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Chemical Engineering Evaluation / Feasibility Study of an On-Line Lithium Removal Process

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Chemical Engineering Evaluation / Feasibility Study of an On-Line Lithium Removal Process

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PRODUCT DESCRIPTION

Pressurized water reactors (PWRs) use enriched lithium-7 to maintain the pH in the reactor coolant at levels that mitigate cracking and reduce corrosion product transport. Natural lithium cannot be used because lithium-6 undergoes a neutron reaction that generates tritium at unacceptable levels for plant operation. The industry is concerned that the enriched lithium-7 supply chain may be vulnerable, and alternate alkali metals (potassium) are not readily qualified for use. From a positive perspective, lithium-7 is generated via neutron reaction with boron-10, which is used as a neutron poison for reactivity control. This document explores the possibility of lithium-7 recovery from the operating fleet and reviews technologies that could perform that recovery while the plant is on-line and off-line.

Objectives

To create a mass balance of lithium-7 in the PWR reactor coolant system (RCS) over a typical operating cycle.

To review possible solutions for recovering lithium.

To develop experiments and discuss the results in order to determine the most efficient means of recovering reusable lithium.

Approach

The project team developed a mass balance of lithium production and removal for a typical PWR operating cycle. Options for onsite and centralized lithium recovery from reactor coolant and spent resins were examined. The team developed chemistry regimes to be tested in the laboratory in order to evaluate the use of electrodeionization and ion exchange regeneration methods for lithium recovery.

Results

The lithium mass balance during a typical 18-month cycle was calculated applying all known input, output, and generation terms. These calculations demonstrated that it is feasible in many cases to recover sufficient lithium for the next cycle using sample line flow, and when not possible, the lithium may be recovered from cation exchange resins. Possible options for recovering lithium were reviewed, and wet-chemistry experiments were performed to determine the most effective means of recovering lithium from the RCS. The results of the experiments showed that while it would be expensive and challenging to implement, the U.S. PWR industry could recover sufficient lithium to maintain operations if the lithium supply were interrupted.

Applications, Values, and Use

The project demonstrated that lithium-7 recovery is feasible, reassuring the industry that operations may be sustained in event of a supply interruption. This report provides a strategy to determine whether on-line or off-line lithium recovery options are needed based on plant configuration and recommends future work to optimize chemistry requirements. This work supports EPRI's mission of ensuring safe and reliable nuclear power operations.

Keywords

Lithium recovery
Electrodeionization
Mass balance
Cation resin
On-line lithium removal

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INTRODUCTION

Background

Many pressurized light water reactors (PWR) use a chemical shim or neutron poison (boron in the form of boric acid) to control reactor power. As a PWR operates over time and fuel is burned, the boron concentration in the reactor coolant system (RCS) must be reduced proportionally to maintain constant reactor power. The rate of boron removal varies among PWRs but generally falls between 3 and 5 ppm/day depending upon the size of the reactor. In general and not considering other burnable poisons, the required boron removal rate from the reactor coolant system to maintain power is linear with time.

Lithium hydroxide enriched in ^{7}Li is initially added as a pH buffer in order to control RCS pH within a required band at the beginning of core life (BOL). Lithium enriched in ^{7}Li is required because ^{6}Li undergoes a neutron reaction to generate tritium at concentrations unacceptable for nuclear power plant operations,

$$^{6}\text{Li}(n,\alpha)^{3}\text{H}$$

^{7}Li must also be removed as boric acid is removed from the RCS for pH to remain within the control band selected by the plant chemistry program. ^{7}Li is also produced in the RCS by way of the following nuclear reaction

$$^{10}\text{B}\ (n,\ \alpha)\ ^{7}\text{Li}$$

^{10}B is approximately 20% naturally abundant, and the ^{7}Li produced from the above reaction is linearly proportional to the boron concentration. Therefore as boron concentration in the RCS is reduced, the ^{7}Li production rate decreases, and less excess lithium must be removed over time to maintain RCS pH within the required band.

^{7}Li is procured from facilities outside the United States, and the supply chain may be susceptible to disruption. This report seeks to develop a process that recovers ^{7}Li both used and generated in the reactor.

Traditionally, boron removal from the RCS is performed using a deborating demineralizer or by adding dilution water. As boron concentration in the RCS goes down during an operating cycle, larger volumes of dilution water are required to reduce the RCS boron concentration by the required amount to maintain 100% power (e.g., 3-5 ppm/day).

During an operating cycle, any required lithium removal beyond that which occurs as a result of boron dilution is performed by periodically aligning a hydrogen form cation bed into the purification stream (downstream or in parallel with the LiOH mixed bed normally in service for RCS purification). The frequency of the delithiation evolutions can vary from twice per day to once per three days for a duration of a few minutes to 20 – 30 minutes. The ^{7}Li trapped on this cation bed is currently not recovered for reuse. [1]

This report presents two options for recovery of ^{7}Li. The first option involves an on-line methodology that could be applied within individual plants. The second option involves off-site recovery of ^{7}Li from exhausted cation resin used for lithium control during operation.

The results indicate that a PWR can generate more ^{7}Li in a single operating cycle than is required for a subsequent cycle of operation for that unit. Laboratory testing demonstrated that sufficient ^{7}Li can be recovered to support a future cycle of operation using either an on-line separation from existing sample lines or via off-line regeneration of cation resin ion exchange beds used for delithiation.

Opportunities for additional study include the following:

- Examine the effect of various types of mid-cycle outages (e.g. mode 3, mode 5) and associated water management impacts on model sustainability.
- The use of certain types of burnable poisons requires a boron and lithium program that differs somewhat from the linear program considered in this evaluation. Expand the model to include these burnable poison scenarios.
- Examine lithium recovery from mixed bed resin in addition to cation resin and the effect of this additional source of ^{7}Li on sustainability.
- Determine the cost of implementation for both on-line separation in individual plants and a centralized facility to recover ^{7}Li from resin.
- Develop a national model to determine how many plants would have to participate in this program in order to generate enough ^{7}Li to supply the entire US PWR fleet.

References

1. EPRI. “Feasibility Study of an On-line Lithium Removal Process.” 3 July 2014.

2

LITHIUM MASS BALANCE ESTIMATES AND REMOVAL RATES

2.1 Industry Li-7 Consumption

Significant masses of ^{7}Li are required for startup of PWRs. The industry may use more lithium than calculated because of startup and forced outage requirements. This section reviews a survey compiled by the EPRI Chemistry Technical Strategy Group to estimate the consumed ^{7}Li.

2.1.1 Industry Survey

EPRI developed an industry survey related to lithium consumption during plant operations and requested support from the EPRI PWR Chemistry Technical Strategy Group (TSG) members in September 2013 [1]. The TSG survey is summarized in Table 2-1. Eleven utilities representing 15 US units plus Angra and EDF plants responded to the survey. The survey provided the following results for lithium-7 hydroxide monohydrate (the ^{7}Li form that is typically purchased):

- The average amount used per operating cycle based upon the responses received was 21.6 kg.
- Plants use an additional 11.1 kg of lithium-7 hydroxide monohydrate during start-up from a refueling. Most plants use pre-lithiated resin during start-up. The lithium used to pre-lithiate the resin is an additional use of lithium that needs to be accounted for in the estimate.
- The highest amount reported was from a plant that does not use pre-lithiated resin. This plant reported using 49.4 kg of lithium-7 hydroxide monohydrate per cycle.
- Two plants reported very high levels of lithium-7 hydroxide monohydrate usage: 49.4 kg and 44.4 kg. If these two are removed, the average from the survey becomes 17.4 kg per cycle. The reasons these two plants are high is likely due to forced shutdowns. One of these plants experienced 5 mid-cycle shutdowns during their previous cycle. Each mid-cycle start-up required another 2-6 kg of lithium-7 hydroxide mononhydrate, depending on when in the fuel cycle the mid-cycle shutdown occurred.

Table 2-1
Survey questions to the EPRI Chemistry Technical Strategy Group

Survey Question	Averaged Results (kg)
What is the typical lithium-7 hydroxide monohydrate consumption per operating cycle?	21.6
What is the typical lithium-7 hydroxide monohydrate consumption during start-up from refueling?	11.1
What is your current inventory?	35.6

2.1.2 Industry Annual Li-7 Consumption

An estimate of the lithium hydroxide monohydrate required to start up a Westinghouse 4-loop PWR reactor with 7 ppm of ^{7}Li (a conservative estimate) is below:

$$(92{,}500\ gal\ H_2O)\left(\frac{3.785L}{gal}\right)\left(1\frac{kg}{L}\right)\left(7e-6\ g\frac{kg\ Li}{kg\ H_2O}\right)\left(\frac{42.03kg\ LiOH\cdot H_2O}{7\ kg\ Li}\right)$$
$$= 14.7\ kg\ LiOH\cdot H_2O$$

The mass of lithium needed to saturate a lithium ion exchange bed in a PWR is estimated by first assuming a 30 cubic foot mixed bed equivalently loaded, which yields 12.2 cubic feet of cation resin.

$$(30\ ft^3\ resin)\left(\frac{1.1\frac{meq}{g}resin}{1.1\frac{meq}{g}+1.6\frac{meq}{g}}\right)=12.2\ ft^3\ cation\ resin$$

$$12.2\ ft^3\left(5.8\ \frac{lb\ LiOH\cdot H_2O}{ft^3}\right)\left(\frac{0.453kg}{lb}\right)=32.1\ kg\ LiOH\cdot H_2O$$

Assuming the overall lithium mass is the amount used for resin lithiation, the calculated coolant mass, and the second value in Table 2-1, the average lithium mass is 21.6 + 11.1 + 32.1 = 64.8 kg lithium hydroxide monohydrate. Assuming 18 month cycles and 65 operating reactors, the annual consumption of Li-7 is approximately 2,808 kg lithium hydroxide monohydrate, or about 468 kg ^{7}Li annually.

2.2 Calculated Lithium Mass Balance Estimates

The first step for determining the feasibility of recovering lithium from operating PWRs is to examine the ^{7}Li use and production rates. This is accomplished by developing a detailed lithium mass balance for a single PWR unit. The ^{7}Li mass balance starts with understanding the lithium added, generated, and removed for a typical operating cycle and typical plant. The overall cycle lithium mass balance is challenging because there are variations in the boron curve, which changes the generation rate from the conversion of ^{10}B to ^{7}Li, and there are lithium losses that vary with plant water movement operations and leak rates. These challenges are addressed in the various assumptions used to develop the model.

2.2.1 Model Inputs and Assumptions

This section explains the two corresponding spreadsheets developed to estimate the mass balance of lithium in the RCS. [2]

For the purpose of this theoretical spreadsheet, certain assumptions were made. These are bulleted below:

- RCS temperature is a function of reactor power and is 586 ºF (307 ºC) at 100% power
- Volume of the RCS is 87,850 gallons (332,548 L) at 586 °F (307 °C).
- ^{10}B fraction is a constant 19.9% and there is no recycling of boron.
- RCS leak rate is a constant 0.2 L/min (or 0.05 gpm) at 586 °F (307 °C).
- RCS sample is 0.1 L/min at 77 °F (25 °C) and is run for four hours a day and is not recycled.
- Cation bed effluent sample is 0.1 L/min at 77 °F (25 °C) and is run for four hours a day once a week and is not recycled.
- Cation bed is 99% efficient. Refer to Section 3.2.1 for the estimate calculation.
- The Boron Thermal Regeneration System (BTRS[1]) column is set at 0 or 1, with 1 meaning that a deborating bed is used. Beginning of cycle deboration is accomplished by feed and bleed and end of cycle deboration is accomplished using anion resin. For this model, BTRS is turned on when boron reaches 400 ppm.
- A pH program of 7.4 is used for the entire cycle to conservatively model (e.g. maximize) lithium usage.
- Mixed bed resin volume is 30 cubic feet (0.85 m^3).
- Cation/anion resin ratios are equivalently mixed, therefore only one-third volume of mixed bed resin is cation resin (10 cubic feet (0.28 m^3) in this case).
- Thermal neutron flux is assumed to be 9.3E11 n/cm^2 and the fast neutron flux is assumed to be 1E10 n/cm^2.

2.2.2 Model Derivation

A lithium mass balance is applied to each unit operation in the spreadsheet:

$$\boldsymbol{Accumulation = Li\ In - Li\ Out + Li\ Generation} \qquad \text{Eq. 2-1}$$

There are three sources of lithium into the system.

Injected lithium—this mass is recorded by the plant and manually input into the spreadsheet.

Lithium generation from the reaction of boron and neutrons–A completely rigorous treatment of lithium production is not needed for this project. Rather, a calculation simplified by holding

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[1] The model is not limited to plants with BTRS. For plants not equipped with BTRS, any deboration with resin would produce the same result.

selected parameters such as neutron fluxes constant, and confirming the calculation results using typical field experience will suffice. This simplified calculation is described below.

The production of ^{7}Li [3] in a PWR is the result of neutron activation reaction on ^{10}B by the following reaction:

$^{10}B(n, \alpha)^7Li$ **Eq. 2-2**

Using the same nuclear theory ^{7}Li is removed each day by the following reaction:

$^7Li(n, \alpha)\alpha$ **Eq. 2-3**

However the concentration of ^{7}Li is almost 1/1000th that of boron and its cross-section for nuclear reaction is 10^5 smaller. So loss of ^{7}Li via nuclear reaction is inconsequential compared to its production rate.

The physical parameters associated with the production of ^{7}Li are as follows:

Table 2-2
Physical Parameters [3] for 7Li Production

^{10}B abundance	Thermal Neutron Flux, φ	Fast Neutron Flux, φ		^{10}B Thermal Neutron Cross-section, σ	^{10}B Fast Neutron Cross-section, σ		^{7}Li Abundance	^{7}Li Thermal Neutron Cross-section, σ	^{7}Li Fast Neutron Cross-section, σ	1 barn
19.9 %	Variable n/cm^2-sec	Variable n/cm^2-sec		3840 barns	1730 barns		92.5 %	0.045 barns	0.020 barns	$1x10^{-24}$ cm^2

The equation that is used to calculate the production of the stable isotope [5] ^{7}Li from reaction (2-2) is:

$N_{new} = N_{target} \times \varphi \times \sigma \times time,$ **Eq. 2-4**

where the parameters have the identities and units defined in Table 2-2. The design of each core is slightly different and therefore the values for the thermal and fast flux will vary somewhat from core to core. The energies for neutrons follow a Maxwell-Boltzmann distribution. The mean value for thermal neutrons is about 0.03 eV and that for fast neutrons about 2 MeV. Additionally the values given for the nuclear cross-sections are nominal values for a neutron energy range. Cross-sections are complex functions of neutron energy.

