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Separation of Carrier-Free ^{90}Y from ^{90}Sr by SLM Technique Using D2EHPA in N-Dodecane as Carrier

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Solvent extraction studies on Sr^{2+} and Y^{3+} are carried out from varying concentrations (0.01–6.0 M) of nitric acid using di-(2-ethylhexyl)phosphoric acid (D2EHPA) as extractant. Extraction of yttrium is observed to be higher than that of strontium at all the acidities and is found to increase substantially with decreasing concentration of nitric acid. Practically negligible extraction ($D < 10^{-3}$) of Sr^{2+} is observed from feed solutions containing nitric acid in the range of 1.0 to 4.0 M. These solvent extraction data are used to optimize the transport of ^{90}Sr and ^{90}Y across the supported liquid membrane (SLM) individually as well as from their mixture (due to insitue growth) under different experimental conditions. Selective separation of ^{90}Y (>90%) from ^{90}Sr is obtained in 6 h, when the concentration of nitric acid in feed is kept at 1.0 M and that of receiving phase is maintained at 4.0 M. 20% D2EHPA in n-dodecane is found to be the optimum carrier concentration for the efficient transport of ^{90}Y in SLM mode. Under these conditions transport of strontium is found to be negligible. Radiochemical purity of the product ^{90}Y is checked by following its decay as well as by extraction paper chromatography. The contamination of ^{90}Sr in ^{90}Y product is found to be < 0.001%. Based on the experimental results, a single stage SLM system for the generation of carrier-free ^{90}Y from ^{90}Sr source is described. The system is amenable for automation and scale up.

Keywords ^{90}Sr-^{90}Y generator; D2EHPA; PUREX-HLLW; solvent extraction; supported liquid membrance

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INTRODUCTION

Strontium-90 ($T_{1/2} = 28$ y) is one of the important fission products present abundantly in high level liquid waste (HLLW) generated during the reprocessing of spent nuclear fuel by PUREX process (1). ^{90}Sr separated from this waste is an ideal source for carrier-free ^{90}Y ($T_{1/2} = 64.4$ h, $E_{max} = 2.28$ MeV) which is used in nuclear medicine due to its good radionuclidic as well as stable binding characteristics with various chelating agents (2–5). Methods based on ion-exchange, extraction chromatography, and solvent extraction either alone or in combination have been reported in literature (6–14) for the separation of carrier-free ^{90}Y from ^{90}Sr for radiotherapeutic applications. The most important criteria in the separation is the radiochemical purity of ^{90}Y product especially from its parent which is a bone seeker (15).

Solvent extraction data are quite useful in selecting the solvent/carrier in membrane based separation techniques and extraction chromatography. The extraction data under identical conditions can be used to optimize the separation conditions. Earlier, similar studies were reported from our laboratory (13) using 2-ethylhexyl 2-ethylhexyl phosphonic acid (KSM-17), an acidic organophosphorous reagent and were used for the development of single stage SLM based generator system for the separation of carrier-free ^{90}Y from ^{90}Sr (10–12). Further, the solvent extraction behavior of strontium and yttrium was studied using octyl(phenyl)-N, N-diisobutyl carbamoylmethylphosphine oxide (CMPO) and based on the findings, a two-stage SLM generator system was developed for milking ^{90}Y in a most pure form useful for radiopharmaceutical applications (13). In view of improving the purity of carrier-free ^{90}Y and imparting operational simplicity, our laboratory is continuously engaged in the development of a superior ^{90}Sr–^{90}Y generator system.

Di-(2-ethylhexyl) phosphoric acid (D2EHPA), yet another reagent of acidic organophosphorous class which has already been studied for the separation of ^{90}Y from ^{90}Sr-^{90}Y mixture in solvent extraction mode (18). The present paper presents the usability of D2EHPA as an extractant for the separation of carrier-free ^{90}Y in SLM mode. Distribution ratios for Sr and Y from nitric acid medium in batch mode are reported using 20% D2EHPA as extractant. These data are used in the selective transport of ^{90}Y during the development of a single stage SLM based generator system using D2EPHA as carrier. Under optimized conditions, a single stage SLM based generator system is tested for milking of carrier-free ^{90}Y from ^{90}Sr. This when coupled with a second stage membrane cell

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using CMPO as carrier similar to that developed earlier (13) will make the system more operator friendly. The merits of the current system are also discussed in the paper.

