Synthesis and Polymerization of Tris(alkylamino)-silanes with Epoxy Prepolymers

N. BILOW and R. F. MURPHY, *Materials Technology Department,*
Hughes Aircraft Company, Culver City, California 90230, and
W. J. PATTERSON, *Marshall Space Flight Center,*
National Aeronautics and Space Administration, Huntsville, Alabama 35812

Synopsis

Phenyltris(methylamino)silane, phenyltris(ethylamino)silane, and vinyltris(hexyl-amino)silane were synthesized and evaluated as curing agents for epoxy resins. Cured resins derived therefrom have very good physical and dielectric properties. The moisture resistance of the resins evaluated was especially good for those derived from the vinyltris(hexylamino)silane. Typical dielectric properties of a phenyltris(ethylamino)-silane-cured epoxy specimen include a dielectric constant of 3.7 at 25°C. and a dissipation factor of 0.8%. Elevated temperature data are also presented.

INTRODUCTION

Silazanes constitute a class of compounds which were first synthesized by Cheronis around the mid-1940's and on which the first patents were issued in 1951.[1] The compounds are generally synthesized by the reaction of halosilanes with a large excess, generally at least fourfold,[2] of ammonia or various other amines.

Polymers of various types have been made from silazanes through hydrolysis,[3] through additions to polyisocyanates, and through condensations with diols.[4] Those silazanes derived from ammonia have also been homopolymerized at 60°C. with the liberation of ammonia.[1]
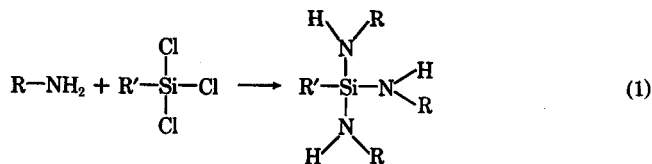
In a report by Weigel[5] the diglycidyl ether of isopropylidene diphenol was polymerized with hexamethylcyclotrisilazane and octaphenylcyclotetrasilazane. These polymerizations were found to proceed at 100°C. or higher. In these cases, gelation occurred within 1 hr. at 175°C., and the polymerizations were complete within 6 hr. at this temperature. Other research in this field was conducted by Burke,[6] whose investigations dealt with the use of difunctional silazanes such as bis(methylamino)diphenylsilane as epoxy polymerization agents rather than the trifunctional silazanes such as those reported herein. Schultz and Zike[7] have prepared epoxysilazane polymers by the *in situ* reaction of ammonia with mixtures of halosilanes and epoxy compounds. These polymers contain functional groups resulting from silazane–epoxy additions as well as from conventional

amine–epoxy additions.   Silazanes were also used by Boyer et al.[8] to polymerize mixtures of epoxy prepolymers with polyhydroxy compounds.

EXPERIMENTAL

In work carried out in the Hughes laboratories, silazanes which were studied as epoxy curing agents were noncyclic compounds derived from primary aliphatic amines.   In this respect they contrast with the ammonia-based cyclosilazanes studied by Weigel.[5]

Specific compounds investigated in the Hughes research are phenyltris-(methylamino)silane (I), phenyltris(ethylamino)silane (II), and vinyltris-(hexylamino)silane (III).   These compounds were synthesized by the reaction of the corresponding trihalosilane with a large excess (tenfold) of the appropriate anhydrous amine.   The general reaction is illustrated in eq. (1), where, for I, R = CH_3, R' = C_6H_5; for II, R = C_2H_5, R' = C_6H_5; for III, R = C_6H_{13}, R' = $-CH=CH_2$.

$$R-NH_2 + R'-\overset{\displaystyle Cl}{\underset{\displaystyle Cl}{\overset{|}{\underset{|}{Si}}}}-Cl \longrightarrow R'-\overset{\displaystyle \overset{H}{\underset{}{N}}\diagup R}{\underset{\displaystyle \underset{H}{\overset{}{N}}\diagdown R}{\overset{|}{\underset{|}{Si}}}-N\diagup^{H}_{\diagdown R} \qquad (1)$$

Various compounds of this type have been reported previously.   For example, a series of such compounds, derived from propyltrichlorosilane was synthesized by L. Tansjo.[9]   However, of the three silazanes synthesized in the Hughes research, only the phenyltris(ethylamino)silane was reported previously.[10]   It was used in curing hydroxyl- and alkoxyl-terminated silicones.

