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Tritium Waste Control: July-September 1978

November 10, 1978

Monsanto

MOUND FACILITY
Miamisburg, Ohio 45342

operated by
MONSANTO RESEARCH CORPORATION
a subsidiary of Monsanto Company

for the
U. S. DEPARTMENT OF ENERGY

Contract No. EY-76-C-04-0053

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Foreword

The work in the first and third sections is funded by 189C AL0219A. The remaining sections are work related to Tritium Waste Control but not directly funded by AL0219A.

Previous documents in this series are:

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MLM-2451	MLM-2519
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The combined Electrolysis Catalytic Exchange system was modified to allow better control of experimental conditions and to prevent the overflow of water into the air detritiation system. A program designed to regenerate the activity of the hydrophobic catalyst was also completed.

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Slight differences in the release rate from the drums are now beginning to appear. The three drums with the highest fractional permeation rate had the least amount of tritium when packaged (Figure 8). The fractional permeation rate of the two octane drums appears to have leveled off at about the same rate as the oil and water drums.

FIXATION OF AQUEOUS TRITIATED WASTE IN POLYMER IMPREGNATED CONCRETE. . . 10

Tests continued on samples of cement and cement-plaster mixtures which were injected with 386 Ci of tritiated water, cured, and then impregnated with catalyzed styrene monomer. After polymerization, the samples were put into uncontaminated water and the tritium concentration was monitored. No significant differences were noted except in two cases when the polyethylene bottle had been removed, which resulted in 35 to 80 times more tritium being released into the surrounding water. Full scale (cold) waste drum No. 5 was polymerized with excellent results.

GAS GENERATION BY SELF-RADIOLYSIS OF POLYMER IMPREGNATED CONCRETE. . . . 18

Pressure increase and gas composition were measured over (1) tritiated water without fixation, (2) polymer-impregnated concrete, and (3) nonpolymer concrete. Activities for all samples were 10 Ci/cm^3. Pressure buildup results are essentially the same for concrete made with tritiated distilled water and tritiated waste water. However, the pressure buildup rate is slightly higher for the polymer impregnated concrete than for the nonpolymer concrete. Mass analysis of the cover gas over tritiated water without fixation and over the polymer and nonpolymer concrete samples made with tritiated waste water show that hydrogen represents about 85% of the gas generated.

Catalytic exchange detritiation studies

M. C. Embury, T. K. Mills and M. L. Rogers

Background

In the operation of a light water reactor, all radioisotopic contaminants except tritium can be removed from the effluent stream by ordinary chemical and physical methods. Tritium removal in this case requires special considerations. Isotopic separation processes, therefore, need to be developed for removing tritium from contaminated water streams.

The objectives of this study are to:

1. Determine technical and economic factors of HT/H_2O exchange as a process for treating tritium contaminated water;

2. Determine suitability of hydrophobic exchange catalysts for use in water detritiation systems; and

3. Through pilot-scale testing, establish design criteria for detritiation systems capable of meeting the tritium control needs of DOE site operations and the U. S. power reactor industry.

A bench scale experimental system was built and operated to demonstrate the detritiation of water via catalytic exchange. In addition, an economic evaluation was made of the HT/HTO catalytic detritiation stripping process as it might apply to a 5 metric ton per day nuclear fuel reprocessing plant.

A pilot-scale Combined Electrolysis Catalytic Exchange (CECE) system was designed and built using a hydrophobic precious metal catalyst developed by AECL at Chalk River, and a solid polymer electrolyte electrolysis cell built by General Electric Company. This system was operated in an experiment which reduced the effluent tritium concentration to below Radiation Control Guidelines (RCG). The system was modified for more reliable operation and operated intermittently over a 2-month period without major equipment failure.

The CECE system was further modified to enhance process equipment reliability and prevent damage to the system during continuous operation. A Height of a Transfer Unit (H_{OG}) of 68 cm (2.2 ft) was calculated for a continuous run with a water flowrate of 4.5 cm^3/min and a hydrogen flowrate of 7,400 std cm^3/min at 32°C and 1.7 atm. Additional experiments at $\tilde{L}/\tilde{G}$ ratios of 0.5, 0.75, and 1.0 agreed with the bench scale data of Butler. However, the data indicated a reduced catalytic activity, which required regeneration of the catalyst before future experiments could be run.

Accomplishments

The Combined Electrolysis Catalytic Exchange (CECE) system was modified to correct inadequacies in the process piping. These piping changes allow the fresh feed tank for the catalytic exchange columns to maintain a constant pressure during operation, assuring a constant liquid feed rate to the columns. These piping changes will also prevent an overflow of water from the CECE system into the air detritiation system, which normally causes a system downtime of a week or more.

Additional work was done on both the air and helium detritiation systems. The manual controls on the air system were improved to provide the operator with more information during operation. A heat exchanger was installed on the oil circulation loop of the helium purifier to remove waste heat produced by the blower system.

Experimental results from previous runs were in good agreement with each other and with the bench scale data of Butler [1]. Although these results were in good internal agreement, replicate values for H_{OG} at $\tilde{L}/\tilde{G}$ ratios of 0.75 and 1.0 were lower than the initial results. While this fact could be explained by experimental error, it may have been caused by a reduction of the activity of the catalyst [2]. Previous experience with the hydrophobic exchange catalyst by the AECL at Chalk River National Laboratory has indicated that the catalyst deactivation is reversible in that as much as 80% of the original catalyst activity can be recovered with regeneration techniques [3]. Of primary concern is the loss of the hydrophobic nature of the catalyst due to the wetting of active sites on the catalyst surface. The situation can be remedied by drying the catalyst with an inert gas, such as nitrogen or argon, at an elevated temperature. Another type of catalyst poisoning is caused by trace impurities in the water stream such as carbon monoxide or mercaptans. These impurities can be burned off the catalyst with oxygen gas [4].

Preheated nitrogen gas was passed through the catalyst beds in order to dry them. The gas flow was continuous at a rate of approximately 11 std liter/min for a one hundred hour period through the work week. This procedure was followed for four weeks and restarted for another week after a 6-week delay due to construction in the gloveboxes. The desired nitrogen temperature for regeneration was 130°C, however, because of the double containment built into the system it was necessary to route the gas through 120 ft (36.6 m) of stainless tubing before reaching the first column. As a result it is possible that the gas had cooled to ambient tempeature (27°C) before entering the column. A gas preheater between the two columns heated the gas entering the second column to the 130°C temperature. Since the columns are sealed, no direct analysis could be made on the water content of the catalyst spheres. However, the exit gas stream was passed through an ethylene glycol bubbler which indicated a very low moisture content in the nitrogen stream leaving the columns.

The same experimental procedure was followed for a 1-week period using oxygen gas to remove other catalyst poisons such as CO or mercaptans. Nitrogen was passed through the columns for 3 days to purge the oxygen before starting the electrolysis system.

Future plans

The CECE will be operated in a series of experiments to determine H_{OG} for $\tilde{L}/\tilde{G}$ ratios of 0.5, 0.75, and 1.0 in order to evaluate the catalyst regeneration procedure. The CECE system will be expanded by the addition of a catalytic recombiner system to provide a liquid reflux at the stripping end of the column.

In addition, a tritium storage system will be installed as an interface between the CECE system and the cryogenic distillation system to enable the simultaneous operation of both units.

Management of high specific activity tritiated liquid wastes

G. E. Gibbs and J. J. Dauby

Background

Mound generates high-activity tritiated liquid waste from a variety of tritium handling operations and from stack effluent control systems [5-7]. There are two general categories of liquid waste which can be described as water (~1000 Ci/liter) and vacuum pump oil (~55 Ci/liter). Since 1972 these materials have been packaged for disposal in an improved container, and the work has been done in a glovebox facility. Handling is accomplished with virtually no release to the environment and no significant operator exposure. The facility, package, and operating experience have been described in detail in the literature [8]. Twenty-five liters of waste oil is sorbed on vermiculite in the polyethylene drum, whereas 27 liters of liquid water waste is sorbed on a cement-plaster mixture in the polyethylene drum. The packaging procedure thereafter is the same, that is sealing in a 30-gal drum and finally in a 55-gal drum.

