

Finally, easy sample prep for IC

Thermo Scientific™ Dionex™ Guardcap™ Vial Caps

Keywords

Cation removal, anion removal, metal removal, surfactant removal, hydrocarbon removal, aromatic dye removal, organic removal, hydrophobic substances removal, carboxylic acid removal, neutralization, automated inline sample preparation, matrix elimination, sample filtration

Benefits

- Removes up to 1000 mg/L of divalent cations (e.g., calcium or transition metals) from 800 μ L of sample (Dionex Guardcap H and Dionex Guardcap Na vial caps)
- Removes non-ionic surfactants, hydrocarbons, aromatic dyes, hydrophobic substances, and protonated carboxylic acids (Dionex Guardcap HRP vial caps)
- Removes up to 800 mg/L of anions (e.g., sulfate) from 250 μ L of sample (Dionex Guardcap AEX vial caps)
- Neutralizes up to 500 μ L of 50 mM NaOH or other base (Dionex Guardcap H vial caps)
- Neutralizes up to 250 μ L of 30 mM MSA, HCl, or other monovalent acid (Dionex Guardcap AEX vial caps)
- Filters better than 80% of particulate matter greater than 20 μ m in diameter
- Use with any ion chromatography system that includes a Dionex AS-DV autosampler
- Reduces time, labor, and cost required for offline sample preparation techniques
- Increases the lifetime of analytical columns and suppressors by removing harmful metals, matrix ions, and particulates from samples



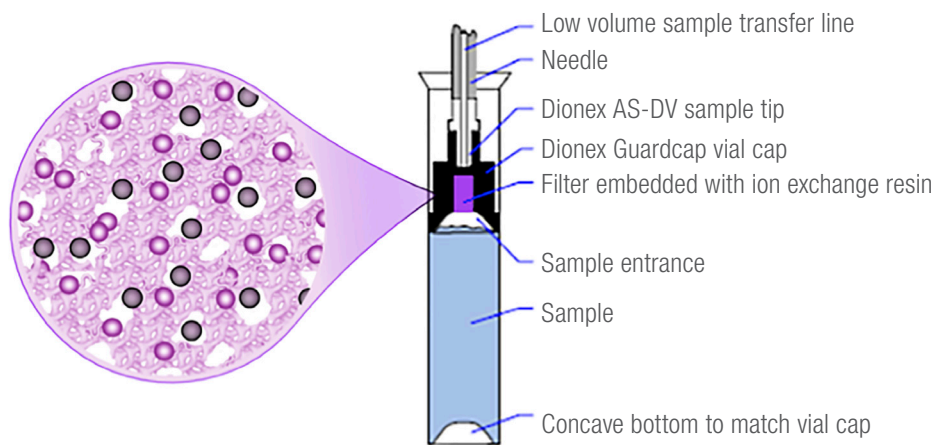


Figure 1. Illustration showing the HDPE filter matrix in the Dionex Guardcap vial cap, which is embedded with spherical Thermo Scientific™ Dionex™ OnGuard™ resin particles

Dionex Guardcap vial caps

Thermo Scientific™ Dionex™ Guardcap™ vial caps are designed to be used with Thermo Scientific™ Dionex™ PolyVial™ sample vials in Thermo Scientific™ Dionex™ AS-DV autosamplers to facilitate automated inline sample preparation for ion chromatography determinations. The unique Dionex PolyVial sample vial incorporates a vial cap that acts as a simple piston to force sample out of the vial. Dionex Guardcap vial caps fit 5 mL Dionex PolyVials and contain ion exchange resin embedded in a high density polyethylene filtration matrix. This vial/cap system can now be used for hassle-free sample preparation, including removal of alkaline earth and transition metals, removal of anions, removal of hydrophobic substances, neutralization of acids and bases, and filtration of particulates using Dionex Guardcap vial caps.

Benefits of automated inline sample preparation

Traditional offline sample preparation cartridges require labor and disposable syringes to first condition the cartridges and then to pass sample through them. Dionex Guardcap vial caps require no conditioning prior to use; they are pre-wetted and ready for use when opened. In addition, they can improve the reproducibility of analyses because important parameters such as timing, flow rate, and delivery volume are easily set and controlled by the autosampler.

