MILITARY SPECIFICATION

LEAD STYPHNATE, NORMAL (COMMERCIAL GRADE)

1. SCOPE

1.1 Scope. - This specification governs the manufacture and preparation for delivery of normal lead styphnate and the methods of inspection and tests upon which acceptance shall be based.

2. APPLICABLE SPECIFICATIONS, STANDARDS, DRAWINGS, AND PUBLICATIONS

2.1 The following specifications, standards, drawings, and publications, of the issue in effect on date of invitation for bids, form a part of this specification:

SPECIFICATIONS

FEDERAL

RR-S-366 - Sieves; Standard, Testing

NAVY DEPARTMENT

General Specifications for Inspection of Materials

STANDARDS

MILITARY

MIL-STD-129 - Marking of Shipments

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

2.2 Other publications. - The following publication, of the issue in effect on date of invitation for bids, forms a part of this specification:

Code of Federal Regulations 49CFR 71.1, Title 49, Transportation, Chapter 1, Interstate Commerce Commission, Parts 71-78: Explosives and Other Dangerous Articles.

(Copies of 49CFR 71.1 are obtainable from the Superintendent of Documents, Government Printing Office, Washington 25, D.C., price $3.75.)

3. REQUIREMENTS

3.1 Color and appearance. - The normal lead styphnate shall be yellow-brown to brown in color and shall be free from visible impurities.

3.2 Crystal form. - The material shall consist of discrete crystals in the form of hexagonal plates.

3.3 Particle size. - The normal lead styphnate shall pass completely through a U. S. Standard No. 100 sieve.

3.4 Apparent density. - The apparent density of the normal lead styphnate shall be within the limits of 1.40 gms./ml. to 1.60 gms./ml.

3.5 Material insoluble in ammonium acetate. - The maximum amount of material in the normal lead styphnate which is insoluble in ammonium acetate shall not exceed 0.2 percent.

3.6 Material soluble in ether. - The ether soluble material present in the normal lead styphnate shall not exceed 0.1 percent.

3.7 Acidity. - The pH of a 1 percent suspension of normal lead styphnate in water shall be within the limits of 5.0 to 7.0.

3.8 Instantaneous flash point. - The normal lead styphnate shall have an instantaneous flash point within the limits of 310°C to 330°C.

3.9 Lead content. - The lead content of the normal lead styphnate shall be within the limits of 43.2 to 44.3 percent.

3.10 Nitrogen content. - The nitrogen content of the normal lead styphnate shall be within the limits of 8.87 to 9.07 percent.

4. SAMPLING, INSPECTION, AND TEST PROCEDURES

4.1 Sampling. -

4.1.1 Lot. - A lot shall consist of not more than 500 pounds of normal lead styphnate (dry weight).

4.1.2 Sampling procedures. - By means of a horn spoon, sufficient material to form a primary sample of approximately 100 grams (dry weight) shall be removed from each barrel or drum in the lot. Primary samples shall consist of equal portions taken from each bag in the barrel or drum. Each primary sample shall be blended on a smooth surface by mixing with a horn spatula. Water shall be added in case the sample appears to be dry. The sample shall be spread out and divided into cubes approximately 1/2-inch on a side by means of the spatula. Small portions shall be taken from each cube to make a retained portion of the primary sample of approximately 20 grams (dry weight). The retained portion of each primary sample, thoroughly wetted, shall be transferred to a smooth-necked bottle, the bottle tightly stoppered with a rubber stopper, and the stopper held in place by means of an adhesive tape. The bottle shall be so labeled that the barrel or drum from which the sample was taken can be easily identified. The remaining portions of all the primary samples shall be thoroughly blended, spread out, marked into approximate 1/2-inch cubes and small portions taken from each cube until a composite sample of approximately 50 grams dry weight is obtained. This composite sample, thoroughly wetted, shall be transferred to a smooth-necked bottle, the bottle tightly stoppered with a rubber stopper, and the stopper held in place by means of an adhesive tape. The bottle shall be labeled to show the name of the material, manufacturer, plant, contract or order number, lot number, and the number of pounds in the lot. Acceptance tests shall be made on the composite sample representative of the lot. The approximate 20 gram primary samples shall be held for possible future examination should the composite sample fail to meet the requirements of this specification.

4.2 Inspection procedures. - For Naval purchases the general inspection procedures shall be in accordance with General Specifications for Inspection of Material.

4.3 Tests. -

4.3.1 Microscopic examination. -

4.3.1.1 Color and appearance. - Transfer a portion of about 10 grams of the wet sample as received to a Buchner funnel and apply suction until the sample is almost dry.

Spread the sample on a watch glass and place in a desiccator over concentrated sulfuric acid for 24 hours or in an oven at 70 - 75°C for at least four hours or until constant weight is obtained. Use this sample for all determinations where a dry sample is required. Take four portions of about 0.5 mg. each from different places in the sample and spread them over an area of about 2 cm. square on a glass microscope slide. Examine the material macroscopically and also microscopically with reflected light using a magnification of about 30 times.

