

MIL P-14067B (MU)  
10 March 1967  
SUPERSEDING  
MIL-P-14067A (MU)  
13 February 1963

MILITARY SPECIFICATION  
  
POWDERS, METAL  
(FOR USE IN AMMUNITION)

1. SCOPE

1.1 This specification covers three types of atomized metal powders and one type of ellipsoidal metal powder. (See 6.3 or 6.5).

1.2 Classification . - The metal powders shall be of the following types as specified (See 6.1 and 6.2):

Type I - Magnesium Powder, Atomized.  
Type II - Aluminum Powder, Atomized.  
Type III - Magnesium-Aluminum Alloy Powder 65/35, Atomized.  
Type IV - Magnesium Powder, Ellipsoidal.

2. APPLICABLE DOCUMENTS

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

SPECIFICATIONS

FEDERAL

UU-P-236 - Paper, Filter, Analytical.

STANDARDS

MILITARY

MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes (ABC-STD-105)  
MIL-STD-109 - Inspection Terms and Definitions.  
MIL-STD-129 - Marking for Shipment and Storage.  
MIL-STD-1233 - Procedures for Determining Particle Size, Particle Size Distribution and Packed Density of Powdered Materials.

FSC: 6810

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MIL-STD-1234 - Pyrotechnics, Sampling, Inspection and  
Testing.

## PUBLICATIONS

"American Society for Testing and Materials.

ASTM Designation E 34-58 - Chemical Analysis of Aluminum  
and Aluminum Base Alloys.

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

(Copies of specifications, standards, drawings and publications required by contractors  
in connection with specific procurement functions should be obtained from the procuring  
activity or as directed by the contracting officer.)

2.2 Other publications. - The following document forms a part of this  
specification to the extent specified herein. Unless otherwise indicated, the issue  
in effect on date of invitation for bids shall apply.

### Code of Federal Regulations

49 CFR 71-90 - Interstate Commerce Commission Rules and Regulations  
for the Transportation of Explosives and Other Dangerous Articles.

(The Interstate Commerce Commission Regulations are now a part of the Code of  
Federal Regulations (1949 Edition and revisions) available from the Superintendent  
of Documents, Government Printing Office, Washington 25, D. C. Orders for  
the above publication should cite "49 CFR 71-90 (latest revision).")

## 3. REQUIREMENTS

### 3.1 Shape

3.1.1 Type I, III, and IV. The shape of metal powders type I, III, and IV  
particles shall be spheroidal when tested as specified in 4.3.1 (see 6.3).

3.1.2 Type II. - The shape of metal powders atomized type II particles  
shall be spheroidal or granular when tested as specified in 4.3.1 (see 6.3).

3.1.3 Type IV. - The shape of the ellipsoidal magnesium shall be spheroidal  
with a maximum of 30 percent oblong shape particles when determined as specified  
in 4.3.17. (See 6.6).

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3.2 Chemical and physical properties. - The metal powders shall conform to the chemical and physical requirements as shown in Table I.

3.3 Granulation. - The metal powder shall conform to the granulation requirements as in Table II when tested as specified in 4.3.15.

3.4 Average particle diameter. The average particle diameter shall conform to the requirements in Table III when tested as specified in 4.4.13.

#### 4. QUALITY ASSURANCE PROVISIONS

4.1 General quality assurance provisions. - The supplier is responsible for the performance of all requirements specified herein. Except as otherwise specified, the supplier may utilize his own or any other inspection facilities and services acceptable to the Government. Inspection records of the examinations and tests shall be kept complete and available to the Government as specified in the contract or order. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements. Reference shall be made to Standard MIL-STD-109 in order to define the terms used herein. Inspection shall be performed in accordance with this specification and other specifications referenced in any of the contractual documents.