Traditional inline sample preparation cartridges require additional switching valves and loading Figure 1. Illustration showing the HDPE filter matrix in the Dionex Guardcap vial cap, which is embedded with spherical Thermo Scientific™

Dionex™ OnGuard™ resin particles pumps that can add significant cost and complexity to the method. Dionex Guardcap vial caps require no additional hardware for systems that use Dionex AS-DV autosamplers. Simply install these functionalized vial caps on your sample vials and your samples are ready for analysis. Sample preparation is performed inline, within the Dionex Guardcap vial cap, and automated by the autosampler.

The chemistry

Dionex Guardcap H vial caps contain sulfonated strong cation exchange resin in the hydronium ion form embedded in a high density polyethylene (HDPE) matrix. A cylinder of this composite material is incorporated into the cap as shown in Figure 1. Each Dionex Guardcap H vial cap has enough capacity to exchange cations and simultaneously neutralize base or remove high concentrations of divalent cations. Some examples are shown in Table 1. Samples with higher concentrations of base should be diluted prior to treatment with Dionex Guardcap H vial caps.

Dionex Guardcap Na vial caps contain sulfonated strong cation exchange resin in the sodium form. Each Dionex Guardcap Na vial cap has enough capacity to remove high concentrations of divalent cations without changing the pH of the sample. This is ideal for pH-sensitive analytes (e.g., nitrite) that are affected by the pH change resulting from sample prep with hydronium-form resins. Removal of metals from samples requiring determination of nitrite must be accomplished through sodium-form resin to ensure quantitative recovery (>99%).

Table 1. Compatible sampling volume and sample matrix concentrations for Dionex Guardcap vial caps

Product	Matrix elimination application	Sampling volume	Maximum sample concentration
Dionex Guardcap H / Dionex Guardcap Na	Divalent cations, metals	Up to 800 µL	1000 ppm (e.g., Ca ²⁺ , Pb ²⁺)
Dionex Guardcap H / Dionex Guardcap Na	Divalent cations, metals	Up to 275 µL	1800 ppm (e.g., Ca ²⁺ , Pb ²⁺)
Dionex Guardcap H	Monovalent cation with neutralization of base	Up to 500 µL	50 mM NaOH or other base
Dionex Guardcap AEX	Anions with neutralization of acid	Up to 250 µL	800 mg/L (e.g., sulfate) 30 mM MSA, HCl, or other monovalent acid
Dionex Guardcap HRP	Hydrophobic organic species	Up to 500 µL	0.01% Triton™ x-405 or other non-ionic surfactant 20 mg benzoic acid or other protonated carboxylic acid

Dionex Guardcap HRP vial caps contain a proprietary styrene-divinylbenzene resin grafted with a hydrophilic monomer that helps maintain the pre-wetted state required for maximum retention of hydrophobic compounds. Since the resin contains no anion or cation exchange sites, Dionex Guardcap HRP vial caps may be used to remove hydrophobic contaminants prior to ion exchange analysis. Dionex Guardcap HRP vial caps have the same selectivity as Dionex OnGuard II RP cartridges and remove species by non-specific adsorption. Each Dionex Guardcap HRP vial cap has enough capacity to remove up to 0.01% non-ionic surfactant (e.g., Triton™ x-405) from 500 µL of sample. Dionex Guardcap HRP vial caps can also remove protonated carboxylic acids (e.g., up to 20 mg benzoic acid at pH <4).

Dionex Guardcap AEX vial caps contain strong anion exchange resin in the borate form. Each Dionex Guardcap AEX vial cap has enough capacity to exchange anions and simultaneously neutralize acids. Some examples are shown in Table 1. Samples with higher concentrations of acid should be diluted prior to treatment with Dionex Guardcap AEX vial caps.

The following application examples illustrate several uses of Dionex Guardcap vial caps for sample preparation.

Removal of metals and protection from metal contamination

Dionex Guardcap H resin is provided in the H⁺ form, meaning that alkali, alkaline earth, and transition metal cations are exchanged for hydronium ions, thus acidifying the sample. This application is useful for removing cations from the sample matrix that could potentially precipitate on system components or become trapped in the anion suppressor. This method of use is analogous to using Thermo Scientific™ Dionex™ OnGuard™ II H cartridges for offline sample preparation removal of metals and other higher valency cations.

