MIL-A-23950A (AS)
1 SEPTEMBER 1966
SUPERSEDING
MIL-A-23950 (WEP)
19 AUGUST 1964

MILITARY SPECIFICATION

ALUMINUM POWDER, SPHERICAL

This specification has been approved by the Naval Air Systems Command, Department of the Navy.

1. SCOPE

1.1 **Scope.** The specification defines the minimum requirements for three types of spherical aluminum powder.

1.2 **Classification.** The spherical aluminum powder shall be of the following types, as specified (see 6.2):

Type	*Particle Size Range*
I	4.5–9.0 microns
II	12.0–18.0 microns
III	25.0–30.0 microns

2. APPLICABLE DOCUMENTS

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

STANDARDS

MILITARY

MIL-STD-414 — Sampling Procedures and Tables for Inspection by Variables for Percent Defective

MIL-STD-129 — Marking for Shipment and Storage

FEDERAL

FED-STD-102 — Preservation, Packaging, and Packing Levels

(When requesting any of the applicable documents, refer to both title and number. All requests should be made via the cognizant Government inspector. Copies of this specification and other unclassified specifications and drawings required by contractors in connection with specific procurement functions should be obtained upon application to the Commanding Officer, Naval Supply Depot (Code 105), 5801 Tabor Avenue, Philadelphia 20, Pennsylvania. All other documents should be obtained from the procuring activity or as directed by the contracting officer.)

3. REQUIREMENTS

3.1 **Preproduction.** The preproduction test sample shall be manufactured using the process methods and equipment proposed for production. In the event of significant changes in the process methods and equipment which in the opinion of the procuring activity may adversely affect the characteristics of the material, additional preproduction samples may be required.

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

TABLE I. *Composition*

Ingredients	*Limits, % by Weight* Minimum	Maximum
Purity	98%	
Volatile @ 220°F		0.2%
Ether Extractables		.05%

TABLE II. *Physical properties*

Type	*Particle Size Mesh*	*%Min.*	*APS (Fischer Sub-Sieve Size)*
I	–325	99.9	4.5–9 μ
II	–325	99.	12.0–18.0 μ
III	–200	100.	25. –30 μ
	–325	85.	

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 three types of aluminum powder furnished under this specification shall be spherical in shape.

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

3.6 Data requirements. No data is required by this specification or by applicable documents referenced in section 2 unless specified in the contract or order (see 6.2).

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 supplies and services conform to prescribed requirements.

4.2 Classification of examinations and tests.

(a) Preproduction Tests (4.4)

(b) Quality Conformance Inspection (4.5)

4.3 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.

4.4 Preproduction tests. Preproduction tests shall consist of all of the Quality Conformance Inspections and requirements of this specification.

4.5 Quality 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. If test methods differ from those specified herein, a copy of those methods or references to available sources for the methods shall be furnished the procuring activity.

4.5.1 *Sampling.* Sampling for quality conformance inspections shall be in accordance with Standard MIL-STD-414, Inspection Level IV. One container shall be considered as one unit of product. Each sample shall be representative of the container.

4.5.2 *Chemical Composition.* The vendor is required to certify that the chemical composition of the aluminum powder furnished in each lot is in accordance with Table I (see 6.2). 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.

4.5.2.1 *Determination of Purity.*

4.5.2.1.1 *Apparatus.*

(a) Beckman Model K automatic titrator or any 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 ml, for use with pH meter.

4.5.2.1.2 *Solutions.*

(a) *Potassium Fluoride, 50%.* Dissolve 250 grams of potassium fluoride dehydrate into 250 ml of distilled water. Add two grams of potassium hydroxide to insure alkalinity. Adjust the pH of this solution to 10.5 as accurately as possible with hydrochloric acid and potassium hydroxide, and store in a polyethylene bottle. This solution must be adjusted to pH of 10.5 before use in aluminum analysis.

