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PROTECTIVE COATINGS FOR MAGNESIUM

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FOREWORD

This report was prepared by The Sherwin-Williams Company under USAF Contract No. AF 33(616)-35. The contract was initiated under Project No. 73121 (C) "Organic Protective Coatings", and was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center with R. L. Stout acting as project engineer.

ABSTRACT

The purpose of this investigational work, which was to strive to develop improved coatings for the protection of magnesium, has been accomplished. The recommended finishing systems based on this work use a vinyl toluene-ether ester vehicle in both the primer and the topcoat. The primers are pigmented at 35% pigment volume concentration with zinc chromate and silicon dioxide. The solvent resistance of these systems has been variable. The two types used in the samples submitted for evaluation show bad softening but fair recovery upon solvent evaporation.

Time consuming difficulties were encountered in establishing a satisfactory test surface. Pretreatment of the magnesium according to MIL-M-3171, Type III, by a preferred commercial processor and the use of replicate test panels was the solution. Wash primers as pretreatments for magnesium or as an addition to dichromate pretreatments show promise but their performance is too variable to warrant inclusion in the recommended system at this time. Improvements in wash primers included the use of strontium chromate and silicon dioxide as the pigmentation and the reduction of acid activator content to 25% of normal with simultaneous control of water content.

Several items were investigated briefly and showed enough promise to warrant further study. These include polysulfide resins, furan resins, a pigment prepared from calcium sulfide and ammonium vanadate, pigmentation changes in wash primers, and a method for determining the permeability of films to chloride ions.

This work was done by the Paint Research Department of The Sherwin-Williams Company under contract AF 33(616)-35 from 29 March, 1952 to 1 June, 1954.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

M. R. WHITMORE
Technical Director
Materials Laboratory
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INTRODUCTION

The purpose of this contract is to strive to develop improved organic coatings for the protection of magnesium parts of aircraft. Such improvements may be in corrosion resistance, adhesion or weight.

The well known and generally excellent finishing system for aluminum, Zinc Chromate Primer, MIL-P-6889a, followed by aluminized lacquer, MIL-L-7178, has been used by the Air Force on aluminum for many years. With the introduction of magnesium into the construction of aircraft frames in conjunction with aluminum, this same finishing system was adopted for magnesium. Here the performance has not been as favorable and failures have been noted both in adhesion and corrosion.

These failures can be explained by the relatively high pH of the corrosion products of magnesium and the presence of an active bimetallic couple at each magnesium-aluminum junction. The vehicle of Zinc Chromate Primer is either an alkyd or an alkyd-phenolic dispersion resin combination. In either case the alkali resistance can be rated only fair. In fact, the resistance of these vehicles to a pH as high as that produced by magnesium oxide or hydroxide is poor. Thus it should be expected that any corrosion of magnesium occurring at breaks in the coating system would tend to creep rapidly under the specification painting system through destruction of the primer vehicle at the paint-metal interface. This corrosion creepage in conjunction with the potential difference between aluminum and magnesium that is present at each bimetallic couple, establishes conditions favorable to severe corrosion.

On these premises, much of the effort of this project has been directed toward developing a paint system utilizing an alkali resistant vehicle. Alkali resistance alone, of course, is not the whole story. The vehicle must also act as a binder for the pigment in such a way that there is a reservoir of pigment available for inhibition of corrosion. Other attributes such as good adhesion, rapid drying at room temperature, flexibility, hardness, and abrasion resistance are also primary functions of the vehicle and were considered.

Pigmentation also was given considerable attention, particularly in the primer. Corrosion inhibitive pigments must be available from the primer in certain proportions over long periods of exposure in order to be effective. Such prolonged availability depends upon controlled aqueous solubility and film permeability. The former quality is an inherent characteristic of the individual pigments but much can be done concerning the latter quality by proper manipulation of the pigment volume concentration or PVC. Thus the relationship of PVC to critical pigment volume concentration (CPVC) was studied in several instances.

Much of the investigation of the corrosion inhibitive properties of various pigments was done using a simple technique. This consisted of exposing metal slips to saturated saline solutions of the various pigments and observing the progress of corrosion visually. All of the more promising pigments were evaluated subsequently in actual coatings to substantiate the water tests.

Although pretreatment of the metal with inorganic chemicals was not a part of the purpose of this contract, it was found necessary to do considerable evaluation work on pretreatments in order to obtain as uniform a substrate as possible. Consideration was given also to the use of a wash primer, or an organic pretreatment, as a replacement for or an adjunct to the regular dichromate pretreatment.

Sixteen monthly reports and seven quarterly reports have been prepared and submitted previously. This is the twenty-fourth and final report and covers the work done during the entire period of the contract, 29 March, 1952 through 1 June, 1954.

SECTION I

TEST METHODS

A. Panels

In any research and development work, it is axiomatic that experimental data should represent variations in results that occur because of controlled variations in the experimental set up and not because of uncontrolled variations. This is particularly difficult in working with paints which are such complex mixtures of natural and synthetic organic and inorganic materials. In addition to the composition of the paint itself there are innumerable possibilities for uncontrolled variations in the nature, cleaning and pretreatment of the substrate; the application, drying and aging of the films; and in the test procedures. Anything that can be done to minimize these undesirable variations is well repaid by the increased reliability of the experimental results. Therefore, considerable effort was expended on the design, pretreatment and assembly of the panels for this project.

At the direction of the Air Force FS-1H magnesium sheet was selected for the main test surface. This sheet, .051 inches thick was obtained from Dow Chemical Company. Panels were cut to the specified size, drilled for riveting and handling, and the edges rounded and deburred prior to pretreatment. FS-1 is notable as a magnesium alloy because of the relatively low content of aluminum (3.0%) as compared, for instance, with J alloy (6.5%)[1]. This, coupled with its high purity with respect to iron, nickel and copper, gives FS-1 relatively good corrosion resistance.

It has been pointed out by Alexander[2] that magnesium alloys may vary sufficiently in composition to affect their corrosion rate materially. Thus Alexander feels that magnesium alloys differ sufficiently within their specifications to overshadow any differences in corrosion rate brought about by wide variances in the quality of applied organic coatings.

The Magnesium Division of Dow Chemical Company does not agree with this viewpoint. They feel that an alloy such as FS-1 has tight enough specifications to give reproducible corrosion rates. At the same time they concede that surface contamination from rolling processes can and does vary and that such variations affect corrosion rates materially. De Long[3] has suggested different pickling operations for the removal of various types of soil such as graphite base lubricants, imbedded foreign materials and mill scale. It has been suggested that even with proper surface conversion treatment of magnesium as specified in MIL-M-3171 all surface contaminants are not removed in every case. These residual contaminants then could cause the variation in corrosion rates found by Alexander.

At any rate it is no more than prudent to confine the magnesium panels in an experiment to those from one lot of magnesium if possible. To this end panels have been ordered in groups of 1000 for the 3 x 6 inch size and 250 for the 6 x 12 inch size. It was specified that each batch of panels must be from sheet from the same lot of magnesium. These sheets were acetic acid pickled by Dow before cutting into panels. Even so they were sometimes given a second pickle prior to the pretreatment if there were any evidences of residual mill scale or other contaminants remaining on the surface.

Again at the direction of the Air Force 24ST Alclad aluminum was selected as the metal for use in couples where galvanic corrosion was of interest. 24ST aluminum was chosen in preference to 75S even though the latter is more prevalent in aircraft construction. This was done with the thought that the use of 24ST would result in a more severe test in a couple with magnesium. Although there is no evidence to support the existence of corrosion variances for aluminum similar to magnesium, similar arrangements were made for the acquisition of aluminum panels in relatively large batches from one lot. The aluminum panels were .032 inches thick.

Most of the experimental work has been done on 3 x 6 inch or 6 x 12 inch panels. In cases where corrosion was of interest, such as in salt spray tests and exterior exposure, an assembly was constructed in which a magnesium and an aluminum panel were lapped one inch along the long edge and fastened with three 56S aluminum rivets conforming to AN-QQ-W-298, AN470B 4-4. Since it is difficult to rivet two thin sheets of metal without some buckling of the sheets a special jig was constructed to facilitate the riveting. In spite of this some buckling occurred and as a consequence there was some applied stress on the metal panels.

B. Paint Application

Through consultation with the project monitor, a standard procedure was established at the beginning of the contract for coating the panels. Pretreated magnesium panels were primed with two coats of primer with a two hour air drying interval. Aluminum panels were given only one coat of primer. After overnight drying the panels were riveted together and the assembly was given two coats of topcoat with a two hour air drying interval. The panels were allowed to age in the laboratory for 48 hours before being subjected to test conditions. All application was by spray. This procedure was followed except where otherwise noted.

Since each coat of paint was applied at about 0.5 mils thickness the total film thickness on the exposed magnesium surface was about two mils and on the exposed aluminum surface, about one and a half mils. The lap joint was separated with about one and a half mils of primer. Electrical contact between each pair of panels was checked with a simple one and a half volt battery and bulb. Lighting of the bulb with the coupled panel in the circuit was considered evidence of good electrical contact.

The edges of all assembled panels were protected by an additional coat of a proprietary alkyd primer and a coat of a mixture of paraffin waxes. The primer was applied by a modified dipping method and covered only about one eighth of an inch from the edge. The wax was applied by dipping and covered about one quarter of an inch from the edge. Obviously the lapped edges could not be given this extra protection.

C. Evaluation Test Methods

It was agreed both contractually and verbally that salt spray testing would be the primary evaluation method with confirmatory exterior exposures of the better coating systems. This scheme has been followed. Since 3% salt spray was selected in preference to 20%, it was felt that humidity cabinet tests were of less importance. Actually with a salt concentration as low as 3%, considerable information on moisture resistance is obtained during the salt spray exposure.

Normally a salt spray cabinet maintained at 20% salt and 95° F is less severe on paint coatings than is one maintained at the same temperature but at 3% salt when the effect on coatings is considered. This is felt to be primarily because of the greater osmotic effects with the latter. It was found, however, that with the magnesium-aluminum coupled panels used in this project, and probably with other dissimilar metal couples, the 20% salt was considerably more corrosive (See Section A, Table 1 of the Appendix). Such an effect with a dissimilar metal panel can be explained by the greater conductivity of the 20% salt spray.

The question of which testing atmosphere was the more desirable, 3% or 20%, was considered in the light of these results. The slower rates of corrosion in the 3% spray are more conducive to differentiation between the performance of similar types of coatings. In a research and development project such differentiation is important for following trends and gaining information. Although the more rapid failures in the 20% salt spray might be desirable in control work or rough screening tests, it was not felt that they would be valuable in a project of this type.

A standard Industrial Filter and Pump salt spray cabinet was used in this project. Exposure panels were mounted at an angle of 15° from the vertical with the magnesium panel on top. The lapped edges were placed in a horizontal position so that the salt solution was shed over the magnesium panel onto the aluminum. The cabinet was operated in accordance with ASTM specification B 117-49T with the exception of the salt concentration.

In those few cases where a humidity test was used a specially designed humidity cabinet using the cold wall principle was employed. Actually there is no accepted standard humidity cabinet in the paint industry. It is contended that any cabinet that does not maintain copious condensation on all parts of all panels all the time does not operate continuously at 100% relative humidity. In order to maintain 100% relative humidity a cabinet must be designed to introduce more water into the atmosphere than is theoretically necessary. This can be accomplished by using a cold wall or by introducing droplets of water by means of a spray. In the cabinet used in this project the cabinet walls were maintained at 90° F while the atmosphere was at 110° F. Failures would be expected to be more severe than in a bubbler type cabinet operated at 105° to 110° F.

Exterior exposures have been conducted on 6 x 12 inch panels of magnesium and aluminum finished and coupled as described previously. They have been exposed in Florida at 45° south under the auspices of contract AF 33(600)-22654. Ratings have been made monthly on such characteristics as gloss and corrosion. Adhesion has been rated by a knife scratch test or the tape test specified in MIL-P-6889a. Other tests that have been conducted, particularly solvent resistance, have been run as specified in MIL-P-6889a or MIL-L-7178.

Film thickness measurements were made during the early part of the contract on duplicate steel panels with a GE Magnegauge. However, during most of the project an Aminco Filmeter was available for direct measurements with much greater accuracy. Gloss measurements have been made on a 60° Photovolt gloss meter. Adhesion measurements have been made for the most part by the knife scratch test although some use has been made of the tape test. Unless otherwise specified ratings have been made on an arbitrary basis of 10 - 0, with 10 representing perfect performance and 0 complete failure.

SECTION II

PANEL PRETREATMENT

A. Inorganic Pretreatments for Magnesium

1. Evaluation of MIL-M-3171, Type III

At the start of this project it was agreed that dichromate treatment, MIL-M-3171, Type III would be the standard pretreatment for magnesium panels. This has been carried out. However, it was found that there was a good deal of variation in dichromate treated magnesium surfaces even though the specification was followed. This led to considerable evaluation work, both of panels pretreated in the laboratory and in commercial pretreating establishments.

In all of the work on magnesium pretreatments close cooperation was maintained with personnel of the Magnesium Division of Dow Chemical Company. A fundamental reason for difficulties with dichromate pretreatments of FS-1 magnesium has been advanced by Dow; namely, that the dichromate pretreatment was developed for the magnesium alloys in existence prior to the introduction of FS-1. All of these alloys contained substantially higher concentrations of aluminum (at least 100% higher). Since the deposition of chromate on magnesium depends on the corrosion of magnesium by the acidic chromate solution and since the lower aluminum content of FS-1 makes it less susceptible to such corrosion it can be seen readily that chromate deposition on FS-1 magnesium would be more difficult to induce and to control.

a. Laboratory Pretreatment

Initial efforts were directed toward pretreatment of magnesium panels in the laboratory according to MIL-M-3171, Type III. All pretreatment was done in suitable four liter beakers. Considerable difficulty was encountered in obtaining pretreatments with uniformly good dichromate films. This was attributed, at least in part, to the relatively large ratio of surface treated to the volume of the solutions. This condition led to relatively rapid depletion of the solution chemicals. One factor was established. The pH range allowed by the specification for the dichromate bath was too high, 4.0 to 5.5. It was found that a pH of 4.1 to 4.6 was more satisfactory. This was confirmed by Dow personnel who stated that the pH should be 4.2 to 5.0.

b. Commercial Pretreatment

Although a fairly satisfactory method for laboratory dichromate treatment of magnesium panels was established it was found that this operation was excessively time consuming. This was due in part to the small scale of the laboratory set up and in part to the necessity of repeating the operation in many instances. A decision was made to have 500 panels pretreated by a commercial establishment. Following Dow's advice this was done by a Chicago concern under the observation of one of the project's chemists according to the procedure outlined in Section B of the Appendix. Although this concern was apparently proficient in such pretreatment operations the results were not satisfactory from either an appearance standpoint or a performance test. (See Section A, Table 2 of the Appendix). Visual observation of the treated panels showed that the marking ink on the magnesium had not been removed completely by the cleaning operation and now was present as a loose powder on the surface of the magnesium. In addition the panels were generally lighter in color than normal and exhibited a slight greenish cast after aging.

Salt spray tests were run on replicate panels of both the commercial pretreated and laboratory pretreated panels using the specification finishing system, MIL-P-6889a with MIL-L-7178, aluminized. The results were quite disappointing. (See Section A, Table 2 of the Appendix.) The commercially pretreated panels allowed early blistering (24 to 48 hours) due to water sensitivity and relatively rapid severe failures in corrosion. While the laboratory pretreated group of panels showed no blistering and reasonable corrosion resistance, neither group of panels showed good reproducibility. The differences noted between supposedly replicate panels were of such a magnitude that it was doubtful if any significant differences could be detected in paint formulations.

c. Variations to Increase the Reproducibility

For this reason various means of increasing the reproducibility of the test method were investigated. The most obvious method would be to increase the number of test specimens to a point where statistical analysis would apply. Another method would be to increase the severity of the test to a point where differences in pretreatment would become minor -- such as removing the coating from the lapped edges of the panels. A third possibility was to eliminate the pretreatment of the magnesium or to eliminate some of the final steps in the pretreatment of the magnesium -- such as the dichromate treatment and the hydrofluoric acid treatment. A fourth, more drastic, method was also considered -- the elimination of the bimetallic couple and the substitution of an alkali resistance test for the salt spray test.

The latter method was dropped except as a last resort because of the difficulty expected in establishing the relationship of results to actual performance. However, the other methods were evaluated by finishing five test assemblies each with the specification coating system and with an ether ester system using the following pretreatments on the magnesium.

(1) Standard -- MIL-L-3171, Type III
(2) Chromic acid pickle
(3) Chromic acid pickle plus a hydrofluoric acid treatment
(4) MIL-M-3171, Type I
(5) MIL-M-3171, Type II
(6) MIL-M-3171, Type III with the paint removed from the lapped edge of the magnesium panel

Salt spray tests on these show that the bare magnesium panels, numbers (1) and (2), showed even more variation in results than did the standard MIL-M-3171, Type III panels. This was true for both types of finishing systems (See Section A, Tables 3 and 4, Appendix.) Similarly, Types I and II of MIL-M-3171 showed inferior reproducibility compared with Type III, especially when the latter was applied in the laboratory (See Section A, Tables 5 and 6, Appendix.) However, it was shown that increasing the severity of the test by removing the paint film from the lapped edge of the magnesium panel did improve the reproducibility. In addition it was shown that such an increase in severity still allowed differentiation between paint systems, at least when the differences were as great as between the specification system and an ether ester system. (See Section A, Tables 7 and 8, Appendix.)

The results thus far then indicated that MIL-M-3171, Type III pretreated magnesium tended to give more reproducible results than did either bare or other dichromate treated surfaces. Pickled magnesium surfaces, particularly when treated with hydrofluoric acid, tended to corrode quite rapidly and non-uniformly. Other dichromate treated surfaces do not give as good salt spray resistance as does MIL-M-3171, nor are they as uniform. This is at variance with the view held by Dow that any dichromate deposit on the surface of magnesium will be an effective inhibitor, but it does not necessarily controvert the idea that properly prepared deposits also will serve as an excellent paint anchor.

In selecting the type of panels to be used during the remainder of the project, both the reproducibility of the test results and the degree of departure from practical systems was considered. From these standpoints the continuation of an MIL-M-3171, Type III panel was favored without any intentional damaging of the film until the test had progressed for some time. In order to increase the reliability of test results, therefore, the number of replicate panels was increased to at least three panels for each variation tested.

At this time another commercial concern was contacted for application of the dichromate pretreatment on the advice of Dow personnel. This concern pretreated a batch of panels using three different methods of surface preparation, namely

(1) Mechanical buffing or satin finish -- to reclaim unsatisfactorily pretreated panels
(2) Chromic acid pickle
(3) Nitric acid pickle

Salt spray tests on magnesium-aluminum panel assemblies using these pretreated magnesium panels with the specification finishing system showed them to have appreciably better corrosion resistance but relatively poor moisture resistance, particularly the nitric acid pickled panels. The mechanically buffed panels were an exception in that they showed both good corrosion resistance and moisture resistance. (See Section A, Table 9, Appendix.)

Inasmuch as it appeared from these results that there may have been excessive water solubles remaining on the pretreated surface after the rinsing operation of the pretreatment, it was decided to investigate the effect of additional rinsing operations. Initial salt spray tests using running tap water rinses of 30 minutes and eight hours duration, respectively, showed promise of controlling blistering and greatly improving initial adhesion. However, this same test indicated that such rinsing operations tended to reduce the effectiveness of the dichromate pretreatment in corrosion inhibition. Thus it appeared that the water solubles in the original dichromate pretreatments not only served to increase the blistering tendencies of subsequent paint films but also acted as corrosion inhibitors. (See Section A, Table 10, Appendix.) A recheck on this experiment using two different ether ester finishing systems as well as the specification system failed to confirm these results. In fact this experiment indicated that additional water rinsing of dichromate pretreatments tended to reduce the moisture resistance of subsequent paint films. (See Section A, Table 11, Appendix.)

Another recheck was conducted using only the specification painting system but elaborating on the rinsing operations. Here a distilled water rinse was added to the half hour tap water rinse; a toluene rinse was added to the half hour tap water rinse, and a toluene rinse was used alone. The latter showed the best performance but even here the superiority over the control panels disappeared within 500 hours of salt spray exposure. (See Section A, Table 12, Appendix.) From these results it was decided to incorporate a toluene rinse into the standard panel preparation procedure in the form of a spray rinse.

Since the mechanically buffed or satin finish panels evaluated previously were only a sample lot, another evaluation was run on a production lot of this type of pretreated magnesium panel. Two different types of magnesium panels were reclaimed in this lot, one that had been inadvertently etched with a phenolic base cutting fluid and another that had been dichromate pretreated unsatisfactorily. This production pretreatment was not satisfactory in contrast to the sample lot in salt spray tests. (See Section A, Table 13, Appendix.) Therefore these panels were not used for evaluation work.

Reasonable reproducibility was obtained through most of the project using panels pretreated by the New York concern. The third lot of panels received from this source obviously was not satisfactory. Visual observation showed incomplete dichromate films, powdery dichromate films, and contamination with a yellow chromate. These panels were rejected for evaluation work and a new lot was obtained. However, there was no basis for refusing to accept these panels as being inadequately treated, since the specification does not include any check test.

d. Cooperative Study With Dow Chemical

The disparity of results this laboratory obtained with dichromate treatments on magnesium was discussed with the Magnesium Division of Dow Chemical Company. Dow has not encountered such variations in similar work in their laboratories. In order to determine why this difference in results existed, duplicate tests on multiple panels were run in the two laboratories using the same paints and the same lot of pretreated magnesium panels for checking adhesion. In this case the agreement between laboratories was good but a definite advantage was discovered for the paint system furnished by Dow although both paint systems fulfilled the specifications. (See Section A, Table 14, Appendix.) Since the main difference in paint formulations from the two sources was the fact that the better primer was Type II and the poorer primer was Type I, an additional test was conducted using similar primers from the same producer. In this study it was shown again that better adhesion and better uniformity of adhesion was obtained using a primer that contained some phenolic dispersion resin, either MIL-P-6889a, Type I Control or Type II. (See Section A, Table 15, Appendix.) It should be pointed out that because of cost considerations any bid on MIL-P-6889a, Type I will be filled with a primer that does not include the phenolic dispersion resin.

2. Pretreatments Other Than Dichromate

At the request of the Air Force certain other types of pretreatments for magnesium have been evaluated briefly.

a. Caustic Anodize

A tentative specification, MIL-M-3171A, Type V describes a magnesium pretreatment that comprises a hydrofluoric acid treatment followed by anodizing in an alkaline solution with either direct or alternating low voltage current. Panels pretreated in this manner by the Materials Laboratory were evaluated for initial adhesion and salt spray performance. A standard magnesium-aluminum couple was used with the specification finishing system, MIL-P-6889a and MIL-L-7178. In general this evaluation, along with similar evaluations in comparison with wash primer under vinyl systems, showed that although caustic anodizing offered a good base for the adhesion of organic coatings it is not as good as dichromate pretreatments for either corrosion or moisture resistance. (See Section A, Tables 16, 17, and 18, Appendix.)

b. Iridite #15, Mag-Coat

Allied Research Products furnished a group of magnesium panels pretreated with Iridite #15, Mag-Coat which is a simple chemical pretreatment that can be applied by a single dip or brush application. As such it was compared with MIL-M-3171, Type I, as well as with Type III. The test panels were finished as standard magnesium-aluminum assemblies with the specification system, MIL-P-6889a, Type I, and MIL-L-7178, Aluminized. Salt spray tests showed the Iridite pretreated panels to be weak in moisture resistance when compared with Type III pretreatment but about equal to Type I. In corrosion resistance the Iridite was inferior to both the Type I and Type III. The initial adhesion over the Iridite was good, actually better than that over Type III and equal to that over Type I. The differences between Type I pretreatment and Iridite were slight. (See Section A, Table 19, Appendix.) It is not felt that the Iridite #15 pretreatment should be accepted or rejected on the basis of this one evaluation test.

c. New Anodic Pretreatment

A relatively new method of pretreating magnesium has been developed by Dow Chemical Company, namely, Dow 17. This method of treatment employs the use of applied electrical current and as such resembles the tentative MIL-M-3171A, Type V and HAE procedures. Dow furnished the contract with magnesium panels pretreated with Dow #17 at two different voltages. Panels pretreated at 75 volts were lighter colored and had a thinner coat than those treated at 90 volts. Dow admitted to certain shortcomings in flexibility with Dow #17 pretreatments. However, they claimed that certain relatively slow drying materials such as spar varnish would tend to "lock" the pretreatment coating to the substrate. Actually it was found that with the pretreatment applied at 75 volts, good flexibility was obtained regardless of the finishing system, while with the 90 volt pretreatment two relatively fast drying primers, MIL-P-6889a, Type I, and a styrenated ether ester, gave better flexibility than did the spar varnish. (See Section A, Table 20, Appendix.)

Comparisons of Dow #17 pretreatments at 75 and 90 volts with MIL-M-3171, Type III pretreatments under a styrenated ether ester system in salt spray tests show them all to be good. With a specification finishing system the 75 volt Dow #17 pretreatment has a slight advantage, both in uniformity of results and in the quality of the protection given. (See Section A, Table 21, Appendix.) However, it was not considered that these advantages of Dow #17 were sufficient to justify changing the type of standard pretreatment for this contract.

B. Pretreatment of Aluminum

During the early part of this project the aluminum panels were simply solvent spray cleaned. It was noticed that adhesion results on this type of surface were quite variable. For this reason a switch was made to an alcoholic phosphoric acid etch with no deposition of phosphate. This type of panel preparation gave better and more uniform adhesion results and was used for the remainder of the project.

Some work was done on the use of Alodine surface pretreatments on aluminum under vinyl copolymer coatings to improve the adhesion. This is discussed in more detail in the section on vehicles.

C. Organic Pretreatments for Magnesium

If an organic coating such as wash primer could be utilized in place of inorganic chemical pretreatments of magnesium, it would be quite beneficial. The organic coating would lend itself more readily to field application and even in production it could very well be more uniform and reliable than the present dichromate pretreatment. With this in mind, a considerable effort was made to adapt wash primers for the pretreatment of magnesium.

The wash primer pretreatment for metals generally is considered favorably by the coatings industry over steel, galvanize and certain other metals. Over magnesium, however, there have been difficulties arising from the greater reactivity of the acid component with the magnesium. This reactivity apparently leads to the formation of sufficient hydrogen gas to form visible blisters that are trapped in the dried wash primer film. Although such blisters have not been found to cause significant degradation in performance, their presence probably is undesirable on aircraft skins from an aerodynamic standpoint as well as from an appearance standpoint.

For the purposes of this project a wash primer has been considered as an organic coating formulation based on a basic pigment, a hydroxyl containing resin and a polybasic acid. Most of the investigational work has been done with formulations along the lines of MIL-C-15328a which is based on basic zinc chromate, polyvinyl butyral and phosphoric acid. Several lines of investigation have been followed in attempts to eliminate or control the residual blistering resulting from hydrogen evolution.

1. Hydrogen Evolution

a. Aging of Activated Wash Primers

It is well known that wash primers lose their effectiveness in attaining adhesion to metals within limited periods of time after the acid activator has been added to the resin and pigment base[4]. Presumably this results from certain reactions between these three components[5]. One thought was that at some period after the addition of the acid component there might be a balance of reactivity so that no blisters would result from hydrogen evolution while there would still be sufficient reactivity to gain adhesion between the magnesium and the wash primer film. While it was found that sufficient aging of the activated wash primer (24 hours in one case) did eliminate all visible blistering, it also was found that such wash primers had little or no adhesion to magnesium. (See Section A, Table 22, Appendix.)

b. Variation in Activator Content

One method of controlling the reactivity of wash primers with magnesium substrates would be to reduce the activator content. This was tried with a wash primer with variations in the activator content from 25% of normal to 175% of normal. Variations in hydrogen evolution when applied over alkaline cleaned magnesium were noted, but these variations did not show any consistent relationship to the acid concentration. (See Section A, Table 23, Appendix.)

Water content has been found to be critical in wash primers as far as package stability is concerned. Unless the water content of wash primers is kept between rather narrow limits gelation is apt to occur immediately upon reduction with the phosphoric acid activator. This would indicate that water might play a very important role in the basic wash primer reactions and conceivably could be critical in the amount of reaction with magnesium substrates. To investigate this the water content of two different wash primers was varied from considerably above normal to considerably below normal over two different types of magnesium surfaces. Over the more reactive magnesium surface (acetic acid-nitrate pickle) it was found that about 75% water content based on the phosphoric acid gave the least hydrogen evolution blistering. These wash primers with 75% water (based on phosphoric acid) still showed excellent adhesion to magnesium. (See Section A, Tables 24, 25, and 26, Appendix.)

This influence of water content on the activity of a wash primer with magnesium led to a reconsideration of the activator content of wash primers with a recognition of the total water content. Water is introduced into wash primers through the use of solvents containing a small percentage of water, through the use of phosphoric acid and through the addition of water as such to the formula. The wash primer used in the initial activator study had sufficient water in its pigmented base to account for half of its total water content after activation, or 50% based on the phosphoric acid. A wash primer fulfilling specification MIL-C-15328a, on the other hand, had only one-tenth of the total water in its base. Thus as the activator content of the proprietary wash primer is reduced the ratio of water to acid increases rapidly whereas as the activator content of the specification wash primer is reduced this ratio increased relatively slowly. Since it is considered that this ratio of water to acid can influence the rate of the wash primer reaction considerably, the specification wash primer was selected for further studies. It was found that over active (acetic acid-nitrate pickle) and normal (abraded) magnesium surfaces a concentration of 25% of the normal activator content gave little or no hydrogen evolution blistering. At an activator concentration of 25% of normal this wash primer had a water content of 150% based on the phosphoric acid. (See Section A, Tables 27 and 28, Appendix.)

Salt spray tests with wash primers over bare magnesium using 25% of the normal activator have shown variable results. Generally such a surface appears to offer poor moisture resistance. Early results of the same wash primer over a dichromate treated surface indicated considerable promise. (See Section A, Tables 29 and 30, Appendix.) Repeat evaluations with such a double pretreatment again were contradictory, sometimes improving corrosion and moisture resistance but sometimes tending to degrade these characteristics. (See Section A, Tables 31 and 32, Appendix.)

c. Effect of Film Thickness

At least two other investigators have suggested that the hydrogen evolution blistering in wash primer films could be eliminated by the use of very thin films. This laboratory did not concur in these findings. Films were applied to bare magnesium by spray at less than 0.1 mil thickness and blistering was still evident. The blisters at such low film builds were quite small and difficult to detect but they were not diminished in frequency. In another test films were laid down by means of a wedge shaped drawdown gage which varied from 0.0 to about 2.5 mils in wet film thickness. No substantial differences were noted in hydrogen evolution blistering.

d. Effect of Atmospheric Moisture

A great deal of difference was noted from day to day in the amount of hydrogen evolution blistering when wash primers were applied to magnesium, even though conditions were held as constant as practicable. One explanation for these differences was the variation in relative humidity. In an attempt to substantiate this theory wash primers were sprayed over bare magnesium surfaces that had been pre-wet with water by exposure in the humidity cabinet which deposited droplets of water on the entire surface, and over panels that had been dried in an oven. No significant differences were noted in the amount of hydrogen evolution blistering. (See Section A, Table 33, Appendix.) A repeat test with less drastic conditions was conducted. Here the panels were pre-wet by cooling them to -10° F and then exposing them to the atmosphere. Again heated panels were used as a control. Here also no significant differences were noted in hydrogen evolution blistering. (See Section A, Table 34, Appendix.)

e. Effect of Alkyl Phosphates as Activators

Hydrogen dialkyl phosphates and dihydrogen alkyl phosphates were investigated as substitutes for the phosphoric acid activator in wash primers. The thought was that the reduced acidity of these phosphates might eliminate the hydrogen evolution blistering on magnesium while still maintaining an ability to gain adhesion. A series of 14 alkyl phosphates with varying chain length alkyl groups from methyl to lauryl was selected and investigated. These phosphates were substituted for the phosphoric acid activator in a wash primer on an equivalent basis. These wash primers were then used in place of the MIL-M-3171, Type III pretreatment over magnesium for standard panel assemblies finished with an MIL-P-6889a, Type I, - MIL-L-7178, aluminized, system.

It was found that none of the alkyl phosphates caused sufficient hydrogen evolution with magnesium to result in blistering of the film. Although good adhesion was attained with the shorter chain length alkyl phosphates it was found that all of the alkyl phosphate activated wash primers tended to blister to some extent during salt spray testing. This would indicate the presence of more water soluble materials from the use of alkyl phosphates than from the use of phosphoric acid. The soluble materials could be either unreacted alkyl phosphates or soluble reaction products. The best performance was shown by mono ethyl acid ortho-phosphate activated wash primer, but even this system showed blistering in the salt spray test. (See Section A, Table 35, Appendix.)

f. Use of Non-Oxidizable Solvent

Rosenbloom[5] has theorized that the alcohol solvents in wash primers enter into a redox reaction in which the chromium is reduced to some intermediate valency and the alcohol is oxidized. The intermediate valency chromium ions tend to form complexes with the polyvinyl butyral resin. This is, then, the main wash primer reaction. If the alcohol solvents of normal wash primers were replaced with a less oxidizable solvent, such as tertiary amyl alcohol, then there would be much less chance for chromium to be reduced. Such wash primers should be stable. Rosenbloom found no pH change in such wash primers but he also found that they lacked the ability to gain adhesion to metals. An attempt was made to duplicate his work, since wash primers with reduced activity should be of interest over magnesium. It was found that a wash primer based on MIL-C-15328a but using tertiary butyl alcohol as the solvent gelled solid within 30 minutes after the addition of the activator.

g. Elimination of Residual Blistering

Since a method had not been found to control consistently the amount of hydrogen evolution while still maintaining adhesion and performance, a method was sought to eliminate the hydrogen from the wash primer film without leaving any disruptions in the film, such as blisters. One avenue that was followed was an attempt to keep the wash primer film open until all of the hydrogen had escaped from the film by the use of slower evaporating solvents such as butyl alcohol. When this was tried the results were inconclusive since neither the control with normal solvents nor the test wash primer with butyl alcohol showed sufficient hydrogen evolution to be visible.

Another possible means of dissipating the hydrogen would be to change the surface characteristics of the wet film in such a way as to facilitate the release of any bubbles that are formed. Silicone oils are known to affect liquid surfaces very strongly. Therefore, a methyl oil DC 200, 500 centistokes, was added to the specification wash primer at varying concentrations (from 0.0004 to 0.2% by weight). When these wash primers were sprayed over bare magnesium the residual hydrogen blistering decreased with increasing silicone content until at 0.2% there was no visible blistering. This investigation was repeated to confirm the results but in this case the control level of blistering was too low to give conclusive results. Neither salt spray tests nor adhesion checks have shown any major deleterious effects from the use of silicone oil. In no case was there any evidence of pock marking, crawling, or other surface defects in either the wash primer, a specification zinc chromate primer applied over the wash primer or in a subsequent aluminum lacquer topcoat. (See Section A, Tables 36 and 37, Appendix.)

One theory as to why the comparatively large amount of silicone oil in wash primers did not develop other problems, hinged on the compatibility of silicone oil and polyvinyl butyral resin. If silicone oil and polyvinyl butyral are compatible the silicone oil would be distributed uniformly throughout the film. Most other resins are incompatible with silicone oils, which tends to concentrate the oil at the surface. It would seem that the actual concentration at the surface would be the determining factor for adhesion or cratering effects. A check on the compatibility of DC 200, 500 ctsk., with polyvinyl butyral, polyvinyl acetate-chloride copolymer, and an alkyd at 0.2% DC 200 showed that all were incompatible to about the same degree. It was necessary to use the Tyndall effect to detect the incompatibility.

h. Discussion

It is obvious from the foregoing discussion of hydrogen evolution blistering that all of the factors that affect wash primer reactions are not understood. Thus variations occur between duplicate experiments that cannot be explained with the present knowledge. In spite of this, progress appears to have been made both in regulating the amount of hydrogen evolved and in eliminating such hydrogen as is generated without disrupting the wash primer film. A contingent problem was one of evaluating such wash primers to determine if they offered any real advantages, either in place of or as an addition to the dichromate pretreatment. The results in these latter cases were not consistent. Generally, wash primers applied directly over bare magnesium show about the same corrosion resistance as MIL-M-3171, Type III, but somewhat poorer moisture resistance. When used over dichromate treated magnesium surfaces wash primers usually increase the corrosion resistance but may lower the moisture resistance.

2. Variations in Wash Primer Formulations

All of the work on wash primers discussed previously has been based on essentially the same formula, namely: one part of polyvinyl butyral resin, XYHL; one part of pigment, basic zinc chromate; and one-half part of phosphoric acid. Any variations in formulation have been minor, such as solvent composition, per cent water or additives in small percentages. Considerable work was done also which encompassed major changes in the wash primer, such as pigmentation, type of vehicle or a combination of these two. Initial work along these lines was based on wash primer formulations that had shown promise over metals other than magnesium. These formulations generally were developed by the resin manufacturer concerned.

a. Special Wash Primer Formulations

One of these formulations was based on a polyvinyl butyral resin with a lower polyvinyl alcohol content, Butvar B-76, modified with Resimene 881. Such a wash primer has shown good performance on aluminum. Another formulation was based on a polyvinyl formal resin, Formvar F-770, pigmented with basic zinc chromate. This formulation has somewhat better pot life than the specification wash primer and should be less sensitive to water in its formulation. Another formulation was based on polyvinyl butyral pigmented with a chromium phosphate pigment. This type of primer uses only 40% as much acid activator as does the specification wash primer and as such should be less reactive with magnesium. In addition it has shown good package stability in the activated form. A fourth formulation was based on a polyvinyl butyral resin XYHL, that had been specially reacted with a mixture of chromic acid and phosphoric acid according to Bakelite's formula XL 5441. This primer was pigmented with basic zinc chromate and has shown fair package stability. No additional acid is used in this formulation. (See Section C, 1 of the Appendix.)

All of these special wash primers were evaluated over bare magnesium and over MIL-M-3171, Type III, treated magnesium. A standard magnesium-aluminum couple was used with a specification finishing system, MIL-P-6889a plus MIL-L-7178, Aluminized. In these tests as in many subsequent tests with wash primers the results of salt spray exposures were not consistent. In the initial evaluation there appeared to be a decided advantage for the one-package type of wash primer, the chrome phosphate pigment and the chromic-phosphoric acid treated polyvinyl butyral resin. (See Section A, Table 38, Appendix.) In a repeat evaluation over bare magnesium this advantage was lost. At the same time in an evaluation over dichromate treated magnesium, the chromium phosphate pigmented wash primer gave outstanding performance. (See Section A, Table 39, Appendix.)

The preceding tests on the chrome phosphate wash primer were carried out with a sample of pigment that subsequently was found to be inferior. Therefore, additional work was done with pigments obtained from du Pont and from the Sherwin-Williams Dry Color Department, both of which had been found to be satisfactory in wash primers over steel. These pigments were incorporated into two different wash primers, one based on a polyvinyl butyral resin, XYHL, and one on a chromic acid-phosphoric acid treated polyvinyl butyral resin, XL 5441. (See Section C, 1, c and d, Appendix, for type formulations.) Each of these four wash primers were tested over dichromated and bare magnesium panels with the normal aluminum couple and specification finishing system.

Salt spray exposures showed those primers based on polyvinyl butyral resin to be markedly superior to those based on treated polyvinyl butyral resin in original adhesion, moisture resistance and corrosion resistance. There appeared to be little to choose from between the two chrome phosphate pigments. A specification wash primer, MIL-C-15328a, with 25% of the normal activator was included in this series for comparison purposes. Although it showed about as good corrosion resistance as the chrome phosphate wash primers, its moisture resistance was relatively poor. Another type of wash primer, based on polyvinyl butyral resin and a B stage phenolic resin, also was included. This wash primer showed poor initial adhesion to dichromate treated magnesium and poor moisture resistance over bare magnesium. (See Section A, Tables 40 and 41, Appendix.)

It should be pointed out that the chrome phosphate pigmented polyvinyl butyral resin described above does not spray well at normal reductions. In addition it was found in subsequent work that the adhesion of such a wash primer to a dichromate treated magnesium surface was not consistently good.

b. Variation in Polyvinyl Butyral Resin

MIL-C-15328a wash primer calls for a polyvinyl butyral resin with a vinyl alcohol content of 18-20% with a certain intrinsic viscosity. Until recently there was only one commercial resin that fulfilled these requirements, Bakelite's XYHL. There was another commercial polyvinyl butyral resin on the market that could be used to prepare a satisfactory wash primer, Monsanto's Butvar B76-1. Other investigators have found that wash primers made from this lower vinyl alcohol content resin (11.5 - 12.5%) gave better moisture resistance over steel surfaces. With the thought that this improved moisture resistance might be effective also on magnesium an evaluation was carried out on MIL-C-15328a type wash primers prepared from the two resins, XYHL and Butvar B76-1. Both wash primers were applied to bare magnesium with 25% of the normal acid activator. Both showed considerable hydrogen evolution blistering, with no advantage for either resin. Evaluations were run on standard magnesium-aluminum couples using a specification finishing system, MIL-P-6889a, Type I - MIL-L-7178, Aluminized. Salt spray tests showed no advantage for the low hydroxyl content resin. Actually the blistering was more severe with this wash primer while the corrosion was about the same for both wash primer systems. Adhesion was excellent with either wash primer. (See Section A, Table 42, Appendix.)