The two drums containing octane waste (113 and 114) are positioned in the water in a like manner to the oil and water drums; however, the internal package is quite different: 250 ml of contaminated octane waste is sorbed on vermiculite contained by a 32-oz polyethylene bottle which is then sealed. Three of these bottles are sealed inside a 50-gal steel drum liner. This liner is then sealed inside a 55-gal steel drum, which is in turned sealed inside a 83-gal steel drum. All void volumes within the package are filled with vermiculite and asphalt.

These octane wastes were packaged in the 55-gal drum about 6 years ago and the 83-gal drum was added in 1977.

The 83-gal drum now becomes the waste package outer container and is immersed in water contained by a 146-gal drum. Here the 146-gal drum becomes the test container, whereas in the case of the water and oil waste drums, the 83-gal drum is the test container.

A study of the tritium release rate from actual burial packages was initiated in May 1975 and is continuing. The study consists of the individual immersion of representative actual burial packages in a known volume of water. The surrounding water is periodically sampled and analyzed for the presence of tritium; a release rate is then calculated from these data. Figure 1 is a schematic of a typical burial package under test.

Accomplishments

The total tritium permeation from each drum varies proportionally with the amount of tritium contained in the package (Figures 2-7). When this total permeation is converted to fractional

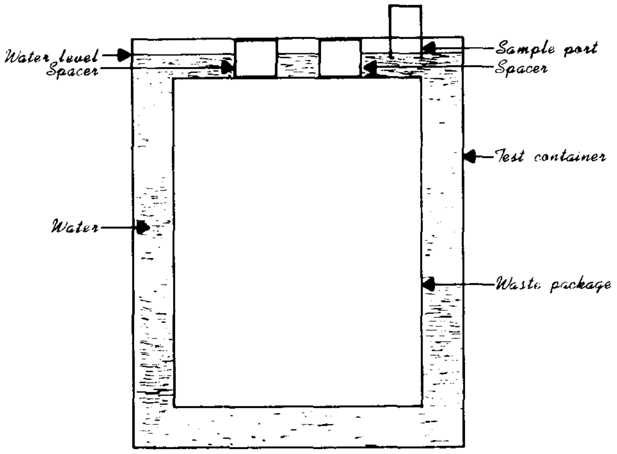

FIGURE 1 - Schematic of test package.

permeation (Figure 8) the rate for each drum is approximately equal. One conclusion to this is that the amount of tritium permeation is directly proportional to the amount of tritium contained inside the package. There does not appear to be any relationship to the type of liquid waste contained.

Slight differences in release rate from the drums are now beginning to appear (Figure 8). The three drums with the highest fractional permeation rate had the least amount of tritium when packaged. These are the two oil drums (217 and 218) and an octane drum (114). The fractional permeation rate from drum No. 318 has been increasing at a greater rate during the past 22 weeks. Future results will show whether the fractional permeation rate of these drums will level off as have the other drums, or continue to increase. The fractional permeation rate of the two octane drums appears to have leveled off at about the same rate as the oil and water drums for the same time period (Figure 8). This indicates a relationship between time since

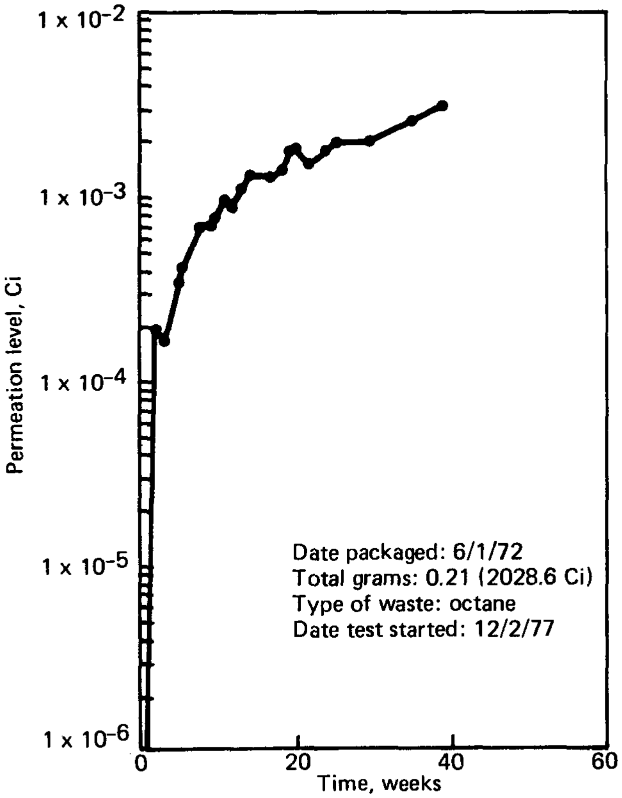

FIGURE 2 - Tritium waste package permeation rate for drum No. 114.

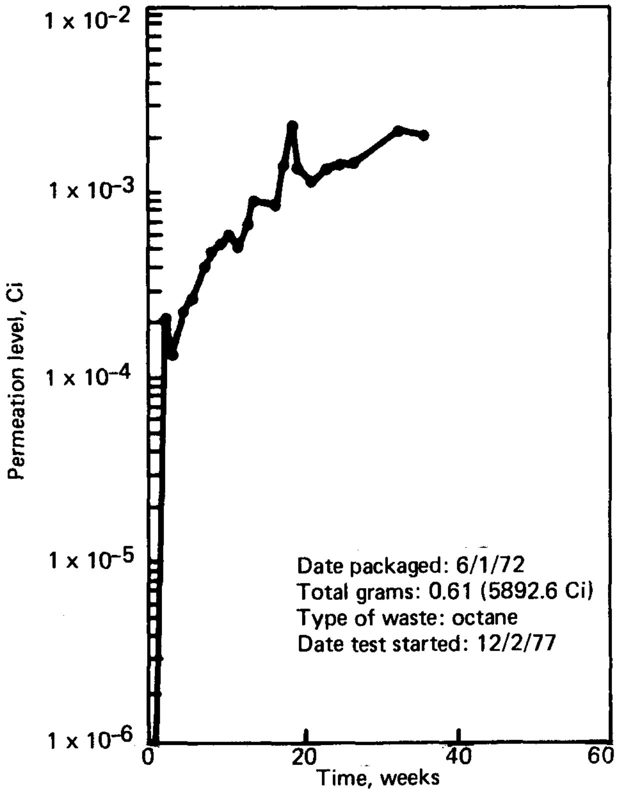

FIGURE 3 - Tritium waste package permeation rate for drum No. 113.

