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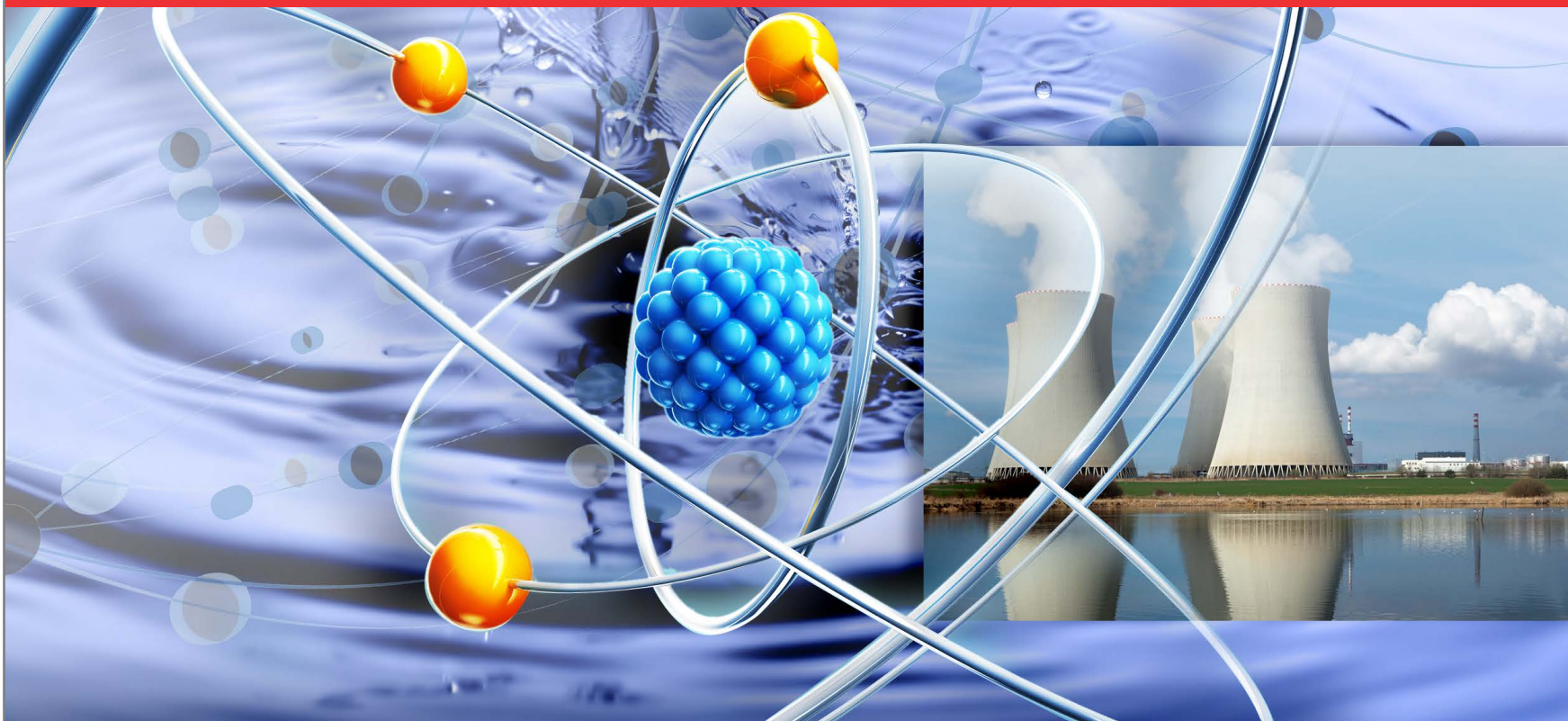
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# Ion Chromatography

## Power Plant Water Applications Summary Notebook



The ionic purity of boiler water, feedwater, borated water, and steam is important to maintain component reliability, safety, and the overall economic viability of nuclear power plant (NPP) and fossil fuel power plant (FFPP) operation. Failure mechanisms in the boiler, turbine, heaters, and condensers of power plants can be directly related to water and steam ionic purity. By understanding the ionic contamination levels for water and steam contaminants, utilities and operators can eliminate or reduce damage from corrosion, scaling, and deposition. Each power plant has a set of water chemistry matrices that are often specific for that plant and usually based on the water chemistry history and metallurgy of the plant. In general though, the typical matrices are:

- Pure water
- Pure water with amine additives
- Borated water
- Closed cooling water (with high nitrite concentrations)
- Environmental samples (effluents, soils, air, etc.)

Reagent-Free Ion Chromatography has been widely adopted in most nuclear and fossil fuel power plants due to its ease of use and low detection limits, which make for a good fit with the typical matrices and analytes which need to be monitored in this industry.

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# Pure Water



The chemistry of the feed water used in NPPs and FFPPs must be strictly controlled in both the primary and secondary systems in order to protect against corrosion, which can compromise the integrity of power plant components leading to faulty and hazardous operation. Identification of ionic contaminants in water is critical for the identification and prevention of corrosive conditions and ion chromatography is an indispensable technique to identify the individual ionic species at sub- to low- $\mu\text{g/L}$  concentrations.

- **Determination of Trace Anions in High Purity Waters by High Volume/Direct Injection Ion Chromatography**
- **Determination of Trace Anions in High-Purity Waters Using Direct Injection and Two-Step Isocratic Ion Chromatography**
- **Determination of Transition Metals at PPT Levels in High-Purity Water and SC2 (D-clean) Baths**
- **Determination of Trace Anions in High-Purity Waters by Ion Chromatography with the Dionex IonPac AS17 Using High-Volume Direct Injection with the EG40**
- **Determination of Trace Sodium and Transition Metals in Power Industry Samples by Ion Chromatography with Nonsuppressed Conductivity Detection**

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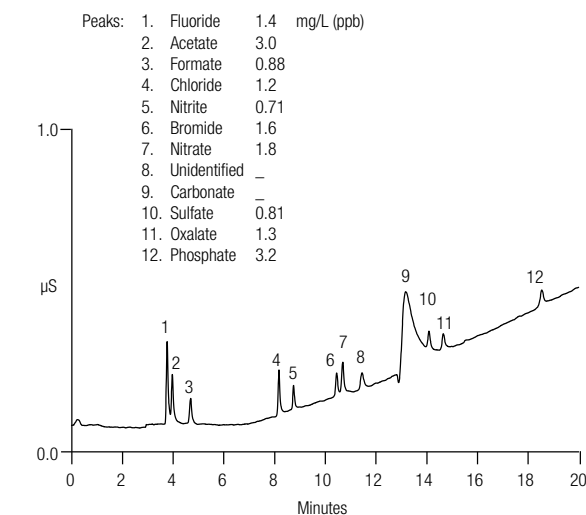
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# Trace Anions in High Purity Waters by High Volume/Direct Injection

There has been considerable interest in the determination of anions at trace levels by ion chromatography (IC). For example, the Electric Power Research Institute (EPRI) has established IC as the analytical technique for determining sodium, chloride, and sulfate down to 0.25 µg/L (ppb) in power plant waters.

This application note demonstrates the use of a gradient separation on the Thermo Scientific™ Dionex™ IonPac™ AS11 Column which enables low to sub-µg/L detection limits for anions in high-purity water by direct injection. This technique eliminates the sample pump, concentrator column, preconcentration time, and recovery problems associated with sample preconcentration. This method simplifies the analysis and reduces run time of high-purity water and power plant treated waters.



Trace anion determination.

