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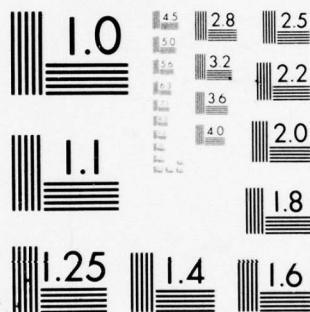
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INVESTIGATION OF COATING COMPOUNDS AND
ADHESIVES FOR SELF-SUPPORTING COLLAPSIBLE
FUEL STORAGE TANKS

FINAL TECHNICAL REPORT

BY

C. T. CHMIEL
D. V. PERKINS
G. W. BERNIER

ENGINEERED SYSTEMS DEPARTMENT

JUNE, 1977

U. S. ARMY MOBILITY EQUIPMENT RESEARCH
AND DEVELOPMENT CENTER
FORT BELVOIR, VIRGINIA 22060

Prepared by
UNIROYAL, Incorporated
Mishawaka, Indiana 46544
Contract Number DAAG53-76-C-0140

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UNIROVAL has evaluated coatings and adhesives as potential candidates from which self-supporting collapsible fuel storage tanks can be fabricated. The evaluation was concerned with polyurethane coatings, and was performed utilizing Glass, Kevlar, Dacron and Nylon fabrics as reinforcing members. Emphasis was placed on spray coating of one-shot polyurethane materials.		

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SUMMARY

UNIROYAL has evaluated coatings and adhesives for the purpose of improving the performance of the coated fabric from which self-supporting collapsible fuel storage tanks are fabricated. The evaluation was concerned with polyurethane coatings, and was performed utilizing Glass, Kevlar, Dacron and Nylon fabrics as reinforcing members. Emphasis was directed toward the following specific areas of fuel storage tank technology:

1. reducing stiffness of coatings at lower temperatures,
2. improving long-term aging of coatings in water at elevated temperatures,
3. improving aging of coatings in acidified fuel,
4. applying coatings by a non-solvent, spray technique.

UNIROYAL's approach involved 2 distinct but complementary efforts:

1. developing new coatings at its Corporate Research Center in Connecticut through the utilization of a non-solvent, one-shot polyurethane system in place of the currently used solvent prepolymer polyurethane system,
2. developing new adhesives at its Mishawaka, Indiana facility for bonding one-shot polyurethane coatings to themselves, to fabrics and to metal.

An optimum polyether and an optimum polyester polyurethane were defined at the UNIROYAL Research Center. At Mishawaka, adhesives were defined for bonding coatings to fabrics, coatings to coatings, and coatings to aluminum. Also at Mishawaka, the spraying technique was evaluated for application of one-shot polyurethane coatings onto fabrics.

The optimum coatings and optimum adhesives were used at Mishawaka to fabricate seams and coated fabric-to-metal laminates. The coated fabric precursors for these seams and laminates were spray applied. Long term aging studies were made on these seams and laminates in order to determine their effectiveness in resisting degradation when exposed to water and fuel at elevated temperatures.

FORWARD

The program is entitled, "Investigations of Coating Compounds and Adhesives for Self-Supporting Collapsible Fuel Storage Tanks". It was performed under Contract No. DAAG53-76-C-0140, under the cognizance of Dr. J. V. Mengenhauser of the U. S. Army Mobility Equipment Research and Development Center, Fort Belvoir, Virginia.

The DA Project Title and Project No. are "Combat Support Technology", No. 1G762708 AH67. The Task Title and Task No. are "Bulk Storage", No. FA.

The program was directed towards improving the service life of self-supporting collapsible fuel storage tanks in tropical climates, as well as, improving its handling characteristics at lower temperatures.

Polyurethane coating studies were conducted at the UNIROYAL Research Center located in Middlebury, Conn. The principle investigator there was Mr. G. W. Bernier, and the Research Manager was Dr. B. M. Murphy.

The adhesion studies were conducted at the UNIROYAL plant facility located in Mishawaka, Indiana. The principle investigator was Ms. K. K. Dyck, and the project leader was Mr. D. V. Perkins. The writers wish to acknowledge the contributions of Messrs. J. R. Kulesia and R. J. Hillebrand who were in charge of the polyurethane mixing equipment.

The Project Manager for the contract was Dr. C. T. Chmiel, and the Contract Administrator was Mr. D. E. Hile. Both are located in Mishawaka, Indiana.

References reviewed in the performance of the contract are listed in Table 1.

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Introduction

The majority of self-supporting collapsible fuel storage tanks are fabricated from polyurethane coated fabrics. The fabric is invariably nylon, and the coating is usually a polyether polyurethane on the outside and a polyester polyurethane on the inside, or next to the fabric. The polyether polyurethane possesses good hydrolytic stability, but lacks resistance to fuels and has a high permeability to fuel. The polyester polyurethane possesses good adhesion to fabric, is highly impermeable to fuel and resists attack by hydrocarbon fuels. But it suffers from susceptibility to hydrolytic degradation.

Field experience with self-supporting collapsible fuel static storage tanks shows a need for improvements in the following areas:

1. increased service life in tropical areas beyond 18 months,
2. better crease resistance of coatings upon handling at temperatures lower than -25° F,
3. increased resistance to fuel and water of adhesive bonds between coating and fabric, coating-to-coating, and coating-to-metal,
4. increased resistance of the outer coating to weathering, particularly in the presence of fuel wet spots,
5. better abrasion resistance of the outside coating.

Currently, improvement in fuel resistance is attained through use of different prepolymers and increased crosslinking. Incorporation of additives also improves the resistance to hydrolysis. But these techniques are limited by the prepolymers available on the market, and by the amount of additives that can be incorporated into a formulation without deteriorating the initial properties of the coating.

The purpose of this contract was to attempt to effect the aforementioned improvements by experimental research and development work on one-shot polyurethane coatings to replace the currently used prepolymer polyurethane coatings. In the latter approach, prepolymer polyester and/or polyether chains of about 5000 molecular weight and possessing isocyanate end groups are cured with diamines in order to achieve workable properties. Again,

the properties attainable are limited by the prepolymers available on the market.

In the one-shot polyurethane case, the precursors which would ordinarily be used in the preparation of the prepolymers are added in the presence of the curative so that chain growth and polymer chain crosslinking proceed simultaneously. More varied properties and better control of properties are expected from the wide choice of types and concentrations of polyols, chain extenders, curatives and additives available.

A concomitant purpose of this contract was to optimize the adhesion in the following interfaces: coating/fabric, coating/coating and coating/metal. The coatings were limited to one-shot polyether and one-shot polyester polyurethanes. The fabrics under investigation were glass, Dacron, Kevlar, and nylon.

The one-shot polyurethane approach, unlike the prepolymer approach, does not involve solvents. Therefore, there is no wastage of solvent, and there is minimal pollution of the atmosphere.

Also, the one-shot polyurethane system is amenable to spray coating. An objective of this contract was to spray coat fabrics using optimum one-shot polyether and one-shot polyester polyurethane formulations. This approach when applied to sleeve-type fabrics will reduce the number of seams in a fuel storage tank. Reducing seams will reduce the number of potential weak spots.

UNIROYAL's effort was directed toward accomplishing the stated objectives through a research and development program consisting of six phases as follows:

- Phase I - Review of Past Work
- Phase II - Coating Materials Evaluation
- Phase III - Adhesives Evaluation
- Phase IV - Coatings Application Evaluation
- Phase V - Aging Evaluation of Seams and Coated Fabric/
Metal Adhesive Bonds
- Phase VI - Data Analysis and Documentation

The effort included in these phases is now complete, and this report describes the work performed and the results obtained.

Investigation

A. Studies at UNIROYAL's Research Center

The effort at the Research Center was directed toward screening of polyether and polyester spines as candidates in the formulation of one-shot polyurethane coatings (Phase II). The screening resulted in the recommendation of the best polyether and polyester type polyol to use in further evaluations performed at Mishawaka.

1. Description of Polyol Spines

Ten (10) different polyol spines were selected for evaluation:

- 5 polyesters
- 3 polyethers
- 1 polyester/polyether mixture
- 1 polycaprolactone

Several of the polyols were studied at two different molecular weights, so in all there were 14 different polyurethane coatings investigated. The list is shown in Table 2, together with their chemical compositions and equivalent weights.

The polyols were converted into polyurethane films using a proprietary UNIROYAL formulation. These formulations have been coded for future reference; the code number/letter designations appear in the tables which depict the properties of the films prepared from the appropriate polyols. All of the cast films were cured 10 min. at 300° F, and then post cured 20 hours at 150° F. Tensile and low temperature stiffness properties were determined on the films, and the data is shown in Table 3. Test procedures used in this study are compiled in Appendix A.

2. Effect of Type of Curative

The properties of a polyurethane film will depend on the type of isocyanate used in curing the polyol. Therefore, the polyols in the screening phase of this contract were cured using 2 types of isocyanates, hereafter to be referred to as isocyanate A and isocyanate B. The effects of different isocyanates on polyurethane film properties are also shown in Table 3, together with the formulation codes.

3. Effect of Catalyst Level on Polyurethane Film Properties

Preliminary work at the Research Center and at Mishawaka indicated a strong independence of tensile properties on catalyst level. In order to optimize the catalyst level in the formulations, one polyester polyol (S1035-55), one polyether polyol (24-32) and one polycaprolactone (PCP-0260) were converted to films using 4 levels of catalyst concentration. At each concentration, each of these films was cured using the 2 isocyanates mentioned in A-2 above. The tensile strength, 200% modulus and elongation values for each coating initially, and after aging for 21 days at 160° F in water, are shown in Table 4, together with the formulation codes.

4. Fuel Aging of Original Films

As an additional aid in screening the polyol spines, the films were exposed to fuel aging at 160° F. The tensile retention properties of the films upon fuel aging are also shown in Table 3. The original intent was to age the films for 42 days, but the rubber workers' strike interfered with normal handling of samples. The films were aged for 63 days instead.

5. Selection of 6 Polyols for Further Evaluation

From an analysis of the data incorporated in Table 3, UNIROYAL reduced the list of polyol spine candidates to 6 for more extensive investigations. The 6 candidates are shown in Table 5.

6. Long Term Aging of Polyurethane Films

The films from the six polyols selected in A-5 were subjected to tests specified in Table 1 of the Purchase Description in Request for Quotation DAAG53-76-Q-0032, and as modified in UNIROYAL's technical proposal EP-608-76, p. 14. Original film properties and retention properties obtained after aging for up to 90 days at 160° F and for up to 28 days at 180° F are shown in Tables 6-10. As per UNIROYAL's intent described in its proposal an analysis of the data was made and a decision was reached as to which one of the 3 polyesters and which one of the other 3 polyols had performed the best after 42 days of aging. Although the water aging studies at 160° F were continued up to 90 days, the optimum polyester and polyether formulations were

transmitted immediately to personnel of the Engineered Systems R & D Section at Mishawaka, Indiana for use in their adhesion evaluations.

B. Studies at UNIROYAL's Facility at Mishawaka, Indiana

The effort at Mishawaka, Indiana was directed toward screening of adhesives for bonding coating to fabric, coating to coating and coating to metal. In addition, spray techniques were investigated for application of the one-shot polyurethane materials to fabrics. And finally, studies were made of the fuel and water aging resistances of seams and coated fabric/metal.

Screening of the adhesives for adhering one-shot polyether and polyester polyurethanes to 4 types of fabrics was done using coating formulations available at the Mishawaka facility. The work at the Research Center was not programmed to be to the point where a decision could be made as to the optimum polyester and polyether coating to use. Fabrics used were glass, Kevlar, Dacron and nylon.

Screening of adhesives for bonding coating to coating and coating to metal was done using the optimum polyether and polyester polyurethanes selected in A-6 above, and nylon fabric.

Final long term agings were done on spray coated samples of each of the four fabrics using the optimized one-shot polyether and polyester formulations selected in A-6 and the best adhesive combinations for bonding coating to fabric, coating to coating and coating to metal as determined in the screening experiments.

1. Description of Adhesives for Bonding Coating to Fabric

Three classes of adhesives were investigated: isocyanate, epoxy and polyurethane. Three separate adhesives from each class were studied, giving a total of 9 adhesives screened. The list of adhesives, their sources, and the prepared solutions for dunking of fabrics is shown in Table 11.

(a) Procedures for Application of Adhesives to Fabrics

(1) Epoxies and Isocyanates

Fabrics were dunked into the adhesive solutions,

air dried, and then heated for 5 min. at 300° F to effect cure of the adhesive.

(2) Polyurethane

Fabrics were dunked into the adhesive solutions, air dried and then heated for 2 min. at 300° F to effect cure of the adhesive.

(b) Procedure for Application of Coatings to Treated Fabrics

The treated fabrics, and gray fabrics as controls, were roll coated with one-shot polyether or polyester polyurethane dispensed from an automatic mixing machine. Before coating, the treated fabrics were equilibrated at 250° F. Immediately after coating, the fabrics were placed in an oven and heated at 250° F for 1 hour.

