

Reducing Carbonate Interference in Anion Determinations with the Carbonate Removal Device (CRD)

REAGENTS AND STANDARDS

Carbonate, a naturally occurring anion in water and aqueous solutions, results from the dissolution of carbon dioxide gas in these solutions. In some beverages, carbon dioxide is purposely added for flavor or effervescence and also acts as a preservative. When samples are analyzed by ion chromatography (IC) with hydroxide and tetraborate eluents, the sample carbonate may coelute and interfere with the quantification of anions of interest (e.g., nitrite, bromide, sulfate, or perchlorate).

Carbonate contamination in IC with hydroxide and tetraborate eluents can be introduced by the eluent or the sample. Because the carbonate acts as an eluent, variable carbonate levels in the mobile phase due to variation in eluent preparation steps cause poor retention time and peak area reproducibilities. This type of carbonate contamination is largely eliminated using a Dionex Reagent-Free™ Ion Chromatography (RFIC™) system.¹ The RFIC system electrolytically generates the hydroxide eluent in situ, thereby eliminating the exposure to air and subsequent carbon dioxide absorption. Carbonate introduced into the IC system with the deionized water (used to supply the RFIC system) is essentially eliminated by the Continuous Regenerating Anion Trap Column (CR-ATC). The CR-ATC is installed before the injection valve and after the EluGen® cartridge and EG degas module.

Until recently, carbonate could not be eliminated from the sample without a sample preparation step that could potentially contaminate the sample. The carbonate peak can now be removed from the chromatogram by the Carbonate Removal Device (CRD) without compromising the sample or adding sample preparation steps.²

This Technical Note discusses the theory, operation, and installation of the CRD, and highlights its advantages

for anion determinations. This note also demonstrates the benefits of using the CRD for three different applications:

- a) Determination of low $\mu\text{g/L}$ concentrations of anions and organic acids in ultrapure water using an IonPac® AS15 column (3×150 mm, $5 \mu\text{m}$), and a gradient separation with a 1-mL injection (Dionex Application Update 142)³,
- b) Determination of low $\mu\text{g/L}$ perchlorate in drinking water using an IonPac AS16 column (4×250 mm) with a 1-mL injection (Dionex Application Update 148)⁴,
- c) Determination of anions in carbonated mineral water using an IonPac AS18 (2×250 mm) column (Dionex Application Note 154)⁵.

Dionex Application Update 142 describes the determination of $\mu\text{g/L}$ concentrations of anions and organic acids in ultrapure water. This is an important application for the semiconductor, disk drive, electronic components, and nuclear power industries. In ultrapure water, carbonate is the most abundant anion, more than 10 \times the concentration of any other anion. In this application, the analytical challenge is to accurately identify and quantify ng/L or low $\mu\text{g/L}$ concentrations of adipate, sulfate, oxalate, and bromide that elute on the tail of a much larger carbonate peak.

Dionex Application Update 148 describes the determination of single digit $\mu\text{g/L}$ concentrations of perchlorate in very high mg/L concentrations of chloride, sulfate, and carbonate. The challenge is to accurately identify and quantify perchlorate as it elutes on the tail of the combined peak for chloride, carbonate, and sulfate.

Application Note 154 describes the determination of anions in environmental waters using an RFIC system.

We selected a carbonated mineral water sample for this application. Carbonated mineral water is a challenging sample when bromide and chlorate must be determined. For this and the other two applications, the CRD successfully removes carbonate just prior to conductivity detection after it exits the anion self-regenerating suppressor (ASRS® ULTRA II). This permits the easy quantification of the target analytes.

EXPERIMENTAL

Equipment

Dionex ICS-2500 system or ICS-3000 equivalent system consisting of:

Gradient Pump (GP50 or DP/SP) with degas option and gradient mixer (GM-4 Dionex, P/N 049136)

Eluent Generator (EG50 or EG) with EluGen II potassium hydroxide (Dionex, P/N 058900), CR-ATC, Continuous Regenerating Anion Trap Column, (Dionex, P/N 060477) and degas module

Conductivity Detector (CD25A with AS50T Thermal Compartment or DC with CD, Dionex P/N 061716)

Autosampler (AS50 or AS)

Chromeleon® Chromatography Workstation with Chromeleon 6.6

(Note: The CRD can be used with any RFIC system.)

Filter unit, 0.2- μ m nylon (Nalgene Media-Plus with 90-mm filter, Nalge Nunc International P/N 164-0020 or equivalent nylon filter)

Vacuum pump

300 cm of green 0.75-mm i.d. (0.030-in.) PEEK tubing to make 1100- μ L loop (P/N 044077, ordered per in.)

Black 0.25-mm i.d. (0.01-in.) PEEK tubing to make 5- μ L loop (P/N 052306 for 5 ft.)

Low pressure Teflon® (E.I. du Pont de Nemours) tubing (1.6-mm or 0.063-in. i.d., P/N 014157) for the CRD and degas waste lines

10-mL polystyrene vials (for AS50 or AS Autosampler), with caps and slit septa (Dionex, P/N 055058)

Micropipettor and tips for preparing samples, standards, and pipetting samples into vials

Application 1: Corning or other brands of similar quality (Corning, P/N 431081 or VWR, P/N 29186-199) 225-mL polystyrene sterile flasks.

Reagents and Standards:

Deionized water, Type 1 reagent-grade, 18 M Ω -cm resistivity or better

(Note: Use only ACS reagent grade chemicals for all reagents and standards).

Application 1: Anions and Organic Acids in Ultrapure Water

Combined Seven Anion Standard II (Dionex, P/N 57590)

Acetic acid (JT Baker, P/N JT9515-03)

Adipic acid (Aldrich, P/N A26357-100G)

Glycolic acid (or hydroxyacetic acid) (Sigma, P/N G8284)

Hydroxyisobutyric acid (HIBA) (Aldrich, P/N 32,359-4)

Methanesulfonic acid (Dionex, P/N 033478)

Oxalic acid, dihydrate (Fisher, P/N A219-500)

Phthalic acid (Aldrich, P/N 402915)

Sodium formate (Fisher, P/N S648-500)

Application 2: Perchlorate in Drinking Water

Sodium carbonate, monohydrate (Fisher, P/N S262-3)

Sodium chloride, crystalline (JT Baker ULTRAPURE BIOREAGENT, P/N JT3624-1)

Sodium perchlorate, anhydrous crystal (EM Science, P/N EM-SX0692-1)

Sodium sulfate, granular (EM Science, P/N EM-SX0760-1)

Application 3: Carbonated Mineral Water

Acetic acid (JT Baker, P/N JT9515-03)

Citric acid, monohydrate (JT Baker, P/N JT0110-1)

Oxalic acid, dihydrate (Fisher, P/N A219-500)

Sodium bromide (Aldrich, P/N 31,050-6)

Sodium carbonate, monohydrate (Fisher, P/N S262-3)

Sodium chlorate (Aldrich ReagentPlus™, P/N 24,414-7)

Sodium chloride, crystals (JT Baker ULTRAPURE BIOREAGENT, P/N JT3624-1)

Sodium fluoride (Fisher, P/N S299-100)

Sodium formate (Fisher, P/N S648-500)
Sodium nitrate, crystalline (Fisher, P/N S343-500)
Sodium nitrite (JT Baker, P/N JT3780-1)
Sodium phosphate, dibasic anhydrous (JT Baker UL-TRAPURE BIOREAGENT, P/N JT4062-1)
Sodium sulfate, granular, (EM Science, P/N EM-SX0760-1)

Samples

Application 1: Type 1 deionized water
Application 2: City of Sunnyvale, CA drinking water
Application 3: Brand A carbonated mineral water, purchased from a grocery store.

