MIL-L-17699(NOrd)
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MILITARY SPECIFICATION

LEAD 2-ETHYL HEXOATE

1. SCOPE

1.1 Scope. - This specification covers one grade of lead 2-ethyl hexoate intended for various military applications.

2. APPLICABLE SPECIFICATIONS, STANDARDS, AND PUBLICATIONS

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

SPECIFICATIONS

NAVY

General Specifications for Inspection of Material.

STANDARDS

MILITARY

MIL-STD-129 - Marking of Shipments

(Copies of specifications and standards 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 - Transportation, Interstate Commerce Commission, Explosives and Other Dangerous Articles.

(Copies of the Code of Federal Regulations may be obtained upon application, accompanied by money order, coupon, or cash, to the Superintendent of Documents, Government Printing Office, Washington 25, D.C. Prices may be obtained from the Superintendent of Documents.)

3. REQUIREMENTS

3.1 Moisture and volatiles. - Maximum, 2.0 percent.

3.2 Acidity and alkalinity. -

3.2.1 Alkalinity. - None.

3.2.2 Acidity to phenolphthalein. - Maximum 1.2 percent as 2-ethyl hexanoic acid.

3.3 Total lead content. - Maximum, 42.5 percent; minimum, 40.5 percent.

3.4 Viscosity. - Minimum of Z-8 (Gardner-Holdt).

3.5 Diethylphthalate insoluble material. - Maximum, 1.0 percent.

3.6 Color. - Not darker than 7 (Gardner Standard, 1933).

4. SAMPLING, INSPECTION, AND TEST PROCEDURES

4.1 Lot. - Unless otherwise specified a lot shall consist of not more than 10,000 pounds.

4.2 Sampling. - Ten percent but in no case more than 10 or less than 3 of the containers shall be selected from each lot by the inspector so as to be representative of the lot. If there are fewer than 3 containers in the lot, all the containers shall be sampled. The material shall be thoroughly mixed and approximately 8 ounces of it shall be taken from each selected container. Each of these primary samples shall be placed in an airtight container and labelled so that the container from which it was taken can be identified. A composite sample of approximately 8 ounces shall be made from equal portions of the primary samples. The composite sample shall be thoroughly mixed and placed in an airtight container labeled to show the name of the material, manufacturer, plant, contract or order number, lot number and lot size. All acceptance tests shall be made on the composite sample. However, if it becomes apparent during

sampling that the lot is not uniform, the inspector may require that any primary sample be tested for compliance with the requirements of the specification. All primary samples shall be held for possible future examination should the composite sample fail to meet the requirements.

4.3 Inspection. - Unless otherwise specified in the contract or order, inspection shall be made at the point of delivery.

4.4 Tests. -

4.4.1 Moisture and volatiles. - Transfer a portion of about 10 gm. of the material to a tared glass dish about 5 cm. in diameter and about 2 cm. deep and accurately weigh the dish and contents. Place in an oven maintained at 100° to 105°C for 2 hours, cool in a desiccator and weigh. Calculate the loss in weight as percent moisture and volatiles.

4.4.2 Acidity and alkalinity. -

4.4.2.1 Alkalinity. - Transfer a weighed portion of about 20 gm. of the sample to a 400 ml. beaker and add 40 ml. of neutral 95-percent ethyl alcohol. Stir the mixture thoroughly for at least 2 minutes with a stirring rod. Add 200 ml. of cold, freshly boiled distilled water to the beaker and stir the mixture vigorously for about 2 minutes. Filter the mixture through a suitable funnel containing a No. 41 Whatman, or equivalent, filter paper. Wash the insoluble matter and filter paper with two 50 ml. portions of distilled water, make up the combined filtrate and washings to 500 ml. in a volumetric flask and then divide it into two equal portions. To the first portion add five drops of phenophthalein indicator and note the color of the solution. Consider the alkalinity to be none if the solution does not turn pink on addition of the phenolphthalein. If the solution does not turn pink on addition of phenolphthalein, determine the acidity to phenolphthalein as specified in 4.4.2.2.

4.4.2.2 Acidity to phenolphthalein. - If the solution to which phenolphthalein has been added as specified in 4.4.2.1 remains colorless, titrate with N/10 sodium or potassium hydroxide solution to a light pink end point. Calculate the percent acidity as follows:

Percent acidity to phenolphthalein (as 2-ethyl hexanoic acid) = $\frac{14.4\ VN}{W}$

where

V = ml. of N/10 sodium or potassium hydroxide solution used

N = normality of sodium or potassium hydroxide solution

W = gm. of sample represented by the aliquot taken

4.4.3 Total lead content. -

4.4.3.1 Reagents. -

1. Glacial acetic acid, C.P., lead content not greater than 0.00005 percent.

2. Acetic acid, approximately 70 percent by weight obtained by diluting C.P. acetic acid with distilled water.

3. Nitric acid, C.P., lead content not greater than 0.00002 percent.

4. Sulfuric acid, C.P., lead content not greater than 0.001 percent.

5. Sulfuric acid, 3N (about 15 percent or 1:11 dilution).

6. Ethyl alcohol, 50 percent.

7. Ethyl alcohol, 95 percent.

4.4.3.2 Procedure. -

1. Accurately weigh a 0.4000-0.5000 gm. sample into a 50 ml. beaker or onto a watch glass and transfer it quantitatively to a 400- or 500 ml. tall-form beaker, using the acetic acid in step 2 below.

2. Add 70-percent acetic acid as needed to total 50 ml., 20 ml. of concentrated nitric acid, and 10 ml. of concentrated sulfuric acid. (Perform in a hood.)

