

METRIC
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**MIL–DTL–3668E(EA)**

w/Amendment 1

26 October 2009

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SUPERSEDING**MIL–DTL–3668E(EA)**

15 August 2006

**DETAIL SPECIFICATION****DYE, 1,4–DIAMINO–2,3–DIHYDROANTHRAQUINONE (BLUE)**

*This specification is approved for use by the U.S. Army Edgewood Chemical Biological Center and is available for use by all Departments and Agencies of the Department of Defense.*

**1. SCOPE**

**1.1 Scope.** This specification covers two types of blue dye (CAS #81–63–0), chemically known as 1,4–Diamino–2,3–Dihydroanthraquinone;

- Type I – for solvent based binders and bowl and V type blenders,  
Type II – for air based fluidized bed type blenders.

**2. APPLICABLE DOCUMENTS**

**2.1 General.** The documents listed in this section are specified in sections 3 or 4 of this specification. This section does not include documents cited in other sections of this specification or recommended for additional information or as examples. While every effort has been made to insure the completeness of this list, document users are cautioned that they must meet all specified requirements of documents cited in sections 3 or 4 of this specification, whether or not they are listed.

Comments, suggestions, or questions on this document should be addressed to: U.S. Army Edgewood Chemical Biological Center, ATTN: RDCB–DEA–S, 5183 Blackhawk Road, Aberdeen Proving Ground, MD 21010–5424 or emailed to <a href="mailto:APGR–EcbsSpecsTeam@us.army.mil">APGR–EcbsSpecsTeam@us.army.mil</a> . Since contact information can change, you may want to verify the currency of this address information using the ASSIST Online database at <a href="http://assist.daps.dla.mil">http://assist.daps.dla.mil</a> .
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AMSC N/A

FSC 6820

**DISTRIBUTION STATEMENT A.** Approved for public release; distribution is unlimited.

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## 2.2 Government documents.

### 2.2.1 Deleted.

**2.2.2 Other Government documents, drawings, and publications.** The following other Government documents, drawings, and publications form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

#### U.S. ENVIRONMENTAL PROTECTION AGENCY

##### EPA Method 6010B – Inductively Coupled Plasma–Atomic Emission Spectrometry

(Copies of these documents are available online at [www.epa.gov](http://www.epa.gov) or from the U.S. Environmental Protection Agency, Ariel Rios Building, 1200 Pennsylvania Avenue, N.W., Washington, DC 20460.)

**2.3 Non–Government publications.** The following documents form a part of this document to the extent specified herein. Unless otherwise specified, the issues of these documents are those cited in the solicitation or contract.

#### ASTM INTERNATIONAL

##### D 1895 – Standard Test Methods for Apparent Density, Bulk Factor, and Pourability of Plastic Materials

(Copies of this document are available online at [www.astm.org](http://www.astm.org) or ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428–2959.)

**2.4 Order of precedence.** Unless otherwise noted herein or in the contract, in the event of a conflict between the text of this document and the references cited herein, the text of this document takes precedence. Nothing in this document, however, supersedes applicable laws and regulations unless a specific exemption has been obtained.

## 3. REQUIREMENTS

**3.1 First article.** When specified (see 6.2), a sample shall be subjected to first article inspection in accordance with 4.2.

**3.2 Chemical and physical characteristics.** The dye shall conform to the chemical and physical characteristics of table I. Yellow corn dextrin conforming to MIL–D–3994 can be

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used to adjust the bulk density of the dye. Only the minimum amount of dextrin required to obtain the apparent density shall be used.

TABLE I. Chemical and physical requirements.

Characteristic	Requirement		Test Paragraph
	Type I	Type II	
Purity, dry basis, percent by weight, minimum	70	85	4.4.1
Heavy metal content			4.4.4
Arsenic (As), ppm maximum	< 3	< 3	
Cadmium (Cd), ppm maximum	< 15	< 15	
Mercury (Hg), ppm maximum	< 3	< 3	
Lead (Pb), ppm maximum	< 20	< 20	
Chromium (Cr), ppm maximum	< 30	< 30	
Zinc (Zn), ppm maximum	< 30	< 30	
Volatile matter, percent by weight, maximum	2.5	2.5	4.4.3
Particle size distribution:			4.4.2
Average particle size, microns, maximum	— — —	45	
Average particle size, microns, minimum	— — —	30	
Particles less than 10 microns, percent maximum	— — —	20	
Distribution shape	— — —	Single mode	
Particles less than 250 microns, percent minimum	97	— — —	
Particles less than 150 microns, percent minimum	90	— — —	
Particles less than 75 microns, percent minimum	70	— — —	
Apparent density, grams per milliliter (dry basis)	$0.35 \pm 0.10$	$0.40 \pm 0.10$	4.4.5
Dextrin content, percent by weight maximum	30.0	15.0	4.4.6

