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# Flooded Cell Permeation Testing of Elastomers

James M. Sloan and Stephen C. Bodnar

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13. ABSTRACT (Maximum 200 words) Permeation studies were carried out using a flooded radian test cell to follow the diffusion of an organic liquid through a polymer barrier. The experimental apparatus consists of a MiniCAMS (Miniature Continuous Air Monitoring System) equipped with a sulphur detector which has the capability of detecting nanogram quantities of permeant at breakthrough. The results yield a time dependent sigmoidal curve that allows determination of the solubility and diffusion coefficients as well as a breakthrough time. DMSO was selected as the test permeant as it is a low swelling solvent which is very sensitive toward detection by the sulphur GC detection system. This method was used to characterize the permeation properties of a number of elastomer systems including NR, SBR, BR, and IIR.					
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- Contents

	Page
Introduction.....	1
Background.....	1
<b>Experimental</b>	
Apparatus.....	2
Materials.....	3
Results and Discussion.....	3
Conclusions.....	4
References.....	5

**Figures**

1. Schematic of automated permeation testing apparatus.....	6
2. Permeation rate-time curve for DMSO through natural rubber.....	6
3. Permeation rate-time curve for DMSO through styrene-butadiene rubber.....	7
4. Permeation rate-time curve for DMSO through neoprene.....	7
5. Permeation rate-time curve for DMSO through nitrile rubber.....	8
6. Permeation rate-time curve for DMSO through fluorocarbon rubber.....	8
7. Permeation rate-time curve for DMSO through hydrin.....	9
8. Polarity index versus solubility constant for six elastomers. The polarity index for FC has been estimated at 5.0 based on permeation curve.....	10
9. Polarity index versus permeation constant for six elastomers. The polarity index for FC has been estimated at 5.0 based on permeation curve.....	10
10. The dependence of the diffusion constant with polymer mobility. Polymer mobility is measured as T-Tg.....	11

**Tables**

1. Permeation data.....	12
2. Polarity index for various liquids.....	12
3. Polarity index for elastomers.....	12

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## Introduction

Permeation testing is becoming a routine method for determining the chemical resistance of polymer barriers. However, the ability to perform these measurements easily, confidently, and accurately remains a challenge. This is made more difficult still, by the large diversity of chemicals requiring evaluation.

Most of the earlier [1-4] studies of permeation have focused on the transport of gases through a polymer barrier. Many of the liquid permeation studies are carried out using the immersion into liquid method, which reveals little about the breakthrough [5,6]. Relatively few have been concerned with the permeation of liquids [7].

Permeation is most often described as the transport of molecules through a barrier of finite thickness. This process is primarily governed by diffusion of the permeant, which is defined by a diffusion coefficient and solubility constant. The advantages of permeation studies are that the diffusion and solubility constants can be measured directly and the breakthrough time of each permeant/barrier pair can be obtained.

The present study focuses on an automated dynamic method of studying the mechanics of permeation and diffusion through a series of various elastomers. This technique allows determination of breakthrough times, solubility, and permeation rates.

## Background

Permeation is defined as a process by which a diffusing molecule (permeant) is transported through a barrier. The mechanism of permeation is usually described as a three-step process which involves: (a) the sorption of the permeant on the outside surface, (b) diffusion through the bulk membrane, and (c) desorption of permeant from the opposite surface into a collecting medium [8].

The fundamental parameters that govern the interactions of a permeant liquid and a polymer barrier material are the diffusion and solubility coefficients. For barrier polymers with a uniform thickness,  $L$ , and a constant concentration of challenge liquid, Fick's first law takes the form [9]:

$$J = \frac{DS}{L}$$

where  $S$  is the solubility in  $\text{g/cm}^3$ ,  $D$  is the diffusion coefficient in units of  $\text{cm}^2/\text{sec}$ , and  $J$  is the flux in  $\text{g/cm}^2 \cdot \text{sec}$ . If one considers the simplest case, unidirectional diffusion, Fick's Law becomes [10]:

$$J = -D \frac{dc}{dx}$$

where the derivative,  $\frac{dc}{dx}$ , is the concentration gradient in the thickness direction.

For the flooded cell permeation experiments conducted in this report, the steady-state permeation rate can be determined by [11,12]

$$J = \frac{FC}{A}$$

where  $F$  is the flow rate of nitrogen in ml/min,  $A$  is the surface area of sample exposed to the permeant, and  $C$  in mg/ml is the steady-state concentration of permeant in the collection medium. This equation can be solved assuming the following conditions are met. Both the diffusion coefficient and liquid concentration are constant, and the concentration of the permeant on the sweep surface is maintained at zero. The diffusion and solubility coefficients can be calculated by using these approximation equations.

$$D = \frac{L^2}{7.2t_{1/2}}$$

and

$$S = \frac{LQ}{D}$$

By constructing a plot of permeation rate versus time, one can use a simple graphical technique to estimate values for  $t_{1/2}$  and  $Q$ . The value  $t_{1/2}$  is defined as the time it takes to reach half of the steady-state rate.  $Q$ , is the steady-state rate permeated in units  $g/cm^2 \cdot sec$ .

