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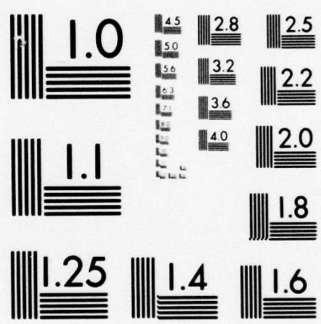

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FABRICATION OF A LOW TEMPERATURE FUEL HOSE FROM PHOSPHONITRILIC FLUOROELASTOMER

NOVEMBER, 1976

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FINAL REPORT - CONTRACT DAAG53-75-C-0187,
P00002

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FABRICATION OF A LOW TEMPERATURE FUEL HOSE FROM PHOSPHONITRILIC FLUOROELASTOMER

November, 1976

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The Firestone Tire & Rubber Company
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SUMMARY

Refueling operations in the Arctic region must be conducted at temperatures as low as -70°F. A critical need exists for fuel resistant elastomers that could be utilized for fuel hoses at such temperatures. This work was directed toward providing such a hose.

The elastomer chosen for this study was a modified phosphonitrilic fluoroelastomer (PNF®). PNF® utilized in an earlier contract (No. DAAK02-73-C-0464) produced hose capable of use at about -45°F. This program was an attempt to lower that use temperature down to -70°F.

The initial phase of this investigation was a compounding study to develop processible compounds which could be fabricated into collapsible and suction type hoses. It was shown that several formulations of the modified PNF® provided adequate processibility. However, the good low temperature serviceability constraint severely limited the number of reinforcing agents. Only relatively large particle size blacks such as FEF would provide both good processibility and low temperature flexibility.

Using an FEF black compound, it was demonstrated that large lengths of both collapsible and suction hoses could be manufactured. These hoses showed good flexibility at -70°F. Furthermore, the hoses possessed very good dimensional stability and physical strength. Rather low tensile and tear strengths were the major deficiencies of these hoses. Low adhesions of tube and cover to inner plies were caused primarily by the low tear strength.

All trial hoses showed good fuel resistance. The final, large lengths of hose showed adequate volume swells but high levels of residue from the

existent gum test. However, it appears that considerable amounts of fuel components were present in the residue along with some low molecular weight PNF®.

It can be concluded that the modified PNF® can be utilized to produce Arctic fuel hose with utility at -70°F. Future studies should be directed toward improving tensile and tear strengths and eliminating any low molecular weight material in the polymer.

PREFACE

This report describes all work performed under Contract No. DAAG53-75-C-0187 and a modification to this contract (P00002). The original contract was for an eight month period from June 30, 1975, to February 28, 1976; the modification was for a 70 day period starting on June 28, 1976. The driving force for this work was development of fuel hose capable of service in Arctic environment (-70°F to +95°F).

This final report was prepared by the Central Research Laboratories of The Firestone Tire & Rubber Company. The work was sponsored and administered by the U. S. Army Mobility Equipment Research and Development Center, Ft. Belvoir, Virginia. Mr. Philip Mitton served as the Contracting Officer's Technical Representative.

Project management was under the direction of Dr. D. P. Tate, Assistant Director of Research, and Dr. J. A. Beckman, Manager of the Elastomer Synthesis Division at Firestone. Special recognition is also due Mr. Robert A. Lord, Mr. Bill Goodwin and Mr. Al Turowsky for directing and handling the hose fabrication work performed at Boston Industrial Products Division of American Biltrite, Inc. We also acknowledge the assistance of many co-workers at the Firestone Central Research Laboratories, especially Mr. J. F. Witner, Dr. G. S. Kyker, Mr. R. J. Sando and Mr. E. K. Sanders, for assistance in the compounding and testing phases of this research..

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INTRODUCTION

The goal of this work was to utilize phosphonitrilic fluoroelastomer (PNF®) to produce fuel resistant hose which would be serviceable in extreme cold environments (-70°F). Such hose would satisfy the Army's requirements for refueling operations in the Arctic region.

The U. S. Army Mobility Equipment Research and Development Center sponsored earlier work in this area (Contract No. DAAK02-73-C-0464; 6/73-12/73). In these prior studies at the Firestone Central Research Laboratories, it was shown that fuel hoses could be fabricated from phosphonitrilic fluoroelastomers. However, a major deficiency of the hose was poor low temperature flexibility (relative to the desired goals). A Gehman T_5 value of -42°F, a torsional stiffness ratio at -70°F of 20 and a cold tension recovery at -70°F of 10% were obtained on stock used in the hose building.

Since the above described work in 1973, a modified phosphonitrilic fluoroelastomer (PNF®) exhibiting improved low temperature flexibility was developed at the Firestone Central Research Laboratories. The present contract utilizes this modified PNF® and is a development study directed toward fabrication of Arctic fuel hose.

The program followed in the present study can be divided into three major parts:

1. Development of processible compounds.
2. Development of all techniques for satisfactory manufacture of hoses.
3. Production of fuel hose.

In the compounding or first phase of the program, the major objectives were:

1. To optimize low temperature (-70°F) serviceability while maintaining fuel and water resistance.
2. To obtain sufficiently high "green" and cured strength for hose manufacture.
3. To attain good mill and calender release.
4. To maintain good rubber to textile adhesion.

Suitable compounds developed in the compounding studies were then utilized in the second phase of the program--prototype hose design. Sufficient stock was mixed to permit fabrication of short lengths of 2" ID fuel hose. Three of these hose building trials were made.

Finally, the best compound was utilized to produce large lengths of both the suction and collapsible 2" fuel hoses. The hose sections produced were then tested for comparison to the desired contract requirements.

INVESTIGATION

1. Polymer

The polymers utilized in these investigations are modified phosphonitrilic fluoroelastomers (PNF®). The improved low temperature flexibility of the modified PNF® was realized by reduction of fluorine content in PNF®. This reduction in fluorine content causes an increase in volume swell in hydrocarbon fuels. Thus, the level of fluorine is critical for maintaining a proper balance of low temperature flexibility and fuel resistance.

For the bulk of the work in this contract, a modified PNF® with a good balance of low temperature flexibility and fuel resistance was utilized (K18161). However, this high DSV polymer could not be processed on a rubber mill. Heat aging at 300°F reduced the "nerve" of this polymer and resulted in adequate processibility.

Additional polymer had to be synthesized for the production phase of the contract. The polymer produced was too low in fluorine content and resulted in unsatisfactory fuel resistance. However, earlier independent studies at the Firestone Central Research Laboratories indicated that good control of the low temperature-fuel resistance balance could be attained through utilization of blends of modified and unmodified phosphonitrilic fluoroelastomer (PNF®-200). This was an important finding since precise control of the fluorine content in these syntheses has not yet been worked out. Thus, after evaluating several blends, it was decided to utilize a 60:40 blend of modified PNF® to PNF®-200. It was also shown that heat treatment of this blend was unnecessary.

These modified phosphonitrilic fluoroelastomers are characterized by lower Tg's (ca. -79°C) than the PNF®-200 (ca. -67°C).

2. General Approach

Since the modified PNF®'s are inherently good low temperature polymers, the basic objective was to compound this polymer in order to achieve good processibility and satisfactory physical properties while still maintaining the low temperature flexibility. Particularly important in the processing area was to develop compounds which showed good mill and calender release.

Compounds which could be calendered and which possessed good low temperature and stress-strain properties were to be used in trial productions of short lengths of hose. The best compounds and hose manufacturing techniques found were then to be utilized in fabrication of large lengths of fuel hose.

3. Compound Development

The initial stage of our compounding studies was an attempt to find stocks which processed adequately. Once good processing was achieved, attempts were made at attaining improved tensile strength, tear strength and adhesion to fabric. In these studies, it was deemed necessary that low temperature flexibility (Gehman T_5 = -70°F) be maintained before the stock would be considered for hose building trials.

4. Trial Hose Fabrications

Satisfactory compounds developed under this contract were utilized in building of short lengths of hose by the Boston Industrial Products

Division of American Biltrite, Inc. (subcontractor). In all of the hose preparations, a laminated calendered sheet construction was utilized. Also, rayon was braided in such a manner as to permit strike through and knitting of the rubber throughout the hose. Both collapsible and suction type fuel hoses were prepared. Tests were run on the completed hoses in order to provide direction for future manufacturing efforts.

5. Final Hose Production

The final hose building effort was an attempt to prepare 125 feet of collapsible hose and 35 feet of suction hose. The best compounds and hose building techniques developed during the contract were utilized in this final effort. The hose design was the same as used in trial runs.

6. Experimental Details

A. Instruments

1. Laboratory Rubber Mills

a. 2" x 6", L. Albert and Son, Model A-6974, capacity: ca. 100 g of PNF®-LT stock

b. 6" x 12", Farrel-Birmingham, Inc., Model 44630, capacity: ca. 2 lbs. of PNF®-LT stock

c. 10" x 12", Farrel-Birmingham, Inc., Model 44667, capacity: ca. 5 lbs. of PNF®-LT stock

2. Brabender Mixer -- Model PL-V150, C. W. Brabender Instruments, Inc., capacity: ca. 120 g of PNF®-LT stock

3. Banbury Mixer -- Model B Banbury, Farrel-Birmingham, Inc., capacity: ca. 1900 g of PNF®-LT stock

4. Laboratory Balances

   a. Sartorius, Model 2403 -- used for weighing of curing agents and pigments for small batches ($\pm$0.01 g)

   b. Toledo, Model 3710 -- used for weighing of pigments for large batches

5. Instron Model No. 1130 -- The Instron Corp. -- was used for stress-strain measurements. This instrument was interfaced with an IBM 1130 Computer for computation of stress-strain data.

6. Shore Durometer -- Shore Instrument and Mfg. Co., Inc.

7. Gehman Torsional Wire Apparatus -- Firestone instrument constructed according to ASTM-D-1053 and a Wallace Test Equipment instrument.

8. Compression Set Jigs -- 25% Deflection, Method B, were constructed at Firestone according to ASTM-D-395

9. Forced Air Oven -- Blue M Electric Co., for heat aging of polymer

10. Cold Tension Recovery -- The test instrument consisted of a measuring board to which are mounted several stretching devices consisting of a movable and a fixed clamp. Lines are engraved on the board at intervals corresponding to each 10% stretch, based on the length of the specimen between the 1/4 inch stubs.

B. Mixing Techniques

Brabender and Banbury Mixes -- A small amount of black is added to the mixer followed by addition of the polymer. The remaining black, MgO and stabilizer are added in increments. The compound is mixed for 8 to 10 minutes and dumped. Curing agent is then added to the masterbatch banded on a warm (130°F) mill.

C. Physical Test Methods

Test specimens were sheeted out on a rubber mill and press cured at 1000 psi unless otherwise stated. Tests were also run on specimens obtained from hose samples.

1. Stress-Strain -- ASTM-D-412, Die C, 73°F. Specimens were cut from press-cured 1.5" x 4" x 0.040" or 6" x 6" x 0.075" slabs.

2. Shore "A" Hardness -- ASTM-D-2240, tests made on small cylinder (0.25" h x 0.53" d)

3. Compression Set -- ASTM-D-395, Method B, 25% Deflection, press cured cylinder. Low temperature tests -- ASTM-D-1229, same sample and conditions.

4. Gehman Low Temperature Measurements -- ASTM-D-1053. Specimen 1.5" x 0.125" was cut either from a hose sample or a press cured 6" x 3" x 0.075" slab. An IBM 1130 Computer was programmed for computation and print-out of Gehman data and graphs. All testing was performed in accordance with guidelines of Attachments #1 and #2 cited under Paragraph F.1 of Section F and Section J entitled "Special Provisions."

5. T-Adhesion Test -- A Firestone test performed as follows:

   a. Using a Hytronic Cutting Machine (Model A; United Shoe Machinery Corp.) and a 6" x 0.50" die prepare an adequate number of sheeted strips (0.110") for pad building.

   b. Ply one piece of rubber stock (6" x 0.50" x 0.110") unto one piece of calendered fabric backing (0.051").

c. Place sample in building mold with fabric side down.

d. Place ten cords (ca. 7" in length) with equal spacing on top of the two piece assembly.

e. Invert another two piece assembly, made as in a. and b., on top of the cords so that cords are between two layers of stock to be tested.

f. This assembly should now fit snugly into mold.

g. Cure adhesion pads as desired (usually 45' @ 320°F in this work).

h. Cords are pulled from the rubber pads by means of an 1130 Instron at a test speed of 10"/minute. The top grip is a special holder made for the cured sample, with a slot in the bottom to permit the sample to be inserted with the cord protruding. The bottom grip is a wedge type designed to exert increasing tightening as the cord is pulled.

i. The ten cords are pulled and averaged. Multiplication by two yields the lbs./in. value reported.

6. Trouser Tear -- Test followed ASTM D1938 except for these modifications:

a. Force necessary to propragate a tear measured on a rubber sheet (0.075") and not a plastic film.

b. The specimens consist of strips 3.5" x 2.0" with a longitudinal slit 2.5" long down the middle of the sheet.

7. Tension Recovery -- This test followed the procedure given in the Purchase Description of this contract and outlined as follows:

a. With the specimens at a temperature ranging from 68°F to 78°F, they are clamped in the stretching devices and pulled back until the 1 1/2 inch portion of the specimen has been stretched to 100% elongation and fixed in that position.

b. The stretching devices and the specimens shall be conditioned in a low temperature chamber for 166 hrs. ± 1 hr. at -70°F ± 2°F. The measuring board shall be conditioned for not less than two hours at the same temperature.

c. With the test instrument and specimens still in the low temperature chamber, the movable clamp is released from its fixed position, and the assembly is conditioned for an additional 30 minutes at -70°F.

d. The final length of the specimen is determined 30 min. (±10 sec.) after release of the clamps and with the stretching devices and specimens held at an angle of 15° from the vertical.

e. The cold tension recovery percentage for each set of three specimens is calculated and averaged. The average value is used to determine compliance with the specification requirements.

f. The percentage of cold tension recovery is computed from the formula:

$$\% \text{ cold tension recovery} = \frac{Ls-Lf}{Ls-Lo} \times 100$$

where: Ls = stretched length of specimen

Lf = final length of specimen

Lo = initial length of specimen

test specimens: 0.080" wide x 1.5" long with 0.25" square at each end

8. <u>Brittleness</u> -- determined in accordance with ASTM designation D746.

9. <u>Torsional Stiffness Ratio</u> -- determined in accordance with Method 5612 of Federal Test Method Standard No. 601.

10. <u>Existent Gum</u> -- This test followed the procedure given in the Purchase Description of this contract and ASTM-D-381-70. A test sample of hose not less than 14 inches long is plugged with a clean corrosion resisting cylinder 2 inches long secured in place with a clamp. The sample of hose is filled to within 2 inches of the top with TT-S-735, Type II fluid. The top of the hose is then plugged in a manner similar to the bottom. The sample is stored in a vertical position for seven days at ambient temperature of 100°F (±2°F). Every 24 hours, the fluid is agitated for five minutes by moving the hose back and forth from vertical to horizontal positions at a rate of two cycles per minute. At the end of seven days, the fuel is agitated again for five minutes and immediately removed.

The fuel is tested for washed and unwashed existent gum in accordance with paragraphs 9.1-9.6 and 9.8-9.12 respectively of ASTM-D-381-70.

A modified version of this test utilizes diced samples of hose compound. A 5.0 g sample ($<$ 70 mils thick) is cut from the hose and diced into approximately 1/16 inch squares and placed into a flask containing 250 ml of TT-S-735, Type II Fluid. The flask is kept for 48 hrs. at 735°F (±5°F) with occasional stirring. After filtration through Whatman 41H (or equivalent) paper, the existent gum content is determined (as above).

7. Preliminary Compounding Studies

A. Unaged Polymer

The 137 pounds of polymer used in this work were prepared in four batches. Stress-strain and Gehman data for the individual batches (Table I) show all to be close to specifications and comparable. Analyses of the raw polymers were quite consistent for the four batches. As a result, the four batches were combined to yield a uniform lot of polymer to be used for all development work. This polymer was designated K18161 and utilized as is in all of the preliminary studies to be described.

