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Washington 16, D. C.
Printed in U. S. A.

SOIL ADSORPTION OF RADIOACTIVE WASTES AT LOS ALAMOS

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The disposal of radioactive wastes by discharge to the ground has been practiced at Oak Ridge (1), Hanford (2), and Savannah River (3). During the early years of operation at Los Alamos, N. Mex., all wastes were discharged to seepage pits or to canyons. About six years ago this practice was stopped and chemical precipitation treatment plants (4) were installed. The areas receiving these plutonium-bearing wastes have been repeatedly monitored since that time, and no appreciable movement of plutonium through the soils has been noted. It has been observed, however, that the concentration of plutonium in the soil of a canyon receiving low-level wastes has progressively moved downstream. This concentration is not high and, although it is measurable, it is still within acceptable tolerance levels. The movement has not been extensive and is confined within the limits of the Los Alamos Project. Because there was some movement, it was deemed advisable to investigate the travel of plutonium through the local soils under varying conditions. Since there is a possibility that wastes containing strontium-90 and cesium-137 might be produced by the Laboratory in the near future it was decided to investigate these isotopes as well.

The Pajarito plateau, upon which Los Alamos is located, is underlain by Bandelier tuff erupted from the Jemez volcano during a recent geologic period. The Bandelier tuff has been subdivided into three member units which, from youngest to oldest, are the Tshirege, Otowi, and Guaje members. The Tshirege member forms the massive pinkish-orange colored cliffs that are the canyon walls in the Los Alamos area. It ranges in thickness from 100 to 200 ft near the Rio Grande to about 1,000 ft under the town site and shows general, irregular jointing and locally well-developed columnar joints. The Otowi and Guaje members, ranging in thickness up to about 200 and 50 ft, respectively, underlie the Tshirege throughout most of the plateau and are best exposed in the lower reaches of the canyons which are cut into the plateau.

The Tshirege member of the Bandelier tuff is composed mainly of welded tuff resulting from two or three ash flow eruptions from the Jemez volcano. The rocks of the Tshirege member generally are porous and friable but only slightly permeable. The permeability varies from 1 to 5 gpd/sq ft under one foot head. The ion exchange capacity for the Tshirege member, which was the tuff selected for this study, varies from 0.5 to 3 millequivalents per 100 grams. The Otowi member is a single thick pumice flow with some ash-fall and water-washed debris at the top, in some locations.

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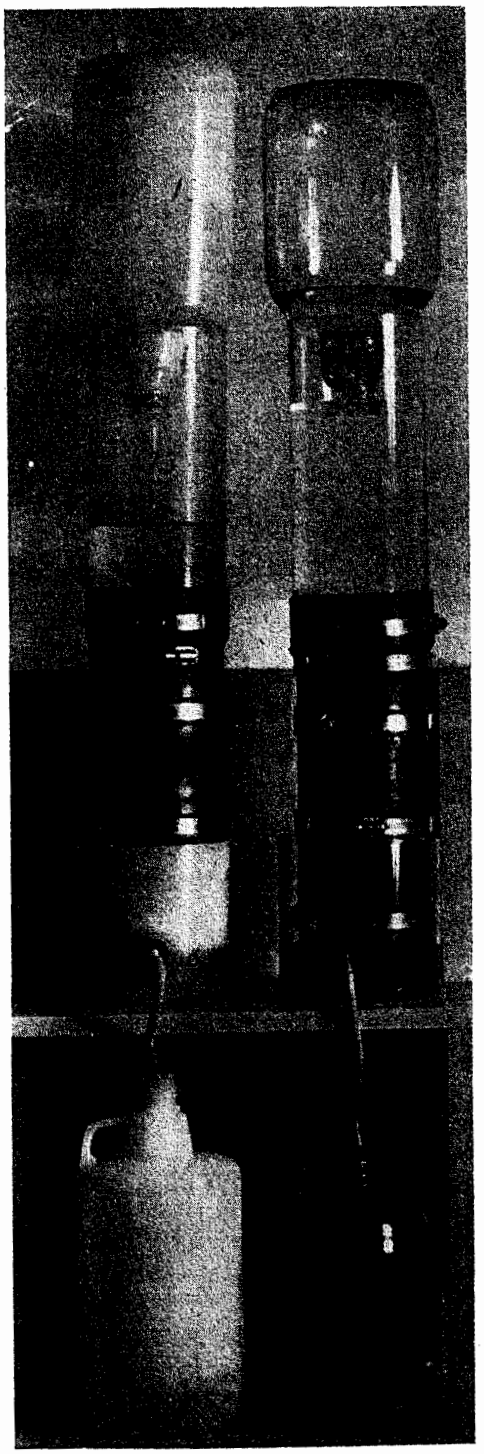
The Guaje member consists of massive unconsolidated pumice.

