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Development of a Silane-Hydrolysate Binder for UV-Resistant Thermal Control Coatings

W. J. Patterson

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W. J. Patterson
George C. Marshall Space Flight Center
Marshall Space Flight Center, Alabama

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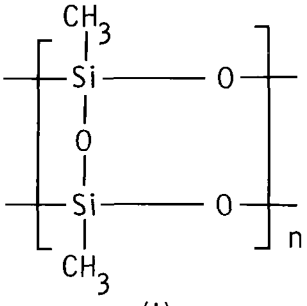
TECHNICAL PAPER

DEVELOPMENT OF A SILANE-HYDROLYSATE BINDER FOR UV-RESISTANT THERMAL CONTROL COATINGS

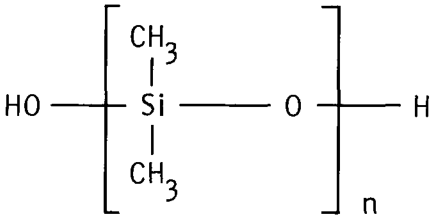
I. INTRODUCTION

This project was initiated to investigate the utility and thermal control coating performance of methyl silicone resins of the type ideally represented by Structure I:



(I)

The effort was to encompass the chemical synthesis of this "laddered" methyl silicone, an assessment of its solubility, tractability and curing behavior, and a study of its ability to be formulated in S-13G type passive thermal control coatings. The current thermal control coating state-of-the-art is represented by S-13G, which is a zinc oxide-filled formulation utilizing a hydroxyl-terminated dimethylsiloxane as a binder (Structure II):



$$HO-\left[Si(CH_3)_2-O\right]_n-H$$

(II)

It is believed that methyl silicones would potentially be more resistant to UV-induced yellowing that dimethylsilicones. Comparison of structures I and II shows that the methyl silicone (I) contains fewer of the radiation-vulnerable Si-CH_3 linkages, based on repeating segment size, than does Structure II. Another limitation of the S-13G coating is its relative susceptibility to handling damage. The proposed methyl silicones are expected to be more highly crosslinked to result in a substantially tougher, more damage-resistant coating.

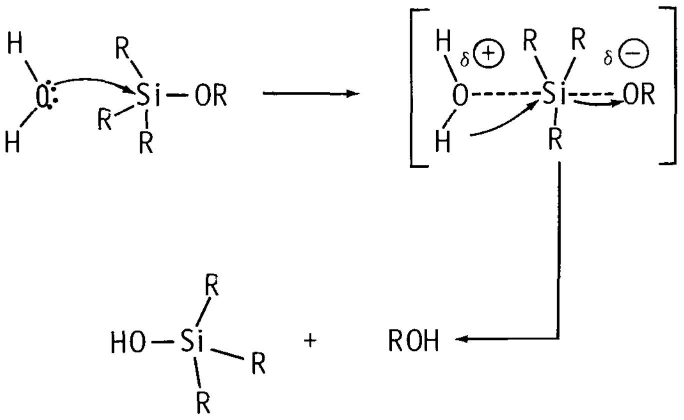
One type of laddered methyl silicone is now in limited commercial production and has been evaluated to the extent of defining erratic cure advancement or "coasting" as a significant problem. When applied to substrates as a thermal control coating formulation, the film progressively embrittles to a degree that detracts seriously from its coating value. This type of material is believed to involve direct hydrolysis and polymerization of the corresponding halosilane. The patent literature also contains reports of alkoxysilane hydrolysates as coating binders [1] but no UV-stability data are given.

II. COATING DEVELOPMENT

The scope of this project was to develop readily hydrolyzable monomers or oligomers with appropriate functionality to result in laddered methyl silicone polymers, and to evaluate these as binders for UV-resistant thermal control coatings.

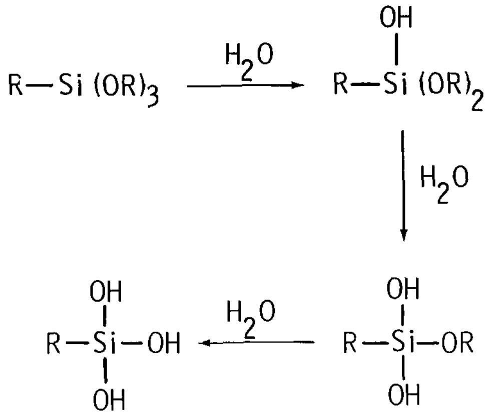
A. Theoretical Considerations

Nucleophilic displacement reactions at silicon, which occur in polar media, have been described by Sommer [2] as having an S_n2-like mechanism. This implies a bimolecular mechanism with backside attack by the nucleophile, resulting in simultaneous departure of the leaving group. Using an alkoxysilane as the substrate and water as the attacking nucleophile the following mechanism is operative:

