DIONEX
CORPORATION

DX-120 OPERATIONAL TECHNIQUES, MAINTENANCE & TROUBLESHOOTING

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DX120 Course Objectives

1. To identify the components of the DX 120 system.
2. Make all liquid, air, and electric line connections required to set up an operating system.
3. Describe how each module performs its functions.
4. Identify the front panel buttons and their functions.
5. Describe how a self-regenerating suppressor works.
6. Perform ion separations using isocratic methods.
7. Troubleshoot common problems associated with hardware and chemistry.
8. Perform routine maintenance of the chromatography system.

DX-120 OPERATIONAL TECHNIQUES, MAINTENANCE & TROUBLESHOOTING

I. INTRODUCTION

II. ELUENTS, COLUMNS AND SUPPRESSION
- A. Eluents
  1. Purpose of the eluent
  2. Key points to remember when preparing and using eluents
  3. Characteristics which affect chromatography
  4. Eluent troubleshooting
- B. Columns
  1. Purpose of the column
  2. Maintenance
  3. Troubleshooting
- C. Suppressors
  1. Purpose
  2. Operation
  3. Maintenance
  4. Troubleshooting

III. INSTALLATION AND OPERATION
- A. Description of components and flow path
- B. DX-120 Installation and Operation (Exercise)

IV. MAINTENANCE AND TROUBLESHOOTING
- A. DXP pump
- B. Tubing and fittings
- C. Valves
- D. Conductivity cell

V. SYSTEM TROUBLESHOOTING
- A. System and maintenance log
- B. Troubleshooting exercises

VI. HARDWIRING AND AUTOMATION
- A. Rear panel hardwire connections
- B. Data collection
- C. System automation

INTRODUCTION

The goal of this course is to provide a basic understanding of Operation, use and Maintenance of the DX-120 chromatography system. Information from this manual, along with notes taken during the course, will provide a thorough understanding of chromatography.

Upon completion of the DX-120 Operational Techniques, Maintenance & Troubleshooting course:

- You should have a good understanding of the purpose and operation of all components that make up the DX-120.
- You should feel comfortable with all maintenance procedures described.
- You should feel confident in using troubleshooting techniques to identify instrument problems.
- You should understand how to hardwire a DX-120 for data collection and automation.

Objectives

Bie the end of this section, each participant should be able to:

- * Describe the purpose of an eluent
- * Describe the key points for preparing and using eluents
- * Identify eluent characteristics which affect chromatography

Purpose of an Eluent

Chromatography is defined as the separation of components in a mixture by partitioning between a mobile phase and a stationary phase. The mobile phase is the eluent and the stationary phase is the column (which will be discussed in a later section). The eluents contain ions (with similar characteristics of the sample ions) which compete with sample ions for sites on the column.

Example: As a sample containing solute "AB" travels through the column, "A" has less attraction than "B" to the column. "A" will therefore remain on the column for a shorter time than "B" and the resulting elution sequence will be "A", then "B". In this example, the eluent first pushes "A" off the column, then "B". The eluent itself will have the same attraction to the column as "A" and "B".

Preparing an Eluent

Proper preparation of eluents is very important. The following guidelines should be followed:

* Prepare solutions from reagent (HPLC or ACS) grade chemicals with a label analysis of 99% (or Better) purity. Be sure to date the reagent when it is received and when it is opened.

* All standards, eluents regenerants and reagents should be prepared with 18 megohm (or more) deionized, filtered water. 18 megohm water will produce a background conductivity of < 1 uS. This will reduce background conductivity. It is not recommended that bottled water be used for conductivity applications.

* For best system performance and results, filter particulates from all solutions including samples. OnGuard cartridges (which will be discussed later) will filter both particulates and interfering ions from a sample.

* Whenever possible, prepare high concentration stock solutions for eluents and standards. High concentration stock solutions are more stable than those of low concentration, and they can be used for serial dilutions.

* After eluents have been prepared, they should be thoroughly degassed using helium prior to introduction into the chromatography system. This will prevent air bubbles from forming in the pump or detector cell.

* Special care should be taken when preparing Sodium Hydroxide eluents. Carbon Dioxide from the atmosphere will readily dissolve in a NaOH solution where it is then converted to a carbonate anion. This carbonate anion will cause contamination problems when put through a chromatography system. Prepare a NaOH solution from a 50% w/w NaOH stock solution. Do not use NaOH pellets, as they are loaded with carbonate. When aliquoting from a NaOH stock solution bottle, it is advisable to take the aliquot from the center of the bottle because carbonate contamination that has already been introcuced will be attracted to the walls of the container. It is also important to degas the reagent water prior to adding the NaOH aliquot to avoid converting any Carbon Dioxide that is dissolved in the water to carbonate upon introduction of the NaOH aliquot.

* When using Sodium Carbonate/Sodium Bicarbonate eluents, it is important to be aware of biological contamination. Because of its neutral pH, Sodium Carbonate/Sodium Bicarbonate is a very good bacterial medium. Bacterial growth will occur after approximately a week to two weeks. Bacterial growth can plug a chromatography system and ruin a set of columns.

Characteristics Which Will Affect Chromatography

There are several characteristics of an eluent which will affect how it interacts with the column and the eluting species. These include the following:

- Eluent Species Valence: Monovalent eluents will push monovalent species off the

  column, and divalent eluents will push divalent species off the column (hence the phrase "Likes elute likes"). The higher the valence of the sample ion, the longer it is retained on the column.

- Eluent Species Selectivity: The eluent ions compete with the sample ions for sites on the column. If the eluent ion selectivity is too high relative to the sample ions, the sample ions will not remain on the column long enough to separate.

- Eluent selectivity is a function of valence, and valence is a function of pH of the eluent. The valence of the sample species can be transformed by altering the pH of the eluent. If the valence of a sample species is changed, the time the species elutes off the column will also be changed.

ELUENT TROUBLESHOOTING

1. Analytes elute at later than normal retention times.

   A. Eluent is too strong
   B. Eluent is too weak
   C. Eluent chemicals are in hydrated form
   D. Both B and C

2. Monovalent analytes are eluting fine but divalent analytes are eluting later.

   A. Monovalent counter ion is too weak
   B. Divalent counter ion is too strong
   C. Divalent counter ion is too weak or do not exist
   D. Monovalent counter ion is too strong

3. Shorter than usual retention times are noticed.

   A. Eluent is too strong
   B. Eluent is too weak
   C. Counter ion concentration is too weak
   D. Both A and C

4. Peak elution order has changed, sensitivity has decreased.

   A. Eluent pH is too high
   B. Eluent pH is too low
   C. Counter ions valence has changed
   D. All of the above
   E. None of the above

5. Notice a loss sensitivity and a decrease in retention times.

   A. Eluent is too weak
   B. Eluent is contaminated
   C. NaOH eluent has too much carbonate in it
   D. Carbonate/Bicarbonate eluent is too old
   E. B,C and D

6. Background conductivity is too high.

   A. Eluent is too weak
   B. Counter ion is not present in a high enough concentration
   C. Both A and B
   D. None of the above

7. The pump will not stay primed.

   A. Eluent is contaminated
   B. Eluent has not been degassed
   C. Eluent concentration is too high
   D. Eluent has not been filtered
   E. Both B and D

ELUENTS

1. Chromatography separation occurs between the ______________ and the ________________.

2. All solutions should be prepared from chemicals of at least ______ purity.

3. All solutions should be prepared with deionized water with a megohm value of at least ________________.

4. _______________ water is not recommended for conductivity applications.

5. All solutions should be ______________ to remove particulates prior to use.

6. _________ concentration eluents and solutions are more stable than _______ concentrations.

7. Eluents should be degassed with ________ to remove dissolved gases prior to use.

8. NaOH ___________ should not be used to prepare NaOH eluents.

9. Deionized water used to prepare NaOH eluents should be __________ prior to adding NaOH.

10. Because of its neutral pH, Carbonate eluents are very good __________ ___________.

11. Divalent eluents will elute ____________ species off the column.

12 If eluent ___________ is too high, sample ions will not remain on the column long enough.

13. A change in pH can possibly change the ___________ of a sample species or eluent.

COLUMN THEORY

Objective:

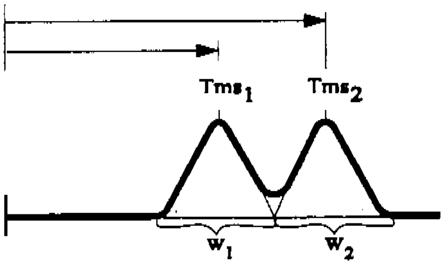
By the end of this section, each participant should be able to:

- Describe the purpose of the column.
- Define chromatography, resolution, efficiency, selectivity, and capacity
- Describe the purpose of a guard column and an OnGuard cartridge
- Recognize column deterioration
- Perform routine maintenance procedures

What is Chromatography?

The purpose of the column is to provide a physical medium where the eluent (mobile phase), the sample (analytes of interest) and the resin (stationary phase) within the column can interact to cause a separation of the sample into the individual analytes of interest. The amount of separation that is achieved can be measured using a chromatographic term called resolution.

Resolution *The measure of separation between two peaks. The figure below shows the resolution between two peaks and the resolution equation.



$$R = \frac{Tms_2 - Tms_1}{\frac{W_1 + W_2}{2}}$$

Using the equation for resolution and the data given in the following three examples, calculate the resolution of fluoride and chloride.

	ANALYTE	RETENTION TIME	PEAK WIDTH	RESOLUTION
1.	fluoride	1.05	2 seconds	
	chloride	1.65	3 seconds	____________
2.	fluoride	0.80	2 seconds	
	chloride	2.55	3 seconds	____________
3.	fluoride	1.05	2 seconds	
	chloride	4.05	3 seconds	____________

What do these resolution values mean? Remember that resolution is the measure of separation of two peaks. The resolution values can be compared with the following convention used to determine two peak resolution:

> If $R<0.5$, the peaks are not resolved enough to integrate properly
>
> If $R>0.5$ but <1.0, the peaks are resolved enough to integrate properly, but are not completely separated.
>
> If $R> 1.0$, the peaks are considered completely (baseline) resolved. The two peaks are completely separated.

The resolution equation does not take into account such things as poor peak symmetry, differences in peak response, etc. It should only be used as a rough estimate of peak resolution.

There are three factors which will affect resolution: efficiency, capacity and selectivity. The following table describes the factors further.

Term and Symbol	**Definition**	**How it Can be Affected**
Efficiency (N)	A measure of peak dispersion as components move through a column. An efficient column will retain components of interest without spreading.	Voids or channels from excessive backpressure, or disruption of column packing.
Capacity (k')	The maximum concentration that can be loaded onto a column before overloading.	Highly retained components, and eluent is not strong enough to elute them off the column: contamination.
Selectivity (α)	A measure of affinity of the sample ion for the resin exchange sites.	Change the resin

Figure 2 shows the effect of varying efficiency, capacity and selectivity on the resolution between two peaks. In the initial chromatogram, resolution between the two peaks is poor. When capacity is varied, resolution does not improve. When efficiency is varied, resolution is better than the initial state. Varying selectivity will give the best resolution.

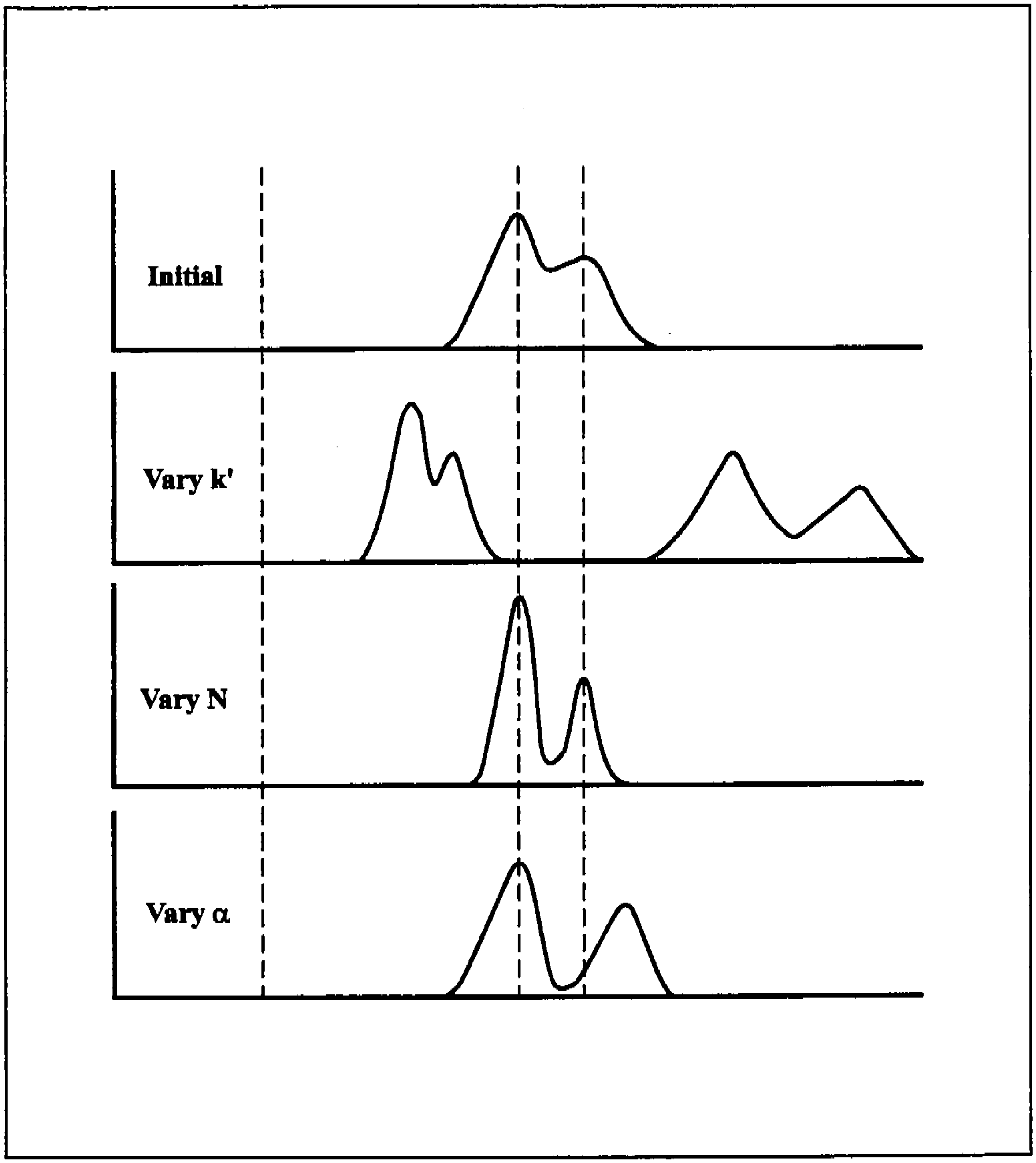


Figure 2, Summary - how k', N, and α Affect Resolution

Guard Columns

Guard columns are smaller versions of the separator columns. They are placed in the system before the separator column. Since they have the same packing material as the separator colun, contaminants will be retained on the guard column.

Guard columns should be used because:

- They are packed with the same material as the separator column.
- They will retain contaminants the same as the separator column.
- They are easier to clean than the separator column.
- They are less expensive to replace than the separator column.

OnGuard Cartridges

OnGuard cartridges can be used to remove contaminants from the sample before it is injected. The cartridges contain packing material similar to columns. Certain components which could contaminate the column or interfere with the analysis are retained in the cartridges.

Cartridge	**Packing Material**	**Interferents Retained**	**Part No.**
On-Guard P	Polyvinylpyrolidone	Tannic & humic acids, phenols, lignins, azo compounds, cationic surfactants	039597
On-Guard RP	Divinylbenzene	Anionic & nonionic surfactants, aromatics, aliphatics, proteins	039595
On-Guard H	H-form cation resin	Cations, including metals and surfactants	039596
On-Guard Ag	Ag-form cation resin	Chloride,bromide, iodine, proteins	039637
On-Guard A	Bicarbonate-form anion resin	Anions	042102
On-Guard Ba	Barium-form Sulfonic acid resin	Sulfate	046072

Before use, OnGuard Cartridges must be pre-treated to activate the resin. Refer to the OnGuard Operators Manual for specific instructions.

Summary-Guard Columns and OnGuard Cartridges

- Guard Columns and OnGuard cartridges can be used to protect columns from contamination.
- Guard columns have the same resin as separator columns and contaminants will be retained on the guard column.
- OnGuard cartridges are used for sample treatment prior to injection.