### Experimental Conditions

Columns:	Dionex IonPac AS11 Analytical, 2 mm i.d. Dionex IonPac AG11 Guard, 2 mm i.d. Thermo Scientific Dionex IonPac ATC Anion Trap Column, 2 mm i.d.
Eluents:	1) DI H <sub>2</sub> O 2) 5 mM NaOH 3) 100 mM NaOH Gradient program—see Table 1.
Flow Rate:	0.5 mL/min
Sample Volume:	750 µL
Detection:	Suppressed conductivity, Thermo Scientific™ Dionex™ ASRS™ Anion Self-Regenerating Suppressor, 2 mm, external water mode, 300 mA current

Time (min)	%E1	%E2	%E3	Comments
0	0	65	35	Initial 38 mM NaOH
1.99	0	65	35	End regeneration
2.00	90	10	0	Start 0.5 mM NaOH
9.00	90	10	0	End equilibration
11.50	90	10	0	0.5 mM to 5.0 mM NaOH
15.00	0	100	0	5.0 mM to 26 mM NaOH
29.00	0	78	22	End at 26 mM NaOH
29.10	0	65	35	Step to 38 mM NaOH

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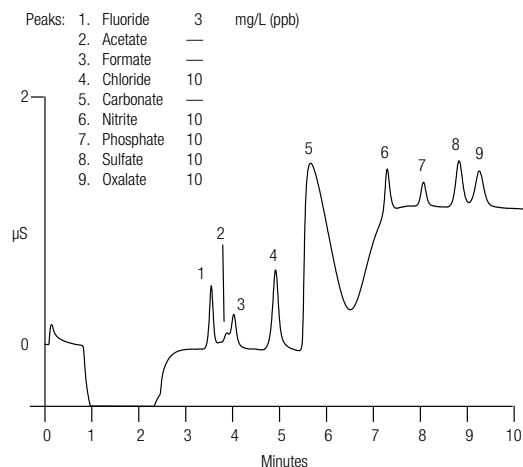
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# Trace Anions in High Purity Waters Using Direct Injection and Two-Step Isocratic IC

Reliable methods to quantify anionic contaminants in high-purity water are essential to both the semiconductor and power generation industries. Anionic contamination is known to cause metal corrosion in microelectronic circuitry. Likewise, the presence of low  $\mu\text{g/L}$  (ppb) concentrations of chloride and sulfate can make the stainless steel components of a power plant (such as steam generators, boiler tubes, condenser tubes, and turbine blades) susceptible to stress corrosion cracking.

To respond quickly to changing conditions, a fast method is needed by those monitoring anionic contamination at trace levels. This application note describes a rapid, high-volume, direct-injection technique that utilizes a Dionex IonPac AS14 2 mm analytical column and a borate eluent with a step change. The separation was applied to high-purity water as well as amine-treated matrices of interest to the power generation industry. Common anions such as fluoride, chloride, nitrate, phosphate, and sulfate were determined at levels below 1  $\mu\text{g/L}$  (ppb) in less than 15 minutes.



Trace anions in 8 mg/L (ppm) morpholine.

Conditions			
Columns:	Dionex IonPac AS14 Analytical, 2 × 250 mm Dionex IonPac AG14 Guard, 2 × 50 mm		
Trap Column:	Dionex IonPac ATC Anion Trap Column, 2 mm		
Eluent A:	9 mM Boric acid/6.75 mM Sodium hydroxide		
Eluent B:	40 mM Boric acid/30 mM Sodium hydroxide		
Pump Program:			
Time (min)	A (%)	B (%)	Comments
Initial	100	0	Equilibrate initial eluent
0	100	0	Load sample loop
5.00	100	0	Inject
8.50	100	0	
8.51	0	100	Step to stronger eluent
15.00	0	100	
Eluent Flow Rate:	0.75 mL/min		
Detection:	Suppressed Conductivity, Dionex ASRS suppressor, 2 mm, AutoSuppression™ external water mode		
SRS Current Setting:	300 mA		
Expected Background			
Conductivity:	2–4 µS		
Expected System			
Backpressure:	17.2 MPa (2500 psi)		
Sample Volume:	1 mL		



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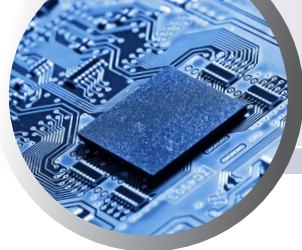
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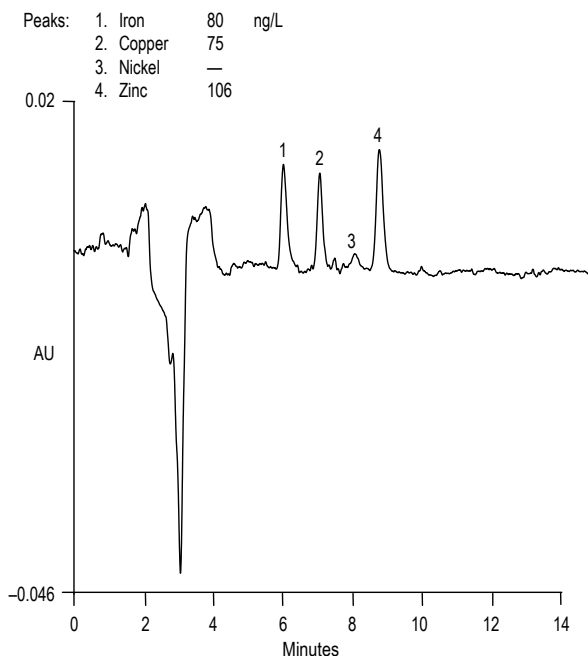
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# Transition Metals at PPT Levels in High Purity Water and SC2 Baths

Metal atoms and conductive particle contaminants are undesirable and potentially damaging in semiconductor manufacturing processes. Oxidative cleaning baths and large volumes of ultrapure rinse water are used to remove metallic contamination from wafer surfaces. For optimal cleaning efficiency, the concentrations of iron and other metals in cleaning solutions should be minimized. To monitor metals in cleaning baths and rinse water, improved analytical methods are needed.

This application note describes a method for determining low ng/L amounts of transition metals in high purity water and semiconductor bath solutions. The Dionex IonPac CS5A column is used for the separation of transition metals. This column has a unique bilayer latex structure consisting of both anion- and cation-exchange retention mechanisms.



SC2 batch (30 mL concentrated) containing 1 mL HCl/5 mL H<sub>2</sub>O<sub>2</sub>/494 mL H<sub>2</sub>O

Conditions	
Columns:	Dionex IonPac CS5A Analytical, 2 × 250 mm Dionex IonPac CG5A Guard, 2 × 50 mm Dionex IonPac TCC-2 Cation Concentrator, 3 × 35 mm
Eluent:	Pyridine-2,6-dicarboxylic acid (PDCA)
Eluent Flow Rate:	0.3 mL/min
LC-30 Temperature:	30.0 °C
Postcolumn Reagent:	0.06 g of PAR in 1 L MetPac PAR Postcolumn Diluent
Postcolumn Flow Rate:	0.15 mL/min
Concentrator Pump Flow Rate:	2.0 mL/min
Run Time:	15 min
Detection:	Visible, High setting, 530 nm
System Backpressure:	1700–2000 psi

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# Trace Anions in High Purity Waters Using High-Volume Direct Injection

This application note describes an ion chromatographic method using the microbore Dionex IonPac AS17 anion-exchange column to determine trace concentrations of common inorganic anions, low-molecular-weight organic acids, as well as acrylate, methacrylate, benzoate, and phthalate. These contaminants can come from some of the following sources: cleaning agents, adhesives, oils, mold release agents, and solder fluxes. Anionic contamination is known to cause corrosion in microelectronic circuitry. The analytes are detected by suppressed conductivity using two different gradient methods: a 27 min separation and 41 min separation.

### **Pump Program Method 1**

Time (min)	Flow (mL/min)	A Valve (%)	Dionex EG40 Generator Conc. (mM)	Comments
Initial	0.50	100 Load	20.0	20 mM KOH
0.00	0.50	100 Load	1.0	Load sample loop, equilibrate 1.0 mM KOH for 5 min
5.00	0.50	100 Inject	1.0	Inject
15.00	0.50	100 Inject	1.0	1 mM KOH
19.00	0.50	100 Load	12.0	12 mM KOH
25.00	0.50	100 Load	20.0	20 mM KOH
27.00	0.50	100 Load	20.0	20 mM KOH

### **Pump Program Method 2**

Time (min)	Flow (mL/min)	A Valve (%)	Dionex EG40 Generator Conc. (mM)	Comments
Initial	0.50	100 Load	40.0	40 mM KOH
0.00	0.50	100 Load	0.3	Load sample loop, equilibrate 0.3 mM KOH for 5 min
5.00	0.50	100 Inject	0.3	Inject
11.00	0.50	100 Inject	0.3	0.3 mM KOH
13.00	0.50	100 Inject	1.0	1 mM KOH
24.00	0.50	100 Load	10.0	10 mM KOH
41.00	0.50	100 Load	40.0	40 mM KOH