Conditions

Application 1: Anions and Organic Acids in Ultrapure Water

Columns: IonPac AS15-5 μ m Analytical, 3 \times 150 mm (P/N 057594)
IonPac AG15-5 μ m Guard, 3 \times 30 mm (P/N 057597)
Flow Rate: 0.7 mL/min
Eluent (EG50 or EG): 7–60 mM potassium hydroxide for 5–12 min
Inj. Valve: Reset to the “Load” position at 4 min
Temperature: 30 °C
Inj. Loop Size: 1100 μ L
Inj. Volume: 1000- μ L partial loop injection of 1100- μ L loop
Detection: Suppressed conductivity with ASRS ULTRA II, recycle mode, 2 mm (P/N 061562)
Current setting, 104 mA
CRD: Microbore 2 mm (P/N 062986)
Background: 0.8 μ S at 7 mM potassium hydroxide
Typical Backpressure: ~2300 psi
Typical Noise: <3 nS
Run Time: 20 min

Application 2: Perchlorate in Drinking Water

Columns: IonPac AS16 Analytical, 4 \times 150 mm (P/N 055376)

IonPac AG16 Guard, 4 \times 50 mm (P/N 055377)
Flow Rate: 1.2 mL/min
Eluent (EG50 or EG): 65 mM potassium hydroxide
Temperature: 30 °C
Inj. Loop Size: 1100 μ L
Inj. Volume: 1000- μ L partial loop injection of 1100- μ L loop
Detection: Suppressed conductivity ASRS ULTRA II, external water mode, 4 mm (P/N 061561)
Current setting, 193 mA
CRD: Standard bore, 4 mm (P/N 062983)
Background: 0.4 μ S
Typical Backpressure: ~2300 psi
Typical Noise: <1.0 nS
Run Time: 15 min

Application 3: Carbonated Mineral Water

Columns: IonPac AS18 Analytical, 2 \times 150 mm (P/N 060553)
IonPac AG18 Guard, 2 \times 50 mm (P/N 060555)
Flow Rate: 0.25 mL/min
Eluent (EG50 or EG): 22–40 mM potassium hydroxide for 7–8 min
Injection Valve: Reset to the “Load” position at 4 min
Temperature: 30 °C
Inj. Volume: 5- μ L full loop injection (PEEK sample loop)
Detection: Suppressed conductivity with ASRS ULTRA II, recycle mode, 2 mm (P/N 061562)
Current setting, 25 mA
CRD: Microbore, 2 mm (P/N 062986)
Background: 0.4 μ S at 22 mM potassium hydroxide
Typical Backpressure: ~2100 psi
Typical Noise: <2 nS
Run Time: 15 min

PREPARATION OF SOLUTIONS AND REAGENTS

Eluent Preparation

It is essential to use high quality, Type 1 water, >18 MΩ-cm. Degas if needed.

Standard Preparation

Application 1: Anions and Organic Acids in Ultrapure Water

Prepare individual stock solutions of 1000 mg/L acetate, formate, glycolate, adipate, oxalate, and phthalate. Dissolve the amount of reagent grade compound (Table 1) in deionized water in a 100-mL Class A volumetric flask and dilute to 100-mL mark with deionized water.

Prepare separate intermediate standards of 1.0 mg/L from each of the stock solutions. Pipette 100 µL of the individual stock solution into a 120-mL polypropylene

Anion	Compound	Mass (g)
Glycolate	Glycolic acid (CH ₂ OHCOOH)	0.101
Acetate	Glacial acetic acid (CH ₃ COOH)	0.102
Formate	Sodium formate (HCOONa)	0.151
Hydroxyisobutyrate	2-Hydroxyisobutyric acid (HIBA) (CH ₃ CHOHCH ₂ COOH)	0.101
Methanesulfonate	Methanesulfonic acid (CH ₃ SO ₃ H)	0.101
Adipate	Adipic acid (COOH(CH ₂) ₄ COOH)	0.101
Oxalate	Oxalic acid dihydrate (HOCCOOH•2H ₂ O)	0.143
Phthalate	Phthalic acid (C ₆ H ₄ (COOH) ₂)	0.101

bottle. Dilute with deionized water to 100.00 g total weight. Prepare an intermediate standard from the Dionex Combined Seven Anion Standard II. Pipette 1000 µL into the 120-mL polypropylene bottle. Dilute 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 Application Up-

date 142 for additional precautions needed for determinations of µg/L anion concentrations⁶.)

Prepare working standards of combined anion and organic standards from the intermediate standards. Pipette 1000 µL and 3000 µL of the intermediate standards of Combined Seven Anion Standard and organic acids, respectively into a 225-mL polystyrene sterile flask. Dilute this working standard with deionized water to 100.00 g total weight. The final concentrations are 1.0 µg/L nitrite, chloride, sulfate, nitrate, and bromide, 0.2 µg/L of fluoride, 2.0 µg/L of phosphate, and 3.0 µg/L of glycolate, acetate, formate, hydroxyisobutyrate, methanesulfonate, adipate, oxalate, and phthalate. Prepare working standards of 0.5, 2.0, 4.0, and 5.0 µg/L of anions with 2.0, 4.0, 5.0, and 10 µg/L of organic acids, in a similar manner as the first working standard. Prepare low µg/L standards daily, higher µg/L standards weekly, and mg/L standards monthly.

Application 2: Perchlorate in Drinking Water

Prepare stock solutions of 1000 mg/L perchlorate and 25,000 mg/L each of carbonate, chloride, and sulfate. Dissolve the amount of reagent grade compound (Table 2) in deionized water and dilute with deionized water to 100.00 g total weight.

Prepare an intermediate standard of 1.0 mg/L perchlorate. Pipette 100 µL of the 1000 mg/L perchlorate stock into a 120-mL polypropylene bottle and dilute with deionized water to 100.00 g total weight.

