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FLEXIBLE URETHANE RESINS AND COATING MATERIALS DEVELOPMENT (U)

FINAL REPORT

25 February 1970 - 24 February 1971

by

J.E. Pregler

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13. ABSTRACT

Research studies were conducted on polyurethane coating formulations to develop thermally stable, low temperature flexible films that would be non-yellowing when exposed to weathering.

A series of different polyols and crosslinking agents were reacted with an aliphatic diisocyanate, cured into films and physical properties determined. Results of this investigation enabled the selection of the most thermally stable polyol components.

Diols, triols and tetrols were investigated. They included polyether, polyester and hydrocarbon backbone structures.

Polyesters in combination with an isocyanurate crosslinker provided the most thermally stable polyol combination when reacted with 4,4-methylene bis(cyclohexylisocyanate) and cured.

In addition to thermal stability and low temperature flexibility, abrasion resistance, energy of rupture, tensile properties at -65°F and 70°F, solvent resistance and accelerated weathering properties were determined.

Non-photochemically reactive solvents were formulated which conform to "Rule 66" legislation.

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4,4'-Methylene bis(cyclohexylisocyanate)						
Heat Stability						
Tris (2-hydroxyethyl) isocyanurate						
Polycaprolactone						
Physical Properties						
Crosslinked						

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Report Period: 25 February 1970 - 24 February 1971

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SUMMARY

A non-yellowing moisture cured urethane coating was developed from 4,4'-methylene bis(cyclohexylisocyanate), poly (epsilon-caprolactone), and crosslinked with tris(2-hydroxyethyl) isocyanurate at a 2:1 NCO:OH ratio. It possesses good abrasive resistance, low temperature (-65°F) flexibility, tensile strength, solvent resistance, elongation and 300°F thermal stability.

This coating was developed after screening the following polyols and crosslinking agents for the thermal stability they contributed to a urethane film:

POLYOLS	CROSSLINKING AGENTS
Polycaprolactones of 530, 840 and 1250 mol.wt.	Mondur HC (triisocyanate)
Polyoxyethylene glycols of 300, 600 and 1000 mol.wt.	Desmondur N (triisocyanate)
Polyoxytetramethylene glycols (650 mol.wt.)	Quadrol (tetrol)
Polyoxypropylene glycols (450 and 750 mol.wt.)	Pentaerythritol (tetrol)
Desmophen 650 (polyester)	Poly-G 630 PG (triol)
RD-18 (polyester)	Trimethylolpropane (triol)
	Castor Oil DB (triol)
	1,2,6-Hexanetriol
	Tris(2-hydroxyethyl) isocyanurate (triol)

Screening was accomplished by measuring the films for tensile strength and elongation, both before and after thermal aging (24 hours @ 300°F), and comparing the results obtained against a standard control system or formulation. This study led to the selection of tris(2-hydroxyethyl) isocyanurate [THEIC] and the polycaprolactones of 530 and 840 molecular

weights as the materials contributing the greatest to film stability. These candidate raw materials were formulated in various combinations with 4,4'-methylene bis(cyclohexylisocyanate) [$H_{12}MDI$] and from this study three potential candidate systems were selected and completely tested, with the best performing system being picked as the final candidate system.

The solvent system chosen for the candidate system consists of 40% p-dioxane, 20% cellosolve acetate, 20% cyclohexane and 20% butyl acetate. The pigment selected was titanium dioxide at a tentative level of 25% based on the resin solids. The candidate system contains 52.2% non-volatiles by weight.

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I. INTRODUCTION

A. Background

The Naval Air Systems Command is interested in obtaining a low temperature flexible, general purpose aircraft coating. The coatings currently in use, yellow after 18 months service and often crack or split along the fuselage seams, requiring frequent and costly maintenance.

One explanation (Philadelphia Navy Yard) holds that cracking occurs when the coatings become brittle at low temperatures (-40°F) and cannot adjust to the various stresses set up in the metal fuselage. It is felt that a coating which remains flexible at -65°F would not crack under conditions encountered during normal in-service use. Aromatic polyurethane coatings are known to possess outstanding low temperature flexibility, but have not gained wide acceptance because they discolor after a short time in service and this prevents the compounding of coatings with stable colors.

Many polyurethane coatings were, and are, based on aromatic isocyanates. The primary source of discoloration in urethane systems is often attributed to the aromatic isocyanate structure. In the past few years it was found that polyurethanes made from aliphatic isocyanates yellowed only slightly, if at all. As a result, it is now possible to construct non-yellowing polyurethane coatings with a wide range of properties, but so far no system has been designed with both low temperature flexibility (-65°F) and high temperature stability.

Under Contract N00019-69-C-0093 [1] we developed a non-yellowing, general purpose aircraft coating from a polytetramethylene ether glycol [4] and 4,4'-methylene bis(cyclohexylisocyanate) [$H_{12}MDI$]. It had outstanding strength, abrasion resistance, and low temperature flexibility (-65°F), but discolored when heated for 24 hours at 300°F and had poor weathering characteristics.

It was our intention, and the purpose of this contract, to develop a coating which was non-yellowing and possessed both low temperature flexibility and high temperature (300°F) stability.

Thermal stability is dependent upon the stability of the chemical bonds involved in building the polymer. Crosslinking contributes to thermal stability by increasing the polymer softening point, preventing melting, and presumably resisting rapid oxygen penetration and assimilation by the polymeric system. Most film degradation starts with oxygen attack on specific chemical bonds, causing them to rupture. If oxygen attack can be retarded, or confined mainly to the film surface, the polymer's upper working range can be extended.

It is, therefore, quite apparent that the types of materials used, and the amount of crosslinking existing within the polymer backbone, are quite important.

B. Approach

Since a non-yellowing coating was desired,this restricted us to using aliphatic isocyanates. Work on Contract N00019-69-C-0093 performed here at Olin showed that 4,4'-methylene bis(cyclohexylisocyanate) [$H_{12}MDI$] is the best choice among aliphatic isocyanates to develop a non-yellowing flexible urethane coating. This then leaves only two major areas to investigate; 1) the stability, amount and type of crosslinking to use and 2) the stability of various polyols.

A logical point to start this program was to use the coating formulation developed under Contract N00019-69-C-0093 as a reference base or control, because it offered many of the physical properties we were targeted to achieve for the coating system under this contract. (See Appendix I) We introduced crosslinking into the control formulation, investigating the effects of crosslinking the polymer backbone through

the isocyanate structure and through the polyol structure. We used percent retention of the polymer's original physical properties after thermal aging as a prime criterion for measuring the effect of a crosslinking agent. This led to the selection of several stable crosslinking agents.

Next, we examined different types of polyols by substituting them into a standard formulation containing a single crosslinking agent and using the same evaluation standards we used for the crosslinking agents. This permitted the selection of several stable polyols.

The selected polyols and crosslinking agents were next formulated in various combinations with each other and the resulting films were again evaluated for retention of properties after thermal aging. This permitted the selection of a single polyol and a single crosslinking agent which were in turn formulated together with $H_{12}MDI$ at several different levels to achieve a range of properties. Those systems having properties most nearly matching the parameters of this contract were thoroughly tested, with the best performing system being selected as the candidate system.

II. RESULTS AND DISCUSSION

A. Evaluation of Crosslinking Agents

At the start of this program we investigated the effect of crosslinking within two different systems; 1) a one-shot or one-package system, and 2) a moisture-cured prepolymer-based system. The one-shot system involved mixing together all of the reagents 4,4'-methylene bis(cyclohexylisocyanate), Polymeg 650 and the crosslinking agent) in a modified MIL Thinner, (45% toluene, 45% MIBK, 5% xylene and 5% Cellosolve Acetate) at an NCO:OH ratio of 1.1:1, catalyzing with 1% D-22 (based on the resin solids) and casting a film. The control was made by mixing $H_{12}MDI$ and Polymeg 650 together in modified MIL Thinner at an NCO:OH ratio of 1.1:1 catalyzing and curing into a film.

The one-shot systems were chosen to eliminate a dependence upon moisture for cure. But, this goal was not achieved since, in all cases the properties of the uncrosslinked control were better than the crosslinked systems. The one-shot approach was not fruitful, and no further one-shot systems were examined. See Tables I through IV for data concerning their formulation and physical properties.

For the moisture-cured systems, a standard prepolymer formulation was used and appears as follows:

REACTANTS	10 EQ. % FORMULATION	20 EQ. % FORMULATION
4,4'-Methylene bis(cyclohexylisocyanate)	0.382 Eq.	0.382 Eq.
Polymeg 650	0.172 Eq.	0.153 Eq.
Crosslinking Agent	0.019 Eq.	0.038 Eq.

The crosslinking agent was substituted into this formulation at levels of 10 equivalents percent and 20 equivalents percent.

There are many ways to express or indicate the relative amount of crosslinking in a polymer system. There are, for example, weight percent, mole percent, and crosslink density, to name a few. None of these methods permitted a direct comparison between systems using reactants of differing formula weight and functionality.

In this investigation, the crosslink levels were compared on an equivalents percent basis, and is defined as the percent of the equivalents supplied by the crosslinking agent as compared to the total equivalents of like functional groups present in the system. This approach had these advantages: 1) It did not disturb the ratio or distribution of urea to urethane bonds. 2) It did not require the introduction of additional compounds in order to adjust the crosslink density. 3) It permitted evaluating crosslinking agents as a single variable by keeping

the percent of total bonds, actually involved in supplying crosslinks constant between systems. The disadvantages to this approach were: 1) It caused minor changes in the crosslink density. 2) It caused changes in the polymer formula weight. However, the differences in crosslink densities caused negligible changes in the physical properties compared to those caused by the crosslinker.

Changes in molecular weight of the polymer may cause slight changes in flexibility or elongation. These changes were not large enough, when compared to the overall formula weight to be measured. One primary interest area was thermal stability, and the drawbacks cited above do not relate to this property.

The following materials were evaluated as crosslinking agents:

COMPOUND	FORMULA WT.*	EQUIVALENT WT.	FUNCTIONALITY
Poly-G 630PG	590	197	3
Quadrol	292	73	4
Trimethylol-propane	134	44.7	3
1,2,6-hexanetriol	134	44.7	3
Pentaerythritol	136	34	4
Mondur HC **	1113	371	3
Desmodur N **	840	280	3
Tris(2-hydroxyethyl) isocyanurate [THEIC]	261	87	3

* Based on OH number or amine equivalent

** Isocyanates

The above crosslinking agents were reacted with Polymeg 650 and $H_{12}MDI$ to form a prepolymer at an NCO:OH ratio of 2:1 in modified MIL Thinner. For the control we used the same prepolymer system at the same NCO:OH ratio but omitted the crosslinking agent. Test films were prepared

by catalyzing with 1% D-22 (based on resin solids), casting and curing with moisture. All films were aged for at least 72 hours before testing for two reasons; 1) a target requirement was to achieve cure in this time period; 2) previous experience has shown that this permits about 90% of the ultimate properties to develop. In the early stages of this program, only selected screening tests were performed on the cured films. A list of the tests and properties measured follows:

TEST	PROPERTIES INDICATED
Tensile Modulus	Elastomeric Properties
Tensile at Break	Strength
Elongation	Flexibility at Room Temperature
Conical Mandrel	Flexibility at -65°F
Shore A	Hardness
Heat Age 24 Hours @ 300°F	Thermal Stability
Energy of Rupture	Toughness

These tests permitted rapid assessment of crosslinking agents nal for improvement in hardness and thermal stability.

Tables V and VII list the materials used to prepare the moisture-cure prepolymers for evaluating all crosslinking agents except THEIC. The THEIC information can be found in Table IX. Included in these tables are reactant stoicheometry, theoretical (calculated) percent free NCO and the actual free NCO found. The test data from the cured films appears in Tables VI, VIII and X and shows how the physical properties changed as the amount of crosslinking varied from 0 to 20 equivalents percent. As crosslinking increased, the values of the film's hardness, thermal stability and tensile modulus also increased, while

the values for elongation, energy of rupture and tensile-at-break decreased. These changes, with the exception of thermal stability, were most pronounced when the system was crosslinked by means of a triol or tetrol (Table VI) and least pronounced when crosslinked by means of a triisocyanate (Table VIII).

The addition of a crosslinking agent to a prepolymer formulation always resulted in the cured film having increased thermal stability and slightly decreased tensile strength. For example, the uncrosslinked control (see Run 1099406 Table VI) lost 67% of its original elongation after thermal aging (24 hours @ 300°F) while none of the crosslinked films lost more than 50% of their original elongation. Many films, which showed good retention of elongation after thermal aging, showed very poor retention of tensile strength. Unless a film showed good retention of both tensile strength and elongation after thermal aging, it was rejected as not suitable, even though this contract required only a 50% retention of elongation as being necessary.

Furthermore, the acceptability of a crosslinking agent, in addition to its performance, was based upon its ease of utilization (solubility, lack of gel formation, etc.).

Poly-G 630PG was eliminated from further consideration because its films constantly showed a high loss of tensile strength after thermal aging.

Mondur HC and Desmondur N (triisocyanates) were both rejected because of limited solubility at the desired levels of use, coupled with sub-par-performance during testing (see Table VIII and XII). 1,2,6-Hexanetriol and trimethylolpropane were also eliminated for lack of solubility.

Pentaerythritol and THEIC had a similar solubility problem, but, because their films showed such good properties, they were selected for further evaluation studies in combination with various stable

polyols. Pentaerythritol showed the best retention of tensile strength at 20 eq. % for any crosslinking agent examined. But, because of solubility, its use was limited to a maximum level of 20 eq. %.

The films containing tris(2-hydroxyethyl) isocyanurate showed the best combined retention of tensile strength and elongation after thermal aging, ranking first in tensile strength and second in elongation at a level of 10 eq. % (see Table XIII).

One additional crosslinking agent, Quadrol, was selected. After thermal aging, its films show good retention of elongation (87% at 20 eq. %) and almost no discoloration. It showed the best solubility of all the crosslinking agents evaluated.

B. Polyol Evaluation

In order to evaluate the available polyols, a standard formulation, containing a single crosslinking agent, was set-up, the various polyols substituted into it and a prepolymer formed (see Table XIV for a list of formulations). The prepolymer was then catalyzed, moisture cured into a clear film, and tested for retention of elongation and tensile strength after thermal aging for 24 hours @ 300°F. The following formulation was used as standard:

Compound	Equivalents
4,4'-Methylene bis(cyclohexylisocyanate)	0.382
Quadrol	0.019
Polyol	0.172

NCO:OH Ratio 2:1

Quadrol was selected as the crosslinking agent for the above formulation because it had both a favorable reaction rate and good solubility

in the normal urethane solvents.

This method permits a relative comparison between the polyols being evaluated for thermal stability and not an absolute evaluation of a polyol. For example, some polyols might be thermally stable in and of themselves, but not make a film which is stable.

The following polyols were examined:

Polymeg 650

Niax 510, 520 and 540

Carbowax 300, 600 and 1000

Poly-G 420P and G 750P

Castor Oil DB

Castor 1066

Polybutene 1000

Desmophen 650

RD-18

Polyols such as Desmophen 650 and RD-18 had limited solubility, and when films could be formed, they had good thermal stability but were hard and brittle. Attempts to make flexible films containing these two polyols always resulted in loss of thermal stability (see Table XVI).

Castor 1066 is a castor oil-based product from Spencer Kellogg while Polybutene 1000 is a hydrogenated hydroxy-polybutadiene resin. Neither formulated polyol gave acceptable products after thermal aging. The film containing Castor 1066 discolored badly while the film based on Polybutene 1000 melted in one case and lost nearly all of its physical properties in the other (see Tables XI and XII; Run F-9396 and F-9398, 99 for data on formulation and physical properties).

