DIFFUSION OF SODIUM IN VARIOUS TYPES OF CONCRETE

by

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INTRODUCTION

It has been suggested, and in many cases proven, that alkalies react chemically with some types of aggregate used in concrete, creating non-uniform pressures within the concrete and leading to eventual structural failure (6), (10). These alkalies are definitely present in the cement itself. But this is not the only source, as the environment in which the concrete is placed may also supply these alkalies. In view of this deleterious alkali-aggregate reaction, the Applied Mechanics Department at Kansas State College became interested in studying the manner in which alkalies, particularly sodium ions, diffuse through various types of concrete.

In order to study the movement of sodium, some means of detecting and measuring the quantity of it at any point in the concrete was required.

The use of chemical analysis, although theoretically possible, was not practical for two reasons. First, the amounts of sodium involved were quite small, making a careful analysis slow and painstaking. Second, it would have been impossible to distinguish between sodium introduced from an outside source and sodium already present. The flame photometer method also has been suggested and used (Blackman, 2), but it suffered from the same weaknesses as the chemical analysis.

A method was proposed and used by Spinks, et al (9) which eliminated the above mentioned difficulties. The ion under investigation was tagged by the use of radioactive tracers. It then became relatively easy to detect and measure the concentration of the ions at any point in the neat cement with which they were working. The Spinks method was chosen as the basis for the present study of the diffusion of sodium in various types of concrete.

METHOD AND PROCEDURE

Theoretical Considerations

The general equation for diffusion is as follows:

where C is the concentration of the diffusing substance and D is the diffusion constant in cm²/sec. If D is assumed to be a true constant independent of the concentration, then the solution of the equation for linear diffusion into a semi-infinite solid is given by (Barrer, 1),

$$1 - \frac{c}{c_0} = \frac{2}{\sqrt{\pi}} \int_0^{\infty} e^{-y^2} dy$$

where Commontration at surface of solid

$$\alpha = \frac{x}{2\sqrt{Dt}}$$

x = depth at which C is measured

t = diffusion time

The right aids of the equation is the probability integral. Thus, in order to obtain the value of D, which is indicative of the rate of diffusion, one need only know the ratio $\frac{C}{C_0}$. Since this is a ratio, it was possible to use the radioactive counting rates rather than the actual concentrations. With the value of $\frac{C}{C_0}$ known, a table of probability integrals gives the value of </c><math>< From this, D is obtained from the equation

$$D = \frac{1}{4t} \quad \left(\frac{x}{\alpha}\right)^2$$

Experimental Procedure

Treatment of the Concrete. Sixty concrete cylinders, two inches in diameter and four inches long, were provided by the applied mechanics department; these are shown in Flate I. Fourteen varieties of concrete were used in order to determine whether or not there was a variation in D with each type. The constituents of the various concretes are given in Table 1. All of the cylinders were subjected to a 30 day curing period in a steam room and subsequently stored in laboratory air for an additional 400 days.

Approximately .5 inch of concrete was removed from each end of the cylinders with a brick saw. This removed surface irregularities resulting from the initial molding of the cylinder. By means of a surface grinder, each end was ground level, and as smooth as the heterogeneity of the concrete would permit. In order that the sodium ion movement would be through the end faces only, the sides of the cylinders were coated with paraffin.

Before the cylinders were placed in an alkali solution, they were immersed in distilled water until saturated. This prevented the sodium ions from being carried into the concrete by hydrostatic pressure rather than by pure diffusion. The greatest influx of water occurred during the first 24 hours; however, the cylinders were kept in water for 34 days to insure complete saturation.

The solution in which the concrete cylinders were then immersed consisted primarily of .15 M Na₂SO₄ plus a small amount of Na²²Cl. This solution was contained in glass battery jars, each capable of holding twelve concrete cylinders in a horizontal position as shown in Plate II. Each jar was filled

EXPLANATION OF PLATE I

Two of the concrete cylinders before being coated with paraffin.

PLATE I



EXPLANATION OF PLATE II

Concrete cylinders in battery jar containing radioactive solution.



Table 1. Constituents of the concrete cylinders.