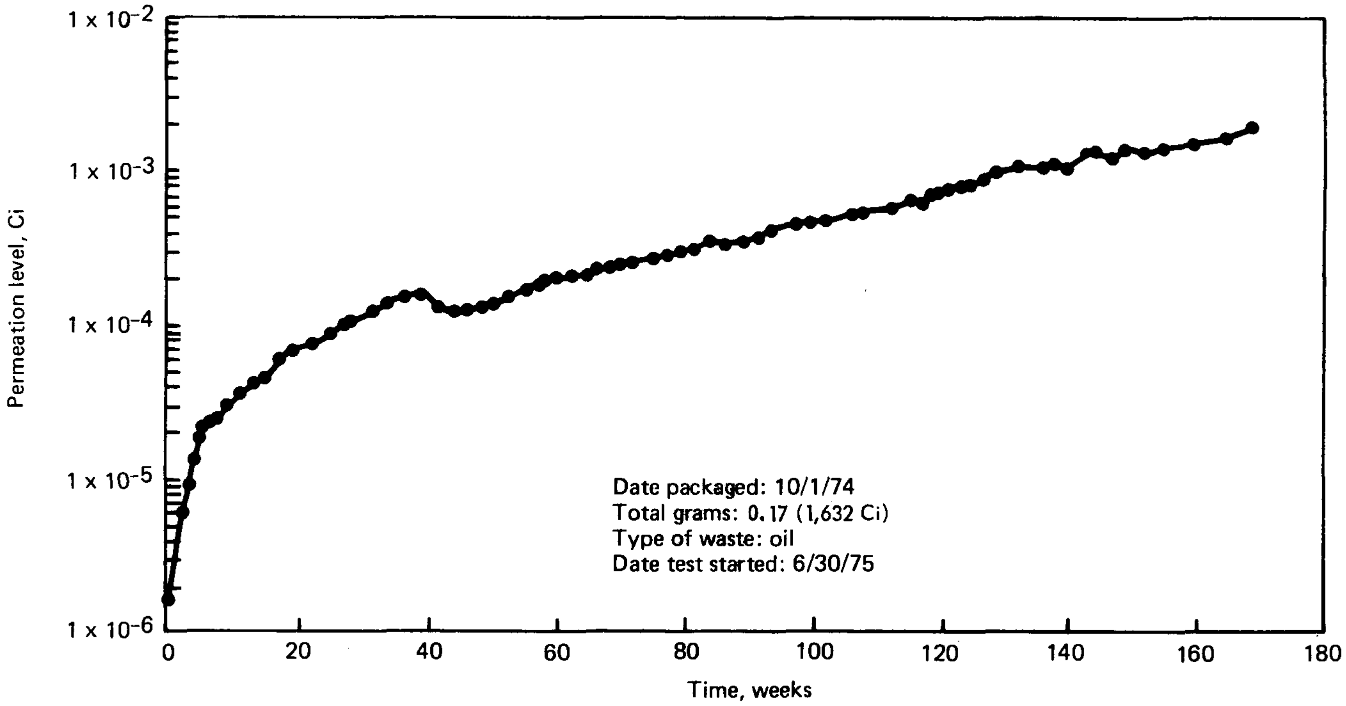

FIGURE 4 - Tritium waste package permeation rate for drum No. 218.

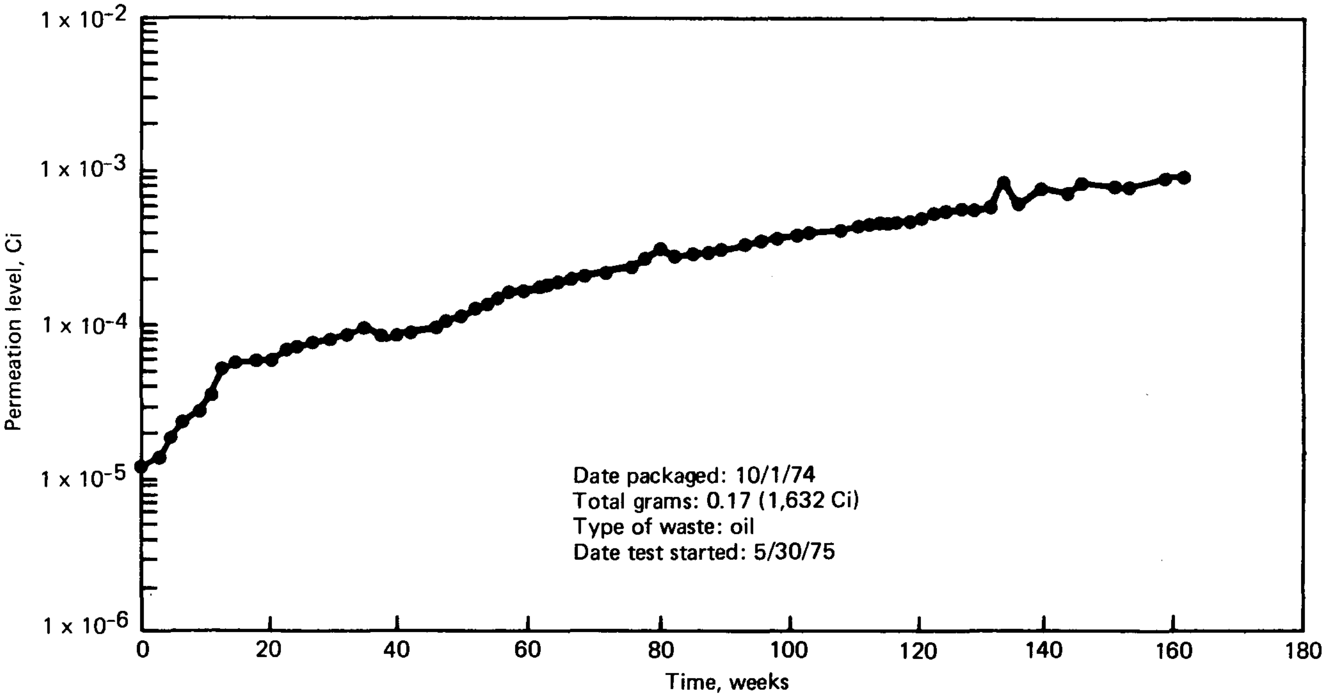

FIGURE 5 - Tritium waste package permeation rate for drum No. 217.

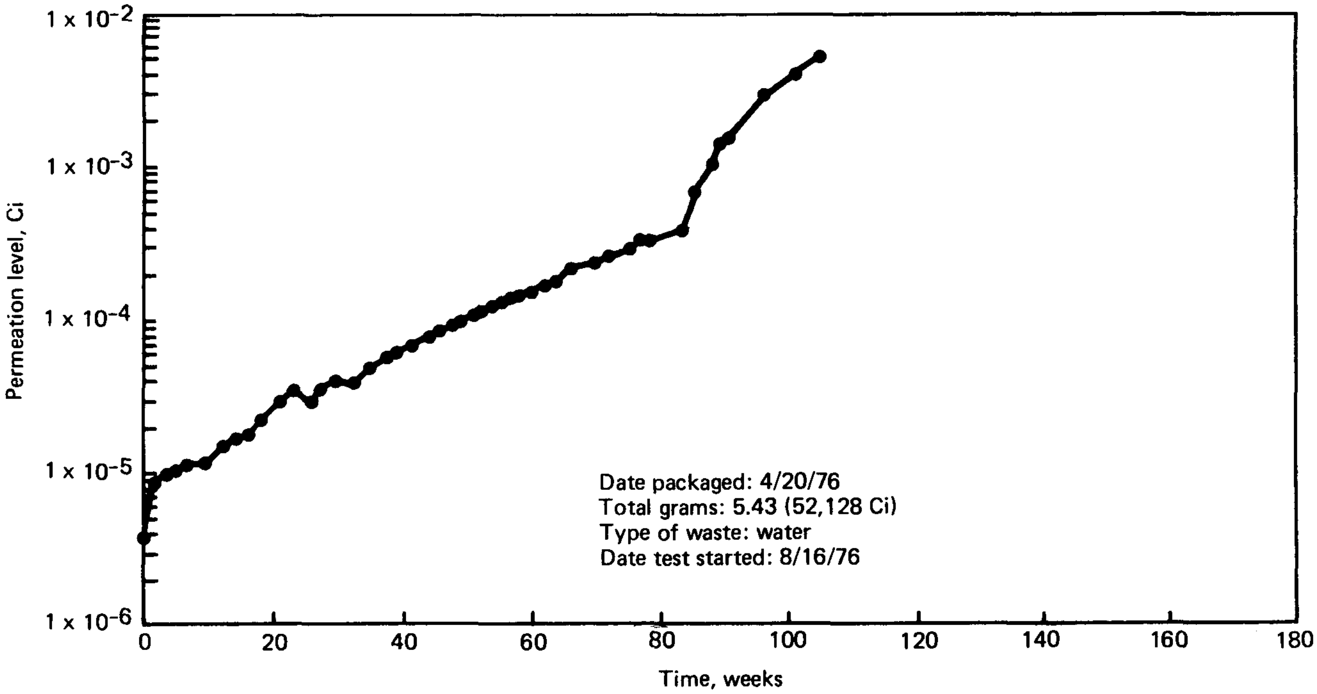

FIGURE 6 - Tritium waste package permeation rate for drum No. 318.

