-ME
10-2-97.

DC 3378-ME
13.4.98

DC 3844-ME
04/04/2005

IND/ME/980(PROV)

IND/ME/980:2016

मोहरबन्द/अनारक्षित/डिविजीठ
प्रमाणित सच्ची प्रति।
Certified Correct Copy of
Sealed SPECIFICATION
दिनांक/Attested date
....22.4.96.....

कृते नियंत्रक गु.आ.इ.न (मै.वि.)
खडकी, पुणे-411003.
FOR CONTROLLER QOA (ME)
KHADKI, PUNE-411 003.

DC-6033-ME
dt 08-06-2017

BORON POWDER

DSCATNO
TYPE-I – 6810-001135
Type-II – 6810-001136

*Authority - CQA(ME) Letter no. CQA(ME)/7212/63 dt 19/08/2017

ISSUED BY

CONTROLLERATE OF QUALITY ASSURANCE

(MILITARY EXPLOSIVES)

AUNDH ROAD, KHADKI PUNE -411003

IND/ME/980(PROV)

AMENDMENT RECORD

AMENDMENT DC NO.	DATE	AUTHORITY LETTER	CLAUSES AFFECTED	REMARKS
3844-ME,	04/04/2005	CQA(ME)7212/61 dt 16/4/05	The existing specification 'BORON POWDER' is amended as under Page 12 For Existing formula against Purity. Read $\frac{(V_1 - V_2) \times F \times 0.1082 \times 5}{m}$ Page 13 For Existing formula against Purity Read: $\frac{\text{Vol of NaOH} \times F \times 1.082}{m}$	Amended (V) Malvelekar 4/6/05 (V.J. MALVELEKAR) AF.

C O N T E N T S

THIS SPECIFICATION OR ANY PATTERN, DRAWING OR ANY OTHER INFORMATION ISSUED IN CONNECTION THEREWITH MAY ONLY BE USED FOR A SPECIFIC ORDER PLACED BY THE COMPETENT AUTHORITY. IT IS NOT TO BE USED FOR ANY OTHER PURPOSE WHATSOEVER WITHOUT THE EXPRESS WRITTEN SANCTION OF THE DIRECTOR GENERAL OF QUALITY ASSURANCE, MINISTRY OF DEFENCE, NEW DELHI 110011.

0 FOREWORD

0.1 This specification has been prepared by the Controllerate of Quality Assurance (Military Explosives), Aundh road, Khadki, Pune 411003.

0.2 For additional copies or any other enquiry regarding this Specification , reference should be made to theQuality Assurance Authority (i.e.CQA(ME) Kirkee)

1 S C O P E

1.1 This specification is meant to govern manufacture , supply and Quality Assurance of Boron Powder.

1.2 The material is suitable for use in the manufacture of Pyrotechnic compositions. Grade I material is used in illuminating Composition No.47 and Gr II material is used in 155 mm smoke composition.

2 RELATED SPECIFICATION & DOCUMENTS

2.1 The related specifications and document as mentioned in clause 2.2 are those applicable at the date of publication of this specification. It is contractors/manufacturer's responsibility to confirm their current applicability and to obtain from CQA(ME), Aundh road, Khadki, Pune 411003 information concerning any change that may be necessary due to cancellation, replacement or supersession of any of these documents.

2.2 The following specifications have been referred to in the preparation of this IND/ME specification.

i)	CS 5507	-	Boron Amorphous
ii)	Bofors F1329-000472b	-	--------"-------
iii)	ERDL/TRIM/IGN/RM/I	-	Boron

2.3 Copies of this specification and other related specifications are obtainable on payment basis as follows.

IND/ME	-	Controllerate of Quality Assurance (Military Explosives) Aundh road, Khadki ,pune-411003
IS	-	Bureau of indian standards Manak Bhavan, 9,B.S.Zafar marg New Delhi 110002
CS & Bofors		Directorate of standardisation 'H' Block M. of D. New DElhi-110011
ERDL SPECN		The Director, HEMRL Sutarwadi Pune - 411 021

3 MATERIAL / FINISH

3.1 The Boron Powder shall be in the the form of fine dark brown powder,free from gritty particles, Visible impurities and foreigh matter.

4 MANUFACTURE

4.1 The Boron Powder shall be manufactured by a process which has received authoritative approval. The Quality Assurance officer / Quality Assurance Authority shall be informed regarding the process used & shall be informed with prior notification of any proposed deviation therefrom.