## Experimental

### Apparatus

A schematic drawing of the experimental apparatus is shown in Figure 1. The 1.5-inch diameter ( $4.9 \text{ cm}^2$  exposed area) test specimens were prepared for testing by using a circular cutting die. The permeation test cell used in the experiment was purchased from the Radian Corporation of Austin, TX. The entire permeation cell is located in an isothermal chamber maintained at  $37^\circ\text{C}$ . The analysis is run for a minimum of six hours and a data point is acquired every three minutes.

The test specimen is clamped between two stainless steel components, forming two compartments. The top compartment is called the challenge reservoir. It holds approximately two milliliters of permeant liquid. The bottom compartment, named the sweep volume, is continuously swept by clean air at a flow rate of  $770 \pm 50 \text{ ml/min}$ . The test procedure is now ready to begin.

The permeant is allowed to diffuse from the challenge reservoir surface of the sample to the sweep volume side. The sweep volume is carried through a short length of heated tubing to a Sample Selection System. This piece of equipment allows the user to pass all or a known fraction of the permeant stream onto the Stream Selection System. Here, the computer follows a pre-programmed pattern to select which test cell (if more than one is being used) the gas chromatograph will receive. Once the cell is chosen, the stream is pulled by vacuum onto a cold ( $50^\circ\text{C}$ ) Tenax solid sorbent tube. The sorbent is then flashed at  $240^\circ\text{C}$ . Nitrogen, at a flow rate of  $300 \text{ ml/min}$ , is used to push the flashed molecules into the cold GC column. The column temperature is then ramped from  $50$  to  $200^\circ\text{C}$  within 30 seconds. The exit of the column is connected to a flame photometric detector, which has a sensitivity of one nanogram to sulfur containing compounds. A value is then received by the computer and interpreted.

The system is calibrated by injecting known amounts of DMSO and measuring the resultant peak height. A calibration curve of peak height response to DMSO amount yields a straight line with a correlation coefficient confidence  $\geq 0.98$ .

## Materials

Six elastomers were evaluated using the previously described system. Included were:

1. sulphur cured natural rubber (NR)
2. thiourea cured hydrin (EC) elastomer
3. oxide cured neoprene (CR)
4. sulphur cured styrene-butadiene rubber (SBR)
5. sulphur cured nitrile rubber (NBR)
6. cured fluorocarbon elastomer (FR)

All elastomers contained carbon black as a reinforcing filler. All elastomeric compounds were formed in 6 x 6 inch sheets by Smithers Scientific and cut prior to testing into 1.5-inch diameter disks. The disk's surfaces were cleaned with isopropyl alcohol.

## Results and Discussion

Figure 2 shows a typical steady-state permeation curve for DMSO permeating through a 12.1-mil thick natural rubber specimen. The curve shows the permeation rate (ng/min) as a function of time (min) and is notably sigmoidal in shape. In general, the curve can be broken down into three discrete sections, (1) lag time, where no permeant has broken through, (2) a transient region, where a steady increase in permeation rate is seen, and finally (3) a plateau of steady-state permeation. The breakthrough time is defined as the duration from time zero to initial detection of permeation within the detection limit of machine.

Figure 3 shows a typical permeation curve for SBR/DMSO. This curve closely resembles the curve obtained for NR. Both of these curves follow classical Fickian diffusion.

Figures 4, 5, and 6 show the permeation curves obtained for CR, NBR, and FR. The curves begin with consistent lag times, followed by a steady increase in permeation rate similar to curves obtained for NR and SBR. However, the permeation rate does not reach a steady-state level. Instead, the permeation rate reaches a maxima then steadily declines. This kind of behavior has been observed previously by other researchers and may be attributed to possible structural modifications during the course of the experiment [13]. These curves all exhibit large permeation rates. These rates can generally be attributed to moderate swelling effects of the polymer membranes.

Figure 7 shows the permeation curve for EC. This curve exhibits a very sharp increase in permeation rate until steady-state is quickly achieved. This can be attributed to a very high degree of swelling. Mobility throughout the polymer is so greatly increased, diffusion is almost instantaneous. From these graphs, the following trend in swelling can be determined.

## Swelling EC>FR>NBR>CR>SBR = NR Non-Swelling

Table 1 summarizes the permeation data for the six elastomers evaluated. In this table we define the parameter P, as the permeation constant. This is simply the product of the diffusion and solubility constants,  $P = DS$ . A distinctive trend can be drawn when comparing the elastomers. The permeation rate is slow for NR and SBR, but significantly faster for FR and EC. This is also seen in the solubility and permeation constants. One possible explanation for this trend is the polarity interactions between the liquid and the polymers.

It has been reported that the solubility and diffusibility are governed by polymer-penetrant interactions [13,14]. Liquid penetrants having polarity indices that most closely match the polarity index of the polymer exhibit faster permeation rates [15]. Therefore, polar compounds permeate faster through polar polymers and non-polar compounds would permeate faster through non-polar polymers. The liquid penetrant used in this experiment was DMSO, a highly polar compound assigned a polarity index of 6.5. Table 2 lists the polarity index for some common solvents. These values can be compared to the polarity index of individual elastomers to estimate permeation rates for any polymer-penetrant combination. Table 3 lists the estimated polarity index for the elastomers used in this study.

An attempt to correlate the solubility and permeation constants with the polarity index is shown in Figures 8 and 9. A general experimental trend is seen between the solubility parameter and polarity index. An even better correlation is found between the permeation constant and polarity index. The polarity index for the fluorocarbon rubber is not known. It has been estimated to have an index of approximately 5.0 from the permeation rate-time curve.

