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EPICOR–II Resin/Liner Investigation: Low-Level Waste Data Base Development Program for Fiscal Year 1990

Annual Report

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Prepared for
U.S. Nuclear Regulatory Commission

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ABSTRACT

The EPICOR-II Resin/Liner Investigation: Low-Level Waste Database Development Program, funded by the U.S. Nuclear Regulatory Commission, is (a) studying the degradation effects in EPICOR-II organic ion exchange resins caused by radiation, (b) examining the adequacy of test procedures recommended in the Branch Technical Position on Waste Forms to meet the requirements of 10 CFR 61 using solidified EPICOR-II resins, (c) obtaining performance information on solidified EPICOR-II ion exchange resins in a disposal environment, and (d) determining the condition of EPICOR-II liners.

Results of the third sampling analysis of ion exchange resins from prefilters PF-8 and PF-20 are compared with baseline data from tests performed on unirradiated resins and with results from the first and second samplings to determine if degradation has occurred because of the high internal radiation dose. Results of compression tests on seven-year old waste forms containing EPICOR-II resins solidified with both Portland Type I-II cement and DOW vinyl ester-styrene are presented and compared to earlier compression test data. Results of the fifth year of data acquisition from the field testing are also presented and discussed.

SUMMARY

The March 28, 1979 accident at Three Mile Island Unit 2 (TMI–2) released approximately 560,000 gal of contaminated water to the Auxiliary and Fuel Handling Buildings. The water was decontaminated using a three–stage demineralization system called EPICOR–II containing organic and inorganic ion exchange media. The first stage of the system was designated the prefilter, and the second and third stages were called demineralizers. Fifty EPICOR–II prefilters with high concentrations of radionuclides were transported to the Idaho National Engineering Laboratory for interim storage before final disposal at a commercial disposal facility in the State of Washington. Research is being conducted on materials from four of those EPICOR–II prefilters under three tasks of the TMI–2 EPICOR–II Resin/Liner Investigation: Low–Level Waste Database Development Program.

In the first task, Resin Degradation, resin cores were obtained from prefilters PF–8 and PF–20 during first, second, and third samplings using special tools developed for that purpose. A series of characterization tests was performed on the resins to determine if degradation due to radiation had occurred during interim storage. These tests included American Society for Testing Materials procedures, infrared spectroscopy, gas chromatography, high performance liquid chromatography, scanning electron microscopy, supercritical fluids chromatography, barium chloride precipitation, inductively coupled plasma–atomic emission spectroscopy, and gamma–ray spectroscopy. Analysis comparing test results of the first, second, and third samplings of resins from EPICOR–II prefilters was compared with earlier work and found to be consistent with those findings. The strong acid cation resins are experiencing a decrease in total exchange capacity as the absorbed radiation dose increases. The analysis of the resins from the third sampling identify that very significant degradation has occurred. While the organic ion exchange media (resins) from these EPICOR–II prefilters have suffered significant physical changes, the contained radionuclides remain within the resin bed. The acceptability of EPICOR–II prefilters for disposal in high integrity containers at a commercial disposal site was confirmed.

For the second task, Resin Solidification, Portland Type I–II cement and vinyl ester–styrene (VES) waste forms incorporating ion exchange resin waste from EPICOR–II prefilters were subjected to the tests specified in the "Technical Position on Waste Form" issued by the U.S. Nuclear Regulatory Commission. Waste form performance data were obtained as a result of the work. Seven–year old EPICOR–II resin waste forms made with Portland Type I–II cement and DOW vinyl ester–styrene were compression tested and the results were compared to similar waste forms tested earlier in the program. Results indicated an increase in strength of both types of waste forms with age.

The third task, Field Testing, is examining the effect of disposal environments on solidified resin wastes from EPICOR–II prefilters. The purpose of this task, using lysimeter arrays at Oak Ridge National Laboratory and Argonne National Laboratory in Illinois, is to expose samples of solidified ion exchange resin to the actual physical, chemical, and microbiological conditions of a disposal environment. The study is designed so that continuous data on nuclide release and movement, as well as environmental conditions, will be obtained over a 20–year period.

Each month, data stored on a cassette tape are retrieved from the data acquisition system and translated into an IBM PC–compatible disk file. At least quarterly, water is drawn from the porous cup soil–water samplers and the lysimeter leachate collection compartment. Those water samples are analyzed for beta– and gamma–producing nuclides.

Results of the fourth year of data acquisition are presented in this report. These results show that radionuclides are continuing to move from the waste forms and through the soil column. Also, some data on waste–form performance are presented. VES is comparable to cement in retaining Sr–90, unlike findings from Savannah River Laboratory, which favor cement to be a better retainer than VES.

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ACRONYMS AND ABBREVIATIONS

ANL–E	Argonne National Laboratory–East
ASTM	American Society for Testing & Materials
DAS	data acquisition system
DOE	U.S. Department of Energy
GC	gas chromatography
HPL	high–performance liquid chromatography
ICP–AES	inductivity coupled plasma–atomic emission spectrometry
INEL	Idaho National Engineering Laboratory
MHC	moisture holding capacity
NRC	U.S. Nuclear Regulatory Commission
ORNL	Oak Ridge National Laboratory
SEM	scanning electron microscope
SFC	supercritical fluid chromatography
TAN–607	Test Area North Building 607
TMI–2	Three Mile Island Unit 2
TP	technical position on waste form
VES	vinyl ester–styrene

ANNUAL REPORT OF THE TMI–2 EPICOR–II RESIN/LINER INVESTIGATION: LOW–LEVEL WASTE DATABASE DEVELOPMENT PROGRAM FOR FISCAL YEAR 1990

INTRODUCTION

The March 28, 1979 accident at Three Mile Island Unit 2 (TMI–2) released approximately 560,000 gal of contaminated water to the Auxiliary and Fuel Handling Buildings. That water was decontaminated using a demineralization system called EPICOR–II developed by Epicor, Inc.[a] The contaminated water was cycled through three stages of organic and inorganic ion exchange media. The first stage of the system was designated the prefilter, and the second and third stages were called demineralizers. After the filtration process, the ion exchange media in 50 of the prefilters contained radionuclides in concentrations greater than the limits for low–level wastes. These prefilters were transported to the Idaho National Engineering Laboratory (INEL) for interim storage before final disposal. A special overpack, or high–integrity container, was developed during that storage period for use in disposing of the prefilters at a commercial disposal facility in the State of Washington. As part of the EPICOR and Waste Research and Disposition Program funded by the U.S. Department of Energy (DOE), 46 prefilters were disposed. Four prefilters used in the U.S. Nuclear Regulatory Commission (NRC) studies were stored in temporary storage casks outside the Hot Shop of Test Area North Building 607 (TAN–607) at the INEL. Those four prefilters were disposed during this reporting year at the Radioactive Waste Management Complex on the INEL Site.

Under the EPICOR and Waste Research and Disposition Program, continuing research has been conducted by EG&G Idaho, Inc. (EG&G Idaho) on materials from those EPICOR–II prefilters.[1,2,3] That work is now directed by the NRC as part of the TMI–2 EPICOR–II Resin/Liner Investigation: Low–Level Waste Database Development Program. Studies are being conducted on organic ion exchange resins from selected prefilters. The resins are being examined to measure degradation and tests are being performed to characterize solidified ion exchange media.

This report discusses the resin degradation studies conducted on the third set of resin samples removed from the EPICOR–II prefilters (PF–8 and PF–20). The results are compared with findings from studies of the first and second sampling, as described in References 4 and 5 with those of the third sampling described in Reference 6. The degradation studies determine the acceptability of EPICOR–II prefilters for disposal in high–integrity containers at the commercial disposal site at Hanford, Washington by identifying (a) degradation effects on the ion–exchange resins caused by contained radiation and (b) possible release of contained radionuclides from the ion exchange resins.

Another aspect of this program, the solidification of EPICOR–II wastes from prefilters PF–7 and PF–24 using Portland Type I–II cement and vinyl ester–styrene (VES) (a proprietary solidification agent developed and supplied by the DOW Chemical Company), was investigated.

The formulations used for the immobilization of EPICOR–II wastes were developed to produce waste forms meeting the regulatory requirements

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a. Mention of specific products and/or manufacturers in this document implies neither endorsement or preference nor disapproval by the U.S. Government, any of its agencies, or EG&G Idaho, Inc., of the use of a specific product for any purpose.

of 10 CFR 61, "Licensing Requirements for Land Disposal of Radioactive Waste." The NRC, in its "Technical Position on Waste Form" (TP),[7] provides guidance to waste generators on waste form test methods and acceptable results for compliance with the waste form requirements of 10 CFR 61. In this study, EPICOR–II waste forms were subjected to the specified test procedures to ensure compliance with stability requirements. During this reporting period, four EPICOR waste forms were compression–tested. The results of those tests compared to the earlier data indicate the waste form strength is increasing with age. Solidified waste forms containing EPICOR–II ion exchange resin waste are currently being field–tested using lysimeters. The intent of the testing is to expose waste form samples to the physical, chemical, and microbiological environment of typical disposal sites in the eastern United States.[1,2,3] It is intended that the lysimeters monitor release of nuclides from the buried waste forms and provide data that accurately determines movement as a function of time and environmental conditions. Emphasis is placed on investigating the requirements of 10 CFR 61. The study is designed so that continuous data on nuclide release and movement, as well as environmental conditions, will be obtained over a 20–year period.

This report contains data from the fifth year of lysimeter operation, as well as cumulative data on water balance and nuclide content of water samples. Data for this report were retrieved from the data acquisition system (DAS) and from beta and gamma analyses of lysimeter water samples.

RESIN DEGRADATION

Materials and Methods

To develop baseline data for the resin degradation studies, unirradiated ion exchange resins representative of those in the EPICOR-II prefilters were provided by Epicor, Inc. The unirradiated resins were identified by functional group, exchangeable species, and matrix (e.g., sulfonic acid, strong acid cation, and styrene). They were characterized for comparison with the irradiated resins obtained from prefilters PF-8 and PF-20. Both the unirradiated and irradiated resins were examined, using similar techniques. American Society for Testing and Materials (ASTM) tests were used to determine moisture content, density, salt-splitting capacity, and exchange capacity.[8] Vapor phase, liquid, and super critical fluid chromatography were used to quantitatively analyze the rinse and soak solutions for leachable organic compounds. Inductively coupled plasma-atomic emission spectroscopy (ICP-AES) determined sulfonic acid groups and scanning electron microscopy determined the physical condition of the resins. Gamma-ray spectroscopy determined radionuclides in aqueous solutions.

Throughout this report, the following nomenclature applies to various sizes and configurations of materials removed from the EPICOR-II prefilters for examination:

- *Resin Core*—One core was removed from each prefilter (PF-8 and PF-20) using coring tools.
- *Resin Samples*—100-mL volumes were removed from the resin cores. Three samples (PF-8#1, PF-8#2, and PF-8#3) were removed from the PF-8 core. One sample (PF-20) was removed from the PF-20 core.

Coring. Resin cores were removed remotely from EPICOR-II prefilters PF-8 and PF-20 in 1983, 1985, and 1989 using coring equipment based on a design developed at Battelle Columbus Laboratories and modified for use at the INEL.[9] The coring equipment consists of the following: (a) coring tool and shutter used for collecting, transporting, and storing the resin core; (b) casing and shutter used for retaining the void space in the bed and preventing collapse of the resin bed after removal of the coring tool; and (c) vibrator tool, which drives the coring tool, casing, and shutters into the resin bed. NUREG/CR-4150[4] further describes the coring equipment.

Gamma Scanning. Full-length isotopic gamma scans were made of each resin core from the first and second samplings to produce axial profiles of radionuclide distribution. Those profiles were used to determine the regions of highest radionuclide loading in the cores. Isotopic spectral gamma measurements then were obtained at locations of highest radionuclide concentration.[10] The two radionuclides having measurable concentrations detected by gamma spectroscopy were Cs-134 and Cs-137.

After removing the cores, full-depth gross gamma scans were made within the resin beds of the prefilters. Those scans were used to estimate the total integrated dose absorbed by the resins.

Radiation Doses. Gamma radiation doses were calculated for the resin samples using gamma dose measurements obtained during full-depth, gross gamma scans within the prefilter resin beds.[4,5] Measurements obtained at elevations of interest were used with the method outlined[11] to calculate the total integrated beta-gamma radiation dose for each resin sample. The calculated doses are given in Table 1.

Sampling. At the Test Reactor Area of the INEL, each resin-filled coring tool was transferred from its cask into the Hot Cell for remote removal of resin samples. The coring tool shutter was withdrawn to expose the layers of different ion exchange media (resin). It was noted that some smearing of material from one layer into another had occurred when the shutter was

Table 1. Calculated radiation doses for EPICOR–II irradiated resin samples

Sample	Calculated Gamma Activity at Location of Sample in Prefilter (R/min)	Total Gamma Radiation Dose (rad)	Total Beta–Gamma Radiation Dose (rad)
PF–8#1	21.2	10.5×10^7	1.5×10^8
PF–8#2	13.5	6.7×10^7	0.9×10^8
PF–8#3	19.8	9.8×10^7	1.4×10^8
PF–20	13.2	6.4×10^7	0.9×10^8

inserted during coring operations and withdrawn for sampling. That smearing required careful removal of the mixed surface material to expose unmixed resin near the center of the core. It was the unmixed material that was the target for collection.

Resin samples were collected with a vacuum pump and a deionized water–filled, graduated glass column.[4,5,6] One end of a flexible rubber tube was attached near the top of the glass column and the other end to a vacuum pump outside the Hot Cell. A segment of rubber tubing was attached from the top of the glass column to a stainless steel tube to form a wand. With the vacuum pump running, the wand was positioned over the target resin with a master–slave manipulator in such a way that resin was drawn into the column. Sample sizes of 100 mL were collected. Three samples were obtained from the PF–8 core, two styrene cation resins (PF–8#1 and PF–8#3) and one phenolic cation resin (PF–8#2). A single styrene cation resin sample was obtained from PF–20. The resin samples were collected from or near those regions of highest radionuclide loading. This study is concerned with degradation of organic ion exchange resins; therefore, only organic resin samples were removed from the cores. No anion samples were collected from either core because of the much lower radionuclide content of the anion exchange resin and low radiation dose indicated by gamma scans (resulting in much less degradation than in the cation exchange resins.)

Sample Preparation. The radiation levels of the samples were of such intensity that analytical work performed on the irradiated resins needed to be done within a hot cell environment. However, that would have made characterization and analysis of the samples very costly and time–consuming. Previous tests performed at the INEL on unirradiated resins had shown that an EPICOR resin could be stripped of 99% of its cations using a 10% hydrochloric acid solution.[6] Based on that information, it was decided to strip the radionuclides from the PF–8 and PF–20 resin samples.

As described in the previous section, samples were removed from the coring tools and drawn into separate ion exchange columns filled with deionized water. The samples were allowed to soak 24 hours in the water–filled columns. At the end of that time, the deionized water used to soak each of the samples (PF–8#1, PF–8#2, and PF–8#3, and PF–20) in the separate columns was examined visually. Each ion exchange column was reconfigured, and the deionized water was removed through the shutoff valve of the column and retained for analysis. The samples were rinsed two times each with deionized water, which was added by the pump through the tubing at the top of each column. Resin samples PF–8#1 and PF–8#3 and PF–20 showed restriction to flow during this initial rinse procedure and required a batch rinse with the water removed by decanting. That deionized water also was retained for gas chromatography (GC) analysis and functional group tests.

A solution of 10% hydrochloric acid was pumped through each resin sample at a rate of 100 mL/min. That procedure continued until 60 sample volumes [illegible] determined to remove 99% of the cesium) or 6 L of acid flowed through each resin sample. Representative quantities of that acid rinse were collected and later analyzed.

The deionized water soak and rinses, and the hydrochloric acid rinse greatly reduced the radionuclide content of the resins. That made it possible to remove the samples from the Hot Cell and perform the analyses in a Type II fume hood containing a high-efficiency particulate air filter on the outlet duct.

Characterization of Unirradiated and Irradiated Resins. The following analytical methods were used to characterize the unirradiated Epicor, Inc. resins (strong acid cation and phenolic cation) and samples from PF-8 (two strong acid cation samples and one phenolic cation sample) and PF-20 (strong acid cation):

- ASTM Procedures for the Physical and Chemical Properties of Particulate Ion Exchange Resins[12]
- Gas, liquid, and supercritical fluid chromatography
- ICP-AES for determination of sulfonic acid groups
- Gamma-ray spectroscopy
- Scanning electron microscopy.

American Society of Testing Materials Tests. ASTM procedures were used to determine the chemical and physical conditions of the ion exchange resins.[4,5,6,12] Results from analysis of the irradiated EPICOR-II resins and the unirradiated resins were compared to determine if degradation had occurred. The following ASTM procedures[a] were used for the strong acid cation and phenolic cation exchange resins:

- The pretreatment phase of the ASTM procedure converted the ion exchange resins to one standard form (usually the sodium form for cation resins). This standard form provided a baseline for other ASTM tests.
- The water retention capacity test indicated the porosity of the resin. The resin's porosity is dependent on the amount of effective cross-linking. The higher the water retention capacity, the lower the effective cross-linking. In the case of the PF-8 and PF-20 resins, the water retention capacity is an indication of the amount of divinylbenzene cross-linking.
- The backwashed and settled density test determined changes in effective cross-linking between new and used resins. The density is proportional to the amount of effective cross-linking in the resin.
- The salt-splitting capacity test showed the number of sulfonic acid groups contained in a cation ion exchange resin. A decrease in salt-splitting capacity would show a loss of functional sulfonic acid groups. Phenolic, carboxylic acid, and phosphonic acid functional groups also will exhibit salt-splitting capacity to some degree.
- The total exchange capacity test determined the exchange capacity of cation ion exchange resins that contain functional groups in addition to, or different from, sulfonic acid functional groups.

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a. The tests were performed in accordance with ASTM standards; deviations are within allowable limits of those standards.

Gas Chromatography. Gas chromatography vaporizes a liquid sample and separates it into components by using a GC column containing a mobile phase and a stationary phase. In the case of the EPICOR–II resin samples, a DB5 30–m–long Durabond column with a 1.5 microfilm thickness from J. W. Scientific was chosen for the GC analysis. Any organic products in the original sample were concentrated by use of hexane. Samples of the soak and rinse solutions from PF–8 and PF–20 were examined.

Liquid Chromatography. High–performance liquid chromatography (HPLC) separates a liquid into its components by means of liquid chromatography, partition chromatography, ion–exchange chromatography, or exclusion chromatography. This study relied on the technique of partition chromatography, an HPLC technique in which the solute is partitioned between two immiscible solvents, one fixed and the other mobile. The fixed phase was an HPLC ODS C–18 column from Waters Chromatography, and the mobile phase was acetonitrile. Aliquots of the hexane extracts of the soak and rinse solutions from PF–8 and PF–20 were injected into a Kratos HPLC using a UV–VISIBLE Detector, and HPLC chromatograms were obtained.

Supercritical Fluid Chromatography. Supercritical fluid chromatography (SFC) raises the mobile phase above its critical temperature and pressure. At this point, called the critical point, the mobile phase is neither a gas nor a liquid. Separations are made based on the different solubilities of analytes by changing the density of the mobile phase. This techniques lends itself well for thermally labile compounds and larger molecular weight polymeric materials that cannot be run using other chromatography techniques. Hexane–extracted liquid samples of soak and rinse water from the EPICOR–II resins were analyzed using a 10 meter SB–Phenyl–50, 100 micron ID, 0.25 micron film thickness capillary column. Carbon dioxide was used as the mobile phase and an isothermal (100°C) pressure program was run starting at 100 atm and going to 375 atm at a rate of 5 atm/min.

Inductively Coupled Plasma – Atomic Emission Spectroscopy for the Determination of Sulfur. It has been shown that the EPICOR–II cation resins are sulfonic acid, divinylbenzene, styrene type resins.[8] The high internal radiation dose received by those resins could cause loss of the active sulfonic acid sites. This would result in finding sulfate in the deionized water soak and rinse solutions from the PF–8 and PF–20 strong acid cation resins. ICP–AES is an elemental technique and can detect sulfur present in low concentrations in aqueous solutions. This work was performed on a Perkin Elmer Plazma II ICP–AES using three analytical wavelengths for sulfur.

Scanning Electron Microscopy. To determine the physical conditions of the resin samples from PF–8 and PF–20, scanning electron microscope (SEM) photomicrographs were obtained of the resins at different magnifications. The photomicrographs allowed examination of the resins for cracks, bead breakage, bead softening, agglomeration, and other defects.

Gamma–Ray Spectroscopy. Gamma–ray spectroscopy determined the total amount of Cs–134 and Cs–137 present in the resin samples. Samples of soak, rinse, and elution liquids were measured.

Test Results

Because the unirradiated resins were seven years old, they might have been expected to show some degradation. However, the unirradiated resins showed little apparent change from previous analyses.[4,5,6,8] The nine–year old irradiated resins from PF–8 and PF–20 also would have been expected to show some degradation due to age.[13,14] Since the unirradiated resins showed no degradation because of age in this study, any degradation of the irradiated resins was assumed to be from radiation damage and not from age.

American Society for Testing and Materials Tests. Results of the tests performed on irradiated and unirradiated resins from the third sampling of PF–8 and PF–20 are listed in Table 2.

Table 2. Results of ASTM tests on irradiated and unirradiated ion exchange resins–third sampling[a]

ASTM Test Parameter	Resin Sample					
	Unirradiated Strong Acid Cation[b]	PF–8#1 Strong Acid Cation	PF–8#3 Strong Acid Cation	PF–20 Strong Acid Cation	Unirradiated Phenolic Cation Resin[b]	PF–8#21 Strong Cation
Water retention capacity	41.48 ± 0.89%	56.56 ± 1.21%	56.06 ± 1.20%	48.53 ± 1.04%	45.36 ± 0.97%	52.78 ± 1.13%
Backwashed and settled density (g/mL)	0.837 ± 0.011	0.812 ± 0.011	0.829 ± 0.011	0.886 ± 0.012	0.705 ± 0.009	0.812 ± 0.011
Salt–splitting capacity (meq/g)[c]	5.21 ± 0.96	3.78 ± 0.70	3.83 ± 0.71	4.42 ± 0.82	2.17 ± 0.04	3.78 ± 0.70
Total exchange capacity (meq/g)[c]	6.28 ± 0.97	3.44 ± 0.53	3.64 ± 0.56	4.77 ± 0.74	4.65 ± 0.71	3.44 ± 0.52

a. The errors associated with the data were experimentally determined – they were calculated as the first standard deviation from the mean.

b. This unirradiated sample was tested at a different time than the other samples.

c. Measured in milliequivalents per gram of dry resin.

