FURANE PLASTICS
INCORPORATED

5121 SAN FERNANDO ROAD WEST, LOS ANGELES, CALIF. 90039

Copy: 20

- FINAL REPORT -

S. L. Lieberman 16 December 1971

Development of Organic Non-Flammable Spacecraft Potting, Encapsulating and Conformal Coating Compounds

NAS 9-8750

(NASA-CR-115364) DEVELOPMENT OF ORGANIC NON-FLAMMABLE SPACECRAFT POTTING, ENCAPSULATING AND CONFORMAL S.L. Lieberman (Furane Plastics, Inc., Los Angeles, Calif.) 16 Dec. 1971 394 p CSCL 11D G3/18
N72-17533
Unclas
14869
(CATEGORY)

N72-17533

DEVELOPMENT OF ORGANIC NON-FLAMMABLE SPACECRAFT POTTING, ENCAPSULATING AND CONFORMAL COATING COMPOUNDS

S. L Lieberman

Furanê Plastics
Los Angeles, California

16 December 1971

N72-17533

FORWARD

This research program was sponsored by the Materials Technology Branch of the Manned Spacecraft Center, National Aeronautics and Space Administration. Mr. H. F. Kline was the Project Monitor for NASA. The Program was conducted by Furane Plastics, Inc., under Contract No. NAS 9-8750. Mr. Sheldon L. Lieberman was the Program Manager, Principal Investigator, and author of this Final Report. Laboratory and test assistance was provided by Mr. David Lufkin.

The author particularly wishes to acknowledge the extensive test support and forbearance provided by the entire staff and, in particular, Messrs. D. Pippen and L. Shluter, of the White Sands Testing Facility of NASA-MSC, and to the testing staff of the Delsen Corporation. Appreciation is also extended to Mr. W. Dewar, of Furane, for his technical guidance and support. Finally, credit is given to R/D Industries for the design, development, and much of the fabrication of the Flammability Test Facility, and the other special molds and tooling utilized for the Program.

ABSTRACT

The overall Program objective was to develop a flexible compound which not only functioned in a manned aerospace environment as an effective electrical insulation, but whose flammability characteristics in 16.5 psia, 60% oxygen/40% nitrogen were evidenced by rapid self-extinguishment and minimal thermal (pyrolysis) degradation.

Based upon extensive contacts with sources of pertinent information (i.e. literature survey, vendors, etc.), a broad array of "non-flammable" polymeric specie, and additives generally noted to have flame retarding properties, were investigated, formulated, and tested.

The following polymeric matrices were examined in depth: fluoroelastomers, modified fluoroelastomers, silicone RTV's, fluorosilicone RTV's, and modified silicone and fluorosilicone RTV's. Almost none of these systems burned in air, but all burned in 6.2 psia oxygen. Inorganic, organic, and inorganic/organic additives were evaluated in conjunction with these polymers in order to achieve the required survival in the 16.5 psia 60/40 environment. Depending upon formulations, it was possible to achieve non-flammable products when tested in the 1/4 in. x 1/4 in. x 1 in. configuration, even up to and including 16.5 psia oxygen. However, in order to assure this level of flame resistance, it was found necessary to heavily "load" the matrix with the additives. This resulted in a significant reduction in mechanical properties and large increases in viscosities. Optimization of formulations to obtain a suitable balance between these properties and flammability resistance led to the final selection of Formulation 387 as the primary system. Its fluorosilicone RTV/halogen-loaded constituency meets almost all Program requirements. The very few low valued properties found are within a realistic level of design toleration.

Complete formulation, processing, and test data is herein provided for F-387, and the other formulations prepared by the Project. These other formulations include several potentially useful for this Program, but which require further development, possibly even synthesis of necessary stable intermediaries.

This report contains details of those test methods and procedures utilized in the Program but which are not either readily available, or are of some special nature. In addition, a description of the special flammability facility designed and fabricated for this Program is also presented.

TABLE OF CONTENTS

Page

VOLUME II

LIST OF TABLES

LIST OF ILLUSTRATIONS

LIST OF APPENDICES

List of Appendices - cont'd.

SECTION 1

INTRODUCTION

Numerous aerospace applications exist for potting and encapsulating compounds, and conformal coatings. These include connectors, printed circuit boards, wire harness terminations, circuit breakers, etc. These materials perform such diverse design and fabrication functions as electrical insulation, abrasion and handling protection, physical locating and orienting restraint, shock and vibration isolation, and atmospheric (particularly moisture and high humidity) and bacteriological protection. There are a broad range of conventional off-the-shelf flexible or semi-flexible materials (e.g. urethanes, polysulfides, epoxies, silicones, and polyesters) which could satisfactorily provide these functions and, in fact, are approved for the above usage under various applicable MIL and NASA specifications (Refs. 1-1, -2, -3, and -4). Some of these materials are inherently flame retardant in air by virtue of their molecular structure or additives. However, none of them are non-flammable, or even completely and rapidly self-extinguishing, in 6.2 psia oxygen or 16.5 psia 60% oxygen/40% nitrogen, even with so-called non-flammable additives.

NASA experience (Refs. 1-5, -6, -7, -8, -9, -10, and -11) has particularly highlighted the necessity of having non-flammable versions of these compounds and coatings for such man-rated space vehicles as the Command and Lunar Modules for the Apollo spacecraft, and for the upcoming SKYLAB and Shuttle operations. As NASA approved Category B, Minor Exposed Materials in the Crew Bay Atmosphere (Ref. 6-1), items, it is essential that these materials, regardless of exposure to cabin atmosphere (i.e., vacuum to 16.5 psia 60% oxygen/40% nitrogen), provide no fuel to any accidental flame initiation. Such criterion is exceedingly difficult to accomplish with polymeric moieties, even when no hydrogen is present in the polymer. For example, "...Teflon, which is a very fire resistant material in air, burns rapidly in 100% oxygen at 16.5 psia and significantly in a 60% O_2/40% N_2 environment at that pressure." (Ref. 1-12).

Besides the flammability aspect, the material selection criteria for the manned space structures must also consider the critical problems of toxicity resulting from outgassing within the closed ecological system of manned space flight.

From the above, it was apparent that there were no presently available potting/encapsulating compounds and conformal coatings which not only comply with the conventional functional and processing requirement, but also meet the unique safety criteria engendered by manned spacecraft operations. It was therefore the objective of this Program's R & D effort to develop, formulate, and test non-flammable, non-toxic, vacuum stable, low temperature curing compounds which would be applicable to potting, encapsulating, and conformal coating of electrical systems typically encountered in manned aerospace systems.

SECTION 2

PROGRAM SCOPE

In addition to the overall Program scope definition established by the Statement of Work (Ref. Appendix A), other realistic constraints were initially applied, some due to obvious technical needs in terms of probability of success, others to budget restrictions. These limitations included:

a. No new polymer synthesis routes would be investigated. "Simple" laboratory and/or shop polymer modifications were not considered herein as synthesis.

b. Only off-the-shelf materials would be utilized. This restriction was later broadened during the Program to include research materials which had achieved or could achieve rapid pilot plant and/or production status.

c. Formulated compound(s) raw materials' costs were not to exceed approximately \$100.00 per pound. Although critical NASA requirements, especially in the area of flammability resistance, have necessitated (and are still doing so) the use of materials which cost \$500.00 to \$1500.00 per pound, a lower cost was essential if wider usage by cost conscious hardware-oriented designers was to be accomplished.

d. Emphasis was placed upon the development of elastomeric and/or flexible formulations. This was due to the practical problem of a) achieving the mechanical stability necessary for compound/component bond integrity, and b) protecting the component from shock and vibration resulting from process/installation procedures, as well as operational environments.

It should be noted that the Statement of Work shown in Appendix A is a modification of the original Program directive. Its changes reflect recent NASA and NASA contractors design philosophy and objectives.

SECTION 3

EXPERIMENT DEFINITION AND TASK SEQUENCE DEVELOPMENT

A graphic description of the Program Plan is shown in Figure 3-1. The phases noted reference the applicable sections of this report. The completion of a specific phase signified a major "Go-No Go" decision point for each formulation.

3.1 PHASE I: MATERIALS SELECTION

3.1.1 State-Of-The-Art Material and Testing Survey

In order to accomplish the Program's objectives within the prescribed constraints, it was deemed necessary to first assess the state-of-the-art primarily of non-flammable materials, and secondarily of fire retarding and quenching agents. Further, the pertinent processing, environmental and functional characteristics of potential materials were also to be sought. Since the binder was clearly the weakest link in the formulation chain, it was essential to contact organizations which were considered potential sources of applicable binder materials and/or data (see Section 4).

Through the course of the Program, accession of 327 pertinent documents was made. These reports covered such applicable areas as:

a. Materials (e.g. elasromers, non-flammable monomers and polymers, flame retarding additives).

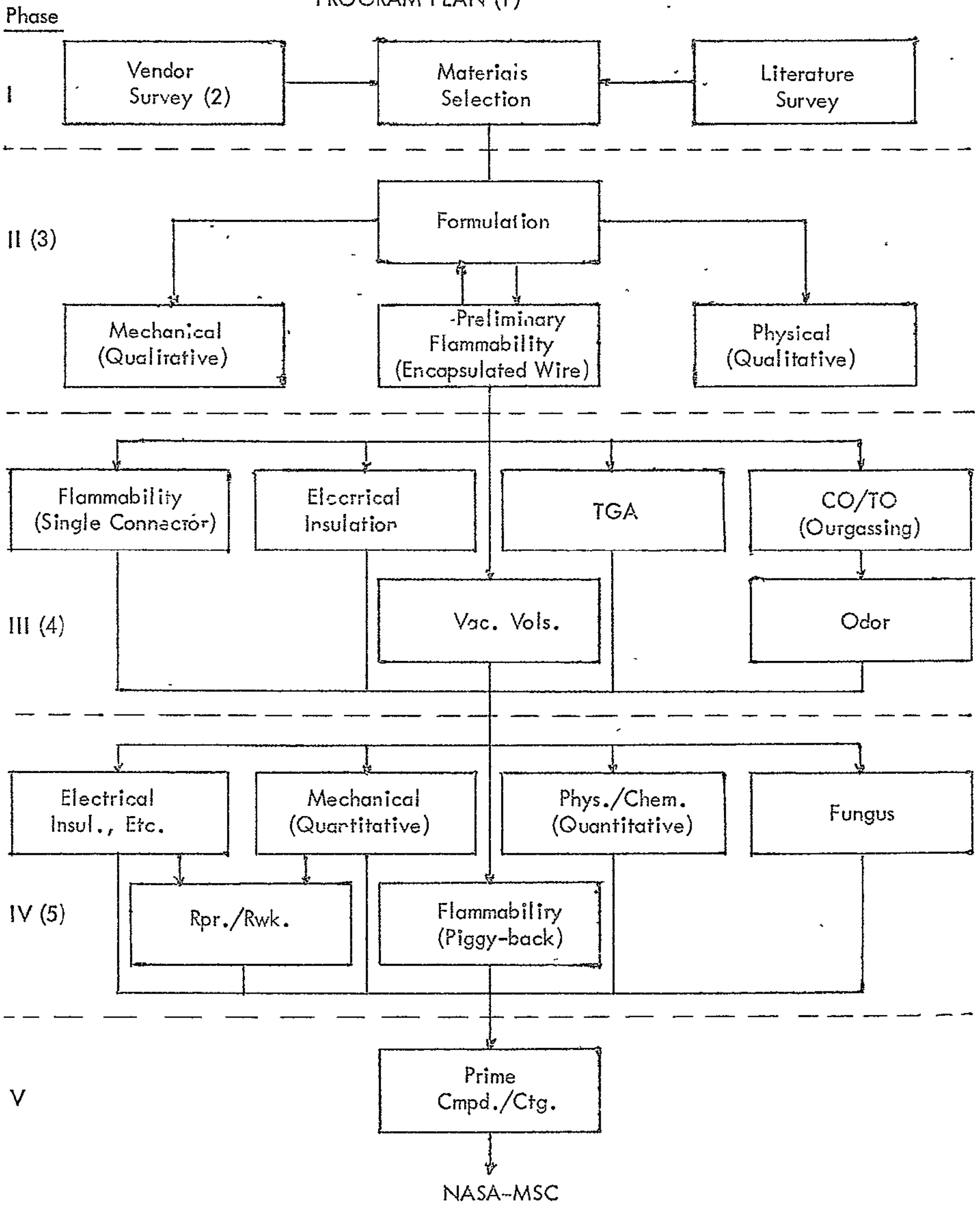
b. Testing/Properties (e.g. flammability, thermal and/or vacuum, outgassing, toxicity, thermal decomposition in high oxygen environments, environmental requirements for manned as well as unmanned space operations).

c. Applications (e.g. potting, encapsulating, wiring, printed circuits).

3.1.2 Material Selection

Based upon an analysis of the available data, the delineation of processing/functional requirements, and a determination of the relative significance of such information, candidate polymeric matrices and flammability reducing agents were selected for preliminary formulation. (See Section 6). Principal emphasis was on the minimization of hydrogen content (preferably by replacement with halogen atoms). Secondary consideration was given to minimum carbon. Unless otherwise noted in this report, the various formulation materials were used in the "as received" condition. Since many were vendor proprietary products, the chemical identification and quantity of their constituents were often not available.

FIGURE 3-1
PROGRAM PLAN (1)



1. Based on Statement of Work (Appendix A)
2. See Section 4.
3. See Section 6.
4. See Section 7.
5. See Section 8.

Although the number of elastomeric, or even flexible, polymers which are rapidly self-extinguishing is quite limited, it was deemed feasible, by judicious incorporation of additives, to utilize somewhat more flammable polymers. The rationale for the specific selection of each of the additives used in the Project's formulations has been reviewed in some detail in the Progress Reports, or are discussed in Section 6.1.1.2 of this report.

The formulations evaluated in this Program were generally based upon the arbitrary decision to initially utilize 150 phr of any flame retardant under consideration. A more relevant method to comparatively evaluate the various fillers would have been to utilize exactly the same particle size for each one, and to add sufficient quantity to achieve a given viscosity. However, it was clearly impractical to attempt to procure a specific particle size for most of the candidate fillers in terms of the Program budget and continuity. Further, conventional viscosity measurement techniques which would be pertinent to the generally high viscosities encountered with many of the formulations considered by the Project, could not have been utilized for the minimal weight quantities (e.g. approximately 10 grams) prepared for the preliminary flammability tests. Hence, a "standard" 150 phr filler content was used in most formulations. Wherever reasonably feasible, particle size was maintained below 170 mesh.

3.2 PHASE II: PRELIMINARY SCREENING

3.2.1 Formulation

Materials selected from the Phase I investigation were compounded in formulations which were then fabricated into preliminary flammability test specimens and qualitative physical and mechanical test strips.

3.2.2 Testing

3.2.2.1 Flammability - As previously noted, the order of testing precedence and the rejection of certain formulations were primarily predicated upon flammability resistance. Therefore, the initial consideration of any formulation was based upon its performance under ignition conditions in environments containing various partial pressures of oxygen and respective oxygen percentages in the total pressure environment. The criteria was non-flammability (or rapid self-extinguishability) in whichever test environment was being used at a given level of test (i.e. air, 6.2 psia oxygen, etc.).

The comparative flammability resistance of the various formulations were initially evaluated using a 1/4" x 1/4" x 1.0" molded specimen, each having a Teflon coated wire embedded in the center of the specimen, parallel to the long dimension. The mold for these specimens is described in complete detail in Section 5.2 (Ref. Appendix O). This preliminary flammability test is clearly a highly simplified version of the required test (see Appendix B). The advantages of thie preliminary test are as follows:

a. Rapid and low cost (labor and material) tests permit a significantly larger number of formulation variations to be evaluated.

b. A "rough" optimization of candidate formulations can be achieved prior to design-type functional data accumulation.

c. The preliminary tests specifically exclude all potentially flammable materials (i.e. other than candidate materials) from the test zone (e.g. neoprene insert of the Bendix pygmy connector) which could tend to obscure the flammability characteristics of the candidates.

The preliminary flammability tests were first performed in air during the early part of the Program. (A shift to starting at 6.2 psia oxygen was later carried out when sufficient familiarity with the formulations was established.) If found to be self-extinguishing or non-flammable in air (Ref. Appendix I) the test was repeated at 6.2 psia oxygen (Ref. Appendix J). If the material was self-extinguishing or non-flammable, the test was repeated in 16.2 psia 60% oxygen/40% nitrogen (Ref. Appendix K). Success at this level advanced the test to 16.5 psia oxygen (Ref. Appendix L). A material was removed from further preliminary flammability testing if the formulation was found to be non-self-extinguishing, regardless of test environment. Subsequent revision of the Statement of Work upgraded the 60/40 test from 16.2 to 16.5 psia to more closely coincide with the then applicable spacecraft environment. This increased the oxygen partial pressure from 9.72 to 9.9 psia. Testing in this revised environment was still performed via Appendix K, but with appropriate changes in pressures.

3.2.2.2 Mechanical/Physical Properties (Qualitative) - All data shown in the various tables of this report which are identified with the sub-title of "Characteristics" were developed from thin strip specimens (see Section 5.3). Degree of cure was also based upon the molded preliminary flammability specimen. Due to the small size of the strip specimen, testing was limited to those forces simply provided by the hands and between the fingers. For example, crack resistance was carried out by folding the strip in half so that the crease was in the width dimension, and the faces met within approximately 1/4 in. from the crease line. While in the folded condition, a crack propagation evaluation was then performed by making a small slit in the convex surface of the fold with a scalpel. Such tests, while being extremely simple and fast to run, are clearly quite subjective by nature and, hence, were only indicative of a trend and/or were a gross approximation of a property.

3.3 PHASE III: FINAL SCREENING

The results of Phase III, consisting of those tests shown in the applicable portion of Figure 3-1, are discussed in complete detail in Section 7. The tests selected for this phase were based upon the widest evaluation (i.e. flammability, electrical properties, space environmental and thermal effects, and toxicity) with the minimum number of tests.

The electrical properties can usually be qualitatively deduced from theoretical considerations of the base polymer's structure, and its skeletal constituents. However, the inclusion of additives sufficiently complexed the analysis so that empirical data were required at this screening level.

It should be noted that the odor test was not conducted until the "safe" concentration level was established during the carbon monoxide/total organics (CO/TO) determinations were made.

3.4 PHASE IV: PRODUCT CHARACTERIZATION

This phase, as shown in Figure 3-1, involved additional tests which provide preliminary design data. This additional data increased the confidence level in the usage of the final selected formulation. For example, the cured materials are exposed to mechanical loads when a) they are handled and shipped, b) installed, c) launched, d) relocated, and e) used to position other materials (e.g. wire harnesses). Since it is not practical to initially test materials to each and every load environment anticipated, various basic tests were used to categorize the materials for subsequent material selection and specific test evaluation by applicable M&P/design staffs. These tests included tensile, elongation, tear, and bond strengths.

The "piggy-back" flammability test was conducted during this phase to determine the effect of one connector's ignition upon an immediately adjacent connector. The results of this test are discussed in Section 8.3.1 and the test is described in Appendix A, Section 3.4.1.2.

Since realistic utilization of the final material may involve some processing and fabrication errors by the electrical component's manufacturers and assemblers, it was considered advisable to determine the feasibility of rework and repair techniques with the material. The techniques were verified by fabrication of a typical repair and determining the quality of the repair with a moisture/resistance test.

3.5 SYSTEMS AND PROCEDURES

3.5.1 Identification

Source and batch data were maintained for all formulations and processing materials. A number was consecutively assigned to each formulation. Individual batches and duplicate specimens of a given formulation were also noted via this technique. For example, 003-05-12 designates the twelfth specimen prepared from the fifth batch of formulation system number 3. Test data were keyed to these numbers.

3.5.2 Data Retention.

Each formulation and flammability test was recorded and filed as an individual unitized package so that access to all data for each was immediately available.

SECTION 4

MATERIALS AND TESTING SURVEY

To accomplish the aforementioned objectives within the program constraints, it was necessary to assess the state-of-the-art of non-flammable materials, and of fire retarding and quenching agents. In addition, the processing, environmental, and functional characteristics of potential materials were also sought. Since the elastomeric, low temperature curing binder is clearly the weakest link in the formulation chain, contacts were established with organizations which were considered potential sources of applicable binder materials and/or data. Also, testing experience of other organizations than the Contractor, especially with respect to flammability, was simultaneously investigated. As a result of these contacts, those organizations whose activities appeared to warrant a more detailed conference were visited. The resultant information is presented in Appendix G of this report.

Subsequent actions with each of these organizations is briefly noted below:

1. NASA - White Sands Test Facility: The connector flammability, odor, outgassing, and TGA tests were conducted by WSTF for this program.
2. NASA - MSC: Program monitoring by MSC was maintained via visits, telecons, and Progress Reports.
3. Rice University: It became apparent that it was not realistic to pursue their direct fluorination route during the course of this program since the "art" was still quite preliminary.
4. Peninsular Chemresearch: As a result of various telecons, it was evident that their extensive (also Pierce) line of fluorine chemicals were generally not applicable to direct polymer formulation. (However, they appeared to be an excellent source of precursors for synthesis of potentially non-flammable polymers.) Nor were they particularly interested in the HFPO patented by DuPont. However, they were prepared (also Pierce) to consider a fluorinated polymer development sub-contract oriented toward the needs of this program. They indicated that a minimum of 2 years would probably be involved. Further, development cost would be high (CNR development being cited as a typical example). Since this was outside the scope of this program, the subject was dropped.
5. NASA - MSFC: No directly applicable polymer technology or data available.
6. NASA - Goddard: Ceramic-based evaluation materials never received.

7. DuPont - Chestnut Run: The Viton formulations were experimentally pursued in some depth. Although no further action could be taken with respect to procurement of HFPO, its potential with epoxies is still evident.

8. US Navy: No further action.

9. Grumman: Their use of glass frit was explored in some Furane formulations.

10. Wright-Patterson AFB: Monsanto Research was contacted concerning perfluorostyrene. Cost at this time (development laboratory quantities) was considered excessive. AFML Viton sealant formulations (similar to those suggested by DuPont) were examined experimentally.

11. 3M: Considerable attention was given to the L-2344 polymer during the program. None of the -COOH terminated Fluorel was available.

12. NASA - Ames: Their primary materials development interest in intumescent-type fire retardants was not too pertinent herein.

It should be noted that a considerable number of other organizations were effectively dealt with via telecon (e.g. Dow Corning and the silicone division of General Electric). The results of these investigations are documented for those interested.

SECTION 5

SPECIAL APPARATUS AND TEST PROCEDURES

5.1 FLAMMABILITY TEST FACILITY

The Project's initial concept for the flammability tests, equipment and facilities, was predicated upon the use of conventional glass reaction flasks as the basic test chamber. In addition, portability was essential to the coordination of tests with the sub-contracror who was scheduled to perform some of the other tests. It became rapidly clear that a more sophisticated design and construction was necessary in order to optimize safety, reproducibility, a high rate of testing (i.e. over 500 specimens were ultimately tested with this facility during the conduct of the program), and cost. As a result, a considerably greater effort was expended on this task than had been previously estimated. Further, subsequent arrangements with the White Sands Tesring Facility of the NASA Manned Spacecraft Center to conduct outgassing tests obviated the need for portability.

A cylindrical section of a surplus vacuum processing system was utilized as the basic test chamber. (Expendable and/or surplus materials and components were used wherever possible to minimize cost.) This structure had various porrs which were used with only minor modifications. The entire facility and system design was then reoriented around this cylinder. Figure 1 (Appendix H) is a block diagram of the final unit and the subsystems of which it is comprised. The interconnecting distribution systems for the electrical power, environmental test atmospheres (oxygen and nitrogen), purging (nitrogen), vacuum, and venting systems are specifically shown in Figure 2 (Appendix H). Tables 1 and 2 of Appendix H further identify materials and components of the test facility. Numerous brass, stainless steel, iron, and copper fittings and tubing were utilized in the assembly of the system. Flexible pressure lines (e.g. Flexline) were also used in order to simplify assembly.

The remaining pertinent design details of the system can be seen in Figures 3 through 13, inclusive, of Appendix H. However, several items worthy of additional comment are:

1. The primary pressure gage, -26 (see Figure 2 of Appendix H), was recalibrated to assure precise pressure readings within the gage limits (i.e. 0.025 psia/div.). Since the gage was used as an absolute pressure indicator, its case was kept evacuated. This was found difficult to achieve in practice due to a very minute, but finite, leakage in the case connections. It was found more expedient to attach the case to the vacuum pump via a vacuum valve, -21 (see Figure 2 of Appendix H), so that the case could be periodically evacuated.

2. Due to the anticipated enormous number of specimens to be tested, cycling speed was an important design parameter. To this end, a manual 4.0 in. quick-opening vacuum valve is the seal on the loading port. The general procedure is to prepare a connector and attach it to P/N 11600 (Figure 7 of Appendix H). The potted connector and its holder are then held by one hand, and are inserted into the chamber via the loading port. The holder is mechanically located onto the specimen holder support plate (Figure 8 of Appendix H), with the aid of the banana plugs. The specimen's wires are then connected to the center bolts of P/N 11500 (Figure 6 of Appendix H), for power. The hand is then removed from the 4.0 in. valve, the valve is closed, and the test can be commenced. The same arrangement can be utilized for both the preliminary and "screening" connectors. The preliminary 1/4 x 1/4 x 1 specimens are mechanically held by their leads to the terminals.

3. An appreciable amount of time was devoted to minimizing system leaks. All pipe threads were sealed with Teflon tape. The exposed excess tape was then trimmed away and the exterior of the connection was then coated with RTV 3140 silicone elastomer (Dow Corning), a one-part moisture curing, methyl alcohol producing system. AN connections were simply tightened and coated with RTV 3140.

Due to the obvious dangers inherent in the test (e.g. high oxygen content, over-ambient pressurization, potential toxicity of decomposition products, etc.), system and procedural safety were part of the fundamental design criteria for the test facility. For example, it is seen from Figure 2 of Appendix H that the relief vents of the low pressure oxygen (-14) and nitrogen (-13) regulators are vented into the "outside" vent line. This eliminates possibility of venting toxic gases near operating personnel. Other safety measures included:

1. A light which is activated by the pressure switch so that personnel in the vicinity of the system will be immediately aware of any pressurization downstream of the O_2 (-16) and N_2 (-15) globe valves.

2. A 12-25 micron filter, (-27) which is to assure that the entire pressurization system, especially the gage (-26) and the regulators, are given some protection in the event that excessive chamber pressure tries to force 12-25 micron particulate matter upstream. Such particles could readily be present in the chamber from the pyrolysis of filled formulations.

3. A rupture disc. Initially, commercially available 0.0006 in. aluminum foil (Reynolds Wrap) was used. Four successive runs with dry nitrogen resulted in rupture at 18.9, 19.5, 19.7 and 19.6 psia; certainly satisfactory for our purposes. (In order to carry out these runs, the relief

valve, -29, was closed off since it opened at approximately 16.8 psia and reclosed at approximately 16.2 psia.) The above thickness of foil was found to develop hairline cuts after several runs. These cuts were obviously in the pattern of the honeycomb core support disc. The damage seems to have resulted from the up and down cycling of the foil onto the core edges during the flammability test runs. After replacing the foil several times, it was decided to use an 0.0011 in. aluminum foil. This significantly increased the rupture disc life. However, it was desired to reduce the failure rate even further so as not to interrupt test series. In order to do so without becoming involved in either machining out the core caviry, or even worse, machining the core down to a smaller thickness so that a non-flammable (e.g. ceramic) coating could be applied to the core edges, it was decided to place a very thin porous screen between the foil and core. A 100 mesh brass screen was selected and is presently in use between the honeycomb core and the foil.

4. A check valve (-29) set to "crack" at 2.5 psi above ambient to back up the manual relief valve (-18).

5. A mirror set at an angle so that the specimen can be observed through the Pyrex window (8.32 in. dia. clear sight).

6. All flushing done with nitrogen; no air permitted in system due to relative ease of having some amounts of organics (e.g. oils) carried into the chamber by the air, even with very good filters.

7. Testing was conducted in outside covered-over area to assure a practical level of air circulation for the vented effluent.

8. Substitution of Viton 'O' rings and gaskets wherever possible, for the usual types (probably Buna N) found in or designed for many of the components which can be exposed to pure oxygen.

9. Oxygen cleaning. Upon completion of fabrication, the entire system was assembled for "fit." It was then disassembled to the extent that all materials and components which would, or conceivably could be, exposed to a high oxygen environment could then be oxygen cleaned. The system was then reassembled, checked out, and put into operation.

The procedures for flammability testing in "air," 6.2 psia oxygen, 16.2 psia 60/40 oxygen/nitrogen, and 16.5 psia oxygen with this facility are shown in Appendices I, J, K, and L, respectively. The components referenced in these procedures are shown on Drawing No. 10010C, included as Appendix M. A typical copy of the test data sheet is also provided for further clarification and information (see

Appendix N). The entire facility is shown in Figure 5-1. In general, the facility was designed so that its operational limits and procedures could closely simulate those specified in MSC-D-NA-0002, Tests 5 and 17.

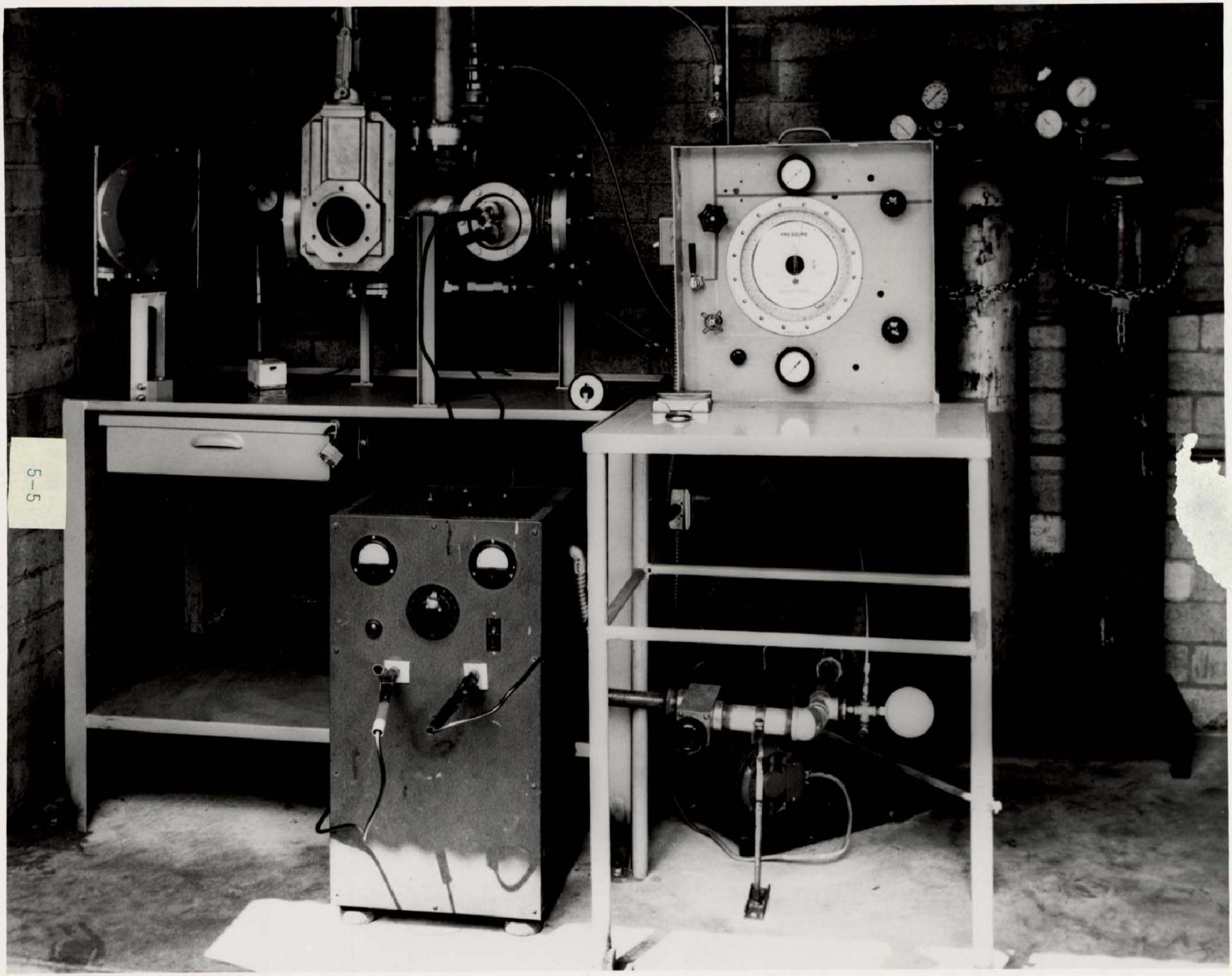
The equipment was put "on stream" with formulation F-001 (see Table 6-49). Figure 5-2 shows the test specimen being inserted into the chamber. The installed specimen is seen in Figure 5-3. Two specimens were tested in air to verify the basic operational procedure. This was extended to 6.2 psia oxygen with another specimen from the same formulation. The data for these specimens are given in Table 6-50. The testing of the fluorocarbon formulations was then commenced in both air and oxygen in order to clear up remaining bugs in equipment and procedures.

5.2 FLAMMABILITY TEST SPECIMEN MOLD

In order to conduct a preliminary evaluation of the comparative flammability resistance of the large number of formulations anticipated, a simple molded specimen configuration was required; one which did not utilize such non-recoverable hardware as expensive connectors, printed circuit boards, etc. Several designs were considered based upon whether the ignition wire was to be embedded within the specimen or whether it was to be peripherally attached. The former would be more difficult to fabricate but would more closely simulate the specimen required for MSC-D-NA-0002, Tests 5 and 17. The latter would be simpler to produce and would be similar to the silicone RTV igniter sometimes used for flammability testing in pure oxygen. The embedded type was finally selecred because of its relationship to the above Tests 5 and 17, specified in the Work Statement.

A four cavity die design was prepared. The simplest (and lowest cost) design of this type would have been based upon simple milled slots in a Teflon (for release) block. It was decided to design the die so that each cavity could be completely disassembled. The advisability of this was in line with the possible poor cures with polymeric species which might result from some of this program's formulations. Thus, even low cohesive strength specimens could be prepared and removed with minimum damage. This also permits shorter mold turn-around time. It was also considered advisable to keep the spacing between the cavities as small as possible to minimize the quantity per "shot" of experimental and/or costly materials. The die sketch is shown in Figure 1 of Appendix O and the Bill of Materials is tabulated in Table 1 of Appendix O.

The removable cavity segments are shown in Figure 5-4. The entire unassembled mold is seen in Figure 5-5. Figures 5-6 and 5-7 show the AWG 20 Teflon insulated wires, used for ignition and attachment, in various stages of assembly prior to insertion of a compound. A cured set of test specimens are seen in Figure 5-8 before demolding. Typical variations in physical quality of resultant specimens are graphically discernible from Figures 5-9 and 5-10. (Variations can occur due to such variables as curative, cure time and/or temperature, or intrinsic cohesive/adhesive strengths.)

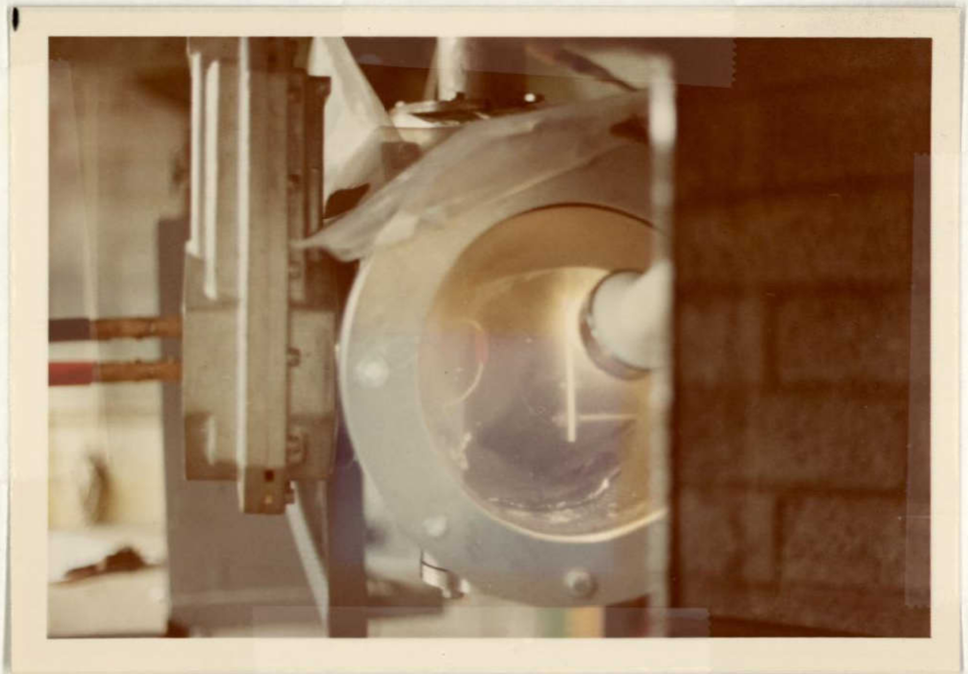


5-5

FIGURE 5-2

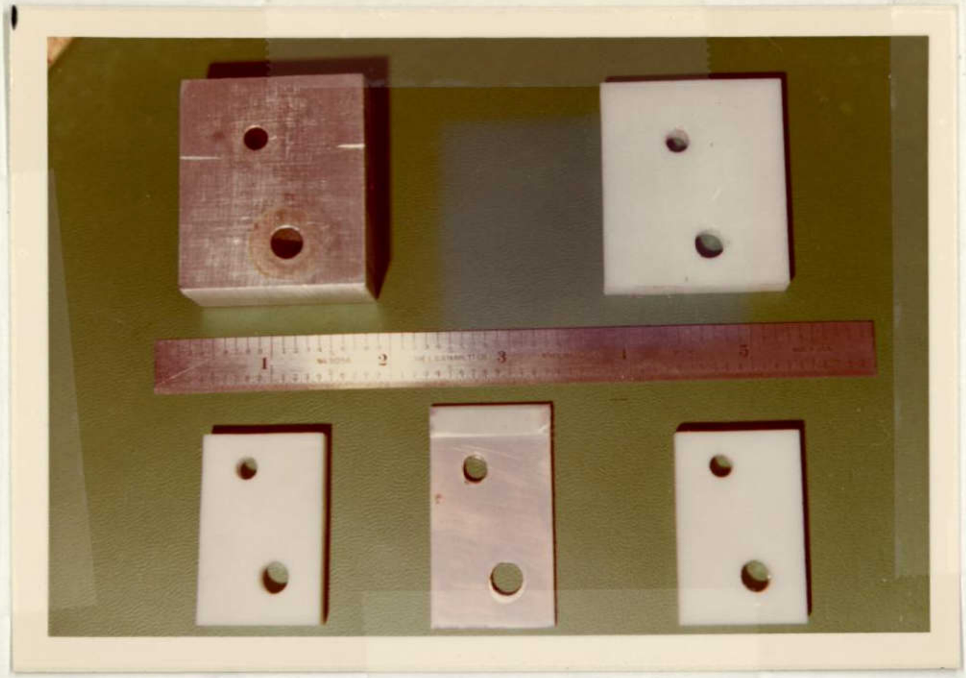
FLAMMABILITY TESTER: SPECIMEN INSERTION

FIGURE 5-3



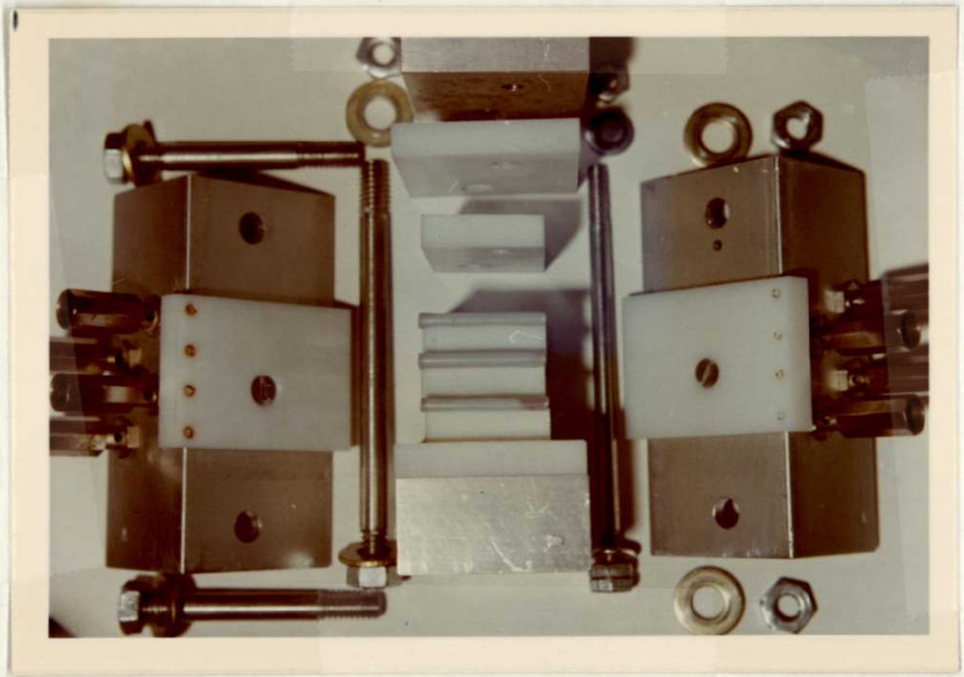
FLAMMABILITY TESTER: TEST IN PROGRESS
(Red Specimen: F-001)

FIGURE 5-4



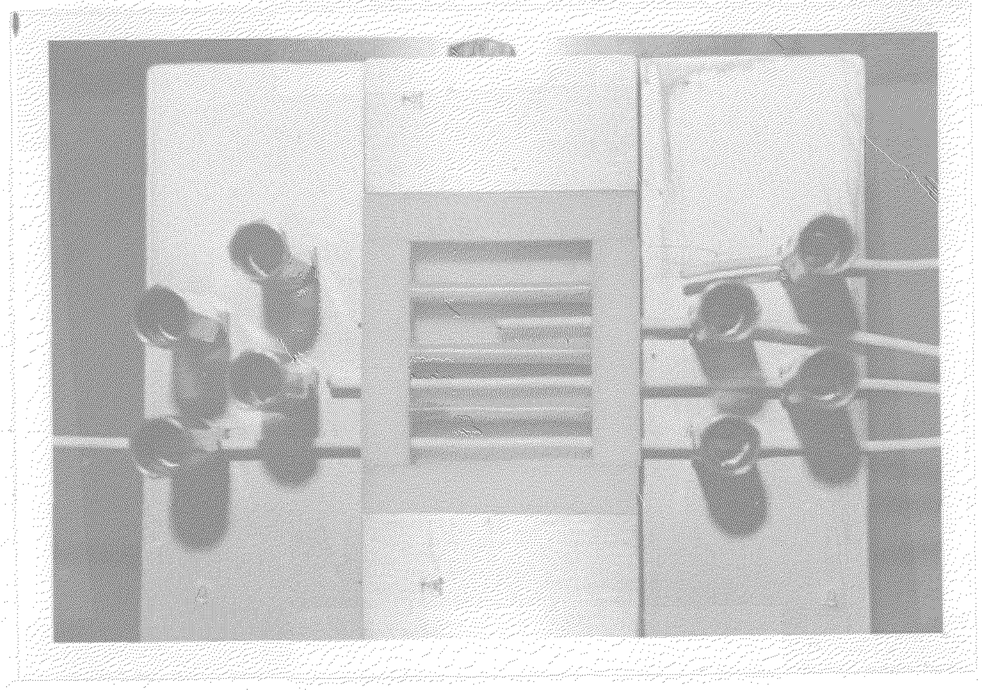
FLAMMABILITY SPECIMEN MOLD: REMOVABLE CAVITY SEGMENTS

FIGURE 5-5



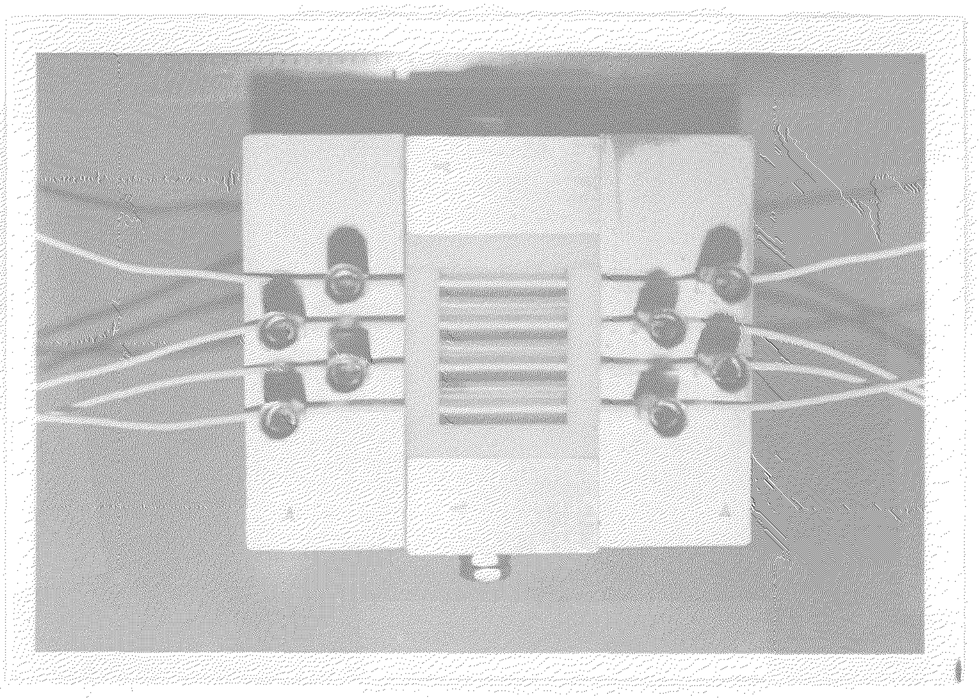
FLAMMABILITY SPECIMEN MOLD: UNASSEMBLED

FIGURE 5-6



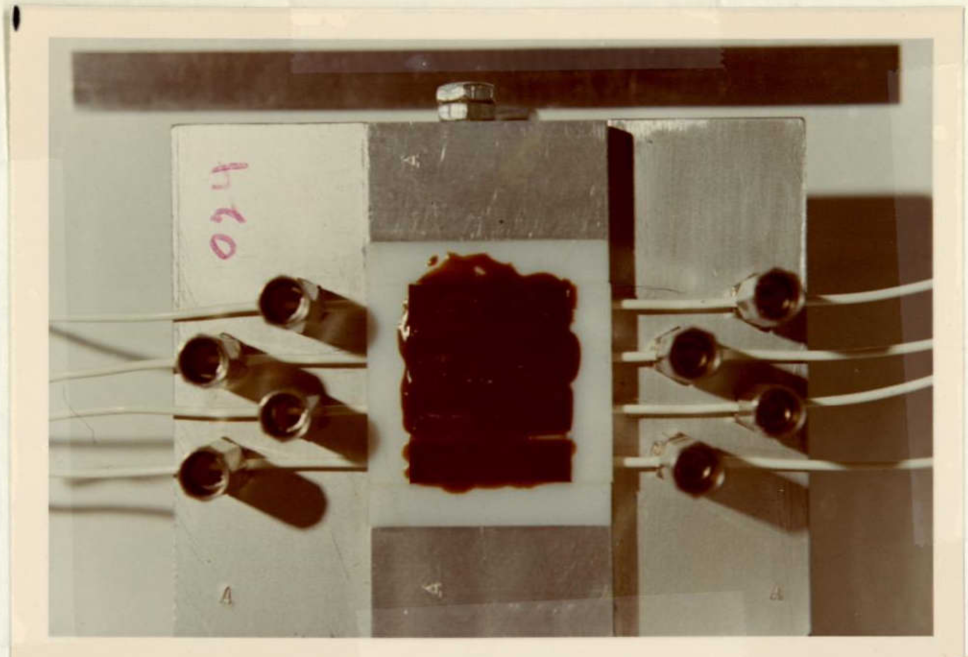
FLAMMABILITY SPECIMEN MOLD: IGNITION WIRES PARTIALLY INSERTED

FIGURE 5-7



FLAMMABILITY SPECIMEN MOLD: IGNITION WIRES INSERTED AND CLAMPED

FIGURE 5-8



FLAMMABILITY SPECIMEN MOLD: CURED F-024 IN PLACE

Figure 5-9

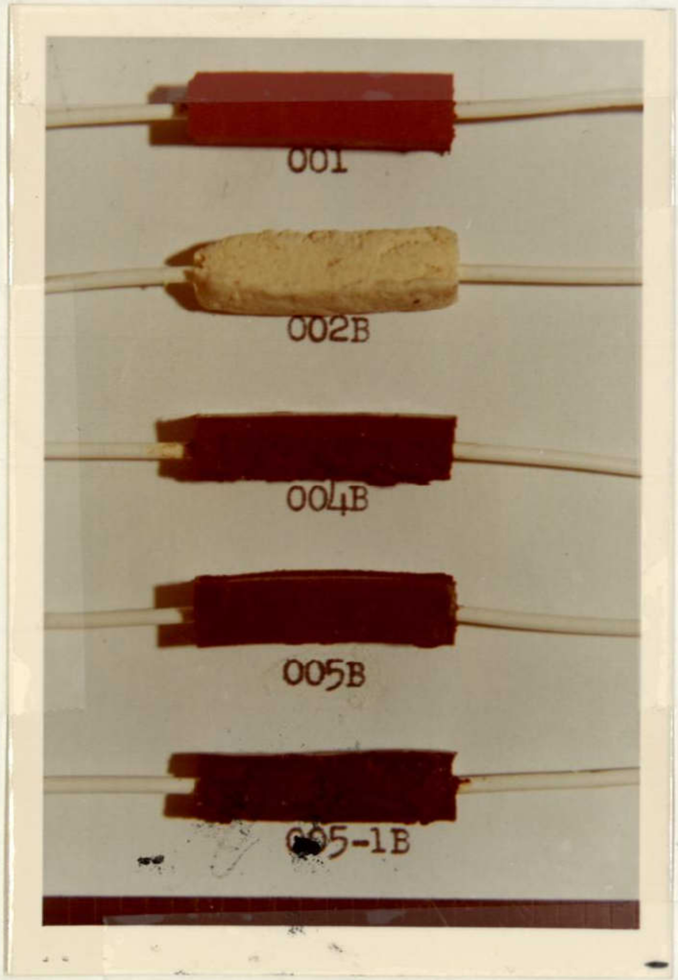
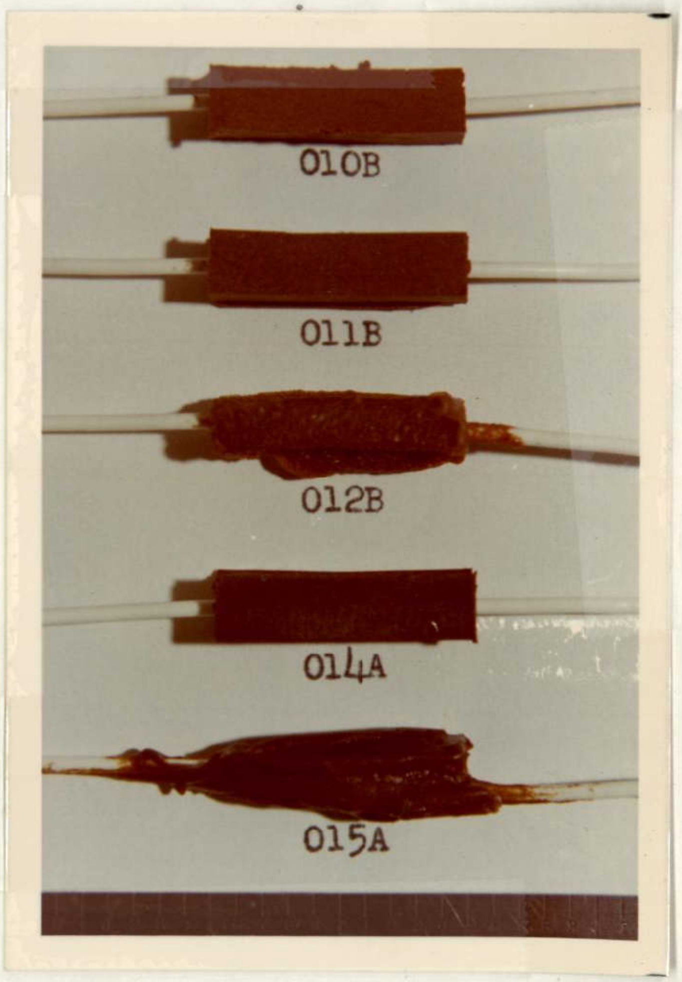


Figure 5-10



FLAMMABILITY SPECIMEN (TYPICAL)

Two units of the mold were fabricated. Several "dry" runs were carried out with the aforementioned formulation F-001. All operations, including cure, were initially performed at room temperature. No mold difficulties were encountered. (These specimens were those also used for check-out of the Flammability Tester since it is essentially the same formulation used as the initiator for several flammability tests, including the "piggy-back" test, Appendix A, para. 3.4.1.2).

Subsequent mold usage indicated that it fully met all design expectations except in one regard. Delamination of the tape from the stainless steel spacers eventually occurred in the specimen area. A careful examination of molded specimens revealed a concave contour on each side of the specimens. Apparently there is sufficient air pressure developed in the delamination area to withstand the potting material's thermal expansion (note: not a closed mold). The more recent specimens evidence very little difference in this contour, suggesting that a relatively constant deformation now exists. Even if it is assumed that configuration and dimensions can affect a specific flammability test, comparative evaluations can still be made with these specimens as long as the contours remain constant. On the other hand, the delamination permits a compound to creep under the tape. Attempts to completely remove it would probably cause serious damage to the tape and require its immediate replacement. Leaving the compound in could conceivably cause inhibition of other compounds. For example, residue of an amine cured fluorocarbon elastomer could inhibit the cure of such addition reaction systems as the Sylgard (Dow Corning) silicone RTV's. However, we did not see the necessity of making any mold changes. It should be noted that two options were available to correct the problem: (1) the steel could be periodically retaped, or (2) it can be coated with a baked Teflon finish. The former being simplest and cheapest, was utilized. It also did not cause a noticeable mold "down time." On the other hand, this solution is obviously only a temporary expedient; delamination eventually occurring. This, in turn, again caused variations in specimen contours until this new delamination also stabilized. Although we were concerned with this problem, it should be reiterated that the overall performance of the mold was excellent.

Complete disassembly, clean-up, reassembly of the mold, as well as wire insertion, proved to be quite rapid, generally 15-20 minutes. A mold having a completely uncured mess was recycled in less than 30 minutes. Since 4 specimens were produced in each cycle, the average specimen time was certainly reasonable.

The relative difficulty in part removal was usually indicative of degree of cure since this is a function of cohesion and tack. It was noted that a well cured formulation demolded either completely clean or resulted in some minor particle pick-off by the mold. The latter results from low cohesion or from a non-homogeneous mass (i.e. filler deposited on the mold surface).

5.3 STRIP MOLD

It was deemed advisable to fabricate a thin specimen of each formulation in order to obtain a better qualitative "feel" for handling characteristics, flexibility, memory, crack initiation and propagation, tear strength, etc.. To this end, a number of very simple and inexpensive strip molds were fabricated from aluminum sheet, pressure sensitive Teflon tape, and spring-loaded paper clips. The resultant specimens were approximately 3-1/4 in. x 3/4 in. x 0.065 in.

SECTION 6

NONFLAMMABLE FORMULATIONS DEVELOPMENT: PRELIMINARY SCREENING

6.1 FLUOROCARBON POLYMERS

Extensive NASA experience (Ref. 6-1) has shown the greater intrinsic effectiveness of this class of polymers to resist burning in a "high" oxygen environment than most other polymers having an organic backbone. In addition, they are either already commercially available, or can readily be made so. Two classes of these polymers were considered within this program: a) vinylidene fluoride/perfluoropropene copolymers, and b) di-hydroxy terminated vinylidene fluoride/perfluoropropene copolymers. (Fluorosilicone RTV's are arbitrarily separately classed herein purely for convenience of data presentation.)

6.1.1 Vinylidene Fluoride/Perfluoropropene Copolymer

These are essentially the Vitons (S-1: See Table 6-1) and Fluorels (S-2). For example, the eight commercially available Vitons vary in molecular weight and, hence, viscosity (see Table 6-2).

TABLE 6-2

VISCOSITY OF VITONS (Ref. S-1)

Grade	Viscosity (Mooney at 212°F)	
LM	2,000 cps (212°F)	
C-10	10	(ML-10)
A-35	35	(ML-10)
B-50	50	(ML-10)
A	67	(ML-4)
D-80	80	(ML-10)
B	117	(ML-4)
A-HV	180	(ML-4)

There are also variations in the ratio of vinylidene fluoride to perfluoropropene. Since a solventless system is desired, the one with lowest viscosity was the most attractive. Unfortunately, it is also the least reactive. A further complication is provided by the thermal process limitation of 150°F (due to the possible presence of thermally sensitive electronic components).

TABLE 6-1
MATERIAL SOURCES

S-	Source
1.	E. I. duPont de Nemours
2.	3M Co.
3.	Merck & Co., Inc.
4.	U. S. Borax Research Corp.
5.	Stauffer Chemical Co.
6.	Shell Chemical Co.
7.	General Electric Co.
8.	Union Carbide Corp.
9.	McKesson Chemical
10.	Matheson, Coleman and Bell
11.	Philadelphia Quartz
12.	Dow Corning
13.	Furane Plastics, Inc.
14.	Swift & Co.
15.	Mobile Chemical Co.
16.	Hooker Chemical
17.	U. S. Borax
18.	M & T Chemicals, Inc.
19.	Buckman Labs.
20.	Harshaw Chemical
21.	Corning Glass
22.	Zonolite Div., W. R. Grace & Co.
23.	Harrison & Crosfield
24.	Standard Industrial Minerals
25.	FMC Corp.
26.	Continental Oil Co.
27.	Chemical Div., Pittsburgh Plate Glass Co.
28.	Michigan Chemical
29.	Alfa Products, Ventron Corp.
30.	K & K Labs., Inc.
31.	El Monte Chemical Co.
32.	Arrowhead Puritas
33.	Microbeads Div., Cataphote Corp.
34.	Anderson Development Co.
35.	Mallinckrodt Chemical Works
36.	Fisher Scientific Co.
37.	General Chemicals Div., Allied Chemical & Dye Corp.
38.	Braun Chemical
39.	Humphrey Chemical Corp.
40.	Monsanto Co.
41.	F. D. Davis & Co.
42.	Marco Div., W. R. Grace & Co.
43.	Diamond Shamrock Chemical Co.
44.	Norac Co.
45.	Chemetron Noury Corp.
46.	Velsicol Chemical Corp.
47.	Erickson Research Chemicals

Although temperatures in excess of 150°F are not permitted by the Work Statement for component processing environments, pre-blending of formulation ingredients could be carried out above 150°F. In fact, pre-contractual efforts indicated the feasibility of reducing injection and/or extrusion viscosity of such thermoset compounds (hence, increasing processing rate) via a "hot gun" technique. This would be contractually permissible as long as the exudate did not contact the components at a temperature in excess of 150°F. Pre-catalyzed, one package systems could then be prepared. Package stability for a minimum of 6 months could probably be achieved, if necessary, by sub-normal temperature storage.

A qualitative estimate of the handling (i.e. viscosity) characteristics of such polymers was therefore obtained at various temperatures in order to establish initial polymer selection. (See Table 6-3). The Viton LM and the L-2344 were clearly usable at these temperatures; both could be poured, as well as pre-blended and injected at 200°F or below.

Although the L-2344 had the lower viscosity of the two, its cure is significantly different from that of the conventional fluoropolymer due to its unusual reactive hydroxyl terminal groups. Emphasis was initially applied to the Viton LM and the L-2344 was dealt with at a later date. However, as noted above, the low temperature processing characteristics of the Viton LM results from its comparatively low molecular weight; its classification as a wax rather than an elastomer may be more appropriate. Cured Viton C-10 would be expected to have intrinsically better mechanical properties than LM, as evidenced by its higher molecular weight than LM. Although C-10 was not found pourable below 250°F, it could be blended or injected at 200°F. Carrying this concept one step further, it was not deemed unrealistic to consider pre-blending a stronger, but high temperature processable fluoroelastomer (e.g. Viton A-35) at temperatures above 250°F, and inject below 200°F. These higher molecular weight types would also be expected to be more reactive than LM.

6.1.1.1 Unfilled Formulations - Considerable formulation and testing by H. F. Kline of NASA-MSC (Ref. 6-2) verified that Viton LM is comparatively unreactive. Their compounds blended even as high at 200°F, or cured at 150°F for seven days, or even up to 400°F resulted in unsatisfactory cures. It should be noted that MSC found that it was possible to achieve a non-flammable mixture with several of these incompletely cured formulations. Hence, initial Furane formulations also included some modifications of Mr. Kline's systems; such modification being based upon in-house technology.

It is well established in the literature that an acid acceptor is necessary for achieving an effective state of cure, regardless of catalytic system used. Typically, magnesium oxide is used for this purpose. Some of the MSC formulations contained Maglite Y (S-3), a commercial grade of magnesium oxide. Some contained zinc oxide, another, but less used acid acceptor. Maglite Y is perfectly suitable for

TABLE 6-3

THERMAL PROCESSING OF FLUOROELASTOMERS & FLUORORESINS

Feasibility to ______________ @ ______________ °F (1)

Grade	S	Pour					Blend (2)					Inject (3)				
		197	249	300	350	400	197	249	300	350	400	197	249	300	350	400
Viton LM	1	X	X				X	X	O	O	O	X	X	O	O	O
L-2344	14	X	X				X	X	O	O	O	X	X	O	O	O
Viton C-10	1						X	X	X	X	X	X	X	X	X	X
KF-2142	14										X	X	X	X	X	X
KF-2146	14										X	?	X	X	X	X
Viton A-35	1										?	X	X	X	X	X
Viton B-50	1											X	X	X	X	X
KF-800	14										?				?	X
KF-2143	14															?
Viton D-80	1															
KF-2140	14															
KF-3700	14															

(1) ± 2°F.
(2) By hand
(3) By air gun
S. . . . . Source (See Table 6-1)
X. . . . . OK to use
O. . . . . Not tested

"normal" Viton formulations which require mill rolling for blending of ingredients, storage life for catalyzed but uncured stock, and are cured above 150°F and post-cured above 400°F. A higher reactivity MgO was considered essential to the low temperature cure conditions prescribed by this program. Table 6-4 shows that Maglite D (S-3), being 2-3 times as reactive as Maglite Y, should accelerate HF elimination.

TABLE 6-4

ACID ACCEPTORS REACTIVITY
(Magnesium Oxide, Commercial Grade)

MAGLITE (S-3)	MgO (%)	Density (gm/cm^3)	Reactivity (Iodine No.)	Cost ($/lb./500 lb.)
K	94.5	3.5	25, max.	.295
L	93.0	3.45	25-30	.365
Y	94.0	3.5	35-55	.305
M	89.0	3.3	75, min.	.35
D	90.0	3.32	100, min.	.285

Another approach was to use calcium oxide instead of, and in conjunction with, magnesium oxide. Water, which results from the HF scavenging action of the acid acceptor, remains in the fluoroelastomer, especially if thick sections are involved (e.g. potted connectors), and if low cure/post cure temperature are required. Presence of this water tends to reverse the preferred reaction direction, hence, preventing a better cure. The importance of water removal or deactivation has been clearly demonstrated (Ref. 6-3). It has been found (Ref. 6-4) that calcium oxide provides the following three advantages over magnesium oxide in Viton formulations:

a) Less shrinkage during cure,
b) Absence of voids in thick sections, and
c) Higher state of cure.

The above advantages can be attributed to the fact that the calcium oxide not only reacts with the HF, but it also readily forms a thermally stable hydrate. It is then not necessary for the water to be eliminated in order to remove it from the reaction sites. Since it remains, the total mass stays relatively unchanged, minimizing shrinkage (and voids at higher temperatures; "blowing" action of the water also can occur). (If used alone, this dual service of calcium oxide suggests the need for higher quantities than would be indicated by its HF reaction alone.)

The inactivation of water and simultaneous incorporation of boron was also attempted by using trimethoxyboroxine (methyl metaborate). The reaction with six moles of water releases three moles of methyl alcohol which boils at 65.65°C (148.4°F), just below the cure temperature "limit." The remaining boric acid somewhat ties up 4.5 moles of water of the six original ones. It was easily dispersed, having a viscosity of 15-100 cps at room temperature for the commercial grade (S-5) and a viscosity of 19.0-19.3 cps for the research grade (S-4). It was considered herein as a reactive diluent.

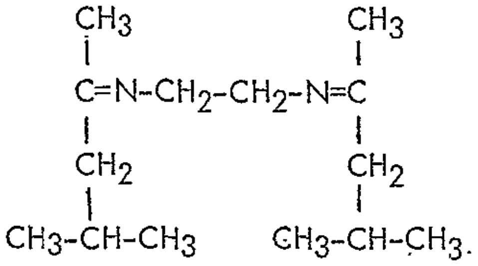
The possibility of increasing the rate of dehydrofluorination of the fluoroelastomer by increasing the quantity of acid acceptor was also examined in the desire to compensate for the lower cure temperature and greater quantity of non-fluoroelastomer additives which were present. Since many such additives are also hydrated or water producers (e.g. most borate, boric acid, some silicates, etc.), additional calcium oxide, or an equivalent, was called for to reduce any inhibition of HF elimination.

The selection of type and quantity of fluoroelastomer catalyst is another area which was evaluated. For example, the aforementioned work by NASA-MSC was essentially based upon a cure system designed to allow Viton LM to be used in a flame resistant sealant formulation recommended by duPont (Appendix G). It involves the use of 5 phr Epon Curing Agent H-1 (S-6) and 1 phr tetraethylene pentamine. It would appear that the H-1 is to provide storage life, while the pentamine is to accelerate the reaction when it does proceed.

The appropriateness of such a catalytic system for the program was analyzed with respect to three factors:

a) Cure rate,
b) Degree of cure, and
c) Fuel source.

The H-1 is designated by its manufacturer as being a "reactive Ketimine" (Ref. 6-5). They define this type of ketimine as one "...having a high free amine content...". Their other two ketimines, H-2 and H-3, are reported as being "essentially pure" ketimines. Although the manufacturer does not stipulate which amines and ketones were used to form these compounds, this information can be deduced with considerable reliability from available data. It is reported (Ref. 6-6) that H-2 is a ketimine of ethylene diamine and methyl isobutyl ketone.



The manufacturer states that the available weights of polyamine (i.e. amount of reactive polyamine released when the curing agent is completely reacted with water) are 39 and 27% respectively, for H-1 and H-2 (Ref. 6-5). A value of 26.8% is calculated for the above presumed structure. This would represent one mole of the amine per two moles of the ketone. If an additional mole of the amine is included to "...having a high amine content...", the value is 38%. This would indicate that the H-1 is a 1:1 mole ratio of ethylene diamine and MIBK. It is then seen that the catalytic system contains an immediately available, moderately reactive polyamine with two primary, and three secondary amines, a highly reactive polyamine with two primary amines but which are only available at a rate determined by the availability or release of water from the other constituents (e.g. absorbed water, water from the HF plus MgO reaction, etc.), and a highly flammable ketone. If, as discussed above, it is granted that "loose" water will slow the fluoroelastomer crosslinking, the formulation ingredients should be pre-dried. Therefore, the only source of "loose" water will be from the HF reaction. Since this is dependent upon the moderately reactive pentamine, it is evident that the cure rate at 150°F, or less, will not be as rapid as it would be if a comparable amount of the ethylene diamine were used alone. Of course, a significant increase in the pentamine can also increase the cure rate by purely statistical reasoning. For example, duPont reported to this Project that the above sealant formulation only has a three minute "pot life" at 150°F when catalyzed by three phr of the pentamine alone. This represents six moles of primary amine (see Table 6-5) while five phr of H-1 plus one phr of the pentamine is equivalent to (5) (.39) (2) + (1) (2) = 5.9 moles of primary amine. It appears that the rate may be more dependent upon availability than upon the differences between the two polyamines.

It should be noted that the existence of the pentamine's secondary amines was deliberately ignored in comparing the above time dependency of the two amines. It has been determined (Ref. 6-7) that the rate and final extent of HF elimination is a function of the type of amine (i.e. Pri. $\gg$ Sec. $>$ Tert.). The data also suggests that only the primary amine causes complete crosslinking. In addition, the same report also states that the difunctional agents are faster curing and require lower cure temperatures. Based upon these cure characteristics, it was considered possible to replace the ketimine with a primary polyamine whose quantity is balanced between the need for sufficient "pot life" and the necessity for a more complete cure.

The "fuel source" factor, as a function of hydrogen, is amply demonstrated by calculation of hydrogen present, based upon the data in Reference 6-1, Table 3-1-1. A simple determination of per cent hydrogen per mole would lead one to the erroneous conclusion that very little difference exists in the hydrogen content of the polyamines shown in Table 6-5. Even considering the hydrogens to total amines ratio, there would be very little basis for selection. However, when only the primary amines are considered, it is seen that the tetraethylene pentamine (TEPA) represents almost a threefold hydrogen penalty as compared to ethylene diamine. In the same manner, it is found that the H-1 has 9 hydrogens/primary amine, or almost 2.5 times the hydrogens per primary amine as the pure ethylene diamine (EDA).

TABLE 6-5

POLYAMINES

Name	Formula	Boiling Point (1) (°C/760mm)	Pressure (1) (mmHg/20°C)	Amines Present			Mol Wgt. (1)	Hydrogen (2)		
				Pri.	Sec.	Tert.		per Mole (%)	per Amine	per Pri. Amine
Ethylene Diamine	$H_2NC_2H_4NH_2$	117	10	2	--	--	60.10	13.3	4.0	4.0
1,3-Diaminopropane	$H_2NC_3H_6NH_2$	139.7	3	2	--	--	74.13	13.5	5.0	5.0
Propylene Diamine	$CH_3CH(NH_2)$-CH_2NH_2	120.9	8	2	--	--	74.13	16.2	6.0	6.0
Diethylene Triamine	$(H_2NC_2H_4)_2NH$	206.9	<1	2	1	--	103.17	12.6	4.3	6.5
Triethylene Tetramine	$H_2N(C_2H_4NH)_3H$	277.4	<0.01	2	2	--	146.24	12.3	4.5	9.0
Tetraethylene Pentamine	$H_2N(C_2H_4NH)_4H$	340 (d)	<0.01	2	3	--	189.31	12.2	4.6	11.5

1. "Chemicals and Plastics Physical Properties--1968", Union Carbide Corp.
2. Calculated.

Specifically, 5 gm. of H-1 is equivalent to (.39)·(5) = 1.95 gms. EDA, 1 gm. of TEPA has a primary amine equivalence to EDA of 60.1/189.3 ÷ .317 gm. EDA. Therefore, an equivalent amount of EDA primary amine for the H-1/TEPA mixture would be 2.267 phr.

Calculation of the hydrogen present in a basic fluoroelastomer and its catalyst placed this concern over the hydrogen content in more perspective. For example, a formulation of Fluorel 2140 and several catalytic systems are shown in Table 6-6 with their respective hydrogen content.

Several observations can be made from Table 6-6. First, the use of the TEPA and ketone-containing ketimine results in a 19.8% increase in hydrogen content when compared to the EDA catalyzed system. Secondly, the high level of catalyst concentration in the "sealant" is clearly demonstrated by the comparable weight of DIAK 1. Normally, this catalyst is ued at a level of 1.0 to 1.75 phr. in "typical" fluoroelastomer recipes which are given "typical" cure/post cure cycles (i.e. to 400°F). This led to the conclusion that simply increasing the catalyst concentration without modifying the other ingredients of the basic sealant formulation probably would not improve the state of cure.

TABLE 6-6

CATALYZED FLUOROELASTOMER
VS. HYDROGEN CONTENT

	Formulation 1			Formulation 2			Formulation 3		
	Cmpd.	Hydrogen		Cmpd.	Hydrogen		Cmpd.	Hydrogen	
Component	(gm)	(%)	(gm)	(gm)	(%)	(gm)	(gm)	(%)	(gm)
F-2140 (S-2)	100	2.0	2.0	100	2.0	2.0	100	2.0	2.0
H-1	5	12.7	0.635						
TEPA	1	12.2	0.122						
EDA				2.27	13.3	0.302			
DIAK 1 (S-1)							6.04 (2)	10.0 (1)	0.604
Total H (gm)			2.757			2.302			2.604

1. M.W. = 160, 16 hydrogens.
2. (160/60.1) = 6.04 gm.

Based upon these considerations, a series of formulations were prepared to optimize cure of the Viton LM. In addition, those Viton LM formulations considered as non-flammable by MSC were prepared as a basis of comparison and to assess processing difficulties. Details of the processing, and the resultant qualitative mechanical and physical properties of this series, shown in Table 6-7, have been presented in the various Progress Reports for this Program. In general, the formulation blending was carried out by hand at temperatures up to 200°F. They were not degassed, were cured at 150°F, and were usually given a 150°F postcure (i.e. not in mold). Upon the addition of acid/water acceptors the viscosity was such as to be barely pourable at 150°F. When flame retardants were also incorporated, mechanical force (e.g. spatula) was needed to distribute the compound, even at 150-200°F. Cured specimen configurations consisted of the preliminary flammability type (see Appendix O) and the thin strips.

Formulation 019 was found to have the best balance of mechanical properties. This formula demolded satisfactorily after 25.5 hours at 150°F, but was slightly tacky. After 7 additional days at room temperature, the specimens were significantly more flexible than other MgO or CaO specimens, did not crack in 180° bend, and were no longer tacky. The clearly superior flexibility of this formulation is seen in Table 6-8 and Figure 6-1.

TABLE 6-8

CRACK RESISTANCE
(180° Bend)

Spec. No.	Acid Acceptor (phr) MgO (1)	CaO	Cure at 150°F (hrs)	Results (% Cracked)
005A	15	---	15	100
005-1A	15	---	47	75
019A	7.5	10.4	25.5	0
008	---	20.8	24	100
008-1A	---	20.8	47	100

(1) Maglite D

It is seen from Table 6-9 that except for the NASA-MSC suggested formulations (i.e. flame retardant filled), the flammability resistance of the "cured" polymer itself is satisfactory in air, but is unquestionably affected by only 6.2 psia oxygen. The filled formulations, however, were significantly more resistant to burning.

