18 March 1968

Superseding
JAN-Z-365
11 July 1946

MILITARY SPECIFICATION

ZINC - DUST
(FOR USE IN PYROTECHNICS)

This specification is mandatory for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope - This specification covers one grade of zinc-dust for use in pyrotechnics.

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.

SPECIFICATIONS

Federal

RR-S-366	Sieve, Test

STANDARDS

Military

MIL-STD-105	Sampling Procedures and Tables for Inspection by Attributes

FSC 6810

MIL-STD-129 Marking for Shipment and Storage

(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.)

3. REQUIREMENTS

3.1 Material - Zinc-dust shall be a product of high quality, suitable for the purpose intended, and so manufactured as to meet the requirements specified herein.

3.2 Chemical requirements - Zinc-dust shall conform to the applicable chemical requirements specified in Table I.

Table I
CHEMICAL REQUIREMENTS

Property	Percentage		Test Paragraph
	Maximum	Minimum	
Total zinc, calculated as zinc	———	97.5	4.4.2
Metallic zinc	———	94.0	4.4.3
Zinc oxide	6.0	———	4.4.4
Impurities (other than zinc oxide)	2.0	———	4.4.3.2.2
Moisture and other volatile matter	0.1	———	4.4.5
Matter soluble in organic solvent mixture	0.0	———	4.4.6

3.3 Granulation - Zinc-dust shall conform to the granulation requirements of Table II when determined in accordance with 4.4.7.

Table II
GRANULATION REQUIREMENTS

Sieve number 1/	Percent through, minimum
100	99
200	90
230	75

1/ Use U. S. Standard sieves conforming to FF-S-366

* 3.4 Workmanship - This material shall be uniform in quality, free from foreign material, and shall conform to the requirements of this specification.

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 in the contract or order, the supplier may use his own or any other facilities suitable for the performance of the inspection requirements specified herein, unless disapproved by 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 supplies and services conform to prescribed requirements.

* 4.2 Quality conformance inspection - Conformance of the zinc-dust to the requirements of this specification shall be determined entirely by means of quality conformance inspection. The quality conformance inspection shall consist of an examination for acceptability of quality control methods used by the manufacturer, examining and testing the quality conformance samples (4.3.2) for all of the requirements of this specification, and an examination of the sample of filled containers (4.3.3) for conformance to the packaging, packing, and marking requirements.

* 4.3 Sampling -

4.3.1 Lot size - For the purpose of sampling, a lot of zinc-dust shall consist of a manufacturer's batch (see 6.3). If the material cannot be identified by batch, a lot shall consist of not more than 1,000 pounds of zinc-dust offered for delivery at one time.

4.3.2 Sample for tests - From each lot offered for acceptance under contract, two-one-pound samples of zinc-dust shall be removed from separate unit containers taken at random.

4.3.3 Sample for examination of filled containers - A random sample of filled containers shall be selected from each lot of zinc-dust offered for acceptance under contract, in accordance with MIL-STD-105 at Inspection Level I and an acceptable quality level (AQL) of 2.5 percent defective.

* 4.4 Test methods -

4.4.1 Visual inspection - Conformance of the zinc-dust to the requirements for material (3.1) and workmanship (3.4) shall be determined by visual inspection.

* 4.4.2 Total zinc - Determine total zinc by the potassium ferrocyanide method.

4.4.2.1 Standardization of Potassium Ferrocyanide Solution - Weigh 0.2 gm of metallic zinc or freshly ignited Z_nO, transfer to a 400 ml beaker and disolve in 10 ml of HCl (sp. gr. 1.18) and 20 ml of water. Drop in a small piece of litmus paper, add NH_4OH until slightly alkaline, then add HCl until just acid and then 3 ml more of HCl. Dilute to about 250 ml with hot water and heat to nearly boiling. Run in the $K_4Fe(CN)6$ solution, prepared by dissolving 22 gm of $K_4Fe(CN)_6.3H_2O$ in water and diluting to 1 liter, slowly while stirring constantly until a drop tested on a white porcelain plate with a drop of uranyl nitrate indicator shows a brown tinge after standing 1 minute. The uranyl nitrate indicator is prepared by dissolving 5gm of $UO_2(NO_3)_2.6H_2O$ in 100 ml of water. During the titration the temperature of the solution shall not be allowed to fall below 70°C.

