MIL-Z-11410B
12 February 1968
SUPERSEDING
MIL-Z-11410A
19 August 1952

MILITARY SPECIFICATION

ZIRCONIUM-NICKEL ALLOY, POWDERED

1. SCOPE AND CLASSIFICATION

1.1 Scope. This specification covers powdered zirconium-nickel alloy for use in delay compositions.

1.2 Classification. Powdered zirconium-nickel alloy shall be of the following types, as specified (see 6.1):

Type I - 70/30 zirconium-nickel alloy
Type II - 30/70 zirconium-nickel alloy

2. APPLICABLE 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:

SPECIFICATIONS

FEDERAL

RR-S-366 - Sieves, Standard, Testing

MILITARY

MIL-A-2550 - Ammunition and Special Weapons, General Specification for

STANDARDS

MILITARY

MIL-STD-129 - Marking for Shipment and Storage
MIL-STD-1233 - Procedures for Determining Particle Size, Particle Size Distribution, and Packed Density of Powder Material

(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 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.

INTERSTATE COMMERCE COMMISSION

49 CFR 71-78 - Rules and Regulations of the Transportation of Explosives and Other Dangerous Articles

(Application for copies should be addressed to the Superintendent of Documents, Government Printing Office, Washington, D. C. 20402. Orders should cite "49 CFR 71-78").

3. REQUIREMENTS

3.1 Composition. Powdered zirconium-nickel alloy of the specified type (see 6.1), shall conform to the corresponding chemical composition specified in table I.

Table I. Chemical composition

Constituent	Type I	Type II
Calcium, percent, maximum	0.15	0.15
Iron, percent, maximum	0.20	0.20
Aluminum, percent, maximum	0.15	0.15
Sulfur, percent, maximum	0.01	0.01
Total zirconium and nickel, percent, minimum	96.0	96.0
Zirconium, percent	70.0 ± 4.0	30.0 ± 4.0
Nickel, percent	30.0 ± 4.0	70.0 ± 4.0
Moisture, percent, maximum	0.20	0.20

3.2 Granulation. Not less than 99.5 percent of the powdered alloy shall pass through a No. 200 and not less than 97.0 percent through a No. 325 sieve when tested as specified in 4.4.9.

3.3 Average particle diameter. The average diameter of the powdered zirconium-nickel alloy particles shall be 5.0 ± 3.0 microns, when determined as specified in 4.4.10.

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, the supplier 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 that supplies and services conform to prescribed requirements.

4.1.1 Contractor quality assurance system. The contractor shall provide and maintain an adequate quality assurance system, acceptable to the Government covering the supplies under the contract. A current written description of the system shall be submitted to the contracting officer prior to initiation of production. The written description will be considered acceptable when, as a minimum, it provides the quality assurance required by this specification. The contractor shall notify the Government of an obtain approval for any change to the written procedure that might affect the degree of assurance required by this specification or other applicable documents referenced herein.

4.2 Lot. A lot shall be defined as that quantity of material which has been subjected to some unit chemical or physical mixing process intended to make the final product substantially uniform and shall consist of 100 pounds minimum unless otherwise specified by the contracting agency.

4.3 Sampling. The inspector shall take three separate 1 pound samples from the lot. If the material was produced in batches, each sample shall be taken from a different batch, possible. If the material was produced in one continuous run, the three samples shall be taken as follows: One from the first part; one from the middle part; and one from the last part of the run. Each sample shall be placed in a clean, dry, metal or glass container which shall be sealed and carefully marked.

4.4 Test methods.

4.4.1 General. Analytical reagent grade chemicals and distilled water shall be used throughout the tests. Blank determinations shall be run in parallel with the tests, using the same quantities of reagents used in the test, and corrections shall be applied when necessary.

