# A SUBSTITUTE POTTING COMPOUND FOR SYLGARD 93-119 BASED ON SYLGARD 184 AND Q3-6527 DIELECTRIC GEL



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## ABSTRACT

This report deals with the development of a substitute potting compound developed by Pantex to replace the no longer available Dow Corning 93-119. The specific approach taken in this development utilized Dow Corning Sylgard 184 as the basic starting material, altering its viscosity with Dow Corning Dielectric Gel Q3-6527 and then modifying the cure rate with Dow Corning Accelerator Q3-6559.

Various tests were run to characterize both chemical and physical properties of the substitutes as well as the initial 93-119 and the starting raw materials. Summaries of the data peculiar to this substitute are presented here whereas a more detailed examination of the test methods and results for the overall project are presented in another report.

The properties of two substitutes developed from the two extremes of viscosity to be expected for Sylgard 184 during production are presented in this report. These substitutes are easily quick-frozen in the catalyzed state for production use upon thawing. They are however somewhat more difficult to deaerate and eventually cure to a harder state than the original DC 93-119.

## INTRODUCTION

With the discontinuation by Dow Corning of the production of several of their addition cured silicone potting compounds used by the DoE complex it became necessary to develop alternate or substitute materials. This task was undertaken jointly by the Lawrence Livermore Laboratory (LLL) and Pantex. This is an interim report and will deal only with the work done by Pantex toward the development of a substitute for 93-119.

The route toward this particular 93-119 substitute employs Dow Corning's Sylgard 184 as the main starting material which is diluted to the appropriate viscosity using Dow Corning's Dielectric

## DISCLAIMER

Gel Q3-6527 and accelerated to the desired cure rate using Dow Corning's Accelerator Q3-6559. For this study two lots of Sylgard 184 were used, one (lot 36) representing the low end of the viscosity range available from Dow Corning and the other (lot 31) representing the higher end.

## VISCOSITY MODIFICATION

One method of obtaining a substitute for Dow Corning's 93-119 uses Sylgard 184 as the base material and Dielectric Gel as the viscosity modifier or diluant. Table I lists the viscosities obtained as the amount of Dielectric Gel was varied for two different lots of Sylgard 184. Fig. 1 graphically represents the same information. Sylgard 184 (lot 31) represents a relatively high viscosity lot of 184 and lot 36 represents a relatively low viscosity lot. Viscosity was measured using a Brookfield LVT viscometer with a No. 3 spindle rotating at 12 rpm.

## CURE RATE MODIFICATION OF A SPECIFIC VISCOSITY MODIFIED SUBSTITUTE

Selecting an operating viscosity of 2.5 Pa•s for each of the two lots of Sylgard 184 fixed the amount of Dielectric Gel used. For each of these two blends the effect on cure rate at 25 C of adding incremental amounts of Accelerator Q3-6559 was evaluated. The data are presented in Table II and Figs. 2 and 3.

Several different tests can be employed to measure the degree of cure. One such test is SNAP TIME. The material will reach a point where an instrument such as a spatula inserted into the sample and withdrawn will produce a stringlike mass that "snaps" like a weak rubber band; hence the term "Snap Time."

Another such test is GEL TIME. An automatic timer such as a "Techne" or a "Mol Rez" periodically inserts a probe of a particular weight and geometric configuration into the material. As gelation of the sample progresses the force required to move the probe through the material becomes larger than some preset value and the instrument shuts off.

A third such measurement is DOUBLE TIME. This is the time measured from the start of mixing, for the viscosity to reach a value twice that of the 10-minute viscosity.

## TEMPERATURE DEPENDENCY OF CURE RATE FOR A SPECIFIC VISCOSITY MODIFIED, CURE RATE MODIFIED SUBSTITUTE

By selecting a snap time of 90 minutes at 25 C, operating conditions for the two cure rate curves fixed the amount of accelerator to be used. Two formulations were developed with 10-minute viscosities of 2.5 Pa•s and snap times of 90 minutes at 25 C for both a high and a low viscosity lot of Sylgard 184. For each of these two formulations the temperature dependency from 20 to 30 C was evaluated. These data are displayed in Table III and Figs. 4 and 5.

