N13-25633

RUBBER COMPOSITIONS

FOR

HYDRAZINE SERVICE

PSI PROJECT NO. 7034000 JPL CONTRACT NO. 952864

FINAL REPORT

Prepared by		
John Repar		
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Concurred by	Date	•
Don Kohorst		

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Pressure Systems, Inc. 2017 Camfield Avenue Los Angeles, California 90040

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#### AESTRACT

Forty-three compounds were formulated and tested for hydrazine compatibility at 160°F. Variables introduced included silicon dioxide filler loading level and particle size. Both butyl and ethylene-propylene rubbers were employed as well as various vulcanization systems. Compatibilities averaged approximately 175 days as compared to a range of 159 to 239 days for the controls. The data showed that compounds containing butyl and butyl blended with ethylene propylene could not be distinguished from ethylene propylene alone as far as physical properties were concerned. A trend noted was that a filler level with higher silicon dioxide loading exhibited better hydrazine compatibility. Particle size variation did not show any consistent trends. Any of the vulcanization systems employed appeared to be satisfactory. A refined technique for dissolving aluminum cores from EPT-10 bladders was also perfected.

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#### SUMMARY

Forty-three rubber compounds, consisting of ethylene-propylene, butyl, and blends of both were prepared for hydrazine compatibility testing in this experimental investigation. The intent was to develop compounds suitable for bladders to be used for hydrazine expulsion on spacecraft.

The program was divided into the following five areas of investigation:

(1) variable filler loading effects on the compound EPT-10, (2) variation of filler type and particle size effects, (3) properties of butyl and butyl-ethylene-propylene blends, (4) vulcanization systems, and (5) refinement of procedures for dissolving aluminum cores from hydrazine bladders during manufacture.

Hydrazine compatibility testing of the compounds was conducted at 160°F. Pressure rises to 50 psia, however, required as long as 223 days (175 days average) with control samples ranging from 158 to 239 days.

A slight trend was noted favoring higher silicon dioxide filler loading in ethylene propylene compounds, but no trend was apparent as far as particle size was concerned.

Butyl and butyl blends with ethylene propylene exhibited hydrazine compatibilities indistinguishable from those exhibited by ethylene propylene alone.

Compounds containing butyl rubber had higher surface tackiness than ethylene propylene compounds. This characteristic is desirable for some types of fabrication.

A study of vulcanization systems produced five compounds having properties suitable for diaphragm and bladder applications. These compounds were either butyl or butyl blends with ethylene propylene.

A procedure for the dissolution of aluminum cores from EPT-10 bladder was successfully carried out.

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The program, as originally conceived, was not completed. Only pressure-rise data from hydrazine immersion of the test compounds were obtained. Weight-change determinations were not made, although the samples have been stored to permit this whenever desired.

Future studies should limit hydrazine exposure of test rubber compounds to one month or less, after which physical properties can be determined. It may be expedient to subject only the most promising compounds to this short-duration testing.

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## INTRODUCTION

The effect of hydrazine on rubber materials has been a source of concern for many years. Rubber compounds normally contain some chemicals which either catalyze hydrazine, causing decomposition, or react with hydrazine directly. A major portion of a rubber compound normally consists of the filler, and the concentration of other various ingredients in the material is largely dependent upon its nature.

In an earlier program (Ref. 1), it was shown that employment of rubber materials containing silicone dioxide or calcium silicate fillers reduced hydrazine decomposition rates drastically when compared to compounds containing carbon black fillers. The initial effort in the present program was directed toward establishing the compatibility of fillers with hydrazine.

The compatibility and weight-change data were expected to give some indication of the mechanism of hydrazine absorption. The structure of hydrazine is similar to that of water. As the filler Silene D is hygroscopic, it was suspected that this filler has a greater affinity for hydrazine than for water, because hydrazine is anhydrous and also very hygroscopic.

In a continued effort to discover a trend of hydrazine absorption by rubber materials, an attempt was made to correlate the compatibility of rubber compounds with hydrazine with the particle size of the fillers used in these compounds. Implicit in this approach is a relationship between compatibility and compound surface area.

