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A laboratory flow system for transport under reducing conditions

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ABSTRACT.

A flow system for laboratory studies of radionuclide transport in natural fissures in granitic rock under reducing conditions is described. The system based on the use of synthetic ground water equilibrated with granitic rock in a well sealed system, allow experiments to be carried out at $-240\,\mathrm{mV}$. In flow experiments with technetium the retardation was found to be dependent on the method used for reducing $\mathrm{TcO_4}^-$.

1. INTRODUCTION.

The study of radionuclide migration through rock is currently of great interest due to its relevance to the possible escape paths into the biosphere of radionuclides released from high level radioactive wastes burried in deep geological repositories.

While water will provide the vehicle for transportation, interaction with geological material may greatly influence the radionuclide movement relative that of water. The identity and thereby the geochemical reactivity of the compounds formed by the radionuclides will depend on the oxidation state of the radionuclides, the redox properties of the ground water and surrounding rock, the pH and concentration of complexing agents in the water. In our earlier studies of radionuclide migration in granitic drill-cores containing natural fissures (Neretnieks, Eriksen and Tähtinen 1982), (Eriksen 1985), (Moreno, Neretnieks and Eriksen, 1985) all but one of the experiments were carried out under oxic conditions. The conditions in a repository will, however, as indicated by the measured redox potentials of deep ground waters be reducing (Wikberg, 1987). The present work describes a laboratory flow system which allows experiments to be carried out under reducing conditions and some experiments carried out with $^{99}\mathrm{Tc}$ and 99mTc.

The long lived fission product ^{99}Tc ($t_{1/2} = 2.1 \cdot 10^5$ years) is present in large quantities in nuclear waste. Under oxidizing

condition technetium is existing in solution as TcO_4^- but is expected to exist as Tc(IV) in a reducing environment. Whereas no or very little sorption of TcO_4^- occurs on geologic material Tc(IV)-species may possibly be sorbed. Experiments were therefore carried out with reduced $^{99m}Tc(VII)$ to obtain information on the sorption of Tc(IV) on granite rock.

EXPERIMENTAL.

2.1 Flow system.

Reducing water can be obtained in laboratory, as demonstrated by Wikberg (1987) by contacting deoxygenated water with granitic rock in a well sealed vessel. Our experimental set up (Fig. 1) is therefore based on the use of granitic rock to secure low Eh of the synthetic water used in our flow experiments. The synthetic ground water, equilibrated with crushed granitic rock in a water reservoir, is fed through granitic drill-cores containing natural fissures by means an encapsulated HPLC-pump. All connecting tubes are of stainless steel quality and the drill-cores are encapsulated in a plexiglas cylinder continuously flushed with argon to avoid any oxygen leakage into the system.

Prior to any tracer experiment water from the reservoir was circulated through the entire flow system for several days to equilibrate the fissure surfaces. The redox potential was continuously monitored with Pt or Ag/AgCl electrodes during the equilibration. The tracer experiments were initiated by connecting an injection loop initially containing

3.40 cm³ tracer solution to the flow system. During the tracer experiments the effluent from the drill-core was fed continuously to a fractonal collector for analysis. The waterflow was characterized by a nonsorbing tracer.

2.2 Rock-material.

The rock sample used in our experiments is granitic rock from the Stripa mine taken at a depth of 360 m below ground level. The drill-core (37 mm diameter, 126 mm long) used in the present experiments had a natural fissure running parallel to the axis. The cylindrical surfaces of the drill-core were sealed with a coat of polyurethane lacquer, and fitted with plexiglas endplates containing shallow inlet and outlet channels slightly wider the the fissures.

2.3 Technetium solutions.

Determination of the Tc(IV) concentration in the effluent by the use of ^{99}Tc as tracer would be very diffic.lt due to the low solubility of Tc(IV), $2.6\cdot10^{-9}$ mol·dm⁻³ according to Meyer et al (1987, 1988), and the long half life of ^{99}Tc (see Table 2). The experiments were therefore carried out using the short lived γ -emitter $^{99}\text{m}\text{Tc}$ as tracer and ^{99}Tc as "carrier" to regulate the overall initial Tc-concentration.

Table 1

Characteristics of tracers used.