In the case of gas diffusion in an elastomer, it has been observed that the diffusion constant varies with polymer mobility, which is dependent upon the difference in the experimental temperature and the polymer's glass transition temperature. Figure 10 presents the change in D with the polymer mobility as estimated by  $T-T_g$ . A linear trend is observed for the six elastomers tested over the 37° to 110°C temperature range. These results suggest that polymer mobility is the key determinant in the liquid and gas diffusion process.

## Conclusions

An automated experimental method is described which effectively can measure diffusion, solubility, and permeation rates through rubbery protective membranes. This technique, depending on the detector of choice, can accurately measure permeation rates down to nanogram levels. This approach offers the potential of studying permeating liquid mixtures.

Utilizing this technique we have studied the diffusion and permeation of six elastomers. Using DMSO, a highly polar liquid, as a permeant it was found that the more polar polymers (FC and EC) exhibited faster diffusion and permeation rates, while the less polar polymers (NR and SBR) showed the opposite.

The permeation performance of these rubbers has been related to the polarity index, which is a measure of the polarity exhibited by the base polymer. Polymers with a high polarity index showed faster permeation rates while non-polar polymers showed slower permeation rates. Additionally, the diffusion is affected by polymer mobility. A linear trend of the diffusion constant with  $T-T_g$  is observed.



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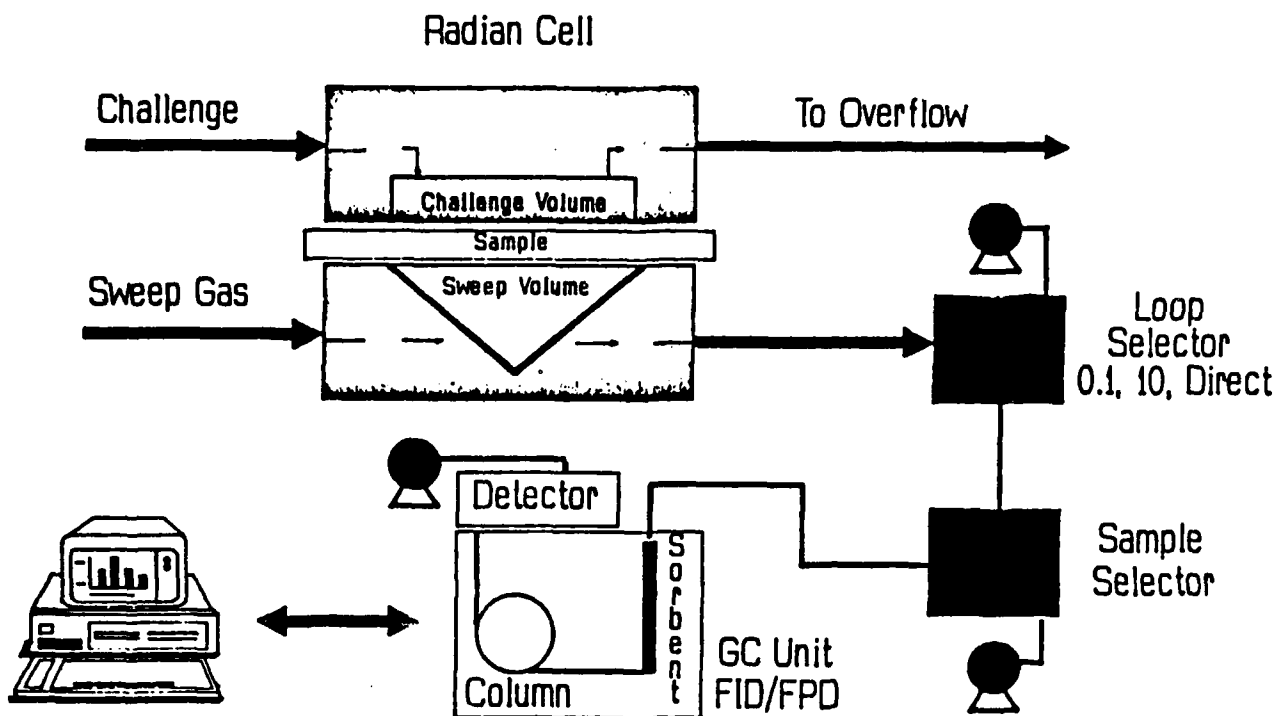


Figure 1. Schematic of automated permeation testing apparatus.