A new polyvinyl butyral resin is available now which also fulfills the requirements of MIL-C-15328a for vinyl alcohol content and intrinsic viscosity. It is Monsanto's Butvar B-90. The characteristics of this resin in comparison with XYHL and Butvar B76-1 are shown below.

Identification	Sp. G.	Polyvinyl Acetate (Maximum)	Polyvinyl Alcohol Min.	Polyvinyl Alcohol Max.	Viscosity Cp.
Bakelite XYHL	1.12	0.3	18	20	13-18
Monsanto Butvar B76-1	1.10	1.5	10.5	13	>23
Monsanto Butvar B-90	1.10	1.0	18	20	13-18

An initial evaluation on MIL-C-15328a type wash primers prepared from these resins was run over bare magnesium using a standard magnesium-aluminum couple finished with a styrenated ether ester system. Salt spray tests showed the Butvar B-90 primer to have an edge over XYHL in moisture resistance, particularly in the early stages. Butvar B76-1 primers were even more deficient than XYHL primers in this respect. However, no differences in adhesion or corrosion resistance were detected. (See Section A, Table 43, Appendix).

In a repeat test using the XYHL and Butvar B-90 wash primers over bare and dichromated magnesium surfaces under a styrenated ether ester system and the specification system, a slight advantage was shown for the Butvar B-90 wash primer in both moisture and corrosion, This advantage was so slight as to classify the two wash primers as about equal in performance. The styrenated ether ester system was substantially superior to the specification system and the dichromate treated magnesium surface offered substantially better moisture and corrosion resistance under all systems. (See Section A, Table 44, Appendix.)

c. B Stage Phenolic Resins

It was found that B stage phenolic resins mixed with a polyvinyl butyral resin exhibited good compatibility as well as good film character. Upon further investigation, it was shown that shellac could be added to the mixture without impairing the compatibility. This three phase system gave better physical characteristics in clear films. A mixture containing 90 parts of B stage phenolic resin, 10 parts of polyvinyl butyral resin and 10 parts of orange shellac was found to have the best properties in clear films. This and a similar mixture containing 25 parts of shellac were pigmented with strontium chromate and applied over bare and dichromate treated magnesium and evaluated both as pretreatments and as normal primers.

In general it was found that these primers were not promising. It appeared that pigmentation of these formulations tended to detract from their adhesion and flexibility, to a point where they were inferior to the specification wash primer in this respect. They also showed no substantial advantage over a normal wash primer in moisture resistance. Unfortunately, the formulation that showed the better water resistance (10% shellac) also showed the poorest physical characteristics. (See Section A, Table 45, Appendix.)

d. Pigmentation

As discussed in another section of this report certain chromate pigments were found to be the better inhibitors of corrosion for magnesium out of about 25 types of common paint pigments. In addition one inert pigment, silicon dioxide, showed unusual inhibitive action on magnesium. MIL-C-15328a type wash primers were prepared in which the various promising chromate pigments were used to replace the basic zinc chromate on a volume basis and silicon dioxide was used to replace the magnesium silicate, also on a volume basis. The chromates were:

Zinc Chromate
Calcium Chromate
Strontium Chromate
Barium-Potassium Chromate

These wash primers were applied to bare magnesium panels which were then used in a standard magnesium-aluminum assembly, finished with the specification system MIL-P-6889a, Type I - MIL-L-7178, Aluminized. All of these wash primers showed excellent adhesion to magnesium. Salt spray tests showed a considerable advantage for the strontium chromate pigmented primer in moisture resistance and reasonably good corrosion resistance for all of the wash primers. The barium-potassium chromate pigmented wash primer was slightly the poorest of the experimental wash primers in corrosion resistance. In this case the silicon dioxide showed no advantages over magnesium silicate as the inert. (See Section A, Table 46, Appendix.)

3. Film Thickness

Normally wash primers are applied at film thicknesses of 0.2 to 0.5 mils. It was thought possible that increasing this film thickness substantially might help to improve the moisture resistance of wash primer systems over bare magnesium. To evaluate this possibility panels were prepared with wash primer coats varying in film thickness from 0.4 to 2.0 mils. An MIL-P-6889a, Type I - MIL-L-7178, Aluminized, system was used over the wash primers and over dichromate treated magnesium as a control. In addition another control was included using a 0.4 mil film of wash primer over dichromate treated magnesium under the same specification finishing system.

Checks on the adhesion of these systems showed little variation over magnesium but a considerable decrease over steel as the film thickness increased. Early salt spray results indicated that increasing the film thickness of wash primers does improve the moisture resistance of the system. As the salt spray tests progressed the advantages for the heavy wash primer films decreased until only the 1.5 to 2.0 mil films were effective. The advantages that were gained in moisture and corrosion resistance were not great and probably would not justify the extra film thickness. (See Section A, Tables 47 and 48, Appendix.)

4. Performance

There have been a number of instances where the specification wash primer with 25% of the normal activator content has been evaluated as a magnesium pretreatment as a secondary purpose of a series. The results of these evaluations will be reported here. In one instance wash primer was evaluated in comparison with MIL-M-3171, Type III and with bare magnesium under a 50:50 VMCH:VAGH vinyl chloride-acetate copolymer system, a 25:75 VMCH:VAGH system, a VAGH system, and a styrenated ether system. All finishing systems were used at three different film thicknesses, four coats, two coats and one coat. In salt spray tests the wash primer pretreatment was as good as or better than the dichromate pretreatment or the bare magnesium in corrosion resistance and in most cases the same holds true for moisture resistance. Particularly good moisture resistance was evident with the 25:75 VMCH:VAGH finishing system. Adhesion also was excellent with the wash primers. (See Section A, Table 49, Appendix.)

In another instance a wash primer was used under a vinyl alkyd system and the specification system. The initial adhesion was excellent in both cases. The vinyl alkyd system showed fair to good corrosion resistance but poor moisture resistance while the specification system was just the reverse. (See Section A, Table 50, Appendix.) Furan resin coatings have shown their best performance over wash primed magnesium from an adhesion and corrosion resistance standpoint but such a system is subject to early failures in moisture resistance. (See Section A, Table 51, Appendix.)

In a comprehensive series of tests near the end of this project designed to summarize the performance of the more promising systems that had been developed, a number of wash primer systems were included. These systems were varied. The wash primers were applied over bare magnesium, dichromate treated magnesium, and over acid etched aluminum. Finishing systems included the specification system, a vinyl copolymer system, and an ether ester primer under a vinyl copolymer. In general the performance of the wash primer systems was good when they were applied over dichromate treated magnesium. The performance under an unmodified vinyl copolymer system where the wash primer was applied over both the dichromate treated magnesium and the aluminum panels was particularly good. (See Section A, Tables 52 and 53, Appendix.)

SECTION III

VEHICLES

The organic binder or vehicle portion of any paint film could be called the most important ingredient. In any paint film that exhibits some degree of gloss, the vehicle is the outer layer and thus is the portion that comes into direct contact with the substrate and the atmosphere. In addition the vehicle forms around each pigment particle a layer of tightly adsorbed molecules which tend to determine the dispersion qualities of the pigment. Thus from the time a paint is dispersed until it has deteriorated in the film, the vehicle is surrounding, suspending and holding the pigment. Certainly the pigment portion of a paint film is important from other than esthetic angles, but the vehicle is primarily responsible for such properties as adhesion, flexibility, and chemical resistance.

At the beginning of this project it was theorized that the alkali resistance of magnesium primers should be a prime factor in the performance of that primer. This was based on the high alkalinity produced by the corrosion products of magnesium, the oxide and hydroxide. With this in mind, vehicles were selected for study partly on the basis of their alkali resistance. This theory has been substantiated as the project has progressed, i.e. vehicles appear to perform as magnesium primers in proportion to their alkali resistance.

One of the important considerations in studying vehicles is the pigment volume concentration. For each combination of pigments and vehicles there is a point in their relative values where there is just sufficient vehicle to satisfy the adsorptive capacity of the pigment and to fill the voids between the pigment particles. This has been called the critical pigment volume concentration or CPVC[6]. Any additional vehicle merely serves to hold the pigment particles apart and has little effect on the permeability of the film. Any additional pigment, on the other hand, will cause voids in the film and will cause a rapid increase in permeability. Normally, topcoats are pigmented with a PVC:CPVC ratio of considerably less than one. Primers are pigmented with a PVC:CPVC ratio approaching one. Attention has been paid to this concept in studying primers.

A. Systematic Study of Alkyd Primers

Although undoubtedly much work has been done on the use of alkyd vehicles in magnesium primers, there are no published data available which describe any systematic investigations. Therefore, at the suggestion of the Project Engineer, such a program was undertaken. Because a considerable amount of effort has gone into the development of Sherwin-Williams proprietary alkyd for use in MIL-P-6889a, Type I, this alkyd was chosen as the guide. The pattern of this alkyd was followed as closely as possible with the only variations being those under investigation and those that were necessary to give practical cooking conditions and satisfactory physical characteristics to the resulting vehicle.

1. Preparation

As the first step nine different drying oils were cooked into alkyds. Although the alkyd guide has more than one oil in its composition it was thought advisable to prepare each experimental alkyd with only one oil. The drying oils included in this program were as follows:

Linseed
Soya
Fish
Castor
Safflower
Oiticica
Tung
Perilla
Varsoy

The alkyd guide is prepared with an excess of polyol of about 20% based on theoretical equivalents. This excess results in an acid value of 25-30 in the standard cook. With the more reactive oils, namely, oiticica and raw tung, it was found to be necessary to increase the excess polyol content to about 30-35% to prevent premature gelation. Even with this additional polyol these oils gave alkyds with relatively high acid values. A high excess polyol content also was necessary with perilla oil as the only available sample of this oil was one that had been bodied. The less reactive oils, namely, linseed, soya, fish, castor, and safflower, gave alkyds with relatively low acid values when 20% excess polyol was used. (See Section A, Table 54, Appendix, for the characteristics of these alkyds.)

Since there were no data available on whether an acid value of 25-30 is the optimum, two batches of the guide were prepared, one with an acid value of 29 and one with an acid value of 38. (See Section A, Table 54, Appendix.)

Two polyols have been used extensively in alkyd preparation, namely, glycerol and pentaerythritol. Generally speaking, pentaerythritol with its four reactive hydroxyl groups results in more cross linking than does glycerol with its three hydroxyls. This greater cross linking tends to give better chemical resistance but less flexibility. An attempt was made to prepare alkyds varying from 100% glycerol to 100% pentaerythritol in 25% steps. It was found to be impractical to prepare the 100% pentaerythritol alkyd, and those alkyds with 50% or more pentaerythritol came out with relatively high acid numbers. (See Section A, Table 55, Appendix.)

Another variable in the preparation of alkyds is the ratio of oil to glycerol phthalate. Variations in this ratio affect many properties of alkyds. Among the most important of these effects are chemical resistance and flexibility. Generally speaking, the chemical resistance is inversely proportional to the concentration of the oil, and the flexibility is proportional to the concentration of the oil. To investigate this variable of oil content for magnesium primers three batches of the proprietary type alkyd were prepared; one with the standard oil length, one with 10% additional oil and one with 10% less oil. (See Section A, Table 56, Appendix.)

2. Primer Characteristics

These alkyds with different composition variables were formulated into MIL-P-6889a, Type I, primers. They were examined for characteristics such as viscosity, drying rates, film hardness, and adhesion to dichromate treated magnesium. Some viscosity variations were apparent but were not felt to be too significant. The greatest variations resulted from changes in the type of oil and the type of polyol. The soya alkyd was particularly slow in drying but eventually gave a tough although somewhat soft film. The linseed, castor and perilla alkyds gave soft films but were satisfactory otherwise. The safflower alkyd gave the poorest film in that it tended to crumble rather than ribbon when scratched with a knife. The oiticica, tung, and Varsoy alkyds gave poorer adhesion to dichromate treated magnesium. (See Section A, Table 57, Appendix.)

Replacing glycerol in the standard alkyd with pentaerythritol tended to increase the brittleness of the resultant film as the pentaerythritol content increased. It is probable that this effect could be overcome by proper changes in oil type and length but such an investigation was not undertaken. (See Section A, Table 58, Appendix.) The only other significant effect with other types of alkyd variations was the poor drying rate of an isophthalic alkyd. (See Section A, Tables 59, 60 and 61, Appendix.)

3. Critical Pigment Volume Concentration

As discussed previously, the PVC:CPVC ratio is important in primers. Since apparently minor changes in vehicle can have a gross effect on the CPVC of a paint, the CPVC was determined for each of the alkyd primers.[6] Actually the CPVC's varied very little from that of the primer made with the standard proprietary alkyd. The determination of CPVC is subject to an error of ± 2%[6]. However, in the case of the oiticica primer, there had been sufficient viscosity increase in the package to make the CPVC determination difficult. This might have introduced an error of more than 2% which could account for a relatively high CPVC with this primer. The only other significant deviation is a tendency for pentaerythritol containing alkyds to have lower CPVC's than do straight glycerol alkyds. (See Section A, Table 62, Appendix.) It was considered satisfactory to evaluate these primers without any adjustments of PVC(held at 25% to 35%).

4. Salt Spray Results

All of the primers were subjected to salt spray tests using a standard dichromate treated magnesium-aluminum panel assembly with an MIL-L-7178, Aluminized, topcoat. With the alkyds prepared from the various oils there was little significant difference in performance, although linseed is favored somewhat. Actually the best results were obtained with a production primer using the proprietary alkyd. It would be expected that this proprietary alkyd on which a considerable amount of development work has been done should give the best results. However, it should be pointed out that this development work was done for aluminum primers. (See Section A, Table 63, Appendix.)

From the series on variations in polyol content it was quite definite that high proportions of pentaerythritol favored the salt spray performance. Thus the 75:25 pentaerythritol:glycerol gave considerably better salt spray resistance than either the 50:50 or 25:75 pentaerythritol:glycerol ratios. Acid value, at least within certain ranges, had no effect on salt spray performance. There appeared to be a tendency for better salt spray performance with increased oil length vehicles. Neither a special metallic catalyst nor the use of isophthalic anhydride improved the salt spray performance materially. (See Section A, Tables 63-67 inclusive, Appendix.)

B. Phenolic Vehicles

A resin supplier recommended to this project that certain phenolic varnishes be evaluated in coatings for magnesium7. Two vehicles were prepared and formulated into primers as per the supplier's suggestions. These were a 25 gallon phenolic dehydrated castor oil varnish using a 100% para phenyl phenol resin and a 15 gallon phenolic dehydrated castor oil varnish using a para tertiary butyl phenol resin. The primers were formulated using a pigmentation of 85% zinc chromate and 15% magnesium silicate at pigment volume concentrations of 50%. Over standard dichromate treated magnesium-aluminum panel assemblies these primers showed some promise, exhibiting excellent moisture and corrosion resistance in salt spray tests. However, both also showed a tendency to be brittle. (See Section A, Table 68, Appendix.)

The same two vehicles were reformulated into primers at a PVC of 40%. These primers showed better flexibility but tended to gain hardness very slowly during the drying period. Two additional varnishes were prepared, using a different para tertiary butyl phenol resin, into 15 and 25 gallon dehydrated castor varnishes. Again primers were formulated at 40% PVC. As observed previously, all of these primers plus one formulated at 50% PVC showed excellent corrosion resistance in a salt spray test. However, their moisture resistance was poor. (See Section A, Table 69, Appendix.)

Since it was thought that this excessive blistering might be the result of equipment failure rather than primer deficiencies this evaluation was repeated. Again the moisture resistance was found to be poor while the corrosion resistance was good. (See Section A, Table 70, Appendix.) In view of these unfavorable results and the slow curing of the flexible type of phenolic primers no further work was undertaken.

C. Polyvinyl Chloride-Acetate Copolymer Vehicles

At the beginning of this contract it was noted that vinyl chloride-vinyl acetate copolymer based coating systems had been found to offer the most promise for magnesium protection by at least three independent laboratories, namely Northrup Aircraft, Inc.[8], Naval Air Experimental Station[9], and Dow Chemical Company[10]. In general these investigators concluded that the favorable results with vinyl type coatings were due primarily to the impermeability of vinyl films to moisture along with their excellent inherent chemical resistance. The Sherwin-Williams Company, independent of this contract, had developed two magnesium primers based on vinyl resins plus modifying resins which had shown outstanding performance in salt spray tests. These combined factors led to a considerable amount of effort during the course of the project toward developments with vinyl copolymer resins.

1. Acrylic and Methacrylic Modifiers

Initial tests comparing two modified vinyl copolymer systems with a specification MIL-P-6889a/MIL-L-7178 system and several ether ester systems were encouraging, not only for the vinyls but also for the ether esters. In salt spray tests all of these alkali resistant systems rated 8-9 for corrosion resistance in contrast to 1-5 for the specification control system. (See Section A, Table 71, Appendix.)

In the investigation of vinyl copolymer coatings under this project it was considered probable that chemical resistance played a more important role than impermeability in performance over magnesium.

Because of this belief it was decided to limit the modifying resins and plasticizers to those known to possess excellent chemical resistance of themselves. In this class are the various acrylate, methacrylate and acrylate-methacrylate copolymer resins and the chlorinated diphenyl plasticizers. Actually it was known that excellent coatings can be prepared from vinyl copolymer resins with no modification other than plasticizer. Such coatings can give excellent adhesion to properly prepared metal surfaces but this adhesion is sometimes difficult to obtain. The primary function of the modifying resin or resins was thought to be to increase the probability of good adhesion to dichromate treated magnesium and to aluminum.

The first step in investigating acrylic and methacrylic esters was to determine their compatibility with vinyl resins. Bakelite's VMCH was selected as the vinyl resin. VMCH is a copolymer of vinyl acetate and vinyl chloride modified with a small percentage of maleic anhydride to give the resin polarity. Rohm and Haas's and du Pont's acrylic and methacrylic esters were investigated for compatibility at a 1:1 ratio with one-third part of a chlorinated diphenyl plasticizer, Monsanto's Aroclor 1254. The following results were obtained:

Resin	Supplier	Compatibility
"Lucite" HG-41 - Methyl Methacrylate	du Pont	C
"Lucite" HG-24 - Ethyl Methacrylate	du Pont	C
"Lucite"4- n-butyl Methacrylate	du Pont	I
"Lucite"5- Isobutyl Methacrylate	du Pont	I
"Lucite"6- 50/50 copolymer n-butyl/Isobutyl Methacrylate	du Pont	I
Acryloid A-10 - Copolymer of Acrylic and Methyl-Acrylic Esters	Rohm and Haas	C
Acryloid B-72 - Similar to A-10	Rohm and Haas	C
Acryloid B-82 - Similar to B-72	Rohm and Haas	C
Acryloid C-10 - Similar to A-10	Rohm and Haas	I

The compatible resins were formulated into primers maintaining the same ratio of resins in the vehicle and using strontium chromate as the active pigment. Zinc chromate was avoided because of the known catalytic action of zinc on vinyl resins. Even with strontium chromate it was found that a differential speed two roll rubber mill could not be used for dispersion because the resultant chip was insolubilized. A pebble mill was used instead. Even with this type of dispersion one of the methacrylate modified vinyl primers (Acryloid C-10) gelled before it could be used. (See Section C, e, Appendix, for formula.)

The physical characteristics of the primer containing Lucite HG 41 were the best in this group, particularly for aged films. This, coupled with good salt spray performance over a standard magnesium-aluminum panel assembly, indicated considerable promise for this primer. (See Section A, Tables 72 and 73, Appendix.) Several repeat evaluations of vinyl copolymer systems have shown that although this coating has good performance in salt spray tests, once it starts to fail it fails quite rapidly. This can be explained at least in part by its relatively high degree of impermeability and its tendency to have only fair adhesion. At any rate it has been found repeatedly that vinyl copolymer coatings may maintain excellent salt spray resistance for a period but once failure starts it progresses rapidly. (See Section A, Tables 52, 74, 75 and 76, Appendix.)

It was thought at one time that the reason for this peculiar behavior of vinyl finishing systems could be attributed to the fact that they formed such an impermeable envelope around the pigment particles that inhibitive pigments could not become effective. However, leaching tests in water at room temperature on a number of primer films over glass showed a vinyl copolymer to allow substantially more leaching of chromate color than did other films which showed a normal progression of corrosion.

2. Alkyd Modifiers

Vinyl copolymer coatings modified with alkyd resins have found some favor as coatings for magnesium. A primer based on the vinyl alkyd MIL-P-15934 and an aluminized topcoat based on the same vehicle were evaluated as a system over a standard wash primed magnesium-aluminum panel assembly. MIL-P-15934 has a vehicle composed of 35 parts of a hydroxyl modified vinyl copolymer, VAGH, and 65 parts of a long oil soya alkyd which is pigmented with zinc chromate and magnesium silicate. Salt spray tests showed the vinyl alkyd system to be weak in moisture but somewhat superior in corrosion resistance when compared with the alkyd-lacquer system. No further work was undertaken with this vehicle. (See Section A, Table 50, Appendix.)

3. Pretreatment of Aluminum

The Naval Air Experimental Station at Philadelphia has reported that vinyl copolymer coatings over bare aluminum have shown poor moisture resistance. For instance, they found that after a rainstorm a vinyl film could be stripped easily from a 2 x 3 foot aluminum panel, whereas after 24 hours recovery the adhesion again was excellent. Pretreatments of the phosphate type have been claimed to overcome this deficiency in moisture resistance.

Since vinyl copolymer coatings were considered as possible superior coatings an evaluation was made of two pretreatments for aluminum that could be applied in the field. These treatments were an amorphous phosphate type and a mixed metal oxide type. The control surfaces were wash primed and phosphoric acid etched aluminum. No magnesium couples were employed. Two different types of vinyl finishes were used, a polyvinyl butyral wash primer and a vinyl copolymer system.

The panels were immersed in water at room temperature for 24 and 48 hour periods. In no case did the vinyl systems fail by blistering, but there was considerable loss in adhesion. As the complexity of the pretreatment increased, the loss of adhesion decreased. Thus the mixed metal oxide pretreatment was found to be excellent. (See Section A, Tables 77 and 78, Appendix.)

4. Curing of Hydroxyl Containing Vinyl Resins

One of the shortcomings of thermoplastic resins in general is their lack of solvent resistance. This is true of the vinyl copolymer resins. With the thought that if a certain amount of curing or cross linking could be brought about the solvent resistance would be improved, an investigation was made of the effect of butyl titanate.

Esters of titanates such as butyl titanate are known to hydrolyze very readily in the presence of water. Such hydrolysis also will take place with alcohols even though the alcohols are very high in molecular weight, such as the tripolymer of vinyl chloride-vinyl acetate-vinyl alcohol, VAGH. It is possible that titanium esters could serve to cross link VAGH sufficiently to increase the solvent resistance substantially. XYHL, the polyvinyl butyral resin containing 18-20% polyvinyl alcohol was also included in this study.

Preliminary work was done using VAGH in a mixture of xylene, methyl isobutyl ketone, and anhydrous ethyl alcohol, and with XYHL in anhydrous ethyl alcohol. It was found that the addition of only three per cent of butyl titanate, based on the weight of the resin, caused immediate gelation of both the VAGH and the XYHL solutions. By diluting the catalyst with a solvent that was compatible with both the resin and the catalyst the rate of gelation was greatly reduced. Further reduction in gelation rates was obtained by replacing the ethyl alcohol in part or completely with butyl cellosolve. However, it was found that resins cast from solutions containing from 0.5 to 3.0% butyl titanate showed only slight improvement in solvent resistance. Work was discontinued on this approach.

D. Bisphenol-Epichlorohydrin Condensates

Resins formed by the condensation of epichlorohydrin and the sodium salt of bisphenol recently have gained a prominent place in the coatings industry because of their excellent chemical resistance, adhesion and flexibility. The chemical resistance of these resins, commonly called ether resins, could be predicted from their composition and structure. Ether linkages are known to be relatively inert, showing no reactivity with sodium, phosphorus trichloride or sodium hydroxide. The reactive hydroxyl and epoxide groups are widely spaced along the chain so that cross linked polymers of such ether resins show good flexibility and chemical resistance. Thus esters of ether resins with fatty acids should possess better chemical resistance than alkyds because the relatively weak ester linkages are spaced further apart. Such has been found to be the case.

Ether resins can be cross linked or cured through the use of amine resins such as urea or melamine formaldehyde or phenolic resins but considerable heat is required. Since the Air Force has stipulated room temperature drying for aircraft finishes this type of cure was not considered. Curing can also be accomplished in ether resins through the use of monomeric polyamines, titanium esters, polyamide resins, and other catalytic or cross linking agents. Some investigational work was done along these lines but an extended cure time and limited pot life has hampered these developments. Ether resins are polyalcohols and as such they can be substituted for part or all of the polyol in alkyds with subsequent improvement in chemical resistance. Here again some investigational work has been done.

The bulk of the investigations concerning ether resins has been on the ether ester type. The rate of cure of these esters can be improved by the copolymerization of the fatty acids with vinyl aryl compounds such as styrene or vinyl toluene. This avenue has been investigated extensively.

1. Ether Esters

Previous work by this laboratory, independent of this project, had indicated that soya and dehydrated castor fatty acids with Shell Chemical's Epon 1004 gave the most promising ether ester vehicles. Therefore these were investigated using two proprietary vehicles.

The first step was the determination of the critical pigment volume concentration. It was found that the castor ester had a CPVC of about 70% while the soya ester had a CPVC of about 46%. On this basis primers were prepared with zinc chromate at PVC's of 60 and 65% with the castor ester and 38 and 42% with the soya ester. Formulations also were prepared at PVC's of 60 and 65% for the soya ester in order to show the effect of using an excess of pigment. In addition, formulations were included with both esters at PVC's of 28% to correspond with the control specification primer. Sprayouts on dichromated magnesium and aluminum panels showed that all of the dehydrated castor ether esters showed considerably better drying than did the soya esters. Salt spray tests on coupled panels showed a slight advantage for the lowest PVC soya ester in corrosion resistance with the lowest PVC castor ester second best. Blistering tended to follow its normal trend, increasing with decreasing PVC. The two higher PVC soya ester paints were relatively poor, as expected. All of the ether ester systems were superior to the specification control. (See Section A, Tables 71, 79, and 80, Appendix and the photograph on the following page.)

The soya ester formulation contained 0.1% cobalt and 0.05% manganese as driers. Various additives and drier variations were tried to speed up and improve the cure of this soya ether ester. Doubling the original drier content, orthophenanthroline, and tetrabutyl titanate showed little if any benefit. The most promising additives were 0.2% calcium octoate and 4.0% of a very short linseed phenolic varnish. Although these additives did not clear up the residual tack they did impart better recoatability at two hours and at 24 hours. (See Section A, Tables 81, 82, and 83, Appendix.) No explanation was found for the tendency for the recoatability to be poorer on the aluminum substrates, as shown in the tables. Some work was done on the effect of extended storage of samples of the various substrates in individual samples of the wet primer but no positive results were obtained.

Although excellent recoatability was observed in these tests it was found in subsequent work that lifting still would occur on occasion, when the soya ether ester primers with the phenolic varnish additive were recoated after overnight dry. For this reason some work was done with a dehydrated castor ether ester, since it had shown better drying in the initial tests. The following drier combinations with percentages based upon resin solids by weight, were tried in attempts to improve the drying characteristics of MIL-P-6889a, Type I, primers containing this vehicle:

(1) 0.5% lead naphthenate
(2) 0.1% cobalt + 0.05% manganese (standard)
(3) 0.5% lead + 0.05% cobalt + 0.05% manganese
(4) 1.0% orthophenanthroline + 0.1% manganese
(5) 1.5% orthophenanthroline + 0.1% manganese
(6) 2.0% orthophenanthroline + 0.1% manganese

Over aluminum substrates all of these primers, with one exception, showed considerable softening when topcoated after 24 hours drying. The exception was the primer with the number (3) drier combination. Since lead is known to be a good drier for through drying these results are not surprising. It has been reported that ether esters will precipitate lead driers. However, a check on clear formulations with the number (3) drier combination did not show any turbidity with either a castor or a soya ether ester.

Salt spray tests were run on the castor ether primer containing drier number (3) along with the same primer containing the standard drier plus 4.0% of a short linseed phenolic varnish, and a styrenated soya ether ester primer containing 0.4% lead and 0.05% cobalt. The same vehicles, aluminized, were used in the topcoats. The results showed excellent performance for all of the systems with an advantage for the styrenated system in moisture resistance and a slight advantage for the castor ether esters in corrosion resistance. (See Section A, Table 84, Appendix.)

Both the soya and the castor ether esters that have been used in this work have had acid values of less than two at 50% solids. Higher acid values have been found to lower the alkali resistance of such vehicles. However, it was thought the higher acid values might also result in better drying characteristics and improved adhesion. A castor ether ester was prepared with an acid value of 5.5. Adhesion and hydrocarbon resistance tests favored the original ester with the lower acid value. (See Section A, Table 85, Appendix.)

A serious deficiency exists in ether resins for exterior usage; namely, a marked tendency to exhibit rapid chalking. This has been cited by Shell as a favorable method of paint film failure since it leaves a good repaintable surface. The effect of aluminizing an ether composition is under study but these tests are not mature. After four months Florida exposure at 45° south no chalk was reported for aluminized ether esters. It was thought to be desirable, therefore, to develop a topcoat for ether esters. MIL-L-7178 type topcoats are not satisfactory directly over ether ester primers. Apparently the incompatibility of nitrocellulose and ether esters leads to poor intercoat adhesion between coatings based on these resins.

Two avenues were followed in arriving at a more satisfactory system with ether ester primers. First the use of a chemically resistant topcoat with good exterior performance was tried directly over the ester primer. A vinyl copolymer topcoat was used in this case. It was found that with the solvents normally used, the vinyl topcoats caused lifting of the primer. This lifting tendency was overcome by using more volatile solvents in the vinyl topcoat. Even here there was a marked softening of the primer that remained for several days.

The second method tried utilized a styrenated ether ester primer, which will be discussed later, in a sandwich coat system. That is, a thin coat of a primer that showed good intercoat adhesion with both the ether ester and the nitrocellulose films was placed between the primer and the topcoat. This system showed good physical characteristics and fair solvent resistance. Such a system would be predicted to give better salt spray performance than a straight specification system because of better alkali resistance where the system is in contact with the metal.

After about 1000 hours of salt spray exposure the ether ester primer-vinyl topcoat system and the sandwich coat system were both much superior to the specification system in corrosion resistance, with some advantage for the former. The ether ester-vinyl system did show relatively weak moisture resistance. This is probably an effect of the retained solvents in the primer film. (See Section A, Table 86, Appendix.)

2. Vinyl Aryl Ether Esters

The difficulties with lifting upon recoating and the probable excessive chalking upon exterior exposure of ether ester based coating systems, pointed to further modification of these vehicles. One possibility would be the copolymerization of the fatty acid portion with vinyl aryl compounds such as styrene or vinyl toluene. This would be expected to promote more rapid curing, and, by dilution, less sensitivity to chalking. Considerable work was done along these lines.

Styrenated products such as alkyds and drying oils have gained a place for themselves as commercial products in the coating field in the past few years. Styrenation of these drying type vehicles has offered the advantages of more rapid drying rates and improved hardness. In many instances they also offer improved soap, water, and alkali resistance. However, they suffer from a sensitivity to solvents, poor mar resistance, and decreased flexibility. The copolymerization of fatty acids with vinyl aryl compounds and the esterification of this product with ether resins has shown indications of overcoming some of these deficiencies while at the same time maintaining the desirable characteristics[11,12].

In particular, the styrenated ether esters have shown good flexibility if the proportion of styrene is kept at a reasonably low level (less than 33%). At the same time the mar resistance has been found to be satisfactory. Although there has been evidence that the styrenated ether esters retain the inherent poor resistance to hydrocarbons it appears possible to circumvent this drawback by certain combinations of vinyl toluene modified ether esters. Actually no difficulty was encountered in recoating of vinyl aryl modified ether esters at the prescribed intervals.

There are, of course, innumerable variations that could be investigated in connection with vinyl aryl ether esters such as:

(1) Type and concentration of vinyl aryl component
(2) Type and concentration of the fatty acid.
(3) Type and concentration of epoxy-bisphenol condensate.
(4) Type and concentration of the styrenation catalyst.
(5) Reaction procedures such as time, temperature, method (solvent or fusion), and order of introducing the ingredients.

In the initial work on this phase six vehicles were prepared and evaluated, including four modified with styrene and two with vinyl toluene. The compositions and characteristics of these vehicles were as follows:

Type and Concentration			Acid	Viscosity	Cure	Color
Vinyl Aryl	Fatty Acid	Ether Ester	Value	G-H	Sec.	Gardner
Styrene-40	Linseed-40	Epon 1004-60	2.90	V	2	7
Styrene-40	Linseed-Soya-40	Epon 1004-60	1.13	O	8	7
Styrene-40	Soya-40	Epon 1004-60	0.52	N	24	7
Styrene-13	Benzoic-9 Dehydrated Castor-20	Epon 1004-60	2.80	Y	6	6
Vinyl Toluene-46	Linseed-40	Epon 1004-60	1.39	V	3	7
Vinyl Toluene-46	Soya-40	Epon 1004-60	1.18	K	18	6

These vehicles were incorporated in MIL-P-6889a, Type I, primers by substituting for the alkyd on an equal volume basis. No consideration was given to critical pigment volume concentration at this time.

These primers showed good physical characteristics with the exception of adhesion in some cases. Over MIL-M-3171, Type III, treated magnesium panels the linseed and dehydrated castor vehicles showed somewhat weak adhesion. In these cases the failures, both by scratch test and in a conical mandrel bend test, were between the dichromate and the metal rather than between the primer and the substrate. Salt spray tests of these primers over standard magnesium-aluminum assemblies under an MIL-L-7178, aluminized, topcoat showed all of the primers to have excellent corrosion and good moisture resistance. (See Section A, Tables 71, 87 and 88, Appendix and the photograph on the following page.)

In a number of subsequent tests a styrenated soya fatty acids ester of Epon 1004 with a ratio of 40:40:60 styrene: fatty acid: ether resin was found to show sufficient promise to warrant recommending this vehicle for the most promising system at the conclusion of the first year of this project. (See Section A, Tables 21, 31, 49 and 89, Appendix.) Consequently, it was deemed advisable to check this vehicle for conformance to all of the tests specified in MIL-P-6889a and MIL-L-7178. This was done using a primer based on the Type I control formula for MIL-P-6889a with the styrenated ether ester vehicle replacing the alkyd only on an equal volume of solids basis, and a topcoat based on MIL-L-7178, Aluminized, with the styrenated ether ester replacing both the nitrocellulose and the alkyd on an equal volume of solids basis.

Two vinyl copolymer primers also were included in these tests as promising coatings. Both were pigmented with strontium chromate and modified with a methacrylate resin, HG 41. One was based on a carboxyl modified vinyl copolymer, Bakelite VMCH, and the other on a hydroxyl modified copolymer, Bakelite VAGH. They were designed for use over dichromate treated magnesium and wash primed magnesium, respectively.

The styrenated ether ester primer passed all of the critical specification tests with the exceptions of 3.5.3 and 3.5.4. These are a hydrocarbon resistance test and an anchorage (tape) test, respectively. Even in these tests the primer tested alone passed in every case. It was only when topcoats were used that failures occurred. In some cases these failures could be attributed directly to poor intercoat adhesion with the lacquer topcoat. However, a styrenated ether ester system, primer and topcoat, gave poor results in the hydrocarbon resistance tests.

Two systems designed to improve the intercoat adhesion over styrenated ether ester primers and possibly the hydrocarbon resistance were included in these two critical tests. One of these was an alkyd enamel, MIL-E-7729, used directly over the styrenated ether ester primer, which gave excellent intercoat adhesion. The other was based on a sandwich coat of the alkyd primer, MIL-P-6889a, between the styrenated ether ester primer and the lacquer topcoat, MIL-L-7178. Again the intercoat adhesion was good. In each case the anchorage (tape) test was passed but the hydrocarbon resistance test was poor. (See Section A, Table 90, Appendix.)

The vinyl coatings passed all of the critical specification tests with the exceptions of 3.5.1, 3.5.2, and 3.5.4. These are a lacquer resistance, primer absorption test; a water resistance test; and an anchorage (tape) test, respectively. The failure in the lacquer resistance test was in surface appearance and was not considered serious since a nitrocellulose lacquer topcoat was used. The incompatibility of vinyl copolymer resins and nitrocellulose would be expected to lead to poor results here. The water resistance and anchorage tests, however, are serious failures. (See Section A, Tables 91 through 100 inclusive, Appendix.)

Prior to the time that these specification tests were completed, a pilot plant batch of a styrenated ether ester vehicle was made. This was done to check laboratory procedures in larger equipment since this type of vehicle had not been produced commercially. (See Section D, Appendix for cooking procedure.) At about this same time it was found that there is a certain degree of incompatibility between the styrenated ether ester vehicle and the phenolic dispersion resin. This incompatibility is evidenced by difficulty in the grinding operation. A third point in question at the same time was the durability of styrenated ether ester topcoats in comparison with alkyd topcoats. These three points for evaluation were combined into one series and a repeat evaluation was made on the effect of the phenolic dispersion resin in a second series. Both salt spray and Weather-Ometer (Atlas Twin Arc) tests were run.

An examination of the results showed that the pilot plant batch was slightly better than the laboratory batch of styrenated ether ester in salt spray performance, while the two vehicles were about equal in Weather-Ometer durability. In the Weather-Ometer it was interesting to note that the specification alkyd-lacquer system showed greater initial drop off in gloss but that the styrenated ether ester system showed an abrupt drop to a still lower gloss between 700 and 850 hours of exposure. The phenolic dispersion resin was not found to be critical from a performance standpoint in the styrenated ether ester primer. The alkyd topcoat over the styrenated ether ester primer improved the Weather-Ometer durability but did not improve the hydrocarbon resistance and was relatively weak in corrosion resistance. (See Section A, Tables 101 through 104, inclusive, Appendix.)

Since the system of styrenated ether ester primer-specification lacquer topcoat with a thin sandwich coat of the alkyd specification primer did show good intercoat adhesion and a little better solvent resistance, an attempt was made to increase the hydrocarbon resistance still more by increasing the drying period of the primer and/or the sandwich coat. This was found to be effective but the recoat times of 24 hours were too extensive to be practical. (See Section A, Table 105, Appendix.) This is interesting, however, in that it suggests that ultimate solvent resistance is possible if the proper rate of cure can be attained. Further efforts to attain solvent resistance in this manner were not undertaken.

The next approaches to solvent resistance with vinyl aryl type ether esters were through cooking methods and variations in the vinyl aryl component. A total of about 36 hours cooking time was required to prepare the styrenated ether esters discussed previously, including both the styrenation of the fatty acid and the esterification of the ether resin. Two laboratory cooks were made in which these two reactions were carried out simultaneously. One cook required 20 hours of cooking time and the other 16 hours because of slight variations in procedure. In both cases solvent resistance and corrosion resistance were improved but moisture resistance was impaired as determined by hydrocarbon resistance on aluminum panels and salt spray tests on standard magnesium-aluminum assemblies. (See Section A, Tables 106 and 107, Appendix.)

The variation in vinyl aryl component consisted of a substitution of vinyl toluene and divinyl benzene for the styrene. In this case the copolymerization reaction is carried to a point of apparent gelation. The resulting vehicle, however, can be cut with normal solvents. Two vehicles were cooked by this technique, both of which were hazy because of gel particles. Both were formulated into MIL-P-6889a, Type I, type primers and MIL-L-7178, Aluminized, type topcoats. These systems exhibited good solvent resistance over aluminum panels and excellent corrosion resistance but poor moisture resistance in salt spray tests. (See Section A, Tables 106 and 107, Appendix.)

In view of the relatively poor moisture resistance of the previous batches of vinyl toluene ether esters, another cook was made. In this case the ratio of vinyl toluene/fatty acid was reduced from 40/40 to 23/40. A primer patterned after MIL-P-6889a, Type I, and a topcoat patterned after MIL-L-7178, Aluminized, were made from this vehicle. In general this system gave excellent results. Hydrocarbon resistance and water soak tests were satisfactory. Both corrosion and moisture resistance were excellent after 1000 hours of salt spray exposure. A repeat cook of this vehicle gave comparable results in a complete system and also gave excellent results in a sandwich coat system using an alkyd specification primer between the vinyl toluene ether ester primer and a nitrocellulose specification topcoat. (See Section A, Tables 108, 109, and 110, Appendix.)

Still another variation on vinyl toluene ether ester vehicles was prepared in which gum arabic was included. The thought was that the gum arabic would aid in the dispersion of the small gel particles that have been present in all batches of this type of vehicle. This vehicle was not successful in that the gel particles still remained and in that it could not be ground into a suitable primer.

In spite of the fact that solvent resistance had been gained with vinyl toluene ether ester systems, there still remained a need for a different type of topcoat because of the questionable chalk resistance for exterior exposure. A series was instigated for the purpose of screening a variety of topcoats for suitable application over vinyl aryl ether ester primers, with particular emphasis on intercoat adhesion.

