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**Diffusion in crystalline rocks of some  
sorbing and nonsorbing species**

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The values of the Freundlich parameter  $k_f$  in Table I (p. 6) are given with the concentration  $q$  in mg/g, instead of in mg/kg according to the notation list. The  $k_f$ -values should therefore be:

0.89	instead of	$8.9 \cdot 10^{-4}$
1.1	"	$1.1 \cdot 10^{-3}$
2.7	"	$2.7 \cdot 10^{-3}$
9.8	"	$9.8 \cdot 10^{-3}$
3.7	"	$3.1 \cdot 10^{-3}$
6.1	"	$6.1 \cdot 10^{-3}$
1.0	"	$1.0 \cdot 10^{-3}$
3.9	"	$3.9 \cdot 10^{-3}$
6.9	"	$6.9 \cdot 10^{-3}$
30	"	$3.0 \cdot 10^{-2}$

These  $k_f$ -values are also given in fig. 3, 4 and 5

Fig. 3:  $q = 9.8 \cdot 10^{-3} \cdot c_p^{0.54}$  should be  $q = 9.8 \cdot c_p^{0.54}$   
 $q = 3.0 \cdot 10^{-2} \cdot c_p^{0.54}$  "  $q = 30 \cdot c_p^{0.54}$   
 $q = 2.7 \cdot 10^{-3} \cdot c_p$  "  $q = 2.7 \cdot c_p$   
 $q = 6.9 \cdot 10^{-3} \cdot c_p$  "  $q = 6.9 \cdot c_p$

Fig. 4:  $q = 1.1 \cdot 10^{-3} \cdot c_p^{0.66}$  should be  $q = 1.1 \cdot c_p^{0.66}$   
 $q = 3.9 \cdot 10^{-3} \cdot c_p^{0.66}$  "  $q = 3.9 \cdot c_p^{0.66}$   
 $q = 8.9 \cdot 10^{-4} \cdot c_p$  "  $q = 0.89 \cdot c_p$   
 $q = 1.0 \cdot 10^{-3} \cdot c_p$  "  $q = 1.0 \cdot c_p$

Fig. 5:  $q = 6.1 \cdot 10^{-3} \cdot c_p^{0.54}$  should be  $q = 6.1 \cdot c_p^{0.54}$   
 $q = 3.7 \cdot 10^{-3} \cdot c_p$  "  $q = 3.7 \cdot c_p$

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NONSORBING SPECIES

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This report concerns a study which was conducted for SKBF/KBS. The conclusions and viewpoints presented in the report are those of the author(s) and do not necessarily coincide with those of the client.

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AND NONSORBING SPECIES

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## SUMMARY

Laboratory experiments to determine the sorption and the rate of diffusion of cesium and strontium in pieces of granite have been performed. The effective diffusivity,  $D_p \cdot \epsilon_p$  was found to be  $1 - 2 \cdot 10^{-12} \text{ m}^2/\text{s}$  for both cesium and strontium.

The diffusion of non-sorbing species in granites and other rock materials have been studied in laboratory scale. The non-sorbing species were iodide, tritiated water, Cr-EDTA and Uranine. In granites the effective diffusivities were determined to be  $0.7 - 1.3 \cdot 10^{-13} \text{ m}^2/\text{s}$  for iodide and  $1.3 - 1.8 \cdot 10^{-13} \text{ m}^2/\text{s}$  for tritiated water.

Electrical resistivity measurements in salt water saturated rock cores have been performed. The resistivity is measured in the saturated core and in the salt solution with which the core has been saturated. The ratio between these two resistivities has a direct relation to the ratio of the effective diffusivity for a component in the rock material and the diffusivity in free water for the same component.

The results from the electrical resistivity measurements and the experiments with diffusion of non-sorbing species are in fair agreement. The effective diffusivity for cesium and strontium (sorbing species) are, however, more than ten times higher than expected from the results of diffusion of non-sorbing species and the electrical resistivity measurements. This is interpreted as an effect of surface diffusion.

## CONTENTS

	Page
1. Introduction	1
2. Sorption of strontium and cesium on granite	2
2.1 Introduction	2
2.2 Experimental	2
2.3 Results	3
2.4 Determination of the sorption capacity and the rate of sorption	4
3. Diffusion of non-sorbing ions in rock material	9
3.1 Diffusion of tritiated water in granite	9
3.1.1 Experimental	9
3.1.2 Results	10
3.1.3 Determination of the diffusivity	10
3.2 Diffusion of iodide, Uranine and Cr-EDTA in rock materials	12
3.2.1 Experimental	12
3.2.2 Results	13
3.2.3 Determination of the diffusivities	14
4. Electrical resistivity measurements in saturated rocks, in order to determine the diffusivity	16
4.1 Theory	16
4.2 Experimental	16
4.3 Results	17
5. Porosity determinations	19
6. Discussion and conclusions	20
Notation	
References	
Appendix	
Figures	

## 1. INTRODUCTION

Questions related to the final disposal of the nuclear wastes is studied intensively in many countries. The Swedish concept for a final repository for radioactive waste is to emplace the waste in crystalline rock. Canisters containing the waste are placed in holes in stable rock at about 500 m depth. The nuclides may, however, eventually leak out from the canisters and be transported with the moving ground water in fissures in the bedrock. To estimate the velocity of the moving nuclides in the fissures it is important to know the interaction mechanisms between the nuclides and the rock. The nuclides may sorb on the fissure surfaces and they may also diffuse through the stagnant fluid in the micropores of the rock and sorb on the micropore surfaces as well. This may have a strong impact on the retardation of the radionuclides. (1)

Three types of experiments in the laboratory scale to determine the sorption and the diffusion of nuclides in crystalline rocks have been performed.

- o Sorption and diffusion experiments with cesium and strontium in two different granites.
- o Diffusion experiments with non-sorbing species (iodide, Uranine and Cr-EDTA) in different rock materials.
- o Electrical resistivity measurements in salt water saturated rock cores.

## 2. SORPTION OF STRONTIUM AND CESIUM ON GRANITE

### 2.1 Introduction

In an earlier investigation (2) the diffusion and sorption of cesium and strontium in crushed granite particles was studied. During crushing of the granite to smaller particles, microfissures and cracks may have been induced. It is therefore possible that the adsorption capacities and determined diffusivities in crushed granite particles are higher than they would be in the undisturbed rock. With that in mind it is interesting to investigate the sorption capacity and the diffusivity for strontium and cesium in cut larger granite pieces.

The granite in these experiments is from Finnsjön (quartz-granodiorite) outside Forsmark on the east coast of Sweden and from the Stripa mine (quartz-monzonite) in central Sweden. The same type of granites were used as in the previous experiments with crushed particles.

### 2.2 Experimental

The Finnsjö granite was in the form of cylindrical plates of 41 mm diameter and 5 mm thickness. They were taken from a drill core from about 100 m depth.

The Stripa granite was in the form of square plates 35 x 35 x 5 mm. These pieces were sawed out from a larger drill core from about 340 m depth in the Stripa mine.

Before starting the adsorption experiments, the granite pieces were saturated with "synthetic" ground water (the composition proposed by B. Allard, see Appendix 1) by the following method: The pieces were heated at 90° C in vacuo for three days. The dried pieces were then placed above a pan of "synthetic" ground water in a vacuum chamber. A pressure close to the boiling point of the water (about 25 mm Hg) was maintained for several hours, and then the samples were dropped into the water.



After being in contact with the synthetic ground water for about a week ten pieces were put in a rack of teflon with a distance of 4 mm between the pieces (Fig. 1). Because of this distance both sides of the plates would be in contact with the solution in the adsorption experiment. The rack of teflon with the granite pieces was placed in a bottle, and 1,100 ml of the solution containing strontium or cesium or both was added. Altogether 5 experiments were performed, strontium adsorption on Finnsjö granite and Stripa granite, cesium adsorption on Finnsjö granite and on Stripa granite and one experiment with simultaneous adsorption of cesium and strontium on Finnsjö granite. The initial concentration of strontium was about 10 ppm and of cesium about 15 ppm. The solid to liquid ratio was in the experiments with Finnsjö granite 178.2 g/1,100 ml and in the experiments with Stripa granite 165.4 g/1,100 ml.

All the bottles were placed in a shaking bath and the water in the bath was tempered at 25° C. At intervals approximately following a geometric progression, small fractions (1 ml) of the solutions were taken out, and the concentration of cesium and strontium was measured by atomic absorption spectrometry.

### 2.3 Results

The relative concentration,  $c/c_0$ , of strontium and cesium versus time are shown in Fig. 2 for both Stripa granite and Finnsjö granite. We assume here that equilibrium is obtained after about 10,000 hours. The adsorption capacity for strontium on Stripa granite is higher than for cesium, but the opposite relation is found for the Finnsjö granite. These results agree with those found for adsorption of cesium and strontium on crushed Finnsjö and Stripa granite (2).

The adsorption capacity for the Finnsjö granite is higher than for the Stripa granite. The solid to liquid ratio are not the same for the two granites, but if the equilibrium concentrations are related to the same solid to liquid ratio it would still give a much higher adsorption capacity for the Finnsjö granite. This is also in agreement with earlier results (2).

Fig. 2 also shows the results of simultaneous adsorption of strontium and cesium on Finnsjö granite. The concentration decrease for both strontium and cesium is changed when the other component is present. When comparing with the result from the experiment with separate adsorption, the adsorption capacity for strontium has increased but for cesium it has decreased. The results from simultaneous adsorption on crushed Finnsjö granite (2) showed a decrease in the adsorption capacity for cesium but no change for strontium compared with separate adsorption.

#### 2.4 Determination of the sorption capacity and the rate of sorption

In the model below the granite pieces are treated as porous bodies. The nuclides diffuse through the micropores and are then sorbed on the inner surfaces of the solid. The diffusion equation which describes this sorption process for onedimensional diffusion is

$$\epsilon_p \cdot \frac{\partial c_p}{\partial t} + \rho_s \cdot \frac{\partial q}{\partial t} = D_p \cdot \epsilon_p \cdot \frac{\partial^2 c_p}{\partial x^2} \quad (1)$$

where  $\epsilon_p$  is the porosity of the rock pieces,  $c_p(x,t)$  and  $q(x,t)$  are concentrations in the micropore fluid and in the solid phase respectively,  $D_p$  is the pore diffusivity and  $x$  is the length coordinate. Local chemical equilibrium is assumed at every point inside the pieces. The relation between the concentrations  $c_p$  and  $q$  within the pieces can be described by the following equilibrium relation, the Freundlich isotherm.

$$q = k_f \cdot c_p^\beta \quad (2)$$

In the sorption experiments, nuclides have been diffusing from a limited solution volume  $V$  to  $n$  pieces of granite, each with a thickness of  $2\ell$ . The area of the piece normal to the diffusing direction is  $A$ . The mass balance for the nuclide then is

$$V \cdot \frac{\partial c}{\partial t} = -n \cdot A \cdot D_p \cdot \epsilon_p \cdot \frac{\partial c_p}{\partial x} \Big|_{x = \pm \ell} \quad (3)$$

where  $c$  is the concentration in the bulk fluid.

The influence of the edges of the pieces is neglected in this analysis. The area of the edges is about 20 % of the total surface.

The film diffusion resistance at the surface of the pieces is negligible in the experiments made here.

The initial and boundary conditions for sorption from a volume with an initial concentration of  $c_0$  to rock pieces initially free from nuclides are

$$c_p(x,0) = q(x,0) = 0 \quad -l \leq x \leq +l \quad (4)$$

$$c(0) = c_0 \quad (5)$$

$$c_p(\pm l, t) = c \quad t > 0 \quad (6)$$

$$\frac{\partial c_p}{\partial x}(0, t) = \frac{\partial q}{\partial x}(0, t) = 0 \quad t > 0 \quad (7)$$

In these sorption experiments on pieces of granite no separate determination of the equilibrium relation i.e. the Freundlich isotherm (Eq. (2)) has been made. In the investigation with adsorption and diffusion of cesium and strontium in crushed Finnsjö and Stripa granite (2), it was found that the isotherm was near linear ( $\beta \approx 1$ ) for strontium adsorption on both Finnsjö and Stripa granite. For cesium adsorption, however, the isotherm was found to be non-linear, with the Freundlich exponent  $\beta = 0.54$  for Finnsjö granite and  $\beta = 0.66$  for Stripa granite.

