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On the interaction of granite with Tc (IV) and Tc (VII) in aqueous solution

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IN AQUEOUS SOLUTION

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ON THE INTERACTION OF GRANITE WITH Tc(IV) AND Tc(VII)

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ABSTRACT

The behaviour of technetium in granite-groundwater systems under reducing conditions was investigated. The anion TcO_4^- was reduced to $Tc(IV)$ and simultaneously precipitated as $TcO_2 \cdot nH_2O$ on the granite surfaces. The electron sources are assumed to be iron oxides and/or iron containing minerals in the granite.

The technetium concentration in ground water under repository conditions may be predicted assuming $TcO_2 \cdot nH_2O$ as the solid phase and $TcO(OH)_2^O$ and TcO_4^- as the predominant aqueous complexes using a formation constant for $TcO(OH)_2^O$ of $\log K = -8.16$ and a standard reduction potential E^O for the reaction $TcO_4^- + 3e^- + 4H^+ = TcO_2 \cdot nH_2O$ of 0.738 V.

The surface related distribution ratio K_a for $TcO(OH)_2^O$ between Stripa granite and ground water is approximately 1 cm based on geometrical surface area.

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1 INTRODUCTION

The long-lived fission product ^{99}Tc ($t_{1/2}$ =2.13·10^5 y) is present in large quantities in nuclear wastes and hence its chemical behaviour in the geosphere is of considerable interest.

When radioactive waste in an underground repository is exposed to groundwater radionuclides it can be leached out and become dispersed by diffusion in the backfill material and the connected microstructure of the host rock. The radionuclides may also be transported through the fracture network of the host rock by advective flow. It is imperative that under no circumstances hazardous quantities of radionuclides reach the biosphere. The radionuclide concentration at the source will be determined by the solubility and the migration retarded if the radionuclide is strongly sorbed on the rock.

The solubility and sorption of multivalent redox sensitive radionuclides, like ^{99}Tc, depend on the redox properties of the groundwater and the surrounding rock, pH and concentration of complexing agents in the ground water.

We have in earlier works studied the migration of Tc in granitic drill cores containing natural fissures under oxic and reducing (+260 to -280 mV) conditions (Eriksen 1985, 1988). Based on the retardation of Tc under reducing conditions and the geometric surface area of the fissure walls the surface related distribution coefficient R_a, defined as the ratio between the concentration of sorbed Tc (mol·cm^{-2}) and the concentration of Tc in ground water (mol·cm^{-3}), was found to be approximately 1 cm.

An important factor in the assessment of of the migration of a radionuclide is the maximum attainable concentration under the given conditions of water chemistry. Under oxic conditions technetium exists as the anionic species TcO_4^- whereas under reducing conditions, expected to exist in a deep bedrock repository for used fuel, it is generally predicted that technetium will be present as the sparingly soluble $Tc(IV)O_2 \cdot 2H_2O$.

Meyer et al (1986, 1987, 1988,1989) have carried out extensive studies on the solubility of precipitated and electrodeposited Tc(IV) oxides in aqueous solution at pH < 9.7 and the dependence of the solubility of $TcO_2 \cdot nH_2O$ in aqueous solution on pH and pCO_2 has recently been studied by Eriksen et al (1991). From the experimental results it is concluded that the solubility in granitic ground water ($2 \cdot 10^{-3}$ $mol \cdot dm^{-3}$ HCO_3^-, pH $\approx$ 8.2) is $6.8 \cdot 10^{-9}$ $mol \cdot dm^{-3}$ with $TcO(OH)_2^O$ as the predominant aqueous complex.

The purpose of this work is to study the behaviour of the $TcO_4^-/TcO_2 \cdot nH_2O$ redox couple in granite-water systems. The study encopasses two laboratory experiments.

In the first experiment we followed the reduction of TcO_4^- in reducing ground water circulating through a column of coarsly crushed granite.

In the second experiment we studied migration of Tc from $TcO_2 \cdot nH_2O$, electrodeposited on a platinum mesh electrode, to crushed granite in moderately reducing water (E_h approx - 90 mV).

2   EXPERIMENTAL

2.1 Materials

All reagents, of analytical grade, were used without further purification.

The radionuclides used were ^{99}Tc, delivered in 0.1 mol·dm^{-3} NH$_4$OH aqueous solution from Amersham, and ^{99m}Tc recovered as TcO$_4^-$ in 0.4 mol·dm^{-3} aqueous KCl solution from a ^{99}Mo generator (Kjeller).

Radionuclide solutions were prepared by diluting aliquots of the stock solutions with ground water.

Reducing groundwater was prepared by contacting argon purged (AGA 5.7-quality) triple distilled water with granitic rock in a quartz reservoir as described by Wikberg (1987) and Eriksen (1988). The rock used is granitic rock from the Stripa mine taken at a depth of 360m below ground level.

The redox potentials were monitored with a Metrohm Pt electrode and a Yokogawa SR 20/AR 24 (Ag/AgCl) reference electrode connected to a Metrohm 632 pH meter. The electrodes were standarized using saturated quinhydrone buffers at pH 4 and 7. The pH measurements were performed with a Radiometer pHM84 pH meter and a GK 2321c combined glass/reference electrode.

Both experiments were carried out in a controlled atmosphere box flushed with AGA 5.7-quality Ar containing < 0.5 ppm O$_2$.

2.2 Column experiment

The experimental set up is depicted in Figure 1. The column was filled with 82 cm^3 granitic coupons with approximate dimensions 1x1x1 cm and the entire flow system (total volume 201 cm^3) flushed with Ar.

Reducing groundwater from the reservoir was thereafter circulated through the column for several days to equilibrate the granite surfaces. The Tc-experiment was initiated by injecting 15 μl TcO_4^- solution ,initially containing 0.335 $mol \cdot dm^{-3}$ ^{99}Tc and approximately 10^{-11} $mol \cdot dm^{-3}$ ^{99m}Tc, into the circulating water. The water flow was 0.18 $cm^3 \cdot min^{-1}$ and the measured potential varied between -440 and -460 mV during the experiment.

The Tc transport through the column was initially followed by monitoring the activity of the shortlived ^{99m}Tc ($t_{1/2}$ = 6h) with a NaI scintillation detector fitted with a 20x4 mm lead collimator.

The water circulation was stopped after 4 days and samples were drawn from the water phase and analyzed for TcO_4^- and Tc(IV). The water samples were added to equivalent volumes of chloroform containing 0.1 $mol \cdot dm^{-3}$ tetraphenylarsonium chloride. The organic phase removes TcO_4^- quite efficiently leaving the Tc(IV)-species in the aqueous phase. Testing the extraction procedure we found that two consecutive extractions were sufficient to ensure complete removal of TcO_4^-. Following each extraction, samples from the organic and aqueous phase respectively were mixed with Ready Safe Liquid scintillation coctail (Beckman). The samples were analyzed for the pure β-

emitter ^{99}Tc (E = 0.293 MeV) in a Beckman model 5801 Liquid scintillation system, calibrated with standard solutions containing appropriate and known ^{99}Tc concentrations. The water was thereafter removed from the column and the granite coupons divided into eight fractions according to height above the TcO_4^- injection point. Each fraction was subdivided into two parts containing approximately 10 g granite and desorption of ^{99}Tc was carried out following two different procedures.

- each sample was quickly rinsed in Ar-purged distilled water before being transferred to 20 cm^3 flasks containing 10 cm^3 aerated water

- the samples were directly transferred to 20 cm^3 flasks containing 10 cm^3 aerated water.

The desorption was carried out for 104 days. The flasks were at appropriate intervals, following additions of H_2O_2 and HNO_3, placed in an ultrasonic bath for 30-40 minutes. The Tc concentration in the aqueous phase was determined by liquid scintillation counting of ^{99}Tc.

2.3 $TcO_2(s)$ experiment

The experimental arrangement is depicted in Figure 2. Small glass vials with porous walls were filled with crushed and size fractionated granite and placed a large vessel (approximately 2 dm^3) together with 130 g small granitic drill cores (2-4 cm diameter). The vessel was sealed and freed from

oxygen by Ar purging for two days and thereafter filled with 1.8 dm^3 water, which had been preequilibrated with granitic rock for eight days in the "ground water generator" (Wikberg, 1987; Eriksen, 1988). TcO$_2$·nH$_2$O, electrodeposited on a platinum net, was immersed into the water and water samples were drawn at intervals and analyzed for Tc(IV) and TcO$_4^-$ as described above.

At the end of the experiment the different size fractions of crushed granite were quickly rinsed with reducing water from the ground water generator and transferred into small flasks containing 19.2 cm^3 Ar purged water from the generator. After two days in the controlled atmosphere box the flasks were taken out of the box ,opened and aerated. At intervals, following additions of H$_2$O$_2$ and/or HNO$_3$, the flasks were placed in an ultrasonic bath for 30-40 minutes. The total Tc concentration in the aqueous phase was determined by liquid scintillation counting of ^{99}Tc.

3. EXPERIMENTAL RESULTS

3.1 Column experiment

The dispersion of Tc in the granite column, monitored by measurement of the ^{99m}Tc activity, following the injection of TcO$_4^-$ to the circulating reducing water is shown in Figure 3 and the break through of Tc into the water phase, measured 2 mm above the granite column, is shown in Figure 4. The Tc(IV) and Tc(VII) concentrations in the circulating water 3 and 4 days after the TcO$_4^-$ addition respectively are given in Table 1.

Table 1.

Concentration of Tc(IV) and TcO_4^- in the circulating water.

Time after TcO_4^- injection (days)	Tc(IV) $mol \cdot dm^{-3}$	TcO_4^- $mol \cdot dm^{-3}$ *
0		$4.22 \cdot 10^{-5}$
3	$1.12 \cdot 10^{-8}$	$3.89 \cdot 10^{-5}$
4	$1.17 \cdot 10^{-8}$	$3.90 \cdot 10^{-5}$

* assuming complete mixing (15 μl 0.335 $mol \cdot dm^{-3}$ TcO_4^- added to 119 cm^3 circulating water).

