

## AN EVALUATION OF THE RADIATION RESISTANCE OF HIGH-DENSITY POLYETHYLENE\*

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### ABSTRACT

Mechanical tests following gamma irradiation and creep tests during irradiation have been conducted on high-density polyethylene (HDPE) to provide data to help assess the adequacy of this material for use in high integrity containers (HICs). Two types of HDPE, a highly cross-linked rotationally molded material and a non-cross-linked blow molded material, were used in these tests. Gamma-ray irradiations were performed at several dose rates in environments of air, Barnwell and Hanford backfill soils, and ion-exchange resins. The results of tensile and bend tests on these materials following irradiation are presented along with results on creep during irradiation.

### INTRODUCTION

High integrity containers (HICs) provide an alternative to solidification for meeting the stability requirements for Class B and C radioactive waste under 10 CFR Part 61 (Licensing Requirements for Land Disposal of Radioactive Waste). The State of South Carolina has licensed HICs for disposal of low-level radioactive waste in the Barnwell, SC, land burial site. High density polyethylene (HDPE) is the material used to fabricate most HICs.

To provide a data base to assist in assessing the adequacy of HDPE for HICs, the U. S. Nuclear Regulatory Commission (NRC) contracted with Brookhaven National Laboratory (BNL) to test the radiation resistance of two types of HDPE, Marlex CL-100 and Chemplex 5701. Marlex CL-100 is a highly cross-linked HDPE produced by the Phillips Chemical Company while Chemplex 5701 is a non-cross-linked, high-molecular weight HDPE which contains a small percentage of hexene as co-polymer. The Marlex CL-100 material used in this study was rotationally molded while the Chemplex 5701 was blow molded.

To investigate the radiation resistance of HDPE, tensile and bend tests were performed following irradiation, and creep testing was conducted during irradiation. For the tensile and bend testing, samples were irradiated at several dose rates in air, backfill soils from the Barnwell, S.C. and Hanford, WA, radioactive waste burial sites and in ion-exchange (IX) resins. Creep tests during irradiation were conducted in air and in IX resins. Irradiation affects polyethylene by causing cross-linking and by introducing unsaturation and free radicals into the polymer.<sup>(1)</sup> Hydrogen evolution accompanies these changes, and the presence of unsaturation and radicals also causes the color to become yellow to brown as the dose increases. Oxidation of polyethylene occurs during irradiation in the presence of oxygen, as in air.<sup>(1,2)</sup> Cross-linking generally leads to some increase in tensile strength and a decrease in elongation at break.<sup>(1)</sup> Oxidation decreases cross-linking and introduces products of oxidation into the polymer which result in decreases in both tensile strength and elongation at break.<sup>(2)</sup>

Irradiations in the soils and IX resins were conducted at 10-11°C to provide a more realistic approximation to burial conditions. The HIC will certainly be in contact with backfill soil. IX resins are perhaps the most common type of radwaste disposed of in these containers. It was of interest to determine whether irradiation of HDPE in the soils or the IX resins might cause any interaction or reaction between an HIC and its external or internal environment. It was also of interest to determine whether mechanical testing following irradiation in the soils and IX resins would be noticeably different from results following irradiation in air. The soils might limit oxygen availability to the HDPE specimens while the IX resins may exclude oxygen entirely since they react with oxygen during irradiation.<sup>(3)</sup>