6.1.1.2 Flame Retarded Formulations - Based upon the above Viton LM polymer investigations, F-019 was primarily used as the binder for evaluating the flame retardancy effectiveness of various additives. As in the case of the other "polymer" systems considered by the Project, it was necessary that the additives incorporated with the LM complied with such selection criteria as:

TABLE 6-7

FLUOROELASTOMERS

Formulations (phr)

Component	S	002	020	003	004	005	005-1	009	006	008	008-1	010	007	019	011	012
Viton LM	1	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
ZnO	9	5.6	11.1	11.1									30.2			
Maglite-Y	3				15.											
Maglite-D	3					15.	15.	22.5						7.5	15.	15.
MgO,hvy.USP	3								15.							
CaO	10									20.8	20.8	31.2		10.4	20.8	
TMB (1)	4															10.8
Boric Acid	9	122.3	100	100												
SS 65 PWD	11		11.1													
Metso Anh.48	11			11.1												
FS-1265	12		11.1	11.1												
X001 (2)	13	6.7	6.7	6.7	6.7	6.7	6.7	6.7	6.7	6.7	6.7	6.7	6.7	6.7	6.7	6.7
H-1	6															

S .... Source (see Table 6-1)
1. Trimethoxy Boroxine
2. Tetraethylene Pentamine (TEPA): H-1/1:5 by weight

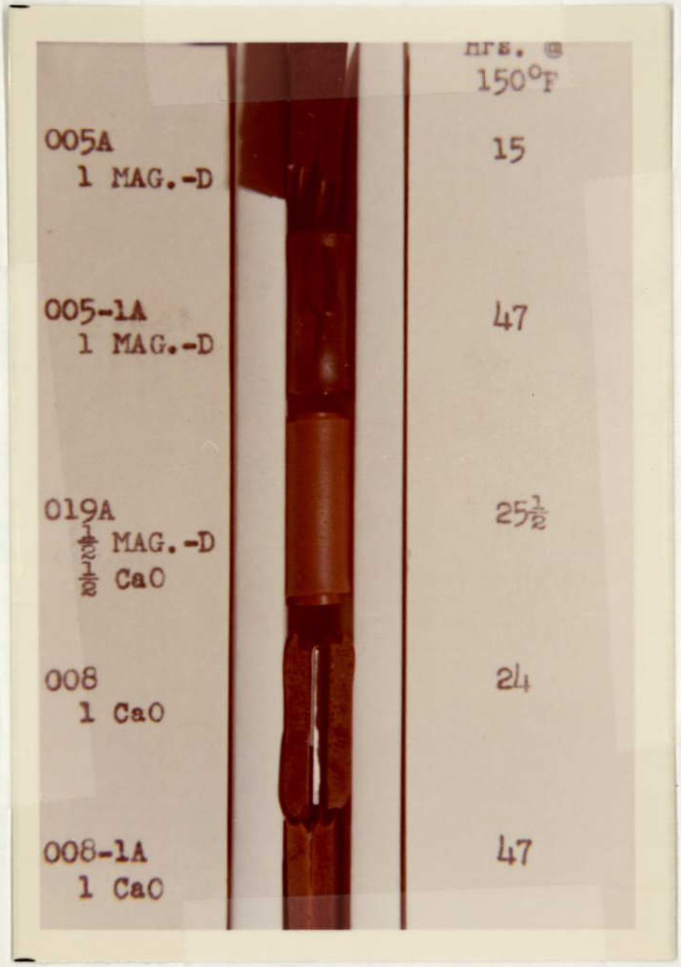
TABLE 6-7(cont'd)

FLUOROELASTOMERS

		Formulations (phr)											
Component	S	013	014	039	015	024	027	016	025	017	052	018	053
Viton LM	1	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.
Maglite-D	3	15.	15.	15.	15.	15.	15.	15.	15.	15.	15.	15.	15.
TEPA (3)	8	12.95	7.15	5.36									
EDA (4)	8				2.27	3.41	6.82						
DTA (5)	8							3.9	5.85				
Hexameth. Dia. (6)	10									4.39	6.59		
Hexameth.Tetra.(7)	10											5.30	10.60

3. Tetraethylene pentamine
4. Ethylene diamine
5. Diethylene triamine
6. Hexamethylene diamine
7. Hexamethylene tetramine

FIGURE 6-1



180° BEND TEST

TABLE 6-9
FLUOROCARBON ELASTOMER PRELIMINARY FLAMMABILITY -TEST CONDITIONS/RESULTS-

			Specimen											
			002B	020A	020B	003A	003B	004B1	004B2	005B	005-1B	009B	006	006
	O_2 Pressure (psia)		6.5	(7)	6.2	(7)	6.2	(7)	(7)	(7)	6.2	(7)	(7)	6.2
	Wire Ignition (1)		T/B	T/B	T/B	T/B	T/B	T	T/B	T/B	T/B	T/B	T/B	T/B
R	Wire Failure (1)			T	T	B	T	T	T	T	B	B	T	B
E	Smoke (2)		N	N		N	N	N	N	N	N	S	N	N
S	Burn Time (sec.) (3)	flame-out	0	0	38	0	4	0	2	1	30	3	0	45
		glow-out	0	0	38	0	4	0	2	1	38	3	0	55
U	Configuration (4)		Y	N	Y	Y	Y	Y	Y	Y	Y	Y	Y	N
L	Residue (%) (5)		100	100	90	100	100	100	100	100	90	100	100	50
T	Melted (6)		Y	Y	N	N	N	N	N	N	N	N	N	N
S	Color	exterior	white	white	black	white	white	brown	brown	brown	black	brown	brown	gray
		interior	"	"	white	"	"	"	"	"	"	"	"	"
	Exterior Texture	powdery (6)	N	N	N	N	N	N	N	N	N	N	N	Y
		crusty (6)	N	N	Y (8)	N	N	N	N	N	Y	N	N	N

1. T.....Top B.....Bottom
2. N.....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. Ambient pressure air

TABLE 6-9(cont.)
FLUOROCARBON ELASTOMER
PRELIMINARY FLAMMABILITY
-TEST CONDITIONS/RESULTS-

		Specimen												
		008	008-1B	010B	007	007	019A	019B	011B	012B	014A	014B	039A	039B
O_2 Pressure (psia)		(7)	6.2	(7)	(7)	6.2	(7)	6.2	(7)	(7)	(7)	6.2	(7)	6.2
Wire Ignition (1)		T	T/B	T/B	T/B	T/B	T/B	T/B	T	T/B	T/B	T/B		T/B
Wire Failure (1)		T	B	T	T		T	T	T	T	T	B		T
Smoke (2)		S		N	N		S		N	N	S		P	
Burn Time (sec.) (3)	flame-out	0	42	1	0	36	4	158	0	0	2	30	0	105
	glow-out	0	145	1	0	107	4	176	0	0	2	152	0	110
Configuration (4)		Y	Y	Y	Y	N	Y	N	Y	N	Y	N	Y	Y
Residue (%) (5)		100	50	100	100	40	100	30	100	100	100	20	90	80
Melted (6)		N	N	N	N	N	N	N	N	Y	N	N	N	N
Color	exterior	brown	white	tan	lt.tan	gray	brown	gray	brown	brown	brown	white	black	wht-gray
	interior	brown	gray	tan	lt.tan	gray	brown	gray	brown	brown	brown	white	black	black
Exterior Texture	powdery (6)	N	N	N	N	Y	N	N	N	N	N	N	N	N
	crusty (6)	N	Y	N	N	Y	N	Y	N	N	N	Y	Y	Y
			(8)			(8)		(8)				(8)		(8)

1. T.....Top B.....Bottom
2. N.....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. Ambient pressure

TABLE 6-9(cont.)
FLUOROCARBON ELASTOMER
PRELIMINARY FLAMMABILITY
-TEST CONDITIONS/RESULTS-

			Specimen											
			015A	015B	027A-1	027A-2	027B	016A	016B	025-A	025-B	017-B	017-C	052
	O_2 Pressure (psia)		(7)	(7)	(7)	(7)	6.2	(7)	6.2	(7)	6.2	(7)	6.2	(7)
RESULTS	Wire Ignition (1)		T/B	T/B	T/B	T/B	T/B	T/B	T/B	T	T/B	T/B	T/B	T/B
	Wire Failure (1)		T	T	T/B	T	T	T	T	T	B	T		T
	Smoke (2)		N	N	N	S	N	S		S	N			
	Burn Time (sec.) (3)	flame-out	0	0	0	4	150	1	50	0	24	0	40	0
		glow-out	0	0	0	4	161	1	86	0	109	0	110	0
	Configuration (4)		N	Y	Y	N	Y	Y	N	Y	Y	Y	N	Y
	Residue (%) (5)		100	100	100	90	90	100	70	100	90	100	60	100
	Melted (6)		Y	N	N	Y	N	N	N	N	N	N	N	N
	Color	exterior	brown	brown	brown	brown	white	brown	black	brown	black	brown	white	brown
		interior	brown	black	black	black	black	black	black	brown	black	black	black	black
	Exterior Texture	powdery (6)	N	N	N	N	N	N	N	N	N	N	Y	N
		crusty (6)	N	N	N	N	Y (8)	N	Y (8)	N	Y (8)	N (8)	Y (8)	N (8)

1. T.....Top B.....Bottom
2. N.....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. Ambient pressure

a. Minimum weight.
b. Minimum volume (i.e. to minimize effects on functional properties and other environmental response characteristics than flammability).
c. Particle size and distribution.
d. Vacuum stability.
e. Thermal stability: non-fugitive or migrating (except if migration required for flammability reduction - see mechanism d, below) and remains integral with matrix upon pyrolysis.

For example, additives were considered of interest to the Program if they could provide any of the following mechanisms.

a. Produce such quenching gases as water vapor, carbon dioxide, nitrogen, halogens, and halo-compounds.
b. Thermally-induce intumescence: bicarbonates.
c. Increase thermal diffusivity; beryllium oxide, magnesium oxide.
d. Cause fusible inorganics to migrate: tetraborates.
e. Create large thermally induced endotherms (low temperature activation).

Detailed selection criteria were established for each of these mechanisms. Thus, the requirements for the quenching gas producing additives generally involved the following:

a. Non-flammability,
b. High specific heat to assist in the dissipation of input heat flux (similar to the "blowing factor" in ablation),
c. Maximum gas volume per unit original weight,
d. Non-toxic, -volatile, or -odorous,
e. Amenable to rapid removal by life support system subsequent to a fire.

These quenching gases could be obtained via such actions as:

a. Thermal decomposition of the polymer itself.
b. Thermal decomposition of additives (e.g. solids having water of crystallization such as:

    1. Asbestos (up to 20% by weight of water).
    2. Unexpanded vermiculite.
    3. Inorganic hydrated salts.
    4. Some borates.

c. Thermally induced phase changes of additives (e.g. halogenated liquids and iodine crystals going to their gaseous form).

It was believed that such "quenching" additives could be incorporated into the basic polymer matrix by:

a. Direct blending: halogenated liquids, solid microparticles, etc.
b. Molecular sieves (Linde) (represents a weight-penalty; does not provide flammability resistance, compound binding, reinforcement, etc.).
c. Microencapsulation (NCR or SRI types) (hydrated inorganic or fluoropolymeric encapsulants were considered preferable in order to reduce the weight penalty).
d. Complexing with other components (additives, curatives, etc.) (e.g. phosphorous based curatives for halogenated epoxies, water of crystallization complexed with thixotropic inorganics, etc.).

The possibility of increased efficiency from synergistic effects was also evaluated. For example, it is well known that antimony oxide in conjunction with a halogen provides a greater flammability reduction than an equivalent weight of either used separately.

Some fire retardants which provide multiple benefits were also explored in the Program. For example, a modified barium metaborate (Busan 11-M1: Buckman Laboratories) also improves the elastomer's fungus resistance.

Considerable analysis was given (Ref. 6-8) to the selection of inorganic borates and silicates for use herein with Viton LM.

The specific formulations wherein potentially flame retarding additives were blended with a base stock of Viton LM, is shown in Table 6-10. Table 6-11 is a tabulation of the processing utilized for each of these formulations, based upon the coding shown in Table 6-12.

TABLE 6-12

PROCESS CODE

1. Add all ingredients together, except for catalyst(s).
2. Preheat all ingredients, except catalyst(s) to ____°F.
3. Preheat mold(s) to ______°F.
4. Add the catalyst(s).
5. Mix for ______min. at ______°F.
6. Cure in mold ______hr/min @ ______°F.
7. Post cure ______days/hr. @ ______°F.

The qualitative mechanical properties of the more interesting of the formulations are given in Table 6-13. The flammability test results are shown in Table 6-14.

The addition of 100 phr of boric acid resulted in specimens which could now survive the 6.2 psia oxygen, but were extremely boardy and brittle. The incorporation of 30 phr of Krytox 143 AC improved the processing by reducing the viscosity slightly. The latter specimen showed moderate resistance to burning in the 60/40 environment.

TABLE 6-10

FLUOROELASTOMERS WITH FLAME RETARDANTS

		Formulations (phr)											
Component	S	237	238	239	240	243	244	245	246	247	248	249	253
Viton LM	1	100.											
Maglite D (1)	3	7.5											
Calcium oxide	10	10.4											
F-237-1	13		117.9	117.9	117.9	117.9	117.9	117.9	117.9	117.9	117.9	117.9	117.9
Boric acid (2)	9		100.	100.	100.			100.	100.	100.	100.	100.	100.
SS 65 PWD (2,3)	11			15.									15.
METSO ANH.48 (2,4)	11				15.								
KRYTOX 143 AC (7)	1		30.	30.	30.					30.	30.	30.	30.
X001 (5)	13		6.7	6.7	6.7		6.7		6.7	10.05	6.7		13.4
H-1	6												
DTA (6)	8					5.85		5.85				5.85	

S......Source (See Table 6-1)
1. Magnesium oxide
2. 100% through 170 mesh
3. Anhydrous sodium silicate (ground glass)
4. Anhydrous sodium metasilicate
5. Tetraethylene pentamine (TEPA): H-1/1:5 by weight
6. Diethylene triamine
7. Halogenated liquid

TABLE 6-11

FLUOROELASTOMERS WITH FLAME RETARDANTS

- FORMULATION PROCESSING -

Formulation No. F-	Steps (2)									
	1	2	3	4	5		6		7	
					(min)	(°F)	(hr/min)	(°F)	(d/hr)	(°F)
237	(1)	200	-	-	5	200	-	-	-	-
238	X	"	200	X	"	150	63/50	150	15/1	150
239	X	"	"	X	10	"	24/35	"	-	-
240	X	"	"	X	"	"	24/-	"	-	-
243-1	-	"	-	X	5	"	95/05	"	-	-
243-2	-	"	-	X	4	"	2/13	"	2/22	150
244	-	"	-	X	5	"	4/15	"	"	"
245	X	"	-	X	"	"	94/10	"	-	-
246	X	"	-	X	"	"	96/05	"	-	-
247	X	"	-	X	"	"	93/35	"	-	-
248	X	"	-	X	"	"	95/30	"	-	-
249	X	"	-	X	"	"	93/15	"	-	-
253	X	"	200	X	10	"	24/-	"	14/-	150

1. Predried MAGLITE D and CaO 1 hr. @ 200°F
2. See Table 6-12.

20

TABLE 6-13

FLUOROELASTOMERS WITH FLAME RETARDANTS CHARACTERISTICS

-CURED/POST CURED-

Properties	Formulation 238	239	240	243	244	253
Cured	Y	N	N	N	Y	N
Tacky	oily	N	N	N	N	oily
Flexible	NG	P	P	Y	Y	
Memory	NG	P	P			
Cohesion	NG	NG	NG			
Tear Resistance	NG	NG	NG			
Crack Resistance (1)	NG	NG	NG			
Crack Propagation (1)	NG	NG	NG			
Porous	Y	Y	Y			
Adheres to unclean alum. Foil cup	P	P	P	F	F	
Cheesy	S	(2)		(2)	N	(2)

Y.....Yes VS.....Very Slight S.....Slight N.....No
X.....Satisfactory F.....Fair P.....Poor NG.....Not Good

1. 180° bend (face-to-face contact)
2. Gummy

TABLE 6-14
FLUOROELASTOMERS WITH FLAME RETARDANTS
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

			Specimen							
			238-I-I	238-I-I	239-I-I	240-I-I	240-I-I	240-I-I	253-IB-I	253-IB-I	253-IB-I
	O$_2$ Pressure (psia)		9.72 (60%)	16.5	6.2	6.2	9.72 (60%)	16.5	6.2	9.72 (60%)	16.5
R	Wire Ignition (1)		T/B	T/B	T	T	T	T	T	T/B	T/B
E	Wire Failure (1)		T	B	T	T	T	T	T	T	T
	Smoke (2)		N	P	N	N	N	N	N	N	P
S	Burn Time (sec.) (3)	flame-out	26	20	0	0	6	10	2	15	36
		glow-out	26	33	0	0	6	10	2	21	36
U	Configuration (4)		Y	N	Y	Y	Y	Y	Y	Y	N
L	Residue (%) (5)		95	10	0	>99	>99	5	>99	90	10
T	Melted (6)		N	N	N	N	N	Y	N	N	Y
	Color	exterior	tan	black	tan	tan	tan	tan	tan	tan	gray
S		interior	"	"	"	"	"	"	"	"	"
	Exterior Texture	powdery (6)	N	N	N	N	N	N	N	N	N
		crusty (6)	Y	Y	N	N	N	N	N	Y	Y

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No

When 15 phr of Metso Anhydrous 48 (i.e. anhydrous sodium metasilicate) was also added, the specimen withstood 16.5 psia oxygen with only minor surface degradation. But all of these additives further increased the viscosity so that the formulation, even at 150°F required noticeable force to pack it into the molds. Consequently, no further formulation effort was conducted with Viton LM or even the more reactive Viton C-10.

6.1.1.3 Fluoroelastomer/Silicone RTV Formulations - The potential utilization of a low viscosity amine-cured silicone RTV such as RTV 602 (S-7) as a reactive diluent for Viton LM was investigated.

Although the RTV 602 is somewhat more flammable than other silicone RTV's under consideration (Refs. 6-1 and 6-9) for this application, it, and its methyl-phenyl analog, RTV 603, were formulated (see Table 6-15) with the higher viscosity, but less flammable Viton LM. The following areas of concern inherent in this approach were investigated:

a. Co-blending of the two polymers,
b. Compatibility with each other and with a mutually acceptable curing agent,
c. Possibility of a cure at or below 150°F,
d. Pot life and storage life,
e. Cured characteristics.

It was found that processing up to 350°F was feasible without causing significant degradation to either polymer (see Tables 6-3 and 6-16). However, the two did not appear to be adequately compatible when blended by hand stirring; separation in the liquid state being noted. (This does not preclude the possibility of achieving compatibility by other blending techniques.) Considerable difficulty was also encountered in keeping the RTV 602 from gelling too fast, and separating into two distinct phases from the LM. Processing details are provided in References 6-10 and 6-11. Cured characteristics were tabulated in Reference 6-11.

TABLE 6-16

THERMAL PROCESSING OF RTV 602

Temp. (°F)	Time (hrs/min)	Weight Loss (1) (gm)	(%)
150	2/-	0.00	0.00
200	8/-	0.02	0.10
250	4/-	0.02	0.10
300	2/-	0.04	0.20
350	2/40	0.05	0.25
400	3/45	0.07	0.35

(1) Original sample weight.... 20.15 gm.

TABLE 6-15

FLUOROCARBON/SILICONE RTV ELASTOMERS

		Formulations (phr)											
Component	S	050	051	026	028	030	029	031	032	037	038	040	041
Viton LM	1			100.	100.	100.	100.	100.	100.	100.	100.	100.	100.
RTV 602	7	100.	100.	100.	100.	100.	100.	12.	20.	20.	20.	20.	20.
Maglite D	3			15.	15.	7.5	7.5	7.5	7.5	7.5	7.5	7.5	7.5
CaO	10					10.4	10.4	10.4	10.4	10.4	10.4	10.4	10.4
X001 (1)	13			6.7	6.7	6.7	6.7	6.7	6.7				
SRC-05	7	.3	.2	.75	.75	.25							
H-1	6									6.7			3.4
H-2	6										6.7	10.1	3.3

S......Source (See Table 6-1)

1. Tetraethylene pentamine (TEPA): H-1/1:5 by weight

TABLE 6-15 (cont'd)

FLUOROCARBON/SILICONE RTV ELASTOMERS

Component	S	057	067	069	068	058	063	064	065	066
		Formulations (phr)								
Viton LM	1	100.	100.	100.			100.	100.	100.	100.
RTV 602	7	20.	40.	100.	40.					
RTV 603	7					100.	30.	30.	30.	30.
Maglite D	3	7.5	7.5				7.5	7.5	7.5	7.5
CaO	10	10.4	10.4				10.4	10.4	10.4	10.4
Boric acid	9		58.4		58.4					
XPI-158 (2)	4			100.						25.
H-1	6	5.						6.7	10.	10.
SRC-05	7					.2				
X001 (3)	13						6.7			
TEPA	8									

2. Zinc borate
3. Tetraethylene pentamine (TEPA): H-1/1:5 by weight

TABLE 6-17
FLUOROCARBON/SILICONE ELASTOMERS PRELIMINARY FLAMMABILITY
-TEST CONDITIONS/RESULTS-

			Specimen											
			031A	031A	031B	032A	032A	032B	037A	037A	037B	057-B-1	068	066
	O_2 Pressure (psia)		(7)	6.2	6.2	(7)	6.2	6.2	(7)	6.2	6.2	6.2	6.2	6.2
RESULTS	Wire Ignition (1)		T/B	T/B	T/B	T/B	T/B	T	T/B	T/B	T/B	T/B	T	T/B
	Wire Failure (1)		T	T	T	B	T	T	T	T	T	T	T	B
	Smoke (2)				N			N	S		S	S	S	P
	Burn Time (sec.) (3)	flame-out	31	86	81	16	85	90	3.5	30	100	33	130	25
		glow-out	31	86	88	16	85	101	3.5	85	103	93	178	116
	Configuration (4)		Y	N	N	Y	N	N	Y	N	N	N	Y	Y
	Residue (%) (5)		85	10	10	95	15	10	100	5	10	1	90	40
	Melted (6)		N	N	N	N	N	N	N	N	N	N	N	N
	Color	exterior	tan	gray	gray	tan	gray	gray	tan	gray	gray	off-white	white	white
		interior	black	gray	gray	black	gray	gray	black	gray	gray	off-white	black	
	Exterior Texture	powdery (6)	N	N	N	N	N	N	N	N	N	N	Y	N
		crusty (6)	Y	Y	Y	Y	Y	Y	N	Y	Y	Y	Y	Y

1. T.....Top B.....Bottom
2. N.....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. Ambient pressure air

Although interest in this system was further reduced when it was found that the presence of either boric acid or zinc borate with the RTV caused an almost immediate sharp increase in viscosity to what appeared to be a gelled and/or gummy state, the preliminary flammability test was run on both "filled" as well as "unfilled" specimens. It is seen from Table 6-17 that none could survive 6.2 psia oxygen.

6.1.2 Di-Hydroxy Terminated Vinylidene Fluoride/Perfluoropropene Copolymers. This material was of considerable interest due to its lower hydrogen content than conventional Fluorels and Vitons (1.4% as compared to 2.0%), its reactive terminal groups, its low viscosity, and its availability (see Appendix G).

6.1.2.1 Isocyanate Modified Formulations - Initial activity with the di-hydroxy terminated vinylidene fluoride/perfluoropropene polymer L-2344 centered around its known (see Appendix G) reactivity with isocyanates. The first formulation, F-137 (see Table 6-18), was cured via the following procedure:

1. The L-2344 was pre-heated to 200°F to facilitate dispensing when weighed.
2. The L-2344 and the isocyanate were weighed out together.
3. They were reheated to 200°F for 10 minutes and then blended for 3 minutes while still being heated at 200°F.
4. Cure: 12 hours at 150°F.
   Post Cure: 11 hours 10 minutes at 200°F.

The specimen did not seem to be cured after the initial cure cycle but it was uniformly brownish transparent (indicating apparent compatibility). It appeared cured after the post cure even though it had a very slightly tacky surface. (This gradually disappeared in air.) Although the resultant specimen had quite a few bubbles, it seemed to have very good tensile strength, good flexibility, elongation, and adhesion to an aluminum foil cup (1).

F-137 was modified (F-138) by the addition of 150 phr boric acid just prior to the cure cycle. In order to assure adequate blending of the filler into the polymer, it was necessary to reheat the mixture several times at 200°F for a total of 10 minutes. This time "cure" was obtained in only 6 hours at 200°F. The lack of tackiness after this cure schedule was probably due to the presence of the filler. The cured specimens (F-138-1B) were somewhat cheesy and did not evidence any of the good mechanical properties of F-137.

The excellent flammability results with F-138 are seen in Table 6-19; only slight surface degradation noted in vicinity of wire junctures with the compound, with

---

(1) Used in the "as received" condition (possible mold lubricant present).

TABLE 6-18

FLUOROCARBON/ISOCYANATE ELASTOMERS

Component	S	137	138	397	398	409	411	412
		Formulations (phr)						
L-2344 (1)	2	100.		100.	100.	100.	100.	100
F-137	13		100.					
TDI (2)	---	5.22		5.22	5.22	5.22	5.22	5.22
Boric acid (3)	9		150.					
Calcium oxide	10						5.	
$CaSO_4$, Anh.	---							5.
Catalyst T-9 (4)	---				0.5			
DTD (5)	7					0.4	0.4	0.4

S:.....Source (See Table 6-1)
1. Dihydroxy terminated fluoropolymer
2. Tolylene diisocyanate
3. 100% through 170 mesh
4. Stannous octoate
5. Dibutyl tin dilaurate

TABLE 6-18 (cont'd)

FLUOROCARBON/ISOCYANATE ELASTOMERS

Component	S	140	124	125	128	318	319	320	321
		Formulations (phr)							
L-2344 (1)	2	100.	100.	100.	100.	100.	100.	100.	100.
TDI (2)	--	26.42	47.62	87.37	129.8				
VIRCOL 82 (6)	15	50.	100.		100.				
BROMINEX 126 (7)	14			100.	100.				
9113 (8)	14					21.3			
9117 (8)	14						16.3	16.3	16.3
DABCO (9)	--							1.0	
TEA (10)	--								1.0

6. Phosphorous-containing polyol
7. Bromine-containing polyol
8. Isocyanate/bromine-containing prepolymer
9. Triethylene diamine
10. Triethylamine

TABLE 6-19
HYDROXYL-TERMINATED FLUOROCARBON WITH INORGANIC FLAME RETARDANT
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

		Specimen			
		138-1B-1	138-1B-2	138-1B-1	138-1B-1
	O_2 Pressure (psia)	6.2	6.2	9.72 (60%)	16.5
RESULTS	Wire Ignition (1)	T/B	T/B	T/B	T/B
	Wire Failure (1)	T	T	T/B	T
	Smoke (2)	N	N	N	N
	Burn time (sec.) (3) — flame-out	5	7	6	5
	Burn time (sec.) (3) — glow-out	5	7	6	5
	Configuration (4)	Y	Y	Y	Y
	Residue (%) (5)	100	100	100	100
	Melted (6)	N	N	N	N
	Color — exterior	tan	tan	tan	tan
	Color — interior	"	"	"	"
	Exterior — powdery (6)	N	N	N	N
	Exterior — crusty (6)	N	N	N	N
				(7)	

1. T.....Top B.....Bottom
2. N.....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. 60% O_2/40% N_2 = 16.2 psia

all specimens, even in 16.5 psia oxygen. Additional experimental effort was conducted to reduce the very high at 200°F and the unworkable at room temperature viscosity by using less flame restardant and/or dilution with low viscosity reactives. Further attempts were made to reduce the cure temperature to no more than 150°F. These efforts included formulations with various accelerators (e.g. stannous octoate).

Processing steps of the other formulations tabulated in Table 6-18 are delineated in Table 6-20, in accordance with the coding of Table 6-12.

The use of different accelerators did speed up the cure to different extents, but all seemed to cause a rapid increase in viscosity and an early gelation, thereby preventing evolution of volatile byproducts (i.e. CO_2 and water). Calcium oxide and anhydrous calcium sulfate were added to reduce the presence of water. This was done to minimize the production of CO_2 from the reaction of tolylene diisocyanate (TDI) with the water. Upon completion of this series, rapid gelation/porosity remained the one primary difficulty.

The addition of potentially reactive diluents to the TDI catalyzed L-2344 was next examined. These formulations are also described in Tables 6-18 and 6-20. The results were as follows:

F-140: Not cured
F-124: Partially cured.
F-125: Brittle.
F-128: Partially cured (brittle in areas, soft and uncured in other areas).
F-318: Not cured.
F-319: " "
F-320: " "
F-321: " "

6.1.2.2 Silane Modified Formulations - Both reactive and non-reactive diluents were considered for the L-2344. This section concerns itself solely with the possible utilization of such silanes as shown in Table 6-21. The following section reviews the Project's activities with other diluents.

TABLE 6-20

FLUOROCARBON/ISOCYANATE ELASTOMERS

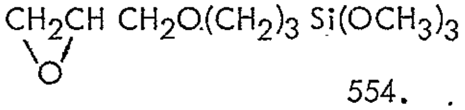
- FORMULATION PROCESSING -

Formulation No. F-	Steps (3)									
	1	2	3	4	5 (min)	(°F)	6 (hr/min)	(°F)	7 (d/hr)	(°F)
397	-	-	-	X	5	203	13/-	150	½/-	150
									"	"
									"	"
									5/22	"
398	X	-	-	X	"	"	12/-	"	1/2	"
409	X	194	-	X	3	74	1/-	74	½/-	"
									5/22	"
411	(1)	"	-	X	"	"	12/-	150	"	"
412	(1)	"	-	X	"	75	"	"	"	"
140	X	200	-	X	5	"	68d	"	-	-
124	X	"	-	X	8	74	"	"	-	-
125	X	"	-	X	5	76	"	"	-	-
128	(2)	"	-	X	"	"	"	"	-	-
318	-	"	-	X	"	68	4d/17hr	"	-	-
319	-	"	-	X	"	"	4d/16hr	"	-	-
320	-	"	-	X	"	"	4d/3hr	"	-	-
321	-	"	-	X	"	"	4d	"	-	-

1. Blended all but DTD.
2. Preheat L-2344 & V-82. Add B-126 when blend at ambient.
3. See Table 6-12.

TABLE 6-21

SILANES (Refs. 6-14)

Vendor's No. (S-8)	Name	Structure	B.P. 760mm (°F)	Mol. Wt.	Hydrogen Atoms (%)
A-151	Vinyltriethoxysilane	$CH_2=CH\ Si\ (OET)_3$	322.	190.3	9.46
A-164	Dimethyldiethoxysilane	$(CH_3)_2\ Si\ (OET)_2$	236.	148.3	10.8
A-187	gamma-Glycidoxypropyl-trimethoxysilane	 $CH_2CH\ CH_2O(CH_2)_3\ Si(OCH_3)_3$	554.	236.	8.47
A-188	Vinyltriacetoxysilane	$CH_2=CH\ Si(OOCCH_3)_3$	446.	232.	5.18
A-1100	gamma-Aminopropyl-triethoxysilane	$NH_2(CH_2)_3\ Si\ (OET)_3$	422.	221.3	10.4

Since the relative reactivity of the L-2344's hydroxyl groups were not sufficiently known, several possible reaction directions with the silanes were examined. A-151 was selected for its vinyl and ethoxy functions. A-1100 was of interest because of its amine and ethoxy functions. A limited test (F-222) (see Tables 6-22 and 6-23) with the A-151 showed no indication of activity. On the other hand, the A-1100 (F-290) appeared partially "cured." Increased quantities of A-1100 were then used. The results were:

F-290: Not cured.
F-291: Partially cured.
F-292: Almost cured and slightly stiff.
F-293: Cured and brittle.
F-294: Cured and brittle.

F-294 was selected as a base binder to determine the comparative flammability of this type system in conjunction with boric acid, since further formulation effort would be required to improve the mechanical properties. F-294 was chosen in preference to F-293 in order to assure a complete (i.e. non-marginal) "cure" and assess a conservatively formulated system with respect to flammability resistance. As seen in Table 6-24, the comparative hydrogen content of F-294 is still low.

TABLE 6-22

FLUOROCARBON/SILANE COPOLYMERS

		Formulations (phr)									
Component	S	222	290	291	292	293	294	285	366	368	369
L-2344 (1)	2	100.	100.	100.	100.	100.	100.	100.		100.	100.
A-151 (2)	8	3.81									
A-1100 (3)	8		5.	10.	25.	50.	80.	80.	221.3		
A-164 (4)	8								148.3		
H_2O	--								36.		
F-366-1	13									55.25	110.5
Boric acid (5)	9							150.			

S......Source (See Table 6-1)
1. Dihydroxy terminated fluoropolymer
2. Vinyl triethoxy silane
3. Gamma-Aminopropyl triethoxy silane
4. Dimethyldiethoxy silane
5. 100% through 170 mesh

TABLE 6-22 (cont'd)

FLUOROCARBON/SILANE COPOLYMERS

Component	S	370	371	372	373	374	431	433
		Formulations (phr)						
L-2344 (1)	2		100.	100.	100.	100.	100.	100.
A-1100 (3)	8	221.3						
A-164 (4)	8	1483.						
A-188 (6)	8		5.	10.	25.	50.		
A-187 (7)	8						25.	25.
H_2O	--	396.						
TDI (8)	--							5.22

6. Vinyl triacetoxy silane
7. Gamma-Glycidoxypropyltrimethoxy silane
8. Tolylene diisocyanate

TABLE 6-23

FLUOROCARBON/SILANE RTV POLYMERS

- FORMULATION PROCESSING -

Formulation No. F-	Steps (3)									
	1	2	3	4	5 (min)	5 (°F)	6 (hr/min)	6 (°F)	7 (d/hr)	7 (°F)
222	-	-	-	X	5	200	24/-	150	-	-
290	-	-	-	X	"	"	20/03	"	-	-
291	-	-	-	X	"	"	19/40	"	-	-
292	-	-	-	X	"	"	1/39	"	17hr/50m	150
293	-	-	-	X	"	"	1/29	"	-	-
294	-	-	-	X	"	"	1/21	"	-	-
285	(1)	-	-	-	15	74	4/35	"	-	-
366	(2)	-	-	-	5	76	20/-	75-80	-/3	185
368	-	-	-	-	<1	194	19/-	150	-	-
369	-	-	-	-	1	158	18/-	"	-	-
370	(2)	-	-	-	5	73	20/-	185	-/20	73
371	-	-	-	X	<1	185	18/-	150	-	-
372	-	-	-	X	<1	167-185	18/-	"	-	-
373	-	-	-	X	<1	"	"	"	-	-
374	-	-	-	X	<1	"	"	"	-	-
431	-	-	-	X	5	150	8/-	150	1/-	150
433	X	-	-	X	3	72	7/30	"	1/-	"

1. Blend A-1100 & boric acid @ R.T. Preheat L-2344 @ 200°F. Add blend to L-2344.
2. Blended Silanes then added water.
3. See Table 6-12.

TABLE 6-24

HYDROGEN CONTENT

Polymer	Hydrogen Atoms (%)	Ref.
L-2344	1.4	App. G
Viton LM	2.0	" . G
Fluorosilicone RTV	4.5	6-15
F-294	5.3	calc.
Dimethyl silicone RTV	8.1	6.16

The resultant specimens (F-285) with boric acid were very boardy and brittle. However, negligible thermal destruction resulted when tested up to, and including, 16.5 psia oxygen (see Table 6-25). It should be noted that within 10 minutes after adding the boric acid, the viscosity significantly increased.

Since the A-1100/L-2344 system gave indications of excessive crosslinking rather than increased linear chains, it was assumed that this was due to not only reactions at the amine, but also at the ethoxy sites. A partial cohydrolysis was attempted with A-164 and water (F-366). No exotherm was noticed when the A-164 and A-1100 were blended at room temperature. A slight exotherm was detected after the addition of the water. After heating this blend at 185°F, considerable small bubbles were seen for 30-45 minutes after removal from the heat. If the cohydrolysis was successful, these volatiles were ethyl alcohol. The resultant liquid was formulated with the L-2344. The resultant products were still brittle and hard (F-368 and F-369). Excessive A-164 was then reacted with A-1100. This approach (F-370) was abandoned when a white residue appeared.

A direct reaction of A-188 was then carried out with the L-2344. Immediate gelation resulted in each case (F-371, -372, -373, and -374). The products were very gummy, tacky, soft, and had very poor cohesion.

Similarly, a reaction between A-187 and L-2344 was sought. The percent hydrogen in F-431 was caluclated to be only 2.81%. No cure resulted. When TDI was added to this system, the product was soft, tacky, gummy, and had very poor cohesion,

6.1.2.3 Diluents - Krytox 143 AC, a perfluoroalkylpolyether "oil," was added (F-212) to L-2344 (see Tables 6-26 and 6-27). The materials were blended at approximately 200°F. Although the room temperature viscosity was only moderately high, some of the K-143 AC appeared to separate out. In addition, there was a distinct "oily" feel to the material.

Hexafluoroisopropanol (HFIP) (S-1) was evaluated as a potential reactive diluent since it only has 1.19% hydrogen present, has a viscosity of 1.021 cs at 77°F

TABLE 6-25
FLUOROCARBON/SILANE POLYMERS PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

		Specimen			
		285-1-1	285-1-1	285-1-1	285-1-2
	O_2 Pressure (psia)	6.2	9.72 (60%)	16.5	16.5
R	Wire Ignition (1)	T/B	T/B	T/B	T/B
E	Wire Failure (1)	B	T	T/B	T
	Smoke (2)	N	N	N	N
S	Burn Time (sec.) (3) flame-out	5	12	11	9
	Burn Time (sec.) (3) glow-out	5	12	11	9
U	Configuration (4)	Y	Y	Y	Y
L	Residue (%) (5)	>99	>99	95	98
T	Melted (6)	N	N	N	N
	Color exterior	cream/	cream/	white/	cream/
S	Color interior	black	black	black	black
	Exterior Texture powdery (6)	N	N	N	N
	Exterior Texture crusty (6)	Y	Y	Y	Y

1. T....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No

TABLE 6-26

FLUOROCARBON POLYMER WITH DILUENTS

		Formulations (phr)								
Component	S	212	357	359	358	360	361	362	363	364
L-2344 (1)	2	100.		165.		164.		100.	100.	100.
KRYTOX 143 AC (2)	1	50.								
HFIP (3)	1		100.		100.		330.			
A-1100 (4)	8		132.				132.			
A-151 (5)	8				113.					
F-357-1	13			232.						
F-358-1	13					213.				
F-361-1,2	13							16.5	82.5	165.

S......Source (See Table 6-1)
1. Dihydroxy terminated fluoropolymer
2. Perfluoroalkylpolyether fluid
3. Hexafluoroisopropanol
4. Gamma-Aminopropyltriethoxy silane
5. Vinyl triethoxy silane

TABLE 6-26 (cont'd)

FLUOROCARBON POLYMER WITH DILUENTS

		Formulations (phr)								
Component	S	365	367	403	404	406	407	408	405	410
L-2344 (1)	2		100.						100.	
HFIP (3)	1	200.		168.	168.	168.			50.	
A-1100 (4)	8	132.								
F-365-1	13		62.9							
F-405-1	13									150.
TDI (6)	--			87.			87.			31.12
PAPI (7)	--				130.			130.		
Catalyst T-9 (8)	--			4.	4.	4.	4.	4.		
DTD (9)	7									1.2

6. Tolylene diisocyanate
7. Polymethylene polyphenylisocyanate
8. Stannous octoate
9. Dibutyl tin dilaurate

TABLE 6-27

FLUOROCARBON POLYMER WITH DILUENTS

- FORMULATION PROCESSING -

Formulation No. F-	Steps (2) 1	2	3	4	5 (min)	5 (°F)	6 (hr/min)	6 (°F)	7 (d/hr)	7 (°F)
212	-	-	-	-	5	200	4/30	150	-	-
357	-	-	-	-	2	77	-	-	-	-
359	-	-	-	-	5	71	12/-	150	-	-
358	-	-	-	-	2	77	-	-	-	-
360	-	-	-	-	5	70	-/8	150	-	-
361					(See text)					
362	-	-	-	-	5	150	45/-	150	-	-
363	-	-	-	-	"	"	"	"	-	-
364	-	-	-	-	"	"	21/-	"	-	-
365					(See text)					
367	-	-	-	-	5	150	20/-	150	-	-
403	(1)	-	-	-	1	77	4/-	77	1/-	77
404	(1)	-	-	-	"	"	"	"	"	"
406	-	-	-	-	"	74	-	-	-	-
407	-	-	-	-	"	"	-	-	-	-
408	-	-	-	-	"	"	-	-	-	-
405	-	-	-	-	5	76	6/-	76	-	-
410	-	-	-	-	"	72	1/-	73	½/-	150

1. Added T-9 4 hrs. after blending HFIP & TDI.
2. See Table 6-12.

(Ref. 6-12), "...exhibits strong hydrogen bonding and will associate and dissolve most molecules with receptive sites, such as oxygen, double bonds, or amine groups." (Ref. 6-13). It also forms "stable and distillable complexes...with many ethers and amines..." (Ref. 6-12). It was considered of use herein as either a) a partial inactivator of A-1100 (see Section 6.1.2.2, this report), or as b) a direct coreacting diluent with L-2344.

A strong exothermic reaction was evidenced when A-1100 was blended with HFIP (F-357) at room temperature; none was detected between A-151 and HFIP (F-358). Both were dried with anhydrous calcium sulfate and then added to L-2344. The one (F-359) compounded with A-1100 was similar to F-294 (see Table 6-22) when cured, and had a faint A-1100 odor. No reaction was noticed with the A-151 blend.

Another series was formulated in which the A-1100 and considerable excess (3 X) HFIP were refluxed together (F-361) at 221°F. The formulations prepared with this blend and L-2344 showed no significant improvement in mechanical properties over the previous group, and had considerably more voids. A third series, containing less HFIP (2 X), also showed no improvement, except that the voids were significantly quite smaller.

If HFIP were used as a diluent for L-2344, it was essential that side reactions be kept to a minimum. F-403 and -404 were prepared to evaluate this problem. Complete miscibility was found between the HFIP and the TDI and PAPI. However, when stannous octoate was added, high exotherms resulted in less than one minute. A hard, white, porous solid was obtained in approximately 45 minutes at room temperature with F-403. A hard, dark brown solid was found in F-404 after approximately 45 minutes at room temperature. F-403 did not melt at 185°F, whereas F-404 did. The stannous octoate was separately blended with the HFIP (F-406), TDI (F-407), and PAPI (F-408) to ascertain the specific coreactants. As expected, no change occurred with the HFIP, but TDI resulted in a hard, brittle, very porous, tan, opaque mass which had poor adhesion to the test tube wall. PAPI gave a hard, brittle, glassy clear dark brown mass which had excellent adhesion to the test tube.

Based upon these results, L-2344 was gently refluxed with HFIP (F-405). After 6 hours at room temperature, TDI and dibutyl tin dilaurate were blended into the solution (F-410). A hard, porous, and somewhat brittle mass was obtained.

6.1.2.4 Silicone RTV Modified Formulations - Mechanical blends of silicone and fluorosilicone RTV's were made with the L-2344 for the purpose of reducing the latter's viscosity (see Tables 6-28 and 6-29). In some cases, the only catalyst used was the one for the silicone (and fluorosilicone) to determine if a copolymer could be developed instead of having merely a mechanical mixture. Other formulations also contained reactants for the L-2344. Regardless of the type of RTV used, the blends showed evidence of not being true copolymers. For example,

TABLE 6-28

FLUOROCARBON/SILICONE RTV POLYMERS

Component	S	Formulations (phr) 021	022	023	219	217	160	220	221	218	271	295	297	322	323
L-2344 (1)	2	100.	100.	100.	100.	100.	100.		100.	100.	100.	100.	100.	100.	
96-004 (2)	12			100.							100.	100.			
SYLGARD 182 (2)	12				100.										
RTV 655A (2)	7					100.									
RTV 11 (2)	7						100.								
RTV 511 (2)	7							100.							
77-024 (3)	12									100.					
RTV 615A (2)	7												100.		
RTV 602 (2)	7													100.	
F-220-1	13								100.3						
F-322-1	13														200.5
Boric acid (4)	9										150.	150.	150.		300.
96-004 Catalyst	12	10.		12.							12.	10.			
S-182 Catalyst	12				10.										
RTV 655B	7					10.									
DTD (5)	7						0.4	0.3							
77-024 Catalyst	12									10.					
RTV 615B	7												10.		
SRC-05	7													0.5	
Catalyst T-9 (6)	--		0.6												
A-1100 (7)	8											5.	5.		

S......Source (See Table 6-1)
1. Dihydroxy terminated fluoropolymer
2. Silicone RTV
3. Fluorosilicone RTV
4. 100% through 170 mesh
5. Dibutyl tin dilaurate
6. Stannous octoate
7. Gamma-aminopropyltriethoxysilane

TABLE 6-29

FLUOROCARBON/SILICONE RTV POLYMERS

- FORMULATION PROCESSING -

Formulation No. F-	Steps (1)									
	1	2	3	4	5 (min)	(°F)	6 (hr/min)	(°F)	7 (d/hr)	(°F)
021	-	-	-	X	5	200	21d/12hr	150	-	-
022	-	-	-	X	"	"	"	"	-	-
023	X	200	-	X	"	78	4/-	"	-	-
219	X	"	-	X	6	76	38/55	"	-	-
217	X	"	-	X	6	75	40/16	"	-	-
160	X	"	-	X	5	78	21d/hr	"	-	-
220	-	-	-	X	5	76	-	-	-	-
221	X	200	-	-	3	75	38/24	150	-	-
218	X	"	-	X	8	75	5/34	"	-	-
271	X	"	-	X	5	76	4/-	"	-	-
295	X	"	-	X	10	82	23/40	"	-	-
297	X	"	-	X	5	85	25d	"	-	-
322	X	"	-	X	3	71	14d/22hr	"	-	-
323	X	"	-	-	15	"	"	"	-	-

1. See Table 6-12.

the very low viscosity of 96-004 seemed to have its iron oxide filler filtered out by the high viscosity L-2344 so that a thin water white layer surrounded its specimen. This was not apparent with the higher viscosity RTV's but their corresponding "polymers" had poorer mechanical properties (see Table 6-30).

Flammability tests were run with F-271, -295, and -297. All contained boric acid and were blends of L-2344 with catalyzed addition reaction dimethyl silicone RTV's. The two containing the low moledular weight RTV, 96-004, clearly had superior flammability resistance to the RTV 615 system. In fact, only negligible damage occurred to the 96-004 specimen even in the 16.5 psia oxygen. F-297 showed significant pyrolysis degradation in 9.72 psia oxygen/6.48 psia nitrogen (i.e. 60/40 16.2 psia mixture) (see Table 6-31).

The high boric acid loading in F-232 made it impossible to prepare flammability specimens.

6.1.2.5 Tolylene Diisocyanate/Silicone RTV Modified Formulations - Since it apparently was not feasible to utilize L-2344 just as an additive to a silicone RTV system and still maintain the useable mechanical strength of the RTV, a series of formulations (see Table 6-32) were prepared (see Table 6-33) which contained catalysts for both the L-2344 and the RTV. It was felt that the resultant mechanical mixture would have better mechanical properties (see Table 6-34) than the formulations in the preceding section, 6.1.2.4.

The low molecular weight dimethyl silicone RTV 96-004 and L-2344 did not blend well (i.e. separation) (F-278) and the cohesive strength of the "cured" specimen was extremely poor when boric acid was added (F-280). However, there was negligible pyrolysis damage at 60/40 (see Table 6-35). The unfilled blend of the fluorosilicone RTV 77-033 and L-2344 (F-284) was quite porous, had fair mechanical properties, but was completely decomposed in 6.2 psia oxygen, even when a 200°F post cure was applied. A group of formulations (F-417, -423, and -425) were prepared with Dechlorane 604, Sylgard 182, and L-2344. Varying the ratios of these ingredients, while still staying within a satisfactory viscosity range, resulted in flammability specimens which could not take 6.2 psia oxygen. Substituting, weightwise, boric acid for one of these created a formulation (F-426) which had an almost unworkable viscosity. In fact, the viscosity rapidly rose while placing the catalyzed compound(s) in the mold. The cured material was also quite porous, had poor cohesion, but did not show damage until exposed to 16.5 psia oxygen. An attempt was made to surface treat the boric acid (F-377) in order to reduce its apparent coreaction with L-2344. A formulation (F-427) with the treated material showed similar very high viscosity to that found in F-426 but had less porosity. The flammability test specimens were badly degraded at 16.5 psia oxygen and showed some degradation at 60/40. A Dechlorane 604 filled blend of 77-033 and L-2344 had fair mechanical properties, showed slight degradation in 60/40, but was completely decomposed in 16.5 psia oxygen.

TABLE 6-30

FLUOROCARBON/SILICONE RTV POLYMERS CHARACTERISTICS

-CURED/POST CURED-

	Formulation									
Properties	219	217	160	220	221	218	271	295	297	322
Cured	Y	Y	?	Y	Y	Y	Y	N	Y	S
Tacky	S	S	Y	N	Y	S	N	S	N	Y
Flexible	Y	Y	Y	Y	Y	Y	F	F	F	Y
Memory	X	X	F	X	P	X	P	P	F	Y
Cohesion	X	F	P	F	P	P	NG	NG	P	P
Tear Resistance	X	F	P	F	P	P	NG	NG	P	P
Crack Resistance (1)	-	-	-	-	-	-	NG	NG	F	X
Crack Propagation (1)	-	-	-	-	-	-	NG	NG	F	X
Porous	N	N	S	N	Y	Y	N	Y	Y	?
Adheres to unclean alum. Foil cup	F	F	N	P	F	-	-	-	X	P
Cheesy	N	N	N	N	N	N	N	N	N	(2)

Y.....Yes VS.....Very Slight S.....Slight N.....No
X.....Satisfactory F.....Fair P.....Poor NG.....Not Good

1. 180° bend (face-to-face contact)
2. Slightly gummy

TABLE 6-31
FLUOROCARBON/SILICONE RTV POLYMERS
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

		Specimen							
		271-I-I	271-I-I	271-I-I	295-I-I	295-I-I	295-I-I	297-I-I	297-I-I
	O_2 Pressure (psia)	6.2	9.72 (60%)	16.5	6.2	9.72 (60%)	16.5	6.2	9.72 (60%)
R	Wire Ignition (1)	T	B	T/B	T/B	T	T/B	T	T/B
E	Wire Failure (1)	T	B	T	T	T	T	T	B
	Smoke (2)	N	N	N	N	N	N	N	P
S	Burn Time (sec.) (3) flame-out	2	~4	20	~10	2	8	2	65
	Burn Time (sec.) (3) glow-out	2	6	20	16	2	17	2	65
U	Configuration (4)	Y	Y	Y	Y	Y	Y	Y	N
L	Residue (%) (5)	>99	>99	>99	95	>99	95	>99	50
T	Melted (6)	N	N	N	N	N	N	N	N
	Color exterior	red	(7)	(7)	(7)	red	red/wht	beige	gray
S	Color interior	"	---	---	---	"	---	"	black
	Exterior Texture powdery (6)	N	N	N	Y	N	N	N	Y
	Exterior Texture crusty (6)	N	Y	Y	Y	N	Y	N	Y

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. Red/white/black.

TABLE 6-32

FLUOROCARBON (TDI CATALYZED)/SILICONE RTV POLYMERS

Component	S	Formulations (phr) 278	280	284	299	417	423	425	426	377	427	424
L-2344 (1)	2	100.		100.	100.	100.	100.	100.	100.		100.	100.
96-004 (2)	12	100.										
77-033 (3)	12			100.								100.
RTV 615A (2)	7				100.							
SYLGARD 182 (2)	12					100.	50.	40.	50.		50.	
F-278-1	13		320.									
F-377-1	13										150.	
Boric acid (4)	9	120.			150.							
Boric acid (5)	17								150.			
Boric acid (6)	9									100.		
DECHLORANE 604	16					250.	150.	150.				150.
96-004 Catalyst	12		12.									
77-033 Catalyst	12			10.								10.
RTV 615B	7				10.							
S-182 Catalyst	12					10.	5.	4.	5.		5.	
TDI (7)	--		6.	5.22	6.	5.22	5.22	5.22	5.22		5.22	5.22
Catalyst T-9 (8)	--		1.2									
A-151 (9)	8									1.5		
A-164 (10)	8									1.5		

S...... Source (See Table 6-1)

1. Dihydroxy terminated fluoropolymer
2. Silicone RTV
3. Fluorosilicone RTV
4. 100% through 170 mesh
5. USP, granular
6. 100-170 mesh
7. Tolylene diisocyanate
8. Stannous octoate
9. Vinyl triethoxy silane
10. Dimethyldiethoxy silane

TABLE 6-32(cont'd)

FLUOROCARBON (TDI CATALYZED)/SILICONE RTV POLYMERS

		Formulations (phr)											
Component	S	413	414	416	418	419	420	421	422	428	429	430	415
L-2344 (1)	2		100.	100.	100.								
96-004 (2)	12								100.				
SYLGARD 182 (2)	12			100.	50.	100.	100.						
SYLGARD 186 (2)	12									100.	100.	100.	
F-413-1	13		10.2	10.2	5.2	10.2	10.2		10.2				
DECHLORANE 604	16			150.	150.								
96-004 Catalyst	12	10.											
S-182 Catalyst	12							10.					10.
S-186 Catalyst	12									10.	10.	10.	
TDI (7)	--		5.22	5.22	5.22			5.22					
DTD (11)	7	0.2											0.2
DTDA (12)	18									0.1			
bio M&T TBTO (13)	18										0.1		
TYZOR TPT (14)	1											0.1	

11. Dibutyl tin dilaurate
12. Dibutyl tin diacetate
13. Bis (Tri-n-butyltin) oxide
14. Organic titanate

TABLE 6-33

FLUOROCARBON (TDI-CATALYZED)/SILICONE RTV POLYMERS

- FORMULATION PROCESSING -

Formulation No. F-	Steps (3)									
	1	2	3	4	5 (min)	5 (°F)	6 (hr/min)	6 (°F)	7 (d/hr)	7 (°F)
278	X	200	–	–	20	200	–	–	–	–
280	–	–	–	X	10	80	186/32	150	9/13	150
284B	(1)	200	–	X	6	86	17/40	"	3/23	"
284C	(1)	"	–	X	"	"	"	"	3/23	200
299	X	200	–	X	5	84	24d/2hr	"	–	–
417A	(1,2)	150	–	X	15	83	12/30	"	–	–
417B	(1,2)	"	–	X	"	"	"	"	1/6	150
423A	(1,2)	"	–	X	3	82	18/30	"	–	–
423C	(1,2)	"	–	X	3	82	18/30	"	4/10	150
425	(1,2)	"	–	X	5	75	23/30	"	–	–
426	(1,2)	"	–	X	"	76	41/–	"	–	–
377	X	–	–	–	60	73	–	–	–	–
427	(1,2)	150	–	X	5	78	40/–	150	–	–
424A	(1,2)	"	–	X	5	82	17/45	"	–	–
424C	(1,2)	"	–	X	"	"	"	"	4/12	150
413	X	–	–	–	1	83	–	–	–	–
414	(1,2)	–	–	X	15	"	2/20	150	–/15	150
416	(1,2)	–	–	X	3	84	7d	"	–	–
418	(1,2)	–	–	X	8	83	"	"	–	–
419	–	–	–	X	1	"	6/45	"	24/2	200
420	–	–	–	X	"	"	"	"	"	"
421	X	–	–	–	5	79	–	–	–	–
422	–	–	–	X	1	75	9/–	150	–	–
428A	X	–	–	X	5	80	23/30	RT	–	–
428B	X	–	–	X	5	80	"	150	–	–
429A	X	–	–	X	3	"	22/–	RT	–	–
429B	X	–	–	X	"	"	"	150	–	–
430A	X	–	–	X	"	"	"	RT	–	–
430B	X	–	–	X	"	"	"	150	–	–

1. Preheat L-2344. Add silicone.
2. Add solid additives at R.T.
3. See Table 6-12.

TABLE 6-34

FLUOROCARBON (TDI CATALYZED)/SILICONE RTV POLYMERS CHARACTERISTICS

-CURED/POST CURED-

Properties	Formulation (3)											
	284B	284C	299	417A	417B	423A	423C	425	426	427	424A	424C
Cured	P	Y	?	Y	Y	P	Y	F	Y	Y	F	Y
Tacky	Y	S	N	S	VS	S(2)	S(2)	S	S	S	S	X
Flexible	X	X	X	X	X	X	X	X	X	F	X	X
Memory	P	X	X	F	F	X	F	F	F	F	X	X
Cohesion	F	X	P	P	P	P	F	P	P	P	P	P
Tear Resistance	F	X	P	P	P	P	F	P	P	P	P	P
Crack Resistance (1)	X	X	F	X	X	X	X	X	P	P	X	X
Crack Propagation (1)	X	X	F	X	X	X	X	X	P	P	X	X
Porous	Y	Y	Y	N	N	Y	Y	Y	Y	Y	S	S
Adheres to unclean alum. Foil cup	-	-	?	-	-	-	-	-	F	F	-	-
Cheesy	N	N	N	N	N	N	N	N	S	S	N	N

Y.....Yes VS.....Very Slight S.....Slight N.....No
X.....Satisfactory F.....Fair P.....Poor NG.....Not Good

1. 180° bend (face-to-face contact)
2. On open surface.
3. Letter designation in formulation number refers to cure/post cure cycle (see Table 6-33).

TABLE 6-35
FLUOROCARBON (TDI CATALYZED)/SILICONE RTV POLYMERS
PRELIMINARY FLAMMABILITY
– TEST CONDITIONS/RESULTS –

			Specimen									
			280-1-1	280-1-2	280-1-1	284-1B-1	284-1C-1	299-1-1	299-1-1	417-1A-1	417-1B-1	423-1-A	423-1C-1
	O_2 Pressure (psia)		6.2	6.2	9.72 (60%)	6.2	6.2	6.2	9.72 (60%)	6.2	6.2	9.72 (60%)	9.72 (60%)
R	Wire Ignition (1)		T/B	T/B	T	T/B	T/B	T	T/B	T/B	T/B	T/B	T/B
E	Wire Failure (1)		T	T	T	B	T	T	B	B	B	B	B
	Smoke (2)		N	N	N	N	N	N	P	P	P	P	P
S	Burn Time (sec.) (3)	flame-out	0	0	12	41	40	3	72	55	51	57	50
		glow-out	0	0	12	52	52	3	72	55	51	57	50
U	Configuration (4)		Y	Y	Y	Y	N	Y	---	Y	Y	Y	N
L	Residue (%) (5)		0	0	>99	0	0	100	75	75	50	25	10
T	Melted (6)		N	N	N	N	N	N	---	N	N	N	N
	Color	exterior	red	red	(7)	gray	gray	brown	---	black	blk/wht	white	white
S		interior	"	"		"	"	"	---	white	"	"	"
	Exterior Texture	powdery (6)	N	N	N	Y	Y	N	---	Y	N	Y	Y
		crusty (6)	N	N	Y	Y	Y	N	---	Y	Y	N	N

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. Red/white/black.

TABLE 6-35 (cond't)
FLUOROCARBON (TDI CATALYZED)/SILICONE RTV POLYMERS
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

			Specimen								
			425-1-1	425-1-2	426-1-1	426-1-2	426-1-1	427-1-1	427-1-1	427-1-1	427-1-2
	O_2 Pressure (psia)		6.2	6.2	6.2	9.72 (60%)	16.5	6.2	9.72 (60%)	16.5	16.5
R	Wire Ignition (1)		T/B	T/B	T/B	T/B	T/B	T/B	T/B	T/B	T/B
E	Wire Failure (1)		T	T	T	T	T	T	T	B	T
	Smoke (2)		S	S	N	N	S	N	N	P	P
S	Burn time	flame-out	73	90	0	4	37	2	18	59	78
	(sec.) (3)	glow-out	105	142	0	4	37	2	18	59	78
U	Configuration (4)		---	N	Y	Y	Y	Y	Y	Y	Y
L	Residue (%) (5)		---	0	100	100	85	100	98	50	50
T	Melted (6)		---	N	N	N	N	N	N	N	N
	Color	exterior	---	---	blk/wht	tan	white	tan	wht/blk	wht/blk	wht/blk
S		interior	---	---	tan	"	black	"	tan	"	"
	Exterior	powdery (6)	---	Y	N	N	N	N	N	N	N
	Texture	crusty(6)	---	N	Y	N	Y	N	Y	Y	Y

TABLE 6-35 (cont'd)
FLUOROCARBON (TDI CATALYZED)/SILICONE RTV POLYMERS
PRELIMINARY FLAMMABILITY
– TEST CONDITIONS/RESULTS –

			Specimen					
			424-1A-1	424-1A-1	424-1B-1	424-1C-1	414-1-1	414-1-2
	O_2 Pressure (psia)		6.2	9.72 (60%)	16.5	16.5	6.2	6.2
R	Wire Ignition (1)		B	T/B	T/B	T/B	T/B	T/B
E	Wire Failure (1)		B	B	T	T	---	T
	Smoke (2)		N	N	S	S	---	S
S	Burn Time (sec.) (3)	flame-out	7	7	45	30	5	59
		glow-out	7	7	75	59	5	79
U	Configuration (4)		Y	Y	Y	Y	Y	N
L	Residue (%) (5)		95	95	5	5	95	0
T	Melted (6)		---	N	N	N	Y	N
	Color	exterior	black	black	white	blk/wht	black	---
S		Interior	tan	tan	"	"	red	---
	Exterior Texture	powdery (6)	N	N	N	Y	N	Y
		crusty (6)	N	N	Y	Y	Y	Y

Some of the above cocatalyzed formulations evidenced some phase separation in the cured specimens. It was assumed that the RTV was gelling much faster than the L-2344. An attempt was made to speed up the latter process by adding dibutyl tin dilaurate to the system. A master batch of DTD with 96-004 catalyst was prepared (F-413). A similar one with Sylgard 182 catalyst was also made (F-415). Dechlorane 604 filled formulations were made (F-414, -416, and -418). None were cured. F-413 was then used with Sylgard 182 alone. No cure occurred with F-419 (degassed) or F-420 (not degassed). The effect upon RTV 96-004 cure was also checked (F-422). Again, no cure resulted. The basic systems were then rechecked (Sylgard 182: F-042-2 and RTV 96-004: F-044-2). Both readily cured. The compatibility of tolylene diisocyanate with these addition reaction silicone RTV catalysts was then evaluated (F-421). They appeared to be non-miscible but non-reacting with each other. It was clear that the DTD was "poisoning" these RTV catalysts. (F-413 became a very weak gel/liquid mixrure when rechecked 6 months later. In the same time, F-415 turned into a cheesy gel, and F-421, kept in a desiccator containing Drierite, remained mostly as a two-phase liquid which had some gel in the TDI layer and some crystals in the 182 catalyst layer.)

Compatibility of other potential TDI "slow" accelerators with Sylgard 186 was then determined.

F-428 (dibutyl tin diacetate): Incomplete cure (cheesy gel state, oily feel).
F-429 (bio-Met TBTO): No cure.
F-430 (Tyzor TPT): Cured somewhat slower than usual, 150°F cured (22 hours) specimens had better mechanical strength than room temperature cured (~3 months recheck) one.

6.2 SILICONE RTV POLYMERS

Although unfilled silicone RTV's have approximately 8.1% hydrogen by weight as compared to approximately 2.0% for conventional fluoroelastomer polymers (see Table 6-24), lower viscosities, significantly easier processing characteristics, excellent electrical properties, and respectable thermal stability of the former clearly necessitated their consideration in this program. None by themselves appear to meet the 60% oxygen/40% nitrogen, 16.5 psia, flammability requirements for this program (Ref. 6-1), let alone the 16.5 psia pure oxygen environment. However, it was felt that the addition of suitable flame retardants might allow them to do so. Since a maximum cure temperature of 150°F is a program parameter, only silicone room and low temperature vulcanizing (RTV's and LTV's) cure systems were permissible. The two principal sources of such materials were Dow Corning and General Electric. Stauffer Chemical recently entered this field, but their product line did not appear to offer anything different than that provided by the other two companies. We therefore arbitrarily confined our analyses to Dow Corning and General Electric products. In addition to those materials which are very similar in both lines, each did offer certain unique grades which were of interest herein. The following very brief description of the applicable (and then available) cure systems and their respective pertinent grades is presented in order to establish the basis of our selections for initial study (see Table 6-36).

A. Condensation Reaction

A.1 One Part: Since these are moisture curing, they were only of minimal interest as the basis for a conformal coating. Although both G.E. and Dow Corning have these moisture cured systems, the various selections from these were limited to one vendor. This was done to minimize duplication. Dow Corning was selected since it has a broader variety of such cure systems (e.g. G.E. apparently does not have the methoxy cured type).

A.1.a <u>Acetoxy Type</u> - Acetic acid is the byproduct. Potential corrosion of substrates minimized interest in this group. There was some indication (Ref. 6-17) that 77-000 was being used by a major aerospace firm as the basis of a flame retardant system. 96-042 is supposedly related to 77-000, and is more readily available.

A.1.b <u>Methoxy Type</u> - The reaction product is methyl alcohol. Hence, this system is non-corrosive. 3140, a typical compound of interest, has a viscosity of 350 poise which was low enough to permit some fire retardant loading.

A.1.c <u>ROH Type</u> - Although similar to the methoxy system, the members of this group produce low molecular weight alcohols other than methyl alcohol.

A.2 Two Part: The silanol condensation reaction is usually initiated by such metallic soaps as stannous octoate, dibutyl tin dilaurate, and lead octoate. The latter is seldom used because of toxicity problems. The tin octoate is the fastest and generally results in fairly good reversion resistance. The dibutyl tin is the most commonly used catalyst for this system. The data reported in References 6-1 and 6-9 seem to infer that the methyl-phenyl, Me-ϕ, silicone RTV's have a lower flame propagation rate than the dimethyl, $(Me)_2$, ones. Since the methyl-phenyl types are commercially most available as metallic soap cured systems, we primarily selected two of them, RTV's 511 and 560, for further consideration (see Table 6-37).

TABLE 6-36

SILICONE RTV'S (& LTV'S) OF INTEREST

		Reaction System						Silicone Type	
		Condensation (2)				Addition			
		1 Part			2 Parts	1 Part	2 Parts (3)		
Grade	S (1)	Acetic Acid	MeOH	ROH	H_2O or ROH			$(Me)_2$	Me-Ø
96-042	12	X						X	
77-000	12	X						X	
3140	12		X					X	
92-007	12			X				X	
92-009	12			X				X	
11	7				X			X	
511	7				X				X
560	7				X				X
577	7				X				X
96-004	12						X	X	
SYLGARD	12						X	X	
93-072 (4)	12						X	X	
93-500 (5)	12						X	X	
615	7						X	X	
616	7						X	X	
655	7						X		X
96-052	12						X		X
602	7				X			X	
603	7				X				X

1. See Table 6-12.
2. Listed by reaction by-products.
3. See Table 6-38.
4. Similar to Sylgard 186, but white.
5. Vacuum grade of Sylgard 184.

TABLE 6-37

TWO PART CONDENSATION SILICONE RTV's (1)

Properties	RTV 511	RTV 560	RTV 602	RTV 603
Viscosity, uncat. (Poise)	200	400	8-15	10-30
Solids, initial (%)	98	98	100	100
Solids, cured (%)	---	---	100	100
Pot life (hrs)	4-6	3-5	2-3 (2)	2-3 (2)
Tack free time (hrs)	6-10	5-8	---	---
Cure cycle (hrs/°F)	16-24/77	16-24/77	5/150 (2)	5/150 (2)
Specific gravity	1.20	1.42	0.995	0.99
Hardness (Shore A)	45	60	15	15
Tensile strength (psi)	350	800	---	---
Elongation (%)	180	160	---	---
Tear resistance, die B (lb/in)	15	45	15	---

(1) General Electric
(2) SRC-05 catalyst (G.E.): 0.25 phr.

Another two part condensation reaction system, RTV 602 (G.E.) is quite unique. It utilizes an amine catalyst for cure. Although it is somewhat more flammable (Refs. 6-1 and 6-9) than the other RTV's which were under consideration for this program, it provided an equally unique opportunity to reduce the viscosity of an amine cured fluoroelastomer (e.g. Viton and Fluorel) (see Section 6.1.1.3, this report). The vendor was subsequently able to supply a methyl-phenyl version, RTV 603.

B. Addition Reaction

B.1 One Part: RTV 815 (G.E.) is typical of this type. It is basically identical to RTV 615 (G.E.), a two part addition system, except that an inhibitor has been added to the polymer to prevent premature activation of the already included catalyst. The inhibitor is thermally removed at a minimum temperature of 212°F, 62°F above the limitation imposed by the Program's Work Statement. Therefore these compounds were not included in the present program.

B.2 Two Part: These are essentially vinyl silicone polymers which can be polymerized by such catalysts as chloroplatinic acid. Pertinent ones are shown in Table 6-38. Except as otherwise indicated, all of those tabulated have the following characteristics:

1. They can be deep section cured.

TABLE 6-38

ADDITION REACTION (1) RTV's & LTV's

	Grade								
Properties	96-004	S-182 (2)	S-184 (3)	S-186 (4)	S-188 (5)	615	616	655	96-052
Viscosity-base (Poise)	15	58	40-65	1000	105	35	40	50	200
-base + cat. (Poise)	5	41	<40	500					100
Transparent	N	Y	Y	Y	Y	Y	N	Y	N
Color	red	water	water	water	water	lt.straw	bl.	water	white
Moisture sens.-uncured	Y	Y	Y	Y	Y				Y
Pot life (hrs./°F)	2/RT	6/77	3/77	>2/RT	6/77	4/77	4/77	4/77	5/77
Cure, min. temp. (hrs./°F)	24/77		7D/77			7D/77	7D/77	7D/77	1D/77
	4/150	4/150	4/150	4/150	4/150	4/150	4/150	4/150	4/150
Reversion resistant	Y	Y	Y	Y	Y				Y
Tensile strength (psi)	200	800	600	650	300	850	800	850	255
Elongation (%)	140	100	100	420	200	150	100	150	280
Hardness (Shore A)	28	35	30	32	17	35	45	35	35
Tear strength (lb/in)		15	15	100					
Specific gravity	1.29	1.05	1.08	1.12	1.05	1.02	1.22	1.07	1.18
Self-extinguishing (in air)	Y	Y	Y	Y	Y				Y

1. See Table 6-36
2. S-183 is black version of S-182; catalyzed viscosity is 50 poise
3. S-185 is black version of S-184; catalyzed viscosity is 50 poise
4. S-187 is black version of S-186; catalyzed viscosity is 500 poise
5. S-189 is black version of S-188;

2. They have a cure inhibition problem. Contact with such materials (or even their vapors) as:–

   a) Amines and amine-cured elastomers and plastics, and certain other nitrogen compounds used to cure elastomers and plastics (Refs. 6-18, -19, -20, and -21),
   b) Tin compounds (Ref. 6-18),
   c) Sulfur and sulfur compounds (and their residues) such as are used in organic rubbers (e.g. butyl and chlorinated rubbers) (Refs. 6-18, -19, -20, -21, and -22),
   d) Acid materials (and their residues) used in some silicone RTV rubbers (Refs. 6-18, -19, -20, -21, and -22),

   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 was therefore maintained during the formulation and cure phases, and could probably be provided in production, or even in the field, with only minimal inconveniences. Where an incompatible material or component could not be avoided, coating it with a barrier or primer to act as an interface will suffice.

3. None of the ingredients (i.e. pre-polymer or catalyst) are considered toxic in the uncured state.

It is seen from Table 6-38 that this reaction system is available in a wide range of viscosities and silicone types. The very low viscosity of 96-003 suggested an excellent opportunity for high loadings, even though its mechanical strength is intrinsically low. Similarly cured, the standard Sylgard (Dow Corning) series, the RTV 630 (G.E.) series, and the 615 and 616 grades of G.E. were also deemed excellent starting points for further loading. The 616 has shown the best flame resistance as compared to the other G.E. compounds mentioned, when tested with the chimney/bunsen burner facility (Ref. 6-9). In the same test, 655, the methyl-phenyl compound, was significantly more flammable than 616 but less so than 615, the unfilled version of 616. It should also be noted that 655 is not reported in Reference 6-1.

6.2.1 Addition Reaction Systems

6.2.1.1 Unfilled Formulations – Initial formulations with the addition reaction systems were without modification in order to establish a base line of respective characteristics, including flammability. Table 6-39 tabulates the commercial

TABLE 6-39

ADDITION REACTION SILICONE RTV'S

Component	S	Formulations (phr)									
		044	042	043	054	055	045	046	047	117	118
96-004	12	100.									
SYLGARD 182	12		100.								
SYLGARD 184	12			100.							
SYLGARD 186	12				100.						
SYLGARD 188	12					100.					
RTV 615A	7						100.				
RTV 616A	7							100.			
RTV 655A	7								100.		
EC-1663B	2									100.	
96-052	12										100.
94-004 Catalyst	12	10.									
S-182 Catalyst	12		10.								
S-184 Catalyst	12			10.							
S-186 Catalyst	12				10.						
S-188 Catalyst	12					10.					
RTV 615B	7						10.				
RTV 616B	7							10.			
RTV 655B	7								10.		
EC-1663A	2									10.	
96-052 Catalyst	12										10.

S.......Source (See Table 6-1)

addition reaction silicone RTV compounds being evaluated. In general, processing (see Table 8 of Reference 6-10 and Sections II.B.1 and II.B.3 of Reference 6-11) was carried out in accordance with the manufacturer's recommendations. All the compounds and their respective catalysts were blended by hand at room temperature and degassed at room temperature. Except for F-117 and -118, the cure cycles were expedited by preheating the molds to 150°F. The molds for F-117 and -118 were not preheated. Primary interest in "base-line" compounds was for evaluation of flammability resistance; mechanical and physical properties considerations were of secondary interest. From F-117 on, all of the silicone RTV addition reaction types were cured in non-preheated molds since the preheating of the others were only carried out to expedite mold turn-around time during initial Program stages. All were cured/post cured at 150°F for at least a total of four hours, except F-045 (due to premature removal from oven); it received 3 hours 20 minutes exposure at 150°F.

The catalyzed systems, in order of increasing viscosity, were: F-044 $<$-042, -043, -045, -046, -047 $<$-055 $<$-118 $<$-117 $\ll$ -054. The ones listed above, from F-042 to -047 are grouped together since they had approximately the same viscosity. It was noticed that F-044 and -043 seemed to start gelling within a few minutes after being put in the hot mold.

At this point, it should be noted that some extra attention was given to F-117 since it (EC-1663B/A: 3M) had been utilized for Lunar Module potting requirements (Ref. 6-23). A standard preliminary flammability molding was attempted with this material. It was catalyzed per the manufacturer's instructions (i.e. 10 parts of EC-1663A to 100 parts EC-1663B. The vendor doesn't mention an elevated temperature cure in its literature except post cure starting at 450°F. However, it was decided to cure this system at 150°F since the other RTV's had also been so cured, to expedite mold recycling. After 31 hours 34 minutes at 150°F, the specimens were still not cured throughout. An 11 hour 10 minute post cure at 200°F did not greatly improve the degree of cure. Ambient temperature air storage for over 60 days resulted in an almost non-tacky state for the original exposed surfaces.

A qualitative evaluation of some of the pertinent characteristics of the formulations, particularly with respect to mechanical and physical properties, is shown in Tables 8 and 9 of Reference 6-10, and Sections II.B.1 and II.B.3 of Reference 6-11. All except F-055 demolded satisfactorily. Although appearing essentially cured after an additional 2.5 hours at 150°F, this formulation was still quite tacky even after this post cure. F-045, -046, and -047 were slightly tacky after the cure but were not after the post cure. It would seem that the F-055 surface somehow became inhibited, whereas the others didn't, or is basically more sensitive to inhibition factors than the others of this group. Although cure inhibition problems with these compounds were known, no special precautions were undertaken other than carefully cleaning the tooling previously used with amines with methyl ethyl ketone. For example, the same ovens were used without a high temperature bake-out. Except in the isolated case of the initial vacuum volatility evaluation of F-387-8 (see Section 7.2, this report), inhibition from such sources has not been of any consequence.

Other than the above specific situations, no problems ensued during the processing phase for any of the formulations discussed in this section.

The results of the preliminary flammability tests of these compounds are shown in Table 6-40 and are discussed in greater detail in References 6-10 and 6-11. All specimens, except F-045 and -047, remained unchanged when tested in ambient pressure air. These two evidenced a slightly burnt condition on one end of each of the specimens.

None of the 6.2 psia oxygen tested specimens survived. Of the unfilled systems, the methyl-phenyl one seemed to have better char strength than the dimethyl varieties. The filled specimens, which didn't contain what appeared to be carbon black, had better char strength than either unfilled or carbon black filled ones. It is believed that the filled ones contain the following:

F-044: Iron oxide and silica (or TiO_2)
F-054: Silica
F-046: Carbon black and silica (or TiO_2)

The flammability specimens' residue of F-117 appears exactly like F-049B (6.2) with respect to both color (inside and outside) and texture. It is apparent that this material is not significantly, or even marginally, better than the other iron oxide filled silicone RTV's in mechanical strength or flame resistance. Also, its viscosity doesn't permit much more loading, and its cure time and conditions are less satisfactory than others evaluated. There was no further activity with this system.

6.2.1.2 Inorganic Flame Retardants - In order to add flame retardants to the base resin, it was obviously essential that the resin have the minimum initial viscosity. It was seen from the experimental results with the addition systems that only F-044, -042, -043, -045, -046, -047, and, possibly, -055 had sufficiently low enough viscosities to be considered as the flame retardant carrier(s). Of these, F-045, -046, and -047 were somewhat better with respect to mechanical properties. Since F-046 is a carbon black filled version of F-045, it was also deleted. F-047 was selected for the carrier in the initial study of flame retardants because it is a methyl-phenyl silicone RTV, whereas F-045 is a dimethyl type. Although the total hydrogen (and carbon) content is greater with the former, it was expected to provide better thermal stability (i.e. both high and low temperatures). In addition, although unfilled, it evidenced some char integrity.

