

MIL-D-48561A (AR)
29 June 1988
SUPERSEDING
MIL-D-48561
26 February 1975

MILITARY SPECIFICATION

DI-2-ETHYLHEXYL AZELATE

This specification is approved for use within the US Army Armament, Munitions and Chemical Command, and is available for use by all Departments and Agencies of the Department of Defense.

1. SCOPE

1.1 Scope. This specification covers the requirements and quality assurance provisions for the manufacture and acceptance of two types of di-2-ethylhexyl azelate (see 6.1).

1.2 Classification. The di-2-ethylhexyl azelate shall be of the following types:

TYPE I - For general use.

Type II - For use in propellant for the M864 system.

2. APPLICABLE DOCUMENTS

2.1 Government documents.

2.1.1 Specifications and standards. The following specifications and standards form a part of this specification to the extent specified herein. Unless otherwise specified, the issues of these documents shall be those listed in the issue of the Department of Defense Index of Specifications and Standards (DODISS) and supplement thereto, cited in the solicitation.

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commander, U.S. Army Armament, Munitions and Chemical Command, Attn. AMSMC-QA, Picatinny Arsenal, New Jersey 07806-5000 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

AMSC N/A

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FSC 6810

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SPECIFICATIONS

MILITARY

MIL-A-48078 .

- Ammunition, Standard Quality Assurance Provisions, General Specification For

STANDARDS

FEDERAL

FED-STD-141

- Paint, Varnish, Lacquer, and Related Materials; Methods of Inspection, Sampling, and Testing

MILITARY

MIL-STD-105

- Sampling Procedures and Tables for Inspection by Attributes
- Marking for Shipment and Storage
- Propellants, Solid: Sampling, Examination & Testing
- Lot Numbering of Ammunition
- ACS Chemicals

MIL-STD-129

MIL-STD-286

MIL-STD-1168

MIL-STD-1218

(Copies of specifications, standards, handbooks, drawings, publications, and other Government documents required by contractors in connection with specific acquisition functions should be obtained from the contracting activity or as directed by the contracting activity.)

2.2 Other publications. The following documents(s) form a part of this specification to the extent specified herein. Unless otherwise specified, the issues of the documents which are DOD adopted shall be those listed in the issue of the DODISS specified in the solicitation. Unless otherwise specified, the issues of documents not listed in the DODISS shall be the issue of the nongovernment documents which is current on the date of the solicitation.

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AMERICAN SOCIETY FOR TESTING AND MATERIALS (ASTM)

- D 92 - Flash and Fire Points by Cleveland Open Cup
- D 97 - Pour Point
- D 445 - Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)
- D 482 - Ash from Petroleum Products
- D 1193 - Reagent Water
- D 3951 - Standard Practice for Commercial Packaging
- E 168 - General Technique for Infrared Quantitative Analysis
- E 300-73 - Recommended Practice for Sampling Industrial Chemicals

(Application for copies should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.)

2.3 Order of precedence. In the event of a conflict between the text of this specification and the references cited herein, (except for associated detail specifications, specification sheets or MS standards), the text of this specification shall take precedence. Nothing in this specification, however, shall supersede applicable laws and regulations unless a specific exemption has been obtained.

3. REQUIREMENTS

3.1 Physical and chemical characteristics. The di-2-ethylhexyl azelate shall conform to the applicable physical and chemical characteristics as given below. Type I shall conform to Table IA and the applicable paragraphs below; Type II shall conform to Table IB and the applicable paragraphs below, when tested as specified therein.

TABLE IA. Physical and chemical characteristics for Type I.

<u>Characteristic</u>	<u>Requirement</u>	<u>Applicable Test Paragraph</u>
Specific gravity (15.6°/15.6°C)	0.921 ± 0.002	4.5.1.1
Pour point, °C	Below -62.22	4.5.1.1
Flash point, °C, min	205	4.5.1.1
Fire point, °C, min	230	4.5.1.1
Viscosity at 40°C, min	10.9 centistokes	4.5.1.1
Viscosity at -40°C, max	1250 centistokes	4.5.1.1
Ash, max	0.0005%	4.5.1.1
Iodine No., max	1.0	4.5.1.1

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TABLE IB. Physical and chemical characteristics for Type II.

