

MIL-T-23624(Wep)
31 December 1962

MILITARY SPECIFICATION

TOLYLENE - 2, 4 - DIISOCYANATE

This specification has been approved by the Bureau of Naval Weapons, Department of the Navy

1. SCOPE

1.1 This specification establishes the requirements of tolylene - 2, 4 - diisocyanate for use as addition and condensation adducts in polyurethane manufacture (see 6.1).

1.2 Classification. The tolylene - 2, 4 - diisocyanate shall be of the following grades as specified (see 6.2):

Grade A - 2, 4 isomer content - 64 percent minimum
 Grade B - 2, 4 isomer content - 79 percent minimum
 Grade C - 2, 4 isomer content - 98 percent minimum

2. APPLICABLE DOCUMENTS

2.1 The following specifications, standards, and publications form a part of this specification. Unless otherwise specified, the issue in effect on date of invitation for bids shall apply:

SPECIFICATIONS

Federal

PPP-C-96	Cans, Metal, 28 Gage and Lighter
PPP-D-729	Drums: Metal, 55-Gallon (for Shipment of Noncorrosive Material)
PPP-P-704	Pails: Shipping, Steel (1 through 12 Gallon)

STANDARDS

Federal

FED-STD-141	Paint, Varnish, Lacquer, and Related Materials, Methods of Inspection, Sampling, and Testing
FED-STD-791	Lubricants, Liquid Fuels and Related Products, Methods of Testing

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Military

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

(When requesting any of the applicable documents, refer to both title and number. All requests should be made via the cognizant Government inspector. Copies of this specification and other unclassified specifications and drawings required by the contractor in connection with specific procurement functions should be obtained upon application to the Commanding Officer, Naval Supply Depot (Code CDS), 5801 Tabor Avenue, Philadelphia 20, Pennsylvania. All other documents 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. Unless otherwise indicated, the issue in effect on date of invitation for bids shall apply:

American Society for Testing and Materials

ASTM Standards

(Copies of ASTM publications may be obtained from the American Society for Testing and Materials, 1916 Race Street, Philadelphia 3, Pennsylvania).

Consolidated Classification Committee

Consolidated Freight Classification Ratings, Rules, and Regulations

(Applications for copies should be addressed to the Consolidated Classification Committee, 202 Chicago Union Station, Chicago 6, Illinois).

National Classification Board

National Motor Freight Classification Rules and Container Regulations

(Applications for copies should be addressed to the National Classification Board, 1424 Sixteenth Street, N. W. Washington 6, D. C.

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Manufacturing Chemists' Association, Inc.

Chemical Safety Data Sheet SD-73-Properties and Essential Information for Safe Handling and Use of Toluene Diisocyanate

(Application for copies should be addressed to the Manufacturing Chemists' Association, Inc., 1925 Connecticut Avenue, N. W., Washington 9, D. C.).

3. REQUIREMENTS

3.1 Material.- The toluene - 2, 4 - diisocyanate shall be a product so formulated as to meet the requirements of this specification (see 4.4.1).

3.2 Requirements.- The toluene - 2, 4 - diisocyanate shall conform to the applicable requirements specified in Table I when tested as specified in 4.4.

Table I - Requirements

	Grade		
	A	B	C
Assay (total isocyanate), min., percent	99.0	99.0	99.0
Hydrolyzable chlorine, max., percent	0.01	0.01	0.01
Total chlorine, max., percent	0.04	0.04	0.04
Freezing point, °C	5.0-6.5	12.5-13.5	20.0-22.0
Color, Hazen or APHA, max.	50	50	50
Specific gravity, 20/4 °C	1.21-1.23	1.21-1.23	1.21-1.23
Refractive index, 25°C	1.564-1.568	1.564-1.568	1.564-1.568
Acidity, as HCl, max., percent	0.04	0.04	0.04
Isomer content:			
2, 4 by infra-red, min., percent	64.0	79.0	98.0
2, 6 by infra-red, max., percent	36.0	21.0	2.0

3.3 Precautionary marking. In addition to the marking specified in 5.3, the following special marking shall also be included on the label:

Precautionary Marking

Warning: Poison
 Avoid contact with skin
 Avoid breathing of vapor
 Use with adequate ventilation
 Keep container closed
 Keep away from moisture
 Keep away from heat and open flame

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3.4 Workmanship.- The tolylene -2,4 - diisocyanate ingredients shall be processed to meet the requirements of this specification (see 4.4.1).

4. QUALITY ASSURANCE PROVISIONS

4.1 The supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own or any other inspection facilities and services acceptable to the Government. Inspection records of the examinations and tests shall be kept complete and available to the Government as specified in the contract or order. 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 supplies and services conform to prescribed requirements.

