

# JAN-B-660

5 AUGUST 1948

## NATIONAL MILITARY ESTABLISHMENT SPECIFICATION

### BARIUM OXALATE (For Use in Ammunition)

This specification was approved by the Departments of the Army, the Navy, and the Air Force for use of procurement services of the respective Departments.

#### A. APPLICABLE SPECIFICATIONS

A-1. *Specifications.*—The following specifications, of the issue in effect on date of invitation for bids, form a part of this specification:

##### U. S. ARMY SPECIFICATIONS

- 50-0-1—General Specification for Ammunition except Small Arms Ammunition.
- 100-2—Standard Specification for Marking Shipments by Contractors.

##### FEDERAL SPECIFICATIONS

- RR-S-366—Sieves; Standard, Testing.

#### B. GRADE

B-1. This specification covers one grade of barium oxalate as hereinafter specified.

#### C. MATERIAL AND WORKMANSHIP

C-1. Barium oxalate shall be manufactured by a precipitation process.

#### D. GENERAL REQUIREMENTS

D-1. See section E.

#### E. DETAIL REQUIREMENTS

- E-1. *Color.*—White.
- E-2. *Moisture.*—Maximum, 0.3 percent.
- E-3. *Material insoluble in dilute acid.*—Maximum, 0.5 percent.
- E-4. *Material soluble in water.*—Maximum, 0.3 percent.
- E-5. *Water-soluble alkalinity.*—Maximum, 0.1 percent.
- E-6. *Water-soluble acidity.*—The water extract shall not be acid to methyl orange.
- E-7. *Barium oxalate.*—Minimum, 92 percent.
- E-8. *Calcium salts.*—Maximum, 0.5 percent.
- E-9. *Grit.*—None.
- E-10. *Granulation.*—Through No. 100 U. S. Standard sieve, minimum 90 percent. Sieve to conform to Federal Specification RR-S-366.
- E-11. *Apparent density.*—Maximum, 0.50 gm. per ml.

#### F. METHODS OF SAMPLING, INSPECTION, AND TESTS

- F-1. *Size of lot.*—Maximum 1,000 pounds.
- F-2. *Sampling.*—Ten percent, but in no case more than 10 or less than 3 of the containers, shall be selected by the inspector so as to be

representative of the lot. If there are fewer than 3 containers in the lot, all the containers shall be sampled. The material shall be thoroughly mixed and approximately 8 ounces of it shall be taken from each selected container. Each of these primary samples shall be placed in an airtight container and labeled so that the container from which it was taken can be identified. A composite sample of approximately 8 ounces shall be made from equal portions of the primary samples. The composite sample shall be thoroughly mixed and placed in an airtight container labeled to show the name of the material, manufacturer, plant, contract or order number, lot number, and lot size. All acceptance tests shall be made on the composite sample. However, if it becomes apparent during sampling that the lot is not uniform, the inspector may require that any primary sample be tested for compliance with the requirements of the specification. All primary samples shall be held for possible future examination should the composite sample fail to meet the requirements.

F-3. *Inspection.*—Inspection shall be made in accordance with the requirements of U. S. Army Specification 50-0-1, and shall be at the point of delivery unless otherwise specified in the contract or order.

F-4. *Tests.*—

F-4a. *Color.*—Determine by visual inspection.

F-4b. *Moisture.*—Weigh approximately 5 gm. of the sample into a tared moisture dish. Place in a vacuum desiccator over concentrated  $H_2SO_4$  for 24 hours. Weigh, and calculate the loss in weight as percent moisture.

F-4c. *Material insoluble in dilute acid.*—Weigh approximately 5 gm. of the sample into a 400-ml. beaker. Add 200 ml. of 10-percent HCl solution and stir until all soluble material has dissolved. Filter the supernatant solution through a tared filtering crucible. Wash the insoluble material remaining in the beaker with three or four 10-ml. portions of 10-percent HCl solution and transfer to the crucible. Wash the material with two or three 10-ml. portions of distilled water. Dry for 1 hour at  $100^\circ$  to  $105^\circ$  C., cool in a desiccator and weigh. Calculate the increase in weight as percent material insoluble in dilute acid.

F-4d. *Material soluble in water.*—Weigh approximately 5 gm. of the sample into a dry 250-ml. Erlenmeyer flask. Add exactly 100 ml. of neutral distilled water at  $25^\circ$  C. Stopper the flask and shake for 5 minutes. Allow the insoluble material to settle for 10 minutes, then filter the solution through a dry filtering crucible, catching the filtrate in a dry receiving flask. Transfer the filtrate to a tared 150 ml. beaker and evaporate to a small volume on a hot plate and to dryness on a steam bath. If preferred, an aliquot of the filtrate may be used. Heat the residue for 1 hour at  $100^\circ$  to  $105^\circ$  C., cool in a desiccator, and weigh. Calculate the weight of the residue as percent material soluble in water.

