# Demonstration of SuperLig® 605 and SuperLig® 644 in a Regeneration Flow Sheet to Remove Radioactive Strontium and Cesium from Seawater

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## ABSTRACT

Remediation techniques are needed for efficient removal of radioactive strontium and cesium from seawater. Molecular Recognition Technology, using selective SuperLig® resins, demonstrates a clean chemistry approach to solving this problem. Molecular Recognition Technology offers high levels of separation of chemically pure metal contaminants, whose subsequent disposal is compatible with simple waste forms (grout and glass). SuperLig® resins can be regenerated with common reagents and used many times, limited by a dose of 1E+9 Rad, which assures low volumes of secondary solid waste. The metal selectivity of the SuperLig® resin ensures that the solid waste is of low residual activity. These properties of sustainability are illustrated in a project that demonstrated the ability of two SuperLig® resins to selectively remove Cs and Sr from a seawater mimic representative of the contaminated seawater in Fukushima, Dai'ichi, Japan harbor. Removal of radioactive Sr-90 and Cs-134/Cs-137 from seawater using conventional ion exchange or a complexing agent is complicated by the presence of large excesses of competing ions such as Na, K, Ca, and Mg, which can be co-extracted. These competing ions, if co-extracted, reduce the capacity of a single pass resin with the consequent increase in volume of spent resin or decrease in capacity of a regenerable resin. The successful demonstration reported in this study has shown the ability of two proprietary resins, SuperLig® 605 and SuperLig® 644, to remove Sr and Cs, respectively, with high selectivity from simulated solutions containing these metal ions together with major constituents of seawater at their normal concentrations. This effort was part of a demonstration project to investigate methods for removal of Sr-90 and Cs-134/Cs-137 from 160,000 cubic meters of seawater impounded at Fukushima, Dai'ichi, Japan harbor. SuperLig® resins can be regenerated by elution with small volumes of simple acid solutions. SuperLig® resins have a high tolerance to radiation, with their performance not being degraded by doses of 1.0E+9 Rad. Estimates, based on the results obtained, are that Cs and Sr levels in the volume of seawater in the Fukushima, Dai'ichi, Japan harbor could be reduced to desired discharge limits in 200 days with some 30

cubic meters of the two SuperLig® resins and that decontamination factors of 10 or more could be achieved. Results of the demonstration, together with advantages of the Molecular Recognition Technology process over conventional procedures for these separations are presented and discussed.

## INTRODUCTION

An enclosed area of about 160,000 cubic meters ($m^3$) within the Fukushima, Dai'ichi, Japan harbor (harbor) contains contaminated seawater, i.e., up to 200 becquerel per liter (Bq/L) Sr-90 and a much lower level of Cs-134/137. This seawater must be decontaminated to allow discharge. IBC Advanced Technologies, Inc. (IBC) participated in 2014-2015 in a competitive search, administered by the Mitsubishi Research Institute on behalf of the Japanese government, for technologies capable of cleaning up seawater contaminated with radioactive Cs and Sr to below discharge limits, without extracting appreciable quantities of the common constituents of seawater, i.e., Na, K, Mg, and Ca [1]. A further requirement was that the technology should be deployed without a conventional pump-and-treat step, i.e., one that would remove seawater for remote treatment and segregate the treated product. The process used needed to be capable of decontaminating the seawater to below discharge limits precluding the need for further treatment, and not generate significant volumes of solid secondary waste. The recovered contaminated product should be easily handled as a conventional waste form. To enable discharge to the harbor, the cleaned-up liquid must not be depleted in Na and other seawater ions. The procedure should avoid carry-over of inactive species into the waste stream. Using a Molecular Recognition Technology (MRT) SuperLig® procedure, IBC demonstrated achievement of these requirements by selectively separating and recovering Cs and Sr from a simulated seawater solution.

Requirements for removal of Cs and Sr from the seawater matrix mandate use of a technology that is simple, yet highly metal-selective, generates minimal waste, and is compatible with necessary waste disposal requirements. MRT is a process that meets these requirements. Highly selective separations of individual metals can be made with MRT using proprietary SuperLig® resins, even in the presence of complex matrices of other metals. MRT processes have been used for over two decades in the global market for highly selective metal separations, including those involving radioactive metals. High selectivity by the SuperLig® resins makes possible a separation process that generates little waste. In the process, simple and relatively harmless chemicals, such as dilute mineral acids are used. No solvents are used.

