

Determination of trace cations in concentrated acids using AutoNeutralization pretreatment and ion chromatography

Authors

Terri Christison and Jeff Rohrer
Thermo Fisher Scientific,
Sunnyvale, CA

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Introduction

Determination of cations in concentrated acid is important to many industries, including chemical, electronics, pharmaceutical, food, and beverage manufacturing, as well as industrial plating, environmental monitoring, and mining. Some cations can combine with anionic contaminants to form insoluble compounds that can cause plating and etching defects in plating and electronics manufacturing processes. Calcium in phosphoric acid has been shown to improve uranium extraction,¹ but calcium in phosphate fertilizers may contaminate soil.² Lithium, sodium, potassium, calcium, and magnesium ions play important roles in biological systems. Persons at risk from certain diseases must monitor their intake of these cations, such as potassium and sodium for cardiovascular disease³ and potassium for patients on dialysis from renal failure.⁴ The U.S. Food and Drug Administration (FDA) requires that these concentrations are reported on ingredient labels of food and beverages. Thus, it is important to determine cations in acid additives and acidic feed materials used for pharmaceutical, food, and beverage manufacturing.

Ion chromatography (IC) with suppressed conductivity detection is the preferred method for determining ionic species, especially at $\mu\text{g/L}$ and ng/L concentrations. Direct injection of concentrated acid samples overloads an IC column with the acid cation, resulting in poor chromatography that makes it difficult or impossible to quantify cations. Sample dilution reduces column overloading, but sacrifices cation determinations at $\mu\text{g/L}$ and ng/L concentrations.

The introduction of AutoNeutralization™ technology by Dionex in 1994, embodied in the Thermo Scientific™ Dionex™ SP10 AutoNeutralization™ module, eliminated the need to dilute concentrated acids to avoid column overloading, and thus allowed the determination of $\mu\text{g/L}$ and ng/L concentrations of anions and cations.⁵ This technique used multiple cycles through a cation neutralizer (Thermo Scientific™ Dionex™ CSRN™) to completely neutralize the acid in the sample.

The second embodiment of Dionex AutoNeutralization technology replaced the Dionex SP10 module with two 6-port valves and an external pump installed in a Thermo Scientific™ Dionex™ ICS-2500 system. This method, reported in the previous version of Thermo Scientific Application Note 94⁶ (AN 94), effectively neutralized strong acids with a single cycle. Trace anions in concentrated bases were determined in a similar way, with multiple passes through an anion neutralizer (Thermo Scientific™ Dionex™ ASRN™) (previous version of AN 93⁷). The method described in the most recent version of AN 93⁸ uses the dual pumps, detectors, and multiple valves of the Thermo Scientific™ Dionex™ ICS-3000 Reagent-Free™ IC (RFIC™) system to make configuration of the AutoNeutralization process easier. Using a technique coined Park and Neutralize, the concentrated base sample was pumped by Pump 2 of the Dionex ICS-3000 system through a 10-port valve and trapped in the neutralizer for a set time. The neutralized sample was released from the neutralizer and the trace anions were collected on the concentrator column, eluted by Pump 1 eluent, separated on the separation column, and detected by suppressed conductivity.

This Application Note replaces the previous version of AN 94⁶ and updates the method for determination of cations in concentrated acids using the dual capabilities of the Dionex ICS-3000 IC system. The AutoNeutralization

process is performed using the ICS-3000 IC system's dual pumps, detectors, and valves for easier configuration without the need for external pumps or valves. The system configurations, plumbing, valve operations, and program are described in this Application Note.

The acid concentrations described in this method do not need to be trapped in the neutralizer to effectively neutralize the acid. The acid is fully neutralized as it flows through the neutralizer and to the concentrator.

This method successfully determines trace cations from low- $\mu\text{g/L}$ to low- mg/L concentrations in 24% sulfuric, 25% phosphoric, and 10% hydrofluoric acids. Acids at concentrations above 30% have higher viscosities that cause longer loading times and result in lower reproducibilities. For concentrated acids greater than 30%, the Park and Neutralize technique can be used. AN 93⁸ describes this technique for the determination of trace anions in concentrated bases.

Experimental Equipment

- Dionex ICS-3000 RFIC system* consisting of:
 - DC Detector/Chromatography module with single temperature zone, AM Automation Manager (P/N 079833), 10-port valve (P/N 061962), one injection valve, a standard bore temperature stabilizer, and two CD Conductivity Detectors and Integrated Cells (P/N 079829)
 - DP Dual Pump module with degas module, Thermo Scientific™ Dionex™ IonPac™ GM-4 gradient mixer (P/N 049136), and Thermo Scientific™ Dionex™ IonPac™ CTC-1 trap column (9 × 24 mm, P/N 040192)
 - EG Eluent Generator module with Thermo Scientific™ Dionex™ EluGen™ EGC III MSA cartridge (P/N 074535), and Thermo Scientific™ Dionex™ Continuously Regenerated Cation Trap Column CR-CTC II, P/N 066262

* Equivalent or improved results can be achieved using the Thermo Scientific™ Dionex™ ICS-5000+ HPIC™ system.

- Dionex AS Autosampler with 10 mL sample tray and vial kit (P/N 055058), and 500 or 1,000 μL sample syringe
- Thermo Scientific™ Dionex™ Chromeleon™ Chromatography Management Software, version 6.8

- Tubing

- Black PEEK™ tubing (0.25-mm or 0.01-in. i.d., P/N 052306 for 5 ft) for liquid line connections for both systems and backpressure loops for the Thermo Scientific™ Dionex™ CSRS™ ULTRA II suppressor
- Yellow PEEK tubing (0.08-mm or 0.003-in. i.d., P/N 052301 for 5 ft) for system backpressure loops
- Green PEEK tubing (0.76-mm or 0.03-in. i.d., P/N 044077 per inch) for eluent waste lines
- Low pressure Teflon® (The Chemours Company) tubing (1.6-mm or 0.063-in. i.d., P/N 014157) for external water regenerant lines for the Dionex CSRN II neutralizer, and Dionex CSRS ULTRA II suppressor regenerant and degas waste lines
- 100 µL sample loop (P/N 42951)
- Thermo Scientific™ Dionex™ Suppressor External Regenerant Installation Kit (P/N 038018) with 4 L bottle for external water suppression
- Corning or other brand of similar quality (Corning P/N 431081 or VWR P/N 29186-199) 225-mL polystyrene sterile flasks for trace cation standards and samples

Reagents and standards

Use only ACS reagent grade chemicals for all reagents and standards.

- Deionized water, Type 1 reagent-grade, 18.2 MΩ·cm resistivity or better
- Calcium chloride, dihydrate (Fisher Scientific, P/N C79-500, 500 g)
- Thermo Scientific™ Dionex™ Combined Six Cation Standard-II, NIST traceable (P/N 046070)
- Lithium chloride (JT Baker®, Baker Analyzed®, P/N 2370-01)
- Magnesium chloride, hexahydrate (JT Baker, Baker Analyzed, P/N 2444-01)
- Potassium chloride (Fisher Scientific, P/N P217-500, 500 g)
- Sodium chloride, crystalline (JT Baker, Baker Analyzed, P/N 3624-01)

Conditions

Sample preparation: System 2

Trap Column: Dionex IonPac CTC-1, 9 × 24 mm (P/N 040192)

Flow Rate: 0.5 mL/min

Carrier: Degassed deionized water

Inj. Volume: 100 µL

Neutralization: Dionex CSRN II Cation Self-Regenerating Neutralizer, external water mode, 500 mA. (Replaced by Thermo Scientific™ Dionex™ CERS™ 500e Cation Electrolytically Regenerated Suppressor for External Water Mode (4 mm) P/N 302663)

Run Time: 1.4 min for AutoNeutralize and Concentrate (needs to be determined experimentally)

