MIL-Z-46189
2 October 1986
SUPERSEDING
AMMRC-PD-106
12 July 1982

MILITARY SPECIFICATION

ZIRCONIUM USED IN GASLESS MIXTURES

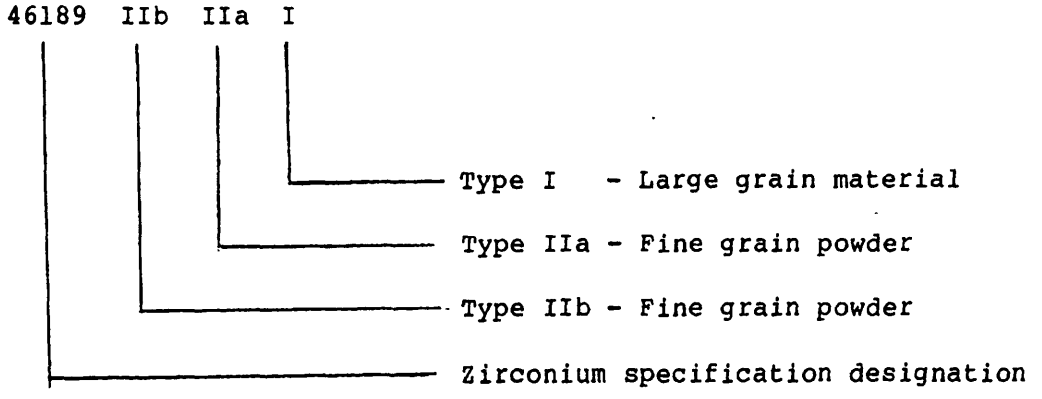
1. SCOPE

1.1 Scope. This specification for zirconium powder applies to zirconium produced by the magnesium reduction process, and covers zirconium metal in fine powder form for use in the manufacture of thermal batteries.

1.2 Classification. This product shall be of the following types: (see 6.2)

Type I - Large grain material of nominal particle size - 2.0 - 3.0 μm
Type IIa - Fine grain powder of nominal particle size - 1.2 - 2.6 μm
Type IIb - Fine grain powder of nominal particle size - 1.5 - 2.3 μm

1.2.1 Part numbering designation.



2. APPLICABLE DOCUMENTS

2.1 Government documents.

2.1.1 Specifications, standards, and handbooks. The following specifications, standards, and handbooks form a part of this specification to the extent specified herein. Unless otherwise specified, the issues of these documents shall be those listed in the issue of the Department of Defense Index of Specifications and Standards (DODISS) and supplement thereto, cited in the solicitation.

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Director, U.S. Army Materials Technology Laboratory, ATTN: SLCMT-MSE, Watertown, MA 02172-0001 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

AMSC N/A /FSC 6810/

SPECIFICATIONS

MILITARY

MIL-P-116 - Preservation; Methods of

STANDARDS

FEDERAL

FED-STD-313 - Material Safety Data Sheets, Preparation and Submission of

MILITARY

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

(Copies of specifications, standards, handbooks, drawings, publications, and other Government documents required by contractors in connection with specific acquisition functions should be obtained from the contracting activity or as directed by the contracting activity.)

2.2 Other publications. The following document(s) form a part of this specification to the extent specified herein. Unless otherwise specified, the issues of the documents which are DOD adopted shall be those listed in the issue of the DODISS specified in the solicitation. Unless otherwise specified, the issues of documents not listed in the DODISS shall be the issue of the nongovernment documents which is current on the date of the solicitation.

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM).

D1193 - Standard Specification for Reagent Water
E146 - Chemical Analysis of Zirconium and Zirconium Alloys, Standard Methods for

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

(Non-Government standards and other publications are normally available from the organizations which prepare or which distribute the documents. These documents also may be available in or through libraries or other informational services.)

U.S. DEPARTMENT OF TRANSPORTATION

49 CFR 100-199 Rules and Regulations for the Transportation of Explosives and Other Dangerous Articles

29 CFR 1910-1200

(Application for copies should be addressed to the Superintendent of Documents, Government Printing Office, Washington, D.C. 20402.)