4.1.1 Contractor quality assurance system. If the contractor desires to utilize a quality assurance system which is at variance with the quality assurance provisions of 4.2 and 4.3 and other documents referenced herein, he shall submit a written description of the system to the contracting officer for approval prior to initiation of production. It shall include a description covering controls for lot formation and identification, inspections to be performed, inspection stations, sampling procedures, methods of inspection and provisions for control and disposition of nonconforming material. The written description will be considered acceptable when, as a minimum, it provides the quality assurance required by the provisions of 4.2 and 4.3 and the other documents referenced herein. The contractor shall not be restricted to the inspection station or the method of inspection listed in this specification provided that an equivalent control is included in the approved quality assurance procedure. In cases of dispute as to whether certain procedures of the contractor's system provide equal assurance, the comparable procedure of this specification shall apply. The contractor shall notify the Government of, and obtain approval for, any change to the written procedure that affects the degree of assurance required by this specification or other documents referenced herein.

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TABLE I

Requirements

Test Paragraph

TYPES

I

II

III

IV

Magnesium  
Powder

Aluminum  
Powder

65/35  
Magnesium  
Aluminum  
Alloy Powder

Ellipsoidal  
Magnesium

Magnesium, percent minimum  
(min.)

98.0

.....

65 plus  
or minus 5

98.0

4.3.5

Aluminum, percent min.

.....

98.75

35 plus or  
minus 5

4.3.10

Volatile material at 105 degrees  
Cent. (°C) percent maximum

0.1

0.1

0.1

4.3.2

Oil and Grease percent (max.)

.....

0.2

0.2

.....

4.3.8

Zinc, percent, max.

1.5

0.3

...

0.5

4.3.6

Silicon percent, max.

...

0.25

0.2

.....

4.3.9

Alloy as Iron, percent, max.

0.5

0.55

0.1

0.05

4.3.4

Carbides percent, max

0.004

...

...

0.004

4.3.3

Other impurities, percent, max

0.3

...

...

1.0

4.3.8

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TABLE I (Continued)

<u>Requirements</u>	<u>TYPES</u>				<u>Test Paragraph</u>
	I	II	III	IV	
	Magnesium Powder	Aluminum Powder	65/35 Magnesium Aluminum Alloy Powder	Ellipsoidal Magnesium	

Total aluminum and magnesium percent min.	...	...	98.0	...	4.3.12
Apparent density gram (gm) per milliliter (ml) min.	0.95	1.0	1.0	0.95	4.3.14
Nitrogen, PPM, max	20.0	...	...	20.0	4.3.18

For Type III Magnesium/Aluminum used method 4.3.12.

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TABLE II GRANULATION

Percent Passing by Weight

Nominal mesh size 20/50 50/100 100/200 200/325

U.S. Standard Sieve No.

Type I Magnesium

16	100 min	....	....	....	....
20	95 min	95 min	....	....	....
30	70 min	85 min	95 min	100 min	....
40	....	....	....	....	....
50	20 max	40 max	70 min	90 min	....
60	10 max	25 max	....	....	....
80	....	....	....	....	....
100	....	5 max	40 max	50 min	....
140	....	....	20 max	....	....
200	....	....	5 max	30 max	99.5 min
325	....	....	....	5 max	60.0 min

Type II Aluminum

Percent Passing by Weight

200	....	....	....	99.5 min
325	....	....	....	60.0 min

Type III 65/35 Magnesium Aluminum

Percent Passing by Weight

30	....	....	100 min	....
50	....	....	90 min	....
100	....	....	50 min	....
200	....	....	30 max	99.5 min
325	....	....	5 max	60.0 min

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TABLE II GRANULATION (Continued)

Percent Retained On

Type IV Magnesium

U. S. Standard Sieve No.

20	.....	0.1 max
30	.....	7.0 max
40	.....	43-53
50	.....	40-56
60	.....	0-30

TABLE III. Average Particle Size, in Microns

Nominal mesh size Powder type	20/50	30/50	50/100	100/200	200/325
I	450 plus or minus 50	350 plus or minus 50	200 plus or minus 25	125 plus or minus 25	22 plus or minus 8
II	-	-	-	-	22 plus or minus 8
III	-	-	-	125 plus or minus 25	22 plus or minus 8
IV	-	350 plus or minus 50	-	-	-

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4.1.2 Submission of product. - At the time the completed lot of product is submitted to the Government for acceptance the contractor shall supply the following information accompanied by a certificate which attests that the information provided is correct and applicable to the product being submitted:

- a. A statement that the lot complies with all quality assurance provisions of the approved current written description of the system.
- b. Quantity of product inspected.
- c. Results obtained for all inspection performed.
- d. Specification number and date together with an identification and date of changes.
- e. Certificates of analysis on all material procured directly by the contractor when such material is controlled by Government specifications listed in any of the contractual documents.
- f. Quantity of items in the lot.
- g. Date submitted.