Appendix D has a table with examples of varying fluxes and the impact on the amount of lithium produced. [4]

Lithium introduced by the mixed bed that has cation resin in the ^{7}Li form.—The mass action reaction equilibrium expression for this is given in equation 2-5, shown as selectivity of hydrogen with respect to lithium (inverse of convention) because the resin is in lithium form.

$$\boldsymbol{K} = \frac{[\boldsymbol{Li}]*[\boldsymbol{R-H}]}{[\boldsymbol{H^+}]*[\boldsymbol{R-Li}]}$$ **Eq. 2-5**

Where:

K = Equilibrium constant

$[Li]$ = The concentration of lithium going into the mixed bed

$[H^+]$ = The concentration of hydrogen going into the mixed bed

$[R-H]$ = Concentration of hydrogen on resin

$[R-Li]$ = Concentration of lithium on resin

Important assumptions about this equation are given below:

- Equilibrium kinetics are applied at all times; no unsteady state conditions, exchange fronts, or zones are considered.
- The resin is well mixed and free of contaminants.
- The equilibrium constant is 1.1
- The concentration of hydrogen is calculated from the pH
- The concentration of more selective cations in the RCS (Co, Zn, Cr, Fe) is very low compared to Li
- $[R-H]+[R-Li]$ = 1.7 eq/L

To find the change in the resin-lithium concentration, the prior day [R-Li] was subtracted from the current [R-Li]. While lithium is increasing day-to-day in the reactor, it will be deposited on the resin. Therefore, for the first 108 days of the 500 day cycle, this column is negative. However, once the ratio of [Li] to [H] decreases, lithium is removed from the resin and brought into the system. For a full derivation, see Appendix A.

There are five locations where lithium can be lost. These are the cation bed, the holdup tank (HUTS)[2], leakage from the reactor, and two sample lines. For a full derivation of lithium lost to the HUTS, see Appendix B. The sample lines included are from the RCS and the cation bed effluent. It was determined that the loss of lithium in the mixed bed and the sample from the mixed bed are insignificant and are not included on the spreadsheet. The equations for the loss of lithium from the other areas are given below:

Holdup Tank: $\frac{C_{Li,t1}+C_{Li,t2}}{2} * \ln\left(\frac{C_B-C_{B,t1}}{C_B-C_{B,t}}\right) * V$ **Eq. 2-6**

During dilution, C_B is 0

RCS Leak: $C_{Li} * v_{LR} * t$ **Eq. 2-7**

RCS Sample: $C_{Li} * v_{RCSS} * t$ **Eq. 2-8**

Cation Bed Sample: $C_{Li} * v_{CBS} * t$ **Eq. 2-9**

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[2] Hold-up tanks have a variety of names in different plants. Throughout this document, this is a generic term for a tank where letdown is directed when water is removed from the RCS to dilute the boron concentration in the RCS.

Where:

C_{Li} = Concentration of lithium

$C_{Li,t1}$ = Concentration of lithium when reactor coolant is first diverted to the holdup tank (HUTS)

$C_{Li,t2}$ = Concentration of lithium when reactor coolant flow to the HUTS is turned off

C_B = Concentration of boron in the reactor makeup stream (e.g. blender outlet or blended flow for Westinghouse design plants)

$C_{B,t1}$ = Concentration of boron in system when reactor coolant is first diverted to the HUTS

$C_{B,t2}$ = Concentration of boron in system when reactor coolant flow to the HUTS is turned off

ν_{LR} = RCS leak rate

ν_{RCSS} = Flow rate to RCS sample

ν_{CBS} = Flow rate to cation bed sample

V = Volume of the RCS

t = Time

These equations are entered in the spreadsheet and converted to pounds of lithium. The cation bed operation for lithium control is modeled by determining lithium production and losses and initiating cation bed operation when lithium deviated from the control program by > 0.5%. Although the EPRI PWR Primary Guidelines [6] establish varying lithium control limits as a function of lithium concentration, a 0.5% control band for purposes of this model does not introduce significant error because the total mass of lithium required to be removed to control reactor coolant pH at a particular value does not change as a function of the control band; adjustments to the band only impact the frequency of intervention required to maintain the lithium concentration within the band.

2.3 Operational Requirements

2.3.1 Cation Bed Operations

One of the modeling objectives is to look at the past few days of lithium data and determine when the cation bed will need to be run to remove lithium from the system. The column titled “Calc Li Conc” is the total lithium in the system if the cation bed is not run. It is the summation of the measured lithium (existing lithium in the reactor coolant) and the input lithium (from generation and manual addition) while subtracting the outputs of lithium (losses calculated in Section 2.1.2). Next, a linear extrapolation using the previous two days of calculated lithium to estimate the lithium concentration for the following day. The calculated lithium concentration was then compared to the measured lithium value. A graph of these two parameters is given in Figure 2-1.

The term “Measured Li” in Figure 2-1 and the model refers to the estimate of what the lithium concentration should be based on a linear extrapolation using the calculated lithium from the previous two days. The term “Calculated Li” in Figure 2-1 and the model refers to the mass balance model output for a particular day.

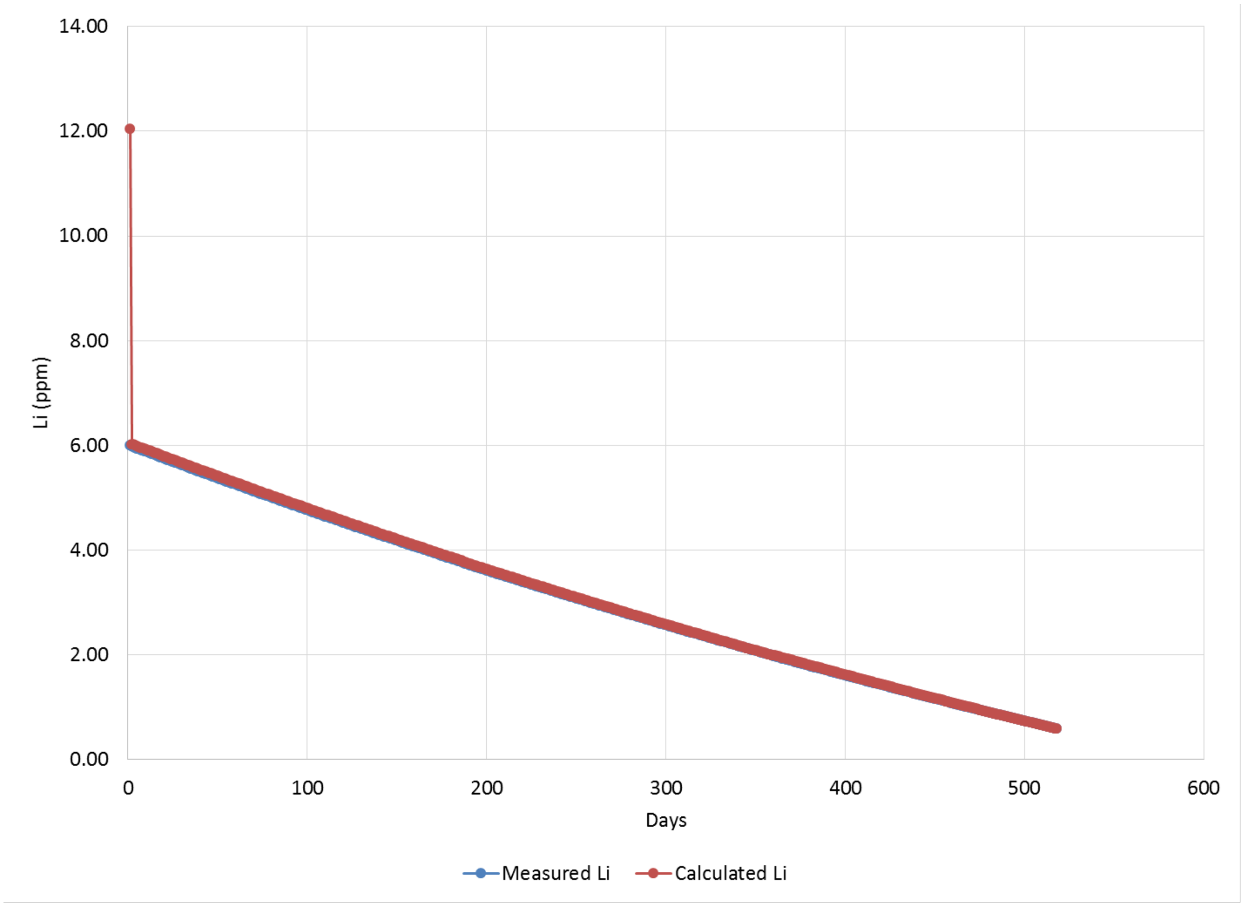

Figure 2-1
Measured and Calculated Lithium

When the calculated lithium concentration is greater than the measured value by 0.5% (an arbitrary adjustable parameter), the spreadsheet calculates how much lithium needs to be removed from the RCS coolant with the cation bed. This lithium mass is the difference of the predicted lithium and the measured lithium. The daily coolant flow rate to the cation bed can be determined by dividing the output of lithium in the cation bed by the measured lithium concentration, and converting to gallons per day. In the column for the daily flow rate to the cation bed, the equation is multiplied by 1.01. This is to reflect that the cation bed is only 99% efficient and therefore must be run 1.01 times as long to get out the desired amount of lithium.

The same logic is used to determine if the concentration of lithium in the system gets too low. If the predicted value is less than the measured value by 5%, the spreadsheet calculates how much lithium needs to be injected by taking the difference of the predicted value and measured value, converted into pounds.

This process is further explained by Figure 2-2 and Table 2-3.

2.3.2 Calculation Flow Diagram

A calculation flow diagram for the model is given below in Figure 2-2.

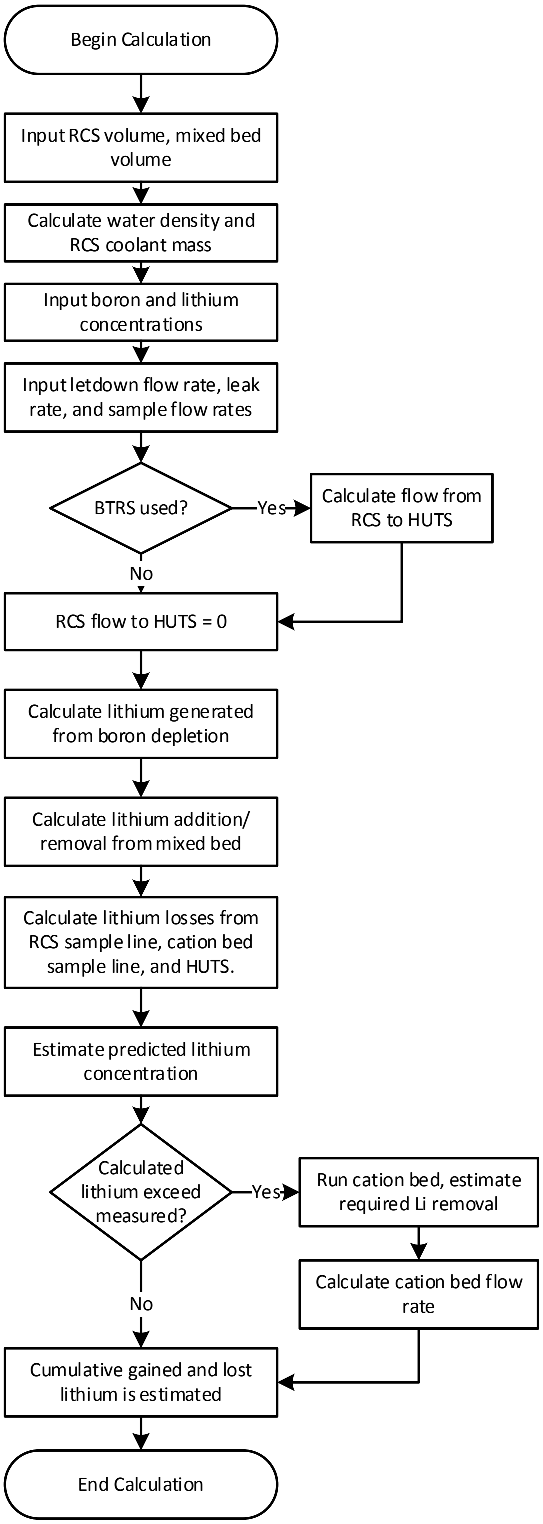

Figure 2-2
Algorithm Description for Cycle Lithium Mass Balance

Table 2-3
Explanation of Flow Chart

Number, Corresponding Column(s)	Explanation	Equation/Assumption
Input RCS volume, mixed bed volume	Input two parameters	Used common values for purpose of theoretical spreadsheet
Calculate water density and RCS coolant mass	Calculates density and mass of water in RCS	Based on equation between relationship of temperature and density for water
Input boron and lithium concentrations	Input two parameters	pH can be calculated from these concentrations. For the purpose of this report, a pH of 7.4 was assumed.
Input letdown flow rate, leak rate, and sample flow rates	Input four flow rates	These four columns are all inputs by the plant. For the purpose of this report, theoretical flow rates are used.
Calculate flow from RCS to HUTS	Input parameter	If no BTRS is used, the lithium flow to the HUTS is calculated with the following equation: $= \frac{C_{Li,t1} + C_{Li,t2}}{2} * \ln\left(\frac{C_B - C_{B,t1}}{C_B - C_{B,t}}\right) * V$ (See Appendix A for full explanation)
Calculate lithium generated from boron depletion	Calculates lithium generated from boron	This is described in section 2.1.2
Calculate lithium addition/removal from mixed bed	This value was calculated and determined to be negligible	N/A
Calculate lithium losses from RCS sample line, cation bed sample line, and HUTS	Calculates lithium lost to mixed resin bed (negative) or lithium gained (positive)	RCS Sample Lithium $= C_{Li} * v_{RCSS} * t$ Cation Bed Sample Lithium $= C_{Li} * v_{CBS} * t$ Lithium to HUTS $= C_{Li} * v_{HUTS} * t$
Estimate predicted lithium concentration	Calculates lithium concentration in RCS before cation bed is used	= Measured lithium + lithium inputs – effluent lithium
Calculated lithium exceed measured?	Compares calculated lithium to goal value of lithium	If the calculated lithium exceeds the goal by 0.5%, the cation bed is run.
Cumulative gained and lost lithium is estimated	All inputs and outputs of lithium have been calculated	The values are used to create graphs and determine where lithium ends up in the RCS.

2.3.3 Results

The cumulative outputs are displayed below in Figure 2-3.