F-4e. *Water-soluble alkalinity.*—Prepare a water extract of the material as described in paragraph F-4d. Add 3 drops of phenolphthalein indicator solution. If the water extract acquires a pink tint, this color indicating an alkaline reaction, titrate an aliquot portion of this water extract with approximately N/10 hydrochloric acid

solution to the disappearance of the pink color. Calculate the percent water-soluble alkalinity in the sample to sodium carbonate as follows:

$$\text{Percent water-soluble alkalinity} = \frac{10.6 VN}{W}$$

where

$V$  = ml. of standard acid used.  
 $N$  = normality of the standard acid.  
 $W$  = gm. of sample.

**F-4f. Water-soluble acidity.**—Prepare a water extract of the material as described in paragraph F-4d. Add 2 drops of methyl orange indicator solution. Note if the water extract is acid to methyl orange as indicated by the solution acquiring a pink tint.

**F-4g. Barium oxalate.**—Transfer a weighed portion of approximately 1 gm. of the sample to a 600-ml. beaker and add 50 ml. of 10-percent HCl solution. Warm and stir the mixture until all soluble matter has dissolved. Filter the solution if necessary and dilute to 200 ml. with distilled water. Add 14-percent NH<sub>4</sub>OH solution slowly until the precipitate which forms on the addition of the NH<sub>4</sub>OH dissolves with difficulty when the contents of the beaker are stirred. Heat the solution in the beaker to the boiling point and add slowly with stirring 5 ml. of a dilute H<sub>2</sub>SO<sub>4</sub> solution prepared by mixing 3 ml. of concentrated H<sub>2</sub>SO<sub>4</sub> with 20 ml. of water. Boil the solution gently for 15 minutes and then set the beaker and contents on a steam bath for 2 hours, or until the precipitate has settled completely. Filter the solution through a tared Gooch crucible, transfer the precipitate to the crucible, and wash thoroughly with hot distilled water. Save the combined filtrate and washings for the calcium determination. (See par. F-4h.) Dry the crucible and contents, ignite at 800° to 900° C. for 15 minutes, cool in a desiccator and weigh. Calculate the increase in weight to percent barium oxalate in the sample as follows:

$$\text{Percent barium oxalate} = \frac{96.56A}{W}$$

where

$A$  = gm. of residue.  
 $W$  = gm. of sample

**F-4h. Calcium salts.**—Use the combined filtrate and washings from the barium sulfate precipitate, prepared as described in paragraph F-4g. Make this solution just ammoniacal to methyl red, boil for several minutes, allow any precipitate to settle and filter the solution. Wash the precipitate several times with hot water. Evaporate the combined filtrate and washings to approximately 150 ml., make slightly acid to methyl red with 19-percent HCl solution and add 10 ml. of a saturated ammonium oxalate solution. Make the solution slightly ammoniacal to methyl red and keep just below the boiling point for approximately 1 hour. Filter the solution through a No. 42 Whatman, or equivalent, filter paper, transfer the precipitate to the filter paper and wash several times with a slightly ammoniacal 1-percent ammonium oxalate solution. Transfer the precipitate and filter paper to a small tared platinum crucible provided with a cover, dry and ignite carefully until all organic matter has been destroyed. Heat the residue at 900° to 1000° C. for 1 hour. Cover the crucible while the

crucible is still red hot, cool the covered crucible and contents in a sulfuric acid desiccator and weigh. Calculate the increase in weight to percent calcium salts, as calcium oxalate monohydrate, in the sample as follows:

$$\text{Percent monohydric calcium oxalate} = \frac{260.5A}{W}$$

where

$A$  = gm. of ignited residue.  
 $W$  = gm. of sample.

**F-4i. Grit.**—Transfer at least 3 portions of the sample to smooth glass slides. Rub the material on the glass by exerting pressure with a smooth steel spatula blade. Note if particles of grit are present as indicated by lack of uniformity of the material and the persistence of a scratching noise when pressing and rubbing of the material on the glass plate is continued.

**F-4j. Granulation.**—Transfer a weighed portion of approximately 100 gm. of the sample to a No. 100 U. S. Standard sieve provided with a bottom pan. Shake the material on the sieve, and brush if necessary with a brush having soft bristles, until no more material passes through the sieve. Weigh the material remaining on the sieve and calculate the percent passing through the sieve.

**F-4k. Apparent density.**—Assemble a Scott volumeter as shown on Figure 1, having a No. 40 U. S. Standard sieve in the upper hopper, so that the tared receiving cube, one cubic inch in volume, is directly under the lower funnel and rests on the base of the apparatus. Slowly pour approximately 15 to 20 gm. of the sample into the hopper and brush if necessary through the sieve in order to fill the receiving cube to overflowing. By means of a spatula, carefully strike off the excess barium oxalate from the cube, taking care not to jar the cube during this operation. Weigh the cube and contents, and from the volume and weight of barium oxalate calculate its apparent density. Make three tests and calculate the average of the apparent density values as follows:

$$D = \frac{W}{V}$$

where

$D$  = apparent density.  
 $W$  = weight of sample.  
 $V$  = volume of receiving cube.

