PRODUCT MANUAL

IonPac® TRACE ANION CONCENTRATOR (TAC-2) COLUMN

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PRODUCT MANUAL

IONPAC® TRACE ANION CONCENTRATOR (TAC-2) COLUMN

(3 x 35 mm, 10-32 Ferrule Fittings)

(P/N 043101)
(P/N 043102 Pack of 4)

Document No. 034467
Revision 06
18 November 2004

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SECTION 1 - INTRODUCTION

The IonPac® Trace Anion Concentrator (TAC-2) Column is designed primarily for high purity water analysis. The function of the TAC-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 TAC-2 leading to a lowering of detection limits by 2-5 orders of magnitude. The unique advantage of the TAC-2 to the analytical chemist is the capability of performing routine trace analyses of sample matrix ions at µg/L levels without extensive and laborious sample pretreatment.

The TAC-2 is packed with a styrene-divinylbenzene copolymer that is agglomerated with Alkyl quaternary ammonium functionalized latex. The capacity of the TAC-2 is 3.4 µeq/column with a void volume of approximately 50 µL. The physical rigidity of this resin allows the TAC-2 to be used at pressures up to 4,000 psi. The TAC-2 can be readily converted between the hydroxide and the salt form without significant changes in the operating pressure and is compatible with samples having up to 5% organic solvent. The recommended maximum flow rate is 3 mL/min.

Table 1
IonPac TAC-2 Concentrator Column Packing Specifications

Column	Partical Diameter µm	Substrate X-Linking %	Latex Diameter nm	Latex X-Linking %	Column Capacity µeq/column	Functional Group	Hydrophoicity
IonPac TAC-2 Concentrator	30	2	250	5	3.4	Alkyl quaternary ammonium	Medium

Table 2
IonPac TAC-2 Concentrator Column Operating Parameters

Column	Typical Back Preasure psi (Mpa) at 30 °C	Standard Flow Rate mL/min	Maximum Flow Rate mL/min	Maximum Operational Preasure psi (Mpa)
IonPac TAC-2 3 x 35 mm Column	65 (0.45)	1	3	4,000

NOTE: ***Always remember, assistance is available for any problem 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.

