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TECHNICAL INFORMATION  
FOR THE APPLICATION OF FLUORORUBBER D<sup>4</sup> (POLY-FBA)  
IN CHEMICAL SEPARATIONS PLANTS

By

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Process Equipment Development  
Chemical Research & Development  
HANFORD LABORATORIES

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TECHNICAL INFORMATION  
FOR THE APPLICATION OF FLUORORUBBER 1F4 (POLY-FBA)  
IN CHEMICAL SEPARATIONS PLANTS

INTRODUCTION

Fluororubber 1F4, also known as Poly-FBA is a product of Minnesota Mining and Manufacturing Company. It is the polymer of 1, 1-dihydroperfluorobutyl acrylate.

The outstanding resistance of this material to organic solvents, including aromatics, ketones, and chlorinated hydrocarbons has prompted the Process Equipment Development Operation to evaluate it for application in chemical separations plants.

SUMMARY AND CONCLUSIONS

Fluororubber 1F4 is resistant to 10 percent and 60 percent nitric acid, 50 percent sodium hydroxide, carbon tetrachloride, Recuplex CAX, Purex HAX and distilled water at room temperature. Exposure to hexone caused a 25 percent increase in length and some softening. It will not tolerate boiling 60 percent nitric acid.

Exposure in a gamma field had little effect on the chemical compatibility until the exposure reached  $10^8$ r. A total exposure of  $10^7$ r caused the samples to degrade quite severely on the surface when submerged in the test solution. The changes in hardness and length were smaller after irradiation than before.

1F4 rubber also tends to cold flow and take a permanent set. These characteristics, coupled with the surface weakness upon irradiation, will severely limit the field of useful application of this elastomer. A completely confined application, such as "O" rings or gaskets for tongue and groove joints offers the most promise. It is doubtful that Fluororubber 1F4 will be useful in any service where total irradiation will exceed  $10^8$ r.

DISCUSSIONManufacturer's and Literature Information

Minnesota Mining and Manufacturing Company's Fluororubber 1F4 is a high molecular weight synthetic rubber which contains over 50 percent fluorine. It is resistant to high temperatures, oils, solvents, and many chemicals. 1F4 rubber is saturated and as a result cannot be vulcanized by the usual sulfur recipes. Carbon black or an inorganic pigment is required for reinforcing. The material is furnished to the fabricator as a gum or latex. It can be handled with conventional rubber compounding techniques on standard equipment.

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Fluororubber 1F<sup>4</sup> shows good resistance to phosphate esters; although there is an initial drop in hardness and strength, longer aging effects little additional change. Immersion in 90 percent nitric acid produces a gradual chemical attack, but rubbery qualities and satisfactory chemical properties are retained for long periods.

The rubber is essentially immune to ozone attack at room temperature. It deteriorates more rapidly in high temperature air than in liquid media. The presence of oxygen causes cure reversion or molecular weight degradation. It is resistant to a wide range of organic solvents, synthetic lubricants, hydraulic fluids and oils at temperatures up to 400° F. 1F<sup>4</sup> rubber is very difficult to dissolve but many solvents will cause some swelling. The loss in physical properties which accompanies this swelling is not usually severe.

#### LABORATORY TEST RESULTS

The samples used in this series of tests were purchased from Stillman Rubber Company. No specific service requirement was specified on the purchase requisition. The tests conducted include (1) static immersion at room temperature in aqueous solutions of nitric acid, caustic soda, and in organic solvents; (2) static immersion in 60 percent nitric acid at its boiling point; (3) exposure to a gamma source followed by the same tests as in (1) above.

Samples for test (1) and (2) were exposed as received from the manufacturer. They were immersed in the test solution and not removed for remeasurement until the completion of the test. For test (3) samples were exposed to a cobalt-60 source to a total dosage of 10<sup>6</sup>, 10<sup>7</sup>, and 10<sup>8</sup>r gamma and then procedure of test (1) repeated for each exposure level. All tests, with the exception of test (2) above, were for 28 days. Test (2) static immersion in boiling 60 percent nitric acid was discontinued after about ten minutes when it became obvious that the rubber was failing.

