PRODUCT MANUAL

IONPAC® TAC-LP1 LOW PRESSURE TRACE ANION CONCENTRATOR COLUMN

(4 x 35 mm, P/N 046026)

IONPAC® TAC-ULP1 ULTRA LOW PRESSURE TRACE ANION CONCENTRATOR COLUMN

(5 x 23 mm, P/N 061400)

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

1. See Section 3, "Operation," for operation instructions.
2. See Section 4, "Example Applications."
3. See Section 5, "Troubleshooting Guide," for problem analysis and operation corrections.
4. See "Appendix A - Column Care," for column cleanup and long-term storage recommendations.

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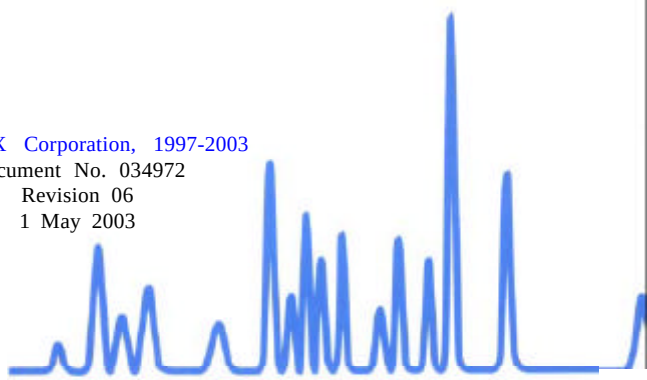

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SECTION 1 - INTRODUCTION

The IonPac® Low Pressure Trace Anion Concentrator (TAC-LP1) and the IonPac TAC-ULP1 Ultra Low Pressure Trace Anion Concentrator (TAC-ULP1) Columns are designed primarily for high purity water analysis. The function of the TAC-LP1 or TAC-ULP1 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-LP1 or TAC-ULP1 leading to a lowering of detection limits by 2-5 orders of magnitude. The unique advantage of the TAC-LP1 or TAC-ULP1 to the analytical chemist is the capability of performing routine Low Pressure Trace analyses of sample matrix ions at µg/L levels without extensive and laborious sample pretreatment.

The TAC-LP1 and TAC-ULP1 are packed with a 18 µm styrene/divinylbenzene copolymer that is agglomerated with an anion exchange latex that has been completely aminated. The latex has a polyvinylbenzyl backbone and carries the actual ion exchange sites. Due to the highly cross-linked structure, the resin is solvent compatible. The TAC-ULP1 uses column hardware with a wider internal diameter of 5-mm and a shorter length, thereby decreasing the column backpressure but maintaining the column capacity. The capacity of the TAC-LP1 and TAC-ULP1 is 25 µeq/column with a void volume of approximately 145 µL. The physical rigidity of this resin allows the TAC-LP1 or TAC-ULP1 to be used at pressures up to 3,000 psi. The TAC-LP1 or TAC-ULP1 can be readily converted between the base and the salt form without significant changes in the operating pressure.

The recommended maximum flow rate is 3 mL/min. The back pressure generated by the TAC-LP1 is less than 60 psi at 2.0 mL/min and the TAC-ULP1 is 30 psi at 2.0 mL/min. The large resin particle size (18 µm) in the TAC-LP1 and TAC-ULP1 make it possible to do manual injections onto the concentrator. Syringes with up to 3 mL capacities can be used to manually push samples through the TAC-LP1 or TAC-ULP1. It takes approximately 1 minute to manually push 3 mL of sample through the TAC-LP1 or TAC-ULP1.

The TAC-LP1 and TAC-ULP1 can be used with hydroxide and carbonate eluents, with or without solvent, to concentrate samples on either 4-mm or 2-mm analytical systems.

This manual assumes that you are familiar with the installation and operation of the Dionex Ion Chromatograph (IC) and the 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 TAC-LP1 and TAC-ULP1 have 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."

