

INCH-POUND

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SUPERSEDING
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DETAIL SPECIFICATION

MAGNESIUM – ALUMINUM ALLOY, POWDERED

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

1. SCOPE

1.1 Scope. This specification covers powdered magnesium-aluminum alloy suitable for use in ammunition.

1.2 Classification. Magnesium-aluminum alloy powder is of the following types:

Type A - 50/50 (magnesium/aluminum)

Type B - 65/35 (magnesium/aluminum)

2. APPLICABLE DOCUMENTS

2.1 General. The documents listed in this section are specified in sections 3 and 4 of this 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 requirements documents cited in sections 3 and 4 of this specification, whether or not they are listed.

Comments, suggestions, or questions on this document should be addressed to: Commander, US Army Armaments Center, ATTN: FCDD-ACE-QSA, Picatinny Arsenal, New Jersey 07806-5000 or emailed to usarmy.picatinny.ardec.list.ardec-stdzn-branch@mail.mil. Since contact information can change, you may want to verify the currency of this address information using the ASSIST Online database at <https://assist.dla.mil>.

AMSC N/A

FSC 1370

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2.2 Government documents.

2.2.1 Specifications, standards, and handbooks. The following specifications, standards and handbooks form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those listed in the solicitation or contract.

DEPARTMENT OF DEFENSE STANDARDS

MIL-STD-1916 - DOD Preferred Methods for Acceptance of Product

(Copies of these documents are available online at <https://quicksearch.dla.mil/>.)

2.3 Non-Government publications. The following documents form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

ASTM INTERNATIONAL

ASTM B215 - Standard Practices for Sampling Metal Powders
 ASTM B329 - Standard Test Method for Apparent Density of Metal Powders and Compounds Using the Scott Volumeter
 ASTM E11 - Standard Specification for Woven Wire Test Sieve Cloth and Test Sieves
 ASTM E300 - Standard Practice for Sampling Industrial Chemicals
 ASTM E3061 - Standard Test Method for Analysis of Aluminum and Aluminum Alloys by Inductively Coupled Plasma Atomic Emission Spectrometry (Performance Based Method)

(Copies of ASTM standards are available online at <http://www.astm.org> or from American Society for Testing and Materials, 100 Barr Harbor Dr., West Conshohocken, PA 19428.)

2.4 Order of precedence. Unless otherwise noted herein or in the contract, 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 Required inspections.

3.1.1 First article. When specified (see 6.2), a sample shall be subjected to first article inspection in accordance with 4.2.

3.1.2 Conformance. A sample shall be subject to conformance inspection in accordance with 4.3.

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3.2 Form. Type A alloy powder shall be granular and shall contain no slivers, chips or machine turnings.

3.3 Chemical composition. Magnesium-aluminum alloy powder shall conform to the requirements shown in Table I.

TABLE I. Chemical composition and physical properties.

Requirement (weight %)	Type A Powder	Type B Powder
Magnesium	50.0 ± 2.0	65.0 ± 2.0
Aluminum	50.0 ± 2.0	35.0 ± 2.0
Total magnesium and aluminum, minimum (min.)	98.0	98.0
Oxides as Al ₂ O ₃ , maximum (max.)	2.0	2.0
Iron as Fe, max.	0.75	0.75
Silicon as Si, max.	0.5	0.5
Other metals, max.	0.5	0.5
Zinc as Zn, max.	0.1	0.1
Grease and fats, max.	0.01	0.01
Moisture, max.	0.05	0.05
Grit, max.	0.1	0.1

3.4 Moisture. The magnesium-aluminum alloy powder shall conform to the moisture requirements as shown in Table I.

3.5 Grease and fats. The magnesium-aluminum alloy powder shall conform to the grease and fats requirements as shown in Table I.

3.6 Grit. The Magnesium-aluminum alloy powder shall conform to the grit requirements as shown in Table I.

3.7 Oxides as Al₂O₃. The Magnesium-aluminum alloy powder shall conform to the oxides as Al₂O₃ requirements as shown in Table I.

3.8 Granulation. Magnesium-aluminum alloy powder shall be in accordance with Table II. All percentages shall be by weight using US Alternative Sieves conforming to ASTM E11. The powder shall pass through the required sieves readily without balling or the particles clinging together.

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TABLE II. Granulation.

Through sieve no.	Type A		Type B	
	Minimum	Maximum	Minimum	Maximum
	Percent	Percent	Percent	Percent
100	99.5	---	90	---
120	98	---	80	---
230	45	65	40	60

3.9 Apparent density (Type A only). The apparent density of the magnesium-aluminum alloy, Type A powder shall be not less than 0.80 grams per cubic centimeter.

3.10 Workmanship. The magnesium-aluminum alloy powder shall be uniform in quality and free from lumps, grit, visible impurities, foreign matter, or other defects that would render the metal powder unsuitable for intended use.

4. VERIFICATION

TABLE III. Requirement/verification cross reference matrix.

