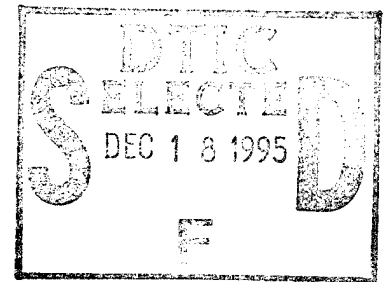


Development of alternate silicone potting compounds

Vol. 7. Development of secondary alternates for Dow Corning's Aerospace Sealant, DC93-120

William E. Cady
Albert T. Buckner



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GLOSSARY

Accelerator (E-244-69) (XCF3-6559), now QCF3-6559	Dow Corning concentrate of platinum complex in silicone polymer
Brookfield viscometers LVT, HBF, RVT	Models of viscometer made by Brookfield Engineering Laboratories Inc., Stoughton, MA 02072
Cabosil Grade MS75	Specific grade of silicone dioxide made by Cabot Corporation, Boston, MA 02110
DC93-120	Dow Corning silicone aerospace sealant
DC1107	Dow Corning methylhydrogen polysiloxane fluid
Dielectric gel Q3-6527	Dow Corning silicone electronic potting compound
Dow Corning	Dow Corning Corporation, Midland, MI 48640
DSC	Differential scanning calorimeter
GPC	Gel permeation chromatography
L84-89 A and B	Specific lot of Sylgard 184 used in the study
L86-58 A and B, L86-52A and B	Specific lots of Sylgard 186 used in the study
L1107-69	Specific lot of DC1107 fluid used in the study
LASL	Los Alamos Scientific Laboratory, Los Alamos, NM 87545
LLNL	Lawrence Livermore National Laboratory, Livermore, CA 94550
M_n	Number-average molecular weight
Mol-Rez gel timer	Mol-Rez gel-time meter Cat. No. VG-3050 made by Gardner Laboratory, Inc. Bethesda, MD 20014
M_w	Weight-average molecular weight
P75-1L236	Specific lot of Cabosil Grade MS75 used in this study
Pantex	Mason & Hanger-Silas Mason Co., Inc., Amarillo, TX 71905
PE-56	Specific lot of accelerator QCF3-6559 used in the study

PQ3-31 A and B	Specific lot of dielectric gel Q3-6527 used in the study
Shore A	Durometer Hardness Tester, Type A, made by Shore Instrument and Manufacturing Co., Jamaica, NY 11431
Sylgards 184 A and B, 186 A and B	Dow Corning silicone electronic potting compounds
Tg	Glass transition temperature
TGA	Thermal gravimetric analyzer
TMA	Thermal mechanical analyzer

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ABSTRACT

A substitute for Dow Corning DC93-120 silicone potting compound can be formulated from Sylgards 184 and 186 resins and either accelerator QCF3-6559 or DC1107 fluid. Substitutes can also be formulated from Sylgard 186, dielectric gel Q3-6527, and DC1107 or from Sylgard 184, silicone dioxide, and accelerator QCF3-6559.

INTRODUCTION

Because Dow Corning Corporation stopped making the silicone potting compounds that we were using in war-reserve production, we had to develop alternate materials for the future needs of LLNL and LASL. We have a cooperative program¹ between LLNL and Pantex (PX) to develop such materials. Our search for alternate compounds centers on Dow Corning's commercially available silicone potting compounds used in relatively large volumes for electronic encapsulation. These compounds are based on polymers, platinum catalysts, and silane curing agents similar to those in the potting compounds we were using.

The primary substitute that we developed for DC93-120 is based on a mixture of Sylgard 184 and 186 resins and accelerator QCF3-6559.² This substitute has not yet been used in production but could potentially be a problem because formulations with large amounts of Sylgard 184 can have entrapped bubbles. It is postulated that the silicate resin used to reinforce 184 creates a high surface tension in 184 that causes bubbles that do not break.

Sylgard 186 is reinforced with treated particulate silicone dioxide and has a lower surface tension than does Sylgard 184. Sylgard 186 can be

thinned with dielectric gel Q3-6527 and accelerated with QCF3-6559. From other phases of the program we found out that 186 with a significant quantity of Q3 could not be accelerated to our goal of a gel time of 90 minutes by QCF-3 alone, so we added DC1107 fluid. DC1107 is a highly reactive methyl hydrogen polysiloxane fluid that supplies additional hydrogen for reactions with vinyl groups and accelerates the reaction.

As an extension of the use of DC1107, we replaced all of the accelerator with 1107 in the 186 and Q3 system. Also, we replaced the accelerator with 1107 in the 184 and 186 system.

As a final attempt to formulate an alternate for DC93-120 we thickened 184 with silicon dioxide, Cabosil Grade MS75, and accelerated the mixture with accelerator QCF3-3.

This report deals with LLNL efforts to develop other alternate DC93-120 formulations based on:

1. Sylgard 186, Q3, and both accelerator and DC1107.
2. Sylgard 186, Q3, and DC1107.
3. Sylgard 184, Sylgard 186, and DC1107.
4. Sylgard 184, silicon dioxide, and accelerator.

CHARACTERISTICS OF THE PRIMARY ALTERNATE FOR DC93-120

The primary alternate is based on Sylgard 186 diluted with Sylgard 184 and accelerated with QCF-3 to a viscosity of 10 ± 0.8 Pa·s and a gel time of 90 ± 30 min at $25 \pm 1^\circ\text{C}$. This intentionally gels about 40 min faster than the DC93-120 it replaces. This alternate has a representative composition as follows:

Component	Parts by weight
Sylgard 186 A	60
B	6
Sylgard 184 A	40
B	4
Accelerator QCF3-6559	6

This is combination 1a of Ref. 2 and is based on 186, lot L86-52, and 184, lot L84-89. Its typical properties are listed in Table 1.

TABLE 1. Typical properties of DC93-120 substitute based on L86-52 and L84-89.

Zero-time viscosity,^a Pa·s	
at 20°C	6.5
25°C	6.8
30°C	7.0
Initial viscosity,^a Pa·s	
at 20°C	9.68
25°C	10.00
30°C	10.40
Time to double initial viscosity,^b min	
at 20°C	30
25°C	25
30°C	22
Gel time, min	
at 20°C	111
25°C	76
30°C	58
Hardness, Shore A	
After 6-h cure at $22 \pm 5^\circ\text{C}$	31.4
After 24-h cure at $22 \pm 5^\circ\text{C}$	37.2
Ultimate	40.0
After 2-y cure at $22 \pm 5^\circ\text{C}$	40.4
Weeks to reach ultimate hardness	
	11
Extractables, wt%	
After 3-d cure at $22 \pm 5^\circ\text{C}$	4.64
After ≥ 30 -d cure at $22 \pm 5^\circ\text{C}$	4.54

^aBrookfield viscometer LVT spindle 3 at 3 rpm.

^bElapsed time from initial viscosity to $2 \times$ initial viscosity.

GOALS

The goals for the alternates for DC93-120 are to reduce or eliminate the potential problem of trapping bubbles in assembly; to compare DC1107 and accelerator as the means of accelerating the cure rate of Sylgard 186 diluted with Sylgard 184 and

also 186 diluted with dielectric gel, Q3; and to evaluate the use of silicon dioxide to thicken Sylgard 184 and accelerator and compare this blend with the other formulations.

PROGRAMS TO DEVELOP SECONDARY ALTERNATES FOR DC93-120

The four LLNL programs are based on the following materials:

- Sylgard 186 resin and curing agent, lots L86-52A and -52B and L86-58A and -58B.

- Sylgard 184 resin and curing agent, lot L84-89A and -89B.

- Dielectric Gel Q3-6527 parts A and B, lot PQ3-31A and -31B.

- Accelerator QCF3-6559, lot PE-56.
- DC1107 fluid, lot L1107-69.
- Silicon dioxide, Cabosil Grade MS75, lot P75-1L236.

In Part X, Appendix A, of the first volume of this series,¹ we described a plan to increase the viscosity of Sylgard 184 with silicon dioxide to a value of 10 Pa·s and to accelerate the reaction of 184, filled with silicon dioxide, with accelerator to achieve a gel time of 90 min. Part X was assigned second priority and was the last part of the program to be done because we anticipated difficulty in achieving a stable blend of 184 and silicon dioxide. The details of the Part X program are as follows:

A. Develop 120 equivalent No. 2.

Note: Incorporate silicon dioxide into the resin component A by premixing in a mixer below 100°C, then milling the premix on a three-roll mill.

1. Establish how adding silicon dioxide in 5 increments to catalyzed 184 affects the initial viscosity of the blend at $25 \pm 1^\circ\text{C}$ over the range of 6 to 14 Pa·s, with LVT viscometer spindle 3 at 3 rpm.
2. Blend the formulation to give 10.0 ± 1 Pa·s.
 - a. Measure viscosity/time curve and gel time at $25 \pm 1^\circ\text{C}$, LVT spindle 3 at 3 rpm,
 - b. Measure hardness at 24 h at $22 \pm 5^\circ\text{C}$.
3. For 10 Pa·s blend, establish the effect of adding accelerator (in 4 increments of 4, 6, 8, and 10 parts per 100 of resin) to catalyzed resin on
 - a. Viscosity with time at $25 \pm 1^\circ\text{C}$, LVT spindle 3 at 3 rpm,
 - b. Gel time at $25 \pm 1^\circ\text{C}$,
 - c. Hardness at 6 and 24 h at $22 \pm 5^\circ\text{C}$.
4. Formulate silicon dioxide and accelerator in catalyzed 184 to give 10.0 ± 0.8 Pa·s and to give a gel time of 90 ± 30 min at $25 \pm 1^\circ\text{C}$.
 - a. Establish viscosity/time curve, gel time, and snap time at $20 \pm 1^\circ\text{C}$, $25 \pm 1^\circ\text{C}$, and $30 \pm 1^\circ\text{C}$, LVT spindle 3 at 3 rpm,
 - b. Measure hardness as a function of time to a constant value at $22 \pm 5^\circ\text{C}$,

- c. Check extractables after 3-d cure and after ≥ 1 -mo cure at $22 \pm 5^\circ\text{C}$.

d. —

e. —

B. —

C. Characterize 120 substitute No. 2 cured for at least 1 mo at $22 \pm 5^\circ\text{C}$.

1. TGA analysis,
2. DSC analysis,
3. Mechanical spectrometer analysis,
4. TMA analysis.

The purpose of Part XI of Appendix A¹ is to use Q3 to reduce the viscosity of Sylgard 186 to a value of 10 Pa·s and to accelerate the reaction of the blend to a gel time of 90 min. As happened in Part XIII,¹ we could not achieve a suitable gel time with accelerator only, so we had to add DC1107 also (Part XI 3'). The details of the Part XI program are as follows:

A. Develop 120 substitute No. 3.

1. Establish how adding 5 increments of gel diluent to catalyzed 186 affects the initial viscosity of the blend at $25 \pm 1^\circ\text{C}$, in the range of 8 to 12 Pa·s, LVT spindle 3 at 3 rpm.
2. Blend the formulation to give 10.0 ± 0.8 Pa·s.
 - a. Measure viscosity with time, gel time, and snap time at $25 \pm 1^\circ\text{C}$, LVT spindle 3 at 3 rpm,
 - b. Measure hardness at 24 h at $22 \pm 5^\circ\text{C}$.
3. For a catalyzed blend of 10 Pa·s, establish how adding accelerator (in 4 increments of 4, 6, 8, and 10 parts per 100 of 186 resin plus gel diluent part A) affects:
 - a. Viscosity with time at $25 \pm 1^\circ\text{C}$, LVT spindle 3 at 3 rpm,
 - b. Gel time and snap time at $25 \pm 1^\circ\text{C}$,
 - c. Hardness at 6 and 24 h at $22 \pm 5^\circ\text{C}$.
- 3'. For a catalyzed blend of about 10 Pa·s that contains 4 parts of accelerator, establish effects of adding increments of active hydrogen concentrate on
 - a. Initial viscosity at $25 \pm 1^\circ\text{C}$, LVT spindle 3 at 3 rpm,
 - b. Gel time at $25 \pm 1^\circ\text{C}$.