Figure 2 shows the determination of methanesulfonate in a lead nitrate matrix using Dionex Guardcap H sample preparation. The inset in Figure 2 shows the results of passing 1 mL of a 500 ppm Pb²⁺ standard through a Dionex Guardcap H vial cap. The Dionex Guardcap H vial cap effectively removes the Pb²⁺ ions from the standard.

Column: Thermo Scientific™ Dionex™ IonPac™ AS20 (2 × 250 mm)
 Eluent: 5-40 mM KOH from 0 to 15 minutes
 Eluent Source: Dionex EGC 500 KOH Cartridge
 Flow Rate: 0.25 mL/min
 Inj. Volume: 2.5 µL
 Sampling Volume: 500 µL
 Detection: Suppressed Conductivity, Thermo Scientific™ Dionex™ AERS™ 500 Suppressor, AutoSuppression, Recycle mode

- Peaks:
1. Fluoride
 2. Methanesulfonate
 3. Chloride
 4. Nitrate
 5. Sulfate
 6. Phosphate

Inset: MQuant™ Lead Test (Merck KGaA)

- A. 1 mL 500 ppm Pb²⁺ through Dionex Guardcap H
 B. 500 ppm Pb²⁺

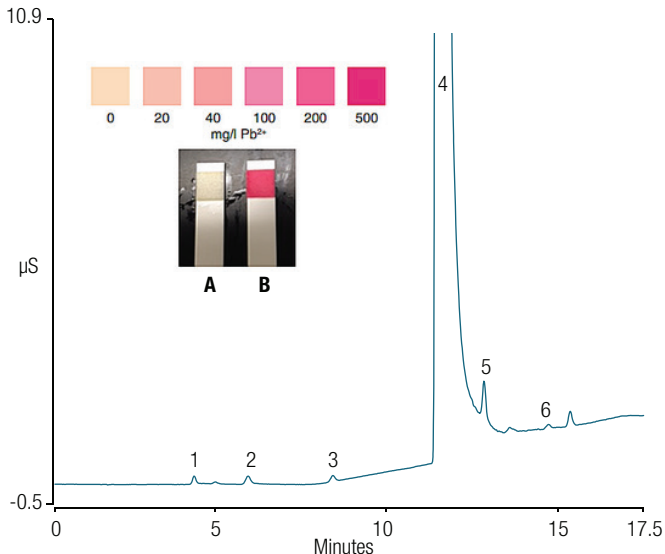


Figure 2. Determination of methanesulfonate in a lead nitrate matrix using Dionex Guardcap H sample preparation

Column: Dionex IonPac AS23 (2 × 250 mm)
 Eluent: 4.5 mM Na₂CO₃ / 0.8 mM NaHCO₃
 Eluent Source: Dionex AS23 eluent concentrate
 Flow Rate: 0.25 mL/min
 Inj. Volume: 2.5 µL
 Detection: Suppressed Conductivity, Dionex AERS 500 Carbonate Suppressor, AutoSuppression, Recycle mode

- A: Chromatogram using a clean Dionex AERS 500 Carbonate suppressor
 B: Chromatogram using a Dionex AERS 500 Carbonate suppressor fouled with 500 µL of 24,000 mg/L Cu(II) from copper nitrate

