



Dionex Guardcap™ H

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Product Manual

for

Dionex Guardcap H

(Package of 125, Item # 302504)

(Package of 12 with sample transfer line, Item # 302765)

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Revision 02, March, 2017, Added the item number for 12 pack (# 302765).

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

1.1 Overview of Automatic Sample Preparation using Guardcap™ H and the Dionex AS-DV Autosampler

Thermo Scientific™ Dionex™ Guardcap™ H vial caps are designed to work with 5-mL Thermo Scientific Dionex PolyVials™ in Thermo Scientific Dionex AS-DV autosamplers to achieve inline removal of cations and neutralization of bases.

The Dionex AS-DV holds 50 vials that can be sampled in any order and multiple samples can be taken from each vial. A full description of operating features and instructions for use can be found in the Dionex AS-DV Autosampler Operator's Manual, Document No. 065259. The unique PolyVial sample vial incorporates a cap that acts as a simple piston to force sample out of the vial. This eliminates the need for an external sampling pump. This vial/cap system can be used for in-line sample preparation removal of cations, neutralization, and filtration by using Guardcap H vial caps.

Guardcap H vial caps fit 5-mL PolyVials and contain H⁺-form cation exchange resin in a high density polyethylene filtration matrix. The Guardcap H filters are brown in color, as compared to the white filter cap filters. Note that if filtration-only is needed, the standard filter cap contains a 20 µm filter to remove solid materials from the sample before injection. By default, the Dionex AS-DV comes with a sample transfer line that is 142 cm long. To maximize the ion exchange capacity in Guardcap H, the Dionex AS-DV autosampler should be configured with an onboard 6-port high pressure injection valve (Item # 068920) and 35 cm sample transfer line (Item # 22120-60051) in order to minimize sample volumes required for adequate flushing between samples. If an onboard injection valve is not available, direct connection to injection valves mounted in IC systems should be made with a 70 cm sample transfer line (Item # 22120-60055).

1.2 Deionized Water Requirements for Ion Chromatography and Guardcap H /AS-DV Methods

For all procedures requiring deionized water, use ASTM Type 1 (18 megohm-cm) filtered and deionized water that meets the following specifications.

Table 1 Specifications for ASTM Type I water (with addition of a limit on metals)

Resistivity	> 18.0 megohm-cm
Organics-TOC	<10 ppb
Iron-Transition metals	<1 ppb
Pyrogens	<0.03 Eu/mL
Particulates >0.2 µm	< 1 units/mL
Colloids-silica	<10 ppb
Bacteria	1 cfu/mL

1.3 Setup



NOTE

The standard sample transfer line in the AS-DV autosampler should be replaced with a shorter, reduced volume line in order to make optimum use of the ion exchange capacity of the Guardcap H caps. Please see the Setup of AS-DV and Appendix A for instructions to change the sample transfer line.

1.3.1 Vials and Caps

Guardcap H vial caps are available for 5-mL PolyVials. Use of an insertion tool (Item # 037987), included in the Dionex AS-DV Ship Kit (Item # 068887), simplifies insertion of the caps. Section 3, Reordering Information, contains part numbers for PolyVials, Guardcap vial caps, and other accessories.

The Guardcap H filters are brown in color, as compared to the white filter cap filters. The brown color appears darker for wet Guardcap H caps than for dry Guardcap H caps. Color does not affect performance. The Guardcap H are shipped slightly damp but perform equally well from a dry state. The Guardcap H packaging is an opaque, resealable bag and it is recommended to re-seal the bag when not in use to avoid airborne contamination such as from amines.

