

PRODUCT MANUAL

IonPac[®] Fast Cation II

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PRODUCT MANUAL

for the

IONPAC FAST CATION II COLUMN
(P/N SP5393)

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SECTION 1 INTRODUCTION

The IonPac Fast Cation II Column (P/N SP5393) is designed specifically for the rapid determination of alkaline earth metals. This manual describes the eluent systems that have been developed for this column, and how to use it in combination with the IonPac Fast Cation I Column (P/N SP5391), to obtain the fastest possible results for the simultaneous analysis of both alkali metals and alkaline earth metals.

Even though this is not its intended function, because of its low backpressure of 550 psi or less at 2 mL/min eluent flow rate, this column may be used as a guard and as a preconcentrator column.

1.1 Initial Inspection

As soon as you unpack the column, inspect it for shipping damage. Because many carriers limit the claim period to 10 days after the delivery date, Dionex recommends that you report any shipping damage to the incoming carrier immediately. To request help in assessing shipping damage, or to report a missing or damaged part, contact the Dionex office nearest you.

1.2 Column Warranty

The 90-day column warranty is in effect from the day the column is shipped from Dionex. After verifying that it was not damaged in transit, install the column in the system and test it by making several injections of the standard used to obtain the test chromatogram. The test chromatogram is shipped with the column. Contact the nearest Dionex Service Office if the performance of the column is unsatisfactory.

SECTION 2 - INSTALLATION

2.1 System Requirements

The IonPac Fast Cation II Column may be run on any Dionex Ion Chromatograph (IC) equipped with a Conductivity Detector and a Cation MicroMembrane Suppressor (CMMS III, P/N 056752).

NOTE: *Do not substitute either a Cation Fiber Suppressor or a packed bed suppressor column for the Cation MicroMembrane Suppressor (CMMS). None of the eluents required for the IonPac Fast Cation II Column will work satisfactorily with these suppressors.*

WARNING: DO NOT USE A GUARD COLUMN WITH THE IONPAC FAST CATION II COLUMN. CHROMATOGRAPHY WILL BE SEVERELY IMPAIRED.

NOTE: *This manual assumes you already know how to install and operate the Dionex Ion Chromatograph (IC) and the Cation MicroMembrane Suppressor (CMMS). If you do not, familiarize yourself with the operator's manuals for these products before beginning an analysis.*

2.2 Installing the Column Switching Valve

To perform rapid determinations of the alkali metals (plus ammonia), and alkaline earth metals from one sample injection under isocratic conditions, it is necessary to use a Dionex Inert 4 way High Pressure Valve (P/N 037143) for switching between the IonPac Fast Cation I Column and the IonPac Fast Cation II Column. Contact the Dionex Sales Representative nearest you to order this valve.

NOTE: *The "A" valve included in the CHA, or the 4 way 4,000 psi valve included in the CHB, may be used for this purpose.*

Figure 2.1 shows the port orientation and plumbing of the column switching valve. The valve has two stacks, each one with four valve ports. The ports are in two different planes, one above the valve's vent hole and the other below it. The label on the valve has an arrow to show the top of the valve. The ports are numbered clockwise starting with the top stack, port 1 is above the hole, port 5 is below the hole, and port 7 is diametrically opposite port 5. The IonPac Fast Cation II Column is connected between ports 4 and 8. The IonPac Fast Cation I Column is connected between ports 2 and 6. Ports 3 and 5 are connected with 7 inches of 0.012" ID tubing.

Port 1 of the column switching valve is connected to port 5 of the injection valve. Port 7 is connected to the inlet of the Cation MicroMembrane Suppressor (CMMS). The outlet of the CMMS is connected to the detector cell inlet.

Use 0.012" ID tubing (P/N 035548) to make the connections to the column switching valve and columns. Keep all tubing lengths as short as practically possible to reduce the amount of dead volume added to the system.