The following primers were included in this study:

1. MIL-P-6889a, Type I, proprietary
2. Castor fatty acids ether ester
3. Styrenated soya fatty acids ether ester
4. Vinyl toluene-soya fatty acids ether ester

Each of the following topcoats were applied over each of the primers:

1. MIL-L-7178, aluminized
2. MIL-L-7178, aluminized (rich ketone free solvent)
3. MIL-L-7178, aluminized (lean ketone free solvent)
4. Hydroxyl modified vinyl copolymer
5. Acryloid-alkyd
6. Vinyl alkyd (proprietary)
7. Vinyl-alkyd (52 R 13)

In general the alkyd specification primer took the topcoats the best, but in specific instances, particularly with the proprietary vinyl alkyd, excellent systems were obtained with the other primers. The vinyl aryl ether ester primers softened under all of the topcoats but recovered to give satisfactory to excellent adhesion in some cases. The Acryloid-alkyd topcoat gave excellent leafing with aluminum but tended to lift the ether ester primers more than any other topcoat. (See Section A, Table 111, Appendix.)

From this test the proprietary vinyl alkyd was selected for further evaluation over all of the primers. In this case the nitrocellulose specification topcoat was used as the control. Water resistance tests favored both of the vinyl aryl ether ester primers while solvent resistance tests favored the alkyd specification primer and the ether ester primer. (See Section A, Table 111, Appendix.) Salt spray tests showed that the styrenated ether ester primer with the proprietary vinyl alkyd had the best moisture resistance, while the vinyl toluene ether ester primer with the same topcoat had the best corrosion resistance. (See Section A, Table 112, Appendix.)

If vinyl aryl ether ester systems are desired for any colors other than aluminum the use of a topcoat such as vinyl alkyd probably would be desirable. Weather-Ometer tests were run on Glossy Sea Blue formulations using two vinyl aryl ether ester vehicles and a lacquer for a control. Both of the ether ester formulations showed poor gloss retention after 700 hours of exposure. (See Section A, Table 113, Appendix.) It was found in the preparation of these enamels that a drier combination of lead and cobalt caused agglomeration. A change of driers to cobalt and manganese corrected this condition.

It was considered desirable at this point to prepare a pilot plant batch of vinyl toluene-ether ester in order to determine if the cook was practical for full scale production. The most promising formulation was used, namely; 60 parts of Epon 1004, 40 parts of soya fatty acid, 23 parts of vinyl toluene and 4 parts of divinyl benzene. (See Section D,2, Appendix, for details of procedure.) The physical characteristics of this batch compared very closely with those of the previous laboratory batches. Salt spray tests on primer-topcoat systems from this batch also showed good results. (See Section A, Tables 114 and 115, Appendix.) However, hydrocarbon resistance tests on aluminum panels were very poor. At the same time it was found that vinyl toluene-ether ester systems that had been prepared from laboratory batches of the vehicle also had poor solvent resistance. Apparently some change had taken place during aging of the primer and/or of the vehicle in the package for little more than one month which caused poor development of solvent resistance.

Immediately two laboratory batches of this vehicle were prepared using, in one case, the same raw materials as used in the plant batch and, in the other case, the same raw materials as used in the previous laboratory batches. Here the latter batch showed satisfactory solvent resistance while the former did not in primer-topcoat hydrocarbon resistance tests. However, a recheck on this solvent resistance after only one week's aging of the coatings in the package showed poor solvent resistance in both cases. Again there is evidence of rapid loss of the ability to attain solvent resistance during aging of coatings in the package. An aged vinyl toluene-ether ester coating system on an aluminum panel retained good solvent resistance for at least one month.

A number of drier variation studies were instigated. These gave variable results, partly, it is presumed, because of changes upon aging of the wet coatings but partly because of the vagaries of paint testing. The most promising drier additives appeared to be calcium octoate and iron. Even these did not give good hydrocarbon resistance after short periods of aging in the package. (See Section A, Tables 116, 117, and 118, Appendix.)

Since the vinyl toluene-ether ester cook is of the solvent type using vinyl toluene as the solvent, there is a possibility that some vinyl toluene monomer remains in the completed vehicle. If so this could explain the loss of the ability to develop solvent resistance upon aging of wet samples. The vinyl toluene monomer would evaporate from films laid down from fresh coatings. On the other hand the vinyl toluene monomer would tend to polymerize to a polyvinyl toluene in aged coatings. The polyvinyl toluene, not being volatile, would remain in films laid down from such aged coatings and would weaken the solvent resistance. On these hypotheses, another vinyl toluene-ether ester was prepared in the laboratory using the same procedure as for the pilot plant batch, but including steam sparging.

The hydrocarbon resistance of this vehicle in a primer-topcoat system was found to be relatively good. A few blisters occurred along the edge of the panel and the entire film softened somewhat. Recovery was rapid since it depended primarily on solvent evaporation. Further testing with this vehicle and a similar one cooked to a lower cure (10 seconds compared with 30 seconds) showed them to undergo somewhat the same loss of solvent resistance upon aging in the package as did unsparged cooks. (See Section A, Table 154, Appendix.)

Presumably a reduction in total vinyl aryl content in an ether ester vehicle would also result in increased hydrocarbon resistance. To this end, a cook was made using 13 parts of vinyl toluene, 20 parts of fatty acid, 60 parts of ether resin and 9 parts of benzoic acid. (See Section D,3, for details on the cooking procedure.) Again it was found that a primer-topcoat system prepared from this vehicle had good hydrocarbon resistance. In this case there were no blisters even on the edges and the film softened only while in the solvent. This vehicle also showed some loss of solvent resistance upon aging in the package in each of two cooks. (See Section A, Table 154, Appendix.)

Both the sparged type of vehicle and the low vinyl aryl content vehicle showed good corrosion resistance in limited salt spray tests. The sparged vehicle showed relatively poor water resistance on salt spray but excellent water resistance on an immersion test. Both vehicles showed good adhesion. (See Section A, Tables 154 and 155, Appendix.)

It is considered that, in spite of some shortcomings in solvent resistance, the vinyl toluene modified ether ester offers the most promise for magnesium coatings for the Air Force. Primers and topcoats based on a sparged ether ester (Section D,2, Appendix) and a low vinyl aryl content ether ester (Section D,3, Appendix) are being submitted to the Air Force for evaluation. Both vehicles are being included since there has been relatively little test work on low vinyl aryl content type.

3. Room Temperature Curing of Ether Resins

Ether resins of the type represented by Shell's Epon resins contain epoxide and hydroxyl groups. It is through the reactivity of these groups that curing is effected. This reactivity can be utilized at room temperature through the use of certain catalysts or cross linking agents.

The catalysts normally used are acidic in nature. Sulfonic acids are favored because of their activity without destructive action on organic materials. Such acids as, for instance, p-toluene sulfonic acid, serve to break the epoxide ring and encourage further polymerization through condensation. This polymerization is rapid even at room temperatures. However, previous workers have found the subsequent films to be brittle in nature.

Either alcohols or amines can be used as cross linking agents but the amines seem to be more effective at room temperature. In order to obtain suitable three dimensional polymers polyamines must be used. Ethylene diamine and its homologues such as diethylene triamine or triethylene tetramine appear to be particularly useful in this respect.

Titanium esters also should be useful in curing hydroxyl resins because of their tendency to hydrolyze even with such high alcohols as the ether resins.

In selecting the ether resin for investigations with cross linking agents, therefore, attention should be given to both the epoxide and hydroxyl concentration. Normally it is accepted that monomeric polyamines cure ether resins primarily through the epoxide groups. From this it would be expected that the lower the molecular weight the faster would be the cure since epoxide groups appear only at the end of the molecules of ether resins. On the other hand, the curing with titanium esters should be more effective with the higher molecular weight resins which contain more hydroxyl groups. This discussion suggests the employment of combinations of polyamines and titanium esters with all but the simplest possible ether resin. This has been done along with some work on various polyamine-ether resin combinations.

a. Polyamines

Two polyamines, ethylene diamine and diethylene triamine, were tried at 6 and 12 parts per hundred parts of resin with four different ether resins in clear films. The resins were Epons 828, 834 and 1001 and an experimental Sherwin-Williams resin with a somewhat different composition. All combinations were found to dry within six hours with the exception of those containing Epon 828. This was unexpected since Epon 828 is the lowest molecular weight of the Epon resins. A recheck on another lot of Epon 828 confirmed these results. Recoating of these films at 24-48 hours with a lacquer resulted in very poor adhesion to the substrate. Blends of Epon 828 and 1001 with 6 PHR of ethylene diamine also were unsuccessful. (See Section A, Tables 119 and 120, Appendix.)

b. Polyamine-Titanium Ester Blends

Various proportions of diethylene triamine and butyl titanate, including 100% amine and 100% titanate, were tried with clear solutions of Epon 1001. Little difference was found in drying rates but high amine ratios were more favorable from the standpoint of solvent resistance (recoat adhesion) and moisture resistance (water immersion). High titanate ratios favored package stability. (See Section A, Table 121, Appendix.) It was noted in this work that heavier films tended to improve the adhesion after recoating with a lacquer. At the same time it was found that the higher boiling alcohol solvents aided package stability more than the lower alcohols. With ethyl alcohol, for instance, immediate precipitation of gel particles occurred upon the addition of the cross linking blend of amine and titanate. Diacetone alcohol was found to give the most favorable solvent blends but even when films 1.0 mil thick were sprayed they failed to give good recoat adhesion. (See Section A, Tables 122 and 123, Appendix.)

c. Ether Modified Alkyds

In investigations separate from this project it has been found that replacing a part of the polyol in alkyds with an ether resin results in improved chemical resistance and especially improved alkali resistance. Using a proprietary alkyd resin designed for use in MIL-P-6889a three different ether modified alkyd resins were prepared. The first, in which a simple substitution of 10% of the glycerol by Epon 1004 was made, an excessively high acid value resulted. In the second, 10% Epon 1004 and 10% glycerol were added to the normal alkyd formulation. Here a more nearly normal acid value was obtained but it was still high. In the third, the reactive oil in the alkyd was replaced with linseed and 10% of the glycerol was replaced with ether resin. All three vehicles were formulated into MIL-P-6889a, Type I, primers and evaluated over standard dichromate treated magnesium-aluminum panel assemblies under an MIL-L-7178, aluminized lacquer. None of these primers showed any promise. They exhibited good solvent resistance but a weakness in corrosion and/or moisture resistance in salt spray tests. (See Section A, Tables 124 and 125, Appendix.)

E. Furan Resins

Furan resins have been used in the chemical industry as tank linings. For this application the resins are used to impregnate fiber glass and generally are cured by heat. They can be cured at room temperature by the use of acid or basic cross linking agents. The acids have been used more extensively. Strong mineral acids such as HCl or H_2SO_4 are the most effective curing agents while acids such as oxalic, maleic and phosphoric increase the package life and exert some curing action. Furan resins have been investigated for this project to some extent.

A commercial coating reputedly based on furan resins was evaluated over bare, wash primed, and dichromate treated magnesium. Salt spray tests on standard magnesium-aluminum assemblies showed superior corrosion resistance for this coating but a tendency to blister after only 24 hours exposure. (See Section A, Tables 126 and 127, Appendix.)

A number of commercial furfuryl alcohol resins were investigated also. These included:

1. F-1003, supplied by the Haveg Corporation
2. Durez 14383, by Durez Plastics and Chemicals, Inc.
3. Jet-Kote, Type X-3M, by Furane Plastics, Inc.
4. X-5A, by Furane Plastics, Inc.
5. Resistojet, by Furane Plastics, Inc.
6. Durez 15789, Durez Plastics and Chemicals, Inc.

Of these the Jet-Kote and Durez 14383 were the most promising. Resin X-5A requires baking at 330° to 500° F for complete curing. Resistojet was similar to Jet-Kote. Durez 15789 is a high molecular weight resin which it was hoped would form a film without catalyst simply by solvent evaporation. Actually it required several days to form a tack free film. F-1003 did not form a good film under any of the conditions tried.

The catalysts investigated included oxalic acid, phosphoric acid, hydrochloric acid, mono ethyl acid orthophosphate, mono octyl acid orthophosphate, and carbamide-phosphoric acid. The results with oxalic acid were poor. Phosphoric acid was a faster catalyst for F-1003 and Jet-Kote than for Durez 14383. Actually the phosphoric acid catalyzed Durez 14383 was still fluid in the package after more than three weeks, whereas the other resins gelled after one week. Although Durez 14383 was slower in curing, the cured film was found to be more resistant to methyl isobutyl ketone than were the other resins.

Hydrochloric acid cured all resins in about 30 minutes and caused instability in the package within 16 hours. Mono ethyl phosphate and mono octyl phosphate were used with Jet-Kote only. As expected, the lower alkyl phosphate was the better catalyst and gave a much tougher film. Both gave extended package stability.

Jet-Kote and Durez 14383 were pigmented with titanium dioxide to give light brown films. Salt spray tests using a proprietary catalyst, Furane's Z-1A, with Jet-Kote and a mixture of sulfuric acid and isopropyl acetate with Durez 14383, gave poor results with both resins. (See Section A, Table 128, Appendix.)

No further work was done on furan resins.

F. Polysulfide Polymers

Pigmentation studies described under Section IV have shown some unusual effectiveness of sulfide compounds in inhibiting the corrosion of magnesium. With the thought that sulfide groups in vehicles might also be effective, a cursory evaluation of one sulfur bearing vehicle, Thiokol LP-3 was undertaken. Thiokol LP-3 is a poly-functional mercaptan with reactive SH terminal groups. Following a formulation suggested by Thiokol Corporation, LP-3 was blended with Epon 828 at a ratio of 2:3. This blend was pigmented with titanium dioxide and catalyzed with tetra ethylene pentamine at a rate of six parts per hundred of resin. (See Section C,f, Appendix.) This finish was used directly over dichromate treated magnesium-aluminum panels as both primer and topcoat. The adhesion and general film character were good. Salt spray tests gave excellent results, actually better than a vinyl toluene-ether ester system, in both corrosion and moisture resistance. (See Section A, Table 129, Appendix.) This type of formulation suffers from a limited package stability after the addition of the amine and from a relatively slow curing rate. Nevertheless it merits further investigation.

SECTION IV

PIGMENTATION

The study of pigmentation of magnesium coatings has not received as much attention as the study of vehicles. This probably results from two factors; first, that vehicles were taken up first and thus received a greater impetus, and second, that more improvement was found from vehicle changes than from pigment changes. The work done on critical pigment volume concentration and on the pigmentation of wash primers was reported in previous sections. This section is concerned with a screening test, with the evaluation of the better pigments from this test and with a particularly promising pigment combination, calcium sulfide-ammonium vanadate.

A. Screening Test

At stages in the project where vehicle selections had not been made, it was deemed desirable to check a number of pigments for their effects on the corrosion of magnesium. These tests were conducted using water as the vehicle. The tests consisted of completely immersing weighed metal strips in saturated solutions of the various pigments in distilled water and in 3% NaCl solutions. The relative inhibition of corrosion for each pigment was judged on visual gradings and actual weight losses. Three different types of metal were used, namely:

1. FS1H magnesium that had been alkali cleaned and chromic acid pickled.

2. FS1H magnesium that had been MIL-M-3171, Type III treated in the laboratory.

3. FS1H magnesium that had been MIL-M-3171, Type III treated in the laboratory and coupled to a 24S aluminum panel with a 56S aluminum rivet. (Evaluated in water solutions only.)

Even in the early stages of this test it was apparent that none of the pigments under investigation were giving complete protection. At first the corrosion in the distilled water and NaCl solutions was about equal, if anything, somewhat more severe in the distilled water solutions. As the experiment progressed the rate of corrosion in the NaCl solution increased more rapidly. This suggests that the salt accelerated corrosion of the magnesium primarily because of the increased conductivity which it imparted to the environment. Such conductivity could have been less important at the beginning of the corrosion when the anodes and cathodes tended to be small and relatively close together. Then as corrosion proceeded and the anodes and cathodes increased in size the distances that the corrosion currents had to flow would be increased and conductivity would have become more important.

Chromate pigments, particularly the more soluble ones, appeared to be the best inhibitors for magnesium corrosion. Lead pigments on the other hand acted as accelerators for this corrosion. Inerts generally were neutral but silicon dioxide appeared to be about as effective an inhibitor as the better chromates.

Other pigments showed a variable performance. Zinc oxide caused very rapid corrosion in salt water but had very little effect in plain water solutions. Chromium phosphate, in spite of previous indications of good results in wash primers, was only fair to poor in these tests. It was proved definitely that not all fluorides are good inhibitors for magnesium, as was hypothesized by some investigators. Sodium silico fluoride, for instance, caused severe corrosion in all of the environments. Both magnesium oxide and zinc sulfide dropped off from an original rating of good to a rating of fair. Lampblack was relatively poor.

Control tests using plain distilled water and NaCl solutions without the pigments showed relatively little corrosion in the water but considerable corrosion in the NaCl solution with either bare or dichromate treated magnesium. This compared with a certain group of pigments which was classified as "Inactive." The pigments that reduced the corrosion in NaCl solutions without any deleterious effects in water solutions were classified as "Inhibitive" while those that allowed appreciable corrosion in water solutions or increased corrosion in NaCl solutions were classified as "Accelerators." A listing of this order is shown below:

Inhibitive Pigments

- Strontium Chromate
- Zinc Chromate
- Silicon Dioxide
- Calcium Chromate
- Barium-Potassium Chromate

Inactive Pigments

- Fibrous Magnesium Silicate
- Diatomaceous Silica
- Calcium Carbonate
- Magnesium Silicate
- Zinc Sulfide
- Barium Chromate
- Magnesium Oxide
- Basic Lead Silico Chromate

Accelerating Pigments

- Calcium Plumbate
- Titanium Dioxide - R-510
- Chromium Phosphate
- Mica
- Lampblack
- Lead Chromate
- Titanium Dioxide - R-610
- Red Lead
- Zinc Oxide
- Basic Silicate White Lead
- Sodium Silico Fluoride
- Lead Cyanamid

B. Evaluation of the Better Pigments

These results were used in the preparation of four experimental MIL-C-15328a type wash primers and five MIL-P-6889a type styrenated ether ester primers. In all cases each of the four "Inhibitive" chromate pigments were used to replace the regular chromate pigment and silicon dioxide was used to replace the regular magnesium silicate inert, both on a volume basis. Salt spray tests with the wash primers showed the corrosion resistance of all of the systems to be about equal to one using MIL-C-15328a. The strontium chromate wash primer had an advantage in moisture resistance. (See Section A, Table 46, Appendix.) In the styrenated ether ester primer calcium chromate was slightly superior to the regular zinc chromate in corrosion resistance but somewhat inferior in moisture resistance. Strontium chromate and barium potassium chromate were poor in both corrosion and moisture resistance. The replacement of the inert by silicon dioxide was definitely beneficial in both corrosion and moisture resistance. (See Section A, Table 130, Appendix.)

C. Inhibitive Pigments in Vinyl Copolymer Vehicles

As discussed in Section III, C, primers based on vinyl copolymer vehicles show good salt spray resistance for a period but once failure starts it proceeds quite rapidly. For this reason it was thought that inhibitive pigments are not as effective in vinyl vehicles as in other types of vehicles, such as alkyds or styrenated ether esters. To investigate this, two primers were prepared from vinyl copolymer vehicles, one pigmented with iron oxide and one with strontium chromate. These primers along with a proprietary vinyl primer pigmented with iron oxide and a modified vinyl copolymer pigmented with strontium chromate were subjected to salt spray exposure on standard magnesium-aluminum test panels under an aluminized vinyl topcoat. In both cases the iron oxide pigmented primers were somewhat better in corrosion resistance and moisture resistance. This served to confirm the theory. (See Section A, Table 131, Appendix.)

D. Ammonium Vanadate-Calcium Sulfide as Magnesium Corrosion Inhibitor

Emerson and Cuming Company have reported that a combination of ammonium vanadate with calcium sulfide is an excellent corrosion inhibitor for magnesium[13]. This combination was evaluated first at two different concentrations in an MIL-P-6889a type primer using a styrenated ether ester vehicle. In one case all of the pigment was replaced on a volume basis with the ammonium vanadate-calcium sulfide while in the second case only 5% of the pigment by volume was replaced. In both cases salt spray results were disappointing. The 100% substitution was very poor in both corrosion and moisture resistance while the 5% substitution was somewhat deficient in moisture resistance and only equal in corrosion resistance to the standard zinc chromate-magnesium silicate pigmentation. (See Section A, Table 132, Appendix.)

The calcium sulfide-ammonium vanadate combination was then checked in a screening test as described previously. Here the inhibition was outstanding. Essentially complete protection was given to magnesium-aluminum couples for six days in a 3% NaCl solution. Calcium sulfide by itself was good but definitely inferior to the combination. Other pigments and pigment combinations that were tried were all poor in the 3% NaCl solution. These were as follows:

- Strontium chromate plus calcium sulfide
- Ammonium vanadate
- Zirconium fluoride
- Potassium zirconium fluoride
- Zinc chromate
- Strontium chromate
- Silicon dioxide
- Magnesium silicate
- Blank - control

It was evident that there was a synergistic effect from the combination of ammonium vanadate with calcium sulfide and that calcium sulfide is the most active of the two components. (See Section A, Table 133, Appendix, and the photograph on the following page.)

The exceptional corrosion inhibition for magnesium by ammonium vanadate-calcium sulfide combinations shown here led to further investigations. First the critical pigment volume concentration was determined. The Asbeck-SW CPVC cell was not adaptable for use with the styrenated ether resin and gloss readings on a PVC ladder were not clear cut so permeability studies were investigated. By this method it was determined that the CPVC of an ammonium vanadate-calcium sulfide pigmented styrenated ether ester primer based on MIL-P-6889a, Type I, was between 75 and 80%. Such primers were then prepared at PVC's of 80 and 85% and used both as normal primers and as sandwich coats between a zinc chromate pigmented styrenated ether ester primer and an aluminized styrenated ether ester topcoat. Salt spray tests again were disappointing. Moisture resistance was very poor and corrosion resistance was slightly inferior to a standard system. (See Section A, Table 134, Appendix.)

Since the application of topcoats over highly pigmented primers does affect the permeability of such primers, the calcium sulfide-ammonium vanadate primer with the 80% PVC was tried as the complete system on standard magnesium-aluminum couples. Salt spray tests using a bare couple as the control showed relatively weak protection from such a system. (See Section A, Table 135, Appendix.)

An opalescent film was noted on aluminum-magnesium couples that had been immersed in saline solutions of calcium sulfide-ammonium vanadate combinations. The possibility that such a film might be a good pretreatment for painting was investigated. Several methods of obtaining such films were tried including immersion of coupled and individual magnesium and aluminum panels in water and 3% saline solutions of calcium sulfide-ammonium vanadate combinations, and using a low voltage direct current on similar set-ups in which the magnesium was made the anode and aluminum the cathode. Salt spray tests of standard test assemblies using these pretreated panels did not show any promising results. (See Section A, Tables 136 and 137, Appendix.)

A ratio of 43:57 calcium sulfide:ammonium vanadate was used in all of the tests described previously. Various ratios of these two components were evaluated by the screening test method. These tests favored increased calcium sulfide content up to 75%. (See Section A, Table 138, Appendix.)

A number of additional screening type tests were carried out in efforts to determine the mechanism by which the combination of calcium sulfide and ammonium vanadate inhibit the corrosion of magnesium. The following conclusions were reached:

(1) The alkalinity of calcium sulfide is not the explanation. Sodium sulfide and calcium hydroxide tests showed poor inhibitive effects.

(2) The formation of ammonium sulfide may be a partial explanation but ammonium sulfide by itself appeared to be exhausted during screening tests.

(3) Of the individual ions calcium, sulfide, ammonium and vanadate the sulfide is the most critical.

(4) Zinc sulfide, cadmium sulfide and antimony sulfide do not show the inhibitive action of calcium sulfide either by themselves or in combination with ammonium vanadate.

(5) The reaction product of calcium sulfide and ammonium vanadate, even when thoroughly water washed shows almost as good inhibitive action as the mixture of the components.

(See Section A, Tables 139 through 143 inclusive, Appendix.)

Following these results, four different lots of pigments were prepared from the reaction product of calcium sulfide and ammonium vanadate using slightly different methods and two different ratios of calcium sulfide to ammonium vanadate. Screening tests showed the lot prepared from a 1:1 ratio of $CaS:NH_4VO_3$ with no aging of the precipitate in the liquor to be slightly the best in inhibitive action. Again the inhibitive action was almost equal to the mixture of calcium sulfide and ammonium vanadate. (See Section A, Table 144, Appendix.)

Another lot of pigment was prepared by the better method with a 1:1 ratio of $CaS: NH_4VO_3$. Again screening tests showed excellent results. This pigment was incorporated in both a styrenated ether ester vehicle and a vinyl toluene-ether ester vehicle to make MIL-P-6889a, Type I, primers. Salt spray tests on these primers over standard magnesium-aluminum test assemblies under aluminized topcoats with the respective vehicles showed good moisture resistance but corrosion resistance only equal to that of zinc chromate. (See Section A, Tables 145 through 147 inclusive, Appendix.) There was not sufficient time to conduct further studies.

SECTION V

MISCELLANEOUS

A. Vinyl Tape

The Navy has developed a special system for the protection of magnesium-aluminum couples using a vinyl tape between faying surfaces. The McDonnell Aircraft Corporation of St. Louis has issued a set of instructions which were followed in this investigation. These instructions encompass the application of three coats of MIL-P-6889a on aluminum and four coats on magnesium followed by vinyl tape along the faying surfaces and extended at least one-eighth inch beyond the lap. A normal topcoat was used. Minnesota Mining and Manufacturing Company's #470 Vinyl Tape was used. As controls a standard number of prime coats (one on aluminum and two on magnesium) and double this number of prime coats were used without the tape. Salt spray tests showed no advantage for the tape in either case. (See Section A, Table 148, Appendix.) It is quite possible that tape systems would show better relative performance on normal exterior exposures. In salt spray tests the panels are continuously wet with the 3% salt solution. This allows practically continuous bridging of the vinyl tape with an electrolyte and in effect negates the usefulness of the tape. Under other conditions of exposure where bridging of the tape would not be as prevalent, considerably more benefit could be obtained from the use of the tape.

B. Red Oxide Primer

Excellent performance has been reported by other investigators for MIL-P-11414, Red Oxide Primer, over magnesium. Salt spray tests of this primer over standard dichromate treated magnesium-aluminum test assemblies under MIL-L-7178, Aluminized, showed it to be about equal to MIL-P-6889a, Type I, in performance. (See Section A, Tables 149 and 150, Appendix.) It is not the purpose of this project to develop lower cost primers for magnesium nor to develop zinc chromate conservation formulations, but rather to develop improved coatings. Therefore these results were not conducive to further work.

C. Reduction in Total Film Weight

For obvious reasons there is a continuous desire on the part of the Air Force to reduce the weight of aircraft structures. Paint systems, too, add an appreciable weight, to a point where it would be advantageous to eliminate as many coats as practical and still maintain adequate corrosion resistance. The more alkaline resistant coatings discussed in the section on vehicles were thought to offer possibilities along this line. For this reason various vinyl copolymer combinations, styrenated ether ester, and the specification were applied over dichromate treated, wash primed and bare magnesium in the following coating systems:

(1) Two coats primer with two coats topcoat on the magnesium and one coat primer with two coats topcoat on the aluminum

(2) One coat primer with one coat topcoat on both the magnesium and aluminum

(3) No primer, one coat topcoat after assembly of the panel

Failures in salt spray tests were very rapid in all cases where only one coat of topcoat was used as was expected where the faying surfaces were not painted. With one coat of primer and one coat of topcoat (total of 1.0 mil) the specification system and vinyl systems were still relatively poor. The styrenated ether ester system was reasonably good, actually slightly better than two coats of primer and two coats of topcoat with the specification system. With two coats of primer and two coats of topcoat the styrenated ether ester system again was superior, while the vinyl copolymer was good during the early stages of the test but only fair at the end of the test. (See Section A, Table 49, Appendix.) This investigation indicated that styrenated ether ester coatings are slightly better than the specification system for corrosion resistance at about one-half of the coating weight.

Another possible method of applying coatings over aircraft where dissimilar metal couples occur would be to prime only the faying surfaces plus a one inch overlap and then topcoat the entire surface. This was tried with the same systems used in the previous tests over the same types of pretreatment on magnesium and aluminum. Again salt spray tests showed early failures in those areas where only one coat of topcoat was used, regardless of the type of paint. (See Section A, Table 151, Appendix.) Thus it is not considered feasible to use only a partial primer coat on any portion of an aircraft.

D. Permeability of Films to Chloride Ions

Kittleberger and Elm[14] along with Mayne[15] have suggested that paint films inhibit corrosion by acting as ion barriers, particularly chloride and sulfate ions, rather than as barriers to oxygen or water. Kittleberger and Elm have reported that phenolic films have a very low diffusion rate of sodium chloride. Work done by the project has found phenolic coatings good resistors toward corrosion but deficient in other properties such as flexibility and solvent resistance. With this apparent confirmation of previous results, it was decided to investigate the diffusion of sodium chloride through various films that were of interest to this project.

Initial tests have been made on phenolic and vinyl toluene-ether ester films. The procedure was to form an unsupported film by the familiar technique of application on a tin substrate and removal by amalgamation. These films were placed between ground glass surfaces of a two section U tube. A 3% sodium chloride solution was placed on one side of the film and distilled water on the other. By following the decrease in resistance of the distilled water a measure of the diffusion of ions through the film was obtained.

The phenolic film showed an initial lag period the first hour of the test and then a steady, even increase in specific conductivity. The vinyl toluene-ether ester film was almost identical to the phenolic film in specific conductance measurements. The initial lag in diffusion can be attributed to the film thickness. Thus a steady rate of diffusion was attained only after the film reached a state of equilibrium with respect to sodium chloride. Time did not permit further investigation of this technique but it appears to have some possibilities.

E. Emerson & Cuming Recommended Magnesium Primer

The Emerson & Cuming Company has recommended a primer based on Epon 1001, pigmented with strontium chromate and calcium sulfide, and catalyzed with diethylene triamine[16]. They have designated this primer as Primer 8553. (See Section C,g, Appendix, for formulation.) This primer showed excellent adhesion to magnesium, excellent solvent resistance and good water resistance in an immersion test for 24 hours. Salt spray tests, however, were not as favorable. Here moisture resistance was only fair and corrosion began after less than 300 hours of exposure. In these respects the specification system MIL-P-6889a - MIL-L-7178, was about equal to Primer 8553. A vinyl toluene-ether ester system included in this test showed no corrosion on any panels and fair to good resistance to moisture. (See Section A, Tables 152 and 153, Appendix.)

SUMMARY AND CONCLUSIONS

The purpose of this contract was to strive to develop improved organic coatings for the protection of magnesium, particularly when in contact with dissimilar metals. This has been done. The culmination of these efforts will be embodied in five gallon samples of primer and aluminized topcoat which will be furnished to the Materials Laboratory, Wright Air Development Center. The final selection of the vehicle for these coatings has been made and consists of two types of vinyl toluene-ether esters as described in Sections D,2, and 3 of the Appendix. The primers will be pigmented with zinc chromate and silicon dioxide at a PVC of 35%. The topcoats will be pigmented with aluminum at the same PVC as MIL-L-7178, Aluminized. (See Section C, h and i, Appendix, for detailed formulation.)

Standard test methods including panel construction, paint application and conditions of exposure were established during the early part of the contract and were followed throughout except where otherwise noted. In this connection, considerable difficulty was encountered in establishing a satisfactory dichromate treated magnesium surface. By direction this dichromate treatment was applied as specified in MIL-M-3171, Type III. Laboratory pretreatment of test panels was found to be too time consuming and non-uniform. Commercial pretreatment varied considerably from one processor to another and even the best was not uniform from lot to lot. In order to minimize the possibility of errors from non-uniform substrates at least three and usually four replicate panels were used for salt spray tests.

A number of other types of inorganic pretreatments were investigated. These included MIL-M-3171, Types I, II and V, Dow #17, and Iridite #15, Mag-Coat, on magnesium as well as Alodine pretreatments on aluminum. None of these showed any outstanding advantages over the standard pretreatments.

Organic pretreatment of magnesium surfaces with wash primer type coatings gave variable results. Apparently certain unknown factors were not being controlled. It appeared that a reduction of the phosphoric acid activator to 25% of normal with coincidental control of the water content offered promise in reducing the amount of blistering due to hydrogen evolution when wash primers were applied over bare magnesium. This move coupled with the use of a small percentage of silicone oil in the wash primer and the application of thin films of wash primer should eliminate problems of hydrogen evolution. The performance of wash primers was variable. In general over bare magnesium they showed corrosion resistance at least equal to that of dichromate treatment, and over dichromate treatments they improved the corrosion resistance. However, their moisture resistance was not always satisfactory in either case. This moisture resistance was the main variable in performance.

Certain variations in wash primer formulations looked particularly interesting. These included one based on a chrome phosphate resin in a polyvinyl butyral resin and one based on MIL-C-15328a but using strontium chromate and silicon dioxide as the pigmentation. These formulations, particularly the latter, are worthy of further investigation. Another method of improving the moisture resistance of wash primers was the use of a more impermeable system, such as aluminized vinyl copolymer topcoats over a vinyl primer.

Alkali resistance was established as a prime factor in the performance of vehicles in coatings over magnesium. This was hypothesized at the beginning of the project on the basis of the high alkalinity of magnesium corrosion products. Thus a systematic variation of alkyd vehicles failed to show any marked increase in the performance of coatings based on these alkyds. But the use of vinyl chloride-vinyl acetate copolymer paints or systems based on epichlorohydrin-bisphenol condensates showed marked improvement.

It was found that the latter type of system gave better protection against corrosion creep, possibly because of a combination of better adhesion and better use of inhibitive pigments. Of the various types of vehicles based on epichlorohydrin-bisphenol condensates the fatty acid esters were found to be the most promising. Copolymerization of the fatty acids in the esters with vinyl aryl compounds was necessary to get proper curing. Solvent resistance of such vinyl aryl ether esters was improved by the substitution of vinyl toluene for styrene but the improvement was found to be somewhat temporary. Further refinements in cooking procedures and composition have given indications of imparting satisfactory solvent resistance to coatings made from this type of vehicle.

Other types of vehicles evaluated included phenolic varnishes, furan resins, and polysulfide resins. The phenolics showed surprisingly good corrosion resistance but suffered from a lack of flexibility or slow drying and from poor solvent resistance. Their relative impermeability to chloride ions may be an explanation of their corrosion resistance. Experimental furfuryl alcohol resin formulations did not show any promise as magnesium coatings but one proprietary coating was excellent for corrosion resistance although weak in moisture resistance. Limited work on a polysulfide resin-epoxy resin formulation gave very promising results as a magnesium coating. The necessity for a two compartment package was a disadvantage but further work is recommended on this type of formulation.

Near the end of this project a comprehensive study of the more promising types of coatings was conducted in one series. The best coating systems from this series were selected for a repeat evaluation. Both of these series served to confirm the selection of the vinyl toluene-ether ester vehicle. Salt spray results are illustrated in the four photographs on the following pages. In the first pair of photographs each panel is representative of four replicate panels. Number 4 is the vinyl toluene-ether ester system over dichromate treated magnesium-aluminum. In the second pair of photographs four replicate panels are shown for each system. "A" designates the vinyl toluene-ether ester system. Detailed identification of these photographs is given in Section A, Tables 52 and 75 respectively, Appendix.

Pigmentation investigations were not as fruitful as vehicle investigations. Certain chromates such as strontium and calcium showed some advantages over zinc chromate but these were not consistent enough nor of sufficient magnitude to justify replacing zinc chromate. A decided advantage was found, however, for silicon dioxide over magnesium silicate as the inert in primers. Considerable work was done with calcium sulfide and ammonium vanadate mixtures and the reaction product of these two compounds. Excellent corrosion inhibition was found for magnesium-aluminum couples immersed in saline solutions saturated with such a mixture or pigment. However, no way was found to incorporate this mixture or the pigment into a paint film and to retain the excellent inhibition of corrosion. The excellent results in immersion tests are sufficiently encouraging to warrant further study of these inhibitors.

Other studies brought the following conclusions:

1. The Navy's system of vinyl tape on faying surfaces shows no advantages in salt spray tests but this method of test may not be a fair representation of performance.

2. The Red Oxide Primer, MIL-P-11414, shows performance about equal to the Zinc Chromate Primer, MIL-P-6889a.

3. One coat of a styrenated ether ester primer and one coat of a topcoat based on the same vehicle are equal to or slightly better in salt spray performance than two prime coats and two topcoats of the specification system.

(Turn to page 65)

4. A method has been devised to measure the permeability of paint films to ions. Application of this method with chloride ions may be an indication of the performance of coatings over magnesium. Further work along these lines should be rewarding.

5. The amine-epoxy primer recommended by Emerson & Cuming does not give as good salt spray performance as vinyl aryl ether esters. It is particularly weak in moisture resistance.

