

INCH-POUND

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MILITARY SPECIFICATION

LEAD STYPHNATE, BASIC

This specification is approved for use by the US Army Armament, Research, Development and Engineering Center and is available for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers the requirements and quality assurance provisions for the manufacture and acceptance of two types of basic lead styphnate for use in explosive priming compositions. (see 6.1).

1.2 Classification. Basic lead styphnate shall conform to one of the following types (see 6.2):

Type I: Color: Yellow to orange yellow
Dimensions: Seventy-five (75) percent shall have a length between 7 to 20 microns.
Form: Needle crystals

Type II: Color: Yellow brown to brown
Dimensions: Ninety-five (95) percent shall have a length between 7 to 35 microns.
Form: Discrete crystals

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commander, U.S. Army ARDEC, ATTN: SMCAR-BAC-S, Picatinny Arsenal, New Jersey 07806-5000 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

AMSC N/A

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2. APPLICABLE DOCUMENTS

2.1 Government documents.

2.1.1 Specifications and standards. 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 listed in the issue of the Department of Defense Index of Specifications and Standards (DODISS) and supplement thereto, cited in the solicitation (see 6.2).

SPECIFICATIONS

FEDERAL

RR-S-366 - Sieves, Standard for Testing Purposes

STANDARDS

MILITARY

MIL-STD-109 - Quality Assurance Terms and Definitions
MIL-STD-129 - Marking for Shipment and Storage
MIL-STD-650 - Explosive: Sampling Inspection and Testing
MIL-STD-1168 - Lot Numbering of Ammunition
MIL-STD-1218 - ACS Chemicals

(Unless otherwise indicated, copies of federal and military specifications, standards, and handbooks are available from the Naval Publications and Forms Center, (ATTN: NPODS), 5801 Tabor Avenue, Philadelphia, PA 19120-5099.)

2.1.2 Other Government document. The following other Government publication forms a part of this document to the extent specified herein. Unless otherwise specified, the issues are those cited in the solicitation.

CODE OF FEDERAL REGULATIONS

49 CFR 100-199 - Department of Transportation Rules and Regulations for the Transportation of Hazardous Materials

(The Interstate Commerce Commission Regulation are now a part of the Code of Federal Regulations, available from the

Superintendent of Documents, US Government Printing Office, Washington, DC 20402. Orders for the above publications should cite, "49 CFR 100-199 (latest revision)".)

2.2 Non-Government publications. The following publications form a part of this document to the extent specified herein. Unless otherwise specified, the issues of the documents which are DoD adopted are those listed in the issue of the DODISS cited in the solicitation. Unless otherwise specified, the issues of documents not listed in the DODISS are the issues of the documents cited in the solicitation (see 6.2).

AMERICAN SOCIETY FOR TESTING AND MATERIALS

- | | |
|------------|--|
| ASTM D2905 | - Statement on Number of Specimens Required to Determine the Average Quality of a Textile Material |
| ASTM E70 | - pH of Aqueous Solutions with Glass Electrode, Test for Material |
| ASTM E300 | - Sampling Industrial Chemicals |

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

(Non-Government standards and other publications are normally available from the organizations that prepare or distribute the documents. These documents also may be available in or through libraries or other informational services.)

2.3 Order of precedence. 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 Color and crystalline form. The lead styphnate shall conform essentially to the following when examined as specified in 4.5.2:

- | | |
|----------|-------------------------------------|
| Type I: | Color: Yellow to orange yellow |
| | Crystalline form: Needles |
| Type II: | Color: Yellow brown to brown |
| | Crystalline form: Discrete crystals |

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3.2 Crystal dimensions. The crystal dimensions shall conform to the following when tested as specified in 4.5.3:

Type I: A minimum of seventy-five (75) percent of the crystals shall have the longest dimension between 7 and 20 microns and that dimension shall not be greater than five times the width of the crystal.

Type II: A minimum of ninety-five (95) percent of the crystals shall have a length between 7 and 35 microns.

3.3 Granulation. One hundred percent of Type I and Type II material shall pass through a US Standard Sieve No. 325 when tested as specified in 4.5.4.

3.4 Chemical and physical properties. The basic lead styphnate shall conform to the properties given in Table I when tested in accordance with the applicable test paragraph.

TABLE I. Chemical and physical properties.