Monomers

Phenyltris(methylamino)silane (I).   A solution of phenyltrichlorosilane (23.3 g., 0.11 mole, Dow Corning Corp.) and anhydrous ether (50 ml.) was gradually added to a solution of dry methylamine (34.2 g., 1.1 mole) in anhydrous ether (200 ml.).   After completing the addition the reaction mixture was heated at reflux for 45 min. and excess methylamine was distilled off.   It was then filtered to remove methylammonium chloride and the filtrate was then distilled at ambient pressure to remove ether.   Distillation of the high-boiling material at 3 torr yielded 11 g. of phenyltris-(methylamino)silane boiling between 104 and 115°C.   Virtually all of the product boiled at 105°C.   The yield represents 51% of theoretical.

ANAL.   Calcd.: Si, 14.41%; N, 21.54%.   Found: Si, 14.76%; N, 20.30%.

Phenyltris(ethylamino)silane (II).   To a mixture of anhydrous ethyl-amine (450 g., 10 mole) in anhydrous ether (1 liter) was added dropwise

a solution of freshly distilled phenyltrichlorosilane (211 g., 1 mole) in anhydrous ether (200 ml.). Upon completion of the addition, the mixture was heated at reflux for 1 hr. After cooling and settling, the fluid phase was decanted and filtered. Ether and excess amine were then removed by simple distillation and the phenyltris(ethylamino)silane was distilled under reduced pressure (b.p. 123°C./5 torr). A yield of 196 g. (0.83 mole, 83%) was obtained.

ANAL. Calcd.: Si, 11.85%; N, 17.72%. Found: Si, 12.2%; N, 16.36%.

Vinyltris(hexylamino)silane (III). Freshly distilled vinyltrichlorosilane (b.p. 90°C.) and *n*-hexylamine (b.p. 132 ± 1°C.) were used in this preparation. The amine was dried over KOH before distilling.
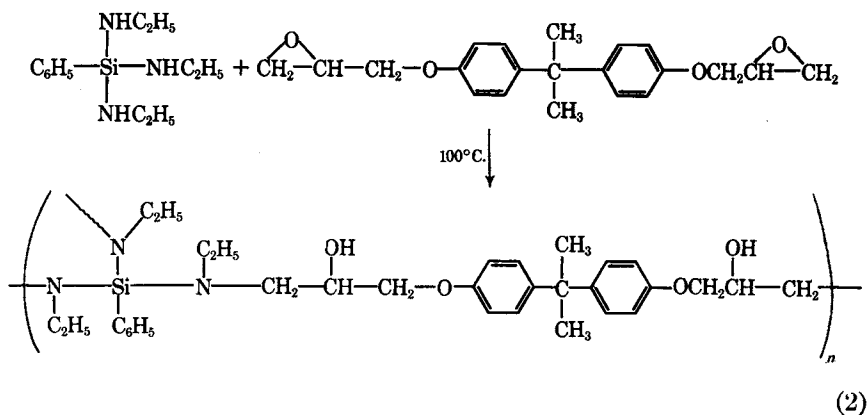
To a cold solution (−10°C.) of *n*-hexylamine (208 g., 2.05 mole) in anhydrous ether (100 ml.) was added dropwise (1 hr.) a solution of vinyltrichlorosilane (32.3 g., 0.20 mole) in anhydrous ether (75 ml.). Upon completion of the reaction the mixture was allowed to warm to room temperature and was then filtered to remove the hexylamine hydrochloride. Ether and excess amine were removed by distillation at ambient pressure and the residue was then extracted with naphtha. This naphtha solution was distilled first at ambient and then at reduced pressure (approximately 5 torr). Because of the high boiling point of the product (132–155°C.) it was redistilled at 1.5×10^{-3} torr and 115–140°C. Some degradation occurred at these temperatures, probably due to the high pot temperature (160–175°C.). A final distillation on a molecular still was carried out at 10^{-3} torr and two fractions were collected, one boiling at 95–105°C. (27.5 g.), pot temperature 98–107°C.; one boiling at 105–150°C. (5 g.), pot temperature 107–160°C. The total product ($\simeq$ 30 g.) represents a 42% yield.