### Natural Rubber/DMSO, 10%, 12.1 mils

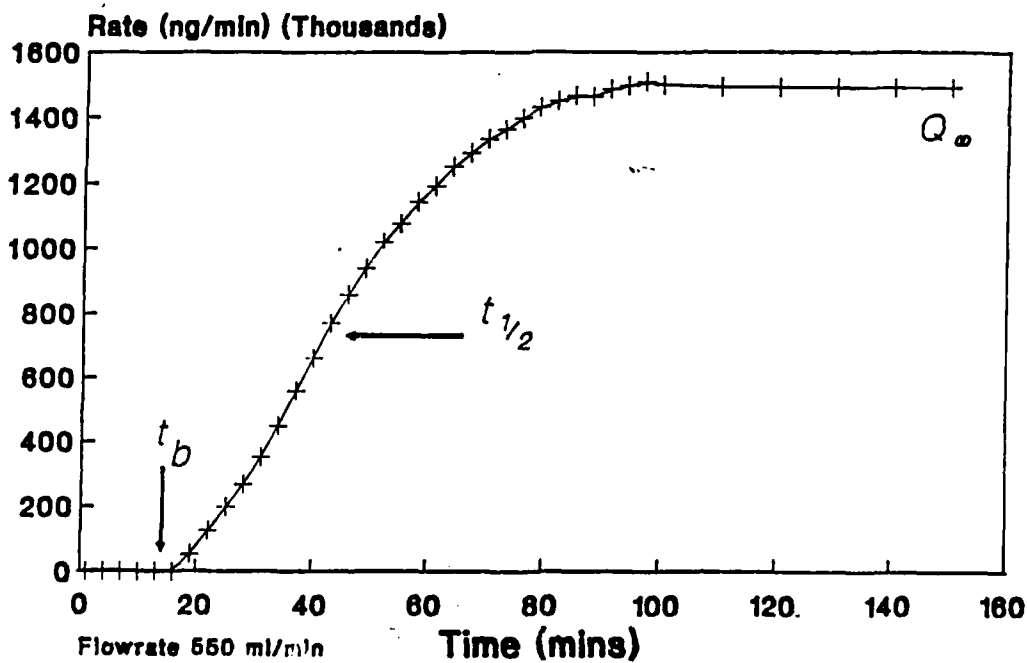


Figure 2. Permeation rate-time curve for DMSO through natural rubber.

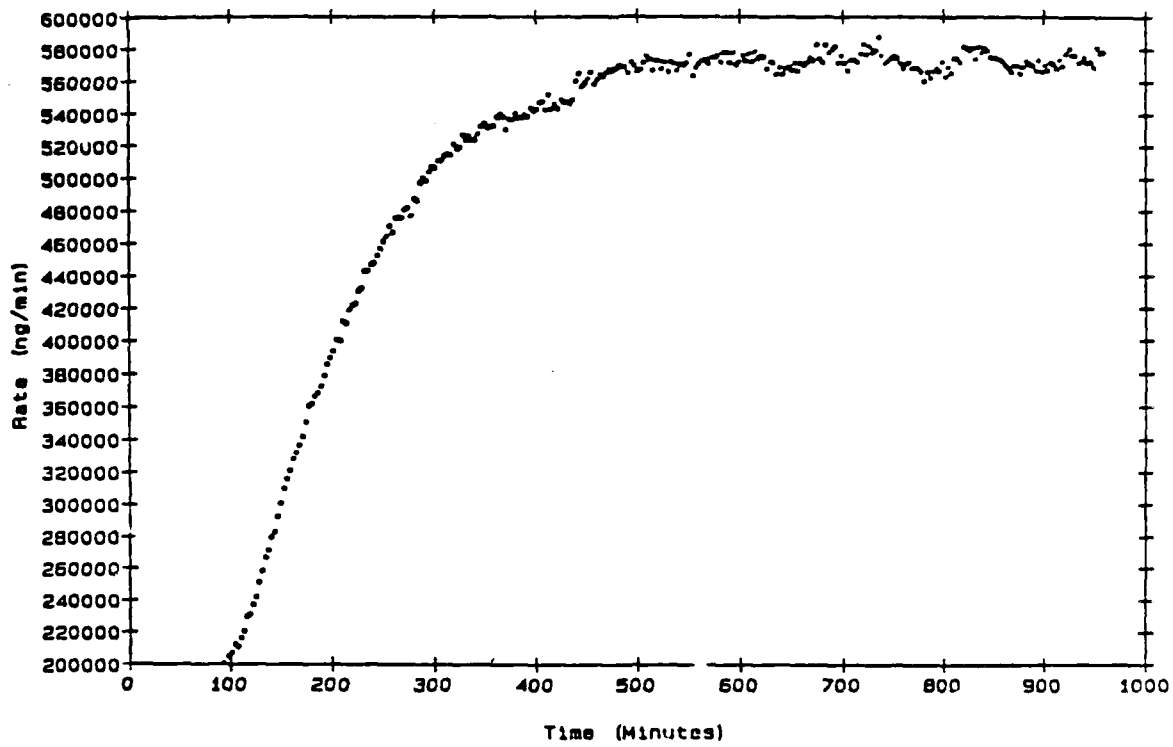


Figure 3. Permeation rate-time curve for DMSO through styrene-butadiene rubber.