4.4.2 Determination of nickel content. Transfer a 1 gm. sample, weighed to 0.1 mg., to a 250 ml. beaker. Add 50 ml. of hydrochloric acid and 25 ml. of nitric acid. Cover with a watch glass and cautiously add 25 ml. of sulfuric acid. Boil gently until fumes of sulfuric acid appear and continue fuming until the solution becomes clear and light yellow in color. Cool to room temperature and add about 100 ml. of water. Heat on the hot plate to dissolve the salts. Wash into a 250 ml. volumetric flask, cool and dilute to the mark with water. Pipet a 25 ml. aliquot into a 400 ml. beaker, keeping the remainder of the solution for the zirconium determination (see 4.4.3). Dilute to about 250 ml. and add 10 gm. of tartaric acid and 20 ml. of 10 percent ammonium chloride solution. Add ammonium hydroxide until the solution is slightly alkaline. Heat nearly to boiling and add, with stirring, 25 ml. of 1 percent alcoholic solution of dimethylglyoxime for type I alloy or 60 ml. for type II alloy. Add ammonium hydroxide until the solution has a distinct ammoniacal odor. Heat on steam bath for 1 hour and then filter through a tared sintered glass crucible of medium porosity. Transfer and wash with water. Dry for 2 hours at 100° to 120°C, cool in a desiccator, and weigh. Calculate the percent nickel as follows:

$$\text{Percent nickel} = \frac{20.32A}{W}$$

where: A = weight of precipitate, gm.
W = weight of sample in the aliquot, gm.

4.4.3 Determination of zirconium content.

4.4.3.1 Reagents.

4.4.3.1.1 Cupferron solution (6 percent). Dissolve 6 gm. of cupferron in 100 ml. of cold water and filter.

4.4.3.2 Procedure. Pipet a 50 ml. aliquot from the solution kept from the nickel determination (see 4.4.2) into a 400 ml. beaker. Dilute to about 200 ml., cool to 10° ± 2°C in an ice bath, and add with stirring an excess of 6 percent cupferron solution. Filter through a medium texture filter paper, and transfer and wash with cold (1 to 10) hydrochloric acid. Transfer the precipitate and filter paper to a tared platinum dish and dry for 2 hours at 110° to 120°C. Heat at 300° to 400°C until complete charring of the paper has occurred and the rush of gases from decomposition of the organic matter has ceased. Increase the temperature to 900° ± 50°C and continue the ignition at this temperature for 2 hours. Cool in a desiccator and weigh. Calculate the percent zirconium as follows:

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

where: A = weight of precipitate, gm.
W = weight of sample in the aliquot portion, gm.

4.4.4 Determination of total zirconium and nickel content. The total percentage of zirconium and nickel shall be determined by adding the percentage values obtained for zirconium (see 4.4.3) and for nickel (see 4.4.2) determined separately.

resistant, variety DW, grade VISC of FF-B-650. Level A unit packs shall be

4.4.5 Determination of calcium content. Transfer a 5 gm. sample to a 400 ml. beaker. Add 150 ml. of 1 to 1 hydrochloric acid solution and boil gently for 30 minutes. Add 50 ml. of water, cool in an ice bath and filter through a fine texture filter paper, collecting the filtrate in a 400 ml. beaker. Wash with 2 percent hydrochloric acid solution and discard the precipitate. Evaporate the filtrate to about 25 ml. to remove most of the acid, and dilute to 100 ml. Heat nearly to boiling and add ammonium hydroxide until the solution is alkaline. Boil for 1 minute, allow to settle for 15 minutes, and filter through a Buchner funnel containing a fine texture filter paper. Wash with 2 percent ammonium chloride solution and discard the precipitate. Transfer the filtrate to a 400 ml. beaker, add 2 ml. ammonium hydroxide, and saturate with hydrogen sulfide. Add 2 ml. more ammonium hydroxide and dilute to about 200 ml. with recently boiled and cooled water. Cover with a watch glass and let stand for several hours, preferably overnight. Filter through a fine texture filter paper, and wash with 2 percent ammonium chloride solution containing a little ammonium sulfide. Test a small portion of the filtrate with dimethylglyoxime to make certain that all the nickel has been removed. If the separation of the nickel is incomplete, treat with hydrogen sulfide again as described above and filter again. Discard the precipitate. Boil off the hydrogen sulfide from the filtrate and then add enough bromine to color the solution red. Heat to boiling, and continue boiling until the bromine has been expelled and the solution is colorless. Test the solution with litmus paper and render slightly acid, if necessary, with hydrochloric acid. Add 1 gm. of ammonium oxalate, bring to boil, and make the solution distinctly alkaline with ammonium hydroxide. Boil 3 minutes, cool, add 5 ml. of ammonium hydroxide and allow to stand for 2 hours. Filter through a fine texture filter paper, and wash with 2 percent ammonium hydroxide solution. Dissolve the precipitate into the original beaker with 100 ml. of hot 20 percent sulfuric acid and then wash with 100 ml. of hot water. Heat to 80° to 90°C and titrate to a faint pink with 0.1N potassium permanganate solution. Calculate the percent calcium and correct for the blank titration as follows:

$$\text{Percent calcium} = \frac{2.00\ (V - v)N}{W}$$

where: V = ml. of potassium permanganate solution used in titration of the sample.
v = ml. of potassium permanganate solution used in the blank titration.
N = normality of the potassium permanganate solution.
W = weight of sample, gm.

4.4.6 Determination of iron content.

4.4.6.1 Reagents.

4.4.6.1.1 Standard iron solution (1 ml. = 0.025 mg. Fe). Dissolve 0.1756 gm. of $Fe(NH_4)_2(SO_4)_2 \cdot 6H_2O$ in 10 ml. of nitric acid (1 to 1). Boil for 1 minute, cool, and dilute to 1 liter in a volumetric flask.

4.4.6.1.2 Dilute hydrogen peroxide. Dilute 1 ml. of 30 percent hydrogen peroxide to 20 ml. with water. Prepare fresh as needed.

4.4.6.2 Preparation of calibration curve. Transfer 3.0, 6.0, 9.0, 12.0, and 15.0 ml. portions of standard iron solution to 100 ml. volumetric flasks. Add 2 ml. of sulfuric acid (1 to 1) and 2 ml. of nitric acid. Dilute to about 50 ml. with water and add 1 ml. of dilute hydrogen peroxide and 20 ml. of 10 percent potassium thiocyanate solution. Dilute to the mark with water. Measure the absorbance within 30 minutes at 470 millimicrons with a spectrophotometer that has been set at 100 percent transmittance with the reagent blank. Plot milligrams of iron against absorbance.

4.4.6.3 Procedure. Transfer a 1 gram sample to a large platinum dish. Add 5 ml. of hydrofluoric acid and 5 ml. of nitric acid and warm to dissolve. Add 10 ml. of sulfuric acid and evaporate to fumes of sulfuric acid. Allow to cool and add about 60 ml. of water. Heat on a hot plate until the salts are in solution. Cool, wash into a 100 ml. volumetric flask, and dilute to the mark with water.

Pipet a 10 ml. aliquot into a 100 ml. volumetric flask, and dilute to about 50 ml. Add 2 ml. of nitric acid, 1 ml. of dilute hydrogen peroxide, and 20 ml. of 10 percent potassium thiocyanate solution. Dilute to the mark with water and measure the absorbance within 30 minutes at 470 millimicrons with a spectrophotometer that has been set to 100 percent transmittance with the reagent blank. Determine the milligrams of iron by consulting the calibration curve. Calculate the percent iron and correct for the slight interference from the green nickel color as follows:

$$\text{Percent iron} = \frac{A}{W \times 10} - 0.0001\ P$$

where: A = mg. of iron as read from calibration curve.
W = weight of sample in aliquot, gm.
P = percent nickel in the sample.