Since earlier studies in our laboratories showed black-filled stocks to be best for low temperature flexibility, we started our investigation with an evaluation of various carbon blacks. A standard formulation illustrated in Table II was also utilized. Data in Table II show that the level of FEF black has significant influence on processing and low temperature flexibility. At lower levels of FEF, the stocks processed poorly and showed excellent low temperature flexibility. Processing was improved considerably at 50 phr FEF, but the low temperature flexibility became poorer with a Gehman T_5 of only -59°F obtained. Stress-strain properties were only fair with modulus increasing and tensile remaining about the same with increasing black level.

GPF and a combination of MT and FEF blacks were also evaluated in the standard formulation (Table III). Although stress-strain properties were fairly good, these stocks could not be given further consideration due to the poor mill processing. The compounds would not form a band, were very lacey and could not be calendered.

HAF black was tried at different levels and presented similar problems. The compounds processed very poorly and low temperature flexibility was poor at high levels of black (Table IV).

It was felt that Austin black would have little effect on low temperature properties and, therefore, was tried in combination with

FEF black (Table V). Even at the high levels of blacks, processing difficulties were evident; also, the low temperature flexibility was not acceptable.

The processing problems experienced were similar to those evident with high nerve polymers. To reduce this nerviness, the polymer was heat-aged in a forced air oven for one hour at 300°F. This treatment had no adverse effects on stress-strain properties (Table VI). However, no improvement in processibility resulted.

Trying to remedy our major problem, we evaluated stearic acid as a processing aid. With the 50 phr FEF compound, excellent mill processing was achieved through addition of stearic acid (Table VII). At lower levels of FEF required for low temperature flexibility, however, the stearic acid did not have any influence on mill processing (Table VIII).

The good processing formulation with 50 phr FEF was unsatisfactory for low temperature flexibility. Using this formulation, lower levels of peroxide were tried to determine if reduced cure states might improve low temperature flexibility. Slightly lower T_5 and G at -55°C values were observed with the lowest peroxide level compound (Table IX), but the improvements were insufficient to warrant use of this formulation for hose building.

An evaluation of a precipitated silica, Quso WR-82, was also made. At all levels of this silica, poor processibility was obtained (Table X).

B. Heat-aged Polymer

The high nerve of the modified PNF® (PNF®-LT) being used still seemed to be a logical cause of our processing difficulties. It was shown earlier that a one hour treatment at 300°F produced no improvements in processing and no adverse effects on normal vulcanizate properties. Thus, the polymer was subjected to more vigorous heat treatments and then compounded in a standard formulation with only 30 phr FEF. The results shown in Table XI indicated that longer heat treatments did indeed remedy our processing problems. Also, at these lower FEF black levels, good low temperature flexibility was achieved as evidenced by the Gehman data shown. Normal stress-strain data were indicative of slight overcure and no serious degradation of the polymer from the heat treatments. It appeared that 8.5 hours at 300°F produced the best results.

8. First Hose Building Trial

Having attained satisfactory processing and low temperature flexibility, we proceeded to a trial hose building effort with the heat-treated polymer. The formulation chosen was our standard one consisting of 100 parts rubber, 30 FEF, 6 MgO, 2 stabilizer and 0.4 Vulcup R.

Our goal in this trial was to determine if our standard formulation could be utilized in the hose building process. The suction type hose seemed to be the more difficult to fabricate, and so we planned to make a 10 foot length of suction hose and only a one foot length of collapsible hose. Both hoses were to be prepared from laminated calendered sheets with braided rayon (2200 denier, 2 plies) requirement.

Five small Banbury mixes were necessary in order to produce the required stock for this initial trial. The stocks mixed very well, and each mix was cure checked. Results of these cure checks (Table XII) indicated that the five batches could be blended and calendered. The calendering, on a 20" calender with rolls at approximately 130°F, proceeded very well. The resulting sheets were smooth and uniform. The dimensions of the calendered stocks are shown in Table XIII.

The suction hose was then built as follows:

1. Tube stock (0.050") was wrapped twice around a 2" OD mandrel.
2. The tube surface was freshened with MEK.
3. Tube was passed through a 48 carrier textile horizontal braider where one layer of rayon (2200 denier, 2 plies) was applied in a 2 over, 2 under pattern.
4. Fabric-tube assembly was covered with a cement consisting of 20% PNF®-LT stock (XS from calendering) dissolved in acetone.
5. One inner ply (0.037") was applied.
6. Steel wire (0.065" OD) was spiraled on at a spacing of 0.25".
7. Another inner ply (0.037") was applied.
8. The entire assembly was passed through the braider for application of another layer of rayon (identical to the previous layer).
9. The PNF®-LT cement was again applied.
10. Two plies of cover stock (0.050") were added to complete the hose.
11. The hose was double wrapped with wet nylon curing tape and cured in a steam autoclave for two hours at 320°F. The mandrel was hollow to allow steam to circulate inside.

The collapsible hose was built in similar fashion but consisted of only two plies of tube stock, braided rayon, one inner ply, braided rayon and two plies of cover stock.

No problems occurred during these hose building operations. Green strength of the stock was adequate to resist any pull down by fabric or wire. The hoses cured satisfactorily and removal of the hoses from the mandrel was relatively easy with McLube 1775 as a lubricant.

Tests were performed on the suction hose only, and results are summarized in Table XIV. In general, the results were encouraging. Hydrostatic pressure test results met or exceeded specifications. One problem area was the low tensile strength and low elongations. Also, the adhesion values were only slightly above specifications.

The stock used in this first hose fabrication trial was further tested for fuel resistance in Type II Fluid (TT-S-735) and for water resistance. Data in Table XV illustrate satisfactory results in both fluids for our compound.

9. Additional Compounding Studies

Following our first hose building trial, our efforts focused on improving stress-strain and tear properties, increasing adhesion of tube and cover, reducing cure times and development of a cover compound which would produce the desired fuel diffusion rate ratio for tube and cover. Practically all of this work was done with polymer (K18161) that was heat-aged 8.5 hours at 300°F.

To improve the elongation of our hose formulation, compounds with lower Vulcup R levels were evaluated (Table XVI). At the lower peroxide levels, the desired elongations (> 150%) were realized while modulus decreased and tensile strengths remained unchanged. Surprisingly, these stocks with lower cure states did not exhibit higher tear strengths. Gehman low temperature properties were essentially the same for all compounds. All in all, it appeared that the reduction in peroxide level would not cause any problems.

The Vulcup R used as curative in all of our work is designed for cures at 340°F. However, the maximum cure temperature attainable in production autoclaves was 320°F. Thus, it was felt that improved properties and shorter cure times could be realized with peroxides that initiate at lower temperatures. Several different peroxides were evaluated with our standard formulation (Table XVII). Monsanto Rheometer data indicate shorter times to 90% cure for Dicup and Luperco after 35'/320°F cures; however, normal stress-strain properties were essentially identical for all compounds, including the Vulcup R formulation. Trouser tear strengths and low temperature flexibilities were also comparable for all compounds. Thus, at 320°F, there seemed to be no advantages evident from these lower temperature curing peroxides.

Our approach to attaining greater fuel diffusion rates in cover than in tube stocks was to add small amounts of polymers with poor fuel resistance to the cover compound. A first attempt with a silicone polymer was futile in that the compound processed poorly and probably would not calender (Table XVIII). A preliminary evaluation of EPDM and polybutadiene containing compounds indicated that processing and normal stress-strain properties were not adversely affected (Table XIX). Fuel diffusion rate ratio determinations showed that less than 5 phr of EPDM would suffice to attain the desired ratio of 1.30 (Table XX). Thus, a compound with only 2.5 phr of EPDM was evaluated, and results were quite good (Table XXI). Physical properties were unaffected by the EPDM and a fuel diffusion rate ratio of 1.42 was achieved. At the same time, low levels of a non-fluorinated polyalkoxyphosphazene were tested. Satisfactory diffusion rates resulted, but the normal stress-strain values fell well below specifications (Table XXI).

A serious problem with our first hose was poor tear strength. Besides the obvious consequences, poor tear also contributes to the adhesion problem because of the strike through of rubber in the hose design used. With high tear strength, this strike through would make separation of tube or cover from rayon difficult. Independent studies in our laboratories with PNF®-200 indicated that small quantities of Teflon 8A improved tear strength significantly. Data in Table XXII illustrate the influence of increasing levels of Teflon 8A on tear and normal stress-strain properties of our standard PNF®-LT formulation. These preliminary results were quite encouraging in that tear strength was improved considerably and modulus and tensile strength also increased. However, upon close inspection of the test pieces, it was evident that a problem existed with the Teflon-containing stocks. The compounds appeared to consist of thin layers of rubber which could readily be delaminated.

Because of the improvements in tear attained by Teflon addition, we attempted a couple of variations in mixing procedure to overcome the delamination difficulty. First, we tried addition of the Teflon in the Brabender rather than on the mill as in our initial efforts. This resulted in improvements in tear and normal stress-strain properties similar to the earlier trials, but delamination of the stocks was still evident (Table XXIII). Another mixing variation, addition of silicone oil to improve Teflon dispersion, also had no influence on the delamination problem (Table XXIV).

A couple of silica fillers were evaluated to determine their influence on stress-strain and tear properties. Quso WR-82 filled compounds showed very poor tear strengths that could have been caused in part by the over-cured nature of the stocks (Table XXV). Hi Sil 233 formulations produced similar results (Table XXVI).

T-adhesion values to rayon were determined for our formulations used in the first hose building trial (Table XXVII). The values observed were quite low, and there was no evidence of rubber on the cord following the test. To remedy this situation, various known adhesion promoters were added to our standard formulation and tested. None of these additives greatly improved adhesion; all, except Cohedur RL, had detrimental effects on normal vulcanizate properties (Table XXVIII), and none showed any rubber on the cord following the test.

Several different black fillers, some of which were evaluated with unaged polymer, were tried with the heat aged polymer to determine their effects on normal stress-strain, tear strength and low temperature flexibility. The results of this study are illustrated in Table XXIX. All of the compounds processed fairly well, although calendering problems probably would have been encountered with the low structure HAF and the GPF stocks. The SAF compound showed considerably higher tear strength, but low temperature flexibility was unacceptable. The HAF compound had fairly good low temperature flexibility, but tear strength was very poor. It appeared that the best overall properties were obtained with the standard FEF formulation.

10. Second Hose Building Trial

Another hose building trial was attempted with the cover stock (R198625) described earlier (Table XXI). Besides attempting to attain the desired fuel diffusion rate ratio between tube and cover, we were also attmpting to increase the elongation values obtained in our first hose building effort. To achieve the latter effect, the Vulcup R level was reduced from 0.4 phr level used in the first trial to 0.2 phr. Otheriwse, the formulation for this trial remained unchanged. Table XXX shows the cure check results on three Banbury mixes each of tube and cover stocks. The data showed that the three batches of each stock could be combined for calendering and that the properties were about as we had desired.

We experienced a little more difficulty in calendering these stocks. The compounds were sticking slightly to the calender rolls. In spite of this stickiness, sufficient stock was calendered to build 10-15 feet of collapsible hose.

In this second trial, we attempted to prepare only collapsible type hose (15 ft.) by the identical process described earlier. The hose building itself went smoothly with no problems encountered up to the curing stage. After curing for 90 min. at 325°F, great difficulty was experienced in removal of the hose from the mandrel. We eventually were forced to cut the hose. Stress-strain properties of the tube and cover sections were very poor (Table XXXI), and the undercured nature of the tube could have contributed to the poor release from the mandrel. The lubricant used was identical to that used in our first trial (McLube 1775).

In an attempt to determine the cause of the poor stress-strain properties obtained on a sample of the hose, we determined physical properties on excess cover stock that was both press and steam cured. The results of these determinations clearly showed that the poor mechanical properties were not caused by steam curing (Table XXXII).

Since cure checks prior to calendering showed good results, it appeared that our problem had arisen during the calendering process. To test this hypothesis, a study was made of the influence of calendering conditions on ultimate physical properties. This investigation illustrated that repeated high temperature calendering could cause a reduction in subsequent cure states (Table XXXIII). In the second hose building trial, we did have more problems which necessitated more than one pass through the calender. Also, our temperature control was not very good.

11. Further Compounding Studies

Following the second hose building trial our efforts continued to focus on improvement of processing, tensile strength, tear strength and adhesion to rayon. We also investigated the replacement of the pure peroxide, Vulcup R, with a 40% dispersion of Vulcup on Burgess KE (Vulcup 40KE). The results presented in Table XXXIV indicate comparable cures with the two peroxides. We decided to utilize the Vulcup 40KE since it would be easier to handle, should give better reproducibility with our small batches and might reduce the possibility of peroxide volatilization during calendering.

It was also felt that improved tensile, green and tear strengths might be attained through use of a polymer that was heat aged for a shorter period (< 8.5 hrs. @ 300°F). We first followed the drop in dilute solution viscosity (DSV) with 300°F aging up to 8.5 hours (Table XXXV). Polymers (K18161) aged for 4.5, 6.0 and 8.5 hours were then compounded, cured and tested (Table XXXV). It was shown that 4.5 hours aging (K18353) was not sufficient to reduce nerve and yield good processing. The polymer aged for 6.0 hours (K18352) did process well. Normal stress-strain properties were improved slightly by the reduced aging times while tear strength and low temperature flexibility were essentially unchanged. It was decided to continue looking at polymers aged for both 6 and 8.5 hours to determine if there were any benefits from shorter aging times.

Continuing our search for improved reinforcement and processing, we evaluated additional carbon blacks and combinations of carbon blacks. Attempts to utilize small amounts of SAF in combination with Austin black did give reasonably good low temperature flexibility, but processing and stress-strain properties were unsatisfactory (Table XXXVI). SAF black in combination with FEF black produced excellent low temperature properties and good tensile strength, but processing was again very poor (Table XXXVII). Use of ISAF and FEF blacks in combination (25 phr total) offered somewhat better processing than the other combinations but still poorer than 30 phr FEF black alone (Table XXXVIII). Improved tensile strength and good processing were realized with ISAF blacks alone (Table XXXVIII). However, tear strengths were poor and low temperature properties only marginal. Finally, a pair of blacks utilized in the printing industry were evaluated. These blacks yielded good mill processing at low temperatures, good stress-strain properties and improved tear strengths. However, low temperature properties were deemed unsatisfactory (Table XXXIX).

An earlier study with the silica Quso WR-82 produced vulcanizates that were overcured. Hence, a re-examination was made at lower peroxide level and showed that reasonably good stress-strain properties could be achieved at 0.75 phr Vulcup 40KE (Table XL). No advantages in processibility or tensile and green strength over the FEF formulation were evident.

Samples of nylon, rayon and polyester that had been treated for improved adhesion to rubber were tested for adhesion to PNF®-LT tube and cover stocks. The treated fabrics did show better adhesions, but the improvements were only slight. Adhesions to all fabrics were poor (Table XLI) with no evidence of rubber on the cords after testing.

12. Third Hose Building Trial

In view of the difficulties experienced in our second hose building effort, a third trial was made in order to produce hose with the desired differences in fuel diffusion rates between tube and cover stocks. We also hoped to remedy our calendering problems and to try Vulcup 40KE and the polymer aged for only 6.0 hours.

Both press and steam cure checks were run on our preferred hose compounds. In addition, a cure check was made after calendering on a small laboratory calender (Table XLII). Little difference was observed between stocks (R199437) that were press and steam cured. However, calendering did produce a sizeable reduction in cure state (R199463). Normal stress-strain properties were still fairly good after calendering (Table XLII). A slight increase in Vulcup 40KE was made in the hose formulations (illustrated in Table XLIII).