EXPERIMENTAL

Materials

Di-(2-ehtylhexyl) phosphoric acid (GR grade) obtained from E. Merck is used. Diluent n-dodecane (~93% C-12) was procured from M/s. Transware Chemia Handelsgeselschaft Hamburg, Germany. All other reagents used are of Analytical Reagent Grade. Commercially available polytetrafluoroethylene (PTFE) membranes supplied by Sartorius AG, Germany are used after impregnating with D2EHPA. The average pore diameter and thickness of membranes are 0.45 and 160 μm respectively and their porosity is about 84%.

The radioactive tracer $^{85+89}Sr$ is procured from the Board of Radiation and Isotope Technology (BRIT), Department of Atomic Energy (India) and ^{90}Y is obtained from the single stage SLM generator system developed in-house earlier (11).

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Radio Assay

Beta activity is assayed using a β-proportional counter standardized against RaDE source and NaI(Tl) gamma scintillation counter standardized against ^{137}Cs source is used for $^{85+89}Sr$ gamma activity. In batch extraction experiments known aliquots (generally 50–100 μL) from each phase are taken for radioassay whereas 20 μL of the samples are drawn from the compartments at different intervals for β-activity assay.

Solvent Extraction Experiments in Batch Mode

In solvent extraction experiments, 2 mL of the aqueous phase containing one of the metal ions (either Y^{3+} or Sr^{2+}) spiked with their respective tracer at known concentration are contacted with equal volume of 20% D2EHPA in n-dodecane for about 10 minutes which was found to be sufficient to attain the equilibrium. The phases are separated and assayed for their activity. Extraction behavior is studied as a function of nitric acid of varying strength (0.01–6.0 M). Distribution ratios under identical conditions are evaluated and used to compute the separation factors.

Impregnation of D2EHPA in PTFE Membrane Support

Impregnation of D2EHPA in PTFE support is carried out by immersing the membranes in the desired carrier solution using n-dodecane as diluent for at least 12 h before use. The membrane is rinsed once with water and used as partition between the feed and receiving compartment in the membrane cell.

Transport Studies

Single-stage SLM measurements are carried out using a two-compartment permeation cell in which two aqueous phases, one source and other receiving phase, (5 mL each) are separated by a supported liquid membrane of area 1.13 cm^2. The solutions in both the compartments are mechanically stirred at room temperature to avoid concentration polarization conditions at membrane interfaces and in bulk solution. A schematic diagram of a single stage permeation cell is shown in Fig. 1.

Experiments are carried out under different experimental conditions. Transport parameters such as the concentration of the carrier in the membrane phase, concentration of nitric acid in the feed, as well as in the receiver phase etc. are studied. The concentration of the carrier is varied from 10 to 80% in n-dodecane. Undiluted D2EHPA (as supplied) is also used as carrier. Concentration of HNO_3 in the source is varied in the range of 1.0–2.0 M whereas that of the receiver phase is varied in the range of 3.0–4.0 M.

Macro Concentrations of Sr

The transport of strontium is also studied from feed solutions containing 5 mg/mL of strontium at 1.0 M HNO_3 spiked with $^{85+89}Sr$ as tracer. In these experiments 4.0 M HNO_3 is used as receiving phase. Concentration of strontium in the feed was knowingly kept at ~5 mg/L as this is the concentration of Sr that is likely to be present in the purified ^{90}Sr product solution from HLLW. In a separate experiment, transport of Sr is also studied for a prolonged period of ~24 h under similar conditions. The presence of higher concentration of Sr (~10 mg/mL) in the feed is also studied under similar conditions.

