METRIC

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30 September 1997
SUPERSEDING
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1 September 1966

PERFORMANCE SPECIFICATION

ALUMINUM POWDER, SPHERICAL

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

1. SCOPE

1.1 Scope. The specification defines the minimum requirements for four types of spherical aluminum powders used as propellant ingredients.

1.2 Classification. The spherical aluminum powder shall be of the following types, as specified:

Type	Particle Size Range	Percent minimum
I	4.5 - 9.0 µm (micrometers)	99.9
II	12.0 - 18.0 µm	99.0
III	25.0 - 30.0 µm	100.0/85.0
IV	12.0 - 18.0 µm	90.0

2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are specified in sections 3 and 4 of this

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commander, Naval Air Warfare Center Aircraft Division, Code 414100B120-3, Highway 547, Lakehurst, NJ 08733-5100, 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 1336

specification. This section does not include documents in other sections of this specification or recommended for additional information or as examples. While every effort has been made to ensure the completeness of this list, document users are cautioned that they must meet all specified requirements documents cited in sections 3 and 4 of this specification, whether or not they are listed.

2.2 Non-Government publications. The following documents form a part of this document to the extent specified herein. Unless otherwise specified, the issues of the documents which are DoD adopted are those listed in the issue of the DoDISS cited in the solicitation. Unless otherwise specified, the issues of documents not listed in the DoDISS are the issues of the documents cited in the solicitation (see 6.2).

AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

ASTM-B330	-	Average Particle Size of Powders of Refractory Metals and Their Compounds by the Fisher Sub-Sieve Sizer, Standard Test Method for (DoD adopted).

(Application for copies should be addressed to the American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959.)

2.3 Order of precedence. In the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1 First article. When specified (see 6.2), a sample shall be subjected to first article inspection in accordance with 4.3.

3.2 Composition. The chemical composition of the four types of spherical aluminum powder shall be in accordance with table I.

3.3 Physical properties. The physical properties of the spherical aluminum powder shall be in accordance with table II.

3.4 Form. The particles of each of the four types of aluminum powder furnished under this specification shall be spherical in shape.

Table I. Composition.

Ingredients	Limits, percent by weight	
	Minimum	Maximum
Purity	98	---
Volatile @ 104°C (220°F)	--	0.2
Ether extractables	--	0.5

Table II. Physical properties.

Particle size			Average Particle Size (APS) (Fisher sub-sieve size) (µm)
Type	Mesh	Percent Minimum	
I	-325	99.9	4.5 - 9
II	-325	99.0	12.0 - 18.0
III	-200	100.0	25.0 - 30
	-325	85.0	
IV	-325	90.0	12.0 - 18.0

3.5 Workmanship. The material furnished under this specification shall be free from foreign contaminates. It shall be uniform in quality and manufactured in accordance with standard manufacturing procedures of the industry.

4. VERIFICATION

4.1 Classification of inspections. The inspection requirements specified herein are classified as follows:

a. First article inspection (see 4.3)

b. Conformance inspection (see 4.4)

4.2 Lot size. A lot shall consist of material produced by one manufacturer in one continuous operation employing not more than one lot of each ingredient and with no change in formulation or process. If manufacture is by batch process, batches may be combined to form a lot provided not more than one lot of each ingredient and no change in formulation or process is used.

4.3 First article inspection. First article inspection shall consist of all of the conformance inspections and requirements of this specification.

4.4 Conformance inspection. The following procedures shall be performed to determine compliance with section 3. Other test methods may be used if they offer assurance of equal results.

4.4.1 Sampling. Sampling shall be determined by the acquiring activity (see 6.2). One container shall be considered as one unit of product. Each sample shall be representative of the container.

4.4.2 Chemical composition. The chemical composition of the aluminum powder furnished in each lot shall be in accordance with table I. Failure to comply with these requirements shall be cause for rejection of the lot. The composition of each lot of material will be determined by the methods explained in the following paragraphs.