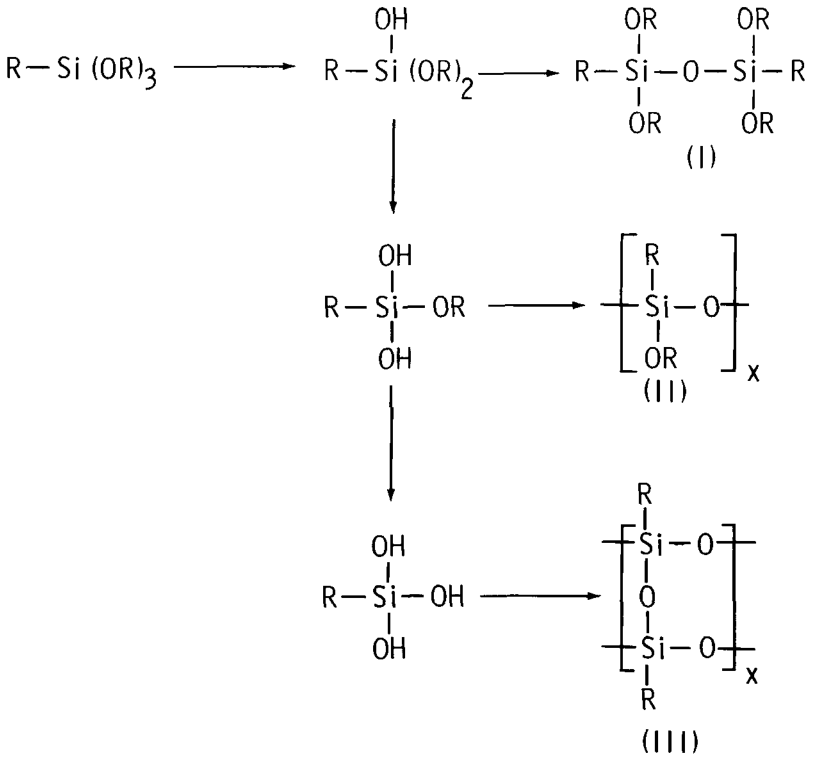


If one considers the analogous case in carbon chemistry, aliphatic ethers are not hydrolyzed under these conditions. The enhanced reactivity at silicon is generally attributed to the 3d electron participation in reducing the free energy of the transition state. An additional factor enhancing the reactions of Si-X relative to C-X is the greater polarizability of the Si-X bond. Even considering this rate enhancement, the hydrolysis of alkoxysilanes proceeds rather slowly. For the purposes of this study, the modest hydrolysis rate was exploited to generate alkoxysilane hydrolysates which remained soluble for further formation into coatings.

The concept of laddered siloxane polymers is based on condensation polymerization of trifunctional monomers [3-6]. The trialkoxysilanes were selected as a starting point in development of a controlled hydrolytic process leading to formation of linear, soluble oligomers with the potential for further crosslinking to an infusible solid. McNeil and coworkers [7] demonstrated, using the substrate $PhSi\ (O\ CH_2\ CH_2\ OCH_3)_3$, that hydrolysis leads to generation of the corresponding triol in a stepwise process:

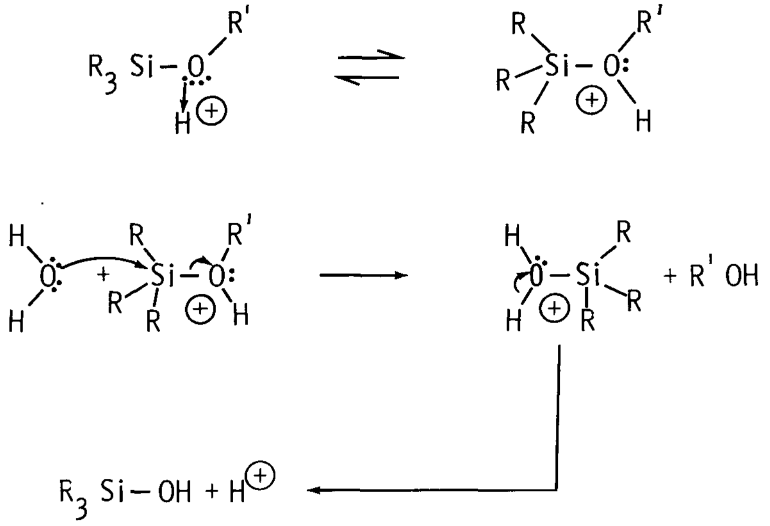


Generation of discrete intermediates was facilitated in this case by the stabilizing effect of the phenyl ring. If one now considers a totally aliphatic system such as $CH_3\ Si\ (OCH_2\ CH_3)_3$ (which would be required for UV-transparency in thermal control coatings), the intermediate silanols would be expected to exist only as transient species in a series of rather complex and competitive hydrolysis reactions:



Structure (III) represents the idealized "ladder" configuration, generated as a result of triol dehydration, although structures (I) and (II) could undergo further hydrolysis/dehydration reactions to form other laddered methyl silicone oligomers.

The hydrolysis of triethoxysilane monomers would utilize a low-cost, commercially available starting material and would generate only ethanol as a reaction product. This hydrolytic process under neutral conditions is somewhat sluggish, but is accelerated by acid catalysis as illustrated below:



Steric factors also profoundly affect hydrolysis rate. The methoxy group would present the least steric hindrance, but has the disadvantage of some toxicity [as $CH_3\ Si\ (OCH_3)_3$] and generates hazardous methanol as a hydrolysis by-product. The corresponding triethoxysilane is characterized by a three-fold reduction in hydrolysis rate due to increased steric hindrance, but acid catalysis accelerates this process to an acceptable time frame for coating formulation studies.

B. Silane Hydrolysate Development

A primary goal of this development was to establish hydrolysis conditions for formation of a soluble ladder polymer at modest temperatures (below 50°C) without addition of solvents. Implicit in this was the requirement for a low concentration of residual ethoxy or hydroxyl groups for subsequent reaction during the crosslinking phase (when applied as a coating binder).

The methyltriethoxysilane monomer was chosen for initial hydrolysis studies. This commercially available compound was purified by fractional distillation (b. 143°/760 torr) to a purity of 99.5 percent (glc) sealed in a dry, nitrogen-purged bottle and stored in the cold prior to use.

Initial hydrolysis studies involved the parameters of H_2O content, temperature and catalysis while monitoring the progress of the hydrolysis via high pressure liquid chromatography (hplc). The hplc measurements utilized a Waters micro-bondapack C_{18} reverse phase column with 50/50 methanol/water as the mobile phase at a flow rate of 1 ml/min.