MRT systems have been used for green chemistry separations of a wide range of metal ions and anions including precious metals, such as platinum group metals, gold, and silver [2-5]; rare earth metals [6]; base metals, such as Cu, Ni, Co, and Sn [5]; toxic metals, such as Pb, Hg, As, and Cd [2,3,7]; radioactive metals, such as Cs, Sr, Ra, Pu, and U [8-10]; anions such as nitrate, Cl, sulfate, pertechnitate [9,11-13]; and metal anions, such as chloro complexes of Ru and Pt [2]. Relevant to the present study are the remarkable selectivities of SuperLig® 605 and SuperLig® 644 for $Sr^{2+}$ and $Cs^{+}$, respectively. These SuperLig® resins were developed for specific and selective removal of Sr and Cs from a high Na solution found in the high level waste (HLW) tanks at Hanford, Washington, where they were tested extensively and successfully against that application.

MRT SuperLig® resins have been of interest to workers in the nuclear industry for over two decades. This interest has involved both bulk separation of radionuclides and analytical determination of these metals. SuperLig® resins have been used at Hanford works in Washington for the separation of $Cs^{+}$, $Sr^{2+}$, and $TcO_4^{-}$ [11,12]. A number of workers have used MRT systems for analysis of radioactive metal ions [9,10]. In one case [8], the number of steps required to separate and prepare radioactive Sr for analysis was reduced from 50 for conventional methods to 6 for MRT and the time required was shortened from days to 20 minutes.

This study describes a series of trials carried out, using MRT, that demonstrated the ability of SuperLig® 605 and SuperLig® 644 to remove Sr and Cs, respectively, with sufficiently high selectivity to make an efficient clean separation from seawater simulant solutions containing concentrations of Na, K, Ca, and Mg equal to those found in natural seawater. The results demonstrate that MRT offers a rapid, cost-effective green chemistry procedure for removal and recovery of these metals from contaminated seawater.

## DESCRIPTION AND DISCUSSION

### Molecular Recognition Technology Process

The MRT process for selective metal separations and recovery has been described [2,3,14]. The SuperLig® system consists of a metal-selective ligand attached by a tether to a solid support, such as silica gel, as shown in Figure 1. The ligand in this case is 18-crown-6. The SuperLig® system in Figure 1 is selective for $K^{+}$, which fits nicely into the 18-crown-6 cavity (good match of cation ionic radius with cavity radius), while $Na^{+}$ and $Cs^{+}$ do not, their radii being too small and too large, respectively. The relative ability of the guest metal ions to fit in the host cavity is supported by log equilibrium constant (K) values valid in methanol at 25 °C for 18-crown-6 interactions with these metal ions [3] *i.e.*, $Na^{+}$ = 4.36, $K^{+}$ = 6.10, and $Cs^{+}$ = 0.99. Similar principles apply to the selective separations described here, where proprietary SuperLig® systems capable of highly selective interactions with $Cs^{+}$ and $Sr^{2+}$ are used for the separations of these ions. Ligand, cation, and system parameters which affect the magnitudes of host host–guest interactions are numerous and the reader is referred to review articles in which these parameters are discussed [15,16].

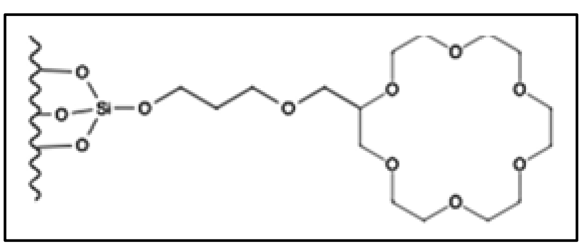

Fig. 1 Representative SuperLig® System Consisting of a Solid Support Particle, Silica Gel, to which a Metal-selective Ligand, 18-crown-6, is Attached by a Tether, which is Chemically Bound to both the Ligand and the Silica Gel.