In some calendering work on a small lab calender, it was observed that our stocks tended to stick more at 130°F than at 150°F. This fact was used to our advantage in calendering for our third hose building trial. The lower roll of the calender which contains the cutting knives was kept at 140°F while the upper roll was maintained at 150°F. This prevented the stocks from going to the top roll and pulling away from the cutting knives. Calendering of both tube and cover stocks proceeded very smoothly.

The building of collapsible and suction hoses (5 and 7 feet respectively) went very well except for difficulties in removal of the collapsible hose from the mandrel. The suction hose released quite readily by applying pressure with a wrapped bar. This same technique resulted in release of the collapsible hose but only after a considerable length of time during which slight damage occurred to the hose.

Results of various tests on the hose compounds are summarized in Tables XLIII to XLV. Stress-strain properties were fair but below the desired specification. Elongations were above the desired 150% level, and tensile strengths were around 1000 psi except for the tube section of the collapsible hose which appeared to be undercured. Press cures on excess stock gave much better normal properties and indicated that the steam cure had produced poorer cures this time. Gehman low temperature test results were excellent for tube sections, but the T_5 was undesirably high for the cover. Apparently the small amount of EPDM was detrimental to low temperature flexibility. Additional low temperature tests were performed by the Department of the Army and are summarized in the letter shown in the Appendix. In general, the results were quite favorable with no serious problems resulting from the conditioning of specimens for 7 days at -70°F. Tear strengths were not very good but about as high as we can expect (Table XLIII). Adhesion values for tube and cover to ply of the suction hose and for cover to ply for the collapsible hose were well within specifications (Table XLIV). However, tube to ply adhesion for the collapsible hose was unsatisfactory. The latter result was difficult to understand since identical compounds were used for both hoses. The hydrostatic pressure tests gave acceptable results for both hoses. Fuel and water resistance was also satisfactory for both tube and cover stocks (Table XLV).

13. Final Compounding Studies

After our third hose building trial, it was evident that we still needed improvements in tear and tensile strengths and a better mandrel lubricant was required. We also had to develop a new cover stock that provided the desired fuel diffusion rate and did not influence low temperature flexibility.

Earlier studies showed that addition of small amounts of polybutadiene produced the desired fuel diffusion rate in cover stock. We repeated this work and checked the effect on Gehman low temperature properties. It was found that the fuel diffusion rate and low temperature properties were acceptable for a cover compound containing 2 phr of polybutadiene (Table XLVI).

Continuing our search for improved physical properties, we evaluated additional black reinforcing agents. A high structure GPF and N 234 ISAF blacks were compared to our standard FEF formulation. The ISAF compound fared quite well in all tests, particularly tear strength, but the Gehman low temperature properties were well below specifications. The GPF compound showed no advantages over the FEF stock and was poorer in tear strength (Table XLVII).

An earlier investigation with combinations of ISAF and FEF blacks indicated that these compounds were close to meeting specifications and that a repeat analysis was warranted. Data in Table XLVIII summarize this reinvestigation. Normal stress-strain properties, low temperature flexibility and tear strengths were essentially the same as our FEF compound. Although the low temperature properties were greatly improved, the control compound was also better than usual. This may indicate some problems in this series of Gehman tests. Since processing of the FEF formulation was slightly better, we would not recommend a switch to the ISAF-FEF combination.

Several other blacks also provided properties close to or better than specifications. Hence, these compounds were evaluated again with some minor adjustments in peroxide and black levels. Good stress-strain properties were realized with the HAF, ISAF (HS) and Rub Corex P stocks, but low temperature flexibility was not very good (Table IL).

An acetylene black, Shawinigan, was also evaluated at 30 phr level. This stock processed well and showed fairly good tensile strength although the stock was obviously overcured (Table L). The Gehman T_5 value was excellent, but the G value at -55°C was higher than desired. Tear strength was very poor, but this was undoubtedly influenced by the tight cure obtained on this stock. All in all, the Shawinigan compound with a reduced cure state would probably be comparable to our FEF formulation. Any future studies, possibly involving extrusion of hose compounds, should consider both the Shawinigan and the ISAF-FEF combination compounds.

Earlier work showed that Teflon increases our tear strength but also produced a laminated stock that could be peeled apart. We tried a different type of Teflon, Teflon-6, to see if this delamination could be avoided. The cured stocks still exhibited some layering, and the tear strengths were not improved significantly (Table LI). We also investigated the effects of Teflon 8-A in combination with Silane A-174, a coupling agent. The stocks could still be delaminated, and low temperature properties were quite poor (Table LII).

14. Evaluation and Compounding of New Polymer for Hose Production

In order to prepare 125 feet of collapsible hose and 35 feet of suction hose, it was necessary to synthesize an additional 168 pounds of PNF®-LT. Table LIII illustrates the raw polymer analyses of six batches of material that would provide sufficient material. These analyses show that the DSV's of these polymers are significantly lower than obtained for earlier polymer (K18161) and that the Tg's are lower. The latter result is due to the lower levels of fluorine observed in these polymers.

We compounded, cured and tested each of the individual batches. Normal stress-strain properties were not very good and mill processing was very poor. The stocks would not form a tight band but simply bagged off the mill. Gehman low temperature properties were exceptionally good, but fuel resistance was very poor (Table LIV).

All of the problems of the above compounded stocks could be ascribed to too low a level of fluorine in the polymer. This conclusion is drawn from previous experience in our laboratories. Our earlier, independent studies also indicated that the PNF®-LT polymers with very low levels of fluorine could be blended with PNF®-200 to attain a good balance of low temperature flexibility and solvent resistance. With this in mind, we evaluated blends of one of the new PNF®-LT's and a PNF®-200. Results of this investigation, summarized in Table LV, were very encouraging. First of all, the addition of PNF®-200 greatly improved mill processing. Also, stress-strain properties were improved so that even at 20 parts of PNF®-200 to 80 parts PNF®-LT, properties equivalent to those observed in our earlier work were obtained. Both at 20 and 40 parts of PNF®-200, acceptable low temperature properties were realized. Fuel resistance was marginal with 20 parts of PNF®-200 and within specifications (<40%) for the 60:40 blend. In going to 60 parts of PNF®-200 and 40 parts PNF®-LT, low temperature properties fell into the undesirable range. Thus, it appeared that blends of the two polymers would yield desired properties provided the blends did not contain predominantly PNF®-200.

The six batches of PNF®-LT were blended in six separate lots on a 20" rubber mill. A cure check on three of the six lots indicated a uniform blend was produced (Table LVI). This blend (K15900) was utilized to optimize and perform further checks on the PNF®-LT and PNF®-200 blends.

Table LVII illustrates results of our investigation of 80:20, 60:40 and 50:50 (PNF®-LT:PNF®-200) blends. Good stress-strain properties and reasonably good processing were again observed for all of the blends.

Low temperature properties were also acceptable for all compositions studied. On the basis of overall properties, the 60:40 blend was chosen for our hose building efforts. A peroxide level study with this blend showed the optimum Vulcup 40KE level to be in the 1.0 to 1.2 phr range (Table LVIII). A final check on fuel resistance with these stocks produced satisfactory results (Table LVIII).

Prior to going to our final, large Banbury mix, we performed a Banbury mix and checked the calendering of recommended tube and cover stocks on small lab equipment. The stocks mixed very well and yielded good stress-strain properties (Table LIX). The stocks were purposely cured tighter than ultimately desired in anticipation of losses in cure state during calendering and steam curing. The slightly higher peroxide level in the tube stock was also used to compensate for the lower cure states usually observed in the tube section of the hose. The calendering was somewhat difficult due to slight sticking of both compounds to the calender rolls. Temperature did not seem to have as great an influence on release, although higher temperatures did improve the calendering slightly. Rather surprisingly, the calendering did not appear to influence the subsequent curing and mechanical properties of these stocks (Table LIX). In view of these results, we proceeded to our large mix of final compounds with the same formulations except for a slight decrease in peroxide levels.

15. Production of Large Lengths of Hose

The final, large hose building effort was performed with tube and cover formulations illustrated in Table LX. A masterbatch totaling 232 pounds and excluding peroxide and polybutadiene was mixed in a Banbury.

The batch was mixed in 6 minutes and dumped at 250°F. No free pigments were evident, and the stock dropped readily. The batch was then divided, and peroxides and polybutadiene were added on a mill. The mill mixing went quite well; both stocks could be cut readily from the mill, and the cover stock, which handled somewhat better, could be rolled on the mill. The stocks reached 212°F during the mill mixing. Cure checks showed good properties for both compounds (Table LX).

The calendering operation was then performed with top rolls maintained around 150°F and the bottom roll kept at 130°F. We managed to calender all of the desired lengths of stock, but we did experience problems. The compounds occasionally stuck to the top rolls causing a tearing of the calendered sheets. A slight improvement in mill release would greatly facilitate this phase of hose production.

Fabrication of the desired lengths of hoses followed exactly the process described earlier. We performed a preliminary preparation of about 36 feet of collapsible hose in which two different mandrel release agents, talc and silicone mold release, were evaluated. The hose building was marred only by a build-up of stock which occurred during the braiding operation and resulted in a small knot in the hose. Release of the hose was quite good with both types of release agents. The hose had to be cut at the location of the knot, and this resulted in only 22 instead of the required 25 feet. The remainder of hose was used for testing purposes.

With the good release obtained in the preliminary run, we continued on to the preparation of the 100 feet of collapsible and 35 feet of suction hoses. These lengths were prepared with no problems and release from the mandrel was good. Talc was utilized as the lubricant for all of these preparations.

Both types of hoses were subjected to hydrostatic pressure testing, and results of these tests are illustrated in Table LXI. All of the requirements of these pressure tests were met for both the collapsible and suction hoses. The diameter and weight of the hoses were checked, and the collapsible hose was slightly above the desired 1 lb./ft. requirement. Since identical compounds were used in the two hoses, adhesions were determined on the collapsible hose only. Both before and after filling with fuel, the adhesion values were below the desired 10 lbs./in. Crush resistance on the suction hose was satisfactory.

Remaining test results from American Biltrite are summarized in Table LXII. These tests were performed on the collapsible hose only, and results should be identical for the suction hose. Tensile strengths of both tube and cover were below specification and lower than expected from tests prior to hose building. Once again, the tube section was not cured as tightly as the cover. Stress-strain measurements after immersion in Type II Fluid of TT-S-735 and distilled water (160°F) indicated marginal retentions of vulcanizate properties. Rather surprisingly, the cover stock which contains the polybutadiene showed better retention of physical properties after 14 days in the Type II Fluid. Volume increases and weight changes in Type II Fluid were within specifications. One test result that was very bothersome was the high existent gum value. This prompted some further examinations in the Firestone Laboratories which will be discussed shortly.

No cracking or checking of cover stock was observed after the required ozone exposure, and retention of stress-strain properties after accelerated weathering was excellent. The low temperature properties of the tube and cover stock were satisfactory as evidenced by the brittleness test and the Gehman test results (Table LXII). The Gehman T_5 values were -68-69°F and G values at -55°C were 429-700 psi.

To check that no problems were incurred during large scale mixing and calendering, some excess stock from the hose building was press-cured and tested. Stress-strain properties show a glaring difference from those obtained on hose samples (Table LXIII). Tensile strengths are close to meeting the 1500 psi specification and 100% modulus in the tube stock is more than twice that observed on a sample taken from the hose. Retentions of stress-strain properties after aging in Type II Fluid of TT-S-735 were also much better for these press cured stocks. Requirements were easily met for these fluid aging studies.

Due to the extremely high levels of existent gum found in American Biltrite's testing, we repeated these tests at Firestone. Utilizing the diced sample technique, a value of 60 mg/100 ml was observed. Use of a 14" length of hose, however, yielded 1880 mg/100 ml. The latter level, confirming American Biltrite's results, prompted an investigation of the residue from the existent gum test. It was found that the residue consisted of two liquid layers. The two phases were separated and analyzed by NMR. The upper layer showed chemical shifts at 6.91 δ, 2.12 δ and 0.78 δ indicative of the aromatic and aliphatic hydrocarbons of the fuel mixture. Also present was a broad peak at 3.95 δ indicative of the methylene protons adjacent to oxygen and present in pendant groups of our modified PNF®. These same peaks were evident in the NMR of the lower layer except that the peak at 3.95 δ was greatly increased. Also evident were peaks at 5.65 δ and 6.0 δ indicative of the terminal proton in our C_5^f fluoroalkoxy pendant groups. The upper phase of this residue was by far the major constituent.

DISCUSSION

The basic problem in this study was to take an inherently good low temperature rubber, our modified phosphonitrilic fluoroelastomer (PNF®-LT) and produce fuel hose from it while maintaining the good low temperature properties. Thus, the problem was one of developing PNF®-LT compounds which satisfied requirements for hose building while still maintaining good low temperature properties and fuel resistance.

The key requirements for compounds utilized in the fabrication of collapsible and suction type fuel hoses are enumerated below:

1. Must be calenderable -- compounds must release well from mill rolls and possess sufficient tear strength to resist damage to stock.
2. Must have building tack -- calendered sheets will be built up from several plies and green stocks must stick slightly to facilitate this operation.
3. Must resist pull down of fabric and wire reinforcement; here again, good green strength is necessary.
4. Must have good adhesion -- layers of rubber and reinforcing fabric must adhere well. With design of hose utilized, this is accomplished by good adhesion of rubber to fabric and by good tear strength. The latter factor is important because of the fabric braid pattern which permits significant strike through of rubber.

5. Cured hose must have reasonable strength to withstand normal wear and tear; thus, high modulus, tensile strength and tear strength are desirable.

6. Good release of hose from the mandrel -- this should be accomplished primarily through use of mandrel lubricants.

The mandrel release and building tack were not of great concern in initial approaches to the problem. Hence, our initial goals were to develop compounds which processed well on rubber mills and showed good green strength, tear strength and normal stress-strain properties. The adhesion problem was addressed separately and only after the above properties were realized.

It was felt that realization of our initial goals would be possible through judicious choice of reinforcing agent. Consequently, major emphasis was given to evaluation of different fillers. As pointed out in our earlier studies, a major restriction in these investigations was the fact that the filler type did influence low temperature performance. The more highly reinforcing or small particle size fillers were detrimental to low temperature flexibility.

A major processing problem with our first large batch of PNF®-LT was overcome by heat aging of the polymer. Apparently these high DSV products possessed too much nerve for good mill processing or calendering. The 300°F treatment for 6-10 hours reduced the nerve of the rubber and resulted in greatly improved processing.

With the improved inherent low temperature flexibility of the PNF®-LT, we felt that it might be possible to withstand some losses in low temperature performance from the use of more highly reinforcing fillers. However, it was found that ISAF and HAF type blacks at the 30 phr level produced unsatisfactory low temperature flexibility. Reduction of the level of these type blacks resulted in poor mill processing.

The best reinforcing agent found was FEF black at 30 phr level. With this compound, we attained satisfactory processing, adequate green strength and stress-strain properties while maintaining good low temperature flexibility and fuel resistance. Although tensile strength was below specifications, it was felt that the low temperature properties and processibility of this compound gave it preference over any other formulations. A couple of other black compounds, the FEF-ISAF combinations at 25 phr and the Shawinigan formulation, were closest to the FEF in overall properties.

A major deficiency of the FEF compound was low tear strength. Other formulations with improved tear did not meet low temperature specifications. Teflon 8-A was found to improve tear resistance significantly, but it also produced a serious delamination problem. Attempts to eliminate the delamination difficulties failed. To optimize our FEF compound, we tried to maintain elongation at break above 150%.

In our hose building trials, it was found that building tack was very good, adhesion of tube and cover to inner plies was marginal and release of the hoses from the mandrel was difficult. The mandrel release problem was solved through use of better lubricants such as talc or silicone mold release. In our initial trials, we used a lubricant, McLube 1775, which offered the lowest probability of remaining in the completed hose.

The adhesion values observed in our trial hose preparations were marginal. We felt this situation could be greatly improved by attaining better tear strength in our stocks and/or better adhesion of our stocks to rayon. The tear strength problem has already been discussed. To improve adhesion to fabric, we evaluated various adhesion promoters and some treated fabrics. Both of these approaches proved fruitless.