As seen, whereas the TcO_4^- concentration in the circulating water decreased by $0.32 \cdot 10^{-5}$ $mol \cdot dm^{-3}$ only $1.17 \cdot 10^{-8}$ $mol \cdot dm^{-3}$ was retrieved as Tc(IV) in solution. The Tc(IV) concentration is but slightly higher than the solubility of $TcO_2 \cdot nH_2O$ as determined by Eriksen et al (1991).

The granite coupons used for desorption/leaching of Tc were sampled at appropriate positions in the granite column (see Figure 5). The release of Tc from rinsed and unrinsed coupons are plotted in Figures 6-8 and 9-10 respectively. In both cases there is a marked increase in the amount of released ^{99}Tc, given as activity (cpm) per gram granite, with increasing height in the upper part of the granite column. Based on the desorption profile in the column and the total weight of the granite we estimate the total amount of Tc retrieved from the granite samples to be $(2.7 +/- 0.5) \cdot 10^{-7}$

moles, corresponding to a $(2.3 +/-0.5) \cdot 10^{-6}$ mol$\cdot$dm^{-3} decrease in the TcO_4^- concentration of the circulating water.
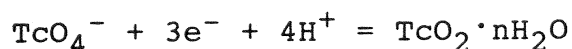
3.2 $TcO_2(s)$ experiment

The redox potential was monitored continuously throughout the various experimental phases. As can be gleaned from section A of Figure 11 constant E_h was obtained in the groundwater generator after approximately one week. Groundwater was thereafter pumped into the vessel containing the crushed granite (section B). The platinum mesh electrode with electrodeposited $TcO_2 \cdot nH_2O$ was lowered into the water at B/C and water samples drawn for $Tc(IV)/TcO_4^-$ analysis after 43, 46, 50 and 51 days respectively. The concentrations and redox potentials on the normal hydrogen electrode scale are given in Table 2.

Applying Nernst equation

$$E = E^O + 2.3(RT/nF) \cdot log(P_i \text{ ox }^{ni}/P_j \text{ red }^{nj})$$

to the reaction

$$TcO_4^- + 3e^- + 4H^+ = TcO_2 \cdot nH_2O$$

we can, using $E^O = 0.738$ V (Meyer et al 1989) calculate the expected equilibrium concentration of TcO_4^- at the experimental pH and redox conditions. As can be seen from Table 2 ,the measured TcO_4^- concentrations are 2-3 times higher than the calculated ones. It should, however, be pointed out that the uncertainty in the measured redox potential is 15-20 mV which may account for the discrepancies between calculated and measured concentrations.

Table 2.

Redox potential, pH, Tc(IV) and TcO_4^- concentrations for samples of the water phase.

Time days	pH	$E_h^{(1)}$ mV	$Tc(IV) \cdot 10^9$ $mol \cdot dm^{-3}$	$TcO_4^- \cdot 10^5$ $mol \cdot dm^{-3}$ measured	calc*
43	9.18	-90	4.8+/-1.7	2.1	0.49
46	9.18	-92	4.2+/-1.5	2.24	0.39
50	9.18	-85	5.6+/-0.1	2.40	0.88
51	9.18	-83	4.7+/-0.2	2.51	1.10

(1) rel normal hydrogen electrode, (ref electrode +226 mV)

 * calculated using the equation:

$$E = 0.738 + 0.0197 \log [TcO_4^-] - 0.0788 \cdot pH$$

The Tc concentrations in reducing water due to release of Tc from the crushed granite are plotted versus the size fractions in Figure 12. As can be seen the measured Tc concentrations correspond to the solubility of $TcO_2 \cdot nH_2O$ (Eriksen et al 1991). The slight increase in the Tc concentrations with time is probably caused by slow intrusion of oxygen into the flasks.

The effect of aerating the bottles on the release of Tc from the crushed granite is very small (Figure 13), indicating a slow reaction with oxygen, whereas addition of H_2O_2 results in a marked increase in the Tc release.

Assuming that the Tc, released from the crushed granite, was sorbed as Tc(IV) we have, based on the concentration of Tc(IV) species in the vessel, calculated a formal distribution ratio (R_d) for each size fraction. The distribution ratios are plotted versus the size fractions in Figure 14.

There is clearly no consistent correlation between the distribution ratio and the particle size, the increase in R_d with decreasing particle size in the size range 250-2000 μm is followed by a sudden decrease in R_d for particels smaller than 250 μm.

4 DISCUSSION

From the column experiment it may be concluded that the oxidation of TcO_4^- in reducing groundwater is very slow. Most, if not all, of the TcO_4^- dissappearing from the circulating water is sorbed or precipitated on the granite. The concentration of Tc(IV) species in the circulating water corresponds to the solubility of $TcO_2 \cdot nH_2O$ clearly indicating surface deposition of the oxide.

In the $TcO_2(s)$ experiment the TcO_4^- concentration in the aqueous phase is, within the experimental uncertainty, in agreement with the concentration calculated for the $TcO_2 \cdot nH_2O/TcO_4^-$ redox couple using the Nernst equation.

In an earlier study of sorption on crushed granite (Eriksen and Locklund 1989) we measured the N_2-BET surface area of crushed and intact granites. The experimental results were accommodated by a model of porous particles with accessible outer and inner surfaces. On linear regression analysis of the

R_d/surface area data for ^{99}Tc in the size range 250-2000 μm we obtain an intercept with the surface area axis at 0.24 m$^2 \cdot$g^{-1}. Corresponding plots of our earlier data on sorption of ^{85}Sr, ^{134}Cs and ^{152}Eu and cationic exchange capacity of crushed granites are shown in Figures 16-21. With the notable exception of ^{152}Eu sorption the regression lines pass close to or through origo. An intercept with the surface area axis at 0.24$\cdot$m^2 g^{-1} for Stripa granite indicates that only outer surfaces have participated in the process. In the case of ^{152}Eu this was explained as being due to strong sorption resulting in depletion of ^{152}Eu in the aqueous phase and thereby slow diffusion and redistribution on the inner surfaces.

We have in an earlier report discussed the migration of Tc in natural granitic fissures under reducing conditions (Eriksen et al 1985, 1988). Based on the retardation of Tc relative to water we estimated the geometric surface based distribution ratio R_a for Tc(IV) between granite and ground water to be 1 cm.

Assuming the crushed granite to consist of spherical particles with diameters equal to the arithmetic means of the sieves bracketing the various size fractions we estimate R_a to be approximately 60 cm in the TcO$_2$(s) experiment, i.e nearly two orders of magnitude higher than the R_d obtained in the migration experiments.

We have hitherto used the term sorption to describe the loss of a radionuclide from an aqueous solution phase to a contiguous solid phase. Sorption may occur by several

mechanisms eg precipitation, (formation of a solid phase) and adsorption (Sposito 1986).

The migration experiments were carried out with total Tc concentration at or lower than the solubility limit of $TcO_2 \cdot nH_2O$ whereas a solid $TcO_2 \cdot nH_2O$ phase as well as Tc(IV) and TcO_4^- in solution were present in the TcO_2(s) experiment. It is evident that the chemical system in the latter experiment is more complicated and several sorption processes may be involved.

A plausible explanation to the apparent disagreement between the R_a values obtained in the two experiments would be that the distribution ratio measured in the migration experiment characterises adsorption of the aqueous Tc(IV) species $TcO(OH)_2^O$ on the granite whereas both reduction/precipitation of TcO_4^- and sorption of $TcO(OH)_2^O$ occured in the TcO_2(s) experiment, thereby yielding a higher R_a value.

The transfer of Tc from the $TcO_2 \cdot nH_2O$ electrode to the crushed granite can be accommodated by a process involving dissolution of Tc(IV) from the electrode, oxidation of aqueous Tc(IV) to TcO_4^- and surface mediated reduction/precipitation of TcO_4^- as $TcO_2 \cdot nH_2O$ on the granite surface.

Data from sorption studies of iron containing minerals (Vandergraaf 1984), iron oxyhydoxides (Walton 1986) and a Fourier transform infrared study of technetium-iron oxide reactions (Haines 1987) clearly demonstrate that TcO_4^- is reduced to $TcO_2 \cdot nH_2O$ and precipitated on the mineral surfaces.

Thus the reduction of TcO_4^- and precipitation of $TcO_2 \cdot nH_2O$ on the granite surfaces is most probably caused by small amounts of iron containing minerals or oxides in the granite. A possible cause of the sudden drop in $TcO_2 \cdot nH_2O$ precipitation for particles <250 μm is Fe-depletion in the smaller size fractions during the crushing and size fractionation process.

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Walton F B, Paquette J, Ross J P M and Lawrence W E (1986).
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Fig. 1
Schematic illustration of granite
Column experiment.