### EXPERIMENTAL

Irradiations were performed in the BNL Co-60 gamma pool facility at 10-11°C, at dose rates from 1.4 krad/h to 93 krad/h in environments of air, backfill soils from the Barnwell, SC, and Hanford, WA, land burial sites and dewatered ion-exchange (IX) resins. Temperature was monitored by observing pool temperature. For irradiations in air, this was found to be accurate to within 1°C by measurements using a thermocouple. For irradiations in the soils and IX resins the temperature was measured by inserting a thermometer into the medium immediately upon removal of the container from the air tube following irradiation. This was also found to be within 1°C of pool temperature. The highest dose rate used in these tests, 93 krad/h, was chosen to allow irradiation to 100 Mrad in a reasonable time (45 days). The dose value of 100 Mrad was based on the NRC's requirement, as stated in the Technical Position on Waste Form, May 1983, that, "No significant changes in material design properties should result following exposure to a total accumulated dose of 10<sup>8</sup> rads." The lowest dose rate used, 1.4 krad/h, was the lowest available. The IX resin formulation used was a 1:1 mixture of a strong-acid cation resin and a strong-base anion resin. The resin mixture was loaded with soluble contaminants and insoluble corrosion products (crud) according to a recipe from an analysis of spent PWR mixed bed IX resins.<sup>(4)</sup> Test samples were placed in 0.075 m x 0.3 m (3-in. diam x 12-in. high) Pyrex containers for irradiation. Air flowed through the container at a rate of 100 cm<sup>3</sup>/min for the air irradiations. For irradiations in the soils and IX resin, the test samples in the container were completely embedded in well tamped soil or resin.

\*Work carried out under the auspices of the U.S. Nuclear Regulatory Commission.

Radiochromic film was used for dosimetry. The accrued dose may vary as  $\pm 10\%$  from the value indicated by the film. The films used are regularly calibrated against other films which are traceable to the National Bureau of Standards.

Test specimens were stamped or machined from Chemplex 5701 and from two varieties of Marlex CL-100. The Chemplex was taken from blow-molded 55-gallon drums purchased from Plasti-Drum Company, Lockport, IL. One variety of Marlex CL-100, non-HIC Marlex, came from a rotationally molded container purchased from Poly-Processing, Inc., Monroe, LA. The other Marlex CL-100 was actual HIC material provided by Chem-Nuclear Systems, Inc.

Tensile testing was performed according to ASTM D-638 (Tensile Properties of Plastics) at a testing speed of 0.05 m/min (2 in./min). ASTM Type IV specimens, for material  $\leq 0.004$  m (0.160 inch) in thickness, sufficed for the Chemplex and non-HIC Marlex materials while ASTM Type III specimens, for material 0.007-0.014 m (0.28 - 0.55 in) in thickness, were required for the Marlex HIC material, which was typically about 0.013 m (0.500 in.) thick. The Type IV specimens were stamped, as recommended in D-638, using Die C as described in ASTM D-412. The Type III specimens were machined. Bend testing was performed according to ASTM D-790 (Flexural Properties of Plastics and Electrical Insulating Materials). Testing was performed within four days of the end of irradiation. Creep testing during irradiation was performed on Type IV tensile specimens in equipment built for this study. The Type IV tensile specimens were clamped into self-aligning holders and lowered down air tubes in the BNL gamma pool and locked into place. Cables from the sample holders passed over pulleys and were attached to weight pans. Weights added to the pans supplied the creep stress and pan movement provided the creep measurement.

## RESULTS AND DISCUSSION

Qualitatively, the tensile results on the three HDPE materials were similar. Differences were noted mainly in details. However, there was at least one notable difference in characteristics between the Chemplex and Marlex materials which appeared to be related more to the container manufacturing processes than to material differences. Bend tests results showed a substantial difference between Chemplex and Marlex. Irradiation increased creep but the increase became noticeable only at relatively large stresses.

### Tensile Testing

Figure 1 shows a series of typical tensile stress vs elongation vs dose curves for Marlex HIC material irradiated in air at 10-11°C at a dose rate of 9.3 krad/h. Curves similar to those shown in Figure 1 were also obtained for the non-HIC Marlex and Chemplex. For this reason, results for the different materials will not be presented separately. The unirradiated and 9.3 Mrad curves show stress rising to a maximum (which is defined as the yield point, stress or strength) followed by a decrease to a constant stress until the break occurs. In the region of decreasing stress following the yield point neck formation occurs. The constant-stress portion of the curve results from neck propagation.

Irradiation eventually causes the loss of necking behavior. The 47 Mrad and 93 Mrad curves in Figure 1 evidence this from the absence of the transition to a region of constant stress. Loss of necking behavior following irradiation is accompanied by the appearance of surface cracks in these materials. In a specimen

irradiated to a dose beyond which no necking occurs, failure results when one of these cracks propagates through the specimen. The cracks appear prior to the yield point and at the yield point the one crack that will propagate through is apparent. The number of cracks increases with increasing dose.

