MIL-C-51077(Ord)
1 February 1962

MILITARY SPECIFICATION

CALCIUM SILICATE, TECHNICAL

1. SCOPE

1.1 This specification covers only one type of calcium silicate, absorbent for use in explosives.

2. APPLICABLE DOCUMENTS

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

SPECIFICATIONS

MILITARY

UU-S-48 - Sacks, Shipping, Paper

STANDARDS

MILITARY

MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes.
MIL-STD-109 - Inspection Terms and Definitions.
MIL-STD-129 - Marking for Shipment and Storage.

PUBLICATIONS

ORDNANCE CORPS

ORD-M608-11 - Procedures and Tables for Continuous Sampling by Attributes.

FSC: 6810

(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 Chemical requirements.-The calcium silicate, absorbent shall conform to the chemical composition specified in Table I, when tested as specified in the applicable paragraph.

Table I

Ingredient	Percent Maximum (max.)	Minimum (min.)	Applicable paragraph
Total volatile matter	9.0	- - - -	4.3.1
Total silicon as silicon dioxide (SiO_2) dry basis	64.0	52.0	4.3.2
Total calcium as calcium oxide (CaO) dry basis	32.0	23.0	4.3.3
Sum of percent SiO_2, CaO, and loss of weight on ignition, dry basis	- - - -	97.0	4.3.4
pH	9.0	7.5	4.3.7

3.2 Physical requirements.-The calcium silicate, absorbent shall conform to the physical requirements specified in Table II, when tested as specified in the applicable paragraph.

Table II

Property	Percent Min.	Percent Max.	Applicable paragraph
Granulation			4.3.5
Retained on 140 mesh sieve	----	1.0	
Retained on 325 mesh sieve	----	6.0	
Meta-nitrotoluene absorption	550 gram (gm.)/ 100 gm.	----	4.3.6
Grit**	----	0.01	4.3.8

**Note: The residue shall not produce a scratching noise or scratch the glass slide.

3.2.1 Color.-The calcium silicate, absorbent shall be white in color.

3.3 Workmanship.-All bags shall be dry and all bags, and the material contained therein, shall be free of dirt, oil, grease and other foreign material.

4. QUALITY ASSURANCE PROVISIONS

4.1 General quality assurance provisions.-The supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own or any other inspection facilities and services acceptable to the Government. Inspection records of the examinations and tests shall be kept complete and available to the Government as specified in the contract or order. 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. Inspection shall be performed in accordance with this specification and other specifications referenced in any of the contractual documents.

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 4.2.2 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 4.2.2. and 4.3 and the other documents referenced herein. The contractor shall not be restricted to the inspection station or the method of inspection listed in this specification provided than 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 procedure of this specification shall apply. The contractor shall notify the Government of, and obtain approval for, any changes to the written procedure that effects 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 submitted:

a. A statement that the lot complies with all quality assurance provisions of the approved current written description of the system.

b. Quantity of product inspected.

c. Results obtained for all inspection performed

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

e. Certificates of analysis on all material covered by referenced government specificationsprocured directly by the contractor.

f. Quantity of product in the lot.

g. 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 Government verification.-Using the contractor's written quality assurance procedure (see 4.1.1), this detail specification, and other contractual documents as a guide, the Government inspector shall verify all quality assurance operations performed by the contractor. Verification shall be in accordance with a. or b. as applicable, the decision being the re-

sponsibility of the procuring activity. In either case, the inspector shall also ascertain, prior to acceptance, that all quality assurance provisions of other specifications referenced in any of the contractual documents have been complied with. Deviations from prescribed or agreed upon procedures discovered by the Government inspector shall be brought to the attention of the supplier. Disposition of the product and remedial action shall be as directed by the Government inspector and, depending on the nature of the deviation, may consist of lot rejection, screening, re-sampling, re-instruction of the supplier's employees, or other appropriate action:

a. Verification at the point of manufacture shall be accomplished at unscheduled intervals in accordance with 4.1.3.1 and 4.1.3.2.

b. Verification at the point of delivery shall be in accordance with 4.1.3.2.

4.1.3.1 Surveillance. - Surveillance shall include, but is not limited to:

a. Observation of procedures concerning lot formation and identification.

b. Observation of sampling procedures and application of acceptance criteria.

c. Determination that all required examinations and tests are performed in accordance with the prescribed procedures of this specification, or approved equivalents thereto.

d. Review of procedures for control and disposition of nonconforming material.