In the past, butyl rubber compounds have been used with hydrazine. Butyl bladders were used for hydrazine expulsion in zero-gravity environment on all of the Ranger spacecraft as well as on the Mariner 4 and 7 spacecraft. Butyls have the added advantage of being characteristically tacky, thus facilitating lay-up fabrication. The main difficulty with butyl rubbers, however, is achieving suitable vulcanization in the presence of fillers other than carbon black. Studies of butyls and vulcanization systems are therefore closely linked.

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In the cited earlier program (Ref. 1), the compound that was found to exhibit the best overall balance of properties for use in bladder fabrication was EPT-10. This compound however has characteristics considered less than optimum, including a relatively high absorption of hydrazine and a lack of tackiness which made lay-up construction difficult. EPT-10 is molded into bladders on an aluminum core. The common procedure for extracting this aluminum core from the bladder has not been completely satisfactory. Principally; EPT-10 was found to be sensitive to the caustic solution used in core dissolution. Development of a coating that would protect the rubber and the perfection of a technique for applying this coating over the exterior and interior surfaces of a bladder while still on the aluminum core were part of the present program.

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## TECHNICAL DISCUSSION

### A. GENERAL

This program was divided into several areas of investigation:

- (1) Effects of variable filler loading on EPT-10
- (2) Effects of variation of filler type and particle size
- (3) Butyl and butyl-ethylene-propylene blends
- (4) Vulcanization systems
- (5) Refinement of aluminum core dissolution procedures.

Rubber compounds were numbered beginning with compound 10. The numbering, system was derived from the earlier program (Ref. 1). The numbering is consecutive except for compound 10A which was basically the same as compound 10 except for the cure system. Compounds 26 through 33 (except 29) were mill mixed for radiation studies which will be evaluated under another program and therefore have not been included in this report.

The compounds were immersed in hydrazine at a temperature of 160°F — a test temperature common in numerous specifications. It was hoped that this temperature would expedite the program by decreasing the time of rise to 50 psia. Many of the samples, however, exceeded 200 days of test duration.

Evaluation of the compounds was limited to pressure rise data during hydrazine immersion. As this is only a partial evaluation of the compounds, samples are presently being kept in storage so that they can be evaluated as to weight gain and hydrazine contamination should such a follow-on procedure be desired.

The pressure-rise data were plotted, and all curves were found to be linear after the first three or four days of immersion and until termination at 50 psia.

Variability of data was reduced considerably by preconditioning the samples as well as the test capsules in hydrazine for two days at ambient conditions. Nonetheless, there still remained a rather wide dispersion which made conclusions somewhat difficult to derive. There is some justification, however, for considering data obtained from longer exposures of samples to hydrazine as having greater validity because leaky capsules were readily

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detectable. Short test durations are likely the result of contaminated capsules and/or test samples. It is interesting to note that the test duration of control samples varied from a low of 158 days to a high of 239 days. In this instance; data were very much better than expected. Samples from compound AFE-332-11, which was developed for hydrazine service by TRW also fared well as far as test duration was concerned, with two samples reaching 182 days and a third 156 days.

The mill processing characteristics of most of the compounds which were prepared under the program were satisfactory. As expected, the addition of butyl rubber to ethylene propylene invariably improved the handling characteristics.

All but one of the compounds were extruded through a 100-mesh screen. An attempt was made to extrude compound 51, but the virgin teflon clogged the screen. A companion compound, number 52, which contained ground, fused teflon, passed through the screen without difficulty. This process was an attempt to improve the tear strength which was accomplished with the virgin teflon (compound 51) having a tear strength of 265 lbs/in. as compared with 195 lbs/in. for compound 52. It appears that the virgin teflon reinforces the compound by simulating a fibrous structure. This can be seen visually in tensile strength and tear samples as they are stressed.

In addition, the compounds were X-rayed, and the test swatches were molded from areas which were free of particles approximately 0.005 in. or larger in cross section.

The core dissolution procedure for bladders from an earlier program was not completely satisfactory. The caustic solution which was employed in dissolving the aluminum core caused a degradation of the EPT-10 bladder material. The main effort in this investigation was directed toward the application of a protective coating over the rubber.