4.2 Acceptance inspection.- Conformance of the tolylene -2,4 - diisocyanate to the requirements of this specification shall be determined entirely by means of acceptance inspection. The acceptance inspection shall consist of an examination for acceptability of quality control methods used by the manufacturer, examining and testing the acceptance samples (4.3.2) for all of the requirements of this specification, and an examination of the sample of filled containers (4.3.3) for conformance to the packaging, packing, and marking requirements.

4.3 Sampling.

4.3.1 Size of lot.- For the purpose of sampling, a lot of tolylene - 2, 4 - diisocyanate shall consist of a manufacturer's batch. If the material cannot be identified by batch, a lot shall consist of not more than 5,000 pounds of tolylene - 2, 4 - diisocyanate offered for delivery at one time.

4.3.2 Sample for tests.- From each lot offered for acceptance under contract, two 1-quart samples of tolylene - 2, 4 - diisocyanate shall be removed from separate unit containers taken at random. The samples so selected shall be placed in small mouth glass-stoppered bottles. Individual samples shall not be mixed, shall be placed in separate air-tight and water-tight containers, which shall be nearly filled, covered and sealed to prevent atmospheric effects, and shall be labeled completely with information on the lot and batch number, date of sampling, contract number, and applicable specification and grade. Effect of temperature variance shall be minimized by maintenance at 75±20F. A batch is defined as the end product of all the raw materials mixed or blended in a single operation.

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4.3.3 Sample for examination of filled containers.- A random sample of filled containers shall be selected from each lot of tolylene -2, 4-diisocyanate offered for acceptance under contract, in accordance with Standard MIL-STD-105 at inspection level I and acceptable quality level (AQL) = 2.5 percent defective.

4.4 Inspection methods.- Unless otherwise specified all examinations and tests shall be conducted in accordance with the provisions of Standard MIL-STD-105. Unless otherwise specified the physical and chemical values specified in Section 3 apply to the average of the determinations made on the unit of product.

4.4.1 Assay.- Add 40 milliliters (ml) of dry toluene to a clean dry glass-stoppered 500 ml erlenmeyer flask. With the aid of a weighing pipet accurately weigh to the nearest milligram between 6.5 and 7.0 grams (gm) of the tolylene -2, 4 - diisocyanate into the flask. Immediately add 50.0 ml of 2 normal (N) dibutylamine (in dried xylene) solution. Rinse the neck and sides of the flask with an additional 10 ml of dry toluene. Let stand for 15 minutes with the flask loosely stoppered. Add 225 ml of isopropyl alcohol and 0.8 ml of bromcresol green indicator from a 1.0 ml pipet and titrate with 1N hydrochloric acid to a color change from green to yellow that persists for at least 15 seconds. Determine a blank by adding 50.0 ml of 2N dibutylamine to 50 ml of toluene in an erlenmeyer flask. Add 225 ml of isopropyl alcohol and 0.8 ml of bromcresol green indicator. Titrate with 1N HCl. Calculate the assay percentage as follows:

$$\text{Assay, percent} = \frac{(B-A) \times N \times 8.708}{W}$$

Where: A = volume of HCl required to titrate sample
 B = volume of HCl required to titrate blank
 N = normality of HCl
 W = weight of sample

4.4.3 Hydrolyzable chlorine.- The hydrolyzable chlorine in the tolylene -2, 4- diisocyanate shall be determined in accordance with ASTM Method No. D1638, sections 39 to 46 inclusive.

4.4.4 Total chlorine.- The total chlorine in the tolylene -2, 4- diisocyanate shall be determined in accordance with ASTM Method No. D808 using a 1 ± 0.100 gm sample

4.4.5 Freezing point.- The freezing point of the tolylene -2,4- diisocyanate shall be determined in accordance with Standard FED-STD-791 Method No. 1411.

4.4.6 Color.- The color of the tolylene -2, 4- diisocyanate shall be determined by comparison with Hazen or APHA platinum-cobalt standards.

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The color standards are dilute HCl solutions of platinum and cobalt in fixed ratios. They are used for measuring the color of essentially water-white liquids and are applicable only to liquids in which the color-producing bodies present have light absorption characteristics nearly identical with those of the platinum-cobalt standards.

The same proportions of platinum and cobalt are used to prepare Hazen and APHA color standards. These are the same both numerically and in concentration.

4.4.6.1 Preparation of color standards.- Dissolve 1.245 gm. of potassium chloroplatinate (K_2PtCl_6) and 1.000 gm. of crystallized cobalt chloride ($CoCl_2 \cdot 6 H_2O$) in distilled water containing 100 ml. of 37 percent HCl and make up to 1000 ml. in a volumetric flask with distilled water. This solution will have a Hazen or APHA color of 500. This solution may also be purchased from Fisher Scientific Company, New York City, under the designation Platinum-Cobalt Color Standard Solution Number 500 or APHA Catalog No. SO-P-120 in 4, 16, or 32-ounce quantities.