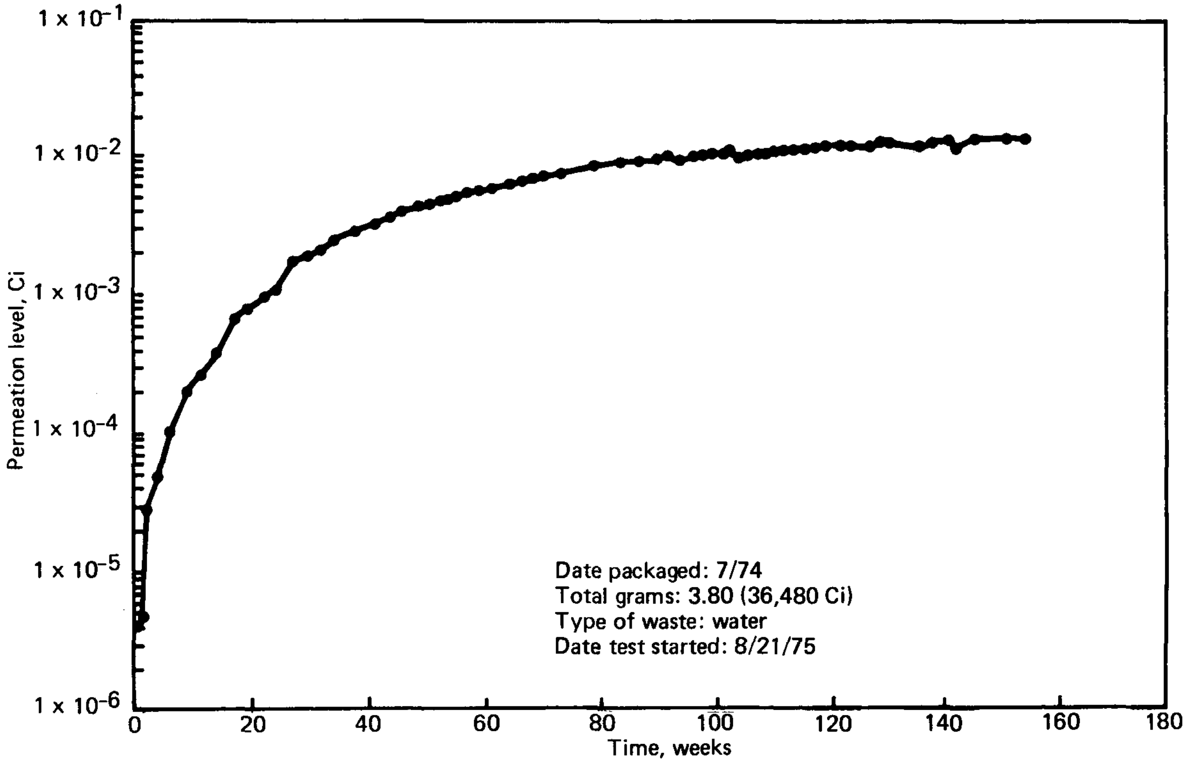

FIGURE 7 - Tritium waste package permeation rate for drum No. 205.

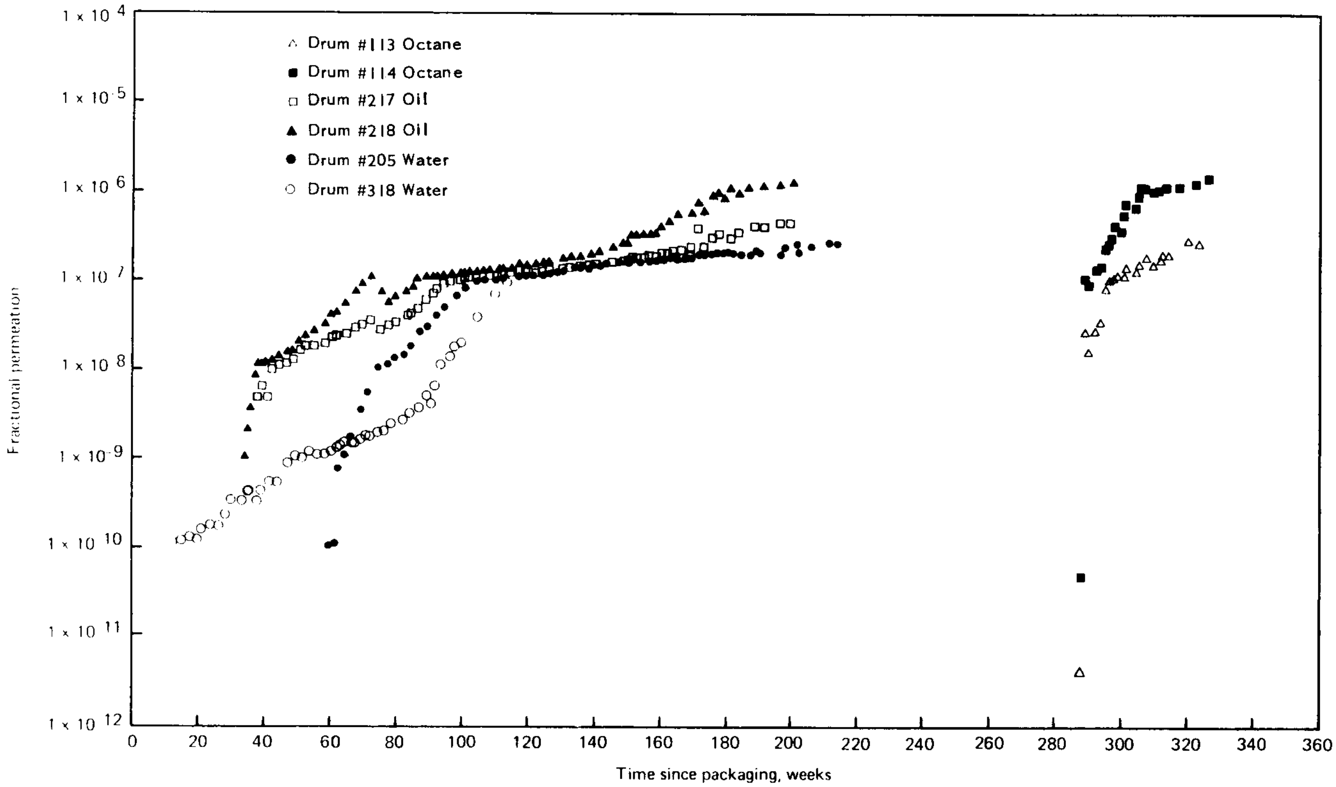

FIGURE 8 - Comparison of fractional releases from all drums.

packaging and the fractional permeation. All six drums in the study reached equilibrium at approximately the same rate and are leveling off at about the same rate.

Samples of oil taken from two different SW Building laboratories were assayed by scintillation counting. One sample from a vacuum pump in a tritium synthesis laboratory contained 140 Ci/liter and a sample of the oil from a vacuum pump in the mass spectrometer laboratory contained 1.84 Ci/liter. The tritium concentration of the oil actually in the pump in the synthesis lab was ∿3 times higher than this because the pump was flushed with 2 volumes of clean oil. The oil is routinely emptied into a 100-liter holding tank where it is sampled and assayed. In the last five years the tritium content of these 100-liter batches ranged from 10 to 185 Ci/liter with the average over the past 5 years being 55 Ci/liter.

Future Plans

The release test will be continued and future data will be reported.

