

JAN-A-667
20 AUGUST 1948

**NATIONAL MILITARY ESTABLISHMENT SPECIFICATION
ALUMINUM POWDER, SUPERFINE**

This specification was approved by the Departments of the Army, the Navy, and the Air Force for use of procurement services of the respective Departments.

A. APPLICABLE SPECIFICATIONS

A-1. The following specifications, of the issue in effect on date of invitation for bids, form a part of this specification:

U. S. ARMY SPECIFICATIONS

50-0-1—General Specification for Ammunition except Small Arms Ammunition.

100-2—Standard Specification for Marking Shipments by Contractors.

FEDERAL SPECIFICATION

RR-S-303—Sieves; Standard, Testing.

B. GRADE

R-1. This specification covers one grade of superfine aluminum powder.

C. MATERIAL AND WORKMANSHIP

C-1. See section E.

D. GENERAL REQUIREMENTS

D-1. See section E.

E. DETAIL REQUIREMENTS

E-1. *Form of particles*.—Flake.

E-2. *Material volatile at 105° C.*.—Maximum, 1.0 percent.

E-3. *Free metallic aluminum*.—Minimum, 85 percent.

E-4. *Silicon, iron, zinc, manganese, magnesium and other metallic impurities*.—Maximum total, 2.0 percent.

E-5. *Other extractive matter*.—Maximum, 3.0 percent.

E-6. *Apparent density*.—Maximum, 0.30 gm. per ml.

E-7. *Granulation*.—Minimum, 99.0 percent through a No. 325 U. S. Standard sieve conforming to Federal Specification RR-S-303.

E-8. *Oxit*.—None.

F. METHODS OF SAMPLING, INSPECTION, AND TESTS

F-1. *Size of lots*.—Maximum, 100 pounds.

F-2. *Sampling*.—Select 3 of the containers in the lot, the containers being selected in such a manner as to be representative of the lot. These containers shall represent the primary samples. By means of a scoop remove from each selected container a portion of approximately 0.5 pound. Mix the portions removed from the primary samples, place the composite sample in a rubber-stoppered bottle, and label so as to show the name of the material, manufacturer, plant, purchase order and number of pounds in the lot. All acceptance tests shall be made on the composite sample representative of the lot. Hold

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the primary samples for possible future examination should the composite sample fail to meet the requirements.

F-3. Inspection. Inspection shall be made in accordance with U. S. Army Specification 10-0-1 and shall be made at the point of delivery unless otherwise specified in the contract or order.

F-4. Tests—

F-4a. Particle form.—Place a small portion of the sample on a glass slide and examine the material under a high-power microscope. A microscope equipped with an oil immersion lens and having a magnification of approximately 500 has been found to be suitable. Note the form of the material.

F-4b. Material volatile at 105° C.—Transfer a portion of approximately 2 gm. of the material to a tared moisture dish. Weigh the dish and contents and calculate the weight of sample taken for test. Heat the dish and contents for 4 hours at $105^{\circ} \pm 3^{\circ}$ C., cool in a desiccator, weigh and calculate the loss in weight as percent material volatile at 105° C.

F-4c. Free metallic aluminum.—Determine the free metallic aluminum content by the hydrogen evolution method, using a suitable apparatus for dissolving the sample and measuring the gas evolved. The apparatus shown in figure 1 has been found satisfactory, and its use is described below:

Assemble the apparatus shown in figure 1, attaching the various parts to suitable supports. Turn the three-way stopcock to the A position, raise the leveling bulb until the meniscus of the water in the gas-measuring buret is at 0-ml. mark, and turn the stopcock clockwise to the B position. Place 100 ml. of a 10-percent sodium hydroxide solution, previously saturated with hydrogen, in the reaction flask, and attach the reaction flask to the apparatus by means of the two-holed rubber stopper. Adjust the position of the thermometer in the rubber stopper so that the bulb of the thermometer dips into the alkali solution. Weigh accurately a portion of approximately 0.38 gm. of the sample and wrap in cellophane or paper in such a manner as to make the following operations possible. Remove the reaction flask from the apparatus and place the wrapped sample in the neck of the flask without allowing the sample to fall into the alkali. Reattach the reaction flask to the apparatus, again taking care not to allow the sample to fall into the alkali. Allow the sample to remain in the neck of the flask for at least 10 minutes or until the atmosphere above the alkali becomes saturated with water vapor. Bend the temperature of the thermometer and turn the three-way stopcock clockwise to the O position. Cause the sample to fall into the alkali by hitting the bottom of the reaction flask against the palm of the hand. Shake the flask to cause the wrapping on the sample to open and the aluminum to come in contact with the alkali. As the evolution of gas proceeds, lower the leveling bulb so that the level of the water in the gas-measuring buret remains at approximately the same level as the water in the leveling bulb. When the reaction is complete, cool the alkali solution in the reaction flask to its temperature prior to the reaction and keep at this temperature for 10 to 15 minutes. Adjust the level of the water in the leveling bulb to the same height as the meniscus of the water in

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the gas measuring buret. Read the volume of gas in the buret and correct for any error in the graduation of the buret. Read the temperature of the gas by means of a thermometer placed close to the buret. Determine the atmospheric pressure by means of a mercurial barometer accurate to 0.1 mm., and make any necessary temperature correction. Calculate the percent free metallic aluminum as follows:

$$\text{Percent free metallic aluminum} = V \times \frac{(P-p) \times 273.1}{760(273.1+T)} \times \frac{0.09027}{W}$$

Where:

V = ml. of hydrogen evolved at temperature T °C.

P = barometric pressure in mm. of mercury corrected for temperature.

p = vapor pressure of water at temperature T °C in mm. of mercury.

T = temperature of gas in °C.

W = gm. of sample.

F-4d. *Silicon, iron, zinc, manganese, magnesium and other metallic impurities.*—Determine the percentages of silicon, iron, zinc, manganese, magnesium in the sample, using standard methods of chemical analysis. If during the course of the analysis for these impurities the presence of other metallic impurities is detected or indicated, determine the percentage of each of these other metallic impurities using standard methods of chemical analysis. Report the total percentage of all the above impurities present.

F-4e. *Ether extractive matter.*—Extract an accurately weighed portion of approximately 2 gm. of the sample with redistilled ethyl ether for 4 hours in a Soxhlet, or similar, extraction apparatus. The sample should be contained in a suitable extraction thimble capable of retaining most of the fine aluminum particles. Evaporate the ether extract in the extracting flask to a volume of approximately 50 ml. on a steam bath. Filter the extract through filter paper and wash the extraction flask and filter paper with three 10-ml. portions of ethyl ether, catching the filtrate and washings in a tared 150-ml. beaker. Evaporate the ether from the beaker by placing the beaker on a steam bath. Heat the beaker and residue at $90^{\circ}\pm 3^{\circ}$ C. for 1 hour, cool in a desiccator and weigh. Reheat the beaker and residue at $90^{\circ}\pm 3^{\circ}$ C. for 0.5 hour, cool in a desiccator and weigh, repeating these operations until a weight constant to within 5 mg. is obtained. Calculate the increase in weight to percent ether extractive matter.

F-4f. *Apparent density.*—Assemble the Scott Volumeter as shown in figure 2 so that the tared receiving cube rests on the base of the apparatus directly under the lower funnel. The volumeter shall have a No. 40 U. S. Standard screen on the upper hopper, and the receiving cube shall have a volume of 1 cubic inch. Using a quantity of the sample sufficient to fill the receiving cube to overflowing, slowly pour the material through the screen into the hopper. By means of a straight-edged spatula carefully strike off the excess material from the top of the receiving cube, taking care not to jar the cube during this operation. In this operation the straight-edge of the blade of the spatula shall move in an imaginary plane passing through the upper edges of the cube, while the flat of the blade shall be held perpendicular to this plane. Weigh the cube and contents. Calculate the apparent density of the sample in gm. per ml. by dividing the increase in weight

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of the cube in gm, by the volume of the cube in ml. Make 3 determinations and report the average of the results obtained.

