MILITARY SPECIFICATION

ZIRCONIUM CARBIDE

This specification is approved for use by the Naval Air Systems Command, Department of the Navy, and is available for use by all Departments and Agencies of the Department of Defense.

1. SCOPE.

1.1 Scope. This specification establishes the requirements for zirconium carbide.

2. APPLICABLE DOCUMENTS.

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

STANDARDS

MILITARY

MIL-STD-129	Marking for Shipment and Storage.
MIL-STD-1218	ACS Chemicals.

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

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commanding Officer, Naval Air Engineering Center, Engineering Specifications and Standards Department (ESSD) Code 93, Lakehurst, NJ 08733, by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

FSC 6810

2.2 Non-Government documents. The following document forms 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.

STANDARDS

American Society for Testing and Materials (ASTM)

ASTM-E-146 — Standard Method for Chemical Analysis of Zirconium and Zirconium-Base Alloys.

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

3. REQUIREMENTS.

3.1 Chemical and physical properties. Properties of the zirconium carbide shall conform to Table I.

TABLE I. Chemical and physical properties.

Property	Min	Max
Weight median diameter, micrometers[1/]	6.0	10.0
Fisher average particle diameter, micrometers	2.0	5.0
Zirconium plus hafnium, percent	87.9	...
Hafnium, percent	...	2.0
Total carbon, percent	11.3	...
Free carbon, percent	...	0.5
Iron, percent	...	0.05
Oxygen	...	0.6
Nitrogen	...	0.8

[1/] MSA analysis (Mine Safety Appliance Company, Pittsburg, PA).

3.2 Stability. The zirconium carbide (when protected from contamination) shall have a storage life of 18 months from date of delivery to the procuring activity.

3.2.1 Storage life extension. The storage life of an individual lot (see 6.3.1) may be extended for an additional 18 months provided the zirconium carbide, upon retest, sucessfully meets the requirement for weight mean diameter.

3.3 Toxic products and safety. Safety regulations and guidelines applicable to the use of zirconium carbide should be complied with to preclude personal injury and damage to equipment and facilities.

3.4 Workmanship. Workmanship shall be such that the zirconium carbide is uniform in appearance, of consistent high quality and free from visible contamination.

4. QUALITY ASSURANCE PROVISIONS.

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order (see 6.2.1), the contractor shall be responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the contractor 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.

4.2 Sampling. The lot shall be sampled in accordance with Table II.

TABLE II. Sampling plan.

Number of containers in lot	Number of containers sampled (primary sample)	Number of composite samples
100 or more	10% (nearest whole number)	5
51-99	10	4
11-50	10	3
1-10	all	2

4.2.1 Primary samples. Physical property tests shall be run on each primary sample (see Table II). From each container to be sampled, take a sample of approximately one pint by repeated fillings of an aluminum tube approximately 3/4-inch in diameter and long enough to reach the bottom of the container. Fill and seal a one pint glass jar with sample. Label each jar with the date, lot number, and container number. Failure of any primary sample to pass all of the physical-properties tests herein shall result in rejection of the lot represented.

4.2.2 Composite samples. Chemical property tests shall be run on each composite sample. Divide the primary samples equally into the number of composites shown in Table II. Blend each composite thoroughly by manipulation of the container. Label each composite with Roman

numerals, also include date, lot numbers, and manufacturer's container identification numbers. The remainder of the primary samples shall be retained pending acceptance or rejection of the lot. Failure of any composite sample to pass all of the chemical-properties tests herein shall result in rejection of the lot represented.

4.3 Quality conformance inspections and tests. Quality conformance inspections and tests shall consist of the following:

a. Tests of Table I properties (see 4.4).

b. Inspection of filled containers (see 4.5.1).

c. Visual inspection (see 4.5.2).

4.4 Test methods. Tests shall be performed using apparatus, reagents, and procedures specified herein. The use of alternate apparatus, reagents, or procedures shall require prior written approval of the procuring activity. All American Chemical Society (ACS) reagents shall conform to MIL-STD-1218.

4.4.1 Weight mean diameter.

4.4.1.1 Apparatus.

a. MSA particle size analysis equipment, or equal.

b. Codex Number 3128 logarithmic probability graph paper, or equal.