A 10-gram sample of methyltriethoxysilane (MTES) was hydrolyzed with 3 mol-equivalents of water. The initially heterogeneous mixture was stirred rapidly at room temperature and aliquots were removed as a function of time for hplc analysis. The disappearance of the MTES peak was monitored with time and indicated significant monomer concentration remaining after 4 hr. The hydrolysis run was then repeated using various catalysts including hydrochloric, sulfuric, acetic and para-toluenesulfonic acids, as well as sodium and potassium hydroxide, stannous octoate, and tetraethylammonium hydroxide. The basic catalysts as well as stannous octoate resulted in partial gellation of the hydrolysate. The acidic catalysis was more effective in promoting hydrolysis without excessive crosslinking. Acetic acid seemed particularly suited to this reaction, and resulted in a modest rate acceleration as shown in Figure 1. In a typical acetic acid-catalyzed run at room temperature, the initially heterogeneous mixture became homogeneous and crystal clear after 10 min of vigorous stirring. The mild exotherm, which accompanied the reaction, peaked as sufficient ethanol was generated to establish a single phase. As indicated in Figure 1, the MTES monomer was virtually consumed after 4 hr.

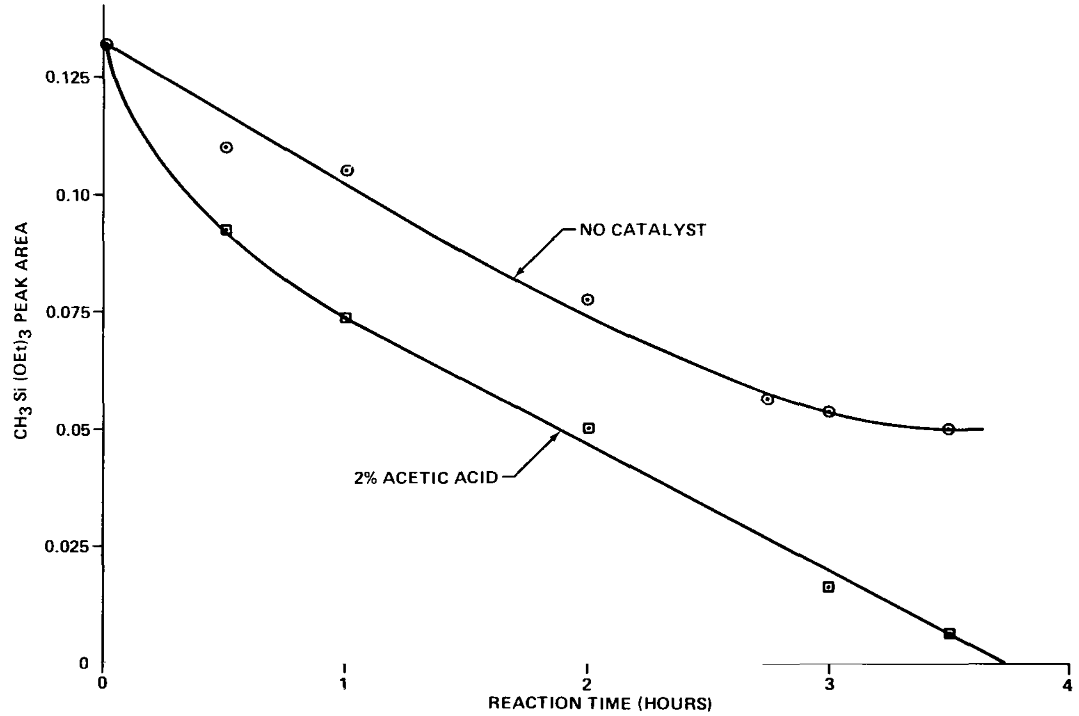


Figure 1. Effect of acid catalysis on rate of methyltriethoxysilane hydrolysis.

Figure 2. Optical clarity of silane hydrolysate polymer.

The effect of temperature on the hydrolysis rate was profound and undesirable. A similar hydrolysis run carried out at 50°C resulted in some gellation and precipitation of insoluble polymer. Considering the effectiveness of acid catalysis at room temperature for coating binder production, further temperature/catalyst combinations were not pursued.

The next step in MTES hydrolysate optimization involved the MTES/H_2O molar ratio. It was initially believed that stoichiometric ratios of H_2O (3 mol-equivalents of H_2O/ mol-equivalent of MTES) would lead to premature crosslinking and subsequent solubility problems for the hydrolysate, as well as embrittled coatings after cure. For this reason the range of 2 to 3 mol-equivalents of water was assessed first. Test solutions were prepared with 2, 2.5, and 3 equivalents of H_2O catalyzed with 2 percent (by weight) acetic acid, and stirred for 24 hr. The resulting clear solutions were extracted with chloroform and dried over Mg_2SO_4. The highly viscous, crystal clear residues (Fig. 2) were analyzed by gel permeation chromatography using micro-styragel columns and chloroform as a mobile phase. The molecular weight data from these chromatograms are given in Table 1. The polymer molecular weights increased as a function of H_2O concentration, but no gellation was detected.

TABLE 1. MOLECULAR WEIGHT DATA FOR METHYLTRIETHOXYSILANE HYDROLYSATES

MTES/H_2O Ratio	$[\eta]$[1]	$\overline{M}_w$	$\overline{M}_n$	MWD[2]
1.0/2.0	0.0241	4650	1020	4.56
1.0/2.5	0.0298	5119	891	5.75
1.0/3.0	0.0316	5340	940	5.68

1. Intrinsic viscosity, measured in $CHCl_3$ at 30°C.
2. Molecular weight distribution.

The liquid hydrolysates were assessed for curing characteristics by painting 3-5 mils onto clean aluminum substrates and allowing to cure for 24 hr at room temperature followed by 24 hr at 150°F. All formulations from Table 1 cured to tack-free clear coatings. Additional MTES/H_2O ratios of 10/1.5 and 10/3.5 were then prepared and applied as coatings. The 10/1.5 ratio remained tacky following the cured schedule, indicating a deficiency of reactive sites on the polymer chain. The 10/3.5 ratio cured but showed some tendency toward brittleness, which would be expected for a highly crosslinked system. Thus, the MTES hydrolysate prepared at room temperatures from 2 to 3 mol-equivalents of water and catalyzed by 2 percent acetic acid was selected for further study as a binder in coating formulations.