In the experiments with larger pieces of granite we assume that the Freundlich exponent  $\beta$  has the same value as in the experiments with crushed granite. From that assumption and the equilibrium concentrations the constant  $k_f$  in the Freundlich isotherm can be calculated.

Table I shows the values of the parameters in the Freundlich isotherm for the adsorption experiments on pieces of granite. In the experiments with simultaneous adsorption the exponent  $\beta$  is assumed to be the same as in the experiments with separate adsorption. As a comparison the corresponding values for adsorption on the largest particles (4-5 mm) (2) are included in Table 1. The constant  $k_f$  is in all cases larger for the crushed particles than for the pieces. The difference is

largest for cesium. This means that the adsorption capacity is higher for the particles, and it might be an effect of microfissures and cracks induced during the crushing of the granite.

		Adsorption on granite pieces		Adsorption on granite particles, (2) $d_p = 4-5$ mm	
		$\beta$	$k_f$	$\beta$	$k_f$
Stripa	Strontium	1	$8.9 \cdot 10^{-4}$	1	$1.0 \cdot 10^{-3}$
	Cesium	0.66	$1.1 \cdot 10^{-3}$	0.66	$3.9 \cdot 10^{-3}$
Finnsjö	Strontium	1	$2.7 \cdot 10^{-3}$	1	$6.9 \cdot 10^{-3}$
	Cesium	0.54	$9.8 \cdot 10^{-3}$	0.54	$3.0 \cdot 10^{-2}$
Finnsjö simultaneous sorption	Strontium	1	$3.7 \cdot 10^{-3}$		
	Cesium	0.54	$6.1 \cdot 10^{-3}$		

Table I: Parameter values in the Freundlich isotherm.

To estimate the diffusivities one needs to solve the diffusion equations. Crank (3) gives the analytical solution to the equations for a linear isotherm ( $\beta = 1$ ). For the non-linear isotherm the equations have been solved numerically by a computer program (TRUMP) developed at the Lawrence Livermore Laboratories (4).

Fig. 3, 4 and 5 show the fit of the diffusion model to the experimental uptake curve for cesium and strontium. Also the fit of the model with the Freundlich isotherm found for the largest crushed particles (2) are presented. If equilibrium is not obtained after 10,000 hours, as we assume it does, then the diffusivity  $D_p \cdot \epsilon_p$  lies somewhere between the values for equilibrium after about 10,000 hours and equilibrium according to the crushed particles. The diffusion model gives, however, a better fit to the experimental values for the former case.

In Table II the estimated diffusivities are presented. This diffusivity,  $D_p \cdot \epsilon_p$ , is an effective diffusivity, and the relation to the diffusivity in free water,  $D_v$ , is

$$D_p \cdot \epsilon_p = D_v \cdot \epsilon_p \cdot \delta_D / \tau^2 \quad (8)$$

where  $\epsilon_p$  is the porosity which restricts the available mean area for transport,  $\delta_D$  is the constrictivity which implies that the pores may be constricted in some parts, and  $\tau$  is the tortuosity which accounts for the effect of a lengthening of the path.

The relation between the effective diffusivity and the diffusivity in free water gives a value to the formation factor  $\epsilon_p \cdot \delta_D / \tau^2$ , which consists of material properties. This factor has been calculated and is presented in Table II.

The diffusivity for cesium and strontium in free water was determined by the following equation (5)

$$D_{AB} = \frac{R \cdot T}{F^2} \frac{(1/n_+ + 1/n_-)}{(1/\lambda_+^0 + 1/\lambda_-^0)} \quad (9)$$

Here R is the gas constant, F the Faraday's constant, T the absolute temperature,  $\lambda_+^0$  the cationic conductance at infinite dilution,  $\lambda_-^0$  the anionic conductance at infinite dilution,  $n_+$  the valence of cation and  $n_-$  the valence of anion. The anion was chloride. The diffusivity for strontium in water was determined to  $1.3 \cdot 10^{-9} \text{ m}^2/\text{s}$  and for cesium  $2.0 \cdot 10^{-9} \text{ m}^2/\text{s}$ .

The interaction of ions in a multi-ion system is important when the several ion conductances differ greatly. An estimation of the diffusivities of cesium and strontium in the synthetic ground water has been made by the following equation (6):

$$n_+ \cdot N_+ = - \frac{R \cdot T}{F^2} \cdot \frac{\lambda_+}{n_+} \left( G_+ - n_+ \cdot c_+ \frac{\sum \lambda_+ \cdot G_+ / n_+ - \sum \lambda_- \cdot G_- / n_-}{\sum \lambda_+ \cdot c_+ + \sum \lambda_- \cdot c_-} \right) \quad (10)$$

Where  $N_+$  is the diffusion flux density of cation,  $c_+$  and  $c_-$  the corresponding ion concentrations and  $G_+$  and  $G_-$  are the concentration gradients in the direction of diffusion. The gradients were assumed to be linear and the diffusion to be into pure water. With this equation the diffusivity for strontium in synthetic ground water was determined to  $1.0 \cdot 10^{-9} \text{ m}^2/\text{s}$  and for cesium  $2.4 \cdot 10^{-9} \text{ m}^2/\text{s}$ .

	Finnsjö granite		Stripa granite	
	$D_p \cdot \epsilon_p$ (m <sup>2</sup> /s)	$\epsilon_p \cdot \delta_D / \tau^2$	$D_p \cdot \epsilon_p$ (m <sup>2</sup> /s)	$\epsilon_p \cdot \delta_D / \tau^2$
Strontium	$2.2 \cdot 10^{-12}$	$1.7 \cdot 10^{-3}$ a) $2.2 \cdot 10^{-3}$ b)	$1.3 \cdot 10^{-12}$	$1.0 \cdot 10^{-3}$ a) $1.3 \cdot 10^{-3}$ b)
Cesium	$1.6 \cdot 10^{-12}$	$0.80 \cdot 10^{-3}$ a) $0.67 \cdot 10^{-3}$ b)	$1.0 \cdot 10^{-12}$	$0.50 \cdot 10^{-3}$ a) $0.42 \cdot 10^{-3}$ b)
Strontium, simultaneous sorption	$3.1 \cdot 10^{-12}$	$2.4 \cdot 10^{-3}$ a) $3.1 \cdot 10^{-3}$ b)		
Cesium, simultaneous sorption	$1.0 \cdot 10^{-12}$	$0.50 \cdot 10^{-3}$ a) $0.43 \cdot 10^{-3}$ b)		

a)  $D_v$  from Eq. (9)

b)  $D_v$  from Eq. (10)

Table II: Effective pore diffusivities and values of the formation factor  $\epsilon_p \cdot \delta_D / \tau^2$  for strontium and cesium diffusion in Finnsjö and Stripa granite.

### 3. DIFFUSION OF NON-SORBING IONS IN ROCK MATERIAL

#### 3.1 Diffusion of tritiated water in granite

In this experiment the diffusion of tritiated water (THO) through a piece of granite was determined. The piece of granite (5 mm thick and 41 mm in diameter) was taken from a drillcore from Finnsjö (quartz-granodiorite) just outside Forsmark on the east coast of Sweden. Two experiments were performed one with THO in distilled water and one with THO in "synthetic" ground water.

##### 3.1.1 Experimental

The apparatus is shown schematically in Fig. 6. It consists of a diffusion cell, a unit for taking out samples, two pumps and a storage bottle. The piece of granite was placed in the diffusion cell and on one side of the piece tritiated water was circulated (high concentration system) and on the other side of the piece distilled water or synthetic ground water (low concentration system).

The diffusion cell (Fig. 7) consists of a cylindrical vessel and a piston. The piston is fixed in the vessel with four bolts. On each side of the granite piece there is a porous plate and a plate with concentric channels. Between the inner wall of the vessel and the granite piece two conical rings of teflon are placed, in order to prevent leakage between the two systems.

Before the granite piece was placed in the diffusion cell it was saturated with distilled water or synthetic ground water by the same method as used in the experiment with cesium and strontium. The activity of the tritiated water in the high concentration system was about 50  $\mu\text{Ci}$ , and the volume 500 ml. The volume of the water in the low concentration system was 80 ml in the experiment with distilled water and 40 ml in the experiment with synthetic ground water. Samples (0.5 ml) were taken out from both the high concentration system and the low concentration system about once a week, and the activity in each sample was measured by a liquid scintillation technique. To keep the volume in the low concentration system constant 0.5 ml distilled water or synthetic ground water was added each time a sample had been taken out.

### 3.1.2 Results

The total amount of tritiated water which had diffused through the granite at different times is shown in Fig. 8. The amount of tritiated water is expressed in counts per minute (cpm). The activity in the high concentration system was about 25,000 cpm/0.5 ml. The background activity in the low concentration system was about 55 cpm/0.5 ml.

In the experiment with synthetic ground water there was a large increase in the activity during the first ten days. This large increase is probably caused by an initial leakage between the two systems. After the first ten days the increase in activity is about the same for the experiments with synthetic ground water and distilled water.

### 3.1.3 Determination of the diffusivity

The rate of transfer of diffusing substance through a porous plate of cross-sectional area  $A$ , can be described by Fick's law of diffusion

$$N = -D_e \cdot A \cdot \frac{dc}{dx} \quad (11)$$

where  $N$  is the flowrate of diffusing substance,  $c$  the concentration of diffusing substance,  $x$  the distance in the diffusing direction, and  $D_e$  is the effective diffusion coefficient.

Consider the case of diffusion through a plane sheet of thickness  $\ell$  and effective diffusion coefficient  $D_e$ , whose surfaces,  $x=0$ ,  $x=\ell$  are maintained at constant concentrations  $c_1$ ,  $c_2$  respectively. After a time a steady state is reached, in which the concentrations remain constant at all points of the sheet, and the rate of transfer of diffusing substance is the same across all sections of the sheet. The flowrate of diffusing substance is then given by the equation

$$N = -D_e \cdot A \cdot \frac{dc}{dx} = D_e \cdot A (c_1 - c_2)/\ell \quad (12)$$

Fig. 8 shows that steady state is obtained after seven days in the experiment with distilled water, and after about ten days in the experiment with synthetic ground water, assuming that the initial leak has been sealed. The flowrate is determined by linear regression. The



concentration in the low concentration system is negligible compared to the concentration in the high concentration system, and the latter concentration is constant with time.

By using Eq. (12) the diffusivity  $D_e$ , which in this case is an effective pore diffusivity  $D_p \cdot \epsilon_p$ , for tritiated water in Finnsjö granite was found to be  $1.3 \cdot 10^{-13} \text{ m}^2/\text{s}$  for the experiment with distilled water and  $1.8 \cdot 10^{-13} \text{ m}^2/\text{s}$  for the experiment with synthetic ground water.

The relation between the effective pore diffusivity and the diffusivity in free water gives a value of the formation factor  $\epsilon_p \cdot \delta_p/\tau^2$  (according to Eq. (8)), which consists of material properties. The self diffusion coefficient for water was determined to  $2.1 \cdot 10^{-9} \text{ m}^2/\text{s}$  by the following equation (7):

$$\frac{D_{AA} \cdot \mu_A}{k \cdot T} = \frac{100}{2 \cdot \pi} \left( \frac{\tilde{N}}{\tilde{V}_A} \right)^{1/3} \quad (13)$$

$D_{AA}$  is the self diffusion coefficient,  $\mu_A$  is the viscosity,  $k$  is the Boltzmann constant,  $T$  is the temperature,  $\tilde{N}$  is the Avogadro's number and  $\tilde{V}_A$  is the molar volume.

The diffusivity for THO in water is  $2.4 \cdot 10^{-9} \text{ m}^2/\text{s}$  (8).

Table III presents the determined  $D_p \cdot \epsilon_p$  and values of the formation factor  $\epsilon_p \cdot \delta_D/\tau^2$  for the experiment in distilled water and in synthetic ground water.  $D_v = 2.4 \cdot 10^{-9} \text{ m}^2/\text{s}$  has been used when calculating the formation factor from Eq. (8).

	Finnsjö granite	
	Distilled water	Synthetic ground water
$D_p \cdot \epsilon_p \text{ (m}^2/\text{s)}$	$1.3 \cdot 10^{-13}$	$1.8 \cdot 10^{-13}$
$\epsilon_p \cdot \delta_D/\tau^2$	$5.4 \cdot 10^{-5}$	$7.5 \cdot 10^{-5}$

Table III: Effective pore diffusivities and values of the formation factor for diffusion of tritiated water in Finnsjö granite.