Table 1
IonPac TAC-LP1 and TAC-ULP1 Trap Column Packing Specifications

Column	Particle Diameter µm	Substrate X-Linking %	Latex Diameter nm	Latex X-Linking %	Column Capacity µeq/column	Functional Group	Hydrophobicity
TAC-LP1 4-mm	18	55	85	6	25	Alkanol quaternary ammonium	Very Low
TAC-ULP1 5-mm	18	55	85	6	25	Alkanol quaternary ammonium	Very Low

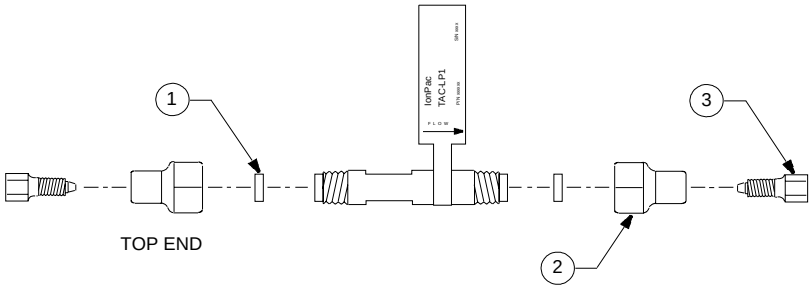
Table 2
IonPac TAC-LP1 and TAC-ULP1 Trap Column Operating Parameters

Column	Typical Back Pressure psi (MPa) at 30°C	Standard Flow Rate mL/min	Maximum Flow Rate mL/min
TAC-LP1 Trap Column 4-mm	≤ 60 (0.21)	1.0	3.0
TAC-ULP1 Trap Column 5-mm	≤ 30 (0.21)	1.0	3.0

SECTION 2 - INSTALLATION

2.1 Column Description

The IonPac Low Pressure Trace Anion Concentrator (TAC-LP1) and the IonPac TAC-ULP1 Ultra Low Pressure Trace Anion Concentrator (TAC-ULP1) Columns consists of the following components:



1. Bed Support Assembly (P/N 042955)
2. 10-32 Ferrule Column End Fitting (P/N 052809)
3. 10-32 Ferrule Plug (P/N 042772)

Figure 1
TAC-LP1 and TAC-ULP1 Column Components

SECTION 3 - OPERATION

3.1 Sample Loading

Sample loading is performed either manually with a ≤ 3 mL syringe (it takes approximately 1 minute to inject 3 mL of sample through the TAC-LP1 or TAC-ULP1) or with a separate positive displacement pump such as the Dionex DQP pump (P/N 035250). 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 TAC-LP1 or TAC-ULP1and/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 TAC-LP1 or TAC-ULP1in the direction opposite to the eluent flow, it is then "backflushed" with eluent onto the guard and analytical columns (see Figures 2 and 3, "Loading the TAC-LP1 or TAC-ULP1 Column"). This configuration concentrates the anions in a tight band at the bottom of the TAC-LP1or TAC-ULP1. When injected, all of the ions are rapidly eluted off the TAC-LP1or TAC-ULP1 and onto the guard and analytical columns. If the sample is loaded onto the TAC-LP1or TAC-ULP1 in the same flow direction as the eluent flow, the anions are concentrated at the head of the column rather than at the bottom. When injected, the anions 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 2 illustrates the configuration for sample loading using a Rheodyne valve.

