PRODUCT MANUAL

IONPAC® FAST CATION I COLUMN

(4 x 250 mm, P/N SP5391)

QUICKSTART STEPS AND LINKS
Click blue text below to get started.

1. See Section 3, "Operation". Note operation precautions and chemical purity requirements. Make the required stock and working solutions for eluents.

3. See "Quality Assurance Report". Run the Production Test Chromatogram as a system check.

4. See Section 4, "Example Applications" for example applications.

5. See "Column Care" for column cleanup and long-term storage recommendations.

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7 November 2002

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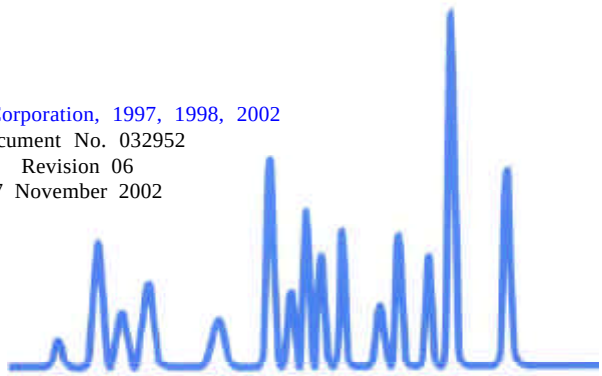

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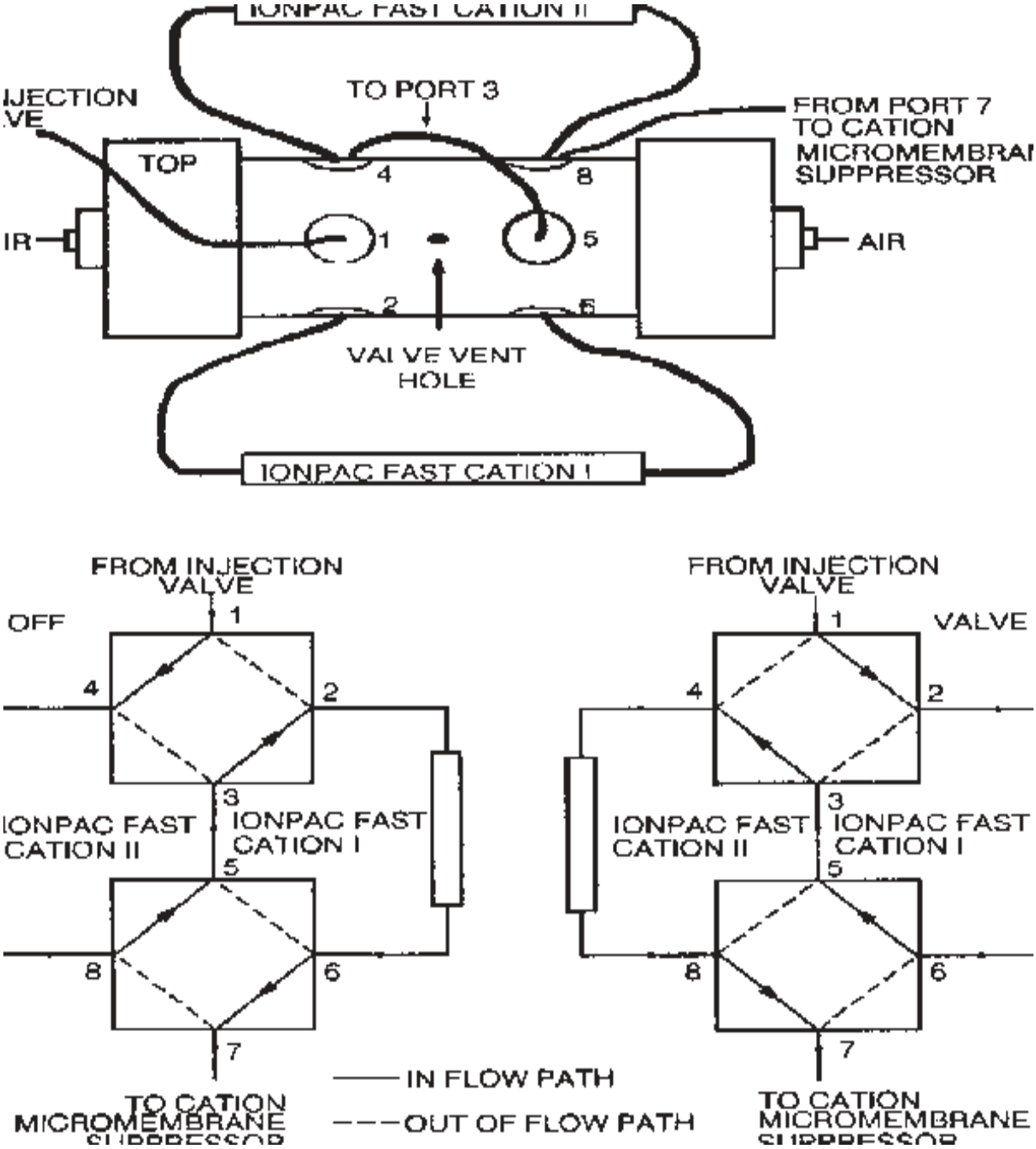
SECTION 1 - INTRODUCTION