All The deviations from the approved process, however slight, shall be recorded immediately and all the material affected shall be set aside pending the decision of QA Officer / QA Authority.

5 TENDER SAMPLE

5.1 The Contractor/manufacturer shall submit a tender sample of 25 g induplicate free of charge from the same batch of manufacture and conforming to this specification.

6. QUALITY ASSURANCE

6.1 INSPECTION

6.1.1 The Boron Powder Amorphous and the packages in which it is contained shall be subject to Quality Assurance by and to the final approval of Quality Assurance Officer/Quality Assurance Authority.

6.1.2 Samples of the material and of the packages may be taken from any portion of the batch/lot of manufacture.

6.1.3 If on examination, any sample be found not to conform to this specification, the whole batch/lot/consignment shall be rejected .

6.1.4 The foregoing provisions shall equally apply contractors and subcontractors, if any.

6.2 SAMPLING

6.2.1 The representative sample of 25 g shall be taken from each package from the batch /lot. The number of samples to be drawn from the batch/lot shall be as given below.

Lot Size			No of Containers to be selected
3	to	50	3
51	to	200	4
201	to	400	5
401	to	650	6
651	to	over	7

6.2.2 Samples shall be drawn in A/C room.

6.2.3 The contents to each container selected for sampling shall be mixed thoroughly before drawal of sample.

6.2.4 The samples shall be placed in sound, clean, dry air tight containers which have no action on the material.

6.2.5 The sample container shall be of such size that it is almost completely filled by the sample. Each container shall be sealed air tight and stored at 27±2 degree Centigrade.

6.3 **TEST REQUIREMENTS**

6.3.1 Samples taken from any portion of the batch / lot shall comply with the clause 3.1 above and in addition shall satisfy the following test requirements.

SL.NO	Characteristic	PASSING STANDARD		TEST METHOD
		Grade I	Grade II	
1	Purity % Min	95	90	Appendix 'A'
2	Volatile matter % Max	0.5	0.5	Appendix 'B'
3	Avarage particle Size micrometre			Appendox 'C'
	Min	0.8	0.4	
	Max	0.9	1.2	

SL.NO	Characteristic	PASSING STANDARD Grade I	GRADE II	TEST METHOD
4	Impurities			
	a) Mg % Max	1.2	5.0	
	b) Fe % Max	0.2	–	Appendix 'D'
	c) Si % Max	0.2	–	
	d) Oxygen % Max	4.2	–	
	e) Nitrogen % Max	1.5	–	
	f) Sodium % Max	–	0.50	
5	Specific gravity at 27±2 deg C	2.44	–	Appendix 'E'

7. SUPPLIER'S INSPECTION OF STORES / CONSIGNMENT

7.1 Before tendering the stores for inspection, The supplier shall carry out a thorough inspection of each delivery to satisfy himself that the store fully conforms to this specification and shall render a certificate to that effect to the QA Officer / QA Authority.

8. WARRANTY

8.1 The stores supplied against the contract shall deem to have been warranted against defective material and performance by the contractor/manufacturer for a period of 12 months from the date of receipt of the store at the consignee's end and if during this period any of the stores supplied is found defective, the same shall be replaced by the Contractor/Manufacturer free of charge at the consignee's premises.

9 PACKING

9.1 Boron Powder shall be packed in polythene bag having film thickness 0.13 mm minimum. The mouth of the bag shall be properly heatsealed. The bag shall then be placed in a plywood drum or fibre drum.

9.2 The inclusion of any foreign matter or impurities in any of the packages shall render the whole consignment liable to rejection.

9.3 Any other form of packages shall have prior approval of QA Officer / QA Authority .

10. MARKING

10.1 All the packages containing the material shall be durably and legibly marked with the following details.

a) Nomenclature and specification number.

* b) Name and address of the consignee.

* c) A/T No. or S.O. NO. and date.

d) Consignment.

e) Lot/batch no. and date of manufacture.

f) Gross and net mass.

g) Consecutive no. of package and total no. of packages in the consignment.

h) Date of supply.

* i) Contractor's initials or recognised trade mark.

* Not applicable when the store is manufactured in Ordnance Factories.

10.2 In addition to above the QA Officer may suggest some more marking/identification suitable at the time of inspection.

10.3 The paint used for marking shall conform to IS 138-1981.

11 DEFENCE STORES CATALOGUE NUMBER

11.1 The defence store catalogue number allotted to this store is _ _ _ _ _ _ _ _ _ _ _ .

12. SAFETY OF OPERATIONS

12.1 Nothing in this specification shall relieve the manufacturer/ user/ Contractor of his responsibility for the safety of his operations during manufacture, handling storage & transport.