The remainder of the films yielded the following data:

AFTER 24 HOURS AT 300°F*

Ref. No.	Polyol	% Tensile Lost	% Elongation Lost	Gardner Color Standard
F-9366	Polymeg 650	80.8	22.7	11
F-9400	Niax 510	32.7	0	6
1099453	Niax 520	65.7	0	6
1099405	Niax 540	83.1	0	6
1099407	Carbowax 300	Brittle	Brittle	4
1099408	Carbowax 600	81.6	83.8	6
1099409	Carbowax 1000	95.3	97.0	11
1099419	Poly-G 420P	Brittle	Brittle	2
1099424	Poly-G 750P	82.4	95.8	4
1099443	Castor Oil DB	10.6	0	8

*Heat aged as unpigmented free films

This data indicates a correlation between the molecular weights of the polyol and the percent retention of tensile strength of the cured films after thermal aging. As the molecular weight of a given polyol family increased, the percent retention of tensile strength decreased. In other words, the greater the molecular weight within a polyol family the more thermolabile the polymer will be (see Figure 1).

The thermal decomposition of a polymer will often result in colored by-products which tint the entire polymer, causing problems in colorfastness. Therefore, a polymer which discolors excessively under thermal conditions was to be avoided, even though it showed no loss of physical properties. The amount of discoloration that the above films acquired were compared by use of the Gardner Color Standard.

This permited a relative comparison between the various polyol-containing systems. A significant amount of discoloration was not present until the color values got above five, and, for the purposes of a thin film, values as high as seven could be masked by pigmentation.

The discoloration values of 6 for the Niax-containing systems were within acceptable limits, while the value of 8 for the Castor Oil DB system was just a shade high. Three other systems, containing respectively Carbowax 300, Poly-G 420P and Poly-G 750P, had lower color values (i.e. better) than the Niax or Castor Oil DB systems, but they had very poor physical properties after thermal aging.

Only the Niax (510, 520) and Castor Oil DB systems showed acceptable physical properties coupled with good color values. The Castor Oil DB system lost only 10.6% of its original tensile strength and no elongation after thermal aging while the Niax polycaprolactones (510 and 520) lost 32.7% and 65.7% of their original tensile strength and no elongation (see Table XV for a summary of physical properties). As a result of this study Niax 510, Niax 520 and Castor Oil DB were selected as candidate polyols for further evaluation.

C. Selection Of The Most Thermally Resistant Building Blocks

Only Quadrol, pentaerythritol, tris(2-hydroxyethyl) isocyanurate, Castor Oil DB and the Niax polycaprolactones (510 and 520) out of the many materials tested provided significant film thermal stability.

These candidate materials were formulated in various workable combinations with $H_{12}MDI$ at a 2/1 NCO/OH ratio to form prepolymers, which were in turn moisture cured into films. These were aged 72 hours and then their physical properties were measured both before and after thermal aging (24 hours at 300°F). See Tables XVI and XVII.

This investigation resulted in selection of stable building blocks. Runs F-9400 (Niax 510), 1099453 (Niax 52.) and 1099405 (Niax 540) (Table XVII) shows the Niax 510 (Mol. wt. 530) system retained 67.7% of its original tensile strength while the Niax 520 (Mol. wt. 840) retained 34.2% and the Niax 540 (Mol. wt. 1250) only 17.4%. As the molecular weight of the polycaprolactone in the formulation increased, the thermal stability of the coating decreased. Another way of saying this is that as the equivalent weight per crosslink (eq.wt./cl.) increases, the thermal stability, as determined by measuring the percent retention of tensile strength after thermal aging, decreases (see Figure 1).

Table XVIII contains a listing of formulations arranged according to increasing equivalent weight per crosslink. The systems selected for inclusion in the table contain either one or more of the selected candidate raw materials or Polymeg 650 (control) in their formulations.

A comparison of F-9400 (Niax 510, H_{12}MDI, Quadrol 10 eq. %) and F-9367 (Polymeg 650, H_{12}MDI, Quadrol 10 eq. %) shows that after thermal aging they retained 67.7% and 19.2% respectively of their original tensile strength. The eq.wt./cl. of the two polymers are nearly equal; 10,300 vs. 11,260. The only major difference between the two systems is the polyol used; Niax 510 vs. Polymeg 650. Therefore, it is concluded that Niax 510 contributes considerably more to film thermal stability than does Polymeg 650.

A comparison of Runs F-9400 (Niax 510, H_{12}MDI, Quadrol 10 eq. %) and 1099446 (Niax 510, H_{12}MDI, THEIC, 10 eq. %) shows that the percent retention of tensile strength of the two systems are nearly equal, 67.7% vs. 69.6%. Both have the same equivalents percent of crosslinking agent but the eq. wt./cl. for 1099446 is 50% greater than F-9400, (15,600 vs. 10,300). Since it has been shown that, as the equivalent

weight per crosslink increases, the thermal stability of the system decreases, it therefore must be concluded that THEIC contributed more thermal stability to the film than did Quadrol.

This data, (Table XVIII) also showed that the crosslink agent giving the greatest thermal stability was pentaerythritol at 20 eq. %, and that it performed equally well with either Polymeg 650 or the Niax polycaprolactones (see Runs F-9378, F-7652 and F-9377).

The various crosslinking agents can be ranked according to decreasing thermal stability: pentaerythritol > THEIC > Quadrol. However, even though pentaerythritol formulations (Table XVII - F-9377, 78, F-7652) showed excellent retention of physical properties after thermal aging, all the films were badly discolored. In addition no more than 20 equivalents percent of pentaerythritol could be used because of its limited solubility.

Castor Oil DB (functionality = 3) formulations (Table XVII, Run 1099437 and 1099464) gave films which showed good thermal stability but either discolored badly when heated for 24 hours at 300°F or did not have enough elongation to pass the -65°F Conical Mandrel bend.

Before a final selection of raw material could be made, an examination of the accelerated and outdoor weathering data was necessary. Accelerated weathering was run in the Q-U-V unit. The Q-U-V test was run for a total of 900 hours which consisted of continuous repeating cycles of 8 hours under UV light at 145°F, followed by a 4 hour condensation cycle at 120°F. The films were examined after every 100 hours. Additional films were placed for outdoor exposure (45° South) at Olin here in New Haven, Connecticut, and these were examined at one week intervals.

The first film to fail in the Q-U-V unit after 195 hours was the Control (Table XIX, Run 1099406) based on H_{12}MDI and Polymeg 650.

Film (F-7643), based on the THEIC/H_{12}MDI prepolymer reacted with Polymeg 650, also failed at 195 hours. A film, 1099405, containing H_{12}MDI, Quadrol, and Niax 540 became soft and mushy after 724 hours. But systems 1099427A and 1099428-2 based upon H_{12}MDI/THEIC and polycaprolactone were hard, glossy, clear, and colorless after 900 hours. This, then, suggests by inference that Quadrol was the weak link in system 1099405 and that Polymeg 650 was the weak link in the other two systems. All films containing castor oil polyols as one component of their formulation showed some yellowing after 900 hours but otherwise had a good appearance.

The first film to fail under outdoor weathering was again the Control (Table XIX, 1099406) which contained Polymeg 650. It failed after only 6 weeks exposure. Sample 1099405 (Table XIX) became soft and tacky after 8 weeks; it contained H_{12}MDI, Niax 540, and Quadrol. Only the films containing both THEIC and Niax polycaprolactones in their formulations (1099427A, 1099428-2) performed well and showed no discoloration during either the outdoor or accelerated weathering tests. To sum up, the data on the physical properties, degree of discoloration, thermal stability, and weathering indicated the Niax polycaprolactones (510 and 520) be selected as the polyols, and THEIC as the crosslinking agent for development of a candidate system with the already selected H_{12}MDI isocyanate.

D. Potential Candidate Systems

THEIC and the Niax polycaprolactones (510 and 520) were formulated in various combinations with H_{12}MDI and reacted in various solvent systems to form prepolymers which were cured into films and tested for physical properties before and after thermal aging (see Table XVII). This and previous data suggested the following as potential candidate

formulations which most nearly match the requirements of this contract:

	Type	Isocyanate	Polyol	Crosslinking Agent
1.	One-Part Moisture-Cure	H_{12}MDI	Niax 520 (70 eq. %)	THEIC (30 eq. %)
2.	One-Part Moisture-Cure	H_{12}MDI	Niax 520 (60 eq. %)	THEIC (40 eq. %)
3.	Two-Part	H_{12}MDI - THEIC Prepolymer	Niax 540	---

These formulations were prepared in a 60% toluene and 40% p-dioxane by weight solvent system. Duplicates of 2 and 3 were prepared using a Rule 66 solvent system of 60% diethylene glycol dimetnyl ether [D_2M] and 40% p-dioxane by weight. For complete formulations see Table XX, "Potential Candidate Systems."

The one-part systems were pigmented with Rutile titanium dioxide at 15 wt. %, based on the resin solids, by ball milling. The polyol portion of the two-part systems was pigmented (Rutile TiO_2) so that, when reacted with the isocyanate containing portion, the overall pigment level in the cured film would be 15% by weight based on the resin solids.

The pigmented systems were then catalyzed (D-22) and two coats sprayed at one hour intervals on two sets of aluminum Q-panels (Type AA Chromic anodized MIL-A-8625); one set was unprimed and one set was primed with MIL-P-23377B. Coatings were allowed to moisture cure at 75°F and 50% relative humidity.

Two commercially available general purpose polyurethane aircraft coatings were selected as controls and for the purposes of this report designated "A" and "B". The manufacturer of Control A recommended the use of their own primer which we referred to as Control A primer.

Both control systems were prepared and sprayed according to the instructions supplied by the manufacturers. All films were then cured at 75°F and 50% RH and after 72 hours tested for physical properties and performance, which are discussed below under their separate headings.

1. Tensile and Elongation

Pigmented free films, both primed (MIL-P-23377B) and unprimed, were used to obtain values for tensile strength and percent elongation. These properties were measured before and after thermal treatment (48 hours at 300°F) at ambient conditions, and again at -65°F. Tensile values for the untreated systems at ambient conditions were 3414 psi and 3995 psi for unprimed Controls A and B respectively. Under these same conditions the tensile values for the potential candidate systems ranged from a low of 4363 psi (F-7662) to a high of 6104 psi (F-7664) (see Table XXI).

After thermal treatment, measurements at 25°C showed the commercial controls retained all of their tensile strength while the potential candidate systems showed minor loss of strength (10% maximum) (see Table XXII).

Elongation values at 25°C for unprimed films ranged from 110% (F-7661) to 170% (F-7660)(see Table XXI) for the moisture cured system and 20% to 30% for the two-part systems. No loss of elongation resulted from thermal treatment.

Elongation values for the commercial controls, at 25°C did not exceed 30%. Control A lost all of its elongation after thermal treatment while Control B showed no loss.

When tensile strengths were measured at -65°F only F-7661 showed a major loss of tensile strength after thermal aging. Sample F-7660 lost only 3.3% unprimed, while Control B showed no loss at all.

Elongation values at -65°F were very low, all running below 10%.

Thermal aging did not affect the elongation values obtained. System F-7660's elongation of 7.6% was the highest value obtained at -65°F. At rupture the commercial controls shattered into many small splinters while our potential candidate systems merely gave a single clean break.

2. Energy of Rupture

Energy of rupture is a measure of the work performed during elongation of a free film. It is calculated from curves obtained during tensile measurements. A summary of the data obtained can be found in Table XXIII.

F-7660 had the best value of 5275 in-lbs/in^3 for the moisture cured systems while F-7664 (two-part system) showed 8,983 in-lbs/in^3. The commercial Controls A & B had 654 and 825 in-lbs/in^3 respectively.

3. 1/8" Conical Mandrel at -65°F

All systems were subjected to the 1/8" Conical Mandrel test both before and after thermal aging. The commercial controls did not pass the Conical Mandrel at -65°F under any conditions. Only F-7663, of the potential candidates, failed for untreated films both primed and unprimed. All of the unprimed potential candidates passed after thermal treatment while the primed ones failed. The degree of failure after thermal aging for the potential candidate systems was of a very minor nature when compared to the controls. For example, F-7660 showed only a tiny flake missing at the panel tip (1/8" end), approximately 0.04" x 0.02". For a summary of results see Table XXIV.

4. Reverse Impact Test At -65°F

Two in-lbs. was the best value obtained for the Controls (A & B) when the Reverse Impact Test was run at -65°F. Whether the films were primed or unprimed made little difference. Two in-lbs. is the lowest scale marking on the Reverse Impact Tester. The potential candidate

systems showed excellent Reverse Impact values ranging from 60 to 160 in-lbs. for the unprimed films. Priming reduced these values to 16-60 in-lbs., still considerably better than the commercial controls (see Table XXIII).

The two-part systems (F-7663, F-7664) are less flexible and had impact values lower than the one-part systems (3-30 unprimed and 2-12 primed) but were still slightly better than the controls.

5. Shore A Hardness

Shore A Hardness was measured on both primed (MIL-P-23377B) and unprimed films. Even though the potential candidate systems exhibited outstanding flexibility, they had Shore A Hardnesses equal to the commercial controls. They ranged from a low of 91 for System F-7662 to a high of 96 for System F-7660 (see Table XXIII).

6. Wet Tape Adhesion Test

All film samples (both controls and candidate) passed the Wet Tape Adhesion Test, except Systems F-7662 and F-7663. These two systems were formulated using a solvent system of 40% p-dioxane and 60% diethylene glycol dimethyl ether (D_2M). Failure may have resulted because some D_2M remained in the cured film and acted as a conductor for moisture penetration to the substrate resulting in an adhesion loss between the film and the substrate. For a summary of results see Table XXIII. D_2M appears to have a detrimental effect upon the films, and thus, it was not considered for use in a Rule 66 system.

7. Abrasion Resistance

Abrasion resistance was measured using an S.S. White Model F Industrial Airbrasive Unit. System F-7660 showed the greatest abrasion resistance with values of 9.6 sec./mil unprimed and 24.8 sec./mil primed. The Control A had values of 1.3 sec./mil and 2.5 sec./mil for its unprimed and primed systems. The best value obtained for

Control B was 4.7 sec./mil with a primed system. For a complete summary of data see Table XXV.

8. Solvent Resistance

The solvents JP-4, MIL-L-7808, Aviation gas and Reference Fuel B had no adverse effect upon any of the films, the "Controls" or the potential candidate systems. Neither primed nor unprimed systems were attacked.

9. Thermal Aging - 48 Hours @ 300°F

All samples showed slight discoloration after aging 48 hours at 300°F, going from a white to a beige-cream-white color. Control A applied over its own primer showed the least discoloration. Control B showed about the same degree of discoloration as F-7661, F-7662 and F-7663. Film F-7660 showed slightly more discoloration than F-7661, probably because of its lower crosslink density which also contributes to its greater flexibility.

Color Eye values measured before and after thermal aging (Table XXVI) indicate that all systems suffered a loss of brightness (y value) under these conditions with the controls performing slightly better than the potential candidate systems.