Identi- fication	: Cement	; (er. of Cemen	: Gr. of t : River S		y : Additions
PDAC	Penn Dix	ie	300	1200	1:4	None
PDAL	90 10		300	1200	1:4	N-Tair-R*
PDA2	16 16		300	1200	1:4	N-Tair-R
PDCC	10 10		214	1286	1:6	None
PDC1	40 10		214	1286	1:6	N-Tair-R
PDC 2	99 99		21.4	1286	1:6	N-Tair-R
PDBC	16 11		250	1251	1:5	None
PDB1	18 18		250	1251	1:5	N-Tair-R
PDB2	N N		250	1251	1:5	N-Tair-R
PDB1F	и и		175	1251	1:5	30% sub fly- ashtN-Tair-I
MAC	Medusa		300	1200	1:4	None
MAI.	10		300	1200	1:4	N-Tair-R
MA2			300	1200	1:4	N-Tair-R
MCC			214	1286	1:6	None
MC1			214	1286	1:6	N-Tair-R
MC2	10		214	1286	1:6	N-Tair-R
MBC	86		250	1251	1:5	None
MB1	11		250	1251	1:5	N-Tair-R
MB2	W		250	1251	1:5	N-Tair-R
MB1F	Ħ		175	1251	1:5	30% sub fly- ashtN-Tair-H
		Gradi	ng of Blue	River S. G.		
Sieve	3/8	4	8	16	30 5	0 100
% Retain	ned 0	18	45	60	80 9	3 95
6	5.5 gal/sk.	all mi	xes			

^{*}air-entrainment.

with seven liters of .15 M Na₂SO₄ plus three milliliters of Na²²Cl having an activity of .0456 mc/ml. A beeswax-coated wooden lid was placed over each jar to reduce evaporation of the solution to a minimum. Thus, only small additions of water were necessary from time to time to keep the concentrations of the solution constant.

Since diffusion processes are somewhat dependent upon temperature, the jars were placed in ovens (Plate III) and the temperature held constant at

EXPLANATION OF PLATE III

Battery jars placed in temperature controlled ovens.

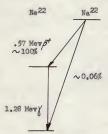
PLATE III



99° F. This value was used since the temperature of the room in which the ovens were situated was expected to be less than this at all times. An appreciably higher temperature could not have been used because the paraffin would have melted from the cylinders.

The sixty cylinders were placed in the radioactive solution on August 13, 1954. Twenty cylinders, selected for this part of the experiment, were removed on May 13, 1955, giving them a diffusion period of 273 days or about 2.38×10^7 seconds.

Counting Arrangement. Na²² is a positron emitter which has a half life of 2.6 years and whose decay scheme is as follows (Hollander, et al, 5):



Since both frand y radiations were present, the following facts had to be considered: The maximum range of the practicles was about 220 mg/cm² of aluminum. This corresponded to .85 mm of concrete or aluminum, both of which have approximately the same density. The half-thickness of concrete for 1.28 Mev y rays is about 5.5 cm. The Geiger Muller tube used for detection had an efficiency of about one percent for y rays. However, the ratio of y count to procure was much higher, due to the greater range of

the χ' rays and the added annihilation χ' count resulting from the decay of the β^+ particles.

Spinks showed in his work that no great error was introduced when the β^+ activity counted at a given depth in the cylinder was used as an indication of the concentration at that depth. This was true because the β^+ particles had a small range in comparison to the expected depths of penetration of the sodium.

Since only the fractivity was to be counted, it was necessary to distinguish between it and the factivity. This was accomplished in the following manner: The total activity due to f, f and background radiation was counted; an aluminum absorber was placed between the cylinder and the Geiger-Mueller tube filtering out the frays so that only the frays and background would be counted; the difference between the two counts was taken as the true frount.

An end window Geiger-Fineller tube having a 2.3 mg/cm² window was used in conjunction with a Berkeley model 2000 decade scaler for detecting and recording the activity. Flates IV, V and VI show the equipment used and the way in which it was arranged. To hold background to a minimum, the tube was encased in a thick lead shield leaving only the window exposed. A lead absorber, having an aperture one inch in diameter, was placed in front of the window so that only the central section of the end of the cylinder would be counted. This eliminated any edge effects which may have been present.

After each thickness of concrete was ground off, the end surface was cleaned with a brush. A small square of Kleenex was placed over the end of the cylinder to prevent contamination of the tube, which was then placed on top of the tissue. Placing the tube directly on top of the cylinder made

EXPLANATION OF PLATE IV

Photograph showing plastic bag in position.