4.4.2.1 Determination of purity.

4.4.2.1.1 Apparatus.

a. Beckman Model K automatic titrator or any hydrogen-ion concentration (pH) meter capable of pH measurement with reproducibility of 0.02 and accuracy of 0.1 pH.

b. Magnetic stirrer, if pH meter is used.

c. Buret, 50 milliliters (mL), for use with pH meter.

4.4.2.1.2 Solutions.

a. Potassium fluoride, 50 percent. Dissolve 250 grams (g) of potassium fluoride dehydrate into 250 mL of distilled water. Add two grams of potassium hydroxide to ensure alkalinity. Adjust the pH of this solution to 10.5 with hydrochloric acid (HCl) and potassium hydroxide, and store in a polyethylene bottle. This solution shall be adjusted to pH of 10.5 before use in aluminum analysis.

b. Aluminum standard solution. React one gram of aluminum of known purity (National Institute of Science & Technology (NIST) standard or equivalent (see 6.3), with 20 mL of 25 percent sodium hydroxide (NaOH) in a 250-mL beaker covered with a watch glass. After the reaction has ceased, boil the solution to ensure complete reaction. Cool and transfer the solution to a one liter volumetric flask. Wash the beaker with six portions of water and add the washings to the volumetric flask. Make up to mark with distilled water.

c. 0.1 normal (N) hydrochloric acid. This solution may be standardized against the aluminum standard solution. The procedure for this standardization is the same one used in

determination of purity, which is given below. The standardization factor, in milligrams (mg) of aluminum (Al), is calculated as follows:

$$\text{mg Al} = \frac{\text{mL of Aluminum aliquot} \quad X \quad \text{sample weight of Aluminum}}{1{,}000}$$

$$\text{Standardization factor} = \frac{\text{mg of Aluminum}}{\text{mL of standard acid}}$$

4.4.2.1.3 Procedure.

a. Weigh 0.25 g of sample to the nearest 0.1 mg.

b. Transfer the sample to the 250-mL beaker and carefully add 10 mL of 25 percent NaOH. Cover the beaker immediately with a watch glass to prevent any loss due to spattering.

c. After initial reaction has ceased, heat the solution to boiling on a hot plate.

d. Cool to room temperature. Wash the watch glass with three portions of distilled water, collecting the washings in the original beaker.

e. Transfer the solution analytically to a 250-mL volumetric flask and make up to the mark with distilled water.

f. Pipette 20 or 25 mL of aliquot into a 250-mL beaker. Add 100 mL of distilled water.

g. Prepare the titrator by filling the buret with the standard HCl solution and turning the instrument on. Set the pH dial to 10.5 and the acid-base switch to set. Turn the selector switch to neutral and the millivolt (mV)-pH switch to pH.

h. Place the beaker containing the sample on the stand and raise the stand so that the electrodes and stirrer are in the solution and the safety switch is activated. Turn the selector switch so that the titrator delivery unit is connected with the rest of the apparatus.

i. Adjust the pH of the solution to just above 10.5 with the aid of strong NaOH and HCl solutions. Set the acid-base switch to base and allow the titration with dilute standard acid to bring the pH of the solution to exactly 10.5.

j. Refill the buret with the standard acid. Add 25 mL of 50 percent potassium fluoride solution to the beaker. The released hydroxyl ions activate the delivery unit and the titration will automatically start and will terminate at exactly 10.5 pH.

k. Calculate as follows:

$$\text{percent Aluminum} = \frac{(\text{mL of standard HCl})(\text{Standardization factor})(250)}{(\text{Sample weight in mg})(\text{mL of aliquot})} \text{ X } 100$$

l. The apparatus can be changed to a continuous pH measurement device such as a Beckman zero-matic pH meter. In that case, a magnastirrer is used for thorough mixing during titration, and the acid is manually added from a 50-mL buret with 0.1-mL graduation.

