MIL-Z-399D
1 October 1971
SUPERSEDING
MIL-Z-399C
19 February 1969

MILITARY SPECIFICATION

ZIRCONIUM (GRANULAR AND POWDERED)

This specification is mandatory for use by all Departments and Agencies of the Department of Defense

1. SCOPE

1.1 Scope. This specification covers two types of zirconium for use in the manufacture of ordnance materials.

1.2 Classification. The zirconium shall be of two types as follows: Each type shall have 3 classes as indicated in tables I and II.

Type I - Granular
Type II - Powdered

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.

SPECIFICATION

FEDERAL

RR-S-366 - Sieve, Test

FSC 6810

STANDARDS

MILITARY

MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes
MIL-STD-129 - Marking for Shipment and Storage.
MIL-STD-1233 - Procedures for Determining Particle Size, Particle Size Distribution of Packed Density of Powdered Materials

(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

D 1193 - Reagent Water

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

(Technical society and technical association specifications and standards are generally available for reference from libraries. They are also distributed among technical groups and using Federal agencies.)

DEPARTMENT OF TRANSPORTATION

49-CFR Transportation Parts 1-199

(These regulations may be purchased from the Superintendent of Documents, Government Printing Office, Washington, D. C. 20402.)

3. REQUIREMENTS

3.1 Particle size requirements. Type I zirconium shall conform to the particle size requirements specified in table I, and type II zirconium shall conform to the particle size requirements specified in table II. The tests for these requirements shall be as specified in 4.3.3.

Table I. Particle size requirements for type I zirconium

Sieve	Class 1	Class 2	Class 3
Thru No. 20 sieve (840 microns), percent by weight	99 min.		
Thru No. 60 sieve (250 microns), percent by weight	5 max.		
Thru No. 80 sieve (177 microns), percent by weight		100	
Thru No. 100 sieve (149 microns), percent by weight		98 min.	
Thru No. 170 sieve (88 microns), percent by weight			100
Thru No. 200 sieve (74 microns), percent by weight		50 max.	98 min.
Thru No. 325 sieve (44 microns), percent by weight		25 max.	
Subsieve			
Less than 20 microns, percent by weight			10.0 max.
Less than 10 microns, percent by weight		2.0 max.	

Table II. Particle size requirements for type II zirconium

Sieve	Class 1	Class 2	Class 3
Thru No. 120 sieve (125 microns), percent by weight	100	100	99.5 min.
Thru No. 200 sieve (74 microns), percent by weight	99 min.	99 min.	
Thru Buckbee Mears No. 26 sieve, (20 microns), percent by weight	96 min.	96 min.	
Subsieve			
Less then 9 microns, percent by weight	85 min.	85 min.	
3 microns, percent by weight	70-90	70-90	
0.75 microns, percent by weight	12-30	12-30	
Average particle size, microns	2.5±1.0	2.0 ± 0.3	3.0 ± 1.0

3.2 Chemical requirements. The zirconium shall conform to the chemical requirements as specified in table III.

Table III. Chemical requirements

Requirement	Type I	Type II			Test par.
		Class 1	Class 2	Class 3	
Total zirconium, percent min.	96.0	94.0	95.0	95.0	4.3.4
Calcium, percent, max.	0.05	0.10	0.05	0.05	4.3.5 (see 6.6)
Iron, percent, max.	0.30	0.20	0.03	0.05	4.3.6 (see 6.6)
Aluminum, percent, max.	0.10	0.30	0.15	0.10	4.3.7 (see 6.6)
Hydrogen, percent, max.	0.20	0.20	0.20	—	4.3.8 (see 6.8)
Chloride (as Cl), percent, max.	0.03				4.3.9
Silicon, percent, max.	0.10				4.3.10 (see 6.6)
Tin, percent, max.	0.75				4.3.11 (see 6.6)
Ignition gain, percent		30.2-33.0	30.2-33.0	30.2-33.0	4.3.12

3.3 Burning time. Type II zirconium, classes 1 and 2, shall have a burning time between 1.3 and 2.4 seconds (per 10 inches) when determined as specified in 4.3.13.

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.

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 and obtain approval for any changes to the written procedure that might affect the degree of assurance required by this specification or other applicable documents referenced herein.

4.2 Inspection provisions.

4.2.1 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 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 MIL-STD-105. 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 Containers for domestic shipment and limited storage, prior to closing.

Categories	Defects	Method of inspection	Code No.
Critical:			
	1. Zirconium insufficiently wet (see 5.1)	Visual	01001
Major:	None defined		
Minor:	None defined		

4.2.2.2 Sealed container.

Categories	Defects	Method of inspection	Code No.
Critical:	None defined		
Major:			
	201. Container leaking	Visual	02001
Minor:			
	202. Marking misleading or unidentifiable	Visual	02002

4.2.3 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. A random sample of filled containers shall be selected from the lot in accordance with MIL-STD-105. However, when a lot comprises less than 10 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 eight (8) ounces. (These portions shall then be thoroughly mixed to form a composite sample which shall be used for the chemical composition and granular tests. The composite sample shall be placed in a suitable container, sealed and identified.)

4.3 Test procedures.

4.3.1 Tests. Except as specified herein, reagent grade chemicals and distilled water in accordance with ASTM D 1193 shall be used throughout the tests.

4.3.2 Sample preparation. Shake the sample so that the solids are dispersed as uniformly as possible in the liquid in which the material is shipped. Quickly pour off an amount of the suspension that will contain enough of solids for all the tests to be run (approximately 65-70 gm. for type I zirconium and 20-25 gm. for type II zirconium) into a Buchner funnel containing a Whatman 42 filter paper. Apply suction and wash with water, then three times with acetone and twice with ether. Allow the ether to drain off completely. Transfer the sample to a large watch glass, spread out the material with a spatula, and allow to dry by standing overnight. Heat in an oven at 50°C (122°F) for 2 hours. Store in a stoppered bottle.

4.3.3 Particle size determination.

4.3.3.1 Type I zirconium.

4.3.3.1.1 Sieve sizing. Weigh 50 gm. of the dry sample into the top sieve of the specified nest of sieves (see table I) conforming to the requirements of RR-S-366. Cover the sample and shake for 30 minutes in a mechanical shaker geared to produce 300 ± 15 gyrations and 150 ± taps of the shaker per minute. Weigh the portion retained by each sieve and calculate the percentage passing through the sieve in question.