(c) Preparation of Peel Adhesion Samples

Peel adhesion samples were prepared by bonding similar pieces of the coated fabrics to each other using as an adhesive the one-shot polyurethane that made up the coating. The test pieces were placed in a press at 250° F for 10 min. followed by 50 min. heating in an oven at 250° F. In all, 80 polyurethane/fabric combinations were prepared (4 fabrics x 9 adhesives x 2 coatings + 4 grey controls x 2 coatings). The peel adhesion values obtained at room temperature are shown in Table 12.

(d) Selection of Adhesives for Further Evaluation

From an analysis of the data in Table 12 UNIROYAL reduced the list of coating/fabric adhesive combinations from 80 to 32. The list of adhesives that were retained for long-term aging is shown in Table 13.

(e) Long-Term Aging of Coating/Fabric Adhesive Combinations

The coating/fabric adhesive combinations shown in Table 13 were immersed in water and fuel for 42 day long-term aging at 160° F. Samples were removed after 14, 28 and 42 days aging. The peel adhesion values obtained are shown in Table 14.

2. Description of Adhesives for Bonding Coating to Coating

Three adhesives were screened for bonding one-shot polyether polyurethane to itself, and three adhesives were screened for bonding one-shot polyester polyurethane to itself.

The adhesives used are shown in Table 28. Adhesives A-1, A-2 and A-3 were used in the case of polyester polyurethane coatings whereas, B-1, A-2 and B-3 were used for the polyether polyurethane coatings.

(a) Preparation of Coated Fabric for Adhesive Screening

Nylon fabric was chosen as the base fabric for the adhesive screening trials. Samples of nylon fabric (untreated) were roller coated using the optimized polyester and polyether one-shot polyurethanes from A-6 above. The nylon fabric was pre-heated to 250° F prior to application of the coating compounds which were dispensed from an automatic mixing machine. Immediately after coating, the fabrics were placed in an oven and heated at 250° F for 1 hr.

(b) Preparation of Coating to Coating Adhesion Samples for Screening Tests

Seam peel adhesion samples were prepared from each of the polyester and polyether one-shot polyurethane coated nylon samples using the adhesives described in Table 28. The adhesives were brush coated and allowed 15-20 min. open time prior to seam fabrication. The fabricated seam samples were then cured at 290° F for 1 hour at a pressure of 90 psi. The original peels were then taken and the samples immersed for fuel and water exposure at 160° F. The results of these exposures is shown in Tables 16 and 17.

3. Description of Adhesives and Primers for Bonding Coating to Metal

Three adhesives were screened for bonding one-shot polyether polyurethane to metal, and three adhesives were screened for bonding one-shot polyester polyurethane to metal. The adhesives are the same used in B-2 above, and shown in Table 28.

(a) Preparation of Coated Fabric for Adhesive Screening

The coated fabric described in B-2a was used.

(b) Preparation of Coating to Metal Adhesion Samples for Screening Tests

Coating to metal adhesion samples were prepared from each of the polyester and polyether one-shot polyurethane coated nylon samples, using the adhesives and primers described in Table 28. The 2" x 12" x 1/8" 2024 T6 aluminum bars were prepared by degreasing, vapor blasting, and degreasing followed by application of the primers. The primers were applied by brush and allowed to dry 20 min. before being fused at 300° F for 20 min. Coating to metal adhesion samples were then prepared using each of 6 adhesive/coated fabric combinations on each of the three primed metal types for a total of 18 sample types. The adhesives were applied by brush to both fabric and metal and allowed 15-20 min. open time prior to cure. The samples were then put together and cured at 290° F for 1 hour at a pressure of 90 psi. The original peels were then taken and the samples immersed for fuel and water exposure at 160° F. The results of these exposures is shown in Tables 16 and 18.

4. Selection of Best Adhesives and Primers for Long Term Aging

Selection of the best coating to coating adhesive and adhesive/primer combinations for bonding coating to metal was made by analysis of the 42 day aging data presented in Tables 15-18.

5. Preparation of Coated Fabric Samples for Use in the Final Coating to Coating, and Coating to Metal Adhesion Study

Each of the four fabrics (glass, Kevlar, nylon and Dacron) was treated with the adhesive which was found to be specific for bonding the polyester and polyether one-shot coatings (See Table 14). The eight treated fabrics were then spray coated with either one-shot polyester or one-shot polyether polyurethane using a Binks Model 101-52212 spraying unit equipped with a Model 18 FMP spray

gun. Each fabric sample was heated to 250° F prior to spray application of the coating and immediately placed in an oven for 1 hour at 250° F after spray coating.

(a) Preparation of Coating to Coating and Coating to Metal Adhesion Samples for Final Aging

Coating to coating and coating to metal adhesion samples were prepared for the eight coated fabric types, (4 polyester-4 polyether) using the adhesives and adhesive/primer systems determined to be the best from analysis of the data in Tables 15-18. The samples were prepared using the same methods as described in 2 (b) and 3 (b) above.

(b) Aging of Final Coating to Coating and Coating to Metal Adhesion Samples

The samples prepared according to the procedures noted in 5 (a) were subjected to the schedule of testing shown in Table 2 of RFQ DAAG53-76-Q-0032 and as modified in UNIROYAL's technical proposal EP-608-76, pps. 26 and 27. The results of these aging tests are presented in Tables 19, 20, 21, 22, 23, 24, 25 and 26.

Discussion

A. Work Conducted at the UNIROYAL Research Center on Screening of Polyols

1. Curing of Polyols and Film Properties

The proposal called for screening of 5 polyethers and 5 polyester type polyols. However, preliminary experiments with casting one-shot polyurethanes from polyether polyols showed that not all of the available spines produced favorable films suitable for further work. In view of this, the 5 polyether polyurethanes originally planned for were replaced by 3 polyether polyurethanes, one polyester/polyether polyurethane mixture and one polycaprolactone. In a way, the program effort was broadened by including other types of polyol spines.

Varying the catalyst concentrations in the formulation did not affect significantly the initial physical properties of a polyester, polyether or a polycaprolactone

polyurethane coating (see Table 4). This is in variance with some preliminary work conducted at the Research Center and at Mishawaka. A more severe postcure used in the current studies may have resulted in a levelling effect. The catalyst level used in casting the films in our screening of the polyols was intermediate between the high and low values used in the catalyst concentration study.

Table 4 also shows that within the range of catalyst concentrations studied there was no significant effect of catalyst level on the physical properties of these films aged 21 days in water at 160° F. This is encouraging because, unlike some observations reported in the literature, the catalyst is not contributing to the acceleration of hydrolytic degradation in the systems under investigation.

The effects of 2 different types of isocyanates are shown in Tables 3 and 4. Isocyanate A gives rise to a stiffer film than does isocyanate B as measured by the torsional stiffness ratio. This reflects itself in lower elongations for the isocyanate A films; the 200% moduli values for the isocyanate A films also tend to be higher, although some exceptions do exist.

But isocyanate A imparts better film resistance toward fuel than isocyanate B as measured by retention of tensile strengths after aging. The overall retention of tensile strength for isocyanate A films after 63 days aging is 34% which reflects good resistance to fuels when compared with the guideline value established for this investigation of 30% minimum after 42 days aging.

In the case of water aging up to 21 days, the type of isocyanate does not affect significantly the hydrolytic stability of the polyether polyurethane, but isocyanate B has a dramatic deteriorating effect on the polycaprolactone polyurethane. For the polyester polyurethane coating, isocyanate A is slightly better than isocyanate B in imparting resistance to hydrolysis. Based on these aforementioned analyses, isocyanate A was retained as the curative for future polyurethane coating evaluations.

The data in Table 3 was used to reduce the list of polyol spine candidates from 14 to 6 for more extensive investigations. In general, the minimum film tensile

strength of 2500 psi is readily obtained from most polyols. Formulation changes can bring the tensile strength up to the minimum where deficiencies still exist.

The 200% moduli and torsional stiffness ratio values for the polyester films are significantly higher than the guideline values. It appears that the guideline maximum values for these properties will be exceeded by the polyester polyurethane coatings.

The torsional stiffness ratio values for the polyether films are rather close to the guideline values, although somewhat on the high side. However, for polyether polyol PTMG-2000 cured with isocyanate A the values are within the guideline range. On the other hand, the 200% moduli values are appreciably on the high side, comparable to that observed for the polyester polyurethane films.

The lower molecular weight spines with isocyanate A produced films possessing better tensile retention in fuel than the higher molecular weight spines.

The properties of the coatings from the 6 polyol spines chosen for future investigations are consolidated in Table 5. All of the polyols were cured with isocyanate A for best performance of the coatings.

In choosing these 6 polyols strong emphasis was placed on the films' resistances to fuel at elevated temperatures as judged by their tensile strength retentions. The isocyanate B films tended to possess 200% moduli and torsional stiffness ratios closer to the guideline values, but these films showed significantly greater deterioration upon fuel aging which ruled them out from further consideration.

Initial properties and retention values for the 6 polyols obtained after aging at elevated temperatures for up to 42 days are compared in Tables 6-10. The data in Table 6 shows that, except for PCP-0260 (a polycaprolactone), all of the polyols produce polyurethane films whose tensile strengths exceed the guideline retention value of 30% of initial after 42 days aging at 160° F in water. The polyester S1034-50 is particularly outstanding, having retained 75% of its tensile properties.

All of the polyols produce films which have initial

tensile strengths above the guideline value of 2500 psi, except for PTMG-2000, which is a polyether. None of the films meet the 500 psi maximum tensile stress value at 200% elongation. Only PCP-0260 meets the minimum ultimate elongation value of 300%.

Table 6 also shows that only half of the polyols produce films which pass the guideline retention value of 65% of initial tensile strength upon Weatherometer aging.

Table 7 shows 3 of the 6 polyurethane films passing the guideline retention value of 40% of initial tensile strength after aging for 28 days in water at 180° F. These are the same 3 which passed the Weatherometer test, and include a polyether polyol (PTMG-2000), a mixed polyether/polyester polyol G443/1014-75 and a polyester polyol (S1014-75).

Table 8 shows tensile strength retention data obtained after exposure to fuel for 42 days at 73.5° F and 160° F. At the lower temperature, the guideline value of 30% retention of tensile strength is met by PTMG-2000 (a polyether) and S1014-75 (a polyester). At the higher temperature it is met only by G443/1014-75 (a mixture of polyether and polyester polyols).

The tensile retention values in Table 8 show a dramatic drop after 14 day aging, and then a significant increase up to 28 day aging. This is indicative of a lower than optimum crosslinking density in the films. The relatively poor retention values observed in fuel is attributed to this fact.

When exposed to acidified fuel, all of the polyurethanes passed the guideline values when exposed at the lower temperature (see Table 9). Only G443-1014-75 met the guideline value at the higher temperature. The data here is complicated by a lower than optimum crosslinking density as it was with the non-acidified samples.

Comparison of Tables 8 and 9 shows that the effect of acid in fuel has no effect at low temperatures, and only a small deteriorating effect at the high temperature, if at all.

The modulus of rigidity data and torsional stiffness ratios are shown in Table 10. Only S1034-50 (a polyester)

meets the initial guideline value for torsional stiffness ratio, but it exceeds the recommended maximum after fuel extraction. The high value after extraction may be due to a lower than optimum crosslinking density in the polyurethane structure.

There are some anomalies in the torsional stiffness ratio values. For example, several of the ratios are lower at -40° F than they are at -25° F. No explanation is offered for this phenomenon at this time. The samples were conditioned the same amount of time prior to testing in order to minimize crystallinity effects.

The unwashed existent gum values in Table 10 for some of the films are relatively high and again reflect a lower than optimum crosslinking density in the polyurethane structure.

The small weight losses in the Taber Test shown in Table 10 reflect good abrasion resistances for the films under investigation. This is particularly true for films PTMG-2000, S1034-50 and PCP-0260.

2. Selection of Optimum Polyether and Polyester Polyols

From an analysis of the data presented in Tables 6-10 UNIROYAL chose S1034-50 (a polyester) and PTMG-2000 (a polyether) for future investigations to be conducted at Mishawaka. In analyzing the data, the choice of a polyester was among S1014-75, S1014-47 and S1034-50; the choice of a polyether, or other polyol than polyester, was among PTMG-2000, G443/1014-75 and PCP-0260. The polyesters S1014-75 and S1034-50, for the most part, performed about the same in aging experiments, and both showed better retention values than S1014-47. Choice of S1034-50 over S1014-75 was based then strictly on its lower stiffness values at low temperatures.

In the polyether, or other than polyester category, PCP-0260 was immediately ruled out because of its deterioration in water and fuel environments. PTMG-2000 and G443/1014-75 were about the same in all other respects, except in stiffness at low temperatures. PTMG-2000 was chosen over G443/1014-75 because of its lower stiffness values at low temperatures. The Contract Officer's Representative, Dr. J. V. Mengenhauser, was apprised of UNIROYAL's emphasis on the low temperature properties of

the candidate polyols, and he concurred on the choices that were made.