C. Coating Formulation/Cure Studies

The coating formulation studies were carried out using a DuPont TiO_2 identified as R-900-01. Samples of this pigment were ball-milled in ethanol and sprayed onto aluminum discs to serve as UV absorptance specimens. The UV absorptance data for this study was generated on a Beckman DK-2A spectrophotometer with a Gier-Dunkle integrating sphere over a wavelength range of 0.27 to 2.5 microns. The pigment was quite UV-transparent in the wavelength range of interest (0.35 to 2.5) and had a calculated absorptance (α) of 0.17. The initial coating formulation parameters were pigment/binder ratio and coating thickness. The TiO_2 was freshly ball milled for 2 hr in ethanol to provide a master batch with a paste consistency. The pigment and MTES hydrolysate binder were blended until a creamy smooth consistency was achieved. The formulation was then filtered through three layers of cheesecloth and sprayed at approximately 3 mil thickness onto aluminum panels using a Paasche air brush. The panels were cured for 24 hr at 70 to 80°F plus 24 hr at 150°F. The pigment/binder ratios evaluated are shown in Table 2, together with bend test results.

TABLE 2. OPTIMIZATION OF COATING PIGMENT/BINDER RATIO

Pigment/Binder Ratio	α	Bend Test
10/26	0.22	Slight Crazing
12/26	0.21	No Crazing
14/26	0.22	No Crazing
16/26	0.20	No Crazing

The bend tests were performed on a conical mandrel with the bend diameter decreasing to approximately 1/4-in. on one side of the test panel (Fig. 3). The bend area was inspected at 4X magnification and the degree of crazing or cracking was qualitatively determined. From the data in Table 2, it appears that a threshold pigment level exists, above which coating integrity (via the bend test) is independent of pigment content. The absorptance appears not to be sensitive to pigment level (the difference in observed α-values is not considered significant).

The effect of coating thickness was evaluated by preparing sprayed test panels in which the number of spray coats was varied from 1 to 10. The cured coating thicknesses varied from 0.7 to 8.7 mils. Corresponding α-values were determined and the data are presented graphically in Figure 4. This relation then allowed the spray operator to approximate, by the number of spray passes, the cured coating thickness. The absorptance was measured for each thickness and plotted as shown in Figure 5. It is apparent that α reaches a limiting value of 0.17 to 0.18 at a thickness of 5 to 6 mils. These same sprayed test panels were then subjected to the conical mandrel bend test with results given in Table 3.

