Dionex OnGuard II Cartridges

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Product Manual

for

Dionex OnGuard II Cartridges

OnGuard II A, 48 Pack, 1 cc, P/N 057091, 2.5 cc, P/N 057092
OnGuard II Ag, 48 Pack, 1 cc, P/N 057089, 2.5 cc, P/N 057090
OnGuard II Ba, 48 Pack, 1 cc, P/N 057093, 2.5 cc, P/N 057094
OnGuard II H, 48 Pack, 1 cc, P/N 057085, 2.5 cc, P/N 057086
OnGuard II Na, 48 Pack, 1 cc, P/N 062948, 2.5 cc, P/N 062962
OnGuard II M, 48 Pack, 1 cc, P/N 057137, 2.5 cc, P/N 057095
OnGuard II P, 48 Pack, 1 cc, P/N 057087
OnGuard II RP, 48 Pack, 1 cc, P/N 057083, 2.5 cc, P/N 057084
OnGuard II Ag/H, 48 Pack, 2.5 cc, P/N 057410
OnGuard II Ba/Ag/H, 48 Pack, 2.5 cc, P/N 063955

OnGuard II RP, 12 Pack, 1 cc, P/N 082760
OnGuard II H, 12 Pack, 1 cc, P/N 082761
OnGuard II Ag, 12 Pack, 1 cc, P/N 082762
OnGuard II Ba, 12 Pack, 1 cc, P/N 082763
OnGuard II Ba/Ag/H, 12 Pack, 2.5 cc, P/N 082764
OnGuard II Ag/H, 12 Pack, 2.5 cc, P/N 082756
OnGuard II A, 12 Pack, 1 cc, P/N 088355
OnGuard II M, 12 Pack, 1 cc, P/N 088356
OnGuard II P, 12 Pack, 1 cc, P/N 088358
OnGuard II Na, 12 Pack, 1 cc, P/N 088357

Revision History:

Revision 07, May, 2013; Rebranded for Thermo Scientific. New cartridge size added to the product line.

Revision 08, February, 2015; Added four new 12 packs for A, M, P, Na.

Revision 09, October, 2015; Corrected part number typos on page 2.

Revision 10, March, 2017; Added Dionex OnGuard Selection Tool and bibliography references.

Safety and Special Notices

Make sure you follow the precautionary statements presented in this guide. The safety and other special notices appear in boxes.

Safety and special notices include the following:

 **SAFETY**	***Indicates a potentially hazardous situation which, if not avoided, could result in death or serious injury.***
 **WARNING**	***Indicates a potentially hazardous situation which, if not avoided, could result in damage to equipment.***
 **CAUTION**	***Indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury. Also used to identify a situation or practice that may seriously damage the instrument, but will not cause injury.***
 **NOTE**	***Indicates information of general interest.***
IMPORTANT	***Highlights information necessary to prevent damage to software, loss of data, or invalid test results; or might contain information that is critical for optimal performance of the system.***
Tip	***Highlights helpful information that can make a task easier.***

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1. Introduction

The Dionex™ OnGuard™ II line of sample pretreatment cartridges includes seven functionalities in two cartridge sizes, 1.0 cc and 2.5 cc. These cartridges are suitable for matrix elimination and some analyte solid phase extraction methods. Matrix elimination is a method of sample preparation that removes interfering matrix species from the sample by specifically binding the matrix species to the phase, leaving the analyte for subsequent determination. In an analyte solid phase extraction method, the analyte is specifically bound to the phase while the matrix is washed through the cartridge. The analyte is subsequently eluted from the cartridge with a suitable eluent for subsequent analysis.

The Dionex OnGuard II cartridges incorporate a new and improved design aimed at ease-of-use. The design adds important performance-based features. Dionex OnGuard II hardware features include the Luer-lock inlet/Luer slip outlet, a full-size sample distribution frit, a two-level seal, and a gripper seal on the outlet bed support. These features allow easy stacking for mixed chemistries, maximum bed usage, and minimum sample loss. The 2.5 cc size reduces application cost by providing a capacity suited to high ionic strength samples. Resins packed in barrel format, suitable for automation, are also available as custom orders.

The Dionex OnGuard Selection Tool (Figure 1) is available for download to help customers find the appropriate Dionex OnGuard cartridge(s) for their application. Use the tool to browse 17 common applications and 10 Dionex OnGuard cartridges, and easily obtain ordering information, specifications, and startup procedures (Figure 2).

Tip ***The Dionex OnGuard Selection Tool requires Microsoft Excel 2010 or later to function properly.***

Figure 1 Browse applications and cartridges with the Dionex OnGuard Selection Tool

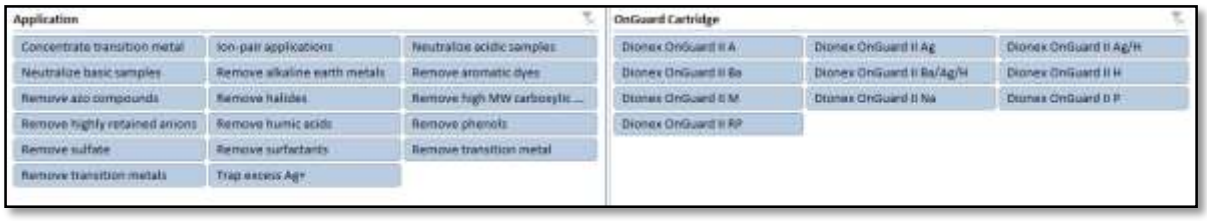

Figure 2 Easily obtain ordering information, specifications, and startup procedures

Thermo Scientific™ Dionex™ OnGuard™ II Cartridges

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Applications

Applications	Dionex OnGuard Cartridge	Formats	Part Numbers
Remove sulfate	Dionex OnGuard II Ba	1.0 cc & 2.5 cc	1.0 cc: 082763 (pkg of 12), 057093 (pkg of 48) 2.5 cc: 057094 (pkg of 48)

Specifications

OnGuard Cartridge	Functional Group	Mode of Use	pH Stability
Dionex OnGuard II Ba	Ba2+ form sulfonate	Precipitation Ion Exchange	0 - 14

Startup Procedures

OnGuard Cartridge	Formats	Startup Procedures
Dionex OnGuard II Ba	1.0 cc	For optimum results with Dionex OnGuard II cartridges, always observe the following basic principles of usage. 1. To provide the necessary pressure on the cartridges in manual pretreatment of samples, use a 5 mL syringe. 2. Before using a cartridge, flush it with 10 mL of deionized water to remove trace ionic contaminants from the cartridge and condition the packing. 3. To maximize loading on the cartridge bed, put the sample through at 2.0 mL/min. Higher flow rates are practical if less than maximum loading is acceptable. 4. Always discard the intial 3 mL of effluent when treating a sample or standard. 5. If the substance to be removed by the cartridge is colored or otherwise visible in the cartridge, use the cartridge until the visible band extends about three-fourths of the way to the cartridge outlet. For absolute determination of loading capabity for a specific application, aliquots of sample can be put through the cartridge and tested by the appropriate detection metod until a breakthrough is observed. 6. Optimum performance is achieved when the cartridges are used in a vertical direction.