Anion	Compound	Mass (g)	Concentration of Anion in Stock Solution (mg/L)
Perchlorate	Sodium perchlorate (NaClO ₄)	0.123	1,000
Carbonate	Sodium carbonate monohydrate (Na ₂ CO ₃ •H ₂ O) ^a	5.166	25,000
Chloride	Sodium chloride (NaCl)	4.121	25,000
Sulfate	Sodium sulfate (Na ₂ SO ₄)	3.697	25,000

^a Or 4.4160 g of anhydrous sodium carbonate

Prepare working standards of 0, 5, and 25 µg/L perchlorate in water, 100MA, and 500MA. One hundred MA (100MA) is defined as a matrix solution of 100 mg/L each

Table 3. Amount (µL) of Stock Solutions Used to Prepare 100 mL of Working Standards

	1.0 mg/L Perchlorate ^a	25,000 mg/L Carbonate	25,000 mg/L Chloride	25,000 mg/L Sulfate
5 µg/L perchlorate in water	500	—	—	—
25 µg/L perchlorate in water	2500	—	—	—
100MA ^b	—	400	400	400
5 µg/L perchlorate in 100MA	500	400	400	400
25 µg/L perchlorate in 100MA	2500	400	400	400
500MA ^c	—	2000	2000	2000
5 µg/L perchlorate in 500MA	500	2000	2000	2000
25 µg/L perchlorate in 500MA	2500	2000	2000	2000
100 mg/L carbonate	—	400	—	—
500 mg/L carbonate	—	2000	—	—

^a 1 mg/L perchlorate is an intermediate standard.

^b 100MA is a matrix solution of 100 mg/L each of chloride, carbonate, and sulfate.

^c 500MA is a matrix solution of 500 mg/L each of chloride, carbonate, and sulfate.

of chloride, carbonate, and sulfate, and 500MA is 500 mg/L each of the same anions. Pipette the amount shown in Table 3 of perchlorate, carbonate, chloride, and sulfate from the stock solutions into a 120-mL polypropylene bottle and dilute with deionized water to 100.00 g total weight.

Prepare 100 and 500 mg/L carbonate spiked samples in deionized water to evaluate carbonate removal. Pipette the amount of carbonate shown in Table 3 from the stock solutions into a 120-mL polypropylene bottle and dilute with deionized water to 100.00 g total weight.

Application 3: Carbonated Mineral Water

Prepare individual stock solutions of 1000 mg/L of fluoride, acetate, formate, nitrite, bromide, nitrate, chlorate, oxalate, phosphate, and citrate and 25,000 mg/L of chloride, sulfate, and carbonate. Dissolve the amount of reagent grade compound (Table 4) in deionized water in a 120-mL polypropylene bottle and dilute with deionized water to 100.00 g total weight.

Table 4. Amount of Compound Used to Prepare 100 mL of Individual Stock Solutions

Anion	Compound	Mass (g)	Concentration of Anion in Stock Solution (mg/L)
Fluoride	Sodium fluoride (NaF)	0.221	1000
Acetate	Glacial acetic acid (CH ₃ COOH)	0.102	1000
Formate	Sodium formate (HCOONa)	0.151	1000
Chloride	Sodium chloride (NaCl)	4.121	25,000
Nitrite	Sodium nitrite (NaNO ₂)	0.150	1000
Bromide	Sodium bromide (NaBr)	0.129	1000
Carbonate	Sodium carbonate monohydrate (Na ₂ CO ₃ •H ₂ O) ^a	5.166	25,000
Sulfate	Sodium sulfate (Na ₂ SO ₄)	3.697	25,000
Nitrate	Sodium nitrate (NaNO ₃)	0.137	1000
Chlorate	Sodium chlorate (NaClO ₃)	0.128	1000
Oxalate	Oxalic acid dihydrate (HOOC-COOH•2H ₂ O)	0.143	1000
Phosphate	Sodium phosphate, dibasic (Na ₂ HPO ₄)	0.150	1000
Citrate	Citric acid monohydrate (HOOC-CH ₂ -C(OH)(COOH)-CH ₂ -COOH•H ₂ O)	0.110	1000

^a Or 4.4160 g of anhydrous sodium carbonate.

Prepare combination working standards of 0.05, 0.10, 1.0, 5.0 mg/L of fluoride, acetate, formate, nitrite, bromide, chlorate, oxalate, phosphate, and citrate with 2.5, 5.0, 10, 50 mg/L of nitrate, and 50, 100, 200, 500 mg/L of chloride, sulfate, and carbonate. Pipette the amount shown in Table 5 from the stock solutions into a 120-mL polypropylene bottle and dilute with deionized water to 100.00 g total weight.

Table 5. Amount (μL) of Stock Solutions Used to Prepare 100 mL of Working Standards

Stock Solutions (mg/L)	Working Standard			
	1	2	3	4
Fluoride, acetate, formate, nitrite, bromide, chlorate, oxalate, phosphate, and citrate	5	10	100	500
Chloride, carbonate, and sulfate	200	400	800	2000
Nitrate	250	500	1000	5000

Sample Preparation

Application 1: Anions and Organic Acids

Type I deionized water was analyzed directly without any sample treatment. Trace anion determinations in ultrapure water require additional steps to maintain a contamination-free system. Powder-free vinyl or nitrile gloves should be worn during all sample preparation and sample handling steps. Sample vials should be rinsed three to five times and soaked 24 h prior to use with Type 1, deionized water. The deionized water in the syringe flush container should be replaced daily with fresh deionized water. (See Application Update 142 for additional precautions needed for determinations of $\mu\text{g/L}$ anion concentrations.)

Application 2: Perchlorate in Drinking Water

The City of Sunnyvale drinking water was analyzed directly without any dilution or sample preparation. The drinking water was spiked with perchlorate at 5, 10, 15, and 20 $\mu\text{g/L}$. To prepare these samples, pipette 500, 1000, 1500, and 2000 μL of the 1.0-mg/L perchlorate stock into separate 120-mL polypropylene bottles and dilute to 100.00 g with drinking water.

Application 3: Carbonated Mineral Water

Brand A carbonated mineral water sample was diluted fivefold with degassed deionized water prior to analysis. Samples were prepared fresh daily from a previously unopened bottle of the same lot.

SYSTEM SETUP

Install the GM-4 Eluent Gradient Mixer, EluGen II potassium hydroxide cartridge, CR-ATC, ASRS ULTRA II suppressor, columns, and backpressure loops for the suppressor and the eluent generator. Install the GM-4 Eluent Gradient Mixer between the pump and the EluGen cartridge according to the start-up instructions in the EG50 product manual.⁷ Follow the Quickstart instructions for the CR-ATC⁸ to hydrate the CR-ATC and install it after the EGC cartridge and before the injection valve and the degas module. Install the columns after the injection valve according to the product manuals.⁹⁻¹¹ Install a backpressure loop on the “cell out” position of the conductivity cell. Follow the Quickstart instructions in the suppressor product manual¹² to hydrate the suppressor and install it between the columns and the conductivity cell. The suppressor should be installed in recycle mode for Applications 1 and 3, and external water mode in Application 2. Measure the system pressure with and without the backpressure coil at the experiment's flow rate.¹³ This must be ~ 40 psi to prevent suppressor damage. After the installations are completed, check the total system pressure. The total system pressure should be >2000 psi for the eluent generator with the optimum operating pressure of 2300 psi. If the system pressure is <2000 psi, install yellow (0.003 in. i.d.) PEEK tubing between the degas module and the injection valve to increase the system pressure to ~ 2300 psi.¹⁴ Do not allow the system pressure to exceed 3000 psi, as this could damage the degas module.