4. Add gel diluent parts A and B, accelerator, and DC1107 to catalyzed 186 to give 10.0 ± 0.8 Pa·s and to give a gel time of 90 ± 30 min at $25 \pm 1^\circ\text{C}$.
 - a. Establish viscosity/time curve, gel time, and snap time at $20 \pm 1^\circ\text{C}$, $25 \pm 1^\circ\text{C}$, and $30 \pm 1^\circ\text{C}$, LVT spindle 3 at 3 rpm,
 - b. Measure hardness with time to constant value at $22 \pm 5^\circ\text{C}$,
 - c. Check extractables after 3-d and ≥ 1 -mo cure at $22 \pm 5^\circ\text{C}$.
 - d. —
 - e. —
 - f. —

- B. Characterize 120 substitute No. 3 cured at least 1 mo at $22 \pm 5^\circ\text{C}$ by
 1. —
 2. TGA analysis,
 3. TMA analysis,
 4. DSC analysis,
 5. Mechanical spectrometer analysis.

The aim of Part XV, which was added to the original program, is simply to substitute DC1107 fluid for accelerator in a standard formulation of Sylgard 186 and Sylgard 184 from Part VI and to adjust the DC1107 to meet the 90-min gel time and the initial viscosity of 10 Pa·s. Details of the Part XV program are:

- A. Develop 120 substitute No. 4
 1. With the formulation

L84-89A	40 parts by weight,
B	4 parts by weight,
L86-52A	60 parts by weight,
B	6 parts by weight,

 establish the effect of adding L1107 (in 3 increments of 0.10, 0.13, and 0.16 parts per 100 of L84-89A + L86-52A) on
 - a. Viscosity with time, gel time, and initial snap time at $25 \pm 1^\circ\text{C}$, approximately 100-g samples, LVT spindle 3 at 3 rpm,
 - b. Hardness at 24 h at $22 \pm 5^\circ\text{C}$.
 2. Select a formulation to give an initial viscosity of 10.0 ± 1 Pa·s and to give a gel time of 90 ± 30 min at $25 \pm 1^\circ\text{C}$.
 - a. Establish viscosity/time curve and gel time at $20 \pm 1^\circ\text{C}$, $25 \pm 1^\circ\text{C}$, and $30 \pm 1^\circ\text{C}$, approximately 300-g sample, LVT spindle 3 at 3 rpm,

1°C , and $30 \pm 1^\circ\text{C}$, approximately 300-g sample, LVT spindle 3 at 3 rpm,

- b. Measure hardness as a function of time at $22 \pm 5^\circ\text{C}$ to a constant value,
- c. Measure extractables after 3-d and ≥ 1 -mo cure at $22 \pm 5^\circ\text{C}$.

- B. Characterize the 120 substitute No.4 cured at least 1 mo at $22 \pm 5^\circ\text{C}$ by
 1. TGA analysis,
 2. TMA analysis,
 3. DSC analysis,
 4. Mechanical spectrometer analysis.

The purpose of Part VXI, which was added to the original program, was to eliminate the accelerator and substitute DC1107 in a formulation selected from Part XI so as to meet these goals: viscosity of 10 Pa·s and a gel time of 90 min. Details of the Part XVI program are as follows:

- A. Develop 120 substitute No. 5
 1. With the following formulations from Part XI

L86-58A	70.6 parts by wt or wt%,
B	7.0 parts by wt or wt%,
PQ3-31A	11.2 parts by wt or wt%,
B	11.2 parts by wt or wt%,

establish the effect of adding L1107 in various increments up to 2.5 parts per 100 of L86-58 plus PQ3-31 on:

- a. Initial viscosity, gel time at $25 \pm 1^\circ\text{C}$, about 100-g sample, LVT spindle 3 at 3 rpm,
- b. Hardness at 24 h at $22 \pm 5^\circ\text{C}$,
- c. Extractables after 3-d cure at $22 \pm 5^\circ\text{C}$.
2. Select a formulation to give an initial viscosity of 10.0 ± 1 Pa·s and to give a gel time of 90 ± 30 min at $25 \pm 1^\circ\text{C}$.
 - a. Measure extractables after 3-d cure at $22 \pm 5^\circ\text{C}$,
 - b. Establish viscosity/time curve and gel time at $20 \pm 1^\circ\text{C}$, $25 \pm 1^\circ\text{C}$, and $30 \pm 1^\circ\text{C}$, approximately 300-g sample, LVT spindle 3 at 3 rpm,
 - c. Measure hardness as a function of time at $22 \pm 5^\circ\text{C}$ to a constant value,
 - d. Measure extractables after ≥ 1 -mo cure at $22 \pm 5^\circ\text{C}$.

B. Characterize 120 substitute No. 5 cured at least 1 mo at $22 \pm 5^\circ\text{C}$ by

1. TGA analysis,

2. DSC analysis,

3. Mechanical spectrometer analysis,

4. TMA analysis.

DEFINITION OF STARTING MATERIALS

The major components in these alternates for DC93-120 are Sylgards 184 and 186 and dielectric gel Q3. In Parts VI and XV, Sylgard 184 is used to dilute 186. In Parts XI and XVI, Q3 is used to dilute 186. In Part X, silicon dioxide MS75 is used to thicken 184. In Part XV we used lot L86-52 and in Parts XI and XVI we used lot L86-58 of Sylgard 186. Both accelerator QCF3-6559 and DC1107 fluid were used either separately or together to accelerate the blends to the 90-min gel time. We used only one lot of 184, accelerator, DC1107, and silicon dioxide in these experiments.

The Sylgards 184 and 186 resin (A) and curing agent (B) components were used in their normal weight ratio of 10 parts of A to 1 part of B. The Q3 A and B components were used in their normal one-to-one weight ratio. The properties of all starting components are listed in Table 2 and the properties of the cured Sylgards and Q3 are listed in Table 3.

The two 186 lots are very similar; the most significant difference is in the viscosity of the resin and that is our intentional variable. Q3 has the highest platinum content, highest vinyl content, and the least amount of active hydrogen of the three resins. Sylgards 184 and 186 have about the same platinum content but, of the two, 186 has less vinyl and much less active hydrogen. The MS75 silicon dioxide we are using has a surface area of $249.2 \text{ cm}^2/\text{g}$. Its water content was originally 1.45 wt% but this is a highly variable quantity. Both lots of 186 react faster than does 184. L86-52 is slightly faster than L86-58 and has a lower extractables content. 184 cures harder than 186. Q3 doesn't cure to any hardness and reacts extremely slowly. It also has a very high extractables content and a very high swell ratio (about 7 for LQ3-37). All this indicates a low degree of cross-linking that is due to the deficiency of active hydrogen in Q3.

TABLE 2. Properties of starting materials.

Property	Sylgard 186				Sylgard 184		Q3-6527		QCF3-	DC1107 L1107-69
	L86-52		L86-58		L84-89		PQ3-31		6559 PE-56	
	A	B	A	B	A	B	A	B		
Platinum content, ppm	7.1	—	6.2	—	6.7	—	16.0	—	237	—
Vinyl content, wt% ^a	0.23	0.27	0.25	0.37	0.61	0.46	0.63	0.58	0.67	0
Active hydrogen content, wt%	—	0.092	—	0.093	—	0.440	—	0.016	—	1.60
Molecular weight by GPC ^b										
M _w	40,100 ^c	38,650	39,800 ^c	38,600	21,110	23,600	14,100	14,000	17,700	—
M _n	13,300	10,500	12,300	9,400	5,100	6,600	5,500	5,700	6,400	—
M _w /M _n	3.0	3.68	3.2	4.11	4.14	3.58	2.56	2.46	2.77	—
Viscosity ^d at $25 \pm 1^\circ\text{C}$, Pa·s										
LVT spindle 4 at 60 rpm	—	—	—	—	5.55	—	—	—	—	—
LVT spindle 1 at 30 rpm	—	—	—	—	—	0.089	—	—	—	—
RVT spindle 7 at 10 rpm	84.0	—	100.0	—	—	—	—	—	—	—
LVT spindle 3 at 12 rpm	—	1.120	—	1.080	—	—	—	—	—	—
LVT spindle 2 at 60 rpm	—	—	—	—	—	—	0.243	0.249	—	—
LVT spindle 3 at 60 rpm	—	—	—	—	—	—	—	—	0.364	0.046

^aPantex measurements.

^bBased on silicone standards.

^cSecond mode data.

^dBrookfield viscometers.

TABLE 3. Properties of starting materials blended in their standard proportions.

Property	L86-52 A & B blended in a 10:1 weight ratio	L86-58 A & B blended in a 10:1 weight ratio	L84-89 A & B blended in a 10:1 weight ratio	PQ3-31 A & B ^a blended in a 1:1 weight ratio
Initial viscosity, Pa·s	38.7 ^b	39.2 ^b	3.45 ^c	0.294 ^d
at 20°C	39.7	42.4	3.34	0.158
25°C	36.0	43.5	3.23	0.250
30°C				
Time to double initial viscosity, ^e min				
at 20°C	158	164	170	973
25°C	131	145	150	377
30°C	71	86	112	230
Gel time, min				
at 20°C	279	320	426	1400
25°C	262	293	357	982
30°C	153	178	243	528
Ultimate hardness, Shore A at 22 ± 5°C	36.8	35.2	43.0	—
Extractables, wt%				
After 3-d cure at 22 ± 5°C	3.98	5.44	5.63	24.9
After ≥1-mo cure at 22 ± 5°C	4.11	5.05	5.59	68.3
Equilibrium swell ratio in chlorobenzene	2.634	2.673	1.941	—

^aPantex measurements.

^bBrookfield viscometer HBF spindle 4 at 10 rpm.

^cBrookfield viscometer LVT spindle 3 at 12 rpm.

^dBrookfield viscometer LVT spindle 2 at 30 rpm.

^eElapsed time from initial viscosity to 2 × initial viscosity.

EXPERIMENTAL RESULTS

Our results are based on programs conducted using Parts X, XI, XV, and XVI as outlined above.

PART X, A.1

To determine the effect of silicon dioxide in increasing the viscosity of 184, we added MS75 (as received) to catalyzed L84-89 in increments of 0.5, 2.0, 4.0, and 6.0 grams MS75 per 100 grams of L84-89A. We then measured the initial viscosity of the blend at 25 ± 1°C. Results are plotted in Fig. 1.

PART X, A.2 and A.3

From Fig. 1, we selected the following composition and measured its properties as shown in Tables 4 and 5.