Typical System

Backpressure: ~800 psi

Analytical: System 1

Columns: Dionex IonPac CG16 guard (5 × 50 mm, P/N 057574) and Dionex IonPac CS16 analytical (5 × 250 mm, P/N 079805)

Flow Rate: 1.0 mL/min

Eluent: 28 mM methanesulfonic acid

Eluent Source: Dionex EluGen EGC II MSA

Temperature: 30 °C

Concentrator: Thermo Scientific™ Dionex™ IonPac™ TCC-ULP1, 5 × 23 mm (P/N 063783)

Detection: Suppressed Conductivity, Dionex CSRS ULTRA II Cation Self-Regenerating Suppressor, recycle mode, 82 mA

Typical

Background: <0.5 µS

Typical System

Backpressure: ~2200 psi

Typical Noise: <0.5 nS

Total Run Time: 35 min

Rinse Program:

System 2: Same conditions without Concentrate

System 1: Bypassed

Inj. Valve 1: Inject position

Total Run

Time: 10 min

Samples

- 24% (w/w) Sulfuric acid prepared from 96% (w/w) JT Baker ULTREX™ II Ultrapure concentrated sulfuric acid (P/N 6902-05)
- 25% (w/w) Phosphoric acid prepared from 86% (w/w) JT Baker, Baker Analyzed concentrated phosphoric acid (P/N 0260)
- 10% (w/w) Hydrofluoric acid prepared from 49% (w/w) concentrated hydrofluoric acid (Ashland)

Preparation of solutions and reagents

Caution: Concentrated acids are highly corrosive and can cause severe burns to exposed skin, eyes, and lungs. Review the Material Safety Data Sheets for handling, personal protective precautions, exposure limits, incompatibility, and waste disposal. Acids have strong exothermic reactions with water. Sulfuric acid has a violent reaction with water and can sometimes boil over during dilution. When diluting acids in water, always add acid to water. Add the acid slowly and gently swirl the solution to prevent hot spots. Wear safety glasses and gloves. Face shields are also recommended during dilution, especially with sulfuric and hydrofluoric acids. Hydrofluoric acid is highly toxic and can cause severe burns and bone damage. Extra precautions should be used when handling hydrofluoric acid: have the calcium gluconate antidote on hand, use gloves specified for hydrofluoric acid, wear double gloves, and wear a face shield. Take care to clean the work area to prevent accidental exposure.

Preparing 1 M (2 N) sulfuric acid conditioning solution

The 1 M sulfuric acid conditioning solution is used to clean the Dionex IonPac CTC-1 trap column and the Dionex CSRN II neutralizer. To prepare the 1 M sulfuric acid conditioning solution, clean a 25-mL disposable graduated plastic pipette with deionized water by filling it with deionized water and dispensing it to waste. Pipette 56 mL of concentrated sulfuric acid into 500 mL of Type 1 deionized water in a 1 L volumetric flask. Cool to room temperature. Bring to volume with Type I deionized water. Transfer the conditioning solution to a 2 L eluent bottle, seal the bottle, and attach the bottle to the System 1 Eluent B line.

Preparing sulfuric acid rinse solution used for calibration

To prepare a 0.5 M (1 N) sulfuric acid solution from concentrated sulfuric acid, pipette 28 mL of concentrated sulfuric acid into 500 mL of deionized water in a 1 L volumetric flask. Cool to room temperature, then bring to volume with Type I deionized water. Transfer the sulfuric acid rinsing solution to a pre-cleaned 1 L HDPE bottle.

Preparing stock standards

Stock standards can be prepared from certified reagents or purchased commercially as certified solutions, such as Dionex Combined Six Cation Standard-II (50 mg/L lithium, 200 mg/L sodium, 250 mg/L ammonium and magnesium, and 500 mg/L potassium and calcium). To prepare individual stock solutions of 1,000 mg/L of lithium, sodium, potassium, magnesium, and calcium, weigh the amount of reagent grade compound (Table 1) into a 125 mL HDPE sample bottle and dilute with deionized water to 100.00 g total weight. Shake each stock solution to fully dissolve the reagent.

Table 1. Amount of compound used to prepare 100 mL of 1,000 mg/L individual stock solutions.

Cation	Compound	Mass (g)
Lithium	Lithium chloride (LiCl)	0.061
Sodium	Sodium chloride (NaCl)	0.254
Potassium	Potassium chloride (KCl)	0.191
Magnesium	Magnesium chloride (MgCl ₂ • 6H ₂ O)	0.837
Calcium	Calcium chloride (CaCl ₂ • 2H ₂ O)	0.367

Preparing intermediate and working standards

To determine cations in concentrated acid samples, prepare separate 1.0 mg/L intermediate standards from lithium, sodium, potassium, magnesium, and calcium stock solutions in water. Pipette 100 µL each of the 1,000 mg/L individual stock solutions into separate 120 mL polypropylene bottles. Dilute with deionized water to 100.00 g total weight. Alternatively, dilute the Dionex Combined Six Cation Standard-II 200-fold by pipetting 500 µL of the standard into a 120 mL polypropylene bottle and diluting with deionized water to 100.00 g total weight.

Prepare the Corning 225 mL polystyrene sterile flasks for the µg/L standards two days or more prior to the standard preparation. Rinse each flask five times with deionized water, fill it to the top with deionized water, and let it soak overnight. Repeat this daily until flasks are needed for the µg/L standards. See Thermo Scientific Application Update 142 for additional precautions needed for determination of µg/L ion concentrations.⁹

Prepare working standards in the calibration ranges of the samples by diluting the intermediate standards appropriately with deionized water. In this Application Note, 10 standards were prepared from the intermediate standard of the Dionex Combined Six Cation Standard-II. Final dilutions were 200,000-, 100,000-, 40,000-, 20,000-, 8,000-, 4,000-, 1,600-, 800-, 400-, and 200-fold. Concentrations of lithium ranged from 0.25 to 250 µg/L (0.25, 0.5, 1.25, 2.5, 6.25, 12.5, 31.0, 62.5, 125, and 250 µg/L), sodium from 1.0 to 1,000 µg/L (1.0, 2.0, 5.0, 10, 25, 50, 125, 250, 500, and 1,000 µg/L), potassium and calcium from 2.5 to 2,500 µg/L (2.5, 5.0, 12.5, 25, 62.5, 125, 312.5, 625, 1,250, and 2,500 µg/L), and magnesium from 1.25 to 1,250 µg/L (1.25, 2.5, 6.25, 12.50, 31.25, 62.5, 156.3, 312.5, 625, and 1,250 µg/L).

Preparing samples

Caution: Concentrated acids are highly corrosive and can cause severe burns to exposed skin, eyes, and lungs. Review the Material Safety Data Sheets and the caution statement in the Preparing the Conditioning Solution section.

To prepare samples of 24% (w/w) sulfuric acid solution from 96% (w/w) 12.5 M sulfuric acid and degassed deionized water:

1. Clean a 25 mL transfer pipette. Rinse three times with deionized water and shake out excess.
2. Clean a 225 mL polystyrene flask as described in Preparing Intermediate and Working Standards.
3. Add 100.0 g of degassed deionized water to 225 mL flask.
4. Slowly pipette 50.0 g of 96% sulfuric acid into flask..
5. Swirl gently.

6. Dilute with degassed deionized water to 200.0 g total weight.
7. Cap flask and gently swirl until thoroughly mixed.
8. Cool to room temperature before testing.

Prepare the 25% (w/w) phosphoric acid solution similarly by pipetting 58.5 g of 85.5% (w/w) phosphoric acid into 100.0 g of deionized water then diluting to 200.0 g total weight.

Prepare the 10% (w/w) hydrofluoric acid solution similarly by pipetting 20.4 g of 49% (w/w) hydrofluoric acid into 50.0 g of deionized water then diluting to 100.0 g total weight.