(Results from the first and second samplings were previously reported.)[4,5]

The results of these analyses are graphically presented in Figure 1. Probably the most important characteristic of ion exchange media is total exchange capacity. This figure shows the marked decrease in exchange capacity as the resins received more radiation doses. While there is some separation of the curves for Samples PF–8#1 and PF–8#3 from PF–8#2 (phenolic) and PF–20, the No. 1 and No. 2 sample curves are very close and all four exhibit common shapes. Differences in these data were introduced during measurement of radiation doses.

Gas, Liquid, and Supercritical Fluid Chromatography. The irradiated resins were soaked and rinsed with deionized water. These water samples were then extracted with hexane in an attempt to determine if water soluble organic molecules could be leached from the irradiated resins. In all three chromatography experiments, no detectable organic material was found. This would imply that the degradation products of the polystyrene divinylbenzene are not readily extractable into water. Therefore, organic decomposition products are expected to stay with the intact resin beads.

Inductively Coupled Plasma – Atomic Emission Spectroscopy. Assay of the solutions collected during resin treatment clearly demonstrate that sulfonic acid groups are being lost due to irradiation of the sulfonic acid containing polystyrene divinylbenze based resins (See Table 3). Some sulfate also was found in the PF–8#2 phenolic resin sample. This can be

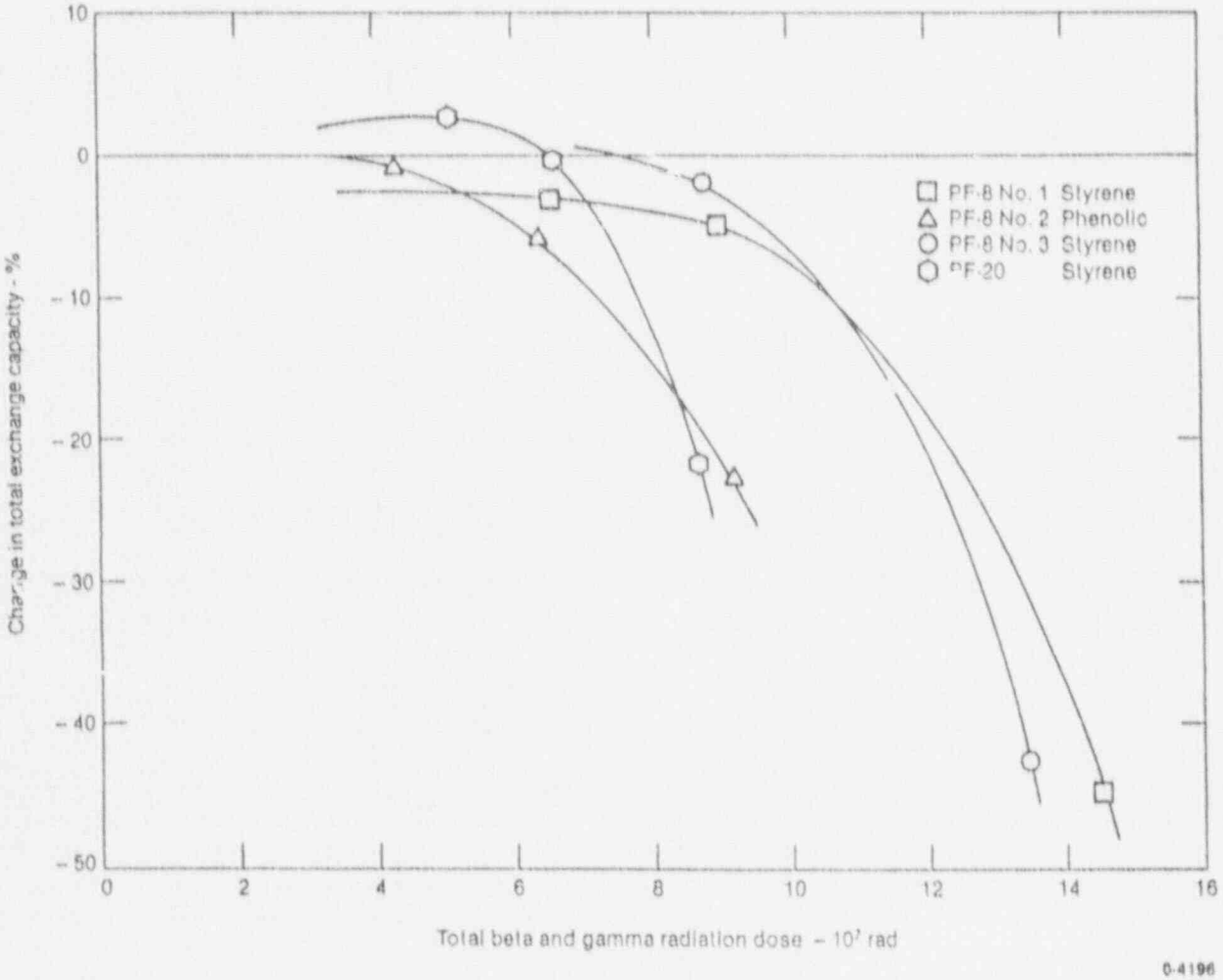

Figure 1. Change in total exchange capacity with increase in radiation dose.

Table 3. Sulfate determined in aqueous solutions from resins by inductively coupled plasma–atomic emission spectroscopy–third sampling

Resin Sample No.	mg SO_4 / 100 mL Sample
PF–8#1	25
PF–8#2	4
PF–8#3	63
PF–20	37
Unirradiate Resin	0.3

explained by the fact that this sample was taken next to the strong acid resin. These resin samples still appeared to be moist, which would allow the sulfate to migrate slowly. Also, because of the proximity of these resins in the sampling tool, a small amount of the strong acid cation resin was collected with the phenolic resin.

Scanning Electron Microscopy. SEM photomicrographs of the unirradiated Epicor, Inc. supplied resins were presented in two NRC reports.[4,5] Figures 2 through 5 show examples of resins from the third sampling of the irradiated EPICOR–II resins. Other SEM photomicrographs are presented in Reference 6.

The SEM photomicrograph of PF–8#1 resin (Figure 2) shows considerable damage to the resin bead. It shows a concentric type of bead cracking not observed during the previous samplings. The photomicrograph of PF–8#3 resin (Figure 3) shows a different type of cracking. Figure 3 shows two beads that have cracked. One also appears to have softened and been deformed by surrounding beads.

The SEM photomicrograph of PF–20 resin (Figure 4) shows a cracked bead that exhibits other damage partially hidden by an adjoining bead. The SEM photomicrograph of PF–8#2 phenolic resin (Figure 5) shows one particle obtained from the third sampling with slight cracking.

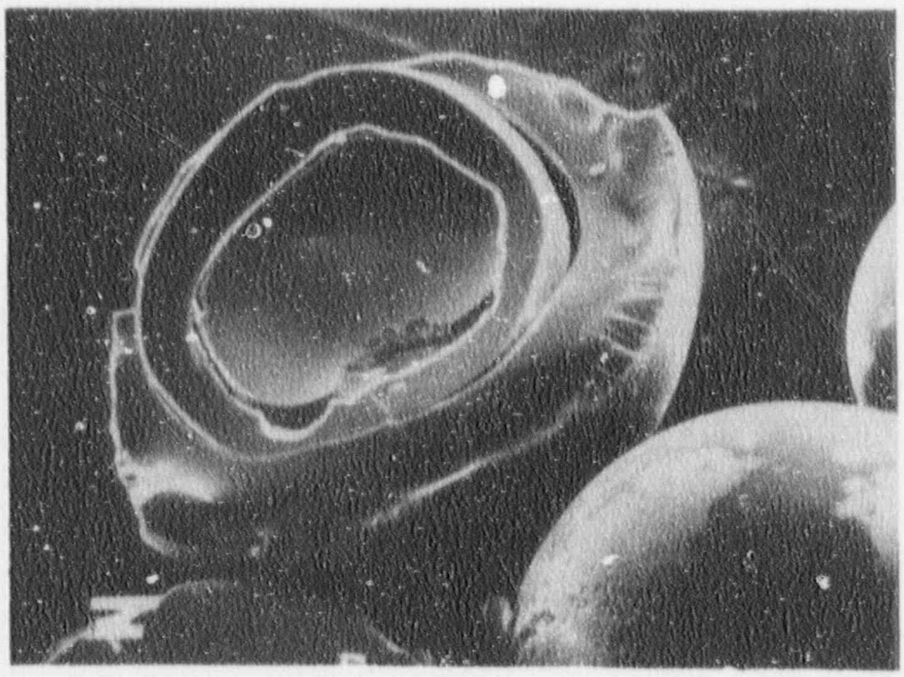

Figure 2. SEM photomicrograph of EPICOR–II strong acid cation resin sample from PF–8#1 showing a closeup of one unusually damaged bead (at 100 magnification).

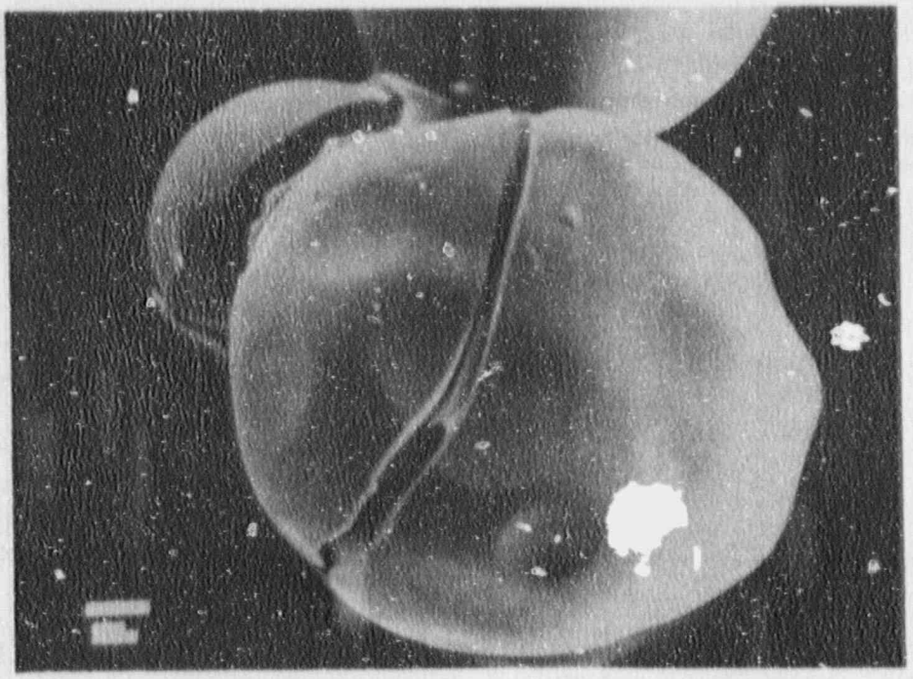

Figure 3. SEM photomicrograph of EPICOR–II strong acid cation resin sample PF–8#3 showing only one resin bead, which appears to have softened and dimpled (at 100 magnification).

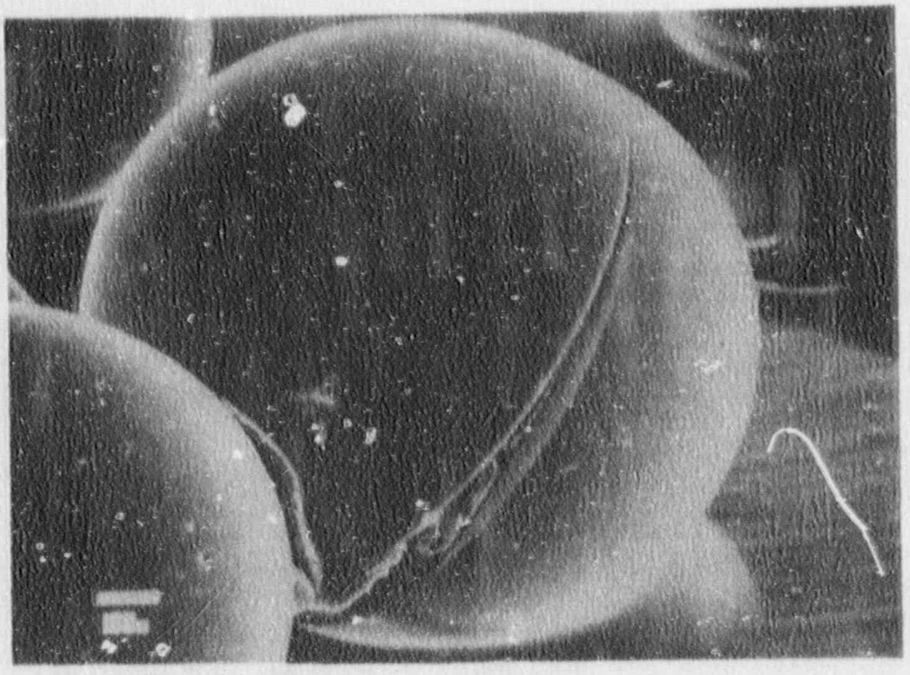

Figure 4. SEM photomicrograph of EPICOR–II strong acid cation resin sample PF–20 showing a closeup of a damaged resin bead (at 100 magnification).

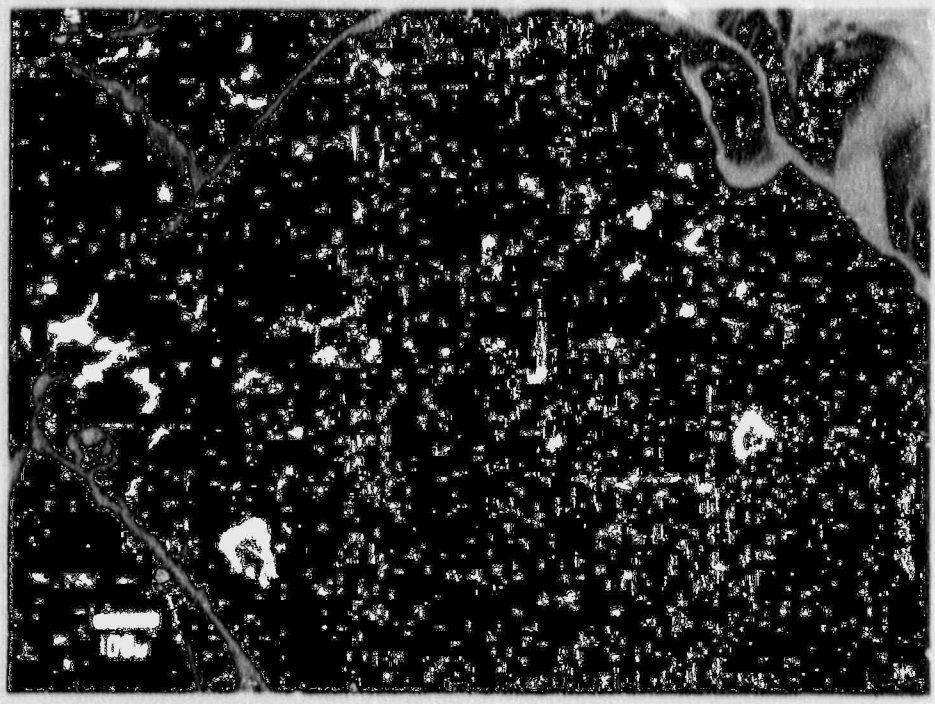

Figure 5. SEM photomicrograph of EPICOR–II phenolic cation resin sample PF–8#2 showing a closeup of one cracked resin particle (at 50 magnification).

Gamma–Ray Spectroscopy. During the third sampling, gamma–ray spectroscopy was used to determine the amount of radionuclides present in the aqueous solutions. Table 4 presents the amount of Cs–134 and Cs–137 found in the deionized water solutions, as well as the total cesium from the resin samples.

Table 4. Cesium measured in aqueous solutions from resins–third sampling

	Total mCi/100 mL Resin Sample		
Resin Sample No.	Cs–134	Cs–137	Total Cs
PF–8#1	650	74,089	74,139
PF–8#2	95	11,183	11,278
PF–8#3	1141	130,242	131,383
PF–20	930	108,215	109,145

Physical Observations. PF–8#1 and PF–8#3 resin samples both appeared dark orange to brown in color. The old unirradiated resin was still a light amber color. PF–20 resin samples also had a significant change in color (mostly orange). When the sampling was performed, the PF–8#1 and PF–8#3 resin samples also appeared to be sticky and tenacious toward the sampling wand. In several instances, the vacuum wand had to be rinsed in water to clear the vacuum tube.

Synopsis of Results. Table 5 presents results of the various analytical tests performed on the irradiated EPICOR–II resin samples. It should be noted that results in the table are expressed in terms of differences between values obtained from tests on the irradiated EPICOR–II resins from the first, second, and third samplings and the values obtained from tests on the unirradiated Epicor, Inc. supplied resins.

Table 5. Synopsis of results from analysis of EPICOR–II irradiated resin samples (first, second and third samplings)

Analytical Technique	Sample Number	Resin Sample: PF–8#1 Strong Acid Cation	PF–8#2 Phenolic Cation	PF–8?3 Strong Acid Cation	PF–20 Strong Acid Cation
ASTM Tests					
Water retention capacity	1,2,3	Increase/Increase/Increase	Increase/increase/Increase	No sample/Increase/Increase	increase/Increase/Increase
Backwashed and settled density	1,2,3	Decrease/Decrease/Decrease	Decrease/Decrease/Decrease	No sample/Decrease/Increase	Decrease/Decrease/Increase
Salt–splitting capacity	1,2,3	Decrease/Decrease/Decrease	Increase/Increase/Decrease	No sample/Decrease/Decrease	Decrease/No change/Decrease
Total exchange capacity	1,2,3	Decrease/Decrease/Decrease	No change/Decrease/Decrease	No sample/Decrease/Decrease	Increase/No change/Decrease
Infrared spectroscopy	1,2	No apparent changes in structure	No apparent changes in structure	No apparent changes in structure	No apparent changes in structure
Gas, liquid and supercritical fluid chromatography	1,2,3	No soluble products determined	No soluble products determined	No soluble products determined	No soluble products determined
$BaCl_2$ precipitation for sulfonic acid groups	1,2	Sulfonic acid groups are being lost	This resin contains no sulfonic acid	Sulfonic acid groups are being lost	Sulfonic acid groups are being lost
Inductively coupled plasma-atomic emission spectroscopy	3	Sulfonic acid groups are being lost	Some sulfonic acid groups found	Sulfonic acid groups are being lost	Sulfonic acid groups are being lost
Scanning electron microscopy observations	1	Resin bead cracking	Damage on a few particles	No sample	No damage noted
	2	Resin bead cracking	No damage noted	Resin bead cracking	No damage noted
	3	Resin bead cracking	Damage on one particle	Resin bead cracking	Damage to one bead

Table 5. (continued)

Analytical Technique	Sample Number	Resin Sample: PF–8#1 Strong Acid Cation	PF–8#2 Phenolic Cation	PF–8#3 Strong Acid Cation	PF–20 Strong Acid Cation
Visual observations	1	Nothing unusual	Contamination with PF–8#1 resin	No Sample	Nothing unusual
	2	Soak and rinse solutions were a brown color	Soak and rinse solutions were a brown color	Soak and rinse solutions were a brown color	Soak and rinse solutions were a brown color
	3	Soak and rinse solutions were a light brown color with sediment	Soak solution contained light brown sediment; contamination with PF–8#1 resin	Soak and rinse solutions were a light brown color with sediment	Soak and rinse solutions were a light brown color with sediment
Physical observations	1	Lack of flow during elution	Nothing unusual	No Sample	Nothing unusual
	2	Lack of flow during elution	Nothing unusual	Lack of flow during elution	Nothing unusual
	3	Lack of flow during elution	Nothing unusual	Lack of flow during elution	Lack of flow during elution

RESIN SOLIDIFICATION

Four EPICOR-II waste forms have been compression-tested. Samples with mass and contact radiation dose readings similar to those tested in 1984 were used.[15,16] One sample of each waste type (all organic and organic with zeolite) in each binder (Portland cement and vinyl ester-styrene) were tested. On June 28, 1990, the seven-year old waste forms were compression-tested per ASTM C39, using a Tinuis Olsen 60,000-lb testing machine calibrated on June 21, 1990. The test results are presented in Table 6.

The specimens were tested dry using poured epoxy leveling caps. The concrete specimens broke in a column cracking pattern with very little flaking. The VES specimens bulged in the center of the cylinders. The column breaking took place within the bulging center area. The VES specimens seemed to be very ductile. The tested specimens are shown in Figures 6, 7, 8, and 9.

Figure 10 shows the above data and previous compressive strength test results (taken at ages after fabrication of 1 month and 24 months) relative to specimen age. All sample types experienced increase in strength with age. The oldest cement containing organic/inorganic ion exchange resins exhibited an unusually high strength. It is noted that only one specimen of each of the four types was tested and the data scatter could account for this apparent anomaly. Further testing planned over the next two years should clarify these results.

These waste form specimens have been stored in shielded drums throughout the project. Dose measurements indicate that the specimens have experienced a total gamma dose of 1.5×10^6 to 2.5×10^6 rad. That dose has caused serious embrittlement of the polyethylene specimen containers, which are beginning to crack. The VES is evidently responding to the high dose by becoming more ductile.

Table 6. Compression test results of seven-year old EPICOR-II waste forms

Specimen	Material of Construction	Radiation at Contract (R/h)	Ultimate Load (lb)	Compress Strength (psi)
C1-23	Organic ion exchange in Portland cement	7	9,890	3667
C2-A7	Organic/inorganic ion exchange in Portland cement	12	17,000	6404
D1-A2	Organic ion exchange in Dow vinyl ester-styrene	25	8,830	3364
D2-27	Organic/inorganic ion exchange in Dow vinyl ester-styrene	28	11,290	4333

Figure 6. Sample C1–23 organic ion exchange resin in Portland cement after compression test.