2.3 Order of precedence. In the event of a conflict between the text of this specification and the references cited herein (except for associated detail specifications, specification sheets or MS standards), the text of this specification shall take precedence. Nothing in this specification, however, shall supersede applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1 Materials covered in this document shall conform to the properties shown in table I.

3.2 Workmanship safety. Zirconium metal powder is a dangerous fire and explosion hazard. The dry and partially dry powder is sensitive to friction, heat, and sparks. It readily accumulates a static electric charge, and is liable to spontaneous combustion. The zirconium powder shall not be stored below the freezing temperature of water.

3.3 Zirconium slurry. Unless otherwise specified, the zirconium shall be thoroughly mixed with water to form a slurry containing not less than 25 percent by weight of water. In order to prevent freezing during shipment or storage in low temperature climates, the zirconium slurry shall contain not less than 25 percent by weight of methylalcohol-water solution or other anti-freeze mixture approved by the contracting officer. The methylalchohol - water solution shall have a freezing point of minus 30°F (-34.4°C) or below.

3.4 Material safety data sheet. A Material Safety Data Sheet will be submitted in accordance with procedures outlined in FED-STD-313 and 29 CFR1910-1200 (see 6.3.2)

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 Inspection provisions

4.2.1 Lot. A lot shall consist of that quantity of zirconium produced by one manufacturer in accordance with the specification of one continuous set of processing conditions with no change in materials. In the event that the process is a batch operation, each batch shall consititute a lot.

4.2.2 Sampling.

4.2.2.1 Examination. Each lot shall be inspected in accordance with the classification of tests. Sampling plans and procedures shall be in accord with MIL-STD-105. Acceptance tests shall be made on a composite sample representative of the lot. The composite sample of 4.2.2.2 shall be subjected to the tests of 4.5.

TABLE I. Chemical, physical and thermal requirements for Type I, Type IIa, Type IIb zirconium.

Requirements	Type I	Type IIa	Type IIb	Test
Total zirconium (Zr) plus hafnium (Hf), percent	95.0 (min)	95.0 (min)	95.0 (min)	4.5.4
Ignition gain, (percent)	30.0 (min) -33.0(max)	30.0 (min) -33.0(max)	30.0 (min) -33.0(max)	4.5.5
Hydrogen content, (percent)	0.25 max	0.25 (max)	0.25 (max)	4.5.6
Geometric median particle size	3.9 - 7.0 um	1.6 - 2.0 um	1.9 - 3.1 um	4.5.7
Surface area	0.9 - 2.0 m^2/g	0.9 - 2.0 m^2/g	0.9 - 2.0 m^2/g	4.5.8
Permeametry particle size	2.3 - 3.5 μm	*1.2 - 2.6 μm	*1.5 - 2.3 μm	4.5.9
Open train burning	3.5 - 8.0 sec/10in	1.3 - 3.5 sec/10in	1.3-3.5 sec/10 in	4.5.10

*POWDER BELOW 1.5 μm IS HAZARDOUS TO WORK WITH

4.2.2.2 Test sample. From all containers comprising the lot, enough material is removed to form a homogeneous composite test sample of approximately two (2) pounds of zirconium slurry when thoroughly mixed. Acceptance tests shall be made on a composite sample representative of the lot. The composite sample shall be placed in a suitable container, sealed and identified. The composite sample of 4.2.2.2 shall be subjected to the tests of 4.5.

4.3 Classification of Tests.

4.3.1 Quality Conformance Tests. Quality conformance tests shall be as specified in table II:

TABLE II.

Test	Requirement paragraph	Test paragraph
Total Zirconium content (including hafnium)	3.1	4.5.4
Ignition gain	3.1	4.5.5
Hydrogen content	3.1	4.5.6
Particle size distribution	3.1	4.5.7

4.4 Preparation for Delivery.

4.4.1 Classification of defects. Categories and defects shall be as specified in table III. Sampling procedure shall be in accordance with MIL-STD-105 using single sampling plan and acceptable quality level (AQL) of 2.5 percent defective.

TABLE III.

