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# U. S. NAVAL AIR ENGINEERING CENTER

PHILADELPHIA, PENNSYLVANIA

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AERONAUTICAL MATERIALS LABORATORY

REPORT NO. NAEC AML RS 7045/17 DATE 2 March 1964

THE PERMEATION OF SALT WATER THROUGH PROTECTIVE COATINGS USING RADIOACTIVE TRACER TECHNIQUES

SEVENTH PROGRESS REPORT ON  
 F.R. PROJECT NAEC AML (17) R 360 FR 101  
 F.R. PROJECT NO. 17 (15 JUN 1963 - 15 DEC 1963)

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#### ABSTRACT

Data obtained on the permeation of sodium ions, chloride ions and water molecules through eight films using radioactive tracer techniques, are presented. Permeation data on various concentrations of sodium chloride in solution show that perm-selectivity by a membrane for a particular ion may occur. Analysis of graphical presentations of data indicates that the diffusion is Fickian and on further rationalization of these data, the theoretical concepts concerning mechanisms by which ions may diffuse through a membrane are substantiated.

## I. INTRODUCTION

A. This is the seventh progress report of work performed to date under Project No. NAEC AML (17) R 360 FR 101 Foundational Research Project No. 17, entitled, "The Permeability of Salt Water Through Protective Coatings Using Radioactive Tracer Techniques", which was established on 15 September 1960.

B. The specific aims of this program are the use of radioisotopes to establish fundamental data regarding the mechanism of salt water permeation through organic protective films; to investigate the effects of resins, pigments and incorporated corrosion inhibitors on this permeation, and finally to utilize the radioactive tracer techniques developed in this investigation to obtain fundamental data concerning the effectiveness of conventional protective coatings as barrier materials for the prevention of corrosion.

C. The reference (a), (b), and (c) reports to the Bureau of Naval Weapons contain descriptions of the equipment and procedures developed to accurately measure the quantity of chloride ions as chlorine-36 diffusing through a membrane and data on the rate of diffusion of chloride ions through various films. The reference (d) report describes the procedure developed to measure the rate of diffusion of water through a membrane and experimental data thereon. The reference (e) report describes a radioactive tracer procedure to determine the rate of diffusion of the sodium ion through a film using sodium-22. The reference (f) report contains complete permeability data for the chloride ion, sodium ion and water for five films. An analysis of these data is presented together with theoretical concepts on the mechanism of permeation of the sodium ion and chloride ion.

D. Complete permeability data for the chloride ion, sodium ion and water is presented for eight films. Permeability data at various concentrations of sodium chloride in solution is presented and analyzed to show that a perm-selectivity may occur. Analysis of graphical presentations indicate that the diffusion is Fickian and further substantiates the theoretical concepts that are offered to establish the mechanisms of permeation of ions through a film.

## II. EXPERIMENTAL PROCEDURES

### A. Permeation of Chloride Ion, Sodium Ion and Water

The procedure for the determination of the rate of diffusion of the chloride ion is fully described in the reference (b) and (c) reports. The reference (d) report describes the determination of the rate of diffusion for water and the procedure for the sodium ion is reported in reference (e). These methods briefly are: a film is fixed in the diffusion cell. The radioactive solution is placed in the right hand compartment of the cell and distilled water is placed in the left hand compartment. The ratemeter is set and the pump and recorder are turned on. When the permeability of the sodium ion or water is

being determined, the counts per minute recorded by the recorder are corrected for decay of sodium-22 and tritium by referring to decay curves. (This is not necessary when the chloride ion permeability is being determined since chlorine-36 has a very long half-life). Finally, by referring to a calibration curve, the c.p.m. are converted to the total weight of the ions or water that diffused through the membrane.

1. Employing these methods, the rates of diffusion for the chloride ions, sodium ions and water were completed for three films in addition to those already reported in reference (f). (Mylar, cellulose acetate and Aclar films). A complete Diffusion Rate Study is now presented in Table 1 for eight films which represent typical protective coatings and barrier materials encountered in the Bureau of Naval Weapons corrosion protection endeavors. All of these measurements were obtained using a 0.053M sodium chloride solution.

2. In order to investigate the effects of the concentration of salt solution on the rates of diffusion of the ions, three films were selected and measurements were obtained using a .027M solution of sodium chloride. These results are tabulated in Table 2.

B. The quantities of the ions (sodium and chloride) that permeated the films at various periods of time were calculated and then plotted against these periods of time. The graphs depicting these relationships are presented in Plates 1, 2, 3, and 4.

C. The rates of diffusion ( $\text{g hr}^{-1}$ ,  $\text{sq.cm}^{-1}$ ,  $\text{mil}^{-1}$ ) for the sodium ions and for the chloride ions were calculated at various periods of time and were then plotted against these periods of time. The graphs depicting these relationships are presented in Plates 5 and 6. In order to present the graphs in as few plates as possible, semi-logarithmic sheets were employed and the rates of diffusion plotted on the logarithmic axis.

### III. RESULTS

A. The completed test results for all eight films selected for this investigation are tabulated in Table 1. Detailed observations of the diffusion test results obtained for five films which were completed at that time were presented in the reference (f) report. Similar observations have been noted of diffusion test results for Aclar, Mylar and cellulose acetate films. These results are briefly restated.

1. The experimental values for the rates of diffusion of both the chloride ion (column 6) and the sodium ion (column 8) are not equal to the theoretical values (columns 4 and 5). These theoretical values were calculated from the concentrations of the ions in the test solution and the experimental values for the rates of diffusion of water (column 3), assuming that the ions diffuse through the films according to their concentrations in the test solution.

