N74-21162

# PRODUCTION DEVELOPMENT OF ORGANIC NONFLAMMABLE SPACECRAFT POTTING, ENCAPSULATING AND CONFORMAL COATING COMPOUNDS

CONTRACT NAS 9-11068

## VOLUME 4

## EXECUTIVE SUMMARY

(31 Jan 1974)

Sheldon L. Lieberman

Furane Plastics, Inc.
5121 San Fernando Rd. W.
Los Angeles, CA 90039

# EXECUTIVE SUMMARY

## INTRODUCTION

Numerous aerospace applications exist for potting and encapsulating compounds, and conformal coatings. NASA requirements for Shuttle particularly highlight the present necessity of having non-flammable versions of these compounds and coatings. These materials must also comply with functional criteria based upon electrical and mechanical strengths, odor and toxicity limitations, vacuum volatility, etc. Other constraints, including ease of application, availability, and cost enter into the selection process.

This program was a logical consequence of several NASA-sponsored (i.e. NAS 9-8749 and 9-8750) programs, as well as pertinent in-house R & D at the NASA Manned Spacecraft Center. As such it:

a. represented second generation refinements of the above R & D efforts,
b. established the logical impetus to pilot plant production of optimized formulation(s),
c. delineated pertinent production variables, and
d. updated property data in accordance with the most recently specified application parameters and their attendant requirements.

The program was conducted in accordance with the Program/Test Plan shown in Figure 1. Upon completion of the formulation, optimization, and evaluation phases, 50. pounds of the NASA-selected compound, EPOCAST 87517-A/B, was prepared and delivered to NASA.

## CONCLUSIONS

### 1.0 EPOCAST 87517-A/B

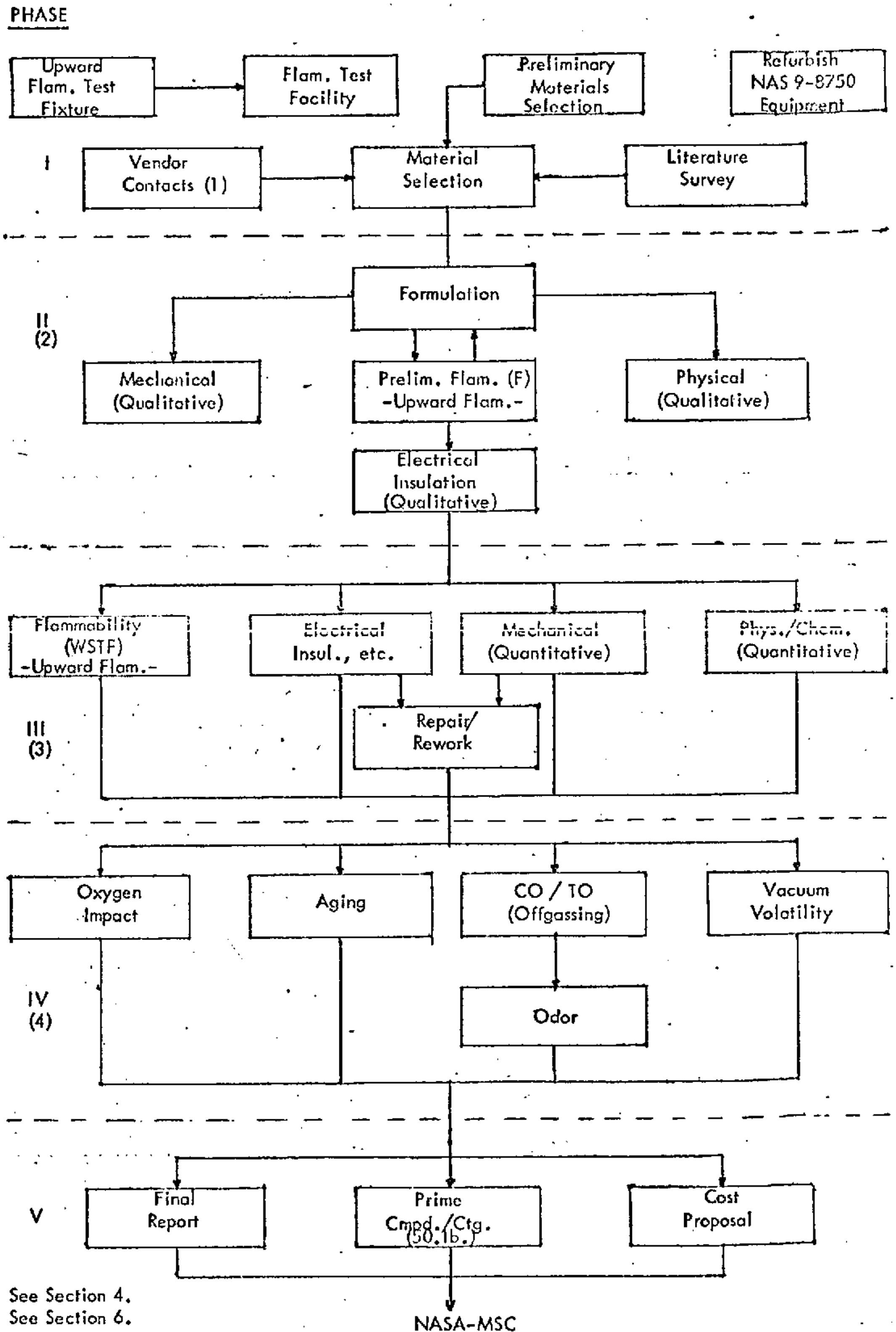
The following formulation, based upon the modified silicone RTV, F-476/596 (NAS 9-11068), was found to have the best balance of thermal, electrical, mechanical, physical, and chemical properties of those formulations evaluated in this program (Table 1).