4.4.1.2 Reagents.

a. Chlorobenzene, American Chemical Society (ACS) reagent.

b. Surfactant - Twitchell Base 8240 (Emery Industries, Inc., Cincinnati, Ohio), or equal.

c. Benzene, ACS reagent.

4.4.1.3 Determination of weight median diameter. Weigh a 0.008 gram (gm) sample onto a tared flat glass plate. Add one drop of Twitchell Base 8240 surfactant. Disperse the sample for 1 minute with the blade of a steel spatula. Using the sharpened edge of the spatula, place the sample in a MSA transfer chamber. Add approximately 1 milliliter (ml) of MSA feed liquid (prepared by mixing chlorobenzene and benzene in a 60:40 volumetric ratio). Holding the chamber firmly between the thumb and index finger, shake briskly for 30 seconds to bring the sample into

suspension. Introduce the sample into a 0.75-millimeter (mm) MSA analysis tube and test in accordance with the following schedule at 25 degrees Celsius (°C).

	MSA Centrifuge, revolutions per minute			
Diameter, Micrometers	300 Min:Sec	600 Min:Sec	1200 Min:Sec	1800 Min:Sec
15	0:15			
9	0:28			
5	1:34			
3		1:10		
2		2:08		
1			2:50	
0.8			2:11	
0.6				2:24
0.4				5:58
		End Point		15:00

The sample height (h) in the capillary of the analysis tube shall be read at each size interval and at the end point (h_{max}). The cumulative weight percent greater than the stated diameter (percent > d) is determined as follows:

$$\text{percent} > d = \frac{h}{h_{max}} \times 100$$

Average the percent > d values of duplicate runs and plot on Codex logarithmetic probability graph paper. Plot the stated sizes in micrometers versus the percent > values. The weight median diameter of the distribution will be that size in micrometers at the 50 percent point on the distribution curve.

4.4.2 Fisher average particle diameter.

4.4.2.1 Apparatus. Fisher subseive sizer and associated equipment, or equal.

4.4.2.2 Determination of Fisher average particle diameter. Weigh exactly 6.73 gm into the Fisher sample tube containing a porous plug covered with a Fisher filter paper disc. Mount the tube on the instrument rack and pinion attachment. Compress the sample to a porosity of 0.45 to 0.47 using a torque wrench. Move the Fisher chart laterally until the tip of the rack pointer coincides with the sample height curve

on the chart. Move the sample tube to the analysis position and clamp in place. Turn on the instrument, previously calibrated with the Fisher Ruby Calibrator. After the water in the manometer has reached its maximum height, raise the pointer to just cover the water level. Read the size in micrometers at the tip of the pointer directly from the chart. Report the average of two analyses as the Fisher average particle diameter.

4.4.3 Zirconium. The zirconium content shall be determined by difference (i.e., sum of the measured elements except hafnium subtracted from the total).

4.4.4 Hafnium. The hafnium content shall be determined by X-ray fluorescence, or equal.

a. Conditions

Analytical line	Hf 1 $L\beta_1$
Background	41.00
Crystal	LiF 200
Medium	Vacuum
Count time	40 sec
Anode material	Cr
KV	50
Ma	30
Detector	Scintillation
Collimator	Fine
Vessel	Steel plus carbon disk

b. Sample preparation

Borax - beads

1. containing 5 g $Li_2B_4O_7$ + 0.2 g sample

2. containing 5 g $Li_2B_4O_7$ + 0.2 g sample plus known amounts of pure HfO_2 (1 percent, 2 percent, 5 percent)

4.4.5 Total carbon. The total carbon shall be determined by the micro-Pregl combustion technique, or equal.

4.4.6 Free carbon. The free carbon shall be determined as follows:

a. Approximately 1 to 2 gm of sample are weighed into a platinum crucible. Add 25 cm^3 of concentrated hydrofluoric acid and then heat on a sand bath. Nitric acid is added dropwise until sample is dissolved. Distilled water is added and the contents of the crucible are quantitatively rinsed into a porcelain filter crucible. Filter crucible and contents are then dried in an oven at 105°C.

b. The filtration residue is carefully scraped off with a sharp spatula (the bottom of the crucible has to be completely white if this is properly performed), onto a porcelain combustion boat and the carbon content determined by the micro-Pregl combustion technique, or equal.