Method of verification		Classes of verification						
1 – Analysis 2 – Demonstration 3 – Examination 4 – Test		A – First article B – Conformance						
Section 3 Requirement	Description	Verification Methods				Verification Class		Section 4 Verification
		1	2	3	4	A	B	
3.1.1	First article	X	X	X	X	X		4.2
3.1.2	Conformance	X	X	X	X		X	4.3
3.2	Form			X		X	X	4.5.1
3.3	Chemical composition				X	X	X	4.5.2
3.4	Moisture				X	X	X	4.5.3
3.5	Grease and fats				X	X	X	4.5.4
3.6	Grit				X	X	X	4.5.5
3.7	Oxides as Al ₂ O ₃				X	X	X	4.5.6
3.8	Granulation				X	X	X	4.5.7
3.9	Apparent density				X	X	X	4.5.8
3.10	Workmanship			X		X	X	4.5.9

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

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- a. First article (see paragraph 4.2).
- b. Conformance (see paragraph 4.3).

4.2 First article inspection. When specified, a sample shall be subject to first article verification in accordance to Table IV and 4.4.

4.2.1 First article quantity. The first article sample shall be 10 kilograms of metal powder from the first production lot produced by the contractor using the same production equipment, process procedures, and material supply sources as will be used in regular production.

4.2.2 Rejection. If any sample fails to comply with any of the applicable requirements the first article quantity shall be rejected.

TABLE IV. First article acceptance inspection.

Classification	Metal Powder First Article Acceptance Test	Requirement Paragraph	Inspection Method Reference
<u>Critical</u>	None defined		
<u>Major</u>			
101	Form	3.2	4.5.1
102	Chemical composition	3.3	4.5.2
103	Moisture	3.4	4.5.3
104	Grease and fats	3.5	4.5.4
105	Grit	3.6	4.5.5
106	Oxides as Al ₂ O ₃	3.7	4.5.6
107	Granulation	3.8	4.5.7
108	Apparent density	3.9	4.5.8
<u>Minor</u>			
201	Workmanship	3.10	4.5.9

4.3 Conformance inspection.

4.3.1 Inspection lot formation. Lot formation shall be in accordance with the lot formation requirements of MIL-STD-1916. A lot shall consist of not greater than one type of powder.

4.3.2 Classification of characteristics. Conformance inspection shall be performed in accordance with inspection provisions set forth herein. The characteristics shown in 3.2 through 3.10 when tested in accordance with 4.5 shall constitute minimum inspections to be performed.

4.3.3 Inspection requirements by classification of characteristics.

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- a. For the conformance inspection paragraph 4.3.3.1, the definitions of critical, major and minor defects are provided in paragraph “Definitions” of MIL-STD-1916.

4.3.3.1	<u>Metal powder.</u>	Conformance Criteria	Requirement Paragraph	Inspection Method Reference
<u>Critical</u>	None defined			
<u>Major</u>				
101	Form	4.4	3.2	4.5.1
102	Chemical composition	4.4	3.3	4.5.2
103	Moisture	4.4	3.4	4.5.3
104	Grease and fats	4.4	3.5	4.5.4
105	Grit	4.4	3.6	4.5.5
106	Oxides as Al ₂ O ₃	4.4	3.7	4.5.6
107	Granulation	4.4	3.8	4.5.7
108	Apparent density	4.4	3.9	4.5.8
<u>Minor</u>				
201	Workmanship	4.4	3.10	4.5.9

4.4 Metal powder sampling. A representative sample of not less than two kilograms shall be obtained from each container sampled of magnesium-aluminum alloy powder using ASTM E300 or ASTM B215. Unless otherwise specified, sampling shall be conducted in accordance with Table V. When lots comprise three or less containers, all containers in the lot shall be selected. Each representative sample shall be rolled and tumbled for five minutes prior to inspection. Composites of samples from multiple containers shall not be allowed. Failure of any test, by any sample, shall be cause for rejection of the lot represented.

TABLE V. Sampling.

Containers in a lot	Number of containers to sample
3 to 150	3
151 to 1,200	5
1,201 to 7,000	8
7,001 to 20,000	10
20,001 to 35,000	15
over 35,000	20

4.5 Test methods and procedures.

4.5.1 Form. Tumble by hand or mechanically the container with the representative sample for five minutes. Then with a scoop, place a few grams of magnesium-aluminum powder

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to form a thin layer on a microscopic slide and examine under a 20 to 100 power microscope to visually verify granular characteristics.

4.5.2 Chemical composition.

4.5.2.1 Aluminum.

4.5.2.1.1 Aluminum, Magnesium, Iron as Fe and Zinc as Zn (Inductively Coupled Plasma (ICP) method, preferred).

4.5.2.1.1.1 Reagents. Trace metal grade acids shall be used for sample dissolution. Unless otherwise noted, references to water shall mean laboratory accepted deionized water. It is advisable to use commercially available stock solutions with certified concentrations traceable to primary standards (National Institute of Standards and Technology or international measurement standards). Stock solutions shall be replaced after one year or in accordance with manufacturer recommendations. Alternately, stock solutions may be prepared in-house from high purity metals or their salts. The maximum recommended shelf life is one year from date of initial preparation. Recommended stock concentrations as follows:

- a. Hydrochloric Acid (HCl), concentrated, 36.5 – 38%
- b. Certified Al stock solution shall contain 10,000 µg/mL Al in 5.00% HCl
- c. Certified Mg stock solution shall contain 10,000 µg/mL Mg in 5.00% HCl
- d. Certified Zn stock solution shall contain 20 µg/mL Zn in 2.00% HCl
- e. Certified Fe stock solution shall contain 200 µg/mL Fe in 2.00% HCl
- f. De-ionized water.