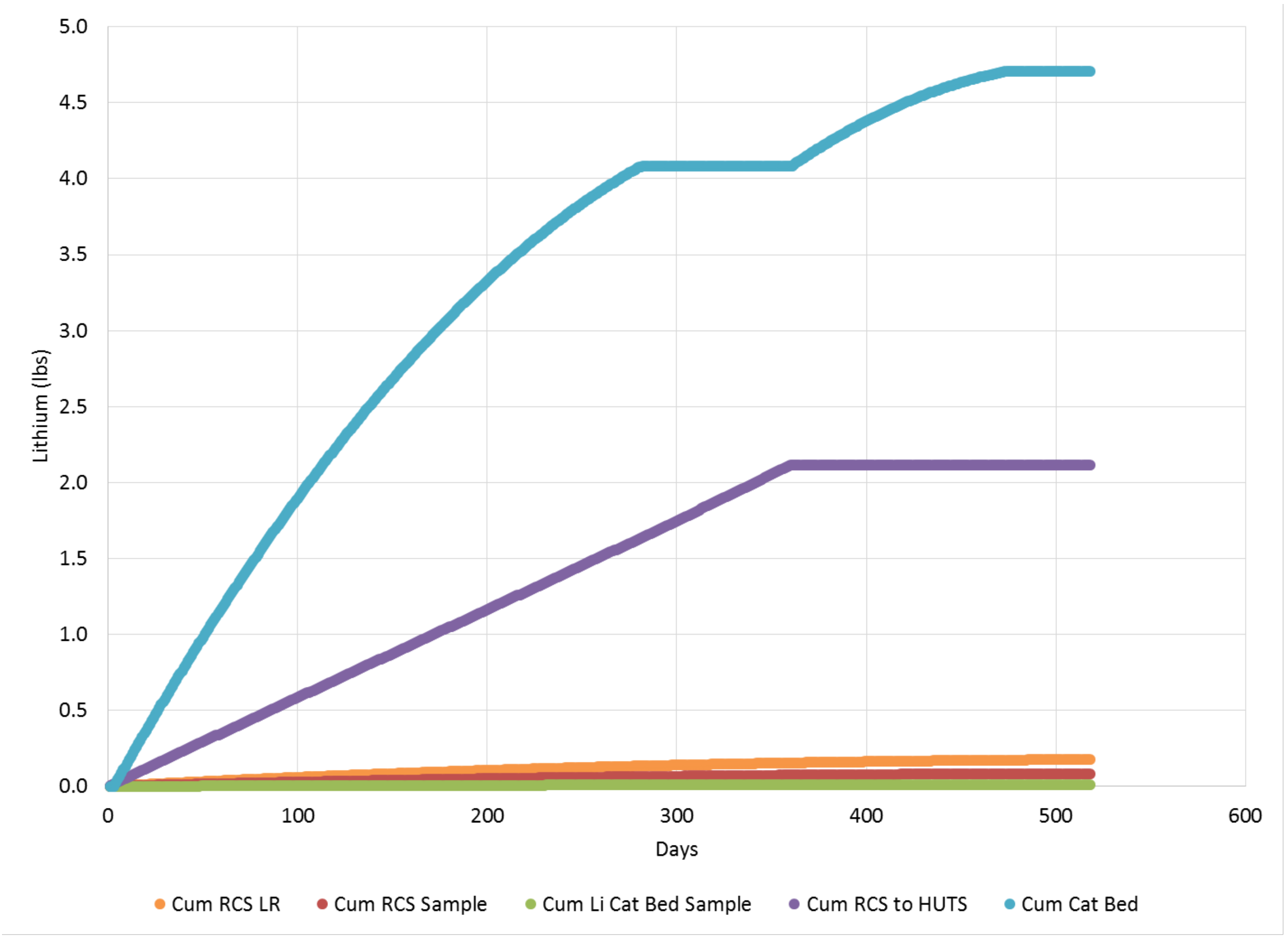

Figure 2-3
Cumulative Lithium Outputs

The cumulative RCS sample is covered by the cumulative lithium bed effluent sample, and both are insignificant losses of lithium. The most significant losses of lithium are due to the cation bed and to the holdup tanks.

These results are summarized in Table 2-4 below.

Table 2-4
Pounds and Percent of Lost Lithium for a Typical Cycle

	Lbs	Kg	%
RCS Sample	0.09	0.04	1.19
RCS Leakage	0.18	0.08	2.49
HUTS	2.11	0.96	29.83
Cation Bed	4.71	2.13	66.32

The model output shows that for typical plant operations, a sustainable lithium inventory (e.g. greater than the mass of lithium required for beginning of cycle requirements) is retained on the cation resin, suggesting that lithium recovery from the cation resin is a possible strategy for creating a source of ^{7}Li if normal supplies are interrupted. This option along with an on-line recovery option are examined in detail in Chapter 3.

2.4 References

1. Memo from resin supplier to J. McElrath, October 18, 2013.
2. Mark Simmons (personal communication, 27 July 2014).
3. Nuclear Constants and Reactions are reproduced from Interactive Chart of the Nuclides, National Nuclear Data Center, Brookhaven National Laboratory, based on ENSDF and the Nuclear Wallet Cards. http://www.nndc.bnl.gov/chart
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5. EPRI 3002000505, “Pressurized Water Reactor Primary Water Chemistry Guidelines, Volume 1, Revision 7,” Final Report, April 2014.

3
LITHIUM RECOVERY OPTIONS

As described in Section 2, one option for lithium recovery is to regenerate the lithium from cation resin used for reactor coolant lithium control. A second option considered in this report is to recover lithium via an on-line process in-plant using a reactor coolant sample stream. Laboratory test conditions were developed for both of these options, and subsequent laboratory testing was completed with the results given in Chapter 4.

Other options to recover lithium from exhausted resins, such as ashing and wet oxidizing, were explored but determined to be less practical due to operational hazards, waste generation, and difficulty associated with obtaining a useful lithium product with sufficient purity from the process. Therefore, these options will not be discussed further in this report.

3.1 Lithium Separation using Electrodeionization Techniques

3.1.1 Evaluation of RCS Sampling Equipment for Recovering Lithium

PWRs use a wide range of sampling system designs for obtaining routine reactor coolant samples. Some plants use portions of a post-accident sample system mandated to be installed at all plants in the wake of the TMI accident. Other plants, particularly those built prior to the post-accident sampling requirements, have a simpler panel located in or near the laboratories. Reactor coolant samples can generally be obtained from the primary system hot leg, or the chemical volume control system (CVCS) inlet. Samples are cooled to approximately room temperature for safe handling and proper operation of any installed instrumentation. Typically, the CVCS inlet sample tap is located downstream of the CVCS heat exchangers, reducing the heat load on the sample panel cooling equipment. Samples can also be collected from the CVCS demineralizer outlet. Many plants can elect to return the sample stream to the volume control tank (VCT) to minimize waste generation.

Reactor coolant samples are generally not left continuously flowing and are only operated for grab sampling evolutions. The sample flow capabilities of reactor coolant sample panels vary widely between plants. Sample cooling equipment may not be sized or maintained in a manner to support continuous hot leg sample flow. Sample flow rates may be as low as 1 liter per minute depending on the design of the sample line and panel.

The mass of lithium needed to be removed from the RCS on a daily basis was determined from the mass balance calculations documented in section 2. Using the reactor coolant lithium concentration for each day, the sample flow rate required to accomplish reactor coolant lithium control was determined. This value ranges from approximately 3 liters per minute to as high as 10.5 liters per minute for the operating cycle modeled. Flow rates as high as 10 – 15 liters per minute are not typically achievable from primary system sample panels. The sample system would likely require modifications to support a continuous reactor coolant sample flow rate in the range required for lithium control throughout the operating cycle.

Although using the primary sample system for complete reactor coolant lithium control is not practically feasible, it may still be possible to recover lithium using the sampling system. The maximum mass of lithium that could be removed from the RCS using a continuous sample flow in the range of 1 – 3 liters per minute was determined using the mass balance model. The results are shown in Table 3-1 below.

Table 3-1
Lithium Removal Using Typical Sample System Flow Rates (18 month cycle)

Flow Rate (L/min)	Lithium Removed lbs (kg)
1	5.09 (2.31)
3	15.28 (6.93)

Although it would not be possible to accomplish complete RCS lithium control during certain parts of the operating cycle using an RCS sampling stream (some lithium removal using cation resin would still be required), it appears feasible to recover the mass of lithium needed for converting the CVCS demineralizer to the lithium form and for manual chemical additions to the RCS, thereby creating a sustainable baseline operating strategy in the event of lithium supply interruption. A typical plant uses 3-4 pounds (1.4-1.8 kgs) of ^{7}Li for an entire cycle (not including mid-cycle outages). If approximately 5 pounds (2.3 kgs) of ^{7}Li can be recovered during a particular operating cycle, a plant would have a sufficient mass of lithium for the following cycle, thereby creating a sustainable alternate lithium supply without recovering lithium from cation resin.

3.1.2 Electrodialysis Cell

An electrodialysis (ED) or electrodeionization (EDI) cell can be used to separate lithium from boric acid to produce lithium hydroxide. A diagram of this cell is shown below.

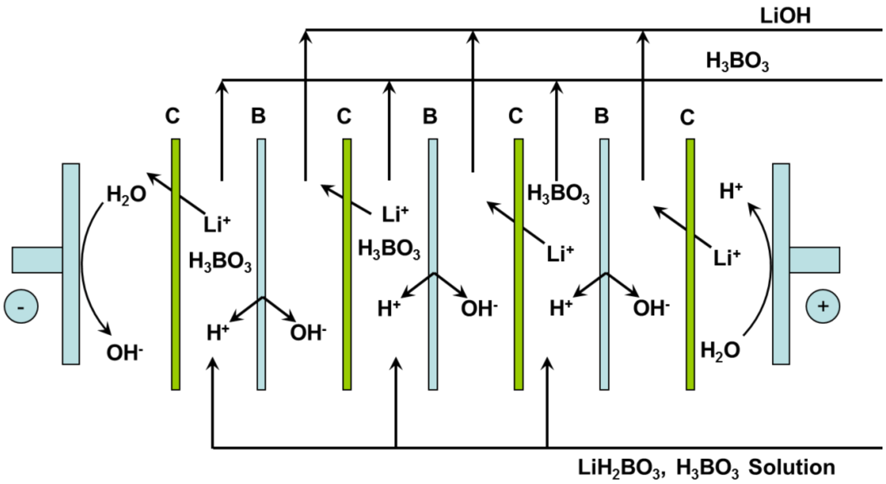


Figure 3-1
Electrodialysis Cell [2]

The EDI cell uses ion exchange resins and membranes and direct electrical current to convert LiH_2BO_3 and water to LiOH, H_3BO_3. Typical continuous electrodeionization (CEDI) modules are made up of alternating cation and anion membranes (the anion membrane would be in place of the bipolar membrane in the above schematic). While there is some size exclusion, the main method of prohibiting unwanted transfer of ions is through charge exclusion. The electric current continuously regenerates this resin so it does not become exhausted. This allows for sustainable separation of lithium. The transport of the anion associated with a strong acid occurs readily through the anion membrane and the product compartment would produce a concentrated salt that could then be processed in a three-compartment bipolar ED cell to produce the acid and base from the salt. However, in the case of boric acid, which is a very weak acid, the first pKa is 9.23, thereby indicating that at this pH, over half of the acid is present as neutral H_3BO_3, and half as monovalent anion, thereby limiting application of anion membranes to higher pH's.[3]

Although its high pKa limits the use of anion membranes, this property helps in the removal of lithium from a solution of lithium borate / boric acid using a cell as depicted in Figure 3-1. As mentioned, a solution consisting of half lithium borate (LiH_2BO_3) and boric acid, will have a pH of around 9.0, and therefore the proton concentration at all times will be limited to very low concentration. As proton is made at the bipolar membrane, it quickly associates with the dissociated acid and therefore does not compete with lithium transport across the cation membrane. High degrees of conversion are possible; the conversion is only limited by the drop in conductivity in the feed product.

Besides recovering the lithium from a feed stream, the EDI can also supply a stream of boric acid that has been substantially depleted of lithium.

Continuous Electrical Deionization (CEDI), a type of EDI, was evaluated in the liquid radwaste (LRW) system at Braidwood. The results of this field demonstration are documented in an EPRI report [3]. The following description of the CEDI process is taken from that report:

> "CEDI uses a combination of ion exchange resins and membranes and direct electric current to continuously deionize water without the need for regeneration chemicals. Like conventional ion exchange, continuous electrodeionization removes dissolved, ionizable materials such as salts, acids and bases… Contaminants such as particles and large organic molecules that do not ionize, do not pass through the ion exchange membrane - and are not removed.
>
> Continuous electrodeionization modules consist of mixed-bed resins sandwiched between alternating anion and cation membranes. These membranes are actually ion exchange resins manufactured in sheet form. Resin compartments in this "sandwich" construction alternate between diluting and concentrating compartments. Compartment sets are called cell pairs and form the basic element in a module.
>
> In the module, direct current is applied to the anode (positive electrode) on one end of the module, and to the cathode (negative electrode) on the other end. This electric potential drives the ions captured by the ion exchange resins through the membrane.
>
> Because the resins in the module are continuously regenerated by the electric current, they do not become exhausted."

---

[3] The incorporation of mixed resin (anion resin) in the feed compartment may help to increase the local pH of the feed, thereby increasing the amount of dissociation.

For a lithium recovery operation, the reject stream consisting of lithium and boron, would be recovered for further processing or re-use.

The CEDI equipment installed at Braidwood was manufactured by Ionpure Technologies. The design parameters for the individual CEDI modules are shown in Table 3-2. Four modules were arranged in parallel to meet the 30 gpm system processing rate. Refer to Figure 3-2 for a picture of the system installed at Braidwood.

Table 3-2
CEDI Module Specifications IP-LXM24MK-1 [9]

Parameter	Value
Nominal Flow Rate	12.5 gpm (2.8 m3/hr)
DC Power	0-600 VDC, maximum 10 amps
Weight	200 lbs (91 kg)
Nominal Pressure Drop	2 to 30 psid (1.4-2.1 bar)
Maximum Feed Pressure	100 psi (7 bar)
Typical Recovery	90 to 95%
Product Conductivity	> 16 megohm

Figure 3-2
CEDI System Installation at Braidwood [2]

3.1.3 Electrodeionization Equipment Footprint

Some plants have little or no room for CEDI equipment placement in the immediate vicinity of the reactor coolant sample panel. However, suitable locations may be found outside of sample rooms or along the route of CVCS demineralizer outlet sample lines. Based on the Braidwood test unit, an approximate equipment footprint conservatively sized at 4 ft (1.2 m) by 12 ft (3.7 m) can be used for determining suitable placement locations. A suitable location is likely available for placing a skid of approximately these dimensions somewhere along the route of the CVCS demineralizer outlet sample line at many PWR plants. Use of the CVCS demineralizer outlet sample is preferred to minimize radionuclides or particulate introduction to the CEDI unit.

