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ON THE FORMATION OF A MOVING REDOX-FRONT BY α-RADIOLYSIS OF COMPACTED WATER SATURATED BENTONITE

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On the formation of a moving redox-front by α-radiolysis of compacted water saturated bentonite.

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Abstract.

The formation of an expanding volume containing the radiolytically formed oxidants H_2O_2 and O_2 has been studied in α-irradiated compacted water saturated bentonite (ρ = 2.12 $g \cdot cm^{-3}$). The G-values (0.67+/-0.05), (0.64+/-0.07) for H_2O_2 and O_2 respectively are in fair agreement with the corresponding G-values obtained in experiments with synthetic ground water. From the leaching of γ-irradiated bentonite it is concluded that only a fraction of the Fe^{2+} content is easily accessible as scavenger for the radiolytically formed oxidants.

CONTENT.

1. INTRODUCTION.

On radiolysis of the pore water in compacted water saturated bentonite oxidizing and reducing species are formed in equal amounts. The radiolytic yields of the radicals e^-_{aq}, $H^\cdot$, $OH^\cdot$, $HO_2^\cdot$ and the molecular products H_2O_2, H_2 and O_2 are strongly dependent on the LET (Linear Energy Transfer) of the radiations, the G-value of the molecular products increasing with increasing LET at the expense of the radical yields.

The primary oxidizing and reducing species react with solutes in the pore water and the redox potential will be strongly influenced by the composition of the aqueous phase eg pH, concentration of carbonate and iron.

Due to its low reactivity and high diffusivity H_2 will diffuse out of the irradiated volume leaving behind an excess of reactive oxidizing species. The Fe^{2+} ion is a good scavenger for oxidizing radicals and H_2O_2, O_2. The solubility of the product Fe^{3+} is much lower than the solubility of Fe^{2+} at the expected environmental condition in a nuclear waste repository ($pH = 8$, $HCO_3^- = 2\ mmol \cdot dm^{-3}$) and Fe^{3+} will most probably be precipitated. The rate of expansion of the volume containing radiolytically created oxidizing species will therefore be strongly dependent on the accessibility of Fe(II) as a sink for oxidizing species.

The diffusion of radiolytically formed H_2 out of irradiated volumes compacted water saturated bentonite has been demonstrated in several studies (Eriksen and Jacobsson 1983, Eriksen et al 1987, 1988) and reasonable agreement was obtained between experimental G-values and G-values calculated using the computer program CHEMSIMUL (Christensen and Bjergbakke, 1986).

The formation of an expanding oxidative volume i.e. a moving redox front on α-irradiation of synthetic ground water has recently been demonstrated in experiments with a glass filter as diffusion barrier (Eriksen et al 1988). The experimental $G(H_2O_2)$ value obtained was lower than the value calculated by CHEMSIMUL, whereas the overall oxidizing capacity assumed to be due to H_2O_2 and O_2 was found to agree within the experimental error.

The aim of the present work is to assess the accessibility of Fe(II) as a sink term for oxidizing species in compacted water saturated bentonite and to demonstrate experimentally the formation of a moving oxidative redox front.

2. EXPERIMENTAL.

2.1 Material.

The clay used in the present study is the American Colloid Co type Mx-80 granulated Na-bentonite. The water used in the radiolysis experiments was, if not otherwise stated, synthetic ground water, the composition of which is given in Table 1.

Table 1

Composition of synethetic ground water*.

Species	Concentration (mg·dm^{-3})
HCO_3^-	123
SO_4^{2-}	9.6
Cl^-	70
SiO_2	12
Ca^{2+}	18
Mg^{2+}	4.3
Na^+	65
K^+	3.9

* pH 8-8.2

2.2 Fe(II)-leaching.

Clay samples were suspended in varying volumes of iron-free double distilled water. After 24 h the Fe^{2+} and Fe^{3+} concentrations in the aqueous phase of the suspensions were determined colorimetrically with o-phenanthroline according to the procedure described in literature (Fortune and Mellon, 1938; Gerstl and Banin, 1980).

