# IDENTIFICATION AND TREATMENT OF LITHIUM AS THE PRIMARY TOXICANT IN A GROUNDWATER TREATMENT FACILITY EFFLUENT

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#### **ABSTRACT**

A stable isotope of lithium (<sup>6</sup>Li) is used in manufacturing nuclear weapons, nuclear shielding and nuclear reactor control rods. Lithium compounds have been utilized at Department of Energy facilities and Li-contaminated waste has historically been land disposed. Seep water from burial grounds near the Oak Ridge Y-12 plant contain small concentrations of chlorinated hydrocarbons, trace amounts of polychlorinated biphenyls, and Li at concentrations of 10 to 19 mg/L. Treatment of the seep water consists of oil-water separation, filtration, air stripping to remove volatile organic compounds and carbon adsorption to remove non-volatile organics.

Routine biomonitoring tests using fathead minnows (*Pimephales promelas*) and *Ceriodaphnia dubia* are conducted for the facility. The no-observed-effect concentration (NOECs) for these species ranged from <1 to 12%. An evaluation of suspected contaminants revealed that toxicity was most likely due to Li. Laboratory tests showed that a concentration of 1 mg Li/L reduced the survival of both species; 0.5 mg Li/L reduced *Ceriodaphnia* reproduction and minnow growth. However, the toxicity was greatly reduced in the presence of sodium (Na). At concentrations up to 4 mg Li/L, Na can fully negate the toxic effect of Li.

Because of the high concentration of Li and low concentration of Na discharged from the treatment facility, selective removal of Li from the groundwater was desired. Columns packed with SuperLig® material (developed by IBC Advanced Technologies, Inc.) were used to evaluate a process for Li removal. The SuperLig® material is composed of a highly lithium-selective organic macrocycle permanently and covalently bonded to silica gel. Bench-scale tests with groundwater demonstrated that the SuperLig® material was very effective at removing Li from the groundwater effluent, but the high selectivity of the material for Li could not be confirmed due to concurrent removal of calcium and other cations by the silica support. Removing the Li from the effluent greatly reduced the toxicity: the NOEC for *Ceriodaphnia* in the nontreated effluent was 3% and in the treated effluent was 50%.

Results of this work are important because a compound that was largely unrecognized as being highly toxic to aquatic biota was identified. Toxicity of the effluent was correlated with Li concentration and the mitigating effects of Na on Li toxicity were defined. In addition, a treatment option for selective removal of Li was identified which successfully removed Li and thus reduced the toxicity of the effluent.

Keywords: lithium, toxicity reduction evaluation, groundwater, Ceriodaphnia, fathead minnow

#### INTRODUCTION

Lithium (Li) is the lightest metal in its elemental form. Lithium has never been found as a pure element; instead it is always bound in stable minerals and salts (Bleiwas and Coffman 1986). Commercial production of Li in the United States began in 1925. Significant usage of Li did not occur until World War II during which Li was used for CO<sub>2</sub> absorption in submarines (as Li hydroxide monohydrate), to inflate emergency signal balloons (as Li hydride), and in high-temperature resistant greases (Comer 1978, Bleiwas and Coffman 1986,

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FMC 1992). In the late 1950s and early 1960s, uses for Li were developed in the air conditioning industry (as Li bromide) and in the synthetic rubber industry (butyllithium). By 1960 Li had come into demand for a wide variety of uses and was well established in the marketplace. In 1994, ceramics and glass production and aluminum smelters were the largest consumers of Li in the United States (Ober 1994). Other uses include synthetic rubber, pharmaceuticals, chemical manufacturing, lubricants, batteries, and air treatment. The consumption of Li in the United States during 1994 is shown in Fig. 1.

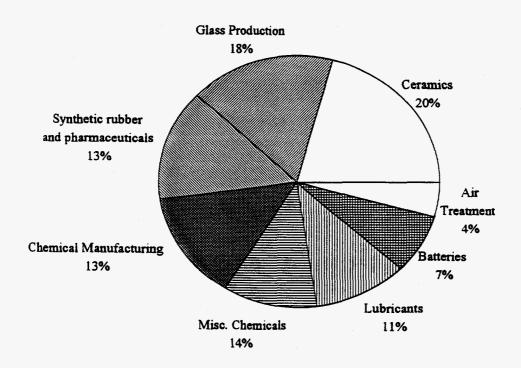


Figure 1. Consumption of lithium in the United States during 1993. From Ober 1994.

