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DETAIL SPECIFICATION
LEAD STYPHNATE, NORMAL

This specification is approved 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 Normal Lead Styphnate for use in primer compositions (Section 6.1).

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 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 sections 3 and 4 of this specification, whether or not they are listed.

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 cited in the solicitation or contract.

Comments, suggestions, or questions on this document shall be addressed to: Commander, US Army ARDEC, Attn: RDAR-QES-E, Picatinny Arsenal, New Jersey 07806-5000 or emailed to ardecstdzn@conus.army.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.daps.dla.mil

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DEPARMENT OF DEFENSE STANDARDS

MIL-STD-650	-	Explosive: Sampling Inspection and Testing
MIL-STD-1168	-	Ammunition Lot Numbering and Ammunition Data Card
MIL-STD-1916	-	DoD Preferred Methods for Lot Acceptance

(Copies of this document are available online at https://assist.daps.dla.mil/quicksearch/ or from the Standardization Document Order Desk, 700 Robbins Avenue, Building 4D, Philadelphia, PA 19111-5094.)

2.2.2 Other Government documents, drawings, and publications. The following other Government documents, drawings, and publications form a part of this document to the extent specified herein. Unless otherwise specified, the issues are those cited in the solicitation.

NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY

NIST SRM 136	-	National Institute of Standards and Technology, Standard Reference Material, Potassium Dichromate

(Copies of this document are available online at http://www.nist.gov/index.html.)

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.

AMERICAN SOCIETY FOR TESTING AND MATERIALS INTERNATIONAL

ASTM E11	-	Standard Specification for Woven Wire Test Sieve Cloth and Test Sieves
ASTM E70	-	Standard Test Method for pH of Aqueous Solutions With the Glass Electrode
ASTM E300	-	Standard Practice for Sampling Industrial Chemicals
ASTM E323	-	Standard Specification for Perforated-Plate Sieves for Testing Purposes

(Copies of ASTM standards may be ordered online at http://www.astm.org or from ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959)

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 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.

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3. REQUIREMENTS

3.1 Requirement inspections.

3.1.1 First article. When specified (see 6.3), a sample shall be subjected to first article inspection in accordance with 4.2.

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

3.2 Appearance. Normal lead styphnate shall be free of visible impurities or foreign materials.

3.3 Crystal form. Normal lead styphnate shall be crystalline. The longest dimension of any crystal shall not be greater than three times the next longest axis. Hexagonal plates shall be acceptable.

3.4 Crystal dimension. Unless otherwise specified, the largest dimension of any normal lead styphnate crystal or aggregate of crystals shall not be greater than 0.5 mm.

3.5 Chemical and physical characteristics. The chemical and physical characteristics of normal lead styphnate shall conform to the requirements given in TABLE I.

TABLE I. Chemical and physical characteristics.

Characteristics	Requirement
Granulation	100% through U.S. standard # 100 sieve
Granulation (alternate method), microns	99% smaller than 176.0 microns 100% smaller than 250.0 microns
Apparent density, gm/cc	1.30 to 1.60
Acidity, pH	5.0 to 6.6
Material insoluble in ammonium acetate, max Percent	0.3
Material soluble in ether, max percent	0.1
Lead, percent	43.2 to 44.3
Nitrogen, percent	8.87 to 9.07
Instantaneous flash point (Differential Scanning Calorimeter, DSC), °C	270 to 290

3.6 Lot numbering. Each lot of lead styphnate shall be identified by a lot number. Lot numbering identification shall be in accordance with MIL-STD-1168.