The certificate shall be signed by a responsible agent of the certifying organization and shall be accompanied by evidence of the agent's authority to bind his principal.

4.1.3 Government verification. - Using the contractor's written quality assurance procedure, when applicable, this detail specification and other contractual documents as a guide, the government inspector shall verify all quality assurance operations performed by the contractor. Verification shall be in accordance with a or b, below, as applicable, the decision being the responsibility of the procuring activity. In either case, the inspector shall also ascertain, prior to acceptance, that all quality assurance provisions of other specifications referenced in any of the contractual documents have been complied with. Deviations from prescribed or agreed upon procedures discovered by the government inspector shall be brought to the attention of the contractor. Disposition of the product and remedial action shall be as directed by the government inspector and, depending upon the nature of the deviation, may consist of lot rejection, screening, resampling, reinstruction of the suppliers employees or other appropriate action.



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Unless otherwise approved by the contracting officer, the inspection lot size of metal powders deliverable under the contract shall be not less than the smallest weekly estimate of quantities contractually scheduled for production during the contract period nor more than the largest quantity contractually scheduled for delivery during any month of the contract period. Inspection lots shall be homogeneous and of a size convenient to the contractor. The classification of defects shall be in accordance with Standard MIL-STD-105.

4.2.2 Examination. - Sampling plans and procedures for the following classifications of defects shall be in accordance with Standard MIL-STD-105 except that inspection for critical defects, when listed, shall be 100 percent. At the option of the procuring activity, AQL's and sampling plans may be applied to the individual characteristics listed using an AQL of 0.40 percent for each major defect and an AQL of 0.65 percent for each minor defect.

4.2.2.1 Drum sealed (see 5.1).

<u>Categories</u>	<u>Defects</u>	<u>Method of Inspection</u>	<u>Code No.</u>
Critical: None defined			
Major:	AQL 0.40 percent		
101.	Weight of contents. . . . .	Scale	01001
102.	Gasket missing or cover improperly closed . . . . .	Visual	01002
Minor:	AQL 0.65 percent		
201.	Marking misleading or unidentifiable	Visual	01003

4.2.3 Testing

4.2.3.1 Sampling lot. - A random sample of 8 containers shall be selected from each lot. When lots are comprised of 10 containers or less each container shall be sampled.

4.2.3.1.1 Preparation of composite for chemical test. - Approximately one ounce primary samples of material shall be removed from each of the ten containers in order to equal eight ounces, and if there are less than 8 containers, equal primary samples in sufficient quantity to equal 8 ounces, shall be removed from each container. The individual primary samples shall then be combined in order to form a homogeneous composite sample of eight ounces and subjected to the tests specified in 4.3. If the composite sample fails to comply with any of the requirements specified, the lot shall be rejected.

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4.2.3.1.2 Sampling for granulation and particle size. - Each container shall be rolled and tumbled for ten minutes or until the metal powder is completely mixed in the container. Then a sample of approximately 8 ounces shall be removed from the container with a sampling thief. The granulation and particle size shall be determined on the sample thus obtain. Each container shall be individually tested. If any sample fails to comply with the requirements of Table II and III the container shall be rejected.

4.3 Test methods and procedures. - The following test methods shall be as classified as major defects.

4.3.1 Shape. - Code number 02001. - A small portion of the sample shall be placed under a microscope and examined for conformance to paragraph 3.1.

4.3.2 Volatile material at 105°C. Code number 03001. - Approximately 5 gm. of the sample shall be weighed into a tared dish and heated in a oven for 3 hours at 105°C., then cooled and weighed. The loss in weight shall be calculated as percent volatile material at 105°C.