In the 1950s the Atomic Energy Commission purchased large amounts of Li hydroxide as feed material for a weapons production process at the Oak Ridge Y-12 Plant (DOE 1995b). From 1955 to 1963, Li hydroxide (LiOH) was used in the Columnar Exchange (COLEX) Process which separated the lithium-6 ( <sup>6</sup>Li) isotope from the lithium-7 ( <sup>7</sup>Li) isotope (DOE 1995b). The <sup>6</sup>Li-enriched stream was used for making lithium hydride and lithium deuteride materials for the weapons production program (DOE 1995a). During production, approximately 50 million kg of LiOH were involved in the program. Much of the lithium-contaminated waste generated in the past was land disposed. One such land disposal unit at the Oak Ridge Y-12 Plant was the Bear Creek Burial Grounds (BCBG). Remediation of the BCBG has included capping and the collection and treatment of contaminated seeps. The seep water contains small concentrations of chlorinated hydrocarbons, trace amounts of oil contaminated with polychlorinated biphenyls (PCBs), and Li at concentrations of 10 to 19 mg/L. Approximately 2.5 million gallons of groundwater are collected and treated annually.

Preliminary treatment where the seep water is collected consists of oil-water separation and filtration; the water is then transferred by tanker to the Groundwater Treatment Facility (GWTF; Fig. 2). The two primary unit operations of the GWTF are air stripping for removal of volatile organic compounds and carbon adsorption for removal of non-volatile organic compounds, primarily PCBs. A chemical feed system is used to precipitate

and remove iron when necessary. Treated effluent is discharged through a National Pollutant Discharge Elimination System (NPDES) monitoring station to East Fork Poplar Creek. Lithium is not removed from the water by this treatment system.

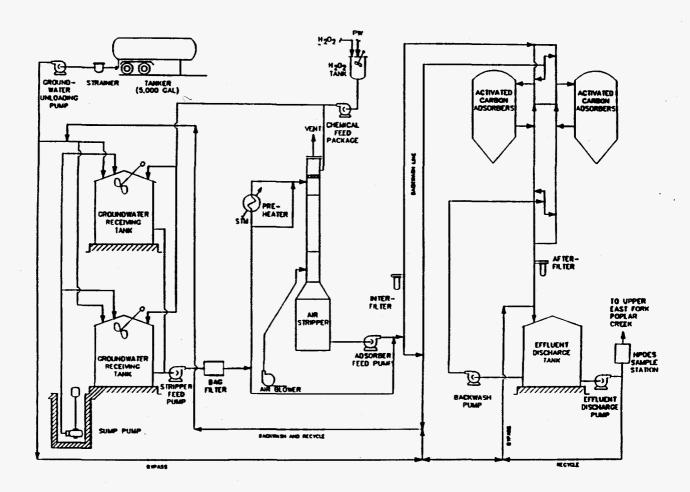


Figure 2. Groundwater Treatment Facility Process Flow Schematic.

Routine effluent biomonitoring tests using fathead minnows (*Pimephales promelas*) and *Ceriodaphnia dubia* have been conducted for the GWTF since December 1991. The no-observed-effect concentration(NOEC) for *Ceriodaphnia* was routinely 25% and for fathead minnows 100% until additional seeps were routed to the collection system for the treatment facility. After these seeps were added, the NOECs for *Ceriodaphnia* and fathead minnows ranged from <1 to 12%. An evaluation of suspected contaminants revealed that the increased toxicity (lower NOEC) coincided with an increase in Li from approximately 0.2 to 15 mg/L. Because there is very little information published about Li toxicity, an investigation was performed on the toxicity of Li.

The objectives of this work were to investigate the toxicity of Li at low concentrations to *Ceriodaphnia dubia* and fathead minnows and to confirm Li as the primary toxicant in the groundwater treatment facility effluent. Two additional objectives were to demonstrate (1) a process for selective removal of the Li from the

groundwater and (2) removal of Li resulted in lower toxicity. No commercially available ion exchange resin was found that would provide selective removal of the Li over other cations present in the water. One company, IBC Advanced Technologies, Inc. (IBC), had developed a product they claimed to be very highly selective for Li. Preliminary tests by IBC indicated that one of their SuperLig® materials would effectively remove Li from water simulating the GWTF effluent. A contract was approved for IBC to prepare a larger quantity of the SuperLig® material, perform additional tests with simulated groundwater, and then ship the test columns to the Y-12 Plant for testing with actual GWTF effluent.