1.3.2 Setup of AS-DV

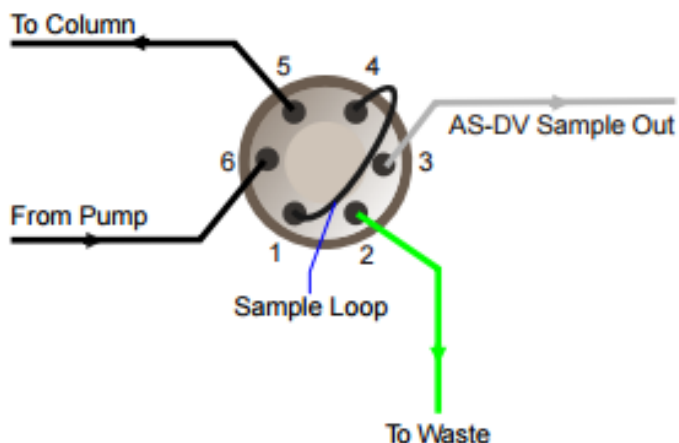
1.3.2.1 Injection valve installed in AS-DV

For best performance and the highest use of the ion exchange capacity of Guardcap H, the AS-DV autosampler should be setup with the on-board injection valve and a reduced volume sample transfer line assembly (Item # 22120-60051). Other configurations with direct connection to injection valves located in IC instruments are discussed in section 1.3.2.2. The standard AS-DV sample line will reduce the capacity of the Guardcap by up to 80%. Large flush volumes and extra tubing volume will exhaust the capacity, depending on the ionic strength on the sample matrix.

Guardcap H can be used at flow rates up to 3 mL/min but optimum performance is achieved at 1 mL/min. The following instructions describe setup of the hardware and method parameters for accomplishing sample preparation using Guardcap H.

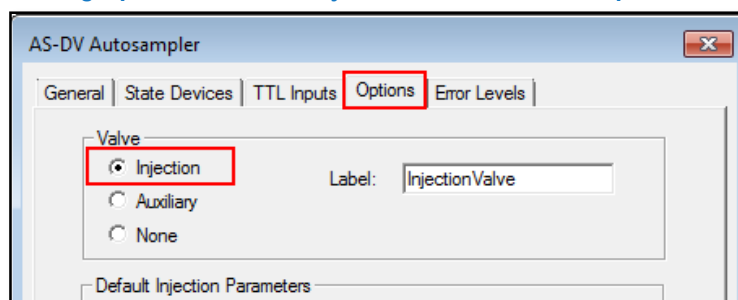
1. Install 6-port injection valve in AS-DV autosampler (see Figure 1). Note that the valve installs upside down.

Figure 1 AS-DV 6-port Injection Valve Plumbing



- a. Install a new, reduced volume AS-DV sample transfer line assembly (Item # 22120-60051). This is a narrower Teflon line shortened to 35 cm. See Appendix A or the AS-DV Operators manual for detailed installation instructions.
2. Add an AS-DV to the Chromeleon Instrument Configuration. Confirm the onboard valve is identified as an Injection Valve in the AS-DV options (Figure 2):

Figure 2 Confirming Optional Onboard Injection Valve in AS-DV Options



3. The sample delivery volume in the AS-DV will be controlled by the Chromeleon Instrument Method/Program using the command line below (Figure 3). The injection volume in the Sequence is not used. The actual injection volume is the volume of the sample loop installed on the injection valve.

Figure 3 Setting Sample Delivery Volume to 175 µL in the Instrument Method

Command	Value
Sampler.FlushFactor	1
Sampler.DeliverSpeed	1.0 [ml/min]
Pump_ECD.Flow	1 [ml/min]
Sampler.LoadPosition	
Sampler.DeliverSample	Volume=175 [µl]
Sampler.EndSamplePrep	

4. The loading flow rate (Delivery Speed) can be specified either in the Chromeleon Instrument Method Wizard (Figure 4) or directly in the Instrument Method/Program (Figure 5).
 - a. Set Loading Mode = **Concentrator Mode**
 - b. Set Delivery Speed = **1.0 mL/min**