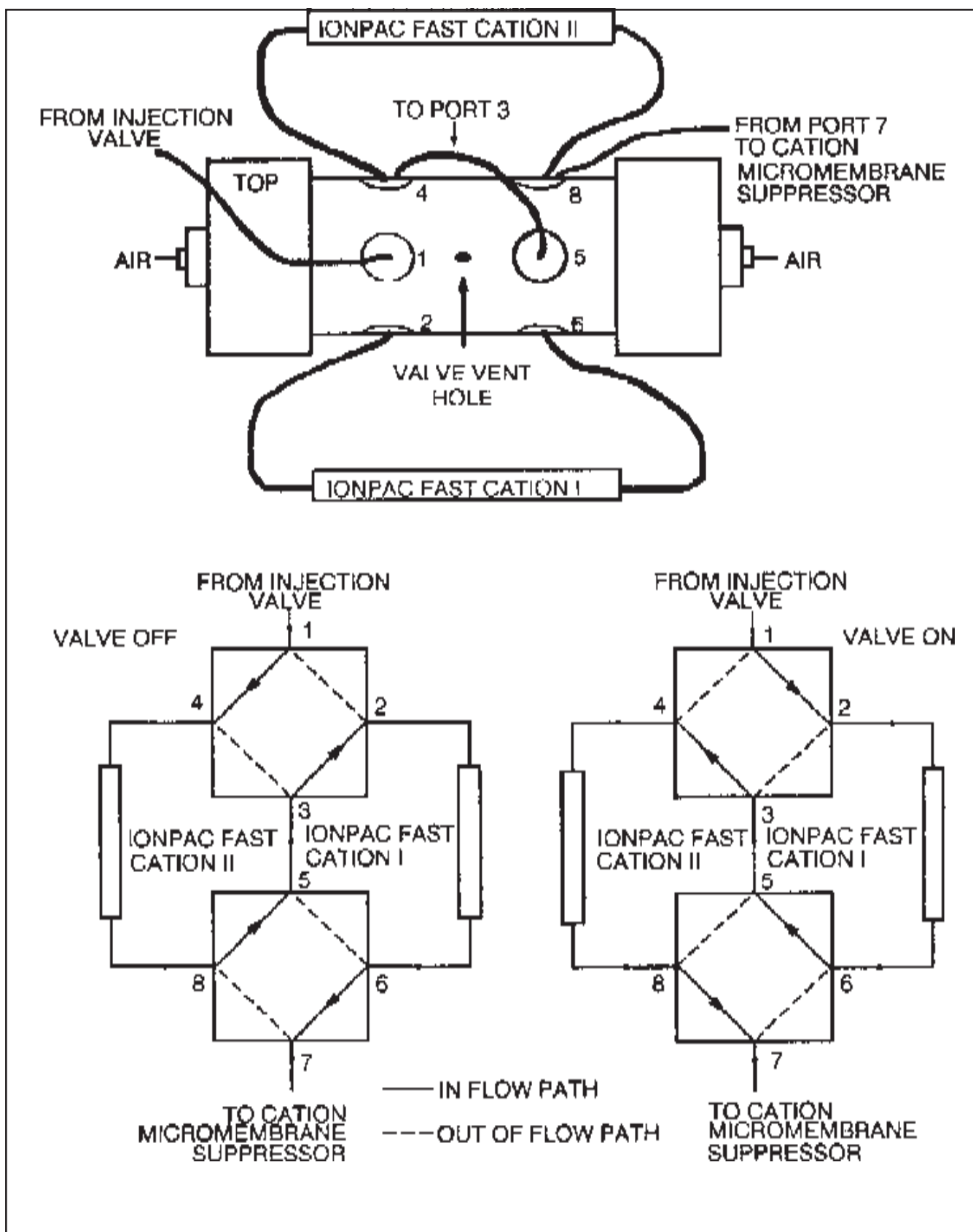
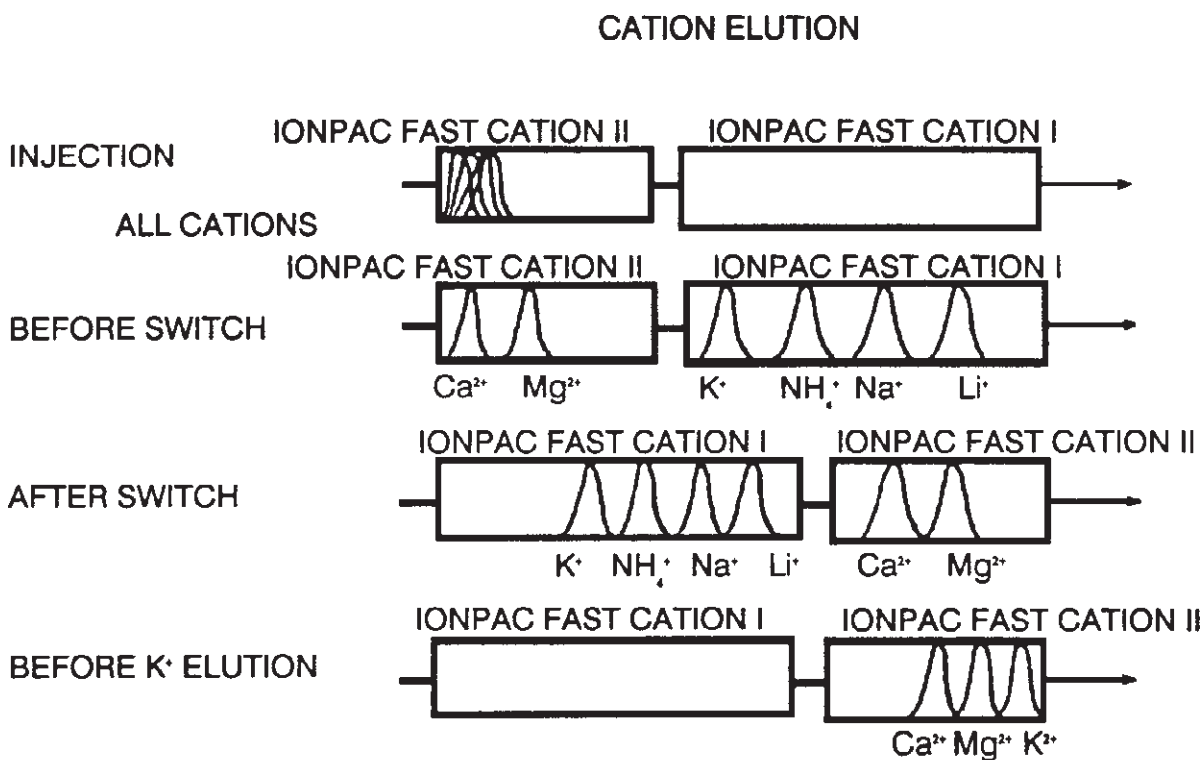