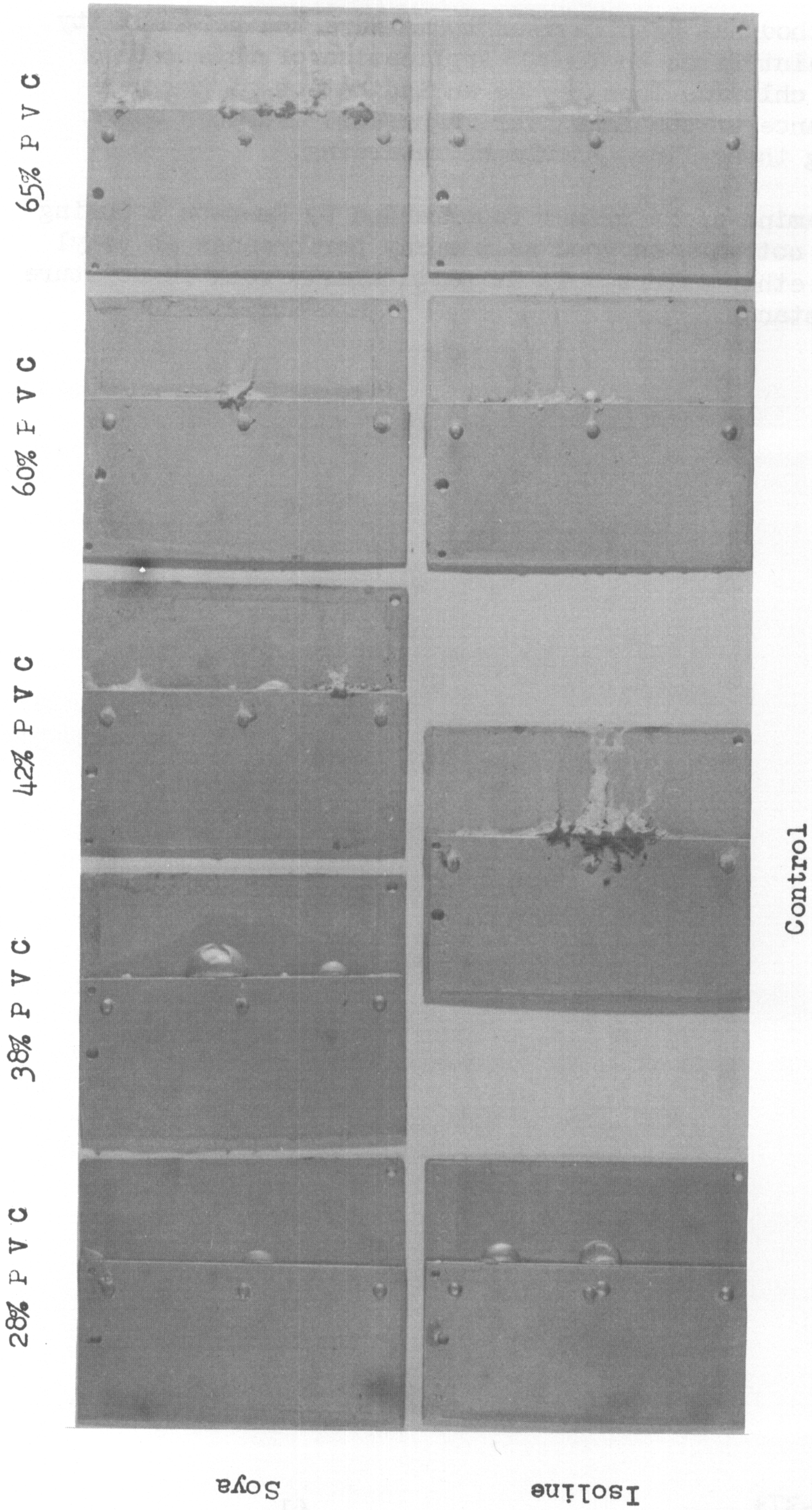

Figure 1. Ether Ester Systems

550 Hours 3% Salt Spray

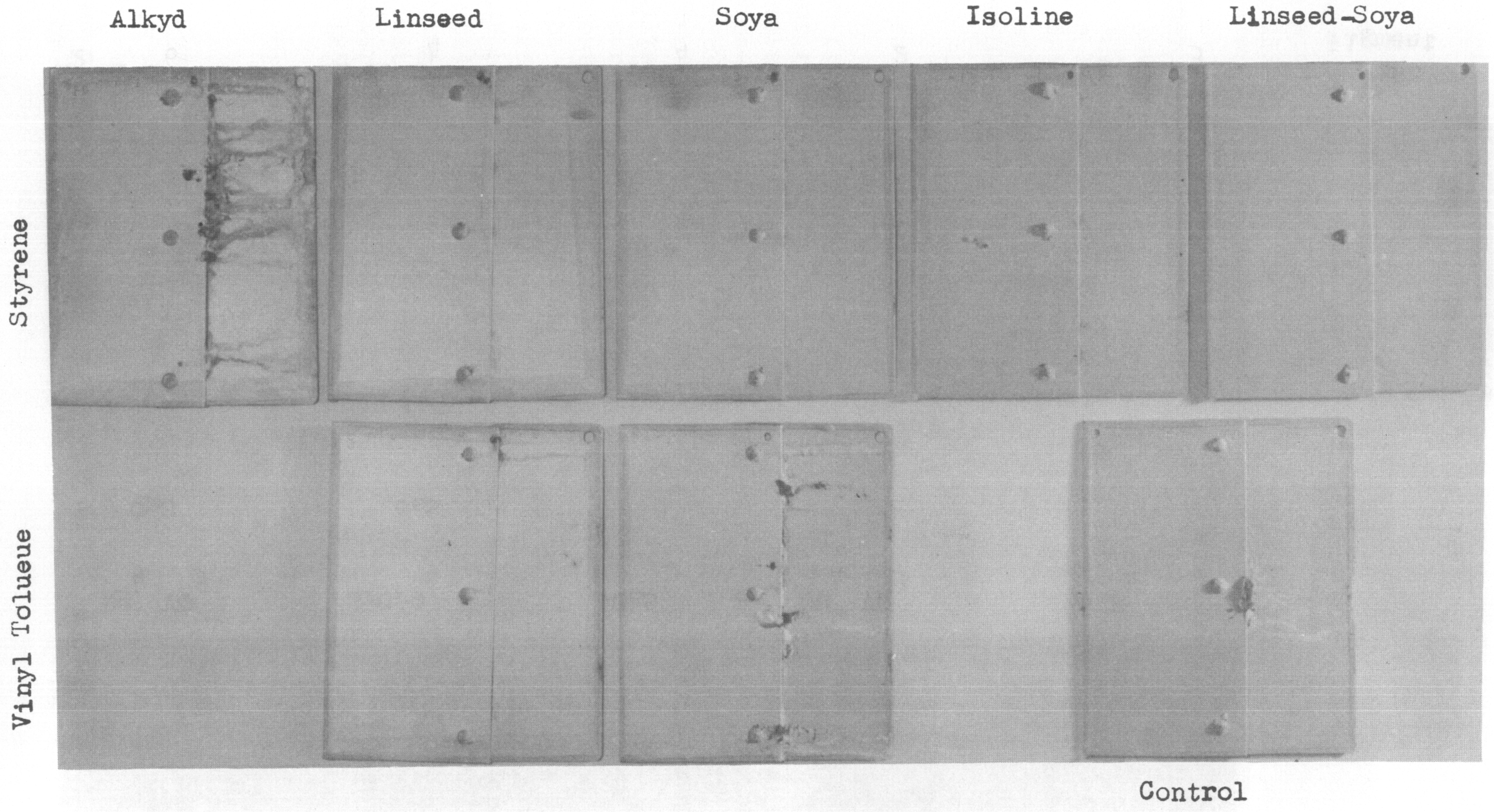

Figure 2. Styrenated Ether Esters

400 Hours 3% Salt Spray

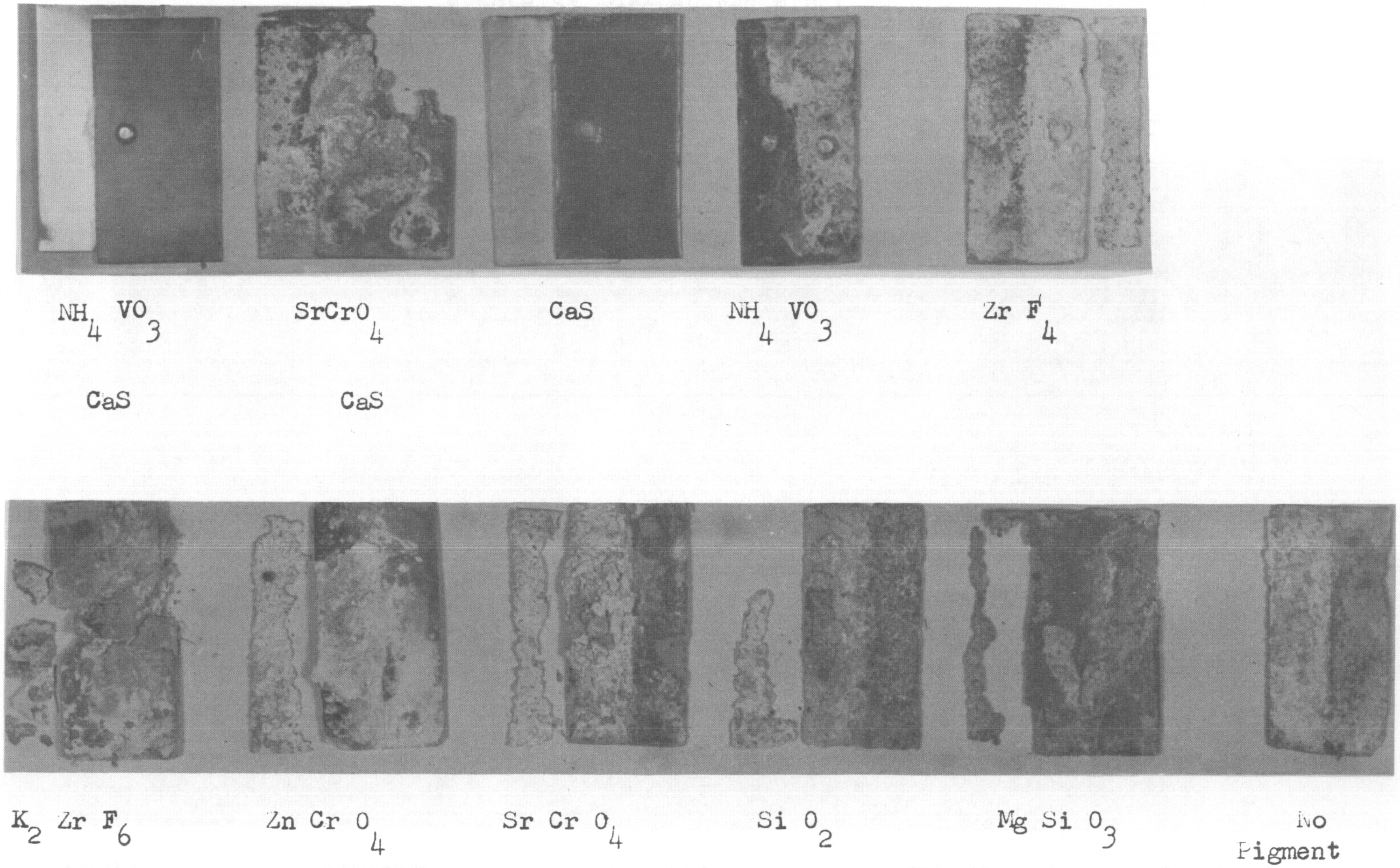

Figure 3. FS1 Magnesium-24ST Aluminum

Pigment Slurries 3% Saline

6 Days at 75°F

AF 141 Series

Summation of Magnesium Finishing Systems
3% Salt Spray - 340 Hours

Magnesium	1.	2.	3.	4.
Pretreatment	MIL-M-3171/III	MIL-M-3171/III	MIL-M-3171/III	MIL-M-3171/III
Primer	MIL-P-6889a/I	Castor-ether ester	Soya-ether ester	VT soya-ether ester
Topcoat	MIL-L-7178	Bakelite VAGH	Soya-ether ester	VT soya-ether ester

Magnesium	5.	6.	7.	8.
Pretreatment	MIL-M-3171/III	MIL-M-3171/III	MIL-M-3171/III	MIL-M-3171/III
Primer	Styrene ether ester + MIL-P-6889a/I	VT ether ester + MIL-P-6889a/I	Bakelite VMCH-VYHH	VT soya-ether ester
Topcoat	MIL-L-7178	MIL-L-7178	Bakelite VAGH	Vinyl alkyd

Magnesium	9.	10.	11.	12.
Pretreatment	MIL-M-3171/III	MIL-M-3171/III	MIL-M-3171/III + MIL-C-15328a	MIL-M-3171/III + MIL-C-15328a
Primer	VT soya-ether ester	Castor phenolic	MIL-P-6889a/I	Castor-ether ester
Topcoat	Acryloid Alkyd	MIL-L-7178	MIL-L-7178	Bakelite VAGH

Magnesium	13.	14.	15.	16.
Pretreatment	MIL-C-15328a	MIL-C-15328a	MIL-M-3171/III + MIL-C-15328a	MIL-M-3171/III
Primer	MIL-P-6889a/I	Castor-ether ester	Bakelite VAGH-VYHH	Bakelite VMCH-VYHH
Topcoat	MIL-L-7178	Bakelite VAGH	Bakelite VYHH	Bakelite VYHH

Note: All aluminum panels were cleaned by acid etching with MIL-C-15328a added to panel 15 and a mixed metal oxide treatment added to panel 16. All topcoats were aluminized.

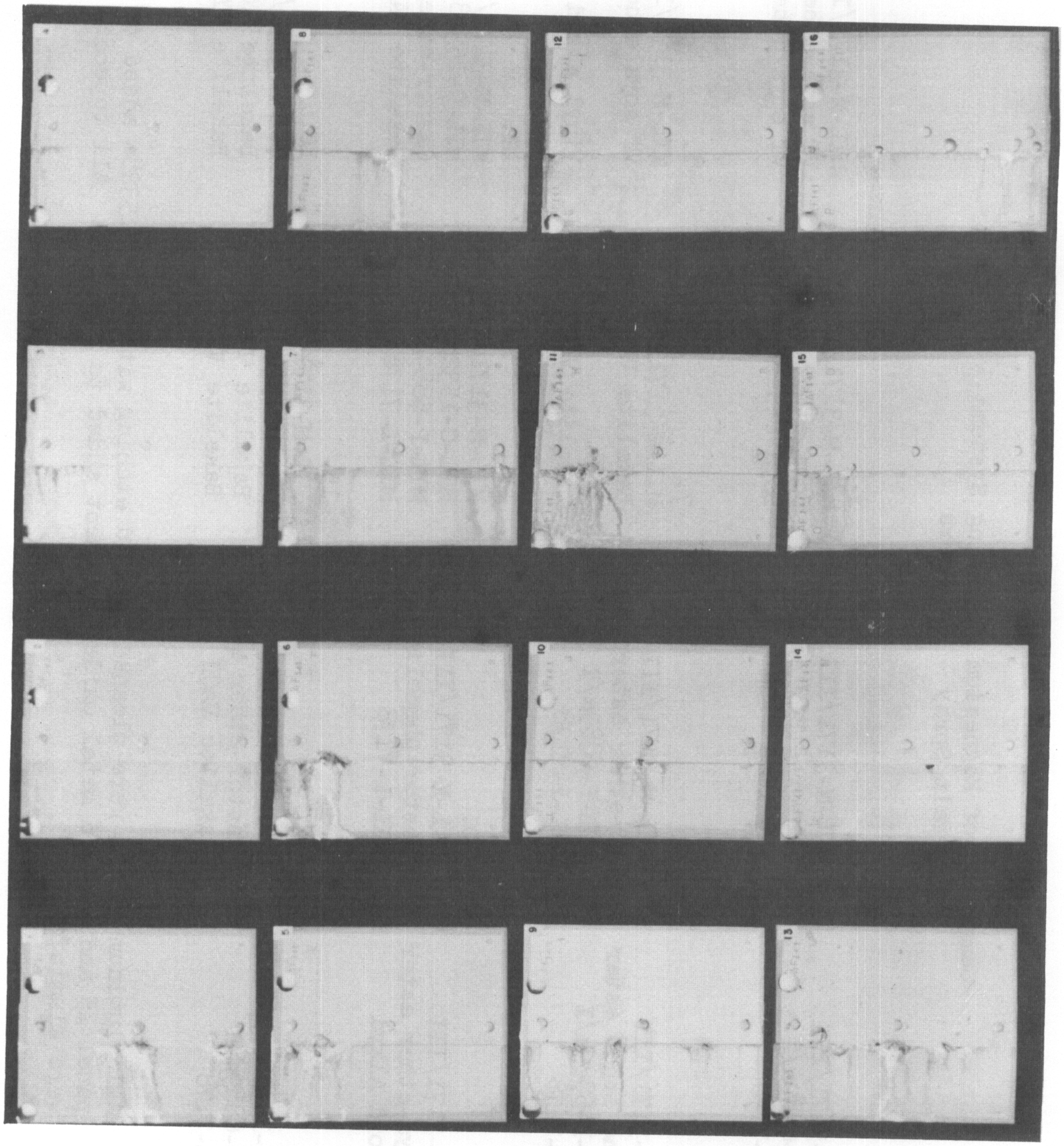

Figure 4. Summation of Magnesium Finishing Systems AF 141 Series

3% Salt Spray 340 Hours

AF 141 Series

Summation of Magnesium Finishing Systems
3% Salt Spray - 340 Hours

Magnesium	1.	2.	3.	4.
Pretreatment	MIL-M-3171/III	MIL-M-3171/III	MIL-M-3171/III	MIL-M-3171/III
Primer	MIL-P-6889a/I	Castor-ether ester	Soya-ether ester	VT soya-ether ester
Topcoat	MIL-L-7178	Bakelite VAGH	Soya-ether ester	VT soya-ether ester
Magnesium	5.	6.	7.	8.
Pretreatment	MIL-M-3171/III	MIL-M-3171/III	MIL-M-3171/III	MIL-M-3171/III
Primer	Styrene ether ester + MIL-P-6889a/I	VT ether ester + MIL-P-6889a/I	Bakelite VMCH-VYHH	VT soya-ether ester
Topcoat	MIL-L-7178	MIL-L-7178	Bakelite VAGH	Vinyl alkyd
Magnesium	9.	10.	11.	12.
Pretreatment	MIL-M-3171/III	MIL-M-3171/III	MIL-M-3171/III + MIL-C-15328a	MIL-M-3171/III + MIL-C-15328a
Primer	VT soya-ether ester	Castor phenolic	MIL-P-6889a/I	Castor-ether ester
Topcoat	Acryloid Alkyd	MIL-L-7178	MIL-L-7178	Bakelite VAGH
Magnesium	13.	14.	15.	16.
Pretreatment	MIL-C-15328a	MIL-C-15328a	MIL-M-3171/III + MIL-C-15328a	MIL-M-3171/III
Primer	MIL-P-6889a/I	Castor-ether ester	Bakelite VAGH-VYHH	Bakelite VMCH-VYHH
Topcoat	MIL-L-7178	Bakelite VAGH	Bakelite VYHH	Bakelite VYHH

Note: All aluminum panels were cleaned by acid etching with MIL-C-15328a added to panel 15 and a mixed metal oxide treatment added to panel 16. All topcoats were aluminized.

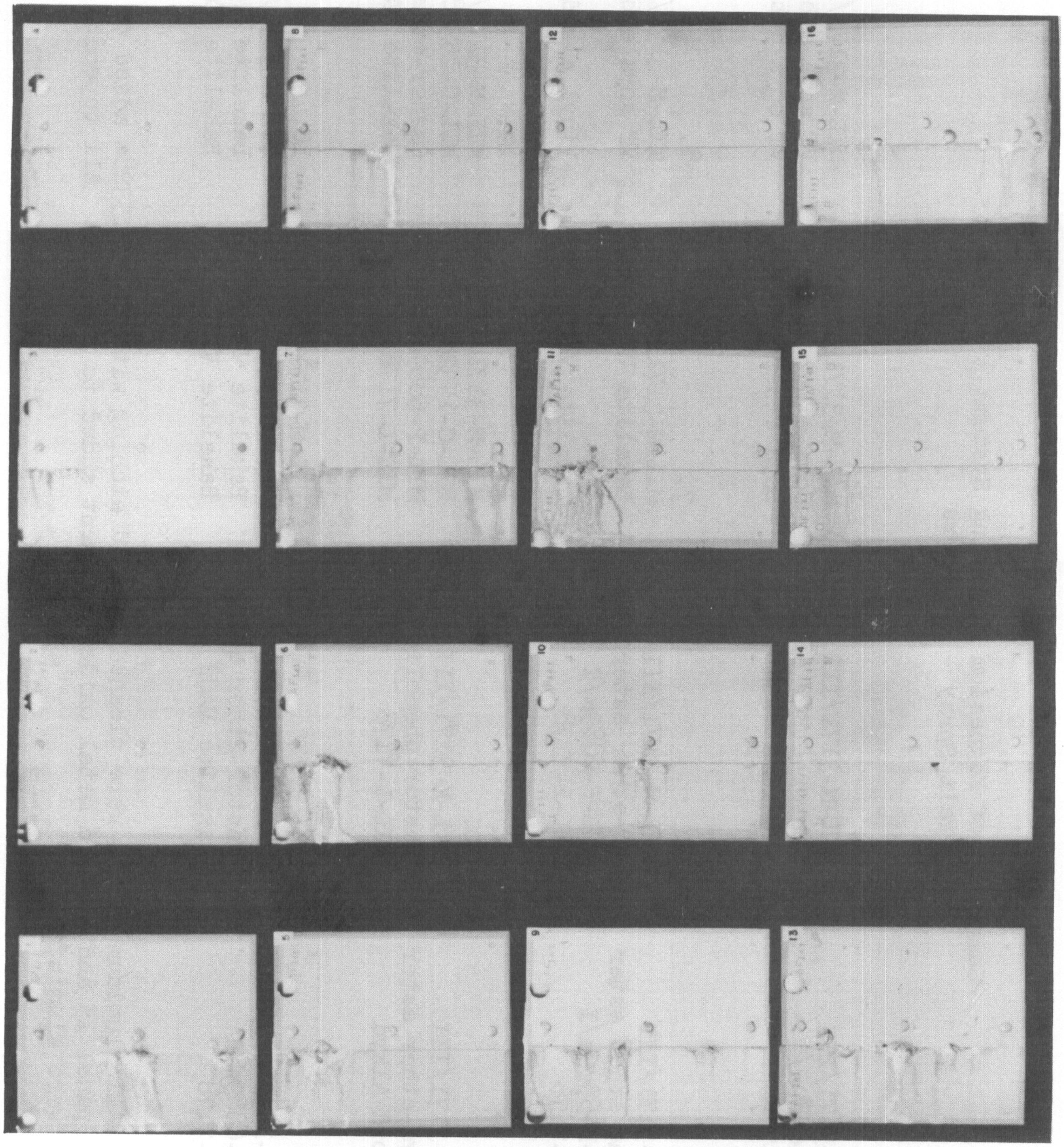

Figure 4. Summation of Magnesium Finishing Systems AF 141 Series

3% Salt Spray 340 Hours

AF 141 Series

Summation of Magnesium Finishing Systems
3% Salt Spray - 672 Hours

	1.	2.	3.	4.
Magnesium Pretreatment	MIL-M-3171/III	MIL-M-3171/III	MIL-M-3171/III	MIL-M-3171/III
Primer	MIL-P-6889a/I	Castor-ether ester	Soya-ether ester	VT soya-ether ester
Topcoat	MIL-L-7178	Bakelite VAGH	Soya-ether ester	VT soya-ether ester
	5.	6.	7.	8
Magnesium Pretreatment	MIL-M-3171/III	MIL-M-3171/III	MIL-M-3171/III	MIL-M-3171/III
Primer	Styrene ether ester + MIL-P-6889a/I	VT ether ester + MIL-P-6889a/I	Bakelite VMCH-VYHH	VT soya-ether ester
Topcoat	MIL-L-7178	MIL-L-7178	Bakelite VAGH	Vinyl alkyd
	9.	10.	11.	12.
Magnesium Pretreatment	MIL-M-3171/III	MIL-M-3171/III	MIL-M-3171/III + MIL-C-15328a	MIL-M-3171/III + MIL-C-15328a
Primer	VT soya-ether ester	Castor phenolic	MIL-P-6889a/I	Castor-ether ester
Topcoat	Acryloid Alkyd	MIL-L-7178	MIL-L-7178	Bakelite VAGH
	13.	14.	15.	16.
Magnesium Pretreatment	MIL-C-15328a	MIL-C-15328a	MIL-M-3171/III + MIL-C-15328a	MIL-M-3171/III
Primer	MIL-P-6889a/I	Castor-ether ester	Bakelite VAGH-VYHH	Bakelite VMCH-VYHH
Topcoat	MIL-L-7178	Bakelite VAGH	Bakelite VYHH	Bakelite VYHH

Note: All aluminum panels were cleaned by acid etching with MIL-C-15328a added to panel 15 and a mixed metal oxide treatment added to panel 16. All topcoats were aluminized.

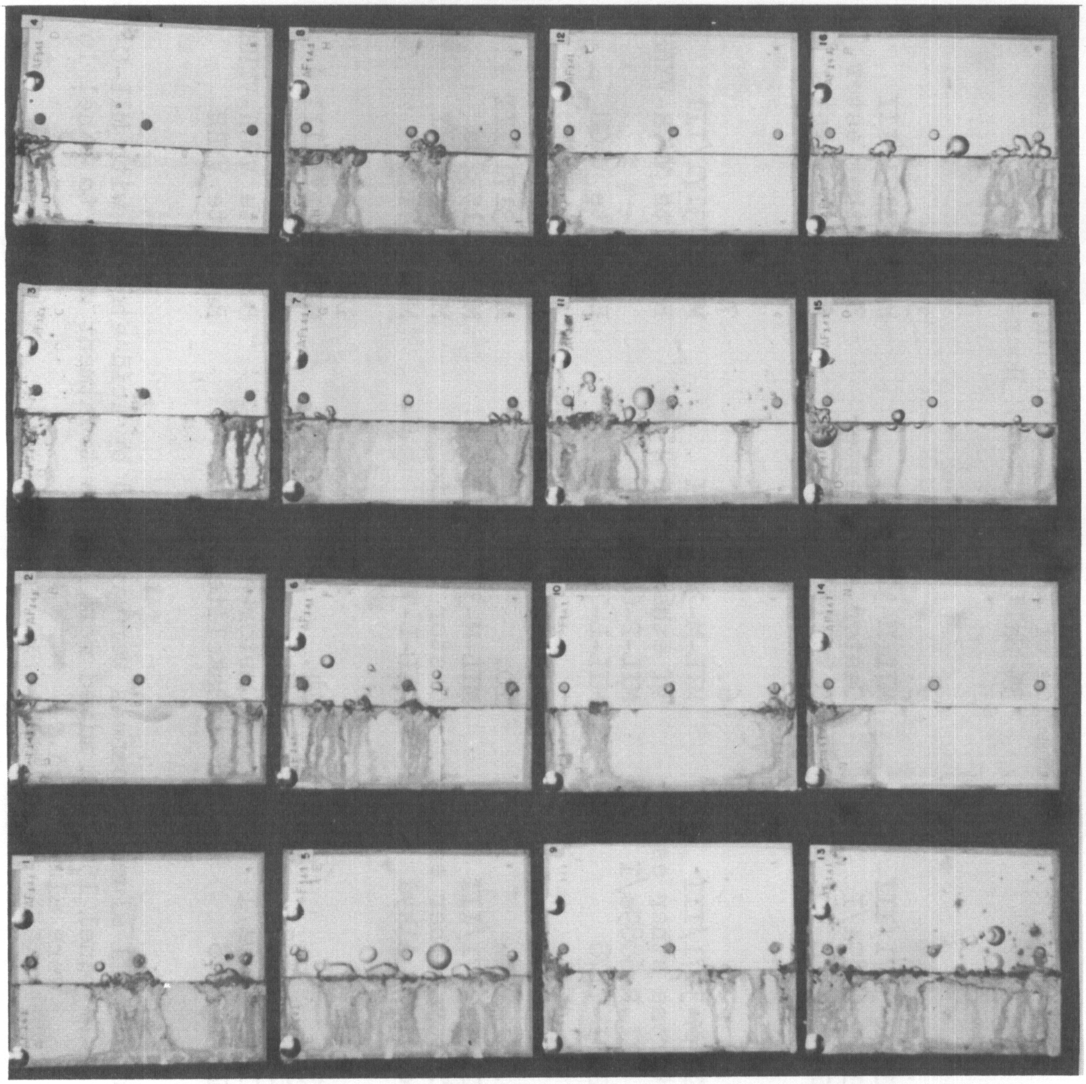

Figure 5. Summation of Magnesium Finishing Systems AF 141 Series

3% Salt Spray 672 Hours

Vinyl Toluene-Ether Ester System

768 Hours - 3% Salt Spray

A	Magnesium Pretreatment	MIL-M-3171, Type III
	Primer	VT soya ether ester
	Topcoat	VT soya ether ester
B	Magnesium Pretreatment	MIL-M-3171, Type III
	Primer	Bakelite VMCH-VYHH
	Topcoat	Bakelite VYHH
C	Magnesium Pretreatment	MIL-M-3171, Type III
	Primer	VT ether ester
	Topcoat	Acryloid Alkyd
D	Magnesium Pretreatment	MIL-M-3171, Type III
	Primer	MIL-P-6889a, Type I
	Topcoat	MIL-L-7178

Note: all topcoats were aluminized.

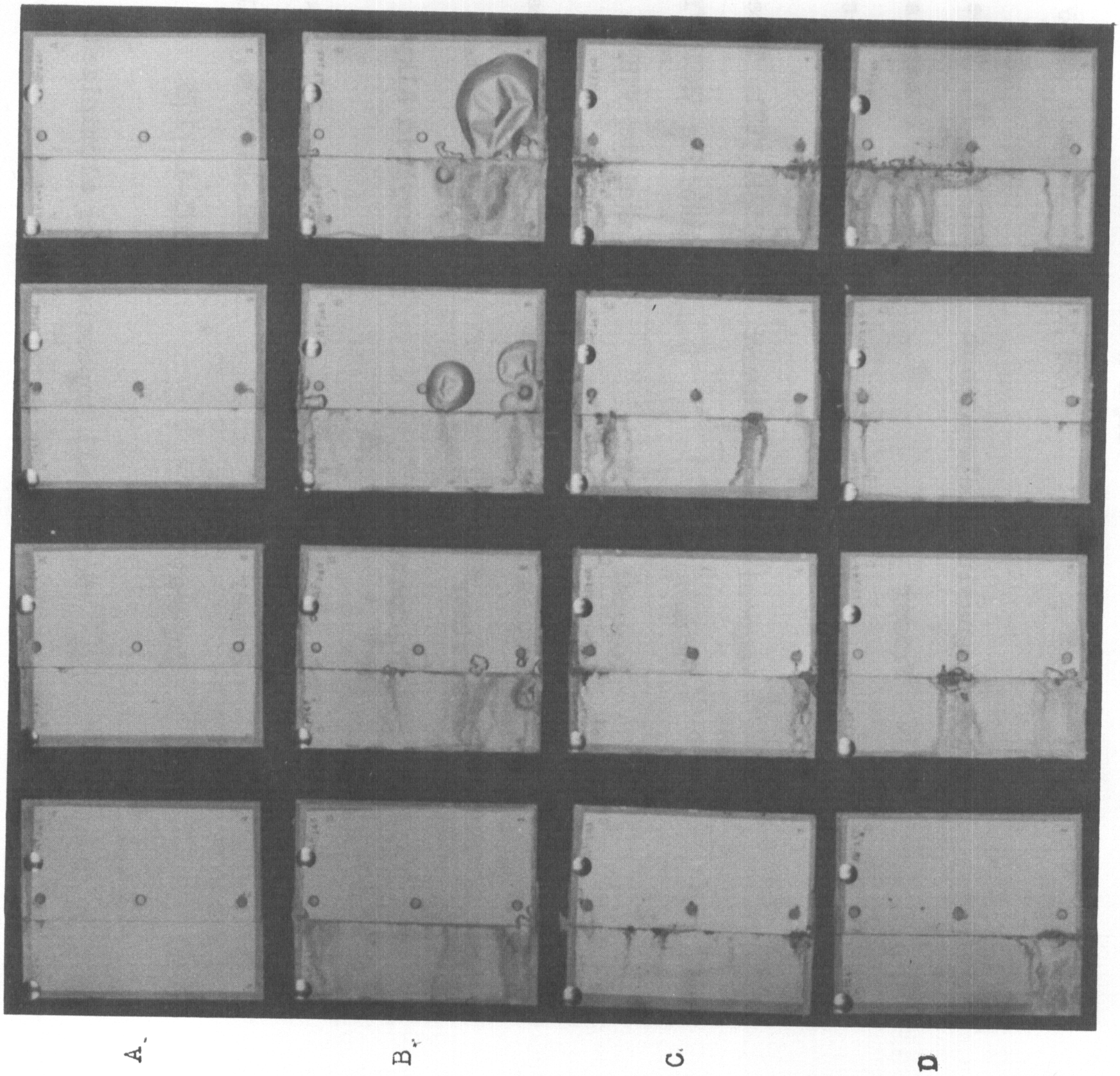

Figure 6. Vinyl Toluene-**E**ther Ester System

768 Hours 3% Salt Spray

Vinyl Toluene-Ether Ester System

1224 Hours - 3% Salt Spray

A	Magnesium Pretreatment	MIL-M-3171, Type III
	Primer	VT soya ether ester
	Topcoat	VT soya ether ester
B	Magnesium Pretreatment	MIL-M-3171, Type III
	Primer	Bakelite VMCH-VYHH
	Topcoat	Bakelite VYHH
C	Magnesium Pretreatment	MIL-M-3171, Type III
	Primer	VT ether ester
	Topcoat	Acryloid Alkyd
D	Magnesium Pretreatment	MIL-M-3171, Type III
	Primer	MIL-P-6889a, Type I
	Topcoat	MIL-L-7178

Note: All topcoats were aluminized.

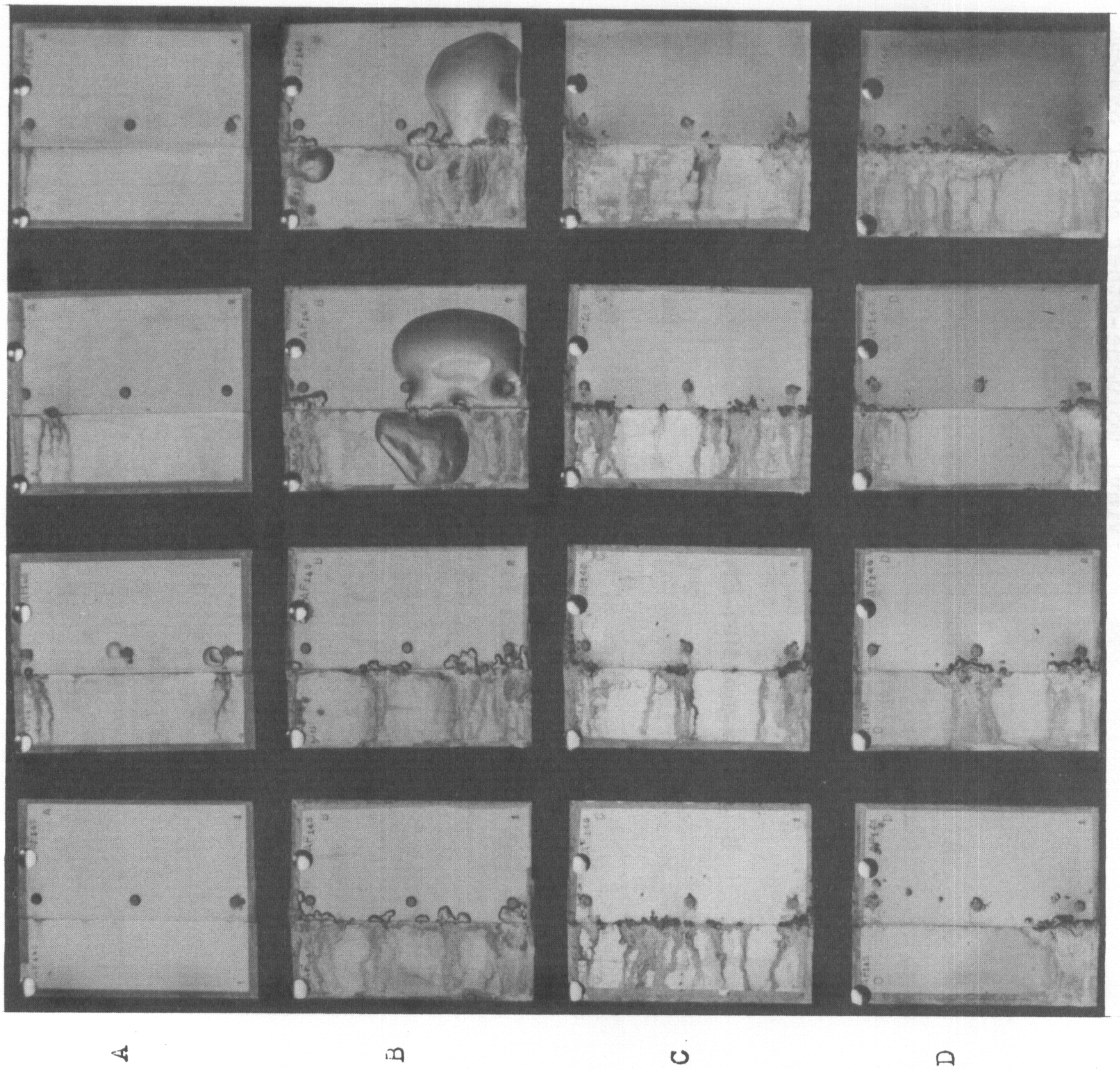

Figure 7. Vinyl Toluene-Ether Ester System

1224 Hours 3% Salt Spray

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APPENDIX

A. Detailed Results

Table I

3% Vs. 20% Salt Spray Testing

Film Thickness		Corrosion Rating					
		25 Hours		100 Hours		225 Hours	
Primer Mils	Topcoat Mils	3%	20%	3%	20%	3%	20%
1.0	1.0	10	6	9	4	8	2
1.0	1.0	10	7	7	2	4	1
1.0	0.9	10	5	7	2	4	1
0.9	0.9	9	4	5	1	3	0

Table 2

Commercial Vs. Laboratory MIL-M-3171 Pretreatments

Identification	Film Thickness	3% Salt Spray Performance - Corrosion					
		24 hr.	48 hr.	72 hr.	168 hr.	216 hr.	312 hr.
Commercial MIL-M-3171, Type III	2.2	10	10	9	8	5	2
	2.2	10	10	10	10	8	7
	2.2	10	10	9	5	3	1
	2.2	10	10	7	4	3	1
	2.1	10	10	9	7	5	2
	2.1	10	9	6	3	3	1
Laboratory MIL-M-3171, Type III	2.1	10	10	10	10	8	8
	2.1	10	10	9	7	4	5
	2.1	10	10	10	9	8	8
	2.2	10	10	8	7	4	4
	2.2	10	10	10	8	6	5
	2.2	10	10	10	10	8	8

Table 3

Ether Ester System Over Bare Magnesium Surfaces

Surface Preparation	Adhesion	3% Salt Spray Performance - Corrosion 216 hr.	336 hr.	432 hr.	672 hr.	840 hr.	936 hr.
Laboratory MIL-M-3171, Type III	8	10	10	10	8	8	7
	8	10	10	10	9	9	8
	8	10	10	10	9	9	9
	8	10	10	9	9	9	9
	8	10	10	10	8	8	8
CrO_3 - NO_3 Treated	6	10	10	9	8	7	4
	6	10	10	10	8	8	6
	6	10	10	10	9	9	8
	6	10	10	10	8	8	8
	6	10	10	10	9	9	9
CrO_3 - NO_3 Treated + HF Treated	2	9	7	5	4	2	1
	2	10	10	9	5	3	2
	2	10	9	9	5	5	5
	2	10	9	9	4	3	3
	2	10	10	9	7	8	8

Table 4

Specification System Over Bare Magnesium Surfaces

Surface Preparation	Adhesion	3% Salt Spray Performance - Corrosion 24 hr.	96 hr.	192 hr.	336 hr.
Laboratory MIL-M-3171, Type III	9	10	10	10	8
	9	10	10	8	5
	9	10	10	10	8
	9	10	10	10	9
	9	10	10	10	9
CrO_3 - NO_3 Treated	9	9	9	8	2
	9	10	10	10	8
	9	10	10	10	8
	9	10	9	9	8
	9	10	9	8	2
CrO_3 - NO_3 Treated + HF Treated	2	10	10	10	8
	2	10	9	8	3
	2	10	9	9	8
	2	10	10	10	8
	2	10	9	9	8

Table 5

Ether Ester System Over Various Chromate Treated Magnesium Surfaces

Surface Preparation	Adhesion	3% Salt Spray Performance - Corrosion						
		216 hr.	336 hr.	432 hr.	672 hr.	840 hr.	936 hr.	1104 hr.
MIL-M-3171, Type I	9	10	9	9	8	7	5	3
		10	10	9	8	7	6	5
		10	10	9	8	7	7	6
		10	10	9	8	7	7	7
		10	10	9	8	8	7	7
Commercial MIL-M-3171, Type III	7	10	10	8	7	6	4	2
		10	10	10	8	8	7	4
		10	10	10	8	8	8	4
		10	10	9	8	8	8	8
		10	9	9	7	5	5	3
Laboratory MIL-M-3171, Type III	8	10	10	10	8	8	7	7
		10	10	10	9	9	8	8
		10	10	9	9	9	9	8
		10	10	10	9	9	9	9
		10	10	9	8	8	8	8
MIL-M-3171, Type II	8	10	10	9	8	8	7	7
		10	10	9	8	8	7	6
		10	10	9	9	9	9	9
		10	10	9	8	8	7	4
		10	10	10	9	9	9	9

Table 6

Specification System Over Various Chromate Treated Magnesium Surfaces

Surface Preparation	Adhesion	3% Salt Spray Performance - Corrosion			
		24 hr.	96 hr.	192 hr.	336 hr.
MIL-M-3171, Type I	9	10	10	10	8
		10	9	9	7
		9	9	8	5
		10	9	9	6
		10	9	7	3
Commercial MIL-M-3171, Type III	9	10	9	8	3
		10	10	9	3
		10	9	9	4
		10	9	8	3
		10	4	10	7
Laboratory MIL-M-3171, Type III (Mottled)	9	10	10	10	9
		10	10	10	9
		10	10	10	7
		10	10	10	8
		10	10	10	8
Laboratory MIL-M-3171, Type III (Good)	9	10	10	10	8
		10	10	8	5
		10	10	10	8
		10	10	10	9
		10	10	10	9
MIL-M-3171, Type II	9	10	10	10	9
		10	9	9	5
		10	9	9	5
		10	9	9	5
		10	9	9	5

Table 7

Ether Ester System With Gross Film Discontinuities

Surface Preparation	Adhesion	3% Salt Spray Performance - Corrosion						
		168 hr.	216 hr.	336 hr.	432 hr.	672 hr.	840 hr.	936 hr.
Commercial MIL-M-3171, Type III	7	10	10	10	8	7	6	4
		10	10	10	10	8	8	7
		10	10	10	10	8	8	8
		10	10	10	10	8	8	8
		10	10	9	9	7	6	5
Commercial MIL-M-3171, Type III Edge Scratched	7	9	8	7	2	1	1	1
		9	8	7	3	1	1	1
		8	7	6	2	1	1	1
		8	7	6	2	1	1	1

Table 8

Specification System with Gross Film Discontinuities

Surface Preparation	Adhesion	3% Salt Spray Performance - Corrosion			
		24 hr.	96 hr.	168 hr.	336 hr.
Commercial MIL-M-3171, Type III	9	10	9	8	3
		10	10	9	3
		10	9	9	4
		10	9	8	3
		10	10	10	7
Commercial MIL-M-3171, Type III Edge Scratched	9	10	8	7	3
		10	6	5	2
		10	5	5	2
		10	6	5	2
		10	6	5	3

Table 9

Performance of Commercial Pretreatment Dichromated Panels

Identification of Pretreatment	3% Salt Spray Performance											
	24 hr.		96 hr.		216 hr.		336 hr.		504 hr.		672 hr.	
	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Commercial #2 Standard Treatment	10	10	9	8	9	7	9	2	7	1	3	2
	10	10	10	9	10	7	9	6	9	4	9	2
	10	10	10	5	9	2	9	1	7	1	4	2
	10	10	10	5	9	6	9	1	9	1	9	2
	10	10	9	9	7	6	5	5	3	4	2	2
Commercial #2 HNO_3 Pickled	10	10	9	2	7	1	6	1	4	1	3	1
	10	10	10	2	10	1	9	1	9	1	7	1
	10	10	9	3	9	1	9	1	9	1	8	1
	10	10	9	2	8	1	7	1	6	1	4	1
	10	10	9	2	9	1	9	1	9	1	9	1
Commercial #1	10	10	9	9	9	7	9	6	8	2	4	1
	10	10	9	9	8	7	7	6	5	2	3	1
	10	10	10	9	10	8	9	7	7	2	4	1
	10	10	9	9	9	6	5	5	3	3	2	1
	10	10	9	3	8	1	6	1	4	1	4	1
Commercial #2 Satin Finish Panels	10	10	10	10	10	10		10	10	9	9	9
	10	10	10	10	10	10		10	9	9	9	9

Table 10

Effect of Additional Rinsing Operations on the Performance of Dichromate Treated Magnesium,#1