Until this time a Techne Gel Timer had been employed. Since the more common gel timer in the DoE complex is a Mol Rez Gel Timer manufactured by Gardner Instruments the temperature dependency test was repeated with the latter instrument. These results are given in Table IV and Figs. 6 and 7.

Table I. 10-Minute Catalyzed Viscosity vs Dielectric Gel Concentration in Sylgard 184 @ 25 C

| Dielectric Gel (pph) | Sylgard 184 10-Minute Viscosity (Pa·s) Lot 31 | Lot 36 |
|---|---|---|
| 0 | - | 2.64 |
| 0 | - | 2.70 |
| 2 | 2.75 | - |
| 2 | 3.02 | - |
| 3 | - | 2.37 |
| 6 | - | 1.98 |
| 6.11 | 2.40 | 2.15 |
| 6.8 | 2.25 | - |
| 10 | - | 1.79 |
| 15 | 1.73 | 1.43 |
| 25 | 0.97 | - |
| 36.7 | 0.97 | 0.97 |

*pph = parts per hundred of Dielectric Gel A to Sylgard 184 A. The amount of Dielectric Gel B is equal to the amount of Dielectric Gel A*

Table II. Accelerator Concentration vs Cure Rate of Viscosity Modified (2.5 Pa·s) Sylgard 184 @ 25 C

| Accelerator (pph) | Sylgard 184 (Lot 31) & 5 pph Dielectric Gel — Techne Gel Time (min) | Snap Time (min) | Double Time (min) | Sylgard 184 (Lot 36) & 2 pph Dielectric Gel — Techne Gel Time (min) | Snap Time (min) | Double Time (min) |
|---|---|---|---|---|---|---|
| 2 | 146.2 | 153.0 | 77.2 | 149.4 | 160.0 | 81.0 |
| 4 | 99.4 | 103.0 | 55.0 | 92.0 | 99.0 | 38.6 |
| 5 | 85.2 | 89.5 | 48.9 | 87.8 | 94.0 | 57.7 |
| 6 | 76.7 | 79.0 | 44.3 | 74.6 | 75.0 | 40.0 |
| 8 | 59.4 | 61.0 | 34.6 | 46.0 | 46.0 | 26.5 |
| 10 | - | - | - | 48.3 | 49.0 | 28.0 |

*pph = parts per hundred of Accelerator to Sylgard 184 A*



Table III. Temperature Dependency of Cure Rate for Specific Viscosity and Cure Rate Modified Substitute

| Temperature (C) | Sylgard 184 (Lot 31) & 5 pph Dielectric Gel & 5 pph Accelerator — Double Time (min) | Techne Gel Time (min) | Snap Time (min) | Sylgard 184 (Lot 36) & 2 pph Dielectric Gel & 5 pph Accelerator — Double Time (min) | Techne Gel Time (min) | Snap Time (min) |
|---|---|---|---|---|---|---|
| 20 | 71.3 | 152.6 | 153.0 | 63.5 | 144.5 | 147.5 |
| 23 | 52.1 | 113.5 | 114.0 | 53.0 | 106.0 | 108.0 |
| 25 | 42.9 | 90.6 | 93.0 | 45.2 | 86.0 | 89.0 |
| 27 | 48.4 | 82.4 | 82.0 | 43.8 | 74.8 | 77.0 |
| 30 | 39.0 | 63.3 | 64.0 | 37.6 | 58.0 | 60.2 |

Table IV. Repeat of Temperature Dependency of Cure Rate for Specific Viscosity and Cure Rate Modified Substitute

| Temperature (C) | Sylgard 184 (Lot 31) & 5 pph Dielectric Gel & 5 pph Accelerator — Double Time (min) | Mol Rez Gel Time (min) | Snap Time (min) | Sylgard 184 (Lot 36) & 2 pph Dielectric Gel & 5 pph Accelerator — Double Time (min) | Mol Rez Gel Time (min) | Snap Time (min) |
|---|---|---|---|---|---|---|
| 20 | 69.5 | 168.0 | 158.0 | 68.0 | 162.0 | 151.0 |
| 23 | 56.5 | 126.0 | 121.0 | 51.2 | 116.0 | 109.0 |
| 25 | 49.1 | 103.0 | 98.0 | 49.9 | 105.0 | 97.0 |
| 27 | 47.5 | 88.0 | 86.0 | 46.5 | 90.0 | 85.0 |
| 30 | 38.1 | 71.4 | 64.0 | 42.5 | 74.0 | 73.0 |

