

Determination of Trace Nickel and Zinc in Borated Power Plant Waters Containing Lithium Hydroxide Using Nonsuppressed Conductivity Detection

Introduction

There are currently more than 400 nuclear power plants worldwide that produce approximately 15% of the global electricity. In the U.S., nearly 20% of the electricity used is derived from nuclear power, and the U.S. Department of Energy conservatively forecasts that 28% more electricity will be needed by 2035.¹ Nearly 60% of the nuclear power plants in operation throughout the globe use pressurized water reactors (PWRs) to generate electricity, compared to only 20% that use boiler water reactors.² Historically, corrosion has been a major contributor to a loss of power generation in PWRs, which has caused a significant increase in repair and maintenance costs and radiation exposure to personnel.³ According to the Electric Power Research Institute (EPRI), stress corrosion cracking and general corrosion of reactor components have cost the nuclear power industry billions of dollars because of forced and extended power outages, replacement and repair of components, and increased regulatory oversight.⁴ Water chemistry plays a critical role in the degradation of materials, including steam generators, reactor components, fuel cladding, and plant piping.³ Therefore, monitoring ionic impurities in nuclear power plants is critical to minimize corrosion and reduce maintenance costs.

In the primary water of a PWR nuclear reactor, boric acid is added as a neutron absorber to control the core reactivity. During the initial stages of the fuel cycle, the concentration is relatively high (1000–2500 ppm boron) and then is gradually reduced to 0 ppm as the fuel is spent.^{5,6} Although boric acid is weakly dissociated, even a slightly acidic environment can cause corrosion of

materials, with the consequence of increased activation and transport of corrosion products.⁷ Therefore, lithium hydroxide is added to achieve a desired $\text{pH}_{300\text{ }^{\circ}\text{C}}$ range between 6.9 and 7.4, which minimizes corrosion, solubility, and transport of activated corrosion products.^{5,7}

Zinc addition to the water of a PWR has been found to suppress the Cobalt-58 and Cobalt-60 activity buildup on stainless steel surfaces.⁸ Cobalt-60 is the principal contributor to out-of-core radiation fields in PWRs. In general, cobalt is released through erosion, corrosion, friction, and other mechanisms that lead to its activation to Cobalt-60 in the reactor flow path. Cobalt-60 is a high-energy gamma emitter with a half-life of 5.3 years that produces long-term radiation areas in the plant.⁹ The use of zinc in PWRs is beneficial because it has a very high binding energy to a tetrahedral position on the metal oxide lattice, which is the preferential location of cobalt. The zinc displaces the cobalt from the tetrahedral site and, therefore, results in a reduction of the activity buildup.⁸ In addition, steam generators used in PWRs consist of Ni-base alloys that can be oxidized by the primary water. During this oxidation process, nickel can be released in the primary coolant, which increases contamination and leads to stress corrosion cracking.¹⁰ Therefore, the determination of $\mu\text{g/L}$ concentrations of zinc and nickel in PWRs is critical to minimize or inhibit corrosion and thereby reduce maintenance costs.

Ion chromatography (IC) can be used for the determination of transition metals in power plant waters. Typically, the determination of transition metals is achieved by forming either an anionic or cationic complex, depending on the complexing agent in the eluent, followed by postcolumn derivatization with 4-(2-pyridylazo)resorcinol (PAR) and absorbance detection at 530 nm. This postcolumn chemistry provides a very broad selectivity and good sensitivity for determining transition metals.¹¹ Cation-exchange chromatography with nonsuppressed conductivity detection can also be used to determine several different transition metals.¹² The formation of insoluble hydroxides from the suppressor reaction precludes the use of a suppressor for this analysis. However, although the use of nonsuppressed conductivity detection is a good alternative to the postcolumn method for determining selected transition metals in simple sample matrices, it does not provide the equivalent specificity and sensitivity for many transition metals when compared to use of the PAR postcolumn reagent.