13. SUGGESTIONS FOR IMPROVEMENT

13.1 Any suggestion for improvement in this document shall be forwarded to the Controller, CQA(ME) Aundh Road, Khadki Pune 411003.

sd/ ----------

(V.R. SONONE)

CONTROLLER

CQA (ME) AUNDH ROAD

Khadki, PUNE 411003.

APPENDIX 'A'

PURITY

1. APPARATUS

a) Platinum crucible

b) Fisher burner

c) 250 ml volumetric flask

2. REAGENT

a) Sodium Carbonate

b) 1:1 Hydrochloric acid

c) 6 N Sodium hydroxide

d) 0.1 N Hydrochloric acid

e) 0.1 N Sodium hydroxide

f) Mannitol

g) Indicator : Methyl Red, Phenolpthalein

3. PROCEDURE

A sample of 0.10 to 0.15 g shall be accurately weighed in a tared platinum crucible. To the crucible 1.5 g of sodium carbonate shall be added and mixed well with a glass rod, taking care that no part of the mixture adheres to the rod. The crucible shall be covered with lid and heated with a low flame of a Fisher burner for approximately five minutes, taking care to avoid loss by spurting or frothing. The heat shall be gradually increased to fuse the mixture and held at fusion temperature for five minutes. The mixture shall be cooled and then dissolved by placing the crucible and cover in a covered 400 ml beaker containing 50 ml of 1:1 hydrochloric acid. The crucible and cover shall be removed and washed with distilled water. When digestion is complete, the washings are to be combined with the solution . The solution shall be transferred to a 250 ml Volumetric Flask and diluted to the mark. A 50 ml aliquot shall be pipetted into a 250 ml Erlenmeyer Flask , three drops of methyl red indicator shall be added and the excess hydrochloric acid neutralised with 6 N Sodium hydroxide. The pH shall be adjusted to the methyl red neutral point with 0.1 N Sodium hydroxide or 0.1 N hydrochloric acid, whichever is necessary, and then 5 ml of

hydrochloric acid added. A few glass beads shall be added, the solution boiled for three minutes to expel carbon dioxide, cooled in a water bath, and again neutralised to the methyl red neutral point with carbon dioxide free 0.1 N Sodium hydroxide. Six to ten g of reagent grade mannitol and 20 drops of phenolphthalein indicator shall be added and titrated with 0.1 N sodium hydroxide to the first Phenolphthalein pink coloration. A blank experiment with the reagents mentioned above shall be run following the above procedure exactly. The purity shall be calculated as follows :

DC 3844-DDE 4/4105

$$\text{Purity} = \frac{(V_1 - V_2) \times F \times 0.1082 \times 5}{m}$$

(V1 - V2) (F) (1.082) X 100

(X) 4/6105

Where

V1 = ml. of Sodium hydroxide required/used for sample

V2 = ml. of Sodium hydroxide required/used for blank

F = Factor of Sodium hydroxide N/10 NaOH

m = Mass of Sample in aliquot, g g

Alternate Method

Place 0.2 g of dried sample in a 30 ml capacity Nickel crucible. Add 8.0 g Sodium Carbonate (AR) and mix until mixture is homogeneous and begin fusion over a Fischer burner at a low temperature. Gradually increase the temperature to bright red hot and then keep in the muffle at 800 degree Centigrade for 2 hours. After removal and cooling add 200ml of distilled water and keep it overnight. Filter through Whatman NO.41 filter paper. Neutralise the filtrate with dil H_2SO_4 using Methyl red indicator when pH 8.5 is attained. (The colour changes from yellow to just pink). Heat and boil till carbon dioxide is removed completely. Cool and make the volume to 500 ml.

Take 50 ml of solution and 1 g of mannitol per 10 ml of solution or 10 to 15 ml of glycerol and titrate against N/10 NaOH using Phenolphthalein indicator.

$$C_3H_5OH + HBO_2 + NaOH = C_3H_5OHNaBO_3 + H_2O$$

1 ml of 1N NaOH = 0.03482 g of B_2O_3

$$\% \text{ Purity} = \frac{\text{Vol. of N/10 NaOH X 0.003482 X 216 X 500 X 100}}{\text{mass of the Sample X 69.6}}$$

$$= \frac{\text{Vol. of N/10 NaOH X F X 1.082 X 100}}{\text{mass of the Sample}}$$

Where F = Factor of N/10 NaOH
m = mass of sample in g

APPENDIX 'B'

VOLATILE MATTER

1. APPARATUS

Glass stoppered weighing bottole. 60mm in diameter Air oven 100 to 105 deg C.