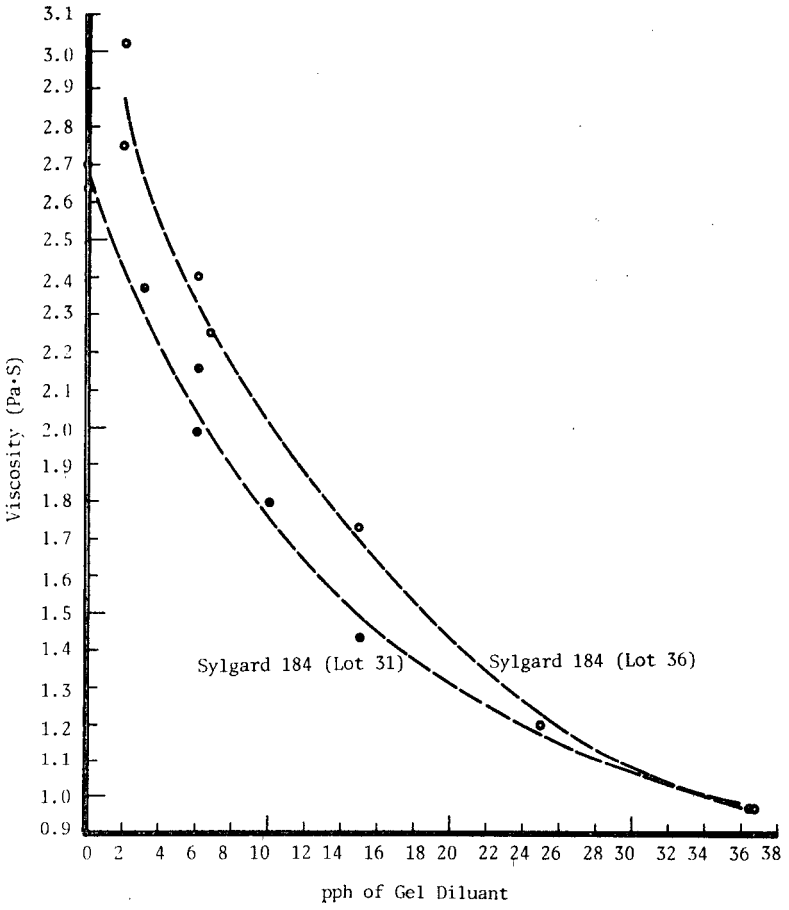


Fig. 1. Ten Minute Catalyzed Viscosity vs pph of Dielectric Gel in Sylgard 184 at 25 C

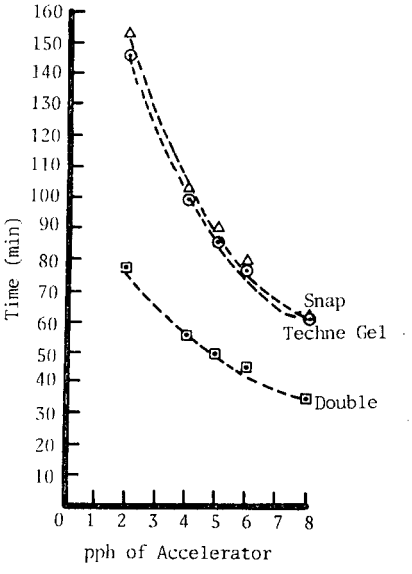


Fig. 2. pph of Accelerator vs Cure Rate of Sylgard 184 (Lot 31) Viscosity Modified to 2.5 Pa·s with 5 pph Dielectric Gel

