MIL-S-230C
12 January 1983
SUPERSEDING
MIL-S-230B
7 August 1969

MILITARY SPECIFICATION

SILICON, POWDERED, TECHNICAL

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

1. SCOPE

1.1 Scope. This specification covers two grades and four classes of technical grade powdered silicon.

1.2 Classification. Silicon shall be of the following grades and classes as specified (see 6.2):

Grade I
- Class A
- Class B
- Class D

Grade II
- Class C

2. APPLICABLE DOCUMENTS

2.1 Government documents.

2.1.1 Specifications, standards, and handbooks. Unless otherwise specified, the following specifications, standards, and handbooks of the issue listed in that issue of the Department of Defense Index of Specifications and Standards (DoDISS) specified in the solicitation form a part of this specification to the extent specified herein.

: 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. :

FSC 6810

SPECIFICATIONS

FEDERAL

RR-S-366 - Sieve Test

STANDARDS

MILITARY

MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes

2.1.2 Other Government documents, drawings, and publications. The following other Government documents, drawings, and publications form a part of this specification to the extent specified herein.

CODE OF FEDERAL REGULATIONS (CFR)

49 CFR 171 to 179 - Department of Transportation Hazardous Materials Regulations

(The Code of Federal Regulations is available from the Superintendent of Documents, US Government Printing Office, Washington, DC 20402. Orders for the above publication should cite "49 CFR 171 to 179.")

(Copies of specifications, standards, handbooks, drawings, and publications required by manufacturers in connection with specific acquisition functions should be obtained from the contracting 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. The issues of the documents which are indicated as DoD adopted shall be the issue listed in the current DoDISS and the supplement thereto, if applicable.

ASTM STANDARDS

D1193 - Reagent Water

(Application for copies should be addressed to ASTM, 1916 Race Street, Philadelphia, PA 19103.)

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

2.3 Order of precedence. In the event of a conflict between the text of this specification and the references cited herein, the text of this specification shall take precedence.

3. REQUIREMENTS

3.1 Chemical characteristics. Silicon shall conform to the applicable chemical characteristics of table I when tested as specified therein.

TABLE I. Chemical characteristics

Characteristic	Grade I	Grade II	Test paragraph
Silicon, minimum percent by weight	97.0	96.5	4.2.4.1
Iron, maximum percent by weight	1.00	---	4.2.4.2(a)
Aluminum, maximum percent by weight	1.00	---	4.2.4.2(b)

3.2 Granulation characteristics. Silicon shall conform to the applicable granulation characteristics of table II when tested as specified in 4.2.4.3.

TABLE II. Granulation characteristics

Sieve size	Percent by weight passing			
	Class A	Class B	Class C	Class D
No. 100, minimum	99.0	---	---	99.0
No. 170, minimum	---	---	98.0	---
No. 200, minimum	80.0	99.9	---	---
No. 230, minimum	---	---	90.0	---
No. 230, maximum	---	---	---	50.0

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or purchase order, 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 prescribed requirements.

4.2 Quality conformance inspection.

4.2.1 Lotting. A lot shall consist of the silicon of one grade and class 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 silicon test. Sampling shall be conducted in accordance with table III. A representative specimen of approximately 500 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 III. Sampling for silicon test

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

4.2.2.3 For container leakage test. Sampling shall be conducted in accordance with MIL-STD-105.

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 2.5 percent defective:

(a) Contents per container not as specified
(b) Container not as specified
(c) Container damaged or leaking
(d) Marking incorrect, missing, or illegible

4.2.3.2 For silicon 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.

4.2.3.3 For container leakage test. The sample unit shall be one container. The sample containers selected in 4.2.2.3 shall be tested as specified in 4.2.5 using an AQL of 1.5 percent defective.