4.4.2.1.1 A blank shall be run using the same amounts of reagents and water, under the same conditions of temperature, volume, acidity as obtained when the standardization sample (see 4.4.2.1) is titrated.

4.4.2.1.2 Calculation - The strength of the $K_4Fe(CN)6$ standard solution in terms of grams of zinc is calculated as follows:

Zinc equivalent in grams per milliter of $K_4Fe(CN)6$ solution

$$= \frac{W}{T_1 - T_2}$$

where

W =weight of zinc sample (or equivalent to the Zn O used)
T_1 =ml x $K_4Fe(CN)6$ used in titration of sample
T_2 =ml $K_4Fe(CN)6$ used in titration of blank

4.4.2.2 Procedure - Weigh accurately 0.25 gm of the powdered zinc, transfer to a 400 ml beaker, moisten with alcohol and dissolve in 10 ml of HCl (sp. gr. 1.18) and 20 ml of water. Continue with the procedure used in 4.4.2.1, beginning with the addition of litmus paper and adjustment of the acidity with NH_4OH and HCl.

4.4.2.3 Calcuation - Calculate the percentage of total zinc by the following formula:

$$\text{Percentage of total zinc} = \frac{(X - X_1)B \times 100}{W}$$

X = ml $K_4Fe(CN)6$ used in titration of sample
X_1 = $K_4Fe(CN)6$ used in titration of blank
B = Zinc equivalent in grams per milliliters of $K_4Fe(CN)6$ solution (see 4.4.2.1.2)

4.4.3 Metallic Zinc - Determine metallic zinc by the hydrogen evolution method.

4.4.3.1 Apparatus - Assemble apparatus as shown on figure 1. Calibrate the bulb D to include capacity from stopcock E to the zero reading of the burette. Also calibrate capacity of flask A and tubes to stopcocks E and B. Saturate the water in leveling bulb F and measuring tube D with hydrogen at room temperature and pressure.

4.4.3.2 Procedure -

4.4.3.2.1 Total Hydrogen evolved - Weigh accurately 1.15 gm of the powdered zinc, transfer to flask A, and about 5.0 gm pure ferrous ammonium sulfate, a piece of sheet platinum having a total about 5 sq cm, and an amount of water exactly 100 ml less than the capacity of the flask and tubes and connect the apparatus as shown on figure 1. Open cocks B and E, raise F to bring the water in the burette to cock E and then close E and B. This leaves 100 ml of air in the apparatus. Read the temperature and barometric pressure and calculate the gas volume to standard temperature and pressure by the following formula:

$$V = \frac{Pv}{760} \quad X \quad \frac{1}{1 + 0.00367t}$$

where

V = corrected volume
v = original volume
P = barometer reading in mm corrected for the vapor pressure of water at t°C.1/
t = temperature in degrees C
0.00367 = coefficient of expansion of hydrogen and air for each degree rise in temperature.

Now pour 43 ml of 1:1 sulfuric acid into the thistle tube, lower F, open cock E and allow the acid to run into flask A partially opening cock B. During the admission of the acid, avoid admission of air. Close cock B and allow the apparatus to stand until the gas volume becomes constant, keeping the level of the liquid in F and D fairly equal throughout the evolution to avoid leakage and hydrogen solubility changes due to pressure differences. The evolution is complete in about 3 hours. Calculate the total volume of gas in the apparatus by adding 60 ml to the gas measuring apparatus reading. This 60 ml is the volume of the gas in the flask A remaining after the addition of 43 ml of acid. (The 43 ml of 1:1 sulfuric acid adds 40 ml to the liquid due to shrinkage of mixing).Read the

1/ In order to evaluate the vapor pressure of water, since A contains acid and the measuring tube contains water, it may be assumed that the entire static system is over water.

temperature and the barometric pressure and correct the total gas volume by the preceding formula for deviations from standard temperature (0°C) and pressure (760 mm). Subtract from the corrected volume the corrected volume of gas originally in the apparatus. Add to the remainder 2.0 ml to correct for the solubility of hydrogen in the solution in flask A. The sum is the hydrogen evolved at standard temperature and pressure.