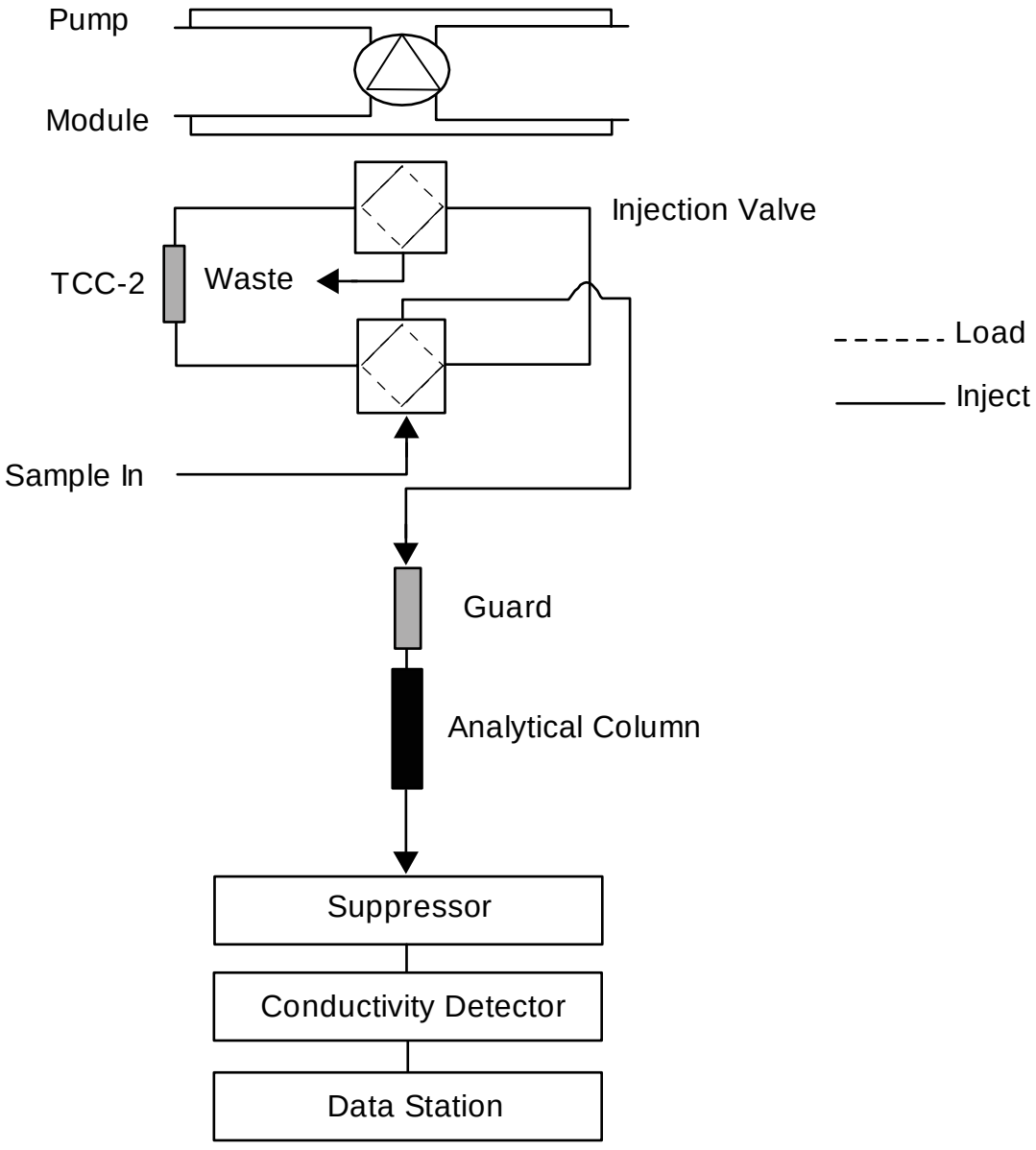

Figure 1
Configuration for Determining Trace Levels of Cations

2.2 Column Description

The IonPac Trace Anion Concentrator (TAC-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)

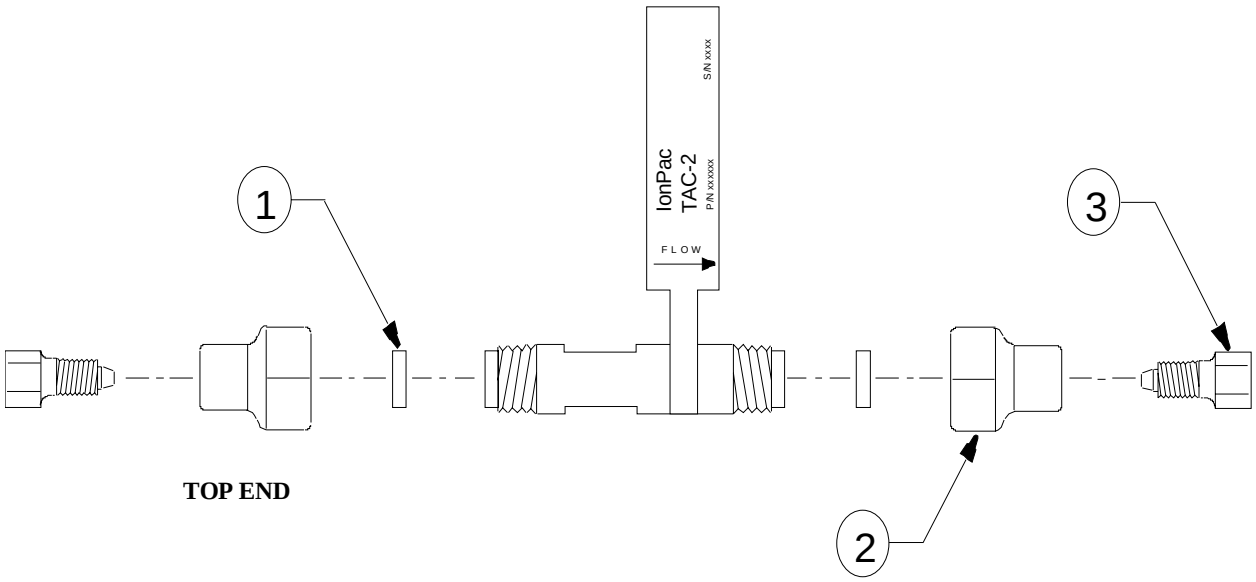

Figure 2
Column Components

SECTION 3. OPERATION

3.1 Sample Loading

Sample loading is performed with a separate positive displacement pump such as the DIONEX DQP pump (P/N 035250). Pump flow rates of approximately 3 mL/min may be used while maintaining sample concentration efficiencies high enough to ensure good quantitation. To prevent overloading of the TAC-2 and/or the loss of sample analytes, concentration linearity over the desired analytical concentration range should be determined (see Section 3.4.1).