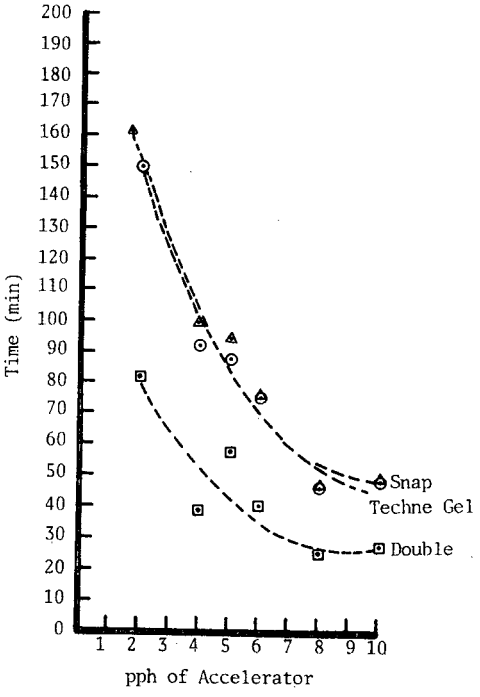


Fig. 3. pph of Accelerator vs Cure Rate of Sylgard 184 (Lot 36) Viscosity Modified to 2.5 Pa·s with 2 pph Dielectric Gel

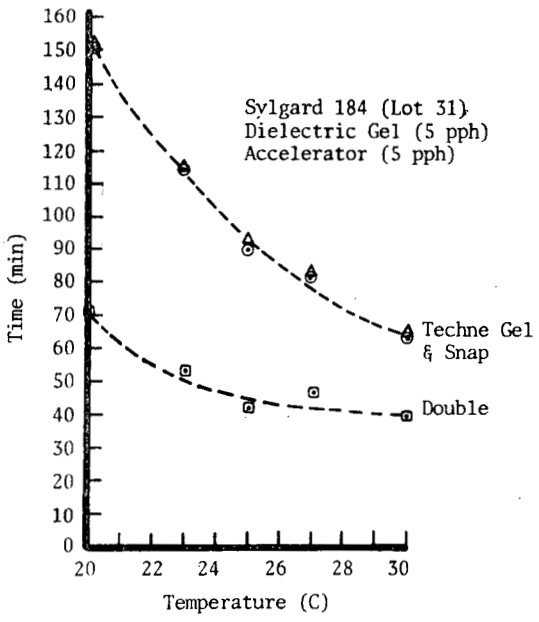


Fig. 4. Temperature Dependency of Substitute (Lot 31)

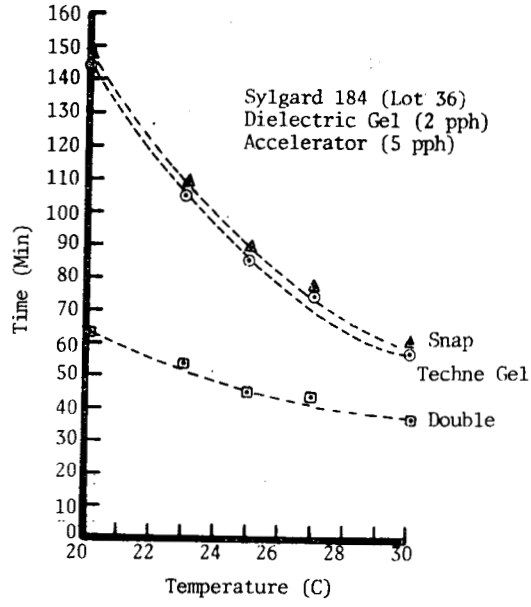


Fig. 5. Temperature Dependency of Substitute (Lot 36)

