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Determination of Trace Lithium in Industrial Process Waters

INTRODUCTION

In a variety of situations, carryover from one body of water to another is measured by monitoring a tracer compound. Lithium is a good choice for a tracer because it is very soluble and has a low background concentration in public water supplies.¹ As a tracer, lithium has been used in a variety of diverse applications such as monitoring travel time of water between stream sites² and tracing water movement in activated sludge reactors.³

The current methodology to assay for lithium is time consuming and insensitive.⁴ There is a need for an improved analytical technique. This Application Update describes a rapid and sensitive ion chromatographic method for the determination of trace lithium using a microbore IonPac® CS12A column.

EQUIPMENT

Dionex DX-500 Ion Chromatography system consisting of:

GP40 Gradient Pump, microbore configuration

CD20 Conductivity Detector with a temperature-controlled conductivity cell (DS3)

LC20 Chromatography Enclosure, PEEK, with a second channel kit / Two Rheodyne valves, rear loading

Dionex DXP Single-Piston Pump

AC2 Power Control Accessory (P/N 046057)

4-L plastic bottle assemblies (two for external water mode)

PeakNet™ Chromatography Workstation

PEEK tubing (P/N 044777), 0.030-in. (0.75-mm) i.d., 60-ft (18.29-m) to construct a 7.5-mL loop

REAGENTS AND STANDARDS

Deionized water (DI H₂O), Type I reagent grade, 18 MΩ-cm resistance or better

Sodium chloride, reagent grade (J.T. Baker, Inc.)

Concentrated sulfuric acid, trace metal grade (J.T. Baker, Inc.)

Lithium chloride, reagent grade (J.T. Baker, Inc.)

Morpholine, 99%, reagent grade (Aldrich Chemical Co.)

Hydrazine, 100% hydrate, equivalent to 64% hydrazine, reagent grade (Sigma Chemical Co.)

Ethanolamine, 99%, reagent grade (Aldrich Chemical Co.)

CONDITIONS

Columns:	IonPac CS12A analytical and CG12A guard, 2 mm
Eluent:	5 mN Sulfuric acid, step change to 30 mN Sulfuric acid at 10 min
Eluent Flow Rate:	0.25 mL/min
DXP Carrier Solution Flow Rate:	2 mL/min
Sample Volume:	7.5 mL
Concentrator Column:	IonPac TCC-LP1
Trap Column:	IonPac CG10, 4 mm
Detection:	Conductivity
Suppression:	CSRS®-II, 2 mm, external water mode
Current Setting:	100 mA
Expected System Backpressure:	1800 psi (12.4 MPa)
Expected Background Conductivity:	< 3 μS

GP40 Pump Program:

<u>Time</u> <u>(min)</u>	<u>A</u> ^a <u>(%)</u>	<u>B</u> ^b <u>(%)</u>	<u>Comments</u>
Initial	17	83	Equilibrate 5 mN H ₂ SO ₄
0.00	17	83	Load Sample Loop
0.10	17	83	Conc. Valve to "Load"
0.50	17	83	DXP Loading Pump "On"
9.50	17	83	DXP Loading Pump "Off"
10.00	17	83	Inject, Conc. Column in-line
20.00	17	83	End 5 mN H ₂ SO ₄
20.10	100	0	Step to 30 mN H ₂ SO ₄
30.00	100	0	End

^a30 mN H₂SO₄ ^bDI H₂O

1 N Sulfuric Acid Eluent

Carefully add 50.04 g of concentrated sulfuric acid to a volumetric flask containing about 500 mL of deionized water. Dilute to the 1-L mark and mix thoroughly.

30 mN Sulfuric Acid Eluent

Pipet 30 mL of the 1 N sulfuric acid stock solution to a 1-L volumetric flask. Dilute to 1 L with deionized water. Degas the eluent.

Matrix Preparation

For this study, three different matrices were prepared: (1) high-purity deionized water containing trace sodium at 0.1 µg/L (ppb); (2) 20-mg/L (ppm) morpholine with 0.05-µg/L sodium; and (3) 70-µg/L hydrazine with 2-mg/L ethanolamine and 0.1-µg/L sodium.

PREPARATION OF SOLUTIONS AND REAGENTS

1000-mg/L Lithium Standard Solution

Dissolve 6.108 g of lithium chloride in 1000 mL of deionized water. Be sure to dry an excess of the salt in a desiccator or oven before weighing it out.

