MILITARY SPECIFICATION

CURING AGENTS, DIMERYL-DI-ISOCYANATE AND ISOPHORONE DI-ISOCYANATE

This specification is approved for use by the Naval Air Systems Command, Department of the Navy, and is available for use by all Departments and Agencies of the Department of Defense.

1. SCOPE.

1.1 Scope. This specification establishes the requirements for two types of di-isocyanate curing agents.

1.2 Classification. The curing agents shall be of the following types:

Type I	Dimeryl-di-isocyanate (DDI)
Type II	Isophorone di-isocyanate (IPDI)

2. APPLICABLE DOCUMENTS.

2.1 Issues of documents. The following documents of the issue in effect on date of invitation for bids or request for proposal, form a part of the specification to the extent specified herein.

STANDARDS

MILITARY

MIL-STD-129	Marking for Shipment and Storage.
MIL-STD-1218	ACS Chemicals.

(Copies of specifications, standards, drawings and publications required by contractors in connection with specified procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

Beneficial comments (recommendations, additions, deletions) and any pertinent data which may be of use in improving this document should be addressed to: Commanding Officer, Naval Air Engineering Center, Engineering Specifications and Standards Department (ESSD) Code 93, Lakehurst, NJ 08733, by using the self-addressed Standardization Document Improvement Proposal (DD Form 1426) appearing at the end of this document or by letter.

FSC 6810

2.2 Other publications. The following document forms a part of this specification to the extent specified herein. Unless otherwise indicated the issue in effect on date of invitation for bids or request for proposal shall apply.

STANDARDS

American Society for Testing and Materials (ASTM)

ASTM-D-1638	Urethane Foam Isocyanate Raw Materials.

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

3. REQUIREMENTS.

3.1 Product characteristics. The curing agents shall be clear liquids with the color of light straw.

3.2 Chemical properties. Chemical properties of the curing agents shall conform to the requirements of Table I.

TABLE I. Chemical properties.

Property	Type I		Type II	
	Min	Max	Min	Max
NCO content, percent	13.50	14.30	37.1	37.8
Moisture, percent	---	0.02	---	---
pH	5.0	6.5	5.0	7.0
Hydrolyzable chlorine, percent	---	0.05	---	0.02

3.3 Infrared spectrum. An infrared spectrum shall be made for Type I and Type II materials in accordance with 4.4.3. The infrared spectrum shall qualitatively conform to Figures 1 and 2.

3.4 Stability. When packaged in accordance with 5.1 and stored in the temperature range of 5 to 22 degrees Celsius (°C), the curing agents shall meet the requirements of this specification for a minimum of 6 months after acceptance. The shelf life may be extended for 6-month intervals after reacceptance testing for conformance to the chemical property requirements in Table I as follows:

a. Type I - All properties.

b. Type II - NCO content and pH.

3.5 Toxic products and safety. Safety regulations and guidelines applicable to the use of the curing agents should be complied with to preclude personal injury and damage to equipment and facilities.

3.6 Workmanship. Workmanship shall be such that the curing agents are uniform in appearance, of consistent high quality and free from visible contamination.

4. QUALITY ASSURANCE PROVISIONS.

4.1 Responsibility for inspection. Unless otherwise specified in the contract or purchase order, the contractor shall be responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the contractor may utilize his own facilities or any commercial laboratory acceptable to the Government. The Government reserves the right to perform any of the inspections set forth in this specification where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements.

4.2 Sampling. The lot shall be sampled in accordance with Table II.

TABLE II. Sampling plan.

Number of containers in lot	Number of containers sampled (primary sample)	Number of Composite samples
100 or more	10% (nearest whole number)	5
51 - 99	10	4
11 - 50	10	3
1 - 10	ALL	2

4.2.1 Primary samples. Physical property tests shall be run on each primary sample (see Table II). The material container may be sampled by use of a clean glass tube, rod, or pipet. If the container is small enough to be handled safely, the sample may be obtained by pouring. The smallest sample size possible that is consistent with test requirements shall be taken. The minimum sample size shall be two ounces. Glass containers shall be used for all liquid samples. Each sample shall be labeled with date, lot number, and manufacturer's container identification number. Failure of any primary sample to pass all of the physical properties tests herein shall result in rejection of the lot represented.