- Peaks:
1. Fluoride
 2. Chloride
 3. Nitrate
 4. Phosphate
 5. Sulfate

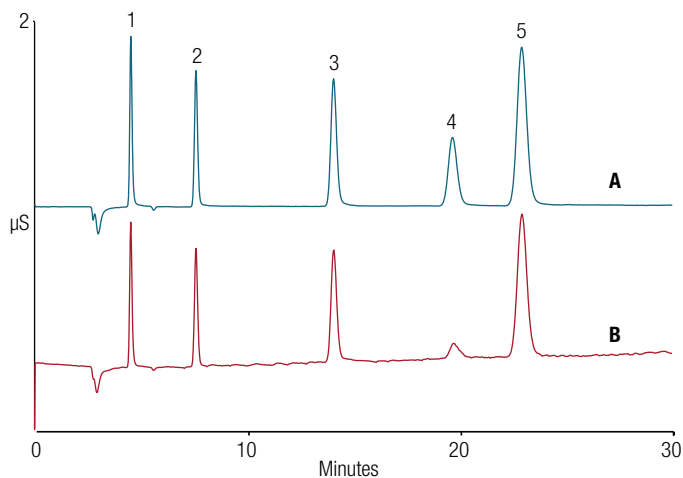


Figure 3. Comparison of inorganic anion analysis using clean and fouled suppressors

Many sample types contain matrix metal cations that can foul the cation exchange sites of anion suppressors with extended use. Figure 3 shows the chromatographic effects of using a suppressor that has been fouled with metals. Fouling of the anion suppressor with metals such as copper results in increased baseline noise and decreased sensitivity for all peaks, especially phosphate. Use of Dionex Guardcap H or Dionex Guardcap Na vial caps can prevent this type of fouling, saving downtime associated with troubleshooting and cost associated with premature replacement.

Dionex Guardcap Na resin is provided in the Na⁺ form, meaning that alkali, alkaline earth, and transition metals in the cation form, and protonated weak bases, including amines, are exchanged for sodium ions. The sodium

content of the sample increases, and all cations that could potentially precipitate or be trapped on system components are removed from the sample without changing the sample pH. This method of use is analogous to using Thermo Scientific™ Dionex™ OnGuard™ II Na cartridges for offline sample preparation removal of metals and other higher valency cations. Since sample pH remains unchanged, a Dionex Guardcap Na vial cap is ideal for treating samples that contain pH-sensitive analytes. Table 2 shows a comparison of nitrite recoveries for a sample of 1 ppm nitrite in a matrix with 500 ppm calcium treated with both Dionex Guardcap H and Dionex Guardcap Na vial caps. Note that quantitative recovery of nitrite (> 99%) is only possible with Dionex Guardcap Na vial caps.

Table 2. Comparison of 1 ppm nitrite recovery in 500 ppm calcium (CaSO₄) using Dionex Guardcap H and Dionex Guardcap Na vial caps

Dionex Guardcap H			Dionex Guardcap Na		
% Recovery*	% RSD (n=7)	pH after sample prep	% Recovery*	% RSD (n=7)	pH after sample prep
66%	9.3%	2	101%	2.7%	6

*compared to 1 ppm nitrite standard in deionized water matrix

Neutralization of base

The Dionex Guardcap H resin is provided in the H⁺ form. When a cation such as sodium is removed from the sample, it is replaced by the hydronium ion and this can neutralize bases that are present in the sample. This is especially useful for lowering the pH of high pH samples that will be injected into a system that determines anions with a gradient separation.

Figure 4 shows the effects of a high pH matrix injected onto a Dionex IonPac AS17-C column and the improvement using Dionex Guardcap H vial caps. The pH of the sample is lowered to a pH that more closely matches the initial conditions of the gradient, thus eliminating disturbances in the ion exchange equilibria.

Similarly, Figure 5 shows the baseline disturbance caused by a high pH matrix injected onto a Dionex IonPac AS23 column and the improvement using Dionex Guardcap H vial caps.

The capacity of a Dionex Guardcap H vial cap for neutralization is approximately 500 µL of 50 mM NaOH or other base.

Removal of anions and neutralization of acid

Dionex Guardcap AEX resin is provided in the borate 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. This method of use is analogous to using Thermo Scientific™ Dionex™ OnGuard™ II A cartridges for offline sample preparation removal of anions and neutralization of acids. Figure 6 shows the effect on peak efficiency of a low pH matrix injected onto a Dionex IonPac CS17 column and the improvement using Dionex Guardcap AEX vial caps.

