

DETAIL SPECIFICATION

GRAPHITE, DRY

(For use in ammunition)

Reactivated after 7 May 2018 and may be used for new and existing

1. SCOPE

- 1.1 <u>Scope</u>. This specification covers the requirements, examinations and tests for commercial grade Graphite and is intended for use in ammunition.
 - 1.2 Classes. The graphite must be of the following grades (see Table I)

Grade I - Manufactured graphite used for lubrication
Grade II - Natural graphite used for lubrication
Grade III - Manufactured graphite used for glazing
Grade IV - Natural graphite used for glazing

Comments, suggestions, or questions on this document should be addressed to: Commander, US Army ARDEC, ATTN: RDAR-EIQ-SA, Picatinny Arsenal, New Jersey 07806-5000 or email 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 1376

<u>DISTRIBUTION STATEMENT A</u>. Approved for public release; distribution is unlimited

2. APPLICABLE DOCUMENTS

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

2.2 Government documents.

2.2.1 <u>Specification and standards</u>. The following specifications and standards 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.

DEPARTMENT OF DEFENSE STANDARDS

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

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

2.3 <u>Non-government publications</u>. 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 E11 - Standard Specification for Woven Wire Test Sieve Cloth and Test Sieves

(Copies of these documents are available from www.astm.org or ASTM International 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959)

2.4 <u>Order of precedence</u>. Unless otherwise specified in this document or in the contract, in the event of a conflict between the text of this document and 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 <u>Required inspections</u>.
- 3.1.1 <u>First article</u>. When specified (See 6.2), a sample shall be subjected to first article inspection in accordance with 4.3.
- 3.1.2 <u>Conformance</u>. A sample shall be subjected to conformance inspection in accordance with 4.4.
- 3.2 <u>Appearance (applicable to Grades II and IV only)</u>. Graphite of Grades II and IV shall appear steel gray or silver gray in color and shall have a metallic luster.
- 3.3 <u>Properties</u>. Graphite shall conform to the limits for the properties specified in Table I, when determined as specified in the applicable subparagraphs of 4.5.

Grade II Grade III Grade IV Grade I Paragraph **Properties** Moisture (max.) 0.20 0.50 0.50 0.50 4.5.2.1 Ash (max.) 0.60 6.0 0.60 6.0 4.5.2.2 Silica (max.) 1.0 2.75 4.5.2.3 Other grit None None None None 4.5.2.4 Acidity 4.5.2.5 None None None None Free sulfur (max.) 0.05 0.05 4.5.2.6 0.02Total Sulfur (max.) 0.20 0.50 0.50 4.5.2.7 Granulation: 4.5.2.8 Through Sieve No. 100 (149 65.0 4.5.2.8 . micron min.) Through Sieve No. 200 (74 96.0 96.0 4.5.2.8 micron min.) Through Sieve No. 325 (44 96.0 4.5.2.8 . micron min.)

TABLE I. Properties.

- 3.4 <u>Lubricating quality (applicable to Grades I and II only)</u>. When specified, Grades I and II graphite shall be tested for use as a pelletizing lubricant. The graphite shall be considered satisfactory if the machine works smoothly and easily, if the powder does not stick to any parts of the machine, and if the resulting pellets do not fall apart (see 6.2).
- 3.5 <u>Glazing quality (applicable to Grades III and IV only)</u>. When specified, Grades III and IV graphite shall be tested for use as a glazing compound. The graphite shall be considered

satisfactory if it is processed through the glazing equipment and provides a uniform coating. Additional requirements may be specified in the contract (see 6.2).

3.6 <u>Workmanship</u>. There shall be no foreign contaminants including dust, debris from machinery, or any other particle that is not intended to be in the material. Care shall be taken to protect the material and its components from moisture and adverse environmental effects. All required markings shall be correct, neat and legible.

4. VERIFICATION

TABLE II. Requirement/verification cross reference matrix.