Following our hose building trials, it was obvious that we could process our compounds, hoses could be built from these compounds and the hoses were satisfactory except for low adhesion and low tear strength. We proceeded with production of larger lengths of hose with the same FEF formulations because the major objectives were fulfilled and no better formulations were available.

The production of large lengths of hose necessitated the synthesis of additional PNF®-LT. This synthesis work pointed out another problem with PNF®-LT--that of good control of fluorine content. These preparations yielded polymers with lower levels of fluorine than desired and resulted in outstanding low temperature flexibility but poor fuel resistance and physical properties. However, this problem was remedied through use of blends of the PNF®-LT with our PNF®-200 which contains high fluorine levels. This utilization of blends of the two PNF's provided an excellent means of controlling the fluorine content and the balance between low temperature flexibility and fuel resistance.

Use of a production size Banbury and rubber mill caused no unusual problems. A Banbury mix of 234 lbs. of stock was completed in 6 minutes and produced a uniform compound with no loose black evident. Peroxide and polybutadiene (to cover stock) were added to the formulations on a mill, and this operation showed that these compounds could be handled readily on a production size mill. The entire batch was then converted to calendered sheets of desired size. Although required lengths were obtained, some holes were later found in the sheets and were caused by occasional sticking of stock to the top roll. The sticking was only slight and sporadic so that only a small improvement in calender release would probably make this operation free of any difficulties.

The building of both collapsible and suction hoses proceeded very well. The few holes produced during calendering were mended by covering with some excess stock. Building tack and green strength of the compounds were good. Following steam cures, all lengths of hose released reasonably well from the mandrel.

Hydrostatic pressure tests showed the hose construction of both types of hoses to be sound. Also, the major objective of maintaining good low temperature flexibility was achieved. Ozone and weathering were also good. Volume increases in Type II Fluid of TT-S-735 were within specifications, but existent gum content of fuel contained in the hose for seven days was quite high. High levels of fuel components were evident in the residue from the existent gum test and make the validity of our results questionable.

Samples cut out from the hoses showed tensile strengths of 912-925 psi. Press cured samples prepared from excess stock showed tensile strengths of 1300 psi. This difference is greater than usually observed between a sample taken from a steam cured hose and a press cured sample. Studies performed during the course of this contract indicated only small differences between press and steam cured samples. Considerably longer cure times, compared to press cured slabs, were utilized in the hose fabrication. Perhaps, with the thickness of the hoses, higher cure temperatures should also be utilized.

The adhesion of cover and tube to inner plies was below specification. We had expected the adhesions to be marginal because of the use of FEF black which results in low tear strength. However, this type black was required in order to maintain processibility and low temperature flexibility. The search for additives to improve tear strength of these FEF compounds should continue. With the good strike through of rubber, the improved tear strength would result in much better adhesions. Additives to improve adhesion to rayon might help some, but poor tear strength of the rubber phase would negate the benefits of better rubber to fabric interaction.

Retentions of stress-strain properties following immersions in Type II Fluid and water were marginal. Big improvements could be realized here if the initial stress-strain properties were improved. This is clearly shown by the excellent retentions observed for press-cured samples of the hose compounds. The latter samples had considerably better initial properties.

Overall, the results of the large scale production of Arctic fuel hoses was fairly satisfactory. Hoses could be built from our modified PNF®, and the resultant hoses showed good low temperature flexibility. Properties such as tensile and tear strength had to be sacrificed somewhat in order to attain good processibility and maintain low temperature flexibility.

CONCLUSIONS

A modified phosphonitrilic fluoroelastomer has been utilized to produce collapsible and suction type Arctic fuel hoses. Based on brittleness and Gehman tests, these hoses showed good flexibility at -70°F. Both types of hoses exhibited excellent dimensional stability and physical strength. In fact, it appears that a reduction in fabric, which would improve flexibility, is feasible. Fuel resistance was generally good except for some questionable existent gum test results on our final, large lengths of hose.

The modified PNF compounds can be handled quite well in factory equipment, and it was shown that large lengths of hose could be made. Banbury and mill mixing proceeded very smoothly. Considerable lengths of material were calendered during the course of this work. It appears that we are on the borderline for very good processing on a calender. Occasional sticking of compounds to calender rolls caused some difficulties. A slight improvement in release would alleviate all problems.

Although the modified PNF® has improved low temperature flexibility, it was found that highly reinforcing filters still could not be utilized because of detrimental effects on desired low temperature flexibility. With larger particle size fillers, such as FEF black, good low temperature properties were achieved, but only marginal tensile and tear strengths resulted. With the particular hose design utilized, the low tear strength resulted in poor adhesion between tube and cover to inner plies.

RECOMMENDATIONS

The compound developed in this work processed well, could be utilized in large scale hose manufacture and exhibited good low temperature flexibility. Thus, major objectives were realized. However, other important area may require improvement and should be focal points for future studies. First of all, both tensile and tear strength of our basic FEF compound should be increased. A study of various additives to the basic FEF formulation should be made.

In large scale production, the use of an extruded tube could be beneficial. Extrudability of the good low temperature compounds of modified PNF® should be examined. For future large scale calendering operations, a slight improvement in calender release would greatly facilitate this operation. Various mill release agents should be added to the FEF compound and evaluated. Finally, the painting of a solution of PNF® compound unto fabric greatly slows rate of hose production. An alternate for this operation should be sought.

Since dimensional stability and strength of all hoses were very good, a hose containing less fabric should be evaluated. Such a hose should possess greater flexibility.

Because of the presence of presumably low molecular weight PNF® in the existent gum residue, future syntheses of these polymers should exclude any low molecular weight species.

GLOSSARY

PNF®	phosphonitrilic fluoroelastomer containing pendant fluoroalkoxy groups. Supplied by Firestone.
Modified PNF®, PNF®-LT	a phosphonitrilic fluoroelastomer with reduced fluorine content.
MgO	Stan Mag ELC.
Stabilizer	Zinc bis(8-oxyquinolate).
Vulcup R	(α,α'-bis(t-butylperoxy)diisopropylbenzene).
Vulcup 40KE	a 40% dispersion of Vulcup R on Burgess 40KE provided by Hercules.
Teflon 8A	fibrous Teflon supplied by DuPont.
Polybutadiene	HD-35, a 35 Mooney viscosity polymer supplied by Firestone.
Shawinigan black	black made from acetylene gas and supplied by Gulf Oil Canada Limited.

TABLE I

PHYSICAL PROPERTIES OF PNF®-LT COMPOUNDS AND RAW POLYMER ANALYSES

Batch No. RPP	-10159	-10165	-10206	-10208	Spec
Physical Properties					
100% Modulus, psi	455	570	575	615	Record
Tensile, psi	1045	1110	1000	1050	1500
Ult. Elongation, %	160	150	165	145	150
Gehman T_5 (°F)	-67	-70	-75	-77	-75 ± 5
G (-70°F) (psi)	319	278	257	145	500 max.
Raw Polymer Analyses					
DSV	7.29	6.78	3.94	3.39	
% Gel	0	0	0	0	
% C_2^f*	65.4	ND	62.3	59.6	
% C_5^f*	19.0	ND	22.5	27.0	
% C_5^h*	14.5	ND	13.8	12.5	
Wt. % Cure Site	1.24	1.16	0.71	0.76	
Wt. % Na	0.093	0.15	0.023	0.042	
Wt. % Cl	0.090	0.14	0.035	0.031	
Tg °C	-78.5	-77.5	-79.0	-79.5	
Wt. % F**	45.6	ND	46.5	48.1	

* Mole % of pendant groups determined by NMR.
**Determined on the basis of pendant group analyses (NMR).

TABLE II

EFFECT OF BLACK LEVEL ON PROCESSING AND PHYSICAL PROPERTIES

Stock	R197 -300	-301	-302	-303
FEF Black	20	30	40	50
Mixing Evaluation				
Mixing	Fair	Fair	Good	Good
Dump	Black Loose	Good	Good	Good
Dump Time, min.	7	8	9-1/2	8
Milling	Sticky	Won't band-Lacey	Fair	Good
Calenderable	No	No	Maybe	Yes
Physical Properties				
Normal Stress-Strain - Cured 35' @ 340°F				
100% Modulus, psi	560	705	975	1050
Tensile, psi	975	1270	1050	1050
Ult. Elongation, %	130	140	110	100
Shore "A" Hardness - Cured 40' @ 340°F	48	56	64	77
Compression Set (70 hrs. @ RT) - Cured 40' @ 340°F				
% Set	10.4	14.7	19.7	28.7
Gehman Low Temp. Properties - Cured 35' @ 340°F				
T_5, °F	-73	-70	-68	-59
G, RT, psi	64	62	88	182
G, -55°C, psi	205	304	490	1109

Formula: 100 polymer, black as shown, 6 MgO, 2 Stabilizer, 0.4 Vulcup R.

TABLE III

EFFECT OF BLACK LEVEL AND TYPE VARIATION ON PROCESSING AND PROPERTIES

Stock	R197	-303	-306	-307	-308	-309	-310
Black							
FEF		50	45	-	-	-	30
GPF		-	-	30	40	50	-
MT		-	-	-	-	-	20
Mixing Evaluation							
Mixing		Good	Good	Fair	Good	Good	Good
Dump		Good	Good	Good but dry	Good	Good	Good
Dump Time, min.		9½	10	10	10	10½	10
Milling		Fair-Good	Fair	Won't band	Won't band	Won't band	Won't band
Calenderable		Yes	Maybe	No	No	No	No
Physical Properties							
Normal Stress-Strain - Cured 35' @ 340°F							
100% Modulus, psi		1390	1220	1070	1300	1410	-
Tensile, psi		1390	1380	1160	1490	1480	1300
Ult. Elong., %		100	120	110	110	110	90
Shore "A" Hardness - Cured 40' @ 340°F		76	72	54	63	72	67

Formula: 100 polymer, Black as shown, 6 MgO, 2 Stabilizer, 0.4 Vulcup R.

TABLE IV

EFFECT OF HAF BLACK ON PROCESSING

Stock R197	315	316	317
HAF Black level	30	40	50
Physical Properties			
Mix Evaluation			
Mixing	Poor	Good	Good
Dump	Crumbly	Good	Good
Dump Time, min.	10½	16	11
Milling	Poor	Lacey	Lacey
Calenderable	No	No	No
Normal Stress-Strain - Cured 35' @ 340°F			
100% Modulus, psi	1040	1220	1200
Tensile, psi	1040	1520	1370
Ult. Elongation, %	100	120	110
Shore "A" Hardness - Cured 40' @ 340°F			
	67	70	82
Gehman Low Temp. Properties - Cured 35' @ 340°F			
T_5, °F	-65	-60	-40
G, RT, psi	132	125	251
G, -55°C, psi	612	804	3269

Formula: 100 Polymer, Black as shown, 6 MgO, 2 Stabilizer, 0.6 Vulcup R.

TABLE V

EFFECT OF AUSTIN BLACK ON PROCESSING

Stock	R197 -330	-331	-332
FEF Black level	30	40	40
Austin Black level	20	10	20
Physical Properties			
Mixing Evaluation			
Mixing	Good	Good	Good
Dump	Good	Good	Good
Milling	Won't Band	Poor	Fair
Calenderable	No	No	Marginal
Torque @ Dump (m-gms.)	3990	3920	3850
Normal Stress-Strain - Cured 35' @ 340°F			
100% Modulus, psi	-	-	-
Tensile, psi	1025	1050	945
Ult. Elongation, %	95	95	80
Shore "A" Hardness - Cured 40' @ 340°F	70	74	76
Gehman Low Temperature Properties - Cured 35' @ 340°F			
T_5, °F	-	-	-59
G, RT, psi	-	-	185
G, -55°C, psi	-	-	1324

Formula: 100 Polymer, Black level as shown, 6 MgO, 2 Stabilizer, 0.4 Vulcup R.

TABLE VI

EFFECT OF HEAT TREATMENT ON PROCESSING

Stock	R197	-304	-305
Feature		Control	Heat Treated Polymer (1 hr. @ 300°F)
Mixing Evaluation			
Mixing		Fair	Fair
Dump		Good	Good
Dump Time, min.		7-1/2	7-1/2
Milling		Won't band-Lacey	Won't band-Lacey
Calenderable		NO	NO
Physical Properties			
Normal Stress-Strain - Cured 35' @ 340°F			
100% Modulus, psi		900	1040
Tensile, psi		900	1225
Ult. Elongation, %		100	110
Shore "A" Hardness - Cured 40' @ 340°F		59	62

Formula: 100 Polymer as shown, 30 FEF, 6 MgO, 2 Stabilizer, 0.4 Vulcup R.

TABLE VII

EFFECT OF STEARIC ACID ON PROCESSING

Stock	R197	-303	-313	-314
Feature		No S.A. 6 MgO 0 ZnO	2 S.A. 0 MgO 2 ZnO	2 S.A. 6 MgO 2 ZnO
Mixing Evaluation				
Mixing		Good	Good	Good
Dump		Good	Good	Good
Dump Time, min.		9	9	9
Milling		Good	Excellent*	Excellent
Calenderable		Yes	Yes	Yes

*Stock was softer than R197314 probably too soft to build suction hose.

Formula: 100 polymer, 50 FEF, Stearic Acid, MgO, and ZnO as shown.

TABLE VIII

EFFECT OF STEARIC ACID AS A PROCESSING AID

Stock R197	-318	-321	-322	-323
Black level	30	40	40	40
Stearic Acid level	2	2	1	0.5
Physical Properties				
Mix Evaluation				
Mixing	Good	Good	Good	Good
Dump	Good	Good	Good	Good
Milling	Lacey	Lacey	Lacey	Lacey
Calenderable	No	No	No	No
Torque @ Dump (m-gms.)	3000	3750	3500	3500
Normal Stress-Strain - Cured 35' @ 340°F				
100% Modulus, psi	485	875	920	1350
Tensile, psi	1250	1250	1360	1465
Ult. Elongation, %	190	150	150	110
Shore "A" Hardness - Cured 40' @ 340°F	57	67	65	68

Formula: 100 polymer, FEF Black and Stearic Level as shown, 6 MgO, 2 Stabilizer, 0.4 Vulcup R.

TABLE IX

EFFECT OF LOWER PEROXIDE LEVEL ON PHYSICAL PROPERTIES

Stock	R197 -303	-311	-312
Peroxide Level	0.4	0.3	0.2
Physical Properties			
Normal Stress-Strain - Cured 35' @ 340°F			
100% Modulus, psi	1285	1000	760
Tensile, psi	1285	1200	930
Ult. Elongation, %	100	120	130
Shore "A" Hardness - Cured 40' @ 340°F	79	75	73
Gehman Low Temperature Properties - Cured 35' @ 340°F			
T_5, °F	-51	-54	-54
G, RT, psi	215	175	148
G, -55°C, psi	1946	1492	1225

Formula: 100 polymer, 50 FEF, 6 MgO, 2 Stabilizer, Vulcup R as shown.

TABLE X

EFFECT OF SILICA ON PROCESSING

Stock	R197	-348	-349	-350
Quso WR-82 Silica		25	30	35
Mixing Evaluation				
Mixing		Good	Good	Good
Dump		Good	Good	Good
Milling		Lacey	Lacey	Lacey
Calenderable		No	No	No
Torque @ Dump (m-gms.)		2400	2400	2500

Formula: 100 Polymer, Silica as shown, 6 MgO, 2 Stabilizer, 0.4 Vulcup R.

