

METRIC

MIL-D-3668D

9 December 1985

SUPERSEDING

MIL-D-3668C(EA)

31 October 1977

## MILITARY SPECIFICATION

## DYE, 1,4-DIAMINO-2,3-DIHYDROANTHRAQUINONE (METRIC)

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

## 1. SCOPE

1.1 Scope. This specification covers one type of anthraquinone dye.

## 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.

## SPECIFICATIONS

## FEDERAL

L-P-378 - Plastic Sheet and Strip, Thin Gauge, Polyolefin

## MILITARY

MIL-D-3464 - Desiccants, Activated, Bagged, Packaging Use and Static  
Dehumidification

MIL-D-3994 - Dextrin, Technical

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: 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 Chemical Research and Development Center, ATTN: SMCCR-SPD-TS, Aberdeen Proving Ground, MD 21010-5423 by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

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AMSC N/A

FSC 6820

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## STANDARDS

## MILITARY

- MIL-STD-105 - Sampling Procedures and Tables for Inspection by Attributes
- MIL-STD-129 - Marking for Shipment and Storage
- MIL-STD-147 - Palletized Unit Loads

(Copies of specifications and standards 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 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 non-government documents which is current on the date of the solicitation.

## UNIFORM FREIGHT CLASSIFICATION RULES

(Application for copies should be addressed to the Uniform Classification Committee, Room 1106, 222 South Riverside Plaza, Chicago, IL 60606.)

## ASTM STANDARDS

- D 1895 - Apparent Density, Bulk Factor, and Pourability of Plastic Materials (DOD Adopted)
- D 3951 - Commercial Packaging (DOD Adopted)
- E 11 - Wire-Cloth Sieves for Testing Purposes (DOD Adopted)

(Application for copies should be addressed to ASTM, 1916 Race Street, Philadelphia, PA 19103.)

(Nongovernment standards and other publications are normally available from the organizations which prepare or which distribute the documents. These documents also may be available in or through libraries or other informational services.)

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.

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## 3. REQUIREMENTS

3.1 Chemical and physical characteristics. Dye, 1,4-Diamino-2,3-dihydro-anthraquinone shall conform to the chemical and physical characteristics of table I when tested as specified therein.

TABLE I. Chemical and physical characteristics

Characteristic	Requirement	Test paragraph
Purity, percent by weight, minimum	70	4.2.4.1
Volatile matter, percent by weight, maximum	2.5	4.2.4.2
Particle size, minimum percent by weight passing:		4.2.4.3
250-micrometer sieve	97	
150-micrometer sieve	90	
75-micrometer sieve	70	
Apparent density, grams per milliliter	$0.35 \pm 0.10$	4.2.4.4

3.2 Diluent. Dextrin conforming to MIL-D-3994 is acceptable as a diluent. No other diluents shall be used.

## 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.

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4.2 Quality conformance inspection.

4.2.1 Lotting. A lot shall consist of the dye produced by one manufacturer, at one plant, from the same materials, and under essentially the same manufacturing conditions provided the operation is continuous. The minimum lot size shall be 2,270 kilograms. In the event the process is a batch operation (see 6.3), several batches may be mixed together to form a lot provided the batches are subjected to some physical mixing operation intended to make the final product substantially uniform and homogeneous; otherwise, each batch shall constitute a lot.

4.2.2 Sampling.

4.2.2.1 For examination of packaging. Sampling shall be conducted in accordance with MIL-STD-105.

4.2.2.2 For test. See 6.6 for sampling and testing precautions. Three representative specimens of approximately 200 grams (g) each shall be randomly removed from the lot of dye offered for acceptance and shall be placed in separate clean, dry containers labeled to identify the lot represented.