2. The measured diffusion rates for the sodium ion (column 8) are not equal to the theoretical diffusion rates (column 7). These theoretical diffusion rates were calculated from the diffusion rates experimentally obtained for the chloride ion (column 6), assuming that sodium ion diffuses in stoichiometric proportions with the chloride ion.

B. 1. The rates of diffusion for the chloride and sodium ion determined at a sodium chloride concentration of 0.027M (Table 2) are less than those determined at a concentration of 0.053M (Table 1). The results of Table 2 also indicate that the rates of diffusion of the sodium ion are not equal to the calculated theoretical diffusion rates based on the measured rates for the chloride ion.

2. Considering the results of Table 2, the following may be observed:

a. The rates of diffusion for the neoprene organic protective coating are approximately one-half of those tabulated in Table 1.

b. The rate of diffusion for the sodium ion permeating through the cellulose acetate film is one-half that measured at a salt concentration of 0.053M, however, the chloride ion rate of diffusion is much less than one-half the chloride rate measured at the greater concentration.

c. The rates of diffusion through the nylon film are considerably less at the 0.027M concentration although they are not one-half of these rates.

C. The quantities of permeating ions as functions of time are shown graphically in Plates 1 to 4. With the exceptions of the graphs depicting the flow of the chloride ion through polyethylene and nylon films, the data fall on straight lines with some indication of a slight curve as the graph approaches the origin.

D. The rates of diffusion, in  $\text{g hr}^{-1}$ ,  $\text{sq. cm}^{-1}$ ,  $\text{mil}^{-1}$ , as functions of time are shown graphically in Plates 5 and 6. An examination of these graphs shows that the rate of diffusion of an ion either increases or decreases with the passing of time until a condition is reached wherein the ions continue to diffuse through the film at a constant rate.

#### IV. ANALYSIS OF RESULTS

A. A detailed analysis of the experimental results, tabulated in Table 1, was presented in the reference (f) report to the Bureau of Naval Weapons. At that time, the experimental work had been completed for five films. Based upon these results, a theoretical discussion of the mechanisms by which ions diffuse through protective films was presented. As mentioned previously, the diffusion data for Mylar, Aclar and cellulose acetate have been completed and included in Table 1. These additional data do not alter the concepts of these mechanisms but further substantiate them. These concepts are briefly summarized in this report as follows:

1. From the analysis of the data of Table 1, reported in paragraph III, A1 and A2, it is evident that the solution of sodium chloride is not flowing through the various films as such, and that a portion of the sodium chloride is withheld. Also, the sodium ions and the chloride ions are in no case diffusing through any of the films in stoichiometric proportions. Characteristic of the film used, either the cation or the anion diffuses to a greater extent than its counter ion. It is known that ions exist in solution in a hydrated state and it is generally accepted that the hydrated sodium ion is larger than the hydrated chloride ion (cf. refs. (g), (h) and (i)). Therefore, since these hydrated ions are not of the same dimensions, the above results indicate that the retention of an ion by a membrane is not solely due to pore diameter, since if the mechanism of permeation were solely of a sieve-like nature, then in no case would a hydrated ion of larger dimensions diffuse at a greater rate than its counter ion with smaller dimensions. An examination of the results in Table 1 shows that the measured diffusion rates of the sodium ion are greater than those calculated from the measured diffusion rates of the chloride ion in the case of three films: neoprene, epoxy, and cellulose acetate. Thus, the sodium ion is diffusing through these membranes at greater concentrations than would occur if the sodium and chloride ions diffused in their stoichiometric ratios in sodium chloride. Consequently, it can be deduced that another factor besides the pore diameter and size of the hydrated ion is involved. This factor is the concept of ionic charges appearing upon the walls of the pores of a membrane as explained below.

2. From electrochemical investigations measuring the electromotive forces (concentration potentials) arising in such membrane concentration cells, K. Sollner, reference (j), offers theoretical electrochemical concepts to explain the mechanisms of the diffusion of ions. It is believed that the walls of pores contain charges caused by the ionization of certain groups which are part of the polymer molecule. If these charges are negative, then anions will be repelled and cations will be attracted, the reverse taking place if the charges are positive. Thus, a membrane may be said to be perm-selective in that the ions of the same charge as the membrane are presented by electrical repulsion from approaching the spots at the pore walls where the fixed charges of the same polarity are located. It can be visualized if the pores are sufficiently narrow, such ions may be entirely excluded, since this charge effect will extend into the solution. On the other hand, a pore may have a sufficiently wide diameter so that the charge on the wall of the pore may not have a sufficient repulsive effect for ions that are of the same charge to stop them, and hence, they pass through in "mid-stream". Consequently, a mixture of ions may diffuse through the membrane and the ions of opposite charge to that on the walls of the pore will permeate at a greater rate.

B. The theoretical concepts that have previously been presented from electrochemical studies include another factor that may affect the permeation of ions through a membrane. This factor is the concentration of an electrolyte in solution. It is thought that with an increase in concentration of the electrolyte solutions, an increasing quantity of anions and cations will enter the pores and the specific influence of the membrane may be decreased thereby. This concept is offered to explain the apparent differences noted in concentration potential measurements where the concentration of the electrolyte in solution is increased.