Column: Dionex IonPac AG17-C (2 × 50 mm) / Dionex IonPac AS17-C (2 × 250 mm)
 Eluent: 1 mM KOH from 0 to 3 minutes, 1–35 mM KOH from 3 to 10 minutes, 35 mM KOH from 10 to 12 minutes
 Eluent Source: Dionex EGC 500 KOH Cartridge
 Flow Rate: 0.25 mL/min
 Inj. Volume: 2.5 µL
 Sampling Volume: 500 µL
 Detection: Suppressed Conductivity, Dionex AERS 500 Suppressor, AutoSuppression, Recycle mode

Peaks:
 1. Fluoride 20 ppb
 2. Acetate 1 ppm
 3. Formate 1 ppm
 4. Chloride 30 ppb
 5. Nitrate 1 ppm
 6. Carbonate ---
 7. Sulfate 1.5 ppm
 8. Oxalate 100 ppb
 9. Phosphate 1.5 ppm

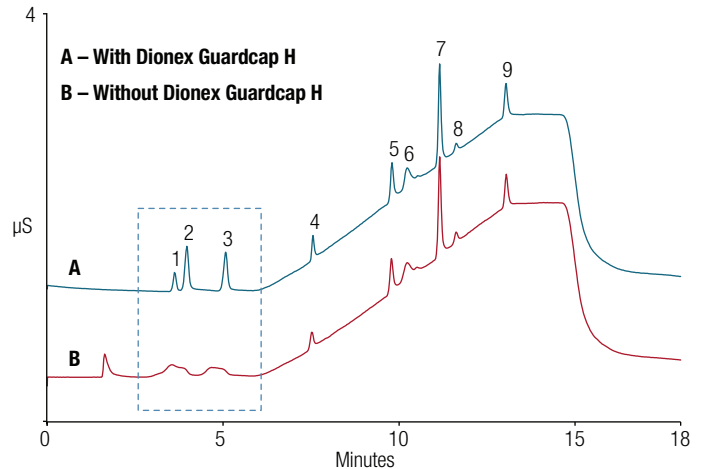


Figure 4. Neutralization of base to improve peak efficiency using Dionex Guardcap H vial caps

Column: Dionex IonPac AG23 (4 × 50 mm) / Dionex IonPac AS23 (4 × 250 mm)
 Eluent: 4.5 mM Na₂CO₃ / 0.8 mM NaHCO₃
 Eluent Source: Dionex AS23 eluent concentrate
 Flow Rate: 1.0 mL/min
 Inj. Volume: 10 µL
 Sampling Volume: 500 µL
 Detection: Suppressed Conductivity, Dionex AERS 500 Carbonate Suppressor, AutoSuppression, Recycle mode

Peaks:
 1. Fluoride
 2. Acetate
 3. Formate
 4. Chloride
 5. Nitrate
 6. Bromide
 7. Nitrate
 8. Phosphate
 9. Sulfate

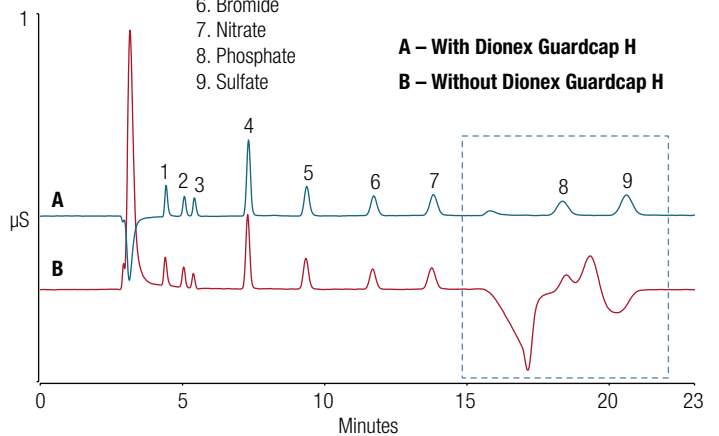


Figure 5. Neutralization of a 50 mM NaOH matrix to improve peak efficiency using Dionex Guardcap H vial caps

The capacity of a Dionex Guardcap AEX vial cap for neutralization is approximately 250 μL of 30 mM MSA, HCl, or other monovalent acid.