| | |
|---|---|
| Sylgard 186 (Dow Corning) | 100. pbw |
| Ammonium Polyphosphate (Phos-Chek P/30, Reg.: Monsanto) | 50. " |
| 7570 Glass Frit (Corning Glass) | 25. " |
| Sylgard 186 Curing Agent (Dow Corning) | 10. " |

This formulation met all of the requirements of the Statement of Work (Appendix A) and its modifications, except for the following:

FIGURE 1

PROGRAM/TEST PLAN (ACTUAL)



1. See Section 4.
2. See Section 6.
3. See Section 7.
4. See Section 8.

## TABLE 1

### FORMULATIONS PROPERTIES

- Summary -

| Property | T/M (6) | Compound Requirement (7) | See Table | Modified Silicone RTV Min. | Modified Silicone RTV Max. | Modified Silicone RTV Ave. | Modified Fluorosilicone RTV Min. | Modified Fluorosilicone RTV Max. | Modified Fluorosilicone RTV Ave. | Epoxy Min. | Epoxy Max. | Epoxy Ave. | Epoxy-Urethane Min. | Epoxy-Urethane Max. | Epoxy-Urethane Ave. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Dielectric Constant | M | 5.0, max. | | 4.8 | 5.1 | 5.0 | 5.2 | 5.3 | 5.3 | 4.6 | 4.9 | 4.7 | 4.1 | 4.1 | 4.1 |
| Power Factor | M | 0.09, max. | | 0.09 | 0.12 | 0.11 | 0.020 | 0.020 | 0.020 | 0.083 | 0.088 | 0.086 | 0.051 | 0.053 | 0.052 |
| Dielectric Strength (v/mil) (1) | M | 350., min. | | 220. | 260. | 240. | 280. | 400. | 350. | 500. | 620. | 550. | 570. | 700. | 610. |
| Volume Resistivity (ambient) (x$10^{12}$ ohms-cm) | M | (P) 1., min.<br>(C) 1., min. | | 1200. | 1400. | 1300. | 4.2 | 4.8 | 4.5 | 0.057 | 0.083 | 0.074 | 34. | 40. | 38. |
| Surface Resistivity (x$10^{12}$ ohms) | M | 1., min. | | 28000. | >56000. | >47000. | 11. | 47. | 16. | 0.041 | 0.049 | 0.046 | 180. | 340. | 240. |
| Arc Resistance (seconds) | T | 45., min. (2) | | 125. | 128. | 127. | 4. | 5. | 4.3 | 44. | 62. | 53. | 23. | 28. | 25. |
| Insulation Resistance (megohms) | M | (3) | | | | | | | | | | | | | |
| ambient (x$10^5$) | | 1., min. | | 1670. | 4500. | 2720. | 3.3 | 4.0 | 3.6 | 0.16 | 0.22 | 0.18 | 1.6 | 2.0 | 1.8 |
| 212°F | | 750., min. | | $9.6x10^6$ | $9.9x10^6$ | $9.7x10^6$ | 1900. | 2700. | 2300. | 1.5 | 2.8 | 2.3 | 160. | 220. | 190. |
| pre-m.c. (4)/ambient (x$10^5$) | | 1., min. | | 1880. | 2000. | 1920. | 4.4 | 4.6 | 4.5 | 0.075 | 0.15 | 0.125 | 9. | 26. | 16. |