	Major Defects	Method of Inspection
101	Container damaged	Visual
102	Container leakage	Visual
103	Container closure incorrect	Visual
104	Container incorrect	Visual
105	Container marking incorrect, missing or illegible	Visual

4.5 Test Methods and Procedures.

4.5.1 Visual examination. Visual examination of samples of zirconium shall be made and found to be uniform in quality. The container must be free from all major defects (see table III). The water should be clear when the powder has settled with no sign of contamination by oils, greases or other impurities that would affect the material in its end use.

4.5.2 Tests. Tests shall be made with distilled water as recommended in ASTM D1193 and analytical reagent grade chemicals. No substitution of specified equipment or methods shall be permitted except with prior approval of the government.

4.5.3 Test preparations

4.5.3.1 Drying Procedure. Thoroughly mix the sample by shaking the zirconium/water slurry. Using a Buchner funnel containing a Whatman No. 42 filter paper, vacuum filter an appropriate size sample to yield about 75g of dried zirconium. Wash the filter cake twice with acetone (500 ml each) and twice with diethyl ether (500 ml each). Suction dry for 1/2 hour.

Cautiously transfer the dried zirconium to a large Petri dish, carefully break up the lumps using a non-conducting probe, and oven-dry at 70°C (158°F) for four hours. Cool to room temperature in a desiccator. Transfer the small amount (8-9g) to a small sample bottle and save for further analysis.

4.5.4 Determination of total zirconium. (Precipitation with Cupferron). A weighed portion of material is dissolved in acid and the zirconium is precipitated as the zirconium-cupferronate and subsequently ignited to zirconium oxide as described in 4.5.4.1.

4.5.4.1 Assay Procedure. Accurately weigh about 0.1 gm zirconium into a clean, dry 400 ml beaker. Rinse down the sides of beaker, and carefully add 25 ml of concentrated sulfuric acid (H_2SO_4). Cover and heat gently on a hot plate until the zirconium has completely dissolved. Cool, and carefully dilute to 150 ml with distilled water. If a silica residue is noticed, add 3-5 drops of hydrofluoric acid (HF), and evaporate again to dense white fumes. Place the diluted sample into an ice bath, and cool to approximately 10°C (50°F). Add 40 ml of 6% Cupferron (60gm of reagent grade Cupferron in 1 liter distilled cold water), stir well, and allow to stand in the ice bath for 15-30 minutes. Filter through a Whatman No. 42 ashless paper filter, using the Cupferron wash solution, (30ml of 6% Cupferron solution diluted to 1 liter with the distilled water) to transfer and wash the precipitate. Air-dry for at least four hours, preferably overnight. Transfer the filter paper and precipitate to a preignited, preweighed platinum crucible. Place crucible in a muffle furnace held at 350-400°C (662-752°F) and allow filter paper to char. If such a muffle is unavailable, the filter paper may be carefully charred by means of a Bunsen or Meeker burner. Do not permit the paper to flame. After the filter paper and precipitate have been charred, place the crucible in a muffle furnace held at 950°C $\pm$ 50° (1742 $\pm$ 122°F) for about one hour. This ignites the precipitate to zirconium oxide (ZrO_2). Ignite to constant weight and weigh as ZrO_2.

% Total Zirconium:

$$\frac{\text{wt } ZrO_2 \times \frac{Zr}{ZrO_2} \times 100}{\text{wt sample}} = \% \text{ Zr}$$

where

$$\frac{Zr}{ZrO_2} = \frac{\text{Mol wgt Zr}}{\text{Mol wgt } ZrO_2} = \frac{91.2}{123.2} = 0.7403$$

then

$$\frac{\text{g } ZrO_2 \times 0.7403}{\text{g sample}} \times 100 = \% \text{ Zr}$$

(The total zirconium content shall be as specified in table I).