4.3.3.1.2 Subsieve. Determine the particle size distribution on 2.0 gm. of the material that passes through the No. 325 sieve by use of the Andreassen pipet method (see 6.3). The prescribed medium is as follows:

Triethylene Glycol	90 percent by volume
Distilled water	10 percent by volume
100 percent aerosol	0.2 percent (wt/volume)

Calculations are derived from "Stokes Law" according to the following formulas:

(1) $d = \sqrt{\frac{18\eta h \times 10^6}{(D_2 - D_1)gt}}$

(2) $k = \frac{18\,\eta}{(D_2 - D_1)\,g}$

(3) $t = \frac{kh \times 10^6}{d^2}$

where: d = diameter in microns in formular 1 and 3
k = a constant in formulas 2 and 3
t = time of fall in seconds in formulas 1 and 3
n = viscosity in centipoises of dispersing medium at a definite temperature in formulas 1 and 2
h = distance of fall in cm. in formulas 1 and 3
D_1 = density of the dispersing medium in a gm cc at the same temperature as "n" in formulas 1 and 2
D_2 = density of particle in gm/cc in formula 1; in formula 2, D_2 is density of sample at same temperature as "n"
g = acceleration due to gravity = 980 cm/sec^2 in formulas 1 and 2

(4) $\frac{\text{wt. of withdrawal x 100}}{\text{wt. of initial withdrawal}}$ = Percentage of particles of fraction passing through a No. 325 sieve

4.3.3.2 Type II zirconium (see 6.9)

4.3.3.2.1 Sieve sizing.

4.3.3.2.2 Sieves No. 120 and 200. Weigh 10 gm. of the dry sample into a tared No. 120 sieve. Rest this sieve on a tared No. 200 sieve, if the latter sieve is to be used (see table II). Both sieves should be 3 inches in diameter and conform to the requirements of RR-S-366. Wash the sample with a needle spray of water until no more material passes through the sieves. Rinse with acetone, allow to air-dry, and then heat at 80°C (176°F) for 2 hours, cool, and weigh. Calculate the percent of the material passing through the sieves.

4.3.3.2.3 Buckbee Mears Sieve No. 26. Place a Buckbee Mears Sieve No. 26 (see 6.4) into a funnel over a 400 ml. beaker. For type I, weigh 2 gm. of the fines from 4.3.3.2.2 into a watch glass; for type II, weigh 2 gm. of the original dried sample into a watch glass. Add a few ml. of 5 percent sodium pyrophosphate solution. Disperse the sample to a smooth paste with a rubber policeman and wash into the sieve with water from a wash bottle. Wash the fine particles through the sieve, using dilute sodium pyrophosphate solution (20 ml. of 5 percent pyrophosphate solution per liter of water) and a camel's hair brush. Wash with acetone and allow the sieve to dry in the air. Transfer the residue to a tared weighing bottle, dry at 80°C (176°F) for 1 hour, cool in a desiccator, and weigh. Calculate the percent of the material passing through the sieve.

4.3.3.2.4 Subsieve. The particle size distribution of type II zirconium shall be determined by use of the Eagle-Picher Turbidimeter (see 6.5).

4.3.3.2.4.1 Preparation of sample. Place approximately 0.1 gm. of the powder sample on a clean watch glass and add 5 drops of 0.11 molar aqueous sodium pyrophosphate solution. Disperse the sample and solution with a rubber policeman until thoroughly blended, and all aggregates broken down. Place a 20 micron sieve in a funnel whose stem leads to a 250 ml. graduated cylinder, fitted with a ground glass stopper. Pour a quantity of saturated sodium pyrophosphate solution onto the 20 micron sieve sufficient to give a total of 2.4 ml. of solution on the watch glass and sieve. Wash the sample carefully from the watch glass and policeman through the sieve, using water from a wash bottle. A small clean camel's hair brush may be used to assure that all particles smaller than 20 microns pass the sieve. Wash the sieve thoroughly with water to remove all the particles from its bottom. Wash the funnel and remove it. Dilute the sample suspension contained in the graduated cylinder with water to 100 ml. Shake the sample contained in the graduated cylinder vigorously to obtain a uniform suspension. Pour approximately 15-20 ml. of the suspension quickly into a second 250 ml. glass stoppered graduated cylinder and dilute to 100.0 ml. The temperature of the diluted sample shall be held constant to plus or minus 1°C during all subsequent operations.

4.3.3.2.4.2 Adjustment of turbidimeter. Turn on the light source at least 15 minutes before use to achieve stability. Connect the microammeter. Clean the transmission cell well and place it in the turbidimeter. Place the copper funnel over the cell and set the light shield in place to obstruct the passage of light through the cell. Pour 100.0 ml. of water into the cell through an unconstricted glass funnel. Adjust the light source iris diaphragm until a reading of 100.0 microamperes is obtained on the microammeter. Allow the turbidimeter to remain undisturbed for 5 minutes and determine that the microammeter reading remains unchanged. Adjust the cell elevator so that the distance from the surface of the Water to the center of the light slot is 1.25 cm.

4.3.3.2.4.3 Adjustment of sample concentration. Remove and empty the transmission cell, and replace it in the turbidimeter. Place the copper funnel over the cell and set the light shield in place to obstruct the passage of light through the transmission cell. Shake the diluted sample suspension in the graduated cylinder to disperse it completely. Pour the sample quickly through an unconstricted glass funnel in the transmission cell, rotating the cylinder as the suspension flows from it. Immediately after the suspension is poured into the cell, remove the light shield, and note the microammeter reading. The initial microammeter reading should be between 60 and 80. If it is not, repeat the applicable parts of above using a new sample concentration, until the initial microammeter reading is between 60 to 80.

4.3.3.2.4.4 Recording of data. Record the reading of the microammeter every 0.2 minute for the first minute; every 0.5 minute for the second and third minutes; every minute up to 20 minutes, and then every 5 minutes until 80 minutes have passed.

