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**THE INFLUENCE OF MOISTURE CONTENT OF
SAND ON LEACH RATES FROM CEMENT AND
BITUMEN RADIOACTIVE WASTE FORMS IN
A SHALLOW GROUND REPOSITORY**

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LIXIVIATION DES DÉCHETS RADIOACTIFS CONDITIONNÉS AU
CIMENT ET BITUME DANS UN DÉPÔT SOUTERRAIN PEU PROFOND**

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Published in the Proceedings of the Spectrum '92 Conference, held in Boise, Idaho, 1992 August 23-27.

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AECL Research

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by

Z. Lovasic^a, J. Torok^b, and L.P. Buckley^b

*(Published in the Proceedings of the Spectrum '92 Conference,
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RÉSUMÉ

Les taux de lixiviation de déchets conditionnés au ciment et au bitume en contact avec du sable ont été déterminés à différentes teneurs en eau jusqu'à la saturation inclusivement. Un déchet conditionné de forme cylindrique occupait l'une des extrémités de la colonne, alors que le reste était rempli de sable tassé. Les profils de concentration axiale des radionucléides ont été déterminés plusieurs fois dans la colonne au cours de l'essai à l'aide d'un système automatisé comprenant un spectromètre gamma et un banc optique. On a déterminé la migration du ⁸⁵Sr, ¹³⁷Cs et ⁶⁰Co d'après les profils de concentration en fonction du temps. Le coefficient de diffusion du ¹³⁷Cs dans le ciment a varié davantage que prévu en fonction de la variation de la teneur en eau du sable.

La lixiviation des déchets conditionnés au bitume est complexe et sa reproductibilité (répétabilité) est mauvaise. Lorsque la teneur en eau du sable était inférieure à 0,067 mL/mL, aucune lixiviation n'a été déterminée avec les déchets conditionnés au bitume, sauf pour un spécimen. On n'a pu établir aucune relation évidente entre le taux de lixiviation et la teneur en eau du sable à une teneur supérieure à 0,18 mL/mL.

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ABSTRACT

Leaching rates from cement and bitumen-conditioned waste in contact with sand were determined with different water content of the sand up to and including saturation. A cylindrical waste form occupied one end of a column, with sand packed in the remainder. Axial radionuclide concentration profiles in the columns were determined several times during the experiment using an automated system consisting of a gamma spectrometer and an optical bench. The migration of ⁸⁵Sr, ¹³⁷Cs and ⁶⁰Co was determined from concentration profiles with time. The diffusion coefficient for ¹³⁷Cs in cement varied more than expected with the change of moisture content of sand.

The leaching of bitumen waste forms is complex, and has poor reproducibility. When the water content of the sand was below 0.067 mL/mL, no leaching was determined from bitumen waste forms in all of the specimens except one. No clear relationship could be established between leach rate and the water content of sand above 0.18 mL/mL water content.

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TABLE OF CONTENTS

	<u>Page</u>
1. INTRODUCTION	1
2. LITERATURE REVIEW	2
3. THEORY	2
3.1 Diffusion	2
3.2 Leaching from the Waste Form	3
4. EXPERIMENTAL PROCEDURE	4
5. DIFFUSION COEFFICIENT CALCULATIONS	5
5.1 Use of the Scan Difference Curves	5
5.2 Fit of the Experimental Leach Results to Equations 4 and 5	6
6. RESULTS	6
6.1 Diffusion of Radioisotopes as Function of the Moisture Content in Sand ..	7
6.2 Bitumen Leaching	7
6.3 Cement Leaching	8
7. DISCUSSION	8
8. SUMMARY AND CONCLUSIONS	10
9. REFERENCES	11

1. INTRODUCTION

Atomic Energy of Canada Limited (AECL) is planning to construct the IRUS (Intrusion Resistant Underground Structure) low- and intermediate-level nuclear waste repository. It is a shallow ground repository with sand as backfill and in a sandy environment. The safety assessment of this repository requires data about the migration of radionuclides, and that became one of the objectives of this investigation.

To achieve reliable predictions of radionuclide migration from a waste disposal site to the biosphere and the impact of the radionuclides on man, one should understand the mechanism of the dominant migration processes and determine the values of the transport parameters. The work reported here deals with the first step in a series of supposed events: the release (leaching) of radioactivity from the waste form to the surrounding soil.

Traditionally, leach rates are determined in a water saturated environment. Such an approach has been standardized by the IAEA, ANS and other regulatory bodies.

However, the above approach has a major disadvantage: it may not be realistic for the near surface disposal, well above the water table. By testing the leaching properties of two dominant waste forms, cement and bitumen, in contact with sand of various water content, a more realistic data base for leaching can be generated. The radionuclides: ^{85}Sr , ^{137}Cs and ^{60}Co were incorporated into the waste forms and their migration was monitored during the experiments. The isotopes were selected because they constitute 80% of the radioactivity in the low- and intermediate-level radioactive waste. They are gamma emitters and thus facilitate the nondestructive monitoring of their migration.

