See also: **II/Chromatography: Thin-Layer (Planar):** Densitometry and Image Analysis; Spray Reagents. **III/Flame Ionization Detection: Thin-Layer (Planar) Chromatography. Impregnation Techniques: Thin-Layer (Planar) Chromatography. Silver Ion:** Thin-Layer (Planar) Chromatography. **Sterols:** Supercritical Fluid Chromatography.

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STRONTIUM FROM NUCLEAR WASTES: ION EXCHANGE
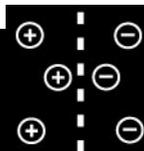
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Introduction

The development of new inorganic ion exchange materials for the selective removal of strontium and other radionuclides from nuclear waste has progressed rapidly in recent years. ^{90}Sr is an important component of many nuclear wastes and is a high yield fission product of ^{235}U. It is relatively short-lived with a half-life of 28.8 years and, along with ^{137}Cs, is the source of a large percentage of the initial radioactivity and heat generation of spent nuclear fuel. During the reprocessing of nuclear fuel, irradiated uranium fuel rods are dissolved in nitric acid and uranium and plutonium are separated from the fission products and other actinides by means of the Purex process. Tributylphosphate (TBP) dissolved in an organic phase, such as odourless kerosene, is contacted with the nitric acid solution, and plutonium and uranium nitrates are selectively complexed by the TBP and extracted into the organic phase. The majority of fission products, including ^{90}Sr, remain in the aqueous acidic phase, which can then be concentrated by means of evaporation and stored prior to permanent disposal. In addition to the acidic high level waste stream, numerous other streams are generated during reprocessing operations as a result of washing, decontamination and scrubbing operations. Details of some specific streams generated by the nuclear industry from which ^{90}Sr needs to be selectively removed from large excesses of inert ions will be given later in this article.

A convenient method of selectively removing contaminant species from higher concentrations of inert ions is by ion exchange. Organic ion exchange resins are used in many industries for the selective removal of ions from aqueous streams. These materials consist of a polymeric backbone (commonly polystyrene) to which has been attached functional groups such as carboxylic or sulfonic acids to produce cation exchangers, or tertiary or quartenary amines to produce anion exchange resins. However, the use of organic resins in the nuclear industry is limited for a number of reasons. These include:

- low resistance to damage by ionizing radiation, thus limiting operational life;
- low thermal stability;
- limited chemical stability;
- low selectivity in comparison to inorganic ion exchange materials;
- incompatibility with grout or cement, making the final disposal of spent resins a problem.

Inorganic ion exchangers offer a number of advantages over conventional organic resins including greater selectivity and both chemical and radiolytic stability. Additionally, they are compatible with current waste encapsulation techniques and are stable enough that they can be used as a final waste form for long-term storage. The major drawback to the use of inorganic ion exchangers is that they are typically synthesized as fine powders, which are unsuitable for use in column operations. However, there are now a number of techniques available to allow these powders to be produced as pellets or particles suitable for column operations, while still retaining fast ion exchange kinetics and the ion selectivity of the original material. A number of reviews of available materials and their ion exchange selectivities have been written, and as new materials and methods of manufacture are being developed, the use of inorganic materials both in the nuclear industry and elsewhere will undoubtedly expand. Some of the major classes of materials that are currently being used (or are under evaluation) for the selective removal of ^{90}Sr from nuclear wastes are described in the following sections.

Zeolites

Zeolites are hydrated aluminosilicates with open framework structures. These consist of building blocks of {SiO$_4$} and {AlO$_4$} tetrahedra which can be interlinked to give a wide range of different materials with regular tunnels and cavities. The presence of trivalent aluminium in the framework results in a net negative charge that is neutralized by the absorption of cations. Specific zeolites exhibit high selectivities for strontium and caesium over other alkali and alkaline earth cations. This has led to their use in the treatment of some nuclear waste streams. Details of zeolite synthesis, structures, applications and information on their ion exchange properties can be found in the literature and will not be detailed in this article.