4.2.3 Inspection procedure.

4.2.3.1 For examination of packaging. The sample unit shall be one filled and closed unit container, ready for shipment. Sample unit containers shall be examined for the following defects using an AQL of 2.5 percent defective:

- (a) Contents per container not as specified
- (b) Container not as specified
- (c) Polyethylene liner or closure thereof not as specified (level B only)
- (d) Container closure not as specified
- (e) Container damaged or leaking
- (f) Desiccant not as specified or missing (level B only)
- (g) Unitization not as specified
- (h) Marking incorrect, missing, or illegible

4.2.3.2 For test. Each specimen taken in 4.2.2.2 shall be tested as specified in 4.2.4. Failure of any test by any specimen shall be cause for rejection of the lot represented.

4.2.4 Tests. See 6.6 for sampling and testing precautions. Reagent grade chemicals shall be used throughout the tests. Where applicable, blank determinations shall be run and corrections applied where significant. Tests shall be conducted as follows:

4.2.4.1 Purity. Prepare and measure the absorbance of a solution of standard 1,4-diamino-2,3-dihydroanthraquinone dye of known purity (see 6.4) and a solution of the specimen as follows: Weigh to the nearest milligram (mg) 0.186

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g of the dye and quantitatively transfer to a 500-milliliter (mL) volumetric flask using small amounts of 95-percent ethanol. Dissolve in approximately 400 mL of 95-percent ethanol, stirring and breaking up all lumps to facilitate solution. Dilute to the mark with 95-percent ethanol. Shake thoroughly to insure uniform mixing. Transfer a 20-mL aliquot of the solution to a second 500-mL volumetric flask, dilute to the mark with 95-percent ethanol, and mix thoroughly. Using a suitable spectrophotometer, read the absorbance value of the more dilute solution at  $456 \pm 2$  nanometers. Use the 95-percent ethanol used in preparing the solution as the reference liquid in the spectrophotometer. Cell spacers or variable path length cells may be used in lieu of the prescribed dilution procedures. Calculate the percent by weight purity as follows:

$$\text{Percent purity} = \frac{ABC}{DE}$$

where: A = Percent purity of standard,  
 B = Absorbance value of specimen,  
 C = Weight of standard in grams,  
 D = Absorbance value of standard dye, and  
 E = Weight of specimen in grams.

4.2.4.2 Volatile matter. Weigh to the nearest milligram approximately 5 g of the specimen in a tared, glass-stoppered weighing bottle. Remove the stopper and dry to constant weight at 70° to 75°C. Cool to room temperature in a desiccator and weigh. Calculate the percent by weight volatile matter as follows:

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

where: A = Weight of specimen and stoppered bottle before heating in grams,  
 B = Weight of specimen and stoppered bottle after heating in grams, and  
 W = Weight of specimen in grams.

4.2.4.3 Particle size. Use sieves conforming to ASTM E 11. Nest the sieves in order of decreasing mesh size (with the sieve of largest mesh on top) on a receiving pan. Weigh to the nearest 0.1 g approximately 10 g of the specimen and then use one of the following procedures:

(a) Dry method. Place the weighed specimen on the top sieve and brush the material with a camel's-hair brush until no more specimen passes through the sieve. Remove the sieve and weigh the material retained. Repeat this procedure with each of the other sieves. Calculate the percent material passing through each sieve.

(b) Wet method. Mix the weighed specimen with sufficient water in a beaker by stirring with a glass rod to produce a smooth paste. Carefully wash the thoroughly wetted specimen through the sieves. (The use of a wetting

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agent is permitted.) Dry the sieves in an oven at 70° to 75°C and weigh the material remaining on each sieve. Calculate the percent material passing through each sieve.

4.2.4.4 Apparent density. Determine the apparent density in accordance with ASTM D 1895, method A.

## 5. PACKAGING

5.1 Unit packing. The dye shall be unit packed level B or commercial as specified (see 6.2).

5.1.1 Level B. Uniform quantities of no less than 65 and no more than 130 kilograms  $\pm 1/4$  percent of the marked net weight of dye shall be packed in a fiber drum conforming to rule 51, section 2 of the Uniform Freight Classification for a weight limit of over 225 but not over 300 pounds, with an aluminum barrier of 7.62 micrometers minimum thickness incorporated into one of the inner plies of the sidewall. Each drum shall be provided with a close-fitting bag liner formed from polyethylene of 101.6 micrometers minimum thickness conforming to type I, class 1, grade and finish optional of L-P-378. Seams shall be completely heat sealed and shall meet the heat-seal strength requirements of L-P-378. The filled bag shall be closed by heat sealing, tying, or knotting. A minimum of eight units of desiccant conforming to MIL-D-3464 shall be placed on top of the closed liner. Drum closure shall be fully gasketed and shall form a tight seal between the rim and the top of the drum.