<u>Property</u>	<u>Requirements</u>		<u>Applicable test paragraph</u>
	<u>Type I</u>	<u>Type II</u>	
Apparent density, gm/ml	0.3 -0.5	1.40-1.60	4.5.5
Acidity, pH	5.0-6.0	6.0-7.0	4.5.6
Insoluble, in ammonium acetate, max, percent	0.2	0.2	4.5.7
Soluble in ether, max percent	0.1	0.1	4.5.8
Lead, percent	59.0-60.0	59.0 -60.0	4.5.9
Nitrogen, percent	5.97-6.17	5.97-6.17	4.5.10
Instantaneous flash point, °C	330-350	330-350	4.5.11

3.5 First article. When specified in the acquisition document (see 6.2), a sample shall be subjected to first article inspection (see 6.3) in accordance with 4.3.

3.6 Workmanship. The manufacturer shall implement procedures and controls to assure that the process and the product are not compromised by foreign materials and contaminants or any other

conditions which may degrade the composition. Determination of foreign materials shall be in accordance with 4.5.12.

4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the supplier is responsible for the performance of required inspections, examinations and tests as specified herein. Except as otherwise specified in the contract or order, the supplier may use his own or any other facilities suitable for the performance of the inspections, examination and tests specified herein, unless disapproved by the Government. In addition, the Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure supplies and services conform to prescribed requirements. Reference shall be made to MIL-STD-109 to define terms used herein.

4.1.1 Responsibility for compliance. Basic lead styphnate shall meet all requirements of sections 3 and 5. The inspections set forth in this specification shall become a part of the contractor's overall inspection system or quality program. The absence of any inspection requirements in the specification shall not relieve the contractor of the responsibility of ensuring that all products or supplies submitted to the Government for acceptance comply with all requirements of the contract. Sampling inspection, as part of manufacturing operations, is an acceptable practice to ascertain conformance to requirements, however, this does not authorize submission of known defective material, either indicated or actual, nor does it commit the Government to accept defective material.

4.2 Classification of inspections. The required inspections specified herein are classified as follows:

- a. First article inspection (see 4.3)
- b. Quality conformance inspection (see 4.4)

4.3 First article inspection.

4.3.1 Submission. When specified in the contract or purchase order, the contractor shall submit a first article sample consisting of one (1) pound of basic lead styphnate. The first article sample shall be obtained from the first production lot which has been produced by the contractor, using the same production processes, procedures and equipment as will be used in fulfilling the contract. All raw materials used in producing the basic lead styphnate shall be from the same sources as will be used in regular production.

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4.3.2 Inspections to be performed. The first article sample may be subjected by the Government to any or all of the examinations or tests specified herein.

TABLE II. First Article Inspection
CLASSIFICATION OF CHARACTERISTICS

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PARAGRAPH	TITLE	SHEET 1 OF 1		DRAWING NUMBER
				NEXT HIGHER ASSEMBLY
CLASSIFICATION	EXAMINATION OR TEST	CONFORMANCE CRITERIA	REQUIREMENT PARAGRAPH	INSPECTION METHOD REFERENCE
	Characteristics prior to packing			
	Color and crystalline form		3.1	4.5.2
	Crystal dimensions		3.2	4.5.3
	Granulation		3.3	4.5.4
	Apparent density		3.4	4.5.5
	Acidity, pH		3.4	4.5.6
	Insoluble in ammonium acetate		3.4	4.5.7
	Soluble in ether		3.4	4.5.8
	Lead content		3.4	4.5.9
	Nitrogen content		3.4	4.5.10
	Instantaneous flash point		3.4	4.5.11
	Workmanship		3.6	4.5.12
NOTES:				

AMSMC Form 1570b, 1 Jul 89

Replaces 1570, 1 Feb 85, which may not be used.

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4.3.3 Rejection. If the sample fails to comply with any of the applicable requirements, the first article quantity shall be rejected. The Government reserves the right to terminate its inspection upon any failure of a sample to comply with any of the stated requirements.

4.4 Quality conformance inspection.

4.4.1 Inspection lot formation. A lot shall consist of one or more batches produced by one manufacturer, in accordance with the same specification, or same specification revision, under one continuous set of operating conditions. Each batch shall consist of that quantity of basic lead styphnate that has been subjected to the same unit chemical or physical process intended to make the final product homogenous. The criteria and procedure for the assignment of lot numbers shall be in accordance with MIL-STD-1168.