# B. EFFECTS OF VARIABLE FILLER LOADING ON EPT-10

Compounds 10 through 14, as shown in Table 1, show considerable overlapping of data with respect to pressure rise, and a clear conclusion concerning filler loading is not obvious. There is some justification, however, for placing a greater degree of confidence in the higher figure of the two samples, because

it is inferred from experience that the presence of contamination will tend to mask the actual behavior of the test sample. Leakage also can be readily detected as pressure either stabilizes or decreases under this condition.

If the longer times are selected as representative of compound behavior, a general trend becomes apparent (compound 13 excepted) which appears to indicate that the dilution of the rubber with silicone dioxide filler tends to reduce the hydrazine decomposition rate.

Physical properties of the compounds indicate that compounds 10, 11 and 12 are entirely suitable for diaphragm and bladder applications, whereas compounds 13 and 14 have tensile strengths which are relatively low.

Table 1. Filler Level, Silene D

		Compound						
Characte	ristic	10	11	12	13	14		
Parts per Hundre by Weight	ed of Rubber,	65	50	35	20	0		
Days to 50 psia	Sample 1	210	172	160	182	141		
in N <sub>2</sub> H <sub>4</sub> at 160°F	Sample 2	125	164	139	170	102		
Tensile Strength	psi	2030	1685	1400	810	280		
Elongation, %		400	460	440	390	330		
Hardness, Shore	A	.78	73	68	60	53		

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# C. EFFECTS OF VARIATION OF FILLER TYPE AND PARTICLE SIZE

Early in this phase of the program, the amount of silicon dioxide added to compounds as a filler had to be reduced, as the particle size of this filler decreased until a level of 35 parts per hundred by weight of rubber was reached. This permitted a comparison of data from all of the silicon dioxide fillers.

The investigation was concentrated on obtaining data from silicon dioxide as there are very few non-carbon-black reinforcing fillers. The data shown in Table 2, however, indicate that the various particle sizes of silicon dioxide have no definite correlation with hydrazine compatibility. The range of 130 to: 220 days of exposure appears to be the result of data scatter rather than tending to establish a trend.

Some other fillers employed in the investigation were Silene E F and Ice Cap KE clay, which are calcium silicate fillers. Compound 41 contained Ice Cap KE clay and had a hydrazine compatibility (196 days to 50 psia) which was within the time span of many of the compounds that contained silicon dioxide. This compound, as well as compound 23, with 210 days to 50 psia, indicates that calcium silicate is suitable as a reinforcing filler for hydrazine applications.

Compounds 51 and 52 each contained teflon powders. Compound 51 contained virgin teflon, while compound 52 was filled with the fused and ground material. The tear strength of compound 51 was 265 lbs/in. and of compound 52, 195 lbs/in. In order to increase tear strength, it is necessary to employ virgin teflon in filler material. No difficulties were encountered in mixing either compound, but compound 51 could not be extruded through the 100-mesh screen. Neither of these two compounds was subjected to compatibility tests; however, the two compounds should behave somewhat like compound 12.

All of the compounds processed more easily than EPT-10 during mixing, extruding, and molding (except for cited difficulties with compound 51 extrusion). The physical properties of the compounds discussed, however, were all within the range of workable expulsion devices.

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Table 2. Filler Study, Types of Silicon Dioxide Particles

	Compound*									
Characte ristic		12	18	19	20	21	22	23	24	25
Filler		Silene D	1	O-Sil	Cab- O-Sil H-5	<b>(</b>	Cab- O-Sil M7	Silene EF	Arcsilica 800	Hi-Sil 233
Particle Size, µ		0.03	0.005	0.012	0.007	0.05	0.012	0.03	0.005	0.022
Days to 50 psia	Sample 1	160	130	155	209	182	172	210	220	215
in Hydrazine at 160°F	Sample 2	139			186	145	137	145	181	154
Tensile Strength,	psi	1400	2785	2590	2455	2425	3180	1390	1390	2735
Elongation, %		440	480	370	320	480	390	350	280	400
Hardness, Shore	A	68	82	81	83	70	84	73	73	79

\*Filler Level - 35 Parts per Hundred of Rubber by Weight

## D. BUTYL AND BUTYL-ETHYLENE-PROPYLENE BLENDS

Often an approach is neglected because an apparent solution receives more attention. The ethylene propylene compounds have overshadowed the butyls for this reason during recent years. Yet butyl rubbers have several favorable characteristics. One of the most important is the resistance to crack growth during flexing, another is the low permeability to gases which should also be duplicated by a low permeability with hydrazine, and a third is the characteristic tackiness which enhances the prospects of lay-up-type fabrication.