2.3 Gamma irradiation of washed bentonite.

Water soluble Fe^{2+} was removed by washing Mx-80 with double distilled water. Bentonite suspensions were thereafter purged with Ar (AGA-SR quality) containing < 1 ppm O_2 and irradiated in a AECL-Co-60 unit with dose rate 240 krad·h^{-1} to a maximum dose of 12 Mrad. Corresponding suspensions containing 10 mmol·dm^{-3} formate were irradiated to 15 Mrad. In this system the ·OH radical is converted to the reducing radical ion $CO_2^{\cdot -}$ by the reaction

$$OH + HCO_2^- \rightarrow CO_2^{\cdot -} + H_2O$$

The irradiated samples were analyzed for Fe^{2+} and Fe^{3+}, as described above.

2.4 Gamma irradiation of untreated bentonite.

1 g Mx-80 in equilibrium with air at ambient humidity and temperature was suspended in 3 cm^3 N_2O saturated synthetic ground water. The N_2O converts the hydrated electron e^-_{aq} to a ·OH radical by the reaction

$$e^-_{aq} + N_2O \rightarrow N_2 + {}^{\cdot}OH + OH^-$$

and a oxidative solution is thus obtained. The irradiated samples were analyzed for Fe^{2+} and Fe^{3+}.

2.5 α-irradiation.

2.5.1 Radiation source.

The radiation source consisted of ^{241}Am incorporated into a gold matrix on silver backing. The α-emitting surface was covered by a 2 μm thick layer of a gold-palladium alloy. The diameter of the active surface was 25 mm and the total activity 35.7 MBq. The average energy of the α-particles leaving the surface was, as determined by α-spectroscopy (Eriksen et al, 1987), 4.6 MeV and the energy deposition $8.22 \cdot 10^{13}$ $eV \cdot s^{-1}$. The range of the α-particles is taken to be 37 m in water (Flügge, 1958) and thus 17 μm in the water saturated compacted bentonite with density 2.12 $g \cdot cm^{-3}$.

2.5.2 Experimental set up.

Bentonite in equilibrium with air at ambient temperature and humidity was compacted to a density of 1.8 $g \cdot cm^{-3}$ and thereafter equilibrated with synthetic ground water in a swelling pressure oedometer. The water was deoxygenated by purging with Ar (AGA SR-quality) containing < 1 ppm O_2 during the equilibration. During the water saturation process the density of the bentonite increased to 2.12 $g \cdot cm^{-3}$.

After water saturation, the oedometer was opened in a glove box with Ar-atmosphere and the ^{241}Am radiation source mounted as shown in Figure 1.

2.5.3 Radiolysis experiment.

The following analysis were carried out:

a) Synthetic ground water was flushed through the filterstone and the solution analyzed for H_2O_2 diffused through the bentonite at different times after the onset of irradiation. The H_2O_2 analyses were carried out using a chemiluminescence method (Kok et al 1978). 100 μl solution was added to 2 cm^3

reagent containing $2 \cdot 10^{-5}$ mol·dm^{-3} luminol (5 amino-2,3 dihydro-1.4 phtalazinedione) and $2 \cdot 10^{-5}$ mol·dm^{-3} Cu^{2+} and the light emission was measured with a LKB-1250 luminometer.

b) Synthetic ground water, initially containing $2 \cdot 10^{-6}$ mol·dm^{-3} Fe^{2+} was flushed through the filterstone and the solution analyzed for Fe^{2+} at different times after the change of flushing solution.

3. EXPERIMENTAL RESULTS.

3.1 Fe-leaching.

The concentration of Fe^{2+} in suspensions containing <100 cm^3 water per gram dry clay has been shown to be constant, $8.6 \cdot 10^{-6}$ mol·dm^{-3} (Eriksen and Jacobsson, 1983) indicating a solubility limited dissolution from the clay. The Fe^{3+} concentration was found to be below the detection limit. On increasing the water volume to more than 1000 cm^3 per gram dry Mx-80 a total Fe^{3+} content of 0.72 mg Fe^{2+}/g dry clay was obtained (Figure 2).