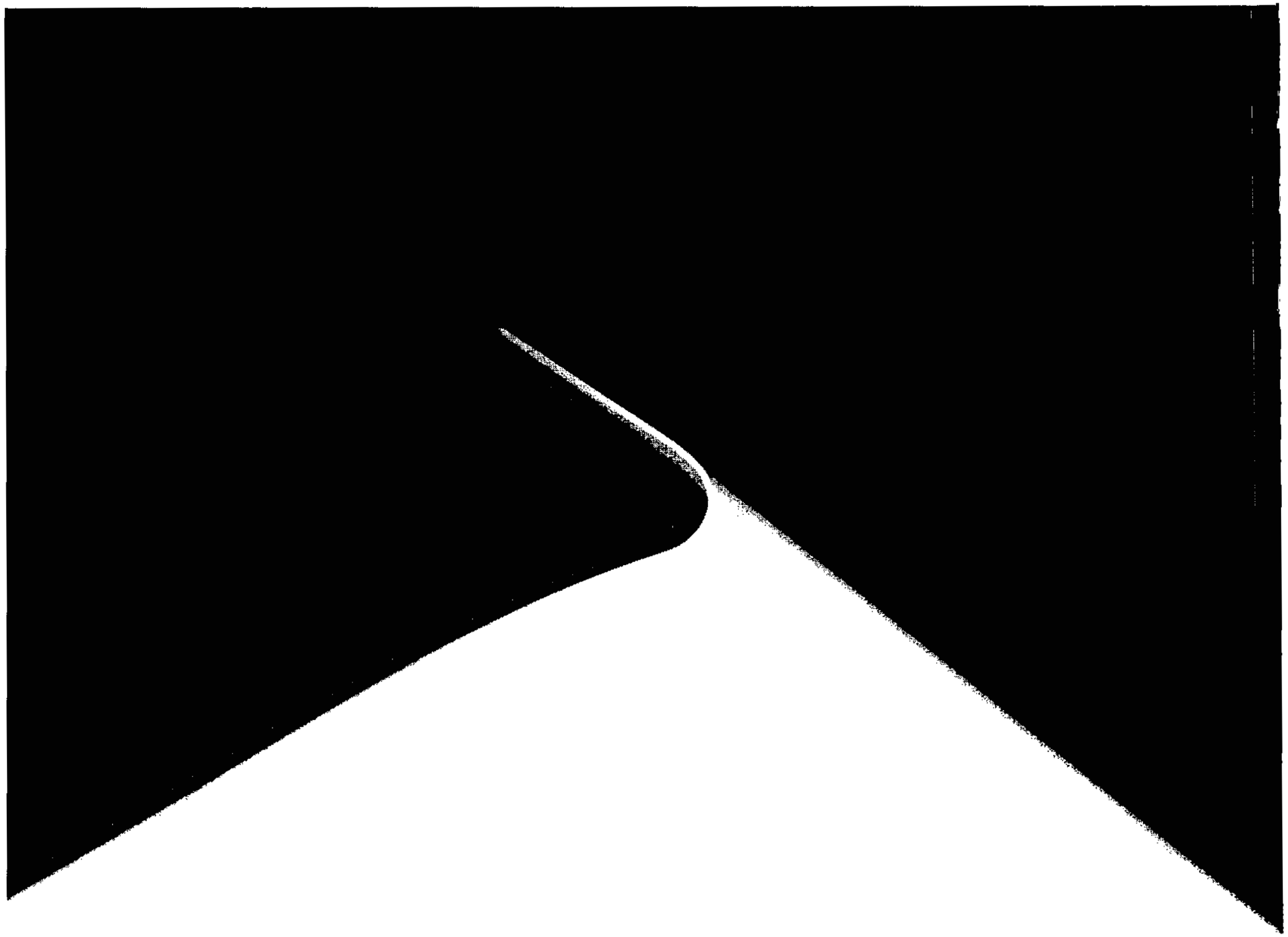


Figure 3. Bend test specimen from conical mandrel.

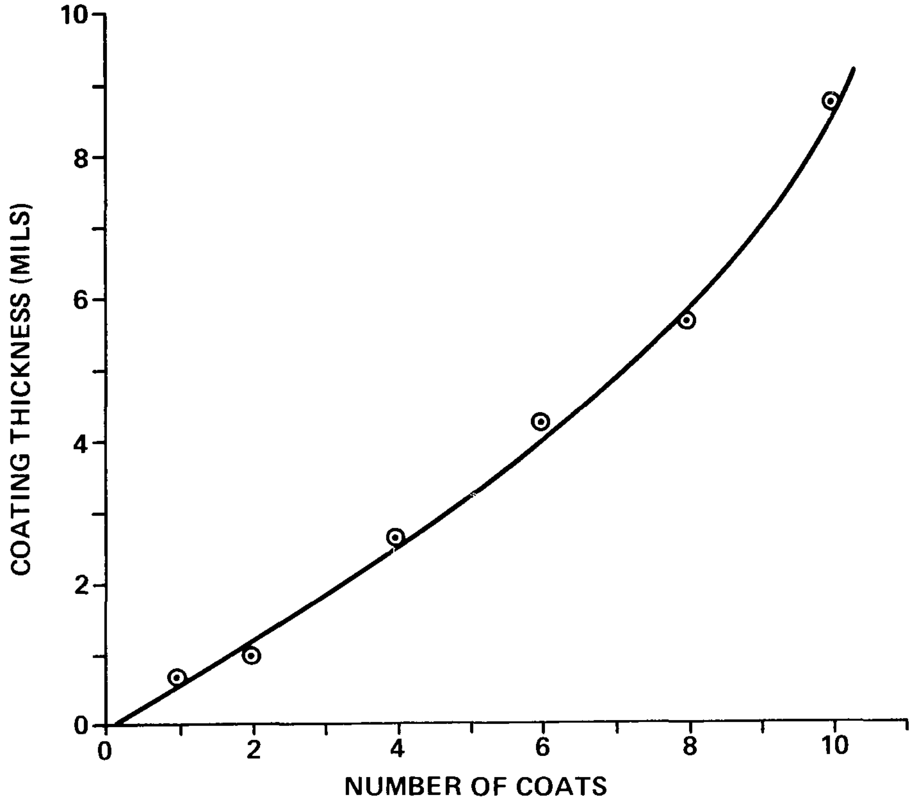


Figure 4. Coating thickness versus number of spray coats.

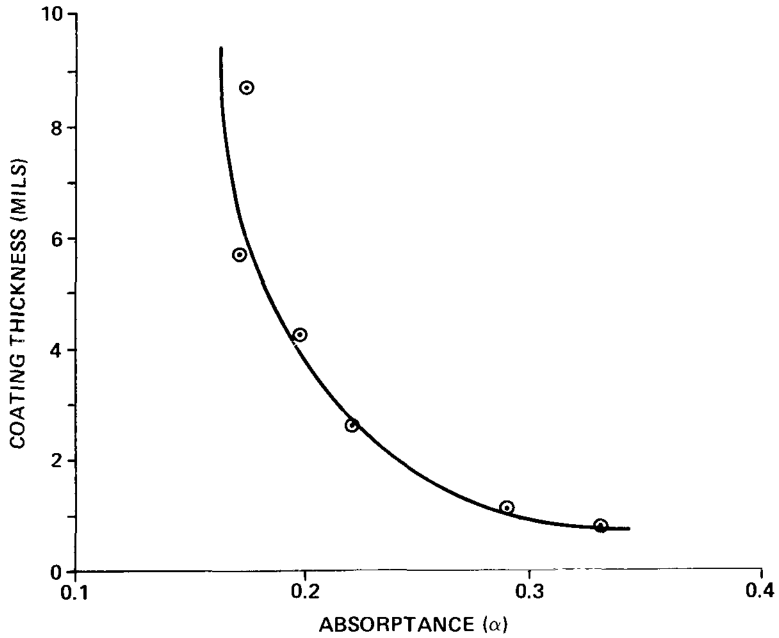


Figure 5. Dependence of α on coating thickness.