Sand is an ideal backfill material for the repository. Because of its relatively large particle size, it is expected to have a low water content in the repository environment. The low water content in turn results in low diffusion rates of water soluble radionuclides. The buffer material underneath the repository has a high retardation factor for many of the radionuclides, resulting in a significant reduction in the migration of those radionuclides.

Two types of experiments were performed in this program:

- i) the measurement of the diffusion of radionuclides within the same media: sand, or cement; and
- ii) leaching of the waste forms, where the waste forms were in contact with initially uncontaminated wet sand. The moisture content of sand was a controlled parameter.

2. LITERATURE REVIEW

Leaching rates are usually measured by IAEA recommended methods, where the specimens are immersed in water. Oblath (1) was the first to evaluate the leaching properties of portland cement in laboratory and field-scale experiments in saturated and in unsaturated environments. While most experimental methods are destructive, and provide only one set of data per experiment, the gamma scanning technique applied in this work is nondestructive. It has been used in several other applications.

3. THEORY

The diffusion of isotopes in waste form, in the adjacent soil and mass transfer at the interface, determines the leach rate from waste to soil. In this section diffusion in a single media is discussed first, followed by an analysis of the more complex leaching process.

3.1 Diffusion

The diffusion of radioisotopes in soil (sand in this case) is governed by Fick's law, but is more complex because of the sorption/desorption process of ionic species which results in the reduced migration rate (retardation) of the isotopes.

The migration of isotopes in soil in one dimension, without bulk flow, can be represented by the differential equation:

$$\frac{\delta C}{\delta t} = D \frac{\delta^2 C}{\delta x^2} \quad (1)$$

The solution of the differential equation in a semi-infinite media with the following boundary conditions:

i) $C = C_0 \quad 0 < x < h$

ii) $C = 0 \quad x > 0$

where:

C = concentration of diffusing component

h = sample length of the high concentration portion of the sample in the direction parallel to diffusion flux (cm)

x = distance vector parallel to "h",

can be represented by (6):

$$\frac{C}{C_o} = 0.5 \left(\operatorname{erf} \left(\frac{h-x}{\sqrt{4D_s t}} \right) + \operatorname{erf} \left(\frac{h+x}{\sqrt{4D_s t}} \right) \right) \quad (2)$$

where the diffusion coefficient in soil (D_s) (cm^2/s) is:

$$D_s = \frac{D_o \tau}{\left(1 + \frac{K_d \sigma}{\theta} \right)} \quad (3)$$

and:

D_o = diffusion coefficient of free ion in water (cm^2/s)

τ = tortuosity factor

K_d = distribution coefficient of the radioisotope
between soil and the solute (cm^3/g)

σ = density of the soil (g/cm^3)

θ = moisture content of soil (cm^3/cm^3)

By repeated gamma scanning of a column a number of axial radionuclide concentration profiles are obtained and from it diffusion coefficients are calculated.

3.2 Leaching from the Waste Form

In the assessment of concrete leaching the coupled diffusion of the critical component in two dissimilar media is measured and modeled. The critical parameters are the diffusion coefficient in the waste form (D_1), in sand (D_2) and the interfacial resistance. The last parameter is not included in the mathematical analysis. The related mathematics is described by Crank (2) and the solution to the diffusion equation is:

$$C_1 = \frac{C_o}{1+k \left(\frac{D_2}{D_1} \right)^{\frac{1}{2}}} \left(1+k \left(\frac{D_2}{D_1} \right)^{\frac{1}{2}} \operatorname{erf} \frac{x}{\sqrt{4D_1 t}} \right) \quad (4)$$

and

$$C_2 = \frac{kC_o}{1+k \left(\frac{D_2}{D_1}\right)^{\frac{1}{2}}} \operatorname{erfc} \frac{|x|}{\sqrt{4D_1t}} \quad (5)$$

where:

C_o = initial concentration in the waste form;

C_1 = concentration in material 1 (sand) and distance x from the waste form-sand interface, where $x > 0$;

C_2 = concentration in material 2 (waste form) and distance x from the waste form-sand interface, where $x < 0$;

D_1, D_2 = diffusion coefficients in materials 1 and 2, respectively; and

k = ratio of equilibrium concentrations in material 1 and 2 which is equal to the ratio of distribution coefficients in materials 1 and 2.

In most leach experiments only the fraction of radioactivity released as a function of leach time is determined. From such data it is impossible to realistically evaluate the values of D_1 and D_2 , the diffusion coefficients in each media. The advantage of using scanning gamma spectrometry is that the concentration profile can be monitored during the experiment, and what is more important, the individual diffusion coefficients can be determined.

