

MIL-L-757A
1 September 1967
SUPERSEDING
SEE SECTION 6

MILITARY SPECIFICATION

LEAD STYPHNATE, NORMAL

1. SCOPE

1.1 This specification covers normal lead styphnate for use in lead styphnate priming compositions.

2. APPLICABLE DOCUMENTS

2.1 The following documents of the issue in effect on date of invitation for bids or request for proposal, form a part of this specification to the extent specified herein:

SPECIFICATION

FEDERAL

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

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

2.2 Other publications. The following document forms a part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on date of invitation for bids or request for proposal shall apply.

FSC 1376

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INTERSTATE COMMERCE COMMISSION

49 CFR 71-90 - Rules and Regulations of the Transportation of Explosives and Other Dangerous Articles.

(Application for copies should be addressed to the Superintendent of Documents, Government Printing Officer, Washington, D. C., 20402. Orders should cite "49 CFR 71-90".)

3. REQUIREMENTS

3.1 Color and appearance. The material shall be yellow-brown to brown in color and shall be free from visible impurities and aggregates of crystals.

3.2 Crystal form. The material shall consist of the discrete crystals of the monoclinic system in which the longest axis is not greater than three times that of the next longest axis. Hexagonal plates are acceptable.

3.3 Crystal dimension. The largest dimensions of any crystal shall not exceed 0.5 mm (see 6.6).

3.4 Granulation. One hundred percent shall pass through a U. S. Standard sieve No. 100.

3.5 Chemical and physical properties. Chemical and physical properties shall be as indicated in table I.

Table I. Chemical and physical properties

Properties	Requirements
Apparent density, gm/cc	1.30 to 1.60
Acidity, pH	5.0 to 6.0
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 °C (see 6.6)	310 to 330

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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 all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own facilities or any commercial laboratory acceptable to the Government. The Government reserves the right to perform any of the inspections set forth in the specification where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements.

4.1.1 Contractor quality assurance system. The contractor shall provide and maintain an adequate quality assurance system, acceptable to the Government covering the supplies under the contract. A current written description of the system shall be submitted to the contracting officer prior to initiation of production. The written description will be considered acceptable when, as a minimum, it provides the quality assurance required by this specification. The contractor shall notify the Government of and obtain approval for any change to the written procedure that might affect the degree of assurance required by this specification or other applicable documents referenced herein.

4.2 Lot. A lot shall consist of one or more batches of lead styphnate not exceeding 500 pounds which has been produced by one manufacturer under one continuous set of operating conditions, in accordance with the same specification or same specification revision. A batch of lead styphnate shall be produced from one chemical and/or physical mixing process. Each lot shall be identified with a lot identification number, name of manufacturer, date of manufacture as well as dry weight of lead styphnate contained therein.

4.3 Sampling. Take from each of the barrels or drums in the lot a sample of approximately 100 gm. by hand or with a horn spoon. Place a 30 gm. portion of each of such primary samples in a tightly stoppered bottle so labeled that the barrel from which the sample was taken can be identified. Blend the remaining portions of the primary samples on a smooth surface with a horn spatula. Spread the lead styphnate out and divide into squares about 0.5 inch on a side with the spatula. Remove from all of these squares enough lead styphnate to form a composite sample of approximately 50 gm. Mix this composite sample thoroughly and place it in a tightly stoppered bottle. Label the sample so as to show the name of the material, manufacturer, plant, purchase order, and number of pounds in the lot. All acceptance tests shall be made on the composite sample representative of the lot. However, if it becomes apparent during sampling that the lot is

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not uniform, the inspector may require that any primary sample be tested for compliance with the requirements of the specification. Hold the primary samples for possible future examination should the composite sample fail to meet the requirements. A lot should be rejected if the sample does not pass the requirements given in section 3.

4.4 Inspection. Inspection shall be made at the point of delivery, unless otherwise specified in the contract or order.

4.5 Test methods.

4.5.1 Preparation of dry sample. Transfer a portion of the wet sample containing about 10 gm. 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 concentrated sulfuric acid for 24 hours or in an oven at 70° to 75°C until constant weight is obtained (this will take at least 2 hours). Use this dry sample for all determinations unless otherwise specified.