TABLE 6-40
ADDITION REACTION SILICONE RTV'S
PRELIMINARY FLAMMABILITY
-TEST CONDITIONS/RESULTS-

		Specimen									
		044B	044B	044B-2	044B-3	042B-1	042B-1	042B-2	042B-3	043B	043B
	O_2 Pressure (psia)	(7)	6.4	6.2	6.2	(7)	6.2	6.2	6.2	(7)	6.2
	Wire Ignition (1)	T	T/B	T/B	T/B	T/B	T/B	T/B	T/B	T/B	T/B
R	Wire Failure (1)	T	T	B	T			B	B	T	T
E	Smoke (2)	N		N	S			N	N	N	
S	Burn Time (sec.) (3) flame-out	0	30	38	43	2	6	53	30	0	40
U	Burn Time (sec.) (3) glow-out	0	65	55	93	2	11	115	52	0	107
L	Configuration (4)	Y	N	Y	Y	Y	N	Y	Y	Y	N
T	Residue (%) (5)	100	90	95	90	100	30	85	80	100	20
S	Melted (6)	N	N	N	N	N	N	N	N	N	N
	Color exterior	red	white	white	white	trans	white	white	white	trans	white
	Color interior	"	brown	lt.brn	beige	"	black	black	black	"	black
	Exterior Texture powder (6)	N	N	N	N	N	N	N	N	N	N
	Exterior Texture crusty (6)	N	Y	Y	Y	N	Y	Y	Y	N	Y
				(8)	(8)			(8)	(8)		

1. T.....Top B.....Bottom
2. N.....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. Ambient pressure air

TABLE 6-40 (cont'd)

ADDITION REACTION SILICONE RTV'S

PRELIMINARY FLAMMABILITY

-TEST CONDITIONS/RESULTS-

										Specimen	
			043B-2	054	054	054-2	054-3	055	055	055-2	055-3
	O_2 Pressure (psia)		6.2	(7)	6.2	6.2	6.2	(7)	6.2	6.2	6.2
RESULTS	Wire Ignition (1)		T/B	T	T/B	T/B	T/B	T/B	T/B	T/B	T
	Wire Failure (1)		T	T	T	B	B	T	B	T	T
	Smoke (2)		N			N	N			N	N
	Burn Time (sec.) (3)	flame out	26	0	30	35	30	3	26	35	25
		glow-out	53	0	56	82	57	3	43	66	49
	Configuration (4)		Y	Y	Y	Y		Y	N	Y	N
	Residue (%) (5)		50	100	90	95	90	100	70	90	50
	Melted (6)		N	N	N	N	N	N	N	N	N
	Color	exterior	white	trans	white	white	white	w.bl.	white	white	white
		interior	black	"	black	black	black	trans	black	black	black
	Exterior Texture	powdery (6)	N	N	N	N	N	N	N	N	N
		crusty (6)	Y	N	Y	Y	Y	N	Y	Y	Y

1. T.....Top B.....Bottom
2. N.....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. Ambient pressure air

TABLE 6-40 (cont'd)
ADDITION REACTION SILICONE RTV'S
PRELIMINARY FLAMMABILITY
-TEST CONDITIONS/RESULTS-

			Specimen								
			045B	045B	045B-2	046B	046B	046B-2	047B	047B-1	047B-2
	O_2 Pressure (psia)		(7)	6.2	6.2	(7)	6.2	6.2	(7)	6.2	6.2
	Wire Ignition (1)		T/B	T/B	T	T	T/B	T/B	T/B	T/B	T/B
R	Wire Failure (1)		T	T	T	T	T	T	T	T	T
E	Smoke (2)				N	N		N			
S	Burn Time	flame-out	0	27	28	0	40	50	2	36	30
U	(sec.) (3)	glow-out	20	35	40	0	60	82	2	60	54
L	Configuration (4)		Y	N	N	Y	Y	Y	Y	N	Y
T	Residue (%) (5)		95	90	95	100	90	90	100	90	90
S	Melted (6)		N	N	N	N	N	N	N	N	N
	Color	exterior	trans	white	white	black	white	white	trans	white	white
		interior	"	black	black	"	black	black	"	black	black
	Exterior	powder (6)	N	N	Y	N	N	N	N	N	N
	Texture	crusty (6)	N	Y	Y	N	Y	Y	N	Y	Y

1. T.....Top B.....Bottom
2. N.....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No

ADDITION REACTION SILICONE RT.V'S
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

		Specimen		
		047B-3	117-1B-1	118-1-1
	O_2 Pressure (psia)	6.2	6.2	6.2
RESULTS	Wire Ignition (1)	T/B	T/B	T/B
	Wire Failure (1)	T	B	T
	Smoke (2)	N	S	N
	Burn time (sec.) (3) flame-out	30	37	45
	Burn time (sec.) (3) glow-out	59	43	80
	Configuration (4)	Y	N	Y
	Residue (%) (5)	90	90	90
	Melted (6)	N	N	N
	Color: exterior	white	white	white
	Color: interior	black	red	black
	Exterior Texture powder (6)	N	N	N
	Exterior Texture crusty (6)	Y	Y	Y

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. Ambient pressure air

Table 6-41 is a tabulation of formulations utilizing RTV 655 with some inorganic flame retardant candidates. Justification for the interest in these particular additives has been reviewed in Progress Report No. 3 (Ref. 6-8) for this program.

Processing steps are shown in Table 11 of Reference 6-11, and in Table 6-42 of this report.

Antimony oxide was found (F-088) to completely inhibit the cure of the RTV 655, even after 18 hours. This apparently reduced its potential usefulness with halogen flame retardants in addition silicone RTV systems. Nevertheless, several attempts were made to utilize such synergistic behavior. Based upon prior art (Ref. 6-24) with polyethylene, the weight ratio of Sb_2O_3 to NH_4BF_4 was set at 2:1 for a total amount of 150 phr. The ratios for the other 2 fluorine compounds, KBF_4 and KPF_6, were adjusted with respect to molecular weights and total fluorine content. None of the formulations cured within 4 hours at 150°F. At the end of 45 hours at 150°F, all were partially cured but tacky. XPI-158 (zinc borate: $2ZnO \cdot 3B_2O_3 \cdot 3.3\text{-}3.7\ H_2O$), which had been found to accelerate addition reaction silicone RTV's, was substituted for 80% pbw for the Sb_2O_3. The ones with NH_4BF_4 and KBF_4 were partially cured and tacky after 4 hours. The one with KPF_6 was not cured at all. At the end of 30 hours, the first two were cured (no tack). The third one was still uncured.

It is seen in Table 12 of Reference 6-11 and in Table 6-43 of this report that except for several of the ammonium salts and the fluorinated compounds, the mechanical properties are generally quite poor, indicating that the approximate limit of their respective loadings (for the given particle size and shape) had been reached.

Table 6-44 shows the results of the preliminary flammability tests. None of the specimens survived the 16.5 psia oxygen environment. F-120 was the only one which was reasonably self-extinguishing and had negligible damage at 60/40, 16.2 psia. This was a formulation of 150 phr boric acid with a Me-Ø silicone RTV, 96-052. The latter, as received, was apparently loaded with titanium dioxide. The initial viscosity was 100 Poise when catalyzed. Upon adding the boric acid, the compound became almost unworkable due to its very high viscosity. F-142, based upon an unfilled Me-Ø silicone RTV, 655, and containing a higher percentage of titanium dioxide and a lower percentage of boric acid, was not acceptable at 6.2 psia oxygen. F-145, which had 150 phr boric acid, but only 50 phr titanium dioxide (all within an RTV 655 matrix), was also unacceptable. F-129 was also not suitable at 6.2. It contained only 150 phr titanium dioxide with the RTV 655. F-033, a blend of 150 phr boric acid and the RTV 655, also did not survive 6.2 psia oxygen. However, as noted above, the viscosity of F-120 is quite high. Further, the qualitative mechanical properties were also unsatisfactory.

TABLE 6-41

ADDITION REACTION SILICONE RTV'S WITH INORGANIC ADDITIVES

Component	S	Formulations (phr)													
		033	079	080	096	034	035	087	098	099	100	101	129	142	088
RTV 655A	7	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.
Boric Acid	9	150.													
Boric Acid (1)	9													100.	
Boric Acid,Anh.(1,2)	17		150.												
Borax (1)	9			150.											
Borax, Anh. (1,2)	17				150.										
Boron Phosphate	4					150.									
XPI-158 (3)	4						75.								
BUSAN 11-MI (4)	19							150.							
METSO ANH.48 (1,5)	11								150.						
SS65 Pwd. (1,6)	11									150.					
METSO 20 (1,2,7)	11										150.				
METSO 200 (1,2,8)	11											150.			
Titanium Dioxide	--												150.	100.	
Antimony Oxide	20														150.
RTV 655B	7	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.

TABLE 6-41 (cont'd)

ADDITION REACTION SILICONE RTV'S WITH INORGANIC ADDITIVES

		Formulations (phr)													
Component	S	102	103	104	112	113	093	092	115	116	107	106	105	110	111
RTV 655A	7	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.
1710 Glass Frit (10)	21	150.													
7570 Glass Frit (11)	21		150.												
7900 Glass Frit (12)	21			150.											
Vermiculite BO-5(13)	22				150.										
Vermiculite BO-4 (-100)(13)	22					150.									
325 Mica (14)	23						150.								
ASTRACAL SF (15)	24							150.							
AVIBEST C (16)	25								75.	25.					
DISPAL Alumina (17)	26										150.				
BURNIL (18)	2											25.			
HISIL 233 (19)	27												150.	75.	25.
RTV 655B	7	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.

TABLE 6-41 (cont'd)

ADDITION REACTION SILICONE RTV's WITH INORGANIC ADDITIVES

Component	S	Formulations (phr)									
		070A	070	071	072	073	074	075	120	145	173
96-004	12	100.	100.	100.							
SYLGARD 182	12				100.						
SYLGARD 186	12					100.					
SYLGARD 188	12						100.				
RTV 655A	7							100.		100.	100.
96-052	12								100.		
Boric acid	9	150.	150.					125.			
Boric acid (1)	9			125.	125.	125.	125.		150.	150.	
TiO_2	--									50.	
MAGLITE D (9)	3										150.
96-004 catalyst	12	10.	12.	10.							
S-182 catalyst	12				10.						
S-186 catalyst	12					10.					
S-188 catalyst	12						10.				
RTV 655B	7							10.		10.	10.
96-052 catalyst	12								10.		

TABLE 6-41 (cont'd)

ADDITION REACTION SILICONE RTV'S WITH INORGANIC ADDITIVES

Component	S	Formulations (phr)							
		272	273-1	273-2	275-2	276	274-1	274-2	277
RTV 655A	7	100.	100.	100.	100.	100.	100.	100.	100.
Ammonium Biborate (1)	10	150.							
Ammonium Borate	10		150.						
Ammonium Borate (1)	10			150.					
Ammonium Phosphate, Monobasic (1)	10				150.				
Ammonium Phos., Dibasic (1)	10					150.			
Ammonium Bromide	10						150.		
Ammonium Bromide (1)	10							150.	
Ammonium Carbonate (20,21)	10								150.
RTV 655B	7	10.	10.	10.	10.	10.	10.	10.	10.

TABLE 6-41(cont'd)

ADDITION REACTION SILICONE RTV'S WITH INORGANIC ADDITIVES

		Formulations (phr)								
Component	S	336	337	338	339	340	341	342	343	344
SYLGARD-186	12	100.	100.	100.	100.	100.	100.	100.	100.	100.
NH_4BF_4	20	150.			50.			50.		
KBF_4	10		150.			56.			56.	
KPF_6	10			150.			56.			56.
Sb_2O_3	20				100.	94.	94.	20.	18.8	18.8
XPI-158(3)	4							80.	75.2	75.2
S-186 Catalyst	12	10.	10.	10.	10.	10.	10.	10.	10.	10.

S.....Source (See Table 6-1)

1. 100% through 170 mesh
2. Dessicated
3. Zinc borate
4. Barium metaborate monohydrate
5. Anhydrous sodium metasilicate
6. Anhydrous sodium silicate (ground glass)
7. Pentahydrate sodium metasilicate
8. Sodium orthosilicate (9.5% H_2O)
9. Magnesium oxide
10. Pyrex, extra hard
11. High lead
12. VYCOR (96% silica)
13. Hydrated magnesium aluminum iron silicate
14. Hydrated sodium potassium aluminum silicate
15. Potassium aluminum silicate
16. Hydrated magnesium iron silicate
17. Hydrated aluminum oxide
18. Lithium magnesium silicate
19. Silicon dioxide
20. Ball Milled
21. Reagent grade

TABLE 6-42

ADDITION REACTION SILICONE RTV'S WITH INORGANIC ADDITIVES

- FORMULATION PROCESSING -

Formulation No. F-	Cure (1)		Post Cure	
	Time (hrs/min)	Temp. (°F)	Time (hrs/min)	Temp. (°F)
071A	-/15	150	13/-	RT
071B	"	"	"	"
			22/40	150
072-1A	"	"	13/-	RT
072-1B	"	"	"	"
			22/40	150
072-2	16/-	"	--	--
073A	-/10	"	13/-	RT
073B	"	"	"	"
			22/40	150
074-1A	"	"	13/-	RT
074-1B	"	"	"	"
			22/40	150
074-2	4/50	"	--	--
075	16/-	"	--	--
145A	1/-	"	--	--
145B	"	"	2/45	150
173	(See text)		--	--
272-1	24/-	150	--	--
272-2	5/-	"	--	--

1. Time in mold

TABLE 6-42 (cont'd)

ADDITION REACTION SILICONE RTV'S WITH INORGANIC ADDITIVES

- FORMULATION PROCESSING -

Formulation No. F-	Cure (1) Time (hrs/min)	Cure (1) Temp. (°F)	Post Cure Time (hrs/min)	Post Cure Temp. (°F)
273-1	22/25	150	--	--
273-2	16/20	"	--	--
275	21/25	"	--	--
276	4/30	"	--	--
274-1	21/45	"	--	--
274-2	15/25	"	--	--
277	28/-	RT	--	--
336	2/-	150	--	--
337	"	"	--	--
338	"	"	--	--
339	45/-	"	--	--
340	"	"	--	--
341	"	"	--	--
342-2	41/-	"	36/-	150
343-2	"	"	"	"
344	4/-	"	26/-	"

TABLE 6-43

ADDITION REACTION SILICONE RTV'S WITH INORGANIC ADDITIVES CHARACTERISTICS

-CURED/POST CURED-

	Formulation								
Properties	071A	071B	072-1A	072-1B	072-2	073A	073B	074-1A	074-1B
Cured	Y	Y	N	Y	Y	Y	Y	N	?
Tacky	N	N	Y	N	N	S	N	-	S
Flexible	Y	Y	Y	F	X	Y	Y	-	Y
Memory	P	F	N	F	F	P	X	-	P
Cohesion	P	P	-	F	P	P	F	-	P
Tear Resistance	P	P	-	P	P	P	P	-	P
Crack Resistance (1)	NG	NG	-	X	X	X	X	-	P
Crack Propagation (1)	F	NG	-	NG	P	X	X	-	P
Porous	N	N	-	N	N	N	N	-	N
Adheres to unclean alum. Foil cup	-	-	-	-	-	-	-	-	-
Cheesy	-	Y	-	N	N	Y	N	-	N

Y.....Yes VS.....Very Slight S.....Slight N.....No
X.....Satisfactory F.....Fair P.....Poor NG.....Not Good

1. 180° bend (face-to-face contact)

TABLE 6-43 (cont'd)

ADDITION REACTION SILICONE RTV'S WITH INORGANIC ADDITIVES CHARACTERISTICS

-CURED/POST CURED-

Properties	Formulation 074-2	075	145A	145B	272-1	272-2	273-1	273-2
Cured	?	Y	Y	Y	Y	Y	Y	Y
Tacky	S	N	N	N	N	N	N	N
Flexible	Y	Y	Y	Y	F	F	X	X
Memory	F	F	X	F	-	F	X	F
Cohesion	P	P	F	F	-	P	P	F
Tear Resistance	P	P	P	P	-	P	P	F
Crack Resistance (1)	X	X	X	X	-	NG	-	X
Crack Propagation (1)	X	F	X	X	-	NG	-	P
Porous	N	N	?	?	N	N	N	N
Adheres to unclean alum. Foil Cup	-	-	-	-	X	F	P	F
Cheesy	N	VS	N	N	Y	Y	S	N

TABLE 6-43 (cont'd)

ADDITION REACTION SILICONE RTV'S WITH INORGANIC ADDITIVES CHARACTERISTICS

-CURED/POST CURED-

	Formulation							
Properties	275	276	274-1	336	337	338	342-2	343-2
Cured	Y	Y	Y	Y	Y	Y	Y(?)	Y(?)
Tacky	N	N	N	N	N	N	S	Y
Flexible	X	X	X	Y	Y	Y	X	Y
Memory	X	X	X	Y	Y	Y	X	X
Cohesion	P	P	F	F	F	F	P	P
Tear Resistance	P	P	F	F	F	F	P	P
Crack Resistance (1)	X	X	-	X	X	X	X	X
Crack Propagation (1)	X	X	-	X	X	X	X	X
Porous	N	N	N	N(2)	-	-	N	N
Adheres to unclean alum. Foil cup	F	X	P.	N	N	N	-	-
Cheesy	N	N	P	-	-	-	-	N

2. Very slight due to large particle size of NH_4BF_4.

TABLE 6-44

ADDITION REACTION SILICONE RTV'S WITH INORGANIC FLAME RETARDANTS
PRELIMINARY FLAMMABILITY
-TEST CONDITIONS/RESULTS-

			Specimen								
			033	033	033-2	033-3	033-2-1	079	079	080	096
	O_2 Pressure (psia)		(7)	6.2	6.2	6.2	6.2	6.2	9.72	6.2	6.2
RESULTS	Wire Ignition (1)		T/B	T/B	B	T/B	T/B	T	T/B	T	T/B
	Wire Failure (1)			T	B	T/B	T	T	B	T	T
	Smoke (2)		P	N	N	N	S	S	S	S	S
	Burn Time (sec) (3)	flame-out	0	75	$\sim$45	7	54	45	37	38	40
		glow-out	0	81	$\sim$54	7	59	47	43	38	45
	Configuration (4)		Y	Y	Y	Y	Y	Y	Y	Y	Y
	Residue (%) (5)		100	90	90	100	90	90	90	85	95
	Melted (6)		N	N	N	N	N	N	N	N	N
	Color	exterior	white	white	white	white	white	white	white	white	white/bl.
		interior	black	black	black	"	black	"	"	black	black
	Exterior Texture	powder (6)	N	N	N	N	Y	N	N	N	N
		crusty (6)	N	Y	Y	N	Y	Y	Y	Y	Y
									(9)		

1. T.....Top B.....Bottom
2. N.....None S.....Some P.....Profuse.
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. Ambient pressure air

9. 60% O_2/40% N_2 = 16.2 psia

TABLE 6-44 (cont'd)

ADDITION REACTION SILICONE RTV'S WITH INORGANIC FLAME RETARDANTS PRELIMINARY FLAMMABILITY -TEST CONDITIONS/RESULTS-

			Specimen							
			034	034	034-2	035-1	035-1-1	035-1-2	087-2	098-2-1
	O_2 Pressure (psia)		(7)	6.2	6.2	(7)	6.2	6.2	6.2	6.2
RESULTS	Wire Ignition (1)		T/B	T/B	T/B	T/B	T	T/B	T	T
	Wire Failure (1)		T	T	B	T	T	B	T	T
	Smoke (2)		N		N			N	N	S
	Burn Time (sec.) (3)	flame-out	0	58	48	3.5	50	53	50	36
		glow-out	0	82	71	3.5	63	69	53	41
	Configuration (4)		Y	Y	Y	Y	Y	Y	Y	N
	Residue (%) (5)		100	90	90	100	90	90	90	95
	Melted (6)		N	N	N	N	N	N	N	N
	Color	exterior	beige	white	white	white	white	white	white	white
		interior	"	black	black	"	black	black	black	black
	Exterior Texture	powder (6)	N	N	N	N	N	N	N	N
		crusty (6)	N	Y	Y	N	Y	Y	Y	Y

1. T.....Top | B.....Bottom
2. N.....None | S.....Some | P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No;
5. Amount left in (4)
6. Yes or No
7. Ambient pressure air

TABLE 6-44 (cont'd)

ADDITION REACTION SILICONE RTV'S WITH INORGANIC ADDITIVES
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

			Specimen							
			099-2B-1	129-2B-1	142-1-1	102-2B-1	103-2B-1	104-2B-1	112-2B-1	113-2B-1
	O_2 Pressure (psia)		6.2	6.2	6.2	6.2	6.2	6.2	6.2	6.2
RESULTS	Wire Ignition (1)		T/B	T/B	T/B	T/B	T/B	T/B	T/B	T/B
	Wire Failure (1)		B	T	B	B	T	B	T	B
	Smoke (2)		N	N	S	N	N	N	N	N
	Burn time (sec.) (3)	flame-out	36	45	43	43	55	40	41	38
		glow-out	44	95	53	84	110	93	68	63
	Configuration (4)		Y	Y	Y	Y	Y	Y	Y	Y
	Residue (%) (5)		95	90	90	95	90	95	95	95
	Melted (6)		N	N	N	N	N	N	N	N
	Color	exterior	white	white	white	white	white	white	white	white
		interior	black	"	"	black	black	tan	black	tan
	Exterior Texture	powder (6)	N	N	N	N	N	N	N	N
		crusty (6)	Y	Y	Y	Y	Y	Y	Y	Y

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No

TABLE 6-44 (cont'd)

ADDITION REACTION SILICONE RTV'S WITH INORGANIC ADDITIVES
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

			Specimen						
			092-2B-1	070A	070A-1	070A	070A	071	072-2
	O_2 Pressure (psia)		6.2	6.2	6.2	9.72 (60%)	16.5	6.2	6.2
RESULTS	Wire Ignition (1)		T	T/B	T/B	T	B	T/B	T
	Wire Failure (1)		T	B	T	T	B	B	T
	Smoke (2)		N	N	N	N	P	S	S
	Burn Time (sec.) (3)	flame-out	42	57	60	39	30	46	48
		glow-out	83	57	60	39	30	46	53
	Configuration (4)		Y	Y	Y	Y	Y	Y	Y
	Residue (%) (5)		90	95	95	>95	90	95	85
	Melted (6)		N	N	N	N	N	N	N
	Color	exterior	white	white	white	red/wht	white	white	white
		interior	tan-gray	black	black	red	black	black	black
	Exterior Texture	powdery (6)	N	N	N	Y	Y	N	Y
		crusty (6)	Y	Y	Y	Y	N	Y	Y

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No

TABLE 6-44 (cont'd)

ADDITION REACTION SILICONE RTV'S WITH INORGANIC ADDITIVES
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

		Specimen						
		073	074-2	075	120-IB-1	120-IB-2	120-IB-1	120-IB-1
	O_2 Pressure (psia)	6.2	6.2	6.2	6.2	6.2	9.72 (60%)	16.5
RESULTS	Wire Ignition (1)	T/B	T/B	T	T/B	T/B	T/B	T/B
	Wire Failure (1)	B	T	T	T	T/B	T	B
	Smoke (2)	S	N	S	N	N	N	P
	Burn time (sec.) (3) flame-out	35	68	68	8	3.5	19	42
	Burn time (sec.) (3) glow-out	41	79	76	8	3.5	19	42
	Configuration (4)	Y	Y	Y	Y	Y	Y	Y
	Residue (%) (5)	90	85	90	100	100	100	95
	Melted (6)	N	N	N	N	N	N	N
	Color exterior	white	white	white	white	white	white	white
	Color interior	black	black	black	"	"	"	gray
	Exterior Texture powdery (6)	N	Y	N	N	N	N	N
	Exterior Texture crusty (6)	Y	Y	Y	N	N	N	Y

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No

TABLE 6-44 (cont'd)

ADDITION REACTION SILICONE RTV'S WITH INORGANIC ADDITIVES PRELIMINARY FLAMMABILITY

- TEST CONDITIONS/RESULTS -

		Specimen						
		145-1B-1	272-2-1	273-2-1	274-2-1	275-2-1	276-2-1	336-2-1
	O_2 Pressure (psia)	6.2	6.2	6.2	6.2	6.2	6.2	6.2
RESULTS	Wire Ignition (1)	T/B	B	T/B	T/B	T/B	T/B	T
	Wire Failure (1)	B	B	T/B	T	T	B	T
	Smoke (2)	S	N	N	N	P	P	P
	Burn time (sec.) (3) flame-out	46	50	75	85	45	49	55
	Burn time (sec.) (3) glow-out	49	52	85	96	45	49	58
	Configuration (4)	Y	Y	Y	Y	Y	Y	Y
	Residue (%) (5)	90	90	90	90	90	90	50
	Melted (6)	N	N	N	N	N	N	N
	Color exterior	white	white	white	white	white	white	white
	Color interior	---	---	---	black	---	---	& black
	Exterior Texture powdery (6)	N	N	N	N	N	N	Y
	Exterior Texture crusty (6)	Y	Y	Y	Y	Y	Y	Y

1. T....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No

TABLE 6-44 (cont'd)

ADDITION REACTION SILICONE RTV'S WITH INORGANIC ADDITIVES
PRELIMINARY FLAMMABILITY
-TEST CONDITIONS/RESULTS-

		Specimen						
		337-2-1	338-2-1	342-2-1	342-2-2	342-2-1	343-2-1	343-2-1
	O_2 Pressure (psia)	6.2	6.2	6.2	6.2	9.72 (60%)	6.2	9.72 (60%)
R	Wire Ignition (1)	T	T	T/B	B	---	---	T/B
E	Wire Failure (1)	T	---	T	B	---	T/B	T
S	Smoke (2)	S	P	N	N	---	---	S
U	Burn time (sec.) (3) flame-out	42	37	0	0	28	0	45
	Burn time (sec.) (3) glow-out	42	37	0	0	38	0	45
L	Configuration (4)	N	---	Y	Y	---	Y	Y
T	Residue (%) (5)	15	---	100	100	---	100	95
S	Melted (6)	N	N	N	N	---	N	N
	Color exterior	(7)	(7)	white	white	---	white	white
	Color interior	(7)	(7)	"	"	---	"	black
	Exterior Texture powdery (6)	Y	Y	N	N	---	N	N
	Exterior Texture crusty (6)	Y	Y	N (8)	N (8)	---	N (8)	Y

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. Black and white
8. Test questionable (TFE pulled away from wire, but didn't ignite)

6.2.1.3 Organic Flame Retardants - Formulations which contained organic halogenated compounds were prepared (see Table 6-45). Selection was further restricted to those which either had no hydrogen present, or which had very little hydrogen.

F-036, containing hexabromobenzene, had a low viscosity, but was difficult to cure. In fact, no cure was achieved at 150°F after almost 9 hours. Over three days at 200°F was required to cure the material even though the pot life was only between 12 and 24 hours. Specimens from this formulation had a foamy interior, a smooth surface, and were flexible but quite cheesy. Some form of inhibition was clearly evident.

Another non-curing formulation, shown in Table 6-45, was F-143. No cure was obtained even after 30 days at 150°F. The filler, FC-134 (S-2) is an organic compound having a stable fluorocarbon tail and a cationic solubilizing group. On the other hand, FC-95 (F-144 and -148), a similar type compound, has an anionic solubilizing group, and did cure after 25 hours at 150°F. (No further information was available for FC-134 or FC-95 due to the vendor, 3M, considering both materials of a proprietary nature.) The resultant product was not tacky, was flexible, had good memory and crack resistance, and was neither cheesy nor porous. However, it had poor tear resistance and cohesion and readily propagated a crack.

When 2 drops of bromotrichloromethane per 1.5 grams of hexabromobenzene was added to F-036, the specimen (F-089) was slightly tacky after 57 hours at 150°F. An increase to 10 drops resulted in a cure in 56 hours (F-090).

F-081 was prepared with Dechlorane 510, a high chlorine, non-hydrogen containing organic compound. F-084 was made with Dechlorane 515. It is not known whether any hydrogen is present. Both of these cured.

The tetrabromobisphenol A of F-083 has only 2.2% hydrogen, whereas neither tetrabromo phthalic anhydride (F-085) nor tetrachloro phthalic anhydride (F-086) have any hydrogen. However, these three organics appear to either inhibit or slow down the cure rate of the RTV 655. F-083 was only semi-cured after 46 hours at 150°F. F-085 was only semi-cured after 4 days. F-086 required almost 7 hours at 150°F to cure. Additional processing and cured characteristics data were reported in Tables 15 and 16, respectively, of Reference 6-11.

The preliminary flammability test results are shown in Table 6-46. Simply stated, at the concentrations indicated in Table 6-45, none of the formulations tested survived 6.2 psia oxygen.

6.2.1.4 Inorganic/Organic Flame Retardants - An attempt to cure the hexabromobenzene/RTV 655 system at 150° and at 200°F (see Table 15 of Reference 6-11) with antimony oxide proved unsuccessful (F-078). Since Busan 11-MI has reportedly

TABLE 6-45

ADDITION REACTION SILICONE RTV'S WITH ORGANIC ADDITIVES

		Formulations (phr)											
Component	S	036	089	090	081	084	083	085	086-1	086-2	144	148	143
RTV 655A	7	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.
Hexabromobenzene	28	150.	150.	150.									
Bromotrichloromethane	28		(5)	(6)									
DECHLORANE 510 (1)	16				150.								
DECHLORANE 515 (1)	16					150.							
Firemaster BP 4A (2)	28						150.						
Firemaster PHT4 (3)	28							150.					
TCPA (4)	16								150.	150. (7)			
FC-95 (8)	1										150.	150.	
FC-134 (8)	1												150.
RTV 655B	7	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.

S......Source (See Table 6-1)
1. Proprietary product: high chlorine content
2. Tetrabromo bisphenol-A
3. Tetrabromo phthalic anhydride
4. Tetrachlorophthalic anhydride
5. 2 drops/1.00 gm. RTV 655A
6. 10 drops/1.00 gm. RTV 655A
7. 100% through 170 mesh
8. Fluorocarbon solid surfactant

TABLE 6-46

ADDITION REACTION SILICONE RTV'S WITH ORGANIC ADDITIVES
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

			Specimen					
		036	036-1	036-2	081	084-2	086-2B	148-1-1
	O_2 Pressure (psia)	(7)	6.2	6.2	6.2	6.2	6.2	6.2
RESULTS	Wire Ignition (1)	T/B	T	T	T	T/B	T/B	T/B
	Wire Failure (1)	B	T	T	T	T	B	B
	Smoke (2)	P	---	---	S	S	S	S
	Burn time (sec.) (3) flame-out	0	5	54	45	48	80	28
	Burn time (sec.) (3) glow-out	0	5	69	133	165	84	40
	Configuration (4)	Y	Y	N	Y	Y	N	N
	Residue (%) (5)	95	100	40	50	85	80	10
	Melted (6)	N	N	N	N	N	N	N
	Color exterior	beige	beige	white	black	black	gray	white
	Color interior	black	black	black	white	white	black	black
	Exterior Texture powdery (6)	N	N	Y	N	Y	Y	N
	Exterior Texture crusty (6)	N	N	Y	Y	Y	Y	Y

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. Ambient pressure air

been used as a partial replacement for antimony oxide, and since there was no difficulty in curing it with the RTV 655 type system, a formulation was prepared (see Table 6-47) with it and hexabromobenzene (F-091). Cure at 150°F was sufficient in 1-1/2 hours to demold the specimens. They were then post cured for an additional 15-1/2 hours. The latter thermal exposure seems to have slightly stiffened the specimen (see Table 16 of Reference 6-11). F-094 was prepared with less of the borate. This somewhat reduced the effect of temperature cycling. F-097 was similar to F-094 with the exception that antimony oxide was also added. This required more time to cure than F-094. An effort was made to cure the hexabromobenzene in conjunction with a potassium aluminum silicate (F-095). The specimen was only semi-cured after 24 hours at 150°F. It didn't appear cured until several weeks later, at room temperature. A Dechlorane 510/antimony oxide system cured in 5 hours 15 minutes.

It is seen from Table 6-48 that none of the flammability test specimens from this group passed the 6.2 psia oxygen environment.

6.2.2 Two-Part Condensation Reaction Systems

As in the case of other condensation systems, a by-product results herein which can be a source of outgassing and can result in reduced mechanical properties, especially in thick sections. Therefore they are generally less desirable than the addition types. However, these condensation systems are much less prone to inhibition.

6.2.2.1 Unfilled Formulations - The formulations are shown in Table 6-49.

F-270 was an evaluation of a dimethyl analog of the two-part condensation, methylphenyl (Me-Ø) system used in F-048. Some cure difficulty was encountered with F-270. In fact, no cure was obtained in 5 hours 15 minutes at 150°F. Cure was achieved after an additional 41 hours 51 minutes at 150°F. At that point, the "cured" material was slightly tacky, but was flexible, had good memory, cohesion, and tear resistance, was crack resistant, didn't propagate an induced crack, and was not porous or cheesy.

The processing data and cured characteristics of the remaining formulations shown in Table 6-49, are tabulated in Tables 5 and 6 of Reference 6-11, respectively.

F-001 is the formulation being used by NASA-MSC as an igniter for flammability testing (Ref. App. A, para. 3.4.1.2, and Ref. 6-1). It was used as the initial mold tryout for the preliminary flammability specimen mold (Ref. Section 5.2 of this report). These specimens were also the first samples tested in the flammability tester (Ref. Section 5.1 of this report).

As noted in Table 5 of Reference 6-11, F-050 had insufficient pot life. F-051 contained less catalyst.

TABLE 6-47

ADDITION REACTION SILICONE RTV'S WITH INORGANIC/ORGANIC ADDITIVES

Component	S	Formulations (phr)					
		078	091	094	097	095	082
RTV 655A	7	100.	100.	100.	100.	100.	100.
Hexabromobenzene	28	150.	150.	150.	150.	150.	
Bromotrichloromethane	28						
DECHLORANE 510 (1)	16						150.
Antimony Oxide	20	75.			50.		75.
BUSAN 11-MI (2)	19		100.	50.	50.		
ASTRACAL SF (3)	24					100.	
Titanium Dioxide	--						
RTV 655B	7	10.	10.	10.	10.	10.	10.

S......Source (See Table 6-1)
1. Proprietary product: high chlorine content
2. Barium metaborate monohydrate
3. Potassium aluminum silicate

TABLE 6-48
ADDITION REACTION SILICONE RTV'S WITH INORGANIC/ORGANIC ADDITIVES
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

		Specimen			
		091-2-1	094-2	097	082
	O_2 Pressure (psia)	6.2	6.2	6.2	6.2
RESULTS	Wire Ignition (1)	B	T/B	T	T/B
	Wire Failure (1)	B	B	T	T/B
	Smoke (2)	P	P	P	P
	Burn time (sec.) (3) flame-out	75	65	53	58
	Burn time (sec.) (3) glow-out	82	97	93	105
	Configuration (4)	Y	Y	N	N
	Residue (%) (5)	90	90	90	50
	Melted (6)	N	N	N	N
	Color: exterior	white	white	white	black
	: interior	black	black	black	gray
	Exterior Texture powdery (6)	N	Y	N	N
	Exterior Texture crusty (6)	Y	Y	Y	Y

1. T.....Top B.....Bottom
2. N.....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No

TABLE 6-49

TWO-PART CONDENSATION REACTION SILICONE RTV'S

Component	S	048	049	001	050	051	058	270
RTV 511	7	100.						
RTV 560	7		100.	85.				
RTV 577	7			15.				
RTV 602	7				100.	100.		
RTV 603	7						100.	
RTV 11	7							100.
DTD (2)	7	0.3	0.3	0.5				0.3
SRC-05	7				0.3	0.2	0.2	

S......Source (See Table 6-1)
2. Dibutyl tin dilaurate (Thermolite 12)

The catalyzed systems, in order of increasing viscosity, were: F-050 /-051, <-048, <-049, <-001.

As in the case of the additional systems, these compounds were essentially unchanged when ignited in ambient pressure air (see Table 6-50). The sole exception was -049B, which was slightly burned on one end. Although those exposed to 6.2 psia oxygen apparently completely burned up, the residue from these maintained their original configuration generally better than the addition types tested to date (i.e. better char structure).

6.2.2.2 Flame Retardants - Various inorganic additives were formulated with either RTV 511 or RTV 560 in order to improve their flammability resistance (see Table 6-51). Both RTV's were prepared with only 125 phr boric acid since 150 phr was physically not feasible due to the enormous increase in viscosity. The viscosity of F-076 was extremely high, but it still could be made into flammability test specimens. However, the viscosity of F-077 was too high even at the 125 phr level, to fabricate any test specimens.

A catalyzed RTV 511 master batch (F-208) was prepared in order to evaluate the fluorinated inorganic compounds shown in Table 6-51. The remaining additive systems were individually blended with the RTV and the catalyst, except for F-202. This formulation was initially prepared without F-208, but was found to have an excessively high viscosity. F-208 was added as a diluent. F-204 was obviously quite hygroscopic. F-205 was prepared first from F-208 (F-205-1), and eventually "cured" to an extremely poor cohesive state, and had slight tack. The same system was separately formulated (i.e. No. F-208) as F-205-2. Again, the specimens did not hold together. F-203 resulted in a powdery product. F-206 was extremely hygroscopic (i.e. "cured" specimens wet after exposure to ambient air), started to "cure" while blending, and became stringy. F-207 was similar to F-206. F-269 appeared to "cure" while blending.

F-211 utilized antimony oxychloride as the potential flame retardant. Antimony oxide, by itself, has not been found by other investigators, as well as by this Project, to be an effective flame retardant. However, if it is used in conjunction with halogen containing compounds, especially those having chlorine, an apparently synergistic relationship is developed with respect to flame retardation. It has been postulated (Ref. 6-25) that antimony oxychloride, SbOCl, is formed upon ignition. The vapor created by this reaction acts as a blanket and tends to inhibit the spread of burning after the ignition source is removed. (It should be noted that SbOCl is somewhat hygroscopic even though it is insoluble in water.)

The processing data and cured characteristics of the above compounds are shown in Table 6-52 and Table 6-53, respectively. The flammability results are tabulated in Table 6-54. None of the specimens from this group satisfactorily survived 6.2 psia oxygen.

TABLE 6-50
TWO-PART CONDENSATION REACTION SILICONE RTV'S
PRELIMINARY FLAMMABILITY
-TEST CONDITIONS/RESULTS-

			Specimen											
			048B	048B	048B-2	048B-3	049B	049B	049B-2	001-1	001-2	001	001-3	001B-4
	O_2 Pressure (psia)		(7)	6.2	6.2	6.2	(7)	6.2	6.2	(7)	(7)	6.2	6.2	6.2
R	Wire Ignition (1)		T	T/B	T	T/B	T/B	T/B	T/B		T/B	T/B	T/B	T/B
E	Wire Failure (1)		T	T	T	T	T	B	T			T	B	B
S	Smoke (2)		N		N	S			N					N
U	Burn Time (sec.) (3)	flame-out	0	40	40	53	1.5	40	53	0	0	55	38	46
		glow-out	0	45	50	60	1.5	50	68	0	0	72	38	61
L	Configuration (4)		Y	N	N	N	Y	N	Y	Y	Y	N	Y	Y
T	Residue (%) (5)		100	90	90	90	100	90	90	100	100	90	90	90
S	Melted (6)		N	N	N	N	N	N	N	N	N	N	N	N
	Color	exterior	white	white	white	white	red	white	white	red	red	white	white	white
		interior	"	black	black	wh/gray	"	red	black	"	"	red	red	black
	Exterior Texture	powder (6)	N	N	N	Y	N	N	N	N	N	N	N	N
		crusty (6)	N	Y	Y	Y	N	Y	Y	N	N	Y	Y	Y

1. T.....Top B.....Bottom
2. N.....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. Ambient pressure air

TABLE 6-50 (cont'd)
TWO-PART CONDENSATION REACTION SILICONE RTV'S
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

		Specimen							
		051B	051B	051B-2	051B-3	058	058	058-2	270-1-1
	O_2 Pressure (psia)	(7)	6.2	6.2	6.2	(7)	6.2	6.2	(7)
RESULTS	Wire Ignition (1)	T/B	T/B	T/B	T/B	T	T	T	T/B
	Wire Failure (1)	T	T/B	B	B	T	T	T	T
	Smoke (2)			S	N	N	N	S	N
	Burn time (sec.) (3) flame-out	0	38	30	30	0	40	40	45
	glow-out	0	82	36	38	0	73	77	77
	Configuration (4)	Y	N	N	N	Y	N	Y	Y
	Residue (%) (5)	100	90	90	90	100	90	80	90
	Melted (6)	N	N	N	N	N	N	N	N
	Color: exterior	trans.	white	white	white	trans.	white	white	white
	: interior	"	black	black	black	"	black	black	---
	Exterior Texture powdery (6)	N	N	N	N	N	N	Y	N
	crusty (6)	N	Y	Y	Y	Y	Y	Y	Y

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. Ambient pressure air

TABLE 6-51

TWO-PART CONDENSATION REACTION SILICONE RTV'S WITH ADDITIVES

Component	S	Formulations (phr)											
		076	077	208	202	204	205-1	205-2	203	206	207	269	211
RTV 511	7	100.		100.	100.			100.				100.	100.
RTV 560	7		100.										
F-208-1	13				25.	100.	100.		100.	100.	100.		
Boric acid (1)	9	125.	125.										
Ca_3B_6	29				150.								
CaBF	29					150.							
$CaSiF_6 \cdot 2H_2O$	29						150.	150.					
SbF_3	29								150.				
$Co_2(BF_4)_2$	29									150.			
$Ni(BF_4)_2 \cdot 6H_2O$	29										150.		
$(NH_4)_2CO_3 \cdot H_2O$ (2)	10											150.	
SbOCl	30												150.
DTD (3)	7	0.3	0.3	0.3	0.3			0.3				0.6	0.3

S......Source (See Table 6-1)
1. 100% through 170 mesh
2. Reagent grade
3. Dibutyl tin dilaurate (Thermolite 12)

TABLE 6-52

TWO-PART CONDENSATION REACTION SILICONE RTV'S WITH ADDITIVES

- FORMULATION PROCESSING -

Formulation No. F-	Cure (1) Time (hrs/min)	Cure (1) Temp. (°F)	Post Cure Time (hrs/min)	Post Cure Temp. (°F)
076-1	4/28	150	--	--
076-2	5/-	"	--	--
077	4/18	"	--	--
202	2/30	"	1/30	150
204	44/-	"	--	--
205-1	"	"	--	--
205-2	48/-	"	--	--
203	44/-	"	--	--
206	1/4	"	--	--
207	-/44	"	--	--
269	(See text)		--	--
211-1	4/20	150	--	--
211-2	20/10	"	--	--

1. Time in mold

TABLE 6-53

TWO-PART CONDENSATION REACTION SILICONE RTV'S WITH ADDITIVES CHARACTERISTICS

-CURED/POST CURED-

	Formulation					
Properties	076-1	076-2	202-1A	202-1B	205-2	211-2
Cured	Y	Y	Y	Y	N	Y
Tacky	VS	N	N	N	N	N
Flexible	Y	Y	Y	Y	Y	Y
Memory	P	F	Y	Y	NG	Y
Cohesion	P	P	F	P	NG	F
Tear Resistance	P	P	P	P	NG	F
Crack Resistance (1)	P	P	X	NG	NG	P
Crack Propagation (1)	F	P	X	NG	NG	P
Porous	N	N(?)	-	-	Y	S
Adheres to unclean alum. Foil cup	N	-	N(3)	-	P	P
Cheesy	VS	N	N(2)	N(4)	gummy	N

Y.....Yes
VS.....Very Slight
S.....Slight
N.....No
X.....Satisfactory
F.....Fair
P.....Poor
NG.....Not Good

1. 180° bend (face-to-face contact)
2. Cured
3. R.T. Cured specimen
4. Cured and post cured

TABLE 6-54
TWO-PART CONDENSATION REACTION SILICONE RTV'S WITH ADDITIVES
PRELIMINARY FLAMMABILITY
– TEST CONDITIONS/RESULTS –

		Specimen							
		076-1-1	076-1-2	076-2-1	076-2-2	076-2-1	076-2-1	202-1-1	211-2-1
	O_2 Pressure (psia)	6.2	6.2	6.2	6.2	9.72 (60%)	16.5	6.2	6.2
RESULTS	Wire Ignition (1)	T	T/B	T/B	T/B	T/B	T/B	T/B	T/B
	Wire Failure (1)	T	B	T	B	B	B	T/B	B
	Smoke (2)	N	N	N	N	S	S	S	P
	Burn time (sec.) (3) flame-out	4	8	3.5	86	75	63	72	45
	Burn time (sec.) (3) glow-out	4	8	3.5	86	82	63	118	54
	Configuration (4)	Y	Y	Y	Y	Y	Y	N	N
	Residue (%) (5)	100	100	100	95	95	90	0	90
	Melted (6)	N	N	N	N	N	N	N	N
	Color: exterior	white	white	white	white	white	white	black	white
	Color: interior	"	"	"	gray	"	"	---	---
	Exterior Texture powdery (6)	N	N	N	N	N	N	N	N
	Exterior Texture crusty (6)	N	N	N	Y	Y	Y	Y	Y

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No

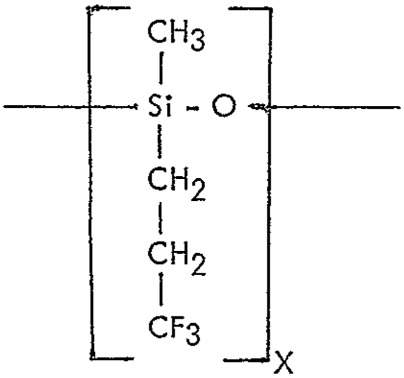
6.2.3 Flame Retardant Polyols

The objective of this group of formulations (see Table 6-55) was to determine if flame retarding polyol liquids could be incorporated into a basic silicone RTV structure to decrease the viscosity while improving the flammability resistance (or at least, not deteriorating it).

The general tacky and/or greasy feel to "cured" (see Table 6-56) specimens in this group, and their poor cohesion and tear resistance (see Table 6-57) were somewhat conclusive indications that the polyols had inadequate compatibility with the RTV's, regardless of the latter's type. In addition, tacky and/or greasy surfaces are not conducive to adhesion to such substrates as wires, PC boards, etc. No further evaluation was conducted.

6.3 FLUOROSILICONE RTV POLYMERS

As seen in Table 6-24, the hydrogen content of the fluorosilicone RTV's is almost half that of the conventional dimethyl silicone RTV's. In addition, the incorporation of 36.5% (i.e. parts by weight) (Ref. 6-15) of fluorine in the molecule of the former's base polymer would clearly be an aid in flame retardation. This data for the base polymer agrees quite well with the following structure:



$$\left[\, -\overset{\displaystyle \mathrm{CH_3}}{\underset{\displaystyle \mathrm{CH_2CH_2CF_3}}{\mathrm{Si}}}-\mathrm{O}- \,\right]_X$$

However, the following factors must be considered in its use herein.

a. Intrinsic viscosity is somewhat higher than that of a comparable dimethyl silicone RTV; loading with flame retardants is, hence, more limited. On the other hand, need for such additives is less than that for a conventional silicone RTV, for a given environment.
b. Dow Corning is at this time, and will be for the forseeable future, the sole source for commercial fluorosilicone RTV's due to their patent position with this class of materials.
c. All presently available fluorosilicone RTV's are non-phenyl types.
d. Dow Corning does not generally recommend their fluorosilicone RTV's for service in excess of 550°F due to possible evolution of toxic thermal decomposition products.

TABLE 6-55

SILICONE RTV'S WITH FLAME RETARDANT POLYOLS

		Formulations (phr)													
Component	S	153	154	156	157	214	158	159	201	155	150	151	162	161	152
SYLGARD 182	12	100.									100.				
RTV 655A	7		100.	100.	100.							100.			
RTV 511	7					100.	100.	100.							
									100.				100.	100.	
RTV 603	7									100.					100.
BROMINEX 126	14	25.	25.	25.	25.	25.	25.	25.	25.	25.					
VIRCOL 82	15										25.	25.	25.	25.	25.
S-182 Catalyst	12	10.									10.				
RTV 655B	7		10.	12.5	15.							10.			
DTD (1)	7					0.25	0.3	0.6	0.25				0.25	0.5	
Catalyst SRC-05	7									0.6					0.6

S......Source (See Table 6-1)
1. Dibutyl tin dilaurate

TABLE 6-56

SILICONE RTV'S WITH FLAME RETARDANT POLYOLS

- FORMULATION PROCESSING -

Formulation No. F-	Cure (1) Time (hrs/min)	Cure (1) Temp. (°F)	Post Cure Time (hrs/min)	Post Cure Temp. (°F)
153	17/35	150	21 days	150
153-C	"	"	--	--
154	17/10	"	21 days	150
154-C	"	"	--	--
156	3/45	"	21 days	150
156-C	"	"	--	--
157	-/45	"	20 days	150
157-C	"	"	--	--
214	21 days	"	--	--
158-1	-/35	"	--	--
158-2	20 days	"	--	--
159	9/40	"	--	--
201	20 days	"	--	--
155	20d/5hr	"	--	--
150	21d/2hr	"	--	--
151	21 days	"	--	--
162	16d/6hr	"	--	--
161	"	"	--	--
152	21 days	"	--	--

TABLE 6-57

SILICONE RTV'S WITH FLAME RETARDANT POLYOLS CHARACTERISTICS

-CURED/POST CURED-

	Formulation												
Properties	156C	157C	214C	158-1C	158-2C	201C	155C	151-1	151-1C	162C	161C	152	152C
Cured	Y	Y	Y	Y	Y	Y	?	Y?	Y	Y	Y	(5)	Y
Tacky	Y(2)	Y	N(2)	?	(2)	?	Y(2)	N	N(2)	Y	S(2)	N(2)	N(2)
Flexible	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y
Memory	Y	Y	Y	Y	Y	Y	Y	?	?	Y	Y	Y	Y
Cohesion	P	X	F	F	F	F	NG	NG	NG	F	F	NG	NG
Tear Resistance	P	P	P	P	P	P	P	NG	NG	F	P	NG	NG
Crack Resistance (1)	X	X	X	X	X	X	X	-	-	X	X	X	X
Crack Propagation (1)	X	X	NG	NG	NG	?	X	-	-	X	X	NG	NG
Porous	N	-	Y	-	-	-	-	N	N	-	-	N	N
Adheres to unclean alum. Foil cup	N	N	N	N	N	N	N	-	Y	F	N	-	N
Cheesy	S	-	N	N	N	-	(6)	(3)	N(4)	-	N	Y	Y

Y.....Yes VS.....Very Slight S.....Slight N.....No
X.....Satisfactory F.....Fair P.....Poor NG.....Not Good

1. 180° bend (face-to-face contact)
2. Greasy
3. Very, very soft
4. Very soft
5. Gelled
6. Gummy

e. Base compound is higher in cost than non-fluorinated analogs (e.g. Sylgard 186: $8.-10./lb., RTV 77-033: $70./lb.). Obviously, this differential in raw material cost is drastically reduced when lower cost additives are incorporated. For example, the inclusion of 150 phr of a $5./lb. additive only reduces the cost of a compound with the former to $6.27-7.12/lb. But formulations with the latter would drop to $32.50/lb.!

Various commercial and experimental fluorosilicone RTV's are shown in Table 6-58. 94-516 is a carbon black loaded commercial grade and was available. We have been led to believe that 77-013 is an unfilled version of 94-516. The lack of data for 77-013 is due to the fact that the vendor, Dow Corning, considers this material as very experimental and has sharply restricted its distribution. 77-024 is alleged to be similar to 94-516, but with a lower carbon black content. After considerable evaluation, the Project was informed (Ref. 6-26) that 77-024 was being discontinued, and that the non-carbon black containing 77-033 was being offered as a substitute.

It is seen from Table 6-58 that the 20-046 is apparently a very high viscosity, (probably thixotropic) high solids compound, whereas the 94-003 is the reverse. They were of some interest either individually or in combination with each other; mutual compatibility being most likely. Unfortunately, the only RTV fluorosilicone condensation reaction systems available are of the acetoxy type; the less corrosive ones were obviously preferred. No action was taken with these two.

6.3.1 Fluorosilicone RTV's with Carbon Black

The carbon black containing two part addition systems were evaluated in this section since 77-013 was not made available to the Project, and until 77-033 could be prepared and shipped to Furane.

6.3.1.1 Phase I: Defective Catalyst - Upon receipt, moldings F-056 and F-108 were prepared from 94-516 and 77-024, respectively. The formulations are described in Table 6-59. 94-516, apparently containing carbon black, silica, and possibly titanium dioxide, was extremely viscous and thixotropic. It readily cured in 2 hours at 150°F (Ref. 6-10) to a flexible non-tacky, non-cheesy elastomer which had only fair cohesion and poor tear resistance, but was able to withstand the 180° bend tests. A 10 gram sample had a pot life of approximately 1.5 hours at 78°F.

After 6 hours 45 minutes at 150°F the F-108 specimens were very soft and tacky. A post cure of 33 hours 5 minutes at 150°F gave no improvement. On the other hand, the pot life of 11 grams was only 4 hours at 78°F. A boric acid formulation of the resin was then processed (F-109) by adding the filler after catalyzation. The viscosity was very high. The moldings still were not cured after 8 hours 22 minutes at 150°F. The possibility of greater inhibition sensitivity was considered. Busan 11-MI

TABLE 6-58

FLUOROSILICONE RTV'S (1)

Properties	Grade 94-516 Sealant (5, 6)	77-013 (5, 6)	77-024 (5, 6)	77-033 Sealant (5, 6)	20-046 Sealant (7, 8)	94-003 Dispersion Ctg. (7, 8)
Viscosity - base (Poise)	6000	800-	475	400-800		
- base + cat. (Poise)	4500	1300				25-75
Rate of extrusion (gm/min) (2)					50	
Transparent	N					
Color	black		grey	white	alum.	buff
Solids - initial (%)						50
- cured (%) (3)	100	100	100	100	95	
Moisture sens. - uncured	Y	Y				
Pot life (hrs/°F)	2/RT			1-2/RT		
Cure, min. temp. (hrs/°F)	7D/77 4/150				7D/77 (9)	7D/77 (9)
Tack free time (hrs)					<2	
Reversion resistant	Y	Y	Y	Y		
Specific gravity	1.37			1.37 (10)	1.35	1.37
Hardness (Shore A)	30			30-40 (10)	30	35
Tensile strength (psi)	550			500-600 (10)	150	
Elongation (%)	400			200-300 (10)	150	
Tear strength (lbs/in)				20 (10)		

1. Dow Corning materials and data.
2. 1/8 in. orifice, 90 psi air.
3. 24 hours at 158°F.
4. Discontinued
5. Two part system.
6. Addition reaction.
7. One part system.
8. Condensation reaction (moisture curing).
9. 50 ± 5% relative humidity.
10. 24 hours at room temperature.

TABLE 6-59

FLUOROSILICONE (WITH CARBON BLACK) RTV: PHASE I

Component	S	056	108	109	114	121	122	123	133	134	126	130	131	132	136	139
		Formulation (phr)														
94-516	12	100.														
77-024	12		100.	100.	100.				100.	100.	100.	100.	100.	100.	100.	100.
77-024 (1)	12					90.	90.	90.								
SYLGARD 182	12								20.							
RTV 615A	7									20.						
RTV 655A	7															
Boric Acid (2)	9			150.			150.	150.								
BUSAN 11-MI	19				100.	100.	50.									
7570 Glass Frit (3)	21							50.								
94-516 Catalyst	12	10.												10.		
77-024 Catalyst (4)	12		10.	10.	10.	10.	10.	10.	10.	10.						
S-182 Catalyst	12								2.		10.					
RTV 615B	7									2.		10.			5.	7.5
RTV 655B	7												10.			

S......Source (See Table 6-1)
1. Fillers removed
2. 100% through 170 mesh
3. 100% through 325 mesh
4. Defective

TABLE 6-59 (cont'd)

FLUOROSILICONE (WITH CARBON BLACK) RTV: PHASE I

Component	S	127	119	135	141	146	147	149	163	164	165	166	167	168	169	172
		Formulations (phr)														
77-024 (1)	12	90.														
77-024	12		100.	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.
RTV 615A	7		100.			100.										
RTV 655A	7			100.												
L-2347	2						10.	10.								
Boric Acid (2)	9				150.	300.		150.	125.					150.	150.	150.
7570 Frit	21								25.							
FC-43	2									20.				20.	20.	
FC-75	2										20.					20.
FC-77	2											40.				
FC-78	2												20.			
77-024 Catalyst (4)	12								10.						10.	
RTV 615B	7	10.	20.		20.	20.	10.	10.		10.	10.	20.	10.	10.		10.
RTV 655B	7			20.												

had been found to somewhat counteract this effect. F-114 was therefore formulated. A cure of 29 hours 30 minutes still resulted in some surface tack. F-121, -122 and -123 were then prepared by first centrifuging out the particulate matter of the 77-024. The blends were then prepared from only the fluid portion. None cured after lengthy cure/post cure cycles. This seemed to eliminate the fillers as the problem source, leaving the polymer and catalyst as potentially defective material.

An attempt was then made to co-cure the 77-024 system with other, but not fluorinated, Sylgard type systems. F-133 and -134 were still tacky after cure cycles which had previously proven successful with these type systems.

When the vendor was contacted, they also suggested the possibility of cure inhibition. The Project did not feel that this was likely since the equipment and facilities, including the ovens and molds, had been used for some time with similar addition reaction silicone RTV systems. Further, the open air "pot life" samples were also not fully cured (i.e. soft and tacky). It was then agreed that other Sylgard type curatives should be considered with the 77-024 while their technical personnel re-examined the entire situation.

F-126, -130, -131 and -132 were formulated with such curatives at the same level of concentration (i.e. 10 phr) as they or 77-024 catalyst would normally be used. All cured satisfactorily, but at different rates. The relative rates were 615B, 655B > 94-516 catalyst >> S-182 catalyst. The RTV 615B was selected for further evaluation with the 77-024 pending resolution of problem by vendor. F-136 contained 5 phr and F-139 had 7.5 phr. The pot lives of approximately 2 gram samples were found to be as follows:

TABLE 6-60

POT LIFE OF 77-024/615B FLUOROSILICONE RTV

Formulation No.	Catalyst Conc. (phr)	Gel Time (min)	Gel Temp. (°F)
130	10.	15	80
139	7.5	28	80
136	5.	38	79

A sample of the 77-024 which had the filler removed was also catalyzed with 10 phr of the 615B (F-127). Ten hours later, the sample was still usable. It was then heated for 36 hours at 150°F before a non-tacky cure was achieved. No further work with the filler-removed 77-024 was carried out.

At this time, the vendor concurred that the 77-024 catalyst was defective and that they would replace it as soon as possible. It was therefore deemed adviseable to continue usage of the 77-024 polymer portion in conjunction with the other catalysts until the new catalyst was received. F-119 and -135 were formulated to determine feasibility of using the regular silicone RTV as a reactive diluent. Both systems cured with no difficulty. However, some separation seemed to occur between the two polymers in F-135. This may be due to the fact that the RTV 655 is a methyl-phenyl type, and the 77-024 is a dimethyl type, or more simply, better blending may have been required. F-141 was identical to F-119 plus boric acid. F-146 was a combination of F-119 and F-141.

F-147 and F-149 contained L-2347, a non-reactive fluorinated diluent, to observe its plasticizing action of the 77-024. (The viscosity of L-2347 is close to that of water.) Its elemental analysis is as follows:

Fluorine	71.6% by weight
Carbon	24.1% " "
Hydrogen	0.3% " "
Oxygen	4.0% " "

The former cured, but F-149, also containing the standard 150 phr boric acid, resulted in a powdery mass. F-163 could not be cured to a non-tacky state and it had very poor cohesion. A series of formulations were attempted with various perfluorinated fluids having different boiling points and viscosities. All appeared to cure to a non-tacky but poor cohesive state. The two highest boiling point ones were selected for preliminary flammability specimens (F-168, -169 and -172).

The detailed cure cycles for all of these formulations are given in Table 25 of Reference 6-11 and in Table 6-61 of this report. Cured characteristics of those not described in the above text are shown in Table 6-62. The preliminary flammability test results for 94-516 and 77-024 are tabulated in Tables 6-63 and 6-64, respectively.

F-056 and F-108 burned with no apparent difficulty, similar to the non-fluorinated versions. (See Table 6-40). F-109 was likewise tested even though not adequately cured. Two specimens tested at 6.2 psia were completely undamaged. A specimen tested at the 60/40 environment (i.e. 60% oxygen + 40% nitrogen = 16.2 psia oxygen) was only very slightly affected. One tested at 16.5 psia oxygen was approximately 10% burnt at one end and 1-2% at the other end. The specimen of F-141 tested at 6.2 psia oxygen was completely charred. There were two obvious differences between F-109 and -141. The latter had half the quantity of boric acid with respect to total polymer weight. Also, the 615 polymer probably has a similar hydrogen content as the resular Sylgards. F-146 could not pass 6.2 psia oxygen. F-163 and F-168 were only slighted degraded at 6.2 psia oxygen and in the 60/40 environment. F-169 had a little greater damage at 60/40, and the specimens were found to have single longitudinal cracks from one end to the other end after the test.

TABLE 6-61

FLUOROSILICONE (WITH CARBON BLACK) RTV: PHASE I

- FORMULATION PROCESSING -

Formulation No. F-	Cure (1) Time (hrs/min)	Cure (1) Temp. (°F)	Post Cure Time (hrs/min)	Post Cure Temp. (°F)
146-1A	2/45	150	--	--
146-1B	"	"	2/15	150
147	4/-	"	--	--
149	(See text)		--	--
163	22/55	150	--	--
164	3/30	"	--	--
165	3/-	"	--	--
166	-/40	"	--	--
167	-/13	"	--	--
168	17/25	"	--	--
169	15/50	"	--	--
172	4/8	"	--	--

1. Time in mold

TABLE 6-62

FLUOROSILICONE (WITH CARBON BLACK) RTV: PHASE I CHARACTERISTICS

-CURED/POST CURED-

Properties	Formulation 146-1A	 146-1B	 168	 169	 172
Cured	Y	Y	Y	N	Y
Tacky	N	N	N	Y	N
Flexible	Y	Y	P	Y	F
Memory	F	F	P	P	F
Cohesion	P	P	P	NG	NG
Tear Resistance	P	P	P	NG	NG
Crack Resistance (1)	P	P	NG	NG	NG
Crack Propagation (1)	P	P	NG	NG	NG
Porous	-	-	N	N	-
Adheres to unclean alum. Foil cup	-	-	-	-	-
Cheesy	S	S	Y	gummy	Y

Y.....Yes VS.....Very Slight S.....Slight N.....No
X.....Satisfactory F.....Fair P.....Poor NG.....Not Good

1. 180° bend (face-to-face contact)

TABLE 6-63

FLUOROSILICONE RTV 94-516
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

		Specimen		
		056-1-1	056-1-1	056-1-2
	O_2 Pressure (psia)	(7)	6.2	6.2
RESULTS	Wire Ignition (1)	T/B	T/B	T
	Wire Failure (1)	T	B	T
	Smoke (2)	---	---	N
	Burn time (sec.) (3) flame-out	2	51	53
	Burn time (sec.) (3) glow-out	2	54	55
	Configuration (4)	Y	N	N
	Residue (%) (5)	100	60	70
	Melted (6)	N	N	N
	Color: exterior	black	white	white
	Color: interior	black	black	black
	Exterior Texture powder (6)	N	Y	Y
	Exterior Texture crusty (6)	N	Y	Y

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. Ambient pressure air

TABLE 6-64
FLUOROSILICONE (WITH CARBON BLACK) RTV: PHASE I
- PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

		Specimen							
		108-1-1	109-1-1	109-1-2	109-2-1	109-2-1	109-1-1	109-1-1	109-2-1
	O_2 Pressure (psia)	6.2	6.2	6.2	6.2	9.72 (60%)	9.72 (60%)	16.5	16.5
RESULTS	Wire Ignition (1)	T/B	T	T/B	B	T/B	T/B	T/B	B
	Wire Failure (1)	T	T	T/B	B	T	B	T/B	B
	Smoke (2)	S	N	N	N	N	N	N	S
	Burn time (sec.) (3) flame-out	42	1.5	5.5	4	5	5	18	38
	glow-out	45	1.5	5.5	4	5	5	18	53
	Configuration (4)	N	Y	Y	Y	Y	Y	Y	Y
	Residue (%) (5)	60	100	100	>99	>99	100	100	85
	Melted (6)	N	N	N	N	N	N	N	N
	Color exterior	black	gray	gray	gray	gray	gray	gray	white
	interior	white	"	"	"	"	"	"	---
	Exterior Texture powder (6)	Y	N	N	N	N	N	N	N
	crusty (6)	Y	N	N	N	N	N	N	Y

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No

TABLE 6-64 (cont'd)
FLUOROSILICONE (WITH CARBON BLACK) RTV: PHASE I
PRELIMINARY FLAMMABILITY
– TEST CONDITIONS/RESULTS –

		Specimen							
		109-2-2	141-1B-1	146-1B-1	163-1-1	163-1-1	163-1-1	168-1-1	168-1-1
	O_2 Pressure (psia)	16.5	6.2	6.2	6.2	9.72 (60%)	16.5	6.2	9.72 (60%)
RESULTS	Wire Ignition (1)	T/B	T/B	T/B	T/B	T/B	T/B	T/B	T/B
	Wire Failure (1)	B	T	B	T	B	B	T	T/B
	Smoke (3)	P	S	S	N	N	P	N	N
	Burn time (sec.) (3) flame-out	57	47	50	11	12	51	5.5	5
	Burn time (sec.) (3) glow-out	57	48	55	11	12	51	5.5	5
	Configuration (4)	Y	Y	Y	Y	Y	Y	Y	Y
	Residue (%) (5)	85	90	80	>99	>99	85	>99	>99
	Melted (6)	N	N	N	N	N	N	N	N
	Color exterior	white	white	white	gray	gray	white	gray	gray
	Color interior	---	black	---	---	"	---	"	"
	Exterior Texture powdery (6)	N	N	N	N	N	N	N	N
	Exterior Texture crusty (6)	Y	Y	Y	N	N	Y	N	N

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No

TABLE 6-64 (cont'd)
FLUOROSILICONE (WITH CARBON BLACK) RTV: PHASE I
PRELIMINARY FLAMMABILITY
– TEST CONDITIONS/RESULTS –

		Specimen						
		168-1-1	169-1-1	169-1-1	169-1-1	172-1-1	172-1-1	172-1-1
	O_2 Pressure (psia)	16.5	5.2	9.72 (60%)	16.5	6.2	9.72 (60%)	16.5
RESULTS	Wire Ignition (1)	T/B	T/B	T/B	T/B	T/B	---	T/B
	Wire Failure (1)	B	T	B	T	T/B	---	B
	Smoke (2)	P	N	N	P	N	S	S
	Burn time (sec.) (3) flame-out	49	5.5	15	54	4	80	45
	glow-out	49	5.5	15	54	4	80	51
	Configuration (4)	Y	Y	Y	Y	Y	Y	Y
	Residue (%) (5)	80	>99	>99	85	>99	85	85
	Melted (6)	N	N	N	N	N	N	N
	Color: exterior	white	gray	gray	white	gray	white	white
	: interior	black	"	"	---	"	---	---
	Exterior Texture powdery (6)	N	N	N	N	N	N	N
	crusty (6)	Y	N	N	Y	N	Y	Y

1. T.....Top B.....Bottom
2. N.....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No

(Probably due to use of the defective 77-024 catalyst.) When the FC-43 of F-168 was replaced by FC-75 (F-172), the usage limit was unquestionably 6.2 psia oxygen.

6.3.1.2 Phase II: Acceptable Catalyst - Upon receipt of the "proper" 77-024 catalyst, reformulation (see Table 6-65) and testing was instituted. Processing data and cured characteristics are reported in Tables 6-66 and 6-67, respectively.

The curing ability of the new catalyst was checked on two separate occasions (F-171 and F-210) by two different members of the Furane laboratory staff. The cure schedule also differed in each case. There were no cure difficulties observed in either test.

It is seen from Table 6-67 that reducing the recommended catalyst concentration (F-170) by 30% (F-174) caused observable deterioration in mechanical properties when 150 phr boric acid was added to the base material. Reducing the boric acid to 125 phr (F-175) improved these properties.

A phosphonitrilic chloride resin, PH 990, was tried as a flame retardant. It was impossible to blend the mixrure until the quantity of PH 990 was reduced to 75 phr. Internal inhibition seemed to prevent a good cure of F-181. Cyclic phosphonitrilic chloride resulted in a better cure (F-180). When a smaller particle size was used (F-184), the mechanical properties improved. However, stored preliminary flammability specimens showed corrosion of the copper wires.

Antimony oxychloride was then added to the 77-024 (F-209-1). The 2.60 gm. of blend was cured in an aluminum dish with no tack. Preliminary flammability and strip specimens (F-209-2) were then prepared. The strip appeared to be almost as well cured (very slight tack) as F-209-1. However, the flammability specimens were almost fluid at the bottom half of each specimen while the top half was similar to the strips. Some form of inhibition had evidenraly occurred. An additional post cure was then attempted, but it made no apparent difference.

A series of formulations was then prepared with various inorganic compounds containing boron and/or fluorine. F-188 was hygroscopic and did not cure. F-186, -183, and -189 also did not cure. F-187 readily cured, but the flammability and strip test specimens tended to fall apart.

As seen in Table 6-68, the only formulations of this section which had any significant flammability resistance were those which contained boric acid. At a level of 150 phr, one out of two specimens survived at 16.5 psia oxygen. At a level of 125 phr, the limit was closer to the 60/40 environment.

6.3.1.3 Diluents - At this point, it was deemed advisable to try reducing the viscosities of the 77-024 with various perfluorinated and halogenated fluids. These were selected from the Krytox and Freon product lines of DuPont because of their availability. 3M's perfluorinated fluids had significantly lower boiling points. It

TABLE 6-65

FLUOROSILICONE (WITH CARBON BLACK) RTV WITH ADDITIVES: PHASE II

Component	S	Formulations (phr) 171	210	170	174	175	175X	181	180	184	209
77-024	12	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.
Boric Acid (1)	9			150.	150.	125.	125.				
PH 990 (2)	31							75.			
Cyclic $PNCl_2$	31								150.		
Cyclic $PNCl_2$ (1)	31									150.	
SbOCl	30										150.
77-024 Catalyst	12	10.	10.	10.	7.	7.	10.	10.	10.	10.	10.

S......Source (See Table 6-1)
1. 100% through 170 mesh
2. Phosphonitrilic resin prepolymer

TABLE 6-65 (cont'd)

FLUOROSILICONE (WITH CARBON BLACK) RTV WITH ADDITIVES: PHASE II

		Formulations (phr)					
Component	S	185	188	186	187	183	189
77-024	12	100.	100.	100.	100.	100.	100.
Ca_3B_6	29	150.					
CaBF	29		150.				
$CaSiF_6 \cdot 2H_2O$	29			150.			
$MgTiF_6$	29				150.		
SbF_3	29					150.	
$Ni(BF_4)_2.6\ H_2O$	29						150.
77-024 Catalyst	12	10.	10.	10.	10.	10.	10.

S.....Source (see Table 6-1)

TABLE 6-66

FLUOROSILICONE (WITH CARBON BLACK) RTV WITH ADDITIVES: PHASE II

- FORMULATION PROCESSING -

Formulation No. F-	Cure (1) Time (hrs/min)	Cure (1) Temp. (°F)	Post Cure Time (hrs/min)	Post Cure Temp. (°F)
171	-/19	150	--	--
210	4/20	"	--	--
170	4/-	"	--	--
174	17/20	"	--	--
175	15/45	"	--	--
175X	7/-	"	--	--
181	44/20	"	--	--
180	11/35	"	--	--
184	15/45	"	--	--
209-1	42/20	"	--	--
209-2	8/30	"	9/30	150
185	1/20	"	3/50	"
188	4 mo.	"	--	--
186	24/-	"	--	--
187-1	2/20	"	--	--
187-2	87/45	"	--	--
183	24/-	"	--	--
189	87/08	"	--	--

1. Time in mold

TABLE 6-67

FLUOROSILICONE (WITH CARBON BLACK) RTV WITH ADDITIVES: PHASE II CHARACTERISTICS

-CURED/POST CURED-

	Formulation									
Properties	171	210	170	174	175	180	184	209-2	185	187-2
Cured	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y
Tacky	N	N	N	N	N	N	VS	VS	N	N
Flexible	Y	Y	S	Y	Y	Y	Y	Y	Y	Y
Memory	Y	Y	S	P	P	F	F	Y	Y	P
Cohesion	F	X	P	P	P	P	F	F	P	P
Tear Resistance	F	P	P	NG	P	P	P	F	P	P
Crack Resistance (1)	X	X	P	NG	P	P	X	X	P	P
Crack Propagation (1)	X	P	P	NG	P	P	X	F	P	P
Porous	-	Y	-	N	N	Y	-	-	-	Y
Adheres to unclean alum. Foil cup	S	N(2)	N	P	N	-	-	-	-	-
Cheesy	N	N	S	Y	S	N	N	N	N	Y

Y.....Yes VS.....Very Slight S.....Slight N.....No
X.....Satisfactory F.....Fair P.....Poor NG.....Not Good

1. 180° bend (face-to-face contact)
2. Room temperature cure.

TABLE 6-68
FLUOROSILICONE (WITH CARBON BLACK) RTV WITH ADDITIVES: PHASE II PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

		Specimen										
		210-1-1	210-1-1	210-1-2	210-1-1	170-1-1	170-1-1	170-1-1	170-1-2	174-1-1	174-1-1	174-1-1
	O_2 Pressure (psia)	(7)	6.2	6.2	9.72 (60%)	6.2	9.72 (60%)	16.5	16.5	6.2	9.72 (60%)	16.5
RESULTS	Wire Ignition (1)	T/B	T/B	T/B	T/B	T/B	T/B	T/B	T/B	T/B	T/B	T/B
	Wire Failure (1)	T	---	T/B	---	B	B	B	T/B	T	B	B
	Smoke (2)	S	N	N	N	N	N	S	N	N	N	S
	Burn time (sec.) (3) flame-out	121	41	48	50	5.2	5	40	10	8	6	40
	Burn time (sec.) (3) glow-out	121	41	48	55	5.2	5	53	10	8	6	47
	Configuration (4)	Y	N	N	N	Y	Y	Y	Y	Y	Y	Y
	Residue (%) (5)	90	90	0	80	>99	>99	85	>99	>99	>99	90
	Melted (6)	N	N	N	N	N	N	N	N	N	N	N
	Color exterior	gray	black	gray	gray	gray	gray	white	gray	gray	gray	white
	Color interior	"	---	---	---	"	"	gray	---	"	"	---
	Exterior Texture powdery (6)	Y	Y	Y	Y	N	N	N	N	N	N	N
	Exterior Texture crusty (5)	Y	Y	Y	Y	N	N	Y	Y	N	N	Y

1. T.....Top B.....Bottom
2. N ....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. Ambient pressure air

TABLE 6-68 (cont'd)
FLUOROSILICONE (WITH CARBON BLACK) RTV WITH ADDITIVES: PHASE II
PRELIMINARY FLAMMABILITY
TEST CONDITIONS/RESULTS

		Specimen								
		175-1-1	175-1-1	175-1-1	181-1-1	180-1-1	184-1-1	185-1-1	186-1-1	187-2-1
	O_2 Pressure (psia)	6.2	9.72 (60%)	16.5	6.2	6.2	6.2	6.2	6.2	6.2
RESULTS	Wire Ignition (1)	T/B	T/B	T/B	T/B	T/B	T/B	T	T/B	T/B
	Wire Failure (1)	T/B	T/B	B	T	T/B	T	T	---	B
	Smoke (2)	N	N	P	P	P	P	S	N	N
	Burn time (sec.) (3) flame-out	5	15	41	55	74	36	70	0	56
	Burn time (sec.) (3) glow-out	5	15	41	60	74	36	110	0	56
	Configuration (4)	Y	Y	Y	N	Y	Y	Y	N	N
	Residue (%) (5)	>99	>99	80	0	85	75	80	0	0
	Melted (6)	N	N	N	Y	N	N	N	Y	N
	Color exterior	gray	gray	white	black	white	wht/blk	black	gray	gray
	Color interior	"	"	---	"	---	---	---	"	"
	Exterior Texture powdery (6)	N	N	N	Y	Y	N	N	N	Y
	Exterior Texture crusty (6)	N	N	Y	Y	Y	Y	Y	N	N

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No

is seen from Table 6-69 thct the diluents were generally added at a 20 phr level. F-192 cured (see Table 6-70) to a flexible non-tacky or -greasy state (see Table 6-71). F-200, containing 150 phr of the same fluid, K-143 AC, cured to a flexible structure which was very oily and porous. In fact, fluid separation was noted. F-193, -194, -195 also cured to flexible structures. Of these, only F-195 had any noticeable greasy "feel." It also had the lowest boiling range (i.e. 289-365°F at 0.8 mm Hg.). It was therefore deleted from further consideration herein.

