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MILITARY SPECIFICATION .

SULFUR, GROUND (FOR USE IN AMMUNITION)

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

1. SCOPE

1.1 This specification covers the requirements for ground sulfur.
(see 6.1).

1.2 Classification - The sulfur shall be of the following grades,
as specified (see 6.2 and Table I)

Grade A - Ground crude
Grade B - Ground crude
Grade C - Ground crude
Grade D - Ground refined
Grade E - Ground crude

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

UU-S-48

Sacks, Shipping, Paper

STANDARDS

Military

MIL-STD-105

Sampling Procedures and Tables for
Inspection by Attributes

MIL-STD-129

Marking for Shipment and Storage

FSC-6810

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(Copies of specifications and standards, 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 documents form 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.

American Society-for Testing and Materials Publications
Standard Method of Tests

(ASTM Standards are published by the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pa. 19103.)

American Chemical Society Publications
Reagent Chemicals, A.C.S. Specifications

(Copies of A.C.S. publications may be obtained from the American Chemical Society, 1155 Sixteenth Street, N.W., Washington, 6, D.C.)

Technical Society and technical association specifications and standards are generally available for reference from libraries. They are also widely distributed among technical groups and using Federal agencies.

3. REQUIREMENTS

3.1 Data - No data is required by this specification or by the applicable documents referenced in Section 2 unless specified in the contract or order. (see 6.2).

3.2 Material - Sulfur shall be produced from American natural deposits. Sulfur made from by-products such as pyrites, hydrogen sulfide, or sulfur dioxide is not acceptable.

3.3 Chemical and Physical Properties - When tested as specified in 4.5, the sulfur shall conform to the applicable requirements specified in Table I.

*3.4 Workmanship - The sulfur shall be free from dirt, grease, grit, and any other contamination. Excepting Grade C, the sulfur shall be free from lumps not easily broken up by hand.

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Table I - Chemical and Physical Properties

Requirement, percent by weight	Grade of sulfur					Test method
	A	B	C	D	E	
Sulfur (min)	99.5	99.5	99.5	99.8	99.5	4.5.1
Moisture (max)	0.20	0.10	0.10	0.05	0.10	4.5.2
Acidity, as H ₂ SO ₄ (max)	0.01	0.002	0.002	0.002	0.01	4.5.3
Ash (max)	0.10	0.10	0.10	0.05	0.10	4.5.4
Chlorides, as NaCl (max)	0.01	0.01	0.01	0.01	0.01	4.5.5
Sulfates, as Na ₂ SO ₄ (max)	-	-	-	0.003	-	4.5.6
Particle size: through U. S. Standard Sieve						
No. 100 (min)	98.0	99.5	99.7	99.0	99.0	4.5.7
No. 200	88.0 min.	95.0 min.	97.0 min.	90.0 min.	85.0 to 95.0	
No. 325 (min)			93.0	80.0		
* Ammonia and ammonium salts	-	zero	zero	-	zero	4.5.8

4. QUALITY ASSURANCE PROVISIONS

4.1 Inspection Responsibility - Unless otherwise specified by the Government, 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 examinations and tests set forth in the specification where such are deemed necessary to assure that supplies and services conform to prescribed requirements.

*4.2 Lot Formation - For purposes of sampling, a lot shall consist of a manufacturer's batch or that part of a manufacturer's batch that has been submitted for inspection at one time. A batch is defined as that quantity of sulfur that has been mixed, blended or processed in a single operation intended to make the final product substantially uniform. If the material cannot be identified by batch, a lot shall consist of sulfur of one grade produced by one manufacturer, at one plant, under essentially the same conditions, processed in a continuous operation and submitted for inspection at one time.

*4.3 Quality Conformance Samples - For the sampling procedures specified in 4.3.1 and 4.3.2, the lot size shall be the number of filled shipping sacks or filled bottles in the lot.

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4.3.1 Samples for Tests - A quantity of filled unit containers shall be selected at random from each lot prior to the closure operation in accordance with MIL-STD-105 at inspection level S-4. A representative specimen (taken from two or more places in the container) shall be taken from each of the selected samples. Half of the specimens shall be thoroughly mixed together and placed in a clean, dry container. The other half shall also be thoroughly mixed together and placed in another clean, dry container. Each of these composite samples shall be marked with the product identification, lot number and composite sample number. The sample containers shall be sealed with a tight fitting lid.

4.3.2 Samples of the End Item and Preparation for Delivery - Samples for the inspections specified in 4.4.2.1 through 4.4.2.4 shall be selected at random from each lot in accordance with MIL-STD-105 and Table II. The lot size shall be the number of unit containers for inspections 4.4.2.1 through 4.4.2.3 and the number of shipping containers for inspection 4.4.2.4.