Preparing spike recovery samples in water and reagents

To prepare spike recovery samples in sulfuric acid, phosphoric acid, and hydrofluoric acid solutions, add 1,000 µL of a dilution of the Dionex Combined Six Cation Standard-II that is 100-times the desired concentration to 99 g of the sample. For example, to prepare a 40,000-fold dilution of the standard (1.25 µg/L lithium, 5.0 µg/L sodium, 12.5 µg/L potassium and calcium, and 6.3 µg/L magnesium) spiked into 24% sulfuric acid, slowly pipette 1,000 µL of a 400-fold dilution of Dionex Combined Six Cation Standard-II into a 225 mL flask containing 99 g of 24% sulfuric acid solution. Cap the flask and shake gently until it is thoroughly mixed. Prepare the 25% phosphoric acid and 10% hydrofluoric acid spike recovery samples and the 24% sulfuric acid robustness experiment samples in a similar manner.

Theory for cation self-regenerating neutralizer Dionex CSRN II operation

The AutoNeutralization pretreatment is based on the neutralization of the strong acid to low conductivity water through the high capacity, electrolytic Cation Self-Regenerating Neutralizer (Dionex CSRN II) device. The Dionex CSRN II device neutralizes the strong acid by exchanging the anion of the acid with electrolytically generated hydroxide. The neutralized sample, predominantly water with trace levels of cations, is collected on a Dionex IonPac TCC-ULP1 concentrator column and injected after 1.4 min. The neutralization process is thoroughly discussed in the Dionex CSRN II product manual.¹⁰

System setup

Setup for the individual modules, components, and system is thoroughly described in the operator's and installation manuals^{11,12} for the Dionex ICS-3000 chromatography system, and in the Chromeleon "Help" menus.

The sample loading and neutralization by the Dionex CSRN II neutralizer occurs on System 2. The remaining trace cations in the neutralized sample are concentrated, injected, separated, and detected by suppressed conductivity on System 1.

System 2: sample preparation

The sample preparation part of the AutoNeutralization application includes the Dionex AS Autosampler, Pump 2 with Dionex IonPac CTC-1 (9 × 24 mm) trap column, Dionex AM Automation Manager with a 10-port, high pressure valve (AM-HP1), Dionex CD 2 Conductivity Detector, and a Dionex CSRN II neutralizer. The Dionex CD 2 detector monitors sample injection, the sample after it has passed through the neutralizer, and the cleanliness of the carrier water. It is not used for quantification.

To set up System 2:

1. Connect black PEEK (0.254 mm or 0.010 in. i.d.) tubing from Pump 2 to the Dionex IonPac CTC-1 trap column¹³ and from trap column to inside of DC.
2. Add a 30 cm (12 in.) length of yellow (0.076 mm or 0.003 in. i.d.) PEEK tubing to make an ~800 psi backpressure loop.
3. Prime Pump 2.
4. Verify that System 2 pressure is between 800 and 900 psi. If needed, remove or add yellow PEEK backpressure tubing to adjust system pressure between 800 and 900 psi.
5. Flush Dionex IonPac CTC-1 trap column with deionized water for 20 min.
6. Open upper chamber door of the Dionex ICS-3000 DC module and locate Automation Manager and Dionex AM-HP1 10-port high pressure valve 1, (top left corner of Automation Manager).

7. Plumb System 2 with black PEEK tubing according to Figure 1:

- a. Add black PEEK tubing from yellow backpressure tubing to Port 1 on Dionex AM-HP1 valve.
- b. Install 100 μ L loop (~49 cm orange PEEK tubing) in Ports 2 and 4.
- c. Connect 6.5 to 7 cm (2.6 to 2.8 in.) of black PEEK tubing from Port 3 to Port 7 to connect sample load side of the valve (Ports 1–5) to neutralization side of the valve (Ports 6–10).
- d. Connect Dionex AS Autosampler injection port tubing into Port 5.
- e. Connect one end of green (0.76 mm or 0.30 in. i.d.) PEEK tubing into Port 6. Direct other end into waste container.
- f. Connect 35 to 37 cm (13.8 to 14.6 in.) of black PEEK tubing from Port 10 to CD 2 Conductivity Detector inlet.

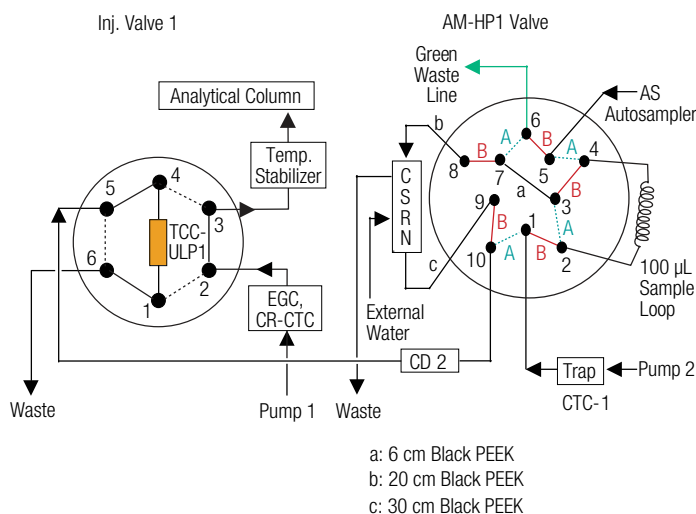


Figure 1. Valve configuration for detection of cations in concentrated acids using AutoNeutralization.

Setting Up the Dionex CSRN II neutralizer

Hydrate the Dionex CSRN II neutralizer for 20 min, according to the QuickStart procedure in the neutralizer product manual.¹⁰

Because the Dionex CSRN II membranes are highly aminated, it is important to condition the neutralizer with 1 M sulfuric acid to reduce the amines to a stable background concentration.

1. Connect black PEEK tubing from Pump 2 to Eluent In on neutralizer.
2. Connect black PEEK tubing from Eluent Out to Regen In with a 10-32 to ¼" fitting.
3. Direct the Regen Out tubing to waste.
4. Pump 100 mL of 1 M sulfuric acid (Eluent B) at 1 mL/min, followed by 300 mL of deionized water (Eluent A). Do not turn on neutralizer.
5. Reroute Eluent Out tubing from neutralizer through CD 2 to Regen In. Flush neutralizer overnight (or until the background conductivity is <2 µS) with deionized water at 1 mL/min.

To install the Dionex CSRN II neutralizer:

1. Disconnect tubing used to condition neutralizer and install neutralizer in the suppressor slot for System 2.
2. Connect 21–23 cm (8.3–9.0 in.) of black PEEK tubing from Port 8 of Dionex AM-HP1 valve to Eluent In on the CSRN II neutralizer, and 44–46 cm (17.3–18.1 in.) of tubing from Eluent Out to Port 9.
3. Install external water regenerant on neutralizer using Dionex Suppressor External Regenerant Installation Kit.
4. Connect Teflon tubing (1.6 mm or 0.063 in. i.d.) to Regen In of Dionex CSRN II neutralizer and from Regen Out to waste.
5. Adjust flow rate to 3–5 mL/min by adjusting the pressure on the external water regenerant system.
6. Determine flow rate by collecting water from the neutralizer into a 25 mL graduated cylinder for 5 min. Total volume should be 15–25 mL.

It is critical that the external water regenerant is flowing through the Dionex CSRN II neutralizer before the pump and the neutralizer are turned on. When the pump is turned off, the regenerant should also be turned off. It is also critical that the backpressure to the Dionex CSRN II neutralizer is <100 psi.

1. Measure backpressure on the neutralizer channel by disconnecting the line out of the eluent out port of the Dionex CSRN II neutralizer ("B").
2. Replace line and measure system pressure with the line in ("A"). The difference "A minus B" should be <100 psi.
3. Install 18–20 cm (7.1–7.9 in.) of black PEEK tubing from CD 2 outlet to Port S (5) in Inj. Valve 1 located in the lower chamber of the DC. System 2 is now connected to System 1.