Method of Verification		Classes of Verification						
1 - Analysis		A - First Article Inspection						
2 - Demonstration (end item test)		B - Conformance Inspection						
3 - Examination								
4 - Test								
Description	Section 3	Section 4	Verification Verification					
	Requirements	Verifications		Method Class		iss		
			1	2	3	4	A	В
First Article	3.1.1	4.3		X	X	X	X	
Conformance	3.1.2	4.4			X	X		X
Appearance	3.2	4.4.2.1			X		X	X
Properties	3.3	4.5.2				X	X	X
Moisture, Maximum	Table I	4.5.2.1				X	X	X
Ash, maximum (max.)	Table I	4.5.2.2				X	X	X
Silica, max.	Table I	4.5.2.3				X	X	X
Other grit	Table I	4.5.2.4				X	X	X
Acidity	Table I	4.5.2.5				X	X	X
Free sulfur, max	Table I	4.5.2.6				X	X	X
Total Sulfur, max	Table I	4.5.2.7				X	X	X
Granulation	Table I	4.5.2.8				X	X	X
Lubricating quality	3.4	4.5.2.9		X			X	
Glazing quality	3.5	4.5.2.10		X			X	
Packaging	5.1	4.4.2.1			X		X	X
Workmanship	3.6	4.4.2.1			X		X	X

^{4.1 &}lt;u>Classification of inspection</u>. The verification requirements specified herein are classified as follows.

a. First article inspection (see 4.3)

- b. Conformance inspection (see 4.4)
- 4.2 <u>Verification conditions</u>. Unless otherwise specified, all verifications shall be performed in accordance with the test condition specified in the applicable test method document of the paragraphs in the specification.
- 4.3 <u>First article inspection</u>. When specified, a sample shall be subjected to first article inspection in accordance with Table III.
- 4.3.1 <u>Inspections to be performed</u>. The first article inspection shall be 100% examination of all characteristics including workmanship requirements for the components listed and the tests specified in Table III.
- 4.3.2 <u>Rejection</u>. The first article sample shall be rejected if any of the inspections/tests in Table III are not acceptable.

	NO. OF SAMPLE	REQUIREMENT	INSPECTION	
	UNITS	PARAGRAPH	METHOD	
Appearance	Note 1	3.2	Visual	
Moisture, maximum	Note 1	3.3	4.5.2.1	
Ash, maximum	Note 1	3.3	4.5.2.2	
Silica, maximum	Note 1	3.3	4.5.2.3	
Other grit	Note 1	3.3	4.5.2.4	
Acidity	Note 1	3.3	4.5.2.5	
Free sulfur, maximum	Note 1	3.3	4.5.2.6	
Total Sulfur, maximum	Note 1	3.3	4.5.2.7	
Granulation	Note 1	3.3	4.5.2.8	
Lubricating quality	As required	3.4	4.5.2.9	
Glazing quality	As required	3.5	4.5.2.10	
Note 1. Samples shall be taken from a 600 gram sample representing the lot.				

TABLE III. First article.

4.4 Conformance inspection.

4.4.1 <u>Lot formation</u>. Inspection lots shall comply with the lot formation provisions of MIL-STD-1916. A lot shall consist of graphite of one grade only, produced by one manufacturer in accordance with the same specification or same specification revision, from the same batch or blending operation that has been subject to the same processing operation or condition. A batch shall be that quantity of graphite that has been subjected to the same unit chemical or physical process intended to make the final product substantially uniform.

4.4.2 Classification of characteristics.

- a. <u>Sampling requirements</u>. Inspection sampling requirements for Critical, Major, and Minor characteristics are as defined in MIL-STD-1916. In addition to the sampling requirements stated herein, Verification Level VII shall be used to verify the 100% screening operation of Critical characteristics. Unless otherwise specified, Inspection Level IV shall be used for all characteristics defined as Majors and Inspection Level II for all Minor characteristics; Critical characteristics shall be addressed in accordance with MIL-STD-1916.
- b. <u>Conformance inspection</u>. Conformance inspection shall be performed in accordance with paragraph 4.4.2.1. For all conformance inspections the same sample specimen may be used for all non-destructive examinations or tests.