4.3.3 Carbides. - Code number 04001.

4.3.3.1 Apparatus. - Apparatus similar to that shown on figure 1 shall be used.

4.3.3.2 Reagent. - A 0.25 gm. portion of gelatin shall be dissolved in hot water. The solution shall be diluted to 500 ml. and 500 ml. of 95 percent ethyl alcohol and 1.25 gm. hydroxylamine hydrochloride shall be added.

4.3.3.3 Procedure. A weighed portion of 50 gm. of magnesium powder shall be added to the reaction flask (A). Approximately 50 gm. of cuprous chloride shall be transferred to test tube (B). Nitrogen shall be passed through the apparatus to remove the air and shall be continued throughout the determination. The test tube shall be lowered and 15 ml. of concentrated ammonium hydroxide ( $\text{NH}_4\text{OH}$ ) and 30 ml. of hydroxylamine hydrochloride reagent added to the reaction flask. The test tube shall be raised, the stopper inserted, 100 ml. of distilled water from dropping funnel (C) added the heater switched on. The mixture shall be heated in the reaction flask just to boiling. A red or pink coloration of the cuprous ammonium chloride solution in the test tube indicates that the magnesium sample contains more than 0.004 percent carbides. Should the cuprous ammonium chloride solution become blue, due to the introduction of air, it shall be decolorized with successive 5 ml. portions of hydroxylamine hydrochloride reagent until the blue color disappears.

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4.3.4 Alloyed iron as Fe. - Code number 06001. An accurately weighed portion of approximately 10 gm. of the sample shall be transferred to a 500 ml. flask. About 200 ml. of 1:5  $H_2SO_4$  shall be added slowly, to minimize effervescence and prevent spattering. The solution shall be allowed to stand until action ceases and all the magnesium has dissolved. The solution shall be titrated immediately with 0.02N  $KMnO_4$  solution. The percent alloyed iron shall be calculated as follows: (see 4.3.16).

$$\text{Percent alloyed iron as Fe} = \frac{5.584 \text{ VN}}{W}$$

where:

V = ml.  $KMnO_4$  required for sample.

N = normality of  $KMnO_4$  solution.

W = gm. of sample.

4.3.5 Free metallic magnesium (eudiometer method). Code number 07001. The free metallic magnesium shall be determined in accordance with method 412.1 of Standard MIL-STD-1234.

4.3.6 Zinc (see 4.3.16). Code number 08001. Ammonium Mercuric Thiocyanate Method or Zinc Oxide Method. The zinc content shall be determined in accordance with Method ASTM Designation E 34-58.

4.3.7 Other impurities. Code 09001. The sum of the percentages of volatile material, carbides, alloyed iron, free metallic magnesium and zinc shall be subtracted from 100 percent. The remainder shall be percentage of other impurities.

4.3.8 Oil and grease. - Code number 10001. A weighed portion of approximately 20. gm. of the sample shall be extracted with ether in a Soxhlet or similar extractor using a tared flask. The ether shall be evaporated when extraction is completed and the flask and contents shall be dried at  $90^\circ C$ . to constant weight, cooled in a desiccator, and weighed. A blank shall be run at the same time. The weight of residue shall be calculated to percent oil and grease, as follows:

$$\text{Percent oil and grease} = \frac{(A-B) 100}{W}$$

where:

A = weight of ether extract.

B = weight of blank.

C = weight of sample.

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4.3.9 Silicon as Si. - Code number 11001. The Silicon content shall be determined in accordance with the mixed acid method of ASTM designation E-34-58.

4.3.10 Free metallic aluminum (eudiometer method). - Code number 12001. The free metallic aluminum shall be determined as specified in method 412.1 of Standard MIL-STD-1234. Except that the reaction flask shall contain 100 ml. 10 percent sodium hydroxide solution saturated with hydrogen. At Standard conditions a 0.33 to 0.34 gms. sample shall be used and calculated as follows:

$$\text{Percent free metallic aluminum} = \frac{0.0288V (P_1 - P_2)}{W (273 \text{ plus } T)} \quad (0.275A \text{ plus } 1.28B)$$

where:

T = temperature of water jacket in °C.

V = volume of gas in buret, ml.

P<sub>1</sub> = barometric pressure, in mm. of mercury.