Clinoptilolite is a common natural zeolite with the ideal formula Na$_6$Al$_6$Si$_{30}$O$_{72}$·24H$_2$O, though due to interactions with natural groundwaters, some of the Na$^+$ ions will have ben replaced by K$^+$, Mg^{2+} and Ca^{2+} ions. It is currently used on a large scale both in the UK and the USA for the treatment of nuclear

waste solutions such as cooling pond water. This is one of the largest waste streams in terms of the volume of liquid, and consists of water used to cool and shield irradiated uranium fuel rods prior to their disposal or reprocessing. For example, spent pressurized water reactor (PWR) fuel is generally stored under water for up to 5 years to allow short-lived radioisotopes, such as ^{131}I ($T_{1/2} = 8.06$ days) and ^{106}Ru ($T_{1/2} = 367$ days), to decay away, and thus make the fuel rods easier to handle. Storage is often accompanied by the release of tiny amounts of radioactivity, primarily ^{137}Cs and ^{90}Sr, from the fuel rods into the cooling water thus necessitating removal of the radioactivity before the water can be discharged to the environment. Modern fuel is typically clad in zircalloy or stainless steel and the release of radioisotopes is minimal. However, older fuel types such as the UK's Magnox fuel and fuel stored at the Hanford site in Washington State, USA, do release significant radioactivity.

Fuel storage pond waters are relatively pure and contain minimal dissolved cations that can compete with the ^{90}Sr and ^{137}Cs for the available ion exchange sites. Compositions of a fuel pond simulant from the Hanford site and the composition of an average pond water from the British Nuclear Fuels plc. (BNFL) Sellafield, UK site are given below in **Table 1**.

At the Sellafield plant, BNFL employs two 9.6 m^3 beds of clinoptilolite in the site ion exchange effluent plant (SIXEP) to decontaminate the pond water used for storage of Magnox fuel before controlled discharge to the sea. This clinoptilolite originates from the Mud Hills deposit in the Mojave Desert, California, and has been crushed and sieved to give a particle size of 0.4–0.8 mm in diameter. A schematic of the SIXEP plant is given in **Figure 1**.

Table 1 Composition of two fuel cooling ponds

Component	Hanford N-basin (ppm)	Sellafield (ppm)
Al	0.78	0.3
B	28.4	nd
Ba	3.1	0
Ca	33.4	1.7
Cs	6.47×10^{-5}	3217 Bq mL^{-1a}
K	2.5	3.6
Mg	0.70	0.3
Na	37.2	48.5
Sr	0.39	287 Bq mL^{-1a}
pH	8.2	11.4

^{a} The activities of ^{90}Sr and ^{137}Cs correspond to pondwater concentrations of 5.38×10^{-5} ppm and 8.69×10^{-4} ppm, respectively. There is unlikely to be significant nonactive caesium present; however, the amount of nonactive strontium is likely to be significantly greater than the ^{90}Sr concentration. nd, not determined.
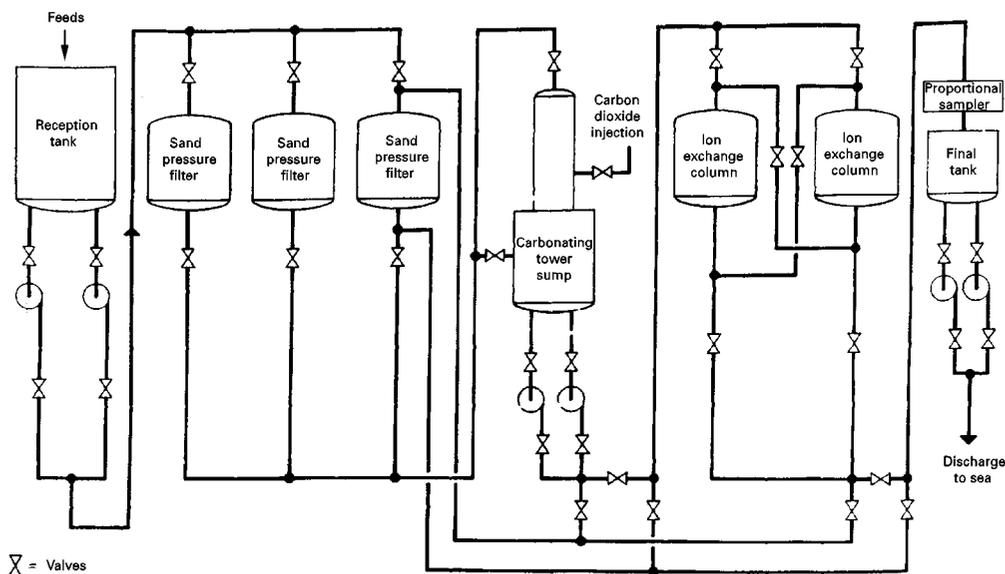
Figure 1 Schematic of BNFL's site ion exchange effluent plant (SIXEP). (Reproduced with the permission of BNFL.)

