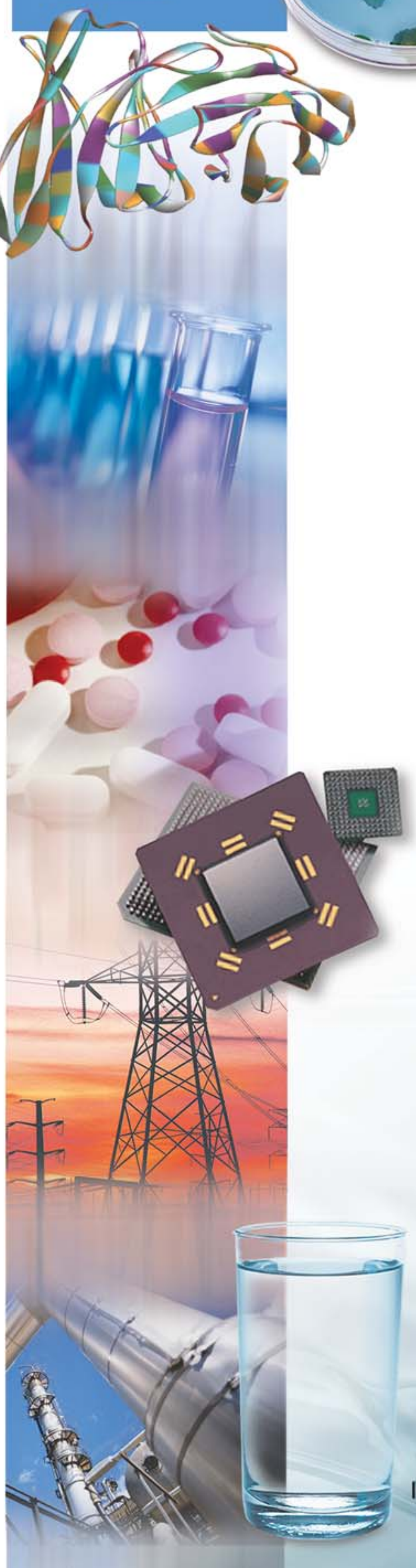

PRODUCT MANUAL

IonPac® Trace Cation Concentrator (TCC-2)

Now sold under the Thermo Scientific brand

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DIONEX

IC | HPLC | MS | EXTRACTION | PROCESS | AUTOMATION

PRODUCT MANUAL

for the

IONPAC® TRACE CATION CONCENTRATOR (TCC-2) COLUMN
(3 x 35 mm, 10-32 FERRULE FITTING)

(EACH, P/N 043103)

(4 PACK, P/N 043104)

Document No. 034466
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SECTION 1 - INTRODUCTION TO TRACE CATION CONCENTRATION

The IonPac® Trace Cation Concentrator (TCC-2) Column is designed primarily for high purity water analysis. The function of the TCC-2 is to strip ions from a measured volume of a relatively clean aqueous sample matrix. This process “concentrates” the desired analyte species onto the TCC-2 leading to a lowering of detection limits by 2–5 orders of magnitude. The unique advantage of the TCC-2 to the analytical chemist is the capability of performing routine trace analyses of sample matrix ions at mg/L levels without extensive and laborious sample pretreatment.

The TCC-2 is packed with a styrene/divinylbenzene copolymer that is surface sulfonated. The capacity of the TCC-2 is 10 µeq/column with a void volume of approximately 150 µL. The physical rigidity of this resin allows the TCC-2 to be used at pressures up to 4,000 psi. The TCC-2 can be readily converted between the acid and the salt form without significant changes in the operating pressure and is compatible with samples and eluents having up to 5% organic solvent. The recommended maximum flow rate is 3 mL/min.

CAUTION: Samples and eluents must contain less than 5% organic solvents.

This manual assumes that you are familiar with the installation and operation of the Dionex Ion Chromatograph (IC) and the Anion MicroMembrane Suppressor. If you do not understand the operation of the system, take the time to familiarize yourself with the various system components before beginning an analysis.