Table II

<u>Inspection</u>	<u>Inspection level</u>	<u>A.Q.L</u>	<u>Sampling unit</u>
End item (4.4.2.1)	S-3	Zero defects	Filled unit container
Filled unit container (4.4.2.2)	S-3	2.5	Filled unit container
Net contents (4.4.2.3)	S-2	see 4.4.2.3	Filled unit container
Shipping containers (4.4.2.4)	II	2.5	Shipping container fully prepared for delivery (just prior to closure)

*4.4 Quality Conformance Inspection -

4.4.1 Inspection of Samples for Tests - Each composite sample selected in accordance with 4.3.1 shall be examined and tested to determine conformance to all the requirements of Section 3 of this specification. Non-conformance of a composite sample to a single requirement shall be cause for rejection of the lot represented by the composite sample.

4.4.2 Inspection of the End Item and Preparation for Delivery - The applicable samples selected in accordance with 4.3.2 and Table II shall be examined for the defects classified in 4.4.2.1 through 4.4.2.4 and to determine compliance with all the other applicable requirements of Section 5 of this specification. If the number of defective samples exceeds the acceptable quality level (AQL) specified in Table II, the lot represented by the sample group shall be rejected.

4.4.2.1 End Item - Examine the end item samples for conformance to the Workmanship requirement 3.4.

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4.4.2.2 Filled Unit Containers -EXAMINEDEFECT

Material

Not material required

Construction

Not size, type or class specified
Closure not as specified
Evidence of leakage

Marking

Warning labels or marking missing.
Marking illegible, incorrect,
incomplete. Not in accordance with
contract requirements.

4.4.2.3 Net Contents - If the average net contents of the samples selected in Table II is less than the amount specified in the contract or order, the lot represented by the sample shall be rejected.

4.4.2.4 Shipping containers fully prepared for delivery -EXAMINEDEFECT

Intermediate Packaging

Not level required by contract or purchase order.
Material or construction not as specified.

Packing

Not level required by contract or purchase order.
Materials or construction not as specified.
Any nonconforming component, incomplete closures, inadequate strapping, bulged or damaged containers.

Count

Less than specified or indicated quantity of primary containers per shipping container.

Markings

Warning labels or marking missing.
Omitted, illegible, incorrect, incomplete or not in accordance with contract requirements.

Shipping containers fully prepared for delivery shall also be examined after closure for closure defects. This inspection, 4.4.2.4, is not necessary if the unit container is also the shipping container.

4.5 Test Methods - Reagent water conforming to ASTM D1193 and reagent chemicals conforming to American Chemical Society (A.C.S) specifications (see 2.2) shall be used throughout the tests. Weighings shall be made to the nearest 0.0001 gram (g). Unless otherwise specified herein, solutions shall be made up on a weight basis.

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4.5.1 Sulfur - Weigh approximately 10g of sample into a beaker and add 100 milliliters (ml) of carbon disulfide. Stir the mixture to dissolve the sulfur. Filter through a tared filtering crucible. Wash the beaker with several portions of carbon disulfide, adding the washings to the crucible. After washing down the sides of the crucible with carbon disulfide, dry the crucible by suction to remove the carbon disulfide. Treat the residue in the crucible with 3 successive 15-ml portions of hot aniline, allowing each portion to filter through slowly. Wash the residue in the crucible with several portions of ethyl alcohol to remove all traces of aniline. Dry the crucible at 100°C for 1 hour; cool in a desiccator and reweigh. Calculate the gain in weight as percent insoluble matter. Calculate percent sulfur as follows:

$$\text{Percent sulfur} = 100.00 - (A+B)$$

where: A = Percent insoluble matter and

B = Percent moisture (see 4.5.2)

*4.5.2 Moisture - Place about 50 grams of sulfur into a tared dry weighting bottle (75 millimeter (mm) diameter by 30 mm high). Record the weight of bottle and sample and place, with lid removed, in an oven maintained at $80 \pm 1^\circ\text{C}$ ($176 \pm 2^\circ\text{F}$) for 16 to 24 hours. Remove from the oven, replace the lid on the bottle, cool in a desiccator and re-weigh. Calculate the loss in weight as percent moisture.