2. PROCEDURE

An accurately weighed portion of approximately 5 g of the sample shall be transferred to a tared, glass stoppered weighing bottle about 60 mm in diameter. The weighing bottle and contents shall be placed, with the stopper removed, in an oven maintained at 100 deg C to 105 deg C for 2 hours. The stopper shall be replaced, the bottle cooled in a desiccator, and weighed. The volatile matter content shall be calculated as follows :

3. CALCULATIONS

$$\text{Volatile matter \% by mass} = \frac{m_2 - m_3}{m_2 - m_1} \times 100$$

where

m_1 = mass of empty bottle, g

m_2 = mass of bottle and sample, g

m_3 = mass of bottle and sample, after drying, g

APPENDIX 'C'

AVERAGE PARTICLE SIZE

The average particle size shall be determined using a Fisher Subsieve Sizer. One of the portion plugs shall be attached to the plug manipulator. A paper disc shall be laid over one end of the sample tube, and the plug pushed into the sample tube for a distance of approximately 12 mm with the perforated surface of the plug against the surface of the paper disc, forcing the paper to crimp around the edges and preced the plug into the sample tube. The sample tube shall be placed in a vertical position with the paper side of the plug up. A 2.45 gm portion of the dry sample obtained after drying shall be accurately weighed and transfered into the sample tube with the aid of a small funnel. The side of the tube shall be tapped to settle the powder. A second paper disc shall be laid over the top of the sample tube, the second portion plug attached to the plug manipulator, and the plug and paper disc forced down into the sample tube, manually compressing the sample as tightly as possible. The manipulator shall be removed. The rack shall be lowered until the pointer lies on the base line. The calculator chart shall be shifted until a porosity value of 0.80 is indicated by the pointer on the porosity scale located along the bottom edges of the calculator chart. The porosity value is a measure of the degree of packing of the sample. After making this setting, the chart shall not be moved until the determination has been completed. The rack shall be raised and the sample tube placed on the brass post beneath the rack and pinion with the lower plug in contact with the upper end of the brass post. The rack shall be lowered until the flat bottom end comes in contact with the upper plug.

The rack shall continue to be loweed by turning the pinion knob manually until the tip of the pointer coincides with a point on the sample height curve on the chart. The sample has now been packed to a porosity value of 0.80. The rack shall be raised and the sample tube removed. The initial level o the water menisous in the manometer tube, miniscus located over the calculator chart, shall be adjusted by means of the manometer control knob so that the meniscus coincides with the upper edge of the metalcrossbar attached on the rack when the tip of the pointer coincides exactly with the base line on the calculator chart. If adjustment of the water meniscus cannot be made because of too little water in the manometer tube. The range control knob, located at the extreme upper right of the front panel shall be turned to the "LO" position turned to the "LO" position. The sample tube shall be mounted, without disturbing the sample in any way, between the rubber cushioned supports of the sample tube holder. The upper cap shall be screwed down onto the sample until an air tight seal is obtained at both ends. The line cord shall be plugged into a 110 volt, 60-cycle alternating current line, and the electrical switch at the lower right hand corner of the front panel thrown to the "ON" position. This turns on the air pump as well as the pilot lamp which illuminates the tip of the bubbler tube in the pressure regulator stand pipe, asobserved through the round window in the lower left-hand corner of the front panel, and the level of water in the stand pipe, as observed through the upper window. The pressure control knob shall be adjusted until the bubbles rise in the stand pipe at the rate of 2 to 3 per second. The water level n the manometer tube shall be allowed to rise to a maximum level. Since the bulk volume of the required weight of sample is too great to permit the use of the calculator chart furnished with this apparatus, the average particle diameter shall be calculated using the following formula :

$$m = \frac{K\,H\,N}{(A\,H\text{-}N)^{3/2}} \sqrt{\frac{F}{P - F}}$$

where

m = average particle diameter, microns

K = permeability constant

A = cross-sectional area of sample tube, square centimeters(cm)

H = height of sample, cm

F = height of water column in manometer tube, cm, when the sample tube contains plugs, filter disks and sample.

P = height of water column in manometer tube, cm, when the sample tube contains plugs and filter disks only.