The TCC-2 has 10-32 threaded PEEK end fittings for use with ferrule/bolt liquid line fittings. If you find it necessary to install a component with 1/4-28 ports and therefore need to obtain or make one or more transition lines between 10-32 and 1/4-28 threaded ThermoFlare™ ports, Dionex recommends the use of Tefzel® liquid lines with a PEEK ferrule/bolt fitting on one end and a 1/4-28 ThermoFlare fitting on the other end. See - Dionex Liquid Line Fittings - for detailed instructions on purchasing or making these lines.

Always remember that assistance is available for any problem that may be encountered during the shipment or operation of DIONEX instrumentation and columns through the DIONEX North America Technical Call Center at 1-800-DIONEX-0 (1-800-346-6390) or through any of the DIONEX Offices listed in, “DIONEX Worldwide Offices.”

SECTION 2 - INSTALLATION

2.1 System Requirements for the IonPac Trace Cation Concentrator (TCC-2) Column

Figure 1 - Configuration for Determining Trace Levels of Cations, shows the generalized flow schematic for trace level cation chromatography.

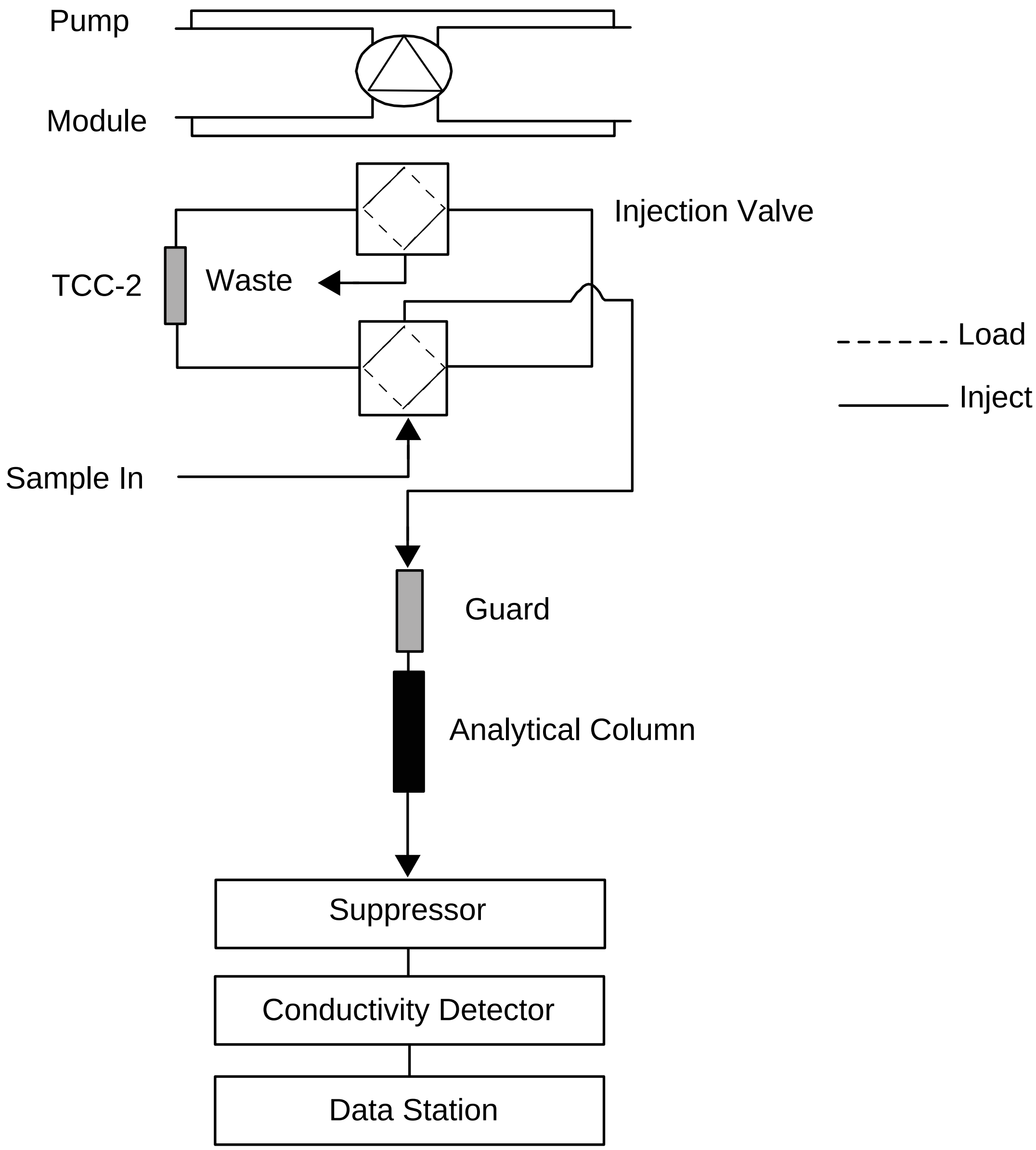
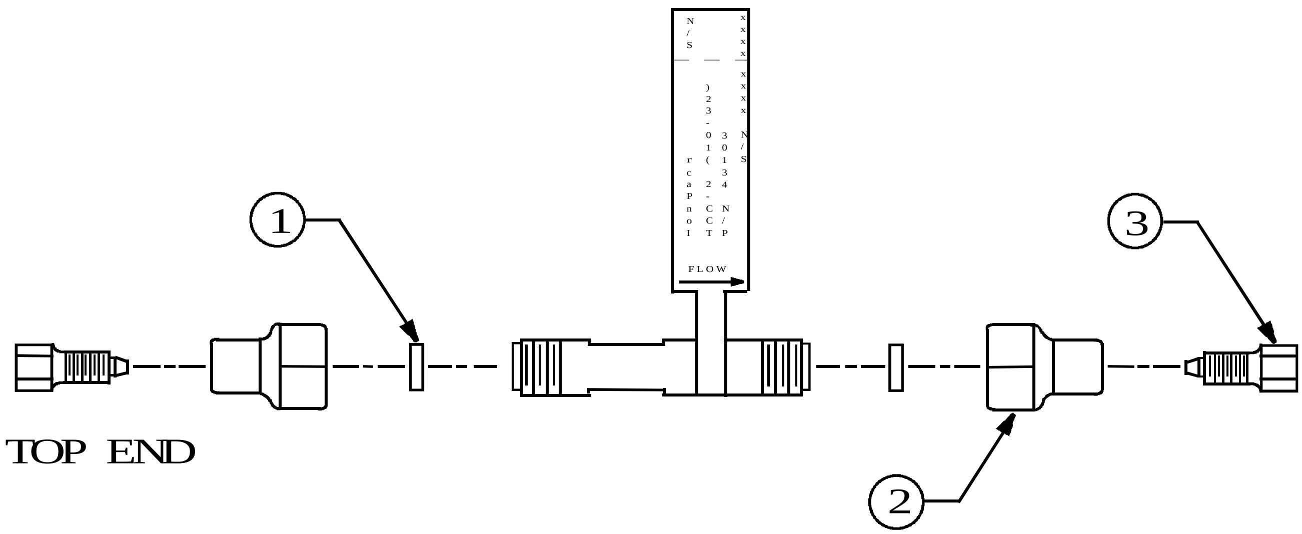