Figure 2.1
Plumbing Connections to the Column Switching Valve

SECTION 3 - OPERATION

3.1 General Operating Instructions

The IonPac Fast Cation II Column is used with the IonPac Fast Cation I Column (P/N 039591) in the Column Switching Mode to obtain the fastest possible results for the simultaneous analysis of both alkali metals and alkaline -earth metals under isocratic eluent conditions. Figure 3.1 is a representation of the separation scheme.



* COLUMN SWITCHING OCCURS AT 40 SECONDS

Figure 3.1
Column Switching Separation Scheme

When the sample is injected, the column switching valve should be in the OFF position: the eluent flows first through the IonPac Fast Cation II Column and then through the IonPac Fast Cation I Column. The divalent cations in the sample will be retained by the IonPac Fast Cation II Column, while the monovalent cations are essentially unretained by this column and move to the IonPac Fast Cation I Column where the monovalent cations are separated.

The switching valve is switched "ON" forty seconds after injecting the column. The eluent flows through the IonPac Fast Cation I Column and then through the IonPac Fast Cation II Column. This prevents the divalent cations in the sample from going through the IonPac Fast Cation I Column, providing rapid divalent cations separation by the IonPac Fast Cation II Column.

If you have a Gradient Pump Module (GPM), you can control the injection valve and the column switching valve automatically through GPM valves 5 and 6 respectively. The program would be as follows:

TIME (minutes)	VALVE5 (Injection Valve)	VALVE6 (Column Switching Valve)
0	ON	OFF
0.6	ON	ON
7.5*	OFF	ON
7.6*	OFF	OFF

* This time depends on the type of the analysis.

If you do not have a GPM, and want to automate control of the valve, contact the Dionex Sales Representative nearest you for automation information.

3.2 General Operating Conditions

Eluent components:	HCl and DL-2,3-diaminopropionic acid monohydrochloride (DAP·HCl)
Eluent flow rate:	2.0 mL/min
Regenerant:	0.1 M tetrabutylammonium hydroxide (TBAOH)
Regenerant flow rate:	greater or equal to 5 mL/min
Detector Range:	10 μ S full scale or as required
Sample loop:	50 μ L- (or as required)

3.3 Chemicals Required

HCl DL 2, 3 diaminopropionic acid monohydrochloride (DAP·HCl) at 2.0 mL/min/0.1 M tetrabutylammonium hydroxide (TBAOH) greater or equal to 5 mL/min/10 μ S full scale or as required 50 μ L sample loop.

It is very important for the eluent to be as free of metallic impurities as possible. Thus, chemicals and water used should be of the greatest available purity.