Component	Parts by weight
L84-89A	100
L84-89B	10
Silicon dioxide	5.6

To this formulation, we added PE56 in increments of 4, 6, 8, and 10 parts per 100 parts of L84-89A by weight. We measured viscosity with time and gel time at 25 ± 1°C and hardness at 6 and 24 hours cure at 22 ± 5°C. The viscosity-time data are tabulated in Table 4 and the other properties are in Table 5. Time to double initial viscosities and gel times are plotted in Fig. 2 and viscosity-time data are plotted in Fig. 3.

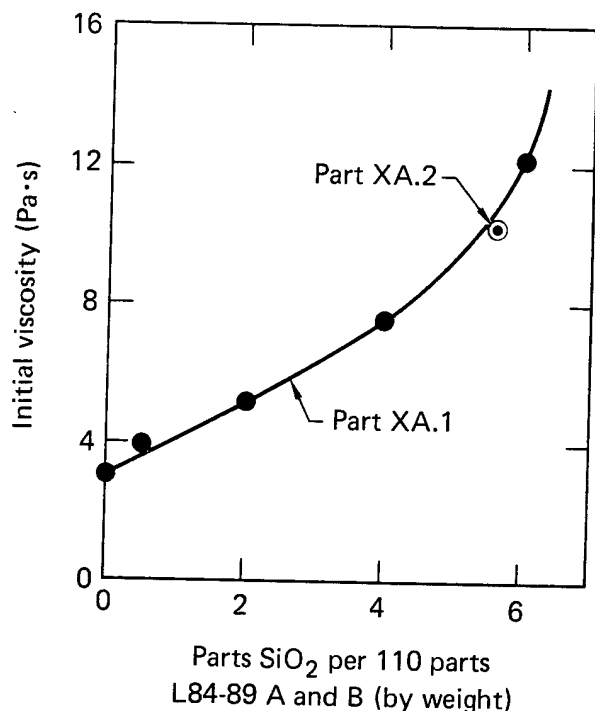


FIG. 1. Effect of silicon dioxide on the initial viscosity at $25 \pm 1^\circ\text{C}$ of catalyzed L84-89 for Part X A.1 and X A.2, LVT spindle 3 at 3 rpm.

PART X, A.4

From the data on Part X, A.1, A.2, and A.3 we selected the following composition as our representative formulation:

Component	Parts by weight	Wt%
L84-89A	100	82.92
L84-89B	10	8.29
PE56	5	4.15
Silicon dioxide	5.6	4.64

For this formulation we established the viscosity with time at $20 \pm 1^\circ\text{C}$, $25 \pm 1^\circ\text{C}$, and $30 \pm 1^\circ\text{C}$. We also measured hardness with time and extractables after 3-d cure and after ≥ 1 -mo cure at $22 \pm 5^\circ\text{C}$. The viscosity/time data are listed in Table 6 and plotted in Fig. 4. The other data are shown in Table 7.

PART XI, A.1

To determine the effect of Q3 in reducing the viscosity of 186, we added catalyzed PQ3-31 to catalyzed L86-58 in 13 increments and measured the initial viscosity at $25 \pm 1^\circ\text{C}$. This was done in connection with Part XIII, so we covered the range of

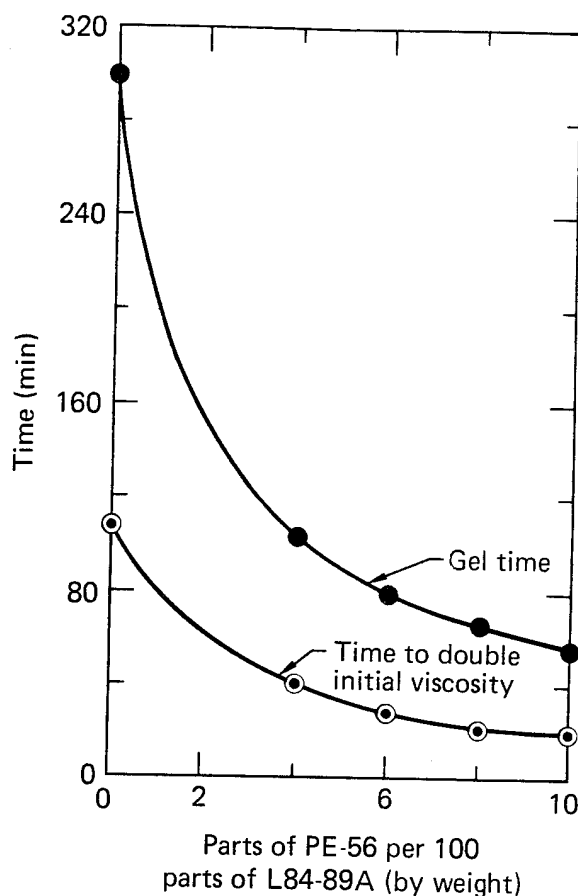


FIG. 2. Effect of PE-56 on the reaction rate at $25 \pm 1^\circ\text{C}$ of a blend of L84-89 A and B and silicon dioxide for Part X A.2 and X A.3, LVT spindle 3 at 3 rpm.

10 to 98 wt% of Q3 in the blend of Q3 and 186. We measured the viscosity with the LVT viscometer and spindle 3, but measured at various spindle speeds. All results are tabulated in Table 8 and some are plotted in Figure 5. As the data show, viscosity is a function of spindle speed and one speed usually cannot be used over a broad viscosity range. The data at 3 rpm are of interest to Part XI.

PART XI, A.2 and A.3

From Fig. 5, we selected the following composition and measured its properties as shown in Table 9 (Part A.2).

Component	Parts by wt or wt%
L86-58A	70.0
PQ3-31A	11.5
L86-58B	7.0
PQ3-31B	11.5

TABLE 4. Viscosity vs time data for blends of L84-89, A and B, and silicon dioxide with varying amounts of PE-56 for Part X A.

Time from start of mixing, min	Viscosity ^a at 25 ± 1°C, Pa·s				
	Parts of PE-56 per 100 parts of L84-89 A by weight				
	0	4	6	8	10
10	10.32	9.96	10.08	10.20	8.92
13	10.60	10.56	10.76	11.04	9.68
16	10.80	11.08	11.28	11.84	10.48
19	10.92	11.52	11.96	12.92	11.44
22	11.12	12.00	12.64	14.04	12.56
25	11.36	12.56	13.44	—	13.96
26	—	—	—	16.04	—
30	11.64	13.68	15.24	18.56	17.08
32	—	—	—	20.04	18.76
34	—	—	—	22.00	—
35	—	—	17.40	—	—
40	12.28	—	20.12	—	—
41	—	—	20.72	—	—
42	—	17.64	—	—	—
45	—	18.84	—	—	—
50	12.84	21.16	—	—	—
60	13.48	—	—	—	—
70	14.32	—	—	—	—
85	15.48	—	—	—	—
90	15.96	—	—	—	—
100	16.96	—	—	—	—
125	20.08	—	—	—	—
130	20.80	—	—	—	—

^aBrookfield viscometer LVT spindle 3 at 3 rpm.

TABLE 5. Properties of blends of L84-89, A and B, and silicon dioxide unaccelerated and with varying amounts of PE-56 for Parts X A.2 and A.3.

Property	Parts of PE-56 per 100 parts of L84-89 by weight				
	0	4	6	8	10
Initial viscosity ^a at 25 ± 1°C, Pa·s	10.32	9.96	10.08	10.20	8.92
Time to double initial viscosity ^b at 25 ± 1°C, min	109	40	30	22	21
Gel time at 25 ± 1°C, min	300	104	80	66	58
Hardness, Shore A after 6-h cure at 22 ± 5°C	—	25.0	—	42	45.0
24-h cure at 22 ± 5°C	47.6	47.4	50	49.6	50.0

^aBrookfield viscometer LVT spindle 3 at 3 rpm.

^bElapsed time from initial viscosity to 2 × initial viscosity.

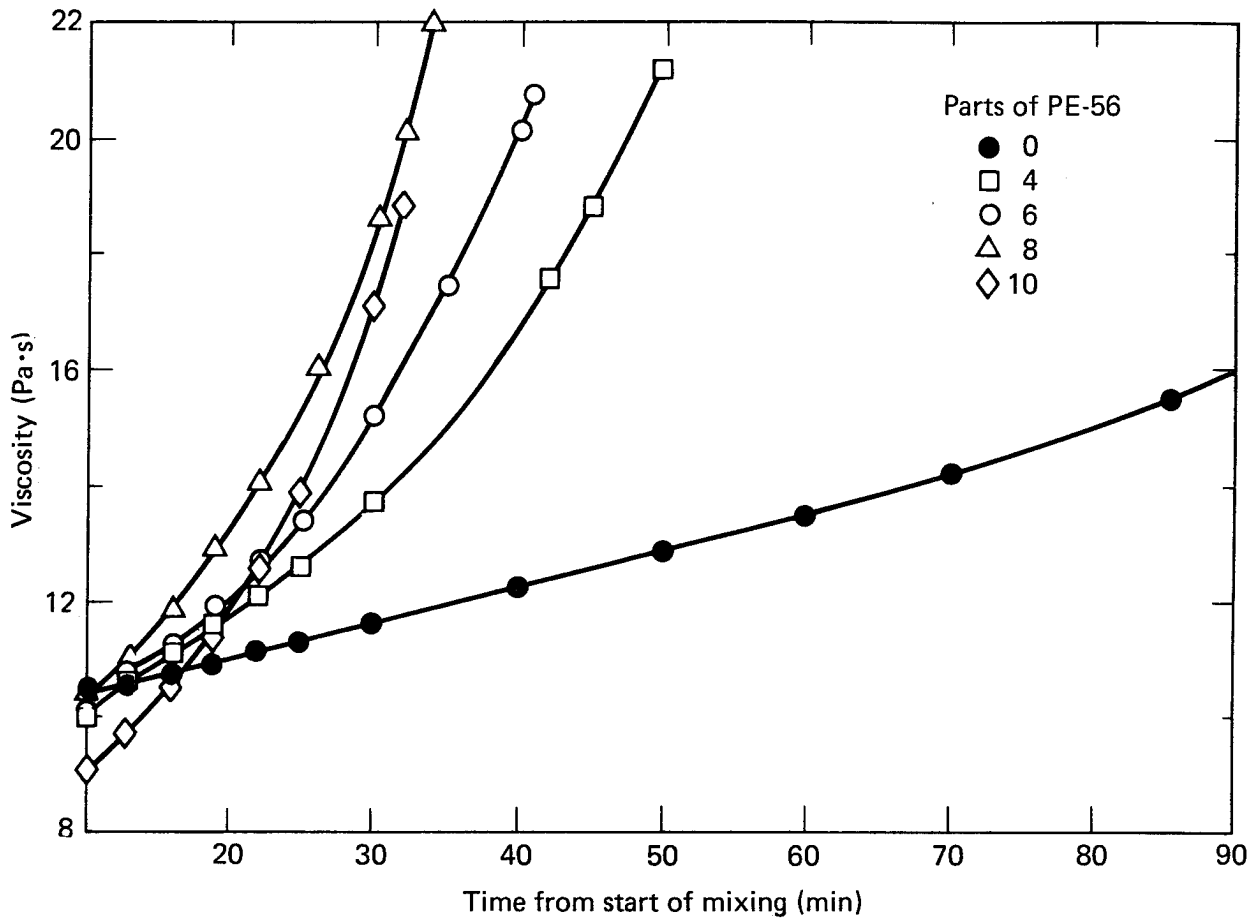


FIG. 3. Viscosity-time curves, at $25 \pm 1^\circ\text{C}$, for a blend of L84-89 A and B and silicon dioxide with varying amounts of PE-56, LVT spindle 3 at 3 rpm (Part X A.3).