Figure 1
Configuration for Determining Trace Levels of Cations

SECTION 3 - OPERATION

3.1 Column Description

The IonPac Trace Cation Concentrator (TCC-2) Column consists of the following components:



1. Bed Support Assembly (P/N 042955)
2. 10-32 Ferrule Column End Fitting (P/N 042367)
3. 10-32 Ferrule Plug (P/N 042772)

Figure 2
Column Components

3.2 SAMPLE LOADING

Sample loading is performed with a separate positive displacement pump such as the Dionex DXP pump (P/N 043047). Pump flow rates of approximately 3 mL/min can be used while maintaining sample concentration efficiencies high enough to ensure good quantification. To prevent overloading of the TCC-2 and/or the loss of sample analytes, concentration linearity over the desired analytical concentration range should be determined (see Section 3.4.1 - Capacity Consideration of Concentrators).

The flow direction during the concentration step is critical. After the sample has been loaded onto the TCC-2 in the direction opposite to the eluent flow, it is then "backflushed" with eluent on to the guard and analytical columns (see Figure 3 - Loading the Trace Cation Concentrator (TCC-2) Column). This configuration concentrates the cations in a compact band at the bottom of the TCC-2. When injected, all of the ions are rapidly eluted off the TCC-2 and onto the guard and analytical columns. If the sample is loaded onto the TCC-2 in the same flow direction as the eluent flow, the cations are concentrated at the head of the column rather than at the bottom. When injected, the cations begin chromatographic separation on the concentrator before reaching the guard and analytical columns. Normally the function of the concentrator is to strip the ions of interest from the sample matrix and not to act as an analytical column. In order to ensure maximum system performance, it is recommended that concentration always be performed in a backflush manner.

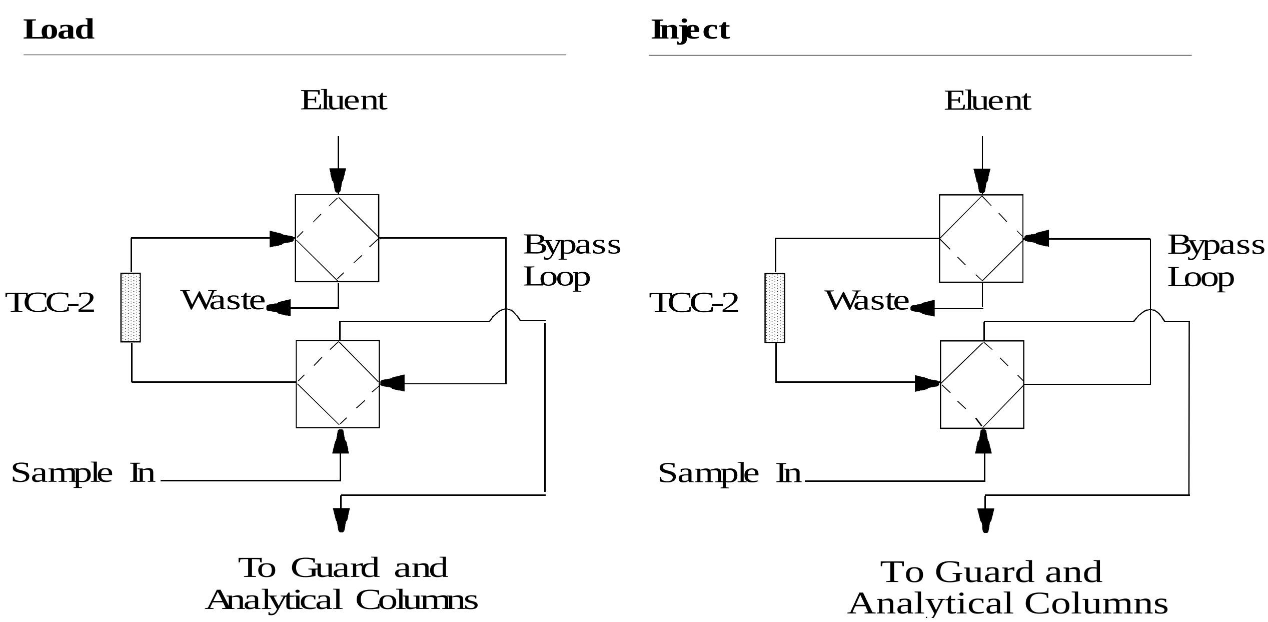


Figure 3
Loading the Trace Cation Concentrator (TCC-2) Column

3.3 REAGENT AND SAMPLE HANDLING

The use of the IonPac Trace Cation Concentrator (TCC-2) Column has certain limitations. At trace analyte concentration levels (µg/L), the results of analysis depend on carefully following good laboratory practices. All sources of contamination must be eliminated. The following sections focus on critical points that must be observed when using concentrator columns. Proper consideration of these points will enable the analyst to obtain accurate and reproducible results at trace analyte levels.

3.3.1 Water Quality

All water used in the preparation of standards and eluents must be deionized water with a specific resistance of 18.2 megohm-cm. The quality of the dilution water must be determined by Ion Chromatography since even deionized water with a specific resistance of 18.2 megohm-cm may contain trace levels of the ions of interest. To do this, analyze your water in exactly the same manner as you would your sample.