Methods

Cylindrical cores of tuff about 4¾ in. in diameter and varying in length from 4 to 18 in. were cut from unweathered rock with a core drill. The cores were sealed in a neoprene jacket which extended 3 in. above the upper surface of the core. A 4-in. lucite tube was inserted in the open jacket and served as a reservoir for the applied spike solution. Solutions of the respective nuclides were prepared so that 1×10^6 cpm/l were obtained. The flow of solution was controlled by adjusting the length of a tube attached to the neck of one-gallon jugs which contained the various spikes. Flow was regulated by adjusting the head so that 100 to 200 ml/hr of effluent would be collected in a glass dressing jar equipped with a delivery spout and delivered to a second gallon jug. The arrangement is shown in Figure 1. One of the set-ups is shown to use a polyethylene jug and receiving reservoir. The use of this material was necessitated by the determination of silicon, since it was felt this element might have some bearing on the retention or release of the various nuclides.

Samples were collected at 24-hr intervals or as required and the volumes recorded. At the time of sample collection 1 ml of each sample was plated on a stainless steel planchet, dried, and counted in an alpha or beta counter as required; gamma counts were determined on a 10-ml sample of the effluent. With strontium solutions, in many cases, original plates were counted two weeks later to determine the growth of yttrium. In the case of those effluents containing a mixed spike, radiochemical separations were made.

When increasing activity of the effluent indicated breakthrough of the nuclide, the core was dismantled, dried and autoradiographs made to deter-



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mine the distribution of the nuclide within the core. With some cores the application of the nuclide was stopped and leach solutions of various cations were applied to the core.

Determinations of pH, nitrate, calcium, sodium, alkalinity, and silicon content were made according to "Standard Methods" (5).

The weight of radioactive isotopes used in a study of this type is extremely small. For example, the drinking water tolerance for plutonium is 1.5×10^{-6} μc/ml which amounts to about 20 parts per trillion and in the case of Sr^{90} the figure is about 50 parts per quadrillion. At these extremely low concentrations, the chemistry of the various elements sometimes does not follow accepted patterns and ordinary ion exchange phenomena do not always pertain to the particular study. The removals are rather efficient, however, and it is possible, by passing a cesium spiked solution through a tuff core to reduce the concentration from 2×10^{-8} mg/l to 2×10^{-11} mg/l. It is obvious from this that the concentration and nature of the stable ionic components of the water will be the determining factors in the ability of tuff, or any soil, to retain radionuclides.

Results

Pu^{239} Cores

Seven cores of varying length were fed water spiked with 1×10^6 cpm/l of Pu^{239} as $Pu(NO_3)_4$. The amount of water fed varied from 100 to 380 l and the time of the test varied from two to six months. In every case the retention of the plutonium was in excess of 96 per cent. The spike was added to tap and distilled water and there was no apparent difference between the two. An analysis of Los Alamos tap water is shown in Table I. In the case of the core receiving 380 l of water this amounts to an application of 380×10^6 cpm and represents about 1,520 in. of water or some 90 yr of Los Alamos rainfall.

TABLE I.—Analysis of Los Alamos Tap Water

Constituent	Concentration (ppm)
Phen. alk.	0
M. O. alk.	116
Total hardness	24
Calcium	9
Magnesium	0.4
Sodium	51
Chloride	5
Fluoride	1.2
Nitrate nitrogen	1
Sulfate	4.5
Silica	35
Total solids	214

Plutonium, because of its high atomic weight and valence, can be expected to be tightly bound on any ion exchange material and replace many other ions. The ion exchange capacity of the tuff cores was exhausted after the passage of only a few liters of tap water, yet the plutonium was retained in a small portion of the core even after several hundred liters had passed through.

Autoradiographs were made by placing photographic film on cores that were cut in half. As can be seen in Figure 2 the activity penetrated to a depth of about 2 in. At this rate it is highly improbable that any plutonium, discharged in this form, would reach the Los Alamos ground water some 1,000 ft below ground surface in the foreseeable future. It should also be noted that there are several "hot" spots (white) in the radiograph which indicate the presence of localized concentrations of high ion exchange material such as montmorillonite clays.

Later attempts were made to leach out the plutonium already adsorbed on the cores by solutions of sodium, calcium, and magnesium in concentrations of 100 ppm as well as a saturated solution of carbon dioxide. None of these solutions removed appreciable amounts of plutonium.

Since this study was directed toward

FIGURE 2.—Autoradiograph of tuff core—Pu^{239} studies.

basic work very little effort was expended on actual wastes or complexing agents. It has been pointed out that plutonium complexes quite easily and in this condition is removed from solution only with great difficulty (4). It is to be expected that, under these conditions, it would not be retained by soils.