4.5.2.1.1.2 Sample preparation.

- a. Weigh 200 mg (+/- 20 mg) of sample to the nearest 1 mg and transfer into a 150 mL beaker.
- b. Add 10 mL of water.
- c. Add 10 mL of 1:1 HCl and cover with watch glass.
- d. Allow reaction to complete and cool to ambient temperature.
- e. Transfer into 100 mL volumetric flask.
- f. Rinse inner surface of watch glass and inner walls of beaker with water into flask.
- g. Fill flask with water to the 100 mL mark and cap. Mix thoroughly.
- h. Pipette 5 mL of the above solution into a 50 mL volumetric flask. Fill to the mark with water. This gives the 10-fold dilute sample solution for Al and Mg determination.

4.5.2.1.1.3 Standard preparation for Type A.

- a. Middle standard (for Zn and Fe determination)
 1. Add stepwise to 100 mL volumetric flask:
 - i. 8 mL of 10,000 µg/mL Al solution
 - ii. 8 mL of 10,000 µg/mL Mg solution

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- iii. 8 mL of 20 $\mu\text{g/mL}$ Zn solution
 - iv. 7 mL of 200 $\mu\text{g/mL}$ Fe solution
 2. Fill to mark with water and cap. Mix thoroughly.
 - b. Dilute middle standard (for Al and Mg determination)
 1. Pipette 5 mL from middle standard solution above into 50 mL volumetric flask
 2. Fill to mark with water and cap. Mix thoroughly.
 - c. High standard (for Zn and Fe determination)
 1. Add stepwise to 100mL volumetric flask:
 - i. 12mL of 10,000 $\mu\text{g/mL}$ Al solution
 - ii. 12mL of 10,000 $\mu\text{g/mL}$ Mg solution
 - iii. 15mL of 20 $\mu\text{g/mL}$ Zn solution
 - iv. 9mL of 200 $\mu\text{g/mL}$ Fe solution
 2. Fill to mark with water and cap. Mix thoroughly.
 - d. Dilute high standard (for Al and Mg determination)
 1. Pipette 5mL from high standard solution above into 50 mL volumetric flask
 2. Fill to mark with water and cap. Mix thoroughly.
 - e. Prepare blank by pipetting 2 mL of HCl into 100 mL volumetric flask and dilute with water to the 100 mL mark.
 - f. Type A (for Zn and Fe determination)
 1. Middle standard shall contain: 800 $\mu\text{g/mL}$ Al, 800 $\mu\text{g/mL}$ Mg, 1.6 $\mu\text{g/mL}$ Zn, 14 $\mu\text{g/mL}$ Fe
 2. High standard shall contain: 1,200 $\mu\text{g/mL}$ Al, 1,200 $\mu\text{g/mL}$ Mg, 3 $\mu\text{g/mL}$ Zn, 18 $\mu\text{g/mL}$ Fe
 - g. Type A dilute (for Al and Mg determination)
 1. Middle standard shall contain: 80 $\mu\text{g/mL}$ Al, 80 $\mu\text{g/mL}$ Mg, 0.16 $\mu\text{g/mL}$ Zn, 1.4 $\mu\text{g/mL}$ Fe
 2. High standard shall contain: 120 $\mu\text{g/mL}$ Al, 120 $\mu\text{g/mL}$ Mg, 0.3 $\mu\text{g/mL}$ Zn, 1.8 $\mu\text{g/mL}$ Fe
- 4.5.2.1.1.4 Standard preparation for Type B.
- a. Middle standard (for Zn and Fe determination)
 1. Add stepwise to 100mL volumetric flask:
 - i. 6mL of 10,000 $\mu\text{g/mL}$ Al solution
 - ii. 12mL of 10,000 $\mu\text{g/mL}$ Mg solution
 - iii. 8mL of 20 $\mu\text{g/mL}$ Zn solution
 - iv. 7mL of 200 $\mu\text{g/mL}$ Fe solution
 2. Fill to mark with water and cap. Mix thoroughly.
 - b. Dilute middle standard (for Al and Mg determination)

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1. Pipette 5mL from mid standard solution above into 50 mL volumetric flask
 2. Fill to mark with water and cap. Mix thoroughly.
- c. High standard (for Zn and Fe determination)
1. Add stepwise to 100mL volumetric flask:
 - i. 8mL of 10,000 $\mu\text{g/mL}$ Al solution
 - ii. 14mL of 10,000 $\mu\text{g/mL}$ Mg solution
 - iii. 15mL of 20 $\mu\text{g/mL}$ Zn solution
 - iv. 9mL of 200 $\mu\text{g/mL}$ Fe solution
 2. Fill to mark with water and cap. Mix thoroughly.
- d. Dilute high standard (for Al and Mg determination)
1. Pipette 5mL from mid standard solution above into 50 mL volumetric flask
 2. Fill to mark with water and cap. Mix thoroughly.
 3. Prepare a sample blank by pipetting 2 milliliters of HCL into 100 milliliters volumetric flask and dilute with water to the 100 milliliters mark.
- e. Type B (for Zn and Fe determination)
1. Middle standard shall contain: 600 $\mu\text{g/mL}$ Al, 1200 $\mu\text{g/mL}$ Mg, 1.6 $\mu\text{g/mL}$ Zn, 14 $\mu\text{g/mL}$ Fe
 2. High standard shall contain: 800 $\mu\text{g/mL}$ Al, 1400 $\mu\text{g/mL}$ Mg, 3 $\mu\text{g/mL}$ Zn, 18 $\mu\text{g/mL}$ Fe
- f. Type B dilute (for Al and Mg determination)
1. Middle standard shall contain: 60 $\mu\text{g/mL}$ Al, 120 $\mu\text{g/mL}$ Mg, 0.16 $\mu\text{g/mL}$ Zn, 1.4 $\mu\text{g/mL}$ Fe
 2. High standard shall contain: 80 $\mu\text{g/mL}$ Al, 140 $\mu\text{g/mL}$ Mg, 0.3 $\mu\text{g/mL}$ Zn, 1.8 $\mu\text{g/mL}$ Fe