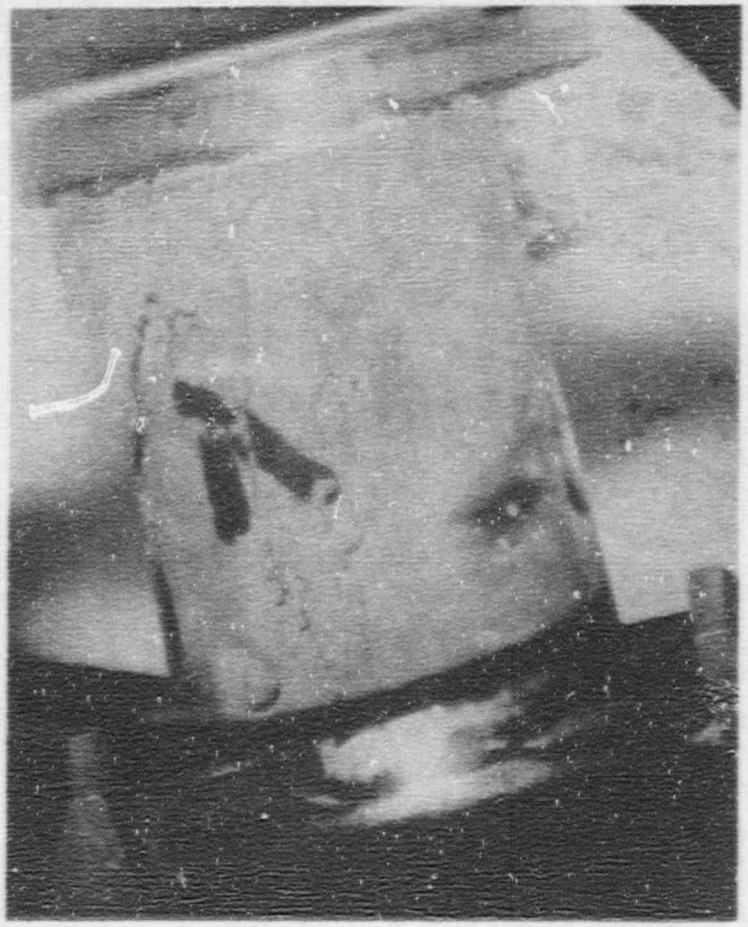

Figure 7. Sample C2A–7 organic/inorganic ion exchange resin in Portland cement after compression test.

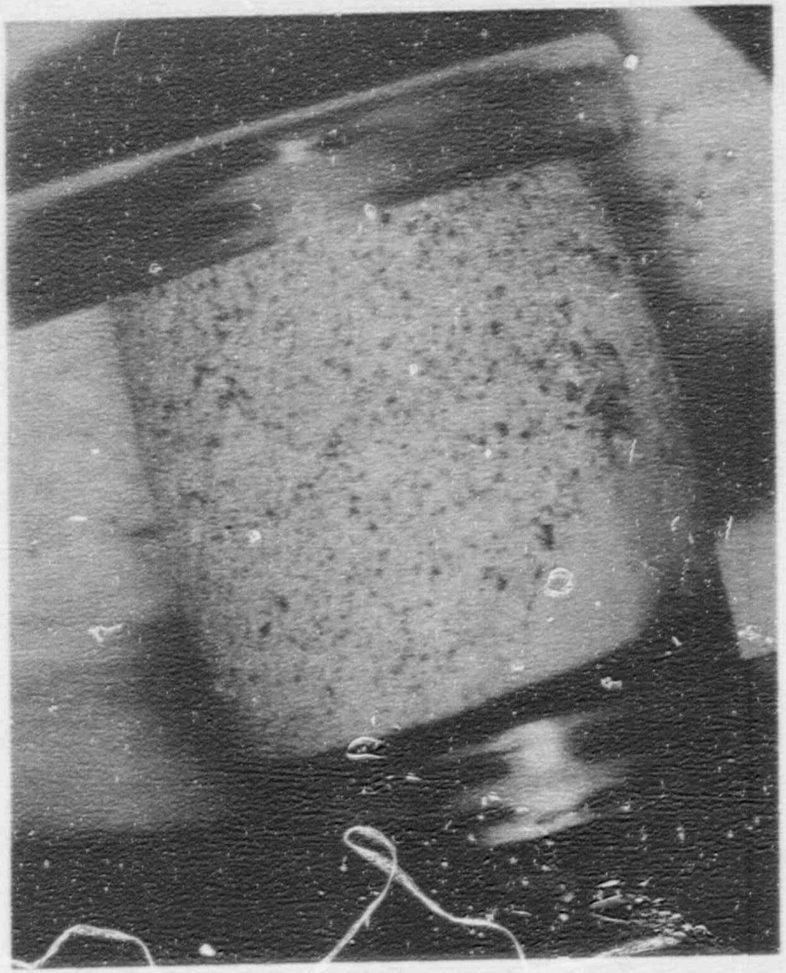

Figure 8. Sample DIA–2 organic ion exchange resin in vinyl ester–styrene after compression test.

Figure 9. Sample D2–27 organic/inorganic ion exchange resin in vinyl ester–styrene after compression test.

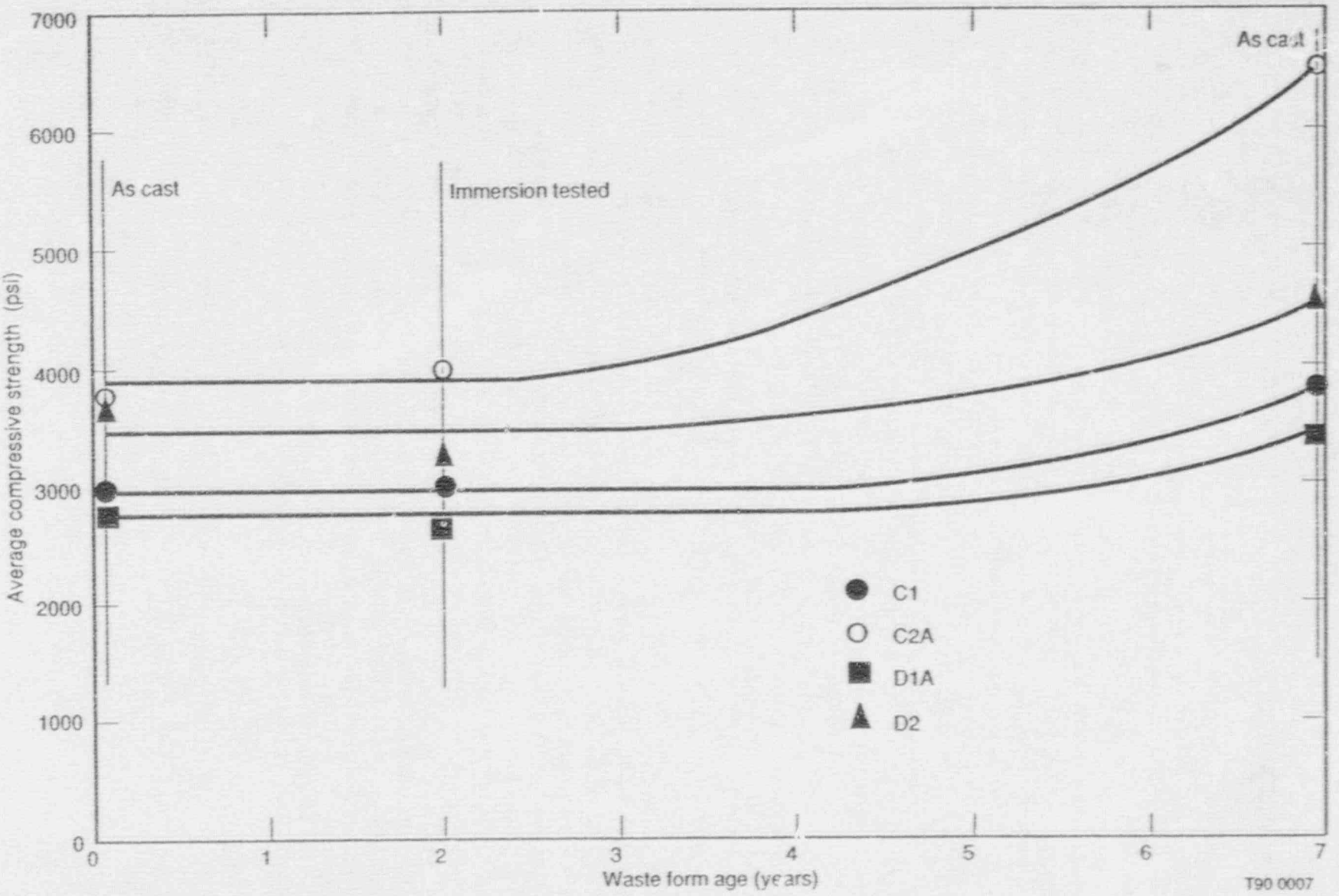

Figure 10. Variation of waste form compressive strength with age caused by self–irradiation and curing.

FIELD TESTING

Materials and Methods

Field testing is described in the following section of this report. Included are descriptions of waste forms, lysimeters, and procedures used to collect and analyze data.

Experiment Description. Solidified waste forms containing EPICOR–II ion exchange resin waste are currently being field–tested using lysimeters. Lysimeter sites have been established at Oak Ridge National Laboratory (ORNL) and Argonne National Laboratory–East (ANL–E). Instrumentation within each of the five lysimeters at each site includes porous cup soil–water samplers and soil moisture/temperature probes. The probes are connected to an onsite DAS, which also collects data from a field meteorological station located at each site. A detailed description of the lysimeters and their installation and data from the first four years of operation are contained in earlier reports.[17,18,19,20,21]

Description of Waste Forms. Waste forms used in the field test are composed of solidified EPICOR–II prefilter resin wastes. Two waste formulations are used in the solidification project (Table 7). Type A is a mixture of synthetic organic ion exchange resins from PF–7 (phenolic cation, strong acid cation, and strong base anion resins), while Type B is a mixture of synthetic organic ion exchange resins from PF–20 (strong acid cation and strong base anion resins) with an inorganic zeolite. Waste Type A contains 25% Sr–90, while Type B contains about 1% Sr–90. Of the other radionuclides in those wastes, Cs–137 and Cs–134 are the major constituents, with Sb–125 found in trace amounts.

Portland Type I–II cement and VES were used to solidify both types of resin wastes. Individual waste forms were manufactured by allowing a mixture of solidification agent and resin waste to solidify in polyethylene molds that were 4.8–cm in diameter by 10.2–cm high. Enough of the mixture was added to each vial to produce waste forms with an average diameter of 4.8 cm and height of 7.6–cm (137.5 cm^3). A complete description of waste form manufacture is given in Reference 15. Bench testing of similar waste forms, per the requirement of the Branch Technical Position on Waste Form, is described in Reference 16.

Description of Lysimeters. The lysimeters are designed as self–contained units that can be easily disposed at the termination of the field test experiment. A total of ten lysimeters are used, with five placed at each field site. Each lysimeter is a right circular cylinder (0.91 m in diameter by 3.12 m in height) constructed of 12–gage, 316 L stainless steel (Figure 11). Internally, the lysimeter is divided into two sections, the upper volume being 1532 L and the lower being 396 L. A 3.8–cm, Schedule 40, stainless steel pipe serves as an access to the lower compartment. Soil, instrumentation, and waste forms are contained in the upper compartment, while the lower compartment serves as a leachate collector.

Table 7. Lysimeter waste form composition

Lysimeter Number	Fill Material	Waste Form Description	Prefilter Number
1	Soil	Cement with Type A waste	PF–7
2	Soil	Cement with Type B waste	PF–24
3	Soil	VES with Type A waste	PF–7
4	Soil	VES with Type B waste	PF–24
5 ANL–E	Silica oxide	Cement with Type A waste	PF–7
5 ORNL	Silica oxide	Cement with Type B waste	PF–24

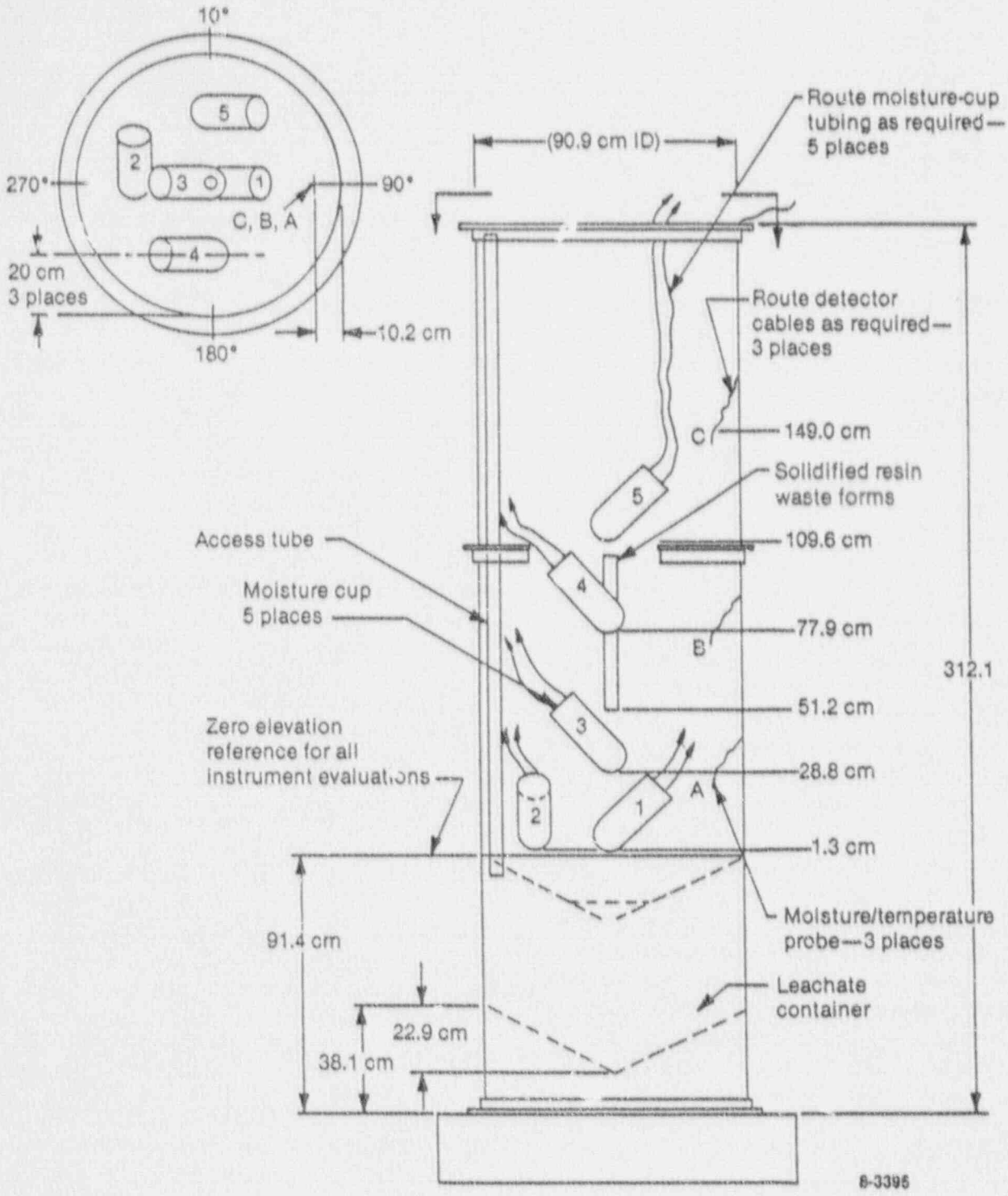

Figure 11. EPICCR–II lysimeter vessel component locations.

Four lysimeters at each field site are filled with soil; the remaining one is a control filled with an inert silica sand.[17] Two different soils were used. One was representative of Midwestern soils, the other was intended to approximate soil found at Barnwell, South Carolina. ANL–E used local indigenous soil that fits the NRC criteria for the midwestern soil. It is a Morley silt loam, with the surface layer removed. The resulting subsurface soil is a clay loam. Soil at the ORNL was not found to be a suitable substitute for Barnwell soil; therefore, acceptable soil was transported to the

ORNL from the Savannah River Plant adjacent to the Barnwell facility in South Carolina.

Each of the lysi ers is consecutively numbered 1 throug , with 1 through 4 containing soil and number 5 being the sand–filled control. The waste form type found in each lysimeter is given in Table 7.

Data Retrieval and Analysis. Electrical impulses from the environmental instruments are collected by, processed in, and stored by the DAS for periodic retrieval. The DAS processes input into recognizable data using programmable steps. Outpu, from the soil moisture probes, for example, is processed by a polynomial equation that was derived from laboratory calibration of the probes.[17]

Data output from the DAS is stored on a cassette tape and, after retrieval, is translated to an IBM PC–compatible disk file. Hard copy from these files is provided either graphically or in a printed format. The graphic display presents data over an extended time peri d. The graphic r esentation was used for this repor.

Water from each lysimeter is drawn from porous cup soil–water samplers and lysimeter leachate collection compartments at least quarterly. These water samples are analyzed routinely for gamma–producing nuclides and, as required, for the beta–producing nuclide Sr–90. Water analyses are performed at ANL–E by the Environmental Services Laboratory and at ORNL by the Environmental Radio Analysis Laboratory. Both of these laboratories have a traceable quality assurance program and use accepted analytical procedures for nuclide determination.

Results and Discussion

This report contains DAS data from ANL–E and ORNL obtained from July 1989 through July 1990. In addition, information on water balance and nuclide content in soil water and leachate is a compilation of data from the initiation of the project (ANL–E, August 1, 1985; ORNL, June 1, 1985) through June 1990. Much of the data is displayed in graphic format so that information can be correlated easily with time.

Weather Data. Precipitation, air temperature, wind speed, and relative humidity, as recorded by the ANL–E and ORNL DAS systems during the 12–month reporting period, are presented in Figures 12 through 19. Total official precipitation (measured by reference rain gauges near each site) for the period was 100.8 cm at ANL–E and 153.3 cm at ORNL. ANL–E, for the first time since 1985, was well above the normal annual rainfall of 85.2 cm,[22] while ORNL was 110% of the normal annual rainfall of 138.8 cm.[23] This is the second time in four years that ORNL has equalled or exceeded the normal amount of yearly precipitation. The monthly precipitation pattern for each site can be seen from the histograms in Figures 12 and 16. Figure 20 shows the cumulative pattern of precipitation for both sites since the initiation of field work. By the end of this reporting period, there had been a cumulative total of 426.7 cm at ANL–E while ORNL had received a total of 631.2 cm.

Air temperature data from ANL–E (Figure 13) show that there were periods of freezing temperatures from mid–November 1988 until near mid–April 1990. In contrast to previous years, ORNL experienced a period of freezing temperatures from mid–November until mid–January and again in mid–February (Figure 17).

Lysimeter Soil Temperature Data. Soil temperature and moisture sensors are physically located within a common housing or probe. These probes are located at three elevations (149, 77.9, and 28.8 cm, as measured from the bottom of the soil column) within each lysimeter. The function of these probes is to provide data on whether or not the buried waste forms experience freezing temperatures and if the surrounding soil is moist. Because all of the soil lysimeters at each site are exposed to the same environment, the current placement of probes provides a planned redundancy in data collection. Therefore, as long as there are functioning probes in any of the soil

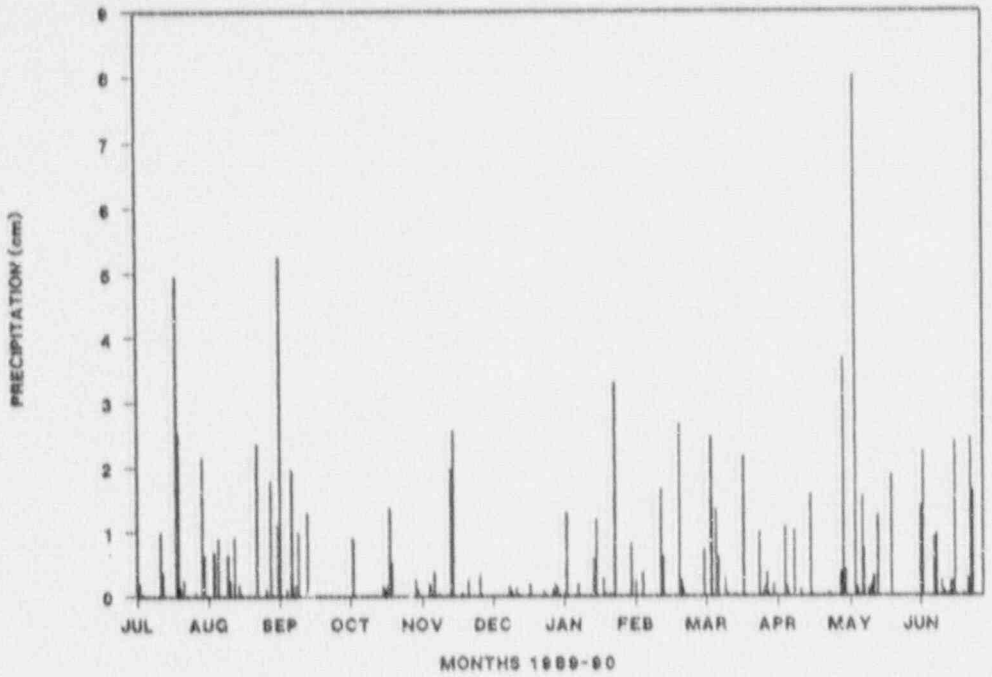

Figure 12. ANL–E weather data–precipitation.

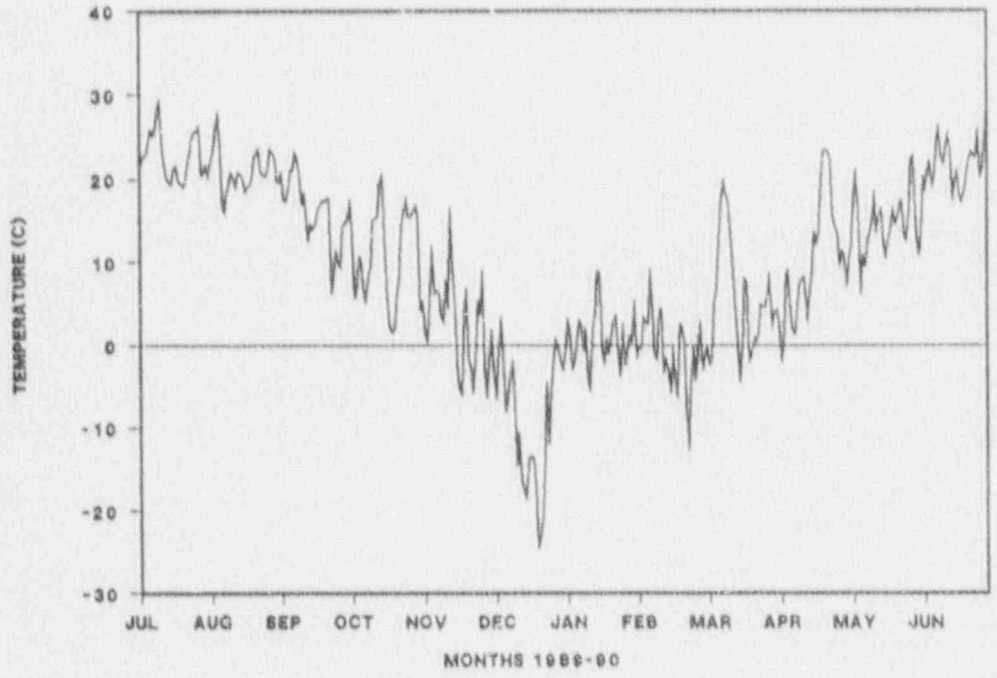

Figure 13. ANL–E weather data–air temperature.