NOTE ***Download the Dionex OnGuard Selection Tool here:***
http://www.thermofisher.com/content/dam/tfs/ATG/CMD/cmd-documents/bro/bro/chrom/ic/col/BR-OnGuard-Cartridge-Selection-Tool-EN.xlsm

The following table summarizes the Dionex OnGuard II product line and the associated chemistries:

Table 1 Recommended Cartridge Applications

Cartridge Type Size	Functional Group	Example Applications
Dionex OnGuard II A 1.0 cc, 2.5 cc Cartridges	HCO^-_3 form quaternary amine	Removal of highly retained anions, pH adjustment
Dionex OnGuard II Ag 1.0 cc, 2.5 cc Cartridges	Ag^+-form sulfonate	Removal of halides
Dionex OnGuard II Ba 1.0 cc, 2.5 cc Cartridges	Ba^{2+}-form sulfonate	Removal of sulfate
Dionex OnGuard II H 1.0 cc, 2.5 cc Cartridges	Sulfonic acid (H^+ form)	Removal of cations and transition metals, pH adjustment
Dionex OnGuard II Na 1.0 cc, 2.5 cc Cartridges	Sulfonic acid (Na^+ form)	Removal of cations and transition metals without pH adjustment
Dionex OnGuard II M 1.0 cc, 2.5 cc Cartridges	Iminodiacetate	Removal/concentration of transition metals
Dionex OnGuard II P 1.0 cc Cartridge	Polyvinylpyrrolidone	Removal/concentration of phenols, azo compounds, humic acid
Dionex OnGuard II RP 1.0 cc, 2.5 cc Cartridges	Divinylbenzene	Removal of hydrophobic species
Dionex OnGuard II Ag/H 2.5 cc Cartridge	Ag^+-form sulfonate Filter Sulfonic acid (H^+ form)	Removal of halides Trapping of excess Ag^+
Dionex OnGuard II Ba/Ag/H 2.5 cc Cartridge	Ba^{2+}-sulfonate Ag^+-form sulfonate Filter Sulfonic acid (H^+ form)	Removal of sulfates Removal of halides Trapping of excess Ag^+

This manual describes recommended manual pretreatment procedures and examples of use for each of the cartridge types. Twelve cartridges can be preconditioned using the Dionex OnGuard Sample Preparation Workstation (P/N 039599).

Table 2 Dionex OnGuard II Characteristics

Cartridge Type	1 cc Cartridge meq*	2.5 cc Cartridge meq*
Dionex OnGuard II A	0.7	1.75
Dionex OnGuard II Ag	2.0 – 2.2	5.0 – 5.5
Dionex OnGuard II Ag/H	n/a	4.5 Ag 0.8 H
Dionex OnGuard II Ba	2.0 – 2.2	5.0 – 5.5
Dionex OnGuard II Ba/Ag/H	n/a	2.2 – 2.6 Ag 2.2 – 2.6 Ba 0.8 H
Dionex OnGuard II H	2.0 – 2.2	5.0 – 5.5
Dionex OnGuard II Na	2.0 – 2.2	5.0 – 5.5
Dionex OnGuard II M	0.4	1.0
Dionex OnGuard II P	6.0	n/a
Dionex OnGuard II RP	0.3 g resin	0.75 g resin

* All capacities are on a water-swollen basis.

Table 3 Sample Pretreatment Phase Chemistries

Phase	Functionality	Mode of Use	Common Uses
A	Anion-exchange Bicarbonate-form	Ion Exchange	pH adjustment Removes anions
Ag	Cation-exchange Silver-form	Precipitation Ion Exchange	Remove halides and others that precipitate with silver
Ba	Cation-exchange Barium-form	Precipitation Ion Exchange	Remove sulfate and others that precipitate with barium
H	Cation-exchange Hydronium-form	Ion Exchange	Remove alkali and alkaline earth metals, cationic transition metals: acidify sample
M	Iminodiacetate Ammonium-form	Chelation	Concentrate and elute transition metals
Na	Cation-exchange Sodium-form	Ion Exchange	Remove alkaline earth and cationic transition metals without a pH change
P	Poly-vinylpyrrolidone	H-bonding Complexation	Remove phenols, azo dyes, humic acids
RP	Poly-divinylbenzene	Adsorption	Remove neutral hydrophobic compounds

2. Cartridge Usage

2.1 Pretreatment Procedures for the Cartridges

For optimum results with Dionex OnGuard II cartridges, always observe the following basic principles of usage.

1. To provide the necessary pressure on the cartridges in manual pretreatment of samples, use a 5 mL syringe, especially when using a Dionex OnGuard II P cartridge. Back pressure in the Dionex OnGuard II P cartridge is higher than in the other types of Dionex OnGuard cartridges.

2. Before using a cartridge, flush it with the volume of deionized water or solvent recommended in Table 4, "Recommended Cartridge Preparation Procedures" (Columns 2-3) to remove trace ionic contaminants from the cartridge and condition the packing.

   EXAMPLE:
   Before application of the sample, flush the Dionex OnGuard II RP cartridge first with 5 mL of methanol and then with 10 mL of deionized water. When doing low level analyses (e.g., 100 ppm to 1 ppm chloride with loop injection), flush the cartridge as directed in Table 4, "Recommended Cartridge Preparation Procedures" (Column 4). Afterward, treat the cartridge with an additional 2 mL of deionized water and inject into the Ion Chromatograph to determine a blank level. If the cleaning was inadequate, flush the cartridge with additional water and then recheck.

3. To maximize loading on the cartridge bed, put the sample through at the recommended flow rate. See Table 4, "Recommended Cartridge Preparation Procedures" (Column 4). The most efficient use of the cartridge beds is obtained at sample flow rates less than 4.0 mL/min for sorption-based processes and less than 2.0 mL/min for ion exchange processes. Higher flow rates are practical if less than maximum loading is acceptable.

4. Always discard the recommended initial effluent volume. See Table 4, "Recommended Cartridge Preparation Procedures" (Column 5) when treating a sample or standard.

   EXAMPLE:
   After filling a 5 mL syringe with sample, push the first 3 mL into a waste container. Collect the next 2 mL for injection into the Ion Chromatograph.

5. If the substance to be removed by the cartridge is colored or otherwise visible in the cartridge (e.g., AgCl precipitate), use the cartridge until the visible band extends about three-fourths of the way to the cartridge outlet. For absolute determination of loading capacity for a specific application, aliquots of sample can be put through the cartridge and tested by the appropriate detection method (e.g., UV, pH, conductivity, or precipitation) until a breakthrough is observed.

   EXAMPLE:
   To test Cl^- breakthrough on the Dionex OnGuard II Ag cartridge, push 2 mL aliquots of sample through the cartridge into several small beakers containing 5-10 mL of 0.1 N $AgNO_3$. Breakthrough occurs when an AgCl precipitate is first observed in one of the beakers.