B. Work Conducted at the Mishawaka, Ind. Facility on Adhesives

1. Screening of Adhesives for Bonding of Coatings to Fabric

Screening of adhesives for bonding polyurethane coatings to fabric involved 9 adhesives, 4 fabrics and 2 polyurethanes. The adhesives studied were 3 each from the following class of compounds: epoxy, isocyanate and polyurethane. The fabrics were glass, Kevlar, Dacron and nylon, and some of their characteristics are shown in Appendix D. Two polyurethanes were made, one each from a polyether and a polyester polyol.

From Table 12 it is seen that the average adhesion values for the polyether polyurethane coating to the fabrics increase in the sequence: nylon > glass > Dacron > Kevlar. The adhesion of the fabric without any adhesive treatment is relatively high for nylon and glass. In general, the isocyanates are effective in enhancing the adhesion to Dacron and nylon, the epoxies are effective for glass, but the Kevlar fabric remains rather insensitive to the adhesive treatments tried in this study.

Table 12 also shows that the average adhesion values for the polyester polyurethane coating to fabric increases for the most part in the same sequence as for the polyether polyurethane coating. However, the difference between Dacron and Kevlar fabrics is not as great as it is in the case of the polyether polyurethane coating. The adhesion of both coatings to nylon fabric are about the same, but the adhesion of the polyester polyurethane coating is noticeably higher to glass, Kevlar and Dacron fabrics than it is for the polyether polyurethane coating.

The best performer for each adhesive class for each of the 2 polyurethane coatings are summarized in Table 13. The samples were immersed in water and fuel for 42 day long-term aging at 160° F.

Table 14 shows that long-term aging of the polyether polyurethane/fabric laminates results in a large drop in peel adhesion values after exposure for 14 days for all samples tested. But there is no significant deterioration in peel adhesion values from then on up to 42

days exposure. The 14 day deterioration may be due to a lower than optimum crosslinking density, resulting in a plasticizing action by the fuel.

In fuel aging of polyether polyurethane/fabric laminates, the best retention values (on a % basis) for glass and Kevlar fabrics are observed with a polyurethane adhesive. In the case of nylon fabric the best retention was observed with an epoxy adhesive, whereas for Dacron fabric the isocyanate and polyurethane adhesives, and no adhesive, are equally effective.

In the case of water aging of polyether polyurethane/fabric laminates, the 14 day aging results do not show the severe drop in adhesion values observed in fuel aging. Further aging through 42 days produced no significant deterioration in adhesion values.

The best adhesion retention values for each fabric to a polyether polyurethane coating were observed with the following adhesives:

Glass	- isocyanate and no adhesive
Kevlar	- epoxy
Nylon	- no adhesive
Dacron	- isocyanate

The peel adhesion data for polyester polyurethane /fabric laminates is also shown in Table 14. The drop in adhesion values after 14 day fuel aging is not as dramatic as in the case of the polyether polyurethane/fabric laminates. Continued fuel aging through 42 days produced no, or small, additional reductions in adhesion values.

The best adhesion retention values for each fabric to a polyester polyurethane coating were observed with the following adhesives:

Glass	- no adhesive
Kevlar	- no adhesive, epoxy and isocyanate
Nylon	- no adhesive, epoxy and isocyanate
Dacron	- no adhesive and epoxy

In the case of water aging, the best adhesion retention values for each fabric to a polyester polyurethane coating were observed with the following adhesives:

Glass - no adhesive and isocyanate
 Kevlar - no adhesive and epoxy
 Nylon - isocyanate
 Dacron - no adhesive and isocyanate

2. Selection of Optimum Coating/Fabric Adhesives

The list of best single adhesive for a particular polyurethane/fabric combination is the following:

	Glass	Fabric		Dacron
		Kevlar	Nylon	
1. Polyether Polyurethane	Isocyanate #3	Epoxy #2	None	Isocyanate #3
2. Polyester Polyurethane	None	Epoxy #2	Isocyanate #3	Isocyanate #2

Because of a suspected non-optimum crosslinking in the polyurethane structures, greater emphasis was placed on the retention values in water. It was felt that fuel would have a more deteriorating effect on the coatings, and therefore, the coating/fabric adhesion values in fuel would not be representative.

3. Screening of Adhesives and Adhesive/Primer Systems for Coating to Coating and Coating to Metal Bonding

Nylon fabric was chosen as the reinforcement for samples fabricated for the coating to coating and coating to metal adhesion screening study. Samples of non treated nylon fabric were roller coated with the optimized one-shot polyester and polyether polyurethane compound from the work done at Oxford. Due to the low viscosity of the optimized polyether compound, it was necessary to apply two roller coats of material per side to insure that sufficient material was present to produce a uniform bonding surface. The polyester coated nylon and the polyether were then used to fabricate coating to coating and coating to metal adhesion samples.

Three adhesives were used for bonding polyester to polyester and three adhesives were used to bond the polyether to itself. The adhesives are as follows:

for polyester A1 - moisture cured polyester
 A2 - 2-part polyester
 A3 - one-shot polyester coating

for polyether B1 - 2-part polyether
 A2 - 2-part polyester
 B3 - one-shot polyether coating

The A2 adhesive was used in forming seams from both the polyether and polyester polyurethane coated fabrics because preliminary experiments showed it to be effective for both types of coatings.

Each of the six adhesive/coated fabric combinations was evaluated against each of the aluminum metal primers: Chemlok 607 from Dayton Chemical Products, Thixon AB1244 from Whittaker Corp. and AP10 from M & T Chemical Co., yielding a total of 18 coated fabric/adhesive/primer combinations.

All samples were fabricated for testing in accordance with the test procedures as outlined in Appendix B and C of this report.

The results of the 160⁰ F water agings up to 90 days and the 160⁰ F Type II fuel aging up to 42 days are shown in Tables 15, 16, 17 and 18.

The peel adhesion data for the polyether adhesion samples is shown in Tables 15 and 16. The data in Table 15 for coating to coating adhesion shows poor initial adhesion values, none of which pass the guideline value of 25 lbs/in. These low values were obtained because the primary mode of failure was between coats of the polyether coating compound. This failure indicates that the first polyether coat reached too high a state of cure to allow proper adhesion of the second coat. The coating to coating adhesion values in water dropped off slightly but then retained a significant percentage of their original values. The order of performance for the adhesives was A2 < B2 < B1. The data for the Type II fuel aging showed a large drop in adhesion initially, however, no change in values was noted through the 42 day aging period. From the % retention figures the performance of the adhesive was A2 < B1 < B2.

The polyether coating to metal adhesion values are presented in Table 16. Analysis of the data shows that one adhesive/primer (B2/AP10) meets the guideline value of 45 lbs/in. initial adhesion. Upon 42 day water aging at 160° F, the data shows three adhesive/primer combinations passing the 90 day guideline figures of 15 lbs/in. or 30%. These combinations are AP10/B1, AP10/A2 and AP10/B2. The order of performance of the adhesives was $A2 < B1 < B2$. The order of performance of the primers was $AP10 < 1244 < 607$. Analysis of the fuel aging data shows a large drop in adhesion initially with little further loss through the 42 day aging period. None of the adhesive/primer combinations passed the 42 day guideline figures of 30 lbs/in. or 50% of initial after the 42 day aging period. The order of performance of the adhesives was $B2 < A2 < B1$. The order of performance of the primers was $607 < AP10 < 1244$.

The peel adhesion data for the polyester adhesion samples is shown in Tables 17 and 18. The data in Table 17 for coating to coating adhesion shows two of the three adhesives (A1 and A2) passing the guideline value of 25 lbs/in. The coating to coating adhesion values in water show two of the adhesives (A2 and A3) passing the 90 day guideline figure of 10 lbs or 40% with the A2 adhesives gaining in strength through the aging period. The order of performance for the adhesives in water was $A2 < A3 < A1$. Analysis of the fuel aging data shows a large drop in adhesion initially, however, there was little further loss of adhesion through the 42 day aging period. The order of performance of the adhesives was as follows: $A2 < A3 < A1$.

The polyester coating to metal adhesion data is presented in Table 16. Analysis of the data shows that two of the adhesive primer combinations (1244/A3 and AP10/A3) pass the guideline initial adhesion figure of 45 lbs/in. Upon 42 day water aging at 160° F the data shows three adhesive primer combinations 1244/A2, AP10/A2 and AP10/A3 passing the 90 day guideline figure of 15 lbs/in. or 30%. The order of overall performance of the adhesives was $A3 < A2 < A1$. The order of the overall performance of the primers was $AP10 < 1244 < 607$. Analysis of the fuel aging data shows a moderate drop in adhesion initially, however, the values are maintained throughout the 42 day period. Although none of the adhesive primer combinations passed the 42 day

guideline values of 30 lbs/in. or 50%, several of the sample types retained 15-20 lbs. of adhesion and 50-75% of their original strength. The order of performance of the adhesives was $A2 < A1 < A3$, and the order of performance of the primers was $AP10 < 607 < 1244$.

Analysis of the combined fuel and water aging data for each type of coating compound, considering the initial adhesion values, water and fuel aging data, including absolute adhesion data, % retention figures, and mode of failure, resulted in choosing the following combinations for the final long term adhesion studies:

	<u>Polyester Coating</u>	<u>Polyether Coating</u>
Coating to Coating	A2	A2
Coating to Metal	A2/AP10	A2/AP10

4. Coatings Applications Evaluation

During the coating to fabric, coating to coating and coating to metal adhesion screening studies investigations into methods of coating the one-shot polyurethane material onto fabric were investigated. With typical viscosity of the one-shot ethers being approximately 1000 cps and a typical ester approximately 2400 cps, machine coating of fabric is feasible. The reactivity of the material necessarily rules out dunk coating since a reservoir of material is required and would gel because of the relatively short pot life, i.e., <10 min. The materials will, however, lend themselves to either a flow coating or knife over roll operation.

During the period of time the screening tests were being done, work was accomplished in optimizing a method for spray applications of the one-shot urethane materials geared toward spraying of large irregular surfaces, such as might be encountered in spray coating a sleeve construction which is being considered for collapsible storage tanks.

A Binks Model 101-52212 spray unit utilizing a Model 102-2100, 18FMP spray gun (air atomization), or a model 102-1700, 43P spray gun (airless atomization) was used in the

spray application work. The 18FMP gun was evaluated using two atomization systems, internal atomization using a 9-41 nozzle and external atomization utilizing a 68PB nozzle. The criteria for evaluation of the various spray methods were porosity, coating uniformity, thickness, and overspray. The method of spray application yielding the least porosity, best uniformity, greatest thickness per pass with acceptable overspray was achieved using the 18FMP gun equipped with a 9-41 nozzle and internal atomization. The conditions established for spray application of the final coated fabric samples was as follows:

	<u>Polyester</u>	<u>Polyether</u>
Formula	101575K	101575U
Nozzel	9-41	9-41
Pot pressure cure (psi)	2	2
Pot pressure polyol (psi)	60	60
Pot pressure solvent (psi)	60	60
Line pressure-main (air) (psi)	92	90
Atomizing air pressure (psi)	70	60
Pump pressure (psi)	20	20
Line pressure cure (psi)	200	100
Line pressure polyol (psi)	200	400
Line temperature ° F	160	160
Polyol heater temperature ° F	160	160
Cure heater temperature ° F	75	75
Polyol pot temperature ° F	160	160
Cure pot temperature ° F	75	75

Both the polyester and polyether coated fabrics sprayed under the above conditions showed some coating porosity. It was necessary to modify the catalyst system for spray application, to minimize the sagging of the sprayed coating when applied to a vertical surface. The resultant compounds possessed a gel time of approximately 20 secs. The modification involved the changing of the type of catalyst rather than the level because gel times of the order desired were not feasible with the original catalyst system.

5. Final Long Term Aging Study of Coating to Coating and Coating to Metal Bonds

Coated fabric samples were prepared for final coating to coating (seam), coating to metal and dead load shear testing.

The coated fabric samples were prepared by spray coating, using the method described in B. 5., page 8.

One set of glass, Kevlar, nylon and Dacron fabrics were spray coated with the optimized one-shot polyether polyurethane and one set spray coated with the optimized polyester polyurethane.

Prior to spray application of the coatings the fabrics were given the optimum treatment specific for the polyester or polyether coating as determined in the coating to coating adhesion study as outlined in B. 2., page 16.

Coating to coating, coating to metal and dead weight load adhesion samples were prepared for each set of polyester and polyether urethane coated fabrics using the A2 adhesive and AP10 metal primer determined as optimum systems in the adhesive and primer screening evaluation.

Samples were made in sufficient quantity to perform the tests as outlined in Table 2 of RFQ DAAG-53-76-Q-0032 and as modified in UNIROYAL's technical proposal EP-608-76, pps. 26 and 27.