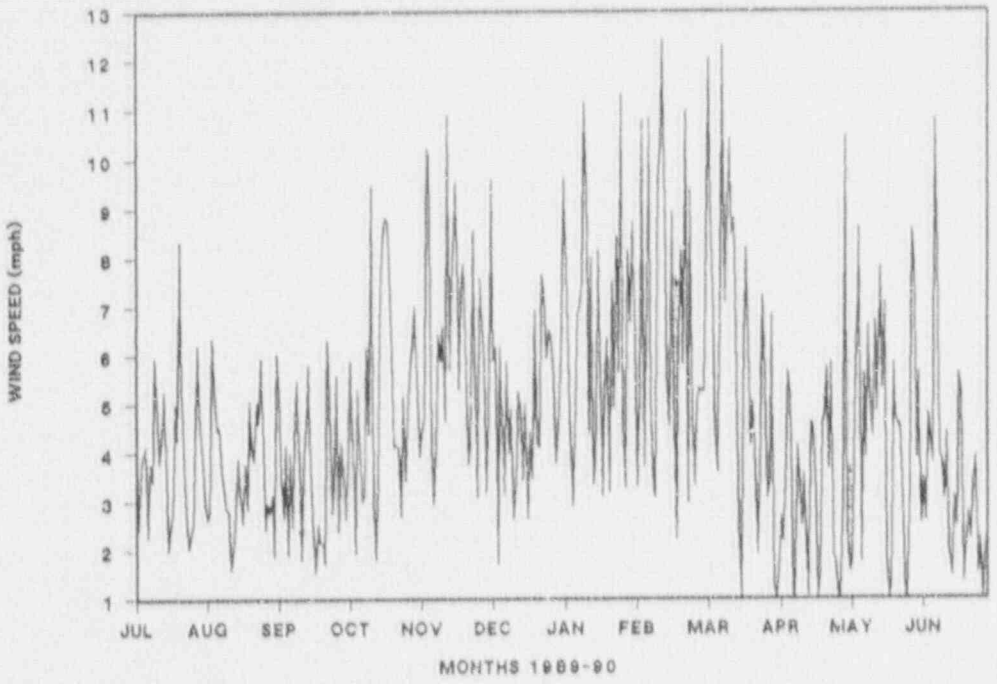

Figure 14. ANL–E weather data–wind speed.

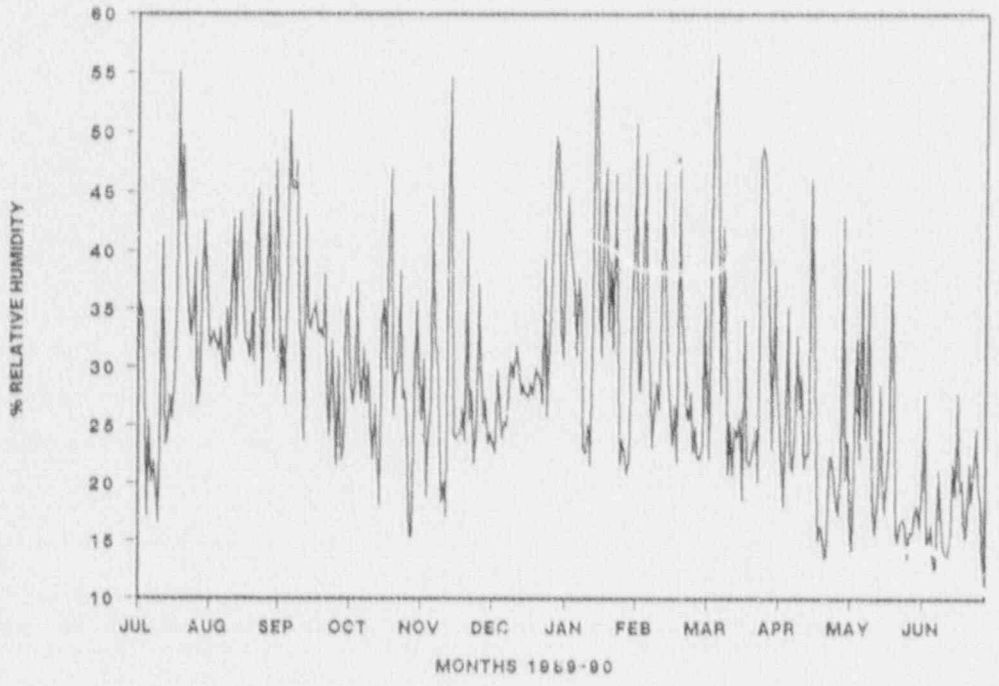

Figure 15. ANL–E weather data–relative humidity.

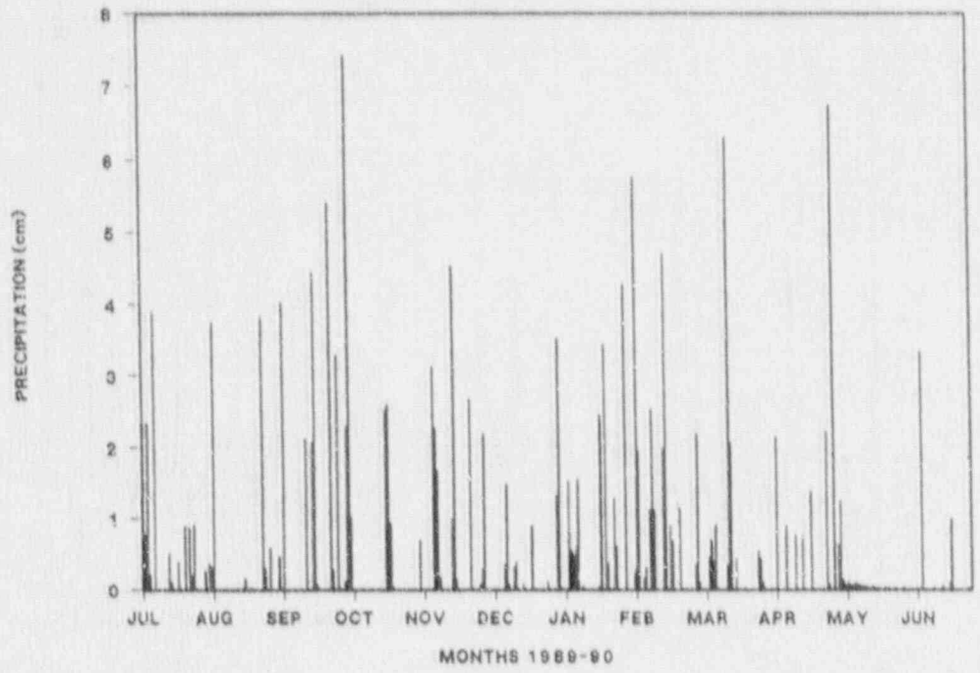

Figure 16. ORNL weather data–precipitation.

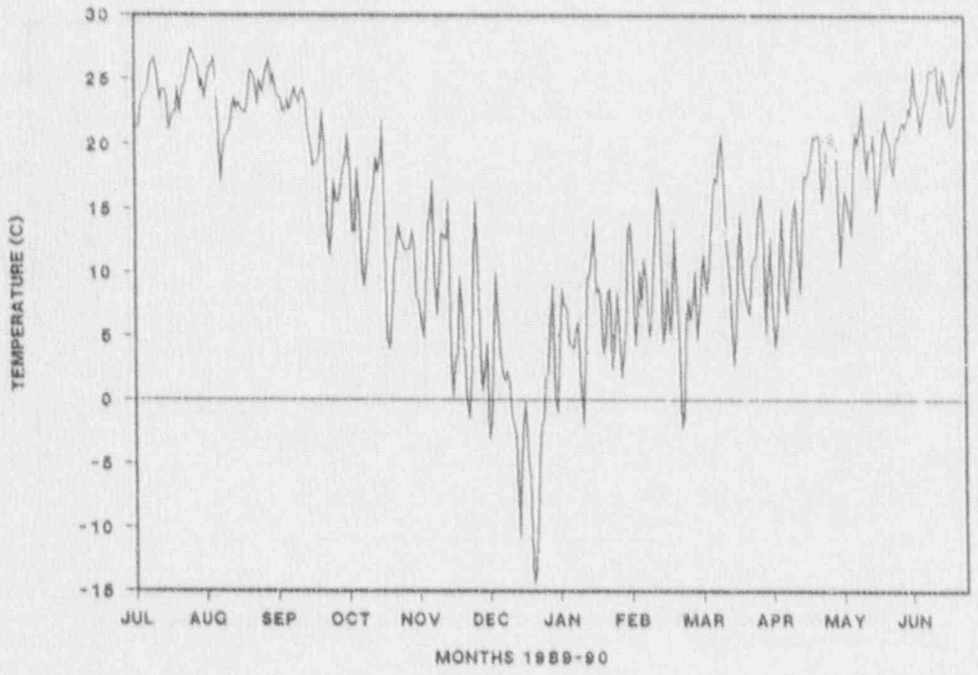

Figure 17. ORNL weather data–air temperature.

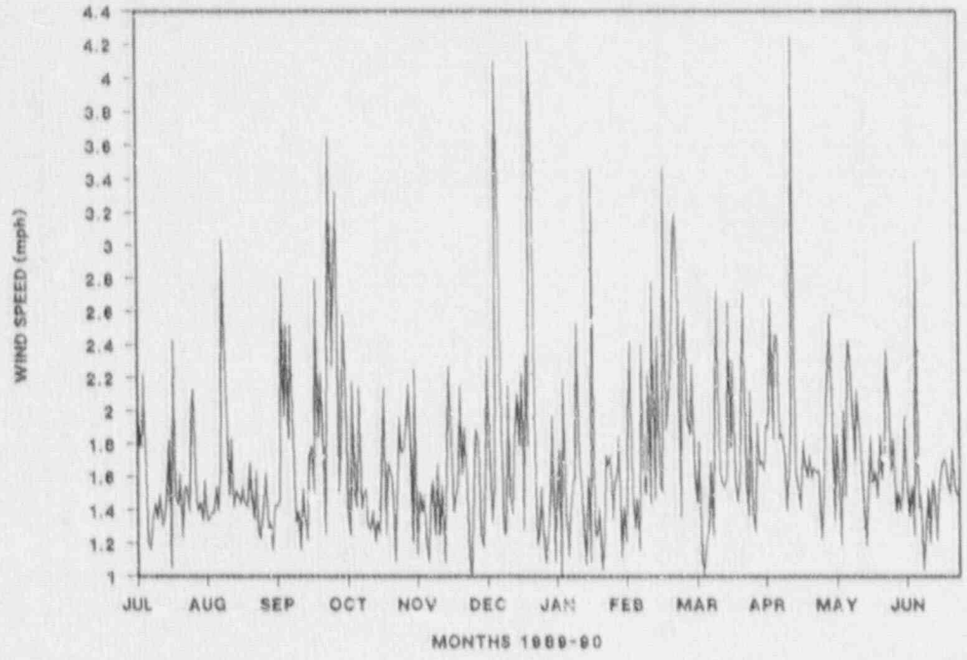

Figure 18. ORNL weather data–wind speed.

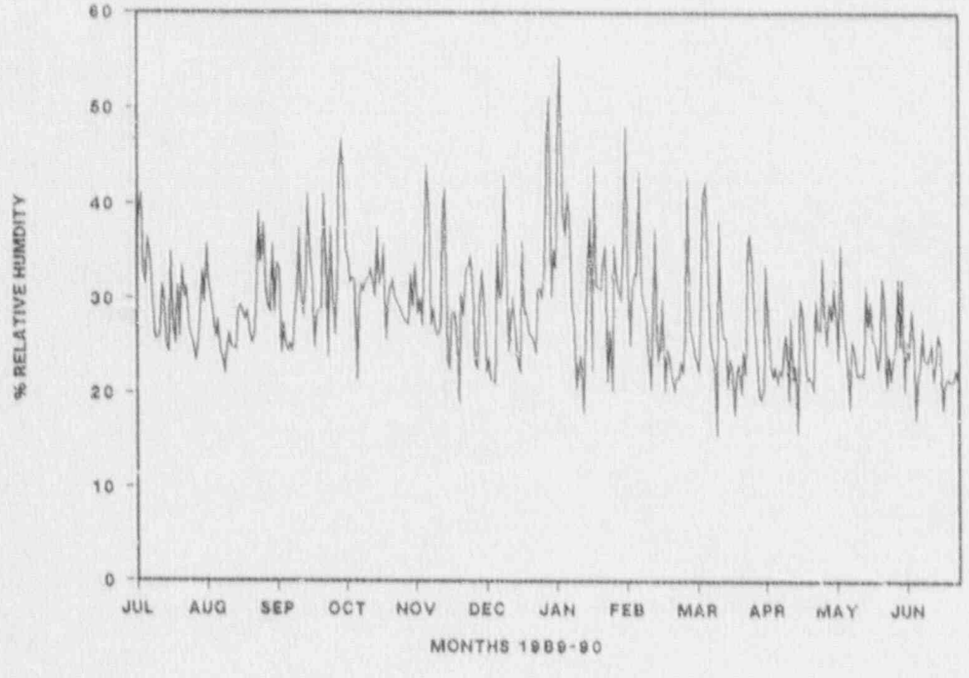

Figure 19. ORNL weather data–relative humidity.

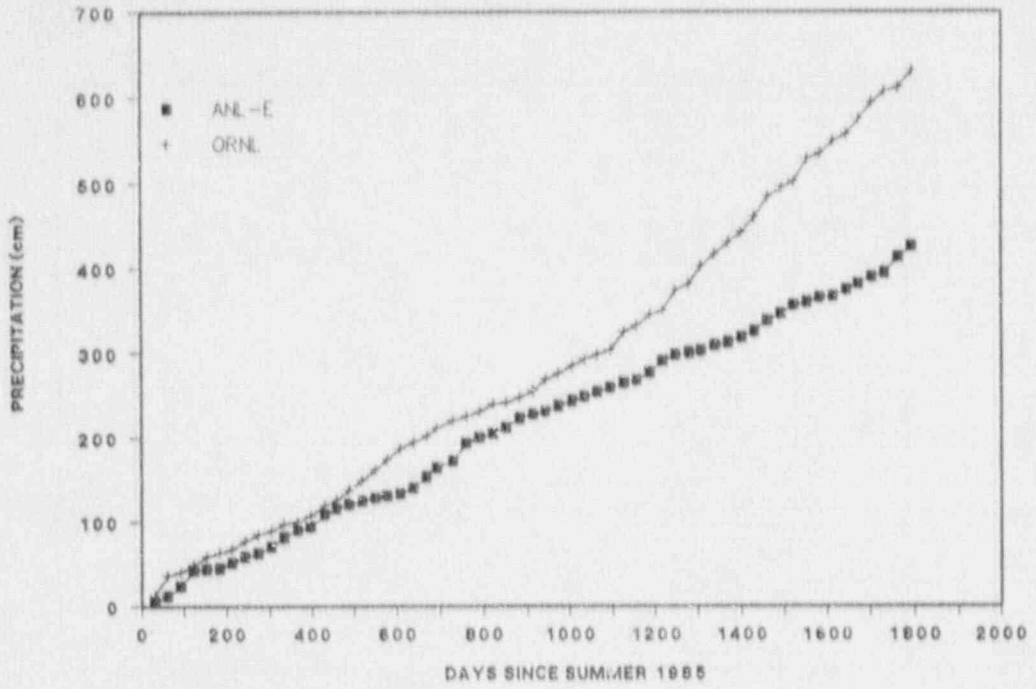

Figure 20. ANL–E and ORNL cumulative precipitation.

lysimeters at each site, sufficient data to satisfy reporting criteria will be available. In addition, temperature data collected during the years of extended service life of the probes will serve as a useful climatological reference for assessing waste form performance in future years.

The lysimeter soil temperature data recorded at ANL–E and ORNL during the reporting period are shown in Figures 21 through 29. Except for ANL–E lysimeter 1 at the 149 cm elevation in January and again in February, at no time during the reporting period was a freezing temperature recorded at any depth by a functioning temperature probe within a lysimeter. A direct correspondence can be seen between air temperature and soil temperatures at both locations.

As stated in past reports, there have been a number of temperature probe failures at ANL–E. During the last two reporting periods, it was obvious that the temperature probes in ANL–4 failed; therefore, data from these probes have not been included in the report. During the 1989 reporting period, it appeared that the probes in ANL–3 were not functioning properly. Further deterioration of these probes and one in ANL–5 has been seen during this reporting period. From past experience, it would appear that the probes have been damaged by corrosion of the metal parts.[19] At the present time, a more damage–resistant replacement for these probes has not been found. All temperature probes at ORNL still appear to be functional.

Lysimeter Soil Moisture Data. Data from the moisture probes at both ANL–E and ORNL, shown in Figures 30 through 39, indicate that the lysimeter soil columns at both sites have remained moist during the reporting period. The probe output from the soil column of each lysimeter over time (as determined by averaging the outputs of the three probes in each lysimeter) showed that the variation in moisture detected for the lysimeters at each site was relatively similar and not excessive. There was a coefficient of variation of 21.3% at ANL–E and 7.8% at ORNL. The probes continue to serve their original purpose of providing some indication of the status of lysimeter soil moisture. As was mentioned in the section on soil temperature, some of the probes at ANL–E are no longer functioning.

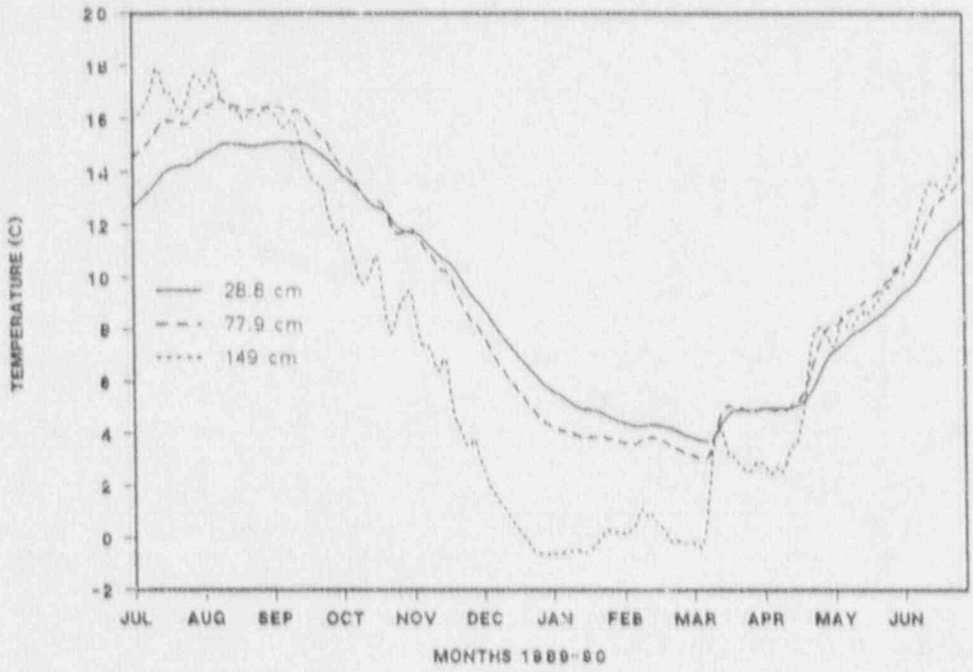

Figure 21. ANL–E lysimeter 1 soil temperature.

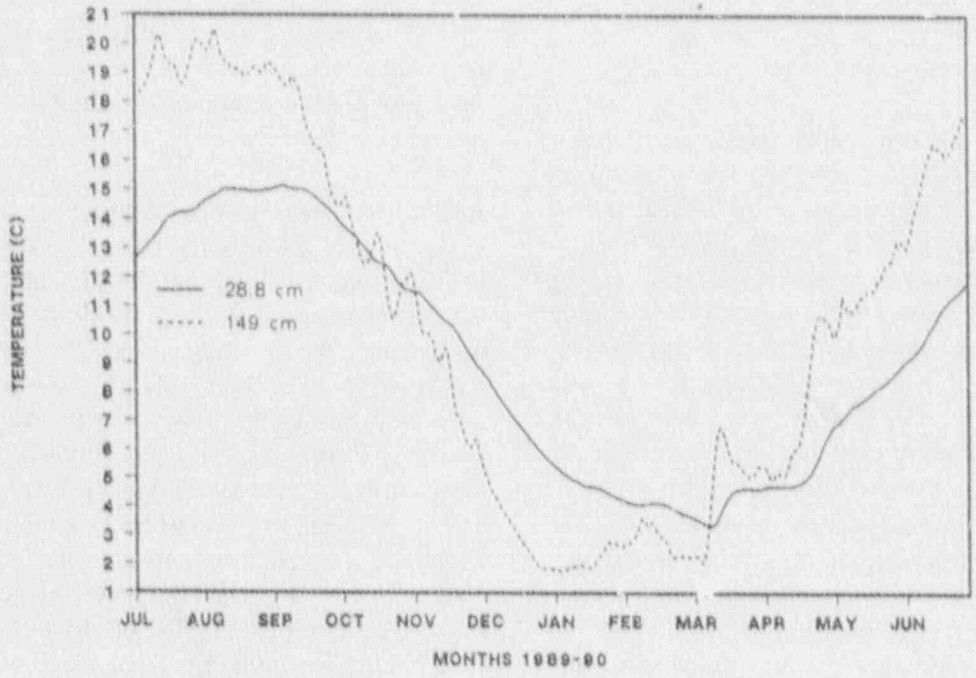

Figure 22. ANL–E lysimeter 2 soil temperature.