#### **METHODOLOGY**

### **Toxicity Tests**

The toxicity of GWTF effluent and Li to early-life-stage fathead minnows (*Pimephales promelas*) and *Ceriodaphnia dubia* were evaluated in seven day or three brood static-renewal tests. Static renewal toxicity tests were conducted using methods similar to those described by the Environmental Protection Agency (EPA; 1994): method 1000.0, Fathead minnow, *Pimephales promelas*, larval survival and growth test and method 1002.0, and Daphnid, *Ceriodaphnia dubia*, survival and reproduction test (EPA 1994). Hereinafter these tests will be referred to as the fathead minnow test and *Ceriodaphnia* test. All fish were obtained from Aquatic BioSysems, Inc., Fort Collins, Colorado. The larvae were shipped overnight and were no more than 48 h old at the time of test initiation. Upon arrival, the fish were aerated and fed approximately 400 µL of brine shrimp (*Artemia salina*). The larvae were then acclimated within 1-5 h to 25±1° C in a temperature controlled water bath. *Ceriodaphnia* were cultured in the Toxicology Laboratory of the Environmental Sciences Division at Oak Ridge National Laboratory. Culture water for *Ceriodaphnia* and control water for both species was 25% DMW (dilute mineral water) consisting of 1 part Perrier water to 3 parts deionized distilled water amended with a low concentration of trace metals.. Water temperature was maintained at 25±1° C. *Ceriodaphnia* were fed a mixture of yeast, Cerophyl<sup>®</sup>, and trout chow (YCT) augmented with the green alga *Selenastrum capricornutum*.

Each fathead minnow test was initiated by putting 10 larvae in each of four replicate 600-mL beakers containing 250 mL test solution. Each *Ceriodaphnia* test was initiated by putting 10 neonates that were <24-h old in 10 replicate 30-mL beakers containing a minimum of 15 mL test solution. Control treatments, consisting of four or ten replicate beakers, each containing 25%DMW were included with each test. During the period of testing, the control water had an average pH of 7.94, conductivity of 208.3 μS/cm, alkalinity of 8.35 mg/L as CaCO<sub>3</sub>, and hardness of 97.38 mg/L as CaCO<sub>3</sub>. The larvae were fed newly hatched brine shrimp (*Artemia salina*) twice daily. *Ceriodaphnia* were fed 100 μL of YCT and 100 μL of *S. capricornutum* daily. The water temperature was maintained at 25±1° C and the photoperiod was 16 light: 8 dark. Mortality and reproduction (for *Ceriodaphnia*) was recorded every 24 h at daily renewal for 6 or 7 d. Fish growth was computed as dry weight at the end of 7 d test period. Reproduction was calculated as offspring/surviving female.

GWTF effluent grab samples and treated samples were collected and held at 4°C for <36 h before first use. Subsamples were removed daily and warmed to  $25 \pm 1^{\circ}$  C. Lithium and sodium test solutions were prepared by using stock solutions of Li (as LiCl), sodium (as NaCl or Na<sub>2</sub>SO<sub>4</sub>), and 25% DMW in polyethylene containers. Each concentration was prepared 24 h before test initiation and stored at approximately 4° C. A subsample of each test concentration was removed daily and warmed to  $25\pm 1^{\circ}$  for renewal. The conductivity of each dilution was measured on the first day of the test and pH, conductivity, alkalinity and hardness were assessed daily for the control water (all measurements were by standard EPA methods). Samples of each concentration were preserved with approximately 100 µL of nitric acid for analysis of Li and sodium by Inductively Coupled Plasma Spectroscopy (ICP). Target nominal Li concentrations were 1 mg Li/L (measured range = 0.92 to 1.3 mg/L), 2 mg Li/L (measured range = 1.7 to 2.1 mg/L), and 4 mg Li/L (measured range = 3.4 to 4.0 mg/L). For purposes of discussion, the test concentrations will be referred as the target nominal concentrations of 1, 2, and 4 mg Li/L. Target nominal sodium concentrations were 10 mg Na/L (measured range = 8.4 to 13 mg/L), 20 mg Na/L (measured range = 20 to 23 mg/L), 40 mg Na/L (measured range = 40 to 47 mg/L). For purposes of discussion, the test concentrations will be referred as (measured range = 150 to 170 mg/L). For purposes of discussion, the test concentrations will be referred as

the target nominal concentrations of 10, 20, 40, 80, and 160 mg Na/L.