* 4.4.3.2.2 Impurities - For accurate results, the volume of hydrogen evolved (see 4.4.3.2.1) must be corrected for the hydrogen evolved by iron which is usually present, and for tin which may be present. To make these corrections, assemble apparatus as shown on figure 2.

* 4.4.3.2.2.1 Iron - Weigh accurately and place in flask B, approximately 11.5 gm of the sample, add a piece of sheet platinum an area of about 5 sq cm and close the apparatus tightly. Pass nitrogen through the apparatus until the air is entirely removed. Close pinchcock G and stop the flow of nitrogen. Add 125 ml of water through separatory funnel E to flask B, then add dropwise through the separatory funnel 50 ml of concentrated sulfuric acid. During the addition of the acid, keep the contents of the flask at or below room temperature by means of an ice bath. After the addition of the acid, allow the flask to stand for the length of time required for the hydrogen evolution in the determination of metallic zinc. (This usually requires 3 hours.) Open pinchcock G and draw the contents of flask B through the asbestos mat in tared filter F and into flask B^1 by means of suction applied at H. Wash flask B four times by adding50-ml portions of water, shaking and drawing over into flask B^1. Remove the stopper from flask B^1 and titrate immediately with potassium permanganate solution to a pink color permanent for one minute. Calculate the reducing matter as percentage of iron by the following formula:

$$\text{Reducing matter calculated as percentage of Fe} = \frac{5.584\ AN}{W}$$

where
A = ml $KMnO_4$ used in titration
N = normality of $KMnO_4$ solution
W = weight of zinc-dust sample

4.4.3.2.2.2 - Residue - Complete the washing of the residue in the tared filter F (see 4.4.3.2.2.1) and dry at 105° to 110°C., cool in a desiccator and weigh. Calculate the percentage of residue insoluble in dilute sulfuric acid by the following formula:

$$\text{Percentage of residue} = \frac{100A}{W}$$

where

A = weight of dried residue
W = weight of zinc-dust sample

* 4.4.3.2.2.3 - Tin Transfer the titrated solution from flask B[1] (see 4.4.3.2.2.1) to a 600 ml beaker. Add NH_4OH until just alkaline, then add 5 ml of HCl or 5 ml of H_2SO_4(1:3) for each 100 ml of solution. Cover the beaker, place it on a hot plate, heat to about 95°C, then pass in a slow stream of H_2S for 1 hour, digest for 1 hour and allow to stand one-half hour longer. Filter, and wash the precipitate of SnS alternately with three portions each of wash solution (100 ml of saturated $NH_4C_2H_3O_2$ solution, 50 ml of glacial $HC_2H_3O_2$, and 850 ml of water) and hot water. Transfer the filter paper and precipitate to a 50-ml beaker, add 10 to 20 ml of ammonium polysulfide, heat to boiling, and filter. Repeat twice the digestion with ammonium polysulfide and the filtration, then wash the filter with hot water. Acidify the combined filtrate and washings with $HC_2H_3O_2$ (1:9), digest on a hot plate for one hour, allow to stand overnight, and filter through an ignited, cooled and weighed Gooch crucible with a suitable asbestos pad. Wash alternately with two portions each of the wash solution and hot water and dry thoroughly. Ignite over a Bunsen flame, very carefully at first to convert the sulfide to the oxide; then partly cover the crucible and heat strongly over a large Bunsen or Meker burner. (SnS must be converted carefully to the oxide, which may be heated to a high temperature without loss by volatilization.) Weigh as SnO_2 and calculate percentage of metallic tin by the following formula:

$$\text{Percentage of Sn} = \frac{78.77\ A}{W}$$

where

A = weight of dried residue
W = weight of sample

* 4.4.3.3 Calculations - Multiply the percentage of tin (see 4.4.3.2.2.3) by 0.94 and subtract the result from the reducing matter calculated as percentage of iron (see. 4.4.3.2.2.1). The remainder is percentage of metallic iron. Correct the evolved hydrogen at standard temperature and pressure (see. 4.4.3.2) for the iron and tin present in the sample taken for the determination of metallic zinc on the basis that 0.01 gm metallic iron evolves 4.012 ml of hydrogen and 0.01 gm metallic tin evolves 1.887 ml hydrogen at standard temperature and pressure. Calculate the volume corrected for the iron and tin present as metallic zinc by the following formula:

$$\text{Percentage of metallic zinc} = \frac{0.29188\ M}{W}$$

where

M = ml of hydrogen corrected to standard temperature and pressure
W = weight of sample

The sum of the percentages of residue, iron and tin, equals the percentage of impurties other than zinc oxide.