4.4.2 Examination and tests.

4.4.2.1 Classification of characteristics. Quality conformance examinations and tests are specified in the following classification of characteristics paragraphs. The contractor's quality program or detailed inspection system shall provide assurance of compliance of all characteristics with the applicable specification requirements utilizing as a minimum the conformance criteria specified herein.

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PARAGRAPH	TITLE	SHEET 1 OF 1		DRAWING NUMBER
4.4.2.1.1	Chemical and physical characteristics			NEXT HIGHER ASSEMBLY
CLASSIFICATION	EXAMINATION OR TEST	CONFORMANCE CRITERIA	REQUIREMENT PARAGRAPH	INSPECTION METHOD REFERENCE
<u>Critical</u>	None defined			
<u>Major</u>				
101	Color and crystalline form	4.4.3.2	3.1	4.5.2
102	Crystal dimensions	4.4.3.2	3.2	4.5.3
103	Granulation	4.4.3.3	3.3	4.5.4
104	Apparent density	4.4.3.2	3.4	4.5.5
105	Acidity, pH	4.4.3.2	3.4	4.5.6
106	Material insoluble in ammonium acetate	4.4.3.3	3.4	4.5.7
107	Material soluble in ether	4.4.3.3	3.4	4.5.8
108	Lead content	4.4.3.3	3.4	4.5.9
109	Nitrogen content	4.4.3.3	3.4	4.5.10
110	Instantaneous flash point	4.4.3.3	3.4	4.5.11
<u>Minor</u>				
201	Workmanship	4.4.3.2	3.6	4.5.12
NOTES:				

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Replaces 1570, 1 Feb 85, which may not be used.

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CLASSIFICATION OF CHARACTERISTICS

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PARAGRAPH	TITLE	SHEET 1 OF 1		DRAWING NUMBER
4.4.2.1.2	Bag, cambric or cloth			NEXT HIGHER ASSEMBLY
CLASSIFICATION	EXAMINATION OR TEST	CONFORMANCE CRITERIA	REQUIREMENT PARAGRAPH	INSPECTION METHOD REFERENCE
<u>Critical</u>				
001	Basic lead styphnate improperly or insufficiently wet	100%		5.1.1/Visual, prior to closing
<u>Major</u>				
101	Cloth cap missing, torn or improperly positioned	100%		5.1.1/Visual, prior to closing
102	Bag pierced or torn	100%		5.1.1/Visual, prior to packing
103	Bag improperly closed	100%		5.1.1/Visual, prior to packing
<u>Minor</u>	None defined			
NOTES:				

AMSMC Form 1570b, 1 Jul 89

Replaces 1570, 1 Feb 85, which may not be used.

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CLASSIFICATION OF CHARACTERISTICS

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PARAGRAPH	TITLE	SHEET 1 OF 1		DRAWING NUMBER
4.4.2.1.3	Bag, velostat, conductive			NEXT HIGHER ASSEMBLY
CLASSIFICATION	EXAMINATION OR TEST	CONFORMANCE CRITERIA	REQUIREMENT PARAGRAPH	INSPECTION METHOD REFERENCE
<u>Critical</u>	None defined			
<u>Major</u>				
101	Cloth cap missing, torn or improperly positioned	100%		5.1.1/Visual, prior to closing
102	Insufficient alcohol/water covering inner bag	100%		5.1.1/Visual, prior to closing
103	Bag improperly closed	100%		5.1.1/Visual, prior to packing
104	Bag pierced or torn	100%		5.1.1/Visual, prior to packing
<u>Minor</u>	None defined			
NOTES:				

AMSMC Form 1570b, 1 Jul 89

Replaces 1570, 1 Feb 85, which may not be used.

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PARAGRAPH 4.4.2.1.4	TITLE Bag, cotton duck	SHEET 1 OF 1		DRAWING NUMBER
				NEXT HIGHER ASSEMBLY
CLASSIFICATION	EXAMINATION OR TEST	CONFORMANCE CRITERIA	REQUIREMENT PARAGRAPH	INSPECTION METHOD REFERENCE
<u>Critical</u> <u>Major</u> 101 102 103 104 <u>Minor</u>	None defined Rubber cloth cap missing Bag pierced or torn Bag improperly closed Marking missing or incorrect None defined	 100% 100% 100% 100%	 	 5.1.1/Visual, prior to closing 5.1.1/Visual, prior to packing 5.1.1/Visual, prior to packing 5.1.1/Visual, prior to packing
NOTES:				

AMSMC Form 1570b, 1 Jul 89

Replaces 1570, 1 Feb 85, which may not be used.