4.3.3.2.4.5 Calculation of results. Using Keuffel and Esser No. 46-100 (old No. 358-81) graph paper, or equivalent, plot the data points. Plot the percent absorption on the linear axis and the time in minutes on the logarithmic axis. Draw a best fit smooth curve through the data points extending the curve at the upper end to the 0.05 minute interval. Draw reference marks on the curves at the time at which the microsizes have fallen past the reading level. The time of fall values are given in table IV for the different size particles. Using projection and work sheets or equivalent, obtainable from the Joplin Printing Company, Joplin, Missouri, lay out a graph in the right hand section plotting the percent light absorbed values from each particle size from 0.5 to 8.0 microns inclusive. Draw a smooth curve through the points using a Dietzgen curve #2152-19 or equivalent and project the curve smoothly to the origin.

In the first column of the lower left hand corner, record the radius intervals listed in table IV with the smallest radius interval at the top. Record the percent absorption values for the different radius intervals in the next column head PA (prior absorption - the percent absorption prior to the interval in question). The values in this column above 8 microns will be taken from the original plot of K & E No. 46-6010 and for 8 microns on down from the second graph. The next column (Δ A) is for incremental light absorbed for the stated radius interval. It is obtained by subtraction of the successive prior absorptions of the previous column. To find the EH (equivalent hidings), multiply each Δ A figure by the appropriate light (equivalent hiding) factor in table IV. Total the EH column and divide each EH figure by the total to determine the weight percent for the next column. Add the percentage cumulatively for the last column.

To determine the final percentage less than 9, 3 and 0.75 microns, select the appropriate figure from the cumulative percent column and multiply each by the fraction less than 20 microns as follows:

$$\text{Final percent corrected for 20 micron removal} = \frac{D(100-A)}{100}$$

where: D = percent cumulative less than the diameter selected.
A = percent on 20 micron sieve as determined in 4.3.3.2.3.

Table IV. Time of fall values for zirconium powder (Eagle-Picher Turbidimeter)

Radius interval (microns)	Time of fall (minutes)	Avg. diameter (microns)	Light factor
15-20	0.080	35	
10-15	0.182	25	33.3
8-10	0.284	18	23.8
6-8	0.525	14	18.7
5-6	0.725	11	14.7
4-5	1.14	9	12.0
3-4	2.02	7	9.3
2-3	4.54	5	6.67
1-2	18.2	3	4.00
0.5-1	72.6	1.5	2.40
0.25-0.5	290	0.75	1.90
0.0-0.25	1161	0.25	1.45

4.3.3.2.5 Average particle size. The average particle size shall be determined by the Fisher subsieve method as described in MIL-STD-1233, method 100.

4.3.4 Determination of zirconium.

4.3.4.1 Reagents.

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

4.3.4.1.2 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.

4.3.4.2 Procedure. Transfer a 0.23 to 0.25 gm. of the dry 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 (176°F)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 at about 80°C (176°F) for 40 minutes while stirring occasionally. Wash down the cover list 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.

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 (176°F) on the hot plate. Place the beaker in an oven at about 80°C (176°F), cover with an inverted 1 liter beaker, and allow to stand overnight. Filter through a Whatman No. 40 filter paper and transfer 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

Note: Hafnium is counted with the zirconium.

4.3.5 Determination of calcium (see 6.6).

4.3.5.1 Reagents.

4.3.5.1.1 Standard calcium solution No. 1 (1 ml. = 1.0 mg. of Ca). Dry reagent grade calcium carbonate for 3 hours in an oven at 110°C (230°F), transfer 1.2486 gm. to a covered 400 ml. beaker, add 10 ml. of dilute hydrochloric acid (1 to 3), wash down the sides of the beaker, boil for 3 minutes, cool, and dilute to 500 ml. in a volumetric flask.

4.3.5.1.2 Standard calcium solution No. 2 (1 ml. = 0.01 mg. of Ca). Pipet 5 ml. of standard calcium solution No. 1 into a 500 ml. volumetric flask and dilute to the mark. Prepare fresh daily.

4.3.5.1.3 Methyl orange indicator. Dissolve 0.1 gm. of methyl orange in 100 ml. of water.

4.3.5.1.4 Murexide solution. Dissolve 40 mg. of good grade murexide in 75 ml. of water at 10°C (50°F) in a glass stoppered 250 ml. pyrex bottle and add 175 ml. of 95 percent ethyl alcohol. Store in a refrigerator at about 10°C (50°F).

4.3.5.2 Preparation of calibration curve. Transfer 2.0, 4.0, 6.0, 8.0, and 10.0 ml. portions of standard calcium solution No. 2 to 800 ml. beakers, and add 50 ml. of water, 2 ml. of sulfuric acid, 2.5 gm. of ammonium chloride, and 3 drops of methyl orange indicator. Carry a reagent blank through the entire procedure (this is essential). While swirling the beaker add ammonium hydroxide (1 to 1) until the solution turns yellow and then add 30 ml. of nitric acid. Boil down without cover glasses at the highest heat of the hot plate to fumes of sulfuric acid and continue heating to the complete disappearance of fumes. Allow to cool, wash down the sides of the beaker from the very top with a wash bottle, and add 25 ml. of nitric acid and 25 ml. of hydrochloric acid. Again heat at highest heat of the hot plate to the complete disappearance of fumes. Allow to cool, wash down the sides of the beaker and boil down to dryness. Again allow to cool, add 30 ml. of water, and boil down to 20 ml. Add 5 ml. of water and wash into 50 ml. volumetric flasks. Cool to 10° to 15°C (50° to 59°F) in a cold water bath.

Remove the volumetric flasks from the water baths, add 2.0 ml. of 0.1N sodium hydroxide solution and wash down the sides with a little water. Add 10.00 ml. of murexide solution, dilute to the mark, and within 30 minutes measure the absorbence at 505 nm with a spectrophotometer that has been set to 100 percent transmittance with the reagent blank. Plot mg. of calcium against absorbence.

4.3.5.3 Procedure. Transfer 0.4 gm. of the sample to a platinum dish and add 10 ml. of water, 4 ml. of sulfuric acid, and 2 ml. of hydrofluoric acid, and swirl. Carry a reagent blank through all steps of the procedure (this is essential). Wash down the sides with water, evaporate to fumes of sulfuric acid and fume for 2 or 3 minutes. Allow to cool.