4. EXPERIMENTAL PROCEDURE

Cylindrical specimens consisting of a waste form adjacent to and in contact with sand were prepared. The specimens were sealed to avoid moisture loss.

Gamma rays emanating from the column were collimated through a 5 mm wide slit. A high purity germanium gamma-ray detector connected to a personal computer analyzer system was used for gamma spectrometry. A stepping motor, controlled by the same computer, moved the column in increments of 0.1 cm. A full gamma spectra was generated in each interval. The data were compiled for each isotope by obtaining gamma count rate as a function of axial distance.

5. DIFFUSION COEFFICIENT CALCULATIONS

All of the calculations were performed with the radionuclide concentration normalized; i.e. the reduced concentrations (C_r) were calculated:

$$C_r = C / C_0$$

The use of reduced concentrations eliminated the need for absolute calibration of the gamma spectrometer.

The results from the gamma scanning of the diffusion cells were corrected for the following two interferences: changes in detector calibration and gamma shine from the neighbouring sections of the specimen.

The results were compensated for possible calibration changes during periods between scanning by normalizing the integral number of counts from each scanning to the first scanning curve.

For the shine interference correction, two approaches were used: application of the deconvolution program and use of the scanning difference curves discussed later.

The experimental results were fit to the diffusion equations using the least squares method.

5.1 Use of the Scan Difference Curves

Immediately after its preparation, each specimen was scanned and in subsequent calculations we assumed that no diffusion had taken place prior to this initial scan. At each location the reduced concentration at time=0 was subtracted from the reduced concentration at time=t, and the concentration difference was plotted as a function of axial location. A typical difference curve is illustrated in Figure 1. The collection of this data set along the column for a specific diffusion time is denoted as the "difference curve". The area under the difference curve is a measure of the quantity of the diffusing species that has crossed the interface. Cumulative fraction released (CFR) was determined by dividing the average of the

- i) total activity lost from the waste form (area under first, negative part of the difference curve); and
- ii) the gain in activity in the sand

by the initial activity. The data were also corrected for the difference in self adsorption of the two materials. The relationship between CFR and the effective

diffusion coefficient was evaluated from Equations 2, 4 and 5. The CFR is related to diffusion time and diffusion coefficient by:

$$CFR = Q * t^{\frac{1}{2}} * D^{\frac{1}{2}} \quad (6)$$

Where Q is a constant

Differentiating with respect to $t^{1/2}$

$$\frac{\delta CFR}{\delta t^{1/2}} = Q * D^{1/2} \quad (7)$$

By plotting CFR as a function of $t^{1/2}$, from Equation 7, the slope of the line is proportional to $D^{1/2}$.

Calculations based on cumulative fraction released using Equation 7 were performed for ^{137}Cs leaching from cement. Results are presented in Figure 2.

5.2 Fit of the Experimental Leach Results to Equations 4 and 5

While this approach provides an estimate of the individual diffusion coefficients, three parameters must be selected to generate the best fit. These parameters are the diffusion coefficient in sand and the waste form and the ratio of the distribution coefficient in the two media (k).

The influence of shine from neighbouring column sections is maximum when the concentration gradients are the highest, a condition characteristic of short diffusion times. As diffusion proceeds, the concentration gradients decline and the influence of shine is reduced. At infinite time concentration gradients vanish along with shine contribution. By plotting the diffusion coefficients as a function of reciprocal diffusion time, and extrapolating the data to $1/t = 0$, we estimated the diffusion coefficient at infinite time. When no shine correction was applied, the diffusion coefficient declined with time. When applying the CFR approach to the calculation of D, the value of D increased with time.

6. RESULTS

Diffusion of radionuclides in sand is presented first, followed by leaching results for bitumen and cement specimens.

6.1 Diffusion of Radioisotopes as Function of the Moisture Content in Sand

Diffusion coefficients were calculated from four consecutive scans of each sample by least squares fitting of the results to Equation 2, followed by extrapolation to infinite diffusion time. Papendick and Campbell (3) proposed the following equation for the calculation of molecular diffusion of an ionized species in soil as a function of soil moisture content :

$$D = D_0 a \theta^b \quad (8)$$

The coefficient "a" accounts for tortuosity and is given as approximately 2.8 by the authors. The value of 3.0 is given for "b".

Taking logarithms of each side of Equation 8 gives:

$$\text{Log } D_m = \text{Log } (D_0 a) + b \text{ Log } \theta \quad (9)$$

Thus a log-log plot of D as a function of θ should be a straight line with a slope equal to b. The data fit well to the general form of Papendick's and Campbell's equation. However the parameters were different from values obtained by Papendick and Campbell; the value of b was close to 1.