6. Optimum performance is achieved when the cartridges are used in a vertical direction.

Table 4 Recommended Cartridge Preparation Procedures

Cartridge Type Size	Flushing Reagent	Volume (mL)	Maximum Flow Rate (mL/min)	Initial Sample Volume to Discard (mL)
Dionex OnGuard II A				
1.0 cc Cartridge	DI Water	10	2	3
2.5 cc Cartridge		15	2	6
Dionex OnGuard II Ag				
1.0 cc Cartridge	DI Water	10	2	3
2.5 cc Cartridge		15	2	6
Dionex OnGuard II Ag/H				
2.5 cc Cartridge	DI Water	15	2	6
Dionex OnGuard II Ba				
1.0 cc Cartridge	DI Water	10	2	3
2.5 cc Cartridge		15	2	6
Dionex OnGuard II Ba/Ag/H				
2.5 cc Cartridge	DI Water	15	2	6
Dionex OnGuard II H				
1.0 cc Cartridge	DI Water	10	2	3
2.5 cc Cartridge		15	2	6
Dionex OnGuard II Na				
1.0 cc Cartridge	DI Water	10	2	3
2.5 cc Cartridge		15	2	6
Dionex OnGuard II M*				
1.0 cc Cartridge	2 M Ammonium acetate pH 5.5	10	2	See Section 2.13, “OnGuard II M”
2.5 cc Cartridge	2 M Ammonium acetate pH 5.5	15	2	See Section 2.13, “OnGuard II M”
Dionex OnGuard II P*				
1.0 cc Cartridge	DI Water	10	4	3
Dionex OnGuard II RP				
1.0 cc Cartridge	1. Methanol	5	4	3
	2. DI Water	10		
For ion-pair applications continue with:				
	3. 0.5 M TMAOH	5		
	4. DI Water	10		
2.5 cc Cartridge	1. Methanol	10	4	
	2. DI Water	15		
For ion-pair applications continue with:				
	3. 0.5 M TMAOH	10		
	4. DI Water	15		6

* The Dionex OnGuard II P and Dionex OnGuard II M cartridges may not appear full when received, but the bed will swell when flushed.

2.2 How to Set Up a Recovery Experiment

A matrix elimination recovery experiment evaluates the amount of analyte that is lost with the matrix on the sample pretreatment cartridge. In most good methods, the recovery should be close to 100%. The concentration of analyte that goes through the cartridge while matrix ions are being removed is determined.

2.2.1 For 1.0 cc and 2.5 cc cartridges

A. Pretreat cartridges as shown in Table 4, "Recommended Cartridge Preparation Procedures."

B. Calibrate the IC with a suitable standard.

C. For a 1.0 cc cartridge, push the IC standard through a cartridge, discard the first 3 mL of effluent and collect the next 2 mL for analysis. For the 2.5 cc cartridge, discard the first 6 mL of effluent and collect the next 2 mL for analysis.

D. Quantify the treated standard versus the calibration.
If the recovery is not acceptable, the sample may have been diluted by a volume of liquid already in the cartridge. Since the 3 mL discard volume is adequate for a 1.0 cc cartridge (6 mL for a 2.5 cc cartridge), the discarded volume may have been incorrect.

E. Make a standard addition to an aliquot of sample matrix and repeat the recovery calculation.
Low recovery in this step suggests that the analyte has co-precipitated and is trapped on the cartridge (in the case of Ag and Ba cartridges), or is bound to the matrix species being removed (in the case of P cartridge). You may be able to adjust sample pH to improve recoveries. For example, the sample pH should be lowered for use with a P cartridge. In the case of Ag and Ba cartridges, the sample should be diluted. When the matrix ion content is high, analyte can be trapped in the precipitate as it forms.

2.3 Determining the Discard Volume

Applications using Dionex OnGuard cartridge setups with large volumes of cartridge resin, such as the 2.5 cc, stacked 1.0 cc, or multilayer cartridges, may require the determination of the discard volume. The following steps provide a protocol for ensuring 0% dilution of the sample with residual wash water which can dilute the sample as it flows into the resin bed.

A. Apply a standard of known concentration.

B. Analyze successive 1-mL fractions and record the area counts for each fraction.

C. When successive 1-mL fractions become equal in area counts, no dilution is occurring.

D. Choose the discard volume to be the total volume collected through the second fraction where the area counts repeat.

E. This should occur between the 6-mL fraction and the 9-mL fraction for 2.5 cc, stacked 1.0 cc, and multilayer cartridges.

2.4 Dionex OnGuard II H

The Dionex OnGuard II H Cartridges (1.0 cc P/N 057085; 2.5 cc P/N 057086) contain sytrene-based, strong acid resin in the H^+ form. The resin has a very high selectivity for multivalent cations such as calcium and the transition metals. The cartridge is designed primarily for the removal of high levels of alkaline earths and transition metals from sample matrices, for the neutralization of caustic samples, and for the removal of carbonate. The cation exchange capacity is 2.0 – 2.2 meq/1.0 cc cartridge on a water-swollen basis and 5.0 – 5.5 meq/2.5 cc cartridge. With one 1.0 cc cartridge, 10 mL of 0.2 M NaOH may be neutralized before analysis; with a 2.5 cc cartridge, 25 mL of 0.2 M NaOH may be neutralized before analysis. The resin is stable over a pH range of 0-14.

2.4.1 Recovery Data

When using the Dionex OnGuard II H, recoveries of a group of carboxylic acids that included acetic, valeric, benzoic, succinic, and tartaric acids were greater than 98% at pH 4 or above, except for aromatic acids such as benzoic acid, where recoveries were greater than 95% at pH 5 or above.

NOTE

Adding a small amount of an organic solvent such as 2% isopropanol or acetonitrile to the sample improves recovery of aromatic acids.

2.4.2 Applications: Removal of Carbonate

Dionex OnGuard II H, in combination with nitrogen or helium sparging, is effective for removing carbonate from samples. The sample is passed through the cartridge in the standard method (See Table 4, "Recommended Cartridge Preparation Procedures"). This process acidifies the carbonate to carbonic acid. The carbonic acid is then removed by sparging the sample for 5 min. More than 400 ppm carbonate can be removed from more than 10 mL of sample, depending on the concentrations of other cations also present in the sample.

2.4.3 Acidification of Samples

Dionex OnGuard II H can be used in conjunction with Dionex OnGuard II RP to pre-acidify a sample prior to removing a matrix containing weak acid by adsorption onto the RP resin. The two cartridges are stacked H – RP for this type of application.

2.5 Dionex OnGuard II Na

The Dionex OnGuard II Na Cartridges (1.0 cc P/N 062948; 2.5 cc P/N 062962) contain a styrene-based, strong acid sulfonate resin in the Na^+ form. The resin has a very high selectivity for multivalent cations such as calcium and the transition metals without reducing the sample pH. This insures recovery of acid labile analytes such as nitrite. The cartridge is designed primarily for the removal of high levels of alkaline earth and transition metals from sample matrices. The cation exchange capacity is 2.0 – 2.2 meq/1.0 cc cartridge and 5.0 – 5.5 meq/2.5 cc cartridge on a water-swollen basis.

In comparison to the OnGuard II H cartridge, there would be no change in sample pH when using this cartridge.

2.5.1 Recovery Data

When using the OnGuard II Na, recoveries of the anions listed in Table 5 were greater than 97%.

Table 5 Recovery Data for OnGuard II Na

Analyte	AVERAGE
Fluoride concentration (ppm) % Fluoride Recovery	2.00 99.8%
Chloride concentration (ppm) % Chloride Recovery	19.7 98.4%
Nitrite concentration (ppm) % Nitrite Recovery	1.96 98.0%
Nitrate concentration (ppm) % Nitrate Recovery	1.94 97.3%
Sulfate concentration (ppm) % Sulfate Recovery	19.9 99.4%

2.6 Dionex OnGuard II Ag

The Dionex OnGuard II Ag Cartridges (1.0 cc P/N 057089; 2.5 cc P/N 057090) contain a high capacity, strong acid, cation exchange resin in the silver form. Except for the ionic form, it is very similar to the Dionex OnGuard II H packing. The Dionex OnGuard II Ag cartridge removes Cl^-, Br^-, I^-, AsO_4^{3-}, CrO_4^{2-}, CN^-, MoO_4^{2-}, PO_4^{3-}, SeO_3^{2-}, $SeCN^-$, SO_3^-, S^{2-}, SCN^- and WO_4^{2-} by precipitation. Recoveries for analytes of interest should be confirmed. Recovery of nitrite may be low in acidic samples, depending on the sample pH and the quantity applied to the cartridge.