In this application note (AN 250), the authors describe the determination of trace concentrations of nickel and zinc in borated water containing lithium hydroxide by cation-exchange chromatography using a 4 mm Thermo Scientific Dionex IonPac SCS 1 column and nonsuppressed conductivity detection. A TCC-ULP1 concentrator column is used to concentrate 3 mL of sample, which enables sub- $\mu\text{g/L}$ detection limits and single-digit $\mu\text{g/L}$ quantification limits for nickel and zinc. This method simplifies the determination of these transition metals by avoiding the additional expense and time required to prepare the PAR postcolumn reagent while still maintaining good sensitivity. The linearity, detection limits, precision, and accuracy of the method for determining the target transition metals in borated samples is described.

Equipment

Thermo Scientific Dionex ICS-3000 IC system* consisting of:

DP Dual Pump module (an SP Single Pump module can also be used)

DC Detector/Chromatography module (single- or dual-temperature zone configuration)

AS Autosampler with a 5 mL syringe (P/N 053915), 8.2 mL sampling needle assembly (P/N 061267)

Dionex IonPac™ Mixer, 500 μL (P/N 079971) for low-noise operation

Thermo Scientific Dionex Chromeleon 6.8 Chromatography Data System (CDS) software

*An ICS -1100, -1600, or -2100 system can also be used for this application.

Reagents and Standards

Deionized water, Type I reagent-grade, 18.2 M Ω -cm resistivity or better

Thermo Scientific Dionex Combined Six Cation Standard-II, 100 mL (P/N 057590)

Zinc sulfate monohydrate, $\geq 99.9\%$ trace metals basis (Sigma-Aldrich®, St. Louis, USA, P/N 307491)

Nickel standard for IC (1000 mg/L), 100 mL (Fluka®, St. Louis, USA, P/N 72224)

Oxalic acid dihydrate, ACS reagent, $\geq 99.5\%$ (Fluka, P/N 75700)

L-Ascorbic acid, 99% (Sigma-Aldrich®, P/N A92902)

Thermo Scientific Dionex methanesulfonic acid (MSA $> 99\%$, P/N 033478)

Samples

Boric acid, 99.5% min. (J.T.Baker®, Mansfield, USA, P/N 0084)

Lithium hydroxide, monohydrate, 99.5% (Sigma-Aldrich, P/N 254274)

Conditions

Columns:	Dionex IonPac SCS 1 Analytical, 4 $\times$ 250 mm (P/N 079809)
	Dionex IonPac SCG 1 Guard, 4 $\times$ 50 mm (P/N 079933)
Eluent:	2.5 mM MSA/1.2 mM oxalic acid/2 mM ascorbic acid
Flow Rate:	1 mL/min
Temperature:	30 °C
Sample Volume:	3 mL
Concentrator	
Column:	Thermo Scientific Dionex TCC-ULP1, 5 $\times$ 23 mm (P/N 063783)
Detection:	Nonsuppressed conductivity
Background:	$\sim 1300 \mu\text{S}$
Noise:	$\sim 10 \text{ nS}$ peak to peak
Backpressure:	$\sim 2350 \text{ psi}$
Run Time:	25 min

Preparation of Reagents and Standards

Eluent Solution

Prepare 1.0 N MSA stock solution by adding 96.10 g of Dionex MSA (> 99%, P/N 033478) to a 1 L volumetric flask containing about 500 mL of deionized water. Dilute to the mark and mix thoroughly. It is recommended to initially prepare a 3 mM MSA eluent to reproduce the QA chromatogram supplied with the column. To prepare 3 mM MSA, add 3 mL of 1.0 N MSA to a 1 L volumetric flask containing about 500 mL of degassed deionized water. Dilute to the mark and mix thoroughly. To prepare the eluent solution used to determine nickel and zinc (2.5 mM MSA/1.2 mM oxalic acid/2 mM ascorbic acid), combine 2.5 mL of 1.0 N MSA, 0.1513 g of oxalic acid dihydrate, and 0.3522 g of ascorbic acid in a 1 L volumetric flask containing about 500 mL of degassed deionized water. Dilute to the mark with degassed deionized water and mix thoroughly. Store the eluent under ~5–10 psi of head pressure with an inert gas in a plastic eluent bottle.