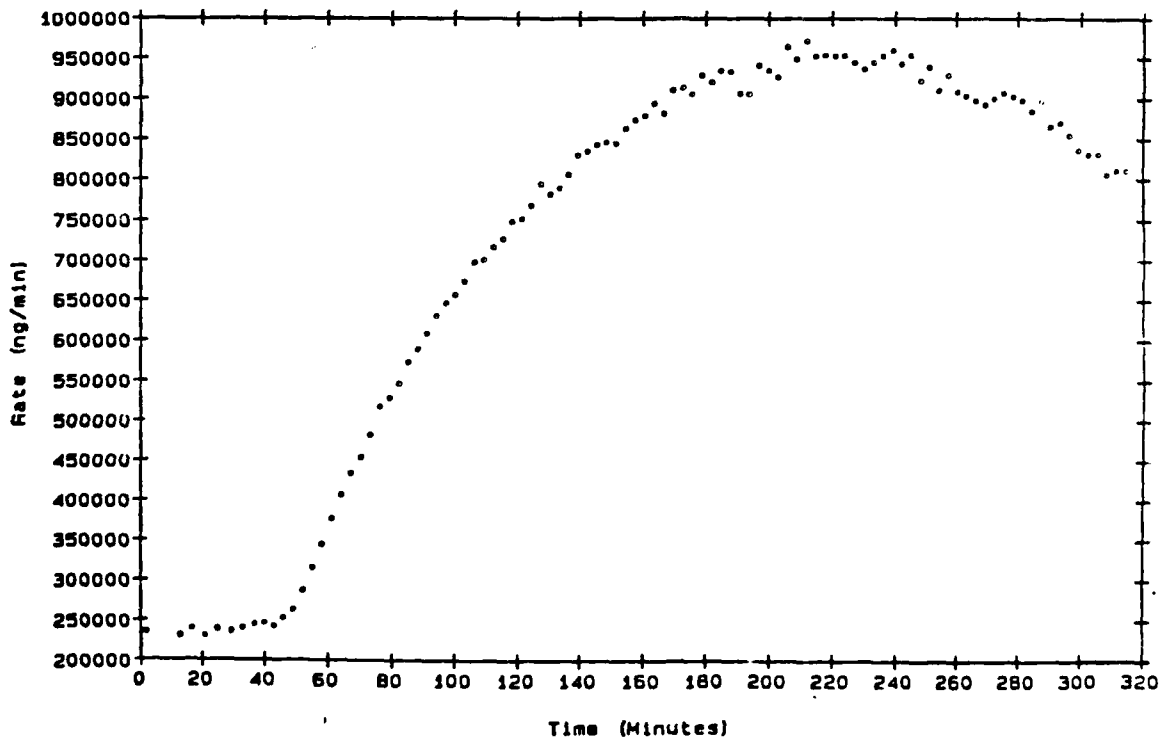


Figure 4. Permeation rate-time curve for DMSO through neoprene.

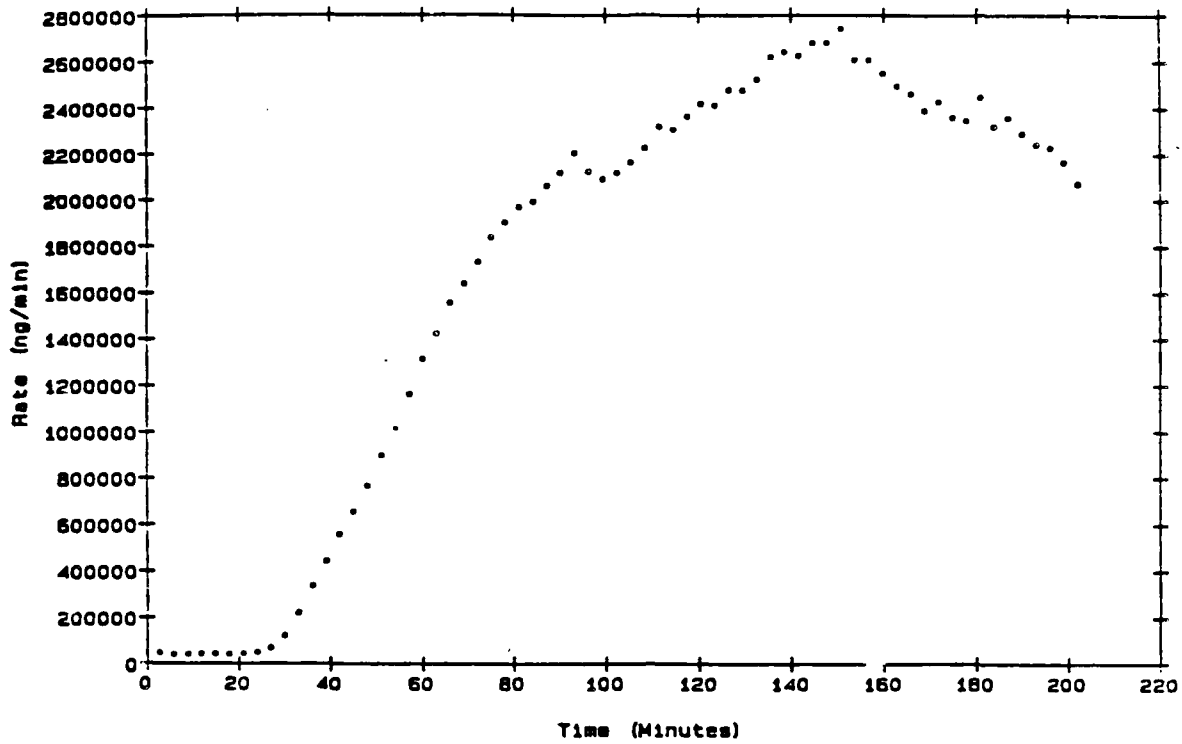


Figure 5. Permeation rate-time curve for DMSO through nitrile rubber.