The 1.0 cc cartridge has a capacity of 2.0–2.2 meq/cartridge on a water-swollen basis and the 2.5 cc cartridge has a capacity of 5.0–5.5 meq/cartridge on a water swollen basis. For example, approximately 9 mL of 1% NaCl may be treated with one 1.0 cc Dionex OnGuard II Ag cartridge and 23 mL of 1% NaCl may be treated with a 2.5 cc cartridge.

The Dionex OnGuard II Ag is often followed by a Dionex OnGuard II H cartridge. The Dionex OnGuard II H traps any silver ions that may breakthrough, thus protecting the analytical column.

NOTE

Samples with pH 7 or greater will form a black silver oxide precipitate in the cartridge.

2.6.1 Recovery Data

Recoveries of 2 ppm fluoride, 10 ppm nitrate, and 15 ppm sulfate are greater than 98%. If analyte recovery is low when removing halides from a high salt matrix, the sample should be diluted. Occasionally, analyte is trapped in the precipitate as it forms. Table 6, "Chloride Removal for Dionex OnGuard II Ag" shows the concentration of chloride remaining in a sample after treatment through a single 1.0 cc Dionex OnGuard II cartridge.

Table 6 Chloride Removal for Dionex OnGuard II Ag

Initial Chloride (mg/L)	Chloride Remaining (mg/L)
0	0.15
100	0.15
200	0.15
300	0.22
400	0.25

When phosphate is an analyte in a matrix passed through the Dionex OnGuard II Ag cartridge, the recovery of phosphate should be determined for the matrix. Phosphate may precipitate with Ag depending on the matrix composition. Phosphate recoveries should be determined by standard addition to the matrix.

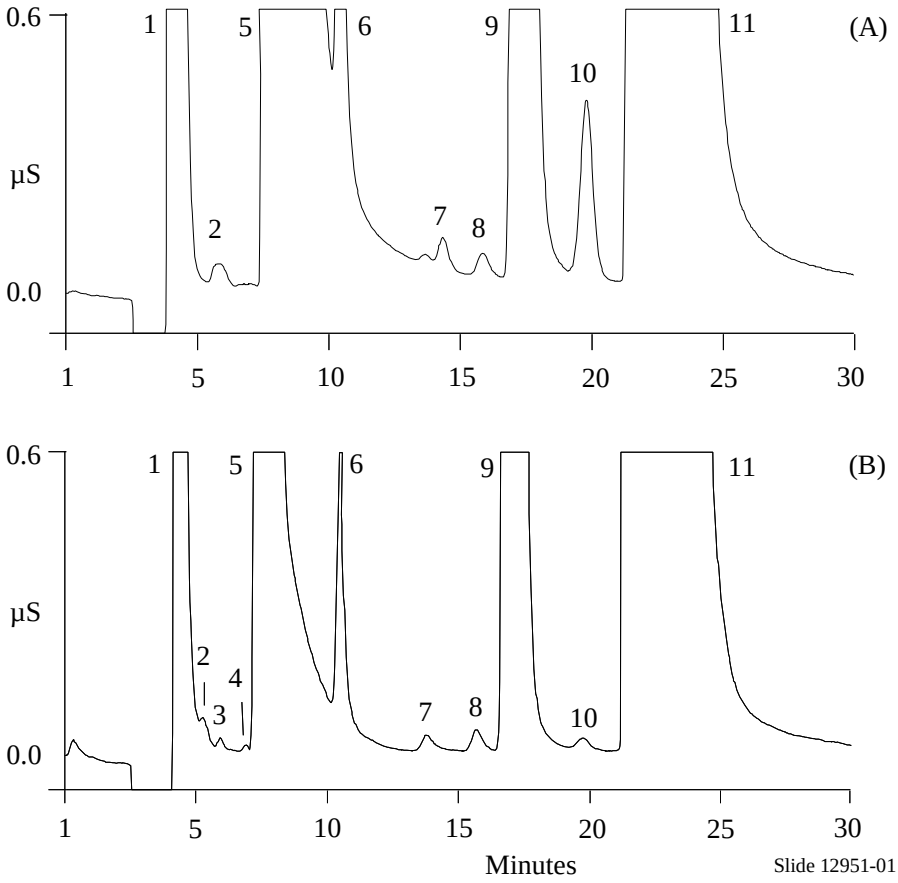
2.6.2 Determination of Bromate

The chromatograms below show the separation of bromide from chlorite after the concentration of chloride was reduced by Dionex OnGuard II Ag. Sample B was pretreated with Dionex OnGuard II Ag, H 1.0 cc cartridges. Samples also contained 200 mg/L bicarbonate.

Columns:	Dionex IonPac AG9-HC + AS9-HC
Eluent:	9.0 mM Sodium carbonate
Flow Rate:	1 mL/min
Inj. Volume:	500 µL
Detection:	Suppressed conductivity, ASRS ULTRA, AutoSuppression external water mode

Figure 3 Trace Bromate Determination Using Matrix Elimination/Preconcentration

Peaks	mg/L
1. Fluoride	1.0
2. Unknown	-
3. Chlorite	0.01
4. Bromate	0.005
5. Chloride	200.0
6. Nitrite	0.1
7. Bromide	0.01
8. Chlorate	0.01
9. Nitrate	10.0
10. o-Phosphate	0.1
11. Sulfate	200.0



2.7 Dionex OnGuard II Ag/H

The Dionex OnGuard II Ag/H Cartridge (2.5 cc P/N 057410) is a layered cartridge that contains Dionex OnGuard Ag II resin and Dionex OnGuard H II resin.

The Dionex OnGuard II Ag/H Cartridges contain a high capacity, strong acid, cation exchange resin in the silver form (Dionex OnGuard Ag II). The Dionex OnGuard II Ag cartridge resin removes Cl^-, Br^-, I^-, AsO_4^{3-}, CrO_4^{2-}, CN^-, MoO_4^{2-}, PO_4^{3-}, SeO_3^{2-}, $SeCN^-$, SO_3^-, S^{2-}, SCN^- and WO_4^{2-} by precipitation. Recoveries for analytes of interest should be confirmed. Recovery of nitrite may be low in acidic samples, depending on the sample pH and the quantity applied to the cartridge.

The Dionex OnGuard II Ag/H Cartridges also contain sytrene-based, strong acid resin in the H^+ form (Dionex OnGuard II H). The resin has a very high selectivity for multivalent cations such as calcium and the transition metals. The cartridge is designed primarily for the removal of high levels of alkaline earth and transition metals from sample matrices, for the neutralization of caustic samples, and for the removal of carbonate. The Dionex OnGuard H resin is placed at the outlet of the two-layer cartridge in order to trap soluble silver and other cations in the sample matrix. This two-layer cartridge can be used in place of two single cartridges used in series and has the added advantage of the higher silver-capacity.

The Dionex OnGuard II Ag/H cartridge has a capacity of 4.5 meq/cartridge (Dionex OnGuard II Ag) and 0.8 meq/cartridge (Dionex OnGuard II H).