(b) *Aluminum Standard Solution.* React one gram of aluminum of known purity (NBS Standard or equivalent) with 20 ml of 25% sodium hydroxide in a 250 ml beaker covered with a watch glass. After the reaction has ceased, boil the solution to insure complete reaction. Cool and transfer the solution to a one liter volumetric flask. Wash the beaker with 6 portions of water and add the washings to the volumetric flask. Make up to mark with distilled water.

(c) *0.1 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 is calculated as follows:

$$\text{mg Al} = \frac{\text{ml of Al aliquot} \times \text{sample wt of Al}}{1000}$$

$$\text{Standardization factor} = \frac{\text{mg of aluminum}}{\text{ml of standard acid}}$$

4.5.2.1.3 *Procedure.*

(a) Weigh 0.25 gram of sample to the nearest 0.1 mg.

(b) Transfer the sample to a 250 ml beaker and carefully add 10 ml of 25% 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 of volumetric flask and make up to mark with distilled water.

(f) Pipette a 20 or 25 ml aliquot into 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 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 the pH of the solution to exactly 10.5.

(j) Refill the buret with the standard acid. Add 25 ml of 50% 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) Calculation:

$$\% \text{ aluminum} = \frac{(\text{ml std. HCl})\ (\text{Standardization}) \times 250}{\text{Sample wt in mg} \times \text{ml of aliquot}} \times 100$$

(l) The apparatus can be changed to a continuous pH measurement device such as a Beckman zeromatic 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.

4.5.2.1.4 *Alternate Procedure.*

(a) Weight 0.9 ± 0.05 gram, accurate to 0.1 milligram, of the sample into a 400 milliliter 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 to 80 degrees C. (158 to 176°F) and add approximately 25 approximately 25 ml of the precipitating agent (5% 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 in the 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:

$$\frac{\text{wt of ppt} \times 117.41}{\text{sample wt (gms)}} = \% \text{ Aluminum}$$

4.5.2.2 *Determination of Volatile Matter at 220°F.*

4.5.2.2.1 *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 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 gms of sample to each weighing bottle, reweigh and record the weight as *Gross.*

(d) Place the unstoppered bottles in the oven at 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.

$$\% \text{ loss at } 220°\text{F} = \frac{(\text{Gross}) - (\text{Residue} + \text{Tare})}{(\text{Gross}) - (\text{Tare})} \times 100$$

4.5.2.3 *Determination of Ether Extractables.*

4.5.2.3.1 *Procedure.*

(a) Weigh 50 gm 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 mean of rubber tubing or equal.

(d) Distill the ether until there is only about 50–75 ml of ether left in the flask.

(e) Pour the remaining ether into a tared 150 ml beaker and exaporate 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.

$$\% \text{ Ether Extractable} = \frac{\text{wt of residue gm} \times 100}{\text{wt sample (gms)}}$$

4.5.2.3.2 *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 to 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 2 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 ½ full. Distill in this manner until about 15–20 ml of 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:

$$\text{Ether Extractables} = \frac{\text{wt of residue, gms}}{\text{Sample wt, gms}} \times 100$$

4.5.3 *Physical Properties.* The vendor is required to certify that the physical properties of the aluminum powder furnished in each lot is in accordance with Table II. Failure to comply with these 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.

4.5.3.1 Determination of Particle Size (Fischer Sub Sieve Sizer or Equal).

4.5.3.1.1 *Procedure.*

(a) Screw one of the porous plugs to the plug manipulator, lay a paper disc over one end of the sample tube, and push the plug into the sample tube with the perforated surface of the plug against the surface of the paper disc, forcing the paper into the sample tube. The plug manipulator is then removed.

(b) The sample tube is placed in a vertical position in the rubber support stand with the paper side of the plug up.

(c) Weigh out (to 0.01 gm) a sample of dry powder equal in grams to the true density of the sample.

(d) With the aid of the powder funnel, completely transfer the weighed sample into the sample tube, tapping the side of the tube to settle the powder.

(e) Lay a second paper disc over the top of the sample tube, attach the second porous plug to the manipulator, and force the plug and paper disc down into the sample tube until the powder is compacted enough to move the lower plug. Remove the manipulator.