If heavy metals such as copper or nickel are present in amounts greater then 0.05 percent, perform a mercury cathode electrolysis, otherwise proceed as in the next paragraph. For the mercury cathode electrolysis, dilute the solution to about 150 ml. with water, wash into a mercury cathode cell, and electrolyze at 5 volts for 45 minutes. Filter through a Whatman No. 42 filter paper into a 400 ml. beaker that has graduated markings.

Dilute to about 300 ml., add 5 gm. of ammonium chloride and 3 drops of methyl orange indicator, and heat to about 80° to 90°C (176° to 194°F). Remove each beaker from the hot plate individually, and add ammonium hydroxide (1 to 1) from a buret with vigorous stirring until the first sign of a precipitate, and then continue the addition dropwise until the solution just becomes yellow. Dilute to the 350 ml. mark with hot water (temperature 80° to 90°C (176° to 194°F)) and allow the beaker to stand near the edge of the hot plate for 5 minutes so that the precipitate settles. Filter immediately through a Whatman No. 41 filter paper into a 400 ml. beaker with graduated markings. Discard the first 25 ml. of solution and collect 175 ml.

If the manganese content is greater than 0.05 percent, make a manganese dioxide precipitation, otherwise proceed as in the next paragraph. To make the manganese dioxide precipitation, add 10 ml. of bromine water and 5 ml. of ammonium hydroxide (1 to 1) to the 175 ml. of solution in the 400 ml. beaker. Digest to 95°C (203°F) for 15 minutes, filter through a Whatman No. 42 filter paper into a 800 ml. beaker, and wash with water.

Add 30 ml. of nitric acid to the solution (contained in a 800 ml. beaker) and proceed with the evaporations and development of the color as described under the preparation of calibration curve. Convert the reading to mg. of calcium by referring to the calibration curve. Calculate the percent calcium as follows:

$$\text{Percent calcium} = \frac{A}{10W}$$

where: A - mg. of Ca as read from curve
W = gm. of sample in 50 ml. volume on which color measurements are made (ordinarily, W = 1/2 x 0.4 = 0.2 gm.).

4.3.6 Determination of iron (see 6.6).

4.3.6.1 Reagents.

4.3.6.1.1 Standard iron solution (1 ml. = 0.100 mg. of Fe). Dissolve 0.1000 gm. of pure iron in 100 ml. of hydrochloric acid (1 to 1) by warming on the hot plate, cool, and dilute to 1 liter in a volumetric flask.

4.3.6.1.2 Hydroxylamine hydrochloride solution (10 percent). Dissolve 50 gm. of hydroxylamine hydrochloride in water and dilute to 500 ml.

4.3.6.1.3 Tartaric acid solution (50 percent). Dissolve 250 gm. of tartaric acid in water and dilute to 500 ml.

4.3.6.1.4 Ortho-phenanthroline solution (0.20 percent). Dissolve 1 gm. of ortho-phenanthroline monohydrate in water at 50°C(122°F), cool, and dilute to 500 ml.

4.3.6.2 Preparation of calibration curve. Transfer 0.5, 1.0, 2.0, 3.0, and 3.5 ml. portions of standard iron solution to 250 ml. beakers. Carry along a reagent blank. Dilute to about 50 ml. with water and add 5 ml. of tartaric acid solution (50 percent), 1 ml. of hydroxylamine hydrochloride solution (10 percent), and 5 ml. of ortho-phenanthroline solution (0.2 percent). Adjust the pH to about 5.5 to 6.0 with ammonium hydroxide (1 to 2) using pH paper. Transfer to 100 ml. volumetric flasks and dilute to about 90 ml. with water. Heat in a water bath at 60° to 70°C (140° to 158°F) for 30 minutes. Cool, and dilute to the mark. Measure the absorbence at 510 nm in a spectrophotometer that has been set to 100 percent transmittance with the reagent blank. Plot mg. of iron against percent transmittance.

4.3.6.3 Procedure. Transfer a 0.5 gm. sample to a platinum dish and add 15 ml. of water and 7 ml. of sulfuric acid. Add 2 ml. of hydrofluoric acid in 0.5 ml. portions. Evaporate to strong fumes of sulfuric acid at moderate heat. Add 50 ml. of water, warm to dissolve the salts if necessary, cool, and dilute to 100 ml. in a volumetric flask. Transfer a 20 ml. aliquot to a 250 ml. beaker. Add 5 ml. of tartaric acid (50 percent) and proceed as in the preparation of the calibration curve. Carry along a reagent blank. Convert the reading to mg. of iron by consulting the calibration curve. Calculate as follows:

$$\text{Percent iron} = \frac{A}{10W}$$

where: A = mg. of iron as read from curve
W = gm. of sample in aliquot

4.3.7 Determination of aluminum (see 6.6).

4.3.7.1 Reagents.

4.3.7.1.1 Standard aluminum solution No. 1 (1 ml. = 0.5 mg. of Al). Dissolve 0.5000 gm. of pure aluminum metal in 25 ml. of hydrochloric acid (1 to 1) by heating on a hot plate, cool, and dilute to 1 liter in a volumetric flask.

4.3.7.1.2 Standard aluminum solution No. 2 (1 ml. = 0.025 mg. of Al). Pipet a 10 ml. aliquot of standard aluminum solution No. 1 into a 200 ml. volumetric flask and dilute to the mark. Prepare fresh daily.

4.3.7.1.3 Sodium hydroxide solution (25 percent). Dissolve 125 gm. of reagent grade sodium hydroxide pellets (preferably obtained from a fresh unopened bottle) in about 400 ml. of water contained in a 500 ml. plastic bottle, cool, and dilute to 500 ml.

4.3.7.1.4 Sulfuric acid (5 percent). Add 25 ml. of sulfuric acid to water, cool, and dilute to 500 ml.

4.3.7.1.5 Phenolphthalein solution (0.1 percent). Dissolve 0.5 gm. of phenolphthalein in a mixture of 250 ml. of ethyl alcohol (95 percent) and 250 ml. of water.