Mixture of ^{90}Y–^{90}Sr

In these experiments, radiochemically pure ^{90}Sr solution in nitric acid medium, separated and purified by multi-step techniques involving solvent extraction, ion-exchange, and precipitation from PUREX-HLLW was used as feed (19). After attaining equilibrium the acidity of the final test solution is adjusted to either 1.0 or 2.0 M HNO_3 depending upon the requirement and used as feed for milking ^{90}Y.

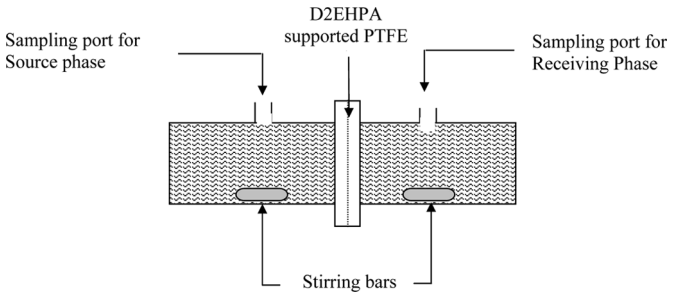

FIG. 1. Schematic diagram of single stage permeation cell.

Percent Transport and Flux

The feed and receiver phases are monitored for beta radioactivity to find the percentage transport at desired time intervals as below:

$$\text{Transport } (\%) = (A_t/A_o) \times 100$$

Where, A_t is the activity at time interval t in the receiver compartment and A_o the initial activity taken in the feed compartment. Corrections for ^{90}Y decay are incorporated wherever necessary while calculating the percent transport.

Membrane permeability is determined by monitoring the ^{90}Y beta activity in the receiving phase as a function of time. The yttrium flux, J_M, is computed as:

$$J_M = [Y^{3+}]_{\text{receiving}} {}^{*}V/(A^{*}t)$$

Where: $[Y^{3+}]_{\text{receiving}} = Y^{3+}$concentration in the receiving phase in mole dm^{-3} using specific activity of ^{90}Y.

V = volume of receiving phase in dm^3

A = effective area of the membrane in m^2

t = time elapsed in sec.

Purity of ^{90}Y Product

Suitable aliquot from the ^{90}Y product compartment is assayed for its β-activity which was followed as a function of time. Activity vs. time semi log graph is plotted to find its $T_{1/2}$. Extraction paper chromatographic technique is used to assay the purity of the ^{90}Y product (20). In this experiment the test solution is diluted appropriately with high accuracy to a concentration of 37 MBq/mL (1.0 mCi/mL). A known small aliquot (10 μL) of this test solution is applied on the paper where a spot is impregnated with KSM-17. The paper is developed in saline. The KSM-17 retains Y^{3+} tightly at the point of application and Sr^{2+} migrates with solvent front, resulting in neat sharp separation. The paper on drying, cutting, and counting gives the data for estimation of amounts of radioactivity in ^{90}Sr and ^{90}Y regions.

RESULTS AND DISCUSSION

Extraction data obtained using 20% D2EHPA in n-dodecane at varying strengths of nitric acid are given in Table 1. In general, the extraction of yttrium is much higher compared to that of strontium which is found to increase substantially with decrease in acidity. Practically negligible extraction of Sr^{2+} is observed from feed solutions at nitric acid concentration greater than 1.0 M ($D_{Sr} < 10^{-3}$).

Using the distribution ratios under identical conditions, separation factors ($D_{Y^{3+}}/D_{sr^{2+}}$) are calculated for Sr–Y couple and are listed in the same table. Results indicate higher separation factors in the acidity range of 0.5–2.0 M with a maximum at 1.0 M HNO_3. These results suggest that D2EHPA does not extract Sr^{2+} from acidic aqueous solutions higher than 1.0 M HNO_3. Extraction of Y^{3+} by 20% D2EHPA is significant ($D_Y \sim 70$) even at 1.0 M HNO_3 which decreases with increasing concentration of nitric acid. Thus, this condition is ideal for separating ^{90}Y from ^{90}Sr in the SLM mode.