Tracer	Half-life	Mode of decay	Measured
Lignosulfonate			Optical abs. at
ion (LS ⁻)			280 nm.
mol wt 24 000			ϵ 3.10 $M^{-1}cm^{-1}$
99 _{TC}	2.1·10 ⁵ y	β	
99m _{TC}	6 h	IT	γ (0.14 MeV)

The radionuclides used were ^{99}Tc delivered as TcO_4^- in 0.1 mol·dm^{-3} aqueous NH_4OH solution from Amersham and ^{99}mTc recovered as TcO_4^- in 0.4 mol·dm^{-3} aqueous NaCl solution from a ^{99}Mo generator (Kjeller). Most radionuclide solutions were prepared by diluting stock solutions with artifical ground water, synthesized according to the composition given in Table 2.

Table 2

Composition of synthetic ground water a) used in the flow experiments.

Species	Concentration mg·dm ⁻³	Species	Concentration mg·dm ⁻³	
нсо ₃ -	123 ,	Ca2+	18	
so ₄ ²⁻	9.5	Mg ²⁺	4.3	
c1 ⁻	70	K ⁺	3.9	
SiO ₂ (total)	12	Na ⁺	65	
3)				

a) ph = 8.2, Eh -240 mV

Two different procedures were used to reduce the ${\rm TcO_4}^-$

1) The reduction was carried out by means of $H_2(Pd)$. The hydrogen gas was freed from oxygen by bubbling the gas through an aqueous solution containing Hg/Zn, HCl and $CrCl_3$. Solutions containing two different total Tc concentrations were used in the experiments.

a)
$$3 \cdot 10^{-12} \text{ mol} \cdot \text{dm}^{-3} 99 \text{m}_{\text{TC}}$$
,

b)
$$3 \cdot 10^{-12} \text{ mol} \cdot \text{dm}^{-3} 99 \text{m}_{TC}, 1 \cdot 10^{-7} \text{ mol} \cdot \text{dm}^{-3} 99 \text{Tc}$$

The reduction was carried out using a redox-buffer with measured reduction potential -520~mV versus a saturated colonnel electrode. The composition is given in Table 3. The initial Tc concentrations, i.e before reduction, were $1\cdot10^{-11}~\text{mol}\cdot\text{dm}^{-3}~99\text{m}_{\text{Tc}}$ and $1\cdot10^{-8}~\text{mol}\cdot\text{dm}^{-3}~99\text{Tc}$.

Species	Concentration mg·dm ⁻³	Species	Concentration mg·dm ⁻³
NaHCO ₃	1 680	CaCO ₃	10 mg (tot)
Na ₂ CO ₃	76	FeSO ₄	88
KCl	10		

a) pH = 8.2, Eh -520 mV versus saturated colonnel electrode

2.4 Tracer measurements.

Sodium lignosulfonate (NaLS) with a mean molecular weight of 24000 was used as nonsorbing tracer. The ${\rm LS}^-$ ion displays a

strong optical absorption band with maximum at 280 nm (ε \cdot 3·10⁵ M⁻¹ cm⁻¹) and the LS⁻ concentration was monitored photometrically at this wavelength. The concentration of ^{99m}Tc in the effluent was determined from measurements of its γ -radiation using a (2-x2 in.-) NaI well type detector. The ^{99m}Tc-activity on the fissure surfaces was measured with a planar (2-x2 in.) NaI detector fitted with a 7x37 mm lead collimator.

3. RESULTS.

3.1 Lignosulfonate.

The lignosulfonate (LS⁻) concentration-time curve is plotted in Figure 3a. The tracer solution loop volume was $3.40~{\rm cm}^3$ and the volume of the tube between the tracer loop and the sampling point of the fractional collector was $0.36~{\rm cm}^3$ (see Fig. 2).

The fissure volume can be calculated from the ${\rm LS}^-$ data using the equation

$$v_f = q_w \cdot t_w \tag{1}$$

where v_f = fissure volume (cm³)

 $q_w = \text{water flux } (\text{cm}^3 \cdot \text{min}^{-1})$

 $t_{\mathbf{w}}$ = residence time of LS⁻ in fissure (min)

The volumes of the flow system are given in Figure 2, the water flux was $0.42~{\rm cm}^3 \cdot {\rm min}^{-1}$ and the measured residence time

(Fig 3a) is $16.5\ \text{min}$. Thus, taking into account the time delay caused by the tube volume we obtain

$$V_f = 0.42(16.5 - 3.4/2.0.42 - 0.360/0.42) = 4.87 \text{ cm}^3$$

Based on the simple assumption of parallel, plane fissure surfaces the fissure width and the surface area/volume ratio is given by the equation

$$\delta = V_f/d\cdot 1 \tag{2}$$

$$a = 2 \cdot d \cdot 1/V_f = 2/\delta$$
 (3)

where a = surface area/volume ratio (cm)

d = drill core diameter (cm)

l = drill core length (cm)

 δ = fissure width (cm).