Guard Column Exhaustion

The guard column, which is a shorter version of the analytical column, filters particulate matter from the eluent and the sample. Strongly retained ionic species are trapped before contamination of the analytical column can occur. The procedure for determining guard exhaustion is as follows:

1. Depending upon your application, prepare the proper standard.
2. Install only the guard column.
3. If using chemical eluent suppression, use the suppressor that will be used during analysis.
4. Equilibrate the guard column with the eluent.
5. Inject the standard selected and record retention time of peaks.
6. Inject a typical sample matrix.
7. After the baseline stabilizes, repeat the injection procedure for the sample matrix.
8. Inject the standard again and record the retention time of peaks.
9. Repeat steps 6 and 8. Record retention time of peaks.
10. For each injection of standard, calculate the capacity factor

$$\text{from } k' = \frac{t_2 - t_1}{t_1}$$

11. Prepare a graph of k' versus number of sample injections.

12. Extrapolate the line until k' intersects the x coordinate.

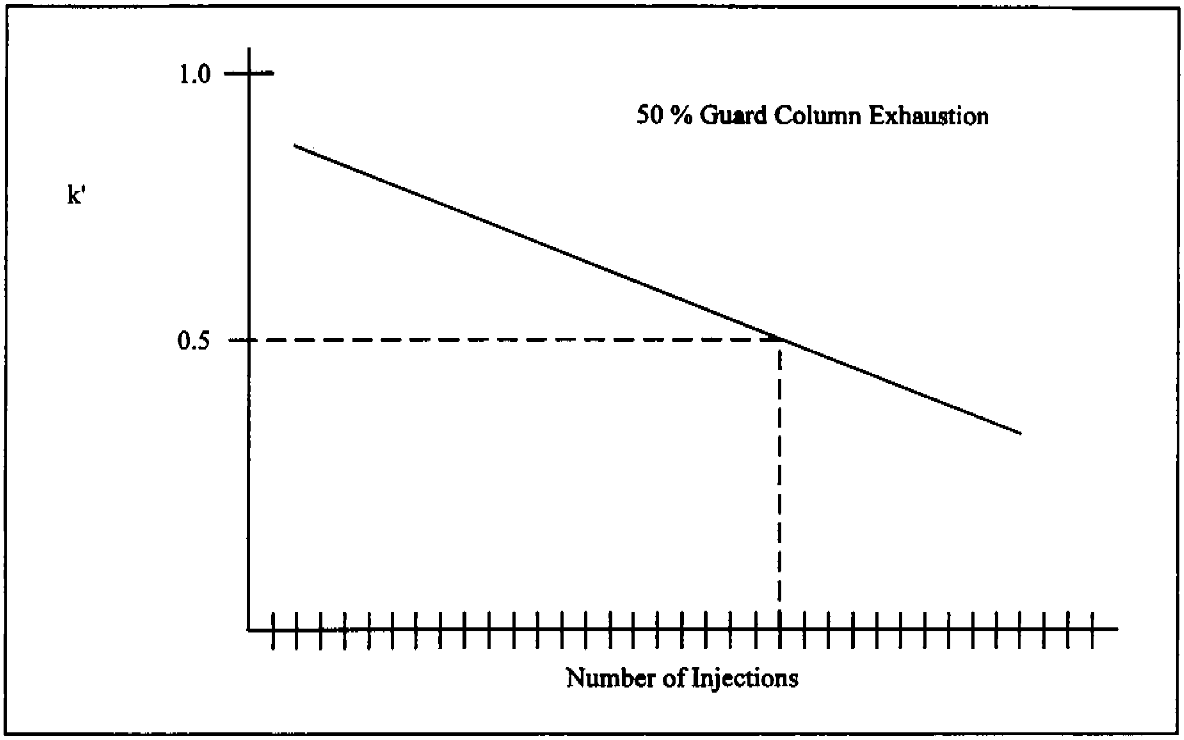
13. A vertical drop from k'(initial)/2 is the 50% exhaustion of the guard column.

$$k' = \frac{t_2 - t_1}{t_1}$$

$$\text{Exhaustion} = \frac{k'\ \textit{intial}}{2}$$

t_1 : retention time peak 1
t_2 : retention time peak 2

NOTE: THE SAME PROCEDURE CAN BE USED TO TEST FOR SEPARATOR COLUMN EXHAUSTION.



The capacity factor, k', can be helpful in determing the condition of a column. For example, the plot above measures k' of [SO_4^{2-}] against the number of injections. This is done using only the guard column. After several injections of the analyte, the k' value will decrease depending on how pure the matrix is and eventually after a finite amount of injections, k' will become a very small number.

Initially, when a column is brand new, the k' value for sulfate say is X_1. Several injections are made from day to day analysis to measure X and at a given time the measurement will delineate from X_1. After sufficient data points have been obtained, a best fit line would be drawn. If the sample matrix contains a lot of contaminants, the slope of k' over the number of injections would be largely negative. The corresponding value that correlates with $X_{0.5}$ indicates that at that injection, 50% of the guard column has been exhausted and at this time it is necessary to clean or change the guard column.

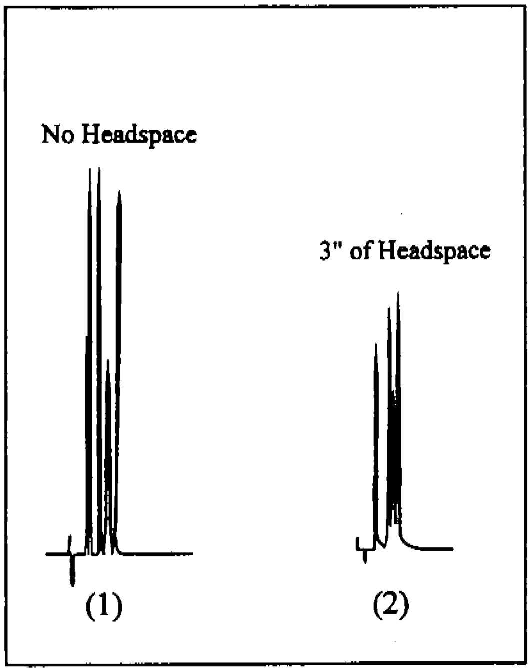
$$k' = \frac{t_2 - t_1}{t_1}$$

Column Deterioration

During routine use, columns may show signs of deterioration. Deterioration of columns will occur no matter what an analyst does to prevent it. However, columns may deteriorate prematurely if contaminants are introduced into the chromatography system. Many times columns can be cleaned in order to restore them to approximately their original condition. Column cleaning will be addressed later in the course. The effects of column deterioration can be seen as any of the following:

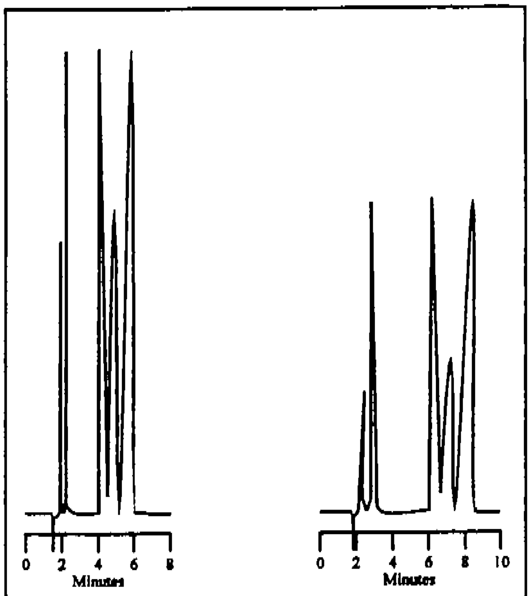
- Decreasing retention times
- Loss in resolution
- Abnormal peak shapes
- Abnormal (usually higher) operating pressures

Some examples of these are given below.



(1) New Column

(2) Deteriorating Column=Loss ofresolution



No leak
(1)

Major leak
(2)

(1) Deteriorating Column= Decreasing retention times

(2) New Column

Column Cleaning

Detailed instructions for column cleaning can be found in the column manuals and Technical Notes 2 and 7. Cleaning and storage solutions for some of the more common Dionex columns are shown in the table below.

Column	Cleaning Solution	Storage Solution
AS4A-SC	1 M HCl plus 0.1 M KCl	0.1 M NaOH
AS9	100 mM oxalic acid plus 50 mM NaOH	Eluent
AS11	0.5 - 1.0 M NaOH	Eluent
→ AS14	10X eluent concentration	Eluent
CS3	1 M HCl plus 0.1 M KCl	Eluent
CarboPac PA1	0.5 - 1.0 M NaOH	Eluent
CS5A	1 M HCl or strong chelator	0.1 M NaOH
CS12, CS14	5X MSA eluent concentration	Eluent

Once a cleaning solution is selected, the following procedure describes steps for cleaning the guard and separator columns.

Column Cleaning Procedure

1. (Disconnect suppressor and detector (column output can go to waste container).*
2. If cleaning both the guard and separator columns, place the guard column after the separator column.
3. Fill eluent bottle with cleaning solution.
4. Set a flow rate of 1.0 mL/min.
5. Pump 60 mL of the cleaning solution through the column.
6. Pump 30 mL of deionized water (18 megohm or greater) through the column.
7. Re-connect the system (suppressor, valves, etc.).
8. Equilibrate with eluent for 30-60 minutes.

* An alternative is to connect the separator and/or guard column directly to the line from the pressure transducer. All valves, suppressors, etc. are then bypassed.

Column Cleaning Tips

- When unsure of the cleaning solution, 10-100 strength eluent can be used.
- To clean the guard and separator columns at the same time, place the guard column after the separator column.
- Pump cleaning solution through the column in the same direction as eluent flow.

Column Maintenance

Bed Supports

Bed supports are small, frit assemblies located in both ends of a column (see Figure 2). They filter out particulates in the eluent stream, and keep the resin from being pushed out of the column. If the inlet bed support becomes clogged with particulates, system pressure will start to increase. Use the following procedure to change the inlet bed supports.

Installation of Inlet Bed Supports

1. Disconnect column from system.
2. Using two wrenches, unscrew the inlet column fitting.
3. Turn the fitting over and tap it against a benchtop to remove the bed support.
4. Discard old bed support.
5. Place new bed support into the end fitting. ***In most bed supports, the frit may be raised above the seal on one end. When changing bed supports, orient the raised frit toward the column. This will help prevent headspace.***
6. Clean any particulate matter from the end of the column tube.
7. Screw end fitting back into the column.

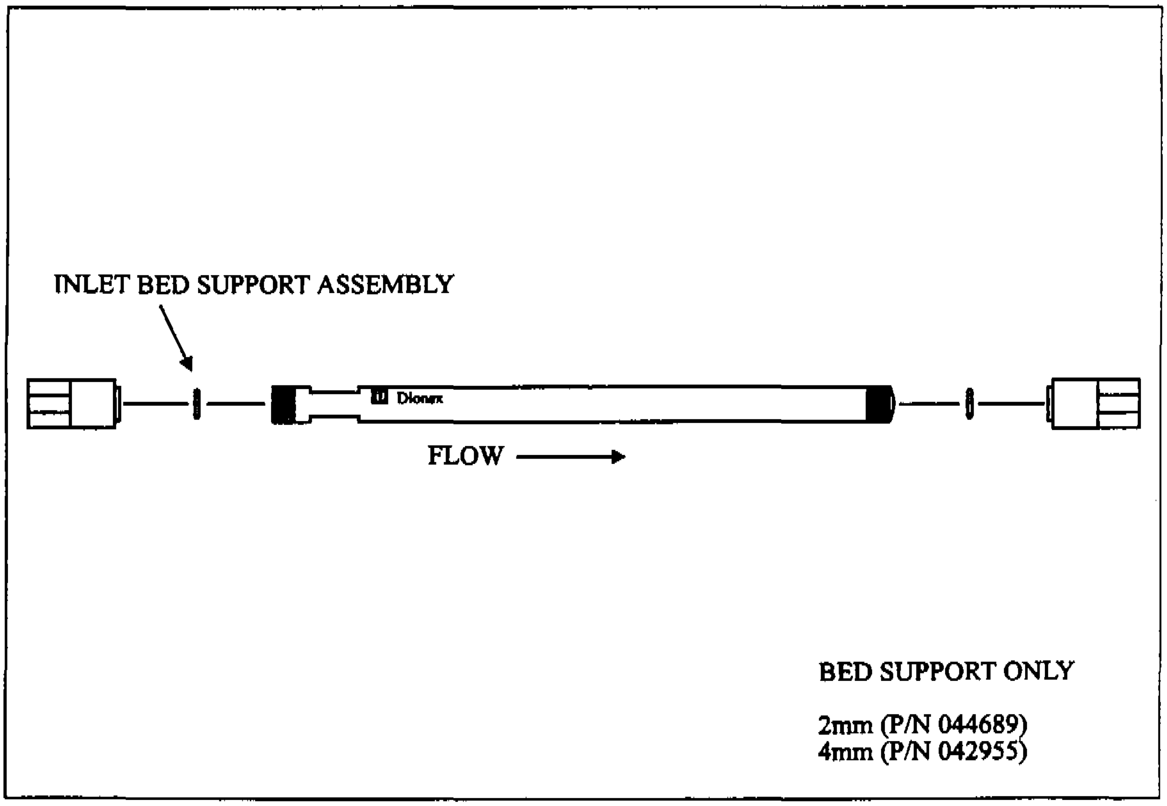


Figure 2. Bed Supports

Objectives

By the end of this section, each participant should be able to:

- Describe how a suppressor works.
- Describe when and how to clean a suppressor
- Describe how an AutoRegen works
- Describe how AutoSuppression works

The Role of Chemical Suppression

The eluents used for ion chromatography contain ions in large quantities and therefore have high. noisy background conductances. It is difficult to detect the small increase in conductance due to the sample components in the presence of this high background. Chemical suppression is used to reduce the eluent background to near zero while simultaneously converting the sample components to their acid or hydroxide forms which have higher conductances than the salt forms (see Figure 1).

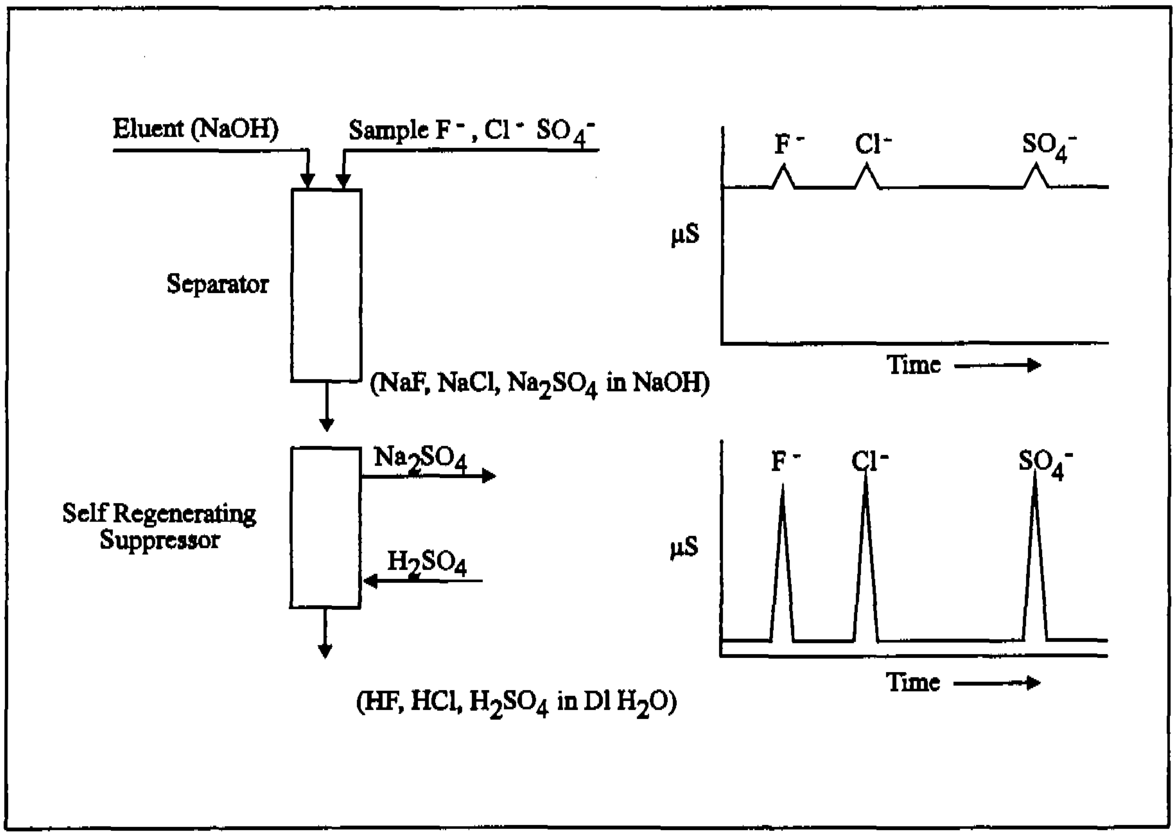
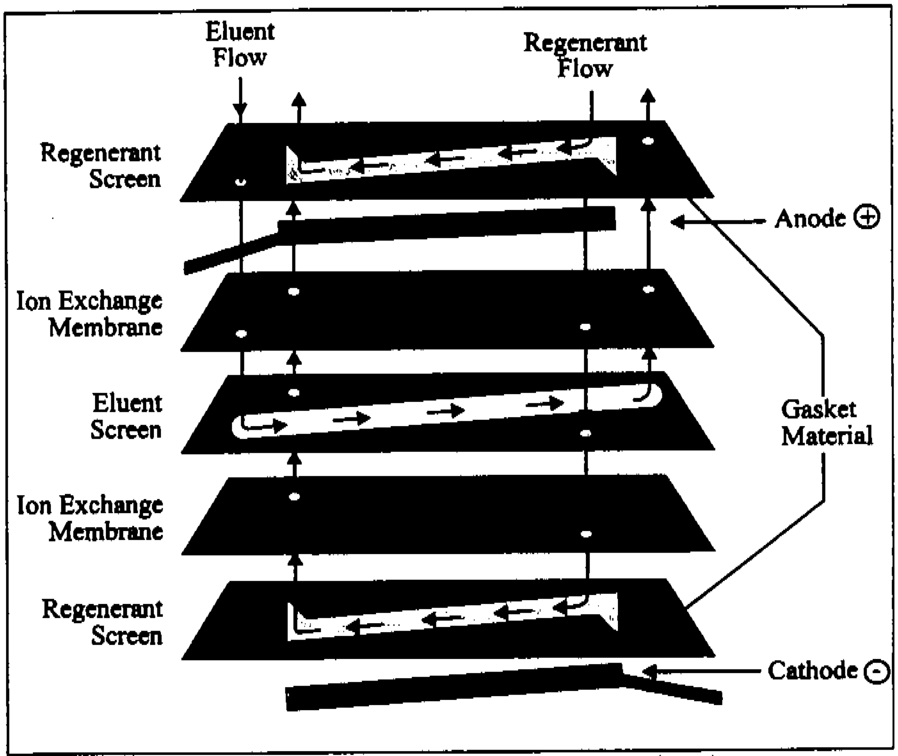


Figure 1. The Role of Chemical Suppression

AUTOSUPPRESSION: THEORY OF OPERATION

Operation of SRS under the AutoSuppression mode is based on electrolysis and electrodialysis chemistry. The SRS consists of two ion exchange membranes sandwiched between three high capacity ion exchange screens positioned in the anodic, eluent, and cathodic chambers. The screens create a convoluted low volume flow path for the eluent and provide an ion transport path for the ion exchange process. The chemical regenerant is generated inside the SRS by electrolysis. The internal construction of the SRS Self-Regenerating Suppressor is shown below.