4.3.7.1.6 Benzoic acid solution (10 percent). Dissolve 50 gm. of benzoic acid in 500 ml. of methanol.

4.3.7.1.7 Acetate buffer for aluminon reagent. Cool 950 ml. of ammonium hydroxide in an ice bath and add 860 ml. of glacial acetic acid slowly with stirring. Cool to room temperature and add more acid or base to bring the pH between 5.25 and 5.35 when the solution is diluted 1 to 20. Dilute to 2 liters with water.

4.3.7.1.8 Gelatin solution (1 percent). Add 7 gm. of gelatin to about 500 ml. of warm water while stirring. Warm until clear, cool, and dilute to 700 ml.

4.3.7.1.9 Composite aluminon reagent. Dissolve 0.7 gm. of aluminon in about 400 ml. of water, add 140 ml. of benzoic acid solution (10 percent), and dilute to 700 ml. with water. Add in order, 700 ml. of buffer solution and 700 ml. of gelatin solution (1 percent). Shake well. Allow to stand three days before using. Store in a dark bottle.

4.3.7.2 Preparation of calibration curve. Add 0.2, 0.5, 1.0, 3.0, 5.0, 8.0, 10.0, and 12.0 ml. portions of standard aluminum solution No. 2 to 250 ml. plastic beakers. Carry along a reagent blank. Add 75 ml. of water, 15 ml. of sodium hydroxide solution (25 percent), 10 ml. of sulfuric acid (5 percent), 5 ml. of hydrochloric acid, and 2 or 3 drops of phenolphthalein solution (0.1 percent). Insert a plastic stirring rod, add hydrochloric acid dropwise from a buret until the solution becomes colorless and then add 3 drops excess hydrochloric acid. Wash into 200 ml. volumetric flasks (the volumes after the washings should be about 125 ml.) and add 25 ml. of composite aluminon reagent measured with a pipet. Place into 1 liter beakers containing about 900 ml. of tap water that is boiling vigorously and keep in the boiling water for 15 minutes with the burners of the hot plate at highest heat. Remove the volumetric flasks from the boiling water and dilute to the neck with water. Cool to room temperature in a running water bath and dilute to the mark. Within 2 hours measure the absorbance at 540 nm with a spectrophotometer that has been set to 100 percent transmittance with the reagent blank. Plot mg. of aluminum against absorbance.

4.3.7.3 Procedure. Weigh 1 gm. of the sample into a platinum dish and add 20 ml. of water, 3 ml. of nitric acid, 5 ml. of sulfuric acid, and 3 ml. of hydrofluoric acid. Carry along a reagent blank. After the vigorous reaction has ceased, wash down the sides. Evaporate to fumes of sulfuric acid and fume for 2 or 3 minutes. Allow to cool, wash down the sides, and again evaporate to fumes. Allow to cool, add about 50 ml. of water, warm to dissolve the salts and cool.

If more than 0.3 percent tin is present, electrolyze with a mercury cathode for 30 minutes. Transfer to 100 ml. volumetric flasks and dilute to mark. Add 30 ml. of sodium hydroxide solution (25 percent) to 500 ml. plastic bottles that have a mark on the outside indicating the 200 ml. volume (thick-walled hydrofluoric acid containers are satisfactory). Place the plastic bottles in 1 liter beakers containing 900 ml. of tap water that is boiling vigorously and keep in the vigorously boiling water for 10 minutes or more, swirling the bottles ocasionally. The large volume of water is necessary to prevent the plastic bottles from adhering to the sides of the beakers. Remove each bottle individually from the boiling water bath and immediately add by means of a pipet 20 ml. of the sulfuric acid solution of the sample while swirling the bottle. Dilute almost to the 200 ml. mark, cool in a running water bath to room temperature, and dilute to the 200 ml. mark.

Filter through Whatman No. 41H filter papers into 250 ml. plastic beakers that have a mark on the outside indicating the 100 ml. volume. Discard the first 25 ml. of filtrate and collect 100 ml. Add 5 ml. of hydrochloric acid and 2 or 3 drops of phenolphthalein solution, and proceed as described under preparation of calibration curve. Convert the reading to mg. of aluminum by referring to the calibration curve. Calculate the percent aluminum as follows:

$$\text{Percent aluminum} = \frac{A}{10W}$$

where: A = mg. of Al as read from curve
W = gm. of sample in 200 ml. volume on which color measurements are made (ordinarily, W = 1/2 x 20/100 x 1 = 0.1 gm.)

4.3.8 Determination of hydrogen (see 6.7 and 6.8)

4.3.8.1 Apparatus. Hot-vacuum-extraction apparatus, as shown in figure 1. The apparatus must be constructed with a known volume, V_1, which is used for calibration purposes. The capacity of this volume should be of the same order of magnitude as the total volume of the system and must be calibrated with ± 1 percent prior to incorporation into the apparatus. Calibration by weighing the bulb empty and filled with water is satisfactory. The McLeod gage is of the triple range type, covering 0 to 50 on the quadratic scale, and 0 to 0.5 mm and 0 to 5 mm on the two linear scales. Any other type of gage that will yield readings of comparable accuracy is satisfactory.

4.3.8.2 Reagents.

4.3.8.2.1 High vacuum grease for all stopcocks and standard taper joints not directly in heating zone (Apiezon N grease is satisfactory for this purpose).

4.3.8.2.2 High vacuum wax for all standard-taper joints associated with heated zones (Apiezon N wax is satisfactory for this purpose).

4.3.8.3 Calibration of volume.

(a) Assemble the apparatus as shown in figure 1. Evacuate the apparatus, with both diffusion pumps operating, to 0.1 micron or less. Isolate the calibrated volumes from the evacuating diffusion pumps and forepump. Determine the leak rate of the volumes by periodic measurement of the pressure with the McLeod gage. The leak rate should be less than 0.3 micron per minute.

(b) Introduce hydrogen into the apparatus until a pressure of approximately 0.25 to 0.5 mm is attained. This may be done by heating a high hydrogen content sample, preferably zirconium hydride, in the furnace assembly. (Alternatively hydrogen may be admitted by heating a small palladium tube, sealed outside the volume system, with a soft yellow gas flame.)