TABLE 1
Extraction of Sr^{2+} and Y^{3+} from HNO_3 medium

$[HNO_3]$ in feed (M)	Distribution ratio (D)		Separation factor $(D_Y)/(D_{Sr})$
	^{90}Y	^{90}Sr	
0.01	1772.60	3.20	5.54×10^2
0.10	959.16	1.47	6.52×10^2
0.50	615.92	0.09	6.84×10^3
1.00	70.06	$<10^{-3}$	$>7.01 \times 10^4$
2.00	7.63	$<10^{-3}$	$>7.63 \times 10^3$
4.00	0.84	$<10^{-3}$	$>8.40 \times 10^2$
5.00	0.70	$<10^{-3}$	$>7.00 \times 10^2$
6.00	0.35	$<10^{-3}$	$>3.50 \times 10^2$

Experimental conditions:
Feed: ^{90}Y, β-activity = $\sim 1.3 \times 10^5$ Bq/mL, $^{85+89}Sr$, γ activity = $\sim 6.4 \times 10^3$ Bq/mL, Extractant: 20% D2EHPA in n-dodecane, Phase ratio: 1:1, Volume: 2 mL, Contact time:15 min.

Figure 2 gives the results of transport studies carried out from nitric acid medium using a yttrium tracer. Based on the results from solvent extraction, in all these cases, the concentration of nitric acid in the feed compartment is maintained at 1.0 M whereas 4.0 M HNO_3 is used as strippant. From the figure it can be seen that when the carrier concentration is 20%, the highest transport of ^{90}Y across the membrane is obtained. Lower carrier concentration at

TABLE 2
Transport of Sr through SLM containing 20% D2EHPA in n-dodecane as carrier

Transport conditions	Time (h)	Transport (%)
Feed: 1.0 M HNO_3 containing Sr at 5 mg/mL spiked with $^{85+89}Sr$, γ-activity = 8550 Bq/mL	6	ND
Receiving phase: 4.0 M HNO_3	24	0.01
Feed: 1.0 M HNO_3 containing Sr at 10 mg/mL spiked with $^{85+89}Sr$, γ-activity = 8850 Bq/mL,	6	ND
Receiving phase: 4.0 M HNO_3	24	0.02

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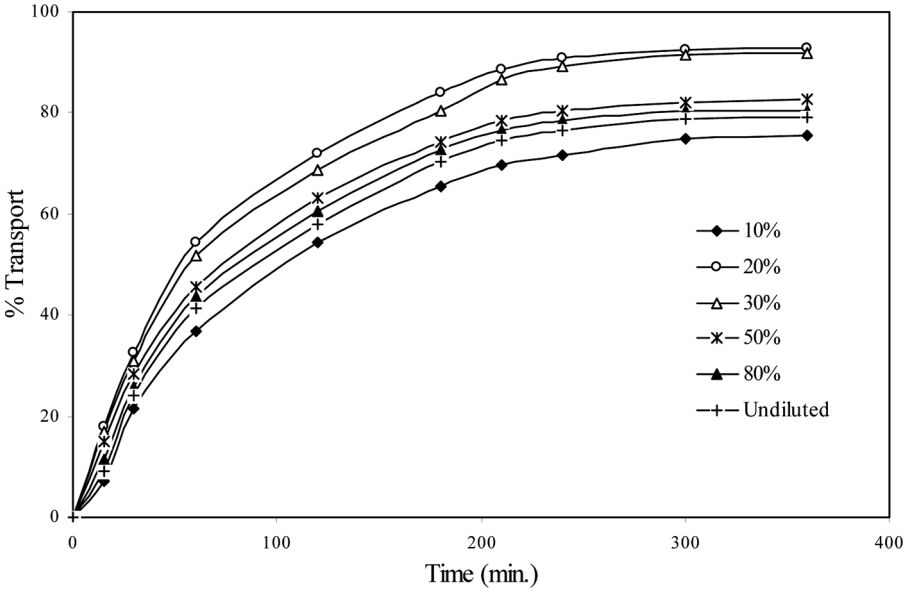

FIG. 2. Transport of ^{90}Y as a function of time using varying conc. of D2EHPA as carrier [Conc. of HNO_3 in Source phase: 1 M, Receiver phase: 4 M, Initial ^{90}Y β-activity in feed: $\sim 1.3 \times 10^5$ Bq/mL].