4.4.2.1	Graphite				
Classification	Examination Or Test	Conformance Criteria	Requirement Paragraph	Inspection Method Reference	
Critical	None defined				
<u>Major</u>		1.5.1			
101	Appearance	4.5.1	3.2	Visual	
102	Moisture, maximum	4.5.1	3.3	4.5.2.1	
103	Ash, maximum	4.5.1	3.3	4.5.2.2	
104	Silica, maximum	4.5.1	3.3	4.5.2.3	
105	Other grit	4.5.1	3.3	4.5.2.4	
106	Acidity	4.5.1	3.3	4.5.2.5	
107	Free sulfur, maximum	4.5.1	3.3	4.5.2.6	
108	Total sulfur, maximum	4.5.1	3.3	4.5.2.7	
109	Granulation	4.5.1	3.3	4.5.2.8	
Minor					
201	Foreign contamination	Level II	3.6	Visual	
202	Container damaged	Level II	5.1	Visual	
203	Container sealed	Level II	5.1	Visual	
204	Weight markings correct	Level II	5.1	Visual	
205	Marking missing or incorrect	Level II	5.1	Visual	
206	Workmanship	Level II	3.6	Visual	
Note. Level II sampling shall be done by container.					

4.5 Methods of inspection.

4.5.1 <u>Sampling</u>. Select 10 percent of the containers in the lot. If there are less than 100 containers in the lot, select 10 containers. If there are less than 10 containers in the lot, all containers shall be selected. Remove a portion of approximately 60 grams (g) of material from

each container. Mix thoroughly each of the primary samples so obtained and remove sufficient material to form a composite sample of approximately 600 g. Mix the composite sample thoroughly, place this in an airtight container, and label so as to show name of the material, manufacturer, plant, contract or order number, lot number, and number of pounds in the lot. All acceptance tests shall be made on the composite sample representative of the lot. If the composite sample fails to comply with any of the requirements specified the lot shall be rejected.

- 4.5.2 <u>Test methods and procedures</u>. The following test methods and procedures shall be performed.
- 4.5.2.1 <u>Determination of moisture</u>. Transfer approximately 2 g of the composite sample to a tared glass weighing dish and weigh accurately. Dry the dish and contents at 100-105 degrees Celsius (°C) for 1 hour, cool the crucible for 1 hour at room temperature (20-22 °C) and weigh. Calculate the loss in weight and percent of moisture.

Percent moisture = $100 \cdot (B - A)/B$

Where:

A= weight of sample after heating, g B= weight of sample before heating, g

4.5.2.2 <u>Determination of ash.</u> Transfer (to a tared, porcelain crucible for Grades I and III, and a platinum crucible for Grades II and IV) a portion of approximately 1 g of the composite sample and weigh accurately. Using a Bunsen burner or muffle furnace, ignite the crucible and contents until all combustible matter has been consumed, cool the crucible for 1 hour at room temperature $(20-22\,^{\circ}\text{C})$ and weigh. Retain the residue for the silica and other grit determinations as specified in 4.5.2.3 and 4.5.2.4.

Calculate the weight of residue as percent ash as follows:

Percent ash = $100 \cdot C/D$

Where:

C= weight of residue after heating, g D= weight of sample before heating, g

4.5.2.3 <u>Determination of silica</u>. (Applicable to Grades II and IV only) To the ash in the platinum crucible, add five milliliters (mL) of hydrofluoric acid and a few drops of sulfuric acid. Evaporate and ignite. Cool the crucible for 1 hour at room temperature $(20 - 22 \, ^{\circ}\text{C})$ and weigh.

Calculate the loss in weight as a percent silica as follows, and retain the residue for grit determination.

Percent silica = $100 \cdot (C - E)/D$

Where:

C= weight of residue after ash determination, as found in paragraph 4.5.2.2, g

D= weight of sample before ash determination in paragraph 4.5.2.2, g

E= weight of ash residue after acid treatment in this paragraph, g

4.5.2.4 <u>Determination of other grit</u>.