3.3.2 Sample Collection and Storage

At trace analyte concentration levels (µg/L), chances of contamination during collection or storage are high. Every container and every procedural step constitutes a potential source of contamination. Polystyrene containers with leak-tight caps can be used to store 1 to 5 µg/L levels of inorganic and organic cations for up to 8 days. Recommended storage vessels are Corning tissue culture flasks. The following procedure should be used for storage of µg/L level samples:

a) Rinse the polystyrene container and cap twice with deionized water having a specific resistance of 18.2 megohm-cm. Fill the container until it overflows, cap it securely, and soak for 4 hours.

b) Empty the container and refill it with deionized water having a specific resistance of 18.2 megohm-cm. Cap the container securely. It should remain filled at least 24 hours before sample collection.

c) Empty the container and rinse it twice with the sample to be collected. Fill the container with the sample until it overflows and then cap the container securely. Be sure that the sample line does not touch the container.

NOTE: ***Never use plastic syringes with rubber pistons for any loading of trace ions. These materials cause non-reproducible results.***

3.3.3 Standards

It is good practice to run standards at the beginning, middle, and end of each day to ensure constant instrument response. Because external standard quantification is used, it is critical that standard solutions are correctly prepared.

a) 1,000 mg/L stock standard solutions should be prepared by accurately weighing amounts of salts as described in your instrument manual. These solutions are stable over a period of several months.

b) 1 mg/L stock standard solutions may be prepared by diluting 1 mL of 1,000 ppm stock standard to 1,000 mL in a volumetric flask. These solutions should then be transferred to clean polystyrene containers. They may be stored for one month.

c) 1 µg/L working standard solutions may be prepared by diluting 1 mL of the 1 mg/L stock standard to 1,000 mL. These working standards are stored in polystyrene containers. They are stable up to 8 days but it is recommended that they be prepared daily since standard response is critical in the results of your analysis.

3.4 CONCENTRATOR CAPACITY

3.4.1 Capacity Considerations for Concentrators

As in all ion exchange systems, the resin has a finite capacity. It can strip a given amount of ions from water. When the capacity of the concentrator is exceeded, the stripping will not be quantitative. This condition is referred to as column overload.

When estimating the capacity of a concentrator, one must remember that the column is used in a dynamic state where the liquid containing the analytes is flowing over the resin at a finite rate. This reduces the capacity somewhat since the analyte ions have less time to interact with the resin surface.

Low concentrator column capacity creates the following practical implications:

a) Trace analysis of an analyte is difficult in the presence of µg/L concentrations of species which exhibit higher or similar affinities for the resin. If the dynamic column capacity is exceeded, high affinity ions will displace the analytes on the ion exchange sites and result in their elution to waste during the loading process.

b) Conversely, qualitative analysis of ions with higher affinities for the resin in the presence of high concentrations of ions with low affinities is possible. Again, the key to successful analysis is that the ionic content of the high affinity ion to be quantitated may not exceed the effective column capacity.

c) Do not dilute samples to be concentrated in eluent because the eluent ions elute the ions of interest.

d) A plot of response versus concentration should be generated as in Figure 4 - Linearity of Ammonium Concentrator Injection, for the determination of the maximum amount of sample or standard that can be quantitatively loaded. In Figure 4 - Linearity of Ammonium Concentrator Injection, the break in the curve where linearity starts to change is at a concentration volume of 10 mL of 1 mg/L ammonium. For practical purposes the amount concentrated for a series of samples should be 75% of this value. This will ensure that there is a safety margin built into the concentration process in case a sample in a series of concentration experiments has a slightly higher ionic concentration.