PLATE IV



EXPLANATION OF PLATE V

Photograph showing plastic bag removed and G.M. tube in position for taking a count.

PLATE V



EXPLANATION OF PLATE VI

Close up showing concrete cylinder in the vise, brass ring for depth measurements and plywood plate for centering of G.M. tube.

PLATE VI



it possible to keep constant the distance between the cylinder and the tube window and to maximize the reometrical efficiency.

Grinding Arrangement. In order to obtain an indication of the concentration of Na²² at various depths in the concrete, it was necessary to remove small increments of concrete from the end of the cylinder and take an activity count after each increment was removed. A surface grinder was employed for the purpose of removing these small increments of concrete. From .1 mm to .4 mm of concrete was removed between counts.

The concrete cylinder under test was held securely to the moving table of the surface grinder by a vise. To prevent the spread of radioactive concrete dust thrown off by the grinding wheel, a plastic bag was placed around the grinding wheel and concrete cylinder. After each grinding operation, the bag was removed so that the cylinder would be exposed for counting purposes. The surface grinder and counting arrangement were set up in such a way that the activity count could be taken without removing the cylinder from the vise. This reduced the number of variables brought into the problem and made possible faster operation.

The thickness of concrete removed at each grinding operation was determined by measuring with a depth gage the distance between the end of the cylinder and a brass ring fastened to the vise, and substracting this measurement from the value obtained in the same manner after the previous grinding operation. Six readings were taken at each depth in order to obtain an average depth measurement.

The plywood plate to which the brass ring was attached served as a centering guide for placing the Ceiger-Mueller tube in the same position each time. The notches in the circumference of the plate were identical to notches placed in the lead shield covering the end of the tube.

It was found that the activity of the dust was actually quite low, as a monitoring instrument failed to show any reading above background. However, several precautions were taken to reduce radiation hazards which might have developed. All the work was done in a small room having a wood frame and lined with cellulose acetate-coated, cotton-cord mesh. This prevented the further spread of dust which may have escaped from the plastic bag surrounding the concrete cylinder and grinding wheel. A vacuum sweeper was used to clean the room at regular intervals. A lab coat, dust mask, and rubber gloves were worn during grinding operations.

EXPERIMENTAL RESULTS

In order to indicate the distribution of the Na²² over the surface of the cylinders, radiographs were made on three cylinders before they were ground. These radiographs are shown in Flate VII.

Counting data obtained from the cylinders are given in Flates VIII-XXI.

The B+Y, , and B+activities are indicated for each cylinder. A sample of the resulting of versus depth curve for one of the cylinders is given in Flate XXII. The important aspects of the graphs were tabulated as shown in Tables 2 and 3. A sample calculation of D is given in the Appendix,

EXPLANATION OF PLATE VII

- Fig. 1. Radiograph of end face of cylinder PDA1.
- Fig. 2. Radiograph of end face of cylinder MBC.
- Fig. 3. Radiograph of end face of cylinder PDB1F.

PLATE VII

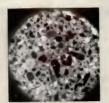


Fig. 1



Fig. 2

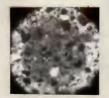
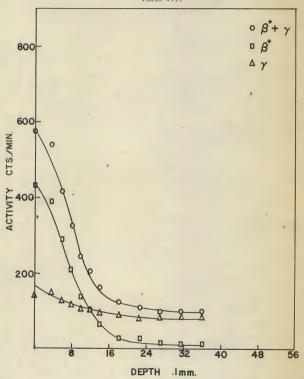


Fig. 3

EXPLANATION OF PLATE VIII.

Graph of activity versus depth in concrete for cylinder PDAC.

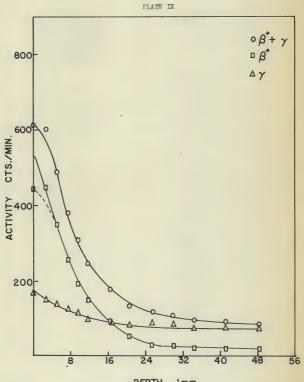




EXPLANATION OF PLATE IX

Graph of activity versus depth in concrete for cylinder PDA1.