4.4.2.1.4 Alternate procedure.

a. Weigh 0.9 ±0.05 g, accurate to 0.1 mg, of the sample into a 400-mL beaker and add 50 mL of distilled water.

b. Add 20 mL concentrated HCl in 5 mL increments and heat to boiling to obtain complete solution of the aluminum.

c. Cool, transfer to a 1-liter volumetric flask and dilute to the mark with distilled water.

d. Mix solution thoroughly and pipette a 50 mL aliquot into a 400-mL beaker.

e. Warm the solution to 70°C to 80°C (158°F to 176°F) and add approximately 25 mL of the precipitating agent (5 percent solution of 8 hydroxyquinoline in 2N acetic acid).

f. Slowly add 30-40 mL 2N ammonium acetate. If the liquid above the precipitate is yellow, enough reagent has been added.

g. Allow to stand for one hour.

h. Collect the precipitate in a previously dried and weighed filter crucible (Selas, 3001 or equivalent) and wash well with distilled water.

i. Dry the precipitate (ppt) in an oven at 115°C (239°F) for one and one-half hours.

j. Allow to cool in a desiccator.

k. Weigh and record as residue.

l. Calculate percent aluminum as follows:

$$\text{percent Aluminum} = \frac{\text{(Weight of ppt) (117.41)}}{\text{(Sample weight in grams)}}$$

4.4.2.2 Determination of volatile matter at 104°C (220°F).

4.4.2.2.1 Procedure.

a. Prepare two weighing bottles for each determination by cleaning with chromic acid solution, rinsing with distilled water and then drying the bottles at 110°C (230°F) for one hour.

b. Cool the bottles in a desiccator for 30 ±5 minutes, weigh to the nearest 0.1 mg, and record weight as Tare.

c. Add approximately 5 g of sample to each weighing bottle, reweigh and record the weight as Gross.

d. Place the unstoppered bottles in the oven at 104°C (220°F) for three hours.

e. Stopper and place the bottles in a desiccator for 30 ±5 minutes. Reweigh the bottles and record weight as Residue.

Note: Make all weighings as rapidly as possible.

f. Calculate as follows:

$$\text{percent loss @ 104°C (220°F)} = \frac{\text{(Gross) - (Residue + Tare)}}{\text{(Gross) - (Tare)}} \text{ X } 100$$

4.4.2.3 Determination of ether extractables.

4.4.2.3.1 Procedure.

a. Weigh 50 g of aluminum powder into a 500 mL round bottom flask and cover with 200 mL of diethyl ether.

b. Fit the flask with an Allihn condenser or equal and reflux for one hour using a heating mantle. Caution: Protect from open flames or sparks.

c. Remove the round bottom flask and filter the ether through filter paper into a Claissen flask or equal. Attach a West condenser and adapter, or equal, to the flask. The West condenser and adapter shall enter a second Claissen flask. The second Claissen flask shall be vented by means of rubber tubing or equal.

d. Distill the ether until there is only about 50 to 75 mL of ether left in the flask.

e. Pour the remaining ether into a tared 150-mL beaker and evaporate over a steam bath in a vented hood.

f. When dry, place the beaker in a 110°C (230°F) oven for one hour. Cool in a desiccator and weigh.

g. Calculate as follows:

$$\text{percent Ether extractable} = \frac{\text{Weight of residue in grams}}{\text{Weight of sample in grams}} \text{ X } 100$$

4.4.2.3.2 Alternate procedure.

a. Weigh a sample of the material, to the nearest mg in a Whatman filter thimble. Choose the sample size so that the thimble is filled to within 10 mm of the rim.

b. Place the thimble in a siphon cup. Attach the cup of the cooling coil of the underwriters extraction apparatus.

c. Add 70 mL of the ether in the 400-mL extraction flask and assemble the apparatus as shown in figure 1.

d. Heat the flask on an electric hot plate, or equal, at low temperature. Turn on the cooling water and extract the material for two hours in this manner.

e. Cool, remove the thimble from the cup and reassemble the unit. The excess ether may now be distilled off in the siphon cup and removed when the cup is one-half full. Distill in this manner until about 15 to 20 mL of the liquid remains in the flask.