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4. VERIFICATION

TABLE II. 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 Method
		1	2	3	4	A	B	
3.1.1	First article	X		X	X	X		4.2, TABLE III
3.1.2	Conformance inspection	X		X	X		X	4.3, TABLE IV
3.2	Appearance			X		X	X	4.4.2
3.3	Crystal form			X		X	X	4.4.3
3.4	Crystal dimension			X		X	X	4.4.4
TABLE I	Granulation				X	X	X	4.4.5
TABLE I	Granulation (alternate method), microns			X		X	X	4.4.5.1
TABLE I	Apparent density	X				X	X	4.4.6
TABLE I	Acidity	X				X	X	4.4.7
TABLE I	Material insoluble in ammonium acetate	X				X	X	4.4.8
TABLE I	Material soluble in ether	X				X	X	4.4.9
TABLE I	Lead	X				X	X	4.4.10
TABLE I	Nitrogen	X				X	X	4.4.11
TABLE I	Instantaneous flash point (DSC)			X	X	X	X	4.4.12
3.6	Lot numbering			X		X	X	4.4.13

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

a. First article inspection (see 4.2)
b. Conformance inspection (see 4.3)

4.2 First article. When specified, a sample shall be subjected to first article verification in accordance with TABLE III.

4.2.1 First article quantity. The first article sample shall consist of 500 g of normal lead styphnate. The first article inspection shall be performed on the quantities listed in TABLE III.

4.2.2 Inspection to be performed. The first article inspection shall be performed in accordance with TABLE III.

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4.2.3 First article rejection. If any item of the sample fails to comply with the first article requirements, the first article shall be rejected.

TABLE III. First article inspection.

Examination or Test	Sampling Quantity	Requirement Paragraph	Inspection Method Reference
Appearance	1 g	3.2	4.4.2
Crystal form	1 g	3.3	4.4.3
Crystal dimensions	1 g	3.4	4.4.4
Granulation	30 g	TABLE I	4.4.5
Granulation (alternate method)	1 g	TABLE I	4.4.5.1
Apparent density	2 g	TABLE I	4.4.6
Acidity	1 g	TABLE I	4.4.7
Material insoluble in ammonium acetate	1 g	TABLE I	4.4.8
Material soluble in ether	2 g	TABLE I	4.4.9
Lead	0.55 g	TABLE I	4.4.10
Nitrogen	0.8 g	TABLE I	4.4.11
Instantaneous flash point (DSC)	.5 mg	TABLE I	4.4.12
Lot numbering	1/	3.6	4.4.13
Notes: 1/ The first article sample shall be manufactured and assigned a first article lot number. All raw materials used in producing the normal lead styphnate shall be from the same sources as will be used in regular production.			

4.3 Conformance inspection.

4.3.1 Lot formation. Lot formation shall be in accordance with the lot formation requirements of MIL-STD-1916, paragraph Formation and identification of lots and batches. A lot shall consist of one or more batches not exceeding 226.5 kg (500 pounds) produced by one manufacturer, in accordance with the same specification revision, under one continuous set of operating conditions.

4.3.2 Inspection by classification of characteristics. Known characteristics are classified in TABLE IV. The definitions of critical, major and minor defects are provided in paragraph “Definitions” of MIL-STD-1916.

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TABLE IV. Conformance inspection classification of characteristics.

	Chemical and physical characteristics			
Classification	Examination or Test	Conformance Criteria	Requirement Paragraph	Inspection Method Reference
Critical	None defined			
Major				
101	Appearance	4.3.3.2	3.2	4.4.2
102	Crystal form	4.3.3.2	3.3	4.4.3
103	Crystal dimensions	4.3.3.2	3.4	4.4.4
104	Granulation	4.3.3.3	TABLE I	4.4.5
105	Apparent density	4.3.3.2	TABLE I	4.4.6
106	Acidity	4.3.3.2	TABLE I	4.4.7
107	Material insoluble in ammonium acetate	4.3.3.3	TABLE I	4.4.8
108	Material soluble in ether	4.3.3.3	TABLE I	4.4.9
109	Lead	4.3.3.3	TABLE I	4.4.10
110	Nitrogen	4.3.3.3	TABLE I	4.4.11
111	Instantaneous flash point (DSC)	4.3.3.3	TABLE I	4.4.12
Minor				
201	Lot numbering	100%	3.6	4.4.13
Notes:				

4.3.3 Testing. (see 6.6)

4.3.3.1 Sampling. Obtain two random 30 g samples from each batch of normal lead styphnate being submitted for inclusion in a lot. The samples shall be obtained from the process vessel prior to pack out using procedures described in ASTM E300 for slurries.