The effect of concentration of the electrolyte is being investigated at the present time, using the radiochemical procedures described in the previously referenced reports. It is thought that, should the phenomenon of perm-selectivity be observed as the concentration of electrolyte in solution is decreased, this would be strong proof that a factor in the mechanisms of the diffusion of ions through a membrane is an electrochemical phenomenon involving a charge on the walls of a pore. Since if the diffusion mechanism were merely that of pore diameter, size of hydrated ions and subsequent flow through pores, then the effect of varying the concentration of the electrolyte would reflect as proportional changes in the rate of diffusion of the ions.

1. The data presented in Table 3 contain calculations based on measurements of rates of diffusion determined at various concentrations of sodium chloride in solution. The rates of diffusion measurements at 0.014M sodium chloride solution for the neoprene film have not been completed to date. The ionic charges reported to be present on the walls of the pores were deduced from the sodium ion and chloride ion rates of diffusion measurements as reported in the Aeronautical Materials Laboratory report, reference (f). It was found that there was no stoichiometric relationship between the measured rates of diffusion of the sodium ion and the chloride ion. Therefore, it was concluded that an ionic charge exists on the walls of the pores and that the polarity of the charge was determined by whether the measured rate of diffusion of the sodium ion was greater or less than the theoretical diffusion rate calculated from the measured diffusion rates of the chloride ion.

An analysis of the data of Table 3 shows that for one ion there is a perm-selectivity definitely taking place as the concentration of the sodium chloride in solution is decreased. This is arrived at by comparing the measured rates of diffusion of the sodium ion, at the various salt concentrations, as a percentage of the stoichiometric equivalent of the sodium ion, calculated from the measured rate of diffusion of the chloride at the same salt concentration, as:

$$\frac{\text{measured rate of diffusion of sodium ion} \times 100}{\text{stoichiometric equivalent of sodium ion calculated from measured chloride ion rate at same salt concentration}} = \%$$

a. The percentage as calculated above for the nylon film decreases as the concentration of the sodium chloride in solution is decreased from 0.053M to 0.014M. This indicates that there is a decrease in the proportion of the sodium ion to the chloride ion diffusing through the film and that the nylon film is exhibiting a perm-selectivity for the chloride ion.

b. The percentages calculated for the cellulose acetate film (Table 3) increase as the concentration of the sodium chloride in solution decreases which indicates that a greater proportion of the sodium ion to the chloride ion is diffusing through the membrane in proportion to the chloride ion and that the cellulose acetate film is exhibiting perm-selectivity for the sodium ion.

c. There is no perm-selectivity apparent as yet by the neoprene film at a concentration of 0.027M. The measurements at 0.014M have not been completed.

In this manner, strong experimental evidence has been obtained that an ionic charge on the walls of the pores in a film is a factor involved in the mechanism of the diffusion of a salt through a membrane.

C. The graphs depicted in Plates 1 to 4, in which the quantities of ions permeating are plotted as function of time, are characteristic of a Fickian diffusion process. The Fick equation (reference (k) and reference (l)).

$$D = - \frac{q}{Kt} \left( \frac{1}{C' - C''} \right)$$

in the differential form is not strictly applicable to these diffusions, since changes in the concentration gradient occur in the course of the diffusion. However, in this experimental work, the changes in the concentrations of the solutions are very minute so that the concentration gradient at the film may be considered constant, and the above equation is a close approximation. In this equation: D = diffusion coefficient, q = the amount of material passing through the film in time t, C' and C'' are the concentrations of the solutions at the film and K is equal to A/l in which A is the cross sectional area of the film and l is the film thickness.

1. The above equation can be converted to the simpler equation shown below, by eliminating  $\left( \frac{1}{C' - C''} \right)$  since the concentration changes are minute and may be considered constant and also the negative sign may be dropped since it is used only to signify a flow from the chamber of greater concentration to that of more dilute concentration. The equation then becomes:

$$q = \frac{DA t}{l}$$

where q = the quantity transferred in time t, D = the permeability constant (diffusion coefficient) (g hr<sup>-1</sup>, sq. cm<sup>-1</sup>, mil<sup>-1</sup>), A is the cross sectional area of the film and l = the film thickness. The permeability constant, D, may be calculated from the above equation by substituting the experimental values for q and t, when a steady state of flow has been reached. Such values have been recorded in Tables 1 and 2 as rates of diffusion. A more exact figure may be obtained by calculating the slopes of the graphs shown in Plates 1 to 4. Since A is a constant and l can be measured, D may be calculated from the slope of the curve at the steady state of flow. In this manner, the permeability constants for the sodium ion were calculated for three films; the slopes were calculated by the least mean squares method from the laboratory data of a series of measurements and the permeability constants or rates of diffusion hr<sup>-1</sup>, sq. cm<sup>-1</sup>, mil<sup>-1</sup> were obtained. The results were:

Plaskon 8200,  $6.37 \times 10^{-8}$  g Na<sup>+</sup>; neoprene,  $7.42 \times 10^{-9}$  g Na<sup>+</sup>; and epoxy organic protective coating  $1.11 \times 10^{-9}$  g Na<sup>+</sup>. These values may be compared to those appearing in Table 1.