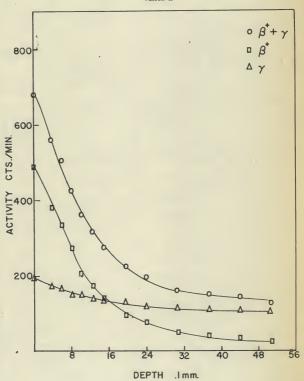


.l mm. DEPTH

EXPLANATION OF PLATE X

Graph of activity versus depth in concrete for cylinder PDCC.

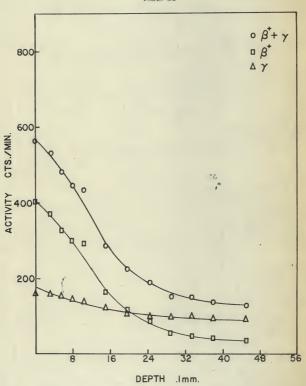




EXPLANATION OF PLATE XI

Oraph of activity versus depth in concrete for cylinder PDC1.

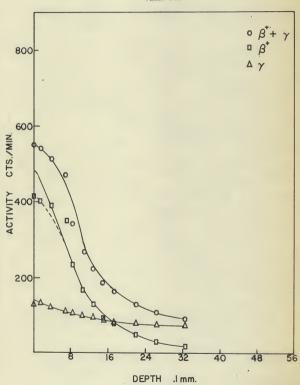
PLATE XI



EXPLANATION OF PLATE XII

Graph of activity versus depth in concrete for cylinder PDBC.

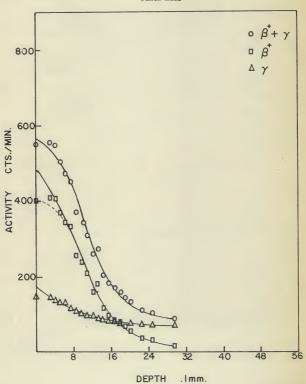
PLATE XII



EXPLANATION OF PLATE XIII

Graph of activity versus depth in concrete for cylinder PDB2.

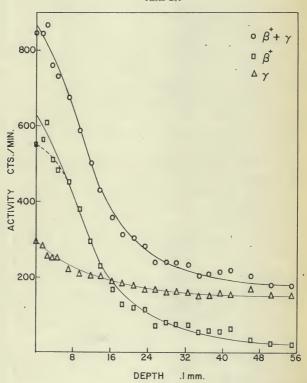
PLATE XIII



EXPLANATION OF PLATE XIV

Graph of activity versus depth in concrete for cylinder PDB1F.

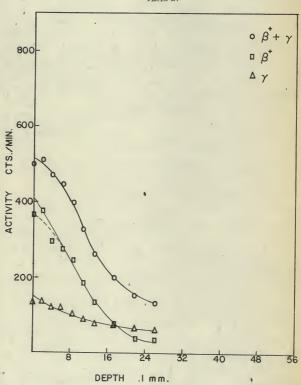
PLATE XIV



EXPLANATION OF PLATE XV

Graph of activity versus depth in concrete for cylinder MAC.

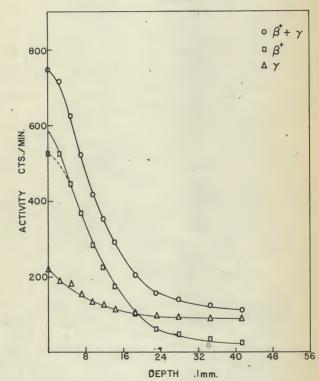
PLATE XV



EXPLANATION OF PLATE XVI

Graph of activity versus depth in concrete for cylinder MAL.

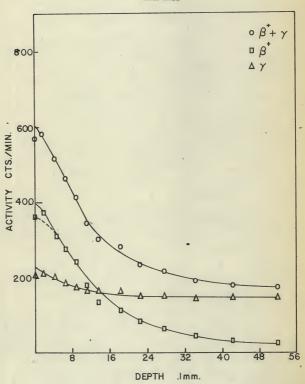




EXPLANATION OF PLATE XVII

Graph of activity versus depth in concrete for cylinder MCC.

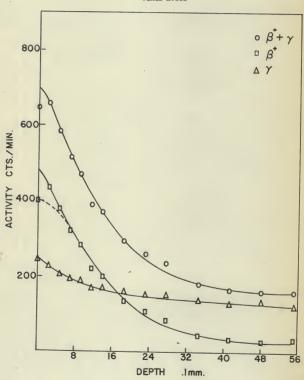




EXPLANATION OF PLATE XVIII

Graph of activity versus depth in concrete for cylinder MCl.