The remaining formulations in this section also contained the standard 150 phr boric acid. F-196, -198, and -199 varied in quantity of K-143 AC. Cured F-199 was quite oily. The Freon ones had even worse mechanical properties, but were not oily.

The flammability results with F-196, -198, -197, -177, or -178 did not appear to differ from that obtained with no diluenr (see F-170 of Table 6-68). F-1.99, containing 100 phr K-143 AC, showed at 16.5 psia oxygen, no effect with one specimen, and oily slight degradation with a second one (see Table 6-72).

6.3.1.4 Carriers Loaded with Flame Retardant Liquids - It was determined from the proceeding section that adding the diluents at a 20 phr level did not significantly reduce the viscosity enough, but did deteriorate the mechanical properties, and generally acted as an internal release agent (preventing adhesion to the wires). But the apparent improvement in flame retardation at a 100 phr level could also not be ignored. In order to achieve the latter benefits without the aforementioned penalties, some of the diluents were combined with molecular sieves (see Table 6-73) prior to addition to the polymer (see Table 6-74).

77-024 cured (see Table 6-75) normally with only 25 phr of the molecular sieves (F-191). However, 150 phr resulted in a dry powdery condition (F-190). F-232 had a slightly oily surface, but F-233 had a noticeable separation of liquid from the cured polymer. It was not possible ti incorporate 100 phr of F-227 into the polymer due to its effect upon the viscosity. It was then arbitrarily reduced to 50 phr. There was then no blending problem (F-229). The difficulty was attributed to the very volatile nature of the Freon 114-B2 (i.e. leaving only molecular sieves in F-227). F-228 also cured, but Freon E-9 separated out.

A comparison of water loaded sieves with a water treated glass frit was made with F-224 and F-182, respectively. The latter evidenced somewhat better mechanical properties (see Table 6-76) than the sieve ones, even with a 150 phr loading. However, it readily burned in 6.2 psia oxygen (see Table 6-77).

Boric acid was added to the Krytox 143 AC loaded sieve formulations F-232 and F-233. It was also used with F-236 which has a lower concentration of the Krytox. The resultant flammability specimens survived the 60/40 environment but F-234 also withstood 16.5 psia oxygen. F-235 almost started to burn and had well defined surface

TABLE 6-69

FLUOROSILICONE (WITH CARBON BLACK) RTV WITH FLAME RETARDANT AND/OR NON-REACTIVE DILUENTS

		Formulations (phr)											
Component	S	192	200	193	194	195	196	198	199	197	176	177	178
77-024	12	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.
Boric Acid (1)	9						150.	150.	150.	150.	150.	150.	150.
Krytox 143 AC	1	20.	150.				20.	40.	100.				
Krytox 143 AD	1			20.						20.			
Krytox 143 AX	1				20.								
Krytox 143 AZ	1					20.							
Freon 114-B2	1										20.		
Freon TF	1											20.	
Freon E-9	1												20.
77-024 Catalyst	12	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.

S......Source (See Table 6-1)
1. 100% through 170 mesh

TABLE 6-70

FLUOROSILICONE (WITH CARBON BLACK) RTV WITH FLAME RETARDANT AND/OR NON-REACTIVE DILUENTS

- FORMULATION PROCESSING -

Formulation No. F-	Cure (1) Time (hrs/min)	Cure (1) Temp. (°F)	Post Cure Time (hrs/min)	Post Cure Temp. (°F)
192	-/61	150	--	--
200	16/32	"	--	--
193	-/36	"	--	--
194	-/34	"	--	--
195	-/41	"	--	--
196	18/10	"	--	--
198	1/-	"	17/14	150
199	17/55	"	--	--
197	16/50	"	--	--
176	46/32	"	--	--
177	45/45	"	--	--
178	44/-	"	--	--

1. Time in mold

TABLE 6-71

FLUOROSILICONE (WITH CARBON BLACK) RTV WITH FLAME RETARDANT AND/OR NON-REACTIVE DILUENTS CHARACTERISTICS

-CURED/POST CURED-

	Formulation						
Properties	196	198	199	197	176	177	178
Cured	Y	Y	Y	Y	Y	Y	Y
Tacky	N	N(2)	N(2)	N	N	N	N
Flexible	Y	Y	Y	Y	F	F	F
Memory	Y	F	Y	Y	F	F	F
Cohesion	P	P	P	P	P	P	P
Tear Resistance	P	P	P	P	P	P	NG
Crack Resistance (1)	P	F	P	P	NG	NG	NG
Crack Propagation (1)	P	P	P	P	NG	NG	NG
Porous	Y	Y	Y	N	-	-	S
Adheres to unclean alum. Foil cup	P	-	P	-	N	N	N
Cheesy	S	N	N	Y	S	S	S

Y.....Yes VS.....Very Slight S.....Slight N.....No
X.....Satisfactory F.....Fair P.....Poor NG.....Not Good

1. 180° bend (face-to-face contact)
2. Slight oily film on surface of strip mold

TABLE 6-72
FLUOROSILICONE (WITH CARBON BLACK) RTV WITH FLAME RETARDANT AND/OR NON-REACTIVE DILUENTS
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

		Specimen							
		196-1-1	196-1-1	196-1-1	198-1-1	198-1-1	198-1-1	199-1-1	199-1-1
	O_2 Pressure (psia)	6.2	9.72 (60%)	16.5	6.2	9.72 (60%)	16.5	6.2	9.72 (60%)
RESULTS	Wire Ignition (1)	T/B	T/B	T/B	T/B	T/B	T/B	T	T/B
	Wire Failure (1)	T	B	B	T	B	B	T	T/B
	Smoke (2)	N	N	P	N	N	P	N	N
	Burn time (sec.) (3) flame-out	4	5	64	4	5	52	1	5
	Burn time (sec.) (3) glow-out	4	5	64	4	5	52	1	5
	Configuration (4)	Y	Y	Y	Y	Y	Y	Y	Y
	Residue (%) (5)	>99	>99	90	>99	>99	70	>99	>99
	Melted (6)	N	N	N	N	N	N	N	N
	Color exterior	gray	gray	white	gray	gray	white	gray	gray
	Color interior	---	---	---	″	″	---	″	″
	Exterior Texture powdery (6)	N	N	N	N	N	N	N	N
	Exterior Texture crusty (6)	N	N	Y	N	N	Y	N	N

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No

TABLE 6-72 (cont'd)
FLUOROSILICONE (WITH CARBON BLACK) RTV WITH FLAME RETARDANT AND/OR NON-REACTIVE DILUENTS
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

		Specimen							
		199-1-1	199-1-2	197-1-1	197-1-1	197-1-1	197-1-2	176-1-1	176-1-1
	O_2 Pressure (psia)	16.5	16.5	6.2	9.75 (60%)	16.5	16.5	6.2	9.72 (60%)
RESULTS	Wire Ignition (1)	T/B	T/B	T/B	T/B	T/B	T/B	T/B	T/B
	Wire Failure (1)	T	T/B	B	B	T/B	B	T	B
	Smoke (2)	N	N	N	N	N	P	N	N
	Burn time (sec.) (3) flame-out	9	22	6	4	5	45	3	6
	Burn time (sec.) (3) glow-out	9	22	6	4	5	56	3	6
	Configuration (4)	Y	Y	Y	Y	Y	Y	Y	Y
	Residue (%) (5)	>99	95	>99	>99	>99	80	>99	>99
	Melted (6)	N	N	N	N	N	N	N	N
	Color exterior	gray	gray/wht	gray	gray	gray	white	gray	gray
	Color interior	"	---	---	---	---	gray	"	"
	Exterior Texture powdery (6)	N	N	N	N	N	N	N	N
	Exterior Texture crusty (6)	N	Y	N	N	N	Y	N	N

1. T.....Top B.....Bottom
2. N.....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No

TABLE 6-72 (cont'd)
FLUOROSILICONE (WITH CARBON BLACK) RTV WITH FLAME RETARDANT AND/OR NON-REACTIVE DILUENTS
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

		Specimen							
		176-1-1	177-1-1	177-1-1	177-1-1	177-1-2	178-1-1	178-1-1	178-1-1
	O_2 Pressure (psia)	16.5	6.2	9.72 (60%)	16.5	16.5	6.2	9.72 (60%)	16.5
RESULTS	Wire Ignition (1)	B	T/B	T/B	T/B	T/B	T	T/B	T/B
	Wire Failure (1)	B	T	T/B	T/B	B	T	B	T/B
	Smoke (2)	S	N	N	N	S	N	N	N
	Burn time (sec.) (3) flame-out	43	3	5	12	56	1.5	22	35
	Burn time (sec.) (3) glow-out	43	3	5	12	56	1.5	22	35
	Configuration (4)	Y	Y	Y	Y	Y	Y	Y	Y
	Residue (%) (5)	85	>99	>99	>99	85	>99	>99	95
	Melted (6)	N	N	N	N	N	N	N	N
	Color exterior	white	gray	gray	gray	white	gray	gray	gray/wht
	Color interior	---	"	"	---	---	"	"	---
	Exterior Texture powdery (6)	N	N	N	N	N	N	N	N
	Exterior Texture crusty (6)	Y	N	N	N	Y	N	N	Y

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No

TABLE 6-73

FLAME RETARDANT LIQUIDS ON NONFLAMMABLE CARRIERS

Component	S	Formulations (phr)						
		236	231	227	226	225	179	231X
Mol. Sieves 13X	8	100.	100.	100.	100.	100.		100.
Glass Frit 7570	21						100.	
Krytox 143 AC	1	200.	298.					300.
Freon 114-B2	1			~29.				
Freon E-9	1				~372.			
Distilled Water (1)	32					25.	excess	

S......Source (See Table 6-1)
1. Ozone-free

TABLE 6-74

FLUOROSILICONE (WITH CARBON BLACK) RTV WITH FLAME RETARDANT LIQUIDS/CARRIERS

Component	S	Formulations (phr)											
		191	190	232	233	229	228	224	182	230	234	235	230X
77-024	12	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.
Boric Acid (1)	9									150.	150.	150.	150.
Mol. Sieves 13X	8	25.	150.										
231 (2)	13			100.	150.					100.	150.		
227 (2)	13					50.							
226 (2)	13						100.						
225 (2)	13							50.					
179 (2)	13								150.				
236 (2)	13											150.	
231X (2)	13												100.
77-024 Catalyst	12	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.

S......Source (See Table 6-1)
1. 100% through 170 mesh
2. See Table 6-73

TABLE 6-75

FLUOROSILICONE (WITH CARBON BLACK) RTV WITH FLAME RETARDANT LIQUIDS/CARRIERS

- FORMULATION PROCESSING -

Formulation No. F-	Cure (1) Time (hrs/min)	Temp. (°F)	Post Cure Time (hrs/min)	Temp. (°F)
191	68/45	150	--	--
190	71/15	"	--	--
232	1/-	"	--	--
233	16/20	"	--	--
229	-/21	"	--	--
228	-/21	"	--	--
224	1/17	"	--	--
182	15/50	"	--	--
230	65/03	"	--	--
234	15/45	"	--	--
235	4/-	"	--	--
230X	9/-	"	--	--

---

1. Time in mold

TABLE 6-76

FLUOROSILICONE (WITH CARBON BLACK) RTV WITH FLAME RETARDANT LIQUIDS/CARRIERS CHARACTERISTICS

-CURED/POST CURED-

Properties	Formulation 232	233	229	228	224	182	230	234	235
Cured	Y	Y	Y	Y	Y	Y	Y	Y	Y
Tacky	(3)	(3)	N	(3)	N	N	(3)	(3)	(3)
Flexible	Y	Y	Y	Y	Y	Y	Y	Y	Y
Memory	Y	Y	Y	Y	Y	Y	F	Y	Y
Cohesion	F	F	F	F	F	F	NG	NG	NG
Tear Resistance	F	F	F	F	F	X	NG	NG	NG
Crack Resistance (1)	-	P	-	-	-	X	NG	NG	NG
Crack Propagation (1)	-	P	-	-	-	X	NG	NG	NG
Porous	Y	Y	Y	-	Y	Y	Y	N	Y
Adheres to unclean alum. Foil cup	F	-	P	F	F	-	P(2)	NG	-
Cheesy	N	N	N	N	N	N	S	Y	Y

Y.....Yes
VS.....Very Slight
S.....Slight
N.....No
X.....Satisfactory
F.....Fair
P.....Poor
NG.....Not Good

1. 180° bend (face-to-face contact)
2. Room Temperature Cure
3. Oily

TABLE 6-77
FLUOROSILICONE (WITH CARBON BLACK) RTV WITH FLAME RETARDANT LIQUIDS/CARRIERS
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

		Specimen										
		182-1-1	230-1-1	230-1-1	230-1-1	234-1	234-1	234-1	234-1	235-1-1	235-1-1	235-1-1
	O_2 Pressure (psia)	6.2	6.2	9.72 (60%)	16.5	6.2	9.72 (60%)	16.5	16.5	6.2	9.72 (60%)	16.5
RESULTS	Wire Ignition (1)	T/B	T/B	T/B	T/B	T/B	T	T	T/B	T/B	T	T/B
	Wire Failure (1)	T	T	T	T	T/B	T	T	T	T	T	B
	Smoke (2)	S	N	N	S	N	N	N	N	N	N	N
	Burn time (sec.) (3) flame-out	40	3	4	55	4	0	0	0	4	2	9
	Burn time (sec.) (3) glow-out	47	3	4	78	4	0	0	0	4	2	9
	Configuration (4)	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y	Y
	Residue (%) (5)	80	>99	>99	90	>99	>99	>99	>99	>99	>99	>99
	Melted (6)	N	N	N	N	N	N	N	N	N	N	N
	Color exterior	white	gray	gray	wht/blk	gray	gray	gray	gray	gray	gray	gray
	Color interior	---	---	---	---	---	---	---	---	---	---	---
	Exterior Texture powdery (6)	N	N	N	N	N	N	N	N	N	N	N
	Exterior Texture crusty (6)	Y	N	N	Y	N	N	N	N	N	N	N

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No

cracks in the molded specimens. It is seen in Table 6-76 that all of these boric acid formulations had very poor mechanical properties.

6.3.2 Fluorosilicone RTV's With No Carbon Black

When the Project received 77-033, the "unfilled" version of 77-024, it appeared to have a white filler which was believed to be silica.

6.3.2.1 Inorganic Flame Retardants - Initial formulation (see Table 6-78) was with the unmodified 77-033 to establish the usual base-line characteristics (F-241 and F-268). F-242 contained the standard 150 phr of boric acid, and F-378 had 125 phr. A series of formulations was then prepared to evaluate a) usefulness of a soda lime (no lead) optical crown glass as a fire retardant, b) effect of particle size on viscosity, and c) effect of particle size and shape on flexibility. Spherical glass beads (see Table 6-79) were used in this phase.

TABLE 6-79

GLASS SHOT

Grade	Micron Range	Softening Point, (°F) Approx.
MSP	500-250	1200
MSH	250-149	"
MSM	149-74	"
MSL	74-44	"
1.65 RI	590-297	1300-1400
1.91 RI (812)	177-125	1400

No discernible advantage in any of the evaluated areas were evidenced by the glass shot. In fact, a small amount added to a boric acid system (F-263) seemed to increase the burning.

No cure (see Table 6-80) was obtained with ammonium carbonate (F-264) or ammonium silicafluoride (F-267). The ferric phosphate, $FePO_4 \cdot 4H_2O$, used for F-441 apparently had too small a particle size to maintain any fluidity in the blend. The remaining fluorinated compounds shown in Table 6-78 readily cured (see Table 6-81).

Boric acid contains 43.70% water (Ref. 6-27) and the XPI-158 (i.e. hydrated zinc borate) used in previous formulations for this Project is reported (Ref. 6-28) to have 13.80-15.21%. Another zinc borate, ZB-237, containing the same proportion of zinc oxide to boric acid, but having 25.35% water, (Ref. 6-28) was obtained and formulated with 77-033 (F-384). Due to its fine particle size, only 90.4 phr could be added. The mechanical properties were quite poor and the 6.2 psia oxygen specimen was completely burnt (see Table 6-82).

TABLE 6-78

FLUOROSILICONE RTV'S WITH INORGANIC ADDITIVES

		Formulations (phr)										
Component	S	241	268	242	257	258	259	260	261	262	263	378
77-033	12	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.
Boric Acid (1)	9			150.							125.	125.
Glass Shot-MSP (2)	33				150.							
Glass Shot-MSH (2)	33					150.						
Glass Shot-MSM (2)	33						150.					
Glass Shot-MSL (2)	33							150.			25.	
Glass Shot-1.65 RI (2)	33								150.			
Glass Shot-812: 1.91 RI (2)	33									150.		
77-033 Catalyst	12	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.

S......Source (See Table 6-1)
1. 100% through 170 mesh
2. See Table

TABLE 6-78 (cont'd)

FLUOROSILICONE RTV'S WITH INORGANIC ADDITIVES

Component	S	Formulations (phr)								
		264	265	266	267	355	356	384	389	441
77-033	12	100.	100.	100.	100.	100.	100.	100.	100.	100.
Ammonium carbonate (3)	35	150.								
Aluminum sodium silicofluoride	36		150.							
Ammonium fluoborate	37			150.						
Ammonium silicafluoride	38				150.					
Potassium fluoborate	10					150.				
Potassium fluophosphate	10						150.			
ZB-237 (4)	39							90.4		
PHOS-CHECK P/30R (5)	40								150.	
Ferric phosphate	--									150.
77-033 Catalyst	12	10.	10.	10.	10.	10.	10.	10.	10.	10.

3. U.S.P. grade
4. Zinc borate
5. Ammonium polyphosphate

TABLE 6-80

FLUOROSILICONE RTV'S WITH INORGANIC ADDITIVES

- FORMULATION PROCESSING -

Formulation No. F-	Cure (1) Time (hrs/min)	Cure (1) Temp. (°F)	Post Cure Time (hrs/min)	Post Cure Temp. (°F)
241	18/27	150	--	--
268	15/50	"	--	--
242	17/45	"	--	--
257	22/29	"	--	--
258	22/15	"	--	--
259	21/55	"	--	--
260-1	21/30	"	--	--
260-2	4/20	"	--	--
261	21/01	"	--	--
262	20/25	"	--	--
263	15/52	"	--	--
378	2/-	"	22/-	150
264	16/05	"	--	--
265	2/30	"	17/10	150
266	1/35	"	"	"
267	16/25	"	--	--
355	2/-	"	--	--
356	2/30	"	--	--
384	19/-	"	--	--
389	1/40	"	22/-	150
441	(See text)		--	--

1. Time in mold

TABLE 6-81

FLUOROSILICONE RTV'S WITH INORGANIC ADDITIVES CHARACTERISTICS

-CURED/POST CURED-

Properties	Formulation 241	 268	 242	 257	 258	 259	 260-1	 260-2	 261
Cured	Y	Y	Y	Y	Y	Y	Y	Y	Y
Tacky	Y	S	N	N	N	N	N	N	N
Flexible	Y	Y	Y	Y	Y	Y	Y	Y	Y
Memory	Y	Y	F	-	-	-	-	Y	-
Cohesion	F	X	P	-	-	-	-	F	-
Tear Resistance	F	X	P	-	-	-	-	F	-
Crack Resistance (1)	F	X	P	-	-	-	-	F	-
Crack Propagation (1)	F	X	P	-	-	-	-	F	-
Porous	Y	Y	N	-	-	-	-	N	-
Adheres to unclean alum. Foil cup	F	F	F	X	X	X	X	F	F
Cheesy	N	N	N	N	N	N	N	N	N

Y.....Yes VS.....Very Slight S.....Slight N.....No
X.....Satisfactory F.....Fair P.....Poor NG.....Not Good

1. 180° bend (face-to-face contact)

TABLE 6-81 (cont'd)
FLUOROSILICONE RTV'S WITH INORGANIC ADDITIVES
CHARACTERISTICS

-CURED/POST CURED-

	Formulation							
Properties	262	263	265	266	355	356	384	389
Cured	Y	Y	Y	Y	Y	Y	Y	Y
Tacky	N	N	S	N	VS	VS	N	N
Flexible	Y	Y	Y	Y	Y	Y	Y	Y
Memory	-	Y	Y	Y	Y	Y	Y	Y
Cohesion	-	P	P	F	P	F	P	NG
Tear Resistance	-	P	P	F	P	P	P	NG
Crack Resistance (1)	-	P	P	X	X	X	NG	NG
Crack Propagation (1)	-	P	P	X	X	X	NG	NG
Porous	-	N	N	N	N	N	N	-
Adheres to unclean alum. Foil Cup	F	P	F	F	-	-	-	-
Cheesy	N	S	N	N	N	-	N	S

Y.....Yes	VS.....Very Slight	S.....Slight	N.....No
X.....Satisfactory	F.....Fair	P.....Poor	NG.....Not Good

1. 180° bend (face-to-face contact)

TABLE 6-82
FLUOROSILICONE RTV'S WITH INORGANIC ADDITIVES
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

		Specimen						
		241-1-1	241-1	268-1-1	268-1-2	242-1-1	242-1-1	242-1-1
	O$_2$ Pressure (psia)	(7)	6.2	(7)	(7)	6.2	9.72 (60%)	16.5
RESULTS	Wire Ignition (1)	T	T/B	T/B	T/B	T	T	T/B
	Wire Failure (1)	T	B	B	T/B	T	T	T/B
	Smoke (2)	N	N	S	N	N	N	N
	Burn time (sec.) (3) flame-out	0	34	101	80	1	3	15
	Burn time (sec.) (3) glow-out	0	34	101	80	1	3	21
	Configuration (4)	Y	N	Y	Y	Y	Y	Y
	Residue (%) (5)	100	75	90	90	99	99	95
	Melted (6)	N	N	N	N	N	N	N
	Color exterior	white	wht/gray	brn/wht	gray	white	white	wht/gray
	Color interior	"	---	---	---	---	---	---
	Exterior Texture powdery (6)	N	N	Y	Y	N	N	N
	Exterior Texture crusty (6)	N	Y	Y	Y	N	N	Y

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. Ambient pressure air

TABLE 6-82 (cont'd)
FLUOROSILICONE RTV'S WITH INORGANIC ADDITIVES
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

		Specimen					
		242-1-2	260-2-1	260-2-2	260-2-1	263-1-1	263-1-1
	O$_2$ Pressure (psia)	16.5	(7)	(7)	6.2	6.2	9.72 (60%)
R	Wire Ignition (1)	T/B	T/B	T/B	T/B	T/B	T/B
E	Wire Failure (1)	B	B	T	T	T	B
S	Smoke (2)	P	N	N	N	S	P
U	Burn time (sec.) (3) | flame-out	48	0	3	37	56	60
L	Burn time (sec.) (3) | glow-out	51	0	31	37	56	60
	Configuration (4)	Y	Y	Y	Y	Y	Y
T	Residue (%) (5)	85	99	90	99	85	75
S	Melted (6)	N	N	N	N	N	N
	Color: exterior	wht/blk	white	wht/blk	white	wht/blk	wht/blk
	Color: interior	---	---	---	---	---	---
	Exterior Texture | powdery (6)	N	N	N	N	N	N
	Exterior Texture | crusty (6)	Y	N	N	N	Y	Y

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. Ambient pressure air

TABLE 6-82 (cont'd)
FLUOROSILICONE RTV'S WITH INORGANIC ADDITIVES
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

			Specimen						
			265-1B-1	266-1B-1	266-1B-1	266-1B-2	266-1B-1	355-1-1	355-1-1
	O_2 Pressure (psia)		6.2	6.2	9.72 (60%)	9.72 (60%)	16.5	6.2	9.72 (60%)
RESULTS	Wire Ignition (1)		T/B	T/B	T/B	B	T/B	---	T/B
	Wire Failure (1)		T/B	B	T	B	T/B	B	B
	Smoke (2)		N	N	N	N	P	N	S
	Burn time (sec.) (3)	flame-out	51	10	4	5	~ 105	0	43
		glow-out	51	10	4	5	~ 105	0	43
	Configuration (4)		Y	Y	Y	Y	N	Y	N
	Residue (%) (5)		0	>99	>99	100	0	100	0
	Melted (6)		N	N	N	N	N	N	(8)
	Color	exterior	---	white	white	white	---	white	blk/wht
		interior	---	---	---	"	---	"	"
	Exterior Texture	powdery (6)	---	N	N	N	---	N	Y
		crusty (6)	---	N	N	N	---	N	Y

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. Ambient pressure air
8. Specimen fell off wire at end of test

TABLE 6-82 (cont'd)
FLUOROSILICONE RTV'S WITH INORGANIC ADDITIVES
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

		Specimen					
		356-1-1	356-1-1	384-1-1	389-1-1	389-1-1	389-1-2
	O$_2$ Pressure (psia)	6.2	9.72 (60%)	6.2	6.2	9.72 (60%)	9.72 (60%)
RESULTS	Wire Ignition (1)	T	T/B	T/B	T/B	T/B	T/B
	Wire Failure (1)	T	T	T	T	T/B	B
	Smoke (2)	N	S	S	S	S	P
	Burn time (sec.) (3) flame-out	0	38	58	25	67	45
	Burn time (sec.) (3) glow-out	0	38	58	25	67	45
	Configuration (4)	Y	N	Y	Y	Y	N
	Residue (%) (5)	100	0	95	95	70	15
	Melted (6)	N	(8)	N	N	N	Y
	Color exterior	white	blk/wht	white	white	white	white
	Color interior	"	"	black	black	black	black
	Exterior Texture powdery (6)	N	Y	N	N	N	N
	Exterior Texture crusty (6)	N	Y	Y	Y	Y	Y

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. Ambient pressure air
8. Specimen fell off wire at end of test

Although both mono- and di-ammonium phosphate were evaluated in preceding sections of this Program and found to be unsatisfactory, a related compound, Phoschek P/30 Regular, an ammonium polyphosphate, was tested with 77-033 (F-389) since it was alleged by the vendor that it had greater phosphorous content (i.e. 32%), very low water solubility, and an almost neutral P^H (i.e. 6.5). The resultant product had an extremely high viscosity and very bad mechanical properties. In addition, the 6.2 psia flammability specimen was quite degraded.

Other than the boric acid loaded 77-033 (F-242), the ammonium fluoborate formulation (F-266) was the only one to survive the 60/40 environment (i.e. 2 undamaged specimens out of 2 tests). However, unlike F-242, no success was obtained at 16.5 psia oxygen. But two compensating factors were of considerable interest. The viscosity of F-266 was noticeably lower and its mechanical properties were significantly better than that of F-242.

6.3.2.2 Organic Flame Retardants - Various organic compounds which had low carbon and hydrogen, and which also contained halogens, were investigated in conjunction with 77-033 (see Table 6-83). KEL-F KF-6061 powder was etched per MSC-SPEC-Q-3 (Ref. Appendix F) prior to formulation blending in order to obtain better adhesion to the polymer (F-283). When the KEL-F was also ground prior to curing (see Table 6-84), the resultant strip molding F-296) had even better mechanical properties (see Table 6-85).

F-385 and F-386 were formulated with an organometallic polymer which contains zinc, phosphorus and antimony. Neither cured and both were white on the surface and black on the bottom of the aluminum molding dish. The apparent problem of inhibition/side reaction was checked with the 77-033 base (F-390) and with boric acid (F-391). The appearance was only white and the systems remained uncured. From this it was assumed that the catalyst and ZB-S10 entered into some unknown side reaction.

A series of formulations were made with Dechlorane 602, 603, and 604. Unlike boric acid, they are essentially insensitive to moisture, and have higher melting points. Other pertinent properties are tabulated in Table 6-86.

TABLE 6-83

FLUOROSILICONE RTV'S WITH ORGANIC ADDITIVES

		Formulations (phr)										
Component	S	283	296	385	386	390	391	442	388	443	454	387
77-033	12	100.	100.	100.	100.	100.		100.	100.	100.	100.	100.
KEL-F KF-6061 (1)	2	150.										
KEL-F KF-6061 (2)	2		150.									
PYROSTAT ZBS10 (3)	34			150.		150.	150.					
PYROSTAT ZBS10 (4)	34				150.							
Boric acid (5)	17						150.					
FIREMASTER BP-6 (6)	28							150.				
DECHLORANE 602	16								150.	150.		
DECHLORANE 603	16										150.	
DECHLORANE 604	16											150.
77-033 Catalyst	12	10.	10.	10.	10.			10.	10.	10.	10.	10.

S......Source (See Table 6-1)
1. Pre-etched
2. Pre-etched and ground
3. Low viscosity
4. High viscosity
5. "Special Quality", milled
6. Hexabromobiphenyl

TABLE 6-84

FLUOROSILICONE RTV'S WITH ORGANIC ADDITIVES

- FORMULATION PROCESSING -

Formulation No. F-	Cure (1) Time (hrs/min)	Cure (1) Temp. (°F)	Post Cure Time (hrs/min)	Post Cure Temp. (°F)
283	17/15	150	--	--
296	4/-	"	--	--
385	26/30	"	--	--
386	25/-	"	--	--
390	26/-	"	--	--
391	25/30	"	--	--
442A	1/10	"	--	--
442B	"	"	13/50	150
388	2/-	"	19/45	"
443A	2/10	"	--	--
443B	"	"	13/-	150
387	2/-	"	21/40	"

1. Time in mold

TABLE 6-85

FLUOROSILICONE RTV'S WITH ORGANIC ADDITIVES CHARACTERISTICS

-CURED/POST CURED-

	Formulation							
Properties	283	296	442A	442B	388	443A	443B	387-2
Cured	Y	Y	Y	Y	Y	Y	Y	Y
Tacky	N	S	VS	N	VS	N	N	VS
Flexible	Y	Y	Y	Y	Y	Y	Y	Y
Memory	Y	Y	Y	Y	Y	F	F	Y
Cohesion	F	F	P	P	P	P	P	P
Tear Resistance	F	F	P	P	P	P	P	P
Crack Resistance (1)	F	X	X	X	X	X	X	X
Crack Propagation (1)	F	X	X	X	X	X	X	X
Porous	N	N	S	S	N	S	S	X
Adheres to unclean alum. Foil cup	P	P	-	-	F	-	-	N
Cheesy	N(2)	N(2)	N(2)	N(2)	N	N	N	N

Y.....Yes VS.....Very Slight S.....Slight N.....No
X.....Satisfactory F.....Fair P.....Poor NG.....Not Good

1. 180° bend (face-to-face contact)
2. Filler powders out when broken edge is rubbed.

TABLE 6-86

DECHLORANE COMPOUNDS (3)

		602	603	604
Halogen Content (%)	Chlorine	69.4	66.7	---
	Bromine	---	---	---
	Cl / Br	---	---	76.9
Melting point (°F.)		554-560	730	354
Density (gm/cc)		2.0 (25°C)	1.85 (24°C)	2.37 (20°C)
Vapor pressure (torr)		0.08 (197°C)	0.008 (197°C)	0.0014 (139°C)
Cost, approx. ($/lb.)		0.70-0.75	3.00	3.00 (1) 1.00 (2)

(1) Introductory.
(2) Volume.
(3) Hooker Chemical data.

F-387, containing Dechlorane 604, clearly had superior flammability resistance to any of the other formularions in this section. Its qualitative mechanical properties seemed reasonably good. See Table 6-87.

6.3.2.3 Inorganic/Organic Flame Retardants - Dechlorane 604 was blended with 77-033 and different types of potentially useful inorganic compounds with the object of achieving a synergistic relationship (see Table 6-88). A good cure was generally obtained (see Table 6-89) except for the one conraining antimony oxide (F-401); slight tackiness being observed (see Table 6-90). The zinc borate (F-415) and iron oxide formulations (F-438, -439, and -440) did not even pass 6.2 psia oxygen (see Table 6-91). At 60/40, the survival rate (i.e. self-extinguishing) was:

F-399: 3 out of 3
F-401: 2 " " 3
F-445: 1 " " 1
F-446: 3 " " 3

F-399 and F-445 were also checked at 16.5 psia oxygen. Both completely degraded.

As seen in Table 6-86, the Dechlorane 604 has between 10 and 14% more total halogen, by weight, than 602 and 603. It was not felt that this weight difference was sufficient to account for their significant difference in flammability resistance. Another possibility was the presence of bromine in the molecule. A third one could be a possible synergistic relationship between the chlorine and bromine (and, perhaps, fluorine from the 77-033). Finally, one had also to consider the respective melting

TABLE 6-87
FLUOROSILICONE RTV'S WITH ORGANIC ADDITIVES
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

		Specimen								
		283-1-1	296-1-1	442-1B-1	388-1-1	443-1B-1	387-2-1	387-2-1	387-2-2	387-2-1
	O_2 Pressure (psia)	6.2	6.2	6.2	6.2	6.2	6.2	9.72 (60%)	9.72 (60%)	16.5
RESULTS	Wire Ignition (1)	T/B	T/B	T/B	T/B	T/B	T/B	T/B	B	T/B
	Wire Failure (1)	T	T	T	B	B	B	B	B	B
	Smoke (2)	P	S	P	S	S	N	N	N	S
	Burn time (sec.) (3) flame-out	57	55	73	75	35	17	15	5	45
	Burn time (sec.) (3) glow-out	57	55	73	75	90	17	15	5	75
	Configuration (4)	Y	Y	Y	Y	Y	Y	Y	Y	---
	Residue (%) (5)	70	70	70	75	50	95	95	98	75
	Melted (6)	N	N	N	N	N	N	---	N	N
	Color exterior	gray	gray	blk/wht	wht/blk	white	black	black	black	wht/blk
	Color interior	"	---	"	"	"	---	"	"	"
	Exterior Texture powdery (6)	Y	Y	Y	Y	Y	N	N	N	Y
	Exterior Texture crusty (6)	Y	Y	Y	Y	Y	Y	Y	Y	Y

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No

TABLE 6-88

FLUOROSILICONE RTV'S WITH ORGANIC AND INORGANIC ADDITIVES

		Formulations (phr)													
Component	S	399	401	415	445	446	438	439	440	462	435	436	444	437	434
77-033	12	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.	100.
DECHLORANE 604	16	75.	150.	150.	150.	150.	125.	125.	125.					75.	
Hexabromobenzene	28									150.		75.	75.	75.	150.
DECHLORANE 510	16										150.	75.			
DECHLORANE 602	16												75.		
Boric acid (1)	17	75.													
Antimony oxide (2)	20		20.												
XPI-158 (3)	4			20.											
Glass Frit 7570 (4)	21				50.										
Glass Shot MS-L (5)	33					50.									
Synthetic iron oxide(6)	4						25.								
Synthetic iron oxide(7)	20							25.							
Synthetic iron oxide(8)	4								25.						
BUSAN 11-MI (9)	19														40.
77-033 Catalyst	12	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.

S......Source (See Table 6-1)
1. "Special Quality", milled
2. White Star - "M" grade
3. Zinc borate
4. 325 mesh, predried 6 hrs./212°F.
5. See Table
6. Red # 179
7. Meteor Red Brown # 7751
8. Ultrafine, Red, Dark # 1121M
9. Barium metaborate

TABLE 6-89

FLUOROSILICONE RTV'S WITH ORGANIC AND INORGANIC ADDITIVES

- FORMULATION PROCESSING -

	Cure (1)		Post Cure	
Formulation No. F-	Time (hrs/min)	Temp. (°F)	Time (hrs/min)	Temp. (°F)
399-1	14/-	150	---	---
399-2	2/-	"	11/-	150
401-1	15/35	"	---	---
401-2	12/30	"	---	---
415	3/10	"	15/30	150
445	12/-	"	---	---
446	11/1	"	---	---
438	1/20	"	32/30	150
439	33/-	"	---	---
440A	1/20	"	---	---
440B	"	"	14/10	150
462	12/30	"	---	---
435A	-/40	"	---	---
435B	"	"	57/-	150
436	55/-	"	---	---
444A	2/-	"	---	---
444B	"	"	13/-	150
437	55/-	"	---	---
434A	1/10	"	---	---
434B	"	"	58/-	150

1. Time in mold

TABLE 6-90

FLUOROSILICONE RTV'S WITH ORGANIC AND INORGANIC ADDITIVES

CHARACTERISTICS

-CURED/POST CURED-

Properties	399-1	401	415	445	446	438	440A	440B
Cured	Y	?	Y	Y	Y	Y	Y	Y
Tacky	N	S	N	N	N	N	VS	N
Flexible	Y	Y	Y	Y	Y	Y	Y	Y
Memory	Y	Y	Y	F	Y	Y	Y	Y
Cohesion	P	P	P	P	P	P	P	P
Tear Resistance	P	P	P	P	P	P	P	P
Crack Resistance (1)	X	X	X	X	X	X	X	X
Crack Propagation (1)	X	X	F	X	X	X	X	X
Porous	N	X	---	N	S	S	N	N
Adheres to unclean alum. Foil cup	F	---	---	---	---	---	---	---
Cheesy	N	N	N	N	---	N (2)	N (3)	N (3)

Y.....Yes VS.....Very Slight S.....Slight N.....No
X.....Satisfactory F.....Fair P.....Poor

1. 180° bend (face-to-face contact)
2. Small particles readily came out
3. Some " " " "

TABLE 6-90 (cont'd)

FLUOROSILICONE RTV'S WITH ORGANIC AND INORGANIC ADDITIVES

CHARACTERISTICS

-CURED/POST CURED-

Properties	435A	435B	436	444A	444B	437	434A	434B
Cured	Y	Y	Y	Y	Y	Y	N	Y
Tacky	Y	Y	N	N	N	N	---	N
Flexible	Y	Y	Y	Y	Y	Y	---	Y
Memory	Y	Y	Y	F	F	Y	---	Y
Cohesion	P	F	P	P	F	P	---	P
Tear Resistance	P	P	P	P	P	F	---	P
Crack Resistance (1)	X	X	X	X	X	X	---	X
Crack Propagation (1)	X	X	F	X	X	X	---	X
Porous	N	N	N	VS	VS	N	---	S
Adheres to unclean alum. Foil cup	---	---	---	---	---	---	---	---
Cheesy	N	N	N	N	N	N	---	N

Y.....Yes VS.....Very Slight S.....Slight N.....No
X.....Satisfactory F.....Fair P... P.....Poor NG.....Not Good

1. 180° bend (fact-to-face contact)
2. Small particles readily came out
3. Some " " " "

TABLE 6-91
FLUOROSILICONE RTV'S WITH ORGANIC AND INORGANIC ADDITIVES
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

		Specimen							
		399-1-1	399-1-2	399-1-1	399-1-2	399-2-1	399-2-1	401-1-1	401-1-2
	O_2 Pressure (psia)	6.2	6.2	9.72 (60%)	9.72 (60%)	9.72 (60%)	16.5	6.2	6.2
RESULTS	Wire Ignition (1)	T/B	T/B	T	T/B	T/B	T/B	B	T/B
	Wire Failure (1)	T/B	T	T	T/B	B	T	T/B	T
	Smoke (2)	N	N	N	N	N	P	N	---
	Burn time (sec.) (3) flame-out	15	7	8	7	9	55	10	5
	Burn time (sec.) (3) glow-out	15	7	8	7	9	60	10	5
	Configuration (4)	Y	Y	Y	Y	Y	Y	Y	Y
	Residue (%) (5)	98	99	98	99	95	85	100	98
	Melted (6)	N	N	N	N	N	N	N	N
	Color: exterior	black	black	black	black	black	blk/wht	white	black
	: interior	"	"	---	---	---	"	"	---
	Exterior Texture powdery (6)	N	N	N	N	N	N	N	N
	Exterior Texture crusty (6)	N (7)	N (7)	Y (7)	Y (7)	Y (7)	Y	N (8)	Y (7)

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left-in (4)
6. Yes or No
7. Only corners of specimen discolored
8. Premature wire failure

TABLE 6-91 (cont'd)
FLUOROSILICONE RTV'S WITH ORGANIC AND INORGANIC ADDITIVES
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

		Specimen							
		401-1-1	401-1-2	401-2-1	415-1-1	445-1-1	445-1-2	445-1-1	445-1-1
	O_2 Pressure (psia)	9.72 (60%)	9.72 (60%)	9.72 (60%)	6.2	6.2	6.2	9.72 (60%)	16.5
RESULTS	Wire Ignition (1)	T/B	B	T/B	T/B	---	T/B	B	T/B
	Wire Failure (1)	T/B	B	B	B	---	T	T	B
	Smoke (2)	N	N	P	P	---	N	N	S
	Burn time (sec.) (3) flame-out	7.5	4	54	60	3	3	9	34
	Burn time (sec.) (3) glow-out	7.5	4	54	60	3	3	9	55
	Configuration (4)	Y	Y	Y	Y	---	Y	Y	Y
	Residue (%) (5)	98	98	75	80	---	98	99	70
	Melted (6)	N	N	N	Y	---	N	N	N
	Color: exterior	black	black	blk/wht	wht/blk	---	black	black	wht/blk
	: interior	---	---	"	---	---	---	---	"
	Exterior Texture powdery (6)	N	N	Y	N	---	N	N	N
	Exterior Texture crusty (6)	Y	Y	Y	Y	---	N	N	Y
		(7)	(7)			(8)	(7)	(7)	(9)

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. Ambient pressure
8. Premature wire failure
9. Completely burnt

TABLE 6-91 (cont'd)
FLUOROSILICONE RTV'S WITH ORGANIC AND INORGANIC ADDITIVES
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

		Specimen							
		446-1-1	446-1-1	446-1-2	446-1-3	438-1B-1	439-1-1	440-1B-1	435-1A-1
	O_2 Pressure (psia)	6.2	9.72 (60%)	9.72 (60%)	9.72 (60%)	6.2	6.2	6.2	6.2
RESULTS	Wire Ignition (1)	B	B	B	B	T/B	T/B	T/B	T/B
	Wire Failure (1)	B	B	T	T	T	B	B	B
	Smoke (2)	N	N	N	N	P	P	P	P
	Burn time (sec.) (3) flame-out	3.5	7	5	3	45	45	34	39
	Burn time (sec.) (3) glow-out	3.5	7	5	3	85	45	38	75
	Configuration (4)	Y	Y	Y	Y	Y	Y	Y	Y
	Residue (%) (5)	98	98	99	99	75	75	75	15
	Melted (6)	N	N	N	N	N	N	N	N
	Color: exterior	black	black	black	black	(10)	(10)	(10)	wht/blk
	: interior	---	---	---	---	(10)	(10)	(10)	"
	Exterior Texture powdery (6)	N	N	---	N	Y	Y	Y	Y
	Exterior Texture crusty (6)	N	N	---	N	Y	Y	Y	Y
		(7)	(7)	(7)	(7)	(9)	(9)	(9)	(9)

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. Ambient pressure air
8. Premature wire failure
9. Completely burnt
10. White/black/red

TABLE 6-91(cont'd)
FLUOROSILICONE RTV'S WITH ORGANIC AND INORGANIC ADDITIVES
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS-

		Specimen						
		436-1-1	444-1B-1	437-1-1	437-1-1	437-1-2	437-1-1	434-1-1
	O_2 Pressure (psia)	6.2	6.2	6.2	9.72 (60%)	9.72 (60%)	16.5	6.2
RESULTS	Wire Ignition (1)	T/B	T/B	---	T/B	T/B	T/B	B
	Wire Failure (1)	B	T	T	T	T	T	N
	Smoke (2)	P	S	N	N	N	S	P
	Burn time (sec.) (3) flame-out	49	60	3	4.5	4	30	66
	Burn time (sec.) (3) glow-out	49	115	3	4.5	4	74	66
	Configuration (4)	Y	Y	Y	Y	Y	Y	Y
	Residue (%) (5)	15	75	100	97	98	75	15
	Melted (6)	N	N	N	N	N	N	N
	Color: exterior	blk/wht	wht/blk	---	black	black	wht/blk	black
	: interior	"	(10)	---	---	---	gray	"
	Exterior Texture powdery (6)	N	Y	N	N	N	Y	N
	Exterior Texture crusty (6)	Y	Y	N	N	N	Y	Y
			(9, 10)	(11)	(7)	(7)	(9)	(9)

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. Ambient pressure air
8. Premature wire failure
9. Completely burnt
10. White/black/red
11. Poor ignition

points. In order to briefly evaluate these factors, several formulations were prepared with the Dechloranes, including D-510, $C_{10}Cl_{12}$ (78% chlorine, m.p. = 905°F), and with hexabromobenzene, C_6Br_6 (85.9% bromine, m.p. = 612°F). Thus, the additive(s) in F-435 had 78% chlorine. F-436 had 39% chlorine and 43.5% bromine (total halogen equal to 82.5%). F-444 had 34.7% chlorine and 43.5% bromine (total halogen equal to 78.2%). F-437 had total halogen equal to 82%. It is seen that the total halogens in F-436 and F-437 are comparable, but a) each halogen type comes from completely separate compounds in F-436, and b) the availability of a comparatively low temperature decomposing halogen compound exists in F-437. F-435, -436, -444, and -434 did not survive 6.2 psia oxygen: F-437 withstood 60/40 (i.e. 2 out of 2 specimens), but completely decomposed in 16.5 psia oxygen.

Only very slight differences were observed between F-387 (150 phr D-604) (see Table 6-83) and F-437 with respect to flammability or tested specimen appearances.

6.3.2.4 Carriers Loaded With Flame Retardant Liquids - The formulations prepared in this section are shown in Table 6-92, their processing is noted in Table 6-93, their mechanical/physical properties after cure are listed in Table 6-94, and their flammability resistance is tabulated in Table 6-95.

TABLE 6-93

FLUOROSILICONE RTV'S WITH FLAME RETARDANT LIQUIDS/CARRIERS

- FORMULATION PROCESSING -

Formulation No. F-	Cure (1) Time (hrs/min)	Cure (1) Temp. (°F)	Post Cure Time (hrs/min)	Post Cure Temp. (°F)
256	17/-	150	---	---
250	-/53	"	---	---
252	-/50	"	---	---
254	20/25	"	---	---
255	15/35	"	---	---
251	18/-	"	---	---
379	22/-	"	---	---

(1) Time in mold.

F-250 was too mechanically weak to consider its use. F-252 and F-254 showed liquid separation. F-256 was satisfactory for use at 6.2 psia oxygen, while F-255 and F-251 were relatively undamaged in 60/40. However, none had sufficient flexibility or cohesive strength.

TABLE 6-92

FLUOROSILICONE RTV'S WITH FLAME RETARDANT LIQUIDS/CARRIERS

Component	S	Formulations (phr)						
		256	250	252	254	255	251	379
77-033	12	100.	100.	100.	100.	100.	100.	100.
Boric acid (1)	9	100.	150.			150.	100.	150.
Glass frit 7570	21			150.	150.	50.		
Mol. Sieves 13X	8		50.	50.	50.	50.		
231 (2)	13	150.						
225 (2)	13						50.	
Freon E-9	1		100.	100.	50.	100.		
231X (2)	13							100.
77-033 Catalyst	12	10.	10.	10.	10.	10.	10.	10.

S......Source (See Table 6-1)
1. 100% through 170 mesh
2. See Table 6-73.

TABLE 6-94

FLUOROSILICONE RTV'S WITH FLAME RETARDANT LIQUIDS/CARRIERS

CHARACTERISTICS

-CURED/POST CURED-

Properties	256	254	255	251
Cured	Y	Y	Y	Y
Tacky	oily	oily	N	N
Flexible	Y	Y	P	Y
Memory	F	---	P	F
Cohesion	NG	X	NG	P
Tear Resistance	NG	---	NG	P
Crack Resistance (1)	NG	---	NG	NG
Cràck Propagation (1)	NG	---	NG	NG
Porous	N	---	Y	N
Adheres to unclean alum. Foil cup	---	---	---	P
Cheesy	S	N	Y	N

Y.....Yes
X.....Satisfactory
F.....Fair
P.....Poor
S.....Slight
NG. ....Not Good

1. 180° bend (face to face contact)

TABLE 6-95
FLUOROSILICONE RTV'S WITH FLAME RETARDANT LIQUIDS/CARRIERS
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

		Specimen							
		256-1-1	256-1-1	255-1-1	255-1-1	255-1-1	251-2-1	251-2-1	251-2-1
	O_2 Pressure (psia)	6.2	9.72 (60%)	6.2	9.72 (60%)	16.5	6.2	9.72 (60%)	16.5
RESULTS	Wire Ignition (1)	T	T/B	T/B	T/B	T/B	T/B	T/B	T/B
	Wire Failure (1)	T	B	T/B	B	T	T	T/B	T/B
	Smoke (2)	N	P	N	N	S	N	N	S
	Burn time (sec.) (3) flame-out	2	60	3	11	67	5	14	50
	Burn time (sec.) (3) glow-out	2	60	3	11	67	5	16	65
	Configuration (4)	Y	Y	Y	Y	Y	Y	Y	Y
	Residue (%) (5)	99	80	99	99	85	99	99	90
	Melted (6)	N	N	N	N	N	N	N	N
	Color: exterior	white	wht/blk	white	white	white	white	white	wht/blk
	Color: interior	---	---	---	---	---	---	---	---
	Exterior Texture powdery (6)	N	N	N	N	N	N	N	N
	Exterior Texture crusty (6)	N	Y	N	N	Y	N	Y	Y

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No

6.4 MISCELLANEOUS FORMULATIONS

A brief examination was given to some less exotic (and less costly) approaches than those heretofore reviewed. This section describes such evaluations.

6.4.1 Polyesters

Initial formulation (see Table 6-96) activity in this section was devoted to the evaluation of a non-flexible system (F-383) developed for NASA by another contractor (Ref. 6-29). Although this formulation was undamaged in 6.2 psia oxygen, it was completely destroyed in the 60/40 environment. Diallyl chlorendate (DAC) was then added as a reactive low hydrogen (i.e. 2.56%) diluent. Although it did reduce the viscosity, the modified formulation, F-392, was totally destroyed in 6.2 psia oxygen (see Table 6-99). An attempt to reduce the viscosity with Pyrostat ZB-S10 resulted in a blend which did not cure (see Tables 6-97 and 6-98).

TABLE 6-97

POLYESTER POLYMERS

- FORMULATION PROCESSING -

Formulation No. F-	Steps (2) 1	2	3	4	5 (min)	5 (°F)	6 (hr/min)	6 (°F)	7 (d/hr)	7 (°F)
380	X	-	-	-	10	80	-	-	-	-
382	X	-	-	-	10	80	-	-	-	-
383	-	-	-	X	5	84	2/40	150	19hr/-	150
392	X	-	-	X	10	"	24/-	"	-	-
393	X	-	-	X	5	82	21/-	"	-	-
394	X	(1)	-	X	3	79	24/-	"	-	-
457	X	212	-	-	-	-	-	-	-	-
458	X	"	-	-	-	-	-	-	-	-
400	-	-	-	X	10	80	61/-	150	-	-
459	X	80	-	-	-	-	-	-	-	-

(1) Approx. 2 min. @ 194°F.

(2) See Table 6-12.

TABLE 6-96

POLYESTER POLYMERS

		Formulations (phr)									
Component	S	380	382	383	392	393	394	457	458	400	459
MR-670	42	100.									
GR-684	42						100.				
DION FR 6430	43							100.	100.		
DION ISO 6481	43								100.		100.
DAC (1)	25				20.			100.			100.
L-2344 (2)	2						100.				
F-380-1	13		440.								
F-382-1	13			440.1	440.1	440.1					
F-458-1	13									100.	
Tabular alumina	--	340.									
PYROSTAT ZBS10 (3)	34					20.					
Co. Naph. (4)	--		0.1					0.2	0.2		0.2
MEKP-5 (5)	44			1.	1.	1.	1.				
TS-50 (6)	45									2.	

S......Source (See Table 6-1)
1. Diallyl chlorendate
2. Dihydroxy terminated fluoropolymer
3. Organo metallic polymer
4. Cobalt napthenate
5. Methyl ethyl ketone peroxide (60%) in dimethyl phthalate
6. 2,4 -Dichlorobenzoyl peroxide (50%) in silicone fluid

TABLE 6-98

POLYESTER POLYMERS CHARACTERISTICS

-CURED/POST CURED-

Properties	Formulation 383	 392	 394
Cured	Y	Y	Y
Tacky	N	N	S
Flexible	N	N	N
Memory	-	-	-
Cohesion			
Tear Resistance			
Crack Resistance (1)			
Crack Propagation (1)			
Porous			
Adheres to unclean alum. Foil cup		F	
Cheesy			

Y.....Yes VS.....Very Slight S.....Slight N.....No
X.....Satisfactory F.....Fair P.....Poor NG.....Not Good
1. 180° bend (face-to-face contact)

The supplier of MR-670 indicated (Ref. 6-30) that it is an unaccelerated 25% styrene filled, brominated polyester. GR-684 is identical but is accelerated. L-2344 was added to the latter as a liquid, albeit high viscosity, flame retardant (1.8% hydrogen) and plasticizer. The cured structure was tacky on the surface and brittle.

A non-styrenated, but brominated solid polyester, Dion FR 6430, was put into very high viscosity liquid states with DAC (F-457) and with a non-halogenated, flexible polyester F-458). F-459 had a low viscosity, but a non-styrenated halogenated version of Dion ISO 6481 could not be located.

6.4.2 Diallyl Chlorendate (DAC)

As noted above, DAC has only 2.56% hydrogen, it is a low viscosity liquid, and has two allyl groups which may be reactive at 150°F if properly catalyzed. Additionally, it was of interest as a diluent (reactive?) for silicone and fluorosilicone addition reaction RTV's, and fluorocarbon elastomers. F-331, -332, -333, and -334 were formulated (see Table 6-100 for formulations and Table 6-101 for cure cycles) to determine compatibility of the Sylgard-type silicone RTV with DAC. Separation was noted in F-334. A similar effort with 77-033 was also fruitless:

TABLE 6-99
POLYESTER POLYMERS
PRELIMINARY FLAMMABILITY
- TEST CONDITIONS/RESULTS -

		Specimen		
		383-1-1	383-1-1	392-1-1
	O_2 Pressure (psia)	6.2	9.72 (60%)	6.2
R	Wire Ignition (1)	T/B	T/B	T/B
E	Wire Failure (1)	T	B	B
	Smoke (2)	N	S	N
S	Burn time flame-out	5	110	40
	(sec.) (3) glow-out	5	120	45
U	Configuration (4)	Y.	Y	Y
L	Residue (%) (5)	100	80	---
T	Melted (6)	N	N	N
	Color: exterior	white	black	white
S	: interior	"	white	"
	Exterior powdery (6)	N	Y	Y
	Texture crusty (6)	N	Y	Y

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No

TABLE 6-100

DIALLYL CHLORENDATE POLYMERS

		Formulations (phr)										
Component	S	331	332	333	334	335	215	455	456	395	396	402
DAC (1)	44	100.	100.	100.	100.	100.	50.	100.		100.	25.	25.
SYLGARD 186 (2)	12				100.							
77-033 (3)	12					100.	100.	100.				
L-2344 (4)	2										100.	100.
F-455-1	13								212.			
Boric acid (5)	17					300.	225.					
DECHLORANE 604	16								300.			
TDI (6)	--									5.22		5.22
Catalyst T-9 (7)	--											0.5
TS-50 (8)	45							2.				
S-186 Catalyst	12	1.	5.	10.								
77-033 Catalyst	12					10.	10.	10.				

S......Source (See Table 6-1)
1. Diallyl chlorendate
2. Silicone RTV
3. Fluorosilicone RTV
4. Dihydroxy terminated fluoropolymer
5. "Special Quality" grade
6. Tolylene diisocyanate
7. Stannous octoate
8. 2,4 - Dichlorobenzoyl peroxide (50%) in silicone fluid

TABLE 6-100 (cont'd)

DIALLYL CHLORENDATE POLYMERS

		Formulations (phr)									
Component	S	432	381	447	448	449	450	451	452	453B	453A
DAC (1)	44	50.	100.	100.	100.	100.	100.	100.	100.	100.	100.
L-2344 (4)	2	100.								100.	100.
DECHLORANE 604	16										147.
TDI (6)	--									5.22	5.22
MEKP-5 (9)	44		2.								
TS-50 (8)	45			2.	2.	2.	2.	2.	2.	2.	2.
Co. Naph. (10)	--		0.5					0.5	0.2	0.2	0.2
TMG (11)	10				0.2						
DMT (12)	47					0.2					
DMA (13)	--						0.2				

9. Methyl ethyl ketone peroxide (60%) in dimethyl phthalate
10. Cobalt napthenate (6% Co)
11. 1,1,3,3 - Tetramethylguanadine
12. N,N - Dimethyl-p-toluidine
13. Dimethyl aniline

TABLE 6-101

DIALLYL CHLORENDATE POLYMERS

- FORMULATION PROCESSING -

Formulation No. F-	Steps (9) 1	2	3	4	5 (min)	5 (°F)	6 (hr/min)	6 (°F)	7 (d/hr)	7 (°F)
331	-	(1)	-	X	2	72	14d/16hr	150	-	-
332	-	(1)	-	X	"	"	14d/16hr	"	-	-
333	-	(1)	-	X	"	71	14d/15hr	"	-	-
334	-	(1)	-	X	3	"	"	"	-	-
335	X	(1)	-	X	10	84	18/-	"	-	-
	X	(1)	-	X	"	83	19/-	"	-	-
455	X	-	-	X	"	80	37/-	"	-	-
456	(2)	-	-	-	5	"	37/-	"	-	-
395	-	-	-	X	2	71	13/30	"	½/-	150
396	(2)	-	-	-	2	73	13/-	"	½/-	"
402	X	185	-	X	3	185	9/10	"	½/-	"
432	-	185	-	X	4	69	8/-	"	1/-	"
381	(3)	-	-	X	3	83	16/-	"	16/-	212
447-1	-	-	-	X	10	"	29/-	"	(4)	-
447-2	-	-	-	X	5	82	24/-	"	(5)	-
447-3	-	-	-	X	"	83	24/-	"	(6)	-
447-4	-	-	-	X	"	82	24/-	"	(7)	-
448	X	-	-	X	10	"	28/30	"	-	-
449	X	-	-	X	5	72	89/-	"	-	-
450	X	-	-	X	8	"	88/-	"	-	-
451	X	-	-	X	5	83	24/-	"	-	-
452	X	-	-	X	"	80	23/-	"	1/-	150
453	(8)	149	-	X	5	83	22/-	"	-	-

1. Warm DAC.
2. Blend all materials.
3. Blend DAC and MEKP-5 first.
4. Cured in air.
5. Cured under argon.
6. Cured under glycerine.
7. Cured under (?) FS-1265 fluorosilicone fluid.
8. Blend DAC and L-2344 first.
9. See Table 6-12.

F-335: soft, not cured
F-215: " " "
F-455: Separation, not cured
F-456: " , " "

The combination of DAC with L-2344 was examined, with the following results:

F-395: no cure (no effect)
F-396: no cure, creamy, opaque, some separation with time.
F-402: cured, porous, good cohesion and adhesion, soft, flexible, opaque, but high viscosity and gummy after blending
F-432: similar to F-396, except more separation.

An attempt was then made to find a suitable 150°F cure system for DAC for use with it alone or in conjunction with other polymeric structures. F-381-1 was degassed, F-381-2 was not. Both showed no change after a cure at 150°F, but these aluminum cup specimens became soft on bottom and dry/brittle on the top exposed surface after a post cure at 212°F. A more reactive free radical initiator was utilized in F-447 to reduce the cure temperature. Various non-reacting atmospheres were tried when a surface inhibition problem arose. These specimens seemed incompletely cured. F-448, -449, -450, -451, and -452 were prepared with known free radical accelerators. Of these, the ones with cobalt naphthenate were the only ones which resulted in a cure. The structure was soft/flexible underneath and dry/brittle on the surface. F-453 was prepared to flexibilize the DAC structure and incorporate a flame retardant. No cure was obtained; separation was evident.

6.4.3 Dimethyl Chlorendate (DMC)

As in the case of DAC, DMC has a high chlorine and low hydrogen content (i.e. 44.5-45.5 and 2.56 percents, respectively, for the former, and 51.08 and 1.92 percents, respectively, for the latter). However, DMC is considered essentially unreactive. If a condensation reaction between its methyl groups and the terminal hydroxyl groups of L-2344 or with an amine cured silicone RTV could be achieved, the residual chlorine/hydrogen content of the DMC portion would shift to 55%/0.517%, respectively. These approaches were considered in the formulations shown in Table 6-102.

The solid DMC was readily dispersed in the high viscosity L-2344 (see Table 6-103). After "cure," the product (F-059) was still fluid, miscible, and clear. F-060, -061, and -062 contained RTV 603 base. Again, complete compatibility was obtained. The "cured" materials were observed to be:

F-060: no cure
F-061: no cure at 150°F. Gelled, tacky, poor cohesion, clear after 200°F post cure.
F-062: No cure, clear, some gel, some crystals after cure. Gelled, tacky (much less than F-061), poor cohesion (better than F-061), and clear after post cure at 200°F.

TABLE 6-102

DIMETHYL CHLORENDATE POLYMERS

		Formulations (phr)										
Component	S	059	060	061	062	324	325	326	327	328	329	330
Dimethyl Chlorendate	46	14.	1.	5.	25.	50.	100.	25.	25.	50.	100.	100.
L-2344 (1)	2	100.										
RTV 603	7		100.	100.	100.				100.	100.	100.	100.
RTV 602	7					100.	100.	100.				
SRC-05	7							0.5	0.5			0.5

S......Source (See Table 6-1)
1. Dihydroxy terminated fluoropolymer

TABLE 6-103

DIMETHYL CHLORENDATE POLYMERS

- FORMULATION PROCESSING -

Formulation No. F-	Steps (1)									
	1	2	3	4	5 (min)	5 (°F)	6 (hr/min)	6 (°F)	7 (d/hr)	7 (°F)
059	X	-	-	-	5	176-194	~3 mo.	150	-	-
060	X	-	-	-	3	"	~3 mo.	"	-	-
061	X	-	-	-	4	"	~3 mo.	"	-	-
062	X	-	-	-	2	176	~3 mo.	"	-	-
324	X	-	-	-	15	176-194	13d/22hr	"	17/-	200
325	X	-	-	-	10	185	"	"	"	"
326	X	176-194	-	X	10	185	"	"	-	-
327	X	"	-	X	30	185	"	"	-	-
328	X	-	-	-	15	185	13d/20hr	"	17/-	200
329	X	-	-	-	"	176-194	"	"	"	"
330-1	X	176-194	-	X	"	185	13d/19hr	"	"	"
330-2	X	"	-	X	20	"	"	200	-	-

1. See Table 6-12.

The relative viscosities were: F-060 < -061 < -062 ≪ -059.

When 50 (F-328) and 100 phr (F-329) DMC were added to the RTV 603, gelation did not occur.

RTV 602 was substituted for RTV 603. The products were similar to F-062.

A 12 day 18 hour exposure of DMC to 150 and 212°F was carried out. There was an approximately 1% weight loss at 150°F, but a 40% loss at 212°F.

It was obviously necessary to "tie-up" the DMC or thermally lose it. One method was to catalyze the RTV. These resulted in castings which showed crystals intermixed with the cured RTV when processing was carried out at 150°F, but none when processed at 200°F. The data in Table 6-104 verifies a) the greater non-miscibility of significant quantities of DMC in the dimethyl RTV 602 as compared to the methylphenyl RTV 603, and b) the loss of DMC at the higher processing temperatures.

TABLE 6-104

DIMETHYL CHLORENDATE POLYMERS CHARACTERISTICS

-CURED/POST CURED-

Properties	Formulation 326	Formulation 327	Formulation 330-2
Cured	Y	Y	F
Tacky	N	VS	Y
Flexible	Y	Y	Y
Memory	Y	Y	Y
Cohesion	P	P	P
Tear Resistance	P	P	P
Crack Resistance (1)	X	X	X
Crack Propagation (1)	Y	S	X
Porous	?	?	N
Adheres to unclean alum. Foil cup	N	N	S
Cheesy	Y	S	gummy

Y.....Yes VS.....Very Slight S.....Slight N.....No
X.....Satisfactory F.....Fair P.....Poor NG.....Not Good

1. 180° bend (face-to-face contact)

SECTION 7

NONFLAMMABLE FORMULATIONS DEVELOPMENT: FINAL SCREENING

7.1 FORMULATIONS/PROCESSING

The formulations selected for final screening are shown in Table 7-1. Their processing is described in Table 7-2. The selection was primarily based upon flammability resistance, and secondarily upon handling characteristics and apparent mechanical properties.

7.2 EVALUATION

The final screening tests consisted of:

a. Flammability (Ref. Appendix B)
b. Thermal Offgassing: Carbon Monoxide/Total Organics (Ref. Appendix C)
c. Thermal Offgassing: Odor (Ref. Appendix D)
d. Thermal Analysis: TGA/DTA (Ref. Appendix A, Section 3.3.8)
e. Vacuum Outgassing (Ref. Appendix A, Section 3.4.4, Paragraphs 1 and 2)
f. Insulation Resistance (Ref. 7-1, Section 4.4.3.5., 75°F).

7.2.1 Flammability

In accordance with the above requirements, the flammability evaluarion was carried out with simulated configurarion tests by using potted pygmy connectors. (See Table 7-3). As stated in the Statement of Work (Ref. Appendix A, Section 3.4.1), the criteria is that the "Candidate materials shall be self-extinguishing with no further evidence of degradation by combustion..." The two key words herein are "self-extinguishing" and "furrher." The primary NASA (and probably in all industry) document (Ref. 6-1) on this subject does not define the former term, but it does define "flammable" and "nonflammable" as follows:

Flammable:	Under specific ignition and environment conditions, the ability of solids, liquids or gases to react with sufficient energy to cause continued self-propagation of a flame front thioughout the material.
Nonflammable:	The antonym of flammable. Under specified ignition and environment conditions, the ability of solids, liquids, or gases to resist and stop chemical reaction so that there is not sufficient energy to sustain self-propagation of a flame front throughout the material.

TABLE 7-1

FINAL SCREENING

Component	S	Formulations (phr) 175X	231X	230X	266	378-1	378-3	379-1	379-3	387	437	462
77-024	12	100.		100.								
77-033	12				100.	100.	100.	100.	100.	100.	100.	100.
F-231X-1	13			100.								
F-231X-2	13							100.	100.			
Boric acid	9	125.										
Boric acid (1)	9			150.								
Boric acid (2)	9					125.		150.				
Boric acid (1, 3)	17						125.					
Boric acid (3)	17								150.			
Mol. Sieves 13X	8		100.									
KRYTOX 143AC	1		300.									
NH_4BF_4	20				150.							
DECHLORANE 604 (4)	16									150.		
DECHLORANE 604 (1)	16										75.	
Hexabromobenzene	28										75.	150.
77-024 Catalyst	12	10.		10.								
77-033 Catalyst	12				10.	10.	10.	10.	10.	10.	10.	10.

S.....Source (See Table 6-1)
1. 100% through 170 mesh
2. 100-170 mesh
3. "Special Quality," milled
4. See Tables 8-2 and 8-3

TABLE 7-2

FINAL SCREENING FORMULATIONS PROCESSING

Formulation No. F-	Cure (1) Time (hrs/min)	Temp. (°F)	Post Cure Time (hrs/min)	Temp. (°F)
175X-1	7/-	150	---	---
230X-1	9/-	"	---	---
266-2	13/45	"	---	---
266-4	24/-	"	---	---
378-1	2/-	"	22/-	150
378-3	24/40	"	---	---
379-1	22/-	"	---	---
379-3	24/-	"	---	---
387	(see Tables 8-2,		8-3, and 8-4)	
437-2, -4	12/30	"	---	---
462-1	12/30	"	---	---
462-2	2/10	"	10/-	150

1. Time in mold

TABLE 7-3

FINAL SCREENING FLAMMABILITY TEST (1)
-CONDITIONS-

Furane Ident. No.	175X-1B-1	175X-1B-2	175X-1B-3	230X-1B-1	230X-1B-2	230X-1B-3
WSTF (2) Ident. No.	70-1671	70-1671	70-1671	70-1664	70-1664	70-1664
Atmosphere (psia)						
Oxygen	9.9	9.9	9.9	9.9	9.9	9.9
Nitrogen	6.6	6.6	6.6	6.6	6.6	6.6
Pressure (psia)						
Initial	16.5	16.5	16.5	16.5	16.5	16.5
Final	16.5	17.1	17.1	16.9	17.0	17.0
Current (amps.)						
Initial	55.	55.	55.	55±1	55.	55.
Final	80.	80.	80.	80±1	75.	80.

Furane Ident. No.	266-2B-1	266-2B-2	266-2B-3	378-1B-1	378-1B-2	378-1B-3
WSTF (2) Ident. No.	70-1665	70-1665	70-1665	70-1731	70-1731	70-1731
Atmosphere (psia)						
Oxygen	9.9	9.9	9.9	9.9	9.9	9.9
Nitrogen	6.6	6.6	6.6	6.6	6.6	6.6
Pressure (psia)						
Initial	16.5	16.5	16.5	16.5	16.5	16.5
Final	17.5	17.2	17.4	17.0	17.1	16.8
Current (amps.)						
Initial	55.	55.	55.	55.	55.	55.
Final	80.	80.	80.	70.	70.	70.

(1) NASA-MSC D-NA-0002, Test Method No. 5

(2) NASA-MSC, White Sands Test Facility

TABLE 7-3 (cont'd)

FINAL SCREENING FLAMMABILITY TEST (1)
-CONDITIONS-

Furane Ident. No.	379-1B-1	379-1B-2	379-1B-3	387-3B-1	387-3B-2	387-3B-3
WSTF (2) Ident. No.	70-1732	70-1732	70-1732	70-1745	70-1745	70-1745
Atmosphere (psia)						
Oxygen	9.9	9.9	9.9	9.9	9.9	9.9
Nitrogen	6.6	6.6	6.6	6.6	6.6	6.6
Pressure (psia)						
Initial	16.5	16.5	16.5	16.5	16.5	16.5
Final	17.1	17.1	16.9	17.2	16.9	16.5
Current (amps.)						
Initial	55.	55.	55.	55.	55.	55.
Final	70.	75.	75.	65.	70.	70.

Furane Ident. No.	437-2B-1	437-2B-2	437-2B-3	462-1B-1	462-1B-2	462-1B-3
WSTF (2) Ident. No.	71-2663	71-2663	71-2663	71-2664	71-2664	71-2664
Atmosphere (psia)						
Oxygen	9.9	9.9	9.9	9.9	9.9	9.9
Nitrogen	6.6	6.6	6.6	6.6	6.6	6.6
Pressure (psia)						
Initial	16.5	16.5	16.5	16.5	16.5	16.5
Final	17.0	17.1	17.9	17.0	17.1	17.1
Current (amps.)						
Initial	55.	55.	55.	55.	55.	55.
Final	70.	70.	75.	75.	75.	75.

(1) NASA-MSC D-NA-0002, Test Method No. 5
(2) NASA-MSC, White Sands Test Facility

The comparable key words in these two definitions are "continued"/"sustain" and "throughout." The inference is thus clearly made that "self-extinguishing" is somewhere in between the two definitions, depending solely on a) how fast the flame front disappears, and b) the amount of degradation which occurs, following removal of the ignition source. This realistic rationale is essentially reitered in the following "Criteria of Acceptability" (Ref. 6-1, Table 2-2) for this flammability test:

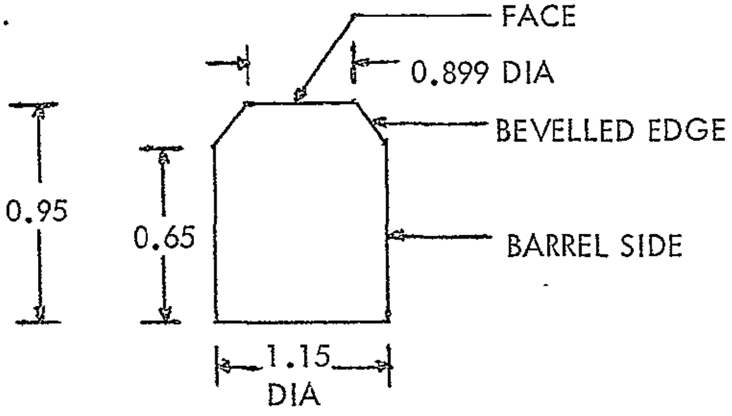
> "Potting material does not sustain combustion following removal of the ignition source with current-overloads up to the melting point of the electrical conductor entering the connector."

However, practical considerations of the test itself indicate that the removal of the ignition source (i.e. cessation of burning Teflon wire insulation) is neither readily nor precisely discernible. Hence, of necessity, we have utilized the <u>total</u> burning criteria. This obviously assumes that the ignition environment and resultant ignition (e.g. temperature, flame rate and location, specimen attitude with respect to gravity, etc.)are identical in every case. The compound burning times and the estimate of fire damaged compound are tabulated in Table 7-4. The calculations for the various surface areas listed in Table 7-4 were based upon the dimensions shown in Figure 7-1.

FIGURE 7-1

CONNECTOR POTTING: WIRE TERMINATION SIDE

(Bendix PTO6CP-18-11-P)



In addition to the above assumption involving the <u>total</u> damage, the results are even more conservative in that no consideration was given to the a) 5.2% coverage of the face surface by the eleven wires, and b) presence of potting compound on the obverse side of the connector (i.e. approximately 40-50% of the total connector potting is there, and is susceptible to burn damage if the potting compound is sufficiently flammable.)

