JAN–C–628

30 JUNE 1948

NATIONAL MILITARY ESTABLISHMENT SPECIFICATION

CALCIUM 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 AND OTHER PUBLICATION

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.[1]

100–2—Standard Specification for Marking Shipments by Contractors.[1]

FEDERAL SPECIFICATION

RR–S–366—Sieves; Standard, Testing.

A–2. *Other publication.*—The following publication, of the issue in effect on date of invitation for bids, forms a part of this specification:

BUREAU OF SUPPLIES AND ACCOUNTS PUBLICATION

Navy Shipment Marking Handbook.[2]

B. GRADE

B–1. This specification covers one grade of calcium oxalate as hereinafter specified.

C. MATERIAL AND WORKMANSHIP

C–1. Calcium oxalate covered by this specification shall be prepared by a precipitation process. It shall consist essentially of the monohydrate.

D. GENERAL REQUIREMENTS

D–1. See section E.

E. DETAIL REQUIREMENTS

E–1. *Color.*—White.
E–2. *Moisture.*—Maximum, 0.5 percent.
E–3. *Material insoluble in dilute acid.*—Maximum, 0.5 percent.
E–4. *Material soluble in water.*—Maximum, 0.4 percent.
E–5. *Barium salts.*—Maximum, 0.5 percent.

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[1] Applicable only to Army purchases.
[2] Applicable only to Navy purchases.

E–6. *Calcium.*—Minimum, 28.6 percent.
E–7. *Oxalate.*—Minimum, 58.4 percent.
E–8. *Grit.*—None.
E–9. *Granulation.*—Through No. 100 U. S. Standard sieve.—Minimum, 99 percent. Sieve to conform to Federal Specification RR–S–366.
E–10. *Apparent density.*—Maximum, 0.60 gm. per ml.

F. METHODS OF SAMPLING, INSPECTION, AND TESTS

F–1. *Size of lot.*—Maximum, 1,000 pounds.
F–2. *Sampling.*—Three containers shall be selected, at random, from the lot for sampling purposes. If there are fewer than three containers in the lot, all the containers shall be selected. A ½-pound representative sample shall be taken from each selected container and placed in a tightly stoppered bottle. The samples shall be thoroughly mixed to form a composite sample representative of the lot. The bottle shall be labeled to identify the sample with the lot represented.
F–3. *Inspection.*—Inspection shall be made in accordance with the requirements of U. S. Army Specification 50–0–1 and shall be made 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.*—Transfer a weighed portion of approximately 5 gm. of the sample to a tared glass weighing dish. Dry the dish and contents at 100° to 105° C. for 4 hours, cool in a desiccator and weigh. Calculate the loss in weight as percent moisture in the sample.
F–4c. *Material insoluble in dilute acid.*—Transfer a weighed portion of approximately 5 gm. of the sample to a 400-ml. beaker. Add 200-ml. of 10-percent hydrochloric acid 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 hydrochloric acid. Transfer the insoluble material to the crucible and wash with two or three 10-ml. portions of distilled water. Heat the crucible and contents for 1 hour at 100° to 105° 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.*—Transfer a weighed portion of approximately 5 gm. of the sample to a dry 250–ml. Erlenmeyer flask. Add exactly 100 ml. of neutral distilled water at 25° C. Stopper the flask and shake for 5 minutes. Allow the insoluble material to settle for 10 minutes and then filter the solution through a dry filtering crucible, catching the filtrate in a dry receiving flask. Transfer the filtrate or an aliquot to a tared beaker, and evaporate to a small volume on a hot plate and then to dryness on a steam bath. Heat the beaker and residue at 100° to 105° C. for 1 hour, cool in a desiccator and weigh. Calculate the increase in weight as percent material soluble in water.
F–4e. *Barium salts.*—Transfer a weighed portion of approximately 1 gm. of the sample to a 400-ml. beaker and add 50 ml. of 10-percent hydrochloric acid solution. Heat the solution until the sample is dissolved, and filter, if necessary. Add slowly a 15-percent ammonium hydroxide solution until the precipitate which is formed on addition of

the ammonium hydroxide solution redissolves with difficulty when the contents of the beaker are stirred. Heat the solution to boiling, add, with stirring, 10 ml. of a 10-percent sulfuric acid solution, and boil for 10 minutes. Place the beaker on a steam bath for 2 hours or until any precipitate which may have formed, has settled out. Filter the solution through a tared Gooch crucible, wash the precipitate several times with hot 1-percent hydrochloric acid solution, transfer the precipitate to the crucible and wash well with hot water. Dry the crucible and contents, heat at 800° to 900° C. for 15 minutes, cool in a desiccator and weigh. Calculate the increase in weight to percent barium salts, as barium oxalate, as follows:

$$\text{Percent barium oxalate} = \frac{96.6A}{W}$$

where A = gm. of precipitate
W = gm. of sample.