| m.c. (4)/ambient | | 200., min. | | $1.x10^6$ | $80.x10^6$ | $45.x10^6$ | $73.x10^3$ | $86.x10^3$ | $78.x10^3$ | 17. | 34. | 27. | $1.5x10^3$ | $3.5x10^3$ | $2.3x10^3$ |
| m.c. (4)/212°F | | 200., min. | | $0.6x10^6$ | $6.9x10^6$ | $3.9x10^6$ | 1000. | 1300. | 1100. | 0.12 | 0.19 | 0.15 | 3.7 | 7.5 | 6.2 |
| pre-rprd./ambient (x$10^5$) | | 1., min. | | 1580. | 1880. | 1740. | 3.7 | 4.6 | 4.1 | 0.29 | 0.38 | 0.34 | 12. | 28. | 19. |
| rprd./m.c. (4)/ambient | | 200., min. | | $1.3x10^3$ | $2.7x10^3$ | $2.0x10^3$ | $48x10^3$ | $51.x10^3$ | $49.x10^3$ | 15. | 19. | 17. | 780. | 1500. | 1020. |
| rprd./m.c. (4)/212°F | | 200., min. | | 530. | 730. | 610. | 620. | 630. | 623. | 0.12 | 0.20 | 0.16 | 3.5 | 4.1 | 3.8 |
| pre-vac./ambient (x$10^5$) | | 1., min. | | 300. | 2000. | 1300. | 2.2 | 2.4 | 2.3 | 0.042 | 0.062 | 0.051 | 75. | 1600. | 1300. |
| vac. (5) (x$10^5$) | | --- | | 1000. | 2000. | 1400. | 5.7 | 16. | 7.8 | 0.041 | 0.050 | 0.044 | (11) | (11) | (11) |
| vac. (5)/m.c. (4)/ambient | | 200., min. | | 31. | 5000. | 1900. | $59.x10^3$ | $81.x10^3$ | $73.x10^3$ | 7.8 | 16. | 11. | (11) | (11) | (11) |
| vac. (5)/m.c. (4)/212°F | | 200., min. | | 4.2 | $6.3x10^6$ | $1.5x10^6$ | 580. | 820. | 740. | 0.093 | 0.11 | 0.10 | (11) | (11) | (11) |
| Tear Strength (lb/in) | M<br>T | 15., min.<br>25., min. | | 50. | 60. | 58. | 14. | 20. | 16. | 51. | 140. | 70. | 120. | 130. | 128. |
| Tensile Strength (psi) | T | 350., min. | | 360. | 450. | 396. | 90. | 160. | 130. | 340. | 1330. | 604. | 1600. (8) | 1860. (8) | 1725. (8) |
| Elongation (%) | T | 125., min. | | 370. | 400. | 380. | 80. | 130. | 118. | 230. | 300. | 290. | 130. (8) | 140. (8) | 135. (8) |
| Shrinkage (%) | M | 1., max. (opaque)<br>3., max. (clear) | | -- | -- | <1. | -- | -- | <1. | -- | -- | <3. | -- | -- | <3. |
| Hardness (after full cure) | M | 30.-85. (Shore A) | | 57. | 59. | 58. | 72. | 75. | 74. | 65. | 79. | 74. | 83. | 90. | 86. |
| Clarity | T | Water clear | | opaque | | | opaque | | | -- | -- | (9) | -- | -- | (10) |
| Viscosity, 77°F (poises) (catalyzed) | T | 100. to 6,000. | | -- | -- | 2520. | -- | -- | 5460. | -- | -- | 5.76 | -- | -- | 70. |
| Specific Gravity | T | 1.25, max. | | -- | -- | 1.40 | -- | -- | 1.77 | -- | -- | 1.37 | -- | -- | 1.44 |