D. The graphs shown in Plates 5 and 6 depict the rates of diffusion for the ions plotted as a function of time. An analysis of these curves indicates a general trend to be taking place. That is, that for the films which were reported to have a positive charge on the walls of the pores (nylon and nitro-cellulose acrylic lacquer), the rate of diffusion for the chloride ion increases with an increase in time until the steady state of flow is reached and vice versa for the sodium ion. On the other hand, for those films which were reported to carry a negative charge on the walls of the pore (neoprene and epoxy organic protective coatings), the rate of diffusion for the sodium ion increases with time (slightly with the epoxy film) until a steady state of flow is reached and vice versa for the rate of diffusion of the chloride ion. Therefore, it is apparent that a phenomenon is occurring which is shown as the ascending or descending segments of the graphs. This phenomenon could be the hydrolysis taking place at the walls of the pores resulting in the formation of the ionic charge so that the ion of the same charge in solution are being deflected as this charge on the walls multiplies. Likewise, the ions of opposite charge in the solution are being attracted and the diffusion becomes more rapid as the charge on the walls builds up.

#### V. CONCLUSIONS

It may be concluded from the radiochemical data presented herein that ionic charges exist on the walls of the pores of a film and that these charges control the flow through the film of ions that are in solution.

#### REFERENCES

- (a) First Quarterly Progress Report on Foundational Research Project NAMC AML RS 7045/17 of 9 Sep 1960
- (b) Second Progress Report on Foundational Research Project NAMC AML RS 7045/17 of 15 Jun 1961
- (c) Third Progress Report on Foundational Research Project NAMC AML RS 7045/17 of 15 Dec 1961
- (d) Fourth Progress Report on Foundational Research Project NAMC AML RS 7045/17 of 15 Jun 1962
- (e) Fifth Progress Report on Foundational Research Project NAMC AML RS 7045/17 of 15 Dec 1962
- (f) Sixth Progress Report on Foundational Research Project NAMC AML RS 7045/17 of 15 Jun 1963
- (g) Electrochemistry, Creighton and Koshler, Vol. I 1941 p. 139
- (h) The Structure of Water and Electrolyte Solutions, R. J. Podolsky, Naval Medical Research Institute, Bethesda, Maryland
- (i) Textbook of Electrochemistry, Kortum and Bockris 1951, Vol. I, p. 131-137
- (j) The Electrochemistry of Porous Membranes, Karl Sollner, Chapter 4 of Electrochemistry in Biology and Medicine, edited by T. Shedlovsky 1955
- (k) Technique of Organic Chemistry, A. Weissberger, Vol. I Physical Methods of Organic Chemistry, Part Two p. 975
- (l) Mathematics of Diffusion, J. Crank, Oxford at the Clarendon Press, 1956 p. 47

DIFFUSION RATE STUDY, SODIUM CHLORIDE SOLUTION 0.053M

1	2	3	4	5	6	7	8
Test	Designation	Diffusion of Water (1)	Theoretical Diffusion of Chloride Ion (1)(2)	Theoretical Diffusion of Sodium Ion (1)(2)	Measured Diffusion of Chloride Ion (1)	Theoretical Diffusion of Sodium Ion Based on Measured Chloride Ion (1)	Measured Diffusion of Sodium Ion (1)
Nylon	8200	$9.24 \times 10^{-3}$	$2.17 \times 10^{-5}$	$1.40 \times 10^{-5}$	$1.31 \times 10^{-7}$	$8.47 \times 10^{-8}$	$5.78 \times 10^{-8}$
Aclar	191	$7.87 \times 10^{-6}$	$1.85 \times 10^{-8}$	$1.20 \times 10^{-8}$	None	None	None
Polyethylene	----	$1.57 \times 10^{-5}$	$3.69 \times 10^{-8}$	$2.39 \times 10^{-8}$	$8.48 \times 10^{-9}$	$5.49 \times 10^{-9}$	None
Nitrocellulose Acrylic Lacquer	MIL-L-19537	$3.50 \times 10^{-4}$	$8.23 \times 10^{-7}$	$5.32 \times 10^{-7}$	$7.29 \times 10^{-9}$	$4.72 \times 10^{-9}$	None
Neoprene Organic Protective Coating	1672A	$4.90 \times 10^{-4}$	$1.15 \times 10^{-6}$	$7.45 \times 10^{-7}$	$2.57 \times 10^{-7}$	$1.66 \times 10^{-9}$	$7.46 \times 10^{-9}$
Epoxy	MIL-C-22750	$1.23 \times 10^{-4}$	$2.89 \times 10^{-7}$	$1.87 \times 10^{-7}$	$1.51 \times 10^{-9}$	$9.77 \times 10^{-10}$	$1.39 \times 10^{-9}$
Cellulose Acetate	----	$1.95 \times 10^{-2}$	$4.58 \times 10^{-5}$	$2.96 \times 10^{-5}$	$4.66 \times 10^{-7}$	$3.02 \times 10^{-7}$	$1.20 \times 10^{-6}$
Mylar	----	$7.78 \times 10^{-5}$	$1.83 \times 10^{-7}$	$1.18 \times 10^{-7}$	None	None	None

NOTES:  
 (1) Diffusion Rates are grams  $hr^{-1}$  sq.  $cm^{-1}$   $mil^{-1}$

(2) Diffusion Rates calculated from Diffusion Rate of Water column (3)

TABLE 1

DIFFUSION RATE STUDY, SODIUM CHLORIDE SOLUTION 0.027M

<u>Film</u>	<u>Designation</u>	<u>Measured Diffusion Rate of Chloride Ion(1)</u>	<u>Theoretical Diffusion of Sodium Ion Based on Measured Chloride Ion(1)</u>	<u>Measured Diffusion Rate of Sodium Ion(1)</u>
Nylon (Capran)	8200	$9.30 \times 10^{-8}$	$6.03 \times 10^{-8}$	$4.80 \times 10^{-8}$
Neoprene Organic Protective Coating	1672A	$1.20 \times 10^{-9}$	$7.77 \times 10^{-10}$	$3.40 \times 10^{-9}$
Cellulose Acetate	-----	$1.40 \times 10^{-7}$	$9.10 \times 10^{-8}$	$6.30 \times 10^{-7}$