4.3.3.2 Testing for batch acceptance. One sample obtained according to 4.3.3.1 from each batch shall be tested in accordance with applicable test methods for conformance with requirements of appearance (see 3.2), crystal form (see 3.3), crystal dimension (see 3.4), apparent density (see TABLE I), and acidity (see TABLE I). If any sample fails to meet any of these requirements, the batch represented by the sample shall be rejected.

4.3.3.3 Testing for lot acceptance. The second samples from the batches not rejected under provisions of 4.3.3.2 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 g each shall be obtained from

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this composite sample. Both sub-samples shall be tested in accordance with the applicable test methods of 4.4 to determine conformance with the requirements of granulation (see TABLE I), material insoluble in ammonium acetate (see TABLE I), material soluble in ether (see TABLE I), lead content (see TABLE I), nitrogen content (see TABLE I), and instantaneous flash point (DSC) (see TABLE I). If either sub-sample fails to meet any of the test requirements, the lot represented by the composite sample shall be rejected.

4.4 Methods of inspection. All tests described in this section shall be performed using prescribed procedures.

4.4.1 Preparation of dry sample. Transfer a portion of the wet sample containing about 10 g of lead styphnate to a Buchner funnel containing a medium texture filter paper and apply suction until sample is almost dry. While working behind a safety shield, spread the sample over a watch glass. Dry the sample by placing the watch glass in a desiccator over drying agent for 24 hours at ambient temperature or in an oven no higher than 75ºC until constant weight is obtained (this will take at least 2 hours). Use this dry sample for all determinations unless otherwise specified.

4.4.2 Appearance. Take a portion of the well stirred wet sample from 4.3.3.1 and spread over an area of approximately 4 cm^2 on a glass microscope slide. Allow the sample to air dry. Examine the material microscopically for impurities using magnification of 50x, under illumination. Any sample containing visible impurities or foreign matter shall be rejected. Retain the slide for the determination of crystal form (see 4.4.3) and crystal dimensions (see 4.4.4).

4.4.3 Crystal form. Using the slide prepared in 4.4.2, observe the crystalline form (shape) under a microscope using a magnification of l00x under reflected light, and optionally, take a photomicrograph at 50x magnification. Any sample containing crystal form with the longest dimension greater than three times the next longest axis shall result in rejection of the batch.

4.4.4 Crystal dimensions. Using the slide prepared in 4.4.2, measure the longest dimension of the larger crystals observed when scanning the field with the aid of a mechanical stage. Use the most accurate means available to determine the longest crystal dimension. Consider the longest dimension to be the distance between the most remote points of any one crystal.

4.4.5 Granulation. Transfer about 30 g of the wet sample to a Buchner funnel and apply suction for 2 to 3 minutes. Weigh out a 10 g portion of the damp sample and transfer it to a clean three inch US Standard #100 Sieve which conforms to the requirements of ASTM E11 and ASTM E323. 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 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 lead styphnate has passed through the sieve, run a gentle stream of water through the sieve while brushing the residue gently with a soft natural hair brush until all in the sample that will pass through the sieve has passed through it. Behind a safety shield, visually examine the screen of the sieve for the presence of lead

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styphnate, at the completion of this operation. Thoroughly rinse the brush to ensure that lead styphnate particles are removed. After the final rinse, a small presence of visible lead styphnate on the sieve shall be cause for rejection of the lot.

4.4.5.1 Granulation (alternate method). Determine lead styphnate particle size distribution using a Microtrac II or equivalent commercially available laser particle size analyzer which has been calibrated using appropriate procedures. Follow sample preparation and instrument operating procedure from the analyzer manufacturer. Only distilled or de-ionized water shall be allowed to be used as liquid medium, if a liquid medium is used to "wet" and disperse a powdered sample. No vigorous physical action, which may cause particle size attrition, shall be performed on the sample prior to loading into the instrument. Analyzer shall be configured to measure longest axis in the sample.