* 4.4.4 Zinc oxide - Determine zinc oxide by difference between percentage total zinc and percentage metallic zinc

4.4.4.1 Calculation - Calculate the percentage of zinc oxide by the following formula:

Percentage of zinc oxide = (A-B) 1.2447

where

A = Percentage of total zinc
B = Percentage of metallic zinc

4.4.5 Moisture and other volatile matter - Weigh approximately 10.000 gm of the sample in a tared, glass-stoppered weighing bottle, approximately 50 mm in diameter by 30 mm high. Remove the stopper and heat for 2 hours in an oven at 100°C to 105°C. Cool in a desiccator and weigh. Calculate the loss in weight as percentage of moisture.

4.4.6 Matter soluble in organic-solvent mixture - Transfer 25 gm of zinc-dust to a 250-ml beaker, add 100 ml of dry solvent, stir with a glass rod for 2 minutes and filter through a Gooch crucible, prepared with an asbestos pad, into a 200-ml tared beaker. Evaporate the solvent on a steam bath, dry the beaker in an oven at 100° to 105°C. for 1 hour, cool in a desiccator and weigh. One hundred ml of the solvent mixture, when treated as specified, shall leave no weighable residue.

Organic solvent mixture
10 volumes ethyl ether
6 volumes benzene
4 volumes methanol (95-100 percent)
1 volume acetone

The solvent mixture shall be neutral. If it tests either acid or alkaline, it should be purified by shaking with pure zinc-dust, decanting therefrom and distilling.

4.4.7 Granulation - Fit three sieves, Nos. 100, 200, and 230 U. S. Standard series, together in the order of decreasing mesh size placing the coarsest mesh on top and a receiving pan at the bottom. Place a weighed portion of 100.0 gm of the zinc-dust sample on the upper sieve, cover the upper sieve, and shake for 5 minutes by means of a mechanical shaker geared to produce 300 ± 15 gyrations and 150 ± 10 taps of the striker per minute. Weigh the quantity retained on each sieve and calculate the percentage of zinc-dust that has passed through each sieve and checked for conformance to table II.

* 4.5 Rejection criteria - If a sample fails to meet any of the test requirements of this specification, the lot represented by the sample shall be rejected.

5. PREPARATION FOR DELIVERY

* 5.1 Packaging - Unless otherwise specified (see 6.2) zinc-dust shall be packaged at Level C.

5.1.1 Level C - The zinc-dust shall be unit packaged in clean, dry containers of the size and type specified by the purchase order or contract. The zinc-dust shall be packaged to afford the minimum degree of protection necessary to prevent deterioration or damage during shipment under normal environmental conditions and commercial modes of transportation.

* 5.2 Packing - Unless otherwise specified (See 6.2) zinc-dust shall be packed at Level C.

5.2.1 Level C - Zinc-dust packaged in accordance with 5.1.1, that requires overpacking, shall be packed in exterior type containers in such a manner as to insure carrier acceptance and safe delivery to destination. Containers shall be in compliance with rules and regulations applicable to the mode of transportation selected.

* 5.3 Marking - In addition to any markings required by contract or order, unit packages and shipping containers shall be marked in accordance with the requirements of MIL-STD-129.

6. NOTES

6.1 Intended use - Metallic zinc powder, commercially known as zinc dust, covered by this specification, is intended for use in pyrotechnics.

6.2 Ordering data - Procurement documents should specify the following:

(a) Title, number and date of this specification
(b) Quantity required in pounds
(c) Applicable levels of packaging, packing and markings, with requirements in detail, if other than specified in Section 5
(d) Size and type of container (see 5.1.1)

* 6.3 Batch - A batch is defined as that quantity of material which has been subjected to some unit chemical or physical mixing process intended to make the final product substantially uniform.