		3% Salt Spray Performance									
		24 hr.		96 hr.		192 hr.		360 hr.		1032 hr.	
Identification	Adh.	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Commercial #2 Eight Hour Water Rinse	7	10	10	10	10	9	8	9	4	6	1
		10	10	10	10	10	8	9	4	8	1
		10	10	10	10	10	8	9	4	5	1
		10	10	10	10	10	7	9	4	8	1
		10	10	10	10	9	8	9	4	5	1
Commercial #2 30 Minute Water Rinse	9	10	10	10	10	8	10	7	3	4	1
		10	10	10	10	10	10	9	3	6	1
		10	10	10	10	9	10	9	3	5	1
		10	10	8	10	6	10	6	3	5	1
		10	10	10	7	9	2	9	2	9	1
Commercial #2 As Received	2	10	10	10	1	9	1	9	1	7	1
		10	10	10	1	10	1	9	1	8	1
		10	10	10	1	10	1	9	1	8	1
		10	10	10	1	10	1	9	1	9	1
		10	10	10	1	10	1	9	1	9	1

Table 11

Effect of Additional Rinsing Operations on the Performance of Dichromate Treated Magnesium, #2

Identification		Adhesion	3% Salt Spray Performance							
			72 hr.		168 hr.		404 hr.		740 hr.	
Pretreatment	Primer		Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Commercial #2 30 Minute Water Rinse	Ether Ester + Calcium	7	9	7	8	6	6	5	4	2
			10	9	9	5	9	5	8	2
			10	8	9	7	8	5	5	2
			10	7	9	4	9	4	8	2
Commercial #2 As Received	Ether Ester + Calcium	8	10	5	9	5	9	5	9	3
			9	4	9	4	9	5	8	2
			9	5	9	4	8	5	4	2
			10	5	9	3	9	4	8	2
Commercial #2 30 Minute Water Rinse	Ether Ester + Phenolic	9	9	5	9	4	8	3	4	2
			9	5	8	5	7	3	3	2
			10	9	9	8	9	7	8	3
			9	8	8	8	7	7	5	3
Commercial #2 As Received	Ether Ester + Phenolic	7	10	9	9	7	7	6	5	4
			10	9	9	8	7	7	5	4
			10	9	9	7	7	7	5	4
			10	9	9	8	9	6	8	5
Commercial #2 30 Minute Water Rinse	MIL-P-6889a Type I	3	10	4	9	3	10	2	9	1
			10	2	9	2	10	2	9	1
			9	2	9	2	9	2	9	1
			10	3	9	2	9	2	9	1
Commercial #2 As Received	MIL-P-6889a Type I	1	9	7	9	6	6	4	3	3
			8	5	8	3	8	3	5	2
			9	6	9	4	9	3	5	2
			9	6	9	3	9	3	6	2

Table 12

Effect of Additional Rinsing Operations on the Performance of Dichromate Treated Magnesium, #3

			3% Salt Spray Performance					
			72 hr.		240 hr.		490 hr.	
Dichromate Treatment	Rinse	Adhesion	Cor.	Blister	Cor.	Blister	Cor.	Blister
Commercial #2	None	8	10	10	10	8	8	4
			10	6	9	4	8	2
			10	8	9	5	8	3
			10	10	9	7	8	3
Commercial #2	Tap Water	8	10	6	9	4	9	2
			10	6	9	4	9	2
			10	8	9	4	9	2
			10	9	9	4	9	2
Commercial #2	Tap Water and Distilled Water	8	10	6	8	4	2	1
			10	7	9	4	9	2
			10	8	9	4	9	2
			10	8	9	4	9	2
Commercial #2	Tap Water - Toluene	3	10	10	9	8	7	4
			10	7	9	4	9	3
			10	7	9	4	7	3
			10	6	9	4	8	2
Commercial #2	Toluene	9	10	9	9	8	9	2
			10	8	9	8	8	2
			10	9	7	8	4	2
			10	9	8	8	6	5
Commercial #1	None	6	10	9	8	6	5	2
			10	9	9	7	6	2
			10	9	7	8	3	6
			10	9	9	8	6	1

Table 13

Performance of Mechanically Reclaimed Magnesium

Identification	Film Thickness (mils) Primer	Film Thickness (mils) Topcoat	Adhesion	3% Salt Spray Performance 96 hr. Cor.	96 hr. Blister	168 hr. Cor.	168 hr. Blister	280 hr. Cor.	280 hr. Blister	376 hr. Cor.	376 hr. Blister
Etched Magnesium, Mechanically Cleaned, Dichromate Treated	1.1	1.0	5	10	7	10	4	9	1	9	1
				10	7	10	3	8	1	8	1
				10	7	10	3	8	1	7	1
				10	4	10	2	7	1	7	1
Dichromated Magnesium, Mechanically Cleaned, Dichromate Treated	1.2	1.0	3	10	4	10	4	9	1	8	1
				9	3	8	3	2	1	1	1
				10	3	10	4	9	1	8	1
				10	3	10	4	9	1	9	1
Pickled Magnesium, Dichromate Treated	1.2	1.0	6	10	10	10	10	9	5	9	5
				10	10	10	10	9	5	9	5
				10	10	10	10	9	8	2	8
				10	10	10	10	9	8	8	8

Table 14

Comparison of Paint Systems From Different Sources Over Dichromate Treated Magnesium

Source of Paint	Type of System	Source of Results	Adhesion Ratings 10	9	8	7	6	5	4	3	2	1	0
Dow Chemical	Primer and Topcoat	Dow		4		3	3						
		Contract		5	1		3		1				
Contract	Primer and Topcoat	Dow		1	2	2		1		1	3		
		Contract				3	4			2			
Dow Chemical	Primer Only	Dow		2		2		1		1			
		Contract						2	1	2			
Contract	Primer Only	Dow		2		1		2	1				
		Contract		1				2	1	1			

Table 15

Effect of Primer Formulation on Adhesion to Dichromate Treated Magnesium

Primer Formulation	Topcoat	Total Film Thickness	Adhesion Mg	Adhesion Al	Adhesion Fe
MIL-P-6889a, Type I, Production	--	1.0	3 3 8	8	4
MIL-P-6889a, Type I, Control	--	0.9	9 9 9	9	4
MIL-P-6889a, Type II, Control	--	1.0	7 6 6	9	3
MIL-P-6889a, Type I, Production	MIL-L-7178, Aluminized	2.0	5 7 8	9	6
MIL-P-6889a, Type I, Control	MIL-L-7178, Aluminized	1.9	9 9 9	9	8
MIL-P-6889a, Type II, Control	MIL-L-7178, Aluminized	1.9	9 9 9	6	5

Table 16

Evaluation of Caustic Anodize as a Magnesium Pretreatment

			3% Salt Spray Performance							
			168 hr.		360 hr.		600 hr.		1008 hr.	
Magnesium Pretreatment	Finishing System	Adhesion	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Dichromate Treatment MIL-M-3171 Type III	MIL-P-6889a MIL-L-7178 Aluminized	7	9	9	9	4	7	2	7	2
			9	9	9	4	7	7	6	2
			10	9	9	4	8	6	8	2
			10	9	9	4	8	8	8	2
			7	9	6	4	4	8	4	2
			9	9	9	2	7	1	2	2
Caustic Anodize MIL-M-3171A Type V	MIL-P-6889a MIL-L-7178 Aluminized	9	9	7	9	3	7	2	4	2
			5	7	3	3	3	2	2	2
			9	7	9	3	7	2	3	2
			6	7	4	3	3	2	2	2
			8	7	8	3	5	2	2	2
			9	7	7	3	2	2	2	2

Table 17

Comparison of Caustic Anodize With
Wash Primer as a Magnesium Pretreatment

Magnesium Pretreatment	Finishing System	Adhesion	3% Salt Spray Performance							
			96 hr.		192 hr.		312 hr.		400 hr.	
			Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Wash Primer	Acrylate-VYHH-	6	9	9	8	9	8	9	7	9
Pretreatment	VMCH Primer +		9	9	9	9	7	9	7	9
MIL-C-15328a	Aluminized		9	9	9	9	8	9	7	9
	VYHH Topcoat		9	9	8	9	7	9	7	9
Caustic	Acrylate-VYHH-	9	9	9	8	8	7	8	7	8
Anodized	VMCH Primer +		9	9	9	8	7	8	6	8
MIL-M-3171A	Aluminized		9	9	7	8	6	8	4	8
Type V	VYHH Topcoat		9	9	7	8	6	8	4	8

Table 18

Comparison of Caustic Anodize With
Wash Primer as a Magnesium Pretreatment With Reduced Paint Loading

Magnesium Pretreatment	Finishing System	Adhesion	3% Salt Spray Performance					
			48 hr.		168 hr.		360 hr.	
			Cor.	Blister	Cor.	Blister	Cor.	Blister
Wash Primer	Acrylate-VYHH-	9	10	10	10	9	9	9
MIL-C-15328a	VMCH Primer +		10	10	9	9	9	9
	Aluminized		10	10	10	9	9	9
	VYHH Topcoat							
Wash Primer	Acrylate-VYHH-	8	9	9	9	9	8	9
MIL-C-15328a	VMCH Primer +		9	9	9	9	7	9
	Aluminized		9	9	9	9	7	9
	VYHH Topcoat							
Caustic Anodize MIL-M-3171A Type V	Acrylate-VYHH-	9	10	9	9	8	8	7
	VMCH Primer +		10	9	8	8	8	7
	Aluminized		9	9	9	8	7	7
	VYHH Topcoat							

Table 19

Evaluation of Iridite #15, Mag-Coat

			3% Salt Spray Performance					
			72 hr.		336 hr.		504 hr.	
Pretreatment	Paint System	Adhesion	Cor.	Blister	Cor.	Blister	Cor.	Blister
MIL-M-3171, Type III	MIL-P-6889a, MIL-L-7178	6	10	8	8	4	8	4
			10	7	8	5	7	5
			10	8	8	4	7	4
			10	10	8	3	8	3
MIL-M-3171, Type I	MIL-P-6889a, MIL-L-7178	9	10	4	9	1	8	1
			10	4	9	1	8	1
			10	3	9	1	6	1
			10	4	8	1	7	1
Iridite #15, Mag-Coat	MIL-P-6889a, MIL-L-7178	8	10	4	6	2	6	1
			10	4	6	2	5	1
			10	5	6	2	5	1
			10	5	6	2	5	1

Table 20

Flexibility Tests of Dow #17 Pretreatments

				Bump Test 32 in./lb.		
Dow #17 Application Volts	Paint Coating	Film Thickness in Mils	Adhesion	Intrusion	Extrusion	Conical Mandrel
75	MIL-P-6889a, Type I	1.2	9	9	7	8
90	MIL-P-6889a, Type I	1.2	7	6	5	5
75	Styrenated Ether Ester Primer	1.3	9	9	6	9
90	Styrenated Ether Ester Primer	1.3	7	6	4	6
75	Phenolic Varnish	0.65	6	4	3	7
90	Phenolic Varnish	0.65	5	4	3	5
75	--	--	--	9	6	8
90	--	--	--	7	3	2

Table 21

Salt Spray Tests on Dow #17 Pretreatments

				3% Salt Spray Performance					
				120 hr.		384 hr.		816 hr.	
Pretreatment	Paint Coating	Film Thick.	Adhesion	Cor.	Blister	Cor.	Blister	Cor.	Blister
Dow #17 - 75 Volt	MIL-P-6889a, Type I MIL-L-7178, Aluminized	1.7	9	8	8	5	7	2	6
				8	8	3	7	2	7
				8	9	5	9	3	8
				8	9	4	8	2	9
Dow #17 - 90 Volt	MIL-P-6889a, Type I MIL-L-7178, Aluminized	2.2	8	9	4	8	4	4	3
				8	4	3	4	2	3
				8	4	6	4	3	3
				9	4	8	4	--	--
Dow #17 75 Volt	Styrenated Ether Ester System	1.9	9	10	10	9	10	8	10
				10	10	9	10	7	10
				10	10	9	10	6	10
				10	10	9	10	8	10
Dow #17 90 Volt	Styrenated Ether Ester System	1.9	8	9	10	8	10	7	10
				9	10	9	10	7	10
				10	10	9	10	7	10
				10	10	9	10	--	--
MIL-M-3171 Type III	MIL-P-6889a, Type I MIL-L-7178, Aluminized	1.8	9	8	4	5	3	3	2
				9	3	5	3	3	2
				8	2	7	2	6	2
				9	3	5	1	3	1
MIL-M-3171 Type III	Styrenated Ether Ester System	1.9	9	10	10	9	10	8	9
				10	9	9	10	8	8
				10	10	9	10	8	9
				10	10	9	10	8	9
MIL-M-3171A Type V	Styrenated Ether Ester System	1.9	9	10	10	9	10	8	8
				10	10	9	10	8	8
				10	10	9	10	8	8
				10	10	9	10	8	8

Table 22

Effect of Aging After the Addition of the Acid Diluent on Wash Primers

Age of Base + Acid	Initial		100% RH, 96 hr.		3% Salt Spray, 96 hr.	
	Adhesion	Blistering	Blistering	Adhesion	Blistering	Adhesion
1 hr.	7	3	10	5	10	8
4 hr.	6	3	6	4	9	6
24 hr.	4	10	2	1	10	3

Table 23

Effect of Acid Concentration on Hydrogen Evolution With a Wash Primer

Parts of Acid	Initial		100% RH, 96 hr.		3% Salt Spray, 96 hr.	
	Adhesion	Blistering	Blistering	Adhesion	Blistering	Adhesion
None	8	10	5	1	3	3
1/8	8	3	4	3	10	8
1/4	8	8	6	3	10	8
3/8	8	5	5	2	10	8
1/2 - Normal	8	5	10	4	10	8
5/8	9	8	9	5	9	9
3/4	9	4	10	6	8	9
7/8	9	4	10	5	10	9

Table 24

Effect of Water Concentration on a Proprietary Wash Primer

Per Cent Water Based on Phosphoric Acid	Amount of Blistering From Hydrogen Evolution		Comments
	General	Activated Areas	
165	7	8	Not too sensitive to activated magnesium but some general blistering. Gels in about 30 minutes.
	7	8	
	8	7	
	6	8	
	7	6	
	8	8	
105	5	2	Bad reaction with magnesium, both general and activated.
	4	2	
	6	2	
	6	3	
	6	2	
	6	3	
75	8	8	Best of group, especially for general blistering.
	8	5	
	8	9	
	9	9	
	9	6	
	9	7	
50	7	6	Sensitive to activated magnesium. Gels in about 15 minutes.
	7	6	
	3	4	
	3	6	
	3	5	
	3	6	

Table 25

Effect of Water Concentration on a Specification Wash Primer

Per Cent Water Based On Phosphoric Acid	Adhesion	Amount of Blistering From Hydrogen Evolution	
		General	Activated Areas
120	9	10	8
		10	8
105	9	10	9
		10	3
93	9	9	8
		9	4
85	9	5	4
		5	4
77	9	9	6
		9	9

Table 26

Effect of Water Concentration on a Specification Wash Primer Over Magnesium Surfaces With Varying Activity

Per Cent Water Based on Phosphoric Acid	Type of Magnesium Treatment	Adhesion	Amount of Blistering From Hydrogen Evolution	
			General	Activated Areas
120	Acetic - Nitrate	4	1	9
		4	1	9
		8	2	9
		5	2	9
77	Acetic - Nitrate	5	1	9
		9	5	9
		9	9	6
		9	9	3
120	Chromic - Nitrate	8	2	9
		9	2	9
77	Chromic - Nitrate	9	7	2
		9	8	2

Table 27

Effect of Activator Concentration on Hydrogen Evolution of Specification Wash Primers Over Active Magnesium*

'er Cent of Activator Based on Normal	Adhesion	Amount of Blistering From Hydrogen Evolution: General	Activated Areas
10	9	7	10
		8	10
25	9	9	10
		9	10
40	9	8	10
		8	10
55	9	8	10
		8	10
70	6	9	10
		9	10
85	7	8	10
		4	8
100	7	4	9
		4	9
115	7	5	7
		4	6
130	7	5	8
		3	9

*Acetic acid-nitrate pickled

Table 28

Effect of Activator Concentration on Hydrogen Evolution of Specification Wash Primers Over Normally Active Magnesium*

Per Cent of Activator Based on Normal	Blistering Over Normally Active Magnesium
25	10 10
100	1 1
130	1 1

*Scratched areas on MIL-M-3171, Type III treated panels

Table 29

Performance of Wash Primers Over Bare Magnesium

			3% Salt Spray Performance							
			360 hr.		552 hr.		720 hr.		816 hr.	
Pretreatment	Adhesion	Initial Blistering	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
MIL-M-3171, Type III	5	10	10 9 9	4 8 7	10 9 8	1 3 8	10 9 4	1 2 8	10 9 3	1 2 2
MIL-P-15328 Normal Activator	9	9	9 7 7	9 9 9	9 4 4	3 3 3	9 3 3	3 3 3	9 3 2	3 3 3
MIL-P-15328 25% Normal Activator	9	10	9 8 10	8 9 9	8 8 9	5 5 5	8 5 5	7 7 8	7 5 4	4 4 3

Table 30

Performance of Wash Primers Over Bare and Pretreated Magnesium - Specification System

Substrate	Pretreatment	Adhesion	3% Salt Spray Performance 72 hr. Cor.	 72 hr. Blister	 240 hr. Cor.	 240 hr. Blister	 490 hr. Cor.	 490 hr. Blister	 658 hr. Cor.	 658 hr. Blister
Bare Magnesium	Dichromate	8	10	10	10	8	8	4	6	1
			10	6	9	4	8	2	3	1
			10	8	9	5	8	3	7	1
			10	10	9	7	8	3	7	1
Dichromate Treated Magnesium	Wash Primer	9	10	9	10	9	10	9	9	8
			10	9	9	9	9	9	9	8
			10	9	10	9	8	9	5	8
			10	9	9	9	9	9	8	8
Bare Magnesium	Wash Primer	9	10	5	10	5	9	1	7	1
			10	5	9	5	9	1	8	1
			10	6	10	6	8	2	8	2
			10	5	10	5	5	1	4	1

Table 31

Performance of Wash Primers Over Bare and Pretreated Magnesium - Styrenated Ether Ester System #1

Pretreatment	Primer	Topcoat	Film Thick.	Adhesion	3% Salt Spray Performance 192 hr. Cor.	 192 hr. Blister	 840 hr. Cor.	 840 hr. Blister	 1608 hr. Cor.	 1608 hr. Blister
MIL-M-3171, Type III	Styrenated Ether Ester	Styrenated Ether Ester	2.0	8	10	10	9	9	6	8
					10	10	9	9	4	8
					10	10	9	9	7	9
					10	10	8	9	4	8
MIL-C-15328a	Styrenated Ether Ester	Styrenated Ether Ester	2.1	8	10	2	-	-	-	-
					10	2	-	-	-	-
					10	2	-	-	-	-
					10	2	-	-	-	-
MIL-M-3171 Type III	MIL-C-15328a Plus Styrenated Ether Ester	Styrenated Ether Ester	1.8	8	9	10	8	9	7	8
					9	9	9	7	6	5
					9	7	-	-	-	-
					9	8	9	6	3	3
MIL-M-3171 Type III	MIL-P-6889a Type I	MIL-L-7178 Aluminized	2.0	8	7	9	4	9	2	7
					9	3	4	2	2	1
					10	6	-	-	-	-
					10	8	6	8	3	2

Table 32

Performance of Wash Primers Over Bare and Pretreated Magnesium - Styrenated Ether Ester System #2

		3% Salt Spray Performance									
		96 hr.		432 hr.		768 hr.		1104 hr.		1440 hr.	
Metal Treatment	Paint Systems	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Alkali Cleaned	MIL-C-15328a with	10	2	10	2	9	2	9	2	8	2
	Styrenated Ether	10	2	10	2	9	2	9	2	8	2
	Ester Primer and	10	2	10	2	9	2	9	2	7	2
	Topcoat, Aluminized	10	2	10	2	9	2	8	2	7	2
MIL-M-3171, Type III	MIL-C-15328a with	10	8	9	5	9	5	8	5	7	5
	Styrenated Ether	10	9	6	6	6	6	8	8	8	5
	Ester Primer and	10	9	10	5	9	5	8	5	8	5
	Topcoat, Aluminized	10	10	10	10	9	9	9	8	9	8
Alkali Cleaned	MIL-C-15328a with	10	5	9	5	8	4	5	4	4	4
	Styrenated Ether	10	6	9	5	8	4	5	3	4	3
	Ester Topcoat,	10	6	10	5	8	4	5	3	4	3
	Aluminized	10	5	10	5	8	3	6	3	4	3
MIL-M-3171, Type III	MIL-P-6889a, Type	10	4	8	3	5	3	4	2	2	2
	I and MIL-L-7178,	10	5	8	3	6	3	5	2	3	2
	Aluminized	10	7	10	7	8	5	7	4	4	2
		10	7	8	7	7	7	6	5	3	3

Table 33

Effect of Surface Moisture on Hydrogen Blistering #1

Type of Cleaning	Type of Pretreatment	Type of Wash Primer	Blister Rate
Alkali Clean	Humidity Cabinet	With Activator	1 1
Alkali Clean and Acetic Acid-Nitrate	Humidity Cabinet	With Activator	2 2
Alkali Clean	Oven 115° F	With Activator	1 2
Alkali Clean and Acetic Acid-Nitrate	Oven 115° F	With Activator	4 4

Table 34

Effect of Surface Moisture on Hydrogen Blistering, #2

Type of Cleaning	Type of Pretreatment	Type of Application	Type of Wash Primer	Blister Rate
Alkali Clean	Freezer -10° F	Spray Draw Down	With Activator	3 1
Alkali Clean and Acetic Acid-Nitrate Pickle	Freezer -10° F	Spray Draw Down	With Activator	5 4
Alkali Clean	Oven 115° F	Spray Draw Down	With Activator	2 2
Alkali Clean and Acetic Acid-Nitrate Pickle	Oven 115° F	Spray Draw Down	With Activator	4 6

Table 35

Performance of Wash Primers
Various Alkyl Phosphate Activators

		3% Salt Spray Performance								
		24 hr.		48 hr.		96 hr.		168 hr.		
Identification Acid Activator	Adhesion	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Adhesion
Control - MIL-M-3171, Type III	8	9	10	9	10	8	10	3	10	9
$CH_3H_2 PO_4$	7	10	5	9	5	7	4	3	2	3
$(CH_3)_2 H PO_4$	7	10	10	9	10	9	8	7	2	7
$(CH_3)_2 H_2P_2O_7$	8	10	10	10	9	10	7	7	2	7
$C_2H_5 H_2 PO_4$	9	10	10	10	10	10	9	9	4	9
$(C_2H_5)_2 H PO_4$	8	10	9	10	9	8	9	6	3	7
$(C_2 H_5)_2 H_2 P_2O_7$	5	10	9	9	9	8	8	3	8	8
$C_4H_9 H_2 PO_4$	5	10	7	9	7	9	6	7	2	2
$(C_4H_9)_2 H PO_4$	2	10	10	10	10	7	10	4	6	2
$C_5H_{11} H_2 PO_4$	2	10	10	10	10	9	10	6	4	3
$C_8H_{17} H_2 PO_4$	3	9	10	9	10	7	10	3	5	2
$C_8H_{17} H_2 PO_4$	2	9	10	9	10	8	9	6	7	2
$(C_8H_{17})_2 H_2 P_2O_7$	2	10	10	8	9	8	8	7	3	3
$C_{12} H_{25} H_2 PO_4$	Acid Activator Insoluble in Primer									
$(C_{12} H_{25})_2 H PO_4$	9	10	10	9	10	7	9	3	2	8

Table 36

Effect of Silicone Oil on the Performance of Wash Primers, #1

Silicone Content of Wash Primer	Finishing System	Residual Blister of Wash Primer	Intercoat Adhesion	3% Salt Spray Performance							
				24 hr.		264 hr.		504 hr.		1008 hr.	
				Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
0.0%	MIL-P-6889a	6	9	10	10	9	6	8	4	7	2
	MIL-L-7178	6		10	10	9	3	8	3	7	2
	Aluminized	8									
0.0004%	MIL-P-6889a	4	9	10	10	9	6	8	5	8	2
	MIL-L-7178	4		10	10	9	6	8	5	8	3
	Aluminized	5									
0.004%	MIL-P-6889a	5	9	10	10	9	6	8	5	8	2
	MIL-L-7178	5		10	10	9	6	8	5	8	2
	Aluminized	6									
0.02%	MIL-P-6889a	9	9	10	10	9	8	8	8	8	4
	MIL-L-7178	9		10	10	9	8	8	7	8	4
	Aluminized	9									
0.04%	MIL-P-6889a	9	9	10	10	9	8	8	6	7	3
	MIL-L-7178	9		10	10	9	7	8	5	6	3
	Aluminized	9									
0.2%	MIL-P-6889a	10	9	10	10	9	8	8	6	6	3
	MIL-L-7178	10		10	10	9	7	8	5	6	3
	Aluminized	10									

Table 37

Effect of Silicone Oil on the Performance of Wash Primers, #2

Silicone Content of Wash Primer	Finishing System	Residual Blister of Wash Primer	Intercoat Adhesion	3% Salt Spray Performance			
				24 hr.		48 hr.	
				Cor.	Blister	Cor.	Blister
0.0%	MIL-P-6889a	9	9	10	7	10	5
	MIL-L-7178	9		10	9	10	7
	Aluminized	9		10	7	10	4
		9		10	5	10	6
0.0004%	MIL-P-6889a	10	9	10	5	10	4
	MIL-L-7178	10		10	6	10	3
	Aluminized	10		10	5	9	3
		10		10	4	10	4
0.004%	MIL-P-6889a	9	9	10	4	10	3
	MIL-L-7178	10		10	4	10	2
	Aluminized	10		10	4	10	2
		10		10	4	9	3
0.02%	MIL-P-6889a	9	9	10	6	10	4
	MIL-L-7178	9		10	7	9	5
	Aluminized	10		10	7	10	6
		10		10	7	10	7
0.04%	MIL-P-6889a	10	9	10	5	10	4
	MIL-L-7178	10		10	4	9	3
	Aluminized	10		10	4	10	3
		10		10	7	10	6
0.2%	MIL-P-6889a	10	9	10	8	10	7
	MIL-L-7178	9		10	7	10	5
	Aluminized	9		10	7	10	4
		9		10	9	9	8

Table 38

Evaluation of Wash Primers, #1

			3% Salt Spray Performance					
	Adhesion		24 hr.		72 hr.		192 hr.	
Description	Magnesium	Aluminum	Cor.	Blister	Cor.	Blister	Cor.	Blister
Proprietary Wash Primer	9	9	5	10	3	10	1	4
Butvar B-76 Resimene 881 Wash Primer	9	9	8	10	3	10	1	10
Formal F-770 Wash Primer	6	9	5	10	1	10	1	7
Chromium Phosphate Wash Primer	4	9	9	10	9	10	6	9
Chromic Acid - Phosphoric Acid Treated Butyral Wash Primer	4	9	9	10	9	3	8	3
Control System Over Bare Magnesium	7	9	3	10	2	1	1	1
Control System Over Dichromate Treated Magnesium	6	9	8	10	7	10	7	6

Table 39

Evaluation of Wash Primers, #2

		3% Salt Spray Performance							
		24 hr.		100 hr.		175 hr.		425 hr.	
Description	Lapped Edge Treatment	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
MIL-M-3171, Type III (Control)	Dip Coated	8	9	3	9	3	9		
Proprietary Wash Primer	Dip Coated	9	8	5	8	5	8		
Chromium Phosphate	Dip Coated	9	9	3	8	4	8		
Chromic Acid - Phosphoric Acid Treated Butyral	Dip Coated	8	8	5	6	4	5		
Butvar B-76 -- Resimene 881	Dip Coated	10	5	9	5	7	5		
MIL-M-3171, Type III (Control)	Uncoated	6	9	3	9	2	9		
Proprietary Wash Primer	Uncoated	5	9	3	9	2	9		
Chromium Phosphate	Uncoated	6	9	4	8	2	6		
Chromic Acid - Phosphoric Acid Treated Butyral	Uncoated	5	8	3	5	2	4		
Butvar B-76 - Resimene 881	Uncoated	4	6	3	6	2	5		
MIL-M-3171, Type III + Proprietary Wash Primer	Coated and Removed	10	8	9	5	9	6	3	5
MIL-M-3171, Type III + Chromium Phosphate	Coated and Removed	10	7	10	6	10	6	9	6
MIL-M-3171, Type III + Chromic Acid-Phosphoric Acid Treated Butyral	Coated and Removed	10	7	10	6	9	6	3	4

Table 40

Chrome Phosphate Pigmented Wash Primers

		Film Thickness (mils)		Adhesion	
Substrate	Identification	Mg	Al	Mg	CRS
Dichromate Treated Magnesium	du Pont Chrome Phosphate in XL 5441	2.0	1.5	4	8
Bare Magnesium	" "	2.4	1.5	10	8
Dichromate Treated Magnesium	S-W Chrome Phosphate in XL 5441	2.2	1.4	2	8
Bare Magnesium	" "	2.5	1.5	10	8
Dichromate Treated Magnesium	du Pont Chrome Phosphate in XYHL	2.0	1.5	9	8
Bare Magnesium	" "	2.6	1.4	10	8
Dichromate Treated Magnesium	S-W Chrome Phosphate in XYHL	2.2	1.5	10	8
Bare Magnesium	" "	2.6	1.6	10	8
Dichromate Treated Magnesium	MIL-C-15328a 25% Normal Activator	2.2	1.5	7	8
Bare Magnesium	" "	2.5	1.6	7	8
Dichromate Treated Magnesium	None	2.2	1.6	3	4
Dichromate Treated Magnesium	XYHL - B Stage Phenolic	2.2	1.5	1	1
Bare Magnesium	" "	2.5	1.6	10	1

Table 41

Chrome Phosphate Pigmented Wash Primers Performance

		3% Salt Spray Performance							
		24 hr.		96 hr.		360 hr.		696 hr.	
Substrate	Identification	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Dichromate Treated Magnesium	du Pont Chrome Phosphate in XL 5441	10	10	10	10	8	3	8	1
		10	10	9	10	5	3	3	1
		10	10	8	10	4	6	1	1
		10	10	9	10	5	8	1	1
Bare Magnesium	du Pont Chrome Phosphate in XL 5441	10	10	6	7	1	1	1	1
		10	10	9	7	7	1	3	1
		10	10	9	6	8	1	6	1
		10	10	10	6	4	1	1	1
Dichromate Treated Magnesium	S-W Chrome Phosphate in XL 5441	10	10	9	10	8	1	6	1
		10	10	10	10	8	1	6	1
		10	10	9	9	7	1	6	1
		10	10	9	7	8	1	7	1
Bare Magnesium	S-W Chrome Phosphate in XL 5441	10	10	9	5	8	1	6	1
		10	10	9	5	8	1	4	1
		10	10	9	4	5	1	3	1
		10	10	9	4	7	1	2	1
Dichromate Treated Magnesium	du Pont Chrome Phosphate in XYHL	10	10	10	10	9	10	4	9
		10	10	9	10	9	9	9	8
		10	10	10	10	6	10	2	8
		10	10	9	10	5	10	3	8
Bare Magnesium	du Pont Chrome Phosphate in XYHL	10	10	8	10	3	10	1	9
		10	10	9	10	9	9	8	9
		10	10	10	10	9	10	6	9
		10	10	8	10	3	10	1	9
Dichromate Treated Magnesium	S-W Chrome Phosphate in XYHL	10	10	10	10	9	8	8	3
		10	10	9	10	9	8	8	2
		10	10	10	10	9	6	8	3
		10	10	10	10	9	2	8	1
Bare Magnesium	S-W Chrome Phosphate in XYHL	10	10	9	10	4	9	1	5
		10	10	9	10	7	9	3	5
		10	10	9	10	7	9	2	5
		10	10	9	10	8	9	6	8

(cont.)

		3% Salt Spray Performance							
		24 hr.		96 hr.		360 hr.		696 hr.	
Substrate	Identification	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Dichromate Treated Magnesium	MIL-C-15328a 25% Normal Activator	9	10	9	9	9	2	7	1
		9	10	9	6	7	1	4	1
		9	10	9	8	9	6	6	1
		9	10	9	9	9	2	7	1
Bare Magnesium	MIL-C-15328a 25% Normal Activator	9	10	9	4	9	1	7	1
		9	10	9	4	9	1	7	1
		9	10	9	4	8	1	6	1
		9	10	9	4	9	1	5	1
Dichromate Treated Magnesium	None	10	10	9	10	1	1	1	1
		9	10	8	10	8	1	1	1
		10	10	7	10	2	3	1	1
		10	10	9	10	2	2	4	1
Dichromate Treated Magnesium	XYHL - B Stage Phenolic	9	10	9	10	9	5	4	4
		9	10	8	10	8	9	2	8
		9	10	10	10	5	5	6	4
		10	10	8	10	2	8	3	8
Bare Magnesium	XYHL - B Stage Phenolic	9	10	9	5	2	2	1	1
		10	10	9	5	9	2	5	1
		10	10	9	5	9	1	2	1
		10	9	9	5	8	1	1	1

Table 42

Effect of Varying Vinyl Alcohol Content of Polyvinyl Butyral Resins in Wash Primers

			3% Salt Spray Performance							
			24 hr.		120 hr.		288 hr.		528 hr.	
Pretreatment	Finishing System	Adhesion	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
MIL-C-15328a With High Hydroxyl Butyral Resin	MIL-P-6889a, MIL-L-7178 Aluminized	9	10	10	8	8	7	6	4	5
			10	10	9	8	8	6	8	4
			10	10	9	8	8	4	8	3
			10	10	9	4	8	4	4	2
MIL-C-15328a With Low Hydroxyl Butyral Resin	MIL-P-6889a, MIL-L-7178 Aluminized	9	10	9	9	3	8	2	7	1
			10	9	9	3	8	2	7	1
			10	9	9	3	8	2	8	1
			10	9	9	3	8	2	5	1

Table 43

Evaluation of Wash Primers Prepared from Different Polyvinyl Butyral Resins, #1

		3% Salt Spray Performance									
		48 hr.		216 hr.		348 hr.		552 hr.		888 hr.	
Identification	Wash Primer Mils	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Bakelite XYHL	0.20	10	10	10	9	10	8	10	8	9	8
		10	10	10	8	10	8	10	8	9	8
		10	10	10	9	10	6	10	6	9	5
		10	10	10	9	10	7	10	7	9	5
Monsanto Butvar B76-1	0.20	10	10	10	4	9	4	9	4	8	4
		10	10	10	3	10	3	10	3	9	3
		10	10	10	3	9	3	9	3	8	3
		10	10	10	3	10	3	10	2	9	2
Monsanto Butvar B-90	0.24	10	10	10	10	10	9	9	8	9	8
		10	10	10	10	10	9	10	7	9	6
		10	10	10	10	10	9	9	9	8	8
		10	10	10	10	10	9	10	8	9	8

Table 44

Evaluation of Wash Primers Prepared From Different Polyvinyl Butyral Resins, #2

			3% Salt Spray Performance							
			96 hr.		432 hr.		600 hr.		768 hr.	
Pretreatment	Resin	Finishing System	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Alkali Cleaned	Butvar B-90	Styrenated Ether Ester System	10	2	10	2	9	2	9	2
			10	3	9	3	9	3	9	2
			10	3	9	3	9	3	9	3
			10	3	10	3	10	3	9	2
MIL-M-3171 Type III	Butvar B-90	Styrenated Ether Ester System	10	8	10	6	10	6	9	5
			10	9	9	8	9	8	9	8
			10	5	10	4	10	4	9	4
			10	3	10	2	10	2	10	2
Alkali Cleaned	Butvar B-90	MIL-P-6889a Type I plus MIL-L-7178, Aluminized	9	2	6	2	5	2	3	2
			10	3	8	3	7	3	6	3
			9	3	7	3	6	3	5	2
			10	3	9	3	8	3	7	3
MIL-M-3171 Type III	Butvar B-90	MIL-P-6889a Type I plus MIL-L-7178, Aluminized	10	7	9	5	8	5	7	5
			10	7	10	7	9	6	9	6
			10	6	9	6	9	6	8	6
			10	7	9	7	8	7	7	7
Alkali Cleaned	XYHL	Styrenated Ether Ester System	10	2	8	2	8	2	7	2
			10	2	10	2	9	2	8	2
			10	2	10	2	10	2	9	2
			10	2	9	2	9	2	9	2
MIL-M-3171, Type III	XYHL	Styrenated Ether Ester System	10	9	9	3	9	3	9	3
			10	5	10	3	10	3	10	3
			10	8	10	4	9	4	9	4
			10	9	10	6	9	6	9	6
Alkali Cleaned	XYHL	MIL-P-6889a Type I plus MIL-L-7178 Aluminized	8	3	5	3	4	3	3	3
			9	3	6	3	5	3	4	3
			9	2	8	2	7	2	4	2
			10	3	7	3	6	3	4	3
MIL-M-3171, Type III	XYHL	MIL-P-6889a, Type I plus MIL-L-7178, Aluminized	10	6	9	3	8	3	7	3
			10	5	10	3	10	3	10	3
			10	5	10	3	10	3	9	3
			10	6	9	3	9	3	8	2
MIL-M-3171 Type III	No Wash Primer	MIL-P-6889a Type I plus MIL-L-7178, Aluminized	10	4	8	3	7	3	6	2
			10	8	8	5	7	5	6	4
			10	7	9	6	9	6	8	6
			10	9	8	8	7	8	7	7

Table 45

B Stage Phenolic Coatings

Pretreatment	Wash Primer Type	Film Thick.	Adhesion	3% Salt Spray Performance 96 hr. Cor.	96 hr. Blister	264 hr. Cor.	264 hr. Blister	432 hr. Cor.	432 hr. Blister	768 hr. Cor.	768 hr. Blister
Alkaline Cleaned FS1H Mag.	MIL-C-15328a	2.25	9	10	6	10	5	10	2	8	2
				10	7	10	5	10	2	9	2
				10	7	10	5	10	2	8	2
				10	7	10	5	10	2	8	2
Alkaline Cleaned FS1H Mag.	Phenolic-Polyvinyl Butyral Plus 10% Shellac	2.25	4	10	7	10	6	9	3	6	3
				10	8	10	8	9	5	6	4
				10	7	10	7	9	4	6	3
				10	7	10	7	9	4	7	3
Alkaline Cleaned FS1H Mag.	Phenolic-Polyvinyl Butyral Plus 25% Shellac	2.20	8	10	6	10	5	8	1	5	1
				10	8	8	7	7	2	3	2
				10	8	9	7	9	5	7	3
				10	7	9	7	7	3	1	2
Alkaline Cleaned FS1H Mag.	Phenolic-Polyvinyl Butyral - No Shellac	2.20	8	10	7	10	6	10	2	10	2
				10	7	9	6	8	2	6	1
				10	7	10	6	8	3	8	1
				10	7	10	6	9	2	5	1
MIL-M-3171 Type III	Phenolic-Polyvinyl Butyral Plus 10% Shellac	2.25	6	10	10	10	9	10	8	9	8
				10	10	10	9	10	9	9	8
				10	10	10	9	10	8	9	8
				10	10	10	9	10	9	9	8
MIL-M-3171 Type III	--	2.03	9	10	10	10	10	10	9	8	8
				10	10	10	10	10	9	9	8
				10	10	10	10	10	9	9	8
				10	10	10	10	10	9	8	8

Table 46

Various Chromate Pigments in Wash Primer

		3% Salt Spray Performance							
		24 hr.		192 hr.		336 hr.		528 hr.	
Pretreatment	Pigmentation	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Wash Primer MIL-C-15328a	Basic Zinc Chromate Magnesium Silicate	9	8	9	2	8	1	6	1
		9	6	9	2	8	1	7	1
		9	6	9	1	8	1	6	1
		9	5	9	1	8	1	8	1
Wash Primer MIL-C-15328a	Basic Zinc Chromate Silicon Dioxide	10	7	9	2	7	1	6	1
		10	7	9	1	7	1	6	1
		10	7	9	2	8	1	7	1
		10	7	9	2	6	1	5	1
Wash Primer MIL-C-15328a	Zinc Chromate Silicon Dioxide	10	7	9	2	7	1	7	1
		10	7	9	2	8	1	8	1
		10	7	9	2	7	1	7	1
		10	7	9	1	8	1	7	1
Wash Primer MIL-C-15328a	Calcium Chromate Silicon Dioxide	10	8	9	5	8	2	7	2
		9	6	9	1	8	1	8	1
		9	8	9	5	8	2	7	2
		10	8	9	4	8	1	7	1
Wash Primer MIL-C-15328a	Strontium Chromate Silicon Dioxide	9	9	8	8	6	7	3	6
		10	8	10	7	9	6	8	5
		10	8	10	7	9	6	8	5
		10	8	9	7	9	6	8	6
Wash Primer MIL-C-15328a	Barium-Potassium Chromate Silicon Dioxide	10	8	9	1	8	1	6	1
		10	8	9	2	7	1	5	1
		10	8	8	1	7	1	3	1
		9	8	5	1	4	1	1	1
Dichromated Magnesium + MIL-P-6889a	Standard	10	9	8	4	7	2	5	1
		8	9	6	4	3	2	1	1
		8	9	3	5	1	2	1	1
		9	9	8	5	8	2	8	1

Table 47

Variations in Film Thickness of Wash Primers
Physical Characteristics

Substrate	Pretreatment	Film Thickness	Adhesion	
			Mag.	Steel
Dichromate Treated Magnesium	Wash Primer	0.4	9	9
Bare Magnesium	Wash Primer	0.4	9	9
Bare Magnesium	Wash Primer	0.7	8	8
Bare Magnesium	Wash Primer	1.0	8	8
Bare Magnesium	Wash Primer	1.5	8	9
Bare Magnesium	Wash Primer	2.0	9	7
Bare Magnesium	Dichromate	--	9	5

Table 48

Variations in Film Thickness of Wash Primers Performance

		3% Salt Spray Performance							
		24 hr.		96 hr.		408 hr.		696 hr.	
Substrate	Pretreatment	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Dichromate Treated Magnesium	Wash Primer	10	10	10	10	9	4	7	1
		10	10	10	10	10	7	8	1
		10	10	10	10	8	4	7	1
		10	10	10	10	9	6	7	1
Bare Magnesium	Wash Primer	10	9	10	3	8	1	4	1
		10	9	10	3	7	1	4	1
		10	9	10	3	8	1	5	1
		10	9	10	3	8	1	5	1
Bare Magnesium	Wash Primer	10	10	10	4	8	3	3	1
		10	10	10	4	8	3	4	1
		10	10	10	4	9	3	6	1
		10	10	10	4	6	3	2	1
Bare Magnesium	Wash Primer	10	10	10	4	9	3	7	1
		10	10	10	4	9	3	5	1
		10	10	10	4	9	3	5	1
		10	10	10	4	9	3	5	1
Bare Magnesium	Wash Primer	10	10	9	6	9	5	8	3
		10	10	10	6	9	5	7	3
		10	10	10	6	9	5	8	2
		10	10	10	5	9	5	7	2
Bare Magnesium	Wash Primer	10	10	10	6	9	7	7	3
		10	10	10	7	9	7	7	3
		10	10	10	7	9	8	8	4
		10	10	10	6	9	6	8	3
Bare Magnesium	Dichromate	10	10	10	10	8	8	6	1
		10	10	10	10	9	9	6	1
		10	10	10	10	9	8	7	1
		10	10	10	9	6	8	2	1

Table 49

Comparison of Various Coatings, Coating Weights and Pretreatments

Pretreatment	Finishing System	Number of Coats	Total Film Thick.	Adhesion	3% Salt Spray Performance* 24 hr. Cor.	24 hr. Blister	192 hr. Cor.	192 hr. Blister	360 hr. Cor.	360 hr. Blister	600 hr. Cor.	600 hr. Blister
MIL-M-3171 Type III	Specification	4	2.2	6	10	8	9	7	8	5	6	5
		2	1.0	7	9	7	6	7	4	6	2	6
		1	0.4	2	4	9						
MIL-M-3171 Type III	VMCH Resin	4	2.4	4	9	5	9	5	8	3	7	3
		2	1.1	5	9	8	8	5	6	3	5	2
		1	0.4	2	3	9						
Bare Magnesium	VMCH Resin	4	1.8	9	10	10	9	9	9	4	6	3
		2	1.0	9	10	10	8	7	6	3	3	3
		1	0.3	7	3	10						
MIL-M-3171 Type III	VMCH/VAGH 50/50 by weight	4	1.8	3	10	8	9	6	8	2	7	2
		2	0.9	4	10	7	9	4	7	2	4	2
		1	0.4	2	3	9						
MIL-C-15328a	VMCH/VAGH 50/50 by weight	4	1.8	5	10	10	9	9	9	9	7	7
		2	1.3	7	10	10	9	10	8	7	6	4
		1	0.7	2	6	10						
Bare Magnesium	VMCH/VAGH 50/50 by weight	4	1.8	5	10	8	9	7	8	3	6	3
		2	0.9	8	10	8	9	5	7	3	4	3
		1	0.4	7	3	9						
MIL-M-3171 Type III	VMCH/VAGH 25/75 by weight	4	1.7	3	9	6	8	5	7	1	4	1
		2	0.8	3	9	5	7	4	4	1	1	1
		1	0.3	2	6	7						
MIL-C-15328a	VMCH/VAGH 25/75 by weight	4	1.8	8	10	10	9	10	8	9	6	9
		2	1.3	8	10	10	9	9	6	8	5	8
		1	0.8	3	9	9						
Bare Magnesium	VMCH/VAGH 25/75 by weight	4	1.8	5	10	7	8	5	6	1	4	1
		2	0.9	6	9	8	7	7	7	2	2	2
		1	0.4	7	3	9						
MIL-M-3171 Type III	VAGH Resin	4	2.4	2	10	9	9	6	8	2	6	2
		2	1.3	2	10	9	8	4	6	2	4	2
		1	0.4	2	3	9						

(Cont.)