Figure 4 Confirming Delivery Speed in Instrument Method Wizard

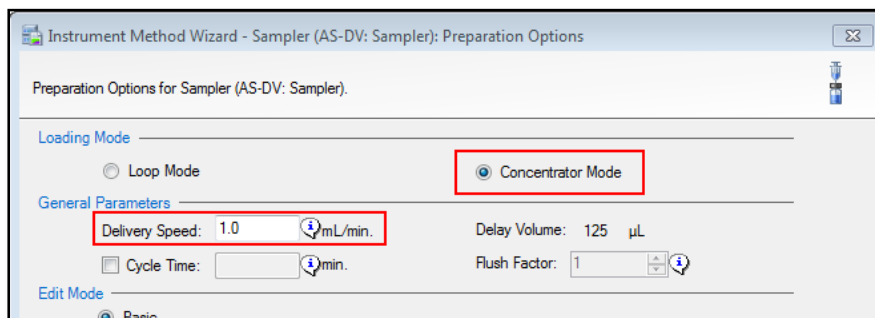


Figure 5 Confirming Delivery Speed in the Instrument Method/Program

Command	Value
Sampler.FlushFactor	1
Sampler.DeliverSpeed	1.0 [ml/min]
Pump_ECD.Flow	1 [ml/min]
Sampler.LoadPosition	
Sampler.DeliverSample	Volume=175 [µl]
Sampler.EndSamplePrep	

This configuration allows about 10x flushing per sample while maximizing the ion exchange capacity available in Guardcap H.

The actual injection loop volumes can be in the range of 2–25 µL for 2-mm or 4-mm operation.

1.3.2.2 Injection valve installed in IC system

1. Install a new, reduced volume AS-DV sample transfer line assembly (Item # 22120-60055). This is a narrower Teflon line shortened to 70 cm. Note: the AS-DV must be placed very close to the IC in order to use this sample line.
2. Setup is the same as steps 3 and 4 of section 1.3.2.1 except the sample delivery volume is defined as 350 µL in the Chromeleon Instrument Method/Program (Figure 6).

Figure 6 Setting Sample Delivery Volume to 350 μL in the Instrument Method

Command	Value
Sampler.FlushFactor	1
Sampler.DeliverSpeed	1.0 [ml/min]
Pump_ECD.Flow	1 [ml/min]
Sampler.LoadPosition	
Sampler.DeliverSample	Volume=350 [μL]
Sampler.EndSamplePrep	

1.3.3 Preparing Sample Vials and Guardcap H caps

Thermo Scientific recommends thoroughly rinsing all PolyVials and caps with deionized water before filling to remove any traces of dust. Thermo Scientific does not recommend reusing vials or caps and does not guarantee their cleanliness or proper operation if reused.

To prevent the introduction of contaminants, extreme care must be used in cleaning. Use only ASTM Type I (18 megohm-cm) filtered and deionized water that meets the specifications listed in Table 1 for the cleaning procedures.

Guardcap H vial caps are provided in a clean state (see Figure 7). However, for trace analysis it may be necessary to pre-rinse the caps. Guardcap H vial caps can be flushed with deionized water using the same procedure used for filter caps in section 3.1 of the AS-DV Operator's manual. Using a clean syringe, force 10-50 mL of deionized water through the Guardcap H from the top of the cap (Luer inlet). Be careful not to dislodge the Guardcap H filter. Rinsed Guardcap H caps can be stored in a clean polyethylene container, filled with deionized water and capped.

PolyVials should be rinsed 10 times with a jet of deionized water. For trace analysis, rinsed PolyVials should be allowed to soak in deionized water for at least 24 hours. When ready to use, empty the PolyVial and rinse two times with sample.

1.3.4 Filling the PolyVial and Installing the Guardcap H vial cap



NOTE

To prevent contamination when doing trace-level analysis, use forceps to handle the vials and caps. Also, avoid touching any surface that will be wetted with sample.