The flow direction during the concentration step is critical. After the sample has been loaded onto the TAC-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). This configuration concentrates the cations in a compact band at the bottom of the TAC-2. When injected, all of the ions are rapidly eluted off the TAC-2 and onto the guard and analytical columns. If the sample is loaded onto the TAC-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.

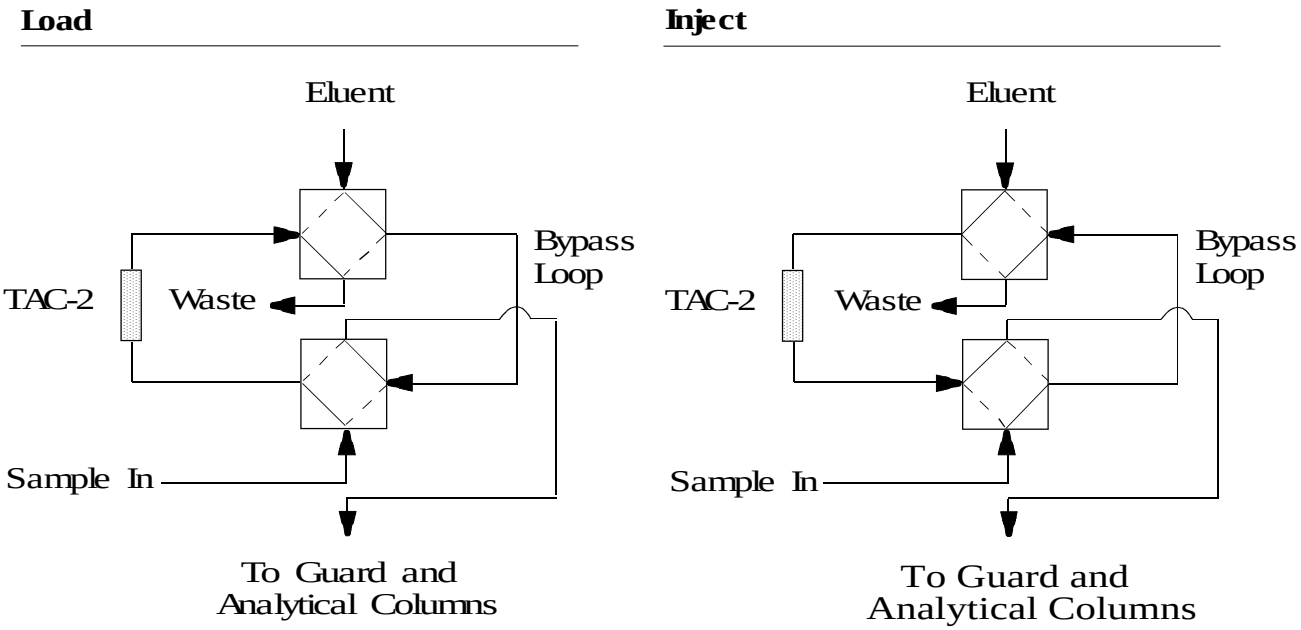

Figure 3
Loading the IonPac Trace Anion Concentrator (TAC-2) Column

3.2 Reagent and Sample Handling

The use of the IonPac Trace Anion Concentrator (TAC-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 which 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.

3.2.1 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.

3.2.2 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 may be used to store 1 to 5 µg/L levels of inorganic and organic anions 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.

B. Fill the container until it overflows, cap it securely, and soak for 4 hours.

C. Empty the container and refill it with deionized water having a specific resistance of 18.2 megohm-cm.

D. Cap the container securely. It should remain filled at least 24 hours before sample collection.

E. Empty the container and rinse it twice with the sample to be collected.

F. Fill the container with the sample until it overflows and then cap the container securely. Be sure 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.***

3.2.3 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 quantitation 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. These 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 these be prepared daily since standard response is critical in the results of your analysis.

3.3 Concentrator Capacity

3.3.1 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 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 (e.g. SO_4^{2-}, NO_3^-, S^{2-}, OH^-, etc.). 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.

NOTE: ***At higher concentrations, all ions which are ion-exchanged on the resin may be potential sources of interference. This includes ions which are not detected by the conductivity detector (e.g., S^2, OH^-, CN^-). The dynamic column capacity should serve as a guideline in evaluating potential interferences.***

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 (e.g., analysis of SO_4^{2-} in the presence of high concentrations of Cl^-). Again, the key to successful analysis is 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 volume concentrated should be generated as in Figure 4 for determination of the maximum amount of sample or standards that can be quantitatively loaded. The break in the curve where linearity starts to change is 100 mL concentrated. 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 for samples in a series having slightly higher ionic concentrations.

