

MIL-Z-50976 (MU)  
30 September 1971

## MILITARY SPECIFICATION

### ZIRCONIUM SPONGE

#### 1. SCOPE

1.1 This specification covers zirconium sponge (see 6.4) for use in the manufacture of ordnance materials.

2.2 Classification.-Zirconium sponge covered by this specification shall be of the following classes as specified (see Table 1).

- Class 1 - Pass through 3/4" screen and retain by #6 screen
- Class 2 - Pass through 3/8" screen and retain by #6 screen
- Class 3 - Pass through 1/4" screen and retain by #6 screen
- Class 4 - Pass through #6 screen and retain by #8 screen

#### 2. APPLICATION 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.

#### SPECIFICATION

##### FEDERAL

RR-S-366 - Sieve, Test

#### STANDARDS

##### MILITARY

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

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MIL-STD-109	-	Quality Assurance Terms and Definitions
MIL-STD-129	-	Marking for Shipment and Storage
MIL-STD-1168	-	Lot Numbering of Ammunition
MIL-STD-1235	-	Single and Multilevel Continuous Procedures and Tables for Inspection by Attributes

(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).

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

ASTM E10-66	-	Test for Brinell Hardness of Materials
ASTM D11-93	-	Reagent Water

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania, 19103).

Analytical Chemistry - Vol. 24, pp 1869-1870 (1952)

#### CODE OF FEDERAL REGULATIONS

Title 49	-	Transportation, Parts 0-190
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(The Interstate Commerce Commission Regulations are now a part of the Code of Federal Regulations, available from the Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402. Orders for the above publications should cite, "49 CFR 0-190 (latest revision)".)

### 3. REQUIREMENTS

3.1 Particle size distribution.-Zirconium sponge shall conform to the particle size distribution specified in Table I, when determined in accordance with 4.4.2.

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TABLE I - Particle size distribution for zirconium sponge

<u>SIEVE SIZE</u>	<u>CLASS 1</u>		<u>CLASS 2</u>		<u>CLASS 3</u>		<u>CLASS 4</u>	
	<u>Thru</u>	<u>On</u>	<u>Thru</u>	<u>On</u>	<u>Thru</u>	<u>On</u>	<u>Thru</u>	<u>On</u>
3/4"	100%							
1/2"	33.3+6%							
3/8"	100%							
1/4"	33.3+6%		50+10%		100%			
#4	50+15%							
#6	0.5% max		0.5% max		0.5% max		100%	
#8	0.5% max.							

3.2 Particle shape.-The shape of zirconium sponge particles shall resemble the particles as shown in Figure 1.

3.3 Hardness of zirconium sponge.-Zirconium sponge shall have a Brinell hardness of not less than 120 nor greater than 190 when tested as specified in 4.4.3.

3.4 Tap density of zirconium sponge.-Zirconium sponge shall meet the following tap density requirements when tested as specified in 4.4.4.

Class 1	120 lbs/cu.ft	min.
Class 2	130 lbs/cu.ft	min.
Class 3	130 lbs/cu.ft	min.
Class 4	135 lbs/cu.ft	min.

3.5 Chemical requirements.-The zirconium sponge shall conform to the chemical requirements as specified in Table 2 when tested in accordance with the applicable paragraph.

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TABLE 2 - Chemical requirements for zirconium sponge

<u>TEST</u>	<u>REQUIREMENT</u>	<u>TEST PARAGRAPH</u>
Zirconium, percent, min.	94.7%	4.4.8
Hafnium, percent, max.	4.5%	4.4.7
Chloride (as Cl <sup>-</sup> ), percent max.	0.15%	4.4.5
Fe, Mg, O <sub>2</sub> + trace elements, percent, max	0.65%	4.4.6

3.6 First article testing.-This specification makes provisions for first article testing. Requirements for the submission of first article samples by the contractor shall be as specified in the contract.

#### 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 prescribed requirements. Reference shall be made to MIL-STD-109 to define terms used herein.

4.4.1 Submission of product.-At the time each completed lot of items deliverable under the contract is submitted to the Government for acceptance, the contractor shall supply the following information accompanied by a certificate which attest that the information provided is correct and applicable to the product being submitted:

- a. A statement that the lot complies with all of the quality assurance provisions specified in this specification.
- b. Specification number and date, together with identification and date of changes thereto.
- c. Certificates of analysis on all materials 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.

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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.

#### 4.2. First article inspection

4.2.1 Submission.-Prior to the start of regular production the contractor shall submit a first article sample (see 6.1c) to a Government approved facility as designated by the contracting officer for evaluation in accordance with the provisions of 4.2.2. The first article shall consist of 50 lbs. of zirconium sponge in accordance with instructions issued by the Contracting Officer. All samples submitted shall have been produced by the contractor 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 analysis. 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.