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CLASSIFICATION OF CHARACTERISTICS

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PARAGRAPH	TITLE	SHEET 1 OF 1		DRAWING NUMBER
4.4.2.1.5	Bag, rubber cloth			NEXT HIGHER ASSEMBLY
CLASSIFICATION	EXAMINATION OR TEST	CONFORMANCE CRITERIA	REQUIREMENT PARAGRAPH	INSPECTION METHOD REFERENCE
<u>Critical</u>	None defined			
<u>Major</u>				
101	Insufficient alcohol/water covering inner bag	100%		5.1.1/Visual, prior to closing
102	Bag improperly closed	100%		5.1.1/Visual, prior to packing
103	Bag pierced or torn	100%		5.1.1/Visual, prior to packing
<u>Minor</u>	None defined			
NOTES:				

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Replaces 1570, 1 Feb 85, which may not be used.

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PARAGRAPH	TITLE	SHEET 1 OF 1		DRAWING NUMBER
4.4.2.1.6	Bag, grain			NEXT HIGHER ASSEMBLY
CLASSIFICATION	EXAMINATION OR TEST	CONFORMANCE CRITERIA	REQUIREMENT PARAGRAPH	INSPECTION METHOD REFERENCE
<u>Critical</u>	None defined			
<u>Major</u>				
101	Bag improperly closed	100%		5.1.1/Visual, prior to packing
<u>Minor</u>	None defined			
NOTES:				

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Replaces 1570, 1 Feb 85, which may not be used.

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CLASSIFICATION OF CHARACTERISTICS

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PARAGRAPH	TITLE	SHEET 1 OF 1		DRAWING NUMBER
4.4.2.1.7	Bag, jute liner			NEXT HIGHER ASSEMBLY
CLASSIFICATION	EXAMINATION OR TEST	CONFORMANCE CRITERIA	REQUIREMENT PARAGRAPH	INSPECTION METHOD REFERENCE
<u>Critical</u>				
001	None defined			
<u>Major</u>				
101	Enclosed grain bag incompletely surrounded by sawdust	100%		5.1.1/Visual, measuring rule prior to closing
102	Sawdust insufficiently saturated with water/alcohol	100%		5.1.1/Visual, prior to closing
103	Jute bag torn	100%		5.1.1/Visual, prior to packing
104	Jute bag improperly close	100%		5.1.1/Visual, prior to packing
<u>Minor</u>				
	None defined			
NOTES:				

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Replaces 1570, 1 Feb 85, which may not be used.

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PARAGRAPH	TITLE	SHEET 1 OF 1		DRAWING NUMBER
4.4.2.1.8	Container, drum or barrel			NEXT HIGHER ASSEMBLY
CLASSIFICATION	EXAMINATION OR TEST	CONFORMANCE CRITERIA	REQUIREMENT PARAGRAPH	INSPECTION METHOD REFERENCE
<u>Critical</u>	None defined			
001	Drum or barrel leaking	100%		5.1.1/Visual
002	Marking misleading or unidentifiable	100%		5.2/Visual
<u>Major</u>				
101	Container improperly closed or sealed	100%		5.1.1/Visual
<u>Minor</u>				
201	Markings blurred or of poor quality	100%		5.1.1/Visual
202	Bare spot, other than slight scratches, on exterior (applicable to metal container)	100%		5.1.1/Visual
NOTES:				

AMSMC Form 1570b, 1 Jul 89

Replaces 1570, 1 Feb 85, which may not be used.

4.4.3 Testing. PRECAUTION: This specification covers sampling and testing of toxic and hazardous material. Accordingly, it is emphasized that all applicable safety rules, regulations and procedures must be followed in handling and processing.

4.4.3.1 Sampling. Obtain two random 30 gm samples from each batch of basic lead styphnate being submitted for inclusion in a lot. The samples shall be obtained from the process vessel prior to packout using procedures described in ASTM-E300 for slurries.