E. Selection of a Candidate System

On the basis of the previous tests System F-7660 (70 eq..% Niax 520, 30 eq.·.% THEIC) was selected as the candidate system. It showed good low temperatu lexibility in combination with good thermal stability. At -65°F it passed the 1/8" Conical Mandrel bend and had Reverse Impact values as high as 160 in-lbs. It has adequate tensile strength, 4376 psi, and good thermal stability, losing no elongation and only 10% of its tensile strength after 48 hours at 300°F. After accelerated weathering, 500 hours in the Q-U-V unit, it lost only 33% of its 60" specular gloss, no elongation and only 5% of its

tensile strength (4100 psi before and 3900 psi after). It is non-yellowing, showing no discoloration after 900 hours exposure in the Q-U-V unit. A complete list of test results follows:

Tensile Strength	4400 psi
Elongation	170%
Reverse Impact @ -65°F	160 in-lbs.; 60 in-lbs.*
Abrasion Resistance	24.6 sec./mil*; 10 sec./mil
1/8" Conical Mandrel @ -65°F	Passed
Wet Tape Adhesion Test	Passed
Accelerated Weathering	900 hours in Q-U-V Unit with no discoloration
Solvent Resistance	No effect caused by standard aircraft fluids and solvents
Thermal Resistance 48 Hours @ 300°F	Retained better than 90% of its original tensile strength and all of its elongation without excessive discoloration

*Over MIL-P-23377B

The formulation of F-7660 can be found in Table XX. F-7660 performed as well as, or better than, the controls in all tests except thermal stability. A direct comparison between Control A and F-7660 candidate system appears below:

	Control A	F-7660 Candidate
Pot Life	~4 hours	> 24 hours
Dry Time	< 1 hour	2.5 - 3.5 hours
Respray Time	1 hour	1 hour
Tack Free	~1 hour	2 - 3 hours
300°F Stability	No visible change after 48 hours	Very slight discoloration
Brightness	99	94
60° Specular Gloss	95	95
Abrasion Resistance	1.1 sec./mil	24.8 sec./mil
1/8" Conical Mandrel @ -65°F	Failed	Passed

Reverse Impact @ -65°F	< 2 in-lbs	60 in-lbs.
Elongation	15%	170%
Tensile Strength	3300	4400
Elongation(1)	0	200
Tensile Strength(1)	5600	4000

(1)After 48 hours @ 300°F

F. Rule 66 Solvent Systems

With the selection of a candidate system, it was now possible to search for a Rule 66 conforming solvent system. The prime problem was the limited solubility of THEIC. THEIC is soluble in water and alcohols but insoluble in all of the hydrocarbons solvents normally used in urethane coating formulations. In addition, prepolymers of THEIC show only very limited solubility in these same urethane solvents. Para-dioxane and tetrahydrofuran were the only solvents we found in which the urethane forming reaction would take place, (see Table XXVII). But, because of their high volatility they did not afford good coatings when sprayed. Various solvent combinations were examined. A solution of 60% toluene and 40% p-dioxane by weight gave good results but was not a Rule 66 solvent system.

Development of a Rule 66 conforming solvent system was based upon a concept described in a release by Eastman (Kingsport, Tenn.) entitled "A Three Dimensional Approach To Solubility", which contends that the solubility of a polymer in a solvent depends upon several intermolecular forces: solubility parameter, δ (a measure of internal pressure between molecules), dipole moment, μ (the electrostatic imbalance of a molecule), and hydrogen bonding γ (an attraction force between molecules). The article states that the value of the solubility parameter for a solvent mixture is volume-wise proportional to the solubility parameters

of the mixtures' individual components and that this relationship can also be extended to values for dipole moment and hydrogen bonding. This is a convenient tool for developing solvent mixtures with specific values (δ,μ,γ) in order to insure proper solvency.

As a starting point we know THEIC and its prepolymer are soluble in p-dioxane ($\delta = 9.9, \gamma = 9.7, \mu = 0.4$) and are insoluble in toluene ($\delta = 8.9, \gamma = 4.5, \mu = 0.4$) but are soluble in a 60/40 mixture of toluene/dioxane ($\delta = 9.3, \gamma = 7.0, \mu = 0.4$) . Therefore, the values being sought for our solvent mixture should lie between those of p-dioxane and toluene. By averaging the values of selected Rule 66 conforming solvents, several solvent combinations were selected (see Table XXVIII).

The candidate prepolymer reaction was carried out in these Rule 66 conforming solvent systems, a list of which can be found in Table XXIX. The resulting prepolymer solutions were catalyzed, sprayed, and cured into films. After 72 hours the films were measured for tensile strength and elongation, both before and after thermal treatment (48 hours at 300°F). Only Systems 1099488 and 491 showed good spray characteristics coupled with physical property retention after thermal treatment. They lost only 3% and 15% respectively of their tensile strength and no elongation after thermal aging. The solvent mixture for these runs consisted of 40% 1,4-dioxane, 20% butyl acetate, 20% cyclohexane, and 20% cellosolve acetate. This solvent mixture will be considered as a part of the candidate system. A summary of the tensile data obtained for the other solvent systems can be found in Table XXIX.

G. Variation in NCO:OH Ratio

A formulation was set up using an NCO:OH ratio of 1.5:1 with THEIC at 30 eq. % and Niax 510 at 70 eq. %. When this system was cured into films and tested it was found to have very poor retention of properties

after thermal aging, retaining less than 50% of its original tensile strength (see Table XXX). The NCO:OH ratio of the candidate system was allowed to remain at 2:1.

H. Conclusions

Polyurethanes make acceptable coatings for Naval aircraft. They possess excellent chemical resistance, abrasive resistance, flexibility and strength. Current primers cause some physical property loss for the composite urethane system but, polyurethanes still offer many advantages over current systems.

The candidate system (H_{12}MDI/Niax 520/THEIC @ 30 eq. %) developed under this contract results in an all purpose, non-yellowing, aircraft coating having excellent hardness and low temperature (-65°F) flexibility as well as good resistance to high temperatures (48 hours @ 300°F). This combination of properties is regarded as unique and not possessed by current commercial systems.

Accelerated weathering data indicate that this system should weather well on outdoor exposure. Actual outdoor weathering data needs to be obtained to substantiate this claim.

III. EXPERIMENTAL

A. Preparation of Prepolymers

In general, all prepolymers were prepared in the following manner: Into a dry three-neck 500 ml. flask, equipped with stirrer, condenser, drying tube and nitrogen purge, were added the polyol, 4,4-methylene bis(cyclohexylisocyanate), crosslink agent, and MIL Thinner (45% toluene, 45% MIBK, 5% xylene and 5% cellosolve acetate). The reaction mixture was then heated with stirring to 80°C overnight. The percent NCO was monitored to determine completion of the reaction. At the end of the reaction, only the excess isocyanate added, over and above

the stoichiometry, should remain free. The solvents used in the reactions were dried over Linde molecular sieves (type 5A, 1/8" pellets) for 48 hours. The Polymeg 650 was dried by heating to 90°C under vacuum (0.5 mm Hg) in a rotary film evaporator for six hours. The glassware was oven dried before use.

B. Preparation of a One-Shot System

A formulation for a typical one-shot system is as follows:

10.3 g. H_{12}MDI 0.0763 equivalents

0.31 g. trimethylolpropane 6.94×10^{-3} equivalents

20.75 g. Polymeg 650 0.0625 equivalents

38.0 g. MIL Thinner

0.31 g. Dibutyl tin dilaurate

The 4,4-methylene bis(cyclohexylisocyanate) and half of the solvent are prepared as one solution. The Polymeg 650, trimethylolpropane, dibutyl tin dilaurate and the rest of the solvent are prepared as a second solution. Both solutions are mixed thoroughly. The polyol solution is then poured into the isocyanate solution and mixed again. A small exotherm occurs to about 41°C. After the exotherm has subsided films are prepared.

C. Coating Application On Aluminum Panels

Coatings are applied on 3" x 6" x .020" either alodine (MIL-C-5541) or anodized (MIL-A-8625) aluminum panels. Before coating, the panels are cleaned of oil and grease by washing with trichloroethylene. The coatings are then applied to the panels with a 6 mil. doctor blade.

D. Preparation of Free Film

Bright coke tin panels 6" x 12" x 20 mils are cleaned with trichloroethylene and films applied with a doctor blade. After curing the free films are removed by amalgamation with mercury.

E. Measurement of Tensile Strength, Modulus and Elongation

Free films were prepared as described above and cut into 1/4" neck x 3" dumbbells as described in ASTM-D-638-641. Along the neck of the dumbbell were inscribed two parallel lines one inch apart. The film thickness was measured to the nearest .0002". Tensile modulus, elongation and ultimate tensile were tested on an Instron Tester (Model TM) at 5"/min. crosshead speed. Elongation was determined by measuring the length of separation between the parallel lines at break. Force in PSI at break was also recorded. The chart speed was run at 2"/min. on the recorder. Tensile modulus was determined by recording the pounds of force (PSI) at 100%, 200% and 300% elongation. Both the tensile and tensile modulus are calculated according to the formula.

$$\text{Tensile or tensile modulus} = \frac{\text{lbs. of force}}{\text{initial cross sectional area}}$$

Where the initial cross sectional area = thickness x width of dumbbell neck before testing.

F. Energy of Rupture

"The energy of rupture is a measure of the toughness of a material".[6] It is determined from the load deflection curves obtained with the Instron Tester when measuring tensile strength (as above). Energy of rupture is the total work required to break a unit volume of material. The energy of rupture can be calculated by measuring the area under the load deflection curve with a planimeter. The working equation[2,3] is as follows:

$$Er = \text{Area under curve (sq.in.)} \times \frac{\text{Tensile at Break PSI}}{\text{Ordinate at Break (in.)}} \times \frac{\text{Elongation at Break (in./in.)}}{\text{Abscissa at Break}}$$

G. 1/8 Inch Conical Mandrel - Low Temperature Flexibility Test

The coated aluminum panels and the Conical Mandrel were cooled to -65°F by placing them in a well insulated box containing a small fan

and a large amount of dry ice. The samples were allowed to remain in the box until the internal temperature was stabilized at -65°F for one hour.

The box has an opening at the top so that the cold air is retained inside while conducting the test; a side port would allow the cold air to flow out. The Conical Mandrel Test was performed as follows: with the operating lever of the Conical Mandrel in a horizontal position, the test specimen was placed between the mandrel and the draw-bar with the test film kept towards the draw-bar. Next the lever was moved at a uniform rate so as to bend the test specimen approximately 180° in about 15 seconds. The bent area is examined under an 8-power scope for cracks at the small 1/8" end. A sample is deemed to pass if no cracks are visible. If any crack is visible (no matter how small) the sample has failed.

H. Hardness

Hardness was measured by a Shore A Hardness Tester.

I. Q-U-V Accelerated Weathering Test

The coated panels were cured at ambient temperature for at least 72 hours and then placed in the Q-U-V unit. The panels were exposed for a total time up to 900 hours over an open trough of water with continuous repeating cycles of 8 hours. UV light at 145°F followed by 4 hours of darkness during a condensation cycle at 120°F. After every 100 hours the panels were inspected for changes in physical appearance such as crazing, loss of gloss or yellowing.

J. Reverse Impact

A Gardner Reverse Impact Tester was used in this test. The sample and impact tester were placed in a cold box and chilled to -65°F. After one hour at -65°F, the test panel (3 x 6 coated aluminum panel) was placed over the 5/8" hole at the base plate of the impact tester.

The four pound weight was raised to a given graduation on the tester scale and released smashing into the aluminum panel. The film is then examined for cracking or flaking. If none occurred, the sample is said to have passed at that graduation setting. The setting is increased until the sample fails.

K. Wet Tape Adhesion

After curing for at least 72 hours, the samples were subjected to the wet tape adhesion test according to Federal Standard 141, Method 6301.

L. High Temperature Aging

After curing the coated samples at 75°F and 50% RH for 72 hours they were placed in a circulating hot air oven at 300°F for 48 hours. The samples were observed for evaporation, flow and discoloration.

M. Solvent Resistance

After curing the coated samples for at least 72 hours, they were placed in JP-4 and MIL-L-7808, Aviation Gas, and Reference Fuel B for one hour at room temperature, removed and inspected for lift-off, softening, tackiness, or discoloration. Other samples were placed in distilled water for 24 hours at room temperature then removed and inspected for lift-off, bubbles, change of color, etc. If no change was detected from either test, the sample was said to show satisfactory solvent resistance.

N. Measurement of Color and Brightness

Measurement of color and brightness was carried out by use of a Model D-1 Color Eye (Instrument Development Laboratories, Inc.) following the instructions of the manufacturer as outlined in Instruction Manual No. 4000.

O. Abrasion Resistance

Panels were coated with pigmented vehicles and cured for at least 72 hours. Abrasion resistance was determined with the use of an S.S.

White Industrial Airbrasive Model F unit. The abrasive powder used was Dolomite No. 2 (calcium, magnesium oxide) under a feed pressure of (50) PSIG @ 80 volts setting, with a nozzle height of 3/8". The powder is allowed to impinge upon the film until a pin hole appears through the coating signifying the end point. The time is recorded, the film thickness is measured on an unexposed area adjacent to the holes and the resistance is calculated in sec./mil.

P. Measurement of 60° Specular Gloss

Measurement of 60° Specular Gloss was carried out using a Gardner Automatic Photometric Unit according to the manufacturer's instructions for use of 60° Specular Gloss head (AUX-3).

IV. REFERENCES

1. J. E. Pregler Final Report; Development of a General Purpose Aircraft Coating; Contract N00019-69-C-0093; Olin Corp.

2. Union Carbide, Niax Caprolactone Polyols for Urethane Elastomers, F-42020.

3. Allied Chemical Company, THEIC; tris(2-hydroxyethyl) isocyanurate 318-60.

4. The Quaker Oats Company Technical Bulletin Number 150-B.

5. Naftone Product Bulletin, Desmodur N-Desmophen.

6. M. L. Wilson; Tensile Studies Made On Navy Aircraft, Organic Protective Coatings Report No. NADC-MA-6877, December 12, 1968.

V. EXPERIMENTAL DATA

TABLES I - XXX

FIGURE I

TABLE I

EFFECT OF CROSSLINKING THRU POLYOL

POLYMER FORMULATION

ONE SHOT SYSTEMS

REFERENCE NUMBER	F-9364	F-9365	F-9369	F-9370	F-9374	F-9375	F-9379	F-9380	F-9384
$H_{12}MDI$									
Grams	10.0	10.0	10.0	10.0	10.0	10	10	10	10
Equivalents	0.0763	0.0763	0.0763	0.0763	0.0763	0.0763	0.0763	0.0763	0.0763
Polymeg 650									
Grams	20.48	18.21	20.48	18.21	20.48	18.21	20.48	18.21	23.2
Equivalents	0.0625	0.0555	0.0625	0.0555	0.0625	0.0555	0.0625	0.0555	0.0694
Crosslinking Agent	Poly-G 630	Poly-G 630	Quadrol	Quadrol	Trimethylol-propane	Trimethylol-propane	Pentaery-thritol	Pentaery-thritol	—
Functionality	3	3	4	4	3	3	4	4	—
Grams	1.18	2.73	0.51	1.01	0.31	0.62	0.24	0.47	—
Equivalents	0.00694	0.0139	0.00694	0.0139	0.00694	0.0139	0.00694	0.0139	—
Solvent	MIL Thin	MIL Thin	MIL Thin	MIL Thin	MIL Thin	MIL Thin	MIL Thin	MIL Thin	MIL Thin
NCO:OH Ratio	1.1:1	1.1:1	1.1:1	1.1:1	1.1:1	1.1:1	1.1:1	1.1:1	1.1:1
Concentration of Solids, %	45	45	45	45	45	45	45	45	45

TABLE II

EFFECT OF CROSSLINKING THRU POLYOL

PHYSICAL PROPERTIES

ONE-SHOT — NCO:OH RATIO 1.1:1 — ISOCYANATE H_{12}MDI

REFERENCE NUMBER	EQUIVALENTS PERCENT		FILM THICKNESS MILS.	TENSILE MODULUS; PSI			TENSILE AT BREAK PSI	ELONGATION %	ENERGY OF RUPTURE IN-LBS/IN^3	SHORE A HARDNESS	CONICAL MANDREL -65°F
	DIOL	TRIOL		100%	200%	300%					
F-9364	90 Polymeg	10 Poly-G 630	2.7	175	175	190	510$^+$	700$^+$	—	75	Passed
24 Hrs. @ 300°F				SAMPLE MELTED							
F-9365	80 Polymeg	20 Poly-G 630	2.5	192	234	285	869	510	—	66	Passed
24 Hrs. @ 300°F				SAMPLE MELTED							
F-9369	90 Polymeg	10 Quadrol	2.3	191	214	244	915	600	1486	74	Passed
24 Hrs. @ 300°F				SAMPLE MELTED							
F-9370	80 Polymeg	20 Quadrol	2.7	295	445	780	1296	350	1738	69	Passed
24 Hrs. @ 300°F				SAMPLE MELTED							
F-9374	90 Polymeg	10 Trimethylol-propane	2.1	198	231	263	789	530	1629	73	Passed
24 Hrs. @ 300°F				SAMPLE MELTED							
F-9375	80 Polymeg	20 Trimethylol-propane	2.0	273	369	639	1610	370	1908	74	Passed
24 Hrs. @ 300°F				SAMPLE MELTED							
F-9379	90 Polymeg	10 Pentaerythritol		PENTAERYTHRITOL DID NOT DISSOLVE, COULD NOT FORM FILM							—
F-9380	80 Polymeg	20 Pentaerythritol		PENTAERYTHRITOL DID NOT DISSOLVE, COULD NOT FORM FILM							—
24 Hrs. @ 300°F				SAMPLE MELTED							
F-9384	100 Polymeg	—	1.7	515	643	825	4268	535	7430	65	Passed
24 Hrs. @ 300°F				SAMPLE MELTED							