### 3.2 Diffusion of iodide, Uranine and Cr-EDTA in rock materials

The diffusion of iodide in different rock materials was determined in a series of experiments. The rock materials were granites from Finnsjön and Stripa, gneiss from Karlshamn at the west coast of Sweden and gabbro from Vipängen just outside Uppsala. The thickness of all the pieces were 10 mm, but they had different cross sectional areas. Also one experiment with diffusion of Uranine and one with diffusion of Cr-EDTA in Finnsjö granite were performed.

#### 3.2.1 Experimental

A hole, with the same dimension as the piece of rock, was made in a 10 mm thick PVC-plate. The piece of rock was fitted in the hole with silicone glue. The plate with the rock sample was then heated in a vacuum chamber and saturated with distilled water by the same method used in the sorption experiments with cesium and strontium. After saturation two chambers made of transparent PVC were fastened on the PVC plate, one on each side (see Fig. 9). One of the chambers was filled with distilled water and the other was filled with a solution containing either sodium iodide, Uranine or Cr-EDTA. The concentrations were 1 mol/l, 10 g/l and 9.9 g/l respectively.

In some of the experiments a magnetic stirrer was placed at the bottom of each chamber. This was done to eliminate the diffusion resistance in the solution very near the surface of the rock piece (film resistance). This was later shown not to be necessary because the film resistance is negligible compared to the diffusion resistance in the rock material even without stirring.

Samples (10 ml) were taken from the chamber which from the beginning contained of distilled water, and the concentration of the diffusing component was measured. The iodide concentration was measured with an ion selective electrode, the concentration of Uranine with UV-spectrophotometry and the concentration of Cr-EDTA with atomic absorption spectrometry. Each time a sample had been taken out 10 ml of distilled water was added to the chamber to keep the volume in the chamber constant.

Two diffusion experiments with iodide were made on each rock material, and stirring was used in one of the experiments on each rock material. One diffusion experiment with Uranine and one with Cr-EDTA were made on the Finnsjö granite.

To investigate if there was any diffusion through the PVC plate or through the silicone glue, two blank tests were performed. In one of the tests the diffusion of iodide through a 10 mm thick PVC plate was measured. In the other test a hole was made in a 10 mm thick PVC plate and a piece of the same PVC material was fitted in the hole with silicone glue using the same procedure as for the granite samples, and the diffusion of iodide through this plate was measured.

### 3.2.2 Results

The total amount of iodide which had diffused through the rock pieces at different times is shown in Fig. 10, 11 and 12. In each diagram the results from two experiments with the same rock material, one with stirring and one without stirring, are shown. The results from the experiments with Stripa granite (Fig. 10) are in good agreement, and the same thing can be said about the results from the experiments with the Finnsjö granite (Fig. 11). In the experiments with gneiss, however, (Fig. 12) the results do not agree. The difference is not caused by stirring, because that did not affect the experiments with Stripa and Finnsjö granite. The temperature in the experiments with stirring was higher than in the experiments without stirring because of heat conduction from the stirrer. It seems that the higher temperature had no effect in the experiments with Stripa and Finnsjö granite so that is probably not the reason either to the difference in the experiments with gneiss. The most probable explanation is that the gneiss pieces are not equal considering mineral composition, grain sizes, porosity or other material properties.

In the iodide diffusion experiments with gabbro the concentrations on the low concentration side were lower than the detection limit for the iodide electrode ( $10^{-6}$  mol/l) even after 200 days. The only thing that can be said about these experiments is that less than  $10^{-6}$  mol iodide has diffused through the gabbro pieces in 200 days. This indicates that the effective diffusivity is less than  $4.0 \cdot 10^{-16}$  m<sup>2</sup>/s.

The blank test showed no diffusion through the 10 mm thick PVC plate. In the other blank test where the diffusion through the silicone glue was determined, a very small amount of iodide (less than 1 % compared with the experiments with rock material) had diffused through the glue. This diffusion was in all cases negligible compared to the diffusion through the rock pieces.

Fig. 13 shows the amount of Cr-EDTA which had diffused through a piece of Finnsjö granite at different times. No results from times shorter than 100 days are given because of problems with the analysis at the beginning of the experiment.

The diffusion of Uranine through a piece of Finnsjö granite is presented in Fig. 14.

### 3.2.3 Determination of the diffusivities

The effective diffusivities were calculated by using Eq. (12). The flowrate of diffusing substance at steady state was determined from Fig. 10 to 14 by linear regression.

The effective diffusivities and the formation factor  $\epsilon_p \cdot \delta_D / \tau^2$  (from Eq. (8)) are presented in Table IV. The diffusivity in free water,  $D_v$ , for iodide is  $1.6 \cdot 10^{-9} \text{ m}^2/\text{s}$  at  $25^\circ \text{ C}$  (8). To correlate this diffusivity to the actual temperatures the following equation is used,

$$\frac{D_1}{D_2} = \frac{T_1}{T_2} \cdot \frac{\mu_2}{\mu_1} \quad (14)$$

where  $D_1$  and  $D_2$  are diffusivities,  $T_1$  and  $T_2$  temperatures and  $\mu_1$  and  $\mu_2$  are viscosities.

	Iodide diffusion			Cr-EDTA diffusion			Uranine diffusion		
	$t(^{\circ}\text{C})$	$D_p \cdot \epsilon_p \text{ (m}^2/\text{s)}$	$\epsilon_p \cdot \delta_D / \tau^2$	$t(^{\circ}\text{C})$	$D_p \cdot \epsilon_p \text{ (m}^2/\text{s)}$	$\epsilon_p \cdot \delta_D / \tau^2$	$t(^{\circ}\text{C})$	$D_p \cdot \epsilon_p \text{ (m}^2/\text{s)}$	$\epsilon_p \cdot \delta_D / \tau^2$
Finnsjö granite diffusion area = $1.32 \cdot 10^{-3} \text{ m}^2$	27	$8.4 \cdot 10^{-14}$	$4.9 \cdot 10^{-5}$	20	$6.9 \cdot 10^{-15}$	$1.6 \cdot 10^{-5}$	20	$2.3 \cdot 10^{-15}$	$0.5 \cdot 10^{-5}$
	21	$7.4 \cdot 10^{-14}$	$5.1 \cdot 10^{-5}$						
Stripa granite diffusion area = $2.92 \cdot 10^{-3} \text{ m}^2$	27	$1.3 \cdot 10^{-13}$	$7.6 \cdot 10^{-5}$	20	$6.9 \cdot 10^{-15}$	$1.6 \cdot 10^{-5}$	20	$2.3 \cdot 10^{-15}$	$0.5 \cdot 10^{-5}$
	19	$1.2 \cdot 10^{-13}$	$8.7 \cdot 10^{-5}$						
Gneiss diffusion area = $1.32 \cdot 10^{-3} \text{ m}^2$	30	$1.4 \cdot 10^{-13}$	$7.6 \cdot 10^{-5}$	20	$6.9 \cdot 10^{-15}$	$1.6 \cdot 10^{-5}$	20	$2.3 \cdot 10^{-15}$	$0.5 \cdot 10^{-5}$
	20	$5.0 \cdot 10^{-14}$	$3.5 \cdot 10^{-5}$						
Gabbro diffusion area = $1.46 \cdot 10^{-3} \text{ m}^2$	21	$<4.0 \cdot 10^{-16}$	$<0.03 \cdot 10^{-5}$	20	$6.9 \cdot 10^{-15}$	$1.6 \cdot 10^{-5}$	20	$2.3 \cdot 10^{-15}$	$0.5 \cdot 10^{-5}$
	18	$<4.0 \cdot 10^{-16}$	$<0.03 \cdot 10^{-5}$						

Table IV: Effective diffusivities and values of the formation factor for diffusion in rock materials.

The diffusivities for Uranine and Cr-EDTA in free water have been estimated by the equation of Hayduk-Laudie (6).

$$D_{AW} = 13.26 \cdot 10^{-5} \cdot \mu_W^{-1.4} \cdot \bar{V}_A^{-0.589} \quad (15)$$

$D_{AW}$  is the diffusion coefficient at infinite dilution,  $\mu_W$  the viscosity of water and  $\bar{V}_A$  the solute molar volume at normal boiling point. With this equation the diffusivity in free water for Uranine was found to be  $4.5 \cdot 10^{-10} \text{ m}^2/\text{s}$  and for Cr-EDTA  $4.2 \cdot 10^{-10} \text{ m}^2/\text{s}$ . These values are rough estimates because of the uncertainty in determining the solute molar volume.

#### 4. ELECTRICAL RESISTIVITY MEASUREMENTS IN SATURATED ROCKS, IN ORDER TO DETERMINE THE DIFFUSIVITY

##### 4.1 Theory

Direct measurements of diffusion in low porosity materials are very time consuming. It is therefore of interest to find a method which can reduce the experimental time. There are some indications that electrical conductivity and molecular diffusion may depend in the same way on the formation factor  $\epsilon_p \cdot \delta_D / \tau^2$  (9). This means that

$$\frac{D_p \cdot \epsilon_p}{D_v} = \frac{\epsilon_p \cdot \delta_D}{\tau^2} = \frac{R_o}{R_s} \quad (16)$$

where  $R_s$  is the resistivity of the salt water saturated rock sample and  $R_o$  is that of the salt water.

The concentration of the salt water solution must not be too low because then the pore surface conductivity might influence the results.

##### 4.2 Experimental

Rock samples were saturated with salt solution (NaCl) using the same method as in the experiments with cesium and strontium (see 2.2). After saturation the samples were dried with a piece of paper on the outer surfaces except on the surfaces that would be in contact with the electrodes. The electrodes were polished copper plates. Between the electrodes and the rock sample a filter paper, saturated with the same salt solution as the rock sample, was placed. The resistance in the rock sample was measured by a conductivity meter (type Wheatstone bridge) at a frequency of 50 Hz. The resistance in the salt solution was measured with the same electrodes and conductivity meter. The electrodes were placed on each side of a transparent PVC cylinder. The cylinder was filled with the salt solution, and the resistance measured.

In an experiment performed by Tom Wallin and his co-workers (10), where the impedance in a saltwater saturated piece of Finnsjö granite was measured at frequencies from  $10^{-2}$  Hz to  $10^6$  Hz, it was found that at a frequency of 50 Hz the measured value was a pure resistance (no capacitance included).

#### 4.3 Results

Two initial investigations were performed. The purpose of these was to decide a suitable concentration of the salt solution and the length of the rock samples (distance between the electrodes). These initial investigations were made on Finnsjö granite rock samples.

Fig. 15 shows the formation factor  $\epsilon_p \cdot \delta_D / \tau^2$  ( $=R_o/R_s$ ) versus the concentration of sodium chloride in the salt solution for three samples of Finnsjö granite ( $\emptyset$  41 mm, length 30 mm). At concentrations below 1 mol/l the factor is dependent on the concentration. That might be an effect of pore surface conductivity. Concentrations of 1 mol/l and higher gives a factor that is almost independent of the concentration. For the subsequent measurements the concentration 1 mol/l was chosen.

In the other initial investigation Finnsjö granite cores with different length ( $l = 10$ -100 mm) were saturated with 1 mol/l sodium chloride solution. The result is presented in Fig. 16 as the formation factor versus the length of the cores. Some of the cores were saturated and the resistivity measured two times. It was found that the shortest cores ( $l = 10$  mm) gave a higher value to the formation factor than the others. The reason for that might be that the shortest cores have more pores going through the whole piece than the longer ones. An other possible explanation is that some of the conductivity occurred on the cylindrical outer surfaces of the cores. When the electrodes were pressed against the ends of the cores a zone of salt solution was spread out on the cylindrical surface near each electrode. When the distance between the electrodes was small (shortest cores) these two zones might have been in contact with each other and conductivity could take place on the outer surface of the core. For the longer cores ( $l = 30$ -100 mm) the formation factor seems to be independent of the core length.

