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Elastomer Compatibility with Liquid Propellant at Elevated Temperature

Prepared by Henry O. Feuer, Jr. and Alan R. Teets

Report Date
December 1990



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Report Number 2498

Elastomer Compatibility with Liquid Propellant at Elevated Temperature (U)

Prepared by Henry O. Feuer, Jr. and Alan R. Teets



US Army Belvoir RD&E Center Materials, Fuels, and Lubricants Directorate Fort Belvoir, Virginia 22060-5606

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PREFACE

This report details the investigation and results in the evaluation of the compatibility of various elastomeric compounds that may be used in the transfer and containment of liquid propellants (LPs) currently under development by the US Army. The work performed is in support of the Liquid Propellant Program at the US Army Ballistics Research Laboratory (BRL), Aberdeen Proving Ground, MD. The studies were conducted by the Rubber and Coated Fabrics Research Group, Materials, Fuels and Lubricants Directorate, US Army Belvoir Research, Development, and Engineering Center (BRDEC), Fort Belvoir, VA.

Hydroxyl Ammonium Nitrate (HAN)-based LPs are monopropellants composed of an oxidizer (HAN) and a fuel (an aliphatic amine nitrate). The HAN-based LP used in this study has the designation of 1846. It is a water-based propellant with a density of approximately $1.4g/cm^3$. The fuel component of the LP-1846 is triethanol ammonium nitrate. The Army foresees using LP-1846 in a regenerative injection system for 155mm guns. The regenerative system involves the use of a piston forcing the LP, in the form of a jet or a spray, into the gun chamber during the combustion process, thereby controlling propellant combustion.

Ammonium nitrates are a family of inorganic compounds of which triethanol and hydroxyl ammonium nitrates are members. The basic chemical structure difference between the two nitrates is the presence of the alcohol or the hydroxyl molecular group. When combined, this produces an explosive mixture-triethanol ammonium nitrate which provides the oxidative fuse, and hydroxyl ammonium nitrate which provides the fuel. Whether separate or mixed, the nitrates' effect on elastomer material properties should be similar; therefore, materials that exhibit resistive properties to alcohol and oxidative environments are good candidates for evaluation.

A literature search of elastomers compatible with ammonium nitrates was performed during prior room temperature LP and elastomer compatibility testing. The results of this search provided candidate materials for testing in LP at room temperature. The results of the room temperature testing provided candidate materials for the current testing. The candidate formulations were selected from the following groups:

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- Nitriles (including nitrile/PVC blends)
- Chloroprenes
- Fluoroelastomers
- Halogenated Butyl
- Ethylene-Propylene

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The storage and handling of LP in Army units will probably follow procedures similar to those used in fuel handling so an analysis of the materials used by the hose and collapsible tank industries was performed. This analysis revealed that most of the commercially available products produced in these fields are manufactured by a relatively small group of companies. Several of these companies were contacted to ascertain what elastomers are in use and if other elastomers could be used in production.

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The collapsible tank industry currently uses various types of chloroprenes, nitriles, buty¹, urethane, and thermoplastic urethane elastomers. In addition to those elastomers, the hose industry uses fluoroelastomers. The collapsible tank companies report that fluoroelastomers could be used in their production. Previous work has shown that thermoplastic urethanes, urethane and butyl elastomers, are incompatible with LPs at room temperature. Nitriles, chloroprenes, fluoroelastomers, and halogentated butyl materials were selected for testing at elevated temperatures.

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SECTION I — INVESTIGATION

SCOPE

The purpose of this project was to determine the compatibility of elastomeric materials with the HAN-based propellant at an elevated temperature of 150°F as traced by the deterioration of the elastomers' physical properties.

Elastomeric Compounds

A total of ten elastomeric compounds were tested. These compounds have been used or have the potential for use in military applications such as gaskets, seals, hoses, tubing, insulators, or in coated fabrics for fuel storage tanks. Table 1 lists the elastomeric compounds used in this test as well as a test code used for identification.

| Table 1. | List of Elastomeric | Materials for | Compatibility | with LPs | at 150°F |
|----------|---------------------|---------------|---------------|----------|----------|
|----------|---------------------|---------------|---------------|----------|----------|

| NITRILE ELASTOMERS | TEST CODE |
|--------------------------|-----------|
| NBR-2 | LP-1 |
| NBR-8 | LP-2 |
| NBR-9 | LP-3 |
| NITRILE/PVC BLENDS | |
| 1203-F60-R2, Radian | LP-4 |
| VT-380, Radian | LP-5 |
| POLYCHLOROPRENES | |
| CR-1A | LP-6 |
| CR-2A | LP-7 |
| FLUOROELASTOMERS | |
| Viton 1A | LP-8 |
| Viton 2A | LP-9 |
| MISCELLANEOUS ELASTOMERS | • |
| PE-100-A-027 | LP-10 |

Of the ten elastomeric compounds, three were obtained as cured sheets from end item manufacturers; the formulations were not provided and remain proprietary. The remaining seven compounds were formulated, mixed, and molded in-house. Standard 6 x 6-inch test sheets were molded in accordance with ASTM D-3182, *Rubber Materials, Equipment and Procedures*, for mixing standard compounds and preparing standard vulcanized sheets. The formulations and curing conditions for these compounds are shown in Table 2. Commercial and chemical names, along with suppliers, are listed in Table 3.

| INGREDIENTS | NBR-2 | NBR-8 | NBR-9 | CR-1A | CR-2A | VITON-1A | VITON-2A | |
|-------------------------------|-----------|--------|--------|----------|--------|----------|----------|--|
| Paracril CJ | | 100 | | | | | | |
| Paracril 1880 | | | 100 | | | | | |
| Zeptol 2020 | 100 | | | | | | | |
| Neoprene GW | | | | 100 | | | | |
| Neoprene WRT | | | | | 160 | | | |
| Viton GLT Viton GF | | | | | | 100 | | |
| Zinc Oxide | F | 5 | 5 | <i>c</i> | - | | 100 | |
| Stearic Acid | 5 2 | 5 2 | 5 2 | 5 5 | 5 | | | |
| SAF Black, N-110 | 45 | 2 | 2 | 5 | 1 | | | |
| SAF-HS, N-121 | •• | 45 | 45 | | 40 | | | |
| ISAF Black, N-220 | | | -0 | 35 | -0 | | | |
| MT Black, N-990 | | | | | | 30 | 20 | |
| Agerite Resin D | | 0.5 | 0.5 | | | | 20 | |
| Agerite White | | 0.5 | 0.5 | | | | | |
| Vanox MTI | | 0.5 | .05 | | 1 | | | |
| Sulfur | 0.8 | 0.8 | 0.8 | | | | | |
| Butyl Tuads | 0.7 | | | | | | | |
| Octoate Z | 1.5 | | | | | | | |
| Amyl Ledate | 1.0 | | | | | | | |
| Amax | 1.9 | | | | | | | |
| Santoflex 13 | 3.0 | 3.0 | 3.0 | | | | | |
| Novor 924 | | 4.2 | 4.2 | | | | | |
| TMTM, Monex | | 1.5 | 1.5 | | | | | |
| Santocure NS Santogard PVI | | 0.1 | 0.1 | | | | | |
| Sundex 790 | | 1.25 | 1.25 | 10 | 10 | | | |
| Magnesium Oxide | | | | 4 | 4 | | | |
| Octamine | | | | 2 | 7 | | | |
| ETU-22 PM | | | | - | 0.75 | | | |
| Agerite Superflex | | | | | | | | |
| Solid G | | | | | 2 | | | |
| MBTS | | | | | 1 | | | |
| Calcium Hydroxide | | | | | | 4 | 3 | |
| Luperco 101XL | | | | | | 4 | 1.5 | |
| Diak #7 Cure Conditions | | | | | | 4 | 2 | |
| (min/°F) | 50/320 | 35/320 | 35/330 | 40/320 | 60/300 | 10/350 | 8/350 | |
| Post Cure | | | | | | 041450 | OAICAO | |
| (hrs/°F) | | | | | | 24/450 | 24/540 | |