TABLE III

EFFECT OF CROSSLINKING THRU ISOCYANATE BACKBONE

POLYMER FORMULATION

ONE-SHOT SYSTEMS

REFERENCE NUMBER	F-9387	F-9388	F-9392	F-9393	F-9384
H_{12}MDI					
Grams	9.27	8.24	9.3	8.24	10
Equivalents	6.87x10 -2	6.1x10 -2	6.87x10 -2	6.1x10 -2	7.63x10 -2
Polymeg 650					
Grams	23.18	23.18	23.2	23.2	23.2
Equivalents	6.94x10 -2	6.94x10 -2	6.94x10 -2	6.94x10 -2	6.94x10 -2
Crosslinking Agent	Desmodur N	Desmodur N	Mondur MC	Mondur MC	—
Functionality	3	3	3	3	—
Grams	2.14	4.27	2.83	5.7	—
Equivalents	7.63x10 -3	1.53x10 -2	7.63x10 -3	1.53x10 -2	—
Solvent	MIL Thin	MIL Thin	MIL Thin	MIL Thin	MIL Thin
NCO:OH Ratio	1.1:1	1.1:1	1.1:1	1.1:1	1.1:1
Concentration of Solids, %	45	45	45	45	45

TABLE IV

EFFECT OF CROSSLINKING THRU ISOCYANATE BACKBONE

—— PHYSICAL PROPERTIES ——

ONE-SHOT – NCO:OH RATIO 1.1:1 — POLYOL = POLYMEG 650

REFERENCE NUMBER	EQUIVALENTS PERCENT		FILM THICKNESS MILS.	TENSILE MODULUS; PSI			TENSILE AT BREAK PSI	ELONGATION %	ENERGY OF RUPTURE in-lbs/in³	SHORE A HARDNESS	CONICAL MANDREL -65°F
	DIISOCYANATE	TRIISOCYANATE		100%	200%	300%					
F-9387	90 H_{12}MDI	10 Desmodur N	1.7	315	420	615	2033	420	2840	81	Passed
24 Hrs. @ 300°F				SAMPLE MELTED							
F-9388	80 H_{12}MDI	20 Desmodur N	2.1	322	593	—	877	245	1269	85	Passed
24 Hrs. @ 300°F				SAMPLE MELTED							
F-9392	90 H_{12}MDI	10 Mondur MC	1.8	319	415	583	3040	455	3265	57	Passed
24 Hrs. @ 300°F				SAMPLE MELTED							
F-9393	80 H_{12}MDI	20 Mondur MC	2.5	356	767	—	1560	265	1644	74	Passed
24 Hrs. @ 300°F				SAMPLE MELTED							
F-9384	100 H_{12}MDI	—	1.7	515	643	825	4268+	535+	7430+	65	Passed

TABLE V

EFFECT OF CROSSLINKING THRU POLYOL BACKBONE

PREPOLYMER PREPARATION

MOISTURE CURED SYSTEMS

REFERENCE NUMBER	F-9360	F-9361	F-9366	F-9367	F-9372	F-9373	F-9377	F-9378	F-9382	F-9383	1099406
$H_{12}MDI$											
Grams	50	50	50	50	51.5	51.5	51.5	51.5	51.5	51.5	51.5
Equivalents	0.382	0.382	0.382	0.382	0.382	0.382	0.382	0.382	0.382	0.382	0.382
Polymeg 650											
Grams	56.4	50.1	56.4	50.1	57.4	51.0	57.4	51.0	57.4	51.0	63.8
Equivalents	0.172	0.153	0.172	0.153	0.172	0.153	0.172	0.153	0.172	0.153	0.191
Crosslinking Polyol	Poly-G 630	Poly-G 630	Quadrol	Quadrol	Trimethylol-propane	Trimethylol-propane	Pentaery-thritol	Pentaery-thritol	1,2,6-Hexanetriol	1,2,6-Hexanetriol	—
Functionality	3	3	4	4	3	3	4	4	3	3	—
Grams	3.93	7.3	1.39	2.79	0.85	1.7	0.65	1.3	0.85	1.7	—
Equivalents	0.019	0.0382	0.0191	0.0382	0.0191	0.0382	0.0191	0.0382	0.0191	0.0382	—
Solvent	MIL Thin.	MIL Thin	MIL Thin	MIL Thin	MIL Thin	MIL Thin	MIL Thin	MIL Thin	MIL Thin	MIL Thin	MIL Thin
NCO:OH Ratio	2:1	2:1	2:1	2:1	2:1	2:1	2:1	2:1	2:1	2:1	2:1
% Theory NCO	3.67	3 8	3.72	3.9	3.6	3.85	3.6	3.9	3.5	3.8	3.5
% NCO Found	3.34	3.9	3.60	3.9	3.2	3.8	3.5	4.0	3.6	3.9	3.5
% Concentration of Solids	50	50	50	50	50	50	50	50	50	50	50

TABLE VI

EFFECT OF CROSSLINKING THRU POLYOL

—— PHYSICAL PROPERTIES ——

NCO:OH RATIO = 2: - MOISTURE CURED - ISOCYANATE IS H_{12}MDI

REFERENCE NUMBER	EQUIVALENTS PERCENT		FILM THICKNESS MILS.	TENSILE MODULUS; PSI			TENSILE AT BREAK PSI	ELONGATION %	ENERGY OF RUPTURE IN-LBS/IN^3	SHORE A HARDNESS	CONICAL MANDREL -65°F
	DIOL *	TRIOL		100%	200%	300%					
F-9360	90 Polymeg	10 Poly-G 630	7.9	2130	3323	—	5913	285	8350	90	Passed
24 Hrs. @ 300°F			7.1	1075	—	—	1614	165			
F-9361	80 Polymeg	20 Poly-G 630	10.7	2835	4772	—	5568	220	7024	95	Passed
24 Hrs. @ 300°F			3.8	1337	—	—	2075	170			
F-9366	90 Polymeg	10 Quadrol	9.7	2001	3370	—	5642	270	15,622	87	Passed
24 Hrs. @ 300°F			6.3	800	1001	—	1086	230			
F-9367	80 Polymeg	20 Quadrol	2.2	3040	—	—	5400	200	6900	88	Failed
24 Hrs. @ 300°F			2.2	1604	—	—	2048	175			
F-9372	90 Polymeg	10 Trimethylol-propane	2.2	2126	3312	—	5300	290	8653	89	Passed
24 Hrs. @ 300°F			2.8	768	—	—	1027	140			
F-9373	80 Polymeg	20 Trimethylol-propane	1.9	3294	4351	—	5047	200	6657	93	Passed
24 Hrs. @ 300°F			2.5	2644	—	—	3024	125			
F-9377	90 Polymeg	10 Pentaery-thritol	3.0	2323	3543	—	4803	255	7452	84	Passed
24 Hrs. @ 300°F			0.8	1750	—	—	2202	150			
F-9378	80 Polymeg	20 Pentaery-thritol	3.2	2411	—	—	3016	150	2450	92	Passed
24 Hrs. @ 300°F			2.0	3109	—	—	3109	100			
F-9382	90 Polymeg	10 Hexanetriol	2.6	2251	3149	4885	5520	315	10,240	87	Passed
24 Hrs. @ 300°F			3.0	—	—	—	1534	90			
F-9383	80 Polymeg	20 Hexanetriol	2.7	2685	4326	—	5172	240	7030	90	Passed
24 Hrs. @ 300°F			4.3	1838	—	—	2831	160			
10-9406	100 Polymeg	—	3.0	1833	2610	4485	7521	395	13,220	82	Passed
24 Hrs. @ 300°F			3.1	1075	—	—	1208	130			

* Polymeg 650

TABLE VII

EFFECT OF CROSSLINKING THRU ISOCYANATE BACKBONE

PREPOLYMER PREPARATION

MOISTURE CURED

REFERENCE NUMBER	F-9389	F-9386	F-9390	F-9391	1099406
H_{12}MDI					
Grams	47.0	41.3	47.0	41.3	41.5
Equivalents	0.348	0.306	0.348	0.306	0.382
Polymeg 650					
Grams	63.8	63.8	63.8	63.8	63.8
Equivalents	0.191	0.191	0.191	0.191	0.191
Crosslinking Isocyanate	Desmodur N	Desmodur N	Mondur HC	Mondur HC	—
Functionality	3	3	3	3	—
Grams	10.7	21.4	12.9	28.2	—
Equivalents	0.0382	0.0764	0.0348	0.0764	—
Solvent	MIL Thin	MIL Thin	MIL Thin	MIL Thin	MIL Thin
NCO:OH Ratio	2:1	2:1	2:1	2:1	2:1
% Theory NCO	3.37	3.17	3.92	GELLED REACTION	3.5
% NCO Found	3.3	3.19	3.90		3.5
Concentration of Solids, %	50	50	50		50

TABLE VIII

EFFECT OF CROSSLINKING THRU ISOCYANATE BACKBONE

—— PHYSICAL PROPERTIES ——

NCO:OH RATIO 2:1 — MOISTURE CURED — POLYOL POLYMEG 650

REFERENCE NUMBER	EQUIVALENTS PERCENT		FILM THICKNESS MILS.	TENSILE MODULUS; PSI			TENSILE AT BREAK PSI	ELONGATION %	ENERGY OF RUPTURE in-lbs/in^3	SHORE A HARDNESS	CONICAL MANDREL -65°F
	DIISOCYANATE	TRIISOCYANATE		100%	200%	300%					
F-9385	90 H_{12}MDI	10 Desmodur N	1.8	2007	3970	—	4539	215	4667	82	Passed
24 Hrs. @ 300°F			2.1	1150			1560	130	—		—
F-9386	80 H_{12}MDI	20 Desmodur N	1.6	2168	—	—	4915	185	4507	89	Passed
24 Hrs. @ 300°F			2.6	1118	—	—	1902	155			
F-9390	90 H_{12}MDI	10 Mondur MC	2.3	2297	4398	—	4465	200	5267	85	Passed
24 Hrs. @ 300°F			2.4	1291	—	—	1639	190			
F-9391	80 H_{12}MDI	20 Mondur MC	S A M P L E G E L L E D D U R I N G P R E P A R A T I O N						—	—	—
1099406	100 H_{12}MDI	—	3.0	1833	2610	4485	7521	395	13,220	82	Passed
24 Hrs. @ 300°F			3.1	1075	—	—	1208	130			

TABLE IX

PREPOLYMER PREPARATIONS CONTAINING TRIS (2-Hydroxyethyl) ISOCYANURATE

FORMULATIONS

REFERENCE NUMBERS	1099411	1099415	1099416	1099418	1099423	1099425	1099426	F-7643	F-7643-1*	1099427A*
REACTANT A	H_{12}MDI	H_{12}MDI	H_{12}MDI	H_{12}MDI	H_{12}MDI	H_{12}MDI	H_{12}MDI	H_{12}MDI	Prepolymer 1099406 ①	Niax 510
Grams	51.5	51.6	51.6	51.6	51.6	51.6	51.6	51.6	15.0	2.8
Equivalents	0.382	0.382	0.382	0.382	0.382	0.382	0.382	0.382	0.0134	0.00997
REACTANT B	THEIC	THEIC	THEIC	THEIC	THEIC	THEIC	THEIC	THEIC	Prepolymer F-7643	Prepolymer F-7643
Grams	2.49	1.66	3.32	1.66	16.6	16.6	16.6	11.05	1.42	10.0
Equivalents	0.0285	0.0191	0.0382	0.0191	0.191	0.191	0.191	0.127	0.0028	0.01995
REACTANT C	Polymeg 650	Polymeg 650	Polymeg 650	Polymeg 650	--	--	--	--	--	--
Grams	57.4	63.8	50.8	57.4	--	--	--	--	--	--
Equivalents	0.1719	0.191	0.153	0.1719	--	--	--	--	--	--
Solvent	Tetrahydro-furan	MIL Thin. Dimethyl Sulfoxide	MIL Thin. Dimethyl Sulfoxide	Toluene	Tetrahydro-furan	Toluene	Tetrahydro-furan	Tetrahydro-furan	MIL Thin. THF	Tetrahydro-furan
NCO:OH Ratio	1.9:1	1.9:1	2:1	2:1	2:1	2:1	2:1	3:1	2.2:1	2:1
% Theory NCO	3.62	3.27	3.8	3.63	5.88	5.5	5.13	8.55	--	--
% NCO Found	THEIC Would Not Dissolve	3.23	3.4	3.70	5.70	Didn't React	5.02	8.4	--	--
% Concentration of Solids		50	50	50	50	47	50	50	50	50

* One Shot System

① 0.382 eq. H_{12}MDI and 0.191 eq. Polymeg 650

TABLE IX (CONTINUED)

PREPOLYMER PREPARATIONS CONTAINING TRIS (2-Hydroxyethyl) ISOCYANURATE

—— FORMULATIONS ——

REFERENCE NUMBERS	1099427B*	1099428-1*	1099428-2*	1099430*	1099431	1099432	1099433	1099435	1099436*	1099437*
REACTANT A	RD-18	Prepolymer 1099426	Prepolymer 1099426	Prepolymer 1099405 ①	H_{12}MDI	Prepolymer 1099431	Prepolymer 1099431	H_{12}MDI (DuPont)	Prepolymer 1099435	Prepolymer 1099435
Grams	2.8	10.0	10.0	10.0	103.4	77.7	62.0	50.0	10	10
Equivalents	0.00665	0.00975	0.00975	.0062	0.764	.096	0.0764	0.382	0.0127	0.0127
REACTANT B	Prepolymer F-7643	Poly-G 750P	Niax 540	Prepolymer 1099426	THEIC	H_{12}MDI	H_{12}MDI	THEIC	Pale 16	Castor Oil DB
Grams	10	1.83	3.04	4.5	33.2	51.6	40.0	16.6	5	2.62
Equivalents	0.01995	0.00488	.00488	.0043	0.382	0.382	0.3056	0.191	--	0.00846
REACTANT C	--	--	--	--	--	Niax 510	Niax 510	--	--	--
Grams	--	--	--	--	--	52.5	47.5	--	--	--
Equivalents	--	--	--	--	--	0.191	0.1719 (See Below)	--	--	--
Solvent	Tetrahydro-furan	Tetrahydro-furan	Tetrahydro-furan	Tetrahydro-furan MIL Thin.	Tetrahydro-furan	Toluene Dioxane	Toluene Dioxane	Dioxane	Dioxane	Dioxane
NCO:OH Ratio	2:1 ② (2.2:1) ③	2:1 ② (1.6:1) ③	2:1 ② (1.6:1) ③	1.44:1 ② (2.7:1) ③	2:1	2.5:1 ② (2:1) ③	2:1 ② (1.7:1) ③	2:1	--	1.5:1 ② (1.2:1) ③
% Theory NCO	--	--	--	Sample	5.13	3.88	2.7	5.54	--	--
% NCO Found	--	--	--	Gelled	5.19	3.88	2.8	5.34	--	--
% Concentration of Solids	50	50	50	50	50	47	Also Quadrol 1.39 g 0.0191 eq.	50	50	50

* One Shot Systems

① Prepolymer Contains: 0.382 eq. H_{12}MDI
0.019 eq. Quadrol
0.172 eq. Niax 540
@ 2:1 NCO:OH Ratio

② NCO:OH Ratio when the THEIC prepolymer is considered a triisocyanate

③ Overall NCO:OH Ratio

TABLE IX (CONTINUED)