3.1.4 Other Considerations

Other considerations for equipment placement may include:

- Access to power, spare bus capacity, and a spare breaker cubicle or capacity on an existing breaker.
- Availability of a spare control room annunciator panel and access to wiring for that annunciator circuit for use as a trouble alarm if required.
- Access to a CVCS demineralizer outlet sample line with sufficient flow to support the program requirements.
- Access to a sample system return line or other means to route system effluent to the VCT for return to the RCS.
- Method for collecting the CEDI “reject” stream and method(s) for additional processing to recover lithium from the “reject” stream in a useable form.
- Sampling capability (including drains) and on-line chemistry monitors on the CEDI skid.
- Heat load and ventilation considerations for auxiliary building temperature control requirements
- Undesirable contaminants from the process stream may be introduced into the CEDI unit if there is not a suitable sample stream or process flow from the CVCS demineralizer outlet.

3.2 Lithium Recovery from Cation Demineralizer Beds

As discussed in Chapter 2, it should be possible to recover a sustainable supply of ^{7}Li from exhausted cation demineralizer resin. This option is developed further in this section.

3.2.1 Demineralizer Bed

A simplified diagram of a cation demineralizer is shown in Figure 3-3.

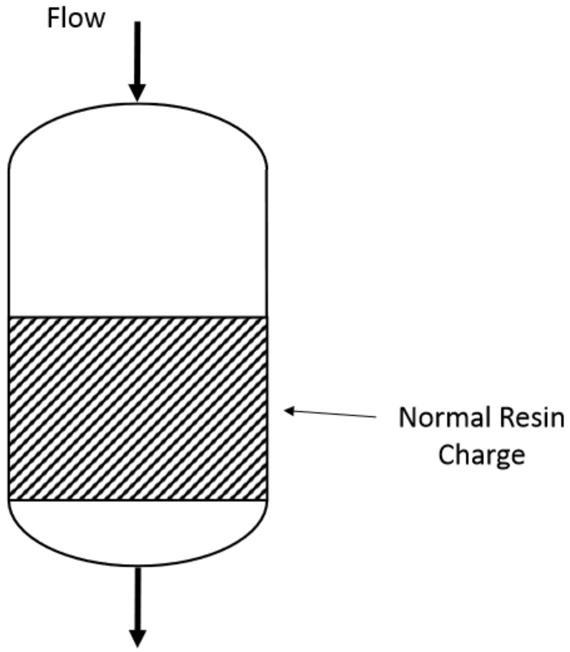


Figure 3-3
Cation Demineralizer

A typical RCS cation demineralizer vessel is 20 ft^3 (0.57 m^3) with 10 ft^3 (0.28 m^3) of resin. When this bed is needed, the RCS passes through and lithium is removed from the system and replaced with hydrogen. In Section 2, the amount of lithium on this bed after a standard cycle was calculated to be 4.7 lbs (2.1 kg) [1]. To remove lithium from the resin, the bed would have to be regenerated with an acidic solution. This is further explained in Section 5.

3.2.2 Chemical Regeneration and Purification of Lithium

Most PWR plants use a hydrogen-form cation demineralizer bed either in parallel with or in series downstream of a lithium-hydroxide form mixed bed demineralizer for the purpose of reactor coolant lithium removal. The cation bed is placed in service periodically as required to maintain proper lithium concentration coordinated with reactor coolant boron concentration. This cation demineralizer resin presents an opportunity for lithium recovery at an off-site facility without requiring costly modifications or impacting plant operations. Rather than shipping exhausted cation demineralizer resin to an offsite burial location, the resin could be shipped to a vendor processing facility for lithium recovery.

The exhausted cation demineralizer resin would contain radionuclides, so the offsite processing facility would need to be properly licensed and designed to handle radioactive materials within the ranges expected from normal operations. Dose or curie content limits would need to be established for individual shipments to ensure that facility license limitations would not be exceeded.

Cation resin regeneration requires the use of an acid with sufficient solution strength to displace the target cation(s) from the resin beads and replace them with a desired cation, often H^+. Most cation resin regeneration agents would introduce a significant contaminant to the lithium solution removed from the resin; this contaminant would not be compatible with reactor coolant system or fuel materials. However, the use of boric acid as the regenerant would be acceptable: boric acid can displace lithium from cation resin, and boric acid is obviously compatible with primary system materials. As a regenerant, boric acid would have limited capability to remove divalent metals and radionuclides from the resin, thereby reducing the amount of post-regeneration processing necessary to further purify the lithium solution.

Following regeneration, the resultant solution is expected to be a combination of lithium borate and boric acid. Any divalent metals or radionuclides in the recovered solution could be removed using a lithium-form cation demineralizer. The concentration of the recovered solution could be adjusted if necessary using reverse osmosis or an evaporator to achieve a lithium and boron concentration acceptable to transportation, storage, and re-use in the primary system at plants. Alternatively, ED or EDI with bipolar membrane could be used to produce lithium hydroxide and boric acid.

3.2.3 Other Considerations

PWRs have the potential to generate spent resin with extremely high dose rates. For example, a refueling outage purification demineralizer can read over 2000 R/hr on contact following an outage. Co-mingling this high activity resin with average or low activity resin in a spent resin tank is undesirable for several reasons:

- The disposal cost for high activity resin is considerably higher than average or low activity resin.
- The in-plant dose consequences of storing high activity resin in a spent resin tank can be unacceptable.

For these and other logistical reasons, most plants have developed methods to directly transfer high activity CVCS resin to a radwaste disposal liner within a shielded transportation cask for cases where plant operating conditions require transfer high activity resin from a CVCS demineralizer vessel. Although the cation resin is expected to be relatively low to average activity concentrations, depending on CVCS system design, these same methods for transferring a CVCS bed directly to a liner could be used to recover lithium-laden cation resin without co-mingling with other resins in a spent resin tank.

3.3 References

1. ChemStaff. (7 August 2014). Feasibility Study of an On-line Lithium Removal Process.
2. "Application of Continuous Electrical Deionization for Liquid Radwaste Processing at Braidwood Generating Station," EPRI 1015116, December 2007.
3. LX-MK Module, Revision F, Pre Installation Instructions, page 2-3.

4

FEASIBILITY TEST OUTCOMES

Laboratory experiments were designed and performed to demonstrate the lithium recovery options developed in the previous chapters:

- Online recovery using in-plant equipment and a reactor coolant sample stream.
- Recovery by regenerating exhausted cation resin.

The two different methods were evaluated with realistic lithium and boron concentrations for the process solution. The results from these experiments were then used to estimate the economics of the process and further define the footprint of the recovery system that might be used for on-line recovery in a plant.

Lithium recovery by regenerating exhausted cation resin could utilize a process similar to that shown in Figure 4-1 below. While Figure 4-1 does not necessarily reflect the pilot-scale experiments performed, it demonstrates what this process could look like on an industrial scale.

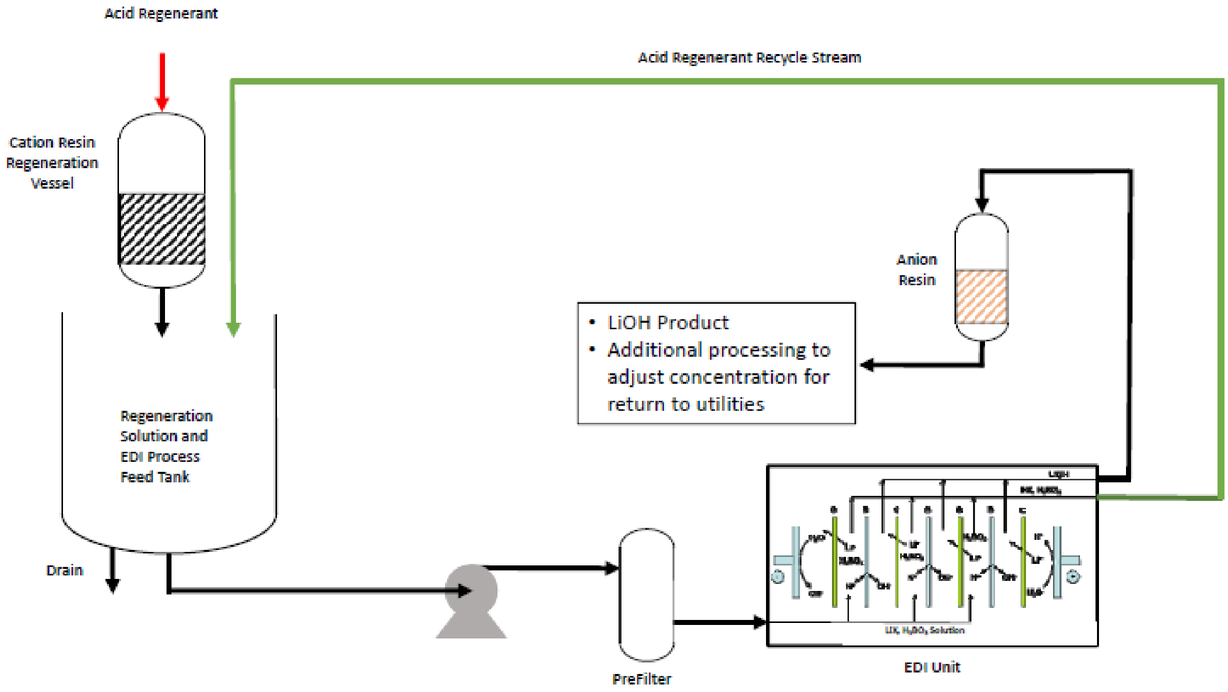


Figure 4-1
Process Diagram

4.1 Recovery of Lithium via On-line Recovery in the Plant

In order to test the applicability of incorporating an on-line lithium recovery system at the plant level, experiments were performed utilizing solution concentrations predicted at various times during the PWR cycle.

- 1200 ppm B, 3.5 ppm Li (~200 days into cycle)
- 800 ppm B, 2.5 ppm Li (~300 days into cycle)
- 400 ppm B, 1.4 ppm Li (~400 days into cycle)
- 100 ppm B, 0.75 ppm Li (~500 days into cycle)

The electrodialysis cell required for this testing was built as shown in Figure 3-1. The cell incorporated Neosepta BP-1E bipolar and CMB cation (Astom, Japan) membranes with Amberlyst 15WET Cation Resin (Rohm and Haas, USA) in the Feed Compartment. The BP-1E membrane is specifically designed to split water into proton and hydroxide; therefore changing the pH of the solutions in contact with either side of the membrane. The CMB membrane allows for the transport of cations and is specially made to decrease the amount of hydroxide back-migration from the lithium hydroxide compartment back into the feed compartment, thereby increasing the current efficiency for lithium hydroxide production. The Amberlyst Resin is a strongly acidic sulfonic acid resin (1.7 eq/L). Other details about the electrodialysis cell and setup can be found in Appendix C.

Results for experiments performed with this cell are shown in Table 4-1 and included:

- Batch operation with the highest solution concentration. In this case, the same solution is recirculated through the cell multiple times.
- Single pass operation with variable flow rates determining conversion per pass for each of the solution concentrations listed above. These experiments also included variation in the applied cell voltage.

Table 4-1
Results for Experiments Utilizing BP-1E/CMB/Cation Resin for Lithium Removal and Lithium Hydroxide Production. 0.2 M LiOH was used as Start Base in All Experiments.

Test	Test time	Start feed conc.	End feed conc.	Cell voltage	Ave. current density	CE Li removal	End OH conc
	min	Li / B (ppm)	Li / B (ppm)	V / cell	mA/cm^2	%	M
Single Pass / Variable Flow							
919-56	75.0	0.68 / 119	0.17 / 119	1.6	6.5	1.8	0.24
919-45	75.0	1.38 / 422	0.33 / 433	1.6	8.5	2.6	0.25
919-40	62.0	2.5 / 1179	0.96 / 1179	1.6	10.7	5.1	0.25
919-48	72.0	3.1 / 1179	0.81 / 1179	1.6	8.2	6.0	0.25
919-52	71.0	3.6 / 1158	1.19 / 1169	0.8	1.0	52.0	0.22
Batch Operation							
919-35	210.0	3.6 / 1179	0.37 / 1179	1.6	10.4	3.2	0.30

4.1.1 Batch Operation for Lithium Removal

In experiment 919-35, a large batch (36 L) of higher concentration solution was continuously circulated through the cell decreasing the bulk reservoir solution from 3.6 ppm lithium down to less than 0.4 ppm lithium. As discussed in further detail in Appendix 5C, a constant voltage of 1.6 V/cell was applied across the active cells, and the resulting current density during the run increased as shown in Figure 4-2. The increase in current density is due to an increase in the conductivity of the lithium hydroxide base product from 0.2 M to 0.3 M over the course of the run. Samples were taken periodically during the test, and the moles of lithium and boron in the feed is shown in Figure 4-3 and Figure 4-4. The lithium decreased throughout the run, with a corresponding increase in lithium in the base compartment (inset). The overall mass balance for the run was very good, producing a 0.3 M (2.1 g/L Li) lithium hydroxide solution containing 0.015 M (160 ppm) boron. The boron concentration in the feed also did not change dramatically.

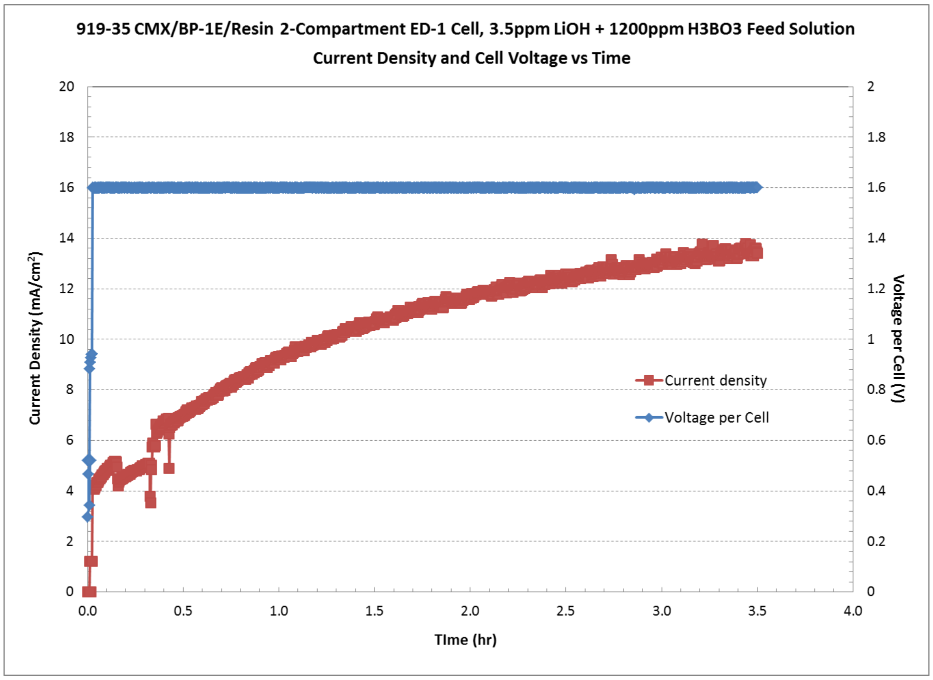


Figure 4-2
Current Density and Individual Cell Voltage for Experiment 919-35 with BP-1E/CMB/Cation Resin Cell.