Contrary to the results obtained in the leach experiments with the sand/cement system, where significant migration of ^{137}Cs was obtained; the diffusion of ^{137}Cs in sand was so low that it could not be measured. The most plausible explanation for this apparent contradiction is the large difference in the pH and ionic strength and especially potassium ion concentration of the cement pore water compared to the water used in the sand diffusion experiments. The high ionic strength and the relatively high concentration of potassium ions released from the cement pore water reduces the distribution coefficient of ^{137}Cs resulting in higher diffusion coefficients (4).

6.2 Bitumen Leaching

Bitumen specimens with 38 wt% salt content were contacted with sand. Cumulative fractions of radioactivity release (CFR) were measured. Migration of all isotopes was observed at higher water concentrations in sand (0.18 and 0.35 mL/mL water content), but virtually no detectable migration of any of the isotopes took place at water concentrations at or below 0.067 mL/mL.

Differences between the CFR of duplicate samples prepared from different batches of concentrates are partially due to difficulties in the preparation of bitumen samples with the exact solids content. It was also difficult to duplicate sample homogeneity.

These difficulties contributed to some of the discrepancies in the results, where higher releases of radionuclides were found with the samples that were in contact with sand containing 0.18 mL/mL water than samples in contact with nearly saturated sand (containing 0.35 mL/mL water). These findings are in agreement with other reports (5) where it was concluded that leaching from bitumen waste forms containing soluble solids (like sodium tetraborate) is non-Fickian.

6.3 Cement Leaching

Only the migration of ^{137}Cs could be measured in the leach experiments, because the diffusivity of the other two isotopes was too low to be measurable.

Results of the leaching experiments were fitted to Equations 4 and 5. There are three adjustable parameters in this calculation: the two diffusion coefficients and the ratio of the distribution coefficients $k = K_d(\text{sand}) / K_d(\text{cement})$. The value of k is, however, very much system dependent and will change with water content and also to a lesser extent with diffusion time. The mobile ions, such as Ca^{++} , Na^+ , K^+ , and Mg^{++} will diffuse out of the cement along with ^{137}Cs . The concentration of these ions, but especially the concentration of K^+ , will lower the value of $K_d(\text{sand})$ (4). At low water content the diffusion coefficient in sand is low and the released ions are concentrated in a region close to the cement/sand interface, resulting in low values for $K_d(\text{sand})$. With increased water content the released ions are spread over a larger volume of sand, resulting in lower ion concentrations and hence increased values for $K_d(\text{sand})$. In Figure 3, the diffusion coefficients, extrapolated to infinite diffusion time, are summarized. Note the much higher dependence of ^{137}Cs diffusion coefficient in sand than ^{137}Cs diffusion coefficient in cement on the water content of sand. In Figure 4, a typical experimental result and the simulation are compared.

7. DISCUSSION

The markedly different leaching behaviour of cement and bitumen waste forms can be attributed to their structural difference.

Cement has an open pore structure much like sand. The major difference between the mass transport behaviour of sand and cement is due to the very different pore size distribution of these two materials. Migration of water and solutes through both media can be described by Fick's laws of diffusion.

Other investigators have measured the diffusion coefficient of ^{137}Cs in cement immersed in water. Their results presented in Table 1 should be comparable to our results obtained with high water content sand. Our results fall in the mid-range of the findings of others.

Table 1: Comparison of the results of ¹³⁷Cs diffusion in Portland cement
From NUREC/CR-5387

Waste Form	Reference	Diffusion Coefficient cm ² /s x 10 ³
Portland III	Walter, 1988	7.2
Portland III	Colombo, 1986	5.9
Portland	Matsuzuru, 1977	0.2 - 2
Portland	Atkinson, 1986	1.4
Portland III	This work	4.5

The release of encapsulated species from bitumen waste forms is much more complicated than the leaching of cement waste forms. Briefly, the continuous phase is bitumen, with the waste particle such as salt or other solid material being the discontinuous phase. The initial step in the release mechanism is the slow diffusion of water from the outside through a thin bitumen film and into a salt crystal. The driving force here is a difference in osmotic pressure between the water outside and the brine inside. Another important mechanism is the hydration of the salt crystals, resulting in an increased volume of the waste particle.

The pressure in the salt crystal bursts the bitumen film, and also likely exerts stress on bitumen films separating the salt crystals. The above breakdown continues to propagate into the waste form. Because the speed of this process is so dependent on a multitude of parameters, such as waste loading, particle size and solubility, expansion of the crystals on hydration, uniformity of waste loading in the waste form, etc., the leaching process is inherently irreproducible. With an open connection to wet soil, the salt dissolves and its contents diffuse out of the waste form. The break-up of the bitumen structure progresses from the surfaces into the bulk of the waste form. Bitumen, a non-polar, hydrophobic material, has a low adsorption capacity, so after the breaching of the bitumen structure the major components contributing to retardation of the radioisotopes are insoluble mineral components in the waste.