4.2.2 Inspections to be performed.-The sample will be subjected by the Government to any or all of the examinations or tests specified in 4.3 and 4.4 of this specification.

4.2.3 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.

#### 4.3 Inspection provisions

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4.3.1 Lot formation.-A lot shall consist of one or more batches of zirconium produced by one manufacturer, in accordance with the same specification, or same specification revision, under one continuous set of operating conditions. Each lot shall consist of that quantity of zirconium 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 MIL-STD-105. The criteria and procedures for the assignment of lot numbers shall be in accordance with Standard MIL-STD-1168.

4.3.2 Examination.-Sampling plans and procedures for the following classifications of defects shall be in accordance with MIL-STD-105 (ABC-STD-105), except that inspection for critical defects shall be 100 percent. Contractor's sampling plans, if used, shall be approved by the Government and shall provide, as a minimum, the protection afforded the Government by the sampling plans in MIL-STD-105. Continuous sampling plans in accordance with 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 except where 100 percent inspection is specified.

## 4.3.2.1 Container

Categories	Defects	Method of Inspection	Code No.
Critical:	None defined		
Major:	None defined		
Minor:	AQL 0.65 Percent		
	201. Marking missing, incorrect or illegible	Visual	01001

4.3.3 Sampling.-All sampling of individual containers shall be accomplished in such a manner that the sample is representative of the entire contents of the container. Individual or composite samples shall be identified in such a manner as to provide positive identification as to the container or containers represented by each sample. For the chemical composition tests, the lot shall be divided up into two (2) parts. From each part, a composite sample shall be prepared in the following manner. A

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random sample of filled containers shall be selected in accordance with MIL-STD-105. However, if each part comprises less than five (5) containers, all containers shall be selected. Approximately equal portions shall be taken from the selected containers in such quantities that the aggregate weight of such portions shall be approximately five (5) pounds (lbs.) These portions shall then be thoroughly mixed to form a composite sample. For the physical tests, the lot shall be divided up into four (4) parts. For each part, a composite sample shall be prepared as described above. The aggregate weight of each composite sample shall be approximately 10 lbs. If any of the above samples fails to meet any of the requirements specified in Section 3, the lot shall be rejected.

#### 4.4 Test methods and procedures

4.4.1 Tests.-Except as specified herein, reagent grade chemicals and distilled water in accordance with ASTM D11-93 shall be used throughout the tests.

4.4.2 Particle size distribution - Major defect - Code No. 02001.-Nest the specified sieves (see 3.1) on a bottom pan. The sieves shall comply with the requirements of RR-S-366. Place a weighed portion of 100 gm of the sample on the upper sieve. Cover the sieve and shake for 30 minutes in a mechanical shaker geared to produce 300 + 15 gyrations and 150 + 10 taps of the shaker per minute. Weigh the amounts retained or passing through the sieves, and calculate the required percentages.

4.4.3 Hardness of zirconium sponge - Major defect - Code No. 03001.-A button shall be prepared from the zirconium sponge metal by a suitable arc melting process. From this button, a hardness test shall be performed in accordance with ASTM E10-66.

4.4.4 Tap density.- Major defect - Code No. 04001.-A 250 ml graduated cylinder containing an accurately weighed sample is inserted into a machine for determining packed density. This machine is described in "Analytical Chemistry" volume 24, Nov. 1952. The machine is then operated at a basic rate of 30 drops per minute, for a period of 10 minutes. After this operation, the volume is obtained. Calculate the tap density as follows:

$$\frac{\text{Weight of sample (grams)}}{\text{Volume of sample (ml)}} = \text{Tap density (g/ml)}$$

$$\text{Tap density (g/ml)} \times 62.4 = \text{Tap density (lbs/cu.ft)}$$



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4.4.5 Determination of chloride - Major defect - Code No. 05001.

4.4.5.1 Reagents

4.4.5.1.1 Boric acid solution (5 percent).-Dissolve 100 g. of boric acid in warm water, cool, and dilute to 2 liters.

4.4.5.1.2 Silver nitrate solution (5 percent).-Dissolve 25 g. of silver nitrate in water and dilute to 500 ml. Store in a dark bottle.