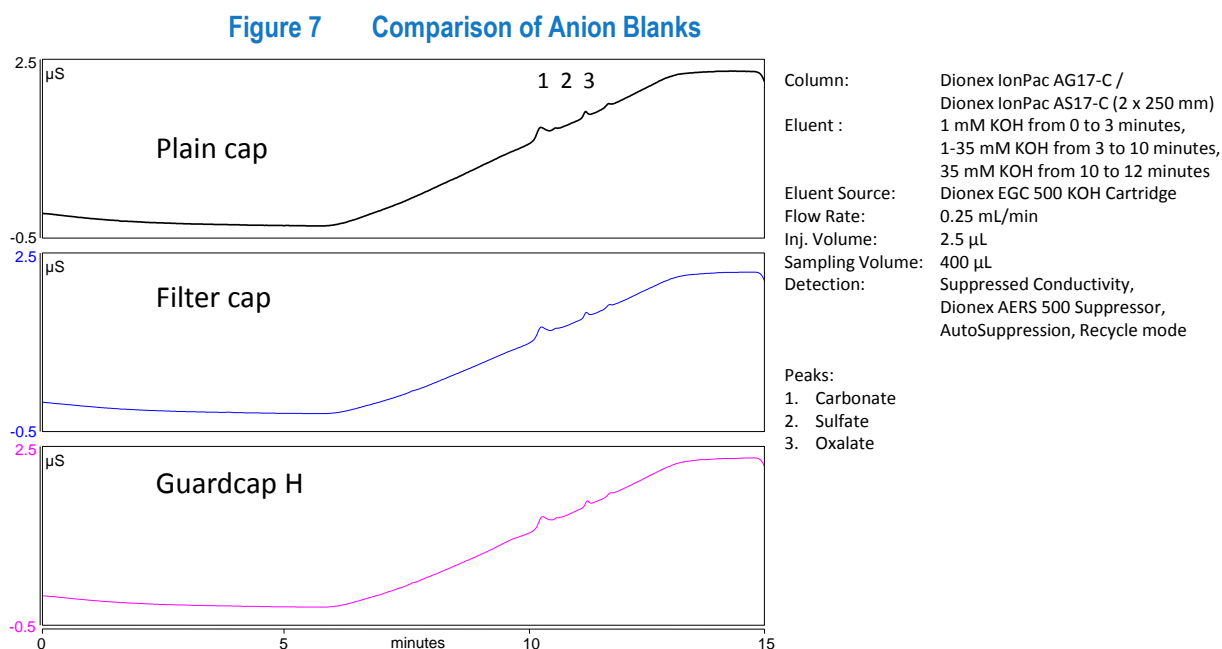
It is best to minimize the initial air bubble under the Guardcap H cap when installed in the 5-mL PolyVial. The procedure below assures that adequate sample is placed in the vial, but does not overflow the vial.

1. Weigh 6.1-6.4 g sample/vial and install the Guardcap H using the flat end of the installation tool so the cap is flush with the top of the vial. Note that this amount of sample is above the top of the vial tray. The top of the Guardcap H cap must be flush with the top of the PolyVial in order for the vial to be properly sensed by the autosampler. The backpressure applied by the user to install the Guardcap H is higher

than when installing a filter or plain cap. This is normal since Guardcap H contains ion exchange resin in the pores of the filter.

2. After installation of the Guardcap H, invert the vial to confirm that no air bubble is freely floating under the cap.
3. Before loading the vial into the carousel, shake off any liquid that has been forced into the cap socket. Do not use laboratory wipes to blot liquid from the cap sockets as wipes can leave fibers that can clog the liquid flow path and cause backpressure.

Figure 7 shows carbonate, sulfate, and oxalate from a system blank. All peaks are less than 50 ppb. The chromatograms in the figure show a comparison using a plain cap, filter cap, and Guardcap H.



1.4 In-line Sample Preparation using Guardcap H caps with PolyVials in the AS-DV Autosampler

Guardcap H caps contain strong cation exchange resin in the hydronium ion form. One Guardcap H can neutralize up to 500 µL of 50 mM sodium hydroxide or remove 1000 mg/L divalent cation, i.e. calcium or transition metals, from 800 µL of sample. The following application examples illustrate several types of methods for sample preparation.