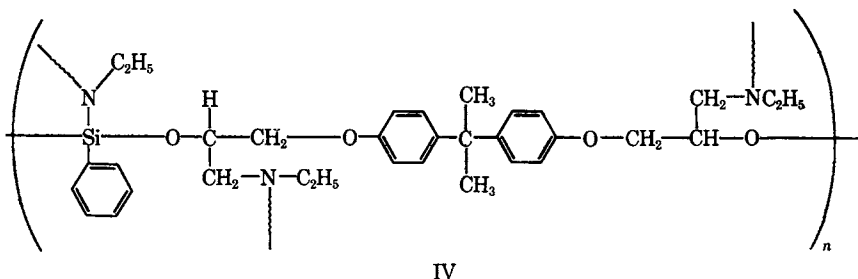ANAL. Calcd.: Si, 7.91%; N, 11.83%. Found: Si, 6.84%; N, 11.40%.

Deviations from ideality in the analyses are attributed to the extremely high moisture sensitivity of these compounds. Fuming invariably occurs in air with the subsequent evolution of the base amine. The most suitable techniques for handling these compounds include storing them in dried syringe bottles and transferring them either in a dry box or via a dry hypodermic syringe. Inadequately dried glassware promotes the formation of flocculent white precipitates in trace amounts, but this problem can be virtually eliminated when meticulously dried glassware is used.

Polymerizations

Silazanes of this type appear to function essentially as polyfunctional amines in their reactions with epoxy compounds as illustrated in eq. (2).

$$
\begin{array}{c}
\text{C}_6\text{H}_5-\underset{\underset{\text{NHC}_2\text{H}_5}{|}}{\overset{\overset{\text{NHC}_2\text{H}_5}{|}}{\text{Si}}}-\text{NHC}_2\text{H}_5 \; + \; \text{CH}_2\!-\!\text{CH}-\text{CH}_2-\text{O}-\!\!\!\bigcirc\!\!\!-\underset{\underset{\text{CH}_3}{|}}{\overset{\overset{\text{CH}_3}{|}}{\text{C}}}-\!\!\!\bigcirc\!\!\!-\text{OCH}_2\text{CH}-\text{CH}_2
\end{array}
$$

$$100^\circ\text{C}\downarrow$$

$$
\left(\!\!\begin{array}{c}
-\underset{\underset{\text{C}_2\text{H}_5}{|}}{\overset{}{\text{N}}}-\underset{\underset{\text{C}_6\text{H}_5}{|}}{\overset{\overset{\text{C}_2\text{H}_5}{\diagdown\;\text{N}\diagup}}{\text{Si}}}-\overset{\overset{\text{C}_2\text{H}_5}{|}}{\text{N}}-\text{CH}_2-\underset{}{\overset{\overset{\text{OH}}{|}}{\text{CH}}}-\text{CH}_2-\text{O}-\!\!\!\bigcirc\!\!\!-\underset{\underset{\text{CH}_3}{|}}{\overset{\overset{\text{CH}_3}{|}}{\text{C}}}-\!\!\!\bigcirc\!\!\!-\text{OCH}_2\overset{\overset{\text{OH}}{|}}{\text{CH}}-\text{CH}_2-
\end{array}\!\!\right)_n
$$

$$(2)$$

It is not unreasonable, however, to consider an entirely different cure process, namely, one which produces a cured resin with the structure IV.

$$
\left(\!\!\begin{array}{c}
-\underset{}{\overset{\overset{\text{C}_2\text{H}_5}{\diagdown\;\text{N}\diagup}}{\text{Si}}}-\text{O}-\underset{\underset{\text{CH}_2-\text{N}-\text{C}_2\text{H}_5}{|}}{\overset{\overset{\text{H}}{|}}{\text{C}}}-\text{CH}_2-\text{O}-\!\!\!\bigcirc\!\!\!-\underset{\underset{\text{CH}_3}{|}}{\overset{\overset{\text{CH}_3}{|}}{\text{C}}}-\!\!\!\bigcirc\!\!\!-\text{O}-\text{CH}_2-\underset{\underset{\text{O}}{|}}{\overset{\overset{\text{CH}_2-\text{NC}_2\text{H}_5}{|}}{\text{CH}}}-\text{O}-
\end{array}\!\!\right)_n
$$

IV

The stoichiometry of these two modes of polymerization is different. In the first process 1 mole of the silazane can be expected to react with $1\tfrac{1}{2}$ moles of the diepoxy compound, whereas in the second process 1 mole of the silazane could react with as many as 3 moles of diepoxy compound since all N—H groups in the latter polymer could react further. No evidence is available to substantiate either mode of reaction. One sample of cured resin was found to undergo a change in physical form after several months, however, and this may be an indication of a gradual change in the polymer structure.