The important finding of this investigation is not the values of the release rates, but the observation that at water contents between of 0.067 and 0.18 mL/mL in sand, a major, probably step-type change in release takes place. Prior to IRUS roof failure and most of the time afterwards, the water content in the repository will correspond to a

sand water content of 0.018 mL/mL (6). This low water content is well within the range, where we measured no radionuclide release from bitumen waste forms. Further work is required to assess the leaching properties in unsaturated soil, of more representative waste forms, such as reverse osmosis concentrates. The above preliminary results are, however, encouraging and suggest that bitumen waste forms may be more stable in a repository environment than previous results have indicated.

8. SUMMARY AND CONCLUSIONS

The scanning gamma spectroscopy method was developed to monitor the radionuclide leaching in unsaturated soil.

Cesium-137 was a suitable isotope to monitor radionuclide release from cement waste forms. The distribution coefficient of this isotope is low, approximately 0.7 mL/g, and thus it is a useful model isotope for the computation of the release of poorly retarded isotopes such as ^{129}I , ^{36}Cl , etc. The leaching rate of ^{85}Sr and ^{60}Co was so low that no measurable release occurred during 100 days of experiments. The results suggest that the latter isotopes are bound strongly in the cement matrix. The diffusion coefficient of ^{137}Cs , ^{85}Sr , and ^{60}Co in sand drops by more than an order of magnitude when the water content of sand is reduced from saturation to 0.016 mL/mL. Much smaller variations in diffusion coefficients with water content change were resumed than found in the literature (7). Wall effects are most likely contributors to this difference, but wall effects will also play a major role in solute transport in a repository.

Based on the pore size distribution of cement, the change in diffusion coefficient of ^{137}Cs with a change in moisture content of the sand should be very small if any, because the very small pores in cement are expected to be filled with water at all of the moisture conditions used in this program. However, a nearly four fold reduction in the diffusion coefficient was observed at 0.0016 mL/mL sand water content relative to the results obtained when nearly saturated sand was used as the leach medium. We have no explanation for this anomaly and further work would be required to identify the cause of the reduction in the diffusion coefficient.

No measurable leaching of radionuclides was observed from simulated bitumen waste forms containing 38 wt% sodium phosphate when the water content of the sand was 0.067 mL/mL or less. At water contents of 0.18 mL/mL sand and above, leaching did take place, but there was no significant effect of the water content in sand on leach rates. There may well be a threshold of moisture content, below which the leaching rate is extremely low. These preliminary results suggest that bitumen waste forms may be much more resistant to leaching in an unsaturated near surface repository than any previous results indicated. There is a general consensus in waste management literature, that the leaching of bitumen waste forms has poor

reproducibility. The uncertainty inherent in bitumen waste form stability reduces the reliability of modelling for repository performance assessments. The question of the range of uncertainty of bitumen waste measurements in comparison to uncertainties of other waste disposal parameter measurements must also be addressed.