SYSTEM PREPARATION AND SET-UP

Configure the IC system as shown in Figure 1. Connect the columns and suppressor in the IC system by using 0.005-in (0.125-mm) tubing. Keep the lengths of connecting tubing as short as possible to minimize system void volume and to ensure efficient 2-mm column operation. Construct a 7.5-mL sample loop with 649 in. (1648 cm) of 0.030-in. (0.75-mm) i.d. PEEK tubing (1 cm = 4.550 µL). To maintain efficient

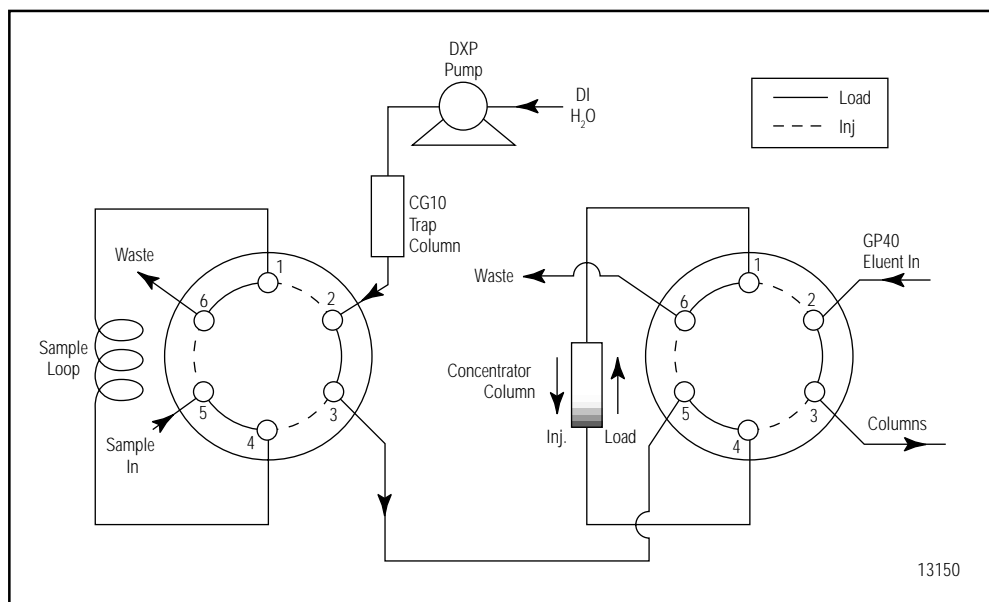


Figure 1. Ion chromatography preconcentration system.

operation of the DXP, the water container feeding this pump should be pressurized with 5 psi (34 kPa) of helium. Set the DXP pump so the flow is no more than 3 mL/min through the TCC-LP1. Prepare the IonPac CG10 trap column by pumping 1.0 N sulfuric acid at 1.0 mL/min for 50 min. Follow with a rinse of deionized water at the same flow rate for 20 min. Establish a blank by loading deionized water as a sample to verify that there is no significant concentration of lithium or other cationic contamination present.

RESULTS AND DISCUSSION

A representative separation for this method in high-purity deionized water is shown in Figure 2. Lithium is the first eluting peak and is well resolved from the other analytes. The method steps to a higher eluent concentration to elute strongly retained cations present in the matrix. A determination of lithium was also demonstrated in the morpholine and hydrazine matrices. Figure 3 shows the separation of a 100 ng/L lithium spike in the hydrazine matrix. Coefficients of determination (r^2) were calculated for lithium at 10, 30, 100, and 300 ng/L spiked in the morpholine and hydrazine matrices. Results showed a linear response, with r^2 values greater than 0.999. This method has a detection limit of 5 ng/L lithium at a signal-to-noise ratio (S/N) of 10.

PRECAUTIONS

Special care must be taken when performing trace analysis. Use only the highest quality deionized water to prepare eluents, standards, and dilutions. Ensure that all moving parts (such as piston seals and check valves) in the gradient pump are in good working order. From startup, a system for trace analysis typically requires about 5 hours to establish a stable background conductivity. For this reason it is best to keep the system running continuously. Ensure that there is an adequate supply of eluent, external water, and waste containers to allow the system to run unattended. The CG10 should be periodically regenerated by using the procedure in the System Preparation and Set-up section. How often this is necessary will depend on the level of contamination in the deionized water. An autosampler is not suitable for this application to achieve the low levels of detection that are required.