To turn on the neutralizer, open the DC chamber and verify by sight that the external water is flowing through the neutralizer before turning on the pump. Select the Detector Compartment tab (System 2) in the Chromeleon Panel (Default Panel Tabset), select SRS as the suppressor type, and enter 500 mA for the current.

Setting Up the Dionex AS Autosampler

To reduce contamination, install a new PEEK needle assembly prior to starting this application. The needle may need to be replaced more often than necessary for most applications, to prevent contamination.

To set up the Dionex AS Autosampler:

1. On the Dionex AS Autosampler module, under Module Setup Menu/Plumbing Configuration, enter sample loop volume of 100 µL or the calibrated volume. Press the enter key.
2. Verify that Sample Syringe and Prep Syringe Volumes are correct. For this application, use a 500 or 1,000 µL sample syringe.
3. For samples with extreme concentration differences, set cut volume to 15 µL (under Module Setup Menu/System Parameters).

4. Put autosampler in Normal mode (Use the ▼ key to select Normal for the Sample Mode. Press Enter.)
5. Set the Flush and Prime volumes on the Chromeleon Panel.
 - a. Enter 300 µL (or 3 times the sample loop) for Flush volume.
 - b. Enter a large volume for the Prime volume (e.g., 5000 µL).
6. The injection volume, entered in the Sequence, is the same as sample loop volume.
 - a. For a full loop injection, the volume removed from the vial is four times the injection volume plus the cut volume.
7. To ensure accurate sample volumes, Prime and Flush the syringes (Chromeleon Panel, Sampler Commands)
8. Change the contents of the flush containers daily.

More information can be found in the Dionex AS Autosampler operator's manual.¹⁴

System 1: Analytical

System 1 analyzes the neutralized sample and includes Pump 1, eluent generation (EG 1 with degas and Dionex CR-CTC trap column), injection (Inj. Valve 1), and concentration (Dionex IonPac Trace Cation Concentrator column, TCC-ULP1, 5 × 23 mm), separation (Dionex IonPac CS16, 5 × 250 mm analytical column and CG16, 5 × 50 mm guard column), and suppressed conductivity detection (Dionex CSRS ULTRA II suppressor). The neutralized sample is moved from the Dionex CSRN II neutralizer into the conductivity detector in System 2 (CD 2), through Inj. Valve 1 in the DC, and into the concentrator column. Then the concentrated neutralized sample is moved from the concentrator column onto the Dionex IonPac CS16 column set.

To set up System 1, install the gradient mixer (GM-4), Dionex EGC II MSA cartridge, CR-CTC trap column, CSRS ULTRA II suppressor, columns, and backpressure loops for the suppressor and the eluent generator.

1. Connect black PEEK (0.254 mm or 0.010 in. i.d.) tubing from Pump 1 to gradient mixer (GM-4) and from gradient mixer into EG module.
2. Install and condition Dionex EGC II MSA cartridge, hydrate and flush Dionex CR-CTC trap column, and install backpressure loop according to the start up instructions in the Dionex ICS-3000 operator's manual.¹¹
3. Install the Dionex CR-CTC trap column after the cartridge and before the degas module and the Port P of Inj. Valve 1.
4. Install backpressure loop after the Dionex CR-CTC trap column and before Port P of Inj. Valve 1 (Figure 1). The ports of Inj. Valve 1 are labeled for clarity according to function: P for pump (Position 2), L for loop (Positions 1 and 4), C for column (Position 3), S for sample (Position 5), and W for waste (Position 6).
5. If a concentrator column is used in place of the sample loop, as in this application, it is necessary to distinguish between Position 1 (L) and Position 4 (L) because the sample is loaded and injected from Port 4. Port 4 (L) is located between Port C and Port S.
6. Install the Dionex IonPac TCC-ULP1 concentrator column in Ports L (1 and 4) according to the product manual instructions.¹⁵
 - a. Position concentrator column closer to Port 4 (between Port C and Port S) than Port 1 with flow arrow on the concentrator pointing from Port 1 to Port 4.
 - b. Install with a minimal amount of black PEEK tubing from column outlet to Port 4.
 - c. Connect inlet of concentrator column to Port 1.
7. Install one end of the black PEEK tubing from temperature stabilizer 1 into Port C and install the other end into guard and analytical columns according to their product manuals.¹⁶
8. Connect green PEEK tubing to Port W and direct other end to waste.

9. Measure backpressure directly from the pump to the cell.
10. Install backpressure loop on “cell out” position of conductivity cell and measure backpressure. The difference of these two measurements is the backpressure due to the cell and backpressure loop. It must be between 30 to 40 psi to prevent suppressor damage.
11. Follow the QuickStart instructions¹⁷ to hydrate and install the Dionex CSRS ULTRA II suppressor.
12. Install suppressor in recycle mode in System 1 suppressor slot between columns and CD 1.
13. Set Pump 1 flow rate at 1.0 mL/min and prime the pump.
14. Adjust system pressure to ~2200 psi by adding or removing yellow PEEK tubing to the system backpressure loop.

Configuration

1. Using the Chromeleon Server Configuration program, create one timebase and add the Dionex ICS-3000 modules and AS Autosampler.¹⁴
2. Under DC properties, assign the Dionex CSRS II suppressor to System 1 and the Dionex CSRN II neutralizer to System 2.
3. Rename the Dionex CSRN II neutralizer as Neutralizer, for convenience and clarity.
4. Verify that the boxes in front of the two high pressure valves (AM-HP1 and Inj. Valve 1) are checked and that no other valves are checked.
5. Assign control of Dionex AM-HP1 valve to Dionex AS Autosampler, and control of Inj. Valve 1 to DC.

6. Verify the Dionex EGC II cartridge is listed on System 1 and is linked to Pump 1.
7. Under AS Autosampler properties, verify Sample Overlap box is checked. Sample Overlap reduces total sample run time.

Creating the program

Use Chromeleon Wizard, the Conditions section, and valve commands in Table 2 to create a program for AutoNeutralization and a rinse program for calibration samples. To enter the suppressor type for the Dionex CSRN II neutralizer, select SRS-MPIC. Set the current at the maximum level (500 mA). The Dionex CSRS ULTRA II, 4 mm is listed under suppressor types. Select it and set the current for 41 mA. Enter the valve commands for Inj. Valve 1 and the AM-HP1 valves in the Relay & State Devices Option section.

In the program review mode, verify the valve timing and the gradient timing. Remove any Duration = number on the valve commands by inserting a semi-colon in front of Duration. Save program.

Hydroxide in the neutralizer can create insoluble compounds and cause alkali metals in water solutions to precipitate. To avoid alkaline-earth metal precipitation during calibration, injections of retention time standards must be alternated with 10-min runs of 24% sulfuric acid. Rinse the neutralizer with acid by injecting the sulfuric acid solution 20 min prior to the standard. To rinse only the neutralizer, Inj.Valve 1 is positioned in Inject mode at time = zero and remains in Inject throughout the 10 min run. The 24% sulfuric acid flows through the Dionex AM-HP1 valve and neutralizer in the same way as the samples but bypasses the concentrator, guard, and analytical columns to waste. Another rinse should be performed after each standard run within 20 min of injection.

Table 2. Chromeleon wizard valve commands to control the injection valve and the Dionex AM-HP1 valves.^a

Time (min)	Valve	Position	Action
0	AM-HP1	B	The sample is neutral-ized as it passes through the CSRN II
0	Inj. Valve 1	Load ^b	The concentrator is positioned to collect the neutralized acid sample
1.4	Inj. Valve 1	Inject	The concentrated neu-tralized acid sample is injected

^aTo neutralize for a longer period, as with acids >30% concentration, refer to Table 4 in AN 93.⁸

^bIn the Rinse program, set to Inject to bypass the analytical column.