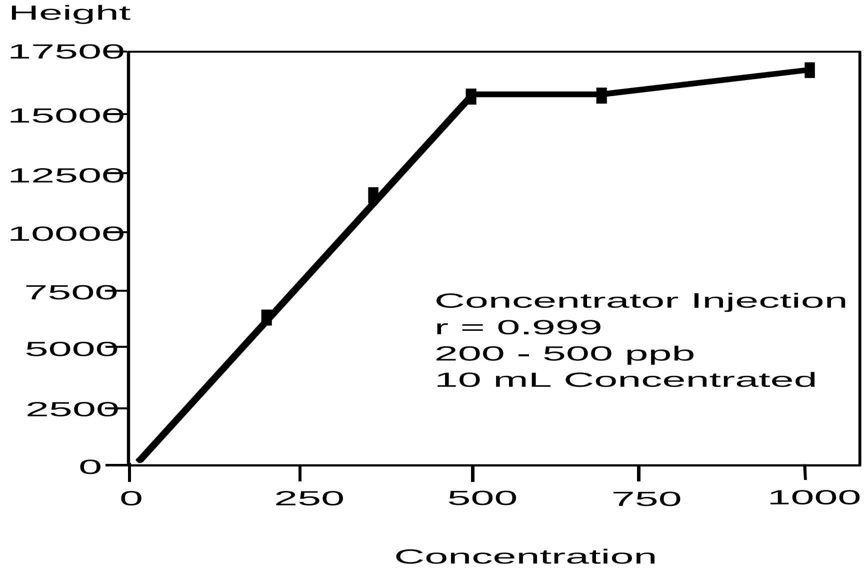


Figure 4
Linearity of Ammonium Concentrator Injection

3.4.2 Determination of Concentrator Column Breakthrough Volume

The breakthrough volume of an analyte ion is that volume of sample which causes an ion of interest to be eluted from, rather than retained or concentrated on, the concentrator column.

The breakthrough is dependent upon the following:

- The volume of sample loaded
- The rate at which the sample is loaded
- The pH of the sample
- The ionic strength of the sample
- The amount and capacity of resin in the column

The breakthrough volume is determined as follows:

a) Prepare 1 L of a solution that closely simulates the type of sample to be analyzed. For example, if the sample contains high levels of ammonia, the simulated sample should also contain ammonia. Ammonia in solution exists as ammonium hydroxide ions. The resulting ammonium ion will act as an eluent.

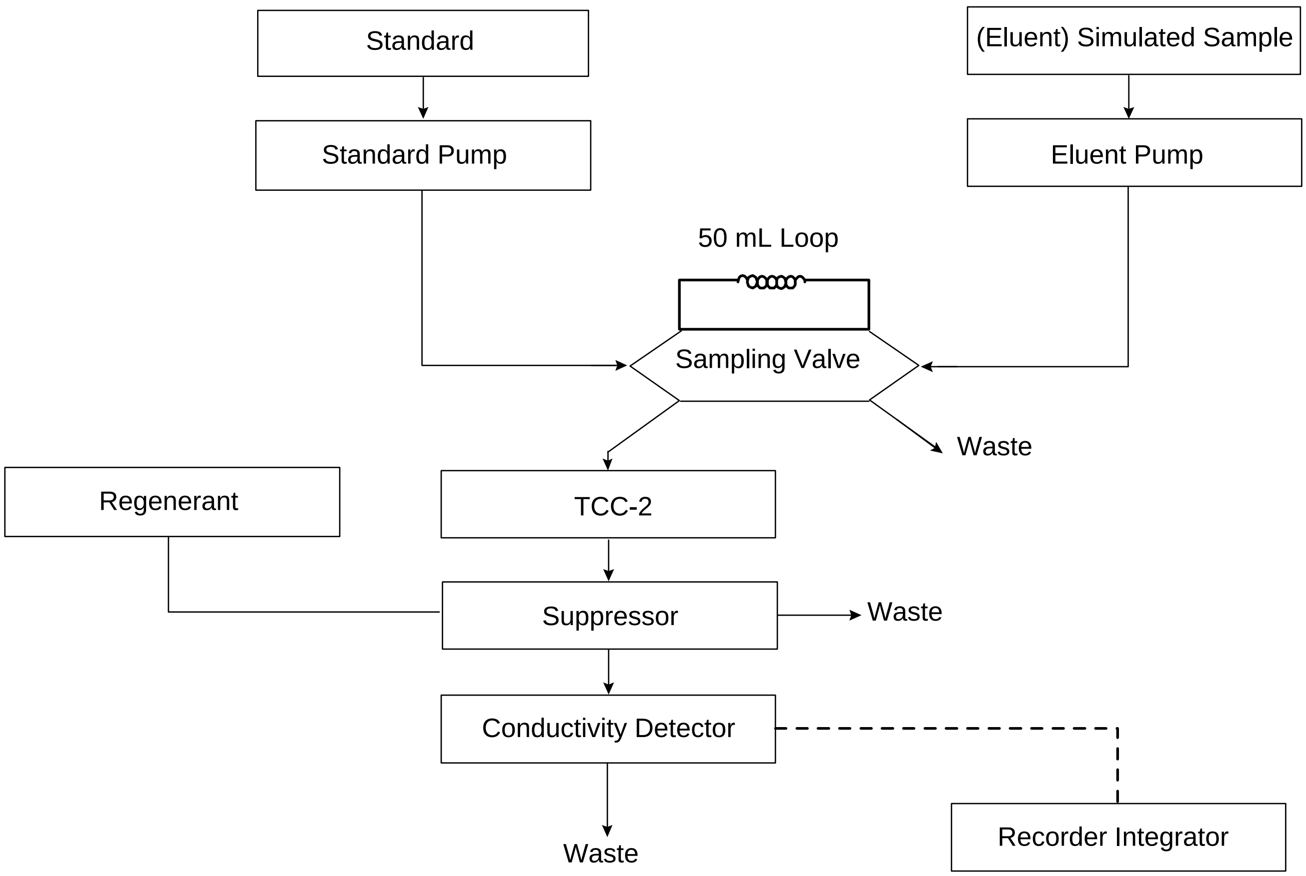
b) Prepare a 1 mg/L standard of the first eluting ion of interest (e.g., Li).