PREPOLYMER PREPARATIONS CONTAINING TRIS (2-Hydroxyethyl) ISOCYANURATE

FORMULATIONS

REFERENCE NUMBERS	1099438*	1099439*	1099440	1099441*	1099442*	1099446
REACTANT A	Prepolymer 1099435	Prepolymer 1099435	TMDI	Prepolymer 1099405 ①	Prepolymer 1099405 ①	H_{12}MDI
Grams	10	10	40.16	10	10	54.3
Equivalents	0.0127	0.0127	0.382	0.00619	0.00619	0.382
REACTANT B	Polycin 58	Polycin 52	THEIC	Prepolymer 1099440	Prepolymer 1099435	THEIC
Grams	3.49	1.37	16.6	1.76	2.3	1.66
Equivalents	0.00846	.00846	0.191	0.00294	0.00294	0.0191
REACTANT C	--	--	--	--	--	Niax 510
Grams	--	--	--	--	--	48.22
Equivalents	--	--	--	--	--	.1719
Solvent	Dioxane	Dioxan	Dioxane	Dioxane MIL Thin.	Dioxane MIL Thin.	Toluene THF
NCO:OH Ratio	1.5:1	1.5:1	2:1	2.5:1 ② (2:1) ③	2.5:1 ② (2:1) ③	2:1
% Theory NCO	--	--	6.9	--	Sample Gelled	3.85
% NCO Found	--	--	6.8	--	--	4.02
% Concentration of Solids	50	50	50	--	--	50

* One Shot Systems

① Prepolymer Contains: 0.382 eq. H_{12}MDI
0.019 eq. Quadrol
0.172 eq. Niax 540
@ 2:1 NCO:OH Ratio

② NCO:OH Ratio when the THEIC prepolymer is considered a triisocyanate

③ Overall NCO:OH Ratio

TABLE X

PREPOLYMER PREPARATIONS CONTAINING TRIS (2-Hydroxyethyl) ISOCYANURATE

REFERENCE NUMBER	REACTANT A	REACTANT B	REACTANT C	FILM THICKNESS MILS.	TENSILE MODULUS, PSI 100%	200%	300%	TENSILE AT BREAK PSI	ELONGATION %	ENERGY OF RUPTURE in-lbs/in^3	SHORE A HARDNESS	CONICAL MANDREL -65°F	GARDNER COLOR STANDARDS
				——— PHYSICAL PROPERTIES ———									
1099411	H_{12}MDI	THEIC	Polymeg 650	REACTION DID NOT GO TO COMPLETION									
1099415	H_{12}MDI	THEIC	Polymeg 650	FILM WAS CLOUDY AND OPAQUE							--	Passed	--
1099416	H_{12}MDI	THEIC	Polymeg 650	3.4	3353	--	--	4706	180	5780	91	--	1
24 Hrs. @ 300°F				3.4	2341	--	--	2435	110	--	--	--	12
1099418	H_{12}MDI	THEIC	Polymeg 650	3.0	2693	4214	--	4662	210	6827	98	Passed	1
24 Hrs. @ 300°F				3.3	1911	--	--	2273	160	--	--	--	11
1099423	H_{12}MDI	THEIC	--	NOT TESTED									
1099425	H_{12}MDI	THEIC	--	DID NOT REACT									
1099426	H_{12}MDI	THEIC	--	NOT TESTED									
F-7643	H_{12}MDI	THEIC	--	TOO BRITTLE TO TEST							99	--	1
24 Hrs. @ 300°F				TOO BRITTLE TO TEST							--	--	1
F-7643-1	Prepolymer 1099406	Prepolymer F-7643	--	2.8	2899	4116	--	6150	290	10,513	82	Passed	1
24 Hrs. @ 300°F				2.7	1986	--	--	2330	130	--	--	--	11
1099427A	Niax 510	Prepolymer F-7643	--	2.6	--	--	--	7846	< 5	--		Failed	1
24 Hrs. @ 300°F				2.6	--	--	--	11,354	< 5	--	--	--	1

TABLE X (CONTINUED)

PREPOLYMER PREPARATIONS CONTAINING TRIS (2-Hydroxyethyl) ISOCYANURATE

REFERENCE NUMBER	REACTANT A	REACTANT B	REACTANT C	PHYSICAL PROPERTIES: FILM THICKNESS MILS.	TENSILE MODULUS, PSI 100%	200%	300%	TENSILE AT BREAK PSI	ELONGATION %	ENERGY OF RUPTURE in-lbs/in^3	SHORE A HARDNESS	CONICAL MANDREL -65°F	GARDNER COLOR STANDARDS
1099427B	RD-18	Prepolymer F-7643	--	TOO BRITTLE TO TEST								Failed	1
24 Hrs. @ 300°F				NOT TESTED								--	1
1099428-1	Prepolymer 1099426	Poly-G 750 P	--	2.4	--	--	--	5828	< 5	--	92	Failed	1 (Cloudy)
24 Hrs. @ 300°F				1.7	--	--	--	5817	< 5	--	--	--	11
1099428-2	Prepolymer 1099426	Niax 540	--	5.1	4863	--	--	5710	120	5250	94	Passed	1
24 Hrs. @ 300°F				3.5	4434	--	--	5486	130	5160	--	--	2
1099430	Prepolymer 1099405①	Prepolymer 1099426	--	NOT TESTED									
1099431	H_{12}MDI	THEIC	--	NOT TESTED									
1099432	Prepolymer 1099431	H_{12}MDI	Niax 510	2.7	--	--	--	5609	~10	7670	92	Crazed (Failed)	1
24 Hrs. @ 300°F				3.1	--	--	--	5790	~20	--	--	--	3
1099433*	Prepolymer 1099431	H_{12}MDI	Niax 510	2.1	--	--	--	4892	<10	--	92	Passed	1
24 Hrs. @ 300°F				2.5	--	--	--	4352	< 5	--	--	--	8
1099435	H_{12}MDI (DuPont)	THEIC	--	TOO BRITTLE TO TEST							91	Failed	1
24 Hrs. @ 300°F				NOT TESTED									1 (Cloudy)
1099436	Prepolymer 1099435	Pale 16	--	2.9	--	--	--	4665	40	1328	89	Failed	3
24 Hrs. @ 300°F				2.3	--	--	--	5165	10	--	--	--	13

* Contains Quadrol Also.

① .382 eq. H_{12}MDI, .0191 eq. Quadrol, and .172 eq. Niax 540

TABLE X (CONTINUED)

PREPOLYMER PREPARATIONS CONTAINING TRIS (2-Hydroxyethyl) ISOCYANURATE

REFERENCE NUMBER	REACTANT A	REACTANT B	REACTANT C	FILM THICKNESS MILS.	TENSILE MODULUS, PSI 100%	200%	300%	TENSILE AT BREAK PSI	ELONGATION %	ENERGY OF RUPTURE in-lbs/in^3	SHORE A HARDNESS	CONICAL MANDREL -65°F	GARDNER COLOR STANDARDS
1099437	Prepolymer 1099435	Castor Oil DB	--	2.7	--	--	--	7898	<5	--	91	Failed	1
24 Hrs. @ 300°F				2.4	--	--	--	7874	<5	--	--	--	2
1099438	Prepolymer 1099485	Polycin 58	--	2.3	--	--	--	6317	<5	476	93	Failed	3
24 Hrs. @ 300°F				2.5	--	--	--	7300	<5	--	--	--	11
1099439	Prepolymer 1099435	Polycin 52	--	3.1	--	--	--	5197	<5	--	90	Failed	1
24 Hrs. @ 300°F				1.6	--	--	--	11,700	<5	--	--	--	2
1099440	TMDI ①	THEIC	--	3.9	--	--	--	10,954	<5	--	94	Failed	1
24 Hrs. @ 300°F				TOO BRITTLE TO TEST									2
1099441 ②	Prepolymer 1099405 ③	Prepolymer 1099440	--	2.7	1329	2138	4754	6256	320	7857	80	Passed	1
24 Hrs. @ 300°F				2.5	528	821	1184	1435	340	2693	--	--	10
1099442 ②	Prepolymer 1099405	Prepolymer 1099435		SAMPLE GELLED									
1099446	H_{12}MDI	THEIC	Niax 510	2.4	4422	--	--	6616	170	7407	94	Passed	1
24 Hrs. @ 300°F				2.3	3078	--	--	4110	180	--	--	--	9

① 2,2,4 Trimethylhexanethylene diisocyanate

② Contains Quadrol

③ .382eq. H_{12}MDI, .0191 eq. Quadrol, and .172eq. Niax 540

TABLE XI

MISCELLANEOUS PREPOLYMER PREPARATIONS

FORMULATIONS

REFERENCE NUMBER	F-9396*	F-9398*	F-9399	1099413	1099414	1099422A*	1099422B*	1099444	1099445
Reactant A	H_{12}MDI	H_{12}MDI	H_{12}MDI	H_{12}MDI	Prepolymer 1099406 (1)	Prepolymer 1099406 (1)	Prepolymer 1099406 (1)	TMDI (2)	H_{12}MDI
Grams	16.0	2.67	18.6	23.63	105.66	25.0	25.0	40.16	50.0
Equivalents	0.119	0.0198	0.138	0.175	0.0942	0.0224	0.0224	0.382	0.382
Reactant B	Castor 1066	Polybutene 1000	Polybutene 1000	Mondur HC	Mondur HC	Mondur HC	Desmodur N	Quadrol	TMP (3)
Grams	6.97	14.53	50.0	16.24	16.24	3.8	2.9	13.94	8.54
Equivalents	0.033	0.02	0.0688	0.0438	0.0438	0.0102	0.0103	0.191	0.191
Reactant C	Polymeg 650	Desmodur N	--	Polymeg 650	Polymeg 650	--	--	--	--
Grams	8.8	0.62	--	34.07	4.85	--	--	--	--
Equivalents	0.027	0.022	--	0.102	0.0146	--	--	--	--
Solvent	MIL Thin.	Toluene	Toluene	MIL Thin.	MIL Thin.	MIL Thin.	MIL Thin.	Dioxane	Dioxane
NCO:OH Ratio	2:1	2.1:1	2:1	2.15:1	2.15:1	2.5:1	2.5:1	2:1	2:1
% Theory NCO	--	--	2.11	3.32	Reaction Gelled	Reaction Gelled	--	7.4	6.8
% NCO Found	--	--	1.96	Reaction Gelled			--	6.6	6.4
% Concentration of Solids	50	50	50	50			50	50	50

* One Shot System

(1) 0.382 eq. H_{12}MDI and 0.191 eq. Polymeg 650

(2) 2,2,4-Trimethylhexamethylene diisocyanate

(3) Trimethylolpropane

TABLE XII

MISCELLANEOUS PREPOLYMER PREPARATIONS

REFERENCE NUMBER	REACTANT A	REACTANT B	REACTANT C	FILM THICKNESS MILS.	TENSILE MODULUS, PSI 100%	200%	300%	TENSILE AT BREAK PSI	ELONGATION %	ENERGY OF RUPTURE in-lbs/in^3	SHORE A HARDNESS	CONICAL MANDREL -65°F	GARDNER COLOR STANDARD
F-9396	H_{12}MDI	Castor 1066	Polymeg 650	1.7	--	--	--	3909	35	--	--	Passed	1
24 Hrs. @ 300°F				1.5	--	--	--	4908	10	--	--	--	12
F-9398	H_{12}MDI	Polybutene 1000	Desmodur N	2.8	156	235	353	1228	530	2752	55	Passed	1
24 Hrs. @ 300°F				SAMPLE MELTED									
F-9399	H_{12}MDI	Polybutene 1000	--	2.7	1136	1496	2022	2773	420	6730	85	Failed	1
24 Hrs. @ 300°F				3.2	--	--	--	432	75	--	--	--	3
1099413	H_{12}MDI	Mondur HC	Polymeg 650	REACTION GELLED									
1099414	Prepolymer 1099406 ①	Mondur HC	Polymeg 650	REACTION GELLED									
1099422A	Prepolymer 1099406 ①	Mondur HC	--	REACTION GELLED									
1099422B	Prepolymer 1099406 ①	Desmodur N	--	3.0	2936	5691	--	6590	210	7910	91	Passed	1
24 Hrs. @ 300°F				3.2	2209	--	--	2911	140	--	--	--	8
1099444	TMDI	Quadrol	--	2.6	--	--	--	9380	< 5	--	93	Failed	1
24 Hrs. @ 300°F				2.9				10483	< 5	--	--	--	3
1099445	H_{12}MDI	TMP	--	2.7	--	--	--	9023	< 5	--	99	Failed	1
24 Hrs. @ 300°F				2.5				10037	< 5	--	--	--	2

PHYSICAL PROPERTIES

① 0.382 eq. H_{12}MDI and 0.191 eq. Polymeg 650

TABLE XIII

PERCENT LOSS OF PROPERTIES AFTER THERMAL AGING

CROSSLINKING AGENTS

Ref.No.	AGENT	FUNCTIONALITY	24 Hours @ 300°F					
			% TENSILE LOST		% ELONGATION LOST		COLOR	
			Equivalents %*		Equivalents % *		Equivalents %*	
			10	20	10	20	10	20
F-9360,61	Poly-G 630 P	3	72.6	60.0	42.0	22.9	15	12
F-9366,67	Quadrol	4	80.8	47.2	22.7	12.5	11	3
F-9372,73	Trimethylolpropane	3	80.6	40.1	51.7	37.6	11	11
F-9382,83	1,2,6-Hexanetriol	3	72.4	45.2	71.4	33.3	12	13
F-9377,78	Pentaerythritol	4	54.2	0	42.2	33.3	6	10
F-9390	Mondur HC	3	73.3	-	5.0	-	5	-
F-9385,86	Desmodur N	3	65.6	48.5	39.5	17.8	9	11
1099418	tris(2-hydroxyethyl) isocyanurate	3	51.8	--	23.9	-	11	-

* Equivalents percent of crosslinking agent in total formulation.