Prepare the lighter color standards by diluting the 500 standard with distilled water. This is best done in two steps for colors below 50 in order to avoid measurements of extremely small volume of the color standard. See Table II for dilution ratios.

TABLE II
HAZEN OR APHA COLOR STANDARDS

Color Standards	ml of 500 STD	ml Distilled Water
200	40	60
150	30	70
100	20	80
75	15	85
60	12	88
50	10	90
Color Standards	ml of 50 STD	ml Distilled Water
50	100	0
40	80	20
30	60	40
20	40	60
15	30	70
10	20	80
5	10	90
0	0	100

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4.4.6.2 Procedure.- Compare the color of the sample visually with an equal depth of the color standards by looking down through the liquid column. It is recommended that the comparison be made in Nessler tubes, however, it can be performed in any uniformly shaped clean glass bottles using similar bottles for the standards.

Report as the color the number of the standard that most nearly matches the sample. In the event that the color lies midway between two standards, report the darker of the two.

Note: Store the color standards in glass-stoppered bottles, preferably pyrex. These standards are permanent for periods of one to two years if stored in tightly stoppered bottles.

4.4.7 Specific gravity.- The specific gravity of the tolylene -2, 4- diisocyanate shall be determined in accordance with ASTM Method No. D287.

4.4.8 Refractive index.- The refractive index of the tolylene -2, 4- diisocyanate shall be determined at 77°F (25°C) in accordance with ASTM Method No. D901 using an Abbe-refractometer or any other equally accurate instrument.

4.4.9 Acidity. Transfer 100 ml of n-propanol by pipette to a clean dry 250-ml beaker containing a stirring bar on a magnetic stirrer and ~~run~~ on stirrer. By pipet add 10 ml of the tolylene -2, 4- Diisocyanate 1/. Cover the beaker with a watch glass and stir for exactly 10 minutes. Immerse the calomel and glass electrodes of the potentiometer and adjust the pH meter to the pH scale. Titrate the solution with 0.01N methanolic potassium hydroxide by adding 0.05 ml increments from a 10-ml buret. Record apparent pH readings after each incremental addition of reagent. Prepare a graph plotting pH versus ml of reagent added. Take the end point of the titration as the mid-point of that portion of the potentiometric curve that is most nearly at right angles to the volume abscissa. The end point normally occurs between an apparent pH of 5.5 and 7.0. Conduct a blank omitting the tolylene -2, 4- diisocyanate sample. Titrate with 0.01N alcohol potassium hydroxide or 0.01N HCl to the apparent pH of the end point observed with the sample present. 2/ Calculate the acidity as percent HCl in the tolylene -2, 4- diisocyanate as follows:

$$\text{Acidity as percent HCl} = \frac{(A-B) \times N \times 3.65}{\text{ml sample} \times 1.22}$$

Where: A = volume of KOH solution to titrate sample, ml
 B = volume of KOH solution to titrate blank, ml
 N = normality of the KOH solution

1/ If the acidity of the tolylene -2, 4- diisocyanate is less than 0.005 percent HCl, use a .20 ml sample and 200 ml of n-propanol.

2/ If HCl is used to obtain the blank, the volume is added to that of the sample titration.

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4.4.10 Isomer content.- The isomer content (tolylene -2, 4- diisocyanate 1/ or tolylene -2, 6- diisocyanate 2/) shall be determined in accordance with ASTM Method No. D1638 sections 14 to 25 inclusive.

1/ A tolylene -2, 4- diisocyanate of known purity is "Hylene-T" - 98 percent tolylene -2, 4- diisocyanate manufactured by E. I. DuPont de Nemours & Company, Inc. Elastomer Chemicals Department, Wilmington, Delaware.

2/ A tolylene -2, 6- diisocyanate of known purity is the analytical standard, "80 percent tolylene -2, 4- diisocyanate, 20 percent tolylene -2, 6- diisocyanate," manufactured by E. I. DuPont de Nemours & Company, Inc., Elastomer Chemicals Department, Wilmington, Delaware.

4.4.11 Rejection criteria.- If a test specimen fails to meet any of the tests required by this specification, the lot represented by the test sample shall be rejected.

5. PREPARATION FOR DELIVERY

5.1 Packaging.- Packaging shall be Level A or C as specified (see 6.2).

5.1.1 Level A.- The tolylene -2, 4- diisocyanate shall be packaged in oblong 1-gallon cans conforming to Type V, Class 4 of Specification PPP-C-96, in 5-gallon pails conforming to Type I, Class 3 of Specification PPP-P 704, or in 55-gallon drums conforming to Type II of Specification PPP-D-729 (see 6.2).