4.4.7 Determination of aluminum content. Transfer a 2 gm. sample to a large platinum dish and add 7 ml. of hydrofluoric acid. Add 7 ml. of nitric acid in small portions. Warm to dissolve. Add 10 ml. of sulfuric acid and evaporate to fumes of sulfuric acid. Allow to cool. Add 10 ml. of water and 10 ml. of hydrochloric acid and heat on the hot plate until the salts are in solution. Wash into a 400 ml. beaker, dilute to about 150 ml. and heat to boiling. Slowly pour the hot solution into 250 ml. of 10 percent sodium hydroxide solution and vigorously stir the resulting solution. Filter through a double thickness of fine texture filter paper container into a Buchner funnel. If the filtrate is not clear, repeat the filtration, using the same paper. Wash with 1 percent sodium hydroxide solution. Discard the precipitate. Make the filtrate acid to litmus paper with hydrochloric acid and then add 5 ml. excess hydrochloric acid. Boil to about 150 ml. Make the solution alkaline to methyl red with ammonium hydroxide, heat to boiling, and boil for a minute. Add some paper pulp, filter through a medium texture filter paper, and wash with 2 percent ammonium chloride solution. Dissolve the precipitate back into the original beaker with hot 1 to 1 hydrochloric acid and wash with hot water. Dilute to about 150 ml., reprecipitate the aluminum, and filter as before. Transfer the precipitate and filter paper to a tared platinum crucible and ignite to dull red heat until the carbon is burned off. Add 1 drop of sulfuric acid and 1 ml. of hydrofluoric acid, and evaporate to dryness. Ignite over a glast burner for 30 minutes, cool in a desiccator, and weigh. Correct the weight of the ignited residue by making a blank determination on the reagents. Calculate the percent aluminum as follows:

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

where: A = weight of test residue, gm.
B = weight of residue of blank, gm.
W = weight of sample, gm.

4.4.8 Determination of sulfur content.

4.4.8.1 Reagents.

4.4.8.1.1 Cadmium chloride solution. Dissolve 10 gm. of $CdCl_2$ in 400 ml. of water and add 600 ml. of ammonium hydroxide.

4.4.8.1.2 Starch solution. Add a cold suspension of 5 gm. of soluble starch in 25 ml. of water to 500 ml. of boiling water, cool, add 50 ml. of cold 10 percent sodium hydroxide solution and 15 gm. of potassium iodide and mix thoroughly.

4.4.8.1.3 Standard potassium iodate solution (0.03N). Dry reagent grade KIO_3 at 180°C for 2 hours, cool, and dissolve 1.0701 gm. in about 20 ml. of water containing 1 gm. of sodium hydroxide and 10 gm. of potassium iodide. When solution is complete, dilute to 1 liter in a volumetric flask. This is a primary standard.

4.4.8.2 Procedure. Transfer a 5 gm. sample to a dry, 300 ml. Florence flask furnished with a thistle tube for the addition of acid and an outlet tube for collecting evolved gases. Connect the apparatus so that the evolved gas will be trapped in a beaker containing 15 ml. of cadmium chloride solution and 200 ml. of distilled water. Add 100 ml. of 1 to 1 hydrochloric acid solution to the flask through the thistle tube. Heat the flask and its contents so that there is a rapid, steady evolution of gas. After the reaction is complete, boil 30 seconds, disconnect the delivery tube, and remove the beaker. Add 2 ml. of starch solution, 40 ml. of 1 to 1 hydrochloric acid, and titrate immediately with 0.03N potassium iodate to a permanent color. Calculate the percent sulfur as follows:

$$\text{Percent sulfur} = \frac{AB \times 0.016 \times 100}{W}$$

where: A = ml. of potassium iodate solution required for titration of sample.
B = normality of potassium iodate solution.
W = weight of sample, gm.

4.4.9 Determination of granulation. Make up a slurry of water and 10 gm. of the sample. Superimpose a tared U. S. standard No. 200 sieve on a tared No. 325 sieve; the sieves shall comply with the requirements of RR-S-366, except that the frames shall have a nominal diameter of approximately 3 inches. Pour the slurry on the top sieve and wash the alloy through each sieve thoroughly with a water spray from a spray nozzle under tap water pressure. A solution of a wetting agent may be added to the weighed sample or to the wash water. Dry each sieve together with the alloy retained thereon in an oven at 105°C until constant weight has been obtained and then cool to room temperature. Weigh the sieves together with the alloy and calculate the percentage of the alloy passing through each sieve.