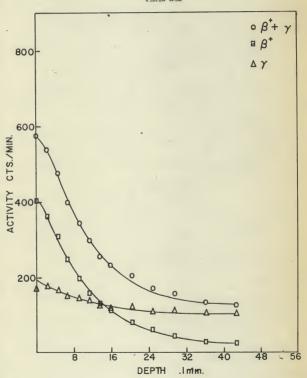
PLATE XVIII



EXPLANATION OF PLATE XIX

Graph of activity versus depth in concrete for cylinder MBC.

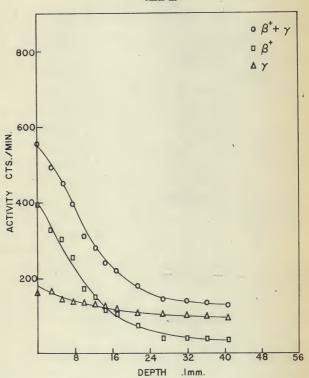




EXPLANATION OF PLATE XX

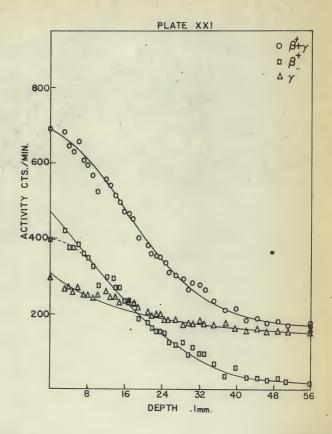
Graph of activity versus depth in concrete for cylinder MBl.

PLATE XX



EXPLANATION OF PLATE XXI

Graph of activity versus depth in concrete for cylinder MBIF.



EXPLANATION OF PLATE XXII

Graph of of versus depth for cylinder FDAC.

PLATE XXII

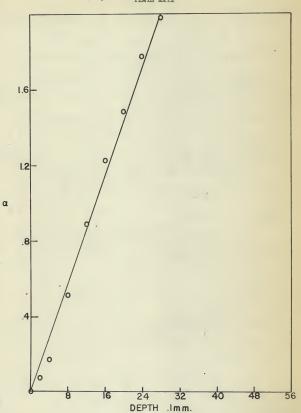


Table 2. Summary of data obtained from Plates VII-XXII.

	: :	Dx 101	o cm ²	Area	under	: 8	C _o	Area	under	: B+	c _o
Mix: by: Wt.:	Addi- :	Dixie :	dusa	: Dixie	: me-	: Dixie	: dusa	: Dixie	: dusa	:Dixie	: dusa
1:4	None	2.1	3.2	.74	1.00	75	77	4.46	5.10	419	390
	N-Tair-R	3.1	4.0	1.22	1.63	98	135	5.73	7.37	500	502
1:5	None	3.1	4.3	.81	.88	72	80	5.43	5.05	460	378
	N-Tair-R	3.3	3.4	1.14	1.84	80	73	6.15	4.80	465	360
1:6	None	4.6	5.5	1.46	1.10	90	65	6.11	5.85	460	375
	N-Tair-R	6.3	4.3	1.24	1.19	65	117	6.54	6.97	373	460
1:5	30% Sub Fly-Ash+ N-Tair-R	5.6	.0.0	2,15	3,16	140	145	9.44	10.00	625	435

Table 3. Average values, illustrating trends.

Term	: Cement	* Additions	: Average : Value
D x 10 ¹⁰ em ² see	Penn Dixie	None N-Tair-R 30% Sub Fly-Ash +N-Tair-R	3.3 4.2 5.6*
	Medusa	None N-Tair-R 30% Sub Fly-Ash tN-Tair-R	4.7 4.3 10.0*
Area Under Curves	Penn Dixie	None N-Tair-R 30% Sub Fly-Ash +N-Tair-R	1.00 1.20 2.15*
	Medusa	None N-Tair-R 30% Sub Fly-Ash +N-Tair-R	•99 1.55 3.16*
Co ets	Penn Dixie	None N-Tair-R 30% Sub Fly-Ash +N-Tair-R	79 87 140*
	Medusa	None N-Tair-R 30% Sub Fly-Ash +N-Tair-R	74 108 145*
rea Under \$\begin{align*} \text{Curves} \end{align*}	Penn Dixie	None N-Tair-R 30% Sub Fly-Ash +N-Tair-R	5.33 6.14 9.44*
	Medusa.	None N-Tair-R 30% Sub Fly-Ash +N-Tair-R	5.33 6.38 10.00*
Co min	Penn Dixie	None N-Tair-R 30% Sub Fly-Ash +N-Tair-R	446 446 625*
Only one sevil	Medusa	None N-Tair-R 30% Sub Fly-Ash +N-Tair-R	381 441 435*