Characteristic	Requirement		Applicable Test Paragraph
	Min.	Max	
Boiling point, at 5mm Hg, °F	230	250	4.5.2.1
Refractive Index, at 25°C	1.446	1.448	4.5.2.2
Specific Gravity, at 25 °C	0.914	0.918	4.5.2.3

3.2 Acid number. (Applies to Type I only.) The acid number shall be no greater than 0.20 when tested in accordance with 4.5.1.2.

3.3 Saponification number. (Applies to Type I only.) The saponification number shall be 274 ± 10 when tested in accordance with 4.5.1.3.

3.4 Infrared spectrum. (Applies to Type II only.) The IR spectrum of the sample shall contain the major peaks shown in Figure 1 when tested in accordance with 4.5.2.4.

3.5 Appearance. (Applies to Types I and II.) The specimen shall be clear and free from suspended matter when tested in accordance with 4.5.1.4.

3.6 Color.

3.6.1 Type I. The color shall be no darker than the standard when tested in accordance with 4.5.1.5.

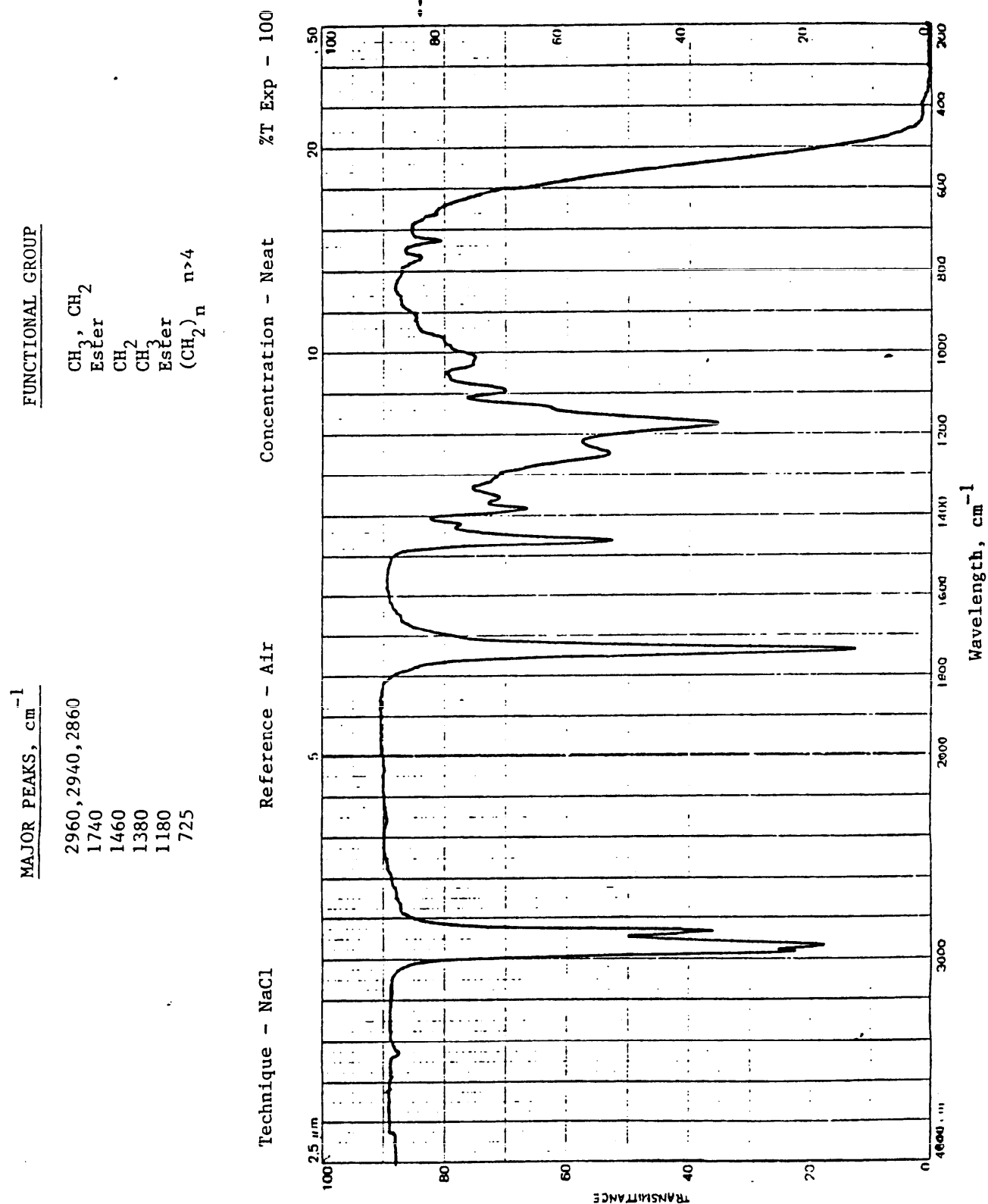
3.6.2 Type II. The color shall be white to slightly yellow when tested in accordance with 4.5.2.6.