Application 1: Preparation of an 1100- μL Sample Loop

To make a 1000- μL injection with the AS50 Autosampler, use an 1100- μL sample loop to make a partial loop injection. To prepare an 1100- μL sample loop, cut a 242-cm length of green 0.75-mm i.d. (0.030-in.) PEEK tubing. The sample loop volume must be calibrated by weight using an analytical balance because the tubing inside diameter can vary by as much as 20%. The sample loop volume is the difference between the loop filled with deionized water and the empty loop. (See Dionex Application Note 166 for an example of this calculation¹⁵.) For this application, the calculated sample loop should be at least 1090 μL .

Table 6. Program Wizard Entries for Determinations of Trace Anions in Ultrapure Water

Parameter	Factor		Section	Action
Gradient type Flow	Multistep gradient 0.6		Pump options	
Retention time -5.1 -5.0 0 5.0 12.0 20.0	Concentration 60.0 7.0 7.0 7.0 60.0 60.0	Gradient curve 5.0 5.0 5.0 5.0 5.0 5.0	Flow gradient options	Equilibration Start and inject Start gradient End gradient High concentration, end
Column temperature Syringe speed Sample needle height Cut volume Flush volume	30 4 2 30 3000		Sampler options	3–5× sample volume
Acquisition time	0 to 20		Acquisition options	
Data collection rate Oven temperature Suppressor Hydroxide	5.0 off ASRS_2mm 60		ECD_1 options	Enter high concentration
Retention time Sampler_inject valve Load position Add	4.0 Select Select Select		Relay & state device option	Reset to load at 4.0 min

Application 1: AS50 Parameters and Program Parameters

For partial loop injection, enter this sample loop volume as the “sample loop size,” on AS50 module under Module Setup Menu, Plumbing Configuration. Enter the program parameters listed in Table 6 during program creation with the Chromeleon Program Wizard.

The flush volume should be 3–5× the sample loop volume. On the AS50 Autosampler, the flush occurs through the injection needle and injection port but not through the sample loop. During the run, the sample loop is open to elute both at the low concentration and high concentration eluent. A high eluent concentration in the loop at the time of sample loading will affect early eluting organic acids, especially at trace anion concentrations. Therefore the injection valve is reset to the load position prior to the gradient ramp, and after the eluent flow has swept the sample loop 2–10×. In this case, with a flow of 0.7 mL/min, every 1.57 min the 1100-µL sample loop is completely swept with eluent and the gradient ramp starts at 5 min. Therefore, the injection valve (see Table 6, Relay & State Device Options) is programmed

to reset to the load position at 4 min. The injection valve command can also be entered manually into the program file, using “Control”, “Command.” The AS autosampler operates in a similar manner. More information can be found in the operator’s manuals of the AS50 and AS autosamplers.^{16,17}

Application 2: Preparation of an 1100-µL Sample Loop

The 1100-µL loop is prepared in the same manner as described in Application 1.

Application 2: AS50 Parameters and Program Parameters

For partial loop injection, enter the sample loop volume as the “sample loop size” on AS50 module under Module Setup Menu, Plumbing Configuration. Enter the program parameters listed in Table 7 with Chromeleon Program Wizard. The flush volume should be 3–5× the sample loop volume.

Table 7. Program Wizard Entries for Determining Perchlorate in mg/L Chloride, Carbonate, and Sulfate

Parameter	Factor		Section	Action
Gradient type Flow	Isocratic 1.2		Pump options	
Retention time -0.1 0.0 15.0	Concentration 65.0 65.0 65.0	Gradient curve 5.0 5.0 5.0	Flow gradient options	Start, Inject End
Column temperature Syringe speed Sample needle height Cut volume Flush volume	30 4 2 30 3000		Sampler options	3-5× sample volume
Acquisition time	0 to 15		Acquisition options	
Data collection rate Oven temperature Suppressor Hydroxide	5.0 off ASRS_4mm 65		ECD_1 options	

Table 8. Program Wizard Entries for Determination of Inorganic Anions in Carbonated Mineral Water

Parameter	Factor		Section	Action
Gradient type Flow	Multistep gradient 0.25		Pump options	
Retention time -5.1 -5.0 0 7.0 8.0 15.0	Concentration 40.0 22.0 22.0 22.0 40.0 40.0	Gradient curve 5.0 5.0 5.0 5.0 5.0 5.0	Flow gradient options	Equilibration Start and inject Start gradient End gradient High concentration, End
Column temperature Syringe speed Sample needle height Cut volume Flush volume	30 4 2 30 3000		Sampler options	3-5× sample volume
Acquisition time	0 to 15		Acquisition options	
Data collection rate Oven temperature Suppressor Hydroxide	5.0 off ASRS_2mm 40		ECD_1 options	Enter high concentration
Retention time Sampler_inject valve Load position Add	4.0 Select Select Select		Relay and state device option	Reset to load at 4.0 min

Application 3: Preparation of a 5- μ L Sample Loop

Prepare a 5- μ L loop from black PEEK 0.25-mm i.d. (0.010-in) tubing. Cut a 10-cm length and calibrate the sample volume by measuring the weight difference of the tubing filled with deionized water and empty tubing. (See Dionex Application Note 166 for an example of this calculation¹⁵.)

Application 3: AS50 Parameters and Program Parameters

In this application, the injection is a full loop injection of 5 μ L. Enter the sample loop volume on the AS50 Autosampler module under Plumbing Configuration, and the Cut Volume of "0" in the System Parameters for a full loop injection. We used a Flush Volume of 250 μ L.

The injection valve was reset to the load position at 4 min (~10× the volume of the sample loop and before the gradient concentration ramp). Enter the program parameters listed in Table 8 with Chromeleon Program Wizard.

CRD

Theory and Operation of the CRD

The CRD is a membrane-based module that transports carbon dioxide across a gas-permeable membrane (Figure 1)¹⁸. Carbonate ion is removed as carbon dioxide from the sample after suppression and just prior to detection, resulting in improved separation and quantification of select anions.

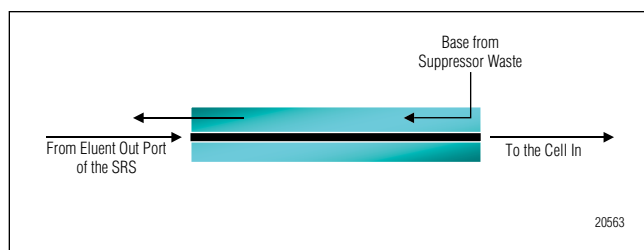


Figure 1. CRD schematic.