4.4.6 Apparent density. Place 3 mL of n-butyl alcohol into a 5 mL graduate which has an internal diameter of about 7.5 mm and is graduated in 0.05 mL divisions. Add approximately 2 g of the dry sample weighed to the nearest 1 mg in small portions and wash down the walls of the graduate with n-butyl alcohol. To insure that the entire sample is wetted, shake the graduate carefully by slowly inverting it at least five times. Adjust the volume by adding butyl alcohol to the 5 mL mark. Allow the graduate to stand for 3 hours and note the volume occupied by the solid material. Calculate the apparent density as follows:

$$\text{Apparent density} = \frac{W}{V}$$

where: W = weight of sample, g
V = observed volume, mL

4.4.6.1 Apparent density (alternate method). Transfer approximately 2 to 3 g of the wet sample obtained in 4.3.3.1 into a Buchner funnel fitted with medium porosity filter paper. Rinse the wet sample with denatured ethyl alcohol to displace water. Rinse the sample with n-butyl alcohol to displace the denatured ethyl alcohol. Transfer the sample to a 5 mL 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. Record this weight as W_1. Allow the cylinder contents to settle for a minimum of 2 hours, then note volume 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) to the nearest 1 mg. Record this weight as W_2. 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

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4.4.7 Acidity. Transfer approximately 1 g of the dry sample weighed to the nearest 1 mg to a 150 mL beaker. Add 100 mL of freshly boiled and cooled distilled water having a pH of 6.0 ± 0.5 and allow the mixture to stand with occasional stirring for 15 minutes. Filter and determine the pH of the filtrate at 25° ± 2°C by means of a pH meter that is capable of measuring pH within 0.1 pH unit. The pH meter shall have glass and calomel electrodes and shall be calibrated with standard buffer solution. Test another 100 mL portion of the water concurrently with the sample solution and correct the pH of the sample solution for any change in pH found in this blank solution.

4.4.7.1 Acidity (alternate method). Transfer approximately 50 mL of the water covering the sample obtained in 4.3.3.1 to a 200 mL beaker. Determine the pH value of this solution in accordance with method given in ASTM E70.

4.4.8 Material insoluble in ammonium acetate. Transfer approximately 1 g of the dry sample, weighed to the nearest 1 mg, to a 250 mL beaker. Add 100 mL of distilled water and then add slowly with constant stirring 100 mL of 20 percent ammonium acetate solution. Stir frequently for 15 minutes and filter the solution through a tared sintered-glass crucible of fine porosity. Transfer the residue to the crucible with distilled water and wash the crucible with distilled water until the washings are colorless. Dry in an oven at 100°C to 105°C for 1 hour, cool in a desiccator, and weigh. Calculate the percent material insoluble in ammonium acetate as follows:

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

where: A = weight of residue, g
W = weight of sample, g

4.4.9 Material soluble in ether. Transfer approximately 2 g 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:

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

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

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4.4.10 Lead. Transfer approximately 0.45 to 0.55 g 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 shall 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 it stand for at least 2 hours. Filter through tared sintered porcelain crucible of fine porosity. Transfer the precipitate to the crucible with 5 percent sulfuric acid and twice with 95 percent ethyl alcohol. Dry in an oven at 100°C to 105°C for 1/2 hour and then ignite in a muffle furnace at 550°C to 600°C for 15 minutes. Cool in a 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, g
W = weight of sample, g

4.4.11 Nitrogen.

4.4.11.1 Apparatus. Storage and titration system are as shown in Figures 1 and 2.

4.4.11.2 Reagents.

4.4.11.2.1 Potassium dichromate solution (0.200 N). Dry standard potassium dichromate (National Institute of Standards and Technology [NIST] reference material 136) in an oven at 110°C for 2 hours, cool, dissolve 9.8070 g in distilled water, and dilute to 1 liter in a volumetric flask. This is a primary standard.

4.4.11.2.2 Acetic acid solution (25 percent). Add 250 mL of glacial acetic acid to 750 mL of distilled water.

4.4.11.2.3 Titanous chloride solution (0.2N). This solution can be prepared from either 20 percent titanous chloride solution or from titanium hydride.