SuperLig® resins consist of small (0.5 millimeter (mm)) particles packed into fixed-bed columns. These columns are present in skid-mounted modular form, and are fully automated for continuous operation. MRT processes operate on a system-cycle basis. A complete system cycle consists of the following sequence: (1) loading phase—target ion is loaded from feed solution onto an appropriate metal-selective SuperLig® product previously charged into the column(s, with remaining feed solution going to raffinate); (2) pre-elution wash phase—any remaining feed solution is washed from the column; (3) elution phase—target ion is eluted with a small amount of eluent, forming an eluate solution concentrated in the metal product; and (4) post-elution wash phase—any remaining eluent is washed from the column. The cycle, with the regenerated column, begins again with Step (1).

Pre-determined high selectivity for target metal species is the distinguishing feature of SuperLig® resins [2,3]. High metal selectivity makes possible high loading on the column of only the target metal resulting in direct recovery, upon elution, of that metal in high yield and high purity from matrices containing competing metals that otherwise would contaminate the separated metal leading to additional separation steps downstream. In the present case, the high selectivity of the SuperLig® resins for $Cs^+$ and $Sr^{2+}$, respectively, over metal ions in the simulated seawater matrix makes effective separations possible. The high affinity of the $Cs^+$ and $Sr^{2+}$ for the SuperLig® resins makes possible the separation of these cations at the milligram per liter (mg/L) level, even in the presence of the seawater matrix, which contains high concentrations of $Na^+$, $K^+$, $Mg^{2+}$, and $Ca^{2+}$. Elution of the selectively separated metal from the column in the MRT process provides a pure and concentrated target metal product without need for further purification.

## Regeneration Flow Sheet for Cesium and Strontium Separation from a Simulated Seawater Matrix

A flow sheet describing the separation of Cs and Sr from contaminated seawater is given in Figure 2. The flow of Cs and Sr is presented from filtration of the contaminated seawater through the separation processes by the SuperLig® resins to

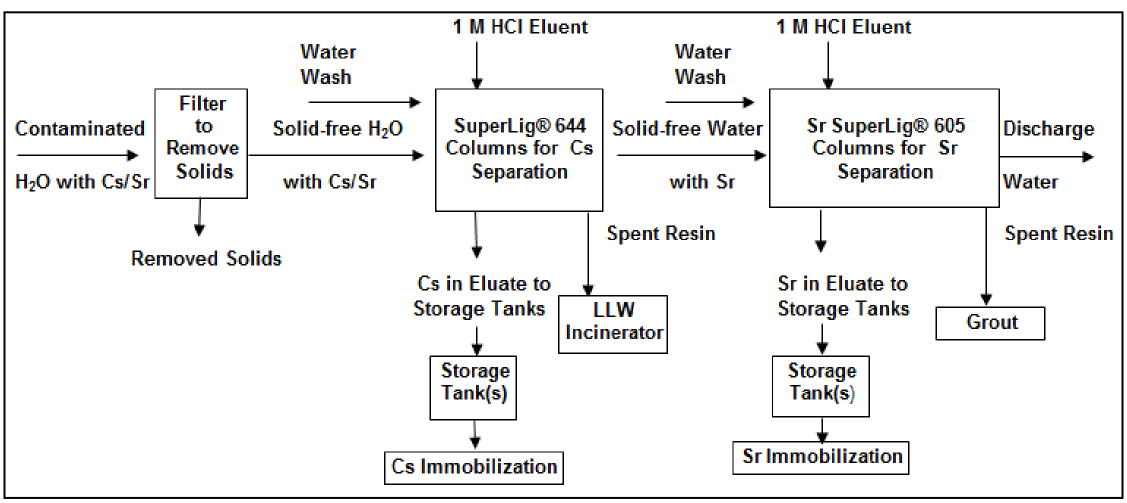


Fig. 2 Regeneration Flow Sheet for Cs and Sr Removal

the eventual disposition of these separated metals by immobilization.