The IonPac Fast Cation I Analytical Column (P/N SP5391) is designed specifically for the rapid determination of alkali metals and 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 II Analytical Column (P/NSP5393) to obtain the fastest possible results for the simultaneous analysis of both alkali metals and alkaline-earth metals.



SECTION 2 - INSTALLATION

2.1 System Requirements

The IonPac Fast Cation I Analytical Column may be run on any DIONEX Ion Chromatograph (IC) equipped with a Conductivity Detector (CDM-II) and a Cation MicroMembrane Suppressor (CMMS III, P/N 037076).

NOTE

Do not substitute either a Cation Fiber Suppressor or a packed bed suppressor column for the Cation MicroMembrane Suppressor. None of the eluents required for the IonPac Fast Cation I Analytical Column will work satisfactorily with these suppressors. To save regenerant preparation time, consumption, and waste, it is recommended that the AutoRegen™ Accessory be used.

The IonPac Fast Cation I Analytical Column can be used alone but is specifically designed to be used in combination with the IonPac: Fast Cation II Analytical Column (P/N SP5393). This requires the use of a column switching valve. Refer to the IonPac Fast Cation II Analytical Column Product Manual (Document No. 032953) for complete installation instructions if you plan to use both columns in a column switching application.

NOTE

Use DIONEX ThermoFlare fittings on all liquid lines.

An IonPac CG3 Guard Column (P/N 037025) may be used with the IonPac Fast Cation I Analytical Column. However, retention times will increase by approximately 20%.

CAUTION

If you are using a Gradient Pump Module, remove any high or low pressure GM-2 Gradient Mixers with serial numbers below 6500 during analyses with IonPac Fast Cation I or IonPac Fast Cation II Analytical Columns. The alumina in these mixers can potentially react with the HCI/DL-2,3-Diaminoproplonic acid (DAP) eluents and contaminate the columns.

For isocratic analyses (such as IonPac Fast Cation I and II column switching analyses) the gradient pump can be operated without mixers.

GM-2 Gradient Mixers with serial numbers 6500 or greater are packed with Teflone packings that will not Interfere with analyses done with the IonPac Fast Cation I or II Analytical Columns. If your Gradient Pump Module is equipped with GM-2 Gradient Mixers having serial numbers of 6500 or greater, there is no need to change them.

It you anticipate developing gradient applications with the IonPac Fast Cation I or II Analytical Columns, install high and low pressure GM-2 Gradient Mixers (P/N 037146) in the Gradient Pump Module having serial numbers of 6500 or greater.

This manual assumes that you already know how to install and operate the DIONEX Ion Chromatograph (IC) and the Cation MicroMembrane Suppressor (CMMS III, P/N 056752). If you do not, familiarize yourself with the Product Manual for these products before beginning an analysis.

SECTION 3 - OPERATION

3.1 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 µS full scale or as required
Sample loop:	50 µL- (or as required)

3.2 Chemicals Required

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, ULTREX12 grade or BAKER INSTRA-ANALYZEDO for trace metals.

B. Use DIONEX DL-2,3-Diaminopropionic acid monohydrochloride (DAP·HCl, P/N039670) ONLY.

C. Use DIONEX Cation Regenerant Solution (0.1 M tetrabutylammonium hydroxide, TBAOH, P/N 039602).

D. Use deionized water, with a specific resistance of 18.2 megohm-cm.

3.3 Solutions Required

A. 1.0 M HCl stock solution: Calculate the amount (in grams) of concentrated HCl you need 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:

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

Carefully (AVOID BREATHING THE VAPORS) 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. Dilute to a final volume of 1 L. 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. 3.0 rnM 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 18.2 megohm-cm.

