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MILITARY SPECIFICATION

CALCIUM CARBONATE

This specification is approved for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 This specification covers calcium carbonate used for small caliber propellants and pyrotechnics, and for impregnation of protective clothing.

2. APPLICABLE DOCUMENTS

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

SPECIFICATIONS

FEDERAL
RR-S-366 - Sieve, Test
UU-S-48 - Sacks, Shipping, Paper

STANDARDS

MILITARY
MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes
MIL-STD-129 - Marking for Shipment and Storage
MIL-STD-650 - Explosive: Sampling, Inspection and Testing

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

FSC 6810

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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

B 330 - Average Particle Size of Refractory Metals and Compounds by the Fisher Sub-Sieve Sizer
D 1193 - Reagent Water
E 300 - Sampling Industrial Chemicals

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 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 Material. The material shall be commercial whiting, either ground limestone or precipitated chalk, of light density and fine particle size.

3.2 Chemical requirements. Calcium carbonate shall conform to the chemical requirements shown in table I, when tested as specified in 4.3.2 through 4.3.8.

Table I. Chemical requirements

	Percent by weight	
	Min.	Max.
Calcium carbonate	98.0	---
Moisture	---	0.20
Iron oxide plus aluminum oxide	---	0.70

3.3 Apparent density. The apparent density of the calcium carbonate shall be 0.40 gm. per cc minimum, when tested as specified in 4.3.5.

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3.4 Granulation. The material shall be free from hard lumps, cakes, or agglomerates, and shall be in such a state of subdivision that no more than 0.3 percent by weight shall be retained on a 325 U. S. standard sieve conforming to the requirements of RR-S-366, when tested as specified in 4.3.6.

3.5 Average particle size. The average particle size shall not be greater than 3.00 microns, when tested as specified in 4.3.7.

3.6 X-ray diffraction. The material shall be identified as calcium carbonate by X-ray diffraction, when tested as specified in 4.3.8.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or order, the supplier 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 Lot size. A lot shall consist of not more than 50,000 pounds of calcium carbonate. If the material is produced by a continuous run process the lot shall contain material from only one continuous run. Material in the lot shall be identified by order of production (in the case of a continuous-run process), or by a batch number (in the case of a batch process) until final action is taken on the acceptance or rejection of the lot. A batch shall be considered as that quantity of material which was processed or blended in such manner as to be substantially uniform.

4.2.2 Sampling.

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

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4.2.2.2 For test. Samples from individual containers shall be selected in accordance with ASTM E 300, using a sampling thief. Samples shall be carefully marked to provide identification of the containers used to obtain the sample. For the tests given in 4.3.1 thru 4.3.7 the containers in the lot shall be divided into two (2) approximately equal groups. For each of these 2 groups, an individual sample shall be prepared in the following manner: Randomly select containers for sampling in accordance with MIL-STD-105. However, if each group contains less than five (5) containers, select all containers. Approximately equal portions of the material shall be taken from each selected container, in such quantity that the aggregate weight of the portions of the samples from each group shall be approximately one (1) pound. Combine these portions and thoroughly mix. Use the two samples formed to perform the tests described in 4.3.1 thru 4.3.7.

4.2.3 Inspection procedure.

4.2.3.1 Examination of preparation for delivery. An examination of the preparation for delivery shall be performed to determine compliance with Section 5. The lot shall consist of all items, packages or shipping containers, as applicable, to be shipped at one time. The sample unit shall consist of one item, package or shipping container. Defects shall be scored in accordance with the classification below. The inspection level shall be S-2 and the acceptance quality level (AQL) shall be 4.0, expressed as defects per hundred units.

EXAMINE	DEFECT
Packaging & Preservation	Not as specified. Not in accordance with contract requirements. Unit packages not uniform. Containers of improper construction.
Packing	Not as specified. Packing material not as specified. Closures not accomplished by specified methods or materials.
Marking	Not as specified. Marking wrong color. Marking incorrect size. Marking illegible, incorrect, incomplete, omitted or not in accordance with contract requirements.

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4.2.3.2 Examination of end item. An examination of the end item shall be performed to determine the condition and appearance of the bagged product. The sample unit shall be one filled bag before closure. The inspection level shall be S-2 and the AQL 4.0 defects per 100 units.

EXAMINE	DEFECTS
Appearance	Not material specified. Presence of foreign material. Caked or presence of lumps.
Loss of material	Evidence of loss or sifting of product from sealed bag.