Fig. 2.
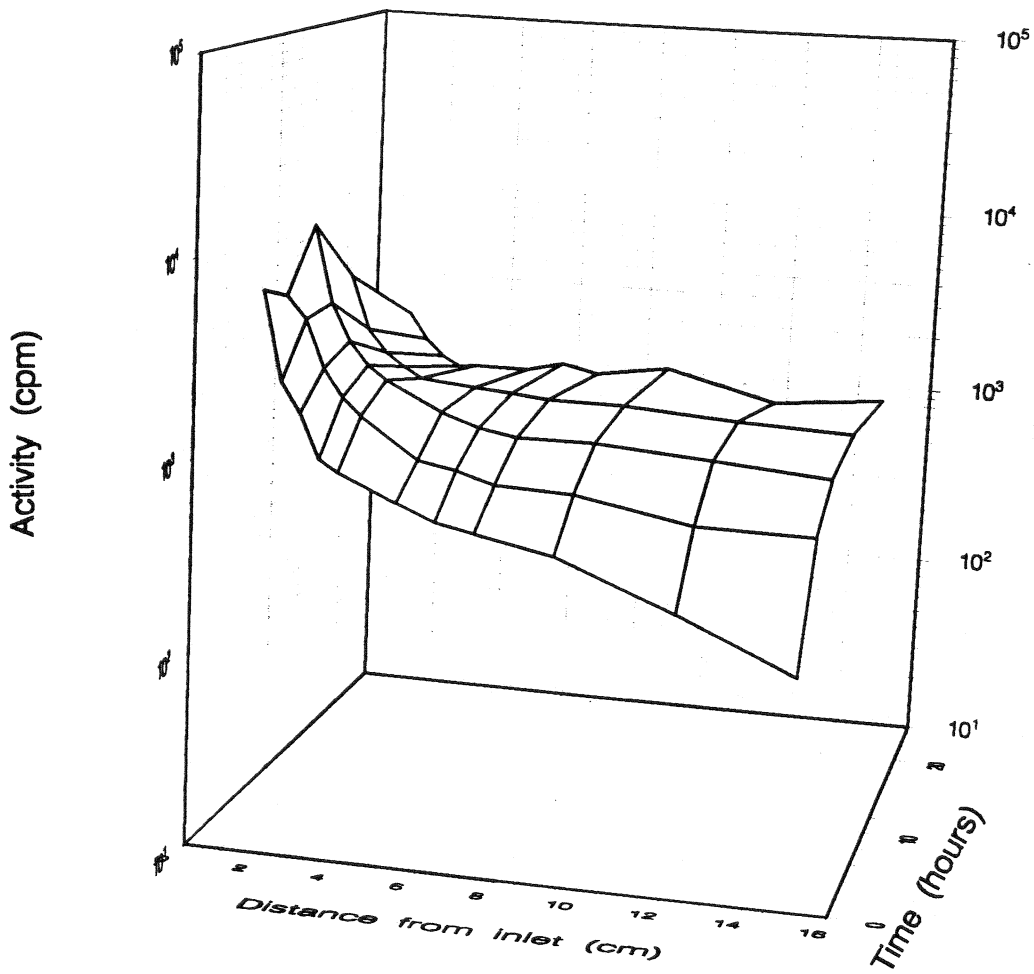Schematic illustration of $TcO_2(s)$ experiment.

Figure 3.

^{99m}Tc distribution in granite column after TcO$_4^-$ injection into circulating water.
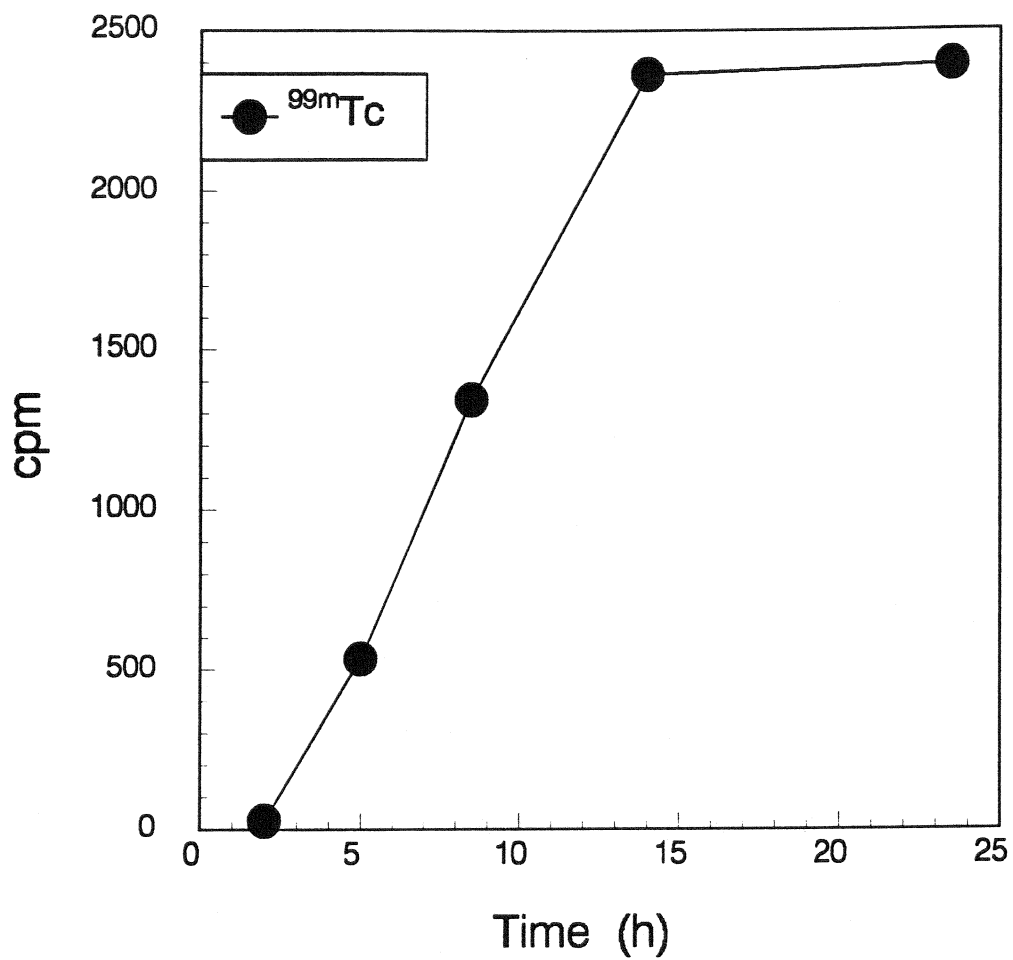
Figure 4.

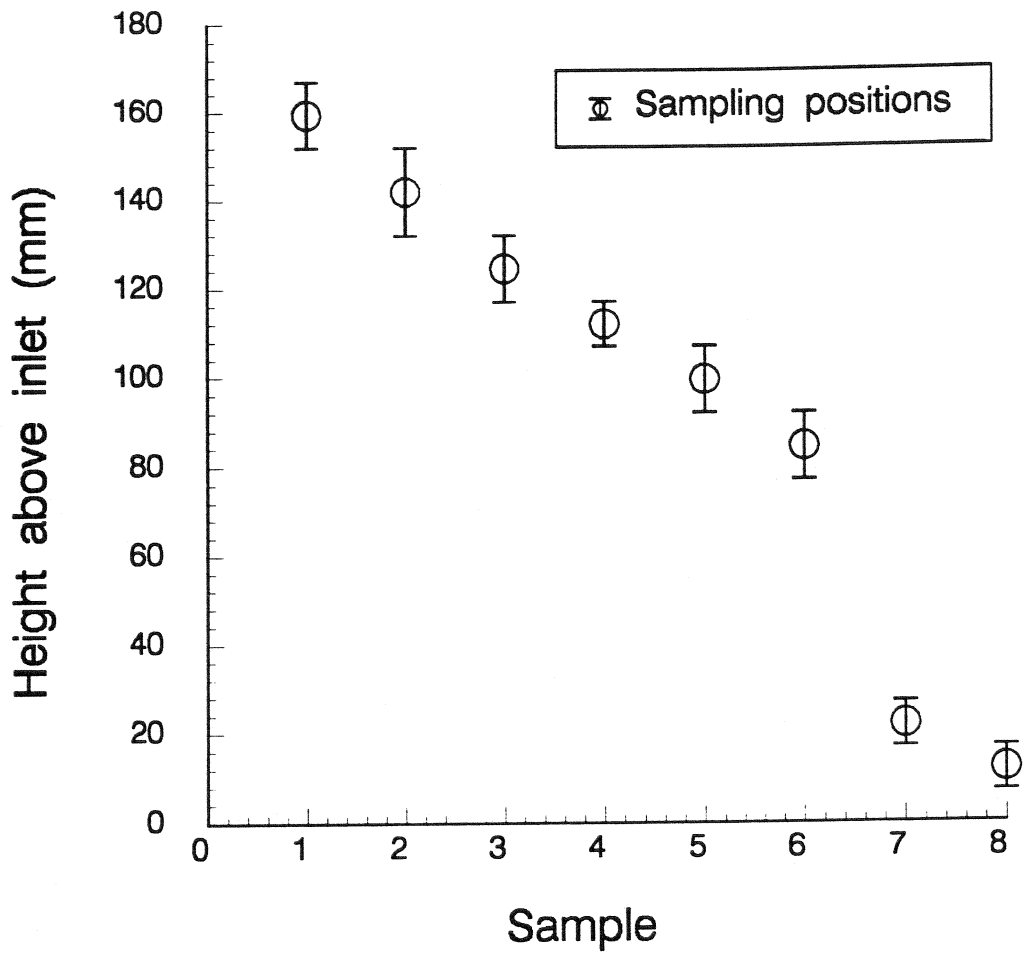^{99m}Tc activity in water phase 2mm above the granite column.

Figure 5.