c) Set up the Ion Chromatograph, as shown in Figure 5 - Determination of the Breakthrough Volume, and equilibrate the concentrator column with eluent at the concentration flow rate needed to achieve a stable baseline.

d) Switch to the simulated sample as an eluent and manually inject a 50 µL portion of the 1 mg/L standard.

e) Record the resulting chromatogram and calculate the breakthrough volume, as shown in Figure 5 - Determination of the Breakthrough Volume.

f) For practical purposes, the volume concentrated should below 75% of the breakthrough volume.



1. Flush concentrator column with eluent.
2. Load 50 µL loop with 1 mg/L standard of first eluting ion of interest.
3. Switch from eluent to simulated sample and inject 50 µL of standard.
4. Determine breakthrough volume as follows:

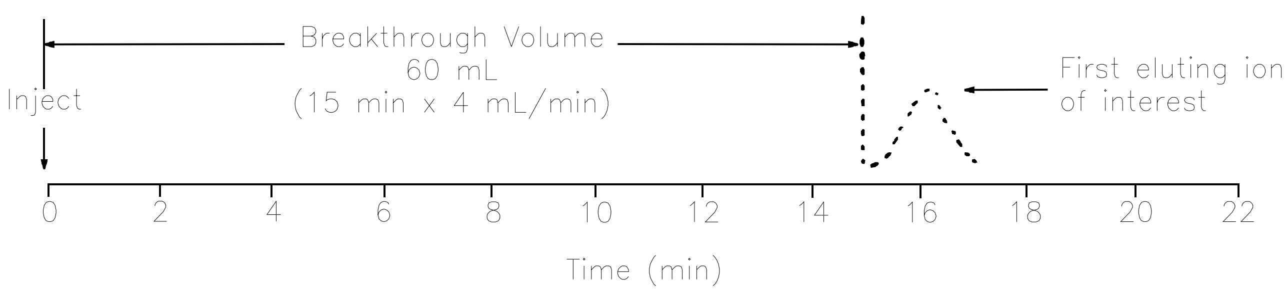


Figure 5
Determination of the Breakthrough Volume

SECTION 4 - TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using the IonPac Trace Cation Concentrator (TCC-2) Column. For more information on problems that originate with the Ion Chromatograph, refer to the Troubleshooting Guide in the appropriate operator's manual. If you cannot solve the problem on your own, call the Dionex Regional Office nearest you (see - Dionex Worldwide Offices).

4.1 High Backpressure from a Contaminated Inlet Bed Support

If the IonPac Trace Cation Concentrator (TCC-2) Column displays high backpressure, the bed support in the column inlet may be contaminated. Follow the instructions below to change the bed support assembly using one of the two spare bed support assemblies included in the ship kit provided with the column.

a) Disconnect the column from the system.

b) Using two open-end wrenches, carefully unscrew the inlet (top) column end fitting.

c) Turn the end fitting over and tap it against a benchtop or other hard, flat surface to remove the bed support and seal 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 you **DO NOT SCRATCH THE WALLS OF THE END FITTING.** Discard the old assembly.

d) Place a new bed support assembly in the end fitting. Use the end of the column to carefully start the bed support assembly into the end fitting.