TABLE 3. COATING ADHERENCE VERSUS THICKNESS

Coating Thickness (mils)	Coating Condition After Bend Test
0.7	No craze/crack; good adherence
1.0	No craze/crack; good adherence
2.6	Very slight edge cracking
4.2	Edge cracking with crazing throughout bend area
5.7	Severe edge cracking and crazing
8.7	Severe cracking, coating loss

The absorptance data in Figure 5 indicate that a coating thickness of approximately 6 mils is required for minimum α values. However, as shown in Table 3, coating thicknesses above 2.6 mils are characterized by substantial loss of adherence and coating integrity. This thickness limitation is typically observed with many coatings and a practical compromise was necessary between coating integrity and UV absorptance. Since a coating thickness of 2 to 2.5 mils appeared to have good hiding power for the substrate, this was chosen as a nominal thickness for subsequent parameter optimization. The α-value at this thickness was approximately 0.2 (this is comparable to typical α-values for the S-13G thermal control coating).

The condition of the aluminum substrate was assessed as a means of improving coating adherence. Test panels were carefully degreased and one set was lightly wiped with a solution of Ludox (colloidal silica) while the other set was primed with DC1200, a standard silane primer. No improvement in coating adherence was observed in either case.

A final 5 by 12 matrix was generated to assess the effect of (1) water concentration in the hydrolysate and (2) cure time/temperature on the coating flexibility/adherence and degree of cure. The hydrolysates were formed using a range of MTES/H_2O ratios from 10/2.5 to 10/3.5. The cure schedules varied from 125 to 200°F and 1 to 7 days. The resulting test panels were subjected to both the mandrel bend test and a 24 hr alcohol exposure to determine degree of cure with results as shown in Table 4. These observations, although somewhat dependent upon the subjectivity of the observer, indicate certain trends. The best bend test data are found for MTES/H_2O ratios of 10/3.0 to 10/3.25 with cure schedules varying from 125°F/1 day to 175°F/3 days. The 200°F cure consistently results in poor coating/substrate adhesion. This is believed due to increased embrittlement of the coating through additional crosslinking at the higher temperatures. However, these same extreme cure conditions provided the greatest resistance to alcohol attack. Again, this is a manifestation of high crosslink density in the methylsilicone binder. Based on the results of Table 4, a practical coating system would reflect a compromise between coating adherence and degree of crosslinking, such as a 10/3.25 ratio cured at 150°F/3 days.

TABLE 4. MTES HYDROLYSATE COATING OPTIMIZATION MATRIX

	MTES/H_2O Ratio				
Cure Schedule	10/2.5	10/2.75	10/3.0	10/3.25	10/3.5
	a. b.				
125°F/1 Day	- / - -	++/- -	-/- -	++/- -	+/- -
125°F/3 Days	-/- -	+/- -	-/- -	++/- -	-/- -
125°F/7 Days	+/- -	+/- -	+/- -	++/-	-/- -
150°F/1 Day	+/- -	+/- -	++/- -	++/-	-/- -
150°F/3 Days	+/- -	+/- -	++/- -	++/-	-/- -
150°F/7 Days	+/- -	+/- -	++/- -	+/-	-/- -
175°F/1 Day	+/- -	+/- -	++/- -	+/-	-/- -
175°F/3 Days	-/- -	-/- -	++/- -	+/-	- -/-
175°F/7 Days	-/+	- -/-	+/+	+/+	- -/-
200°F/1 Day	-/+	- -/+	-/++	-/++	- -/+
200°F/3 Days	-/++	- -/+	-/++	- -/++	- -/++
200°F/7 Days	-/++	- -/+	-/++	- -/++	- -/++

a. Bend test results

b. Solvent resistance, indicating degree of crosslinking.

Rankings:

(++) = very good

(+) = marginal to good

(-) = poor to marginal

(- -) = very poor

III. COATING ASSESSMENT

The preferred coating formulation at this point in the development was based on methyltriethoxysilane hydrolysate (10/3.25 ratio of MTES/H_2O), formulated as a binder with ball-milled TiO_2 in a weight ratio of 12/26 TiO_2/binder. This formulation was spray-applied at a thickness of 2 to 2.5 mils to aluminum substrate and cured 24 hr/75°F plus 3 days/150°F. Figure 6 shows a typical coating application.

The UV-stability of this coating formulation was determined using a Spectrolab X-25 solar simulator which simulates, within 5 percent, the actual solar spectrum over the wavelength range of 0.25 to 1.3 microns. The samples were mounted on a water-cooled plate and the test chamber was evacuated to 10^{-6} torr. The absorptance data were collected over a 930 hr exposure period as shown in Table 5. These measurements were made in air and do not represent the worst case, since they do not include any oxygen-bleachable damage in the TiO_2 pigment.

TABLE 5. ULTRAVIOLET ABSORPTANCE OF SILANE HYDROLYSATE COATING

Sample	T_0	T_{266}	T_{506}	T_{930}
Experimental Coating #1	0.20	0.20	0.20	0.22
Experimental Coating #2	0.23	0.22	0.23	0.23
S-13GLO #1	0.20	0.21	0.22	0.23
S-13GLO #2	0.20	0.21	0.22	0.24

Figure 7 shows the specimens following the 930-hr exposure. No visible degradation was observed. Slightly yellowed, epoxy-based coatings (square samples) are also shown for contrast. The S-13GLO samples were run at the same time for comparative purposes.

The silane hydrolysate coating was cured for 24 hr at temperatures of 100, 150, and 175°F for outgassing assessment. The samples were tested in accordance with the requirements of MSFC Drawing 50MO2442. The samples were maintained at 10^{-6} torr and heated from 25 to 100°C at 2°C/min. The rate of weight loss in each case was less than 0.2 percent/cm^2/hr, which is considered acceptable for coating materials in the space environment.

Additional bend testing was performed to assess the coating integrity following thermal cycling. Appropriate samples were subjected to the mandrel bend test, and identical samples were thermal cycled from -150°F to 150°F for 5 cycles. Bend testing on the cycled specimens showed no significant increase in the tendency of the coating to craze or crack.

