[METRIC]

MIL-Z-12063B(EA)
19 January 1981
SUPERSEDING
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16 December 1952

MILITARY SPECIFICATION

ZINC BORATE, TECHNICAL (METRIC)

This specification is approved for use by US Army Armament Research and Development Command, Department of the Army, and is available for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers one technical grade of zinc borate.

2. APPLICABLE DOCUMENTS

2.1 Issues of documents. The following documents of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein.

SPECIFICATIONS

FEDERAL

RR-S-366 - Sieve, Test.

STANDARDS

FEDERAL

Fed. Std. No. 123 - Marking for Shipment (Civil Agencies).

FSC 6810

: Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed: to: Commander, US Army Armament Research and Development Command, ATTN: DRDAR-TSC-S, Aberdeen Proving Ground, MD 21010 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

MILITARY

MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes.
MIL-STD-129 - Marking for Shipment and Storage.

(Copies of specifications, standards, drawings, and publications required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

2.2 Other publications. The following documents form a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM) STANDARDS

D1193 - Reagent Water.

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

(Technical society and technical association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups and using Federal agencies.)

3. REQUIREMENTS

3.1 Chemical and physical characteristics. The zinc borate shall conform to the chemical and physical characteristics of table I when tested as specified therein.

TABLE I. Chemical and physical characteristics

Characteristic	Percent by weight		Test paragraph
	Minimum	Maximum	
Zinc (as ZnO)	---	46.0	4.2.4.1
Boron (as B_2O_3)	32.0	---	4.2.4.2
Loss in weight at 110°C	---	0.5	4.2.4.3
Loss in weight at 600°C	21.0	---	4.2.4.4
Sulfur (as SO_3)	---	0.5	4.2.4.5
Particle size:			4.2.4.6
Passing a No. 270 sieve	99.5	---	
Passing a No. 325 sieve	99.0	---	

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract, the contractor may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to specified requirements.

4.2 Quality conformance inspection.

4.2.1 Lotting. A lot shall consist of the zinc borate produced by one manufacturer, at one plant, from the same materials, and under essentially the same manufacturing conditions provided the operation is continuous. In the event the process is a batch operation, each batch shall constitute a lot (see 6.3).

4.2.2 Sampling.

4.2.2.1 For examination of packaging. Sampling shall be conducted in accordance with MIL-STD-105.

4.2.2.2 For test. Sampling shall be conducted in accordance with table II. A representative specimen of approximately 450 grams (g) shall be removed from each sample container and placed in a suitable clean, dry container labeled to identify the lot and container from which it was taken.

TABLE II. Sampling for test

Number of containers in batch or lot	Number of sample containers
2 to 25	2
26 to 150	3
151 to 1,200	5
1,201 to 7,000	8
7,001 to 20,000	10
Over 20,000	20

4.2.3 Inspection procedure.

4.2.3.1 For examination of packaging. The sample unit shall be one filled container ready for shipment. Sample containers shall be examined for the following defects using an AQL of 4.0 percent defective:

(a) Container damaged or leaking
(b) Marking incorrect, missing or illegible

4.2.3.2 For test. Each sample specimen taken in 4.2.2.2 shall be tested as specified in 4.2.4. Failure of any test by any specimen shall be cause for rejection of the lot represented.

4.2.4 Tests. Water in accordance with type III of ASTM D1193 and reagent grade chemicals shall be used throughout the tests. Where applicable, blank determinations shall be run and corrections applied where significant. Tests shall be conducted as follows:

4.2.4.1 Zinc. Weigh to the nearest 0.1 milligram (mg) approximately 0.5 g of the specimen into a 400-milliliter (ml) beaker, dissolve in 25 ml of 30-percent sulfuric acid, dilute to approximately 200 ml with water, and add 10 g of ammonium sulfate. Heat to approximately 60°C and add 2 drops diphenylamine or diphenylbenzidine indicator. Titrate with standardized potassium ferrocyanide solution [dissolve 21.6 g of potassium ferrocyanide, $K_4Fe(CN)_6 \cdot 3H_2O$, and 0.3 g of potassium ferricyanide, $K_3Fe(CN)_6$, in 1,000 ml of water, allow to stand 15 days, and standardize against 0.2000 g of pure zinc using the same procedure as above]. At the beginning of the titration add approximately 10 ml of the standardized potassium ferrocyanide solution and stir vigorously until the solution becomes blue; then titrate slowly until approximately 2 ml from the end point. The solution at this stage should be a light lavender. Continue the titration, stiring vigorously after each small addition of the potassium ferrocyanide solution, until 1 drop changes the color from light lavender to pea green. Calculate the percent zinc as zinc oxide as follows:

$$\text{Percent zinc} = \frac{124.5AB}{W}$$

where: A = Milliliters of potassium ferrocyanide solution used,
B = Grams of zinc per milliliter of potassium ferrocyanide solution, and
W = Weight of specimen in grams.