Positions of granite column samples for leaching of adsorbed/precipitated ^{99}Tc.
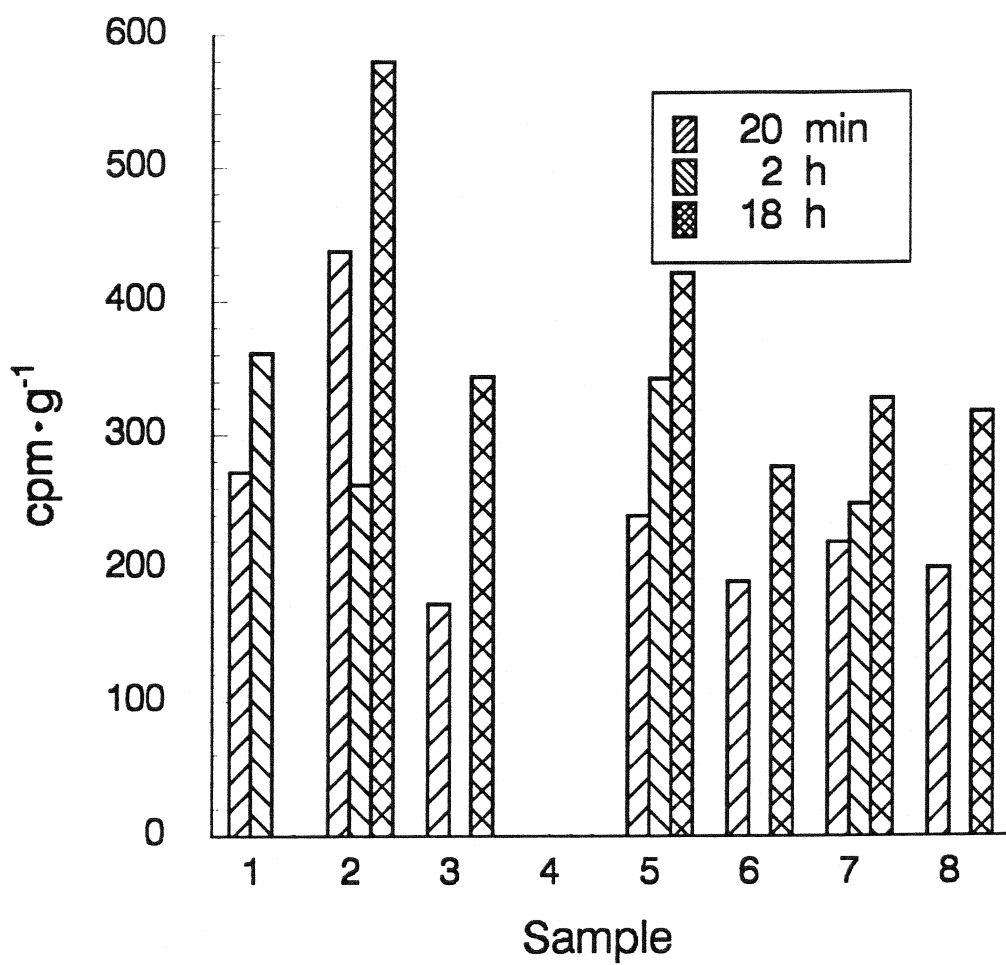
Figure 6.

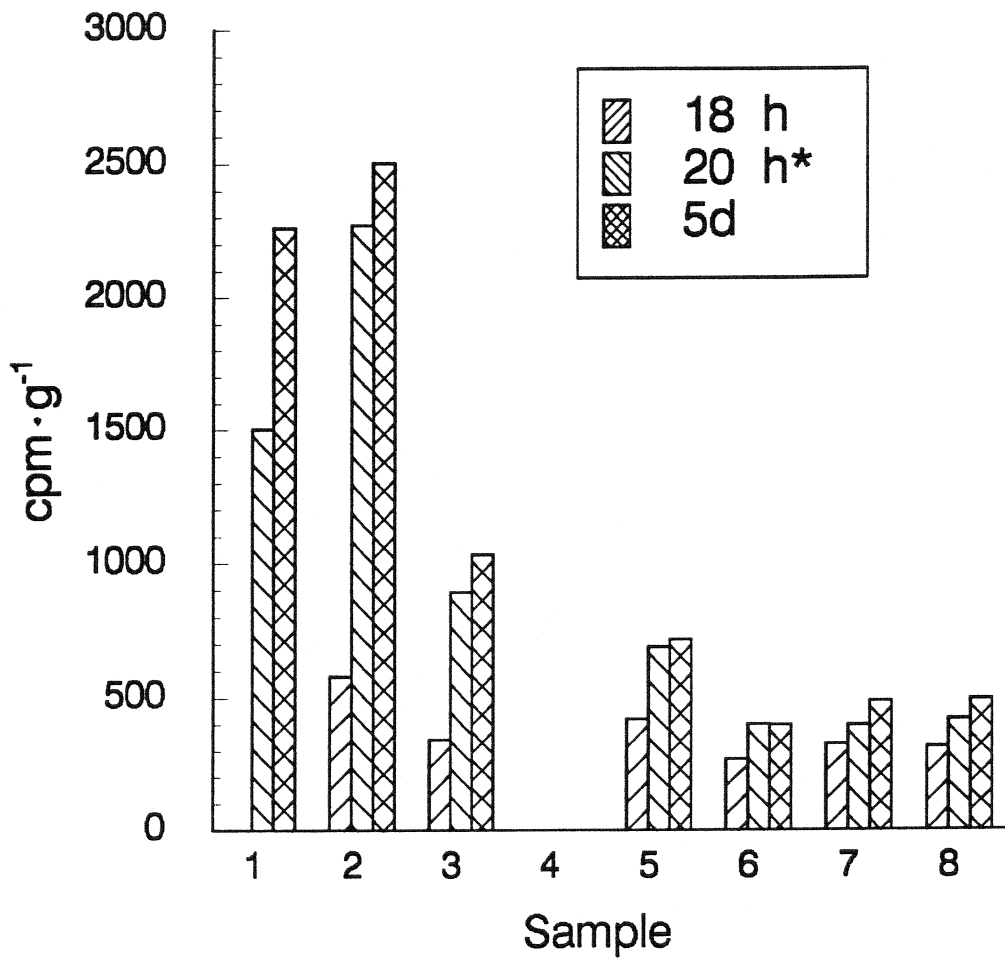Activity of ^{99}Tc released from rinsed granite-column samples.

Figure 7.

Activity of ^{99}Tc released from rinsed granite-column samples.

* 0.1 cm^3 0.2N HNO_3 added to samples 2,3,6,7,8.
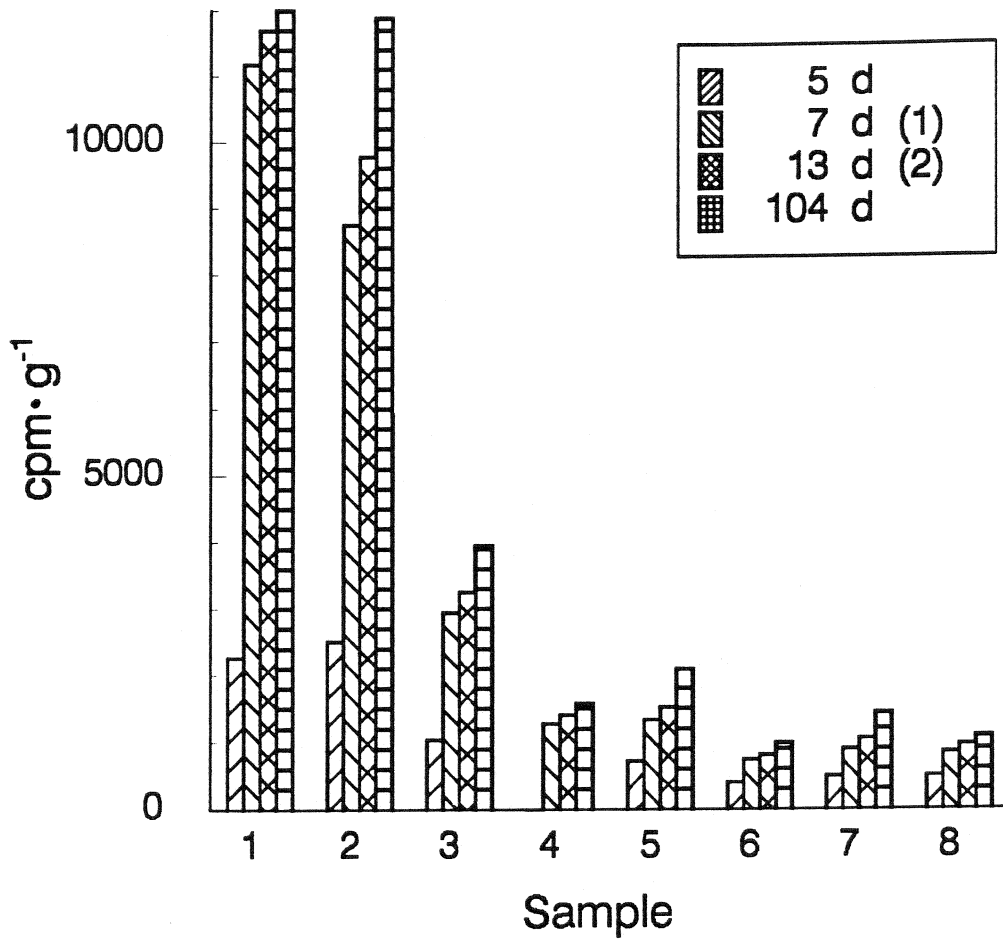Ultrasonic agitation for 30 min on addition.

Figure 8.

Activity of ^{99}Tc released from rinsed granite-column samples.

(1) 0.1 ml   0.2M HNO$_3$ added to 1.4.5
(2) 0.15 ml H$_2$O$_2$ added to all samples.