4.5.5 Ignition gain. Accurately weigh a 0.2 - 0.4 gm sample of dried Zr powder into a 30 ml Vycor crucible. Place the uncovered crucible in a silica triangle 1 inch above the top of a Meeker burner. The sample should ignite within 20 seconds. If it fails to ignite within this time, repeat the procedure with a fresh sample. Continue heating over the burner for 5 minutes after ignition. Transfer the crucible and contents to a muffle furnace heated to 980° ± 10°C (1796° ± 18°F). Heat in the muffle furnace for 2 hours. Cool in a desiccator and weigh to constant weight. Calculate the gain in weight as percent ignition gain:

$$\% \text{ ignition gain} = \frac{\text{Gain in weight}}{\text{wt sample}} \times 100$$

$$\% \text{ Free Zirconium} = \frac{\% \text{ ignition gain}}{\text{theoretical \% ignition gain}} \times 100$$

$$= \frac{\% \text{ ignition gain}}{35.08} \times 100$$

$$\text{where } 35.08 = \frac{\text{Mol wgt } ZrO_2 - \text{Mol wgt Zr}}{\text{Mol wgt Zr}} \times 100$$

(The gain in weight on ignition shall be as specified in table I.)

4.5.6 Geometric median hydrogen content. Determine the hydrogen content by the vacuum fusion method or Perkin-Elmer Carbon, Hydrogen, Nitrogen Analyzer. Hydrogen can be determined by the procedure recommended in ASTM E 146.

4.5.7 Geometric median particle size distribution. The particle size distribution will be determined by the X-ray SediGraph (see 6.4). This is a particle size analyzer based upon Stokes' law of sedimentation. The instrument measures the sedimentation rates of particles in suspension and automatically presents these data as a cumulative percent distribution in

terms of the Stokesian or equivalent spherical diameter. By means of a finely collimated beam of X-rays, the concentration of particles remaining in suspension at various sedimentation depths are determined as a function of time. An accepted standard material of known particle size distribution shall be used for calibration of the X-ray sedigraph before use.

4.5.7.1 SediGraph operation. The as-received powder slurry shall be mixed for at least 15 minutes in its original container with a high speed mixer using a plastic coated stirrer. Further mixing is done with a magnetic stirrer until all the powder becomes dispersed in the water. For Type I powders, a 0.1 wt percent Na-pyrophosphate in water solution shall be used with 1- drop of Photo-Flo (see 6.4). For Type II powders, de-ionized water shall be used with 1- drop Photo-Flo. Using a small syringe, a sample is obtained, while being mixed, by moving the syringe around in the slurry and extracting small amounts from different parts of the container. The sample is then placed into 25 ml of the proper solution, (the pump should be on), for the type powder to be analyzed until the pen reads over 100% on the chart paper. The beaker is placed in an ultrasonic vibrator for 1 min. The sample is quickly returned to the SediGraph, while the magnetic stirrer in the SediGraph is used to keep the particles from settling out. The 100% dial is set and then the program is run. Each sample is run at least twice to check for reproducibility. All runs are started at 35 μm.

Re-plot the data at 10, 20, 30, 40, 50, 60, 70, 80 and 90 cumulative percent on log-probability paper (ex. K & E 46 8080) with three log cycle. The cumulative mass at 50% shall be recorded as the median particle size. Visually compare the log-probability plot of the data to the overlay transparency of acceptable powders. Figure 1. (Overlay attached to specification).

(The particle size distribution shall be as specified in figure 1).

(The median particle size shall be as specified in table I).

4.5.8 Determination of Surface Area (B.E.T. Method - Brunauer S., Emmett P.H., Teller E., Method). A technique using B.E.T. theory shall be used. This measurement may be carried out by a single point or multiple point determination using a static or dynamic (flowing gas) technique. A surface area measurement requires that the solid in question, zirconium particles in this case, be freed of gases and vapors that are acquired from exposure to the atmosphere. This can be done by using either a vacuum or gas-purging technique.

The re-exposure of the prepared solid, to an adsorbate gas while at the temperature of liquid nitrogen, permits the determination of the quantity of gas required for monolayer coverage. The area of the solid on a molecular scale is calculated using the cross-sectional area of adsorbed nitrogen gas molecules. The key consideration is that the chosen method be periodically calibrated to accepted surface area standards. The standard to be used is Duke Scientific Corporation Standard Alumina #355 (see 6.4). The equipment shall be calibrated once a month.

(The surface area shall be specified as in table I).

4.5.9 Permeametry. The term permeametry particle size shall be used instead of median particle size to distinguish data from particle size distribution.