Fixation of aqueous impregnated concrete

J. J. Dauby, R. E. Wieneke and
G. E. Gibbs

Background

Mound Facility, in cooperation with Brookhaven National Laboratory (BNL), has undertaken a program to test and develop the BNL method [9] for fixation

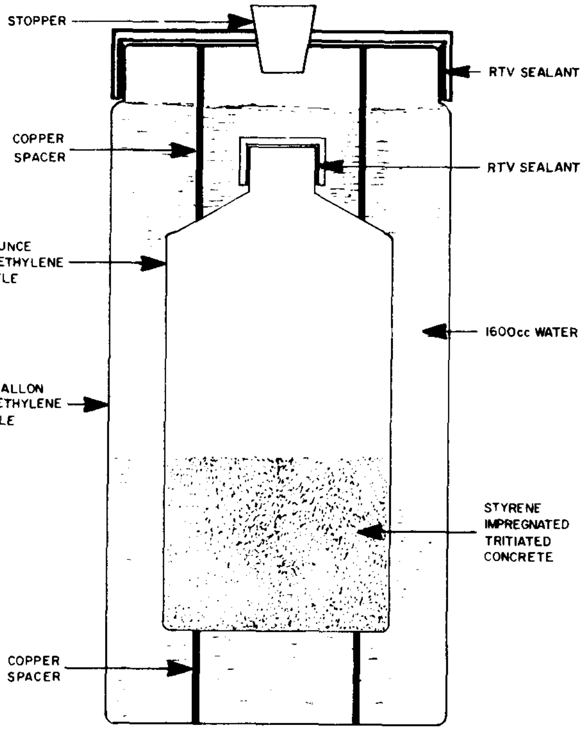

FIGURE 9 - Styrene impregnated tritiated concrete test.

of aqueous tritiated waste and adapt it to current procedures and facilities for packaging tritiated water waste. Concrete has been used at Mound since 1972 as a fixing agent for aqueous wastes containing 0.1 to 0.2 g of tritium per liter. In the BNL polymer impregnation technique, the cured concrete is impregnated with styrene monomer. The monomer fills the pores; then polymerization of the monomer binds and immobilizes the radioactive waste in the concrete. This decreases the tritium release from the concrete and reduces contamination hazards at the burial site. Because the styrene permeates the concrete the waste is, in effect, "microencapsulated". This will significantly reduce the tritium release rate in the event the containment is breached. Thus incorporation of the BNL procedure can improve the present waste package and allow us to establish a lower tritium release rate for a waste burial container.

As a method for transferring the BNL technology to Mound, "cold" samples were prepared using the techniques and parameters suggested by BNL. The details of this work have been given in previous reports in this series. Our work confirmed the work done at BNL.

A test program was instituted to measure and compare the release of tritium from tritiated concrete with and without styrene impregnation. All samples were prepared in 16-oz polyethylene bottles with a 2.625 in. (6.67 cm) diameter and filled to a height of 2.28 in. (5.79 cm).

Approximately 62.5 ml of tritiated water containing 386 Ci was injected into each of the 16 samples containing 250 g of dry mix. This is six times the concentration of tritium that is in a normal tritiated waste package. Hot samples consisted of triplicate mixtures of cement-plaster mixtures in a 1:1 volume ratio, and 10 samples of cement III (high early strength). Details of the preparation of these "hot" samples may be found in a previous report [10].

The samples were put into a test program to measure the release rate of the tritium. The test method (shown in Figure 9) consists of submerging each sample, still contained in the polyethylene bottle, in 1600 cm^3 of water which is sampled periodically to measure the tritium released from the sample.

Attempts were made to prepare full-scale, cold prototype burial packages. Earlier tests without adequate temperature monitoring revealed unexpected damage to the polyethylene drum liner. It was later learned that the BNL work was done using linear high-density polyethylene (LHPE), whereas the drum liners used by Mound were linear low-density polyethylene (LPE). As expected, tests showed that LHPE is more resistant to attack by styrene monomer than is LPE. Options for investigation included: 1) reducing the bulk temperature while styrene monomer is in contact with the drum liner, 2) reducing the contact time of the drum liner and the monomer, and 3) using drums of cross-linked high-density polyethylene (CLPE).

Accomplishments

The permeation data for the test samples are given in Table 1. No significant differences can be noted in the data on the first four groups: cement III (Groups 1 and 4), 1:1 by weight of cement and plaster, or the 1:1 by volume of cement and plaster (see Figures 10 through 13). The fifth group did not have styrene added, therefore it was not heated as the other groups. After 54 weeks there is no significant difference from the other four groups (see Figure 14). However, no significant difference is expected since the polyethylene bottle is much thicker than the layer of polystyrene and constitutes a considerable barrier to tritium permeation.

Samples 59 and 72 were prepared with cement III and styrene with 5% and 1/2% catalyst, respectively, as in Groups 1 and 4. They were not put into the test program until 25 and 21 weeks after their preparation and then with the polyethylene bottle removed. The amount of tritium that has leached into the water bath is 35 to 80 times greater than the diffusion of tritium through the polyethylene bottles. The data are shown in Figures 10 and 13 for Groups 1 and 4. The greatly increased tritium release from these samples is attributed to the removal of the polyethylene bottle. Without the polyethylene there is only a thin layer of polystyrene between the concrete and the water.

The effect of the catalyst concentration is shown in samples 59 and 72. The release from sample 72 is one-half the release from sample 59. This is caused by a lower rate of polymerization which yields a lower bulk temperature and results in the polystyrene containing fewer bubbles.

The sample data on the first four groups for the last 40 weeks show little increase in the amount of tritium diffusing through the polyethylene bottle from the concrete blocks. An equilibrium is being approached. The samples of Group 5 which had no styrene added may be showing a greater increase of tritium permeation.

Examination of the data shows that all samples taken during the 9-week period from September 6 to November 8, 1977, were 1-μl aliquots, whereas all other samples have been 10-μl aliquots. Data points from 1-μl samples are more erratic and slightly higher than the curves fitted to the 10-μl sample points. For Groups 1, 2, and 3 this 9-week period (Figures 10, 11, and 12) was from Week 15 to Week 24. For Group 4 (Figure 13) this period was Week 12 to Week 21. Finally, for Group 5 (Figure 14) this period was Week 4 to Week 13.

The probable cause for the difference between Samples 66, 67, and 68 of

Table 1 - PERMEATION TESTS

Group	Sample No.	Curies	Preparation Date	Type	Water/Dry Mix Ratio	Styrene (%)	Catalyst (%)	Polymerization Temperature (°C)	Total Permeation (Ci)	Group Fractional Release
1	55	0	5-10-77	Cement III	0.250	20	5	40		
1	56	0	5-10-77	Cement III	0.250	20	5	40	None	
1	57	386.6	5-10-77	Cement III	0.250	20	5	40	2.67	7.0×10^{-3}/65 wk
1	58	386.5	5-10-77	Cement III	0.250	20	5	40	2.43	
1	59	386.3	5-10-77	Cement III	0.250	20	5	40	200.4	
2	61	0	5-12-77	1:1 by weight	0.250	23	1/2	55	None	
2	62	386.1	5-13-77	1:1 by weight	0.250	23	1/2	55	2.24	6.0×10^{-3}/64 wk
2	63	385.9	5-13-77	1:1 by weight	0.250	23	1/2	55	2.47	
2	64	386.6	5-13-77	1:1 by weight	0.250	23	1/2	55	2.34	
3	65	0	5-12-77	1:1 by volume	0.305	20	1/2	55	None	
3	66	386.6	5-13-77	1:1 by volume	0.305	20	1/2	55	1.31	6.0×10^{-3}/65 wk
3	67	386.9	5-13-77	1:1 by volume	0.305	20	1/2	55	3.14	
3	68	386.6	5-13-77	1:1 by volume	0.305	20	1/2	55	4.93	
4	60	0	5-12-77	Cement III	0.250	20	1/2	55	None	
4	69	386.5	6-03-77	Cement III	0.250	20	1/2	55	2.85	4.2×10^{-3}/62wk
4	70	386.6	6-03-77	Cement III	0.250	20	1/2	55	2.27	
4	71	386.3	6-03-77	Cement III	0.250	20	1/2	55	1.11	
4	72	386.5	6-03-77	Cement III	0.250	20	1/2	55	97.8	
5	73	386.6	7-22-77	Cement III	0.250	None	None	None	1.80	4.2×10^{-3}/54wk
5	74	387.0	7-22-77	Cement III	0.250	None	None	None	1.52	
5	75	193.4	7-22-77	Cement III	0.250	None	None	None	1.14	