The conditions utilized for polymerization of the epoxy intermediate, Epon X-24, with silazanes are summarized in Table I.

The first three specimens, namely 1, 2, and 3, were unfilled resins derived from Epon X-24 and phenyltris(methylamino)silane, phenyltris(ethylamino)silane, and vinyltris(hexylamino)silane, respectively. In formulating these resin compositions, it was noted that the phenyltris(methylamino)silane yielded homogeneous prepolymer mixtures which tended to cure very rapidly, and care was required to obtain specimens free of cracks. Phenyltris(ethylamino)silane, in contrast, also formed homogeneous prepolymer mixtures, but no exotherm problems were encountered. Cures proceeded smoothly at 85–100°C. with the higher temperature being

TRIS(ALKYLAMINO)SILANES 2113

TABLE I
Composition of Epoxysilazane Dielectric Test Specimens

Sample no.	Components[a]	Weight, g.	Equivalents	Time, hr.	Temperature, °C.
1	A	9.75	0.15	16	85
	D	25.5	0.15	6	100
2	B	11.1	0.14	2	23
				5.5	75
	D	23.8	0.14	15	85
				4	100
3	C	14.3	0.12	18	100
	D	20.4	0.12		
4	B	5.5	0.07	64	75
	D	11.9	0.07	5	100
	I	c			
5	C	10.7	0.09	21	100
	D	15.3	0.09		
	I	c			
6	B	7.9	0.10	15	75
Same as 4	D	17.0	0.10	4	100
except for	I	c			
milder post-					
cure					
7	B	5.5	0.07	1	23
	D	11.9	0.07	5	75
				15	85
	G	43		4	100
8	B	6.3	0.08	2.5	75
	E	2.2	0.08	15	85
	D	27.3	0.16	4	100
9	B	5.5	0.07	1.5	75
	F	7.9	0.07	15	85
	D	23.8	0.14	4	100
10	B	5.5	0.07	15	85
	H	7.4	0.07	4	100
	D	23.8	0.14		

[a] Code: A = phenyltris(methylamino)silane; B = phenyltris(ethylamino)silane; C = vinyltris(hexylamino)silane; D = 2,2-bis(2,3-epoxypropoxyphenyl)propane, Epon X-24, Shell Chemical Co.; E = m-phenylenediamine (Eastman-white label); F = N-methylaniline formaldehyde (synthesized at Hughes); G = 00 smoked silica; H = N,N'-diphenylethylenediamine (Eastman-white label); I = Eccospheres SI (dried at 250°C. from Emerson and Cuming, Los Angeles, California).
[b] All cures carried out under nitrogen.
[c] Maximum obtainable: about 20 wt.-%.

preferred. Vinyltris(hexylamino)silane, on the other hand, was difficult to work with in that it was incompatible with Epon X-24. To obtain homogeneous cured resins, therefore, it was necessary to react this epoxy compound with the silazane under nitrogen at 90–100°C. with constant stirring until sufficient interaction occurred to produce a homogeneous

mixture. This mixture could then be cast and cured at 100°C. to produce colorless, transparent, homogeneous resin specimens.

The second set of specimens, namely 4–7, were filled resins derived from Epon X-24 and either phenyltris(ethylamino)silane or vinyltris(hexylamino)silane. Phenyltris(methylamino)silane was not used in making filled samples. Samples 4–6 contained silica microballoons (Eccospheres SI) and sample 7 contained OO smoked silica. Because of its high packing density, the smoked silica did not yield void-free specimens when vacuum impregnation techniques were used; however, this filler was not evaluated extensively. Microballoon filled specimens were prepared from vacuum-degassed resin–filler-premixes.

Specimens 4 and 6 were phenyltris(ethylamino)silane-cured resins and differed in their cure procedure. The former, which had the more extensive cure, had significantly better dielectric properties as will be noted later.

Specimens in the last group, specifically 8, 9, and 10, were terpolymers derived from phenyltris(ethylamino)silane, Epon X-24, and an aromatic amine. In each case, stoichiometrically equivalent amounts of the silazane and the aromatic amine were used. These amines included m-phenylenediamine, N-methylaniline formaldehyde, and N,N'-diphenylethylenediamine. As far as dielectric properties were concerned, the copolymerization of the aromatic amines with the silazane did not appear to have any special merit and, in fact, detracted from the dielectric properties.