4.2.4.2 Boron. Weigh to the nearest milligram approximately 1 g of the specimen into a 600-ml beaker, moisten with water, and add 5 ml of 1 to 4 sulfuric acid. Warm to complete solution. Dilute to 50 ml with water and neutralize with 15-percent sodium hydroxide solution using methyl orange as the indicator. Acidify with 1 to 4 sulfuric acid using an excess of 4 to 5 drops. Dilute to 200 ml with cold water and pass a rapid stream (at least 8 bubbles per second) of hydrogen sulfide through the solution for 40 minutes. Filter, using a 12.5-centimeter No. 2 Whatman paper or equivalent and wash three times with cold water. Boil the filtrate gently for one-half hour. Cool, neutralize most of the acidity with 15-percent sodium hydroxide solution using methyl orange as the indicator, and complete the neutralization with 0.5N sodium hydroxide solution to the first definite yellow color. Add approximately 5 g of mannitol and titrate with 0.5N sodium hydroxide solution which has been standardized against 0.600 g of boric acid using the procedure herein and phenolphthalein as the indicator. At the appearance of the first pink end point add approximately 2 g more of the mannitol and titrate until the end

point again appears. Continue the addition of mannitol and titrate until the end point again appears. Continue the addition of mannitol and the titration until the end point persists on the addition of more mannitol. (If desired, 100 ml of glycerine, all added at the start of the titration, may be used in place of mannitol.) Calculate the percent boron as boric anhydride as follows:

$$\text{Percent boron} = \frac{100AB}{W}$$

where: A = Milliliters of standardized sodium hydroxide solution used,
B = Grams of boric anhydride per milliliter of standardized sodium hydroxide solution, and
W = Weight of specimen in grams.

4.2.4.3 Loss in weight at 110°C. Place approximately 4 g of the specimen in a tared, wide-form, weighing bottle provided with a glass stopper, and weigh to the nearest milligram. Heat, with stopper removed, for 2 hours in an oven at 105°C to 110°C. Insert stopper, cool, and weigh. Repeat the heating procedure until a constant weight in obtained. Calculate the percent loss in weight as follow:

$$\text{Percent loss in weight} = \frac{100\ (A - B)}{A}$$

where: A = Weight of specimen before drying in grams and
B = Weight of specimen after drying in grams.

4.2.4.4 Loss in weight at 600°C. Weigh to the nearest 0.01 g approximately 15 g of the specimen into a tared porcelain crucible. Ignite in a muffle furnace at 600° $\pm$ 10°C. Cool in a desiccator and weigh. Repeat the heating procedure until a constant weight is obtained. Calculate the percent loss in weight as follows:

$$\text{Percent loss in weight} = \frac{100\ (A - B)}{A}$$

where: A = Weight of specimen before drying in grams and
B = Weight of specimen after drying in grams.

4.2.4.5 Sulfur. Weigh to the nearest 0.01 g approximately 15 g of the specimen into a tall 400-ml beaker. Add sufficient ethanol to make a paste, 50 ml of saturated bromine water, 100 ml of water, and 50 ml of hydrochloric acid having a specific gravity of 1.20. Boil until all the bromine is expelled. Test by adding a few drops of methyl orange solution. If the color is destroyed, continue to boil until the color obtained from the addition of methyl orange is not destroyed. Cool and add 3 to 5 g of granular aluminum. Heat to boiling, filter through a No. 2 Whatman paper or equivalent and wash thoroughly with hot water. Dilute the filtrate to 300 ml with water and neutralize with ammonium hydroxide solution having a specific gravity of 0.90 using methyl orange as an indicator. Dissolve any white precipitate with hydrochloric acid having a specific gravity of 1.20 and add 6 drops in excess. Heat

to boiling and add, dropwise with constant stirring, 25 ml of 10-percent barium chloride solution. Allow to stand in a warm place for at least 2 hours. Filter through a tared Gooch crucible and wash the precipitate with hot water. Dry in an oven at 105° to 110°C and ignite at a dull red heat. Cool in a desiccator and weigh. Calculate the percent sulfur as sulfur trioxide as follows:

$$\text{Percent sulfur} = \frac{34.3\ A}{W}$$

where: A = Weight of barium sulfate in grams and
W = Weight of specimen in grams

4.2.4.6 Particle size. Weigh to the nearest 0.01 g approximately 25 g of the specimen, transfer to a mortar, and add 95-percent ethanol to make a slurry. Mix thoroughly with a pestle, using only enough pressure to break up lumps. Wash the suspension with additional 95-percent ethanol through a clean, dry and tared No. 270 sieve conforming to RR-S-366. A camel's hair brush may be used to facilitate washing the particles through the sieve. Dry the sieve for 1 hour at 105° to 110°C. Cool in a desiccator and weigh. Calculate the percent residue remaining on the sieve. Repeat the above procedure using a second 25-g specimen and a No. 325 sieve conforming to RR-S-366.

5. PACKAGING

5.1 Interplant shipment. Zinc borate shall be unit packed and packed to prevent damage in shipment from the supply source to the first receiving activity for immediate use or further processing. Shipping containers shall be in accordance with the rules and regulations applicable to the mode of transportation.

5.2 Unitization. Containers shall be unitized in a manner to assure carrier acceptance, safe arrival at destination, and stability of the stacks of the unitized containers in storage.

5.3 Marking. Containers shall be marked in accordance with Fed. Std. No. 123 and shall include the contract or purchase order number, lot or batch number, date of manufacture, and name and address of the prime contractor. Unitized loads shall be marked in accordance with MIL-STD-129.

6. NOTES

6.1 Intended use. The zinc borate is intended for use in smoke mixtures.

6.2 Ordering data. Procurement documents should specify the title, number, and date of this specification.

6.3 Batch. A batch is defined as that quantity of material which has been manufactured by some unit chemical process or subjected to some physical mixing operation intended to make the final product substantially uniform.

6.4 Significant places. For the purpose of determining conformance with this specification, an observed or calculated value should be rounded off "to the nearest unit" in the last right-hand place of figures used in expressing the limiting value, in accordance with the rounding-off method of ASTM E29.

Custodian:

Army - EA

Review activities:

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Project No. 6810-0545

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DOCUMENT IDENTIFIER *(Number)* AND TITLE

MIL-Z-12063B(EA) Zinc Borate, Technical (Metric)

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