- 4.5.2.4.1 <u>Applicable to Grade I and Grade III only</u>. Transfer at least three portions of approximately 0.1 g each of the composite sample, to smooth glass slides and cover each with a clean glass slide. Rub each material between glass slides determining the presence of grit by scratching noise and or scratches on the glass slides.
- 4.5.2.4.2 <u>Applicable to Grade II and Grade IV only</u>. Transfer the residue from silica determination to a smooth glass slide and cover with another clean glass slide. Rub the material between the glass slides and determine the presence of grit by scratching noise and or scratches on the glass slides.
- 4.5.2.5 <u>Determination of acidity</u>. Transfer a portion of approximately 10 g of the sample to a 250 mL beaker, add 100 mL of neutral distilled water and heat quickly to boiling while stirring. Filter immediately and cool to room temperature. Test the filtrate by adding two drops of phenolphthalein and then two drops of methyl red. If colorless to phenolphthalein and yellow to methyl red, acidity shall be reported as none.
- 4.5.2.6 Determination of free sulfur. Extract an accurately weighed portion of approximately 25 g of the composite sample with 50 mL of the boiling ethyl ether for approximately 15 minutes. Filter, wash the residue with ether, and concentrate the combined filtrate and washings to dryness. To the ether soluble residue add 10 mL of concentrated nitric acid, 5 mL of concentrated hydrochloric acid and again concentrate the mixture to dryness. Moisten the residue with 2-4 mL of concentrated hydrochloric acid and then add 30-40 mL of hot distilled water, filter, wash the residue with distilled water, keeping the final volume of filtrate and washings within 100 mL. Heat to boiling. Then rapidly add 10 mL of 3 percent barium chloride solution with vigorous agitation, remove from heat and allow the barium sulfate precipitate to settle for at least 24 hours. Filter through a tared fine porosity crucible, wash the residue with hot distilled water, and test the last few washings of the precipitate with 2 drops of

silver nitrate to see that all chloride is removed. Heat over a Bunsen burner until dull red-hot, then cool the crucible for 1 hour at room temperature $(20-22\,^{\circ}\text{C})$ and weigh. Calculate the increase in weight, corrected for the barium sulfate obtained in a blank determination on all reagents, to percentage of free sulfur in the specimen as follows:

Percent free sulfur = $100 \cdot 13.73$ (F – G)/X

Where:

F= weight of precipitate in sample, g G= weight of precipitate in blank, g X= weight of sample, g

4.5.2.7 <u>Determination of total sulfur</u>. Transfer an accurately weighed portion of approximately 1 g of the composite sample to a beaker. Add 30 mL of concentrated nitric acid and 0.1 g of sulfate-free sodium carbonate. Heat the mixture to boiling and then add 60 mL of concentrated hydrochloric acid, slowly at first, until violent reaction has subsided. Concentrate the solution to dryness. Moisten the residue with 2 to 4 mL of concentrated hydrochloric acid, add 30 to 40 mL of hot distilled water and digest for a few minutes on a steam bath. Filter the mixture and wash the residue with distilled water, keeping the final volume of filtrate washing within 100 mL. Heat the filtrate to boiling and rapidly add 10 mL of 5 percent barium chloride solution, with vigorous agitation. Allow the precipitate of barium sulfate to settle for at least 24 hours. Filter the mixture through a tared fine porosity crucible. Wash the residue with hot distilled water and test the last few washings of the precipitate with 2 drops of silver nitrate to see that all chloride is removed. Heat over a Bunsen burner until dull red-hot, then cool the crucible for 1 hour at room temperature (20 – 22 °C) and weigh. Calculate the increase in weight, corrected for the barium sulfate obtained in a blank determination of all reagents, to percentage of total sulfur in the specimen as follows:

Percent total sulfur = $100 \cdot 13.73 (H - I)/Y$

Where:

H= weight of precipitate in sample, g I= weight of precipitate in blank, g Y= weight of sample, g

4.5.2.8 <u>Determination of granulation</u>. Place an accurately weighted portion of 25 g of the sample and two metallic washers on the appropriate U.S. alternative sieve, in accordance with ASTM E11, assembled to a bottom pan. Place a cover on the sieve and shake the assembly for