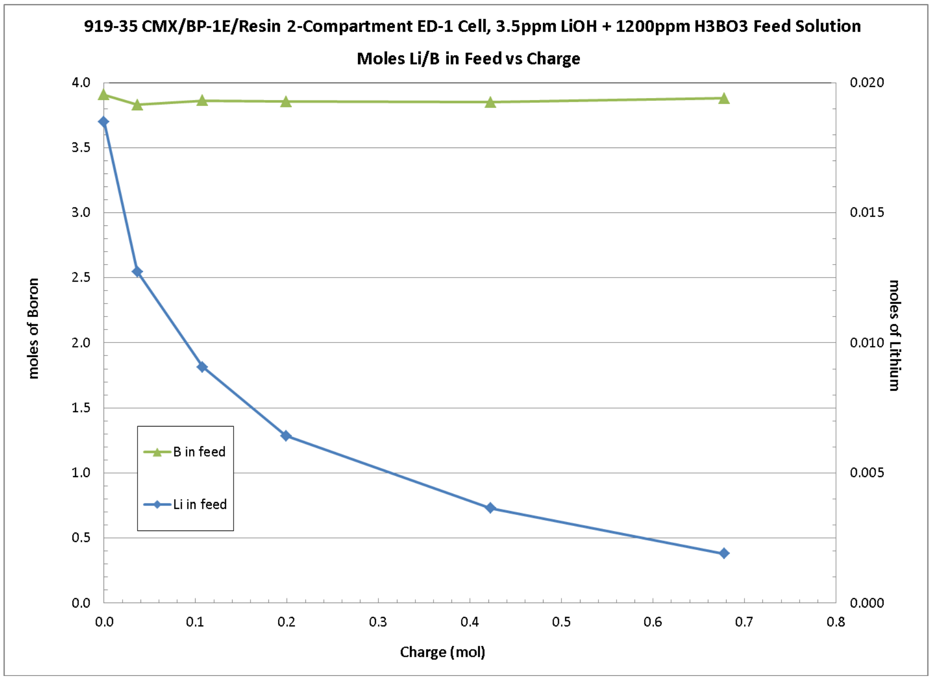


Figure 4-3
Moles of Boron and Lithium in the Feed during Batch Electrodialysis 919-35

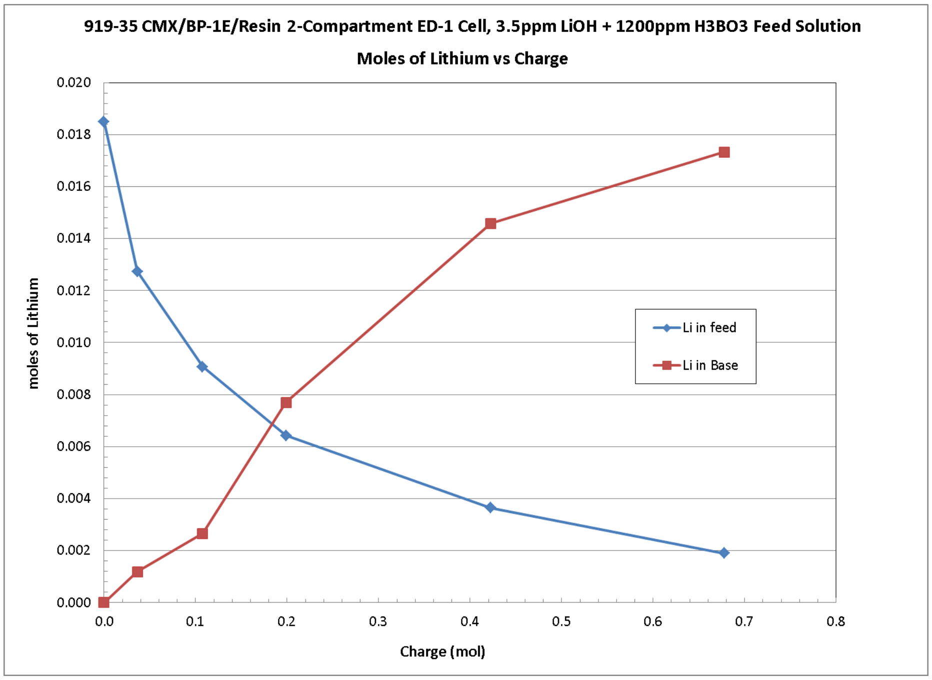


Figure 4-4
Moles of Lithium in the Feed and Base during Batch Electrodialysis 919-35

The extent of conversion during the course of the run could also be monitored via pH and conductivity measurement. As shown in Figure 4-5, the outlet pH and conductivity both decreased considerably during the experiment. The initial pH of the solution at room temperature was about 6.3 dropping to about 5.0, once most of the lithium had been removed.

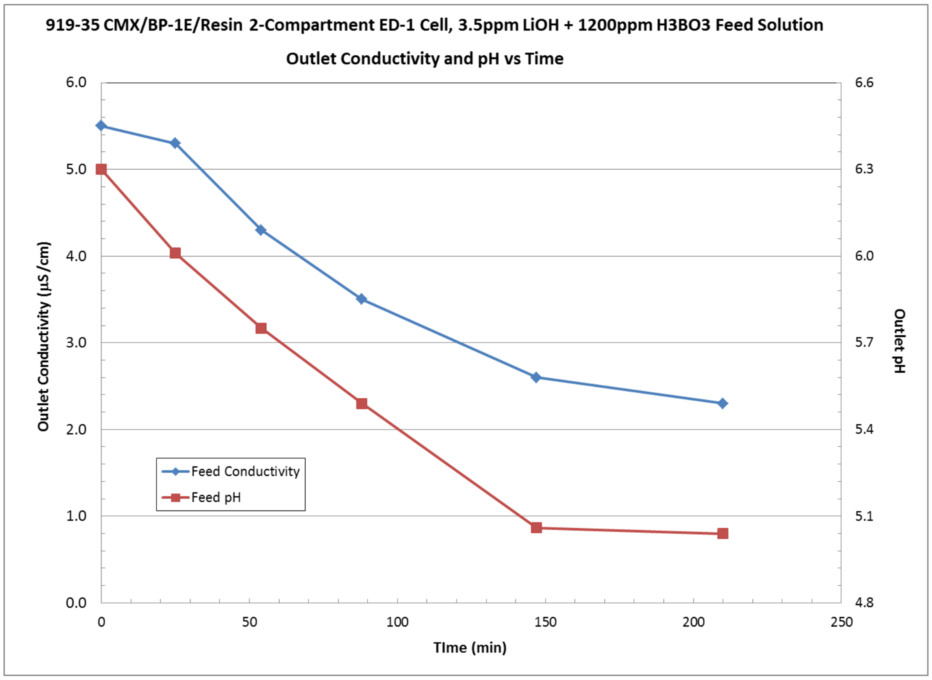


Figure 4-5
Feed Outlet pH and Conductivity During Batch Electrodialysis 919-35

These results indicated that lithium could be effectively removed from a simulated RCS solution, but that the rate of lithium removal decreases as the cycle progresses. Part of this decrease is associated with the lower concentration, but it is also from mixing in the large feed tank. For example, the second sample as measured in the overall drum was 2.5 ppm lithium; however, the solution exiting the cell was already at 0.85 ppm. It was therefore decided to investigate single pass operation of the cell while varying the flow rate to determine the extent of lithium removal.

4.1.2 Single Pass/Variable Flow Experiments

The other experiments listed in Table 4-1 were all performed in single pass mode with the majority of the tests performed over a range of flow rates. The testing can provide estimates as to the removal rate and can be used to size the ED cell for the main plant along with its power consumption. An example of the current density profile obtained during a test with high concentration solution is shown in Figure 4-6. Even though the flow rate is decreased, the current density increases. The overall increase during the run is mostly a function of increase base conductivity, but there is also flow rate dependence as the current density decreases when the flow rate is increased in the last 25 % of the run. Paired with this observation is that the measured rate of lithium removal (mg/min) decreases as the flow rate is decreased as shown in Figure 2-1Figure 4-7. Because the rate of lithium removal decreases while the current density increases, the overall efficiency for lithium removal also decreases as the flow rate is decreased.

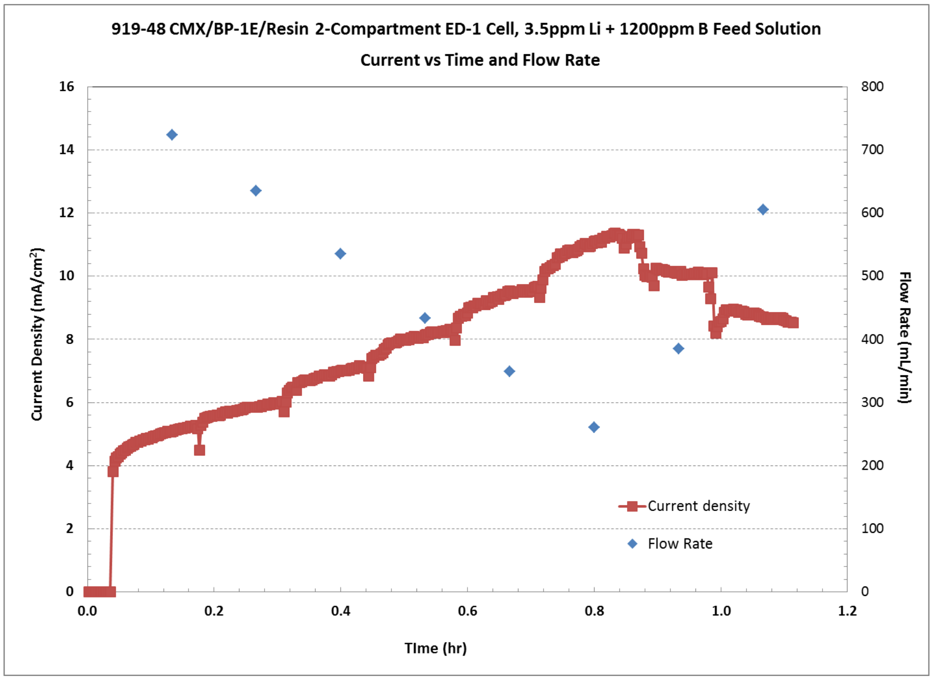


Figure 4-6
Current Density Versus Time and Volumetric Flow Rate for 919-48

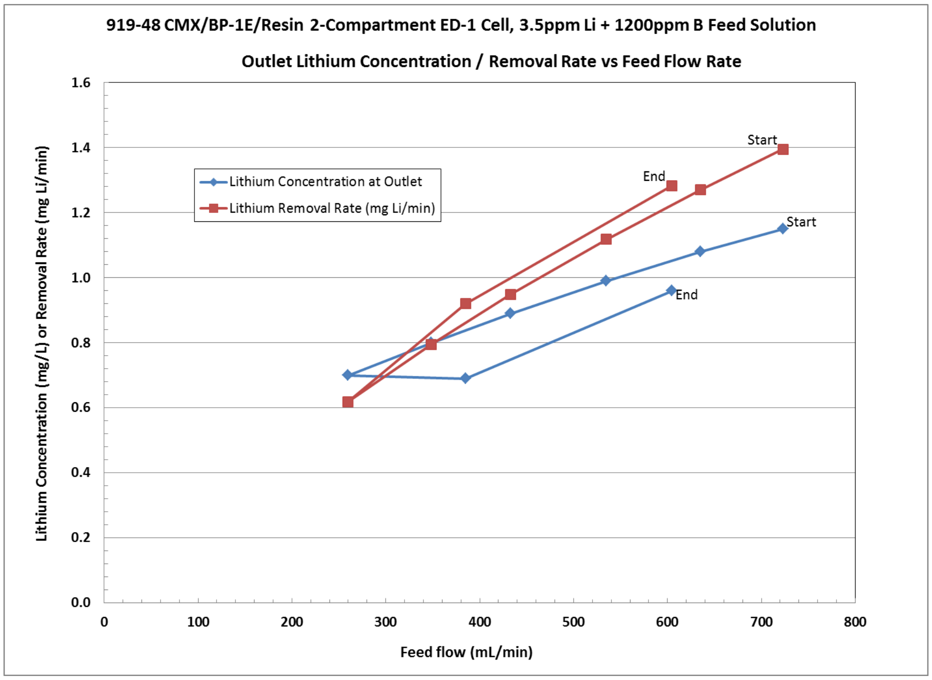


Figure 4-7
Lithium Concentration at Outlet of Cell as a Function of Flow Rate for 919-48

As shown in Table 4-1, the current efficiency for the majority of these tests was a maximum of 6% of the current being associated with lithium removal. Current efficiency is a measure of how much of the current performs the required reaction. Therefore, in the above example, about 94% of the current is not performing lithium removal, but some other reaction. By also monitoring the lithium concentration in the electrode rinse, we have been able to associate the large inefficiency with proton shuttling. Proton generated at the bipolar membrane in the feed is transported across the feed compartment through the ion exchange resin. The lower the amount of lithium on the ion exchange resin bed, the greater the percentage of ion exchange groups on the resin being present as proton, thereby increase the conductivity of the bed (proton mobility is greater than lithium). The higher conductivity of the feed compartment increased the obtained current density. Thus, the lower the lithium concentration in the bed, the higher the overall current obtained and more and more of the current is carried by proton transport. In order to verify this mechanism and to improve the current efficiency of the process the test was performed in a less traditional manner, by limiting the overall current applied to the cell and allowing the applied voltage to drop lower than typically utilized for the bipolar membrane ED.

As shown in Figure 4-8, applying a constant but much lower current density provided a very stable voltage for the majority of the run, and was mostly independent of the flow rate. As listed in Table 4-1, the voltage was about half that applied in previous experiments, and the current efficiency for lithium removal was considerably higher at 52%. i.e. the current passed over the course of the run was more than half associated with lithium removal with much less proton shuttling decreasing the power requirement of the cell Further work optimizing the current/voltage may provide even further improvements. This test provided a decrease in lithium in the feed from 3.57 ppm lithium to 1.19 ppm lithium in a single pass producing a lithium hydroxide concentration of 0.22 M in the receiving/base.