Some measurements of the resistivity in salt water saturated Finnsjö granite cores have been performed by U. Öquist and his co-workers at

the Swedish Geological Survey in Luleå (11). They used an instrument specially developed for measurements of resistivity and induced polarization on rock samples in laboratory conditions. The Finnsjö cores were 50-60 mm long and saturated with 0.025 mol/l or 0.25 mol/l sodium chloride solutions. Their results are presented in Fig. 15 and they also indicate that the formation factor is dependent on the concentration in the low concentration range.

Fig. 17 shows the result from electrical resistivity measurements in Finnsjö and Stripa granite, gneiss and gabbro. The rock samples had a length of 30 mm and they were saturated with a 1 mol/l sodium chloride solution.

The formation factor has about the same value for the Finnsjö and the Stripa granite. Gneiss gives a higher value, and gabbro a somewhat lower value than the granites. The difference between the lowest and the highest value for the same rock material is about a factor 1.5-2.



## 5. POROSITY DETERMINATIONS

Some porosity determinations of the Finnsjö and Stripa granites, gneiss and gabbro have been made by Dr. M. Müller-Vonmoos at the Institut für Grundbau und Bodenmechanik in Zürich (12). The porosities were determined with a mercury porosimeter. The samples were crushed particles and the size was about 5–6 mm. The result is presented in Table V. This investigation also showed that most of the pores in the rock samples had a radius larger than 0.1  $\mu\text{m}$ .

The porosity of larger rock samples ( $\phi$  41 mm, length 10–30 mm) have been determined by the following method. The samples were heated at 90° C in vacuo for three days. The dried samples were then weighed and after that placed above a pan of distilled water in a vacuum chamber. A pressure close to the boiling point of the water (about 25 mm Hg) was maintained for several hours and then the samples were dropped into the water. After being in the water for about two weeks the samples were weighed again (surface dry) and the water content determined. The so determined porosities are also presented in Tabel V.

	Finnsjö granite	Stripa granite	Gneiss	Gabbro
Mercury porosimetry (crushed material)	$9.1 \cdot 10^{-3}$	$3.4 \cdot 10^{-3}$	$4.0 \cdot 10^{-3}$	$3.7 \cdot 10^{-3}$
Water saturation method (large samples)	$1.8-2.1 \cdot 10^{-3}$	$3.1-3.2 \cdot 10^{-3}$		$2.9-3.4 \cdot 10^{-3}$

Table V: Porosities of the rock materials

The porosities obtained by the mercury porosimeter are higher than the porosities obtained by the water saturation method, especially for the Finnsjö granite. In the porosity determination with mercury crushed rock material was used. The Finnsjö particles were taken from crushed material obtained during drilling of some deep holes and the other rock materials were pieces crushed down to a size of about 5–6 mm in an agate stone mortar. The drilling and crushing might have induced cracks and micro fissures and that would give a higher porosity. In the water saturation method larger rock pieces were used (not crushed). It is, however, not certain that the whole piece is saturated with water. If this is the case the water saturation method would give a porosity that is too low.

## 6. DISCUSSION AND CONCLUSIONS

In all the experiments that have been performed the formation factor has been calculated either from determined values of the pore diffusivity or from measured electrical resistivities. If this factor for a certain porous material is known, as well as the diffusivity of a component in free water, then the effective diffusivity for the component in the porous material can be predicted.

Type of experiment		Finnsjö granite	Stripa granite	Gneiss	Gabbro
Diffusion exp.		$5.3-6.2 \cdot 10^{-5}$			
Tritiated water					
conc. range	$10^{-9}$ M	$7.4-8.6 \cdot 10^{-5*}$			
Sorption exp.	Sr	$170-220 \cdot 10^{-5*}$	$98-130 \cdot 10^{-5*}$		
conc. range	$10^{-4}$ M	$66-78 \cdot 10^{-5*}$	$41-49 \cdot 10^{-5*}$		
Simultaneous	Sr	$230-300 \cdot 10^{-5*}$			
sorption	Cs	$43-49 \cdot 10^{-5*}$			
Diffusion exp. Cr-EDTA		$2.0 \cdot 10^{-5}$			
conc. range	$10^{-2}$ M				
Diffusion exp. Uranine		$0.5 \cdot 10^{-5}$			
conc. range	$10^{-2}$ M				
Diffusion exp. Iodide		$4.9 \cdot 10^{-5}$	$7.6 \cdot 10^{-5}$	$7.6 \cdot 10^{-5}$	$<0.03 \cdot 10^{-5}$
conc. range	1 M	$5.1 \cdot 10^{-5}$	$8.7 \cdot 10^{-5}$	$3.5 \cdot 10^{-5}$	$<0.03 \cdot 10^{-5}$
Resistivity measurements		$8.3-13 \cdot 10^{-5}$	$6.7-10 \cdot 10^{-5}$	$14-25 \cdot 10^{-5}$	$2.2-4.3 \cdot 10^{-5}$
conc. range	1 M				

\* Synthetic ground water is used in the experiment.

Table VI: Values of the formation factor for different rocks and determined with different methods.

In Table VI the values of the formation factor from all the experiments are summarized. For Finnsjö granite the diffusion experiment with tritiated water, the diffusion experiment with iodide and the electrical resistivity measurements give results that are in fair agreement. The diffusion experiments with Cr-EDTA and Uranine give values that are lower. These values are, however, rather uncertain because the method that is used to determine the diffusivities in free water for the components might give rather large errors, mostly depending on the difficulty in estimating the solute molar volume (see Eq. (15)).

In the sorption experiments with cesium and strontium the factor is about an order of magnitude higher, or more for strontium, than in the iodide diffusion experiment. This is illustrated in another way in Fig. 18. The diagram shows the best fit of the pore diffusion model to the experimental uptake curve for strontium diffusion in Finnsjö granite.

It also shows a curve calculated with the same pore diffusion model but with a pore diffusivity given by the formation factor  $\epsilon_p \cdot \delta_D / \tau^2 = 7 \cdot 10^{-5}$ , which is of the same magnitude as those for the diffusion experiments with tritiated water and iodide and the resistivity measurements. It is seen that in the experiment with sorption and diffusion of strontium in Finnsjö granite equilibrium is obtained about 20 times faster than would be expected. This indicates that there is another mechanism of migration in addition to pore diffusion. Similar observations have been made in studies of cesium and strontium migration in clays (13).

The iodide diffusion experiment and the resistivity measurements in Stripa granite give values of the formation factor that are in good agreement. The factor determined in the experiments with cesium and strontium is markedly higher also for Stripa granite.

In the experiments with gneiss there are differences. Gneiss is a rather inhomogeneous material and there are probably variances in the material properties between different pieces of gneiss. This could be an explanation to the different results between the iodide diffusion experiment and the resistivity measurements and also to the differences in the results from the same type of experiment.

In the experiments with gabbro the results do not agree at all. It is probably the iodide diffusion experiment that gives a much too low value. The resistivity measurements and the porosity determination indicate that there are pores in the material where diffusion could take part. Maybe the iodide reacts chemically in some way with the rock material.

Experiments where the diffusion in low porous materials is determined are often very time consuming. The results from the experiments here indicate that electrical resistivity measurements, which are a faster method, can give approximate values of the diffusivity. It must be noticed, however, that this is only valid when the transport mechanism consists of only pore diffusion. When surface conductivity is large the method may overestimate the effective diffusivity.

## NOTATION

A	diffusion area of sample (cross-section)	$m^2$
c	concentration in bulk fluid	mol/l, cpm/l, mg/l
$c_o$	initial concentration in bulk fluid	mg/l
$c_p$	concentration in pore fluid	mg/l
$c_+, c_-$	cation and anion concentrations	equiv/cm <sup>3</sup>
D	diffusion coefficient	$m^2/s$
$D_e$	effective diffusion coefficient	$m^2/s$
$D_{AA}$	self diffusion coefficient for comp. A	$m^2/s$
$D_p$	pore diffusivity	$m^2/s$
$D_v$	diffusivity in water	$m^2/s$
F	Faraday's constant = $9.6487 \cdot 10^4$	As/mol
$G_+, G_-$	cation and anion concentration gradients	equiv/cm
k	Boltzmann constant = $1.38 \cdot 10^{-23}$	J/ <sup>o</sup> K
$k_f$	parameter in Freundlich isotherm	
l	thickness or length of a piece	mm
N	rate of transfer	mol/s, cpm/s, mg/s
$N_+$	diffusion flux density of cation	equiv/cm <sup>2</sup> , s
$\tilde{N}$	Avogadro's number = $6.02 \cdot 10^{23}$	(mol) <sup>-1</sup>
n	number of granite pieces	
$n_+, n_-$	valence of cation and anion	
q	concentration in the solid material	mg/kg
R	gas constant = 8.3143	J/mol, <sup>o</sup> K
$R_o$	electrical resistivity in salt water	$\Omega m$
$R_s$	electrical resistivity in salt water saturated rock sample	$\Omega m$
T	temperature	<sup>o</sup> K
t	time	seconds, hours
V	bulk fluid volume	$m^3$
$\tilde{V}_A$	molar volume of component A	cm <sup>3</sup> /mol

$x$	length coordinate	
$\beta$	exponent in Freundlich isotherm	
$\delta_D$	constrictivity for diffusion	
$\epsilon_p$	porosity of the materials	
$\lambda_+^0, \lambda_-^0$	cationic and anionic conductance at infinite dilution	$\text{cm}^2/\Omega, \text{equiv}$
$\mu_A$	dynamic viscosity for component A	$\text{Ns/m}^2$
$\rho_s$	density of the solid material	$\text{kg/m}^3$
$\tau$	tortuosity	
$\epsilon_p \cdot \delta_D / \tau^2$	formation factor	

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Composition of the synthetic groundwater according to B. Allard (14)

$ \text{HCO}_3^- $	$2.014 \cdot 10^{-3}$ M,	123 ppm
$ \text{H}_4\text{SiO}_4 $	$2.056 \cdot 10^{-4}$ M,	12 ppm
$ \text{SO}_4^{2-} $	$1.000 \cdot 10^{-4}$ M,	9.6 ppm
$ \text{Cl}^- $	$1.973 \cdot 10^{-3}$ M,	70 ppm
$ \text{Ca}^{2+} $	$4.477 \cdot 10^{-4}$ M,	18 ppm
$ \text{Mg}^{2+} $	$1.774 \cdot 10^{-4}$ M,	4.3 ppm
$ \text{K}^+ $	$1.000 \cdot 10^{-4}$ M,	3.9 ppm
$ \text{Na}^+ $	$2.836 \cdot 10^{-3}$ M,	65 ppm
pH	8.0 - 8.2	