TABLE XI

HEAT TREATED POLYMER

Stock R197	-353	-355	-361	-354	-356
Heat Treatment — in forced air oven	16 hrs. @ 250°F	16 hrs. @ 275°F	16 hrs. @ 290°F	16 hrs. @ 302°F	8½ hrs. @ 302°F
Physical Properties					
Mixing Evaluation					
Mixing	Good	Good	Good	Good	Good
Dump	Good	Good	Good	Good	Good
Dump Time, min.	9	8	9	9	10
Milling	Good*	Good*	Good	Good**	Good
Calenderable	Marginal	Marginal	Yes	Yes	Yes
Viscosity @ Dump (m-gms.)	4250	3250	2850	2750	2800
Normal Stress-Strain - Cured 35' @ 340°F					
100% Modulus, psi	1330	1240	-	-	-
Tensile, psi	1330	1240	880	930	1210
Ult. Elongation, %	100	100	75	80	90
Shore "A" Hardness - Cured 40' @ 340°F	60	63	-	65	64
Gehman Low Temperature Props. - Cured 35' @ 340°F					
T_5, °F	-70	-72	-67	-72	-70
G, RT, psi	85	104	116	131	117
G, -55°C, psi	340	392	587	519	509

Formula: 100 Polymer (heat treated as shown), 30 FEF, 6 MgO, 2 Stabilizer, 0.4 Vulcup R.

* Small Bank on mill, cool rolls.
**Probably too soft to build suction hose.

TABLE XII

STRESS-STRAIN PROPERTIES ON HOSE STOCK

R197	-369-1	-2	-3	-4	-5
Normal Stress-Strain - Cured 35' @ 320°F					
100% Modulus, psi	975	995	1060	930	1000
Tensile, psi	1170	1085	1180	1060	1160
Ult. Elong., %	120	110	110	110	110

Formula: 100 Polymer (Heat treated 8½ hrs. @ 302°F), 30 FEF, 6 MgO, 2 Stabilizer, 0.4 Vulcup R.

These were 5 batches mixed in a Banbury Mixer (Type B) and blended for hose fabrication.

TABLE XIII

DIMENSIONS OF STOCK FOR HOSE FABRICATION

Suction Hose	
Tube	13.625" x .050"
Inner Plies	7.625" x .037" 8.125" x .037"
Cover	16.875" x .050"
Discharge Hose	
Tube	13.375" x .037"
Inner Ply	7.375" x .015"
Cover	15.125" x .037"

TABLE XIV

TEST RESULTS ON SUCTION HOSE

The following tests were performed by American Biltrite on the first suction hose built on November 5, 1975. All tests are compared to standards specified in Purchase Description of this contract and in MIL-H-370C.

Test	Standard	Test Results
Inside Diameter	2" ± 1/16"	2"
Outside Diameter	2.656 ± .062	2.60
Hydrostatic Proof	No Leaks @ 100 psi	No Leaks
125 psi	Max. Twist 7°/ft.	No Twist
	± 3% Length Change	+ 1.31%
Minimum Burst	200 psi Min.	750 psi
Original Tube Tensile Strength	1500 psi Min.	960 psi
Original Tube Elongation	150% Min.	140%
Original Cover Tensile Strength	1500 psi Min.	950 psi
Original Cover Elongation	150% Min.	110%
70 hrs. @ 73°F - Reference Fuel B		
Tube Tensile Strength	600 psi Min.	803 psi
Cover Tensile Strength	600 psi Min.	770 psi
Tube Elongation	100% Min.	100%
Cover Elongation	100% Min.	100%
Adhesions (Original)		
Tube to Ply	1" Max. separation	3/4"
Cover to Ply	Under 10 lb. Load	9/16"
Adhesions (ASTM #3 Oil)		
Tube to Ply	1" Max. separation	3/4"
Cover to Ply	Under 6 lb. Load	7/8"
Volume Increase - 70 hrs. @ 73°F - Reference Fuel B		
Tube	60% Max.	18.8%
Cover	100% Max.	18.8%
Shore "A" Hardness		
Tube	--	56
Cover	--	60

TABLE XIV (CONTINUED)

TEST RESULTS ON SUCTION HOSE

Test	Standard	Test Results
Low Temperature Flexibility		
After 36 hours at -70°F the hose was very flexible		
Existant Gum	Max. 20 MG/100 Ml.	4.2 MG
Test (Tube)		
Crush Resistance	-15% Max. Deformation 95% Min. Recovery	-9.6% 97.4%
Ozone Cover Resistance		
72 hrs. @ 50 PPHM	No cracking 7X Mag.	No. Cracks

The hose manufactured weighed approximately 1.85 lbs./ft.

TABLE XV

PROPERTIES ON STOCK IN FIRST HOSE BUILD

R197	-369

Formula

K18161-302A*	100
FEF Black	30
MgO	6
Stabilizer	2
Vulcup R Peroxide	0.4

Physical Properties

Normal Stress-strain - press cured 35' @ 320°F

100% Modulus, psi	750
Tensile, psi	1120
Ult. Elongation, %	150

Aging in Solvents - press cured (35'/320°F) samples

Aged Stress-strain - 94 hrs. @ 73°F in Type II Fluid**

	Control	Aged	% Retention	Spec
100% Modulus, psi	780	650	--	
Tensile, psi	1110	860	77.5	60
Ult. Elong., %	140	125	89.5	80

Aged Stress-Strain - 14 days @ 73°F in Type II Fluid

	Control	Aged	% Retention	Spec
100% Modulus, psi	880	700	--	
Tensile, psi	1130	840	74.5	60
Ult. Elong., %	140	125	89.5	80

Aged Stress-strain - 14 days in distilled H_2O @ 160°F

	Control	Aged	% Retention	Spec
100% Modulus, psi	910	770	--	
Tensile, psi	1100	940	85.5	80
Ult. Elongation, %	120	140	117	80

* K18161 heated 8 1/2 hrs. @ 302°F.

** A control from the same slab was tested with each aged sample.

TABLE XV (CONTINUED)

PROPERTIES ON STOCK IN FIRST HOSE BUILD

R197 -369

Volume Change

Type II Solvent	Sample	Spec
94 hrs. @ 73°F, % change	19.8	40
14 days @ 73°F, % change	18.5	40
Distilled H_2O		
14 days @ 160°F, % change	10.9	15
42 days @ 160°F, % change	Not Completed	

Weight change

Type II Solvent	Sample	Spec
94 hrs. @ 73°F, % change	2.1	5
14 days @ 73°F, % change	1.1	5
Distilled H_2O		
14 days @ 160°F, % change	-1.2	5
42 days @ 160°F, % change	Not Completed	

TABLE XVI

EVALUATION OF LOWER PEROXIDE LEVELS

Stock R197	-356	-362	-363
Peroxide Level	0.4	0.3	0.2
Physical Properties			
Normal Stress-Strain - Cured @ 320°F			
100% Modulus, psi			
25' cure	980	700	500
35' cure	1050	850	675
Tensile, psi			
25' cure	1080	1120	1050
35' cure	1110	1190	1100
Ult. Elongation, %			
25' cure	120	150	200
35' cure	110	150	170
Shore "A" Hardness - Cured 40' @ 340°F	64	59	54
Compression Set @ RT - Cured 40' @ 340°F			
70 hrs, % Set	16.8	20.0	23.2
Trouser Tear @ RT - Cured 35' @ 320°F			
lbs./in.	11	8	12
Gehman Low Temperature Properties - Cured 35' @ 320°F			
T_5, °F	-65	-69	-67
G @ RT, psi	99.0	96.0	82.8
G @ -55°C, psi	524.5	406.0	408.4

Recipe: 100 Heat treated polymer, 30 FEF, 6 MgO, 2 Stabilizer, Vulcup R peroxide as shown.

TABLE XVII

EVALUATION OF DIFFERENT PEROXIDE TYPES

R197	-369	-370	-371	-372
Peroxide Type				
Vulcup R	0.4	-	-	-
Dicup 40C	-	1.6	-	-
Vulcup 40KE	-	-	1.0	-
Luperco 230XL	-	-	-	1.7
Physical Properties				
Monsanto Rheometer @ 320°F, 1° Arc, 100 RPM				
Scorch (2 unit rise) min.	6.0	5.9	6.8	3.2
90% Cure, min.	38.0	24.3	39.8	11.7
Torque (min.), dn·m	9.8	9.1	9.1	10.9
Torque (90% Cure), dn·m	20.6	18.1	19.9	18.9
Torque (100% Cure), dn·m	21.8	19.1	21.1	19.8
Normal Stress-Strain - Cured 35' @ 320°F				
100% Modulus, psi	770	465	740	800
Tensile, psi	1260	1150	1170	1210
Ult. Elong., %	150	160	140	140
Trouser Tear @ RT - Cured 35' @ 320°F				
lbs./in.	11	11	11	10
Gehman Low Temp. Properties - Cured 35' @ 320°F				
T_5, °F	-	-67	-71	-71
G @ RT, psi	-	85.9	82.9	77.5
G @ -55°C, psi	-	390.8	320.5	273.9

Recipe: 100 Heat treated polymer, 30 FEF, 6 MgO, 2 Stabilizer, Peroxide as shown.

TABLE XVIII

EVALUATION OF SILICONE AS AN ADDITIVE IN COVER STOCK

Stock R197	-356	-379	-380	-381
Polymer System				
K18161-302A*	100	95	92.5	90
Silicone**	-	5	7.5	10
Physical Properties				
Mixing and Processing Evaluation				
Mixing	Good	Good	Good	Good
Dump	Good	Good	Good	Good
Dump Time, min.	8	9	8	8
Milling	Good	Won't Band	Won't Band	Won't Band
Calenderable	Yes	No	No	No
Normal Stress-Strain - Cured 35' @ 300°F				
100% Modulus, psi	1150	870	800	820
Tensile, psi	1310	1075	975	1010
Ult. Elongation, %	120	120	120	120
Shore "A" Hardness - Cured 35' @ 320°F	63	58	57	59

* Heat treated K18161, 8½ hrs. @ 302°F

**Union Carbide W-982 Silicone Rubber.

Recipe: Polymer as shown, 30 FEF, 6 MgO, 2 Stabilizer, 0.4 Vulcup R.

TABLE XIX

EVALUATION OF POLYBUTADIENE AND EPDM AS ADDITIVES FOR THE COVER STOCK

R197	-356	-391	-392	-393	-394
Polymer System					
K18161-302A	100	95	90	95	90
EPDM	-	5	10	-	-
Polybutadiene	-	-	-	5	10
Physical Properties					
Mixing	Good	Good	Good	Good	Good
Dump	Good	Good	Good	Good	Good
Dump Time, min.	7	10	6	8	8
Milling	Good	Good	Fair	Good	Good
Calenderable	Yes	Yes	Marginal	Yes	Yes
Normal Stress-Strain - Cured 35' @ 320°F					
100% Modulus, psi	1130	1000	1000	-	-
Tensile, psi	1320	1075	1120	1050	1020
Ult. Elongation, %	120	110	105	85	60

Recipe: Polymer as shown, 30 FEF, 6 MgO, 2 Stabilizer, 0.4 Vulcup R

TABLE XX

FUEL DIFFUSION RATIO OF EPDM COVER STOCK

Stock	R197	-356	-391	-392
Polymer System				
K18161-302A		100	95	90
EPDM		-	5	10
Physical Properties				
Mix Evaluation				
Mixing		Good	Good	Good
Dump		Good	Good	Good
Dump Time, min.		7	10	6
Milling		Good	Good	Fair
Calenderable		Yes	Yes	Marginal
Monsanto Rheometer @ 320°F, 1°Arc, 100 RPM				
Scorch (minutes)		7.1	5.5	5.3
90% Cure (minutes)		46.0	44.0	45.3
Torque (Min.), dN·m		10.0	11.0	11.0
Torque (90%), dN·m		22.1	29.0	30.8
Torque (Max.), dN·m		23.4	31.0	33.0
Normal Stress-Strain - Cured 35' @ 320°F				
100% Modulus, psi		1130	1000	1000
Tensile, psi		1320	1075	1120
Ult. Elongation, %		120	110	105
Fuel Diffusion Rate				
Rate - fl. oz. ft.$^{-2}$ .24 hrs.$^{-1}$		0.94	1.87	2.87
Diffusion Ratio		-	1.99	3.05

Recipe: Polymer as shown, 30 FEF, 6 MgO, 2 Stabilizer, 0.4 Vulcup R.

TABLE XXI

Cover Stocks for Optimum
Fuel Diffusion Rate Ratio

Stock	R197363	R198625	R198626	R198627
Polymer				
K18161-302A	100.0	97.5	97.5	95.0
EPDM	--	2.5	--	--
K18315[1]	--	--	2.5	5.0
Mix Evaluation				
Mixing	Good	Good	Good	Good
Dump	Good	Good	Good	Good
Milling	Good	Good	Good	Good
Calenderable	Yes	Yes	Yes	Yes
Monsanto Rheometer				
(@ 320°F, 1° Arc, 100 RPM)				
Scorch (min.)	11.3	12.6	12.8	17.8
90% Cure (min.)	50.3	45.8	44.8	44.3
Torque (min.), dN · m	7.8	7.9	7.8	6.8
Torque (90%), dN · m	13.1	13.2	12.4	10.7
Torque (max.), dN · m	13.7	13.8	12.9	11.1
Normal Stress-Strain				
(cure: 320°F)				
100% M, psi				
35'	560	580	451	377
45'	563	616	452	478
Tensile, psi				
35'	1020	1000	852	738
45'	1038	1038	853	768
Ult. Elong., %				
35'	160	155	198	205
45'	135	150	190	185
Fuel Diffusion Rate Ratio				
	Tube	1.42	1.36	1.74

1. A non-fluorinated polyalkoxyphosphazene.

Recipe: Polymer as shown, 30FEF, 6 MgO, 2 stabilizer, 0.2 Vulcup R.

TABLE XXII

EVALUATION OF TEFLON 8A AND ITS EFFECT ON TEAR RESISTANCE

R197	-356	-382	-383	-384	-385
Teflon 8A	0	2.5	5	7.5	10
Physical Properties					
Mix and Processing Evaluation					
Mixing	Good	Good	Good	Good	Good
Dump	Good	Good	Good	Good	Good
Dump Time, min.	-	8	7	7	5
Milling	Good	Good	Fair	Fair	Fair
Calenderable	Yes	Yes	No	No	No
Normal Stress-Strain - Cured 35' @ 320°F					
100% Modulus, psi	1100	1370	1790	-	-
Tensile, psi	1170	1490	1790	1750	1970
Ult. Elongation, %	110	120	100	80	80
Shore "A" Hardness - Cured 35' @ 320°F	58	63	70	71	68
Trouser Tear @ RT - Cured 35' @ 320°F					
lbs./in.	7	15	36	59	56
Crescent Tear (Die B) @ RT - Cured 35' @ 320°F					
lbs./in.	54	128	203	266	313

Recipe: 100 Heat treated polymer, 30 FEF, 6 MgO, Teflon 8A as shown, 2 Stabilizer, 0.4 Vulcup R.

TABLE XXIII

Evaluation of Teflon-8A (Added in Brabender)

Stock R199	-417	-418	-419
Teflon-8A (phr)	0	2	5
Mix Evaluation			
Mixing	Good	Good	Good
Dump	Good	Good	Good
Milling	Good	Good	Fair
Calenderable	Yes	Yes	Maybe
Normal Stress-Strain (cure: 320°F)			
100% M, psi			
35'	784	948	1201
45'	784	1013	1201
Tensile, psi			
35'	1126	1244	1263
45'	1094	1162	1400
Ult. Elong., %			
35'	150	140	110
	145	125	135
Trouser Tear @ R. T. (cure: 40' @ 320°F)	12.3	34.4	93.8
Shore "A" Hardness (73°F) (cure: 40' @ 320°F)	52.5	64.0	63.0
Compression Set 70 hrs. @ R. T. (cure: 40' @ 320°F)	17.6	20.0	27.7

Recipe: 100 polymer (K18161-302A), 30 FEF, 6 MgO, 2 stabilizer, 0.2 Vulcup R, Teflon as shown.