Results and discussion

This application uses both systems on the Dionex ICS-3000 IC system. Before discussing the experiments, it is helpful to review the flow path of the Dionex AM-HP1 valve. The valve has two states: A and B. In sample load (Figure 2) the valve is in state A. The autosampler loads the sample onto the sample loop, and the water carrier by-passes the Dionex CSRN II neutralizer and sample loop.

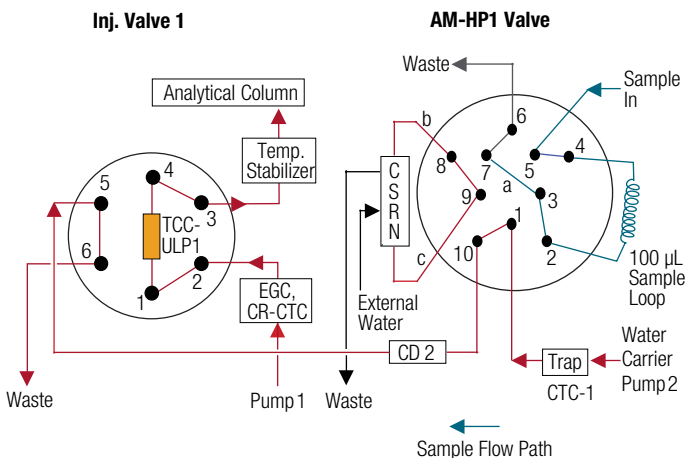


Figure 2. The acid sample is loaded into the 100 µL sample loop [AM-HP1 Valve (A)].

At time zero, the sample is injected as the Dionex AM-HP1 valve switches to state B (Figure 3) and the water carrier flushes the sample from the sample loop. The valve remains in state B as the sample is carried through the neutralizer, where the acid is neutralized and the cations are converted to their base forms. The neutralized sample passes through CD 2 to Port 5 in the System 1 Inj. Valve 1, and is collected for 1.4 min on the concentrator in the load position. After concentrating the cations, the neutralized and concentrated sample is injected (Inj. Valve 1 to Inject at 1.4 min) to the analytical system (Figure 4) and the data collection is started.

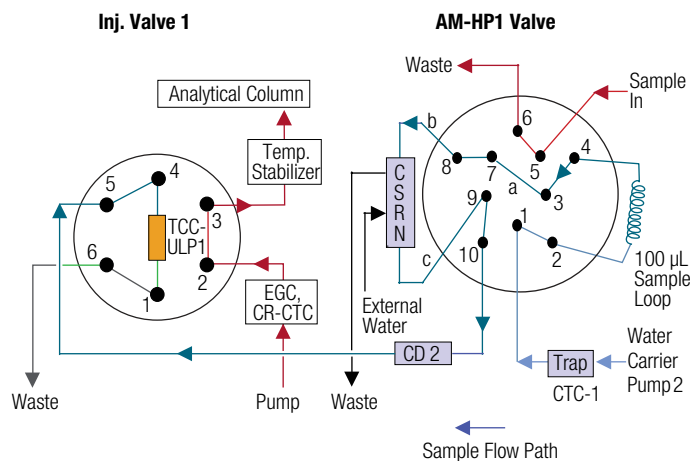


Figure 3. The acid sample is injected and neutralized [AM-HP1 Valve (B)], and collected and concentrated on the concentrator column (Injection Valve 1 in load position).

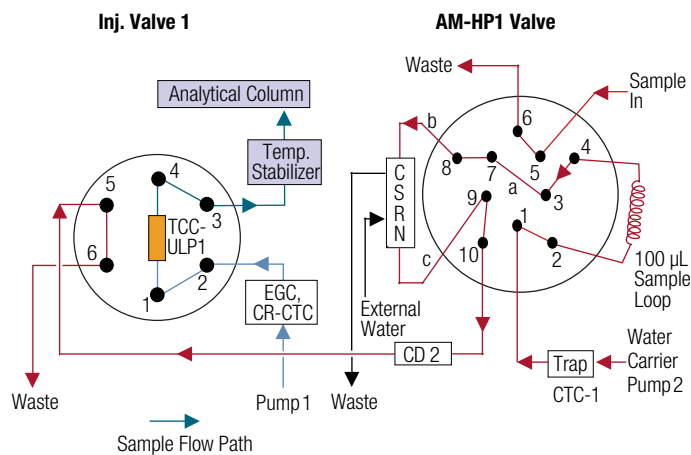


Figure 4. The neutralized and concentrated sample is eluted from the concentrator column (Injection Valve 1 in inject position) on to the analytical column.

To determine the system blank, deionized water was injected and analyzed until the background stabilized. The chromatogram of this system blank (Figure 5) had background concentrations of methylamine, dimethylamine, trimethylamine, and ammonium, and approximate concentrations of 1.73 ± 0.10 µg/L sodium, and 0.41 ± 0.08 µg/L potassium.

Sample Prep.

Trap Column: Dionex IonPac CTC-1,
9 × 24 mm
Carrier: Deionized water
Carrier Flow Rate: 0.5 mL/min
Inj. Vol.: 100 µL
Temperature: 30 °C
Neutralization: AutoNeutralize,
Dionex CSRN II,
external water mode,
500 mA

Analytical

Column: Dionex IonPac CG16,
CS16, 4 mm
Eluent: 28 mM Methanesulfonic acid
Eluent Source: Dionex EGC II MSA
Flow Rate: 1.0 mL/min
Temperature: 30 °C
Detection: Suppressed conductivity,
Dionex CSRS ULTRA II,
recycle mode, 82 mA
Concentrator: Dionex IonPac TCC-U1P1

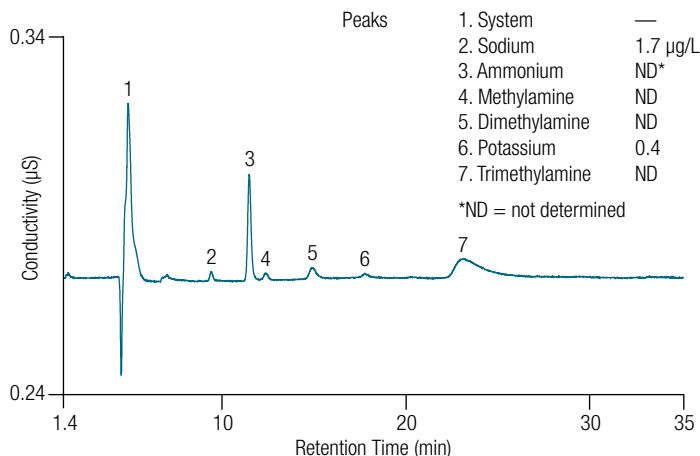


Figure 5. Typical water blank.

Cation retention times were determined in separate 5 µg/L standards and triplicate injections of an 8000-fold dilution of the Dionex Combined Six Cation Standard-II in water (2.5 µg/L lithium, 25 µg/L sodium, 12.5 µg/L ammonium, 62.5 µg/L potassium and calcium, and 31.3 µg/L magnesium). Each standard injection was preceded and followed by an injection of a 24% sulfuric acid rinse solution. See the Creating a Program section for more information. The 8,000-fold dilution of the Dionex Combined Six Cation Standard-II showed all cations elute within 35 min (Figure 6). Methylamine, dimethylamine, and trimethylamine were fully separated from the standard cations.

Method qualification

The AutoNeutralization method was qualified in water and 24% (w/w) sulfuric acid solutions prior to determining trace cations in other concentrated acid samples. The linearity of five cations over a 1000-fold concentration range, typical noise, and method detection limits (MDLs) were determined in water solutions. Reproducibilities, spike recoveries, and 5-day robustness were determined in 24% sulfuric acid solutions.