Precaution: Ascorbic acid can be rapidly oxidized to dehydroascorbic acid by increased temperatures, light, presence of oxygen, and other factors.¹³ Therefore, it is strongly recommended that any eluent containing ascorbic acid be prepared daily. Ascorbic acid is used to reduce Fe(III), if present, to Fe(II), which can be separated on the SCS 1 column. Although iron is not quantified in this method, iron may be present in some nuclear power plant samples, and the presence of ascorbic acid in the eluent is effective at removing iron from the column.

1000 mg/L Zinc Standard Solution

Dissolve 0.2744 g of zinc sulfate monohydrate in 100 mL of deionized water. Store this solution at 4 °C when not in use.

Intermediate Working Standard

Prepare a 10 mg/L combined intermediate solution for nickel and zinc by adding 200 µL of the 1000 mg/L zinc stock solution and 200 µL of the 1000 mg/L nickel standard IC solution in a 20 mL scintillation vial with 19.60 g of deionized water. Prepare a secondary intermediate standard by diluting the primary intermediate standard 1:10 to prepare a combined 1 mg/L each of Ni and Zn. Store this solution at 4 °C when not in use.

Working Standard Solutions

The working standards for Ni and Zn were prepared in the range of 2.5 to 25 µg/L by adding the appropriate volumes of the 1 mg/L intermediate working standard solution to a 100 mL volumetric flask and then diluting to the mark with deionized water. These standards were found to be stable during the duration of this study when stored at 4 °C. However, if the quality control check standards deviate from an acceptable range, then prepare a fresh standard from the stock solution.

Sample Preparation

1000 mg/L Lithium

Add 0.6046 g lithium hydroxide monohydrate to a 120 mL HDPE bottle and dilute to 100 mL with deionized water. Store this solution at 4 °C when not in use.

Simulated Borated/Lithium Samples

A boron concentration in the range of 1000 to 2500 mg/L with an added lithium concentration in the range of 1.8 to 5 mg/L was analyzed in this study. To prepare 1000 mg/L boron with 1.8 mg/L lithium, add 0.5719 g boric acid and 180 µL of 1000 mg/L lithium solution to a 120 mL HDPE bottle and dilute to 100 mL with deionized water. Agitate the solution to fully dissolve the boric acid. A 2000 mg/L boron solution with 3.8 mg/L lithium contained 1.1438 g boric acid with 380 µL of the 1000 mg/L lithium solution. For the 2500 mg/L boric acid with 5 mg/L lithium, dissolve 1.4296 g boric acid, then add 500 µL of 1000 mg/L lithium. Each solution was prepared as described for the 1000 mg/L boron with 1.8 mg/L lithium sample.

System Preparation and Setup

The Dionex ICS-1100, -1600, -2100 IC systems, or the ICS-3000 system may be used for nonsuppressed cation determinations. The correct setup and system preparation will be described for the Dionex ICS-3000 system for nonsuppressed cations. Configure the IC system by installing the 4 × 50 mm Dionex IonPac SCG 1 and 4 × 250 mm Dionex IonPac SCS 1 columns in the column compartment. Connect the outlet of the analytical column directly to the inlet of the conductivity detector and connect the outlet of the detector to a waste line. To reproduce the QA chromatogram supplied with the analytical column, install a 25 µL sample loop on the injection valve. A Dionex IonPac mixer is required for use with the SCS 1 column to average any eluent changes due to temperature fluctuations or pump pulsations and, therefore, reduce background noise. Install the mixer before the eluent inlet of the injection valve. Reverse the signal polarity to obtain positive analyte peaks by opening the System Configuration screen and double clicking DC Detector Compartment in the corresponding Timebase. Then, click the Detector panel and double-click CD1. In the CD1 panel, click the Signals tab and double-click CD_1, then change the Factor from 1.000 to -1.000. Repeat this process for the CD_1_Total signal channel. Detailed instructions for changing the conductivity polarity for the IC integrated systems can be found in application note 158.¹²

Install and configure the AS autosampler. This method requires the configuration of the AS autosampler sample mode to concentrate using the AS front panel (under System Parameters). The concentrate sample mode allows the AS to deliver sample to a low-pressure concentrator at a maximum pressure of 100 psi. Because this application requires large sample injection volumes, a minimum sample syringe size of 5 mL (P/N 053915) should be installed. To accommodate the larger volume, an 8.2 mL sampling needle assembly (P/N 061267) is required for operation.