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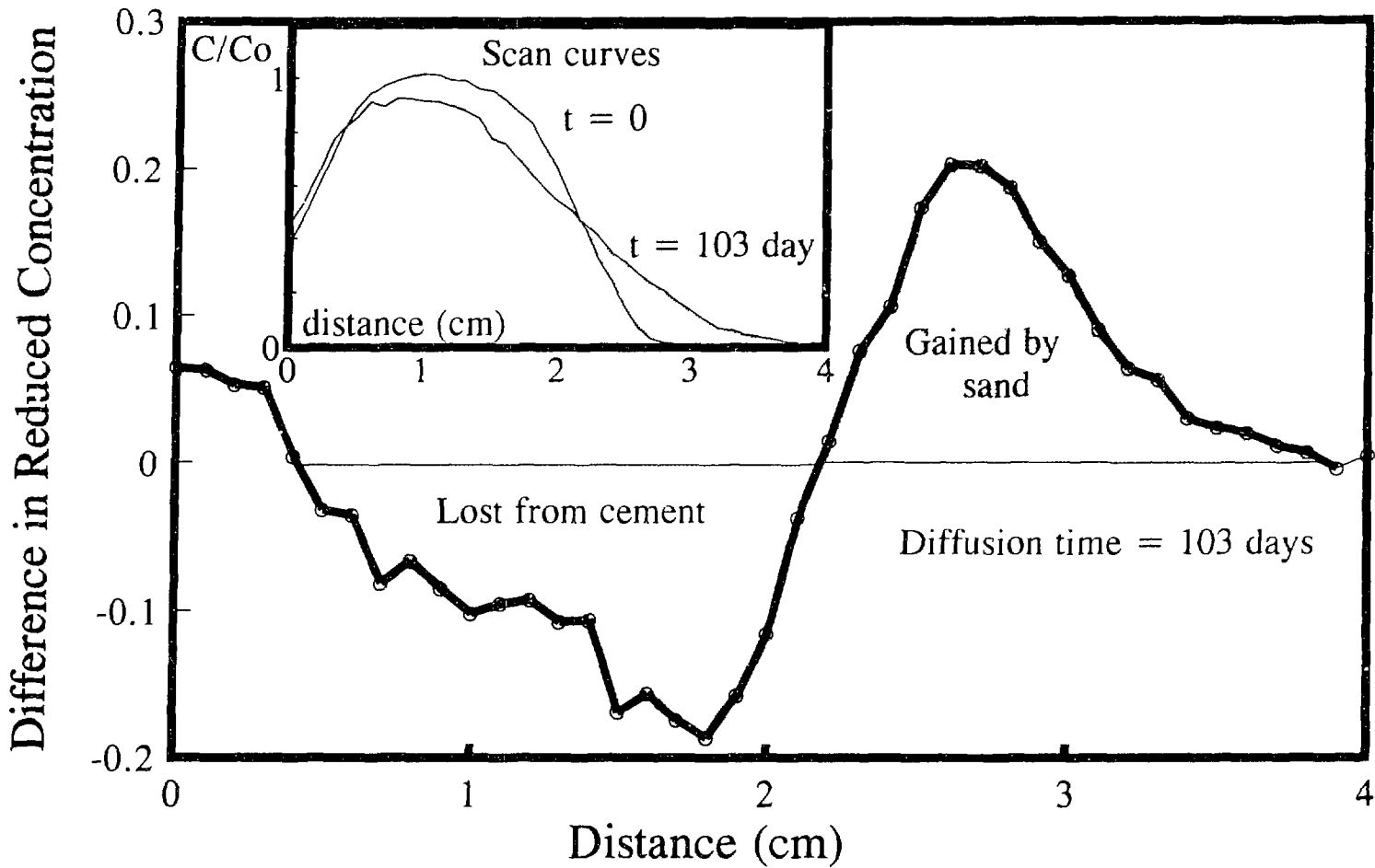


Figure 1
 Scan Difference Curve for Cs-137
 Leaching of cement with sand containing 0.016 mL/mL water