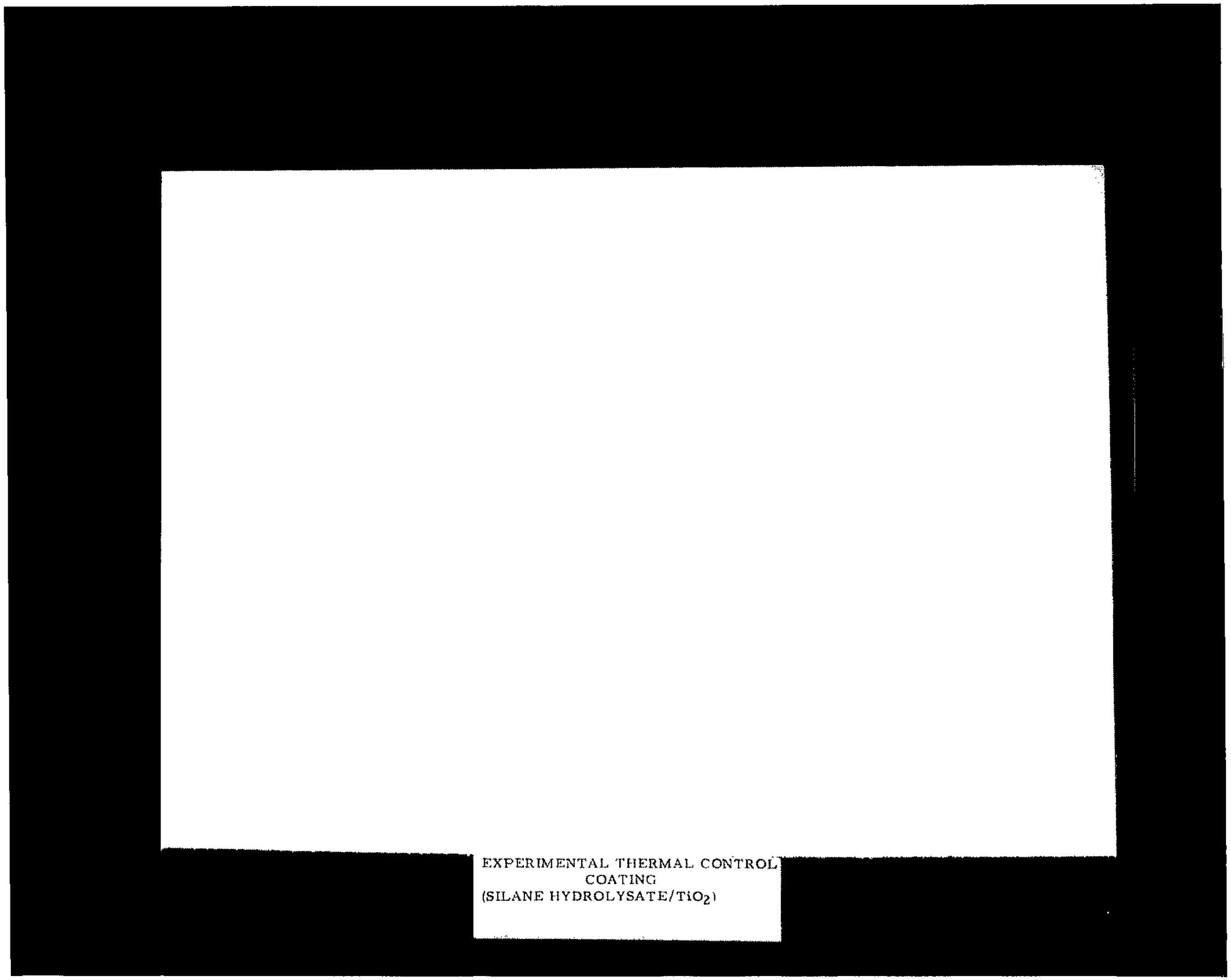


Figure 6. Typical sprayed panel of silane hydrolysate coating.