4.5.3 Acidity - Fit the base of the chromatographic tube (see figure 1), equipped with a ground glass joint, into a 500-ml suction flask. Cover the perforations of the base with a plug of absorbant cotton to provide a support for the column of sulfur. Apply suction and add 100 g of the sample. Tamp smooth with a rubber stopper which just fits inside the tube. Apply suction and pass two 100-ml and three 50-ml portions of freshly boiled (see 4.5) water through the column of sulfur. Allow each portion to drain through the column before adding the succeeding portion. Combine the effluents and titrate with 0.1N sodium hydroxide to a methyl red end point. (Retain the titrated solution for the chloride determination, 4.5.5). Make a blank run using the same procedure, without the sample. Calculate as follows:

$$\text{Percent acidity, as sulfuric acid} = \frac{4.904 (A-B) N}{W}$$

where: A = ml of sodium hydroxide for sample.

B = ml of sodium hydroxide for blank.

N = Normality of sodium hydroxide.

W = Weight of sample, in grams.

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*4.5.4 Ash - Ignite a porcelain or platinum crucible for 5 minutes at 900 to 1000°C in an electric muffle furnace. Transfer the crucible to a desiccator and allow to cool. After weighing the crucible, add a 20 to 25 g sample to the crucible and reweigh. Heat the crucible and sample under a hood until the vapors continue to burn after the flame is withdrawn. Allow spontaneous burning until the sample is consumed. Then place the crucible in the 900 to 1000°C muffle furnace for 30 minutes. Remove from the furnace, cool in a desiccator and weigh to the nearest 0.0001 gram. Calculate as follows:

$$\text{Percent ash} = \frac{A-B (100)}{W}$$

where: A = weight of crucible plus ash, in grams.
 B = weight of crucible, in grams.
 W = weight of sample, in grams.

4.5.5 Chlorides - Filter the titrated solution retained from 4.5.3 into a 600 ml beaker. Add 10 ml of concentrated nitric acid and a few crystals of ammonium persulfate. Boil the solution for 5 minutes, cool to room temperature and dilute to 400 ml. To a 40 ml aliquot of this solution in a 6 inch test tube, add 10 drops of a 10 percent silver nitrate solution and mix. Observe the turbidity and compare to that formed when 40 ml of distilled water, 1 ml of concentrated nitric acid, 10 drops of 10 percent silver nitrate and 0.5 ml of a standard Na Cl - distilled water solution (containing 0.0010 gram of Na Cl per ml) are also mixed in a 6 inch test tube.

If the turbidity of the sample is less than that of the standard, report chloride content as less than 0.005 percent. If the turbidity of the sample is greater than the standard, proceed as follows: Combine the sample turbidity solution with the original sample solution. Add 10 ml of 10-percent silver nitrate solution and heat to boiling. Remove the solution from the source of heat and allow the precipitated silver chloride to settle for a minimum of 2 hours. Quantitatively transfer the precipitate to a tared filtering crucible and wash the precipitate with 1-percent nitric acid. Dry at 130° ± 2°C for one hour, cool in a desiccator, and weigh. Calculate chloride content as follows:

$$\text{Percent chlorides, as sodium chloride} = \frac{40.78 (A-B)}{W}$$

where: A = Weight of crucible and precipitate, in grams.
 B = Tare weight of crucible, in grams.
 W = Weight of sample, in grams (equal to the sample weight for acidity test, 4.5.3)

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4.5.6 Sulfates - Repeat the preparation of the hot water extract of a fresh 100 gram sample of sulfur as described in 4.5.3. Transfer the 350 ml hot water extract to a 600 ml beaker, add 5 ml of concentrated hydrochloric acid and heat to a rolling boil. While boiling, add dropwise 10 ml of 5 percent barium chloride solution. After precipitation of the barium sulfate, place the 600 ml beaker on a steam bath for 2 hours. Filter the solution through a tared filtering crucible, quantitatively transferring the barium sulfate precipitate to the crucible with a stream of hot distilled water. Then wash the sides of the filtering crucible and the precipitate five times with hot distilled water. After drying the crucible and precipitate in an oven at 105°C to 110°C ignite at a cherry red heat for 15 minutes. Cool in a desiccator and weigh. Calculate sulfate content as follows:

$$\text{Percent sulfate, as sodium sulfate} = \frac{60.86 (A-B)}{W}$$

where: A = Weight of crucible and precipitate, in grams.

B = Tare weight of crucible, in grams.

