

MIL-S-14760 (AR)
10 May 1968

MILITARY SPECIFICATION

SILICA
(FOR USE IN AMMUNITION)

1. SCOPE

1.1 This specification covers one type of finely divided silica for use as a thickening agent and in thixotropic control in Aerial Mines. (See 6.4)

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 this specification to the extent specified herein.

SPECIFICATIONS

FEDERAL

PPP-D-723 - Drums, Fiber.

STANDARDS

MILITARY

MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes (ABC-STD-105).
MIL-STD-109 - Quality Assurance Terms and Definitions.
MIL-STD-129 - Marking for Shipment and Storage.
MIL-STD-1235 - Single and Multilevel Continuous Sampling Procedures and Tables for Inspection by Attributes.

FSC: 1345

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(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).

3. REQUIREMENTS

3.1 The material shall consist of finely divided silica which conforms to the following requirements: (See 6.4)

Property	Min.	Max.	Applicable Paragraph
Surface area m ² /gm	175	225	4.3.1
Density, lbs/cu ft.	-	2.3	4.3.2
Moisture	-	1.5	4.3.3
pH	3.5	4.2	4.3.4
Suspension	No settling in 10 minutes		4.3.5

3.2 Workmanship.-The composition shall be free of dirt, chips, and other foreign matter.

4. QUALITY ASSURANCE PROVISIONS

4.1 General quality assurance provisions.-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, the supplier may utilize his own facilities or any commercial laboratory acceptable to 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 prescribed requirements. Reference shall be made to Standard MIL-STD-109 in order to define the terms used herein.

4.1.1 Contractor quality assurance system.-If the contractor desires to utilize a quality assurance system, which is at variance with the quality assurance provisions of 4.2 and 4.3 and other documents referenced herein, he shall submit a written description of the system to the contracting officer for approval prior to initiation of production. It shall include a description covering controls for lot formation and identification, inspections to be performed, inspection stations, sampling procedures, methods of inspection, (Measuring and Testing equipment), and provisions for control and disposition of non-conforming material. The written description will be considered acceptable when, as a minimum, it provides the quality assurance provisions required by the provisions

of 4.2 and 4.3 and other documents referenced herein. The contractor shall not be restricted to the inspection station or the method of inspection listed in this specification provided that an equivalent control is included in the approved quality assurance procedure. In cases of dispute as to whether certain procedures of the contractor's system provide equal assurance, the comparable procedures of this specification shall apply. The contractor shall notify the Government of, and obtain approval for, any changes to the written procedure which affect the degree of assurance required by this specification or other documents referenced herein.

4.1.2 Submission of product.-At the time the completed lot of product is submitted to the Government for acceptance the contractor shall supply the following information accompanied by a certificate which attests that the information provided is correct and applicable to the product being submitted:

a. A statement that the lot complies with all requirements and provisions of the approved current written description of the silica material.

b. Specification number and date, together with an identification and date of changes.

c. Certificates of analysis on all material used directly by the contractor when such material is controlled by Government specifications, shall be made available upon request by the contracting officer.

d. Quantity of product in the lot.

e. Date submitted.

The certificate shall be signed by a responsible agent of the certifying organization. The initial certificate submitted shall be substantiated by evidence of the agent's authority to bind his principal. Substantiation of the agent's authority will not be required with subsequent certificates unless, during the course of the contract, this authority is vested in another agent of the certifying organization.

4.1.3 First article inspection

4.1.3.1 Submission.-The contractor shall submit a first article quantity as designated by the Contracting Officer for evaluation in accordance with the provisions of 4.1.3.2 (See 6.1). The first article sample shall consist of 500 grams of material.

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All samples submitted shall have been produced by the contractor (or furnished by a supplier) using the same production processes, procedures, and equipment as will be used in fulfilling the contract. All materials, including packaging and packing, shall be obtained from the same sources of supply as will be used in regular production. The sample shall be accompanied by certificates of conformance. A first article quantity, or portion thereof, as directed by the Contracting Officer, shall also be submitted whenever there is a lapse in production for a period in excess of 90 days, or whenever a change occurs in manufacturing process, material used, drawing, specification or source of supply as to significantly affect product uniformity as determined by the Government. Prior to submission, the contractor shall inspect the sample to the degree necessary to assure that it conforms to the requirements of the contract and submit a record of this inspection with the sample. A sample containing known defects will not be submitted unless specifically authorized by the Contracting Officer.

4.1.3.2 Inspections to be performed.-The sample will be subjected by the Government to any or all of the examinations or tests specified in 4.2 and 4.3 of this specification and any or all requirements of the applicable drawings.