4.1.3.2 Product inspection. - Product inspection shall consist of Government inspection of product which has been previously inspected by the contractor and found to meet the quality assurance provisions of this specification. The inspection by the Government shall be performed in order to determine that the product is of the quality required by this specification and that the contractor's records are reliable.

4.2 Inspection provisions. -

4.2.1 Lot formation. - A lot shall consist of one or more batches of calcium silicate, absorbent, 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 calcium silicate, absorbent that has been subjected to the same unit chemical or physical mixing process intended to make the final product homogeneous.

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 Handbook ORD-M608-11 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 0.25 percent for each major defect and an AQL of 0.40 percent for each minor defect.

4.2.2.1 Paper shipping sack, prior to filling (see 5.1).-

Categories	Defects	Method of inspection
Critical: None defined		
Major:	AQL 0.25 percent	
101.	Sack torn, cut or punctured	Visual
102.	Seam incomplete, or improperly finished	Visual
Minor:	AQL 1.00 percent	
201.	Marking misleading or unidentifiable . . .	Visual
202.	Evidence of moisture	Visual-Tactile

4.2.2.2 Paper shipping sack, after filling (see 5.1).-

Categories	Defects	Method of inspection
Critical: None defined		
Major:	AQL 0.25 percent	
101.	Closure incomplete or improper.	Visual
Minor: None defined.		

4.2.3 Testing.-

4.2.3.1 Sampling for tests.- A sample of three pounds shall be selected from each lot for tests. If any sample fails to comply with any of the requirements, the lot shall be rejected.

4.3 Test methods and procedures.-

4.3.1 Determination of total volatile matter.- A portion of approximately five gm. weighed to the nearest milligram (mg.) shall be transferred to a tared glass or aluminum dish. The specimen shall be spread loosely over the bottom of the dish. The dish and contents shall be heated in a drying oven maintained at 105° ± 5 degrees centigrade (°C.) for four hours. The dish shall then be removed and placed in a desiccator. When cool, the dish shall be weighed to the nearest mg. The procedure of heating cooling and weighing shall be repeated at hourly intervals until constant weight is reached or the sample gains weight. The percent of total volatile matter shall be calculated as follows:

$$\text{Percent total volatile matter} = \frac{(A-B)\ 100}{A}$$

where:

A = original weight of the specimen in gms.

B = final weight of the specimen in gms.

4.3.2 Determination of total silicon as SiO_2. - A portion of approximately 0.5 gm. weighed to the nearest 0.1 mg., shall be transferred to a 250 milliliter (ml.) beaker. A 25 ml. portion of concentrated hydrochloric acid shall be added and the mixture shall be carefully evaporated to dryness on a hot plate in the hood. The residue shall be baked for 30 minutes in an oven maintained at 105° ± 5°C. At the end of this period, the beaker shall be removed and allowed to cool to room temperature. The residue shall be moistened with three ml. of concentrated hydrochloric acid and then 50 ml. of water shall be added and the mixture heated to boiling. This mixture shall be filtered through a Whatman Number (No.) 42 paper, or equivalent. The beaker shall be rinsed alternately with three portions of hot water, 20 ml. each, and three portions, 2.0 ml. each of 1:20 hydrochloric acid. Each portion of wash water shall be filtered through the original filter paper. The filtrate shall be evaporated to dryness on a hot plate and the residue treated as above beginning with baking in the oven. The residue shall be allowed to cool and then ten ml. of concentrated hydrochloric acid shall be added and then evaporated to dryness on a hot plate. The residue shall be diluted with water, heated to boiling, filtered and washed as before. The filtrate and washings shall be reserved for the determination of total calcium as CaO. Both filter papers shall be placed in an ignited and tared platinum crucible. The papers shall be dried over a low flame and then permitted to char without flaming. The crucible shall be transferred to a muffle furnace maintained at 950° ± 50°C. for one hour. The crucible shall then be removed and cooled to room temperature in a desiccator. The residue should be a powdery materiel ranging in color from a light gray to a white or off white. If the residue is darker than a light gray, it shall be moistened with concentrated sulfuric acid and then gently heated over a flame in the hood to evaporate the sulfuric acid. It shall be transferred to a muffle furnace and ashed as before for 15 minutes. The crucible shall be removed from the furnace and cooled in a desiccator and weighed. Hydrofluoric acid shall be cautiously added dropwise to the crucible until there is no further reaction and the residue is covered with hydrofluoric acid. Five drops of concentrated sulfuric acid shall be added and the crucible shall be carefully heated on a hot plate until fumes of sulfur trioxide are emitted. The residue shall be allowed to cool, and then five drops of hydrofluoric acid shall be added. The crucible shall be heated as before until fumes are no longer evolved. The crucible shall be transferred to a muffle furnace, maintained at 950° ± 50°C., for one-half hour. The crucible shall then be removed and placed in a desiccator and weighed when cool. The percent of total silicon as SiO_2 shall be calculated as follows:

$$\text{Percent total silicon as } SiO_2 = \frac{(A-B)\ 100}{S\ (1 - \frac{V}{100})}$$

where:

A = weight of crucible in gms. with residue prior to addition of hydrofluoric acid.
B = weight of crucible in gms. after evaporation with hydrofluoric and sulfuric acids.
S = weight of the specimen in gms.
V = percent total volatile matter (see 4.3.1)

4.3.3 Determination of total calcium as CaO.- The filtrate and washings reserved from the SiO_2 determination shall be quantitatively transferred to a 400 ml. beaker. The volume shall be adjusted to approximately 200 ml. and three drops of methyl red indicator added. Five ml. of concentrated hydrochloric acid shall be added, followed by 75 ml. of four percent ammonium oxalate. The solution shall be heated to 75° ± 5°C., and 1-1 ammonium hydroxide added with stirring until just alkaline. The solution shall be allowed to stand at room temperature for one hour with occasional stirring and then filtered through Whatman No. 42 paper, or equal. The precipitate shall be washed four to five times with cold 0.1 percent ammonium oxalate solution. The filtrate shall be discarded. Small portions of hot 1:4 hydrochloric acid, making a total of approximately 50 ml. shall be poured on the precipitate. The filtrate shall be caught in a 400 ml. beaker. The filter shall be washed with several small portions of 1:100 hydrochloric acid. The filtrate shall then be diluted to 200 ml. A 50 ml. portion of four percent ammonium oxalate solution shall be added. The solution shall be heated to incipient boiling. Calcium oxalate shall be precipitated by adding 1:1 ammonium hydroxide until the solution is just alkaline to methyl red and then by adding five ml. in excess. The solution shall be allowed to stand for one hour and then filtered through a Whatman No. 42 filter or equal. The beaker and the precipitate shall be washed with small portions of ice cold water until the washings are free of chloride ions. The minimal amount of water required shall be used. The filter paper and precipitate shall be returned to the beaker in which the precipitation took place. The precipitate shall be dissolved by adding 100 ml. of warm 1:10 sulfuric acid to the beaker, using a stirring rod to facilitate solution while warming. The solution shall be heated to 80°C. and then titrated at this temperature with standard 0.1 normal (N) potassium permanganate solution. The percent of total calcium as CaO shall be calculated as follows:

$$\text{Percent total calcium as CaO} = \frac{A \times N \times 2.804}{S\left(1 - \frac{V}{100}\right)}$$

where:

A = volume in ml. of standard permanganate solution used in titration.
N = normality of the standard permanganate solution.
S = weight of the specimen in gms.
V = percent total volatile (see 4.3.1).

4.3.4 Sum of percents SiO_2, CaO, and loss in weight on ignition.- The loss in weight on ignition shall be determined as follows: A 2.0 gm. portion of the sample, weighed to the nearest 0.1 mg., shall be transferred to a previously ignited and tared crucible. The crucible and contents shall be gently ignited over a small flame in the hood. After the contents have been thoroughly charred, or fumes are no longer emitted, the crucible shall be more strongly ignited. After such treatment for ten minutes, the crucible and its contents shall be transferred to a muffle furnace, maintained at 980° ± 15°C., for one hour. At the end of this period the crucible shall be removed and placed in a desiccator. When cool, the crucible shall be weighed. The percent loss in weight on ignition shall be calculated as follows:

$$\text{Percent loss in weight on ignition} = \frac{(A-B)\ 100}{A\left(1 - \frac{V}{100}\right)}$$

where:

A = weight of original specimen in gms.
B = weight of specimen in gms. after ignition.
V = percent total volatile matter (see 4.3.1).

The sum of percents SiO_2, CaO, and loss in weight on ignition shall be calculated as follows:

Sum of percents SiO_2, CaO and loss in weight on ignition = A+B+C

where:

A = percent SiO_2 (see 4.3.2).
B = percent CaO. (see 4.3.3).
C = percent loss in weight on ignition (see 4.3.4).