^{*}Only one sample of each type of cement was tested in the case of the fly-ash substituted concrete.

DISCUSSION OF RESULTS

It was found in all the samples tested that the β curves shown in Plates VII to XX tended to level off at between 10 and 30 counts/minute above the base line. This was thought to be due to the natural radio-activity of the concrete. In support of this theory, one concrete cylinder which had not been treated with radioactive sodium was tested and showed an activity of about 10 counts/minute above background. Therefore, in calculating the values of α , the base line was assumed to be the line to which the β curve became asymptotic.

In most cases it was noted that the initial surface count was significantly lower than the counts at depths of .1 or .2 mm. This was believed to be due to a loss of sodium which occurred when the cylinders were rinsed after being removed from the radioactive solution. Hence, the solid line curves were considered to be more indicative than the dotted line curves and were used in the calculation of \propto .

In studying the data obtained, trends were observed. The initial χ and β^+ activities and the areas under the χ and β^+ curves, which were proportional to the amount of sodium in the cylinder, were given with the D values in Tables 2 and 3. It was noted, in general, that the amount of sodium moving into the concrete increased with increasing ag regate-cement ratios, with the use of air-entrainment, and to a greater degree with the use of 30% substituted fly-ash plus air-entrainment. The Medusa cements, on the average, appeared to contain more radioactive sodium than the Fenn Dixie cements.

There were discrepencies in some of the samples tested but this was expected due to the presence of aggregate in the cement. The addition of

the aggregate increased the disorder of the system over the small depths of about .5 cm to which the sodium diffused. If the sodium had moved over large distances in comparison with the aggregate size, this disorder would have decreased. It was observed that more consistent data was obtained from the 1:4 cement aggregate ratios than from either the 1:5 or 1:6 cement aggregate ratios.

The increased amounts of sodium entering the concretes in which air was entrained was explained by the greater porosity produced in the concrete. A study of diffusion processes has shown that surface diffusion is greater than either grain-boundary or lattice diffusion (Barrer, 1). It seemed possible therefore, that some diffusion of sodium had taken place along the surfaces of the aggregate and that this might be an explanation of the increase of sodium in the concrete with higher aggregate-cement ratios.

The diffusion along the surface of the aggregate seemed particularly likely, since the area of cement presented to the sodium solution would be less, the higher the aggregate-cement ratio.

It has been shown that the substitution of fly-ash tends to increase the density of the concrete and reduce its porosity (Chilcote, 4). However, since air-entrainment was utilized in conjunction with the fly-ash, it was assumed that the porosity would be nearly the same as that of the other air-entrained concretes. In attempting to explain the large amounts of sodium moving into the fly-ash substituted concretes, two theories were suggested. The first suggestion was that as the sodium moved into the concrete, the ions in the leading edge combined chemically with the fly-ash, or compounds resulting from the chemical reaction between the fly-ash and cement, and were removed from the liquid. This maintained a high concentration gradient in the aqueous solution within the grain boundaries. Consequently, this high

concentration gradient tended to increase the amount of sodium moving into the concrete. The second sugrestion was that the fly-ash, in reacting with the cement, removed the major part of the free lime (Lea, 7). Hence the lime was not available for reaction with the incoming Na,SO, and the formation of insoluble gypsum was prevented. Gypsum was believed to form in the pores of the concrete and, since it has a greater volume per mole than lime, it seemed logical that the porosity of the concrete would decrease (Burke and Pinckney, 3). Therefore, the fly-ash caused the porosity to remain relatively constant, whereas in the other concretes tested the porosity was lowered. It was interesting to note that in the work by McConnell (McConnell, 8), the addition of fly-ash appeared to hinder the movement of the sodium. However, he did not use air-entrainment with his fly-ash substituted concretes, and hydrostatic pressure contributed to the movement of the sodium. Hence, the movement of sodium observed was possibly a function of the water movement rather than the diffusion of the sodium, as was presumed in this work.