NOTES:

(1) Diffusion Rates are grams  $hr^{-1}$  sq.  $cm^{-1}$   $mil^{-1}$

TABLE 2

EFFECT OF CONCENTRATION OF SODIUM CHLORIDE ON PERM-SELECTIVITY

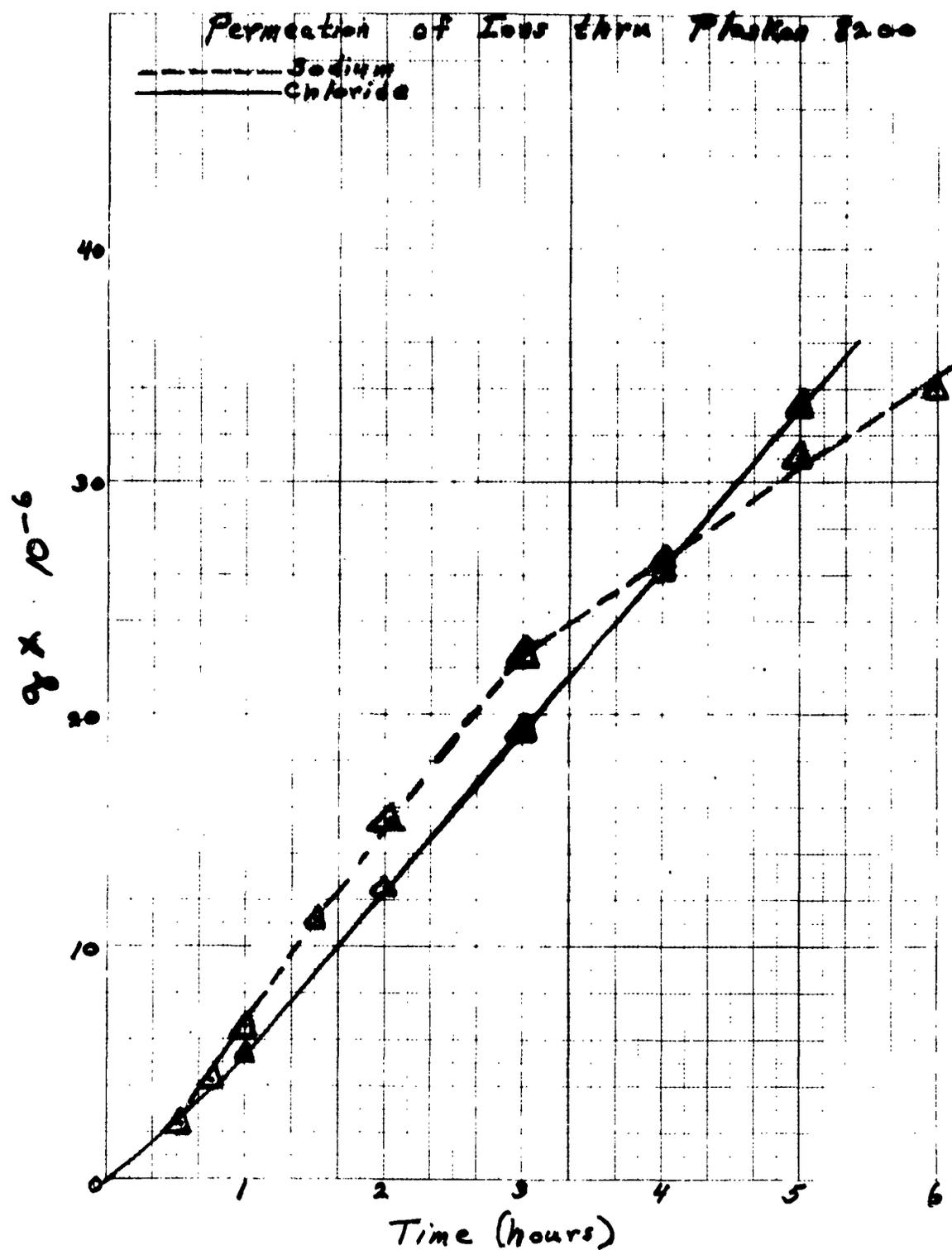
Film	Designation	Charge on Pores(1)	Rate of Diffusion		% of Theoretical Rate of Diffusion		Rate of Diffusion		% of Theoretical Rate of Diffusion	
			0.053M	8.47 x 10 <sup>-8</sup>	0.027M	6.50 x 10 <sup>-8</sup>	0.014M	1.10 x 10 <sup>-7</sup>	0.014M	1.10 x 10 <sup>-7</sup>
Nylon	8200	+	5.78 x 10 <sup>-8</sup>	68.2	3.58 x 10 <sup>-8</sup>	55.1	3.3 x 10 <sup>-8</sup>	30.0	3.3 x 10 <sup>-8</sup>	30.0
Neoprene Organic Protective Coating	1672A	—	7.46 x 10 <sup>-9</sup>	449	3.40 x 10 <sup>-9</sup>	439	-----	-----	-----	-----
Cellulose Acetate	-----	—	1.20 x 10 <sup>-6</sup>	397	6.30 x 10 <sup>-7</sup>	692	3.57 x 10 <sup>-7</sup>	1248	2.86 x 10 <sup>-8</sup>	1248