Conditions	
Columns:	IonPac AS17 analytical, 2 × 250 mm
Columns:	IonPac AG17 guard, 2 × 50 mm
Eluent:	Potassium hydroxide (Thermo Scientific Dionex EG40 Eluent Generator as the source)
Temperature:	30 °C
EG40 Offset Volume:	0 µL
Eluent Flow Rate:	0.5 mL/min
Detection:	Suppressed conductivity, Dionex ASRS ULTRA suppressor, gas-assisted recycle mode
ASRS Current Setting: 100 mA	
Expected Background	
Conductivity:	1 µS (40 mM KOH)
Expected System	
Backpressure:	15.2–16.6 MPa (2200–2400 psi)
Sample Volume:	1 mL



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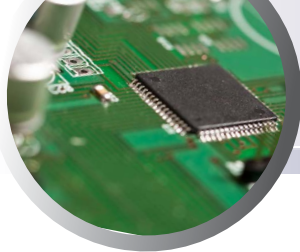
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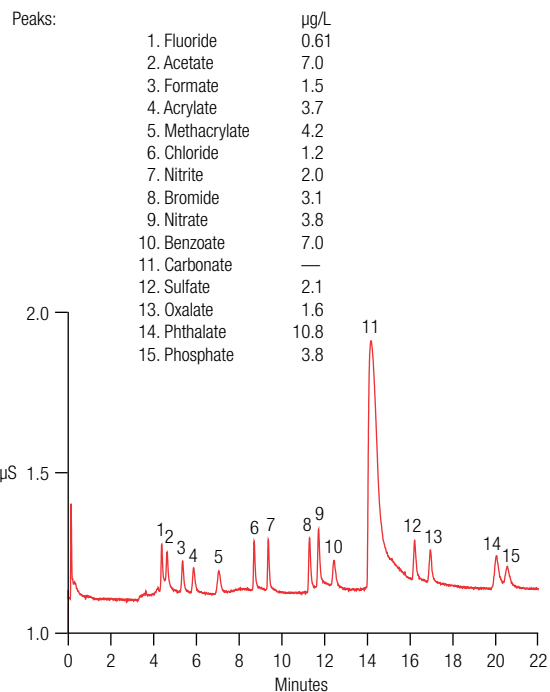
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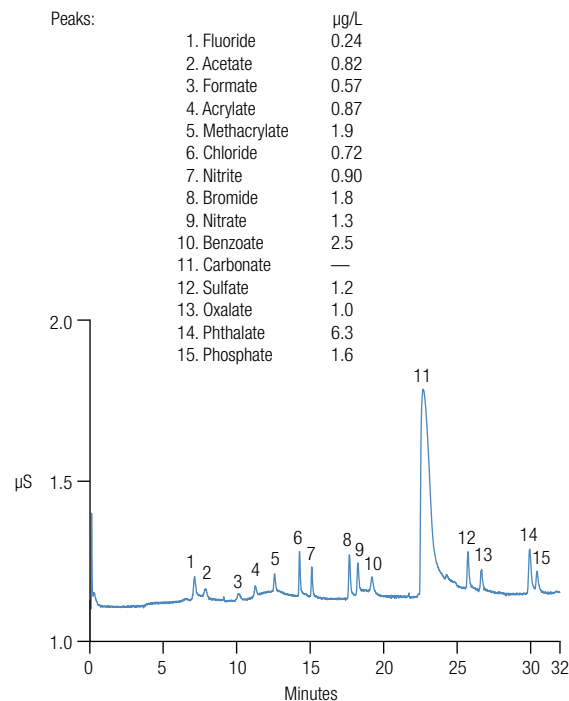
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# Trace Anions in High Purity Waters Using High-Volume Direct Injection



Trace anion determination using the Dionex IonPac AS17 column with Method 1 (for fast sample throughput).



Trace anion determination using the Dionex IonPac AS17 column with Method 2 for samples with target analytes at disparate concentrations.





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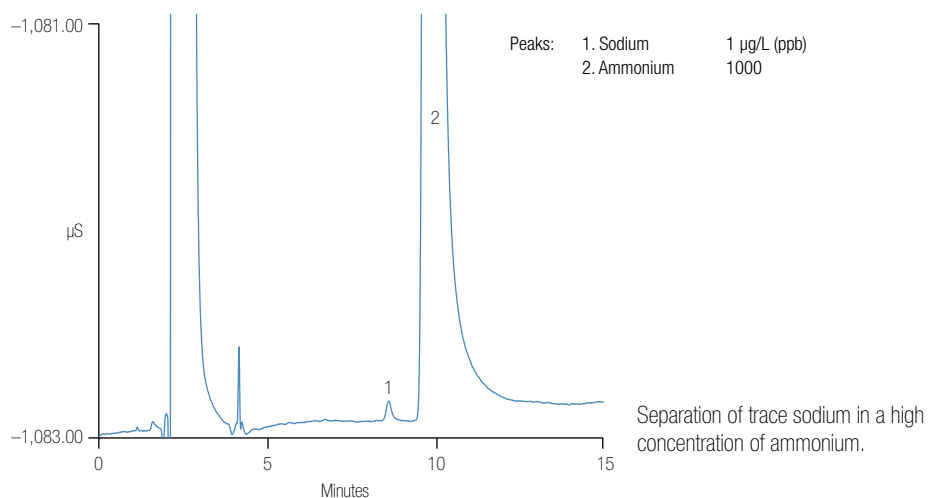


# Trace Sodium and Transition Metals in Power Industry Samples

Corrosive contaminants—particularly sodium, chloride, and sulfate—have been implicated as a major source of corrosion and deposition-related plant shutdowns in nuclear and fossil-fueled plants. IC can measure these contaminants and has been implemented in several U.S. power plant water chemistry monitoring programs. This methodology allows analysts to achieve parts per trillion (ppt) detection limits for individual anionic and cationic species and operate on-line to effectively measure and eliminate corrosive hideout, thereby reducing the plant's operating costs.

This application note demonstrates the capability of nonsuppressed conductivity detection for the determination of trace sodium in ethanolamine-treated samples and the determination of some transition metals. This method yielded good recoveries for sodium spiked at the sub-ppb concentrations in ethanolamine samples containing up to 5 ppm. However, higher ethanolamine concentrations will overload the concentrator and analytical columns, causing decreased peak response and increased peak broadening for sodium. In effect, the low breakthrough volume of the Thermo Scientific Dionex TCC-LP1 column in combination with the Dionex IonPac SCS 1 column with nonsuppressed conductivity detection reduces the sensitivity that can be achieved by this method. This characteristic makes cation exchange with suppressed conductivity detection a more favorable approach because it does not suffer from these limitations.

Conditions	
Columns:	Dionex IonPac SCS 1 Analytical, 4 × 250 mm Dionex IonPac SCG 1 Guard, 4 × 50 mm
Eluent:	3 mM MSA
Flow Rate:	1 mL/min
Temperature:	30 °C
DXP Flow Rate:	2 mL/min
Sample Volume:	3 mL
Concentrator	
Column:	Thermo Scientific Dionex TCC-LP1 Trace Cation Concentrator Column, 4 × 35 mm
Detection:	Nonsuppressed conductivity
Background:	~1100 µS
Noise:	~5–10 nS peak-to-peak
Backpressure:	~2100 psi
Run Time:	15 min



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# Pure Water with Amine Additives

Corrosion inhibitors and oxygen scavengers are added to control pH and create a reducing environment in both liquid and vapor phases of an NPP cooling system. Morpholine and ethanolamine (ETA), the most commonly used organic amines in E.U. and U.S. NPPs, respectively, minimize corrosion by maintaining the water and vapor phase pH between 9.5 and 9.8. Ion-exchange chromatography with suppressed conductivity detection is a well established method to determine  $\mu\text{g/L}$  to  $\text{mg/L}$  concentrations of common cations and amines; therefore, it is the method of choice in the power industry.