1.4.1 Removal of Metals and Other Cations

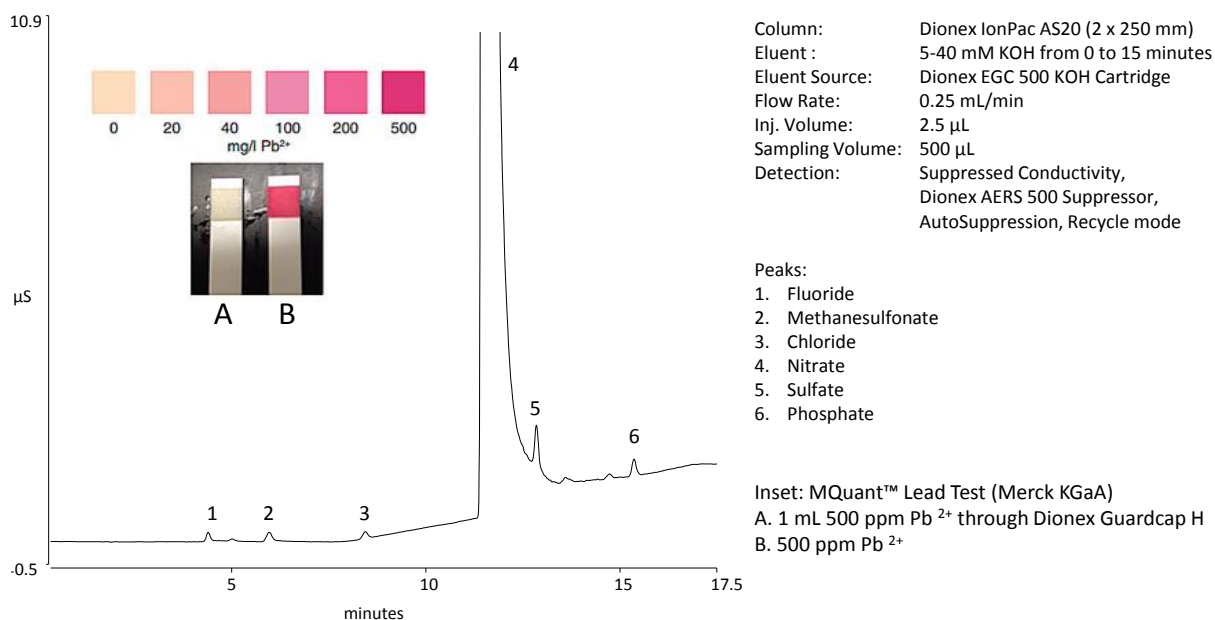
The Guardcap H resin is provided in the H⁺-form, meaning that alkali, alkaline earth, and transition metals in the cation form, and protonated weak bases including amines are exchanged for hydronium ion. One Guardcap H cap contains about 50 µEq of cation exchange capacity, available for removing cations from samples. As an example, one Guardcap H cap can remove 1000 ppm calcium from 800 µL of sample, or 500 ppm of a divalent cation from 1.6 mL of sample. It is important to note that the necessary capacity needed for an application must be

calculated based on the sample volume passing through the Guardcap H rather than the volume of the injection loop.

The sample can become acidic in this mode as cations are exchanged for the hydronium ion. This application is useful to remove cations that can foul the anion suppressor, which is a cation exchanger, or cations that can build up by precipitation as insoluble salts on system components. This method of use is exactly analogous to using Dionex OnGuard™ II H Cartridges for offline sample preparation removal of metals and other higher valency cations.

Figure 8 below shows the determination of methanesulfonate in a lead nitrate matrix using Guardcap H sample preparation. The inset in Figure 8 shows the results of passing 1 mL of a 500 ppm Pb²⁺ standard through a Guardcap H vial cap. Guardcap H effectively removes all of the Pb²⁺ from the standard.

Figure 8 Determination of Methanesulfonate in a Lead Nitrate Matrix Using Guardcap H Sample Preparation



1.4.2 Neutralization of Base

The 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 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 gradient anion system.

Figure 9 below shows the band broadening effects of a high pH matrix injected onto a Dionex™ IonPac™ AS17-C column and the improvement using Guardcap H. The pH of the sample is lowered to a pH that more closely approximates the initial conditions of the gradient, thus eliminating disturbances in the ion exchange equilibria. This is also useful for large loop injections, which for 2-mm systems, are injections volumes in the range of 25 µL. When using standard loop injection, the most common effect of high hydroxide concentration in the sample is peak broadening of early-eluting peaks, most commonly fluoride.