The feed is pumped through a sand filter to remove any particulates and is then treated with carbon dioxide to decrease the pH to approximately 7. The feed passes through two beds of clinoptilolite in series before being sampled and discharged to the Irish Sea. On average, approximately 3000 m^3 of effluent per day pass through the plant, which corresponds to a contact time of only 4.6 minutes per ion exchange bed. The clinoptilolite is very effective and typically removes 98.7% of the strontium and over 99.7% of the caesium from the stream prior to its discharge, and each bed lasts for approximately 6 months online.

Another area in which zeolites can be used for the removal of radioactive strontium is in groundwater remediation. Groundwaters have relatively low ionic strengths similar to pond waters, but differ in that the predominant inactive ions in solution are magnesium and calcium rather than sodium. There is also natural, nonradioactive strontium present, typically in the order of a few tenths of a part per million, which will also be removed along with the radioactive ^{90}Sr. Since Mg^{2+} and Ca^{2+} compete strongly with Sr^{2+} for the available ion exchange sites on the zeolites, the observed distribution coefficients (K_ds) for Sr tend to be considerably lower than in pond waters, and the higher concentration of strontium results in a shorter ion exchange bed life. However, the low cost of natural zeolites (less than US $0.5 per lb for clinoptilolite) means that they are economically viable.

Sodium Nonatitanate

Sodium nonatitanate (NaTi), $Na_4Ti_9O_{20} \cdot xH_2O$, displays a very high selectivity for strontium in basic media. The synthetic procedure is relatively simple and has been scaled up to allow the titanate to be produced on an industrial scale. A soluble source of titanium, such as titanium isopropoxide, is added to a 50% sodium hydroxide solution, resulting in the immediate formation of a white precipitate. The mixture is then heated in a hydrothermal bomb for approximately 21 h at a temperature of 200°C. The product is filtered, washed to remove excess NaOH, and dried to produce a white powder. The final material has a low crystallinity and consequently it has not been possible to determine the crystal structure. However, the titanate is believed to consist of layers of TiO_6 octahedra separated by exchangeable sodium ions and water molecules. At room temperature, the interlayer space is approximately 10 Å, but the distance can vary considerably depending upon the drying temperature, and hence the number of water molecules in the interlayer space. This material is now available from Allied Signal Inc. (Des Plaines, Illinois, USA) and similar products can also be obtained from Selion Inc. (Merden, Connecticut, USA) and Boulder Scientific Company (Mead, Colorado, USA).

Sodium nonatitanate exhibits a very high selectivity for strontium over alkali and other alkaline earth metals in basic media. In acidic media, the material has a high affinity for protons, so strontium selectivity is negligible. Consequently, this allows the nonatitanate to be stripped of absorbed strontium using dilute acid and reused. Sodium nonatitanate readily hydrolyses in water, exchanging protons for sodium ions; this results in a considerable increase in the solution pH. Consequently its use for treating groundwaters contaminated with ^{90}Sr is limited. However, its stability in highly

alkaline conditions makes it ideally suited for the treatment of alkaline nuclear wastes. This aspect will be discussed later.

Titanosilicates

Two separate classes of titanosilicate ion exchange materials have been developed for the selective extraction of strontium from nuclear wastes. Both classes are composed of a titanosilicate framework, but the crystal structures and the Ti : Si ratios are different and hence so are the ion exchange properties.