Table 19 shows the 160° F fuel and water aging data for polyether coating to coating and coating to metal adhesion samples. As can be seen from the fuel data, very poor initial peel values and after aging data exists. The modification to the catalyst system to shorten the gel time is believed to be responsible for this very poor performance of the polyether compound in fuel. Polyether samples made for the 180° F water aging, 73.5° F Type II fuel aging and dead weight load testing were made using the polyether compound containing a different catalyst system in an attempt to improve the original and fuel aged properties. In addition, the 42 day 160° F fuel samples were remade. This data is presented in Table 26. Comparison of the 160° F water aging and the 180° F water aging also shows that the catalyst type affected the initial and aged properties of the polyester compound. The poor performance of the 160° F fuel and water coating to metal samples shown in Table 20 also appears to have been caused by the catalyst modification. Examination of the failure mode for all samples made for the 160° F aging study indicates that not only the polyether compound was affected, but also the performance of the A2 adhesive, since failure within

the A2 adhesive was not characteristic during the screening trials.

Analysis of the more representative "Remake" 160⁰ F fuel aging data presented in Table 26 shows that none of the samples pass the guideline figure of 25 lbs/in. for original peel or the 15 lb/in. - 50% figure for 42 days, however, both the nylon and Kevlar samples retained 46% of their original 13 lbs/in. after the 42 day aging period.

The analysis of the 180⁰ F water aging data for the polyether coated fabrics shown in Table 23 shows none of the samples passing the guideline figure of 25 lbs/in. for original peel with the polyether glass sample the highest at 22 lbs/in. The adhesion for the glass, Kevlar and nylon gradually dropped through the 28 day aging period, however, the % retention at 28 days was relatively high at 36% for glass, 47% for Kevlar and 27% for nylon. The Dacron sample increased in adhesion from an initial of 10 lbs/in. to 23 lbs/in. after 7 days and gradually decreased to a value of 7 lbs/in. or 70% retention.

Analysis of the 73.5⁰ F fuel aging data for the polyether coated fabrics presented in Table 24 shows initial peel adhesion of all sample types to be below the 25 lbs/in. guideline figure and a large initial drop in adhesion after 14 days followed by little further degradation through the remainder of the 42 day aging period.

The results of the dead weight load testing of the one-shot polyether coated fabrics is presented in Table 27. The glass, Kevlar and nylon samples passed the guideline requirement for coating to coating and coating to metal bonds. The Dacron sample failed to meet the coating to coating and coating to metal requirements. Failure of the Dacron samples appears to be due to the relatively low coating to fabric adhesion exhibited by the polyether/Dacron samples.

The coating to coating adhesion data for one-shot polyester polyurethane coated fabrics subjected to water and fuel aging at 160⁰ F is presented in Table 21. Analysis of the data shows both the polyester coated nylon and polyester Dacron passed the guideline value of 25 lbs/in. for original peel adhesion. The water aging data

shows a gradual decline of adhesion values for all sample types to 70 days with complete failure of all samples at 90 days, with the mode of failure being primarily 100% coating to fabric for all sample types. The 160° F fuel aging data shows a sharp drop in adhesion values for the polyester coated glass and polyester coated Dacron with the polyester coated glass maintaining its adhesion level through the 42 day period and the polyester coated Dacron sample showing a gradual drop in adhesion through the 42 day period. The polyester coated Kevlar and polyester coated nylon samples, however, show good retention up to 28 days with the polyester Kevlar dropping off at 42 days and the polyester nylon sample maintaining a high level of adhesion to 42 days and passing the guideline figure of 15 lbs/in. or 50%.

The coating to metal adhesion data for one-shot polyester coated fabric samples is presented in Table 22. All original adhesion values, although relatively high, failed to meet the guideline figure of 45 lbs/in. except the polyester nylon samples, which did pass the requirement. Upon aging at 160° F in water all samples show relatively good absolute values at 14 days, however, from 14 to 70 days exposure they show a steady decline with the 90 day exposure resulting in complete failure of all samples.

Fuel aging of the polyester coating to metal samples shows an initial drop for all but the Kevlar sample after 14 days then a moderate decline in all adhesion values except those for the polyester coated Kevlar. The polyester coated Kevlar value at 42 days was 6 lbs/in. higher than the original peel adhesion value. None of the polyester coating to metal adhesion samples passed the guideline values of 30 lbs/in. or 50% for 42 day 160° F fuel aging.

Examination of the 180° F water aging data for the one-shot polyester coating to coating adhesion shows the glass and Dacron samples pass the guideline values for original peel adhesion. Upon aging all samples show a steady decline up to the 28 day testing. Values for the glass and Dacron samples at 28 days are 3 lbs/in. and 1 lb/in., and the Kevlar nylon samples show a complete loss of bond.

The fuel aging data for the coating to coating adhesion samples made from one-shot polyester coated fabrics is shown in Table 24. The data shows that good initial adhesion values were obtained with the Kevlar and Dacron

samples passing the guideline figure of 25 lbs/in. for initial adhesion. Good adhesion was maintained by all the samples throughout the 42 day aging period with all samples passing the guideline adhesion figure of 15 lbs/in. or 50% for the 42 day aging.

The dead weight load test data for the coating to coating and metal to coated fabric bonds made using the one-shot polyester coated fabrics is shown in Table 27. The glass, Kevlar and nylon all passed the seam test with the Dacron sample failing. The coating to metal tests showed the Kevlar and Dacron passing and the glass and nylon failing. This leaves the one-shot polyester Kevlar as being the only sample set passing both the coating to coating and coating to metal tests.

pH measurements of the water in which the polyester and polyether one-shot adhesion samples were aged are not being reported because the samples were inadvertently replaced into different jars after removal for testing.

Conclusions

1. The polyether polyol, PTMG-2000 (a polytetramethylene glycol) and the polyester polyol, SI034-50 (a 1,6 hexandiol azelate), produce the best polyurethane properties of the 14 polyols screened. In choosing the optimum polyols, emphasis was placed on the low temperature flex properties of the films.
2. Tensile properties of the one-shot polyurethane films are not affected by moderate changes in catalyst level; however, aging and adhesion characteristics seem to be affected by the type of catalyst employed.
3. Attaining the guideline low temperature properties of coating compounds and simultaneously obtaining the guideline fuel and water resistances is not possible using current commercially available polyols and isocyanates. In particular, the stiffness of the one-shot polyurethane coatings tend to be higher than the guideline values.
4. Aging properties of polyurethane coatings in this study were strongly dependent upon the isocyanate used in curing the polyol.
5. Acid in fuel does not have a significant effect on the retention properties of coatings upon aging.

6. The best performing adhesives for bonding one-shot polyester polyurethanes and one-shot polyether polyurethanes to four fabric types were determined from initial screening and fuel/water aging studies.
7. A 2-part polyester adhesive proved to be optimal in bonding one-shot polyester polyurethane coated fabric to itself and one-shot polyether polyurethane coated fabric to itself.
8. A 2-part polyester adhesive/metal primer combination proved to be optimal in bonding one-shot polyester polyurethane coated fabric to metal and one-shot polyether polyurethane coated fabric to metal.
9. In the long term aging studies the one-shot polyether polyurethane showed better adhesive bond performance in water, and the one-shot polyester polyurethane showed better adhesive bond performance upon fuel aging.
10. In the long term aging studies in fuel and water none of the coating to coating or coating to metal adhesion samples passed all of the guideline values.
11. Seams prepared from Kevlar and Dacron polyester polyurethane coated fabrics using the 2-part polyester adhesive passed the guideline values for original peel adhesion. Glass and nylon seam samples were deficient. However, all of the polyester polyurethane seam samples passed the guideline values for fuel resistance when aged 42 days at 73.5° F.
12. It is believed that the formulating done for the attainment of the desired low temperature properties produced compounds with crosslink densities much lower than the optimum needed to impart a high degree of hydrolysis resistance and resistance to swelling by aromatic and aliphatic hydrocarbons.
13. A method was developed for spray coating fabric substrates with one-shot polyurethane materials yielding a coated fabric. Microscopic examination showed the presence of some porosity in the coating.

Recommendations

1. In order to improve coating properties, studies should be made to increase crosslinking density of the polyurethane chains. This may compromise low temperature properties but should lead to increased resistance to fuel and water degradation.
2. In view of recommendation 1., emphasis in future studies should be concentrated on increasing long term aging properties even to the acceptance of non-optimal low temperature properties.
3. Isocyanate A should be used in the curing of the polyols in order to obtain good properties.
4. The anomalies in the torsional stiffness ratios suggest transitions occurring as a function of temperature. In screening several coatings for low temperature performance, a curve should be generated rather than 2 points taken of stiffness properties as a function of temperature.
5. The tensile properties of the coatings were not sensitive to changes in concentration of catalyst investigated, but results showed perceptible affects due to the type of catalyst used. Therefore, future studies should incorporate additional catalyst type/concentration studies.
6. Coating to fabric adhesion was optimal in many cases after treatment of the fabric with isocyanate or epoxy. Future studies should incorporate the effect of fabric treatment on tear and penetration properties of the coated fabric.
7. Future studies on spraying of one-shot polyurethanes should be conducted at various relative humidities to determine effect of moisture level in the air on porosity in films.

Table 1

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Table 2

Description of Polyols

<u>Polyol Type</u>	<u>Chemical Composition</u>	<u>Equivalent Weight</u>
A. Polyester		
1. Hooker S1014-47	1,6 hexanediol adipate/phthalate	1194
2. Hooker S1014-75	1,6 hexanediol adipate/phthalate	748
3. Hooker S1019-55	1,6 hexanediol adipate/phthalate (different ratio from 1 & 2)	1020
4. Hooker S1035-55	1,6 hexanediol azelate/phthalate	1020
5. Hooker S1035-110	1,6 hexanediol azelate/phthalate	510
6. Hooker S1034-50	1,6 hexanediol azelate	1122
7. Hooker S103-55	Mixed ethylene/propylene glycol adipate	1020
B. Polyether		
1. Niax 24-32 (Union Carbide)	Styrene/acrylonitrile graft on polypropylene glycol	-
2. Niax PPG-2025	Polypropylene glycol	2025
3. Polymeg PTMG-1000 (Quaker Oats)	Polytetramethylene glycol	1000

Table 2 (Continued)

<u>Polyol Type</u>	<u>Chemical Composition</u>	<u>Equivalent Weight</u>
4. Polymeg PTMG-2000	Polytetramethylene glycol	2000
5. Olive Poly G 443/Hooker 1014-75	Blend of ethylene oxide capped PPG and polyester	462
C. Polycaprolactone		
1. Niox PCP-0230	Diol terminated caprolactone	634
2. Niox PCP-0260	Diol terminated caprolactone	1496

TABLE 3

POLYESTERS

POLYOL	SI035-55		SI035-110		SI014-47		SI014-75		SI019-55		SI034-50		SI03-55	
	A	B	A	B	A	B	A	B	A	B	A	B	A	B
Isocyanate	4895	5720	7518	6108	4406	4867	5408	5340	4368	4671	3207	4214	2930	5004
Tensile Strength (2500 psi minimum)	2293	1374	--	3642	1152	831	4526	2012	1468	970	1253	812	923	1702
200% Modulus (500 psi maximum)	257	340	27	360	297	423	213	307	267	400	290	403	313	540
Elongation (30% minimum)	163	32	Stiff	Stiff	276	62	224	47.8	245	30	87.4	10.7	189	24
Torsional Stiffness Ratio	265	67	Stiff	Stiff	297	147	240	54.7	308	70	126.7	31.2	420	68.8
-25°F (5 maximum)														
-40°F (10 maximum)														
63 Days, 160°F Medium #5 Fuel	1298	423	2712	1326	1467	*	1959	208	1302	*	946	160	*	*
Tensile Strength	26.5	7.5	36.1	21.7	33.3		36.2	3.9	29.8		29.5	3.8		
% Retention	317	483	225	390	382		357	288	335		303	330		
Elongation	123	142	833	108	129		168	94	125		104	82		
% Retention	G	H	I	J	A	B	C	D	E	F	K	L	M	N
Code 101575														

* Samples too degraded to test.

TABLE 3 (Continued)

POLYETHERS

POLYOL	PPG-2025		PTMG-1000		PTMG-2000		24-32
	A	B	A	B	A	B	
Isocyanate							A
Tensile Strength (2500 psi minimum)	2375	5141	2442	3845	2159		
200% Modulus (500 psi maximum)	1879	-- 2975	1367	819	1514		
Elongation (300% minimum)	280	150 253	287	483	283		
Torsional Stiffness Ratio -25°F (5 maximum) -60°F (10 maximum)	9.2 36	15.9 11 18.3 14	3.7 6.9	6.2 8.9	10.6 24.4		
63 Days, 160°F Medium #5 Fuel							
Tensile Strength	Fuel evaporated during run	1058 1216	993	Fuel evaporated during run	807		
% Retention	" "	31 23.6	40.7	" "	37.2		
Elongation	" "	176 347	307	" "	243		
% Retention	" "	117 137	107	" "	86		
Code 101575	Q	S T	U	V	0		

TABLE 3 (Continued)

POLYESTER/POLYETHER MIXTURE AND POLYCAPROLACTONE

POLYOL	G443/1014-75**		PCP-0230		PCP-0260	
	A	B	A	B	A	B
Isocyanate						
Tensile Strength (2500 psi minimum)	3367	1194	4871	5548	3768	4982
2002 Modulus (500 psi maximum)	1650	1129	3811	1610	530	533
Elongation (300% minimum)	253	317	217	343	370	533
Torsional Stiffness Ratio						
-25°F (5 maximum)	202.3	18.6	154	49	20.7	4.0
-40°F (10 maximum)	221.1	41.9	163	78	24.2	12.0
63 Days, 160°F Medium #5 Fuel						
Tensile Strength	No fuel aging	No fuel aging	1267	562	1846	*
Z Retention			26	10	49	
Elongation			252	245	633	
Z Retention			116	71	171	
Code 101575	W	X	Y	Z	AA	BB

* Samples too degraded to test.