DISCUSSION

Cured resins derived from 2,2-bis(p-epoxypropoxyphenyl)propane [Epon X-24, Shell Chemical Company] and the above-mentioned silazanes were completely colorless transparent products having good dielectric and physical properties. For example, the phenyltris(ethylamino)silane(II)-cured resin had the following characteristics: compressive strength, 21,600 psi; modulus of elasticity in compression, 3.4×10^6 psi; modulus of elasticity in tension, 3.5×10^5 psi; tensile strength, 3,020 psi; elongation, 0.9%; density, 1.23 g./cc. at 23°C.

Measurements of three electrical properties of the epoxy–silazane polymers, namely dielectric constant, dissipation factor, and insulation resistance, were made to determine the potential of these materials in electrical potting formulations.

Dielectric properties of the various silazane cured epoxies were determined by using cylindrical specimens $4\frac{1}{2}$ in. long and $\frac{5}{8}$ in. wide, in which were embedded four 0.16-in. diameter brass electrodes placed 0.33 in. apart on center. The test results are presented in Table II.

Coefficients of linear thermal expansion were not determined on pure resin but were determined on specimens filled with silica microballoons (Emerson and Cuming, Eccospheres SI, approximately 20 wt.-%). The following results were obtained: between −56 and −4°C., 6.20×10^{-5}

TABLE II

Dielectric Properties of Various Silazane-Cured Epon X-24 Specimens

Sample No.	Dielectric constant (1 kc.)					Dissipation factor, %					Insulation resistance, megohm at 500 v. d.c.					Filler[c]	Curing agent[b]
	25°C.	100°C.	125°C.	150°C.	25°C.[a]	25°C.	100°C.	125°C.	150°C.	25°C.[a]	25°C.	100°C.	125°C.	150°C.	25°C.[a]		
2	3.68	3.74	3.97	4.27	3.68	0.81	1.12	2.14	7.30	0.81	0.5×10^7	1×10^3	1×10^3	85	1×10^7		A
3	3.47	4.17	4.35	4.87	3.42	0.04	0.76	0.94	0.55	0.07	$>5 \times 10^7$	2×10^4	7×10^2	90	$>5 \times 10^7$		B
4	2.40	2.54	2.56	2.86	2.34	0.12	0.44	0.55	2.26	0.14	3×10^6	9×10^3	2×10^2	40	5×10^6	G	A
4[d]	2.31	2.29			2.30	1.71	4.69			1.71	3×10^6	5.8			3×10^6	G	A
5	2.41		2.91	2.94	2.41	0.02	0.13	0.28	1.21	0.06	4×10^6	1×10^3	2×10^2	50	$>5 \times 10^7$	G	B
6[e]	2.38	2.80			2.41	1.96	7.29			2.32	3×10^6	4.0			3×10^6	G	A
7	6.62	7.10	6.92	7.19	6.59	0.42	1.22	1.78	3.10	2.54	1×10^7	3.4×10^3	1.4×10^3	260	5×10^7	F	A
8	4.00	4.57	5.23	6.17	4.27	1.20	1.78	5.96	10.18	1.22	5×10^7	1.1×10^4	4×10^2	28	5×10^7		E
9	3.79	4.04	4.38	5.10	3.82	0.91	0.96	3.46	7.02	0.86	5×10^7	4×10^6	7.5×10^3	120	5×10^7		C
10	3.70	5.86	6.31	6.44	3.69	0.80	9.6	22.36	34.82	0.64	$>10^7$	2.5×10^3	15	2.2	5×10^7		D

[a] Reevaluated after the elevated temperature tests were completed.

[b] A = phenyltris(ethylamino)silane; B = vinyltris(hexylamino)silane; C = phenyltris(ethylamino)silane plus *N*-methylaniline formaldehyde (1:1); D = phenyltris(ethylamino)silane plus *N,N'*-diphenylethylenediamine (1:1); E = phenyltris(ethylamino)silane plus *m*-phenylenediamine (1:1).

[c] F = OO smoked silica; G = silica microballoons, Eccospheres SI.

[d] Reevaluated after being submerged in boiling water for 40 min. and redrying for 3 days at 24°C./5 torr.