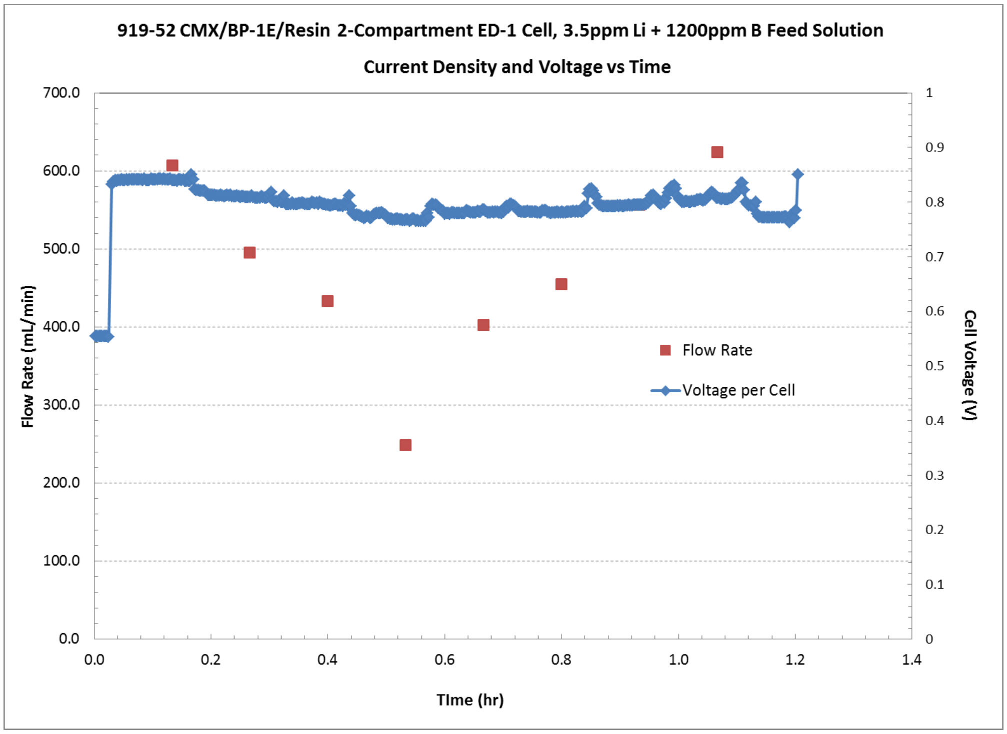


Figure 4-8
Individual Cell Voltage as a Function of Time and Flow Rate at a Constant Current of 1 mA/cm^2 for 919-52

4.1.3 Comparison of Results, Scaled for 2 L/min of RCS Feed

As mentioned in Section 3.1.1, the typical sample system flow rate is between 1-3 L/min. Assuming an average of 2 L/min, the results of the various tests were used to predict a suitable cell area to treat this flow and remove a certain percentage of the lithium in the line. These are presented in Table 4-2. In this case, the solution flowing through the sample line would be treated in a single pass through the electrodialysis cell and sent back to the plant.

Table 4-2
Results obtained from Single Pass/Variable Flow Experiments and Predicted Cell Area, Rate of Lithium Removal and Power Consumption for 2 L/min feed from the RCS Sample Line

Test	Flow	Start Li	End Li	Average Current Density	Percent	Cell Area Required	Mass of Lithium Removed	Cell Power Consumption
	mL/min	ppm	ppm	mA/cm^2	Removed	cm^2	g / day	kWh/day
919-56	563	0.68	0.17	7.6	75%	1778	1.5	0.52
919-45	343	1.38	0.29	9.7	79%	2915	3.1	1.08
919-40	545	2.54	1.0	13.3	61%	1835	4.4	0.94
919-48	385	3.08	0.69	11.8	78%	2598	6.9	1.17
919-52	402	3.57	1.19	1.0	67%	2485	6.9	0.10

Although there is some scatter in the results, in all cases, an electrodialysis cell with 3000 cm^2 (0.3 m^2) of membrane area would be suitable to recover at least 70% of the lithium in the stream. This is not a large ED cell, and therefore increasing the cell area to 0.4 m^2, would allow for a greater amount of recovery. The cell also indicates that the power consumption for a cell running with low current density and voltage (as in 919-52) would have very little power consumption (0.5 kWh /day for the ED cell (including electrode rinse) but not including pumps etc.). These variables could be further optimized with continued testing.

The system footprint would be considerably smaller than the Braidwood system and could have a footprint of about 4 feet by 3 feet (4 feet tall), if a separate feed tank/pump is not used and solution is flowed directly from the sample line through the cell and back to the plant. The system would produce about 1-5 L/day of 0.25 M lithium hydroxide depending on the time within the RCS cycle.

4.2 Recovery of Lithium from Ion Exchange Beds.

Various methods of recovering lithium from the ion exchange beds were outlined in Section 2.2, with specific experiments outlined in Section 3.1.2. This section details the results of two different methods of removing lithium from the ion exchange media; regenerating the resin with boric acid or sulfuric acid.

4.2.1 Boric Acid Regeneration

In order to determine the effectiveness of using boric acid for removing lithium from a lithiated ion exchange bed, regeneration of the ion bed was performed with the following concentrations of boric acid.

- 3% Boric Acid at 20ºC
- 5.5% Boric Acid at 20ºC
- 8% Boric Acid at 50ºC (higher temperature was required to obtain this concentration of acid)

In each case, the bed was first lithiated by adding excess lithium hydroxide to the solution, rinsed with water until neutral, and then placed into the column. The boric acid solution was then added at a flow rate of 2 bed volumes per hour (BV/hr). Solution was collected for 30 minutes (1 BV), analyzed for lithium content and the results are shown in Figure 4-9. The higher the boric acid concentration, the higher the amount of lithium removed. However, even in the best case, only 0.22 g of the 3-4 g of lithium (~5%) that was placed on the bed was recovered.

Section 2.2.3 calculated that up to 4.7 lbs or 2.1 kg of lithium was lost on the Cation Bed. The above regeneration was found to recover about 5% of that on the bed or a possible 100 g total per ~500 day cycle. The solution recovered from the ion bed would have a lithium concentration of 75 mg/L Li in 8000 ppm B. As these concentrations are still higher than those processed in the above EDI tests, it would also be possible to process this solution to produce lithium hydroxide. However, it would only be capable of recovering a small amount of the lithium on the resin bed.

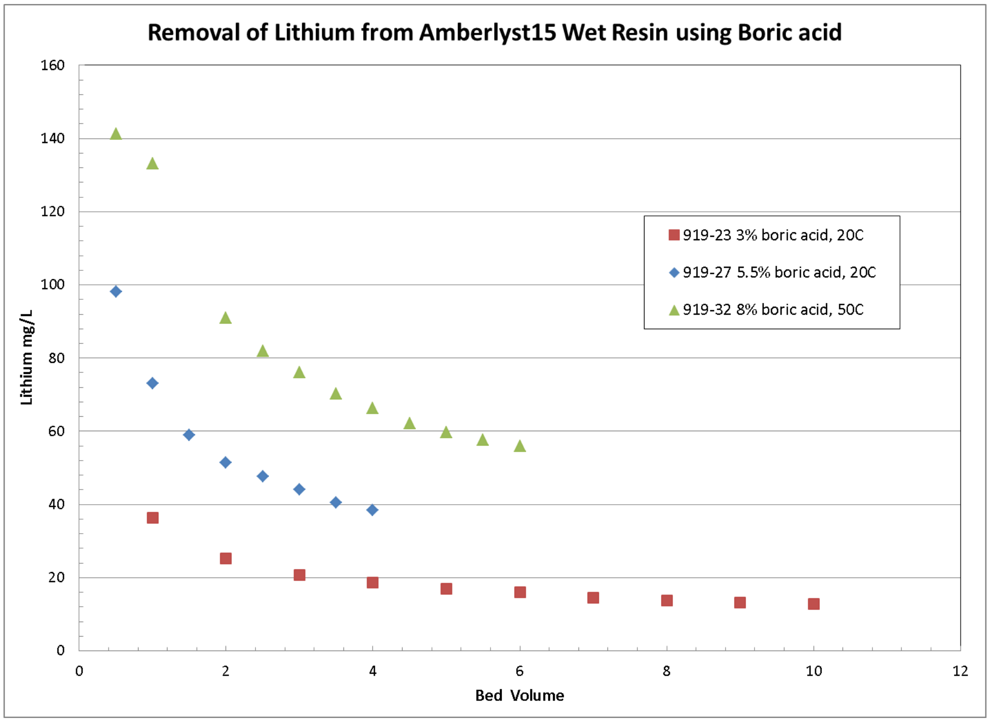


Figure 4-9
Concentration of Lithium Recovered with Boric Acid

4.2.2 Sulfuric Acid Regeneration

Because the boric acid regeneration was not very efficient, a similar experiment to those performed with boric acid was performed with 0.5 M sulfuric acid (1 N). In order to increase the overall concentration of lithium in the regenerate, the resin was regenerated at a slow flow rate of 0.5 BV/hr. Again samples were taken periodically and measured for lithium, and these are shown in Figure 4-10.

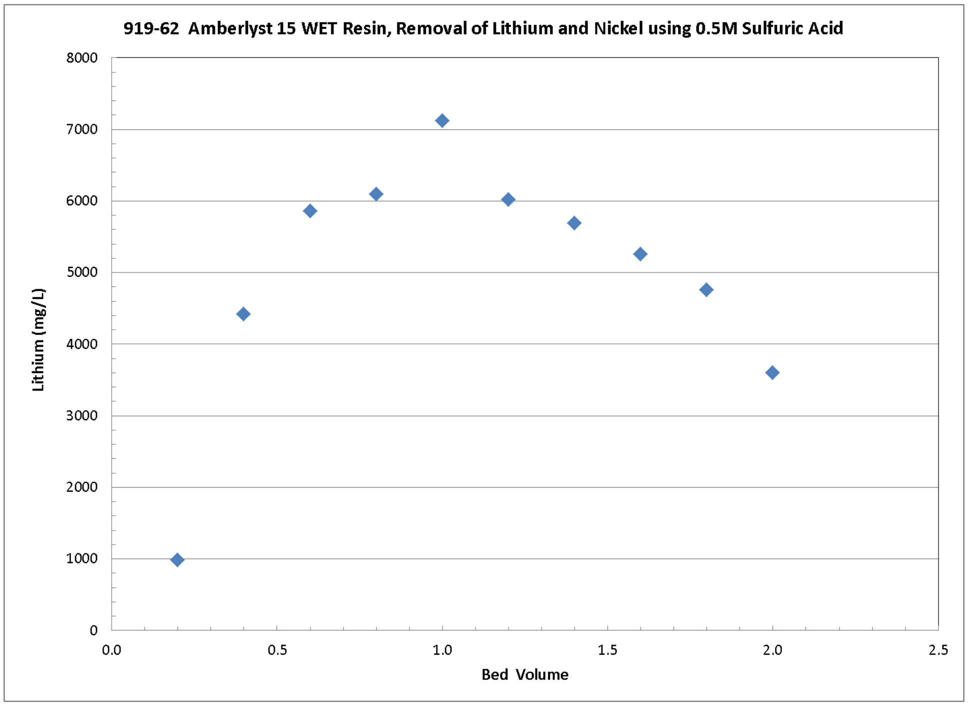


Figure 4-10
Concentration of Lithium Recovered with Sulfuric Acid

Combination of the individual samples collected from the regeneration recovered over 5 g of lithium in the one liter volume of acid solution. This is close to 100% recovery of the amount estimated to have been loaded on the column and is 85% of the capacity of the resin. The solution used to load the column also included 5.4 mg of nickel. The solution after absorption was found to not contain nickel, indicating that the full amount was on the resin. The sulfuric acid regeneration solution was analyzed at 1.3 ppm nickel indicating that about 76% of the nickel stayed on the column which can then be disposed with spent resin. The regeneration could also be performed with hydrochloric acid, but its use would also remove other divalent cations (nickel, cobalt etc.), which would be better left on the resin for disposal/removal from the system.

4.2.3 Electrodialysis of Lithium Sulfate Solution from IX Regeneration

The solution recovered from the ion exchange resin regeneration was processed in a three-compartment bipolar membrane electrodialysis cell as shown in Figure 4-11. This cell is a more traditional bipolar electrodialysis (BPED) cell consisting of bipolar, cation and anion membranes. BP-1E and CMB (Astom, Japan) were again used along with a proton blocking anion membrane AAV (Asahi Glass, Japan), which is designed to allow higher acid concentration by decreasing the amount of proton back migration from the acid compartment back into the feed. Because of the high concentration of lithium in solution, resin is not required in this cell.

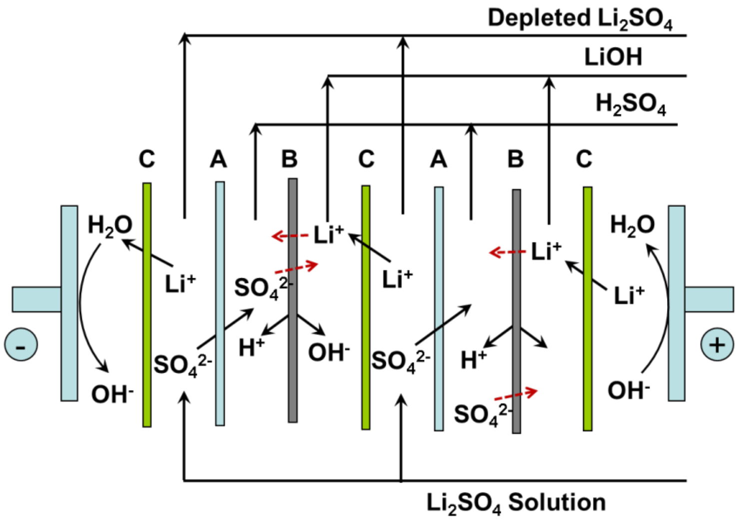


Figure 4-11
Schematic of a 3-Compartment Bipolar Membrane ED for Salt Splitting Lithium Sulfate

The electrodialysis experiment (details of the experiment are given in Appendix 5C) on the IX regenerate stream proceeded well, recovering 99% of the lithium from the feed. 900 mL of 5.0 g/L (0.72 M) lithium sulfate (small amount of bisulfate) was processed into 1 L of 4.4 g/L (0.64 M) lithium hydroxide and 1.5 L of 0.44 M H_2SO_4. As shown in Figure 4-12, the current peaked at 50 mA/cm^2, then dropped considerably as the lithium sulfate concentration decreased to less than 10 mM by the end of the batch as shown in Figure 4-13. The average current density for the batch was 38 mA/cm^2 with 66% current efficiency for lithium removal. However, higher current densities could be obtained if the batch was stopped earlier providing slightly lower recovery of lithium.