The first class of materials is exemplified by sodium titanosilicate (NaTS), with the ideal formula $Na_2Ti_2O_3SiO_4 \cdot 2H_2O$. This can be synthesized in a crystalline form which has allowed its structure to be determined using X-ray powder methods. The titanosilicate was found to have a tetragonal unit cell with $a = b = 7.8082(2)$ Å and $c = 11.9735(4)$ Å. Edge-sharing TiO_6 clusters reside in all eight corners of the unit cell and silicate tetrahedra are located midway between the clusters and link them together. This arrangement produces tunnels parallel to the c-axis where the exchangeable sodium ions and the water molecules reside. The remaining sodium ions are located in the framework, bonded by silicate oxygens and are thus not exchangeable. Due to steric repulsions and space limitations, some of the sodium ions in the tunnels are replaced by protons, leading to an actual formula of $Na_{1.64}H_{0.36}Ti_2O_3SiO_4 \cdot 1.84H_2O$. This exchanger is synthesized by hydrothermally heating a titanium silicate gel of appropriate stoichiometry in $6 \, mol \, L^{-1}$ NaOH at $170°C$ for 2 days.

This material has been shown to have a high selectivity for Cs^+ ions in both acid and alkaline pH and a high selectivity for strontium in alkaline media. Caesium ions exchanged onto the titanosilicate are strongly held in the tunnels, as shown in **Figure 2**, and are not readily leached off, thus the material is not regenerable. Strontium is readily removed by washing with dilute acid. A related material is currently marketed by UOP as a crystalline silicotitanate (CST) under the tradename IE-910 for the powder, and IE-911 for an engineered form suitable for use in column operations. The CST has shown excellent selectivity for ppm levels of caesium ions in the presence of $7 \, mol \, L^{-1}$ Na^+ ions and is currently being considered for use removing ^{137}Cs and ^{90}Sr from alkaline nuclear wastes in the USA.

The second class of titanosilicate materials has the crystal structure of the natural mineral pharmacosiderite. Pharmacosiderite has the ideal formula $KFe_4(AsO_4)_3(OH)_4$ and crystallizes in the cubic
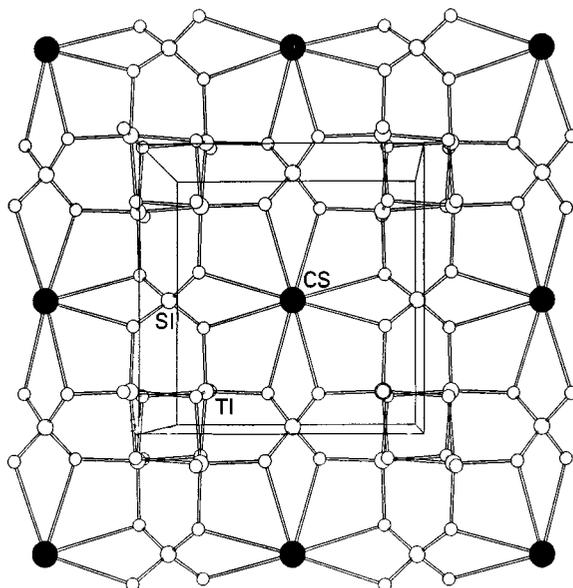

Figure 2 The structure of the caesium-exchanged titanosilicate showing Cs^+ ion in the centre of the tunnel.

system. Titanosilicates with the general formula $M_3H(AO)_4(BO_4)_3 \cdot 4{-}6H_2O$ (M = H, K, Na, etc.; A = Ti, Ge; B = Si, Ge) have been prepared using hydrothermal techniques. A homogeneous gel of appropriate stoichiometry was hydrothermally treated in an excess of either KOH or CsOH at $200°C$ for 1–3 days. Sodium and proton forms were then prepared by exhaustively ion exchanging the material with either HCl or NaCl. The best studied of these materials is the potassium pharmacosiderite, $K_3H(TiO)_4(SiO_4)_3 \cdot 4H_2O$ (KTS-Ph), in which $a = b = c = 7.7644(3)$ Å. Each unit cell consists of