3.4 Sample Concentration

Limits of detection can be enhanced by concentrating the sample onto a concentrator column prior to doing an analysis 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 can be used for sample concentration with the IonPac Fast Cation I and II Analytical Columns:

the IonPac CG3 Guard Column (P/N 037025), the Trace Cation Concentrator (TCC-1, P/N 037032) and the IonPac Fast Cation II Analytical Column (P/N 039633).

A. The Trace Cation Concentrator (TCC-1) provides the least baseline disturbance when used as a concentrator column and placed in line with the system. This is due to its low backpressure contribution.

B. The IonPac CG3 Guard Column and the IonPac Fast Cation II Analytical Column have higher capacities than the TCC-1, but will create a larger baseline disturbance as their backpressure contribution is appreciably larger.

SECTION 4 - EXAMPLE APPLICATIONS

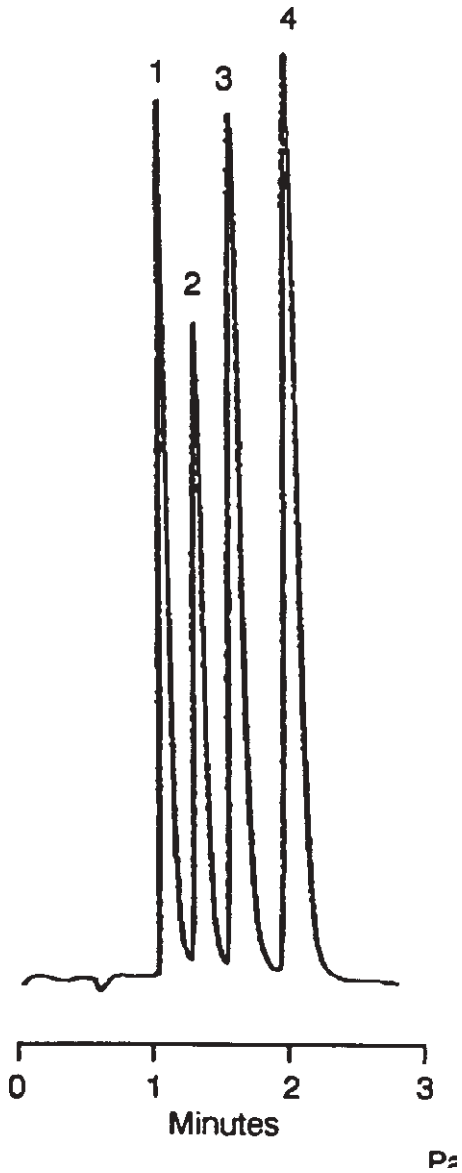
4.1 Analytes: Lithium, Sodium, Ammonium and Potassium

These 4 monovalent cations are determined isocratically with the IonPac Fast Cation I Analytical Column in about 2.5 minutes using 20 mM HCl/0.3 mM DAP·HCl as eluent.

Eluent Preparation

Eluent: 20 rnM HCl/0.3 rnM DAP-HCl. To a 1 liter volumetric flask containing about 500 mL of deionized water with a specific resistance of 18.2 megohm-cm , add 20 mL of the 1 M HCl stock solution and 100 mL of the 3 mM DAP·HCl stock solution prepared in Section 3.3. Dilute to the mark.