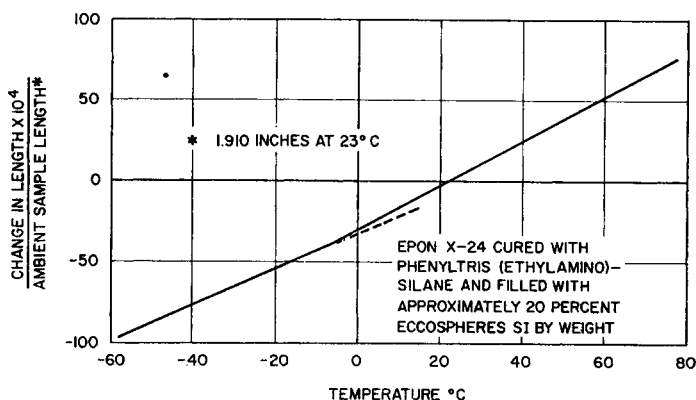[e] Same as 4 except for a much shorter cure period.

Fig. 1. Thermal expansion of filled epoxysilazane.

in./in./°C.; between −4 and +100°C., 7.35 × 10⁻⁵ in./in./°C. This resin appeared to have a transition point at about −4 to −6°C. as seen in Figure 1. Presumably this results from a form of crystallization.

Tests were also carried out on Eccosphere SI-filled samples to determine the extent to which they outgassed in high vacuum both at ambient and at elevated temperature. Duplicate samples were evaluated, and the results obtained are given in Table III.
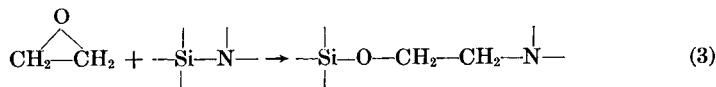
TABLE III

Time and temperature	Pressure range, torr	Total weight loss, %
72 hr. at 23°C.	3 × 10⁻⁶	0.080 ± 0.002
72 hr. at 23°C. plus 24 hr. at 100°C.	3 × 10⁻⁶	1.06 ± 0.04

Results of these tests were highly promising, not only because of the very small weight losses noted, but more significantly because the volatiles were noncondensable at ambient temperature, even when the specimens were heated at 100°C. Consequently the resins tested appear suitable for aerospace applications.

Ethylene oxide–Freon sterilization tests were also carried out and were in accordance with Voyager spacecraft specifications.[11] Samples of both filled and unfilled resin were tested. The resin in both was the phenyltris-(ethylamino)silane-cured Epon X-24 with the filled specimen containing silica microballoons (Eccospheres SI, approximately 20% by weight). Surfaces of both specimens were softened by the sterilization conditions but they rehardened when removed from the sterilizing environment. Whether this was due to physical absorption or due to chemical attack was not established. Both processes are feasible. For example, an attack by ethylene oxide on the Si—N bond might proceed as shown in eq. (3).

$$CH_2\!\!-\!\!CH_2 + -\!\!\overset{|}{\underset{|}{Si}}\!\!-\!\!\overset{|}{N}\!\!- \rightarrow -\!\!\overset{|}{\underset{|}{Si}}\!\!-\!\!O\!\!-\!\!CH_2\!\!-\!\!CH_2\!\!-\!\!\overset{|}{N}\!\!- \qquad (3)$$

However, the subsequent loss in weight observed when the exposed pure resin was heated in the second part of the test essentially negates the possibility that a chemical reaction has occurred. In all probability, therefore, the changes were due to physical sorption only. Results of these tests are shown in Table IV.

TABLE IV
Results of Sterilization Tests on Cured Epoxysilazanes

Sample number	Test conditions[a]	Weight, g.	Weight change, %	Volume, cc.	Volume change, %
C2425-79-3	—	0.8464	—	0.69	—
(unfilled)	A	0.8720	+3.0	0.71	+2.9
	B	0.8351	−1.3	0.75	+8.7
C2425-79-3F	—	0.7245	—	0.86	—
(filled)	A	0.8178	+12.9	—	—
	B	0.7180	−0.9	—	—