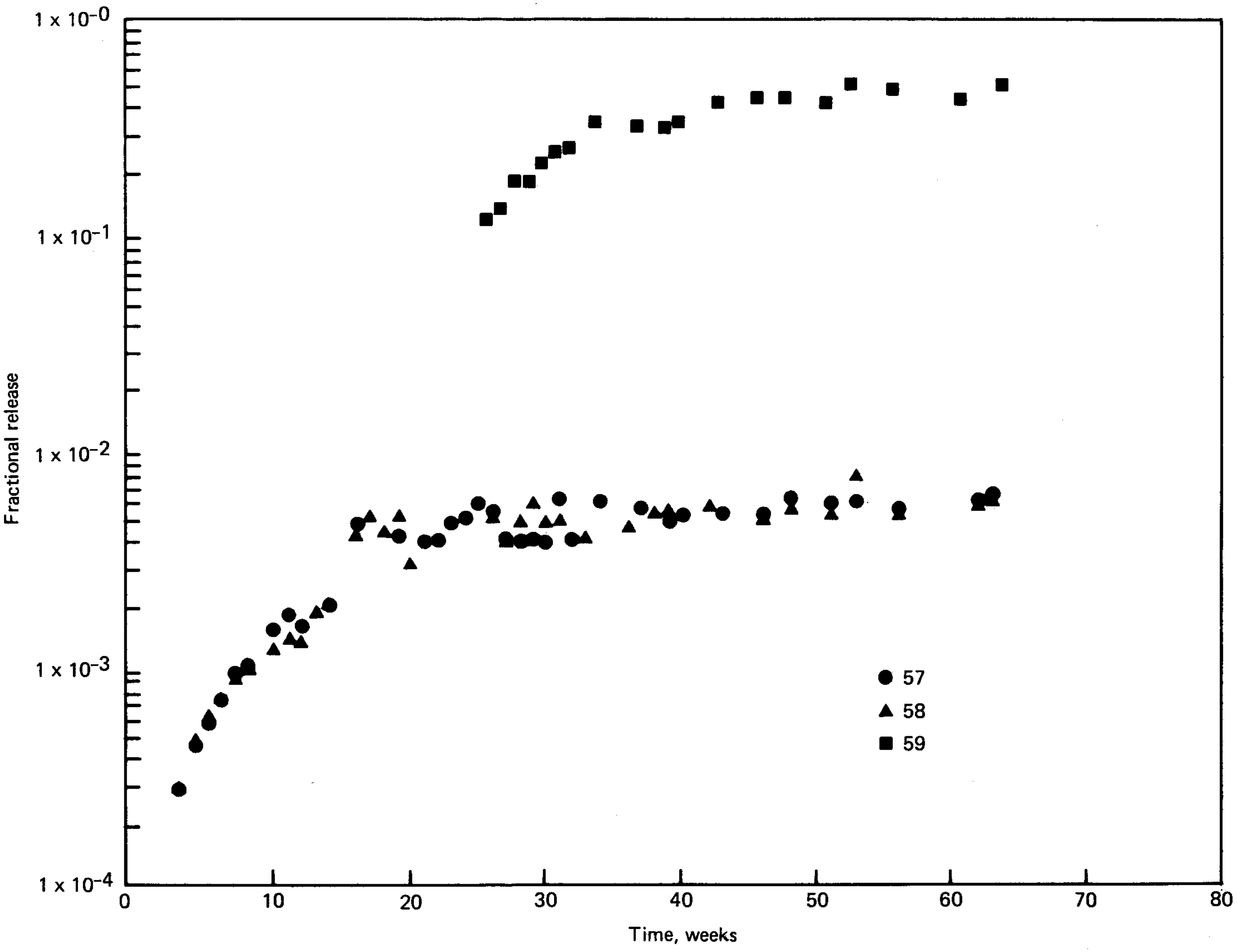

FIGURE 10 - Fractional release from cement III samples, Group 1.

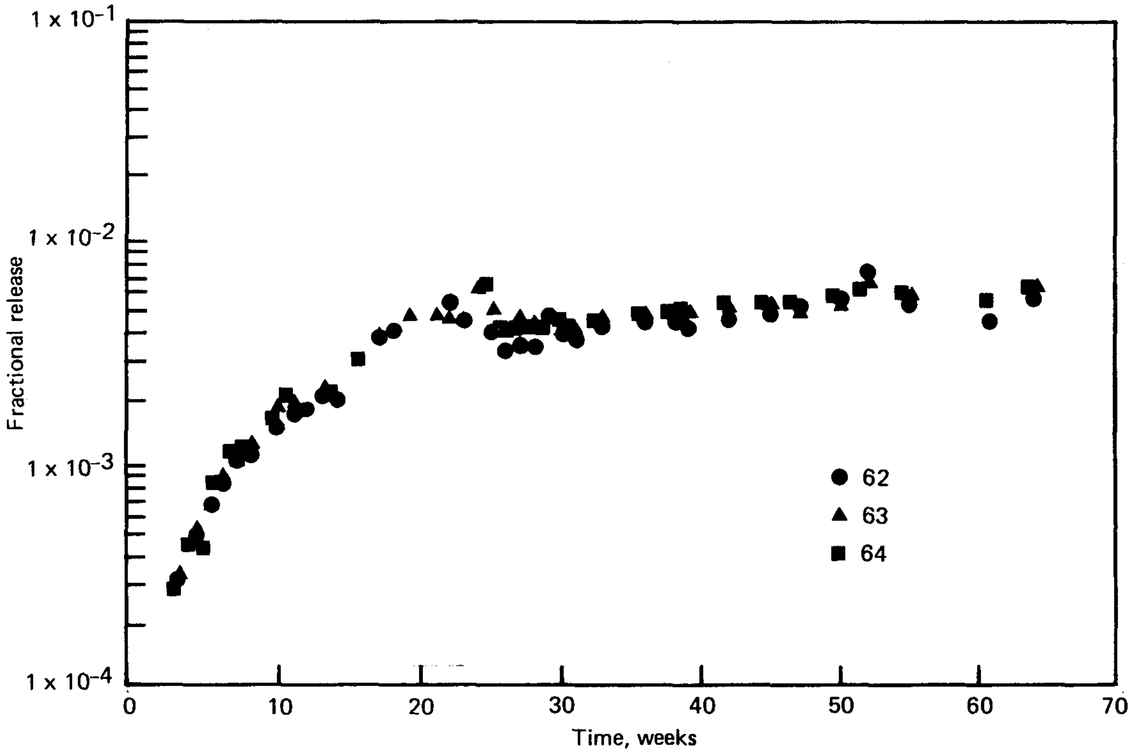

FIGURE 11 - Fractional release from cement/plaster (1:1 by weight), Group 2.