The flow parameters calculated from the LS^- break through curve are given in Table 4.

Table 4

Drill-core parameters calculated from LS flow data.

Fissure	Fissure	Fissure	A _f /V _f
volume (V_f) cm ³	surface (A _f)	width ($^{\delta}$)	cm^{-1}
4.87	93.2	0.105	19.1

3.2 Technetium.

The measured potential of the ground water was in the range -(485-495) mV versus the saturated calomel electrode during all experiments. For solution (a) with very low overall Tc-concentration no change in 99 mTc activity on reduction with $_{12}$ H2/(Pd) was observed and as can be seen from the break-through curves in Figure 3b no delay in the 99 mTc migration relative to LS-was observed. On adding $_{10}$ - $_{7}$ -mol·dm- $_{3}$ - $_{99}$ -Tc as carrier a brownish deposit was formed on the electrode and the $_{99}$ mTc activity in the solution was lowered by about 50 per cent on reduction. No delay in the $_{99}$ mTc transport through the fissure was, however, observed (Fig. 3c).

In earlier experiments with a redox-buffer (method 2) a significant retardation (R=40) in the 99m Tc migration through a natural granitic fissure was observed (Eriksen, 1985).

In the present experiment, the tracer solution was prepared using the same redox-system but artificial ground water was used to eluate the $^{99\text{m}}\text{Tc}/^{99}\text{Tc}$. During the reduction more than 95 per cent of the $^{99\text{m}}\text{Tc}/^{99}\text{Tc}$ was precipitated. No $^{99\text{m}}\text{Tc}$ was detected in the effluent during 50 minutes eluation.

The fissure was thereafter opened and the $^{99\text{m}}\text{Tc}$ activity on the fissure surfaces measured. From the activity data plotted in Figure 4 and the LS- data we can calculate the $^{99\text{m}}\text{Tc}$ retardation using the equation

$$R = (V_w/V_f) \cdot (1/X_{rm})$$

where $V_{\mathbf{w}}$ = volume of water and tracer solution pumped through fissure (cm³)

 V_f = volume of fissure (cm³)

1 = length of fissure (cm)

X_{rm} = mean distance travelled by the radionuclide (cm)

The experimental Tc-data are summarized in Table 5, together with results obtained in earlier experiments (Eriksen 1985).

Table 5

Experimental transport parameters calculated from effluent $99m_{\mbox{Tc-activity-time}}$ curves and activity distribution on fissure surfaces.

Radionuclide sol. Method of red.	$99m_{Tc}$ a) mol·dm ⁻³		_	Retardation R
H ₂ (Pd)	3·10 ⁻¹² 3·10 ⁻¹²	10-7		1
Redox-buffer (see Table 2)	3.10-11	10-8	19.1	14
TcO ₄ -b)		3.10-6	52.4	1
Redox-buffer b) (-280 mV)			41.6	40

a) initial conc., before reduction

4. DISCUSSION.

All Tc-experiments were carried out using reducing synthetic ground water with Eh $_{\odot}$ -240 mV. The great differences in retardation obtained in the experiments are therefore somewhat

b) Eriksen 1985

astonishing, but must be due to differences in the reduction procedures used. According to Meyer et al (1987, 1988) the reduction potential E^{O} for the Tc(VII)-Tc(IV) redox couple is 0.747 V and the solubility of Tc(IV) species $2.56\cdot 10^{-9}$ mol·dm⁻³. Very little, if any TcO_4^- should therefore be present in the reducing ground water used in our experiments.

The reduction potential of the $H_2(Pd)$ system is given by $Eh(V) = -0.059 \cdot pH - 0.0295 \cdot log H_2$. At equilibrium the potential of the reducing solution should therefore be -484 mV, assuming $p(H_2) = 1$, ie much lower that the potential of the synthetic ground water. A possible explanation may therefore be that TcO_4^- was reduced to lower oxidation states eg Tc(III).