TABLE XIV

POLYOL THERMAL STABILITY STUDY

PREPOLYMER PREPARATION

REFERENCE NUMBER	F-9366	F-9400	1099453	F-9405	1099407	1099408	1099409	1099419	1099424	1099443
$H_{12}MDI$										
Grams	51.6	50	50	50	51.6	51.6	51.6	51.6	51.6	51.6
Equivalents	0.382	0.382	0.382	0.382	0.382	0.382	0.382	0.382	0.382	0.382
Polyol	Polymeg 650	Niax 510	Niax 520	Niax 540	Carbowax 300	Carbowax 600	Carbowax 1000	Poly-G 420 P	Poly-G 750 P	Castor Oil DB
Grams	56.4	47.5	73.1	106.9	26.6	30.6	92.3	34.8	64.4	59.4
Equivalents	0.172	0.172	0.172	0.172	0.172	0.172	0.172	0.172	0.172	0.172
Quadrol										
Grams	1.39	1.39	1.39	1.39	1.39	1.39	1.39	1.39	1.39	1.39
Equivalents	0.0191	0.0191	0.0191	0.0191	0.0191	0.0191	0.0191	0.0191	0.0191	0.0191
Solvent	MIL Thin	MIL Thin	MIL Thin	MIL Thin	MIL Thin	MIL Thin	MIL Thin	MIL Thin	MIL Thin	MIL Thin
NCO:OH Ratio	2:1	2:1	2:1	2:1	2:1	2:1	2:1	2:1	2:1	2:1
% Theory NCO	3.72	4.06	3.22	2.60	5.04	3.87	2.76	4.57	3.42	3.62
% NCO Found	3.60	3.96	2.91	2.68	5.19	4.10	2.65	4.58	3.31	3.35
% Concentration of Solids	50	50	50	50	50	50	50	50	50	50

TABLE XV

POLYOL THERMAL STABILITY STUDY

PHYSICAL PROPERTIES

NCO:OH RATIO 2:1 - MOISTURE CURED - ISOCYANATE IS H_{12}MDI

CROSSLINKING AGENT QUADROL (10 EQUIVALENTS %)

REFERENCE NUMBER	POLYOL	FILM THICKNESS MILS.	TENSILE MODULUS, PSI			TENSILE AT BREAK PSI	ELONGATION %	ENERGY OF RUPTURE in-lbs/in^3	SHORE A HARDNESS	CONICAL MANDREL -65°F	GARDNER COLOR STANDARD
			100%	200%	300%						
P-9366	Polymeg 650	9.7	2001	3370	--	5642	270	15,622	87	Passed	1
After 24 Hrs. @ 300°F		6.3	800	1001	--	1086	230	--	--	--	11
P-9400	Niax 510	2.8	4402	6205	--	6205	200	8,535	86	Passed	1
After 24 Hrs. @ 300°F		3.0	2819	3895	--	4208	240	--	--	--	6
1099453	Niax 520	2.3	1866	2674	4988	6824	350	10,660	89	Passed	1
After 24 Hrs. @ 300°F		2.5	1368	1600	1925	2335	390	--	--	--	6
1099405	Niax 540	2.4	1029	1394	2338	6901	440	10,633	81	Passed	1
After 24 Hrs. @ 300°F		2.8	557	701	822	1168	570	--	--	--	6
1099407	Carbowax 300	3.1	--	--	--	7753	<5	--	93	Failed	1
After 24 Hrs. @ 300°F		TOO BRITTLE TO TEST									4
1099408	Carbowax 600	3.0	1990	3480	5148	5793	310	8,987	90	Passed	1
After 24 Hrs. @ 300°F		2.9	--	--	--	1066	50	--	--	--	6
1099409	Carbowax 1000	2.8	672	901	1154	3339	550	7363	81	Passed	1
After 24 Hrs. @ 300°F		2.6	--	--	--	157	40	--	--	--	11
1099419	Poly-G 420P	3.0	--	--	--	5050	50	--	95	Failed	1
After 24 Hrs. @ 300°F		TOO BRITTLE TO TEST									2
1099424	Poly-G 750P	2.2	2117	3595	--	4794	240	6,077	85	Passed	1
After 24 Hrs. @ 300°F		2.4	--	--	--	844	<10	--	--	--	4
1099443	Castor Oil DB	3.0	--	--	--	3475	~20		95	Passed	1
After 24 Hrs. @ 300°F		3.0	--	--	--	3114	40		--	--	8

TABLE XVI

OPTIMIZATION STUDY

FORMULATIONS AND PREPOLYMER PREPARATIONS

REFERENCE NUMBER	1099447	1099448	1099449	1099450 (1)	1099451	1099452 (2)	1099453	1099454 (1)	1099455	1099456 (1)
REACTANT A	H_{12}MDI	H_{12}MDI	H_{12}MDI	H_{12}MDI	H_{12}MDI	H_{12}MDI	H_{12}MDI	Desmodur N	H_{12}MDI	H_{12}MDI
Grams	50.0	50.0	50.0	5.53	25.4	50.0	50.0	7.07	51.6	3.5
Equivalents	0.382	0.382	0.382	0.0422	0.1932	0.382	0.382	0.0252	0.382	0.0267
REACTANT B	Quadrol	Desmophen 650	Desmophen 650	Desmophen 650	Polybutene 1000	Niax 520	Niax 520	Desmophen 650	Niax D-600	Niax D-600
Grams	1.39	39.8	7.95	8.0	65.0	73.1	73.1	5.0	6.92	4.4
Equivalents	0.0191	0.191	0.0382	0.0384	0.0859	0.1719	0.1719	0.02405	0.0382	0.0243
REACTANT C	Desmophen 650	--	Polymeg 650	--	Quadrol	Quadrol	Quadrol	--	Polymeg 650	--
Grams	35.8	--	51.04	--	0.78	1.39	1.39	--	51.1	--
Equivalents	0.1719	--	0.153	--	0.0107	0.019	0.019	--	0.1528	--
SOLVENT	Toluene/ Cellosolve Acetate 52 / 48	Ethyl Acetate/ Cellosolve Acetate 60 / 40	Ethyl Acetate/ Dioxane 50 / 50	Cellosolve Acetate/ Dioxane 60 / 40	Toluene	MIL Thin.	MIL Thin.	Ethyl Acetate	MIL Thin.	MIL Thin.
NCO:OH RATIO	2:1	2:1	2:1	1.1:1	2:1	2:1	2:1	1.1:1	2:1	1.1:1
% THEORY NCO	4.6	4.47	3.68	--	2.23	3.22	3.22	--	3.66	--
% NCO FOUND	Gelled	Gelled	3.82	--	2.39	2.96	2.91	--	3.54	--
% CONCENTRATION OF SOLIDS	50	50	50	50	50	50	50	50	50	50

(1) One-Shot

(2) No Films Made

TABLE XVI (CONTINUED)

OPTIMIZATION STUDY

FORMULATIONS AND PREPOLYMER PREPARATIONS

REFERENCE NUMBER	1099458	1099459	1099460	1099464 ③	1099465 ①	Part I F-7646	Part II F-7647	F-7648	F-7650	F-7650-1 ④
REACTANT A	H_{12}MDI	H_{12}MDI	H_{12}MDI	H_{12}MDI	Prepolymer 1099445 ②	H_{12}MDI	TiO_2	F-7646	H_{12}MDI	Prepolymer F-7650
Grams	51.6	51.6	51.6	51.6	17.7	51.6	32.3g	10g	51.6	66
Equivalents	0.382	0.382	0.382	0.382	0.02685	0.382	--	.0083	0.382	0.0668
REACTANT B	Niax 510	Niax 520	Niax 510	Niax 520	Niax 540	THEIC	Niax 540	F-7647	THEIC	Niax 520
Grams	37.5	48.7	37.5	36.5	15.18	16.6	100g	7.33g	16.6	9.37
Equivalents	0.1337	0.1146	0.1337	0.08595	0.0244	0.191	--	.00758	0.191	0.0223
REACTANT C	THEIC	THEIC	Quadrol	Castor Oil DB	--	--	--	--	--	--
Grams	4.98	7.64	4.19	26.6	--	--	--	--	--	--
Equivalents	0.0572	0.0878	0.0573	0.08595	--	--	--	--	--	--
SOLVENT	Toluene/ Dioxane 80 / 20	Toluene/ Dioxane 50 / 50	Toluene/ Dioxane 66 / 34	Toluene/ Dioxane 80 / 20	Toluene/ Dioxane 80 / 20	n-Butyl Acetate/ Dioxane 50 / 50	Dioxane/ Butyl Acetate 70 / 30	Dioxane	DMF Dioxane	Dioxane
NCO:OH RATIO	2:1	1.9:1	2:1	2:1	1.1:1	2:1	--	2:1	2:1	3:1
% THEORY NCO	3.9	2.9	3.8	3.06	--	4.1	--	--	4.1	2.23
% NCO FOUND	3.98	2.5	3.7	3.0	--	3.6	--	--	4.2	2.24
% CONCENTRATION OF SOLIDS	45	41	45	50	50	50	55	50	37	40

① One-Shot

② 8.54g Trimethylolpropane (0.191 eq.) + 50g H_{12}MDI (0.382 eq.)

③ Contains 10 eq. % THEIC

④ Physical Properties Not Measured

TABLE XVI (CONTINUED)

OPTIMIZATION STUDY

FORMULATIONS AND PREPOLYMER PREPARATIONS

REFERENCE NUMBER	F-7651	F-7652	F-7653	F-7654	F-7655	F-7655-1 ①	F-7655-2 ①	F-7656	F-7656-2	F-7657 ②
REACTANT A	H_{12}MDI	H_{12}MDI	H_{12}MDI	H_{12}MDI	H_{12}MDI	Prepolymer F-7655	Prepolymer F-7655	H_{12}MDI	H_{12}MDI	H_{12}MDI
Grams	51.6	51.6	51.6	51.6	51.6	10	10	14.35	19.1	1.23
Equivalents	0.382	0.382	0.382	0.382	0.382	0.0122	0.0122	0.1063	0.1417	.0091
REACTANT B	Pentaery-thritol	Pentaery-thritol	THEIC	THEIC	THEIC	RD-18	Niax 540	RD-18	RD-18	RD-18
Grams	1.3	1.3	16.6	16.6	16.6	2.6	3.9	30	30	3.5
Equivalents	0.0382	0.0382	0.191	0.191	0.191	0.00612	0.00612	0.0708	0.0708	.0083
REACTANT C	Niax 520	Niax 520	--	Niax 540	--	--	--	--	--	--
Grams	64.2	64.2	--	40.5	--	--	--	--	--	--
Equivalents	0.1528	0.1528	--	0.06367	--	--	--	--	--	--
SOLVENT	Dioxane	DMSO/ Xylene 50 / 50	Dioxane/ Diethylene Glycol Dimethyl Ether 80 / 20	Dioxane/ Diethylene Glycol Dimethyl Ether 75 / 25	Dioxane/ Pentane-dione 75 / 25	Dioxane	Dioxane	Butyl Acetate	Butyl Acetate	Butyl Acetate
NCO:OH RATIO	2:1	2:1	2:1	1.5:1	2.5:1	2:1	2:1	1.5:1	2:1	1.1:1
% THEORY NCO	3.43	3.44	4.1	1.97	5.94	--	--	1.67	3.03	--
% NCO FOUND	Gelled	3.8	Gelled	Gelled	5.2	--	--	Gelled	Gelled	--
% CONCENTRATION OF SOLIDS	50	50	35.0	40.0	40.0	40.0	40	50	50	50

① One-Shot, Film Gelled During Formation

② Films Too Brittle To Test

TABLE XVII

OPTIMIZATION STUDY

PHYSICAL PROPERTIES

REFERENCE NUMBER	F-9366 ①	F-9367 ①	F-9400 ①	1099453	1099405 ①	1099460	F-9377 ①	F-9378 ①
REACTANT A	H_{12}MDI	H_{12}MDI	H_{12}MDI	H_{12}MDI	H_{12}MDI	H_{12}MDI	H_{12}MDI	H_{12}MDI
Equivalents, %	100	100	100	100	100	100	100	100
REACTANT B	Polymeg 650	Polymeg 650	Niax 510	Niax 520	Niax 540	Niax 510	Polymeg 650	Polymeg 650
Equivalents, %	90	80	90	90	90	70	90	80
REACTANT C	Quadrol	Quadrol	Quadrol	Quadrol	Quadrol	Quadrol	Pentaery-thritol	Pentaery-thritol
Equivalents, %	10	20	10	10	10	30	10	20
FILM THICKNESS, MILS.	9.7	2.2	2.8	2.3	2.4	2.8	3.0	3.2
TENSILE @ BREAK, PSI	5642	5400	6205	6824	6901	5402	4803	3016
After 24 Hrs. @ 300°F	1086	2048	4208	2335	1168	4695	2202	3109
ELONGATION, %	270	200	200	350	440	10	255	150
After 24 hrs. @ 300°F	230	175	240	390	570	60	150	100
ENERGY OF RUPTURE in-lbs/in^3	15,622	6,900	8535	10,660	10,633	--	7452	2450
SHORE A HARDNESS	87	88	86	89	81	100	84	92
CONICAL MANDREL, -65°F	Passed	Failed	Passed	Passed	Passed	Passed	Passed	Passed
GARDNER COLOR STANDARD	1	1	1	1	1	1	1	1
After 24 hrs. @ 300°F	11	3	6	6	6	10	6	10

① Previously Reported

TABLE XVII (CONTINUED)

OPTIMIZATION STUDY

PHYSICAL PROPERTIES

REFERENCE NUMBER	F-7652	1099418 ①	1099458	1099459	1099437 ①	1099464 ②	1099455	1099456
REACTANT A	H_{12}MDI	H_{12}MDI	H_{12}MDI	H_{12}MDI	H_{12}MDI	H_{12}MDI	H_{12}MDI	H_{12}MDI
Equivalents, %	100	100	100	100	100	100	100	100
REACTANT B	Niax 520	Polymeg 650	Niax 510	Niax 520	THEIC	Niax 520	Polymeg 650	--
Equivalents, %	80	90	70	60	68	95	80	--
REACTANT C	Pentaery-thritol	THEIC	THEIC	THEIC	Castor Oil DB	Castor Oil DB	Niax D-600	Niax D-600
Equivalents, %	20	10	30	40	42	45	20	100
FILM THICKNESS, MILS.	2.2	3.0	2.5	2.5	2.7	1.9	1.7	2.9
TENSILE @ BREAK, PSI	4237	4662	5753	5655	7898	4784	4257	4547
After 24 hrs. @ 300°F	4350	2273	5052	5641	7874	3478	2505	6033
ELONGATION, %	140	210	80	150	5	120	170	105
After 24 hrs. @ 300°F	180	160	40	190	5	160	150	20
ENERGY OF RUPTURE in-lbs/in^3	2863	6827	--	6527	--	3747	4977	2713
SHORE A HARDNESS	86	98	95	95	91	92	93	92
CONICAL MANDREL, -65°F		Passed	Passed		Failed	Passed	Passed	Failed
GARDNER COLOR STANDARD	1	1	1	1	1	1	1	1
After 24 hrs. @ 300°F	10	11	2	4	2	10	9	2

① Previously Reported

② Contains 10 equivalents per cent THEIC

TABLE XVII (CONTINUED)

OPTIMIZATION STUDY

PHYSICAL PROPERTIES

REFERENCE NUMBER	REACTANT A	REACTANT B	REACTANT C	FILM THICKNESS MILS.	TENSILE AT BREAK PSI	ELONGATION %	ENERGY OF RUPTURE in-lbs/in^3	SHORE A HARDNESS	CONICAL MANDREL -65°F	GARDNER COLOR STANDARD
1099449	H_{12}MDI	Polymeg 650	Desmophen 650	2.9	5357	140	5310	88	Passed	1
	24 hrs. @ 300°F			3.2	3682	105	2936	--	--	10
1099450	H_{12}MDI	--	Desmophen 650	3.1	10,996	5	--	98	Failed	1
	24 hrs. @ 300°F			2.6	10,883	5	--	--	--	1
1099451	H_{12}MDI	Polybutene 1000	Quadrol	2.6	2148	220	3080	79	--	1
	24 hrs. @ 300°F			2.7	820	70	--	--	--	3
1099454	Desmodur N	Desmophen 650	--	2.7	4338	10	--	99+	Failed	1
	24 hrs. @ 300°F			3.1	10,515	5	--	--	--	2
1099465	H_{12}MDI	TMP	Niax 540	2.3	2375	130	2146	83	Passed	1
	24 hrs. @ 300°F			2.5	1901	260	1828	--	--	8
F-7648-1 ①	H_{12}MDI	THEIC	Niax 540	1.9	4358	40	2490	94		--
	24 hrs. @ 300°F			2.0	4845	120	4730	--	--	--
F-7648-2	H_{12}MDI	THEIC	Niax 540	2.1	4522	40	4005	92		--
	24 hrs. @ 300°F			2.1	5223	90	3490	--	--	--
F-7655-1	H_{12}MDI	THEIC	RD-18	2.9	8490	5	--	92		1
	24 hrs. @ 300°F			2.6	11,516	5	--	--	--	5
F-7655-2	H_{12}MDI	THEIC	Niax 540	2.2	4237	140	3780	94	--	1
	24 hrs. @ 300°F			2.1	4350	180	4407	--	--	3

① Pigmented

TABLE XVIII

THERMAL STABILITY VS. EQUIVALENT WEIGHT PER CROSSLINK

REFERENCE NUMBER	COMPONENT A FUNCTIONALITY 2	COMPONENT B		Eq. Wt. Crosslink	% RETENTION OF TENSILE After 24 Hrs. @ 300°F
		Eq %	f *		
1099460	Niax 510	30% Quadrol	4	3,260	86.4
1099459	Niax 520	40% THEIC	3	3,630	95.6
1099458	Niax 510	30% THEIC	3	4,840	87.8
F-9366	Polymeg 650	20% Quadrol	4	5,380	37.9
F-9378	Polymeg 650	20% Pentaery-thritol	4	5,430	100.0
F-7652	Niax 520	20% Pentaery-thritol	4	6,020	100.0
F-9400	Niax 510	10% Quadrol	4	10,300	67.7
F-9367	Polymeg 650	10% Quadrol	4	11,260	19.2
F-9377	Polymeg 650	10% Pentaery-thritol	4	11,500	45.7
1099453	Niax 520	10% Quadrol	4	12,960	34.2
1099446	Niax 510	10% THEIC	3	15,600	69.6
1099405	Niax 540	10% Quadrol	4	16,380	17.4
1099418	Polymeg 650	10% THFIC	3	17,030	48.8