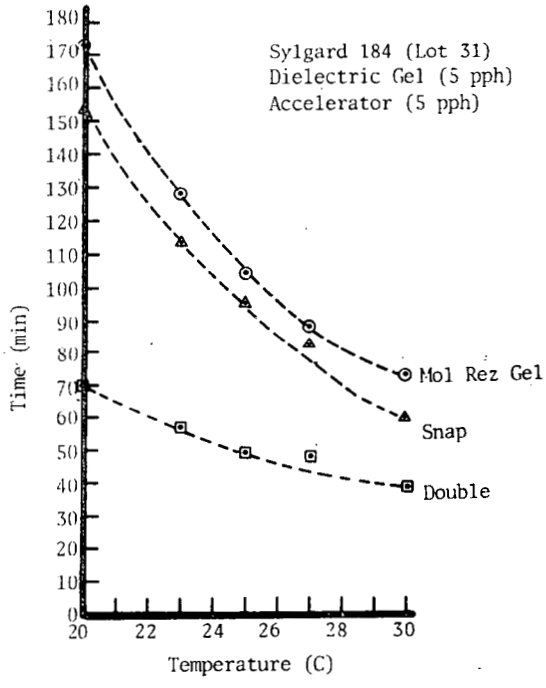


Fig. 6. Repeat of Temperature Dependency of Substitute (Lot 31)

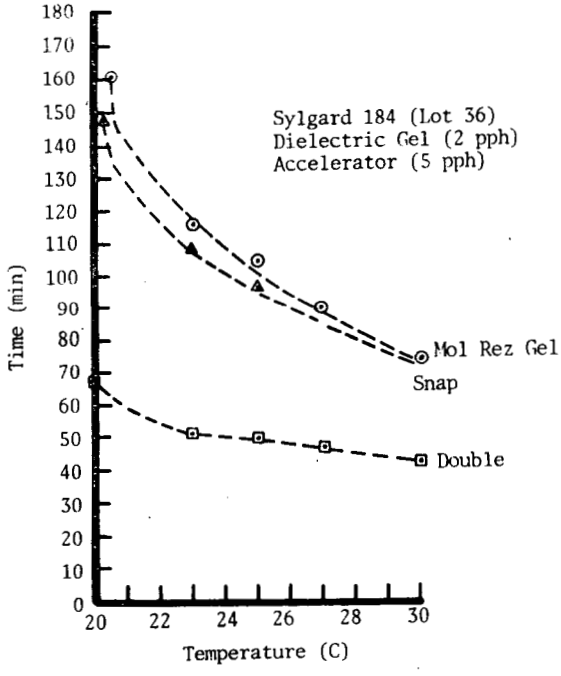


Fig. 7. Repeat of Temperature Dependency of Substiture (Lot 36)

## SHORE A DUROMETER RESULTS

The Shore A durometer hardness for one of the formulations was determined as a function of time and is shown in Fig. 8 along with a curve for 93-119, lot 38. The substitute developed was designed to have somewhat faster cure properties than 93-119. This difference is easily seen in Fig. 8. Unfortunately, the ultimate hardness of the substitute (approximately 35 Shore A) is considerably harder than the ultimate hardness of 93-119 (approximately 25 Shore A).

## BACKGROUND DATA OF MATERIAL PROPERTIES

Material properties of 93-119, the starting materials of its substitutes, and the substitutes themselves are outlined in Tables V and VI. A more detailed examination of these properties for all of the materials in the entire project as well as a more complete explanation of the test methods are presented in MHSMP-78-18.(*1*)

Pantex developed two formulations based on lots of materials at Pantex. LLL also developed two formulations based on different lots of material they had obtained. One formulation from each location was then selected for further testing at Pantex. These were the Pantex formulation based on 184 (lot 31) and the LLL formulation based on 184 (lot 89).

(*1*) *G. L. Flowers and S. T. Switzer, Background Material Properties of Selected Silicone Potting Compounds and Raw Materials for Their Substitutes, MHSMP-78-18, May 1978.*

## DYNAMIC VISCOSITY, LOSS AND STORAGE MODULI

A mechanical spectrometer manufactured by Rheometrics Inc. was used to measure the dynamic viscosity ($\eta$) the dynamic loss modulus ($G''$) and the dynamic storage modulus ($G'$) of the material during cure. The test method employed two parallel plates 25 mm in diameter separated by a gap of 1.4 mm. The stationary bottom plate was attached to a torque measuring transducer. The top plate was oscillated at a frequency of 1 hz with a maximum strain rate of 630%. After the sample was catalyzed it was placed between these plates and monitored during cure. A graph for 93-119 (lot 38) is presented in Fig. 9 and for the 93-119 substitute (lot 31) in Fig. 10.