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10% and higher viscosities above 30% D2EHPA may be the reason for decrease in the transport of ^{90}Y at lower and higher ends. Hence further optimization studies are carried out using 20% carrier concentration. Transport data obtained under these concentrations of D2EHPA are used to calculate ^{90}Y flux using the formula described in the experimental section.

Figure 3 gives ^{90}Y flux as a function of time which indicates the highest flux of ^{90}Y within 30 min of transport in all the cases and decrease further. Maximum flux reaching to above 35×10^{-14} moles/$m^2 \cdot$ sec is observed for 20% D2EHPA in n-dodecane.

Figure 4 shows the effect of the concentration of nitric acid on the transport of ^{90}Y through SLM containing 20% D2EHPA in n-dodecane as carrier. As mentioned in the experimental section, the acidity of the source phase is varied in the range of 1.0–2.0 M whereas that of receiver phase 3.0–4.0 M HNO_3. From the figure it can be seen that the maximum transport of ^{90}Y is obtained when the acidity of the source phase is kept at 1.0 M and that of the receiver phase 4.0 M HNO_3.

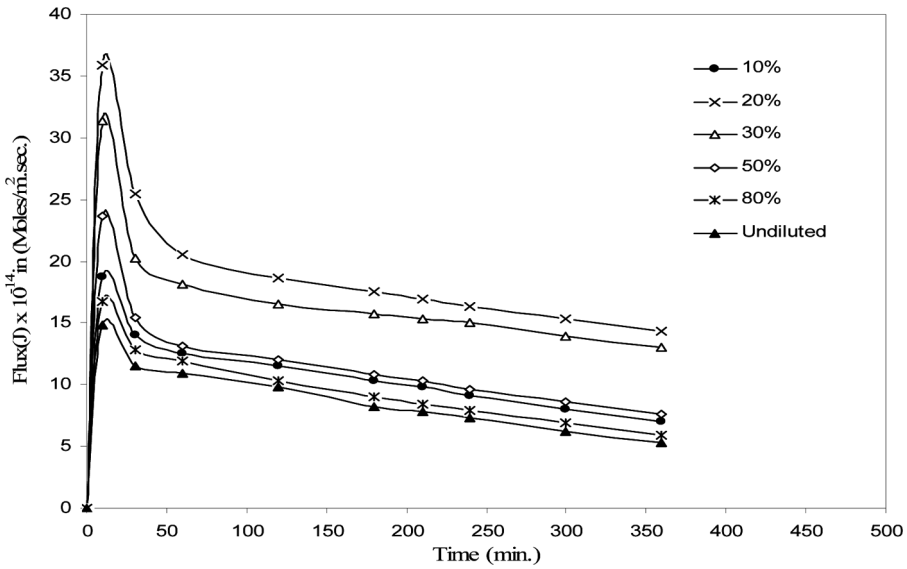

FIG. 3. Flux vs. time graph for ^{90}Y as a function of D2EHPA in n-dodecane as carrier.