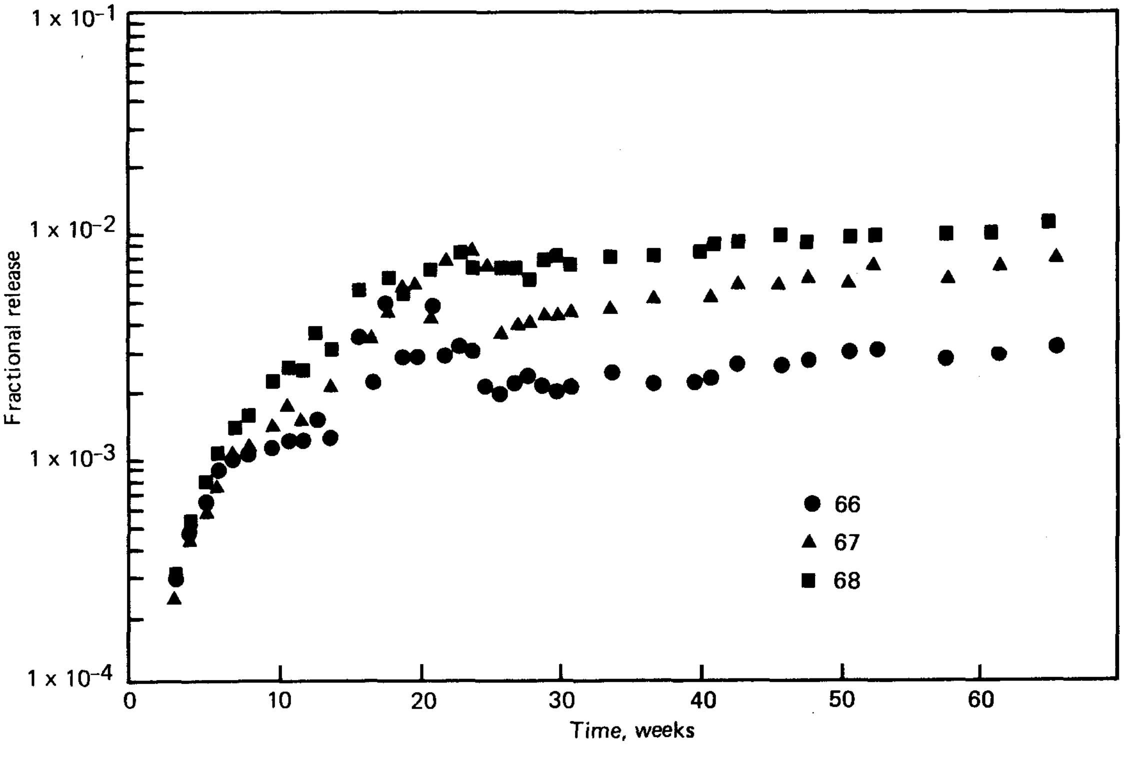

FIGURE 12 - Fractional release from cement/plaster (1:1 by volume), Group 3.

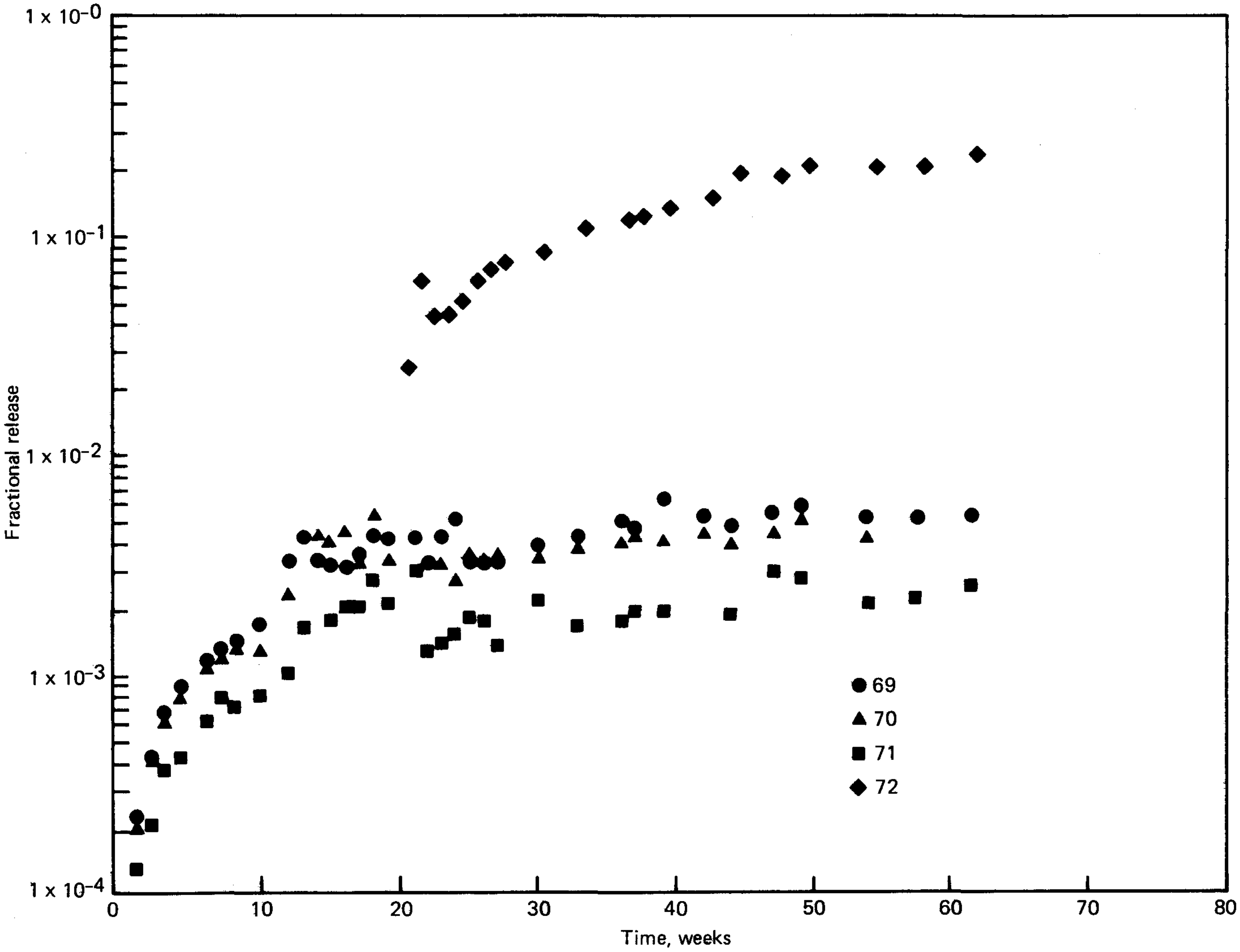

FIGURE 13 - Fractional release from cement III, Group 4.

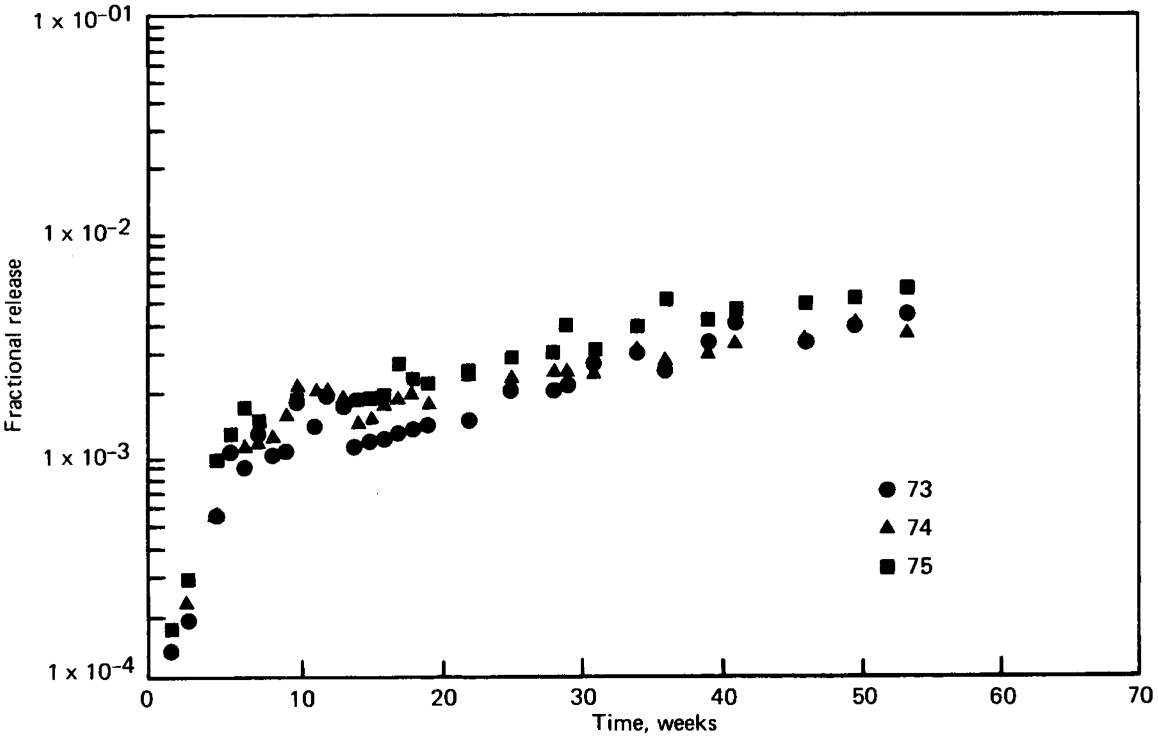

FIGURE 14 - Fractional release from cement III, Group 5.