Pretreatment	Finishing System	Number of Coats	Total Film Thick.	Adhesion	3% Salt Spray Performance* 24 hr. Cor.	24 hr. Blister	192 hr. Cor.	192 hr. Blister	360 hr. Cor.	360 hr. Blister	600 hr. Cor.	600 hr. Blister
MIL-C-15328a	VAGH Resin	4	2.4	9	10	10	9	9	7	7	4	6
		2	1.5	8	10	10	8	10	7	9	4	8
		1	0.7	2	9	9						
MIL-M-3171 Type III	Styrenated Ether Ester	4	2.3	6	10	9	9	7	9	5	8	5
		2	0.9	8	10	8	9	7	8	6	7	4
		1	0.3	4	3	8						
MIL-C-15328a	Styrenated Ether Ester	4	2.5	8	10	10	9	4	8	2	7	2
		2	1.7	9	10	10	9	4	8	2	7	1
		1	0.7	4	10	4						
Bare Magnesium	Styrenated Ether Ester	4	2.5	9	10	10	9	7	8	3	7	2
		2	1.0	9	10	9	9	6	8	2	8	2
		1	0.6	9	5	8						

*Each figure under salt spray performance represents the average performance of four replicate panels.

Table 50

Evaluation of a Vinyl Alkyd System

Pretreatment	Finishing System	Adhesion - Mg Orig.	Adhesion - Mg After Exp.	3% Salt Spray Performance 24 hr. Cor.	24 hr. Blister	96 hr. Cor.	96 hr. Blister	264 hr. Cor.	264 hr. Blister	672 hr. Cor.	672 hr. Blister
MIL-C-15328a	MIL-P-6889a, Type I MIL-L-7178 Aluminized	9	5	10	5	10	5	9	4	4	4
				10	5	9	5	6	5	2	4
				10	6	9	5	9	5	2	4
				10	7	10	7	8	5	3	4
MIL-C-15328a	MIL-P-15934 Aluminized Vinyl Alkyd Topcoat	9	3	10	5	10	3	9	2	7	1
				10	5	10	3	9	2	7	1
				10	6	10	3	9	2	8	2
				10	5	10	3	9	2	6	2

Table 51

Evaluation of a Furan Resin Coating

Magnesium Pretreatment	3% Salt Spray Performance											
	24 hr.		240 hr.		576 hr.		1152 hr.		1920 hr.		2800 hr.	
	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
None	10	6	9	6	7	5	--	--	--	--	--	--
	10	6	10	6	9	5	6	3	4	2	3	1
	10	6	10	6	9	5	6	3	4	2	3	2
Wash Primer	10	7	9	7	9	6	--	--	--	--	--	--
	10	7	10	7	9	6	9	6	9	5	9	5
	10	7	10	7	9	6	9	6	8	5	8	5
MIL-M-3171 Type III	10	9	9	7	8	7	--	--	--	--	--	--
	10	9	9	7	9	7	9	5	9	4	9	1
	10	9	9	7	9	7	9	5	9	4	8	2

Table 52

Summation of the Promising Magnesium Finishing Systems, #1

System Number	Magnesium Pretreatment	Aluminum Pretreatment	Primers	Topcoats
1	MIL-M-3171 Type III	Acid-Etched	MIL-P-6889a, Type I, Proprietary	MIL-L-7178, Aluminized
2	MIL-M-3171 Type III	Acid-Etched	Dehydrated Castor Fatty Acid Ether Ester	Hydroxyl Modified Vinyl Copolymer
3	MIL-M-3171 Type III	Acid-Etched	Styrenated Soya Fatty Acid Ether Ester	Styrenated Soya Fatty Acid Ether Ester
4	MIL-M-3171 Type III	Acid-Etched	Vinyl Toluene-Soya Fatty Acid Ether Ester	Vinyl Toluene-Soya Fatty Acid Ether Ester

(Cont.)

System Number	Magnesium Pretreatment	Aluminum Pretreatment	Primers	Topcoats
5	MIL-M-3171 Type III	Acid-Etched	Styrenated Ether Ester and MIL-P-6889a, Type I, Proprietary	MIL-L-7178
6	MIL-M-3171 Type III	Acid-Etched	Vinyl Toluene-Ether Ester and MIL-P-6889a, Type I, Proprietary	MIL-L-7178
7	MIL-M-3171 Type III	Acid-Etched	Vinyl Copolymer	Hydroxyl Modified Vinyl Copolymer
8	MIL-M-3171 Type III	Acid-Etched	Vinyl Toluene-Soya Fatty Acid Ether Ester	Vinyl Alkyd
9	MIL-M-3171 Type III	Acid-Etched	Vinyl Toluene-Soya Fatty Acid Ether Ester	Acryloid Alkyd
10	MIL-M-3171 Type III	Acid-Etched	Phenolic-Dehydrated Castor	MIL-L-7178
11	MIL-M-3171 Type III plus MIL-C-15328a	Acid-Etched	MIL-P-6889a, Type I, Proprietary	MIL-L-7178
12	MIL-M-3171 Type III plus MIL-C-15328a	Acid-Etched	Dehydrated Castor Fatty Acid Ether Ester	Hydroxyl Modified Vinyl Copolymer
13	Alkaline Degreased plus MIL-C-15328a	Acid-Etched	MIL-P-6889a, Type I, Proprietary	MIL-L-7178
14	Alkaline Degreased Plus MIL-C-15328a	Acid-Etched	Dehydrated Castor Fatty Acid Ether Ester	Hydroxyl Modified Vinyl Copolymer
15	MIL-M-3171 Type III Plus MIL-C-15328a	Acid-Etched Plus MIL-C-15328a	Vinyl Copolymer	Hydroxyl Modified Vinyl Copolymer
16	MIL-M-3171 Type III	Acid-Etched Plus Mixed Metal Oxide	Vinyl Copolymer	Hydroxyl Modified Vinyl Copolymer

Table 53

Summation of the Promising Magnesium Finishing Systems, #2

	3% Salt Spray Performance											
	24 hr.		144 hr.		216 hr.		408 hr.		576 hr.		700 hr.	
System* Number	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister**	Cor.	Blister
1	10	10	8	9	7	8	6	7	6	7	6	6
	10	10	8	10	8	9	7	9	7	5	7	4
	10	10	9	10	8	9	7	9	7	4	7	4
	10	10	8	8	7	5	7	3	7	3	7	3
2	10	10	10	10	10	9	10	5	10	4	9	4
	10	10	10	9	10	7	10	3	10	3	10	3
	10	10	10	9	10	7	10	3	10	3	9	3
	10	10	10	9	9	6	9	4	9	4	9	4
3	10	10	10	9	9	8	9	8	8	6	8	6
	10	10	10	9	10	8	10	8	9	6	8	6
	10	10	10	9	9	8	9	8	8	6	8	6
	10	10	10	7	10	6	9	6	9	4	9	4
4	10	10	10	10	10	10	9	10	8	2	8	2
	10	10	10	10	10	10	9	10	9	2	9	2
	10	10	10	10	10	10	9	10	8	2	8	2
	10	10	10	10	10	10	9	10	9	2	9	2
5	10	10	9	10	8	9	7	9	6	6	6	6
	10	10	9	10	9	10	7	9	7	6	6	6
	10	10	9	10	8	10	7	9	7	6	7	6
	10	10	9	10	9	9	8	9	6	6	6	6
6	10	9	9	9	9	8	8	8	7	6	7	6
	10	7	8	7	8	7	7	7	7	6	6	6
	9	7	8	7	7	7	6	7	6	6	6	6
	10	7	9	7	9	7	8	6	7	3	7	3
7	10	10	10	10	9	10	9	9	8	9	8	8
	10	10	10	10	9	10	8	9	8	6	7	6
	10	10	10	10	10	10	9	10	8	6	8	6
	10	10	10	10	10	10	8	9	8	7	7	7
8	10	10	9	10	8	10	8	9	8	9	8	9
	10	10	10	10	10	10	9	10	9	10	8	9
	10	10	9	10	9	10	8	10	7	10	7	9
	10	10	9	10	9	9	8	9	7	9	7	9

(Cont.)

3% Salt Spray Performance

System* Number	24 hr. Cor.	24 hr. Blister	144 hr. Cor.	144 hr. Blister	216 hr. Cor.	216 hr. Blister	408 hr. Cor.	408 hr. Blister	576 hr. Cor.	576 hr. Blister**	700 hr. Cor.	700 hr. Blister
9	10	9	9	8	8	7	8	7	7	7	6	7
	10	8	9	7	9	6	8	6	7	5	5	5
	10	7	10	7	10	7	8	7	8	7	7	7
	10	9	9	8	9	7	7	7	7	7	7	6
10	9	10	9	10	9	10	8	10	8	6	8	6
	10	10	9	10	9	10	8	10	7	6	7	6
	10	10	9	10	9	10	8	10	8	6	7	5
	10	10	9	10	9	10	8	9	8	2	8	2
11	10	10	10	8	10	8	8	5	8	5	7	4
	10	10	9	8	9	8	7	4	7	3	7	3
	10	10	8	8	8	8	7	5	7	3	6	3
	10	10	8	7	8	7	7	4	7	3	7	3
12	10	10	10	10	9	8	9	6	9	4	9	4
	10	10	10	10	10	8	10	6	10	4	9	4
	10	10	10	9	10	8	10	6	9	4	8	4
	10	10	10	10	10	8	9	6	9	4	9	4
13	10	10	9	7	8	7	7	5	6	3	5	3
	10	10	9	7	9	5	7	3	6	3	6	3
	10	10	8	7	7	6	6	3	5	3	4	3
	10	10	9	8	8	7	6	6	5	3	5	3
14	10	10	10	9	10	8	9	8	8	7	8	7
	10	10	10	8	10	7	9	6	9	6	8	6
	10	10	10	8	10	7	10	6	9	6	8	5
	10	10	10	9	9	8	8	7	8	7	7	6
15	10	10	10	10	10	10	9	10	9	8	8	8
	10	10	10	10	9	10	9	10	8	8	8	8
	10	10	10	10	10	10	9	9	8	8	8	8
	10	10	10	10	9	10	9	9	8	8	8	7
16	10	10	10	10	9	9	9	9	8	8	8	7
	10	10	10	10	9	9	8	9	7	7	7	7
	10	10	10	10	9	9	8	9	7	7	7	7
	10	10	10	10	9	9	8	9	8	6	7	6

*Identifications of system numbers are shown in Table 52.

**It appears that at sometime around 500 hours exposure time on this series the controls failed to function properly. Probably the thermostat allowed the temperature to rise considerably above the 95° F held during normal operation. At any rate all of the panels on exposure at that time showed abnormally severe blistering. This is particularly evident with system #4. The blister results beyond the 408 hour reading have not been considered in interpreting the results.

Table 54

Alkyds With Various Oils Characteristics

Oil Type	Excess Alcohol Per Cent	Cooking Time, Hr.	Acid Value	Cure Sec.	Viscosity G-H	Color Gardner
Guide 1	17	3	38	14	X	12
Guide 2	20	3	29	16	Y	12 +
Linseed	20	5-1/2	8	10	Z	9 +
Soya	20	5	8	11	Z_2	9 +
Fish	20	4-1/2	12	12	Y-Z	13
Castor	20	4-1/3	13	13	Z_3 +	9
Safflower	20	4-2/3	8	16	Z_1-Z_2	7
Oiticica	30	4-3/4	54	20	Z-Z_1	12
Tung	34	5	50	25	V	11
Perilla	30	5	22	12	Z_5-Z_6	15 +

Table 55

Alkyds With Varying Polyol Characteristics

Per Cent Glycerine	Per Cent Penta-erythritol	Excess Alcohol Per Cent	Cooking Time Hr.	Acid Value	Cure Sec.	Viscosity G-H	Color Gardner
100	0	20	3	29	16	Y	12 +
75	25	26	3	35	15	Z_1-Z_2	12
50	50	26	2-1/3	40	14	Z_2	12
25	75	20	2-1/4	56	15	Z_4-Z_5	11 +

Table 56

Alkyds With Varying Oil Length Characteristics

Oil Content	Excess Alcohol Per Cent	Cooking Time, Hr.	Acid Value	Cure Sec.	Viscosity G-H	Color Gardner
10% Less	20	3	32	14	Z-Z_1	12 +
Standard	20	3	29	16	Y	12 +
10% Excess	20	2-1/2	28	13	X-Y	12 +

Table 57

Alkyds With Various Oils Primer Characteristics

Oil Type	Viscosity #3 S-W Cup in Seconds	Settling	Drying - Set-to-touch Clear	Drying - Set-to-touch Pigmented	Film Character	Pencil Hardness	Adhesion to Mg
Proprietary (Production)							9
Proprietary (Laboratory)	45	9	50'	<5'	Tough, flexible	HB	9
Proprietary (Laboratory)	45	8	42	<5	Tough, flexible	HB	9
Linseed	11	9	41	<5	Soft, flexible	5B	9
Soya	9	9	35	O'nite	Slow drying but tough	2B	9
Fish	8	7	84	<5	Tough, flexible	B	9
Castor	188	10	38	<5	Soft, flexible	5B	9
Safflower	11	9	67	<5	Soft, crumbles	B	9
Oiticica	*	9	81	<5	Tough, flexible	F	3
Tung	159	9	34	<5	Tough, flexible	HB	5
Perilla	*	10	20	<5	Slightly soft, flexible	3B	9
Varsoy	*	9	27	<5	Tough, flexible	HB	4

*Too heavy to measure.

Table 58

Alkyds With Varying Polyol
Primer Characteristics

Per Cent Glycerol	Viscosity #3 S-W Cup in Seconds	Settling	Drying - Set-to-touch		Film Character	Pencil Hardness	Adhesion to Mg
			Clear	Pigmented			
100	45	9	45'	<5'	Tough, flexible	HB	8
75	92	9	37	<5	Tough, slightly brittle	HB	9
50	79	9	36	<5	Tough, slightly brittle	HB	7
25	*	10	26	<5	Hard, crumbles	HB	6

*Too heavy to measure.

Table 59

Alkyds With Varying Acid Value
Primer Characteristics

Identification	Viscosity #3 S-W Cup in Seconds	Settling	Drying - Set-to-touch		Film Character	Pencil Hardness	Adhesion to Mg
			Clear	Pigmented			
Production Control							9
Laboratory Control							9
AV 38.3	45	9		<5'	Tough, flexible	HB	9
AV 29.4	45	8	50'	<5	Tough, flexible	HB	9

Table 60

Alkyds With Varying Oil Length
Primer Characteristics

Oil Content	Viscosity #3 S-W Cup in Seconds	Settling	Drying - Set-to-touch Clear	Pigmented	Film Character	Pencil Hardness	Adhesion to Mg
10% Less	54	9	38'	<5'	Tough, flexible	HB	9
Standard	45	9	45	<5	Tough, flexible	HB	9
10% Excess	28	9	39	<5	Tough, flexible	HB	9

Table 61

Miscellaneous Alkyds
Primer Characteristics

Identi-fication	Viscosity #3 S-W Cup in Seconds	Settling	Drying - Set-to-touch Clear	Pigmented	Film Character	Pencil Hardness	Adhesion to Mg
Standard	45	9	45'	<5'	Tough, flexible	HB	9
Metallic Catalyst	26	9	29	<5	Tough, flexible	HB	9
Isophthalic	6	9	--	O'Nite	Tough, flexible	2B	9

Table 62

Critical Pigment Volume Concentration of Alkyd Primers

Identification	CPVC
Proprietary Alkyd - high acid value	41 ± 1
Proprietary Alkyd - normal acid value	40 ± 1
Linseed Alkyd	41 ± 1
Soya Alkyd	38 ± 1
Fish Alkyd	41 ± 1
Castor Alkyd	39 ± 1
Safflower Alkyd	39 ± 1
Oiticica Alkyd	45 ± 1
Tung Alkyd	39 ± 1
Perilla Alkyd	38 ± 1
Varsoy Alkyd	40 ± 1
25% Pentaerythritol/75% Glycerol	38 ± 1
50% Pentaerythritol/50% Glycerol	35 ± 1
75% Pentaerythritol/25% Glycerol	37 ± 1
Proprietary Alkyd - 10% Less Oil	38 ± 1
Proprietary Alkyd - 10% Excess Oil	39 ± 1
Proprietary Alkyd - Metallic Oxide Catalyst	37 ± 1
Isophthalic Alkyd	42 ± 1

Table 63

Alkyds With Various Oils
Salt Spray Performance

	3% Salt Spray Exposure - Corrosion					
Identification	24 hr.	72 hr.	144 hr.	240 hr.	458 hr.	504 hr.
Production Control	10	10	10	10	9	8
Laboratory Control	10	10	8	7	3	3
Laboratory Control	10	9	8	8	5	4
Linseed Alkyd	10	10	9	8	8	8
Soya Alkyd	10	10	9	8	4	3
Fish Alkyd	10	10	7	7	4	3
Castor Alkyd	9	8	5	4	1	1
Safflower Alkyd	10	10	8	7	4	4
Oiticica Alkyd	8	6	4	3	2	2
Tung Alkyd	10	10	8	7	3	3
Perilla Alkyd	10	10	8	6	2	2
Varsoy Alkyd	10	9	6	5	3	2

Table 64

Alkyds With Varying Polyol
Salt Spray Performance

	3% Salt Spray Performance - Corrosion			
Identification	24 hr.	96 hr.	264 hr.	504 hr.
Laboratory Control	10	10	8	5
25% PE/75% Glycerol	10	10	7	2
50% PE/50% Glycerol	10	10	5	1
75% PE/25% Glycerol	10	8	9	7

Table 65

Alkyds With Varying Acid Value
Salt Spray Performance

Identification	3% Salt Spray Performance - Corrosion 24 hr.	96 hr.	168 hr.	264 hr.	432 hr.	504 hr.
Production Control	10	7	6	3	2	1
Laboratory Control	10	9	8	7	4	3
Control Alkyd - AV 39	10	8	7	5	3	2
Control Alkyd - AV 29	10	10	9	6	3	2

Table 66

Alkyds With Varying Oil Length
Salt Spray Performance

Identification	3% Salt Spray Performance - Corrosion 24 hr.	96 hr.	264 hr.	504 hr.
Laboratory Control	10	9	6	4
Proprietary Alkyd - 10% Less Oil	10	8	7	4
Proprietary Alkyd - 10% Excess Oil	10	10	9	7

Table 67

Miscellaneous Alkyds
Salt Spray Performance

Identification	3% Salt Spray Performance - Corrosion 24 hr.	96 hr.	264 hr.	504 hr.
Laboratory Control	10	9	9	8
Metallic Catalyst	10	10	7	5
Isophthalic	10	10	9	7

Table 68

Phenolic Primers at 50% PVC

		3% Salt Spray Performance									
		96 hr.		504 hr.		780 hr.		948 hr.		1452 hr.	
Primer Vehicle	Film Thick.	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
25 gal. Phenolic DCO Varnish	1.9	9	10	9	10	8	9	8	9	5	9
		10	10	9	10	9	10	8	10	7	9
		9	10	9	10	8	9	8	9	7	9
		10	10	9	9	8	9	8	9	7	8
15 gal. Phenolic DCO Varnish	2.0	10	10	9	8	9	7	8	7	7	7
		10	10	9	9	9	7	8	7	7	7
		10	10	9	9	8	7	8	7	7	7
		9	10	9	7	8	6	8	6	6	6
Specification Alkyd	1.9	8	2	7	2	6	2	4	2	2	2
		8	2	7	2	6	2	4	2	2	2
		8	7	6	6	5	5	4	5	2	4
		8	4	5	3	3	2	3	2	1	2

Table 69

Phenolic Primers at 40% PVC, #1

Primer Vehicle	Total Film Thickness (mils)	3% Salt Spray Performance							
		96 hr.		264 hr.		768 hr.		1272 hr.	
		Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Specification Alkyd	2.10	10	9	9	5	6	4	3	3
		10	9	10	5	6	3	3	3
		10	9	10	5	6	3	4	3
		10	9	10	5	6	3	3	3
25 gal. DCO Varnish With Para Phenyl Phenol Resin (50% PVC)	2.10	10	10	10	6	9	3	9	2
		10	10	10	6	9	3	9	3
		10	9	10	5	9	3	9	3
		10	10	10	6	9	3	9	3
15 gal. DCO Varnish With Proprietary Para Tertiary Butyl Phenol Resin (40% PVC)	2.00	10	10	10	5	9	3	9	2
		10	10	10	5	9	3	9	3
		10	10	10	5	9	4	9	4
		10	10	10	5	9	3	8	3
25 gal. DCO Varnish With Proprietary Para Tertiary Butyl Phenol Resin (40% PVC)	2.05	10	10	9	6	9	4	9	3
		10	10	10	6	9	4	9	3
		10	10	9	6	9	4	9	3
		10	10	9	6	8	4	7	3
25 gal. DCO Varnish With Para Phenyl Phenol Resin (40% PVC)	1.95	10	10	10	5	9	4	9	4
		10	10	10	5	9	4	9	4
		10	10	10	5	9	4	9	3
		10	10	10	5	9	4	8	4
15 gal. DCO Varnish With Para Tertiary Butyl Phenol Resin (40% PVC)	1.95	10	10	10	6	9	5	9	4
		10	10	10	5	9	5	9	4
		10	10	10	5	9	5	9	4
		10	10	10	6	9	5	9	4

Table 70

Phenolic Primers at 40% PVC, #2

Primer Vehicle	Total Film Thickness (mils)	3% Salt Spray Performance							
		72 hr.		240 hr.		408 hr.		576 hr.	
		Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Specification Alkyd	2.00	9	9	8	6	6	5	5	4
		10	9	9	6	6	5	5	5
		10	10	10	8	10	8	9	8
		10	10	9	9	8	9	8	8
25 gal. DCO Varnish With Para Phenyl Phenol Resin (50% PVC)	2.00	10	9	9	9	9	8	7	8
		10	10	10	9	9	8	8	7
		10	10	10	9	10	8	9	6
		10	10	10	9	10	7	9	7
15 gal. DCO Varnish With Proprietary Para Tertiary Butyl Phenol Resin (40% PVC)	2.00	9	10	9	9	9	8	8	5
		9	10	9	10	9	9	8	7
		9	10	9	10	9	9	8	7
		10	10	9	9	8	8	8	6
25 gal. DCO Varnish With Proprietary Para Tertiary Butyl Phenol Resin (40% PVC)	2.05	10	10	10	8	9	6	9	5
		10	10	10	9	9	5	9	5
		10	10	10	10	10	9	9	5
		10	10	10	9	10	5	9	5
25 gal. DCO Varnish With Para Phenyl Phenol Resin (40% PVC)	2.15	10	10	9	10	9	9	9	7
		10	10	9	10	9	8	9	6
		10	10	9	10	9	9	9	6
		10	10	9	10	9	9	9	6
15 gal. DCO Varnish With Para Tertiary Butyl Phenol Resin (40% PVC)	2.10	10	10	10	9	10	8	9	6
		10	10	9	9	9	8	9	7
		10	10	9	9	9	8	9	7
		10	10	10	9	9	8	9	7

Table 71

Alkali Resistant Vehicles

Identification	3% Salt Spray Performance Hours of Exposure	Corrosion Rating
Control System with Styrenated Ether Esters	825	5
Control System with Ether Esters	800	1
Control System With Vinyl Copolymers	700	2
Styrene/Linseed Ester of Epon 1004	825	8
Vinyl Toluene/Linseed Ester of Epon 1004	825	8
Dehydrated Castor Ester of Epon 1004	800	8
Soya Ester of Epon 1004	800	8
Acrylic Ester Modified Vinyl Copolymer	700	8
Natural Resin Modified Vinyl Copolymer	700	9

Table 72

Acrylic and Methacrylic Modified Vinyls
Physical Characteristics

Modifying Agent	Package Stability		Drying		Film Character				
					Initial			30 Day	
	Viscosity	Settling	Set-to-Touch Min.	2 hr. Recoat	Pencil Hard.	Adhesion	Toughness	Adhesion	Toughness
Acryloid B-82 Acrylate Copolymer	OK	6	<5	OK	2B	3	7	3	5
Acryloid C-10 Acrylate Copolymer	Gels	6	<5	--	3B	4	5	--	--
Lucite HG 24 Ethyl Methacrylate	OK	2	<5	OK	2B	7	6	4	2
Lucite HG 41 Methyl Methacrylate	OK	2	<5	OK	2B	8	6	8	8
Acryloid A-10 Acrylate Copolymer	OK	6	<5	OK	2B	9	3	9	6
MIL-P-6889a Type I	OK	9	<5	OK	2B	9	8	9	5

Table 73

Acrylic and Methacrylic Modified Vinyls
Salt Spray Performance

	3% Salt Spray Performance									
	72 hr.		168 hr.		240 hr.		504 hr.		672 hr.	
Modifying Agent	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Acryloid B-82 Acrylate Copolymer	10	10	10	10	10	10	10	10	9	10
	10	10	10	10	10	10	10*	10*	8	10
	10	10	10	10	10	10	10	10	9	10
Lucite HG 24 Ethyl Methacrylate	10	10	10	10	10	10	10	9	9	9
	10	10	10	10	10	10	10*	9*	7	9
	10	10	10	10	10	10	10	9	9	7
Lucite HG 41 Methyl Methacrylate	10	10	10	10	10	10	10	10	10	10
	10	10	10	10	10	10	10*	10*	7	9
	10	10	10	10	10	10	10	10	10	10
Acryloid A-10 Acrylate Copolymer	10	10	10	10	10	10	10	9	9	8
	10	10	10	10	10	10	10*	9*	8	8
	10	10	10	10	10	10	10	9	9	7
MIL-P-6889a Type I	10	7	10	7	10	7	9	6	9	2
	10	7	10	6	10	6	9*	5*	6	2
	10	7	10	6	10	6	9	5	9	2

*The coating was removed from one half of each lapped edge on the magnesium panel during this reading.

Table 74

Recapitulation of Vinyl Copolymer and Ether Ester Systems

			3% Salt Spray Performance							
			24 hr.		168 hr.		504 hr.		980 hr.	
Primer	Topcoat	Adhesion	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Acrylate-Vinyl Copolymer	Acrylate Vinyl Copolymer	4	10	10	10	10	10	10	9	9
			10	10	10	10	10	10	9	9
			10	10	10	10	10	10	9	9
Acrylate-Vinyl Copolymer	MIL-L-7178, Aluminized	3	10	10	10	9	10	9	9	6
			10	10	10	4	9	2	9	1
			10	10	10	9	8	8	8	6
Soya-Epon 1004 Ester	Soya-Epon 1004 Ester	8	10	10	10	6	8	4	6	3
			10	10	10	8	8	6	7	8
			10	10	10	7	8	4	7	3
Soya-Epon 1004 Ester	MIL-L-7178, Aluminized	7	10	--	10	3	9	3	7	1
			10	--	9	3	8	2	5	1
			10	--	10	7	8	3	8	1
Styrenated Soya-Epon 1004 Ester	Styrenated Soya-Epon 1004 Ester	8	10	10	10	10	8	10	8	10
			10	10	10	10	8	10	7	10
			10	10	10	10	8	10	5	10
Styrenated Soya-Epon 1004 Ester	MIL-L-7178, Aluminized	8	10	10	9	10	8	10	8	10
			10	10	10	10	8	10	8	10
			10	10	9	10	8	10	8	10
MIL-P-6889a, Type II	MIL-L-7178, Aluminized	5-9	10	10	9	8	8	3	7	1
			10	10	9	8	8	3	3	1
			10	10	9	8	8	3	7	1

Table 75

Recapitulation of Better Magnesium Finishing Systems
Film Characteristics

System	Adhesion	Solvent Resistance 1 hr. Recovery	Solvent Resistance 24 hr. Recovery	Water Resistance Tape Test	Water Resistance Adhesion 24 hr. Recovery
Vinyl Toluene-Ether Ester System	8	6	7	10	8
Vinyl Copolymer System	5	4	5	0	0
Vinyl Toluene Primer-Acryloid Alkyd Topcoat System	9	9	9	10	9
Standard Alkyd Nitrocellulose Control	9	8	9	2	9

Table 76

Recapitulation of Better Magnesium Finishing Systems
Salt Spray Performance

	3% Salt Spray Performance											
	72 hr.		168 hr.		336 hr.		504 hr.		672 hr.		936 hr.	
System	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Vinyl Toluene-Ether Ester Primer-Aluminized Topcoat	10	10	10	10	10	9	10	8	10	3	10	3
	10	10	10	10	10	10	10	9	9	3	9	3
	10	10	10	10	10	10	10	9	9	3	9	3
	10	10	10	10	10	10	10	9	10	3	10	3
Vinyl Copolymer System	10	8	10	8	9	8	8	6	8	6	7	5
	10	9	10	9	9	9	9	7	9	7	7	6
	10	9	10	9	9	9	9	7	9	7	8	5
	10	9	10	9	9	9	9	6	8	6	7	5
Vinyl Toluene-Primer-Acryloid Alkyd Aluminized Topcoat	10	8	10	8	9	8	9	8	8	8	7	6
	9	9	9	9	9	9	9	9	8	8	8	6
	10	9	10	9	9	9	9	7	8	4	7	4
	10	9	9	9	9	9	8	9	8	9	7	9
MIL-P-6889a, Type I plus MIL-L-7178, Aluminized	10	9	9	9	9	8	9	6	8	5	8	5
	9	9	9	9	9	9	8	6	7	6	7	6
	10	8	10	8	9	8	9	3	9	2	8	2
	10	8	10	8	9	8	7	7	6	7	6	7

Table 77

Pretreatments on Aluminum Surfaces, #1

Pretreatment	Type of Coating	Original Adhesion	24 hr. Water Immersion	
			Adhesion	Blister
Solvent Spray	Vinyl Copolymer	7	7	10
Solvent Spray	Wash Primer	9	8	6
H_3PO_4 Etch	Vinyl Copolymer	7	7	10
H_3PO_4 Etch	Wash Primer	9	8	7
Amorphous Phosphate	Vinyl Copolymer	8	8	10
Amorphous Phosphate	Wash Primer	8	6	8
Mixed Metal Oxide	Vinyl Copolymer	9	9	10
Mixed Metal Oxide	Wash Primer	9	9	9

Table 78

Pretreatments on Aluminum Surfaces, #2

Pretreatment	Type of Coating	Original Adhesion	Water Immersion			
			24 hr.		48 hr.	
			Adh.	Blister	Adh.	Blister
Solvent Spray	Vinyl Copolymer	7	7	10	5	10
H_3PO_4 Etch	Vinyl Copolymer	7	7	10	5	10
Amorphous Phosphate	Vinyl Copolymer	8	8	10	7	10
Mixed Metal Oxide	Vinyl Copolymer	9	9	10	9	10

Table 79

Ether Ester Systems - Characteristics

Description			Drying		Film Thickness (Primer Only) Mils		Adhesion	
	Per Cent							
Fatty Acid	CPVC	PVC	Mg	Al	Mg	Al	Mg	Al
Isoline	70	28	10	10	0.9	0.5	9	8
Isoline	70	60	10	10	0.8	0.5	9	8
Isoline	70	65	10	10	0.7	0.4	9	8
Soya	46	28	8	4	0.7	0.4	7	8
Soya	46	38	9	4	0.7	0.4	10*	10*
Soya	46	42	9	6	0.7	0.4	8	9
Soya	46	60	7	4	0.7	0.4	9	10*
Soya	46	65	5	4	0.8	0.5	10*	10*
Control Alkyd	35	40	10	10	0.8	0.5	10*	8

*The rating 10, in this case denotes excellent adhesion but is not meant to denote "Perfect" adhesion.

Table 80

Ether Ester Systems - Salt Spray Performance

	3% Salt Spray Performance									
	25 hr.		150 hr.		225 hr.		350 hr.		500 hr.	
Fatty Acid	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Isoline	10	9	9	8	8	7	8	6	8	6
Isoline	10	10	10	9	9	8	7	6	7	4
Isoline	10	10	10	10	9	9	8	8	7	7
Soya	10	7	10	7	10	6	10	6	9	6
Soya	10	10	9	10	8	10	8	10	8	9
Soya	10	10	9	10	9	10	7	9	7	9
Soya	9	10	9	9	8	8	7	7	6	6
Soya	9	10	6	10	5	10	4	8	4	7
Control Alkyd	10	10	9	10	7	9	6	9	3	9

Table 81

Effect of Additives on the Drying of a Soya Ether Ester Primer

Additive	Tack Free Rating			
	One Hour		Two Hour	
	One Coat	Two Coats	One Coat	Two Coats
None	6	6	6	6
0.1% Co 0.05% Mn (vehicle Solids)	6	6	6	6
0.2% Ca (Vehicle Solids)	6	6	6	6
0.13% Orthophenanthroline (Total Volume)	6	6	6	6
4.0% Linseed-phenolic varnish (Total Volume)	6	6	6	6
6.0% Tetrabutyl-Titanate (Vehicle Solids)	6	6	6	6
(MIL-P-6889a, Type I)	9	9	9	9

Table 82

Effect of Additives on the Recoatability of a Soya Ether Ester Primer

Additive	Recoatability					
	Lifting at Two Hr.			Lifting at 24 Hr.		
	Mg	Al	Fe	Mg	Al	Fe
None	1	1	3	1	1	2
0.1% Co 0.05% Mn (Vehicle Solids)	1	1	3	1	1	2
0.2% Ca (Vehicle Solids)	10	10	10	10	10	10
0.13% Orthophenanthroline (Total Volume)	10	8	10	10	6	10
4.0% Linseed Phenolic Varnish (Total Volume)	10	10	10	10	10	10
6.0% Tetrabutyl Titanate (Vehicle Solids)	10	3	9	10	3	10
(MIL-P-6889a, Type I)	10	10	10	10	10	10

Table 83

Effect of Additives on the Physical Characteristics of a Soya Ether Ester Primer

	Standard System Characteristics at 24 Hr.					
	Magnesium		Aluminum		Steel	
Additive	Adh.	Flex.	Adh.	Flex.	Adh.	Flex.
None	5	8	3	8	5	8
0.1% Co 0.05% Mn (Vehicle Solids)	7	7	6	7	5	7
0.2% Ca (Vehicle Solids)	9	8	8	8	7	8
0.13% Orthophenanthroline (Total Volume)	9	8	8	8	7	8
4.0% Linseed-Phenolic Varnish (total volume)	9	8	7	8	8	8
6.0% Tetrabutyl-Titanate (Vehicle Solids)	6	8	0	--	8	8
(MIL-P-6889a, Type I)	3*	8	1*	8	1*	8

*This same batch of primer has shown excellent adhesion to all three metals.

Table 84

Dehydrated Castor Fatty Acid Ether Ester

Primer Vehicle	Driers Used	Total Film Thick. (Mils)	3% Salt Spray Performance									
			360 hr.		528 hr.		936 hr.		1272 hr.		1804 hr.	
			Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Styrenated	0.4% Pb	1.95	10	10	9	10	9	10	8	10	8	10
Soya Ether			10	10	9	10	9	10	7	10	7	10
Ester			9	10	8	10	7	10	6	10	6	10
			9	10	8	10	7	10	6	10	6	10
Dehydrated	0.5% Pb	1.90	10	10	9	9	9	8	9	6	8	6
Castor	0.05% Co		10	10	9	9	9	8	8	6	7	6
Ether Ester	0.05% Mn		10	10	10	9	10	8	9	6	9	5
			10	10	10	9	10	8	9	6	7	6
Dehydrated	4.0% Linseed	1.90	10	10	9	10	9	9	8	7	7	6
Castor	Phenolic*		10	10	9	10	9	9	9	6	8	6
Ether Ester	0.05% Mn		10	10	9	9	8	8	8	6	7	6
	0.1% Co		10	10	9	9	8	9	8	6	7	6

*4.0% linseed phenolic varnish based upon total volume

Table 85

Effect of Acid Value on Ether Esters

Vehicle	Acid Value	Total Film Thickness (mils)	Adhesion	Hydrocarbon Resistance Immediately After Immersion	Hydrocarbon Resistance 24 hr. After Immersion
Castor Ether Ester	1.2	1.6	8	6	7
Castor Ether Ester	5.5	1.5	7	4	6

Table 86

Topcoats for Ether Ester Primers
Salt Spray Performance

System	Total Film Thick. (mils)	3% Salt Spray Performance							
		144 hr.		408 hr.		744 hr.		1008 hr.	
		Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
MIL-P-6889a, Type I Proprietary-MIL-L-7178, Aluminized	2.00	9	9	7	9	6	8	5	8
		10	9	9	9	8	9	6	8
		10	9	8	9	7	8	5	8
		10	9	8	9	6	8	5	7
Ether Ester Primer-Vinyl Topcoat, Aluminized	1.90	10	8	9	3	9	3	9	2
		10	7	10	3	9	3	9	2
		10	8	10	3	10	3	9	2
		10	9	10	3	10	3	9	2
Sandwich Coat System*	2.05	10	10	9	9	9	9	9	9
		10	10	9	9	8	9	8	9
		10	10	9	9	9	9	8	9
		10	10	9	9	8	9	8	9

*Styrenated ether ester primer with a thin intermediate coat of MIL-P-6889a, Type I, and a topcoat of MIL-L-7178, aluminized.