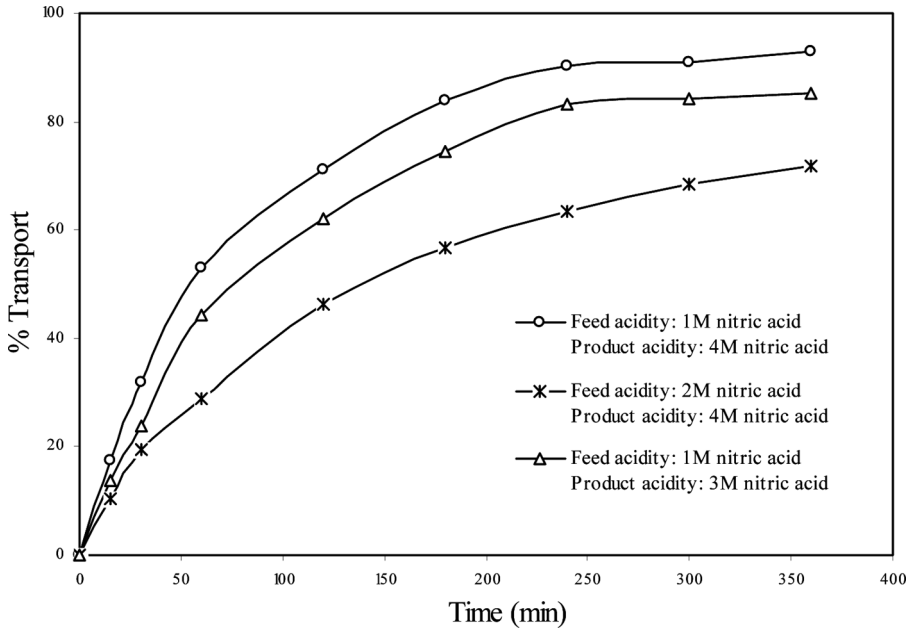

FIG. 4. Transport of ^{90}Y from nitric acid medium using 20% D2EHPA in n-dodecane as carrier [Initial ^{90}Y β-activity in feed: $\sim 1.3 \times 10^5$ Bq/mL].

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Transport of Sr

Since the radiochemical purity of ^{90}Y is very important in bio-medical applications, it is necessary to study the transport of strontium. Results of Sr transport at two different concentrations are given in Table 2. Practically no transport of strontium is observed even after 12 h from feed solution spiked with $^{85+89}Sr$ tracer containing 5 mg/mL Sr at 1.0 M HNO_3 using 4.0 M HNO_3 as receiver phase. About 0.01% of the strontium activity was detected in the receiver phase only after ~24 h. Under similar conditions, even in the presence of higher concentration of 10 mg/mL of strontium in the feed, the receiving compartment did not show any detectable strontium activity after 6 h. Based on these experiments it can be concluded that the generator system can be operated for 6 h under the above conditions to produce ^{90}Y product free from strontium activity.

Above transport parameters are used for separating the carrier-free ^{90}Y from a feed solution containing an equilibrium mixture of ^{90}Sr-^{90}Y. Multi-step separation techniques are employed for getting this radiochemically pure ^{90}Sr from PUREX-HLLW (19). The transport results are shown in Fig. 5. The transport data again confirm the