Reproducing the QA chromatogram supplied with the column is recommended. This can be accomplished by first equilibrating the columns with 3 mM MSA for at least 60 min. A system equilibrated with 3 mM MSA has a background signal of < 1100 μ S and peak-to-peak noise of < 10 nS. Analyze a deionized water blank first to be sure that no peaks elute at the same retention time as the cations of interest. Prepare a 100 $\times$ dilution of the Dionex Six Cation Standard-II and make a 25 μ L full-loop injection. Confirm that the resulting chromatogram is similar to the QA chromatogram supplied with the column. Install a 5 $\times$ 23 mm TCC-ULP1 concentrator in place of the sample loop using black PEEK tubing with the direction of sample loading opposite the direction of analytical flow. Next, equilibrate the columns with 2.5 mM MSA/1.2 mM oxalic acid/2 mM ascorbic acid for at least 90 min, but preferably overnight. A system equilibrated with this eluent has a background signal of $\sim$ 1300 μ S and peak-to-peak noise of approximately 10 nS. Inject 3 mL of a 25 μ g/L standard of nickel and zinc. The column is equilibrated when duplicate injections of the standard produce identical retention times.

Results and Discussion

U.S. nuclear power plant utilities initially began using zinc injection in the mid-1990s, and its use has steadily increased in the last 15 years. The addition of lower zinc concentrations (5 to 10 μ g/L) to the reactor coolant systems is typically used to reduce plant radiation fields while higher concentrations (15 to 40 μ g/L) are used to mitigate pressurized water stress corrosion cracking in Alloy 600 materials. In 2003, EPRI issued a guideline that recommended plants consider zinc injection to improve radiation field management. Since these guidelines were initially issued, additional data has indicated that even low concentrations of zinc are beneficial in mitigating primary water stress corrosion cracking.¹⁴ Therefore, the determination of low concentrations of zinc in nuclear power plants is a critical part of a proper water chemistry program.

The determination of other transition metals, such as nickel and iron, provide data to minimize or prevent stress corrosion cracking in nuclear power plants. Iron is a typical impurity found in the primary water from the corrosion of stainless steels. The oxidizing reaction on the surface of the metals releases Fe(II), which is then oxidized to Fe(III) by either water or by the oxidizing radiolysis products.⁶ Only Fe(II) can be separated and detected by nonsuppressed conductivity. However, in the typical pH range of 6.9–7.4 for borated power plant samples, iron is primarily found in the ferric form because this species is favored at pH $\geq$ 6.¹⁵ For example, if a borated sample containing Fe(III) is injected on the column using the eluent described in the Conditions section (pH $\sim$ 2.6), a response for iron will not be observed. This is due to the formation of $\text{Fe}(\text{C}_2\text{O}_4)_3^{3-}$, a kinetically-stable trivalent oxalate anion complex. This complex does not interact with the cationic stationary phase due to its high stability and therefore elutes in the void volume.¹⁶ Therefore, Fe(III) must be reduced to Fe(II) at low pH prior to analysis using a reducing agent, such as ascorbic acid, to be determined by the method described in this AN.

There are other variables to also consider before determining iron by this method. The reduction rate of Fe(III) to Fe(II) by ascorbic acid decreases significantly as the pH increases; therefore, lowering the sample pH prior to adding ascorbic acid is recommended. In general, standards are usually prepared using Fe(II), not Fe(III), in solutions containing ascorbic acid at low pH to minimize the oxidation of iron. Even in this case, a small fraction of Fe(III) may be present.

Another consideration is that ascorbic acid itself can be oxidized to dehydroascorbic acid, which cannot effectively reduce Fe(III) because of poor coordination with Fe(II).

Therefore, given the complexities of the reactions of iron, it is strongly recommended to simultaneously determine both iron species using PAR with absorbance detection.¹¹ In this application note, the data for iron determinations in nuclear power plants will not be presented because of these inherent analytical challenges. However, if iron is present in the sample, it can be effectively eluted from the column as Fe(II) and separated from other cations if ascorbic acid is present in the eluent and if other criteria discussed above are met (Figure 1).