An explanation of the apparent variation in sodium movement with the type of cement used was not attempted at this time, as additional data, needed to confirm this effect, are still to be taken.

CONCLUSIONS

The purpose of this work was to study the movement of sodium in various types of concrete. It was found that the use of air-entrainment increased the amount of sodium moving into the concrete, and the use of fly-ash plus air-entrainment further increased the amounts. These results were explained on the basis of the increased porosity in the case of the air-entrained

concretes, and on the basis of a chemical reaction and increased concentration gradient in the case of the fly-ash substitute plus air-entrained concrete. Probably reduction of gypsum formation, with resultant loss of porosity, was also effective.

Addition of more aggregate to the concrete tended to increase the average amount of sodium entering; this was explained as being due to increased rate of diffusion along the surfaces of the aggregate. No explanation was offered for the difference brought about by the use of different cements.

Because of the heterogeniety of the concrete, it was felt that more samples should be tested to confirm the trends noted here.

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APPENDIX

A sample calculation of D is given below using the data obtained from cylinder PDAC.

x cm	i	C cts/min :	1 - <u>C</u>	· ×
0	- 1	419 = C ₀		
.02		385	.082	.073
.04		339	.191	.171
.08		197	.530	.511
.12		87	.792	.890
.16		35	.917	1.226
.20		15	.964	1.483
.24		5	.988	1.776
.28		2	.9 95	1.984

The first two columns were obtained from Plate VIII. $1-\frac{C}{C_0}$ was calculated from the second column and C was obtained by using a table of probability integrals and the value of $1-\frac{C}{C_0}$ (see page 2). The values of C were plotted against x as shown in Plate XXII. The alope C) was thus found to be .125. The value was inserted into the equation for D,

$$D = \frac{1}{4t} \left(\frac{x}{\alpha}\right)^2$$

where t = 2.38 x 10^7 sec. By substituting the given values of t and ($\frac{x}{\alpha}$) and solving, one obtains the following value for D

$$D = 2.1 \times 10^{-10} \text{ cm}^2/\text{sec.}$$

DIFFUSION OF SODIUM IN VARIOUS TYPES OF CONCRETE

by

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AN ABSTRACT OF A THESIS

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KANSAS STATE COLLEGE OF AGRICULTURE AND APPLIED SCIENCE It was the purpose of this experiment to study the movement of sodium ions in various types of concrete. This was accomplished by using radio-active tracer techniques for detecting and measuring the amount of sodium present at various depths in concrete cylinders, and from this data determining the diffusion constant, D.

The concrete cylinders were two inches in diameter and three inches long. The cylindrical surfaces were coated with paraffin, but the end faces were left exposed. This allowed the sodium to move through the end faces only; hence, the movement of sodium could be considered as linear diffusion into a semi-infinite solid. The diffusion constant was assumed to be independent of the concentration.

Since the concentrations appeared only in ratios, it was not necessary to know the true concentration. Instead, the counting rates obtained were used in the calculations.

A surface grinder was employed to remove small increments of concrete from the end faces of the cylinders, so that the activity could be counted at various depths. Necessary precautions were taken to prevent radiation hazards during grinding operations.

The \$f^* count was utilized in all calculations. Since \$f^* radiation was also present it was first necessary to count \$f^* f_\$, and then filter out the \$f^* f_*, counting only the \$f^* f_*. The difference in the two counts gave the true count.

The data obtained for the fourteen types of concrete tested showed certain trends. It was found that the use of air entrainment increased the amount of sodium moving into the concrete, and the use of fly ash plus air entrainment further increased the amounts. These results were explained on the basis of the increased porosity in the case of the air-entrained concretes, and on the

basis of a chemical reaction and an increased concentration gradient in the case of the fly-ash substituted plus air-entrained concretes. Probably reduction of gypsum formation, with resultant loss of porosity, was also effective.

Addition of more aggregate to the concrete tended to increase the average amount of sodium entering; this was explained as being due to increased rate of diffusion along the surfaces of the aggregate. No explanation was offered for the difference brought about by the use of different cements.

Because of the heterogenisty of the concrete, it was felt that more samples should be tested to confirm the trends noted here.