Using the Fisher Sub-Sieve Sizer (FSSS) Permeametry instrument, the procedure is fairly simple. A sample of dry powder (6.50 ± 0.01 gm), dried in vacuum oven at 105° (221°F) for 1 hour, is weighed and transferred into a sample tube while tapping the sides of the tube to settle the powder. The powder sample is then compressed in a manner that the permeametry particle size can be read directly from the calculator chart. This is done by setting the pointer at the liquid level of the manometer. The permeametry particle size is determined by a number of measurements on the sample while it is progressively compressed until the particle size value measured is no longer a function of sample porosity. This value is recorded as the permeametry particle size. The calibration procedure described in the operation manual shall be employed prior to each set of measurements.

It has been empirically established that the median particle sizes below 1.5 microns are extremely susceptable to spontaneous combustion. The technique described is exceptionally useful in monitoring the particle size under safe conditions.

4.5.10 Burning Time. A steel bar mold, one inch wide, 5/8 in. thick and 30 inches long, shall be machined to produce a groove 1/8 in. wide by 1/16 in. deep in the center of one of the 1 in. flat sides for the full length of the bar. A burning base shall be constructed of a 36 inches by 4 inches by 1/4 inch piece of transite board (see 6.3.1) or similar insulating material having scribed marks 8 inches from each end. This will provide a 20 in. long time-distance path.

Very slowly and from the lowest height practicable, pour the dry zirconium powder into the groove, completely filling it. The zirconium shall be leveled with the top of the groove by scraping off excess material with the edge of a plastic spatula. It is not to be packed. The transite board shall be placed on top of the bar mold so that the powder extends approximately 5 inches beyond each mark toward each end. The board and mold shall be held together inverted, placed on the bench and then the bottom of the bar mold shall be tapped with the spatula to loosen sample particles. The bar mold shall then be removed. Uniformity and continuity of powder train must be verified. The zirconium shall be ignited at one end. With a stop watch, start timing when the train burns to the first mark and stop when the burning reaches the next mark. The time for 20 inches of burning shall be recorded and divided by 2. The test shall be run 4 times for each sample. The first test is only used to warm the board; the successive three test times are to be averaged. The transite board must be kept in a warm, dry place when not in use.

4.5.10.1 Alternative Methods. The following are alternative methods of measuring the burn rate of the zirconium powder train. These techniques eliminate the necessity of direct visual observation of the burning powder to determine the elapsed time between length markers.

a. The use of a system comprised of two electronic elapse timers connected to a series of photo sensors coupled to fiber optic cables and positioned at the 0, 10" and 20" length markers of the powder train. The burning zirconium powder will automatically trigger off the timers at the selected markers and provide elapsed time measurements for 0-10" and 0-20" lengths.

b. The use of a high speed recording system utilizing instant developing color movie film. A Mekel 300 Instant Analysis Camera is specifically designed to accept Polaroid Polavision Phototape Cassettes and has precise operating speeds that can be varied in single frame increments from 4 to 300 frames per seconds. The exposed film is processed in a Polaroid Polavision Analyzer automatically in ninety seconds and can be viewed in the analyzer immediately after processing. Four discrete slow motion pulse rates are available including 2, 4, 6, and 9 frames per second as well as manual single frames per second. The manual mode allows an accurate count of the number of frames it takes the burning train to advance from marker to marker. These features are especially useful for visual measurement of the burning rate of the zirconium powder train as well as a study of the burning characteristics of the material without direct exposure to the burning powder.

4.5.11 Inspection of packaging. Except when industrial packaging is specified, the sampling and inspection of the preservation and interior package marking shall be in accordance with groups A and B quality conformance inspection requirements of MIL-P-116. The sampling and inspection of the packing and marking for shipment and storage shall be in accordance with the quality assurance provisions of the applicable container specification shown in section 5 and the marking requirements of MIL-STD-129. The inspection of industrial packaging shall be as specified in the contract.

5. PACKAGING

5.1 Preservation.

5.1.1 Unit Packs. One five gallon glass or non-carbon polyethylene container, with a zirconium slurry having a net weight of not nore than 10 pounds comprises one unit.