4.4.7 Iron The iron content shall be determined in accordance with ASTM E 146 using a 1.000 gm sample that is first calcined in air. Dissolve the oxide in about 20 cm^3 of hydrofluoric acid by gently heating. After complete dissolving, cautiously add 10 cm^3 of sulfuric acid and boil the solution to dense white fumes. Continue as described in the ASTM method.

4.4.8 Oxygen. The oxygen content shall be determined by the micro Unterzaucher combustion technique, or equal.

4.4.9 Nitrogen. The nitrogen content shall be determined by the Dumas method, or equal.

4.5 Examinations.

4.5.1 Inspection of filled containers. All filled containers shall be inspected prior to shipment or use for accuracy of markings and for defects in containers and closures. All defective containers and closures shall be repaired or replaced, and contents therein shall be reinspected prior to shipment or use.

4.5.2 Visual inspection. All samples shall be visually inspected to determine conformance to the requirements of 3.4.

4.6 Records. Certification and test data shall be prepared as required by the procuring activity (see 6.2.2).

5. PACKAGING.

5.1 Packaging and packing. Unless otherwise specified in the purchase order(see 6.2.1), packaging and packing of the zirconium carbide shall be in accordance with commercial practice to ensure carrier acceptance and shall be of such construction and materials that the contents will be adequately protected against loss or contamination.

5.2 Marking for shipment. Unless otherwise specified in the purchase order (see 6.2.1), each shipping container shall be marked in accordance with the requirements of MIL-STD-129. Container marking shall include the following:

a. The supplier's lot number.

b. Procuring activity purchase order number.

c. Container identification (applied in numerical sequence as the containers are filled).

d. Date of manufacture.

e. Manufacturer's Code Ident.

f. Net and tare weight of the container.

g. Material identification.

6. NOTES AND CONCLUDING MATERIAL.

6.1 Intended use. The intended use of the material described herein is as an ingredient in solid propellant formulations for rocket motors.

6.2 Ordering data.

6.2.1 Procurement requirements. Procurement documents should specify the following:

a. Title, number and date of this specification.

b. Responsibility for inspection and inspection facilities if different than 4.1.

c. Special packaging, packing, or shipping requirements, if applicable (see Section 5).

6.2.2 Data requirements. When this specification is used in a procurement which incorporates a Contract Data Requirements List (DD Form 1423) and invokes the provisions of 7-104.9(n) of the Defense Acquisition Regulations (DAR), the data requirements identified below will be developed as specified by an approved Data Item Description (DID) (DD Form 1664) and delivered in accordance with the approved DD Form 1423 incorporated into the contract. When the provisions of DAR-7-104.9(n) are not invoked, the data specified below will be delivered by the contractor in accordance with the contract requirements. Deliverable data required by this specification is cited in the following paragraphs:

Paragraph	Data Requirement	Applicable DID
4.6	Certification	UDI-A-23264B
	Test data	DI-T-4024

(Copies of DIDs required by the contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

6.3 Definitions.

6.3.1 Lot. At place of manufacture, a lot consists of one batch (6.3.2) or a uniform blend of two or more batches. At place of delivery, a lot consists of zirconium carbide from one supplier's lot received in a single shipment. Partial shipments may be considered as a single shipment by the procuring activity.

6.3.2 Batch. A batch consists of zirconium carbide made as one unit in an unchanged manufacturing process.

6.4 Suggested source of supply. A product that has met the requirements of this specification in past procurement actions is marketed by Herman C. Starck, Inc., 280 Park Ave (West Building), New York, N.Y. 10017, as Vacuum Quality Zirconium Carbide. This information is for the convenience of the procuring activity and is not to be construed as a waiver of any requirement of this specification nor as any limitation of additional potential sources of supply.

Preparing activity:
Navy - AS

(Project 6810-NB25)

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DOCUMENT IDENTIFIER *(Number)* AND TITLE

MIL-Z-85500(AS), ZIRCONIUM CARBIDE

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specification shall be performed

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