* Functionality

TABLE XIX

ACCELERATED WEATHERING

CLEAR UNPROTECTED FILMS ON ANODIZED ALUMINUM

REFERENCE NUMBER	COMPONENTS						Q-U-V UNIT TOTAL TEST 900 Hrs.			OUTDOOR WEATHERING Start 6/16/70 Still in Progress	
	A	Eq.%	B	Eq.%	C	Eq.%	TOTAL HRS	TOTAL HRS, UV LIGHT	REMARKS	TIME TO FAILURE	REMARKS
1099406	H_{12}MDI	100	Polymeg 650	100	-	-	195	132	Badly cracked, some lift off.	6 weeks	Cracks and lift off.
F-7643-1	H_{12}MDI	100	Polymeg 650	90	THEIC	10	195	132	Very many fine cracks.	-	———
1099405	H_{12}MDI	100	Niax 540	90	Quadrol	10	724	492	Soft, mushy.	8 weeks	Soft, picking up soot badly.
1099441	H_{12}MDI	100	TMDI	100	-	-	-	-	———	8 weeks	Soft, picking up soot badly.
1099427A	H_{12}MDI	100	THEIC	67	Niax 510	33	900	606	Hard, clear, glossy, colorless.	-	———
1099428-2	H_{12}MDI	100	THEIC	75	Niax 540	25	900	606	Hard, clear, glossy, colorless.	-	———
1099437	H_{12}MDI	100	THEIC	75	Castor Oil DB	25	900	606	Trace yellow, hard glossy, clear.		Colorless, Hard, glossy, clear.
1099439	H_{12}MDI	100	THEIC	75	Polycin 52	25	900	606	very slight yellow, hard, glossy, clear.		colorless, hard, glossy, clear.
1099443	H_{12}MDI	100	Castor Oil DB	90	Quadrol	10	900	606	yellow, clear, glossy, hard.	-	———

TABLE XX

POTENTIAL CANDIDATE SYSTEMS

— PREPOLYMER PREPARATION ——

REFERENCE NUMBER	F-7660	F-7661	F-7662	F-7663 2 Part System		P-7664 2 Part System	
				A	B	A	B
REACTANT A	H_{12}MDI	H_{12}MDI	H_{12}MDI	H_{12}MDI	-	H_{12}MDI	-
Grams	147.7	155.2	155.2	105.9	-	111.3	-
Equivalents	1.094	1.149	1.149	0.784	-	.824	-
REACTANT B	Niax 520	Niax 520	Niax 520	THEIC	-	THEIC	-
Grams	160.7	144.8	144.8	34.1	-	28.7	-
Equivalents	0.383	0.345	0.345	0.392	-	0.330	-
REACTANT C	THEIC	THEIC	THEIC	-	Niax 540	-	Niax 540
Grams	14.3	20.0	20.0	-	95.6	-	135.6
Equivalents	0.164	0.23	0.23	-	.154	-	0.218
SOLVENT	Toluene/ Dioxane 60/40	Toluene/ Dioxane 60/40	Dioxane/ D_2M 60/10	Dioxane/ D_2M 60/40	Dioxane/ D_2M 60/40	Dioxane/ Toluene 60/40	Dioxane/ Toluene 60/40
NCO:OH RATIO	2:1	2:1	2:1	2:1	-	2.5:1	-
% THEORY NCO	2.87	3.01	3.01	4.12	-	5.19	-
% NCO FOUND	2.60	2.93	3.10	3.16	-	4.48	-
% CONCENTRATION OF SOLIDS	40	40	40	35	50	35	47.5

TABLE XXI

POTENTIAL CANDIDATE SYSTEMS

- Tensile Strength and Elongation Measurements -

REFERENCE NUMBER	PRIMER	MEASUREMENTS TAKEN AT AMBIENT TEMPERATURE						MEASUREMENTS TAKEN AT -65°F					
		UNTREATED			AFTER 48 HRS. @ 300°F			UNTREATED			AFTER 48 HRS. @ 300°F		
		MILS.	TENSILE STRENGTH PSI	ELONGATION %	MILS	TENSILE STRENGTH PSI	ELONGATION %	MILS.	TENSILE STRENGTH PSI	ELONGATION %	MILS.	TENSILE STRENGTH PSI	ELONGATION %
Control A	Control A Primer	2.7	--	15	2.6	--	0	2.6	--	2.3	2.8	--	3.4
	MIL-P-23377B	3.6	--	10	3.1	--	0	3.8	--	3.6	3.7	--	1.8
	Unprimed	2.5	3414	30	2.6	4846	0	2.6	6716	2.7	2.6	7900	2.8
Control B	MIL-P-23377B	3.6	--	5	3.2	--	5	3.1	--	3.5	3.2	--	3.7
	Unprimed	1.6	3995	30	1.6	4283	25	1.6	7713	2.1	1.6	11,823	3.2
F-7660	MIL-P-23377B	5.2	--	0	5.0	--	0	5.1	--	3.2	5.2	--	2.9
	Unprimed	2.9	4376	170	2.9	3941	200	3.8	11,128	7.6	3.2	10,755	5.6
F-7661	MIL-P-23377B	5.0	--	0	4.8	--	0	--	--	--	--	--	--
	Unprimed	2.6	4381	110	2.4	4256	100	2.6	14,239	7.0	2.7	10,319	4.6
F-7662	MIL-P-23377B	3.0	--	0	3.8	--	0	--	--	--	--	--	--
	Unprimed	1.9	4363	130	2.0	4078	100	1.8	13,059	6.4	1.9	11,201	4.9
F-7663	MIL-P-23377B	4.3	--	0	4.7	--	0	--	--	--	--	--	--
	Unprimed	3.0	5132	30	2.9	6486	20	--	--	--	--	--	--
F-7664	MIL-P-23377B	4.6	--	0	5.3	--	0	--	--	--	--	--	--
	Unprimed	3.3	6104	20	3.1	6832	20	3.2	14,174	3.5	3.4	12,083	4.3

TABLE XXII

POTENTIAL CANDIDATE SYSTEM

PERCENT RETENTION OF TENSILE STRENGTH AFTER THERMAL AGING *

- For Unprimed Films -

REFERENCE NUMBER	PRIMER	TESTED AT ROOM TEMP. % LOSS OF TENSILE	TESTED AT -65°F % LOSS OF TENSILE
CONTROL A	UNPRIMED	0	0
CONTROL B	UNPRIMED	0	0
F-7660	UNPRIMED	9.9	3.3
F-7661	UNPRIMED	3.1	27.5
F-7662	UNPRIMED	6.5	14.2
F-7663	UNPRIMED	0	--
F-7664	UNPRIMED	0	14.7

* 48 Hrs. @ 300°F

TABLE XXIII

EVALUATION OF POTENTIAL CANDIDATE SYSTEMS

REFERENCE NUMBER	PRIMER	REVERSE IMPACT @ -65°F IN-LBS	WET TAPE ADHESION	ENERGY OF RUPTURE IN-LBS/IN^3	SHORE A HARDNESS
Control A	MIL-P-23377B	<2	Passed	--	96
	Control A Primer	<2	Passed	--	95
	Unprimed	<2	--	654	93
Control B	MIL-P-23377B	<2	Passed	--	94
	Unprimed	--	--	825	92
F-7660	MIL-P-23377B	60	Passed	--	99
	Unprimed	160	Passed	5275	96
F-7661	MIL-P-23377B	30	Passed	--	96
	Unprimed	80	Passed	3816	92
F-7662	MIL-P-23377B	16	Failed	--	98
	Unprimed	60	Failed	4523	91
F-7663	MIL-P-23377B	2	Failed	--	96
	Unprimed	3	Failed	1014	94
F-7664	MIL-P-23377B	12	Passed	--	96
	Unprimed	30	Passed	8983	91

TABLE XXIV

1/8" CONICAL MANDREL BEND AT -65°F

BEFORE AND AFTER THERMAL AGING

REFERENCE NUMBER	PRIMER	AGED 72 HRS @ 25°F 1/8" CONICAL MANDREL BEND @ -65°F	AFTER 48 HRS. @ 300°F 1/8" CONICAL MANDREL BEND @ -65°F
Control A	Control A Primer	Failed	Failed (crack = 5.86" Long)
	MIL-P-23377P	Failed	Failed (crack = 5.64" Long)
	Unprimed	Failed	--
Control B	MIL-P-23377P	Failed	Failed (crack = 0.69" Long)
	Unprimed	Failed	--
F-7660	Unprimed	Passed	Passed
	MIL-P-23377B	Passed	Failed (crack = 0.04" Long at Tip)
	Hughson 9924	Passed	Passed
F-7661	Unprimed	Passed	Passed
	MIL-P-23377B	Passed	Failed (crack = 3.12" Long)
F-7662	Unprimed	Passed	Passed
	MIL-P-23377B	Passed	Failed (crack = 0.14" Long)
F-7663	Unprimed	Passed	Passed
	MIL-P-23377B	Failed	Failed (crack = 0.04" Long)
F-7664	Unprimed	Passed	Passed
	MIL-P-23377B	Passed	Failed (crack = 2.63" Long)

ALL SYSTEMS PIGMENTED @ 15% ON SOLIDS WITH TiO_2

TABLE XXV

ABRASION RESISTANCE AS MEASURED ON S.S. WHITE

MODEL E INDUSTRIAL AIRBRASIVE UNIT

REFERENCE NUMBER	FORMULATION	PRIMER	FILM THICKNESS MILS.	TIME SEC.	SEC./MILS
Control A	Commercial System	Control A	2.0	2.4	1.2
			2.0	2.6	1.3
			2.0	3.2	1.6
			Ave. 2.0	2.7	1.3
Control A	Commercial System	MIL-P-23377B	2.5	5.0	2.0
			2.0	6.0	3.0
			2.0	5.2	2.6
			Ave. 2.2	5.4	2.5
Control B	Commercial System	MIL-P-23377B	2.6	13.5	5.2
			2.8	12.4	4.4
			2.5	11.5	4.6
			Ave. 2.6	12.4	4.7
F-7660	$H_{12}MDI$, 70 eq.% Niax 520	--	2.0	18.0	9.0
	30 eq.% THEIC		2.0	19.5	9.75
	Solvent-Toluene/dioxane		2.0	20.1	10.05
			Ave. 2.0	19.2	9.6
F-7660	$H_{12}MDI$, 70 eq.% Niax 520	MIL-P-23377B	2.0	46.7	23.35
	30 eq.% THEIC		2.0	49.6	24.8
	Solvent-Toluene/dioxane		2.0	50.2	25.1
			Ave. 2.0	48.8	24.8

TABLE XXV (CONTINUED)

ABRASION RESISTANCE AS MEASURED ON S.S. WHITE

MODEL F INDUSTRIAL AIRBRASIVE UNIT

REFERENCE NUMBER	FORMULATION	PRIMER	FILM THICKNESS MILS.	TIME SEC.	SEC./MILS
F-7661	H_{12}MDI, 60 eq.% Niax 520	--	4.0	29.2	7.3
	40 eq.% THEIC		4.0	25.2	6.3
	Solvent-Toluene/dioxane		4.0	22.8	5.7
			Ave.4.0	25.7	6.4
F-7661	H_{12}MDI, 60 eq.% Niax 520	MIL-P-23377B	2.0	46.8	23.4
	40 eq.% THEIC		2.0	43.2	21.6
	Solvent-Toluene/dioxane		2.5	38.4	15.3
			Ave.2.2	42.8	20.1
F-7662	H_{12}MDI, 60 eq.% Niax 520	--	2.5	19.8	7.9
	40 eq.% THEIC		2.5	18.4	7.4
	Solvent-Dioxane/D_2M		2.5	15.4	6.3
			Ave.2.5	17.8	7.2
F-7662	H_{12}MDI, 60 eq.% Niax 520	MIL-P-23377B	2.5	25.8	10.3
	40 eq.% THEIC		2.5	20.2	8.1
	Solvent-Dioxane/D_2M		2.5	24.3	9.7
			Ave.2.5	23.4	9.3
F-7663	Two Part System	--	3.0	19.5	6.5
	H_{12}MDI, Niax 540, THEIC		4.0	22.2	5.5
	Solvent-Dioxane/D_2M		4.0	26.9	6.7
			Ave.3.7	22.8	6.2

TABLE XXV

ABRASION RESISTANCE AS MEASURED ON S.S. WHITE

MODEL F INDUSTRIAL AIRBRASIVE UNIT

REFERENCE NUMBER	FORMULATION	PRIMER	FILM THICKNESS MILS.	TIME SEC.	SEC./MILS
Control A	Commercial System	Control A	2.0	2.4	1.2
			2.0	2.6	1.3
			2.0	3.2	1.6
			Ave. 2.0	2.7	1.3
Control A	Commercial System	MIL-P-23377B	2.5	5.0	2.0
			2.0	6.0	3.0
			2.0	5.2	2.6
			Ave. 2.2	5.4	2.5
Control B	Commercial System	MIL-P-23377B	2.6	13.5	5.2
			2.8	12.4	4.4
			2.5	11.5	4.6
			Ave. 2.6	12.4	4.7
F-7660	$H_{12}MDI$, 70 eq.% Niax 520	--	2.0	18.0	9.0
	30 eq.% THEIC		2.0	19.5	9.75
	Solvent-Toluene/dioxane		2.0	20.1	10.05
			Ave. 2.0	19.2	9.6
F-7660	$H_{12}MDI$, 70 eq.% Niax 520	MIL-P-23377B	2.0	46.7	23.35
	30 eq.% THEIC		2.0	49.6	24.8
	Solvent-Toluene/dioxane		2.0	50.2	25.1
			Ave. 2.0	48.8	24.8

TABLE XXVI

EVALUATION OF POTENTIAL CANDIDATE SYSTEMS

THERMAL AGING - 48 HOURS @ 300°F

REFERENCE NUMBER	PRIMER	BEFORE THERMAL AGING: 48 HRS @ 300°F			AFTER THERMAL AGING: 48 HRS @ 300°F			% LOSS OF BRIGHTNESS (Y-Y')
		COLOR EYE			COLOR EYE			
		X	Y	Z	X'	Y'	Z'	
CONTROL A	Control A Primer	101.9	102.3	102.1	92.3	99.2	94.9	2.9
	MIL-P-23377B	100.6	100.6	100.7	88.1	95.5	90.5	5.1
	Unprimed	101.4	101.4	101.5	65.9	78.6	68.8	24.8
CONTROL B	MIL-P-23377B	98.8	99.2	99.1	86.0	94.1	88.8	5.1
F-7660	MIL-P-23377B	92.1	92.9	92.5	60.6	72.4	63.4	20.5
	Unprimed	86.3	85.5	86.2	70.1	78.0	72.4	7.5
F-7661	MIL-P-23377B	91.7	92.3	92.0	76.4	84.8	78.9	7.5
	Unprimed	89.1	88.4	88.9	75.0	82.1	77.2	6.3
F-7662	MIL-P-23377B	90.8	90.3	90.7	77.3	84.5	79.6	5.8
	Unprimed	89.1	87.7	88.7	73.3	80.6	75.5	7.1
F-7663	MIL-P-23377B	88.9	88.8	89.1	76.5	83.0	78.8	5.8
	Unprimed	88.2	87.1	87.8	76.1	81.8	78.0	5.3
F-7664	MIL-P-23377B	87.8	87.8	88.0	57.9	69.6	60.6	18.2
	Unprimed	85.8	84.5	85.3	75.2	80.5	77.1	4.0