1. Lithium
2. Sodium
3. Ammonium
4. Potassium



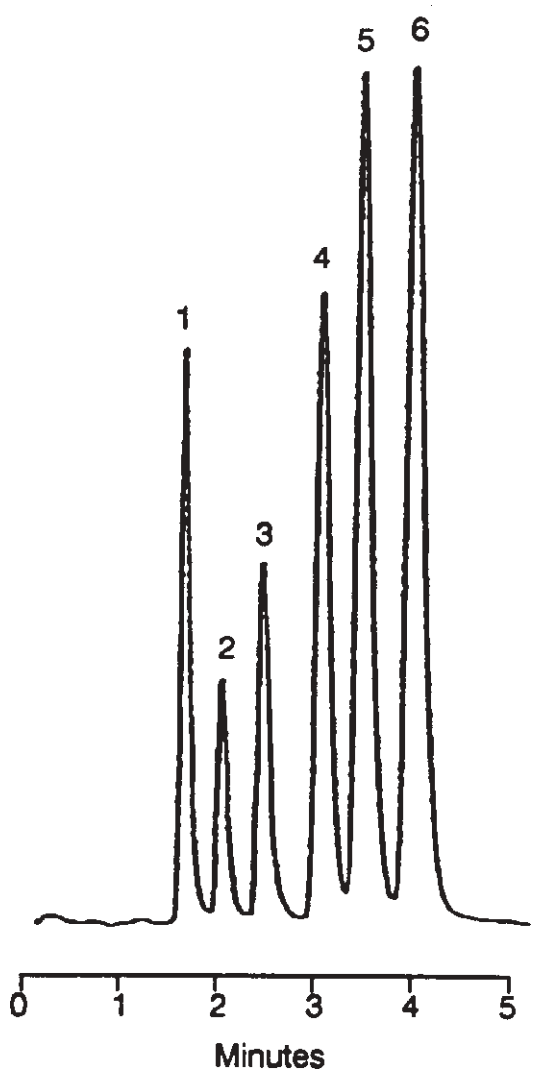
4.2 Analytes: Lithium, Sodium, Ammonium, Potassium, Rubidium and Cesium

These 6 monovalent cations are determined isocratically using the IonPac Fast Cation I Analytical Column in about 3.5 minutes using 17 mM HCl/ 0.26 mM DAP·HCl as eluent.

Eluent Preparation

Eluent: 17 rnM 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, add 17 mL of the 1 M HC1 stock solution and 85 mL of the 3 mM DAP·HCl stock solution. Dilute to the mark.

1. Lithium
2. Sodium
3. Ammonium
4. Potassium
5. Rubidiurn
6. Cesium



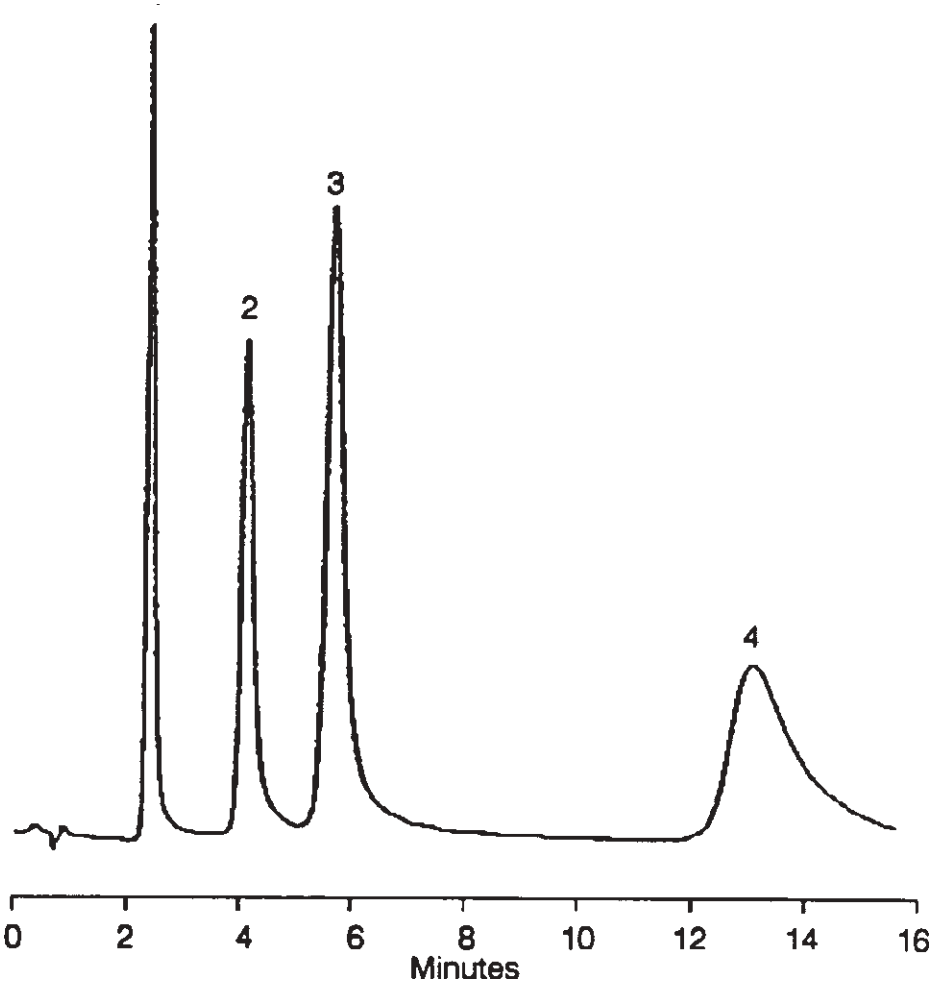
4.3 Analytes: Magnesium, Calcium, Strontium and Barium