4.5.2.1.1.5 Preparation of calibration curve.

- a. Emission intensities shall be measured as follows:
1. Al emission intensity shall be measured using radial view at 308.215nm.
 2. Mg emission intensity shall be measured using radial view at 279.806nm.
 3. Fe emission intensity shall be measured using radial view at 259.940nm.
 4. Zn emission intensity shall be measured using radial view at 213.856nm.
- b. Aspirate standard solutions into ICP spectrometer in order of increasing concentration. Between aspirating each standard solution, aspirate the blank solution sufficiently to prevent cross-contamination.
- c. Take emission measurements for each element of interest and generate a calibration curve utilizing the instrument's software. Repeat the calibration if the correlation determination (R2) for each element is < 0.999 .

4.5.2.1.1.6 Sample analysis.

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- a. Aspirate the starting sample into the ICP spectrometer and record the emission measurement for Zn and Fe.
- b. Aspirate the 10-fold dilute solution into the ICP spectrometer and record the emission measurement for Al and Mg.
- c. Software generated concentrations for each element shall be inserted into the formula below as A_1 .
- d. Aspirate blank solution and insert into formula below as A_2 .

$$X = 100 \cdot (A_1 - A_2)/M$$

Where: X = content of the element of interest in sample [% (mass fraction)]

A_1 = amount of the element of interest detected in the sample, g

A_2 = amount of element of interest detected in the blank test solution, g

M = amount of sample weighed out, g

4.5.2.1.2 Aluminum (alternate method).

4.5.2.1.2.1 Preparation of solutions.

- a. 8-Hydroxyquinoline solution. Dissolve five grams of 8-Hydroxyquinoline in 10 milliliters of glacial acetic acid. The solution shall be diluted to 100 milliliters, with distilled water and filtered if necessary.
- b. Bromophenol blue indicator. Dissolve 0.100 grams of bromophenol blue powder in 20 milliliters of 95 percent ethyl alcohol and dilute to 100 milliliters with distilled water.

4.5.2.1.2.2 Procedure. Transfer an accurately weighed sample 1.000 ± 0.0500 grams of Type A alloy or 0.7700 ± 0.0300 grams of Type B alloy to a 250 milliliter beaker. Add ten milliliters of distilled water to the sample beaker. The beaker shall be covered with a watch glass and 20 milliliters of concentrated HCl shall be added dropwise through the opening at the lip of the beaker. After the reaction in the beaker has subsided, the solution shall be heated to boiling to assure complete solution. The watch glass shall be washed thoroughly with distilled water and the washings drained back into the beaker. The solution shall be filtered through a No. 41 Whatman, or equivalent, filter paper with the filtrate caught in an accurately calibrated, 1 liter volumetric flask. The filter paper shall be washed thoroughly with hot distilled water and the solution made up to the 1 liter volume with the distilled water. Mix by inverting flask five (5) or more times. A 100 milliliter aliquot shall be transferred to a 250 milliliter beaker using an accurately calibrated buret or pipet. Three drops of bromophenol blue indicator shall be added to the solution neutralized by adding dropwise and with constant stirring, a filtered 1:1 NH_4OH solution until the yellow color turns to blue (see 4.5.2.4.2). Two drops of 1:1 NH_4OH solution shall then be added to the excess solution. The solution shall be heated to boiling and 18 milliliters of the 8-hydroxyquinoline solution added in small portions, stirring after each addition to dissolve the precipitate that forms. Any precipitate remaining after the completion of the addition of the reagent shall be disregarded. The solution shall be heated to boiling and 40 milliliters of 2 molar (M) ammonium acetate solution (154 grams per liter) added dropwise with

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vigorous stirring and then placed in a steam bath for 30 minutes. The solution shall be filtered with a suction through a tared, fine porosity, fritted glass bottomed crucible. The precipitate shall be washed eight times with 15 milliliter portion of distilled water, releasing the suction before each addition and allowing the precipitate to remain in contact with the wash water for at least one minute prior to the application of suction. The precipitate shall be dried for 3 hours in an oven at 120° to 140°C, cooled in a desiccator and weighed as aluminum hydroxyquinoline, $\text{Al}(\text{C}_9\text{H}_6\text{ON})_3$. The percent aluminum shall be calculated as follows:

$$\text{Percent aluminum} = \frac{5.87 \cdot (A - (0.0874 \cdot B \cdot W))}{W}$$

Where: A = weight of precipitate, g

B = percent iron (from 4.5.2.5)