Table 87

Vinyl Aryl Ether Ester Primers
Characteristics

Description	Drying	Film Thickness Mg Mils	Film Thickness Al Mils	Film Thickness Total (Mg) Mils	Adhesion Mg	Adhesion Al	Adhesion Fe
Styrenated Alkyd	10	1.0	0.6	2.3	7	8	8
Styrenated Linseed Ether Ester	10	1.1	0.6	2.4	5	7	8
Styrenated Linseed-Soya Ether Ester	10	1.1	0.6	2.5	8	7	8
Styrenated Soya-Ether Ester	10	1.0	0.4	2.2	9	9	9
Styrenated Castor-Benzoic Ether Ester	10	1.0	0.5	2.4	5	8	8
Vinyl Toluene-Linseed Ether Ester	10	1.1	0.6	2.5	4	7	8
Vinyl Toluene-Soya Ether Ester	10	1.0	0.6	2.4	9	7	8
Control MIL-P-6889a, Type I	10	1.0	0.4	2.2	9	9	8

Table 88

Vinyl Aryl Ether Ester Primers
Salt Spray Performance

	3% Salt Spray Performance					
	25 Hours		200 Hours		350 Hours	
Description	Corrosion	Blister	Corrosion	Blister	Corrosion	Blister
Styrenated Alkyd	10	10	6	8	5	8
Styrenated Linseed Ether Ester	10	10	9	9	9	9
Styrenated Linseed Soya Ether Ester	10	10	9	9	9	9
Styrenated Soya Ether Ester	10	10	5	7	5	8
Styrenated Castor-Benzoic Ether Ester	10	10	8	9	9	7
Vinyl Toluene-Linseed Ether Ester	10	10	9	9	9	7
Vinyl Toluene-Soya Ether Ester	10	10	8	9	9	9
Control MIL-P-6889a, Type I	10	10	9	9	7	6

Table 89

Review of Previously Selected Systems

			3% Salt Spray Performance							
			24 hr.		168 hr.		504 hr.		980 hr.	
Primer	Topcoat	Adhesion	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Acrylate-Vinyl Copolymer	Acrylate Vinyl Copolymer	4	10	10	10	10	10	10	9	9
			10	10	10	10	10	10	9	9
			10	10	10	10	10	10	9	9
Acrylate-Vinyl Copolymer	MIL-L-7178, Aluminized	3	10	10	10	9	10	9	9	6
			10	10	10	4	9	2	9	1
			10	10	10	9	8	8	8	6
Soya-Epon 1004 Ester	Soya-Epon 1004 Ester	8	10	10	10	6	8	4	6	3
			10	10	10	8	8	6	7	8
			10	10	10	7	8	4	7	3
Soya-Epon 1004 Ester	MIL-L-7178, Aluminized	7	10	--	10	3	9	3	7	1
			10	--	9	3	8	2	5	1
			10	--	10	7	8	3	8	1
Styrenated Soya-Epon 1004 Ester	Styrenated Soya-Epon 1004 Ester	8	10	10	10	10	8	10	8	10
			10	10	10	10	8	10	7	10
			10	10	10	10	8	10	5	10
Styrenated Soya-Epon 1004 Ester	MIL-L-7178, Aluminized	8	10	10	9	10	8	10	8	10
			10	10	10	10	8	10	8	10
			10	10	9	10	8	10	8	10
MIL-P-6889a, Type II	MIL-L-7178, Aluminized	5-9	10	10	9	8	8	3	7	1
			10	10	9	8	8	3	3	1
			10	10	9	8	8	3	7	1

Table 90

Adhesion and Hydrocarbon Resistance of Styrenated Ether Ester Systems

Primers	Topcoats	Adhesion	Hydrocarbon Resistance	Recovery
MIL-P-6889a	MIL-E-7729	7	10	10
Styrenated Ether Ester	MIL-E-7729	8	1	1
Styrenated Ether Ester-MIL-P-6889a	MIL-L-7178	9	2	9

Table 91

Primer Viscosities, 3.3.4

Primer Identification	Viscosity - #1 Zahn - Sec.
MIL-P-6889a, Type I Control	54
Styrenated Ether Ester	69
Carboxyl Modified Vinyl Copolymer	94
Hydroxyl Modified Vinyl Copolymer	128

Table 92

Primer Adhesion Tests, 3.4.6, 3.4.7 and 3.5.4

Primer Identification	Metal Anchorage 3.4.6	Coating Anchorage 3.4.7	Anchorage (Tape) 3.5.4
MIL-P-6889a, Type I Control	7	8	9
Styrenated Ether Ester	7	5	6
Carboxyl Modified Vinyl Copolymer	9	9	1
Hydroxyl Modified Vinyl Copolymer	9	9	2

Table 93

Lacquer Resistance, Primer, Absorption, 3.5.1

Primer Identification	Time Interval Between Primer and Topcoat	Surface Appearance	Adhesion
MIL-P-6889a, Type I, Control	10 Minutes	5	8
	1 hour	6	8
	6 hours	8	8
	16 hours	10	8
	48 hours	10	8
Styrenated Ether Ester	10 minutes	9	8
	1 hour	10	8
	6 hours	10	8
	16 hours	10	8
	48 hours	10	8
Carboxyl Modified Vinyl Copolymer	10 minutes	6	8
	1 hour	7	8
	6 hours	7	8
	16 hours	6	8
	48 hours	7	8
Hydroxyl Modified Vinyl Copolymer	10 minutes	5	9
	1 hour	7	9
	6 hours	7	9
	16 hours	6	9
	48 hours	7	9

Table 94

Water Resistance of Primers, 3.5.2

Primer Identification	Topcoat	Substrate	Adhesion	Blistering
MIL-P-6889a, Type I, Control	None	Acid Etched Al	9	10
	"	Wash primed Al	9	10
	"	Wash Primed Mg	9	9
	"	Dichromated Mg	9	8
	Test Lacquer	Acid Etched Al	7	6
	"	Wash Primed Al	7	9
	"	Wash Primed Mg	8	10
	"	Dichromated Mg	8	10
Styrenated Ether Ester	None	Acid Etched Al	9	10
	"	Wash Primed Al	9	9
	"	Wash Primed Mg	9	8
	"	Dichromated Mg	9	8
	Test Lacquer	Acid Etched Al	4	10
	"	Wash Primed Al	6	10
	"	Wash Primed Mg	6	10
	"	Dichromated Mg	7	10
Carboxyl Modified Vinyl Copolymer	None	Acid Etched Al	7	3
	Test Lacquer	Acid Etched Al	3	3
Hydroxyl Modified Vinyl Copolymer	None	Wash Primed Al	7	5
	Test Lacquer	Wash Primed Al	4	3

Table 95

Hydrocarbon Resistance of Primers, 3.5.3

Primer Identification	Topcoat	Substrate	Adhesion	Blistering
MIL-P-6889a, Type I, Control	None	Acid Etched Al	8	10
	"	Wash Primed Al	8	10
	"	Wash Primed Mg	8	10
	"	Dichromated Mg	8	8
	Test Lacquer	Acid Etched Al	9	4
	"	Wash Primed Al	9	8
	"	Wash Primed Mg	9	9
	"	Dichromated Mg	9	6
Styrenated Ether Ester	None	Acid Etched Al	8	10
	"	Wash Primed Al	8	7
	"	Wash Primed Mg	8	8
	"	Dichromated Mg	8	10
	Test Lacquer	Acid Etched Al	4	0*
	"	Wash Primed Al	4	8
	"	Wash Primed Mg	4	4
	"	Dichromated Mg	7	6
Carboxyl Modified Vinyl Copolymer	None	Acid Etched Al	9	10
	Test Lacquer	Acid Etched Al	8	10
Hydroxyl Modified Vinyl Copolymer	None	Wash Primed Al	9	7
	Test Lacquer	Wash Primed Al	9	8

*Cracked and peeled upon removal from test solution.

Table 96

Topcoat Viscosities, 3.6.3

Topcoat Identification	Viscosity - #3 Ford-Sec.
MIL-L-7178	40
Styrenated Ether Ester	20
Vinyl Copolymer (VYHH)	71

Table 97

Topcoat Adhesion Tests, 3.7.2 and 3.9.4

Primer Identification	Topcoat Identification	Time Interval Between Topcoats	Lifting	Adhesion 48 hr.	Adhesion 1 Mo.
MIL-P-6889a, Type I, Control	MIL-L-7178, Aluminized	4 hr.	10	8	9
		7 hr.	10	8	9
		24 hr.	10	9	9
MIL-P-6889a, Type I, Control	Styrenated Ether Ester, Aluminized	4 hr.	10	5	8
		7 hr.	10	5	8
		24 hr.	10	7	8
Styrenated Ether Ester	MIL-L-7178, Aluminized	4 hr.	10	6	8
		7 hr.	10	6	8
		24 hr.	10	7	8
Styrenated Ether Ester	Styrenated Ether Ester, Aluminized	4 hr.	10	5	8
		7 hr.	10	5	8
		24 hr.	10	6	8
Carboxyl Modified Vinyl Copolymer	Vinyl Copolymer VYHH	4 hr.	10	9	9
		7 hr.	10	9	9
		24 hr.	10	9	9
Hydroxyl Modified Vinyl Copolymer	Vinyl Copolymer VYHH	4 hr.	10	9	9
		7 hr.	10	9	9
		24 hr.	10	9	9

Table 98

Flexibility of Primer-Topcoat Systems, 3.9.6 and 3.9.6.1

Primer Identification	Topcoat Identification	Flexibility	Cold Cracking
MIL-P-6889a	MIL-L-7178, Aluminized	8	3
Styrenated Ether Ester	Styrenated Ether Ester	9	9
Carboxyl Modified Vinyl Copolymer	Vinyl Copolymer VYHH	8	2
Hydroxyl Modified Vinyl Copolymer	Vinyl Copolymer VYHH	8	1

Table 99

Water Resistance of Topcoat Systems, 3.10.1

Primer Identification	Topcoat Identification	Blistering	Adhesion
MIL-P-6889a	MIL-L-7178, Aluminized	10	7
Styrenated Ether Ester	Styrenated Ether Ester, Aluminized	10	5
Carboxyl Modified Vinyl Copolymer	Vinyl Copolymer (VYHH)	3	2
Hydroxyl Modified Vinyl Copolymer	Vinyl Copolymer (VYHH)	2	2

Table 100

Hydrocarbon Resistance of Topcoat Systems, 3.10.2

Primer Identification	Topcoat Identification	Blistering	Adhesion
MIL-P-6889a	MIL-L-7178, Aluminized	10	8
Styrenated Ether Ester	Styrenated Ether Ester, Aluminized	1	3
Carboxyl Modified Vinyl Copolymer	Vinyl Copolymer (VYHH)	10	9
Hydroxyl Modified Vinyl Copolymer	Vinyl Copolymer (VYHH)	10	9

Table 101

Styrenated Ether Ester Systems
Salt Spray Performance
(Pilot Plant Vs. Laboratory)

Primer	Glossy Sea Blue Topcoat	Adhesion	Hydrocarbon Resist.	3% Salt Spray Performance 96 hr. Cor.	96 hr. Blister	360 hr. Cor.	360 hr. Blister	864 hr. Cor.	864 hr. Blister	1200 hr. Cor.	1200 hr. Blister
MIL-P-6889a Type I Proprietary	MIL-L-7178	7	8	10	9	9	9	8	9	7	9
				10	9	10	9	8	9	8	9
				10	8	10	8	9	8	9	8
				10	8	10	8	9	7	8	7
Styrenated Primer With Phenolic	Styrenated	9	5	10	10	9	10	8	10	8	10
				10	10	9	10	8	10	8	10
				10	10	9	10	6	10	6	10
Styrenated Primer Without Phenolic*	Styrenated*	9	5	10	10	10	10	8	10	7	10
				10	10	8	10	3	10	2	10
				10	10	10	10	8	10	7	10
				10	10	9	10	8	10	7	10
Styrenated Primer Without Phenolic	Styrenated	9	7	10	10	10	10	9	10	9	10
				10	10	10	10	10	10	9	10
				10	10	10	10	9	10	9	10
				10	10	10	10	9	10	9	10
Styrenated Primer Without Phenolic	MIL-E-7729	6	6	9	10	8	10	6	8	4	6
				9	10	8	10	7	8	6	6
				9	10	8	10	7	9	6	8
				9	10	9	10	7	9	6	8

*This styrenated ether ester vehicle was a laboratory batch. In all other cases the styrenated vehicle was made in the pilot plant.

Table 102

Styrenated Ether Ester Systems
Weather-Ometer Tests
(Pilot Plant Vs. Laboratory)

Primer	Glossy Sea Blue Topcoat	Gloss Readings, 60° Photovolt						
		Initial	126 hr.	227 hr.	350 hr.	526 hr.	706 hr.	851 hr.
MIL-P-6889a, Type I Proprietary	MIL-L-7178	84	38	39	40	40	41	43
Styrenated Primer With Phenolic	Styrenated	98	91	85	88	84	82	8
Styrenated Primer Without Phenolic*	Styrenated*	100	75	90	88	90	87	4
Styrenated Primer Without Phenolic	Styrenated	100	87-88	84	88	84	83	12
Styrenated Primer Without Phenolic	MIL-E-7729	87	81	82	80	73	74	75

*This styrenated ether ester vehicle was a laboratory batch. In all other cases the styrenated vehicle was made in the pilot plant.

Table 103

Styrenated Ether Ester Systems - Effect of Phenolic Dispersion Resin

Primer	Total Film Thick. (mils)	Adhesion	24 hr. Recovery	
			Water Resistance	Hydrocarbon Resistance
Styrenated Primer With Phenolic	1.45	8	6	5
Styrenated Primer Without Phenolic	1.40	9	8	8

Table 104

Styrenated Ether Ester Systems - Effect of Phenolic Dispersion Resin Salt Spray Performance

	3% Salt Spray Performance									
	264 hr.		432 hr.		768 hr.		936 hr.		1172 hr.	
Primer	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Styrenated Primer With Phenolic	10	10	9	10	8	10	5	10	4	10
	10	10	10	10	9	10	8	10	6	10
	10	10	10	10	9	10	7	10	6	10
	10	10	9	10	8	10	5	10	4	10
Styrenated Primer Without Phenolic	10	10	9	10	8	10	6	10	5	10
	10	10	9	10	9	10	7	10	5	10
	10	10	9	10	8	10	6	10	5	10
	10	10	9	10	9	10	7	10	4	10

Table 105

Drying Times With a Sandwich Coat System

Recoat Time - Sandwich Coat	Recoat Time - Topcoat	Adhesion	Hydrocarbon Resistance	Adhesion - 24 hr. Recovery
24 hr.	24 hr.	6	7	3
6 hr.	18 hr.	7	4*	2
24 hr.	72 hr.	8	9	7

*Small blisters formed.

Table 106

Variations in Vinyl Aryl Ether Ester Vehicles, #1

		3% Salt Spray Performance									
		72 hr.		168 hr.		336 hr.		504 hr.		1176 hr.	
Vehicle	Solvent Resistance	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Styrenated	2	10	10	10	10	10	10	10	10	8	9
Ether Ester-	2	10	10	10	10	10	10	9	10	7	9
Pilot Plant		10	10	9	10	9	10	9	10	6	9
Batch-Control		10	10	9	10	9	10	8	9	5	8
Styrenated	6	10	9	10	3	10	2	10	2	9	2
Ether Ester-	6	10	8	10	2	10	2	9	2	9	2
Reduced Cook-		10	8	10	2	10	2	9	2	9	2
ing Time		10	8	10	3	10	3	9	3	9	3
(20 hr.)											
Vinyl Toluene-	8	10	10	10	5	10	3	10	2	9	2
Divinyl Benzene	8	10	9	10	8	9	5	9	2	9	2
Ether Vehicle		10	9	10	8	10	4	9	2	9	2
"Gel" Technique		10	9	10	8	9	4	9	2	8	2

Table 107

Variations in Vinyl Aryl Ether Ester Vehicles, #2

		3% Salt Spray Performance									
		72 hr.		408 hr.		648 hr.		816 hr.		1162 hr.	
Vehicle	Solvent Resist.	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Styrenated	6	10	10	10	10	10	10	9	10	8	9
Ether Ester	6	10	10	10	10	10	9	9	9	9	8
Pilot Plant		10	10	10	10	9	10	9	9	8	9
Batch-Control		10	10	10	10	9	10	9	10	8	9
Styrenated	8	10	9	10	2	9	2	8	2	5	2
Ether Ester	8	10	9	10	2	9	2	9	2	7	2
Reduced		10	10	10	4	8	4	7	3	4	3
Cooking Time (16 hr.)		10	10	9	7	7	4	6	4	3	3
Vinyl Toluene	8	10	10	10	9	8	9	8	8	7	4
Divinyl Benzene	8	10	10	10	9	10	9	10	8	9	4
Ether Vehicle		10	10	10	9	10	9	9	8	8	4
"Gel" Technique		10	10	10	7	9	6	8	2	7	2

Table 108

Reduced Vinyl Toluene Content Ether Ester, #1

	3% Salt Spray Performance							
	72 hr.		168 hr.		408 hr.		480 hr.	
System	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Sandwich Coat*	9	10	9	10	8	9	8	9
	10	10	10	10	9	10	9	10
	10	10	10	10	9	10	9	10
	10	10	10	10	8	9	8	9
Vinyl Toluene Primer - Aluminized Topcoat	10	10	10	10	9	10	9	9
	10	10	10	10	10	10	9	10
	10	10	10	10	9	10	9	10
	10	10	10	10	10	9	9	8
Styrenated Ether Ester Primer-Aluminized Topcoat	9	10	9	10	8	10	8	10
	10	10	10	10	8	10	8	10
	10	10	10	10	10	10	10	10
	10	10	10	10	9	10	8	10
MIL-P-6889a, Type I, Proprietary-MIL-L-7178, Aluminized	9	9	8	9	7	9	7	8
	8	9	8	9	7	9	6	9
	9	10	8	10	7	10	7	9
	9	10	9	10	7	9	7	9

*Vinyl Toluene Primer with an intermediate coat of MIL-P-6889a, Type I, and a topcoat of MIL-L-7178, aluminized.

Table 109

Reduced Vinyl Toluene Content Ether Ester, #2
Film Characteristics

System	Original Adhesion	Solvent Resistance		Water Resistance	
		Blister	Adhesion 24 hr. Recovery	Tape Test	Adhesion 24 hr. Recovery
Sandwich Coat System	8	10	8	10	8
Vinyl Toluene Ether Ester System	7	10	7	10	7
Styrenated Ether Ester System	6	5	6	10	6
Standard Alkyd Nitro-cellulose System	9	10	9	0*	9

*An abnormal result. This system is expected to give perfect performance on this test.

Table 110

Reduced Vinyl Toluene Content Ether Ester, #2
Salt Spray Performance

	3% Salt Spray Performance											
	72 hr.		408 hr.		480 hr.		672 hr.		840 hr.		1008 hr.	
System	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Sandwich Coat*	9	10	8	9	8	9	7	8	7	6	7	6
	10	10	9	10	9	10	8	9	8	6	7	6
	10	10	9	10	9	10	8	9	8	6	8	6
	10	10	8	9	8	9	7	8	7	6	7	6
Vinyl Toluene Primer-Aluminized Topcoat	10	10	9	10	9	9	8	9	8	6	7	6
	10	10	10	10	9	10	9	10	8	6	8	6
	10	10	9	10	9	10	8	10	8	6	8	6
	10	10	10	9	9	8	9	7	8	5	8	5
Styrenated Ether Ester Primer-Aluminized Topcoat	9	10	8	10	8	10	7	9	7	8	7	8
	10	10	8	10	8	10	7	10	7	7	7	7
	10	10	10	10	10	10	9	10	8	7	8	7
	10	10	9	10	8	10	8	10	7	8	7	8
MIL-P-6889a, Type I Proprietary MIL-L-7178, Aluminized	9	9	7	9	7	8	5	7	5	4	4	4
	8	9	7	9	6	9	5	8	5	5	5	5
	9	10	7	10	7	9	6	9	5	5	5	5
	9	10	7	9	7	9	7	8	6	5	6	5

*Vinyl toluene primer with an intermediate coat of MIL-P-6889a, Type I and MIL-L-7178, Aluminized

Table 111

Topcoats for Ether Ester Primers

Primer	Topcoat	Age of System	Water Resistance Recovery 0 hr.	Water Resistance Recovery 24 hr.	Hydrocarbon Resistance Recovery 0 hr.	Hydrocarbon Resistance Recovery 24 hr.
MIL-P-6889a, Type I	MIL-L-7178	48 hr.	1	9	9	9
		30 days	0	9	9	9
MIL-P-6889a, Type I	Vinyl-alkyd (Proprietary)	48 hr.	1	9	6	6
		30 days	1	9	6	8
Dehydrated Castor Fatty Acid Ether Ester	MIL-L-7178	48 hr.	1	8	8	9
		30 days	1	9	7	8
Dehydrated Castor Fatty Acid Ether Ester	Vinyl-Alkyd (Proprietary)	48 hr.	2	9	5	7
		30 days	1	9	6	8
Styrenated Soya Fatty Acids Ether Ester	MIL-L-7178	48 hr.	9	9	8	8
		30 days	9	9	7	8
Styrenated Soya Fatty Acids Ether Ester	Vinyl-Alkyd (Proprietary)	48 hr.	9	9	1	3
		30 days	9	9	3	7
Vinyl Toluene-Soya Fatty Acids Ether Ester	MIL-L-7178	48 hr.	9	9	2	8
		30 days	9	9	3	8
Vinyl Toluene-Soya Fatty Acids Ether Ester	Vinyl-Alkyd (Proprietary)	48 hr.	9	9	1	3
		30 days	9	9	2	8
Vinyl Toluene-Soya Fatty Acids Ether Ester	Acryloid-Alkyd	48 hr.	9	9	7	9
		30 days	8	9	7	9

Table 112

Topcoats for Ether Ester Primers
Salt Spray Performance

			3% Salt Spray Performance							
			72 hr.		144 hr.		312 hr.		720 hr.	
Primer	Topcoat (Aluminized)	Total Film Thickness (Mils)	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
MIL-P-6889a, Type I (Proprietary)	MIL-L-7178	2.00	9	10	9	9	8	8	3	6
			9	10	8	9	8	9	7	7
			10	10	10	10	10	9	9	7
			10	10	10	10	9	9	6	8
MIL-P-6889a, Type I (Proprietary)	Vinyl-Alkyd (Proprietary)	2.05	8	10	6	2	5	2	2	2
			9	10	9	9	7	7	2	2
			9	10	9	9	8	7	4	2
			9	10	9	8	6	7	2	2
Dehydrated Castor Fatty Acids Ether Ester	MIL-L-7178	2.10	10	10	9	10	9	8	6	4
			10	10	10	9	10	6	9	2
			10	10	10	10	10	8	9	7
			10	10	10	10	10	8	9	7
Dehydrated Castor Fatty Acids Ether Ester	Vinyl-Alkyd (Proprietary)	2.00	9	10	9	9	9	3	8	2
			9	10	9	9	9	3	9	2
			9	10	9	9	9	3	9	2
			10	10	10	9	10	3	10	2
Styrenated Soya Fatty Acids Ether Ester	MIL-L-7178	2.15	9	10	9	10	9	10	6	9
			9	10	8	10	8	10	4	9
			9	10	9	10	9	10	5	9
			9	10	9	10	9	10	6	9
Styrenated Soya Fatty Acids Ether Ester	Vinyl-Alkyd (Proprietary)	2.10	9	10	9	10	9	10	7	9
			9	10	9	10	9	10	8	10
			9	10	9	10	9	10	8	10
			9	10	9	10	9	10	8	10
Vinyl Toluene-Soya Fatty Acids Ether Ester	MIL-L-7178	2.10	10	10	9	10	9	10	9	6
			10	10	9	10	9	9	9	4
			10	10	10	10	10	8	9	3
			10	10	10	10	10	9	9	3
Vinyl Toluene-Soya Fatty Acids Ether Ester	Vinyl-Alkyd (Proprietary)	2.15	10	10	10	10	10	8	9	4
			10	10	10	10	10	10	9	7
			10	10	10	10	10	6	9	4
			10	10	10	8	10	4	9	3
Vinyl Toluene-Soya Fatty Acids Ether Ester	Acryloid-Alkyd	1.85	10	8	9	8	9	8	8	4
			10	8	9	8	9	7	9	4
			9	7	9	7	9	7	8	4
			10	8	10	8	9	7	9	4

Table 113

Glossy Sea Blue Vinyl Aryl Ether Ester Topcoats Weather-Ometer

Glossy Sea Blue System	Gloss Readings*				
	Original	86 hr.	207 hr.	387 hr.	700 hr.
Vinyl Toluene-Ether Ester System	86	68	60	57	2
Styrenated Ether Ester System	97	95	90	88	1
Standard Alkyd Nitrocellulose System	89	86	86	80	68

*Taken on a 60° Photovolt Gloss Meter.

Table 114

Reduced Vinyl Toluene Content Ether Ester Vehicles Characteristics

Vehicle	#1	#2	#3	#4	#5
Solids Content	50%	50%	50%	50%	50%
Body	U-V	U +	X-Y	U +	T
Acid Value	0.7	0.9	0.9	0.6	0.6
Cure	8 sec.	8 sec.	8 sec.	12 sec.	9 sec.
Color	6 hazy	5-6 hazy	7 hazy	5 hazy	5 hazy
Wt./Gal.	7.99 lb.	7.97 lb.	8.01 lb.	7.96 lb.	8.00 lb.

Table 115

Vinyl Toluene-Ether Ester Systems
Pilot Plant Batch

	3% Salt Spray Performance											
	24 hr.		192 hr.		360 hr.		528 hr.		696 hr.		864 hr.	
Vehicle	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Vinyl Toluene-Ether Ester Vehicle #2	10	10	10	9	9	8	9	8	9	8	9	8
	10	10	10	9	10	9	10	9	9	9	9	9
	10	10	10	10	10	9	10	9	9	8	9	8
	10	10	10	10	10	7	10	7	9	6	9	5
Vinyl Toluene-Ether Ester Vehicle #3 (Pilot Plant Batch)	10	10	10	10	10	9	10	8	10	7	10	7
	10	10	10	10	10	7	10	7	10	6	10	6
	10	10	10	10	10	9	10	8	10	7	10	7
	10	10	10	10	10	7	10	7	10	6	10	6
	120 hr.		168 hr.		336 hr.		504 hr.					
Vinyl Toluene-Ether Ester Vehicle #4	10	10	10	10	10	5	9	5				
	10	10	10	10	10	6	10	6				
	10	9	10	9	10	5	10	5				
	10	10	10	10	10	6	10	6				
Standard Alkyd Nitrocellulose Vehicle-Control	9	9	9	9	8	8	8	7				
	9	9	9	9	9	8	8	7				
	9	10	9	9	8	8	7	8				
	10	7	9	6	8	5	7	5				

Table 116

Vinyl Toluene-Ether Ester Vehicles
Drier Study #1

Driers in Primers	Driers in Topcoats	Solvent Resistance 0 hr. Recovery	Solvent Resistance 24 hr. Recovery
0.4% Pb 0.05% Co (Naphthenates)*	0.1% Co 0.05% Mn (Naphthenates)*	1	2
0.4% Pb 0.05% Co (Naphthenates)* 1 hr. @ 180° F	0.1% Co 0.05% Mn (Naphthenates)* 1 hr. @ 180° F	2	3
0.4% Pb 0.05% Co. (Naphthenates)* 4 hr. @ 180° F	0.1% Co 0.05% Mn (Naphthenates)* 4 hr. @ 180° F	5	6
0.4% Pb 0.05% Co (Acetates)	0.1% Co 0.05% Mn (Acetates)	1	2
0.4% Pb 0.05% Co (Naphthenates)**	0.1% Co 0.05% Mn (Naphthenates)**	7	8
0.4% Zn 0.05% Co** (Naphthenates)	0.1% Co 0.05% Mn (Naphthenates)**	6	7
0.4% Ca 0.05% Co** (Naphthenates)	0.1% Co 0.05% Mn (Naphthenates)**	6	7
0.4% Ca 0.05% Co** (Naphthenates)	0.1% Ca 0.1% Co** (Naphthenates)	5	6
0.4% Pb 0.05% Co (Octoates)	0.1% Co 0.05% Mn (Octoates)	4	6
0.4% Ca 0.05% Co (Octoates)	0.1% Co 0.05% Mn (Octoates)	4	6

(cont.)

Driers in Primers	Driers in Topcoats	Solvent Resistance 0 hr. Recovery	 24 hr. Recovery
0.4% Ca 0.05% Co (Octoates)	0.1% Ca 0.1% Co (Octoates)	4	5
0.4% Pb 0.05% Co (Naphthenates)*	0.1% Co 0.05% Mn (Naphthenates)**	1	2
0.4% Pb 0.05% Co (Naphthenates)* 1 hr. @ 250° F	0.1% Co 0.05% Mn (Naphthenates)* 1 hr. @ 250° F	9	9
0.4% Pb 0.05% Co (Naphthenates)* 4 hr. @ 250° F	0.1% Co 0.05% Mn (Naphthenates)* 4 hr. @ 250° F	9	9

*Purchased driers
**Sherwin-Williams driers

Table 117

Vinyl Toluene-Ether Ester Vehicles
Drier Study #2

Primer Vehicle Number	Naphthenate Driers in Primers	Topcoat Vehicle Number	Naphthenate Driers in Topcoats	Solvent Resistance Recovery 0 hr.	 24 hr.
1	0.4% Pb* 0.05% Co*	--	--	1	3
2	0.4% Pb* 0.05% Co*	--	--	1	3
4	0.4% Pb* 0.05% Co*	--	--	8	8
4	0.4% Pb** 0.05% Co**	--	--	8	8
4	0.4% Zn 0.05% Co**	--	--	8	8
4	0.4% Ca 0.05% Co**	--	--	8	8
--	--	2	0.1% Co* 0.05% Mn*	6	8

(cont.)

Primer Vehicle Number	Naphthenate Driers in Primers	Topcoat Vehicle Number	Naphthenate Driers in Topcoats	Solvent Resistance Recovery 0 hr.	24 hr.
--	--	4	0.1% Co* 0.05% Mn*	1	2
--	--	4	0.1% Co** 0.05% Mn**	1	2
--	--	4	0.1% Ca 0.1% Co**	5	6
1	0.4% Pb* 0.05% Co*	5	0.1% Co** 0.05% Mn**	1	2
2	0.4% Pb* 0.05% Co*	2	0.1% Co* 0.05% Mn*	3	5
2	0.4% Pb* 0.05% Co*	5	0.1% Co** 0.05% Mn**	1	2
4	0.4% Pb* 0.05% Co*	4	0.1% Co* 0.05% Mn*	1	2
4	0.4% Pb** 0.05% Co**	4	0.1% Co** 0.05% Mn**	1	2
4	0.4% Zn 0.05% Co**	4	0.1% Co** 0.05% Mn**	1	2
4	0.4% Ca 0.05% Co**	4	0.1% Co** 0.05% Mn**	3	6
4	0.4% Ca 0.05% Co**	4	0.1% Ca 0.1% Co**	5	7
5	0.4% Pb** 0.05% Co**	2	0.1% Co* 0.05% Mn*	5	7
5	0.4% Pb** 0.05% Co**	5	0.1% Co** 0.05% Mn**	3	5
MIL-P-6889a, Type I, Proprietary	0.3% Pb 0.03% Co	--	--	9	9

(Cont.)

Primer Vehicle Number	Naphthenate Driers in Primers	Topcoat Vehicle Number	Naphthenate Driers in Topcoats	Solvent Resistance Recovery 0 hr.	Solvent Resistance Recovery 24 hr.
--	--	MIL-L-7178	--	9	9
5	0.4% Pb** 0.05% Co**	--	--	9	9
--	--	5	0.1% Co** 0.05% Mn**	9	9

*Purchased driers
**Sherwin-Williams driers

Vehicle #1 -- Initial laboratory batch of 60:40:40:4 Epon 1004: soya fatty acids:vinyl toluene:divinyl benzene

Vehicle #2 -- Repeat laboratory cook of vehicle #1

Vehicle #4 -- Repeat laboratory cook of vehicle #1 using plant raw materials

Vehicle #5 -- Repeat laboratory cook of vehicle #1 using laboratory raw materials.

Table 118

Vinyl Toluene-Ether Ester Vehicles
Drier Study #3

Driers in Primer (Pilot Plant Vehicle)	Driers in Topcoat (Pilot Plant Vehicle)	Solvent Resistance Recovery 0 hr.	Solvent Resistance Recovery 24 hr.
0.4% Pb 0.05% Co	0.1% Co 0.05% Mn	1	2
0.4% Pb 0.05% Co 0.05% Mn	0.1% Co 0.05% Mn	1	2
0.4% Ca 0.1% Co	0.1% Co 0.05% Mn 0.4% Ca	1	2
0.4% Ca 0.05% Co 0.05% Mn	0.1% Co 0.05% Mn 0.4% Ca	1	2

(Cont.)

Driers in Primer (Pilot Plant Vehicle)	Driers in Topcoat (Pilot Plant Vehicle)	Solvent Resistance Recovery	
		0 hr.	24 hr.
0.3% Pb 0.1% Co 0.2% Ca	0.1% Co 0.05% Mn 0.4% Ca	2	3
0.4% Pb 0.05% Co 0.1% Fe	0.1% Co 0.05% Mn 0.1% Fe	2	3
0.4% Pb 0.05% Co 0.1% Fe 0.2% Ca	0.1% Co 0.05% Mn 0.1% Fe 0.2% Ca	1	2
0.4% Ca 0.05% Co 0.1% Fe	0.4% Ca 0.05% Mn 0.1% Fe	3	5
0.5% Fe 0.05% Co 0.05% Mn	0.5% Fe 0.05% Co 0.05% Mn	3	4
0.5% Ca 0.05% Co 0.05% Mn	0.5% Ca 0.05% Co 0.05% Mn	2	3
0.4% Pb 0.05% Co 0.05% Zr	0.1% Co 0.05% Mn 0.05% Zr	1	3
0.4% Pb 0.05% Co 0.05% Rare Earths	0.1% Co 0.05% Mn 0.05% Rare Earths	1	2
0.4% Pb 0.05% Co 0.1% Fe 0.1% Sn	0.1% Co 0.05% Mn 0.1% Fe 0.1% Sn	1	2
0.4% Pb 0.1% Co	0.1% Co 0.05% Mn	1	2
0.4% Pb 0.05% Co*	0.1% Co 0.05% Mn	1	2
0.4% Pb 0.05% Co 0.05% Mn*	0.1% Co 0.05% Mn	1	2
0.4% Ca 0.1% Co*	0.1% Co 0.05% Mn 0.4% Ca	1	2

(Cont.)

Driers in Primer (Pilot Plant Vehicle)	Driers in Topcoat (Pilot Plant Vehicle)	Solvent Resistance Recovery 0 hr.	24 hr.
0.3% Pb 0.1% Co 0.2% Ca*	0.1% Co 0.05% Mn 0.4% Ca	2	3
0.4% Pb 0.05% Co 0.1% Fe*	0.1% Co 0.05% Mn 0.1% Fe	2	3
0.4% Pb 0.05% Co* 0.1% Fe 0.2% Ca	0.1% Co 0.05% Mn 0.1% Fe 0.2% Ca	1	2
0.4% Pb 0.05% Co 0.05% Zr*	0.1% Co 0.05% Mn 0.05% Zr	1	2
0.4% Pb 0.05% Co 0.1% Sn*	0.1% Co 0.05% Mn 0.1% Fe 0.1% Sn	1	2

*Ball mill dispersions

Table 119

Polyamine Curing of Epoxide Resins

Resins	Polyamine	Conc. PHR	Viscosity (#4 Ford-Sec.) 1 hr.	6 hr.	72 hr.	Adhesion 72 hr.	Pencil Hardness 72 hr.
Epon 828	Ethylene Diamine	6	12	16	Gel	--	--
"	Diethylene Triamine	6	12	13	Gel	--	<7B
Epon 834	Ethylene Diamine	6	16	35	Gel	9	F
"	Diethylene Triamine	6	16	49	Gel	5	2H
Epon 1001	Ethylene Diamine	6	18	35	Gel	2	F
"	Diethylene Triamine	6	19	32	Gel	3	H
"	Ethylene Diamine	12	17	35	Gel	7	2H
Experi-mental	Ethylene Diamine	6	17	25	Gel	5	F
	Diethylene Triamine	6	17	28	Gel	4	2H

Table 120

Epon 828 - Epon 1001 Blends With Polyamines

Resin	Viscosity - Sec. #4 Ford @ 75° F Initial	5 (Hours)	24 (Hours)	48 (Hours)	Adhesion 7 Days	Foil Drying 1/2 (Hours)	1 (Hours)	4 (Hours)	6 (Hours)	24 (Hours)	48 (Hours)	5 (Days)	14 (Days)
100 Epon 828	12	17	Gel	--	--	0	0	0	0	0	0	0	0
90 Epon 828/ 10 Epon 1001	13	26	Gel	--	--	0	0	3	3	5	5	5	5
75 Epon 828/ 25 Epon 1001	14	45	Gel	--	9	0	1	5	8	10	--	--	--
50 Epon 828/ 50 Epon 1001	18	112	Gel	--	9	0	1	6	10	--	--	--	--
25 Epon 828/ 75 Epon 1001	18	82	Gel	--	5	3	3	6	10	--	--	--	--
100 Epon 1001	22	65	171	Gel	5	3	3	6	10	--	--	--	--

Table 121

Various Amine:Titanate Ratios With Epon 1001

Amine to Titanate Ratio	Total Catalyst PHR	Foil Drying 1/2 hr.	Foil Drying 1 hr.	Foil Drying 2 hr.	Foil Drying 3 hr.	Pencil Hardness 48 hr.	Recoat Adhesion 24 hr.	Moisture Resistance 48 hr. Whiten	Moisture Resistance 48 hr. Adhe-sion	Moisture Resistance 48 hr. Tough-ness	Viscosity - Sec. #4 Ford @ 75° F Initial	4 hr.	24 hr.	72 hr.	8 day	14 day
60/40	7	0	5	6	10	2H	8	9	1	7	18	19	20	38	Gel	--
23/77	5	0	5	6	10	H	2	2	4	1	18	19	19	19	19	20
23/77	7	0	5	6	10	2H	3	9	3	2	18	19	19	24	47	Gel
50/50	7	0	3	6	10	2H	4	10	1	2	18	19	19	24	55	Gel
67/33	7	0	3	5	10	2H	7	9	2	5	18	19	19	30	Gel	--
33/67	7	0	5	6	10	2H	3	7	4	3	18	19	19	21	29	58
100 Amine	7	1	3	4	10	2H	8	10	1	8	18	20	24	69	Gel	--
100 Titanate	7	1	5	6	10	H	2	6	5	4	18	19	19	19	19	19

Table 122

Effect of Solvent Blends on Package Stability of Amine-Titanate Treated Epon 1001 Solutions

Solvents (Per Cent of Solvent Mixture)					
MIBK	Toluene	Alcohol	Diacetone Alcohol	Butyl Cellosolve	Stability
50	50	--	--	--	Gel particles immediately.
44.7	44.6	10.7	--	--	Gel particles immediately. All gel in two weeks.
40	40	20	--	--	Gel particles immediately.
35	35	30	--	--	Gel particles immediately. Soft gel in two weeks.
44.7	44.6	--	10.7	--	Gel particles immediately.
40	40	--	20	--	Gel particles immediately.
35	35	--	30	--	Gel particles immediately.
--	--	--	50	50	No gel.
45	45	--	--	10	Gel particles immediately.
40	40	--	--	20	Gel particles immediately, but less than above.
35	35	--	--	30	Gel particles immediately, but less than last two.
--	50	--	--	50	No gel.
--	33-1/3	--	33-1/3	33-1/3	No gel.
25	25	--	25	25	No gel.