Ionic strength: 0.0085



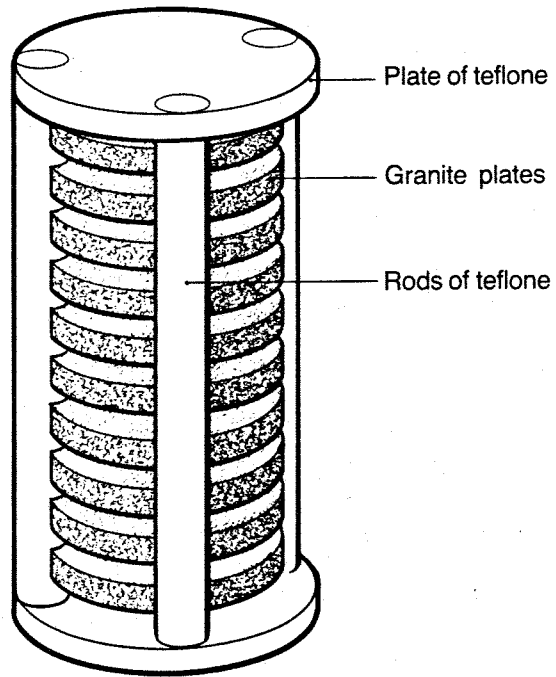


Fig 1: A rack of teflon with granite pieces

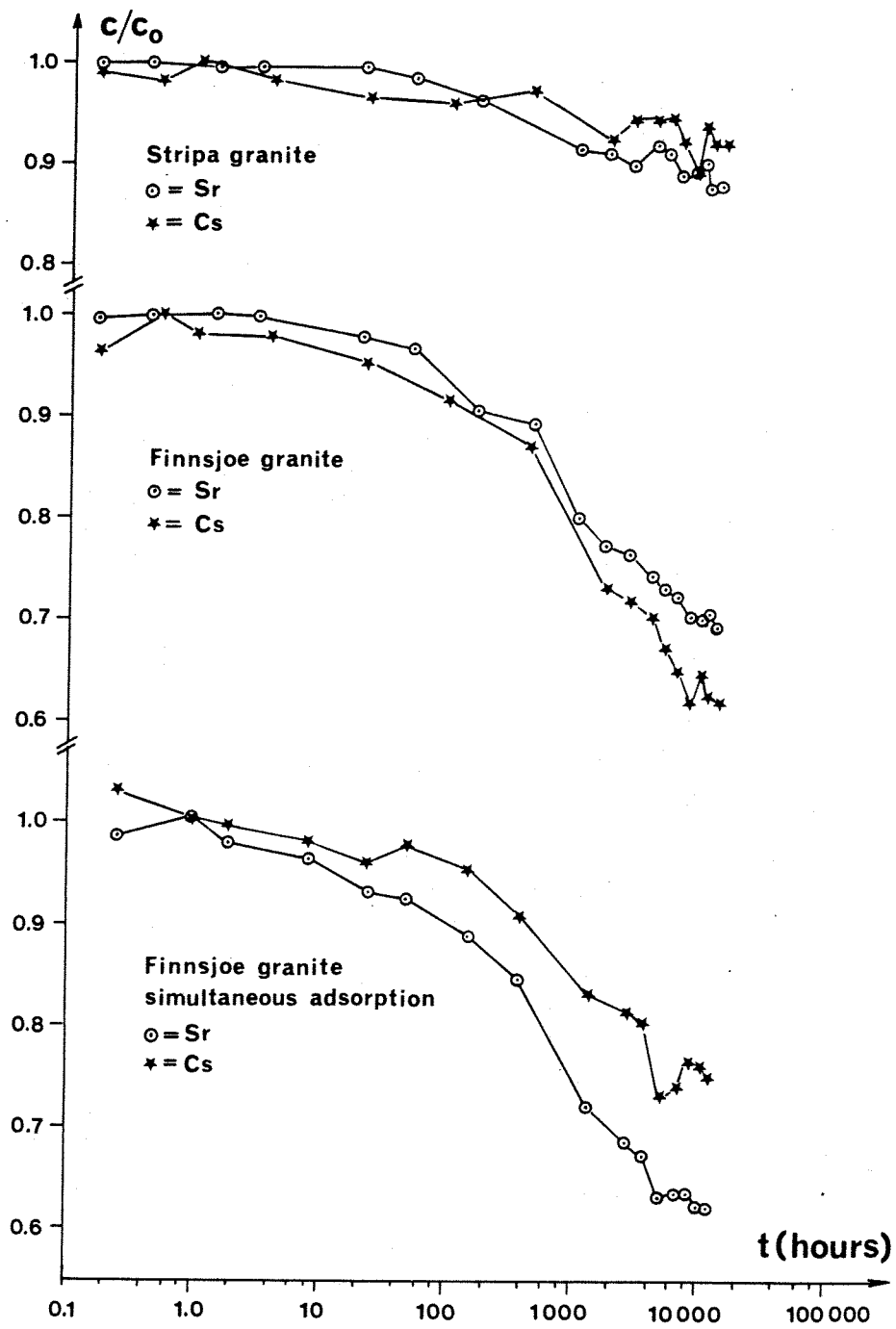


Fig 2: Adsorption of cesium and strontium on Finnsjoe and Stripa granite

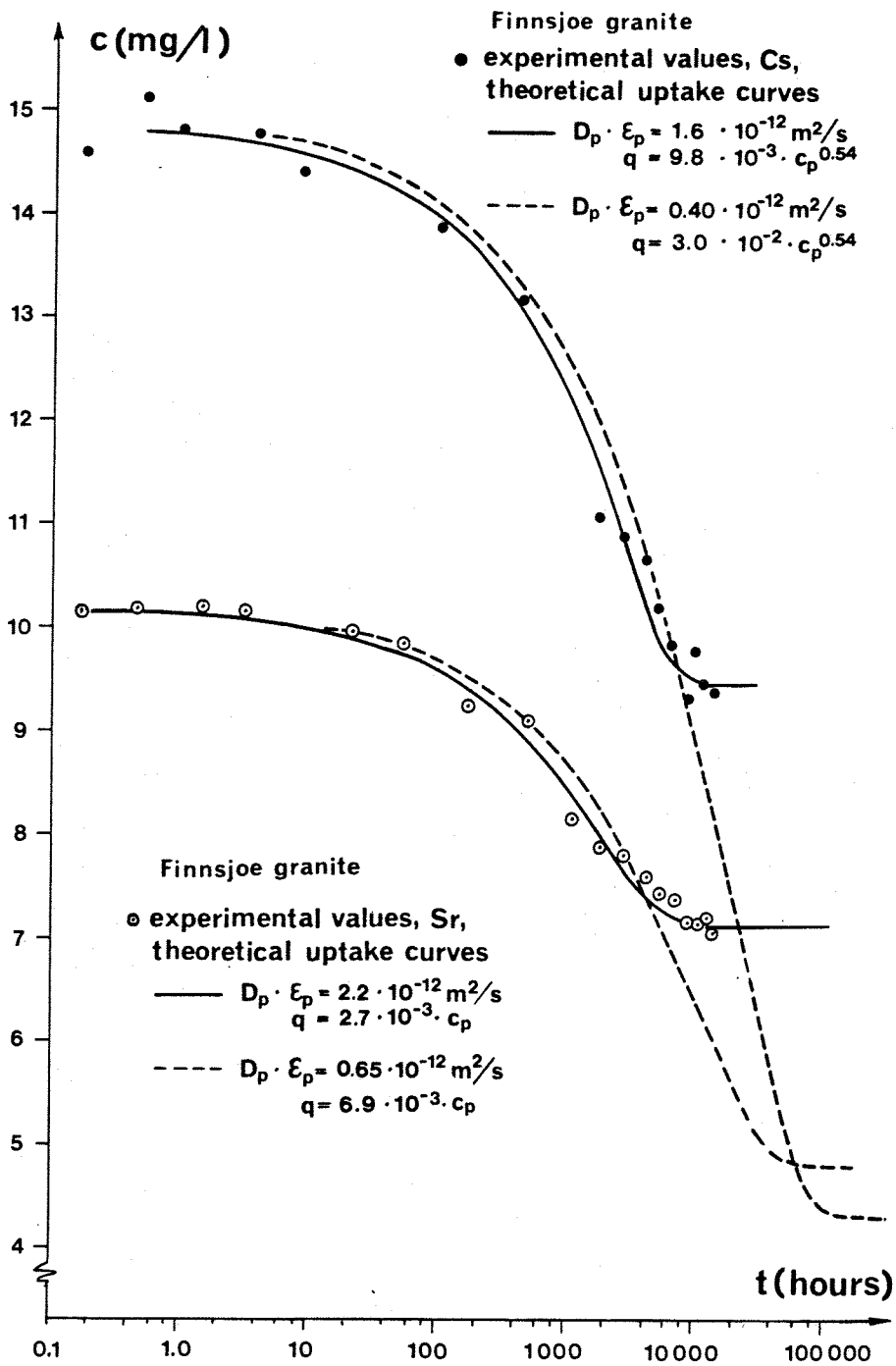


Fig 3: The fit of the diffusion model to the experimental uptake curve for cesium and strontium in Finnsjoe granite

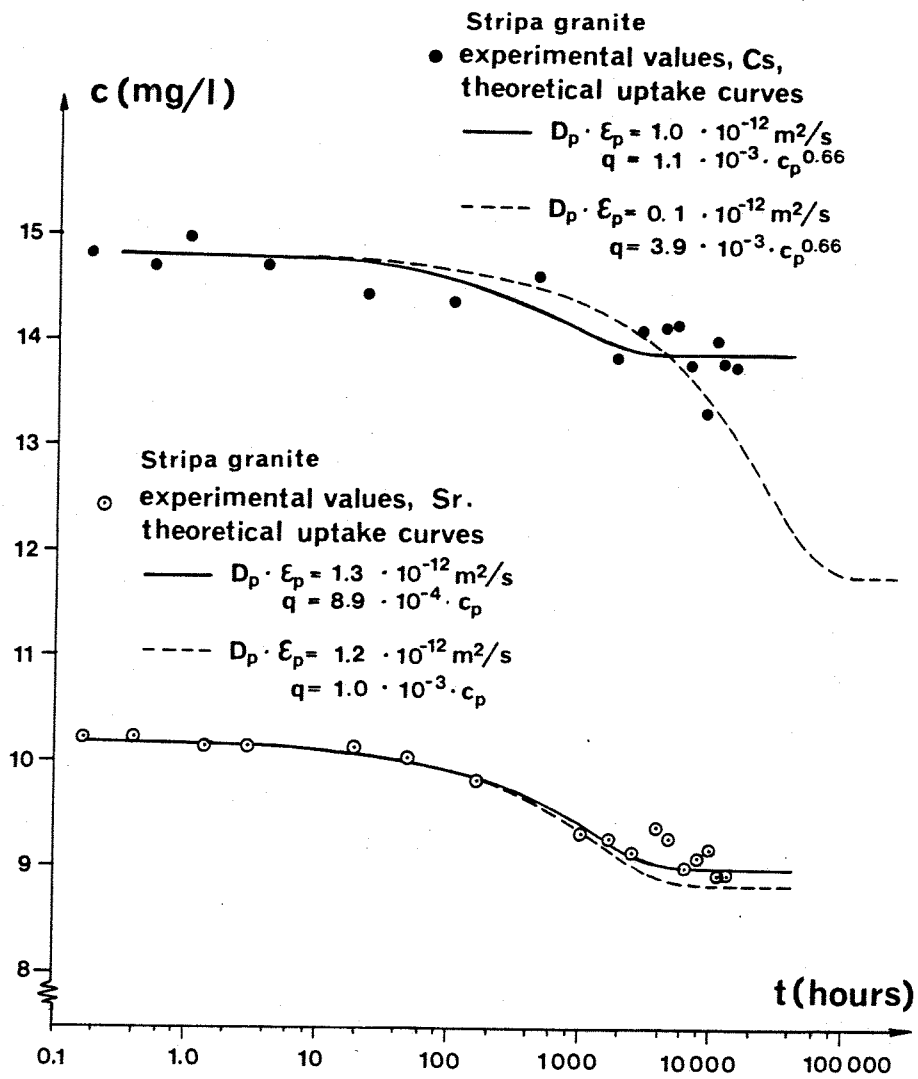


Fig 4: The fit of the diffusion model to the experimental uptake curve for cesium and strontium in Stripa granite