Table 2. Formulations of In-House Elastomers Used in LP Testing

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| COMMERCIAL NAME | CHEMICAL NAME | SUPPLIER |
|---------------------------|--|-------------------|
| Paracril CJ | Hot polymerized, 38.5% ACN, nitrile rubber | Uniroyal Chemical |
| Paracril 1880 | Hot polymerized, 22% ACN, nitrile rubber | Uniroyal Chemical |
| Zeptol 2020 | Acrylonitrile-ethylene-butadiene terpolymer, 37% ACN | Zeon Chemical |
| Neoprene GW | Sulfur-modified polychloroprene | Dupont |
| Neoprene WRT | Co-polymer of chloroprene and 2, 3-dichloro | |
| | -1, 3-butadiene | Dupont |
| Viton GLT | Peroxide curable fluoroelastomer | Dupont |
| Viton GF | Peroxide & coagent curable fluoroelastomer | Dupont |
| Agerite Resin D | 1, 2-dihydro-2, 2,4 trimethylouinoline | R. T. Vanderbilt |
| Agerite White | Di-beta-naphtynyl-P-pnenylene-oiamine | R. T. Vanderbilt |
| Vanox MT1 | 2-mercaptotoivlimidazole | R. T. Vanderbilt |
| Butyl Tuads | TetrabutyIthiuram disulfide | R. T. Vanderbilt |
| Octoate Z | Zinc 2-ethylhexoate | R. T. Vanderbilt |
| Amyl Ledate | Lead diamyldithiocarbamate | R. T. Vanderbilt |
| Amax | N-oxydiethylene benzothiazole-2-sulfenamide | R. T. Vanderbilt |
| Novor 924 | Diurethane vulcanizing agent | Akron Chemical |
| TMTM, Monex | Tetramethyl thiuram monosulfide | Akron Chemical |
| Santocure NS | N-T-butyl-2-benzothiazole sulfenamide | Monsanto |
| Santogard PVI | N-(cyclohexylthio) phthalimide | Monsanto |
| Sundax 790 | Aromatic oil, ASTM D2226, Type 101 | R. E. Carroll |
| Octamine | Diphenyl-amine and diisobutylene | Uniroyal Chemical |
| Agerite Superflex Solid G | Diphenylamine | R. T. Vanderbilt |
| MBTS | 2, 2'benzothiazyl disulfide | Akron Chemical |
| Luperco 101XL | 2, 5-dimethyl-2, 5-BIS (1-but; -peroxy) nexane | Pennwalt |
| Diak #7 | Polyfunctional triazine derivative | Dupont |
| Santoflex 13 | N-1, 3-Dimethylbutyl-N'-phenyl-p-phenylenediamine | Monsanto |
| ETU-22 PM | Ethylene thiourea | Akron Chemical |

Table 3. List of Commercial and Chemical Names and Suppliers

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Liquid Propellant

The LP-1846 used in this program was supplied by the Ballistics Research Laboratory (BRL). It was received as a Class II explosive. Personnel and laboratory safety procedures were established for the safe handling, storage, and disposal of the LP as suggested by the BRL. The LP was used as received.

PREPARATION

Sample Preparation

Test specimens were prepared from the vulcanized sheets in accordance with ASTM D-412, Rubber Properties in Tension, using a Die-C type dumbbell. The samples were cut and initial thicknesses and weight, were measured. Sets of three samples of each type elastomer were prepared for differing time intervals. The sets of elastomers were immersed in test tubes containing LP and sealed with teflon-lined caps to prevent evaporation. The time intervals used were 0 days (original), 7 days, 14 days, 28 days, 42 days, and 70 days. The samples in the LP were conditioned at an elevated temperature of $150^{\circ}F \pm 5^{\circ}F$.

Procedures

The immersion of the elastomeric materials and the determination of the affect on their properties was performed in accordance with ASTM D-471, *Kubber Properties—Effects of Liquids*. After specimens were exposed to the LP, they were cooled to room temperature in the original test fluid approximately 4 hours. The samples were then individually removed and rinsed in deionized water prior to measuring. The change in volume was determined by the water displacement method at room temperature as described in Section 10 of ASTM D-471. The volume of the liquid propellant was kept constant during the conditioning periods to eliminate concentration variations due to any extracted ingredients from the elastomers.

RESULT DATA

The results reported were produced by the average of three specimens. Statistical analysis was performed on all test data to ascertain the accuracy of results. The physical properties of all the elastomeric materials are shown in Tables 4 through 8. Table 4 shows tensile strength data, Table 5 shows ultimate elongation data, Table 6 shows volume change data, while Tables 7 and 8 show tensile strength retention and ultimate elongation retention, respectively.

| CODE | COMPOUND | ORIGINAL | 7 DAYS | 14 DAYS | 28 DAYS | 42 DAYS | 70 DAYS |
|-------|-----------|----------|--------|---------|------------------|---------|---------|
| LP-1 | NBR-2 | 5070 | 3450 | 2300 | 1730 | 1650 | 1200 |
| LP-2 | NBR-8 | 3640 | 2920 | 2300 | 1760 | 1430 | 1310 |
| LP-3 | NBR-9 | 2700 | 2600 | 2340 | 1980 | 1840 | 1430 |
| LP-4 | 1203F60R2 | 2370 | 2470 | 2390 | 240 ^r | 2120 | • |
| LP-5 | VT-380 | 3140 | 1970 | 2240 | 1630 | 1430 | 960 |
| LP-6 | CR-1 | 1600 | 1690 | • | 1520 | 1610 | 1570 |
| LP-7 | CR-2 | 3660 | 3870 | * | 3720 | 3870 | 3750 |
| LP-2 | Viton-1 | 2480 | 2400 | • | 2880 | 2270 | 2670 |
| LP-9 | Viton-2 | 2000 | 2110 | • | 2260 | 2080 | 2200 |
| LP-10 | PE100A027 | 1760 | ** | 1780 | •• | ** | • • |

Table 4. Tensile Strength (psi) of Elastomers in LP at 150°F

* No data collected due to experimental error.