Internal construction of the SRS Self-Regenerating Suppressor.

AutoSuppression Chemistry of the Anion Self-Regenerating Suppressor (ASRS)

The chemistry of AutoSuppression with the ASRS follows and is illustrated in Figure 5. This example uses sodium hydroxide as the eluent, but sodium carbonate/sodium bicarbonate and boric acid/tetraborate eluents may also be used.

In the ASRS, the hydronium ions generated in the anode chamber move across the cation exchange membrane and react with hydroxide ions in the eluent, forming water. Simultaneously, cations such as sodium in the eluent enter the cathode chamber and combine with regenerant hydroxide ions to form sodium hydroxide, which is then removed to waste.

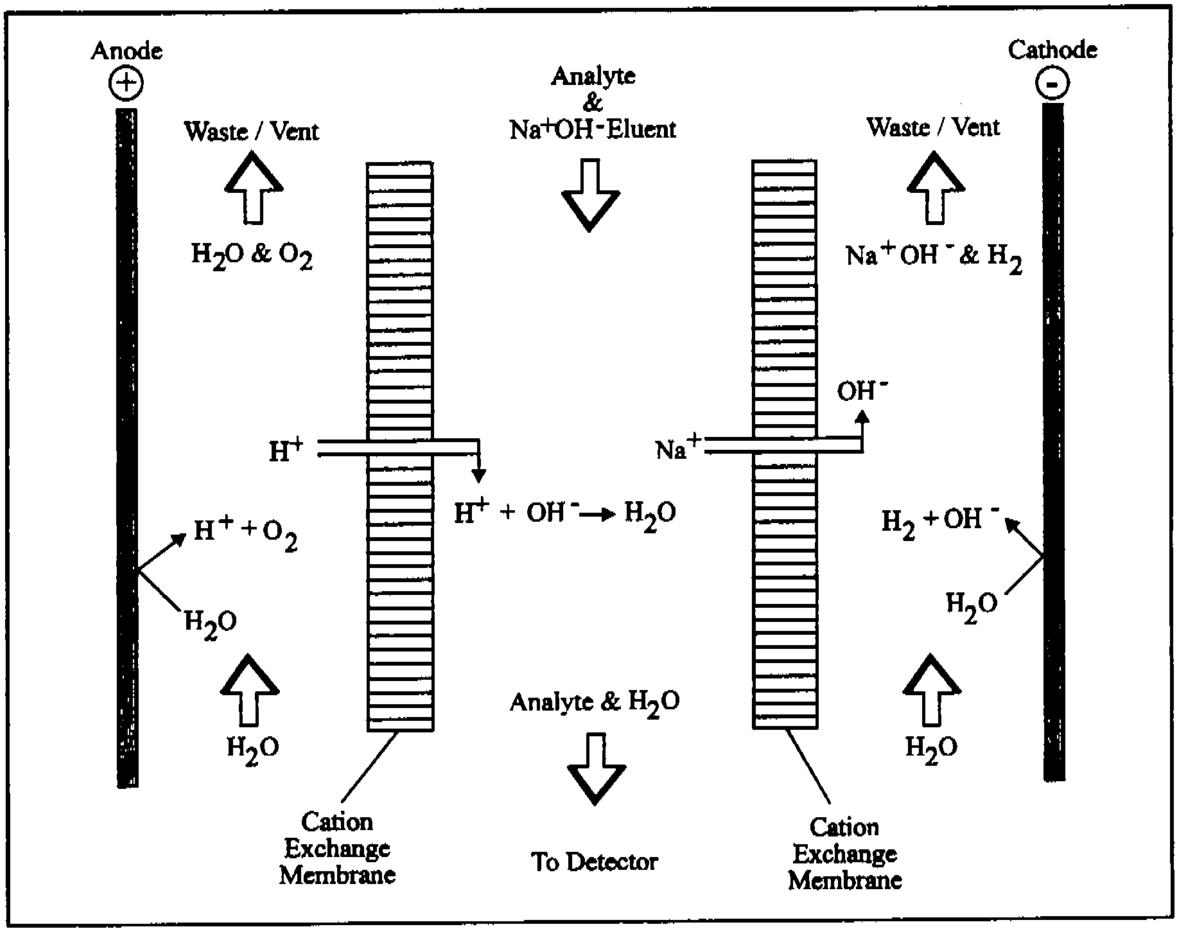


Figure 6 AutoSuppression with the Anion Self-Regenerating Suppressor (ASRS).

AutoSuppression with the Cation Self-Regenerating Suppressor (CSRS)

The chemistry of AutoSuppression with the CSRS follows and is illustrated in Figure 6. Methanesulfonic acid eluents are used with the CSRS in the AutoSuppression Recycle Mode.

The CSRS follows the same principles as the ASRS, but with charges flowing in the opposite direction. In the CSRS, hydroxide ions generated in the cathode chamber move across the anion exchange membrane and react with the hydronium ion in the eluent, forming water. Simultaneously, anions in the eluent enter the anode chamber and are removed by the continuous flow of water.

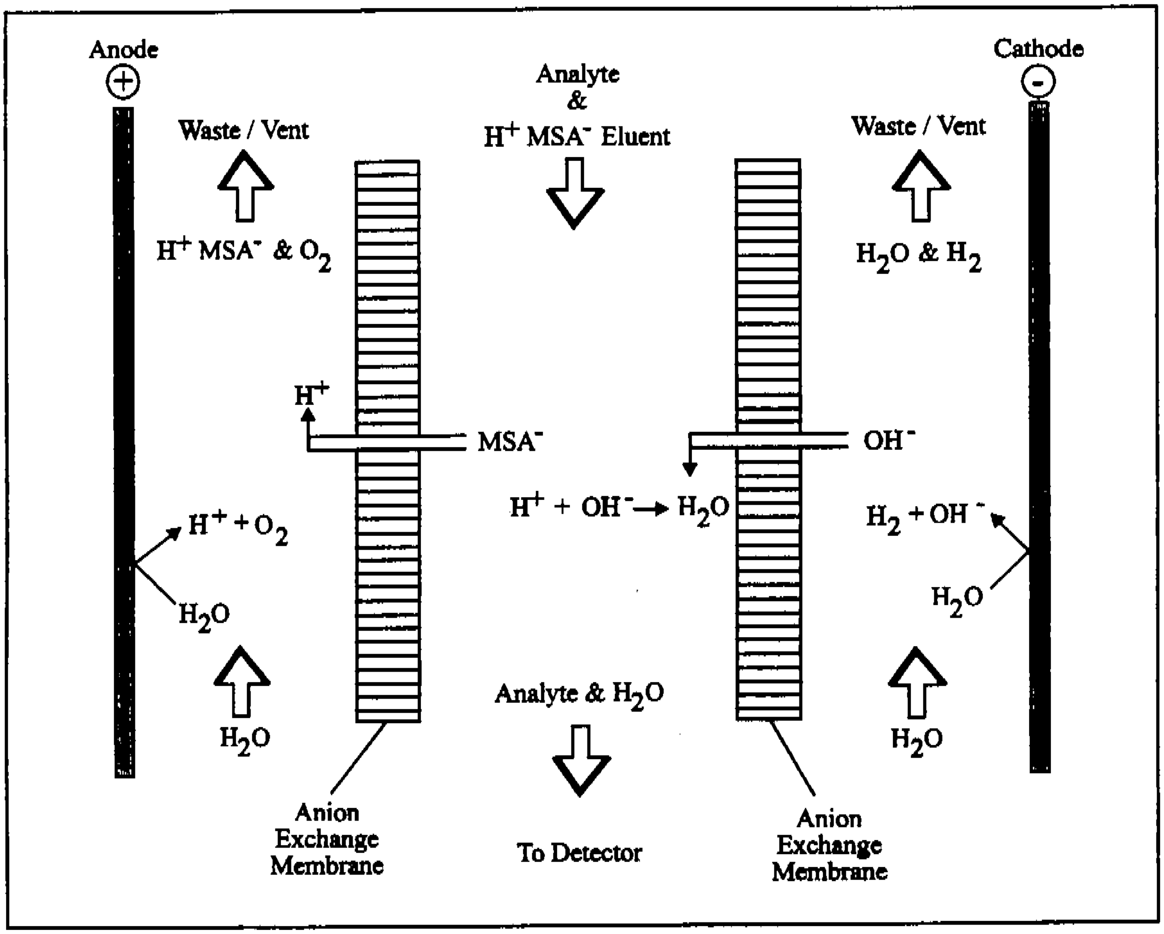
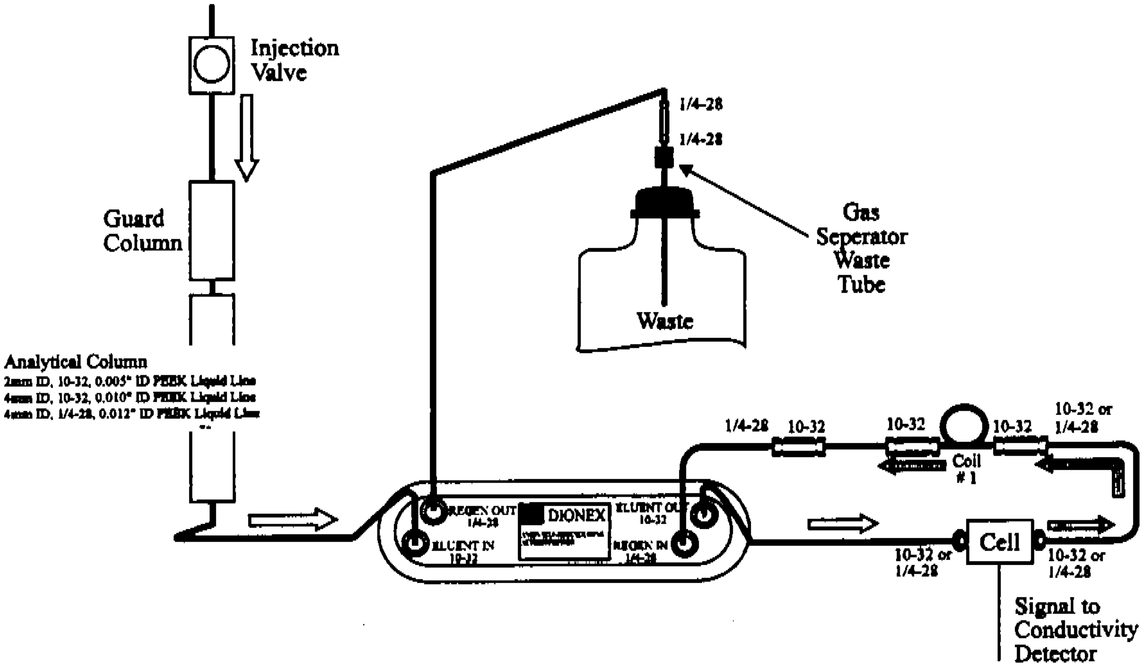


Figure 7 AutoSuppression with the Cation Self-Regenerating Suppressor (CSRS.)

PLUMBING FOR THE AUTOSUPPRESSION RECYCLE MODE OPERATION

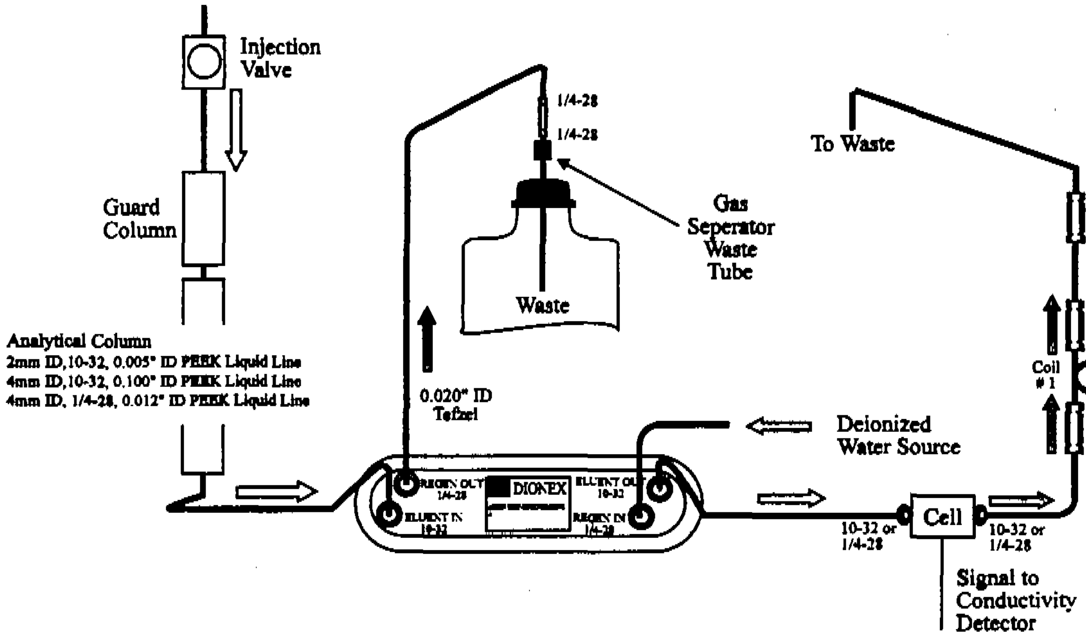


The AutoSuppression Recycle Mode Plumbing Diagram

Coils for ASRS-I Back Pressure Requirements

ASRS-I Type	Flow Rate	ID of Tubing	Length of Each Coil	Number of Coils
4 mm	0.5 - 1.5 ml/mm	0.010" (black)	2.5 feet	2
4 mm	1.5 - 3.0 ml/mm	0.010" (black)	2.5 feet	1
2 mm	0.12 - 0.37 ml/mm	0.005" (brown)	1.0 feet	2
2mm	0.37 - 0.75 ml/mm	0.005" (brown)	1.0 feet	1

PLUMBING FOR THE AUTOSUPPRESSION EXTERNAL WATER MODE



The AutoSuppression External Water Mode Plumbing Diagram

Any analysis that can be performed using the AutoSuppression Recycle Mode can be done using the AutoSuppression External Water Mode. The AutoSuppression External Water Mode can be used to increase suppression capacity above that achieved using the AutoSuppression Recycle Mode (see Figure The AutoSuppression External Water Mode Plumbing Diagram). Suppression capacity increases as the flow rate of water through the regenerant chambers increases. The AutoSuppression External Water Mode achieves higher suppression capacity than that achieved by the AutoSuppression Recycle Mode because the water flow rate is not restricted to the eluent flow rate. A constant source of de-ionized water having a specific resistance of 10 meg-ohm or greater, is supplied to the regenerant chambers to generate hydronium ions for neutralization.

Determining Eluent and Regenerant Concentrations in the Chemical Suppression Mode

The ASRS-I (4mm) and ASRS-I (2mm) have the ability to provide continuous suppression of eluents using chemical regeneration with an acid such as sulfuric acid. See table: Matching Regenerant Concentrations and Flow Rate to Eluent Concentration and Flow Rate lists the eluent concentrations and flow rates of standard eluents used in anion separations and the regenerant concentrations and flow rates required to suppress them. The operation of the ASRS-I (4mm) and the ASRS-I (2mm) requires a constant flow of the regenerant over the membrane in a direction that is countercurrent to the flow of the eluent. The maximum allowed regenerant flow rate is 15 mL/min.

Matching the Current Setting to the Eluent concentration and Flow Rate

Eluent	**Eluent Flow Rate (mL/min)**	**Current Setting**	**Recycle Mode**	**External Water Mode**
Anion Self-Regenerating Suppressor (ASRS-I, 4mm)				
1.7 mM Na HCO_3 1.8 mM Na_2CO_3	0.5-2.0	1	Yes	Yes
2.8 mM $NaHCO_3$ 2.2 mM Na_2CO_3	0.5-2.0	1	Yes	Yes
1-100 mM NaOH	0.5-1.0	3	Yes	Yes
	1.1-2.0	4	Yes	Yes
1-20 mM $Na_2B_4O_7$	0.5-1.0	2	Yes	Yes
	1.1-2.0	3	Yes	Yes
20-50 mM $Na_2B_4O_7$	0.5-1.0	3	Yes	Yes
	1.1-1.5	4	No	Yes
Organic Solvent Containing Eluents	0.5-2.0	OFF	Chemical Suppression Mode ONLY	
Anion Self-Regenerating Suppressor (ASRS-I, 2mm)				
1.7 mM $NaHCO_3$ 1.8 mM Na_2CO_3	0.1-0.50	1	Yes	Yes
2.8 mM $NaHCO_3$ 2.2 mM Na_2CO_3	0.1-0.50	1	Yes	Yes
1 - 100 mM NaOH	0.1-0.35	2	Yes	Yes
	0.36-0.50	3	Yes	Yes
100 - 150 mM NaOH	0.1-0.50	3	Yes	Yes
	0.36-0.50	4	Yes	Yes
1- 30 mM $Na_2B_4O_7$	0.1-0.35	2	Yes	Yes
	0.36-0.50	3	Yes	Yes
30 -75 mM $Na_2B_4O_7$	0.1-0.24	3	Yes	Yes
	0.25-0.38	4	No	Yes
Organic Solvent Containing eluents	0.1-0.5	OFF	Chemical Suppression Mode ONLY	

Matching the Current Setting to the Eluent Concentration and Flow Rate

Eluent	Eluent Flow Rate (mL/min)	Current Setting	Recycle Mode	External Water Mode
Cation Self-Regenerating Suppressor (CSRS-I, 4mm)				
5-25 mM MSA	0.5-1.0	2	Yes	Yes
	1.1-2.0	3	Yes	Yes
25-50 mM MSA	0.5-1.0	3	Yes	Yes
50-100 mM MSA	0.5-1.0	4	Yes	Yes
All HCl/DAP, HNO_3 or Organic Solvent Containing Eluents	0.5-2.0	OFF	Chemical Suppression Mode <u>ONLY</u>	
Cation Self-Regenerating Suppressor (CSRS-I, 2mm)				
5-25 mM MSA	0.1- 0.35	2	Yes	Yes
	0.36 - 0.5	3	Yes	Yes
25-50 mM MSA	0.1- 0.35	4	Yes	Yes
All HCl/DAP,HNO_3 or Organic Solvent Containing Eluents	0.1- 0.05	Off	Chemical Suppression Mode <u>ONLY</u>	

Current	
1	50
2	100
3	300
4	500

Suppressor Cleaning

Since the suppressor contains a membrane with active sites, those sites can become occupied by contaminants. Cleaning the suppressor may restore the performance of the system. Signs of a contaminated suppressor include:

1.