[a] Test condition A: samples carried through six 24-hr. cycles at 23°C. in an atmosphere of 12% ethylene oxide and 88% Freon 12 (difluorodichloromethane) humidified to 50% R.H.; test condition B: specimens heated at 135°C. for 200 hr. under dry nitrogen upon completion of the ethylene oxide–Freon exposure.
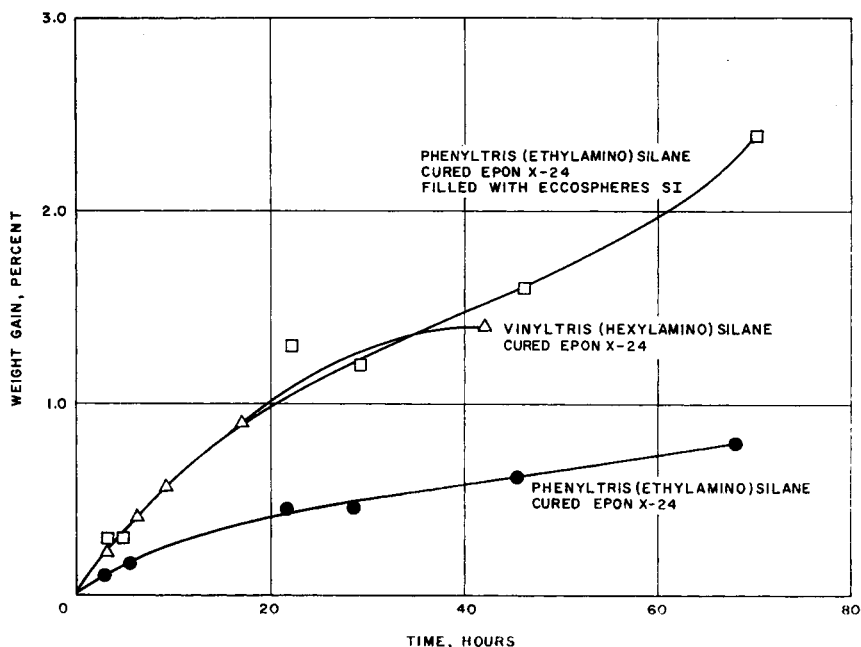

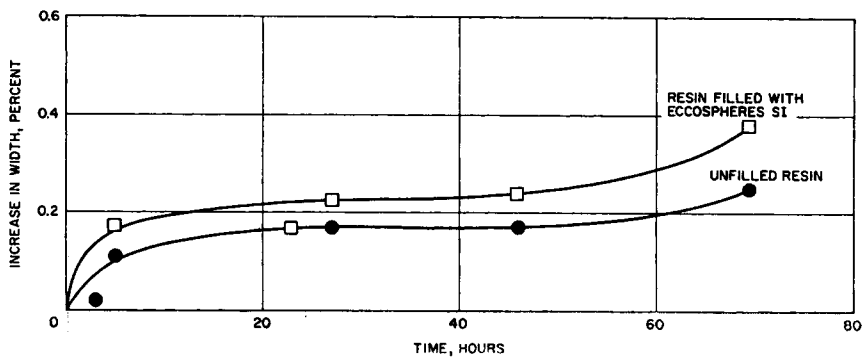Fig. 2. Effect of water on silazane-cured epoxy resins (total immersion at 24°C.).

