METRIC

MIL-D-3277E
9 December 1985
SUPERSEDING
MIL-D-003277D(EA)
31 October 1977
MIL-D-3277C
22 June 1971

MILITARY SPECIFICATION

DYE, SOLVENT GREEN 3 (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 Solvent Green 3 dye, chemically known as 1,4-di-p-toluidinoanthraquinone.

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

: 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.

AMSC N/A FSC 6820

MILITARY

MIL-D-3464 - Desiccants, Activated, Bagged, Packaging Use and Static Dehumidification
MIL-D-3994 - Dextrin, Technical

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.

3. REQUIREMENTS

3.1 Chemical and physical characteristics. Dye, Solvent Green 3 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, dry basis, percent by weight, minimum	90	4.2.4.1
Volatile matter, percent by weight, maximum	2.5	4.2.4.2
Particle size, percent by weight passing:		4.2.4.3
850-micrometer sieve, minimum	100	
300-micrometer sieve, minimum	97	
45-micrometer sieve, maximum	40	
Apparent density, grams per milliliter, dry basis, minimum	0.38	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.

4.2 Quality conformance inspection.

4.2.1 Lotting. A lot shall consist of the Dye, Solvent Green 3 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 6.7 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 6.7 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 area of the chromatographic peak of a standard Solvent Green 3 dye solution (see 6.4) and a solution of the specimen as follows: Weigh to the nearest 0.1 milligram (mg) exactly 100.0 mg of

the dye, which has been previously dried to constant weight at 70° to 75°C and transfer quantitatively to a 100-milliliter (mL) volumetric flask. Dissolve the dye in methylene chloride (dichloromethane), mix thoroughly, and dilute to 100 mL with methylene chloride (this stock solution contains 1 mg dye per milliliter). Using a suitable high pressure liquid chromatograph, inject 10 microliters of the solution and using a suitable integrator, measure the area under the Solvent Green 3 dye peak (retention time is 3.2 minutes) with the detector set at either 407 or 290 nanometers. The mobile phase is methylene chloride flowing at 2 mL per minute (1900 pounds per square inch) through a 3.9 by 25 millimeter guard column filled with 37 to 50 micrometer silica and a 4.6 by 250 millimeter analytical column containing 5 micrometer silica. Calculate the percent by weight purity as follows:

$$\text{Percent purity} = \frac{AC}{B}$$

where: A = Area of specimen solution peak,
B = Area of standard solution peak, and
C = Percent purity of the standard.

Repeat the injections three more times and report the percent purity as the average of the four results.

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

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 except that the specimen shall be dried as specified in 4.2.4.2 prior to testing.

5. PACKAGING

5.1 Unit packing. Dye, Solvent Green 3 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 ± 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 ± 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 dust or fumes.
Use with adequate ventilation.

6. NOTES

6.1 Intended use. Dye, Solvent Green 3 is intended for use in the manufacture of colored 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. Solvent Green 3 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 Color index. The Color Index number of Solvent Green 3 dye is C161565.

6.7 Sampling and testing precautions. This specification requires inspection of chemical material and use of reagents which are potentially hazardous to personnel. All applicable safety rules, regulations and procedures must be followed in the handling and processing of these materials.

Custodians:

Army - EA
Navy - OS

Review activities:

Army - MD
DLA - GS

Preparing activity:

Army - EA

Project No. 6820-0083

U.S. GOVERNMENT PRINTING OFFICE: 1986-605-036/40915