Figure 9 Neutralization of Base to Improve Peak Efficiency

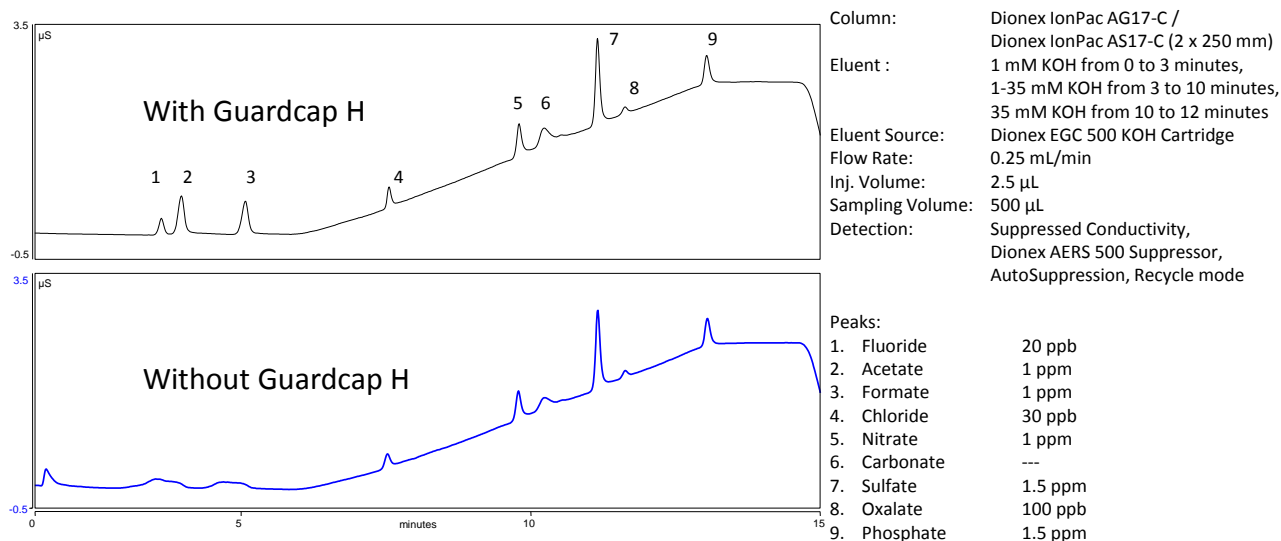
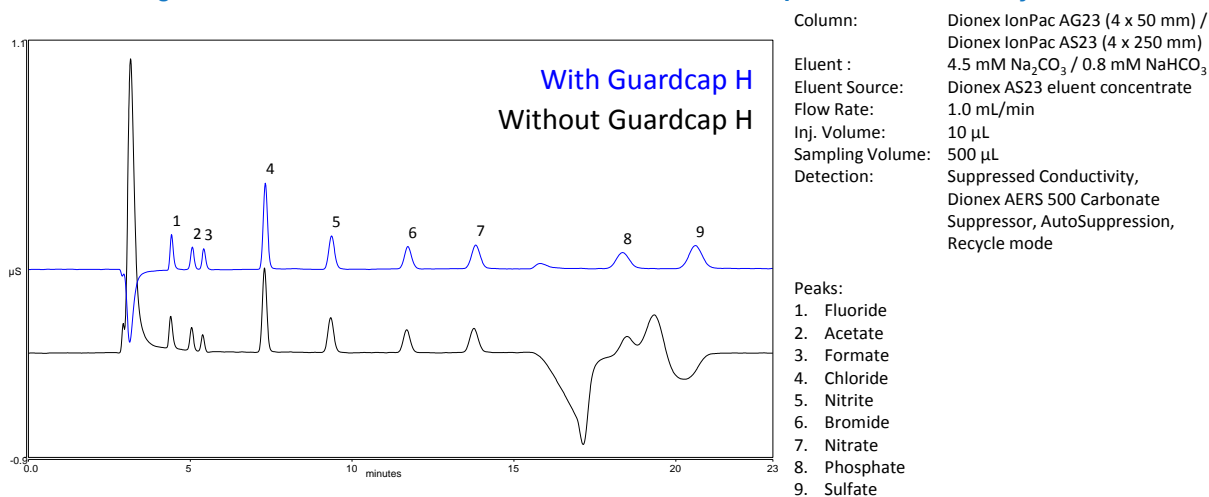


Figure 10 below shows the baseline disturbance caused by a high pH matrix injected onto a Dionex™ IonPac™ AS23 column and the improvement using Guardcap H.