## Determination of Trace Cations in Power Plant Waters Containing Morpholine

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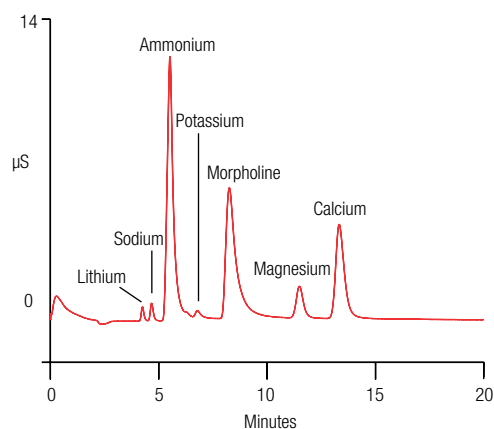
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# Trace Cations in Power Plant Waters Containing Morpholine

Morpholine and ammonium are used as additives in power plant waters. Morpholine acts as a corrosion inhibitor, whereas ammonium is used to control pH. In this matrix, it is critical to determine the presence of inorganic cation contaminants. This method uses the Dionex IonPac CS14 column to quantify trace concentrations of lithium, sodium, potassium, magnesium, and calcium in the presence of high levels of ammonium and morpholine. Acetonitrile can be added to the eluent to improve peak shape and optimize resolution for some of the cations of interest.



Trace cations in Morpholine mix: Method 2 (MSA with 5% acetonitrile).

Conditions	
Columns:	(2) Dionex IonPac CG14 (2 mm) guard columns, one used as a guard column, the other as a concentrator column (1) Dionex IonPac CS14 (2 mm) analytical column
Eluent:	8 mM Methanesulfonic acid or 8 mM Methanesulfonic acid in 5% acetonitrile (v/v)
Eluent Flow Rate:	0.25 mL/min
Rinsing Flow Rate:	1.0 mL/min
Sample Volume:	1.0 mL
Detection:	Suppressed Conductivity
Suppressor:	Thermo Scientific™ Dionex™ CSRS™ I Cation Self-Regenerating Suppressor (2 mm), AutoSuppression, Recycle Mode (without acetonitrile); External Water Mode (with acetonitrile)

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# Borated Water



In NPPs boric acid is used in pressurized water reactor (PWR) power plants to control the nuclear reaction, because boron is a good neutron absorber. In PWR plants, measuring anionic contaminants in borated waters is an important part of corrosion monitoring.

- **Application of Eluent Generation for Trace Anion Analysis of Borated Waters**
- **Determination of Trace Nickel and Zinc in Borated Power Plant Waters Containing Lithium Hydroxide Using Nonsuppressed Conductivity Detection**
- **Determination of Organic Acids and Inorganic Anions in Lithium-Containing Boric Acid-Treated Nuclear Power Plant Waters**
- **Determination of Trace Organic Acids and Inorganic Anions in Boric Acid-Treated Power Plant Waters Using an Automated Reagent-Free Ion Chromatography System**
- **Determination of Trace Fluoride, Chloride, and Sulfate in Lithium-Containing Borated Waters**

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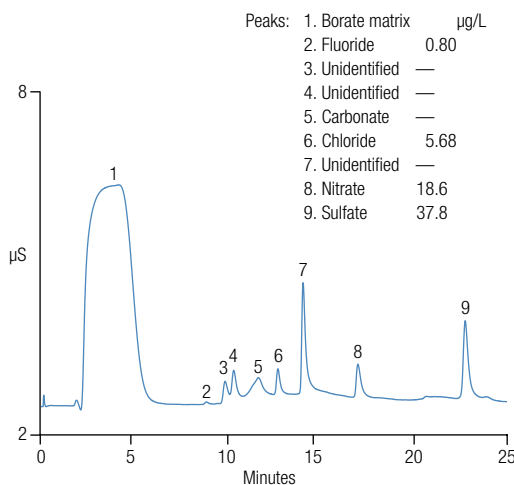
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# Trace Anion Analysis of Borated Waters

Boric acid is used in PWR power plants to control the nuclear reaction, because boron is a good neutron absorber. The presence of low- $\mu\text{g/L}$  (ppb) concentrations of chloride and sulfate can make the stainless steel components of a power plant, such as steam generators, boiler tubes, condenser tubes, and turbine blades, susceptible to stress-induced corrosion cracking. Therefore, measuring anionic contaminants in borated waters is an important part of corrosion monitoring. A reliable method is needed to determine anionic contamination at trace levels in borated waters.

This application note describes a rapid, high-volume, direct injection technique that uses a Dionex IonPac AS14 2 mm analytical column. Common anions such as fluoride, chloride, nitrate, phosphate, and sulfate can be determined at levels below 1  $\mu\text{g/L}$  (ppb) in borated waters with up to 7500 mg/L boron in less than 30 min.



Trace anions in 2000 mg/L boron.

Conditions	
Columns:	Dionex IonPac AS14 Analytical, 2 mm i.d. Dionex IonPac AG14 Guard, 2 mm i.d.
Eluent:	50 mM boric acid titrated with potassium hydroxide from an Dionex EG50 Eluent Generator
Temperature:	30 °C
Gradient:	4.0 mM KOH from 0–6.0 min with 50 mM boric acid 4.0 to 30.0 mM KOH from 6–18 min with 50 mM boric acid
Flow Rate:	0.38 mL/min
Detection:	Suppressed conductivity
Suppressor:	Dionex ASRS ULTRA II suppressor, 2 mm, AutoSuppression, external water mode
Suppressor current:	38–46 mA
Expected Background Conductivity:	3 $\mu\text{S}$ (30 mM potassium hydroxide/ 50 mM boric acid)
Recommended System Backpressure:	15.2–16.6 MPa (2200–2400 psi)

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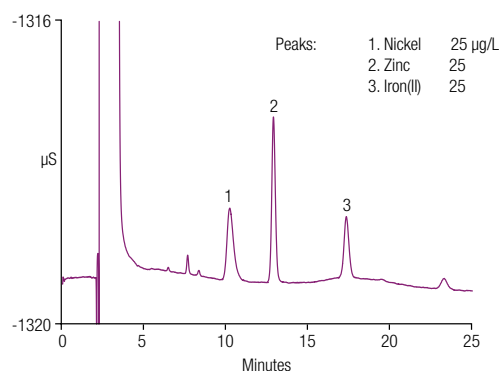


# Trace Nickel and Zinc in Borated Power Plant Waters

The determination of transition metals in power plant waters 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. Cation-exchange chromatography with nonsuppressed conductivity detection can also be used to determine several different transition metals.

This application note describes the determination of trace concentrations of nickel and zinc in borated water containing lithium hydroxide by cation-exchange chromatography using a 4 mm Dionex IonPac SCS 1 column and nonsuppressed conductivity detection. A Dionex 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.

Conditions	
Columns:	Dionex IonPac SCS 1 Analytical, 4 $\times$ 250 mm Dionex IonPac SCG 1 Guard, 4 $\times$ 50 mm
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:	Dionex TCC-ULP1, 5 $\times$ 23 mm
Detection:	Nonsuppressed conductivity
Background:	~1300 $\mu\text{S}$
Noise:	~10 nS peak to peak
Backpressure:	~2350 psi
Run Time:	25 min



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

\*The determination of iron by this method is not recommended



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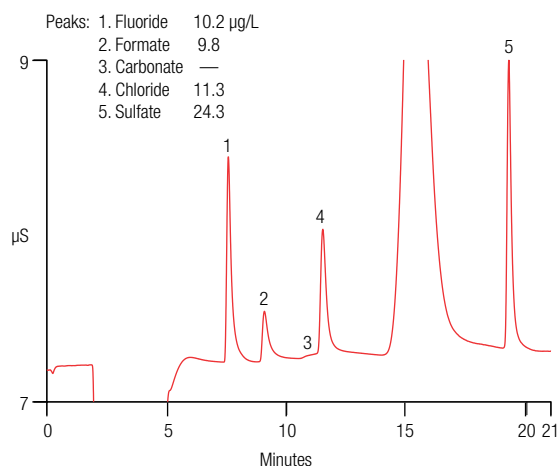
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# Organic Acids and Inorganic Anions in Lithium-Containing Boric Acid-Treated Power Plant Waters

For nuclear power plants using a PWR, the primary coolant includes boric acid as a water-soluble neutron absorber to control the nuclear reaction. Boron concentrations at the beginning of the fuel cycle may be as high as 2500 mg/L (ppm), corresponding to 1.4 percent boric acid. At the high temperature and pressure in the PWR, this boric acid concentration can form residue that deposits metal oxides on the fuel rods. To prevent residue buildup, lithium hydroxide is added to increase the pH to 6.9 or higher. However, trace anionic impurities at low- or sub- $\mu\text{g/L}$  (ppb) concentrations from the water source (or materials such as ion-exchange polisher resins) can serve as catalysts for corrosion.