## Removal of Cesium from Simulated Seawater Matrix and from the Entire Harbor

Results of a representative set of column tests with Cs input of 0.8 mg/L are presented in Table 1. These results show the effectiveness of SuperLig® 644 in removing Cs from a seawater mimic containing Cs, Na, K, Mg, and Ca. It is seen that decontamination factor (DF) values (DF = concentration in divided by concentration out) of 19-615 are achieved with SuperLig® 644. The performance of SuperLig® 644 for Cs removal in a single, 20 gram (g) column from a seawater mimic is excellent with greater than (>) 98 percent (%) of the Cs removed during the high DF (polishing) portions of the test prior to significant breakthrough and only 0.25-0.45% Na, 0.02-1.1% K, 2-3% Ca, or 1-2% Mg removed from the feed solution by the resin using a water wash. The amount of Na, less than (<) 0.07%; K, 0.018%; and Mg, 0.4-0.5% removed from the feed solution is even less when a dilute acid wash as well as water wash is used. Elution of the SuperLig®-bound Cs with a small amount of weak acid eluent (regeneration of the resin) produces a concentrated eluate in which Cs is concentrated by a large factor. These results demonstrate a high selectivity of the SuperLig® 644 resin for Cs over the other major ions in seawater.

TABLE 1. Removal of Cesium using a 20 g bed of SuperLig® 644 from a Solution of Seawater Mimic Containing Cs.

| Volume (L) | Cs Input (mg/L) | Cs Output (mg/L) | DF |
|---|---|---|---|
| 0.4 | 0.8 | 0.0013 | 615 |
| 0.8 | 0.8 | 0.0018 | 444 |
| 1.2 | 0.8 | 0.002 | 400 |
| 1.6 | 0.8 | 0.002 | 400 |
| 2.0 | 0.8 | 0.0021 | 381 |
| 2.4 | 0.8 | 0.0025 | 320 |
| 2.8 | 0.8 | 0.0033 | 242 |
| 3.2 | 0.8 | 0.0066 | 121 |
| 3.6 | 0.8 | 0.0177 | 45 |
| 4.0 | 0.8 | 0.0432 | 19 |

A knowledge of the results of the Cs single column removal tests, the estimated volume of the harbor to be treated, and the DF value required allows a calculation to be made of the time that would be required to remove Cs from the harbor. This calculation involves the following assumptions:

1. Harbor volume requiring treatment is 160,000 $m^3$.
2. DF value is 10.

3. A non-pump-and-treat system is assumed with seawater treated being immediately returned to the harbor leading to a gradual reduction of Cs in the entire harbor rather than a high polish of the Cs from individual segments of the harbor.

4. A 9,838 kilogram (kg) amount of SuperLig® 644 is used, being divided between the canisters holding the resin.

5. The mass of Cs present in 316 L of seawater per kg of SuperLig® 644 can be removed per cycle at the start of the processing. A loading volume of 133% of this volume is used to complete the full loading and breakthrough.

6. Wash volumes (4 L/kg resin) of deionized water and elution volumes (three segments of 4 L/kg resin) of 1 M HCl (two recycled segments and one fresh elution segment at the end) are used.

7. All flow rates are 0.2 L/kg/minute leading to a cycle time including load/wash/elution/post wash of 1,730 minutes.

Results of the calculations indicate that the fraction of Cs initially in the harbor could be reduced by 90% over 200 days (see Figure 3.) Over the same time period, the volume containing the Cs would be reduced from the harbor volume, 160,000 cubic meters ($m^3$), to 5,355 $m^3$ involving re-use of the resin. The Cs waste-form would be produced from the 5,355 $m^3$. The treatment time includes a four-hour time allocation to accommodate crane skip handling. The calculations show an efficient cleanup of the harbor to a DF value of 10; well above the DF value of 3 required.