To this formulation, we then added PE56 in increments of 4,6,8, and 10 parts by weight and measured viscosity with time, gel time, and snap time at $25 \pm 1^\circ\text{C}$ and hardness at 24 h at $22 \pm 5^\circ\text{C}$ (Part A.3). The data are listed in Table 9. There was no measurable hardness at 24-h cure. A gel time of 90 min cannot be achieved with the use of accelerator only. It might be possible that, with 8 to 10 parts of PE56 and a decrease in PQ3-31 to get the initial viscosity back up to 10, the gel time would speed up to 90 min. We did not take this approach. Instead, we added DC1107 fluid as in Part XIII.

TABLE 6. Viscosity vs time data for the representative blend of Part X A.4 at three temperatures.

Time from start of mixing, min	Viscosity, ^a Pa·s at		
	$20 \pm 1^\circ\text{C}$	$25 \pm 1^\circ\text{C}$	$30 \pm 1^\circ\text{C}$
10	9.52	9.60	10.24
13	10.36	10.44	11.12
16	10.92	11.20	11.76
19	11.48	12.00	12.72
22	11.96	12.76	13.68
25	12.24	13.88	14.64
30	—	15.44	16.64
32	13.64	—	—
35	—	—	19.64
40	—	21.24	23.88
42	15.80	—	—
50	18.16	—	—
53	19.04	—	—

^aBrookfield viscometer LVT spindle 3 at 3 rpm.

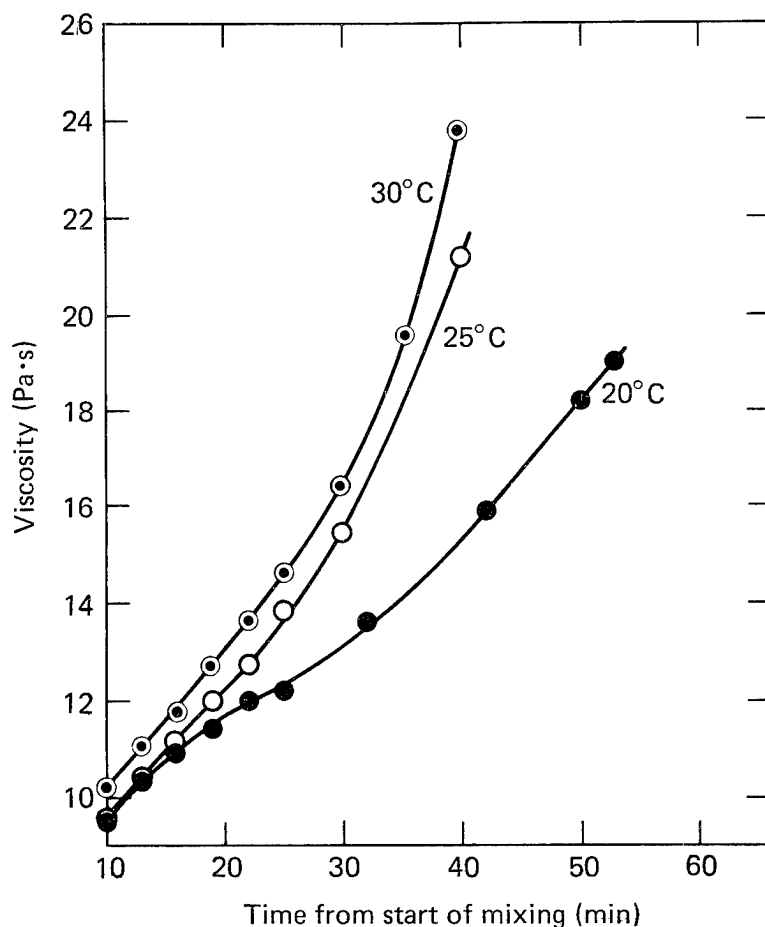


FIG. 4. Viscosity-time curves for the representative blend of Part X A.4 at 3 temperatures, LVT spindle 3 at 3 rpm.

TABLE 7. Properties of the representative blend of Part X A.4 at three temperatures.

Property	20 ±1°C	25 ±1°C	30 ±1°C
Initial viscosity, ^a Pa·s	9.52	9.60	10.24
Time to double initial viscosity, ^b min	43	27	26
Gel time, min	131	94	71
Hardness, Shore A			
After 24-h cure at 22 ±5°C	—	50.1	—
Ultimate	—	55.1	—
Time to reach ultimate, mo	—	1	—
Extractables, wt%			
After 3-d cure at 22 ±5°C	—	5.16	—
After >30-d cure at 22 ±5°C	—	5.12	—

^aBrookfield viscometer LVT spindle 3 at 3 rpm.

^bElapsed time from initial viscosity to 2 × initial viscosity.

PART XI, A.3'

From Part A.3 we selected the formulation with 4 parts of PE56 and added 0.10 part of L1107-69 per 100 parts of PQ3-31 and L86-58 by weight. We measured initial viscosity and gel time at 25 ± 1°C as shown in Table 10. The addition of only 0.10 part of L1107-69 is sufficient to bring the gel time down to our goal.

PART XI, A.4

To bring the initial viscosity within our desired goal, we slightly reduced the amount of Q3 from that used in A.3 and tested the following formulation:

Component	Parts by weight	Wt%
L86-58A	70.6	67.82
L86-58B	7.0	6.72
PQ3-31A	11.2	10.76
PQ3-31B	11.2	10.76
PE56	4.0	3.84
L1107-69	0.10	0.10

We measured viscosity as a function of time, snap time, and gel time at $20 \pm 1^\circ\text{C}$, $25 \pm 1^\circ\text{C}$, and $30 \pm 1^\circ\text{C}$. We measured hardness as a function of cure time at $22 \pm 5^\circ\text{C}$ and extractables after 3-d cure and ≥ 1 -mo cure at $22 \pm 5^\circ\text{C}$. Viscosity-time data are listed in Table 11 and plotted in Fig. 6. The other data are shown in Table 12. We have achieved a suitable initial viscosity and gel time but the extractables content is considerably higher than our maximum of 6 wt%.

PART XV, A.1

To determine how substituting DC1107 for accelerator QCF3-6559 affected the properties of a blend of Sylgard 186 diluted with Sylgard 184, we selected the following formulation of Part VI, which

is the same as combination 1A except for the absence of accelerator.

Component	Parts by weight
L86-52A	60
L86-52B	6
L84-89A	40
L84-89B	4

To this formulation we added L1107-69 in 5 increments and measured viscosity with time, gel time, and snap time at $25 \pm 1^\circ\text{C}$. We also measured hardness at 24 h at $22 \pm 5^\circ\text{C}$. The viscosity-time data are listed in Table 13 and the other data are in Table 14. We were able to achieve a gel time of 90 min with 0.32 part L1107-69, but the initial viscosity exceeded our goal of $10 \pm 0.8 \text{ Pa}\cdot\text{s}$. Therefore, we held the amount of L1107-69 at 0.32 part and increased the proportion of L84-89 to L86-52. The data are in Tables 15 and 16.

PART XV, A.2

We still could not select a formulation to meet our goals from the data generated in A.1, so we

TABLE 8. Effect of Q3 on the viscosity of Sylgard 186 for Parts XI A.1 and XIII A.1.

Blend by wt%		Initial viscosity ^a at $25 \pm 1^\circ\text{C}$, Pa·s.				
Q3	186	3 rpm	6 rpm	12 rpm	30 rpm	60 rpm
10	90	28.32	b	—	—	—
20	80	13.40	12.34	b	—	—
30	70	6.40	—	5.69	b	—
40	60	3.52	—	3.22	2.99	b
50	50	1.96	—	1.89	—	1.71
60	40	1.08	—	1.25	—	1.14
70	30	1.00	—	—	—	0.812
75	25	0.480	—	—	—	0.652
80	20	0.400	—	—	—	0.556
90	10	0.360	—	—	—	0.396
93	7	0.600	—	—	—	0.348
95	5	0.322	—	—	—	0.322
98	2	0.320	—	—	—	0.288

^aBrookfield viscometer LVT, spindle 3.

^bOff scale.

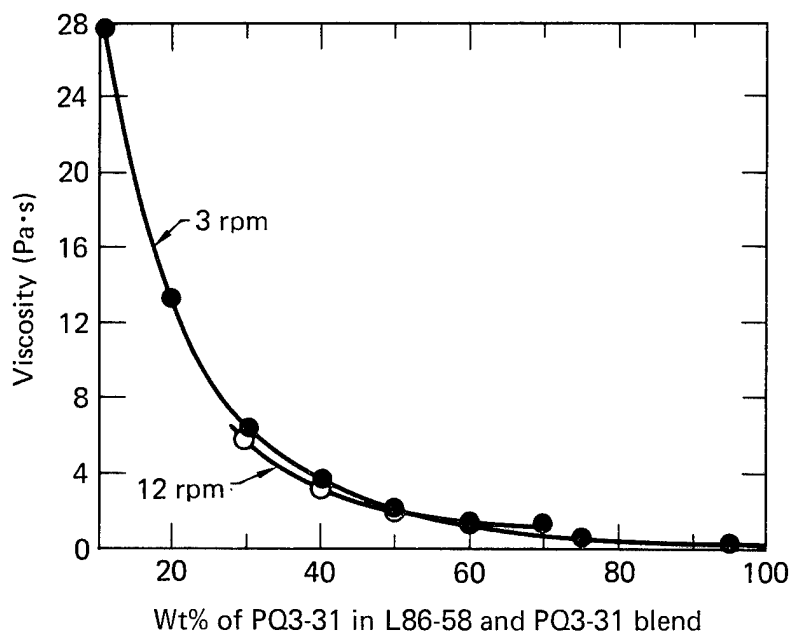


FIG. 5. Dilution effect of PQ3-31 on L86-58 at $25 \pm 1^\circ\text{C}$ for Parts XI A.1 and XII A.1, LVT spindle 3.

TABLE 9. Properties of blends of PQ3-31 (A and B) and L86-58 (A and B), unaccelerated and accelerated with varying amounts of PE-56 for Parts XI A.2 and A.3.

Property	Parts of PE-56 per 100 parts of PQ3-31A and B plus L-86-58 A and B, by weight				
	0	4	6	8	10
Initial viscosity ^a at $25 \pm 1^\circ\text{C}$ Pa·s	10.24	8.88	8.24	7.80	7.52
Time to double initial viscosity ^b at $25 \pm 1^\circ\text{C}$, min	—	79	59.5	54	54.5
Snap time at $25 \pm 1^\circ\text{C}$, min	375	155	—	115	122
Gel time at $25 \pm 1^\circ\text{C}$, min	443	199	148	126	131

^aBrookfield viscometer LVT spindle 3 at 3 rpm.

^bElapsed time from initial viscosity to $2 \times$ initial viscosity.