4.5.2 Color and appearance. Take four portions of about 0.5 mg. each from different places in the dry sample and spread them over an area of about 2 sq. cm. on a glass microscopic slide. Examine the material macroscopically for color and impurities under daylight illumination and microscopically for impurities and aggregates of crystals using a magnification of about 30 times and reflected light.

4.5.3 Crystal form. Select four portions each of approximately 0.5 mg. from different areas of the dry sample and spread over an area of approximately 2 sq. cm. on a glass microscopic slide. Observe the crystalline shape of the material microscopically using a magnification of about 350 times and reflected light.

4.5.4 Crystal dimensions. Using the slide prepared for determining the crystal form, measure the longest dimension of the larger crystals observed when scanning the field with the aid of a mechanical stage. Use transmitted light and an ocular micrometer which has been calibrated by use of a stage micrometer. Consider the longest dimension to be the distance between the two most remote points on any one crystal.

4.5.5 Granulation. Place a portion of the wet sample containing approximately 10 gm. of lead styphnate on a U. S. standard sieve No. 100 (RR-S-366) 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.

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4.5.6 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 2 gm. of the dry sample weighed to 1 mg. in small portions and wash down the walls of the graduate with n-butyl alcohol. To insure that all the 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, gm.
V = observed volume, ml.

4.5.7 Acidity. Transfer 1 gm. of the dry sample weighed to 1 mg. to a 150 ml. beaker. Add 100 ml. of freshly boiled and cooled 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 $25^\circ \pm 2^\circ\text{C}$ 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.

4.5.8 Material insoluble in ammonium acetate. Transfer 1 gm. of the dry sample, weighed to 1 mg., to a 250 ml. beaker. Add 100 ml. of water and then add with constant stirring slowly 100 ml. of 20 percent ammonium acetate solution. Allow to stand for 15 minutes with frequent stirring and filter the solution through a tared sintered-glass crucible of fine porosity. Transfer the residue to the crucible with water and wash the crucible with water until the washings are colorless. Dry in an oven at 100° 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, gm.
W = weight of sample, gm.

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4.5.9 Material soluble in ether. Transfer 2 gm. of the dry sample weighed to 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 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, gm.
 B = weight of residue from blank, gm.
 W = weight of sample, gm.

4.5.10 Lead. Transfer 0.45 to 0.55 gm. of the dry sample weighed to 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. 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 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 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 (or a Gooch crucible with an asbestos mat). Transfer the precipitate to the crucible with 5 percent sulfuric acid. Wash the crucible three times with the 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 desiccator and weigh. Calculate the percent lead in the sample as follows:

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

where: A = weight of ignited residue, gm.
 B = weight of sample, gm.

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4.5.11 Nitrogen.

4.5.11.1 Apparatus. Storage and titration system are as shown in figures 1 and 2.

4.5.11.2 Reagents.

4.5.11.2.1 Potassium dichromate solution (0.2000 N). Dry National Bureau of Standards potassium dichromate (NBS Standard 136) in an oven at 130°C for 2 hours, cool, dissolve 9.8070 gm. in water, and dilute to 1 liter in a volumetric flask. This is a primary standard.

4.5.11.2.2 Acetic acid solution (25 percent). Add 250 ml. of glacial acetic acid to 750 ml. of water.

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

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

4.5.11.2.3.2 Preparation from titanium hydride. Warm 100 ml. of concentrated hydrochloric acid to 20° to 80°C on a hot plate in a well ventilated hood. Add in small portions, 12 gm. of titanium hydride and cover with a watch glass. Remove the watch glass only to add to the solution. Warm until evolution of hydrogen has nearly ceased. Cool to room temperature, add 250 ml. of oxygen-free water, and mix by means of a current of inert gas (oxygen-free carbon dioxide or nitrogen). Filter through a No. 41 Whatman fluted filter paper. If the solution is not clear, refilter through the same paper. Add 100 ml. of concentrated hydrochloric acid and dilute to 1 liter with oxygen-free water. Again mix by means of a current of inert gas. Store in an amber bottle under carbon dioxide supplied by a Kipp generator 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.