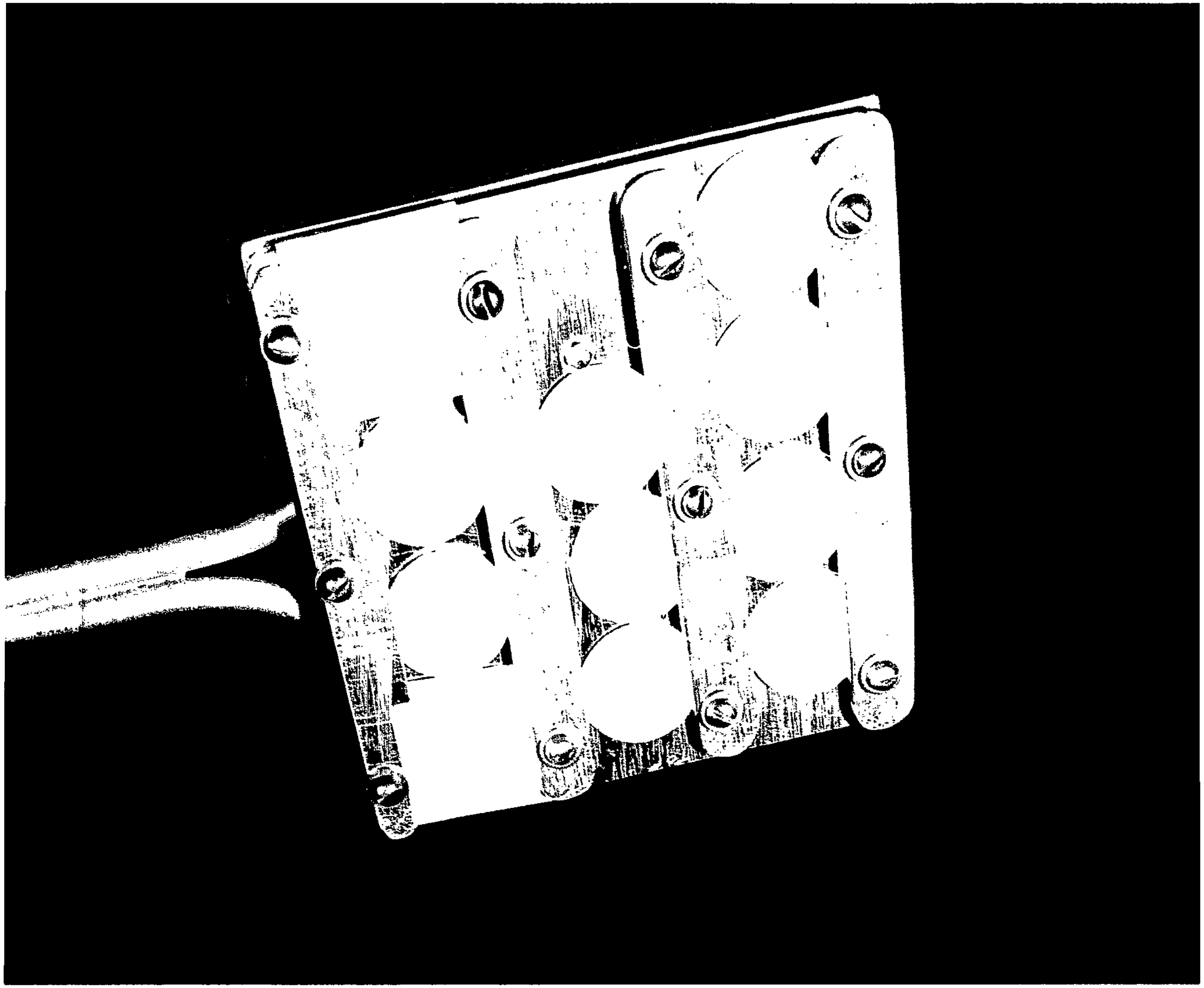


Figure 7. Test fixture with experimental coatings following 930 hour exposure.

One of the greatest potential advantages of the silane hydrolysate coating as opposed to the S-13GLO is the significant increase in damage resistance offered by the experimental system. The cured experimental coatings were not visibly damaged by thumbnail pressure, while the S-13G was readily removed. The abrasion resistance of these coatings was compared on a Taber abrasion tester, using CS-10 abrasive wheels with 500 grams of weight per wheel. At the end of 3000 revolutions, the S-13G samples had lost 4 to 5 times more coating weight than the experimental silane hydrolysate coating.

IV. CONCLUSIONS

This development study has led to the definition of a specific methyltriethoxysilane hydrolysate binder suitable for formulation as a candidate thermal control coating. The ease of spray application and cure under modest conditions (150°F/3 days) provide realistic processing conditions for flight hardware. The high inherent thermal stability and low outgassing characteristics of the binder would appear well suited for use on spacecraft with contamination-sensitive hardware. The UV absorptance values over 930 hr were not significantly different from those of S-13G, indicating that the reduction in silicon-methyl group concentration along the laddered polymer chain does not reduce the UV-damage susceptibility (based on short term exposures) to a measureable extent. Longer exposure times and in situ measurement techniques are needed to force a measurable degradation in the S-13G and assess the relative performance of the experimental coating.

The experimental coating is characterized by a substantially greater resistance to abrasion and accidental damage than the S-13GLO coating. This feature together with reasonable flexibility in bending of the coating substrate, should provide a very durable coating.

One of the greatest drawbacks of commercial resins with a "ladder" structure has been their tendency to "coast" or develop a higher degree of crosslinking and subsequently embrittle with time. The flex test results on the experimental coating did not indicate a significant tendency toward embrittlement, based on comparison of cure for 1 day or 7 days at the various test temperatures. The testing should be extended to periods of several months at various temperatures to conclusively assess the embrittlement tendency.

The silane hydrolysate binder system should be investigated with pigments other than TiO_2. Limited trials during this project with both S-13GLO and zinc orthotitanate pigments (both have greater intrinsic UV-stability than TiO_2) indicated binder compatibility problems. Similar effects in dimethylsilicone binders have been resolved by modifications in pigment surface chemistry, and further modifications should yield stable silane hydrolysate-based coatings.

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16. ABSTRACT

Detailed characterization and formulation studies have been performed on a methyltriakoxysilane hydrolysate as a binder for thermal control coatings. The binder was optimized by varying hydrolysis temperature, time, catalyst type, and water concentration. The candidate coating formulations, based on this binder with TiO_2 pigment, were optimized via a detailed series of sprayed test panels that included the parameters of binder/pigment ratio, ethanol content, pigment particle size, coating thickness and cure conditions. A typical optimized coating was prepared by acetic acid-catalyzed hydrolysis of methyltriethoxysilane with 3.25 mol-equivalents of water over a 24 hour period at room temperature. The resulting hydrolysate was directly mixed with pre-milled TiO_2 (12 grams pigment/26 grams binder) to yield a sprayable consistency. Panels were sprayed to result in a nominal cured coating thickness of 2 mils. Cure was affected by air drying for 24 hr at room temperature plus 72 hr at 150°F. These coatings are typically extremely tough and abrasion-resistant, with an absorptance (α) of 0.20 and emittance (ϵ) of 0.89. No significant coating damage was observed in the mandrel bend test, even after exposure to thermal cycling from -160° to 160°F. Vacuum exposure of the coatings for 930 hours at 1 equivalent UV sun resulted in no visible degradation and no significant increase in absorptance.

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