TABLE 7-4

FINAL SCREENING FLAMMABILITY TEST

- RESULTS -

Specimen No. F-	Potting Compound Burning Time (sec.) (ea.)	(ave.)	Damaged Area (estimated) (1) Face (0.64 in. = 15.8%) (%)	(in.)	Beveled Edge (1.05 in. = 26.0%) (%)	(in.)	Barrel Side (2.35 in. = 58.2%) (%)	(in.)	Total (4.04 in. = 100.0%) (%)	(in.)	Total (ave.) (%)
175X-1B-1	80.		90.	0.576	10.	0.105	-0-	-0- (2)	0.681	16.9	
-2	130.	108.	95.	0.608	20.	0.210	-0-	-0- (2)	0.818	20.3	18.6
-3	115.		(3)	(3)	(3)	(3)	(3)	(3)	(3)	(3)	(3)
230X-1B-1	30.		50.	0.32	-0-	-0-	-0-	-0-(2)	0.32	7.9	
-2	50.	42.	50.	0.32	-0-	-0-	-0-	-0- (2)	0.32	7.9	9.2
-3	45.		75.	0.48	-0-	-0-	-0-	-0-	0.48	11.9	
266-2B-1	267.		100.	0.64	100.	1.05	100.	2.35	4.04	100. (2,4)	
-2	525.	424.	100.	0.64	100.	1.05	100.	2.35	4.04	100. (2,4)	100.
-3	480.		100.	0.64	100.	1.05	100.	2.35	4.04	100. (4,5)	
378-1B-1	80.		80.	0.512	25.	0.263	-0-	-0-	0.775	19.2 (5)	
-2	85.	83.	80.	0.512	10.	0.105	-0-	-0-	0.617	15.3 (2)	12.8
-3	85.		25.	0.158	-0-	-0-	-0-	-0-	0.158	3.9	

1. See Figure 7-1
2. Insert damage in both connector sides between center pins which carry the "fusing" current
3. Specimen at NASA-MSC
4. Considerable sub-surface degradation
5. Inserts in both connector sides burnt

TABLE 7-4 (cont'd)

FINAL SCREENING FLAMMABILITY TEST

- RESULTS -

Specimen No. F-	Potting Compound Burning Time (sec.) (ea.)	Potting Compound Burning Time (sec.) (ave.)	Damaged Area (estimated) (1) Face (0.64 in. = 15.8%) (%)	Face (0.64 in. = 15.8%) (in.)	Beveled Edge (1.05 in. = 26.0%) (%)	Beveled Edge (1.05 in. = 26.0%) (in.)	Barrel Side (2.35 in. = 58.2%) (%)	Barrel Side (2.35 in. = 58.2%) (in.)	Total (4.04 in. = 100.0%) (%)	Total (4.04 in. = 100.0%) (in.)	Total (ave.) (%)
379-1B-1	95.		30.	0.192	-0-	-0-	-0-	-0-	0.192	4.8 (2)	
-2	0.	32.	20.	0.128	-0-	-0-	-0-	-0-	0.128	3.2 (2)	3.5
-3	0.		15.	0.096	-0-	-0-	-0-	-0-	0.096	2.4	
387-3B-1	37.		45.	0.286	10.	0.105	-0-	-0-	0.391	9.7	
-2	32.	48.	75.	0.48	-0-	-0-	-0-	-0-	0.48	11.9 (2)	12.4
-3	75.		90.	0.576	5.	0.053	-0-	-0-	0.629	15.6 (2)	
437-2B-1	55.		95.	0.608	35.	0.368	-0-	-0-	0.976	24.2	
-2	100.	169.	100.	0.64	40.	0.42	3.	0.071	1.131	28.1 (2)	50.8
-3	353.		100.	0.64	100.	1.05	100.	2.35	4.04	100. (2)	
462-1B-1	(6) 155.		95.	0.608	30.	0.315	-0-	-0-	0.923	22.8 (2)	
-2	(7) 166.	159.	95.	0.608	20.	0.21	-0-	-0-	0.818	20.3 (2)	21.1
-3	155.		95.	0.608	20.	0.21	-0-	-0-	0.818	20.3 (2)	

6. Potting material ignited and extinguished 3 times during this period.
7. " " " " " 2 " " " "

More precisely stated, the various aspects of the (arbitrary) selection criteria were as follows:

1. Pyrolysis degradation, if present, must be essentially limited to the surface.

2. Pyrolysis degraded surfaces (i.e. "Damaged Area" of Table 7-4) must not exceed 50%, average, or 75%, maximum, of the potentially degradable area, for a minimum of three specimens.

3. Burning time must not exceed 60 seconds, average, or 90 seconds, maximum, for a minimum of three specimens.

A summary of the results is presented in Table 7-5 and is also graphically shown in Figure 7-2.

TABLE 7-5

FINAL SCREENING FLAMMABILITY TEST
-RESULTS-
(SUMMARY) (1,4)

Formulation No. F-	Primary Degradation		Damaged Area (%) (2) Average Value	P/F	Damaged Area (%) (2) Maximum Value	P/F	Burning Time (sec.) (2) Average Value	P/F	Burning Time (sec.) (2) Maximum Value	P/F
		P/F								
175X	Surface (3)	P	18.6(3)	P	20.3 (3)	P	108.	F	130.	F
230X	"	P	9.2	P	11.9	P	42.	P	50.	P
266	Interior	F	100.	F	100.	F	424.	F	525.	F
378	Surface	P	12.8	P	19.2	P	83.	F	85.	P
379	"	P	3.5	P	4.8	P	32.	P	95.	F
387	"	P	12.4	P	15.6	P	48.	P	75.	P
437	"	P	50.8	F	100.	F	169.	F	353.	F
462	"	P	21.1	P	22.8	P	159.	F	166.	F

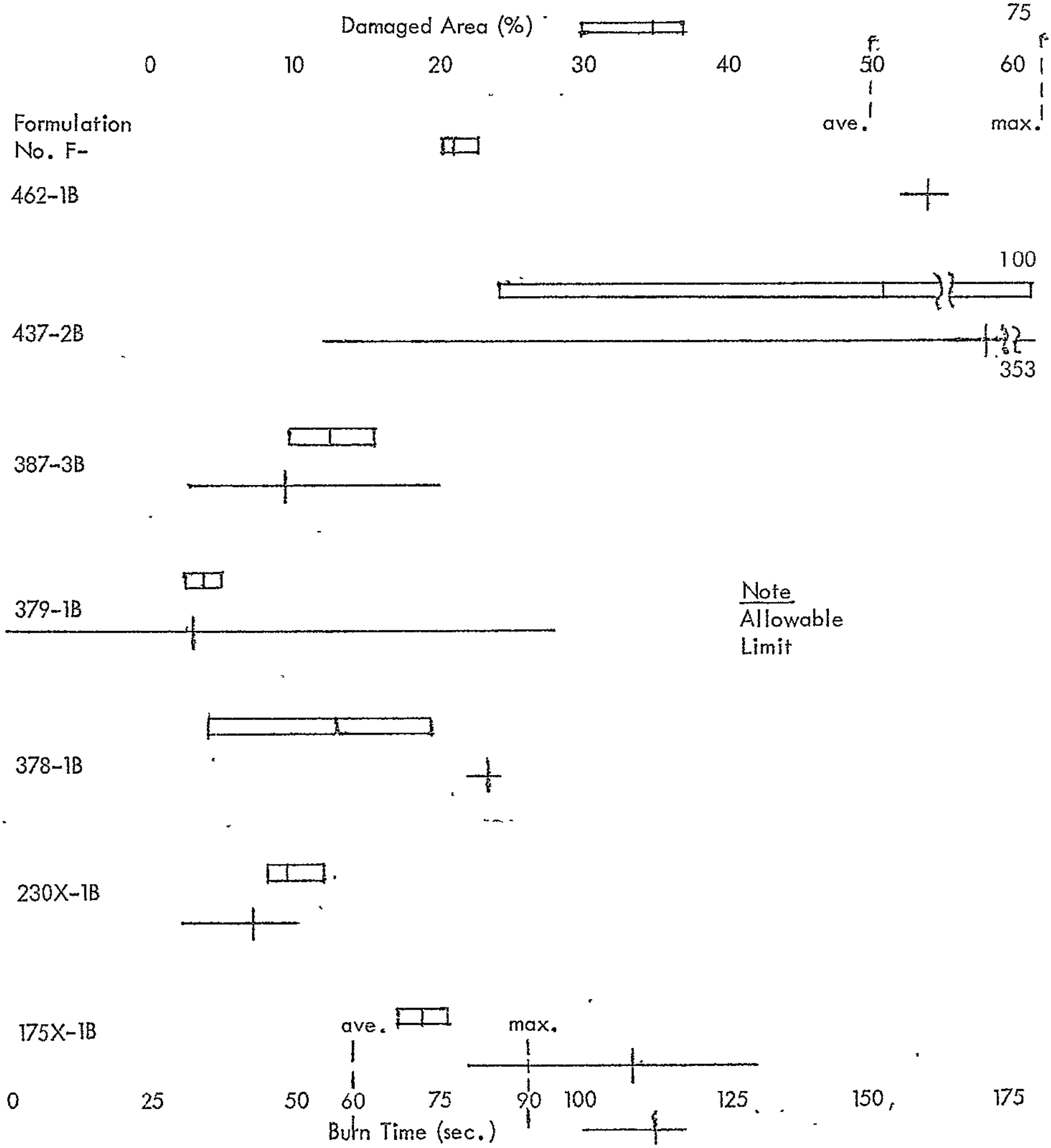
(1) See Table 7-4.
(2) Three specimens.
(3) Two specimens.
(4) P.....Pass F.....Fail

Based upon the above criteria, the flammability resistance of these formulations was considered to be in the following order:

F-379 > -230X > -387 > -378 > -175X > -462 > -437 >> -266

FIGURE 7-2

FINAL SCREENING FLAMMABILITY TEST
-RESULTS-
(Summary)



However, it was necessary to also consider such ancillary factors as:

a) All three of the obverse plugs of F-230X, and two of the three obverse plugs of F-379 were loose. Their poor adhesion is no doubt attributable to the Kryrox 143 AC in their formulations.
b) Two of the three obverse plugs of F-462-1B were loose. It was conjectured that some form of inhibition was involved since it was quite difficult to cure a hexabromobenzene/RTV 655 system (F-036) (Ref. 6-10, Section 3, page 30).
c) Significant soot was noticed with F-437-2B (two out of three) and F-175X-1B (three out of three). One specimen of the latter also "dripped" during the test and one specimen had a loose obverse plug.

Solely from these results, F-378 and F-387 were preferred formulations.

7.2.2 Carbon Monoxide/Total Organics

Unless otherwise noted, the compounds for this test were cast between methyl ethyl ketone-washed Mylar sheets which were backed up with heat resistant glass, to form 1/8" x 6.0" x 6.0" sheets. Dam material (3 sides only) was natural rubber tubing supported by brass wire. Spring clips were used to provide the pressure. The same technique was utilized for all sheet material required for the Program. The test conditions and results are shown in Table 7-6.

As specified in the Statement of Work (Ref. Appendix A, Section 3.4.2), the carbon-monoxide can not exceed 25 micrograms per gram of sample, and the total organics can not exceed 100 micrograms per gram of sample after exposure to 155°F for 72 hours. Based upon these requirements, the results of Table 7-5 have been summarized in Table 7-7 in ascending order of contaminant content.

TABLE 7-7

CO/TO: SUMMARY OF RESULTS

Contaminant (μ gm/gm)

	Passed		Not Passed	
	155°F	200°F	155°F	200°F
CO	378-3(1):0.3	266-1A:0.3		
	379-3(2):0.3	378-1A:0.4		
	387-6A:0.4	230X-1A:0.6		
	437-2A:0.4	379-1A:0.6		
	462-1A:0.4	387-3A:0.9		
	266-4(1):0.5	266-1A(2):1.1		
	387-7C:0.7	175X-1A:1.9		

TABLE 7-7 (cont'd)

CO/TO: SUMMARY OF RESULTS

Contaminant (μ gm/gm)

	Passed		Not Passed	
	155°F	200°F	155°F	200°F
TO	379-3(1):1.2	387-3A:19.	266-4(1):226.	266-1A(2):111.
	387-7C:1.5	230X-1A:35.		379-1A:124.
	387-6A:3.4	175X-1A:63.		378-1A:196.
	437-2A:5.7			266-1A:276.
	378-2(1):6.3			
	462-1A:8.6			

1. Aluminum dish mold.
2. Rerun.

In addition, identification of major infrared spectroscopy peaks, via 10 meter infrared and gas chromatographic/mass spectrographic scans, was to be carried out. Also "...none of the outgassed products shall constitute a toxic hazard to the crew, based upon the criteria of acceptability of Test No. 6, Appendix A" (Ref. Appendix C). It is seen from Table 7-6 that this aspect of the analysis was not investigated in F-175X-1A, -230X-1A, or -266-1A (original and rerun). The base polymer in F-175X and F-230X is no longer available. F-266 was analyzed in batch No. 4 (i.e. F-266-4). The remaining formulations were analyzed in this regard and the results are noted in the table, with F-266-4 being the only unsatisfactory one.

It should be noted at this point that it was difficult to rationally reconcile some of the date in Table 7-6. For example, F-175X-1A, -230X-1A, -378-1A, and -379-1A, were all tested at 200°F. The polymer in F-175X-1A and F-230X-1A, 77-024, contained carbon black, whereas the related polymer in F-378-1A and F-379-1A, 77-033, did not. Further, the cure cycles (see Table 7-2) for the two 77-024 formulations were significantly shorter than for the other two. However, comparing the four compounds, we see that F-175X-1A and F-230X-1A have significantly less total organics than the other two, and that the carbon monoxide is essentially the same in all cases, except for F-175X-1A (i.e. approximately 300% more). These particular disparities are of interest since the total organics values for F-378-1A and F-379-1A exceed the acceptable limits at 200°F, but their carbon monoxide values are the lowest at 155°F and are still quite low at 200°F.

7.2.3 Odor

The criterion for this test is specified in the Statement of Work (Ref. Appendix A, Section 3.4.3), the value of which "...must not exceed 25, for the sum of 10 odor

TABLE 7-6

CARBON MONOXIDE AND TOTAL ORGANICS (1)
-CONDITIONS/RESULTS-

Furane Ident. No.	175X-1A	230X-1A	266-4	266-1A	266-1A (3)	378-3	378-1A
WSTF (2) Ident. No.	70-1671	70-1664	71-2583	70-1665	70-1665	71-2584	70-1731
Initial wt. (gm.)	20.0572	20.0629	20.0354	20.0025	10.0094	20.0114	20.0637
Test Chamber Vol. (liters)	4	4	4	4	2	4	4
Exposure							
Time (hours)	72	72	72	72	72	72	72
Temperature (°F) ± 5°F	200	200	155	200	200	155	200
Weight Loss (gm./gm.)	0.0054	0.0113	0.0016	0.0029	0.0023	0.0002	0.0033
Weight Loss (%)	0.54	1.13	0.16	0.29	0.23	0.02	0.33
Carbon Monoxide (μgm./gm.)	1.9	0.6	0.5	0.3	1.1	0.3	0.4
Total Organics (μgm./gm.)	63.0	35.0	226.0 (4)	276.0	111.0	6.3 (5)	196.0 (7)

Furane Ident. No.	379-3	379-1A	387-6A	387-7C	387-3A	437-2A	462-1A
WSTF (2) Ident. No.	71-2585	70-1732	71-2586	71-2587	70-1745	71-2665	71-2666
Initial wt. (gm.)	20.0228	20.0155	20.0803	10.0888	20.0126	20.1311	20.0722
Test Chember Vol. (liters)	4	4	4	2	4	4	4
Exposure							
Time (hours)	72	72	72	72	72	72	72
Temperature (°F) ± 5°F	155	200	155	155	200	155	155
Weight Loss (gm./gm.)	0.0006	0.0070	0.0009	0.0012	0.0016	0.0006	0.0006
Weight Loss (%)	0.06	0.70	0.09	0.12	0.16	0.06	0.06
Carbon Monoxide (μgm./gm.)	0.3	0.6	0.4	0.7	0.9	0.4	0.4
Total Organics (μgm./gm.)	1.2 (6)	124.0 (7)	3.4 (5)	1.5 (5)	19.0 (7)	5.7 (5)	8.6 (5)

(1) NASA-MSC D-NA-0002, Test Method No. 6
(2) NASA-MSC, White Sands Test Facility
(3) Rerun
(4) More than 10μgm./gm. 1-fluoro-1-chloroethyl benzene, aliphatic chloro compound (via 10 m. infrared and GC-MS scans)
(5) Less than 10μgm./gm. toluene (via 10 m. infrared and GC-MS scans)
(6) No significant peaks (" " " " " " " ")
(7) No significant peaks (" " " " scan)

TABLE 7-8

ODOR TEST (1)
-CONDITIONS/RESULTS-

Furane Ident. No.	175X-1A	230X-1A	266-4	266-2A	378-3	378-1A	379-3
WSTF (2) Ident. No.	70-1671	70-1664	71-2583	70-1665	71-2584	70-1731	71-2585
Material per Liter (gm.)	5.069	5.057	5.100	5.063	5.042	5.053	5.008
Test Chamber Vol. (liters)	6	6	6	6	6	6	6
Exposure							
Time (hours)	72	72	72	72	72	72	72
Temperature (°F) ± 5°F	200	200	155	200	155	200	155
Dilution with Oxygen	(3)		(4)			(5)	
1:29	0.0	0.5	0.1	0.3	0.3	0.1	0.2
1:9	0.1	1.1	0.1	0.2	0.3	0.0	0.5
1:1	1.4	1.9	1.5	0.8	1.5	0.5	1.2

Furane Ident. No.	379-1A	387-6A	387-7C	387-3A	437-2A	462-1A
WSTF (2) Ident. No.	70-1732	71-2586	71-2587	70-1745	71-2665	71-2666
Material per Liter (gm.)	5.014	5.016	5.0444	5.012	5.006	5.003
Test Chamber Vol. (liters)	6	6	2	6	6	6
Exposure						
Time (hours)	72	72	72	72	72	72
Temperature (°F) ± 5°F	200 (6)	155	155	200	155	155
Dilution with Oxygen						
1:29	0.0	0.1	0.4	0.1	0.1	0.0
1:9	0.1	0.2	0.5	0.3	0.3	0.7
1:1	0.5	0.9	1.6	2.1	1.3	1.7

(1) NASA-MSC D-NA-0002, Test Method No. 7 (average of 10 member panels)

(2) NASA-MSC, White Sands Test Facility

(3) Diluted 1050 ml concentrate with O_2 in 2. liter desiccator to meet WSTF 620 ppm safety requirement

(4) Diluted 395 ml concentrate with O_2 in 2. liter desiccator to meet WSTF 620 ppm safety requirement

(5) Diluted 340 ml concentrate with O_2 in 2. liter desiccator to meet " " " " "

(6) Diluted 430 ml concentrate with O_2 in 2. liter desiccator to meet " " " " "

evaluations..." (or 2.5 for the average of a 10 member panel). In addition to the required exposure at 155°F, some of the compounds were also exposed to 200°F prior to test. Table 7-8 tabulates the test conditions and final results. It is seen that some of the formulations apparently outgassed too greatly to use as is and were therefore diluted to the extent noted in the table, prior to test. A summary of the 1:1 ratio dilution results for the "tested" and for the "actual" conditions is shown in Table 7-9, in the ascending order of "actual" values at 155°F and 300°F. The "actual" values are based upon the "tesred" values and any pre-dilution factor. This, of course, assumes a proportional effect.

TABLE 7-9

ODOR: SUMMARY OF RESULTS

Formulation	Tested			Actual (calc.)	
No. F-	155°F	200°F		155°F	200°F
266-2A	---	0.8	pass	---	0.8
387-6A	0.9	---		0.9	---
379-3	1.2	---		1.2	---
437-2A	1.3	---		1.3	---
378-3	1.5	---		1.5	---
387-7C	1.6	---		1.6	---
462-1A	1.7	---		1.7	---
230X-1A	---	1.9		---	1.9
387-3A	---	2.1		---	2.1
379-1A	---	0.5		---	2.3
175X-1A	---	1.4	fail	---	2.7
378-1A	---	0.5		---	3.0
266-4	1.5	---		7.7	---

In three out of the four cases where tests were run at both 155 and 200°F, the expected increase at the higher temperature is noted. However, F-266 shows the reverse. Calculations also indicate this same trend for the other two dilutions for this formulation. Peculiarities with this formulation were also present in the data for CO/TO (see Tables 7-6 and 7-7).

Discounting the anomalies of F-266, we see that F-387-6A has the lowest average value. But we find that although F-387-7C (i.e. another batch of the same formulation) also passes the requirement, it has a test value of almost twice that of F-387-6A. Again, referring to the CO/TO values in Tables 7-6 and 7-7, it is seen that F-387-6A has over twice the total organics and approximately half the carbon monoxide of F-387-7C.

7.2.4 Thermal Analysis: TGA/DTA

The conditions utilized for the determination of the thermogravimetric analysis (TGA) and differential thermal analysis (DTA) of the compounds which were used for the final screening, and the ensuing data, are tabulated in Table 7-10. Upon examination of the data traces in conjunction with the flammability results, the first 600°F appeared to be the most significant. Specifically, endotherms below 400°F seemed to strongly indicate chemical changes which affect the flammability resistance of these compounds.

The cause for the weight loss under partial vacuum (and no heat) of F-387-1A is unknown, and is especially inexplicable since no such loss was ascertained in F-437-2A. Nevertheless, it should be noted that the total weight loss up to 600°F was only 8.4%. In comparison, reference is made to an original, but now obsolete, requirement for this program that, with respect to the specimens, "Not more than 10% shall outgas or pyrolyze below this (600°F) temperature."

Although the endotherm for F-387-1A occurs at 374°F, the trace shows only approximately 1.0 mg (i.e. 0.35%) weight loss at 493°F. It is 10. mg (i.e. 3.5%) at approximately 570°F.

7.2.5 Vacuum Outgassing

The formulations evaluated for outgassing while exposed to the combined environment of vacuum and heat, are listed in Table 7-11 with their test conditions and their respective results. As the various final screening test results were analyzed, it became evident that F-387 was of primary interest. Thus, when the property requirements in the Statement of Work (Ref. Appendix A) were amended to reflect limitations in cure condition requirements (i.e. 150°F, maximum), F-387 was retested at 150°F while exposed to vacuum (F-387-8). The weight loss and maximum loss rate were higher than anticipated, considering the polymer's and flame retardant's respective known characteristics. For example, Dow Corning states that (Ref. 7-2) at 212°F/24 hours/1 x 10^{-6} torr, there is normally less than approximately 3.% weight loss; such loss being primarily due to low molecular weight cyclic material. They also maintain that their "special cleaning" procedures can achieve a weight loss of 0.3-0.4%, with only 0.03% condensables. The Dechlorane 604 is reported (Ref. 7-3) to have a 354°F melting point, and a vapor pressure at 282.2°F of 0.0014 torr.

Specimens of the flame retardant, Dechlorane 604, and of the compound, F-387-3A, were then subjected to the following investigation:

> Each specimen was divided into two approximately equal portions and weighted. One portion of each material type was then preconditioned at 212°F. for four hours, minimum. The two dried portions were then weighed and kept in a desiccator when not in a vacuum or being weighed. The other two portions were tested in the "as is" state.

TABLE 7-10

THERMAL ANALYSIS
-CONDITIONS/RESULTS-

Furane Ident No.	175X-1A	230X-1A	266-2A	378-1A
WSTF (1) Ident. No.	70-1671	70-1664	70-1665	70-1731
	(2)	(3)	(4)	(5)
Oxygen (psia)	6.2	6.2	6.2	6.2
Initial Wt. (mg.)	94.9	140.0	84.3	199.5
Heating Rate (°F/min.)	18.	18.	18.	18.
Test Temp. Max. (°F)	941.	932.	932.	599.
Weight Loss (mg.)	58.7	87.6	91.8	55.4
Weight Loss (%)	61.9	62.6	97.3	27.8
Temp. Max. Wt. Change (°F)	761.	770.	680.	356.
Temp. Max. Exotherm (°F)	770.	788.	none	>599.
Temp. Max. Endotherm (°F)	374.	~374.	680.	392.
Temp. Initial Wt. Loss (°F)	212.	212.	374.	176.
Temp. Initial Decomp. (°F)	266.	302.	482.	230.

Furane Ident. No.	379-1A	387-1A	437-2A	462-1A
WSTF (1) Ident. No.	70-1732	70-1745	71-2665	71-2666
	(5)			(9)
Oxygen (psia)	6.2	6.2	6.2	6.2
Initial Wt. (mg.)	231.0	285.0	377.1	464.2
Heating Rate (°F/min.)	18.	18.	18.	18.
Test Temp. Max. (°F)	599.	599.	599.	599.
Weight Loss (mg.)	55.9	24.1 (5)	17.3	12.6
Weight Loss (%)	24.2	8.4	4.9	2.7
Temp. Max. Wt. Change (°F)	356.	599.	>599.	>599.
Temp. Max. Exotherm (°F)	>599.	>599.	>599.	~536.
Temp. Max. Endotherm (°F)	374.	374.	374.	none
Temp. Initial Wt. Loss (°F)	176.	(6)	266.	266.
Temp. Initial Decomp. (°F)	266.	410. (7)	473. (8)	554.

TABLE 7-10 (cont'd)

THERMAL ANALYSIS
-CONDITIONS/RESULTS-

Notes:

1. NASA-MSC, White Sands Test Facility
2. Sample was shrunk and hard at test termination (941°F)
3. Residue was a hard cake, gray in color, and friable.
4. At approximately 626°F, the crucible was obscured by a cloud of white off-gas material and coated the inside of the furnace tube. No notable sample remained at test termination (932°F).
5. Residue was firmer and less yielding to touch than original specimen.
6. Sample lost 10 mg. under partial vacuum without application of heat.
7. Sample started to char. Residue was charred and fused.
8. " " " " . Sample was charred black at 554°F. Residue was soft, sticky, and spongy. Heavy deposit of white elongated crystals was left on inside of furnace tube.
9. No apparent change in sample.

TABLE 7-11

VACUUM OUTGASSING
-CONDITIONS/RESULTS-

	Weight							
Formulation No. F-	Initial (mg)	After Post Cure (1) (mg)	Loss After Post Cure (mg)	Loss After Post Cure (%)	After Conditioning (2) (mg)	Loss After Conditioning (2) (mg)	Loss After Conditioning (2) (%)	Conditioning Loss Rate, Max. (%/hr)
305-1 (4,8)	16.70	---	---	---	11.5	5.20	31.1	10.6
307-3A (5,8)	106.34	106.13 (7)	0.21 (7)	0.20 (7)	77.87	28.26	26.63	3.76
175X-1A	96.78	87.08	9.70	10.02	68.82	18.26	20.97	9.2
230X-1D (6)	96.00	---	---	---	72.28	23.72	24.71	23.1
266-2A	97.46	96.64	0.82	0.84	92.70	3.94	4.08	0.47
378-1 (6)	94.93	---	---	---	70.80	24.13	25.42	32.9
379-1 (6)	94.84	---	---	---	71.30	23.54	24.82	26.0
387-3 (6)	97.30	---	---	---	47.29	50.01	51.40	25.0
387-8 (6)	88.54	87.17	1.37	1.55 (9)	84.81 (3,10)	2.41 (3)	2.44 (3)	0.16 (3)

1. 8 hrs./150°F/10^{-4} - 10^{-5} torr
2. 24 hrs./250°F/10^{-4} - 10^{-5} torr
3. 24 hrs./150°F/10^{-4} - 10^{-5} torr
4. "Strip" mold specimen
5. 0.050" sheet
6. 2.4" ID mold (standard laboratory alum. dish)
7. 64 hrs./77°F/10^{-5} torr
8. NASA-MSC-12SKI formulation.
9. Initial weight loss rate = 0.34%/hr.
10. After post cure, specimen was allowed to reach ambient conditions over a 15 hr period. "Initial" value at start of conditioning = 87.22 mg.

The four portions were weighed and exposed to 10^{-4} torr vacuum at ambient temperature for sufficient time to reach a "steady" state condition. The portions were then reweighed (with minimum air exposure). They were next subjected to 155 ± 5°F and 10^{-4} torr vacuum for 24 hours. After cooling under vacuum, they were then reweighed.

The resultant data is tabulated in Table 7-12. (The raw data is included to clarify conditioning cycles and the respective computations.) Upon examination of the data, the following several observations appear reasonable and valid:

a. The cured polymer has almost an order of magnitude greater percent loss in weight than the filler, Dechlorane 604, at 212°F. This might have been due either to adsorbed moisture on the polymer, or a very low molecular weight polymer fraction.
b. The ambient temperature vacuum data also indicates that the slight D-604 weight loss and the more significant loss by the cured compound is probably adsorbed moisture.
c. The vacuum/thermal data, taken in context with the previous data, suggests that although there appears to be no loss of D-604 at either 212°F or in vacuum alone, a very slight amount of D-604 (i.e. approximately 0.035% per 25 1/2 hrs.) is lost in the combined environment. This data also further confirms that the higher loss by the cured compound is apparently only due to adsorbed moisture and not to polymer fractions.

Based upon the above, it was felt that the "loss after conditioning" and the "maximum conditioning loss rate" for F-387-8, shown in Table 7-11, were excessive. Subsequent review of the processing data showed that the formulation for this test was molded in a standard laboratory aluminum dish in the "as received" condition. It was then conjectured that these dishes were die stamped with the aid of a mold lubricant. Such a lubricant could range from a simple natural fat to more sophisticated types as silicone oils and/or greases. These lubricants could readily cause abnormally high readings by transfer from the die to the aluminum cup, and then to the molding with:

a. volatilization then taking place in the combined environment, and/or
b. Inhibition of the polymer cure occurring. This could conceivably cause non-crosslinked low molecular weight polymer fractions to be present during the exposure to the combined environment.

In order to determine the validity of these suppositions, part of a trimmed-away section of a molded insulation resistance "reworked" specimen was carefully tested at 150°F/10^{-4} - 10^{-5} torr. The mold for these specimens is made of aluminum and steel with steel bolts. All of the formulation contacting surfaces of the mold have skived Teflon tape as a release agent; said tape being secured to the metal substrates with a silicone pressure sensitive adhesive. Final cleaning of the mold consists of mechanical removal of prior molding's residues and a careful wiping of all mold

TABLE 7-12
VACUUM OUTGASSING
-ENVIRONMENTAL EFFECTS-

Test Conditions Or Weight Identification	Calculations: Weight or Weight Loss			
	DECHLORANE 604		Formulation 387-3A	
	Predried	No Predry	Predried	No Predry
Initial Wt. (gm)	4.2215	4.5131	1.2305	1.0245
P.C. 'A' Wt. (gm)	4.2196	---	1.2261	---
Wt. Loss (gm)	0.0019	---	0.0044	---
Wt. Loss (%)	0.045	---	0.358	---
Initial Wt. (gm)	4.2215	4.5131	1.2305	1.0245
P.C. 'B' Wt. (gm)	4.2202	4.5116	1.2302	1.0227
Wt. Loss (gm)	0.0013	0.0015	0.0003	0.0018
Wt. Loss (%)	0.031	0.033	0.024	0.180
P.C. 'A' Wt. (gm)	4.2196	---	1.2261	---
P.C. 'B' Wt. (gm)	4.2202	4.5116	1.2302	1.0227
Wt. Loss (gm)	[0.0006]	---	[0.0041]	---
Wt. Loss (%)	[0.014]	---	[0.335]	---
Initial Wt. (gm)	4.2215	4.5131	1.2305	1.0245
P.C. 'C' Wt. (gm)	4.2185	4.5099	1.2259	1.0209
Wt. Loss (gm)	0.0030	0.0032	0.0046	0.0036
Wt. Loss (%)	0.071	0.071	0.374	0.359
P.C. 'A' Wt. (gm)	4.2196	---	1.2261	---
P.C. 'C' Wt. (gm)	4.2185	4.5099	1.2259	1.0209
Wt. Loss (gm)	0.0011	---	0.0002	---
Wt. Loss (%)	0.026	---	0.016	---
P.C. 'B' Wt. (gm)	4.2202	4.5116	1.2302	1.0227
P.C. 'C' Wt. (gm)	4.2185	4.5099	1.2259	1.0209
Wt. Loss (gm)	0.0017	0.0017	0.0043	0.0018
Wt. Loss (%)	0.040	0.038	0.350	0.180

Post Condition 'A': 4 1/4 hr/212°F.
" " 'B': Ambient temp./10 min/1 x 10^{-4} torr.
" " 'C': 155 ± 5°F/25 1/2 hr/ 8 x 10^{-5} torr.

surfaces with methyl ethyl ketone. A 15 minute bake of each cleaned mold part at 150°F completes the process. It was therefore felt that the F-387-8C trimmings were reasonably uncontaminated. It should be noted that the designation F-387-8 and -8C indicate that both were not only from the same formulation, but also from the same batch and cured identically at the same time.

The conditions and results of this test are shown in Table 7-13. (Again, raw data are included to facilitate a better understanding of the results.) Some pertinent comments are:

a. The 24 hour combined environment weight loss percentages for the cured compounds shown in Tables 7-12 and 7-13 are quite consistent (i.e. 0.374 vs. 0.71). The somewhat higher value for F-387-8C could be attributable to the fact that the weighings for Table 7-12 were carried out in air, whereas those for Table 7-13 were conducted with an in-situ microbalance. The former was obviously more subject to moisture pick-up during the weighing process.

b. Without resorting to the allowable (Ref. Appendix A, Section 3.3.4) post cure of 8 hours at 150°F, F-387 meets the requirement for a 1.0% maximum weight loss for 24 hours, and barely (i.e. 0.12%/hr) falls outside of the requirement for 0.1% per hour maximum loss rate for 24 hours. If the post cure is utilized, it is seen that this formulation easily meets both requirements. In fact, the conservatively calculated data for a specimen which had been post cured and then exposed to <u>48</u> hours of the combined environment shows that the <u>total</u> percentage of weight loss, as well as the maximum weight loss <u>rate</u>, would still comply with the "1.0% maximum weight loss for <u>24</u> hours" requirement (i.e. 0.99% and 0.025%, respectively).

c. It is assumed that the rapidly declining loss rate from the initial high value of 0.12% per hour is, as noted above, primarily due to the immediate loss of adsorbed water.

d. The rate of weight loss decreased with exposure time.

e. There was a <u>moderate</u> amount of white deposit or condensate left on the walls of the glass hangdown tube when F-387-8 was run. The temperature of the glass at the point where the condensate began was approximately 120°F. A sample of the deposit was found to have a melting point of 356-360°F. This indicated that the deposit was D-604. Only a <u>slight</u> amount of deposit was detected in the F-387-8C run.

7.2.6 Insulation Resistance

The results of a preliminary evaluation of the electrical properties of the formulations which were given a final screening are shown in Table 7-14. The results showed that of the formulas evaluated in this screening phase, those primarily dependent upon organo-halogen compounds as the flame retardant, readily passed the insulation resistance test, and had the best insulation resistance. The rest either marginally passed, marginally failed, or clearly failed.

TABLE 7-13
VACUUM OUTGASSING
-CONDITIONS/RESULTS-
(Formulation 387-8C) (1, 2)

Time	Weight	Weight Loss		Weight Loss Rate	Weight Loss Rate, Max.
(hrs)	(mg)	(mg)	(%)	(%/hr) (4)	(%/hr)
0	95.780	-0-	-0-	0.12	↑
8	95.466	0.314	0.33 (3)	0.025	
24	95.104	0.676	0.71 (3)	0.022	
32	94.931	0.849	0.89 (3)	0.019	0.12
48	94.657	1.123	1.17 (3)	0.017	
56 (5)	~94.521	~1.259	~1.32	<0.017	↓
8-32	---	0.535	0.56 (6)	---	0.025
8-48	---	0.809	0.85 (6)	---	0.025
48-56 (5)	---	~0.136	~0.14 (6)	---	0.017
8-56 (5)	---	~ 0.945	~0.99 (6)	---	0.025
24-48	---	0.447	0.46 (6)	---	0.022

1. Part of trimmed-away section of insulation resistance "reworked" specimen.
2. 150°F/10^{-4} - 10^{-5} torr.
3. Based on 95.780 mg.
4. Tangent value at hour indicated; indicated hour weight taken as base weight.
5. Conservatively calculated from weight at T=48 hours, and weight loss rate at T=48 hours.
6. Based on initial weight of measurement period.

50

TABLE 7-14

INSULATION RESISTANCE (1,2)

Formulation No. F-	Results (3) ($\times 10^4$ megohms) Min.	Max.	Ave.
462-2	770.	950.	890.
437-4	410.	540.	470.
387-12C (4,5)	400.	490.	460.
387-6C (4,5)	220.	270.	240.
387-4 (4,5)	88.	100.	94.
230X-2	11.	13.	12.
Requirement (6)	10.	---	---
387-2	9.6	12.	11.
379-2	9.3	17.	13.
175X-3	3.7	4.9	4.4
266-3	0.35	0.38	0.36

1. Tested at 77-79°F/51% relative humidity.
2. Electrification time: 1-2 minutes. Applied voltage: 500 VDC.
3. Single specimen data; 4 readings per specimen.
4. Tested at 75°F/53% R.H.
5. Reported also for Final Characterization (see Table 8-5)
6. Ref. Appendix A, Table I.

SECTION 8

NONFLAMMABLE FORMULATIONS DEVELOPMENT: PRODUCT CHARACTERIZATION

8.1 FORMULATION SELECTION

A summary of the Final Screening Tests are tabulated in Table 8-1. The general superiority of F-387 is noticeable, and was therefore selected for the complete product characterization.

8.2 FORMULATIONS/PROCESSING

The compositions of the batches which were prepared for the final screening and product characterization of F-387, are defined in Table 8-2. The associated processing data is tabulated in Table 8-3.

8.3 EVALUATION

Table 8-4 is provided herein in order to clarify the genesis of the individual test specimens with respect to the applicable specific batch and cure cycle.

8.3.1 Thermal Properties

The so-called "piggy back" test (Ref. Appendix A, Section 3.4.1.2) was the only flammability test not heretofore conducted. The test was carried out with the two potted connectors at their closest positions, with one directly above the other. The ignition squib was placed immediately below and against the bottom surface of the bottom connector on the wire termination sides of the two connectors. The test environment was 16.5 ± 0.1 psia, 60% oxygen/40% nitrogen.

At test initiation (T_o) an initial current of 5 amperes was applied to the nichrome wire igniter coil. At T_o plus 30 seconds this current was increased to 10 amperes. Smoke was first noted emitting from the silicone igniter at T_o plus 41 seconds; ignition of the silicone igniter occurred at T_o plus 43 seconds. Flames from the silicone igniter enveloped the lower connector and large amounts of black smoke were noted; the smoke increased in intensity until the flames extinguished. At T_o plus 60 seconds the current to the nichrome wire igniter coil was increased to 15 amperes; wire failure occurred at T_o plus 85 seconds. Any evidence of flame was not visible at T_o plus 95 seconds. The final chamber pressure was 16.7 psia.

The acceptance criteria for the material (Ref. Appendix A, para. 3.4.1.2) is either that the lower connector fails to ignite or if the lower connector burns, it must not ignite the upper connector. Hence, F-387 passed since the upper connector did not ignite.

TABLE 8-1
FINAL SCREENING RESULTS
- SUMMARY (1) -

	Formulations							
Test	175X	230X	266	378	379	387	437	462
Flammability	F	F	F	P	F	P	F	F
CO/TO (155°F)	P	P	F	P	P	P	P	P
" " (200°F)	P	P	F	F	F	P	---	---
Odor (155°F)	---	---	F	P	P	P	P	P
" (200°F)	F	P	P	F	P	P	---	---
TGA (2)	3/6	3/5	1/2	4/7	4/6	-/4	2/3	2/1
Vac. Outgassing (150°F)	---	---	---	---	---	P	---	---
" " (250°F) (3)	2	3	1	5	4	6	---	---
Insul. Resistance	F	P	F	F	F	P	P	P

P.....Pass
F.....Fail
1. Based upon "criteria" (Page 7-9) and "ancillary factors" (Page 7-11).
2. Based on temperature of initial weight loss and initial decomposition:
    1.....Highest
    8.....Lowest
3. Based on % total wt. lost and max. rate of loss:
    1.....Best
    8.....Worst

TABLE 8-2

FLUOROSILICONE RTV'S WITH ADDITIVE

(For Final Screening and Characterization)

Component	S	387-3	387-4	461-1	387-5	387-6	387-7	387-8	461-2	387-9	387-10
		Formulations (phr)									
77-033	12	100.	100.								
77-033 (1)	12			100.					100.		
DECHLORANE 604	16	150.	150.								
DECHLORANE 604 (2)	16			150.					75.		
DECHLORANE 604 (3)	16								75.		
461-1	13				250.	250.	250.	250.		250.	
461-2	13										250.
77-033 Catalyst	12	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.

S......Source (See Table 6-1)
1. De-aerated at 29-30 in. Hg./2 hrs.
2. 100-170 mesh
3. 100% through 170 mesh

TABLE 8-2(cont'd)

FLUOROSILICONE RTV'S WITH ADDITIVE
(For Final Screening and Characterization)

Formulations (phr)

Component	S	461-3	387-11	387-12	387-13	(4) 387-14	(5) 387-15	(6) 387-16	387-17	387-18
77-033 (1)	12	100.								
DECHLORANE 604 (2)	16	75.								
DECHLORANE 604 (3)	16	75.								
461-2	13		250.							
461-3	13			250.	250.	250.	250.	250.	250.	250.
77-033 Catalyst	12		10.	10.	10.	10.	10.	10.	10.	10.

S......Source (See Table 6-1)
4. Half of specimen is 387-7C (Repair Test)
5. Half of specimen is 387-8C (Repair Test)
6. Half of specimen is 387-9C (Repair Test)

TABLE 8-3

FLUOROSILICONE RTV'S WITH ADDITIVE
(For Final Screening and Characterization)
- FORMULATION PROCESSING -

	Cure (1)		Post Cure		Total
Formulation No. F-	Time (hrs/min)	Temp. (°F)	Time (hrs/min)	Temp. (°F)	Time @ 150°F (hr/min)
387-3	12/50	150	---	---	12/50
-4	1/50	"	14/45	150	16/35
-5	9/20	"	2/40	"	12/-
-6	7/32	"	4/28	"	12/-
-7	6/20	"	5/50	"	12/10
-8	15/30	"	---	---	15/30
-9	12/38	"	---	---	12/38
-10	12/-	"	---	---	12/-
-11	6/-	"	7/15	150	13/15
-12	9/40	"	2/20	"	12/-
-13	16/-	"	---	---	16/-
-14	24/43	"	---	---	24/43
-15	9/35	"	2/50	150	12/25
-16	~ 12/-	"	---	---	~12/-
-17	12/30	"	---	---	12/30
-18	12/30	"	---	---	12/30

1. Time in mold

TABLE 8-4

FLUOROSILICONE RTV'S WITH ADDITIVE
-SPECIMEN DISPOSITION-

(For Final Screening and Characterization)

Specimen No. F-	Test
387-3A	Odor and outgassing (200°F), TGA
B	Flammability (connectors):D-NA-0002, Test No. 5
387-4	Insulation resistance (no preconditioning)
387-5A	Fungus resistance
B	Flammability (connectors): Work Statement, para. 3.4.1.2 (piggy-back)
C	Insulation resistance (moisture cycle)
D	Shrinkage
387-6A	Odor and outgassing (155°F)
B	Dielectric constant, power factor, and arc resistance
C	Insulation resistance (no preconditioning)
387-7A	Tear, tensile strength, and elongation: partial
B	Dielectric constant, power factor and arc resistance
C	Work Statement, para. 3.4.5.4 (insulation resistance/ repairability), odor and outgassing (155°F)
387-8A	Tear, tensile strength, and elongation: partial
B	Dielectric constant, power factor, and arc resistance
C	Work Statement, para. 3.4.5.4 (insulation resistance/ repairability), vacuum volatility (No. 2)
D	Vacuum volatility (No. 1)
387-9A	Dielectric strength
B	Surface and volume resistivity
C	Work Statement, para. 3.4.5.4 (insulation resistance/ repairability)
387-10A	Dielectric strength
B	Surface and volume resistivity
C	Insulation resistance (vacuum exposed)
387-11A	(Additional molded sheet)
B	Surface and volume resistivity
C	Insulation resistance (moisture cycle)

TABLE 8-4 (cont'd)

FLUOROSILICONE RTV'S WITH ADDITIVE
-SPECIMEN DISPOSITION-
(For Final Screening and Characterization)

Specimen No. F-	Test
387-12A	Dielectric strength
B	Dielectric strength
C	Insulation resistance (no preconditioning)
387-13A	Dielectric strength
B	Insulation resistance (moisture cycle)
C	Pot life
387-14	Work Statement, para. 3.4.5.4 (insulation resistance/ repairability of F-387-7C)
387-15	Work Statement, para. 3.4.5.4 (insulation resistance/ repairability of F-387-8C)
387-16	Work Statement, para. 3.4.5.4 (insulation resistance/ repairability of F-387-9C)
387-17	Wire bond strength
387-18	Wire bond strength

8.3.2 Electrical Properties

A summary of the electrical results are given in Table 8-5. It is seen that except for two tests, all of the electrical requirements are readily met.

Although the average value for dielectric strength meets the requirement for 350. volts/mil, one of the five tested specimens had a value of 340. volts/mil. This is less than approximately 3% low; considered well within the test accuracy, and less than the amount that the average value exceeds the allowable minimum.

The arc resistance results were initially of concern. When the test was run, the typical conductive carbonaceous track was observed after approximately six seconds. In order to place this observation in its proper perspective, the insulation resistance of the two connectors which had been exposed to the "piggy-back" flammability test, was checked with a standard multimeter. The insulation resistance exceeded the instrument's maximum range (i.e. 1×10^7 ohms) when measurements were made from a) pin to pin, b) pin to carbon coated surfaces, c) pin to carbonized surfaces, or d) carbonized surfaces to carbonized surfaces. It is evident that this phenomena necessitates further investigation to determine:

a. effect of high voltage dielectric breakdown on other electrical properties,
b. whether sub-surface high voltage breakdown for this compound has a similar behavior, and
c. whether this characteristic can be utilized for a self-indicating potting/coating compound to detect a high voltage dielectric breakdown, while normally functioning as a good electrical insulating self-extinguishing system. This could provide a unique visual method which not only called attention to a high voltage failure, but also designated the failure's location.

Since a large proportion of the electrical evaluation was based upon the insulation resistance test, some attention was given to the inherent accuracy of this test (i.e. ASTM Test Method D-257) with respect to material, temperature, and humidity. Regarding material, ASTM states (Ref. 8-1) that:

> "...determinations are usually not reproducible to closer than 10%, and often are even more widely divergent (a range of values of 10 to 1 may be obtained under apparently identical conditions)."

As to temperature and humidity, this definitive reference (Ref. 8-1) further states that:

> "...For some materials a change from 25 to 100°C may change insulation resistance or conductance by a factor of 100,000; a change from 25 to 90 percent relative humidity may change them by as much as a factor of 1,000,000 or even more..."

TABLE 8-5
ELECTRICAL PROPERTIES
(Formulation 387)

Property	Requirement (9) T/M (1)	Requirement (9) Value	No. (2)	Results Min.	Results Max.	Results Ave.
Dielectric Constant	T	5.0, max.	3	4.7	4.8	4.7
Power Factor	T	0.09, max.	3	0.020	0.021	0.020
Dielectric Strength (V/mil) (3)	M	350., min.	5	340.	390.	370.
Volume Resistivity (4) (x 10^{12} ohms-cm)	T	1.0, min.	3	19.	30.	24.
Surface Resistivity (x 10^{12} ohms)	T	1.0, min.	3	230.	720.	390.
Arc Resistance (seconds) (5)	T	45., min.	3	6.	6.	6.
Insulation Res. (megohms) (6)						
<u>Set</u>						
A. No Precond., amb. (x 10^5)	M	1.0, min.	3	9.4	46.0	26.5
No Precond., 212°F	M	750., min.	3	3800.	8000.	5900.
B. No Precond., amb. (x 10^5)	M	1.0, min.	3	21.0	61.0	44.7
Precond. (7), amb.	M	200., min.	3	7.9	9.6	8.8 x 10^4
Precond. (7), 212°F	M	200., min.	3	860.	1130.	980.
C. No Precond., amb. (x 10^5)	M	1.0, min.	3	22.0	29.0	25.3
Repaired, amb. (x 10^5)	M	1.0, min.	3	59.0	69.0	64.0
Repaired, Precond. (7) amb.	M	200., min.	3	7.7	8.2	8.0 x 10^4
Repaired, Precond. (7) 212°F	M	200., min.	3	870.	880.	870.
D. No Precond., amb. (x 10^5)	M	1.0, min.	1	---	---	24.0
Precond. (8,7) amb.	M	200., min.	1	---	---	10.0 x 10^4
Precond. (8,7) 212°F	M	200., min.	1	---	---	960.

TABLE 8-5 (cont'd)
ELECTRICAL PROPERTIES
(Formulation 387)

1. T.....Target value M.....Mandatory value
2. Number of individually fabricated specimens.
3. 50 mils.
4. Potting.
5. Based on average value out of 5 for each specimen.
6. Based on average value out of 4 for each specimen.
7. Moisture cycle: 75-160°F/95% R.H./ 1 day per cycle/5 days.
8. Ambient temp./24 hrs/1 x 10^{-4} torr.
9. Ref. Appendix A.

With this in mind, a series of tests were conducted on a single insulation resistance specimen, F-387-11C. The test conditions and results are tabulated in Table 8-6.

TABLE 8-6

INSULATION RESISTANCE TEST CONDITIONS
(Formulation 387-11C)

	Test Conditions (1)			Results (2)		
Steps	Time	Temp. (°F)	Rel. Humid. (%)	Min.	Max.	Ave.
1.	0.5	75.	53.	5.6	6.4	6.1×10^{12}
2.	5 cycles of moisture conditioning per MSFC-SPEC-202A, para. 4.4.3.11.					
3.	0.5	82.	68.	8.5	9.2	8.9×10^{10}
4.	0.5	212.	---	9.1	10.0	9.5×10^{8}
5.	0.5	83.	$\sim$ -0- (3)	4.6	9.8	6.8×10^{11}

1. Successive events.
2. Four readings per test condition.
3. Tested in 57% relative humidity immediately (test completed in <5. minutes) after removal from a 72. hour desiccation over silica gel.

It is seen from Table 8-5 that for an approximately identical set of test conditions, the results for all of the ten ambient tested specimens were well within one order of magnitude. Comparing the temperature change from ambient to 212°F (i.e. steps 3 and 4 of Table 8-6), we see that the results are within two orders of magnitude. Less than one order of magnitude change occurred between approximately zero R.H. and 68% R.H. All of these comparisons show that F-387 is quite consistent, regardless of specimen/batch tested, and variations in temperature and relative humidity.

8.3.3 Mechanical Properties

The data in Table 8-7 indicates that the mechanical strength of F-387 is generally lower than the required values. However, the extensive "handling" of the cured compound while preparing the test specimens, and during the actual testing phases, clearly showed that the mechanical properties were still sufficiently high for practical usage. The 130% elongation permits better load sharing than what occurs with the more rigid potting/coating compounds.

It should be noted that although only three tear, tensile, and elongation specimens are normally required (Ref. 8-2) five were utilized from two separately molded sheets in order to better observe anomalies and extent of spread in the data.

TABLE 8-7
MECHANICAL PROPERTIES
(Formulation 387)

	Requirement (1)			Results		
Property	T/M (2)	Value	No. (3)	Min.	Max.	Ave.
Tear Strength (lb/in)	M	25., min.	5	11.	14.	13.
Tensile Strength (psi)	M	250., min.	5	84.	98.	90.
Elongation (%)	M	125., min.	5	120.	140.	130.
Wire Bond Strength (lb/wire)	M	10., min.				
TFE (4) Wire			15	10.3	11.8	11.0
Kapton/FEP (5) Wire			15	25.8	32.9	28.6
Specimens with Cohesive Failure (6)						
TFE	T	(Cohesive	15			
K/FEP	T	(failure	15			

1. Ref. Appendix A
2. T.....Target value M.....Mandatory value
3. Number of individually fabricated specimens.
4. Polytetrafluoroethylene coating.
5. Polyimide/FEP fluorocarbon laminated films with top coating of FEP fluorocarbon dispersions.
6. Number of specimens having cohesive failure greater than 50%.

8.3.4 Physical and Chemical Properties

Viscosity data is presented in Table 8-8. The data shows that:

a. the base is very slightly thixotropic,
b. the catalyst more than compensates for the increase in viscosity caused by the addition of the Dechlorane 604,
c. the viscosity of the catalyzed compound is less than the "minimum" 100 poise shown as the requirement,
d. the viscosity of the base is less than that quoted by the vendor (i.e. 400-800 poise) (Ref. 8-3).

The catalyzed compound is white. It was consistently found to be homogeneous, with no coarse particles, when prepared from selected particle sized D-604 and carefully blended. No readily seen voids were present in oven cured specimens. The presence of microscopic voids was contingent upon the extent of degassing of the catalyzed formulation. The degassing was carried out at room temperature since the polymer's vendor feels that "...the crosslinker in the polymer might volatilze under a thermal/ vacuum treatment." (Ref. 7-2). A conventional 29-30 in. Hg. vacuum system was used in the Project's degassing activities.

Although the Project's principal concern with hardness was the necessity of complying with the applicable requirement of the Statement of Work, it was also utilized as a quality assurance criteria in evaluating the effectiveness of several batches of 77-033 catalyst. This latter situation arose when mislabeled catalyst was sent by the vendor. At their suggestion (Ref. 8-4), the hardness of cured polymer castings was measured, using different batches of catalyst (Table 8-9). Dow Corning reports (Ref. 8-3) a hardness range of 30-40 (Shore A) when cured 24 hours at room temperature. The data shows that when the same batch of polymer base is catalyzed with the required 10 phr of both the original and the most recent catalyst batches, there is a significant difference in the hardness. The "original" specimen is clearly not satisfactory, whereas the "most recent" specimen is exactly in the expected range. It is also evident that the degassed specimen had slightly lower results, but with less data spread. Batch 319B catalyst was so obviously defective (i.e. well defined separation of two liquids present in shipping containers, and rust particles in container) that no attempt was made to use it. Subsequent investigation by the vendor substantiated our rejection of this batch.

The above results suggested that a closer examination be made of the data obtained from formulations F-387-1, -2, -3, and -4, which were prepared from the batch 271B catalyst. Although these formulations were used extensively in the final screening tests, the only specimen which could be comparatively evaluated with similar ones made from another 77-033 catalyst batch was F-387-4, a non-preconditioned insulation resistance specimen. This specimen had a room temperature average test value of 9.4×10^{11} ohms, whereas the other nine specimens similarly tested were

TABLE 8-8

VISCOSITY
(Formulation 387)

Material	Requirement		Test Conditions (2)				Viscosity (poise)	
	T/M (1)	Value (poise)	Volume Beaker	Volume Mat'l.	Speed (rpm)	Temp. (°F)	Min.	Max.
77-033 Base (3)	(6)	---	600.	450.	10. (8)	77.	71.6	77.0
			250.	150.(7)	10. (8)	77.	73.2	75.0
			600.	450.	10. (9)	77.	71.0	76.5
			250.	150.(7)	10. (9)	77.	75.7	75.7
77-033/150 phr Dechlorane 604 (4)	(6)	---	600.	450.	1.(10)	77.	96.3	99.8
			250.	150.(7)	1.(10)	77.	93.8	100.+
77-033/150 phr Dechlorane 604/ 77-033 catalyst (5)	M	100.- 6000.	250.	150.(7)	1.(10)	78.	52.8	56.5

1. T.....Target value M.....Mandatory value
2. Brookfield Model RVT Viscometer, spindle No. 6.
3. Inventory No. 319A-2/3.
4. F-461-1.
5. F-387-5.
6. Test not required by Statement of Work.
7. Resin level: approx. 1/4" below spindle mark.
8. Pre-stirred 50. hours.
9. Pre-evacuated 2 hours at 77°F under 29-30" Hg. vacuum.
10. Pre-stirred 1. hour.

TABLE 8-9

HARDNESS

Specimen No. F-	Polymer Base	Inventory No. Polymer Catalyst	DECHLORANE 604	Cure at 150°F (hrs/min)	Hardness (Shore A-2) Aluminum Cup Mold (1) Exposed Surface Min.	Max.	Ave.	"Aluminum" Surface Min.	Max.	Ave.
268-2	319A-3	271B (2)	---	2/- (6)	22.	32.	25.	22.	26.	24.
268-3	319A-3	345 (3)	---	" "	35.	45.	40.	32.	46.	34.
268-4	319A-2/3 (9)	345 (3)	---	" "	38.	40.	38.	35.	39.	38.

Specimen No. F-	Polymer Base	Polymer Catalyst	DECHLORANE 604	Cure at 150°F (hrs/min)	Requirement T/M (10)	Value	No. (12)	Sheet Surface (11) Min.	Max.	Ave.
387-6B	319A-2/3 (9)	345 (3)	335 (4)	12/- (6)	↑	↑	7	54.	61.	59.
-6C	" "	" "	" "	" "	↑	↑	4	52.	59.	56.
-7A	" "	" "	" "	12/10 "	T	50-85	7	50.	70.	66.
-8A	" "	" "	" "	15/30 "	↓	↓	6	65.	72.	68.
-11A	" "	" "	" (5)/ 346 (5)	13/15 (7)	↓	↓	6	75.	80.	77.
-12C	" "	" "	335 (5)/ 346 (5)	12/- (8)	↓	↓	4	63.	67.	65.
				Total	---	----	34	---	---	---
				Ave.	T	50-85	---	60.	68.	65.

1. In "as received" condition.
2. Original catalyst batch.
3. Most recent catalyst batch.
4. 100 - 170 mesh.
5. Partly 100 - 170 mesh, partly less than 170 mesh.
6. Not degassed.
7. Degassed 10. minutes/80°F/29-30" Hg. vac.
8. Degassed 10. minutes/ 74°F/29-30" Hg. vac.
9. Degassed 2 hrs/ambient temp./29-30" Hg. vac.
10. T.....Target value M.....Mandatory value
11. Mylar mold surface.
12. Readings per sheet.

in a range of 2.1-6.1 x 10^{12} ohms. Likewise, the tests at 212°F showed that F-387-4 again had the lowest resistance (i.e. 3.8 x 10^9 ohms as compared to 5.9 and 8.0 x 10^9 ohms). Even though F-387-4 specimen had the lowest resistance, the insulation results did not deviate that significantly from the other insulation results to unequivocally establish a relationship with the quality of the catalyst. However, it is certainly suspect and would justify further analysis for its future use.

It is seen from Table 8-9 that the inclusion of 150 phr Dechlorane 604 almost doubles the hardness. It is also evident that all of the 34 readings from the six separate sheets fall within the required range.

The specific gravity of three molded 1." x 1." x 1." cubes of F-387 were determined to be:

Minimum: 1.65
Maximum: 1.69
Average: 1.67

Since the requirement is 2.2, maximum, F-387 meets this objective.

These some cubes were measured and found to have less than 1.% shrinkage. This also complies with the requirement of the Statement of Work (i.e. 3.%, maximum).

Four replicares of F-387-5A were inoculated with a spore suspension of four pure cultured fungi per Reference 7-1, Section 4.4.3.9, for 28 days at 86°F. There was no visible change in the appearance of the specimens following this exposure. This met the requirement of the Statement of Work (Ref. Appendix A, Section 3.4.5.1).

Based upon the remaining requirements of the Statement of Work (Ref. Appendix A), the following pertinent comments apply to F-387:

a. Section 3.2.2: An adhesion promoting primer, 1200 (Dow Corning Corp.), was evaluated in the wire bond pull test and some of the electrical insulation specimens. The primer was applied by dipping the substrate into it. It can also be applied by spraying, or wiping on with a lint-free cheese cloth. The primer was allowed to dry for 1.-1.5 hours (the vendor recommends 1.0 hour at 77°F and 30.0%, minimum, relative humidity). No difficulty was encountered in its use.
b. Section 3.2.3: The vendor's data sheet for the polymer (Ref. 8-3) does not provide any information regarding its toxicity or the need for special handling precautions. However, based upon information pertaining to somewhat similar polymeric systems, it is believed that no unudual precautions are needed in its use. The vendor for the Dechlorane 604 stipulates (Ref. 7-3) that:

> "Tests conducted on rats indicate that Dechlorane 604 may be relatively non-toxic by oral ingestion and non-irritating to the skin. It may be slightly irritating to the eyes."

Although the above information regarding the principal constituents of F-387 suggest that no potentially toxic materials are present, it is still advisable to utilize normal industrial hygiene practices during the processing of F-387.

c. Section 3.2.4: Due to the inherent characteristics of the constituents in F-387, it is not feasible to make it transparent. Although no special pigments or color additives are required, it may be deemed useful as a quality control device to pigment the catalyst in order to assure complete blending of the two component system. Suitable non-toxic pigments have been selected and used in similar polymeric systems (see Section 10.).

d. Section 3.3.2: The compound can readily be applied by spatula, or by injection or extrusion gun. Slightly greater difficulty is encountered when applying F-387 by brush or dipping. A mastic-type gun would be needed if spraying was desired.

e. Section 3.3.3: F-387 has a pot life of approximately 2-2 1/2 hours at 75-80°F when used as a potting material; 1-2 hours when used as a conformal coating.

f. Section 3.3.4: F-387 is tack-free in 5-8 hours at room temperature. The Project has generally cured F-387 during the data gathering phases of this program for approximately 12 hours at 150°F. The polymer's vendor states (Ref. 8-3) that full physical properties and best adhesion can be achieved in only four hours at 150°F or seven days at 77°F. The 12 hour cycle was selected in case polymer growth was in any way slowed by the presence of the D-604. This concern may be groundless; additional testing is needed to clarify this issue.

g. Section 3.3.5: F-387 has not yet been exposed to a six month/70°F, maximum, storage cycle and retested to the criteria of the Statement of Work (Ref. Appendix A). However, the polymer's vendor claims (Ref. 8-3) that 77-033 has a shelf life of 6 months.

SECTION 9

NASA-MSC FORMULATION SG-12KI

9.1 FORMULATION SELECTION

In accordance with contractual requirements (Ref. 9-1), the Project prepared and tested a NASA-specified flame retardant formulation (Ref. 9-2).

9.2 FORMULATION/PROCESSING

The compositions of the batches which were prepared per SK-12KI are listed in Table 9-1. The associated processing data is shown in Table 9-2. Formulation blending and cure cycles were as specified by NASA (Ref. 9-2).

9.3 EVALUATION

Table 9-3 is herein provided to clarify the genesis of the individual test specimens with respect to the applicable specific batch and cure cycle. Table 9-4 tabulates qualitative data for various mechanical properties. It is seen from this table that these qualitative values were unchanged whether the post cure was 25. or 96. hours.

The evaluation criteria for SK-12KI is as stipulated in the Statement of Work (revised 13 Aug. 1968) and as modified in Reference 9-1.

SG-12KI was tested via the Furane "preliminary" flammability procedures (Ref. Appendices H and N), using the 1/4" x 1/4" x 1.0" specimen (Ref. Appendix O). The resultant data are presented in Table 9-5. None passed the test in 6.2 psia oxygen.

All of the thermal property data are summarized in Tables 9-6 and 9-7. The electrical properties are shown in Table 9-8. The mechanical properties are tabulated in Table 9-9. Physical/optical properties are listed in Table 9-10. The chemical properties are covered in Table 9-11.

The poor flammability resistance and unsatisfactory electrical properties after the moisture cycle were the most objectionable characteristics of this formulation. In addition, the thermal/vacuum weight losses were excessive. The solubility and hygroscopic nature of the ammonium phosphate was the source of the difficulties in high humidity environments.

This formulation appears suitable for use as a potting/encapsulating compound in a low, or even moderate, humidity environment, and/or in the presence of a low

TABLE 9-1

FORMULATION NASA-MSC-SG-12KI

Component	S	300-1	301-1	302-1	306-1	308-1	311-1	311-2	311-3	304-1	303-1	303-2	303-3	303-4	305-1
		Formulations (phr) (1)													
SYLGARD 186	12	100.			100.	100.	100.	100.	100.						
$NH_4H_2PO_4$	10	75.			75.	75.	75.	75.	75.						
300-1	13		175.	175.											
301-1	13									225.					
302-1	13										225.	225.	225.	225.	225.
7570 Glass Frit(2)	21		50.												
7570 Glass Frit(3)	21			50.											
7570 Glass Frit(4)	21				50.										
7570 Glass Frit(5)	21					50.	50.	50.	50.						
S-186 Catalyst	12									10.	10.	10.	10.	10.	10.

Component	S	307-1	307-2	307-3	309-1	310-1	310-2	310-3	310-4	312-1	313-1	314-1	314-2	314-3	314-4	314-
		Formulations (phr) (1)														
306-1	13	225.	225.	225.	95.8											
308-1	13				129.2	225.	225.	225.	225.	100.5						
311-1	13									124.5	225.					
312-1	13											225.	225.	225.	225.	225
S-186 Catalyst	12	10.	10.	10.	10.	10.	10.	10.	10.		10.	10.	10.	10.	10.	10

TABLE 9-1 (cont'd)

FORMULATION NASA-MSC-SG12K1

		Formulations (phr) (1)												
Component	S	315-1	315-2	315-3	315-4	315-6	315-7	315-8	(6) 315-9	(7) 315-10	(8) 315-11	315-12	316-1	317-1
311-1	13	225.	225.	225.	225.	225.	225.	225.	225.	180.				
312-1	13									45.				
311-2	13										225.	225.		225.
314-5	13												235.	
315-1	13													
S-186 Catalyst	12	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.	10.		10.

S......Source (See Table 6-1)
1. Processed per NASA-MSC-SG 12K1)
2. Not predried
3. Predried 14 hrs./212°F
4. Predried 7 days/212°F
5. Predried 6 hrs./212°F
6. Half of specimen is 315-2 (Repair Test)
7. Half of specimen is 314-3 (Repair Test)
8. Half of specimen is 314-4 (Repair Test)

TABLE 9-2

FORMULATION NASA-MSC-SG-12KI
-FORMULATION PROCESSING-

Formulation No. F-	Cure (1) Time (hrs/min)	Cure (1) Temp. (°F)	Post Cure Time (hrs/min)	Post Cure Temp. (°F)
303-1	2/-	150	24/-	100
303-2	"	"	48/-	"
303-3	"	"	51/-	"
303-4	"	"	48/-	"
304-1A	"	"	25/-	"
304-1B	"	"	96/-	"
305-1	"	"	97/30	"
307-1	"	"	48/-	"
307-2	"	"	47/-	"
307-3	"	"	48/-	"
309-1	"	"	"	"
310-1	"	"	44/30	"
310-2	"	"	49/15	"
310-3	"	"	48/15	"
310-4	"	"	66/25	"
313-1	"	"	48/-	"
314-1	"	"	"	"

1. Time in mold

TABLE 9-2(cont'd)

FORMULATION NASA-MSC-SG-12KI
-FORMULATION PROCESSING-

Formulation No. F-	Cure (1) Time (hrs/min)	Cure (1) Temp. (°F)	Post Cure Time (hrs/min)	Post Cure Temp. (°F)
314-2	2/-	150	48/11	100
314-3	"	"	48/19	"
314-4	"	"	49/16	"
314-5	(used	for	F- 316 - 1)	
315-1	("	"	")	
315-2	2/-	150	48/-	"
315-3	"	"	49/40	"
315-4	"	"	51/-	"
315-5	"	"	58/30	"
315-6	"	"	56/-	"
315-7	"	"	54/-	"
315-8	"	"	"	"
315-9	"	"	49/16	"
315-10	"	"	48/-	"
315-11	"	"	"	"
315-12	"	"	"	"
316-1	"	"	50/40	"
317-1	"	"	48/-	"

1. Time in mold

TABLE 9-3

FORMULATION NASA-MSC-SG-12KI
- SPECIMEN DISPOSITION -

Specimen No. F-	Test
303-1	Flammability (preliminary): Appendices I and J
303-2	Outgassing (200°F) (1), TGA (1), and 600°F thermal decomposition (1)
303-3	Dielectric constant, power factor, and arc resistance
303-4A	Fungus resistance
B	Dielectric constant, power factor, and arc resistance
304-1A	Flammability (preliminary): Appendices I and J
B	Flammability (preliminary): Appendices I and J
C	Qualitative mechanical properties
305-1A	Flammability (preliminary): Appendices I and J
B	Flammability (Preliminary): Appendices I and J
C	Qualitative mechanical properties and vacuum volatility
307-1A	Surface (R.T.) and volume (R.T. and 212°F) resistivity
B	Surface (R.T.) and volume (R.T. and 212°F) resistivity
C	Surface (R.T.) and volume (R.T. and 212°F) resistivity
307-2A	Dielectric strength
B	Dielectric strength
C	Dielectric strength
D	Insulation resistance (moisture cycle) (2)
307-3A	Vacuum volatility
B	Dielectric strength
C	Dielectric strength
D	Insulation resistance (moisture cycle) (2)
309-1A	Dielectric constant, power factor, and arc resistance
B	Fungus resistance
C	Odor (1)
D	Insulation resistance (moisture cycle) (2)
E	Shrinkage

TABLE 9-3 (cont'd)

FORMULATION NASA-MSC-SG-12KI
- SPECIMEN DISPOSITION -

Specimen No. F-	Test
310-1A	Flammability (connector): D-NA-0002, Test No. 5
B	Flammability (connector): D-NA-0002, Test No. 5
C	Flammability (connector): D-NA-0002, Test No. 5
D	Insulation resistance (moisture cycle) (3)
310-2A	Flammability (connector): D-NA-0002, Test No. 5
B	Flammability (connector): D-NA-0002, Test No. 5
C	Flammability (connector): D-NA-0002, Test No. 5
D	Flammability (connector): D-NA-0002, Test No. 5
E	Flammability "piggy back" (connector): Ref. Appendix A, Section 3.4.1.2
F	Flammability "piggy back" (connector): Ref. Appendix A, Section 3.4.1.2
G	Flammability (connector): D-NA-0002, Test No. 5
H	Insulation resistance (moisture cycle) (3)
310-3	Insulation resistance
310-4	Insulation resistance (moisture cycle) (3)
313-1A	Insulation resistance
B	Compression set (preliminary)
314-1A	Insulation resistance
B	Flammability (preliminary): Appendices I and J
C	Compression set (final)
D	Qualitative mechanical properties
314-2	High potential resistance
314-3	Work Statement, para. 3.4.5.4 (insulation resistance/repairability)
314-4	Work Statement, para. 3.4.5.4 (insulation resistance/repaieability)
315-2	Work Statement, para. 3.4.5.4 (insulation resistance/repairability)
315-3	High potential resistance
315-4	(Additional insulation resistance specimen)
315-5	Insulation resistance (vacuum exposed)

TABLE 9-3 (cont'd)

FORMULATION NASA-MSC-SG-12KI
- SPECIMEN DISPOSITION -

Specimen F-	Test
315-6	(Additional insulation resistance specimen)
315-7	Tear, tensile strength, and elongation
315-8A	Tear, tensile strength, and elongation
315-9	Work Statement, para. 3.4.5.4 (insulation resistance/repairability of F-315-2)
315-10	Work Statement, para. 3.4.5.4 (insulation resistance/repairability of F-314-3)
315-11	Work Statement, para. 3.4.5.4 (insulation resistance/repairability of F-314-4)
315-12A	Low temperature flexural strength
B	Wire bond strength
316-1	High potential resistance
317-1A	(Additional 1/8" x 6." x 6." molded sheet)
B	(Additional 0.080" x 6." x 6." molded sheet)
C	Ozone resistance
D	Compression set

1. Test terminated per NASA-MSC instructions (Ref. 9-3)
2. Test terminated during moisture cycle due to equipment failure.
3. Replacements for 2.

TABLE 9-4

FORMULATION NASA-MSC-SG-12KI

CHARACTERISTICS

-CURED/POST CURED-

Properties	304-1A	304-1B	305-1	314-1
Cured	Y	Y	Y	Y
Tacky	N	N	N	N
Flexible	Y	Y	Y	Y
Memory	Y	Y	Y	Y
Cohesion	X	X	X	X
Tear Resistance	X	X	X	X
Crack Resistance (1)	X	X	X	X
Crack Propagation (1)	X	X	X	X
Porous	N	N	N	N
Adheres to unclean alum. Foil cup	---	---	X	---
Cheesy	N	N	N	N

Y.....Yes N.....No
X.......Satisfactory

1. 180° bend (face-to-face contact)

TABLE 9-5

FORMULATION NASA-MSC-SG-12KI PRELIMINARY FLAMMABILITY - TEST CONDITIONS/RESULTS -

		Specimen					
		303-1-1	303-1-2	303-1-1	304-1A-1	304-1B-1	304-1A-1
	O$_2$ Pressure (psia)	(7)	(7)	6.2	(7)	(7)	6.2
RESULTS	Wire Ignition (1)	T/B	T/B	T	T	T/B	T
	Wire Failure (1)	T	T	T	T	T	T
	Smoke (2)	N	N	S	N	N	P
	Burn time (sec.) (3) flame-out	0	0	43	0	1	45
	Burn time (sec.) (3) glow-out	0	0	43	0	1	50
	Configuration (4)	Y	Y	Y	Y	Y	Y
	Residue (%) (5)	>99	>99	85	>99	>99	85
	Melted (6)	N	N	N	N	N	N
	Color exterior	white	white	white	white	white	white
	Color interior	"	"	"	"	"	"
	Exterior Texture powdery (6)	N	N	N	N	N	N
	Exterior Texture crusty (6)	N (8)	N	Y	N	N	Y

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. Ambient pressure air
8. Ignition not satisfactory

TABLE 9-5 (cont'd)

FORMULATION NASA-MSC-SG-12KI
PRELIMINARY FLAMMABILITY
– TEST CONDITIONS/RESULTS –

		Specimen				
		304-1B-1	305-1-1	305-1-1	314-1B-1	314-1B-1
	O_2 Pressure (psia)	6.2	(7)	6.2	(7)	6.2
RESULTS	Wire Ignition (1)	T/B	T/B	T/B	T	T/B
	Wire Failure (1)	T	T	T	T	T/B
	Smoke (2)	P	N	P	N	P
	Burn time (sec.) (3) flame-out	39	1	45	0	73
	Burn time (sec.) (3) glow-out	42	1	47	0	75
	Configuration (4)	Y	Y	Y	Y	Y
	Residue (%) (5)	85	>99	85	>99	85
	Melted (6)	N	N	N	N	N
	Color exterior	white	white	white	white	wht/blk
	Color interior	"	"	"	"	"
	Exterior Texture powdery (6)	N	N	N	N	N
	Exterior Texture crusty (6)	Y	N	Y	N	Y

1. T.....Top B.....Bottom
2. N....None S.....Some P.....Profuse
3. Zero time: start of specimen burning
4. Generally similar to original specimen: Yes or No
5. Amount left in (4)
6. Yes or No
7. Ambient pressure air
8. Ignition not satisfactory

TABLE 9-6

THERMAL PROPERTIES (SUMMARY)
(NASA-MSC-SG12KI)

Flammability	Results				Requirement	
	Degradation (1, 2, 3)			S. E. (4)	T/M (5)	Value
	N	S	E			
Preliminary (6):						
Ambient Air	5	--	--	--	--	
6.2 psia O_2	--	--	4	--	--	
60% O_2/40% N_2: 16.2 psia	--	--	--	--	--	
16.5 psia O_2	--	--	--	--	--	S. E. (4)
Screening:						
6.2 psia O_2	--	--	2	No	M	
60% O_2/40% N_2: 16.2 psia	--	--	2	No	M	
60% O_2/40% N_2: 16.2 psia (6, 8)	--	--	1	No	--	
TGA, ambient to 932°F (7)	(9)				(2)	
Weight Loss (7)						
200°F / 5 psia O_2	(9)				(2)	
250°F. /1 x 10^{-4} Torr/24 hrs. (10)	No (see Table 9-7)				M	1.0, max.
600°F. /14.7 psia O_2 (%)	(9)				M	10, max.
Weight Loss Rate, 1 x 10^{-4} Torr (7)						
250°F./6 hrs. (10)	No (see Table 9-7)				M	Stabilize Rate
250°F. /6 to 24 hrs. (%/hr.) (10)	No (see Table 9-7)				M	0.02, max.; rate decreases with time
Vac. Deposition of Outgassed Mat'l., 150°F, max. / 1 x 10^{-4} Torr (7)	None				M	None

(1) N. . . . . None S. . . . . Some E. . . . . Extensive
(2) Value not specified in Work Statement.
(3) Number designates quantity of individual specimens.
(4) S. E. . . . . . Self-extinguishing immediately
(5) T. . . . . Target value M. . . . . Mandatory value
(6) Test not specified in Work Statement (revised 13 Aug., 1968)
(7) Data from single tests.
(8) "Piggy back" flammability propagation test.
(9) Testing terminated at W. S. T. F. (Ref. 9-3).

TABLE 9-6 (cont'd)
THERMAL PROPERTIES (SUMMARY)
(NASA-MSC-SG12K1)

(10) The sample was analyzed using a vacuum thermobalance. A weighed sample was placed on the balance pan and the pressure reduced to 10^{-4} - 10^{-5} for 64 hours. The sample was maintained at room temperature, 77°F ± 10°F, during this time. The sample was then heated to 250°F ± 2°F and maintained for 24 hours. The weight of the sample was continuously recorded during this time. The chamber volume was 40 cc.

Sample Weight	
1. Initial	106.34 mg.
2. After 64 hrs. /10^{-5} Torr/77°F	106.13 "
3. After attaining thermal equilibrium (10 min.)	105.95 "
4. After 24 hrs. /10^{-5} Torr/250°F	77.87 "

TABLE 9-7

THERMAL/VACUUM WEIGHT LOSS
24 Hour Weight Loss at 250°F/1 x 10^{-4} Torr (1)
(NASA-MSC-SG12KI)

Time (Hours)	Percent Loss
0 (thermal equilibrium)	0.17
0.3	0.52
0.6	0.73
0.9	0.87
1.2	1.02
1.5	1.18
2.1	1.41
3.0	1.79
4.5	2.81
6.0	4.27
7.5	5.98
9.0	7.80
10.5	9.73
12.0	11.85
13.5	14.16
15.0	16.61
16.5	16.99
18.0	21.09
20.5	23.16
21.0	25.04
22.5	26.55
24.0	26.63

1. The specimen was exposed to a vacuum cleaning at 77°F for 64 hours prior to this test. It is therefore not known whether a pre-test vacuum cleaning at 150°F would reduce the weight loss in this test.