4.4.5.2 Procedure.-Weigh a 2 gm. sample into a plastic bottle and add 20 ml. of water. Add 3 ml. of hydrofluoric acid in 0.5 ml. portions, swirling vigorously after each addition. Add 2 ml. of nitric acid and 120 ml. of boric acid solution (5 percent). Filter through a Whatman No. 42 filter paper into a plastic beaker and wash with water. Add 5 ml. of silver nitrate solution (5 percent) and place the plastic beaker into a 1 liter beaker containing about 700 ml. of tap water that is boiling on the hot plate. Allow to stand for 15 minutes in the boiling water (the temperature of the solution in the plastic beaker will rise to 75° to 80°C during this time). If the supernatant liquid is not clear after this period of time, let the solution stand until the supernatant liquid clears. Remove the plastic beaker and test for completeness of precipitation by adding a few drops of the silver nitrate solution. If additional precipitate forms, add an additional 2 - 3 ml. of silver nitrate solution and repeat the above operation. After the plastic beaker is removed, cover it with a watch glass and allow to stand for 3 or more hours in a dark place. Filter through a tared sintered glass crucible of fine porosity, transfer and wash with 1 percent nitric acid and finally wash once with water. Dry at 125°C for 1 hour, cool, and weigh. Carry through a blank. Calculate as follows:

$$\text{Percent chloride} = \frac{24.74 (A-B)}{W}$$

Where: A = gm. of precipitate from sample

B = gm. of precipitate from blank

W = gm of sample

4.4.6 Determination of iron, magnesium, oxygen and trace elements - Major defect - Code No. 06001.-Iron, magnesium, oxygen, and trace elements shall be determined by satisfactory spectrographic methods.



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4.4.7 Determination of hafnium - Major defect - Code No. 07001.-  
Hafnium shall be determined by a satisfactory spectrographic method.

4.4.8 Determination of zirconium - Major defect - Code  
No. 08001.

#### 4.4.8.1 Reagents

4.4.8.1.1 Mandelic acid solution (15 percent).-Dissolve  
75 gm. of mandelic acid in water and dilute to 500 ml.

4.4.8.1.2 Mandelic acid wash solution.-Dissolve 25 gm. of  
mandelic acid in 400 ml. of water, add 10 ml. of hydrochloric  
acid, and dilute to 500 ml.

4.4.8.2 Procedure.-Transfer a 0.23 to 0.25 gm. of the  
sample, weighed to 0.1 mg. to a large platinum dish. Add 15 ml.  
of water, 10 ml. of sulfuric acid (1 to 1), 5 ml. of nitric acid  
and 2 ml. of perchloric acid. Cover with a plastic watch glass.  
Add 5 ml. of hydrofluoric acid in 0.5 ml. portions from a plastic  
medicine dropper, while swirling the platinum dish and allowing  
about 1 minute between additions. After the final addition, allow  
to stand for five minutes to complete solution, then wash down  
the watch glass with water. Evaporate to fumes of sulfuric acid  
at gentle heat without the watch glass, remove the dish from the  
hot plate, cool it in water, wash down the sides with water, and  
swirl. Again evaporate to fumes of sulfuric acid at gentle heat,  
then raise the temperature of the hot plate to moderate heat, and  
evaporate to dryness and the disappearance of fumes. Cool somewhat,  
add 50 ml. of water and 10 ml. of hydrochloric acid, swirl, and  
heat on the hot plate at gentle heat for 5 to 10 minutes until  
the solution is clear. Wash into a 400 ml beaker and police the  
platinum dish.

Dilute to approximately 120 ml., put in a stirring rod, and heat  
to about 80°C on the hot plate. Add 75 ml. of mandelic acid  
solution (15 percent) from a graduate over a period of 15 to 30  
seconds while stirring the solution with the stirring rod. Stir  
frequently during the next 5 minutes. Cover with a watch glass and  
allow to stand for about 80°C for 40 minutes while stirring  
occasionally. Wash down the cover glass with water, filter through  
a Whatman No. 40 filter paper, and collect the filtrate in a  
clean 400 ml. beaker. Police the stirring rod and beaker, and  
wash the filter paper and precipitate with mandelic acid wash  
solution. Place the filter paper and precipitate into a tared  
platinum crucible and heat on the hot plate at highest heat for  
1 hour or more.

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Within 30 minutes after the filtration, wash the filtrate into the original 400 ml. beaker, cover with a watch glass (but do not insert a stirring rod), and heat to about 80°C on the hot plate. Place the beaker in an oven at about 80°C, cover with an inverted 1 liter beaker, and allow to stand overnight. Filter through a Whatman No. 40 filter paper and wash with mandelic acid wash solution. Place the filter paper into the crucible containing the first precipitate and heat on the hot plate at the highest heat for 1 hour or more. Burn off the carbonaceous material over the low flame of a Meker burner and then heat over the full flame of the Meker burner until the precipitate is white. Ignite over a blast burner for 20 minutes, cool in a desiccator, and weigh. Calculate as follows:

$$\text{Percent zirconium} = \frac{74.03A}{W}$$

Where: A = gm. of precipitate  
W = gm of sample

Hafnium is counted with the zirconium. Therefore subtract the value of hafnium (as determined in 4.4.7) from the above to obtain the true zirconium value.