4.4.3.2 Testing for batch acceptance. One sample obtained according to 4.4.3.1 from each batch shall be tested in accordance with applicable test methods of 4.5 for conformance with the requirements for color and crystalline form (see 3.1), crystal dimensions (see 3.2), apparent density (see 3.4), acidity, pH (see 3.4), and workmanship (see 3.6). If any sample fails to meet any of these requirements, the batch represented by the sample shall be rejected. Rework of non-conforming batches is permitted.

4.4.3.3 Testing for lot acceptance. The second samples from the batches not rejected under the provisions of 4.4.3.2 shall be placed in a suitable container of sufficient volume to hold a composite sample from all batches in the lot. The composite sample shall be thoroughly mixed to assure homogeneity, and two sub-samples of approximately 30 gm each shall be obtained from this composite sample. Both sub-samples shall be tested in accordance with the applicable test methods of 4.5 to determine conformance with the requirements of granulation (see 3.3), material insoluble in ammonium acetate (see 3.4), material soluble in ether (see 3.4), lead content (see 3.4), nitrogen content (see 3.4), and instantaneous flash point (see 3.4). If either sub-sample fails to meet any of the test requirements, the lot represented by the composite sample shall be rejected.

4.4.4 Inspection equipment. For the performance of all test and examinations specified in 4.3 and 4.4, the equipment specified should be employed. The contractor shall have this equipment available, utilize it in accordance with the test method, and is responsible to assure that it is properly calibrated. Government approval of all contractor designed inspection equipment is required prior to its use for acceptance purposes (see 6.4).

4.5 Methods of inspection. All tests described in this section shall be performed using prescribed procedures for replicate determination given in standard analytical chemistry textbooks or ASTM D 2905. Unless otherwise specified herein, all

chemicals shall be Reagent Grade or ACS Grade in accordance with MIL-STD-1218. If these grades are not available, all chemicals shall be the highest purity grade commercially available. See 6.5 for the use of equivalent test methods.

4.5.1 Preparation of dry sample. Transfer an approximately 10 gm portion of the wet sample obtained in 4.4.3.1 to a Buchner funnel fitted with a medium porosity filter paper. Apply suction to the apparatus until sample is almost dry. Dry sample in an oven maintained at 70-75°C for two (2) hours or until constant weight to the nearest 1 mg is obtained. Use this dried sample for the following determinations unless otherwise specified.

4.5.2 Color and crystalline form. Take a portion of the well stirred wet sample from 4.4.3.1 and spread over an area of approximately 4 sq. cm. on a glass microscope slide. Allow the sample to air dry. Examine the material microscopically for color and impurities under daylight illumination. Observe the crystalline form (shape) under a microscope using a magnification of 300X under reflected light and, optionally, take a photomicrograph at about 225X magnification. Use this slide for crystal dimensions determination in 4.5.3.

4.5.3 Crystal dimensions. Using the same slide and magnification as in 4.5.2, measure the longest dimension of the larger crystals observed when scanning the field with the aid of a mechanical stage. Use transmitted and reflected light and an ocular micrometer which has been calibrated by means of the stage micrometer. Consider the longest dimension to be the distance between the two most remote points of any one crystal. Report the average length of 20 crystals.

4.5.4 Granulation. Transfer about 30 gm of the wet sample to a Buchner funnel and apply suction for 2-3 minutes. Weigh out a 10 gm portion of the damp sample and transfer it to a clean three inch US Standard No. 325 sieve which conforms to the requirements of RR-S-366. Carefully examine the sieve to be sure it is free of foreign material. Place the sieve in a porcelain evaporating dish which contains enough water to cover the wire screen of the sieve to a depth of about one half inch. Shake the sieve for a few minutes in such a manner that the basic lead styphnate is below the surface of the water and any material passing through the sieve is caught in the evaporating dish. When most of the basic lead styphnate has passed through the sieve, run a gentle stream of water through the sieve while brushing the residue gently with a soft camel-hair brush until all of the basic lead styphnate that will pass through the sieve has passed through it. Be sure that all of the material passing through the sieve is caught in a container and none is washed down the drain. At the completion of this operation, visually

examine the screen of the sieve for the presence of basic lead styphnate or foreign material. (see 4.5.12)

4.5.5 Apparent density. Determine the apparent density according to Method 201.2, MIL-STD-650 using n-butyl alcohol as the liquid medium.