Fig. 3. Effect of water on phenyltris(ethylamino)silane-cured Epon X-24 (total immersion at 24°C.).
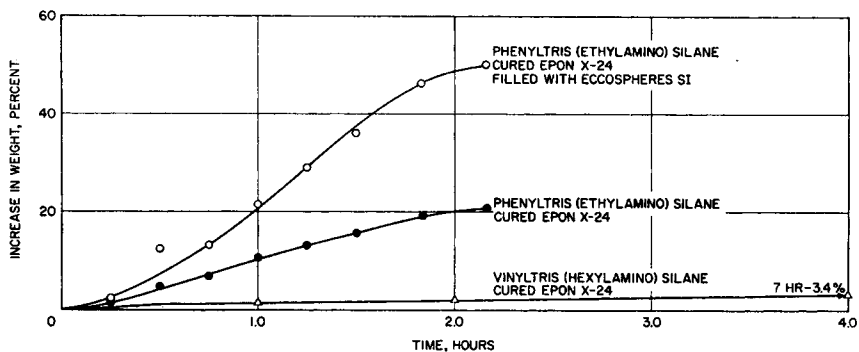
Fig. 4. Water absorption of silazane-cured Epon X-24 resins (total immersion at 100°C.).
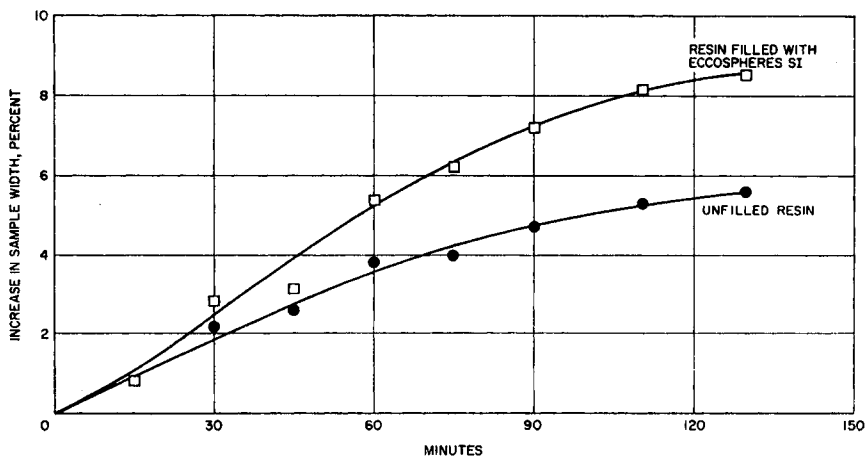
Fig. 5. Effect of water on dimensional stability of phenyltris(ethylamino)silane-cured Epon X-24 (total immersion at 100°C.).

It is quite apparent that the microballoon-filled specimen was somewhat more susceptible to ethylene oxide–Freon absorption than was the unfilled sample. This effect can be attributed to the higher degree of porosity of the filled specimen and can probably be remedied by the use of less than the maximum amount of filler.

A study has also been made on the effect of water on the silazane-cured Epon X-24 resins. Specimens used in these tests were approximately $^1/_8 \times \ ^1/_4 \times \ ^1/_2$ in. Moisture resistance was far better than anticipated both from the standpoint of dimensional stability and weight. With total immersion at ambient temperature the phenyltris(ethylamino)silane-cured samples, both filled and unfilled, swelled about 0.1–0.2% and then showed no significant further change for about 50 hr. Weight gains in this period were below 1% for the phenyltris(ethylamino)silane-cured sample and below 1.6% for the vinyltris(hexylamino)silane-cured sample.

With total submersion at 100°C., the vinyltris(hexylamino)silane-cured resin was highly superior to the phenyltris(ethylamino)silane-cured resin in that the former increased in weight by only 3.4% in 7 hr., whereas the latter gained 20% in weight in 2 hr. It was also noted that the silica microballoon-filled specimens were inferior in all cases to the corresponding unfilled resins. This can probably be attributed to a high degree of porosity. Moisture resistance of the filled samples thus probably can be improved markedly if less than the maximum amount of filler is incorporated.

Data showing the effect of water on these resins are presented in graphical form in Figures 2–5.

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Résumé

On a synthétisé le phényltris(méthylamino)silane, phényltris(éthylamino)silane et vinyltris(hexylamino)silane et ils ont été évalués comme agents de traitement de résines

époxy.   Les résines traitées avaient des propriétés physiques et diélectriques très bonnes. La résistance à l'humidité évaluée de ces résines était particulièrement bonne pour celles dérivées du vinyltris(hexylamino)silane. Les propriétés diélectriques typiques d'un échantillon époxy traité au phényltris(éthylamino)silane incluent une constante diélectrique de 3.7 à 25°C. et un facteur de dissipation de 0.8%.   Des résultats à température élevée sont également présentés.

Zusammenfassung

Phenyltris(methylamino)silan,   Phenyltris(äthylamino)silan   und   Vinyltris(hexylamino)silan wurden synthetisiert und als Härtungsmittel für Epoxyharze erprobt. Davon abgeleitete gehärtete Harze besitzen sehr gute physikalische und dielektrische Eigenschaften.   Die Feuchtigkeitsbeständigkeit der von Vinyltris(hexylamino)silan abgeleiteten Harze war besonders gut.   Zu den typischen dielektrischen Eigenschaften einer mit Phenyltris(äthylamino)silan gehärteten Epoxyprobe gehören eine Dielektrizitätskonstante von 3,7 bei 25°C. und ein Dissipationsfaktor von 0.8 Prozent. Schliesslich werden Daten für erhöhte Temperatur angegeben.