P<sub>2</sub> = vapor pressure of water at T, in mm. of mercury.

W = weight of sample, gm.

A = percent zinc.

B = percent silicon.

4.3.11 Test applicable to Type III, magnesium-aluminum alloy powdered 65/35, atomized.

4.3.11.1 Aluminum. - Code number 13001.

4.3.11.1.1 Preparation of solutions.

4.3.11.1.1.1 8-Hydroxyquinoline solution. - Five gm. of 8-hydroxyquinoline shall be dissolved in 10 ml. of glacial acetic acid. The solution shall be diluted to 100 ml. with water and filtered, if necessary.

4.3.11.1.1.2 Bromphenol blue indicator. - Into 20 ml. of 95 percent ethyl alcohol 0.100 gm. of bromphenol blue powder shall be dissolved and diluted to 100 ml. with water.

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4.3.11.1.2 Procedure. An accurately weighed sample of 0.7700 plus or minus 0.0300 gm. of Type III alloy shall be transferred to a 250 ml. beaker. Ten ml. of water shall be added. The beaker shall be covered with a watch glass and 20 ml. of concentrated HCl shall be added dropwise through the opening at the lip of the beaker. After the reaction has subsided, the solution shall be heated to boiling to assure complete solution. The watch glass shall be washed thoroughly and the washings drained back into the beaker. The solution shall be filtered through a No. 41 Whatman, or equivalent, filter paper. The filtrate shall be caught in an accurately calibrated, 1-liter volumetric flask. The filter paper shall be washed thoroughly with hot water and the solution made up to volume with water. A 100 ml. aliquot shall be transferred to a 250 ml. beaker using an accurately calibrated buret or pipet. Three drops of bromphenol blue indicator shall be added and the solution neutralized by adding dropwise and with constant stirring, a filtered 1:1  $\text{NH}_4\text{OH}$  solution until the yellow color turns to blue. Two drops of  $\text{NH}_4\text{OH}$  solution shall then be added in excess. The solution shall be heated to boiling and 18 ml. 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 ml. of 2 molar (M) ammonium acetate solution (154 gm. per liter) added dropwise with vigorous stirring and then placed on a steam bath for 30 minutes. The solution shall be filtered with a suction through a tared, fine porosity, fritted glass bottom crucible. The precipitate shall be washed eight times with 15 ml. portion of water, releasing the suction before each addition and allowing the precipitate to remain in contact with the wash water for at least 1 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:

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

where:

A = gm. of precipitate.

B = percent iron.

W = gm. of sample contained in the aliquot.

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4.3.11.2 Magnesium. Code number 14001. An 100 ml aliquot of the solution, prepared as specified in 4.3.11.1.2, shall be transferred to a 400 ml beaker by means of an accurately calibrated buret or pipet. Three drops of bromphenol blue indicator shall be added and the solution neutralized with filtered 1:1  $\text{NH}_4\text{OH}$  solution until the yellow color changes to blue. One gm of ammonium chloride and 1 ml of glacial acetic acid shall be added to the solution. Twenty ml of hot 10 percent ammonium benzoate solution shall be added slowly while stirring. The solution shall be heated on a hot plate and boiled gently for 5 minutes. Then filtered through Whatman No. 41, or equivalent, filter paper catching the filtrate in a 600 ml beaker. The precipitate shall be washed 10 times with a hot wash solution containing 1 gm of ammonium benzoate and 2 ml of glacial acetic acid per 100 ml of solution. Any crystallization that occurs in the filtrate as it cools shall be disregarded. The combined filtrate and washing shall be made alkaline to phenolphthalein with filtered concentrated  $\text{NH}_4\text{OH}$  solution. Five ml of  $\text{NH}_4\text{OH}$  solution shall be added in excess. The solution shall be heated from  $60^\circ$  to  $70^\circ$  C and 15 ml of 8-hydroxyquinoline solution added rapidly drops from a buret. The liquid shall be stirred vigorously with a thermometer keeping the temperature of the solution at  $60^\circ$  to  $70^\circ$  C during addition. The solution shall be heated almost to boiling with frequent stirring and transferred to a steam bath for 20 minutes. The solution shall be filtered through a tared, fine porosity, sintered glass bottom crucible. The suction shall be disconnected and 10 ml of warm 1:10  $\text{NH}_4\text{OH}$  wash solution added to the precipitate with a wash bottle agitating the precipitate as much as possible with the force of the steam of wash solution. The suction shall be applied until the precipitate remains only moist enough to prevent cracking. This procedure shall be repeated 7 times finally washing with two 10 ml portions of cold water. The precipitate shall be dried to constant weight in an oven at  $155^\circ$  to  $160^\circ$  C., cooled in a desiccator, and weighed. The percent magnesium shall be calculated as follows:

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

where:

A = gram of precipitate

W = gram of sample contained in aliquot.

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4.3.12 Total aluminum and magnesium. Code number 15001. The percentages of aluminum and magnesium as determined in 4.3.11.1 and 4.3.11.2 shall be added to obtain the total aluminum and magnesium.

4.3.13 Determination of average particle diameter (all types). Code number 16001. - The average particle diameter of metal powders shall be determined by means of an instrument based on the air permeability principle such as the Fisher Sub Sieve Sizer or Picatinny Particle Sizer. The average particle size shall be determined in accordance with Standard MIL-STD-1233, Method 100 for the Fisher Sub Sieve Sizer and Method 200 for the Picatinny Arsenal Particle Sizer.

4.3.14 Apparent density (all types). Code number 17001. - A funnel having a stem of 1/4 inch internal diameter shall be cut so that the tip of the stem is just above the uppermost graduation of a tared 100 ml graduated cylinder. The cylinder shall be filled to the 100 ml mark by pouring small quantities of the sample on the side of the funnel and allowing it to fall by gravity through the stem. Jarring or tapping of the cylinder shall be avoided. The cylinder shall be weighed and the gain in weight divided by 100 shall be reported as the apparent density.

4.3.15 Granulation (all types). Code number 18001. - The granulation shall be determined in accordance with Method 201.1 of Standard MIL-STD-1234.

4.3.16 Spectrochemical Analysis (alternate method). A spectrochemical analysis shall be determined for alloyed iron, zinc, and silicon in accordance with Federal Standard, Federal Test Method Standard No. 151, method 112.

4.3.17 Ellipsoidal Type IV. Code Number 19001. Microscopic Analysis. One hundred particles shall be examined under a microscope with a magnification of 50. Each particle shall be counted individually and the percentage of oblong particles shall be determined.

4.3.18 Nitrogen. - Code number 20001. (Type I and IV). Weigh four grams of powder into 250 ml beakers. Add 25 ml water and cover with watch glasses. Add 20 ml concentrated sulfuric acid in small increments. When solution is completed, transfer to 100 ml volumetric flask and dilute to volume with ammonia free distilled water. After thoroughly mixing, pipette a 25 ml aliquot into the Kjeldahl flask. Add 150 ml ammonia free water and 35 ml 30 percent sodium hydroxide solution and connect to steam distillation assembly.



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Place the delivery tube from the condenser below the surface of 10 ml of N/50 sulfuric acid in 100 ml volumetric flask or 100 ml beaker. Distill at the rate of 3 to 5 ml per minute until the volume is approximately 90 ml. Transfer to 100 ml volumetric flask unless this was used to receive the distillate. Add 2 ml Nessler's (see Note 4) reagent and make to volume and mix. Wait 3 to 5 minutes and read on the photometer preferably using a cell provided 4 cm for the light path. If the Klett type instrument is available, use the blue filter. For the wave length selector type instruments, set the wave length to obtain the maximum absorbance. Run the blank in the same manner and subtract the blank reading from the sample. Obtain micrograms nitrogen from the calibration curve. Prepare the calibration curve to cover the range 0-80 micrograms nitrogen. Use ammonium chloride as the source of nitrogen.

