Errata

Product Manual for the Dionx IonPac™ Cation Polisher
061777-01

For new orders of the following parts discussed in this manual, please use the updated part numbers listed below.

Part	Old Part Number in this manual	Updated Part Number to use for new orders
ASSY,COIL,2ML/MIN,1000PSI,EG40	*053763*	*AAA-053763*
ASSY,ICS3/5 AS,-IV,+SP,-ST	*061777*	*AAA-061777*

Product Manual

For

IonPac® Cation Polisher

IonPac CP1 Na^+ Form Cation Polisher Column 6 x 16 mm (P/N 064930)

IonPac CP2 H^+ Form Cation Polisher Column 9 x 24 mm (P/N 064931)

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1. Introduction

The CP Cation Polisher columns are designed for removal of metallic contaminants and other multivalent cations such as calcium and magnesium from the sample stream when pursuing anion analysis. The CP Cation Polisher columns improve the performance of Ion Chromatography Systems particularly when pursuing phosphate analysis in the presence of metallic contaminants. Samples that contain high levels of metals or cations can be deposited on the guard/analytical columns or the suppressor which can lead to performance issues such as poor peak shapes or poor recoveries for some analyte ions. The contaminants can adversely affect the performance of the guard/analytical columns or suppressor. The CP Cation Polisher columns address the potential precipitation issue of cation contaminants and aid in extending the column and suppressor lifetimes when pursuing anion analysis and when the matrix ions contain multivalent cations.

In some cases the CP Cation Polisher columns aid removal of matrix cations when pursuing anion analysis with sample preconcentration. The matrix cations can elute species of interest from the concentrator column and lead to poor peak shapes and recovery particularly for early eluting anions. By removing the matrix cations using the CP Cation Polisher column good chromatography performance can be restored.

The CP1 Na^+ Form (6x16 mm) Cation Polisher column is packed with 20 µm styrene/divinylbenzene copolymer that is grafted to yield a resin with carboxylate functional groups. The resin was converted to sodium form before packing and is shipped in the sodium form. Due to its highly crosslinked structure, the resin is solvent compatible. The capacity of the column is 170 µeq/column with a void volume of approximately 250 µL. The physical rigidity of this resin allows the column to be used up to 3,000 psi (20.68 MPa). The CP1 Na^+ Form Cation Polisher column can be readily regenerated to the sodium form using sodium containing regenerants such as sodium hydroxide. The CP1 Na^+ Form Cation Polisher column is packed in a (6 x 16 mm) low pressure format to accommodate operation with an autosampler (AS40 or AS). The CP1 Na^+ Form Polisher Cation column is recommended for small sample volume applications.

NOTE	**When pursuing preconcentration applications with the CP1 Na^+ Form Cation Polisher column, for samples that contain high levels of weak acids conversion to the sodium form may result in the formation of highly dissociated species which may act like an eluent. The effect will result in poor recovery and/or peak shapes particularly for early eluting analytes. For example a borated water sample containing high levels of boric acid may be converted to the sodium borate form which being fully dissociated can elute retained anions off the concentrator column. In this case the CP2 H^+ Form Cation Polisher column is recommended.**

The CP2 H^+ Form (9 x 24 mm) Cation Polisher column is packed with 20 µm styrene/divinylbenzene copolymer that is grafted to yield a resin with carboxylate functional groups. The resin was converted to hydronium form before packing and is shipped in the hydronium form. Due to its highly crosslinked structure, the resin is solvent compatible. The capacity of the column is 700 µeq/column with a void volume of approximately 825 µL. The physical rigidity of this resin allows the column to be used up to 3,000 psi (20.68 MPa). The CP2 H^+ Form Cation Polisher column is recommended for large volume sample preconcentration using an external pump. Due to the large delay volume, the CP2 H^+ Form Cation Polisher column is not recommended for small sample volume applications.

The maximum recommended flow rate for the CP1 Na^+ Form Cation Polisher column and CP2 H^+ Form Cation Polisher columns is 5 mL/min. The back pressure generated by the CP1 Na^+ Form Cation Polisher column is less then 10 psi at 2 mL/min. The back pressure generated by the CP2 H^+ Form Cation Polisher column is less than 40 psi at 2 mL/min.