The linearity of peak response was determined for lithium, sodium, potassium, magnesium, and calcium with three replicates each of the 10 standards from

Sample Prep.

Trap Column: Dionex IonPac CTC-1,
9 × 24 mm
Carrier: Deionized water
Carrier Flow Rate: 0.5 mL/min
Inj. Vol.: 100 µL
Temperature: 30 °C
Neutralization: AutoNeutralize,
Dionex CSRN II,
external water mode,
500 mA

Analytical

Column: Dionex IonPac CG16,
CS16, 4 mm
Eluent: 28 mM Methanesulfonic acid
Eluent Source: Dionex EGC II MSA
Flow Rate: 1.0 mL/min
Temperature: 30 °C
Detection: Suppressed conductivity,
Dionex CSRS ULTRA II,
recycle mode, 82 mA
Concentrator: Dionex IonPac TCC-U1P1

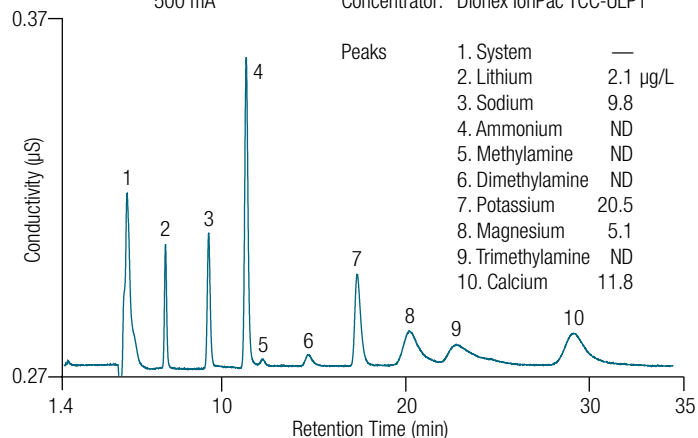


Figure 6. Cation standard in water using AutoNeutralization.

the 200,000-fold to 200-fold dilutions of the Dionex Combined Six Cation Standard-II. The peak response to concentration was measured for lithium from 0.25 to 2 50 µg/L, sodium from 1.0 to 1,000 µg/L, potassium and calcium from 2.5 to 2,500 µg/L, and magnesium from 1.25 to 1,250 µg/L. A rinse sample of 24% sulfuric acid was injected before each standard injection using the 10 min rinse program. The calibration results showed good linearities over these concentration ranges: $r^2 > 0.999$, (0.9993, 0.9996, 0.9995, 0.9993, and 0.9992 for lithium, sodium, potassium, magnesium, and calcium, respectively). The system blank had variable amounts of ammonium that affected the calibration plot. Ammonium can not be quantified with this application.

The noise was determined over five 90 min runs, when water was injected, by measuring the noise in 1-min intervals from 45 to 90 min. The noise value determined by this experiment was 0.29 ± 0.06 nS ($n = 5$). The method detection limit (MDL) is defined as three times the noise level, 0.86 ± 0.18 nS. For this application, the MDL concentrations for the five cations in sulfuric acid ranged from 0.06 to 2.0 µg/L (Table 3). The lithium, sodium, potassium, magnesium, and calcium peaks of a 100,000-fold dilution of the Dionex Combined Six Cation Standard-II (0.5, 2.0, 5.0, 2.5, and 5.0 µg/L, respectively) had a signal to noise ratio of 33.4, 45.6, 22.6, 7.7, and 7.7. These MDLs are comparable to the results reported in the previous version of AN 94.

Table 3. MDLs and recoveries of lithium, sodium, potassium, magnesium, and calcium in 24% sulfuric acid.^a

Cation	MDL (µg/L) ^b	Unspiked (µg/L)	Added (µg/L)	Recovery (%)	RSD
Lithium	0.06	<0.06	13.5	101.9	0.3
Sodium	2.0	7.2 ± 0.3	5.0	101.8	1.1
Potassium	0.7	3.3 ± 0.2	12.5	96.3	1.3
Magnesium	0.8	6.0 ± 0.4	6.3	94.1	0.7
Calcium	1.0	2.0 ± 0.1	12.5	100.1	0.9

^an = 5 injections.

^bMDL was determined as three times the signal to noise ratio (0.29 ± 0.06 nS, n = 5).

Spike recovery was determined for 5.0 µg/L sodium, 12.5 µg/L potassium and calcium, and 6.3 µg/L magnesium in 24% sulfuric acid by adding a 400-fold diluted solution of the Dionex Combined Six Cation Standard-II as 1% of the total volume (40,000-fold dilution). The recovery was good: 94.2 to 103.6% (Table 3). The chromatogram of the 24% sulfuric acid showed good chromatography (Figure 7).

The reproducibility and robustness of the AutoNeutralization method was determined over 240 injections, ~ 120 h. The retention times and peak areas of the five cations were determined for 13.5 µg/L

lithium, 54.0 µg/L sodium, 135.1 µg/L potassium and calcium, and 67.6 µg/L magnesium in 24% sulfuric acid by adding a diluted solution of the Dionex Combined Six Cation Standard-II as 1% of the total volume (3,700-fold dilution). Deionized water injections were inserted between the sample groups of seven replicate injections. The results show that the chromatography was good (Figure 8) and the retention times and peak areas were stable over 128 h of the experiment, with the exception of peak areas for magnesium. The peak areas for magnesium were stable during 200 injections (0.081 ± 0.002 µS-min) but declined during the later injections.

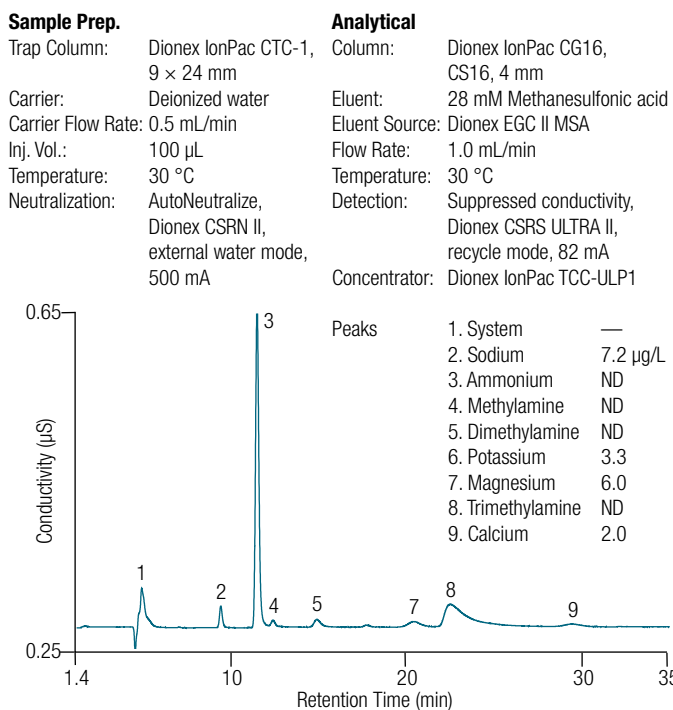


Figure 7. Cations in a 24% sulfuric acid sample using AutoNeutralization.