The CRD contains a narrow-bore capillary membrane tube that is thinly coated with a carbon dioxide permeable silicone film (Figure 2)¹⁹ that takes advantage of the carbonic acid-carbon dioxide equilibrium (Equation 1) chemistry to remove carbon dioxide, thus removing the carbonate peak.

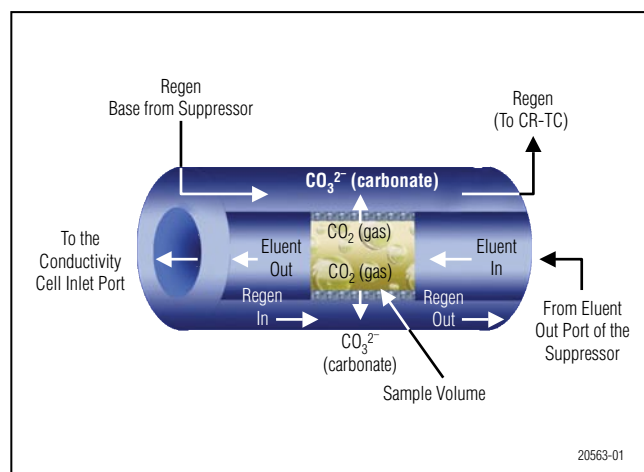
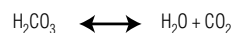


Figure 2. Carbonate Removal Device (CRD) schematic.



Equation 1. CRD equilibrium.

The efficiency of the carbonate peak removal will be called “Apparent % Removal Efficiency” (Equation 2).

$$\text{Apparent \% Removal Efficiency} = 100 - \left(\frac{\text{Response}_{\text{w/CRD}}}{\text{Response}_{\text{w/oCRD}}} \times 100 \right)$$

Equation 2. Apparent removal efficiency.

The CRD is positioned between the ASRS ULTRA II suppressor and the conductivity cell. The eluent and separated sample flow out of the column and into the suppressor. The suppressor converts carbonate ion to carbonic acid (H_2CO_3) in equilibrium with carbon dioxide. As the suppressed eluent flows into the CRD and through the inner CRD membrane tube, carbon dioxide flows across the membrane. This drives the equilibrium to form more carbon dioxide. Concurrently, regenerant waste from the suppressor flowing through the exterior surface of the CRD membrane tube converts carbon dioxide to carbonate ion. The carbonate ion is carried to waste.

CRD Installation

The CRD is easily installed on top of the ASRS ULTRA II suppressor (Figure 3)²⁰ and is intended for use with hydroxide and borate chemistries, and a Dionex RFIC system. To install, spread the bottom metal clips open and place the CRD over the ASRS ULTRA II suppressor with the “Eluent In” port of the CRD facing the “Eluent Out” port of the suppressor. Connect the 1.6-mm i.d. (0.063-in.) Teflon tubing to the regenerant lines and PEEK tubing (red for 2 mm, Dionex P/N 052310; black for 4 mm, Dionex P/N 052306) on the eluent lines.

Application of pressures higher than those recommended will damage the CRD. Always remove all plugs from the CRD before connecting it to the suppressor or the conductivity cell. Ensure that the pressure drop is between 30 and 40 psi for the cell with the backpressure tubing installed. This measurement was discussed in the System Setup section.

The 2-mm and 4-mm CRDs cannot be used interchangeably. The 2-mm CRD should only be used with 2-mm and 3-mm column sets; the 4-mm CRD with the 4-mm column sets. Dionex recommends that the CRD be hydrated before operation. See the CRD product manual for the hydration and backpressure measurement instructions.

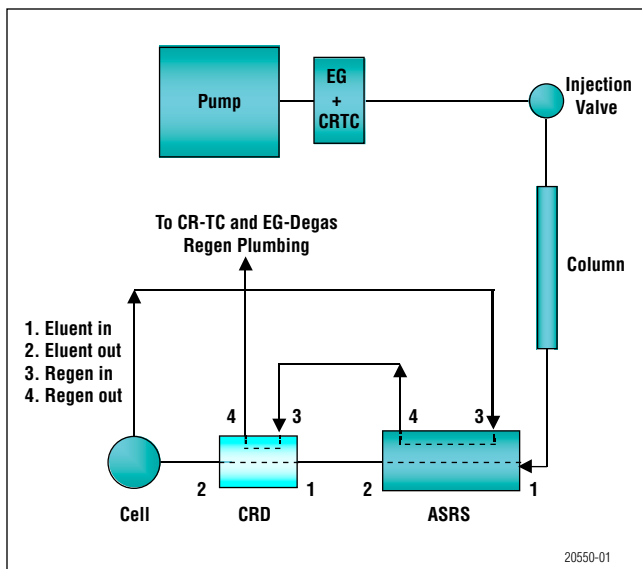


Figure 3. CRD plumbing.

RESULTS AND DISCUSSION

Application 1: Anions and Organic Acids

Trace anion analysis in ultrapure water is intended for detection of ng/L to low $\mu\text{g/L}$ levels of anions. Carbonate, which is easily and readily absorbed in this very clean matrix, is the most abundant anion present, and interferes or coelutes with sulfate, adipate, oxalate, and other anions. Application Update 142 describes trace anions concentrations in ultrapure water using an IonPac AS15-5 μm , 3-mm column set, gradient separation, a 1-mL injection, and suppressed conductivity detection.

We chose this application to demonstrate that the CRD significantly reduces the sample carbonate peak and improves trace-level anion determinations, especially for sulfate and adipate. We first determined the retention times of 7 inorganic anions and 8 organic acids with 10 $\mu\text{g/L}$ single anion standards and then analyzed anions in a combined standard containing 1 $\mu\text{g/L}$ chloride, nitrite, bromide, nitrate, and sulfate, 0.2 $\mu\text{g/L}$ fluoride, 2 $\mu\text{g/L}$ phosphate, and 3 $\mu\text{g/L}$ of glycolate, acetate, formate, hydroxyisobutyric acid (HIBA), methanesulfonate, adipate, oxalate, and phthalate. This standard was analyzed with and without the CRD installed. Figure 4 shows the effect of the CRD on removing the carbonate peak from the combined anion and organic acid standard. Carbonate remains the most abundant peak in these samples but most of the carbonate peak was removed by the CRD ($85 \pm 4.5\%$, Apparent % Removal