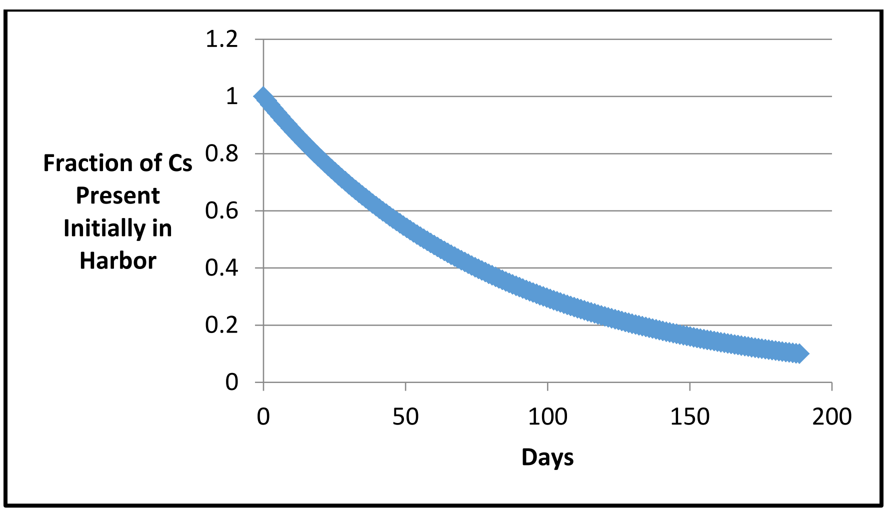


Fig. 3. Cesium Removal in Harbor as a Function of Time in Days Using SuperLig®644.

**Removal of Strontium from Simulated Seawater Matrix and from the Entire Bay**

Results of a representative set of column tests, presented in Table 2, show the effectiveness of SuperLig® 605 in removing Sr at an input level of 1.7 mg/L from a seawater mimic containing Sr at actual natural seawater levels (12.5 mg/L). It is seen that DF values of 11 to 142 can be achieved with SuperLig® 605.

TABLE 2. Removal of Strontium using a 20 g bed of SuperLig® 605 from a Solution of Seawater Mimic Containing Strontium at Natural Seawater Levels (12.5 mg/L).

| **Volume (L)** | **Sr Input (mg/L)** | **Sr Output (mg/L)** | **DF** |
|---|---|---|---|
| 0.4 | 1.7 | 0.012 | 142 |
| 0.8 | 1.7 | 0.0206 | 83 |
| 1.2 | 1.7 | 0.0258 | 66 |
| 1.6 | 1.7 | 0.0353 | 48 |
| 2.0 | 1.7 | 0.0386 | 44 |
| 2.4 | 1.7 | 0.0368 | 46 |
| 2.8 | 1.7 | 0.0587 | 29 |
| 3.2 | 1.7 | 0.0665 | 26 |
| 3.6 | 1.7 | 0.0816 | 21 |
| 4.0 | 1.7 | 0.0999 | 17 |
| 4.4 | 1.7 | 0.115 | 15 |
| 4.8 | 1.7 | 0.134 | 13 |
| 5.2 | 1.7 | 0.138 | 12 |
| 5.6 | 1.7 | 0.16 | 11 |
| 6 | 1.7 | 0.163 | 11 |

Calculations for Sr, similar to those made in the case of Cs, indicate that the fraction of Sr initially in the harbor could be reduced by 90% over 200 days (Figure 4). Over the same time period, the volume containing Sr would be reduced from the harbor volume, 160,000 $m^3$, to 5,770 $m^3$ involving re-use of the resin and achieving a DF value of >10. Results of the calculations, presented in Figure 4, show that 252 cycles of treatment with the SuperLig® 605 resin would be used to effect the treatment. The results show an efficient cleanup of the harbor to the 10 fold DF value required. The cleanup would lead to around 5,770 $m^3$ of eluate concentrate containing the Sr, from which the Sr waste-form would be produced.