W = weight of sample contained in the aliquot, g

4.5.2.2 Magnesium.

4.5.2.2.1 Magnesium (ICP method) (preferred). See procedure described in 4.5.2.1.1.

4.5.2.2.2 Magnesium (alternate method).

4.5.2.2.2.1 Reagents.

- a. Bromophenol blue indicator.
- b. 1:1 NH_4OH solution.
- c. Ammonium chloride.
- d. Glacial acetic acid
- e. Ammonium benzoate solution
- f. Ammonium benzoate
- g. 8-hydroxyquinoline solution
- h. 1:100 NH_4OH solution

4.5.2.2.2.2 Procedure. Transfer a 100 milliliter aliquot of the solution, prepared in accordance with 4.5.2.1.2.2, to a 400 milliliter beaker by means of an accurately calibrated buret or pipet. Add three drops of bromophenol blue indicator and neutralize the solution with filtered 1:1 NH_4OH solution until the yellow color changes to blue. Add 1 gram of ammonium chloride and 1 milliliter of glacial acetic acid to the solution. Add 20 milliliters of hot 10 percent, ammonium benzoate solution slowly while stirring. Heat on a hot plate and boil gently for 5 minutes. Filter through a 11 centimeter Whatman No. 41, or equivalent, filter paper catching the filtrate in a 600 milliliter beaker. Wash the precipitate 10 times with a hot wash solution containing 1 gram of ammonium benzoate and 2 milliliters of glacial acetic acid per 100 milliliter of solution. Any crystallization that occurs in the filtrate as it cools shall be disregarded. Make the combined filtrate and washing alkaline to phenolphthalein with filtered concentrated NH_4OH solution. Add 5 milliliter NH_4OH solution in excess. Heat to 60° to 70°C, and add 15 milliliters of 8-hydroxyquinoline solution rapidly by drops from a buret. Stir the liquid vigorously with a thermometer keeping the temperature of the solution at 60° to 70°C during

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titration. Heat almost to boiling with frequent stirring and transfer to a steam bath for 20 minutes. Filter by means of a tared, fine porosity, sintered glass bottom crucible. Disconnect the suction and apply 10 milliliters of warm 1:100 NH₄OH wash solution to the precipitate with a wash bottle agitating the precipitate as much as possible with the force of the stream of wash solution. Apply the suction until the precipitate remains only moist enough to prevent cracking. Repeat washing with 10 milliliters of warm 1:100 NH₄OH solution and then suctioning seven (7) times and finally wash with two 10 milliliter portions of cold water. Dry to constant weight in an oven at 155° to 160°C. Cool in a desiccator and weigh. The percent magnesium shall be calculated as follows:

$$\text{Percent magnesium} = \frac{7.78 * A}{W}$$

Where: A = Weight of precipitate, g

W = Weight of sample contained in the aliquot, g

4.5.2.3 Total magnesium and aluminum. Add the percentages of aluminum and magnesium as determined in 4.5.2.1 and 4.5.2.2. Verify conformance with requirement.

4.5.2.4 Silicon as Si.

4.5.2.4.1 Silicon as Si (photometric method, preferred). Verify the silicon content by photometric examination (see 6.6).

4.5.2.4.1.1 Reagents.

- a. Aluminum nitrate solution (Al(NO₃)₃) – Transfer 1.0 g of aluminum (99.9% purity or higher), low-silicon, to a 250-mL nickel beaker. Add 100 mL of NaOH solution and cover. Allow to react without applying heat until the reaction subsides, and then warm gently to complete the dissolution. Cool, and transfer to a 400 mL beaker containing 125 mL of HNO₃ (1:1). Cover and warm gently until the salts dissolve and the solution becomes clear. Cool, transfer to a 250 mL volumetric flask, dilute with distilled water to volume, and mix by inverting flask five (5) or more times.
- b. Ammonium molybdate solution (100 g/L) – Dissolve 100 g of ammonium molybdate tetrahydrate ((NH₄)₆Mo₇O₂₄•4H₂O) in distilled water and dilute to 1 L in a volumetric flask. Store in a plastic bottle. Before using, filter through a plastic funnel and Whatman 41 filter paper or equivalent. If a precipitate forms on standing, the solution shall be discarded.
- c. Silicon, standard solution (1 mL = 0.05 mg Si) – Fuse 0.1070 g of anhydrous silicon dioxide (SiO₂) with 1.0 g of sodium carbonate (Na₂CO₃) in a covered platinum crucible. Cool, dissolve the melt completely in distilled water in a plastic beaker, cover, and heat on a steam bath for ½ to 1 hour. Cool, transfer to a 1 L volumetric flask, dilute to volume, and mix by inverting flask five (5) or more times. Store the

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solution in a plastic bottle. If anhydrous SiO_2 is not available, the weight shall be adjusted according to the actual silicon content of the SiO_2 used, as determined by gravimetric analysis.

- d. Sodium hydroxide solution (NaOH) (300 g/L) – Dissolve 300 g of sodium hydroxide (NaOH) in about 600 mL of distilled water, using a nickel or stainless steel beaker. Cool and dilute to 1 L in a volumetric flask. Store in a plastic bottle.
- e. Nitric acid solution (HNO_3 (1:1)) – Thoroughly mix one part reagent-grade hydrogen nitrate (HNO_3 (aqueous) (approximately 70%)) with one part distilled water.