4.4.11.2.3.1 Preparation from 20 percent titanous chloride solution. Filter about 150 mL of 20 percent titanous chloride solution through a glass filtering crucible. Add 100 mL of concentrated hydrochloric acid and mix by means of a current of inert gas (oxygen-free carbon dioxide or nitrogen). Add 750 mL of oxygen-free distilled water and again mix by means of a current of inert gas. Store in an amber bottle under carbon dioxide supplied by a Kipp generator

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as shown in figure 1. The solution need not be standardized, as it is compared with the standard ferric ammonium sulfate each time an analysis is made.

4.4.11.2.4 Ammonium thiocyanate solution (20 percent). Dissolve 20 g of NH_4CNS in 80 g of distilled water.

4.4.11.2.5 Sodium acetate solution (20 percent). Dissolve 20 g of CH_3COONa in 80 g of distilled water.

4.4.11.2.6 Stannous chloride solution. Dissolve 50 g of $SnCl_2$ $2H_2O$ in 100 mL of concentrated hydrochloric acid and dilute to 1 liter with distilled water.

4.4.11.2.7 Diphenylamine sulfonate indicator solution. Add 0.095 g of the barium salt of diphenylamine sulfonic acid to 100 mL of distilled water and stir until solution is complete. Add 10 mL of dilute sulfuric acid (1 to 1), let stand several hours, and filter. Add 500 mL of 85 percent phosphoric acid and dilute to 1 liter.

4.4.11.2.8 Ferric ammonium sulfate solution (0.2N). Dissolve approximately 100 g of ferric ammonium sulfate ($FeNH_4(SO_4)_2 \cdot 12H_20$ in one liter of 5 percent sulfuric acid. The normality is determined as follows: Pipet 25 mL of the ferric ammonium sulfate solution into a 500 mL wide mouth Erlenmeyer flask and add 25 mL of concentrated hydrochloric acid. Heat nearly to boiling point and add the stannous chloride solution drop-wise and with stirring until the yellow color of ferric chloride is discharged and then add 1 drop stannous chloride solution in excess (no more). Cool and add 10 mL of a saturated mercuric chloride solution. Dilute to about 200 mL and occasionally shake for 5 minutes. Add 15 mL diphenylamine sulfonate indicator solution and titrate with a standard 0.2000 N potassium dichromate solution. The change at the end point is from light green to purple. The normality of the ferric ammonium sulfate solution is calculated as follows:

$$\text{Normality of ferric ammonium sulfate solution} = \frac{A \times N}{B}$$

where: A = potassium dichromate solution, mL.
N = normality of potassium dichromate solution.
B = ferric ammonium sulfate solution, taken by titration, mL.

4.4.11.3 Procedure. Transfer approximately 0.6 to 0.8 g of the dry sample weighed to nearest 0.1 mg to a 100 mL beaker. Add 50 mL of 25 percent acetic acid solution and stir to dissolve. Transfer to a 250 mL volumetric flask and dilute to the mark. Attach the source of inert gas (oxygen-free carbon dioxide or nitrogen) to the titration flask as shown in Figure 1 and allow the gas to flow for 5 minutes to displace the air. Continue the flow of gas during the entire titration procedure. Pipet a 25 mL aliquot of the solution of the sample into the titration flask (Figure 2). Add 30 mL of 20 percent sodium acetate solution and 20.00 mL of 0.2 N titanous chloride solution and swirl the flask for about 20 seconds. Add 25 mL of 15 percent hydrochloric acid. Titrate with ferric ammonium sulfate solution until the purple color begins to fade, then add 5 mL of 20 percent ammonium thiocyanate solution and continue the titration to a faint permanent pink. A blank run shall be made using the quantities of reagents as above, but

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omitting the sample. This will provide a value for the amount of ferric ammonium sulfate equivalent to the 20.00 mL of titanous chloride solution. Calculate the percent nitrogen in the sample as follows:

$$\text{Percent nitrogen} = \frac{(A - B) \times 0.00233N \times 1000}{W}$$

where: A = ferric ammonium sulfate solution for the blank, mL.
B = ferric ammonium sulfate solution for the sample, mL.
N = normality of ferric ammonium sulfate solution.
W = weight of sample, g.