(c) Turn off the furnace power, allow the furnace to cool, and measure the hydrogen pressure in the total volume with McLeod gage.

(d) Isolate the precalibrated volume by closing the proper stopcock. Open the remainder of the volume to the evacuating diffusion pump and forepump and allow the system to pump down to 1 micron or less. Isolate the volume from the evacuating diffusion pump and forepump and expand the gas from the precalibrated volume by opening the appropriate stopcock. Measure the pressure in the volume with the McLeod gage.

(e) Repeat this procedure described in paragraph (d) at least eight more times. Calculate the total calibrated volume as follows:

$$V_T = \frac{P_1 V_1}{P_T}$$

where: V_T = total volume, including the McLeod gage, in ml.
P_1 = initial pressure reading-pressure in precalibrated volume in microns.
V_1 = precalibrated volume in ml.
P_T = pressure, in microns, in total volume after expansion from precalibrated volume

The uncertainty of the mean of the calibration values so obtained should not exceed ± 1 percent (95 percent confidence level). Room temperature should not vary by more than ± 2°C (± 3.6°F) during the calibration and subsequent analytical operations. A temperature correction of 0.3 percent per °C may be made for larger temperature fluctuations.

4.3.8.4 Procedure.

4.3.8.4.1 Blank rate of apparatus. Insert the crucible assembly in the furnace and seal the sample receiver to the apparatus with Apiezon N wax. Load 0.5 gm. samples of the zirconium powder, loosely wrapped in tin foil, in the sample arms. Seal and evacuate the apparatus to about 0.05 micron. Outgas the crucible by heating at 1400°C (2552°F). When the crucible is sufficiently outgassed, lower the temperature to 1200° ± 50°C (2192° ± 90°F) and evacuate to 0.02 micron or less. Determine the blank rate of the calibrated volume in microns per minute by isolating the system from the evacuating diffusion pump and forepump. Measure the pressure increase with the McLeod gage at intervals of 3 minutes for 10 minutes. If the blank rate is greater than 0.3 micron per minute, continue the outgassing procedure until the desired blank rate is obtained.

4.3.8.4.2 Analysis of samples. When a satisfactory blank rate is obtained, open the calibrated volume to the evacuating diffusion pump and forepump and again evacuate to 0.02 micron or less. Isolate the volume from the evacuating pumps and manipulate a sample into the crucible. Turn on the induction furnace power and heat the crucible and sample at 1200° ± 50°C (2192° ± 90°F). Read the pressure on the McLeod gage at 5 minute intervals until the pressure increase equals the blank rate. This concludes the analysis of the first sample. Open the system to the evacuating diffusion pump and forepump and again evacuate to 0.02 micron or less. During this evacuation, turn off the power to the induction coil, allow the crucible to cool for about 3 minutes and then by means of the magnetic crucible suspension system, dump the degassed sample into the lower portion of the furnace head. Repeat the procedure for successive samples.

4.3.8.5 Calculation. Calculate the percent hydrogen as follows:

$$\text{Percent hydrogen} = \frac{PVM}{7600\ RTW}$$

where: P = corrected pressure from McLeod gage, in microns.
V = calibrated volume use, in ml.
M = molecular weight of hydrogen = 2.016
R = gas constant (82.05 ml. atmosphere per degree Kelvin mole)
T = temperature of calibrated volume, in degrees Kelvin
W = gm. of sample used

4.3.9 Determination of chloride.

4.3.9.1 Reagents.

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

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

4.3.9.2 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 (167° to 176°F) during this time. Remove the plastic beaker, cover 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 (257°F) 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

4.3.10 Determination of silicon (see 6.6).

4.3.10.1 Reagents.

4.3.10.1.1 Hydrofluoric acid (10 percent). Dilute 50 ml. of hydrofluoric acid (48 percent) to 500 ml. in a plastic bottle.

4.3.10.1.2 Boric acid solution (0.85M). Dissolve 26.3 gm. of boric acid in warm water, cool, and dilute to 500 ml. Store in a plastic bottle.

4.3.10.1.3 Ammonium carbonate solution (20 percent). Dissolve 50 gm. of ammonium carbonate in water and dilute to 250 ml. Store in a plastic bottle. Prepare fresh daily.

4.3.10.1.4 Ammonium molybdate solution (10 percent). Dissolve 25 gm. of ammonium molybdate in water and dilute to 250 ml. Store in a plastic bottle. This solution will keep 3 or 4 days.

4.3.10.1.5 Sodium sulfite solution (17 percent). Dissolve 42.5 gm. of sodium sulfite in water and dilute to 250 ml. Store in a plastic bottle. This solution will keep for 2 days.

4.3.10.1.6 Ammonium tartrate solution (40 percent). Dissolve 100 gm. of ammonium tartrate in warm water, cool, and dilute to 250 ml. Store in a plastic bottle.

4.3.10.1.7 Standard silicon solution (1 ml. = 0.01 mg. of Si). Fuse 0.1070 gm. of anhydrous silicon dioxide with 1 gm. of sodium carbonate in a platinum crucible. Cool the melt, dissolve completely in water, and dilute to 1 liter in a volumetric flask. Store in a plastic or paraffin-lined bottle. Transfer a 200 ml. aliquot to a 1 liter volumetric flask and dilute to the mark. Store in a plastic or paraffin-lined bottle.

4.3.10.2 Preparation of calibration curve. Transfer 10.0, 20.0, 30.0, 40.0, and 50.0 ml. portions of standard silicon solution to plastic beakers. Carry along a reagent blank. Dilute to about 100 ml. and using a pH meter, adjust pH to 2.5 by adding ammonium carbonate solution (20 percent). Add 5 ml. of ammonium molybdate solution (10 percent). Adjust the pH to 2.3 by adding hydrochloric acid (1 to 1). Let stand for 10 ± 0.5 minutes and add 10 ml. of ammonium tartrate solution (40 percent). Transfer to 200 ml. volumetric flasks and dilute to the mark. Let stand for 1 hour and measure the absorbance at 710 nm in a spectrophotometer that has been set to 100 percent transmittance with the reagent blank. Plot mg. of silicon against percent transmittance.