Table 1
IonPac CP1 and CP2 Column Specifications

Column	Particle Diameter	Substrate Crosslinking	Column Capacity	Functional Group	Hydrophobicity
CP1 Na^+ Form (6 x 16 mm)	20 µm	55%	170 µeq/col	Carboxylic Acid	Very low
CP2 H^+ Form (9 x 24 mm)	20 µm	55%	700 µeq/col	Carboxylic Acid	Very low

2. Installation and Operation

2.1. Hardware Configuration

2.1.1 CP1 Na^+ Form Cation Polisher Column Configuration

1. 10-32 Ferrule Plug (P/N 042772)
2. Column Body 6 x 16 mm (P/N 063561)
3. Bed Support (Frit) (P/N 063606)
4. Bed Support Assembly (P/N 063688)
5. 9-mm Column End Fitting (P/N 048298)

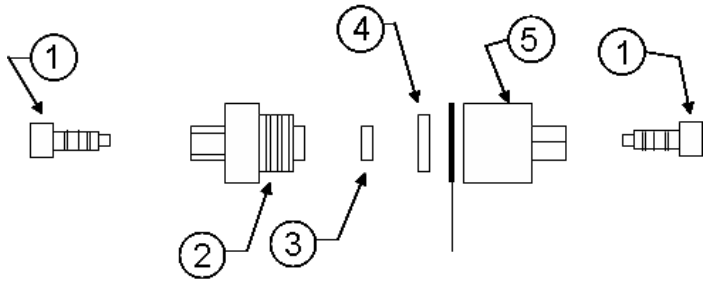

Figure 1
6 x 16 mm CP1 Na^+ Form Cation Polisher Column Hardware

2.1.2. CP2 H^+ Form Cation Polisher Column Configuration

1. 9-mm Column End Fitting (P/N 045287)
2. Bed Support Assembly (P/N 060270)
3. Column Body 9 x 24 mm (P/N 0390391)

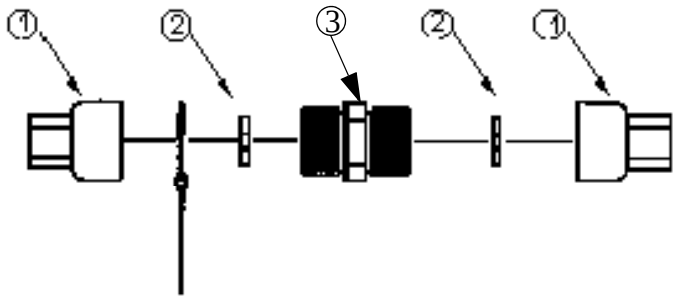

Figure 2
9 x 24 mm CP2 H^+ Form Cation Polisher Column Hardware

2.2. Installation and Operation

2.2.1 Sample Pretreatment with CP1 Na^+ Form Cation Polisher Column

The CP1 Na^+ Form Cation Polisher column was designed to operate with an autosampler. Connect the sample line from the autosampler to the CP1 Na^+ Form Cation Polisher column. Connect a line from the CP1 Na^+ Form Cation Polisher column to the injection valve to load the injection loop. Typically port 5 is used for loading the sample loop which is located between ports 1 and 4. The line from port 6 is diverted to waste. Ports 2 and 4 are used for the eluent in line from the pump and the eluent out line to the column respectively.

In operation, the sample stream is pushed through the CP1 Na^+ Form Cation Polisher column and the sample cations are exchanged for sodium. The sodium form of the sample anions are then routed to an injection loop for further analysis. The CP1 Na^+ Form Cation Polisher column will need to be regenerated to the sodium form when the capacity is depleted. A bi-weekly regeneration protocol is recommended when the number of samples to be analyzed is high. Refer to section 3.1. for regenerating the CP1 Na^+ Form Cation Polisher column.