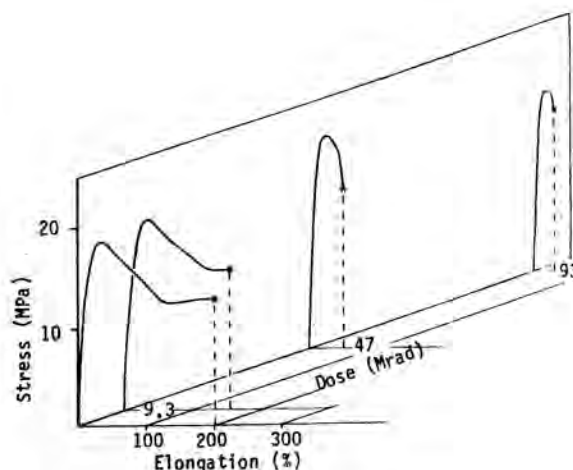


Fig. 1. Three-dimensional plot of tensile stress vs elongation vs gamma ray irradiation dose for Marlex CL-100 HIC material. The irradiations were performed in air at 10-11°C and 93 krad/h.

In Marlex, the cracking always appeared on the inside surface of the container and the cracks were generally evenly spaced along the entire narrow section of the test piece. Figure 2 shows a series of Marlex HIC material tensile specimens and Figure 3 shows a closeup of the cracking that occurs after necking behavior is lost. Examples of the cracking in Marlex HIC material is shown in Figure 3. The cracking observed in the Chemplex is usually limited to the vicinity of the break and occurs on both surfaces of the test piece. These differences in the nature of the cracking behavior appear to be related to surface differences arising in the manufacturing processes. There are no obvious differences between the inner and outer surfaces of the blow-molded Chemplex containers. However, the inside surfaces of the rotationally molded Marlex containers are very smooth and glossy whereas the outside surfaces are dull and somewhat roughly textured. Rotational molding involves melting resin beads on the inside of a mold rotating simultaneously about two perpendicular axes. The outside surface of a container during fabrication is in contact with the mold, whereas, the inside surface is in contact with hot air.<sup>(5)</sup>

Tensile data for Marlex HIC material irradiated at 10-11°C at various dose rates in air, Barnwell and Hanford soils and IX resins are listed in Table I. Figures 4-7 illustrate the data from Table I. Figure 4 plots yield stress vs dose while Figure 5 shows the effect of dose rate on yield stress for a dose of approximately 10 Mrad. Figure 6 plots elongation at break vs dose while Figure 7 shows the effect of dose rate on elongation at break for doses of about 10 Mrad. A quick scan of Figures 4-7 suggests that there is a lot of scatter in the data and that there is no clearly evident segregation of data points by environment (i.e., air, soils, or IX resin) in any of these plots.

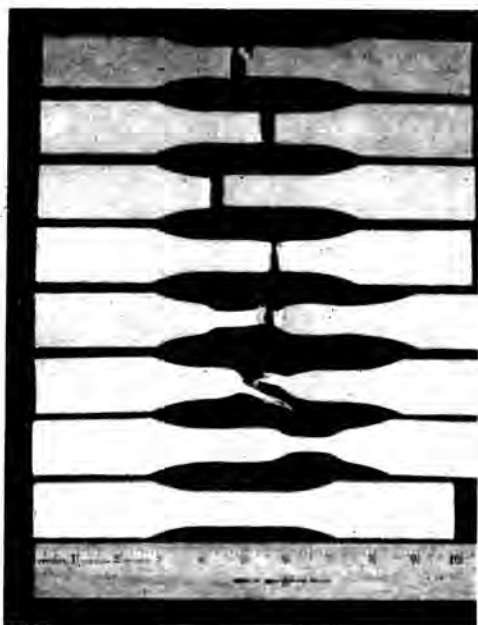


Fig. 2. Photograph of a series of Marlex HIC material Type III tensile specimens irradiated in air at 10-11°C which show the transition from necking to breaking without necking. From bottom: untested specimen; specimen with 1-in. neck; specimen irradiated 2.7 Mrad at 2.5 krad/h; specimen irradiated 9.3 Mrad at 93 krad/h; specimen irradiated 8.6 Mrad at 17 krad/h; specimen irradiated 25 Mrad at 14 krad/h; specimen irradiated 47 Mrad at 93 krad/h and, at top, a specimen irradiated 93 Mrad at 93 krad/h.