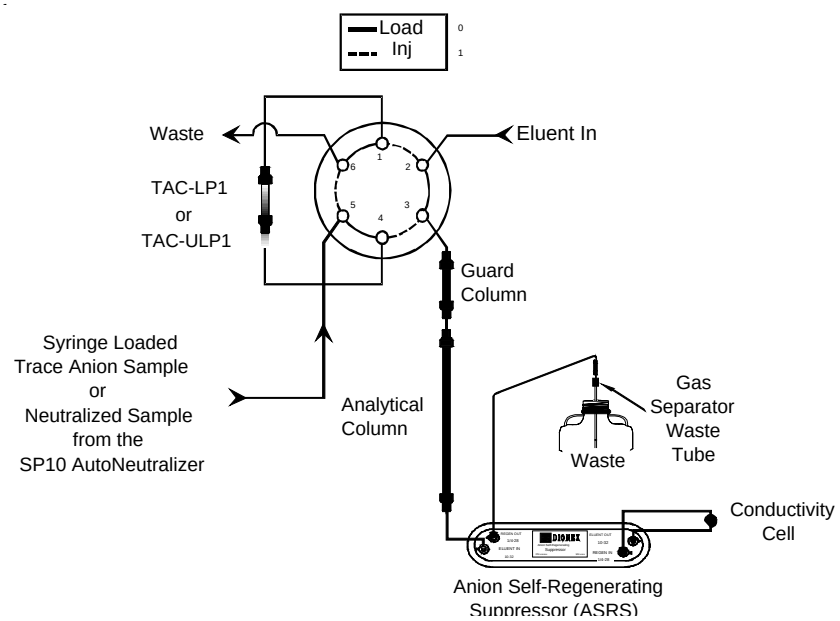


Figure 2
Configuration for Determining Trace Levels of Anions

Figure 3 illistrates the configuration for sample loading using a slider valve.

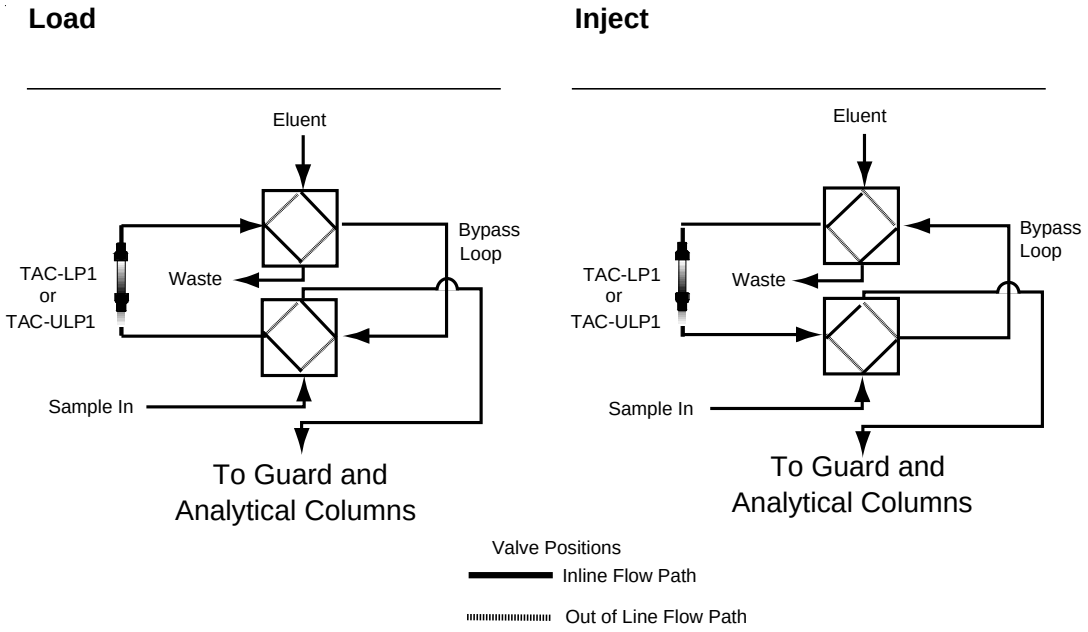


Figure 3
Loading the TAC-LP1 or TAC-ULP1 Column

3.2 Reagent and Sample Handling

The use of the TAC-LP1 or TAC-ULP1Column has certain limitations. At trace analyte concentration levels (µg/L), the results of the 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 the TAC-LP1 or TAC-ULP1 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 can 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. 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.

3.2.3 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 quantification is used, it is critical that standard solutions are correctly prepared.

A. 1,000 mg/L (1 mg/L = 1 ppm) 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 mg/L 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.