4.5.11.2.4 Ammonium thiocyanate solution (20 percent). Dissolve 20 gm. of NH_4CNS in 100 ml. of water.

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4.5.11.2.5 Sodium acetate solution (20 percent). Dissolve 20 gm. of CH_3COONa in 100 ml. of water.

4.5.11.2.6 Stannous chloride solution. Dissolve 50 gm. of $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ in 100 ml. of concentrated hydrochloric acid and dilute to 1 liter with water.

4.5.11.2.7 Diphenylamine sulfonate indicator solution. Add 0.095 gm. of the barium salt of diphenylamine sulfonic acid to 100 ml. of 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.

4.5.11.2.8 Ferric ammonium sulfate solution (0.2N). Dissolve 100 gm. of ferric ammonium sulphate ($\text{FeNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$) in 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 dropwise 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 let stand for 5 minutes with occasional shaking. 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.

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4.5.11.3 Procedure. Transfer 0.6 to 0.8 gm. of the dry sample weighed to 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 should be made using the quantities of reagents as above, but 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, gm.

4.5.12 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 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.

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5. PREPARATION FOR DELIVERY

5.1 Packing. Normal lead styphnate shall be packed wet with not less than 20 percent by weight of water in bags 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), 17H (metal drums, single trip with removable head), 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 3 inches of well packed wood sawdust saturated with water and the jute bag shall be closed by secure sewing to prevent escape of the sawdust. The outer container (drum or barrel) shall be watertight. If the shipment of lead styphnate is to take place at a time when freezing weather is to be anticipated, it must be wet with a mixture of denatured ethyl alcohol and water of such proportions to prevent freezing in transit. Samples shall be packed in accordance with the requirements of 49 CFR 71-90.

5.2 Marking. Each container shall be marked on the upper two-thirds of one surface as follows:

(Qty) WET NORMAL LEAD STYPHNATE
 LOT (Mfr Code and No.)
 PACKED (Month/year)

Marking shall be by stenciling or lithographing using commercial ink in contrasting color to that of the container color. Letters shall be all-upper case and minimum size of letters and numerals shall be 7/16 inch. In addition, each container shall be plainly marked "INITIATING EXPLOSIVE - DANGEROUS - DO NOT STORE OR LOAD WITH ANY HIGH EXPLOSIVES" with 7/16 inch minimum letters.

6. NOTES

6.1 Intended use. Lead styphnate covered by this specification is intended for use in priming compositions.

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6.2 Ordering data. Procurement documents should specify the following:

- (a) Title, number, and date of this specification.
- (b) Packing and packaging required (see 5.1).

6.3 Normal lead styphnate is lead trinitroresorcinate with one molecule of water of crystallization ($C_6H(NO_2)_3O_2Pb \cdot H_2O$).

6.4 Lead styphnate is classified as a high explosive. All necessary precautions in the manufacture, handling, and storage of such a material should be observed.

6.5 Supersession. This specification supersedes JAN-L-757 dated 13 April 1949, MIL-L-17186 dated 20 October 1958, FA-PD-MI-2458 dated 17 March 1961, and PA-PD-160.

6.6 This requirement and corresponding test method can be waived at the discretion of the purchasing agency.

Custodian:

Army - MU
Navy - OS

Preparing activity:

Army - MU

Project No. 1376-0028

Review activities:

Army - MU, MD
Navy - OS

User activities:

Army - MI

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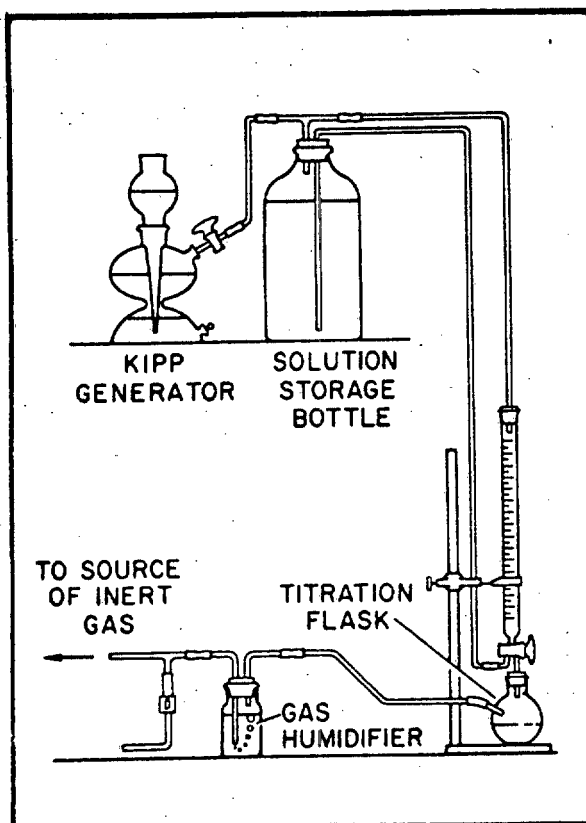