Dilutions of plutonium salts in basic or slightly acid solutions show a tendency to form polymerized, hydrated molecules which are extremely large (6). These polymerized molecules are entirely too large to enter into an ion exchange reaction and could be expected to pass through almost any type of soil. In addition to this effect other experiments have indicated that complexing agents such as: versene, citric acid, soaps, etc., have an adverse effect on the retention of plutonium in tuff. The data are not complete enough to report on at this time. However, it is known that such agents do interfere with removal of plutonium from liquid wastes by chemical precipitation and they might be expected to show a comparable effect in soils. Experiments on the effect of these agents as well as other ions are in progress.

Cs^{137} Cores

Eight cores of varied length were fed water spiked with 1×10^6 cpm/l of Cs^{137} applied as $CsNO_3$. In addition one core received a spike of 10×10^6 cpm/l. Both distilled and tap water were used as diluents to bring the spikes to the required concentration.

Swope (7) has reported that cesium is not held tightly by the resin, Nalcite HCR. However, she points out further that there is a difference among the various synthetic ion exchangers in their ability to retain cesium. That

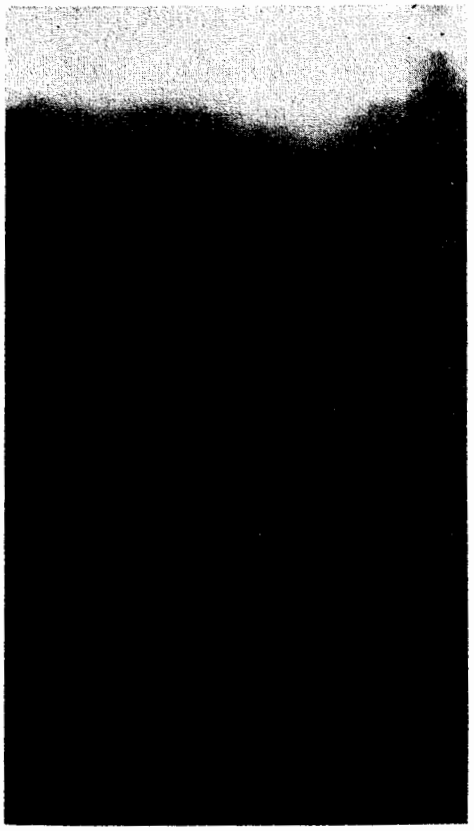


FIGURE 3.—Autoradiograph of tuff core—Cs^{137} studies.

there is a great difference between synthetic ion exchangers and soils has been borne out by the investigations reported here. It was found that cesium was bound very tightly by the tuff with little regard for other ions present in tap water. The lowest percentage retention was 99.6 and the highest was 99.99 +. Autoradiographs (Figure 3) support the above observations and indicate the cesium to be concentrated in the upper 1 in. of core volume. The amount of water passed through the core varied up to 330 l which represents some 1,300 in. of water added over a period of nearly six months. There was no apparent difference between the tap and distilled water solutions. Later attempts to dislodge the adsorbed cesium by leaching with solutions of sodium, calcium, magnesium, and carbonic acid were futile and the cesium remained stationary (see Table II).

Another investigation in progress, Project Green Thumb, has yielded some results which are applicable to the present study. Project Green Thumb is primarily a study of the effect of calcium on the adsorption from spiked soils of Sr^{90} and Cs^{137} by plants. When a spike of these two radionuclides was uniformly distributed through 25 sq ft of soil to a depth of 18 in., Sr^{90} was found to leach from the soil and to appear in the seepage from the bottom of the plots. The amount of Sr^{90} leaching from the soil was proportional to the concentration of calcium applied. The observation did not hold true for Cs^{137} which remained evenly distributed within the soil. The Sr^{90} from a mixed spike applied at the surface migrated downward through the soil but the Cs^{137} remained at the point of application. The results are similar to those observed when a mixed spike of Sr^{90} and Cs^{137} was applied to the surface of a tuff core.

TABLE II.—Summary of Tuff Core Study Results

Core	Nuclide	Core Length (in.)	Breakthrough Volume (l)	Total Volume Through (l)	Nuclide Removal (%)
1*	Pu^{239}	7.25	—†	331	96.2
2‡	Pu^{239}	7.00	—†	209	96.1
5*	Pu^{239}	6.25	—†	98	96.0
6‡	Pu^{239}	6.25	—†	99	96.7
3‡	Sr^{90}	5.50	30.0	37§	26.4¶
3A‡	Sr^{90}	7.25	50.0	41§	64.0¶
4‡	Sr^{90}	13.75	—†	23	97.0
4A‡	Sr^{90}	13.00	143	63§	88.0¶
7‡	Sr^{90}	6.25	27	22§	65.0¶
8‡	Sr^{90}	12.50	92	65§	73.0¶
13*	Sr^{90}	12.75	400	98§	88.0¶
19‡	Sr^{90}	9.75	20	29§	80.0¶
22*	Sr^{90}	9.75	—†	198	98.0
11‡	Cs^{137}	6.00	—†	62	99.9
14*	Cs^{137}	12.50	—†	330	99.7
16‡	Cs^{137}	6.80	—†	93	99.6
17‡	Cs^{137}	7.00	—†	98	99.9
18‡	Cs^{137}	7.50	—†	261	99.5
23*	Cs^{137}	10.30	—†	240	99.7

* Distilled-water solvent.
† No breakthrough observed.
‡ Tap-water solvent.
§ Throughput volume after breakthrough.
¶ Removal after breakthrough.