#### 4. VERIFICATION

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

- (a) First article inspection (see 4.2)
- (b) Conformance inspection (see 4.3)

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## **4.2 First article inspection.**

**4.2.1 Lotting.** The first article lot shall consist of at least 970 kg of blue dye manufactured using the same methods, materials, equipment, and processes as will be used during regular production. The first article sample shall be submitted for inspection and approval in accordance with the terms of the contract. If there is any change of vendors, methods, materials, equipment, facilities, or if there is a break in production of 90 days or more, the contracting Officer shall be notified and at the discretion of the Government, a new first article sample may be required.

**4.2.2 Sampling.** The contractor shall randomly select three representative samples, of approximately 200g each, from the first article lot of dye and place each sample in a clean dry container labeled to identify the first article lot.

**4.2.3 Inspection procedure.** Each first article sample shall be subjected to all of the inspections specified in the classification of characteristics in 4.3.5.

**4.2.4 Acceptance criteria.** If any first article sample fails to comply with any of the applicable requirements, the first article sample shall be rejected.

## **4.3 Conformance inspection.**

**4.3.1 Lotting.** A lot shall consist of the dye produced by one manufacturer, at one plant, from the same materials, under the same manufacturing conditions, and at the same time. The minimum lot size shall be 2270 kg. In the event the lot is a batch operation (see 6.7), 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 uniform and homogenous.

**4.3.2 Sampling.** The contractor shall randomly select three representative samples, of approximately 200g each, from the lot of dye and place each sample in a clean dry container labeled to identify the lot represented.

**4.3.3 Inspection procedure.** Each dye sample shall be inspected in accordance with the classification of characteristics in 4.3.5. Failure of any sample to conform to any characteristic in the classification of characteristics based acceptance criteria specified therein shall be cause for rejection of the lot represented.

**4.3.4 Inspection characteristics.** Critical characteristics are characteristics whose nonconformance to specified requirements is likely to result in hazardous or unsafe conditions for individuals who use or maintain the product. Characteristics whose nonconformance to specified requirements is likely to prevent performance of the tactical function of a major end item are also critical characteristics. Major characteristics are characteristics whose nonconformance to specified requirements is likely to result in failure or to reduce materially the usability of the item for its intended purpose. Minor characteristics are characteristics whose nonconformance

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to specified requirements is not likely to reduce materially the operation or usability of the item for its intended purpose.

**4.3.5 Classification of characteristics.** Conformance inspections shall be as specified in the following classification of characteristics paragraphs.

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**CLASSIFICATION OF CHARACTERISTICS**

PARAGRAPH	TITLE	SHEET 1 OF 1		DRAWING NUMBER	
4.3.5(a)	Dye, 1,4–Diamino–2,3–Dihydroanthra- quine				
CATEGORY	CHARACTERISTIC	SAMPLING AND ACCEPTANCE CRITERIA	REQUIREMENT PARAGRAPH	INSPECTION METHOD	
<b>Critical</b>  <b>Major</b>  101	None defined	Note 1.	3.2		
	Chemical and physical properties				
	a. Purity			4.4.1	
	b. Heavy metals content			4.4.4	
	c. Volatile matter			4.4.3	
	d. Particle size			4.4.2	
	e. Apparent density			4.4.5	
	f. Dextrin content			4.4.6	
<b>NOTES:</b>  1. Sampling shall be in accordance with 4.3.2. One specimen shall be taken from each sample to perform each test.					

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#### 4.4 Tests.

**4.4.1 Purity.** Prepare and measure the absorbance of a standard 1,4–Diamino–2,3–Dihydroanthraquinone dye solution and specimen solution as follows: Weigh to the nearest milligram approximately 0.186g of dye and quantitatively transfer to a 500–milliliter (mL) volumetric flask using small amounts of ethanol for washing. Dissolve specimen in approximately 400mL of ethanol, stirring and breaking up all lumps to facilitate the solution. Dilute to 500mL with ethanol. Shake thoroughly to insure uniform mixing. Remove a 20mL aliquot, transfer to a second 500mL volumetric flask, dilute to 500mL with ethanol, and mix thoroughly. Using a spectrophotometer, read the adsorbance value of the more dilute solution at  $456 \pm 2$  nanometers. Use the ethanol that was used in preparing the solution as the reference liquid in the spectrophotometer. Calculate the percent purity as follows:

$$\text{Percent purity} = (ABC)/(DE)$$

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

**4.4.2 Particle size.** Particle size analysis shall be performed using the laser diffraction method outlined below. Particle size analysis and histogram are required with material certification.