1. 34-59 mils thick.
2. Based on average value out of 5 for each specimen.
3. Based on average value out of 4 for each specimen
4. Moisture cycle (m.c.): 75-160°F/95% R.H./1 day per cycle/ 5 days.
5. Vacuum (vac): 1x$10^{-6}$ torr/150°F/24 hrs.
6. T.....Target Value M.....Mandatory Value.
7. Statament of Work, NAS 9-11068.
8. Four specimens.
9. Transparent/pale yellow.
10. Transparent/brown.
11. A malfunction in a temperature controller caused the destruction of the specimen during the vacuum exposure.

| Test | Requirement | T/M (1) | Results (ave.) |
|---|---|---|---|
| Power factor | 0.09 | M | 0.11 |
| Dielectric strength (v/mil) | 350., min. | M | 240. |
| Specific gravity | 1.25, max. | T | 1.40 |
| Clarity | water clear | T | opaque |

1. T.....Target value M.....Mandatory value

It appears to be suitable for potting, encapsulating, and conformal coating of electrical and electronic systems requiring immediate self-extinguishment in air or low flammability in augmented oxygen environments. It is noted that its insulation resistance significantly exceeds the 5. x $10^8$ ohms, minimum, required by MIL-specifications for regular printed circuit boards and for multilayer printed circuit boards. The high surface (4.7 x $10^{16}$ ohms) and volume (1.3 x $10^{15}$ ohms) resistivity, as well as its high arc resistance (127. seconds), are also noteworthy.

In comparison to previously developed low flammability systems based upon a silicone RTV system (e.g. NASA SG-12 KI: NAS 9-8750), EPOCAST 87517-A/B is far superior in all electrical respects except for power factor (significant difference) and dielectric strength (negligible difference). Its mechanical properties are higher, and its hardness and specific gravity are lower. EPOCAST 87517-A/B is, by comparison to SG-12 KI, significantly less affected by the high humidity cycle.

EPOCAST 87517-A/B's two part system is readily blended, degassed, and is capable of being applied by spatula, injection or extrusion gun, or even slowly poured. It can also be brushed or dipped with ease; spraying, while possible, would be more difficult unless thinned with a solvent. Similarly, if a very thin electrical coating is desired, it may be possible to dilute the compound with a low boiling perfluorinated solvent to obtain a lower viscosity system. Usage of such solvents may necessitate the frequent agitation of the blend to assure homogeneity during application. Solvent removal would be essentially complete subsequent to a thermal/vacuum post cure.

The data indicates that a vacuum/thermal post cure reduces the percent weight loss, percent volatile condensable material (VCM), outgassing (CO/TO), and odor.

This polymeric system, while readily processed, is sensitive to contamination, as are all addition reaction silicone RTV's. The "B" component apparently reacts with certain chemical specie, thereby inactivating it. Contact with such materials (or even their vapors) as amines and amine-cured elastomers and plastics, and certain other nitrogen compounds used to cure elastomers and plastics, tin compounds, sulfur and sulfur compounds (and their residues), such as are used in organic rubbers (e.g. butyl and chlorinated rubbers), and acid materials (and their residues) used in some silicone RTV rubbers, can inhibit cure at the surface, or possibly the entire structure of the compound. This can conceivably create compatibility problems ranging from contacted components and substrates, to tooling and facilities (i.e. cure ovens). Careful selection and cleanliness of contacting materials, equipment, and facilities is therefore maintained during the formulation and cure phases, and can probably be provided in production, or even in the field, with only minimal inconveniences. Where an incompatible material or component can not be avoided, coating it with a barrier or primer to act as an interface will suffice.

The ingredients in this formulation are essentially non-toxic from the standpoint of skin absorption. Normal handling and usage should present no particular hazards.

Table 2 is a compilation of all data developed on EPOCAST 87517-A/B during the course of the program.

## 2.0 EVALUATED FORMULATIONS

### 2.1 Modified Silicone RTV Polymers

Preliminary flammability tests did not show any significant difference between the presence of 75 or 50 phr of Phos-Chek P/30, or 50 or 25 phr of the 7570 glass frit. A reduction of the P/30 to 25 phr becomes significant. The removal of the frit doesn't change the flame-out time, but there is evidence of increased degradation and in the degradation distance. The P/30 appears to limit the burn time. When Dechlorane 604 is used as the flame retardant instead of P/30 and the frit, the flame-out time and residue percentage improved, but a greater surface char distance is noted.