4.3.5 Granulation. - A 10.0 gm. portion of the material, weighed to the nearest 0.01 gm. on a torsion balance or equivalent, shall be transferred to a 250 ml. beaker. Slowly and carefully, 100 ml. of water shall be added down the side of the beaker so as not to disturb the powder. The mixture shall be permitted to stand undisturbed until the powder has been completely wetted. The slurry shall be transferred with the aid of a gentle stream of water, to a No. 140 United States (U.S.) Standard sieve nested over a No. 325 U. S. Standard sieve seated in a large casserole or dish. The balance of the residue n the beaker shall be transferred quantitatively to the No. 140 sieve, by means of small washes of water. When the entire slurry has been washed onto the No. 140 sieve, the washing shall be continued using a fine camel's hair brush to manipulate the slurry on the sieve. When the water passing through the sieve is clear, the sieve shall be removed and dried in an oven, maintained at $105^{\circ} \pm 5^{\circ}C$. for four hours. The sieve shall then be removed, cooled to room temperature and weighed. This residue shall be retained for the grit determination. The gain in weight is due to particles too large to pass through the meshes of the sieve. In the interim, while the No. 140 sieve is drying, the slurry remaining on the No. 325 sieve shall be washed using the same procedure as for the No. 140 sieve. The No. 325 sieve shall be dried as the No. 140. This residue shall also be retained for the grit determination. The percent by weight retained on the No. 140 and No. 325 sieves shall be calculated as follows:

a. Percent retained on No. 140 sieve $= \frac{(A-B) \times 100}{S}$

where:

A = weight of No. 140 sieve and residue in gms.
B = weight of No. 140 sieve in gms.
S = weight of specimen in gms.

b. Percent retained on No. 325 sieve $= \frac{(C-D) \times 100}{S}$

where:

C = weight of No. 325 sieve and residue in gms.
D = weight of No. 325 sieve in gms.
S = weight of specimen in gms.

4.3.6 Meta-nitrotoluene absorption. - A 5.0 gm. specimen weighed to the nearest 0.01 gm. shall be transferred to a 500 ml. casserole and meta-nitrotoluene shall be added from a burette at the rate of 0.15 to 0.20 ml. per second. During the addition of the liquid, the mixture shall be thoroughly stirred with a four inch blade spatula. The flow from the burette shall be stopped when the mixture becomes slightly fluid and the

liquid shall be added drop by drop until the end point is reached. The end point shall be reached when the mixture is sufficiently fluid to drop off the end of the spatula in individual drops when held in a vertical position. This test shall be completed within 10 minutes (max.).

4.3.6.1 Calculation.- Multiply the volume delivered by 23.2 and report as gms. meta-nitrotoluene absorbed per 100 gms. of sample.

4.3.7 pH.- A ten gm. portion of the sample shall be weighed to the nearest 0.01 gm. on a torsion balance or equivalent scale, and placed in a 250 ml. beaker. A 100 ml. portion of distilled water shall be added slowly down the side of the beaker. The mixture shall be stirred with a glass stirring rod and then allowed to stand for ten minutes. The solution shall then be filtered with suction through a Buchner funnel using a previously washed Whatman No. 1 filter paper or equivalent. The first ten ml. of the filtrate shall be discarded. The pH of the filtrate shall be taken using a Beckman glass electrode pH meter, or equivalent. (The pH of the distilled water employed in this determination, should be between 6.0 and 7.0).

4.3.8 Grit.- The residue obtained from the granulation determination in paragraph 4.3.5 shall be transferred to a tared weighing bottle, dried at 100° to 105°C. for one hour, cooled in a desiccator and weighed. Calculate the weight of residue to percent grit. After performing the test place the material between two glass slides. Rub the slides together to determine the presence of grit by scratching noise and scratches on the glass slide.

5. PREPARATION FOR DELIVERY

5.1 Preservation and packaging.-

5.1.1 Level A.- The calcium silicate absorbent shall be packaged in conformance with Specification UU-S-48, sack construction No. 2X of table I.

5.2 Marking.- Marking shall be in accordance with Standard MIL-STD-129.

6. NOTES

6.1 Ordering data.- Procurement documents should specify the title, number, date of this specification, and quantity required.

6.2 Intended use.- The calcium silicate is intended for use in explosive compositions containing Trinitrotoluene (TNT) (to prevent exudation of TNT).

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 in any way be related thereto.

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