2.

3.

4.

To clean a suppressor, a cleaning solution is pumped across the membrane. The following table lists appropriate cleaning solutions.

TYPE OF CONTAMINANT	CLEANING SOLUTION
Organics	90% ACN/MeOH in deionized water
Metals	1.0 M HCl/.1 M KCl
Base-soluble	1 M NaOH
Acid-soluble	1 M HCl

Once a cleaning solution is chosen, use the following procedure to clean the suppressor membrane.

Suppressor Cleaning

1. Turn off SRS Power Supply Control Unit
2. Disconnect guard and separator columns from the injection valve and the Suppressor.
3. Turn off the regenerant, and disconnect the line from the suppressor REGEN IN port.
4. Disconnect the liquid line from the suppressor ELUENT OUT at the cell, and reconnect it to the REGEN IN port.
5 Connect a container of cleaning solution (see above table) to the Eluent supply line leading to the pump.
6. Pump the cleaning solution at 1-2 mL/min for 30 min.
7. Flush columns and suppressor separately with deionized water for 10 minutes..
8. Reinstall columns, pump eluent through the system for equlibration.

Columns

1. Resolution is the measure of _______________ between two peaks.

2. Guard column ____________ is a measure of the number of runs it takes for a guard column to lose 50% of its capacity.

3. When cleaning columns, it is important to maintain the __________ direction.

4. When cleaning a guard column and separator column at the same time, always plumb the guard column _____________ the separator column.

Suppressor

5. The purpose of _____________ is to reduce the eluent background, while increasing the analyte response.

6. Autosuppression uses the _____________ of water to form the hydronium or hydroxide ion to be used in the exchange process.

The following are a list of true/false statements that deal with columns and suppression.

1. ________ Chromatography is the separation of components in a mixture by partitioning between a mobile phase and the analytes.

2. ________ Resolution is affected by efficiency, capacity and selectivity.

3. ________ Guard columns are short columns packed with a different resin the separator column in order to better eliminate contamination.

4. ________ OnGuard Cartridges are used for pretreatment of the eluent.

5. ________ Guard column exhaustion is a measure of the number of runs that can be made through

6. ________ Decreasing retention times and loss of resolution are two major signs of a deteriorating column.

7. ________ When cleaning columns, always reverse the flow direction though the columns to be cleaned.

8. ________ High system backpressure could be a sign of clogged bed supports.

9. ________ Chemical suppression reduced the eluent background conductivity while converting the analytes to their salt forms.

10. ________ Autosuppression uses electrolysis and electrodialysis chemistry to create exchange ions.

11. ________ Autosuppressors should be run in the recycle mode when using organic eluents.

12. ________ When cleaning a suppressor, the ELUENT OUT line should be plumbed directly into the REGEN IN port on the suppressor.

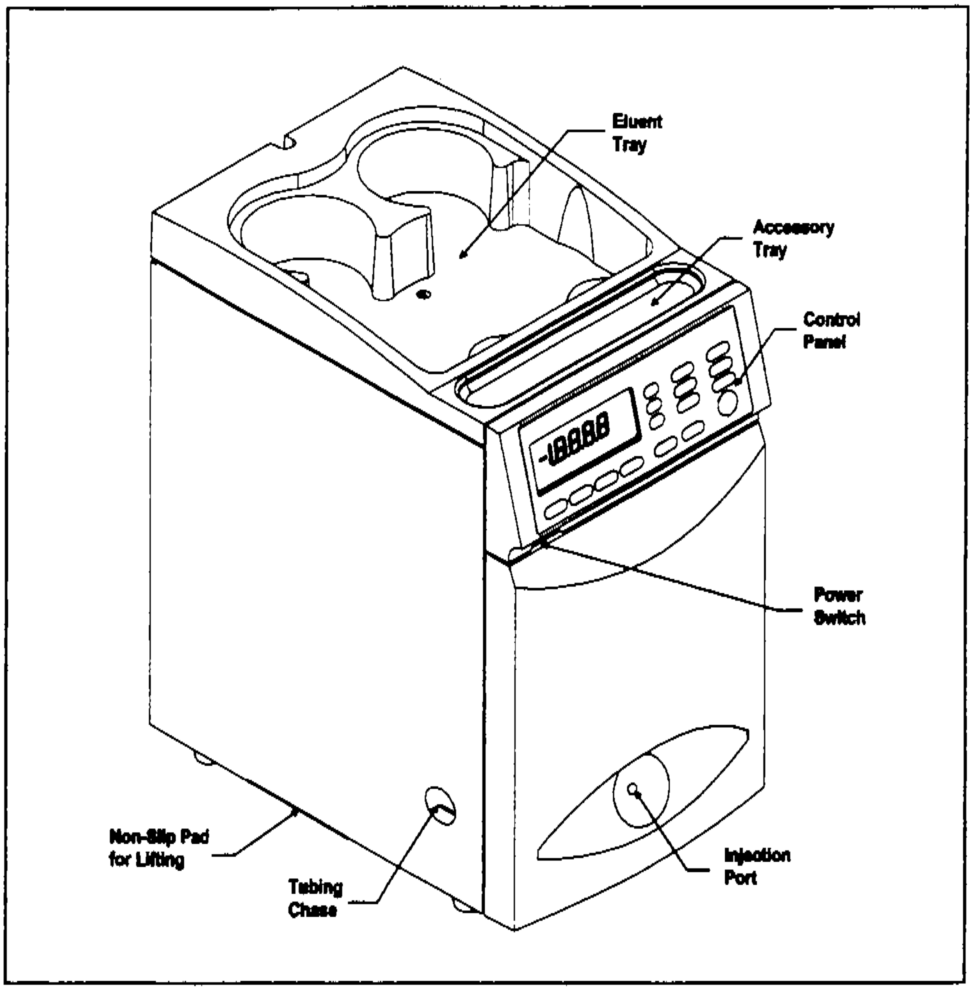
Objectives

By the end of this section, each participant should be able to:

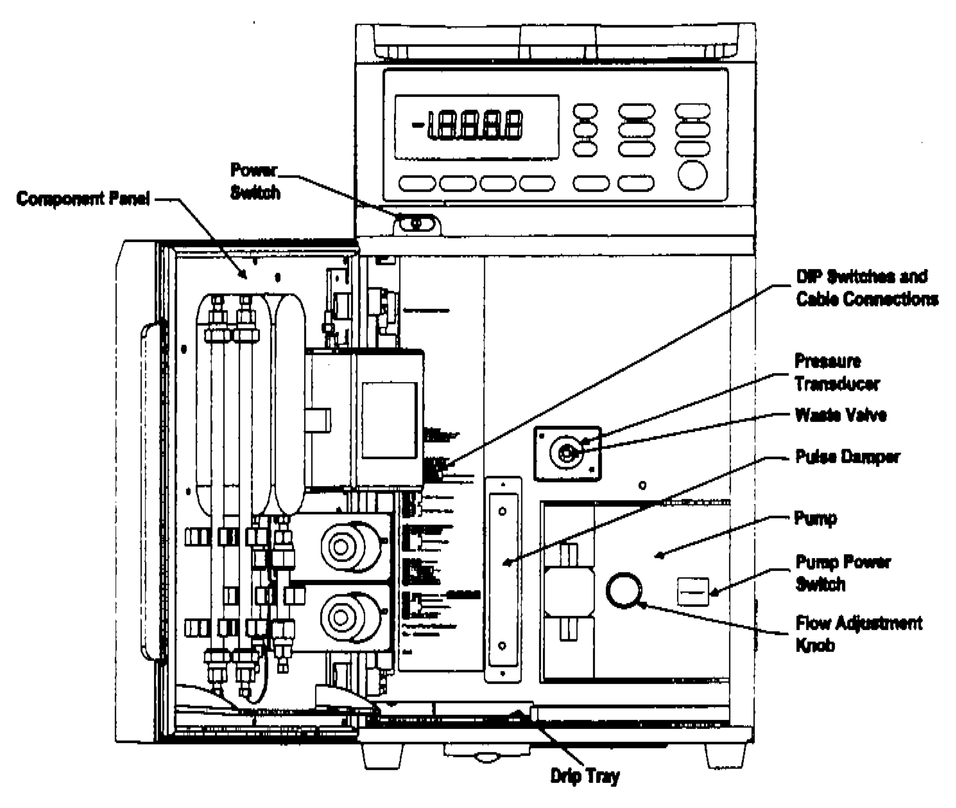
- Describe the components within the DX-120 and trace the flow path
- Install a single column/single eluent DX-120
- Operate a DX-120 using front panel control

Installation Overview

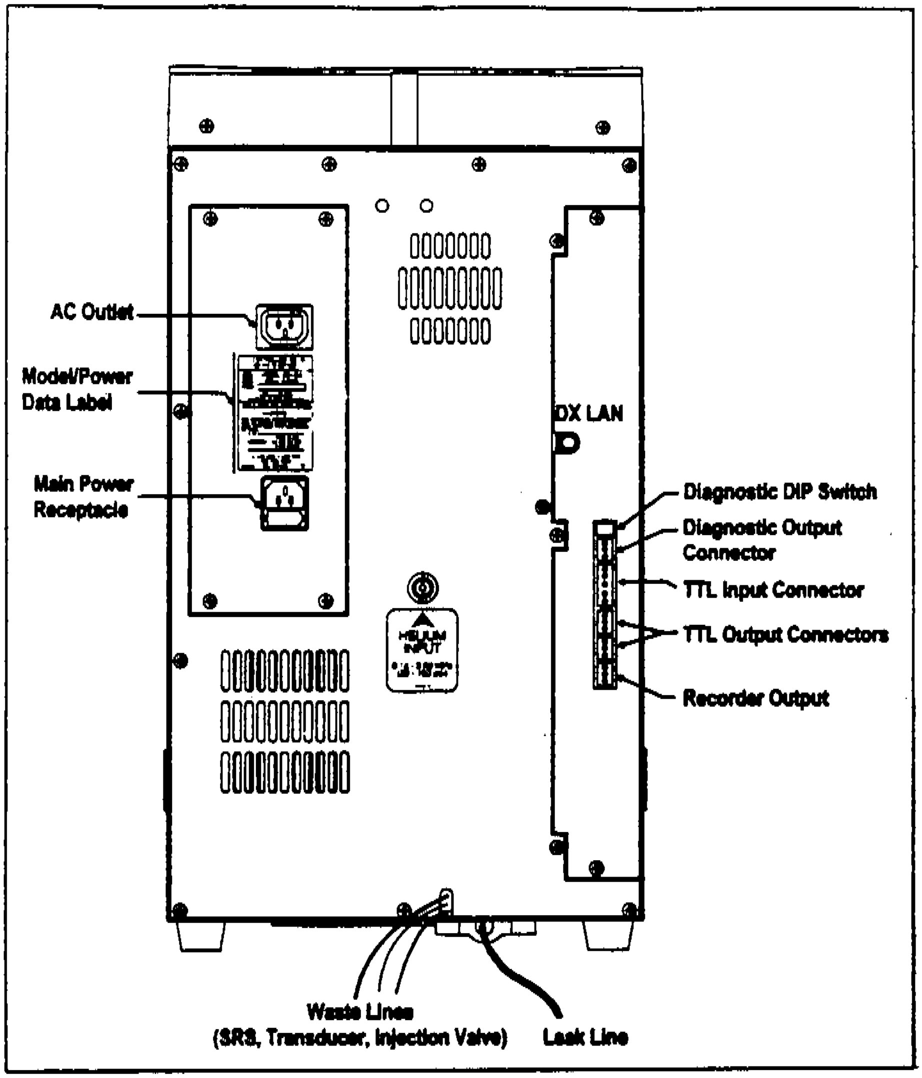
- Connect the power cord
- Place the waste lines in a waste container
- Connect the gas supply line
- (Optional) Connect the DX LAN network cable
- Connect the eluent reservoir(s)
- Set up the pump
- Install the chromatography components (SRS and columns)
- (Optional) Connect an autosampler
- (Optional) Connect a chart recorder or integrator
- Set the configuration DIP switches



DX-120 Operating Features (Exterior)



DX-120 Operating Features (Interior)



DX-120 Rear Panel

The following is a description of the flow path through the DX-120 and a description of the front panel.

CHROMATOGRAPHY STREAM

Eluent Flow

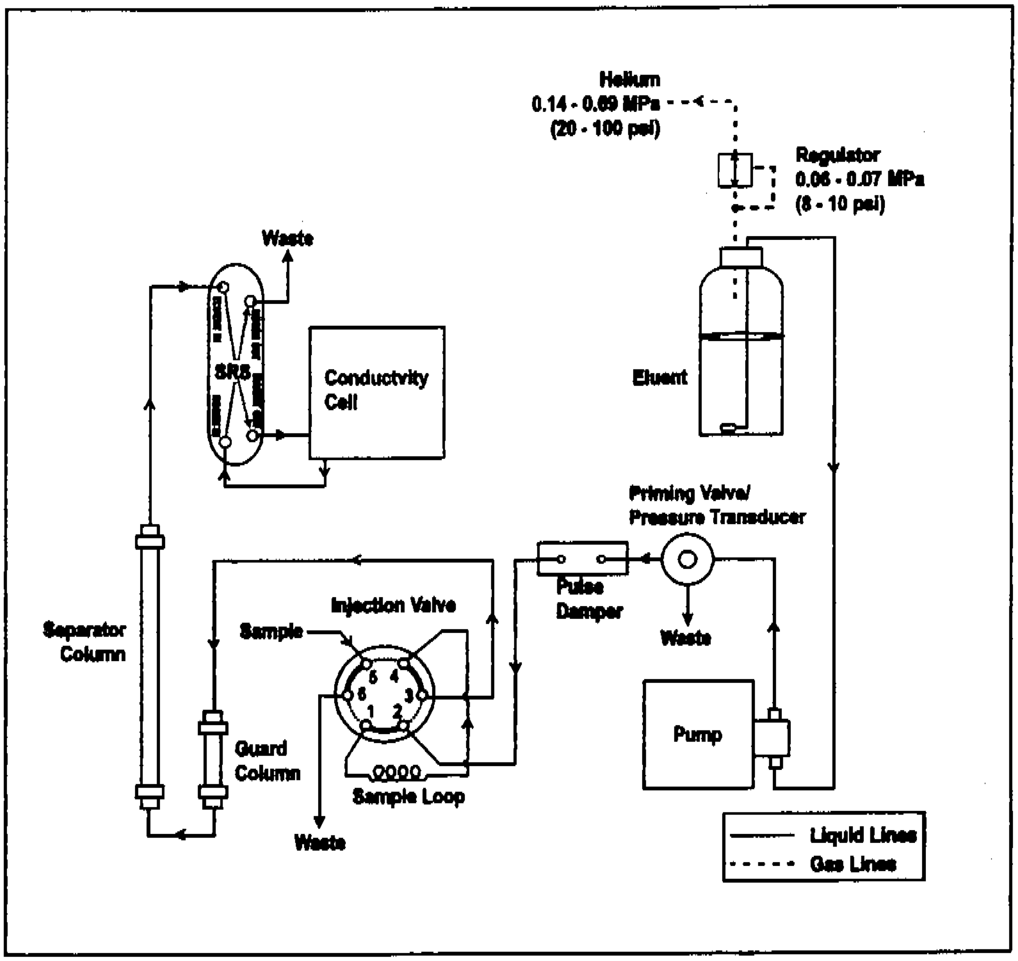
The eluent flows from the eluent bottles, in the upper portion of the system cabinet, to the pump. The eluent is then pumped through the priming valve assembly and pulse damper to the injection valve. A sample is loaded into the injection valve in a fixed volume loop, and is then injected into the chromatography stream. The Stream then goes to the guard column and the separator column. When using chemical eluent suppression, the stream flow is from the separator column to the suppressor and then to the detector cell. If you use a non-suppressed application, the chromatography stream goes directly from the separator column to the cell for detection.