These 4 divalent cations are determined isocratically with the IonPac Fast Cation I Analytical Column in about 13.5 minutes using 34 mM HCl/9 mM DAP·HCl as eluent.

Eluent Preparation

Eluent: 34 mM HCI/9 rnM DAP·HCI. To a 1 L volumetric flask containing about 500 mL of deionized water with a specific resistance of 18.2 megohm-cm, add 34 mL of the 1 M HCl stock solution. Dissolve in it 1.27 g of DAP·HCl. Dilute to the 1 liter mark.

1. Magnesium
2. Calcium
3. Strontium
4. Barium



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

These 6 monovalent and divalent cations are determined isocratically with the IonPac Fast Cation I Analytical Column AND the IonPac Fast Cation II Analytical Column by using column switching. The eluent used is 20 mM HCl/0.3 mM DAP·HCl.

A column switching valve and means of activating it are required to perform this analysis. The column switching valve is activated 40 seconds (0.6 min.) after the sample is injected. The column switching valve plumbing diagram is included in the IonPac Fast Cation II Analytical Column Product Manual (Document No. 032953).

NOTE

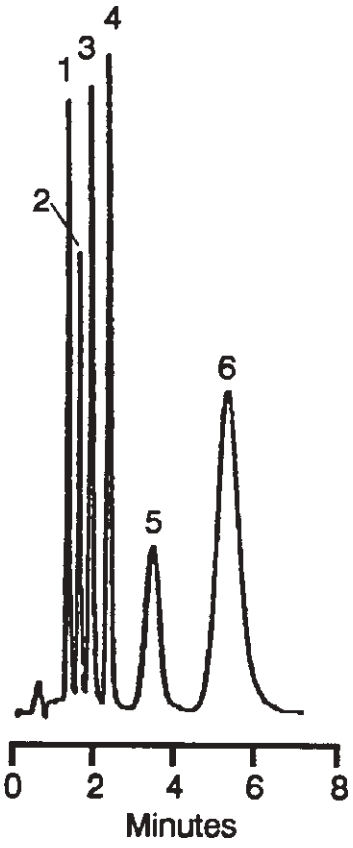
The IonPac Fast Cation II Analytical Column and the column switching valve are not included. They may be ordered from DIONEX: IonPac Fast Cation II Analytical Column (P/N SP5393); DIONEX Inert 4-way High Pressure Valve (P/N 037143). The A valve included in the CHA or the 4-way valve included in the CHB 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 Regional Office (see DIONEX Worldwide Offices) nearest you for more information on this subject.

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, add 20 mL of the 1 M HCl stock solution and 100 mL of the 3 mM DAP·HCl stock solution (see Section 3.3). Dilute to a final volume of 1 L.