f. Transfer the remaining liquid in a tared aluminum pan and evaporate to dryness.

g. Cool in a desiccator and weigh.

h. Calculate as follows:

$$\text{percent Ether extractable} = \frac{\text{Weight of residue in grams}}{\text{Weight of sample in grams}} \text{ X } 100$$

4.4.3 Physical properties. The physical properties of the aluminum powder furnished in each lot shall be in accordance with table II and shall be determined in accordance with ASTM-B330. Failure to comply with the requirements shall be cause for rejection of the lot. The physical properties of each lot of material will be determined by the method explained in the following paragraphs.

4.5 Acceptance criteria. All test results shall indicate compliance with the requirements of section 3. Failure to meet these requirements shall be cause for rejection of the lot.

5. PACKAGING

5.1 Packaging. For acquisition purposes, the packaging requirements shall be as specified in the contract or order (see 6.2). When actual packaging of material is to be performed by DoD personnel, these personnel need to contact the responsible packaging activity to ascertain requisite packaging requirements. Packaging requirements are maintained by the Inventory Control Point's packaging activity within the Military Department or Defense Agency, or within the Military Department's or Defense Agency's automated packaging files, CD-ROM products, or by contacting the responsible packaging activity.

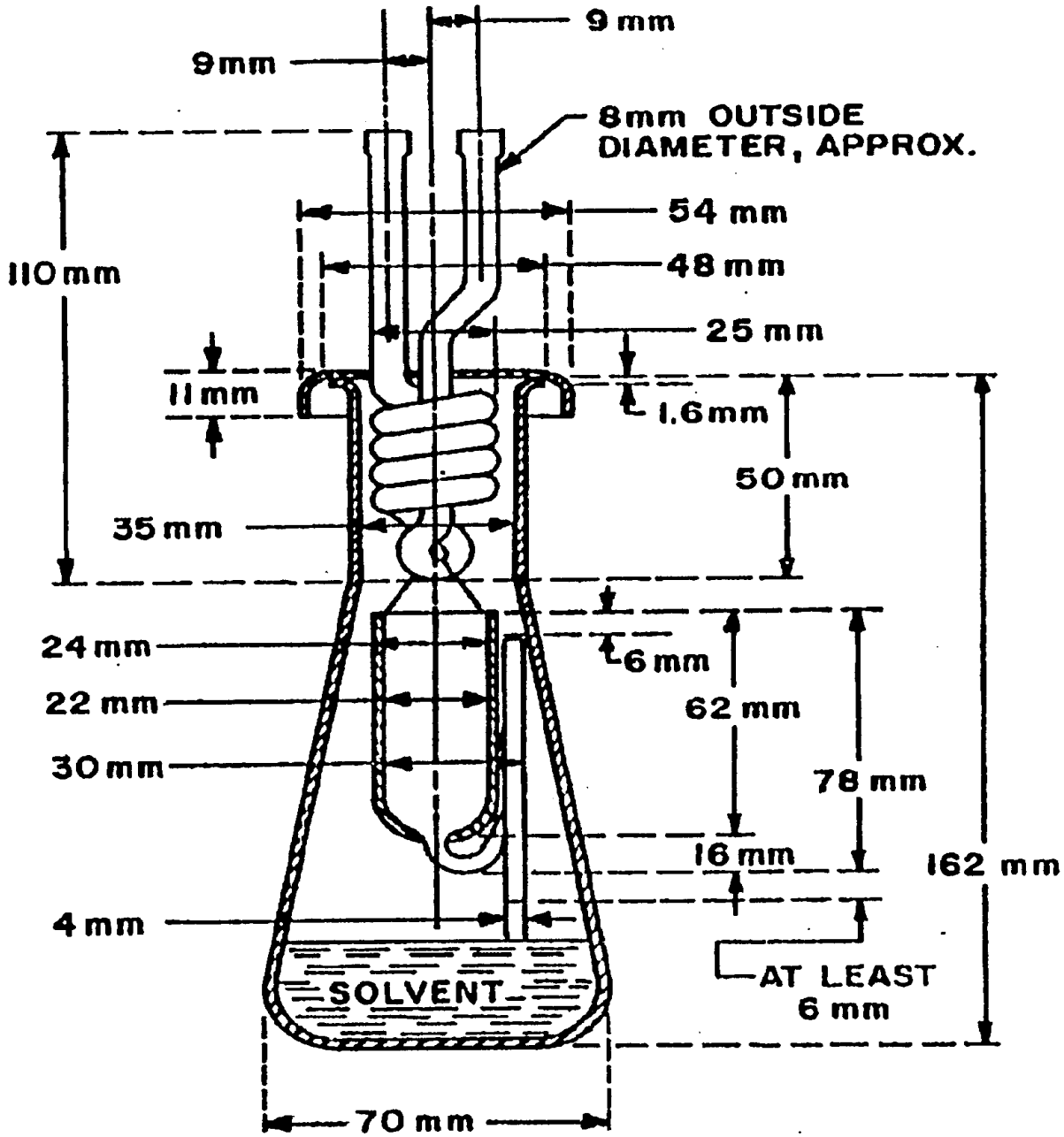

FIGURE 1. Extraction equipment.

6. NOTES

(This section contains information of a general or explanatory nature which may be helpful, but is not mandatory.)

6.1 Intended use. The aluminum powder described by this specification is intended for use as a solid propellant ingredient.

6.2 Acquisition requirements. Acquisition documents should specify the following:

a. Title, number, and date of this specification.

b. Sampling (see 4.4.1).