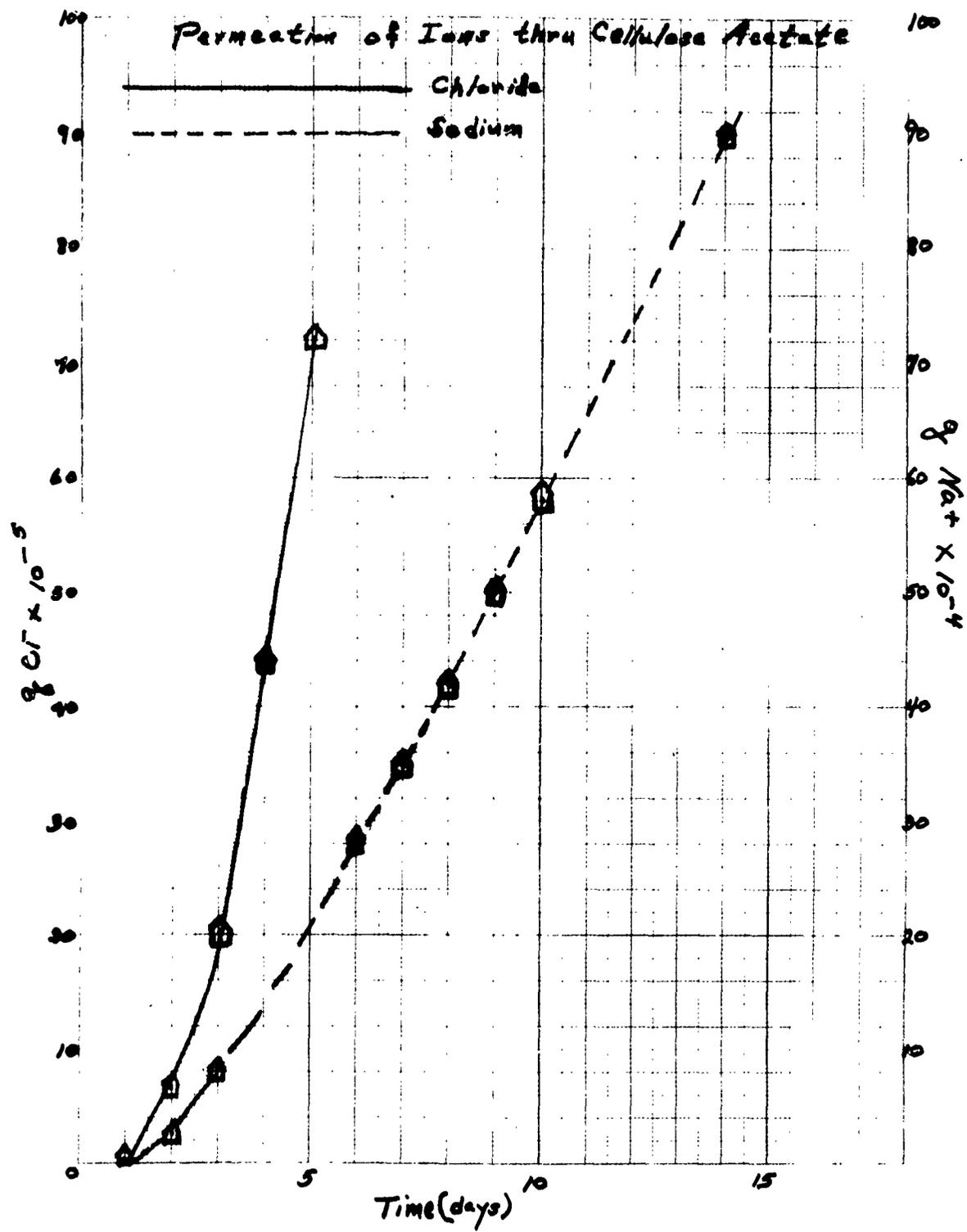
NOTES:

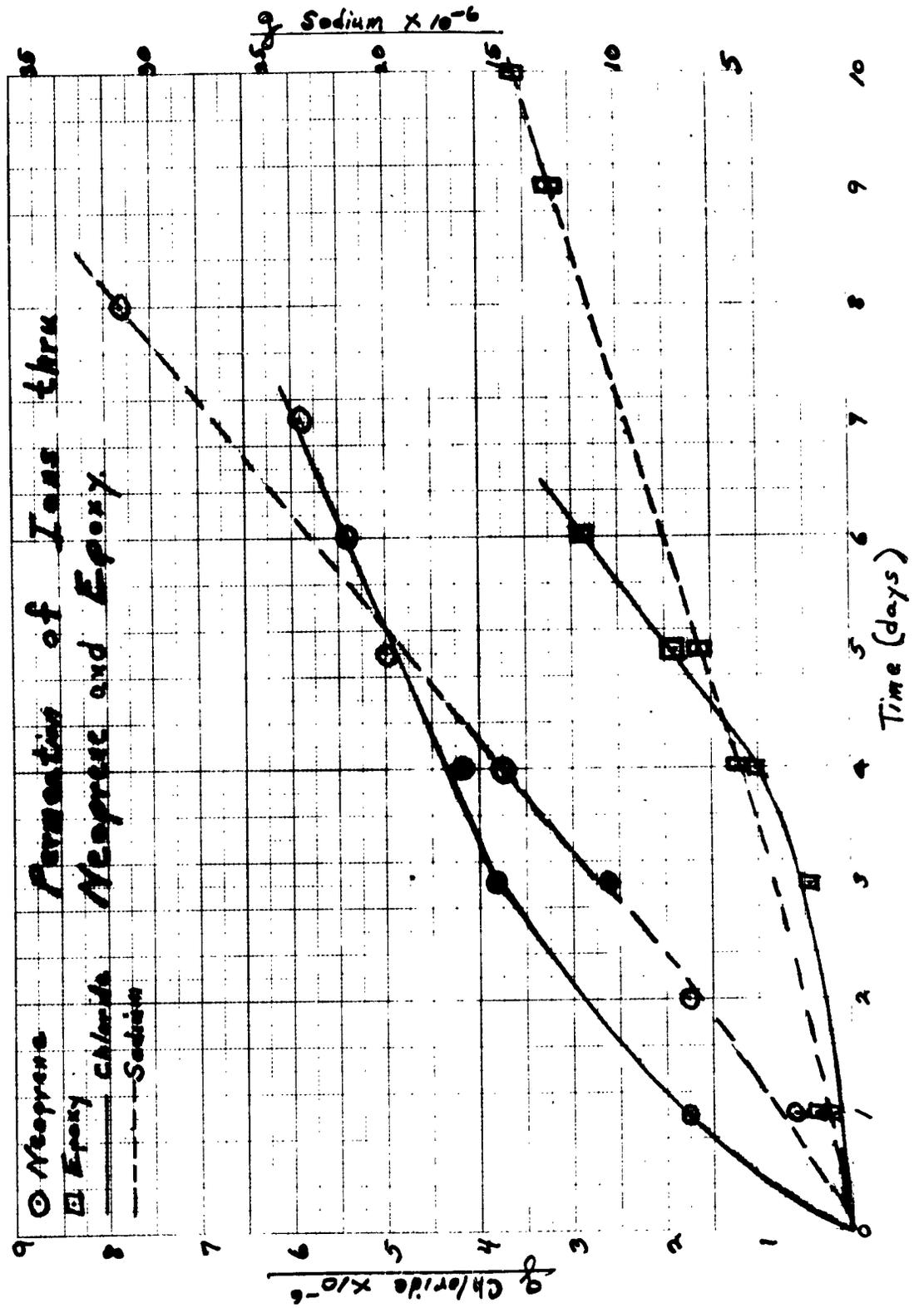
(1) Charge on walls of pores as deduced from experimental work reported in reference (f)

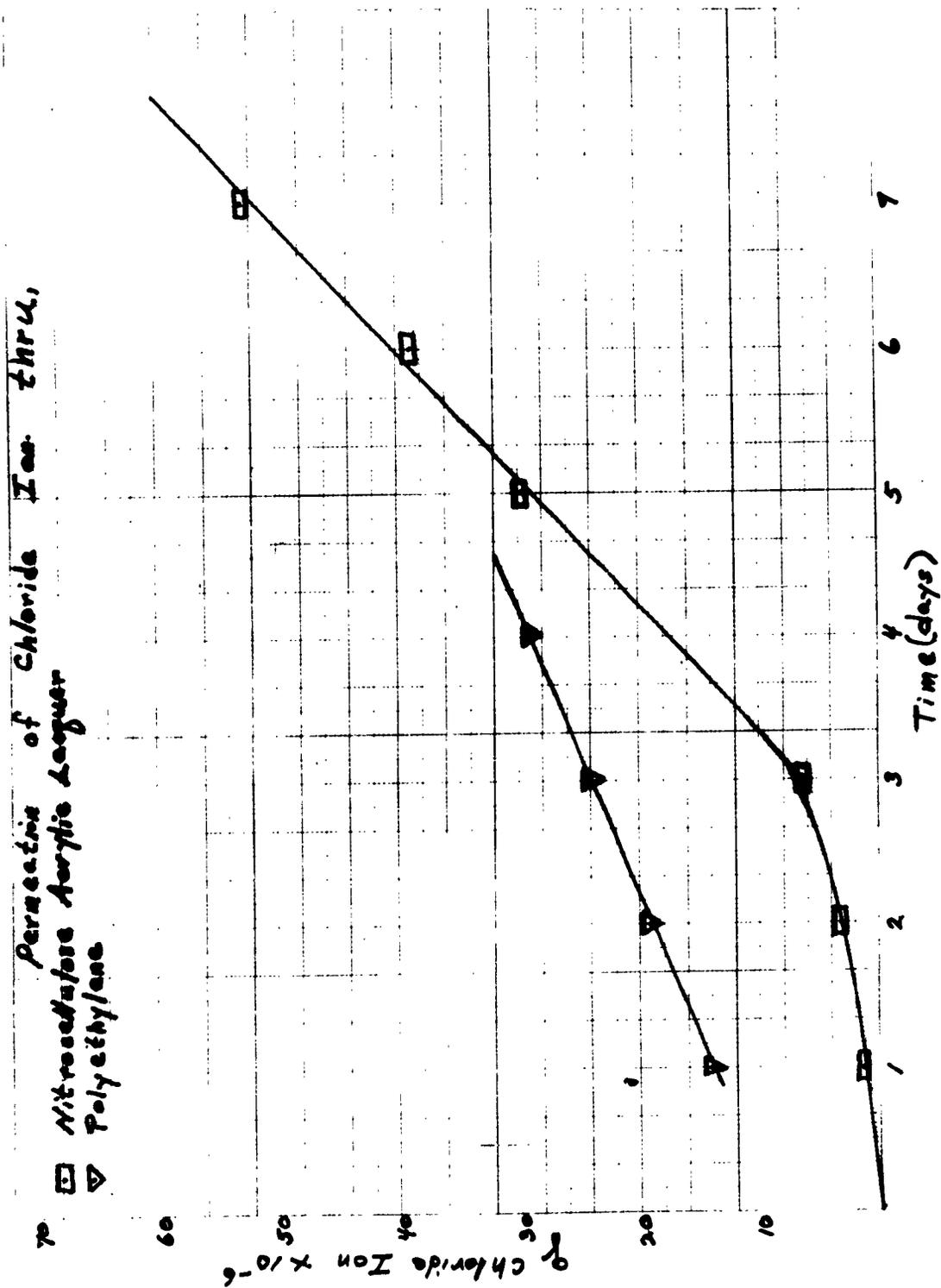
(2) Theoretical diffusion rate for Na<sup>+</sup> ion calculated from measured Cl rate of diffusion