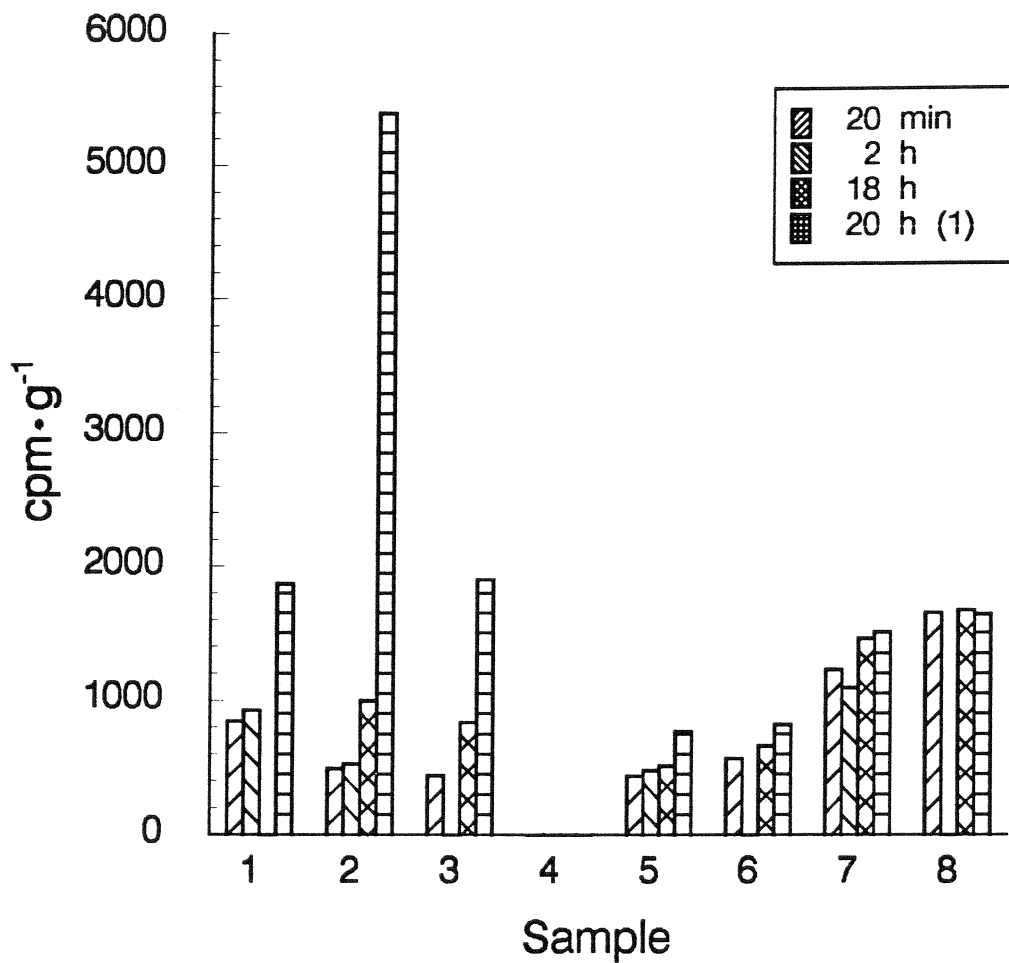Ultrasonic agitation for 30 min on addition.

Figure 9.

Activity of ^{99}Tc released from granite-column samples (not rinsed).

(1) 0.1ml 0.2M HNO$_3$ added to 2,3,6,7,8.
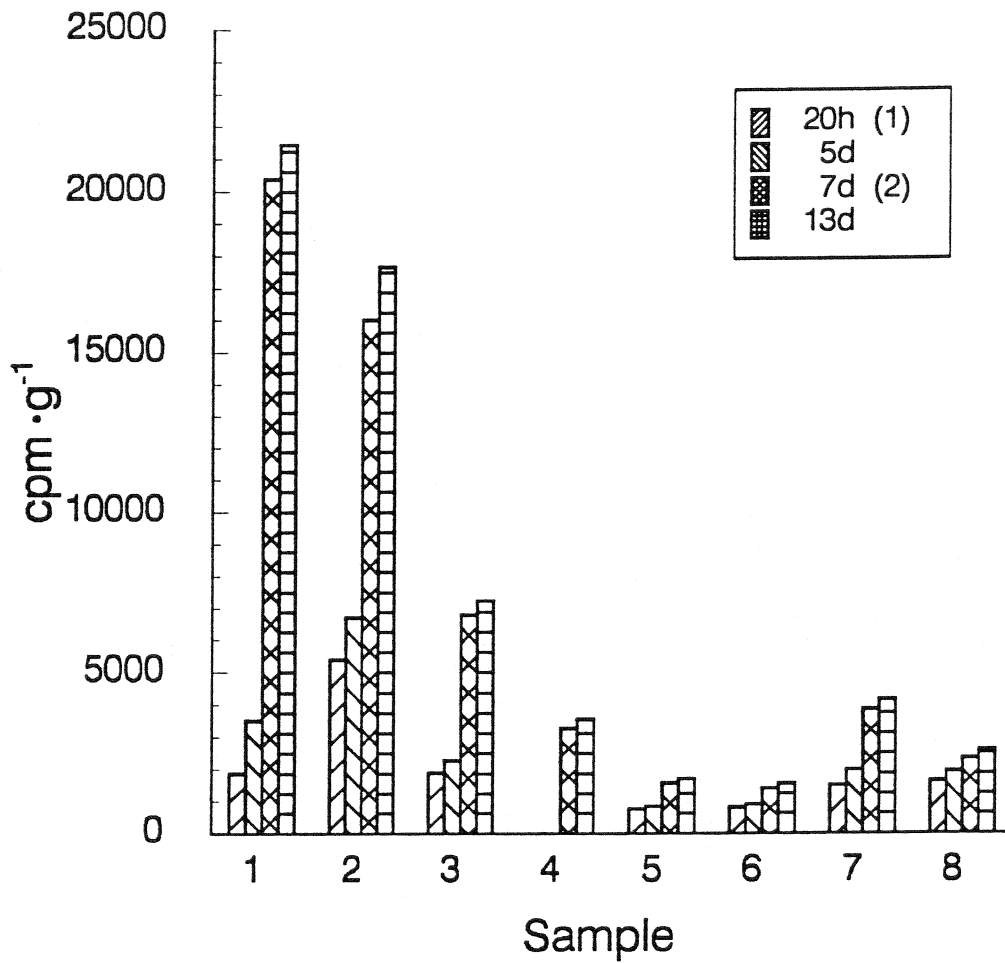
Ultrasonic agitation for 30 min on addition.

Figure 10.

Activity of ^{99}Tc released from granite-column samples (not rinsed).

(1) 0.1 cm^3 0.2M HNO$_3$ added to 2,3,6,7,8.

(2) 0.1 cm^3 0.2M HNO$_3$ added to 1,4 0.15 cm^3

H$_2$O$_2$ added to all samples.
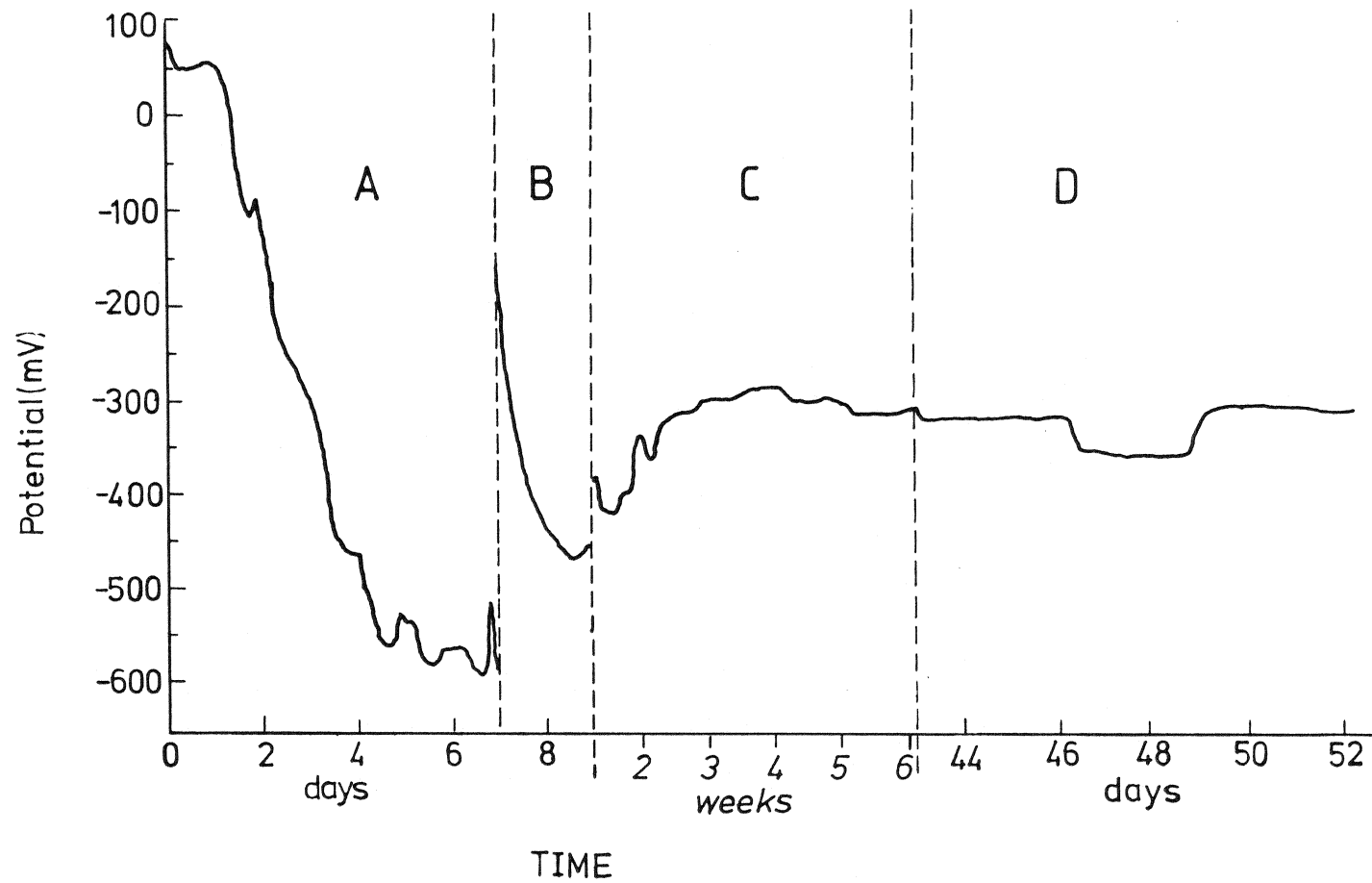
Fig. 11