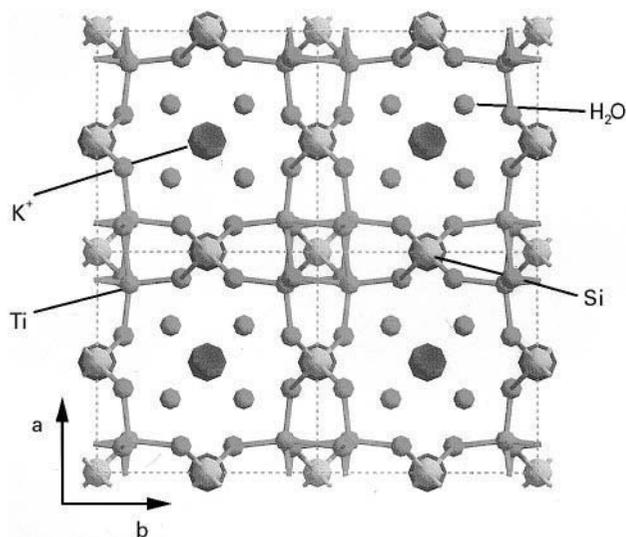**Figure 3** The structure of potassium pharmacosiderite $HK_3(TiO)_4(SiO_4)_3 \cdot 4H_2O$ with the K^+ ion located in the centre of the tunnels. $a = b = c = 7.7644(3)$ Å.

clusters of four titania octahedra linked to each other by silicate groups, as shown in **Figure 3**. This produces a series of intersecting tunnels parallel to the a, b and c axes with the exchangeable ions residing close to the face-centres of the unit cell.

It has proved possible to substitute Ge for both Si and Ti in the pharmacosiderite framework, thus allowing the size of the tunnels to be carefully tailored. These materials have shown selectivity towards both Cs^+ and Sr^{2+} but are not as effective as the sodium titanosilicate, NaTS, described previously. However, the caesium ion can be eluted from the exchanger, so unlike NaTS, the pharmacosiderites are regenerable making them more cost-effective.

Removal of Strontium from High Ionic Strength Wastes

In the USA, there are over 100 million gallons of radioactive mixed waste stored in 332 tanks distributed over a number of Department of Energy (DOE) sites. Much of this tank waste is highly alkaline and is typically over 7 mol L^{-1} in Na^+. The majority of this waste is found at the Hanford site in Washington State, where there is approximately 65 million gallons of high-level waste stored in 177 tanks. All of the Hanford tanks are highly alkaline and were generated as by-products of the production of ^{239}Pu for nuclear weapons manufacture. Initially, the waste was in a nitric acid matrix, but to minimize corrosion of the steel tanks, sodium hydroxide was added to neutralize the wastes and to precipitate much of the radioactivity. The composition of each tank is different, but in general, the wastes consist of three distinct phases. At the bottom of the tank is a metal hydroxide sludge, at the top is a salt cake, predominantly made up of nitrate salts, and between these layers is an alkaline supernate. ^{90}Sr is found in all three layers, but tends to predominate in the sludge layer. However, in cases where there are significant amounts of complexing agents, considerably greater ^{90}Sr activity is found in the supernate. The composition of two supernate simulants developed at Pacific Northwest National Laboratory (PNNL) to mimic the tank wastes are given in **Table 2**.

Both waste simulants represent dilution of actual tank wastes, which is envisaged to be necessary to allow ease of handling without excessive precipitation of salts occurring. NCAW represents tank 241-AZ-102, and 101-SY represents tank 241-SY-101, both diluted to approximately 5 mol L^{-1} in Na^+. This latter tank contains significant amounts of complexants such as ethylenediaminetetraacetic acid (EDTA) and citric acid. Zeolites are unsuitable for the treatment of these tank wastes because they lack sufficient selectiv-

ity for strontium in the presence of high sodium concentrations, and they are also unstable in highly alkaline conditions. However, the titanate and the titanosilicates are synthesized in strongly alkaline media and thus exihibit a high stability in these tank wastes. In addition, all of the exchangers exhibit good radiation stability, thermal stability and excellent resistance to extreme chemical environments.

The sodium nonatitanate and the titanosilicate ion exchange materials were evaluated in preliminary batch experiments using ^{89}Sr as a surrogate for ^{90}Sr. Here, 0.05 g of exchanger was contacted with 10 mL of waste simulant spiked with ^{89}Sr, giving a volume to mass ratio of 200:1, for 18 h using a rotary shaker. The mixtures were then filtered through a 0.2 μm filter and the activity of the aqueous phase determined using liquid scintillation counting. K_d values for strontium were then calculated according to eqn [1] below:

$$K_d = (A_i - A_f)/A_f \times v/m \qquad [1]$$

where A_i is the initial activity of solution (counts per minute mL^{-1}); A_f is the final activity of solution (counts per minute mL^{-1}); v is the volume of solution (mL); and m is the mass of exchanger (g).