4.5.5.1 Apparent density (Alternate Method). Transfer approximately 2 to 3 grams of the wet sample obtained in 4.4.3.1 to a Buchner funnel fitted with a medium porosity filter paper. Rinse the wet sample with denatured ethyl alcohol to displace water. Rinse the sample with n-butyl alcohol to displace denatured ethyl alcohol. Transfer the sample to a 5 or 10 mL graduate, having 0.1 mL graduations. Rinse down the sides of the cylinder with enough n-butyl alcohol to slurry the sample. Stir with a wooden stick, or agitate by some means in order to free any trapped air bubbles. Fill to the top cylinder graduation with n-butyl alcohol as accurately as possible, being sure the sides are rinsed clean. Weigh the cylinder and contents to the nearest mg (0.001 gm). Record this weight as W1. Allow the cylinder contents to settle for a minimum of 2 hours, then note volume in mL to the nearest 0.05 mL, occupied by the sample. Record this volume as V. Determine the weight of the cylinder filled to the top cylinder graduation with n-butyl alcohol only (no sample). Record this weight as W2.

Calculate the apparent density as follows:

$$\text{Apparent density} = \frac{D_s(W_1 - W_2)}{V(D_s - D_b)}$$

Where: D_s = Density of normal lead styphnate
 D_b = Density of N-butyl alcohol

4.5.6 Acidity, pH. Transfer approximately 50 mL of the water covering the sample obtained in 4.4.3.1 to a 200 mL beaker. Determine the pH value of this solution in accordance with method given in ASTM-E70.

4.5.7 Material insoluble in ammonium acetate. Transfer 1 gm of the dry sample, weighed to the nearest 1 mg to a 250 mL beaker. Add 100 mL of water and then add slowly with constant stirring 100 mL of 20 percent ammonium acetate solution. For fifteen minutes frequently stir the contents of the beaker and then filter the solution through a tared sintered-glass crucible of fine porosity. Transfer the residue to the crucible and wash the crucible with water until the filtrate is colorless. Dry in an oven at 100°C to 105°C for 1 hour, cool in a desiccator, and weigh to the nearest 0.1 mg. Calculate the percent material insoluble in ammonium acetate as follows:

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Percent material insoluble in ammonium acetate = $\frac{100A}{W}$

where: A = weight of residue from sample, gm.
W = weight of sample, gm.

4.5.8 Material soluble in ether. Transfer 2 gm of the dry sample weighed to the nearest 1 mg to a 150 mL beaker, add 50 mL of anhydrous ethyl ether and allow to stand with occasional stirring for about 15 minutes. Filter through a dry No. 40 Whatman filter paper or equivalent and catch the filtrate in a tared 100 mL beaker. Wash with a total of 20 mL of ether. Evaporate off the ether in the 100 mL beaker on a steam bath or by means of a current of dry air. Place in a vacuum desiccator containing sulfuric acid until constant weight to the nearest 0.1 mg is obtained. Run a blank determination of 70 mL of ether. Calculate the percent material soluble in ether as follows:

Percent material soluble in ether = $\frac{100(A-B)}{W}$

where: A = weight of residue from sample, gm.
B = weight of residue from blank, gm.
W = weight of sample, gm.

4.5.9 Lead. Transfer 0.45 to 0.55 gm of the dry sample weighed to the nearest 0.1 mg to a 250 mL beaker. Add 10 mL of concentrated nitric acid and 10 mL of concentrated sulfuric acid. Cover the beaker with a watch glass and heat at moderate temperature on a hot plate until dense fumes of sulfur trioxide are evolved. (Oxides of sulfur and nitrogen are toxic, precautions should be taken to assure personnel are not exposed to these fumes.) Allow the beaker to cool for about 2 minutes, add 5 mL of concentrated nitric acid, and heat again until dense fumes of sulfur trioxide are evolved. Repeat the addition of nitric acid and heating to fumes until all the organic matter is destroyed and the solution is practically colorless. Remove the beaker from the hot plate and allow it to cool. Wash down the watch glass and side walls of the beaker with about 10 mL of distilled water. Place the uncovered beaker on the hot plate and again heat until dense fumes of sulfuric acid are evolved. Allow to cool, then cautiously dilute to about 100 mL with distilled water and again allow to cool. Add 25 mL of 95 percent ethyl alcohol and let stand for at least 2 hours. Filter through tared sintered porcelain crucible of fine porosity (or a Gooch crucible with glass fiber filter equivalent to a Whatman glass microfiber, grade 934AS). Transfer the precipitate to the crucible with 5 percent sulfuric acid and twice with 95 percent ethyl alcohol. Dry in an oven at 100° to 105°C for 1/2 hour and then ignite in a muffle furnace at 550° to 600°C for 15 minutes. Cool in a