In 60 percent nitric acid at room temperature, Fluororubber 1F<sup>4</sup> softened from 78 to 37 Duro "A" after 28 days, and swelled 12 percent in length. In 10 percent nitric acid for 28 days, the sample softened to Duro "A" 65 and swelled only 3 percent. The changes caused by 50 percent caustic soda were negligible. Carbon tetrachloride caused 1F<sup>4</sup> rubber to soften to 58 Duro "A" and to increase 10 percent in length. Purex HAX also caused a softening to 58 Duro "A" with only 4 percent increase in length. Both Recuplex CAX and hexone caused a softening to 50 Duro "A". The sample in CAX swelled only 4 percent while that in hexone swelled 25 percent. In distilled water, 1F<sup>4</sup> rubber softened to 67 Duro "A" and swelled 4 percent.

The results obtained when the samples were irradiated to 10<sup>6</sup> and 10<sup>7</sup>r were not significantly different from the test of the unirradiated samples except that samples exposed to 50 percent caustic soda solutions tended to degrade on the surface. The samples which received a dosage of 10<sup>8</sup>r were damaged severely on the surface when immersed in the test solutions, 10 percent nitric acid and carbon tetrachloride excepted.

In spite of the fact that the surface of the samples exposed to  $10^8$ r was crumbly and rubbed off easily, the changes in hardness and in length were smaller than for the unirradiated samples of the material. Even distilled water caused the  $10^8$ r sample to swell and the surface was severely damaged (see Tables I and II, and Figures 1 and 2 for detailed results).



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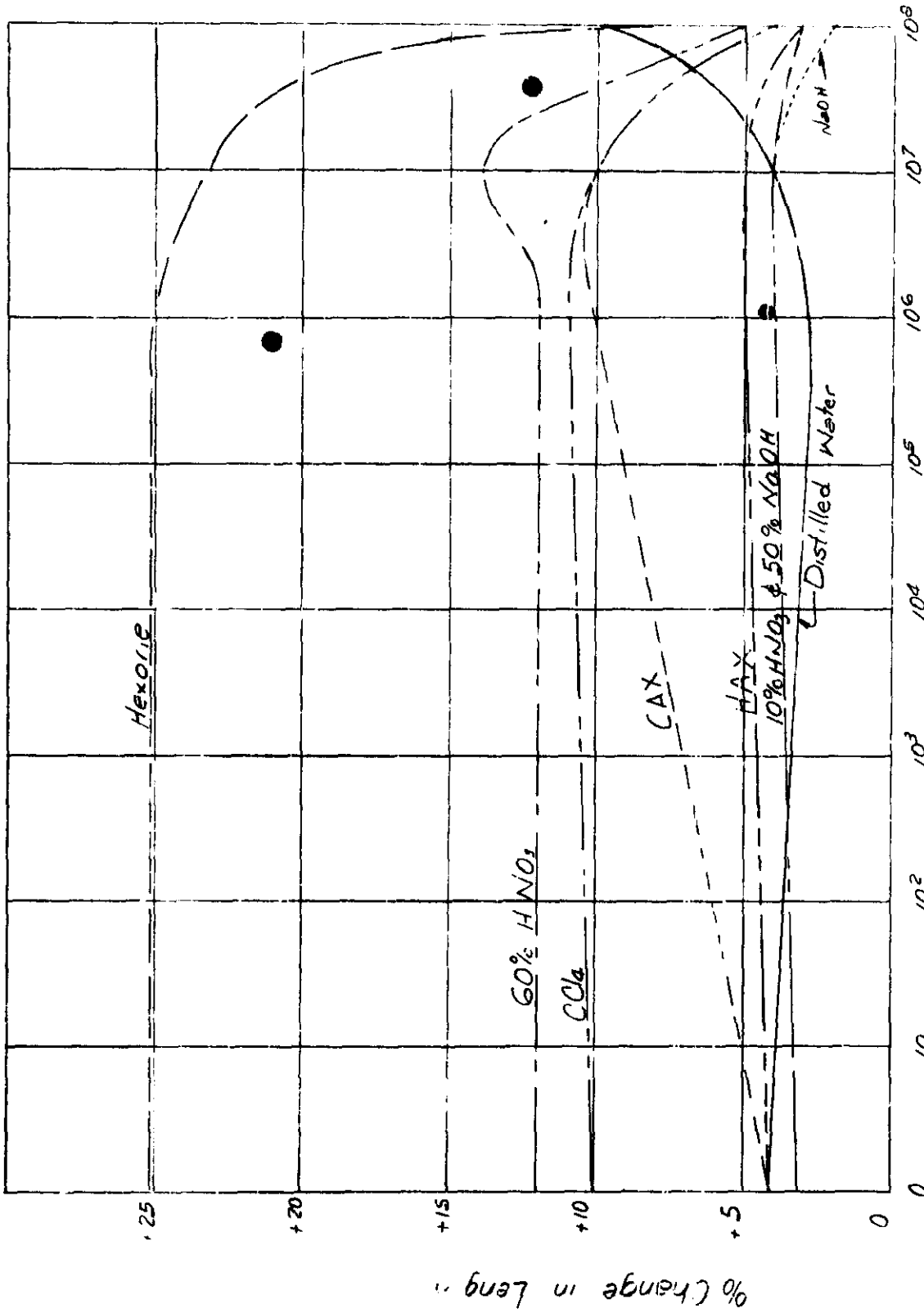
TABLE IChanges in Hardness of Fluororubber 1F4  
After 28 Days Exposure to Test Solution