TABLE XXIV

Evaluation of Teflon-8A and Silicone Oil

Stock R199	-422	-423	-424
Teflon-8A[1]	0	2.0	5.0
Silicone Oil[1]	0	2.0	5.0
Mix Evaluation			
Mixing	Good	Good	Good
Dump	Good	Good	Good
Milling	Good	Good	Won't Band
Calenderable	Yes	Yes	No
Normal Stress-Strain			
(cure: 320°F)			
100% M, psi			
35'	788	900	816
45'	904	853	941
Tensile, psi			
35'	1085	1204	995
45'	1021	1123	941
Ult. Elong., psi			
35'	140	195	150
45'	120	185	100
Shore "A" Hardness (73°F) (cure: 40' @ 320°F)	52.0	63.0	67.0
Trouser Tear @ R. T. (cure: 40' @ 320°F)	11	49	155

[1] Dow Corning Fluid (710)

Recipe: 100 polymer (K18161-302A), 30 FEF, 6 MgO, 2 stabilizer, 0.2 Vulcup R, Teflon 8-A and silicone oil as shown.

TABLE XXV

EVALUATION OF QUSO WR-82 SILICA AS A FILLER

Stock	R198	-603	-604	-605	-606
Quso WR-82 Silica, phr		20	25	30	35
Physical Properties					
Mix Evaluation					
Mixing		Fair	Fair	Fair	Fair
Dump		Loose Powder on all Stocks			
Dump Time, min.		10	10	10	10
Milling		Sticky	Good	Good	Good
Calenderable		No	Yes	Yes	Yes
Monsanto Rheometer @ 320°F, 1°Arc, 100 RPM					
Scorch (minutes)		5.0	3.9	4.0	4.0
90% Cure (minutes)		37.6	37.8	38.8	38.8
Torque (minutes), dN·m		5.3	6.1	6.0	6.5
Torque (90%), dN·m		15.7	22.2	23.5	26.7
Torque (Max), dN·m		16.8	24.0	25.4	28.1
Normal Stress-Strain - Cured 35' @ 320°F					
100% Modulus, psi		-	-	-	-
Tensile, psi		840	960	810	820
Ult. Elong., %		90	90	70	60
Trouser Tear @ RT - Cured 35' @ 320°F					
lbs/in		3	4	5	-

Recipe: 100 K18161-302A, Filler as shown, 6 MgO, 2 Stabilizer, 0.4 Vulcup R

TABLE XXVI

EVALUATION OF HI SIL SILICA AS A FILLER

Stock R198	-608	-609	-610	-611
Hi Sil 233, phr	20	25	30	35
Physical Properties				
Mixing Evaluation				
Mixing	Good	Good	Good	Good
Dump	Good	Good	Good	Good
Dump Time, minutes	10	12	12	12
Milling	Sticky	Sticky	Fair	Good
Calenderable	No	No	No	Yes
Monsanto Rheometer @ 320°F, 1°Arc, 100 RPM				
Scorch, (minutes)	4.7	5.6	5.3	4.3
90% Cure, (minutes)	32.5	35.7	25.5	15.1
Torque (min.), dN·m	10.8	10.4	16.8	32.1
Torque (90%), dN·m	23.9	25.2	36.4	68.9
Torque (Max), dN·m	25.4	26.8	38.6	73.0
Normal Stress-Strain - Cured 35' @ 320°F				
100% Modulus, psi	-	-	-	-
Tensile, psi	500	560	740	880
Ult. Elong., %	60	50	60	50
Shore "A" Hardness	63	68	78	82
Trouser Tear @ RT - Cured 35' @ 320°F				
lbs/in	7	9	8	12

Recipe: 100 K18161-302A, Filler as shown, 6 MgO, 2 Stabilizer, 0.4 Vulcup R

TABLE XXVII

ADHESION OF STOCK TO RAYON

Stock	R197	-369
T-Adhesion to Rayon Used In Hose - Cured 35' @ 320°F - Tested @ RT		
Beaver Rayon		
lbs/in		20
% Coverage		0
Beaunit Rayon		
lbs/in		18
% Coverage		0

Recipe: 100 K18161-302A, 30 FEF, 6 MgO, 2 Stabilizer, 0.4 Vulcup R

TABLE XXVIII

Evaluation of Potential Promoters of Adhesion of Rayon to PNF®-200

Stock	R197363	R198615	R198616	R198617	R198618	R198619
Additive	None	Cohedur RL	Resorcinol + Hexa	Cymel 301	Resorcinol + Cymel 301	Manobond C
Normal Stress-Strain						
(cure: 35' @ 320°F)						
100% M, psi	693	859	--	471	530	533
Tensile, psi	1070	1067	800	651	530	777
Ult. Elong., %	165	135	85	155	100	190
T-Adhesion @ R. T. to Beaver Rayon;						
(cure: 35' @ 320°F)						
lbs./in	16	15	15	13	27	8
% coverage	0	0	0	0	0	0

Recipe: 100 K18161-302A, 30 FEF, 6 MgO, 2 Stabilizer, 0.2 Vulcup R, all additives were used at 2.0 phr (for combinations, total additive = 4.0 phr).

TABLE XXIX

Evaluation of Various Blacks with Heat-Treated Polymer

Stock R199	-400	-401	-402	-403	-404
Black	FEF	HAF(LS)	HAF	SAF	GPF
Mix Evaluation					
Mixing	Fair	Fair	Fair	Fair	Good
Dump	Good	Good	Good	Good	Good
Milling	Good	Fair	Good	V. Good	Fair
Calenderable	Yes	Maybe	Yes	Yes	Maybe
Normal Stress-Strain					
(cure: 35' @ 320°F)					
100% M, psi	554	740	743	519	512
Tensile, psi	1200	1184	1512	1257	1144
Ult. Elong., %	185	130	155	205	180
Shore "A" Hardness (73°F) (cure: 40' @ 320°F)	47.5	50.5	54.5	61.0	40.5
Compression Set					
70 hrs. @ R. T.	16.8	19.2	19.2	34.9	16.0
(cure: 40' @ 320°F)					
Trouser Tear @ R. T. (cure: 35' @ 320°F)	16.8	10.9	9.1	34.1	10.4
Gehman Low Temp. Properties -- cure: 35' @ 320°F					
T_5 °F	-77	-70	-67	-58	-73
G @ R. T., psi	84.9	72.4	93.4	122.9	60.5
G @ -55°C, psi	198.9	254.9	386.2	1006	186.1

Recipe: 100 polymer (K18161-302A), 30 black, 6 MgO, 2 stabilizer, 0.2 Vulcup R for -400 and -404, 0.5 Vulcup R for -401, -402, -403.

TABLE XXX

Stocks Used for Second Hose Building Trial

Stock R199415	-1	-2	-3	R199416	-1	-2	-3
Polymer							
K18161-302A	100.0	100.0	100.0		97.5	97.5	97.5
EPDM	--	--	--		2.5	2.5	2.5
Mix Evaluation							
Banbury Mixing	Good	Good	Good		Good	Good	Good
Dump condition	Good	Good	Good		Good	Good	Good
Dump time/temp. °F	8'/302	8'/305	8'/310		8'/305	8'/305	8'/305
Milling	Good	Good	Good		Good	Good	Good
Calenderable	Yes	Yes	Yes		Yes	Yes	Yes
Normal Stress-Strain							
(cure: 35' @ 320°F)							
100% M, psi	964	891	970		850	851	722
Tensile, psi	1156	1157	1175		1102	1087	1059
Ult. Elong., %	130	140	130		140	140	160

R199415-1, -2, -3 mill blended-tube stock
R199416-1, -2, -3 mill blended-cover stock
} Polymers as shown above, 30 FEF, 6 MgO, 2 stabilizer, 0.2 Vulcup R.

TABLE XXXI

Stress-Strain Properties on Second Hose

Stock	R199415 (Tube)	R199416 (Cover)
Stress-Strain Measured at American Biltrite - cure: 90' @ 325°F		
Tensile, psi	488	690
Ult. Elong. %	235	200
Stress-Strain Measured at Firestone (on cover)		
100% M, psi		531
Tensile, psi		790
Ult. Elong., %		165

Specimens were cut from the hose and buffed.

Table XXXII

Cure Checks on Calendered
Stocks used in Second Hose Building

Stock	R199416 (cover)
Stress-strain (steam cure - 320°F)	
100% M, psi	
45'	522
60'	553
90'	540
Tensile, psi	
45'	851
60'	873
90'	799
Ult. Elong., %	
45'	200
60'	170
90'	160
(press cure - 45' @ 320°F)	
100% M, psi	487
Tensile, psi	937
Ult. Elong., %	205

Table XXXIII

Effect of Calendering on Stress-Strain Properties

	Stock - Treatment	100% M, psi	Tensile, psi	Ult. Elongation, %
1.	R199444 - no calendering	879	1208	145
2.	R199444 - calendered at 130-150°F - two passes	692	1173	170
3.	R199444 - calendered at 130-150°F - several passes	763	1157	165
4.	R199444 - calendered at 180-200°F - two passes	704	1013	145
5.	R199444 - calendered at 170-200°F - several passes	600	814	145

All of the above stocks were press-cured at 320°F for 35 min.

R199444 Recipe: 100 polymer (K18352), 30 FEF, 6 MgO, 2 stabilizer, 0.5 Vulcup 40KE.

TABLE XXXIV

Use of Vulcup 40KE in Place of Vulcup R

Stock R199	-408	-409	-410	-411
Peroxide				
Vulcup R	0.2	0.3	--	--
Vulcup 40KE	--	--	0.5	0.75
Normal Stress-Strain				
(cure: 320°F)				
100% M, psi				
25'	750	1168	651	1060
35'	728	1132	667	930
45'	770	1284	730	--
Tensile, psi				
25'	1106	1266	1100	1291
35'	1111	1132	1006	1254
45'	1197	1284	1107	1020
Ult. Elong., %				
25'	170	110	190	130
35'	170	100	160	135
45'	165	100	165	80

Recipe: 100 polymer (K18161-302A), 30 FEF, 6 MgO, 2 stabilizer, peroxide as shown.

TABLE XXXV

Evaluate Polymers with Reduced Heat Aging Times

DSV vs. Aging Time	Time @ 300°F	DSV	% Gel
	0	3.50	0.0
	2 hrs.	2.66	0.0
	4 hrs.	2.13	0.0
	6 hrs.	1.76	0.0
	8.5 hrs.	1.51	0.0
Stock R199	-412	-413	-414
Polymer	K18161-302A	K18352	K18353
300°F Aging Time	8.5 hrs.	6.0 hrs.	4.5 hrs.
Mix Evaluation			
Mixing	Good	Good	Good
Dump	Good	Good	Good
Milling	Good	Good	Fair
Calenderable	Yes	Yes	Probably
Normal Stress-Strain			
(cure: 320°F)			
100% M, psi			
35'	643	812	787
45'	734	761	880
Tensile, psi			
35'	1010	1184	1208
45'	1021	1150	1213
Ult. Elong., %			
35'	165	150	155
45'	145	150	140
Trouser Tear @ R. T. (cure: 40' @ 320°F)	16.2	14.4	13.2
Gehman Low Temp. Properties			
T_5 °F	-70	-67	-70
G @ R. T., psi	86.6	82.6	88.0
G @ -55°C, psi	303.4	357.9	331.3

Recipe: 100 polymer, 30 FEF, 6 MgO, 2 stabilizer, 0.2 Vulcup R

Table XXXVI

Evaluation of SAF-Austin Black Combinations

Stock - R199	-425	-426	-427	-428
Black	30 FEF	10 SAF 20 Austin	15 SAF 15 Austin	10 SAF 30 Austin
Mix Evaluation				
Brabender mixing	good	good	good	good
Dump	good	sticky	sticky	sticky
Milling	good	bands both rolls	bands both rolls	bands both rolls
Calenderable	yes	no	no	no
Normal Stress-strain - cure: 320°F				
100°M, psi				
35'	862	468	597	476
45'	825	538	615	483
Tensile, psi				
35'	1092	637	894	613
45'	1091	706	835	624
Ult. Elong., %				
35'	140	155	170	170
45'	140	150	150	170
% Compression Set (73°F) (25%/70 hrs.) - cure: 40' @ 320°F				
	16.8	18.4	20.0	18.4
Shore "A" Hardness (73°F) - on compression set buttons				
	52.5	45.5	50.0	52.0
Trouser Tear (73°F)- cure: 40' @ 320°F				
lbs./in.	12.5	11.8	12.7	11.1
Gehman Low Temp. Properties - cure: 40' @ 320°F				
T_5, °F	-62.0	-67.0	-62.5	-65.0
G @ R.T., psi	79.0	67.8	72.0	63.7
G @ -55°C, psi	566	353	546	388

Recipe: 100 polymer (K18161-302A), black - as shown, 6 MgO, 2 stabilizer, Vulcup 40KE - 0.5 to 1.0 (higher for higher SAF).

Table XXXVII

Evaluation of SAF-FEF Black Combinations

Stock - R199	-429	-430	-431	-432
Black	30 FEF	15 FEF	20 FEF	10 FEF
		5 SAF	5 SAF	10 SAF
Mix Evaluation				
Brabender Mixing	good	good	good	good
Dump	good	good	good	good
Milling	good	sticks both rolls	sticks both rolls	sticks both rolls
Calenderable	yes	no	no	no
Normal Stress-Strain - cure:	320°F			
100% M, psi				
35'	560	464	631	--
45'	585	564	644	628
Tensile, psi				
35'	1129	1296	1204	656
45'	1189	1252	1256	836
Ult. Elong., %				
35'	185	210	165	100
45'	190	180	165	120
% Compression Set (73°F) (25%/70 hrs.) - cure: 40' @ 320°F				
	30.0	20.8	20.8	21.6
Shore "A" Hardness (73°F) - on compression set buttons				
	49.0	41.0	45.0	48.5
Trouser Tear (73°F) - cure: 40' @ 320°F				
lbs./in.	28	11	9	8
Gehman Low Temp. Properties - cure: 40' @ 320°F				
T_5, °F	-71.5	-73.0	-71.0	-70.0
G @ R.T., psi	81.7	62.3	53.8	60.1
G @ -55°C, psi	301	216	207	283

Recipe: 100 polymer (K18352), black - as shown, 6 MgO, 2 stabilizer, Vulcup 40 KE - 0.5 to 1.0 (higher for higher SAF)

Table XXXVIII

Evaluation of ISAF and ISAF-FEF Black Combinations

Stock - R199	-455	-456	-457	-458
Black	25 ISAF (HS)	25 ISAF	15 ISAF 10 FEF	10 ISAF 15 FEF
Vulcup 40KE	1.25	1.25	1.0	1.0
Mix Evaluation				
Brabender Mixing	good	good	good	good
Dump	good	good	good	good
Milling	good	good	good	good
Calenderable	yes	yes	probably	probably
Normal Stress-Strain - cure: 320°F				
100% M, psi				
35'	1256	1106	1056	1167
45'	1282	1073	1086	1137
Tensile, psi				
35'	1486	1526	1358	1359
45'	1578	1467	1391	1203
Ult. Elong., %				
35'	120	135	135	120
45'	120	135	130	110
% Compression Set (73°F) (25%/70 hrs.) - cure: 40' @ 320°F				
	14.4	16.8	13.6	12.0
Shore "A" Hardness (73°F) - on compression set buttons				
	55.0	55.0	51.0	51.0
Trouser Tear (73°F) - cure: 40' @ 320°F				
lbs./in.	8	11	11	7
Gehman Low Temp. Properties - cure: 40' @ 320°F				
T_5, °F	-65.0	-65.0	-69.0	-69.0
G @ R.T., psi	90.8	105.3	79.5	47.9
G @ -55°C, psi	540	643	346	203

Recipe: 100 polymer (K18352), black - as shown, 6 MgO, 2 stabilizer, Vulcup 40KE as shown.