##### 1. SAMPLE PREPARATION:

- A. Weigh approximately 0.2 Grams (200 mg) of dye sample into a 30 mL beaker. Add 25 mL deionized water to the dye sample.
- B. Add 1 or 2 drops of surfactant into the dye/water mixture.
- C. Place an external ultrasonic probe (Sonics 130 watt) in the dye sample mixture. Sonicate the sample mixture for 2 minutes at 100% power setting.

##### 2. INTRODUCTION OF SAMPLE INTO THE INSTRUMENT AND TESTING:

- A. After sonication is completed, place a clean stirring bar into the sample and move the sonicated sample to a stirrer set to medium speed to keep all of the particles in the liquid in suspension.
- B. Set the laser diffraction particle size analyzer for MIE calculation with a Relative Refractive Index at  $1.50 - .010$ .
- C. Using a pipette transfer a  $7.5 \pm 0.5$  mL sample from the beaker into the reservoir with the system circulating at a slow speed until proper particle loading is attained in the instrument.

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D. Run the sample immediately.

E. 1. For Type I dye report the percentages less than 250, 150 and 75 microns for the sample.

2. For Type II dye report the mean particle size, distribution shape and the percentage below 10 microns for the sample.

**4.4.3 Volatile matter.** Weigh to the nearest milligram approximately 5g 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:

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

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 before heating in grams.

**4.4.4 Heavy metal contamination.** Determine the heavy metals content of the sample in accordance with EPA Method 6010B.

**4.4.5 Apparent density.** Determine the apparent density in accordance with ASTM D 1895, Method A, except that the sample shall be dried as specified in 4.4.3 prior to testing.

**4.4.6 Dextrin content.** Verify the dextrin content by certification. The certification shall include the dye density before adding the dextrin, the amount of dextrin used and the final density after addition of the dextrin.

## 5. PACKAGING

**5.1 Packaging.** For acquisition purposes, the packaging requirements shall be as specified in the contract or order (see 6.2). When actual packaging of materiel is to be performed by DoD or in–house contractor personnel, these personnel need to contact the responsible packaging activity to ascertain requisite packaging requirements. Packaging requirements are maintained by the Inventory Control Point’s packaging activities within the Military Service or Defense Agency, or within the military service’s system commands. Packaging data retrieval is available from the managing Military Department’s or Defense Agency’s automated packaging files, CD–ROM products, or by contacting the responsible packaging activity.

## 6. NOTES

(This section contains information of a general or explanatory nature that may be helpful, but is not mandatory.)



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**6.1 Intended use.** Dye, 1,4–Diamino,–2,3–Dihydroanthraquinone, covered by this specification is intended for use in the manufacture of colored signaling smoke mixtures.

**6.2 Acquisition requirements.** Acquisition documents should specify the following:

- (a) Title, number, and date of this specification.
- (b) First article:
  - (1) Time allowed for contractor submission of samples for Government test and evaluation after award of contract when testing is performed by the Government.
  - (2) Name and address of test facility and shipping instructions when testing is performed by the Government.
  - (3) Time required for the Government to notify the contractor whether or not to proceed with production.
- (c) Packaging requirements (see 5.1).
- (d) Material Safety Data sheets preparation (see 6.4)
- (e) Certificate of Analysis (see 6.5)

**6.3 Subject term (key word) listing.**

Smoke grenade

Colored signaling smoke

**6.4 Material Safety Data Sheets.** Material Safety Data Sheets for the dye should be prepared and submitted in accordance with Fed. Std. No. 313.

**6.5 Certificate of Analysis.** A certificate of analysis including all the requirements in Table I should be prepared for the dye. The dextrin content should be explicitly stated in the analysis.

**6.6 Supersession data.** This document supersedes MIL–DTL–3668E, dated 15 August 2006.

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

**6.8 Changes from previous issues.** The margins of this specification are marked with vertical lines to indicate where 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. 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.

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**6.9 Compatibility.** Type I dye is provided for compatibility with pyrotechnic formulations based on the original MIL-D-3668 dye. Type II is provided for use with water based binders in air type blenders.

Preparing activity:

Army – EA

(Project 6820-2010-005)

NOTE: The activities listed above were interested in this document as of the date of this document. Since organizations and responsibilities can change, you should verify the currency of the information above using the ASSIST Online database at <http://assist.daps.dla.mil>.