3.2 Gamma irradiation of washed bentonite.

The total radiolytic production of the reducing radicals e^-_{aq} and $CO_2^{\cdot -}$ was 6-8 mmol/g dry clay. No dissolution of Fe^{2+}, Fe^{3+} was observed after the irradiation.

3.3 Gamma irradiation of untreated clay.

On irradiation of a N_2O saturated suspension of Mx-80 the total amount of Fe^{2+} going into solution decreased with increasing dose. The experimental data are plotted in Fig. 2.

3.4 α-irradiation.

The break through curve of H_2O_2 diffusing out of the thin layer (17 m) of irradiated water saturated compacted bentonite is shown in Figure 3. Assuming steady state after 36 hours irradiation the H_2O_2 production is $2.66 \cdot 10^{-13}$ $mol \cdot sec^{-1}$.

The Fe^{2+} consumption by oxidizing species diffusing out of the irradiated volume, following the addition of Fe^{2+} to the flushing solution, is plotted in Figure 4. From the slope we obtain the Fe^{2+} consumption $1.72 \cdot 10^{-12}$ $mol \cdot sec^{-1}$. Assuming the oxidation of Fe^{2+} to be caused by the long lived products O_2 and H_2O_2, the following reactions may be written

$$Fe^{2+} + 1/2\ H_2O_2\ (aq) + H^+ \rightarrow Fe^{3+} + H_2O$$

$$Fe^{2+} + 1/4\ O_2\ (aq) + H^+ \rightarrow Fe^{3+} + 1/2\ H_2O$$

and the O_2 production is thus

$$1/4\ (1.72 \cdot 10^{-12} - 2 \cdot 2.66 \cdot 10^{-13}) =$$

$$= 2.97 \cdot 10^{-13}\ mol \cdot sec^{-1}$$

3.5 Calculation of radiolytic yields.

The watersaturated bentonite is a two phase system and the calculation of G-values are based on the following assumption. The water/dry clay ratio of bentonite in equilibrium with air at ambient temperature is 0.08-0.11 (Pusch et al 1985). On water saturation the compacted bentonite density increases from 1.8 to 2.12 $g \cdot cm^{-3}$. The overall watercontent is thus taken to be 23+/-2 per cent.

The α-energy deposition in the water phase is thus $(1.9+/-0.15) \cdot 10^{13}$ $eV \cdot s^{-1}$. Energy transfer from bentonite is, as in previous work (Eriksen et al, 1987) assumed to increase the dose with a factor 1.3 (Christensen 1966). G-values calculated from the plots in Figs 2-4 are summarized in Table 2.

Table 2

Irradiated system	Radiation	$G(H_2O_2)$	$G(-Fe^{2+})$	$G(O_2)$
washed Mx-80, dist H_2O, 10 mM HCO_2^-	γ	nm*	0	
untreated Mx-80, 1 g clay 3 $g \cdot N_2O$-sat. synth. ground water	γ	nm	5.1±0.5	nm

compacted water saturated Mx-80 (ρ=2.12 g·cm^{-3}) synthetic ground water	α	0.67±0.05	3.9±0.3	0.64±0.07
synthetic ground water (Eriksen et al, 1988)	α	0.69±0.03	3±0.3	0.4±0.07

* nm = not measured

4. DISCUSSION.

The Fe-content of Mx-80 is according to Müller-Vonmoos and Kahr 1983, taking pyrite into account, (see Table 3) 2.4 mg·g^{-1} Fe^{2+} and 25 mg·g^{-1} Fe^{3+}. The Fe^{2+} going into solution in synthetic ground water was in this work found to be 0.72 mg·g^{-1}.

The γ-irradiation experiments clearly indicates that Fe^{3+} is not on a short time scale accessable for reactions with radiolytically produced reducing radicals in the pore water. Whereas the primary radiolytic yields in G-units are well known for fast electrons and γ-radiation this is not the case for α-radiation (Bibler 1974, Burns and Simic 1981, Cohen 1969). "Best estimate values" given Table 3 are used when discussing the experimental results.

Table 3

Primary G-values for radiolysis of water.