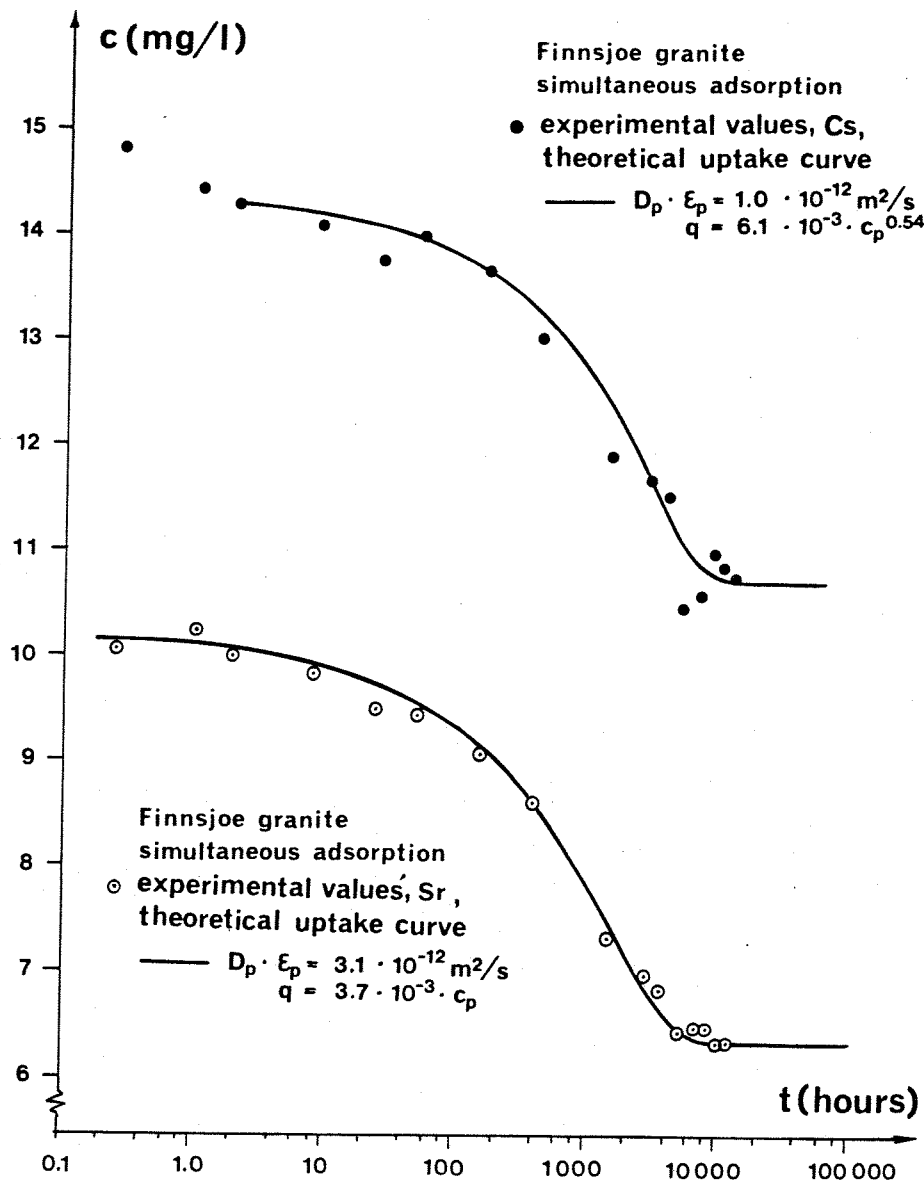


Fig 5: The fit of the diffusion model to the experimental uptake curve for simultaneous sorption of cesium and strontium in Finnsjoe granite

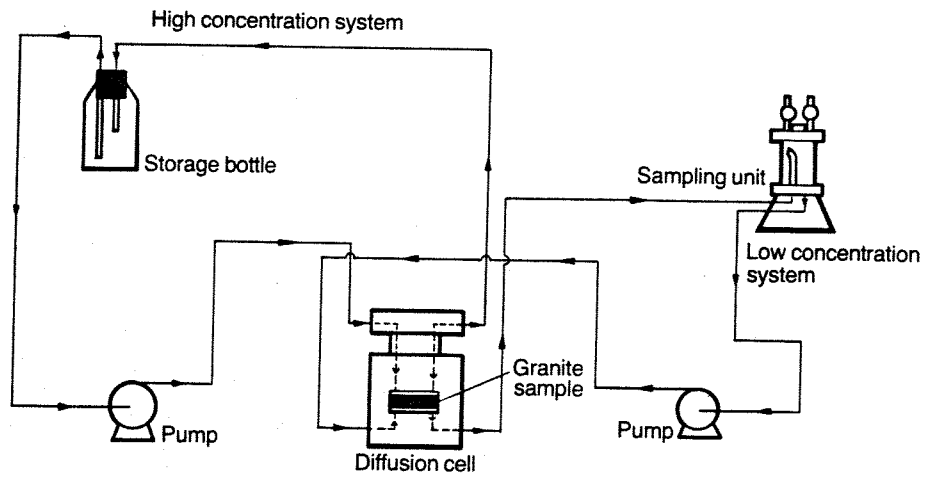


Fig 6: The apparatus in the diffusion experiment with tritiated water

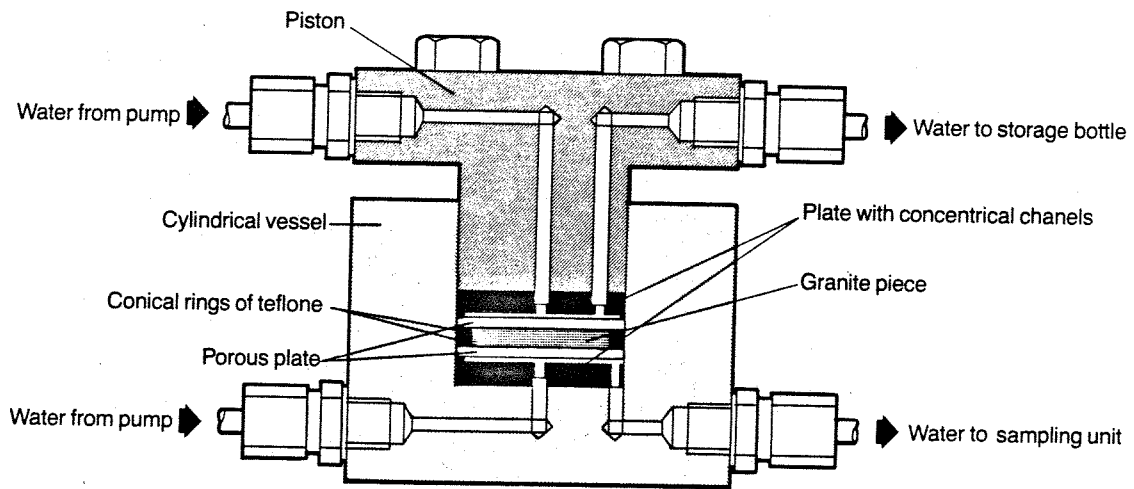


Fig 7: The diffusion cell

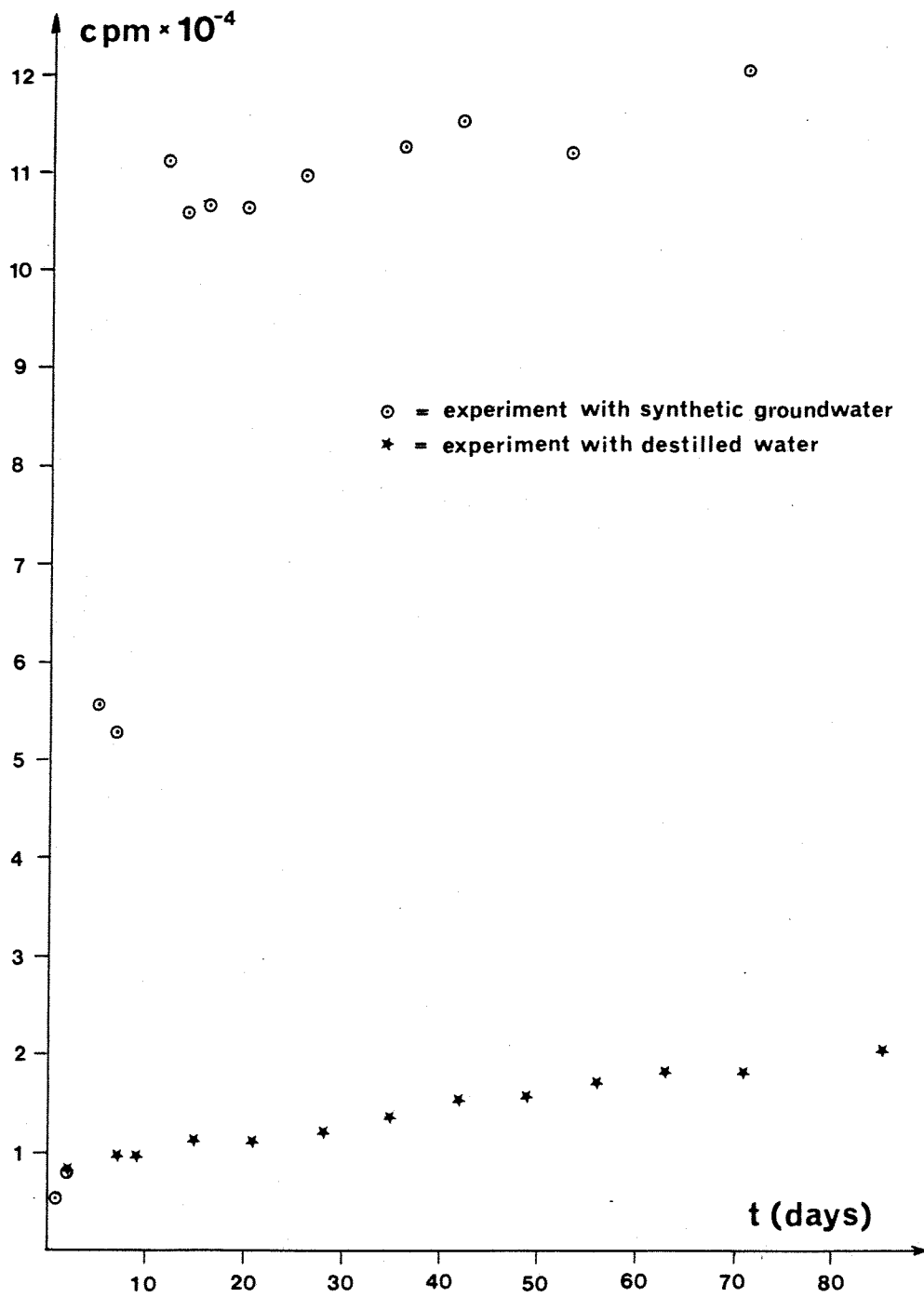


Fig 8: The amount of tritiated water in the low concentration system at different times

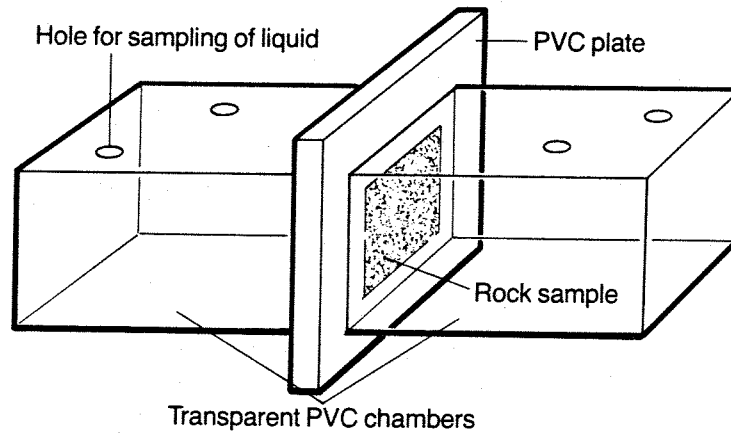


Fig 9: The diffusion cell used in the experiments with iodide, Cr-EDTA and Uranine