E_h time plot for $TCO_2(s)$ experiment
A: ground-water gerator ; B: water in electrode vessel
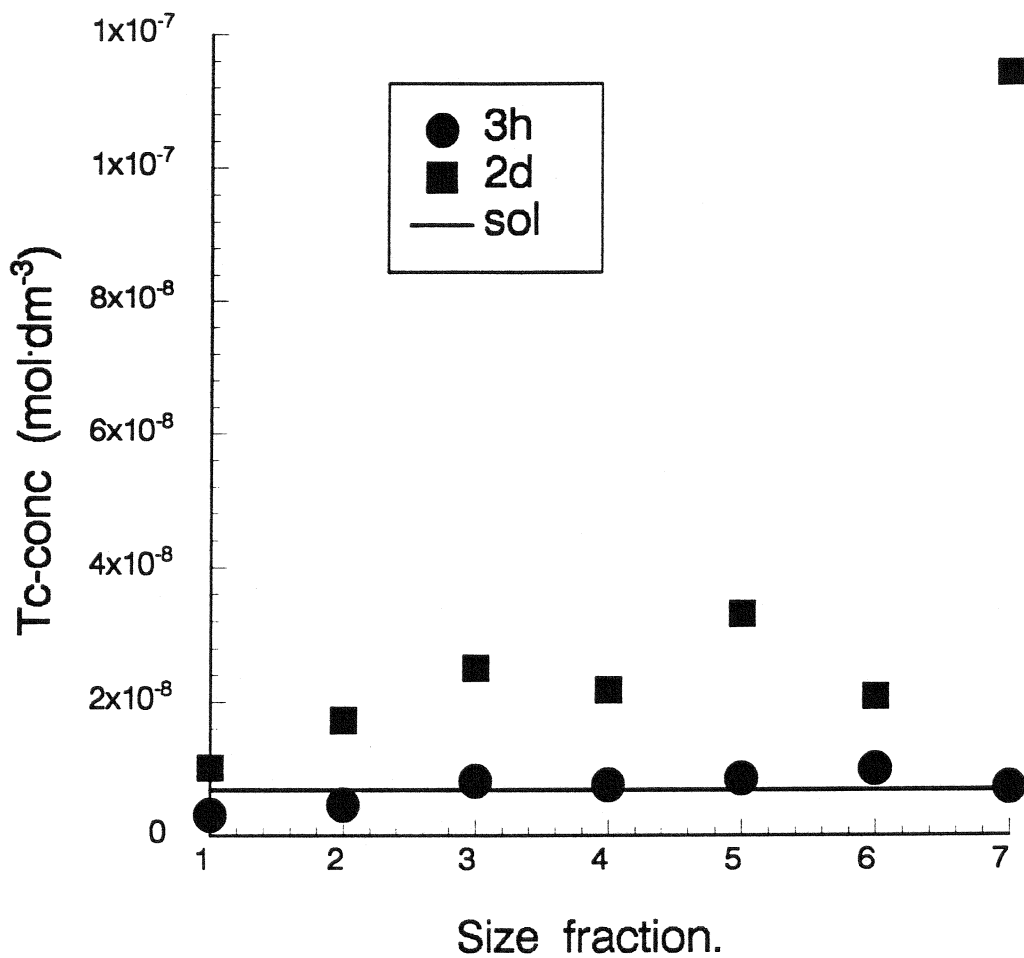B/C: immersion of TcO_2-electrode ; D: sampling period

Figure 12.

[99]Tc released into reducing water from crushed Stripa granite. Full line corresponds to solubility of $TcO_2(s)$.

Size fractions (µm): 1 (63-125); 2 (125-250)
3 (250-500); 4 (250-750); 5 (500-750); 6 (1000-2000)
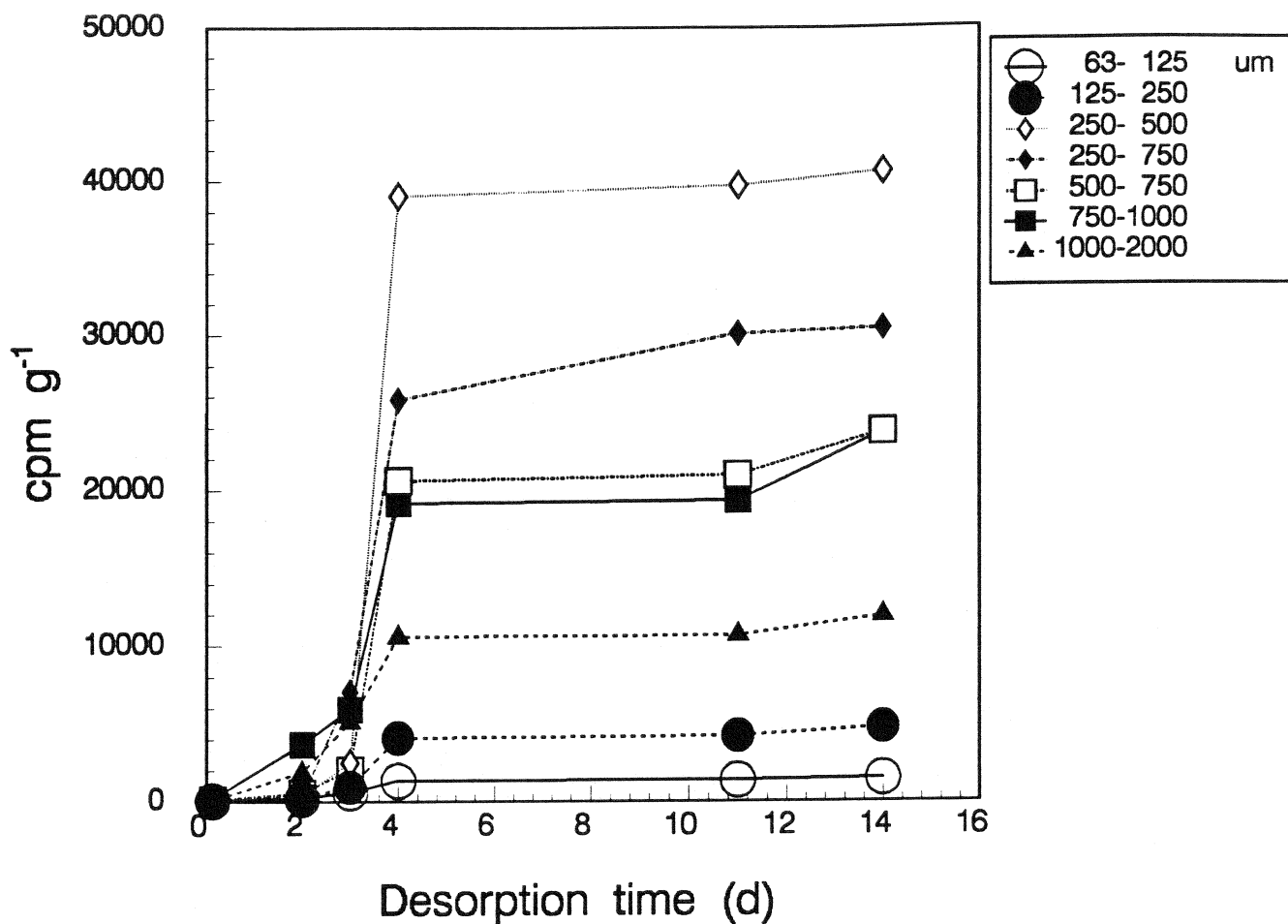7 (1000-2000)

Figure 13.

Activity of ^{99}Tc released from crushed Stripa granite.

0-2d reducing water, 2-3d air saturated water
3-4d 0.6M ,4-11d 1.4M H_2O_2
11d 100 µl HNO_3.

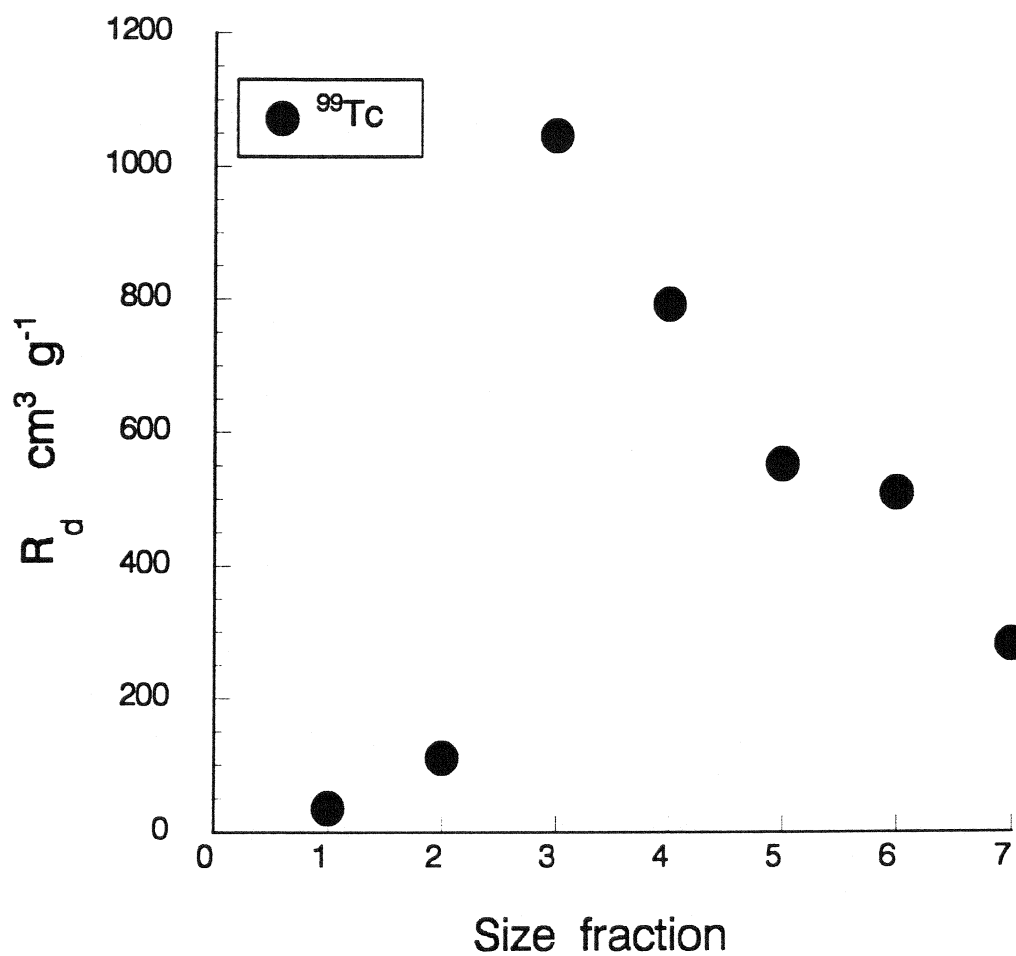Ultrasonic agitation on air saturation and additions.