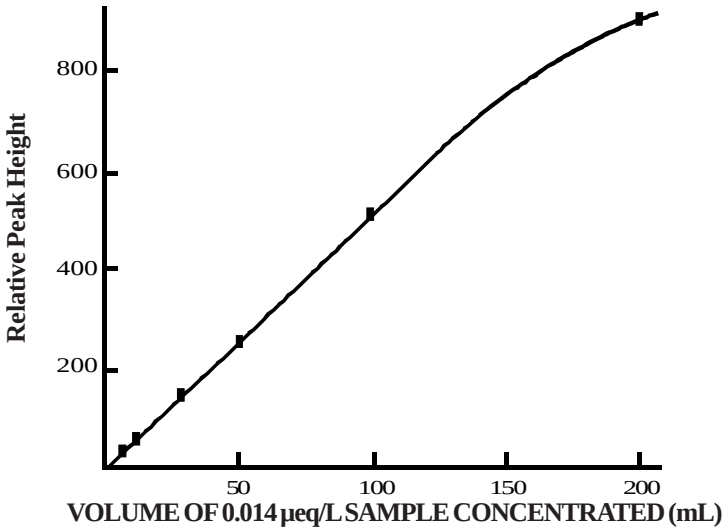

Figure 4
Response versus Volume Concentrated

3.3.2 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:

A. The volume of sample loaded

B. The rate at which the sample is loaded

C. The pH of the sample

D. The ionic strength of the sample

E. The amount and capacity of resin in the column

3.3.2.1 The breakthrough volume is determined as follows:

A. Prepare 1 L of a solution which closely simulates the 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., Cl^-).

C. Set up the Ion Chromatograph, as shown in Figure 5, 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 6.

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

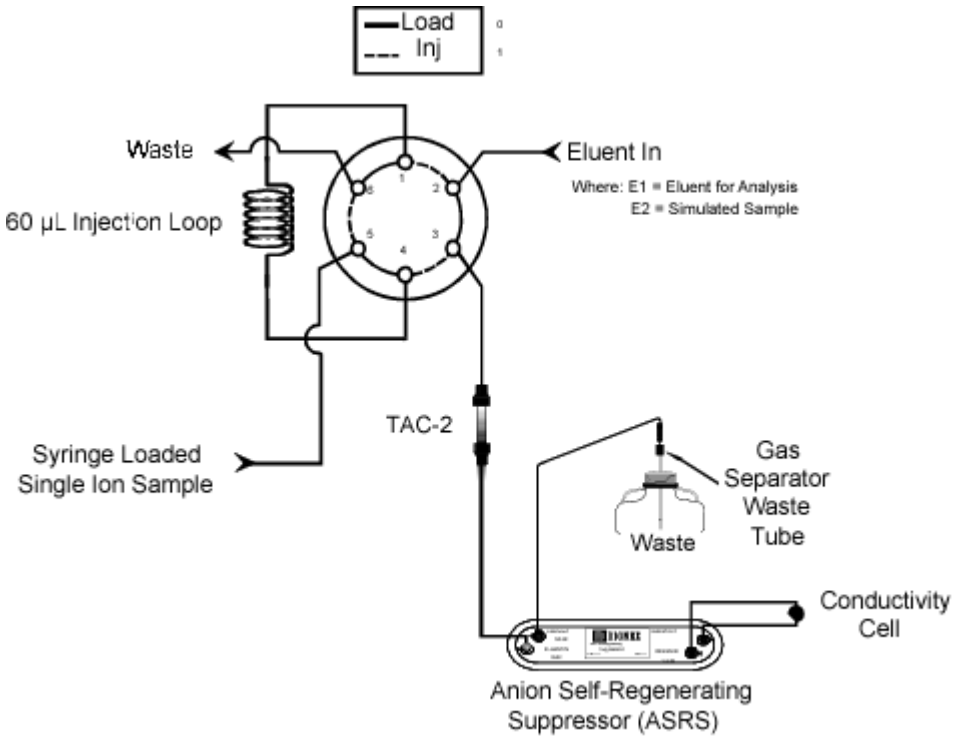

Figure 5
Instrument Setup for the Determination of the Breakthrough Volume

1. Flush concentrator column (TAC-2) 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:

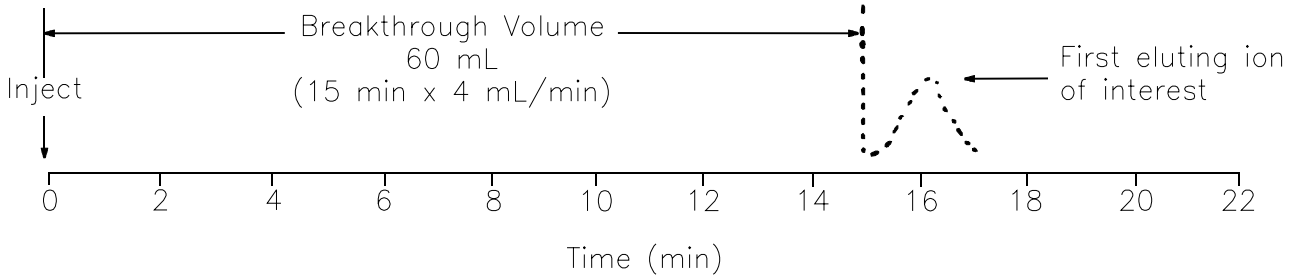

Figure 6
Determination of the Breakthrough Volume

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 Anion Concentrator (TAC-2) Column. For more information on problems which 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.

4.1 High Backpressure from a Contaminated Inlet Bed Support

If the IonPac Trace Anion Concentrator (TAC-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 bench top 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 you **DO NOT SCRATCH THE WALLS OR SURFACES OF THE END FITTING.** Discard the old assembly.

D. Place a new bed support assembly (PN 042955) in the end fitting (PN 042367). Use the end of the column to carefully start the bed support assembly into the end fitting.

E. Screw the end fitting back onto the column. Tighten it finger tight and then using two open-end wrenches, tighten it 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 the washer and the end of the column are clean 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 Anion Concentrator (TAC-2) Column. These problems usually originate in either the analytical column or the post-column detection chemistry. Before checking the TAC-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 TAC-2, then proceed with the following steps:

A. Make sure the eluents and regenerant are correctly formulated.

B. Make sure the eluents are made from chemicals with the recommended purity (see Section 3).

C. Make sure 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 may be caused by a contaminated TAC-2. To clean the TAC-2, see Appendix A, "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 Anion Concentrator (TAC-2) Column is 4,000 psi.

A.2 Column Start-up

The column is shipped with 0.1 M NaOH as the storage solution.

A.3 Column Storage

The IonPac Trace Anion Concentrator (TAC-2) Column should be stored in the hydroxide form.

A.4 Column Cleanup

A.4.1 Polyvalent Anions and Base-soluble Contaminants

A. Prepare a 500 mL solution of 0.5 M NaOH.

B. Disconnect the guard, analytical columns and the suppressor from the injection valve and the Conductivity Detector. Disconnect the Gradient Mixer or Anion Trap Column (ATC) from the gradient pump. Connect the IonPac Trace Anion Concentrator (TAC-2) Column directly to the gradient pump. Direct the effluent from the TAC-2 directly to a waste container.

C. Set the flow rate to 1 mL/min.

D. Pump the 0.5 M NaOH solution through the column for 15-30 minutes.

E. Using eluent, equilibrate the TAC-2 for 15 minutes at 1 mL/min before resuming normal operation.

F. Reconnect the guard, analytical column and the suppressor between the injection valve and the Conductivity Detector. Reconnect the Gradient Mixer or Anion Trap Column (ATC) between the gradient pump and the Injection Valve. Resume operation.

A.4.2 Organic/Anionic Contaminants

A. Prepare a 500 mL solution of 0.1 M NaOH and 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 Anion Trap Column (ATC) from the gradient pump. Connect the IonPac Trace Anion Concentrator (TAC-2) Column directly to the gradient pump. Direct the effluent from the TAC-2 directly to a waste container.

C. Set the flow rate to 1 mL/min.

D. Pump the 0.1 M NaOH/5% CH_3OH solution through the column for 15-30 minutes.

E. Using eluent, equilibrate the TAC-2 for 15 minutes at 1 mL/min before resuming normal operation.

F. Reconnect the guard, analytical column and the suppressor between the injection valve and the Conductivity Detector. Reconnect the Gradient Mixer or Anion Trap Column (ATC) between the gradient pump and the Injection Valve. Resume operation.

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LPN XXXXX 10M 09/04
Printed in U.S.A.

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