## ADHESIVE PROPERTIES

A simple test was employed to evaluate the adhesive properties of 93-119 and its substitute. The test employed bonding two aluminum butt tensile cylinders together with a glue gap of approximately 0.13 mm. These were then evaluated for tension to failure at a crosshead rate of 1.27 mm/min.

| | 93-119 | Substitute |
|---|---|---|
| Strength (MPa) | 2.2 | 2.02 |
| Std. Dev. (MPa) | 0.20 | 0.15 |

Table V. Background Chemical Properties

VISCOSITY OF STARTING MATERIALS

| | Resin (A) (Pa·s) | Model/Spindle No. @ Speed (rpm) | Catalyst (B) (Pa·s) | Model/Spindle No. @ Speed (rpm) |
|---|---|---|---|---|
| Sylgard 184 (Lot 31) | 6.37 | LVF #4 @ 60 | 0.069 | LVT #1 @ 30 |
| Sylgard 184 (Lot 36) | 3.85 | LVF #4 @ 60 | 0.074 | LVT #1 @ 30 |
| Dielectric Gel (Lot 31) | 0.25 | LVT #3 @ 60 | 0.24 | LVT #3 @ 60 |
| Accelerator | 0.36 | LVT #3 @ 60 | NR | |
| Substitute (Lot 31) | 4.04 | LVT #3 @ 12 | NR | |
| Substitute (Lot 89) | 3.6 | LVT #3 @ 12 | NR | |

MOLECULAR WEIGHT

| | Resin A | | | Catalyst B | | |
|---|---|---|---|---|---|---|
| | $M_n$ | $M_w$ | MW | $M_n$ | $M_w$ | MW |
| 93-119 (Lot 30) | 12,300 | 42,500 | 3.46 | 12,200 | 44,300 | 3.63 |
| 93-119 (Lot 31) | 15,800 | 50,300 | 3.18 | 4,220 | 14,000 | 3.32 |
| Sylgard 184 (Lot 31) | 5,800 | 24,100 | 4.16 | 7,470 | 28,000 | 3.75 |
| Sylgard 184 (Lot 36) | 6,190 | 27,700 | 4.47 | 6,770 | 26,000 | 3.84 |
| Dielectric Gel | 5,450 | 14,400 | 2.64 | 5,090 | 13,900 | 2.73 |
| Accelerator | 7,000 | 19,800 | 2.83 | - | - | - |
| Substitute (Lot 31)[a] | | | | | | |
| Substitute (Lot 89)[a] | | | | | | |

[a] *Molecular weight data for these substitutes is merely an algebraic combination of the individual components and would have little meaning by itself.*

*NR - Not Run*

Table VI. Background Chemical Properties

| | Specific Gravity (A) | Specific Gravity (B) | Cured Specific Gravity | Vinyl Content (wt %) (A) | Vinyl Content (wt %) (B) | (A) Platinum Content (ppm) | (B) Active Hydrogen (wt %) | (B) Hydroxyl Content (wt %) | 3 Day Toluene Extractables (wt %) | 1 Month Toluene Extractables (wt %) |
|---|---|---|---|---|---|---|---|---|---|---|
| 93-119 | 0.98 | 0.96 | 0.97 | 0.19 | 1.89 | 8.9 | 0.15 | 0.478 | 4.03 | NR |
| Sylgard 184 (Lot 31) | 0.96 | 0.94 | 1.04 | 0.60 | 2.32 | 8.5 | 0.46 | 0.109 | 5.41 | 3.29 |
| Sylgard 184 (Lot 36) | 1.03 | 0.98 | 1.03 | 0.60 | 2.14 | 6.5 | 0.44 | 0.099 | 5.59 | 5.23 |
| Dielectric Gel (Lot 31) | 0.98 | 0.98 | 0.96 | 0.79 | 1.21 | 15.5 | 0.18 | 0.518 | > 20.00 | > 20.00 |
| Accelerator (Lot 56) | 0.98 | - | - | 0.77 | - | 184.0 | - | - | - | - |
| Substiture (Lot 31) | 1.05 | 0.98 | 1.02 | 0.47 | 3.37 | 44.7 | 0.31 | NR | 5.45 | 5.10 |
| Substitute (Lot 39) | 1.04 | 0.98 | 1.02 | 0.57 | 3.05 | 49.9 | 0.30 | NR | 5.66 | 5.06 |