2.8 Dionex OnGuard II P

The Dionex OnGuard II P Cartridge (1.0 cc P/N 057087) contains a polyvinylpyrrolidone (PVP) polymer with a very high selectivity for phenolics, azo containing compounds, aromatic carboxylic acids, and aromatic aldehydes. PVP also has a very high selectivity for triiodide. The cartridge is recommended for removing the phenolic fraction of humic acids, tannic acids, lignins, anthocyanins, and azo dyes from samples prior to either anion or cation analyses. These substances are all known foulants of anion exchange resins. The Dionex OnGuard II P can successfully treat these samples because the cartridge packing does not contain anion or cation exchange functionality. PVP provides selective removal of the species as compared to RP which removes species by non-specific adsorption.

A major application for Dionex OnGuard II P cartridges is the removal of phenolics from sample matrices such as ground water or inks. Loading capacities for phenolics on Dionex OnGuard II P are maximized at a sample pH of 2–4. Lignin sulfonate, pH adjusted to pH 3.7, can be removed from aqueous samples at a capacity of about 6 meq/1.0 cc cartridge.

2.8.1 Recovery Data

Recoveries of 2 ppm fluoride, 3 ppm chloride, 10 ppm nitrate, 15 ppm phosphate, and 15 ppm sulfate are greater than 98%. Recovery of nitrate at the 1 ppm level is greater than 95%.

Recoveries of species that bind to humic acids may be low since the humic acids bind to the PVP. There is a discussion of this problem in Bibliography Reference A.6. Table 7, "Recovery Data for Dionex OnGuard II P," shows the results of recovery experiments for some carboxylic acids. Selectivity of the Dionex OnGuard II P packing for protonated aliphatic carboxylic acids is slight compared to that for phenolics, azo-containing compounds, aromatic carboxylic acids, and aromatic aldehydes. When using the cartridge for an organic acid analysis, recovery determinations should be made for accurate results.

Table 7 Recovery Data for Dionex OnGuard II P

	pH 4.5	pH 6.1
Acetic acid, 5 ppm	100%	100%
Valeric acid, 20 ppm	94.5%	98.5%
Benzoic acid, 10 ppm	63.9%	100%
Succinic acid, 10 ppm	94.3%	98.1%
Tartaric acid, 10 ppm	99.2%	100.5%

NOTE

Results are accurate to within + 5%.

2.9 Dionex OnGuard II RP

The Dionex OnGuard II RP Cartridges (1.0 cc P/N 057083; 2.5 cc P/N 057084) contain a macroporous divinylbenzene reversed phase packing. This resin has a very high selectivity for hydrophobic substances, especially unsaturated or aromatic substances. It is compatible with all HPLC solvents. The resin is stable over a pH range of 0–14. Dionex OnGuard II RP can replace C-18 bonded phases for many applications, especially where sample pH is extreme. Dionex OnGuard II RP is recommended for removing the following types of substances from sample matrices: aromatic dyes, hydrocarbons, surfactants, and some carboxylic acids. It is also useful for trace enrichment, a technique in which substances are isolated and concentrated on the cartridge from sample matrices and then eluted from the cartridge in a clean minimum volume solvent. Since the packing contains no anion or cation exchange sites, the Dionex OnGuard II RP cartridge may be used prior to ion exchange analysis to remove hydrophobic contaminants.

In a reversed phase mode, the divinylbenzene resin functions as a hydrophobic substrate. Before using the cartridge with aqueous samples, treat it with methanol (see Table 4, “Recommended Cartridge Preparation Procedures”) to wet the packing and promote the most efficient use of the resin surface. Consider the following experimental results: two Dionex OnGuard II RP cartridges were treated with 5 mL of 1000 ppm aqueous sodium lauryl sulfate (SLS) at 4.0 mL/min. One cartridge was pretreated with methanol; the other was not. The eluate from the pretreated cartridge contained 24 ppm of SLS, while the eluate from the untreated, semi-dry cartridge contained 979 ppm of SLS. Very little of this anionic surfactant was retained on the untreated cartridge.

2.9.1 Recovery Data

Recovery of carboxylic acids is pH dependent. At an acidic pH, where the carboxylic acids are protonated, significant losses occur. Table 8, "Recovery Data for OnGuard II RP," summarizes the data generated for a group of carboxylic acids that included monovalents, an aromatic, and divalents. The initial 3 mL of sample from the cartridge was discarded (see Table 4, "Recommended Cartridge Preparation Procedures") and the data was calculated using the following 2 mL of cartridge effluent.

As indicated in Table 8, "Recovery Data for Dionex OnGuard II RP," the addition of a small amount of isopropanol or other organic solvent aids recovery of the more hydrophobic acids.

Table 8 Recovery Data for Dionex OnGuard II RP

Cartridge	pH 2.5 (aq)	pH 4.5 (aq)	pH 4.5 (2% IPA)	pH 6 (aq)
Acetic acid, 5 ppm	50%	81%	100%	100%
Valeric acid, 20 ppm	0%	0%	10%	3%
Benzoic acid, 10 ppm	0%	3%	14%	8%
Succinic acid, 10 ppm	50%	100%	100%	98%
Tartaric acid, 10 ppm	100%	101%	100%	100%

NOTE

Results are accurate to within + 5%.

This data illustrates that organic acids can be removed from sample mixtures following acidification to pH < 2.5. See Section 2.3.3, "Acidification of Samples."

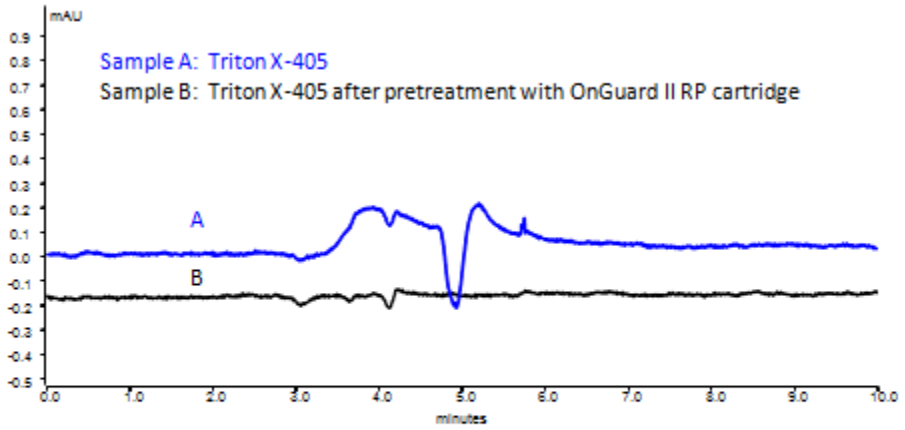
2.9.2 Applications: Removal of Ionic Surfactants

In addition to use in a reversed phase mode, Dionex OnGuard II RP can also be used in an ion pairing mode to remove ionic substances. The recommended ion pairing reagent is tetramethylammonium hydroxide (see Table 4, "Recommended Cartridge Preparation Procedures," for use conditions). Significant losses of the inorganic anions occur when quaternary ammonium type ion pairing reagents larger than tetramethyl (e.g., tetraethyl and tetrapropyl) are used. Any substance that can be retained by MPIC using NH_4OH or TMAOH reagents can be removed from a matrix on Dionex OnGuard II RP in the ion pairing mode. In general, higher loading capacities for anionic surfactants can be obtained in the ion pairing mode than in the reversed phase mode: about 20 mg of sodium lauryl sulfate (SLS) / 1.0 cc cartridge can be removed in the reversed phase mode, while 90 mg of sodium lauryl sulfate (SLS) / 1.0 cc cartridge can be removed in the ion pairing mode with TMAOH reagent. Recoveries of fluoride, chloride, phosphate, nitrate, and sulfate are greater than 98% in the reversed phase mode. Recoveries for these anions in the ion pairing mode, using TMAOH reagent, are greater than 96%.