(f) Place the sample tube on the brass post beneath the rack and pinion with the lower plug in contact with the upper end of the brass post.

(g) Lower the rack, guiding it until the flat bottom end comes in contact with the upper plug. Turn the Pinion Knob firmly until the sample is packed to the desired porosity. (If a particular porosity is desired, first set the Calculator Chart to indicate that porosity. Then pack the sample until the pointer coincides with the Sample Height Curve.)

(h) If not done under Step (g) shift the Calculator Chart laterally until the extreme tip of the point just coincides at some point with the Sample Height Curve on the chart. *The Chart should not be moved after this setting until the determination is finished.*

(i) Mount the sample tube, without disturbing the sample in any way, between the rubber-cushioned supports just to the right of the brass post. Clamp the upper cap down onto the sample tube until an air-tight seal is obtained at both ends.

(j) Plug the line cord into a 110 volt, 60 cycle Alternating Current line. Throw the electrical switch at the lower right hand corner of the front panel to the "ON" position. This turns on the pilot lamp as well as the air pump. The pilot lamp illuminates the tip of the bubbler tube in the pressure regulator stand-pipe, as observed through the round win-

dow in the lower left-hand corner of the front panel as well as the level of the water with relation to the calibration mark as observed through the upper window.

(k) Adjust the Pressure Control Knob, located near the Bubble Observation Window at the lower left of the panel, until the bubbles rise in the stand-pipe at the rate of two to three bubbles per second. This will cause the water level to rise above the calibration mark on the upper end of the stand-pipe. This is normal and does not mean that the calibration is in error.

(l) The liquid level in the manometer tube will rise slowly and reach a maximum within 30 seconds to several minutes, depending on the the particle size. After the maximum rise has been obtained, using care not to disturb the chart, the rack is turned up until the upper edge of the cross bar coincides with the liquid menscus in the manometer. The Particle Size is indicated by the location of the tip of the pointer with relation to the curves on the Calculator Chart. The Chart is like an ordinary graph with the exception that the normally horizontal lines are curved. The fractional parts are obtained by interpolating between curves in the usual manner as interpolating between straight lines on the ordinary graph. (Notice that the diameter value between 0.2 to 4.00 microns representing 0.1 micron, each curve between 4.00 and 8.00 representing 0.2 micron, each curve between 8.00 and 15.0 microns representing 0.5 micron, each curve between 15.0 and 20.0 microns representing 1.0 micron, and the space between 20.0 and 25.0 microns representing 5.0 microns.

(m) If the Average Particle Diameter falls in the range 0.2 to 20.0 microns, read the chart directly with the Range Control Indicator found at the extreme upper right of the front panel, turned to the right. If the Average Particle Size falls between 20.0 and 50.0 microns, turn the Range Control Indicator to the left and multiply the Chart readings by two to secure Average Particle Diameter.

4.6 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. PREPARATION FOR DELIVERY

5.1 Preservation and packaging. Preservation and packaging shall be level C, as defined in Federal Standard FED–STD–102, or as specified (see 6.2). The requirements of section 5 apply only to direct purchases by or direct shipments to the Government.

5.1.1 *Level C.* Preservation and packaging shall be in accordance with standard commercial practice to afford protection against damage, contamination of the product by moisture, corrosion, or other foreign matter.

5.2 Packing. Packing shall be level C, as defined in Federal Standard FED–STD–102, or as specified (see 6.2).

5.2.1 *Level C.* The product packaged as specified in 5.1 shall be packed in a manner to insure carrier acceptance and safe delivery at destination. Containers shall be in accordance with the Uniform Freight Classification Rules or regulations of other carriers applicable to the mode of transportation.