### 2.2 Modified Fluorosilicone RTV Polymers

RTV 77-033 (Dow Corning), the base for F-387 (NAS 9-8750), is no longer available. RTV 94-531 (Dow Corning), an available "substitute", does not have quite the same flammability resistance as the original material, especially at lower levels of Dechlorane 604. Other than a slightly lower self-extinguishing distance, this system as formulated herein, has poorer flammability resistance than the EPOCAST 87517-A/B. The mechanical properties of these modified fluorosilicone RTV systems are less satisfactory than the modified silicone RTV ones. This system does not meet the Statement of Work requirements for dielectric constant, arc resistance, tear strength, tensile strength, elongation, or specific gravity. The $70.00/lb. cost of the RTV 94-531 is the highest cost precurser used in the program, and is more than a magnitude higher in cost than the Sylgard 186. In general, the properties of this system were inferior to those obtained with F-387.

### 2.3 Polyester Polymers

Brominated, but solid, polyester resins can be satisfactorily dissolved in the phosphorous-containing reactive diluent dimethyl allyl phosphonate (DMAP) and

## TABLE 2

## FORMULATION PROPERTIES

### - Summary -

| Property | T/M (1) | Compound Requirement (2) | EPOCAST 87517-A/B (Average) |
|---|---|---|---|
| Dielectric Constant | M | 5.0., max. | 5.0 |
| Power Factor | M | 0.09., max. | 0.11 |
| Dielectric Strength (v/mil) (3) | M | 350., min. | 240. |
| Volume Resistivity (ambient) (x $10^{12}$ ohm-cm) | M | (P) 1., min.<br>(C) 1., min. | 1300 |
| Surface Resistivity (x $10^{12}$ ohms) | M | 1., min. | > 47000. |
| Arc Resistivity (seconds) | T | 45., min. (4) | 127. |
| Insulation Resistance (megohms) | M | (5) | |
| ambient (x $10^5$) | | 1., min. | 2720. |
| 212°F | | 750., min. | 9.7 x $10^6$ |
| pre-m.c.(6)/ambient (x $10^5$) | | 1., min. | 1920. |
| m.c. (6)/ambient | | 200., min. | 45. x $10^6$ |
| m.c. (6)/212.°F | | 200., min. | 3.9 x $10^6$ |
| pre-rprd./ambient (x $10^5$) | | 1., min. | 1740. |
| rprd./m.c. (6)/ambient | | 200., min. | 2.0 x $10^3$ |
| rprd./m.c. (6)/212.°F | | 200., min. | 610. |
| pre-vac./ambient (x $10^5$) | | 1., min. | 1300. |
| vac. (7) (x $10^5$) | | --- | 1400. |
| vac. (7)/m.c. (6)/ambient | | 200., min. | 1900. |
| vac. (7)/m.c. (6)/212.°F. | | 200., min. | 1.5 x $10^6$ |
| Tear Strength (lb/in) | M<br>T | 15., min.<br>25., min. | 58 |
| Tensile Strength (psi) | T | 350., min. | 396. |
| Elongation (%) | T | 125., min. | 380. |
| Flammability | | | |
| Atmosphere (psia) | | | |
| oxygen | | 3.0 | 14.7 |
| nitrogen | | 7.0 | air |
| Ignitor | | silicone | propane |
| Specimen Geom. (L"xW"xH") | | 12.x2.5.x? | 9.x2.5x0.085 |
| Flame Propagation Rate (in./sec.) | | | |
| min. | | --- | 0.0 |
| max. | | --- | 0.0 |
| ave. | | | 0.0 |
| spec. no. | | --- | 3 |
| Self-extinguishing | M | Yes | Yes (8) |
| Burn Length (in.) | | --- | 0.1 |