2.9.3 Applications: Removal of Nonionic Surfactants

Neutral surfactants can interfere with early-eluting peaks, especially with UV detection. The chromatogram below shows a sample of Triton X-405 before and after the concentration of surfactants was reduced by Dionex OnGuard II RP. Sample B was pretreated with a Dionex OnGuard II RP cartridge (1.0 cc).

Figure 4 Use of OnGuard II RP to remove early-eluting surfactants



2.9.4 Applications: Removal of Fat

Fat can cause loss in column capacity and easily foul a detector electrode by adsorbing to the metal surface and interfering with the adsorption of the analyte. The peak response will be low and will decrease injection-to-injection. In the following example, milk was passed through an Dionex OnGuard II RP cartridge prior to analysis by IC with pulsed amperometric detection. The analyte of interest is iodide. A complete discussion of this method is available in Bibliography Reference A.7, "Determination of Iodide in Milk Products."

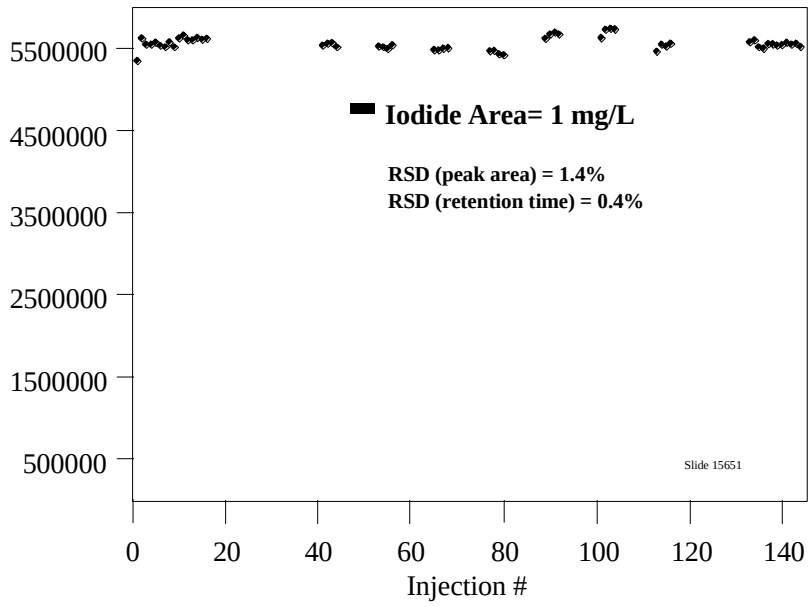
Sample Preparation:

A. Pre-wet an Dionex OnGuard II RP 1.0 cc cartridge with 5 mL methanol followed by 10 mL deionized water; for a 2.5 cc cartridge, use 10 mL methanol followed by 15 mL deionized water.
B. Precipitate the milk protein by adding 2 mL 3% acetic acid to 10 mL sample.
C. Filter.
D. Pass 5 mL filtrate through the cartridge, discarding the first 3 mL.

Failure to remove fat prior to IC with pulsed amperometric detection will lead to high column back pressure and loss in column capacity.

The plot below shows the reproducibility achieved for iodide determinations made between injections of 2% milk. The blank sections were injections of milk samples. For more information, see Dionex Application Note 37, "Determination of Iodide in Milk Products."

Figure 5 Fat Removal from Milk for Iodide Analysis Using IC with Pulsed Amperometric Detection



2.10 Dionex OnGuard II A

The Dionex OnGuard II A Cartridges (1.0 cc P/N 057091; 2.5 cc P/N 057092) contain styrene-based, strong base anion exchange resin in the HCO_3^- form. The resin is selective for anionic species and is designed primarily for the removal of anionic contaminants in sample matrices or neutralization of acidic samples. The Dionex OnGuard II A cartridge is also very useful in removing anionic amino acids, peptides, proteins and other contaminants from glycoproteins, hydrolysates, or carbohydrate samples. The anion exchange capacity is about 0.7 meq / 1.0 cc cartridge and 1.75 meq / 2.5 cc cartridge on a water-swollen basis. The resin is stable over a pH range of 0–14.

2.10.1 Pretreatment of Low pH Samples for Cation Analysis on the IonPac CS12A-5µm

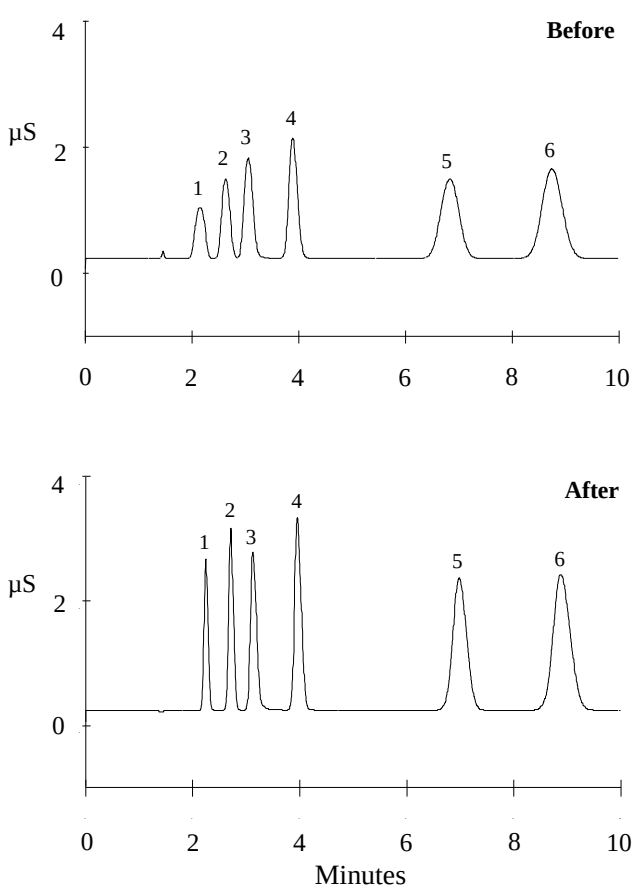
Weak cation exchange columns have a limited pH range because the weak cation exchange sites on the packings protonate at low pH. The chromatographic result is that there is a loss in peak efficiency, as shown in Figure 6, "Pretreatment of Low pH Samples for Cation Analysis on the IonPac CS12A-5µm." Dionex OnGuard II A 1.0 cc contains anion exchange resin in the bicarbonate form. Low pH samples can be adjusted to about pH 6 using this cartridge and the expected peak efficiencies are obtained.

Figure 6 Pretreatment of Low pH Samples for Cation Analysis on the Dionex IonPac CS12A-5µm

Column:	Dionex IonPac CS12A-5µm
Eluent:	22 mN Sulfuric Acid
Flow Rate:	0.5 mL/min
Loop size:	25 µL
Sample:	Standard in 50 mM methanesulfonic acid

Analytes	**mg/L**
Lithium	0.12
Sodium	0.5
Ammonium	0.62
Potassium	1.25
Magnesium	0.62
Calcium	1.25

where 1 mg/L = 1 ppm



2.10.2 Recovery Data

Recoveries of 5 ppm lithium, 20 ppm sodium, 20 ppm potassium, 20 ppm magnesium, and 100 ppm calcium are greater than 98%.