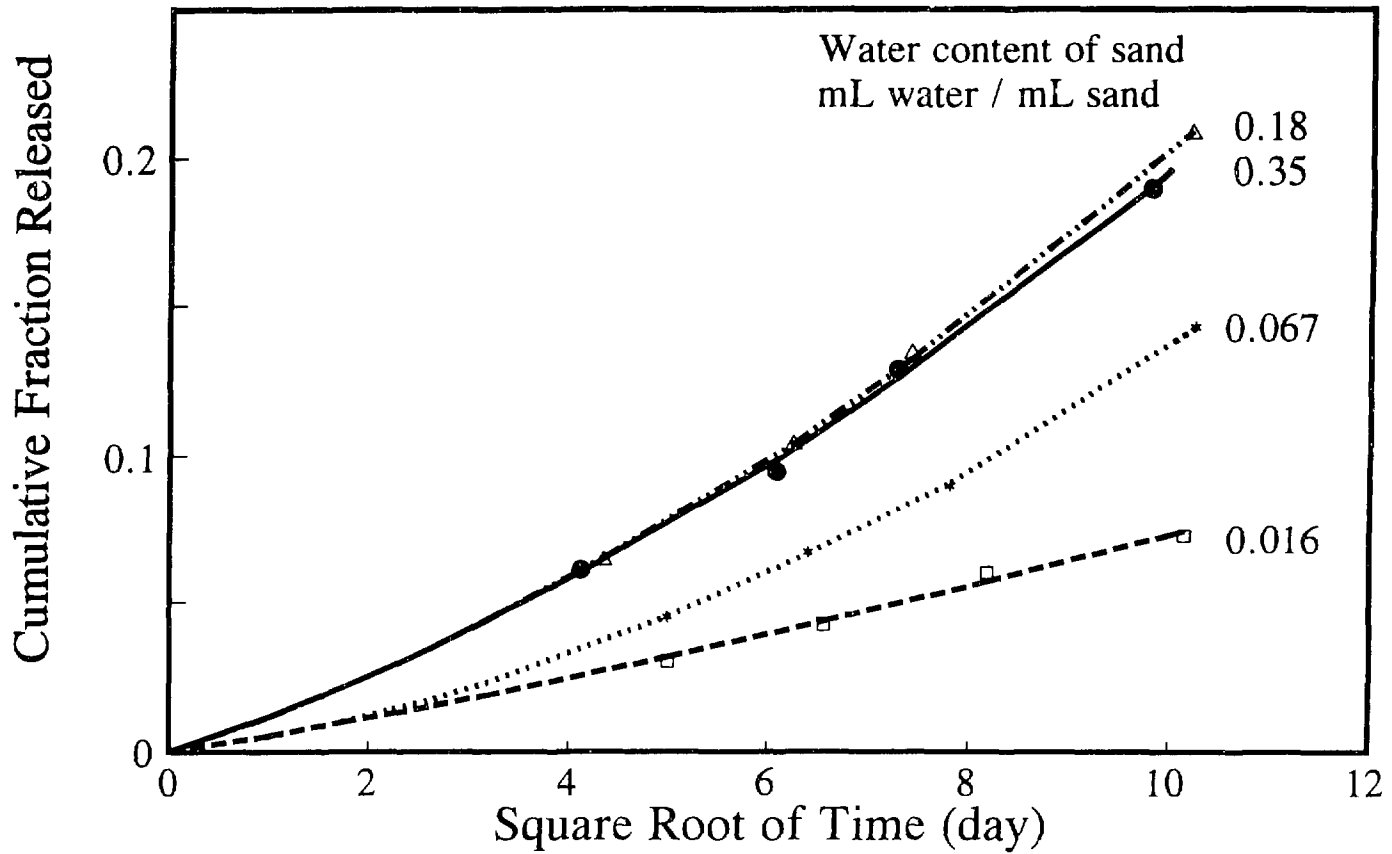


Figure 2
Cumulative Fraction of Cs-137 Released from Cement
As a Function of the Water Content of Sand