** First samples were inadequate because of poor raw materials. Second samples were prepared too late for aging.

Table 4

S1035-55/Isocyanate A

<u>Catalyst Level</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
<u>21 Days R.T.</u>				
Tensile Strength	4385	4537	4270	4469
200% Modulus	2294	2049	2218	2084
Elongation	245	270	255	250
<u>21 days 160°F H₂O</u>				
Tensile Strength	4643	4230	3832	4265
200% Modulus	1514	1511	1439	1345
Elongation	310	305	300	320
<u>Percent Retention</u>				
Tensile Strength	105	93	90	91
200% Modulus	66	74	64	65
Elongation	126	112	117	128
Code 101576	A	B	C	D

TABLE 4 (Continued)
S1035-55/Isocyanate B

<u>Catalyst Level</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
<u>21 Days R.T.</u>				
Tensile Strength	4677	4166	4345	3778
200% Modulus	1298	1346	1354	1215
Elongation	385	375	370	370
<u>21 days 160°F H2O</u>				
Tensile Strength	2746	3491	4033	3207
200% Modulus	888	909	949	1004
Elongation	545	454	450	445
<u>Percent Retention</u>				
Tensile Strength	59	84	93	85
200% Modulus	68	68	70	83
Elongation	142	121	121	120
Code 101576	E	F	G	H

Table 4 (Continued)

24-32/Isocyanate A

<u>Catalyst Level</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
<u>21 days R.T.</u>				
Tensile Strength	1675	1692	2019	1703
200% Modulus	1621	1570	1254	1185
Elongation	200	220	267	237
<u>21 days 160°F H₂O</u>				
Tensile Strength	1383	1838	1941	1701
200% Modulus	926	1161	925	862
Elongation	237	253	267	280
<u>Percent Retention</u>				
Tensile Strength	86	108	96	100
200% Modulus	57	74	73	73
Elongation	118	115	100	118
Code 101576	I	J	K	L

Table 4 (Continued)

24-32/Isocyanate B

<u>Catalyst Level</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
<u>21 days R.T.</u>				
Tensile Strength	1242	1353	1622	1513
200% Modulus	916	950	988	931
Elongation	500	440	437	470
 <u>21 days 160°F H₂O</u>				
Tensile strength	1120	1351	1247	1475
200% Modulus	828	864	851	896
Elongation	440	463	417	425
 <u>Percent Retention</u>				
Tensile Strength	90	100	77	98
200% Modulus	90	91	86	96
Elongation	88	105	95	90
Code 101576	M	N	O	P

Table 4 (Continued)

PCP-0260/Isocyanate A

<u>Catalyst Level</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
<u>21 days R.T.</u>				
Tensile Strength	3157	3533	3414	3575
200% Modulus	602	540	564	531
Elongation	365	380	370	410
<u>21 days 160°F H₂O</u>				
Tensile Strength	3487	3654	2972	3624
200% Modulus	418	393	443	377
Elongation	455	470	425	505
<u>Percent Retention</u>				
Tensile Strength	110	103	87	101
200% Modulus	69	67	79	71
Elongation	125	123	115	123
Code 101576	Q	R	S	T

Table 4 (Continued)

PCP-0260/Isocyanate B

<u>Catalyst Level</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
<u>21 days R.T.</u>				
Tensile Strength	4577	4595	4624	4602
200% Modulus	605	584	572	554
Elongation	480	510	525	490
 <u>21 days 160°F H₂O</u>				
Tensile Strength	1136	1119	1130	1207
200% Modulus	-	-	-	-
Elongation	50	45	35	45
 <u>Percent Retention</u>				
Tensile Strength	25	24	24	26
200% Modulus	-	-	-	-
Elongation	10	9	7	9
Code 101576	U	V	W	X

Table 5

POLYOLS CHOSEN FOR LONG-TERM AGING

POLYOL	S1014-47 A	S1014-75 A	S1034-50 A	PTMG-2000 A	G443/1014-7 A	PCP-0260 A
Isocyanate						
Tensile Strength (2500 psi minimum)	4406	5408	3207	2442	3367	3768
200% Modulus (500 psi maximum)	1152	4526	1253	1367	1650	530
Elongation (300% minimum)	297	213	290	287	253	370
Torsional Stiffness Ratio -25°F (5 maximum) -40°F (10 maximum)	276 297	224 240	87.4 126.7	3.7 6.9	202.3 221.1	20.7 24.2
63 Days, 160°F Medium #5 Fuel						
Tensile Strength	1467	1959	946	993	No fuel aging	1846
% Retention	33.3	36.2	29.5	40.7	" "	49
Elongation	382	357	303	307	" "	633
% Retention	129	168	104	107	" "	171
Code 101575	A	C	K	U	W	AA

TABLE 6

TENSILE STRENGTH RETENTION OF POLYURETHANE
COATINGS AFTER EXPOSURE TO WATER AT 160° F,
AND UPON WEATHEROMETER AGING

	PTMG- 2000	G443/ 1014- 75	S1014- 75	S1014- 47	S1034- 50	PCP- 0260
<u>Initial</u>						
Tensile Strength (psi)	2153	4462	5738	5824	4135	4376
200% Modulus (psi)	1775	-	5194	2272	2228	581
Elongation (%)	225	170	210	280	260	367
<u>Days Aging</u>						
<u>14 Days</u>						
Tensile Strength Retention (%)	56	60	68	47	85	63
Elongation Retention (%)	118	172	119	134	128	144
pH	4.30	4.00	5.30	4.45	4.25	4.52
<u>28 Days</u>						
Tensile Strength Retention (%)	68	69	62	47	76	46
Elongation Retention (%)	136	190	133	145	140	171
pH	4.90	3.98	4.30	4.40	4.30	4.82
<u>42 Days</u>						
Tensile Strength Retention (%)	62	69	62	43	75	18
Elongation (%)	130	190	137	154	155	22
pH	6.16	4.26	4.48	6.29	5.27	5.88
<u>70 Days</u>						
Tensile Strength Retention (%)	68	56	46	15	49	Dec.
Elongation Retention (%)	137	199	151	158	176	Dec.
pH	5.91	4.34	4.62	5.30	4.82	3.94
<u>90 Days</u>						
Tensile Strength Retention (%)	65	43	30	6	23	Dec.
Elongation Retention (%)	136	190	130	58	135	Dec.
pH	5.32	4.99	5.41	4.7	5.41	3.64
Code 101575	U	W	C	A	K	AA

TABLE 6 (Cont'd.)

WEATHEROMETER AGING

	PTMG 2000	G443/ 1014- 75	S1014- 75	S1014- 47	S1034- 50	PCP- 0260
<u>250 Hrs.</u>						
Tensile Strength Retention (%)	85	70	64	59	75	70
Elongation Retention (%)	93	94	87	87	86	94
<u>500 Hrs.</u>						
Tensile Strength Retention (%)	71	71	68	57	59	54
Elongation Retention (%)	87	79	81	92	83	89
<u>750 Hrs.</u>						
Tensile Strength Retention (%)	57	59	65	37	61	44
Elongation Retention (%)	82	98	78	78	80	71
Code 101575	U	W	C	A	K	AA

Table 7

Tensile Strength Retention of Polyurethane
Coatings After Exposure to Water at 180° F

	Polyols					
	PTMG- 2000	G443/ 1014- 75	S1014- 75	S1014- 47	S1034- 50	PCP- 0260
<u>Initial</u>						
Tensile Strength (psi)	2153	4462	5738	5824	4135	4376
200% Modulus (psi)	1775	-	5194	2272	2228	581
Elongation (%)	225	170	210	280	260	367
<u>Days Aging</u>						
<u>7 Days</u>						
Tensile Strength Retention (%)	79.2	54.6	74	54.1	84.2	82
Elongation Retention (%)	112	145	109	117	122	133
<u>14 Days</u>						
Tensile Strength Retention (%)	112.5	52.4	58.9	41.8	60.4	50.9
Elongation Retention (%)	136	149	111	115	116	158
<u>21 Days</u>						
Tensile Strength Retention (%)	92	53	51.9	33	47	21
Elongation Retention (%)	127	133	109	128	125	9.5
<u>28 Days</u>						
Tensile Strength Retention (%)	82	41	87*	16	29	0
Elongation Retention (%)	159	160	115	140	136	0
Code 101575	U	W	C	A	K	AA

*Inconsistent with other data - extrapolated value is about 46.

Table 8

Tensile Strength Retention of Polyurethane
Coatings After Exposure to #5 Fuel at 73.5° F and 160° F

	Polyols					
	PTMG- 2000	G443/ 1014- 75	S1014- 75	S1014- 47	S1034- 50	PCP- 0260
<u>Initial</u>						
Tensile Strength (psi)	2153	4462	5738	5824	4135	4376
200% Modulus (psi)	1775	-	5194	2272	2228	581
Elongation (%)	225	170	210	280	260	367
<u>Days Aging (73.5° F)</u>						
<u>14 Days</u>						
Tensile Strength Retention (%)	27	14	19	12	15	10
Elongation Retention (%)	112	81	63	68	66	44
<u>28 Days</u>						
Tensile Strength Retention (%)	40	18	27	12	21	21
Elongation Retention (%)	83	86	102	54	60	70
<u>42 Days</u>						
Tensile Strength Retention (%)	36	22	31	17	17	16
Elongation Retention (%)	71	91	105	73	54	61
<u>Days Aging (160° F)</u>						
<u>14 Days</u>						
Tensile Strength Retention (%)	25	-	29	21	16	14
Elongation Retention (%)	84	-	119	122	96	97
<u>28 Days</u>						
Tensile Strength Retention (%)	29	31	25	21	35	23
Elongation Retention (%)	103	155	127	140	134	132
<u>42 Days</u>						
Tensile Strength Retention (%)	21	34	16	18	18	0
Elongation Retention (%)	205	115	113	162	107	0
Code 101575	U	W	C	A	K	AA

Table 9

Tensile Strength Retention of Polyurethane Coatings
After Exposure to Acidified #5 Fuel at 73.5° F and 160° F

	Polyols					
	PTMG- 2000	G443/ 1014- 75	S1014- 75	S1014- 47	S1034- 50	PCP- 0260
<u>Initial</u>						
Tensile Strength (psi)	2153	4462	5738	5824	4135	4376
200% Modulus (psi)	1775	-	5194	2272	2228	581
Elongation (%)	225	170	210	280	260	367
<u>Days Aging (73.5° F)</u>						
<u>14 Days</u>						
Tensile Strength Retention (%)	18	12	12	13	17	12
Elongation Retention (%)	79	73	78	82	58	58
<u>28 Days</u>						
Tensile Strength Retention (%)	38	31	29	24	29	19
Elongation Retention (%)	88	138	119	113	88	80
<u>42 Days</u>						
Tensile Strength Retention (%)	35	40	25	21	27	25
Elongation Retention (%)	84	134	98	95	85	89
<u>Days Aging (160° F)</u>						
<u>14 Days</u>						
Tensile Strength Retention (%)	17	31	23	11	16	11
Elongation Retention (%)	109	215	203	118	132	147
<u>28 Days</u>						
Tensile Strength Retention (%)	27	38	31	22	33	6
Elongation Retention (%)	129	229	217	194	168	74
<u>42 Days</u>						
Tensile Strength Retention (%)	18	27	16	8	16	0
Elongation Retention (%)	106	211	142	123	129	0
Code 101575	U	W	C	A	K	AA