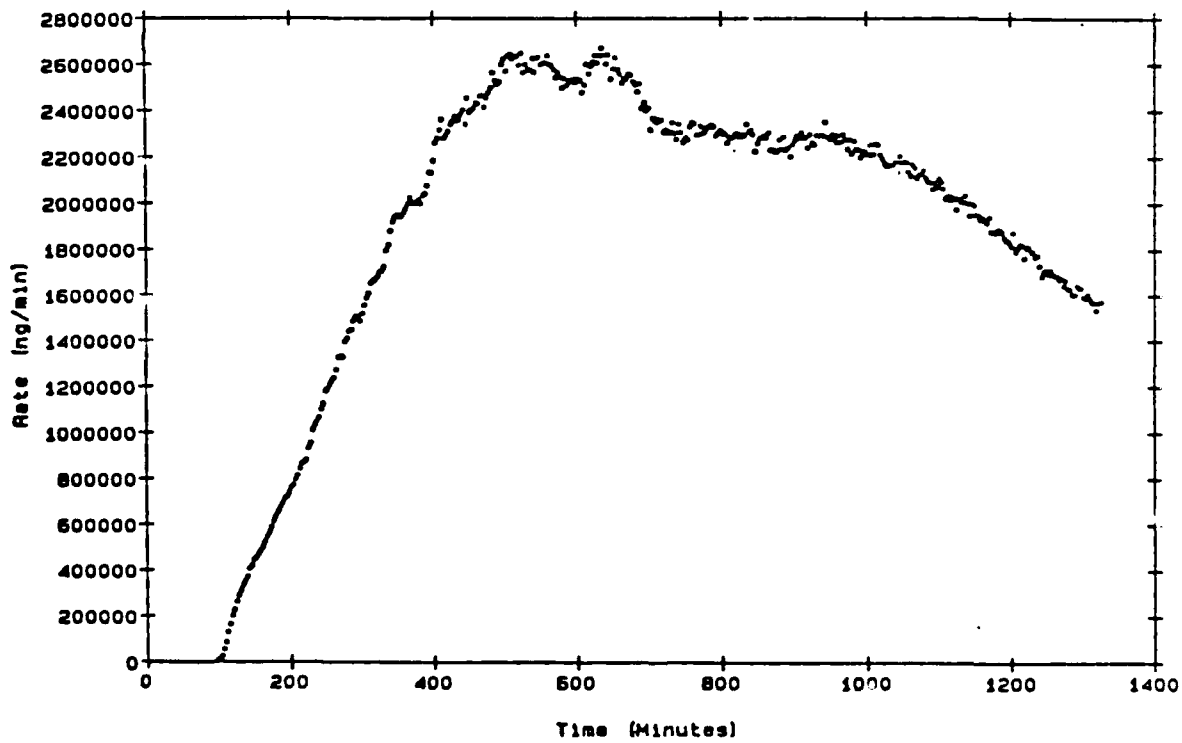


Figure 6. Permeation rate-time curve for DMSO through fluoro-carbon rubber.

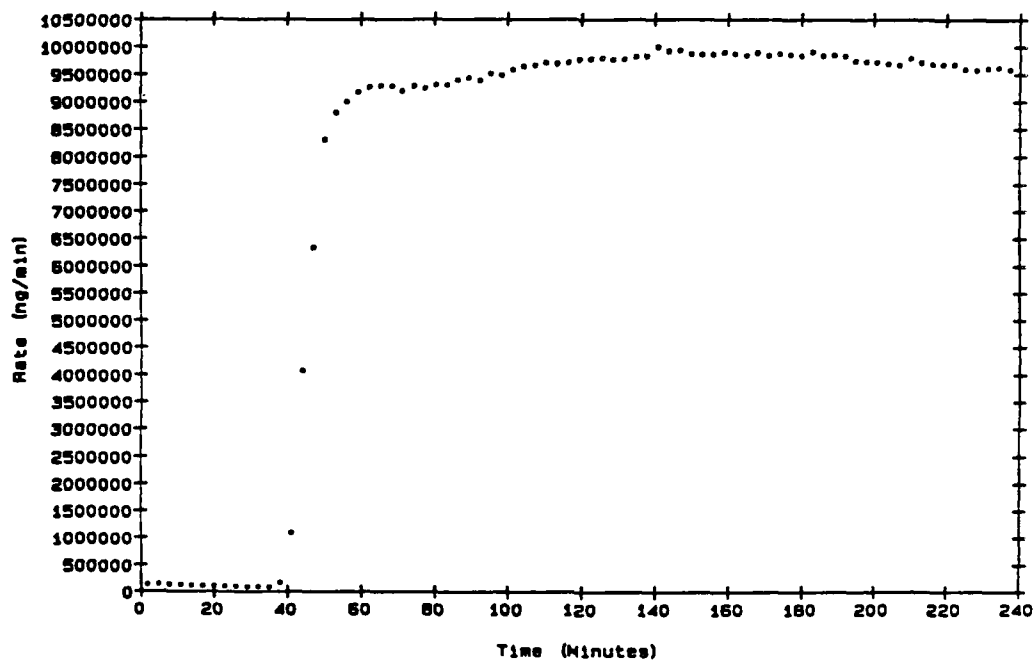


Figure 7. Permeation rate-time curve for DMSO through hydrin.