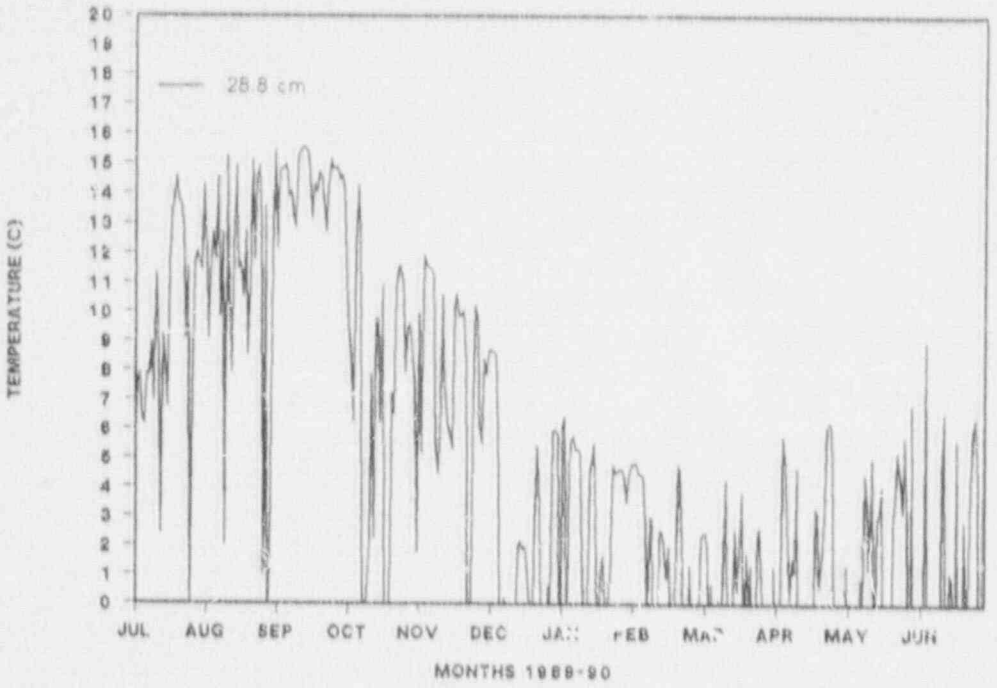

Figure 23. ANL–E lysimeter 3 soil temperature.

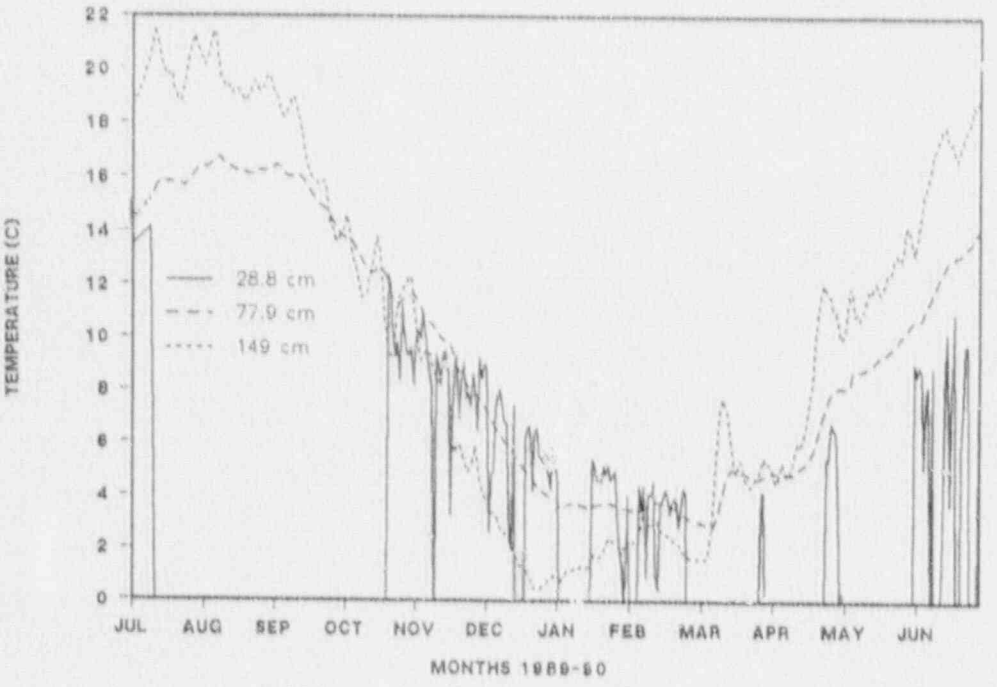

Figure 24. ANL–E lysimeter 5 soil temperature.

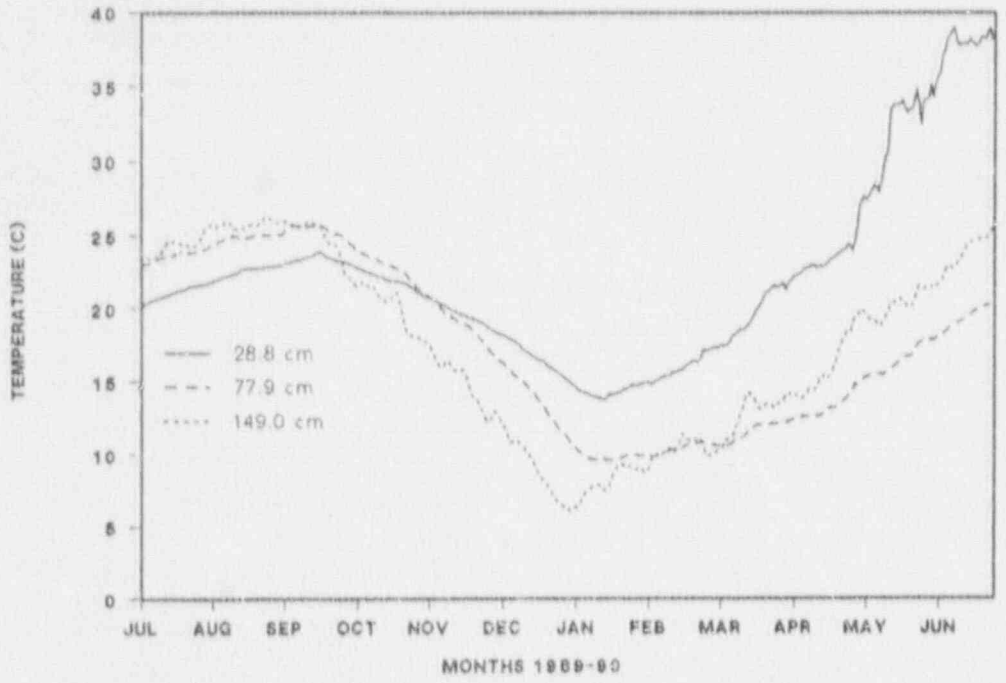

Figure 25. ORNL lysimeter 1 soil temperature.

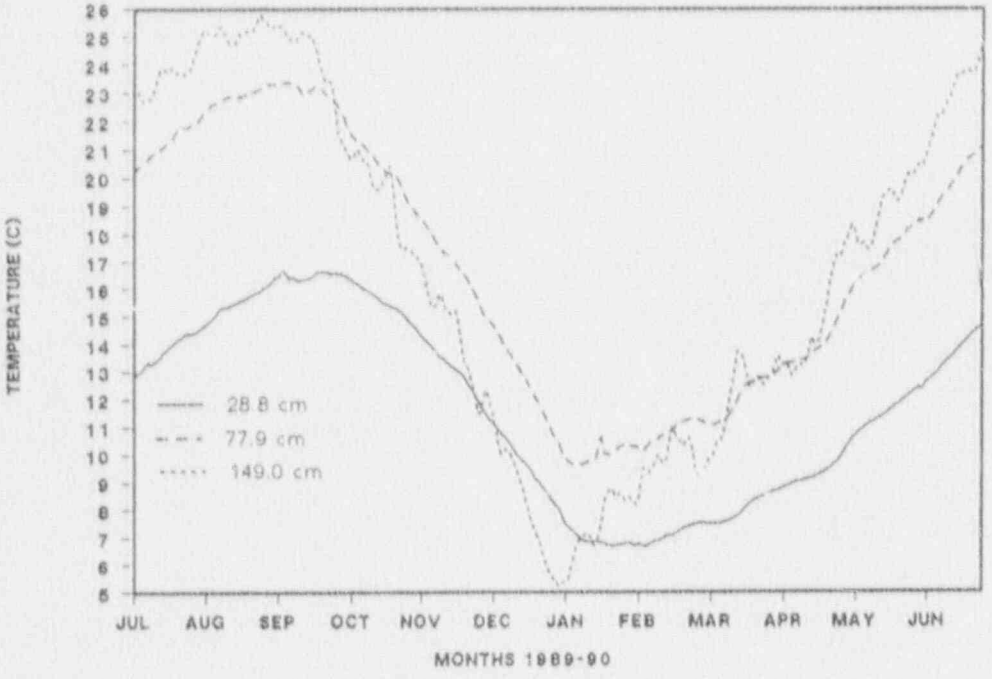

Figure 26. ORNL lysimeter 2 soil temperature.

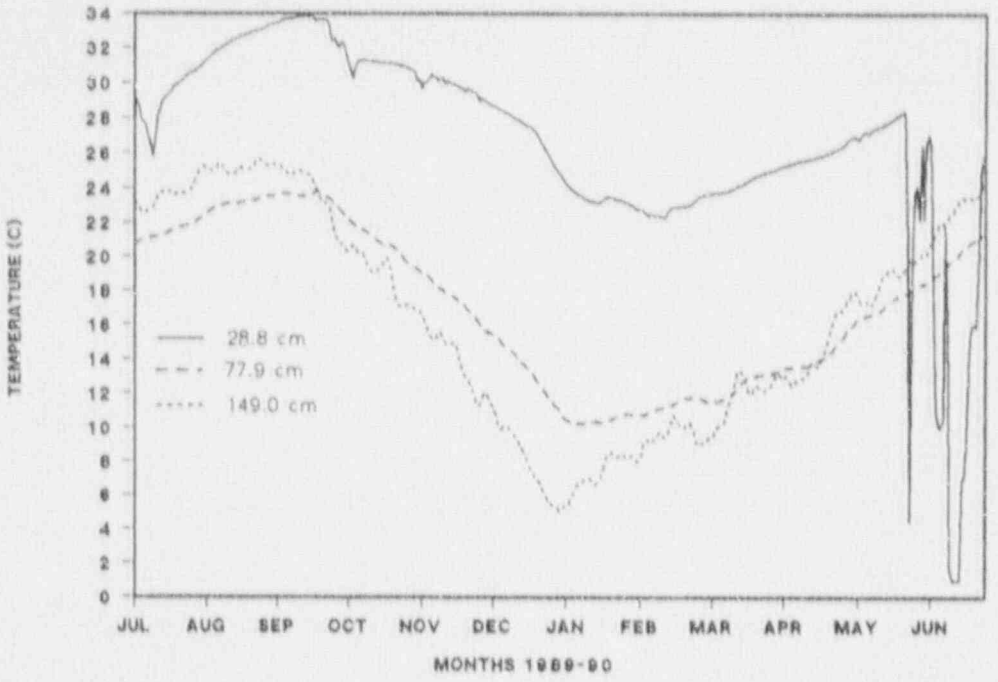

Figure 27. ORNL lysimeter 3 soil temperature.

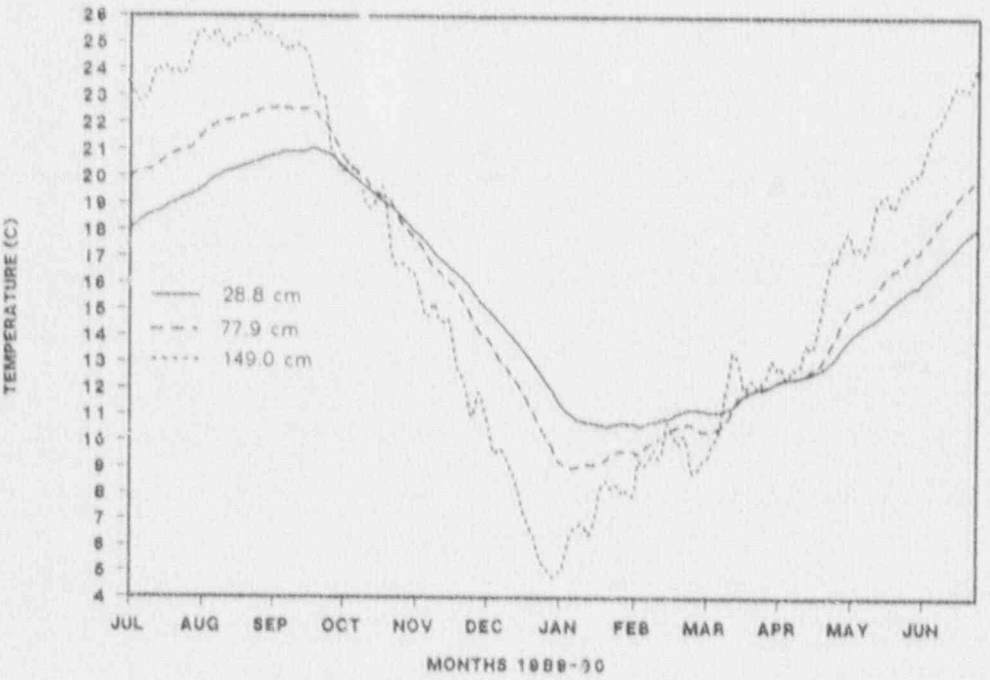

Figure 28. ORNL lysimeter 4 soil temperature.

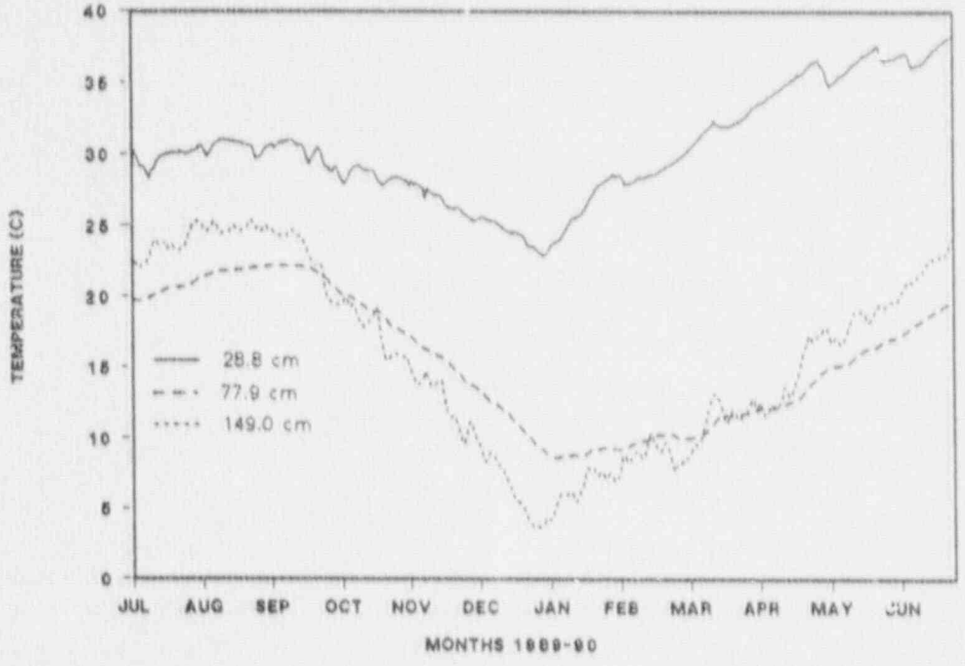

Figure 29. ORNL lysimeter 5 soil temperature.

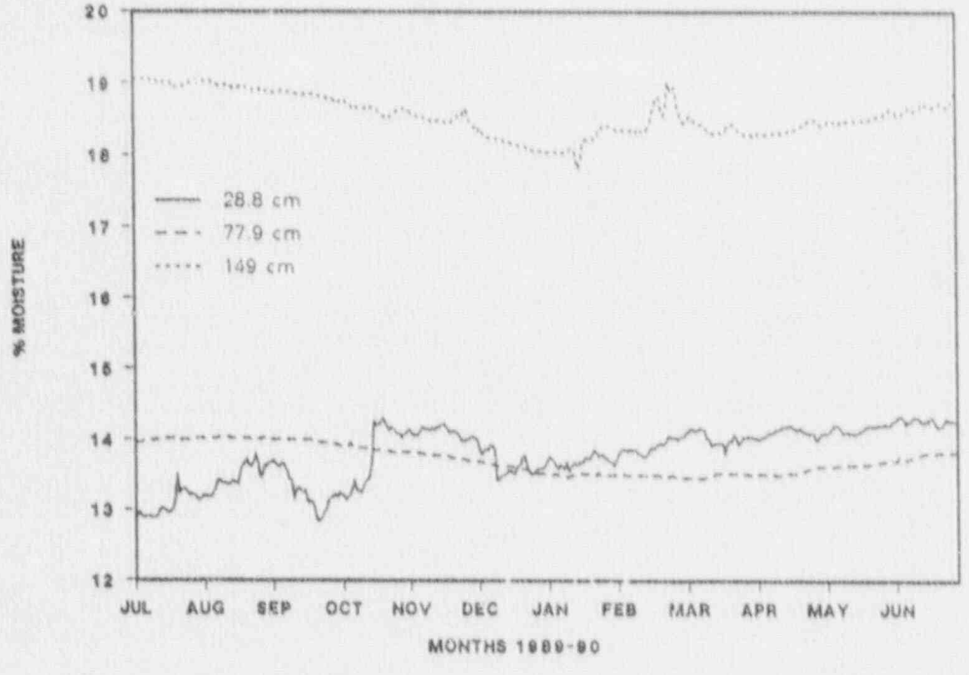

Figure 30. ANL-E lysimeter 1 soil moisture.

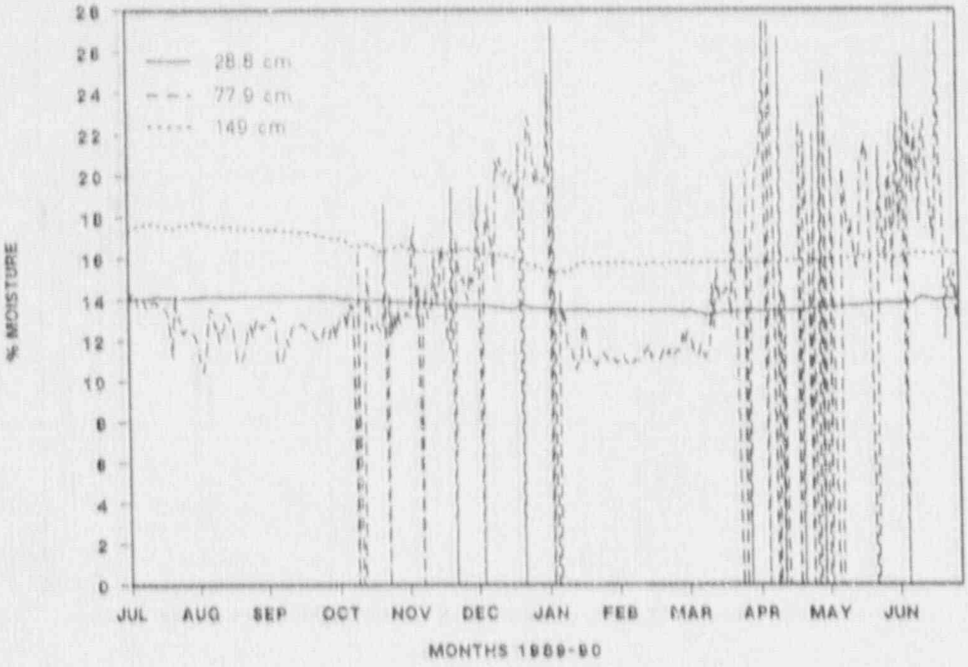

Figure 31. ANL–E lysimeter 2 soil moisture.

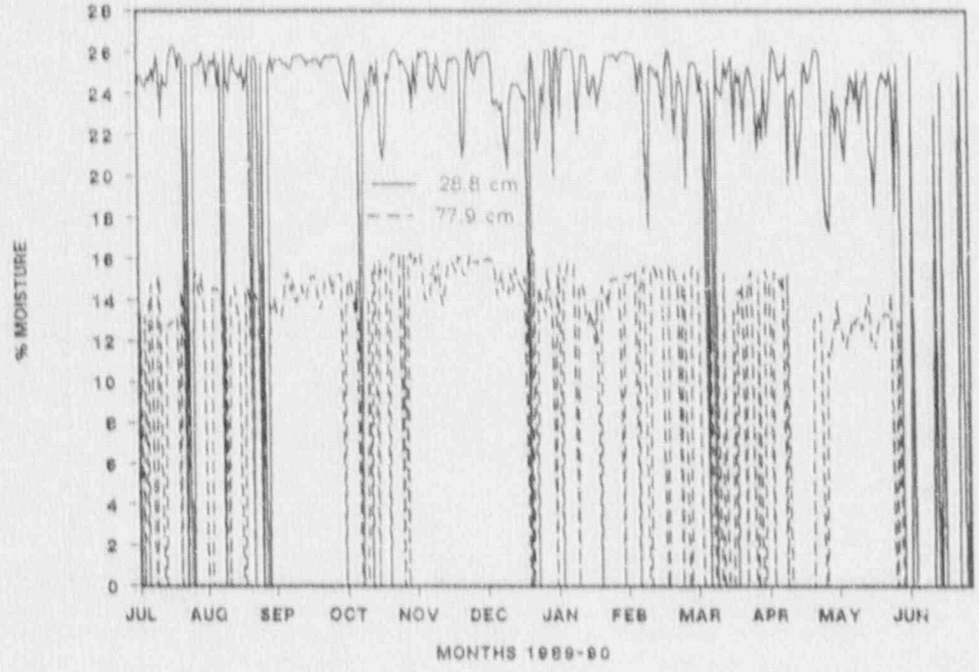

Figure 32. ANL–E lysimeter 3 soil moisture.