The retardation R calculated from the flow data obtained in experiments with ${\rm TcO_4}^-$ reduced in the redox-buffer system with Eh -280 mV agree, within a factor two with the retardation obtained in earlier experiments when the ${\rm A_f/V_f}$ ratio is taken into account.

It is evident from the experimental results that the reduction procedure is of great importance. The time required to reduce Tc(VII) also indicates very sluggish redox reactions. Electrodeposition of Tc(IV) oxides at a well defined potential and equilibration of the electrodeposited Tc(IV)-oxides with ground water may possibly be the preferred technique to obtain well defined tracer solutions. The dynamic of the TcO_4^- reduction is to be studied in further experiments.

5. CONCLUSION.

Flow experiments can be carried at Eh $\,-240\,\,\mathrm{mV}$ in synthetic ground water.

The preparation of the tracer solutions is crucial, as some of the redox-reactions may be very slow. The dynamics of the Tc(VII) reduction and also speciation need to be carried out in separate experiments.

ACKNOWLEDGEMENTS.

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FIGURE LEGENDS.

- Figure 1 Schematics of experimental set up for flow experiments through granitic drill-cores containing natural fissures.
- Figure 2 Details of flow system. Volumes of sample injection system.
- Figure 3 Break through curves obtained with 10 min tracer pulse, flow 0.43 cm³·min⁻¹, sampling time 1 min
 - a) LS⁻, b) 99 mTc Tot* Tc-3·10⁻¹² mol·dm⁻³
 - c) 99m_{Tc} , Tot $\text{Tc} \times 1.10^{-7} \text{ mol} \cdot \text{dm}^{-3}$
 - * concentration before reduction of TcO₄
- Figure 4 $99^{\rm m}{\rm Tc}$ distribution on fissure walls after 50 minutes eluation.

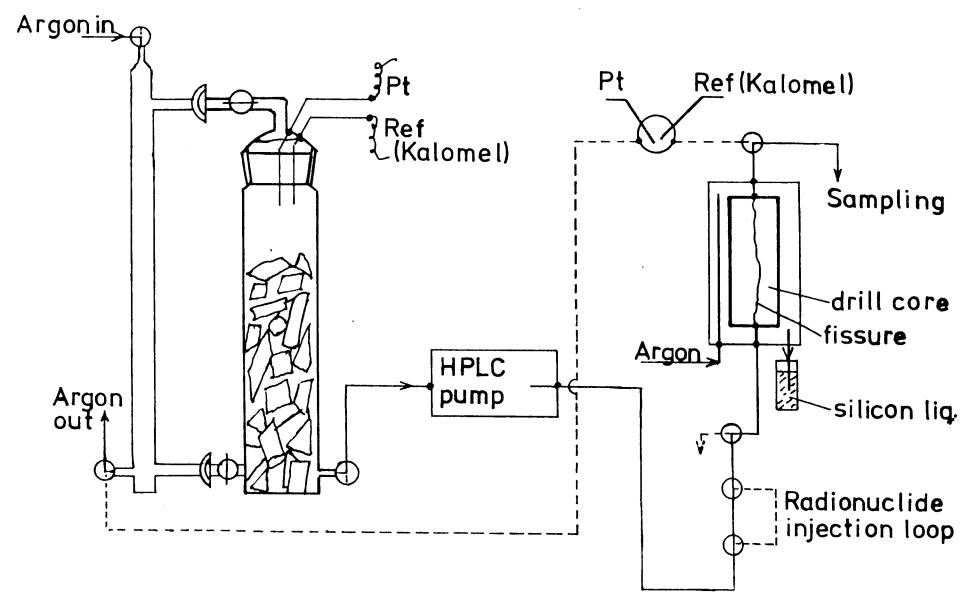
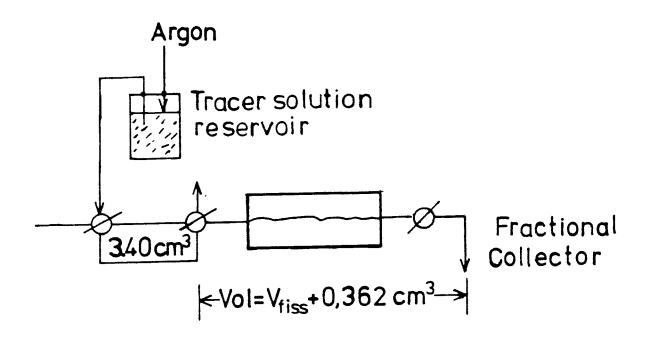