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desiccator and weigh to the nearest 0.1 mg. Calculate the percent lead in the sample as follows:

$$\text{Percent lead} = \frac{68.32A}{W}$$

Where: A = weight of ignited residue, gm.
W = weight of sample, gm.

4.5.10 Nitrogen. Determine the nitrogen content of the basic lead styphnate in accordance with procedure given in method 409.1 of MIL-STD-650.

4.5.11 Instantaneous flash point. Use samples of the size that may be held on about 1/16 inch of the flat end of an ordinary toothpick and a heated Dennis melting point bar. The instantaneous flash point is determined by: (1) noting the time t, required for the explosion of samples dropped on the bar at several temperatures T, slightly lower than the instantaneous flash point, (2) plotting 1/T vs t, and (3) extrapolating linearly to zero t. The inverse of the extrapolated 1/T is the instantaneous flash point. To determine the temperatures on a Dennis bar, use a thermistor type digital thermometer. The thermistor is placed on the Dennis bar and applied with constant pressure until a steady reading is reached. However, the temperature readings will not reflect the true temperature due to the heat loss of this arrangement. A two point calibration is thus required to obtain the corrected temperature from the Dennis bar. This can be achieved by assuming a linear relationship along that portion of the bar which is used for these measurements. Using two compounds as reference whose melting points are accurately known, the true temperatures for correcting the observed temperature readings can be obtained.

4.5.12 Workmanship. The product shall be visually inspected for the presence of foreign material during sampling (see 4.4.3.1, and 4.5.1) and during the color and crystalline form test (see 4.5.2).

5. PACKAGING

5.1 Packing. Basic lead styphnate shall be packed in a wet condition as directed in 49 CFR 100-199. A mixture of water plus denatured ethyl alcohol in such proportions to prevent freezing shall be used unless otherwise directed.

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5.1.1 Level A. A maximum of 25 lbs (dry weight) basic lead styphnate shall be packed wet (see 5.1) directly into a cambric diaper or similar cloth bag. A cap of the same diameter and fabric as the bag shall be placed inside the bag and over the basic lead styphnate. The bag shall then be tied closed and placed inside a bag made of conductive velostat. This velostat bag shall then be filled with the water/alcohol mixture which meets requirements of 5.1. This water/alcohol mixture must completely cover the bag contents. The velostat bag is then tied closed and placed inside a 4 oz. or heavier duck bag. A cap of the same material and of the same diameter as a duck bag shall be placed over the velostat bag. The duck bag is then securely tied closed. This bag is now the unit bag which shall be marked in accordance with 5.2. An appropriate number of unit bags such that the combined total dry weight of normal lead styphnate does not exceed 150 pounds shall be placed into a rubber cloth bag. Inside this bag and over the unit bags of basic lead styphnate shall be placed a cap of the same fabric and same diameter as this bag. This rubber cloth bag shall be filled with a water/alcohol mixture meeting requirements of 5.1 so that the unit bags are completely covered. The bag is then securely tied closed. The rubber cloth bag shall be placed inside a strong grain bag which shall then be securely tied closed. The grain bag and its contents shall be packed in the center of a Department of Transportation Specification 5 or 5B metal drum or barrel which has been lined with a heavy, close-fitting, jute bag. The grain bag shall be entirely surrounded by not less than three inches of well-packed sawdust saturated by the previously referenced water/alcohol mixture. The jute bag shall then be closed by secure sewing to prevent the escape of the sawdust. After closure of the barrel or drum, the package shall be inspected carefully and all leaks stopped. The dry weight of basic lead styphnate in one outside container shall not exceed 150 pounds.