** No data collected because samples reacted with LP.

Table 5. Ultimate Elongation (%) of Elastomers in LP at 150°F

| CUDE | COMPOUND | ORIGINAL | 7 DAYS | 14 DAYS | 28 DAYS | 42 DAYS | 70 DAYS |
|-------|-----------|----------|--------|---------|---------|---------|---------|
| | | | | | | | |
| LP-1 | NBR-2 | 470 | 330 | 240 | 170 | 140 | 90 |
| LP-2 | NBR-8 | 410 | 290 | 210 | 150 | 120 | 100 |
| LP-3 | NBR-9 | 310 | 270 | 240 | 200 | 180 | 140 |
| LP-4 | 1203F60R2 | 340 | 270 | 200 | 150 | 110 | • |
| LP-5 | VT-380 | 520 | 290 | 280 | 220 | 200 | 100 |
| LP-6 | CR-1 | 240 | 240 | | 220 | 230 | 220 |
| LP-7 | CR-2 | 480 | 480 | ٠ | 440 | 440 | 420 |
| LP-8 | Viton-1 | 210 | 210 | • | 230 , | 100 | 220 |
| L2-3 | Viton-2 | 300 | 310 | * | 310 | 300 | 310 |
| LP-10 | PE100A027 | 840 | ** | 820 | ** | ** | ** |

* No data collected due to experimental error.

** No data collected because samples reacted with LP.

| CODE | COMPOUND | 7 DAYS | 14 DAYS | 28 DAYS | 42 DAYS | 70 DAYS |
|-------|-----------|--------|---------|---------|---------|---------|
| LP-1 | NBR-2 | 0.36 | 0.44 | 1.60 | 2.35 | 7.37 |
| LP-2 | NBR-8 | 0.13 | 0.85 | 5.01 | 9.10 | 15.85 |
| LP-3 | NBR-9 | -0.34 | -0.12 | 1.74 | 2.31 | 4.83 |
| LP-4 | 1203F60R2 | 0.38 | 1.13 | 4.76 | 7.10 | ٠ |
| LP-5 | VT-380 | -0.18 | 0.52 | 2.56 | 11.12 | 9.22 |
| LP-6 | CR-1 | 0.29 | 0.78 | 1.50 | 2.35 | 3.45 |
| LP-7 | CR-2 | 0.78 | 1.07 | 1.97 | 2.77 | 3.91 |
| LP-8 | Viton-1 | -0.12 | 0.11 | 0.79 | 1.13 | 2.43 |
| LP-9 | Viton-2 | -0.05 | 0.21 | 0.26 | 0.71 | 0.67 |
| LP-10 | PE100A027 | •• | 0.10 | ** | ** | ** |

Table 6. Volume Change (%) of Elastomers in LP at $150^{\circ}F$

* No data collected due to experimental error.

** No data collected because samples reacted with LP.

| CODE | COMPOUND | 7 DAYS | 14 DAYS | 28 DAYS | 42 DAYS | 70 DAYS |
|-------|-----------|--------|---------|---------|---------|---------|
| LP-1 | NBR-2 | 68.2 | 45.2 | 33.8 | 32.0 | 22.6 |
| LP-2 | NBR-8 | 80.2 | 62.8 | 46.8 | 37.1 | 32.6 |
| LP-3 | NBR-9 | 96.5 | 86.7 | 72.5 | 67.1 | 51.3 |
| LP-4 | 1203F60R2 | 104.0 | 100.1 | 98.2 | 85.5 | • |
| LP-5 | VT-380 | 62.8 | 71.1 | 51.0 | 42.5 | 28.8 |
| LP-6 | CR-1 | 105.4 | • | 94.1 | 99.1 | 95.9 |
| LP-7 | CR-2 | 105.2 | • | 100.3 | 103.8 | 99.9 |
| LP-8 | Viton-1 | 96.9 | • | 115.5 | 90.8 | 106.0 |
| LP-9 | Viton-2 | 105.5 | • | 112.8 | 103.5 | 109.5 |
| LP-10 | PE100A027 | •• | 101.1 | ** | •• | ** |

Table 7. Tensile Strength Retention (%) of Elastomers in LP a. \50°F

* No data collected due to experimental error.

** No data collected because samples reacted with LP.

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| CODE | COMPOUND | 7 DAYS | 14 DAYS | 28 DAYS | 42 DAYS | 70 DAYS |
|-------|-----------|--------|---------|---------|---------|---------|
| LP-1 | NBR-2 | 70.2 | 51.1 | 36.2 | 29.8 | 19.1 |
| LP-2 | NBR-8 | 70.7 | 51.2 | 36.6 | 29.3 | 24.4 |
| LP-3 | NBR-9 | 87.1 | 77.4 | 64.5 | 58.1 | 45.2 |
| LP-4 | 1203F60R2 | 79.4 | 58.8 | 44.1 | 32.4 | • |
| LP-5 | VT-380 | 55.8 | 53.8 | 42.3 | 38.5 | 19.2 |
| LP-6 | CR-1 | 100.0 | • | 91.7 | 95.8 | 91.7 |
| LP-7 | CR-2 | 100.0 | • | 91.7 | 91.7 | 87.5 |
| LP-8 | Viton-1 | 100.0 | • | 109.5 | 90.5 | 104.8 |
| LP-9 | Viton-2 | 103.3 | • | 103.3 | 100.0 | 103.3 |
| LP-10 | PE100A027 | ** | 97.6 | ** | ** | ** |

Table 8. Ultimate Elongation Retention (%) of Elastomers in LP at $150^{\circ}F$

* No data collected due to experimental error.

** No data collected because samples reacted with LP.

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SECTION II — TEST RESULTS

NOTE: Data was not obtained for LP-6, LP-7, LP-8, and LP-9 for 14-day immersion, and for LP-4's 70-day immersion due to experimental error. LP-10 data for 7-day, 28-day, 42-day, and 70-day immersions was not obtained due to a reaction that blew the test tube top open.

TEST DESCRIPTIONS

Tensile Strength

Tensile strength is a standard control measurement used in the rubber industry. It is useful for comparing compounds for quality control, and as a test to determine if an elastomer has deteriorated when exposed to a hostile environment. In most cases, if an elastomer has a large reduction in tensile strength after immersion in a test fluid, a relatively short life span for the elastomer can be expected. Conversely, a very small loss in tensile strength indicates a longer life span which is a necessary condition for dynamic applications.

Four of the elastomeric compounds tested—LP-1, LP-2, LP-3, and LP-5—showed significant decreases in their tensile strength. The decreases ranged from approximately 50% to over 75% after 70-day immersion. The remaining elastomers' tensile strengths stayed relatively constant during testing. The data for the tensile strength is exhibited in Table 2 (page 2) and shown in Figures 1 and 2 (see Appendix for all figures).

Ultimate Elongation

Elongation is defined as the increase in length expressed as a percentage of the initial gage length. The ultimate elongation is the length reached when the sample breaks. Elongation measurements are used in much the same way that tensile strength data is used; however, a large increase or decrease in elongation may represent deterioration.

The ultimate elongation of five of the materials—compounds LP-1, LP-2, LP-3, LP-4, and LP-5—showed decreases of up to 80% after 70-day immersion. The large change in ultimate elongation is a definite sign of excessive degradation. The remaining compounds displayed relatively small changes in ultimate elongation. The results for ultimate elongation are listed in Table 3 (page 3) and displayed graphically in Figures 3 and 4.