5.2 Packing.

5.2.1 Level A. The zirconium slurry shall be packed in a DOT specification container 6D cylindrical steel drum with DOT 2S polyethylene insert. The net weight of the contents may not exceed 50 lbs dry material. Packing shall conform to the regulations listed in 49 CFR Section 173.214C(1).

5.3 Marking. All shipping containers shall be marked in accordance with MIL-STD-129 and shall include as a minimum the following:

Manufacturer's Name
Material-Zirconium Metal Powder (minimum 25% water)
DANGER - Flammable Solid
Suppliers Batch or Lot Number
Net Weight of Zirconium

In addition to any special marking required by the contract order, all markings shall be in accordance with the Code of Federal Regulations 49 CFR 100-199.

6. NOTES

6.1 Intended Use. Powdered zirconium metal covered by this specification is intended for use in the manufacture of pyrotechnic heat powder for thermal battery applications.

6.2 Ordering Data. Procurement documents should specify the following:

a. Title, number and date of this specification
b. Specify Type required (see 1.2)

6.3 Warning. All operations involving handling and use of zirconium are extremely dangerous. The dry powder is sensitive to friction, readily accumulates a static electric charge and is liable to spontaneous combustion. Face shield and gloves should be worn during operations. Water suspensions of zirconium on long storage form a hard cake. Special precautions are needed to repulverize or resuspend the solid zirconium. Under no circumstances, should manual pry bars be used to loosen or break up the cake. It is recommended that special instructions be obtained from a safety officer of the manufacturer for handling or disposing of badly caked zirconium.

Zirconium stored under water which has frozen, is also particularly hazardous to handle or disturb. Every effort should be made to prevent freeezing during shipment and storage, by the use of proper anti-freeze additions to the water as recommended by the manufacturer.

6.3.1 Transite. Transite is described as an asbestos product trade marked formerly by Manville, Inc. A non-asbestos insulating board should be considered for use in this particular operation since a hazard is created when cutting the board to size.

6.3.2 Material Safety Data Sheet (MSDS). Contracting officers will identify those activities requiring copies of completed Material Safety Data Sheets prepared in accordance with FED-STD-313. The pertinent government mailing addresses for submission of data are listed in appendix B of FED-STD-313.

6.4 Reference Data. This specification requires the use of specific materials and equipment which are noted in the text.

(a) Sedigraph Micromeritics - 5000 D Particle Size Analyzer.
(b) Eastman Kodak - Photo-Flo
(c) Duke Scientific Corp. - Standard Alumina #355

6.5 Subject term (key word) listing.

1. Particle size
2. Part-number system
3. Zirconium metal
4. Zirconium powder
5. Zirconium slurry
6. Military specification

Custodians:

Army -- MR
Navy -- AS
Air Force - 99

Review activity:

Army -- AR, SM, MI
Air Force -- 15, 79

DLA -- GS

User activity:

Navy -- MC
Air Force -- 79

Preparing activity:

Army -- MR

Project No. 6810-B381

(KBWP# ID-0723A/DISK 0102A. FOR MTL USE ONLY)