4.5.2.4.1.2 Preparation of calibration curve.

- a. Calibration solutions – Transfer 12.5 mL of $\text{Al}(\text{NO}_3)_3$ solution and 1.0 mL of HNO_3 (1:1) to seven 100 mL beakers. Transfer 1.0, 2.0, 4.0, 8.0, 12.0, 16.0, and 20.0 mL of silicon solution (1 mL = 0.05 mg Si) to each of the 100 mL beakers. Dilute with distilled water to 80 mL and mix.
- b. Color development – Using a pH meter, check the pH of each of the solutions. If the pH is not within 1.1 to 1.3, adjust it to this range using HNO_3 (1:1) or NaOH solution as required. Add 10 mL of ammonium molybdate solution, transfer the solution to a 100 mL volumetric flask, dilute to volume with distilled water, and mix. Let stand at least 5 minutes but no longer than 20 minutes before taking the photometric reading.
- c. Reference solution – Transfer 12.5 mL of $\text{Al}(\text{NO}_3)_3$ solution and 1 mL of HNO_3 (1:1) to a 100 mL beaker, dilute with distilled water to 80 mL and mix.
- d. Color development – Using a pH meter, check the pH of the solution. If the pH is not within 1.1 to 1.3, adjust it to this range using HNO_3 (1:1) or NaOH solution as required. Add 10 mL of ammonium molybdate solution, transfer the solution to a 100 mL volumetric flask, dilute to volume with distilled water, and mix. Let stand at least 5 minutes but no longer than 20 minutes before taking the photometric reading.
- e. Photometry – Transfer a suitable portion of the reference solution to an absorption cell with a 2-centimeter light path and adjust the photometer to the initial setting, using a light band centered at approximately 400 nanometers. While maintaining this adjustment, take the photometric readings of the calibration solutions.
- f. Calibration curve – Plot the photometric readings of the calibration solutions against milligrams of silicon per 100 mL of solution.

4.5.2.4.1.3 Sample analysis.

- a. Test solution
 - 1. Transfer 0.10 g of the sample, weighed to the nearest 0.1 mg, to a 50 mL nickel crucible, add 10.00 mL of NaOH solution, and cover. Allow to react without applying heat until the reaction subsides, wash down the cover and sides

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of the crucible with a minimum amount of distilled water, and boil gently until the reaction is complete and the silicon completely oxidized (see 6.6.1). Cool, dilute to approximately 30 mL with distilled water, and transfer quantitatively to a 150 mL beaker containing 12.5 mL of HNO₃ (1:1). Cover and warm gently until the solution becomes clear (see 6.6.2-3). Cool, transfer to a 100 mL volumetric flask, dilute to volume with distilled water, and mix by inverting flask five (5) or more times until solution is visually homogenous.

2. Transfer a 50 mL aliquot to a 100 mL beaker, add 1.0 mL of HNO₃ (1:1), dilute to approximately 80 mL, and mix by inverting flask five (5) or more times until solution is visually homogenous.

b. Reagent blank

1. Transfer 10.0 mL of NaOH solution to a 100 mL volumetric flask containing 11.5 mL of HNO₃ (1:1). Dilute to approximately 50 mL, cool, dilute to volume with distilled water, and mix. Transfer a 50 mL aliquot to a 100 mL beaker, add 1 mL of HNO₃ (1:1), dilute to 80 mL, and mix by inverting flask five (5) or more times until solution is visually homogenous.

2. To the remaining portion of the reagent blank add 1 mL of HNO₃ (1:1), dilute to volume with distilled water, and mix. This portion is used as the reagent blank for the reference solution in 4.5.2.4.1.3.d.

c. Color development – Using a pH meter, check the pH of the solution. If the pH is not within 1.1 to 1.3, adjust it to this range using HNO₃ (1:1) or NaOH solution as required. Add 10 mL of ammonium molybdate solution, transfer the solution to a 100 mL volumetric flask, dilute to volume, and mix. Let stand at least 5 minutes but no longer than 20 minutes before taking the photometric reading.

d. Reference solution – To the remaining portion of the test solution add 1 ml of HNO₃ (1:1), dilute to volume, and mix. Take the photometric reading of the reference solution as directed in 4.5.2.4.1.2.e using the reagent blank obtained in sample analysis paragraph 4.5.2.4.1.3.b.2 for the initial setting of the photometer.

4.5.2.4.1.4 Calculation. Convert the photometric readings of the test and reference solutions to milligrams of silicon by means of the calibration curve. Calculate the percentage of silicon as follows:

$$\text{Percent silicon} = \frac{(A - B)}{(C \times 10)}$$

Where:

A = silicon found in 100 mL of the final solution, mg

B = reference solution correction, expressed as milligrams of silicon

C = sample represented in 100 mL of the final solution, g

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4.5.2.4.2 Silicon as Si (alternate method). Place a weighed portion of magnesium-aluminum powder approximately 2 grams in a beaker. Cautiously add 70 milliliters of a solution made by mixing 475 milliliters of water, 125 milliliters of 95 percent H₂SO₄, 200 milliliters of 70 percent HNO₃, and 200 milliliters of 38 percent HCl. Evaporate until heavy fumes of SO₃ have been evolved for a few minutes. Cool and add 10 milliliters of 50 percent H₂SO₄. Dilute to 150 milliliters with hot water, heat until salts are dissolved and transfer the insoluble residue to a paper filter. Wash the residue thoroughly with hot water. Retain the filtrate and washings for use in determination of iron (see 4.5.2.5.2). Ignite the residue in a platinum crucible, cool, and weigh. Moisten the residue with a few drops of 50 percent H₂SO₄ and add 2 milliliters of 47 percent HF. Evaporate to dryness, ignite, cool, and weigh. Calculate the loss in weight, which represents silicon dioxide, to percent silicon.