4.2.2 Composite samples. Chemical properties tests shall be run on each composite sample. Divide the primary samples equally into the number of composites shown in Table II. Blend each composite thoroughly by manipulation of the container. Label each composite with Roman numerals, also include date, lot numbers, and manufacturer's container identification numbers. The remainder of the primary samples shall be retained pending acceptance or rejection of the lot. Failure of any composite sample to pass all of the chemical properties tests herein shall result in rejection of the lot represented.

4.3 Quality conformance inspections and tests. Quality conformance inspections and tests shall consist of the following:

a. Tests of Table I properties (see 4.4 and 3.3).

b. Inspection of filled containers (see 4.5.1).

c. Visual inspection (see 4.5.2).

4.4 Test methods. Tests shall be performed using apparatus, reagents, and procedures specified herein. The use of alternate apparatus, reagents, or procedures shall require prior written approval of the procuring activity. All American Chemical Society (ACS) reagents shalll conform to MIL-STD-1218.

4.4.1 Isocyanate (NCO) content.

4.4.1.1 Apparatus.

a. Magnetic stirrer, variable speed, with Teflon-coated stirring bars, or equal.

b. Iodine flask, 250-milliliter (ml).

c. Pipet, 5-ml.

4.4.1.2 Reagents.

a. Isopropanol, ACS reagent.

b. Toluene, ACS reagent (dried over silical gel).

c. Di-n-butylamine (Eastman No. 1260 or equal) solution, 1 Normal (N) (25.8 grams (gm) in 200 ml of dry toluene).

d. Standardized hydrochloric acid in isopropanol, 0.2 N (use tris hydroxymethylamino methane (THAM) procedure for standardization).

e. Bromphenol blue indicator solution (0.01 gm in 25 ml of ethanol).

f. Alcoholic potassium hydroxide solution, 0.5 N.

g. Phenolphthalein.

h. Nitric acid solution, 8.0 N.

i. Standard silver nitrate solution, 0.02 N.

j. Nitrobenzene.

k. Forty percent ferric ammonium sulfate indicator solution.

l. Potassium theocyanate solution, 0.01 N.

4.4.1.3 Determination of isocyanate. Weigh accurately 0.6 to 0.8 gm for Type I or 0.15 to 0.17 gm for Type II into a 250-ml iodine flask. Add 50 ml of dry toluene to the sample flask and to two other flasks to be used as blanks. Pipet 5 ml of 1 N di-n-butylamine solution into the flasks, replace the stopper, stir with a magnetic stirrer, and let stand 30 minutes. Add 50 ml of isopropanol and 0.5 ml of bromophenol blue indicator solution. Titrate with standardized hydrochloric acid in isopropanol solution to the yellow end point.

Calculation:

$$\text{Percent NCO} = \frac{(B-V) \times N \times 4.202}{W}$$

Where: B = average volume of standardized hydrochloric acid for blanks, ml

V = volume of standardized hydrochloric acid for sample, ml

N = normality of standardized hydrochloric acid solution

W = weight of sample, gm

Report the NCO to the nearest 0.01 percent.

4.4.2 Moisture.

4.4.2.1 Apparatus. Beckman "Aquameter", Model KF-4, or equal.

4.4.2.2 Reagents.

a. Karl Fischer (KF) reagent, commercial strength.

b. Standardized KF reagent, stabilized, water equivalent of 2.0 to 3.0 milligrams/milliliter (mg/ml).

c. Pyridine, ACS reagent.

d. Chloroform, ACS reagent.