The movement of Cs^{137} was further investigated in the laboratory by passing a solution of Cs^{137} through a column of soil. When the nuclide, in tap water, was fed to the column it accumulated at the surface as was shown by radioautography. A spike applied to the surface of the soil was not moved by a tap water leach but remained as a thin band at the point of application.

Sr^{90} Cores

The investigation of the passage of Sr^{90} * through tuff cores proved to be the most interesting of the three nuclides studied. Sr^{90} is not nearly so tightly bound to the tuff core as plutonium or cesium. Furthermore, percolation through the cores demonstrated a method of separating strontium and yttrium. Something over 90 per cent of the activity coming through the core was due to Sr^{90}.

Seven cores were set up using tap and distilled water as the Sr^{90} solvent. An autoradiograph of a typical core after breakthrough is shown in Figure 4. It will be noted that the strontium has penetrated the full depth of the core. It is also interesting to note the many "hot" spots throughout the core where activity concentrated. This is similar to the phenomenon noted on the autoradiograph for the plutonium core. One other feature to be noted is the density gradation throughout the core indicating a wide variation of ion exchange capacities.

Two typical runs with tap and distilled water are shown in Figure 5. The shape of the curves is quite similar to the typical curves obtained in determining ion exchange capacities by the method of Frysinger and Thomas (8). The first sharp break in the curve is considered to be the breakthrough point. In these particular cores some 80 l were passed through before an appreciable amount of strontium appeared in the effluent. In the case of the tap-water solvent, after 140 l had been applied the concentration of Sr^{90} in the effluent was approximately the same as in the influent. This volume increased to over 200 l when distilled water was used as the solvent.

The slope of the curve for tap water is greater than for distilled water; however, both curves level off at about the same point. The difference in slopes may be explained on the basis of the competition of elements in tap water and of Sr^{90} for the adsorption sites on the adsorbing surfaces, hence a more rapid saturation of the core when tap water is applied. An explanation might be sought for the continued removal of strontium after the core has apparently been saturated with calcium. It is probable that some small amount of calcium is being replaced by strontium; however, the amount of calcium replaced would be so small as to be undetected by the analytical pro-

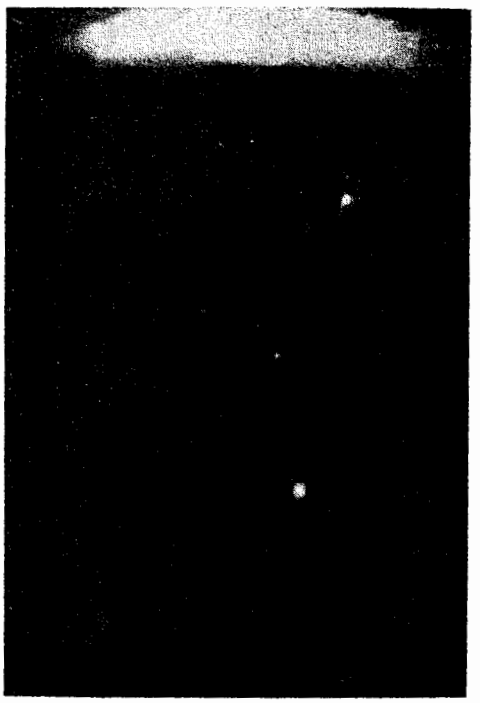


FIGURE 4.—Autoradiograph of tuff core—Sr^{90} studies.

* Sr^{90} feed implies an equilibrium mixture of Sr^{90} with its daughter, Y^{90}.