F–4f. *Calcium.*—Transfer an accurately weighed portion of approximately 1 gm. of the sample to a small platinum crucible and ignite carefully, until all organic matter has been destroyed. Heat the crucible and contents at 900° to 1,000° C. for 15 minutes, cool and transfer as much as possible of the material in the crucible to a 250-ml. beaker. Add 50 ml. of water to the beaker. Rinse the crucible with several small portions of hot 5-percent hydrochloric acid solution and transfer the rinsings to the beaker. Add 10 ml. of concentrated hydrochloric acid to the beaker, heat the contents of the beaker to the boiling point and stir until all soluble material has dissolved. Cool the solution in the beaker slightly, and add 14 percent ammonium hydroxide solution until the solution is just alkaline to methyl red. Boil the liquid in the beaker for approximately 1 minute, allow the precipitate to settle and then filter the solution through a No. 41 Whatman, or equivalent, filter paper. Wash the precipitate and filter paper thoroughly with hot water. Dilute the combined filtrate and washings to approximately 300 ml. and make the solution acid by adding 5 ml. of concentrated hydrochloric acid. Heat the solution almost to boiling and add slowly, with stirring, 50 ml. of a saturated ammonium oxalate solution. Heat the solution to boiling and make just alkaline to methyl red by adding 14 percent ammonium hydroxide solution. Keep the solution in the beaker just below the boiling point for approximately 1 hour and then allow the precipitate to settle out. 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 hot, slightly ammoniacal 2-percent ammonium oxalate solution. Transfer the filter paper containing the precipitate to a tared platinum crucible provided with a cover. Dry the filter paper and precipitate in the crucible, ignite carefully until all organic matter has been destroyed and then ignite the residue at 900° to 1,000° 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. Heat the residue again at 900° to 1,000° C. for 15 minutes, cool the covered crucible and contents in a desiccator as before and weigh, repeating these operations if necessary until constant weight is obtained. Calculate the increase in weight to percent calcium as follows:

$$\text{Percent calcium} = \frac{(A - 0.0088BW)\,71.47}{W}$$

where A = gm. of ignited precipitate
B = percent barium salts (as determined in paragraph F–4e)
W = gm. of sample.

F–4g. *Oxalate.*—Transfer an accurately weighed portion of approximately 0.3 gm. of the sample to a 400-ml. beaker. Add 200 ml. of water and 5 ml. of concentrated sulfuric acid. Heat the solution almost to boiling and stir until the sample has dissolved. Titrate the hot solution with approximately N/10 potassium permanganate solution to a pink end point which does not fade in 30 seconds, keeping the solution at a temperature above 60° C. Make blank determination. Calculate the percent oxalate as follows:

$$\text{Percent oxalate} = \frac{4.401\,(V - v)\,N}{W}$$

where V = ml. of potassium permanganate solution required to titrate the oxalate in the sample.
v = ml. of potassium permanganate solution required for the blank.
N = normality of potassium permanganate solution.
W = gm. of sample.

F–4h. *Grit.*—Transfer at least three 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–4i. *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. If the material is slightly caked, break up the lumps by rubbing and pressing them between the fingers. Weigh the material remaining on the sieve and calculate the percent passing through the sieve.

F–4j. *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, 1 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 magnesium 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 calcium 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. *Rejection and resubmission.*—If the composite sample fails to conform to any of the requirements of this specification, the inspection lot shall be rejected. The contractor shall have the option of having a partial or complete analysis made on each container in the lot at no expense to the Government. The contractor may then remove defective portions of the inspection lot and resubmit the lot for acceptance.

G. PACKAGING, PACKING, AND MARKING FOR SHIPMENT

G–1. *Packing.*—Calcium 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 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; for the Navy in accordance with the requirements of the Navy Shipment Marking Handbook.

H. NOTES

H–1. Calcium oxalate covered by this specification is intended for use in pyrotechnic compositions.