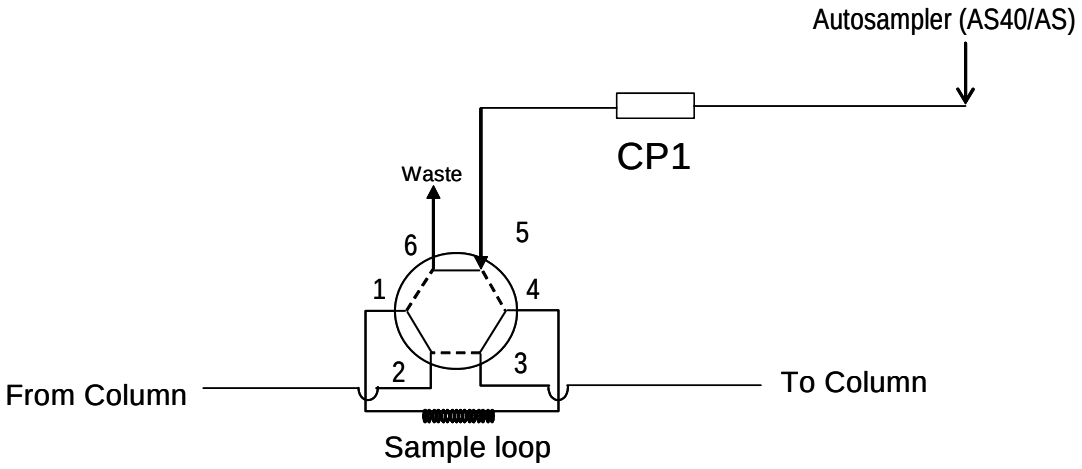

Figure 3
Plumbing Scheme for CP1 Na^+ Form Cation Polisher Column

AS40 autosampler: Use 5 mL vials (P/N 038141) for the sample. Ensure that the delay volume from the autosampler to the injection valve is minimal. Ensure that the sample volume in the vial (typically 1.5 mL to 5 mL) exceeds roughly 3X to 5X the delay volume of the CP Cation Polisher column and any added delay volume from the tubing connecting the autosampler to the injection valve. This will ensure that the sample stream flushes the injection line between injections. The delay volume of the CP1 Na^+ Form Cation Polisher column is 250 µL. For more information on the AS40 autosampler operation, refer to the AS40 Automated Sampler Operator's manual (Document No. 034970).

AS autosampler: Use 10 mL vials (P/N 055058) for the sample. Ensure that the delay volume from the autosampler to the injection valve is minimal. A calibration of the port volume as outlined in Section 5.9. of the AS Autosampler Operator's manual (Doc No. 065051) is recommended after the CP1 Na^+ Form Cation Polisher column is installed inline between the autosampler and the injection valve. Calibrating will ensure that the sample is delivered accurately to the sample injection loop and all delay volume contributions are accounted for.

An alternative method would be to state that the injection loop size is comprised of the loop volume and the delay volume of the CP Cation Polisher column. For example when pursuing a 25 µL injection, state that the loop size is (25 + 250 µL) 275 µL and input this value in the front panel of the AS and in the Chromeleon pgm or Chromeleon wizard. This will ensure that a full loop injection is maintained. This approach circumvents the need to calibrate the port volume in the autosampler.

If there are metals in the sample stream a cleanup for the CP1 Na^+ Form Cation Polisher column is recommended. It should be done on a bi-weekly basis with 500 mN sulfuric acid followed by 500 mM oxalic acid and DI water as outlined in section 4.4.

2.2.2. Sample Pretreatment with CP2 H^+ Form Cation Polisher Column

The CP2 H^+ Form Cation Polisher column is designed to remove cations from sample streams when pursuing anion analysis in conjunction with a concentrator column. An example is the analysis of borated waters in the nuclear power industry.

In most Pressurized Water Reactors (PWRs) the primary coolant water contains matrix components that are predominantly borate and lithium ions and analysis of trace anions such as fluoride, chloride and sulfate is usually pursued at trace levels. Depending on the level of borate ions the analysis is pursued either using EGC generated hydroxide eluents or with EGC generated borate eluents (refer to Application Note 166). Typically when the level of borate ion in the sample matrix is high, borate eluents are preferred.