3. Cover the beaker with a ribbed watch glass and heat on a 3-heat electric plate (a 7-inch diameter, 1200 watt or an 8½ inch diameter, 2000-watt unit is satisfactory) at low heat for 20 minutes.

4. Add 10 ml. concentrated nitric acid and increase the heat to medium. Continue heating until copious white fumes of sulfur trioxide appear. Use "high" heat if "medium" seems too slow when the bulk of the nitric acid has been expelled.

5. Remove the beaker from the hot plate and allow it to cool for 5-10 minutes in order to avoid spitting or too rapid evolution of gases on the addition of acid. Then add 5 ml. of concentrated nitric acid and reheat until fumes of sulfur trioxide reappear.

6. Repeat the cooling, adding nitric acid and reheating until the solution when "fumed" is colorless or very pale yellow. Then "fume" strongly for 10 minutes.

7. Remove the beaker and allow it to cool thoroughly.

8. Cautiously add 50 ml. of distilled water, washing down the cover glass and the sides of the beaker.

9. Boil the solution vigorously for 2 or 3 minutes.

10. Allow it to cool thoroughly and add about 15 ml. of 95 percent ethyl alcohol.

11. Let the solution stand at room temperature for about 2 hours.

12. Filter the solution by suction through a tared, ignited Gooch crucible with a good asbestos mat.

13. Using 5 washings, of about 15-20 ml. each of 3 N sulfuric acid, carefully transfer the lead sulfate precipitate from the beaker to the crucible (this is a dense material and care is required in making a good quantitative transfer).

14. Continue washing the lead sulfate in the Gooch crucible. Use 2 washes with 50 percent alcohol and one with 95 percent alcohol.

15. Dry the crucible by suction for about 5 minutes and then complete the drying in an oven for 15-30 minutes at 105-110°C.

16. Ignite the crucible in a furnace at 500-600°C. for 15 minutes. (Alternatively, ignition may be accomplished by placing the Gooch crucible inside a large porcelain crucible which is directly fired in the full blast of a Fisher burner. With this type of equipment ignite for 5 minutes. The usual crucible used is a Coors No. 3).

17. Cool the Gooch crucible in a desiccator and reweigh.

18. Calculate the percent total lead by multiplying the weight of lead sulfate obtained by 68.32 and dividing by the weight of sample used. The mean of duplicate determinations shall be reported.

4.4.4 Viscosity. - Fill a standard Gardner viscosity tube so that the size of the air bubble approximates that of the bubbles in the standard tubes. Bring the sample and standard tubes to a temperature of 25°C (77°F) by immersing them in a water bath. Start the bubbles to rising from the flat end of the tubes. Use the bottom meniscus of the bubbles for purposes of comparison. Owing to unavoidable differences in bubble sizes and in the shapes of the tubes, it may be necessary to raise one of the tubes so that the points of comparison will be on the same level. The observation is made after the bubbles are completely formed which takes place about 1 1/2 - 2 cm. from the bottom of the tube. The tubes may be held vertically or inclined not less than 45 degrees but all tubes must be held at the same angle. The rate of rise of the bubble for the sample shall not be faster than that of the Z-8 standard tube.

4.4.5 Diethylphthalate insoluble material. - Transfer about 2.0 g. of the material to a suitable tared flask or large test tube and weigh to get the exact weight of the sample. Add 18.0 ml. of C.P. diethylphthalate and mix well. Immerse the test tube in a water bath held at 70°C ± 1°C for 30 minutes; mix occasionally to insure complete dissolution of the material. Cool the test tube and contents to room temperature. The solution should appear to be substantially clear, not opaque. Filter through a tared medium porosity pyrex crucible using suction. Rinse the test tube three times with 5 ml. portions of diethylphthalate, pouring the rinsings through the crucible. Wash the crucible contents twice with 5 ml. portions of 95 percent ethanol and aspirate to air dry

the residue. Place the crucible and contents in a 110°C oven for one hour. Cool in a desiccator and weigh. The weight of residue multiplied by 100 and divided by the weight of sample will give the percent insoluble in diethylphthalate.

4.4.6 Color. -

4.4.6.1 Apparatus. -

a. Gardner Color Standards (1933), Nos. 1 to 18 and rack.

b. Gardner Color Tubes (10.75 mm. inside diameter).

4.4.6.2 Procedure. - Fill a standard Gardner Color Tube (10.75 mm. inside diameter) to the mark with the sample. Place in the rack and compare by daylight with the standard colors. The sample is rated by the number of the Gardner Color Standard which it most nearly matches. If the color is between that of two standards, it is rated plus or minus the standard nearest to it in color.

4.5 Resubmission and retests. - If the composite sample, or any primary sample subjected to test, fails to pass the tests, the lot shall be rejected. The contractor shall then have the option of having a partial or complete analysis made on samples from any or all of the containers in the lot at no expense to the government. The contractor may then remove the defective portions of the lot and resubmit the lot for acceptance, provided complete replacement of the defective portions can be made to the satisfaction of the inspector. The resubmitted lot shall be accepted, provided that new samples, selected in accordance with 4.2 pass all the tests required by this specification.

5. PREPARATION FOR DELIVERY

5.1 Packaging and packing. - Unless otherwise specified, lead 2-ethyl hexoate shall be packaged and packed in substantial commercial containers so constructed as to insure acceptance by common or other carrier for safe transportation at the lowest rate to the point of delivery.

5.2 Marking. - In addition to any special marking required by the contract or order, shipments shall be marked in accordance with Standard MIL-STD-129. Each container shall be labeled "Lead 2-Ethyl Hexoate" and all markings shall conform with the applicable regulations of the Interstate Commerce Commission.

6. NOTES

6.1 Ordering Data. - Procurement documents should specify the number, the date, and the title of the specification.

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.