1 . Lithium
2. Sodium
3. Ammonium
4. Potassium
5. Magnesium
6. Calcium



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

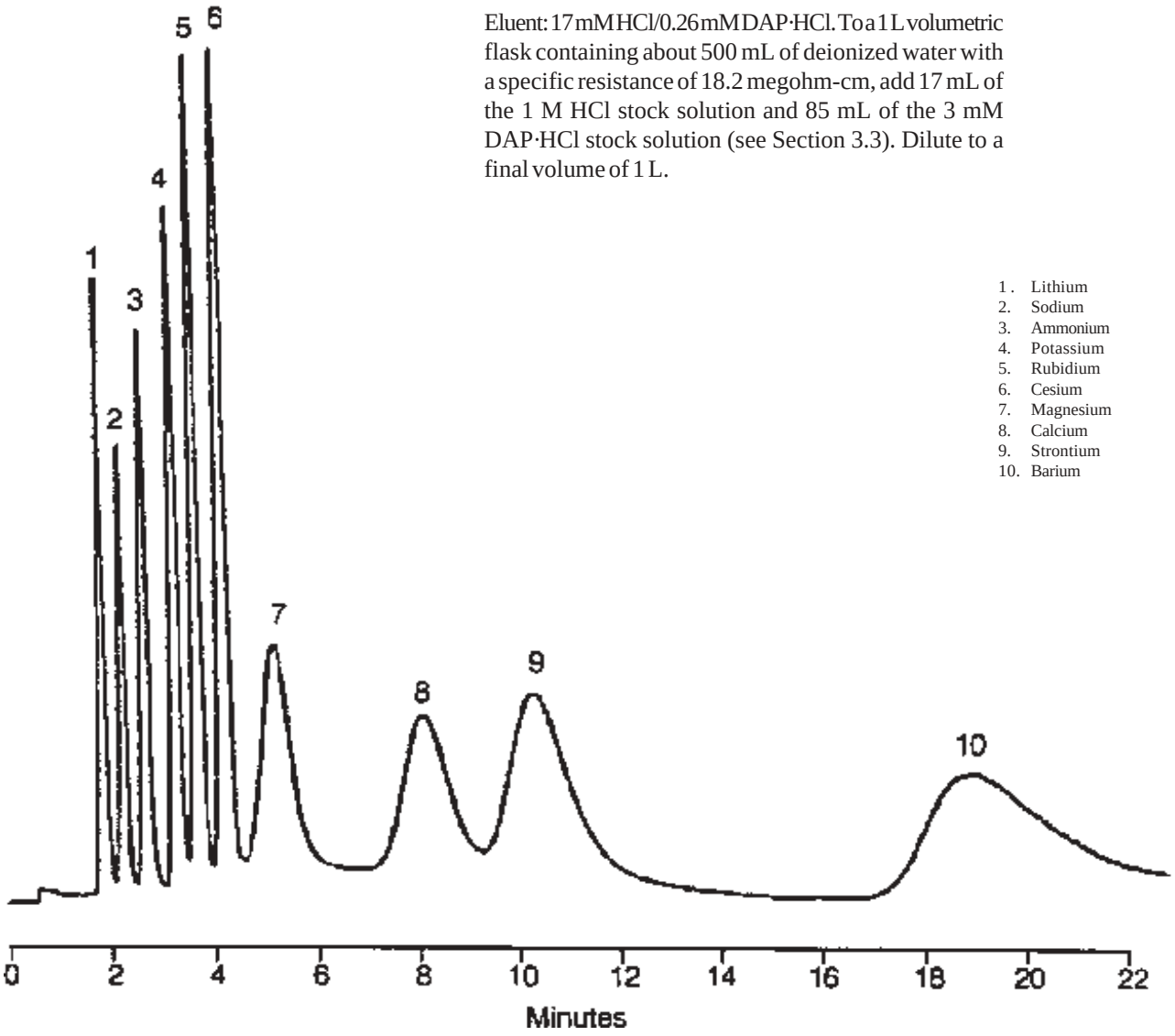
These 10 monovalent and divalent cations are determined isocratically with the IonPac Fast Cation I Analytical Column AND the IonPac Fast Cation II Analytical Column by using column switching. The eluent used is 17 mM HCl / 0.26 mM DAP·HCl.

A column switching valve and means of activating R are required to perform the column switching. The column switching valve is activated 40 seconds after the sample is injected. The column switching valve plumbing diagram is included in the IonPac Fast Cation II Analytical Column Product Manual (Document No. 032953). The IonPac Fast Cation II Analytical Column and the column switching valve are not included. They may be ordered from DIONEX: IonPac Fast Cation II Analytical Column (P/N 039633); 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 Regional Office (see DIONEX Worldwide Offices) nearest you for more information.

Eluent Preparation

Eluent: 17 mM HCl/0.26 mM DAP·HCl. To a 1 L volumetric flask containing about 500 mL of deionized water with a specific resistance of 18.2 megohm-cm, add 17 mL of the 1 M HCl stock solution and 85 mL of the 3 mM DAP·HCl stock solution (see Section 3.3). Dilute to a final volume of 1 L.