When lithium is present in the above matrix, lithium borate, being fully dissociated, acts as an eluent eluting trace components off the concentrator column, leading to poor recovery for weakly held species such as fluoride. The CP2 H^+ Form Cation Polisher column was designed for the above application to remove lithium from the sample streams while converting the sample matrix ions (borate) to a weakly dissociated species (boric acid). Being weakly dissociated the boric acid does not act like an eluent and complete recovery is achieved with the CP2 H^+ Form Cation Polisher column installed. The advantage of using a carboxylated resin over a sulfonated resin for the CP2 H^+ Form Cation Polisher column is that there is no issue with sulfate leaching from the CP Cation Polisher column leading to improved quantification of sulfate.

An example set-up is shown in Figure 4 using an ICS-3000 two channel system. One channel in the ICS-3000 is used for sample preparation while the other channel is used for analysis. The pump in channel 1 is used for pumping a DI water stream or sample carrier stream. A CR-ATC anion trap column is used to remove any anionic contaminants in the DI water stream. An ATC-HC anion trap column can be used in place of the CR-ATC anion trap column.

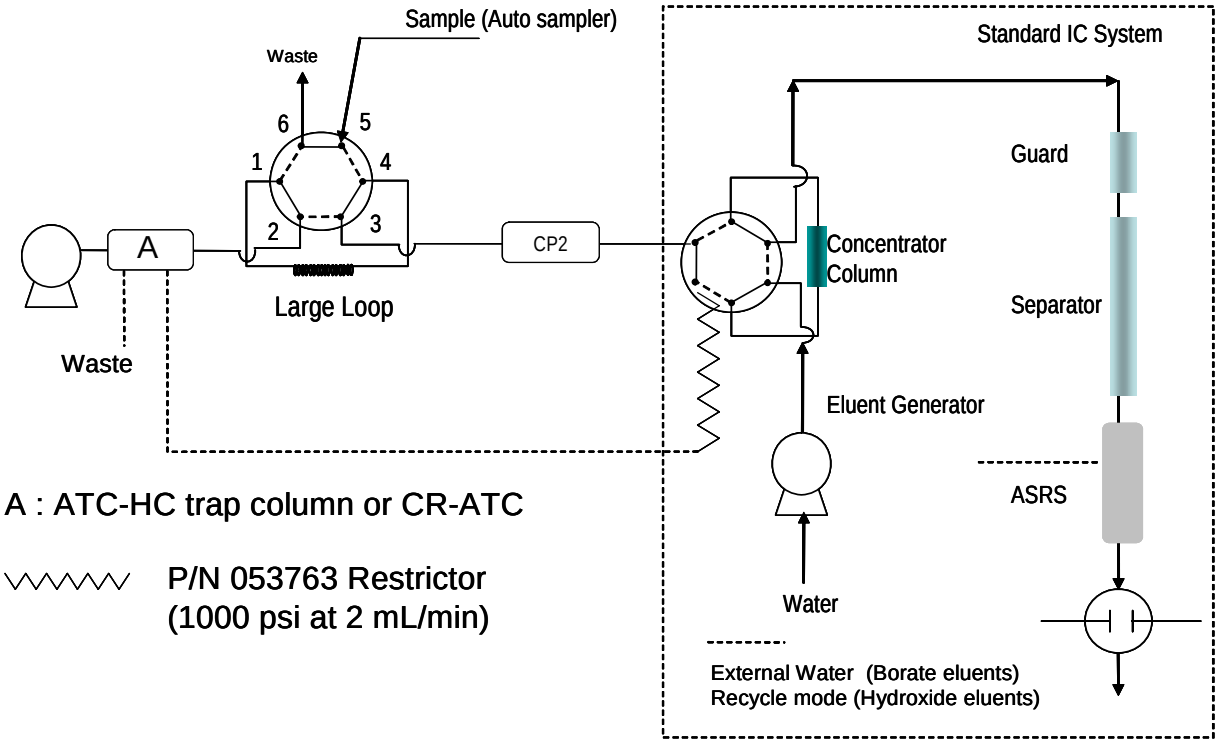

Figure 4
Plumbing Scheme for CP2 H^+ Form Cation Polisher Column

The purified DI water is then diverted into a 6 port valve which has a sample injection loop for the sample (located between 1 and 4). When loading the loop the autosampler (AS or AS-HV) loads the loop from port 5 to 4 to 1 to 6 to waste. During this time port 2 is connected to port 3 and the DI water flows through the CP2 H^+ Form Cation Polisher column and is then diverted into the injection valve on the IC system.