4.1.3.3 Rejection.-If any sample fails to comply with any of the applicable requirements, the first article quantity shall be rejected. The Government reserves the right to terminate its inspection upon any failure of a sample to comply with any of the stated requirements. In the event of rejection, the Government reserves the right to require the contractor to take corrective action and submit a new first article quantity or portion thereof. Until a first article quantity is accepted, the contractor is in no way authorized by the Government to resume regular production unless otherwise directed by the Contracting Officer.

4.2 Inspection provisions

4.2.1 Lot formation.-A lot shall consist of one or more batches of silica produced by one manufacturer in accordance with the same specification, or same specification revision under one continuous set of operating conditions. Each batch shall consist of that quantity of the silica that has been subjected to the same unit chemical or physical mixing process intended to make the final product homogeneous. The product shall be submitted for inspection in accordance with Standard MIL-STD-105 (or Standard MIL-STD-1235 when applicable).

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4.2.2 Examination.-Sampling plans and procedures for the following classification of defects shall be in accordance with Standard MIL-STD-105. Continuous sampling plans, in accordance with Standard MIL-STD-1235, may be used if approved by the procuring activity. Also, at the option of the procuring activity, AQL's and sampling plans may be applied to the individual characteristics listed using an AQL of 0.40 percent for each major defect and an AQL of 0.65 percent for each minor defect.

4.2.2.1 Drums, prior to filling Polyethylene liner.

Categories	Defects	Method of Inspection	Code No. (see 6.2)
Critical: None defined			
Major: AQL 0.40 percent			
101.	Liner cut, torn or punctured.....	Visual	01001
Minor: None defined.			

4.2.2.2 Drums, prior to closing (Polyethylene liner).

Categories	Defects	Method of Inspection	Code No.
Critical: None defined			
Major: AQL 0.40 percent			
101.	Liner not completely sealed.....	Visual	02001
Minor: None defined.			

4.2.2.3 Drum, sealed (fiber).

Categories	Defects	Method of Inspection	Code No.
Critical: None defined			
Major: AQL 0.40 percent			
101.	Weight of contents.....	Scale	03001
102.	Closure incomplete or damaged to the extent that contents sift out..	Visual	03002
Minor: AQL 0.65 percent			
201.	Markings misleading or unidentifiable.....	Visual	03003

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4.2.3 Sampling

4.2.3.1 A representative composite sample of 500 grams shall be taken from each batch of material. (Note: If the manufacturer blends his batches to form a lot, the sample shall be selected from the resultant lot.) This will be accomplished by selecting equal quantities from twenty (20) shipping containers and thoroughly mixing the quantities thus taken into a composite sample. This sample shall be tested for conformance to the requirements of 3.1 as specified in 4.3. If the sample fails to comply with any requirement the lot or batch shall be rejected.

4.2.3.2 In addition to the sample of 4.2.3.1 five (5) individual samples of 50 grams each shall be taken from five (5) randomly selected shipping containers. Each of these samples shall be tested separately as specified in 4.3.1. If any sample fails to comply with the surface area requirement of 3.1, the lot or batch shall be rejected.

4.3 Test methods and procedures

4.3.1 Surface area - Major Defect - (Defect Code 04001).

4.3.1.1 Apparatus (Equivalent apparatus may be used):

a. Beckman Model G pH Meter using Beckman External lead and Beckman External Type 42 Glass Electrode (Beckman No. 40485) or

b. Beckman Model 76 pH Meter using Beckman No. 39096 Thermo-Compensator in conjunction with No. 41236 Silver-Silver Chloride Fiber Type Reference Electrode and No. 40471 Silver-Silver Chloride General Purpose Glass Electrode.

c. Cenco Magnetic Stirrer, (No. 18851) with No. 2 1-5/8" Teflon Coated Stirring Bars (No. 18854).

d. 50 ml. burette, capable of delivering quantities accurate to within 0.05 ml. (i.e., Kemax No. 17060).

4.3.1.2 Reagents

0.1N NaOH Solution

0.1N HCl Solution

20% NaCl Solution (W/W)

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4.3.1.3 Procedure.-Transfer 2.5 grams of the silica sample into a 400 ml beaker and add 250 ml of 20% sodium chloride solution. Stir the suspension using a magnetic stirrer at the maximum rate and adjust the pH to 4.0 by adding hydrochloric acid or sodium hydroxide. Titrate the resultant solution to a pH of 9.0 by the addition of 0.1 Normal sodium hydroxide at a rate of approximately one drop per second. Allow to stir for five minutes more while holding the pH at 9.0 by addition of sodium hydroxide in one drop increments. A blank run using 250 ml of 20% sodium chloride solution, adjusting the pH to 4.0 and titrating to 9.0 as above, shall be made.