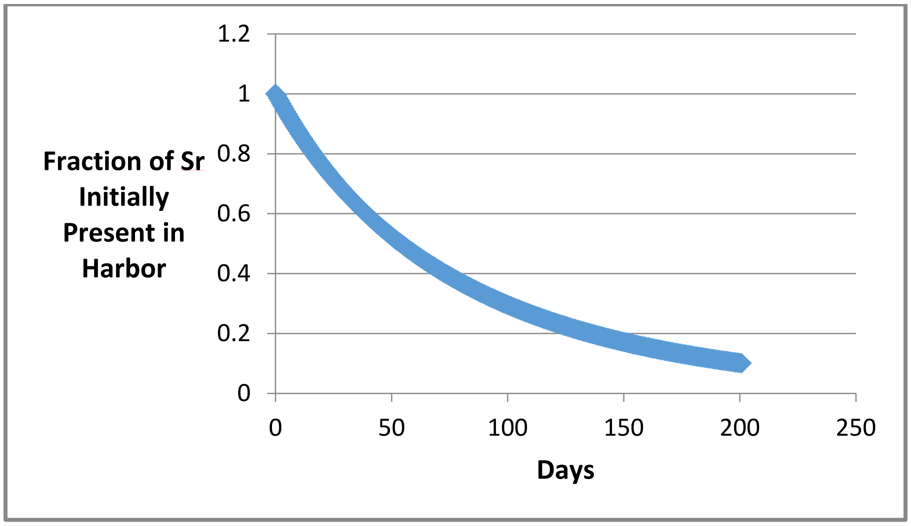


Fig. 4. Strontium Removal in Harbor as a Function of Time in Days Using SuperLig®605 for 200 Day Treatment.

The following assumptions, used in calculating the curve in Figure 4, are based on the results of the Sr single column removal tests, the estimated volume of the harbor to be treated, and the DF value required.

1. Harbor volume is 160,000 $m^3$.

2. DF value is 10.

3. A non-pump-and-treat system is proposed with seawater treated being immediately returned to the harbor leading to a gradual reduction of Sr in the entire harbor rather than a high polish of the Sr from individual segments of the harbor.

4. 6,604 kg of SuperLig® 605 is used for a 200-day treatment, being divided between the canisters holding the resin.

5. The mass of Sr present in 300 L of seawater per kg of SuperLig® 605 is treatable, initially, per cycle. This number gradually increases to 600 L of seawater per kg of SuperLig® 605 per cycle as the Sr concentration in the feed goes from 12 mg/L towards 1.2 mg/L. A loading volume of 133% of this volume is used to complete the full loading and breakthrough. The increase in loading volume from a feed level of 12 mg/L to 1.2 mg/L Sr corresponds to the increase in distribution coefficient ($K_d$) [$K_d$ = {[(initial concentration Sr) – (final concentration Sr)] / (final concentration Sr)} /(test solution volume, ml) / (resin mass, g)] and volume treated with decreasing Sr input level, as seen in the results of both $K_d$ and single column tests, where the $K_d$ and volume treated increase as the feed concentration decreases until a virtually constant $K_d$ level and treatable volume per cycle occurs. This effect is commonly seen for selective resins. This effect does not apply to Cs which is already at a low enough feed level to be at the essentially constant $K_d$ level and treatable volume per cycle.

## RESULTS

Results of the work carried out under this study are summarized in Table 3.

TABLE 3. Summary of Results from this Study

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|---|
| 1. MRT has significant time, labor, and process advantages over conventional methods in its green chemistry approach to highly selective metal separations. |
| 2. SuperLig® 644 and SuperLig® 605 resins meet and exceed the target levels for removing the nuclides Cs and Sr from seawater. |
| 3. IBC has the manufacturing capability to supply SuperLig® 644 and SuperLig® 605 resins in sufficient quantities for the harbor clean-up project. |
| 4. The regeneration flow sheet in MRT processes ensures that a low volume of secondary solid waste in the form of spent resin is generated; in the case of harbor decontamination, 15 $m^3$ of SuperLig® 605 ((based on 200 days processing), and 22.36 $m^3$ of SuperLig® 644 would be produced. |
| 5. The spent SuperLig® resin is amenable to treatment by grouting or incineration and ash grouting, which will result in a further volume reduction in the case of spent SuperLig® 644. |
| 6. Use of resins capable of regeneration reduces spectacularly the amount of secondary solid waste formed compared to single pass resins and, hence, storage/disposal/processing costs. |
| 7. Resin costs for the MRT system are low compared to those for once through processes by virtue of SuperLig® resin regeneration. |
| 8. The regeneration flow sheet, over the period of the harbor clean-up, will generate eluate volumes of 5,355 $m^3$ and 5,770 $m^3$ containing Cs-134/137 and Sr-90, respectively, using a DF value of 10 and somewhat conservative estimates of resin capacity retention. |
| 9. The secondary waste produced is free from other radioactive contaminants and is low in seawater ions (Na, Ca, Mg and K). |
| 10. The secondary waste produced is amenable to a range of treatment options; if grouted directly, this waste will generate 16,000 $m^3$ of grouted product. |
| 11. SuperLig® resin extraction (Skips), if required, can be protected with a filtration step for the harbor input ranging from a simple pre-filter to an electro coagulation unit. |
| 12. The process is configured around a submerged approach which conforms to the need for a non-pump-and-treat method. |
| 13. The process can also be operated as a pump-and-return process which would not require reducing the water level in the impounded area. |
| 14. The skip process, to be employed here, has been widely used in the nuclear industry, but, in particular, at Sellafield, UK for pond water clean-up. |
| 15. The MRT process and the low level of radioactivity in the seawater ensure that the dose to operators is minimized. |
| 16. High radiation resistance of the SuperLig® resin coupled with its high selectivity, ensures that the small volume of solid waste contains low radioactivity. |
| 17. High decontamination factors have been demonstrated that meet any discharge criteria for the target ions. |
| 18. The regeneration flow sheet ensures that target metals are selectively separated from a complex and difficult matrix; a low volume of secondary waste, |