#### Calculations

$$\text{PPM N}_2 = \frac{\text{Micrograms N}_2}{\text{Sample Weight (gr ams)} \times \text{Aliquot}}$$

#### NOTES:

Note 1) It is often difficult to remove the last traces of ammonia from the distillation equipment. This must be done by continuous distillation from alkaline aqueous solutions (make alkaline with sodium hydroxide) until approximately 80 ml of the distillate + 10 ml N/50 sulfuric acid + 2 ml Nessler's reagent made to 100 ml volume gives essentially the same reading as 10 ml N/50 sulfuric acid + 80 ml ammonia free distilled water + 2 ml Nessler's reagent made to 100 ml volume.

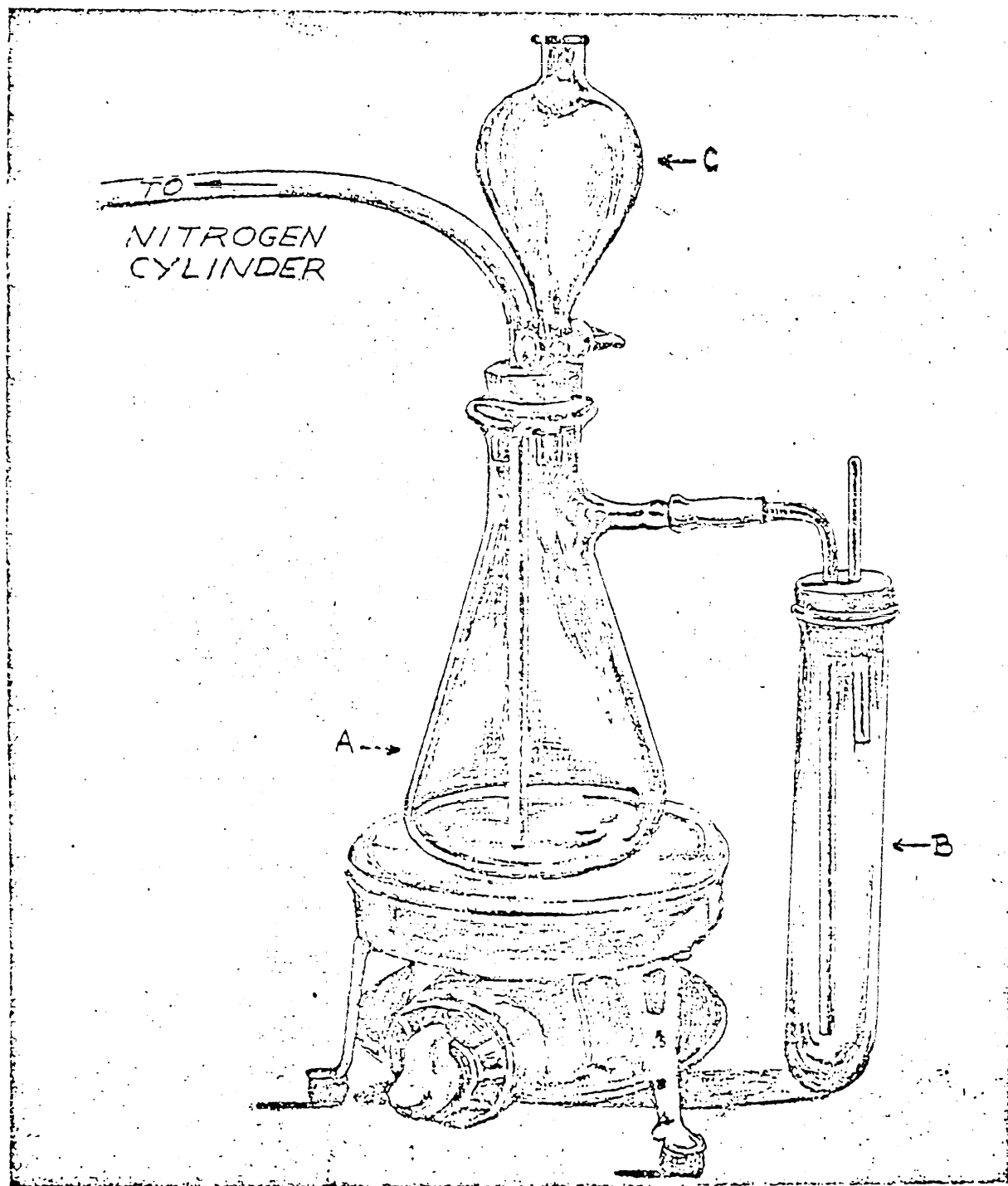
Note 2) The presence of ammonia fumes in the laboratory necessitate frequent blank checks.