G	G, events/100 eV (γ-radiation)	(α-radiation)[1]
H_2O	4.08	2.71
H_2	0.45	1.3
$H^{\cdot}$	0.55	0.21
e^-_{aq}	2.63	0.06
H_2O_2	0.68	0.985
$HO_2^{\cdot}$	0.008	0.22
OH	2.72	0.24

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[1] Christensen and Bjergbakke (1982)

[2] Spinks and Wood (1976).

In gamma-irradiated N_2O saturated aqueous solutions the hydrated electron e^-_{aq} is converted to a $\dot{}$OH radical. The reactions between Fe^{2+} and H_2O_2, O_2 are slow and the H_2O_2, O_2 concentrations will most probably build up and be high enough to scavenge the hydrogen atoms. The expected Fe^{2+} consumption $G(-Fe^{2+})$ is thus given by

$$G(-Fe^{2+}) = 2 \cdot G(H_2O_2) + G(e^-_{aq}) + G(OH)$$
$$-G(H^{\cdot}) = 6.16$$

The experimentally determined Fe^{2+} consumption corresponds to $G(-Fe^{2+}) = (5.1+/-0.5)$ i.e. in fair agreement with consumption calculated using primary G-values. In earlier calculations of $G(H_2O_2)$ in α-irradiated synthetic ground water (Eriksen et al, 1988) using the computer program CHEMSIMUL we found poor agreement between calculated and experimental $G(H_2O_2)$ values. The experimentally $G(H_2O_2) = 0.69+/-0.03$ measured in synthetic ground water is, however, in very good agreement with $G(H_2O_2) = 0.67+/-0.05$ obtained in this work.

The total G-value for oxidizing species produced on α-radiolysis may be calculated if it is assumed that e^-_{aq} and $H\cdot$ react with H_2O_2/O_2 in the irradiated water. The expected $G(-Fe^{2+})$ is thus given by

$$G(-Fe^{2+}) = 2\cdot G(H_2O_2) + G(OH) + 3\cdot G(HO_2\cdot) \\ -G(e^-_{aq}) - G(H) = 2.6$$

The experimental consumption is somewhat higher, corresponding to $G(-Fe^{2+}) = 3.9$, which may be partly due to residual oxygen present in the flushing solution after Ar-purging.

5. CONCLUSIONS.

The formation of an "expanding" volume containing oxidants formed on α-radiolysis of washed compacted watersaturated bentonite has been demonstrated. The G-value for H_2O_2 productions in the pore water is in agreement with the G-value obtained in synthetic ground water. The overall Fe^{2+} consumption of oxidants diffusing through the irradiated bentonite is in fair agreement with the oxidative-capacity calculated using primary G-values. Only a fraction of the total Fe-content of Mx-80 is accessible for reactions with radiolytically formed oxidative and reducing species.

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FIGURE LEGENDS.

Fig. 1 Schematic of α-irradiation cell and flushing system.
Bentonite: $\rho = 2.1\ g{\cdot}cm^{-3}$.

Fig. 2 Effect of γ-radiolysis on dissolved Fe(II).

Fig. 3 Accumulation of H_2O_2 diffusing through the α-irradiated 8 mm thick water saturated compacted Mx-80 disc.
($\rho = 2.12\ g{\cdot}cm^{-3}$).

Fig. 4 Fe(II)-consumption by oxidants diffusing through the α-irradiated 8 mm thick water saturated compacted bentonite disc.
($\rho = 2.1\ g{\cdot}cm^{-3}$).