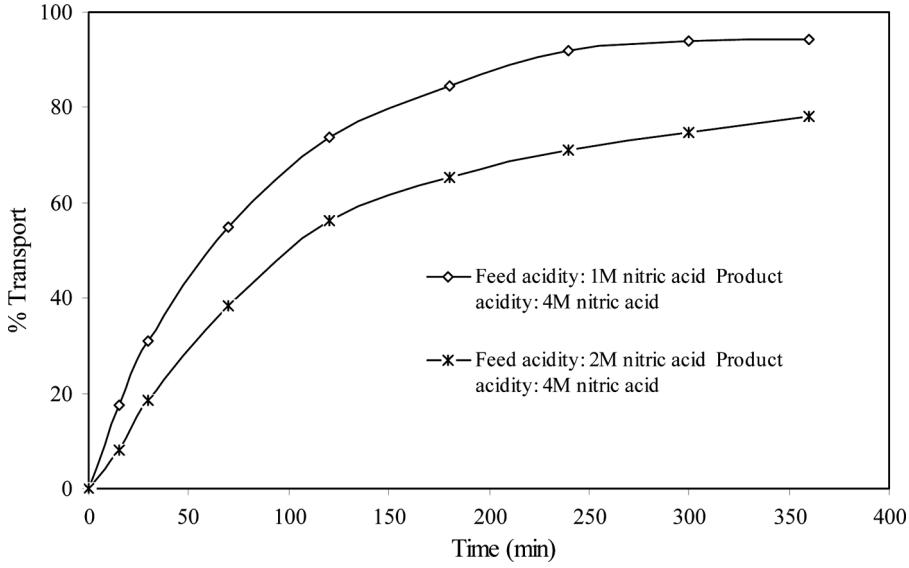

FIG. 5. Selective transport of ^{90}Y from activity mixture of ^{90}Sr–^{90}Y using 20% D2EHPA in n-dodecane as carrier.

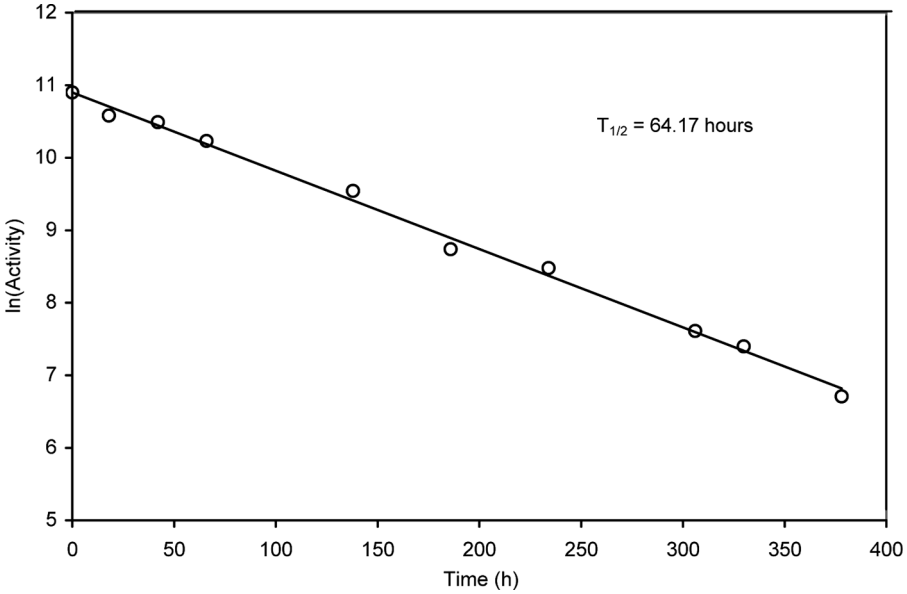

FIG. 6. Decay curve of carrier-free ^{90}Y from receiver compartment.

findings of the experimental results shown in Fig. 4. The transport of ^{90}Y is $>90\%$ in ~ 6 h.

The gross beta activity handled in the feed compartment is 444 MBq ($\sim$12 mCi) level. The system is used repeatedly to check the reproducibility. The results show the ^{90}Y separation yield of $94.2 \pm 3\%$ from a set of five different transport experiments.

The separated ^{90}Y product under the optimized transport conditions is checked for its purity by following its decay. The semi-log of activity vs. time is shown in Fig. 6. From the slope of the graph the $t_{1/2}$ is calculated and found to be 64.17 h. Radiochemical purity of ^{90}Y is also checked for ^{90}Sr contamination using extraction paper chromatography and found to be $<0.001\%$ at 37 GBq/L level of ^{90}Y. A typical extraction paper chromatographic activity bar chart for a ^{90}Y product at 185 MBq (5 mCi) level indicating the retention of all the spotted activity at the point of application is shown in Fig. 7. The activity in the strontium region is found to be below detection level indicating high purity of the product.