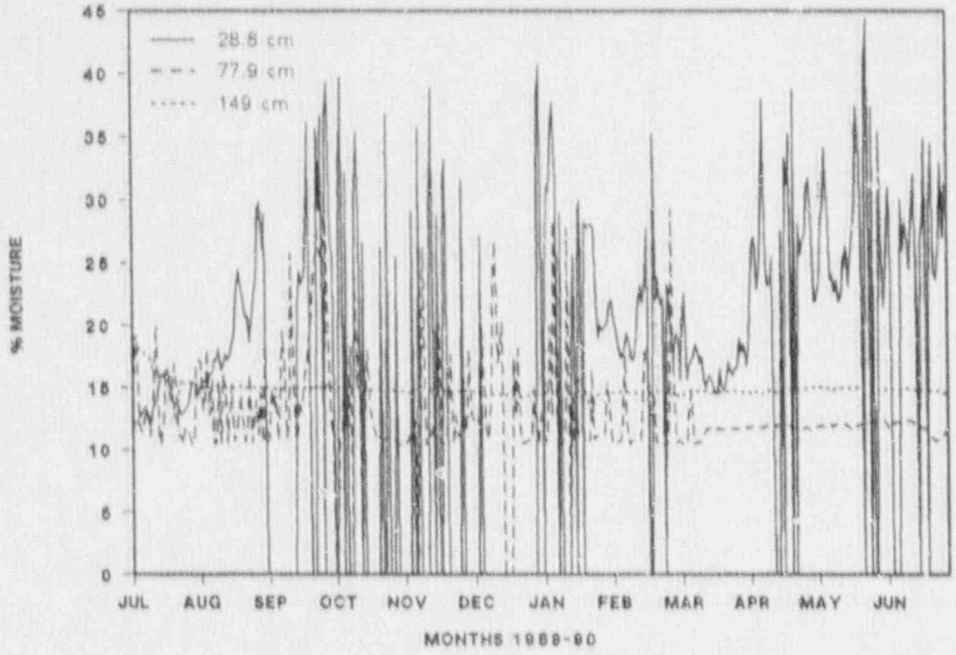

Figure 33. ANL–E lysimeter 4 soil moisture.

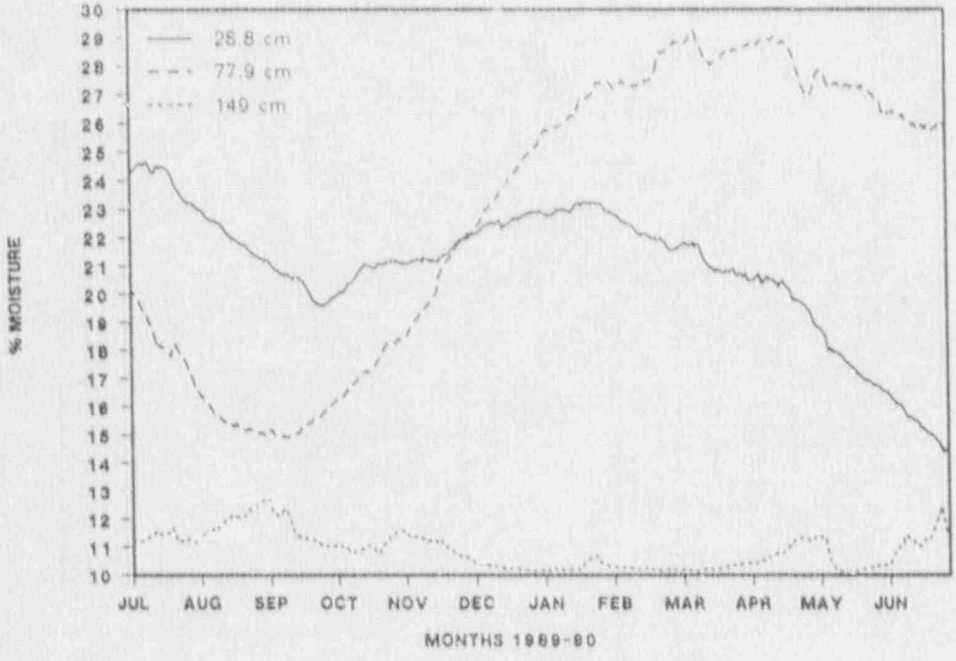

Figure 34. ANL–E lysimeter 5 soil moisture.

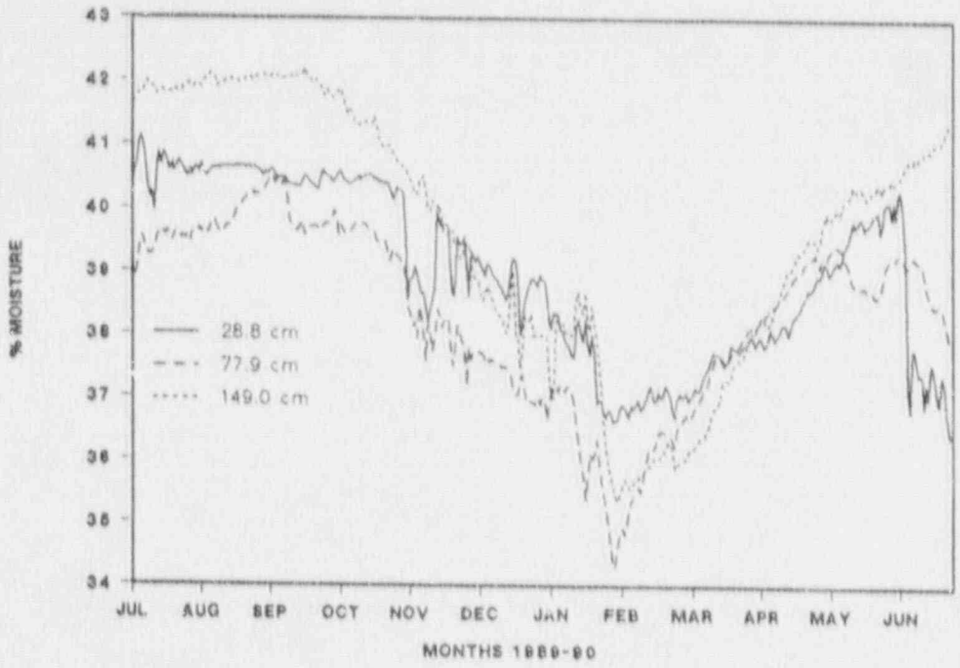

Figure 35. ORNL lysimeter 1 soil moisture.

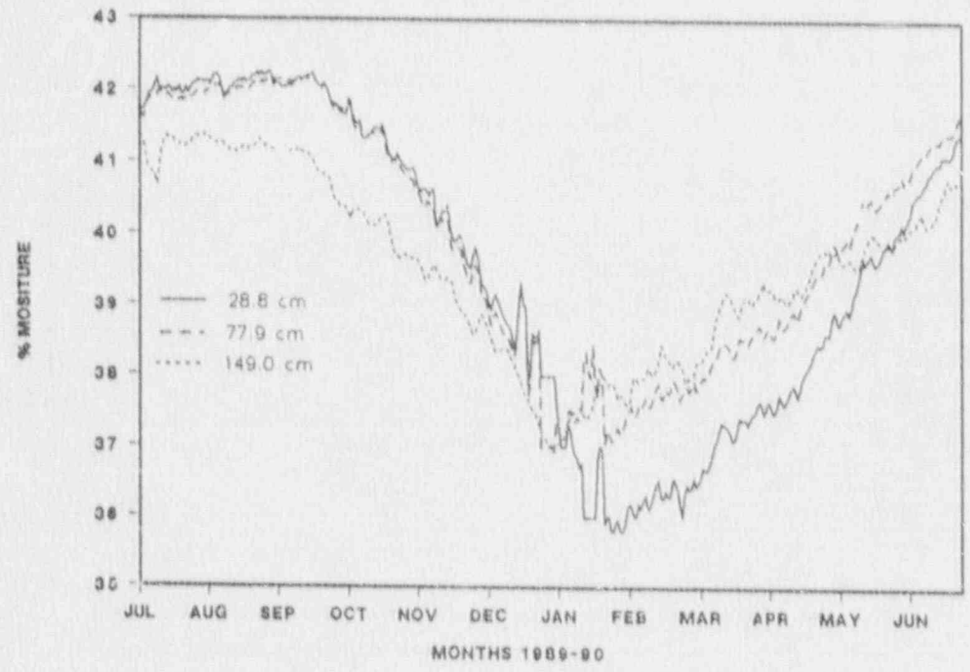

Figure 36. ORNL lysimeter 2 soil moisture.

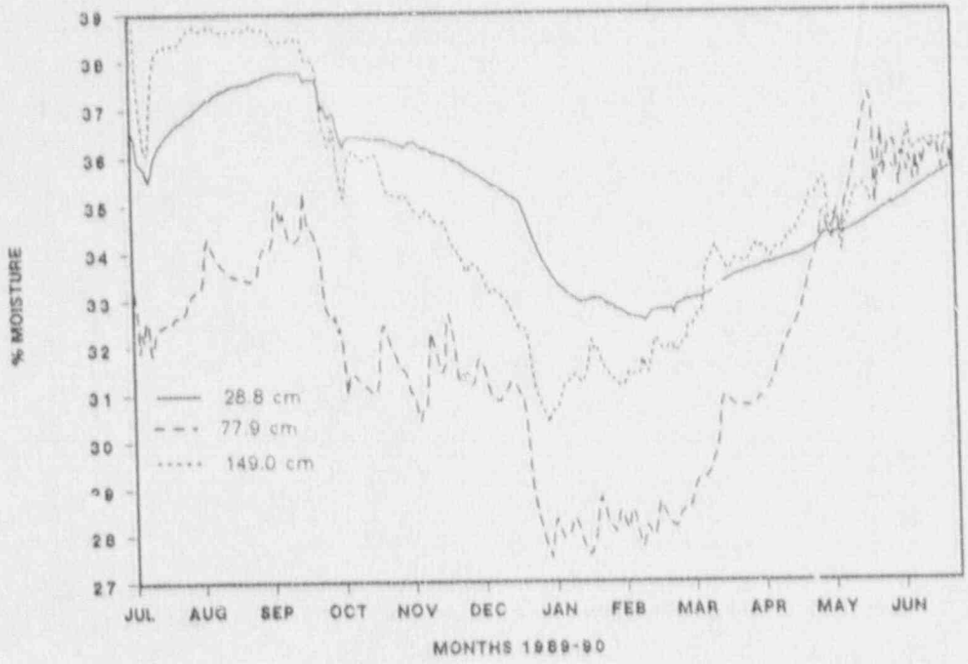

Figure 37. ORNL lysimeter 3 soil moisture.

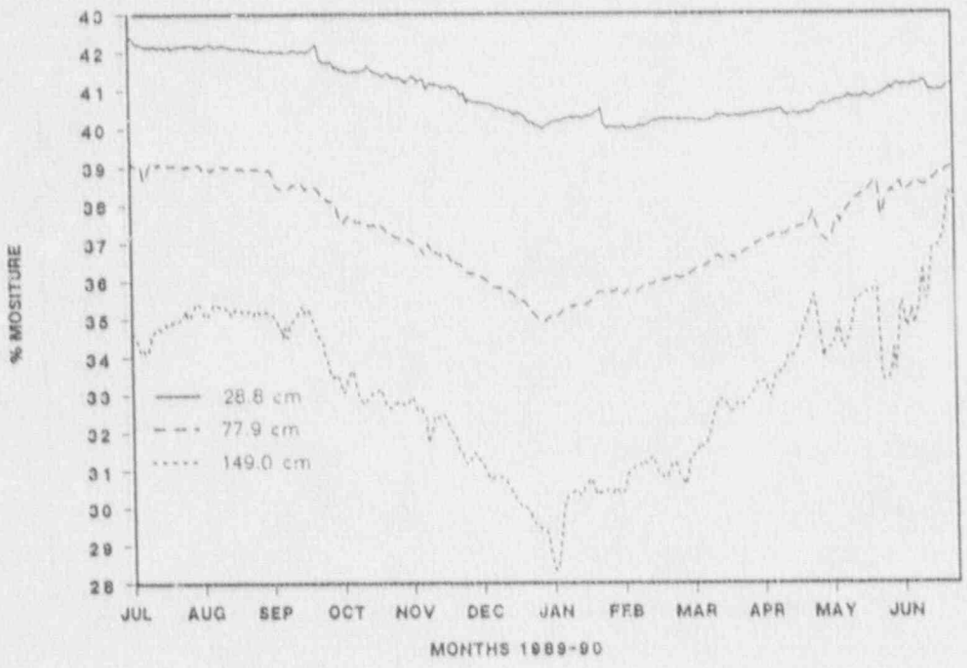

Figure 38. ORNL lysimeter 4 soil moisture.

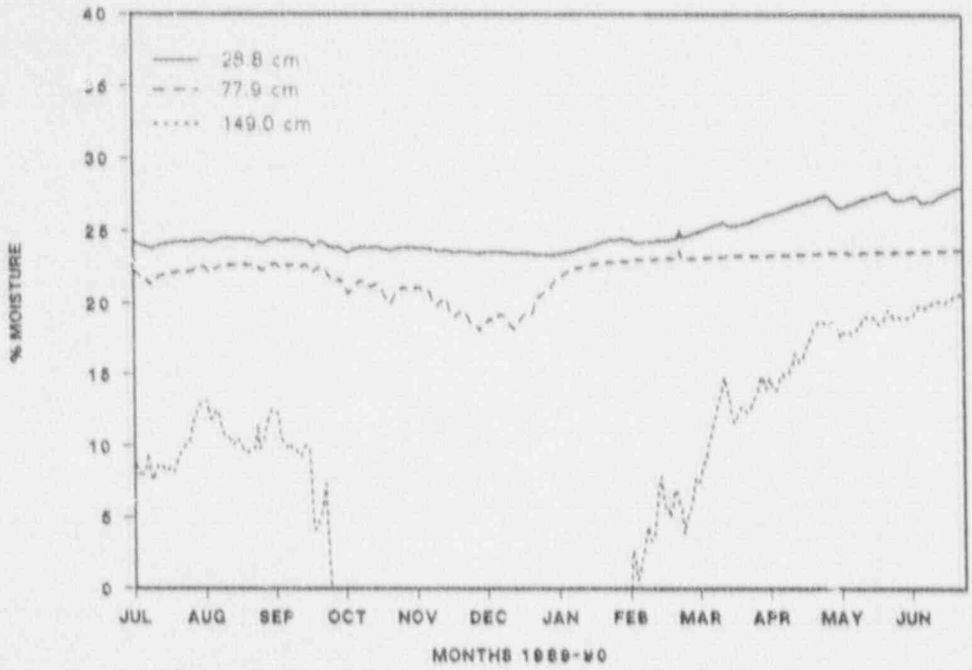

Figure 39. ORNL lysimeter 5 soil moisture.

Soil moisture was discussed in a previous report.[19] Soil moisture in the soil column of the lysimeters at each site is quantified gravimetrically once each year (see Tables 8 and 9). Some idea of the accuracy of the soil moisture probes can be calculated by comparing the once–a–year gravimetric soil moisture data of each soil lysimeter to yearly averaged probe data (Table 10). Percent differences between the gravimetric data and probe data for ANL–E lysimeters range between 28.0 and 37.6%. These values are still within a reasonable range given the use of the information. While data from the ORNL probes continue to overestimate the actual percent soil moisture, these data have remained consistent year after year.

Soil moisture (as gravimetrically determined) at each sampling depth has remained uniformly consistent between intrasite lysimeters during the past several years (Figures 40 and 41). The uniformity of soil moisture in the ANL–E lysimeters (Figure 40) continues to be of interest given the long–term, nonuniform decrease in water infiltration in to ANL 1, 2, and 4. Action to improve drainage of these lysimeters has been taken; however, it has now become obvious that the initial rate of drainage cannot be restored. The present conditions are now thought to be indicative of what would be found if a disposal trench were constructed in this soil. It was decided in fiscal year 1989 that no further efforts would be made to improve drainage of these lysimeters. Instead, water is no longer allowed to pond on the soil surface. Water in excess of 2–3 cm in depth is now removed from the lysimeter surfaces. Total quantities of water removed from the three lysimeters during the year were

- ANL 1, 431 L
- ANL 2, 428 L
- ANL 4, 210 L.

It is apparent from data presented in Figures 40 and 41 that after initial wetting, the water storage within the lysimeter soil columns at each of the sites appears to have remained fairly constant (Tables 8 and 9 and References 17, 18, 19, 20, and 21). At the time of the last sampling, the average soil moisture of ANL–E soils was 56.1% of

Table 8. Moisture profile of ANL–E lysimeters 1 through 4 based on gravimetric measurement of water content[a]

Lysimeter	Depth (cm)	Moisture (dry weight) (%)
1	0–41	17.4
1	41–62	20.8
1	62–82	21.2
1	82–107	21.4
1	107–133	21.9
1	133–153	22.6
1	153–182	23.0
1	182–202	23.3
2	0–41	18.5
2	41–62	22.3
2	62–82	21.1
2	82–107	22.6
2	107–133	22.6
2	133–153	23.3
2	153–182	23.8
2	182–202	23.8
3	0–41	22.2
3	41–62	22.7
3	62–82	24.7
3	82–107	24.9
3	107–133	24.6
3	133–153	24.6
3	153–182	24.9
3	182–202	24.3
4	0–41	22.3
4	41–62	22.6
4	62–82	22.7
4	82–107	23.7
4	107–133	24.2
4	133–153	24.0
4	153–182	23.8
4	182–202	23.9

a. Samples were collected on July 28, 1989.

Table 9. Moisture profile of ORNL lysimeters 1 through 4 based on gravimetric measurement of water content[a]

Lysimeter	Depth (cm)	Moisture (dry weight) (%)
1	0–25	15.8
1	25–50	16.6
1	50–75	17.8
1	75–100	18.0
1	100–125	17.8
1	125–150	18.6
2	0–25	16.0
2	25–50	16.7
2	50–75	17.3
2	75–100	17.8
2	100–125	18.0
2	125–150	18.2
3	0–25	15.2
3	25–50	16.4
3	50–75	17.6
3	75–100	17.9
3	100–125	18.5
3	125–150	18.8
4	0–25	15.7
4	25–50	17.1
4	50–75	17.5
4	75–100	18.1
4	100–125	18.2
4	125–150	18.9

a. Samples were collected on July 10, 1989.

the soil moisture holding capacity (MHC). At ORNL, this value was 39.2%. These values have remained fairly constant from year to year.

By using the cumulative rainfall data from each site since the time the lysimeters were placed in operation (Figure 20), it is possible to calculate the approximate volume of water that

Table 10. Comparison of the average percent moisture values in lysimeter soil column as determined from probe and gravimetric data

Lysimeter Number	Average Percent Moisture for Soil Column Probes[a]	Average Percent Moisture for Soil Column[b]	Percent Difference
ANL 1	15.4 ± 2.6	21.4 ± 1.9	28.0
ANL 2	14.5 ± 1.7	22.2 ± 1.8	34.7
ANL 3	16.2 ± 8.5[c]	24.1 ± 1.0	32.8
ANL 4	14.6 ± 1.7	23.4 ± 0.7	37.6
ORNL 1	39.0 ± 0.6	17.4 ± 1.0	124.1
ORNL 2	39.6 ± 0.2	17.4 ± 0.8	127.6
ORNL 3	34.0 ± 1.7	17.4 ± 1.4	95.4
ORNL 4	37.2 ± 3.9	17.6 ± 1.1	111.4

a. July 1989 through June 1990.

b. Determined gravimetrically for July 1990.

c. Average from two probes.

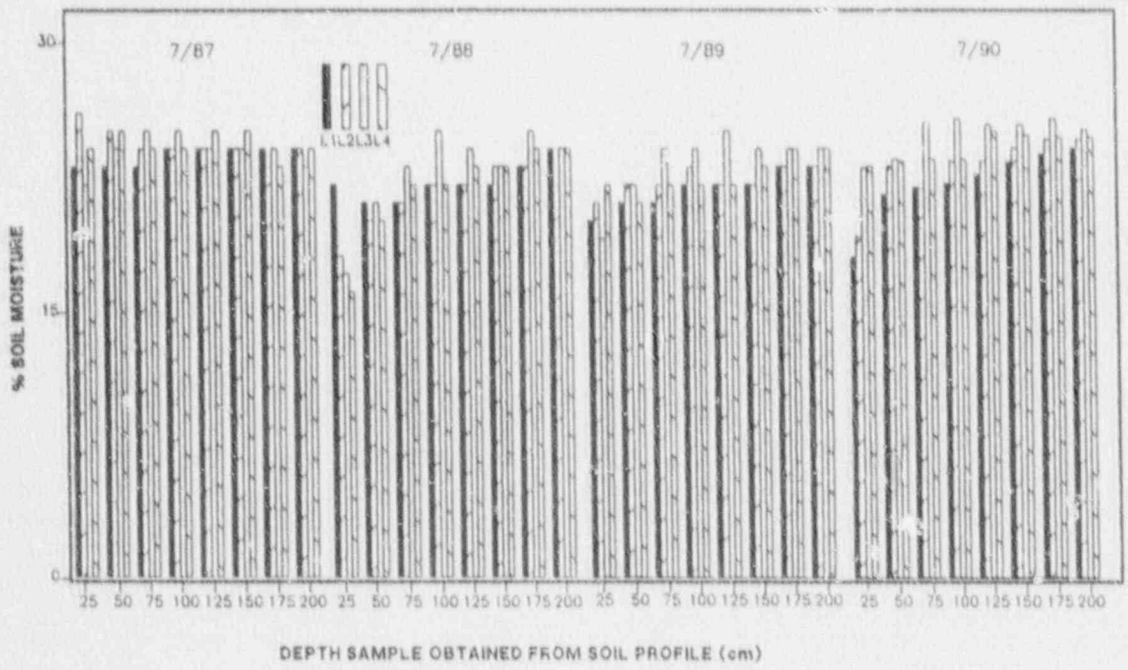

Figure 40. Moisture profile of ANL–E lysimeters 1 through 4 by year based on gravimetric measurement of water content.