#### Lithium Removal

Lithium-selective SuperLig® material was prepared by IBC using 35-60 mesh silica as the support. Approximately 210 g of the material was packed in each glass column. Two columns were used in series, each 5 cm in diameter and 25 cm in height. Glass wool pads supported the SuperLig® material at the top and bottom of each column. A peristaltic pump was used to feed the water being treated through the columns and to rinse and elute the columns, always in an upflow direction.

Three separate tests were performed with the same columns containing Li-selective SuperLig® material. The first two tests were conducted by IBC at their facilities using a simulated groundwater effluent. All solutions were prepared using reagent grade saits or acids and deionized water. Test columns were eluted with 0.5M or 1M HCl. The elution step was preceded and followed by flushing of the columns with deionized water. Feed solutions were kept below a pH of 10.5 to avoid degradation of the silica support. The lithium selective ligand was deprotonated with 0.05M K<sub>2</sub>CO<sub>3</sub>, adjusted with 1M HCl to a pH <10.5. For the second test by IBC and the test with actual groundwater the silica was preloaded with Ca<sup>2+</sup> using 0.1M CaCl<sub>2</sub>.

The third test was conducted at the treatment facility site using actual GWTF effluent. For this test, 20 L of 25% DMW at a pH of 8.6 was initially fed through the column to determine if there were any toxic compounds added from the column. About 21 L of GWTF effluent (adjusted to pH 8.8 with NaOH) were then processed through the two test columns. About 20 L of the treated GWTF effluent was then used for the toxicity test procedures described above. The columns were then fed deionized water to displace the remaining groundwater from the columns. This was followed by an acid elution step in which 1.1 L of 1 M HCl were pumped through the lead column. Feed rates for deionized water and acid were 40 mL/min. About 1 L of deionized water was used to flush the remaining acid from the columns. All elemental analyses for the Li removal tests with actual groundwater were performed using Inductively Coupled Plasma Emission Spectroscopy (ICP-ES).

#### **RESULTS**

#### **Toxicity Tests with Lithium and Sodium**

Routine biomonitoring tests of the GWTF effluent were conducted beginning in 1991 and the NOECs for *Ceriodaphnia* were routinely 25% and for fathead minnows were 100% until June 1993 when the NOECs for *Ceriodaphnia* and fathead minnows ranged from <1 to 12%. Coincident with the increased toxicity (lower NOECs), additional seeps were routed to the collection system for the GWTF and Li in the effluent increased from approximately 0.2 to 15 mg/L.

Results of the fathead minnow and *Ceriodaphnia* toxicity tests using lithium chloride demonstrated that the concentration of Li in the effluent was high enough to cause the observed effects. A concentration of 1 mg Li/L reduced the survival of both test species; 0.5 mg Li/L reduced *Ceriodaphnia* reproduction and minnow growth. The tests with *Ceriodaphnia* were repeated using the receiving stream water (EFPC) as the dilution water and the concentration of Li that was toxic to *Ceriodaphnia* increased: 1 mg Li/L did not reduce *Ceriodaphnia* survival or reproduction. Because the major difference between the two dilution water types appeared to be the sodium concentration, toxicity tests were conducted using combinations of LiCl and sodium sulfate. Results of these tests showed that reproduction of *Ceriodaphnia* exposed to 1 - 4 mg/L was not reduced when the molar ratio of Na:Li was≥2 (Fig. 3). Similar results were observed for fathead minnow survival and growth (Fig. 4). Short-term survival of *Ceriodaphnia* at concentrations of Li above 4 mg/L was mitigated by additional Na (Fig 5); additional Na did not mitigate the effects of Li on long-term survival or reproduction of *Ceriodaphnia*.