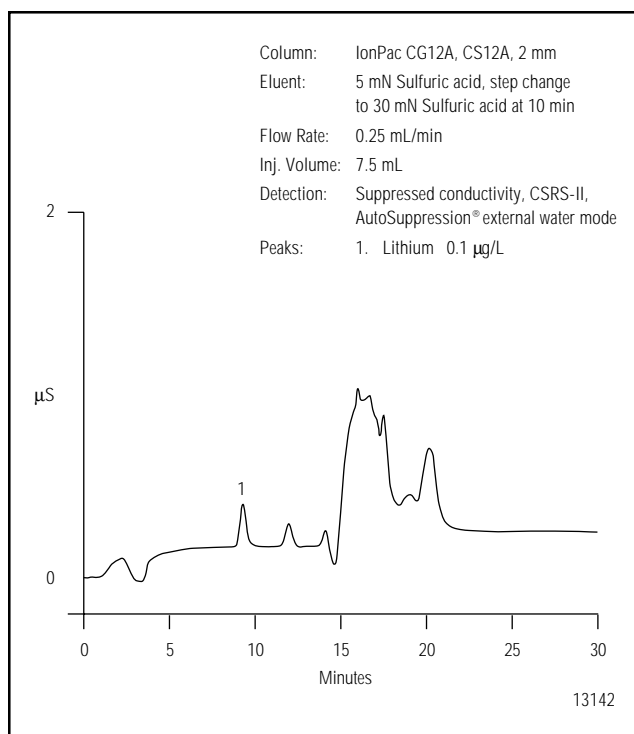


Figure 2. Trace lithium in high-purity DI water.

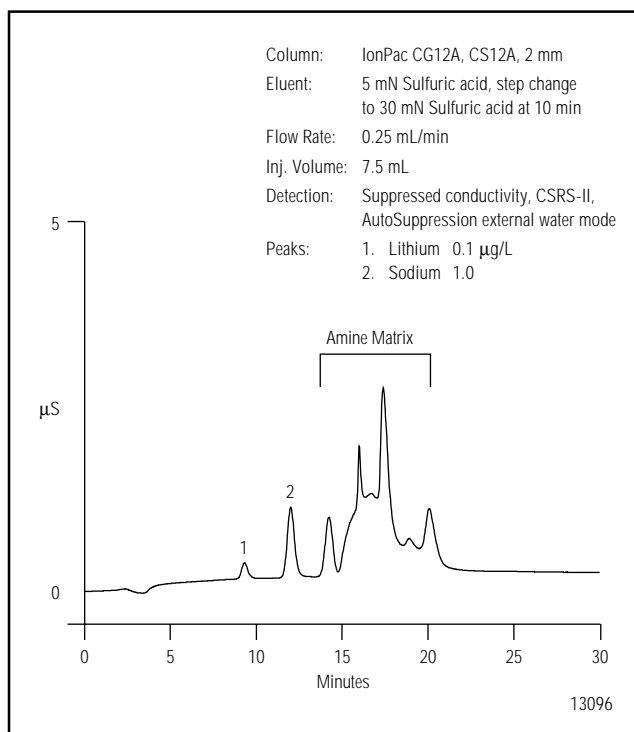


Figure 3. Trace lithium in a hydrazine sample matrix.

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3. Olenik, T.J. *Pollution Engineering.* **1974**, *6* (12), 31–38.
4. Horan, N.J.; Naylor, P.J. *Environ. Tech.* **1991**, *12* (7), 607–608.

LIST OF SUPPLIERS

J.T. Baker, Inc., 222 Red School Lane, Phillipsburg, New Jersey, 08865, USA. Tel.: 800-582-2537.

Aldrich Chemical Co., 1001 West Saint Paul Avenue, P.O. Box 355, Milwaukee, Wisconsin, 53233, USA. Tel.: 800-558-9160.

Sigma Chemical Co., P.O. Box 14508, St. Louis, Missouri, 63178, USA. Tel.: 800-325-3010.



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Dionex Corporation
1228 Titan Way
P.O. Box 3603
Sunnyvale, CA
94088-3603
(408) 737-0700

Dionex Corporation
Salt Lake City Technical Center
1515 West 2200 South, Suite A
Salt Lake City, UT
84119-1484
(801) 972-9292

Dionex U.S. Regional Offices
Sunnyvale, CA (408) 737-8522
Westmont, IL (630) 789-3660
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