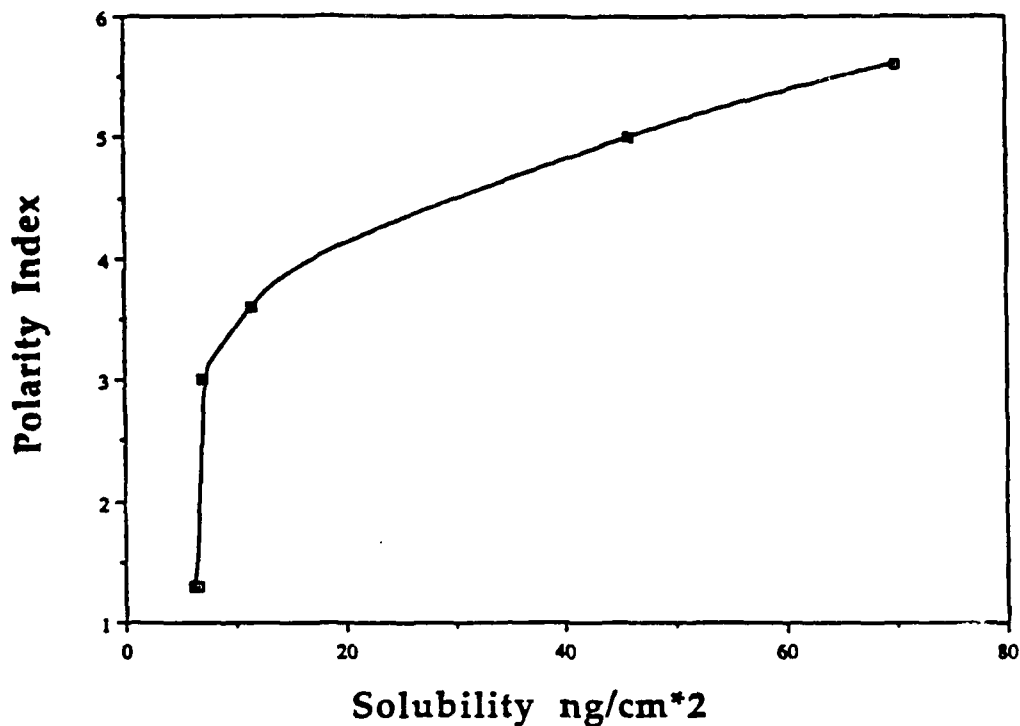


Figure 8. Polarity index versus solubility constant for six elastomers. The polarity index for FC has been estimated at 5.0 based on permeation curve.

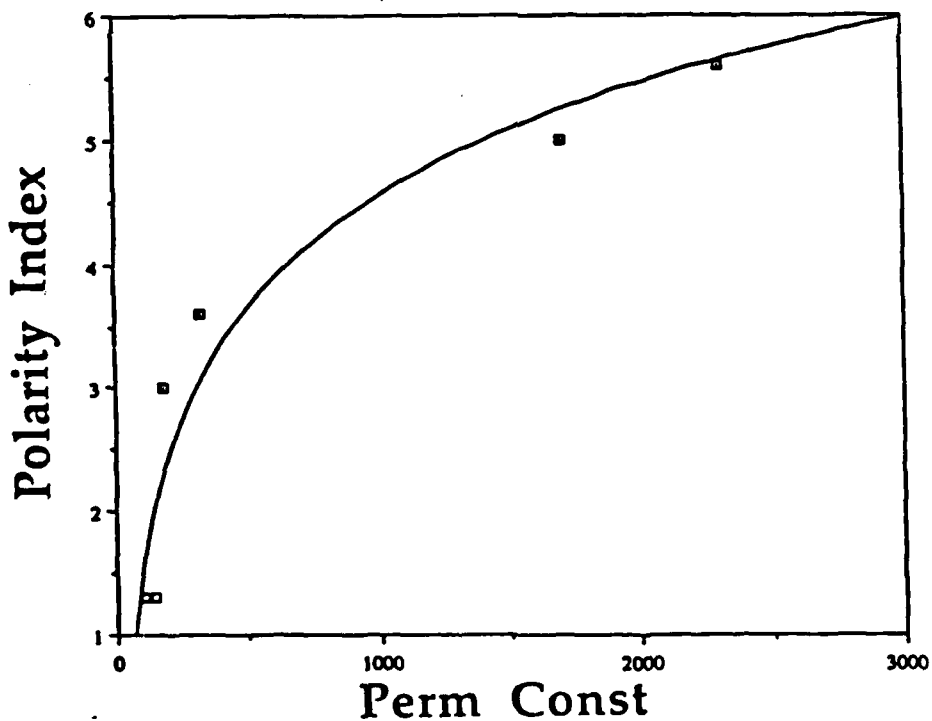


Figure 9. Polarity index versus permeation constant for six elastomers. The polarity index for FC has been estimated at 5.0 based on permeation curve.