In this program the main problems have centered around the physical properties of the butyls and butyl blends. This stems from the difficulty of obtaining satisfactory vulcanization in the presence of silicone dioxide fillers. Peroxides cannot be employed, as they will depolymerize the butyl polymers. Generally the sulfur or resin types offer the best possibilities. Both are also useable with ethylene-propylene terpolymers.

The data in Table 3 indicate that the hydrazine compatibility of butyl compounds and butyl-ethylene-propylene blends is comparable to that of ethylene propylene compounds. Therefore, it is necessary to look elsewhere for differences between these types of compounds. One of these areas is the processing characteristics. Butyl and butyl blends have better processing characteristics than ethylene propylene alone. The increase in surface tackiness would enhance the possibility of hand lay-up and vacuum-bagging type of construction. Elongations in the range of 500% and higher permit the removal of relatively large cores from butyl and butyl blend bladders by stretching the mouth openings sufficiently.

Table 3 summarizes the properties of several butyl compounds that show promise with hydrazine. The butyl bladders which were employed in Mariners 4, 6, and 7 were made from fargo rubber compound FR-6-60-26 which had a Shore-A hardness of 55, an elongation of 550%, and a tensile strength of 1200 psi. Two of the butyl compounds tested, namely 36 and 37, have physical properties similar to those of fargo rubber compound.

The test samples are in storage; weight-change determinations are not available at this time.

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Table 3. Butyl Compounds

T4		Compound*					
Item	35	36	37	47	48		
	Rubbe	r					
Butyl HT-1066	100		50	50			
Butyl 365		50			50		
Royalene 301		50	50				
Nordel 1320			·	50			
Vistalon 2504					50		
V	ulcanizing	Agent					
Sulfur	1.5	1.5	1.5				
Thionex	1.5	1.5	1.5				
MBT	0.5	0.5	0.5				
Sulfasan R				1.5	1.5		
Ethyl Zimate				1.5			
Ledate					2.0		
	Character	ristic					
Days to 50 psia Sample 1	198	182	203	223	178		
in Hydrazine Sample 2	190	157	148	170	166		
Tensile Strength, psi	1915	1080	1245	1015	1335		
Elongation, %	990	450	520	800	900		
_ 8	1		53	43	1		

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### E. VULCANIZATION SYSTEMS

The study of vulcanization systems was directed toward several objectives. The elimination of sulfur was accomplished in compounds 10A (similar to EPT-10) and 41 with the utilization of a dicumyl peroxide cure. Both compounds had elongations which were below 200% and would therefore be somewhat marginal for expulsion bladders or diaphragms. As peroxide cores are not suitable for butyl-type elastomers, it was necessary to investigate other possibilities. Therefore compounds 42 through 50 served a double purpose by combining the study of butyls as well as of vulcanization systems.

Table 4 contains selected data showing the best properties of each basic vulcanization system. For compounds 42 through 45 a resin, ST 1055, was employed. The ultimate tensile strength was low, ranging from 595 to 775 psi. Compounds 47 and 48 employ Sulfasan R as the main agent with ethyl zimate and ledate, respectively, acting as activators. Both compounds show promise in hydrazine applications. With respect to compatibility, any of the vulcanization systems would appear to be satisfactory. Compound 41, which was cured with a peroxide, showed a duration of 196 days in the hydrazine test. This counterbalances the relatively poor results obtained with compound 10A.

### F. REFINEMENT OF ALUMINUM CORE DISSOLUTION PROCEDURES

The process for extracting the aluminum core from a bladder in an earlier program was not completely satisfactory as there was an indication that compound EPT-10 was sensitive to caustic solutions. The data in Table 5 show why EPT-10 required isolation from caustic; a decrease in both the tensile strength and hardness are caused by EPT-10 in contact with caustic. It is also apparent from Table 5 that the oven post-cure phase of vulcanization for this compound should be performed after the caustic soak. As two EPT-10 bladders were still on cores, the problem was to arrive at a procedure for extracting the cores.