Column: Dionex IonPac CG17 (2 x 50 mm) /
Dionex IonPac CS17 (2 x 250 mm)
Eluent: 0.5-0.8 mM MSA from 0 to 25 min,
9 mM MSA from 25 to 35 min
Eluent Source: Dionex EGC III MSA
Flow Rate: 0.35 mL/min
Temperature: 35°C
Inj. Volume: 5 μL
Sampling Volume: 175 μL
Detection: Suppressed Conductivity,
Dionex CERS 500 Suppressor (2 mm),
AutoSuppression, Recycle mode

Peaks:	mg/L
1. Lithium	0.1
2. Sodium	0.4
3. Ammonium	0.5
4. Ethanolamine	1.0
5. Potassium	1.0
6. Diethanolamine	1.0
7. Triethanolamine	5.0
8. Magnesium	0.5
9. Calcium	1.0

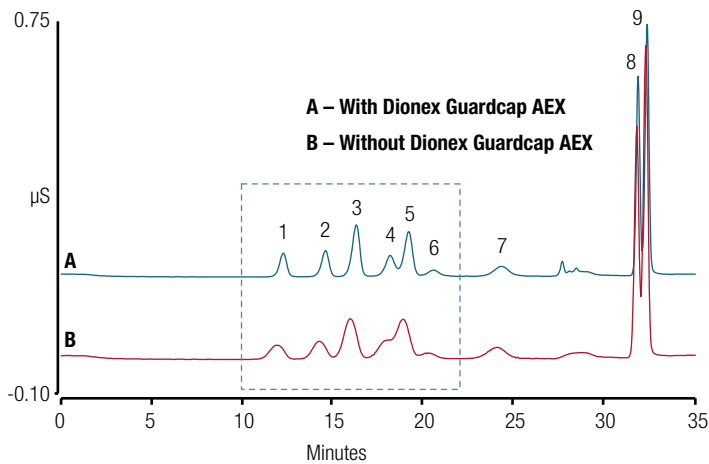


Figure 6. Neutralization of an acidic sample to improve peak efficiency and recovery of cations using Dionex Guardcap AEX vial caps

Removal of surfactants and other hydrophobic organic compounds

Dionex Guardcap HRP vial caps contain hydrophilic reversed phase resin and remove hydrophobic species by non-specific adsorption. This method of use is analogous to using Thermo Scientific™ Dionex™ OnGuard™ II RP cartridges for offline sample preparation removal of aromatic dyes, hydrocarbons, non-ionic surfactants,

and protonated carboxylic acids. Figure 7 shows the determination of fluoride in a synthetic wastewater sample containing 0.005% Triton™ x-405 using Dionex Guardcap HRP vial caps for sample preparation. Optimum matrix removal using Dionex Guardcap HRP vial caps is achieved with a reduced sampling flow rate of 0.25–0.5 mL/min. Each Dionex Guardcap HRP vial cap has enough capacity to remove up to 0.01% non-ionic surfactant (e.g., Triton™ x-405) from 500 μL of sample.

Column: Dionex IonPac AS20 (2 x 250 mm)
Eluent: 5 mM KOH
Eluent Source: Dionex EGC 500 KOH Cartridge
Flow Rate: 0.35 mL/min
Inj. Volume: 5 μL
Sampling Volume: 400 μL
Delivery Speed: 0.5 mL/min
Detection: Suppressed Conductivity,
Dionex AERS 500 Suppressor,
AutoSuppression, Recycle mode

Peak: 1. Fluoride 2 ppb

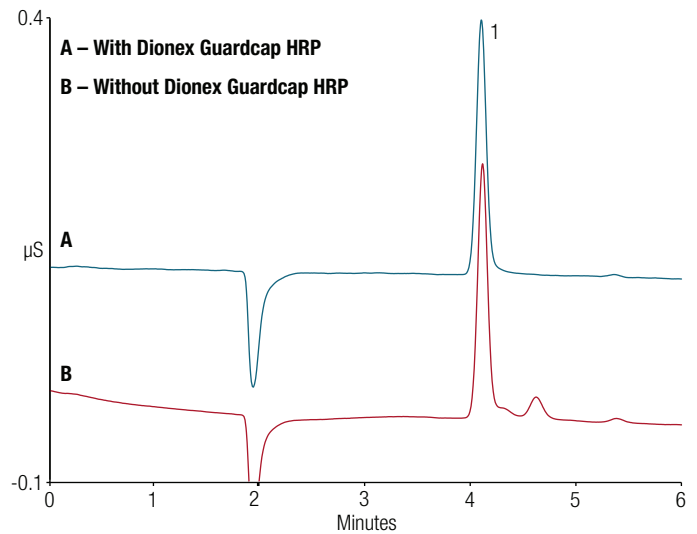


Figure 7. Determination of fluoride in Triton x-405 using Dionex Guardcap HRP vial caps

Filtering of particulates

All Dionex Guardcap vial caps will remove more than 80% of particulate matter greater than 20 μm in diameter.