Table 10

Stiffness, Abrasion and Fuel Extraction
Properties of Polyurethane Coatings

	<u>Polyols</u>					
	<u>PTMG- 2000</u>	<u>G443/ 1014- 75</u>	<u>S1014- 75</u>	<u>S1014- 47</u>	<u>S1034- 50</u>	<u>PCP- 0260</u>
<u>Modulus of Rigidity</u>						
<u>Initial</u>						
Room Temp.	262.8	327.5	2478.9	231.6	218.1	185.5
-25° F	1164.8	77693.2	85307.4	66422.7	24219.2	1194.8
-40° F	2530.3	89311.7	107715.3	78522.0	66945.2	27412.3
<u>After Fuel Extraction</u>						
Room Temp.	284.9	211.7	268.3	218.7	131.2	155.4
-25° F	978.8	46384.8	77182.5	39148.8	9699.9	583.3
<u>Torsional Stiffness Ratio</u>						
<u>Initial</u>						
-25° F	39.37	95.46	37.24	40.42	2.73	24.28
-40° F	2.74	112.86	41.45	52.98	4.15	1.56
<u>After Fuel Extraction</u>						
-25° F	86.54	39.74	25.37	42.01	14.92	1.29
-40° F	3.12	39.13	38.58	43.29	34.60	1.31
<u>Taber Abrasion</u>						
<u>H-18 Wheel, 1,000 revolutions, 1000 g</u>						
Weight loss (g)	0.0215	0.3433	0.4061	0.1114	0.0779	0.0404
<u>Fuel Contamination</u>						
Unwashed existent Gum (mg/100 ml)	47.7	2.8	4.8	13.1	28.6	40.2
Heptane washed existent gum (mg/100 ml)	5.56	0.40	0.82	1.44	2.56	7.52
Code 101575	U	W	C	A	K	AA

Table 11

Adhesives for Bonding Polyurethane
Coatings to Fabrics

A. Epoxies

1	75 parts	Epon 828 (Shell Chemical Co.)
	73 "	Vibrathane B600 (UNIROYAL, Inc.)
	52 "	Methylene Dianiline - MDA (Dow Chemical Co.)
	9800 "	Methyl Ethyl Ketone - MEK
2	10 "	Epon 828
	10 "	Versamid 140 (General Mills Co.)
	980 "	MEK
3	156 "	DEN 438 (Dow Chemical Co.)
	44 "	MDA
	9800 "	MEK

B. Isocyanates

1	20 "	Mondur CB75 (Naftone Co.)
	980 "	MEK
2	20 "	PAPI (Upjohn Co.)
	980 "	MEK
3	20 "	143L (Upjohn Co.)
	980 "	MEK

C. Polyurethanes

1	180 "	Permuthane U-23-006 (Permuthane Co.)
	5 "	" U-40-752
	357 "	Isopropyl Alcohol } Mixed
	357 "	Toluene } first
2	129 "	Permuthane U-23-171
	5 "	" U-40-752
	766 "	Acetone
3	46 "	Vibrathane 6007 (UNIROYAL, Inc.)
	1 "	Staboxal #1 (Naftone Co.)
	950 "	MEK
	3 "	Tonox (UNIROYAL, Inc.)

Table 12

A. Original Pulls in lbs/in on Polyether
Polyurethane Coated Samples

<u>Adhesives</u>	<u>Glass</u>	<u>Kevlar</u>	<u>Fabrics</u>	<u>Dacron</u>
			<u>Nylon</u>	
Epoxy #1	22,18	8,9	36,44	14,13
#2	28,27	13,17	36,24	15,16
#3	15,15	9,15	36,40	11,12
Isocyanate #1	19,19	10,8	54,51	15,12
#2	18,19	8,8	54,50	17,12
#3	20,18	10,14	52,48	17,18
Polyurethane #1	19,24	13,12	26,30	11,12
#2	19,22	12,10	43,40	16,16
#3	20,20	11,12	53,47	13,14
Control (no adhesive)	21,21	12,10	43,43	10,11

B. Original Pulls in lbs/in on Polyester
Polyurethane Coated Fabrics

Epoxy #1	24,24	13,16	52,53	16,20
#2	32,36	18,15	38,35	15,17
#3	36,29	16,16	50,50	12,13
Isocyanate #1	32,27	21,16	43,40	20,20
#2	30,43	19,17	29,30	34,33
#3	26	17,17	42,48	21,23
Polyurethane #1	26,25	15,14	34,38	25,20
#2	29,25	14,17	21,25	17,17
#3	15,8	16,16	24	20,23
Control	17,30	14,14	60,32	12,17

Table 13

Fabric/Coating Adhesives Retained
for Long-Term Aging

	<u>Glass</u>	<u>Kevlar</u>	<u>Nylon</u>	<u>Dacron</u>
1. Polyether	Epoxy #2 Isocyanate #3 Polyurethane #1 Control	Epoxy #2 Isocyanate #3 Polyurethane #1 Control	Epoxy #1 Isocyanate #1 Polyurethane #3 Control	Epoxy #2 Isocyanate #3 Polyurethane #2 Control
2. Polyester	Epoxy #2 Isocyanate #2 Polyurethane #2 Control	Epoxy #2 Isocyanate #1 Polyurethane #3 Control	Epoxy #1 Isocyanate #3 Polyurethane #1 Control	Epoxy #1 Isocyanate #2 Polyurethane #1 Control

Table 14

Adhesives Evaluation in Bonding
Polyether Polyurethane to Fabrics

Adhesive	Initial Peel Adhesion Lbs/in	Fuel			Water				
		14 Days Aging Lbs/in	% Retention	28 Days Aging Lbs/in	42 Days Aging Lbs/in	% Retention	28 Days Aging Lbs/in	42 Days Aging Lbs/in	% Retention
1. Glass Fabric									
None	21	4	19	5	24	6	10	48	11
Epoxy #2	28	6	21	6	21	8	9	32	10
Isocyanate #3	19	3	16	4	21	4	10	53	10
Polyurethane #1	22	8	36	9	41	10	6	27	8
2. Kevlar Fabric									
None	11	2	18	4	36	4	6	54	7
Epoxy #2	15	4	27	6	40	7	8	53	11
Isocyanate #3	12	2	17	3	25	4	8	67	8
Polyurethane #1	12	8	67	8	67	7	6	50	6
3. Nylon Fabric									
None	43	4	9	6	14	4	24	56	20
Epoxy #1	40	8	20	11	28	10	14	35	12
Isocyanate #1	52	8	15	10	19	10	16	31	17
Polyurethane #3	50	6	12	7	14	7	15	30	15
4. Dacron Fabric									
None	10	3	30	4	40	4	9	90	9
Epoxy #2	16	6	38	7	44	5	12	75	14
Isocyanate #3	18	6	33	7	39	7	18	89	18
Polyurethane #2	16	6	38	6	38	7	15	125	14

Table 14 (Cont'd.)
Adhesives Evaluation in Bonding
Polyester Polyurethane to Fabrics

Adhesive	Initial Peel Adhesion Lbs/in	Fuel				Water			
		14 Days		28 Days		14 Days		28 Days	
		Aging Lbs/in	% Retention	Aging Lbs/in	% Retention	Aging Lbs/in	% Retention	Aging Lbs/in	% Retention
1. Glass Fabric									
None	24	10	42	12	50	14	58	14	58
Epoxy #2	34	11	32	9	26	11	32	11	32
Isocyanate #2	36	10	28	11	31	14	39	14	39
Polyurethane #2	27	14	52	9	33	16	59	12	44
2. Kevlar Fabric									
None	14	9	64	8	57	8	57	6	43
Epoxy #2	17	9	53	8	47	9	53	8	47
Isocyanate #1	18	9	50	9	50	8	44	8	44
Polyurethane #3	16	8	50	8	50	8	50	6	38
3. Nylon Fabric									
None	46	11	24	9	20	22	48	24	52
Epoxy #1	52	19	36	12	23	25	48	28	54
Isocyanate #3	45	14	31	9	20	33	73	35	78
Polyurethane #1	36	17	47	11	31	22	61	15	42
4. Dacron Fabric									
None	14	8	57	8	57	12	86	14	100
Epoxy #1	18	8	44	8	44	12	67	11	61
Isocyanate #2	34	14	41	10	29	25	74	34	100
Polyurethane #1	22	8	36	6	27	13	59	14	64

TABLE 15
ADHESIVES EVALUATION IN BONDING
POLYETHER POLYURETHANE COATING TO ITSELF

Water at 160° F										
Adhesive	Original lbs/in	Failure* Mode	14 Days lbs/in.	% Retention	Failure Mode	28 Days lbs/in.	% Retention	Failure Mode	42 Days lbs/in.	Failure Mode
B-1	9	100, b-c	6	67	50/50 b-c/a	6	67	100, a	4	100, a
A2	10	100, b-c	8	80	100, b-c	10	100	100, b-c	10	100, b-c
B2	13	50/50 b-c/a	8	62	100, a	10	77	100, a	9	100, a
Type II Fuel at 160° F										
Adhesive	Original lbs/in	Failure* Mode	14 Days lbs/in.	% Retention	Failure Mode	28 Days lbs/in.	% Retention	Failure Mode	42 Days lbs/in.	Failure Mode
B1	10	65/35 a/b-c	2	20	100, b-c	2	20	100, b-c	2	100, b-c
A2	9	100, b-c	2	22	80/20 b-c/a	2	22	100, c-a	2	100, b-c
B2	13	50/50 b-c/a	1	8	75/25 b-c/a	2	15	100, c-a	2	100, c-a

*See Appendix E for failure mode description

TABLE 16
ADHESIVES AND PRIMERS EVALUATIONS IN
BONDING POLYETHER POLYURETHANE COATING TO METAL

Primer	Adhesive	Original lbs/in.	Failure* Mode	14 Days lbs/in.	Water at 160° F				28 Days lbs/in.	% Retention	Failure Mode	42 Days lbs/in.	% Retention	Failure Mode
					% Retention	Failure Mode	% Retention	Failure Mode						
1244	B1	32	20/40/40 b-c/c-f/a-p	6	19	90/10 p-m/c-a	5	16	75/25 a-p/a-c	5	16	75/25 a-p/c-a		
AP-10	B1	21	90/10 b-c/c-f	18	86	50/25/25 b-c/c-a/c-f	22	105	50/50 b-c/a	22	105	70/25/5 a-c/c-f/b-c		
607	B1	21	60/40 b-c/p-m	0	0	100, p-m	0	0	-	-	-	-		
1244	A2	19	100, b-c	6	32	100, p-m	6	32	100, a-p	4	21	100, a-c		
AP-10	A2	24	85/15 b-c/a-p	22	92	40/50/10 b-c/c-f/a-p	24	108	40/15/45 b-c/a/c-f	27	112	70/30 c-f/a-c		
607	A2	18	100, p-m	0	0	100, p-m	0	0	-	-	-	-		
1244	B2	39	50/50 c-f/b-c	2	5	100, c-a	3	8	100, p-m	3	8	100, a-p		
AP-10	B2	52	100, c-f	40	77	100, c-f	30	58	100, a-p	30	58	85/15 a-p/c-f		
607	B2	15	100, p-m	0	0	100, p-m	0	0	-	-	-	-		

*See Appendix E for failure mode description

TABLE 16

ADHESIVES AND PRIMERS EVALUATIONS IN
BONDING POLYETHER POLYURETHANE COATING TO METAL (CONT'D.)

Primer	Adhesive	Original lbs/in.	Failure* Mode	14 Days lbs/in.	Fuel at 160° F			28 Days lbs/in.	42 Days lbs/in.	Failure Mode	% Retention	Failure Mode	Failure Mode
					% Retention	Failure Mode	% Retention						
1244	B1	21	85/15 b-c/p-m	2	10	60/40 b-c/a	19	4	4	50/50 a/b-c	19	90/10 b-c/a	90/10 b-c/a
AP-10	B1	21	100, b-c	2	10	100, b-c	14	3	4	100, b-c	19	100, b-c	100, b-c
607	B1	19	60/40 p-m/b-c	4	21	90/10 b-c/a	32	6	5	90/10 b-c/a	26	70/30 b-c/a	70/30 b-c/a
1244	A2	21	90/10 b-c/p-m	2	10	100, b-c	14	3	3	100, b-c	14	100, b-c	100, b-c
AP-10	A2	20	75/25 b-c/a-p	4	20	90/10 b-c/a	20	4	4	100, b-c	20	100, b-c	100, b-c
607	A2	16	100, p-m	4	25	90/10 b-c/a	31	5	5	100, b-c	31	80/20 b-c/a	80/20 b-c/a
1244	B2	34	95/5 b-c/a-p	5	15	60/40 a/b-c	18	6	4	30/20/50 p-m/a/b-c	12	65/15/20 p-m/a/b-c	65/15/20 p-m/a/b-c
AP-10	B2	48	100, c-f	6	12	100, b-c	12	6	6	100, b-c	12	100, b-c	100, b-c
607	B2	10	100, a-m	6	60	100, b-c	70	7	6	100, b-c	60	100, b-c	100, b-c