4.2.4 Silicon tests. Water in accordance with ASTM D1193, type as applicable, 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 Silicon. Prepare a fusion mixture by grinding 10 g of anhydrous sodium carbonate and 1 g of potassium nitrate to a fine powder. Place 3 to 4 g of this mixture in a platinum crucible having a 35 to 50 milliliter (mL) capacity. Weigh into the crucible to the nearest 0.1 milligram (mg) approximately 0.5 g of the specimen. Cover the specimen with all but approximately 1 g of the fusion mixture. Thoroughly mix the fusion mixture and specimen by stirring with a vertically held platinum rod moving in a circular path. Care must be taken to keep a layer of fusion mixture that is free from silicon on the bottom of the crucible to prevent alloying of silicon with the platinum at high temperatures. When the mixture appears uniformly gray, brush any material adhering to the rod into the crucible. Add the remaining portion of the fusion mixture, distributing it evenly to form a cover which will help prevent spattering. Place the uncovered crucible on a silica triangle and apply a small flame of a bunsen burner to the outside of the crucible above the surface of the mixture. Carefully heat the upper half of the crucible for about 20 minutes. As a crust forms on the surface and it pulls away from the side, heat the lower half of the crucible on the sides, taking care not to get it too hot or the reaction will become violent and spatter. Cover the crucible to prevent loss of contents if the reaction becomes violent. After heating the main portion of the mixture with the bunsen burner for 30 minutes, use a blast burner to continue the heating. When the mixture has been well sintered and there is no evidence of continued reaction, increase the heat on the bottom of the crucible slowly to prevent loss of contents due to liberation of carbon dioxide from the pasty mass. Fusion is completed when all of the mixture has been reduced to a clear liquid containing no undissolved particles. Continue heating the liquid for an additional 5 minutes to insure complete reaction. Fuse any material adhering to the lid by heating. The total fusion time is 1.5 to 2 hours. Grasp the crucible with platinum-tipped tongs and rotate it to spread the liquid mass on the sides. This will hasten solution of the mass when it is reacted with hydrochloric acid. Allow the crucible to cool. Place the crucible and lid into a 750-mL porcelain casserole and cautiously add 200 mL of 22-percent hydrochloric acid. Cover with a watchglass supported by a glass triangle and place on a steam bath. When effervescence ceases, remove the crucible and lid and wash any material adhering to them into the casserole with the minimum amount of water necessary. Carefully evaporate the solution to dryness. Add 10 mL of concentrated hydrochloric acid to the residue, mix thoroughly, and then add 100 mL of hot water. Heat and stir until all soluble salts are dissolved. Filter the solution through Whatman No. 40 filter paper and wash the residue at least three times with hot water (this residue is designated as residue I). Return the filtrate and washings to the casserole, evaporate to dryness on a steam bath, and heat in an oven for 1 hour at 150° ± 2°C. Cool, add 10 mL of concentrated hydrochloric acid and 100 mL of hot water and stir thoroughly. Heat the solution, filter, and wash the residue with hot water (this residue is designated as residue II). Save the combined filtrate and washings for the determination of iron and aluminum in 4.2.4.2. During the second evaporation, transfer residue I and filter paper to the same crucible used for the fusion. Place the crucible in a cold muffle furnace with the lid removed and slowly heat to 1200°C in approximately 1-1/2 hours. Keep the crucible at 1200°C for another 30 minutes. Cover the crucible, cool to room temperature in a desiccator and weigh to the nearest 0.1 mg. Using a Meker burner, and keeping the

lid on the crucible, repeat the heating, cooling, and weighing until a constant temperature is obtained. Moisten the residue with a few drops of 1 to 1 sulfuric acid and 2 to 3 mL of water. Add hydrofluoric acid carefully until the cruible is approximately half full. (All operations using hydrofluoric acid should be performed in a well-ventilated hood.) Evaporate the solution to dryness on a hotplate. Ignite the crucible in a Meker flame, cool, and weigh to constant weight. The loss in weight of the crucible represents silicon dioxide. Treat residue II in the same manner as residue I. The combined losses in weight from both volatilizations represents the total amount of silicon dioxide. Calculate the percent by weight silicon as follows:

$$\text{Percent by weight silicon} = \frac{46.74\ (A + B)}{W}$$

where: A = Loss in weight in residue I volatilization in grams,
B = Loss in weight in residue II volatilization in grams, and
W = Weight of specimen in grams.

4.2.4.2 Iron and aluminum. Fuse residue I and residue II (see 4.2.4.1) with a small amount of potassium persulfate. Dissolve the fused mass with concentrated hydrochloric acid and add the resulting solution to the combined filtrate and washings reserved from the same silicon determination in 4.2.4.1. Heat the solution to 80° to 90°C and saturate thoroughly with hydrogen sulfide. Let the solution stand for approximately 30 minutes, filter, and wash with water which has been saturated with hydrogen sulfide and acidified with 1 to 20 sulfuric acid. Boil the solution until all of the hydrogen sulfide has been expelled and then oxidize the iron by the addition of bromine water until the solution becomes distinctly yellow. Boil the solution until all of the bromine has been expelled and adjust the volume of the solution to approximately 150 mL with water. Add three or four drops of methyl red indicator solution. Add a 1 to 1 solution of ammonium hydroxide until the solution is alkaline to methyl red, then add 2 or 3 mL in excess. Allow to digest for 10 to 15 minutes, filter, and wash several times with hot 1-percent ammonium nitrate solution, discarding the filtrate and washings. Dissolve the precipitate on the filter paper with hot 1 to 1 hydrochloric acid and wash the paper thoroughly with hot water. Using the above procedure, reprecipitate the iron and aluminum and filter through a Whatman No. 41 filter paper. Wash with hot 1-percent ammonium nitrate solution. Place the filter paper and precipitate in a platinum crucible which has been ignited to constant weight at 800° ± 50°C. Ignite gently until the paper is charred, and then ignite to constant weight in a muffle furnace at 800° ± 10°C. Cool to room temperature in a desiccator and weigh to the nearest 0.1 mg. The weight of the iron and aluminum oxide precipitates is used in the calculation for percent aluminum.