**F-5. Resubmission and retests.**—If the composite sample, or any primary sample subjected to test, fails to pass the tests, the lot shall be rejected. The contractor shall have the option of having a partial or complete analysis made on a sample from any or all of the containers in the lot at no expense to the Government. The contractor may then remove the defective portions of the lot and resubmit the lot for acceptance, provided complete replacement of the defective portions can be made to the satisfaction of the inspector. The resubmitted lot shall be accepted, provided that new samples, selected in accordance with paragraph F-2, pass all the tests required by this specification.

**G. PACKAGING, PACKING, AND MARKING FOR SHIPMENT**

**G-1. Packing.**—Barium oxalate shall be packed in standard commercial containers of not more than 100 pounds capacity and so constructed as to insure acceptance by common or other carrier for safe transportation, at the lowest rate, to the point of delivery. The containers shall be provided with a liner or be of such construction as to prevent contamination by dust or other foreign material.

**G-2. Marking for shipment.**—In addition to any special marking required by the contract or order, and marking to insure safe handling, shipments for the Army shall be marked in accordance with the requirements of U. S. Army Specification 100-2.

**H. NOTES**

**H-1. Use.**—Barium oxalate covered by this specification is intended for use in pyrotechnic compositions.

**H-2.** This specification replaces Picatinny Arsenal Tentative Specification PXS-1182.

**H-3.** Copies of this specification may be obtained from the Office, Chief of Ordnance, Department of the Army, Washington 25, D. C.

**Notice.**—When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication, or otherwise, as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

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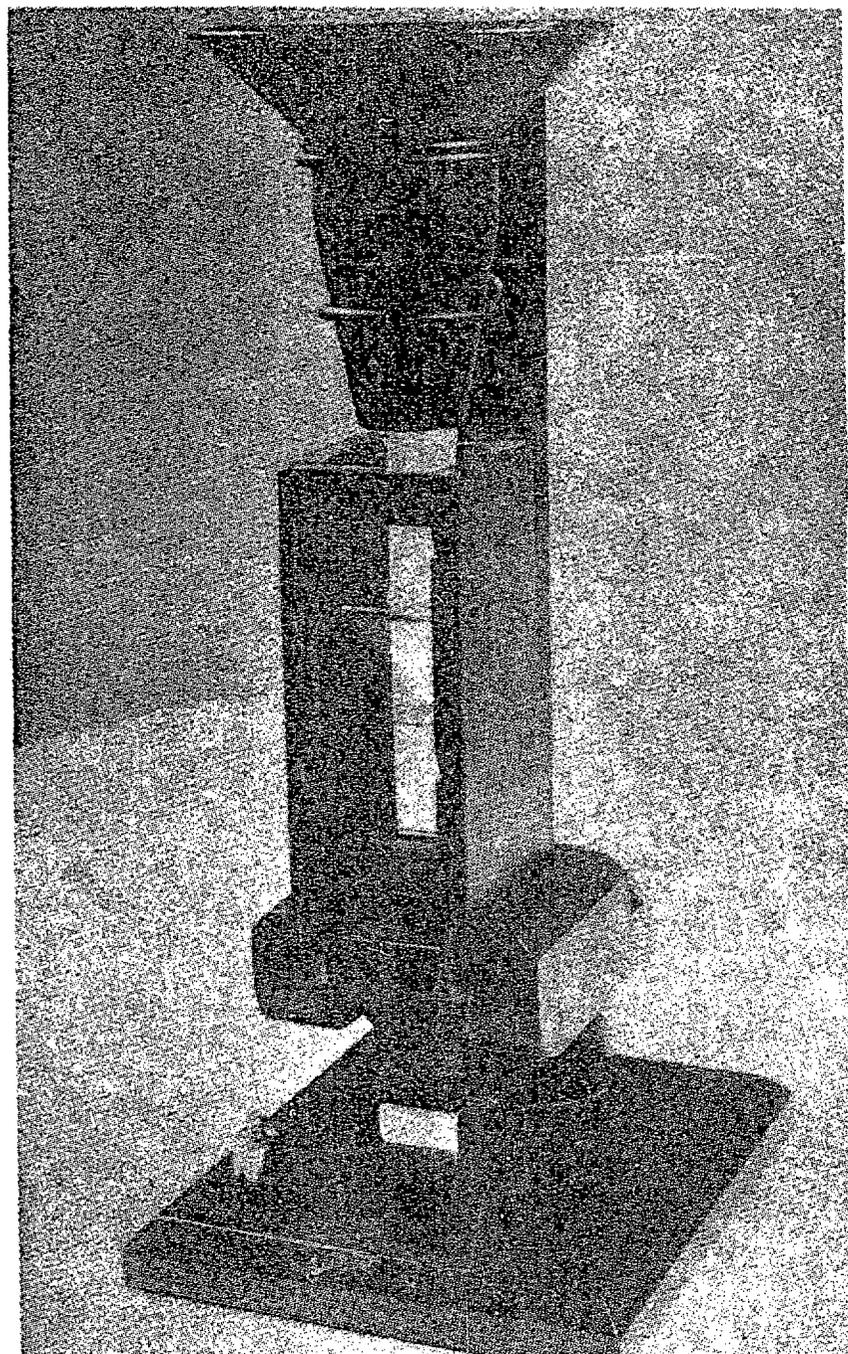


FIGURE 1.—Scott Volumeter.

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