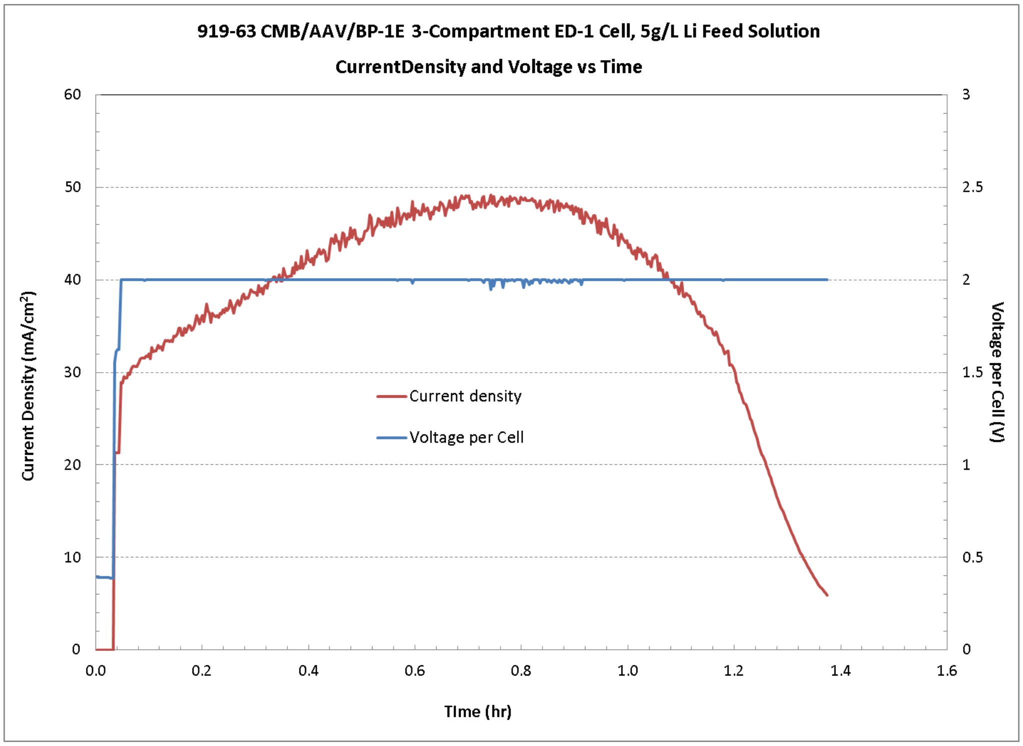


Figure 4-12
Current Density and Voltage per Cell for 3-Compartment BPED with lithium sulfate

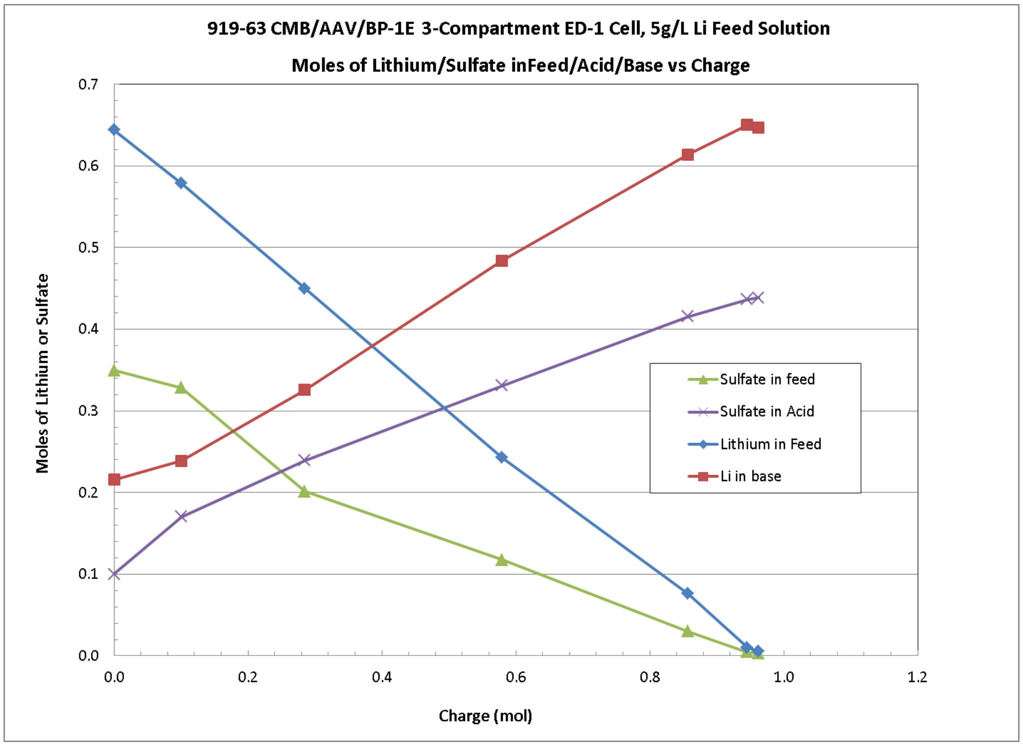


Figure 4-13
Moles of Lithium/Sulfate in the Feed, Acid and Base

The bipolar membrane is not 100% efficient, and does not block lithium as well as other ions. The sulfuric acid was found to contain 665 mg/L (~0.1 M) of lithium, which is not a problem as the acid solution is returned to another ion exchange bed to perform regeneration and recover lithium. Therefore, the lithium is not lost but is a load in the recycle stream. The amount of sulfate that leaks through the bipolar membrane is much smaller (due to its size and divalent charge). The 4.4 g/L Li lithium hydroxide was found to contain 24 ppm sulfate. If this solution was used for the start of an RCS operating cycle, and was diluted to 6 ppm Li, then the process solution would contain 30 ppb sulfate. Additional processing, such as ion exchange, can be performed on the lithium hydroxide product solution to achieve the required sulfate specifications established by utilities participating in such a program.

The cation bed will typically contain 3 kg of Li over a RCS cycle. If the acid regeneration can recover 85% of the lithium, then this will produce about 500 L of 0.72 M lithium sulfate. In order to process this solution in a period of 8 hours, an ED cell of approximately 5 m^2 would be required (a smaller area would be needed if the solution was processed over a longer period of time). A total of 2.6 kg of Li in about 580 L of 0.64 M lithium hydroxide (4.4 g/L Li) would be produced containing about 24 ppm of sulfate. The power required for the ED cell would be about 35 kWh per batch (cell power only). The equipment required for 5 m^2 of cell area would be substantially larger than the system required for on-line recovery (0.4 m^2). However, this system would be able to recover about 60% of the lithium lost from a typical RCS cycle every day of operation and therefore would be able to process the lithium from multiple plants.

4.3 Electrodialysis of Lithiated Water

The electrodialysis cell configurations incorporating bipolar membranes, are capable of removing lithium from the feed stream producing lithium hydroxide. However, in some cases, it would also be beneficial to remove boron from the stream. As previously noted at low pH, the majority of the boron is present as a neutral acid molecule and would not be available for transport. However with lithium present (at higher pH's), an equivalent amount of $H_2BO_3^-$ anion is thought to be present and may transport through an anion membrane. The cell configuration used for this testing is shown in Figure 4-14 with Neosepta CMX and AMX (Astom, Japan) membranes incorporated in the cell along with ion exchange resin in the feed compartment.

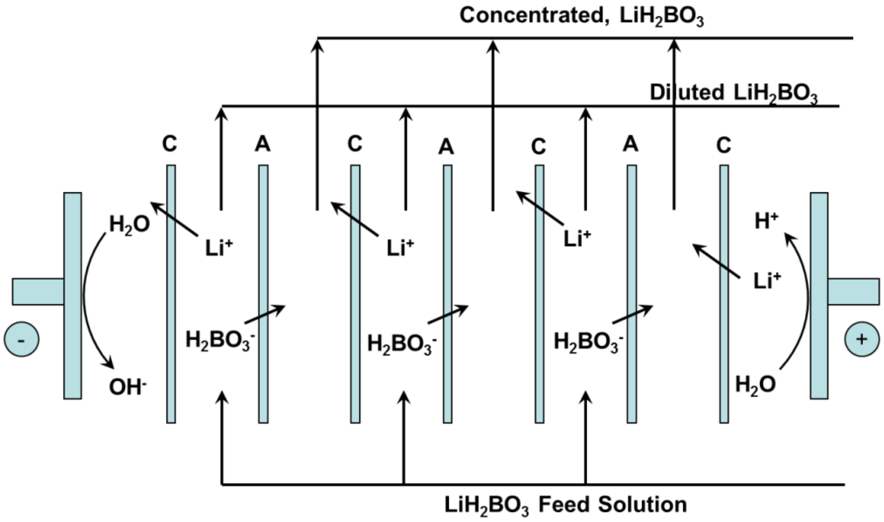


Figure 4-14
Two Compartment EDI cell for removal of lithium and boron

The test was performed with the higher concentration plant solution (3.5 ppm Li/1200 ppm B) incorporating an initial receiving/concentrate solution containing 0.1 M LiH_2BO_3. Although the test performed at low current density (0.33 mA/cm^2), it ran at very high current efficiency for both lithium and boron removal. The lithium current efficiency was 95% providing a decrease in lithium concentration from 3.5 ppm to 1.4 ppm. The boron removal corresponded to a current efficiency of around 250%, assuming transport of $H_2BO_3^-$. The higher than theoretical rate indicates that some neutral H_3BO_3 is likely transported through the membranes as well. When ions are transported through ion exchange membranes, some water of hydration is also transported with them, which can carry neutral molecules under osmotic drag. This is well known for electrodialysis of weak organic acids. In many cases, this transport of neutral molecules is undesirable, but in this case the extra transport benefits the boron removal. The high amount of water transport during this test limited the end concentration of the receiving compartment, producing a higher volume of solution containing 0.1 M Li (concentration unchanged) and 0.13 M borate.

5

RESULTS SUMMARY AND CONCLUSIONS

5.1 Comparison of Recovery Methods

The ability to use electrodialysis to recover lithium from the PWR cycle has been demonstrated via different methods, which are summarized in Table 5-1. Both the on-line recovery option (assuming a 2 L/min flow) and the recovery from the IX Bed with sulfuric acid recover about the same amount of total lithium per PWR cycle.

Table 5-1
Comparison of Recovery Methods

Method	Recovery Rate	Equipment	Cost, Power and Comments
On-Line Recovery	1-8 g /day ~2 kg / cycle	2-compartment EDI Cell, 0.4 m^2 Power Supply (30 W) Electrode Rinse and Base tanks, pumps and piping Conditioning Equipment (Cooling, Radiation Removal) Piping into sample line and return	ED cell 0.5 kWh/day Low power (110V) requirement ~ 4 ft by 3 ft by 4 ft Provides 0.25 M LiOH at plant (with low level B)
Sulfuric Acid Regeneration	2.5-3.0 kg/cycle Capable of treating a PWR plant per day	Rad treatment of resin IX regen. 3-compartment BPED Cell, 5 m^2 Power Supply (4 kW) Feed, Base, Acid and ER tanks, pumps and piping Cooling	ED cell 35 kWh/ 8 hours ~ 6 ft by 4 ft by 6 ft Provides 0.65 M LiOH Central site so that post treatment could be available (sulfate removal or further concentration)
Boric Acid Regeneration	150-200 g / PWR cycle (ED cell sized to treat in 1 day) Capable of treating a PWR plant per day	Similar to Sulfuric Acid Plant 2-Compartment BPED or BPEDI cell, 0.5 m^2 Power supply (300 W)	ED cell 5 kWh/ 8 hours ~ 4 ft by 4 ft by 6 ft Provide between 0.3-0.65 M LiOH Central site so that post treatment could be available (borate removal or further concentration)

The on-line recovery occurs over the full 500 days removing a greater amount of lithium at the beginning of the cycle. The almost 2 kg of lithium is recovered on-site over the 1.5 year period as a 0.25 M lithium hydroxide with about 160 ppm boron. The power requirement (110 V would be suitable) and footprint is small.

The recovery from ion exchange allows for a more rapid recovery of the lithium and recovers about 50% more lithium. A central processing plant could process the ion exchange bed capacity from multiple plants (one per day) providing much larger quantities of about 3 kg/day (dependent on the number of plants). The footprint is still small, possibly allowing this to be placed on an existing site. The power requirements of 4 kW could be easily achieved with 230 V service. The lithium hydroxide in this study was produced as 0.65 M lithium hydroxide with 24 ppm sulfate. The concentration of hydroxide produced from this type of cell could be increased to 2 M with appropriate use of volumes/cell area. The sulfate content would still be expected to be proportional. A central site could also be used for post treatment of the lithium hydroxide if sulfate removal is required. High purity material could be recovered by evaporation and crystallization of lithium hydroxide monohydrate.

Boric acid could also be used, but this treatment would need to be performed off-line, and would only produce 150-200 g of lithium per PWR cycle. A large amount of the lithium would be wasted. If performed in a central facility, a small plant could process this amount of lithium each day, thereby supporting multiple plants.

The tests incorporating an EDI cell with cation and anion exchange membranes showed that it was possible to remove both lithium and borate from plant solution, recovering a more concentrated lithium borate solution. Since bipolar membranes are considerably more expensive than anion membranes, it may be beneficial to use the process shown in Figure 4-14 to remove both lithium and boron from the system, and then performed bipolar ED as in Figure 3-1 on the more concentrated solution producing separate streams of lithium hydroxide and boric acid. More work is required on both systems to determine the optimum configuration.

Based on some of the work already demonstrated, if a separate process to remove only borate is required a cell similar to Figure 3-1 could be used incorporating only bipolar and anion membranes and anion exchange resin. The feed solution would be fed to the hydroxide producing side of the bipolar membrane, and borate would be transported through the anion membrane producing a stream of boric acid.

The test work provided has shown that lithium and borate recovery is feasible, further test work is required to optimize the processes, which can further decrease membrane area, power cost and/or increase the concentration and purity of the lithium hydroxide produced. Other membranes from alternate suppliers should also be evaluated as well as membranes manufactured in the United States. This work focused on a strong-acid cation exchange resin for use in the EDI cell and the ion exchange bed. A weak-acid resin may work better in both the EDI cell and for the boric acid regeneration.

The project team has also demonstrated that borate can be removed and is transported through the anion membrane of the ED cell as long as the pH of the solution is high enough that some of the borate is present as $H_2BO_3^-$. Further work is required to demonstrate whether this can be used to remove the required borate in order to remove the requirement for large amount of dilution in the PWR process.

5.2 Conclusions

The following conclusions have been made based on the results of this work.