Several factors contributed to data scatter including inhomogeneities in the HDPE materials and dimensional variations in the test specimens. The Marlex materials contained small bubbles, which presumably were air bubbles trapped during the container fabrication process. Additionally, the inside and outside surface of the Marlex were noticeably different. The inner Marlex surfaces were also oxidized, presumably from exposure to hot air during the rotational molding process.<sup>(6,7)</sup> The surfaces of the materials corresponding to the inside and outside of the container were left as received and not machined smooth. Thus, the thickness of each test specimen varied at different places along its length. The HIC material varied up to a millimeter in thickness along the length of any individual test specimen while the non-HIC Marlex and Chemplex varied up to a quarter of a millimeter. The thinnest section of each test specimen was used for calculating the cross sectional area.

In Figure 4, which plots yield stress vs dose, most of the data lies above the value for the unirradiated material, as indicated by the dashed line. This indicated that irradiation tended to increase the tensile strength under the conditions of these tests. The unirradiated value is the average of 11 specimens tested over the time period of this task along with the standard deviation about this average. This result was typical of all three HDPE materials tested. Some differences due to irradiation environment appeared at higher doses. The 100 Mrad IX resin point is well above that for air at 93 Mrad. A similar effect

occurred in both the Chemplex and non-HIC Marlex data. Additionally, the IX resin and soil irradiation data for the Chemplex and non-HIC Marlex lie well above that for air at 40-50 Mrad.

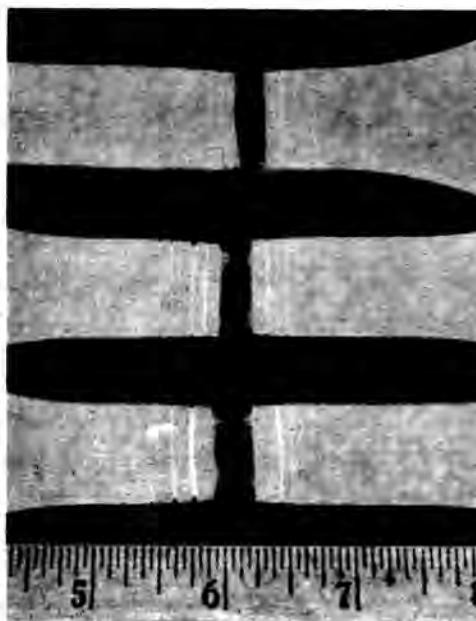


Fig. 3. Closeup of the cracks that occur in irradiated Marlex CL-100 once necking behavior is lost. From bottom: specimen irradiated in IX resins to 10 Mrad at 8.7 krad/h, specimen irradiated in Barnwell soil to 20 Mrad at 11 krad/h and, top, specimen irradiated in Hanford soil to 50 Mrad at 58 krad/h. Irradiations were conducted at 10-11°C.

Figure 5 shows no dose rate effect on the yield stress. There is no trend for these data to either increase or decrease with dose rate. There does not appear to be a noticeable effect of irradiation environment on these results.

There are two striking features evident in Figure 6. First, there is a significant decrease in break elongation with dose. Second, there is a large uncertainty associated with the break elongation of the unirradiated HIC material. The decrease in break elongation correlated with the loss of necking behavior. The break elongation plateaus out at about 50% once necking behavior is lost. Unirradiated Marlex specimens typically fail from a tear which starts at one of the four corners in the necked portion of the specimens. These tears often appear to be started by a small bubble in the material coming to the surface near a corner during formation of the neck and popping. This bubble defect mechanism of failure initiation for the Marlex materials may explain the large variation in the elongation at break. The variation in break elongation for Chemplex was much smaller and occurred at much greater elongation.