TABLE 10. Properties of a blend of PQ3-31 and L86-58 accelerated with 4 parts of PE-56, both with and without 0.10 part L1107-69 per 100 parts of Q3 and 186 by weight, for Parts XI A.3 and A.3'.

Property	Parts of L1107-69 per 100 parts of PQ3 and L86, by weight	
	0	0.10
Initial viscosity ^a at 25 ± 1°C, Pa·s	8.88	8.88
Gel time at 25 ± 1°C, min	199	85

^aBrookfield viscometer LVT spindle 3 at 3 rpm.

TABLE 11. Viscosity vs time data for the representative blend of Part XI A.4 at three temperatures.

Time from start of mixing, min	Viscosity, ^a Pa·s at		
	20 ± 1°C	25 ± 1°C	30 ± 1°C
10	10.36	9.72	11.08
13	11.68	11.20	12.32
16	12.68	12.60	—
17	—	—	14.04
19	13.52	13.92	15.00
22	14.32	15.16	16.52
25	15.00	16.00	18.40
28	—	17.04	—
30	16.16	—	22.48
31	—	18.48	—
34	—	19.72	—
35	—	—	—
36	17.52	—	—
40	18.48	—	—
50	20.84	—	—

^aBrookfield viscometer LVT spindle 3 at 3 rpm.

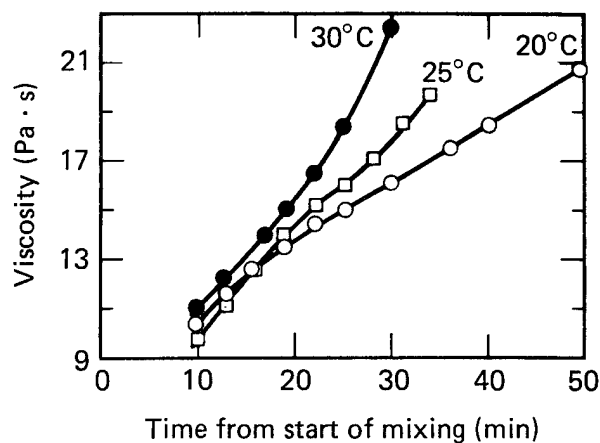


FIG. 6. Viscosity-time curves at 3 temperatures for the representative formulations of Part XI A.4, LVT spindle 3 at 3 rpm.

TABLE 12. Properties of the representative blend of Part XI A.4 at three temperatures.

Property	20 ± 1°C	25 ± 1°C	30 ± 1°C
Zero-time viscosity, ^a Pa·s	5.2	5.0	7.1
Initial viscosity, ^a Pa·s	10.36	9.72	11.08
Time to double initial viscosity, ^b min	39.5	23	19.5
Snap time, min	88	61	37
Gel time, min	151	97	62
Hardness, Shore A			
After 24-h cure at 22 ± 5°C	—	15.2	—
Ultimate	—	17.7	—
Extractables, wt%			
After 24-h cure at 22 ± 5°C	—	9.47	—
After >30-d cure at 22 ± 5°C	—	8.42	—

^aBrookfield viscometer LVT spindle 3 at 3 rpm.

^bElapsed time from initial viscosity to 2X initial viscosity.

TABLE 13. Viscosity vs time data for a 40:60 blend (by weight) of L84-89 and L86-52 accelerated with varying amounts of L1107-69 for Part XV A.1.

Time from start of mixing, min	Viscosity ^a at 25 ± 1°C, Pa·s					
	Parts of L1107-69:110 parts of L86-52 plus L84-89 by weight					
	0	0.10	0.13	0.16	0.25	0.32
10	10.84	11.68	12.88	13.16	13.56	13.64
13	—	12.28	13.64	14.20	14.84	15.00
16	—	13.40	14.28	15.16	15.92	16.12
19	—	—	14.92	15.96	—	17.24
20	—	14.32	—	—	—	—
22	—	14.68	15.60	16.80	18.04	18.52
25	—	15.32	16.28	17.60	19.28	19.96
30	—	—	17.52	19.12	21.48	22.52
40	—	—	19.92	22.24	26.60	29.24
42	—	—	—	—	27.92	—
45	—	—	23.60	23.92	—	—
49	—	18.28	—	—	—	—
50	—	18.40	26.00	25.68	—	—
52	—	—	—	26.64	—	—
60	—	20.76	—	—	—	—
70	—	23.36	—	—	—	—

^aBrookfield viscometer LVT spindle 3 at 3 rpm.

TABLE 15. Effect of varying the ratio (by weight) of L84-89 to L86-52 at 0.32 part of L1107-65 per 110 parts of L84-89 plus L86-52 on the properties of the blend for Part XV A.1.

Property	Ratio of L84-89A to L86-52A by weight		
	40:60	45:55	50:50
Zero-time viscosity ^a at 25 ± 1°C, Pa·s	9.2	8.7	8.4
Initial viscosity ^a at 25 ± 1°C, Pa·s	13.64	11.36	9.32
Time to double initial viscosity ^b at 25 ± 1°C, min	27	33	38
Gel time at 25 ± 1°C, min	90	110	122
Hardness, Shore A after 24-h cure at 22 ± 5°C	40	41.8	41.2

^aBrookfield viscometer LVT spindle 3 at 3 rpm.

^bElapsed time from initial viscosity to 2 × initial viscosity.

TABLE 14. Properties of a 40:60 blend (by weight) of L84-89 and L86-52 accelerated with varying amounts of L1107-69 for Part XV A.1.

Property	Parts of L1107-69:110 parts of L86-52 plus L84-89 (by weight)					
	0	0.10	0.13	0.16	0.25	0.32
Zero-time viscosity ^a at 25 ± 1°C, Pa·s	8.0	7.9	10.7	9.8	9.0	9.2
Initial viscosity ^a at 25 ± 1°C, Pa·s	10.84	11.68	12.88	13.16	13.56	13.64
Time to double initial viscosity ^b at 25 ± 1°C, min	87	50	50	41	31	27
Gel time at 25 ± 1°C, min	294	185	160	150	107	90
Snap time at 25 ± 1°C, min	356	133	125	111	79	68
Hardness, Shore A after 24-h cure at 22 ± 5°C	34.6	40.4	40.6	—	39.4	40.0

^aBrookfield viscometer LVT spindle 3 at 3 rpm.

^bElapsed time from initial viscosity to 2 × initial viscosity.

TABLE 16. Viscosity vs time data for various ratios of L84-89 to L86-52 accelerated by 0.32 part of L1107-69 per 110 parts of L84-89 and L86-52 at $25 \pm 1^\circ\text{C}$.

Time from start of mixing, min	Viscosity, ^a Pa·s		
	Ratio of L84-89 A to L86-52 by weight		
	40:60	45:55	50:50
10	13.64	11.36	9.32
13	15.00	12.12	9.68
16	16.12	12.88	10.24
19	17.24	13.68	10.88
22	18.52	14.56	—
23	—	—	11.64
25	19.96	15.24	12.00
30	22.52	17.08	12.20
40	29.24	21.20	16.04
45	—	23.76	—
50	—	—	19.60

^aBrookfield viscometer LVT spindle 3 at 3 rpm.

selected a different formulation to which to add L1107-69. It is,

Component	Parts by weight
L86-52A	50
L86-52B	5
L84-89A	50
L84-89B	5

To this we added L1107-69 in increments of 0.32, 0.35, 0.40, and 0.50 parts by weight per 100 parts of L86-52A plus L84-89A. We measured viscosity with time and gel time at $25 \pm 1^\circ\text{C}$ and hardness at 24-h cure at $22 \pm 5^\circ\text{C}$. The data are in Tables 17 and 18. The effects of L1107-69 on the reaction rates of two blends of L84-89 and L86-52 from Part XV, A.1 and A.2 are shown in Fig. 7.

From the data generated in Parts XV, A.1 and A.2, we selected the 50:50 blend (by weight) of L84-89 with L86-52 accelerated with 0.50 part by weight of L1107-69 as our representative formulation. We measured viscosity as a function of time and gel time at $20 \pm 1^\circ\text{C}$, $25 \pm 1^\circ\text{C}$, and $30 \pm 1^\circ\text{C}$. The viscosity-time data are shown in Table 19 and

TABLE 17. Viscosity vs time data for a 50:50 blend (by weight) of L84-89 and L86-52 accelerated with various amounts of L1107-65 for Part XV A.2.

Time from start of mixing, min	Viscosity ^a at $25 \pm 1^\circ\text{C}$, Pa·s			
	Parts of L1107-69 per 110 parts of L86-52 plus L84-89 by weight			
	0.32	0.35	0.40	0.50
10	9.32	9.64	9.64	10.12
13	9.68	10.08	10.24	10.88
16	10.24	10.40	11.00	11.56
19	10.88	10.88	11.64	12.44
22	—	11.56	12.24	13.56
23	11.64	—	—	—
25	12.00	12.20	12.84	14.76
30	12.20	13.16	14.20	16.92
40	16.04	15.76	17.60	23.24
45	—	—	19.48	—
50	19.60	19.44	—	—

^aBrookfield viscometer LVT spindle 3 at 3 rpm.

TABLE 18. Properties of 50:50 blends (by weight) of L84-89 and L86-52 accelerated with varying amounts of L1107-69 for Part XV A.2.

Property	Parts of L1107-69:110 parts of L84-89 plus L86-52 by weight			
	0.32	0.35	0.40	0.50
Zero-time viscosity ^a at $25 \pm 1^\circ\text{C}$, Pa·s	8.4	8.6	7.4	7.8
Initial viscosity ^a at $25 \pm 1^\circ\text{C}$, Pa·s	9.32	9.64	9.64	10.12
Time to double initial viscosity ^b at $25 \pm 1^\circ\text{C}$, min	38	40	34	26
Gel time at $25 \pm 1^\circ\text{C}$, min	122	119	112	82
Hardness, Shore A				
After 24-hour cure at $22 \pm 5^\circ\text{C}$	41.2	42	42	42

^aBrookfield viscometer LVT spindle 3 at 3 rpm.

^bElapsed time from initial viscosity to $2 \times$ initial viscosity.

Fig. 8. We measured hardness as a function of time at $22 \pm 5^\circ\text{C}$ and extractables after 3-d cure at $22 \pm 5^\circ\text{C}$. These data are listed in Table 20.