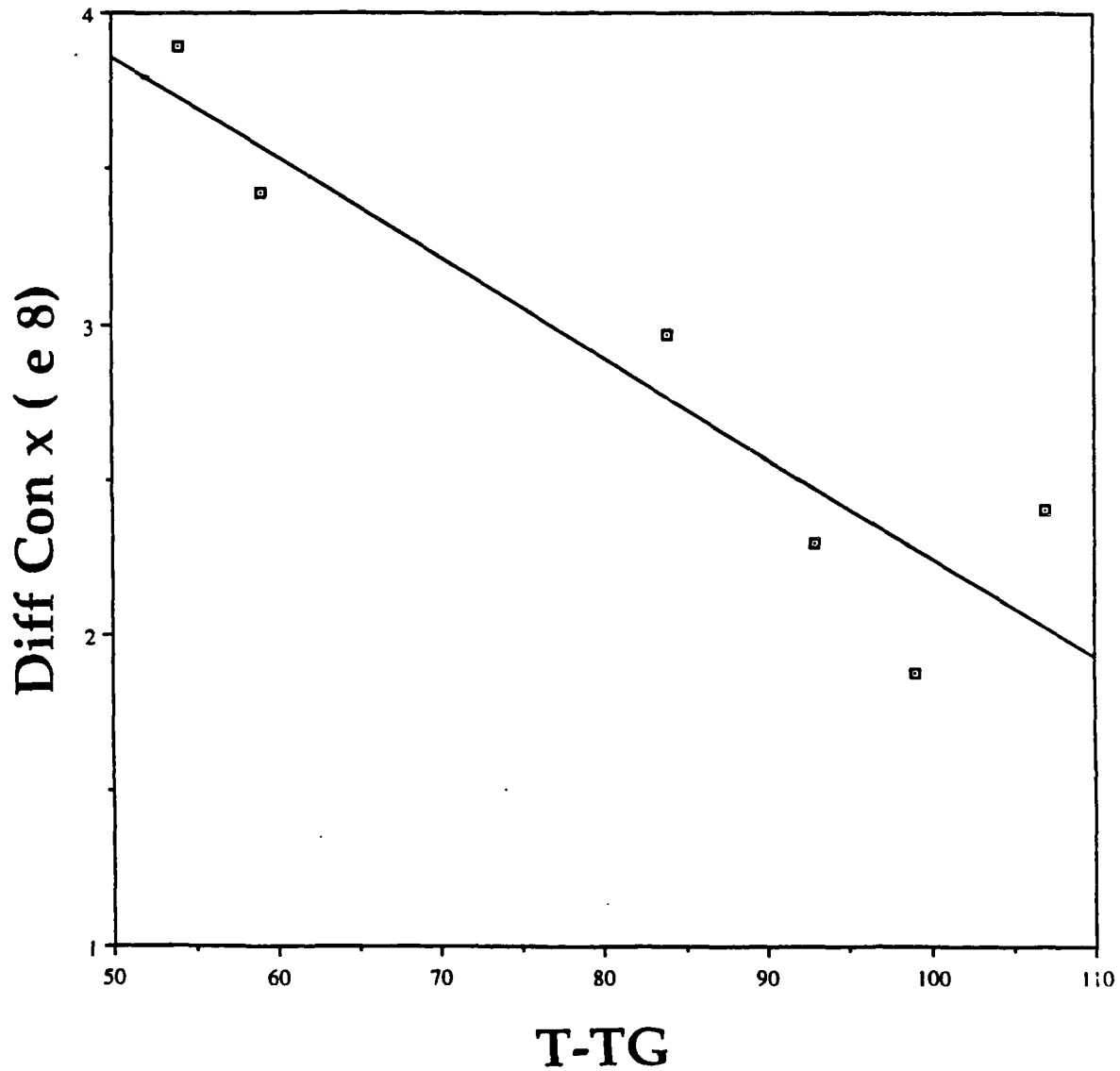


Figure 10. The dependence of the diffusion constant with polymer mobility. Polymer mobility is measured as T-Tg.

Table 1. Permeation data

<u>ELASTOMER SAMPLE</u>	<u>THICKNESS mils</u>	<u>BREAKTIME min</u>	<u>DIFFUSION cm<sup>2</sup>/sec</u>	<u>SOLUBILITY ng/cm<sup>3</sup></u>	<u>PERMEATION ng/sec*cm</u>
EC	18.1	38	3.89*10 <sup>-8</sup>	6.99*10 <sup>10</sup>	2233
FC	25.0	59	3.72*10 <sup>-8</sup>	4.58*10 <sup>10</sup>	1708
NBR	18.5	25	2.97*10 <sup>-8</sup>	1.15*10 <sup>10</sup>	341
CR	22.0	40	2.36*10 <sup>-8</sup>	7.03*10 <sup>9</sup>	179
NR	24.0	136	2.41*10 <sup>-8</sup>	6.08*10 <sup>9</sup>	146
SBR	21.0	102	1.88*10 <sup>-8</sup>	6.57*10 <sup>9</sup>	104

Table 2. Polarity index for various liquids

<u>LIQUIDS</u>	<u>POLARITY INDEX</u>
n-Hexane	0.0
Carbon Disulfide	1.0
Carbon Tet.	1.7
Toluene	3.2
n-Propyl Alcohol	4.3
Ethyl Alcohol	5.2
Acetone	5.4
Acetonitrile	6.2
DMSO	6.5
Formamide	7.3
Water	9.0

Table 3. Polarity index for elastomers

<u>POLYMER</u>	<u>POLARITY INDEX</u>
NR	1.3
SBR	1.3
CR	3.0
NBR	3.6
FC	5.0*
EC	5.6

\* Polarity index for FC is estimated from the permeation curves.



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