3.7 First article inspection. This specification contains technical provisions for first article inspection. Requirements for the submission of first article samples by the contractor shall be as specified in the contract.

3.8 Workmanship. The manufacturer shall implement procedures and controls to assure that the process and the product produced are not compromised by foreign materials and contaminants or any other conditions which may degrade the composition. Determination of foreign materials shall be in accordance with 4.5.1.4.

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Figure 1 - IR Spectrum of Di-2-Ethylhexyl Azelate



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4. QUALITY ASSURANCE PROVISIONS

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the contractor is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified in the contract or purchase order, the contractor 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.

4.1.1 Responsibility for compliance. All items must meet all requirements of sections 3 and 5. The inspection set forth in this specification shall become a part of the contractor's overall inspection system or quality program. The absence of any inspection requirements in the specification shall not relieve the contractor of the responsibility of assuring that all products or supplies submitted to the Government for acceptance comply with all requirements of the contract. Sampling in quality conformance does not authorize submission of known defective material, either indicated or actual, nor does it commit the Government to acceptance of defective material.

4.2 Classification of inspections. The inspection requirements specified herein are classified as follows:

- a. First article inspection (see 4.3).
- b. Quality conformance inspection (see 4.4).

4.3 First article inspection

4.3.1 Submission. The contractor shall submit a first article sample as designated by the Contracting Officer for evaluation in accordance with provisions of 4.3.2. The first article sample shall be ten (10) pounds of di-2-ethylhexyl azelate. The sample shall be obtained from the first production lot which has been produced by the contractor using the same production processes, procedures and equipment as will be used in fulfilling the contract. All materials shall be obtained from the same sources of supply as will be used in regular production.

4.3.2 Inspection to be performed. The sample will be subjected by the Government to any or all of the examinations or tests specified in Table II (see MIL-A-48078).