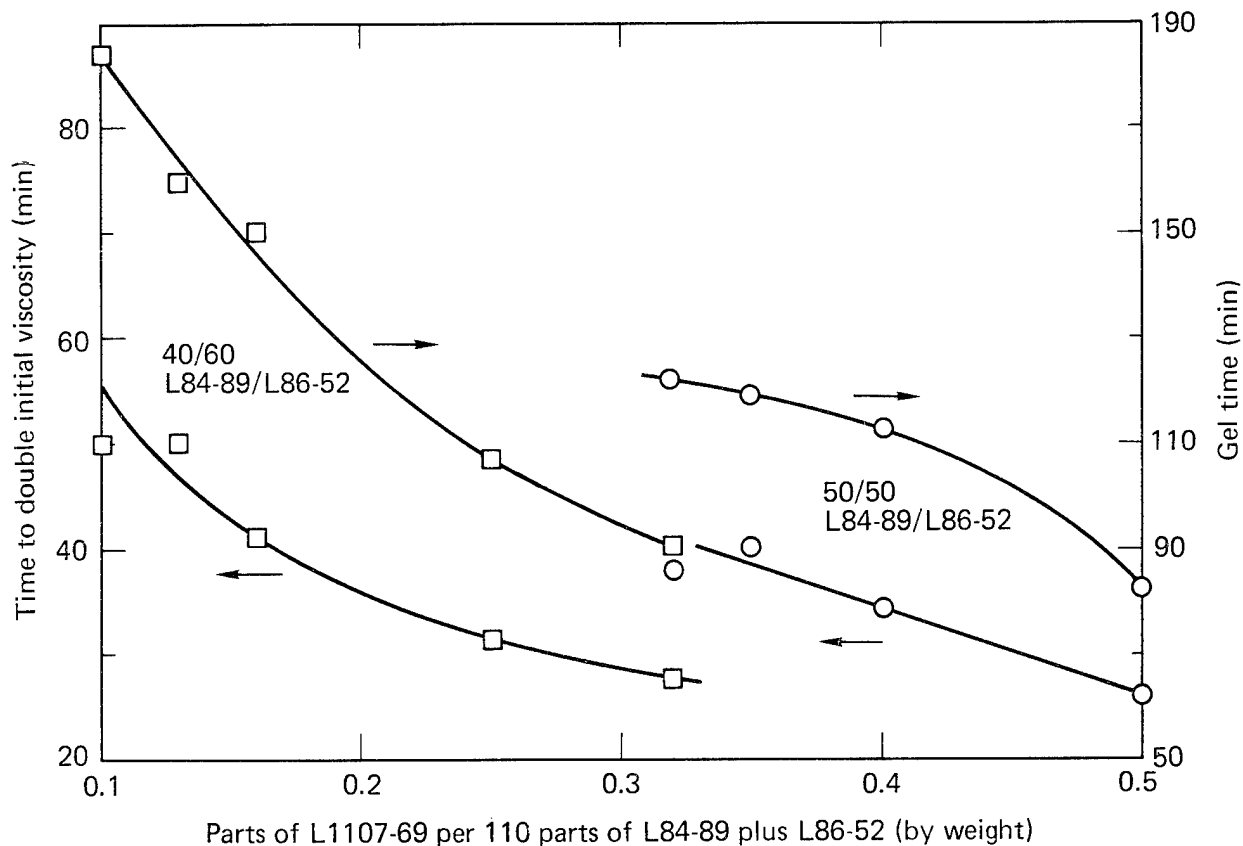


FIG. 7. Effect of L1107-69 on the reaction rates at $25 \pm 1^\circ\text{C}$ of 2 different blends of L84-89 and L86-52 for Parts XVI A.1 and XVI A.2, LVT spindle 3 at 3 rpm.

TABLE 19. Viscosity vs time data for the representative blend of Part XV A.2 at three temperatures.

Time from start of mixing, min	Viscosity, ^a Pa·s at		
	$20 \pm 1^\circ\text{C}$	$25 \pm 1^\circ\text{C}$	$30 \pm 1^\circ\text{C}$
10	10.00	9.64	10.40
13	10.64	10.20	11.44
16	11.52	11.36	12.44
19	—	12.20	13.40
21	12.84	—	—
22	13.04	13.04	14.56
25	13.84	14.00	16.04
30	—	15.72	—
32	—	—	20.80
34	16.68	—	—
35	—	17.76	23.96
40	18.84	20.36	—
44	20.32	—	—

^aBrookfield viscometer LVT spindle 3 at 3 rpm.

TABLE 20. Properties of the representative blend of Part XV A.2 at three temperatures.

Property	$20 \pm 1^\circ\text{C}$	$25 \pm 1^\circ\text{C}$	$30 \pm 1^\circ\text{C}$
Zero-time viscosity, ^a Pa·s	7.45	7.00	7.20
	—	7.80	—
Initial viscosity, ^a Pa·s	10.00	9.64	10.40
	—	10.12	—
Time to double initial viscosity, ^b min	33	28	22
	—	26	—
Gel time, min	138	94	65
	—	82	—
Hardness, Shore A after 24-h cure at $22 \pm 5^\circ\text{C}$	39.6	42	—
Extractables, wt% after 3-d cure at $22 \pm 5^\circ\text{C}$	—	4.73	—

^aBrookfield viscometer LVT spindle 3 at 3 rpm.

^bElapsed time from initial viscosity to $2 \times$ initial viscosity.

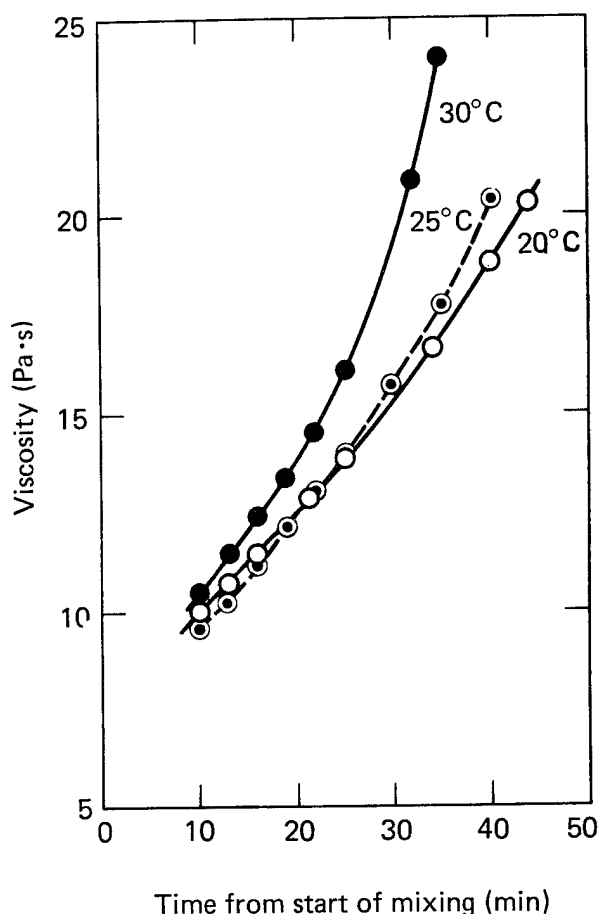


FIG. 8. Viscosity-time data at 3 temperatures for the representative formulations of Part XV A.2, LVT spindle 3 at 3 rpm.

PART XVI, A.1

To determine the effect of substituting DC1107 for accelerator QCF3-6559 on the properties of a blend of Sylgard 186 diluted with dielectric gel Q3-6527, we selected the following formulation of Part XI A.4.

Component	Parts by weight
L86-58A	70.6
L86-58B	7.0
PQ3-31A	11.2
PQ3-31B	11.2

We added L1107-69 to this formulation in various increments and measured initial viscosity and gel time at $25 \pm 1^\circ\text{C}$. We measured hardness after 24-h cure at $22 \pm 5^\circ\text{C}$ and extractables after 3-d cure at $22 \pm 5^\circ\text{C}$ when appropriate. The data are in Table 21. For the range of gel times of interest, the viscosity of blend is too high because the small amount of L1107-69 which we add does not dilute the mixture nearly as much as the much larger amount of PE56 (4.0 parts/100) added to the Part XI formulation. We ran a series of blends of varying proportions of L86-58 to PQ3-31 at a constant amount L1107-69 (0.32 part/100) and measured the usual properties. These are listed in Table 22 and gel times are plotted in Fig. 9. Between 24 and 30% Q3 in the blend with 186, there is

TABLE 21. Properties of a 77.6:22.4 blend (by weight) of L86-58 and PQ3-31 accelerated with varying amounts of L1107-69 for Part XVI A.1.

Property	Parts of L1107-69:100 parts of L86-58 plus PQ3-31 by weight						
	0	0.25	0.3	0.4	0.5	1.5	2.5
Initial viscosity ^a at $25 \pm 1^\circ\text{C}$, Pa·s	—	14.52	14.60	—	—	—	—
	—	—	14.28	—	—	—	—
Gel time at $25 \pm 1^\circ\text{C}$, min	442	101	80	56	45	8	<4
	—	—	84	—	—	—	—
Extractables, wt%							
After 3-d cure at $22 \pm 5^\circ\text{C}$	—	—	4.84	—	—	—	4.27
Hardness, Shore A							
After 24-h cure at $22 \pm 5^\circ\text{C}$	—	—	27.2	—	—	—	—
After 3-d cure at $22 \pm 5^\circ\text{C}$	—	—	—	—	—	—	30.6

^aBrookfield viscometer LVT spindle 3 at 3 rpm.

TABLE 22. Properties of blends of L86-58 with PQ3-31 in varying proportions with a constant 0.3 part (by weight) of L1107-69 per 100 parts of L86-58 plus PQ3-31 for Part XVI A.1.

Property	Weight Ratio of PQ3-31 to L86-58					
	22.4	24.0	24.8	25.5	27.0	30.0
	77.6	76.0	75.2	74.5	73.0	70.0
Initial viscosity ^a at 25 ± 1°C, Pa·s	14.60	11.36	9.08	8.64	8.32	7.25
	14.28	—	—	—	8.42	—
Gel time at 25 ± 1°C, min	80	110	113	—	100	108
	84	—	—	—	101	—
Extractable, wt%						
After 3-d cure at 22 ± 5°C	4.84	—	—	—	5.50	—
Hardness, Shore A						
After 24-h cure at 22 ± 5°C	27.2	—	18.8	—	18.4	—
Ultimate	39.0	—	37.4	—	37.4	—

^aBrookfield viscometer LVT spindle 3 at 3 rpm.

little change in the gel time. The viscosity effect diminishes abruptly at about 24.75% Q3. This change occurs in the viscosity range of interest to us (10 ± 1 Pa·s).

PART XVI, A.2

From this data, we selected the following composition as our representative formulation for Part XVI:

Component	Parts by weight
L86-58A	68.4
L86-58B	6.8
PQ3-31A	12.4
PQ3-31B	12.4
L1107-69	0.3

We measured viscosity with time and gel time at 20 ± 1°C, 25 ± 1°C, and 30 ± 1°C; the viscosity/time data are listed in Table 23 and shown in Fig. 10. Hardness was measured as a function of time at 22 ± 5°C. These data are shown in Table 24.

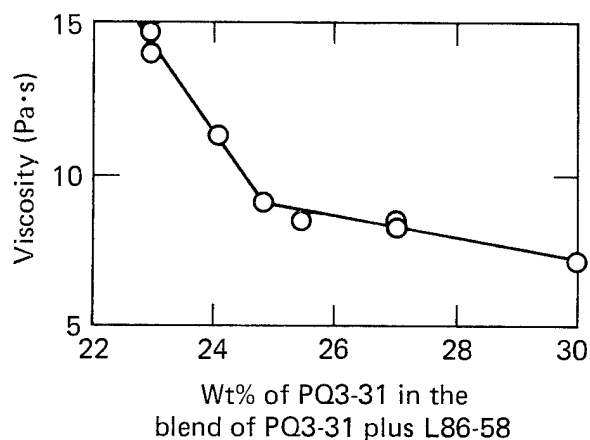


FIG. 9. Effects of the dilution of L86-58 with PQ3-31 with 0.32 part per 100 by weight of L1107-69 added to the blend on the viscosity at 25 ± 1°C for Part XVI A.2, LVT spindle 3 at 3 rpm.