Figure 10 Neutralization of a 50 mM NaOH Matrix to Improve Peak Efficiency



The capacity of Guardcap H for neutralization using the AS-DV flow rate of 1 mL/min is about 500 µL of 50 mM NaOH. If the concentration of base in the sample is higher than about 50 mM, the sample should be diluted prior to treatment using Guardcap H.

1.5 Filtering of Particulates

Similar to filter caps, Guardcap H will remove better than 80% of particulate matter greater than 20 µm in particle diameter.

2. Troubleshooting

Symptom	Possible Issue	Action
AS-DV chatters, grinds, or stalls during sample loading	Loading flow rate (Delivery Speed) is too high.	Reduce loading flow rate to 1 mL/min.
	There is a clog in the sample loading path.	Work through each connection to identify the source of the clog. Replace lines or components as necessary, taking care not to overtighten fittings.
		Clean/replace injection valve components (rotor and stator face assembly).
Carryover	Insufficient sample volume to adequately flush lines between samples.	Ensure that a reduced volume sample transfer line was installed and that the instrument method/program specifies the appropriate sample delivery volume (see Section 1.3.2.1 or 1.3.2.2).
		Increase sample delivery volume by 100 μ L in the instrument method/program.
High blank	System may be contaminated or Guardcap H may be beyond Best If Used By date.	Flush the AS-DV with 5 mL deionized water using a clean PolyVial and plain vial cap.
		Pre-rinse Guardcap H vial caps with clean deionized water as noted in Section 1.3.3.
		Check box label for Best If Used By date.
	Shared AS-DV used for both anions and cations can become contaminated with insoluble salt deposits that contribute to background peaks.	Replace sample loop, sample transfer line, and connecting PEEK tubing from the injection valve to the column.
	Large carbonate peak in blank.	Use a Carbonate Removal Device (CRD) and/or degas samples and deionized water in eluent reservoir.

3. Reordering Information

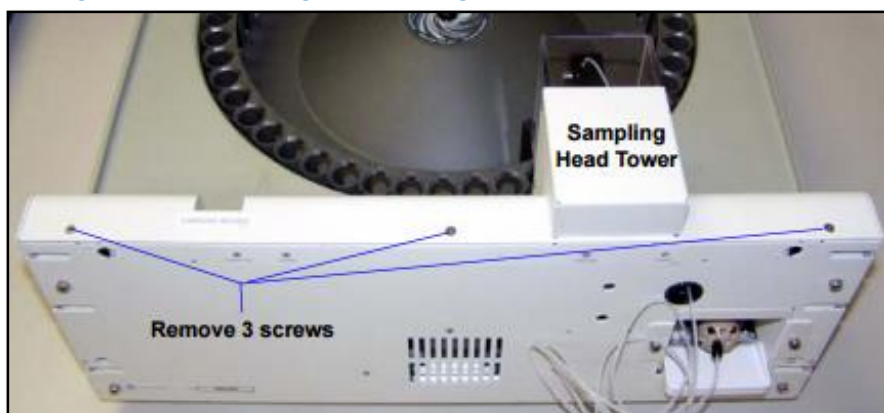
Part Number	Description	Quantity
038008	PolyVials, 5 mL	250
302504	Guardcap H vial caps, 5 mL	125
302765	Guardcap H vial caps, 5 mL <i>Includes a 70 cm sample transfer line</i>	12
037987	Tool, vial cap insertion	1
068920	6-port High Pressure Valve Kit	1
068947	Tray for 5 mL vials	1
22120-60051	35 cm Sample Transfer Line Assembly <i>For use with injection valves mounted in AS-DV</i>	1
22120-60055	70 cm Sample Transfer Line Assembly <i>For use with injection valves mounted in an IC system</i>	1

Appendix A – Installing the Sample Transfer Line

The following tools are needed to complete this procedure: #1 Phillips screwdriver and 3-mm Allen wrench.