3.3 Concentrator Capacity

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 Determinations for Concentrator Injection,” for the determination of the maximum amount of sample or standard that can be quantitatively loaded. In Figure 4, “Linearity Determinations for Concentrator Injection,” the break in the curve where linearity starts to change is at a concentration volume of 2 mL of 30 µg/L fluoride. 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.

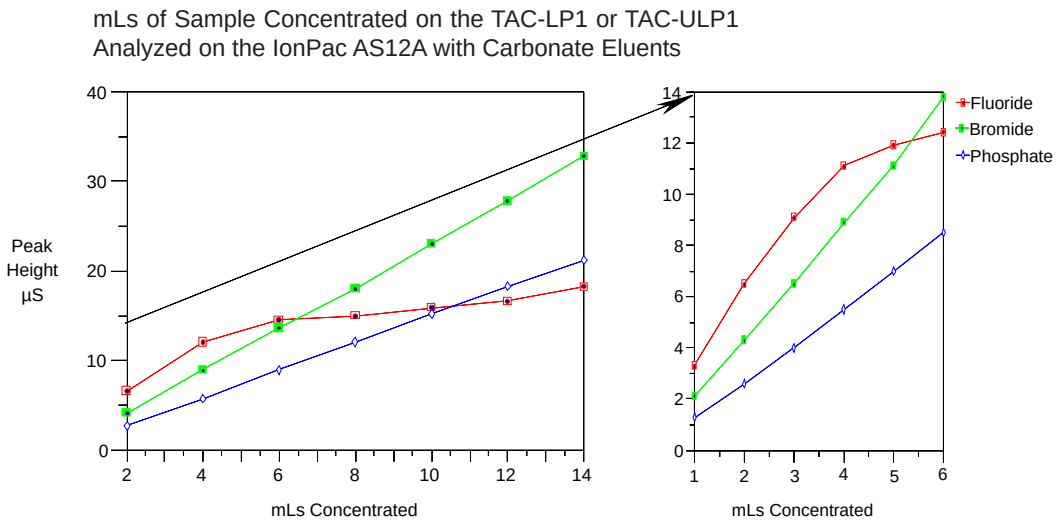


Figure 4
Linearity Determinations for Concentrator Injection

3.3.1 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 volume for a concentrator column is usually defined as the volume of sample necessary to elute the most weakly retained ions of interest in the sample. The more strongly retained ions in the sample, such as SO_4^{2-}, can elute the more weakly retained ions in the sample, such as F^-.

It is also possible for a high concentration of a weakly retained ion such as Cl^- to elute a more strongly retained ion present at low concentration. This can occur if one is attempting to concentrate trace ions in a concentrated matrix.

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.

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 sulfate, the simulated sample should also contain sulfate. The sulfate ion will act as an eluent (E2).

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

C. Set up the Ion Chromatograph, as shown in Figure 5, “Instrument Setup for the Determination of the Breakthrough Volume.”

D. Equilibrate the TAC-LP1 or TAC-ULP1 with the eluent (E1) to be used in the analysis. Set the flow rate necessary to achieve a stable baseline and wash the column in this manner for at least 10 minutes.

E. Switch to the simulated sample as an eluent (E2). Without delay, manually inject 50 µL of the 1 mg/L standard.

F. Record the resulting chromatogram and calculate the breakthrough volume, as shown in Figure 6, "Typical Data Obtained in the Determination of the Breakthrough Volume."