MISCELLANEOUS

In the set of experiments to measure the effect of adding PQ3-31 to L86-58 accelerated with 0.3 part of L1107-69 per 100 parts of blend, we had difficulty in obtaining consistent data. We finally discovered that the variation resulted from dissolved gas in the blend. Results with and without vacuum degassing are shown in Table 25. With degassing, our data became consistent, so we adopted vacuum degassing as a standard procedure. It is probable that the gas is hydrogen released from the DC1107 fluid in a surge as mixing begins. It is not clear why the trapped gas should increase the initial viscosity.

In Part XVI we diluted L86-58 with PQ3-31 to a viscosity of 10 Pa·s with about 23 wt% PQ3-31 in

TABLE 23. Viscosity vs time data for the representative formulation of Part XVI A.2. at three temperatures.

Time from start of mixing, min	Viscosity, ^a Pa·s		
	20 ± 1°C	25 ± 1°C	30 ± 1°C
10	8.84	9.08	10.20
13	10.80	10.40	11.24
16	11.64	11.28	12.52
19	12.64	12.04	13.52
22	13.32	12.76	14.80
25	14.00	13.40	16.48
30	14.92	14.72	19.68
35	15.92	—	24.40
40	17.04	18.08	—
45	18.16	19.84	—

^aBrookfield viscometer LVT spindle 3 at 3 rpm.

the blend. In Part XVII, we diluted L86-58 to a viscosity of 2.5 Pa·s, which required about 45 wt% PQ3-31 in the blend. Both of these systems were accelerated with varying amounts of L1107-69 to achieve a gel time of about 90 min. These data are plotted in Figure 11 to show the difference between the two levels of PQ3-31 in L86-58. With more PQ3-31, more L1107-69 is required to achieve a set gel time.

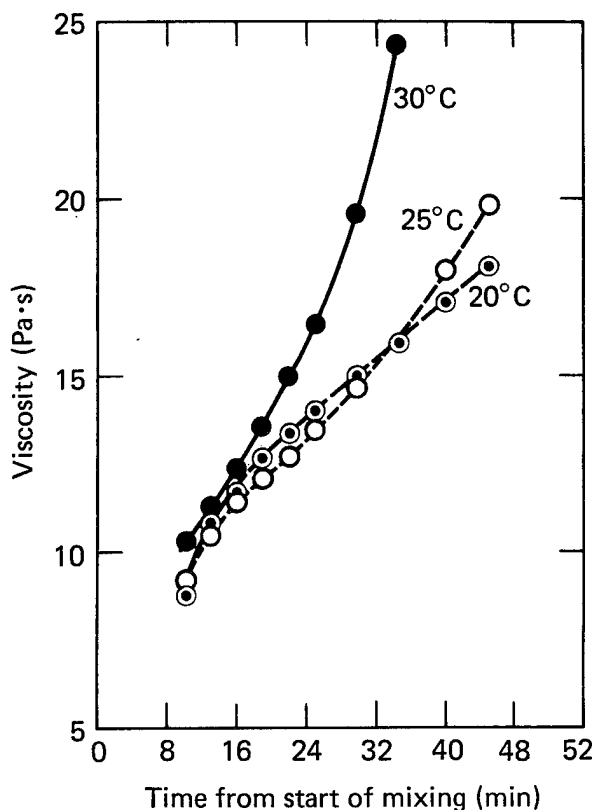


FIG. 10. Viscosity-time data at 3 temperatures for the representative blend of Part XVI A.2, LVT spindle 3 at 3 rpm.

TABLE 24. Properties of the representative formulation of Part XVI A.2. at three temperatures.

Property	20 ± 1°C	25 ± 1°C	30 ± 1°C
Zero-time viscosity, ^a Pa·s	6.7	—	6.6
Initial viscosity, ^a Pa·s	8.84	9.08	10.20
Time to double initial viscosity, ^b min	33	30	21
Gel time, min	155	113	64
Hardness, Shore A			
After 24-h cure at 22 ± 5°C	—	18.8	—
Ultimate	—	37.4	—
Extractables, wt%			
After >1-mo cure at 22 ± 5°C	—	4.57	—

^aBrookfield viscometer LVT spindle 3 at 3 rpm.

^bElapsed time from initial viscosity to 2 × initial viscosity.

TABLE 25. Effect of deaeration on the viscosity of blends of PQ3-31 and L86-58 accelerated with L1107-69.

Percent PQ3-31 in PQ3-31 and L86-58 blend	Parts L1107-69 per 100 parts of blend	Initial viscosity ^a at 25 ± 1°C, Pa·s	
		Deaerated	Not deaerated
27.0	0.3	8.32	9.88
		8.42	
25.5	0.3	8.64	10.96

^aBrookfield viscometer LVT spindle 3 at 3 rpm.

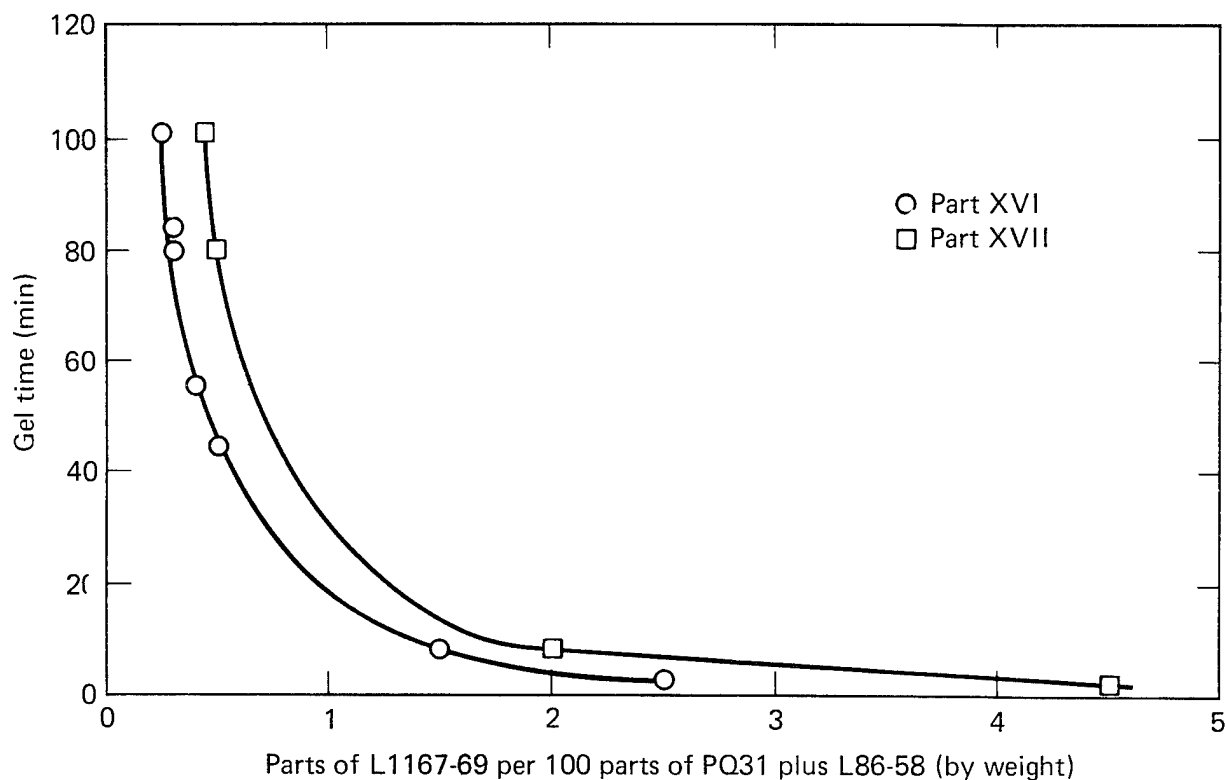


FIG. 11. Effect of L1107-69 on the gel time of 2 blends of PQ3-31 plus L86-58.

COMPARISON OF REPRESENTATIVE FORMULATIONS

Experimentally, we produced substitutes for DC93-120 by five different combinations of available starting materials. The compositions of representative formulations of each of these combinations are listed in Table 26. Properties of these formulations are listed in the ensuing tables and graphs.

The viscosity-time response at $25 \pm 1^\circ\text{C}$ of each formulation is plotted in Fig. 12. Other temperature-dependent properties are listed in Table 27. There are no significant differences in the reactivity of the five substitutes at 25°C . The gel time of the formulation from Part XVI is slower than the other four at 25°C but is about the same at 30°C .

Time-dependent properties (such as hardness and extractables content) and other properties (such as density and equilibrium swell ratio V/V_0) are listed in Table 28.

Formulations from VI and XV are related in that they are blends of Sylgards 184 and 186, but VI is accelerated by accelerator and XV is accelerated by DC1107. The formulations are very much alike in ultimate hardness, V/V_0 , and extractables.

Formulations from XI and XVI are related in that they are blends of Sylgard 186 and dielectric gel Q3-6559, but XI is accelerated with accelerator and

a touch of DC1107 and XVI is accelerated by DC1107 only. The formulations differ: formulation from XI is very soft, has a low degree of cross-linking, and has high extractables compared with XVI. The substitution of DC1107 for accelerator results is an acceptable formulation.

The formulation from X is distinct in that it is a blend of liquid Sylgard 184 and solid particulate silicon dioxide. Its properties are also distinct in some cases. The material is much harder than any other DC93-120 substitute, presumably because of the reinforcing action of the silicon dioxide. Surprisingly, the material is also highly cross-linked ($V/V_0 = 1.8$) compared with all other substitutes. This increased cross-linking may be because bonds are established between the silicone polymer chains and OH or other reactive groups on silicon dioxide particles.

All of our representative formulations, except the one from Part XI, meet our goals for a substitute for DC93-120.

As outlined in the program, we have characterized these representative formulations cured for at least 30 d at $22 \pm 5^\circ\text{C}$ by thermal gravimetric analysis, thermal mechanical analysis, differential scanning calorimetry, and the Rheometrics mechanical spectrometer. Data obtained by all

TABLE 26. Composition of representative formulations of substitutes for DC93-120 potting compound.

Component	Section of program				
	VI ^a	X	XI Wt%	XV	XVI
L84-89A	34.8	82.9	—	45.3	—
B	3.5	8.3	—	4.5	—
L86-52A	52.2	—	—	45.2	—
B	5.2	—	—	4.5	—
L86-58A	—	—	67.8	—	68.4
B	—	—	6.7	—	6.8
PQ3-31A	—	—	10.8	—	12.4
B	—	—	10.8	—	12.4
PE-56	4.3	4.2	3.8	—	—
L1107-69	—	—	0.1	0.5	0.3
SiO ₂ P75-1L236	—	4.6	—	—	—

^aReference 2: Table 15, combination 1b.