Group 3 and differences in the other groups is the variation in the stock polyethylene bottles.

Sample 59, which had the polyethylene bottle removed, is approaching tritium equilibrium with the leach water. In this sample 200 Ci of tritium (52% of the original amount) had moved from the concrete into the leach water, which is 80 times greater than the 2.5 Ci released from similar samples still in polyethylene bottles

In further attempts to develop a workable procedure for preparing a full-scale cold prototype burial package, test drum No. 5 was prepared. Tests were run to determine the thermal characteristics of drum No. 5, and parameters are being finalized for the next polymerization.

Full scale (cold) waste drum No. 5 was polymerized with excellent results. The package consisted of 114 kg of cement inside a 27-gal polyethylene liner, inside a 30-gal steel drum. Twenty-eight liters of water were injected into the center and the cement was allowed to cure.

After heating to 45°C, 28 kg of styrene monomer containing 0.75% catalyst was allowed to soak into the cement. After 4 hr the heaters were increased to maximum, then turned off when the temperature at the circumference of the cement reached 60°C. Polymerization occurred about 14 hr after styrene addition and the maximum temperature at the center of the package was 90°C.

Future plans

Funding for this project ended 9/30/78. A final report will be written on the progress achieved during the past 3 years in the area of polymer impregnated tritiated concrete (PITC). Funding is expected early in FY 1979 for a general plant project (GPP) to permit the application of the PITC concept to all high-activity aqueous tritiated waste at Mound. A greatly reduced level of development effort will be maintained in the PITC area. Some final polymerization experiments will be performed prior to the preparation of a full-scale, "hot" test package. Development will be continued as required to define equipment and operating parameters for the new Tritiated Waste Fixation Facility. The five groups of samples currently in the test program will be maintained and checked periodically to determine the tritium release rate.

Gas generation by self-radiolysis of polymer impregnated concrete

W. Tadlock and G. Abell

Background

The procedure for processing of tritiated waste water for burial purposes developed by Brookhaven National Laboratory makes use of a catalyzed styrene monomer to microencapsulate concrete made from tritiated water. Self-radiolysis of the polymer impregnated concrete generates gas, the volume and constituents of which merit study.

A matrix consisting of four polymer and four nonpolymer concrete samples each having an activity of 10 Ci/cm^3 was made up. This activity, approximately 10 times that associated with Mound waste water, was chosen to obtain accelerated results. Tritiated distilled water was used in mixing the concrete for four of the samples (two polymer and two nonpolymer). The concrete for the remaining samples was mixed using tritiated waste water; the activity of which had been increased with the addition of the higher-activity tritiated distilled water. Two samples containing 10 g each of the tritiated distilled water alone were also made up. The purpose of these two samples was to obtain base line pressure buildup information of the tritiated distilled water without fixation.

These samples were contained in stainless steel storage vessels equipped with a pressure transducer and reusable seal incorporating a knife-edge flange with a copper gasket. The range of the pressure transducer used for the tritiated water samples was 0-30 psia (0-207 kPa) while that for the remaining samples was 0-100 psia (0-690 kPa).

Each concrete sample was made up using 10 g of tritiated water to 40 g of No. 1 Portland cement. After aging in a sealed container for 11 days, 10 g of a catalyzed styrene monomer (0.5% catalyst) was added to four of the samples. These four samples were then heat treated at 55°C overnight in order to polymerize the styrene. The initial cover gas used for all samples was argon containing 8.3% nitrogen and 2.3% oxygen as the major impurities.

Accomplishments

Figure 15 illustrates gas generation due to self-radiolysis of the tritiated water and concrete samples. This data has been normalized to STP conditions with the use of the following equation:

$$\mathrm{cm}^3\,(\mathrm{STP})/\mathrm{Ci} = \frac{P_1 - P_0}{760}\,\frac{V}{\mathrm{Ci}}\,\frac{273}{298}$$

where P_1 = Final Pressure (Torr)
P_0 = Initial Pressure (Torr)
V = Void Volume
Ci = Activity of material in curies

The gas generated from the concrete samples is nearly the same for a period of 90 days whereupon the gas generated from the polymer impregnated concrete begins to exceed that of the nonpolymer samples. In addition, comparison of the gas generated from the concrete prepared with tritiated distilled water versus that from the tritiated waste water does not show any large difference. Therefore the gas generation data was averaged to give two sets of data, one for the polymer impregnated concrete and one for the nonpolymer concrete. At the end of a period of 235 days the cover gas from the concrete samples prepared with tritiated waste water was analyzed; the mass analysis indicated that 84-86% of the gas generated was hydrogen, the remainder being mainly helium-3 with very small amounts of HT and HD. Mass analysis of the cover gas over the tritiated water after a storage time of 75 days gave essentially the same results.

Future plans

The pressure buildup data will continue to be monitored for the concrete samples.

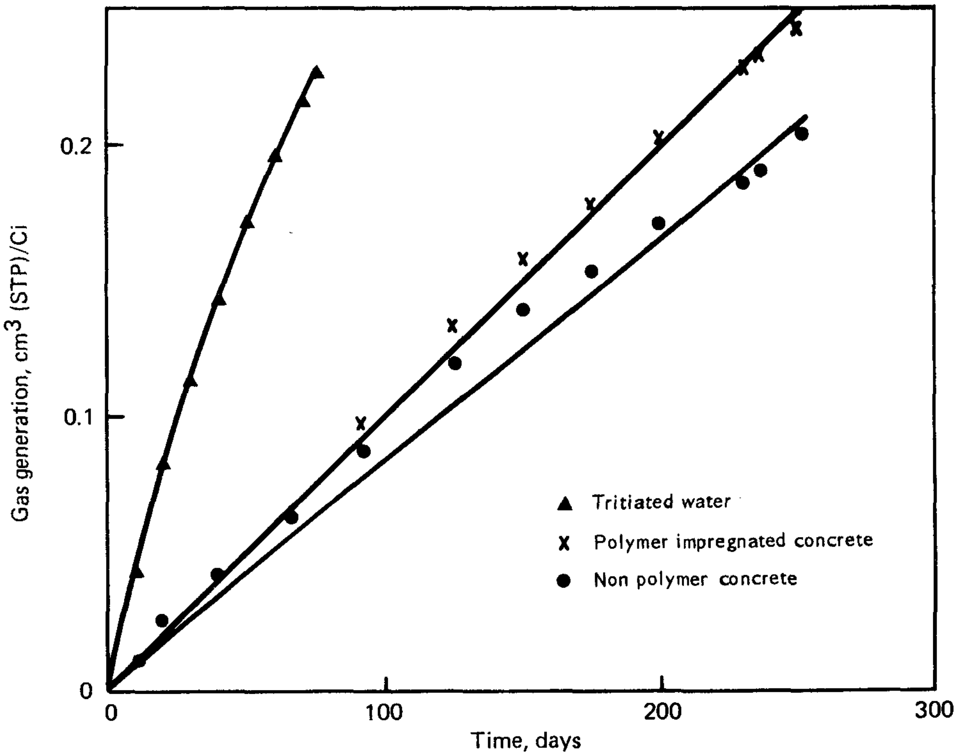

FIGURE 15 - Gas generation of tritiated water, polymer, and nonpolymer concrete.

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