5.1.2 Level B. Level B shall be the same as Level A.

5.1.3 Level C. Level C shall be the same as Level A except DOT Specification 17H metal drum (single trip), as specified in CFR, Title 49, may be used.

5.2 Marking. Markings shall be in accordance with 49 CFR 100-199 and MIL-STD-129. Markings shall be not less than one-fourth (1/4) inch, nor more than one-half (1/2) inch in height. DOT markings must be one-half (1/2) inch in height. Ink shall conform to that specified in MIL-STD-129 and shall be a contrasting color to the bag.

5.2.1 Unit container. Each unit bag as defined in 5.1.1 shall be marked with the following:

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- a. Lead Styphnate, Basic
- b. Quantity (dry weight) in pounds
- c. Lot number

As an alternative, each unit bag may be identified by using a waterproof tag that is marked with the above information, imprinted with a suitable water/alcohol proof ink.

5.2.2 Exterior container. The side and top of each drum or barrel shall be marked: "INITIATING EXPLOSIVE - DANGEROUS - DO NOT STORE OR LOAD WITH ANY HIGH EXPLOSIVE."

The following additional information will be marked on the side of each drum or barrel:

- a. NSN/NATO Stock number, Type I -
Type II - 1376-01-142-0591
- b. Lead Styphnate, Basic
- c. Quantity (net weight in pounds)
- d. Lot number
- e. Gross weight
- f. Any special cautionary marking and labeling

6. NOTES

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

6.1 Intended use. Basic lead styphnate is lead trinitroresorcinate with hydroxy groups, $C_6H(NO_2)_3(OPbOH)_2$, and is intended for use in priming compositions and detonators. Basic lead styphnate is classified as a high explosive. All necessary precautions in the manufacture, handling, and storage of such material should be observed. Only conductive plastic or horn implements shall be used for stirring or transferring. Never use metallic implements and never scrape residual material out of glass equipment. Destroy residual or excess material by dissolving in ammonium acetate solution, collecting the washings in a large beaker and then slowly adding dilute sulfuric acid to precipitate lead sulfate.

6.2 Acquisition requirements. Acquisition documents must specify the following:

- a. Title, number and date of this detail specification.
- b. Issue of DODISS to be cited in the solicitation, and if required, the specific issue of individual documents referenced (see 2.1.1 and 2.2).
- c. Type of lead styphnate required (see 1.2).
- d. First article requirements (see 3.5, 4.3 and 6.3).

e. Level of packing protection required.

6.3 First article. When first article inspection is required the contracting officer should specify that the contractor submit a first article sample as specified in 4.3.1. The contracting officer should also include specific instructions in acquisition documents regarding arrangements for examinations, approval of first article test results, and disposition of first articles. Invitations for bids should provide that the Government reserves the right to waive the requirement for samples for first article inspection to those bidders offering a product which has been previously acquired or tested by the Government and that bidders offering such products, who wish to rely on such production or test, must furnish evidence with the bid that prior Government approval is presently appropriate for the pending contract. Bidders should not submit alternate bids unless specifically requested to do so in the solicitation.

6.4 Submission of inspection equipment designs for approval. Submit contractor equipment designs, as required, to: Commander, SMCAR, ATTN: SMCAR-QAR-R, Picatinny Arsenal, NJ 07806-5000 (see MIL-A-48078).

6.5 Equivalent test methods. The test methods given in this specification are the official methods to be used. The contractor may request to use other method(s) providing that the proposed method is equivalent (accuracy and precision) to the method given in this specification. Prior approval of the contracting officer is required for use of equivalent test methods. A description of the proposed method should be submitted through the contracting officer to Commander, SMCAR-QAR-R, Picatinny Arsenal, NJ 07806-5000. This description should include the procedures used, the accuracy and precision of the method, test data to demonstrate the accuracy and precision and drawings of any special equipment required.

6.6 Material Safety Data Sheets. Contracting officers will identify those activities requiring copies of completed Material Safety Data Sheets prepared in accordance with FED-STD-313. The pertinent Government mailing addresses for submission of data are listed in FED-STD-313.

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6.7 Change from previous issue. Asterisks are not used in this revision to identify changes with respect to the previous issue due to the extensiveness of the changes.

Custodian
Army-AR

Preparing activity:
Army-AR
(Project 1376-0405)

Review activities:
Navy - OS