Volume Change

The volume change is the increase or decrease of the volume of an elastomer after it has been in contact with a fluid. Volume change is measured as a percentage based on the elastomer's original volume. An increase in volume or swell is usually accompanied by a decrease in hardness. Shrinkage, or a decrease in volume, is usually accompanied by an increase in hardness. Shrinkage is far more critical than swelling, especially if the elastomer is used in a sealing application. The amount of swelling or shrinkage permitted is dependent upon the application. In dynamic o-ring usage, swelling of 15 to 20% is a reasonable maximum, and shrinkage of 3 to 4% is the limit.

The volume change of all the elastomers exhibited swelling after 70-day immersion, although five materials—compounds LP-1, LP-3, LP-5, LP-8, and LP-9—displayed some modest initial shrinkage. The maximum swelling value was about 16% and would still be usable in a dynamic o-ring application. The small amounts of shrinkage correlated well with discoloration in the test fluid. The extraction of compound ingredients was evident in many cases by the discoloration and by the loss in the specimens' weight. The volume increase is due to the test fluid entering in the polymers' matrix causing the sample to expand and increase in weight. Data concerning the elastomers' volume change is shown in Table 5 (page 5) and in Figures 5 and 6.

Tensile Strength Retention

Tensile strength retention is the measured tensile strength after aging, divided by the original tensile strength and corrected for the volume change. It is usually expressed as a percentage. The tensile strength retention often shows trends in the tensile strength more clearly than just the tensile strength data.

Figures 7 and 8 display graphs of the tensile strength retention while Table 7 (page 6) lists the data. In comparing the tensile strength to the tensile strength retention values, most trends were obvious in both cases. The tensile strength retention more clearly defines what percentage of the tensile strength was lost.

Elongation Retention

Elongation retention is the measured elongation, after aging, divided by the original elongation. Since the volume swell is not a factor in these measurements, no correction is necessary. The elongation retention, expressed as a percentage, often helps define trends in elongation more clearly than the elongation data alone. The elongation retention data is listed in Table 8 (page 7) and displayed graphically in Figures 9 and 10. The elongation retention did not define any unobserved trends in the elongation data; however, it is a convenient way to express the loss or gain in elongation for the samples.

ELASTOMER TESTING RESULTS

Nitrile Elastomers

The nitrile elastomers tested were represented by compounds LP-1, LP-2, and LP-3. All three compounds showed poor resistance to LP at 150°F. Compound LP-1 is based on a highly saturated (90%) acrylonitrile-ethylene-butadiene terpolymer with 37% bound acrylonitrile. This compound showed a loss in tensile strength retention of 77% after 70-day immersion and a loss of elongation retention of 80% during the same testing period. The compound exhibited an increase in swelling during testing to 7% after 70-day immersion. Since compound LP-1 loses a large percentage of its original properties, it is unsuitable for elevated LP applications.

Compound LP-2 is based on Paracril CJ nitrile elastomer with 38.5% acrylonitrile content. This material behaved similarly to compound LP-1 in that there was a loss of tensile strength retention and elongation retention at 70 days of 77% and 75%, respectively. Compound LP-2 showed an increasing trend in the volume change of 16% after 70 days. The loss of original properties and the large volume swell would make this material unacceptable.

Compound LP-3 is based on Paracril 1880 nitrile elastomer with 22% acrylonitrile content. A loss of 49% in tensile strength retention after 70-day immersion was recorded as was a loss of 55% in elongation retention during the same time period. This compound exhibited a volume swell of 5% after the 70-day immersion. Although this compound did not deteriorate as severely as compounds LP-1 and LP-2, it still degraded to a point where it would be unacceptable for this application.

The results indicate that nitrile elastomers are not compatible with LP at 150°F. The large loss of properties would make nitrile compounds fail rapidly thereby making them unacceptable.

Nitrile/PVC Blends

Two materials were tested that were nitrile/PVC blends. Compounds LP-4 and LP-5 were both provided by industry and most information concerning them is proprietary.

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Compound LP-4 is based on Hycar 1203-F60 nitrile elastomer blended with PVC with a ratio of 70% NBR/30% PVC. The formulation, additives, and curing system are unknown. The tensile strength for compound LP-4 remained fairly constant through the 28-day immersion test but, at 42 days, it showed a decrease in tensile strength of 11%. The tensile strength retention showed

the trend as a slight increase then gradually losing strength until a loss of 14% was recorded at 42 days. No data was obtained for the 70-day test due to experimental error. The ultimate elongation and elongation retention data showed more serious deterioration. The elongation displayed a constant decrease with only 32% of the original elongation remaining after 42-day immersion. The volume change showed an increasing swelling trend with a value of 7% at 42 days. Based on the deterioration observed in the elongation data, this material should not be used with LP at 150° F.

Compound LP-5 is proprietary and information concerning the nitrile/PVC ratio is unknown. The tensile strength and tensile strength retention results displayed a large decrease. The retention value was only 29% after 70-day immersion. The elongation values also showed decreasing trends with a 70-day immersion value of only 19%. The volume change data started with slight shrinkage then increased in volume to 9% at 70 days. Although the volume change was within acceptable limits, the tensile strength and elongation data clearly showed severe deterioration. Compound LP-5 should not be used with LP at elevated temperatures.

As with the nitrile compounds, the nitrile/PVC blend compounds showed a great deal of deterioration after immersion in LP at 150°F. The nitrile/PVC blends should not be used in LP at elevated temperatures.

Polychloroprenes

Compounds LP-6 and LP-7 were two different types of polychloroprenes tested. These polychloroprenes, also called neoprenes, were produced in-house. The data concerning the 14-day immersion of compounds LP-6 and LP-7 was not collected properly; therefore, with the exception of information concerning the volume change, the data was not reported.

The tensile strength of compound LP-6 displayed some fluctuation around the original tensile strength but remained fairly constant. This was also evident in the tensile strength retention which displayed the same fluctuations. At 70-day immersion, the tensile strength retention was 96%. The ultimate elongation and elongation retention behaved similarly to the tensile strength with a retention value of 92% after 70-day immersion. The volume change displayed consistent swelling with a value of 3.5% after 70-day immersion.

Compound LP-7 displayed tensile strength values slightly above the original value. The tensile strength retention also displayed above original values until the 70-day immersion where the value was 100%.

The elongation and elongation retention displayed a decreasing trend to 88% at 70-day immersion. The volume change exhibited increased swelling throughout the testing to 3.9% after 70-day immersion.

The polychloroprene elastomers used in this testing were supplied by *Dupont*. The formulations were compounded, mixed, and molded in-house. Compound LP-6 was based on Neoprene GW, which is a general purpose neoprene with high tear, flexing, and heat resistance based on interpolymerized chloroprene with sulfur added. Compound LP-7 was based on Neoprene WRT, a more specialized polychloroprene designed for low temperature properties. Neoprene WRT is a copolymer of chloroprene and 2, 3-Dichloro-1, 3 butadiene.