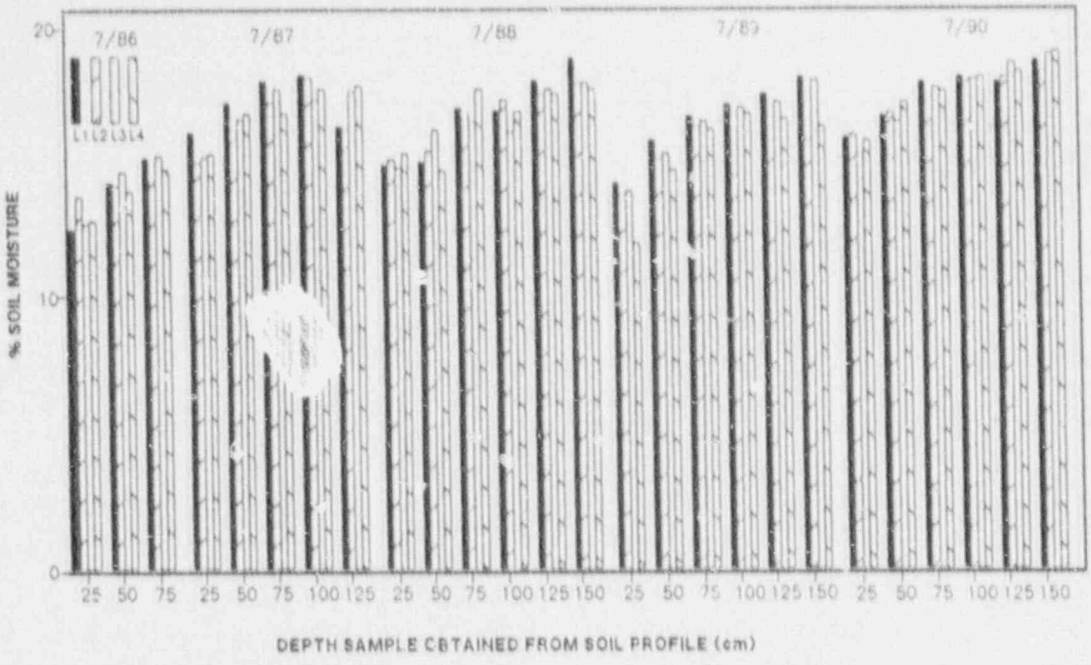

Figure 41. Moisture profile of ORNL lysimeters 1 through 4 by year based on gravimetric measurement of water content.

has been received by the exposed lysimeter surfaces (6489.5 cm^2). Each ANL–E lysimeter received 2769 L; each ORNL lysimeter received 4138 L. The volume of the precipitation that has passed through the lysimeters can be seen in Figures 42 and 43. It has become apparent with time that the throughput of precipitation is dependent on site conditions and lysimeter fill material. At ANL–E, an average of 1214.0 $\pm$ 437.2 L, with a range of 30.1 to 64.9% of total precipitation, has passed through the soil lysimeters; while for the control, this value was 2761 L, or 99.7% of available precipitation. For ORNL, the values were 3511.5 $\pm$ 21.0 L (84.8%) for the soil–filled lysimeters and 4084 L (98.7%) for the control. These data are comparable to the previous year's data (Reference 21). The ORNL lysimeter soils are more permeable than the ANL–E soils (an observation made by comparing the control lysimeter at each site with that site's soil lysimeters). Also, the ORNL lysimeters continue to perform as a unit as compared to the individual drainage activity of the ANL–E lysimeters.

The total volumes of precipitation that have moved through the lysimeters represent an average 1.71 pore volumes for the ANL–E soil lysimeters and 4.94 pore volumes for soil lysimeters at ORNL, while the controls at ANL–E and ORNL were 4.70 and 7.29 pore volumes, respectively.

Radionuclide Analysis. Water samples are normally collected on a quarterly basis from leachate collectors and moisture cups of each of the lysimeters during the 12–month period. At each sampling, only water from the leachate collectors (1 L of collected quantity) and those cups (0.1 L or as noted of the collected quantity) closest to the waste forms (cup 3) are generally analyzed for gamma–producing nuclides and the beta–producing nuclide Sr–90. The analysis protocol, however, triggers the analysis of water from additional cups in a sequential manner if nuclides are found in a cup 3 sample. For example, when nuclides are found in a cup 3 of a lysimeter, water from cup 1 (directly below cup 3), then cup 4, followed by cup 2 should be analyzed (see Figure 11 for cup placement). Because of funding levels, however, it has not been possible to follow this protocol. Rather, only water samples from cups 3 are routinely analyzed at the sites.

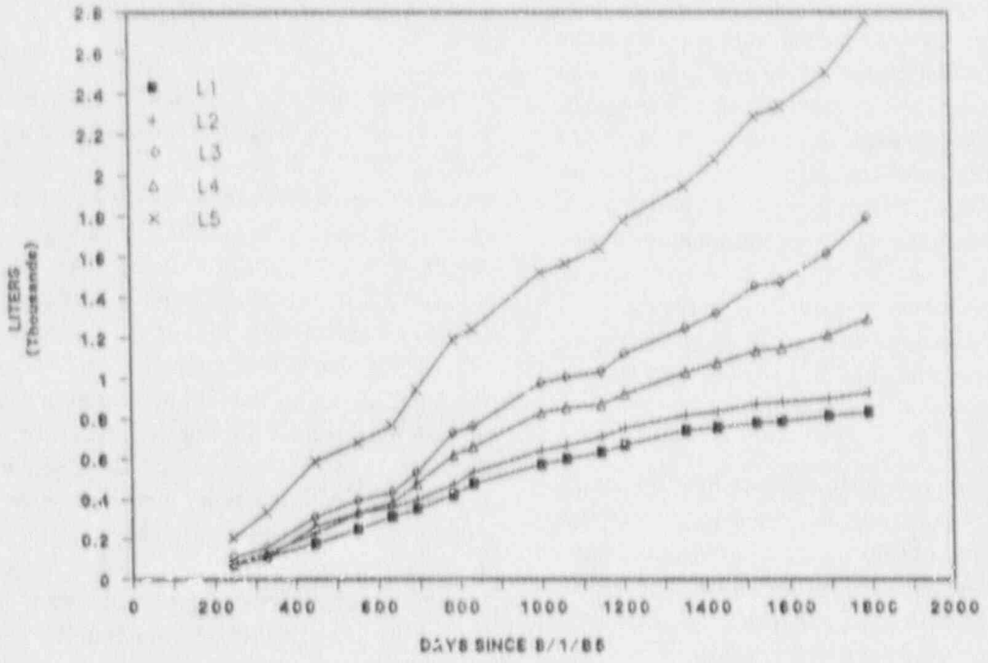

Figure 42. ANL–E cumulative volume of leachate from lysimeters.

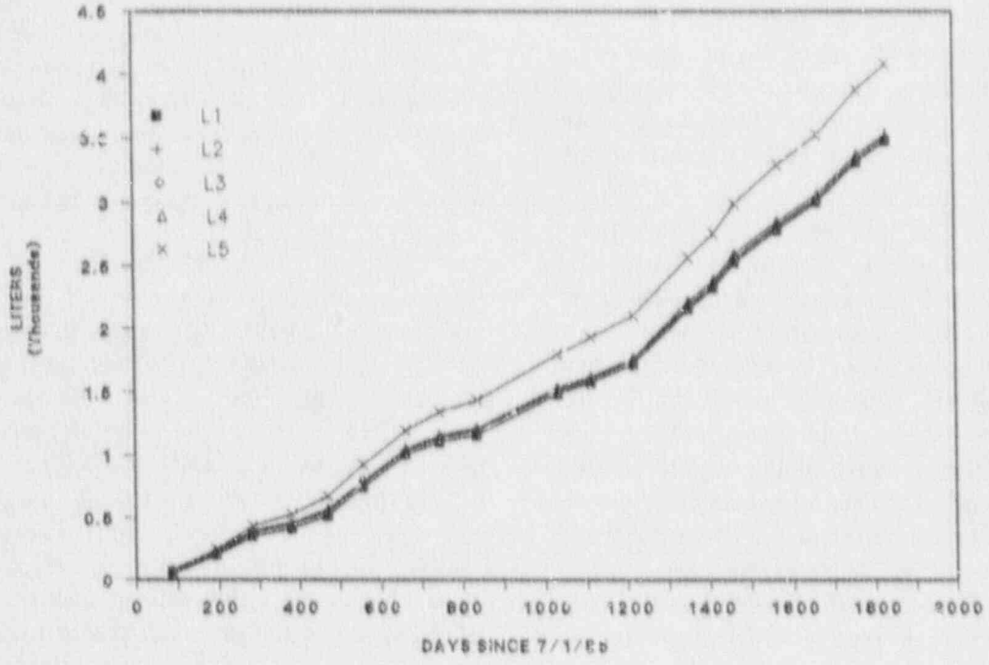

Figure 43. ORNL cumulative volume of leachate from lysimeters.

Tabulated results of beta and gamma analysis for the samples taken during the period are found in Tables 11, 12, and 13. Four samples were taken at each site during the 12 -onth period. The cumulative amounts of nuc.i.s as determined in water samples obtained from lys.meter cup 3 and leachate collectors for all sampling periods are displayed graphically in Figures 44 through 52. It should be noted that water samples have not been withdrawn from ORNL cup 3–3 since the summer of 1989 due to a malfunction of that cup. The lack of data from this cup could give the erroneous impression that nuclides are no longer available from this source (Figure 45).

As has been reported in the past[18,19,20,21] not all nuclides are appearing consistently in either the water obtained from the cups or the leachate collectors. The nuclide that appears with the most regularity at both sites continues to be Sr–90. Consistent, significant occurrences of this nuclide continue in all the cup 3 samples at both ANL–E and ORNL (Figures 4? and 45). There continues to be standout amounts of Sr–90 retrieved from cup 3 samples at both sites. Those include a cumulative total of 452,840 pCi from 3–3 at ANL–E (an increase of 168% over last year) (Figure 44) and 37,286 pCi from 1–3 at ORNL (a 216% increase over last year) (Figure 45). The releases into ANL 3–3 and ORNL 1–3 continue to be steady and almost linear.

During the past 12 months, leachate water from the control lysimeters at each site have continued to contain amounts of Sr–90 orders of magnitude larger than the soil lysimeters (Figures 46 and 47). This is comparable to the previous years findings (References 18, 19, 20, and 21) and as shown above continues to be in sharp contrast to the cup 3 data, which continue to demonstrate that substantial amounts of Sr–90 are still being released from the waste forms in the soil lysimeters. The percent of total Sr–90 being measured in the leachate water and cup 3 samples is somewhat inconsistent between the two sites (Table 14). This could indicate differences in waste form performance at the two sites. However, there is still a comparable percent of total Sr–90 in the leachate water of the control lysimeters for the two sites (Table 14).

Gamma–producing nuclides continue to occur with regularity at ANL–E and are again present at ORNL. ANL 2–3, below a cement waste form containing large amounts of Cs–137, continues to receive Cs–137 (Table 11) with a significant increase in the quantities of this nuclide appearing this year after initially peaking in the February 1987 sample (Figure 48). Since June of 1987, Cs–137 has been appearing in ANL 5–3. The quantity of this nuclide increased in each of the sampling periods during the last year,[21] with an abrupt increase during the last sampling period (Figure 40). This year, there were continued substantial releases (Figure 48). There continues to be no sustained occurrence of Cs–137 in any of the ANL leachate water. Detectable amounts of Cs–137 have been consistently found in water from ORNL–5 and sporadically in the other ORNL waters (Tables 12 and 13). Measurable amounts of Cs–137 began to occur in ORNL 5–3 during the May 1988 sample and have continued in subsequent samplings for a total of 480 pCi. Breakthrough of Cs–137 into the ORNL 5 leachate collector occurred in November 1988, some 7 months after its occurrence in the moisture cup ORNL 5–3 (Figure 49). Thus far, a total of 153,198 pCi have passed through the lysimeter (Figure 50). There have been no occurrences of Sb–125 this year (Figures 51 and 52).

By using a matrix, several comparisons can be made based on the data. Such a matrix is seen in Table 14, which includes intra– and intersite comparisons. Overall, of the nuclides contained in the waste forms,[17] there has been a greater recovery of Sr–90 in terms of quantity and percent of inventory than other nuclides. Cs–137 would be followed by Sb–125 (not listed in Table 14). Compared to Sr–90, the recovery of Cs–137 appears insignificant but remember that it has only been in the past two years that this nuclide has been consistently occurring in number 3 cups at ORNL (Figure 49) and has begun to occur consistently in the leachate collector of the ORNL–5 lysimeter (Figure 50).

Table 11. Results of beta and gamma analysis of ANL–E soil moisture and leachate samples, year 5 (1989 – 1990)

	Concentration (pCi/L)[a]											
	Co–60				Cs–137				Sr–90			
Sample Identification	89 Oct	90 Dec	90 Mar	90 Jul	89 Oct	90 Dec	90 Mar	90 Jul	89 Oct	90 Dec	90 Mar	90 Jul
Lys 1[b]	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
Lys 2	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1	<1
Lys 3	<1	<1	<1	<1	<1	<1	<1	<1	7.9 ± 0.2	2.3 ± 0.1	11.4 ± 0.2	31 ± 1
Lys 4	<1	<1	<1	<1	<1	<1	<1	<1	<1	18.4 ± 0.3	<1	<1
Lys 5	<1	<1	<1	<1	<1	<1	2 ± 1	4.7 ± 1.6	384 ± 1	345 ± 1	452 ± 1	432 ± 1
Lys 1–3[c]	<5	<5	<5	<5	<5	<5	<5	<5	1.8E4 ± 129	4942 ± 50	2762 ± 36	176 ± 12
Lys 2–3	<5	<5	<5	<5	965 ± 2	1563 ± 55	2556 ± 67	1295 ± 53	1946 ± 9	1564 ± 55	2480 ± 12	2773 ± 11
Lys 3–3	<5	<5	<5	<5	<5	<5	<5	<5	7.9E5 ± 1452	6.7E5 ± 1343	1.0E6 ± 1503	1.01E6 ± 1531
Lys 4–3	<5	<5	<5	<5	<5	<5	<5	<5	2353 ± 9	4519 ± 13	3321 ± 2	2280 ± 10
Lys 5–3	<5	<5	<5	<5	1.9E4 ± 134	1.5E4 ± 50	2.1E4 ± 141	2.7E4 ± 234	9377 ± 76	1.3E4 ± 80	1.0E4 ± 70	1.03E4 ± 72

a. Concentration ± 2 sigma

b. 1–L subsample from leachate collector

c. Total moisture cup sample size is ≈ 0.1–L

Table 12. Results of beta and gamma analysis of ORNL soil moisture and leachate samples, year 5 (1989–1990)

Sample Identification	Concentration[a] (Pci/L)							
	CO–60				Cs–137			
	Oct 89	Jan 90	Apr 90	Jul 90	Oct 89	Jan 90	Apr 90	Jul 90
Lys 1[b]	–1.6 ± 4.0	0.5 ± 5.7	<108	3.2 ± 4.6	0.5 ± 3.5	1.3 ± 4.0	40.5 ± 37.8	0.8 ± 5.9
Lys 2	6.2 ± 3.8	2.7 ± 5.7	<108	0.8 ± 3.5	1.6 ± 4.6	5.1 ± 4.9	27.0 ± 59.5	0.5 ± 3.2
Lys 3	1.9 ± 7.0	0.8 ± 4.9	<81	0.7 ± 3.0	–3.2 ± 7.8	0.3 ± 3.8	2.7 ± 64.9	1.1 ± 4.9
Lys 4	–0.5 ± 3.2	–0.8 ± 3.5	<81	1.2 ± 7.0	0.3 ± 3.2	0.8 ± 3.0	<54	3.5 ± 6.2
Lys 5	2.4 ± 4.3	1.1 ± 5.1	<54	2.2 ± 4.3	64.9 ± 5A	37.8 ± 5.4	<81	22 ± 4.6
Lys 1–3[c]	13.5 ± 29.7	–13.5 ± 43.2	<54	2.7 ± 48.7	2.7 ± 29.7	–8.1 ± 35.1	<54	19 ± 35
Lys 2–3	5.4 ± 46.0	16.2 ± 59.2	<81	46 ± 38	2.7 ± 43.2	24.3 ± 54	<54	24 ± 57
Lys 3–3	46.0 ± 56.8	—[d]	—	—	27 ± 64.9	—	—	—
Lys 4–3	–8.1 ± 37.8	24.3 ± 35.1	<54	22 ± 103	2.7 ± 29.7	2.7 ± 043.7	<54	5 ± 76
Lys 5–3	8.6 ± 23.8	13.5 ± 32.4	<54	5.4 ± 62	595 ± 27	432 ± 27	514 ± 27	946 ± 81

a. Concentration ± 2 sigma.

b. 1–L subsample from leachate collection.

c. Total moisture cup sample size is = 0.1–L.

d. Sample not available.

Table 13. Results of beta and gamma analysis of ORNL soil moisture and leachate samples, year 5 (1989–1990)

Sample Identification	Concentration[a] (Pci/L)							
	Sb–125				Sr–90			
	89 Oct	90 Jan	90 Apr	90 Jul	89 Oct	90 Jan	90 Apr	90 Jul
Lys 1[b]	<8.1	<54	<108	<8.1	2.7 ± 29.7	3.5 ± 3.2	48.7 ± 8.1	18.1 ± 5.1
Lys 2	<5.4	<54	<108	<8.1	3.8 ± 3.2	1.3 ± 2.54	0.3 ± 3.2	4.6 ± 3.2
Lys 3	<10.8	<108	<108	<8.1	0.4 ± 2.5	1.9 ± 3.0	0.8 ± 3.2	1.9 ± 2.7
Lys 4	<5.4	<54	<108	<8.1	2.7 ± 3.2	1.1 ± 3.0	2.7 ± 3.5	4.3 ± 3.2
Lys 5	<10.8	<54	<108	<8.1	194.6 ± 13.3	405 ± 27	297 ± 27	3.2 ± 27
Lys 1–3[c]	<54	<54	<81	<108	4.0E4 ± 2703	3.5E4 ± 2703	4.0E4 ± 2703	8.4E4 ± 2703
Lys 2–3	<54	<81	<81	<108	4325 ± 270	4325 ± 270	7839 ± 270	8920 ± 2703
Lys 3–3	<108	—	—	—	2.2E4 ± 270	—	—	—
Lys 4–3	<54	<54	<81	<108	62.2 ± 13.5	70.3 ± 13.5	183.8 ± 27	297 ± 27
Lys 5–3	<54	<54	<81	<108	175.7 ± 21.6	221.6 ± 27.3	460 ± 54	568 ± 27

a. Concentration ± 2 sigma.

b. 1–L subsample from leachate collection.

c. Total moisture cup sample = 0.1–L sample size.

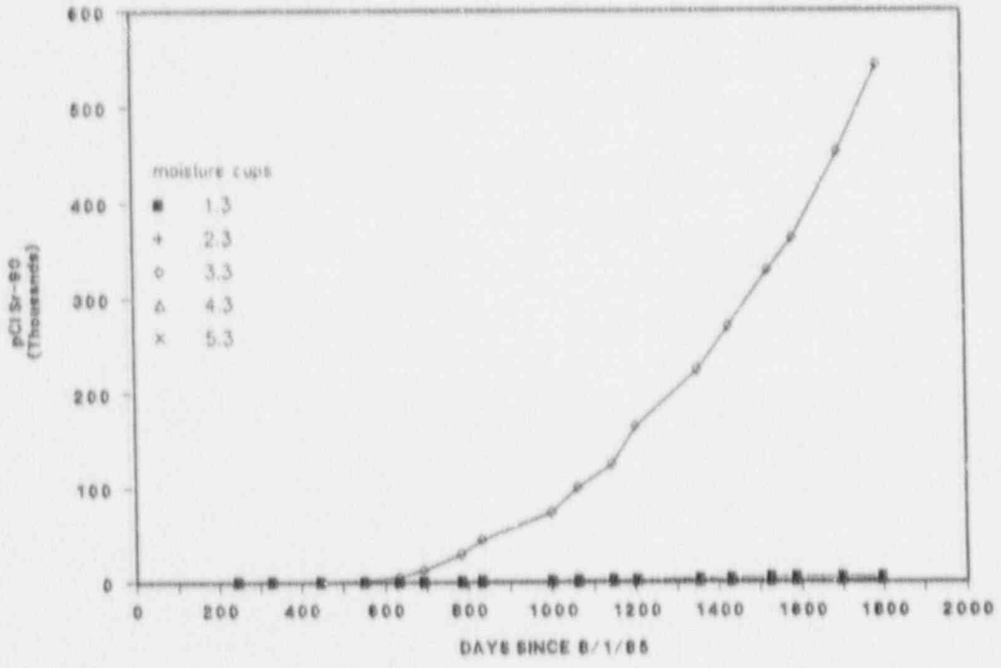

Figure 44. ANL–E cumulative SR–90 collected in moisture cup number 3.

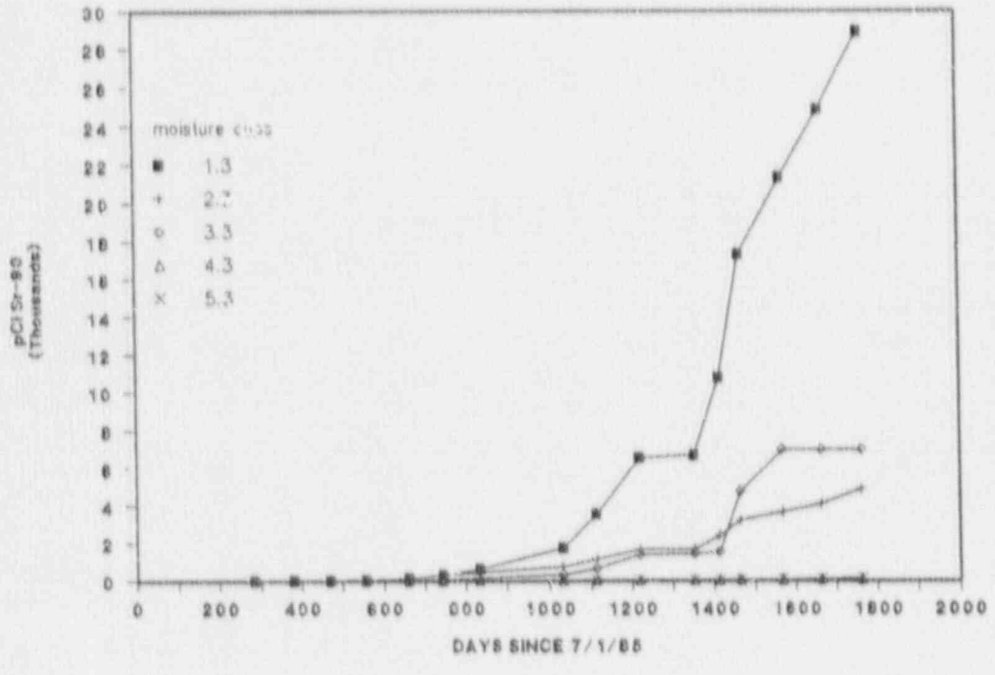

Figure 45. ORNL cumulative Sr–90 collected in moisture cup number 3.