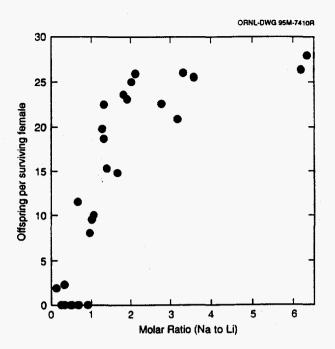


Figure 3. Reproduction of *Ceriodaphnia dubia* in response to Li and Na. Highest Li and Na concentrations tested were 4 mg Li/L and 48 mg Na/L.

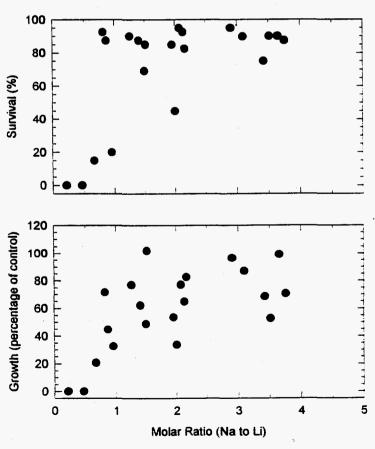


Figure 4. Survival and growth of fathead minnows in response to Li and Na. Highest Li and Na concentrations tested were 4 mg Li/L and 46 mg Na/L.

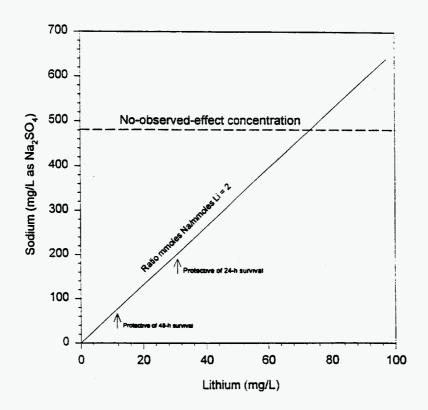


Figure 5. Levels of sodium that mitigate short-term mortality of Ceriodaphnia due to lithium.

# Lithium Removal from the Groundwater Treatment Facility Effluent

The composition of the three samples used to evaluate the SuperLig® material is shown in Table 1. The GWTF effluent used with the test columns had lower than average calcium and higher than average sodium but contained typical levels of Li when compared to typical GWTF effluent. Prior to Li removal testing, the columns were pH acclimated with 0.05 M potassium carbonate which served to deprotonate the ligand sites on the SuperLig® material; potassium also became bound to the silica support. In the first Li removal test performed by IBC, it was found that calcium was bound to the blank silica in significant quantities, displacing the potassium from the pH acclimation. This caused an increase in potassium and decrease in calcium concentration in the initial volume of treated water. To avert this shift in potassium and calcium concentrations during the Li removal step, a second test was performed in which 0.1M calcium chloride and water were pumped through the columns after the potassium carbonate acclimation. This removed K\* and preloaded Ca²\* onto the blank silica, resulting in minimal change of these elements in the water undergoing Li removal.

Table 1. Chemical Composition of Actual and Simulated Groundwater Effluent Samples

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Element	Average GWTF effluent concentration (mg/L)	GWTF effluent adjusted to pH 8.8 and used with test columns (mg/L)	Simulated effluent used with test columns at IBC Facilities (mg/L)
Li	15	12	18
Ca	100	49	98
Na	22	86	43
Mg	20	17	•
K	10	9	12
В	60	41	-

Lithium removal data for the first two tests, using simulated effluent, were nearly identical. Lithium was successfully removed in the columns at a feed rate of 120 mL/min, and the loading capacity was determined to be about 0.15 mmoles Li per gram of SuperLig® material.

For the test with actual groundwater an assumption was made that the loading capacity would be the same as for the simulated groundwater. Based on this loading rate, the test was stopped just prior to predicted Li breakthrough from the trailing column. Sample analyses later showed that Li breakthrough had already occurred in the trailing column; hence, the loading capacity was not as high during the test with actual groundwater effluent (Fig 6). The feed rate through the columns was maintained at 133 mL/min.