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

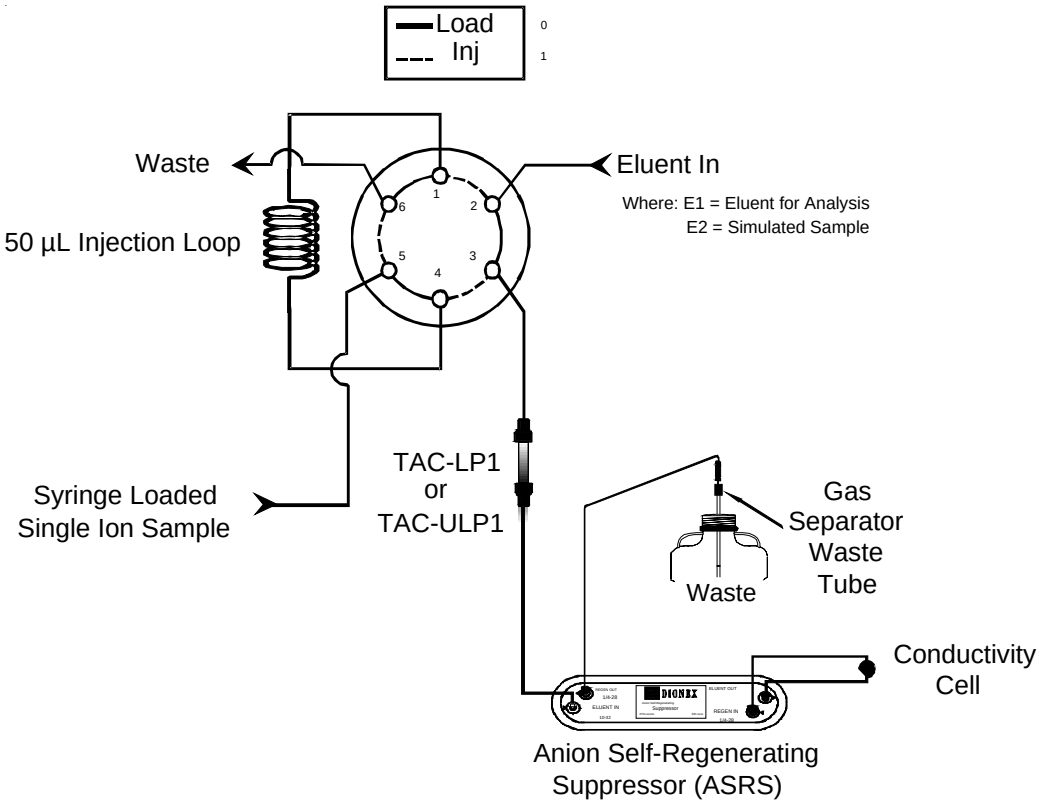


Figure 5
Instrument Setup for the Determination of the Breakthrough Volume

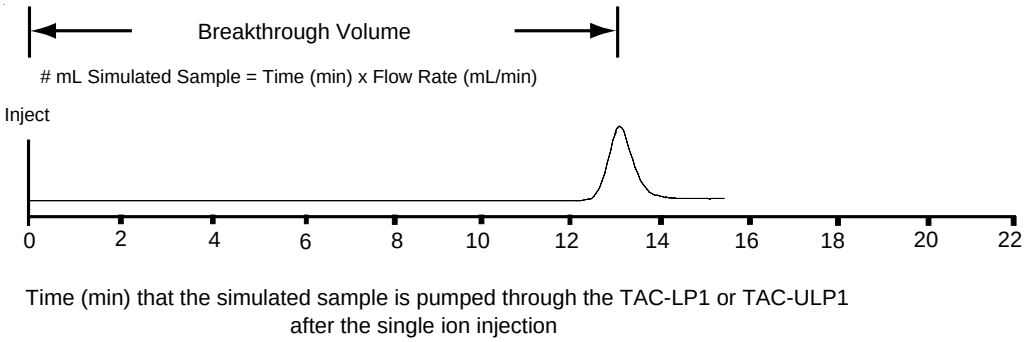


Figure 6
Typical Data Obtained in the Determination of the Breakthrough Volume

SECTION 4 - EXAMPLE APPLICATIONS

4.1 Manual Concentration on TAC-LP1 Versus Direct Injection

The following example demonstrates the advantages of sample concentration with low sensitivity detection versus direct injection with high sensitivity detection.