4.4.2.3 Determination of moisture. To the reaction beaker containing 60 ml of a 1:1 ratio neutralized pyridine-chloroform solution, add 9 to 10 gm of sample which is weighed by difference to the nearest 0.01 gm. The stirrer should be off while adding the sample. Turn on the stirrer to the same position used in the neutralization. Titrate automatically by pressing the titrate button to a 30-second end point.

Calculation:

$$\text{Percent moisture} = \frac{V \times E}{10 \times W}$$

Where: V = volume of standardized KF reagent, ml

E = water equivalent of KF reagent, mg/ml

W = weight of sample, gm

Report the average of duplicate analyses to the nearest 0.001 percent.

4.4.3 Infrared spectrum.

4.4.3.1 Apparatus. Infrared spectrophotometer, Perkin-Elmer Model 21 or equal, equipped with sodium chloride windows.

4.3.3.2 Procedure. Prepare a smear of the sample to be tested on the sodium chloride windows in a thin continuous film. Scan the infrared spectrum from 4000 centimeters^{-1} (2.5 micrometers) to 666 centimeters^{-1} (15 micrometers) for the material being tested. Compare the spectrum obtained with Figure 1 for Type I or Figure 2 for Type II.

4.4.4 pH.

4.4.4.1 Apparatus.

a. pH indicator, Leeds and Northrup, No. 7664, or equal, equipped with Calomel electrode standard 1199-31 and glass electrode standard 1199-30 or equal.

b. Separatory funnel, 250-ml.

4.4.4.2 Reagents.

a. Sodium hydroxide, 0.01 N.

b. Distilled water, pH 7.0.

c. Hydrochloric acid, 0.01 N.

d. Standard buffer solution, pH 7.0.

4.4.4.3 Instrument standardization. Adjust the pH indicator to read a pH of 7.0 using the standard buffer solution.

4.4.4.4 Preparation of pH of 7.0 distilled water. Adjust the pH of 200 ml of distilled water to 7.0 with approximately 0.01 N sodium hydroxide or hydrochloric acid.

4.4.4.5 Determination of pH. Pour 50.0 ml of sample into a 250-ml separatory funnel and add 50.0 ml of 7.0 pH distilled water. Stopper the funnel and shake vigerously for 1 minute. Let stand for 5 minutes or until separated in definite layers, draw off the lower phase for Type I and upper phase for Type II into a 150-ml beaker, and determine the pH of the aqueous solution by using the pH meter and following the instructions supplied with the instrument. Report the pH to the nearest 0.1 pH unit.

4.4.5 Hydrolyzable chlorine.

4.4.5.1 Type I. Hydrolyzable chlorine shall be determined in accordance with ASTM-D-1638, Methods 40 through 46. Report hydrolyzable chlorine to the nearest 0.01 percent.

4.4.5.2 Type II. Hydrolyzable chlorine shall be determined as follows:

a. Weigh 5.0 gm to the nearest mg sample into a round bottom flask.

b. Add 50.0 ml of 0.5 N alcholic potassium hydroxide solution and reflux for 45 minutes; cool the solution, add several drops of phenolphthalein, and make slightly acidic by adding concentrated nitric acid from a dropping bottle.

c. Add 5.0 ml of 6 N nitric acid solution.

d. Add exactly 10.0 ml of standard 0.02 N silver nitrate solution from a buret.

e. Add 5.0 ml of nitrobenzene plus 2.0 ml of 40-percent ferric ammonium sulfate indicator solution.

f. Back titrate to first reddish coloration with standard 0.01 N potassium thiocyanate solution.

g. Calculate hydrolyzable chlorine as follows:

Percent hydrolyzable chlorine =

$$\frac{(V_1 \times N_1 - V_2 \times N_2) \times 3.546}{W}$$

Where: V_1 = volume of standard silver nitrate, ml

N_1 = normality of standard silver nitrate

V_2 = volume of standard potassium thiocyanate, ml

N_2 = normality of standard potassium thiocyanate

W = weight of sample, gm

Report hydrolyzable chlorine to the nearest 0.01 percent.