Table 2 The composition of two Hanford tank waste simulants

Species	NCAW (mol L^{-1})	101-SY (mol L^{-1})
Al	0.43	0.42
Ca	0	4.20×10^{-3}
Cs	5.00×10^{-4}	4.19×10^{-5}
Fe	0	1.96×10^{-4}
K	0.12	0.034
Mo	0	4.20×10^{-4}
Na	4.99	5.1
Ni	0	2.50×10^{-4}
Rb	5.00×10^{-5}	4.20×10^{-6}
Sr	2.70×10^{-7}	4.10×10^{-6}
Zn	0	5.00×10^{-4}
Carbonate	0.23	0.038
Fluoride	0.09	0.092
Hydroxide	3.4	3.78
Hydroxide (free)	1.68	2.11
Nitrate	1.67	1.29
Nitrite	0.43	1.09
Sulfate	0.15	4.75×10^{-3}
Phosphate	0.025	0.02
Citric acid	0	5.00×10^{-3}
Na$_4$EDTA	0	5.00×10^{-3}
HEDTA	0	3.75×10^{-3}
Iminodiacetic acid	0	0.031
Na$_3$ nitrilotriacetate	0	2.50×10^{-4}
Sodium gluconate	0	0.013
pH	14.5	14.4

Na$_4$EDTA, ethylenediaminetetraacetic acid, tetra sodium salt. HEDTA, N-(2-hydroxyethyl)ethylenediaminetriacetic acid.

Table 3 The removal of strontium from Hanford tank waste simulants by inorganic ion exchange materials

Ion exchanger	NCAW, K_d (mL g^{-1})	% Sr removed	101-SY, K_d (mL g^{-1})	% Sr removed
Clinoptilolite	48	19.35	Not tested	–
NaTi	235 000	99.93	295	61.1
NaTS	270 000	99.93	231	54.7
KTS-Ph	20 200	99.55	31	13.2

The results obtained for the three ion exchangers are displayed in **Table 3**. For comparative purposes, the strontium K_d for the Mud Hills clinoptilolite was also included, although this number should be viewed with caution because clinoptilolite is not stable in highly alkaline media and will have undergone substantial decomposition.

It can be seen that all of the synthetic ion exchange materials exhibited a very high selectivity for strontium from NCAW with K_d values in the tens or hundreds of thousands. Clinoptilolite performed very poorly, with a K_d of only 48 mL g^{-1} compared with the best material, the sodium titanosilicate, which had a K_d of 270 000 mL g^{-1}. By contrast, the K_d values from the 101-SY simulant were very low for all of the materials, indicating that the presence of relatively high concentrations of EDTA, citric acid and other complexants has resulted in the strontium being strongly chelated and thus not readily extractable by ion exchange. However, recent studies have indicated that this problem can be overcome by the addition of Ca^{2+} or other ions to the waste in sufficient quantity to saturate all of the EDTA and other complexants present, and thus release the strontium into solution where it can be removed by strontium-selective ion exchangers. Alternatively, the complexants can be destroyed using an appropriate chemical oxidation technique and the strontium removed by ion exchange.

Column Experiments

Column experiments using ^{89}Sr-spiked NCAW were performed to further evaluate the efficiency of the sodium nonatitanate, the sodium titanosilicate and the potassium pharmacosiderite at removing Sr under dynamic conditions. This necessitated pelletizing the ion exchange materials using approximately 15% by weight of a hydrous titania binder. Hydrous titania also shows some affinity for strontium in alkaline media, but tests proved that the K_d values were insignificant in comparison to the ion exchange materials.

Approximately 1 mL of material was slurried into a column and NCAW, spiked with ^{89}Sr to give a total strontium concentration of 2.7×10^{-7} mol L^{-1}, was then passed through at a flow rate of approximately 20 bed volumes per hour (BV h^{-1}). The ^{89}Sr activity

in the solution exiting the column was then analysed using liquid scintillation counting. Percentage breakthrough was calculated according to eqn [2]:

$$\%\text{Breakthrough} = (A_f/A_i) \times 100 \qquad [2]$$

where A_f is the ^{89}Sr activity exiting the column and A_i is the ^{89}Sr activity entering the column.