5.1.2 Level C.- The tolylene -2, 4- diisocyanate shall be packaged in accordance with the manufacturer's commercial practice.

5.1.3 Interior coatings.- The 1-, 5-, and 55-gallon containers shall be coated on the inside with a coating which shall neither affect nor be affected by the tolylene -2, 4- diisocyanate packaged. The supplier shall certify to the contracting officer that the coating used complies with this requirement.

5.2 Packing.- Packing shall be Level A, B, or C as specified (see 6.2).

5.2.1 Level A.- The tolylene -2, 4- diisocyanate, packaged in 1-gallon cans in accordance with 5.1.1, shall be packed for overseas shipment in accordance with the appendix of Specification PPP-C-96. No overpack is required for 5- or 55-gallon containers.

5.2.2 Level B.- The tolylene -2, 4- diisocyanate, packaged in 1-gallon cans in accordance with 5.1.1, shall be packed for domestic shipment in accordance with the appendix of Specification PPP-C-96. No overpack is required for 5- or 55-gallon containers.

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5.2.3 Level C.- The tolylene -2, 4- diisocyanate shall be packed to insure that the shipment arrives in a satisfactory condition at destination. The shipment shall conform to the applicable carriers rules and regulations in effect at the time of shipment.

5.3 Marking.- All unit and shipping containers shall be marked in accordance with Standard MIL-STD-129.

6. NOTES

6.1 Intended use.- The tolylene -2, 4- diisocyanate covered by this specification is intended for use as an ingredient in polyurethane resin homologues.

6.2 Ordering data.- Procurement documents should specify the following:

- (a) Title, number, and date of this specification
- (b) Grade of material (see 1.2)
- (c) Quantity of tolylene -2, 4- diisocyanate required in terms of U. S. gallons
- (d) Type and capacity of containers in which the tolylene -2, 4- diisocyanate is to be furnished, if other than as specified in Section 5
- (e) Applicable levels of packaging and packing with requirements in detail, if other than as specified in Section 5
- (f) Instructions regarding availability of inspection records (see 4.1)

The tolylene -2, 4- diisocyanate shall be furnished by volume, the unit being a U. S. gallon of 231 cubic inches at 60°F (15.6°C).

6.3 Packaging and Packing Levels

6.3.1 Level A - Military Pack.- This level shall be used for those items which are for shipment, handling, indeterminate storage, and world-wide redistribution.

6.3.2 Level B - Limited Military Pack.- This level shall be used only when it is definitely known that the item will be shipped and handled under cover and stored in warehouses or other structures affording equivalent protection from weather.

6.3.3 Level C - Minimum Military Pack.- This level shall be used only when it is definitely known that the packaged item is to be shipped from the supply source to the first receiving activity for immediate use.

6.4 Toxicity.- Precautions for the handling of liquid diisocyanates are thoroughly discussed in "Chemical Safety Data Sheet SD-73, Properties and Essential information for Safe Handling and Use of Tolylene Diisocyanate" issued by the Manufacturing Chemists' Association, Inc.

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Notice: When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any other person or corporation or conveying any rights or permission to manufacture, use, or sell any patented invention that may in any way be related thereto.

SPECIFICATION ANALYSIS SHEET
 NAVWEP8 FORM 4121/3 (8-62)

 FORM APPROVED
 BUDGET BUREAU NO. 45-R309

INSTRUCTIONS

This sheet is to be filled out by Government or contractor personnel involved in the use of this specification in procurement of products for ultimate use by the activity shown on the reverse of this sheet.

of this specification suitable products can be procured with a minimum amount of delay and at the least cost.

Comments and the return of this sheet will be appreciated.

This sheet is provided for obtaining information which will aid the activity shown in insuring that through use

Fold on dotted lines on reverse side, staple in corner, and send to the activity shown.

SPECIFICATION (No. and abbrev. title)

ORGANIZATION		CITY	STATE
CONTRACT NUMBER	QUANTITY OF ITEMS PROCURED		DOLLAR AMOUNT \$
MATERIAL PROCURED UNDER A DIRECT GOVERNMENT CONTRACT		OR A SUBCONTRACT	

1. HAS ANY PART OF THIS SPECIFICATION CREATED PROBLEMS OR REQUIRED INTERPRETATION IN PROCUREMENT USE?
 (If so, give paragraph number and wording, and recommendations for correcting the deficiencies.)

2. COMMENTS ON ANY SPECIFICATION REQUIREMENT CONSIDERED TOO RIGID

3. IS THE SPECIFICATION RESTRICTIVE?

YES NO

IF YES, IN WHAT WAY?

4. REMARKS (Attach any pertinent data which may be of use in improving this specification. If additional papers are attached, send this form and papers in an envelope. This form is addressed for use in window envelope when appropriate.)

SUBMITTED BY (Print name and title)

DATE

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