4.5 Examinations.

4.5.1 Inspection of filled containers. All filled containers shall be inspected prior to shipment or use for accuracy of markings and for defects in containers and closures. All defective containers and closures shall be repaired or replaced, and contents therein shall be reinspected prior to shipment or use.

4.5.2 Visual inspection. All samples shall be visually inspected to determine conformance to the requirements of 3.6.

4.6 Records. Certification and test data shall be prepared as required by the procuring activity (see 6.2.2).

5. PACKAGING.

5.1 Packaging and packing. Unless otherwise specified in the purchase order (see 6.2.1), packaging and packing of the curing agents shall be in accordance with commercial practice to insure carrier acceptance and shall be of such construction and materials that the contents will be adequately protected against loss or contamination.

5.2 Marking for shipment. Unless otherwise specified in the purchase order (see 6.2.1), each shipping container shall be marked in accordance with the requirements of MIL-STD-129. Container marking shall include the following:

a. The supplier's lot number.

b. Procuring activity purchase order number.

c. Container identification (applied in numerical sequence as the containers are filled).

d. Date of manufacture.

e. Manufacturers' Code Ident.

f. Net and tare weight of the container.

g. Material identification.

WARNING

Toxic material. Avoid skin contact.
Do not breathe vapors.

6. NOTES AND CONCLUDING MATERIAL.

6.1 Intended use. The intended uses of the material described herein are as curing agents in solid propellant and rocket motor case liner formulations.

6.2 Ordering data.

6.2.1 Procurement requirements. Procurement documents should specify the following:

a. Title, number and date of this specification.

b. Responsibility for inspection and inspection facilities if different than 4.1.

c. Special packaging, packing, or shipping requirements, if applicable (see Section 5).

6.2.2 Data requirements. When this specification is used in a procurement which incorporates a Contract Data Requirements List (DD Form 1423) and invokes the provisions of 7-104.9(n) of the Defense Acquisition Regulations (DAR), the data requirements identified below will be developed as specified by an approved Data Item Description (DID) (DD Form 1664) and delivered in accordance with the approved DD Form 1423 incorporated into the contract. When the provisions of DAR-7-104.9(n) are not invoked, the data specified below will be delivered by the contractor in accordance with the contract requirements. Deliverable data required by this specification is cited in the following paragraphs:

Paragraph	Data Requirement	Applicable DID
4.6	Certification Test Data	UDI-A-23264B DI-T-4024

(Copies of DIDs required by the contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting officer.)

6.3 Definitions.

6.3.1 Lot. At place of manufacture, a lot consists of one batch (see 6.3.2) or a uniform blend of two or more batches. At place of delivery, a lot consists of curing agent from one supplier's lot received in a single shipment. Partial shipments may be considered as a single shipment by the procuring activity.

6.3.2 Batch. A batch consists of one type of curing agent made as one unit in an unchanged manufacturing process.

6.4 Suggested source of supply. A product that has met the requirements of this specification in past procurement actions is manufactured by the Henkel Corporation, 4620 W. 77th Street, Minneapolis, Minnesota 55435 for Type I and the Thorson Chemical Corporation, 645 5th Avenue, New York, NY 10022 for Type II. This information is for the convenience of the procuring activity and is not to be construed as a waiver of any requirement of this specification nor as any limitation of additional potential sources of supply.

Preparing activity:
Navy - AS

(Project 6810-NB24)