Regenerant Flow

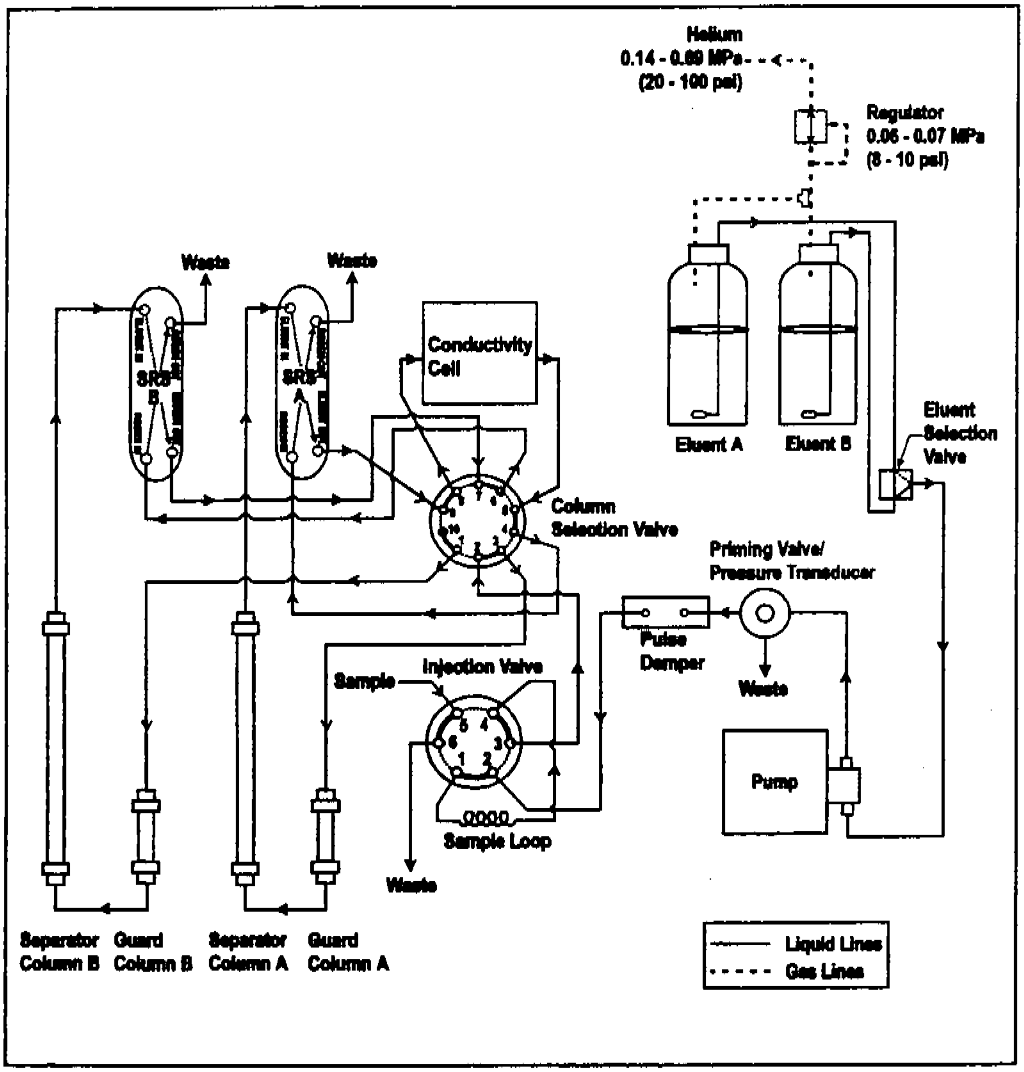
Suppressor regenerant enters the system in several ways. In the case of external water or chemical suppression it can be pumped from an outside AutoRegen unit through the lower rear panel of the DX-120 into the REGEN IN port of the suppressor and out the REGEN OUT port to a waste container. Instead of from the AutoRegen unit, suppressor regenerant can also come from a regulated pressurized regenerant bottle located outside the system.

In the case of the self-regenerating suppressor mode, the line from the detector cell outlet is connected to the REGEN IN port of the suppressor and the REGEN OUT port is directed to the waste container.

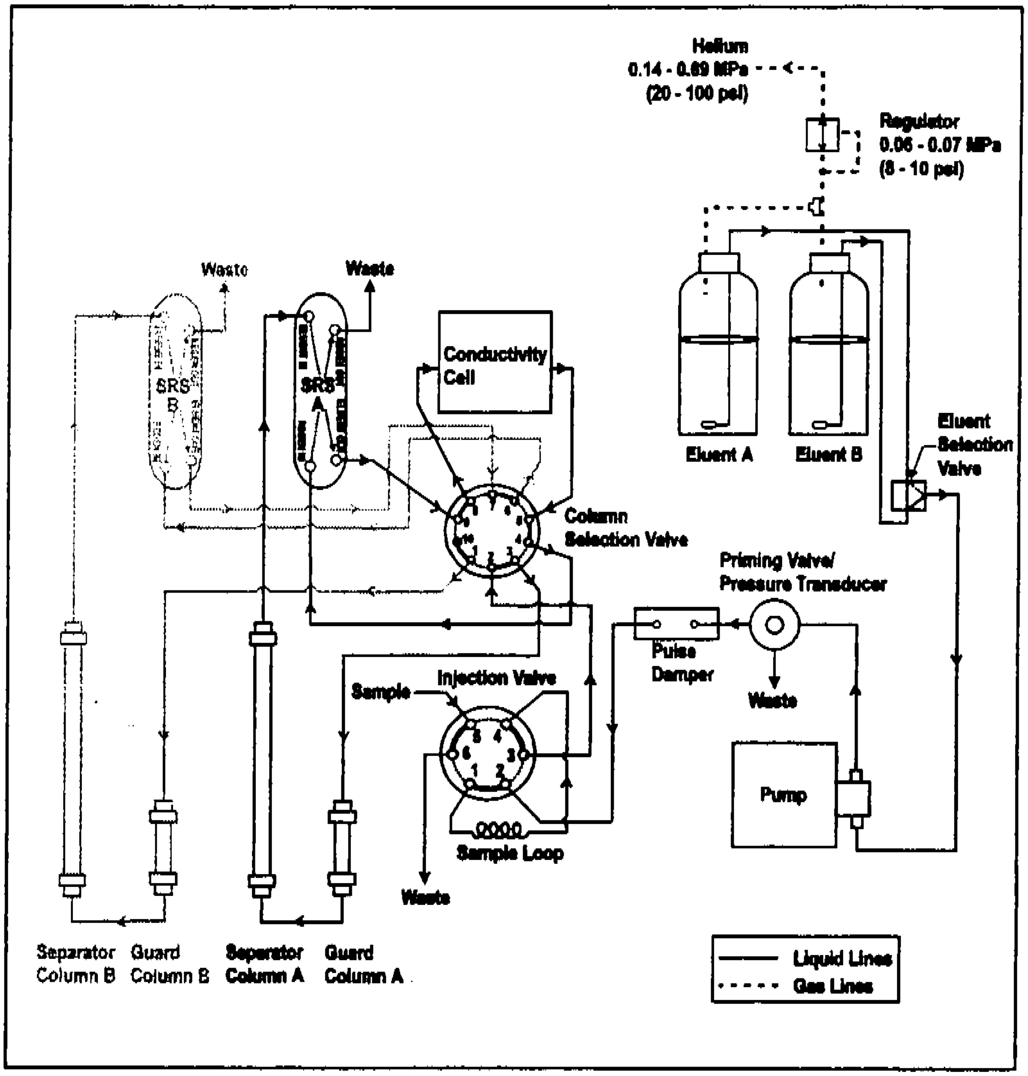
Consult your Suppressor instruction manual for further information on self-regeneration or external water mode of suppression.



DX-120 Flow Schematic: Single-Column



DX-120 Flow Schematic: Dual-Column System
Column Select Mode



DX-120 Flow Schematic: Dual-Column System Eluent Select Mode (Column Set A Active)

Eluent Reservoir Connections

- Untape the coil of red air tubing from the reservoir tray on the top of the DX-120. Push the end of the tube firmly onto the barbed connector on the eluent reservoir cap assembly.
- Untape the coil of white eluent tubing from the reservoir tray and screw the tube end-fitting securely into the eluent fitting on the reservoir cap.

Pump

NOTE

Always filter eluents through a 0.45mm filter to remove small particulates that may contaminate the pump. Install an end-line filter (P/N 045987) on the end of each eluent reservoir line. Filters are supplied in the pressurizable reservoir Ship Kits. *See the Pressurizable Reservoir Installation Instructions* **for details.**

Priming the Pump

1. Fill the eluent reservoir with the eluent required for your application or with deionized water if you will be calibrating the pump flow rate.
2. Lower the outlet line of the reservoir cap assembly into the reservoir. Handtighten the cap.
3. Press **Eluent Pressure** on the front control panel to pressurize the reservoir.
4. Open the pressure transducer waste valve by turning the knob counterclockwise two turns. When the valve is open, eluent flows directly from the pump to waste.
5. Press **Pump** on the front control panel to turn on the pump. Press **Flow Setting** to display the pump flow rate.
6. Pull out the pump flow adjustment knob and adjust the flow rate to 2.0 mL/min. Push the knob back in.

7. After about 10 seconds, adjust the flow rate to the setting required for your application.

8. Close the pressure transducer waste valve and let the system flush and equilibrate for 10 minutes.

Checking the Pump Flow Rate Calibration

Check the pump flow rate calibration at installation and whenever you change applications.

1. Disconnect the line from port 3 of the injection valve if it is currently connected to either the guard column or the column switching valve.

2. Connect an 11 MPa (1600 psi) backpressure device, or a piece of narrow bore peek tubing that will provide 11MPa (1600 psi) of backpressure, to port 3 on the injection valve. Route the other end of the backpressure tubing to waste.

3. Fill the eluent reservoir with deionized water.

4. Turn on the pump and set the flow rate to 1.2 mL/min.

5. Pump deionized water through the system for at least 15 minutes to allow the system to equilibrate.

6. Collect the water into a tared beaker for 5 minutes. Weigh the collected water. It should be 6.00 ±0.05 g.

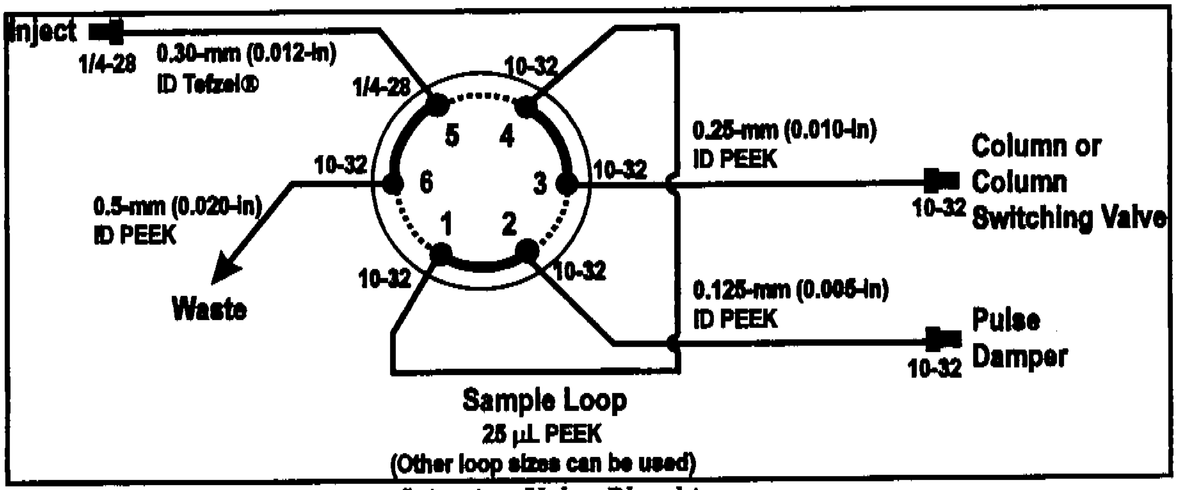
7. If the collected water is not within the above range, see flow rate calibration instructions.

Injection Valve Connections

NOTE

The following information is included for your reference, should you need to replace any tubing or fittings.

The injection valve is plumbed at the factory with all tubing and fittings for connections to the pump, injection port, and column (or to the column switching valve in dual-column systems). A 25mL PEEK sample loop (P/N 042857) is installed between ports 1 and 4.Other sample loop sizes are available. Contact your Dionex office for information.



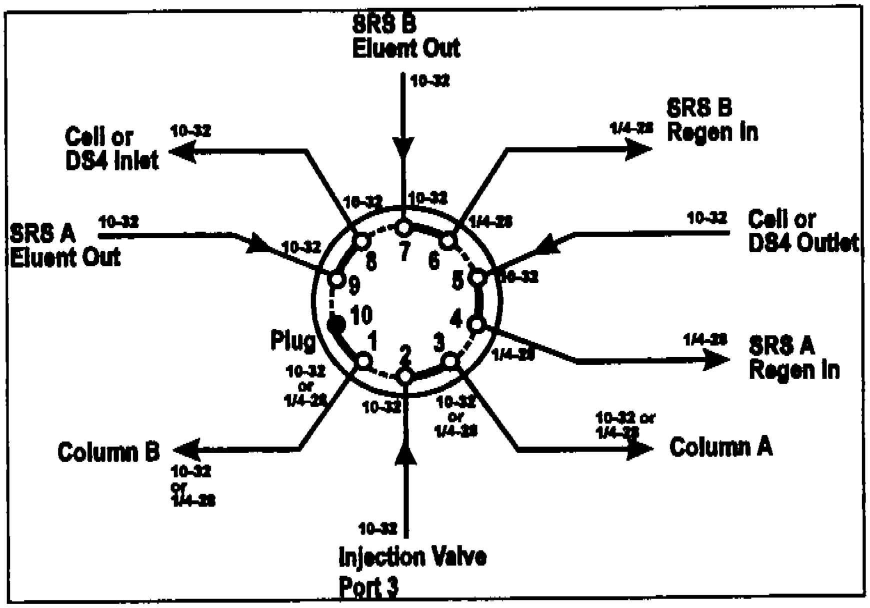
Injection Valve Plumbing

Column Switching Valve Connections (Optional)

NOTE

The following information is included for your reference, should you need to replace any tubing or fittings.

The column switching valve for dual-column configurations is plumbed at the factory with the necessary tubing and fittings to connect the valve to the columns, suppressors, injection valve, and cell.



Column Switching Valve Plumbing

Connections to Chromatography Components

The DX-120 injection valve, column switching valve (if installed), pump, and cell or DS4 connections are plumbed before shipping. The DX-120 Ship Kit contains tubing assemblies for completing the SRS and column connections.

NOTE

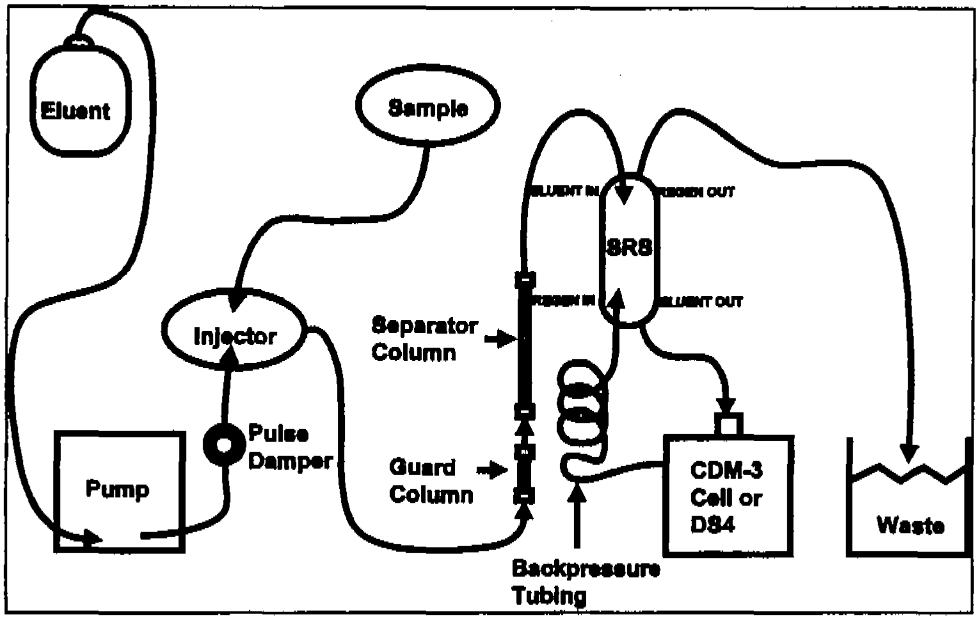
For shipping purposes, unions are installed between unconnected lines from the cell and valves. Remove these unions before beginning the SRS and column installation.

The DX-120 can be plumbed for three operating modes:

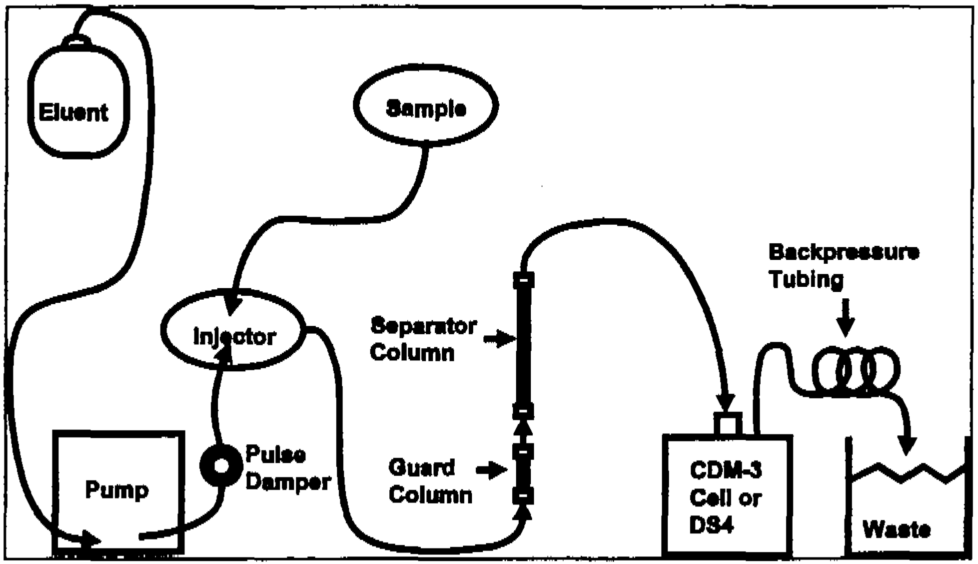
- AutoSuppression ™ Recycle mode (SRS required).
- Nonsuppressed mode, (Conductivity measurements made when no suppressor is in-line may show noticeable baseline drift).
- AutoSuppression with External Regenerant mode. This mode is required if the eluent contains organic solvents.