	P/N
Bed Support Assembly	042955
End Fitting (10-32 ferrule type)	042367

e) Screw the end fitting back onto the column. Tighten it fingertight and then using two open-end wrenches, tighten it 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 cl*****ean before screwing the end fitting back onto the column.**

f) Reconnect the column to the system and resume operation.

4.2 High Background, Noise or Baseline Instability

Normally, problems such as high background, noise or baseline instability will not be attributable to the IonPac Trace Cation Concentrator (TCC-2) Column. These problems usually originate in either the analytical column or the post-column detection chemistry. Before checking the TCC-2 as the source of system background noise, consult the appropriate troubleshooting sections in the analytical column Product Manual, the Ion Chromatograph Operator's Manual and the detector manual.

If the source of the high background noise is isolated to the TCC-2, then proceed with the following steps:

a) Make sure that the eluents and regenerant are correctly formulated.

b) Make sure that the eluents are made from chemicals with the recommended purity (see Section 3 - Operation).

c) Make sure that the deionized water used to prepare the reagents has a specific resistance of 18.2 megohm-cm.

4.3 Poor Peak Shape

In some instances, poor peak shape in Ion Chromatography may be caused by a contaminated TCC-2. To clean the TCC-2, see - Column Cleanup of Polyvalent Cations and Acid-Soluble Contaminants, (See "Column Care").

APPENDIX A - COLUMN CARE

A.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 Trace Cation Concentrator Column (TCC-2) is 4,000 psi.

A.2 Column Start-up

The column is shipped with 4 mM HCl as the storage solution. Flush the column for 30 minutes with eluent before attempting to concentrate sample.

A.3 Column Storage

The TCC-2 should be stored in the acid form. Flush approximately 5 mL of any typical HCl or HCl·DAP cation eluent through the TCC-2.

A.4 Column Cleanup of Polyvalent Cations and Acid-soluble Contaminants

a) Prepare a 500 mL solution of 1 M HCl/0.1 M KCl.

b) Disconnect the guard, analytical columns and the suppressor from the injection valve and the Conductivity Detector. Disconnect the Gradient Mixer or Cation Trap Column (CTC-1) from the gradient pump. Connect the Trace Cation Concentrator (TCC-2) Column directly to the gradient pump. Direct the effluent from the TCC-2 directly to a waste container.

c) Set the flow rate to 1 mL/min.

d) Pump the 1 M HCl/0.1 M KCl solution through the column for 15-30 minutes.

e) Equilibrate the TCC-2 with eluent for 15 minutes at 1 mL/min before resuming normal operation.

f) Reconnect the cation guard, analytical column and the suppressor between the injection valve and the Conductivity Detector. Reconnect the Gradient Mixer or Cation Trap Column (CTC-1) between the gradient pump and the Injection Valve. Resume operation.

A.5 Column Cleanup of Organic/Cationic Contaminants

CAUTION: Samples and eluents must contain less than 5% organic solvents.

a) Prepare a 500 mL solution of 0.1 M HCl/5% CH_3OH.

b) Disconnect the guard, analytical columns and the suppressor from the injection valve and the Conductivity Detector. Disconnect the Gradient Mixer or Cation Trap Column (CTC-1) from the gradient pump. Connect the Trace Cation Concentrator (TCC-2) Column directly to the gradient pump. Direct the effluent from the TCC-2 directly to a waste container.

c) Set the flow rate to 1 mL/min.

d) Pump the 0.1 M HCl/5% CH_3OH solution through the column for 15-30 minutes.

e) Equilibrate the TCC-2 with eluent for 15 minutes at 1 mL/min before resuming normal operation.

f) Reconnect the cation guard, analytical column and the suppressor between the injection valve and the Conductivity Detector. Reconnect the Gradient Mixer or Cation Trap Column (CTC-1) between the gradient pump and the Injection Valve. Resume operation.

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