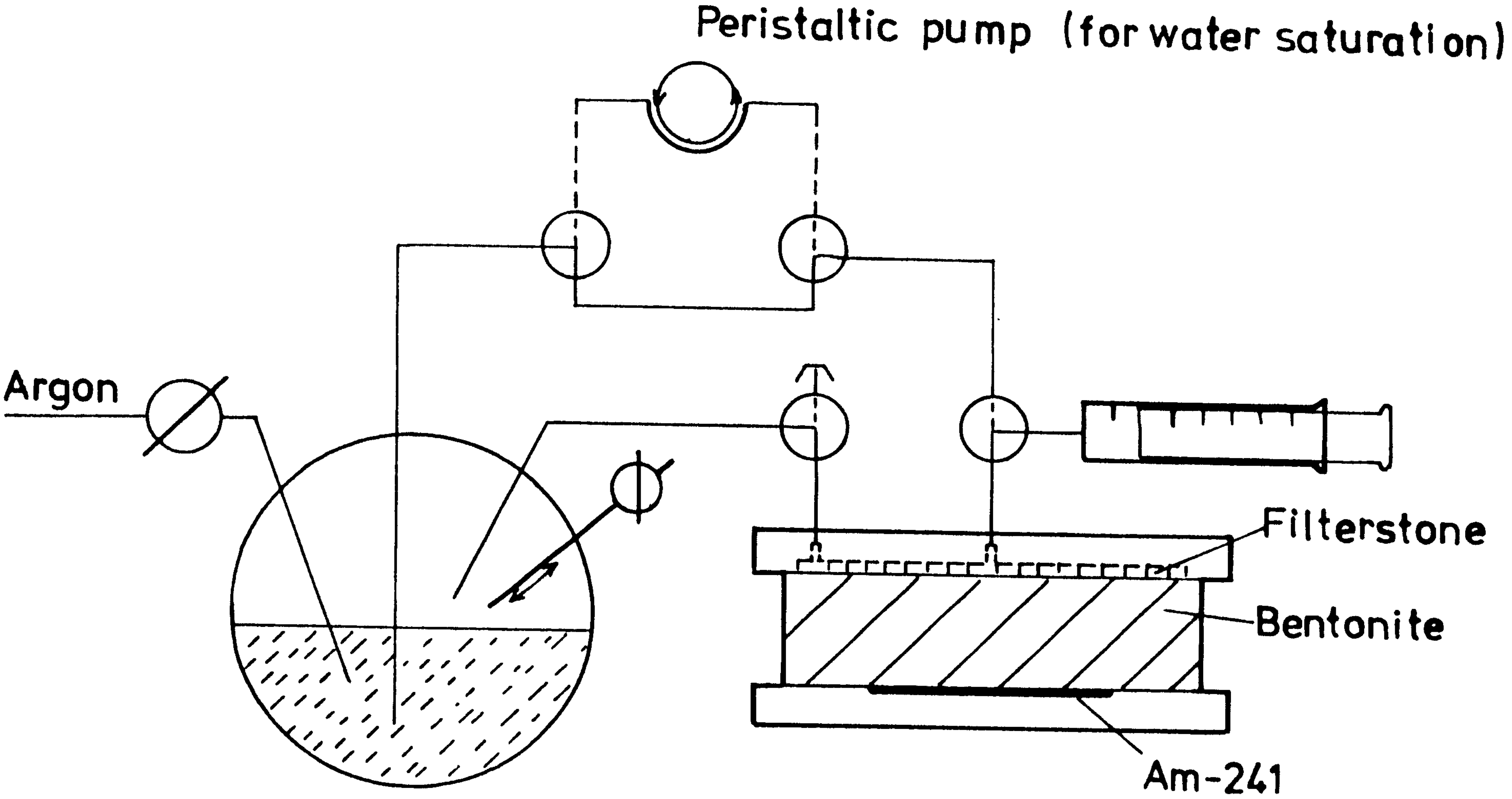

Fig. 1 Schematic of irradiation cell (oedometer)