Note 3) The ammonia free water is prepared by boiling distilled water made alkaline with sodium hydroxide. Portions of distillate are discarded until they cease to develop color with Nessler's reagent.

Note 4) Nessler's reagent - dissolve 50 g potassium iodide in the smallest possible quantity of cold water. Add a saturated solution of mercuric chloride until a faint show of excess is indicated. Add 400 ml of 50 percent KOH solution. After the solution has clarified by settling, make up to 1 liter with water, allow to settle and decant.



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APPARATUS FOR DETERMINATION OF CARBIDES IN MAGNESIUM

FIGURE 1

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## 5. PREPARATION FOR DELIVERY

5.1 Packing. - Unless otherwise specified on the contract or order, powder shall be shipped in unlined, full open head steel drums protected against corrosion and conforming to the requirements of ICC Specification 37A or B stipulated in the Code of Federal Regulations 49 CFR 71-90. Containers shall contain a maximum of 150 pounds. Each drum shall be provided with a tubular rubber cover gasket, firmly cemented in place. When the jackscrew is tightened, the rim shall be tapped with a mallet to insure complete and proper sealing of the gasket. If a drum is to be reused for the shipment of powder, new gaskets shall be used each time. In addition, each drum reused shall pass the tests and conditions for new drums.

5.2 Markings. - Marking to insure safe handling shall conform to the ICC Regulations stipulated in the Code of Federal Regulations 49 CFR 71-90 and in addition shipments shall be marked in accordance with Standard MIL-STD-129. Unless otherwise specified, each container shall be plainly marked with the following information completed:

- (1) Minimum percentage of free metallic magnesium or aluminum.
- (2) Type of powder (Type I, II, III, IV)
- (3) Nominal mesh size.

## 6. NOTES

6.1 Intended use. - These metal powders are intended for use in pyrotechnics compositions.

6.2 Ordering data. - Procurement documents should specify the following:

- (1) Title, number, and date of this publication.
- (2) Type of metal powder (see 1.2).
- (3) Granulation and average particle diameter required (see 3.3 and 3.4).

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6.3 "Atomization" is defined as the dispersion of a molten metal into a spray, the droplets of which are then allowed to freeze under the primary influence of surface tension. "Granular" is defined as particles that have approximately equi-dimensional nonspherical shapes.

6.4 Inspection code numbers. - The five digit code numbers assigned to the inspection herein are to facilitate future data collection and analysis by the Government.

6.5 Ellipsoidal particles are made by chipping magnesium ingots and then hammering the particles in a hammering machine.

6.6 An oblong particle is one that has a length/width ratio greater than 1.5.

Custodian:  
Army - MU

Preparing Activity:  
Army - MU

Project Number : 6810-A149

**INSTRUCTIONS:** In a continuing effort to make our standardization documents better, the DoD provides this form for use in submitting comments and suggestions for improvements. All users of military standardization documents are invited to provide suggestions. This form may be detached, folded along the lines indicated, taped along the loose edge (*DO NOT STAPLE*), and mailed. In block 5, be as specific as possible about particular problem areas such as wording which required interpretation, was too rigid, restrictive, loose, ambiguous, or was incompatible, and give proposed wording changes which would alleviate the problems. Enter in block 6 any remarks not related to a specific paragraph of the document. If block 7 is filled out, an acknowledgement will be mailed to you within 30 days to let you know that your comments were received and are being considered.

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1. DOCUMENT NUMBER

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3a. NAME OF SUBMITTING ORGANIZATION

4. TYPE OF ORGANIZATION (Mark one)

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b. ADDRESS (Street, City, State, ZIP Code)

5. PROBLEM AREAS

a. Paragraph Number and Wording:

b. Recommended Wording:

c. Reason/Rationale for Recommendation:

6. REMARKS

7a. NAME OF SUBMITTER (Last, First, MI) – Optional

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