Figure 1. Storage System

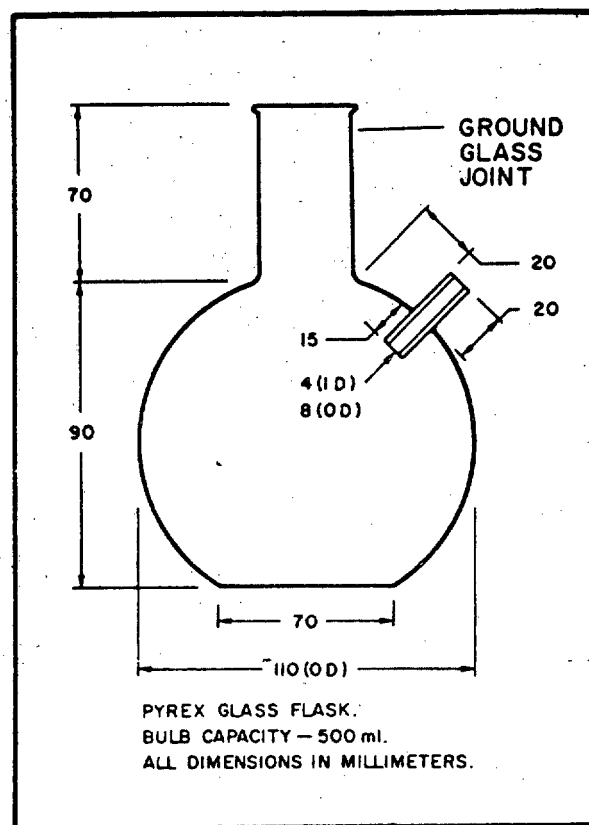


Figure 2. Titration Flask

SPECIFICATION ANALYSIS SHEET		Form Approved Budget Bureau No. 22-R255
INSTRUCTIONS: This sheet is to be filled out by personnel, either Government or contractor, involved in the use of the specification in procurement of products for ultimate use by the Department of Defense. This sheet is provided for obtaining information on the use of this specification which will insure that suitable products can be procured with a minimum amount of delay and at the least cost. Comments and the return of this form will be appreciated. Fold on lines on reverse side, staple in corner, and send to preparing activity. Comments and suggestions submitted on this form do not constitute or imply authorization to waive any portion of the referenced document(s) or serve to amend contractual requirements.		
SPECIFICATION		
ORGANIZATION		
CITY AND STATE	CONTRACT NUMBER	
MATERIAL PROCURED UNDER A <input type="checkbox"/> DIRECT GOVERNMENT CONTRACT <input type="checkbox"/> SUBCONTRACT		
1. HAS ANY PART OF THE SPECIFICATION CREATED PROBLEMS OR REQUIRED INTERPRETATION IN PROCUREMENT USE? A. GIVE PARAGRAPH NUMBER AND WORDING.		
B. RECOMMENDATIONS FOR CORRECTING THE DEFICIENCIES		
2. COMMENTS ON ANY SPECIFICATION REQUIREMENT CONSIDERED TOO RIGID		
3. IS THE SPECIFICATION RESTRICTIVE? <input type="checkbox"/> YES <input type="checkbox"/> NO (If "yes", in what way?)		
4. REMARKS (Attach any pertinent data which may be of use in improving this specification. If there are additional papers, attach to form and place both in an envelope addressed to preparing activity)		
SUBMITTED BY (Printed or typed name and activity - Optional)		DATE

DD FORM 1426

1 JAN 66

REPLACES EDITION OF 1 OCT 64 WHICH MAY BE USED.