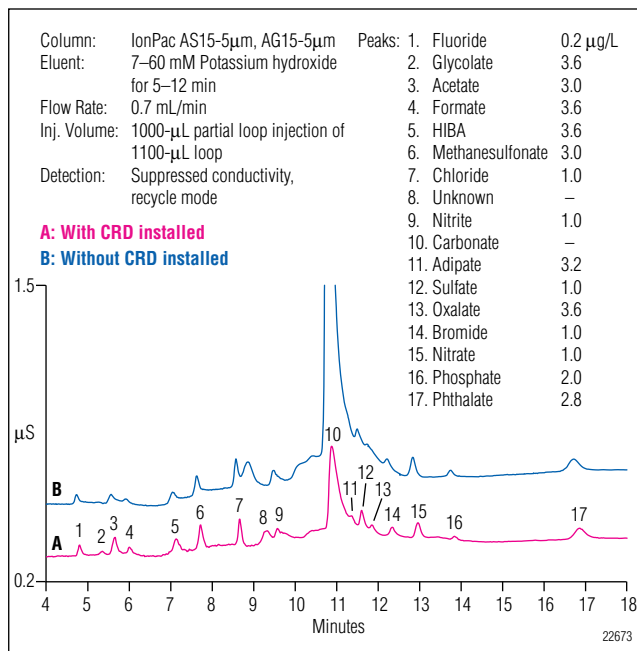


Figure 4. Determination of mixed anion and organic acid standard with (A) and without (B) a CRD.

Table 9. Carbonate Peak Removal by the CRD from Ultrapure Water

Sample	Without CRD	With CRD	Apparent % Removal Efficiency
	Carbonate Peak Area ($\mu\text{S}\cdot\text{min}$)	Carbonate Peak Area ($\mu\text{S}\cdot\text{min}$)	
Combined anion and organic acid standard	10.89 ± 0.01	0.123 ± 0.029	85.6 ± 4.5

n=10 for each data point.

Efficiency, Table 9). Adipate was only detected with the CRD installed (Table 10). The results show that there is an increase in retention times for all anions, +0.08 min to +0.15 min. This is expected because the CRD adds to the delay volume.

Application 2: Perchlorate in Drinking Water

Perchlorate is typically determined at single digit $\mu\text{g/L}$ concentrations in the presence of 100–1000 mg/L levels of carbonate, chloride, and sulfate, a 1:100,000 to 1:1,000,000 ratio. Chloride, sulfate, and carbonate elutes as a large peak and perchlorate elutes on the tail of that peak at 9.6 min. Application Update 148 describes the determination of $\mu\text{g/L}$ concentrations of perchlorate levels in a matrix containing high concentrations of carbonate, chloride, and sulfate using an IonPac AS16

Table 10. Effect of the CRD on Retention Times of µg/L Anions in Ultrapure Water

Combined Anion and Organic Acid Standard	Without CRD Retention Time (min)	With CRD Retention Time (min)
Fluoride	4.73 ± 0.01	4.81 ± 0.03
Glycolate	5.26 ± 0.00	5.35 ± 0.03
Acetate	5.57 ± 0.01	5.65 ± 0.02
Formate	5.92 ± 0.01	6.01 ± 0.02
HIBA ^a	7.06 ± 0.01	7.14 ± 0.01
Methanesulfonate	7.63 ± 0.00	7.72 ± 0.01
Chloride	8.57 ± 0.00	8.67 ± 0.02
Nitrite	9.47 ± 0.01	9.63 ± 0.02
Carbonate	10.79 ± 0.01	10.88 ± 0.01
Adipate	Not detected	11.30 ± 0.01
Sulfate	11.48 ± 0.07	11.60 ± 0.01
Oxalate	11.72 ± 0.07	11.86 ± 0.01
Bromide	12.20 ± 0.01	12.33 ± 0.01
Nitrate	12.83 ± 0.01	12.96 ± 0.01
Phosphate	13.75 ± 0.01	13.85 ± 0.02
Phthalate	16.72 ± 0.02	16.87 ± 0.03

n=10 for each data point.

^a Hydroxyisobutyric acid

column set (4 mm), a 1-mL partial loop injection, and suppressed conductivity detection.

This application demonstrates that the 4-mm CRD can reduce the carbonate peak and improve perchlorate quantifications. Samples of 5 µg/L and 25 µg/L perchlorate spiked in water (100MA and 500MA) were analyzed with and without the CRD installed (Table 11). As expected, perchlorate elutes later with the CRD installed. Peak efficiency (USP) is defined as:

$$16 \left(\frac{\text{Retention time}}{\text{Peak width}} \right)^2$$

and therefore it is expected that the efficiencies will decrease with the longer delay time added by the CRD. However in this case, the perchlorate peak efficiencies remained about the same with the CRD installed. Due to the presence of large concentrations of chloride and sulfate, it is difficult to measure the carbonate peak removal, but the chromatograms show significant reduction in the combined chloride-sulfate-carbonate peak (Figure 5). Figure 5 shows that large amounts of carbonate were removed from 500MA samples. The large peak is reduced in size and its tail approaches the baseline earlier.

Table 11. Summary of Perchlorate Peak Retention Times, Peak Widths, and Peak Efficiencies

Perchlorate Sample	Perchlorate without CRD			Perchlorate with CRD		
	Perchlorate Retention Time (min)	Perchlorate Peak Width (min)	Efficiency (USP)	Perchlorate Retention Time (min)	Perchlorate Peak Width (min)	Efficiency (USP)
5 µg/L in water	9.71 ± 0.02	0.454 ± 0.005	7347 ± 185	9.76 ± 0.01	0.452 ± 0.004	7408 ± 95
5 µg/L in 100MA ^a	9.72 ± 0.03	0.455 ± 0.007	7297 ± 211	9.73 ± 0.01	0.453 ± 0.005	7435 ± 120
5 µg/L in 500MA ^a	9.55 ± 0.02	0.601 ± 0.027 ^b	4067 ± 387 ^b	9.71 ± 0.01	0.602 ± 0.003 ^b	4157 ± 46 ^b
25 µg/L in water	9.77 ± 0.01	0.460 ± 0.000	7430 ± 28	9.79 ± 0.00	0.450 ± 0.000	7591 ± 89
25 µg/L in 100MA ^a	9.60 ± 0.01	0.457 ± 0.009	7106 ± 250	9.71 ± 0.01	0.460 ± 0.000	7186 ± 63
25 µg/L in 500MA ^a	9.63 ± 0.01	0.618 ± 0.004 ^b	3883 ± 52 ^b	9.76 ± 0.00	0.604 ± 0.011 ^b	4184 ± 165 ^b

n=10 for each data point.

^a MA indicates a mixed common anion solution of chloride, sulfate, and carbonate included in the sample matrix at the parenthetical mg/L concentration for each anion.

^b Both the peak widths and the efficiencies are manually calculated. In these samples, perchlorate is a rider peak on the combined peak of chloride and sulfate.