4.3.1.4 Calculations

Surface Area (Sq. Meters/gr.) = (ml-ml blank) N (NaOH) (138.6)
correction

CORRECTION

ML. 0.1N HCl Used	CORRECTION
0	0
0.2	7
0.4	13
0.6	20
0.8	27
1.0	33

4.3.2 Density - Major Defect - (Defect Code 05001)

4.3.2.1 Apparatus

- Cylindrical container of approximately 800cc volume.
- Balance accurate to 0.1 gram.

4.3.2.2 Procedure.-The silica is slowly poured into the container and allowed to settle (care must be taken not to jar container). As the Silica settles, more is added, keeping the silica heaped in the container. When settling has stopped (normally 3 to 5 minutes are required), the excess is scraped off with a straight edge. The container is then weighed and the net weight of material determined.

4.3.2.3 Calculations.-Material

$\frac{\text{Wt. of material}}{\text{vol. Container in cc}} \times 62.43 = \text{Density silica\#/Pt.}^3$

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4.3.3 Moisture (Code No. 06001).-Place a quantitatively weighed portion of approximately 10 grams of the sample in a tared weighing dish. Heat the dish and contents for 2 hours in an oven maintained at 100 ± 5 degrees C. Cover the dish, remove from oven, and cool the specimen to room temperature in a desiccator. Determine the loss in weight by weighing and calculate the percentage of moisture as follows:

$$\text{Percent Moisture} = \frac{A}{B} \times 100$$

where:

A = loss in weight, gm.

B = weight of sample, gm.

4.3.4 pH - Major Defect - (Defect Code 07001)

4.3.4.1 Apparatus (Equivalent apparatus may be used)

a. Beckman Model G pH Meter using Beckman External Fiber Type Calomel Electrode (Beckman No. 39170) with 30" lead and Beckman External Type 42 Glass Electrode (Beckman No. 40485) or

b. Beckman Model 76 pH Meter using Beckman No. 39096 Thermo-Compensator in conjunction with No. 41236 Silver-Silver Chloride Fiber Type Reference Electrode and No. 40471 Silver-Silver Chloride General Purpose Glass Electrode.

c. Cenco Magnetic Stirrer, (No. 18851) with No. 2 1-5/8" Teflon Coated Stirring Bars (No. 18854).

4.3.4.2 Procedure.-Weigh 4 grams of sample into a 250 ml. beaker and add 100 ml. of purified water with a pH of 5.5 minimum. (The purified water is prepared by passing water through an ion exchanger to remove salts and carbon dioxide.) Boiling may also be necessary to remove carbon dioxide to give a 5.5 minimum pH. (Do not adjust the pH of the water with caustic). Stir the silicate water mixture vigorously for 5 minutes. Stop the stirrer. When the liquids agitation ceases, record the pH reading.

4.3.5 Suspension - Major Defect - (Defect Code 08001).-Transfer three (3) grams of silica to a tube containing 100 ml of trichlorotrifluoroethane and shake for two (2) minutes to disperse the silica. Allow the tube and contents to stand for ten (10) minutes; then visually inspect for settling of the silica.

5. PREPARATION FOR DELIVERY

(NOTE: The packing described in 5.1.1 is advisory rather than mandatory. If the supplier wishes to deviate from the provisions in 5.1.1, he shall obtain the approval of the procuring activity.)

5.1 Packing

5.1.1 Drums.-Unless otherwise specified in the contract or purchase order, the material shall be packed in non-returnable fiber drums in accordance with Specification PPP-D-723, (Type III, Grade A). The drums shall be finished with a nominal 0.004 inch thick polyethylene liner, properly heat sealed or otherwise closed to afford protection to the contents equivalent to that given by a heat seal closure.

5.1.2 Level C.-The material shall be packed to afford protection against damage during direct shipment from supply source to the first receiving activity for immediate use. Containers shall comply with Uniform Freight Classification Rules and Container Specifications for rail shipment or National Motor Freight Rules and Container Specifications for truck shipment as applicable.

5.2 Marking.-In addition to any special markings required by the contract or purchase order, containers shall be marked in accordance with MIL-STD-129. Marking shall include, but is not limited to, the following information:

- a. Manufacturers name
- b. Product designation
- c. Lot number
- d. Date of manufacture
- e. Number of this specification

6. NOTES

6.1 Ordering data.-Procurement documents should specify the title, number, class and date of this document.

6.2 Inspection code numbers.-The five digit code numbers assigned to the inspection herein are to facilitate future data collection and analysis by the Government.

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6.3 Intended use.-The silica covered in this specification is intended to be used as a thickening agent and in thixotropic control in Aerial Mine applications.

6.4 One type of silica which has been found satisfactory for the intended purpose is Cab-O-Sil, Grade M-5, made by the Cabot Corp., Boston, Massachusetts. Use of similar materials must be approved by Picatinny Arsenal, Dover, New Jersey 07801 ATTN: SMUPA-ND8 prior to use.

Custodian:
Army-AR

Preparing activity:
Army-AR

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