Table I

Tensile Test Data on  
Irradiated Marlex CL-100 HIC\* Material<sup>a</sup>

Dose (Mrad)	Dose Rate (krad/h)	Environment	Yield Stress (MPa)	Elongation at Break (%)
0 <sup>b</sup>	---	---	20.1 ± 1.1	220 ± 90
9.3	93	air	21.7	160
47	93	air	23.5	51
93	93	air	19.9	32
8.6	17	air	22.1	66
25	14	air	22.8	57
9.5	5.7	air	21.7	47
2.7	2.5	air	23.2	120
3.6	2.5	air	21.2	66
9.7	58	Barnwell soil	21.4	130
50	58	Barnwell soil	25.5	44
8.5	11	Barnwell soil	20.7	64
20	11	Barnwell soil	24.6	60
3.0	4.0	Barnwell soil	22.4	130
8.0	3.7	Barnwell soil	22.3	43
2.0	1.4	Barnwell soil	21.0	110
50	58	Hanford soil	22.8	52
8.5	11	Hanford soil	21.0	49
20	11	Hanford soil	22.3	50
3.0	4.0	Hanford soil	21.1	170
13	79	IX resin	20.3	110
49	79	IX resin	23.2	47
100	79	IX resin	24.3	36
20	11	IX resin	20.8	76
10	8.7	IX resin	20.1	64
3.0	4.0	IX resin	22.1	200
3.0	3.7	IX resin	21.4	60
3.0	4.0	IX resin/ Barnwell soil	21.4	56
10	11	IX resin/ Barnwell soil	21.0	62

\*EnviroSAFE is the trademark of the high integrity containers vended by CHEM-NUCLEAR SYSTEMS, Inc. Containers are rotationally molded using MARLEX CL-100 high density, highly cross-linked polyethylene.

<sup>a</sup>Irradiations were performed at 10-11°C. Tensile testing was performed according to ASTM D-638 (Tensile Properties of Plastics) using one Type III specimen per test.

<sup>b</sup>These data are from 11 unirradiated specimens tested over the time period of this task.

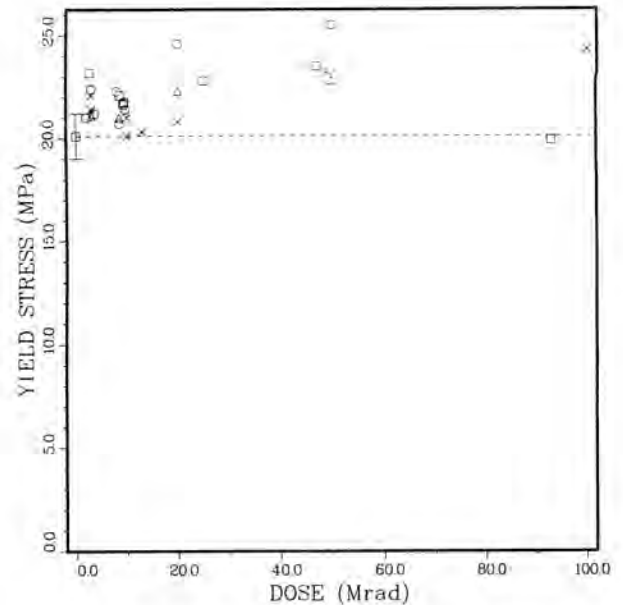


Fig. 4. Yield stress vs dose of Marlex CL-100 HIC material irradiated at 10-11°C. Symbols indicate irradiation environment: air-□, Barnwell soil -○, Hanford soil -△, IX resin -X.