Sample Loop Volume:	See Chromatogram
Column:	IonPac AS12A Analytical Column + IonPac AG12A Guard Column
Eluent:	2.7 mM Na_2CO_3/0.3 mM $NaHCO_3$
Eluent Flow Rate:	1.5 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS ULTRA (4-mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS III (4-mm)
MMS Regenerant:	50 mN H_2SO_4
or AES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES
Expected Background Conductivity:	14-16 µS

	Analyte	µg/L (ppb)
1.	Fluoride	40
2.	Chloride	50
3.	Nitrite	100
4.	Bromide	200
5.	Nitrate	200
6.	Phosphate	300
7.	Sulfate	200

In water with density = 1

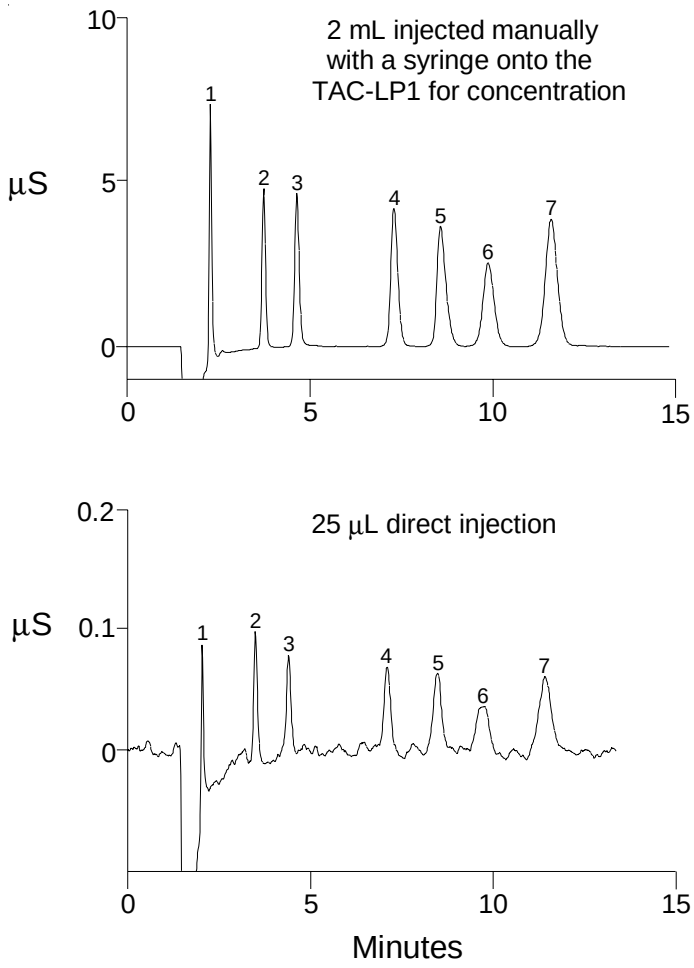


Figure 7
Manual Concentration versus Direct Injection

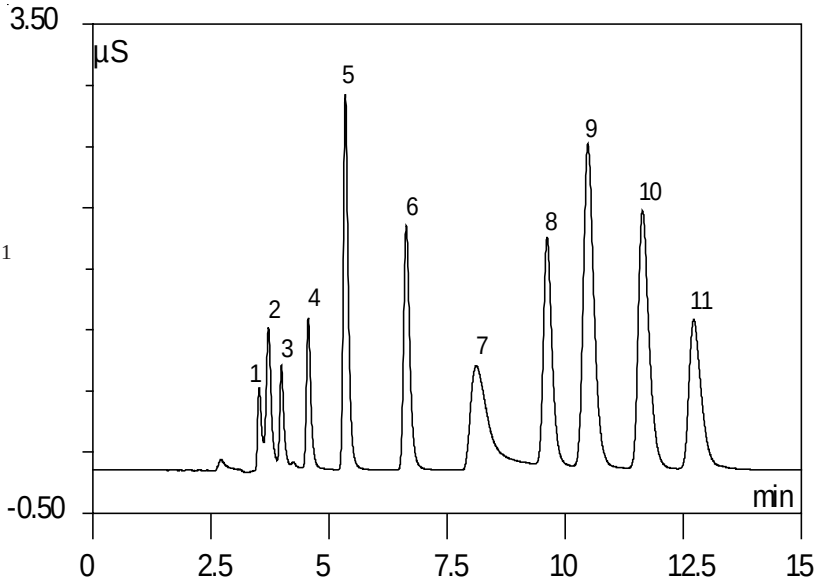
4.2 Manual Concentration on TAC-ULP1 Versus Direct Injection