The results for compounds LP-6 and LP-7 are mixed. Compound LP-6's tensile strength displayed more deterioration than compound LP-7. However, compound LP-7's elongation showed more degradation than compound LP-6. The volume change displayed equivalent swelling for both compounds. Although compounds LP-6 and LP-7 showed some deterioration and swelling, they performed well enough to be considered for applications with LP at elevated temperatures. Thorough testing of any polychloroprene should be performed prior to use with LP at elevated temperatures.

Fluoroelastomers

Two fluoroelastomer compounds were tested—LP-8 and LP-9—both produced in-house and based on Viton fluoroelastomers.

After conditioning in LP at 150°F, both compounds exhibited tensile strength and elongation results above the original values for most of the immersion intervals. Data for the 14-day immersion test was deleted, with the exception of the volume change information, due to incorrect testing procedures.

Compound LP-8's tensile strength retention varied between 91% and 116% of original with a value of 106% after 70-day immersion. The elongation retention data varied from 91% to 110% with a value of 105% after 70-day immersion. The volume change displayed a small amount of shrinkage after 7-day immersion, then indicated consistent swelling up to 2.4% after 70-day immersion.

The tensile strength retention of compound LP-9 was greater than the original value throughout the testing, ranging between 104% and 113%. A value of 110% was recorded for the 70-day immersion. The elongation retention also remained at 100% or higher during the testing with a value of 103% after 70-day immersion. The volume change behaved similarly to compound LP-8 in that, at 7-day immersion, there was very slight shrinkage, then increasing swelling to .7% at 70-day immersion.

Fluoroelastomer compounds performed the best of the materials tested with regard to tensile strength, elongation, and volume swell. Fluoroelastomers, therefore, are the best candidate compounds for use with LP at elevated temperatures. However, testing with any

fluoroelastomer compound should be performed prior to its use in the field to insure that other compounding ingredients will be compatible with the LP fluid.

Halogenated Butyl

Compound LP-10 is a halogenated butyl elastomer supplied by industry. Information concerning the formulation and curing is proprietary. Only original and 14-day immersion data was collected for this material. A slight reaction was apparently occurring in the test tubes while the samples were immersed and aging. This reaction caused the tops of some of the test tubes to blow off and the LP to overflow the tube and spill in the oven. There was no pattern as to when this event would occur. The reaction would occur in as quickly as 1 day or not until several weeks into the test. In the case of the 14-day immersion samples, the reaction never occurred. Attempts were made to retest time periods that failed; however, these samples also reacted. The cause of this reaction is unknown but this material clearly cannot be used with LP at elevated temperatures.

COMPARISON TO ROOM TEMPERATURE TEST

Previous work (BRDEC Technical Report 2476, *Elastomer Compatibility with Liquid Propellant, January 1990*) tested elastomers for their compatibility with LP at room temperature. The elastomers that performed well during the room temperature testing were used for this elevated temperature in LP test. Not all of the elastomers that did well at room temperature passed the elevated temperature testing. Graphs comparing the tensile strength retention are shown in Figures 11 to 13; elongation comparisons are presented in Figures 14 to 16; and volume change comparisons are shown in Figures 17 to 19. Tables 9 and 10 list the data for the tensile strength and elongation retention, respectively, of room temperature testing.

| CODE | COMPOUND | 7 DAYS | 14 DAYS | 28 DAYS | 42 DAYS | 70 DAYS |
|-------|-----------|--------|---------|-------------------|--------------|---------|
| LP-1 | NBR-2 | 89.4 | 96.5 | 91.8 | 90.9 | 91.1 |
| LP-2 | NBR-8 | 96.5 | 109.1 | 104.5 | 97. 3 | 98.4 |
| LP-3 | NBR-9 | 89.5 | 101.6 | 102.5 | 99.1 | 102.0 |
| LP-4 | 1203F60R2 | 103.8 | 101.7 | 101.9 | 76.7 | 88.2 |
| LP-5 | VT-380 | 100.2 | 101.0 | 101. 9 | 98.5 | 100.6 |
| LP-6 | CR-1 | 114.5 | 116.8 | 113.5 | 113.5 | 113.4 |
| LP-7 | CR-2 | 96.0 | 99.9 | 96.2 | 95.3 | 105.3 |
| LP-8 | Viton-1 | 107.9 | 123.0 | 111.7 | 117.1 | 107.6 |
| LP-9 | Viton-2 | 106.5 | 105.2 | 109.7 | 99.2 | 106.1 |
| LP-10 | PE100A027 | 101.5 | 102.4 | 102.5 | 98.9 | 103.7 |

Table 9. Tensile Strength Retention (%) of Elastomers in LP at Room Temperature

| CODE | COMPOUND | 7 DAYS | 14 DAYS | 28 DAYS | 42 DAYS | 70 DAYS |
|-------|-----------|--------|---------|---------|---------|---------|
| LP-1 | NBR-2 | 92.6 | 92.6 | 91.0 | 93.2 | 89.6 |
| LP-2 | NBR-8 | 94.5 | 100.7 | 96.0 | 94.5 | 98.8 |
| LP-3 | NBR-9 | 84.8 | 96.1 | 90.9 | 94.8 | 98.5 |
| LP-4 | 1203F60R2 | 93.9 | 94.7 | 81.6 | 55.3 | 60.5 |
| LP-5 | VT-380 | 98.7 | 102.3 | 101.4 | 105.9 | 102.3 |
| LP-6 | CR-1 | 95.5 | 95.5 | 93.2 | 93.9 | 93.9 |
| LP-7 | CR-2 | 85.4 | 89.9 | 82.8 | 87.3 | 92.6 |
| LP-8 | Viton-1 | 107.6 | 121.8 | 107.6 | 121.8 | 105.9 |
| LP-9 | Viton-2 | 103.8 | 105.5 | 107.7 | 109.3 | 102.2 |
| LP-10 | PE100A027 | 102.4 | 102.4 | 103.7 | 100.4 | 102.4 |

Table 10. Ultimate Elongation Retention (%) of Elastomers in LP at Room Temperature

Nitrile Elastomers

The nitrile elastomers—compounds LP-1, LP-2, and LP-3—performed well at room temperature in LP but deteriorated at elevated temperatures in LP. Figures 11, 14, and 17 show comparisons of the room temperature data to the elevated temperature data of the tensile strength retention, elongation retention, and volume change, respectively. The deterioration of the elastomer that is aged at elevated temperature is clearly seen in the tensile strength retention and elongation retention data. The volume change data shows an increase in the swelling of the samples at elevated temperatures. Nitrile elastomers are not a good material for use with LP at elevated temperatures.

Nitrile/PVC

Compounds LP-4 and LP-5 were nitrile/PVC blends tested in LP. Comparisons of the room temperature *versus* elevated temperature data for tensile strength retention, elongation retention, and volume change are shown in Figures 12, 15, and 18, respectively. The tensile strength retention for compound LP-4 showed the same deterioration for both compounds; however, the elongation retention exhibited much worse degradation when exposed to elevated temperature. The volume change for LP-4 showed more swelling after aging at elevated temperature. Compound LP-5 tensile strength retention and elongation retention clearly showed more deterioration after aging at elevated temperature. The volume change for LP-5 at elevated temperature. The volume change for LP-5 at elevated temperature showed an increased amount of swelling. The deterioration of nitrile/PVC compounds, especially as traced by the elongation retention, would make them unacceptable for use with LP at elevated temperature.