1. Lithium
2. Sodium
3. Ammonium
4. Potassium
5. Rubidium
6. Cesium
7. Magnesium
8. Calcium
9. Strontium
10. Barium

SECTION 5 - TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using the IonPac Fast Cation I Analytical Column. For more information on problems that originate with the Ion Chromatograph (IC) or the Cation MicroMembrane Suppressor (CMMS III, P/N 056752) refer to the Troubleshooting Guide in the appropriate Installation Instructions. If you cannot solve the problem on your own, call the DIONEX Regional Office nearest you (see DIONEX Worldwide Offices).

5.1 High Backpressure

Total system pressure, when using the IonPac Fast Cation I Analytical Column only, should be approximately 1000 psi. If it is higher than this, 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. Disconnect the analytical column from the CMMS III and measure the flow rate with a graduated cylinder and a stopwatch.

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 analytical or guard column might have particulates plugging the inlet bed support. The CMMS III or conductivity detector (CDM-II) might be plugged.

   To find out which part of the system is causing the problem, disconnect the pump eluent line from the injection valve and turn the pump on. Watch the pressure. It should not be more than 50 psi. Successively add the system components (injection valve, column, CMMS III and CDM-II) into the eluent flow path. Watch the pressure. The pressure should increase to approximately 1000 psi when the IonPac Fast Cation I Analytical Column is connected. The CMMS III will add approximately 100 psi. No other component should add more than 100 psi of pressure. Refer to the appropriate Product Manual for cleanup of the problem component.

C. If the IonPac Fast Cation I Analytical 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 R on against a bench top or other hard, flat surface to remove the bed support and sea[assembly. Discard the old assembly.

   4. Place a new bed support assembly (P/N 042310, consisting of Seal Washer, P/N 039835 and Bed Support (P/N 041375) into the end fitting (P/N 041353). Use the end of the column to carefully push the bed support assembly into the end fitting.

   5. Screw the end fitting back onto the column. Tighten it fingertight, then an additional 1/4 turn (25 in x lb). 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.

   6. 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 discussed in Section 3.1 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 a specific resistance of 18.2 megohm-cm.

D. Remove the analytical and guard columns from the system. Is the background still high?

E. To make sure it is not contaminated hardware that is causing the high background, use deionized water with a specific resistance of 18.2 megohm-cm as eluent. The background should be low (2 psi or less). Check the detector and conductivity cell by injecting deionized water with a specific resistance of 18.2 megohm-cm directly into ft.

F. 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 III REGEN OUT port. This flow rate should be greater than or equal to 5 mL/min.

2. Check the eluent flow rate. It should be 2.0 mL/min. Check the eluent flow rate after the analytical column using a graduated cylinder and a stopwatch.

3. Prepare fresh regenerant solution. If you are using an AutoRegen Accessory, pump about 200 mL of regenerant through the Cation AutoRegen Regenerant Cartridge (P/N 039563) to waste before recycling the regenerant back to the regenerant reservoir.

4. If the background is still high, bypass the Cation AutoRegen Regenerant Cartridge in the AutoRegen Accessory. If the background is now low, you probably need to replace the Cation AutoRegen Regenerant Cartridge (P/N 039563).

5. You may need to clean your CMMS III (P/N 056752). For instructions on how to do this, refer to the CMMS III Product Manual (Document No 031728). If the cleanup procedure does not work, you may have to replace the CMMS III.

5.3 Poor Peak Resolution

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

5.3.1 Loss of Column Efficiency

A. Check to see if headspace has developed in the analytical 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's inlet end fitting (see Section 5.1 Step C). If the resin does not fill the column body all the way to the top, it means that the resin bed has collapsed, creating a headspace. The column must be replaced.

B. Extra-column effects can result in sample band dispersion, making the peaks' elution less efficient. Make sure you are using 0.012" ID tubing, in all cases, between the injection valve and the detector cell inlet, and that the tubing lengths are minimized. Check for leaks.

5.3.2 Short Retention Times

A. Check to see if eluent flow rate is faster than 2.0 mL/min. Check the eluent flow rate after the analytical column using a graduated cylinder and a stopwatch.

B. Check to see if the eluent composition and concentration is correct. An eluent that 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.

C. 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 / 0.3 mM DAP·HCl) these take a very long time to elute.

5.4 Spurious Peaks

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

5.4.1 Sources of Column Contamination

A. 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. 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 Analytical Columns (See Section - 2, Caution).