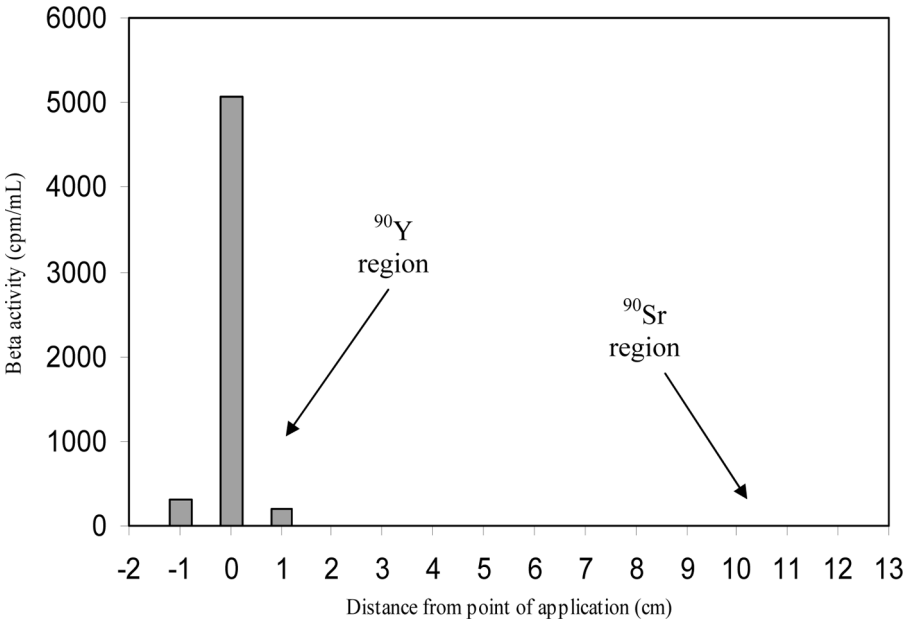

FIG. 7. Extraction paper chromatographic bar chart for ^{90}Y product.

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Based on these results, a single stage SLM system is being operated routinely at 185–370 MBq (5–10 mCi) level for the generation of carrier-free ^{90}Y from ^{90}Sr. The designed system has been tested with feed solution having about 100 mCi of each ^{90}Sr and ^{90}Y mixture. Results indicate >90% ^{90}Y can be milked with the same purity as that obtained using lower lavels. The standard automation aids used in pharmaceuticals can be easily integrated to the system.

Merits of D2EHPA Over KSM-17 as Carrier

As reported earlier from our laboratory (13) KSM-17 carrier is also capable of selectively transporting ^{90}Y from the mixture of ^{90}Sr–^{90}Y, but the system is very sensitive to variation in the acidity of the feed compartment which has to be controlled in a very narrow range of pH *viz.* 1–2. Any significant deviation in the pH can affect the ^{90}Y separation yield and purity. This can put additional burden on the operator and can expose him to radiation when higher quantities are handled. In the present system, where D2EHPA is used as carrier, the ideal acidity of 1.0 M in the feed compartment can be easily controlled and a small variation in acidity does not affect the ^{90}Y separation yield or purity significantly. As the acidity of the receiving compartment is 4.0 M coupling this system to a second stage SLM using CMPO carrier can be easily accomplished similar to that of the second stage of the two stage SLM based generator system reported earlier ( ). The second system will act as a guard to the first and will eliminate the remote chance of ^{90}Sr entering to the product compartment due to any unforeseen mechanical failure or other inadvertent action then by preserving the radiopharmaceutical quality of ^{90}Y product.

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CONCLUSIONS

From solvent extraction and transport data it is concluded that the extraction of ^{90}Y is favored at lower concentration of nitric acid which can be stripped at higher concentration. Selective transport of ^{90}Y (>90%) from ^{90}Sr is achieved in 6 h when the concentration of nitric acid in the feed is maintained at 1.0 M and that of receiver at 4.0 M. 20% D2EHPA in n-dodecane is found to be optimum carrier concentration for the efficient transport of ^{90}Y in SLM mode. A modified two-stage SLM based ^{90}Y generator system in which D2EHPA is used as carrier in the first stage and CMPO in the second stage can form an ideal generator that may be operated in radiophamaceutical centers.

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