This section contains installation instructions for the AutoSuppresion and Nonsuppressed modes. Refer to the SRS manual for information about using the AutoSuppression with External Regenerant mode.

For this course, we will plumb the DX-120 for autosuppression recycle mode.



Auto Suppression Recycle Mode



Nonsuppressed Mode

Column Installation

Before installing the column(s) in the DX-120, refer to the column manual for specialized installation and start-up instructions.

Column Setup

1. Before installing the separator column, pump the eluent required for your application through the injection valve at 1.0 to 3.0 mL/min for 2 to 5 minutes to clear any air from the liquid lines. Trapped air reduces the column efficiency.

2. Switch the position of the injection valve between **INJECT** and **LOAD** several times to allow water to flow through the sample loop and each valve port.

3. Set the Low Pressure Alarm DIP switch (SW1-8) to off. Set the flow rate to 2.0 mL/min and verify that the pressure through the system, with no columns installed, is less than 690 KPa (100 psi). Reset the Low Pressure Alarm switch to on.

4. Stop the pump.

5. Remove the end plugs from the separator column(s) and store them in a safe place. You must reinstall the end plugs in the column(s) before placing them in storage.

6. If they are not already installed, install the column mounting clips (P/N 046699) on the component panel. Orient each clip with the larger clasps toward the front of the panel; squeeze the open ends of the clip together, and insert them into the square opening. Install two clips for the separator column(s) and one for the guard column(s).

NOTE

An arrow on each column indicates the liquid flow direction. It points away from the column inlet, toward the column outlet.

7. Orient the guard column with the outlet facing down and snap the column into its clip. For dual-column systems, snap the back column in first and then the front.

8. Orient the separator column with the outlet facing up and snap the column into its clip. For dual-column systems, snap back column in first and then the front.

The remaining steps depend upon whether you are installing one or two column sets.

Note:
For this course, we are installing only one column.

Single-Column Set Connections

1. Connect the line from port 3 of the injection valve to the inlet of the guard column.

2. Connect the outlet of the guard column to the inlet of the separator column. Use 0.25-mm (0.010-in) ID tubing (P/N 042690), 10-32 fittings (P/N 043275), and ferrules (P/N 043276).

3. For AutoSuppression mode, connect the outlet of the separator column to the SRS ELUENT INLET. For Nonsuppressed mode, connect the outlet of the separator column to the cell or DS4 inlet. Use 0.25-mm (0.010-in) ID tubing (P/N 042690), 10-32 fittings (P/N 043275), and ferrules (P/N 043276).

Dual-Column Set Connections (Optional)

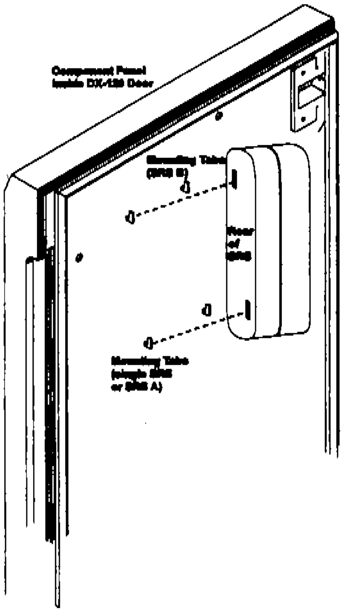
1. Connect the line from port 1 of the column switching valve to the inlet of guard column B.
2. Connect the line from port 3 of the column selection valve to the inlet of guard column A.
3. Connect the outlet of guard column A to the inlet of separator column A. Use 0.25-mm (0.010-in) ID tubing (P/N 042690), 10-32 fittings (P/N 043275), and ferrules (P/N 043276). Repeat for guard and separator columns B.
4. Connect the outlet of separator column A to the **ELUENT INLET** of SRS A and connect the outlet of separator column B to the **ELUENT INLET** of SRS B. Use 0.25-mm (0.010-in) ID tubing (P/N 042690), 10-32 fittings (P/N 043275), and ferrules (P/N 043276).

Self-Regenerating Suppressor (SRS) Installation

NOTE

The instructions here do not replace the instructions in your SRS manual. For complete SRS installation instructions, and for the initial SRS start-up procedure, please refer to the SRS manual.

The SRS mounts on a bracket tabs on the component panel. Orient the SRS with the **REGEN OUT** port and the cable at the top; align the slots on the back of the SRS with the tabs on themounting bracket. Press in, and then down, to lock the SRS in place. Lift up and pull out to remove the SRS.



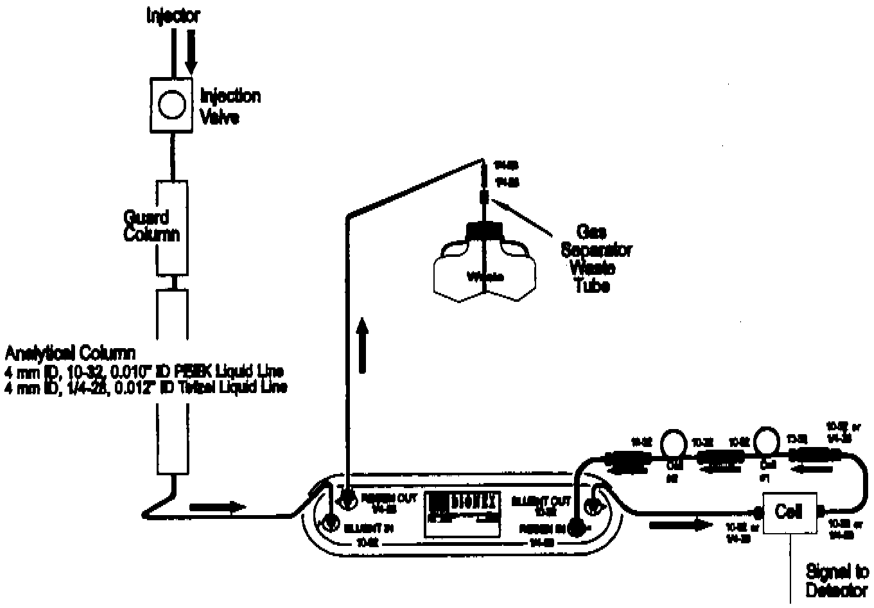
Installing the SRS

The DX-120 Ship Kit includes certain items required for SRS installation:

- SRS gas separator waste tube (P/N 045460)
- Backpressure coils for 4 mm SRS (P/N 045877)

The SRS plumbing connections depend on whether you are installing one or two column sets. Refer to the appropriate following section.

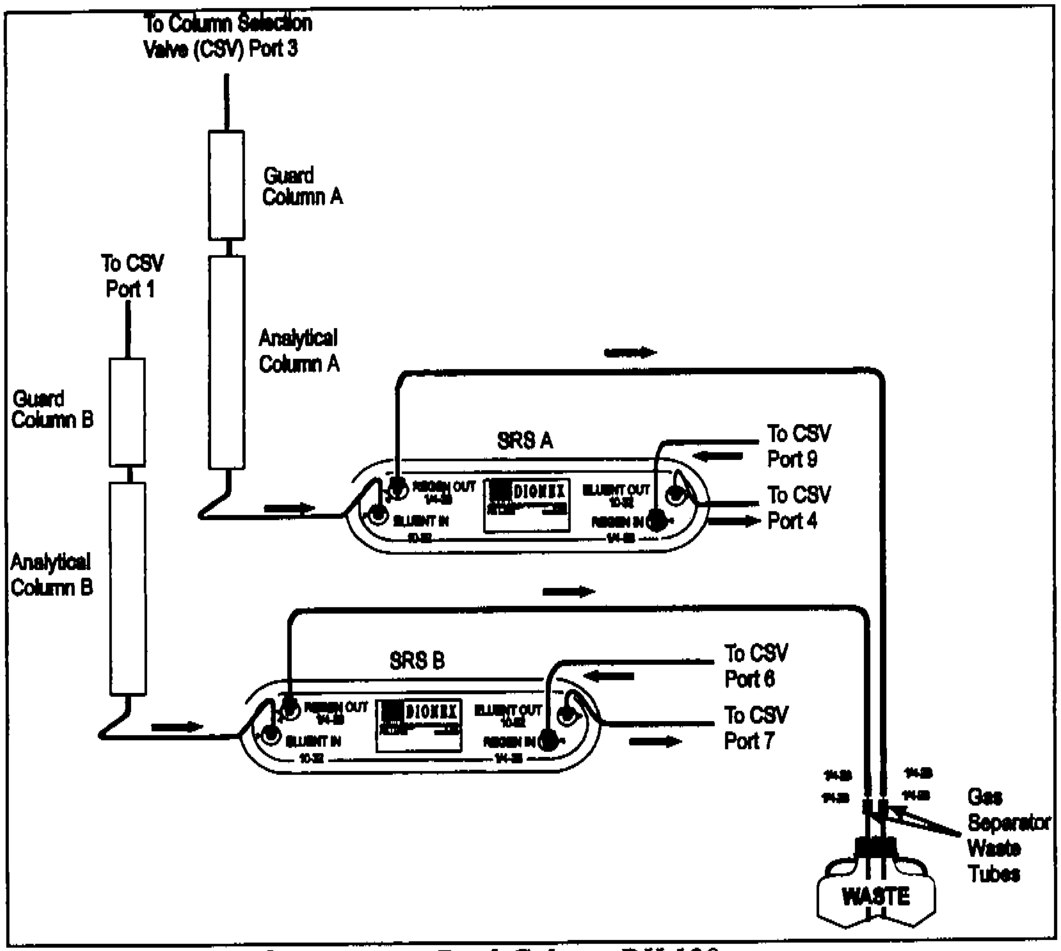
Single-Column SRS Installation



SRS Connections: Single-Column DX-120

1. Connect the line from the cell or DS4 outlet to the SRS **REGEN INLET**.
2. Connect the line from the cell or DS4 inlet to the SRS **ELUENT OUTLET**.
3. Connect backpressure tubing and a waste line to the SRS **REGEN OUTLET**. Refer to the SRS manual for instructions.

Dual-Column SRS Installation (Optional)



SRS Connections: Dual-Column DX-120

1. Connect the line from port 4 on the to the **REGEN INLET** of SRS A.

2. Connect the line from port 6 to the **REGEN INLET** of SRS B.

3. Connect port 9 to the **ELUENT OUTLET** of SRS A.

4. Connect port 7 to the **ELUENT OUTLET** of SRS B.

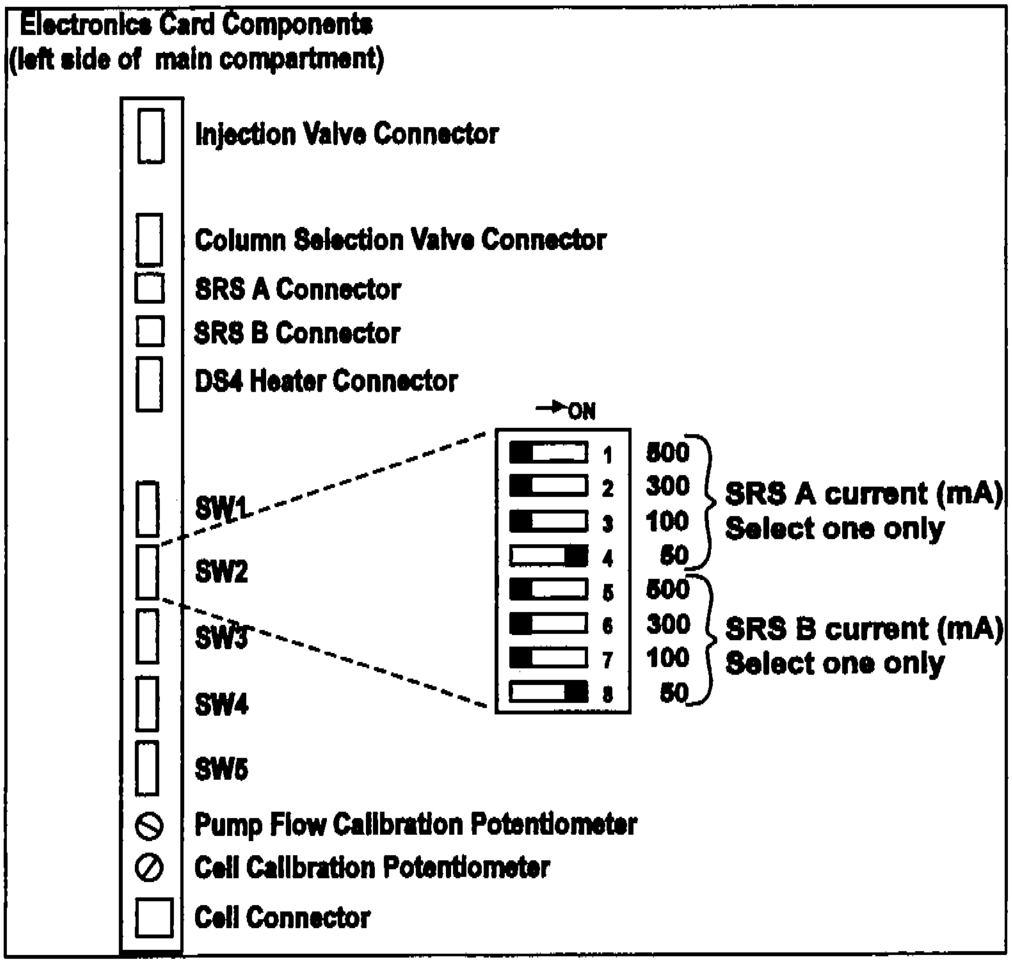
5. Connect waste lines to the **REGEN OUTLET** ports on both suppressors. Refer to the SRS manual for instructions.

Completing the SRS Installation

1. For a single-column system, plug the cable from the SRS into the matching cable extending from the SRS A connector on the electronics card.

   For a dual-column system, also plug the cable from SRS B into the matching cable extending from the SRS B connector.

2. Select the SRS current setting(s) with SW2. Refer to the SRS manual for the recommended setting for your application.



Electronics Card Connections

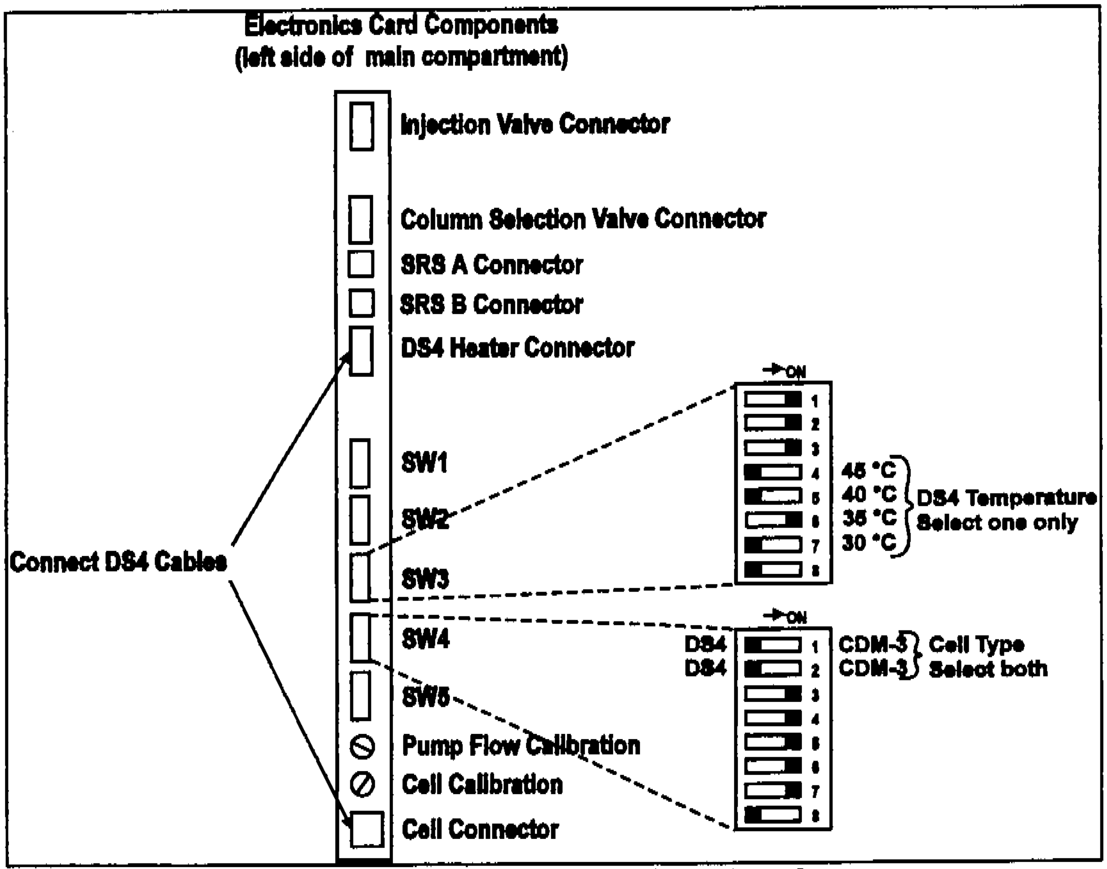
DS4 Detection Stabilizer Installation

If the DS4 is ordered at the same time as the DX-120, it is installed in the DX-120 at the factory before shipping. Follow the installation instructions below when you order a DS4 separately.

1. Position the keyhole slots on the rear of the DS4 over the shoulder washers on the DX-120 component panel, then pull the DS4 down into position.

2. Route both DS4 cables to the electronics card at the left rear of the main compartment.

3. Plug the connectors on the cables into the appropriate jacks on the electronics card



DS4 Cable Connections and DIP Switches

4. Connect the eluent outlet of the column or suppressor to the DS4 inlet, using 0.25-mm (0.010-in) ID tubing (P/N 042690), 10-32 fittings (P/N 043275), and ferrules (P/N 043276).