## TABLE 2 (cont)

## FORMULATION PROPERTIES

- Summary -

| Property | T/M (1) | Compound Requirement (2) | EPOCAST 87517-A/B (Average) | |
|---|---|---|---|---|
| Burn Time (sec.) | | --- | 13.3 | |
| Flame Color | | --- | orange glow | |
| Smoke/Soot | | --- | little | |
| Sparks | | --- | none | |
| Mass Transfer | | --- | none | |
| Residue - type | | --- | bl./wht./brn. soot | |
| - amount | | --- | little | |
| Offgassing | | | | |
| Preconditioning | | --- | none | vac/ht (9) |
| Loading Conditions | | | | |
| Gas | | --- | $O_2$ | $O_2$ |
| Pressure (psia) | | --- | 4.3 | 4.3 |
| Temperature (°F) | | --- | ambient | ambient |
| Exposure | | | | |
| Time (hours) | | --- | 72. | 72. |
| Pressure (psia) | | --- | 5.0 | 5.0 |
| Temperature (°F) | | --- | 155. | 155. |
| Weight Loss (%) | | --- | 0.0045 | 0.0020 |
| Carbon Monoxide ($\mu$gm./gm.) | M | 25., max. | 0.1 | 0.1 |
| Total Organics ($\mu$gm./gm.) (12) | M | 100., max | 42.0 | 3.4 |
| Constituents Analysis ($\mu$gm./gm.) | | | | |
| TF - Freon | | --- | 4. | 2. |
| Benzene | | --- | 2. | --- |
| Xylene | | --- | --- | 0.8 |
| Siloxane dimer | | --- | 160. (10) | 2. |
| Siloxane trimer | | --- | --- | 10., <29. (11) |
| Trisiloxanes | | --- | 30., >20. (11) | --- |
| Tetrasiloxane | | --- | 10., <20. (11) | --- |
| No. unidentified/each | | --- | -, 1>20. (11) | --- |
| Odor | | | | |
| Preconditioning | | --- | none | vac/ht (9) |
| Exposure | | | | |
| Time (hours) | | --- | 72. | 72. |
| Temperature (°F) | | --- | 120. | 120. |

## TABLE 2 (cont)

## FORMULATION PROPERTIES

### - Summary -

| Property | T/M (1) | Compound Requirement (2) | EPOCAST 87517-A/B (Average) | |
|---|---|---|---|---|
| Dilution with Oxygen | | | | |
| 1:29 | | --- | 0.0 | 0.0 |
| 1:9 | | --- | 0.4 | 0.0 |
| 1:1 | M | 2.0, max. | 0.8 | 0.0 |
| Vacuum Effects (13) | | | | |
| Preconditioning | | --- | none (14) | vac/ht (15) |
| Weight loss (%) | M | 1., max. | 0.2208 | 0.2100 |
| VCM (16) (%) | M | 0.1, max. | <0.0007 | <0.0 |
| Oxygen Aging (17) | | | | |
| Tensile strength change (%) | T | 20., max. | 2.2 | --- |
| Percent elongation change (%) | T | 20., max. | 4.6 | --- |
| Oxygen Impact Stability (psia) | | | | |
| Mechanical impact (18) | | --- | 3333. | --- |
| Pneumatic impact | | --- | 375. | --- |
| Shrinkage (%) | M | 1., max. (opaque)<br>3., max. (clear) | <1. | |
| Hardness (after full cure) | M | 30.-85. (Durometer A) | 58. | |
| Clarity | T | Water clear | Opaque | |
| Viscosity, 77.°F (poises) (catalyzed) | T | 100. to 6,000. | 2520. | |
| Specific Gravity | T | 1.25, max. | 1.40 | |

1. T.....Target M.....Mandatory
2. Statement of Work, NAS 9-11068.
3. 34-59 mils.
4. Based on average value out of 5 for each specimen.
5. Based on average value out of 4 for each specimen.
6. Moisture cycle (m.c.): 75.-160.°F/95% R.H./1 day per cycle/5 days.
7. Vacuum (vac.): 1 x $10^{-6}$ torr/150.°F/24. hrs.
8. Self-extinguishing when ignitor flame removed.
9. $10^{-6}$ torr/150.°F/8. hrs.
10. Identification uncertain.
11. Infrared scan.
12. Based on pentane equivalents.
13. Test environment:
    Final pressure: 4. x $10^{-6}$ torr
    Time: 24. hours
    Sample temp.: 150.°F
    Condenser temp.: 77.°F
14. Preconditioning: 24. hours/ambient temperature/ 50.% relative humidity
15. Preconditioning: $10^{-6}$ torr vac./150.°F/8. hrs.
    24. hours/ambient temperature/
    50.% relative humidity
16. Volatile condensable material (VCM)
17. Conditioned per ASTM D572 for 96. hours at 300. psi and 156.°F
18. 50. ft. lb.

cured at room temperature of 150.°F with a ketone peroxide/cobalt system to a clear semi-hard material which has poor mechanical properties such as tear resistance. This material is immediately self-extinguishing with a match ignitor in air and, hence, has some further interest as a clear, flame retarding system.