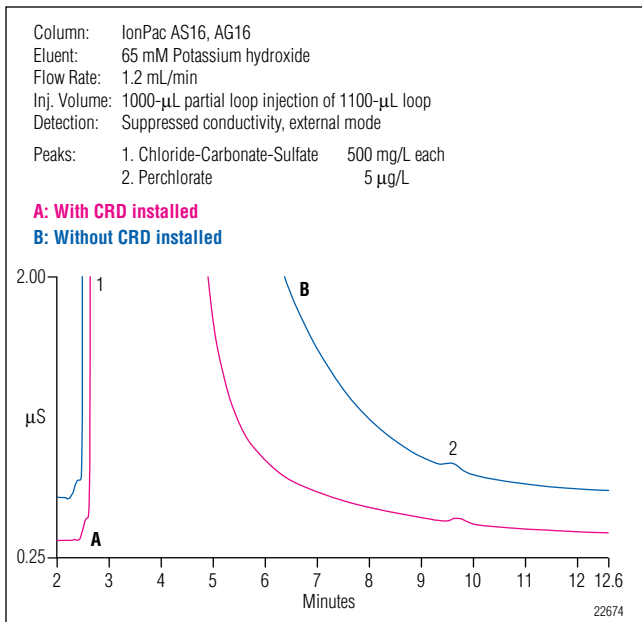


Figure 5. Chromatograms of 5 μ g/L perchlorate in 500MA, with (A) and without (B) a CRD.

To better assess carbonate removal, 100 mg/L and 500 mg/L carbonate were spiked into deionized water and the samples analyzed with and without the CRD (Table 12). These data show that >86% of the carbonate peak was removed from the two samples.

The City of Sunnyvale drinking water spiked with 5 μ g/L increments of perchlorate was analyzed with this method. Good spike recovery was obtained with and without the CRD installed. The City of Sunnyvale drinking water spiked with 5, 10, 15, and 20 μ g/L of perchlorate without the CRD installed had 104 \pm 3%, 106 \pm 2%, 110 \pm 2%, and 112 \pm 1% recoveries, respectively. The same samples with the CRD installed had similar recoveries: 105 \pm 1%, 108 \pm 2%, 106 \pm 1%, and 108 \pm 1%, respectively (Table 13). Carbonate peak tailing was reduced with the CRD (Figure 6).

Application 3: Carbonated Mineral Water and Artificial Drinking Water

Carbonated mineral water contains high levels of carbonate that interfere with routine anion determinations. Typically, the sample must be degassed prior to analysis in order to obtain acceptable results. The concern in any sample preparation is potential contamination or degradation of the sample. A sample preparation also requires several control samples to monitor the process.

Table 12. Effect of the CRD on Carbonate Peak Retention Times, Peak Areas, and Apparent % Removal Efficiencies

Carbonate Spiked Deionized Water Sample	Retention Time (min)	Peak Area (μ S-min)	Apparent % Removal Efficiency
100 mg/L without CRD	3.61 \pm 0.01	10.8 \pm 0.2	N/A
100 mg/L with CRD	3.69 \pm 0.01	1.51 \pm 0.11	86.0 \pm 0.9
500 mg/L without CRD	3.89 \pm 0.01	25.1 \pm 0.4	N/A
500 mg/L with CRD	4.01 \pm 0.01	3.42 \pm 0.13	86.4 \pm 0.6

n=6 for each data point.

Table 13. Spike Recovery (%) of Perchlorate in City of Sunnyvale Drinking Water

City of Sunnyvale Water	+ 5 μ g/L	+ 10 μ g/L	+ 15 μ g/L	+ 20 μ g/L
Without CRD	104 \pm 3	106 \pm 2	110 \pm 2	112 \pm 1
With CRD	105 \pm 1	108 \pm 2	106 \pm 1	108 \pm 1

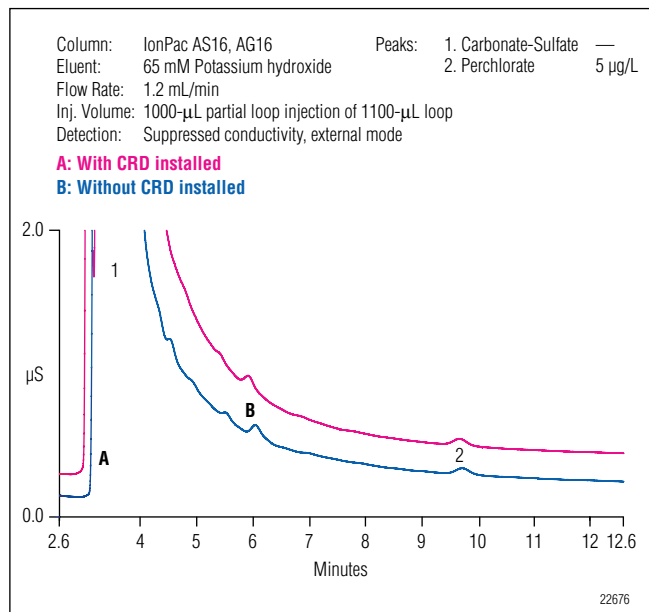


Figure 6. City of Sunnyvale Drinking Water with 5 μ g/L perchlorate spike, with (A) and without (B) a CRD.

Table 14. Effect of the CRD on Retention Times and Peak Efficiencies for Artificial Drinking Water Sample #1

Simulated Drinking Water #1 ^a	Without CRD Retention time (min)	With CRD Retention time (min)	Without CRD Efficiency (USP)	With CRD Efficiency (USP)
Fluoride	3.30 ± 0.00	3.46 ± 0.00	9605 ± 610	7339 ± 319
Chloride	5.10 ± 0.00	5.29 ± 0.00	11334 ± 27	9289 ± 26
Nitrite	6.38 ± 0.00	6.59 ± 0.00	14814 ± 111	13011 ± 403
Carbonate	7.55 ± 0.01	7.91 ± 0.01	985 ± 31	223 ± 35
Bromide	9.30 ± 0.00	9.58 ± 0.00	19276 ± 440	15653 ± 623
Sulfate	9.93 ± 0.01	10.3 ± 0.01	5542 ± 133	5360 ± 26
Nitrate	11.2 ± 0.00	11.5 ± 0.00	15690 ± 198	15487 ± 51
Chlorate	12.0 ± 0.00	12.3 ± 0.00	28382 ± 1551	26301 ± 2720
Phosphate	14.8 ± 0.01	15.1 ± 0.01	47614 ± 1960	44655 ± 1526

n=10 for each data point.

^a Artificial Drinking Water #1: 0.05 mg/L of fluoride, nitrite, chlorate, bromide, and phosphate; 2.5 mg/L nitrate; 25 mg/L sulfate; 50 mg/L chloride and carbonate.