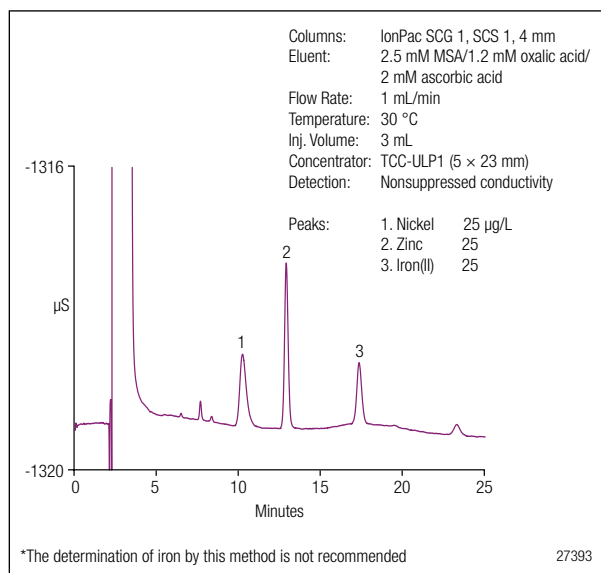


Figure 1. Separation of nickel, zinc, and iron(II) on the Dionex IonPac SCS 1 column.

Method Performance

It is important to establish a deionized water blank by injecting 3 mL on the concentrator column. To obtain a representative blank, the same set of containers and other components used to prepare the boric acid samples should be used for the deionized water blank. As shown in Figure 2, trace concentrations of common cations, which do not interfere with the target metals, were detected in the water blank. Calibration curves from 2.5 to 25 µg/L for nickel and zinc were prepared in deionized water from the 1 mg/L standard solutions. The limit of detections (LODs) and limit of quantifications (LOQs) were determined by first measuring the peak-to-peak noise from the deionized water blank injection. The typical baseline noise using the conditions described in this application note was ~10 nS. The calculated LODs for nickel and zinc were 0.76 and 0.43 µg/L, respectively,

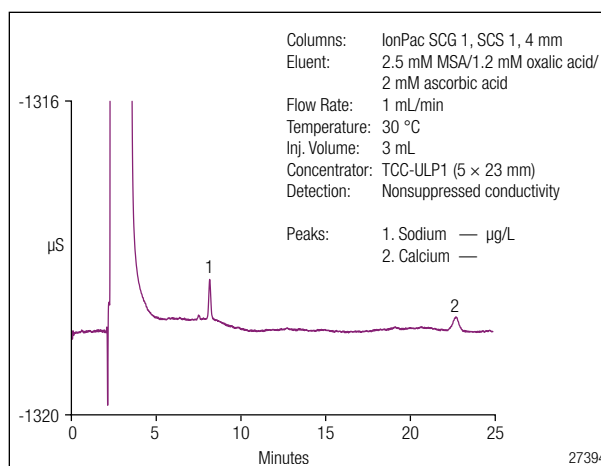


Figure 2. Representative deionized water blank.

Table 1. Linearity, LODs, and LOQs for Nickel and Zinc in Deionized Water

Analyte	Range (µg/L)	Correlation Coefficient (r ²)	RSD	LODa (µg/L)	LOQb (µg/L)
Nickel	2.5–25	0.9999	0.73	0.76	2.50
Zinc	2.5–25	0.9998	1.96	0.43	1.42

^aS/N = 3.

^bS/N = 10.

based on three times the signal-to-noise ratio (S/N). The calculated LOQs for nickel and zinc were 2.5 and 1.42 µg/L, respectively, based on 10 times the S/N. This demonstrates the capability of nonsuppressed conductivity detection to achieve sub-µg/L LODs for the target transition metals when using large injection volumes. Table 1 summarizes the calibration data, LODs, and LOQs for nickel and zinc. Figure 3 shows a representative chromatogram of a 2.5 µg/L nickel and zinc standard in deionized water using the Dionex IonPac SCS 1 column with an eluent solution containing 2.5 mM MSA/1.2 mM oxalic acid/2 mM ascorbic acid.