TABLE 9-8

ELECTRICAL PROPERTIES (SUMMARY)
(NASA-MSC-SG12KI)

Property	Results				Requirement	
	No. (1)	Min.	Max.	Ave.	T/M (2)	Value
Dielectric Constant	3	6.6	6.7	6.7	T	5.0, max.
Power Factor	3	0.003	0.008	0.005	T	0.09, max.
Dielectric Strength (V/mil)	5	260	310	290	M	500, min. (3)
Surface Resistance (x 10^6 Megohm)	3	5.	22.	15.	T	1.0, min.
Volume Res., amb. (x 10^6 Megohm-cm.)	3	0.062	0.55	0.30	T	1.0, min.
Volume Res. (4), 212°F (x 10^6 Megohm-cm.)	3	33.	110.	67.	T	1 x 10^{-3}, min.
Volume Res. (4), 212°F (9) (x 10^6 Megohm-cm.)	3	28.	120.	61.	(10)	--
Arc. Res. (seconds) (5)	3	122	124	123	T	45, min.
High Potential Resistance 60 Hz/sec.	3	No breakdown			M	No breakdown
Insulation Res. (Megohms) (6)						
Set						
A. No Precond., amb. (x 10^5)	3	21.	660.	330.	M	1.0, min.
No Precond., 212°F. (x 10^5)	3	48.	69.	60.	M	7.5 x 10^{-3}, min.
B. No Precond., amb. (x 10^5)	3	580.	1200.	860.	M	1.0, min.
Precond. (7), amb.	3	1.9	27.	10.	M	200, min.
Precond. (7), 212°F.	3	0.44	0.78	0.65	M	200, min.
C. No Precond., amb. (x 10^5)	3	25.	1080.	320.	M	1.0, min.
Repaired, amb. (x 10^5)	3	510.	1200.	820.	M	1.0, min.
Repaired, Precond. (7), amb.	3	0.095	0.39	0.23	M	200, min.
Repaired, Precond. (7), 212°F	3	0.025	0.057	0.039	M	200, min.

TABLE 9-8 (cont'd)

ELECTRICAL PROPERTIES (SUMMARY)
(NASA-MSC-SG12KI)

Property	Results				Requirement	
	No. (1)	Min.	Max.	Ave.	T/M (2)	Value
D. No Precond., amb. (x 10^5)	1	180.	1800.	1400.	M	1.0, min
Precond. (8), amb. (x 10^5)	1	900.	3000.	2000.	M	1.0, min.
Precond. (8, 7), amb.	1	0.037	1.8	0.76	M	200, min.
Precond. (8, 7), 212°F	1	0.01	0.22	0.10	M	200, min.

(1) Number of individually fabricated specimens.
(2) T..... Target value M..... Mandatory value
(3) 50 mils.
(4) Temperature resistance requirement of Work Statement (revised 13 Aug. 1968).
(5) Based on average value out of 5 for each specimen.
(6) Based on average value out of 4 for each specimen.
(7) Moisture cycle: 75-160°F. /95% R. H. /5 days.
(8) 1 x 10^{-4} Torr/ambient temp. / 24 hrs.
(9) Preconditioned at 212°F, tested at ambient.
(10) Test not specified in Work Statement (revised 13 Aug. 1968).

TABLE 9-9

MECHANICAL PROPERTIES (SUMMARY)
(NASA-MSC-SG12K1)

Property	Results				Requirement	
	No. Spec.	Min.	Max.	Aver.	T/M (1)	Value
Tensile Strength (psi)	5	250.	260.	260.	M	800, min.
Elongation (%)	5	310.	330.	318.	M	100, min.
Tear Strength (lb. /in.)	4	32.	37.	35.	M	75, min.
Compression Set (%)	3	0. 8	1. 0	0. 9	M	35, max.
Low Temp. Flexibility, 0 °F	3	No cracking or separation			M	No cracking or separation
Wire Bond Strength (lb. /wire)					M	10. , min.
TFE (2) Wire	15	10. 0	13. 9	12. 4		
Kapton/FEP (3) Wire	15	21. 8	37. 9	30. 6		
Specimens with Cohesive Failure (4)	TFE		-0-		M	Cohesive failure
	K/FEP		-0-		M	

(1) T. . . . . Target value M. . . . . Mandatory value
(2) Polytetrafluoroethylene coating.
(3) Polyimide/FEP fluorocarbon laminated films with top coating of FEP fluorocarbon dispersion.
(4) Number of specimens having cohesive failure greater than 50%.

TABLE 9-10

PHYSICAL/OPTICAL PROPERTIES
(NASA-MSC-SG12KI)

Property	Results				Requirement	
	No. Spec.	Min.	Max.	Ave.	T/M (1)	Value
Viscosity, 77 ± 1°F (poises)						
Base Polymer	942				(2)	--
"Filled" Polymer (uncat.)	8,144				(2)	--
"Filled" Polymer (cat.)	2,340				M	100-450
Homogeneous	Yes				M	Yes
Lumps/Coarse Particles	No lumps, slightly coarse				M	None
Voids	None (with vacuum)				M	None
Transparent	No				T	Yes
Shrinkage (%)	3	--	Nil	--	M	3., max.
Hardness, cured (Shore A)	5(3)	64	68	66	M	30-99
Specific Gravity	3	1.55	1.61	1.58	T	2.2, max.

(1) T..... Target value
M..... Mandatory value
(2) Test not required by Work Statement (revised 13 Aug. 1968)
(3) Number of separate sheets; 4-5 points/sheet.

TABLE 9-11

CHEMICAL PROPERTIES (SUMMARY)
(NASA-MSC-SG12K1)

Property	Results		Requirement	
	No. Spec.	Data	T/M (1)	Value
Pyrolysis Products	1	(5)	M	(2, 3)
Thermal Decomposition				
200°F/5 psia O_2/72 hrs.	1	(5)	M	(2, 3)
600°F/14.7 psia O_2	1	(5)	M	(2, 3, 4)
Odor: 200°F/5 psia O_2/ 72 hrs.	1	(5)	M	25 per 10 evaluations
Fungus: 86°F/28 days	1	No visible change	M	No visible change
Ozone: 50 PPHM/100°F/7 days	5	No cracking or crazing (6)	M	No cracking

(1) T. . . . . Target value M. . . . . Mandatory value

(2) Identify all peaks, and those items listed in 9.3.1.2 of MSC-D-NA-0002, Test Method No. 6, which exceed 10 μgm./gm.

(3) Total Organic: 100 μ gm./gm., maximum.
Carbon Monoxide: 25 μgm./gm., Maximum.

(4) None of the items listed in 9.3.1.2 of MSC-D-NA-0002, Test Method No. 6, shall be present above 10 μgm./gm.

(5) Testing terminated at W. S. T. F. (Ref. 9-3).

(6) 7X magnification.

oxygen content atmosphere. Further, it is not affected by an extended exposure to ozone nor does it act as a nutrient to fungus. It is easily and safely handled by even non-technical personnel, and its catalytic system proportioning is relatively non-critical. It is also readily repaired. Its electrical properties, from ambient to 212°F were satisfactory. The mechanical properties at 0°F were also acceptable. Finally, based upon the raw material costs and the associated processing tasks, it is anticipated that the resultant compound could be made available at a comparatively low cost.

SECTION 10

NASA-MSC FIRE SAFETY CONFERENCE (6, 7 MAY 1970) EXHIBIT SPECIMENS

At the request of NASA-MSC (Ref. 10-1), various potted, conformal coated, and encapsulated specimens were prepared for the above conference. The formulations are shown in Table 10-1 and a description of the processing and identification of the individual application-oriented specimens are given in Table 10-2.

As seen from Table 10-1, the pigments cover a broad array of colors. Except for the cadmium red, all are considered non-toxic. All were completely compatible with the various polymeric systems and the flame retardant additives. At the time these specimens were prepared, Dechlorane 604 had not been evaluated by the Project. We have no reason to doubt the compatibility of these pigments with F-387.

TABLE 10-1

NASA-MSC FIRE SAFETY CONFERENCE (6,7 MAY 1970) EXHIBIT SPECIMENS

Component	S	175X -2, 3	345	346	347	348	349	350	351	352	353	354
		Formulations (phr)										
77-024	12	100.										
77-033	12										100.	100.
F-301-1 (1)	13		225.		225.	225.						
F-311-3 (1)	13			225.			225.					
F-311-2,3 (1)	13							225.	225.	225.		
Boric acid	9	125.										
NH_4BF_4	20										150.	150.
Cadmium Red (2)	20		2.									
Meteor Pink (3)	20			1.								
Dark Red (4)	41				1.							1.
Sun Yellow 'C' (5)	20					1.						
Meteor Green 'B' (6)	20						1.					
Meteor Red Brown (7)	20							1.				
Sun Green 'L' (8)	20								1.			
Meteor Cobalt Blue (9)	20									1.	1.	
77-024 Catalyst	12	10.										
S-186 Catalyst	12		10.	10.	10.	10.	10.	10.	10.	10.		
77-033 Catalyst	12										10.	10.

S......Source (See Table 6-1)
1. See Table 9-1
2. F-3629
3. 7132
4. Ultrafine Synthetic Iron Oxide #1121M
5. H-463 Master Standard
6. 7465 (541-321-37)
7. 7751 (541-319-37)
8. (539-010-38)
9 7540 (541-039-37)

TABLE 10-2

NASA-MSC FIRE SAFETY CONFERENCE (6,7 MAY 1970) DEMONSTRATION SPECIMENS

- PROCESSING AND IDENTIFICATION -

Formulation No. F-	Furane No. Epocast	Cure (1) Time (hrs/min)	Cure (1) Temp. (°F)	Color	Demonstration Specimen
175X-2	N9520	2/25	150	grey	Vacuum tube shell (metal).
175X-3	"	2/-	"	"	Vacuum tube shell (metal).
345	-	13/-	"	red	(color and cure evaluation)
346	N9011	2/-	"	pink	Plywood
347	-	2/-	"	dk. red	(color and cure evaluation)
348	N9012	2/-	"	yellow	Plywood
349	N9013	12/-	"	green	Neoprene covered cable
350	N9015	2/-	"	red brown	Fiberglass fabric for cable overwrap
351	N9014	2/-	"	lt. green	Motor coil
352	N9016	2/-	"	blue	Printed circuit board
353	N9511	2/-	"	blue	Capacitors
354	N9512	2/-	"	dk. red	1N34 glass diodes

1. No post cure

SECTION 11

CONCLUSIONS

11.1 FORMULATION 387

a. Formulation 387 was found to have the best balance of thermal, electrical, mechanical, physical and chemical properties of those formulations evaluated within this Program.

b. The other formulations which received Final Screening are in the following order of preference:

F-387, -462, -437, -230X, -175X, -379, and -266.

c. F-387 met all of the requirements of the Statement of Work (Ref. Appendix A) except for the following:

Test	Requirement	T/M (1)	Results (Ave.)
Arc Resistance (Seconds)	45., min.	T	6.
Tear Strength (lb/in)	25., min.	M	13.
Tensile Strength (psi)	250., min.	M	90.
Viscosity (poise)	100.-6000.	M	56.5 (max.)
Transparent	Yes	T	No

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

d. F-387 is apparently suitable for potting, encapsulating, and conformal coatings. It should be noted that the insulation resistance of F-387 significantly exceeds the 5×10^8 ohms, minimum, required for regular printed circuit boards (Ref. 11-1) and for multilayer printed circuit boards (Ref. 11-2). If a thin coating is desired, it may be possible to dilute the F-387 with a low boiling perfluorinated solvent to obtain a lower viscosity system. (Usage of such solvent will necessitate the frequent agitation of the blend to assure uniformity; settling of the D-604, et al, would be self-defeating. Solvent removal would be essentially complete subsequent to a thermal/vacuum post cure.)

e. The potential ramifications of the arc resistance results may include the usage of F-387, or similar filled systems, as self-indicating (i.e. black track on surface of non-black compound, extending between overloaded circuit components and/or points) electrical coatings, etc., to detect a high voltage dielectric breakdown, while normally functioning as a good electrical insulating, self-extinguishing system. This ancillary application may open up a new field for electronic materials beyond the unique area of manned spacecraft operations; commercial and/or industrial utilization is indicated.

f. Procurement problems (e.g. mislabeled containers, defective catalyst, etc.) with the major constituents of F-387 have already occurred. Tight Quality Assurance standards for these raw materials will be required. Hardness may be a suitable test for the polymer. Melting point may be sufficient for the D-604.

g. The vacuum volatility tests bore out the polymer, 77-033, supplier's caution regarding contamination during processing. This polymer's sensitivity to contamination is typical of the addition reaction silicone RTV's.

h. The basic formulation can be varied with respect to the quantity of Dechlorane 604 in order to optimize other properties while obtaining that degree of flammability protection desired at a given oxygen level. Protection for a 16.5 psia oxygen environment may not be feasible with the basic formulation.

11.2 EVALUATED FORMULATIONS

11.2.1 Fluorocarbon (Conventional) Polymers

a. Conventional fluorocarbon polymers do not lend themselves to the applications herein prescribed (i.e. low temperature cure of thick sections) because of their comparatively high ambient viscosities in the 100% solids state. Viscosity reductions via a) heat, or b) solvent dilution were rejected for potting (primarily) due to the a) high processing temperatures required for flame retardant filled systems for all grades of conventional fluorocarbon considered, and for unfilled grades except the "wax," Viton LM, and b) voids remaining in thick sections, respectively.

b. A Viton LM formulation, F-019, was developed which could be cured at 150°F to a flexible, crack resistant structure. The cured compound appeared to be, at the least, mechanically similar to those Viton LM formulations which "normally" have required cure/post cure temperatures to 480°F. It is believed that this represents an advancement in the pertinent state-of-the-art.

c. The unfilled (i.e. flame retardants) Viton LM formulations did not survive the preliminary flammability tests at 6.2 psia oxygen. Those filled with boric acid withstood the 6.2 psia oxygen. However, the boric acid significantly increased the viscosity. It has been conjectured that the boric acid is complexing with the other ingredients of the formulation.

d. Attempts to lower the viscosity of Viton LM with RTV 602 or 603 were not successful due to the lack of compatibility (i.e. separation occurred) at RTV concentrations high enough to provide an ambient useable blend.

11.2.2 Fluorocarbon (L-2344 : 3M) Polymers

a. Tolylene diisocyanate (TDI) catalyzed L-2344 resulted in good tensile strength, elongation, flexibility, cohesion, and adhesion. However, a minimum of 200°F was required for blending and for cure.

b. Excellent flammability resistance was observed up to, and including, 16.5 psia oxygen when sufficient boric acid was added to L-2344/TDI.

c. A satisfactory balance between 150°F cure time and gelation time was not obtained with the catalytic systems used herein.

d. The use of reactive polyols and isocyanate prepolymers, including those containing bromine and phosphorous, with L-2344 either did not cure, or resulted in brittle structures. A cured brittle product was also obtained when more than 50% A-1100 (gamma-aminopropyltriethoxysilane) was added to L-2344.

e. The blend of silicone RTV's with L-2344 resulted in poor mechanical properties. If the L-2344 was first catalyzed, the flammability was poor. No cure occurred when dibutyl tin dilaurate (i.e. accelerator for TDI) was added. Subsequent investigation revealed that DTD reacts with the catalysts used for the addition reaction silicone RTV's.

11.2.3 Silicone RTV Polymers

a. The commercial silicone RTV's, as received (generally unfilled), did not survive 6.2 psia oxygen. This included F-001, which is essentially the same formulation used for the upward flammability igniter.

b. A number of inorganically filled systems formulated by the Project survived 6.2 psia oxygen. Boric acid loaded 90-052 survived 16.2 psia 60% oxygen/40% nitrogen, but its viscosity was excessive. None of the filled systems withstood 16.5 psia oxygen.

c. None of the silicone RTV's loaded with organic or organic/inorganic flame retardants withstood 6.2 psia oxygen.

d. The condensation reaction silicone RTV's generally had better char structure than the addition reaction types.

e. The following additives, as received, tended to accelerate the cure of addition reaction silicone RTV's:

   Bromotrichloromethane
   Busan 11-MI (barium metaborate monohydrate)
   Dechlorane 510
   Potassium aluminum silicate
   XPI-158 (zinc borate)

f. The following additives, as received, tended to inhibit the cure of addition reaction silicone RTV's:

   Antimony oxide
   FC-134 (cationic fluoro surfactant)
   Hexabromobenzene
   Tetrabromobisphenol A
   Tetrabromophthalic anhydride
   Tetrachlorophthalic anhydride

g. Potentially useful halogenated and phosphorous containing polyols were not miscible with the silicone RTV's.

11.2.4 Fluorosilicone RTV Polymers

a. The cured RTV 77-033 did not survive 6.2 psia oxygen. Boric acid, Dechlorane 604, and a mixture of D-604 and hexabromobenzene withstood 16.5 psia 60/40.

b. The following additives, as received, tended to inhibit the cure of the addition reaction fluorosilicone RTV's:

   Ammonium carbonate
   Ammonium silicafluoride
   ZB-S10 (organo metallic polymer)

c. Based upon the empirical data accumulated by the Project, it is believed that additives which accelerate the addition reaction silicone RTV's will also accelerate the addition reaction fluorosilicone RTV's. The converse (i.e. inhibition) is also deemed applicable.

d. Intrinsic polymer viscosities of the fluorosilicone RTV's are higher than analogous silicone RTV's.

11.2.5 Miscellaneous Polymers

a. The flammability resistance (i.e. preliminary encapsulated wire specimens) of F-387 (and others developed herein) was found to be superior to a rigid "non-flammable" polyester-based potting compound developed for NASA (Ref. 6-24). Thus, F-387 is hardly degraded in the 16.5 psia 60/40 environment, whereas the polyester one is completely destroyed under identical environmental conditions.

b. The polyester approach may still be valid if a non-styrenated (the aforementioned one contains 25% styrene monomer) polyester is used as the matrix.

c. The Project was unable to prepare useful diallyl chlorendate or dimethyl chlorendate formulations to meet the Program's objectives.

11.3 GENERAL

a. The outstanding flammability performance of the Dechlorane 604 formulations is attributed to its comparatively low temperature decomposition, thereby quickly providing its halogen free-radical scavengers, and to a significant low temperature endotherm. Similarly, the superior flammability performance of boric acid in all formulations is believed to be due to its large amount of readily available water, and to its large low temperature endotherm.

b. Boric acid was not used in the final compound because of its poor thermal stability and high solubility in water.

c. Although F-230 and F-379 were somewhat better than F-387 with respect to flammability resistance, a more effective means to isolate the Krytox 143 AC from the polymer is essential before those two compounds can be used. Complete encapsulation will be needed. Carriers, such as molecular sieves, still permit enough of the perfluorinated fluid to reach the polymer and the substrate surfaces.

d. Non reactive diluents were generally not found to be satisfactory since they either left voids upon volatilization, or reduced the formulation's adhesion and cohesion.

e. Based upon the application development activities involved in the preparation of specimens for the NASA-MSC Fire Safety Conference (see Section 10), all of the Final Screening formulations, including F-387, can be readily colored with a broad range of non-toxic pigments. Since these utilize very small amounts of water insoluble inorganic compounds, they should have minimal, if any, effect upon other properties.

f. The NASA-MSC Formulation 12SKI did not pass the 6.2 psia flammability test either in the configuration of the encapsulated wire specimens or as a potted pygmy connector. Also, there was extensive loss in insulation resistance subsequent to the moisture cycle conditioning. As a result of these, and several other deficiencies, it was concluded that this formulation, by itself, was unsatisfactory for the intended usage. An impermeable, flexible, non-flammable top coating would be required if F-12SKI were to be used.

g. Extensive usage of the various molds and test apparatus, especially designed and fabricated for this Program, and their attendant procedures, clearly indicated that they fully met all design expectations and experimental needs.

SECTION 12

RECOMMENDATIONS

a. Establish a prototype/pilot plant production R & D effort for those formulations which have shown via preliminary data that they are potential substitutes for either flexible, but flammable potting, encapsulating, and conformal coating compounds, or rigid, but non-flammable compounds.

b. Include F-387 as a candidate in the above effort.

c. Consider other formulations or their modifications, developed herein, as potential candidates for the prototype/pilot plant program.

d. Examine raw material, processing, storage, and handling variables, as well as their effects upon processing techniques and final properties, with the objective of preparing procurement specifications for the raw material and the resultant compound of the finally selected formulation. Write a process specification for preparing potted components, sub-assemblies, et al, from said formulation. Consider such factors as:

   1) Minimum cure time at ambient and at 150°F with respect to cured properties.

   2) Six month storage life of the formulation on final handling/processing and on cured compound's properties.

   3) Varying quantities of Dechlorane 604 in F-387 on cured and uncured properties.

e. Use an 8 hour/150°F/10^{-4} torr post cure for F-387 if outgassing is critical.

f. Determine suitable solvent and solvent concentration for low viscosity F-387 type systems.

g. Carefully solvent clean molds and other substrates (e.g. wire insulation, connectors, etc.) in contact with F-387 (or other addition reaction silicone or fluorosilicone RTV's) during cure.

h. Determine effects of the non-toxic inorganic pigments, herein utilized, on other properties, especially flammability and insulation resistance (moisture cycle).

i. Optimize F-019 as a base formulation in the development of a low temperature curing, non-flammable Viton LM system which can be used as is, or solvent cut for a coating.

j. Investigate the feasibility of upgrading the mechanical strength of 150°F cured higher molecular weight conventional fluorocarbon polymers than Viton LM (e.g. Viton C-10 or A-35).

k. Formulate and flammability test a D-604 modified F-019.

l. Optimize the cure of L-2344/TDI/D-604.

m. Determine the effect of Tyzor TPT on the cure rate of L-2344/TDI.

n. Formulate and flammability test a D-604 modified RTV 96-052.

o. Formulate and flammability test a D-604 modified liquid, non-styrenated, flexible (and possibly halogenated) polyester.

p. Permanently Teflon coat the metal surfaces of the Flammability Test Specimen Mold which contact formulations.

SECTION 13

REFERENCES

Reference

1-1 MIL-I-0016923F, "Insulating Compound, Electrical, Embedding," 22 July 1971, (epoxy type).

1-2 MIL-S-0023586B, "Sealing Compound, Electrical, Silicone Rubber, Accelerator Required," 7 Nov. 1969.

1-3 MSFC-SPEC-202B, "Compound, Potting and Molding, Elastomeric," 24 Jan. 1969, (urethane type).

1-4 MSFC-SPEC-393A, Amendment 1, "Compound, Printed Circuit Board, Conformal Coating, Elastomeric," 17 Jan. 1966, and MSFC-SPEC-393A, 9 Oct. 1964 (urethane type).

1-5 "What Happened to Apollo," J. B. Campbell, Space Aeronautics, Aug. 1967.

1-6 "Apollo Accident-Part 1," Hearing Before The Committee on Aeronautical and Space Sciences, U. S. Senate, 90th Congress, 7 Feb. 1967.

1-7 Ibid - Part 2, 27 Feb. 1967.

1-8 "Apollo 204 Accident," Report No. 956, Report of the Committee on Aeronautical and Space Sciences, U. S. Senate, 90th Congress, 30 Jan. 1968.

1-9 "How Soon The Moon?," Time, 14 April 1967.

1-10 "Apollo 204 Board Submits Findings," Roundup (NASA-MSC), 14 April 1967.

1-11 "Apollo Fire Report - Oxygen Hiked Before Fire," The Spaceland News Citizen (Clear Lake City, Texas), 18 April 1967.

1-12 "Materials Considerations of Man in Space," H. M. Clancy and R. V. Shelton, North American Rockwell, SAMPE Journal, June/July 1971.

6-1 "Nonmetallic Materials Design Guidelines and Test Data Handbook," MSC-02681, 29 May 1970.

Reference

6-2 Private Communication - H. F. Kline (NASA-MSC) to S. L. Lieberman (Furane), 19 Dec. 1968.

6-3 "The Mechanism of Post Cure of Viton 'A' Fluorocarbon Elastomer," J. F. Smith, Rubber and Plastic Age, 42 No. 1, 59 (1961).

6-4 "Curing Viton with Calcium Oxide," Viton Bulletin No. 11, June 1963, E. I. duPont de Nemours & Co.

6-5 "Ketamine Curing Agents for Epon Resin Coatings," Bulletin SC:62-36, Shell Chemical Co.

6-6 "Hydrofluorocarbon High Temperature Integral Fuel Tank Sealants," W. F. Anspach, AFML-TR-66-399, AFML, April 1967.

6-7 "The Chemistry of Vulcanization of Viton A Fluorocarbon Elastomer," J. F. Smith (E. I. duPont de Nemours & Co.).

6-8 "Development of Organic Nonflammable Spacecraft Potting, Encapsulating and Conformal Coating Compounds," Contract No. NAS 9-8750, Progress Report No. 3, 25 March 1969, S. L. Lieberman (Furane).

6-9 "Flame Retardance of RTV Compounds," TSR-1725, G. E., 4 April 1968.

6-10 Ibid, Progress Report No. 6, 22 April 1969, S. L. Lieberman (Furane).

6-11 Ibid, Progress Report No. 7, 22 May 1969, S. L. Lieberman (Furane).

6-12 "Hexafluoroisopropanol," Technical Report DP-4A, duPont, 1968.

6-13 W. J. Middleton and R. V. Lindsey, Jr., J. Am. Chem. Soc., 86, 4948, 1964.

6-14 "Properties of 'Union Carbide' Silane Coupling Agents," Bulletin PIB 15-17, UCC, Dec. 1966.

6-15 Telecon, R. Campbell (Dow Corning) to S. L. Lieberman (Furane), 8 May 1969.

6-16 Telecon, R. Campbell (Dow Corning) to S. L. Lieberman (Furane), 3 June 1969.

6-17 Telecon, J. Wessel (Dow Corning) to S. L. Lieberman (Furane), 30 Oct. 1968.

Reference

6-18 DC 94-531 Bulletin, Dow Corning, 2 Feb. 1971.

6-19 "How to Process Sylgard Brand Resins," Dow Corning.

6-20 "Sylgard 188 and 189 Potting and Encapsulating Resin", Bulletin 07-284, Dow Corning, June 1968.

6-21 "Engineers Guide to Sylgard Resin Electronic Packaging Materials," Bulletin 07-104, Dow Corning, 1964.

6-22 "Sylgard 182 Potting and Encapsulating Resin," Bulletin 07-214, Dow Corning, Aug. 1966.

6-23 Telecon, S. L. Lieberman (Furane) to T. Martins (Grumman Aircraft Co.), 10 Nov. 1968.

6-24 Brit. Pat. 990,472 (1965), Raytheon Corp.

6-25 "Antimony Oxide" Bulletin, Harshaw Chemical Co.

6-26 Telecon, D. Stuck (Dow Corning) to S. L. Lieberman (Furane), 1 April 1970.

6-27 "Boric Acid," Technical Data Sheet IP-14, U. S. Borax & Chemical Corp.

6-28 "Boron Compounds As Fire Retardants In Coating Materials," W. G. Woods and J. G. Whiten, U. S. Borax Research Corp., 3 June 1968.

6-29 "Development of Inorganic Non-Flammable Spacecraft Potting, Encapsulating, And Conformal Coating Compounds," S. H. Foster and K. H. Lothrop, Final Report NAS 9-8749, Emerson & Cuming, Inc., 1 Oct. 1969.

6-30 Telecon, S. L. Lieberman (Furane) to J. Stuart (Marco Div., W. R. Grace), 25 Feb. 1970.

7-1 MSFC-SPEC-202A, "Compound, Potting and Molding, Elastomeric," dated 11 Dec. 1964.

7-2 Telecon, S. L. Lieberman (Furane) to D. Stuck (Dow Corning), 1 March 1971.

7-3 "DECHLORANE 604," Preliminary Data Sheet No. 349, Industrial Chemicals Div., Hooker Chemical Corp., 24 Aug. 1970.

References

8-1 ASTM Test Method D-257-66, Appendix A1.

8-2 Federal Test Method Standard No. 601, Methods 4111, 4121, and 4211.

8-3 "DOW CORNING 77-033 Experimental Fluorosilicone Sealant," New Product Information, Dow Corning Corp., 5 Aug. 1969.

8-4 Telecon, S. L. Lieberman (Furane) to R. Campbell (Dow Corning), 12 March 1971.

9-1 NAS 9-8750, Amendment 6S, 16 June 1969.

9-2 Correspondence, H. F. Kline (NASA-MSC) to S. L. Lieberman (Furane), 7 Oct. 1969.

9-3 Telecon, H. F. Kline (NASA-MSC) to M. W. Finnie (NASA-WSTF), 19 Nov. 1969.

10-1 Telecon, H. F. Kline (NASA-MSC) to S. L. Lieberman (Furane), 26 March 1970.

11-1 MIL-P-55110B Amendment 2, "Printed Wiring Boards," 18 April 1969, and MIL-P-55110B, "Printed Wiring Boards," 2 Oct. 1968.

11-2 MIL-P-55640A, "Printed Wiring Boards, Multilayer (Plated-Through Hole)," 29 Oct. 1970.

APPENDIX

- A -

NAS 9-8750

STATEMENT OF WORK

NONFLAMMABLE SPACECRAFT POTTING, ENCAPSULATING, AND CONFORMAL COATING COMPOUNDS

(Revised: 9 Nov. 1970)

TABLE OF CONTENTS

1.0 Introduction

The presently available polymeric potting, encapsulating, and conformal coating compounds, although suited to their conventional applications in ambient air, do not provide adequate flame safety for use in the cabin oxygen environment of manned spacecraft. The primary flame propagation hazard is associated with the relatively large surface areas of conformal coatings which are required to provide dielectric protection and moistureproof terminations for spacecraft wire harnesses at terminal strips and circuit breaker panels. Only slightly less serious is the combustibility of present electrical potting compounds used in the pure oxygen atmosphere. Encapsulating compounds may be classed as less critical, since they are normally used in sealed components and are protected from direct contact with ambient oxygen.

The flammability of these present compounds requires that they be encapsulated with relatively thick layers of inorganic coatings which may be brittle, tend to flake and crack, add substantial weight, and hamper any wiring rework required.

2.0 Objectives

It is required that new potting, coating, and encapsulating compounds be developed which will not sustain flame when subjected to the special oxygen flammability tests described in this statement of work. These compounds must not evolve toxic gases at elevated temperatures, and the toxicity of pyrolysis products must be minimal. They must exhibit low volatility at operating temperatures in both 5 psia nominal oxygen atmosphere and vacuum. See requirements of paragraph 3.4 for compliance with these objectives.

Additional requirements described in this statement of work include the usual stringent electrical and physical properties, high bond strength to etched Teflon and Kapton wire, and ease of repair and rework.

A desirable goal is to obtain a clear or transparent potting and coating compound to allow post-fabrication inspection.

3.0 Technical Requirements

3.1 Approach

The requirements of nonflammability, low volatility, and low toxicity of pyrolysis products may be achieved by any of the standardized techniques, by unique and hitherto unproven methods, or by any combination thereof. The study should include investigation of conventional flame inhibition techniques such as addition of halogen bearing compounds singly or in synergistic combination with other components such as antimony trioxide, with special emphasis on anticipated volatility/toxicity problems. The use of nontoxic inorganic fillers as flame retardant "heat sinks", and organometallic curing agents should also be considered.

3.2 Materials

3.2.1 Basic Compounds - Any materials inherently possessing adequate electrical and physical properties may be considered. A detailed literature search and compilation of state-of-the-art test data is required to justify the proposed ultimate choice or choices as a starting point for the development program.

3.2.2 Primers - The use of a primer for potting, encapsulating, and/or conformal coatings, to improve the adhesion to the base structure is permitted, provided the composite primer and basic compound successfully pass all tests of paragraph 3.4.

3.2.3 Toxicity - Any potential toxic materials associated with the processing or handling of the compounds shall be identified, and appropriate safety precautions shall be recommended.

3.2.4 Clarity - It is desirable for the cured product to be as transparent as possible to allow post-fabrication inspection for bubbles or voids. No special pigments or color additives shall be used.

3.3 Performance and Product Characteristics

3.3.1 Appearance - The as-poured and cured compounds shall be homogenous and free from lumps and coarse particles and voids.

3.3.2 Application - Potting and encapsulating compounds shall be capable of being readily applied by an injection or extrusion gun. Conformal coatings shall be capable of being applied by brush, spray or dipping.

3.3.3 Application Life - It is desired that the compound shall be suitable for application for a minimum of 60 minutes, but a minimum of 30 minutes is required.

3.3.4 Curing Time - The tack life of the compound should not exceed 6 hours at room temperature, but a maximum of 12 hours is required. The curing time to obtain optimum properties shall not exceed 7 days at room temperature and 50 percent maximum humidity. Elevated temperatures up to 150°F may be used to accelerate the cure time. A vacuum post cure, not to exceed 8 hours at a maximum of 150°F, may be utilized when necessary to meet the requirements for the compound.

3.3.5 Storage Life - The uncured compounds shall be capable of meeting the requirements of this statement of work for 6 months when stored below 70°F.

3.3.6 Repair and Rework - The cured compounds must be capable of being removed, either mechanically or chemically, without damaging wiring, solder joints, or electronic components. The adherence of patch potting to previously applied potting must be adequate to withstand the 200 megohm moisture resistance test of paragraph 3.4.5.

3.3.7 Temperature Rating - The compounds shall retain useful electrical properties at 212°F, as verified by the applicable insulation resistance tests of paragraph 3.4.5.

3.3.8 Decomposition Temperature - A Thermogravimetric Analysis (TGA) shall be performed from ambient to 932°F on the 10 formulations selected by the contractor for final screening, and the data shall be immediately submitted to the MSC Technical Monitor.

3.4 Properties and Test Requirements

3.4.1 Flammability - Candidate materials shall be self-extinguishing with no further evidence of degradation by combustion in 16.5 psia, 60% oxygen/40% nitrogen when tested as follows:

3.4.1.1 Overload Ignition - Potting and encapsulating compounds shall be tested in accordance with Test No. 5, Appendix A. Conformal coatings shall be tested in accordance with Test No. 17, Appendix A. Power shall be discontinued at first indication of ignition of either wire insulation or compound.

3.4.1.2 Upward Propagation - The compounds shall be tested in accordance with Test No. 1, Appendix A. As an alternative for potting and encapsulating compounds, the following test may be used:

Prepare electrical connector potting sample as instructed in Test No. 5 of D-NA-0002. This connector shall be a Bendix Pygmy PT07CP-18-115 as shown in Figure 2 of Test No. 5. Mount an identical connector above first connector in an aluminum panel in close proximity to the first connector. An igniter shall be prepared as follows:

Positive igniter for flammability tests
The igniter shall be a silicone rubber rod, wound six to eight turns of 20 AWG nichrome wire having a nominal resistivity of 0.7 ohms per foot. The silicone rubber rod shall be prepared according to the following procedure:

82.5 parts by weight of RTV-560 shall be blended with 17.5 parts by weight of RTV-577. To the mix, 0.5 parts by weight of Thermolite T-12 shall be added and thoroughly mixed. The blend shall immediately deairated in a vacuum of not less than 28 inches of mercury for a period of not more than ten minutes. The compound shall then be cast into a mold which will provide rods, when cured and finished, of 0.22 inches diameter and of sufficient length to give a burn time 25 ± 5 seconds in the applicable atmosphere. The compound shall be cured either for 24 hours at room temperature or for 4 hours at room temperature followed by 10 hours at 130 ± 10° F. The cured material shall show a shore "A" durometer reading of not less than 40.

Apply this igniter to the lower surface of the potting material of the bottom connector. Apply electrical current to the nichrome wire starting with 5 amperes and increasing each 30 seconds thereafter until the potting material catches on fire or the nichrome wire fails. The material passes if the lower connector burns but does not ignite the upper connector or if the lower connector fails to ignite. If the upper connector burns the material fails to pass.

3.4.2 Outgassing - As a screening test, the total organics expressed as pentane equivalents evolved from any of the candidate potting, coating, or encapsulating materials shall not exceed 100 micrograms per gram of sample, when tested at 5.0 psia oxygen in accordance with Test No. 6, Appendix A. Outgassed carbon monoxide shall not exceed 25 micrograms per gram when tested in accordance with the same test. Identification of major peaks, during analysis in 5 psia oxygen are required prior to final MSC acceptance of candidate materials. MSC acceptance shall be predicated upon the requirement that none of the outgassed products shall constitute a toxic hazard to the crew, based upon the criteria of acceptability of Test No. 6, Appendix A.

3.4.3 Odor - The total odor rating of candidate material must not exceed 25, for the sum of 10 odor evaluations, when tested at 5.0 psia oxygen in accordance with Test No. 7, Appendix A.

3.4.4 Vacuum Volatility - The rate of weight loss in $1X10^{-4}$ Torr at 150°F shall not exceed 0.1 percent per hour when measured continuously in vacuum with an in-situ microbalance. The total weight loss for the initial 24 hour period shall not exceed 1.0 percent.

There shall be no visible condensate deposited on a glass slide maintained at 100°F and located in close proximity to the test specimen within the vacuum chamber.

An additional vacuum volatility test specimen shall be prepared per MSFC-SPEC-202A, paragraph 4.4.3.11 (See Appendix C) for potting compounds, and per MSFC-SPEC-393A, paragraph 4.5.2.10 (See Appendix D) for conformal coatings. Upon completion of 24 hours vacuum testing, there shall be no apparent visual degradation, and the compounds shall withstand the required 200 megohm moisture resistance tests of paragraph 3.4.7, Appendix C, for potting compounds, and paragraph 3.4.6, Appendix D, for conformal coatings.

3.4.5 Physical and Electrical Properties

3.4.5.1 Potting and Encapsulating Compounds - Candidate potting and encapsulating compounds shall meet the requirements of Table I and MSFC-SPEC-202A, paragraph 3.4.6 (See Appendix C), when tested in accordance with the applicable test methods of paragraph 4.4, Appendix C. Test results shall be submitted for NASA-MSC review.

TABLE I

REQUIREMENTS

PROPERTIES	T/M (1)	VALUE
Dielectric Constant	T	5.0, maximum
Power Factor	T	0.09, maximum
Dielectric Strength (50 mils)	M	350 volts/mil, minimum
Volume Resistivity (ambient)	T	(P) 1×10^{12} ohms-cm., minimum (C) 1×10^{11} ohms-cm., minimum
Surface Resistivity	T	1×10^{12} ohms, minimum
Arc Resistance	T	45 seconds, minimum
Insulation Resistance (ambient)	M	100,000 megohms, minimum
Insulation Resistance (212° F)	M	750 megohms, minimum
Insulation Resistance (moisture cycle)	M	200 megohms, minimum
Tear Strength	M	25 lb./in., minimum
Tensile Strength	M	250 psi, minimum
Elongation	M	125%, minimum
Shrinkage	M	3%, maximum
Hardness (after full cure)	M	50-85 (Shore A)
Viscosity (77°F)	M	100 to 6,000 poises
Specific Gravity	T	2.2, maximum

(1) T - Target Value

M - Mandatory Value

(Rev.: 9 Nov., 1970)

3.4.5.2 Conformal Coatings - Candidate conformal coatings shall meet the requirements of Table I and MSFC-SPEC-393A, paragraph 3.4.5 (See Appendix D) when tested in accordance with the applicable test methods of paragraph 4.5, Appendix D. Test results shall be submitted for NASA-MSC review.

3.4.5.3 Bond Strength to Etched Wire - The bond strength of both potting and conformal coating compounds to etched Teflon and Kapton wire must exceed ten (10) pounds pull-out, and the failure must be cohesive (in the compound) rather than adhesive (at the wire-compound interface), when tested per Appendix B.

3.4.5.4 Patch Potting Adherence - The adherence of patch potting or coating material to previously applied compounds must be adequate to withstand the 200 megohm moisture resistance tests of paragraph 3.4.7, Appendix C, for potting compounds, and paragraph 3.4.6, Appendix D, for conformal coatings. The test samples shall be prepared with the required electrodes encapsulated by the applicable candidate compound. The upper portion of the cured compound shall be removed to a depth of at least one inch (1.00"), exposing both electrodes, to demonstrate compliance with paragraph 3.3.6. Patch potting or coating shall then be applied to replace the original compound, in accordance with the recommended process. The moisture resistance test shall be conducted on the " repaired" specimen to demonstrate dielectric integrity of the repair.

APPENDIX

- B -

ELECTRICAL CONNECTOR

POTTING

FLAMMABILITY TEST (1)

---

(1) Test No.5 of MSC-D-NA-0002, "Procedures and Requirements for the Flammability and Offgassing Evaluation of Manned Spacecraft Non-metallic Materials."

TEST NO. 5

ELECTRICAL CONNECTOR POTTING FLAMMABILITY TEST

1.0 PURPOSE

This test evaluates the flammability characteristics of spacecraft potting compounds in a specific gas atmosphere environment. It is designed to simulate a short circuit or dielectric breakdown on current carrying wires, or connector contacts within the potting used to environmentaly seal electrical connectors.

2.0 TEST CONDITIONS - PRESSURES AND ATMOSPHERE

The test pressure and gas mixture conditions for the pertinent materials category shall be designated by the requirements for each program.

3.0 TEST DISCIPLINE

3.1 Each test shall be directed by the cognizant Test Engineer or his appointed alternate.

3.2 Approval of the test shall be indicated at the end of the test procedure by the signatures of the responsible Test Engineer. Entires transcribed to the test data sheets will also be verified by the Test Engineer.

4.0 CRITERIA OF ACCEPTABILITY

Connector potting material shall not be capable of sustaining combustion in the designated test atmosphere following removal of the ignition source with current overloads up to the melting point of the electrical wire conductor.

5.0 TEST EQUIPMENT

5.1 Test Chamber - Test chamber shall have sufficient volume to ensure complete combustion of the potting compound specimen and shall be suitably constructed and protected to ensure safe operation. A window or viewing port for visual observations shall be provided. A test chamber shall contain inlets for vacuum, source of power for wire overload, air, oxygen, and gas mixture. A horizontal sample holder and a central connector mount shall be included and positioned within the test chamber. See Figure 1.

5.2 Pressure Gauge - The pressure gauge shall be capable of measuring absolute pressure with an accuracy of ± 0.1 Torr.

5.3 Gas Supply - The gas shall be commercially available conforming to appropriate specifications.

5.4 Sample Holder and Connector Mount - The sample holder shall consist of two horizontally mounted electrical connections (bolt with knurled nuts) spaced 12 inches apart. The electrical terminals shall be connected to the ignition power source. A central connector consisting of a vertical panel drilled to receive a horizontal Bendix PT07 Jam Nut Receptacle or equivalent shall be provided.

5.5 Ignition Source - An external power supply shall be provided which is capable of providing a large steady DC current so that a very high temperature will be achieved quickly. The power source must be capable of supplying 100 amperes of current through a 18 AWG fourteen inch long wire.

6.0 SAMPLE PREPARATION

6.1 Prepare three (3) samples for each candidate potting compound per paragraphs 6.2 through 6.8 below.

6.2 Verify material identifications as one of the items below:

6.2.1 Manufacturer's Certification
6.2.2 NASA Certification
6.2.3 Contractor Certification
6.2.4 Definite Identification not available

6.3 A fourteen inch (14") length of AWG 18 white teflon insulated wire (MIL -W-16878, Type E, or MIL -W-22759, Type MS21985) shall be prepared as follows:

6.3.1 Form a "U" bend in the middle of the wire and etch the center 4 inch per MSC Specification Q-3.

6.3.2 Strip one half inch insulation from each end.

6.3.3 Cut the wire in half such that two inches of etched insulation remains on the unstripped end of each seven inch length.

6.4 Obtain a Bendix Pygmy PT06CP-18-11P Straight Plug or equivalent. Strip 1/4" from the end of each 7" wire prepared per 6.3. Crimp one 7" wire to contact K and the second 7" wire to adjacent contact L.

6.5 Crimp nine (9) etched white AWG 20 Teflon insulated wires, each 3" long, in the remaining contacts.

6.6 Place the potting boot on the connector and fill with the candidate potting compound, per the manufacturer's instructions. Ensure that the potting compound is within recommended shelf life. Ensure that all steps are followed exactly as they would be in flight hardware, including cleaning and priming of connector rear insert for bondability, degassing potting compound, proper humidity control, etc.

6.7 Cure per applicable user's procedure or manufacturer's recommended time/temperature for optimum properties.

6.8 Remove potting boot.

7.0 CENTRAL CONNECTOR PREPARATION

7.1 Prepare three (3) Central Connectors per paragraphs 7.2 through 7.5 below.

7.2 Obtain a Bendix Pygmy PT07CP-18-11S Jam Nut Receptacle or equivalent. Crimp a short AWG 16 Teflon insulated jumper between contact K and contact L.

7.3 Place the potting boot on the connector and fill with the compound to be tested.

7.4 Cure per manufacturer's instruction.

7.5 Remove potting boot.

8.0 TEST PROCEDURE

The procedure shall be carried out in the following sequences:

8.1 The sample prepared per Paragraph 6.0 shall be mounted in the sample holder by locking the PT06CP plug to the PT07CP receptacle which has been placed in the vertical panel provided for it, and fastened with a threaded locknut. The stripped ends of the 7 inch lengths of wire crimped into the plug shall be fastened to the current supply terminals of the sample holder.

8.2 The test chamber shall be evacuated to a pressure of 1 Torr and repressurized to the test pressure with oxygen. Allow the chamber to stand for one minute. A leak is indicated if an increase in test chamber pressure of 1 Torr is observed during one minute after the vacuum pump is closed off from the system. The system shall be brought to atmospheric pressure and the leak corrected before any additional tests are carried out. Repeat the above procedure.

8.3 After the test chamber has been stabilized at the test pressure, soak the specimens ten minutes. Apply a current of 55 amperes to the wire. If ignition or considerable degradation is not obtained in one minute, the current shall be increased by 5 amperes (i.e., from 55 to 60 and 60 to 65, etc.) until such time as the wire fails or ignition occurs. If the wire fails, voltage shall remain applied to the open wire until it is positively established that current does not flow by bridging the gap to adjacent conductors.

8.4 Three samples of each potting compound shall be tested. The failure of any one sample to meet the criteria of Paragraph 3.0 shall be cause for rejection of that compound.

9.0 REPORT

The following test data and pertinent information concerning the materials shall be reported:

9.1 Name and positive identification (para. 6.2) of the material

9.2 Vendor designation and vendor.

9.3 Usage, quantity, and surface area in the spacecraft.

9.4 Test pressure and test atmosphere.

9.5 Results of each test including combustion phenomena if ignition occurs.

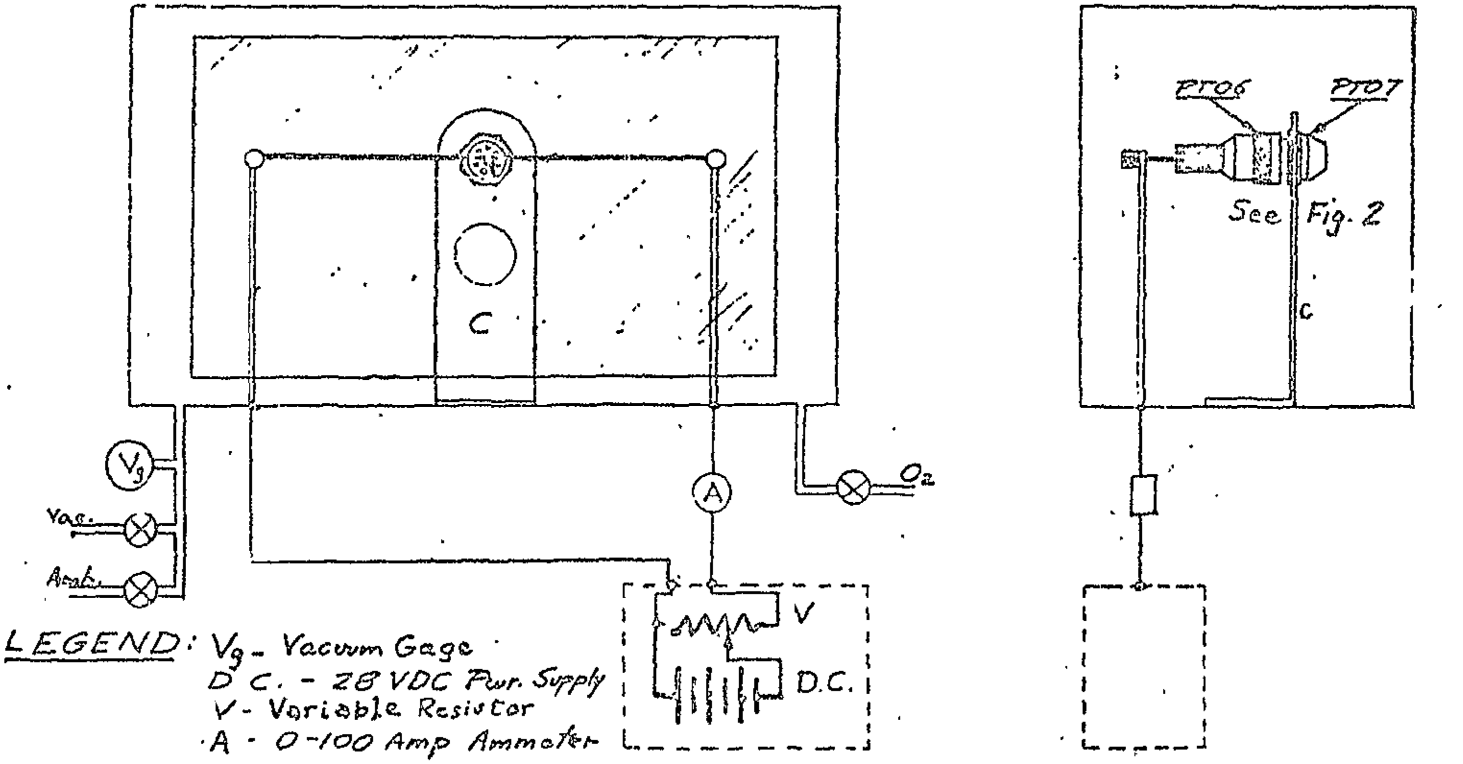
9.6 Date of test.

9.7 Test number.

9.8 Identity of the testing agency.

9.9 Names & signature of Test Coordinator.

FIG. 1 POTTING FLAMMABILITY TEST EQUIPMENT SCHEMATIC

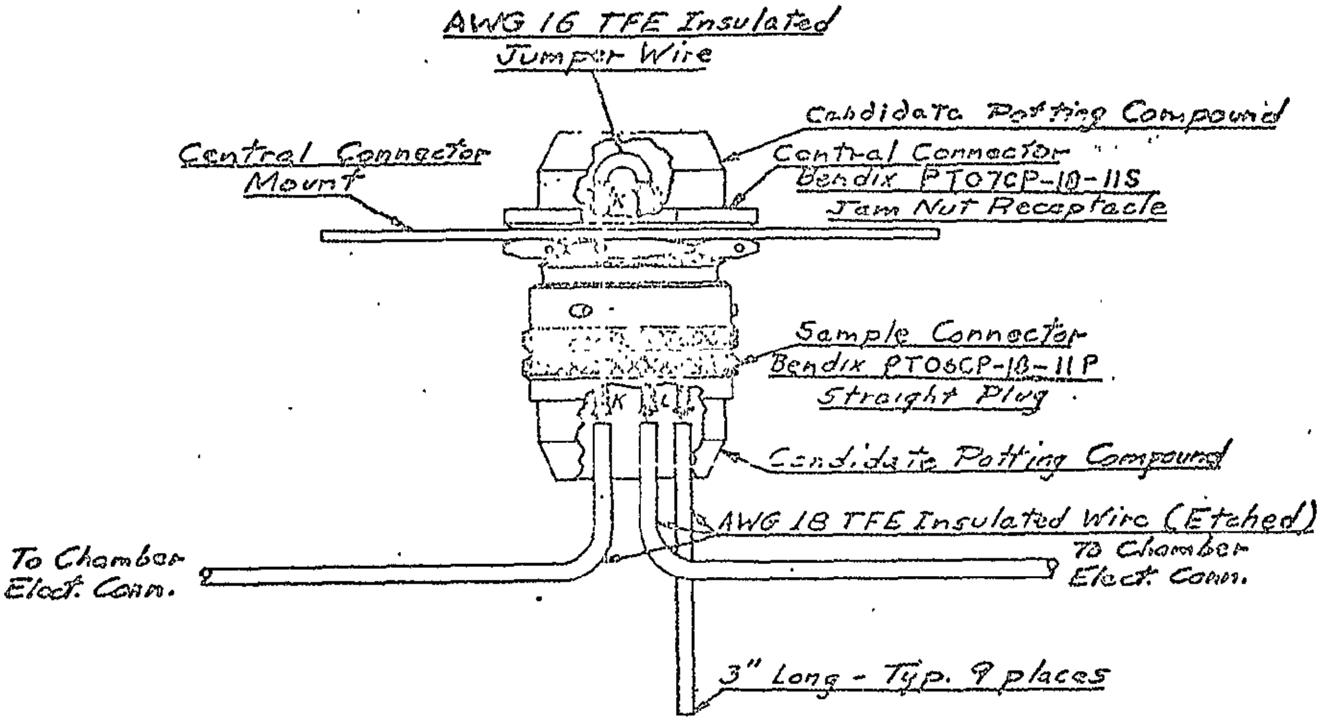


LEGEND: V_g - Vacuum Gage
D.C. - 28 VDC Pwr. Supply
V - Variable Resistor
A - 0-100 Amp Ammeter

C - Central Connector Mount
PT06 - Bendix PT06-CP-10-11P Straight Plug
PT07 - Bendix PT07-CP-10-11S Jam Nut Receptacle

aww
9-13-66

FIG. 2 SAMPLE CONFIGURATION



aww
9-13-65

APPENDIX

- C -

DETERMINATION OF ORGANIC OFFGASSING PRODUCTS AND CARBON MONOXIDE (1)

---

(1) Test No.6 of MSC-D-NA-0002, "Procedures and Requirements for the Flammability and Offgassing Evaluation of Manned Spacecraft Non-metallic Materials."

TEST NO. 6

DETERMINATION OF ORGANIC OFFGASSING PRODUCTS AND CARBON MONOXIDE

NOTE: This test will not be conducted prior to applicable flammability tests nor will it be conducted on materials which have failed a flammability test unless specifically requested.

1.0 PURPOSE

This procedure establishes the criteria for a screening test, which will determine the suitability of nonmetallic materials for use in the space vehicle crew compartment environments. The criteria is established with respect to production by out-gassing of potentially toxic or objectionable volatiles. The volatiles are separated into two categories: Carbon Monoxide and Total Organics.

2.0 TEST CONDITIONS - PRESSURES & ATMOSPHERE

The test pressure and gas mixture conditions for the pertinent materials category shall be designated by the requirements for each program.

3.0 CRITERIA OF ACCEPTABILITY

3.1 The reporting laboratories shall report total organics as micrograms per gram ($\mu g/g$) of sample material using methane as a standard.

3.2 The maximum allowable level of total organics in the tested configuration shall not exceed 100 micrograms of total organics per gram of sample.

3.3 Carbon monoxide shall be reported as the number of micrograms produced by one gram of sample material.

3.4 The maximum allowable level of carbon monoxide in the tested configuration shall not exceed 25 micrograms of carbon monoxide per gram of sample.

4.0 TEST DISCIPLINE

4.1 Each test shall be directed by the cognizant test engineer or his appointed alternate.

4.2 Approval of the test shall be indicated at the end of the test procedure by the signatures of the responsible test engineer. Entries transcribed to the test data sheets will be also verified by the test engineer.

5.0 TEST EQUIPMENT

5.1 Test Chamber - The test chamber shall have a minimum main chamber volume of two liters. It shall have a configuration and be fabricated of materials which allow ready cleaning. A thermometer or thermocouple and pressure gauge for temperature and pressure determination, respectively, shall be included in the test chamber. The chamber shall be connected to a manifold system to permit evacuation, pressure readings, gas introduction, and sample withdrawal. An alternate system would be to connect the chamber directly to a gas chromatograph and/or mass spectrometer for the gas analyses.

5.2 Heating Source - The oven shall maintain constant temperature control over the test chamber to ±5°F. The temperature during sample exposures shall be recorded.

5.3 Vacuum Pump - The vacuum pump shall be capable of producing a vacuum less than one Torr.

5.4 Analytical Equipment - The analytical equipment shall consist of the following types of equipment and any other instruments which the tester desires to use efficiently to evaluate offgassing products.

5.4.1 Gas Chromatograph System - Recorder, thermal conductivity and hydrogen flame ionization detectors. The columns shall have the capability of separating light organic and inorganic gases, organic sulfides and mercaptans, halogenated hydrocarbons, representative aliphatic and aromatic hydrocarbons including aldehydes, ketones, alcohols, and esters. The hydrogen flame ionization detector has greater sensitivity to organic materials. Conversely, because of the flame ionization detector's lack of sensitivity to the inorganic compounds listed above, the thermal conductivity detector is used for their identification.

5.4.2 Recording Infrared Spectrophotometer capable of analyzing 3 microliters ($3\mu l$) or less of liquid with accessories which include an infrared gas cell with 10m path length.

5.4.3 Mass Spectrometer

5.4.4 Electron Capture Detector

5.4.5 Trapping System suitable for trapping and transfer of microliter quantities of liquid from the gas chromatograph to the infrared spectrophotometer or mass spectrometer. A direct G. C. mass spectrometer connection can replace the trapping system if desired.

5.4.6 Gas Sampling System suitable for transfer of measured volumes of gas samples from the test chamber to the gas chromatograph.

5.4.7 Calibration Gas Samples as required to quantify detector sensitivity and readout. These may be bought from a manufacturer or made up in the laboratory.

6.0 PREPARATION OF TEST EQUIPMENT

6.1 Prior to loading of sample into a container, the container shall be loaded to test pressure with test atmosphere, heated at 155° ± 5° F for 24 hours, and the gas analyzed for total organics and carbon monoxide. The container shall be certified clean for use if the total organics value is 5 ppm by volume methane or less over the test atmosphere and the carbon monoxide value is 5 ppm by volume or less. After use, the container shall be reused without cleaning if the sample values are equal to or less than the above. If not, the container shall be heated and purged with air or nitrogen by some convenient method such as a heat gun with blower, loaded to test pressure with test atmosphere, and tested to the above specifications.

6.2 Leak Check - The test system shall not increase in pressure more than one Torr. while remaining at a reduced pressure of one Torr for a time period of one hour.

7.0 PRETEST PROCEDURE

7.1 Verify material identification as one of the following:

- 7.1.1 Manufacturer's certification
- 7.1.2 NASA certification
- 7.1.3 Contractor certification
- 7.1.4 Definite identification not available

8.0 TEST SPECIMENS

8.1 All the materials tested in the program shall be classified into four categories; surface, volume, weight, or specialized items.

8.2 Samples based on Surface

This classification is defined as all those materials that are essentially two-dimensional. This would include films, fabrics, coatings, finishes, inks, primers, adhesives, thin film lubricants, tapes, and electrical insulating material.

The sample tested shall have a surface area of 46.5 ± 2.5 square inches per liter of test container. Coatings, finishings, etc. shall be coated on clean aluminum substrate of 0.020 ± 0.01 inch thickness. Material thickness, curing process, and method of application shall be in accordance with the manufacturer's recommendations. Material may be coated on both sides of the aluminum panel. Tapes and other similar materials with an adhesive surface shall be fastened to a similar aluminum panel. In all cases, only the outer surface of a material on the aluminum panel is counted in the surface area determinations. Films, fabrics, and similar materials shall be cut to give 46.5 ± 2.5 square inches surface area. Since these materials are two-surfaced in use, both the top and bottom surface shall be counted in determining total surface area. Heat shrinkable tubing shall be applied and shrunk to simulate actual use configuration.

8.3 Samples based on Volume

This classification is defined as all those materials having an indefinite volume but having a large real surface area due to surface convolutions or matting. These shall include foams and other blown or foamed materials and insulation padding.

Samples of these materials shall be cut to a thickness of 0.50 ± 0.05 inches unless the existing thickness is less than 0.40 inches. In this case, the existing thickness shall be used. The material shall be cut to such a size as to give 7.75 ± .75 square inches of total surface per liter of test container volume. All surface, tops, bottoms, and sides shall be used to compute total surface area. In cases where the natural thickness is such that the material cut would be too large to be placed into the container, two or more pieces may be cut as long as the total surface area requirement is met.

8.4 Samples based on Weight

This classification is defined as all those materials having a definite bulk and not falling into the volume classification. This shall include potting compounds, molding compounds, cast or formed objects, solid wires, and thick plastics. Liquids that are not used or applied as coatings or thin films shall be included.

The samples shall be used as much as possible in the supplied configuration and cut to give 5.0 ± 0.25 grams per liter of test container. Potted or molded materials shall be prepared and cured per manufacturer's directions and cut to weight. Liquids shall be placed in an aluminum dish 2.25 ± 0.25 inches in diameter. Sample weight shall be 5.0 ± 0.25 grams per liter.

8.5 Specialized Items

It must be recognized that some materials will not meet the above classifications and must be specially handled. This will most often occur with nonhomogeneous materials. These cases will be tested in the manner designated by the Test Engineer. The manner of testing and sample preparation shall be fully reported.

9.0 PROCEDURE

9.1 Purge the test chamber until the minimum test atmosphere concentration is 95 percent and begin heating. Bring the chamber to 155° ± 5°F and adjust the chamber pressure to test pressure.

9.2 After twenty-four hours, check the test gas for contaminants with the gas chromatograph. Proceed to 9.3 if the total contamination is less than 5.0 ppm by volume over the test atmosphere. If the contamination exceeds the above value, the test chamber shall be flushed and rechecked after an additional twenty-four hours. This procedure shall be continued until the contamination is below the specific level of 5 ppm. If the gassing contamination continues indefinitely, revision of the test chamber is indicated.

9.3 Place a clean weighed specimen prepared per Section 8.0 in the test chamber.

9.3.1 The chamber shall be evacuated through a liquid nitrogen trap to below one Torr. The chamber shall then be closed off and the test gas, filtered through a 5X Molecular Sieve, bled into the chamber to test pressure. The conditions of exposure are held at 155° ± 5° F for a total period of 72 hours. Following the designated exposure period, the chamber is brought to room temperature. After the final gas samples are taken, the test specimen is removed from the chamber and weighed. The gases are then analyzed for the following:

9.3.1.1 Total organics expressed as methane equivalents.

9.3.1.2 Outgassing components exceeding $10 \mu g/g$ including but not limited to the following: HCN, Benzene, Xylene, MEK, Chloroform, n-Butanol, Dichloromethane, 1-4 Dioxane, Formaldehyde Trichloroethylene, HCL, Ammonia, Hydrogen Fluoride, Carbonyl Fluoride and Silicon Tetrafluoride. Condensates in the cold trap may be recovered as gas and likewise analyzed and plotted.

9.3.1.3 Quantity of carbon monoxide evolved.

9.4 Determination of Total Organics - The gas chromatograph with the proper columns, Beckman Total Hydrocarbon Analyzer or an equivalent instrument may be used for the determination.

9.5 Outgassing components, including those listed in 9.3.1.2, that exceed $10 \mu g/g$ shall be identified and recorded in the reporting format.

9.6 Determination of Carbon Monoxide - The carbon monoxide content of the evolved gases shall be determined under Section 9.4 above by utilizing an appropriate separation and analytical technique having the sensitivity to detect within $0.5 \mu g/g$ CO.

9.7 All charts and data are to be retained in the event further identification or evaluation is necessary.

10.0 REPORTING DATA

10.1 The following test data and pertinent information concerning the material tested shall be reported:

10.1.1 Name and positive identification of the material (paragraph 7.1)

10.1.2 Vendor designation and vendor

10.1.3 Usage, and surface area in the spacecraft

10.1.4 Weight and size - length, width and thickness of sample tested

10.1.5 Test pressure and atmosphere

10.1.6 Results of tests

    10.1.6.1 Carbon monoxide in micrograms per gram

    10.1.6.2 Total organics in micrograms per gram

    10.1.6.3 Identity of organics greater than 10 micrograms/gm

    10.1.6.4 Weight loss, percent.

10.1.7 Name and number of test procedure

10.1.8 Date of test

10.1.9 Test number

10.1.10 Identity of the testing agency

10.1.11 Name and signature of test coordinator.

APPENDIX

- D -

ODOR TEST (1)

---

(1) Test No.7 of MSC-D-NA-0002, "Procedures and Requirements for the Flammability and Offgassing Evaluation of Manned Spacecraft Non-metallic Materials."

TEST NO. 7

ODOR TEST

1.0 PURPOSE

These procedures are designed to eliminate unsuitable materials from use in the habitable area of spacecraft. A material that fails these requirements shall not be used.

2.0 TEST CONDITIONS - PRESSURES AND ATMOSPHERE

The test pressure and gas atmosphere conditions for the pertinent materials category shall be designated by the requirements for each program.

3.0 ODOR CRITERIA

3.1 Selection of Test Panel for Odor Evaluation - The test conductor shall establish a pool of qualified personnel.

3.1.1 Members of the pool shall be male and each member be capable of detecting seven basic odors from the following solutions:

Primary Odor	Standard Compound	Amount Dilution in Water
Ethereal	1, 2 - dichoroethane	0.4 ml in 500 ml
Camphoraceous	1, 8 - cineole	5μl in 500 ml
Musky	15 - hydroxypentadecanoic acid lactone	1 mg in 1,000 ml
Floral	1 - methyl-1-ethyl-2-phenyl propanol-1	0.075 ml in 500 ml
Minty	menthone (dl)	2μl in 333 ml
Pungent	formic acid	25 ml of 90 percent solution in 500 ml

Putrid (methyl disulphide) methyl dithiomethane 1μl in 10,000 ml

3.1.2 Members of the pool shall be given three odorless solutions along with the seven primary standards for the detection of odor.

3.1.3 The solutions shall be freshly prepared once a month or as needed.

3.1.4 The established pool for odor evaluation shall be requalified every three months.

3.1.5 A panel of five or ten members shall be selected from the pool for odor evaluations. In the event that the test panel consists of five members, each member shall evaluate each sample twice for odor.

3.1.6 Odor panel members should receive a nose and throat examination by a medical staff prior to, and after, each odor test session.

3.1.7 Members of the pool shall not participate on the panel if their sense of smell is affected in any manner.

3.1.8 At least one of the seven basic odors shall be presented to the panel members as a standard for sensing odor prior to evaluation of odors from any sample material.

3.1.9 Panel members shall not be permitted to see the material or to know the gas sample being evaluated for odor, nor to see the ratings of the other panelists.

3.1.10 Odor evaluations on sample materials shall be performed in a suitable room, free from extraneous odors.

3.1.11 Odor evaluations shall be performed on every new bottle of oxygen or gas used for the tests.

3.3 Odor Evaluation - Each panel member shall evaluate the odor of a sample according to the following scale:

Members Rating	Test Conductor's Rating
Undetectable	0
Barely detectable	1
Easily detectable	2
Objectionable	3
Irritating	4

3.3.1 A total score of 25 or less, for the sum of ten odor evaluations of any sample material by the panel members, signifies the material passes the odor test. A total score above 25 at any dilution signifies that the material fails.

4.0 TEST DISCIPLINE

4.1 All materials shall have been tested for carbon monoxide content and total organics before being subjected to the odor test. If a material has over 25 micrograms/gram of carbon monoxide, or over 100 micrograms/gram of total organics, no odor test shall be performed. If the offgassing produces above 10μg/g are analyzed and are judged by the appropriate space medicine division to be noxious, this fact shall be noted on the test report, and no odor test need be performed.

4.2 Each test shall be directed by the cognizant test engineer or his appointed alternate.

4.3 Approval of the test shall be indicated at the end of the test procedure by the signature of the responsible Test Engineer. Entries transcribed to the test data sheets will also be verified by the Test Engineer.

5.0 TEST EQUIPMENT

5.1 Test Chamber - The test chamber shall be made of pyrex glass and its internal volume shall be two liters minimum. The test chamber shall have the following:

5.1.1 A gas tight removable cover.

5.1.2 A sampling valve.

5.1.3 A sampling port capable of being sealed with a septum.

A laboratory vacuum dessicator may be utilized as a test chamber.

5.2 Oven - The oven shall be capable of providing a constant temperature of 200°F.

5.3 Test Gas Supply - The test gas shall be commercially available & conform to the appropriate specifications. Suitable equipment for transferring gas to test chamber shall be used.

5.4 Sample Transfer Equipment - Glass syringes, of 30 cc minimum capacity, shall be used for measuring and transferring the sample atmospheres from the test chambers to panel members face mask.

5.5 Pressure Gauge - The pressure gauge shall be capable of measuring absolute pressures to within ± 0.1 Torr accuracy.

5.6 Olfactometer - The olfactometer shall consist of a mask made of odorless flexible material which can be applied to a panel member's face.

5.7 All odor testing equipment shall be nonproducers of odor and carbon monoxide, under test conditions set forth under Section 8.0.

6.0 PRETEST PROCEDURE

6.1 Verify material identification as one of the items below:

6.1.1 Manufacturer's certification

6.1.2 NASA certification

6.1.3 Contractor certification

6.1.4 Definite identification not available

7.0 TEST SPECIMENS PREPARATION

7.1 All the materials tested shall be classified into four catagories; surface, volume, weight, or specialized items.

7.2 Samples based on Surface

This classification is defined as all those materials that are essentially two-dimensional. This would include films, fabrics, coatings, finishes, inks, primers, adhesives, thin film lubricants, tapes, and electrical insulating material.

The sample tested shall have a surface area of 46.5 ± 2.5 square inches per liter of test container. Coatings, finishings, etc. shall be coated on clean aluminum substrate of 0.020 ± 0.01 inch thickness. Material thickness, curing process, and method of application shall be in accordance with the manufacturer's recommendations. Material may be coated on both sides of the aluminum panel. Tapes and other similar materials with an adhesive surface shall be fastened to a similar aluminum panel. In all cases only the outer surface of a material on the aluminum panel is counted in the surface area determinations. Films, fabrics, and similar materials shall be cut to give 46.5 ± 2.5 square inches surface area. Since these materials are two-surfaced in use, both the top and bottom surface shall be counted in determining total surface area. Heat shrinkable tubing shall be applied and shrunk to simulate actual use configuration.

7.3 Samples based on Volume

This classification is defined as all those materials having a definite volume but having a large real surface area due to surface convolutions or matting. These shall include foams and other blown or foamed materials and insulation padding.

Samples of these materials shall be cut to a thickness of 0.50 ± 0.05 inches unless the existing thickness is less than 0.40 inches. In this case, the existing thickness shall be used. The material shall be cut to such a size as to give 7.75 ± 0.75 square inches of total surface per liter of test container volume. All surface, tops, bottoms, and sides shall be used to compute total surface area. In cases where the natural thickness is such that the material cut would be too large to be placed into the container, two or more pieces may be cut as long as the total surface area requirement is met.

7.4 Samples based on Weight

This classification is defined as well as all those materials having a definite bulk and not falling into the volume classification. This shall include potting compounds, molding compounds, cast or formed objects, solid wires, and thick plastics. Liquids that are not used or applied as coatings or thin films shall be included.

The samples shall be used as much as possible in the supplied configuration and cut to give 5.0 ± 0.25 grams per liter of test container. Potted or molded materials shall be prepared and cured per manufacturer's directions and cut to weight. Liquids shall be placed in an aluminum dish 2.25 ± 0.25 inches in diameter. Sample weight shall be 5.0 ± 0.25 grams per liter.

7.5 Specialized Items

It must be recognized that some materials will not meet the above classifications and must be specially handled. This will most often occur with non-homogeneous materials. These cases will be tested in the manner designated by the test engineer in charge. The manner of testing and sample preparation shall be fully reported.

8.0 TEST CONDITIONS

8.1 The atmosphere and pressure in the test chamber at the start of exposure shall be that specified for each program.

8.2 The sample materials shall be heated at test temperature of 155° ± 5°F for a test duration of at least 72 hours.

8.3 Leak Check - The test system shall not increase in pressure more than one Torr. while remaining at a reduced pressure of one torr for a time period of one hour.

8.4 Odor evaluations shall be started within three hours of the conclusion of the thermal treatment.

8.5 All measuring equipment shall have the proper calibration stickers.

8.6 All equipment shall be cleaned in accordance with commonly accepted laboratory practices and shall be given a distilled water rinse and oven-dried at a minimum temperature of 200° F. The equipment shall be free of extraneous odors.

9.0 TEST PROCEDURE

The procedure shall be conducted in the following order.

9.1 Sample materials shall be prepared according to the conditions outlined under sample preparation in Section 7.0.

9.2 After placing the sample material in the test chamber, the chamber shall be evacuated to one Torr. or less. The test chamber shall then be pressurized to test pressure and test atmosphere.

9.3 The test chamber shall be exposed to a temperature of 155 ± 5°F in an oven for a time period of at least 72 hours, allowing time for initial warm-up.

9.4 Following the isothermal exposure, the test chamber shall be removed from the oven and allowed to return to room temperature.

9.5 The pressure in the test chamber shall be measured and recorded.

9.6 Observation of distillable residues on interior chamber walls shall be made and recorded.

9.7 The test chamber shall be pressurized to one atmosphere with test gas and a sampling septum installed.

9.8 Odor Test - Known volumes of sample atmosphere shall be extracted from the test chamber by means of a syringe and diluted with fresh gas in the following proportions:

9.8.1 One part of sample atmosphere to 29 parts of test gas.

9.8.2 One part of sample atmosphere to 9 parts of test gas.

9.8.3 No dilution, or as drawn from the flask.

9.8.4 A material that fails the criterion at any of the above dilutions shall terminate additional testing. Testing shall begin with the greatest dilution and progress toward no dilution.

10.0 REPORTING

Unless otherwise specified, the following test data and pertinent information concerning the materials shall be reported.

10.1 Name and positive identification (para. 6.0) of material.

10.2 Vendor designation and vendor.

10.3 Total rating numbers determined by the panel members.

10.4 Definition of the odor as interpreted by each panel member.

10.5 Status of the material as a result of the test.

10.6 Name and number of the test procedure.

10.7 Test pressure, atmosphere, and temperature.

10.8 Date of test.

10.9 Identity of the testing agency.

10.10 Name and signature of test coordinator.

APPENDIX

- E -

BOND STRENGTH
TO ETCHED
TEFLON AND KAPTON WIRE (1)

---

(1) Appendix B of NAS 9-8750 Statement of Work (Ref: Appendix A of this report).

TEST NO. 2

BOND STRENGTH TO ETCHED TEFLON AND KAPTON WIRE

1.0 PURPOSE

This test is designed to evaluate the bondability of the candidate potting, encapsulating, and conformal coating compounds to etched TFE Teflon wire insulation and FEP Teflon dispersion-coated Kapton wire insulation.

2.0 CRITERIA OF ACCEPTABILITY

Compounds shall fail in cohesive shear at no less than ten (10) pounds load, when the wiring they are encapsulating is subjected to a standardized tensile test. Adhesive failures allowing the wire to pull out of the compound are indicative of inadequate etching, and require retesting.

3.0 TEST EQUIPMENT

3.1 Tensile Tester, Instron, or equal.

3.2 Sample Holder, per Figure 1.

4.0 SAMPLE PREPARATION

4.1 The following wire insulations shall be used in testing each compound:

4.1.1 Three (3) samples of TFE Teflon insulated AWG 20 hookup wire, per MIL-W-22759, Type MS 21985. Each sample shall consist of five (5) wires, 24" long.

4.1.2 Three (3) samples of FEP Teflon overcoated Kapton insulated AWG 20 hookup wire, per MIL-W-81381/1 (AS). Each sample shall consist of five (5) wires, 24" long.

4.2 The wiring specimens of 4.1 shall be etched per MSC-SPEC-Q-3 (Appendix F).

4.3 The etched specimens shall be encapsulated in the sample holder of Figure 1, using the candidate compounds. The samples shall be cured, using recommended cure time/temperature cycle.