Table 123

Effect of Solvent Blends on Adhesion of Amine-Titanate Treated Epon 1001 Solutions

Solvent Composition				Film Thickness (mils)		Adhesive Characteristics	
MIBK	Toluene	Diacetone Alcohol	Butyl Cellosolve	Panel #1	Panel #2	Undercoat Alone	With Topcoat
40	40	--	20	1	1	7	2
35	35	--	30	1	1	8	3
--	50	--	50	0.7-0.9	0.5-0.7	9	4
--	--	50	50	0.3-0.5	0.4-0.6	4	4
--	33-1/3	33-1/3	33-1/3	0.8	0.8	4	3
25	25	25	25	1	0.9	6	2

Table 124

Ether Modified Alkyd Vehicles, #1

	Film Thick.		3% Salt Spray Performance									
			72 hr.		168 hr.		264 hr.		336 hr.		504 hr.	
Identification	Primer	Total	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
10% Epon 1004 replacing glycerol in standard alkyd	1.17	1.80	10	8	10	8	9	7	9	7	7	5
			10	7	9	7	9	6	8	6	7	6
			9	8	8	8	7	7	6	7	6	6
10% Epon 1004 and 10% Glycerol added to standard alkyd	1.25	1.96	8	9	6	9	6	8	5	7	5	7
			9	9	7	8	7	8	6	7	6	7
			9	8	7	8	7	7	6	7	6	7
			10	9	8	9	8	8	7	7	7	7
Standard Alkyd	1.25	1.95	10	9	8	8	8	8	7	7	7	7
			10	8	10	7	10	7	9	7	9	7
			10	9	9	7	9	7	9	7	8	7
			10	9	9	7	9	7	8	7	8	7

Table 125

Ether Modified Alkyd Vehicles, #2

		3% Salt Spray Performance											
		72 hr.		168 hr.		336 hr.		504 hr.		672 hr.		840 hr.	
Vehicle	Solvent Resist.	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
10% Epon 1004 in 100% Linseed Oil Alkyd Vehicle	8	10	10	10	3	8	2	7	2	6	2	6	2
	8	10	10	10	5	8	2	7	2	6	2	5	2
		10	10	10	3	9	2	8	2	7	2	6	2
		10	10	10	7	10	2	10	2	9	2	9	2
Standard Alkyd Control	9	9	10	9	10	9	10	8	9	8	9	7	8
	9	9	10	9	10	9	10	9	9	8	9	8	8
		9	10	9	10	8	9	7	8	7	8	6	7
		9	10	9	10	9	10	9	9	8	9	7	7
Styrenated Ether Ester	2	10	10	10	10	10	10	9	10	9	9	8	9
	2	10	10	9	10	9	10	9	9	8	9	8	9
		10	10	10	10	10	10	9	10	9	9	8	9
		10	10	10	10	9	10	9	10	9	9	8	9

Table 126

A Commercial Furan Resin Coating Over Various Pretreatments

	3% Salt Spray Performance											
	24 hr.		240 hr.		576 hr.		1152 hr.		1920 hr.		2800 hr.	
Magnesium Pretreatment	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
None	10	6	9	6	7	5	--	--	--	--	--	--
	10	6	10	6	9	5	6	3	4	2	3	1
	10	6	10	6	9	5	6	3	4	2	3	2
Wash Primer	10	7	9	7	9	6	--	--	--	--	--	--
	10	7	10	7	9	6	9	6	9	5	9	5
	10	7	10	7	9	6	9	6	8	5	8	5
MIL-M-3171 Type III	10	9	9	7	8	7	--	--	--	--	--	--
	10	9	9	7	9	7	9	5	9	4	9	1
	10	9	9	7	9	7	9	5	9	4	8	2

Table 127

A Commercial Furan Resin Coating Over Wash Primed Magnesium

		3% Salt Spray Performance									
		72 hr.		504 hr.		912 hr.		1248 hr.		1488 hr.	
Magnesium Pretreatment	Film Thick. (Mils)	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Wash Primer	1.85	10	8	10	7	9	7	9	6	8	6
		10	8	10	7	10	7	9	6	8	6
		10	8	10	7	10	7	9	5	9	5
		10	8	10	7	9	7	9	6	8	5

Table 128

Corrosion Resistance of Furan Coatings

		3% Salt Spray Performance					
		24 hr.		192 hr.		360 hr.	
System	Total Film Thickness (mils)	Cor.	Blister	Cor.	Blister	Cor.	Blister
MIL-P-6889a, Type I, proprietary - MIL-L-7178, Aluminized	2.00	10	10	9	9	9	9
		10	10	9	9	9	9
		10	10	10	9	10	9
		10	10	10	9	10	9
Vinyl Toluene-Ether Ester Primer and Topcoat	2.30	10	10	10	10	10	9
		10	10	10	6	10	3
		10	10	10	9	10	9
		10	10	10	9	10	9
Jet-Kote-Without Catalyst	2.25	9	10	7	8	--	--
		9	10	7	8	--	--
		9	10	7	8	--	--
		9	10	7	9	--	--
Jet-Kote-With Catalyst	2.25	9	9	9	7	8	5
		9	9	8	7	8	5
		9	9	9	7	7	5
		9	9	8	6	7	5
Durez 14383-With Catalyst	2.30	8	9	5	9	The films cracked within 240 hours of salt spray exposure.	
		9	9	5	9		
		9	9	5	9		
		9	9	5	9		

Table 129

Thiokol LP-3 With Epon 828

System	Total Film Thick. (Mils)	Adhesion	3% Salt Spray Performance									
			24 hr.		192 hr.		528 hr.		696 hr.		864 hr.	
			Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
MIL-P-6889a, Type I, Proprietary-MIL-L-7178, Aluminized	2.00	9	10	10	9	9	8	9	6	8	5	8
			10	10	9	9	8	8	7	8	6	6
			10	10	10	9	10	8	9	7	7	7
			10	10	10	9	9	9	8	8	7	7
Vinyl Toluene Ether Ester Primer and Topcoat	2.30	8	10	10	10	10	10	9	9	9	9	8
			10	10	10	6	10	2	9	2	8	2
			10	10	10	9	10	3	9	2	9	2
			10	10	10	9	9	7	9	5	8	4
			72 hr.		240 hr.		568 hr.		736 hr.		904 hr.	
Thiokol-Ether Resin Catalyzed With an Amine	2.30	8	10	10	10	10	10	10	9	10	9	10
			10	10	10	10	10	10	9	9	9	9
			10	10	10	10	10	10	9	9	9	9
			10	10	10	10	10	10	9	10	9	10

Table 130

Various Chromate Pigments in
Styrenated Ether Ester Primers

	3% Salt Spray Performance									
	264 hr.		432 hr.		560 hr.		1064 hr.		1600 hr.	
Identification	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Strontium Chromate-Silicon Dioxide, Styrenated Ether Ester	10	9	9	8	8	5	5	4	4	2
	9	10	8	9	7	8	2	6	1	4
	10	10	9	9	8	8	3	7	2	4
	9	10	9	9	8	7	4	6	3	4
Barium-Potassium Chromate-Silicon Dioxide, Styrenated Ether Ester	9	10	9	10	8	9	5	8	3	5
	9	10	9	10	8	9	7	9	6	5
	10	10	9	10	8	9	5	8	2	6
	10	10	8	10	7	9	3	7	1	6
Calcium Chromate-Silicon Dioxide, Styrenated Ether Ester	10	10	10	10	10	10	9	9	9	8
	10	10	10	10	10	10	8	7	7	7
	10	10	10	10	10	10	8	8	8	8
	10	10	10	10	9	9	9	9	9	8
Zinc Chromate-Silicon Dioxide, Styrenated Ether Ester	10	10	10	10	10	10	9	9	8	9
	10	10	10	10	10	10	10	9	9	9
	10	10	10	10	9	10	8	9	7	9
	10	10	9	10	9	10	7	9	7	9
Zinc Chromate-Magnesium Silicate, Styrenated Ether Ester	10	10	10	10	9	9	8	9	6	8
	10	10	10	10	10	10	9	9	8	9
	10	10	10	10	9	10	9	9	8	8
	10	10	10	10	8	10	8	9	8	8
Zinc Chromate-Silicon Dioxide Alkyd	10	9	9	9	9	9	7	8	4	8
	10	9	8	9	7	9	5	8	4	7
	10	9	9	8	8	8	7	7	2	7
	10	9	6	9	5	9	4	7	3	7
MIL-P-6889a, Type I	6	5	6	5	5	5	2	2	2	2
	5	5	6	5	5	3	1	2	1	2
	7	6	9	6	9	5	4	3	2	3
	7	6	6	5	5	3	1	2	1	2

Table 131

Effectiveness of Inhibitive Pigments in Vinyl Copolymer Vehicles

Pretreatment	Primer	Film Thick. (Mils)	Adhesion	3% Salt Spray Performance							
				72 hr.		168 hr.		432 hr.		840 hr.	
				Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
MIL-M-3171 Type III	Proprietary Iron Oxide Vinyl Copolymer	2.1	7	10	9	10	9	9	8	9	8
				10	9	10	9	9	9	8	7
				10	10	10	10	9	9	9	7
				10	10	9	10	9	8	9	8
MIL-M-3171 Type III	Iron Oxide Vinyl Copolymer	2.0	6	10	3	10	3	8	2	8	2
				10	5	9	5	9	3	6	2
				10	2	9	2	9	2	8	2
				10	5	9	5	8	4	6	2
MIL-M-3171 Type III	Strontium Chromate-Vinyl Copolymer	2.0	8	10	9	9	8	8	7	7	3
				9	7	7	6	7	4	5	2
				10	10	10	9	8	7	5	3
				10	9	10	6	4	4	8	2
MIL-M-3171 Type III	Strontium Chromate-Modified Vinyl Copolymer	2.1	6	10	2	9	2	8	2	5	1
				10	3	9	3	8	2	5	1
				10	2	10	2	8	2	3	1
				10	2	9	2	8	2	6	1

Note: All panels were topcoated with an aluminized vinyl copolymer topcoat.

Table 132

Evaluation of Ammonium Vanadate and Calcium Sulfide
in a Styrenated Ether Ester Vehicle

				3% Salt Spray Performance							
				24 hr.		168 hr.		452 hr.		956 hr.	
Pretreatment	Primer	Film Thick. (Mils)	Adhesion	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
MIL-M-3171 Type III	Styrenated Ether Ester	1.9	8	10	10	10	10	9	10	6	10
				10	10	10	10	9	10	7	10
				10	10	10	10	8	10	8	10
				10	10	10	10	9	10	--	--
MIL-M-3171 Type III	Styrenated Ether Ester 5% $NH_4 VO_3$ + CaS	2.0	8	10	10	10	9	8	9	6	9
				10	10	9	9	9	9	8	9
				10	10	9	9	8	9	7	9
				10	10	10	10	8	10	6	10
MIL-M-3171 Type III	Styrenated Ether Ester 100% $NH_4 VO_3$ + CaS	2.1	8	10	10	8	2	7	1	2	1
				10	10	8	2	6	1	2	1
				10	10	8	2	6	1	--	--
				10	10	8	2	7	1	1	1

Note: All panels were topcoated with an aluminized styrenated ether ester finish.

Table 133

Calcium Sulfide-Ammonium Vanadate Screening Tests

Pigments	3% NaCl Water			Distilled Water		
	MIL-M-3171 Type III	Bare Mg FS1H	MIL-M-3171 Type III Coupled to 24S Al	MIL-M-3171 Type III	Bare Mg FS1H	MIL-M-3171 Type III Coupled to 24S Al
Ammonium Vanadate Plus Calcium Sulfide	10	10	10	10	10	10
Strontium Chromate Plus Calcium Sulfide	10	10	2	10	10	7
Calcium Sulfide	10	9	8	10	9	9
Ammonium Vanadate	8	6	0	8	7	9
Zirconium Fluoride	5	5	0	6	6	5
Potassium Zirconium Fluoride	6	3	0	9	9	8
Zinc Chromate	8	8	0	10	10	10
Strontium Chromate	8	7	0	10	10	10
Silicon Dioxide	7	5	0	9	9	9
Magnesium Silicate	7	6	0	10	10	10
No Pigment - Control	7	6	0	10	10	10

Table 134

Calcium Sulfide-Ammonium Vanadate Primers at PVC's Above the CPVC

		3% Salt Spray Performance							
		44 hr.		72 hr.		240 hr.		408 hr.	
Coating System	PVC	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Sandwich Coat Between Styrenated Ether Ester Primer and Aluminized Topcoat	80%	9	3	9	2	8	1	8	1
		9	3	9	2	8	1	8	1
		9	3	9	2	8	1	8	1
		9	3	9	2	8	1	8	1
Sandwich Coat Between Styrenated Ether Ester Primer and Aluminized Topcoat	85%	9	9	9	7	8	5	8	5
		9	9	9	7	8	5	8	5
		9	9	9	6	8	5	8	4
		9	6	9	4	8	4	8	4
As Primer Coating With Styrenated Ether Ester Aluminized Topcoat	80%	9	5	9	4	8	4	8	4
		9	6	9	3	8	3	8	3
		9	6	9	4	8	4	8	4
		9	5	9	4	8	4	8	4
As Primer Coating With Styrenated Ether Ester Aluminized Topcoat	85%	9	8	8	6	8	5	8	5
		9	9	9	6	8	5	8	5
		9	9	9	6	8	5	8	5
		9	9	9	6	8	5	8	5
Styrenated Ether Ester Primer and Aluminized Topcoat - Control	35%	10	10	10	10	10	10	9	9
		10	10	10	10	10	10	10	9
		10	10	10	10	9	10	9	9
		10	10	10	10	9	10	9	9
Standard Alkyd - Control	35%	10	9	10	9	9	9	9	9
		10	9	10	8	9	8	9	8
		10	9	10	8	10	8	9	8
		10	9	10	8	8	8	7	8

Table 135

Calcium Sulfide-Ammonium Vanadate
80% PVC Primer as the Entire Protective Coating System

	3% Salt Spray Performance				
Panel Coating	72 hr. Corrosion	168 hr. Corrosion	336 hr. Corrosion	504 hr. Corrosion	575 hr. Corrosion
Calcium Sulfide-	9	8	8	7	7
Ammonium Vanadate	9	8	7	7	7
Pigmented Coating	9	8	7	7	7
	9	8	8	7	7
Unpainted Panels	7	5	1	--	--
	7	6	3	--	--
	7	5	2	--	--
	7	5	3	--	--

Table 136

Evaluation of Pickle Film Formed From Calcium Sulfide-Ammonium Vanadate Solutions

Magnesium Type	Film Formed by	3% Salt Spray Performance 72 hr. Cor.	72 hr. Blister	168 hr. Cor.	168 hr. Blister	336 hr. Cor.	336 hr. Blister	504 hr. Cor.	504 hr. Blister
Alkali Cleaned	Immersion in Distilled Water Solution	10	3	9	3	8	3	7	2
		10	3	9	3	9	3	9	2
		10	4	9	4	8	4	7	3
		10	4	9	4	9	4	8	3
MIL-M-3171 Type III	Immersion in Distilled Water Solution	10	5	10	5	9	5	9	5
		10	4	9	4	9	4	8	4
		10	4	10	4	9	4	8	4
		10	4	10	4	9	4	9	4
Alkali Cleaned	Immersion in 3% NaCl Solution	8	3	6	3	5	3	4	3
		9	2	8	2	6	2	5	2
		10	2	9	2	8	2	8	2
		10	2	7	1	5	1	5	1
MIL-M-3171 Type III	Immersion in 3% NaCl Solution	10	4	8	4	8	4	7	4
		9	4	9	4	8	4	8	4
		9	3	9	3	9	3	8	3
		10	3	10	3	9	3	8	3
Alkali Cleaned	Magnesium as Anode-Aluminum as Cathode	9	3	9	3	9	3	8	3
		10	3	10	3	9	3	8	3
		8	3	7	3	7	3	7	3
		10	3	10	3	9	3	9	3
MIL-M-3171 Type III	Magnesium as Anode-Aluminum as Cathode	10	2	10	2	9	2	9	2
		10	2	10	2	9	2	9	2
		10	3	10	2	9	2	8	2
		10	3	10	2	9	2	9	2
Alkali Cleaned	No Pretreatment Control	9	3	9	3	8	3	8	3
		9	3	9	3	8	3	7	3
		10	4	9	4	8	3	8	3
		9	3	8	3	8	3	7	3
MIL-M-3171 Type III	No Pretreatment Control	10	9	8	9	8	8	8	8
		10	5	9	3	8	3	8	3
		9	8	8	7	8	5	8	5
		9	9	8	8	7	8	7	7

Table 137

Evaluation of Calcium Sulfide-Ammonium Vanadate Pickle Film Formed by Electrical Means

		3% Salt Spray Performance							
		72 hr.		168 hr.		336 hr.		504 hr.	
Applied Voltage	Time	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
0.8	1 min.	9	7	8	4	8	4	7	4
		10	8	10	5	10	5	9	4
		10	3	10	2	9	2	9	2
		10	4	10	3	9	3	8	3
0.65	5 min.	10	9	10	7	10	7	9	7
		9	9	8	7	8	7	8	7
		9	9	8	8	8	7	7	7
		9	5	9	4	8	3	7	3
0.7	30 min.	9	9	8	7	8	6	7	6
		10	9	9	9	8	9	8	8
		10	9	10	8	9	8	8	8
		10	10	10	9	9	9	8	9
0.1	30 min.	9	10	8	5	8	5	8	5
		10	9	10	4	10	4	10	4
		9	10	8	4	8	4	8	4
		10	9	10	6	10	6	10	5
1.5	5 min.	9	9	8	7	7	7	7	7
		10	9	9	7	8	7	8	7
		10	9	9	8	8	7	8	7
		10	9	9	8	8	8	8	8
1.5*	30 min.	10	4	9	4	7	4	7	4
		9	4	8	4	7	4	7	4
		10	4	9	4	7	4	7	4
		10	4	9	4	7	4	7	4
Control No Pickle Film Formed		10	9	10	7	8	7	8	7
		10	9	9	7	9	7	8	7
		10	9	10	6	10	6	9	6
		10	9	9	7	8	7	8	7

*This set contained alkaline degreased magnesium panels. All others were pretreated according to MIL-M-3171, Type III.

Table 138

Ratio of Calcium Sulfide to Ammonium Vanadate

Calcium Sulfide	Ammonium Vanadate	Length of Test	Couple Performance		
			Magnesium-Aluminum Couple	Individual Panel in Couple	
				Magnesium	Aluminum
0	100	6 Days	2	0	3
10	90	6 Days	3	2	4
25	75	27 Days	5	5	6
43	57	50 Days	6	6	7
50	50	50 Days	9	8	9
75	25	50 Days	9	9	9
90	10	27 Days	8	8	9
100	0	6 Days	4	4	5

Table 139

Screening Tests on Calcium Hydroxide, Sodium Sulfide and Ammonium Sulfide (Six Days Immersion in 3% Salt Solution)

Inhibitor	Magnesium-Aluminum Couple	Individual Panel in Couple	
		Magnesium	Aluminum
No Pigment	2	1	3
Calcium Hydroxide	2	0	4
Sodium Sulfide	2	10	0
Ammonium Sulfide	5	5	9
Calcium Sulfide	7	7	9
Calcium Sulfide + Ammonium Vanadate	10	10	10

Table 140

Screening Tests on Individual Ions and Their Combinations
(Six Days Immersion in 3% Salt Solution)

Inhibitor	Ions Present	Magnesium-Aluminum Couple	Individual Panel in Couple	
			Magnesium	Aluminum
Calcium Hydroxide	Ca^{++}	2	1	4
Hydrogen Sulfide	S^{--}	7	7	9
Ammonium Hydroxide	$(NH_4)^+$	1	1	1
Vanadium Pentoxide	$(VO_3)^-$	2	0	3
Calcium Hydroxide + Ammonium Hydroxide	Ca^{++}, $(NH_4)^+$	1	0	2
Hydrogen Sulfide + Vanadium Pentoxide	S^{--}, $(VO_3)^-$	8	8	8
Ammonium Sulfide	$(NH_4)^+$, S^{--}	9	9	9
Calcium Sulfide	Ca^{++}, S^{--}	7	7	9
Calcium Vanadate	Ca^{++}, $(VO_3)^-$	2	0	2
Ammonium Vanadate	$(NH_4)^+$, $(VO_3)^-$	2	1	4
Calcium Sulfide + Ammonium Hydroxide	Ca^{++}, S^{--}, $(NH_4)^+$	9	9	9
Calcium Sulfide + Vanadium Pentoxide	Ca^{++}, S^{--}, $(VO_3)^-$	10	9	10
Calcium Hydroxide + Ammonium Vanadate	Ca^{++}, $(NH_4)^+$ $(VO_3)^-$	6	7	7
Ammonium Sulfide + Ammonium Vanadate	$(NH_4)^+$, S^{--}, $(VO_3)^-$	9	9	10
Calcium Sulfide + Ammonium Vanadate	Ca^{++}, S^{--}, $(NH_4)^+$, $(VO_3)^-$	10	10	10
None	None	2	1	3

Table 141

Ammonium Sulfide Exhaustion in Screening Tests
(Six Days Immersion in 3% Salt Solution)

Inhibitor	Explanatory Remarks	Magnesium-Aluminum Couple	Individual Panel in Couple Magnesium	Individual Panel in Couple Aluminum
Ammonium Sulfide	Ammonium Hydroxide Added 72 hr.	7	6	8
Ammonium Sulfide	Hydrogen Sulfide Added 72 hr.	9	9	10
Ammonium Sulfide	Ammonium Sulfide Added 72 hr.	8	7	9
Ammonium Sulfide	Couple Removed 72 hr.	10	9	10
Ammonium Sulfide	Couple Removed 96 hr.	9	9	10
Ammonium Sulfide	Couple Removed 144 hr.	9	9	10
Ammonium Hydroxide		2	1	2
Hydrogen Sulfide		7	7	7
None		2	1	3

Table 142

Various Sulfides in Screening Tests
(Six Days Immersion in 3% Salt Solution)

Inhibitor	Corrosion Performance		
	Magnesium-Aluminum Couple	Individual Panel in Couple	
		Magnesium	Aluminum
Zinc Sulfide	1	1	1
Zinc Sulfide Plus Ammonium Vanadate	2	0	3
Antimony Sulfide	2	0	3
Antimony Sulfide Plus Ammonium Vanadate	2	0	3
Cadmium Sulfide	1	0	1
Cadmium Sulfide Plus Ammonium Vanadate	2	0	3
Thiourea Plus Ammonium Vanadate	8	7	8
Calcium Sulfide Plus Ammonium Vanadate	10	10	10
None	2	1	3

Table 143

Screening Tests on the Reaction Product of Calcium Sulfide and Ammonium Vanadate
(Six Days Immersion in 3% Salt Solution)

Inhibitor	Corrosion Performance		
	Magnesium-Aluminum Couple	Individual Panel in Couple	
		Magnesium	Aluminum
Calcium Sulfide Plus Ammonium Vanadate Control	10	10	10
Light Brown Precipitate	9	9	9
Dark Brown Precipitate	1	0	1
Filtrate	1	0	1
Precipitate Dried and Washed	6	6	7
Precipitate Dried, Unwashed	8	8	8

Table 144

Screening Tests on Pigments Prepared From
Calcium Sulfide and Ammonium Vanadate, #1
(Eight Days Immersion in 3% Salt Solution)

Inhibitor	3% Salt Solution Slurry Performance					
	24 hr.		48 hr.		192 hr.	
	Magnesium	Aluminum	Magnesium	Aluminum	Magnesium	Aluminum
None	0	8	0	6	0	2
Zinc Chromate	7	9	0	8	0	3
$CaS + NH_4 VO_3$ 1-1 ratio, unaged	10	10	10	10	8	9
$CaS + NH_4 VO_3$ 1-1 ratio, aged	10	10	10	10	7	9
$CaS + NH_4 VO_3$ 1.5-1 ratio, unaged	10	10	10	10	8	9
$CaS + NH_4 VO_3$ 1.5-1 ratio, aged	10	10	10	10	5	8
$CaS + NH_4 VO_3$ Fresh Mixture	10	10	10	10	10	10

Table 145

Screening Test of a Pigment Prepared From
Calcium Sulfide and Ammonium Vanadate, #2
(Seven Days Immersion in 3% Salt Solution)

Inhibitor	Magnesium-Aluminum Couple	Individual Panel in Couple	
		Magnesium	Aluminum
Zinc Chromate	2	0	2
$CaS-NH_4 VO_3$ Prepared Pigment	9	9	9
$CaS + NH_4 VO_3$, Fresh Mixture	10	10	10

Table 146

Evaluation of a Calcium-Vanadium Pigmented Primer, #1

		3% Salt Spray Performance									
		48 hr.		144 hr.		240 hr.		408 hr.		576 hr.	
Vehicle in System	Primer Pigment	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Vinyl Toluene-Ether Ester	Reacted Calcium Sulfide-Ammonium Vanadate	10	10	10	9	10	8	9	7	9	7
		10	10	10	7	9	6	9	6	9	6
		10	10	10	10	10	7	10	5	9	5
		10	10	10	10	9	9	9	7	8	7
Styrenated Ether Ester	Unreacted Calcium Sulfide-Ammonium Vanadate	10	10	10	8	10	2	10	2	9	2
		10	10	10	8	10	2	9	2	8	2
		10	10	10	8	10	2	9	2	9	2
		10	10	10	9	10	2	9	2	8	2
Vinyl Toluene-Ether Ester	Zinc Chromate	10	10	9	7	9	6	9	4	8	3
		10	10	10	7	10	6	9	4	9	4
		10	7	10	7	9	7	9	6	8	6
		10	10	10	9	10	8	10	5	9	5

Table 147

Evaluation of a Calcium-Vanadium Pigmented Primer, #2

	3% Salt Spray Performance											
	96 hr.		264 hr.		432 hr.		600 hr.		768 hr.		936 hr.	
Primer Pigment	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
Reacted Calcium Sulfide-Ammonium Vanadate	10	10	9	9	9	8	9	8	8	7	7	7
	10	10	10	8	10	7	9	7	8	6	7	6
	10	10	10	8	10	8	10	7	9	6	8	6
	10	10	10	7	9	6	9	6	8	5	7	5
Unreacted Calcium Sulfide-Ammonium Vanadate	10	10	10	10	9	8	9	7	8	7	7	7
	10	10	9	10	9	8	9	6	8	5	7	5
	10	10	10	10	10	7	9	4	9	4	8	4
	10	10	10	10	9	5	9	4	8	4	7	4
Zinc Chromate	10	9	10	9	10	4	10	2	10	2	9	2
	10	10	9	10	9	6	9	2	8	2	8	2
	10	10	10	10	10	6	10	2	9	2	8	2
	10	10	10	10	10	5	10	2	9	2	8	2

Table 148

Evaluation of Insulative Vinyl Tape on Magnesium-Aluminum Faying Surfaces

		3% Salt Spray Performance									
		96 hr.		264 hr.		504 hr.		840 hr.		1104 hr.	
Type of Coatings	System	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
MIL-P-6889a,	1 coat Al	10	10	10	8	9	2	9	2	7	2
Type I, Plus	2 coats Mg	10	9	10	2	9	2	9	2	7	2
MIL-L-7178,	No tape	10	10	10	6	9	2	9	2	7	2
Aluminized											
MIL-P-6889a,	1 coat Al	10	7	9	7	9	7	8	2	6	2
Type I, Plus	2 coats Mg	10	7	10	7	10	2	9	2	7	2
MIL-L-7178,	Plus Tape	10	8	10	7	9	7	9	2	7	2
Aluminized											
MIL-P-6889a,	3 coats Al	10	8	10	6	9	4	9	2	8	2
Type I, Plus	4 coats Mg	10	8	10	7	9	4	8	2	7	2
MIL-L-7178,	No Tape	10	8	9	7	9	4	7	2	7	2
Aluminized											
MIL-P-6889a,	3 coats Al	10	7	10	3	9	3	8	2	8	2
Type I, Plus	4 coats Mg	10	7	10	6	10	5	9	2	9	2
MIL-L-7178,	Plus Tape	10	7	9	6	9	3	9	2	9	2
Aluminized											
As Above With	3 coats Al	10	10	10	9	9	3	8	2	7	2
Soya Ether	4 coats Mg	10	10	10	9	9	4	8	2	7	2
Ester as	No Tape	10	10	9	9	9	2	8	2	7	2
Vehicle											

Table 149

Red Oxide Primer, MIL-P-11414
Physical Characteristics

Identification	Substrate	Topcoat	Film Thickness (Mils)	Adhesion
MIL-P-11414	Dichromate Treated Magnesium	MIL-L-7178	2.5	2
MIL-P-6889a	Dichromate Treated Magnesium	MIL-L-7178	2.5	3-8

Table 150

Red Oxide Primer, MIL-P-11414
Salt Spray Performance

	3% Salt Spray Performance							
	24 hr.		168 hr.		432 hr.		832 hr.	
Identification	Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
MIL-P-11414	10	10	10	9	9	7	6	1
	10	10	10	9	9	8	6	5
	10	10	9	9	9	7	5	3
	10	10	10	9	9	7	6	3
	10	10	10	9	8	7	6	1
MIL-P-6889a	10	10	9	9	9	8	7	1
	10	10	9	9	9	8	3	1
	10	10	9	9	9	8	7	1
	10	10	9	9	9	7	7	1
	10	10	9	9	9	8	4	2

Table 151

Evaluation of Various Coatings at Reduced Coating Weights

Pretreatment	Finishing System	Total Film Thick.	Adhesion	3% Salt Spray Performance							
				24 hr.		264 hr.		432 hr.		600 hr.	
				Cor.	Blister	Cor.	Blister	Cor.	Blister	Cor.	Blister
MIL-M-3171, Type III	Specification	2.1	8	10	6	6	6	3	6	1	6
MIL-M-3171, Type III	VMCH Resin	1.5	3	10	10	9	8	8	6	6	6
Bare Magnesium	VMCH Resin	1.5	5	10	10	9	6	7	5	5	4
MIL-M-3171, Type III	VMCH/VAGH 50/50 by wt.	1.3	3	10	10	9	4	7	4	6	3
MIL-C-15328a	VMCH/VAGH 50/50 by wt.	1.3	9	10	10	8	8	7	8	6	6
Bare Magnesium	VMCH/VAGH 50/50 by wt.	1.7	4	10	10	9	6	7	3	2	2
MIL-M-3171, Type III	VMCH/VAGH 25/75 by wt.	1.4	2	10	10	9	6	8	5	7	5
MIL-C-15328a	VMCH/VAGH 25/75 by wt.	1.6	7	10	10	8	8	6	7	3	7
Bare Magnesium	VMCH/VAGH 25/75 by wt.	1.7	4	10	9	8	3	6	3	3	2
MIL-M-3171, Type III	VAGH Resin	1.4	2	10	10	8	5	6	4	5	2
MIL-C-15328a	VAGH Resin	1.7	8	9	10	7	8	5	5	4	3
MIL-M-3171, Type III	Styrenated Ether Ester	2.0	7	10	10	10	4	8	4	8	4
MIL-C-15328a	Styrenated Ether Ester	1.7	8	10	10	9	2	7	1	5	1
Bare Magnesium	Styrenated Ether Ester	1.5	9	10	10	9	2	7	2	6	2

Note: All primers were applied to the faying surfaces plus a one inch overlap only.

Table 152

Emerson & Cuming Amine Catalyzed Epon Physical Characteristics

Identification of System	Total Film Thickness (Mils)	Adhesion*	Water Resistance**	Solvent Resistance
Specification MIL-P-6889a, Type I MIL-L-7178, Aluminized	2.10	9	1	10
Vinyl Toluene-Ether Ester, Pilot Plant Batch	2.20	9	9	2
Amine Catalyzed Ether Resin With $SrCrO_4 + CaS$	2.10	9	9	9

*Over magnesium after about 30 days aging.
**Adhesion by tape test after 24 hours immersion.

Table 153

Emerson & Cuming Amine Catalyzed Epon Salt Spray Performance

Identification of System	3% Salt Spray Performance					
	24 hr.		72 hr.		312 hr.	
	Cor.	Blister	Cor.	Blister	Cor.	Blister
Specification MIL-P-6889a, Type I MIL-L-7178, Aluminized	10	10	10	6	8	3
	10	10	10	7	8	4
	10	10	10	6	8	3
	10	10	10	6	10	3
Vinyl Toluene-Ether Ester, Pilot Plant Batch	10	10	10	10	10	6
	10	10	10	10	10	6
	10	10	10	9	10	6
	10	10	10	10	10	6
	24 Hr.		192 hr.		264 hr.	
Amine Catalyzed Ether Resin With $SrCrO_4 + CaS$	10	7	10	5	9	3
	10	6	10	4	9	3
	10	6	10	4	9	3
	10	6	10	4	9	3

Table 154

Physical Characteristics of Final Vinyl Toluene Ether Ester Systems

System	Solvent Resistance Fresh Paints Recovery 0 Hr.	 24 Hr.	 Aged Paints* Recovery 0 Hr.	 24 Hr.	Water Resistance Tape Test	 24 Hr. Recovery	Adhesion Orig.	 20 Days
Vinyl Toluene-Benzoic Acid-Ether Ester System (Low vinyl aryl content)	8	8	4	5	8	9	7	9
Vinyl Toluene-Divinyl Benzene Ether Ester System (Sparged)	7	8	2	3	9	9	7	9

*Paints aged 11 days at 135° F

Table 155

Salt Spray Evaluation of Final Vinyl Toluene Ether Ester Systems

System	Film Thick. (Mils)	3% Salt Spray Performance 24 hr. Cor.	 Blister	 96 hr. Cor.	 Blister	 264 hr. Cor.	 Blister	 432 hr. Cor.	 Blister
Vinyl Toluene-Benzoic Acid-Ether Ester System (Low vinyl aryl content)	2.00	10	10	10	10	10	10	10	10
		10	10	10	10	10	10	10	10
		10	10	10	10	10	10	9	10
		10	10	10	10	10	10	10	10
Vinyl Toluene-Divinyl Benzene-Ether Ester System (Sparged)	2.00	10	10	10	10	9	4	9	3
		10	10	10	9	10	3	10	3
		10	10	10	9	10	5	10	3
		10	10	10	7	9	3	9	2

B. Commercial Pretreatments

1. Chicago Concern

This pretreatment was done by a Chicago concern under the observation of one of the project chemists. Since the procedure varied somewhat from MIL-M-3171, Type III it is outlined as follows:

1. Trichlorethylene degrease
2. Hot alkali clean (dubois Cleaner, 10 to 15 minutes immersion followed by a cold water rinse)
3. Hydrofluoric acid dip (21% HF at room temperature. Five minute dip followed by a cold water dip)
4. Chromic acid dip. (10 pounds CrO_3 in 50 gallons of water with the pH held below 3. Three minute dip followed by a cold water rinse.)
5. Hydrofluoric acid dip. (21% HF at room temperature. Two minute dip followed by hot water rinse.)
6. Dichromate treatment. (1.5 pounds $Na_2Cr_2O_7$ per gallon of water plus sufficient Ca F_2 in a bag to maintain an essentially saturated solution. Forty five minute dip at room temperature followed by a cold and a hot water rinse.)

2. New York Concern

This was the initial lot done by this concern and included three variations in pretreatment, as follows:

1. Mechanical buffing or satin finish
2. Chromic acid pickle
3. Nitric acid pickle

C. Formulations

1. Wash Primers

a. Butyral - Melamine Vehicle

Base Composition	Weight
Butvar B-76	40.5 gm
Resimene 881	31.5 gm
Basic Zinc Chromate	54.0 gm
Celite 266	22.5 gm
Methyl Ethyl Ketone	162.0 gm
Ethanol, 95%	153.0 gm

Acid Diluent	
85% Phosphoric Acid	35.0 gm
Normal Butanol	43.0 gm

b. Polyvinyl Formal

Base Composition	Weight
Basic Zinc Chromate	54.0 gm
Mica	9.0 gm
Formvar F-770	58.5 gm
Normal Butanol	166.5 gm
Xylene	216.0 gm

Acid Diluent*	
85% Phosphoric Acid	4.5 gm
Normal Butanol	48.0 gm
Xylene	48.0 gm

*Use one volume of diluent per two volumes of base.

c. Chrome-Phosphate Pigment

Base Composition	Weight
Polyvinyl Butyral, XYHL	45.0 gm
Chromium Phosphate	45.0 gm
Talc	7.0 gm
Isopropanol	272.5 gm
Methyl Isobutyl Ketone	80.5 gm

Acid Diluent	Weight
85% Phosphoric Acid	9.0 gm
Water	9.0 gm
Isopropanol	32.0 gm

d. Chrome-Phosphate Butyral Resin Solution

Composition	Weight
Chrome-Phosphate Butyral Resin Solution	365.0 gm
Basic Zinc Chromate	43.0 gm
Talc	7.0 gm
Normal Butanol	85.0 gm

e. Methacrylate Modified Vinyl Copolymer Primer

Strontium Chromate	313 gm
Bakelite VAGH	281 gm
Aroclor 1254	89 gm
Magnesium Silicate	313 gm
Lucite HG-41	299 gm
Butyl Cellosolve	86 gm
Toluene	1061 gm
Xylene	390 gm
Methyl Ethyl Ketone	304 gm
Methyl Isobutyl Ketone	452 gm

f. Polysulfide-epoxy Coating

Epoxy resin base:

TiO_2, Rutile, Non-Chalking	100 gm
Lampblack	3 gm
Epon 834	258 gm
Xylene	160 gm
Total	521

Polysulfide Resin Base:

Thiokol LP-3	170 gm
Beetle 216-8	4 gm
Tetraethylene pentamine	26 gm
Total	200

These two bases are mixed together in the above proportions just prior to use.

g. Emerson & Cuming Primer 8553

Physical Constants

- Weight per gallon ---------------------- 10.2 lb.
- Per cent solids ------------------------ 53.5%
  - Resin solids -------------- 44%
    - Epon 1001 --------------- 97%
    - Beetle 216-8 ------------ 3%
  - Non-Resin solids ---------- 56%
    - Inhibitive pigment ------ 84%
      - $SrCrO_4$ -------- 70%
      - CaS ----------- 30%
  - Extender, etc. ------------ 16%
- Per cent solvent ----------------------- 46.5
  - Thinner composition
    - Methyl isobutyl ketone -- 50%
    - Toluene ----------------- 50%

Accelerator Composition

Diethylene triamine (Tech.) ------------ 50%
Toluene -------------------------------- 50%

Reduction and Application

For application mixture add accelerator to primer in ratio 1 ml. accelerator to 30 g. primer. Mix well by shaking. Apply by spray within 4 hours. Thin as necessary with MIBK/toluene (1/1), using minimum amount of thinner.

h. Vinyl Toluene-Ether Ester Primer

Zinc Chromate	969 gm
Silicon Dioxide	165 gm
Lead Naphthenate 24%	12 gm
Cobalt Naphthenate 6%	6 gm
Xylene	480 gm

Vinyl Toluene Ether Ester, 50% NVM	1446 gm
Xylene	75 gm

i. Vinyl Toluene-Ether Ester Topcoat

Vinyl Toluene Ether Ester, 50% NVM	185 gm
Manganese Naphthenate, 6%	0.8
Cobalt Naphthenate, 6%	1.5
Toluene	124
Aluminum Paste, Std.	32

D. Vehicle Cooking Procedures

1. Styrenated Ether Ester (Pilot Plant Batch)

In a 100 gallon stainless steel kettle equipped with agitator, gas inlet tubes, thermometer, condenser and water separator 143 lb. of soya fatty acids were copolymerized with 143 lb. of monomeric styrene. This addition requires 14 hours, during which time 425° F is maintained. An inert gas blanket is turned on at the end of the copolymerization. The mixture is cooled to 350° F and 71 lb. of Epon 1004 is added. When this resin melts out and 350° F is reached another 71 lb. of Epon 1004 is added. This procedure is followed once again so that three 71 lb. additions of Epon 1004 are made. The processing temperature is then raised to 400° F and an inert gas blow of 3 cfm is begun. Then the mixture is held for 1 hour at 420° F. Finally the batch is heated to 470° F and held until the acid value is below 1, the viscosity (at 50% in xylol) is L-N (Gardner-Holdt tube) and the cure is 20 seconds maximum. When these characteristics are achieved the batch is thinned with xylol to 50% solids.

Final Characteristics

Base Yield	430 lb.
Xylol	430 lb. (50%)
Output	860 lb. (107 gal.)
Viscosity	L-N (Gardner-Holdt Tube)
Color	9 max. (Gardner)
AV (NVM)	1 max.
Wt./Gal.	7.97
Cure (sec.)	20 max.
Over-all batch time	Approx. 28 hours

2. Vinyl Toluene-Ether Ester (Pilot Plant Batch)

In a 100 gallon stainless steel kettle equipped as for 1., heat 157-3/4 lb. of soya fatty acids to 350° F under an inert gas blanket. Add 236 lb. Epon 1004 in three equal increments, melting the resin and heating back to 350° F before each addition. After these additions begin adding a mixture of 92 lb. of vinyl toluene, 13-1/4 lb. of 20-25% divinyl benzene and 1 lb. of di tertiary butyl peroxide. This addition requires 2 hours with the batch temperature at 350° F. (The inert gas blanket is discontinued during this addition.) The batch is held 30 minutes at 350° F after the addition. The mixture is then heated to 470° F. At this point a 1 cfm inert gas blow is begun. After one hour the blow is increased to 2 cfm and one hour later to 3 cfm. The mixture is then held at 470° F with the 3 cfm blow until the acid value is below one, the viscosity is T-V and the cure is 10 seconds maximum. When these characteristics are obtained the batch is cut with xylol to 50% solids.

Final Characteristics

Base Yield	464 lb.
Xylol	464 lb. (50%)
Output	928 lb. (116 gal.)
Viscosity	T-V (Gardner-Holdt Tube)
Color	6 max. (Gardner)
AV (NVM)	1 max.
Wt./gal.	7.97
Cure (Sec.)	10 max.
Over-all batch time	Approx. 12 hours

3. Vinyl Toluene-Ether Ester (Increased Epon Content)

In a 100 gallon stainless steel kettle equipped as for 1 and 2 heat 300 lb. of Epon 1004 and 44 lb. of technical benzoic acid to 470° F under an inert gas blanket. This temperature is maintained for 1-1/2 hours. Then 100 lb. of dehydrated castor fatty acids are added and the batch temperature is brought to 470° F and held until an acid value of 4-5 is reached. The mixture is cooked to 350° F and a mixture of 66.6 lb. of vinyl toluene and 1 lb. of di tertiary butyl peroxide is added over a three hour period. The batch temperature is then raised to 440° F. At this point a 1 cfm inert gas blow is begun. These conditions are maintained until a 6 second cure and acid value of 3 maximum is attained. When these characteristics are achieved the mixture is thinned with xylol to 50% solids.

Final Characteristics

Base Yield	480 lb.
Xylol	480 lb.
Output	960 lb. (116 gal.)
Viscosity	V-X (Gardner-Holdt Tube)
Color	6 max. (Gardner)
AV (NVM)	3 max.
Wt./gal.	8.15
Cure (Sec.)	8 max.
Over-all batch time	Approx. 12 hours