5. Connect the outlet of the DS4 (the 10-32 coupler) to either the SRS **REGEN IN** port (for AutoSuppression Recycle mode) or to the backpressure line leading to waste (for Nonsuppressed mode).

6. Direct the DS4 spill/overflow tubing to the spill tray at the bottom front of the DX-120.

7. Select a DS4 temperature set point (SW3-4, 5,6, or 7). Select a temperature at least 5 °C above the highest expected ambient temperature.

8. Set the Cell Type DIP switches (SW4-1, 2) to the off (DS4) position

Backpressure Requirements

All detector cells require enough backpressure to prevent eluent in the cell from degassing due to abrupt volume changes between the small inner diameter of the connecting tubing and the relatively larger volume of the cell. Degassing creates bubbles in the cell and disrupts detector responsiveness.

The DX-120 Ship Kit includes backpressure coils (P/N 045877) for installation after the cell outlet. Table B-1 indicates the number of coils to install, depending on the application flow rate and the type of system.

Flow Rate (mL/min)	Tubing ID	Coil Length	Number of Coils Single-column Systems	Number of Coils Dual-Column Systems
0.5 to 1.5	0.25-mm (0.010-in)	0.76 M (2.5 feet)	2	1
1.5 to 3.0	0.25-mm (0.010-in)	0.76 M (2.5 feet)	1	0

Table B-1. Coils for Backpressure Requirements

In the AutoSuppression mode, the backpressure generated by the DS4 (if installed), cell, and backpressure tubing is applied to the SRS. For example, with 1 meter of 0.25-mm (0.01-in) ID tubing at a flow rate of 1.0 mL/min, the backpressure is about 0.21 MPa (30 psi). The optimal backpressure is 0.28 MPa (40 psi). Refer to the SRS manual for details about SRS backpressure requirements.

In the Nonsuppressed mode, the backpressure generated by the backpressure tubing is applied to the cell. The optimal backpressure is 0.21 to 0.34 MPa (30 to 50 psi).

Integrator Connections

Chart Recorder Connections

1. Connect one end of the Recorder Signal Wire assembly (P/N 043598) to the Recorder Output connectors on the DX-120 rear panel.

2. Connect the other end to the appropriate input connectors on the chart recorder.

4400 or 4600 Integrator Connections

To use the integrator as a recorder with integration functions:

1. Connect one end of the Recorder Signal Wire assembly (P/N 043598) to the Recorder Output connectors on the DX-120 rear panel

2. Connect the other end to the 4400 or 4600 Integrator input connectors.

Configuration DIP Switch Settings

The DIP switches on the left side of the main compartment set various DX-120 operating parameters. The factory default settings are appropriate for most applications.

NOTE

The DIP switch labeled DIAGNOSTIC TESTS on the rear panel is for service procedures only. Do not change any of its settings unless directed to do so by a Dionex Service representative. For routine operation, all settings are off.

Switch Set	Default Settings	→ON			
SW1		□■	1	Auto Inject Reset	
		□■	2	Pump Time-out	
	Single	□■	3	Dual — Column Configuration	
	MPa	■□	4	psi — Pressure Units	
		□■	5	24.1 MPa (3500 psi	High Pressure Alarm Select one only
		■□	6	17.3 MPa (2500 psi)	
		■□	7	13.8 MPa (2000 psi)	
		□■	8	2.0 MPa (250 psi) Low Pressure Alarm	
SW2		■□	1	500	SRS A Current (mA) Select one only
		■□	2	300	
		■□	3	100	
		□■	4	50	
		■□	5	500	SRS B Current (mA) Select one only
		■□	6	300	
		■□	7	100	
		□■	8	50	
SW3		□■	1	Column Select Mode	
		□■	2	Alarm Sound	
		□■	3	Auto Offset	
		■□	4	45 °C	DS4 Temperature Select one only
		■□	5	40 °C	
		■□	6	35 °C	
		■□	7	30 °C	
		■□	8	Reserved 1	
SW4	DS4	□■	1	CDM-3	Cell Type Select both
	DS4	□■	2	CDM-3	
	100 µS	□■	3	1000 µS Range	Recorder Settings
	10 V	□■	4	1 V — Full Scale Voltage	
	0%	□■	5	Offset 10%	
	Neg (-)	□■	6	Pos (+) — Polarity	
		□■	7	Inject Mark	
		■□	8	Reserved 2	
SW5		□■	1	0.5 sec ■□ 1 · 1.0 sec □■ 1 · 2.0 sec ■□ 1 · 4.0 sec □■ 1	Response Time
		■□	2	0.5 sec ■□ 2 · 1.0 sec ■□ 2 · 2.0 sec □■ 2 · 4.0 sec □■ 2	
		□■	3	Filter Enable	
		■□	4	1.9%	Signal Temperature Compensation Select one only
		□■	5	1.7%	
		■□	6	1.5%	
		■□	7	Reserved 3	
		■□	8	Test Cell	

KEY

□■ = ON—Selects function on right

■□ = OFF—Turns off function on right or selects function on left (if any)

Default DIP Swithch Settings

DIP Switch Function	Description
Auto Inject Reset **(SW1-1)**	When on, the injection valve automatically returns to the load position 60 seconds after it is switched to the inject position.
Pump Time-out **(SW1-2)**	When on, if the DX-120 is idle for 90 minutes, the pump flow is reduced to 1/20th of its current rate. The LED on the **Pump** button flashes when the pump flow is reduced. Press any button to resume the last selected flow rate.
Column Configuration (SW1-3)	Selects the column configuration: dual (on) or single (off).
Pressure Units **(SW1-4)**	Selects the pressure units (psi or MPa) shown on the screen.
High Pressure Alarm (SW1-5, 6, 7),	Selects at what pressure the high pressure alarm occurs.
Low Pressure Alarm (SW1-8)	Enables an alarm when a low pressure condition (2.0 MPa or less) occurs.
SRS A Current **(SW2-1, 2, 3, 4),**	Selects the operating current for the SRS (or SRS A in a dual-column system).
SRS B Current **(SW2-5, 6, 7, 8),**	Selects the operating current for SRS B in a dual-column system.
Column Select Mode **(SW3-1)**	For dual-column configurations (SW1-3 on). When on,, selects the column select mode . When off, selects the eluent select mode.
Alarm Sound **(SW3-2)**	Enables or disables the alarm tone.
Auto Offset **(SW3-3)**	When on, an auto offset is performed when injection occurs.
DS4 Temperature **(SW3-4, 5, 6, 7)**	Selects the DS4 operating temperature. Select a temperature at least 5 °C above the highest expected ambient temperature.

DIP Switch Function	Description
Cell Type **(SW4-1, 2)**	Select the type of cell (CDM-3 or DS4) installed in the DX-120. Both switches must be set.
Range **(SW4-3)**	Selects the microSiemens output (100mS or 1000mS) to the recorder, of a full-scale detector response.
Full-Scale Voltage **(SW4-4)**	Selects the voltage output (1 V or 10 V) to the recorder, of a full-scale detector response.
Offset **(SW4-5)**	When on, the analog output is offset 10% of full-scale. The 10% offset allows the recording device to plot the signal if it becomes negative.
Polarity **(SW4-6)**	Selects whether the output signal to the recording device is positive or negative. In nonsuppressed applications in which the analyte output is lower than the background conductance, the polarity must be inverted to have peaks instead of dips
Inject Mark **(SW4-7)**	When on, a mark 10% of full-scale is sent to the recording device when the injection valve switches to the inject position.
Response Time **(SW5-1, 2)**	When analog filtering is enabled, on/off combinations of these switch positions select the filter response time.
Filter Enable **(SW5-3)**	Enables or disables filtering of the analog output. Filtering is typically needed only for nonsuppressed applications, which have higher background and noise.
Signal Temperature Compensation **(SW5-4, 5, 6)**	Selects by how much to adjust the value of the temperature coefficient used by the detector.
Test Cell **(SW5-8)**	Allows testing of the cell electronics for troubleshooting purposes.

Operation

During this exercises you will install and operate a DX-120

1. Verify that the DX-120 has nitrogen connected to it and that the nitrogen pressure is 100 psi.

2. Verify that: a)the DX-120 has power running to it; b) the DX-120 is connected to a data collection device; and c) you have a suitable waste container.

3. Plumb the entire system from eluent bottle to waste connector bottle. Refer any questions to the instructor.

4. After the system has been correctly plumbed, pressurize the eluent and prime the pump.

5. Verify that the pump is set to the correct flow rate for the separation you are going to perform. start the pump.

6. If you are using a self-regenerating suppressor, verify that the current is set to the correct value.

7. Allow the system to equilibrate while you monitor the system pressure and conductivity.

8. After the system has equilibrated, obtain a standard from the instructor and inject the standard into your system.

9. To run a standard properly, load the standard into the sample loop with the injection valve in the **LOAD** position. Press the **AUTO OFFSET** button. Finally, press the **LOAD/INJECT** button while simultaneously starting the data collection device.

EXERCISE

DAY 1

Configure the system appropriately to answer the following:

1. What is the conductivity of non-suppressed deionized water?
2. What is the conductivity of non-suppressed eluent?
3. What is the conductivity of suppressed eluent?

Perform the following analyses:

1. Run a 1:10 and 1:100 dilution of the standard supplied by you instructor.
2. Prepare a 2 point calibration curve for each of the components in your standard.

DAY 2

1. Repair the problems in your system. To verify proper performance. run one of the diluted standards from the previous day to compare results.

2. Clean the column following the suggested procedure on parge 2-17. Use a 20x concentration of your eluent to clean. Re-equilibrate the column with operating eluent.

This section is a guide to troubleshooting problems that may occur while operating the DX-120. If an alarm sounds, check Section for possible causes. If an error code is displayed, check Section for the possible causes. To resolve other problems, turn to the section that best describes the operating problem. There, you will find the possible causes of the problem listed in order of probability. If you cannot eliminate a problem on your own, notify your Dionex office.

Alarms

Three events signal an alarm condition: a tone sounds, the LED on the **Alarm Reset** button blinks, and the display indicates ALARM and the alarm's source. To clear the alarm, press **Alarm Reset**. If the alarm condition still exists the alarm will reappear after 15 seconds. The alarm tone can be disabled with a DIP switch (SW3-2).

- **LEAK ALARM**

There is liquid in the drip tray. Wipe up spills in the tray and check for leaks.

- **CELL LEAK ALARM**

There is a leak in the DS4.

- **LOW PRESSURE ALARM**

The system pressure is less than or equal to the low pressure limit of 1.7MPa (250psi). This condition automatically turns off the pump and SRS. This alarm can be turned off with a DIP switch (SW1-8).

1. Make sure the eluent reservoirs are full.

2. Check for liquid leaks.

3. Make sure the pressure transducer waste valve is closed. To close the valve, turn the knob clockwise, just until tight. **Do Not Overtighten! Overtightening may damage the valve and the pressure transducer housing.**

4. Restart the pump.

5. Prime the pump.

- **HIGH PRESSURE ALARM** ALARM: PRESSURE HI

The system pressure is greater than or equal to the high pressure limit selected (SW1-5, 6, 7). This condition automatically turns off the pump and SRS.

1. Make sure the selected flow rate is not too high.

2. Check for blockages in the liquid line from the pump pressure transducer to the waste container.

3. Make sure the columns are not the source of the high pressure.

4. Set the high pressure limit to 3.4MPa (500psi) above the normal system operating pressure.

5. Restart the pump.

- **SRS ALARM** [illegible] SRS

The SRS has exceeded the accepted current/voltage range.

1. The SRS may be dry. Make sure the eluent reservoir is filled and sufficiently pressurized. Check all liquid lines and valves for leaks, crimping, or blockage.

2. Make sure the cable connecting the SRS to the DX-120 electronics card is connected.

3. Refer to the SRS manual for additional troubleshooting information.

Error Codes

When an error occurs, an error code number displays in the middle of the screen. The error code remains for several seconds, and then the display returns to normal.

- **E000**

Cause: No Moduleware is installed. Moduleware is the DX-120 instrument control microprocessor code.

Action: Download a new copy of the Moduleware if the system includes PeakNet software, or contact Dionex for assistance.

- **E001**

Cause: The **Column B** button was pressed, although the system is configured for a single column.

Action: If it is a dual-column system, verify that the Column Configuration DIP switch (SW1-3) is set to dual column.

- **E002**

Cause: Two or more conflicting DIP switch settings are set.

Action: Reset the DIP switches (see Section).

- **E003**

Cause: More than one high-pressure alarm setting is selected. The pump and SRS will turn off when this error occurs.

Action: Check the high-pressure alarm setting (SW1-5, 6, 7). One switch position must be on and the other two off.

- **E004**

Cause: During the rinse portion of the column switching sequence, a command to switch columns was received from the **Column A** or **Column B** button, or from PeakNet.

Action: The DX-120 cannot begin another column switching sequence during the rinse cycle. Wait until the column switching sequence is completed.

- **E005**

Cause: A command to switch columns was received from either the **Column A** or **Column B** buttons or from PeakNet and the pump is off or the flow rate is very low. The rinse cycle step of the column switching sequence requires an adequate flow rate to flush the previous eluent from the system before switching.

Action: Turn on the pump or increase the flow rate.

- **E006**

Cause: The SRS will not turn on. Either the pump is not on or the suppressor is disconnected.

Action: Turn on the pump; make sure the cable connecting the SRS to the DX-120 electronics card is connected.

Liquid Leaks

- **Leaking fitting**

Locate the source of the leak. Tighten or, if necessary, replace the liquid line connection. Refer to Installation of Dionex Ferrule Fittings for tightening requirements.

- **Broken liquid line**

Replace the line and fittings.

- **Blocked or improperly installed waste line**

Make sure the waste lines are not crimped or otherwise blocked. Also make sure waste lines are not elevated at any point after they exit the DX-120.

- **Loose pump check valve housing**

Make sure the check valves are firmly seated in the pump head. If they are not, tighten them carefully with an open end wrench just until the leak stops (25 in-lb torque maximum).

- **Damaged pump piston seal**
  1. Replace the piston seal (see Section).
  2. If the problem persists, replace the pistons (see Section).

- **Pump head not tight against casting**

  Carefully tighten the pump head mounting screws just until the leak stops (12 intorque). **DO NOT OVERTIGHTEN!**

- **Leaking pressure transducer**

  Make sure the liquid line connections into the check valves are tight. Refer to *Installation of Dionex Ferrule Fittings* for tightening requirements. Replace any damaged fittings.

  Make sure the waste valve is closed. To close the valve, turn the knob clockwise, just until tight. DO NOT OVERTIGHTEN! Overtightening may damage the valve and the pressure transducer housing.

  Inspect the pressure transducer. If the waste valve is the source of the leak, replace the waste valve O-ring (see Section). If the leak is from the rear of the transducer, replace the pressure pad and O-ring.

- **Leaking SRS**

  See the SRS manual for troubleshooting procedures.

- **Leaking DS4.**

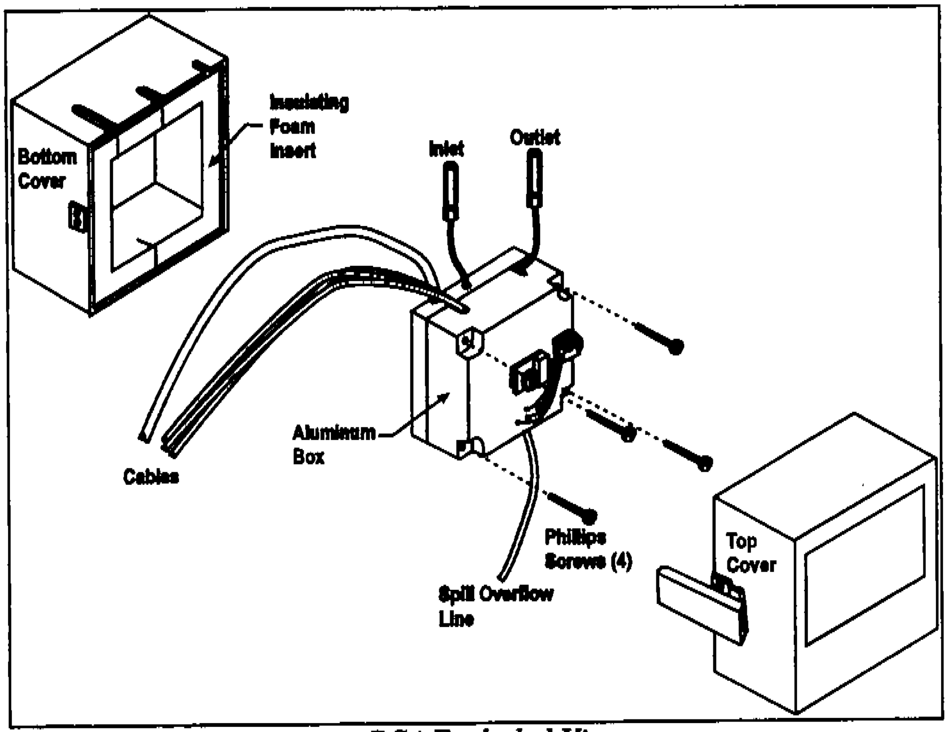
  Check the waste lines for blockage; trapped particles can plug the lines, causing a restriction and/or leak. If necessary, clear the waste lines by reversing the direction of flow.

  Make sure the plumbing downstream from the DS4 is clear; a blockage may overpressurize the DS4, causing it to leak.