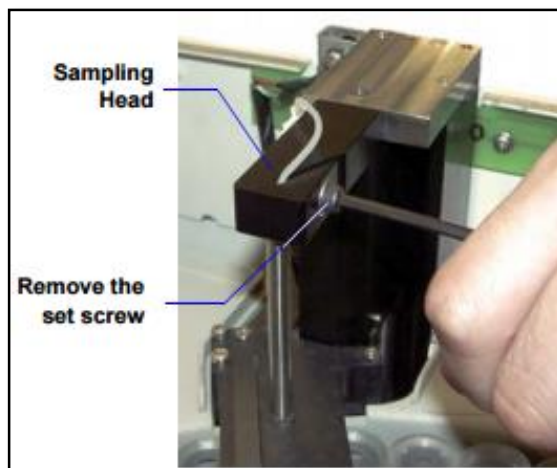
1. Disconnect the sample out line from the injection valve. Remove the fitting.
2. Press the Carousel Release button to retract the sampling tip to its fully up position.
3. Turn off the Dionex AS-DV power and disconnect the power cord. Raise the top cover.
4. Remove the Dionex AS-DV front cover by gripping the indentations at each side and pulling the cover straight off toward you.
5. Use a #1 Phillips screwdriver to remove the three flathead screws from the sampling head tower cover (see Figure 11). Lift up the cover to remove it.

Figure 11 Removing the Sampling Head Tower Cover



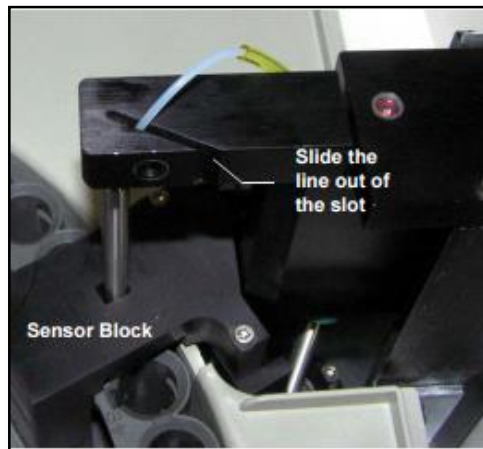
6. Use a 3-mm Allen wrench to remove the set screw on the sampling head (Figure 12).

Figure 12. Removing the Set Screw



7. Pull slightly on the sample line to gain a small amount of slack in the line. Then, slide the tubing out of the slot in the sampling head (Figure 13).

Figure 13 Sliding the Sample Transfer Line out of the Sampling Head Slot



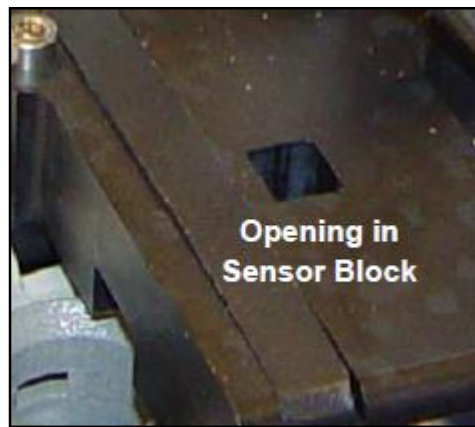
8. Push down on the sampling head to move the needle into the carousel a small amount. Twist the needle to allow the sampling tip to exit through the opening in the sensor block.
9. Unscrew the sampling tip from the needle and pull out and discard the tip and tubing.
10. Locate the new sample transfer line assembly. This assembly consists of a length of tubing with a new sampling tip installed on one end.
11. Thread the free end of the tubing up through the needle and screw the tip into place on needle (Figure 14).

Figure 14 Sampling Tip



12. Continue threading the free end of the tubing up through the sampling head and out the aperture on the front of the Dionex AS-DV.
13. Slide the needle into place and slide the sampling needle tubing back into the slot in the sampling head.
14. Align the flat sides of the sampling tip so that they fit through the opening in the sensor block (Figure 15).

Figure 15 Sensor Block Opening



15. Make sure that the needle is pushed as far as it will go up into the sampling head and then tighten the set screw firmly.

NOTE: The set screw must be very tight to secure the sampling needle. This will require a firm turn of the set screw. Test the line to make sure it is tightly held.

16. Reinstall the sampling head tower cover.
17. Reinstall the front cover and close the top cover.
18. Reconnect the sample out line to the valve.
19. Reconnect the power cord and turn on the power.