4.3.2 Particle size. - Place approximately 10 grams of wet normal lead styphnate on a No. 100 U.S. Standard sieve and wash by lifting and lowering the screen in a container of water. After most of the material has passed through, wash the remainder with a gentle stream of water played over the screen.

4.3.3 Apparent density. - Place 3 ml. of n-butyl alcohol into a 5 ml. graduate having an internal diameter of 7.5 mm and graduated in 0.05 ml. divisions. Add 2.00 grams (dry weight) of normal lead styphnate in small portions and wash down the walls of the graduate with butyl alcohol. To insure that all the sample is wetted, shake the graduate carefully by slowly inverting it a number of times. Adjust volume by adding butyl alcohol to the 5 ml. mark. Allow the graduate to stand for three hours and note the volume occupied by the normal lead styphnate.

$$\text{Apparent Density} = \frac{\text{Sample weight in grams}}{\text{Observed volume in ml.}}$$

4.3.4 Material insoluble in ammonium acetate. - Transfer an accurately weighed portion of about 1.0 gram of dry sample to a 250 ml. beaker. Add 100 ml. of distilled water and then, with constant stirring slowly add 100 ml. of a 20 percent ammonium acetate solution. Stir the mixture until no further solution of the sample is apparent. Filter the solution through a tared, fritted glass filtering crucible of porosity F. Wash all particles from the beaker into the crucible with a jet of distilled water from a wash bottle. Wash the crucible until the wash water comes through colorless. Dry the crucible and contents in an oven at 100° to 105°C for 40 minutes. Cool in a desiccator and weigh. Calculate the gain in weight as percent material insoluble in ammonium acetate.

4.3.5 Material soluble in ether. - Transfer an accurately weighed portion of about 2.0 grams of sample to a 150 ml. beaker. Add 50 ml. of anhydrous ethyl ether and allow the mixture to stand with occasional stirring for about 15

minutes. Decant the ether into a funnel containing a dry No. 41 Whatman paper or equivalent filter paper and catch the filtrate in a tared 100 ml. beaker. Wash the residue in the beaker and funnel twice with 10 ml. portions of ether. Evaporate the ether on a steam bath or by means of a current of dry air. Bring the beaker and contents to a constant weight in a vacuum desiccator containing sulfuric acid. Calculate the gain in weight, after correcting for the blank determined on 70 ml. of anhydrous ethyl ether, as percent material soluble in ether.

4.3.6. Acidity. - Transfer 1.00 gram of sample 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 which reads directly in pH units and is capable of measuring accurately within 0.1 pH unit. The pH meter shall be equipped with a glass electrode and a saturated calomel electrode. It shall be calibrated with standard buffer solutions.

4.3.7 Instantaneous flash point. - The instantaneous flash point of the material shall be determined by placing samples of the size that may be held on about 1/16 inch of the flat end of an ordinary toothpick onto a heated Dennis melting point bar and determining the temperature at which the explosion is instantaneous. This point is determined by noting the times required for the explosion of samples dropped on the bar at several temperatures slightly lower than the instantaneous flash point. Then, by plotting a time-temperature curve and extrapolating to zero time, the instantaneous flash point is obtained.

4.3.8 Lead content. - Weigh out samples of 150 to 175 mgs. into 250 ml. beakers and moisten with a few ml. of distilled water. Add 15 ml. of concentrated nitric acid (Sp.gr. 1.42). Heat gently until all material is in solution and then dilute to 125 ml. Heat the solution to about 80°C and place on an electrolytic apparatus. Keep the solution hot during electrolysis. Electrolyze at 0.5 ampere for one hour using a platinum gauze rotating anode to receive the lead dioxide. To test for completeness of deposition, add enough distilled water to cover fresh electrode surface and after a few minutes note whether any further deposition occurs on the fresh surface. When deposition of lead dioxide is complete, raise the electrodes (with the current still on) from the solution, simultaneously rinsing them down with a gentle stream of water. Cut off the current and rinse the anode in 95 percent ethyl alcohol. Allow to air dry and then weigh. Obtain the weight of lead dioxide by subtracting

the weight of the anode.

$$\frac{\text{gm. of } PbO_2 \text{ x } 86.62}{\text{sample weight}} = \text{percent lead}$$

4.3.9 Nitrogen content. - Nitrogen is calculated on the basis of a determination of nitro groups by reduction with titanous chloride solution. The following solutions are required:

4.3.9.1 Ferric ammonium sulfate, 0.2N. - Dissolve 100 grams of ferric ammonium sulphate in one liter of 5 percent sulfuric acid (by volume). This solution may be standardized as follows: Pipette 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 to near boiling and add stannous chloride solution (50 gm. of $SnCl_2.2H_2O$ in 100 ml. hydrochloric acid and dilute to 1 liter with distilled water) dropwise and with stirring until the yellow color of ferric chloride is discharged. Avoid more than one drop excess. Cool the solution to room temperature and add 10 ml. of a saturated solution of mercuric chloride in water. Dilute to 200 ml. and let stand, with occasional shaking, for about five minutes. Titrate with a standard 0.2N potassium dichromate solution prepared from National Bureau of Standards Sample 136. Use 15 ml. of the indicator solution as prepared in 4.3.9.3. The change at the end point is from light green to purple. The normality of the ferric ammonium sulfate solution is given by

$$\frac{\text{ml. of } K_2Cr_2O_7 \text{ x } 0.2}{25}$$

One ml. of 1 N ferric ammonium sulfate is equivalent to 0.00233 gram of nitrogen.