4.4.11.4 Nitrogen (alternate method). Determine the nitrogen content of the lead styphnate in accordance with Method 409.1 of MIL-STD-650.

4.4.12 Onset of Decomposition. Determine the onset of exotherm peak temperature using any commercially available differential scanning calorimeter, or other suitable thermal analyzer. Set the ramp rate at 5ºC per minute and the starting temperature at 200ºC. The sample cell of the instrument shall be at ambient temperature when loading the sample into it. Remove the excess water from a sample of lead styphnate with a Whatman 40 ashless filter paper or equivalent. Using the minimum amount of sample required for the instrument being used, typically 0.1 – 0.5 mg, load the damp lead styphnate into the cell. Initiate the run. Upon completion of the run, record the temperature at the maximum point of the exotherm. The accuracy and precision of the instrument shall be periodically verified by means of standards that melt above and below the specification range (indium and zinc are suggested). The test shall be performed in triplicate with no greater than ± 10ºC variation between runs.

4.4.13 Lot numbering. Visually verify that an ammunition lot number has been applied IAW MIL-STD-1168.

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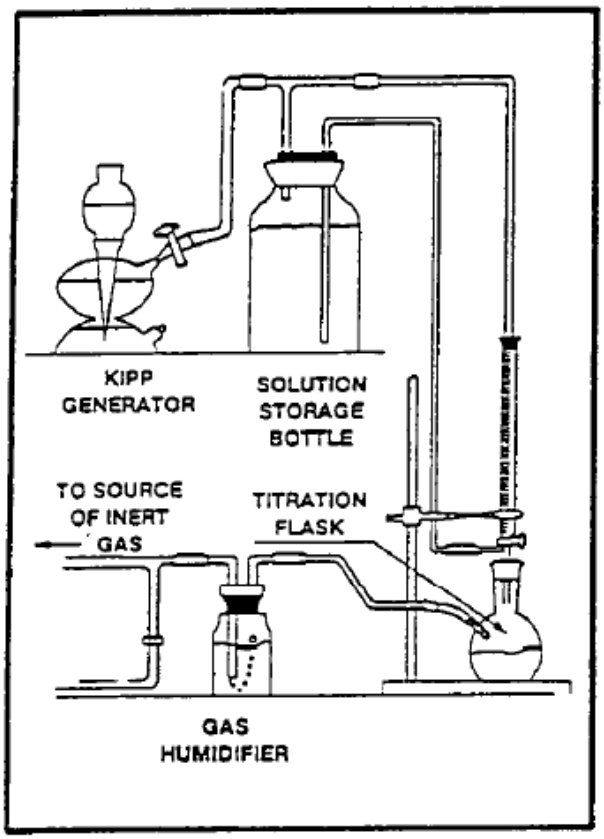

Figure 1. Storage system.

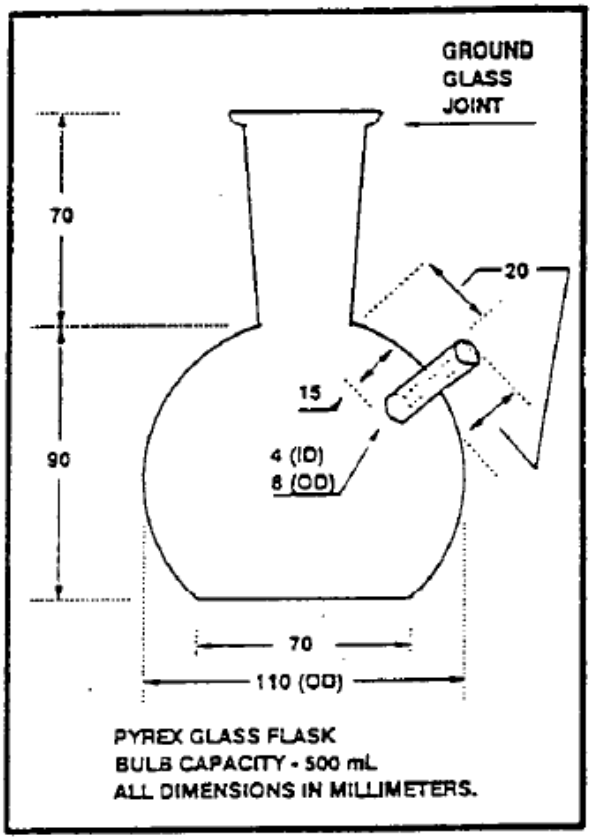

Figure 2. Titration flask.