Initial efforts were performed on test swatches which were coated with several materials, namely, ethyl celulose, polyvinyl chloride plasticsol, and polyvinyl chloride dissolved in a mixture of ketones. Ethyl cellulose was heated to 350 °F. Test samples were dipped and allowed to cool. The coating thickness could be controlled at will. The polyvinyl chloride plastisol was likewise dipped

Table 4. Basic Vulcanization System Properties

T.		• • •	Com	pound*		
Item	10A	22	45	46	47	48
Vul	canizatio	n System				
Dicup 40 c	7					
Vinyl Silante A-172	1		·			
Triallyl Cyanurate	1.5					
Sulfur	·	1.5				
Thionex		1.5				•
MBT		0.5		0.5	·	
ST1055			12			·
TMTDS				1.0		
TDEDC				1.0		-
Sulfasan R				1.0	1.5	1.5
Ethyl Zimate					1.5	
Ledate				·		2.0
	Characte	eristic				·
Days to 50 psia ) Sample 1	137	172	190	180	223	178
in Hydrazine Sample 2	127	137	164	147	170	166
Tensile Strength, psi	1645	3180	775	720	1015	1335
Elongation, %	170	390	620	780	800	900
Hardness, Shore A	73	84	47	50	43	47
Hardness, Shore A  *Compounds are based on par	<u> </u>				<u>t.</u>	

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but had to be heated to 350°F in order to cause it to solidify. The third prospective coating, polyvinyl chloride dissolved in ketones, was dipped and then allowed to dry at room temperature. The first two of the enumerated coatings were not difficult to apply, but they would have been difficult to employ in the final process, where the interior of the bladder had to be coated with the plastisol. The polyvinyl chloride solution offered the most promise.

A small mold was constructed so that bladders could be molded on aluminum cores. The mold produced bladders which were approximately 5 in in diameter. The cores were made from hydroformed aluminum hemispheres which were welded at the girth.

Table 5. Effects of Caustic Solution on Compound 10

	Physical Property					
Sample Description	Tensile Strength, psi	Elongation, %	Hardness, Shore A			
Control (not immersed)	2180	310	77			
Postcured, Not Coated, Immersed	1390	340	62			
Postcured, Coated with Ethyl Cellulose	1500	300	: 70			
Not postcured, * Coated with Ethyl Cellulose	2025	280	74			
Not postcured, * Coated with Polyvinyl Chloride Film	2300	300	77			

<sup>\*</sup>Postcured 4 hr at 350°F after completion of caustic soak; data obtained after final postcure.

<sup>12.5%</sup> sodium hydroxide solution by weight for one week at ambient temperature.

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As had been encountered in the previous program, a release coating was required with EPT-10. Teflon and FEP coatings were tried. Both required high temperatures (700°F+) during application which could be harmful to the properties of aluminum components. No deformation was noted, however. The teflon coating peeled with the rubber after several molding attempts. Therefore it was replaced with an FEP coating which performed satisfactorily. The coating was applied employing the following procedure:

- (1) Clean and grit blast surface to be coated, then bake at 705°F.
- (2) Spray with Dupont 850-204 and fuse at 700 °F.
- (3) Spray with Dupont 856-204 and fuse at 610 °F.
- (4) Spray with Dupont 856-200 and fuse at 610°F.

Following the molding of suitable bladders, coating of the rubber was undertaken. The exterior caused no difficulty, but the interior required the insertion of stainless steel wires between the core and the bladder. The polyvinyl chloride solution would run to the bottom of the bladder along the channels paralleling the wires. The wires could then be moved about so that the entire inner surface was covered. Pinholes were encountered in the polyvinyl chloride coatings. Additional coats or entirely new coatings had to be applied to the exterior as pinholes would permit the caustic to come in contact with the rubber and cause localized discoloration.

Several cores were dissolved. The following procedure was developed for core dissolution:

- (1) Prepare a 2.5% sodium hydroxide solution
  Water 70.00 lbs
  50% caustic solution 3.60 lbs
- (2) Add to dissolving tank
- (3) Insert pump outlet into aluminum core
- (4) Start nitrogen gas bubbling (about one bubble per second)
- (5) Start circulation pump
- (6) Replace solution every 2-1/2 to 3 days.

The nitrogen gas bubbling was included in order to minimize formation of sodium carbonate.