4.3.9.2 Titanous chloride, 0.2N. - Filter about 150 ml. of a 20% titanium trichloride solution through a glass filtering crucible. Add 100 ml. of concentrated hydrochloric acid 36 percent and mix with a rapid current of carbon dioxide. Add 750 ml. of distilled water and again mix with the current of carbon dioxide. The solution should be stored under carbon dioxide supplied by a Kipp generator. The solution need not be standardized, as it is compared with the standard ferric ammonium sulfate solution each time a group of samples is run.

4.3.9.3 Diphenylamine sulfonate indicator. - Weigh out 0.095 gram of the barium salt of diphenylamine sulfonic acid and add it to 100 ml. of water. Stir until solution

is complete. Add 10 ml. of dilute sulfuric acid (H_2SO_4;1:1). Let stand several hours and then filter. Add 500 ml. of 85 percent phosphoric acid and dilute to 1 liter. Use 15 ml. for each titration.

4.3.9.4 Ammonium thiocyanate, 20 percent - Dissolve 25 grams of ammonium thiocyanate in 100 ml. of distilled water.

4.3.9.5 Procedure. - Weigh out 0.60 to 0.80 gram of sample into a 100 ml. beaker . Add 50 ml. of 25 percent acetic acid solution to dissolve the material and transfer the solution to a 250 ml. volumetric flask. Make up to volume and mix thoroughly. Transfer a 25 ml. portion of the solution to a 500 ml. widemouth Erlenmeyer flask having an atmosphere of carbon dioxide. The flask carries a No. 8 rubber stopper with a glass tube extending to the bottom. A large hole in the stopper allows insertion of the buret tip and escape of carbon dioxide gas which is bubbled through during the titration. Add 30 ml. of a 20 percent sodium acetate solution, followed by 20 ml. of a 0.2N titanous chloride solution. Mix the solution by swirling the flask for about 20 seconds. Add 25 ml. of 15 percent hydrochloric acid and titrate with ferric ammonium sulfate solution. When the purple color begins to fade, add 5 ml. of 20 percent ammonium thiocyanate. Continue the titration to a faint permanent pink. A blank run following the above procedure should be made in which the same quantities of reagents are used but the sample is omitted. This provides a value for the amount of ferric ammonium sulfate equivalent to the 20 ml. of titanous chloride. Calculate as follows:

$$\frac{(A-B) \times 0.00233N \times 1000}{\text{sample weight}} = \text{percent nitrogen}$$

A = ml. of ferric ammonium sulfate in blank run

B = ml. of ferric ammonium sulfate in sample run

N = normality of ferric ammonium sulfate

5. PREPARATION FOR DELIVERY

5.1 Packing. - Normal lead styphnate shall be packed with not less than 20 percent by weight of a solution of denatured ethyl alcohol and water containing not less than 50 percent of denatured ethyl alcohol, and in this wet condition shall be packed in bags made of rubberized cloth, each bag containing approximately 25 pounds (dry weight) of normal lead styphnate. There shall be placed in each bag over the lead styphnate a cap of the same cloth and of the diameter of the bag. The bag

shall be tied securely. Not more than five of these bags shall be placed in a larger bag made of the same rubberized material. This large bag shall be tied securely. The large rubberized cloth bag shall contain not more than 150 pounds (dry weight) of normal lead styphnate. This bag shall be placed in the center of a container complying with Interstate Commerce Commission Container Specifications 5 or 5B (metal barrels or drums), 17E (metal drums, single trip) or 10B (wooden barrels or kegs), such container to be lined with a heavy, close fitting bag made of jute, or of other suitable bag material of equal strength. The large rubberized bag shall be completely surrounded within the jute bag, by not less than three inches of well packed sawdust saturated with the same 50 percent solution of denatured ethyl alcohol and the jute bag shall be closed by secure sewing to prevent escape of sawdust. The outer container (drum or barrel) shall be watertight.

5.2 Marking. - Marking to insure safe handling shall conform to Interstate Commerce Commission Regulations for Transportation of Explosives and Other Dangerous Articles, etc. In addition to any special marking required by the contract or order, shipments shall be marked in accordance with MIL-STD-129. In addition, each container shall be plainly marked - "WET NORMAL LEAD STYPHNATE - DANGEROUS - DO NOT STORE OR LOAD WITH ANY HIGH EXPLOSIVE."

6. NOTES

6.1 Ordering data. - Invitation for bids, contracts or other purchasing documents should specify number, title and date of the specification. The point of inspection if other than specified in 4.2 should be stated.

Notice. - When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data, is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation, or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.