W = Weight of sample, in grams

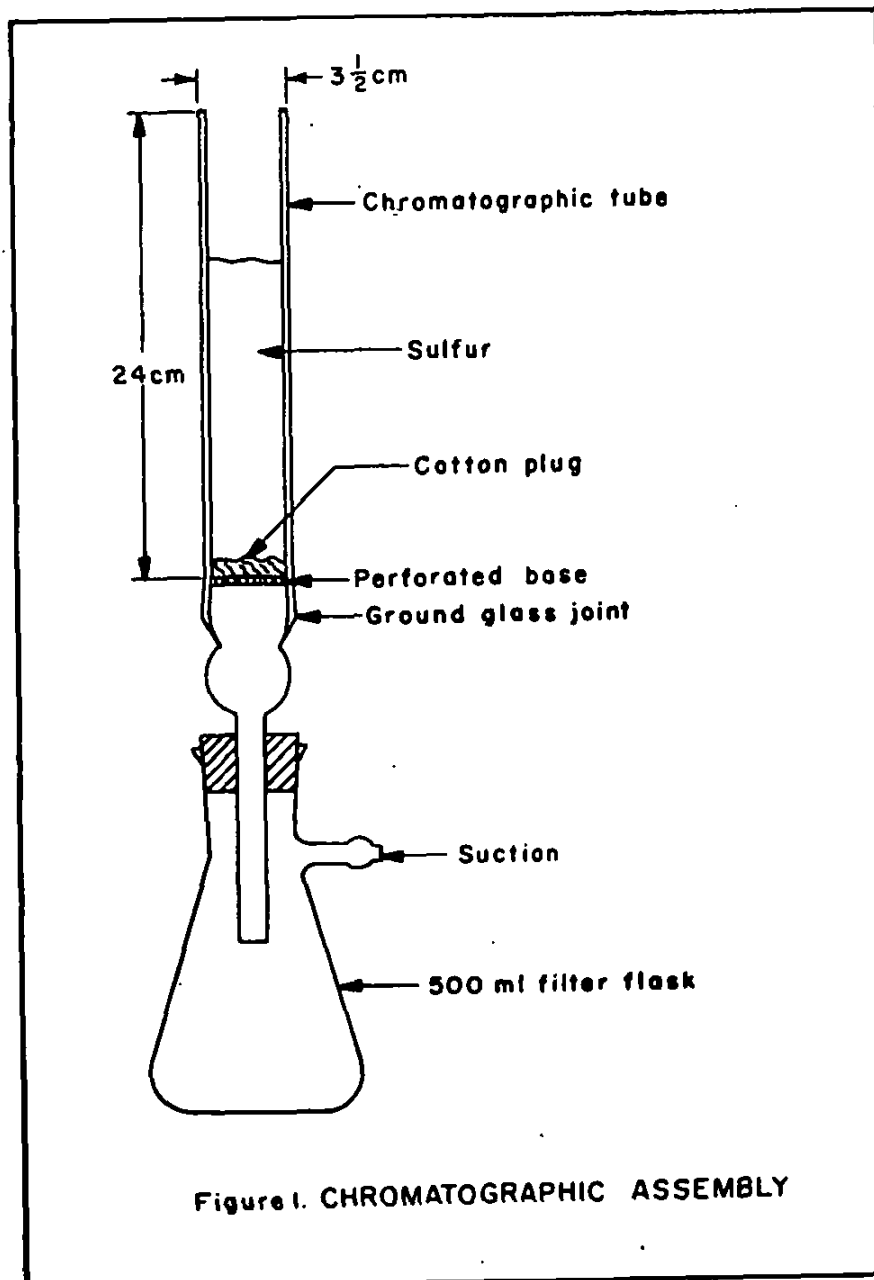
4.5.7 Particle Size - Particle size shall be determined in accordance with ASTM Test Method B 214-63T. Two metal washers weighing not more than 15 grams shall be added to a 100 gram portion of the sample in the top sieve to help break up lumps.

*4.5.8 Ammonia and Ammonium Salts - Weigh out approximately 20 grams of sulfur and place in a beaker with 100 ± 0.1 ml of reagent grade ammonia free water. The water shall be prepared and tested in accordance with ASTM D 1426. The water shall be at laboratory room temperature. Thoroughly mix the sulfur with the water by stirring 5 to 10 minutes. Filter the mixture and then thoroughly rinse the beaker and the filter with three 10 ml portions of reagent grade ammonia free water. Test the effluent for the presence of ammonia or ammonium salts by the Direct Nesslerization Method of ASTM D 1426.

5. PREPARATION FOR DELIVERY

*5.1 Packaging(Bottles only) -

5.1.1 Level A - The sulfur, filled into one or five pound bottles conforming to Type 1, Class 1 of PPP-C-301, shall be packaged in intermediate containers as specified in PPP-C-301.



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5.1.2 Level C - The sulfur shall be packaged in uniform quantities in accordance with the manufacturer's commercial practice. Unit and intermediate containers shall be of uniform size, shape and material. Intermediate containers shall contain only one grade of sulfur.