Outlet concentrations from the lead and trail columns were also measured for sodium, calcium and potassium (Fig 7). The concentrations of sodium and potassium remained relatively constant during the Li removal test. The calcium concentration decreased, indicating continued removal of calcium in the columns. Lithium and calcium were the primary cations removed from the columns by the elution step (Fig 8). About 1 L of deionized water was used to flush the remaining acid from the columns.

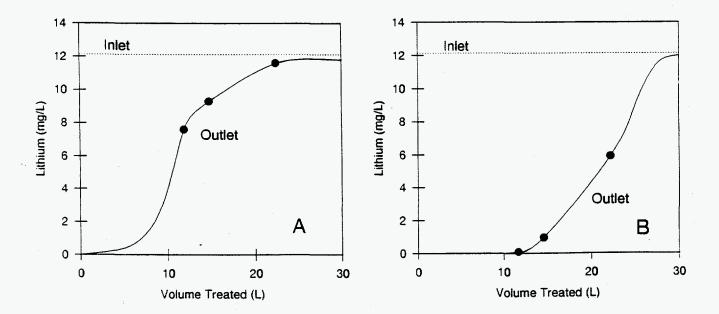


Figure 6. Lead column (A) and trail column (B) breakthrough curves for lithium removal from groundwater treatment facility effluent using SuperLig® material.

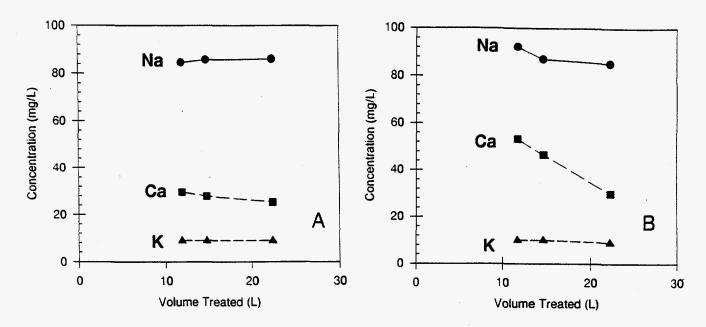


Figure 7. Lead column (A) and trail column (B) outlet concentrations for lithium removal from groundwater treatment facility effluent using SuperLig® material.

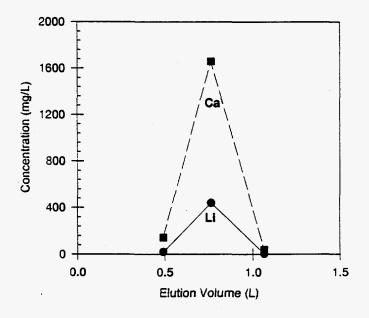


Figure 8. Lead column lithium and calcium elution curves from SuperLig® material using 1M HCl after loading.

# **Toxicity Assessment of Treated Groundwater**

Results of the toxicity assessment of the groundwater sample processed through the columns to remove Li are shown in Tables 2 and 3.

Table 2. Ceriodaphnia and fathead minnow survival in non-treated and resintreated Ground Water Treatment Facility effluent. NT = not tested.

	Ceriodaphnia survival		Fathead minnow survival	
Effluent Concentration	Non-treated	Treated	Non-Treated	Treated
Control	100	100	92.5	62.5
100	NT	0	NT	57.5
50	NT	100	NT	27.5
25	0	100	0	42.5
12	10	80	32.5	77.5
6	100	80	95.0	65.0
3	100	NT	77.5	NT
1	100	ŇT	92.5	NT

Table 3. Ceriodaphnia reproduction and fathead minnow growth in non-treated and resin treated GWTF effluent. NT = not tested.

	Ceriodaphnia reproduction (offspring per female; mean +SD)		Fathead minnow growth (mg/larvae; mean +SD)	
Effluent Concentration	Non-treated	Treated	Non-Treated	Treated
Control	26.6 ± 2.9	28.7± 3.0	0.51 ± 0.04	0.59 + 0.08
100	NT	0	NT	0.21 ± 0.03
50	NT	24.4 ± 3.3	NT	0.51 ± 0.06
25	0	27.9 ± 2.3		$0.52 \pm 0.04$
.12	3.7 ± 5.4	21.7 ± 10.9	$0.09 \pm 0.03$	0.61 ± 0.08
6	19.3 ± 4.4	22.8 ± 6.7	$0.47 \pm 0.03$	0.57 ± 0.03
3	26.0 ± 3.0	NT	$0.58 \pm 0.04$	NT

The removal of Li from the GWTF effluent clearly reduced the toxicity. The NOEC for *Ceriodaphnia* in the non-treated effluent was 3% compared to a NOEC of 50% in the treated effluent. Similarly, the NOEC for fathead minnows in the non-treated effluent was 6% compared to a NOEC of 12% in the treated effluent.