TABLE XXVII

SOLVENT COMPATABILITY

H_{12}MDI THEIC PREPOLYMER SYSTEMS

REFERENCE NUMBER	SOLVENT SYSTEMS		REMARKS
	A	B	
1099423	No Solvent		Added THEIC to molten H_{12}MDI at 70°C - No reaction
1099423A	No Solvent		Added THEIC to molten H_{12}MDI at 70°C with catalyst (D-22) - No reaction
1099425	Toluene	-	Mixed all reactants together with catalyst (D-22) @ 100°C - No reaction
1099426	THF	-	Reaction went almost to completion in one hour @ 70°C (catalyst D-22) Trace of insolubles left which did not react
1099435	Dioxane	-	Reaction went to completion in 30 min. No insolubles Catalyst D-22
F-7646	54% Dioxane	46% Butyl Acetate	Reaction went but became cloudy and gelled after 1 week Catalyst D-22
F-7649	54% DMF	46% Dioxane	Reaction went; solution looked good; viscosity increased on storage. Catalyst D-22
F-7653	75% Dioxane	25% Diethylene Glycol Dimethyl Ether	Near end of reaction solution became cloudy started to show gel formation. Catalyst D-22
F-7655	75% Dioxane	25% 1,5-Pentane-dione	Reaction went to theory, but gelled after 3 days storage Catalyst D-22
F-7664	60% Toluene	40% Dioxane	Reaction went, solution clear. Systems sprayed Ok. Catalyst D-22

TABLE XXVIII

RULE 66 SOLVENTS SYSTEMS

SOLVENT MIXTURES	VOLUME % OF MIXTURE	SOLUBILITY PARAMETER δ	HYDROGEN BONDING γ	DIPOLE MOMENT μ	AVERAGE VALUES FOR SOLVENT MIXTURE		
					δ	γ	μ
DIOXANE	40	9.9	9.7	0.4	9.3	6.6	0.4
TOLUENE	60	8.9	4.5	0.4			
BENZENE	16	9.2	0	0	9.3	7.8	0.9
BUTYL ACETATE	16	8.5	8.8	1.9			
METHYL ETHYL KETONE	4	9.3	7.7	2.7			
DIOXANE	44	9.9	9.7	0.4			
CELLOSOLVE ACETATE	16	8.5	9.4	1.8			
DIOXANE	50	9.9	9.7	0.4	9.4	8.2	1.00
TOLUENE	20	8.9	4.5	0.4			
METHYL ETHYL KETONE	20	9.3	7.7	2.7			
CELLOSOLVE ACETATE	10	8.5	9.4	1.8			
DIOXANE	50	9.9	9.7	0.4	9.7	7.2	1.2
TRICHLOROETHANE	25	9.6	1.5	1.2			
METHYL ETHYL KETONE	25	9.3	7.7	2.7			
DIOXANE	40	9.9	9.7	0.4	9.0	7.5	0.9
BUTYL ACETATE	20	8.5	8.8	1.9			
CYCLOHEXANE	20	8.2	0	0			
CELLOSOLVE ACETATE	20	8.5	9.4	1.8			

TABLE XXIX

CANDIDATE SYSTEM WITH VARIOUS RULE 66 SOLVENT SYSTEMS

MECHANICAL PROPERTIES

REFERENCE NUMBER	FILM THICKNESS MILS.	TENSILE STRENGTH PSI	ELONGATION %	SHORE A HARDNESS	CONICAL MANDREL	SOLVENT SYSTEM
1099484	3.0	5477	240	91	Passed	8.2% Methyl Ethyl Ketone, 16.1% Benzene, 16.1% Butyl Acetate, 44.3% Dioxane, 15.3% Cellosolve Acetate.
48 Hrs.@ 300°F	3.6	3494	310	--	--	
1099486	2.7	5560	240	94	Passed	50% Dioxane, 20% Toluene, 20% Methyl Ethyl Ketone, 10% Cellosolve Acetate.
48 Hrs.@ 300°F	2.9	4226	320	--	--	
1099487	2.4	4054	210	93	Passed	50% Dioxane, 20% Toluene, 20% Methyl Ethyl Ketone, 10% Cellosolve Acetate.
48 Hrs.@ 300°F	2.6	3579	300	--	--	
1099488	3.2	5039	220	93	Passed	40% Dioxane, 20% Butyl Acetate, 20% Cyclohexane, 20% Cellosolve Acetate.
48 Hrs.@ 300°F	3.2	4868	330	--	--	
1099491	2.9	4963	180	95	Passed	40% Dioxane, 20% Butyl Acetate, 20% Cyclohexane, 20% Cellosolve Acetate.
48 Hrs.@ 300°F	3.2	4190	180	--	--	

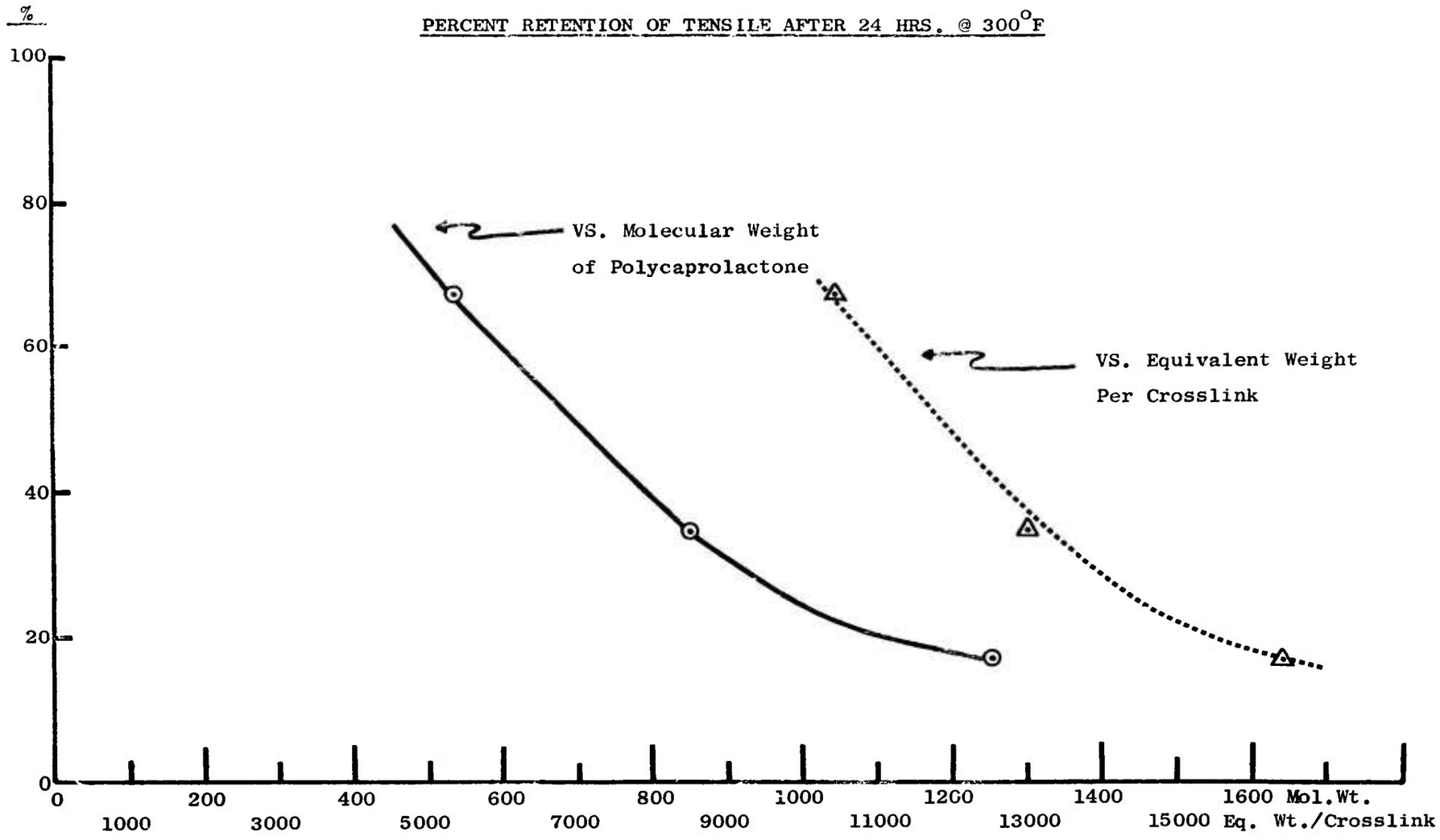
TABLE XXX

MECHANICAL PROPERTIES OF CANDIDATE SYSTEM

WHEN FORMULATED AT AN NCO:OH RATIO OF 1.5:1

REFERENCE NUMBER	FILM THICKNESS MILS.	TENSILE STRENGTH PSI	ELONGATION %	SHORE A HARDNESS
1099494	3.7	4029	20	90
48 Hrs. @ 300°F	3.9	1865	70	--
1099496	2.9	5644	180	94
48 Hrs. @ 300°F	3.1	2543	180	--

FIGURE 1


%
PERCENT RETENTION OF TENSILE AFTER 24 HRS. @ 300°F
100
80
60
40
20
0
VS. Molecular Weight
of Polycaprolactone
VS. Equivalent Weight
Per Crosslink
0
200
400
600
800
1000
1200
1400
1600 Mol.Wt.
1000
3000
5000
7000
9000
11000
13000
15000 Eq. Wt./Crosslink

APPENDIX I

OBJECTIVES OF CONTRACT N00019-70-C-0309

Polyurethane coatings for Naval Aircraft.

The coating should meet the following requirements:

1. Flexible at -65°F as determined by

   (a) 1/8" Conical Mandrel Test

   (b) Reverse impact strength test

   (c) Instron measurements at -65°F for elongation

2. Cure at ambient temperature:

Tack-free time	1½ hrs. max.
Fully cured	72 hrs. max.
Pot-life	8 hrs. min.

3. Pass 500 hr. accelerated weathering test (Atlas Weather-O-Meter). Must retain gloss and should not chalk.

4. Abrasion resistance - at least 10 sec/mil of thickness using S.S. White Industrial Abrasive Unit.

5. High temperature aging at 300°F for 48 hrs. without yellowing or losing more than 50% of its flexibility.

6. Good adhesion even after 24 hrs. immersion in distilled water.

7. Good resistance to jet fuels, hydraulic fluids and water.

8. Soluble in non-air-polluting solvents.

APPENDIX II

DETERMINATION OF NCO GROUPS IN ISOCYANATE PREPOLYMERS AND BLOCKED PREPOLYMERS

(Procedure Using Toluene-PolySolv EE Acetate)

A. PRINCIPLE

The free NCO groups of the dissolved sample react with an excess of di-n-butylamine in a toluene-PolySolv EE Acetate mixture. After the reaction is complete, the excess di-n-butylamine is determined by titration with standard hydrochloric acid solution.

B. APPARATUS

1. 250 ml. Erlenmeyer flasks.

2. Glass stoppers to fit above flasks.

C. REAGENTS

Needed

1. Toluene-PolySolv EE Acetate mixture (50/50 by volume) dried with No. 5A molecular sieve pellets (Note 1).

2. Di-n-butylamine solution (0.1 N.) in dry toluene-PolySolv EE Acetate mixture - 8.9 ml. dba per 500 ml. solution. (This solution is not standardized; strength handled by blank.)

3. Hydrochloric acid solution (0.1 N.) standardized using bromophenol blue indicator.

4. Bromophenol blue indicator solution, 0.1% - Triturate 0.1 grams dry indicator with 1.5 ml. 0.1 N. sodium hydroxide and dilute to 100 ml. with distilled water.

5. Isopropyl alcohol, anhydrous grade.

Sources

(a) Toluene - Baker & Adamson ACS Reagent, Code 2398.

(b) PolySolv EE Acetate - Olin - urethane grade.

(c) Di-n-butylamine - Distillation Products - Cat.# 1260.

(d) Bromophenol blue (3', 3", 5', 5" - tetrabromophenolsulfonephthalein) Distillation Products, Cat. #752.

(e) Molecular Sieves No. 5A - Linde Company.

(f) Isopropyl alcohol - Mallinckrodt A.R., Cat. #3032.

D. PROCEDURE

SAMPLE SIZE

Approximating 1.5 Milli-equivalents of NCO

Estimated % NCO	Gram Sample
100-90	0.05 - 0.06
90-70	0.06 - 0.075
70-50	0.075 - 0.105
50-30	0.105 - 0.175
30-10	0.175 - 0.50
10-5	0.50 - 1.05
5-0	1.05 - 10.00

Weigh the sample containing 1.5 milli-equivalents of NCO (based on above table) into a dry 250 ml. Erlenmeyer flask. Add 25 ml. dry toluene-PolySolv EE Acetate mixture, stopper, and swirl until the sample is completely dissolved (Note 2). Add 25.00 ml. (pipet or buret) of 0.1 N. di-n-butylamine solution and agitate until solids are completely dissolved (Note 3).

Add 100 ml. isopropyl alcohol and 4 to 6 drops bromophenol blue indicator solution. Titrate with 0.1 N. HCl solution to a yellow-green end point.

Run a blank including all reagents but omitting the sample.

E. CALCULATIONS

$$\frac{(B - V) \times N \times 4.202}{\text{Weight of sample (g.)}} = \% \text{ NCO}$$

B = ml. of 0.1 N. HCl required for blank.

V = ml. of 0.1 N. HCl required for the sample.

NOTE 1: Shake toluene-PolySolv EE Acetate mixture with No. 5A molecular sieve pellets several times and allow to stand for at least 24 hours before use to insure adequate drying.

NOTE 2: If the sample is a solution or readily soluble material, agitate in the Erlenmeyer flask until the solution appears homogeneous. Some blocked isocyanate prepolymers are solids (eg. MONDUR S). It has been difficult to dissolve these samples without destroying the blocked NCO groups. The technique which has been shown to be the least harmful has been the preparation of a 50-50 solution (by weight) of the sample in the mixed solvent by vigorous agitation such as obtained with the Red Devil paint conditioner. One hour agitation is normal for this procedure. This solution then can be handled easily.

NOTE 3: Agitate for 10-15 minutes to allow complete reaction of the di-n-butylamine solution with the isocyanate groups and to redissolve any precipitating material.

APPENDIX III

GLOSSARY OF TERMS

Carbowax 300	Polyoxyethylene glycol 300 mol.wt; Union Carbide
Carbowax 600	Polyoxyethylene glycol 600 mol.wt; Union Carbide
Carbowax 1000	Polyoxyethylene glycol 1000 mol.wt; Union Carbide
Castor 1066	Glyceryl tri-ricinoleate, Baker Castor Oil Co.
Castor Oil DB	Especially refined Castor Oil, Baker Castor Oil Co.
D-22	Dibutyl tin dilaurate - catalyst,Union Carbide Corp.
D_2M	Diethylene glycol dimethylether, Olin Corporation
Desmodur N	Biuret based triisocyanate, Naftone
Desmophen 650	Polyester, Naftone
$H_{12}MDI$	Methylene bis (cyclohexyl isocyanate), aliphatic diisocyanate, Allied
MIL Thinner	Solvent mixture 45% toluene, 45% methyl isobutyl ketone, 5% xylene and 5% cellosolve acetate.
Mondur HC	Triisocyanate prepolymer, 2 parts aliphatic and 1 part aromatic isocyanates; Mobay
Niax 510	Caprolactone polyol of 530 mol.wt; Union Carbide
Niax 520	Caprolactone polyol of 840 mol.wt; Union Carbide
Niax 540	Caprolactone polyol of 1250 mol.wt; Union Carbide
Niax D-600	A trifunctional polycaprolactone, Union Carbide
Pale 16	Polymerized Castor Oil, Baker Castor Oil Co.
Polycin 52, 58	A Castor Oil based prepolymer; Baker Castor Oil Co.
Poly-G 420P	Oxypropylated glycol of 400 mol.wt; Olin Corporation
Poly-G 750P	Oxypropylated glycol of 750 mol.wt; Olin Corporation
RD-18	Polyester, Naftone
Quadrol	N,N,N',N'-tetrakis (2-hydroxypropyl) ethylene diamine; Wyandotte
THEIC	Tris (2-hydroxyethyl) isocyanurate; Allied Chemicals
TMDI	Trimethylhexamethylene diisocyanate; International Chemical Corporation.