## 5. PREPARATION FOR DELIVERY

5.1 Packing, Level C.-Unless otherwise specified, zirconium sponge shall be packed in commercial, open top, steel drums, 30-gallon capacity of suitable strength for the weight. Net weight shall be approximately 500 pounds per drum; all drums of any lot shall be packed to the same net weight. Drums shall be clean and free of any coating or foreign material which may contaminate the contents. Plastic liners may be used, if necessary, to prevent contact between the contents and coatings used on the interior of the drum. If a plastic liner is used, the contractor shall specify its nature in the bid. Drums from one manufacturer shall be of one type and color.

5.2 Labeling and marking.-Marking shall be in accordance with MIL-STD-129.

## 6. NOTES

6.1 Ordering data.-Procurement documents should specify the following:

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- a. Title, number and date of this document.
- b. Data cards shall be prepared for each lot in accordance with Standard MIL-STD-1167.
- c. Provisions for submission of first article samples.

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.

6.3 Intended use.-The zirconium covered by this specification is intended to be used in explosives.

6.4 Zirconium sponge shall be produced from the magnesium reduction process.

CUSTODIAN:  
ARMY-MU

PREPARING ACTIVITY:  
ARMY-MU

PROJECT NUMBER: 6810-A-914



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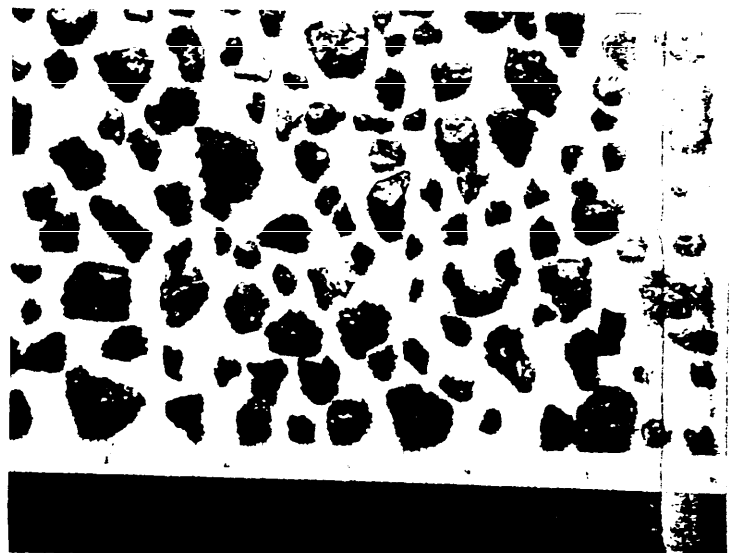


Figure 1 - Particle Shape of Zirconium Sponge



SPECIFICATION ANALYSIS SHEET		Form Approved Budget Bureau No. 22-R255
<p><b>INSTRUCTIONS:</b> This sheet is to be filled out by personnel, either Government or contractor, involved in the use of the specification in procurement of products for ultimate use by the Department of Defense. This sheet is provided for obtaining information on the use of this specification which will insure that suitable products can be procured with a minimum amount of delay and at the least cost. Comments and the return of this form will be appreciated. Fold on lines on reverse side, staple in corner, and send to preparing activity. Comments and suggestions submitted on this form do not constitute or imply authorization to waive any portion of the referenced document(s) or serve to amend contractual requirements.</p>		
SPECIFICATION		
ORGANIZATION		
CITY AND STATE		CONTRACT NUMBER
MATERIAL PROCURED UNDER A <input type="checkbox"/> DIRECT GOVERNMENT CONTRACT <input type="checkbox"/> SUBCONTRACT		
1. HAS ANY PART OF THE SPECIFICATION CREATED PROBLEMS OR REQUIRED INTERPRETATION IN PROCUREMENT USE? A. GIVE PARAGRAPH NUMBER AND WORDING.		
B. RECOMMENDATIONS FOR CORRECTING THE DEFICIENCIES		
2. COMMENTS ON ANY SPECIFICATION REQUIREMENT CONSIDERED TOO RIGID		
3. IS THE SPECIFICATION RESTRICTIVE? <input type="checkbox"/> YES <input type="checkbox"/> NO (If "yes", in what way?)		
4. REMARKS (Attach any pertinent data which may be of use in improving this specification. If there are additional papers, attach to form and place both in an envelope addressed to preparing activity)		
SUBMITTED BY (Printed or typed name and activity - Optional)		DATE

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1 JAN 66

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