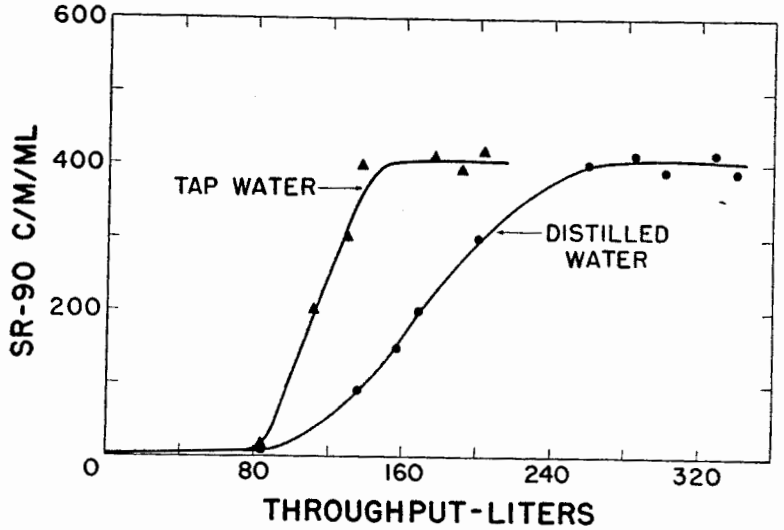


FIGURE 5.—Breakthrough curves for Sr^{90}.

cedures used. It seems reasonable to propose also that within the heterogeneous tuff there exists a wide variety of adsorbents and that some of these might bind the strontium molecule preferentially whereas little or no attraction is exhibited for the calcium molecule. Hence strontium is removed for a period of time after the calcium adsorbing sites have been satisfied. Such a phenomenon is not unknown and is the basis of differential removal of components from biological reaction mixtures by successive treatment with a variety of adsorbing resins. The various synthetic resins show a wide range of preference for different cations so it seems reasonable to assume that the components of tuff could easily demonstrate a similar phenomenon. Unfortunately, there is little, if any, information on ion exchange reactions in extremely low concentrations and much work must yet be done to understand these phenomena.

As noted above, over 90 per cent of the activity in the effluent of the column was due to Sr^{90}. Apparently the relatively high concentration of the divalent ions, calcium and magnesium, does not prevent the adsorption of the trivalent ion, yttrium. The effect of other trivalent ions such as aluminum and iron is being studied.

Mixed Nuclides

In general a waste is a mixture of several nuclides, therefore, the transport of mixed nuclides through tuff cores was investigated. Spikes of both Sr^{90} plus Cs^{137} and Sr^{90} plus Cs^{137} plus Pu^{239} were used as influent at a level of 1,000 cpm/ml each.

One core received a total of 190 l of a Sr^{90}-Cs^{137} spike. Sr^{90} broke through at about 80 l. No Cs^{137} appeared in the effluent after 190 l had passed.

Autoradiographs of the core, Figure 6, indicate the travel of the nuclides. The picture on the left, made with no shielding, reflects the concentration of both Sr^{90} and Cs^{137}. The picture on the right was exposed through 1/16 in. of lead and reflects only the location of Cs^{137}. It will be noted that the Cs^{137} was again concentrated in the top 2 in. of the core while the Sr^{90} was spread throughout.

The core treated with Sr^{90}-Cs^{137}-Pu^{239} mixed spike was 18 in. long. A total of 82 l of spiked solution was passed through. The volume of spiked solution was held low to avoid break-

through in order to obtain a distribution picture of the adsorbed nuclides.

Figure 7 shows the comparison of these radiographs. The picture on the left was made with aluminum foil shielding and reflects the strontium and cesium distribution. The center picture was exposed through lead and reflects the cesium distribution. The picture on the right is a series of photo-micrographs of nuclear track plates placed on the top 3 in. of the core. This type of plate is made by exposing microscope slides coated with photographic emulsion. The alpha particle, because of its relatively large size, does not penetrate the emulsion as is the case with X-rays, gamma rays and beta particles but instead travels along the emulsion leaving a track. A photomicrograph is then taken of the exposure to locate the alpha particle. The magnification of a typical "sunburst" shown in Figure 7 is 400 ×. From Figure 7 it will be noted again that cesium is located at the top, plutonium travels somewhat further, and the strontium penetrated to a depth of nearly 10 in.

Leaching Studies

Early in this investigation it was observed that Sr^{90} in tap water showed a different rate of movement in the core from that observed for the radionuclide in distilled water. In some