F-4g. *Granulation*.—Fill two straight-walled containers, approximately 12 inches in diameter, to a depth of not less than 4 inches with mineral spirits. Fill a similar container to a depth of not less than 3 inches with acetone. Transfer a weighed portion of approximately 5 gm. of the sample to a 250-ml. beaker. Add, in small portions, a total volume of approximately 150 ml. of mineral spirits, mixing the contents of the beaker thoroughly after each addition. It is necessary that all lumps in the sample be thoroughly dispersed in the mineral spirits while the sample is still in the beaker. Clamp a No. 325 U. S. Standard sieve, 8 inches in diameter, just above the level of the mineral spirits in the first container and pour the dispersed sample through the sieve. Wash the beaker with clear mineral spirits until all of the sample has been removed. Pour the washings through the screen. Unclamp the sieve and rock it from side to side, working the bottom of each side of the sieve alternately just under and then just above the level of the liquid in the first container. It is important that the operator secure maximum sieving in each container. After the bulk of the sample has passed through the sieve, the time required being usually about 3 minutes, repeat this procedure in the second mineral spirits container. When it appears that practically none of the residue on the sieve is passing through it, repeat the procedure in the acetone container for 3 minutes or until no more material passes through the sieve. Rinse down the sides of the sieve with a small stream of acetone and collect the residue on one side of the sieve. Transfer the residue to a tared evaporating dish using a small stream of acetone. Evaporate the acetone from the dish on a steam bath. Heat the dish and residue at 105°±3° C. for 15 minutes, cool, weigh and calculate the increase in weight as percent material retained on the 325-mesh sieve. By subtraction from 100 percent, the percent material passing through the sieve is obtained. Retain the residue in the dish for the grit determination.

F-4h. *Grit*.—Transfer the residue retained on the No. 325 U. S. Standard sieve, as obtained by the method prescribed in paragraph F-4g, to a smooth glass slide. Rub the material on the glass by exerting pressure with a smooth steel spatula blade. Note if particles of grit are present as indicated by lack of uniformity of the material and the persistence of a screeching noise when pressing and rubbing of the material on the glass slide is continued.

F-5. *Rejection and resubmission*.—If the composite sample fails to conform to any of the requirements of this specification, the inspection lot shall be rejected. The contractor shall have the option of having a partial or complete analysis made on each container in the lot at no expense to the Government. The contractor may then remove defective portions of the inspection lot and resubmit the lot for acceptance.

G. PACKAGING, PACKING, AND MARKING FOR SHIPMENT

G-1. *Packaging*.—The superfine aluminum powder shall be packaged in standard commercial containers of not more than 5 pounds nor less than 1 pound capacity and so constructed as to insure acceptance by common or other carriers for safe transportation, at the lowest rate, to the point of delivery. The containers shall be provided with a liner

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or be of such construction as to prevent contamination by dust or other foreign materials.

G-2. *Marking*.—In addition to any special marking required by the contract or order, shipments for the Army shall be marked in accordance with the requirements of U. S. Army Specification 100-2.

H. NOTES

H-1. *Use*.—Superfine aluminum powder covered by this specification is intended for use in primer compositions.

H-2. This specification replaces Picatinny Arsenal Tentative Specification P X S-1105.

H-3. Copies of this specification (required for Army purchases) may be obtained from the Office, Chief of Ordnance, Department of the Army, Washington 25, D. C.