The large-volume, direct injection approach for the determination of selected organic acids and inorganic anions in borated waters has been extended to include lithium-containing matrices. The method uses a Thermo Scientific Dionex CR-CTC II Continuously Regenerated Cation Trap Column to remove mg/L lithium levels in the simulated matrices and a high-capacity 2 mm Dionex IonPac AS22 column set to separate the anions after a 1 mL sample injection. With attention to careful sample and system handling, low- $\mu\text{g/L}$  concentrations of fluoride, formate, chloride, and sulfate can be determined with good linearity, precision, and accuracy.



Typical chromatogram of trace anions spiked into 2000 mg/L boron plus 4 mg/L lithium surrogate matrix.

Conditions	
Columns:	Dionex IonPac AG22 Guard column, 2 × 50 mm Dionex IonPac AS22 Analytical column, 2 × 250 mm
Temperature:	35 °C
Flow Rate:	0.38 mL/min
Eluent:	200 mM Boric acid titrated with KOH from EGC II KOH
Gradient:	

Time (min)	KOH Conc. (mM)	Comments
-1.0	14	Re-Equilibration
0	14	Inject*
9.9	14	Start Gradient
10.0	90	End Gradient
24.9	90	Start Re-Equilibration Gradient
25.0	14	End Re-Equilibration Gradient

\*Aspirate sample at speed 2, deliver sample at speed 1.

Injection Volume:	1000 $\mu\text{L}$ (Full loop)
Detection:	Suppressed conductivity
Suppressor:	Dionex ASRS 300 suppressor, 2 mm, external water mode (1.5 to 2 mL/min)
Suppressor Current:	85 mA
Expected Background Conductivity:	7 to 8 $\mu\text{S}$ (14 mM Potassium hydroxide, 200 mM boric acid)
Noise:	<5 nS/min peak-to-peak
System Backpressure:	16.6 to 18.0 MPa (2400 to 2600 psi)

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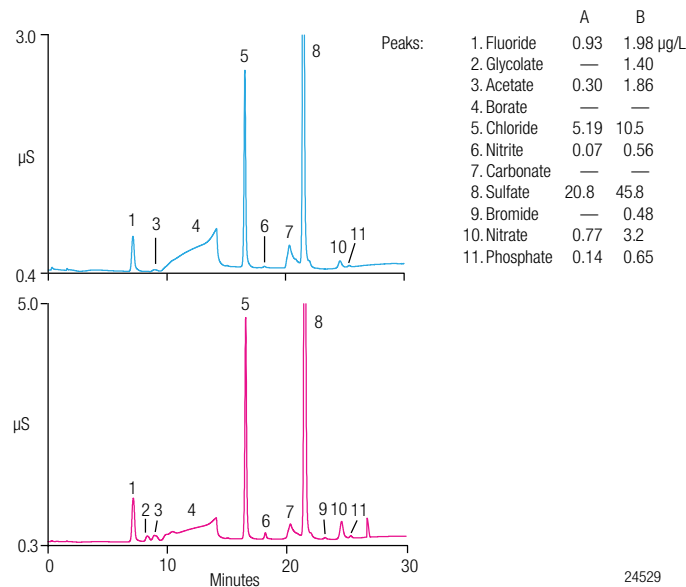


# Trace Organic Acids and Inorganic Anions in Boric Acid-Treated Waters

In pressurized water reactors (PWRs) boric acid is used as a water soluble neutron absorber to control the nuclear reaction. The determination of sub- $\mu\text{g/L}$  anionic contaminants, such as chloride and sulfate, in boric acid treated waters is critical for nuclear power plants to minimize or inhibit corrosion and thereby reduce the associated maintenance costs.

This application note describes an IC method using a 2 mm Dionex IonPac AS15 column and electrolytically generated potassium hydroxide eluent for the determination of low  $\mu\text{g/L}$  concentrations of target organic acids and inorganic anions in simulated boric acid-treated power plant waters.

Conditions	
Columns:	Dionex IonPac AG15, AS15, 2 mm
Eluent:	7 mM KOH 0–10 min, 7–40 mM KOH 10–16 min, 40–60 mM KOH 16–22 min, 60 mM KOH 22–30 min
Eluent Source:	EGC II KOH with CR-ATC
Flow Rate:	0.40 mL/min
Sample Volume:	2 mL
Matrix Elim. Volume:	10 mL
Temperature:	30 °C
Detection:	Suppressed conductivity, ASRS ULTRA II, 2 mm, recycle mode, 65 mA
Concentrator:	UTAC-ULP1 (5 × 23 mm)
Samples:	A) Unspiked 2000 mg/L boron + 3.8 mg/L Li B) Spiked 2000 mg/L boron + 3.8 mg/L Li



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# Trace Fluoride, Chloride, and Sulfate in Lithium-Containing Borated Waters

Nuclear power plants are an important source of electrical energy. For nuclear power plants using PWRs, boron is added in the form of boric acid as a neutron absorber in the reactor coolant to control the nuclear reactivity in the primary reactor. The pH of the coolant is also crucial in maintaining the system because a pH lower than 6.9 (at an elevated temperature of ~300 °C) can cause accelerated corrosion of the fuel rod cladding. Lithium hydroxide is added to the coolant to achieve the desired pH to prevent residue buildup. Trace anionic impurities in borated waters can catalyze corrosion. Therefore, monitoring the concentrations of trace anions is important to reduce the risks associated with operation and to maintain plant reliability.

This study demonstrates the determination of trace fluoride, chloride, and sulfate in simulated borated waters containing lithium using a borate eluent and a large volume direct injection. This method is based on Thermo Scientific Application Note 114: Determination of Anions in High-Purity Waters Using Direct Injection and Two-Step Isocratic Ion Chromatography and extends the analysis of anions in high-purity water to lithium-containing borated waters. Using a slightly modified eluent condition, the determination of fluoride, chloride, and sulfate is achieved with the early eluting borate peak resolved from fluoride and other target anions. Three different simulated samples are examined and the method is validated by recovery and precision studies on these simulated samples.

Conditions			
Columns:	Dionex IonPac AG14 Guard, 2 × 50 mm Dionex IonPac AS14 Analytical, 2 × 250 mm		
Eluents:	A: 100 mM boric acid/75 mM sodium hydroxide B: DI water		
Eluent Conditions:			
Time (min)	A (%)	B (%)	Comment
0.00	10	90	Inject
4.00	10	90	—
4.01	65	35	Step to stronger eluent
8.50	65	35	—
8.51	10	90	Equilibrate eluent for next injection
15.00	10	90	—
Flow Rate:	0.5 mL/min		
Inj. Volume:	1 mL		
Temperature:	25 °C (upper compartment) 30 °C (lower compartment)		
Detection:	Suppressed conductivity, Dionex ASRS 300 suppressor 55 mA, external water mode		
System			
Backpressure:	~2170 psi		
Background			
Conductance:	1.3–3.4 µS		
Noise:	~1.2 nS/min peak-to-peak at the first step, ~8.4 nS/min peak-to-peak at the second step		
Run Time:	15 min		



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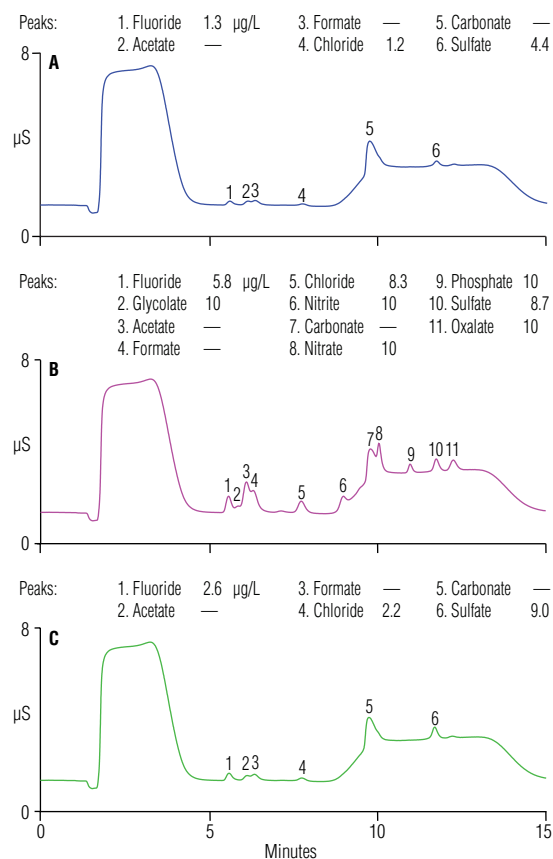
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# Trace Fluoride, Chloride, and Sulfate in Lithium-Containing Borated Waters



Chromatograms of (A) a 2500 mg/L B + 5.0 mg/L Li-simulated sample; (B) the same sample spiked with trace anions; and (C) the same sample spiked with 1.3  $\mu\text{g/L}$  fluoride, 1.0  $\mu\text{g/L}$  chloride, and 4.5  $\mu\text{g/L}$  sulfate.