Polychloroprenes

The polychloroprene compounds LP-6 and LP-7 performed well in testing of LP at room temperature and were satisfactory in LP at elevated temperature. Comparisons of compound LP-6's tensile strength retention, elongation retention, and volume change are shown in Figures 12, 15, and 18, respectively. The tensile strength retention and elongation retention show slight losses at elevated temperature. The volume change of compound LP-6 shows an increase in swelling with immersion time at elevated temperature, while the room temperature test fluctuates between swelling and shrinkage.

Compound LP-7's comparison of tensile strength retention is shown in Figure 13. The tensile strength retention of the elevated temperature testing averaged slightly higher values during the test periods. The comparison of the elongation retention is graphed in Figure 16. The room temperature immersion test drops significantly and shows fluctuations averaging about 87% while the elevated temperature test shows elongation retention starting from 100% then decreasing to 87% after 70-day immersion. The most obvious difference occurred in the volume swell data exhibited in Figure 19. The room temperature test fluctuated with both swelling and shrinkage, but less than 1%. The elevated temperature test displayed gradual swelling during all time periods with a final volume swell of 4%.

Although there was more volume swelling in compounds LP-6 and LP-7 when tested at elevated temperatures, the swelling was not severe enough to preclude usage in most sealing applications. The tensile strength retention and elongation retention exhibited basically the same behavior for the tensile strength retention and elongation retention testing. Based on these results, polychloroprenes should be considered for use with LP at room and elevated temperature applications.

Fluoroelastomers

Comparisons for fluoroelastomer compounds LP-8 and LP-9 are shown in Figures 13, 16, and 19. The tensile strength retention (Figure 13) shows the fluctuation of compound LP-8 for both room and elevated temperature testing; however, the room temperature values were all over 100%, while the elevated temperature values averaged 100% with drops to 90% during the testing. The elongation retention (Figure 16) demonstrates the same pattern as the tensile strength retention with approximately the same values. The volume change (Figure 19) showed the largest difference between room and elevated temperature testing for compound LP-8. The room temperature exhibited only shrinkage during testing, while the elevated temperature had increased swelling after 14-day immersion in LP rising to 2.5%.

Compound LP-9's tensile strength retention and elongation retention (Figures 13 and 16, respectively) behaved similarly in both the room temperature and elevated temperature tests.

Here again, the primary difference was in the volume change (Figure 19). The room temperature volume change testing displayed shrinkage, while at elevated temperature there was slight swelling only.

The fluoroelastomer compounds performed well in both room and elevated temperature testing. The volume change for room temperature may make some sealing applications questionable but the swelling observed at elevated temperature should not be detrimental. Fluoroelastomers should be considered for LP applications.

Halogenated Butyl

No graphs for comparison purposes were produced for the halogenated butyl compound LP-10 since very little data was collected during the elevated temperature test. Compound LP-10 displayed very consistent results for the room temperature testing and was a good candidate material. However, the somewhat explosive reactivity of this material with LP at elevated temperature makes it totally unsuitable for use with LP at higher temperatures. This material might be considered for room temperature or low temperature applications, but the conditions must be well defined.

SECTION III — CONCLUSIONS

Based on the results obtained in this study, two types of polymers are still deemed acceptable for use with LPs. Polychloroprenes and fluoroelastomer compounds showed resistance to LP even at an elevated temperature. Before their use in any LP application, especially sealing applications, thorough testing should be performed to ascertain that all compounding ingredients are compatible with the LP fluid.

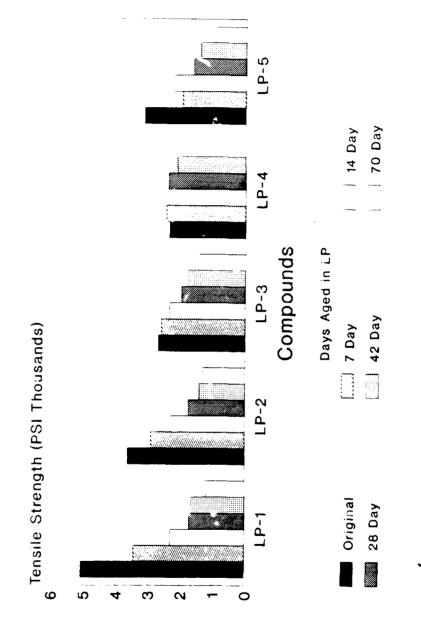
Several elastomers lost enough of their properties to make them unacceptable for use with LP at elevated temperatures. Both nitrile and nitrile/PVC blends lost their physical properties to a point where failure could occur.

One elastomer tested, a halogenated butyl, reacted with the LP at elevated temperature causing the tops of the test tubes to blow off. The reaction causing this is unknown and its determination is beyond the scope of this work. However, such a reaction would prevent halogenated butyl elastomers from being used with LPs at elevated temperatures.

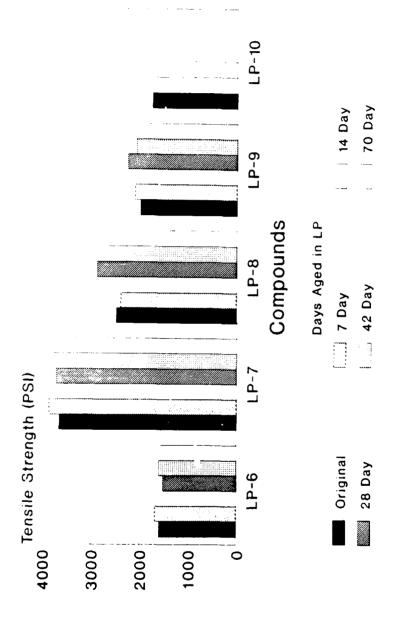
Another concern during this testing is the contamination of the LP by the elastomer. Fourier Transform Infrared Spectroscopy (FTIR) analysis will be used to try and identify any foreign chemicals in the LP after exposure to an elastomer. At the time of this writing, the FTIR work is ongoing. A report concerning the FTIR test will be forthcoming.

APPENDIX OF FIGURES

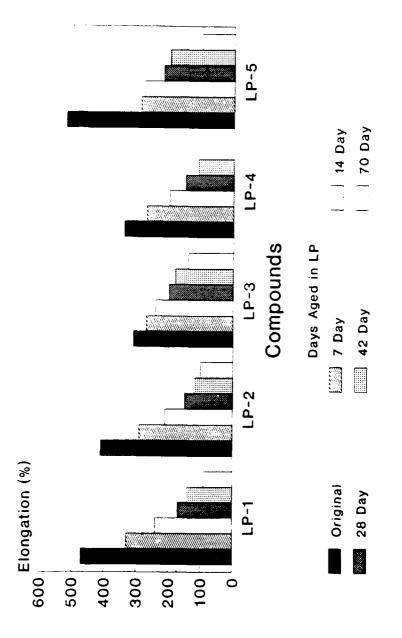
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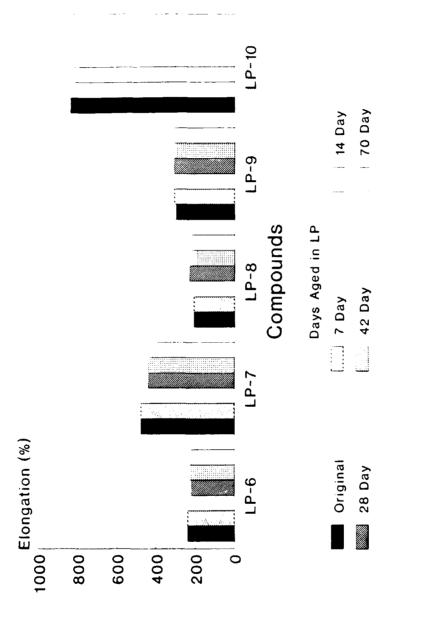