|                        | <u>0</u> | <u>10<sup>6</sup></u> | <u>10<sup>7</sup></u> | <u>10<sup>8</sup></u> |
|------------------------|----------|-----------------------|-----------------------|-----------------------|
| Air                    | 78       | 78                    | 81                    | 92                    |
| 60% HNO <sub>3</sub>   | 37       | 30                    | 30                    | 55                    |
| 10% HNO <sub>3</sub>   | 65       | 64                    | 67                    | 81                    |
| 50% NaOH               | 80       | 72                    | 75                    | 92                    |
| CCl <sub>4</sub>       | 58       | 55                    | 60                    | 85                    |
| CAX                    | 51       | 45                    | 50                    | 78                    |
| HAX                    | 58       | 57                    | 62                    | 80                    |
| Hexone                 | 50       | 48                    | 52                    | 78                    |
| Dist. H <sub>2</sub> O | 67       | 62                    | 67                    | 74                    |

TABLE IIPercent Linear Expansion of Fluororubber 1F4  
After 28 Days Exposure to Test Solutions

|                        | <u>0</u> | <u>10<sup>6</sup></u>  | <u>10<sup>7</sup></u> | <u>10<sup>8</sup></u>         |
|------------------------|----------|------------------------|-----------------------|-------------------------------|
| Air                    | 0        | 0                      | 0                     | 0                             |
| 60% HNO <sub>3</sub>   | 12       | 12                     | 14                    | 5 (Surface<br>Damage)         |
| 10% HNO <sub>3</sub>   | 3        | 4                      | 4                     | 3                             |
| 50% NaOH               | 3        | 4 (Su. face<br>Damage) | 4 (Surface<br>Damage) | 2 (Surface<br>Damage)         |
| CCl <sub>4</sub>       | 10       | 11                     | 10                    | 4                             |
| CAX                    | 4        | 10                     | 10                    | 4 (Surface<br>Damage)         |
| HAX                    | 4        | 5                      | 5                     | 3 (Surface<br>Damage)         |
| Hexone                 | 25       | 25                     | 23                    | 8 (Surface<br>Damage)         |
| Dist. H <sub>2</sub> O | -        | 3                      | 4                     | 10 (Surface Badly<br>Damaged) |