- A. Only use concentrated HCl, Ultrex grade or Baker Instra Analyzed for Trace Metals.
- B. Use Dionex DL 2,3 Diaminopropionic acid monohydrochloride (DAP·HCl, P/N 039670) ONLY.
- C. Use Dionex Cation Regenerant Solution (0.1 M tetrabutylammonium hydroxide, TBAOH, P/N 39602).
- D. Use deionized water with a specific resistance of 18.2 megohm cm or better.

3.4 Solutions Required

- A. For 1.0 M HCl Stock Solution: Calculate the amount, in grams, of concentrated HCl needed to add to a 1 liter volumetric flask by using the HCl composition stated on the label of the particular HCl bottle you are using:

$$\text{Grams HCl} = \frac{36.46 \text{ g/mole} \times 1 \text{ mole} \times 100}{\% \text{ HCl}}$$

SAFETY: AVOID BREATHING THE VAPORS

Carefully add this amount of HCl to a 1 liter volumetric flask containing about 500 mL of deionized water with a specific resistance of 18.2 megohm cm, and dilute to the mark. For example, if the HCl concentration was 38%, to make a 1 M HCl solution you would have to weigh out 95.95 g of concentrated HCl.

- B. For 3.0 mM DAP·HCl Stock Solution: Dissolve 0.422 g of DAP·HCl in a 1 liter volumetric flask. Dilute to the 1 liter mark with deionized water having a specific resistance of 18 megohm cm.
- C. For 0.1 M TBAAOH: Use as supplied.

3.5 Sample Preconcentration

The limit of detection can be enhanced by preconcentrating the sample onto a concentrator column and using this column in lieu of the sample loop. The sample should be loaded onto the column in the **OPPOSITE** direction of the eluent flow; otherwise the chromatography will be compromised.

The following columns may be used for preconcentration with the IonPac Fast Cation I and II Columns:

- IonPac CG3 Guard Column (P/N 037025)
 - The Trace Cation Concentrator (TCC-1) provides the least baseline disturbance when the preconcentrator column is placed in line with the system. This is due to its low backpressure contribution.
 - Trace Cation Concentrator (TCC-1, P/N 037032)
 - IonPac Fast Cation II Column.
 - The IonPac CG3 Guard Column and the IonPac Fast Cation II Column will have higher capacity than the Trace Cation Concentrator (TCC-1), but will create a larger baseline disturbance as their backpressure contribution is appreciably larger.
-

SECTION 4- EXAMPLE APPLICATIONS

4.1 Analytes: Lithium, Sodium, Ammonium, Potassium, Magnesium, and Calcium

By using column switching between an IonPac Fast Cation I column and an IonPac Fast Cation II column, these six monovalent and divalent cations may be determined isocratically. The eluent is 20 mM HCl / 0.3 mM DAP·HCl.

A column switching valve, and means of activating it, is required to perform this analysis. The column switching valve is activated 40 seconds (0.6 min.) after the sample is injected.

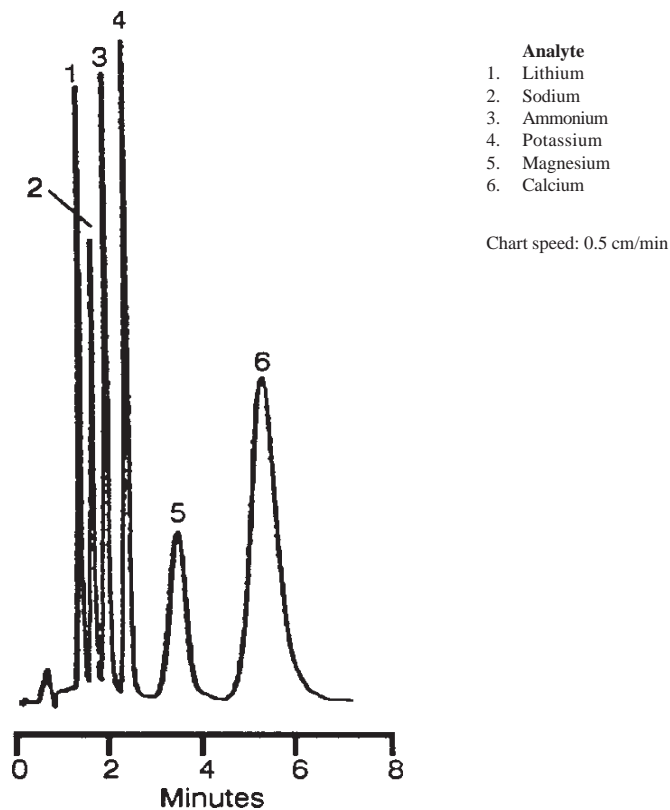
NOTE: *The IonPac Fast Cation I Column and the column switching valve are not included. They may be ordered from Dionex: IonPac Fast Cation I Column (P/N SP5391); Dionex Inert 4 way High Pressure Valve (P/N 037143). The "A" valve included in the CHA, or the 4 way valve included in the CHB, may be used for this purpose.*