2.11 Dionex OnGuard II Ba

The Dionex OnGuard II Ba Cartridges (1.0 cc P/N 057093; 2.5 cc P/N 057094) contain a barium-form, high capacity, strong acid cation exchange resin. The cation exchange capacity is 2.0 to 2.2 meq/ 1.0 cc cartridge on a water-swollen basis and 5.0 to 5.5 meq / 2.5 cc cartridge. When used properly, the Dionex OnGuard II Ba cartridge removes sulfate and chromate by precipitation. Recoveries for analytes of interest should be confirmed.

Precipitation of sulfate by barium-form resin is dependent on the elution of barium ion from the resin by sample matrix cations. Results therefore can be highly variable. The 1.0 cc Dionex OnGuard II Ba Cartridge will reduce sulfate from a 100 ppm sodium sulfate sample to about 15 ppm. If the spiking technique described in 2.11.1 is used, the sulfate from a 100 ppm sodium sulfate sample is reduced to about 0.5 ppm.

2.11.1 Applications: Optimized Removal of Sulfate

NOTE

This technique uses several types of Dionex OnGuard Cartridges.
Please check that your analytes of interest are not removed by any of the cartridges.

Thermo Fisher Scientific has developed a technique for assuring optimum removal of sulfate by Dionex OnGuard II Ba independent of sample matrix composition. In this method, samples are spiked with a divalent displacing cation, such as Ca^{2+} or Mg^{2+} in order to ensure that Ba^{2+} is available for precipitation with SO_4^{2-} from the sample. Co-precipitation of various analyte anions with $BaSO_4$ is minimized by spiking the sample with CO_3^{2-}. Low recovery of some oxyanions can be significantly improved by the addition of CO_3^{2-} to the samples prior to pretreatment with the barium-form device. The chloride salt of the displacing cation is used because the Cl^- counterion can be trapped on the silver-form resin. Carbonate can be removed by acidifying through H-form resin and sparging. Low recoveries of anions in the presence of barium sulfate may be due to co-precipitation. The addition of carbonate improves recoveries to varying degrees depending on the analyte (see Table 9, “Recovery of Oxyanions from Ba^{2+}-form Resin”). Recoveries of analytes of interest should always be determined in the sample matrix. In this technique, as in any other for sample pretreatment, the recovery for the analytes of interest must be determined for each sample type.

Lower concentrations of Ca^{2+} may be used if less than maximum sulfate removal is required. The Ca^{2+} is effective in eluting the Ba^{2+} from the resin so that it can precipitate with sulfate. Dionex OnGuard II Ba is followed by Dionex OnGuard II Ag in order to remove the chloride contributed to the sample by the calcium chloride as well as the other ions indicated in Section 2.4, “Dionex OnGuard II Ag.” This technique is outlined below.

Materials Needed:

A. Dionex OnGuard II Ba

B. Dionex OnGuard II Ag

C. Dionex OnGuard II H (or in-line MetPac CC-1 for Ag^+ trapping only if removal of carbonate is not required)

D. Extra-pure calcium chloride

E. 0.22 µm, 25 mm disposable filter, polypropylene or polysulfone

Procedure:

A. Prepare Dionex OnGuard cartridges as described in Table 4, "Recommended Cartridge Preparation Procedures."

B. Connect these cartridges in order of Ba – Ag – H. See Figure 7, "Manual Use of Multiple Dionex OnGuard Cartridges for Sulfate Removal," In Section 2.11.4.

C. Prepare a 0.5 M $CaCl_2$ solution from extra-pure calcium chloride.

D. Spike the sample to 100 mg/L Ca^{2+} or Mg^{2+} using chloride salts. Spike with CO_3^{2-} if oxyanions are the analytes of interest.

E. Apply spiked sample to the cartridge train at less than 2 mL/min. Follow the manual protocol i.e., discard the first 3 mL for 1.0 cc cartridge and 6 mL for the 2.5 cc cartridge.

2.11.2 Recovery Data

Table 9 Recovery of Oxyanions from Ba^{2+}- form Resin Following Figure 7, "Manual Use of Multiple Dionex OnGuard Cartridges for Sulfate Removal"

Matrix	Sample Matrix Composition (in meq/L) SO_4^{2-}	Cl^-	Na^+	Ca^{2+}	CO_3^{2-}	Comments
1	1.0	5.0	2.1	5.0	0	Matrix without CO_3^{2-} but with Ca^{2+}
2	1.0	5.0	5.4	5.0	3.3	Matrix with both CO_3^{2-} and Ca^{2+}

Analytes	Sample Matrix Composition 1 Added ng	1 Recovered %	2 Added ng	2 Recovered %
NO_3^-	10	46	10	90
BrO_3^-	10	37	10	100
SeO_3^-	20	7	20	18
ClO_3^-	15	75	15	98
SeO_4^{2-}	25	89	25	97
AsO_4^{3-}	10	94	10	96
PO_4^{3-}*	20	0	na	

*Samples in a matrix of 1 mM Na_2SO_4 (without Ca^{2+}) show a 42 % recovery of phosphate.

Please see Appendix A, Bibliography References A.3 for a complete discussion of this method.

2.11.3 Applications: Trace Level Bromate Analysis

Complete details for this method can be found in Application Note 101, "Trace Level Determination of Bromate in Ozonated Drinking Water by Ion Chromatography." The following is a summary of the procedure.

A. Set up a 3-cartridge Dionex OnGuard train as described in Section 2.9.1, "Applications: Optimized Removal of Sulfate."

B. Prepare 0.5 M $MgCl_2 \cdot 7H_2O$.

C. Prepare 0.167 M Na_2CO_3.

D. Spike 9.8 mL of sample with 100 µL of the magnesium chloride solution and 100 µL of the sodium carbonate solution. This produces a sample with 0.005 M Mg^{2+} and 100 mg/L CO_3^{2-}. The addition of carbonate to the sample aids bromate recovery. Mg^{2+} is used rather than Ca^{2+} because $MgCO_3$ is soluble at this level, as compared to $CaCO_3$.

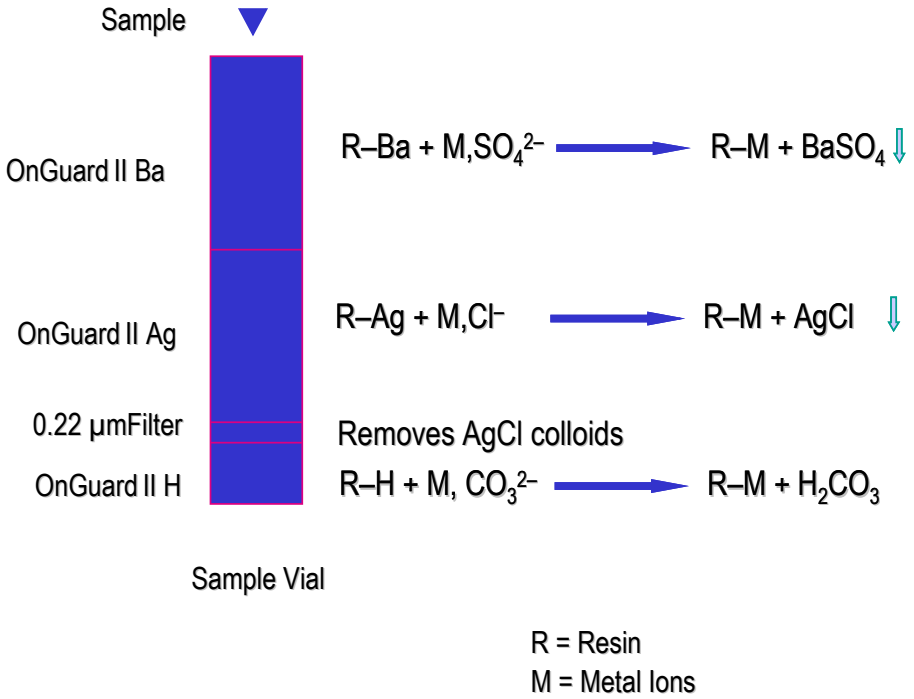
E. Apply sample as in Figure 7, "Manual Use of Multiple Dionex OnGuard Cartridges for Sulfate Removal," including sparging to remove the carbonate prior to ion chromatographic analysis.