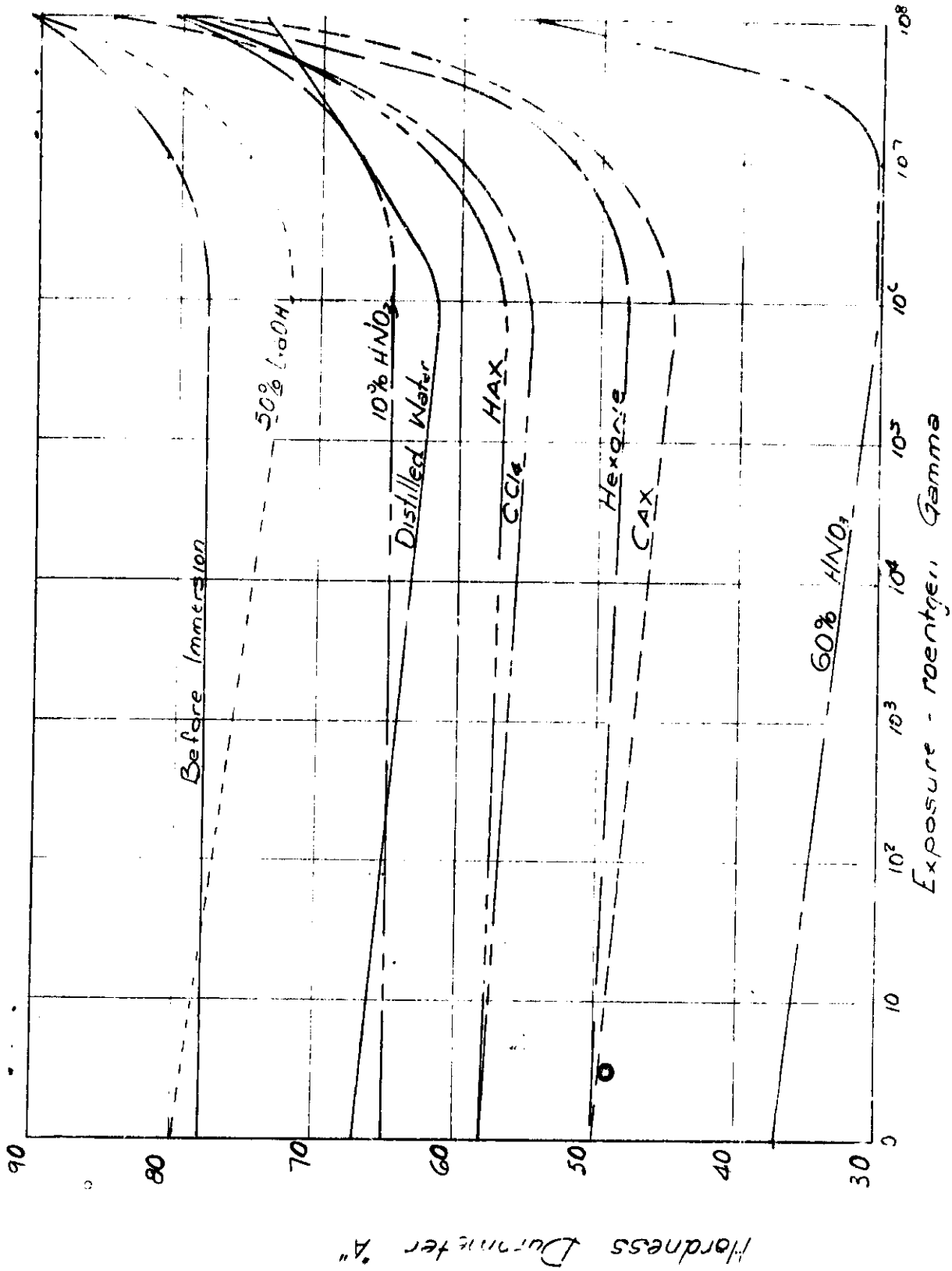
FIGURE 1



Gamma Exposure (r) Before Static Immersion

Fluoro Rubber 1F4 Change in Length of Irradiated Samples  
After 20 Days Static Immersion at Room Temperature

**FIGURE 2**



Fluoro Rubber 1F4  
Hardness of Irradiated Samples After  
28 Days Room Temperature Static Immersion