*See Appendix E for failure mode description

TABLE 17

ADHESIVES EVALUATION IN BONDING
POLYESTER POLYURETHANE COATING TO ITSELF

Water at 160° F										
Adhesive	Original lbs/in.	Failure* Mode	14 Days lbs/in.	% Retention	Failure Mode	28 Days lbs/in.	% Retention	Failure Mode	42 Days lbs/in.	% Retention
A1	31	80/20 c-f/a	12	39	90/10 a/c-f	4	13	100, a	1	3
A2	17	100, c-f	28	165	100, c-f	34	200	90/10 c-f/a	31	182
A3	36	100, c-f	24	67	100, f	24	67	100, c-f	21	58
Fuel at 160° F										
Adhesive	Original lbs/in.	Failure* Mode	14 Days lbs/in.	% Retention	Failure Mode	28 Days lbs/in.	% Retention	Failure Mode	42 Days lbs/in.	% Retention
A1	29	70/30 c-f/a	10	34	80/20 c-f/a	11	38	40/40/20 a/c-f/c	9	31
A2	21	100, c-f	8	38	70/30 c-f/a	8	38	90/10 c/a	7	33
A3	42	100, c-f	8	19	50/50 a/c-f	7	17	85/15 a/c-f	8	19

*See Appendix E for failure mode description

TABLE 18
ADHESIVES AND PRIMERS EVALUATIONS IN
BONDING POLYESTER POLYURETHANE COATING TO METAL

Primer	Adhesive	Original lbs/in	Failure* Mode	14 Days lbs/in.	Water at 160° F				28 Days lbs/in.	% Retention	Failure Mode	42 Days lbs/in.	% Retention	Failure Mode
					% Retention	Failure Mode	% Retention	Failure Mode						
1244	A1	29	85/15 p-m/c-f	6	21	100, a	17	100, a	5	17	100, a	4	14	100, a
AP-10	A1	26	60/40 p-m/c-f	19	73	50/50 a-p/a	46	100, a	12	46	100, a	4	15	100, a
607	A1	36	85/15 p-m/c-f	0	0	100, p-m	-	-	-	-	-	-	-	-
1244	A2	38	85/15 p-m/c-f	8	21	100, a-m	37	95/5 a/c-f	14	37	95/5 a/c-f	16	42	85/15 a/c-f
AP-10	A2	38	40/60 a-p/c-f	34	89	70/30 a-m/c-a	95	100, a	36	95	100, a	30	79	100, a-p
607	A2	22	100, a-p	0	0	100, p-m	-	-	-	-	-	-	-	-
1244	A3	51	100, c-f	1	2	100, p-m	0	100, p-m	0	0	100, p-m	-	-	-
AP-10	A3	46	60/40 c-f/a-p	33	72	85/15 c-f/a-p	78	75/25 c-f/a	36	78	75/25 c-f/a	32	70	60/40 c-f/a
607	A3	22	100, p-m	0	0	100, p-m	-	-	-	-	-	-	-	-

*See Appendix E for failure mode description

TABLE 18
ADHESIVES AND PRIMERS EVALUATIONS IN
BONDING POLYESTER POLYURETHANE COATING TO METAL (CONT'D.)

Primer	Adhesive	Original lbs/in.	Failure* Mode	Type II Fuel at 160° F						Failure Mode	% Retention	42 Days lbs/in.	% Retention	Failure Mode
				14 Days lbs/in.	% Retention	Failure Mode	28 Days lbs/in.	% Retention	42 Days lbs/in.					
1244	A1	26	80/20 p-m/c-f	7	27	100, p-m	8	31	7	100, a	27	7	27	100, a
AP-10	A1	31	100, p-m	20	64	80/20 c-f/a-p	20	64	16	60/40 a/c-f	52	16	52	65/35 a/c-f
607	A1	35	75/25 a/c-f	18	51	80/20 p-m/c-f	20	57	16	60/40 a/c-f	46	16	46	80/20 a/c-f
1244	A2	40	90/10 c-f/a	20	50	80/20 c-f/p-m	22	55	17	80/20 c-f/a	42	17	42	70/30 c-f/a
AP-10	A2	35	90/10 a/c-f	20	57	65/35 c-f/a	22	63	20	85/15 c-f/a	57	20	57	50/50 a/c-f
607	A2	26	100, a-m	24	92	50/50 c-f/p-m	23	88	20	70/30 c-f/a	77	20	77	80/20 c-f/a
1244	A3	40	100, c-f	0	0	100, p-m	-	-	-	-	-	-	-	-
AP-10	A3	47	100, c-f	20	42	50/50 c-f/p-m	21	45	20	65/35 c-f/a	42	20	42	80/20 c-f/a
607	A3	23	100, p-m	16	70	100, p-m	16	70	14	50/50 c-f/a	61	14	61	50/50 a/c-f

*See Appendix E for failure mode description

TABLE 19

POLYETHER COATING-TC-COATING
ADHESION EVALUATION UPON AGING AT 160° F

Fabric	Original lbs/in	Water Aging															
		Failure* Mode	14 Days lbs/in.	Retention %	Failure Mode	28 Days lbs/in.	Retention %	Failure Mode	42 Days lbs/in.	Retention %	Failure Mode	70 Days lbs/in.	Retention %	Failure Mode	90 Days lbs/in.	Retention %	Failure Mode
Glass	10	100, a	4	40	100, a	2	20	100, a	2	20	100, a	1	10	100, a	0	0	100, a
Kevlar	12	100, a	4	33	100, a	3	25	100, a	2	17	100, a	2	17	100, a	4	33	100, a
Nylon	13	100, a	3	23	100, a	1	8	100, a	1	8	100, a	1	8	100, a	0	0	100, a
Dacron	10	100, a	5	50	100, a	2	20	100, a	2	20	100, a	2	20	100, a	3	30	100, a

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Type II Fuel Aging

Fabric	Original lbs/in	Failure* Mode	14 Days lbs/in.	Retention %	Failure Mode	28 Days lbs/in.	Retention %	Failure Mode	42 Days lbs/in.	Retention %	Failure Mode						
Glass	8	100, c-f	0	0	100, c-f	-	-	-	-	-	-	-	-	-	-	-	-
Kevlar	7	100, c-f	0	0	100, c-f	-	-	-	-	-	-	-	-	-	-	-	-
Nylon	7	100, c-f	0	0	100, c-f	-	-	-	-	-	-	-	-	-	-	-	-
Dacron	4	100, c-f	0	0	100, c-f	-	-	-	-	-	-	-	-	-	-	-	-

*See Appendix E for failure mode description

TABLE 20
POLYETHER COATING-TO-METAL
ADHESION EVALUATION UPON AGING AT 160° F

Water Aging																	
Fabric	Original lbs/in	Failure* Mode	14 Days lbs/in.	Retention %	Failure Mode	28 Days lbs/in.	Retention %	Failure Mode	42 Days lbs/in.	Retention %	Failure Mode	70 Days lbs/in.	Retention %	Failure Mode	90 Days lbs/in.	Retention %	Failure Mode
Glass	32	100, c-f	4	12	100, a-p	1	3	100, a-p	0	0	100, a-p	-	-	-	-	-	-
Kevlar	36	100, c-f	7	19	100, a-p	2	6	100, a-p	0	0	100, a-p	-	-	-	-	-	-
Nylon	36	100, c-f	6	17	100, a-p	1	3	100, a-p	0	0	100, a-p	-	-	-	-	-	-
Dacron	47	100, c-f	4	8	100, a-p	1	2	100, a-p	0	0	100, a-p	-	-	-	-	-	-
Type II Fuel Aging																	
Fabric	Original lbs/in	Failure* Mode	14 Days lbs/in.	Retention %	Failure Mode	28 Days lbs/in.	Retention %	Failure Mode	42 Days lbs/in.	Retention %	Failure Mode						
Glass	35	100, c-f	4	11	75/25 c-a/a-p	1	3	100, a	0	0	100, a						
Kevlar	36	100, c-f	3	8	100, a-p	1	3	100, a	0	0	100, a						
Nylon	42	100, c-f	4	10	50/50 a-c/a-p	1	2	100, a	0	0	100, a						
Dacron	42	100, c-f	3	7	100, a-p	1	2	100, a	0	0	100, a						

*See Appendix E for failure mode description

TABLE 22

POLYESTER COATING-TO-METAL
ADHESION EVALUATION UPON AGING AT 160° F

Fabric	Original lbs/in	Water Aging									
		Failure* Mode	14 Days lbs/in.	Retention %	Failure Mode	28 Days lbs/in.	Retention %	Failure Mode	42 Days lbs/in.	Retention %	Failure Mode
Glass	21	100, c-f	25	119	95/5 c-f/p-a	6	29	100, a	3	14	100, a
Kevlar	19	100, c-f	18	95	95/5 c-f/a-p	16	84	100, a	5	26	100, a
Nylon	55	100, c-f	18	33	100, a-p	6	11	100, a	1	2	100, a
Dacron	22	100, c-f	20	91	100, a-p	9	41	100, a	4	18	100, a
											95/5 a/c-f
											95/5 c-f/a
											100, a
											75/25 a/c-f
Fabric	Original lbs/in	Fuel Aging									
		Failure* Mode	14 Days lbs/in.	Retention %	Failure Mode	28 Days lbs/in.	Retention %	Failure Mode	42 Days lbs/in.	Retention %	Failure Mode
Glass	23	100, c-f	13	56	100, c-f	14	61	100, c-f	13	57	100, c-f
Kevlar	18	100, c-f	19	106	100, c-f	20	111	100, c-f	24	133	50/50 c-f/a
Nylon	37	100, c-f	30	81	100, c-f	20	54	95/5 c-f/a	13	35	100, a
Dacron	26	100, c-f	21	81	100, c-f	18	69	60/40 a/c-f	12	46	100, a

*See Appendix E for failure mode description

TABLE 23

SEAM ADHESION
EVALUATION UPON AGING IN WATER AT 180° F

POLYESTER

Fabric	Original lbs/in.	Failure* Mode	7 Days lbs/in.	% Retention	Failure Mode	14 Days lbs/in.	% Retention	Failure Mode	21 Days lbs/in.	% Retention	Failure Mode	28 Days lbs/in.	% Retention	Failure Mode
Glass	28	75/25 c-f/a	20	71	75/25 c-f/a	5	18	100, a	3	11	65/35 a/c-f	3	11	85/15 c-f/a
Kevlar	21	50/50 a/c-f	16	76	50/50 a/c-f	5	24	100, a	3	14	70/30 a/c-f	0	0	70/30 c-f/a
Nylon	17	90/10 a/c-f	17	100	90/10 a/c-f	4	24	100, a	3	18	100, a	0	0	100, c-f
Dacron	25	80/20 a/c-f	11	44	100, a	4	16	100, a	4	16	100, a	1	4	95/5 a/c-f

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POLYETHER

Fabric	Original lbs/in.	Failure* Mode	7 Days lbs/in.	% Retention	Failure Mode	14 Days lbs/in.	% Retention	Failure Mode	21 Days lbs/in.	% Retention	Failure Mode	28 Days lbs/in.	% Retention	Failure Mode
Glass	22	80/20 c-f/a	16	73	40/40/20 c-f/a/c	15	68	85/15 c-f/a	13	59	70/30 a/c-f	8	36	100, a
Kevlar	15	45/55 a/c-f	7	47	60/40 a/c-f	8	53	65/35 a/c-f	9	60	60/40 a/c-f	7	47	70/30 a/c-f
Nylon	15	95/5 a/c-f	5	33	90/10 c-f/a	4	27	100, c-f	4	27	100, c-f	4	27	100, c-f
Dacron	10	100, a	23	230	90/10 a/c-f	10	100	80/20 a/c-f	10	100	90/10 a/c-f	7	70	100, a

*See Appendix E for failure mode description

TABLE 24

SEAM ADHESION
EVALUATION UPON AGING IN FUEL AT 73.5° F

POLYESTER

Fabric	Original lbs/in.	Failure* Mode	14 Days lbs/in.	% Retention	Failure Mode	28 Days lbs/in.	% Retention	Failure Mode	42 Days lbs/in.	% Retention	Failure Mode
Glass	23	100, c-f	15	65	100, c-f	12	52	100, c-f	15	65	100, c-f
Kevlar	26	100, c-f	17	65	55/45 a/c-f	15	58	70/30 c-f/a	17	65	50/50 c-f/a
Nylon	23	70/30 a/c-f	18	78	100, a	18	78	95/5 a/c-f	16	70	80/20 a/c-f
Dacron	27	60/40 a-c/f	24	88	90/10 a/c-f	22	81	80/20 a/c-f	21	78	80/20 a/c-f

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POLYETHER

Fabric	Original lbs/in.	Failure* Mode	14 Days lbs/in.	% Retention	Failure Mode	28 Days lbs/in.	% Retention	Failure Mode	42 Days lbs/in.	% Retention	Failure Mode
Glass	13	50/50 a/c	3	23	60/40 c/a	3	23	50/50 a/c	3	23	70/30 c/a
Kevlar	18	80/20 c/a	6	33	80/20 c/a	5	28	70/30 c/a	5	28	75/25 a/c
Nylon	11	85/15 a/c-f	3	27	80/20 a/c	4	36	100, a	4	36	100, a
Dacron	8	80/20 a/c	3	38	65/35 a/c	3	38	70/30 a/c	3	38	85/15 a/c

*See Appendix E for failure mode description

TABLE 25

POLYETHER COATING-TO-METAL (REMAKE)
ADHESION EVALUATION UPON AGING AT 160° F

Fabric	Original lbs/in	Type II Fuel Aging									
		Failure* Mode	14 Days lbs/in.	% Retention	Failure Mode	28 Days lbs/in.	% Retention	Failure Mode	42 Days lbs/in.	% Retention	Failure Mode
Glass	28	60/40 a/c-f	7	25	65/35 c/a	6	21	50/50 c-a/c	9	32	50/50 c-a/c
Kevlar	28	80/20 c-f/a	9	32	100, c	9	32	100, c	10	36	100, c
Nylon	32	95/5 c-f/a	8	25	100, c	8	25	100, c	10	31	100, c
Dacron	34	85/15 c-f/a	9	26	90/10 c/a	9	26	80/20 c/c-a	8	24	75/25 c-a/c

*See Appendix E for failure mode description

TABLE 26
POLYETHER COATING-TO-COATING (REMAKE)
ADHESION EVALUATION UPON AGING AT 160° F

Type II Fuel Aging											
Fabric	Original Lbs/in	Failure* Mode	14 Days lbs/in.	% Retention	Failure Mode	28 Days lbs/in.	% Retention	Failure Mode	42 Days lbs/in.	% Retention	Failure Mode
Glass	19	100, c	4	21	70/30 c/a	4	21	70/30 c-a/c	7	37	70/30 c/a
Kevlar	13	100, c	4	31	100, c	4	31	60/40 c-a/c	6	46	75/25 c/a
Nylon	13	50/50 a/c	5	38	90/10 c/a	4	31	90/10 c/c-a	6	46	50/50 c/a
Dacron	16	100, c	4	25	60/40 a/c	4	25	100, c-a	7	44	80/20 c/a

*See Appendix E for failure mode description

TABLE 27

DEAD LOAD SHEAR RESISTANCE OF SEAMS
AND ALUMINUM TO COATED FABRIC BONDS

50 lb/in STRESS at 200⁰ F for 8 hrs.