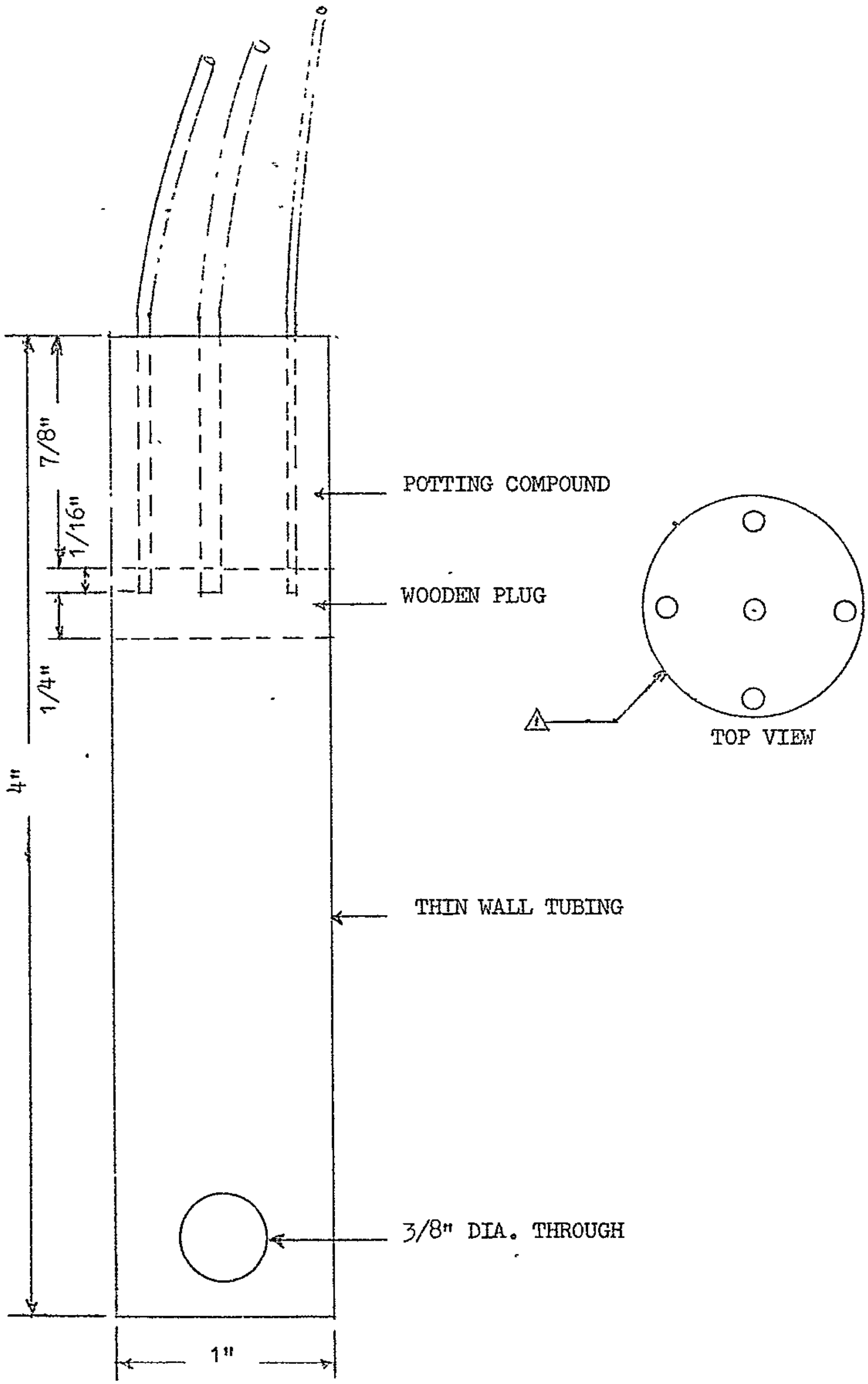
5.0 TEST PROCEDURE

5.1 Insert a pin through the hole in the bottom of the sample holder and attach to a clevis at the lower crosshead of the tensile tester.

5.2 Attach the wires under test to a lug provided at the upper crosshead.

FIGURE 1

WIRE BOND STRENGTH SAMPLE HOLDER



⚠ HOLE DIAMETERS IN WOODEN PLUG TO CLEAR WIRE

5.3 Assure that clevises/lugs are attached to the tensile tester crossheads with spherically seated bolts to assure alignment of the force along the wire axis.

5.4 Set the moving crosshead speed at one inch per minute free head travel.

5.5 Conduct the test, assuring that the test wires are pulled completely free of the candidate compound.

6.0 RESULTS

6.1 Report the highest load carried by the wire under test as the ultimate load, for the three samples of each of the wire insulations of paragraph 4.1.

6.2 Report whether each specimen failed cohesively or adhesively.

APPENDIX

- F -

PRE-TREATMENT
(ETCHING)
OF TEFLON SURFACES
PRIOR TO POTTING PROCESS,
SPECIFICATION FOR

MSC-SPEC. Q-3

(1)

---

(1) Appendix E of NAS 9-8750 Statement of Work
(Ref: Appendix A of this report)

MSC-SPEC-Q-3
September 1966

MANNED SPACECRAFT CENTER

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

HOUSTON, TEXAS

PRE-TREATMENT (ETCHING) OF TEFLON SURFACES PRIOR TO POTTING PROCESS

SPECIFICATION FOR

This specification has been approved by the Manned Spacecraft Center (MSC) for use by MSC contractors, MSC, and other Centers as appropriate.

1.0 SCOPE

This procedure describes the materials and techniques required to adequately etch Teflon TFE and FEP surfaces to provide a bondable surface prior to potting.

2.0 MATERIALS

2.1 Etchant

"Tetra-Etch", manufactured by W. L. Gore and Associates, Neward, Delaware or approved equivalent.

2.2 Cleaning Compound

"Freon" Precision Cleaning Agent (PCA), manufactured by E. I. DuPont "Freon" Products Division, Wilmington, Delaware.

2.3 Neutralizer

Naptha solvent, Laboratory Quality, Fisher Scientific Company, or equivalent.

2.4 Gaseous Blanket

Nitrogen, Specification B-B-N-411b, Amendment 1 of January 19, 1966, Type I, Class I, Grade B.

2.5 Dryer

Silica Gel, indicating commercial grade.

2.6 Rags

Grade A, commercial grade.

2.7 Bags

Kraft paper, commercial grade.

2.8 Shrink Tubing

Thermofit SCL, per Rayclad Bulletin AT-625.

3.0 PROCEDURE

3.1 Trimming

Trim all wires (except those described in paragraphs 3.3.5 and 3.3.6) to the dimensions required by the applicable drawing or specification, retaining 3" - 6" in excess of these dimensions. If shielded wires are to be terminated within the potting so that only the shield jacket protrudes, the primary wire insulation does not require etching and terminations should be made after etching only the shield jacket. If shielded wires are to be terminated outside of the potting, the terminations should be made prior to etching. In the latter case, shield jackets do not require etching, but all primary wires, shield termination wires, and any other jumper wires which will exit the potting should be trimmed at this time, leaving 3" - 6" excess length.

3.2 Cleaning

All dirt, grease, finger prints, and any exterior coatings previously applied to wire to be etched shall be removed by wiping vigorously with "Freon" Precision Cleaning Agent (PCA). This shall be followed by a clean oven bake at 120 degrees F for 30 minutes.

3.3 Etching

3.3.1 Obtain fresh "Tetra-Etch" from refrigerated storage. Assure that maximum shelf life of six months when stored at 40 degrees F to 50 degrees F has not been exceeded. Fresh "Tetra-Etch" is dark greenish-black. It should be discarded after 8 hours at room temperature or if it begins to lighten.

3.3.2 Pour "Tetra-Etch" into a 500cc glass beaker to a depth of approximately 3". Return unused "Tetra-Etch" to refrigerated storage immediately.

3.3.3 If the etching process is to be continuous, provide a sufficient flow of dry nitrogen gas passed through a silica gel dryer to form a gaseous blanket over the container. If the process is used only intermittently, the nitrogen is not required, but the beaker should be kept covered when not in use.

3.3.4 Make a "U"-bend in the wires to be etched and immerse in "Tetra-Etch" for approximately 30-45 seconds, keeping cut wire ends and/or shield terminations above the surface of etchant. If the bundle to be etched does not include any white Teflon insulated wires, a sample of white wire should be included. Once the wires have been immersed, do not remove until the 30 seconds have expired. (NOTE: If etchant is exposed to air, a coating forms which prevents further etching if the wire is re-inserted into the solution.) After 30 seconds, remove the sample of white wire and examine for typical continuous light tan to dark brown color. If satisfactory, remove all wires and drain. If the sample wire has any original white color showing in the etched area, a satisfactory etch has not been obtained and wires shall be reprocessed starting with paragraph 3.2.

3.3.5 In the event that insufficient wire length exists to make a satisfactory "U"-bend (for example, if the wires to be etched have already been cut to length by a component supplier), the open ends of the wires and/or shields shall be sealed from etchant penetration with Thermofit SCL tubing of the appropriate diameter. A $\frac{1}{8}$" piece of SCL shall be placed over the last ¼" of wire and shrunk down. The ¼" of tubing extending beyond the end of the wire shall be pinched together while molten to effect the seal. An alternate means of protecting open ends is to dip them approximately ½" deep in molten candle wax.

After sealing open ends, the wires shall be immersed in "Tetra-Etch" per paragraph 3.3.4.

3.3.6 It is highly desirable that etching be accomplished prior to termination of wires, however in the event that etching of wires is required after the wires have already been terminated (such as after electrical connectors or circuit boards have been installed by a lower tier supplier), the etchant shall be applied to all accessible wire insulation surfaces with a thin polyethylene strip, after orienting critical parts (such as connectors, circuit boards, etc.) so that they are not exposed to etchant. A brush shall not be used because air entrained around bristles may reduce the degree of etch obtained.

To complete the etching process, follow the procedure of paragraph 3.3.4.

3.3.7 Dip satisfactorily etched wires in a 500cc beaker containing approximately 3" of clean Naptha solvent and agitate gently. Remove and drain.

3.3.8 Repeat paragraph 3.3.5, using a second beaker of clean Naptha solvent.

3.3.9 Oven bake etched wires in a clean oven at 120 degrees F for 30 minutes. Minimize handling of insulation in etched areas.

3.3.10 Cut wires to length per applicable drawing or specification. Remove only ½" from ends of wires prepared per paragraph 3.3.5.

3.3.11 Place freshly etched wires in clean paper bags and seal to protect from dirt and light.

3.3.12 Etched wires shall be potted as soon as possible after etching. If more than three weeks have elapsed, the wires shall be reprocessed per paragraphs 3.2 and 3.3.

3.3.13 Etched wires shall be protected from light and excessive handling prior to potting.

4.0 QUALITY ASSURANCE PROVISIONS

4.1 Inspection & Acceptance

Acceptance of the etching process shall be based upon the requirements outlined on paragraph 3.1 through 3.3.13 above.

4.2 Wire bundles containing white Teflon insulation - the white wires shall have a continuous light tan to dark brown coloring. Mottled or spotty appearance is cause for rejection and reprocessing per paragraphs 3.2 and 3.3.

4.3 Wire bundles containing only colored Teflon insulation and/ "H"-Film insulation with Teflon dispersion overcoat - the sample white Teflon wire shall be tagged to identify it with the etched bundle. This sample shall be inspected per paragraph 4.2. If the sample exhibits mottled or spotty appearance, the bundle shall be rejected and reprocessed per paragraphs 3.2 and 3.3.

5.0 ETCHANT CHARACTERISTICS

5.1 Shelf Life of Etchant

5.1.1 Shelf life of "Tetra-Etch" is six months maximum when stored between 40 degrees F and 50 degrees F.

5.1.2 Small containers should be used in preference to large containers in minimize reuse of previously opened containers. These containers shall be kept closed and wax sealed under refrigeration except when actually removing or replacing etchant.

5.1.3 Used etahent shall be discarded in lieu of returning to original containers (see paragraph 5.2).

5.2 Discarding Spent Etchant

5.2.1 Spent "Tetra-Etch" shall be poured slowly into a large container of water until well diluted.

5.2.2 Pour diluted mixture down the drain and flush with clean water.

5.3 Etchant Composition

"Tetra-Etch" is manufactured as a super-saturated mixture of a sodium aryl compound in highly polar solvents. As no free metallic sodium is present, "Tetra-Etch" is non-phrophoric; will not ignite when exposed to air or poured into water. The solution is dark greenish-black, with a specific gravity of approximately .97 and a vapor pressure of approximately 50 mm Hg at 20 degrees C. Its flash point is approximately 33 degrees F; the auto-ignition temperature is greater than 745 degrees C. .

5.4 Safety Precautions

"Tetra-Etch" is flammable and should be kept away from open flames. Caustic soda is formed when the sodium compound reacts with moisture; therefore, any solution that gets on the skin should be flushed off immediately with water. Rubber gloves are advisable and safety glasses should be worn. Provide good ventilation to prevent prolonged breathing of fumes from the solution.

NOTICE: When Government drawings, specifications, or other data are used for any purpose other than in connection with a definitely related Government procurement operation, the United States Government thereby incurs no responsibility nor any obligation whatsoever; and the fact that the Government may have formulated, furnished, or in any way supplied the said drawings, specifications, or other data is not to be regarded by implication or otherwise as in any manner licensing the holder or any person or corporation.

Custodian:	Prepared by:
NASA - Manned Spacecraft Center	Quality Assurance Branch, AR5 Flight Safety Office

APPENDIX

-G-

MATERIALS
AND
TESTING
SURVEY

1. NASA, WHITE SANDS TEST FACILITY, 11-6-68

The laboratories conducting ignition, flammability, burn rate, outgassing, carbon monoxide/total organic, and odor tests were toured; they have excellent equipment, including a new mass spectrometer and new TGA/DTA. Experience is low on the new equipment. Subject program was reviewed with Dave Pippen (Mgr., Systems & Materials Evaluation Laboratory) and Len Schluter (Mgr., Materials and Processes Laboratory). Both are NASA personnel but have LTV/AGC staffs who actually perform the tasks. Dick Lindquist, Chief Chemist of the AGC staff supporting Len Schluter was particularly involved in the conversations. Some of Schluter's people believe the contract's work statement outgassing test condition of 1 x 10^{-4} torr as being considerably difficult to achieve (and hold) with simple pump. They also consider 10μ gm/gm as their chamber "clean" limit, "a lower limit would present serious practical problems in measurement." They have approximately 10 stainless steel cylindrical chambers, 120 liters each, for the different forms of pyro testing, but they utilize the same basic design in each one. The variations in testing are essentially achieved via vertical or horizontal positioning of the chamber, and appropriate specimen fixtures. They insist on direct chamber venting to building exterior, with vacuum assist being available (and usually utilized). Each chamber has both a rupture disc and a relief valve. They have not observed significant pressure increases upon ignition. They have heard of MSC's use of silicone igniters to obtain increased initial flammability. They are generally using the tissue wrapped hot wire technique. They would, theoretically, prefer a massless igniter with a maximum area. In general, they put the specimen in the chamber, evacuate to 0.1 torr, backfill with the test gas to test pressure, "soak" the specimen for ten minutes, and ignite. A total specimen cycle can take fifteen to ninety minutes, depending upon specimen condition and post-test clean-up time.

Mr. Pippen offered to do some of the testing for this program if it didn't interfere too greatly with their normal work schedule. For example, he could slip in a few samples at a time without static from anyone. However, they couldn't guarantee how long they could maintain such an arrangement if they got a sudden influx of "normal" work. They presently can totally handle 10-15 materials per week on a maximum effort basis (but no overtime). Turn-around time for 2-3 samples for CO/TO/odor would be approximately one week if received on Thursday; two weeks if any other day. Their usual procedure is to prepare the specimen for test by Friday, place it in the environmental chamber that afternoon, evaluate CO/TO on Monday, odor (if O.K. to run) on Tuesday, do a quick-look at the data on Wednesday. Thus, Furane could receive a preliminary report via telephone Wednesday afternoon.

They can also conduct the potting and conformal coating flammability tests of MSC-D-NA-0002, Test Methods Nos. 5 and 17, respectively. Film coverage would also be provided, with a turn-around time of 1-2 weeks if processed at WSTF (option: 1 day delivery of raw film to MSC for processing; MSC turn-around time not stipulated as yet). In addition, the pyrolysis outgassed products would be examined via IR and GC for any peaks. These peaks would then be identified.

Mr. Pippen wasn't sure if MSC would approve specimens going directly from Furane to White Sands (usually specimens are funneled through Houston). Mr. Lieberman stated that he would consult with MSC to avoid the unnecessary transshipment. Before firming up the whole process, its obvious advantages must be carefully weighed against some potential inherent problems: The cost of establishing the test facilities in dollars and learning curve time (equipment as well as personnel) must be weighed against increased travel cost/time and the possibility that their "normal" work load could increase to a point where they would not be able to provide the above turn-around time, or even test Furane specimens at all.

The Furane concept for the flammability chamber was reviewed. They didn't see any major objections if complete safety precautions were utilized. Furane/MSC will have to go through L. Maurice Clelland, Chief, Program Control Office, for equipment prints/photos, and test and specimen preparation procedures.

2. NASA-MSC (HOUSTON, TEXAS) 11-7-68, 11-8-68.

Mr. Lieberman first met with Harry Kline (Contract Technical Monitor for NAS 9-8750), Structures and Mechanics Division. Formulation possibilities with commercial elastomers were reviewed, including Mr. Kline's prior and present laboratory activities in this area. Flammability testing of his latest formulations in 16.5 psia oxygen was observed. The potential testing arrangement with White Sands (e.g. CO/TO, odor, etc.) was reviewed. Mr. Kline concurred in the direct shipment of specimens from Furane to WSTF. He suggested consideration of "NASA-MSC Non-Metallic Materials Test Request/Report, MSC Form 2035B (Rev. Apr. 68)." The format will be established upon consultation with Dave Pippen of WSTF. Mr. Kline commented that WSTF has done considerable flammability studies in rate of burning, but they do not have significant experience with flammability of connectors. On the other hand, his lab is constantly testing these latter types as well as unenclosed connector-size specimens. However, WSTF is probably better equipped and has had greater experience in the analysis of offgassed products, a requirement of the flammability screening test. In responce to the suggestion that MSC run some of the flammability tests, Mr. Lieberman commented that the pre-preliminary and preliminary test phases will require the investigation of numerous specimens in a very short time (schedule limitations) and that formulation modifications may be required as soon as a flammability test is completed. This would necessitate conducting these tests at Furane. However, the final flammability screening test, as specified in the Work Statement, could be carried out at either MSC or WSTF.

Mr. Kline's technicians (contractor: Brown & Root/Northrop) "pull down" the chamber to 5 torr, leak check for five minutes, backfill, and test. His chamber is a rectangular cast aluminum shell, 1' x 1' x 2' with 3/4" x 3/4" reinforcing angles. The door has a glass face and is sealed via an expanding 'O' ring-type gasket. This seals for vacuum or overpressure. He has a relief valve but no rupture disc. (The door bolts are sufficiently weak so that an overpressure pops them.) The Furane approach for a chamber was reviewed; no significant objections were raised.

His latest formulation efforts include a mixture of Viton LM, boric acid (or sodium metaborate), zinc oxide, EPON H-1, and DTA (or DETA). He is having considerable trouble trying to get a cure with this system at temperatures under 150° F. (thermal limit for certain types of transistors). He has been casting his ignition specimens in a 50 ml. beaker. The cured (?) specimens are removed from the beaker and helically wrapped with several turns of wire which is then fastened to the chamber holding fixture. A fusible wire is placed directly under the specimen and attached to the power source. Usually, the test is conducted with tissue paper wrapped around the wire to assure flame initiation. Another ignition method is to use a silicone rubber igniter, containing 15 phr GE-577 and 85 phr GE-560. The RTV rubber is cast into 1/4" diameter rods and cut into 1.0" lengths. Six helical wraps of 20 gauge nichrome wire surrounds the igniter. Approx. 55 amps is used to fuse the wire in both methods. It has been arbitrarily established that the igniter must burn for 25-30 seconds in order to have acceptable ignition. The observed test with the silicone igniter evidenced considerable smoke and some flaming, dripping material. A subsequent examination of the specimen revealed that the silicone had puffed sufficiently to contact the specimen and adhere to it quite firmly. It is clearly a more drastic igniter than the tissue paper.

Mr. Kline also tests the compounds with a connector per Test No. 5 of D-NA 0002 (MSC). However, due to the cost of the non-recoverable connectors he usually leaves such testing until after some evaluations have been conducted with the simple 50 ml. beaker specimens.

He will forward copies of his previous formulations, their test records, and loan copies of photos of the tested specimens to Furane.

Following this, a meeting was held with Dr. Elliot Harris, M.D., who heads the MSC group concerned with toxicity of spacecraft materials.

Dr. Harris readily acknowledged that no "cook-book" existed which specifies (for more than the conventional industrial chemicals) toxic species, their critical limit of toxicity (expressed in ppm) in pure oxygen (or even air), additive/synergistic effects of mixed species, and applicable test methods. However, he suggested the following general guidelines for our material selection process: Avoid

1. Acetic acid forming RTV's.
2. Phenyl silanes. They appear to lead to steroid interactions when overheated.
3. Non-polymerized constituents.
4. Excessive use of solvents and volatile diluents. ("Vacuum treatment not always effective.")
5. Benzene or phenyl derivatives.
6. Carbon monoxide producing systems; although of relatively low toxicity, it must not be present since the Life Support Equipment is not capable of adequately removing CO.

Personnel of the Structures and Mechanics Div. maintain that when ignition occurs as a result of an electrical malfunction, all organic materials will be effected. Dr. Harris feels that this isn't the real issue. He states that primary consideration should be given to 1) the temperature at which serious decomposition takes place; 2) the rate of decomposition at a given temperature; and 3) the nature of the byproducts. Mr. Lieberman commented that both were actually correct; thermal decomposition and pyrolysis were both of significance, and hence, of concern.

Dr. Harris is presently establishing an in-house chemical and animal testing capability. He requested Furane to submit the candidate materials to him for his evaluation whenever we established a firm interest in a material. Messrs. Kline and Lieberman later agreed that Furane should submit specimens, if possible within the prescribed budget and schedule limitations, upon completion of the screening phase.

Dr. Harris stipulated that CNR should not be used above room temperature. He further stated that 15 gms of CNR decomposing in the spacecraft would be lethal (Dr. Thomas' results?). He also stated that a Mr. Jerry Smith at White Sands had found hydrolyzables produced by CNR at 150° F. and TGA/mass spectrometer analyses of 150° F. decomposition products indicated presence of such highly toxic species as carbonyl fluoride. An attempt will be made to procure the reports covering this work.

Dr. Harris was not sure how to most effectively test animals with condensables. He was considering use of oral and/or sub-cutaneous injections, but was not fully satisfied with either approach. He hopes to shortly arrive at a more realistic method.

Messrs. Kline and Lieberman met with Charlie Nagle, Tech. Services (shop), and some of Mr. Nagle's crew. They are still potting with such flammable compounds as GE 560 and 630, 3M EC 1090, and E. C. Stycast 2061. However, very little of their work is presently going inside the manned areas. Where it does, MSC personnel apply a Fluorel-fiberglass "bag" around the potted component. The Fluorel coated system is then exposed to 160° F. for twelve hours to remove the MEK carrier and partially cure the Fluorel. Mr. Nagle believes that North American Rockwell testing of this system shows significant outgassing. He also noted that present procedure calls for a materials approval prior to each flight. In general, this group either follows various Huntsville processing specs, manufacturer's data sheets, or their own experience.

Mr. Lieberman then conferred with Tom Mancuso, buyer for NAS 9-8750, primarily to seek his aid in procuring test procedure and facilities information from White Sands. He immediately contacted them and conveyed our request. Mr. Mancuso will pursue this by letter to WSTF.

3. RICE UNIVERSITY (HOUSTON) 11-8-68.

Based upon a prior contact by Dr. Margrave (Rice Chem. Dept.) with Mr. Kline, a meeting was held by Mr. Kline and Mr. Lieberman with Dick Lagow, a graduate student of Dr. Margrave, and Joe Nalle, Business Manager and Legal Counsel for Rice University (Dr. Margrave was out of town on this date). The Rice personnel claim that Margrave and Lagow have developed a unique process (patent application in) to directly fluorinate any hydrocarbon with F_2 gas and achieve essentially 100% yield. The visitors were permitted to see the actual apparatus and various samples as they appear before and after processing. For example, they were shown two small vials of white powder; one which was said to be a commercially available polyethylene powder and the other which Lagow claims was their processed perfluorinated version of same, it having similar properties to TFE. Their present rate of production is only 3-4 gm/day for solids. Lagow believes that they would obtain only about 0.5 gm/day for liquids with their present lab equipment. Lagow noted that if the precursor material has double bonds, fluorine saturation would very probably occur. Thus, if unsaturation was desired, a reduction step would be necessary. Mr. Lagow was not certain of the success with such a reduction. No further action would be taken until Dr. Margrave was appraised of the situation.

A subsequent telecon with Dr. Margrave elicited the information that some working arrangement was in process between Rice and Ozark Mahoning, Tulsa, Oklahoma. Dr. Margrave said that Dr. Wayne White of Ozark Mahoning should be contacted directly since quantity requirements for this program far exceed present Rice capabilities.

4. PENNINSULAR CHEM RESEARCH, INC. (GAINSVILLE, FLA.) 11-11-68.

Mr. Lieberman was unable to get into Gainsville due to weather. Dr. E. C. Stump was contacted to establish a future discussion via telecon and mail.

5. NASA MARSHALL SPACE FLIGHT CENTER (HUNTSVILLE, ALA.) 11-12-68

The first meeting was held with Don Knott of the Astrionics Lab. He is electrical hardware oriented, has been involved in their past development efforts re potting, et al. He said that PRC 1535 and 1538 are extensively used, with 1538 being the "official" Astrionics material.

The only exception to the latter being material used per Kennedy Space Center Spec. 001, "Thin Film Conformal Coating". He thought that there was a strong feeling among MSFC designers to minimize thickness of coatings (lower mass; hence, less loads during shock and vibration). He thought that Bill Fussel of Spacecraft, Inc., Huntsville, was working on space-worthy (?) conformal coatings, particularly for PC cards requiring "ruggedization."

The second meeting was with Dr. James Stuckey, Chf., Composite Materials and Coatings Section, Non-Metallic Materials Br., Materials Div., Propulsion and Vehicle Eng. Lab.; W. Jerry Patterson, Rubber and Plastics Technology Section, Non-Metallic Materials Br., and Dr. Wm. Hill, Polymer Chemistry Section, Non-Metallic Materials Br.

The extensive fluorine chemical and monomer activities which they had previously contracted to Penninsular and Narmco were reviewed. Dr. Hill noted that most of the Narmco work dealt with aromatic compounds, none of which were saturated. Reports on these programs are available; no further work on them is presently in progress. "Lack of budget" was given as the reason for cancellation of further work on the following programs:

"Develop Improved Potting, Encapsulating, and Conformal Coatings" - Monsanto (NAS-8-20402). Flammability was a consideration, but was not emphasized.

"Liquid Prepolymer for Better Low Temp. Properties" - Thiokol (NAS-8-21197). A one year report is due any day.

These people have also done extensive work with substituted silane-diol polymers for elevated temp. stability. To date neither elastomeric or even flexible polymers have been prepared. Mr. Patterson suggested the triazines. "Research Study on Development of Conformal Coating Material and Potting Compound for High Freq. Circuitry" (NAS-8-20375) was one of their contracts. The final report is available.

The third meeting was with Dr. Nunnelley, Chf., Chemistry Br., Materials Div., concerning MFSC flammability testing in 100% oxygen. They have written their own document, MSFC-Spec-101 (Aug. 20, 1968), " Flammability Requirements and Test Procedures for Materials In Gaseous Oxygen Environments." The testing for potting and coating compounds is almost identical to MSC D-NA-0002, Test Method 5. They expect to shortly issue a report on their flammability results. C. Frank Key, Special Asst. to Dr. Nunnelley, will be contacted for the report. They also expect to issue a report on TGA/DTA vs auto-ignition data, in air and in GOX, within the next six weeks. They are hoping to depend upon MSC for toxicity and odor data.

Following this meeting, a Mr. Smith in the Thermal Physics Section, Engineering Physics Br., Materials Div., was contacted re outgassing data. They are preparing a designer-oriented "cook book" for material selection with respect to outgassing. It is "ATM Material Control for Contamination Due to Outgassing." (ATM: Apollo Telescope Mount project.) The document will be periodically updated to insure maximum usefulness of the data. Mr. Smith was also queried about the degree of difficulty in obtaining the 10^{-4} torr vacuum specified in the Work Statement. He felt that it would be quite a problem to maintain such a vacuum over a specimen without going to an oil diffusion pump.

6. NASA GODDARD SPACE FLIGHT CENTER, Greenbelt, Md. - 11/13/68

The first meeting was with Dr. H. E. Frankel, Hd., Materials Research and Development Br., Systems Div., Technology Directorate; A. Fisher, Hd., Polymer Chemistry Section, Materials R & D Br.; and Ed Nelson, Polymer Chemistry Section. An enormous amount of outgassing data has been obtained over the years by the Polymer Chemistry Section in order to consider use of materials in deep space for as long as five years. However, the data is clearly not available in useable form for "outsiders." There are no up-to-date published documents covering this area. Several old ones suggested include:

> "Polymers for Spacecraft Applications" - R. F. Muraca and J. S. Whittick, Stanford Res. Inst., 1967, JPL Contract No. 950745 under NAS 7-100.
>
> "Spacecraft Materials Guidebook" - R. A. Happe, Materials Section, JPL.
>
> "Grumman is also doing outgassing work but they get a magnitude higher values. Dow-Corning is also active in outgassing studies, and their data quality is superior to most."

The Materials R & D Br. does not do toxicity, flammability, or polymer development studies. Their outgassing work can be considered as accelerated testing of 200-400 mg. of sample maintained at 10^{-6} to 10^{-7} torr for 24 hrs. at 125°C. Qualitative and quantitative data is obtained from effluent gases and condensables collected on a plate approx. 1 to 2 cm. away from the heat source. Mr. Nelson claimed that the plate is at 25°C. They almost invariably find hydrocarbons present. The dimethyl siloxanes, phthalate esters, and Freons are also very often present. It was suggested that Dr. I. N. Einhorn at Wayne State Univ. be contacted about his NASA and AF sponsored work to evaluate flammability and thermal characteristics of polymers. Dr. Frankel promises to get the data out in a useful form very shortly.

Next meeting was with Dr. John B. Schutt, Hd., Coating Sect., Thermophysics Br., Spacecraft Technology Div., Technology Directorate, and such members of his staff as F. G. Cunningham, John W. Stuart, and Walter A. Wappaus. This group has built up an excellent reputation in the area of thermal control coatings, primarily based upon inorganic compounds. I am not certain how they got into the potting picture, but, nevertheless, they have been semi-active in developing ceramic based potting compounds. They readily agree that the salt and alkali metal silicates are not acceptable due to their deleterious effect upon electrical properties, especially with typical levels of humidity. They are also avoiding such potentially toxic materials as oxychloride cements.

Their primary suggestion at this time is to use calcium aluminate, either alone, or in conjunction with an elastomer. The elastomer could act as a shock and vibration support, and as a porosity filler. The possibility of vacuum impregnation of the elastomer into the aluminate was suggested. Their electrical testing has shown that the slightest trace amounts of such alkali metals as sodium or potassium degrades the calcium aluminate. This has caused them to prepare their own material from comparatively pure aluminum oxide and calcium oxide. They are willing to prepare some for Furane to evaluate.

7. DuPONT, CHESTNUT RUN LABORATORIES, Wilmington, Del. - 11/14/68

A meeting was held with Dr. E. P. Lieberman and Al Moran of the Elastomer Chemical Department, Elastomers Laboratory. They believe that Viton is DuPont's best recommendation in terms of standard products. Approximately four to five months ago, Dr. Lieberman discussed Viton sealants, coatings, and various mechanical goods with personnel from A. D. Little Co. (Cambridge, Mass.). He was "reasonably sure" that Little had a contract with NASA. He will check his records for more details. DTA curves for both Viton polymers and compounds will also be forthcoming. Toxicity data is sparse, but HF is clearly a byproduct of vulcanization. In fact, type and amount of acid acceptor may be a means to modify cure rate and temperature. They are interested in developing a non-flammable elastomer, but no funds have been allocated for such effort. They hope for funds early in 1969. Even if funds were made immediately available, they would not envisage lab "samples" for at least two years. A reactive terminal end group program is also not in operation at this time. They have requested approval to investigate this area; approval not received as yet. Time scale from day zero; approx. one year for samples, approximately 1-1/2 years for a commercial product. (Note: see section of this report re 3M). They have taken only a cursory look at feasibility to develop a fluid type Viton. "This would be quite difficult, requiring redesign of polymer and curative systems. Market still too low for DuPont (20,000 lbs./yr. is recent best estimate). Dan Supkis of NASA-MSC has tried, (but to date unsuccessfully) to develop low temperature curatives for Viton flame resistant sealants." They believe that Bill Anspach, AFML-WPAFB, has also worked on Viton sealants. DuPont has not, to date, considered any Viton curatives other than amines in their flammability studies. They will provide a list of other possibilities. They claim to have found that Vitons A, B, and D-80 have similar flame rates in 16.5 psia oxygen. They suggest using Viton LM as a plasticizer for other Vitons (2,000 cps at 100°C.). Due to its low reactivity, LM is not generally recommended for use by itself. However, Dr. Lieberman did cite the following recipe which uses LM alone:

Ingredients	Parts by Weight
Part A (Base)	
Viton LM	100
Hydral 710 (1)	40
Maglite Y (2)	10
FS 1265 (1000 centistokes) (3)	10
Part B (Curing Agent): Stir into Part A just prior to use	
Tetraethylene pentamine	1
Epon Curing Agent H-1 (4)	5

Flammability: 100% oxygen at 6.2 psia (in./sec.)

Uncured	0.25
Cured 6 hrs. at 250°F.	0.15
Cured 24 hrs. at 400°F.	No ignition

(1) Hydrated Alumina (Alcoa)
(2) Magnesia (Merck & Co.)
(3) Fluorosilicone oil (Dow Corning Corp.)
(4) Ketimine (Shell Chemical Co.)

A hotter reaction can be obtained with the above recipe by increasing the amine and decreasing the H-1. For example, if three parts by weight of the amine is used alone, the sealant has only a three minute pot life at 150°F. An even stronger base (KOH?) would conceivably speed up the reaction. When such "hot" curatives are used, it is essential that they be added last to the master batch.

Viton A-HV was not considered due to its very high viscosity. It should be noted that there is a greater plasticizing effect by LM on D-80 than on A or B. However, the Shore A is reduced by 8-10 points with only 10 parts LM. D-80 is also of interest since an effective cure can be obtained with compounds other than amines (e.g., hydroquinone) whereas amines appear best with A or B. Dr. Lieberman did not believe that there would be any major difference between Viton A/diamine and Viton D-80/hydroquinone with respect to flammability in 16 psia oxygen. He thinks R. E. Darling ran such tests. Dr. Lieberman will provide a review of their flammability work with respect to filler selection. They said that the lowest viscosity Vitons were LM, C-10, and A-35, in the order of increasing viscosity.

Several papers were cited by Dr. Lieberman with regard to Viton cure mechanism(s). He readily admitted that this subject is still one of conjecture. The papers were:

> "The Chemistry of Vulcanization of Viton A Fluorocarbon Elastomer" - J. F. Smith, Elastomers Chem. Dept., DuPont, circa 1959.
>
> "The Mechanism of Post Cure of Viton A Fluorocarbon Elastomer" - J. F. Smith, Elastomers Chem. Dept. DuPont; Rubber and Plastics Age, 1961, 42, No. 1, 59.

Fluorochemicals were reviewed with E. C. Coyner, Organic Chemicals Department, Freon Products Division. There seems to be some question about future availability of DuPont's line of fluoro alcohols and/or ethers (E-oils). This will be clarified. At present, they are offering hexafluoroacetone, hexafluoroacetone sesquihydrate, hexafluoroisopropanol, vinyl fluoride, and vinylidene fluoride (data to be sent). Mr. Lieberman inquired about hexafluoropropylene oxide and was told that it is not for sale by DuPont; "All of it is being used internally by the Plastics Dept."

Bob Ribbans of the Plastics Dept. was next contacted. He reaffirmed DuPont's position on HFPO, but suggested that someone like Peninsular Chemical Research might make it (they do). He also agreed that it was an excellent consideration for a non-flammable epoxy. He also suggested the following approaches:

1. A finely divided FEP (their T-100, obtained from L. N. P. in finely divided form) added to an E-oil to form a paste, heat to 250-300°F. A plasticized, non-hydrogen containing material is obtained.
2. T-120 (55% FEP plus water) dispersion or Primer 100 as conformal coatings.
3. Anhydrous perfluoro acetone plus diphenyl ether (fluorinated ?) to form a polymeric material. This appears to be spontaneous and fast with a base. He believes General Chemical Div. of Allied recently reported this work.

8. U. S. NAVY, NAVAL SHIP ENGINEERING CENTER, Washington, D. C. - 11/15/68

Mr. Lieberman first met with Bill Shetterly who has been active in high temperature stable polymers. These include the carboranes, carborane phthalocyanine silicones, carborane silicones (Dexsil - Olin Mathieson), carborane-xylylenes, etc. He has also been involved with the Bureau of Ships supported program at Quantum, Inc., Wallingford, Conn., which resulted in a technique that uses graft polymerization to fluorinate the surface of elastomers. Recent directives have reduced his emphasis on high temperature polymers and increased his efforts on non-flammability. Primary concern is to reduce total quantity of shipboard flammable items; cost being a significant factor. They are presently considering such polymers as the phosphorus nitrozines (they may shortly give some contracts in this area). He also supported considerable phosphonitrilic polymer work at U. S. Rubber Co. Objections to phosphonitriles: glassy type easier to make than elastomers, high molecular weight hard to get, most have poor hydrolytic stability, highly crosslinked, generally hard to handle. In addition, very little physical, mechanical, or toxicity data available. He believes that Thiokol had a Navy contract (not recently) to develop silicone carboranes. He isn't specifically pursuing non-flammable elastomers, but U. S. Naval Applied Science Lab., Brooklyn, N. Y., may be.

E. A. Bukzin, Non-Metallics, Ships Materials, Structures and Ocean Technology Br., who provides technical direction and funds for Mr. Shetterley's non-flammability programs, was then contacted. He is clearly quite concerned about toxicity and smoke. It is also obvious that their program is a long term one; no immediate usefulness for the MSC program. In fact, service may flow the other way. Mr. Lieberman also spoke to B. B. Rosenbaum, Metallics and Energy Conversion Systems, Ships Materials, concerning elastomers for high pressure oxygen systems required for fuel cells, and to John Harrison, Head, Fuel Cell Br., and his assistant, Robert Bowen. They have a definite interest in the results of this program which may be applicable to theirs.

A conversation with John Kerr, NAVELECT, elicited the fact that he doesn't use any potting for his connectors because of potential maintenance and repair problems. He is interested in conformal coatings. He would like to be informed of our results. Flammability is not yet an urgent issue.

9. <u>GRUMMAN AIRCRAFT,</u> Bethpage, N. Y. - 11/18/68

The meeting was held with Bob Holden, Supv., Materials Labs., and one of his engineers, Thomas Martins, Non-Metallics. They are only interested in 6.0 psia oxygen for LM. Therefore, all their testing is at 6.0. They are presently using EC-1663 (3M) for potting, etc., in almost all systems, with a β-fiberglass bag over the unit after it is potted. Some CNR foam is present. The present list of approved materials is available if O. K. is obtained from LM Program Office. A similar O. K. is required for any other data. No changes are being considered at this time in the list for remaining LM hardware. Mr. Holden was not aware of any Grumman materials R & D re flammability. He suggested contacting George Young of M. & P. Engineering for additional information. (Mr. Young was out of the plant on visit date). Mr. Holden strongly feels that our program should require a 16.5 psia mixed gas test environment rather than 16.5 psia oxygen if the former will be the actual one used. Suitable contact will be made for the release of data discussed, but not provided.

10. WRIGHT-PATTERSON A. F. B., OHIO - 11/19 & 20/68

First meeting was with Dr. Anthony A. Thomas, Chief, Toxic Hazzards Division, Aerospace Medical Research Laboratories, and Dr. Shelden London, Chief, Chemical Br., Toxic Hazzards Division. They are conducting work for Apollo and MOL. The work for Apollo is via Dr. E. Harris of NASA-MSC. Non-metallic cabin materials were, and are, furnished by him to Dr. Thomas for outgassing and toxicological evaluations. Monsanto Research Corporation is carrying out the exposure in 9 liter flasks at 155°F. in 5.0 psia, oxygen. They analyze the outgassed products with a mass spectrometer, IR, and GLC They are also doing TGA. (Copies of TGA curves obtained from them appear to have been run in air.) They are aware of work at White Sands along same line but not in detail. The toxicological tests are conducted mainly with rats and mice. (Other animals, including monkeys, dogs and guinea pigs, were also observed in test chambers.) Both acute (7 days) and chronic (60 days) exposures are carried out with material which is heated to 155°F. in a separate chamber from that of the animals. In order to evaluate synergistic as well as additive effects, the materials are run in either combined groups of 10-15 or 100. To date, no problem has been found in any of these toxicologicals tests (except an anomolous one a long time ago, in one of the 10-15 group samples. They have not been able to duplicate its toxicity since). It was not made clear whether CNR had been involved in any of these tests. Dr. Thomas agrees with Dr. Harris about non-use of CNR above room temperature. Dr. London did not seem so sure. Dr. Thomas feels that consideration should actually be given to 100-180 day missions. He also believes that the "leaky" cabins, heretofore actually used, intrinsicly provided the best means for decontaminating the manned volume. Hence, as cabins become more leak-proof, the outgassing problem will become worse. Dr. Thomas suggested that Dr. Zapp, Haskel Laboratories, Du Pont, be contacted for data on decomposition products from TFE, FEP, Viton, etc. Dr. Thomas would like to see Dr. Harris initiate a request for a comparative evaluation of all of these types of halogenated polymers. In general, Dr. Thomas feels that Furane should "shoot for a material which gives good stability at operating temperatures". In response to a question about boron-type flame reducing additives, he strongly objects to anything which would result in boron hydrides; their experience shows that these compounds range from minimum (pulmanary irritant) to maximum (death) toxicity.

Some of the pertinent data available from this group can be found in:

"Identification of Volatile Contaminants of Space Cabin Materials"; J. V. Pustinger, F. N. Hodgson, W. D. Ross; AMRL-TR-66-53; Aerospace Medical Research Laboratories, Wright-Patterson Air Force Base, Ohio; 1966.

"Identification of Volatile Contaminants of Space Cabin Materials"; J. V. Pustinger, F. N. Hodgson; AMRL-TR-67-58; Monsanto Research Corporation; June, 1967.

"Identification of Volatile Contaminants of Space Cabin Materials"; J. V. Pustinger, F. N. Hodgson; AMRL-TR-68-27; Montsanto Research Corporation; July, 1968.

"Man's Tolerance to Trace Contaminants"; A. A. Thomas, M. D.; AMRL-TR-67-146; Aerospace Medical Research Laboratories, Wright-Patterson Air Force Base; January, 1968.

"Proceedings of the Conference on Atmospheric Contamination in Confined Spaces, 30 March - 1 April, 1965"; AMRL-TR-65-230; Aerospace Medical Research Laboratories, Wright-Patterson Air Force Base.

"Proceedings of the Second Annual Conference on Atmospheric Contamination in Confined Spaces, 4 and 5 May, 1966"; AMRL-TR-66-120; Aerospace Medical Research Laboratories, Wright-Patterson Air Force Base.

"Proceedings of the Third Annual Conference on Atmospheric Contamination in Confined Spaces, 9 - 11 May, 1967; AMRL-TR-67-200; Aerospace Medical Research Laboratories, Wright-Patterson Air Force Base.

The Fourth Conference was held in September, 1968. The proceedings have not been published.

The next meeting was with Warren Johnson, Chief, Elastomers and Coatings Br., Non-Metallic Materials Division, AFML, and the following members of his staff: Roger Headrick, Chief, Elastomer Group, Warren Griffin, Jerry Sieron, and Tom Graham. In addition, Dr. Cris Tamborski, Polymer Group, Non-Metallic Materials Division, also participated. It was immediately stipulated by all the AFML personnel that they didn't know of any polymeric specie which would be elastomeric, non-toxic, non-flammable in 16.5 psia oxygen and be appliable at 150°F. or lower. They still felt CNR was presently the best material available for Furane's purpose, even though NASA is apparently rejecting it. Further, they take this position despite the very negative report on CNR written by Aerospace Corporation for the MOL Office.

It should be noted that AFML has had considerable experience with CNR; most of which has dealt with its use as an expulsion bladder for N_2O_4. Some of their data can be found in:

"Elastomeric and Compliant Materials for Liquid Rocket Fuel and Oxidizer Applications"; W. R. Sheehan, N. B. Levine, et al; ML-TDR-64-107, Parts I - IV; Thiokol Chemical Corporation.

"Elastomeric and Compliant Materials for Contact with Liquid Rocket Fuels and Oxidizers"; J. Green, N. B. Levine, et al; ASD-TR-61-76, Parts I - III; Thiokol Chemical Corporation.

During the past several years, the primary polymer emphasis at AFML has been with the triazines, including the fluorinated ones. They have not only carried out research internally with these compounds but they also have supported applicable programs at Hooker Chemical, 3M, and the University of Florida. In general, these compounds are cured with tetraphenyl tin at 320° - 350°F. for 20 hours. However, they admit not really understanding the cure mechanism. They haven't specifically sought low temperature cure systems. They do not have much hope of finding a 100% liquid triazine which can be cured below 150°F. Mr. Graham suggested that Dr. Ed Dorfman, Hooker, Grand Island, N. Y., be contacted for further details on triazine curatives.

Some of the AFML in-house and supported triazine programs were reported in the following:

> "Linear Poly (perfluoroalkylene) triazines"; O. R. Pierce, G. A. Grindahl, et al; AFML-TR-65-78, Parts I - IV, Dow Corning Corporation.
>
> "Perfluoro Triazine Elastomers"; W. R. Griffin; ASD-TDR-62-1114; Directorate of Materials and Processes, Wright-Patterson Air Force Base; June, 1963.
>
> "Perfluoroalkylenetriazine Elastomeric Polymers"; E. Dorfman, W. E. Emerson, et al; ML-TDR-64-249, Parts I - IV; Hooker Chemical Corporation.
>
> "Crosslinking and Filler Reinforcement of Perfluoroalkylene Triazine Elastomers"; T. L. Graham; AFML-TR-66-402, AFML, Wright-Patterson Air Force Base; August, 1967.

Both Messrs. Graham and Tamborski believe that Olin reported in the literature that they had made a perfluoro analog of either a deca borane or Dexsil(carborane-silicone). Dr. Tamborski was sure that a perfluoro alkyl group on a silicone would be thermally and hydrolyticly unstable.

Dr. Tamborski has just made some perfluorostyrene. He doesn't have any available for samples. However, he suggested that Dr. Stanley Koch, Monsanto Research, Dayton, be contacted for a quote, since they will also be making it for AFML. He said that Dr. Leo Wall of NBS, Washington, D. C. (Gaithersberg, Md) has also made some in the past.

Mr. Sieron advised contacting C. S. Joseph, Du Pont Industrial Fabric Sales, Cincinnati, Ohio, about his recent claims for a compounded Viton, non-flammable in oxygen. Mr. Graham said that he and several others in the group have, at various times, taken cursory looks at developing low viscosity/low temperature curing Vitons, (generally for fuel resistant sealant applications). Some of their research was reported in the following papers:

"New Hydrofluorocarbon High Temperature Integral Fuel Tank Filleting Sealant"; W. F. Anspach and T. L. Graham; ML-TDR-64-265; AFML, Wright-Patterson Air Force Base; Sept., 1964.

"Hydrofluorocarbon High Temperature Integral Fuel Tank Sealants"; W. F. Anspach; AFML-TR-66-399, AFML, Wright-Patterson Air Force Base; April, 1967.

Several times between 1959 and 1964, they tried to accelerate H F removal from fluoroelastomers and provide unsaturation in the elastomer by adding amines or guanidines to solutions of the elastomer. For example, they tried tetramethyl guanidine alone, and sometimes with a conventional diamine. Since the TMG is an active base, it may be possible to substitute it for some, or all, of the more commonly used MgO. They did not remember being impressed with the results. Some of this work was reported in:

"Mechanism of Cross-Linking in the Vulcanization of New Elastomeric Polymers"; K. L. Paciorek, R. G. Spain, et al; WADC-TR-59-129; Wyandotte Chemicals Corporation; July, 1959.

"Stress Relaxation and Curing Mechanism of Fluorinated Elastomers"; Dr. N. Luyenlijk; ASD-TDR-63-806; Rubber Research Institute T. N. O. (Delft, Netherlands); May, 1964.

Mr. Graham also suggested the possibility of reducing the molecular weight of conventional fluoroelastomers with a KM_nO_4 treatment. Cure mechanisms of fluoropolymers are discussed in some detail in:

"Mechanism of Cross-Linking in the Vulcanization of New Elastomeric Polymers"; K. L. Paciorek, W. G. Lajineos, and B. A. Merkl; ASD-TDR-62-30, Part I; Wyandotte Chemicals Corporation, June, 1962.

"Mechanisms of Cross-Linking Perfluorinated Polymers"; P. Davis and B. Merkl; ML-TDR-64-305, Parts I (Sept. 1966) and II (June, 1967); Wyandotte Chemicals Corporation.

Jack Ross, Chief, Fibrous Materials Br., is one of the two liaison men for AFML to MOL (Ted Rinehart is the other one). He suggested that Furane obtain a copy of the AFMOL material criteria and test method document, Number 10010, from the MOL Office at SAMSO, or from George Epstein at the Aerospace Corporation.

11. <u>3M, St. Paul, Minnesota</u> - 11/21/68

This meeting was held with Dr. R. Bringer, Chief, Fluoroelastomers Laboratory, and Technical Manager, Industrial Resins, Chemicals Division. He made it clear that he wasn't at liber'y to discuss any materials or developments not fully covered by a patent or patent disclosure. 3M has been working with NASA-MSC, particularly with the Crew Systems Division. 3M submitted everything they had which could be of interest in the "non-flammability" search. The net result of these evaluations was that Fluorel appeared to be best (even better than non-hydrogen containing Kel-F elastomer).

3M apparently has looked into processing and formulation variables which could improve Fluorel non-flammability. They screened additives but were not impressed with any conventional ones. However; Dr. Bringer claims that they have come up with something (additive, processing, or?) which results in a non-flammable (in oxygen) elastomer. They refer to it as KF-2140 "Special Grade for NASA use". (The KF-2140 is their standard, unplasticized, unfilled Fluorel.) They are presently supplying this "special" to Mosites Rubber Company (Houston) and Raybestos-Manhattan (South Carolina) in accordance with a private agreement with both companies. It appears that both companies are further modifying the "special" to suit their own applications. A NASA Program Apollo Working Paper, Number 1337, "Non-Flammable Fluorel Compounds", reviews these applications. It also appears that each 3M-2140 "special" batch goes to MSC first, for flammability evaluation, before being sent to Mosites or R/M. Dr. Bringer will check on whether Furane could be included in the arrangement if the "special" were used in its NASA Program.

Comparisons between Fluorel and Viton were discussed. The relative reactivities of Fluoroelastomer curatives were reviewed. For example, such typical ones as Diak (Du Pont) Numbers 1, 2, 3, and 4 have decreasing rates with increasing number. The diamines are faster and more reliable than the monoamines. The straight diamines are faster than their carbamates (e.g. Diak 1). If a perfluoroamine were used, it would be somewhat slower (i.e. lower basicity) than the amine. This might be overcome with more M_gO. Dr. Bringer also feels that the fluoroelastomer curing mechanism(s) is (are) not clearly defined. He seems to remember that Bill Anspach (AFML) attempted to get some reactivity (i.e. unsaturation) in the backbone of Du Pont's C-10 by putting it into a solution with an amine and curing it with a dimercaptan. He believes Warren Griffin may also have been involved.

Dr. Bringer then discussed their latest (11/8/68) public disclosure on Fluorel developments. It concerned a highly fluorinated (approximately 68% F by weight. This compares to approximately 63% F for 2140) hydroxyl-terminated binder resin, L-2344. Although there are 2 hydrogens on the hydroxyl carbon, in addition to the 2 hydrogens present in the typical repetitive section of the Fluorel backbone, the total hydrogen is calculated to be only 1.4% by weight in L-2344, as compared to 2.0% for 2140. This is because only 65 mole % vinylidene fluoride is present in L-2344, whereas 2140 has 80%. This material was developed primarily for a high impulse propellant binder system. They are working closely with Ed Mastrolia, AGC, Sacramento, California. He has been using it with CTI (cyclohexane triisocyanate) and PAPI (polymethylene polyphenyl isocyanate). Typically, they have been mixing the ingredients at elevated temperature, degassing, and curing at 70° - 80°C. The hydroxyl functionality of L-2344 is estimated at approximately 1.8. Long chain perfluoro alcohols were too polar to be useful as a diluent. Some work was carried out in creating a -COOH terminal group. Dr. Bringer will check into this. It would be quite acidic due to adjacent fluorine atoms.

Dr. Bringer made several other comments worthy of note:

1. L-2146 flows at 150°F.

2. Kel-F 800 has less hydrogen than the Fluorels (25 M % vinylidene fluoride/75% CTFE), but it is only slightly flexible. It can be cured in similar manner as Fluorel, or with peroxides.

3. Vinylidene fluoride increases solubility of the polymer.

4. High surface area silicas (Aerosil, Cabosil) seem to increase flammability.

5. 3M has not conducted decomposition studies. However, he has been in contact with an outfit in Cincinnati which apparently is (or was) doing pyrolysis studies. He will also check with the 3M toxicologist.

6. Samples of KF-2140 "special", L-2344, and any laboratory samples of a -COOH terminated Fluorel will be sent A. S. A. P. Conventional materials are available out of Los Angeles 3M stock.

Jerry Hartke was also contacted re applicable 3M contracts with governmental agencies. Mr. Hartke is to forward a list containing contract number, agency, description of work, reports, etc.

12. NASA, AMES RESEARCH CENTER, Moffett Field, California - 11/22/68

Mr. Lieberman met with Dr. John Parker, Sal Riccitiello, and Carr Neel of Ames. John Haney of NCR, Dayton, Ohio, one of their contractors was also present. Their entire program is for the suppression or quenching of aircraft and spacecraft fires, including reentry bodies. They have emphasized two approaches: (a) polyurethane foams and, (b) intumescent coatings. They have utilized (a) halogenated polymers, (b) inorganic salts and, (c) encapsulated volatile or reactive halogen - containing compounds, all via thermal activation. To date, they have not been particularly concerned with toxicity, odors, smoke, or electrical properties. In general, they are mainly interested in protecting equipment, not men.

I saw several films which showed hardware demonstrations of these materials.

They have developed intumescent, solvent release coatings based upon P - nitroaniline bisulfate. They are presently working on new formulations which will have lower moisture sensitivity and may be 100% solids.

They have investigated N, P, B, etc. type compounds. Mr. Riccitiello was reticent about revealing masses of data (good and bad results) except to the extent that they have published. They have no objections to Furane's use of the same materials which they report, including the special micro encapsulated ones from NCR.

They have a contract with Atlantic Research to "Evaluate Fire Quenching Materials". Dominic Cagliostro is the Ames monitor. It was commented that Atlantic Research has added microencapsulated agents to RTV's for potting, etc. Someone thought that it may have been for NASA-MSC.

Some of the data has been reported in:

"Preliminary Data from Studies of Fire Retardant Materials"; G. Goodwin, NASA, Ames Research Center; Feb. 27-28, 1968.

"Development of Polyurethane as Thermal Protection Systems for Controlling Fuel Fires in Aircraft Structures"; J. A. Parker, S. R. Riccitiello, et al; NASA, Ames Research Center.

"The Use of a Salt of P - Nitroaniline as a Component for Intumescent Coatings"; J. A. Parker, G. M. Follen, et al; NASA, Ames Research Center; SAMPE Jnl; August/September, 1968.

More details on many of the above items will be requested from Carr Neel.

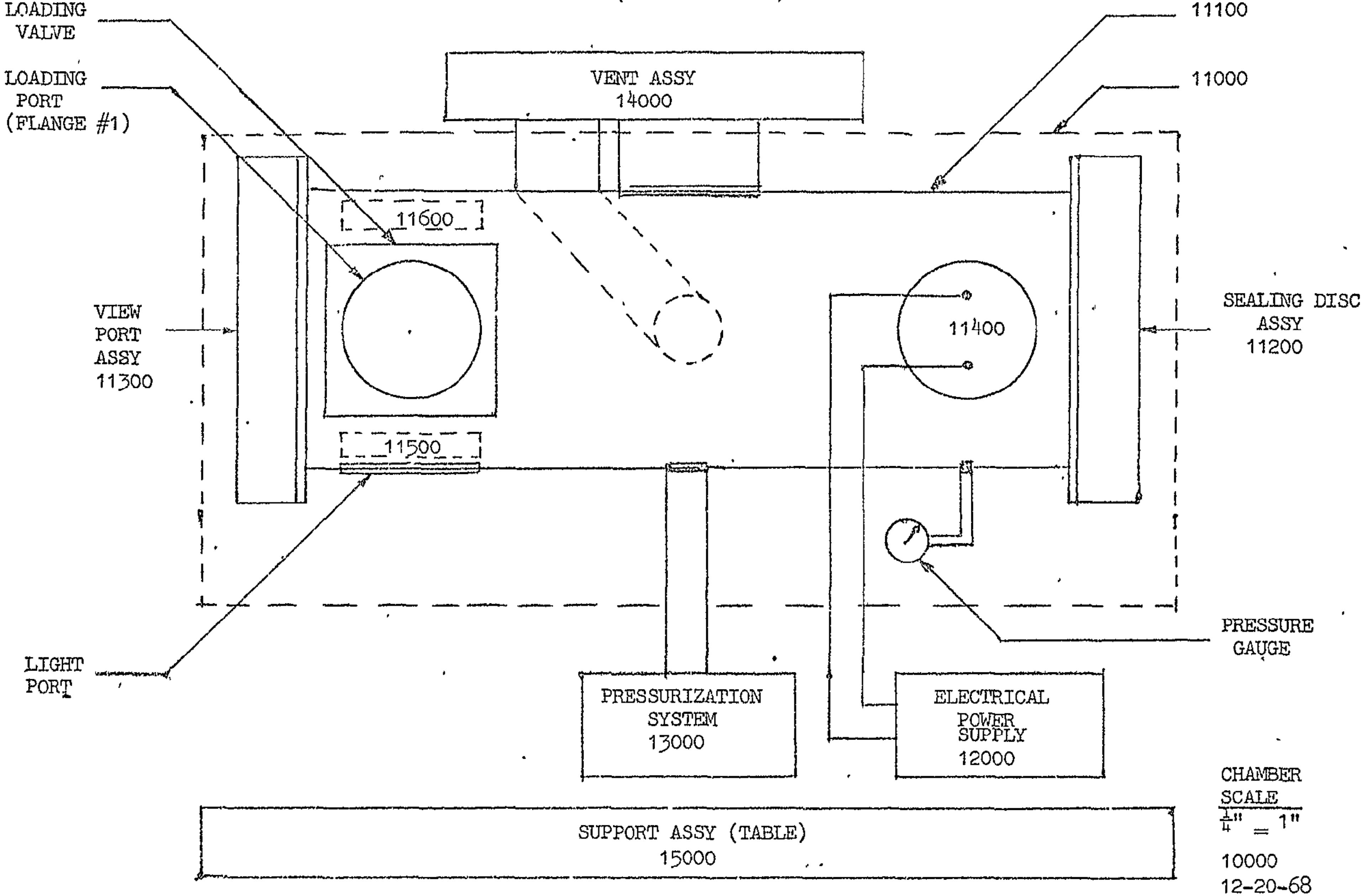
APPENDIX

- H -

NASA/FURANE
FLAMMABILITY
TEST FACILITY

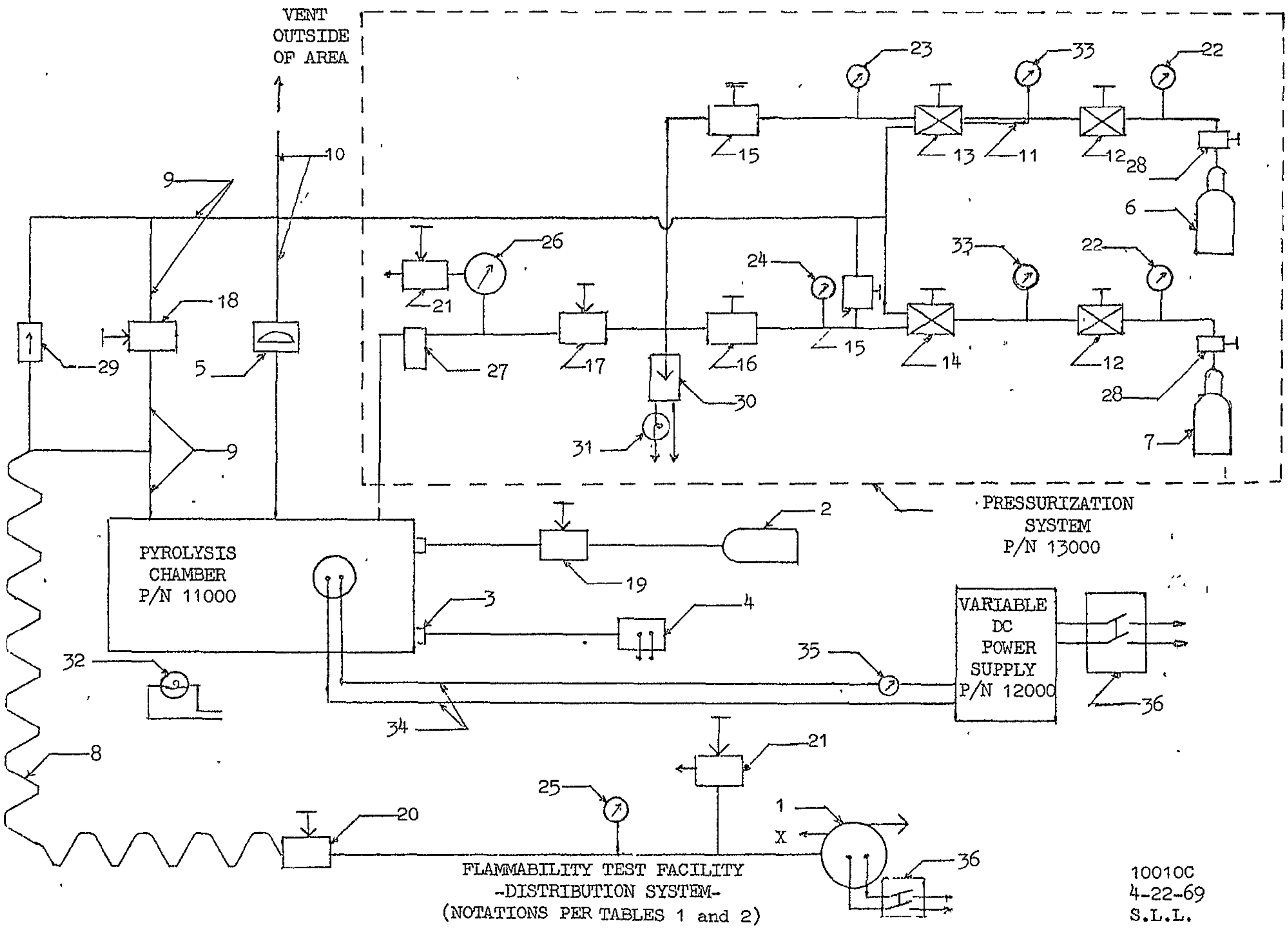
7④

FIGURE 1
FLAMMABILITY TEST FACILITY
(BLOCK DIAGRAM)



CHAMBER SCALE
$\frac{1}{4}" = 1"$

10000
12-20-68
SLL



FLAMMABILITY TEST FACILITY
-DISTRIBUTION SYSTEM-
(NOTATIONS PER TABLES 1 and 2)

FIGURE 2

10010C
4-22-69
S.L.L.

TABLE 1

LEGEND

(See Figure 2)

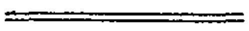
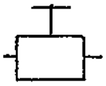
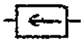
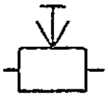
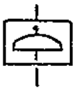
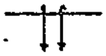
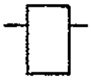
Schematic	Name
	Bellows line
	Copper tubing
	Pressure valve (manual)
	Check valve
	Vacuum valve (manual)
	Regulator
	Gauge
	Pressure switch
	Vacuum pump
	Burst diaphragm
	Light
	110 v. AC power feed
	Gas cylinder
	Filter

TABLE 2

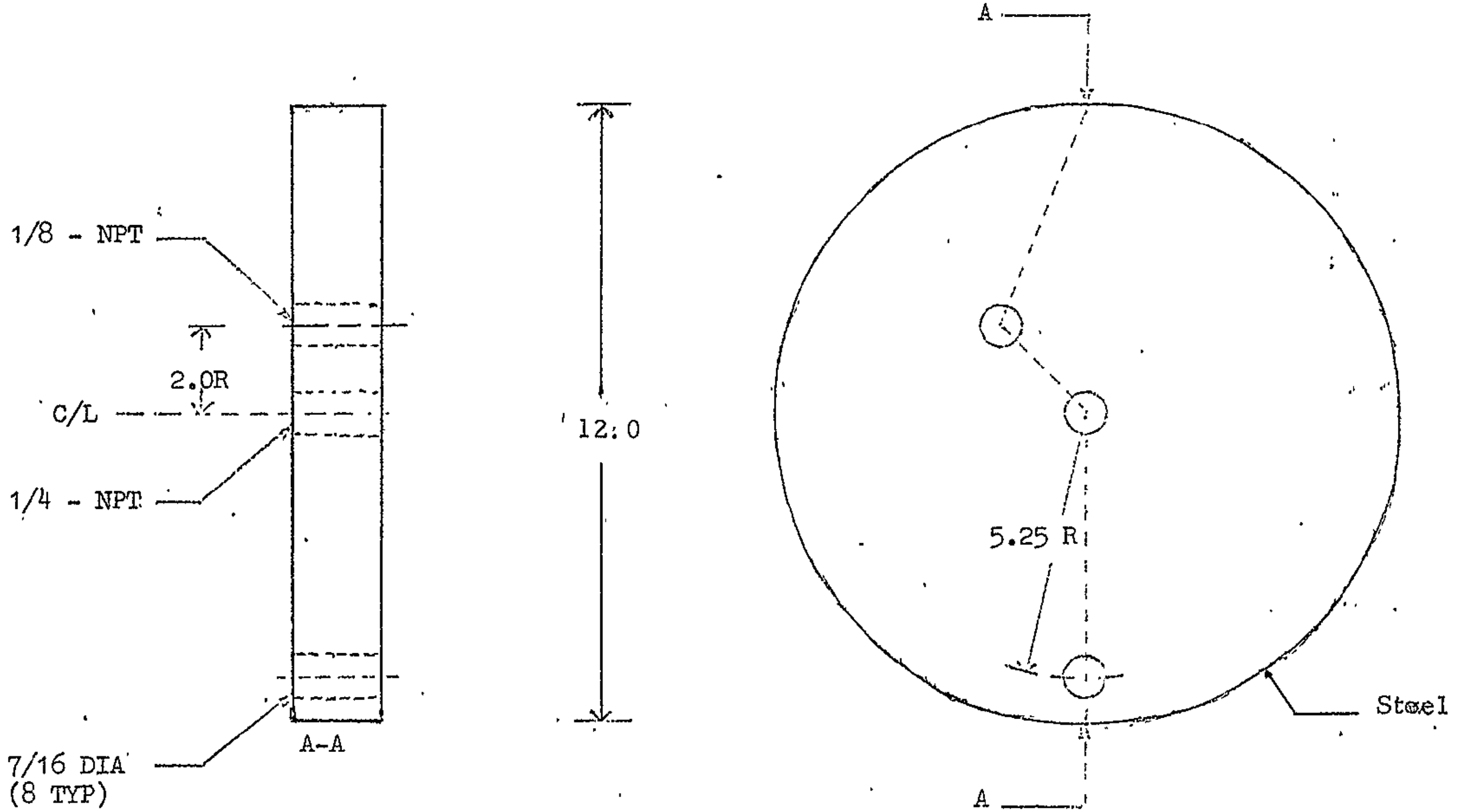
BILL OF MATERIALS
(See Figure 2)

PART NO.	REQ/ASSY.	MATERIAL/COMPONENT	STOCK SIZE
-1	1	Mechanical Vacuum Pump	KC-8 (Kinney)
-2	1	Stainless Steel	4 Liter Flask
-3	1	TG Transducer Gland with I/C Thermocouple	MTG-20-A4 (Conax)
-4	1	Temperature Recorder	Mod. 122 (Rust Rak)
-5	1	P/N 14100	
-6	1	N_2 Container	1A (hi-pressure)
-7	1	O_2 Container	1A (hi-pressure)
-8	1	Bellows Copper Tubing with Flange and Pipe Fittings	1" I.D. x 3'
-9	1	S.S. Bellows with Wire Overwrap	2.0" OD x 20.6" L
-10	1	T-304 Tubing	3.0" OD x .083 W x 34.74 L
-11		Copper Tubing	1/4" diameter, length as required
-12	2	Valve	Type 11-010-027 (Norgren)
-13	1	Valve	Mod. 40 AE 50 (Moore)
-14	1	Valve	Mod. 40 AE 30 (Moore)
-15	2	Valve	1/4" Mod. 400 WOG (Lunkenheimer)
-16	1	Valve	1/4" Mod. 180 (Powell)
-17	1	Valve	1/4" RB 493 (Hoke)
-18	1	Valve	2" VST - 22M2 (CVC)
-19	1	Valve	1/4" 4112-MAY (Hoke)
-20	1	Valve: Brass with Copper Bellows	1-1/2" (Veeco)

TABLE 2 (continued)

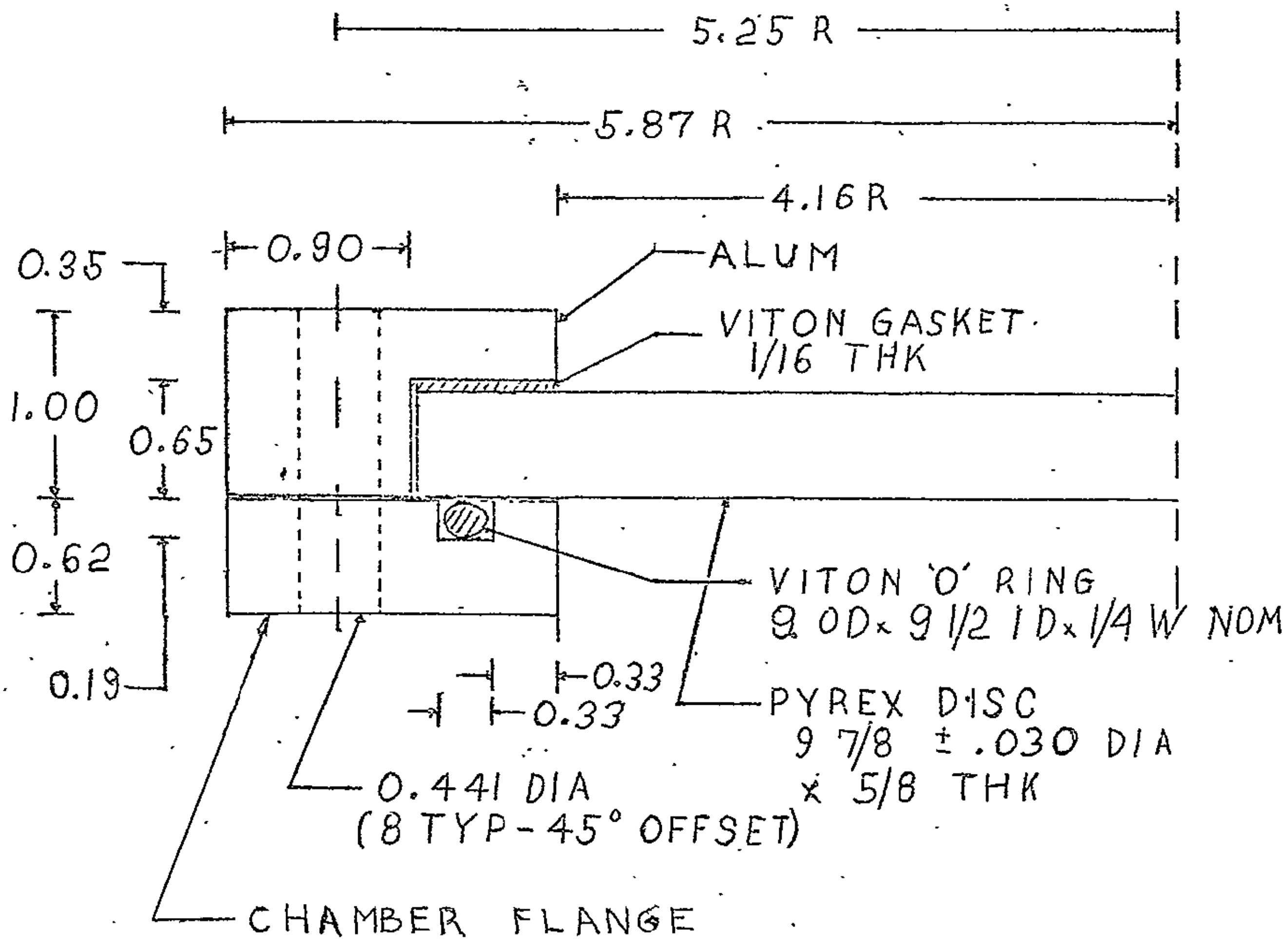
[illegible]RT NO.	REQ/ASSY.	MATERIAL/COMPONENT	STOCK SIZE
-21	1	Valve	1/4" A432 (Hoke)
-22	2	Gauge: 0-3,000 psi	683 (Motometer)
-23	1	Gauge: 0-5 psi	(U.S. Gauge)
-24	1	Gauge: 0-5 psi	(Marshalltown)
-25	1	Gauge: 0-30 inches	(Marshalltown)
-26	1	Gauge: 0-20 psi	FA145181 (Wallace & Tiernan)
-27	1	12-25 micron bronze element	3545 (Hoke)
-28	2	Tank Valve	(Airco)
-29	1	Check Valve	230B (Circle Seal)
-30	1	Pressure Switch	68000 (Hoke)
-31	1	Red Flood Light	100 PAR/P (G.E.)
-32	1	High Intensity Lamp	8034 (Lightolier)
-33	2	Gauge: 0-200 psi	2 1/2 - 1009 KGS (Ashcroft)
-34	2	Cable: Automotive Battery	Open Source
-35	1	Ammeter: 150A	(Simpson)
-36	2	Main Ckt. breaker	Open Source

FIGURE 3
SEALING DISC ASSY.



11200
1/21/69
S.L.L.