*5.2 Packing -

5.2.1 Level A -

5.2.1.1 Bottles - The sulfur, packaged as specified in 5.1.1, shall be packed in accordance with PPP-C-301, Level A.

5.2.1.2 Bulk - Fifty pounds of sulfur shall be packed in a shipping sack in accordance with construction 4x of UU-S-48, type of closure optional. The moisture proof barrier paper shall be polyethylene coated and conform to Type E of UU-S-48 with a basis weight of 1/15 PE-50.

Valve sack closures, when supplied, shall be constructed with moisture proof tuck-in-sleeves, except that the sleeves shall be polyethylene coated in accordance with Type E of UU-S-48, basis weight 1/15 PE-50. After filling, the sleeves shall be heat sealed or closed by tape by the procedure specified in UU-S-48.

In addition, both ends of the sack shall be completely sealed by either heat sealing prior to the closing procedures specified in UU-S-48, or by dipping the closed ends as specified in UU-S-48.

5.2.2 Level C (Bottles and bulk) - The sulfur shall be packed to insure carrier acceptance and safe delivery at destination in containers complying with the rules and regulations applicable to the mode of transportation.

*5.3 Marking - In addition to any marking required by the contract or order, all containers shall be marked in accordance with MIL-STD-129.

5.3.1 Additional Marking - Unit containers shall be marked with the following:

- (a) lot number
- (b) Precautionary instructions to cover hazards that may be encountered during storage or use of the sulfur.

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6. NOTES

6.1 Intended Use - Sulfur covered by this specification is intended for use as follows:

Grade A-For black powder
 Grade B-For pyrotechnic composition
 Grade C-For pyrotechnic composition
 Grade D-For nongaseous powder and primer compositions
 Grade E-For pyrotechnic composition.

6.2 Ordering Data - Procurement documents should specify the following:

- (a) Title, number and date of this specification
- (b) Grade required (see Table I) and quantity.
- (c) Levels of packaging and packing required (see 5.1 and 5.2).
- (d) Additional data, if required (see 3.1)

6.3 Manufacture -

6.3.1 Grade D sulfur is a special high purity grade of refined flour sulfur intended for use in explosive compositions which are sensitive to small quantities of moisture, acids, and electrolytes. Small lots of this grade have been made by washing refined flour sulfur with water and drying at 70°C. The refined sulfur known as "flowers of sulfur" containing up to 1.0 percent acidity and up to 30 percent amorphous sulfur is not a suitable raw material for manufacture of grade D sulfur.

6.3.2 Lumping or caking of grade C sulfur (finest particle size) may be expected inasmuch as the requirements of this specification are intended to prevent the addition of conditioning agents, either organic or inorganic.

6.4 Safety precautions - Sulfur dust or vapors are flammable and can explode when mixed with air and exposed to heat or flame. The lower limit of flammability in air is 30 milligrams per liter. Adequate ventilation should be provided to keep the concentration of sulfur in air below this limit. Sulfur is not considered toxic, but many of its compounds are poisonous. Contact with sulfur can result in conjunctivitis and irritation to sensitive skins.

Personnel who are sensitive to sulfur should wear protective clothing to avoid skin contact and chemical safety goggles for eye protection and a respirator to avoid excessive inhalation.

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Personnel who must fight fires of sulfur should be protected from the very toxic sulfur dioxide fumes. Water is the best extinguisher but small fires may also be controlled by smothering with sand or even with more sulfur. Since sulfur dust should not be scattered when fighting a fire, the use of a water spray is preferable to a solid stream or pressure hose.

6.4.1 Storage - Store sulfur in a cool, ventilated area away from heat, open flame, acute fire hazards and powerful oxidizing agents. Under these ideal conditions, the shelf life of sulfur is indefinite.

6.5 Marginal Notations - The outside margins of this specification have been marked to indicate where changes (deletions, additions, etc) from the previous issue have been made. This has been 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 as written irrespective of the marginal notations and relationship to the previous issue.

Custodians:

Army - MU
Navy - AS
Air Force - 68

Preparing Activity

Navy - AS

Review activities:

Army - MU, MI
Navy - AS, OS
Air Force - 68

DoD Project No. 6810-0431

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-487B Sulfur, Ground (For Use in Ammunition)			
ORGANIZATION (of submitter)		CITY AND STATE	
CONTRACT NO.	QUANTITY OF ITEMS PROCURED	DOLLAR AMOUNT \$	
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)		DATE	

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Naval Air Engineering Center
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