C. 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 17.8 megohm-cm or greater.

D. The system should be as metal-free as possible. Gripper tube fittings are a potential source for metal contamination. Use DIONEX ThermoFlare fittings on all liquid lines in the eluent flow path. The eluent pump should be inspected periodically for signs of leaks.

E. Impurities may come from glass eluent bottles. Polyethylene 2 liter eluent containers (P/N 039163) are preferred.

F. Diluting the eluent will improve peak resolution but will also increase the analytes' retention times. 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 let it 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 any contaminant metals should have been eluted from the column.

5.4.2 Baseline Upsets

When an injection valve (or a column switching valve) is actuated, the possibility of creating a baseline disturbance exists. 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

retorqued (see system manual). Small baseline disturbances at the beginning or at the end of the chromatogram can be overlooked as long as they do not interfere with the quantitation of the peaks of interest.

If cleaning and retorquing the valve does not help, replace the valve. Use a DIONEX High Pressure Injection Valve (P/N 037142) or a DIONEX High Pressure Inert Valve (P/N 037143) as required.

SECTION 6 - COLUMN CARE

6.1 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 Analytical Columns is 2000 psi.

6.2 Column Start-Up

The column is shipped with eluent (see Section 4.1) as the storage solution. This eluent is the same one shown in the test chromatogram (20 mM HCl/0.3 mM DAP·HCl).

Prepare the eluent listed on the test chromatogram, install the column in the chromatography module and test the column performance under the conditions described in the test chromatogram. Let the column equilibrate with the eluent for a few minutes. Equilibration is complete when consecutive injections of the standard give reproducible retention times.

The column can be used with any of the other eluents discussed in Section 4 of this 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.

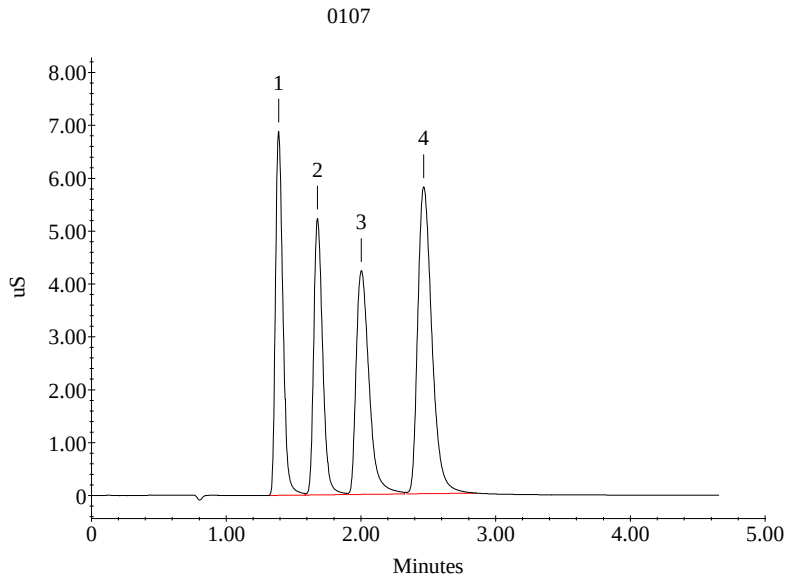
6.3 Column Storage

The column's storage solution should be the eluent used for the particular application (see Section 4).

If the column will not be used for one week or more, prepare it for long term storage. Flush the column for a few minutes with the eluent. Cap both ends securely, using the plugs supplied with the column.

IonPac® Fast Cation I
Analytical (4 x 250 mm)
Product No. SP5391

Serial No. : 0107 **Pressure (PSI) : 850** **Date : 9/8/00 3:29:34 PM**



Eluent: 20 mM HCl, 0.3 mM DL-2 3-diaminopropionic acid monohydrocloride

Flow Rate: 2.0 mL/min

Detection: Suppressed Conductivity at 30 µSFS

Injection Volume: 50 µL

Storage Solution: Eluent

Peak Information : Found Components

Peak No.	Retention Time	Name		Efficiency	Asymmetry (10%)	Resolution
1	1.39	Lithium	0.8	2973	1.4	2.54
2	1.68	Sodium	2.0	2896	1.6	2.18
3	2.00	Ammonium	3.0	2082	1.7	2.52
4	2.47	Potassium	6.0	2624	1.6	n/a

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