4.3.3 Rejection. See MIL-A-48078.

TABLE II - First article inspection

CLASSIFICATION OF DEFECTS & TESTS

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PARAGRAPH	TITLE	EXAMINATION OR TEST	NO. OF SAMPLE UNITS	SHEET OF		DRAWING NUMBER	
				AQL OR 100%	REQUIREMENT PARAGRAPH	NEXT HIGHER ASSEMBLY	PARAGRAPH REFERENCE / INSPECTION METHOD
	Type I Specific gravity Pour point Flash point Fire point Viscosity Ash Iodine No. Acid No. Saponification No. Color Workmanship/appearance				3.1 3.1 3.1 3.1 3.1 3.1 3.1 3.1 3.2 3.3 3.6 3.5,3.8		4.5.1.1 4.5.1.1 4.5.1.1 4.5.1.1 4.5.1.1 4.5.1.1 4.5.1.1 4.5.1.1 4.5.1.2 4.5.1.3 4.5.1.5 4.5.1.4
	Type II Boiling point Refractive index Specific gravity Infrared Color Workmanship/appearance				3.1 3.1 3.1 3.4 3.6 3.5,3.8		4.5.2.1 4.5.2.2 4.5.2.3 4.5.2.4 4.5.2.6 4.5.1.4
NOTE:							

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4.4 Quality conformance inspection

4.4.1 Lot formation. A lot shall consist of one or more batches of Di-2-ethylhexyl Azelate produced by one manufacturer, in accordance with the same specification, or same specification revision, under one continuous set of operating conditions. Each lot shall consist of that quantity of Di-2-ethylhexyl Azelate that has been subjected to the same unit chemical or physical process intended to make the final product homogeneous. The lot shall comply with the provisions for submission of product as specified in MIL-STD-105. The criteria and procedure for the assignment of lot numbers shall be in accordance with MIL-STD-1168. Also, MIL-A-48078 applies.

4.4.2 Examination. See MIL-A-48078.

a. Workmanship. Examination for workmanship shall be conducted on each sample selected for testing in accordance with 4.4.3.1. If any sample fails to meet any test requirement the batch represented by the sample shall be rejected.

4.4.3 Testing. PRECAUTION: This specification covers sampling and testing of toxic and hazardous material. Accordingly, it is emphasized that all applicable safety rules, regulations and procedures must be followed in handling and processing the Di-2-ethylhexyl Azelate.

4.4.3.2 Sampling for type II Di-2-ethylhexylazelate. Approximately 500 grams of the composition shall be selected from each batch to be sampled using ASTM Method E300-73 for liquids. Samples shall be selected for inspection in accordance with table below. If any sample fails to meet any test requirement the batch represented shall be rejected. Each required test shall be performed in duplicate. No composite samples shall be used. The classification of defects shall be as given in Table III.

Number of containers in batch			Number of containers to be sampled	
2	-	100		2
100	-	200		5
200	-	300		8
300	-	500		10
500	-	1000		13
1000	-	3000		20
3000	-	10,000		32

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TABLE III. Classification of defects

<u>Test/Examination</u>	<u>Requirement Paragraph</u>	<u>Defect Classification</u>
<u>Type I:</u>		
Specific gravity	3.1	Major
Pour point	3.1	Major
Flash point	3.1	Major
Fire point	3.1	Major
Viscosity (40°C)	3.1	Major
Viscosity (-40°C)	3.1	Major
Ash	3.1	Major
Iodine No.	3.1	Major
Acid No.	3.2	Major
Saponification No.	3.3	Major
Color	3.6	Major
Workmanship	3.8	Major
<u>Type II:</u>		
Boiling point	3.1	Major
Refractive index	3.1	Major
Specific gravity	3.1	Major
Infrared spectrum	3.4	Major
Color	3.6	Major
Workmanship	3.8	Major

4.4 Inspection equipment. For the performance of all tests and examinations specified in 4.4 and 4.5, commercial inspection equipment should be employed. The contractor shall have available, and utilize correctly, this equipment and is charged with the responsibility of assuring that proper calibration procedures are followed.

4.5 Methods of inspections. All tests given in this section shall be performed using prescribed procedures for replicate determinations given in standard analytical chemistry test-books. Also, unless otherwise specified herein, all chemicals shall be Reagent Grade or ACS Grade in accordance with MIL-STD-1218. See 6.3 for use of equivalent test methods.

4.5.1 Method of inspection for Type I material.

4.5.1.1 General procedure. Perform tests in accordance with Table IV and 4.5.1 to 4.5.2 as applicable. Use reagent water conforming to the requirements for type III of ASTM method D 1193 and reagent-grade chemicals in all tests, unless otherwise specified. Run blank determinations and apply corrections when necessary.