<u>Coating/Fabric</u>		<u>Seams</u>	<u>Aluminum/Coated Fabric</u>
<u>Polyether</u>	Glass	OK	OK
	Kevlar	OK	OK
	Nylon	OK	OK
	Dacron	Failed	Failed
<u>Polyester</u>	Glass	OK	Failed
	Kevlar	OK	OK
	Nylon	OK	Failed
	Dacron	Failed	OK

TABLE 28

ADHESIVES AND PRIMERS UTILIZED IN
SCREENING EVALUATIONS

<u>Adhesives</u>		
<u>Code</u>	<u>Material and Supplier</u>	<u>Parts w/w</u>
A1	Urethane 6001 (UNIROYAL, Inc.)	48
	Saran F310 (Dow Chemical)	8
	Staboxal M (Mobay Chemical)	2
	Methyl Ethyl Ketone	<u>44</u>
A2	PA30 (K. J. Quinn Corp.)	100
	Saran F310 (Dow Chemical)	10
	Q303 (K. J. Quinn Corp.)	50
	Methyl Ethyl Ketone	<u>1667</u>
A3	101575K UNIROYAL proprietary compound	
B1	Vibrathane B600 (UNIROYAL, Inc.)	100
	MDA-Methyl Dianiline (Dow Chemical)	3
	Tonox (UNIROYAL, Inc.)	2.33
	Methyl Ethyl Ketone	<u>187</u>
B2	101575U UNIROYAL proprietary	

Primers

AP10 Primer (M & T Chemical Corp.)
Chemlok 607 (Dayton Chemical Corp.)
Thixon AB1244 (Whittaker Corp.)

APPENDIX A

Test Methods For Coating Compounds ^{1/}

<u>Property</u>	<u>Test Method</u>	
	<u>Spec/Std</u>	<u>Para/Method</u>
Tensile strength (initial)	Fed. Std. 601	4111
Tensile stress at 200% elongation	Fed. Std. 601	4131
Ultimate elongation	Fed. Std. 601	4121
Initial tensile strength retained after immersion in distilled water for:		
90 days at 160 ⁰ F	Fed. Std. 601	6111 incl. 4.8.1
28 days at 180 ⁰ F	Fed. Std. 601	6111 incl. 4.8.1
Initial tensile strength retained after fuel immersion in Medium No. 5 in sealed containers ^{2/} for:		
42 days at 160 ⁰ F	Fed. Std. 601	6111 incl. 4.8.1
42 days at 73.5 ⁰ F	Fed. Std. 601	6111 incl. 4.8.1
Initial tensile strength retained after acid fuel immersion in sealed containers for: ^{3/}		
42 days at 160 ⁰ F	Fed. Std. 601	6111 incl. 4.8.1
42 days at 73.5 ⁰ F	Fed. Std. 601	6111 incl. 4.8.1

APPENDIX A (Continued)

<u>Property</u>	<u>Test Method</u>	
	<u>Spec/Std</u>	<u>Para/Method</u>
Initial tensile strength retained 500 hours of accelerated weathering at 10% elongation <u>3/</u>	Fed. Std. 601	7311 <u>4/</u>
Fuel contamination: <u>5/</u> Unwashed existent gum	MIL-T-52766	4.6.8
Heptane washed existent gum	MIL-T-52766	4.6.8
Cracking resistance of sheets of coating compound 40 mils thick: at minus 25° F	MIL-T-52766	4.6.11
at minus 40° F	MIL-T-52766	<u>6/</u>
Modulus of rigidity (initial)		<u>7/</u>
at room temperature	Fed. Std. 601	5611-1
at minus 25° F	Fed. Std. 601	5611-1
at minus 40° F	Fed. Std. 601	5611-1
Modulus of rigidity (after fuel extraction):		<u>8/</u>
at room temperature	Fed. Std. 601	5611-1
at minus 25° F	Fed. Std. 601	5611-1
Torsional stiffness ratio (initial):		
at minus 25° F	Fed. Std. 601	5612
at minus 40° F	Fed. Std. 601	5612
Torsional stiffness ratio (after fuel extraction):		
at minus 25° F	Fed. Std. 601	5612
at minus 40° F	Fed. Std. 601	5612

APPENDIX A (Continued)

Test Procedures for Coating Compounds

1. Specimens. Three specimens shall be prepared for each modulus of rigidity test and for each torsional stiffness ratio test. Specimens shall be 1.5 inches long, 0.080 inch wide, and 0.75 (+0.010 or -0.035) inch thick. The specimens shall be cut with a sharp die.
2. Fuel extraction procedure. Three specimens shall be immersed in test medium No. 5 or Method 6001, Fed. Test Method Std. No. 601. The volume of the test medium shall be not less than 20 times that of the three specimens, and shall be sufficient to completely cover the specimens after swelling. Specimens shall remain immersed in the test medium for 94 hours \pm 2 hours at $73.5^{\circ}\text{F} \pm 2^{\circ}\text{F}$. After fuel extraction, the specimens shall be dried in circulating air at room temperature for not less than 12 hours, then dried in a forced draft air oven at 130°F until successive weighings at 4 hour intervals show no change in weight.
3. Low-temperature cabinet. The low-temperature cabinet to be used for conditioning specimens shall be of the mechanically refrigerated or dry-ice type, and shall conform to the following requirements.
 - a. The heat-transfer medium in the test chamber shall be air, carbon dioxide, nitrogen, or a mixture of these gases.
 - b. Temperatures shall be maintained within a range of plus or minus 2°F of the specified temperature.
 - c. The heat-transfer medium shall be thoroughly circulated in the test chamber by means of mechanical agitation, which shall function at all times during both the conditioning and testing periods.
 - d. Automatic temperature control shall be used.
 - e. Each of the dimensions of the test chamber interior shall be at least 24 inches.
 - f. If incandescent lamps are provided for illumination, they shall be located at least 16 inches from the test specimens.
 - g. For cabinets other than those of the walk-in type, the following shall apply:

APPENDIX A (Continued)

- 1) Two observation windows shall be provided, one in the side of the box, and one in the top. Windows shall be either 3- or 5-ply glass construction. Windows shall be at least 7 inches square.
- 2) For operation of the test equipment, hand holes equipped with insulated sleeves shall be installed in the wall of the cabinet. Hand holes shall be at least 5 inches in diameter.
- 3) In the dry-ice type of cabinet, a fan or blower in the dry-ice compartment shall circulate the heat-transfer medium from the dry-ice compartment into the test chamber, and back. The amount and movement of the heat-transfer medium shall be adjusted to minimize the fluctuations of the temperature in the test chamber.
4. Modulus of rigidity. The modulus of rigidity shall be determined according to Method No. 5611-1 of Fed. Test Method Std No. 601. Determinations of the modulus at low temperatures shall be performed after the specimens have been refrigerated for 166 hours \pm 1 hour at the prescribed temperature \pm 2° F in a low-temperature cabinet of the type prescribed in paragraph 3 above, and without removing the specimens from the cabinet.
5. Torsional stiffness ratio. The torsional stiffness ratio shall be calculated according to Method No. 5612 of Fed. Test Method No. 601

APPENDIX A (Continued)

Footnotes:

- 1/ Properties after cure.
- 2/ Medium No. 5 of Method 6001 of Fed. Test Method Std. No. 601.
- 3/ Applicable to all exterior compounds. That is, compounds between the fabric and the outside of the tank.
- 4/ Alternate corex D filters in place.
- 5/ Applicable to all interior compounds. That is, compounds between the fabric and the inside of the tank.
- 6/ Procedures of paragraph 4.6.11 shall be followed except that the temperature shall be minus 40° F instead of minus 25° F.
- 7/ See paragraphs 1, 3 and 4 of Test Procedures for Coating Compounds.
- 8/ See paragraphs 1 through 4 of Test Procedures for Coating Compounds.
- 9/ Acid fuel shall be prepared by acidifying Medium No. 5 of Method 6001, Fed. Test Method Std. No. 601 with glacial acetic acid to a Total Acid Number (TAN) of 3.0 as determined by ASTM Designation: D-664 "Neutralization Number by Potentiometric Titration".

APPENDIX B

Test Methods for Seams ^{1/}

<u>Property</u>	<u>Test Method</u>
Dead load shear resistance under 50 lb./in. stress at 200 ⁰ F for 8 hours	Para. 4.6.15, MIL-T-52766
Peel adhesion (initial)	Method 8011, Fed. Std. 601
Peel adhesion after immersion in distilled water for:	
90 days at 160 ⁰ F	Methods 8011 and 6001, Fed. Std. 601, para. 4.6.14, MIL-T-52766
28 days at 180 ⁰ F	Methods 8011 and 6001, Fed. Std. 601, para. 4.6.14, MIL-T-52766
Peel adhesion after immersion in Medium No. 5 in sealed containers for:	
42 days at 160 ⁰ F	Methods 8011 and 6001, Fed. Std. 601, para. 4.6.14, MIL-T-52766
42 days at 73.5 ⁰ F	Methods 8011 and 6001, Fed. Std. 601, para. 4.6.14, MIL-T-52766

Footnotes:

1/ Properties after cure.

2/ Medium No. 5 of Method 6001 of Fed. Test Method Std. No. 601.

APPENDIX C

Test Methods For Coatings-to-Metal Bonds 1/

<u>Property</u>	<u>Test Method</u>
Dead load shear resistance under 50 lb./in. stress at 200° F for 8 hours	Para. 4.6.16.3, MIL-T-52766 <u>2/</u>
Peel adhesion of aluminum strip to coated fabric (initial)	Para. 4.6.17, MIL-T-52766
Peel adhesion of aluminum strip to coated fabric after immersion in distilled water at 160° F ± 2° F for 90 days	Method 8031, Fed. Std. 601, para. 4.6.17, MIL-T-52766
Peel adhesion of aluminum strip to coated fabric after immersion in Medium No. 5 <u>3/</u> at 160° F ± 2° F for 42 days	Method 8031, Fed. Std. 601, para. 4.6.17, MIL-T-52766

Footnotes:

1/ Properties after cure.

2/ Test specimen shall be prepared according to paragraph 4.6.16 of MIL-T-52766, except that the coated fabric strip 1.0 inch ± 0.02 inch wide shall overlap an end of the aluminum strip for a distance of 1.0 inch ± 0.02 inch.

3/ Medium No. 5 of Method 6001, Fed. Test Method Std. No. 601

APPENDIX D

Characteristics of Fabrics

<u>Type</u>	<u>Weight (oz./yd²)</u>	<u>Weave</u>	<u>Manufacturer</u>
Kevlar	6.8	Plain	J. P. Stevens
Glass	19	Plain	J. P. Stevens
Dacron	14.8	2 x 2 basket	UNIROYAL
Nylon	13	2 x 2 basket	UNIROYAL

APPENDIX E

Failure Mode Description

Type

- c-f - coating to fabric bond failure
- c-a - coating to adhesive bond failure
- a - bond failure within the adhesive
- c - bond failure within the coating
- a-p - adhesive to metal primer bond failure
- b-c - bond failure between coating layers
- p-m - bond failure between primer and metal

Degree

The numerical figure represents the percentage of the failure attributed to the specific type indicated by the letter description.