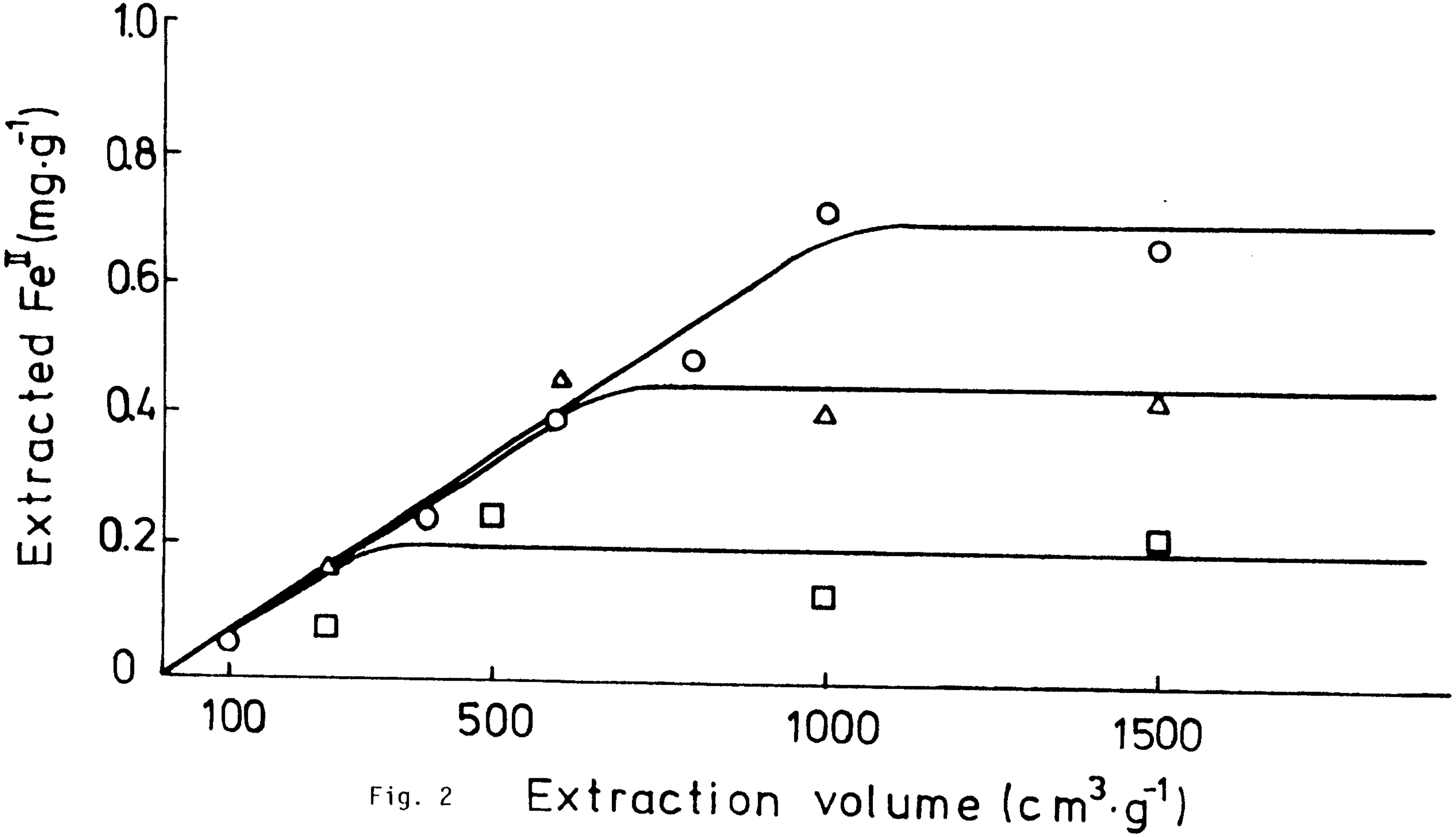
1.0
0.8
0.6
0.4
0.2
0
Extracted Fe^{II} ($mg \cdot g^{-1}$)
100
500
1000
1500
Extraction volume ($cm^3 \cdot g^{-1}$)