4.4.10 Average particle diameter. The average particle diameter shall be determined in accordance with MIL-STD-1233, method 100. The density of type I alloy is 7.20 gm/cc and the density of type II alloy is 8.10 gm/cc.

4.4.11 Determination of moisture. Transfer a 2 gm. sample to a tared weighing dish and heat for 1 hour at 105° ± 5°C. Cool in a desiccator and weigh. Calculate the percent moisture as follows:

$$\text{Percent moisture} = \frac{100A}{W}$$

where: A = loss in weight, gm.
W = weight of sample, gm.

4.5 Resubmission and retest. If the composite sample, or any primary sample subjected to tests, fails to pass the tests, the lot shall be rejected. The contractor shall have the option of having a partial or complete analysis made on samples taken from any or all of the containers in the lot at no expense to the Government. The contractor may then remove the defective portion of the lot, and resubmit the lot for acceptance provided complete replacement of the defective portion can be made to the satisfaction of the inspector. The resubmitted lot shall be accepted provided the new samples selected in accordance with 4.3 pass all the required tests.

5. PREPARATION FOR DELIVERY

5.1 Packaging and packing. Unless otherwise specified, powdered zirconium-nickel alloy shall be packed in heat sealed inert plastic liners, such a polyethylene, contained in new, clean, No. 28 gauge minimum metal containers of approximately 1 gallon capacity (similar to commercial paint pails), or in new, clean, plain steel drums conforming to specifications 6A, 6B, 6C, 17C, 37D, 37E, or 37F of Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles, etc. Each drum or pail shall have a removable head, and shall be hermetically sealed when the cover is secured in place. The gross weight of the packed metal container shall not exceed 110 pounds. When shipped in containers of less than 5 gallon capacity, the containers shall be packed in nailed wooden boxes conforming to specification 15A of Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles, etc. When more than 1 container is shipped in a box, the containers shall be separated, and braced to prevent contact with each other and their movement within the box.

5.2 Marking. Each drum or pail shall be clearly and indelibly marked with the lot number of the alloy powder therein. In addition to any special markings specified by the contract or order, shipments shall be marked in accordance with Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles, without regard to destination or quantity shipped, and with MIL-STD-129.

6. NOTES

6.1 Ordering data. The procurement documents should specify the following:

(1) Title, number, and date of this specification.
(2) Type of powdered alloy required (see 1.2).
(3) Size and type of container required (see 5.1)

6.2 Dichromating. Agitate the zirconium-nickel alloy for approximately 1/2 hour in a hot (90° to 100°C) solution of technical grade potassium dichromate in the ratio of 1 pound technical grade potassium dichromate and 10 pounds water to 4 pounds zirconium-nickel alloy. For each pound potassium dichromate used, add 0.02 pound technical grade potassium acid sulphate to the solution. Filter the alloy slurry on a vacuum pan and wash the alloy once with a volume of clear water equal to the volume of the dichromating solution. Dry the alloy in suitable equipment. If the contractor desires to deviate from this process, he shall submit in writing a description of the process to be used to the contracting agency and receive written approval prior to its use.

Custodian:
Army - MU
Air Force - 84

Review activities:
Army - MR, MD

User activities:
Army - MI, EL

Preparing activity:
Army - MU

Project No. 9630-0012

FOLD

Department of the Army

Frankford Arsenal
Philadelphia, Pa. 19137

OFFICIAL BUSINESS

POSTAGE AND FEES PAID
Department of the Army

Commanding Officer
Frankford Arsenal
ATTN: SMUFA-N1100
Philadelphia, Pa. 19137

FOLD

SPECIFICATION ANALYSIS SHEET

Form Approved
Budget Bureau No. 22-R285

INSTRUCTIONS: 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.

SPECIFICATION

ORGANIZATION

CITY AND STATE | CONTRACT NUMBER

MATERIAL PROCURED UNDER A

[ ] DIRECT GOVERNMENT CONTRACT [ ] 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?

[ ] YES [ ] 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

DD FORM 1 JAN 66 1426 REPLACES EDITION OF 1 OCT 64 WHICH MAY BE USED.