Notice.—When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any right or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

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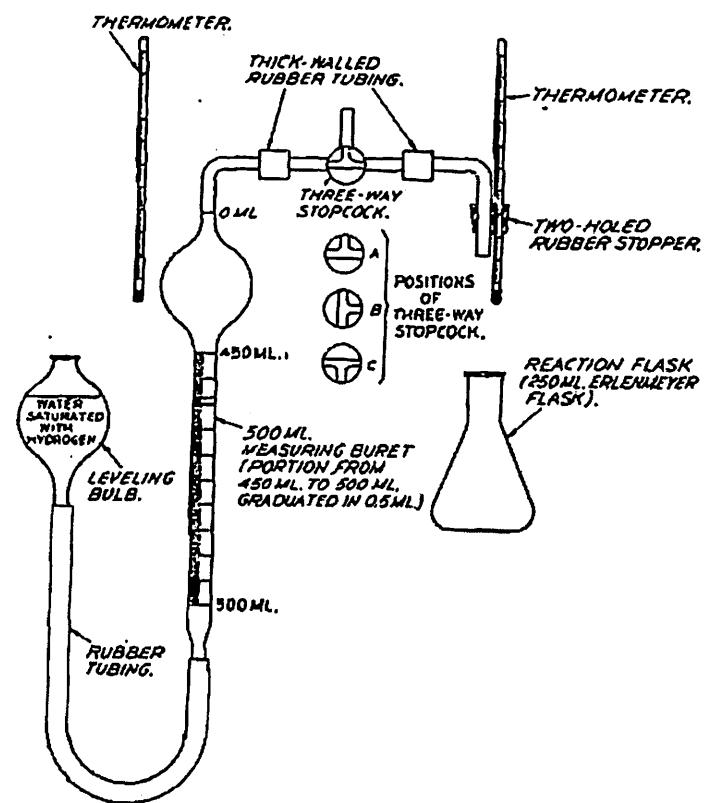


FIGURE 1.—Assembly of apparatus (for determining free metallic content).
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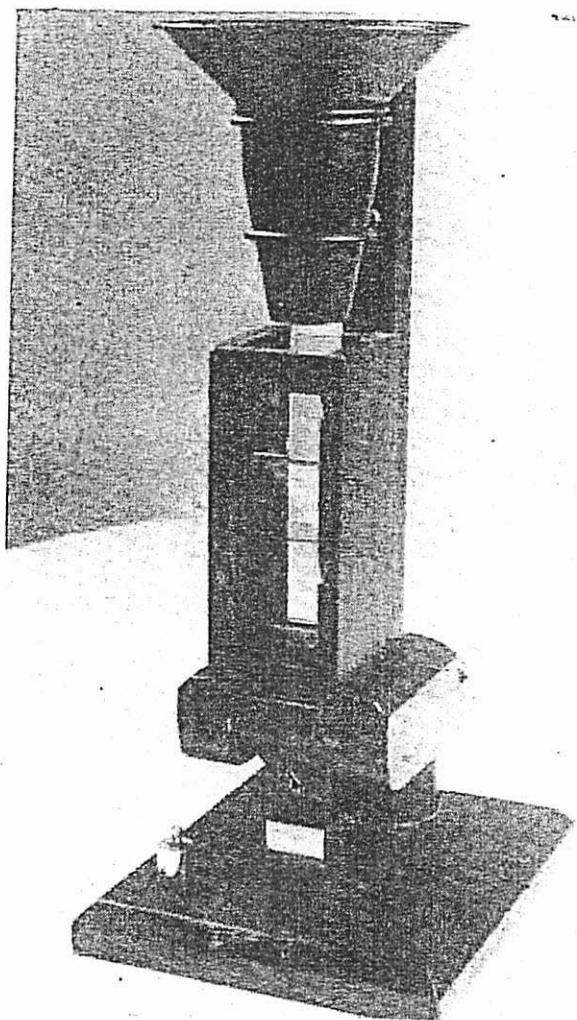


FIGURE 2.—Scott Volumeter.
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