Fig. 2

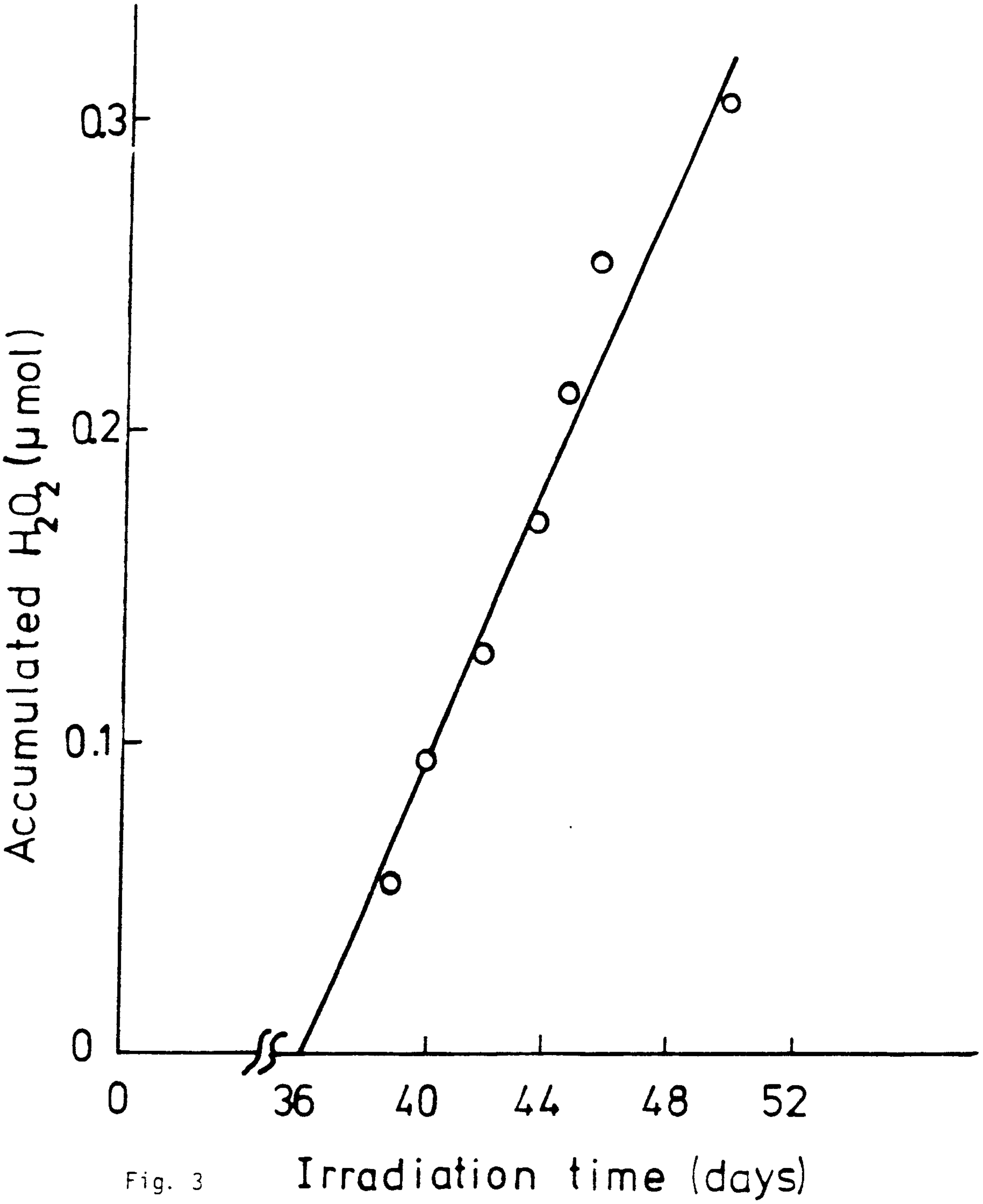
Accumulated H_2O_2 (μmol)
0
0.1
0.2
0.3
0
36
40
44
48
52
Irradiation time (days)