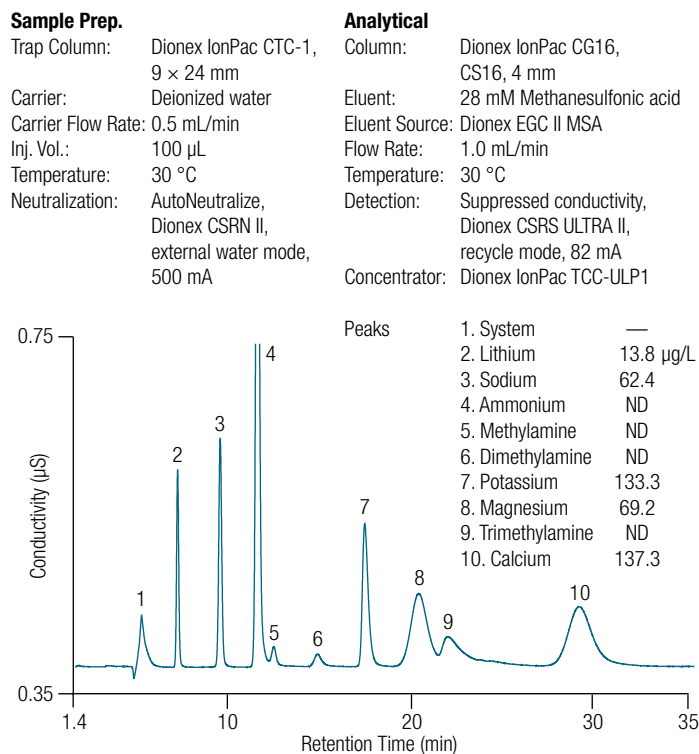


Figure 8. Trace cations in 24% sulfuric acid spiked with the Dionex Combined Six Cation Standard-II.

The retention time reproducibilities for lithium, sodium, potassium, magnesium, and calcium over 240 injections were 6.97 ± 0.01 , 9.27 ± 0.02 , 17.1 ± 0.1 , 20.1 ± 0.2 , and 28.8 ± 0.3 min respectively (Figure 9). The retention times for lithium, sodium, and potassium had low RSDs (<0.3%) for retention times over the 240 injections. The late eluting magnesium and calcium ions had higher RSDs (1.6, and 2.3%, respectively) over the five day robustness test.

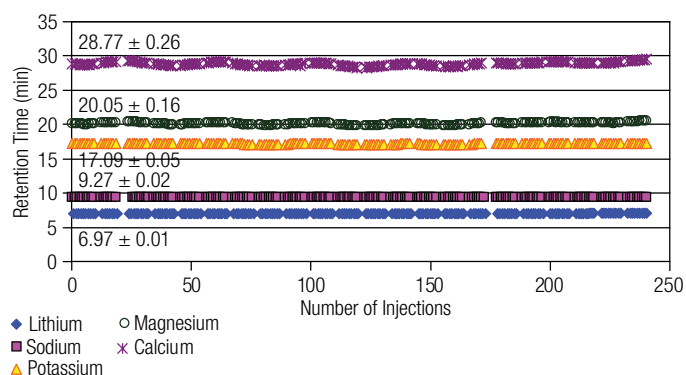


Figure 9. Retention time reproducibilities for cations in 24% sulfuric acid spiked with the Dionex Combined Six Cation Standard-II. Temperature control was disabled during these experiments, causing minor periodic fluctuations in magnesium and calcium retention times.

The peak areas were reproducible (0.3–3.5% RSD) and stable (0.030 ± 0.001 , 0.043 ± 0.001 , 0.061 ± 0.001 , and 0.094 ± 0.001 $\mu\text{S}\cdot\text{min}$, for lithium, sodium, potassium, and calcium, respectively, Figure 10).

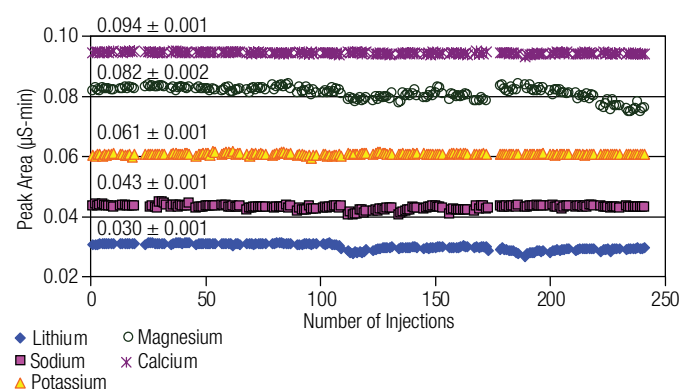


Figure 10. Peak area reproducibilities for cations in 24% sulfuric acid spiked with the Dionex Combined Six Cation Standard-II.

Determination of trace cations in 25% phosphoric and 10% hydrofluoric acids

The AutoNeutralization method was applied to determine trace cations in 25% phosphoric acid and 10% hydrofluoric acid. Spike recoveries of cations were determined for both solutions by adding diluted solutions of the Dionex Combined Six Cation Standard-II, as 1% of the total volume. The 25% phosphoric acid solution (Figure 11) had moderately high concentrations of potassium and magnesium (67, 151 $\mu\text{g}/\text{L}$), and high concentrations of sodium and calcium (459, 818 $\mu\text{g}/\text{L}$) (Table 4). Spike recoveries of a 1,000-fold dilution of the Dionex Combined Six Cation Standard-II (50, 200, 500, 250, and 500 $\mu\text{g}/\text{L}$ of lithium, sodium, potassium, magnesium, and calcium, respectively), in 25% phosphoric acid (Figure 12) were good: 100.6–109.5% (Table 4).

Sample Prep.

Trap Column: Dionex IonPac CTC-1, 9×24 mm
 Carrier: Deionized water
 Carrier Flow Rate: 0.5 mL/min
 Inj. Vol.: 100 μL
 Temperature: 30 $^{\circ}\text{C}$
 Neutralization: AutoNeutralize, Dionex CSRN II, external water mode, 500 mA

Analytical

Column: Dionex IonPac CG16, CS16, 4 mm
 Eluent: 28 mM Methanesulfonic acid
 Eluent Source: Dionex EGC II MSA
 Flow Rate: 1.0 mL/min
 Temperature: 30 $^{\circ}\text{C}$
 Detection: Suppressed conductivity, Dionex CSRS ULTRA II, recycle mode, 82 mA
 Concentrator: Dionex IonPac TCC-ULP1

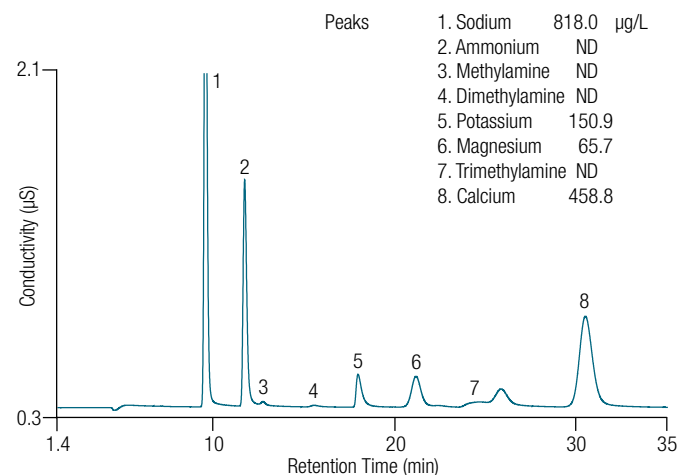


Figure 11. Trace levels of cations in 25% phosphoric acid using AutoNeutralization.

Sample Prep.