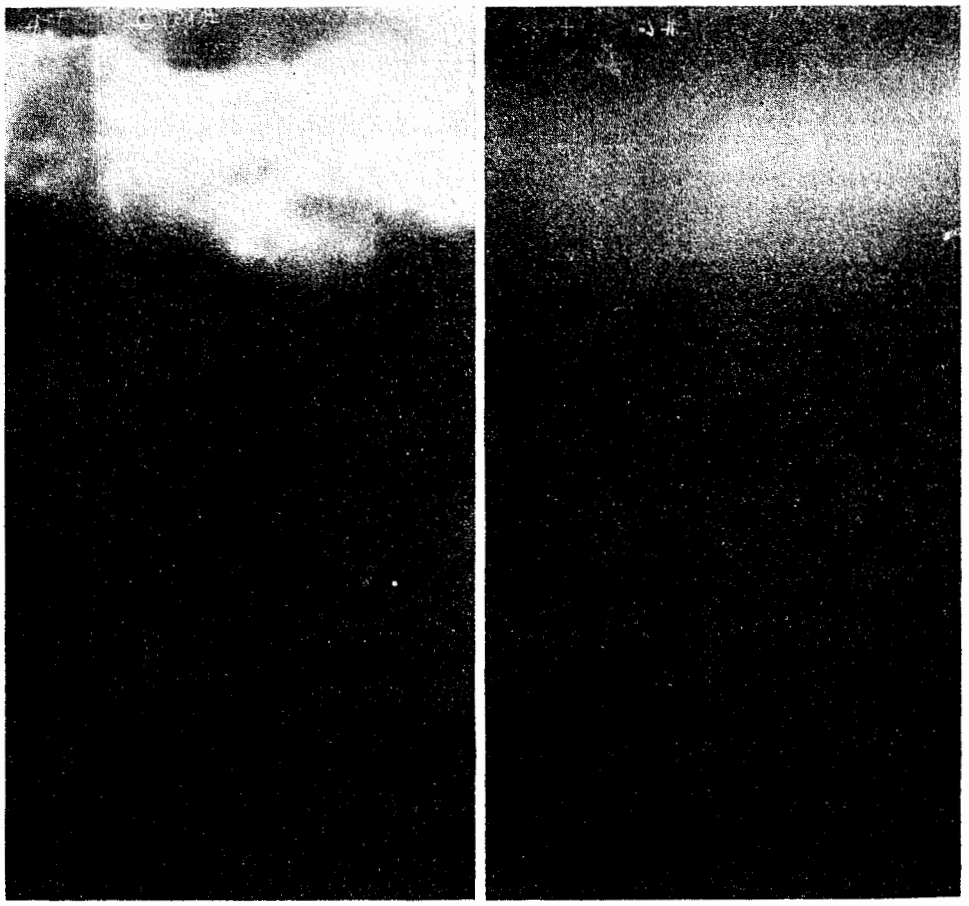
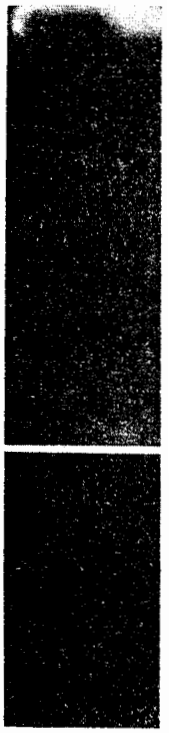


FIGURE 6.—Autoradiographs of tuff core which received a mixed solution of Cs^{137} and Sr^{90}. Picture on left unshielded; picture on right lead shielded.



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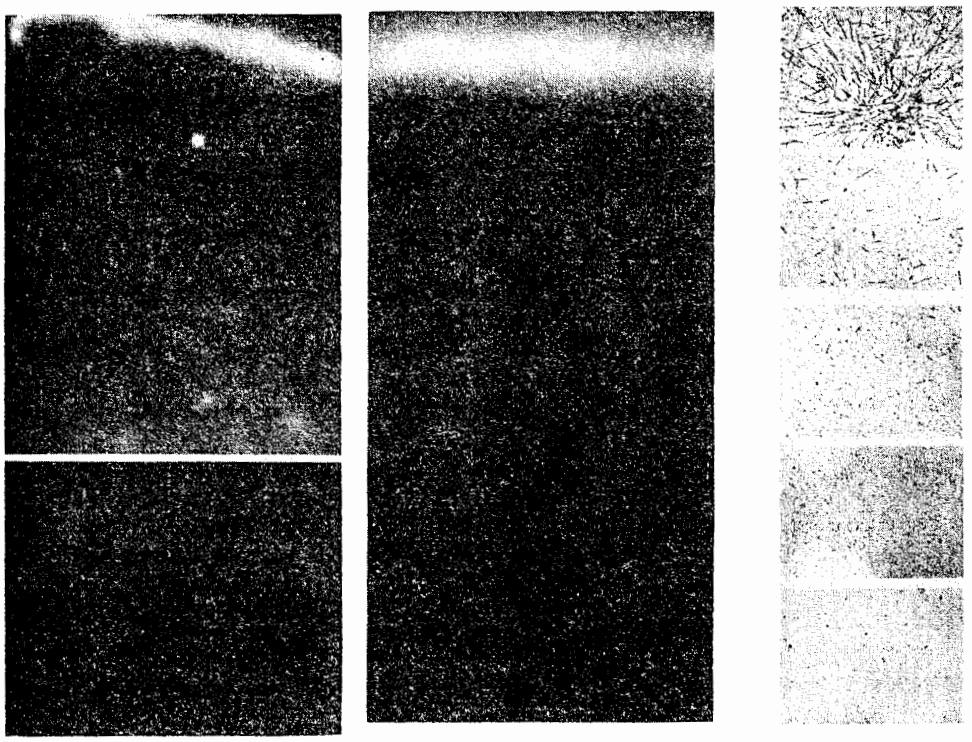