Table XXXIX

Evaluation of Degussa Blacks

Stock - R199	-433	-434
Black	Printex 60	RUB Corex P
Mix Evaluation		
Brabender mixing	good	good
Dump	good	good
Milling	good (@ 80-100°F)	good (@ 80-100°F)
Calenderable	yes	yes
Normal Stress-Strain - cure: 320°F		
100% M, psi		
35'	484	557
45'	588	618
Tensile, psi		
35'	1256	1296
45'	1296	1305
Ult. Elong., %		
35'	225	225
45'	215	210
% Compression Set (73°F) (25%/70 hrs.), cure: 40' @ 320°F	35.7	36.7
Shore "A" Hardness (73°F) - cure: 40' @ 320°F	57.0	58.0
Trouser Tear (73°F) - cure: 40' @ 320°F		
lbs./in.	27	42
Gehman Low Temp. Properties - cure: 40' @ 320°F		
T_5, °F	-62.5	-62.0
G @ R.T., psi	106.7	107.7
G @ -55°C, psi	756	675

Recipe: 100 polymer (K18352), 30 black, 6 MgO, 2 stabilizer, 0.75 Vulcup 40 KE.

AD-A036 903 FIRESTONE TIRE AND RUBBER CO AKRON OHIO CENTRAL RESE--ETC F/G 13/11
FABRICATION OF A LOW TEMPERATURE FUEL HOSE FROM PHOSPHONITRILIC--ETC(U)
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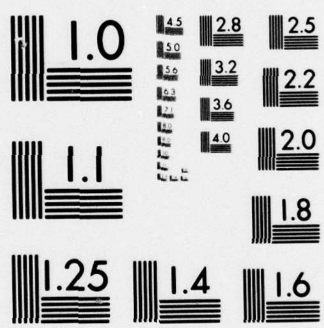

MICROCOPY RESOLUTION TEST CHART
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TABLE XL

EVALUATION OF QUSO WR-82 AT LOWER PEROXIDE LEVELS

Stock R199	-439	-440	-441
Vulcup 40KE	0.75	0.50	0.25
Normal Stress-Strain - cure: 320°F			
100% M, psi			
35'	935	642	399
45'	957	658	398
Tensile, psi			
35'	935	969	860
45'	1026	895	846
Ult. Elong., %			
35'	110	165	270
45'	110	155	255

Recipe: 100 - polymer (K18161-302A), 30 - Quso WR-82, MgO - 6, stabilizer - 2, Vulcup - as shown.

Table XLI

Evaluation of Adhesion of Hose Compounds to Various Fabrics

Stock - R199	-420	-421
K18161-302A	100.0	97.5
EPDM	--	2.5
FEF	30.0	30.0
MgO	6.0	6.0
Stabilizer	2.0	2.0
Vulcup R	0.2	0.2
	138.2	138.2
Normal Stress-Strain - cure: 35' @ 320°F		
100% M, psi	781	762
Tensile, psi	876	1102
Ult. Elong., %	110	165
T-adhesion @ R.T. (lbs./in.) - cure: 45' @ 320°F		
Nylon (treated)	13	12
Nylon (untreated)	7	6
Rayon (treated)	10	11
Rayon (untreated)	7	9
Polyester (treated)	10	10
Polyester (untreated)	8	8

Table XLII

Cure Checks on Compounds for Third Hose Building Trial

Stock - R199	-437	-437	-462	-463	-463	-463
Polymer						
K18352	100.0	100.0	97.5	100.0	100.0	100.0
EPDM	--	--	2.5	--	--	--
Peroxide						
Vulcup R	0.3	0.3	--	--	--	--
Vulcup 40 KE	--	--	0.5	0.6	0.6	0.6
Cure (320°F)	press (35')	steam (45')	press (35')	press (35')	steam (40')	press (35')
Normal Stress-Strain						
100% M, psi	1237	1188	624	826	604	578
Tensile, psi	1343	1188	1096	1270	1067	1091
Ult. Elong., %	110	100	180	165	175	195
					after calendering	after calendering

Recipe: polymer - as shown, FEF - 30, MgO - 6, stabilizer - 2, peroxide - as shown.

TABLE XLIII

VARIOUS TEST RESULTS ON THIRD HOSE COMPOUNDS

Stock R199	-464 (Tube)		-465 (Cover)	
Formulation				
Polymer K18352	100.0		97.5	
EPDM	--		2.5	
FEF	30.0		30.0	
MgO	6.0		6.0	
Stabilizer	2.0		2.0	
Vulcup 40KE	0.7		0.7	
	138.7		138.7	
Normal Stress-Strain - cure: 60' @ 320°F in steam - specimens cut from hose				
	collapsible	suction	collapsible	suction
100% M, psi	424	493	580	688
Tensile, psi	871	981	953	1016
Ult. Elong., %	197	193	170	153
Normal Stress-Strain - on excess calendered stock (after hose building) cure: press, 60' @ 320°F				
100% M, psi	773		724	
Tensile, psi	1184		1179	
Ult. Elong., %	155		165	
Gehman Low Temp. Properties - press cure: 60' @ 320°F				
T_5, °F	-73		-60	
G @ RT, psi	123.9		101.9	
G @ -55°C, psi	442.6		709.5	
Tg, °C	-76		-76	
Low Temp. Testing @ MERDC (after one day @ -70°F)- additional tests in Appendix				
TSR	3.2		4.4	
G @ RT, psi	81		105	
G @ -70°F, psi	250		462	
% Tension Recovery	45		40	
Compression Set	59.6		62.6	
Trouser Tear (73°F) - press cure: 60' @ 320°F				
lbs./in.	11		14.5	
T-adhesion to Rayon - press cure: 60' @ 320°F				
lbs./in.	13		17	

Table XLIV

Test Results on Third Hose

Test		Results: Suction	Results: Discharge
Inside Diameter		2"	2"
Outside Diameter		2.70"	2.47"
Hydrostatic Proof		no leaks	no leaks
125 psi		5° twist, 1.82% length Δ	--
100 psi		--	no twist, 0.53% length Δ
Minimum Burst		850 psi	750 psi
Adhesions (10# load, 1 min.)	tube to ply	0.312" separation	1.75" separation
	cover to ply	0.187" separation	0.175" separation

All tests were done on hose or sections cut from hose.

TABLE XLV

FUEL AND WATER RESISTANCE OF STOCKS IN THIRD HOSE BUILDING TRIAL

Stock	R199464 (Tube)		R199465 (Cover)	
Immersed in Type II Fluid (TT-S-735) @ 73°F for	94 hrs.	14 days	94 hrs.	14 days
Tensile retained, %	93.0	93.8	80.4	105
Stress (100% E) retained, %	93.8	102	83.9	107
Ult. Elong. retained, %	102	91.3	94.1	97.5
Vol. increase, %	9.6	22.1	20.2	32.3
Wt. decrease, %	0.94	1.07	1.03	0.91
Immersed in Distilled Water (pH = 7) @ 160°F for	14 days	42 days	14 days	42 days
Tensile retained, %	78.7	75.5	86.8	86.4
Stress (100% E) retained, %	84.1	78.8	81.8	82.5
Ult. Elong. retained, %	93.3	81.6	97.5	103
Vol. increase, %	13.2	18.4	10.6	15.1
Wt. decrease, %	1.49	1.57	1.56	1.64

Table XLVI

Poly-Bd as Additive for Cover Stock

Stock - R199	-473	-474	-475
Polymer			
K18161-302B	100.0	98.0	96.0
Poly-Bd	--	2.0	4.0
Vulcup 40 KE	0.6	0.5	0.5
Normal Stress-strain - cure: 35' @ 320°F			
100% M, psi	871	758	717
Tensile, psi	1166	1125	1044
Ult. Elong., %	145	165	160
Fuel Diffusion Rate Ratio (cover/tube) - cure: 35' @ 320°F			
	tube	1.62	1.58
Gehman Low Temp. Properties - cure: 40' @ 320°F			
T_5, °F	-69.7	-68.8	-70.6
G @ R.T., psi	79.7	117	138
G @ -55°C, psi	309	534	591

Recipe: Polymer - as shown, 30 FEF black, 6 MgO, 2 stabilizer, peroxide - as shown

TABLE XLVII

EVALUATION OF GPF (HS) AND ISAF (N234)

Stock R199	-466	-467	-468
Black	FEF	GPF (HS)	ISAF (N234)
Mix Evaluation			
Brabender mixing	good	good	good
Dump	good	good	good
Milling	good	good	good
Calenderable	yes	yes	yes
Normal Stress-Strain - cure: 320°F			
100% M, psi			
35'	1059	1155	752
45'	1157	1054	806
Tensile, psi			
35'	1242	1155	1465
45'	1294	1182	1422
Ult. Elong., %			
35'	135	100	225
45'	120	125	200
% Compression Set (73°F) (25%/70 hrs.) - cure: 40' @ 320°F	15.2	15.2	43.8
Shore "A" Hardness (73°F) - on compression set button	53.0	54.0	62.0
Trouser Tear (73°F) - lbs./in.	20	9	72
Gehman Low Temp. Properties - cure: 40' @ 320°F			
T_5, °F	-67.9	-67.9	-51.6
G @ RT, psi	74	79	134
G @ -55°C, psi	293	311	1373

Recipe: 100 - polymer (K18161-302B), 30 - Black, 6 - MgO, 2 - stabilizer, 0.6 - Vulcup 40KE.

TABLE XLVIII

EVALUATION OF ISAF-FEF COMBINATIONS

Stock R199	-478	-479	-480	-481	-482
Black					
ISAF (HS)	10.0	15.0	--	--	--
ISAF	--	--	10.0	15.0	--
FEF	15.0	10.0	15.0	10.0	30.0
Normal Stress-Strain - cure: 320°F					
100% M, psi					
35'	1255	1155	1077	1021	1223
60'	1260	1319	1156	1014	1290
90'	1262	1270	1198	1158	1181
Tensile, psi					
35'	1307	1414	1258	1333	1301
60'	1260	1319	1256	1286	1330
90'	1262	1325	1296	1309	1223
Ult. Elong., %					
35'	110	125	125	130	110
60'	100	105	115	130	105
90'	100	110	115	120	110
Trouser Tear (73°F) - cure: 45' @ 320°F					
lbs./in.	9.2	9.3	9.5	10.6	11.1
Gehman Low Temp. Properties - cure: 45' @ 320°F					
T_5, °F	-82.3	-78.7	-79.6	-81.4	-80.5
G @ RT, psi	79	85	69	76	88
G @ -55°C, psi	186	251	183	187	251

Recipe: 100 - polymer (K18161-302A), black - as shown, 6 - MgO, 2 - stabilizer, 1.0 - Vulcup 40KE except for 482 (0.75).

TABLE IL

EVALUATION OF ADJUSTED HAF. ISAF (HS), RUB COREX P COMPOUNDS

Stock R199	-486	-487	-488
Black	30 HAF	25 ISAF (HS)	27 Rub Corex P
Vulcup 40KE	1.2	1.1	1.0
Mix Evaluation			
Brabender Mix	good	good	good
Dump Condition	good	good	good
Milling*	fair	fair	fair
Calenderable	yes	yes	yes

* Rating given for 130°F mill temperature. With these blacks, milling was better at 100°F.

Normal Stress-Strain - cure: 320°F			
100% M, psi			
35'	882	814	806
60'	984	970	946
Tensile, psi			
35'	1496	1392	1348
60'	1332	1346	1284
Ult. Elong., %			
35'	155	160	155
60'	130	130	135
% Compression Set (73°F) - 70 hrs./25%, cure: 40' @ 320°F	8.8	8.0	9.6
Shore "A" Hardness (73°F) - on compression set button	58.0	56.0	59.0
Trouser Tear (73°F) - cure: 40' @ 320°F			
lbs./in.	13.6	11.5	12.5
Gehman Low Temp. Properties - cure: 40' @ 320°F			
T_5, °F	-46.3	-62.5	-58.9
G @ RT, psi	83	101	94
G @ -55°C, psi	780	690	668

Recipe: 100 - polymer (K18161-302A), black - as shown, 6 - MgO, 2 - stabilizer, Vulcup 40KE - as shown.

TABLE L

EVALUATION OF SHAWINIGAN BLACK

Stock	R199489
Mixing Evaluation	
Brabender Mix	good
Dump Condition	good
Milling	good
Calenderable	yes
Normal Stress-Strain - cure: 320°F	
50% M, psi	
35'	952
60'	947
Tensile, psi	
35'	1298
60'	1226
Ult. Elong., %	
35'	70
60'	65
% Compression Set (73°F) - 70 hrs./25%, cure: 40' @ 320°F	
	13.6
Shore A Hardness (73°F) - on compression set button	
	66.5
Gehman Low Temp. Properties - cure: 40' @ 320°F	
T_5, °F	-78.7, -75.1
G @ RT, psi	156, 185
G @ -55°C, psi	528, 700
Trouser Tear (73°F) - cure: 40' @ 320°F	
lbs./in.	4.0

Recipe: 100 - polymer (K18161-302A), 30 - Shawinigan black, 6 - MgO, 2 - stabilizer, 1.1 - Vulcup 40KE.

TABLE LI

EVALUATION OF TEFLON 6

Stock R199	-484	-485
Teflon 6	2.0	4.0
Normal Stress-Strain - cure: 40' @ 325°F		
100% M, psi	820	1147
Tensile, psi	1144	1440
Ult. Elong., %	145	150
% Compression Set (73°F) - 70 hrs./25%, cure: 50' @ 320°F	12.3	17.7
Shore "A" Hardness (73°F) - on compression set button	63.0	65.0
Trouser Tear (73°F) - cure: 45' @ 320°F		
lbs./in.	17.5	14.8
Gehman Low Temp. Properties - cure: 45' @ 320°F		
T_5, °F	-70.6	-74.2
G @ RT, psi	95	146
G @ -55°C, psi	476	540

Recipe: 100 - polymer (K18161-302A), 30 - FEF black, 6 - MgO, 2 - stabilizer, Teflon 6 - as shown, 0.6 Vulcup 40KE.

TABLE LII

EVALUATION OF TEFLON 8-A IN COMBINATION WITH SILANE A-174

Stock R199	-492	-493	-494
Teflon 8A	4.0	2.0	4.0
Silane A-174	--	2.0	2.0
Normal Stress-Strain - cure: 320°F			
100% M, psi			
35'	1596	1300	--
60'	1144	--	--
Tensile, psi			
35'	1650	1300	1348
60'	1248	1194	1310
Ult. Elong., %			
35'	125	100	80
60'	140	80	85
% Compression Set (73°F) - cure: 40' @ 320°F	16.9	9.5	14.7
Shore "A" Hardness (73°F) - on compression set buttons	67.0	71.0	72.0
Trouser Tear (73°F) - cure: 40' @ 320°F			
lbs./in.	50	14	50
Gehman Low Temp. Properties - cure: 40' @ 320°F			
T_5, °F	-42.7	-51.7	-29.2
G @ RT, psi	103	128	115
G @ -55°C, psi	1328	1137	1878

Recipe: 100 - polymer (K18161-302B), 30 - FEF black, 6 - MgO, 2 - stabilizer, Teflon and Silane - as shown, 0.6 - Vulcup 40KE.

TABLE LIII

ANALYSES OF NEW PNF®-LT

No. RPP	-10721	-10743	-10749	-10754	-10758	-10759
DSV	1.91	1.70	1.58	1.39	2.39	2.51
% Gel	0	0	0	0	0	0
Tg, °C	-82.5	-84.5	-85.0	-84.0	-83.0	-82.0
% Na	0.039	0.022	0.018	0.023	0.26	0.02
% Cl	0.033	0.05	0.05	0.036	0.39	0.18
% R_fO*	82	69	67	67	79	75
% RO*	18	31	33	33	21	25
wt. % F**	45.2	39.6	38.7	38.7	44.0	41.3

* Mole % of pendant groups based on NMR determination.
** Determined on the basis of pendant group analyses (NMR).