$$\text{Percent silicon} = \frac{46.7 * (W - A)}{W}$$

Where: W = original weight of sample, g
A = weight of sample after ignition, g

4.5.2.4.3 Silicon as Si (ICP method) (alternate method). Determine silicon content in accordance with ASTM E 3061.

4.5.2.5 Iron as Fe.

4.5.2.5.1 Iron as Fe (ICP method) (preferred). See procedure described in 4.5.2.1.1.

4.5.2.5.2 Iron as Fe (alternate method). Pass H₂S gas for 20 minutes through the combined filtrate and washings from the determination of silicon (see 4.5.2.4.2). Filter and wash the precipitate with H₂S water acidified with 1 percent H₂SO₄. Boil the combined filtrate and washings vigorously for 25 to 30 minutes to remove the H₂S. Cool the solution and titrate with 0.1N KMnO₄ solution. Retain the solution after titration for the determination of zinc (see 4.5.2.6.2). Calculate percent iron as follows:

$$\text{Percent iron} = \frac{55.8 * V * N}{W}$$

Where: V = volume of standard solution KMnO₄ solution used in titration, mL
N = normality of KMnO₄ solution used
W = original weight of sample measured in 4.5.2.4.3, g

4.5.2.6 Zinc as Zn.

4.5.2.6.1 Zinc as Zn (ICP method) (preferred). See procedure described in 4.5.2.1.1.

4.5.2.6.2 Zinc as Zn (alternate method). Using the entire solution retained from the iron determination (see 4.5.2.5.2), add 25 milliliters of 25 percent tartaric acid solution and dilute to 250 milliliters. Neutralize with 1:1 NH₄OH solution using methyl red indicator. Add 25

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milliliters of a solution of ammonium formate prepared by diluting 200 milliliters of 90 percent formic acid to 970 milliliters of with water and adding 30 milliliters of 28 percent NH_4OH solution. Heat the solution almost to boiling and pass in a rapid stream of H_2S for 30 minutes. Warm the solution until the precipitate has coagulated. Filter and wash the precipitate with a solution prepared by diluting to 1 liter, 25 milliliter of the ammonium formate solution described herein, and saturating it with H_2S . Dissolve the precipitate on the paper by means of a hot 10 percent HCl solution and wash thoroughly. Boil the filtrate and washings for 5 minutes. Cool to some extent, add 5 milliliters of a 25 percent solution of tartaric acid and make just alkaline to methyl red with NH_4OH . Dilute to 100 milliliters and heat to almost boiling. Pass H_2S through the solution for 3 minutes, add 10 milliliters of the undiluted ammonium formate solution as originally prepared, and continue to pass H_2S through the solution for 5 minutes and any additional time required to coagulate the precipitate. Filter and wash with diluted ammonium formate solution. Ignite the precipitate at approximately 700°C in a tared porcelain crucible. Cool and weigh as zinc oxide. Calculate percent zinc as follows:

$$\text{Percent zinc} = \frac{80.3 * B}{W}$$

Where: B = weight of precipitate, g

W = original weight of sample measured in 4.5.2.4.3, g

4.5.2.7 Other metals. If the presence of metals other than those determined in 4.5.2.1 through 4.5.2.6 is indicated, standard procedures for the analysis of such metals shall be used and their percentage determined.

4.5.3 Moisture. Transfer a portion of approximately 5 grams of the sample to a tared dish and heat in an oven for three hours at 105°C . Remove the sample from the oven and place into a desiccator at room temperature to cool. Reweigh sample and calculate the loss in weight as percent moisture as follows:

$$\text{Percent moisture} = \frac{A * 100}{B}$$

Where: A = loss in weight, g

B = weight of sample, g

4.5.4 Grease and fats. Extract a weighed portion of approximately 20 grams of magnesium-aluminum powder with hexanes or diethyl ether in a Soxhlet or similar extractor using a tared flask. When extraction is complete, evaporate the hexanes or diethyl ether and dry the flask and contents at 90°C to constant weight. Cool in a desiccator and weigh. Calculate the weight as percent grease and fats.

4.5.5 Grit. Transfer a weighed portion of approximately 5 grams of the sample to a 250 milliliter beaker. Add cautiously 40 milliliters of a solution made by mixing 10 milliliters of 70% HNO_3 and 30 milliliters of 38% HCl . Boil for 15 minutes. Dilute to 200 milliliters with hot water, filter and wash the insoluble material with hot water. Ignite the residue gently in a crucible, transfer it to a beaker and add 10 milliliters of 50% solution of NaOH . Boil for 10

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minutes, cool, and dilute to 100 milliliters with hot water. Filter and wash with hot water until the washings are free of alkali. Ignite the residue in a tared crucible, cool and weigh. Calculate the increase in weight as percent grit.