* 6.4 Metallic zinc - A rapid method for determining metallic zinc is given in ASTM D521, Chemical Analysis of Zinc Dust. The results are inclined to be somewhat low. For the highest accuracy, the hydrogen evolution method detailed in 4.4.3 should be used.

* 6.5 Supersession data - The material furnished under MIL-Z-365A has chemical and physical requirements identical to that furnished under the previous issue, JAN-Z-365, dated 11 July 1946.

* 6.6 Changes from previous issue - The margins of this specification are marked with an asterisk to indicate where changes (additions, modifications, correction, deletions) from the previous issue were made. This was done as a convenience only and the Government assumes no liability whatsoever for any inaccuracies in these notations. Bidders and contractors are cautioned to evaluate the requirements of this document based on the entire content irrespective of the marginal notations and relationship to the last previous issue.

Custodians:

Army - MU
Navy - OS
Air Force - 68

Preparing Activity:

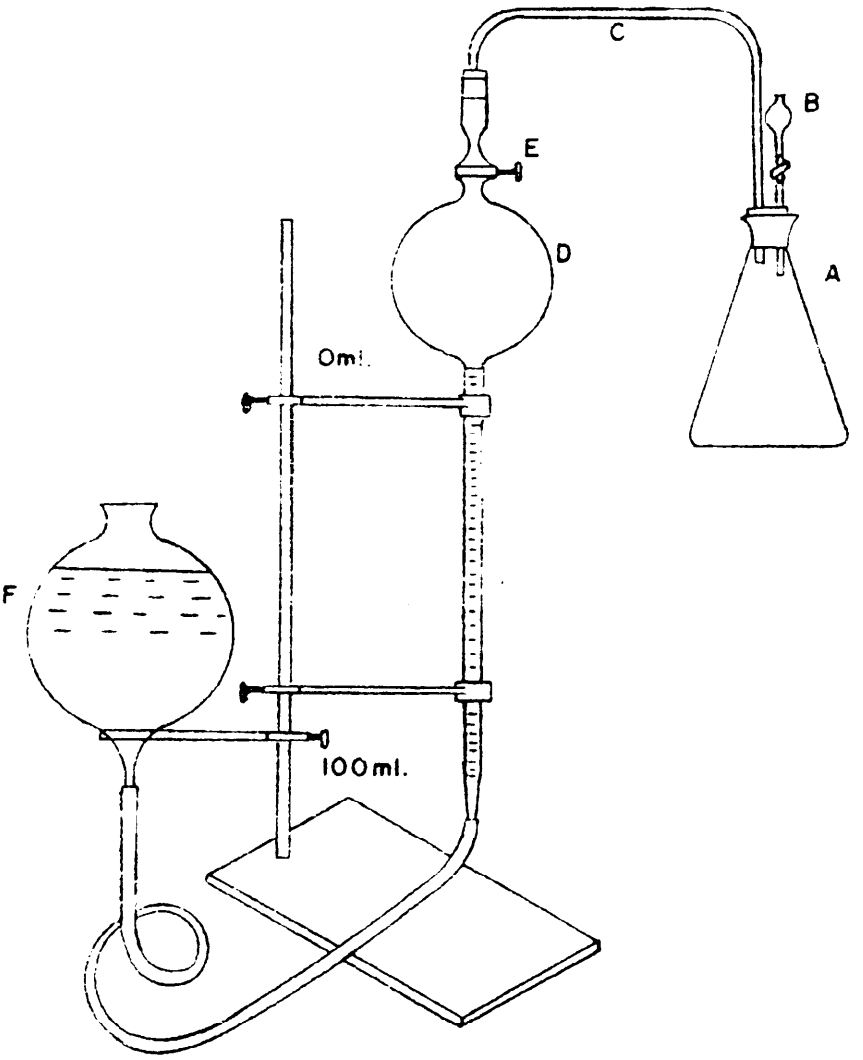
Navy - OS

Project No. 6810-0366

Review activities:

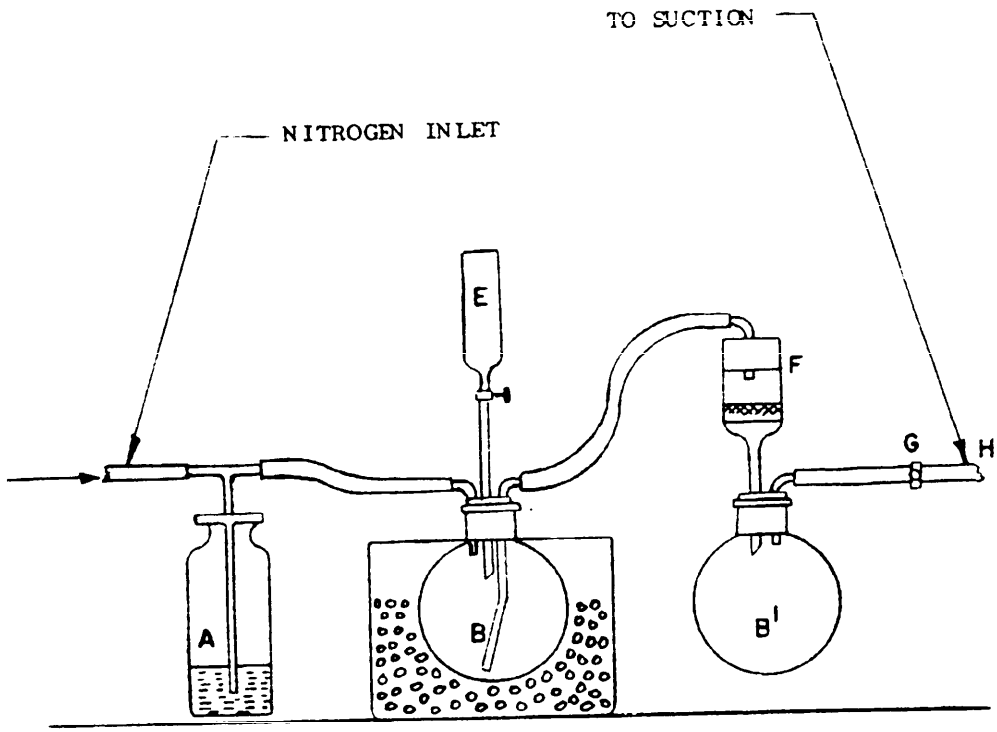
Army - MU, WC
Navy - OS, AS
Air Force - 68
DSA-GS

Army - MI, SM



A- Flask-Actual capacity when filled to stopper 220 ml
B- Thistle tube with stopcock
C- Glass tubing connection
D- Bulb of 400 ml capacity with gas burrette graduated from 0 to 100 ml in 0.2 ml subdivision
E- Stopcock for gas burrette
F- Leveling Bulb

Figure 1--Determination of Metallic Zinc.
Apparatus for the Measurement of Total Hydrogen Evolved.



A -WIDE MOUTH BOTTLE, 16 OUNCES, CONTAINING 2 INCHES OF WATER
B AND B' -ROUND BOTTOM RING NECK FLASKS, 500- ml
E -SEPARATORY FUNNEL, 60-ml, WITH 15-cm STEM
F -FRITTED GLASS FILTER 1 1/2 INCHES IN DIAMETER, PROVIDED WITH AN ASBESTOS MAT.
G -PINCHCOCK

FIGURE 2--DETERMINATION OF METALLIC ZINC. APPARATUS FOR DETERMINATION OF IRON AND TIN.

SPECIFICATION ANALYSIS SHEET

Form Approved
Budget Bureau No. 119-R004

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 (as indicated on reverse hereof).

SPECIFICATION
MIL-S-365

ORGANIZATION (Of submitter) | CITY AND STATE

CONTRACT NO. | QUANTITY OF ITEMS PROCURED | DOLLAR AMOUNT
$

MATERIAL PROCURED UNDER A

☐ DIRECT GOVERNMENT CONTRACT ☐ 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?

☐ YES ☐ 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) | DATE

DD, FORM 1426 APR 61

REPLACES NAVSHIPS FORM 4863, WHICH IS OBSOLETE

S-0270

FOLD

DEPARTMENT OF THE NAVY

NAVAL WEAPONS SERVICES OFFICE
CODE SSS
PHILADELPHIA, PA. 19112

POSTAGE AND FEES PAID
NAVY DEPARTMENT

OFFICIAL BUSINESS

Commanding Officer
Naval Weapons Services Office
Philadelphia, Pa. 19112

FOLD