5.1.2 Commercial. Uniform quantities of no less than 65 and no more than 130 kilograms  $\pm 1/4$  percent of the marked net weight of dye shall be unit packed in accordance with ASTM D 3951.

5.2 Packing. Dye, unit packed as specified in 5.1, shall require no further protection for shipment other than unitization.

5.3 Unitization. Shipping containers shall be unitized in accordance with the applicable requirements of MIL-STD-147 except that the pallet shall be as specified in the contract (see 6.2).

5.4 Marking. Marking for level B shipments shall be in accordance with MIL-STD-129. Marking for commercial shipments shall be in accordance with ASTM D 3951. All shipments shall be marked to show lot number and date of manufacture. In addition, each container shall be durably and legibly marked with contrasting letters and background to show the net weight of contents and the following information:

### CAUTION

Avoid contact with skin or clothing.  
In case of contact, flush with water.  
Avoid breathing dye or combustion products.  
Mask during manufacture or use where potential  
for exposure exists.

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## 6. NOTES

6.1 Intended use. Dye, 1,4-Diamino-2,3-dihydroanthraquinone is intended for use in the manufacture of violet signaling smoke mixtures.

6.2 Ordering data. Acquisition documents should specify the following:

- (a) Title, number, and date of this specification,
- (b) Level of unit packing required (see 5.1), and
- (c) Type of pallet required (see 5.3).

6.3 Batch. A batch is defined as that quantity of material which has been manufactured by some unit chemical process.

6.4 Dye of known purity. 1,4-Diamino-2,3-dihydroanthraquinone dye of known purity for use in 4.2.4.1 may be obtained by authority of the Procuring Contracting Officer (PCO). Alternately, the contractor may prepare his own standard dye and obtain verification of its purity by authority of the PCO from Commander, U.S. Army Armament, Munitions and Chemical Command, Product Assurance Directorate, Aberdeen Proving Ground, MD 21010-5423.

6.5 Significant places. For the purpose of determining conformance with this specification, an observed or calculated value should be rounded off "to the nearest unit" in the last right-hand place of figures used in expressing the limiting value, in accordance with the rounding-off method of ASTM E 29.

6.6 Sampling and testing precautions. This specification requires inspection of chemical material which is potentially hazardous to personnel. All applicable safety rules, regulations and procedures must be followed in the handling and processing of this material.

Custodian:

Army - EA

Review activities:

Army - MD  
DLA - GS

Preparing activity:

Army - EA

Project No. 6820-A084





## STANDARDIZATION DOCUMENT IMPROVEMENT PROPOSAL

(See Instructions – Reverse Side)

1. DOCUMENT NUMBER MIL-D-3668D		2. DOCUMENT TITLE DYE, 1,4-DIAMINO-2,3-DIHYDROANTHRAQUINONE (METRIC)	
3a. NAME OF SUBMITTING ORGANIZATION		4. TYPE OF ORGANIZATION (Mark one)	
		<input type="checkbox"/> VENDOR <input type="checkbox"/> USER <input type="checkbox"/> MANUFACTURER <input type="checkbox"/> OTHER (Specify): _____	
b. ADDRESS (Street, City, State, ZIP Code)			
5. PROBLEM AREAS			
a. Paragraph Number and Wording:			
b. Recommended Wording:			
c. Reason/Rationale for Recommendation:			
6. REMARKS			
7a. NAME OF SUBMITTER (Last, First, MI) – Optional		b. WORK TELEPHONE NUMBER (Include Area Code) – Optional	
c. MAILING ADDRESS (Street, City, State, ZIP Code) – Optional		8. DATE OF SUBMISSION (YYMMDD)	

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