4.1.2 Eluent Preparation

- **Eluent: 20 mM HCl / 0.3 mM DAP·HCl.**

To a 1 liter volumetric flask containing about 500 mL of deionized water with a specific resistance of 18.2 megohm-cm:

- a) Add 20 mL of the 1 M HCl stock solution
- b) Add 100 mL of the 3 mM DAP·HCl stock solution prepared in Section 3.4.
- c) Dilute to the mark.



4.2 Analytes:

Lithium, Sodium, Ammonium, Potassium, Rubidium, Cesium, Magnesium, Calcium, Strontium, and Barium

By using column switching between an IonPac Fast Cation I column and an IonPac Fast Cation II column, these ten monovalent and divalent cations may be determined isocratically. The eluent is 17 mM HCl / 0.26 mM DAP·HCl.

A column switching valve, and means of activating it, is required to perform this analysis. The column switching valve is activated 40 seconds (0.6 min.) after the sample is injected.

NOTE: *The IonPac Fast Cation I Column and the column switching valve are not included. They may be ordered from Dionex: IonPac Fast Cation I Column (P/N 039591); Dionex Inert 4-way High Pressure Valve (P/N 037143). The "A" valve included in the CHA or the 4-way valve included in the CHB may be used for this purpose.*

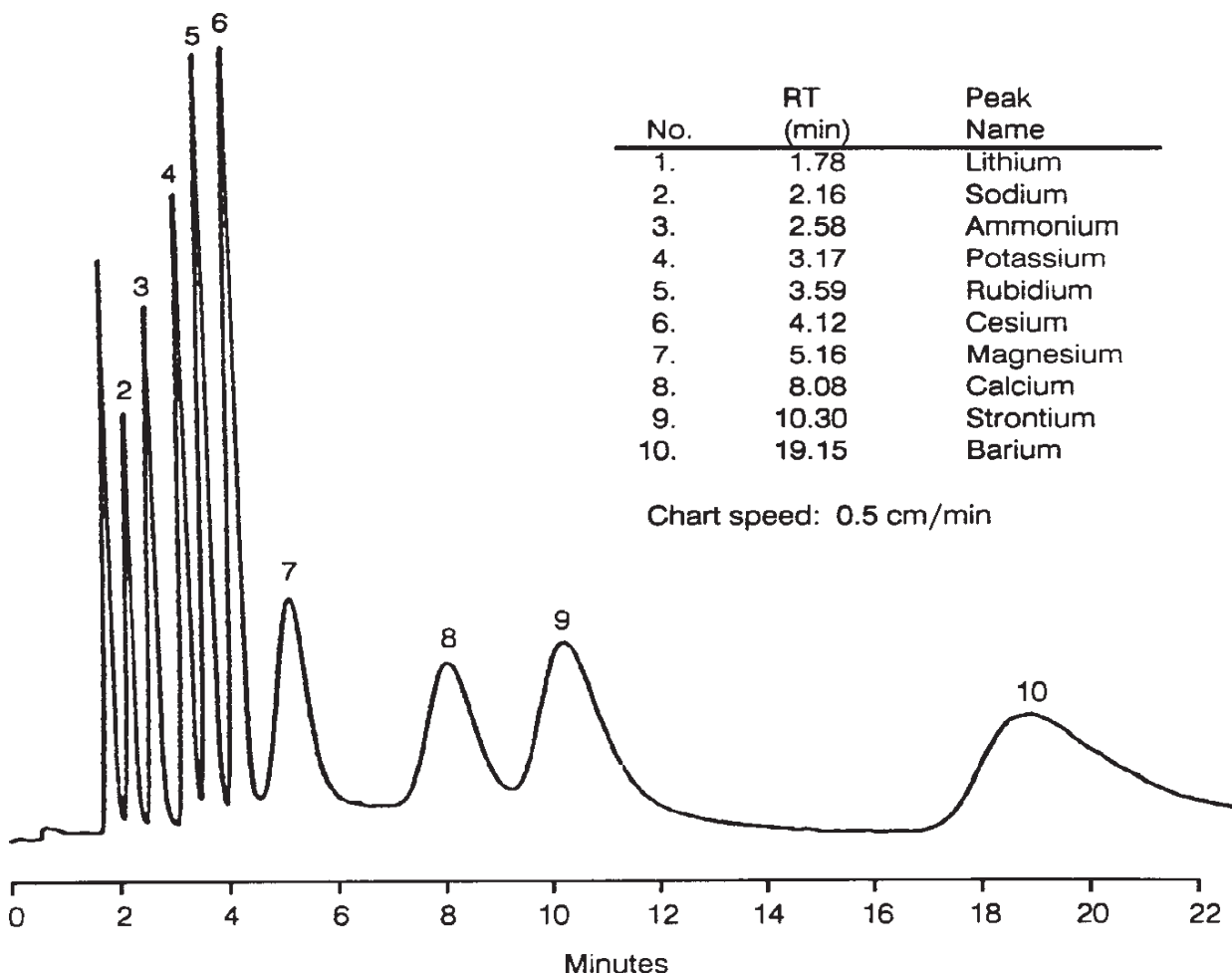
If you have an Analytical Pump Module (APM) instead of a Gradient Pump Module (GPM), you may need to order other parts to automate the column switching valve. Contact the Dionex Sales Representative nearest you for more information on this subject.