FIGURE 7.—Autoradiographs of tuff core which has received a mixed solution of Pu^{239}, Cs^{137}, and Sr^{90}. Picture on left aluminum shielded; center picture lead shielded. Picture on right is alpha track.

instances there were marked differences in rates of movement. It was theorized that certain elements in the tap water might be competing with the Sr^{90} molecules for position at the site of adsorption. The theory was further investigated by leaching cores which had previously received Sr^{90} up to the point of breakthrough. Referring to Figure 8, it will be noted that the first portion of the curve represents the application of the radionuclide in tap water to the core. This portion is typical of other curves obtained in the study except that it is foreshortened. The maximum level (flat portion) of the curve is followed by a rapid decrease in the release of nuclide as distilled water is applied to the core. This decrease has been observed when other similarly treated cores have been leached with distilled water.

Of interest is the increased amount of Sr^{90} released by a solution of H_2CO_3 prepared by saturation of distilled water with CO_2. It was noted that the released radioactivity was about 95 per cent Sr^{90} as was the case with the adsorption studies. Although H_2CO_3 was applied at a continuous rate to a total of 72 l, a sharp decline occurred in released counts after a small increment of leach solution had passed through the core. A similar pattern has been noted with all leach solutions employed. It is possible that the radionuclide is bound to different tuff components with different binding energies and after the weakly bound Sr^{90} has been released, no further leaching occurs until a more rigorous treatment is employed. Concurrently there is a release of calcium from the core and its concentration peaks at about the same point as the peak concentration of strontium. After peak

release the concentration curves of calcium and radionuclides are parallel. McHenry *et al.* (9) have shown the effect of pH on the adsorption of Sr^{90} by soil. Since the pH of the carbonic acid feed was about 4.4, it is possible that the leaching of tuff core is, in fact, a pH effect, i.e., a hydrogen regeneration of certain ion exchange components.

Leaching with sodium chloride resulted only in a decrease of calcium and strontium in the effluent. It is to be expected from the lyotropic series that sodium would have little effect on the release of ions higher in the series.

The application of magnesium chloride as a leach resulted in a rapid rise and equally rapid fall in the concentration of Sr^{90} released to the effluent. Magnesium would appear to be about as efficient as carbonic acid in releasing Sr^{90}, however, it is more effective in that it releases bound radionuclides that the latter does not release.

Magnesium also releases calcium from the core in comparatively high concentrations. After the peak has been reached the Ca and Sr^{90} concentrations show a parallel decrease.

The application of calcium resulted in little release of radionuclides; in fact the Sr^{90} concentration in the effluent from the core gradually dropped. The magnesium concentration likewise dropped to zero and remained at that level.

A mixed leach of calcium, sodium, and magnesium had no appreciable effect on the release of Sr^{90} from the core. The concentration of magnesium in the effluent rose rapidly to 100 ppm, the applied concentration, and remained at that level. The increased concentration to 150 ppm in the effluent is real but difficult to explain.