c. Packaging requirements (see 5.1).

6.3 National Institute of Science & Technology (NIST). Information regarding calibration standards can be obtained by contacting NIST at NIST Public Affairs Division, Administration A903, National Institute of Science & Technology, Gaithersburg, MD 20899-0001. Information may also be obtained by contacting NIST at their Internet Site (http://www.nist.gov).

6.4 Subject term (key word) listing.

Ether extractable
Propellants

6.5 Changes from previous issue. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extent of the changes.

CONCLUDING MATERIAL

Custodian:
Navy - AS

Preparing activity:
Navy - AS

(Project 1336-0009)

STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL

INSTRUCTIONS

1. The preparing activity must complete blocks 1, 2, 3, and 8. In block 1, both the document number and revision letter should be given.
2. The submitter of this form must complete blocks 4, 5, 6, and 7.
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I RECOMMEND A CHANGE:	1. DOCUMENT NUMBER MIL-PRF-23950B(AS)	2. DOCUMENT DATE (*YYMMDD*) 970930

3. DOCUMENT TITLE
Aluminum Powder, Spherical

4. NATURE OF CHANGE (*Identify paragraph number and include proposed rewrite, if possible. Attach extra sheets as needed.*)

5. REASON FOR RECOMMENDATION

6. SUBMITTER

a. NAME (*Last, First, Middle Initial*)	b. ORGANIZATION	
c. ADDRESS (*Include Zip Code*)	d. TELEPHONE (*Include Area Code*) (1) Commercial: (2) DSN: (*If Applicable*)	7. DATE SUBMITTED (*YYMMDD*)

8. PREPARING ACTIVITY

a. NAME COMMANDER NAVAL AIR WARFARE CENTER AIRCRAFT DIVISION	b. TELEPHONE NUMBER (*Include Area Code*) (1) Commercial (732) 323-7488 (2) DSN 624-7488
c. ADDRESS (*Include Zip Code*) CODE 414100B120-3 HIGHWAY 547 LAKEHURST, NJ 08733-5100	IF YOU DO NOT RECEIVE A REPLY WITHIN 45 DAYS, CONTACT: Defense Quality and Standardization Office, 5203 Leesburg Pike, Suite 1403, Falls Church, VA 22041-3466 Telephone (703) 756-2340 DSN 289-2340