4.2.1 Eluent Preparation

• **Eluent: 17 mM HCl / 0.26 mM DAP·HCl.**

To a 1 liter volumetric flask containing about 500 mL of deionized water with a specific resistance of 18.2 megohm-cm:

- a) Add 17 mL of the 1 M HCl stock solution
- b) Add 85 mL of the 3 mM DAP·HCl stock solution.
- c) Dilute to the mark.



SECTION 5 - TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help solve operating problems which may arise while using the IonPac Fast Cation II Column. For more information on problems which originate with the Ion Chromatograph (IC), or the Cation MicroMembrane Suppressor (CMMS III, P/N 056752), refer to the Troubleshooting Guide in the appropriate operator's manual. If you cannot solve the problem on your own, call the Dionex Service Office nearest you.

5.1 High Backpressure

Total system pressure when using the IonPac Fast Cation II Column should be approximately 550 psi; however, when combined with an IonPac Fast Cation I Column in a column switching application the total system pressure should be less than 1550 psi. If it is much higher than 1550 psi., it is advisable to find out what is causing the high pressure.

- A. Make sure that the pump is set to 2.0 mL/min. Higher flow rates will cause higher pressure.
- B. Find out what part of the system is causing the high backpressure.
 - A connecting line of tubing could be plugged or crimped.
 - The injection valve might have a plugged port.
 - The column might have particulates plugging the inlet bed support.
 - The Cation MicroMembrane Suppressor or the detector might be plugged.

To find out which part of the system is causing the problem:

- 1) Disconnect the pump eluent line from the injection valve and turn the pump on.
- 2) Watch the pressure. It should not be more than 50 psi.
- 3) Add the system components (injection valve, column, suppressor and detector) one by one while watching the pressure.
 - The pressure should increase to approximately 550 psi (or 1550 psi when combined with an IonPac Fast Cation I Column in column switching applications) when the IonPac Fast Cation II Column is connected. The Cation MicroMembrane Suppressor will add approximately 100 psi. No other component should add more than 100 psi of pressure. Refer to the appropriate manual for cleanup of the problem component.
- C. If the column is the cause of high backpressure, its inlet bed support may be contaminated. To change the bed support, follow the instructions below using one of the two spare bed support assemblies included in the Ship Kit.
 - 1) Disconnect the column from the system.
 - 2) Using two open end wrenches, carefully unscrew the inlet (top) column fitting.
 - 3) Turn the end fitting over and tap it against a bench top or other hard, flat surface to remove the bed support and seal assembly.
 - 4) Discard the old assembly.
 - 5) Place a new bed support assembly (P/N 042955, consisting of Seal Washer, P/N 042956, and Bed Support P/N 053889) into the end fitting (P/N 042367).
 - 6) Use the end of the column to carefully push the bed support assembly into the end fitting.
 - 7) Screw the end fitting back onto the column.
 - Tighten it finger tight, then an additional 1/4 turn (25 lbs - in). Tighten further only if leaks are observed.

NOTE: If any of the column packing becomes lodged between the end of the column and the bed support washer assembly, no amount of tightening will seal the column. Make sure that the washer and the end of the column are clean before screwing the end fitting back onto the column.