4.2.3.3 Examination for contents of bag. The average net weight of contents of the bags sampled shall not be less than the amount specified in the contract or purchase order. The inspection level shall be S-2.

4.2.3.4 For test. Each of the two samples shall be subjected to all the tests specified in 4.3. If any sample is found to be not in conformance with the requirements of this specification, the lot represented shall be rejected. Where the material is produced by a continuous-run process, a rejected lot shall not be resubmitted for inspection. In case, however, material in the lot is identifiable by batch number, the manufacturer shall have the option of having partial or complete analysis of the batches of a rejected lot made at no expense to the Government. The manufacturer may then remove or replace defective portions of the lot and resubmit the lot for acceptance, provided the markings on the containers are such that complete removal of the defective portions of the lot can be made to the satisfaction of the inspector.

4.3. Test methods.

4.3.1 General. Distilled water, in accordance with ASTM D 1193 Type III and analytical grade reagents, shall be used throughout the tests. Blank determinations shall be run in parallel with the tests, using the same quantities of reagents used in the test, and corrections shall be applied when necessary.

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4.3.1.1 Inspection Equipment. The government reserves the right to inspect contractor's equipment and determine that he has available and utilizes correctly, measuring and test equipment of the required accuracy and precision and that the instruments are of the proper type and range to make measurements of the required accuracy. Commercial inspection equipment, shall be employed where applicable for all tests and examinations specified in Section 4. The contractor is responsible for assuring proper calibration procedures are followed. Government approval of all inspection equipment is required prior to its use for acceptance purposes.

4.3.2 Calcium carbonate. Transfer a 2.5 gm. sample, weighed to 0.1 mg., to a 600 ml. beaker. Cover with a watch glass and carefully add 100 ml. of hydrochloric acid (1 to 3). Heat to boiling and boil for 3 minutes to expel the carbon dioxide. Cool and dilute to 1 liter in a volumetric flask. Pipet a 100 ml. aliquot into the original beaker, neutralize with ammonium hydroxide (1 to 1) to methyl red, and add 1 ml. excess ammonium hydroxide (1 to 1). Dilute to about 200 ml. with water, heat to boiling and add 125 ml. of saturated ammonium oxalate solution (approximately a 4 percent solution) slowly from a graduate while stirring vigorously. Heat to boiling and boil for 1 or 2 minutes. Digest the solution at about 95°C for 30 minutes, remove from the hot plate and allow to stand for 1 hour (no longer). Filter through a sintered glass crucible of medium porosity and wash several times with water at room temperature. Transfer the crucible and contents to a 400 ml. beaker and add 100 ml. of sulfuric acid (1 to 10). Heat to 80°C and stir to dissolve the oxalate. Add about 100 ml. of water at a temperature of 80°C. Titrate the hot solution with 0.1N potassium permanganate solution to a faint pink. Calculate the percent calcium carbonate as follows:

$$\text{Percent calcium carbonate} = \frac{5.005 \times V \times N}{W}$$

where V = potassium permanganate solution for titration of sample, ml.
N = normality of potassium permanganate solution.
W = weight of sample in aliquot, gm.

4.3.3 Moisture. Transfer approximately 3 gm. of sample, weighed to 1 mg. to a weighing dish at least 6 cm. in diameter. Weigh the dish plus sample to 0.1 mg. Heat at 110°C for 2 hours, cool in a desiccator and weigh to 0.1 mg. Calculate the percent moisture as follows:

$$\text{Percent moisture} = \frac{A \times 100}{W}$$

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where A = loss in weight, gm.
W = weight of sample, gm.

4.3.4 Iron oxide plus aluminum oxide. Transfer approximately 3 gm. of the sample, weighed to 1 mg., to a 400 ml. beaker. Carefully add 30 ml. of hydrochloric acid (1 to 1). Add 1 ml. of nitric acid, evaporate to dryness and bake at 100° to 110°C for 1 hour. Take up the residue in 100 ml. of water and 10 ml. of hydrochloric acid. Filter through a Whatman No. 40 filter paper, wash with water and discard the residue. Neutralize the filtrate to methyl red with ammonium hydroxide (1 to 1) and add 3 drops excess ammonium hydroxide (1 to 1). Heat to boiling and boil for a minute. Add more ammonium hydroxide, one drop at a time, if necessary, to maintain slight alkalinity. Add a little paper pulp, filter through a Whatman No. 41 filter paper, and wash several times with hot 2 percent ammonium chloride solution. Transfer the filter paper and precipitate to a tared platinum crucible, then char and burn off the filter paper at low heat. Ignite at approximately 1000°C for 30 minutes, cool in a desiccator, and weigh. Calculate the percent of iron oxide plus aluminum oxide as follows:

$$\text{Percent iron oxide plus aluminum oxide} = \frac{A \times 100}{W}$$

where A = weight of residue, gm.
W = weight of sample, gm.