Isophthalic and bisphenol polyesters are not sufficiently compatible with such reactive flame-retarding diluents as DMAP, FYROL Bis-Beta (Stauffer Chemical Co.), or (BIS(DBP) H (White Chemical Co.). The pre-blends which consisted of a flexible polyester, a brominated polyester, and a reactive phosphorus-containing polyester, considered in this program, are not adequately cured by either a) redox-promoted aromatic diacyl peroxide (i.e. 2,4-dichlorobenzoyl peroxide), or b) teritary butyl peroctoate.

## 2.4 Epoxy Polymers

The epoxy formulations utilized in this program do not provide a satisfactory balance of flammability resistance in 10. psia, 30.% $O_2$/70.% $N_2$, flexibility, and good electrical and mechanical strength. Their slight tack, slight melting during flammability testing, poor odor test results and only fair outgassing resistance indicate a possible stoichiometry imbalance. It should be noted that its weight loss percentage and VCM are higher than the modified silicone RTV and the epoxy-urethane, but are still within the allowable limits.

Solid brominated epoxy resins can be solubilized with a reactive, brominated epoxy diluent, ERX-67 (Shell Chemical Co.), to a 75.°F shelf stability of approximately 2. weeks. D.E.R. 741 (Dow Chemical) is also a suitable diluent which can impart some flexibility, but it does not contain any flame-retarding constituents. ERX-67 blends with D.E.R. 741. This latter preblend system has a very low viscosity, high bromine content, and is stable at room temperature.

It is not feasible to solubilize tetrabromophthalic anhydride in bis dibromopropanol phosphoryl dimethylol amide (bis(DBP)AM (White Chemical Co.) at a low enough temperature at ambient pressure.

Therefore, a preliminary step of reaction between the two moieties could not occur and, hence, a low temperature cure with a flexibilized brominated epoxy pre-blend can not be carried out. Also, a pre-blend of the anhydride dissolved in D.E.R. 741 is not readily accomplished without causing an insoluble reaction product from forming in part of the mixture.

Mono dibromopropanol maleate (DBP-ES: White Chemical Co.) has low solubility in DMAP even when heated. This prevents their being reacted with each other via a free radical initiator, which, in turn, would produce available carboxyl groups which can be reacted with a flexibilized brominated epoxy pre-blend.

The flexible amine-cured RTV 602 (G.E.) does not seem to be compatible with the epoxies tested in this program. Even though the individual components are clear, the castings are opaque. They are also quite stiff.

## 2.5 Urethane Polymers

Non-accelerated (e.g. dibutyl tin dilaurate or stannous octoate) formulations of a bromine/phosphorus-containing dihydroxy polyol, Brominex 160P (Swift Chemical Co.), with various diisocyanates have no voids, but are only poorly cured after a minimum of 100. hours at 150.°F. At similar concentrations, stannous octoate gives a better cure and less voids than dibutyl tin dilaurate. Stannous octoate, at an 0.08% level with these diisocyanates and the Brominex 160P, provides void free castings in 55.-100. hours at 150°.F. Although higher concentrations of the stannous octoate shortened the cure time, it increased the void content.

The rates of gelation for the three diisocyanates utilized for the urethanes in this program were found to be: Isonate 143L $>$ PAPI 901$\gg$Brominex 9107. Castings with Isonate 143L have a much lighter color than those made with PAPI 901. Urethanes based upon the formulations herein cured, do not as readily cure as prepolymer systems.

The flammability resistance of the urethane system tested in 10. psia 30.% $O_2$/ 70.% $N_2$ was not as good as the modified silicone RTV or the epoxy-urethane, but was comparable to the epoxy. None of the urethane formulations, when conditioned in high humidity and heat, are hydrolytically stable.

## 2.6 Epoxy-Urethane Polymers

Commercially available pre-reacted epoxy-urethanes are not sufficiently flexible for the applications intended in this program. There are no commercially available co-reacting epoxy-urethane systems, let alone ones which would be flame-retarding. An apparently unique reaction sequence was developed under this program which utilizes, in terms of their respective ratios of equivalents: diepoxide/dihydroxy polyol/diisocyanate: 1/1/2.

Based upon the ingredients used, it is possible to incorporate on the resultant polymeric structure bromine, phosphorus, and nitrogen. PAPI 901, although darker in color, is preferable to Isonate 143L because its package stability is apparently better. Diethyl N,N-bis(2-hydroxy ethyl) amino methyl phosphonate (e.g. Fyrol 6: Stauffer Chemical Co.) appears to accelerate the cure of the co-reacting system developed in this program.