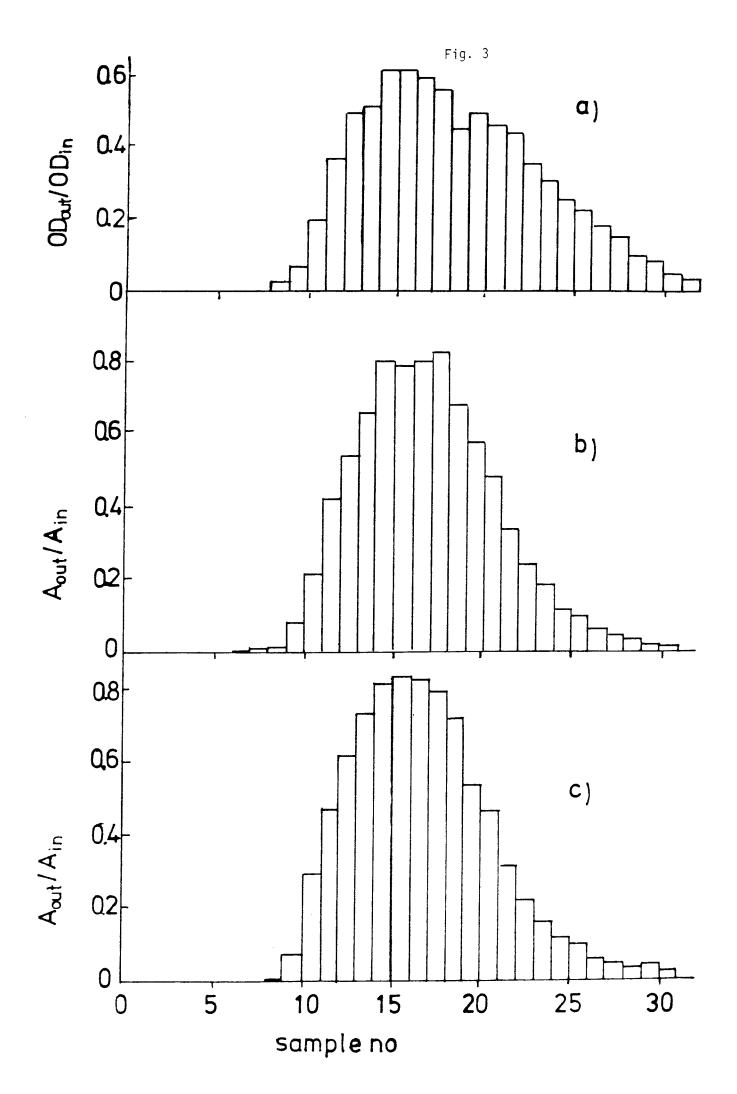
Fig. 1



Puls length: 3.4/q min q=water flux cm³min⁴

Delay:0.362q/min 0.362=tubevol.

Fig. 2



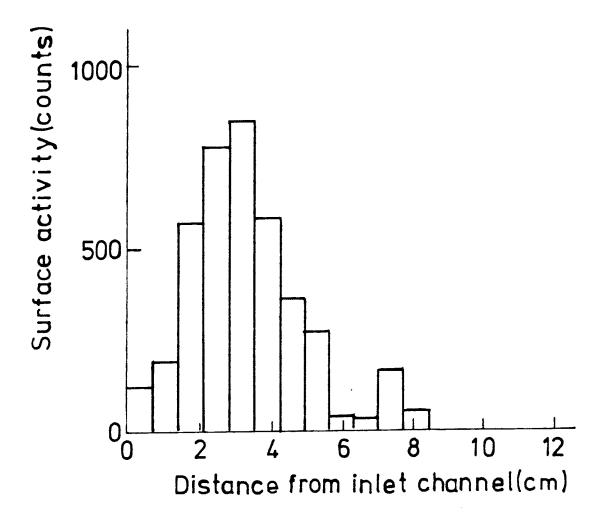


Fig. 4

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Karsten Pedersen University of Göteborg December 1987

TR 88-02

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Thomas Ittner¹, Börje Torstenfelt¹, Bert Allard²

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Luis Moreno¹, Yvonne Tsang², Chin Fu Tsang², Ivars Neretnieks¹
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Trygve E Eriksen, Pierre Ndalamba The Royal Institute of Technology, Stockholm Department of Nuclear Chemistry