The following example demonstrates the advantages of sample concentration with low sensitivity detection versus direct injection with high sensitivity detection.

Sample Loop Volume:	See Chromatogram
Column:	IonPac AS18 Analytical Column + IonPac AG18 Guard Column
Eluent:	23 mM Potassium hydroxide
Eluent Flow Rate:	1.0 mL/min
Temperature:	30 °C
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS ULTRA (4-mm) AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS III (4-mm)
MMS Regenerant:	50 mN H_2SO_4.
or AES Suppressor:	Anion Atlas Electrolytic Suppressor, AAES

	Peaks	mg/L
1.	Fluoride	0.01
2.	Acetate	0.05
3.	Formate	0.02
4.	Chlorite	0.1
5.	Chloride	0.06
6.	Nitrite	0.1
7.	Carbonate	0.40
8.	Bromide	0.20
9.	Sulfate	0.20
10.	Nitrate	0.20
11.	Chlorate	0.20

1 mL injected with AS50 Autosampler onto the TAC-ULP1 for concentration



25 µL direct injection

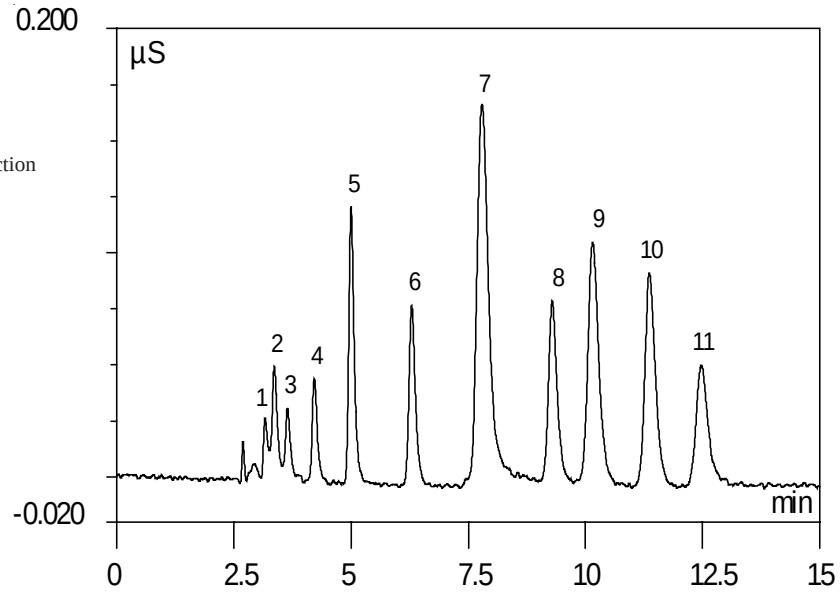


Figure 8
Manual Concentration versus Direct Injection

SECTION 5 - TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using the TAC-LP1and TAC-ULP1 Columns. 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 your nearest Dionex Regional Office (see, "Dionex Worldwide Offices").

5.1 High Back Pressure from a Contaminated Inlet Bed Support

If the TAC-LP1 or TAC-ULP1 Column displays high back pressure, the bed support in the column inlet may be contaminated. **For proper operation in the SP10 AutoNeutralizer, the TAC-LP1 or TAC-ULP1 should generate no more than 70 psi back pressure at a flow rate of 0.5 mL/min.** 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. Before assembling, clean the column body threads and the end fitting threads of any resin particles. If any resin remains on the threads of either the column body or the end fitting, the column may leak regardless of how tight the end fitting is turned onto the column body. Use the end of the column to carefully start the bed support assembly into the end fitting.