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# NEW TECHNOLOGY

No reportable items of new technology have been identified.

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#### CONCLUSIONS

The program, as originally conceived, was not completed. Data from hydrazine immersion consisted of pressure rise only. Consequently, weight-change determinations will not be available until some future date, and the optimization of compounds could not be undertaken during the course of this program.

Although there is still a large spread in the data, this was considerably reduced by keeping samples and test apparatus for 48 hours at room temperature. The hydrazine compatibility test was conducted at 160 °F with the expectation that the time to reach 50 psia, when the tests were to be terminated, would be rather short. While control samples ranged from 159 to 239 days, however, test samples averaged approximately 175 days and went as long as 223 days.

The data have indicated a slight trend that implies that higher ratios of silicon dioxide promote compatibility with hydrazine. There is no evident trend as far as particle size is concerned.

Calcium silicate fillers can be employed successfully, but lower the physical properties of the rubber.

Butyl compounds and butyl ethylene-propylene blends compare favorably with ethylene-propylene alone, with respect to hydrazine compatibility. In addition, butyl enhances the property of surface tackiness of the raw compound. This characteristic is desirable where layups are required during the fabrication process.

The variations in vulcanization included peroxide, sulfur, sulfur substitution, and resin. Compounds which were suitably vulcanized so that they could be employed in diaphragms and bladders included peroxide, sulfur, and sulfur substitute formulations. None of the resin-cured compounds had adequate tensile strength properties (1000 psi or more).

The core dissolution procedure for bladder fabrication was refined so that EPT-10 could be used in the production of bladders. The basic element found necessary for successful core removal was the addition of a protective coating over both the interior and exterior surfaces of the bladder. The coating is a polyvinyl chloride solution in a mixture of ketones.

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#### RECOMMENDATIONS

Samples remain from this program that still require evaluation with respect to weight change. The sample capsules contain the gaseous decomposition products as well as residues which are dissolved in the hydrazine so that no loss of material can occur.

As the compatibility tests ran for such an unexpected long duration, future studies should include a limited exposure, not to exceed one month, after which properties such as weight, ultimate tensile strengths, elongation changes, and other data can be obtained. It may be expedient to subject only the most promising compounds to short-duration testing.

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### APPENDIX

## TEST DATA

Test results are presented in tabular form in this appendix to substantiate the findings elaborated under "Technical Discussion" and "Conclusions."

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Table A-1. Compatibility with Hydrazine, at 160°F, in psia

24	23	22	21	20	19	18	17	16	15	14	13	12	11	10A	10	P	Compound
20 17	23 20	18 20	17 20	16 17	20	22	21	19 38	17	23 30	20 16	22 22	20	28	15 22	50	
31 27	38 29	31 38	28 36	27 29	34	40	36	29	28	37 49	32 27	3 5 7	33 1	42	26 40	100	
42 38	38	45	42	38 40	48		48	41	40		45	49	46 44		37	150	Days
48	48			49		-			•						48	200	78
181 220	145 210	172 137	182 145	209 186	155	130	<u>Б</u>	190 95	184	141 102	179 182	160 139	164 172	127 137	210 125	To 50 psia	

Compatibility with Hydrazine, at 160°F, in psia (Cont'd) Table A-1.

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	To 50 psia	215	187	190	157	203	198	155	142	196	173	220 176	224	190 164	147	170
S	200	47	-			49						48	45			45
Days	150	36.	43	38	4 4 8 2 2	37	40	4 4 43		39 38	44 43	36 43	34	441	41	33
	100	25 36	30	28 27	31	26 35	31 28	33	33	26.	29	25 28	24	30	37	28
	50	16 21	18	18	19	16 20	18	18	19	15	16	16 15	14	18	23	16 13
Compound		25	<b>3</b> 4	 	36	37	38	. 36	40	41	42	43	44	45	46	47

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Table A-1. Compatibility with Hydrazine, at 160 °F, in psia (Cont'd)

Compound			Day	7S	
Compound	50	100	150	200	To 50 psia
48 AFE 332-11	17 16 17 20 18	31 29 30 35 30	46 42 41 49 42		166 178 182 156 182
Control	14 14 12 20 18	26 23 21 33 33	38 32 30 44 48	50 42 41	200 236 239 172