Table VII. Chemical Reactivity Test Data for Silicone with High Explosives

Total Gas Evolved (mℓ Corrected to STP)

| | Elastomeric Material | PBX 9404 | PBX 9501 | HMX | RDX | PETN |
|---|---|---|---|---|---|---|
| HE Only | | | | | | |
| Run 1 | | 0.412 | 0.021 | 0.020 | 0.015 | 0.089 |
| 2 | | 0.399 | 0.016 | 0.005 | 0.018 | 0.100 |
| 3 | | 0.401 | 0.039 | 0.016 | 0.021 | 0.069 |
| 4 | | 0.405 | 0.038 | 0.017 | 0.027 | 0.561 |
| 5 | | | | 0.027 | | 0.287 |
| 93-119 | | | | | | |
| Run 1 | 0.009 | 0.376 | 0.021 | 0.018 | 0.013 | 0.158 |
| 2 | 0.009 | 0.384 | 0.025 | 0.029 | 0.012 | 0.176 |
| 3 | 0.011 | | | | | |
| 4 | 0.004 | | | | | |
| 5 | 0.014 | | | | | |
| Sylgard 184 (Lot 31) | | | | | | |
| Run 1 | 0.005 | 0.354 | 0.035 | 0.016 | 0.025 | 0.221 |
| 2 | 0.008 | 0.362 | 0.030 | 0.016 | 0.017 | 0.108 |
| 3 | 0.006 | | | | | |
| Sylgard 184 (Lot 36) | | | | | | |
| Run 1 | 0.019 | 0.362 | 0.019 | 0.017 | 0.034 | 0.229 |
| 2 | 0.011 | 0.367 | 0.028 | 0.016 | 0.018 | 0.209 |
| 3 | 0.008 | | | | | |
| 4 | 0.010 | | | | | |
| 93-119, Sylgard 184 (Lot 31) 5 pph Dielectric Gel 5 pph Accelerator | | | | | | |
| Run 1 | 0.013 | 0.312 | 0.020 | 0.052 | 0.026 | 0.408 |
| 2 | 0.009 | 0.334 | 0.029 | 0.039 | 0.030 | 0.522 |
| 3 | 0.023 | | | | | |
| 93-119, Sylgard 184 (Lot 36) 2 pph Dielectric Gel 5 pph Accelerator | | | | | | |
| Run 1 | 0.007 | 0.312 | 0.049 | 0.028 | 0.023 | 0.358 |
| 2 | 0.008 | 0.312 | 0.054 | 0.025 | 0.059 | 0.351 |
| 3 | 0.009 | | | | | |
| 4 | 0.020 | | | | | |
| 5 | 0.024 | | | | | |