| | |
|---|---|
| primarily spent SuperLig® resin, is produced in harbor clean-up; and a concentrated eluate containing Sr or Cs is produced that is amenable to a range of treatment options, including grouting or incineration. |
| 19. Use of the SuperLig® resins is underpinned by a body of literature and operating experience, including wide application in the nuclear liquid waste clean-up field, and SuperLig® technology responds to the basic principles of nuclear plant design, including significant limitation of secondary wastes. |

## Stability to Radiation Damage

The SuperLig® resins have been demonstrated (Table 3, 16) to have a high tolerance to radiation, and their performance is not degraded by doses of 1.0 E + 9 Rad (R) [17].

### Green Chemistry Aspects of MRT Applied to the Nuclear Industry

Green chemistry can be defined [18] as “the design of chemical products and processes to reduce or eliminate use and generation of hazardous substances.” An axiom in green chemistry applications is that it is better to prevent waste than to clean up waste after it is formed. Generation of waste by the nuclear industry has been extensive and on-going since the World War II period. Today, cleaning-up this waste is a major global challenge. Results presented in Table 3 for the application of MRT to major clean-up of Cs and Sr from the harbor demonstrate that these metals can be selectively and rapidly removed from contaminated seawater by this green chemistry method with minimal waste generation.

The amount of waste generated during the projected harbor clean-up is very low due to (1) the specificity of the SuperLig® resins for Sr and Cs (Table 3: 9), and (2) the ability to regenerate the SuperLig® resin (Table 3: 6). Specificity for Sr and Cs means that these metals can be concentrated selectively from the contaminated seawater reducing the volume to be disposed of in each case from 160,000 $m^3$ to about 5,500 $m^3$ (Table 3: 8). Regeneration of the SuperLig® resin reduces significantly the amount of spent resin, which would need to be treated (Table 3: 5, 6, 7). These green chemistry characteristics of MRT make it a desirable process for the clean-up of radioactive contamination in the harbor (Table 3: 18).

## CONCLUSIONS

The use of SuperLig® 605 and SuperLig® 644, in a regeneration flow sheet, has been demonstrated as an efficient technique to remove radioactive strontium and cesium from seawater. A large portion of the waste generated in the nuclear industry is related to separation processes. MRT is a mature separation process with important green chemistry features that make it effective in promoting clean separations in the nuclear industry [3,8-14]. First, high selectivity results in clean, efficient metal separations with reduced labor, space, and time requirements. Second, recovery of the separated metal in high concentration allows it to be treated by a reduced amount of grouting or other material for disposal. Third, SuperLig® resins can be renewed for re-use, which reduces significantly the amount of secondary solid waste generated. Fourth, MRT processes are simple in design and operation reducing capex and opex expenses significantly [6]. Fifth, SuperLig® resins have been tested and found to be effective for green chemistry separations of a wide range of radioactive and non-radioactive metal ions of interest to the nuclear industry.

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