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TABLE IV. Methods of inspection

Test	ASTM Method No.	FED-STD141 Method No.
Specific gravity		4183, para. 5
Four point	D 97	
Flash point	D 92	
Fire point	D 92	
Viscosity	D 445	
Ash	D 482	
Iodine No.		5061

4.5.1.2 Acid number. Transfer approximately 100 ml of a mixture of equal parts by volume of 95% alcohol and reagent grade benzene to a 300-ml Erlenmeyer flask. Heat the flask and contents to incipient boiling. Add 3 drops of 1% phenolphthalein solution and titrate with standard 0.01N sodium hydroxide solution to a faint pink color. Add 50 ml of the sample and titrate again to a faint pink color. Calculate the acid number as follows:

$$\text{Acid number} = \frac{56.1 \times A \times N}{M \times D}$$

where: A = ml of sodium hydroxide solution required for sample.
 N = normality of sodium hydroxide solution.
 M = ml of sample taken.
 D = specific gravity of sample.

4.5.1.3 Saponification number. Transfer a 2.0 g sample, weighed to 0.2 mg, to a 250 ml Erlenmeyer flask (24/40 ground glass joint) and add 50.00 ml of approximately 0.5% alcoholic potassium hydroxide solution. Carry along a blank determination on 50 ml of alcoholic potassium hydroxide solution. Reflex under a water-cooled condenser on an electric hot plate for 2 hours. Allow the flask and contents to cool to room temperature. Add 30 ml of 95% ethyl alcohol through the top of the condenser and disconnect the condenser. Add 3 drops of 1% phenolphthalein solution and titrate with standard 0.5N hydrochloric acid to the disappearance of the pink color. Calculate the saponification number as follows:

$$\text{Saponification number} = \frac{56.1 (B-A)N}{W}$$

where: B = ml of hydrochloric acid required for blank.
 A = ml of hydrochloric acid required for sample.
 N = normality of hydrochloric acid.
 W = g of sample.

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4.5.1.4 Appearance. Visually examine the specimen for clarity and suspended matter.

4.5.1.5 Color. Add 1.0 ml of 0.01N potassium dichromate (prepared by dissolving 0.4904 g of $K_2Cr_2O_7$ in water and diluting to 1 liter) to 49.0 ml of water in a Nessler tube and compare the color with 50 ml of specimen in a Nessler tube.

4.5.2 Methods of inspection for Type II material.

4.5.2.1 Boiling point.

4.5.2.1.1 Apparatus

- a. 500 ml, 3 neck flask
- b. Vacuum distilling apparatus
- c. Glas - Col heating unit
- d. Variable transformer
- e. Ground glass thermometer (range 10-360°C, subdivisions of 1°C)
- f. Glass wool insulating material
- g. Source of vacuum, 110 a.c. electricity and cold water (60-70°C)
- h. Bleed-off apparatus to control pressure (stop clock)
- i. Manometer accurate to 0.1 mm Hg
- j. Flexaframe and support clamps

4.5.2.1.2 Procedure.

- a. Weigh out 500 g of sample
- b. Assemble distillation apparatus on the Flexaframe. Assemble Glas-Col heater independently so it can be removed from flask. Insulate flask and distillation apparatus with glass wool.
- c. Pour sample into flask and insert thermometer in a position to measure vapor temperature.
- d. Apply vacuum and regulate to desired pressure (5mm on DOZ) using bleed-off and measure with manometer. Control heat with variable transformer to a gentle boil.
- e. After the temperature of vapor has stabilized, record temperature to nearest 1°C and the pressure at which it was obtained.

4.5.2.2 Refractive index.

4.5.2.2.1 Apparatus.

- a. Abbe-3L Refractometer (Bausch and Lomb)

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- b. Solvent for sample
- c. Soft tissues
- d. Acetone .