H–2. This specification replaces Picatinny Arsenal Tentative Specification PXS–1181.

H–3. Copies of National Military Establishment, Joint Army-Navy, and Federal specifications (required for Army and Air Force purchases) and U. S. Army specifications may be obtained, as indicated in the "Index of United States Army, Joint Army-Navy and Federal Specifications and Standards." Copies of this Index may be obtained from the Superintendent of Documents, Government Printing Office, Washington 25, D. C. Agencies within the Departments of the Army and Air Force will obtain copies of Joint Army-Navy, U. S. Army and Federal specifications through established Departmental channels. Both the title and identifying symbol number should be stipulated when requesting copies.

H–4. Copies of National Military Establishment, Joint Army-Navy, and Federal specifications (required for Navy purchases), Navy Department specifications and the Navy Shipment Marking Handbook may be obtained upon application to the Bureau of Supplies and Accounts, Navy Department, Washington 25, D. C., except that Naval activities should make application to the Supply Officer in Command, Naval Supply Center, Norfolk 11, Va. Both the title and identifying symbol number should be stipulated when requesting copies.

H–5. Copies of this National Military Establishment specifications (required for Department of the Army purchases) may be obtained from the Office, Chief of Ordnance, Department of the Army, Washington 25, D. C.

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NAVY: OS.
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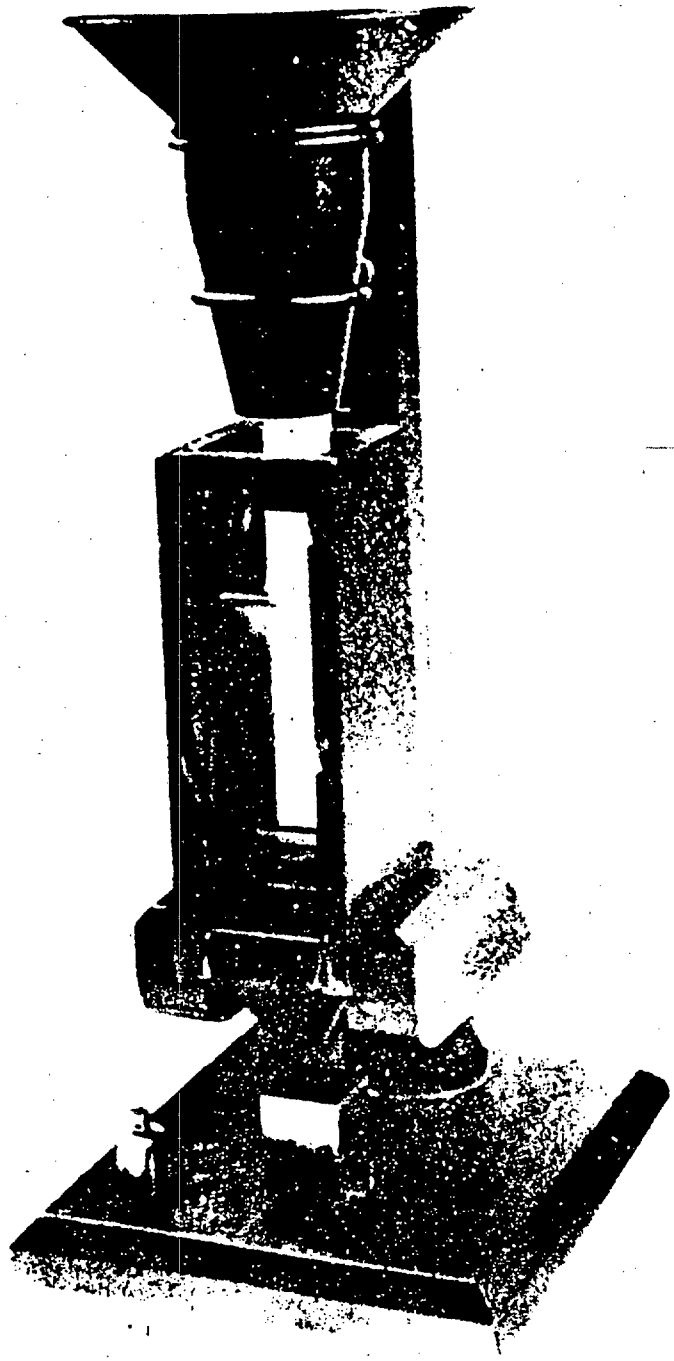

FIGURE 1.—*Scott volumeter.*

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