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Table A-2. Effect of Silene D Loading l

Item		С	ompound*		
Item	10	11	12	13	14
	Var	riable			
Silene-D	65	50	35	20	0
	Con	stant			·
Royalene 301	100	100	100	100	100
Zinc Oxide	5	5	5	5	5
Sulphur	1.5	1.5	1.5	1.5	1.5
Thionex	1.5	1.5	1.5	1.5	1.5
MST	0.5	0.5	0.5	0.5	0.5
	Physica	l Property	7		
Tensile Strength, psi	2030	1685	1400	810	280
Elongation, %	400	460	440	390	330
Hardness, Shore A	78	73	68	60	53
Sp. Gr.	1.08	1.05	1.02	1.0	1.0
*Parts per hundred of r	ubber by w	eight.			

 $<sup>^{1}\</sup>mathrm{Press}$  cured 1 hr at 320 °F and oven postcured 4 hr at 350 °F.

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Table A-3. Composition of Compounds with Hystl\*

Ingredient	Com	pound
ingredient	49	50
Nordel 1440	. <u>-</u> -	75
Nordel 1320	25	
Butyl HT-1066	75	25
Hystl B-200	10	10
Silene D	35	35
Zinc Oxide	5	5
Sulfur	1.5	
Thionex	1.5	
MBT	0.5	
ST 1055		. 15

<sup>\*</sup>Parts per hundred of rubber by weight; neither compound could be vulcanized satisfactorily.

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Table A-4. Filler Study\*

Thomas	Compound										
Item	15	16	17	18	19	20	21.	22	23	24	25
				Rubbe	er						
Royalene 301	100	100	100	100	100	100	100	100	100	100	100
				Fille	r						
Silene EF Arc Silica 800 Hi Sil 233	50	50	50					•	35	35	35
Cab-O-Sil EH5 Cab-O-Sil M-5 Cab-O-Sil H-5 Cab-O-Sil L-5				35	35	35	35	35			
Cab-O-Sil M-7			371	canizing	. Sugta	22		33			
			vui	Camzing	g byster	. 1 1			·		
Zinc Oxide Sulfur MBT Thionex	5 1.5 1.5 0.5	1.5 1.5 0.5									
Physical Property											
Tensile Strength, psi Elongation, % Hardness, Shore A Specific Gravity	1485 380 77 1.08	2485 500 78 1.10	2975 440 88 1.10	2785 480 82 1.05	2590 370 81 1.05	2455 320 83 1.05	2425 480 70 1.05	3180 390 84 1.05	1390 350 73 1.05	1390 280 73 1.05	2735 400 79 1.05

<sup>\*</sup>Press cured, 1 hr at 320 °F, and oven cured, 4 hr at 350 °F.

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Table A-5. Compounds with Fluorocarbon Filler\*

Item		Compound	
	51	52	12
	Rubber	· · ·	
Royalene 301	100	100	100
	Filler		
Silene D	35	35	35
Zinc Oxide	5	5	5
Teflon, TFE T-58	10	<del></del>	
Teflon, TL-126		10	
Sulphur	1.5	1.5	1.5
Thionex	1.5	1.5	1.5
MBT	0.5	0.5	0.5
Physic	al Properties		
Tensile Strength, psi	1330	1350	1330
Elongation, %	280	390	440
Hardness, Shore A	77	65	68.
Tear Strength, lbs/inch	265	195	

<sup>\*</sup>Press cured 1 hr at 32°F and oven postcured 4 hr at 350°F.

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Table A-6. Butyl Compounds and Blends\*

Ingredient	Compound						
ingredient	34	35	36	37	38	39	40
Butyl 365	100		50	·	50		gr t
Butyl HT-1066		100		50		50	100
Royalene 301	-	:	50	50			
Nordel 1440	** ****:		er e e		50	50	14
Silene D	35	35	35	35	35	35	Ĩ.
Zinc Oxide	5	5	5	5	5	5	5
Sulphur	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Thionex	1.5	1.5	1.5	1.5	1.5	1.5	1.5
MBT	0.5	0.5	0.5	0.5	0.5	0.5	0.5

<sup>\*</sup>Parts per hundred of rubber by weight.