- 8) Reconnect the column to the system and resume operation.
-

5.2 High Background Noise

In a properly working system, the background conductivity level under the operating conditions should be below 5 μS . A system with a high background will probably also have high noise, with subsequent increase of detection limits.

- a) Make sure that the eluents and regenerant are made correctly.
- b) Make sure that the eluents are made from chemicals with the recommended purity.
- c) Make sure that the deionized water used to prepare the reagents has close to 18.2 megohm/cm specific resistance.
- d) Remove the column from the system.
- e) Check the background noise.
 - If still too high, then continue troubleshooting.
 - If the background noise reduced to operational levels, then replace the column.
- f) To ensure contaminated hardware is not the cause of the high background noise, use deionized water with a specific resistance of 18.2 megohm/cm as eluent. The background should be $\leq 2 \mu\text{S}$.
- g) Check the detector and conductivity cell by injecting deionized water with a specific resistance of 18.2 megohm/cm directly into it.
- g) If the above items have been checked and the problem still persists, the Cation MicroMembrane Suppressor, CMMS III, is probably causing the problem.
 1. Check the regenerant flow rate at the CMMS REGEN Out port. This flow rate should be $\geq 5 \text{ mL/min}$.
 2. Check the eluent flow rate. It should be 2.0 mL/min.
 3. Prepare fresh regenerant solution.
 - If you are using an AutoRegen accessory, pump about 200 mL of regenerant through the AutoRegen Cation Cartridge (P/N 039563) to waste before recycling the regenerant back to the regenerant reservoir.
 4. If the background is still high, bypass the AutoRegen Cation Cartridge in the AutoRegen Accessory.
 5. If the background is now low, you probably need to replace the AutoRegen Cation Cartridge (P/N 039563).
 6. You may need to clean the Cation MicroMembrane Suppressor (CMMS III, P/N 056752). For instructions on how to do this, refer to the CMMS manual. If the cleanup procedure does not work, you may have to replace the CMMS.

5.3 Poor Peak Resolution

Poor peak resolution may be due to the loss of column or system efficiency. It can also be observed if the column loses capacity or selectivity.

- A. The loss of column efficiency
 1. Check to see if headspace has developed in the column (e.g., due to improper use of the column such as using the column with organic solvents, submitting it to high pressures, or high pH). Remove the column inlet end fitting. If the resin does not fill the column body all the way to the top, the resin bed has collapsed creating a headspace. The column must be replaced.
 2. Extra column effects can result in sample band dispersion making the peak elution less efficient. Make sure you are using 0.012" ID tubing between the injection valve and the detector cell inlet, and that the tubing lengths are minimized. Check for leaks.
 - B. Shortened solute retention times will compromise resolution. Potential reasons for shorter retention times are the following:
 1. Check to see if eluent flow rate is faster than 2.0 mL/min. Check the eluent flow rate after the column.
 2. Check to see if the eluent composition and concentration is correct. An eluent which is too strong will make the peaks elute sooner. Prepare fresh eluent. If you are using a GPM to proportion the eluent components from two or three different eluent reservoirs, the resulting eluent composition might not be accurate enough for this application. Use one reservoir containing the correct eluent composition to see if this is the problem.
 3. Column contamination can lead to a loss of column capacity because all of the exchange sites will no longer be available for the sample ions. Polyvalent cations might be concentrating on the column. Under the eluent conditions used for the monovalent cations (20 mM HCl/0.3 mM DAP·HCl) these would take a very long time to elute.
 - C. Possible sources of contamination are:
 1. Gradient Mixers (GM2, P/N 037146) in the Gradient Pump Module (GPM) with serial numbers below 6500 should be removed from the system or replaced with Gradient Mixers having serial numbers above 6500 when using the IonPac Fast Cation I and II Columns.
 2. There may be impurities in the chemicals or in the deionized water being used. Care should be taken to ensure that the recommended chemicals are used. The deionized water should have a specific resistance of at least 18.2 megohm/cm.
 3. The system should be as metal free as possible. Gripper tube fittings are a potential source for metal contamination. The new Dionex ThermoFlare fittings are preferred. The eluent pumps should be inspected periodically for signs of leaks.
 - Impurities may come from glass eluent bottles. Polyethylene 2 liter eluent containers (P/N 039163) are preferred.
 - Diluting the eluent will improve peak resolution, but will also increase the retention times of the analytes.
 - If a 10% dilution of the eluent is not sufficient to obtain the desired peak resolution or if the resulting increase in retention times is unacceptable, clean the column as described in Column Care.
 - After cleaning, reinstall the column in the system and equilibrate with eluent for about 15 minutes. The column is equilibrated when consecutive injections of the standard give reproducible retention times. The original column capacity should be restored by this treatment, since the contaminant metals should be eluted from the column.
-

5.4 Spurious Peaks

Spurious peaks can be due to column contamination or system component malfunctions.