FIGURE 4
VIEW PORT ASSY

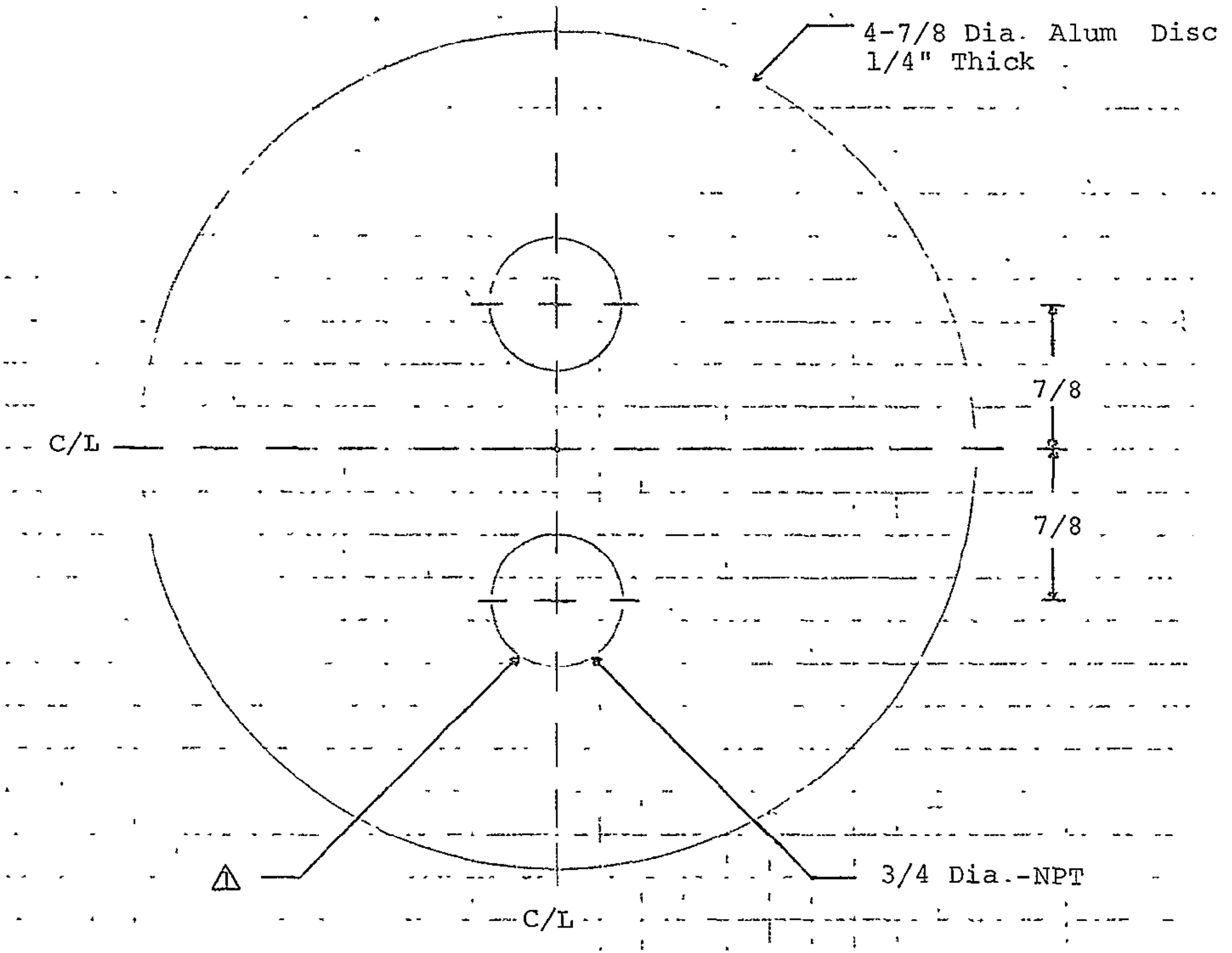


1130.0
12-5-68
S.L.L.

FIGURE 5

P/N 11400A

DC POWER FEEDTHROUGH



NOTE:

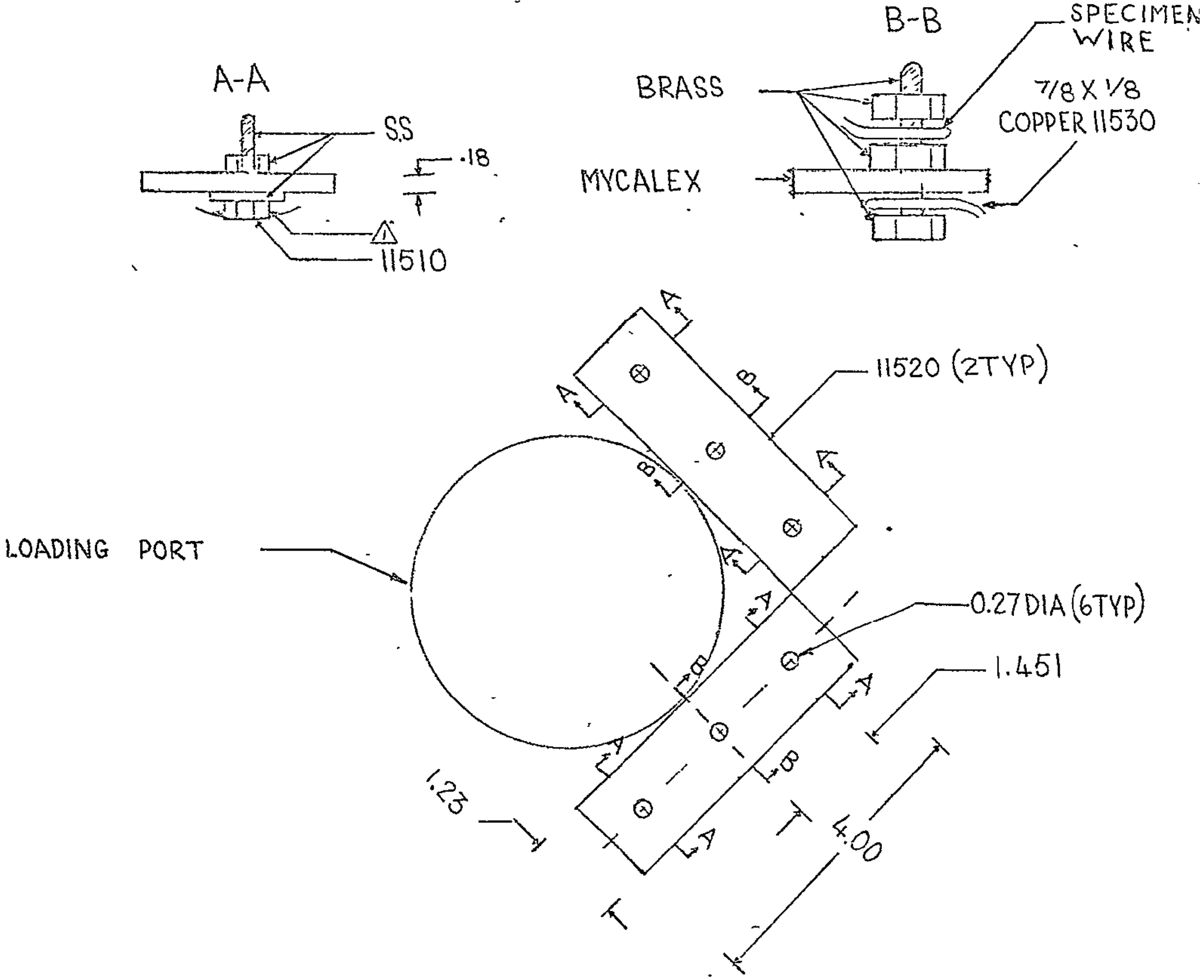
⚠ Using teflon sealing tape on threads, mount EGT-375-A-CU electrical conductor sealing gland (Conax Corp) in each hole. Torque to 90-100 ft -lbs

2-10-69
S.L.L.

FIGURE 6

SPECIMEN POWER ASSY.

P/N 11500

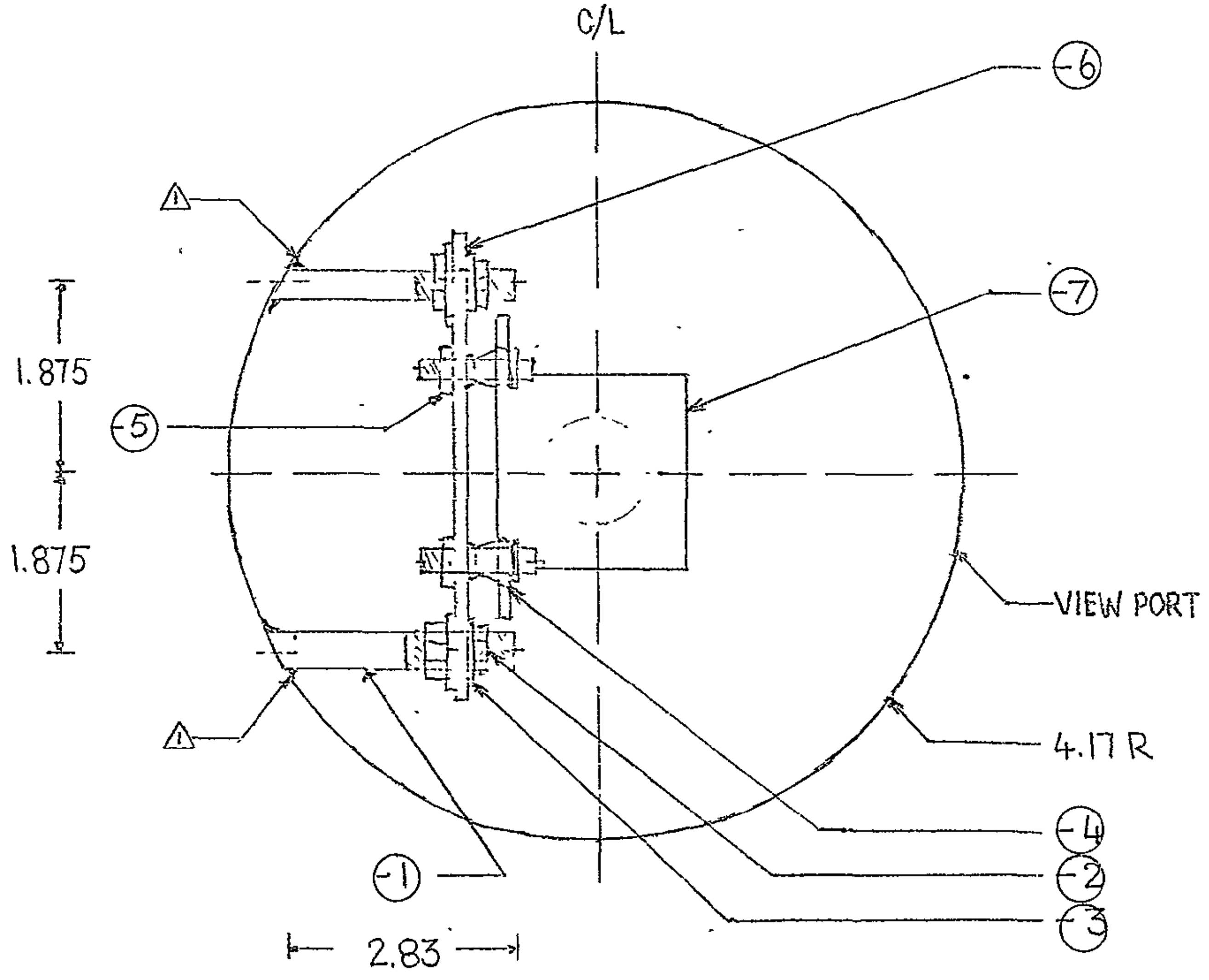


NOTE:

1 WELD BOLT HEADS TO P/N 11100

S.L.L.
11-29-68

FIGURE 7
SPECIMEN HOLDING FIXTURE ASSEMBLY
P/N 11600



NOTE:

△1 WELD AS REQUIRED

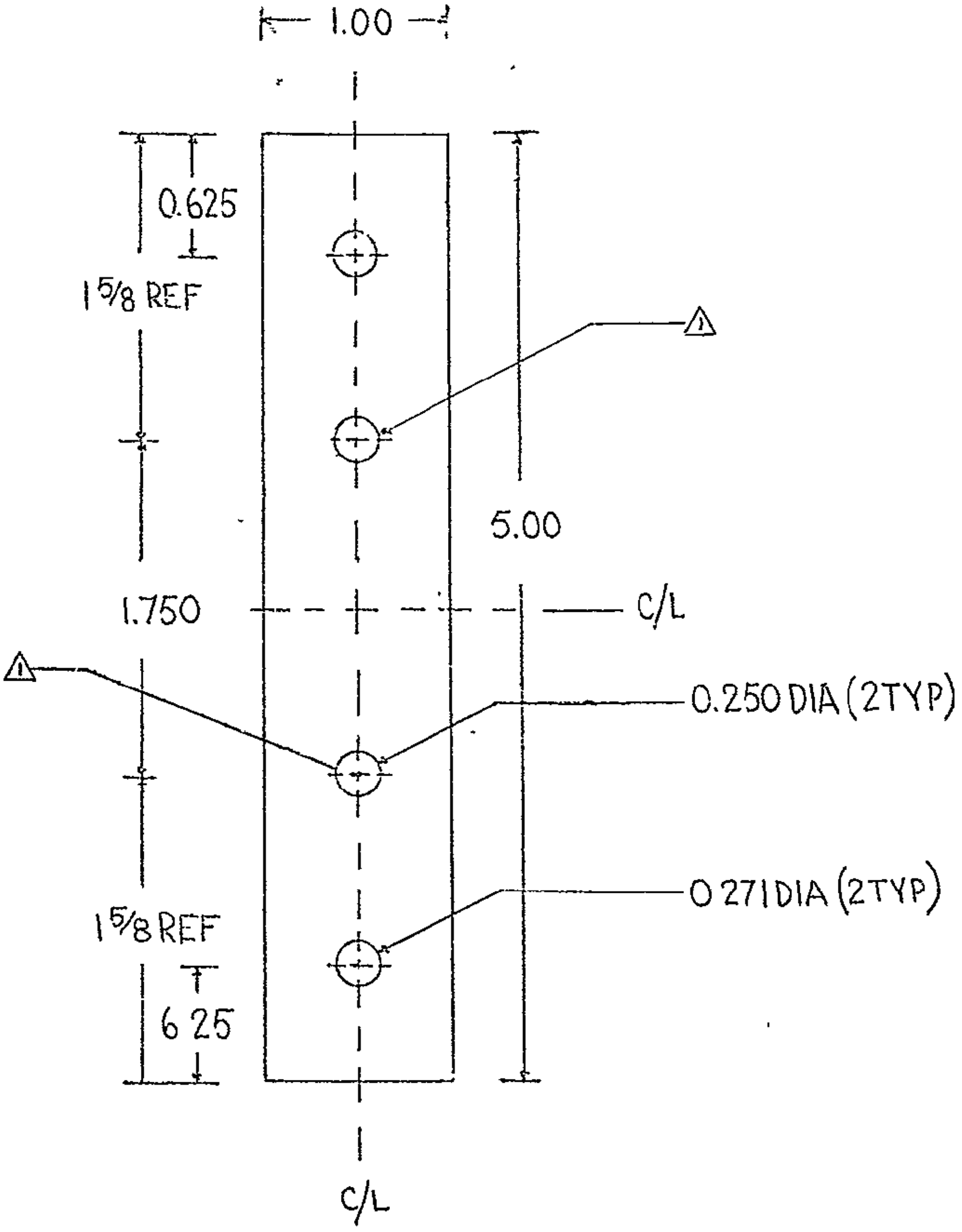
BILL OF MATERIAL

PART Nº	REQ/ASSY	MATERIAL	STOCK SIZE
-1	2	SS. BOLT	2.83"LX 1/4-20, LAST 0.87"
-2	4	SS NUT	1/4-20
-3	4	SS WASHER	
-4	2	BANANA PLUG - MALE	P/N 33-058(GC ELECTRONICS)
-5	2	BANANA PLUG - FEMALE	P/N 33-192(GC ELECTRONICS)
-6	1	P/N 11620	
-7	1	P/N 11630	

S.L.L
12-14-68

FIGURE 8

SPECIMEN HOLDER SUPPORT PLATE



NOTE:
⚠ MOUNT BANANA JACK
IN 33-192 (GC ELECTRONICS)

TOLERANCE
.XX ± .030
.XXX ± .010

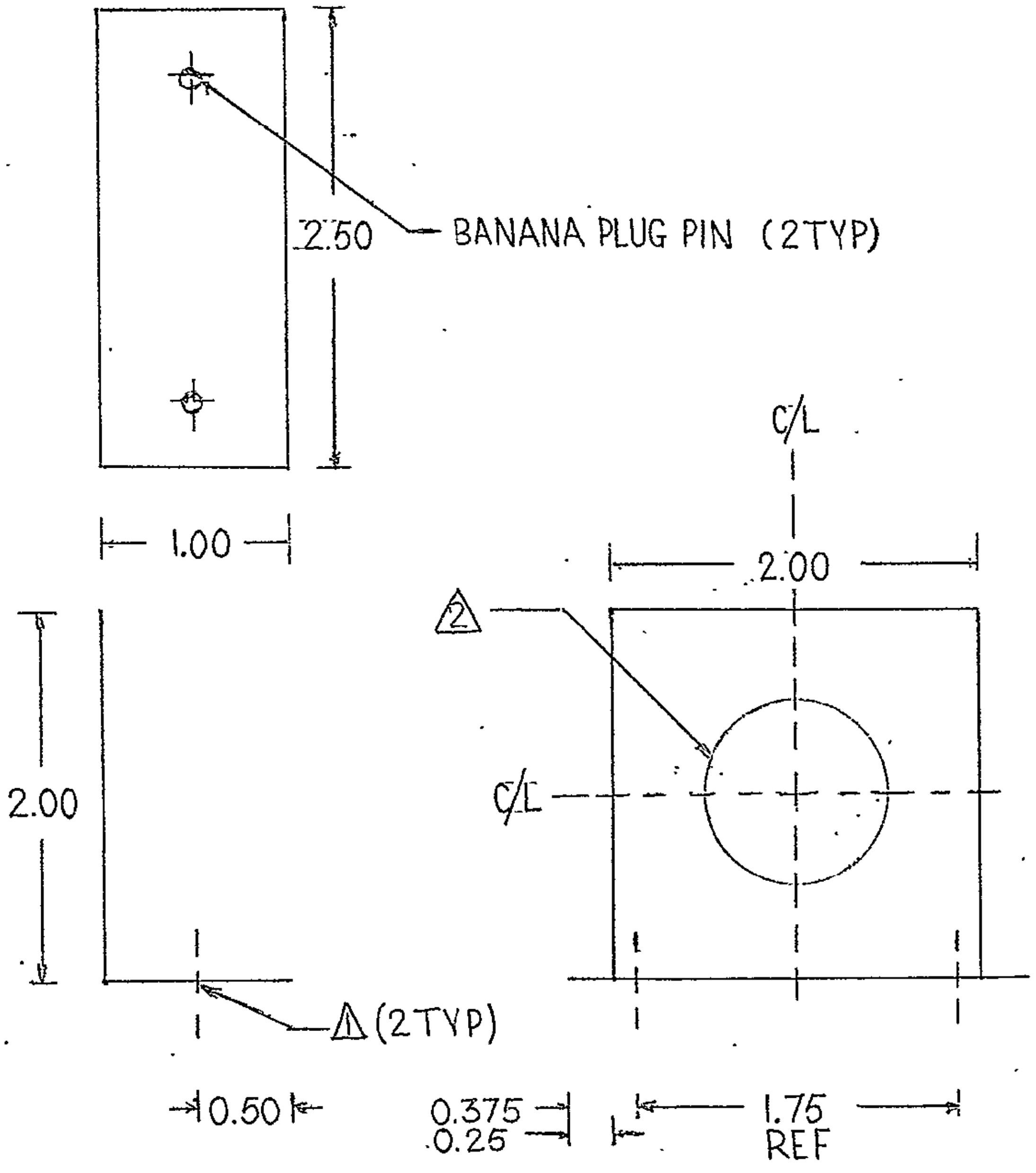
MATERIAL
ALUM, 1/8" THK

11620

S.L.L.
1-4-69

FIGURE 9

CONNECTOR HOLDER



NOTE

△1 DRILL FOR 8-32 THREADED END OF BANANA PLUG PIN 33-058 (G.C. ELECTRONICS)

△2 DRILL TO FIT BENDIX PYGMY CONNECTOR

TOLERANCE:

.XX ± .030
.XXX ± .010

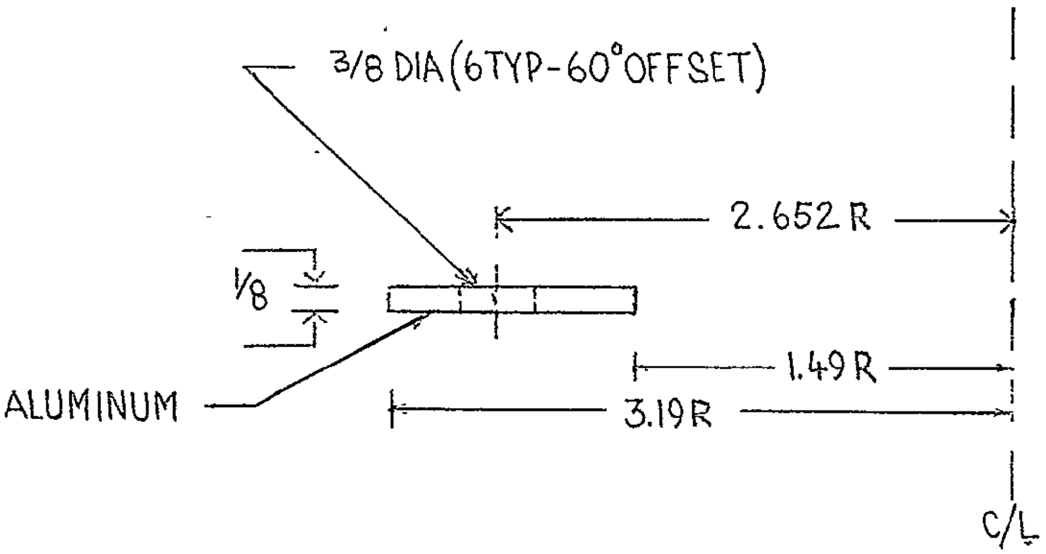
MATERIAL:

0.040 ALUMINUM OR ALCLAD

11630
S.L.L. 1-4-69

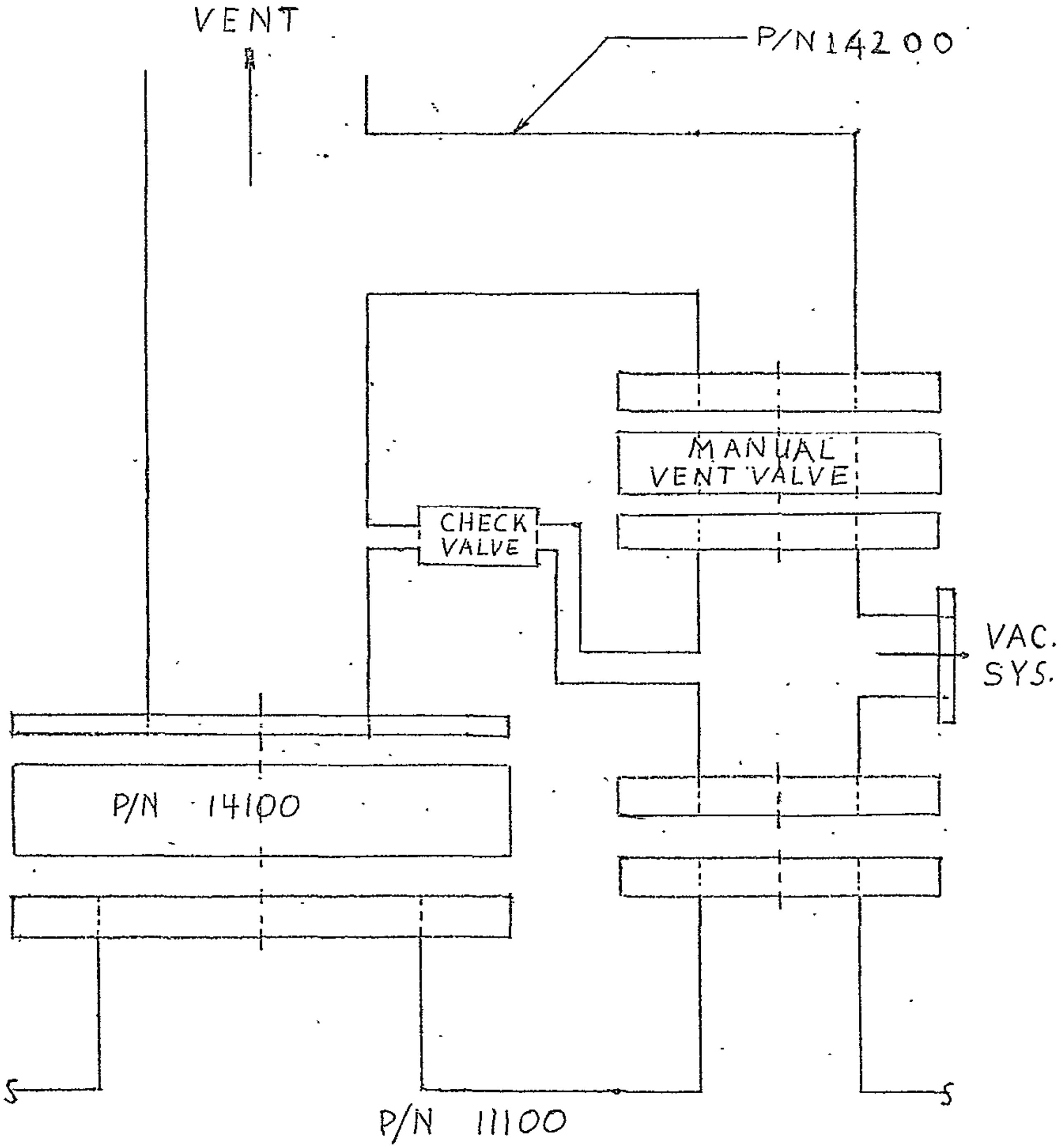
FIGURE 10

P/N 11700: LOADING VALVE 'O' RING INTERFACE RING



S.L.L.
12·2·68

FIGURE 11
VENT ASSY
(BLOCK DIAGRAM)

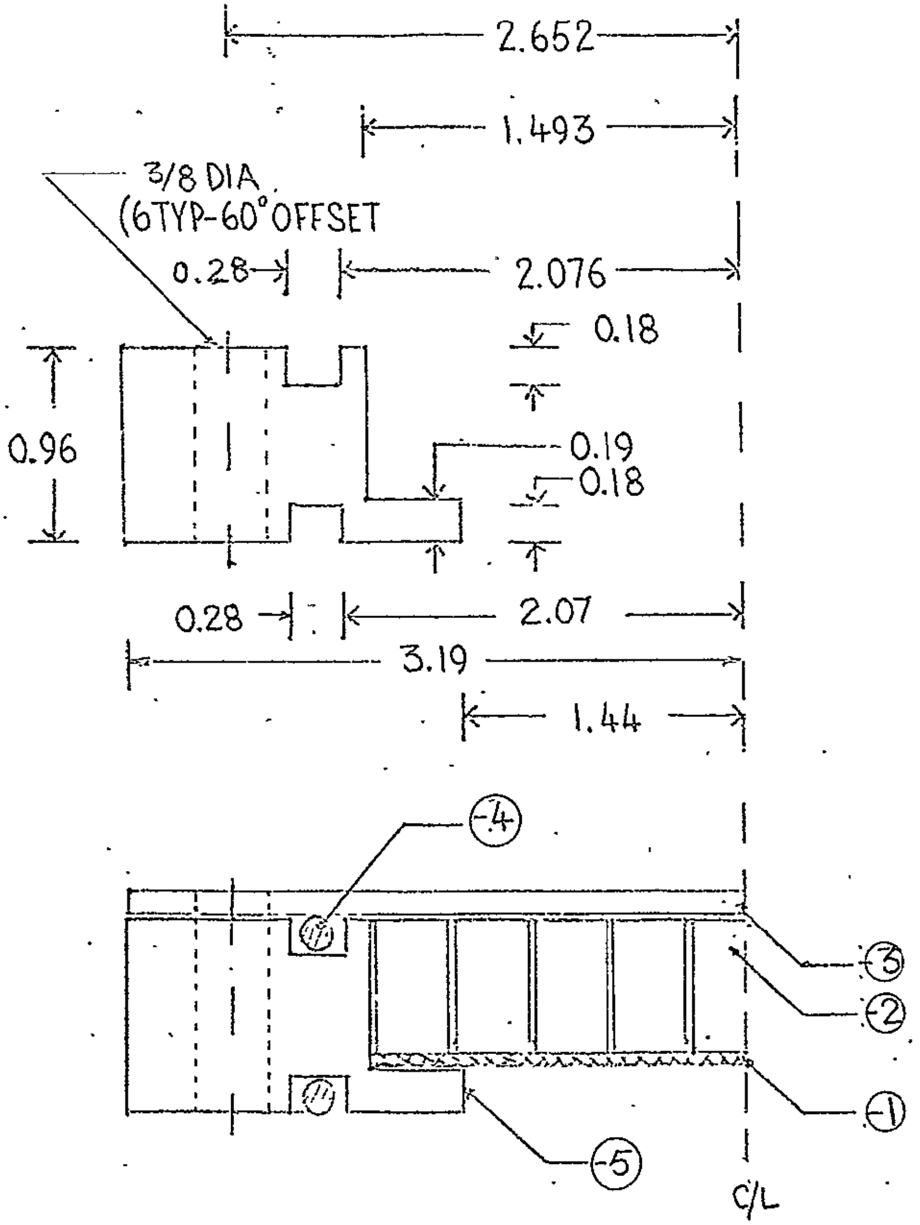


14000
SLL
12-24-69

FIGURE 12

P/N 14100A

RUPTURE DISC ASSY

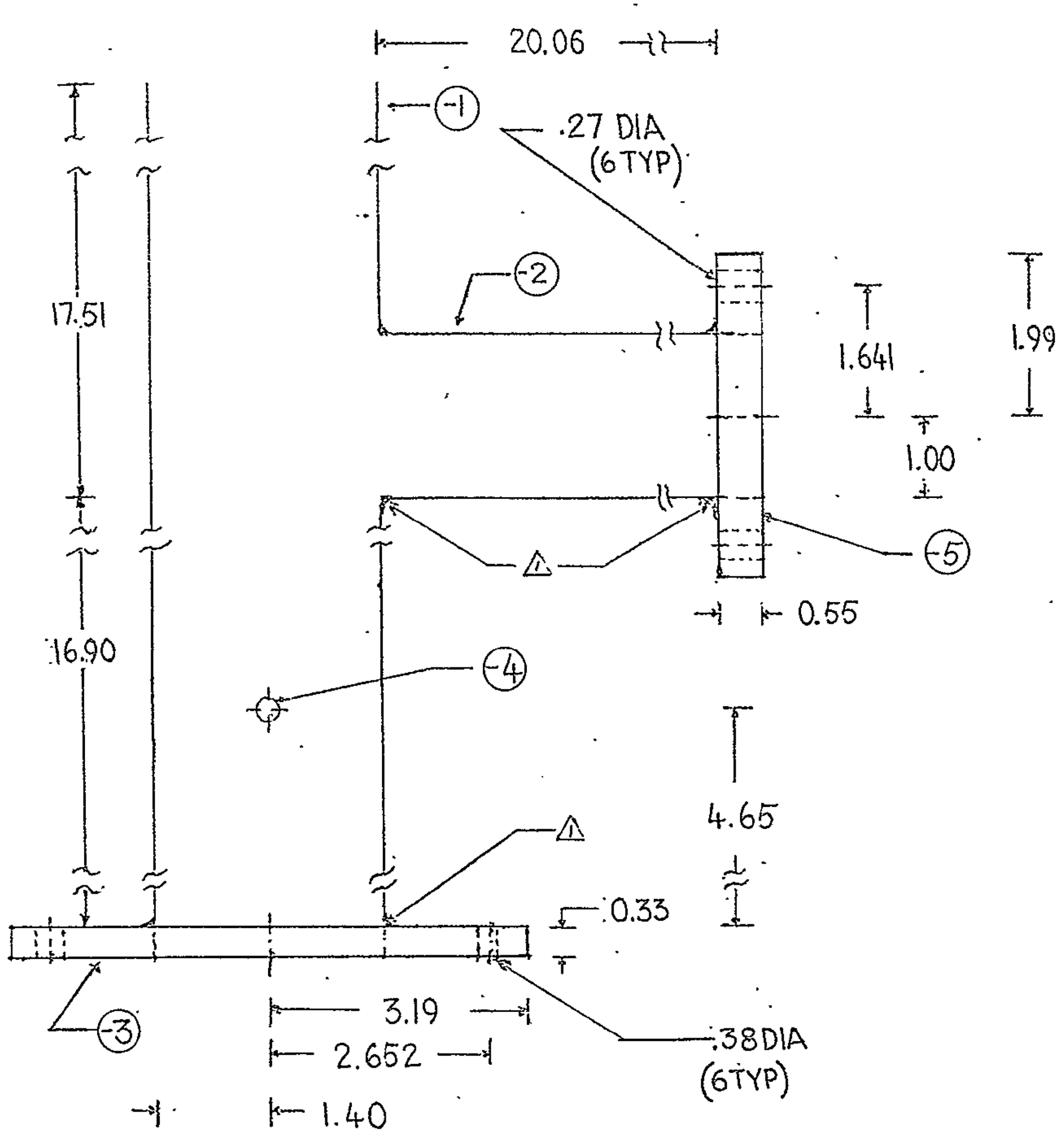


BILL OF MATERIALS:

PART Nº	REQ/ASSY	MATERIAL	STOCK SIZE
-1	1	S.S. WOVEN WIRE FABRIC	0.050 (NOMINAL) THK
-2	1	HONEYCOMB CORE PH 15-7MO WELDED	3/4 THK X 1/4 CELLS X .002 FOIL (ROHR)
-3	1	FOIL, ALUM	A.R. FOR 20 PSIA RUPTURE
-4	2	VITON 'O' RING	2-346-77-545 (PARKER)
-5	1	ALUM	6.38 DIA X .96 THK. SHT.

S.L.L.
12-11-68

FIGURE 13
VENT TUBE
P/N 14200



BILL OF MATERIALS:

PART No.	REQ/ASSY.	MATERIAL	STOCK SIZE
-1	1	T-304 TUBING	3.0" OD X .083 W X 34.74 L
-2	1	S.S. BELLOWS WITH WIRE OVERWRAP	2.0" OD X 20.6"
-3	1	S.S FLANGE	3.19 OD X 1.40 ID X 0.33 W
-4	1	S.S. STUB (NPT. LAST .44")	1.93 L X .48 OD X .07 W
-5	1	S.S FLANGE	1.99 OD X 1.00 ID X 0.55 W

S.L.L.
1·7·69

- APPENDIX I -

LABORATORY CHECKOUT & OPERATIONAL PROCEDURE

TITLE ELECTRICAL POTTING AND COATINGS FLAMMABILITY TEST

TM 9-8750- 1

DATE 22 April 1969

PAGE 1 OF 5

PREPARED BY: S. L. Lieberman

APPROVED BY: W. J. Dewar

FURANE Q C: A. George

FURANE S O: J. Ramsey

GAS TYPE	PRESSURE (psia) PARTIAL	TOTAL
Air	~14.7	~ 14.7
-	-	

-GENERAL SAFETY PRECAUTIONS-

ALL PERSONNEL MUST WEAR SAFETY GLASSES OR GOGGLES AND FACE SHIELDS WHILE IN IMMEDIATE VICINITY OF TEST CHAMBER DURING TESTING OR PRESSURIZED CHECKOUT.

-WARNING-

SMOKING, MATCHES, OR OPEN FLAMES ARE NOT PERMITTED IN THE IMMEDIATE TESTING AREA.

1.0 EQUIPMENT CHECKOUT (Delete if already carried out within preceeding 4 hours.) (REF: DWG. No. 10010C)

1.1 Close valves V-3 through-14 inclusive, and turn switches SW-1 and SW-2 to OFF (down).

1.2 Turn on power at main fuse box in laboratory.

1.3 Open vent valve V-9.

1.4 Turn vacuum pump switch SW-1 to ON (up).

1.5 Fully open gage vacuum valve V-8.

1.6 When a constant reading is observed on gage G-7, close valve V-8.

1.7 Turn switch SW-1 to OFF (down).

1.8 After one minute, gage G-7 shall not have lost more than 0.019 psi vacuum (1 gage div. = 0.025 psi). Testing shall not proceed until any leakage greater than 0.019 psi/min. is reduced to less than or equal to this allowable limit.

1.9 Close valve V-9.

1.10 Turn switch SW-1 to ON (up).

1.11 Fully open vacuum pump line valve V-12.

1.12 When a constant reading is observed on gage G-7, close valve V-12.

1.13 Turn switch SW-1 to OFF (down).

1.14 After one minute, gage G-7 shall not have lost more than 0.019 psi vacuum. Testing shall not proceed until any leakage greater than 0.019 psi/min. is reduced to less than or equal to this allowable limit.

1.15 Open nitrogen cylinder valve V-11.

1.16 Nitrogen cylinder gage G-2 shall indicate not less than 100 psi.

1.17 Set nitrogen gage G-4 to read 10 ± 0.1 psi by adjusting the center screw on the nitrogen high pressure regulator V-2.

1.18 Set gage G-6 to read 2 ± 0.1 psi by adjusting the nitrogen low pressure regulator V-4.

1.19 Open vacuum isolation valve V-7.

1.20 Slowly open nitrogen low pressure valve V-6.

1.21 When the reading on gage G-7 is constant, adjust the pressure to 16.500 ± 0.025 psia with regulator V-4.

1.22 Close valve V-7.

1.23 After one minute, gage G-7 shall not have lost more than 0.019 psi pressure. Testing shall not proceed until any leakage greater than 0.019 psi/min. is reduced to less than or equal to this allowable limit.

1.24 Close valve V-4 so that gage G-6 reads zero.

1.25 Close valve V-6.

1.26 Close valve V-11.

1.27 Open valve V-9.

2.0 TEST

NOTE: UNLESS OTHERWISE STATED, ALL TEST DATA IS RECORDED ON FORM 9-8750-2A.

2.1 Record gage G-7 pressure on "Vacuum on case" line of Form 9-8750-2 A.

2.2 Prepare test specimen from cured material.

2.2.1 ¼" x ¼" x 1" wired block (Ref: DWG. No. 20100)

2.2.1.1 Strip 7/8" of insulation from each end of the wire.

2.2.1.2 Put a single clockwise, approximately 5/16" Dia., "U" loop in one stripped wire end, and a similar one, but counterclockwise, at the other end.

2.2.2 Connector (Ref: Test No. 5,MSC-D-NA-0002)

2.3 Open the loading port, insert the specimen, and connect each wire to the nut/bolt electrical connection on the center of each Micalex insulator (Ref: DWG. No. 11500).

2.4 Position the block specimen inside the chamber by bending, as required, its wires so that it can be easily observed in the view port via the mirror.

2.5 Remove hand from chamber without disturbing the specimen and close the loading port.

2.6 Close valve V-9.

2.7 Turn ON (up) power supply switch SW-2 and adjust the current on gage G-8 to 55. ± 2.5 amperes. If ignition or considerable degradation is not obtained in one minute, increase the current by 5 amperes (i.e. from 55 to 60, and 60 to 65, etc.) until such time as the wire fails or ignition occurs. If the wire fails, maintain the voltage to the wires until it is positively established that current does not flow by bridging insulation gaps.

WARNING: DO NOT TOUCH POWER SUPPLY
TERMINALS OR LEADS DURING
THE REMAINDER OF THE TEST

2.8 Observe the test and record data specified on Test Form 9-8750-2A.

2.9 When visible specimen degradation no longer occurs, turn switch SW-2 to OFF (down).

2.10 Open valve V-11.

2.11 Adjust regulator V-4 to 2. ± 0.1 psi on gage G-6.

2.12 Open valve V-7.

2.13 Fully open valve V-6 slowly.

2.14 When gage G-7 reads 16. ± 0.5 psia, open valve V-9.

WARNING: TEFLON INSULATION IS USED ON
EVERY TEST FOR NAS 9-8750.
PURGE THE CHAMBER FOR NOT LESS
THAN THREE MINUTES.

2.15 After three minutes, close Valve V-9.

2.16 Close valve V-7.

2.17 Close valve V-6.

2.18 Adjust regulator V-4 until gage G-6 reads zero.

2.19 Turn switch SW-1 to ON (up).

2.20 Fully open valve V-12.

2.21 When a constant reading is observed on gage G-7, close valve V-12.

2.22 Turn switch SW-1 to OFF (down).

NOTE: OPEN VALVE V-9 SLOW ENOUGH
TO PREVENT JERKING MOTION
OF GAGE G-7 NEEDLE.

2.23 Fully open valve V-9.

2.24 Open loading port. Observe and record condition of specimen.

2.25 Carefully remove all sample residue from the chamber.

2.26 Close the loading port.

2.27 Close valve V-9

2.28 Close valve V-11.

2.29 Turn off power at main fuse box in laboratory.

- APPENDIX J -

LABORATORY CHECKOUT & OPERATIONAL PROCEDURE

TITLE ELECTRICAL POTTING AND COATINGS FLAMMABILITY TEST

TM 9-8750- 2

DATE 22 April 1969

PAGE 1 OF 6

PREPARED BY: S. L. Lieberman

APPROVED BY: W. J. Dewar

FURANE Q C: A. George

FURANE S O: J. Ramsey

GAS TYPE	PRESSURE (psia) PARTIAL	TOTAL
Oxygen	6.2	6.2
-	-	

-GENERAL SAFETY PRECAUTIONS-

ALL PERSONNEL MUST WEAR SAFETY GLASSES OR GOGGLES AND FACE SHIELDS WHILE IN IMMEDIATE VICINITY OF TEST CHAMBER DURING TESTING OR PRESSURIZED CHECKOUT.

-WARNING-

SMOKING, MATCHES, OR OPEN FLAMES ARE NOT PERMITTED IN THE IMMEDIATE TESTING AREA.

1.0 EQUIPMENT CHECKOUT (Delete if already carried out within preceeding 4 hours.) (REF.: DWG. No: 10010C).

1.1 Close valves V-3 through -14, inclusive, and turn switches SW-1 and SW-2 to OFF (down).

1.2 Turn on power at main fuse box in laboratory.

1.3 Open vent valve V-9.

1.4 Turn vacuum pump switch SW-1 to ON (up).

1.5 Fully open gage vacuum valve V-8.

1.6 When a constant reading is observed on gage G-7, close valve V-8.

1.7 Turn switch SW-1 to OFF (down).

1.8 After one minute, gage G-7 shall not have lost more than 0.019 psi vacuum (1 gage div. = 0.025 psi). Testing shall not proceed until any leakage greater than 0.019 psi/min. is reduced to less than or equal to this allowable limit.

1.9 Close valve V-9.

1.10 Turn switch SW-1 to ON (up).

1.11 Fully open vacuum pump line valve V-12.

1.12 When a constant reading is observed on gage G-7, close valve V-12.

1.13 Turn switch SW-1 to OFF (down).

1.14 After one minute, gage G-7 shall not have lost more than 0.019 psi vacuum. Testing shall not proceed until any leakage greater than 0.019 psi/min. is reduced to less than or equal to this allowable limit.

1.15 Open nitrogen cylinder valve V-11.

1.16 Nitrogen cylinder gage G-2 shall indicate not less than 100 psi.

1.17 Set nitrogen gage G-4 to read 10 ± 0.1 psi by adjusting the center screw on the nitrogen high pressure regulator V-2.

1.18 Set gage G-6 to read 2 ± 0.1 psi by adjusting the nitrogen low pressure regulator V-4.

1.19 Open vacuum isolation valve V-7.

1.20 Slowly open nitrogen low pressure valve V-6.

1.21 When the reading on gage G-7 is constant, adjust the pressure to 16.500 ± 0.025 psia with regulator V-4.

1.22 Close valve V-7.

1.23 After one minute, gage G-7 shall not have lost more than 0.019 psi pressure. Testing shall not proceed until any leakage greater than 0.019 psi/min. is reduced to less than or equal to this allowable limit.

1.24 Close valve V-4 so that gage G-6 reads zero.

1.25 Close valve V-6.

1.26 Close valve V-11.

1.27 Open valve V-9.

2.0 TEST

<u>NOTE:</u> UNLESS OTHERWISE STATED,
ALL TEST DATA IS RECORDED
ON FORM 9-8750-2A.

2.1 Record gage G-7 pressure on "Vacuum on case" line of Form 9-8750-2A.

2.2 Prepare test specimen from cured material.

2.2.1 1/4" x 1/4" x 1" wired block (Ref: DWG. No. 20100).

2.2.1.1 Strip 7/8" of insulation from each end of the wire.

2.2.1.2 Put a single clockwise, approximately 5/16" dia. "U" loop in one stripped wire end, and a similar one, but counterclockwise, at the other end.

2.2.2 Connector (Ref: Test No. 5, MSC-D-NA-0002).

2.3 Open the loading port, insert the specimen, and connect each wire to the nut/bolt electrical connection on the center of each Micalex insulator (Ref: DWG. No. 11500).

2.4 Position the block specimen inside the chamber by bending, as required, its wires so that it can be easily observed in the view port via the mirror.

2.5 Remove hand from chamber without disturbing the specimen and close the loading port.

2.6 Close valve V-9.

2.7 Turn switch SW-1 to ON (up).

2.8 Fully open valve V-12.

2.9 When a constant reading is observed on gage G-7, close valve V-12.

2.10 Turn switch SW-1 to OFF (down). Record pressure reading on gage G-7 on Form 9-8750-2 in the box under the word "vac".

2.11 Open valve V-11.

2.12 Gage G-4 should read 10 ± 0.1 psi. Adjust center screw of regulator V-2 until such reading is obtained.

2.13 Open oxygen cylinder valve V-10.

2.14 Oxygen cylinder gage G-1 shall indicate not less than 100 psi.

2.15 Set oxygen gage G-3 to read 5 ± 0.1 psi by adjusting the center screw on the oxygen high pressure regulator V-1.

2.16 Set oxygen gage G-5 to read 2 ± 0.1 psi by adjusting the oxygen low pressure regulator V-3.

2.17 Open valve V-7.

2.18 Add 6.200 psia oxygen to the reading recorded in step 2.10 by bleeding oxygen with oxygen line pressure valve V-5, into system so that gage G-7 indicates this total pressure. Record this pressure on form in top box of "Test Setting".

2.19 Close valve V-5.

2.20 Close valve V-7.

2.21 Repeat steps 2.17 through 2.20, inclusive, until gage G-7 remains within ± 0.019 psi of required test setting for one minute. If pressure exceeds the limit value, determine cause of leak, repair as necessary, and repeat entire procedure to this point.

NOTE: VENT AND NITROGEN FLUSH
CHAMBER BEFORE CARRYING
OUT REPAIRS.

2.22 Turn regulator V-3 counterclockwise until gage G-5 reads zero.

2.23 Soak specimens five minutes for block specimens and ten minutes for connector specimens.

2.24 Turn power supply switch SW-2 to ON (up) and adjust the current on gage G-8 to 55. ± 2.5 amperes. If ignition or considerable degradation is not obtained in one minute, increase the current by 5 amperes (i.e. from 55. to 60., and 60. to 65., etc.) until such time as the wire fails or ignition occurs. If the wire fails, maintain the voltage to the wires until it is positively established that current does not flow by bridging insulation gaps.

WARNING: DO NOT TOUCH POWER
SUPPLY TERMINALS OR
LEADS DURING THE
REMAINDER OF THE TEST.

2.25 Observe the test and record data specified on Test Form 9-8750-2A.

2.26 When visible specimen degradation no longer occurs, turn switch SW-2 to OFF (down).

2.27 Adjust regulator V-4 to 2 ± 0.1 psi on gage G-6.

2.28 Open valve V-7.

2.29 Fully open valve V-6 slowly.

2.30 When gage G-7 reads 16. ± 0.5 psia, open valve V-9.

<u>WARNING:</u> TEFLON INSULATION IS USED ON EVERY TEST FOR NAS 9-8750. PURGE THE CHAMBER FOR NOT LESS THAN THREE MINUTES.

2.31 After three minutes close valve V-9.

2.32 Close valve V-7.

2.33 Close valve V-6.

2.34 Adjust regulator V-4 until gage G-6 reads zero.

2.35 Turn switch SW-1 to ON (up).

2.36 Fully open valve V-12.

2.37 When a constant reading is observed on gage G-7, close valve V-12.

2.38 Turn switch SW-1 to OFF (down).

<u>NOTE:</u> OPEN VALVE V-9 SLOW ENOUGH TO PREVENT JERKING MOTION OF GAGE G-7 NEEDLE.

2.39 Fully open valve V-9.

2.40 Open loading port. Observe and record condition of specimen.

2.41 Carefully remove all sample residue from the chamber.

2.42 Close the loading port.

2.43 Close valve V-9.

2.44 Close valves V-10 and V-11.

2.45 Turn off power at main fuse box in laboratory.

LABORATORY CHECKOUT & OPERATIONAL PROCEDURE

TITLE ELECTRICAL POTTING AND COATINGS FLAMMABILITY TEST

TM 9-8750- 3

DATE 22 April 1969

PAGE 1 OF 7

PREPARED BY: S. L. Lieberman

APPROVED BY: W. J. Dewar

FURANE Q C: A. George

FURANE S O: J. Ramsey

GAS TYPE	PRESSURE (psia) PARTIAL	TOTAL
Oxygen	9.72	16.20
Nitrogen	6.48	

-GENERAL SAFETY PRECAUTIONS-

ALL PERSONNEL MUST WEAR SAFETY GLASSES OR GOGGLES AND FACE SHIELDS WHILE IN IMMEDIATE VICINITY OF TEST CHAMBER DURING TESTING OR PRESSURIZED CHECKOUT.

-WARNING-

SMOKING, MATCHES, OR OPEN FLAMES ARE NOT PERMITTED IN THE IMMEDIATE TESTING AREA.

1.0 EQUIPMENT CHECKOUT (Delete if already carried out within preceeding 4 hours.) (REF: DWG. No. 10010C).

1.1 Close valves V-3 through -14, inclusive, and turn switches SW-1 and SW-2 to OFF (down).

1.2 Turn on power at main fuse box in laboratory

1.3 Open vent valve V-9.

1.4 Turn vacuum pump switch SW-1 to ON (up).

1.5 Fully open gage vacuum valve V-8.

1.6 When a constant reading is observed on gage G-7, close valve V-8.

1.7 Turn switch SW-1 to OFF (down).

1.8 After one minute, gage G-7 shall not have lost more than 0.019 psi vacuum (1 gage div. = 0.025 psi). Testing shall not proceed until any leakage greater than 0.019 psi/min. is reduced to less than or equal to this allowable limit.

1.9 Close valve V-9.

1.10 Turn switch SW-1 to ON (up).

1.11 Fully open vacuum pump line valve V-12.

1.12 When a constant reading is observed on gage G-7, close valve V-12.

1.13 Turn switch SW-1 to OFF (down).

1.14 After one minute, gage G-7 shall not have lost more than 0.019 psi vacuum. Testing shall not proceed until any leakage greater than 0.019 psi/min. is reduced to less than or equal to this allowable limit.

1.15 Open nitrogen cylinder valve V-11.

1.16 Nitrogen cylinder gage G-2 shall indicate not less than 100 psi.

1.17 Set nitrogen gage G-4 to read 10 ± 0.1 psi by adjusting the center screw on the nitrogen high pressure regulator V-2.

1.18 Set gage G-6 to read 2 ± 0.1 psi by adjusting the nitrogen low pressure regulator V-4.

1.19 Open vacuum isolation valve V-7.

1.20 Slowly open nitrogen low pressure valve V-6.

1.21 When the reading on gage G-7 is constant, adjust the pressure to 16.500 ± 0.025 psia with regulator V-4.

1.22 Close valve V-7.

1.23 After one minute, gage G-7 shall not have lost more than 0.019 psi pressure. Testing shall not proceed until any leakage greater than 0.019 psi/min. is reduced to less than or equal to this allowable limit.

1.24 Close valve V-4 so that gage G-6 reads zero.

1.25 Close valve V-6.

1.26 Close valve V-11.

1.27 Open valve V-9.

2.0 TEST

<u>NOTE</u>: UNLESS OTHERWISE STATED, ALL TEST DATA IS RECORDED ON FORM 9-8750-2A.

2.1 Record gage G-7 pressure on "Vacuum on case" line of Form 9-8750-2A.

2.2 Prepare test specimen from cured material.

2.2.1 1/4" x 1/4" x 1" wired block (Ref: DWG. No. 20100).

2.2.1.1 Strip 7/8" of insulation from each end of the wire.

2.2.1.2 Put a single clockwise, approximately 5/16" dia. "U" loop in one stripped wire end, and a similar one, but counterclockwise, at the other end.

2.2.2 Connector (Ref: Test No. 5, MSC-D-NA-0002).

2.3 Open the loading port, insert the specimen, and connect each wire to the nut/bolt electrical connection on the center of each Micalex insulator (Ref: DWG. No. 11500).

2.4 Position the block specimen inside the chamber by bending, as required, its wires so that it can be easily observed in the view port via the mirror

2.5 Remove hand from chamber without disturbing the specimen and close the loading port.

2.6 Close valve V-9.

2.7 Turn switch SW-1 to ON (up).

2.8 Fully open valve V-12.

2.9 When a constant reading is observed on gage G-7, close valve V-12.

2.10 Turn switch SW-1 to OFF (down). Record pressure reading on gage G-7 on Form 9-8750-2A in the box under the word "vac".

2.11 Open valve V-11.

2.12 Gage G-4 should read 10 ± 0.1 psi. Adjust center screw of regulator V-2 until such reading is obtained.

2.13 Open oxygen cylinder valve V-10.

2.14 Oxygen cylinder gage G-1 shall indicate not less than 100 psi.

2.15 Set oxygen gage G-3 to read 5 ± 0.1 psi by adjusting the center screw on the oxygen high pressure regulator V-1.

2.16 Set oxygen gage G-5 to read 2 ± 0.1 psi by adjusting the oxygen low pressure regulator V-3.

2.17 Open valve V-7.

2.18 Add 9.720 psia oxygen to the reading recorded in step 2.10 by bleeding oxygen with oxygen line pressure valve V-5, into system so that gage G-7 indicates this total pressure. Record this pressure on form in top box of "Test Setting" (with "0_2" in top box and "N_2" in bottom box, under "GAS" notation).

2.19 Close valve V-5.

2.20 Close valve V-7.

2.21 Repeat steps 2.17 through 2.20, inclusive, until gage G-7 remains within ± 0.019 psi of required test setting for one minute. If pressure exceeds the limit value, determine cause of leak, repair as necessary, and repeat entire procedure to this point.

NOTE: VENT AND NITROGEN FLUSH CHAMBER BEFORE CARRYING OUT REPAIRS.

2.22 Turn regulator V-3 counterclockwise until gage G-5 reads zero.

2.23 Set nitrogen gage G-6 to read 2. ± 0.1 psi by adjusting regulator V- 4.

2.24 Open valve V-7.

2.25 Add 6.480 psia nitrogen to reading recorded in step 2.18 by bleeding nitrogen into system with valve V-6 so that gage G-7 indicates this total pressure. Record this total pressure on form in bottom box of "Test Setting".

2.26 Close valve V-6.

2.27 Close valve V-7.

2.28 Repeat steps 2.24 through 2.27, inclusive, until gage G-7 remains within ± 0.019 psi of required test setting for one minute. If pressure exceeds the limit value, determine cause of leak, repair as necessary, and repeat entire procedure to this point.

NOTE: VENT AND NITROGEN FLUSH CHAMBER BEFORE CARRYING OUT REPAIRS.

2.29 Turn regulator V-4 counterclockwise until gage G-6 indicates zero.

2.30 Soak specimens five minutes for block specimens and ten minutes for connector specimens.

2.31 Turn power supply switch SW-2 to ON (up) and adjust the current on gage G-8 to 55. ± 2.5 amperes. If ignition or considerable degradation is not obtained in one minute, increase the current by 5. amperes (i.e. from 55. to 60., 60. to 65., etc.) until such time as the wire fails or ignition occurs. If the wire fails, maintain the voltage to the wires until it is positively established that current does not flow by bridging insulation gaps.

WARNING: DO NOT TOUCH POWER SUPPLY TERMINALS OR LEADS DURING THE REMAINDER OF THE TEST.

2.32 Observe the test and record data specified on Test Form 9-8750-2A.

2.33 When visible specimen degradation no longer occurs, turn switch SW-2 to OFF (down).

2.34 Adjust regulator V-4 to 2 ± 0.1 psi on gage G-6.

2.35 Open valve V-9.

2.36 Open valve V-7.

2.37 Fully open valve V-6 slowly.

WARNING: TEFLON INSULATION IS USED ON EVERY TEST FOR NAS 9-8750. PURGE THE CHAMBER FOR NOT LESS THAN THREE MINUTES.

2.38 After three minutes close valve V-9.

2.39 Close valve V-7.

2.40 Close valve V-6.

2.41 Adjust regulator V-4 until gage G-6 reads zero.

2.42 Turn switch SW-1 to ON (up).

2.43 Fully open valve V-12.

2.44 When a constant reading is observed on gage G-7, close valve V-12.

2.45 Turn switch SW-1 to OFF (down).

<u>NOTE:</u> OPEN VALVE V-9 SLOW
ENOUGH TO PREVENT
JERKING MOTION OF
GAGE G-7 NEEDLE.

2.46 Fully open valve V-9.

2.47 Open loading port, Observe and record condition of specimen.

2.48 Carefully remove all sample residue from the chamber.

2.49 Close the loading port.

2.50 Close valve V-9.

2.51 Close valves V-10 and V-11.

2.52 Turn off power at main fuse box in laboratory.

- APPENDIX L -

LABORATORY CHECKOUT & OPERATIONAL PROCEDURE

TITLE ELECTRICAL POTTING AND COATINGS FLAMMABILITY TEST

TM 9-8750- 4

DATE 22 April 1969

PAGE 1 OF 7

PREPARED BY: S. L. Lieberman

APPROVED BY: W. J. Dew[illegible]

FURANE Q C: A. George

FURANE S O: J. Ramsey

GAS TYPE	PRESSURE (psia) PARTIAL	TOTAL
Oxygen	16.5	16.5
-	-	

-GENERAL SAFETY PRECAUTIONS-

ALL PERSONNEL MUST WEAR SAFETY GLASSES OR GOGGLES AND FACE SHIELDS WHILE IN IMMEDIATE VICINITY OF TEST CHAMBER DURING TESTING OR PRESSURIZED CHECKOUT.

-WARNING-

SMOKING, MATCHES, OR OPEN FLAMES ARE NOT PERMITTED IN THE IMMEDIATE TESTING AREA.

1.0 EQUIPMENT CHECKOUT (Delete if already carried out within preceeding 4 hours.) (REF.: DWG. No. 10010C).

1.1 Close valves V-3 through -14, inclusive, and turn switches SW-1 and SW-2 to OFF (down).

1.2 Turn on power at main fuse box in laboratory.

1.3 Open vent valve V-9.

1.4 Turn vacuum pump switch SW-1 to ON (up).

1.5 Fully open gage vacuum valve V-8.

1.6 When a constant reading is observed on gage G-7, close valve V-8.

1.7 Turn switch SW-1 to OFF (down).

1.8 After one minute, gage G-7 shall not have lost more than 0.019 psi vacuum (1 gage div. = 0.025 psi). Testing shall not proceed until any leakage greater than 0.019 psi/min. is reduced to less than or equal to this allowable limit.

1.9 Close valve V-9.

1.10 Turn switch SW-1 to ON (up).

1.11 Fully open vacuum pump line valve V-12.

1.12 When a constant reading is observed on gage G-7, close valve V-12.

1.13 Turn switch SW-1 to OFF (down).

1.14 After one minute, gage G-7 shall not have lost more than 0.019 psi vacuum. Testing shall not proceed until any leakage greater than 0.019 psi/min. is reduced to less than or equal to this allowable limit.

1.15 Open nitrogen cylinder valve V-11.

1.16 Nitrogen cylinder gage G-2 shall indicate not less than 100 psi.

1.17 Set nitrogen gage G-4 to read 10 ± 0.1 psi by adjusting the center screw on the nitrogen high pressure regulator V-2.

1.18 Set gage G-6 to read 2 ± 0.1 psi by adjusting the nitrogen low pressure regulator V-4.

1.19 Open vacuum isolation valve V-7.

1.20 Slowly open nitrogen low pressure valve V-6.

1.21 When the reading on gage G-7 is constant, adjust the pressure to 16.500 ± 0.025 psia with regulator V-4.

1.22 Close valve V-7.

1.23 After one minute, gage G-7 shall not have lost more than 0.019 psi pressure. Testing shall not proceed until any leakage greater than 0.019 psi/min. is reduced to less than or equal to this allowable limit.

1.24 Close valve V-4 so that gage G-6 reads zero.

1.25 Close valve V-6.

1.26 Close valve V-11.

1.27 Open valve V-9.

2.0 TEST

NOTE: UNLESS OTHERWISE STATED,
ALL TEST DATA IS RECORDED
ON FORM 9-8750-2A.

2.1 Record gage G-7 pressure on "Vacuum on case" line of Form 9-8750-2A.

2.2 Prepare test specimen from cured material.

2.2.1 1/4" x 1/4" x 1" wired block (Ref: DWG. No. 20100).

2.2.1.1 Strip 7/8" of insulation from each end of the wire.

2.2.1.2 Put a single clockwise, approximately 5/16" dia. "U" loop in one stripped wire end, and a similar one, but counterclockwise, at the other end.

2.2.2 Connector (Ref: Test No. 5, MSC-D-NA-0002).

2.3 Open the loading port, insert the specimen, and connect each wire to the nut/bolt electrical connection on the center of each Micalex insulator (Ref. DWG. No. 11500).

2.4 Position the block specimen inside the chamber by bending, as required, its wires so that it can be easily observed in the view port via the mirror.

2.5 Remove hand from chamber without disturbing the specimen and close the loading port.

2.6 Close valve V-9.

2.7 Turn switch SW-1 to ON (up).

2.8 Fully open valve V-12.

2.9 When a constant reading is observed on gage G-7, close valve V-12.

2.10 Turn switch SW-1 to OFF (down). Record pressure reading on gage G-7 on Form 9-8750-2A in the box under the word "vac".

2.11 Open valve V-11.

2.12 Gage G-6 should read 10 ± 0.1 psi. Adjust center screw of regulator V-2 until such reading is obtained.

2.13 Open oxygen cylinder valve V-10.

2.14 Oxygen cylinder gage G-1 shall indicate not less than 100 psi.

2.15 Set oxygen gage G-3 to read 5 ± 0.1 psi by adjusting the center screw on the oxygen high pressure regulator V-1.

2.16 Set oxygen gage G-5 to read 2 ± 0.1 psi by adjusting the oxygen low pressure regulator V-3.

2.17 Open valve V-7.

<u>NOTE:</u> OPEN VALVE V-5 SLOW ENOUGH TO PREVENT JERKING MOTION OF GAGE G-7 NEEDLE.

2.18 Fully open oxygen line pressure valve V-5.

2.19 Allow pressure reading on gage G-7 to stabilize.

2.20 Adjust regulator V-3 so that gage G-7 indicates a pressure equal to the sum of 16.500 ± 0.019 psia and the pressure recorded in step 2.10.

2.21 Close valve V-7.

2.22 Reopen valve V-7 if gage G-7 pressure reading decreased more than 0.019 psia/min. until required pressure is obtained.

2.23 Close valve V-7.

2.24 Repeat steps 2.22 and 2.23 until pressure remains stable within the required limits.

2.25 If it is not possible to stabilize the pressure, determine cause of leak, repair as necessary, and repeat entire procedure to this point.

<u>NOTE:</u> VENT AND NITROGEN FLUSH CHAMBER BEFORE CARRYING OUT REPAIRS.

2.26 Turn regulator V-3 counterclockwise until gage G-5 reads zero.

2.27 Close valve V-5.

2.28 Soak specimens five minutes for block specimens and ten minutes for connector specimens.

2.29 Turn power supply switch SW-2 to ON (up) and adjust the current on gage G-8 to 55. ± 2.5 amperes. If ignition or considerable degradation is not obtained in one minute, increase the current by 5 amperes (i.e. from 55. to 60., and 60. to 65., etc.) until such time as the wire fails or ignition occurs. If the wire fails, maintain the voltage to the wires until it is positively established that current does not flow by bridging insulation gaps.

WARNING: DO NOT TOUCH POWER SUPPLY TERMINALS OR LEADS DURING THE REMAINDER OF THE TEST.

2.30 Observe the test and record data specified on Test Form 9-8750-2A.

2.31 When visible specimen degradation no longer occurs, turn switch SW-2 to OFF (down).

2.32 Adjust regulator V-4 to 2 ± 0.1 psi on gage G-6.

2.33 Open valve V-9.

2.34 Open Valve V-7.

2.35 Fully open valve V-6 slowly.

<u>WARNING:</u> TEFLON INSULATION IS USED ON EVERY TEST FOR NAS 9-8750. PURGE THE CHAMBER FOR NOT LESS THAN THREE MINUTES.

2.36 After three minutes close valve V-9.

2.37 Close valve V-7.

2.38 Close valve V-6.

2.39 Adjust regulator V-4 until gage G-6 reads zero.

2.40 Turn switch SW-1 to ON (up).

2.41 Fully open valve V-12.

2.42 When a constant reading is observed on gage G-7, close valve V-12.

2.43 Turn switch SW-1 to OFF (down).

<u>NOTE:</u> OPEN VALVE V-9 SLOW ENOUGH TO PREVENT JERKING MOTION OF GAGE G-7 NEEDLE.

2.44 Fully open valve V-9.

2.45 Open loading port, Observe and record condition of specimen.

2.46 Carefully remove all sample residue from the chamber.

2.47 Close the loading port.

2.48 Close valve V-9.

2.49 Close valves V-10 and V-11.

2.50 Turn off power at main fuse box in laboratory.

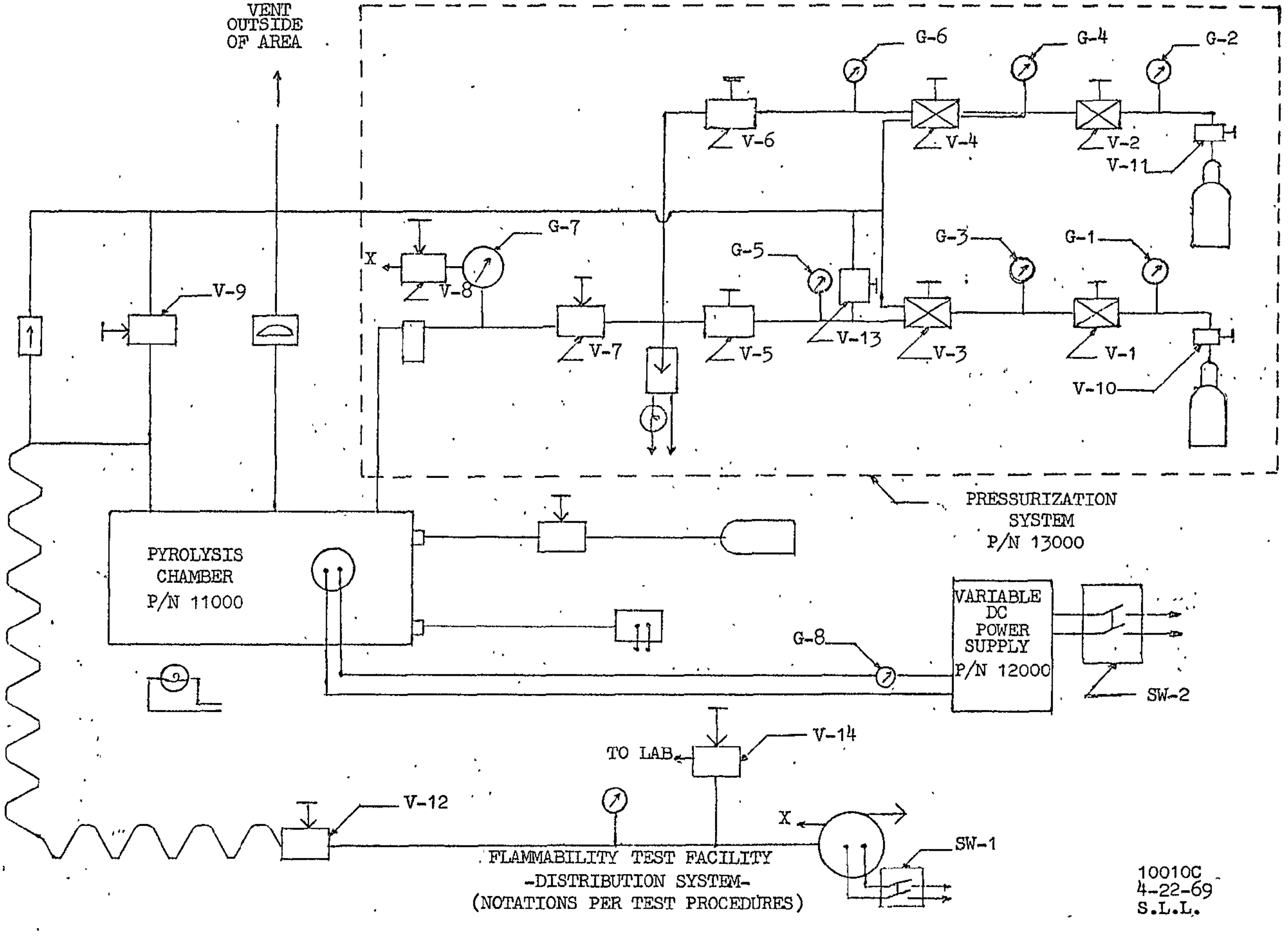
APPENDIX

- M -

FLAMMABILITY
TEST FACILITY
-DISTRIBUTION SYSTEM-
(1)

---

(1) Ref: Appendices I, J, K, and L of this report



FLAMMABILITY TEST FACILITY
-DISTRIBUTION SYSTEM-
(NOTATIONS PER TEST PROCEDURES)

10010C
4-22-69
S.L.L.

APPENDIX

- N -

PRELIMINARY FLAMMABILITY

-TEST CONDITIONS/RESULTS-

PRELIMINARY FLAMMABILITY
-TEST CONDITIONS/RESULTS-

Specimen No.: ____________ Date: ____________ Test Engr: ____________

TEST CONDITIONS

1. Pressure: Gate (W&T) - Vacuum on case: __________ psia

- Atmos: __________ psia (source: ______________)

- Chamber (psia)

Gas	Vac.	test setting	actual

2. Voltage: __________ v. Current: __________ amps,

3. Comments: ______________________________

TEST RESULTS

1. TEST DATA: Pressure (chamber), Peak: __________ psia

Net Increase: __________ psia

Wire Ignition: top bottom

Wire Failure: top bottom

Smoke: none some profuse

Burn Time:	start	flame-out	glow-out	
t =	0	______	______	sec.
Δ =	0	______	______	sec.

2. Comments: ______________________________

____

3. Specimen Post-Test Appearance

3.1 Maintained General Configuration: Yes No

3.2 Residue in Original Configuration: ______ %

3.3 Melted: Yes No

3.4 Color; Exterior: __________

Interior: __________

3.5 Texture; Powdery: Yes No

Crusty: Yes No

4. Comments: ____

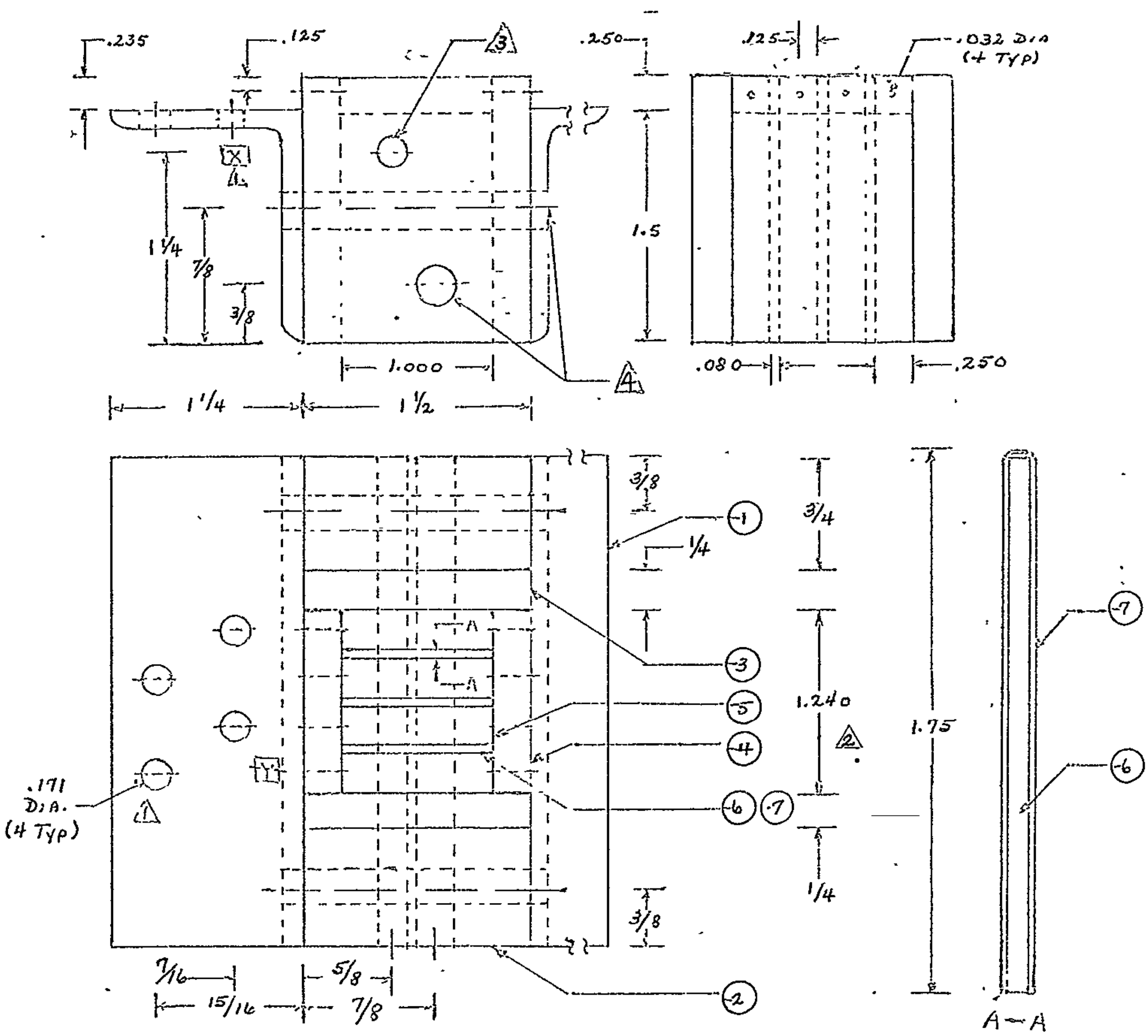
____

APPENDIX

- O -

PRELIMINARY
FLAMMABILITY
TEST SPECIMEN MOLD

FIGURE
PRELIMINARY SAMPLE MOLD



(2 x ACTUAL)

NOTES:

1. Mount 8 Binding Posts with stud facing $\boxed{X}$. Align and center 3/32 hole in binding post with $\boxed{Y}$ and .032 hole in -4.

2. 1.240 approx.; size to fit.

3. Drill for 3/16-NC close fit.

4. Drill for 1/4- NC close fit.

5. Assemble with -9 through -15, as required.

.20100
SLL
1/20/69

TABLE 1

BILL OF MATERIALS:

Part No.	Req/ Assy.	Material	Stock Size
1	2	6061-T6 Angle	1-1/2 x 1-1/4 x 1/8
2	2	Alum.	1-1/2 x 1-3/4 x 3/4
3	2	TFE Teflon	1-1/2 x 1-3/4 x 1/4
4	2	TFE Teflon	1-3/16 x 1-3/4 x 1/4
5	4	TFE Teflon	1 x 1-3/4 x 1/4
6	3	Stainless Steel	1 x 1-1/2 x .055
7	As req.	Tape, TFE, Press. Sens. Adh. 1 side	1" Wide x .012 Thick
8	8	Binding Post	33-286 (G.C. Elect.)
9	1	S.S. or CRES Bolt	1/4-20 NC (Last in.) x 4
10	1	S.S. or CRES Bolt	3/16-20 NC (Last in.) x 4
11	2	S.S. or CRES Bolt	1/4-20 NC (Last 3/4 in.) x 2-1/2
12	6	S.S. or CRES Washer	1/4 x 1/2 x 1/16
13	2	S.S. or CRES Washer	3/16 x 1/2 x 1/16
14	3	S.S. or CRES Nut, Locking	1/4-20 NC
15	1	S.S. or CRES Nut, Locking	3/16-20 NC