During the injection step the DI water is diverted from port 2 to 1 and displaces the large sample loop (previously loaded) via port 4 and 3. The sample slug is diverted into a CP2 H^+ Form Cation Polisher column which traps all cations including lithium, the lithium free sample stream containing all anions of interest. It is then diverted into a concentrator column in the injection valve in a standard IC system.

This set-up eliminates the lithium hence there is no issue with recovery or peak shapes for the early eluting anions and good quantification of all anions is expected. The CP2 H^+ Form Cation Polisher column should be regenerated using acid regenerants when the capacity is depleted. The regeneration frequency is dependent on the concentration of the cations in the sample.

For example, if the total concentration of cations is 10 ppb the CP2 H^+ Form Cation Polisher column would last roughly for 450 injections based on a 40 mL sample concentration. If there are metals in the sample stream, a cleanup of the CP2 H^+ Form Cation Polisher column is recommended on a bi-weekly basis with 500 mN sulfuric acid followed by 500 mM oxalic acid and DI water as outlined in section 4.4.

In experimental set-ups that pump the sample stream directly into the concentrator column using a sample pump, the CP2 H^+ Form Cation Polisher column is installed in the sample stream. The set-up is similar to Figure 4 except the anion trap column should be removed and the sample stream is directly pumped into the CP2 H^+ Form Cation Polisher column. All other plumbing requirements are similar to what is shown in Figure 4.

2.3. Recommended Operating Pressure

The recommended maximum operating pressure for the CP1 Na^+ Form Cation Polisher column and CP2 H^+ Form Cation Polisher column is 3,000 psi. Operating a column above its recommended pressure limit can cause irreversible loss of column performance.

3. REGENERATION AND STORAGE

3.1. Regeneration of CP1 Na^+ Form Cation Polisher Column (6 x 16 mm)

The CP1 Na^+ Form Cation Polisher column has a capacity of 170 µeq/col. When this capacity is exceeded, the column requires regeneration. It is recommended to use the Dionex Trap Column/Suppressor Clean-up Kit (P/N 059659) or the autosampler to regenerate the CP1 Na^+ Form Cation Polisher column to the sodium form. A stand-alone pump can also be used for the purpose of regeneration.

Regeneration steps are outlined below:

1) Pump at least 10 mL of 200 mM MSA through the CP1 Na^+ Form Cation Polisher column.
2) Pump at least 10 mL of 200 mM NaOH through the CP1 Na^+ Form Cation Polisher column.
3) Rinse off the base by pumping at least 5 mL of DI water.

The column is ready for polishing samples.

NOTE	**When implementing the above with the autosampler the concentrator column or injection loop should be in the inject position thereby avoiding contact with the regenerant.**

3.2. Regeneration of CP2 H^+ Form Cation Polisher Column (9 x 24 mm)

The CP2 H^+ Form Cation Polisher column has a capacity of 700 µeq/col. When this capacity is exceeded the column requires regeneration. It is recommended to use the Dionex Trap Column/Suppressor Clean-up Kit (P/N 059659) to regenerate the CP2 H^+ Form Cation Polisher column to the hydronium form. A stand-alone pump can also be used for the purpose of regeneration.

Regeneration steps are outlined below:

1) Pump at least 20 mL of 500 mM sulfuric acid or methanesulfonic acid through the CP2 H^+ Form Cation Polisher column.
2) Rinse off the acid by pumping at least 10 mL of DI water.

The column is ready for polishing samples.

3.3. Column Storage

The CP Cation Polisher columns are shipped with deionized water as the storage solution. The columns should be stored in the respective regenerated forms and then stored with deionized water as the storage solution. For regeneration steps follow section 3.1. and 3.2. respectively and after step 2 (DI water rinse) plug the column inlet and outlet ports for storage.