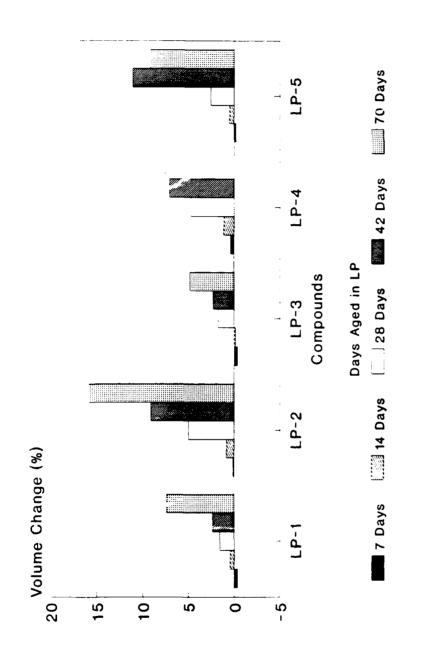














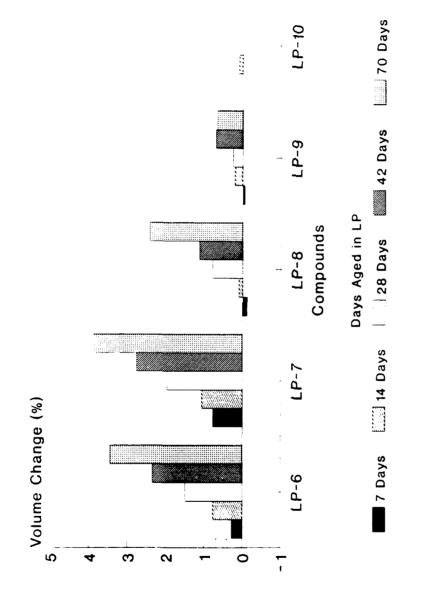
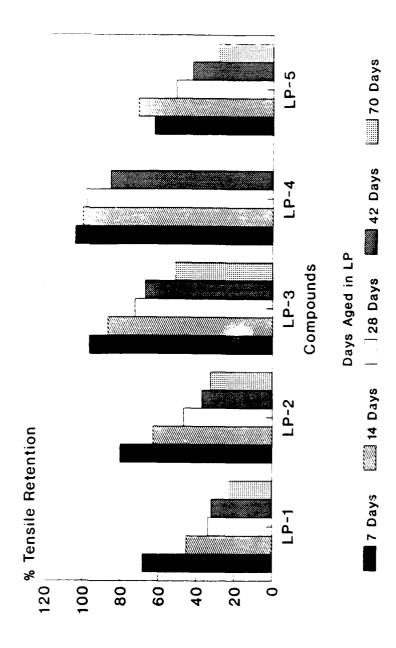
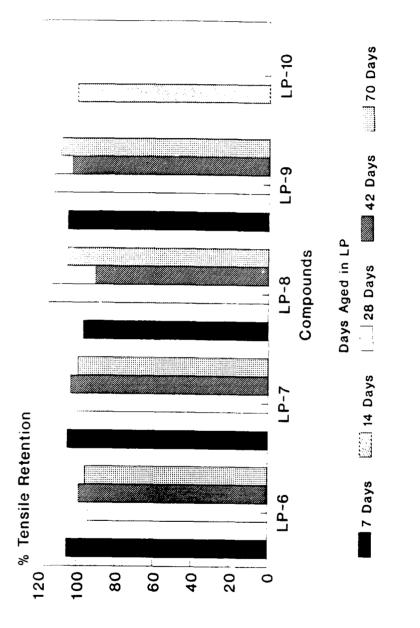


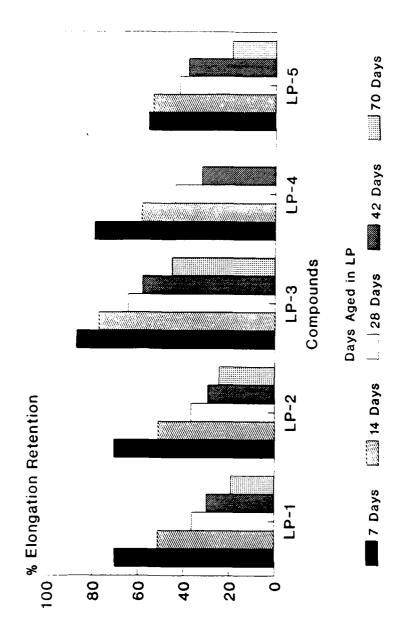
Figure 6. Volume Change for Elastomers Immersed in LP at 150°F



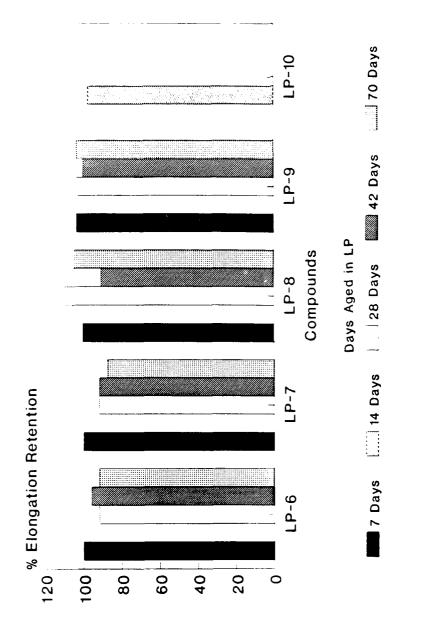














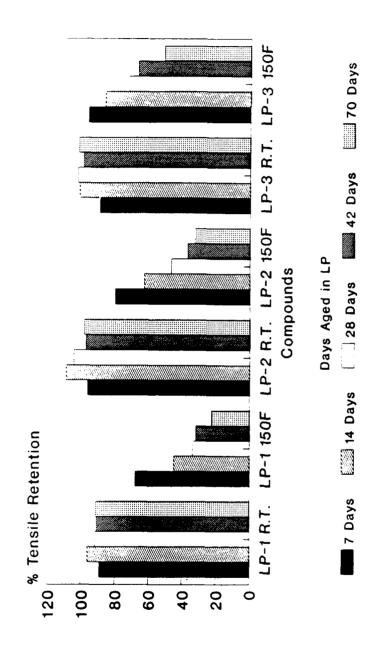
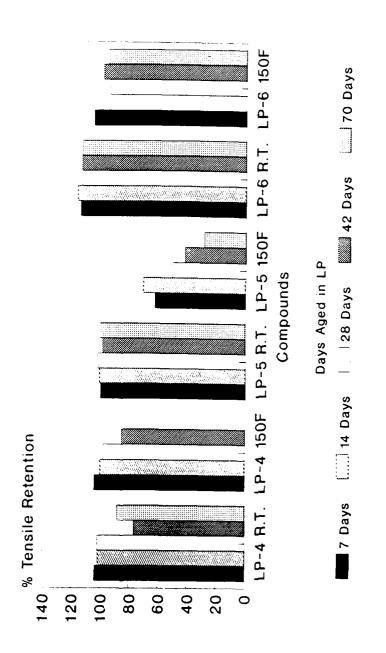