Fig. 3

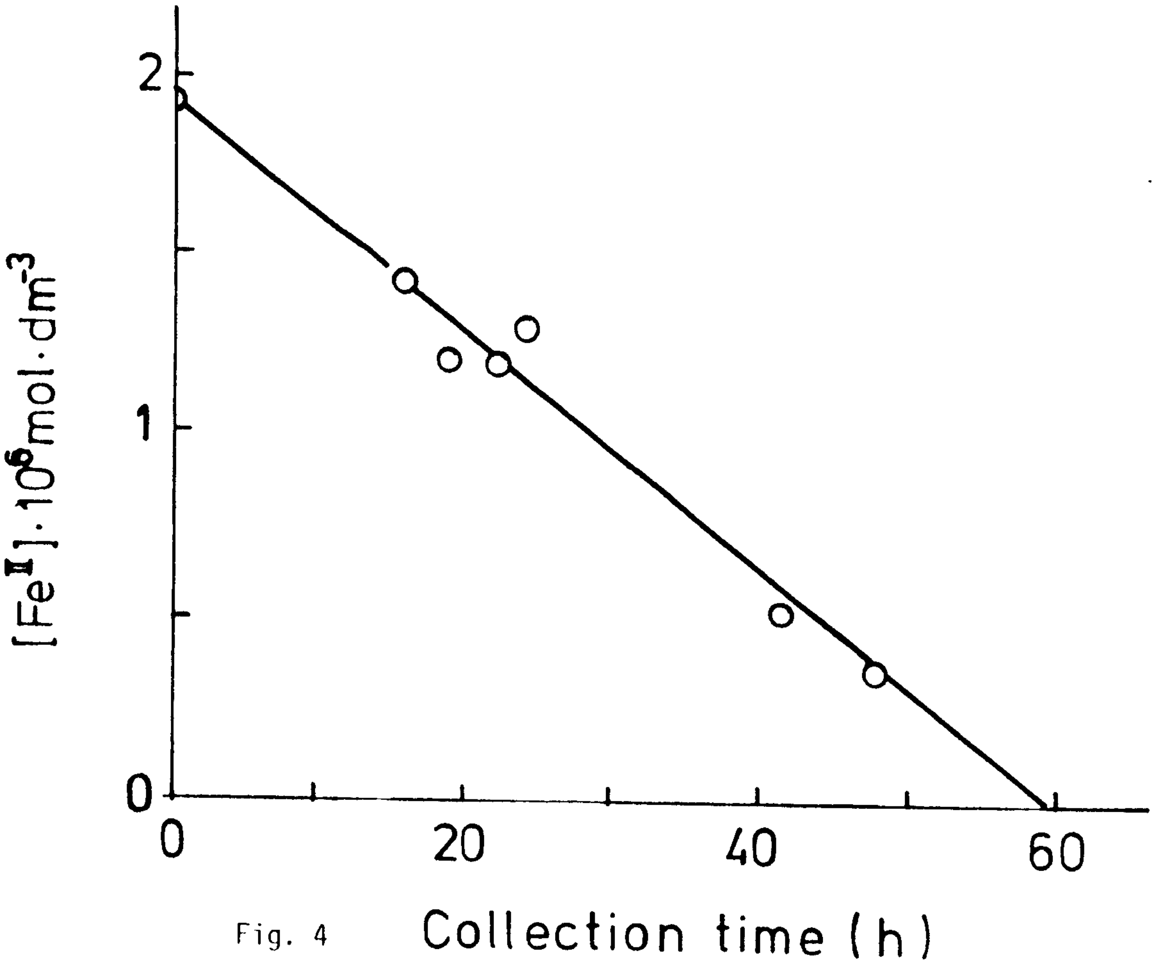

Fig. 4

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[1] Chalmers University of Technology
[2] University of Linköping
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[1] Royal Institute of Technology, Stockholm, Sweden
[2] Lawrence Berkeley Laboratory, Berkeley, CA, USA
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Linköping University, Linköping
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[1] Royal Institute of Technology, Stockholm
[2] Israel Institute of Technology, Haifa
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[1] Swedish Geological Company, Uppsala
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T E Eriksen[1], P Ndalamba[1], I Grenthe[2]
[1] The Royal Institute of Technology, Stockholm
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Department of inorganic chemistry
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I Puigdomenech[1], J Bruno[2]
[1] Studsvik Nuclear, Nyköping
Environmental Services
[2] Royal Institute of Technology, Stockholm
Department of Inorganic Chemistry
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TR 88-22

Radiolysis of ground water: influence of carbonate and chloride on the hydrogen peroxide production

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Seismological Department, Uppsala University,
Box 12019, S-750 12 UPPSALA
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