The epoxy-urethane is almost as flame retardant as the modified silicone RTV. They are both self-extinguishing when the ignitor flame is removed. The burn time and burn length is just slightly greater. Its mechanical strength properties are better than the other formulations so tested. Low electrical resistance at 212.°F, especially after a high humidity/thermal conditioning cycle, and its low arc resistance, minimizes its usefulness as an electrical insulation. The high humidity/thermal conditioning cycle reduces surface transparency but does not seem to affect the interior.

The outgassing from this formulation is greater than for the modified silicone RTV or the two epoxies tested. Although its carbon monoxide is within the allowable limits, the total organics are very high. However, the vacuum/thermal post cure clearly reduces both values. Odor results, even after a vacuum/thermal post cure, are still unsatisfactory. The effect of vacuum exposure is tolerable; the specifications are met. The target value of 20.%, maximum, change in tensile strength after oxygen aging wasn't met. The elongation value was only a 16.5% difference. Therefore, it is considered satisfactory in this latter regard. This formulation may be used for applications whose limits are:

Mechanical impact: 3333. psia
Pneumatic impact: 375. psia.

## 3.0 GENERAL

Production quantities of EPOCAST 87517-A/B can be readily made on available Furane equipment. Preparation of cured samples and checking their hardness is the simplest method to determine degree of cure. Pigmenting of EPOCAST 87517-A/B does not significantly affect hardness.

The modified silicone RTV's are the easiest to fabricate into cured structures; the epoxy-urethanes are the most difficult.

Anti-foaming agents added to the epoxy-urethanes do not result in a lower void content. However, wiping the molds with these agents significantly helps in surface void removal. Cured epoxy-urethane specimens containing ERX-67 had a lighter color than those which did not.

# RECOMMENDATIONS

## 1.0 EPOCAST 87517-A/B

Examine processing variables in greater detail. Consider such factors as:

a. Minimum cure time at ambient and at 150.°F with respect to cured properties,
b. Six month storage life of the "A" and "B" portions on final handling/processing and on cured compound's properties,
c. Optimum vacuum/thermal post cure cycle,
d. Suitable solvent and solvent percentages for low viscosity versions.

Carefully solvent clean molds and other substrates (e.g. wire insulation, connectors, etc.), oven dry, if possible, and keep in non-contaminating environment. Establish appropriate quality control limits which are relevant to specific end product performance.

Evaluate Phos-Chek P/30 "Regular" in comparison with the "fine" grade with respect to processability and end product properties. Reformulate this system so that a non-toxic, non-flammable, etc., pigment is incorporated with the curing agent in such a manner as to stabilize it for extended storage periods. Verify suitability of the recommended pigment with respect to end product properties.

Evaluate the vacuum weight loss and VCM similar to the manner in which the present data was obtained, except that the exposure temperature should be 257.°F instead of 150.°F in order to more realistically compare this formulation with other materials previously tested in accordance with the specified test method.

## 2.0 EVALUATED FORMULATIONS

Improve the mechanical strength of the polyester system and consider its use where a solvent-free, clear, low viscosity, low flammability system is required.

Review the stoichiometry of the epoxy system F-600. Note clarity of casting, and potential of good aging properties in oxygen and/or radiation environment due to presence of ERX-67.

Further development work is recommended for the epoxy-urethanes to reduce their processing difficulties and improve their electrical properties. This continued interest is due to their low viscosity, low flammability, transparency, minimal effect by the moisture cycle, and high mechanical strength.

a. Consider the use of Fyrol 6 as a partial substitute for the Brominex 160P since this may accelerate the reaction and lighten the color of the casting.
b. Determine optimum vacuum/thermal post cure cycle. Particularly note whether post curing removes the bromoethane and benzene on a linear or decreasing exponential curve.
c. Check stoichiometry with respect to <u>exact</u> hydroxyl and NCO numbers.

Evaluate the vacuum weight loss and VCM at 257.°F for the epoxy-urethane. Compare their oxygen aging data with specimens exposed to air for 96. hours at 156.°F to determine if a similar hardening of the casting occurs. Also consider use of the epoxy-urethanes in such non-electrical applications as flame retardant sealants.

Evaluate ERX-67 as an anti-aging reactive chemical.