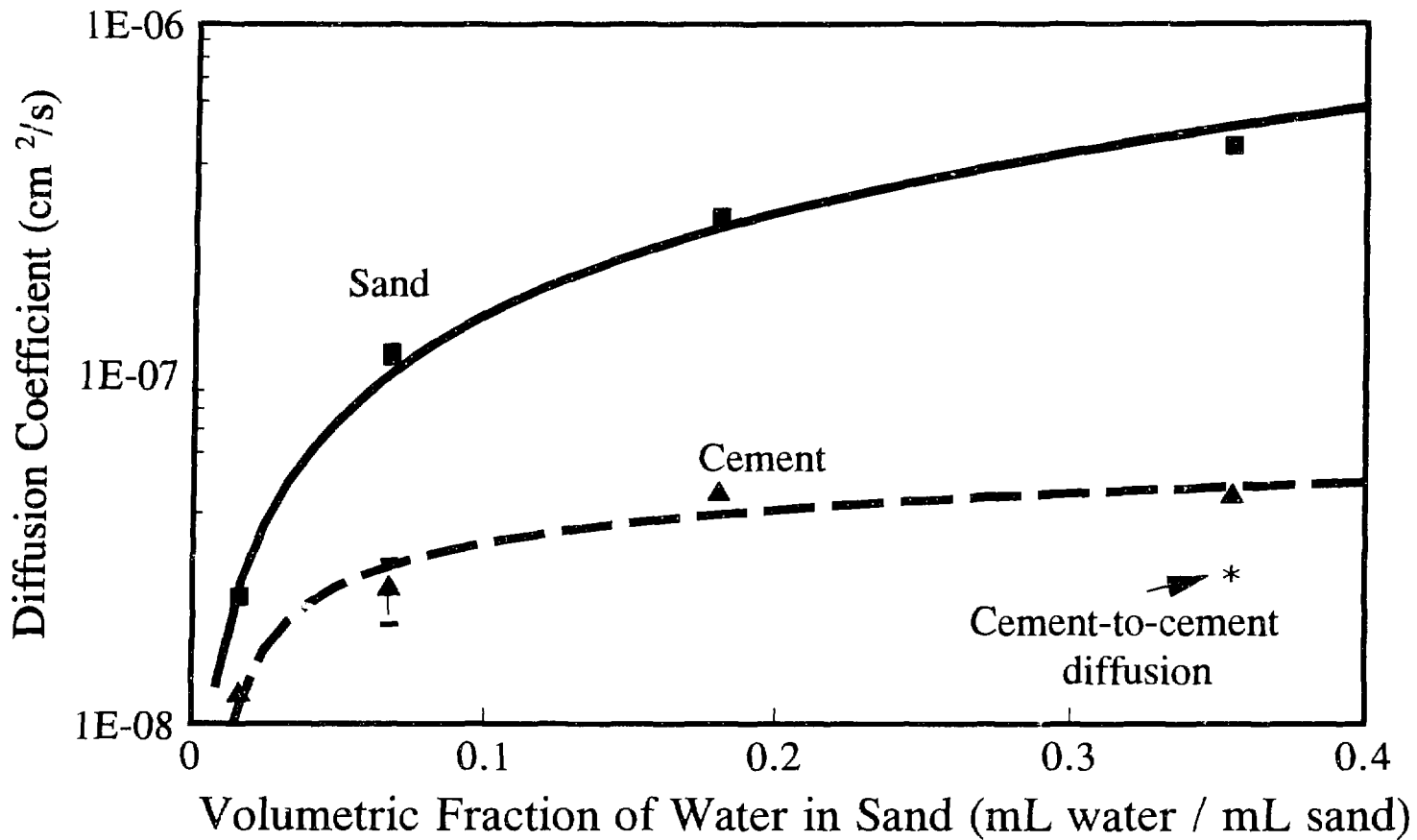


Figure 3
 Cs-137 Diffusion Coefficients Calculated from
 Cement Leach Experiments

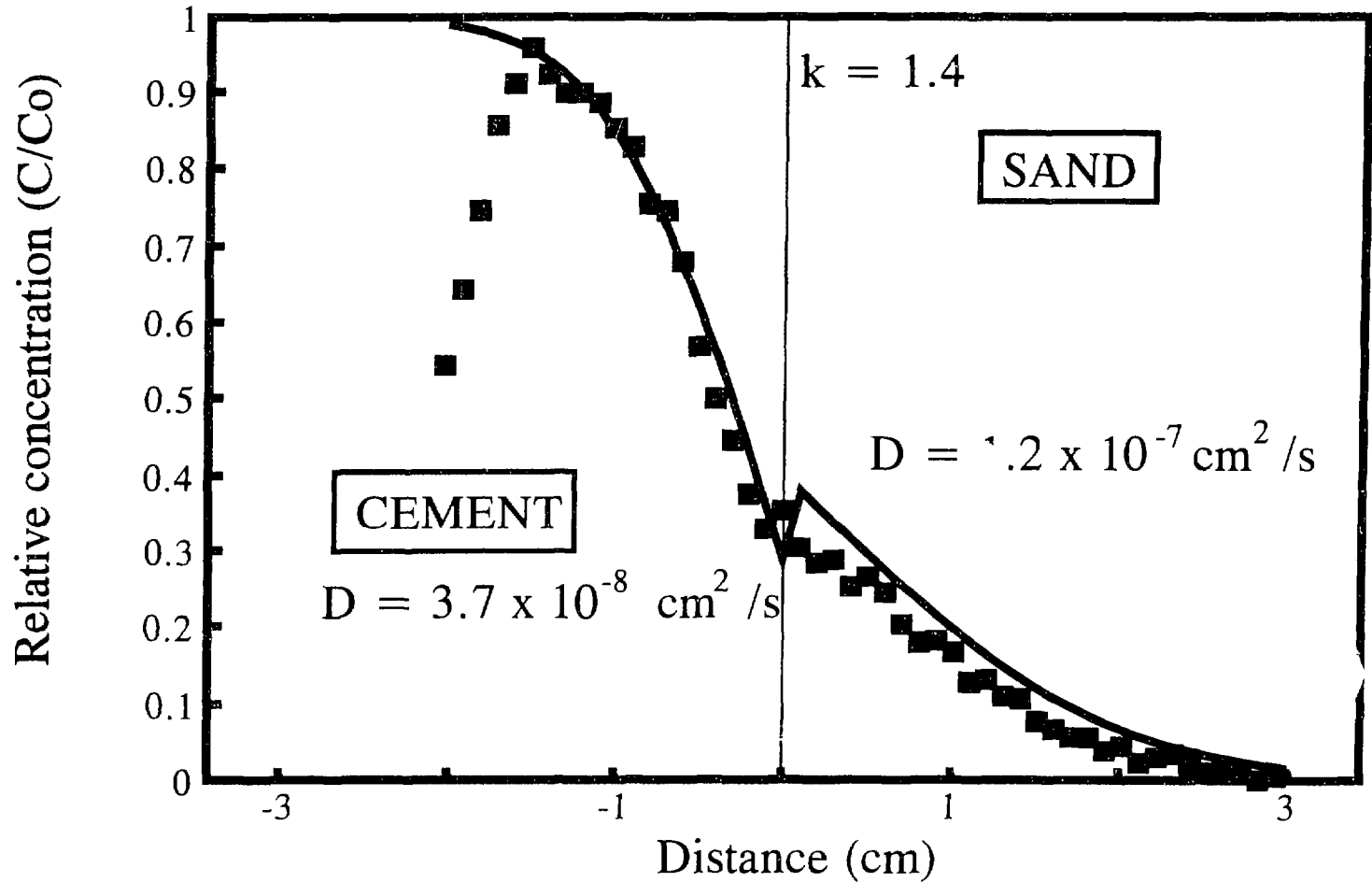


Figure 4

Cs-137 leaching from cement waste form
 Diffusion time = 105 days

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