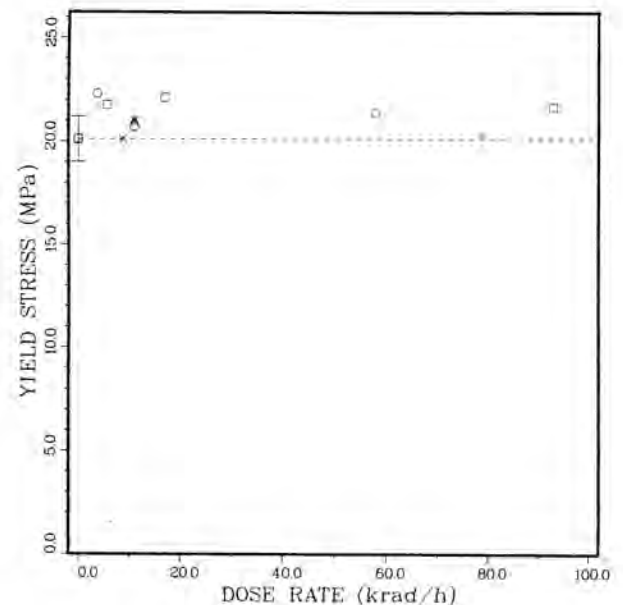


Fig. 5. Yield stress vs dose rate of Marlex CL-100 HIC material irradiated at 10-11°C for total doses from 8.0-13 Mrad. Symbols indicate irradiation environment: air-□, Barnwell soil -○, Hanford soil -△, IX resin -X.



In order to solve the differential equation (6), the boundary and initial condition (7) (8) has been assumed

$$C(+0, t) = C_0 \quad (t > 0) \quad (7)$$

$$C(X, +0) = 0 \quad (X > 0) \quad (8)$$

Thus, the solution of equation (6) has been obtained in equation (9), introducing tortuosity ( $\sqrt{2}$ )

$$\frac{C(\alpha, \beta)}{C_0} = 1 - \frac{2}{\sqrt{\pi}} \int_0^{\frac{\alpha}{2\sqrt{\beta}}} \exp\left(-\xi^2 - \frac{\alpha}{4\xi}\right) d\xi \quad (10)$$

$$\text{where } \alpha = \frac{V}{D} X \quad (11)$$

$$\beta = \frac{V^2}{R_f D} t \quad (12)$$

The experimental results were the counted activities as a function of X and the regression of the concentration profile along the axis of cylindrical bed, gives  $C_0$  and  $R_f \cdot D$  respectively. The intrinsic diffusion coefficient D is given by the limiting ionic conductances and the solid phase density and porosity can be measured, therefore, the distribution coefficient  $K_d$  can be obtained by this experiment. The regression was performed with the methods of least squares numerically.

#### 4. RESULTS AND DISCUSSION

Though stainless steel and teflon filters are inserted at the end of the bed, the radial disturbance of non uniform distribution at the contact surface to the feed solution might cause the interference on the  $K_d$ -value evaluation. Therefore, in the first experiment, the sliced dish-shaped samples were divided into two parts to find if the activity per unit weight in the center was different from that in the outer. Two different slices of  $X \sim 1\text{mm}$  and  $3\text{mm}$  were taken to be analysed. Table I shows the measured count rate /g - minerals. According to the result, no great differences of their activity levels were observed between the center and the outer of the samples.

Table I. Relative concentrations in the center and the outer of the sample

X	Count rate/g-minerals at the center (cpm/g) $\times 10^4$	Count rate/g-minerals at the outer (cpm/g) $\times 10^4$
1	$2.52 \times 10^4$	$2.56 \times 10^4$
3	$7.74 \times 10^3$	$7.87 \times 10^3$

Table II shows the properties of the bed materials, the flow rates and operation time employed here. Mixing either quartz or zeolite to bentonite increases the hydraulic conductivity. The feed pressure to the column has been kept constant. Therefore the flow rate of the mixture is higher than that of pure bentonite bed in Table 2.

Figure 5 shows the relative concentration profile of cesium along the axis of the column after 42 hours operation in the mixture of 70 % bentonite - 30% quartz. The ordinate is

is the relative concentration  $C/C_0$  and the abscissa is the distance from the contact surface. The solid curve shows the result of regression. Good agreement of the experiment and theoretical analysis has been observed. The interstitial velocity was estimated to be  $2.4 \times 10^{-8}$  (m/sec). The temperature was ambient ( $\sim 20^\circ\text{C}$ ) and the feed concentration was kept at  $10^{-6}$  M for all the experiment as explained in section 2. Similar analysis have been made for other materials. Though the profiles are not the same because of the different flow rate and operation time, the experimental results are well described by equation (10).