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 material 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 activity 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 general or explanatory nature that may be helpful, but is not mandatory.)

6.1 Intended use. Normal lead styphnate, lead 2, 4, 6 tri-nitro-resorcinate ($C_6H(NO_2)_3O_2Pb \cdot H_2O$), described in this specification is an explosive intended for use in priming compositions. Normal lead styphnate is classified as a primary high explosive. (All necessary precautions in the manufacture, handling and storage of such materials should be observed.)

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6.2 Acquisition requirements. Acquisition documents should specify the following:

a. Title, number and date of this detail specification.
b. First article requirements (see 3.1.1, 4.2 and 6.4).
c. Level of packaging 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 4.2.1. 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, should 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, U. S. Army ARDEC, ATTN: RDAR-QEM-D, Picatinny Arsenal, NJ 07806-5000. This address will be specified on the Contract Data Requirements List, DD Form 1423 in the contract.

6.4.1 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 should have the equipment available, utilize it in accordance with the test method, and is responsible to assure that it is properly calibrated. Approval of all contractor designed inspection equipment is required prior to its use for acceptance testing (see 6.5).

6.4.2 Submission of contractor test. The POP tests (see 6.10.8) should be submitted to the Commander, U.S. Army ARDEC. ATTN: RDAR-EIL-P. Picatinny Arsenal, New Jersey 07806-5000. (Note: If a POP test report is prepared against an acceptable analogy, the analogy POP test report should also be submitted to RDAR-EIL-P.

6.5 Equivalent test methods. The contractor may request to use alternate test method(s) providing that the proposed method is as a minimum 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, RDAR-QEM-D, 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 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 should be followed in handling and processing.

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6.7 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.

6.8 Acceptance and description sheets. Acceptance and description sheets should be submitted IAW MIL-STD-1171 Acceptance and Description Sheets for Propellants and Explosives.

6.9 Submission of alternative quality conformance provisions. All contractor proposed alternative quality conformance provisions should be submitted to the Government for evaluation/approval by the technical activity responsible for the preparation of this specification.

6.10 Packing. Normal Lead Styphnate should be packaged and shipped in a wet condition as directed in the Code of Federal Regulations CFR 49, for manufacturers within the United States. For foreign manufacturers, all transportation regulations should be in accordance with the Foreign National Transportation Authority and the Recommendations on the Transport of Dangerous Goods, as published in document ST/SG/AC.10/1, current revisions, prepared by the U.N. Committee of Experts on the Transport of Dangerous Goods. A mixture of water plus denatured ethyl alcohol in such proportions to prevent freezing should always be used unless otherwise directed.

6.10.1 Level A. Up to 25 lbs. (dry weight) of Normal Lead Styphnate should be packed wet (50/50 solution of water/denatured ethanol) directly into a cambric diaper or similar cloth bag. A cap of the same diameter and fabric as the bag should be placed inside the bag and over the Normal Lead Styphnate. The bag should then be tied closed and placed inside a bag made of conductive velostat. This velostat bag should then be filled with 50/50 water/denatured ethanol solution. 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 materials and of the same diameter as the duck bag should be placed over the velostat bag. The duck bag is then securely tied closed. This bag is now the unit bag which should be marked in accordance with 6.10.5. An appropriate number of bags (whose combined total dry weight of Normal Lead Styphnate should not exceed 150 lbs.) are placed into a rubber cloth bag. Inside this bag and over the unit of bags of Normal Lead Styphnate should be placed a cap of the same fabric and same diameter as this bag. This rubber cloth bag should then be filled with a 50/50 water/denatured ethanol solution so that the unit bags inside are completely covered. The bag is then securely tied closed. This rubber bag is placed inside a strong grain bag which must be securely tied closed. The grain bag and its contents must be packed in the center of an open head steel drum conforming to UN 1A2, standards for Steel Drums, which has been lined with a heavy, close fitting jute bag. The grain bag should be entirely surrounded by not less than 3 inches of well-packed sawdust saturated by the previously mentioned water/alcohol mixture. The jute bag should then be closed by secure sewing to prevent the escape of the sawdust. The dry weight of Normal Lead Styphnate in one outside container should not exceed 150 lbs.