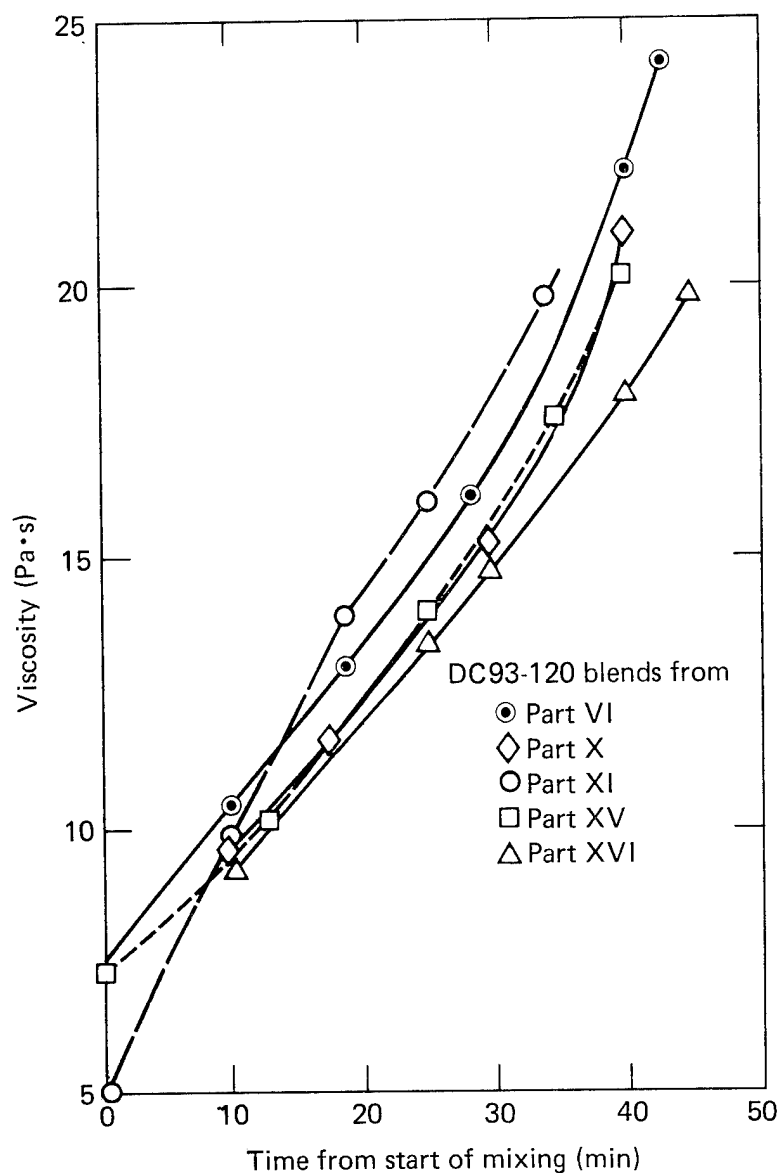


FIG. 12. Viscosity-time curves for representative blends of DC93-120 substitutes at $25 \pm 1^\circ\text{C}$, LVT spindle 3 at 3 rpm.

methods, except the mechanical spectrometer, will be presented in this report.

Thermal gravimetric analyses of all five representative formulations were made on 8- to 29-mg samples in a dry-nitrogen atmosphere at a heating rate of $10^\circ\text{C}/\text{min}$. After the samples reached 400°C , they were maintained there for a total heating time of 100 min. Weight loss data are shown in Table 29.

Formulations VI and XV, which are very similar except for the cross-linking agent, are alike

in thermal stability so that accelerator and DC1107 both have the same effect on cross-linking. Formulations XI and XVI are another pair similar except for the cross-linking agent but they are not alike in thermal stability: XI is more stable after extended heating at 400°C . In fact, XI, which is the most lightly cross-linked and has the highest extractables content of any of the formulations, is the most stable at 400°C . The silicon dioxide-reinforced formulation, X, is the least stable even though it is most highly cross-linked. Perhaps this is evidence

TABLE 27. Temperature-dependent properties of representative formulations of substitutes for DC93-120 potting compound.

Property	20°C					25°C					30°C					
	VI	X	XI	XV	XVI	VI	X	XI	XV	XVI	VI	X	XI	XV	XVI	
Viscosity, ^a Pa·s																
Zero time	7.6	—	5.2	7.5	6.7	7.4	—	5.0	7.0	—	—	—	—	7.1	7.2	6.6
Initial	10.00	9.52	10.36	10.00	8.84	10.40	9.60	9.72	9.64	9.08	10.08	10.24	11.08	10.40	10.20	
Time to double initial viscosity, ^b min	39.5	43	39.5	33	33	28	27	23	28	30	25.5	26	19.5	22	21	
Gel time, min	143	131	151	138	155	87	94	97	94	113	67	71	62	65	64	

^aBrookfield viscometer LVT spindle 3 at 3 rpm.

^bElapsed time from initial viscosity to 2 × initial viscosity.

TABLE 28. Time-dependent and other properties of representative formulations of substitutes for DC93-120 potting compound.

Property	VI	X	XI	XV	XVI
Hardness, Shore A,					
After 24-h cure at 22 ± 5°C	35.0	50.1	15.2	39.6	18.8
Ultimate	39.6	55.1	17.7	39.6	37.4
Time to reach ultimate, weeks	6	1	2	0.14	7.5
After 2-y cure at 22 ± 5°C	40.2	—	—	—	—
Extractables, wt%					
After 3-d cure at 22 ± 5°C	4.64	5.16	9.47	4.73	—
After >30 d	4.66	5.12	8.42	—	4.57
Density, kg/l	1.047	1.049	1.036	1.077	1.073
Equilibrium swell ratio in chlorobenzene, V/V ₀	2.320	1.799	3.297	2.370	2.579

TABLE 29. Thermal stability^a of representative formulations of substitutes for DC93-120 potting compounds.

Property	VI	X	XI	XV	XVI
Onset of weight loss, °C	112	80	110	105	92
Cumulative loss, wt%					
100°C	0	~0.05	0	0	0.1
200°C	0.4	0.4	0.5	0.4	0.6
300°C	1.4	1.6	2.0	1.4	2.0
400°C	3.8	4.3	3.6	3.6	4.1
Loss after 100-min total heating time, wt%	9.1	10.0	6.1	9.0	8.5

^aMeasured by TGA.

that some cross-links are less stable bonds between the silicone polymer and reactive groups on the particulate silicon dioxide.

Thermal mechanical analyses of all five representative formulations were made on samples varying in thickness from 5 mm to 7 mm, with a 10-g penetration probe in a dry-nitrogen atmosphere at a heating rate of 10°C per min over the range of -150°C to +225°C. Data are shown in Table 30. Substituting DC1107 for accelerator eliminates low-temperature softening. Formula X is different from the other four in that no melt penetration takes place. From about -25°C to +225°C, all TMA curves are essentially straight lines.

All five formulations have been tested by differential scanning calorimetry in nitrogen at a heating rate of 10°C/min over the temperature range of about -150°C to 150°C in the DuPont 900 instrument. Results are listed in Table 31. Both DSC and TMA analyses show that the formulation from VI is the most complex and that from X is the simplest. By DSC, the other three formulations are very similar to each other and to VI below 0°C.

Data from the Rheometrics mechanical spectrometer analyses of these representative formulations are too complex to be summarized easily so they will not be included in this report.

TABLE 30. TMA analysis of representative formulations of substitutes for DC93-120 potting compound.

Property	VI	X	XI	XV	XVI
Possible T_g , °C	-124	—	-115	-105	-108
Melt penetration, °C					
Start	-115	—	—	—	—
End	-85	—	—	—	—
Softening, °C					
Start	-68	-115	-90	—	—
End	-38	-45	-65	—	—
Melt penetration, °C					
Start	-38	—	-40	-55	-45
End	-32	—	-26	-35	-32

TABLE 31. DSC analysis of representative formulations of substitutes for DC93-120.

Event	VI			X			XI			XV			XVI		
	Onset	Peak	End	Onset	Peak	End	Onset	Peak	End	Onset	Peak	End	Onset	Peak	End
T_g , °C	-124	-118	-107	-130	-120	-93	—	-125	—	-128	-124	-117	—	-118	—
Exotherm, °C	-101	-87	-74				-108	-101	-83	-117	-84	-64	-107	-98	-90
		Large						Large			Large			Small	
Endotherm, °C	-60	-43	-30				-72	-46	-35	-60	-47	-41	-78	-46	-36
		Large						Large			Large			Large	
Endotherm, °C	31	35	45												
		Small													
Endotherm, °C	117	125	130												
		Very Small													
Exotherm, °C	219	222	227												
		Small													

CONCLUSIONS

The five approaches to a substitute for DC93-120 are:

1. Sylgard 186 diluted with Sylgard 184 and accelerated with accelerator QCF3-6559. (Part VI).
2. Sylgard 186 diluted with Sylgard 184 and accelerated with DC1107 (Part XV).
3. Sylgard 186 diluted with dielectric gel Q3-6527 and accelerated with accelerator QCF3-6559 and a very small amount of DC1107 (Part XI).
4. Sylgard 186 diluted with dielectric gel Q3-6527 and accelerated with DC1107 only (Part XVI).
5. Sylgard 184 thickened with silicon dioxide, Cabosil Grade MS75, and accelerated with accelerator QCF3-6559 (Part X).

All these can be formulated to meet the goals of a viscosity of $10 \pm 1 \text{ Pa}\cdot\text{s}$ at $25 \pm 1^\circ\text{C}$ and a gel time of $90 \pm 30 \text{ min}$ at $25 \pm 1^\circ\text{C}$. The formulation based on 186 Q3, E, and 1107 (number 3 above) had an extractable content of around 9 wt%. That is considerably greater than our goal of 6 wt% maximum.

The blend of 186 with 184 can be accelerated by either accelerator or DC1107. The two formulations

have very similar properties. If there is a problem with bubble entrapment with the QCF3-6559, this problem could be exaggerated with the DC1107 system because of rapid evolution of hydrogen from the 1107. Probably both systems could be made to work in assembly operations but they have not been tried.

The formulation based on 186, Q3, and 1107 (number 4 above) should have the least bubble problem because Sylgard 184 is not present. It has not been evaluated in assembly.

The formulation based on 184, SiO₂, and QCF3 (number 5 above) could be unsuitable for long-term storage because of the added silicon dioxide. The aging characteristics of Sylgard 184 reinforced with untreated silicon dioxide are not well understood. The hardness of this formulation could be a problem on disassembly. Again, this formulation has not been evaluated in assembly.

At the present time, no DC93-120 substitute is being used in production.

SUMMARY

Sylgard 186 can be diluted with Sylgard 184 to meet a viscosity specification of $10 \pm 1 \text{ Pa}\cdot\text{s}$ at $25 \pm 1^\circ\text{C}$. This blend can be accelerated with either accelerator QCF3-6559 or DC1107 fluid to a gel time of $90 \pm 30 \text{ min}$ at $25 \pm 1^\circ\text{C}$. The two formulations have very similar properties and both should be usable in production assembly.

Sylgard 186 can be diluted with dielectric gel Q3-6527 to a viscosity of $10 \pm 1 \text{ Pa}\cdot\text{s}$ at 25°C . This blend cannot be accelerated with QCF3 to the desired gel time. Adding a small amount of DC1107 to the blend with QCF3 achieves the gel time of 90

$\pm 30 \text{ min}$ at 25°C but the extractables content exceeds our goal of 6 wt% maximum. If all the QCF3 in the 186/Q3 blend is removed and DC1107 only is used as the accelerator, the blend achieves all of our goals.

Sylgard 184 can be thickened with silicon dioxide to a viscosity of $10 \text{ Pa}\cdot\text{s}$ at 25°C and can be accelerated with accelerator to a gel time of $90 \pm 30 \text{ min}$ at 25°C .

A substitute for DC93-120 potting compound can be achieved by any of four different formulations of materials.

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