Figure 6 shows one of the strontium concentration profiles obtained in this experiment. As in the case of cesium, the evaluated relative concentration profiles agree well with the solution of convective-diffusion equation (10)

Table III shows the evaluated  $R_f \cdot D$  and  $K_d$  values for strontium and cesium in a variety of bed materials. In general  $K_d$  values for cesium is larger than that for strontium by a factor of 10. Adding zeolite,  $K_d$  values for cesium increases much greater than those for Sr. Though quartz mixing had been expected to decrease the  $K_d$  values, the experimental results indicate that the  $K_d$  values might be even increased especially in the case of strontium. Distribution coefficient  $K_d$  at equilibrium has to be evaluated, in order to find the compaction and the interstitial flow effects on the sorption characteristics. For this purpose, batch sorption experiment has been made in a 50 cc beaker for the same minerals.

Table IV shows the  $K_d$  values in this batch experiment. The zeolite mixture gives higher  $K_d$  than pure bentonite for Sr and Cs as in Table 5. Since the water to mineral ratios ( $W_L/W_s$ ) are quite different between the diffusion experiment and the batch experiment ( $\sim 0.25$  to  $100$ ), the cation concentration in liquid phase in the diffusion experiment is much higher than that in the batch experiment ( $\sim 4000$  to  $40$  ppm in sodium ion concentration). Therefore, the  $K_d$  values in Table 5 and 6 may not agree well.

The obtained  $K_d$  values are plotted by the zeolite content to find the effect in Fig. 7. For both Sr and Cs, the  $K_d$  value are increased with increasing the zeolite content. The inclinations in the batch experiment are about the same for Sr and Cs as those in the diffusion experiment. The  $K_d$  values for cesium agree well in the batch and the diffusion experiment, while the  $K_d$  values for strontium is about 10 times larger in the batch experiment than that in the diffusion experiment.

Figure 8 shows the effect of quartz content on  $K_d$  values. In comparison to the zeolite mixture the remarkable increase of  $K_d$  values can not be observed with increasing the quartz content. As in Fig. 5, the batch and diffusion experiments show the better agreement in cesium than in strontium.