10 minutes by hand or 5 minutes by means of a mechanical shaker geared to produce between 268 and 315 gyrations per minute and 150 + 10 taps of the striker per minute. When no more material passes through, weigh the portion retained on the sieve and calculate the percentage of specimen passing through the sieve as follows:

Percent Through =
$$((Z - (J + K))/Z) \cdot 100$$

Where:

J= weight retained on designated sieve, g K= weight retained on sieves nested above designated sieve, g Z=weight of sample, g

- 4.5.2.9 <u>Lubricating quality</u>. When specified a sufficient amount of material shall be used to verify the requirement of paragraph 3.4 (see 6.2 and 6.6). If multiple batches of material are to be used in production a sample from each shall be used.
- 4.5.2.10 <u>Glazing quality</u>. When specified a sufficient amount of material shall be used to verify the requirement of paragraph 3.5 (see 6.2 and 6.6). If multiple batches of material are to be used in production a sample from each shall be used.

5. PACKAGING

5.1 <u>Packaging</u>. The container shall be sealed and undamaged with the markings including weight present and correct (see 6.2b).

6. NOTES

- 6.1 <u>Intended use</u>. Graphite is intended for the following uses:
- 6.1.1 Grades I and II as lubricants in pelleting explosives.
- 6.1.2 Grades III and IV as glazing agents.
- 6.2 Acquisition requirements. Acquisition documents should specify the following:
- a. Title, number, and date of this specification.
- b. Packaging requirements.
- c. Requirements for First article samples (see 4.3).
- d. Provision for submission of first article samples.
- e. Requirements for submission of acceptance inspection equipment (see 6.3).
- f. Grade of material required (see 1.2).
- g. Requirements and tests for lubricating quality, if required, for Grade I and Grade II only (see 3.4, 4.5.2.9, & 6.6).
- h. Requirements and tests for glazing quality, if required, for Grade III and Grade IV only (see 3.4, 4.5.2.9, & 6.7).
- 6.3 <u>Submission of contractor acceptance inspection equipment (AIE) designs for approval</u>. Submit copies of designs as required to: Commander, U.S. Army ARDEC, ATTN: RDAR-EIQ-MA, Picatinny, NJ 07806-5000. This address will be specified on the Contract Data Requirements List, DD Form 1423 in the contract.
- 6.4 <u>Equivalent test method approval</u>. Prior approval of the contracting officer is required for use of equivalent test methods. A description of the proposed method should be submitted to the contracting officer. This description should include, but not be limited to, the procedures used, the accuracy and precision, and drawings of any special equipment required.
 - 6.5 Material.
 - 6.5.1 Grades I and III graphites are manufactured graphites
 - 6.5.2 Grades II and IV graphites are natural graphites.
- 6.6 <u>Historic pelletizing and glazing quality tests</u>. Prior revisions of MIL-G-155 called out methods for performing testing. Specifically desired requirements and tests should be called out on the drawing, contract, or purchase order (see 6.2).
- 6.6.1 <u>Historic pelletizing quality</u>. Prior revisions specified "Mix two parts by weight of the sample and 100 parts of weight of tetryl complying with the requirements of specification JAN-T-339. Make at least 25 pellets of the mixture by subjecting portions of the mixture to pressure in a pelletizing machine."

6.6.2 <u>Historic glazing quality</u>. Prior revisions specified "The graphite [should] be subjected to an actual factory glazing operation and to such additional test as should be considered necessary by the contracting officer."

6.7 Change history.

Specification Revision	ECP Number	ERR Release Date
MIL-G-155	-	13 December 1944
MIL-G-155A	-	20 September 1962
MIL-G-155A w	-	12 January 1967
Amendment 1		
MIL-G-155A w	-	13 July 1967
Amendment 2		
MIL-DTL-155B	Y18Q2002	-

6.8 Subject term (key word) listing.

Glazing

Lubrication

Pelletization

6.9 <u>Changes from previous issue</u>. Marginal notations are not used in this revision to identify changes with respect to the previous issue due to the extent of the changes.

Custodians: Preparing Activity:

Army-AR

Navy – OS (Project 1376-2014-044)

Air Force – 11

Review activities:

Army - AV, MI

Navy - AS

Air Force - 70