4.3.10.3 Procedure. Weigh a 0.5 gm. sample into a plastic beaker. Carry along a reagent blank. Add 15 ml. of water and 1 ml. of nitric acid (1 to 2) then add 15 ml. of hydrofluoric acid (10 percent) in 3 ml. portions. Warm to about 60° to 70°C (140° to 158°F) in a beaker of hot water to aid solution. Add 150 ml. of boric acid solution (0.65M), mix thoroughly, and cool. Transfer to a 200 ml. volumetric flask and dilute to the marks. Pipet a 50 ml. aliquot into a plastic beaker. Dilute to about 100 ml., adjust the pH to 2.5 by adding ammonium carbonate solution, and develop the color as described under preparation of calibration curve. Convert the reading to mg. of silicon by consulting the calibration curve. Calculate as follows:

$$\text{Percent Silicon} = \frac{A}{10W}$$

where: A = mg. of silicon as read from curve
W = gm. of sample in aliquot

4.3.11 Determination of tin (see 6.6).

4.3.11.1 Reagents.

4.3.11.1.1 Standard potassium iodate solution (0.025N). Dry potassium iodate (KIO_3) at 180°C (356°F) for 2 hours. Dissolve 1.7835 gm. in 100 ml. of water containing 2 gm. of sodium hydroxide and 20 gm. of potassium iodide. Dilute to 2 liters in a volumetric flask. This is a primary standard.

4.3.11.1.2 Antimony trichloride solution (2 percent). Dissolve 2 gm. of antimony trichloride in 50 ml. of hydrochloric acid and dilute to 100 ml. with water.

4.3.11.1.3 Potassium iodide solution (10 percent). Dissolve 10 gm. of potassium iodide in water and dilute to 100 ml. Prepare fresh daily.

4.3.11.1.4 Starch solution. Dissolve 1 gm. of soluble starch in 90 ml. of boiling water, boil for 5 minutes, cool, and dilute to 100 ml.

4.3.11.2 Procedure. Transfer a 2 gm. sample to a platinum dish and add 50 ml. of water and then 10 ml. of hydrofluoric acid in small portions. Add 25 ml. of sulfuric acid and evaporate to fumes. Allow to cool, add 50 ml. of water, and wash into a 500 ml. Erlenmeyer flask. Add 75 ml. of hydrochloric acid and 3 drops of antimony trichloride solution and dilute to about 300 ml. Add 10 gm. of granulated lead (also known as test lead). Boil moderately for 45 minutes while passing carbon dioxide into the flask. Cool to below 10°C (50°F) while passing carbon dioxide into the flask. Add 5 ml. of potassium iodide solution (10 percent) and 5 ml. of starch solution. Titrate with standard potassium iodate solution to a permanent blue color. Run a blank determination. Calculate as follows:

$$\text{Percent tin} = \frac{5.935\ (A-B)\ C}{W}$$

where: A = ml. of potassium iodate solution for titration of sample
B = ml. of potassium iodate solution for titration of blank
C = normality of potassium iodate solution
W = gm. of sample

4.3.12 Ignition gain. Transfer a 0.2 to 0.3 gm. sample, weighed to 0.1 mg., to a tared porcelain crucible. Cover with a porcelain lid and heat over a low flame until the ignition appears complete. Open the cover and heat over the full heat of the Meker burner for 15 minutes. Cool in a desiccator and weigh. Calculate as follows:

$$\text{Percent gain in weight} = \frac{100A}{W}$$

where: A = gm. gain in weight
W = gm. of sample

4.3.13 Burning time.

4.3.13.1 Test equipment.

4.3.13.1.1 Mold bar. One inch by 5/8 inch by 30 inches steel bar with a groove in the center of the one side (the one inch flat side) 1 16 inch deep and 1/8 inch wide running the length of the bar.

4.3.13.1.2 Place of transite. 36 inches by 4 inches by 1/4 inch with a mark 8 inches from each end, giving a 20 inches long timing distance.

4.3.13.2 Procedure. Gently pour the dry sample into the groove, completely filling same. Handle the material with care (see 6.9). Scrape off the excess sample with the edge of a plastic spatula so that the zirconium is level flush with the top of the groove. Do not pack. Place the transite board on top of the bar mold so that the powder extends approximately 5 inches beyond each mark towards each end. Hold board and bar mold with handle together and invert. Place on bench and tap bottom of bar mold with handle of spatula to loosen sample particles. Remove bar mold. With stop watch ready in hand, ignite the zirconium at one end with a match. Start timing when the train burns to the first mark and stop when the burning reaches the record mark. Record time for 20 inches burning and divide by 2. Run four tests on each sample, using the first test to warm up the board. Record the average of the second, third, and fourth tests. Keep the transite board in a warm place when not in use.

5. PREPARATION FOR DELIVERY

5.1 Packing, level C. The zirconium shall be thoroughly mixed with water to form a slurry containing not less than 25 percent by weight of water. It must be packed in wooden boxes, Department of Transportation Specification 15A or 15B, with inside containers of glass or non-carbon polyethylene having net weight of not over 10 pounds each. 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 methyl alcohol-water solution or other anti-freeze mixture approved by the contracting officer, and which has a freezing point of -30°F (-34°C). The zirconium shall be packed for shipment in accordance with the latest Department of Transportation rules and regulations for the transportation of hazardous materials.

5.2 Labeling and marking. Each container should be durably and legibly marked to read:

(a) Zirconium Metal Powder (minimum 25 percent water).
(b) Danger! Flammable solid. May explode if water content is 10 percent or below.
(c) Keep wet in storage - dry powder may be ignited by friction, static electricity, or heat.
(d) Do not attempt to loosen or remove material from container with any tool.
(e) Keep away from heat, sparks, and open flame.
(f) Keep from freezing.
(g) In case of spillage, keep wet and remove carefully.
(h) In case of fire, smother with foam-type fire extinguisher or sand.
(i) Do not use carbon tetrachloride, CO_2 extinguishers or water.
(j) Wear goggles or face shield and fire-retardant clothing when handling.

In addition to any special marking, marking shall be in accordance with MIL-STD-129.

6. NOTES

6.1 Intended use. The zirconium covered by this specification is intended to be used in pyrotechnic and special ignition compositions.