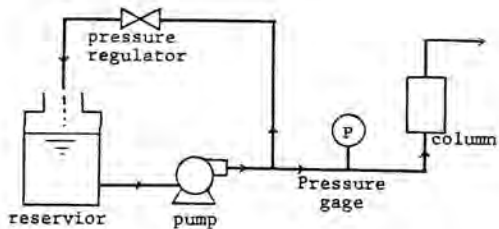


Fig.1 Schematic diagram of experiment

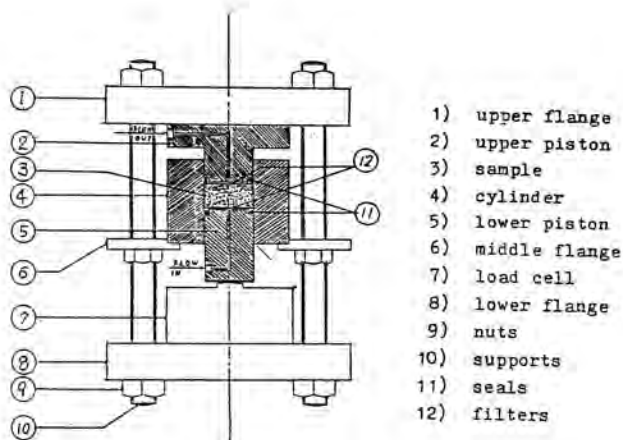


Fig.2 Equipment for migration tests

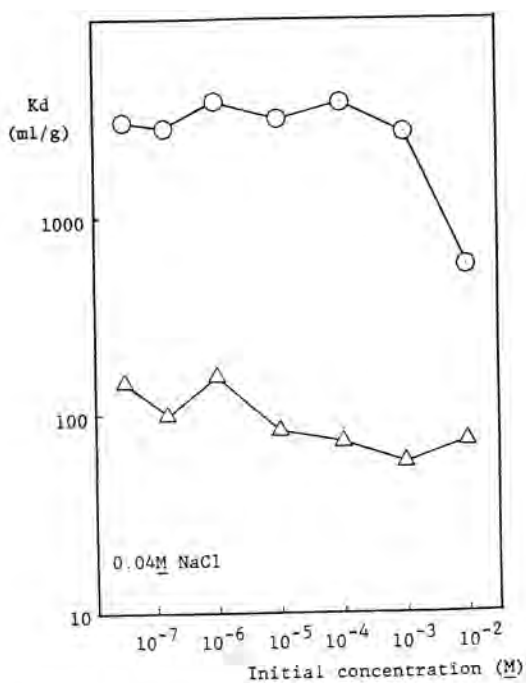


Fig.3 Cs distribution coefficient vs Cs concentration in liquid phase

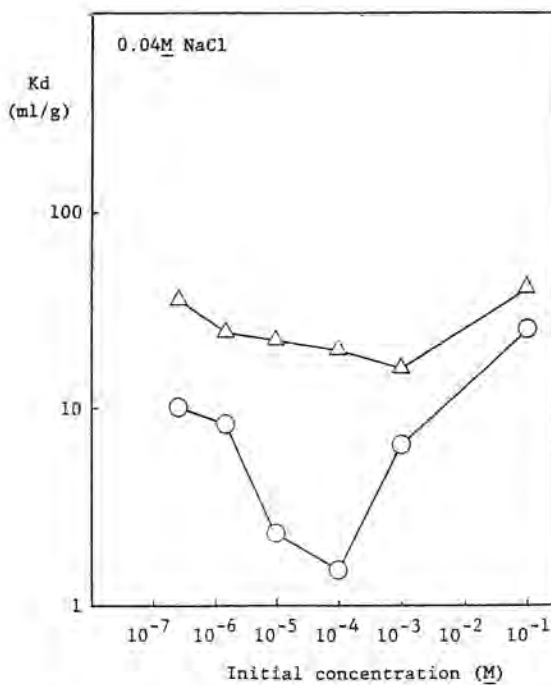


Fig.4 Sr distribution coefficient vs Sr concentration in liquid phase

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Table II presents estimates of  $D_N$  and the time to reach  $D_N$  for several dose rates using these equations. The values of  $D_N$  and the time to dose at dose rates less than 2000 rad/h in Table II represent extrapolations from the data obtained in this study. The dose rates were chosen to bracket estimated initial dose rates for highly loaded IX resin waste. For wastes whose activity is dominated by isotopes with half lives on the order of 30 yrs (e.g., Cs-137), such that the total accumulated dose would be  $10^8$  rad, the dose rate to which the container may be exposed upon loading would be approximately 250 rad/h. Based on this loading, one year after loading, the accumulated dose would be approximately 2.3 Mrad. Similarly, wastes whose activity is dominated by isotopes with half lives of 5 years (e.g., Co-60), loaded such that the total accumulated dose would be  $10^8$  rad, the dose rates to which the container would be exposed upon loading is approximately 1500 rad/h. In this case, one year after loading, the accumulated dose would be approximately 13 Mrad. It should be noted that these estimates of anticipated dose rates and doses may be conservatively high since they neglect container geometry and self-shielding by the resin wastes. On the other hand, it should be remembered that these estimates are based on irradiation data taken at 10-11°C. If the higher temperatures that might be encountered in storage speed up the transition, then these estimates may not be conservative. Using these dose rates as a benchmark for expected field conditions leads one to conclude that embrittlement of HDPE HICs could occur within a few months to a year. It would appear that the consequences of such embrittlement during storage and following burial should be considered in the design of HICs made from HDPE.

Table II

Estimates of the Dose and Time-to Dose for the Necking to Breaking Without Necking Transition for Marlex CL-100 and Chemplex 5701

Material	R(rad/h)	$D_N$ (Mrad)	Time to $D_N$ (Days)
Marlex CL-100	2000	3.0	63
Marlex CL-100	1000	2.1	88
Marlex CL-100	500	1.5	125
Marlex CL-100	100	0.7	292
Chemplex 5701	2000	6.3	130
Chemplex 5701	1000	5.0	209
Chemplex 5701	500	4.0	335
Chemplex 5701	100	2.4	1000

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