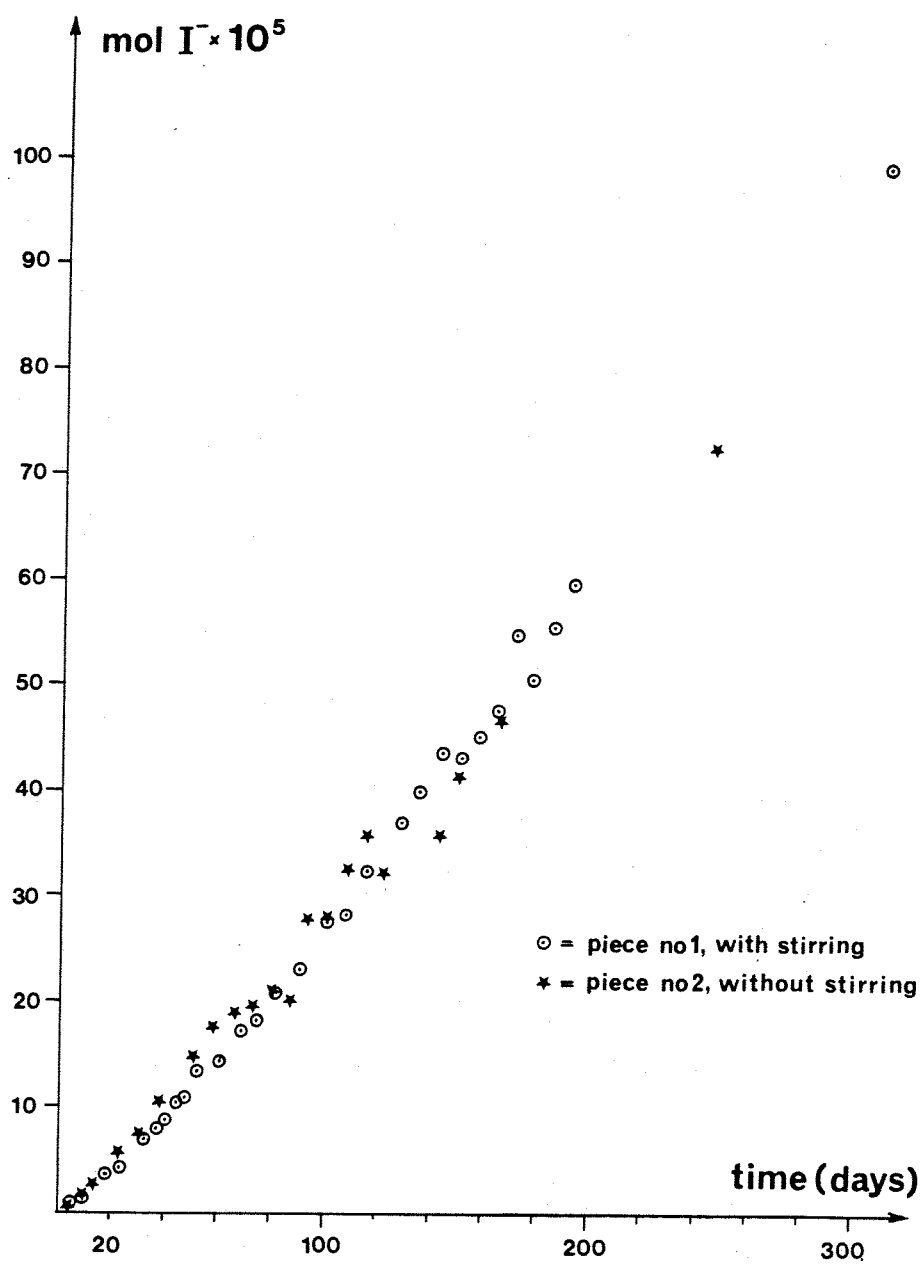


Fig 10: The amount of iodide which had diffused through pieces of Stripa granite at different times

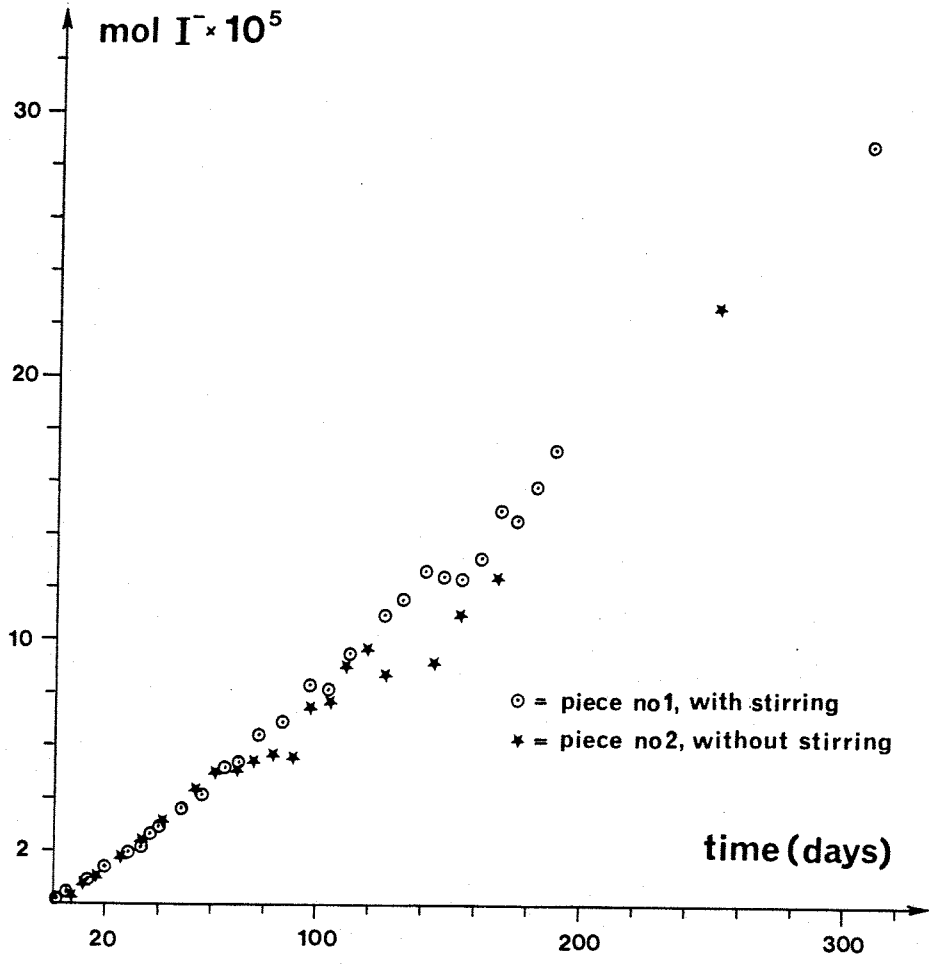


Fig 11: The amount of iodide which had diffused through pieces of Finnsjoe granite at different times

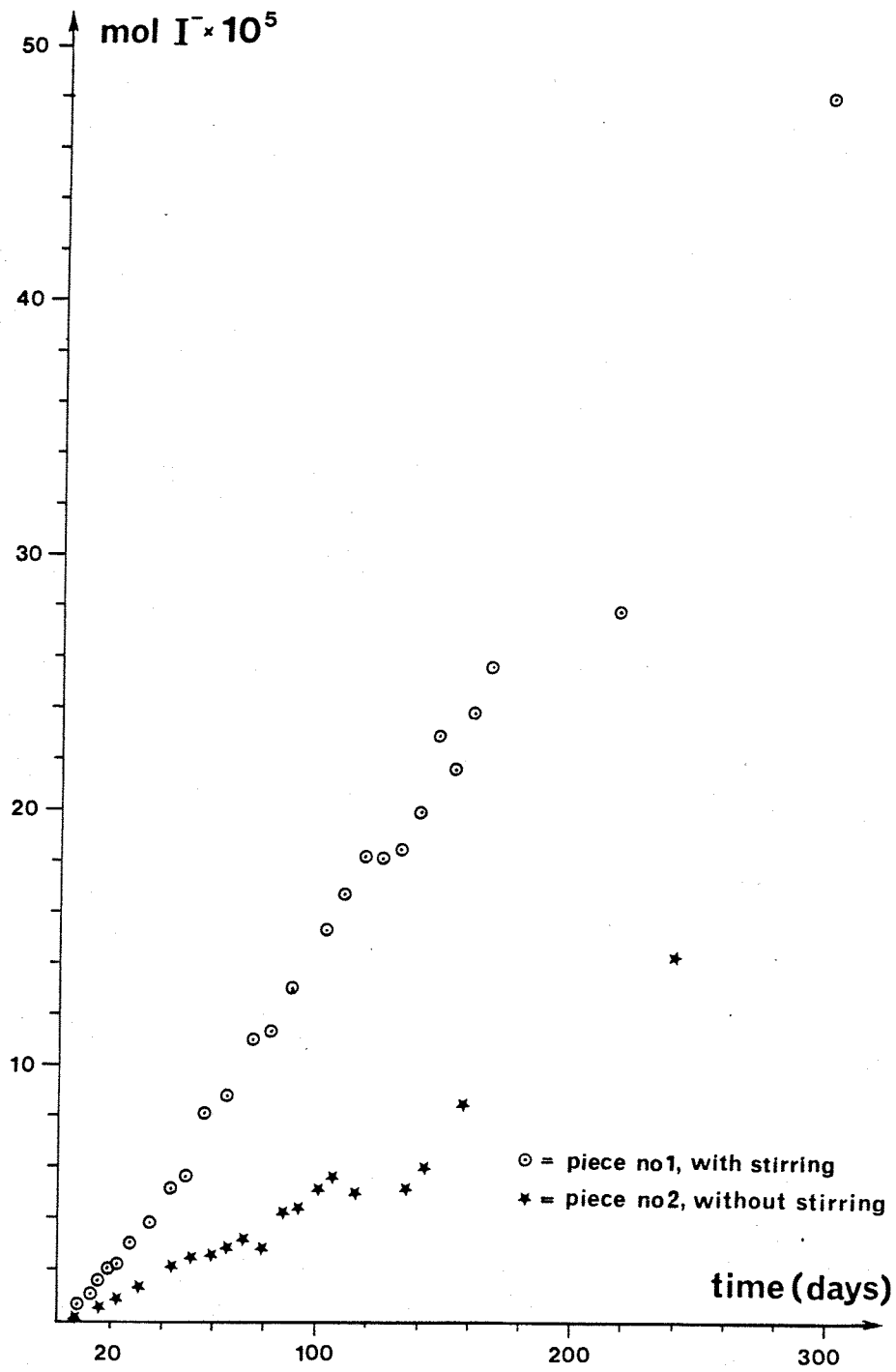


Fig 12: The amount of iodide which had diffused through pieces of gneiss at different times

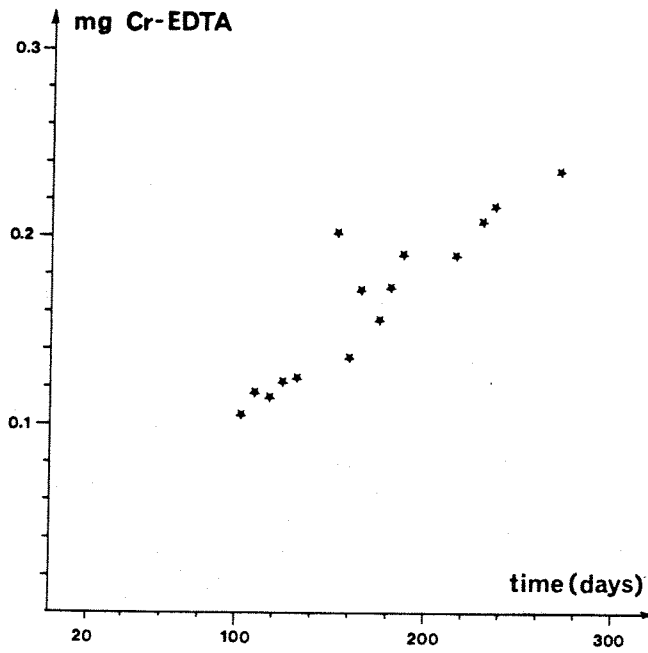


Fig 13: The amount of Cr-EDTA which had diffused through a piece of Finnsjoe granite at different times

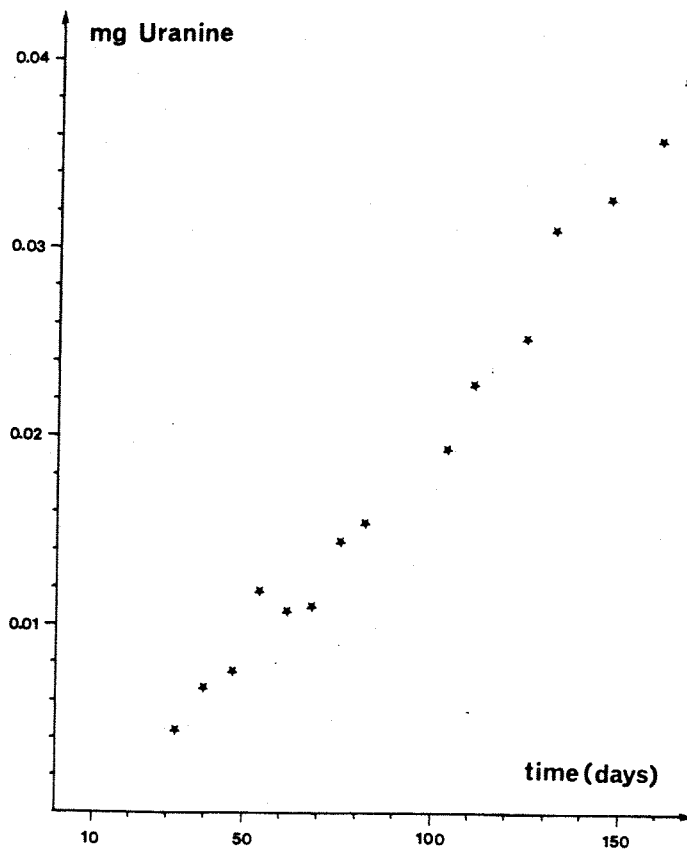


Fig 14: The amount of Uranine which had diffused through a piece of Finnsjoe granite at different times