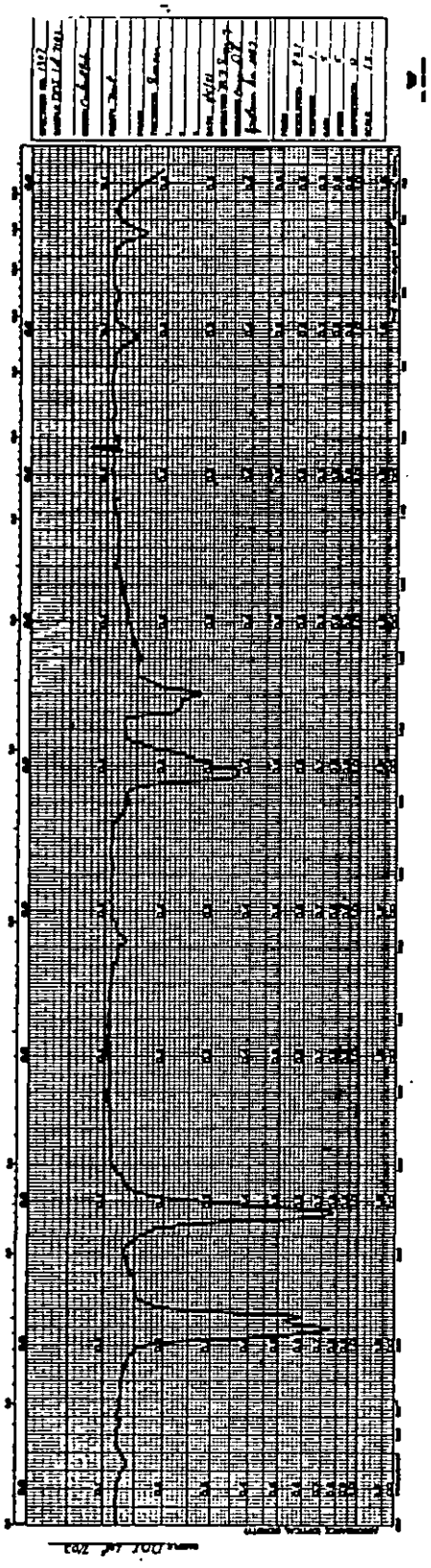

Figure 1. Typical infrared spectrum (Type 1).

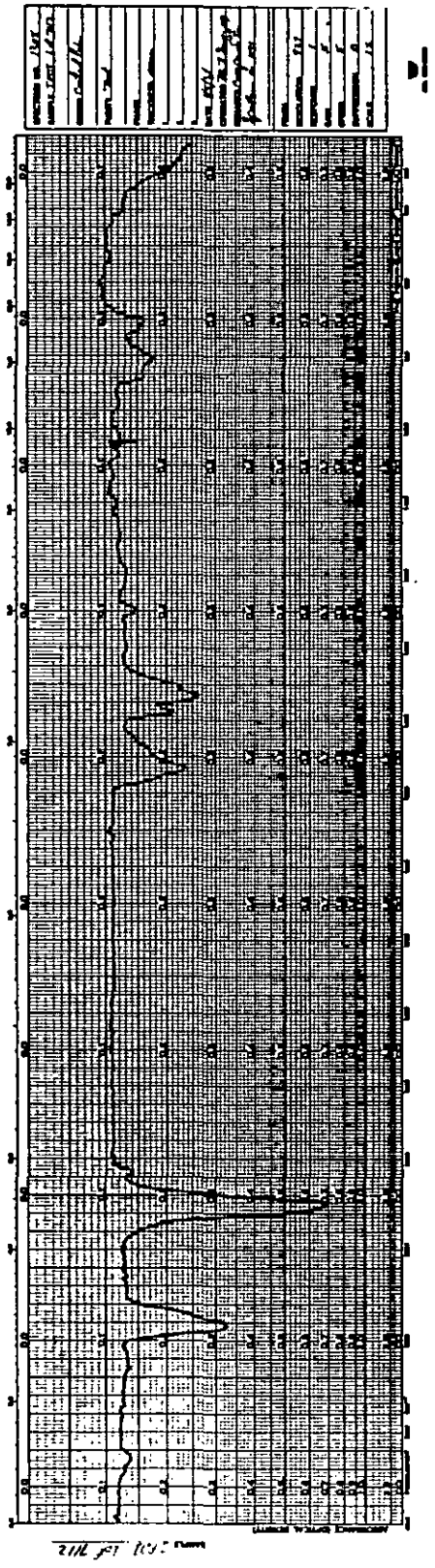

Figure 2. Typical infrared spectrum (Type II).

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