The method performance was evaluated by analyzing synthetic water samples containing 1000–2500 mg/L boron as boric acid with corresponding lithium concentrations as lithium hydroxide in the range of 1.8 to 5 mg/L. In each of the samples evaluated in this application note, the concentrations of nickel and zinc were less than their respective LODs. To evaluate the accuracy of the method, the samples were spiked with approximately 5 µg/L each of nickel and zinc. Figure 4 demonstrates the applicability of the method for this analysis by comparing chromatograms with and without the addition of trace concentrations of nickel and zinc in 1000 mg/L boron containing 1.8 mg/L lithium. As shown, the large lithium matrix peak begins to elute from the column at 6.3 min and returns to baseline well before nickel is eluted.

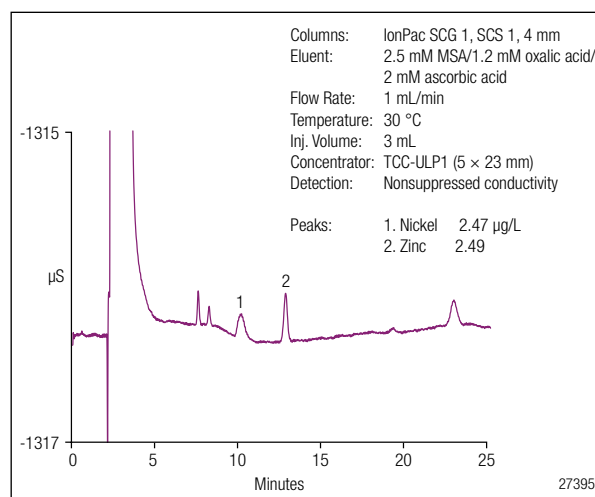


Figure 3. Separation of 2.5 µg/L each of nickel and zinc in deionized water.

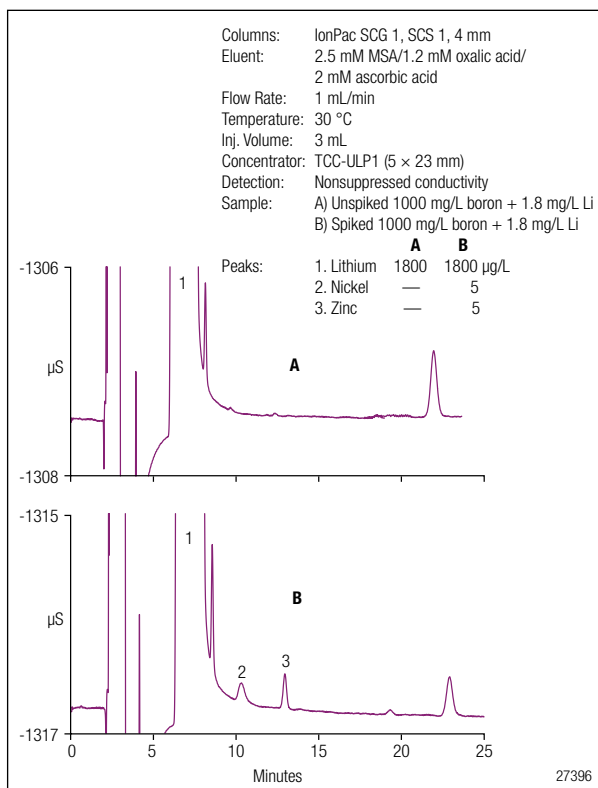


Figure 4. Chromatograms of A) borated water and B) borated water spiked with trace concentrations of nickel and zinc.

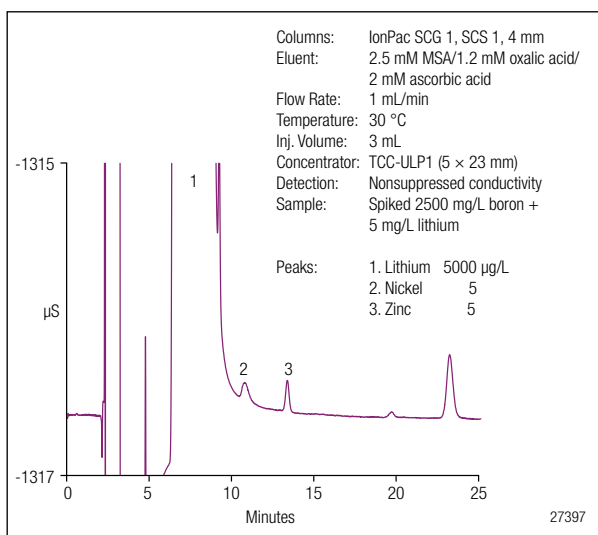


Figure 5. Chromatogram of 2500 mg/L boron and 5 mg/L lithium spiked with trace concentrations of nickel and zinc.