5.3 Marking. Unless otherwise specified by the contract or order, unit packages, intermediate packages, and shipping containers shall be marked in accordance with the requirements of MIL-STD-129 and shall include but not be limited to the following (see 6.2):

(a) Manufacturer's name and address

(b) Gross and net weights

(c) Lot number and date of manufacture

(d) The number of this specification

(e) Safety precautions or unusual storage requirements

(f) Purchase order or contract number

6. NOTES

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

6.2 Ordering data. Procurement documents may specify but not be limited to the following information:

(a) Title, number, and date of this specification

(b) Type and size of container desired

(c) Place of inspection

(d) Minimum lot size, if applicable

(e) Place of delivery

(f) Request for copies of inspection data, if source inspected

(g) Selection of applicable levels of packing and packaging (see 5.1 and 5.2)

(h) Marking requirements (see 5.3)

(i) Data requirements (see 3.6)

6.3 Conflicting requirements. Conflicting requirements arising between this specification or any specifications, publications, or drawings listed herein shall be referred in writing to the procuring activity or appointed agent for interpretation and clarification.

6.4 Request for deviation. Request for deviation from this specification, applicable drawings, specifications, or materials or processes shall be forwarded to the procuring activity prior to incorporating the desired change into production. All deviations shall be limited to the contract under which they were granted. A deviation is a before-the-fact request.

6.5 Request for waiver. Request for waiver from this specification, applicable drawings, specifications, or materials shall be forwarded to the procuring activity. A waiver is an after-the-fact request. A waiver request shall include the following:

(a) Exact nature of the defect

(b) CD number and defect classification, if any

(c) Quantity of items involved

6.6 Value engineering. Manufacturers of material covered by this specification are encouraged to submit value engineering suggestions to the procuring activity in an effort to reduce cost.

6.7 Safety precautions. All detailed safety precautions shall be strictly observed.

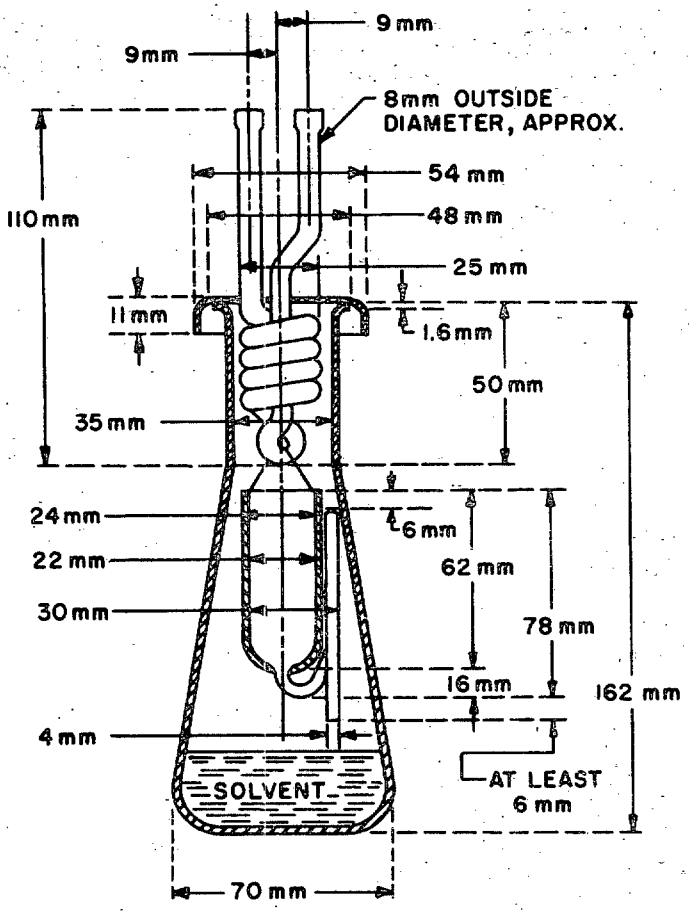

FIGURE 1. EXTRACTION EQUIPMENT

SPECIFICATION ANALYSIS SHEET

Form Approved
Budget Bureau No. 119-R004

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 *(as indicated on reverse hereof)*.

SPECIFICATION

MIL-A-23950A(AS) ALUMINUM POWDER, SPHERICAL

ORGANIZATION *(Of submitter)*	CITY AND STATE

CONTRACT NO.	QUANTITY OF ITEMS PROCURED	DOLLAR AMOUNT
		$

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)*	DATE

DD, FORM 1426
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