Figure 14.

Distribution ratio R_d plotted vs particle fraction. Calculation based on amount of ^{99}Tc released from crushed granite and Tc(IV) solubility ($6.8 \cdot 10^{-9}$ $mol \cdot dm^{-3}$).

Particle size (μm): 1 (63-125); 2 (125-250); 3 (250-500) 4 (250-750); 5 (500-750); 6 (750-1000); 7 (1000-2000)

Figure 15.

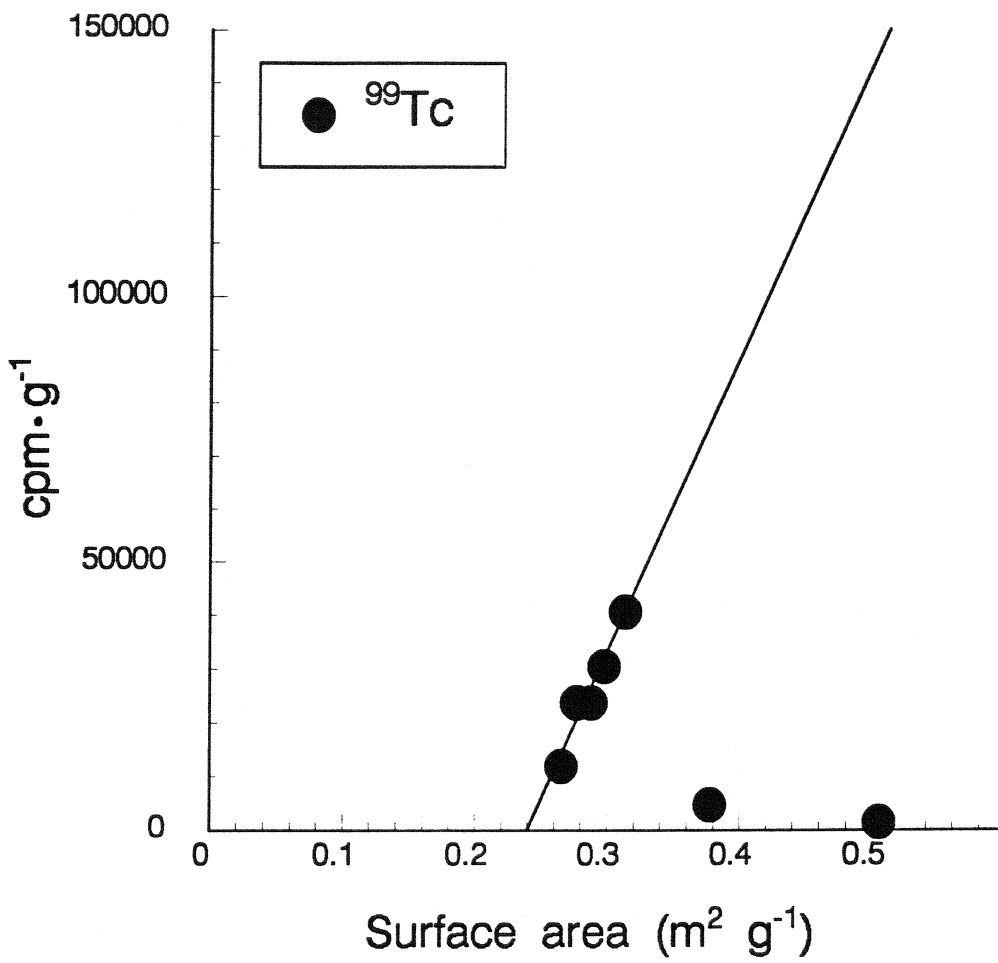Activity of ^{99}Tc released from crushed Stripa granite plotted versus (N_2) BET surface area.

Figure 16.

[152]Eu sorption on crushed Stripa granite.
Surface area measured by N$_2$ sorption.

Figure 17.

Cationic exchange capacity (CEC) of crushed
Stripa granite plotted vs (N_2) BET surface
area.

Figure 18.

[134]Cs sorption on crushed Stripa granite.
Surface area measured by N_2 sorption.

Fig 19.

[85]Sr sorption on crushed Stripa granite.
Surface area measured by (N_2) sorption.

Figure 20.

^{85}Sr sorption on crushed granite plotted versus surface area measured by N_2 sorption.

Figure 21.