6.10.2 Level B. Level B should be the same as Level A.

6.10.3 Level C. Level C should be the same as Level A.

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6.10.4 Marking. Marking should be in accordance with MIL-STD-129.

6.10.5 Unit packaging. Each unit bag as defined in 6.10.1 should be marked in contrasting color to the bag with letters not less than ¼ inch or greater than ½ inch high with water/alcohol proof ink as follows:

a. Normal Lead Styphnate
b. Quantity (dry weight) in pounds
c. Lot number
d. Batch numbers

6.10.6 Alternative. As an alternative, each unit bag may be identified by means of a waterproof tag imprinted with a water/alcohol-proof ink with the above information and attached with a non-metallic tie.

6.10.7 Exterior container. The side and top of each drum or barrel should be marked: "LEAD STYPHNATE, WETTED, UN0130 – DANGEROUS – DO NOT STORE OR LOAD WITH ANY HIGH EXPLOSIVE" in ½ inch high letters. The following additional information will be marked on the side of each drum or barrel in letters not less than ¼ inch or greater than ½ inch high:

a. NSN
b. Normal Lead Styphnate
c. Quantity (dry weight) in pounds
d. Lot number
e. Gross weight
f. MIL-DTL-757

6.10.8 Performance oriented packaging. Prior to shipment, the contractor should ensure either that the container has been tested by either a U.S. Government approved Performance Oriented Packaging (POP) test laboratory or by the government itself in compliance with POP requirements in accordance with Title 49, Code of Federal Regulations. Test will be to a weight at least 10% greater than the actual gross weight or the certification weight specified on the packaging drawings, (whichever is higher) which should be marked on the tested container. POP marking, however, should not be applied to the container until verified by the U.S. Government as follows:

a. The POP test report should be prepared by the POP test laboratory in accordance with DI-PACK-81059 (Data Item Description) following the test.

b. The report must be kept on file by the contractor and must also be submitted to the Army (ARDEC Packaging Div) for verification as directed in the contract data requirements list (CDRL) DD Form 1423.

The US Army will review the report, prepare an endorsement letter, and submit the package to the Defense Logistics Agency (DLA).

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For multi-year contracts, the contractor should re-perform POP testing at a certified POP test laboratory if:

a. The initial POP test report expires before the end of the contract (single pack POP certification is valid for a period of 1 year; combination pack POP certification is valid for a period of 2 years) or

b. There is a change in container manufacturer, design, or manufacturing processes of the exterior shipping container.

No re-test is needed if all packaging is purchased while under an unexpired POP test.

EXCEPTION TO POP MARKINGS: If the container is manufactured outside the USA, the contractor should not apply the UN POP certification mark provided in this contract (if applicable). The contractor/container manufacturer (outside the USA) is responsible to perform the UN POP certification tests and apply the marking authorized by the Transportation Competent Authority of the country of manufacture. The contractor should also provide a copy of the POP Test Report to the U.S. Army (ARDEC Packaging Div) for concurrence prior to shipment.

6.11 Subject term (key word) listing.

Lead 2, 4, 6-tri nitro resorcinate.
Primer
Primary high explosive

6.12 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 – 70

Preparing activity:
Army-AR
(Project 1376-2010-005)

Review activities:
Army – TE
Navy – AS, NP
Air Force – 11, 99

Civil agency:
GSA – FAS

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.daps.dla.mil