4.5.6 Oxides as Al₂O₃. From 100 percent, subtract the sum of the percentages of metals, moisture, grease and fats and grit. The remainder shall be considered to be the percent combined oxygen. Calculate to percent aluminum oxide as follows:

$$\text{Percent aluminum oxide} = 2.12 * \text{percent oxygen}$$

4.5.7 Granulation. Place a weighed portion of approximately 50 grams of the magnesium-aluminum powder on the top sieve of a nest of sieves assembled as specified in Table II, properly superimposed and assembled with a cover and a bottom pan. Shake for 30 minutes by means of a mechanical shaker geared to produce 268 to 315 gyrations and 150 ± 10 taps of the striker per minute. Weigh the material remaining on each sieve and bottom pan and calculate the percent passing through each sieve (see 6.5).

4.5.8 Apparent density (Type A only). Determine the apparent density of the magnesium-aluminum powder in accordance with the method described in ASTM B329. Make 3 separate determinations on each sample, and calculate the mean apparent density value of the powder.

4.5.9 Workmanship. Take approximately 200 to 300 grams of the magnesium-aluminum powder and spread out on a clean sheet of white paper. The sample shall be examined with unaided eyes by personnel with normal vision or normal corrected vision. The sample shall be uniform in quality and free from lumps, grit, visible impurities, foreign matter or other defects that would render the material unsuitable for the intended use. Failure to meet the workmanship inspection and/or visual inspection of workmanship during the test methods of 4.5 shall result in lot rejection.

5. PACKAGING

5.1 Packaging. For acquisition purposes, the packaging requirements shall be as specified in the contract or order (see 6.2). When packaging of materiel is to be performed by DoD or in-house contractor personnel, these personnel need to contact the responsible packaging activity to ascertain packaging requirements. Packaging requirements are maintained by the Inventory Control Point's packaging activities within the Military Service or Defense Agency, or within the military service's system commands. Packaging data retrieval is available from the managing Military Department's or Defense Agency's automated packaging files, CD-ROM products, or by contacting the responsible packaging activity.

6. NOTES

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

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6.1 Intended use. Powdered magnesium-aluminum alloy is intended for use in ammunition.

6.2 Acquisition requirements. Acquisition documents should specify the following:

- a. Title, number and date of this specification
- b. Types required (see 1.2)
- c. Whether a first article sample is required (see 3.1.1)
- d. Packaging requirements (see 5.1)

6.3 Change history.

Specification Revision	ECP Number	ERR Release Date
JAN-M-454	-	21-Feb-1947
JAN-M-454 Amend 1	-	15-Feb-1952
MIL-DTL-454	R18Q2001	

6.4 Units.

μm = micrometer
 μg = microgram
 g = gram
 mg = milligram
 mL = milliliter
 m = meter
 min = minimum
 max = maximum

6.5 Additional particle size requirements and analysis. Programs and suppliers may consider it desirable to impose additional requirements on particle size above and beyond those within the tolerances specified so as to specify further refinement of requirements or receive product optimized for their manufacturing processes. Furthermore, they may consider using alternate or additional methods and equipment to specify, characterize, and analyze size. Such analysis techniques may include:

- a. Gravitational sedimentation (See ASTM B761 and ISO 10076)
- b. Microscopy (see ISO 13322 and BS 3406-4)
- c. Laser diffraction (see ASTM B882 and ISO 13320)

Those who are interested in alternate characterization techniques are cautioned that analysis and results from one technique are generally not directly comparable to other techniques due to numerous factors including particle morphology, measurement technique, etc. Beneficial information may be found on alternate techniques from the National Institute of Standards and

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Technology (NIST) Special Publication 960-1, Recommended Practice Guide on Particle Size Characterization, available at <http://www.nist.gov>.

6.6 Silicon test method. The following notes are included for informational purposes regarding the silicon test method in 4.5.2.6. This test method originally appeared in ASTM E34.

6.6.1 Achieving complete dissolution. With certain alloys it may be necessary to evaporate the caustic solution to pastiness in order to completely dissolve the silicon. Care should be taken to avoid mechanical loss by spattering.

6.6.2 Solution cloudiness and excessive heating. Cloudiness (gray) of the solution at this point usually indicates incomplete dissolution of the silicon and another portion of the sample must be taken. Prolonging the heating after the NaOH attack will probably correct the difficulty. However, excessively prolonged heating will tend to cause precipitation of the manganese.

6.6.3 Brownish-yellow solutions. If the sample contains high manganese, the solution may be colored brownish-yellow due to suspended manganese dioxide (MnO_2). To bleach the color, add a saturated sodium sulfite (Na_2SO_3) solution drop-wise to the hot solution. Avoid any appreciable excess. Add potassium permanganate ($KMnO_4$) solution (3.2 g/L) until the solution is tinted pink. Finally add just enough oxalic acid (10 g/L) to destroy the pink color. Transfer the solution to a 100-mL volumetric flask, dilute to volume, and mix.

6.6.4 Correction for reference solution. For routine work, the reference solution correction may be omitted unless the test solution is colored yellow.

6.7 Subject term (key word) listing.

Ammunition
Chemical
Pyrotechnics
Incendiary

6.8 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.

Custodian:
Army – AR
Navy- OS
Air Force - 170

Preparing activity:
Army – AR
(Project: 1370-2020-001)

Review Activities:
Army – AR
Navy- OS
Air Force - 170

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NOTE: The activities listed above were interested in this document as of the date of this document. Since organizations and responsibilities can change, you should verify the currency of the information above using the ASSIST Online database at <https://assist.dla.mil>.