Carbonated mineral water samples typically have high µg/L levels of fluoride, nitrite, bromide, chlorate, and phosphate in mg/L levels of nitrate, carbonate, chloride, and sulfate. These samples can be analyzed using an IonPac AS18 column set (2-mm), and suppressed conductivity detection. We designed these experiments to show that the CRD reduced carbonate concentrations and improved low-level anion determinations in carbonated drinking water. Artificial Drinking Water Standards

Table 15. Carbonate Peak Removal Results From Using a CRD on Artificial Drinking Water

Standard	Without CRD Carbonate Peak Area (µS-min)	With CRD Peak Area (µS-min)	Apparent % Removal Efficiency
Drinking water #1 ^a	1.28 ± 0.00	0.11 ± 0.01	91.7 ± 0.0
Drinking water #2 ^b	1.64 ± 0.04	0.11 ± 0.01	93.5 ± 0.3
Drinking water #3 ^c	2.65 ± 0.01	0.12 ± 0.00	95.4 ± 0.0
Drinking water #4 ^d	3.82 ± 0.01	0.05 ± 0.02	98.8 ± 0.4

n=10

^a Artificial Drinking Water #1: 0.05 mg/L of fluoride, nitrite, chlorate, bromide, and phosphate; 2.5 mg/L nitrate; 25 mg/L sulfate; 50 mg/L chloride and carbonate.

^b Artificial Drinking Water #2: 0.1 mg/L of fluoride, nitrite, chlorate, bromide, and phosphate; 5 mg/L nitrate; 50 mg/L sulfate; 100 mg/L chloride and carbonate.

^c Artificial Drinking Water #3: 0.2 mg/L of fluoride, 1.0 mg/L of nitrite, chlorate, bromide, and phosphate; 10 mg/L nitrate; 100 mg/L sulfate; 200 mg/L chloride and carbonate.

^d Artificial Drinking Water #4: 0.4 mg/L of fluoride, 5 mg/L of nitrite, chlorate, bromide, and phosphate; 50 mg/L nitrate; 200 mg/L sulfate; 500 mg/L chloride and carbonate.

were created to simulate high µg/L concentrations of fluoride, nitrite, bromide, chlorate, and phosphate, and low mg/L concentrations of nitrate in high mg/L concentrations of chloride, carbonate, and sulfate. Anion peak retention times and peak efficiencies results were compared with and without the CRD installed (Table 14). The results show similar carbonate removal by the CRD, 91.7 ± 0.0% to 98.8 ± 0.4% Apparent % Removal Efficiency (Table 15). Figure 7 shows that with the CRD installed, it is now easier to quantify bromide. The results again showed small increases in retention time and small peak efficiency losses, ~60 to 160 USP plates, with the CRD installed.

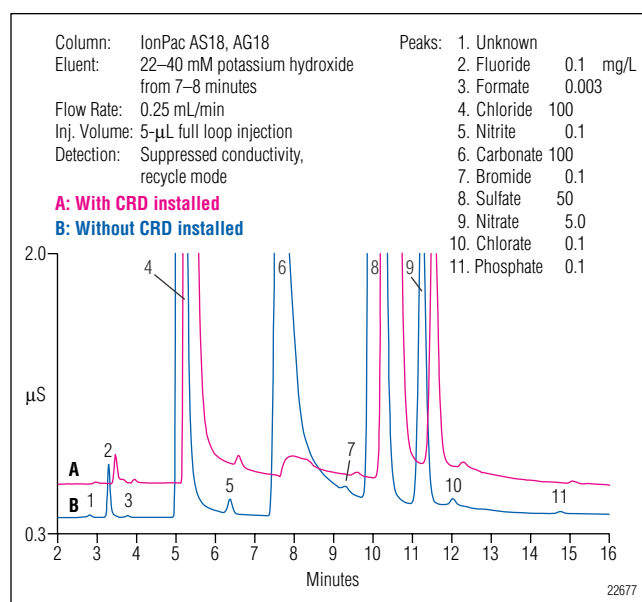


Figure 7. CRD removal of carbonate from an artificial drinking water sample #2.

We then applied the CRD to a carbonated mineral water sample. Brand A carbonated mineral water was labeled as a “low sodium, sparkling mineral water with lime essence and other natural flavors.” The ingredients list stated that it contains mineral water, natural flavors, and CO₂. A 5- μ L injection of this sample overloaded the column, therefore we diluted the sample fivefold with degassed deionized water. Due to the fluctuations in the initial carbonate concentration in the sample, carbonate peak removal is difficult to measure, but our data suggests that over 98% of the carbonate peak was removed (Figure 8).

CONCLUSION

The CRD removes most of the carbonate introduced by the sample, 86.0–98.8% in the three applications described here. This reduces carbonate interference when quantifying neighboring anions, including sulfate, adipate, and bromide. Due to the added delay volume, the CRD causes a slight increase in retention times and a slight decrease in efficiencies but these changes do not negate the significant advantages afforded by the removal of carbonate.

The CRD is easily installed on top of the ASRS ULTRA II suppressor and works efficiently in both the recycle and external modes of the suppressor. The applications presented here demonstrated the removal of carbonate from samples containing a wide range of carbonate concentrations and a wide range of analyte concentrations from μ g/L to mg/L. The CRD is suitable for hydroxide applications with Dionex systems using EluGen eluent generation, CR-ATC, and ASRS ULTRA II suppressor. The CRD has also been shown suitable for borate applications, but borate applications were not reviewed in this Technical Note.

More information on all these Dionex applications and products, including application notes and updates, technical notes, and product and installation manuals, can be found on the Dionex website, www.dionex.com.

PRECAUTIONS

As described in the CRD Product Manual the internal parts of CRD can be damaged by overtightening. All fittings on the CRD should be tightened by hand to “finger-tight”.

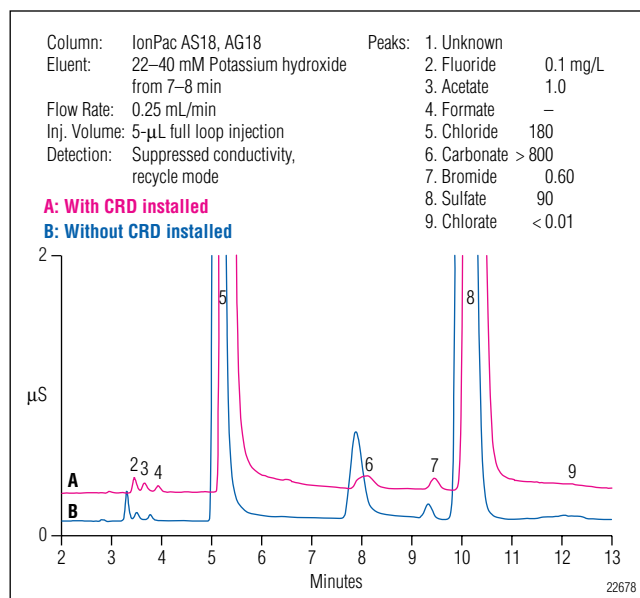


Figure 8. CRD removal of carbonate from a fivefold dilution of “Brand A” carbonated mineral water.

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