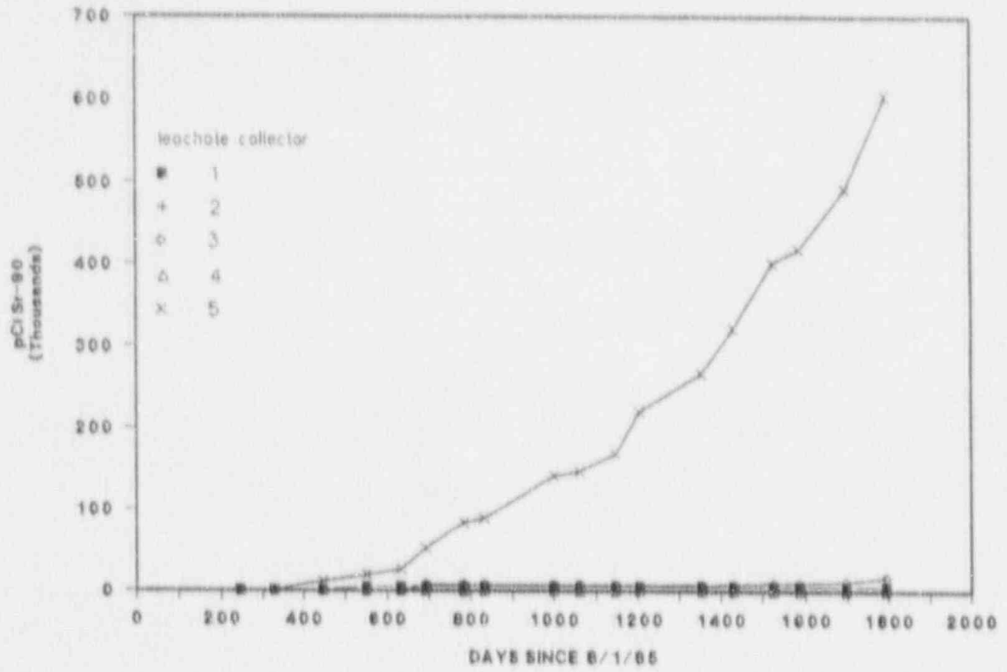

Figure 46. ANL–E cumulative Sr–90 collected in lysimeter leachate collectors.

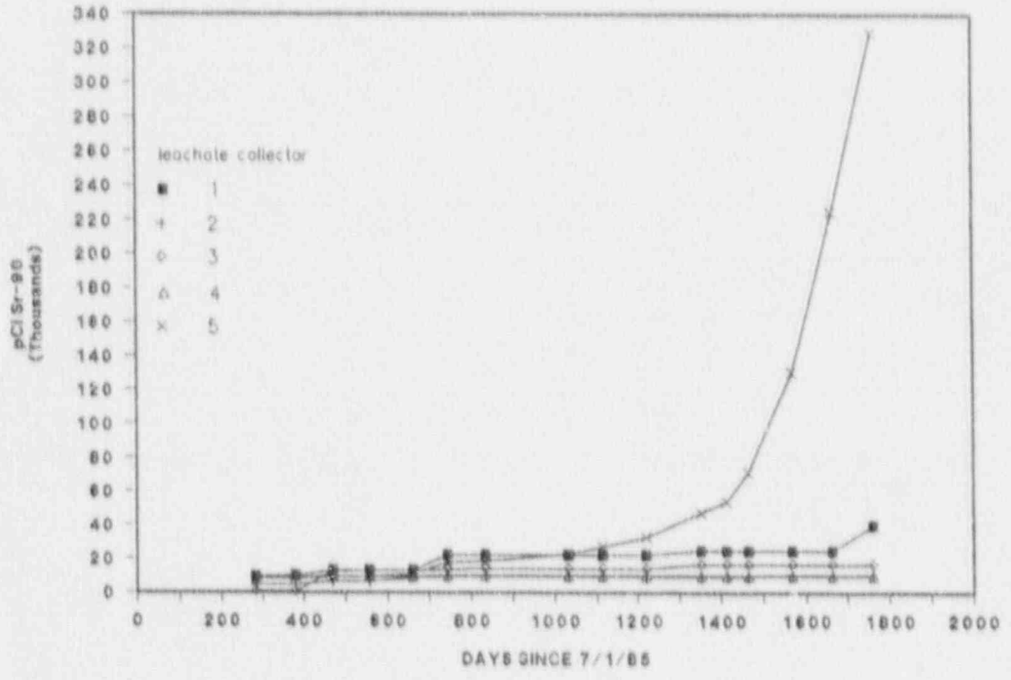

Figure 47. ORNL cumulative Sr–90 collected in lysimeter leachate collectors.

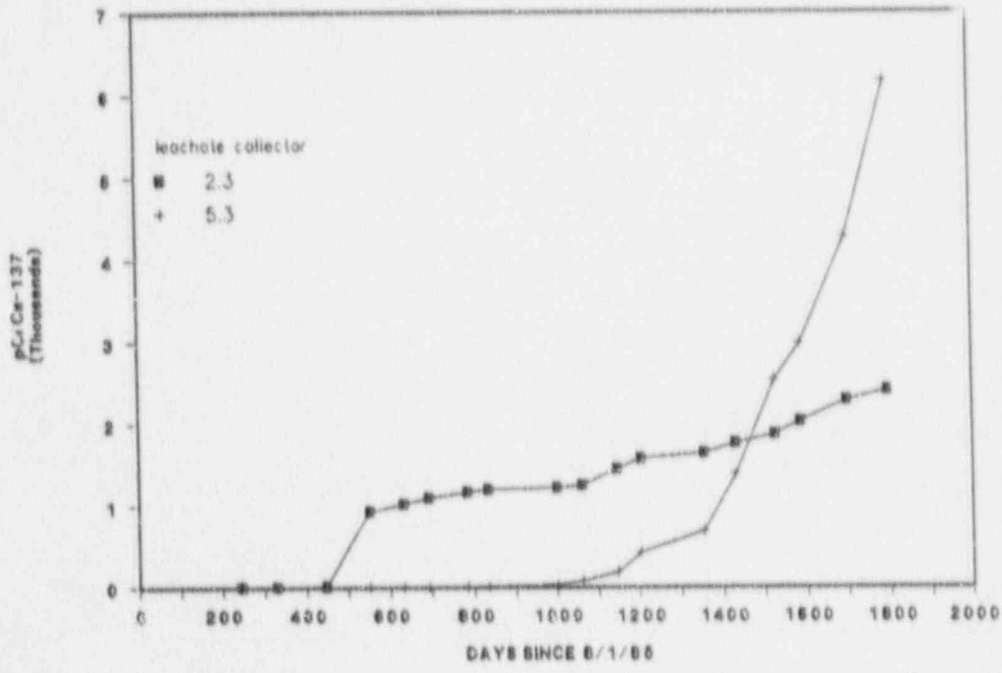

Figure 48. ANL–E cumulative Cs–137 collected in moisture cup number 3.

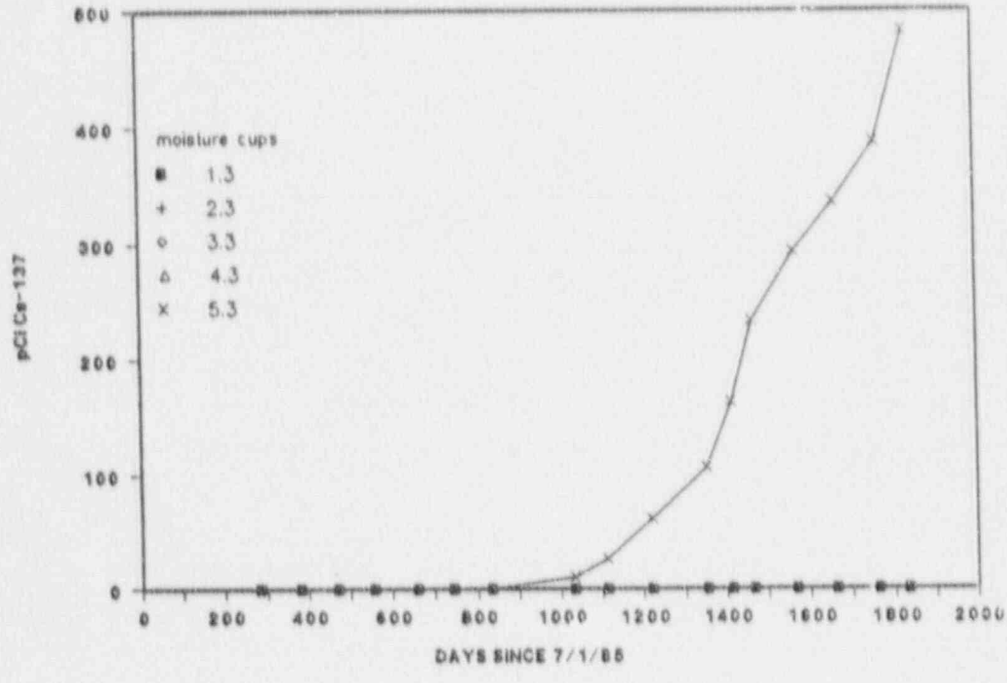

Figure 49. ORNL cumulative Cs–137 collected in moisture cup number 3.

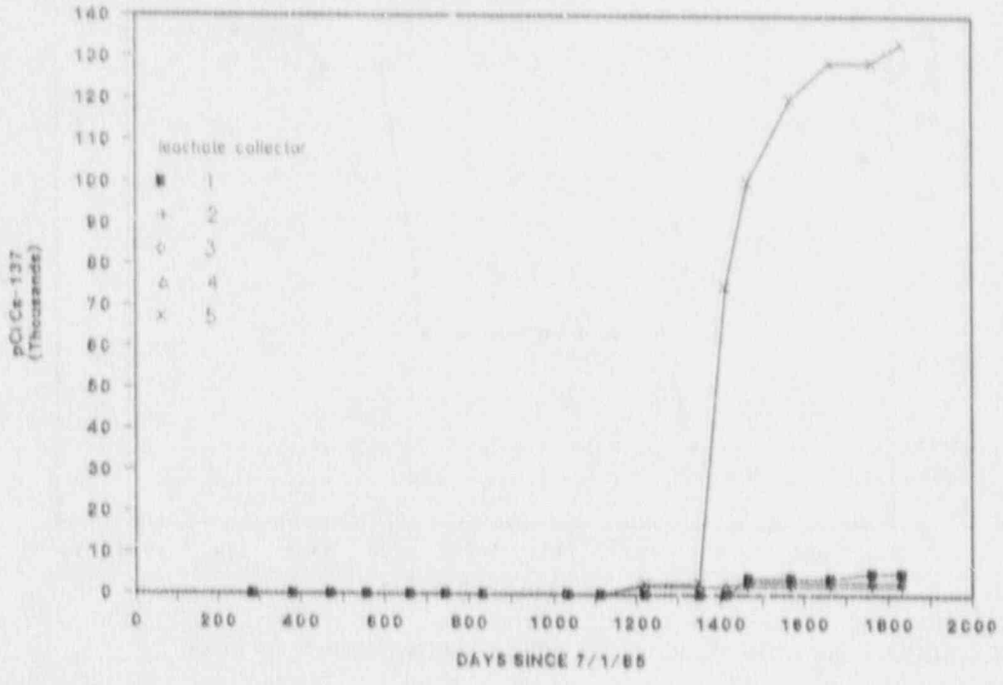

Figure 50. ORNL cumulative Cs–137 collected in lysimeter leachate collectors.

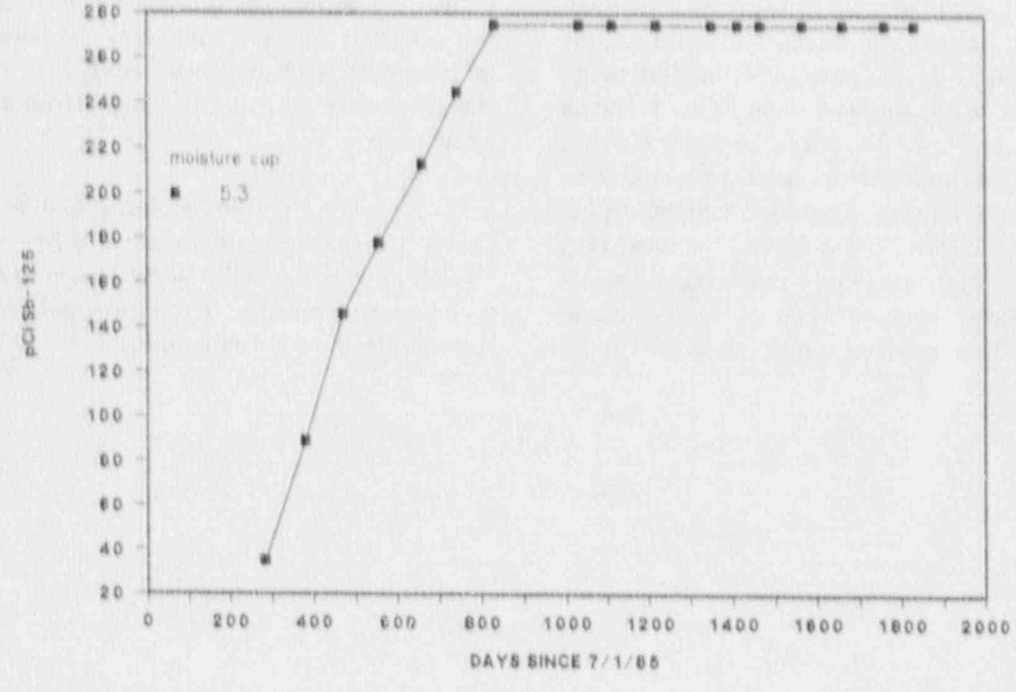

Figure 51. ORNL cumulative Sb–125 collected in moisture cup number 3.

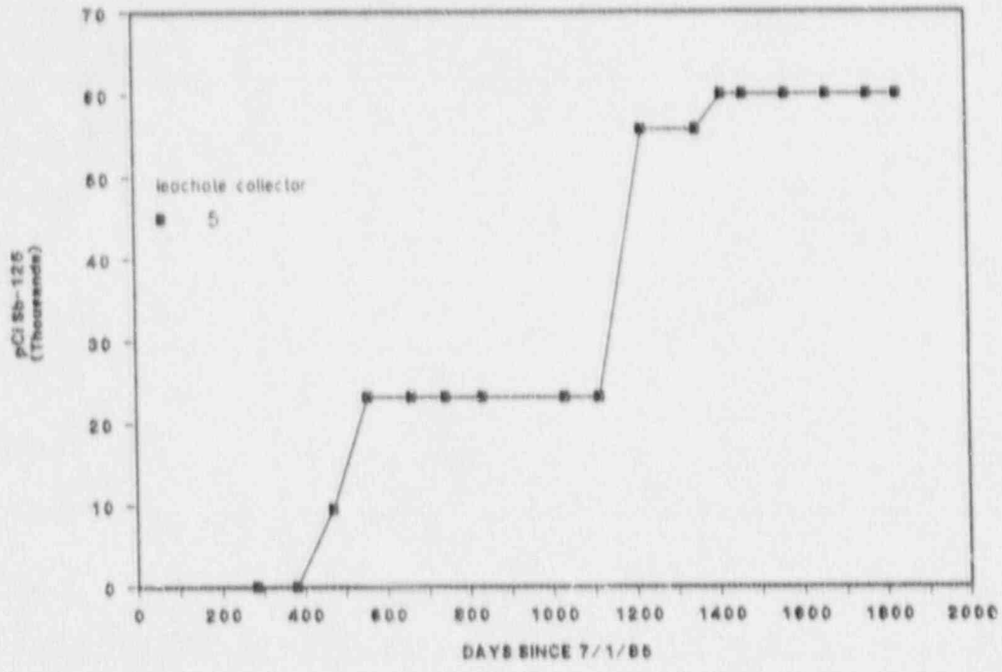

Figure 52. ORNL cumulative Sb–125 collected in lysimeter leachate collector.

At ANL–E, recovery of Sr–90 in cup 3 and the leachate collectors continues to indicate a uniformity of waste form performance. More Sr–90 is found in ANL–3 (VES waste form) than in the other ANL cups but the effect appears to be moderated by the distance traveled in soil from the waste form to the leachate collector. Movement of the nuclide into the leachate of ANL–5 is much greater than that of the other lysimeters and thus provides continued evidence of the moderating effect of soil. Greater quantities of Sr–90 appear to be moving through the ORNL lysimeter than the ANL–E lysimeter. Once again, there appears to be no correlation between the type of waste form and the amount of nuclide recovered in the leachate collector. About 0.1% of the Sr–90 contained in ORNL–5 has now been recovered in leachate from that lysimeter. Recovery of Sr–90 in the ORNL cups has been greater in those lysimeters containing the cement waste forms but any conclusions drawn based on those data must be tempered with data from ANL 3–5, which shows greater recovery of Sr–90 from a VES waste form.

On an intersite comparison it can be seen (Table 14) that larger quantities of Sr–90 and Cs–137 are moving in the ORNL lysimeters. Soil type and precipitation (environmental factors) appear to be the controlling factors.

Table 14. Comparison of total Sr–90 and Cs–137 inventory per lysimeter to total amounts in lysimeter water

Lysimeter Number	Solidifying Agent	Percent Total Inventory Sr–90 × 10^6				Percent Total Inventory Cs–137 × 10^6			
		Moisture Cups		Leachate Water		Moisture Cups		Leachate Water	
		ANL	ORNL	ANL	ORNL	ANL	ORNL	ANL	ORNL
1	Cement	29	205	27	235	—	—	—	2.0
2	Cement	70	174	49	313	0.2	—	—	0.1
3	VES	1981	5	60	64	—	—	—	0.7
4	VES	47	2	6	219	—	—	—	0.1
5	Cement	32	6	3322	10000	10.0	0.3	—	9.0

CONCLUSIONS

The INEL study of degradation of EPICOR–II organic ion exchange resins correlates with findings of other researchers (References 13, 14, 24, 25, 26, 27, and 28) and degradation has been identified in the EPICOR–II resins at a lower than predicted gamma radiation dose.[2,3,4,11] The internal radiation dose received by the organic ion exchange resins in EPICOR–II prefilters PF–8 and PF–20 was sufficient to initiate degradation at the time of analysis of resins from the first sampling. Degradation was continuing from the second sampling at the time of resin analysis and increased noticeably by the third sampling. The equilibrium of the polymer structure has been shifted toward polymer breakdown, as can be seen by the further change in characteristics between the first, second, and third analyses. These results indicate that the threshold dose for the onset of degradation is between 5.1×10^7 and 6.6×10^7 rads because of internal radiation.

One important indicator of the capability of ion exchange media to retain radionuclides is the total exchange capacity. The four samples examined at the INEL exhibited different reactions to radiation. PF–8#1 and PF–8#3, the strong acid cation resins with the highest radiation doses, showed further reduced total exchange capacity. PF–20, the strong acid cation with the lowest radiation dose, showed a decrease from no change. PF–8#2, the phenolic cation with a similar low dose, showed further decreased capacity. Those results indicate that the PF–8#1 and PF–8#3 resins and the PF–8#2 phenolic cation sample had reached a dose high enough to cause the exchange capacity to rapidly decrease. The PF–20 resin had moderately decreased capacity at the third sampling indicating further progress of degradation. Those changes agree with other findings.[14]

These analyses have assisted in determining the extent that organic ion exchangers are degraded by internal radiation under conditions representative of actual use and storage. Degradation of the resins has been related directly to total integrated radiation dose. This has aided in identifying the effects of degradation on release of radionuclides from the ion exchange media. The resin bed in the higher radiation zone is being converted to an agglomerated mass, a substance with unique new nuclide retention capabilities. It has been shown that the contained radionuclides remain within that agglomerated ion exchange bed, which prevents release by restricting water through flow. The acceptability of EPICOR–II prefilters for disposal at a commercial site is thus confirmed.

The findings of this study can be related to commercial disposal of spent ion exchange media used in power reactors. Determining the onset of degradation and, later, significant loss of exchange capacity with resultant loss of radionuclides provides a database useful in planning for and regulating disposal of ion exchange resins.

The seven–year old waste forms fabricated with EPICOR–II ion exchange resins exhibited a slight increase in ultimate compressive strength with age when compared to the strength of 1 and 24–month old waste forms. It appears that strength has not been detrimented by accumulated radiation dose but has increased with age, while one Portland cement specimen failed somewhat above the expected strength. It would be anticipated that the cement specimens would retain strength to a higher radiation dose than VES because low–level radiation does not adversely affect cement strength. However, the high strength exhibited by the one cement specimen is thought to be anomalous and attributed to data scatter. Polyethylene specimen containers are becoming embrittled, which is a precursor to degradation of the VES and the EPICOR–II ion exchange resin contained in the waste form specimen. The compression tests planned over the next several years should confirm the observed trends.

Lysimeter operation during the fifth year at ANL–E and ORNL has been successful. Analyses of data collected during the past 60 months are continuing to show a pattern in nuclide availability and movement such that the

cumulative data are beginning to provide significant insight on waste form performance.

Strontium–90 is still the most prevalent nuclide in collected liquid samples. It appears that waste form performance is similar with respect to release of Sr–90 (except for a very high release from ANL–3). It is also apparent that Sr–90 is able to move more freely through the Savannah River Laboratory soil at ORNL. During the past 36 months, Sr–90 continued to be found in leachate water in the control lysimeters at both sites. It continued to appear that the limited step in receiving Sr–90 in the leachate is not release of the nuclide from the waste forms (since Sr–90 is found in cup 3 samples), but rather it is the soil characteristics (including soil and quantity of soil water) that limit movement.

Data on waste form performance presented in this report continue to suggest that VES is comparable to cement in its ability to retain Sr–90.

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11. ABSTRACT (200 words or less)

The EPICOR-II Resin/Liner Investigation: Low-Level Waste Database Development Program, funded by the U.S. Nuclear Regulatory Commission, is (a) studying the degradation effects in EPICOR-II organic ion exchange resins caused by radiation, (b) examining the adequacy of test procedures recommended in the Branch Technical Position on Waste Forms to meet the requirements of 10 CFR 61 using solidified EPICOR-II resins, (c) obtaining performance information on solidified EPICOR-II ion exchange resins in a disposal environment, and (d) determining the condition of EPICOR-II liners.

Results of the third sampling analysis of ion exchange resins from prefilters PF-8 and PF-20 are compared with baseline data from tests performed on unirradiated resins and with results from the first and second samplings to determine if degradation has occurred because of the high internal radiation dose. Results of compression tests on seven-year old waste forms containing EPICOR-II resins solidified with both Portland Type I-II cement and DOW vinyl ester-styrene are presented and compared to earlier compression test data. Results of the fifth year of data acquisition from the field testing are also presented and discussed.

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