N = a factor which is equivalent to the weight of the sample divided by its true density.

The above determination shall be repeated at lower porosities, taking care to readjust the chart as required, until the bed is tightly compressed. All these determinations shall be made on the same sample. The porosities shall be plotted against the corresponding values of average particle diameter. Record the average particle size for which a change in porosity no longer affects the average particle diameter.

APPENDIX 'D'

A] DETERMINATION OF SILICA

Weigh 0.5 g of the sample in a Nickel crucible with lid. Add 8.0 g of Na_2CO_3. Mix thoroughly. Then keep over a burner at a low temperature. Gradually increase the temperature and then keep in a muffle furnace maintained at 800 ± 25 deg C for two hours. Remove the fused mixture, cool and dissolve in water and acidify the solution with 25 ml of Conc. HCl. Add 25 ml of Methyl Alcohol and evaporate slowly to dryness. Evaporate more with addition of 5 ml of Conc. HCl and 25 ml of Methyl Alcohol. To the residue add 10 ml of 1:1 HCl and 50 ml of hot water. Warm until all soluble salts are dissolved. Filter through No. 41 filter paper. Wash the residue with 2 % hot Conc. HCl and then 5 times with hot water. Preserve the filtrate for determination of Fe & Mg. Burn the filter paper in a platinum cucible, cool and weigh. Add few drops of water, two drops of Conc. H_2SO_4 and 5 to 10 drops of Hydroflouric Acid. Evaporate till all acid fumes are removed. Cool and weigh. The loss in mass represents silica content.

$$\% \text{ silica} = \frac{\text{Loss in mass X 100}}{\text{mass of the sample}}$$

B] DETERMINATION OF IRON AND MAGNESIUM

To the filtrate above add solid Ammonium Chloride and Ammonium Hydroxide to precipitate Iron compound. Filter through filter paper No. 41. Wash with dilute Ammonia. Burn the filter paper on the burner and then keep in a muffle furnace maintained at 800 ± 25 deg C for two hours. Cool and weigh as Iron Oxide.

$$\% \text{ Fe} = \frac{\text{Mass of residue} \times 0.3497 \times 100}{\text{Mass of sample}}$$

MAGNESIUM :-

Acidify the filtrate after removal of Iron with Acetic Acid and add 5 ml of Conc. HCl. Dilute the solution to 150 ml with distilled water. Add 5 to 10 drops of Methyl Red indicator and 10 ml of freshly prepared Dihydrogen Ammonium Orthophosphate (25 g of Dihydrogen Ammonium Orthophosphate dissolved in 100 ml of water). Add pure concentrated Ammonia in excess. Magnesium is precipitated as Magnesium Pyrophosphate. Keep overnight. Filter through No. 42 filter paper. Dry the filter paper alongwith residue at 105 ± 1 deg C for four hours. Place the filter paper in a previously dried & weighed gooch crucible. Burn it completely. Transfer it to muffle furnace maintained at 1000 ± 25 deg C for one hour. Cool & weigh.

$$\% \text{ Magnesium} = \frac{\text{Mass of residue} \times 48.61 \times 100}{222.5534 \times \text{Mass of the sample}}$$

APPENDIX 'E'

DETERMINATION OF SPECIFIC GRAVITY

Weigh the clean, dry pycknometer to the nearest 0.1mg (M_1). Fill the pycknometer with boiled, cooled water. Place the filled pycknometer without cap in the waterbath maintained at 27 ± 2 deg C & allow to remain until it has attained temperature equillibrium with the bath. If the liquid level in the capillary in the stopper has dropped, bring it level with the top of the stopper by adding more liquid. Wipe the outside of the pycknometer dry, cap and weigh (M_2).

Clean & dry the pycknometer. Add 1 to 2 g of the mateial & weigh accuately (M_3).

Place the sufficient liquid in the pycknometer to cover the material. Place the pycknometer without the stopper in the vacuum desiccator & apply vacuum gradually until all the air has been removed. Take care not to permit any particles to be splashed out of the pycknometer or on to the gound portion of the neck, where they might prevent the stopper from sealing properly.

Add additional liquid to fill the pycknometer. Insert the stopper, transfer to the bath, adjust the teemperature, adjust the final volume, wipe, cap & weigh (M_4).

$$\text{Specific gravity at } 27 \pm 2 \text{ deg C} = \frac{(M_3 - M_1) \times d'}{M_2 + (M_3 - M_1) - M_4}$$

****