^{85}Sr sorption on crushed Pingstaberg granite. Surface area measured by N_2 sorption.
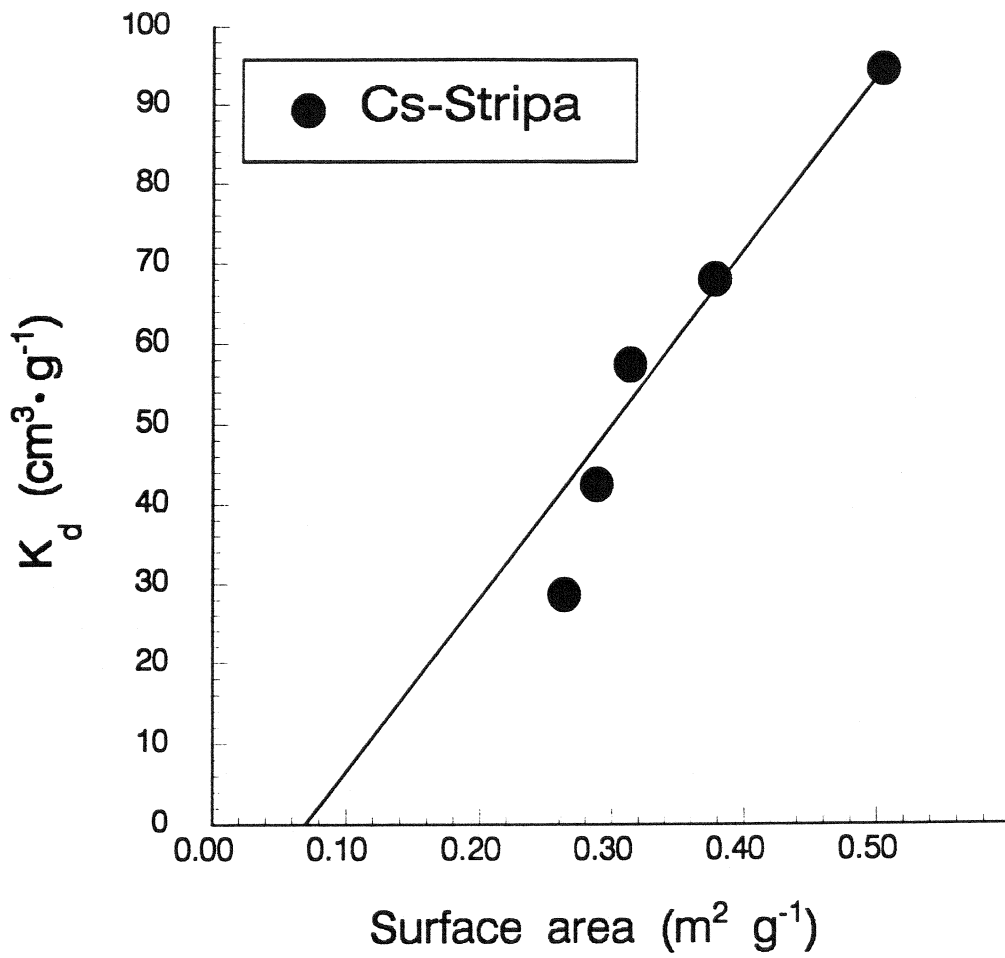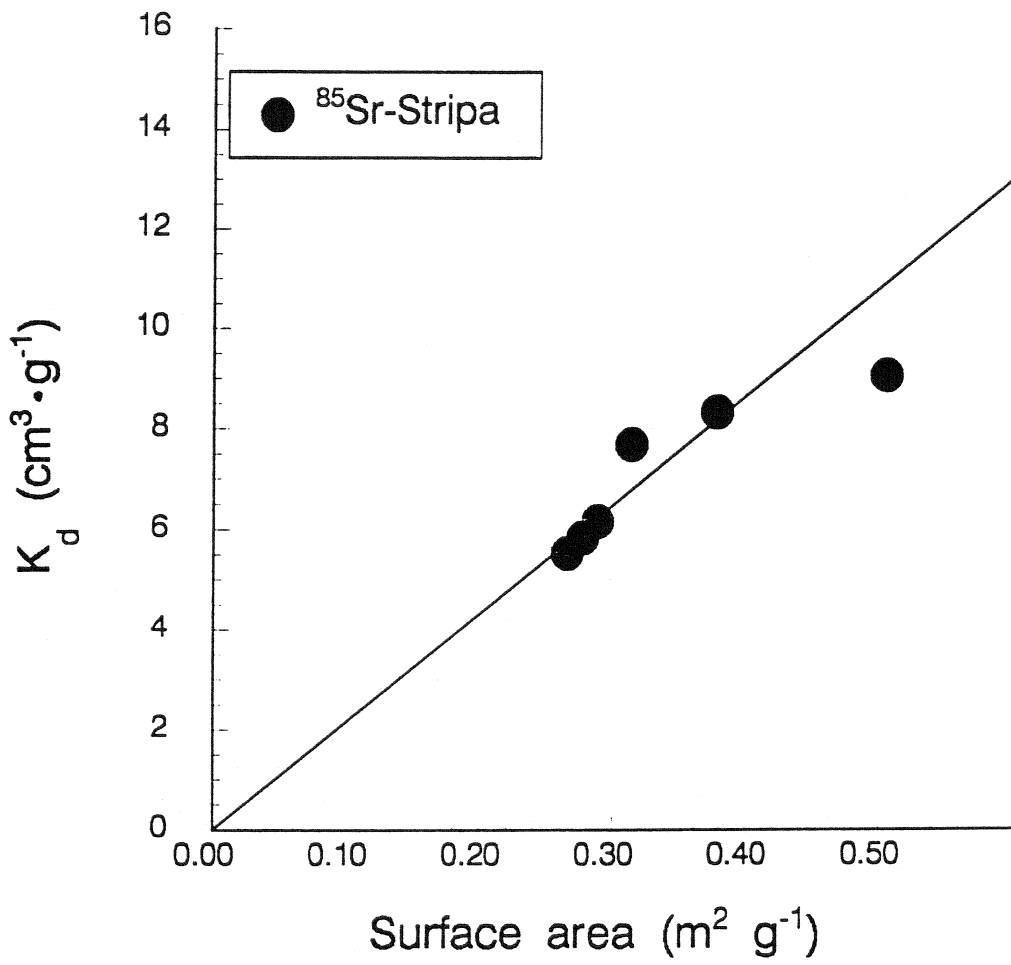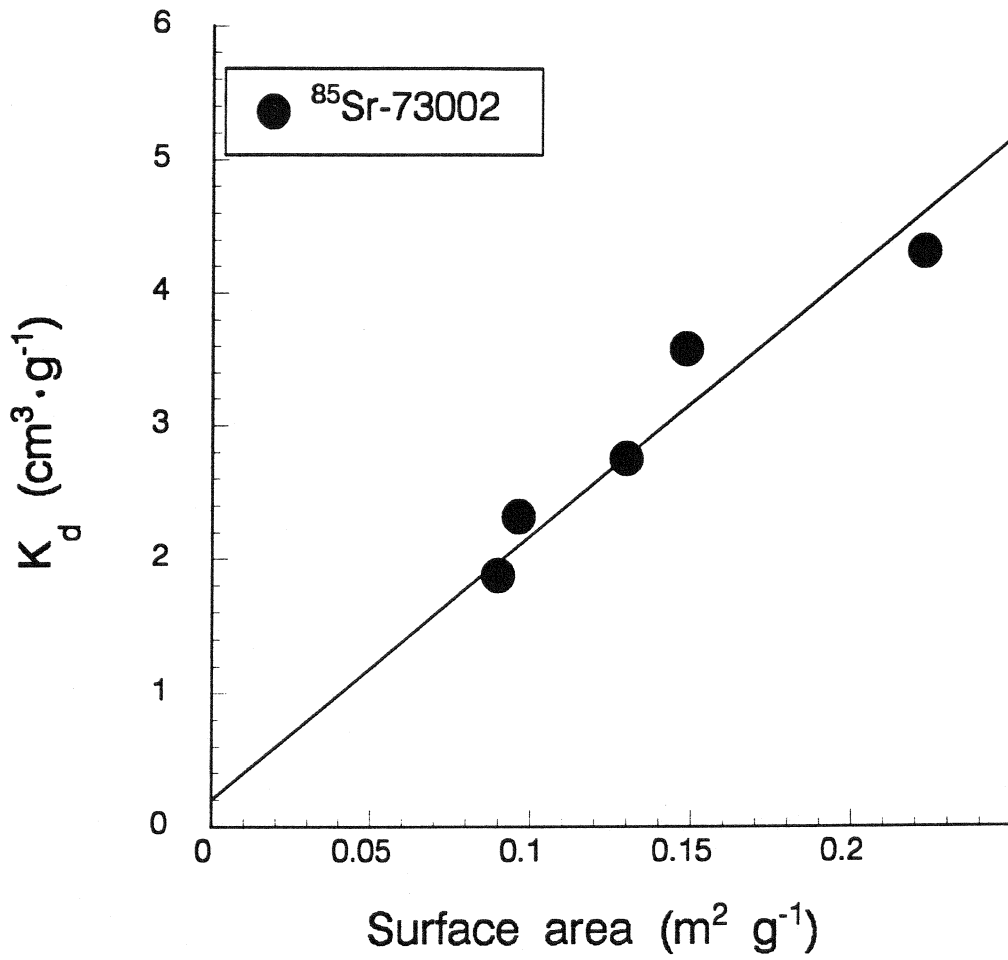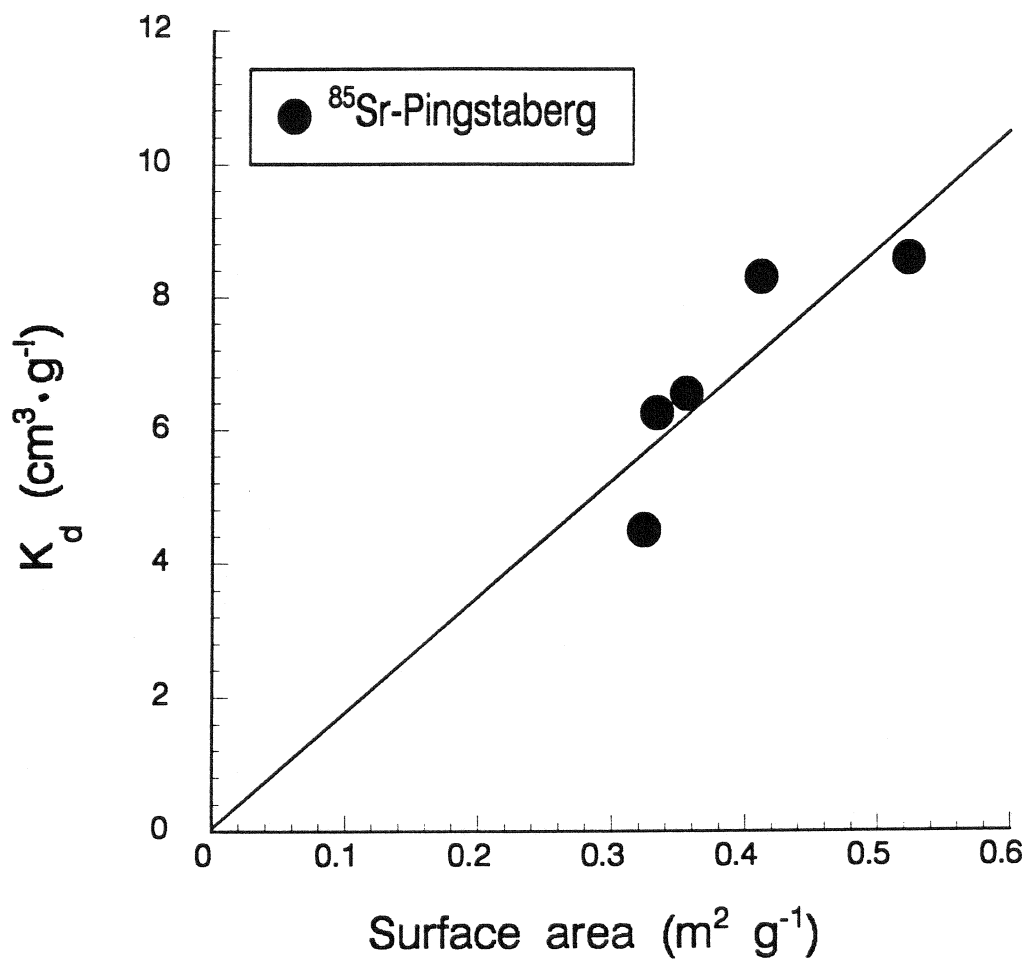
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I Puigdomènech[1], J Bruno[2]
[1]Enviromental Services, Studsvik Nuclear, Nyköping, Sweden
[2]MBT Tecnologia Ambiental, CENT, Cerdanyola, Spain
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[1]SGAB, Luleå
[2]SGAB, Uppsala
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Kaj Ahlbom[1], Sven Tirén[2]
[1]Conterra AB
[2]Sveriges Geologiska AB
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[1]Vattenfall HydroPower AB
[2]Golder Geosystem AB
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Akke Bengtsson[1], Bertil Grundfelt[1], Anders Markström[1], Anders Rasmuson[2]
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[2]Chalmers Institute of Technology
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[1]Starprog AB
[2]SKB AB
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[1]KEMAKTA Consultants Co
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Department of Chemical Engineering, Royal Institute of Technology, Stockholm
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[1]Conterra AB
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[1]MBT Tecnologia Ambiental, CENT, Cerdanyola, Spain
[2]KTH, Dpt. of Inorganic Chemistry, Stockholm, Sweden
[3]VTT, Tech. Res. Center of Finland, Espoo, Finland
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[1]Tyréns
[2]Clay Technology AB
[3]ABB Atom
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