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Table A-7. Vulcanization Systems, A\*

Ingredient			Compound		
ingredient	42	43	44	45	46
Butyl 365	100		50		50
Butyl HT-1066	·	100		50	
Royalene 301			50	50	
Nordel 1440					50
Vistalon 2504					
Silene D	35	35	35	35	35
Zinc Oxide	5	5	5	5	5
ST 1055	12	12	12	12	
MBT					0.5
TMTDS					1.0
TDEDC					1.0
Sulfasan R					1.0

<sup>\*</sup>Parts per hundred of rubber by weight.

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Table A-8. Vulcanization System, B\*

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	Compound				
Ingredient	10A	41	47	48	
	Rubber				
Royalene 301	100	100		· ·	
Butyl 365		<u></u>		50	
Butyl HT-1066		·	50	-	
Nordel 1320			50	<b></b>	
Vistalon 2504	<b></b> .	<b></b>	<b></b>	50	
	Filler				
Silene D	65	<b></b>	3.5	35	
Ice Cap KE Clay		100	, <del></del>		
Dicup 40C	7	7			
Vinyl Silene A-172	1	1			
Tri Allyl Cyanurate	1.5	1.5			
Vulcanizing System					
Zinc Oxide		also total	5	5	
Sulfasan R			1.5	1.5	
Ethylzimate			1.5		
Ledate				2.0	

<sup>\*</sup>Parts per hundred of rubber by weight.

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Compound 8  $\omega$ 10A Press Cure, 320°F hr Ġ 0.75 0.75 0.75 0.75 0.75 1.5 Oven Cure, 350°F hr Table A-9. Physical Properties Tensile Strength, 100% Elongation, %Modulus, 100% .60 <u>ភ</u> Hardness, Shore A  $\sigma_{\omega}$ 

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Table A-10. List of Ingredients

Ingredient	Description	Source
Polymers		
Butyl 365	Isobutylene-Isoprene	Enjay
Butyl HT-1066	Chlorinated Isobutylene- Isoprene	Enjay
Nordel 1320	Ethylene Propylene Terpolymer	Dupont
Nordel 1440	Ethylene Propylene Terpolymer	Dupont
Royalene 301	Ethylene Propylene Terpolymer	Uniroyal
Vistalon 2504	Ethylene Propylene Terpolymer	Enjay
Fillers		
Arc Silica 800	Silicone Dioxide	Pittsburgh Plate & Glass
Cab-O-Sil EH5	Silicone Dioxide	Cabot
Cab-O-Sil H-5	Silicone Dioxide	Cabot
Cab-O-Sil L-5	Silicone Dioxide	Cabot
Cab-O-Sil M-5	Silicone Dioxide	Cabot
Cab-O-Sil M-7	Silicone Dioxide	Cabot
Hi Sil 233	Silicone Dioxide	Pittsburgh Plate & Glass
Ice Kap KE Clay	Calcium Silicate	Burgess
Silene D	Calcium Silicate	Pittsburgh Plate & Glass
Silene EF	Calcium Silicate	Pittsburgh Plate & Glass
Teflon, TFE T-58	Tetrafluro Ethylene	Dupont
Activators and Vulcanizing Agents		. *
Dicup 40 C	Dicumyl Peroxide	Hercules
Ethyl Zimate	Zinc Dirthyl-Dithiocarbamate	R. T. Vanderbilt
Ledate	Lead Dimethyl- Dithiocarbamate	R. T. Vanderbilt
MBT	Mercapto Benzothiazole	R.T. Vanderbilt
TDEDC	Tellurium Dirthyl- Dithiocarbamate	R. T. Vanderbilt
Thionex	Tetramethyl Thiuram Monosulfide	Dupont

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Table A-10. List of Ingredients (Cont'd)

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Ingredient	Description	Source
Activators and Vulcanizing Agents (Cont'd)		
TMTDS	Tetramethyl Thiuram Disulfide	
Tri Allyl Cyanurate		American Cyanamid
ST-1055	Polyhalomethyl Phenol Resin	Schenectady 's
Sulfasan R	4,4 Dithiodimorpholine	Monsanto ,
Sulfur		L.A. Chemical
Vinyl Silane A-172	. <del></del>	Union Carbide
Zinc Oxide	<b></b>	New Jersey Zinc

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