- PWRs that apply BTRS systems with adequate sample line flow (~3 liters/minute) may recover enough ^{7}Li to sustain operations for that unit without an outside source using on-line purification. The studied PWR uses 3-4 pounds (1.4-1.8 kg) of ^{7}Li between lithiating resin and startup. A CEDI used on-line recovers 4.4 lbs (2 kgs) of ^{7}Li per cycle.
- To supply ^{7}Li for the United States PWR reactors using on-line purification, all plants would need to use an on-line CEDI in order to recover their lithium in the event that the supply of ^{7}Li is interrupted (i.e. one plant cannot supply enough lithium for additional plants). This is a limitation of the small flow rate in the RCS sample line.
- Initial research has indicated that not all plants have the sample system capability to recover sufficient lithium, therefore a recovery strategy that is not on-line would be required. Cation bed regeneration followed by lithium separation would be sufficient for off-line lithium recovery.
- Cation bed and mixed bed resin regeneration can recover the needed ^{7}Li per cycle.

Based upon these conclusions, while it would be expensive and inconvenient, it is technically feasible for the United States nuclear industry to be independent of an outside source for ^{7}Li if the supply were interrupted.

5.3 Recommendations

Based on the results of this project, it appears feasible to recover sufficient ^{7}Li to sustain PWR fleet operation in the event of a supply chain interruption. This outcome warrants additional study to further refine the ^{7}Li recovery model, determine the number of plants that would be required to participate, and estimate the cost of implementation. The following are recommendations for future work:

- Examine the effect of various types of mid-cycle outages (e.g. mode 3, mode 5) and associated water management impacts on model sustainability.
- The use of certain types of burnable poisons requires a boron and lithium program that differs somewhat from the linear program considered in this evaluation. Expand the model to include these burnable poison scenarios.
- Examine lithium recovery from mixed bed resin in addition to cation resin and the effect of this additional source of ^{7}Li on sustainability.
- For off-line regeneration processes, experimentally confirm the post-regeneration lithium hydroxide purification options.
- Determine the cost of implementation for both on-line separation in individual plants and a centralized facility to recover ^{7}Li from resin.
- Develop a national model to determine how many plants would have to participate in this program in order to generate enough ^{7}Li to supply the entire US PWR fleet.

A
DERIVATION OF THE LITHIUM ON MIXED BED RESIN

Lithium ions on the resin can be replaced by hydrogen ions, and vice versa.

$$H^+ + R - Li \rightleftharpoons Li^+ + R - H$$

The equilibrium constant of this equation is:

$$K_{eq} = \frac{[Li^+] * [R - H]}{[H^+] * [R - Li]}$$

Important to note is that typically [R-Li]+[R-H] = 1.7 eq/L.

Combining these two equations gives:

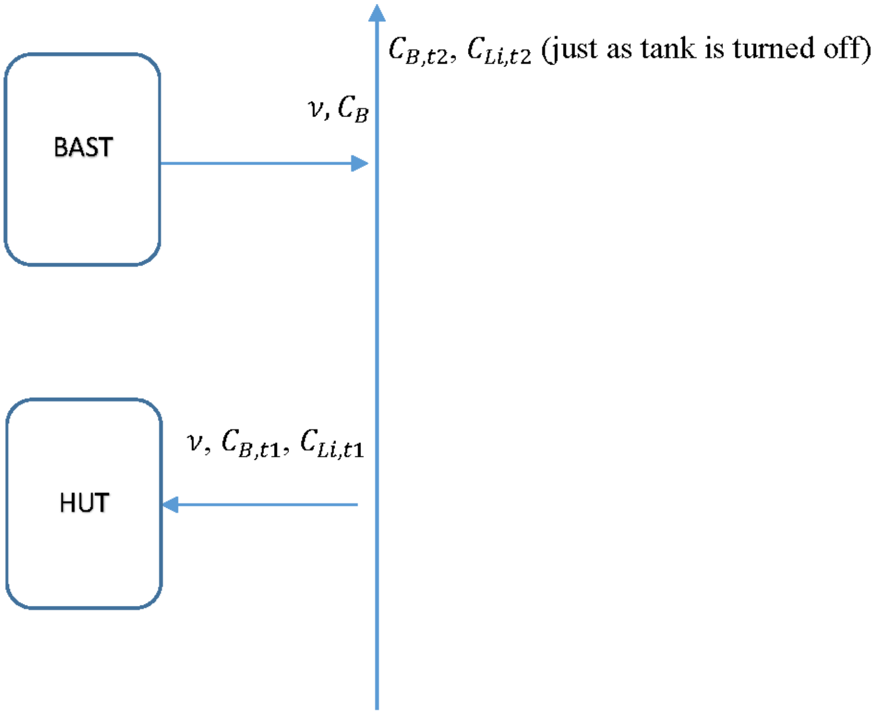
$$[R - Li] = \frac{1.7 * Li}{K_{eq} * H + Li}$$

K_{eq} is typically 1.1, which is the value used in the spreadsheet. Hydrogen can be calculated from pH by:

$$[H^+] = 10^{-pH}$$

And the lithium value is known. By subtracting yesterday's [R-Li] from today's [R-Li], the change in lithium in the system due to the mixed bed can be determined. If this number is negative, lithium was taken from the system and put onto the bed. If the number is positive, lithium was taken from the bed and put into the system.

B
DERIVATION OF HUTS EQUATION 6



Water is taken out of the system and put into the HUT. The water that was lost is replaced by borated water. The equation below shows the mass balance around boron:

$$\frac{V}{v} * \frac{dC}{dt} = C_B - C$$

When solved from time 0 to t and from $C_{B,t1}$ to $C_{B,t2}$, the following equation describes the relationship between time and concentration of boron:

$$\upsilon * \mathrm{t} = \ln\left(\frac{C_B - C_{B,t1}}{C_B - C_{B,t}}\right) * V$$

$\upsilon * \mathrm{t}$ is the total amount of water lost to the HUT. To find the lithium lost, this needs to be multiplied by the concentration of lithium. An important assumption is the while borating/diluting, the change in lithium concentration is linear. Therefore, the average of the starting lithium and final lithium concentrations are used. This is shown below:

$$Li\ Lost = \frac{C_{Li,t1} + C_{Li,t2}}{2} * \ln\left(\frac{C_B - C_{B,t1}}{C_B - C_{B,t}}\right) * V$$

The situation described above is during a boration period. This is when strong concentration of boron are entering the system. However, during dilution, pure water is coming in. In this case, C_B = 0.

C
EXPERIMENTAL DESCRIPTION AND DATA FOR ELECTRODIALYSIS EXPERIMENTS

Electrodialysis experiments were performed on synthetic solution produced from ACS grade lithium hydroxide and boric acid. Electrodialysis cells used for this testing were the ED1 (two compartment) and ED1-BP (three-compartment) cells (Electrosynthesis Company, USA). These ED cells have 100 cm^2 membrane area per cell, and for all experiments a total of 5 repeat units were used providing for an effective membrane area of 500 cm^2. All runs were conducted at 40 °C. Membranes used in this testing were all manufactured by Astom (Japan), except for the AAV membrane which is made by Asahi Glass (Japan). The electrode rinse used in all experiments was 0.1 M LiOH.

The base/receiving/concentrate and the electrode rinse were run in batch mode, i.e. solutions were continuously recirculated through the cell. The electrode rinse solutions from the cathode and anode compartments are recombined such that the net reaction is the splitting of water into hydrogen and oxygen. (Note that in a commercial version of this operation, the streams would need to be safely de-gassed before recombining and typically water is added). Depending on the experiment, the feed solution was sometimes circulated in batch mode, or as a single pass.

In an experimental cell, the cell voltage required for the electrode reactions and potential drop in electrode rinse solution is large in comparison to the voltage drop across the working cells. It therefore needs to be accounted for in experimental trials. However, in commercial cells (with hundreds of cells between end electrodes), this component becomes a minor contribution to the overall cell voltage. The laboratory cell was therefore built with platinum wires positioned in the base compartments closest to the electrode rinse, so that the voltage drop across the working cells could be measured. Most of the experiments were run by restricting the voltage across the platinum wires such that the voltage per cell was equal to a maximum of 1.2 V for a biolar membrane and 0.4 V for a cation or anion membrane. Therefore most of the two-compartment bipolar tests were performed at 1.6 V/cell (1.2 V for the bipolar and 0.4 V for the cation), and the three-compartment bipolar ED was performed at 2.0 V /cell. The two compartment EDI with cation and anion membranes was limited to 0.8 V/cell. In one experiment (919-52), the current density was limited to 1 mA/cm^2 and the voltage allowed to vary.

Samples of each compartment were taken periodically throughout the run and analyzed for lithium, borate, hydroxide and/or proton. The lithium was analyzed using cation ion exchange chromatography and hydroxide/proton by simple acid/base titration. Borate was analyzed by conversion to a strong acid by complexation with mannitol and titrating with hydroxide.

Table C-1 provides a set of all measured parameters for the various reported experiments.

Table C-1
Results for Various ED Tests

Test	Test time	Start feed conc.	End feed conc.	Start OH Conc	End OH conc	Feed Volume Start/End	Base Volume Start/End
	min	Li / B (ppm)	Li / B (ppm)	M	M	L	L
Bipolar/Cation/Resin: 2- Compartment EDI							
Single Pass / Variable Flow							
919-56	75.0	0.68 / 119	0.17 / 119	0.2	0.24	33.0/33.2	1.00/1.02
919-45	75.0	1.38 / 422	0.33 / 433	0.2	0.25	34.5/34.5	1.00/1.03
919-40	62.0	2.5 / 1179	0.96 / 1179	0.2	0.25	33.8/33.9	1.00/0.97
919-48	72.0	3.1 / 1179	0.81 / 1179	0.2	0.25	33.7/33.8	1.00/1.05
919-52	71.0	3.6 / 1158	1.19 / 1169	0.2	0.19	33.5/33.5	1.00/1.04
Batch Operation							
919-35	210.0	3.6 / 1179	0.37 / 1179	0.2	0.30	35.8/35.7	1.00/1.03
3-compartyment BPED (regeneration with sulfuric acid)							
919-63	80	5024 / 0	57 / 0	0.2	0.62	0.89/0.71	0.85/0.9
2-Compartment EDI: Cation/Anion/Resin (Removal of Lithium and Boron)							
				Start Rec. Li / B (ppm)	**Start Rec. Li / B (ppm)**		
919-68	69	3.5 / 1271	1.4 / 1244	730 / 1170	680 / 1330	35/34.7	0.85/0.92

Table C-1 (continued)
Results for Various ED Tests

Test	Charge voltage	Ave. current density	End Feed pH	End Feed Conductivity	Current Efficiency Li removal
	V / cell	mA/cm^2		µS/cm	%
Bipolar/Cation/Resin: 2- Compartment EDI					
Single Pass / Variable Flow					
919-56	1.6	6.5	6.20	1.20	1.8
919-45	1.6	8.5	5.90	1.70	2.6
919-40	1.6	10.7	5.54	4.00	5.1
919-48	1.6	8.2	5.40	3.80	6.0
919-52	0.8	1.0	5.85	--	52.0
Batch Operation					
919-35	1.6	10.4	5.04	2.30	3.2
3-compartyment BPED (regeneration with sulfuric acid)					
919-63	2.0	38.5	3.1	1.5 mS/cm	66
2-Compartment EDI: Cation/Anion/Resin (Removal of Lithium and Boron)					
919-68	0.8	0.33	6.0	4.8	95

D

LITHIUM GENERATED FROM BORON

Table D-1 shows several examples of how the concentration of boron, the thermal flux, and the fast flux affect the production of lithium. Note that the production of ^{7}Li at 1,000 ppm boron is about 0.15 ppm per day based on operating experiences at many PWRs.

Table D-1
Lithium Production from Boron

B, ppm	^{10}B Fraction	Thermal Flux n/cm^2-sec	Atoms ^{7}Li/L-s Produced from Thermal Flux	Atoms ^{7}Li/L-d	ppm ^{7}Li/day	Fast Flux, n/cm^2-sec	Atoms ^{7}Li/L-s Produced from Fast Flux	Atoms ^{7}Li/L-d	Ppm ^{7}Li/day	Total ^{7}Li ppm Produced/day
1E3	1.99E-1	5E13	2.3E15	1.99E20	2.31E0	5E12	1.04E14	8.95E18	1.04E-1	2.41E0
1E3	1.99E-1	1E13	4.6E14	3.97E19	4.62E-1	1E12	2.07E13	1.79E18	2.08E-2	4.83E-1
1E3	1.99E-1	6E12	2.76E14	2.38E19	2.77E-1	5E11	1.04E13	8.95E17	1.04E-2	2.88E-1
1E3	1.99E-1	5E12	2.3E14	1.99E19	2.31E-1	5E11	1.04E13	8.95E17	1.04E-2	2.41E-1
1E3	1.99E-1	4E12	1.84E14	1.59E19	1.85E-1	5E11	1.04E13	8.95E17	1.04E-2	1.95E-1
1E3	1.99E-1	3E12	1.38E14	1.19E19	1.39E-1	5E11	1.04E13	8.95E17	1.04E-2	1.49E-1
1E3	1.99E-1	2E12	9.2E13	7.95E18	9.24E-2	5E11	1.04E13	8.95E17	1.04E-2	1.03E-1
1E3	1.99E-1	1E12	4.6E13	3.97E18	4.62E-2	1E10	2.07E11	1.79E16	2.08E-4	4.64E-2
1.5E3	1.99E-1	5E13	3.45E15	2.98E20	3.47E0	5E12	1.55E14	1.34E19	1.56E-1	3.62E0
1.5E3	1.99E-1	1E13	6.9E14	5.96E19	6.93E-1	1E12	3.11E13	2.69E18	3.12E-2	7.24E-1
1.5E3	1.99E-1	5E12	3.45E14	2.98E19	3.47E-1	5E11	1.55E13	1.34E18	1.56E-2	3.62E-1
1.5E3	1.99E-1	1E12	6.9E13	5.96E18	6.93E-2	1E10	3.11E11	2.69E16	3.12E-4	6.96E-2
5E1	1.99E-1	5E13	1.15E14	9.94E18	1.16E-1	5E12	5.18E12	4.48E17	5.21E-3	1.21E-1
5E1	1.99E-1	1E13	2.3E13	1.99E18	2.31E-2	1E12	1.04E12	8.95E16	1.04E-3	2.41E-2
5E1	1.99E-1	5E12	1.15E13	9.94E17	1.16E-2	5E11	5.18E11	4.48E16	5.21E-4	1.21E-2
5E1	1.99E-1	1E12	2.3E12	1.99E17	2.31E-3	1E10	1.04E10	8.95E14	1.04E-5	2.32E-3

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