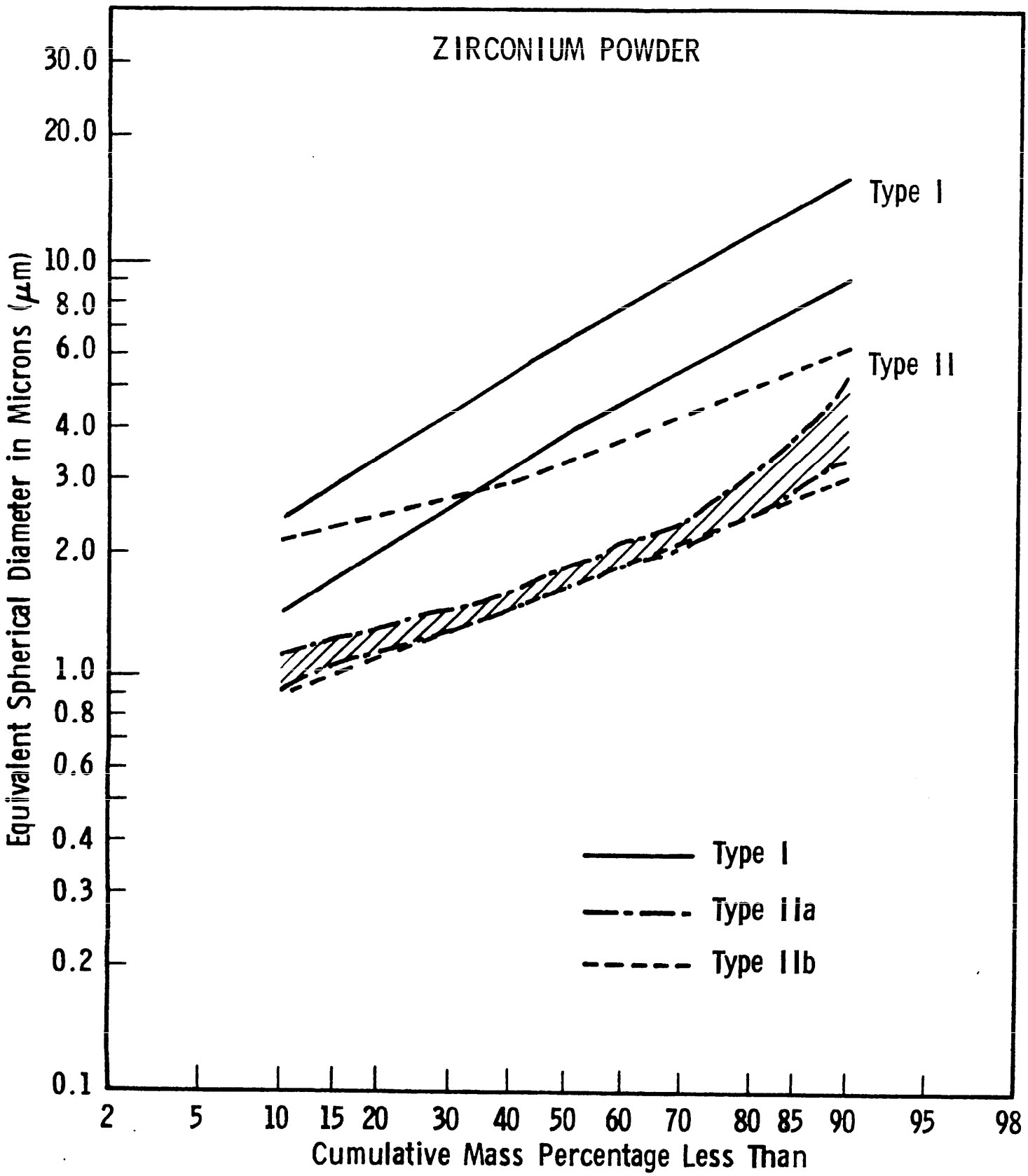


Figure 1. LOG PROBABILITY PARTICLE SIZE DISTRIBUTION RANGES

STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL

(See Instructions – Reverse Side)

1. DOCUMENT NUMBER	2. DOCUMENT TITLE
MIL-Z-46189	ZIRCONIUM USED IN GASLESS MIXTURES FSC 6810-B381

3a. NAME OF SUBMITTING ORGANIZATION	4. TYPE OF ORGANIZATION *(Mark one)*
	☐ VENDOR
b. ADDRESS *(Street, City, State, ZIP Code)*	☐ USER
	☐ MANUFACTURER
	☐ OTHER *(Specify)*: _____

5. PROBLEM AREAS

a. Paragraph Number and Wording:

b. Recommended Wording:

c. Reason/Rationale for Recommendation:

6. REMARKS

7a. NAME OF SUBMITTER *(Last, First, MI)* – Optional	b. WORK TELEPHONE NUMBER *(Include Area Code)* – Optional
c. MAILING ADDRESS *(Street, City, State, ZIP Code)* – Optional	8. DATE OF SUBMISSION *(YYMMDD)*

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NOTE: This form may not be used to request copies of documents, nor to request waivers, deviations, or clarification of specification requirements on current contracts. Comments submitted on this form do not constitute or imply authorization to waive any portion of the referenced document*(s)* or to amend contractual requirements.

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