TABLE 3



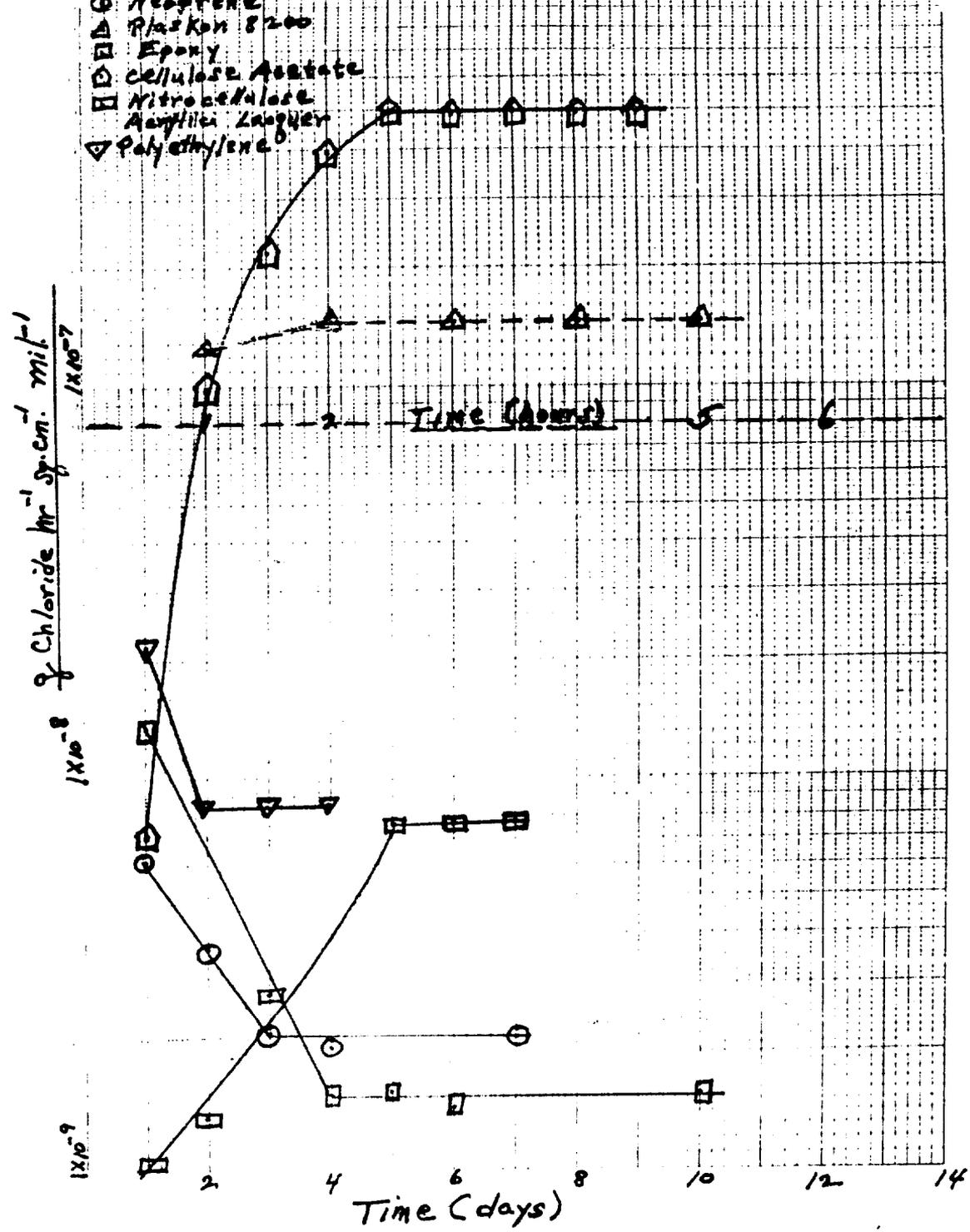






### Rate of Permeation of Chloride Ion Vs. Time

- ⊙ Neoprene
- △ Plaskon 8200
- Epoxy
- ⊠ Cellulose Acetate
- ▣ Nitrocellulose
- ⊞ Methyl Methacrylate
- ▽ Polyethylene



Rate of Permeation of Sodium Ions vs. Time

- Neoprene
- △ Plaskon 8200
- Epoxy Organic Protective Coating
- ⊠ Cellulose Acetate