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# Closed Cooling Water



In NPPs, water in secondary and cooling systems controls the heat created from the fission of radioactive isotopes, to produce steam, which is used to generate electricity. Stress-corrosion cracking and flow-assisted corrosion in the NPP boiler, secondary, and cooling systems can cause increased maintenance time and cost, and loss of power generation. Therefore, it is important to minimize corrosion in NPPs to avoid or reduce these associated events. Ion chromatography is the method of choice for the determination of potentially corrosive ionic impurities that include fluoride, chloride, and sulfate.

- **Determination of Trace Anions in High-Nitrate Matrices by Ion Chromatography**
- **Fast and Sensitive Determination of Transition Metals in Power Industry Waters Using Ion Chromatography**
- **Determination of Polyacrylic Acid in Nuclear Power Plant Pressurized Water Reactor Secondary Feed Water**
- **Determination of Trace Anions in Nuclear Power Plant Secondary Feed Water Containing Polyacrylic Acid**

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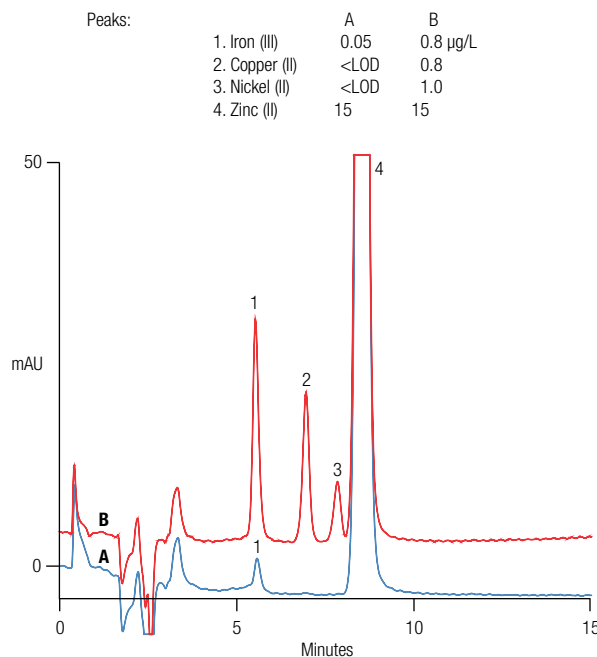
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# Fast and Sensitive Determination of Transition Metals in Power Industry Waters

In pure water, the boiling water reactor (BWR) environment is oxidizing due to the radiolytic generation of species such as oxygen and hydrogen peroxide, increasing the propensity of metals to undergo intergranular stress corrosion cracking (IGSCC). Injecting hydrogen into the feedwater and adding noble metal suspensions promote recombination of hydrogen and oxidants on the metal surfaces, thus lowering IGSCC. Control of zinc, iron, copper, and other impurities in BWR final feedwater is highly dependent on the type of condensate polishing system in use.

An IC method with 530 nm absorbance detection was evaluated for the determination of iron (III), copper (II), nickel (II), and zinc (II) at sub- and low- $\mu\text{g/L}$  concentrations in boiling water and the primary cooling circuit of pressurized water nuclear power plant reactors. The Dionex IonPac CS5A column with a PDCA eluent allowed the separation of the four target transition metals in less than 10 min., even in the presence of high concentrations of boric acid and lithium that constitute the PWR primary coolant surrogate matrix. Excellent recoveries were achieved for the target analytes in deionized water and boric acid/lithium hydroxide matrices, with and without additional zinc added.



Chromatogram of transition metals in surrogate PWR matrix (2000 mg/L B + 3.8 mg/L Li + 15  $\mu\text{g/L}$  Zn<sup>2+</sup>). Trace A: PWR surrogate matrix. Trace B: PWR surrogate matrix spiked with 0.8  $\mu\text{g/L}$  Fe<sup>3+</sup>, Cu<sup>2+</sup>, and 1.0  $\mu\text{g/L}$  Ni<sup>2+</sup>.

### Conditions

Columns:	Dionex IonPac CG5A, 2 × 50 mm Dionex IonPac CS5A, 2 × 250 mm
Eluent:	7.0 mM Pyridine-2,6-dicarboxylic acid (PDCA) 66 mM Potassium hydroxide 5.6 mM Potassium sulfate 74 mM Formic acid
Flow Rate:	0.3 mL/min
Concentrator:	Dionex IonPac TCC-2
Vol. Conc.:	4.7 mL
Column Temp.:	30 °C
Postcolumn:	0.24 mM 4-(2-Pyridylazo) resorcinol (PAR) 1.0 M 2-Dimethylaminoethanol 0.50 M Ammonium hydroxide 0.30 M Sodium bicarbonate
PCR Flow Rate:	0.15 mL/min (40–60 psi)
PC Mixer:	125 $\mu\text{L}$ Knitted reaction coil, 2 mm
Detection:	Absorbance at 530 nm
Noise:	~0.12 mAU/min
Backpressure:	2150 psi
Run Time:	20 min



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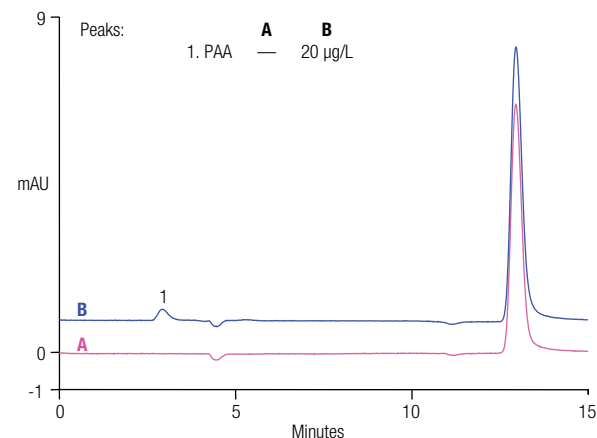


# Polyacrylic Acid in Nuclear Power Plant Pressurized Water

The cost of corrosion-related failures in NPPs is significant, prompting extensive study of corrosion-inhibiting mechanisms for nuclear power reactor systems. In PWRs, deposit fouling in the secondary system (steam generator) can cause undesired consequences such as heat-transfer losses, corrosion of heat-exchange tubes, and reduction in plant output. Studies have shown that the addition of polyacrylic acid (PAA) polymer dispersant to the steam generator water increases the corrosion resistance of steel and promotes removal of iron-based deposits from surfaces.

This study demonstrates the determination of low concentrations of PAA in secondary feed water containing ETA and hydrazine. PAA is resolved from the matrix using a Thermo Scientific™ Acclaim™ SEC-1000 polymer-based size-exclusion column. Water is used as the eluent to maximize sensitivity and eliminate the need for eluent preparation. PAA is detected at 200 nm with a MDL of ~2.6 µg/L in secondary water samples. This method provides a relatively simple and economical solution for NPPs to determine PAA in secondary feed water.

Conditions	
Columns:	Acclaim SEC-1000 Analytical, 7.8 × 300 mm
Eluent:	DI Water
Flow Rate:	1.0 mL/min
Inj. Volume:	300 µL
Temperature:	30 °C
Detection:	200 nm
System Backpressure:	~1044 psi
Noise:	~20 µAU peak-to-peak
Run Time:	15 min



A) Simulated secondary feed water matrix blank and B) simulated secondary feed water spiked with 10 µg/L PAA, with a 10% signal offset applied.