2.11.4 Applications: Optimized Removal of Sulfate

NOTE

This technique uses several types of Dionex OnGuard Cartridges. Please check that your analytes of interest are not removed by any of the cartridges.

Figure 7 Manual Use of Multiple Dionex OnGuard Cartridges for Sulfate Removal



2.12 Dionex OnGuard II Ba/Ag/H

The Dionex OnGuard Ba/Ag/H cartridge (2.5 cc P/N 063955) is a layered cartridge that contains Dionex OnGuard II Ba resin, Dionex OnGuard Ag resin, and Dionex OnGuard II H resin.

The Dionex OnGuard II Ba/Ag/H cartridges contain a barium-form, high capacity, strong acid cation exchange resin (Dionex OnGuard II Ba). The Dionex OnGuard II Ba resin removes sulfate and chromate by precipitation. Recovery for analytes should be confirmed.

The Dionex OnGuard II Ba/Ag/H cartridges also contain a silver form, high capacity, strong acid cation exchange resin (Dionex OnGuard II Ag). The Dionex OnGuard II Ag resin removes Cl^-, Br^-, I^-, AsO_4^{3-}, CrO_4^{2-}, CN^-, MoO_4^{2-}, PO_4^{3-}, SeO_3^{2-}, $SeCN^-$, SO_3^-, S^{2-}, SCN^- and WO_4^{2-} by precipitation. Recoveries for analytes of interest should be confirmed. Recovery of nitrite may be low in acidic samples, depending on the sample pH and the quantity applied to the cartridge.

The Dionex OnGuard II Ba/Ag/H cartridges also contain sytrene-based, strong acid resin in the H^+ form (Dionex OnGuard II H). The resin has a very high selectivity for multivalent cations such as calcium and the transition metals. The cartridge is designed primarily for the removal of high levels of alkaline earth and transition metals from sample matrices, for the neutralization of caustic samples, and for the removal of carbonate. The Dionex OnGuard II H resin is placed at the outlet of the three-layer cartridge in order to trap soluble silver and other cations in the sample matrix. This three-layer cartridge can be used in place of two single cartridges used in series and has the added advantage of the higher silver-capacity.

The Dionex OnGuard II Ba/Ag/H cartridge has a capacity of 2.2 – 2.6 meq/cartridge for Ba resin, 2.2 – 2.6 meq/cartridge for Ag resin, and 0.8 meq/cartridge for H resin.

2.13 Dionex OnGuard II M

Dionex OnGuard II M cartridges (1.0 cc P/N 057137; 2.5 cc P/N 057095) contain iminodiacetate resin in the ammonium form and are available in both 1.0 cc (0.4 meq) and 2.5 cc (1.0 meq) sizes. The Dionex OnGuard II M cartridges are designed for concentrating or removing metals from complex matrices at pH > 4 while allowing alkali and alkaline-earth metals to pass through. The transition metals can then be eluted using 0.5 M nitric acid.

The 1.0 cc product is better suited for matrix elimination of samples above pH 4. The 2.5 cc product is better suited for concentration of transition and re-elution for analysis by IC or ICP-MS. The resin is supplied in the ammonium-form, ready for use at pH 5.5. The resin shrinks at least 50% in acid or when dry. Depending on the ionic strength and sample pH, considerable channeling in the resin bed can occur, causing premature breakthrough of the sample. Therefore, the larger bed has been designed to allow good analytical precision for concentration work.

2.13.1 Concentration of Transition Metals

Transition metals cannot be concentrated if the sample pH is below 4 or if they are not in the cationic form. Therefore, metals that form anions in water cannot be concentrated. Metals that can be concentrated include Cd, Cu, Co, Fe, Mn, Ni, Pb, and Zn. While Cr(III) can be concentrated, it is very difficult to elute. The Cr(VI) forms an anion as chromate and cannot be concentrated. The 2.5 cc bed of Dionex OnGuard II M allows for changes in bed volume as the resin-form changes during use. The bed will shrink about 50% when acid is applied during the elution step.

A. Equipment

All water, reagents, and equipment must be metal-free. Vials and other containers should be acid-washed by soaking in 0.5-1 M metal-free nitric acid (Trace metal grade) for 1 hour. Samples containing particulates should be filtered through 0.45 µm polypropylene filters. These filters can be attached to the top of the cartridges so that filtering and concentration are accomplished in one step.

1. Dionex OnGuard II M, 2.5 cc.
2. Syringes – all plastic, 10 mL such as disposable GRAF Fortuna available from Alltech as P/N Z11,687-4 or Hamilton Gastight all glass/Teflon construction.
3. Ammonium acetate, 2 M, pH 5.5, Dionex P/N 033440.
4. Nitric acid, 0.5 M; make from Dionex 2.0 M nitric acid P/N 033442, trace metal grade.
5. Scintillation vials.
6. Barrel-type reservoirs such as Dionex P/N 041233, 5 mL.
7. Vacuum station or filter flask with 3-hole stopper.
8. Luer-adaptor for backflushing a cartridge; female-to-female connection.

B. Method for Concentration and Elution of Transition Metals

1. Weigh the liquid sample.
2. Dilute 1:1 with ammonium acetate to buffer to above pH 4. Less buffer can be used if the sample pH is less acidic.
3. Attach reservoir to cartridge and place on vacuum station.
4. Pour 3-5 mL ammonium acetate into barrel and aspirate through system.
5. Pour sample into reservoir and aspirate through to remove all free liquid.
6. Remove barrel from vacuum station and attach an all-plastic syringe and adaptor to outlet of the cartridge. Backflush with about 3 g of 0.5 M nitric acid. If eluting metals in the same direction as the concentration, flush with 9 g of 0.5 M nitric acid. It is convenient to elute into a scintillation vial placed on an analytical balance.
7. Record the elution weight for future calculations.

C. Interferences

Chelators, such as EDTA, interfere with the concentration of metals on the Dionex OnGuard II M resin. This is seen as a smearing of the yellow color of iron along the cartridge and as poor concentration efficiency. If organic chelators are present, the samples should be digested using EPA method 200.8. After digestion, we achieved recoveries of the listed metals in the range of 99–117%, with the exception of manganese. Recovery of manganese was considerably lower (about 32%).

2.13.2 Matrix Elimination of Transition Metals

Transition metals are concentrated at pH 4 or above and eluted at pH 2 or below. Therefore, samples for matrix elimination where the transition metals will be retained on the cartridge must have a pH 4 or above. This can be achieved by buffering as described in Section 2.9.1.2, "Method for Concentration and Elution of Transition Metals."

Matrix elimination of transition metals can also be achieved using Dionex OnGuard II H, a cation exchange resin in the hydronium ion form. The anion blanks for chloride and sulfate will be somewhat higher from the H resin although the mechanism is not pH dependent.

Appendix – Dionex OnGuard Bibliography

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