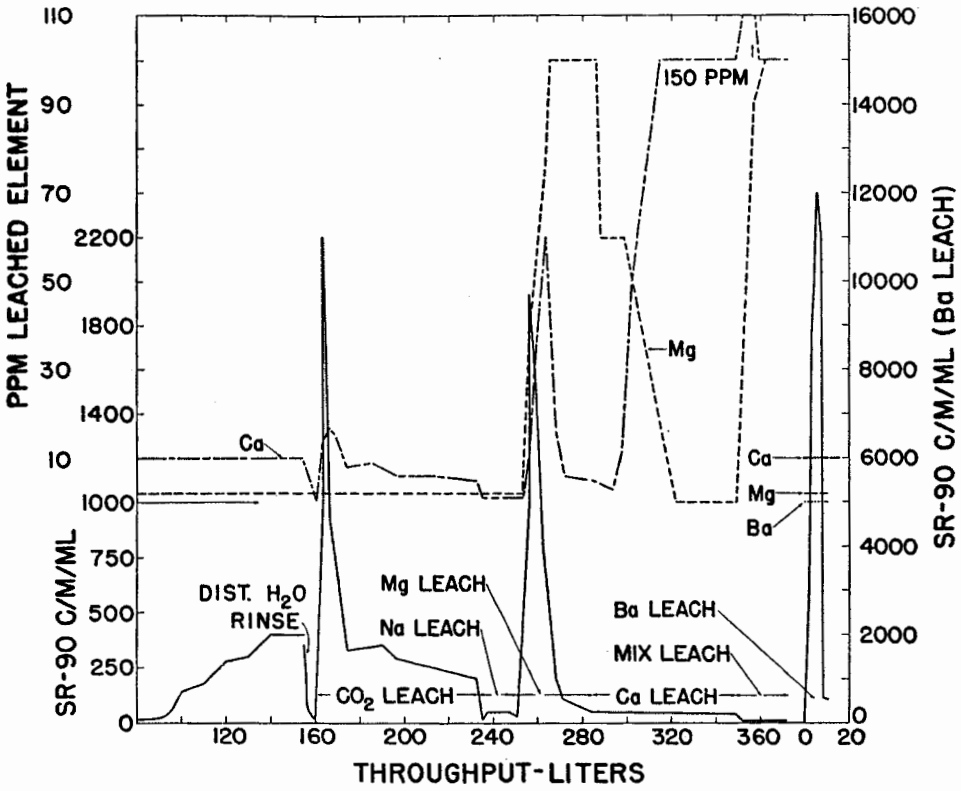

FIGURE 8.—Effects of leach solutions on tuff core—Sr^{90} studies.

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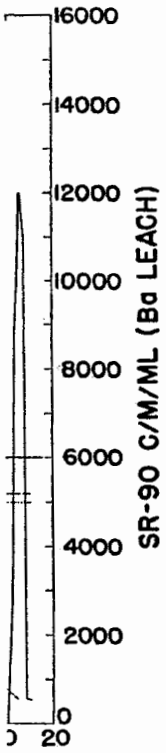

ies.

Due to its position in the lyotropic series, barium should be extremely efficient in the replacement of Sr^{90} adsorbed on an exchange medium. The application of barium chloride to a core previously receiving Sr^{90} resulted in the release of the radionuclide at above 12,000 cpm/ml (note the different scale for barium leach in Figure 8). This release is about 12 times the concentration of radionuclide applied to the core. Barium also released increased concentrations of magnesium and calcium as would be predicted.

Distilled water was added to rinse the barium from the core. This was followed by a leach solution of magnesium chloride resulting in an extensive release of radionuclide. It was expected the release would occur but at a lower value. The result is not explained by data obtained to date.

The leaching studies to date have shown the important effect of water percolating through soil on the movement of Sr^{90}. It is recognized that the concentrations of leaching ions employed in this investigation are above those which would normally be encountered in nature; however, it is well within the realm of possibility that such concentrations might appear in wastes containing radionuclides. The effect of such concentrations of Sr^{90} in a water table tapped for domestic use is self evident.

Discussion

Certain of the results obtained in this study are not in complete agreement with those of Swope (7) who found that both cesium and hardness broke through resin columns at about the same point. McHenry *et al.* (9) indicate a marked effect of the presence of cesium on the breakthrough of strontium. These studies did not show a similar effect. It is possible that the discrepancies are related to the different exchange properties of the basic adsorbents under study.

Orcutt *et al.* (10) have developed excellent expressions for dispersion and exchange phenomena applicable to radionuclides as they move through soils. Whether nuclides in low concentrations that are amenable to soil disposal will follow accepted physical laws is not known. Thomas (11) has stated that it remains to be proved that elements at concentrations of 10^{-7} M follow classical chemistry or the accepted physical laws of ion exchange. It is possible that a demonstration of the applicability of the mathematical treatment of Orcutt *et al.* (10) to solutions where concentrations of solute approach 10^{-7} M will indicate the nature of their chemistry.

It has been demonstrated that the tuff local to Los Alamos has a rather high capacity for the retention of various nuclides. This is especially notable since this particular material has an ion exchange capacity which is about as low as any to be found in nature. Cs^{137} is apparently very tightly bound to the tuff and resists leaching by any of the common agents. Pu^{239} likewise is readily retained by the tuff and from actual experience at Los Alamos, plutonium in wastes discharged into the ground appear to remain at the point of discharge. However, from what is known about the chemistry of plutonium, it is entirely possible that this nuclide could be released at some future time by inadvertent discharge of solutions such as versene in the same area. Work on this phase is being conducted at Los Alamos.

Sr^{90} is not retained by the tuff nearly so well as cesium and plutonium and it is much more easily released. It is becoming increasingly apparent that its fixation poses a most important problem, and it remains the controlling isotope in the disposal of radioactive wastes. Disposal of this isotope to soils is to be undertaken with extreme caution and only with foreknowledge of the nature of the soil and its capacity for the ions known

to be present in the waste. Because Sr^{90} can be leached by other ions, a disposal area receiving this isotope must be closely guarded so that no other wastes will be discharged which might contain concentrations of ions sufficient to dislodge the already adsorbed nuclide.

Acknowledgment

The authors wish to express appreciation to Julia Marie Wellnitz for the autoradiotography entailed in this paper.

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