Product	TAC-LP1 P/N	TAC-ULP1 P/N
Bed Support Assembly	042955	042955
End Fitting (10-32 ferrule type)	052809	052809

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 clean before screwing the end fitting back onto the column.

F. Reconnect the column to the system and resume operation.

5.2 High Background, Noise or Baseline Instability

Normally, problems such as high background, noise, or baseline instability will not be attributable to the TAC-LP1 or to theTAC-ULP1 column. These problems usually originate in either the analytical column or the post-column detection chemistry. Before checking the TAC-LP1 or TAC-ULP1 as the source of system background noise, consult the appropriate troubleshooting sections in the analytical column's 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-LP1 or to the TAC-ULP1, then proceed with the following steps:

A. Make sure that the TAC-LP1 or the TAC-ULP1 is not leaking.

B. Make sure that the eluents are correctly formulated.

C. If you are using a Anion MicroMembrane Suppressor III (AMMS® III), make sure that the regenerant is formulated correctly.

D. Make sure that the eluents are made from chemicals with the recommended purity (see Section 3, "Operation").

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

F. Make sure that the Anion Self-Regenerating Suppressor, the ASRS® ULTRA, the Anion MicroMembrane Suppressor, the AMMS III, or the Anion Atlas® Electrolytic Suppressor, the AAES, is suppressing correctly by bypassing the TAC-LP1 or TAC-ULP1 and making direct injections.

5.3 Poor Peak Shape

In some instances, poor peak shape in Ion Chromatography may be caused by a contaminated TAC-LP1 or TAC-ULP1. To clean the TAC-LP1 or TAC-ULP1, see, "Column Cleanup of Polyvalent Anions and Base-Soluble Contaminants" in the Column Care Appendix.

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 Low Pressure Trace Anion Concentrator Column (TAC-LP1) is 3,000 psi.

A.2 Column Start-up

The column is shipped with 20 mM NaOH as the storage solution. Flush the column for 30 minutes with eluent before attempting to concentrate sample. Pump the effluent directly to a waste container while washing the column. DO NOT pump this effluent through the guard column, analytical column and/or the suppressor.

A.3 Column Storage

The TAC-LP1 should be stored in the base form. Flush approximately 5 mL of 20 mM NaOH through the TAC-LP1.

A.4 Column Cleanup of 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 Module. Disconnect the Gradient Mixer or Anion Trap Column from the Gradient Pump. Connect the Low Pressure Trace Anion Concentrator (TAC-LP1) Column directly to the Gradient Pump. Direct the effluent from the TAC-LP1 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. Equilibrate the TAC-LP1 with eluent for 15 minutes at 1 mL/min before resuming normal operation.

F. Reconnect the anion guard, analytical column and the suppressor between the injection valve and the Conductivity Module. Reconnect the Gradient Mixer or Anion Trap Column between the Gradient Pump and the Injection Valve. Resume operation.

A.5 Column Cleanup of Organic/Anionic Contaminants

A. Prepare a 500 mL solution of 200 mM HCl/80% acetonitrile.

B. Disconnect the guard, analytical columns and the suppressor from the injection valve and the Conductivity Module. Disconnect the Gradient Mixer or Anion Trap Column from the Gradient Pump. Connect the Low Pressure Trace Anion Concentrator (TAC-LP1) Column directly to the Gradient Pump. Direct the effluent from the TAC-LP1 directly to a waste container.

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

D. Pump the 200 mM HCl/80% acetonitrile solution through the column for 15-30 minutes.

E. Equilibrate the TAC-LP1 with eluent for 15 minutes at 1 mL/min before resuming normal operation.

F. Reconnect the Anion Guard, analytical column and the suppressor between the injection valve and the Conductivity Module. Reconnect the Gradient Mixer or Anion Trap Column between the Gradient Pump and the Injection Valve. Resume operation.