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# Trace Anions in Nuclear Power Plant Feed Water

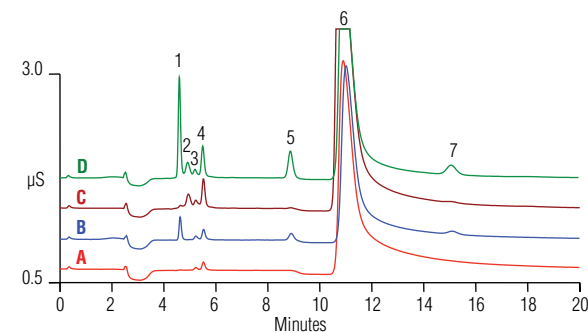
In 2011, NPPs provided 12.3% of the world's electricity production. In the U.S., PWRs comprise approximately two-thirds of the NPPs. The PWR consists of a primary reactor system and a secondary steam system. The chemistry of the feed water must be strictly controlled in both the primary and secondary systems. To protect the secondary steam system from corrosion, an amine such as ETA is added to the feed water to obtain an alkaline pH, while a volatile oxygen scavenger such as hydrazine is used to ensure a reducing environment to form protective magnetite on the surfaces of the steam generator.

This study describes the determination of low concentrations ( $<1 \mu\text{g/L}$ ) of fluoride, chloride, and sulfate in simulated PWR feed water that contains PAA. The method demonstrated efficient trapping of the target anions on the Dionex IonPac UTAC-ULP2 concentrator column, while the sample cations (i.e., ETA and hydrazine) and PAA were diverted to waste. The anions were separated within 20 min on a Dionex IonPac AS15 column set using isocratic elution with electrolytically generated potassium hydroxide. Excellent retention time and peak area reproducibilities were demonstrated from 69 consecutive injections of a spiked simulated sample.

Conditions	
Columns:	Dionex IonPac AG15 and AS15 set, 2 mm
Eluent Source:	Dionex EGC III KOH with Dionex CR-ATC
Eluent:	35 mM KOH
Flow Rate:	0.25 mL/min
Inj. Volume:	1 mL
Temperature:	30 °C
Detection:	Suppressed Conductivity, Dionex ASRS 300 Suppressor, 2 mm, 22 mA, recycle mode

Peaks:	A	B	C*	D
1. Fluoride	—	0.5	0.005	2.0 $\mu\text{g/L}$
2. Glycolate	—	—	—	—
3. Acetate	—	—	—	—
4. Formate	—	—	—	—
5. Chloride	—	0.5	—	2.0
6. Carbonate	—	—	—	—
7. Sulfate	—	0.5	0.015	2.0

\*Estimate



Chromatograms of A) a water blank; B) 0.5  $\mu\text{g/L}$  fluoride, 0.5  $\mu\text{g/L}$  chloride, and 0.5  $\mu\text{g/L}$  sulfate in DI water; C) simulated PAA water; and D) simulated PAA water spiked with 2  $\mu\text{g/L}$  fluoride, 2  $\mu\text{g/L}$  chloride, and 2  $\mu\text{g/L}$  sulfate.

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# Environmental Samples



Ammonium and organic acids from the degradation of morpholine, ethanolamine, and hydrazine accumulate in the secondary and condenser water systems of nuclear power plants (NPPs), which require periodic flushing and blowout. This wastewater effluent, regulated by national wastewater discharge permits, limits amine discharges at levels specified for the individual NPP that range from low  $\mu\text{g/L}$  to low  $\text{mg/L}$  concentrations. Therefore, a sensitive method, such as ion chromatography, is needed to determine  $\mu\text{g/L}$  concentrations for compliance monitoring



### **Determination of Morpholine, Ethanolamine, and Hydrazine in Simulated Nuclear Power Plant Wastewater**



### **Determination of Inorganic Cations and Ammonium in Environmental Waters by Ion Chromatography Using the Dionex IonPac CS16 Column**

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# Morpholine, Ethanolamine, and Hydrazine in Simulated Nuclear Power Plant Wastewater

NPP wastewater can contain high salt content from periodic flushes of the boiler (blowout) to remove salt buildup (i.e., scale). The determination of morpholine, ETA, and hydrazine in the presence of mg/L concentrations of ammonium is an analytical challenge due to their similar selectivity on cation-exchange phases and the presence of higher concentrations of ammonium and other common cations. These challenges were met by optimizing one method to determine both hydrazine and morpholine and a second method to determine ETA.

This application note describes two methods to determine  $\mu\text{g/L}$  concentrations of hydrazine, morpholine, and ETA in disparately larger concentrations of ammonium and other cations in a simulated NPP wastewater sample. Hydrazine and morpholine were determined in the simulated NPP wastewater matrix on the Dionex IonPac CS16 column by suppressed conductivity detection and integrated pulsed amperometric detection (IPAD). This hydrazine and morpholine method takes advantage of the dual pump and detector capabilities of the Thermo Scientific Dionex ICS-3000 system to accurately and precisely deliver the sodium hydroxide PCR required to selectively detect hydrazine and morpholine in the presence of ammonium by IPAD. Ethanolamine is separated by cation-exchange chromatography on the Dionex IonPac CS15 column with suppressed conductivity detection using the column's selectivity to elute ethanolamine before ammonium, along with the precise eluent preparation capabilities of the Reagent-Free™ Ion Chromatography Eluent Generation (RFIC-EG™) system.



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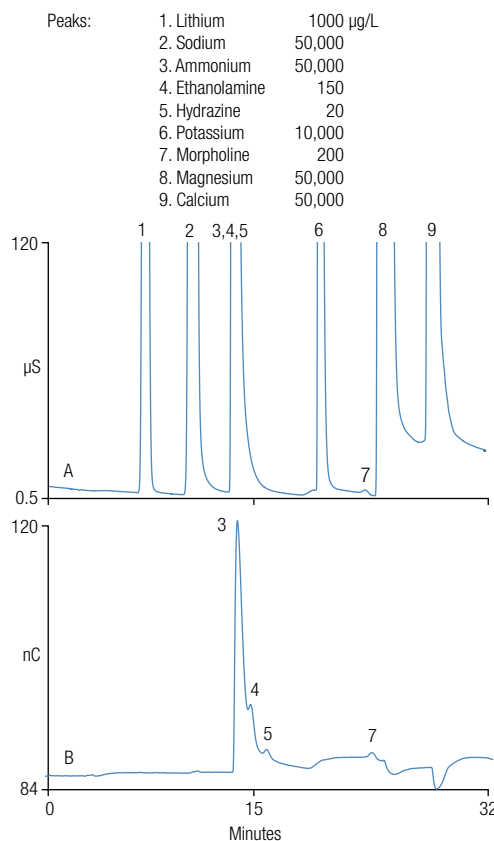
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# Morpholine, Ethanolamine, and Hydrazine in Simulated Nuclear Power Plant Wastewater



Comparison of hydrazine and morpholine in a simulated NPP wastewater matrix by A) suppressed conductivity detection and B) IPAD.

## Conditions

Column: Dionex IonPac CG16 Guard, 3 × 50 mm  
Dionex IonPac CS16 Analytical, 3 × 250 mm

Eluent (EG): 15 mM Methanesulfonic acid (MSA) from  
–10 to 15 min, 40 mM from 15 to 23 min,  
40 to 65 mM from 23 to 25 min, 65 mM from  
25 to 32 min

Flow Rate: 0.4 mL/min

Temperature: 40 °C (Lower compartment)  
30 °C (Upper compartment)

Inj. Volume: 25 µL

Detection: Suppressed conductivity, Thermo Scientific™  
Dionex™ CSRS™ 300 Cation Self-Regenerating  
Suppressor, 2 mm, external water mode,  
77 mA