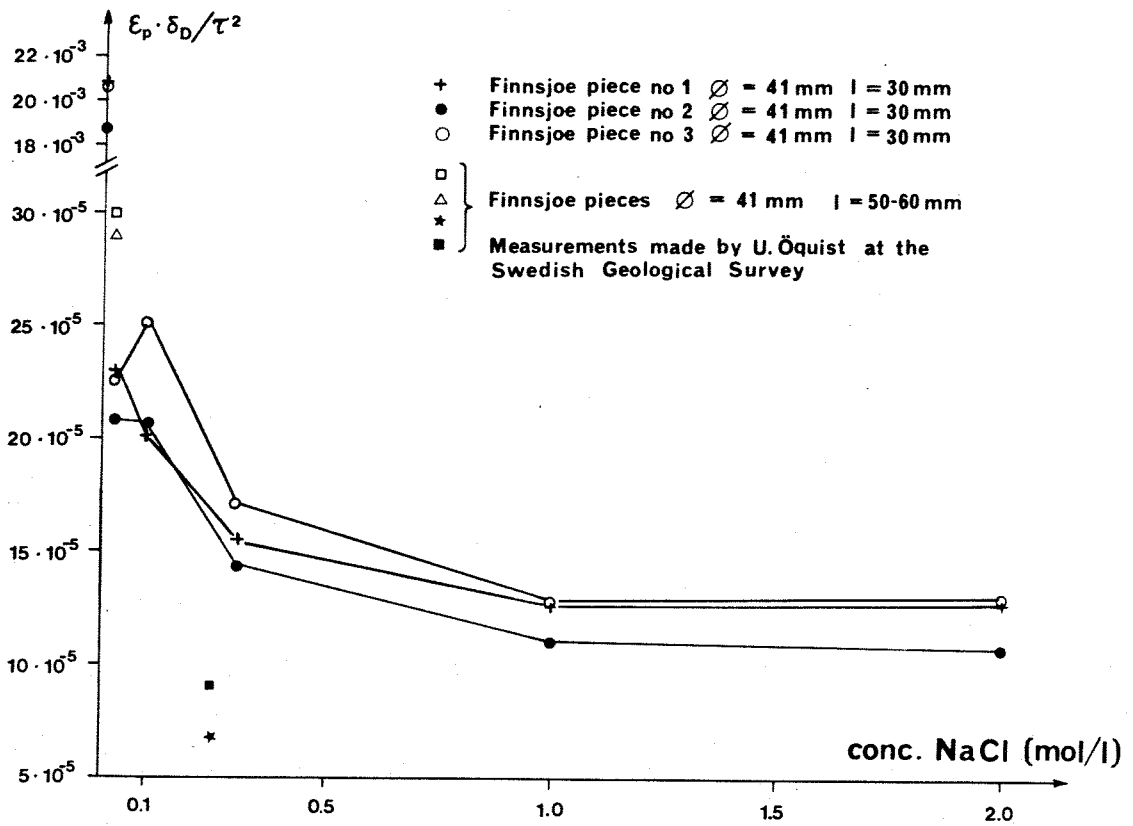


Fig 15: The formation factor versus the concentration of NaCl in the salt solution

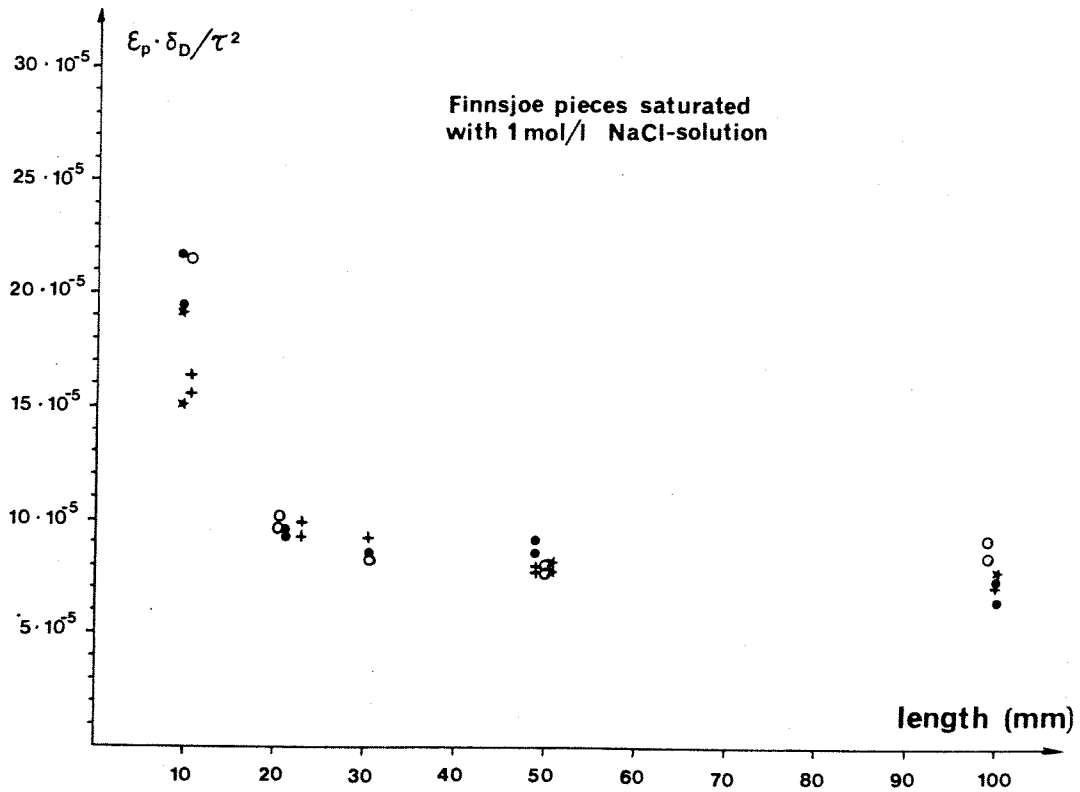


Fig 16: The formation factor versus the length of the rock cores

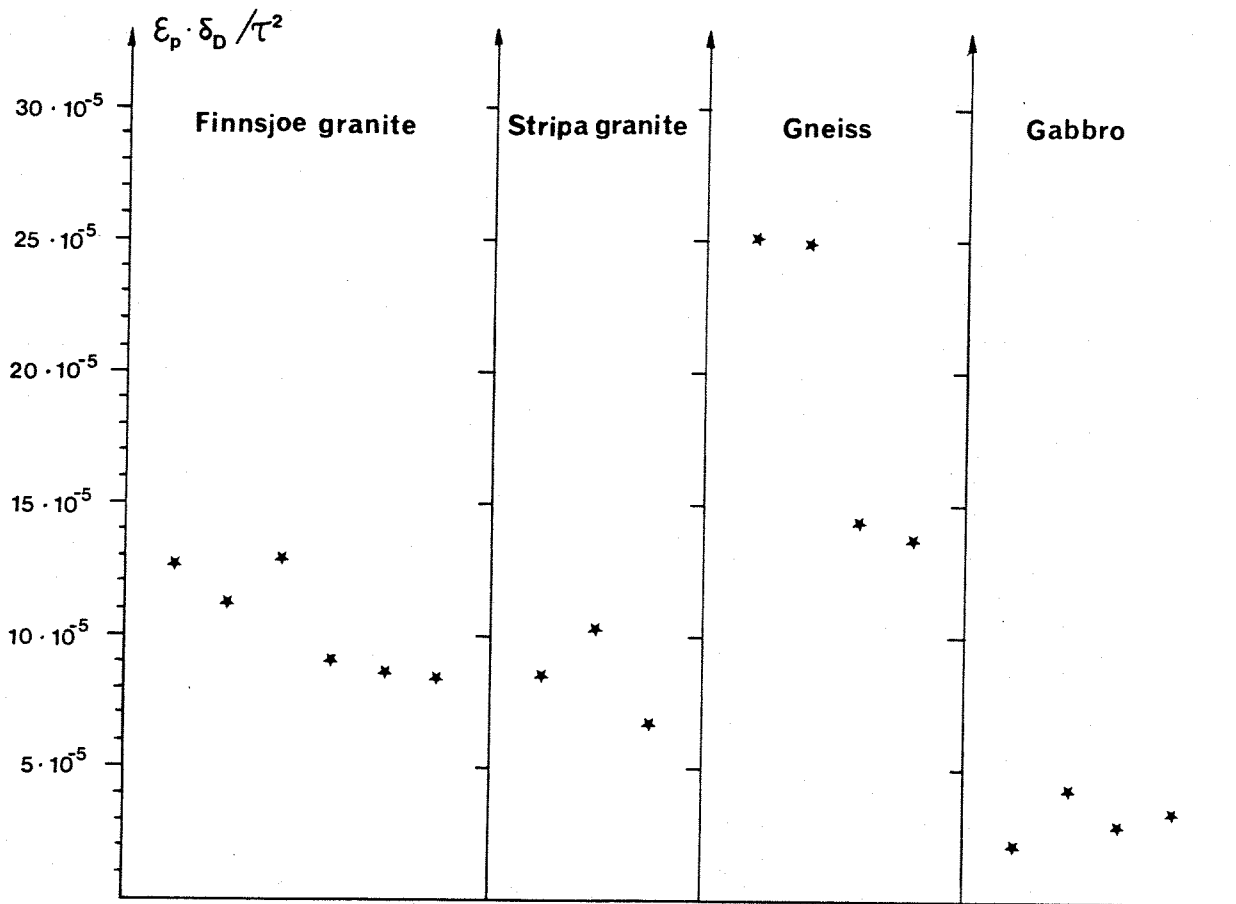


Fig 17: The formation factor for different rock materials determined by electrical resistivity measurements

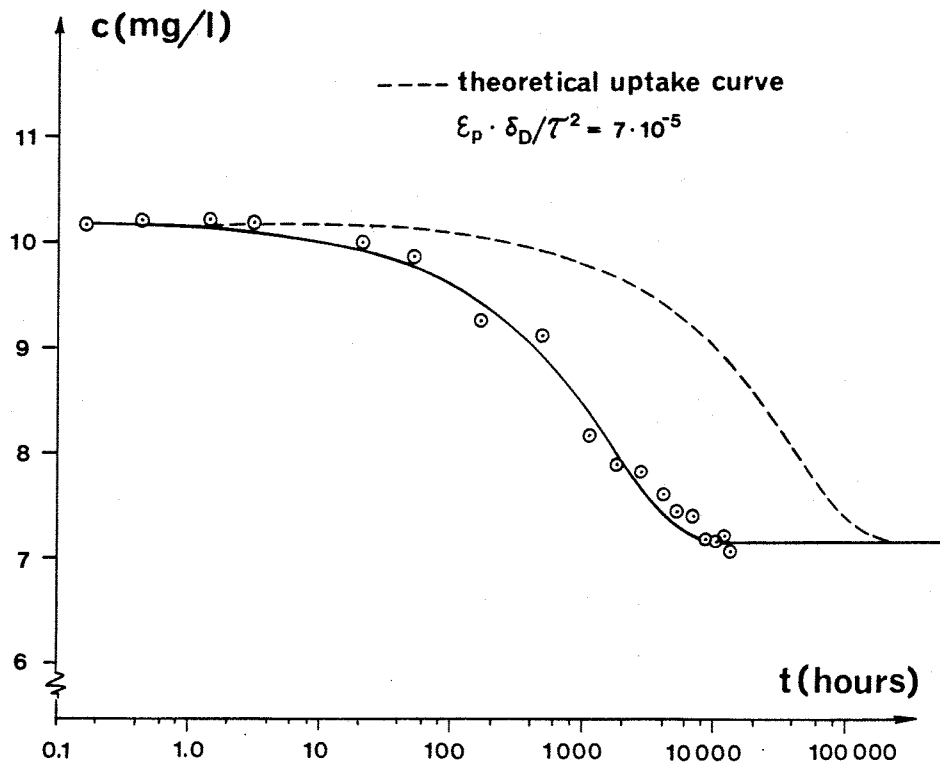


Fig 18: A comparison between the best fit of the pore diffusion model to the experimental data for strontium diffusion in Finnsjoe granite, and the pore diffusion model based on a value of the formation factor for Finnsjoe granite given by the iodide diffusion experiment and the resistivity measurements

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