(a) Iron. Fuse the residue in the crucible from 4.2.4.2 with 5 to 10 g of potassium bisulfate. Allow to cool, and dissolve the cooled melt by heating with 100 mL of water containing 5 mL of 1 to 1 sulfuric acid in a beaker. Remove the crucible and wash thoroughly with hot water, adding the washings to the beaker. Pass hydrogen sulfide gas through the solution for

approximately 30 minutes. Filter through Whatman No. 4 filter paper and wash the precipitate with water saturated with hydrogen sulfide and acidified with 1 to 20 sulfuric acid. Catch the filtrate and washings in the same beaker, boil vigorously for 30 minutes to remove the hydrogen sulfide, cool to room temperature, and titrate with 0.1N potassium permanganate solution. Calculate the percent by weight iron as follows:

$$\text{Percent by weight iron} = \frac{5.585\ AB}{W}$$

where: A = Milliliters of potassium permanganate solution used,
B = Normality of potassium permanganate solution, and
W = Weight of specimen in 4.2.4.1 in grams.

(b) Aluminum. Calculate the percent aluminum as follows:

$$\text{Percent aluminum} = \frac{52.91\ A}{W} - 0.757\ B$$

where: A = Weight of iron and aluminum oxide precipitates in 4.2.4.2 in grams,
B = Percent iron calculated in 4.2.4.2(a), and
W = Weight of specimen in 4.2.4.1 in grams.

4.2.4.3 Granulation characteristics. Use sieves conforming to RR-S-366.

(a) Classes A, B, and D. Select the applicable sieves specified in table II. Weigh each sieve to the nearest 0.01 g and then nest them with the coarser sieve on top and place on a bottom pan. Weigh to the nearest 0.1 g approximately 100 g of the specimen and place on the top sieve. Cover the assembly and shake for 5 minutes by hand or by means of a mechanical shaker geared to produce 300 ± 15 gyrations and 150 ± 10 taps of the striker per minute. Weigh the material remaining on each sieve to the nearest 0.01 g and calculate the percent specimen passing through each sieve.

(b) Class C. Weigh to the nearest 0.1 g approximately 50 g of the specimen and transfer to a No. 230 sieve. Wash the specimen with a stream of water until the water comes through clear. Dry the residue at 105° to 120°C for 20 to 30 minutes and shake through the applicable sieves specified in table II using the procedure specified in (a).

4.2.5 Container leakage test. Place the container in each of the following positions, and leave it in each for a period of 15 minutes:

(a) Upright
(b) Upside down
(c) On one side (or one quadrant)
(d) On one end (or second quadrant)
(e) On other side (or fourth quadrant)

Examine the container after each period for any evidence of leakage.

5. PACKAGING

5.1 Unit packing, level C. A quantity of 500 (+5 or -0) pounds of silicon shall be unit packed in a clean, dry, corrosion free steel drum in accordance with Department of Transportation (DOT) Regulations. There shall be no evidence of leakage of contents when tested as specified in 4.2.5.

5.2 Packing. Silicon, unit packed as specified in 5.1, shall require no further protection for shipment.

5.3 Marking. Containers of silicon shall be marked in accordance with DOT regulations and as follows:

HAZARDS:

FLAMMABLE SOLID
TOXIC BY INHALATION

WARNING! FLAMMABLE
WHEN HEATED, REACTS VIOLENTLY WITH WATER, LIBERATING AND IGNITING HYDROGEN.
Keep away from heat, sparks, and open flame.
Keep dry.
WARNING! HARMFUL IF INHALED
Avoid breathing dust.
Keep container closed.
Use with adequate ventilation.
First Aid: If inhaled, remove to fresh air. If not breathing, give artificial respiration, perferably mouth-to-mouth. If breathing is difficult, give oxygen. Call a physician.

6. NOTES

6.1 Intended use. Silicon is intended for use as an ingredient in pyrotechnic compositions.

6.2 Ordering data. Acquisition documents should specify the following:

(a) Title, number, and date of this specification
(b) Grade and class of silicon required (see 1.2)

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.

6.5 Sampling and testing precautions. This specification covers inspection of chemical material which is potentially hazardous to personnel. Powdered silicon is a flammable solid, toxic by inhalation. All applicable safety rules, regulations, and procedures must be followed in the handling and processing of this material.

Custodians:

Army - EA
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Project No. 6810-B337

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5. PROBLEM AREAS

a. Paragraph Number and Wording:

b. Recommended Wording:

c. Reason/Rationale for Recommendation:

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7a. NAME OF SUBMITTER *(Last, First, MI)* – Optional

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