However, due to the high lithium concentration, the retention times for nickel and zinc increased by 2.2% and 1.5%, respectively, relative to the standard retention times. For a sample containing 2000 mg/L boron with 3.8 mg/L lithium, the retention times increased by 4.3% and 3.3% for nickel and zinc, respectively.

Figure 5 demonstrates the separation of 5 µg/L nickel and zinc added to a 2500 mg/L borated sample containing 5 mg/L lithium. In this case, the retention times further increased by 6.2% and 4.2% for nickel and zinc, respectively. To reduce these shifts in retention times, the sample injection volume would have to be decreased to minimize the concentration of lithium on the column. However, this would consequently increase the respective LODs and LOQs for the method. Despite the observed retention time shifts, the method accuracy was not affected as indicated from the excellent recoveries (Table 2). In general, the average recoveries ranged from 95.4–102.5% for nickel and 94.0–100.4% for zinc. The method precision was evaluated by calculating the retention time and peak area relative standard deviations (RSDs) from six replicate injections. The retention time RSDs for nickel and zinc ranged from 0.08 to 0.34 and 0.019 to 0.035, respectively. The peak area precision based on the replicate injections was 1.4–2.3% and 0.74–1.28% for nickel and zinc, respectively. These values are not significantly different than the same nickel and zinc concentrations prepared in deionized water, which produced retention time RSDs of 0.18 and 0.036, respectively, and peak area RSDs of 1.79 and 1.38, respectively. These results demonstrate that the method accuracy appears to be independent of the boron concentration, which suggests that the method performed well for determining low µg/L concentrations of nickel and zinc in borated nuclear power plant waters.

Table 2. Average Recoveries of Nickel and Zinc Spiked in Simulated Borated Nuclear Power Plant Waters

Analyte	n	1000 mg/L boron + 1.8 mg/L Li		2000 mg/L boron + 3.8 mg/L Li		2500 mg/L boron + 5 mg/L Li	
		Amount Added (µg/L)	Avg. Recovery (%)	Amount Added (µg/L)	Avg. Recovery (%)	Amount Added (µg/L)	Avg. Recovery (%)
Nickel	6	5.06	95.4	5.25	102.5	5.25	95.7
Zinc	6	4.97	94.0	5.16	94.4	5.15	100.4

Conclusion

A cation-exchange chromatography method combined with nonsuppressed conductivity detection was developed for the determination of low µg/L concentrations of nickel and zinc in borated power plant waters. This method eliminates the time and expense associated with preparing a PAR postcolumn reagent for this determination and, therefore, further enhances its ease of use. Use of the silica-based Dionex IonPac SCS 1 column, combined with a low MSA eluent concentration with oxalic acid as an effective complexing agent, enabled the resolution of low concentrations of nickel from high lithium concentrations

at concentration ratios up to 1:1000. Despite these disparate concentration ratios, excellent recoveries were achieved for nickel and zinc. These recoveries also demonstrated that high boron and lithium concentrations had a minimal influence on the method performance. This is further demonstrated by the fact that no significant difference was observed in retention time and peak area RSDs for the investigated samples matrices, relative to the standards in deionized water. In addition, the method demonstrated good linearity for the evaluated concentration range, and sub- to low-µg/L LODs and LOQs that are applicable to the nuclear power industry.

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Suppliers

VWR International, 1310 Goshen Parkway, West Chester, PA, 19380, 1-800-932-5000, www.vwr.com.

Sigma-Aldrich, PO Box 14508, St. Louis, MO, 63178, 1-800-325-3010, www.sigma-aldrich.com.

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