6.2 Ordering data. Procurement documents should specify the following:

a. Title, number and date of this specification
b. Type and class of zirconium

6.3 The principle, technique and calculations for the Andreassen pipet method are described by C. Orr and J. M. Dallavalle in "Fine Particle Measurement: Size, Surface and Pore Volume", MacMillan Co., New York, 1959, Andreassen Pipets may be purchased from Fisher Scientific Company.

6.4 Buckbee Mears Sieve. The Buckbee Mears sieve may be obtained from the Buckbee Mears Company, St. Paul, Minnesota.

6.5 A booklet entitled "Turbidimetric Particle Size Analysis" by Dr. J. R. Musgrave and H. R. Harner may be obtained from the Eagle Picher Co., P. O. Box 47, Joplin, Mo.

6.6 Alternate methods for calcium, iron, aluminum, silicon, and tin. Calcium, iron, aluminum, silicon, and tin may also be determined by satisfactory spectrographic or atomic absorption methods.

6.7 This is essentially the procedure recommended by ASTM for the determination of hydrogen in zirconium and zirconium-base alloys in E 146-65T.

6.8 Alternate method for hydrogen. The hydrogen may also be determined by a satisfactory combustion method.

6.9 Zirconium is a hazardous pyrophoric material, particularly when dry. Handle with care avoiding open flames and unnecessary friction. Type II zirconium is especially pyrophoric.

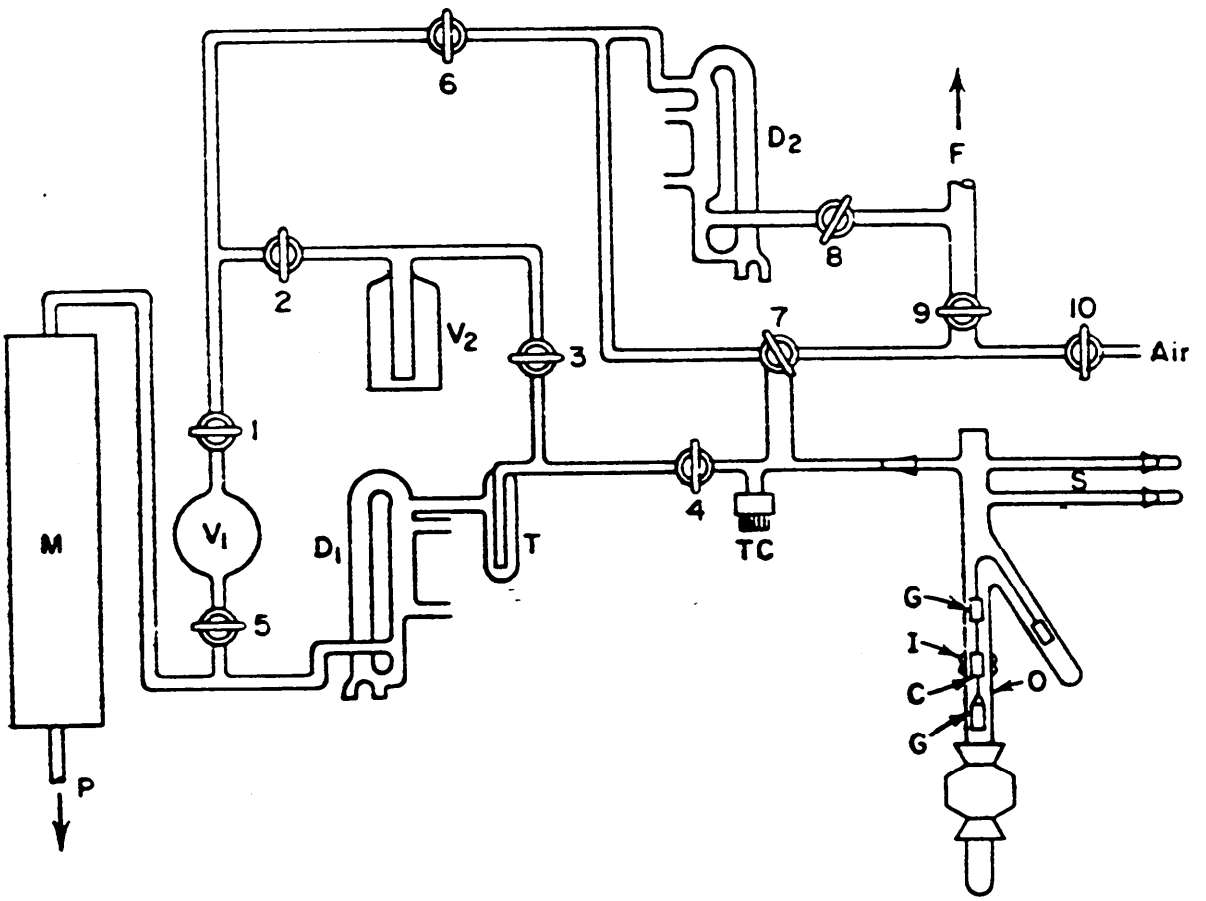
Custodians:
Army - MU
Navy - OS
Air Force - 68

Preparing activity:
Army - MU

Project No. 6810-0904

Review activities:
Army - MD, MU
Navy - OS, AS
Air Force - 68

User activities:
Army - MI, SM



C— Molybdenum crucible.
D_1—Transfer diffusion pump.
D_2—Evacuation diffusion pump.
F—Fore pump.
G—Iron guide tube.
I—Induction heating coil.
M—McLeod gage.
O—Quartz furnace tube.
P—Mechanical pump for McLeod gage.
S—Sample tube.
T—Cold trap.
TC—Thermocouple gage.
V_1—Small volume.
V_2—Large volume.

FIG. 1.—Hot Extraction Apparatus for Determination of Hydrogen in Zirconium.

SPECIFICATION ANALYSIS SHEET

Form Approved
Budget Bureau No. 22-R255

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 JUL 68 1426

REPLACES EDITION OF 1 OCT 64 WHICH MAY BE USED.

FOLD

POSTAGE AND FEES PAID
Department of the Army

OFFICIAL BUSINESS

Commanding Officer
Frankford Arsenal
ATTN: SMUFA-T1000
Philadelphia, Pa. 19137

FOLD