TABLE LIV

EVALUATION OF NEW PNF®-LT'S FOR PRODUCTION OF ARCTIC FUEL HOSE

Stock R	-199498	-203807	-203808	-203811	-203812	-203814
Polymer RPP	-10721	-10743	-10749	-10754	-10758	-10759
Vulcup 40KE	0.7	1.0	1.0	1.3	1.0	1.0
Normal Stress-Strain - cure: 35' @ 320°F						
100% M, psi	372	317	570	229	852	687
Tensile, psi	1232	892	900	795	852	985
Ult. Elong., %	195	185	125	210	100	145
Mill Processing - poor for all stocks--would not form tight bond.						
Trouser Tear (73°F) - cure: 40' @ 320°F						
lbs./in.	16.6	21.0	12.5	31.0	13.0	11.0
Gehman Low Temp. Properties - cure: 40' @ 320°F						
T_5, °F	-74.2	-76.0	-79.6			
G @ RT, psi	43	29	49			
G @ -55°C, psi	136	94	136			
% Vol. Increase in Type II Fluid (TT-S-735) (73°F)						
94 hrs.	36.5	72.7	60.7			
14 days	38.3	--	--			

Recipe: 100 - polymer, 30 - FEF black, 6 - MgO, 2 - stabilizer, Vulcup - as shown.

TABLE LV

EVALUATE BLENDS OF PNF®-LT AND PNF®-200

Stock R	-203809	-203810	-203805	-203806
Polymer				
RPP10743 (LT)	80.0	60.0	40.0	--
RPP10380 (200)	20.0	40.0	60.0	100.0
Vulcup 40KE	1.1	1.1	1.1	1.2
Mixing Evaluation				
Brabender Mix	good	good	good	good
Dump Condition	good	good	good	good
Milling	fair	fair	fair	
Calenderable	probably	probably	probably	
Normal Stress-Strain - cure: 35' @ 320°F				
100 M, psi	957	--	956	1200
Tensile, psi	1126	1286	1599	1960
Ult. Elong., %	105	100	140	120
Trouser Tear (73°F) - cure: 40' @ 320°F				
lbs./in.	5.7	6.8	14.8	22.8
Gehman Low Temp. Properties (73°F) - cure: 40' @ 320°F				
T_5, °F	-65.2	-67.9	-53.5	-55.3
G @ RT, psi	77.0	139.4	57.5	96.6
G @ -55°C, psi	369.1	565.1	472.0	921.0
% Vol. Increase in Type II Fluid (TT-S-735) (73°F) - cure: 35' @ 320°F				
94 hrs.	43.1	31.8	25.3	6.9
14 days	43.4	33.2	26.2	7.7

Recipe: polymer - as shown, 30 - FEF, 6 - MgO, 2 - stabilizer, Vulcup 40KE - as shown.

TABLE LVI

EVALUATION OF BLEND OF 6 BATCHES OF PNF®-LT FOR PRODUCTION OF ARCTIC FUEL HOSE

Stock R	-203816	-203817	-203818
Normal Stress-Strain - cure: 320°F, measurements on ring specimens			
100% M, psi			
35'	364	415	369
45'	377	441	398
Tensile, psi			
35'	978	1077	1045
45'	978	1029	963
Ult. Elong., %			
35'	200	203	220
45'	193	187	193

Recipe: 100 - polymer (K15900 - three samples from three of six lots obtained from blending), 30 - FEF black, 6 - MgO, 2 - stabilizer, 1 - Vulcup 40KE.

TABLE LVII

OPTIMUM BLEND OF PNF®-LT AND PNF®-200

Stock R	-203824	-203825	-203826
Polymer			
K15900 (LT)	80	60	50
RPP10424 (200)	20	40	50
Vulcup 40KE	1.0	1.0	0.9
Normal Stress-Strain - cure: 320°F			
100% M, psi			
35'	658	897	1098
60'	665	1062	958
Tensile, psi			
35'	1041	1250	1440
60'	946	1328	1145
Ult. Elong., %			
35'	165	145	130
60'	140	135	120
% Compression Set (73°F) - 70 hrs./25%, cure: 45' @ 320°F	20.0	13.2	10.1
Shore "A" Hardness (73°F) - on compression set button	50.0	56.0	56.0
Gehman Low Temp. Properties - cure: 40' @ 320°F			
T_5, °F	-74.2	-66.6	-67.5
G @ RT, psi	64	85	97
G @ -55°C, psi	219	406	432
Trouser Tear (73°F) - cure: 40' @ 320°F			
lbs./in.	15	17	10

Recipe: polymer - as shown, 30 - FEF, 6 - MgO, 2 - stabilizer, Vulcup 40KE - as shown

TABLE LVIII

OPTIMUM PEROXIDE LEVEL FOR 60:40 PNF®-LT:PNF®-300 BLEND

Stock R	-203820	-203821	-203822
Polymer			
K15900 (LT)	60	60	60
RPP10424 (200)	40	40	40
Vulcup 40KE	0.8	1.0	1.2
Normal Stress-Strain - cure: 35' @ 320°F			
100% M, psi	554	740	733
Tensile, psi	1231	1260	1235
Ult. Elong., %	190	150	150
% Vol. Inc. in Type II Fluid (TT-S-735) (73°F)			
94 hrs.	34.0	31.9	31.1

Recipe: polymer - as shown, 30 - FEF black, 6 - MgO, 2 - stabilizer, Vulcup

TABLE LIX

TRIAL BANBURY MIX, CALENDERING

Stock R	-203828	-203829
K15900 (PNF®-LT)	60.0	60.0
RPP10424 (PNF®-200)	40.0	40.0
Polybutadiene (HD-35)	--	2.0
FEF	30.0	30.0
MgO	6.0	6.0
Stabilizer	2.0	2.0
Vulcup 40KE	1.3	1.1
Normal Stress-Strain - cure: 35' @ 320°F		
100% M, psi	1264	1045
Tensile, psi	1360	1236
Ult. Elong., %	110	125
Stress-Strain After Calendering - cure: 35' @ 320°F		
100% M, psi		
After calendering @ RT	1354	1137
After calendering @ 140°F	1250	1164
After calendering @ 180°F	--	1069
Tensile, psi		
After calendering @ RT	1354	1228
After calendering @ 140°F	1250	1164
After calendering @ 180°F	--	1160
Ult. Elong., %		
After calendering @ RT	100	110
After calendering @ 140°F	100	100
After calendering @ 180°F	--	115

TABLE LX

BANBURY, MILL MIXING OF STOCKS FOR FINAL HOSE BUILDING

Stock R	-203833 (Tube)	-203834 (Cover)
K15900 (PNF®-LT)	60.0	60.0
RPP10424 (PNF®-200)	40.0	40.0
Polybutadiene (HD-35)	--	2.0
FEF	30.0	30.0
MgO	6.0	6.0
Stabilizer	2.0	2.0
Vulcup 40KE	1.2	1.0
Normal Stress-Strain After Calendering - cure: 320°F		
100% M, psi		
45'	1148	994
90'	1114	923
Tensile, psi		
45'	1246	1195
90'	1216	1184
Ult. Elong., %		
45'	120	135
90'	110	145

Table LXI

Testing Results on Final Hoses - Physical Requirements

A. Collapsible Hose

Test	Result	Spec.
Inside Diameter	2 1/16"	2 ± 1/10"
Weight	16.32 oz./ft.	16 oz./ft. (max.)
Hydrostatic Proof	No leaks or imperfections	No leaks or imperfections
Length Change and Twist	Length change - 0 Twist - 0	Length ± 3% max. Twist 7°/ft. max.
Burst Pressure	650 psi (coupling)	200 psi min.
Initial Adhesions		
Tube to ply	8 lbs./in.	10 lbs./in. min.
Between plies	7 lbs./in.	10 lbs./in. min.
Cover to ply	7 lbs./in.	10 lbs./in. min.
Adhesion after filling (Type II Fluid)		
Tube to ply	5 lbs./in.	6 lbs./in. min.
Between plies	6 lbs./in.	6 lbs./in. min.
Cover to ply	5 lbs./in.	6 lbs./in. min.

B. Suction Hose

Test	Result	Spec.
Inside Diameter	2 1/16"	2 ± 1/16"
Weight	30.72 oz./ft.	32 oz./ft. max.
Length Change and Twist	Length Change - 2% Twist 0.89°/ft.	Length ± 3% max. Twist 7°/ft.
Burst Pressure	400 psi (coupling)	200 psi min.
Crush Resistance - % of original O.D.	92.3% under load 98.7% after load release	85% under load max. 95% after load release max.

LXII

Properties of Tube and Cover of Final Hose

Initial	Tube Obtained	Tube Spec.	Cover Obtained	Cover Spec.
100% M, psi	470	--	626	--
Tensile, psi	955	1500	912	1500
Ult. elong., %	200	150	163	150

Immersed in Type II Fluid of TT-S-735 @ R.T. for	Tube 94 hrs.	Spec.	14 days	Spec.	Cover 94 hrs.	Spec.	14 days	Spec.
100 % M retained, %	103	---	84.5	---	70.4	---	69.9	----
Tensile retained, %	79.3	60	51.3	60	76.3	40	67.2	40
Ult. elong. retained, %	81.5	85	62.5	80	92.0	80	84.3	75
Volume increase, %	24.2	40	32.3	40	32.7	70	32.5	70
Wt. change, %	----	---	3.8	5.0	----	----	3.7	5.0

Immersed in Distilled Water @ 160°F for	Tube 14 days	Spec.	42 days	Spec.	Cover 14 days	Spec.	42 days	Spec.
100% M retained, %	106	----			77.4	---		
Tensile retained, %	85.1	80			79.3	80		
Ult. elong retained, %	83.8	80			92.0	80		
Volume increase, %	24.9	15			17.7	15		

After Accelerated Weathering (500 hrs)	Cover only: Found	Spec.
Tensile retained, %	106	85
Ult. elong. retained, %	95.5	85

After Ozone Exposure — Cover only: no cracking or checking

Existent Gum	Found	Spec.
Unwashed, mg/100ml	1880	20
Washed, mg/1000ml	16.5	5

Brittleness - after 166 hrs @ -70°F — No cracking

Gehman Properties	Tube	Cover
T5°F	68	69
G @ R.T., psi	86	145
G @ -55°C., psi	429	700

Table LXIII

Tests on Press - Cured Samples of Excess Stock from Final Hose Building

Stock	R203 833 (Tube)	R203 834 (Cover)
Normal Stress - Strain - cure: 35' @ 320°F		
100% M_1 psi	1118	1054
Tensile, psi	1308	1322
Ult. elong.	125	135

Stock	R203 833 (Tube)			R203 834 (Cover)		
Aged 94 hrs in Type II Fluid (R.T.) -	original	aged	% retained	original	aged	% retained
100% M	1103	975	88.4	1022	789	77.2
Tensile	1245	1186	95.3	1219	1071	87.9
Ult. elong.	113	123	109	125	147	118
Aged 14 days in Type II Fluid (R.T.)						
100% M	----	1030	----	1010	990	98.0
Tensile	1250	1190	95.2	1200	1120	93.3
Ult. elong.	90	120	133	130	110	84.6

A P P E N D I X

DEPARTMENT OF THE ARMY
US ARMY MOBILITY EQUIPMENT RESEARCH & DEVELOPMENT COMMAND
FORT BELVOIR, VIRGINIA 22060

DRXFB-VU

7 September 1976

Evaluation of Firestone PNF Elastomer Compounds for Arctic Fuel Hose

Report No: 06343 EBBY

Requested by: Fuels Handling Equipment Div, Lab 2000, ATTN: Mr. P. Mitton

Authority: A6H67FD0231

1. The purpose of this work was to evaluate PNF rubber compounds developed by Firestone for fabricating an arctic fuel hose.

2. The PNF press cured compounds submitted by Firestone were identified as follows:

a. R 197-369 - Compound used in hose fabrication studies in Contract DAAG53-75-C-0187 and described in Interim Report dated Nov 75.

b. R199-464 - Tube Compound used in third hose fabrication studies in Contract DAAG53-75-C-0187 and described in Letter Report dated May 17, 1976.

c. R 199-465 - Jacket compound used in third hose fabrication studies in Contract DAAG53-75-C-0187 and described in Letter Report dated May 17, 1976.

3. The formulations and test data are presented in Table 1.

4. The candidate hose compounds exhibited low original tensile strength. All other hose properties such as water and fuel resistance as well as low temperature flexibility were met by the PNF compounds.

5. Conclusions and recommendations are withheld at this time pending a complete evaluation of the hose fabricated by Firestone to be submitted at a later date.

SUBMITTED BY: Paul Touchet
PAUL TOUCHET
Chief, Rub & Ctd Fab Rsch Grp

1 Incl
Table I

FORWARDED BY: Emil J. York
EMIL J. YORK
Chief, Lab 2000

TABLE I

Formulations and Test Results of Firestone Arctic Fuel Hose Compounds

	Polymer I.D.	R197-369	R199-464	R199-465	Hose PD Requirements	
PNF	K18161-302A	100	---	---		
PNF	K18352	---	100	97.5		
EPDM		---	---	2.5		
FEF Black		30	30	30		
Mg Oxide		6	6	6		
Stabilizer		2	2	2		
Vulcup R		.4	---	---		
Vulcup 40KE		---	.7	.7		
Cured at 320°F		35'	60'	60'		
					Tube	Jacket
Original Properties						
Tensile Str.	PSi	940	870	860	1500min	1500min
Elongation	%	150	170	180	150min	150min
Hardness	Shore A	60	58	58	---	---
100% Modulus	PSi	610	350	400	---	---
Mod. of Rigidity, "G", PSi		71	81	105	---	---
After Immersion in Distilled Water 14 Days at 160°F						
Tensile Ret.	%	70.	87	94	80min	80min
Elong. Ret.	%	93.	97	103	80min	80min
Hardness Ch.	Points	0	+2	+2	---	---
Volume Swell	%	9	9	8	15max	15max
100% Mod Ret	%	77	113	83	---	---
After Immersion in Distilled Water 42 Days at 160°F						
Tensile Ret.	%	63(1)	72	97	60min	60min
Elong. Ret.	%	100(1)	94	79	60min	60min
Hardness Ch.	Points	-5(1)	0	+2	---	---
Volume Swell	%	15(1)	15	13	20max	20max
100% Mod. Ret.	%	58(1)	99	89	---	---
After Immersion in Type II Fluid of TT-S-735 for 94 Hrs at 73°F						
Tensile Ret.	%	58	56	55	60min	60min
Elong Ret	%	93	85	83	85min	80min
Hardness Ch	Points	-13	0	-6	---	---
Volume Swell	%	22	26	33	40max	70max
100% Mod Ret	%	57	60	59	---	---

TABLE I (Cont.)

Formulations and Test Results of Firestone Arctic Fuel Hose Compounds

	Polymer I.D.	R197 369	R199-465 464	R199-465	Hose PD Requirements	
After 7 Days at -70^oF						
Ten. Rec.	%	40	43	39	20min	20min
Comp. set	%	66.1	60	63	---	---
TSR	%	4.9	3.2	5.2	5max	5max
G	~~PSi~~	347	257	543	---	---
After 7 days at -40^oF						
Ten. Rec.	%	73	60	60	---	---
Comp Set	%	54	42	43	---	---
TSR	---	2.1	2.1	2.7	---	---
G	PSi	146	168	283	---	---
Existent Gum						
Unwashed mg/1000ml		4			20	---
Washed mg/1000ml		2			5	---

NOTES:

1. The properties were determined after immersion in water for 70 days at 160^oF.

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

This report discusses work directed toward preparation of fuel resistant hose that is serviceable at temperatures as low as -70°F. The bulk of the report describes compounding studies which attempted to provide a balance of properties suitable for Arctic fuel hose. A description is also provided of the fabrication of large lengths of collapsible and suction type hoses.

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It was found that the polymer, PNF®-LT, a modified phosphonitrilic fluoroelastomer, could be compounded to permit production of Arctic fuel hoses which possess good low temperature flexibility. The hoses possessed good dimensional stability and physical strength, but tensile and tear strengths on samples from the hoses were lower than desired.

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