Figure 11. Tensile Retention of Elastomers Comparison of Room Temperature to 150°F After Immersion in LP





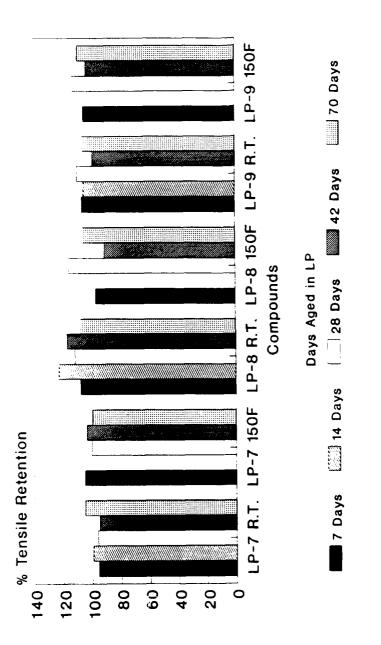
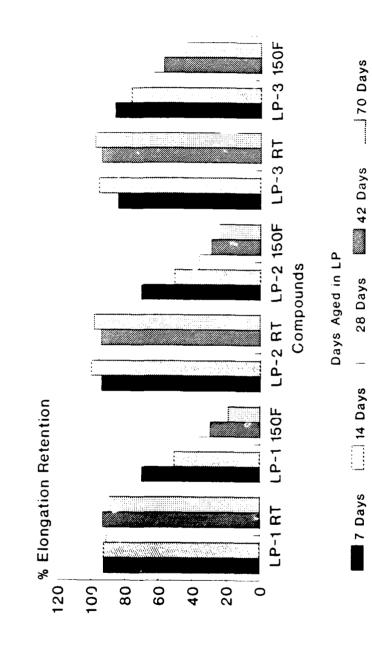


Figure 13. Tensile Retention of Elastomers Comparison of Room Temperature to 150°F After Immersion in LP





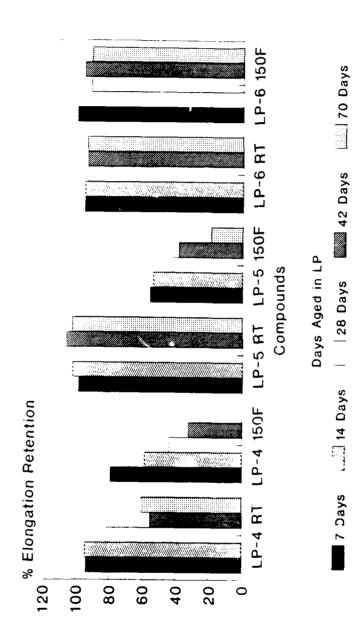


Figure 15. Elongation Retention of Elastomers Comparison of Room Temperature to 150°F After Immersion in LP

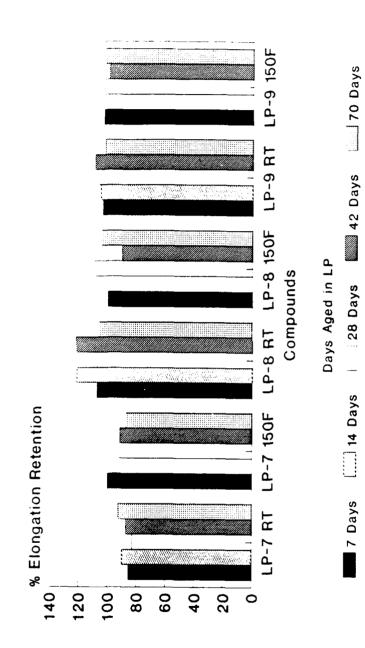


Figure 16. Elongation Retention of Elastomers Comparison of Room Temperature to 150°F After Immersion in LP

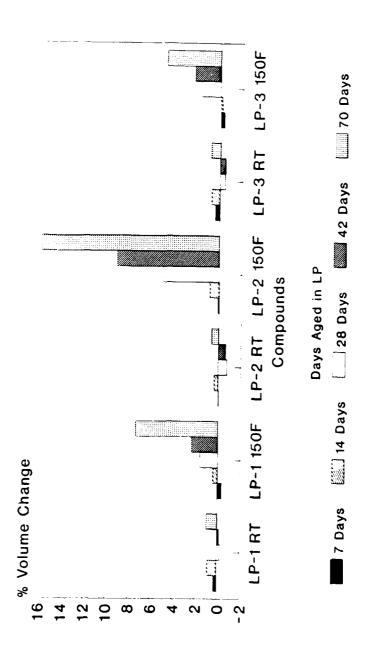


Figure 17. Volume Change of Elastomers Comparison of Room Temperature to 150°F After Immersion in LP

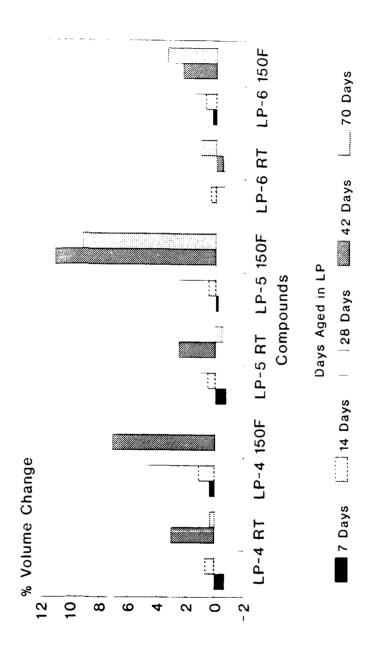


Figure 18. Volume Change of Elastomers Comparison of Room Temperature to 150°F After Immersion in LP

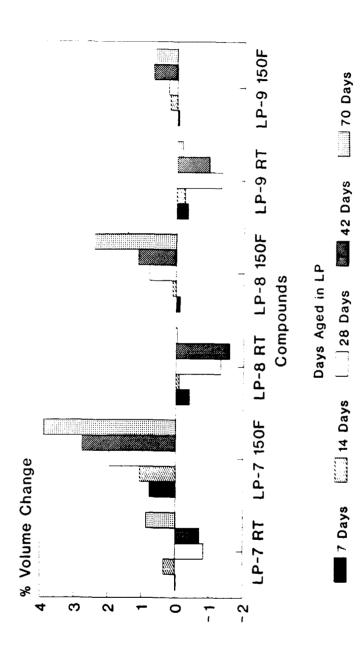


Figure 19. Volume Change of Elastomers Comparison of Room Temperature to 150°F After Immersion in LP

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