Trap Column: Dionex IonPac CTC-1,
9 × 24 mm
Carrier: Deionized water
Carrier Flow Rate: 0.5 mL/min
Inj. Vol.: 100 µL
Temperature: 30 °C
Neutralization: AutoNeutralize,
Dionex CSRN II,
external water mode,
500 mA

Analytical

Column: Dionex IonPac CG16,
CS16, 4 mm
Eluent: 28 mM Methanesulfonic acid
Eluent Source: Dionex EGC II MSA
Flow Rate: 1.0 mL/min
Temperature: 30 °C
Detection: Suppressed conductivity,
Dionex CSRS ULTRA II,
recycle mode, 82 mA
Concentrator: Dionex IonPac TCC-ULP1

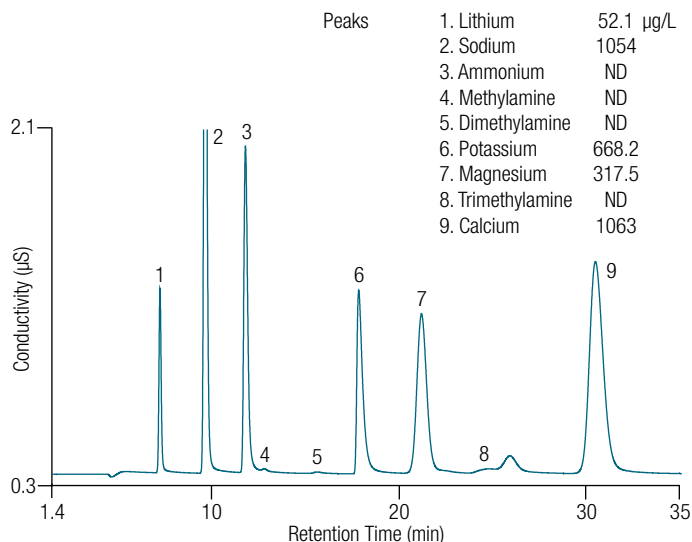


Figure 12. Trace levels of cations in 25% phosphoric acid spiked with the Dionex Combined Six Cation Standard-II.

The 10% hydrofluoric acid solution (Figure 13) had low concentrations of sodium, potassium, and calcium (<10 µg/L), and a moderately high concentration of magnesium (114.2 ± 2.6 µg/L). Spike recoveries of a 13,300-fold dilution of the Dionex Combined Six Cation Standard-II (3.8, 15, 38, 19, and 38 µg/L of lithium, sodium, potassium, magnesium, and calcium, respectively) in 10% hydrofluoric acid (Figure 14) were good: 90.7 to 106.1% (Table 4).

Sample Prep.

Trap Column: Dionex IonPac CTC-1,
9 × 24 mm
Carrier: Deionized water
Carrier Flow Rate: 0.5 mL/min
Inj. Vol.: 100 µL
Temperature: 30 °C
Neutralization: AutoNeutralize,
Dionex CSRN II,
external water mode,
500 mA

Analytical

Column: Dionex IonPac CG16,
CS16, 4 mm
Eluent: 28 mM Methanesulfonic acid
Eluent Source: Dionex EGC II MSA
Flow Rate: 1.0 mL/min
Temperature: 30 °C
Detection: Suppressed conductivity,
Dionex CSRS ULTRA II,
recycle mode, 82 mA
Concentrator: Dionex IonPac TCC-ULP1

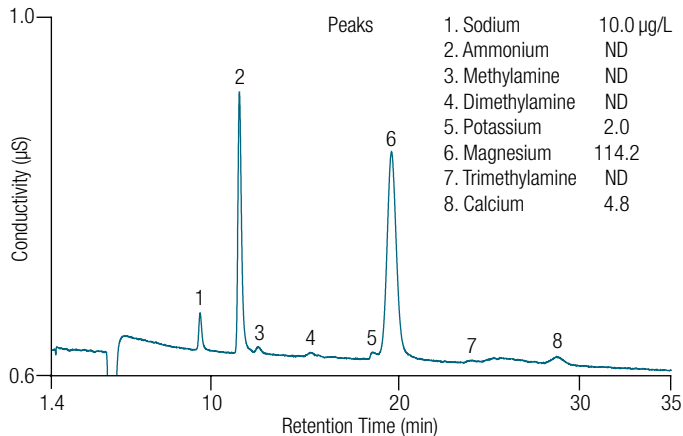


Figure 13. Trace levels of cations in 10% hydrofluoric acid using AutoNeutralization.

Sample Prep.

Trap Column: Dionex IonPac CTC-1,
9 × 24 mm
Carrier: Deionized water
Carrier Flow Rate: 0.5 mL/min
Inj. Vol.: 100 µL
Temperature: 30 °C
Neutralization: AutoNeutralize,
Dionex CSRN II,
external water mode,
500 mA

Analytical

Column: Dionex IonPac CG16,
CS16, 4 mm
Eluent: 28 mM Methanesulfonic acid
Eluent Source: Dionex EGC II MSA
Flow Rate: 1.0 mL/min
Temperature: 30 °C
Detection: Suppressed conductivity,
Dionex CSRS ULTRA II,
recycle mode, 82 mA
Concentrator: Dionex IonPac TCC-ULP1

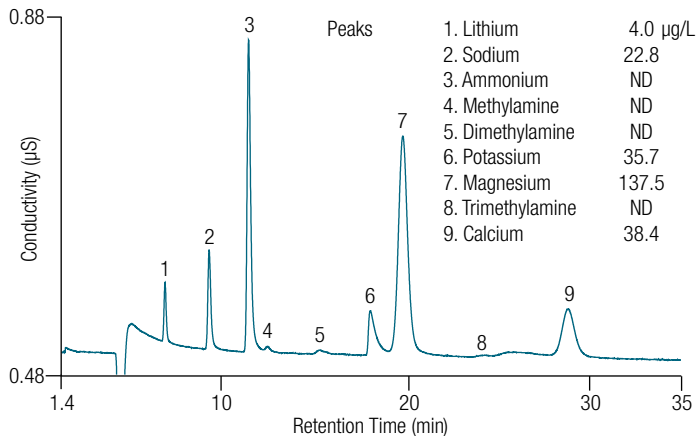


Figure 14. Trace levels of cations in 10% hydrofluoric acid spiked with the Dionex Combined Six Cation Standard-II.

Table 4. Recoveries of sodium, potassium, magnesium, and calcium from 25% phosphoric acid and 10% hydrofluoric acid.

Cation	25% Phosphoric Acid					10% Hydrofluoric Acid				
	Unspiked (µg/L)	RSD	Added (µg/L)	Recovery (%)	RSD	Unspiked (µg/L)	RSD	Added (µg/L)	Recovery (%)	RSD
Lithium	<0.06	—	51.7	100.7	1.2	<0.06	—	3.8	106.1	3.0
Sodium	818 ± 1.8	0.2	208	102.8	<0.1	10.0 ± 0.2	2.4	15.0	91.2	0.6
Potassium	150.9 ± 0.9	0.6	512	100.8	1.3	2.0 ± 0.3	1.5	37.5	90.4	0.6
Magnesium	65.7 ± 1.9	2.9	250	100.6	0.4	114.2 ± 2.6	2.3	19.0	103.2	3.6
Calcium	458.8 ± 1.4	0.3	512	109.5	0.1	4.8 ± 0.1	0.3	37.5	90.7	1.2

n = 5 injections.

Conclusion

Using the Automation Manager, dual pump, dual detector, and eluent generation of a Dionex ICS-3000 system, this Application Note demonstrates that strong acids can be neutralized to allow determination of µg/L cation concentrations. The neutralization occurs automatically within the temperature-controlled environment of the Dionex ICS-3000 DC using the Dionex CSRN II neutralizer. The setup described here provides an easy, automated, and reproducible method to determine µg/L cation concentrations in strong acids necessary for the chemical manufacturing, industrial plating, computer, environmental, mining, pharmaceutical, food, and beverage industries.

Although not discussed in this Application Note, trace concentrations of anions can also be determined in concentrated bases using the same techniques and the corresponding anion components. (See AN 93.⁸)

Precautions

Always handle concentrated acids with caution. Consult the Material Safety Data Sheets (MSDS) for protective clothing, storage, disposal, and health effects. Use only low pressure concentrator columns, such as the Dionex IonPac TCC-ULP1 column, with the Dionex CSRN II neutralizer. Pressures greater than 100 psi can cause irreversible damage to the neutralizer. Always verify that the external water regenerant is flowing through the neutralizer before applying current and turn it off when the pump is off. Ammonium can not be determined quantitatively with this method. Always use high quality deionized water (>18.2 MΩ·cm resistivity) for preparing reagents and samples.

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