The reduced survival, growth and reproduction in the higher concentrations of effluent (treated and non-treated) was related to the Li concentration. Lithium in the full-strength resin treated GWTF effluent was 1.62 mg/L and in the 25% concentration of the non-treated GWTF effluent was 3.1 mg/L.

#### DISCUSSION

Successful identification of Li as the primary toxicant in effluent from the GWTF was the result of chemical analyses of the effluent combined with chemical-specific toxicity tests. It was also fortuitous that NPDES biomonitoring tests of GWTF effluent were ongoing when the new seeps containing Li were added. Lithium had previously been identified as toxic to freshwater organisms representing a salmonid habitat at similar levels to those identified in this study (Emery et al. 1981). The Emery et al. (1981) study was addressing the potential for increased levels of Li in the aquatic environment due to eventual development of fusion power reactors. We have identified that in this instance, the past use and disposal of Li has contaminated groundwater with Li at levels that are toxic to aquatic biota. Our finding that the toxicity of Li to *Ceriodaphnia* and fathead minnows is partially negated by sodium, indicates that in-stream effects will be modified by the ionic composition of water in the receiving stream.

The removal of Li from the GWTF was successful using the resin columns from IBC. The Li loading capacity for the test columns was higher with simulated groundwater than with actual groundwater effluent. As a result, breakthrough of Li occurred sooner than predicted for the actual groundwater, but this was not known until laboratory analyses were completed. Due to the early breakthrough of Li, the 20-liter outlet water sample used for toxicity evaluation contained 1.6 mg/L of Li. While this represented an 87% decrease from the inlet Li concentration, the Li level was still high enough to cause an observable toxic effect. An outlet sample that was collected after 11.7 L of water were processed had a Li concentration <0.08 mg/L, demonstrating that all detectable Li can be removed. The Li removal test with actual groundwater will be repeated using an on-line analyzer to ensure that the columns are regenerated prior to Li breakthrough. Toxicity tests will then be performed with non-treated and resin-treated GWTF effluent to determine if Li fully accounts for the observed toxicity.

Although the SuperLig® material proved to be very effective at removing Li from the groundwater effluent, the high selectivity of this material for only Li could not be confirmed due to concurrent removal of calcium and other cations by the silica support. The removal of cations by the silica support detracted from the Li removal process in the following ways. A larger volume of 0.05 M K<sub>2</sub>CO<sub>3</sub> was required for column acclimation, and CaCl<sub>2</sub> acclimation was required to pre-load the silica with Ca<sup>2+</sup>. Column elution removed Li from the SuperLig® material and calcium from the silica, necessitating a larger volume of eluting acid than for Li alone and generating a less pure eluent. These problems will be addressed by preparing the SuperLig® material on a more inert solid support such as polystyrene or polyacrylate.

#### CONCLUSIONS

Lithium was identified as the toxicant in effluent from the GWTF effluent. Laboratory tests showed that a concentration of 1 mg Li/L reduced the survival of *Ceriodaphnia* and fathead minnows; 0.5 mg Li/L reduced *Ceriodaphnia* reproduction and minnow growth. However, the toxicity was greatly reduced in the presence of sodium (Na). At concentrations up to 4 mg Li/L, Na can fully negate the toxic effect of Li.

Lithium was effectively removed from a groundwater effluent in two columns containing SuperLig® material. The toxicity of the groundwater effluent to *Ceriodaphnia* and fathead minnows was reduced significantly as a result of the Li removal step. Elution with 1M HCl readily stripped both Li and calcium from the test columns.

The use of silica as the solid support for the SuperLig® material has certain disadvantages. Because the silica removes calcium and some other cations from the groundwater effluent, it complicates the column acclimation and elution steps. The lithium removal step should be improved by preparing the SuperLig® material on a more inert solid support.

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