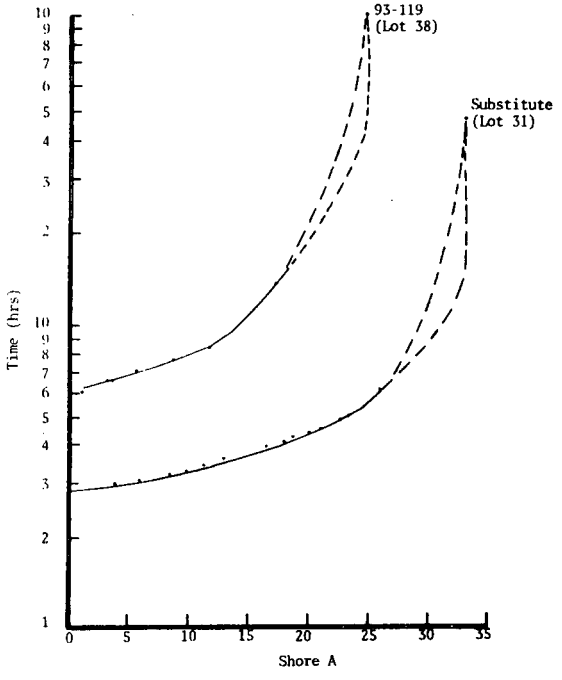


Fig. 8. Shore A Hardness vs Time @ 25 C

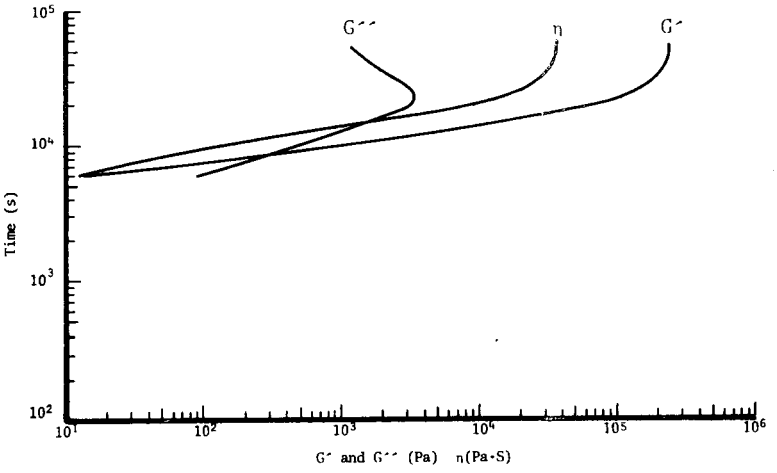


Fig. 9. Dynamic Viscosity, (η), Loss (G'') and Storage (G'), Moduli of Sylgard 93-119 (Lot 38)

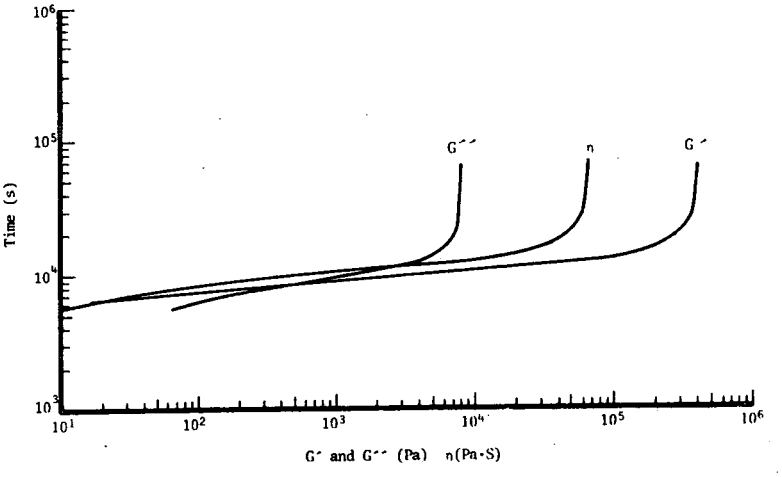


Fig. 10. Dynamic Viscosity (η), Loss (G'') and Storage (G') Moduli of Substitute (Lot 31)

## CHEMICAL REACTIVITY TEST (CRT)

The CRT is designed to provide an early indication of compatibility between a material sample and a high explosive (HE). The test involves placing a sample in a container, evacuating the container and back filling with helium. The container is then subjected to 120 C for 22 hours and the evolved gases measured. Separate tests are conducted for a 0.250 g sample of the material of interest, a 0.250 g sample of the HE, and a mixture of 0.250 g of the test material and 0.250 g of the HE. Should the amount of gases evolved from the mixture be significantly greater than the algebraic sum of the gases from the two individual components then the material is said to be incompatible with the HE.

All of the starting raw materials and historical samples of 93-119 were aged at least one month and then tested against PBX 9404, PBX 9501, HMX, RDX and PETN. No compatibility problems were observed. These results are summarized in Table VII.

## CONCLUSIONS

As a result of this study it is now possible to create a wide range of viscosities and cure rates of silicone potting compounds. One of the compounds yields a material similar to Dow Corning's 93-119 with two notable exceptions—this substitute is approximately 50% harder and it is more difficult to remove entrapped gases. The substitutes are compatible with various types of high explosives and presently there are no apparent shelf life problems. Other than containing more individual components than 93-119, the substitutes are easily mixed and quick-frozen for line usage.

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