Background  
Conductance: 0.3–0.5 µS

Conductance  
Noise: 0.3–0.6 nS

Typical System  
Backpressure: 2200 psi

## Postcolumn Reagent Addition

Detection: IPAD, disposable Dionex AAA-Certified Au,  
Waveform in Table 1

Reagent Flow: 50 mM Sodium hydroxide at 0.14 mL/min

Data Collection  
Rate: 1.66 Hz

IPAD  
Background: 40–60 nC

IPAD Noise  
with Suppressor: 60–80 pC

IPAD System  
Backpressure: 2200 psi

Typical pH: 12.1–12.3

Run Time: 32 min

Time (s)	Potential vs pH (V)	Gain Region <sup>a</sup>	Integration	Ramp <sup>a</sup>
0.00	+0.13	Off	Off	Ramp
0.04	+0.13	Off	Off	Ramp
0.05	+0.33	Off	Off	Ramp
0.21	+0.33	On	On (Start)	Ramp
0.22	+0.55	On	On	Ramp
0.46	+0.55	On	On	Ramp
0.47	+0.33	On	On	Ramp
0.56	+0.33	Off	Off (End)	Ramp
0.57	–1.67	Off	Off	Ramp
0.58	–1.67	Off	Off	Ramp
0.59	+0.93	Off	Off	Ramp
0.60	+0.13	Off	Off	Ramp



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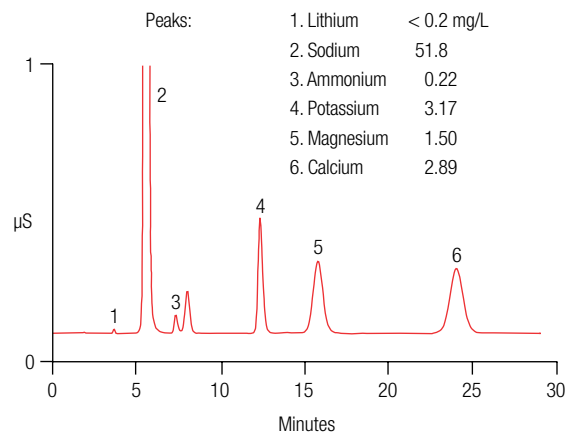


# Inorganic Cations and Ammonium in Environmental Waters

Alkali and alkaline earth cations are commonly determined by using spectroscopic techniques such as AAS or ICP, but ammonium cation in the same sample must be measured separately by a wet chemical technique such as titrimetry, colorimetry (Nesslerization, phenate, or automated phenate methods), or ammonia-selective electrode. Furthermore, the latter two methods may also require a separate distillation step before ammonia can be determined in wastewater. IC in a single run can determine ammonium plus all the important inorganic cations, including lithium, sodium, potassium, magnesium, and calcium.

This application note describes the use of ion chromatography with a Dionex IonPac CS16 cation-exchange column to determine dissolved alkali and alkaline earth cations and ammonium in drinking water, wastewater, and aqueous soil extracts. This application note also evaluates the effect of potential interferences on method performance during the analysis of typical environmental samples.

Conditions	
Columns:	Dionex IonPac CS16 Analytical, 5 × 250 mm Dionex IonPac CG16 Guard, 5 × 50 mm
Eluent:	26 mM MSA
Eluent Source:	Thermo Scientific Dionex EG40 Eluent Generator
Flow Rate:	1.5 mL/min
Temperature:	30 °C
Injection:	10 µL
Detection:	Suppressed conductivity, Dionex CSRS ULTRA suppressor, 4 mm, Autosuppression Recycle mode, current setting 100 mA
Background:	< 1 µS
Noise:	~0.2 nS peak-to-peak
Backpressure:	~2300 psi
Run Time:	30 min



Determination of inorganic cations and ammonium in industrial wastewater with the Dionex IonPac CS16 column.



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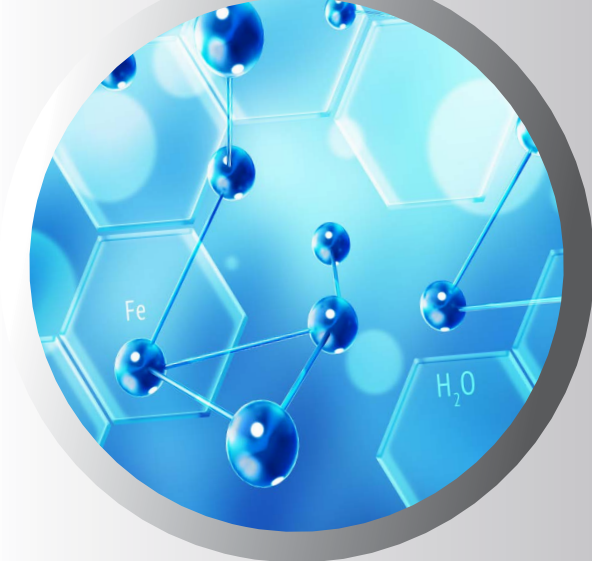
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- **Reagent-Free Ion Chromatography**
- **High-Pressure Ion Chromatography, 4 µm Particle Columns**
- **Chromatography Data Systems**
- **Ion Chromatography and RFIC Systems**

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# Reagent-Free Ion Chromatography

## Eluent Generation

RFIC-EG systems have redefined IC by making it possible to just add water to operate an IC system. These systems allow for a simpler and more reliable way to help deliver superior results while simultaneously saving time and labor. Eluent generation allows the automatic production of high-purity IC eluents. This is made possible through precise control of the electric current applied to the electrolysis of water to generate hydroxide and hydronium ions. Eluent generation eliminates the need to manually prepare eluents from concentrated acids and bases. The only routine reagent needed is deionized water. Furthermore, because the instrument pump seals and pistons only come in contact with deionized water, overall pump maintenance is significantly reduced. With eluent generation, a pair of electrodes is positioned with an ion-exchange membrane separating them; when a current is applied to the electrodes, electrolysis of water generates hydroxide at the cathode and hydronium at the anode. The ion-exchange membrane prevents the species from recombining into water, and allows a counterion from the Eluent Generation Cartridge to migrate across the membrane to form the eluent. The eluent concentration is varied by changing the applied current to generate eluent within a given range 0–100 mM or 0–200 mM (capillary IC). This entire process can be done without the use of extra pumps, fittings, valves or any moving parts.

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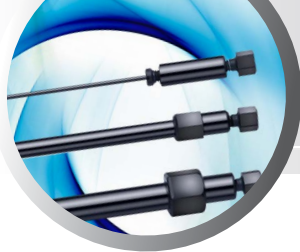
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# High-Pressure IC, 4 $\mu$ m Columns, Capillary IC

## High-Pressure Ion Chromatography

HPIC systems redefine the way ion chromatography is performed due to their continuous operation up to 5000 psi when configured as an RFIC system. High backpressure tolerance lets you increase flow rates to maximize your throughput while still benefiting from the advantages of electrolytic eluent generation and suppression. This feature allows the use of new high-efficiency 4  $\mu$ m particle-size columns which produce fast run times using 150 mm long columns and high resolution using 250 mm long columns.

## 4 $\mu$ m Particle Columns

Chromatographic separations using packed columns benefit from a high number of theoretical plates per column. The number of theoretical plates can be increased by packing smaller particles into the columns. Typically ion chromatography columns use resin particles ranging from 7–9  $\mu$ m in diameter. Recent developments in resin technology have allowed the use of 4  $\mu$ m resin particles in ion exchange columns. The benefits of columns packed with smaller particles include more efficient peaks, better resolution, faster run times, easier integration, and more reliable results.

## Capillary Ion Chromatography

Capillary IC takes performance to a whole new level while saving time and resources. Capillary IC systems use columns with internal diameters of 0.4 mm and typical flow rates of 10  $\mu$ L/min. At this rate, only 15 mL of water a day (5.2 L a year) is consumed allowing these systems to be left always on so that they are always ready to run samples.

The waste produced by a capillary IC system is dramatically reduced, compared to that of a system using 4 or 2 mm i.d. columns, which decreases disposal costs. When operated as a RFIC system, the eluent generation cartridge lasts for 18 months under continuous operation. Using eluent generation, only water flows through the pumps which greatly extends the life of seals and decreases maintenance costs.

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# Chromatography Data Systems

Software is more than just an essential component of a modern chromatography data system – it's often the most important factor in getting the desired results. Whether your needs are basic or complex – whether you use instruments from Thermo Fisher Scientific, or from other manufacturers, or both – there's a Thermo Scientific™ Dionex™ Chromeleon™ Chromatography Data System (CDS) software solution that's right for you.

An unparalleled suite of tools for rapid run start and automated, fast data processing:

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# Ion Chromatography and RFIC Systems

Which IC system is right for your application and budget? From basic starter-line to highly customizable high-pressure IC systems, feel confident that you are selecting quality Thermo Scientific Dionex products, support, and service from the IC technology innovator and leader.



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