4.3.5 Apparent density. Brush approximately 75 gm. of the sample through a U. S. Standard No. 30 sieve and collect in a pan. Transfer approximately 50 gm. of the sieved portion, weighed to 0.1 gm., to a 250 cc graduated cylinder which has a height between the 10 and 250 cc marks of approximately 9-5/8 inches. Drop the cylinder 50 times from a height of 1 1/2 inches onto a piece of hard leather. The cylinder should be rotated slightly during the dropping period. Calculate the apparent density as follows:

$$\text{Apparent density} = \frac{W}{V}$$

where W = weight of sample in graduate, gm.
V = volume occupied by sample in graduate, cc

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4.3.6 Granulation.

4.3.6.1 Visually examine the material to ascertain it is free from hard lumps, cakes, or agglomerates.

4.3.6.2 Residue retained on No. 325 sieve. Weigh out a sample of approximately 50 gm. to 0.1 gm. and wash it through a No. 325 U. S. Standard sieve using a stream of water and a camel hair brush. Carefully wash the residue into a tared porcelain weighing dish. Decant the excess water from the weighing dish, after allowing the residue to settle. Dry the dish on the hot plate, cool in a desiccator, and weigh. Calculate the percent of material retained on a No. 325 sieve as follows:

$$\text{Percent material retained on a No. 325 sieve} = \frac{A \times 100}{W}$$

where A = weight of residue, gm.
W = weight of sample, gm.

4.3.7 Average particle size. Determine the average particle size by use of the Fisher sub-sieve sizer, using a sample size of 2.70 gm. The procedure is described in MIL-STD-650 (method 205.1) and in ASTM B 330.

4.3.8 X-ray diffraction. Calcium carbonate shall be identified using a powder X-ray diffraction test as described in a standard or reference text book.

5. PREPARATION FOR DELIVERY

5.1 Packaging.

5.1.1 Levels B and C. Calcium carbonate shall be packaged in accordance with the applicable requirements of UU-S-48.

5.2 Packing.

5.2.1 Levels B and C. Calcium carbonate shall be packed in accordance with the applicable requirements of UU-S-48.

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5.3 Marking. In addition to any special marking required by the contract or order, shipments shall be marked in accordance with MIL-STD-129.

5.4 Palletization. The item shall be palletized to meet carrier acceptance and safe delivery to destination at lowest rate in compliance with requirements of carrier rules and regulations applicable to the mode of transportation.

6. NOTES

6.1 Intended use. Calcium carbonate procured under this specification is intended to be used in small arms propellants and pyrotechnic mixtures for cartridges not exceeding 20 mm, and for impregnation of protective clothing.

6.2 Ordering data. Invitation for bids or request for proposal, contracts or purchase orders should specify the following:

a. Title, number and date of this specification.

b. Level of packaging and packing required (see 5.1 and 5.2).

c. Unit quantity required.

d. Palletization (see 5.4).

Custodian:
Army - MU

Review interest:
Army - MU, WC, EA
DSA - GS

User interest:
Army - MI
Navy - OS

Preparing activity:
Army - MU

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DOCUMENT IDENTIFIER AND TITLE

MIL-C-293B Calcium Carbonate

NAME OF ORGANIZATION AND ADDRESS

CONTRACT NUMBER

MATERIAL PROCURED UNDER A

☐ DIRECT GOVERNMENT CONTRACT ☐ SUBCONTRACT

1. HAS ANY PART OF THE DOCUMENT CREATED PROBLEMS OR REQUIRED INTERPRETATION IN PROCUREMENT USE?

A. GIVE PARAGRAPH NUMBER AND WORDING.

B. RECOMMENDATIONS FOR CORRECTING THE DEFICIENCIES

2. COMMENTS ON ANY DOCUMENT REQUIREMENT CONSIDERED TOO RIGID

3. IS THE DOCUMENT RESTRICTIVE?

☐ YES ☐ NO *(If "Yes", in what way?)*

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