4.5.2.2.2 Procedure.

- a. Clean the upper and lower prisms with soft, non-abrasive, absorbent, tissues wetted with acetone; allow them to dry.
- b. Introduce the sample upon the prism by means of a pipette or dropper. Only a few drops are required. The prisms are then closed slowly allowing the excess to squeeze out into the space between the metal mounts. NEVER USE GLASS OR METAL APPLICATORS AGAINST THE PRISM FACE.
- c. After the sample is in position on the instrument, set the scale at the approximate value expected. (To see the scale, depress the momentary contact switch on the side of the instrument.)
- d. Release the switch. Bring the borderline which will probably be strongly colored, near the crosshair and compensate the color by adjusting the position of the dial (see Figure 1). The borderline should be faintly blue on one side and faintly red at the other.
- e. Observe the crosshair, sharply focusing the eyepiece if necessary and bring the dividing line upon their intersection by means of the course or fine hand controls.
- f. Read the refractive index by depressing the momentary contact switch. Estimate the fourth place.

4.5.2.2.3 Special notes.

- a. The refractive index decreases with a temperature rise because the density decreases. The prisms can be temperature controlled to meet the need.
- b. Free-flowing liquids can be introduced with an eyedropper into the channel which is alongside the prisms. When a mixture to be analyzed contains a volatile component, this technique is particularly useful because it minimizes the loss by evaporation of the volatile component and thus affects the accuracy of the readout.

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c. Never touch the prisms with any hard object. Always clean with soft tissues moistened with alcohol, acetone, or ether. After cleaning, always leave the prisms in a locked position.

4.5.2.3 Specific gravity. The specific gravity shall be determined in accordance with Method 510.1.1 of MIL-STD-286.

4.5.2.4 Infrared spectrum. An infrared spectrum shall be obtained using a procedure which has had prior approval of the Contracting Officer. ASTM E168 shall be used as a basis of the procedure.

4.5.2.5 Appearance. See 4.5.1.4.

4.5.2.6 Color. Visually examine the specimen for compliance with color requirement.

5. PACKAGING

5.1 Packaging and packing.

5.1.1 Level A. - When applicable packaging shall be as specified in the contract.

5.1.2 Level B. - When applicable packaging shall be as specified in the contract.

5.1.3 Level C. - Unless otherwise specified in contract or order, packaging and packing shall be in accordance with ASTM-D-3951.

5.2 Marking. - In addition to any special markings required by the contract or order, marking shall be in accordance with MIL-STD-129 and shall include, but not limited to, the following information:

STORE AT AMBIENT TEMPERATURES

6. NOTES

6.1 Intended use. The di-2-ethylhexyl azelate covered in this specification is intended for use in lubricants (Type I) and M864 propellant (Type II).

6.2 Ordering data. See MIL-A-48078 (AR).

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6.3 Equivalent test methods. The test methods given in this specification are the official methods to be used. The contractor may request using other methods providing that the proposed method is equivalent (accuracy and precision) to the method given in this specification. Prior approval of the Contracting Officer is required for use of equivalent test methods. A description of the proposed method should be submitted through the Contracting Officer to: Commander, ARDEC, ATTN: AMSMC-QAR-R (D), Picatinny Arsenal, NJ 07806-5000. This description should include, but not be limited to, the procedures used, the accuracy and precision of the method, test data to demonstrate the accuracy and precision and drawings of any special equipment required (see MIL-I-45208).

6.4 Subject term (key word) listing.

Propellant
Plasticizer
Projectile

6.5 Changes from previous issue. The margins of this standard are marked with vertical lines to indicate where significant changes 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.

Custodian:
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Project (6810-A013)

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NOTE: This form may not be used to request copies of documents, nor to request waivers, deviations, or clarification of specification requirements on current contracts. Comments submitted on this form do not constitute or imply authorization to waive any portion of the referenced document(s) or to amend contractual requirements.

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