The breakthrough curves obtained for the materials are given in **Figure 4**. Also included is the breakthrough curve for the commercially available IE-911 determined under the same operating conditions. The nature and percentage binder present in the IE-911 is unknown, but in simple batch equilibrium experiments Sr K_d values in excess of 25 000 mL g^{-1} were obtained for ^{89}Sr in NCAW. Figure 4 shows that breakthrough of ^{89}Sr from the IE-911 bed is almost instantaneous, indicating very poor kinetics of exchange. By contrast, all three of the other exchangers show < 5% ^{89}Sr breakthrough for over 1500 bed volumes. Breakthrough was first obtained for the potassium pharmacosiderite and was followed by the sodium titanate and had reached approximately 25% and 17% respectively after 3000 bed volumes had been passed. By contrast, the breakthrough for the titanosilicate was still only around 5% when the experiment was terminated after the passage of 3500 bed volumes. This indicates rapid kinetics for all of the materials except the IE-911 and also an appreciable capacity for
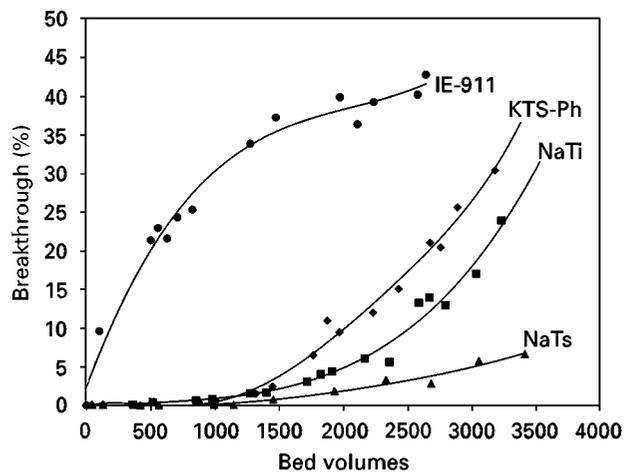

Figure 4 ^{89}Sr breakthrough curves for IE-911, NaTi, NaTS and KTS-Ph for NCAW at a flow rate of 20 BV h^{-1}.

strontium. Thus, all of the materials, particularly the sodium titanosilicate, have good potential for the decontamination of high-salt, alkaline nuclear wastes.

Conclusions

Inorganic ion exchangers have a wide number of applications within the nuclear industry and are preferred over conventional organic resins. Zeolites are ideal for the treatment of dilute wastes, provided that the pH is not too extreme, and their relatively low costs make their use highly economical. For more extreme wastes like those encountered in the Hanford storage tanks, new titanium-based materials have been developed that are able to withstand the high alkalinity and have sufficiently high selectivity to remove trace levels of strontium in the presence of molar quantities of other ions. Although these synthetic exchangers cost hundreds of US dollars per kilogram, their extreme selectivity and ability to be regenerated makes them viable options for the treatment of these extremely complex wastes.

Acknowledgements

I would particularly like to acknowledge Professor Abraham Clearfield, Dr. Elizabeth Bluhm and Gina Graziano at Texas A&M University, who worked with me on the titanate and titanosilicate ion exchange materials.

See also: **I/Ion Exchange. II/Ion Exchange:** Catalysis: Organic Ion Exchangers; Historical Development; Inorganic Ion Exchangers; Novel Layered Materials: Non-Phosphates; Novel Layered Materials: Phosphates; Organic Ion Exchangers; Surface Complexation Theory: Multispecies Ion Exchange Equilibria; Theory of Ion Exchange.

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SUB-CRITICAL WATER: EXTRACTION

See **III / SUPERCRITICAL FLUID EXTRACTION-SUPERCRITICAL FLUID CHROMATOGRAPHY**

SUGAR DERIVATIVES: CHROMATOGRAPHY
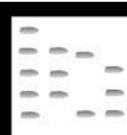
S. C. Churms, University of Cape Town, South Africa

Because sugar derivatives are generally present as complex mixtures, chromatographic techniques are crucial in their analysis. The spectrophotometric methods, and other methods mentioned in this article, serve primarily as chromatographic detection systems, and spectroscopic methods are frequently used in conjunction with chromatography.

Detection Reagents for Planar Chromatography and for Qualitative and Spot Tests

Detection reagents that are specific for particular derivatives, or can distinguish certain classes from