A. Column Contamination:

If the samples contain an appreciable level of polyvalent ions and the column is used with a weak eluent system such as 20 mM HCl/0.3 mM DAP·HCl, polyvalent cations may be contaminating the column. The retention times for the analytes will then decrease, and spurious — inefficient peaks can show up at unexpected times. Clean the column as indicated in Column Care. Using the recommended eluent will ensure that strongly retained polyvalent cations are eluted before the next injection.

B. Baseline Upsets:

The possibility of creating a baseline disturbance exists when an injection valve or a column switching valve is actuated. This baseline upset can show up as a peak of varying size and shape. It will happen when the particular valve needs to be cleaned or torqued (see system manual). Small baseline disturbances at the beginning or end of the chromatogram can be overlooked as long as they do not interfere with the quantitation of the peaks of interest.

If baseline disturbances still occur after the valve has been cleaned, reassembled, and torqued (see manual), replace the valve with a Dionex High Pressure Inert Valve, a Dionex 3 or 4 way valve, or a Dionex Micro injection valve as required.

COLUMN CARE

RECOMMENDED OPERATING PRESSURES

Operating a column above its recommended pressure limit can cause irreversible loss of column performance. The maximum recommended operating pressure for the IonPac Fast Cation I and II Column system is 2000 psi.

COLUMN START-UP

The column is shipped with eluent as the storage solution. This eluent is the same one shown in the test chromatogram (20 mM HCl/0.3mMDAP-HCl).

- a) Prepare the eluent listed on the test chromatogram
- b) Install the column in the chromatography module
- c) Let the column equilibrate with the eluent for a few minutes.
- d) Test the column performance under the conditions described in the test chromatogram.

NOTE: *The column can be used with any of the other eluents discussed in the Manual. Remember to let the column equilibrate with the new eluent. It may take as much as one hour. The column is equilibrated and ready for use when consecutive injections of the standard give reproducible retention times.*

COLUMN STORAGE

The column's storage solution should be the eluent used for the particular application. If the column will not be used for one week or more, prepare it for long term storage.

- a) Flush the column for a few minutes with the eluent.
- b) Cap both ends securely, using the plugs supplied with the column.

COLUMN CLEANUP

The column can be cleaned as follows:

- a) Connect a container of 1 M HCl (prepared from concentrated HCl, Baker Instra-Analyzed for Trace Metals only) directly to the bottom (inlet) of the pump's priming block (i.e. by-passing the eluent select valves).
- b) Pump 40 mL of 1 M HCl (prepared from concentrated HCl, Baker Instra-Analyzed for Trace Metals only) through the column at 2.0 mL/min.
- c) After pumping the acid through the column, disconnect the reservoir from the priming block and reconnect the eluent valve line.
- d) Rinse with 5 mL of eluent.

NOTE: *When cleaning columns, disconnect the line between the Gradient Pump Module pressure transducer and the Chromatography Module at the Chromatography Module end. Connect this line to the top of the column to be cleaned and direct the effluent from the column to a waste container. Do not run any of the cleaning solutions through any of the other columns or modules in line (such as the other IonPac Fast Cation Column, I or II, in column switching applications, the MicroMembrane Suppressor or the detector).*

- Although it might not be as efficient for column cleanup, you may want to try pumping 60 ml of a solution containing ten times the eluent concentration (e.g. 200 mM HCl/3 mlVI DAP-HCl) through the column at 2mL/min. This can be especially advantageous when you have such a solution at hand for eluent stock or for proportioning (diluting) with deionized water in a Gradient Pump Module. The effluent from the IonPac Fast Cation II Column cleanup should be directed directly to a waste container. The effluent from this cleanup should not be pumped over an IonPac Fast Cation I Column if it is present for a column switching application.
- e) After cleanup of the IonPac Fast Cation II Column, reinstall it in the system
 - f) Let it equilibrate with eluent for about 15 minutes. The column is equilibrated and ready for use when consecutive injections of the standard give reproducible retention times.
-

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