4. TROUBLESHOOTING GUIDE

The purpose of this section is to assist you with problems that may arise while using CP Cation Polisher columns. For more information on problems that originate with the Ion Chromatography System (ICS), the Anion Suppressor (ASRS, AMMS, AAES), the EG50 Eluent Generator System, refer to the Troubleshooting Guide in the appropriate operator's manual. If you cannot solve the problem on your own, contact the nearest DIONEX Office (see, "DIONEX Worldwide Offices" on the Dionex Reference Library CD-ROM, P/N 053891).

4.1. High Back Pressure

If the CP Cation Polisher column is the cause of high back pressure, its inlet bed support may be contaminated. To change the bed support, follow the instructions below using one of the two spare bed supports included in the Ship Kit.

A. Disconnect the CP Cation Polisher column from the IC system setup.

B. Using two open-end wrenches, carefully unscrew the inlet column end fitting.

C. Turn the end fitting over and tap it against a bench top or other hard, flat surface to remove the bed support assembly. If the bed support must be pried out of the end fitting, use a sharp pointed object such as a pair of tweezers, but be careful that the walls of the end fitting are not scratched. Discard the old bed support assembly.

D. Place a new bed support assembly (P/N 063688) for CP1 Na^+ Form Cation Polisher column or (P/N 060270) for CP2 H^+ Form Cation Polisher column into the end fitting. Carefully screw the end fitting onto the column so that the seal washer sits properly between the end fitting and the end of the column.

E. Screw the end fitting onto the column until it is finger tight and then use wrenches to tighten it an additional 1/4 turn (25 in x lb). Tighten further only if leaks are observed.

 **CAUTION**	**If the column tube end is not clean when it is inserted into the end fitting, particulate matter may prevent a proper seal between the end of the column tube and the bed support assembly. If this is the case, additional tightening may not seal the column but instead damage the column tube or the end fitting.**

F. Reconnect the CP Cation Polisher column to the Ion Chromatography System and resume operation.

If the CP1 Na^+ Form Cation Polisher or CP2 H^+ Form Cation Polisher column continues to give high pressure particularly after exposure to metal containing samples then a cleanup as outlined in section 4.4. is recommended. This cleanup needs to be done on a bi-weekly basis.

4.2. Poor Peak Shapes or Recovery

If poor peak shapes or recovery is observed especially for early eluting analytes this could be due to the depletion of the capacity of the CP Cation Polisher columns. A regeneration protocol as outlined in Section 3.1. or 3.2. as applicable should be pursued. If poor peak shapes or recovery persists after regeneration then this has to do with other components of the system. Systematically troubleshoot each component.

4.3. Leaking Column

If the pressure inside the CP Cation Polisher column increases due to precipitation then there is a possibility that the column might leak. Identify the source of the leakage by examining the end fittings and replace the appropriate bed support (inlet or outlet) for the leaking side. Follow instructions written in 4.1. to replace the bed support. If leakage persists a cleanup may be required as outlined in 4.4.

4.4. Metal Contamination

The CP1 Na^+ Form Cation Polisher or CP2 H^+ Form Cation Polisher column is compatible with low levels of metal ions in the µg/L (ppb) range. A bi-weekly cleanup is recommended for the CP1 Na^+ Form Cation Polisher and CP2 H^+ Form Cation Polisher columns. This cleanup can be automated for the CP1 Na^+ Form Cation Polisher column using the autosampler. It is recommended to use the Dionex Trap Column/Suppressor Clean-up Kit (P/N 059659) to cleanup the CP2 H^+ Form Cation Polisher column.

The cleanup steps for both CP1 Na^+ Form Cation Polisher column and CP2 H^+ Form Cation Polisher columns are:

1) Pump at least 10 mL of 500 mM sulfuric acid or methanesulfonic acid through the CP Cation Polisher column.
2) Pump at least 10 mL of 500 mM Oxalic acid through the CP Cation Polisher column.
3) Rinse off the acid by pumping at least 10 mL of DI water.

Regenerate the CP1 Na^+ Form Cation Polisher or CP2 H^+ Form Cation Polisher columns following the regeneration steps outlined in section 3.1. or 3.2. Step 3 above may be skipped for the CP2 H^+ Form Cation Polisher column since the regeneration step would require an acid rinse.