Use considerations

Sample delivery volume vs. injection volume

The Dionex AS-DV autosampler pushes sample through the vial cap and into the sample loop on the injection valve. This means that the Dionex Guardcap vial cap is exposed to the entire sampling volume, not only the volume of the injection loop. The sample delivery volume must be

sufficient to flush previous contents of the sample transfer line to waste in order to minimize carryover between samples. A 10x volume is the usual recommended flush volume. Reduced-volume sample transfer lines have been developed for use with Dionex Guardcap vial caps in the Dionex AS-DV autosampler in order to minimize the necessary flush volume and, therefore, the sample delivery

volume. These reduced volume sample transfer lines maximize the ion exchange capacity of Dionex Guardcap vial caps and can be used interchangeably with samples that are capped with standard Dionex AS-DV filter caps or plain caps. Table 3 shows the sample transfer line volume and sampling volume for two different lengths of sample transfer lines.

Table 3. Minimum sampling volumes.

Injection valve mounting location	Recommended sample transfer line length / volume	Minimum recommended sampling volume (10x)
Dionex AS-DV autosampler	35 cm / 17.5 µL	175 µL
Dionex IC systems	70 cm / 35 µL	350 µL

Dionex Guardcap Vial Caps Specifications					
Product	Functionality	Capacity µeq/cap	Solvent compatibility	pH Stability	Mode of use
Dionex Guardcap H	Cation-exchange Hydronium form	50	0–20% methanol	0–14	Removal of alkaline earth and transition metals; pH adjustment of basic samples
Dionex Guardcap Na	Cation-exchange Sodium form	50	0–20% methanol	0–14	Removal of alkaline earth and transition metals
Dionex Guardcap HRP	Hydrophilic reversed phase	20 mg benzoic acid at pH <4	0–20% methanol	0–14	Removal of hydrophobic organics and surfactants
Dionex Guardcap AEX	Anion-exchange Borate form	50	0–20% methanol	0–14	Removal of anions; pH adjustment of acidic samples

Ordering information

To order in the U.S., visit thermofisher.com, call (800) 346-6390 or contact the Thermo Fisher Scientific office nearest you. Outside the U.S., order through your local Thermo Fisher Scientific office or distributor. Refer to the following part numbers.

Description	Part Number
Dionex Guardcap H vial caps, 5 mL, Introductory Package of 12 Includes a 70 cm reduced volume sample transfer line assembly	302765
Dionex Guardcap Na vial caps, 5 mL, Introductory Package of 12 Includes a 70 cm reduced volume sample transfer line assembly	302796
Dionex Guardcap HRP vial caps, 5 mL, Introductory Package of 12 Includes a 70 cm reduced volume sample transfer line assembly	302798
Dionex Guardcap AEX vial caps, 5 mL, Introductory Package of 12 Includes a 70 cm reduced volume sample transfer line assembly	302981
Dionex Guardcap H vial caps, 5 mL, Package of 125	302504
Dionex Guardcap Na vial caps, 5 mL, Package of 125	302797
Dionex Guardcap HRP vial caps, 5 mL, Package of 125	302799
Dionex Guardcap AEX vial caps, 5 mL, Package of 125	302982
35 cm reduced volume sample transfer line assembly Recommended for use with injection valves mounted in Dionex AS-DV autosamplers	22120-60051
70 cm reduced volume sample transfer line assembly Recommended for use with injection valves mounted in IC systems	22120-60055
Dionex PolyVials, 5 mL, Package of 250	038008

Find out more at www.thermofisher.com/Guardcap