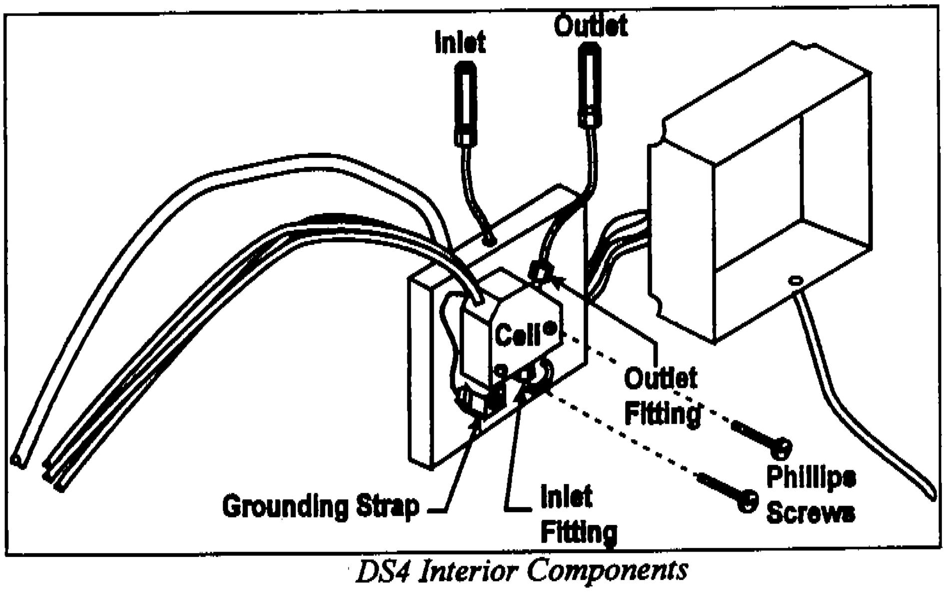
  Make sure the downstream backpressure coils are appropriate for the operating flow rate.

Follow the steps below to disassemble the DS4 and inspect it for the source of the leak.

1. Turn off the DX-120 power.
2. Disconnect the DS4 cables.
3. Disconnect the two 10-32 fitting bolts. Do not misplace the ferrule fittings at the end of the tubing.
4. Remove the DS4 by lifting it upward and then pulling it away from its mounting location. Place the DS4 on the workbench.
5. Open both latches on the DS4 and carefully lift off the top half of the cover, along with its insulating foam insert, exposing an aluminum box .
6. Remove the box from the bottom half of the cover.
7. Remove the four Phillips screws securing the top of the box to its bottom plate. Carefully separate the two parts, exposing the cell. Lay the top aside, being careful not to pull or stress the group of wires that connect the two parts.
8. Check the inlet and outlet cell fittings for leaks. Tighten or replace if necessary.
9. Dry the DS4, test for leaks, and reassemble. Make sure the foam insert is adjusted evenly around the various DS4 components, with no pinching or folding. Check that the top and bottom inserts meet on all sides before latching the cover.



DS4 Exploded View



DS4 Interior Components

- **Liquid seeping from around cell cables**
  The cell has an internal leak and is inoperable; return it to Dionex for repair or exchange.

Pump Difficult to Prime

- **Empty reservoir and/or no eluent connected**
  Connect the pump inlet line to the eluent reservoir. Fill the reservoir.

- **Eluent reservoir not pressurized**
  Connect the air line to the reservoir and press Eluent Pressure to turn on the pressure to the reservoir.

- **Partially blocked end-line filter**
  If the end-line filter (P/N 045987) is no longer pure white, replace it.

- **Liquid leaks at junction between pump head and pump casting**

  1. Replace the piston seals (see Section).

  2. Clean the inlet and outlet check valves.

Pump Loses Prime

- **Eluent reservoir empty**
  Refill the reservoir.

- **Liquid leaks at junction between pump heads and pump casting**
  1. Replace the piston seals.
  2. Clean the inlet and outlet check valves.

Pump Does Not Start

Power switch on pump or Pump button on front panel is off
Turn on both switches.

No power (LED indicators on the control panel are not lighted)
Check that the power cord is plugged in.
Check the main power fuses and replace if needed.

No Flow

Pump not primed

Prime the pump .

Flow rate set to zero

Reset the flow rate.

Broken pump piston

Replace the piston (P/N 014203).

Excessive System Backpressure

- **Restriction in the hydraulic system**

  Check all liquid lines for crimping or blockage. Make sure the ferrule fittings are not overtightened onto tubing. Refer to Installation of Dionex Ferrule Fittings for details.

- **Plugged or damaged fitting**

  Isolate the faulty fitting by loosening fittings one-by-one until the pressure returns to normal. Repair or replace the fitting.

- **Flow rate through the columns too high**
    1. Check the column flow rate, and verify that it matches the flow rate set for the pump.
    2. Measure the pump flow rate using a 10 mL graduated cylinder and stopwatch. Calibrate the flow rate if needed.

- **Clogged column bed supports**
  Replace the bed supports as instructed in the column manual.

- **Contaminated columns**
  Clean the columns (see Column Rejuvenation Procedures, Technical Note 2R, Document No. 032036, or refer to the column manual).

- **Plugged Rheodyne valve passages**
  Refer to the Rheodyne Valve Operator's Manual for troubleshooting information.

Peak "Ghosting"

Ghosting is the appearance of extraneous peaks in a chromatogram. These may be late-eluting peaks from a previous injection or they may result from a contaminated, malfunctioning, or incorrectly installed injection valve. These peaks may co-elute with peaks of interest, resulting in non-reproducible peak heights/areas.

- **Insufficient time between sample injections**
  Wait until the previous sample has been completely eluted before making another injection.

- **Insufficient flush between samples**
  Flush the sample loop with at least 10 loop volumes of deionized water or sample between sample injections.

- **Malfunctioning or incorrectly installed injection valve**
  Refer to the *Rheodyne Valve Operator's Manual* for troubleshooting information.

Nonreproducible Peak Height or Retention Time

- **Column overloading**
  1. Dilute the sample.
  2. Change to a sample loop with a smaller volume.

- **Liquid leaks**
  Locate and eliminate the leaks.

- **Incomplete or imprecise filling of the sample loop**
  1. Fill the sample loop until excess sample exits the waste line.
  2. Inspect the syringe (P/N 016387, 10cc; 016388, 1cc) and replace it if damaged.

Abnormal Retention Time or Selectivity

- **System not equilibrated following an eluent change**
  Allow the system to equilibrate with at least 20 column volumes of eluent (for example, 30 minutes at 2.0 mL/min for 4 mm anion separator columns).

- **Incorrect flow rate through system**
  1. Check that the correct flow rate is selected.
  2. Calibrate the pump flow rate.
  3. Locate and eliminate any liquid leaks.

- **Contaminated or incorrect eluent**
  Remake the eluent using reagent grade chemicals and ASTM filtered, Type I (18-megohm) deionized water.

- **Contaminated or degraded sample**
  Take appropriate precautions when preparing and storing samples to prevent contamination and degradation.

- **Contaminated column**
  1. Clean the column (see Column Rejuvenation Procedures, Technical Note 2R, Document No. 032036, or refer to the column manual).
  2. If cleaning is unsuccessful, replace the column.

DS4 Temperature Inaccurate

- **CELL TEMP HI displays continuously**

  Verify that the set temperature is at least 5 ° C above ambient (SW4-4, 5, 6, 7). Allow 30 to 60 minutes for the initial warm-up period. If you later select a higher set point, allow an additional 3 to 7 minutes for each 5-degree increment in the set point.

The DS4 can take from 1 to 2 hours to completely cool down.

- **LO CELL TEMP displays continuously**
  At high flow rates and temperature settings far above ambient, the DS4 requires more time to heat. In extreme cases, such as a very cold room and a high DS4 set point, the DS4 may not be able to reach the set point temperature.

- **DS4 does not heat**
  Make sure the Cell Type DIP switches (SW4-1, 2) are set to the DS4 position (off).

Make sure that one of the DS4 temperature switch positions (SW4-4, 5, 6, 7) is on. If they are all off, the DS4 will not heat.

Remove the cover and inspect the DS4 for broken or shorted wires or for moisture bridging the control thermistor. If a wire is broken or shorted, replace the wire or call Dionex for assistance

If a leak has caused a short, fix the leak and dry the control sensor.

No Detector Response

- **Cell not connected**

  Check the cell cable connection.

- **Analog output range too high; although the display indicates a response, no recorder response observed**

  Select the 100mS range setting (SW4-3, off).

- **Full-scale output too low**

  Select the 10.0 V full-scale setting (SW4-4, off).

- **No flow from pump**

  Check that the LED on the Pump button is lighted. Check the power switch on the front of the pump.

  Make sure the flow rate is not set to zero.

- **Detector offset out of range**

  Press **Auto Offset** on the front control panel.

Cell electronics malfunctioning

Perform the following test procedure to check the electronics:

1. Disconnect the cell cable from the electronics card at the left side of the pump compartment.
2. Set SW4-1, 2 to the off positions.
3. Set SW5-8 to the on position.
4. The conductivity reading on the display should be 25.0 mS. If this is not the case, there may be a problem with the cell electronics. Contact Dionex for assistance.

Low Detector Output

- Analog output range set too high; although the display indicates a response, no recorder response observed

  Select the 100mS range setting (SW4-3, off).

- **Insufficient sample injected**
  Increase the injection size or concentration.

- **Cell out of calibration**
  Recalibrate the cell.

High Detector Output

- **Auto offset not activated recently**
  Press **Auto Offset** on the front panel before making an injection.

- **Background not suppressed by SRS**
  Check that the SRS is on (the LED on the SRS button should be illuminated).

Check the SRS regenerant out line for bubbles; if there are no bubbles, the suppressor may be contaminated. Refer to the SRS manual for troubleshooting guidance.

- **Sample concentration too high**
  Dilute the sample or install a smaller sample loop.

- **Wrong eluent**
  Make sure you are using the correct eluent.

- **Cell out of calibration**
  Recalibrate the cell.

Noisy or Drifting Baseline

- **Flow system leak ahead of cell; erratic baseline**

  Check all fittings and liquid lines for leaks. Tighten or, if necessary, replace all liquid line connections. Refer to *Installation of Dionex Ferrule Fittings* for tightening requirements.

- **Pump not properly primed**
  Prime the pump.

- **Rapid changes in ambient temperature**
  Redirect heating and air conditioning vents away from the DX-120.

  Replace the CDM-3 cell with a DS4 Detection Stabilizer (P/N050218).

- **Insufficient system equilibration following any changes to operating parameters; especially apparent when operating at high sensitivities**

  Allow longer system equilibration time before starting operation.

- **Air trapped in cell; excessive regular pulses in baseline**
  Check that the correct backpressure coils are installed after the cell and before the SRS.

- **Incorrect SRS operating conditions**
  Refer to the SRS manual for troubleshooting information.

- **Temperature compensation setting not optimized**
  Optimize the setting.

- **DS4 above or below set point**
  Wait for the DS4 to reach the selected temperature before beginning operation. The display will indicate **CELL TEMP** and the selected temperature. If the temperature is above or below the set point, **CELL TEMP HI** or **LO CELL TEMP** is displayed.

MAINTENANCE

This chapter describes routine service procedures for the DX-120. Before replacing any parts, refer to the troubleshooting information in to isolate the cause of the problem. When ordering replacement parts, please include the model and serial numbers of the DX-120.

Substituting non-Dionex parts may impair DX-120 performance, thereby voiding the product warranty. Refer to the warranty statement in the Dionex Terms and Conditions for more information.

NOTE

The DX-120 electronic components are not customer-serviceable. Any repairs involving the electronics must be performed by Dionex.

Replacing Tubing and Fittings

Tubing Size and Type	Used For
0.125-mm (0.005-in) ID (P/N 044221)	Connection from the pump pulse damper to the injection valve
0.25-mm (0.010-in) ID (P/N 042690)	Connections between other system components
0.5-mm (0.020-in) ID (P/N 042855)	Detector cell waste line
Waste Separator Tubing Assembly (P/N 045460)	SRS **REGEN OUT** connections
4-mm Backpressure Tubing Assembly (P/N 045877)	SRS **REGEN IN** connections

- 10-32 fittings (P/N 043275) and ferrules (P/N 043276) are used for most tubing connections. For tightening requirements, refer to *Installation of Dionex Ferrule Fittings*

- 1/8-in flangeless fittings (P/N 048951) and ferrules (P/N 048949) are used for the SRS **REGEN OUT** port connections.

- 1/16 flangeless fittings (P/N 048952) and ferrules (P/N 048950) are used for the SRS **REGEN IN** port.

Changing the Sample Loop

Peak response is directly related to the sample concentration and the injection volume. You can change the sample volume by changing the volume of the injection valve sample loop. With most samples, use of a sample loop larger than 100 mL results in column overloading and nonlinear response. This overloading volume can be even smaller with some sample types.

1. Turn off the pump.

2. Open the DX-120 door.

3. Disconnect the sample loop from ports 1 and 4 on the injection valve.

4. Sample loops of different sizes are available from Dionex. You can also prepare a new sample loop from 0.3 mm (0.012 in) ID tubing (P/N 035548).

   Allow 1.42 cm (0.54 in) of tubing, including the fittings, for each microliter of loop volume.

   When making sample loops of 100 mL or more, use 0.5-mm (0.020-in) ID tubing (P/N 035864). Allow 51 cm of tubing, including fittings, for each 100 mL.

5. Install the new sample loop between ports 1 and 4 on the injection valve.

Isolating a Restriction in the Liquid Plumbing

A restriction in the liquid plumbing will cause excessive system backpressure.

1. Begin pumping eluent through the system (including the columns) at the flow rate normally used.

2. Follow the appropriate hydraulic schematic and work backward through the system, beginning at the cell exit. One at a time, loosen each fitting and observe the pressure. The connection at which the pressure drops abnormally indicates the point of restriction.

If the restriction has caused such high pressure that the system cannot be operated, you must work forward through the flow schematic, adding parts one at a time until an abnormal pressure increase (and hence, the restriction) is found.

3. If the restriction is in the tubing or fitting, remove the restriction either by back flushing or by replacing the tubing or fitting.

Replacing the DS4 Cell

Follow the steps below to disassemble the DS4 and replace the cell. After replacing the cell you must recalibrate it

1. Turn off the DX-120 power.

2. Disconnect the DS4 cables.

3. Disconnect the two 10-32 fitting bolts. Do not misplace the ferrule fittings at the end of the tubing.

4. Remove the DS4 by lifting it upward and then pulling it away from its mounting location. Place the DS4 on the workbench.

5. Open both latches on the DS4 and carefully lift off the top half of the cover, along with its insulating foam insert, exposing an aluminum box.

6. Remove the box from the bottom half of the cover.

7. Remove the four Phillips screws securing the top of the box to its bottom plate. Carefully separate the two parts, exposing the cell. Lay the top aside, being careful not to pull or stress the group of wires that connect the two parts.

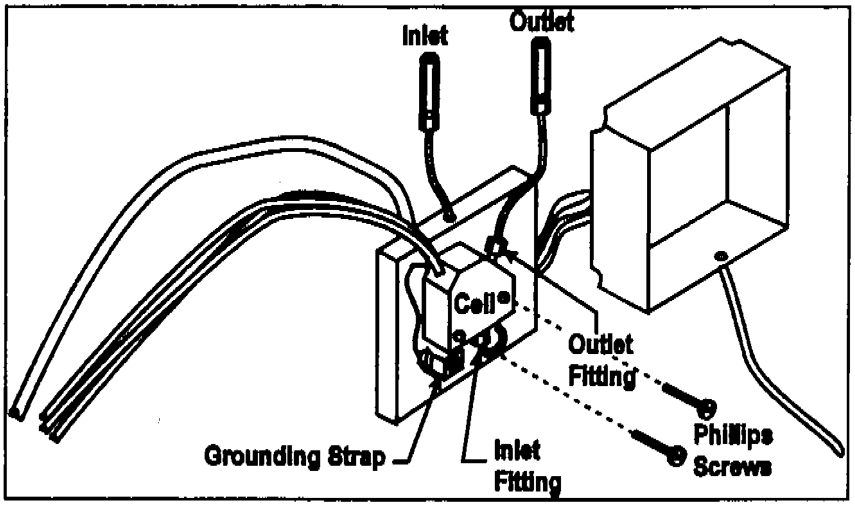
8. Disconnect the grounding strap. Remove the two cell mounting screws.

9. Rotate the cell inlet tube fitting counterclockwise. Let the cell body back away from the fitting until the fitting threads are fully disengaged.
Do not lose the ferrule.

10. Check that the end of the inner Tefzel tubing is flush with the end of the plastic sleeve. If necessary, trim the sleeve slightly to prevent dispersion. Push the tubing into the new cell until it bottoms out in the hole; then, hold the tubing in place while tightening the nut.

11. After testing for liquid leaks, dry the DS4 and reassemble. Make sure the foam insert is adjusted evenly around the various components, with no pinching or folding. Check that the top and bottom inserts meet on all sides before latching the cover.

12. Reconnect the DS4 cables and turn on the DX-120 power.
Calibrate the cell constant.



Interior Components

Cleaning Cell Electrodes

If fouling is suspected, the cell must be cleaned and recalibrated.

1. Prepare the solutions listed below, using filtered ASTM Type I (or better) deionized water. Pour the prepared solutions into labeled containers.

CAUTION

HNO3 is corrosive and a strong irritant. Avoid breathing the vapors. Always prepare the cleaning solution in a fume hood. Wear gloves and goggles.

* a. 3 M HNO3 cleaning solution: dilute 200 mL concentrated HNO3 (s.g. 1.42) to one liter with deionized water.

* b. 0.01 M KCl stock solution: dissolve 0.7456 g of reagent grade KCl in one liter of deionized water.

* c. 0.001 M KCl calibration solution: dilute 100 mL of the stock solution to 1 liter with deionized water.

2. Connect the container of 0.001 M KCl to the pump.

3. Disconnect the line between the suppressor outlet and the cell (or DS4 inlet) at the suppressor. In a dual-column system, disconnect the cell inlet line from port 8 on the column switching valve.

4. Connect a female luer adapter (P/N 024305) to the tubing, using a union (P/N 024447).

5. Fill a 10mL syringe (P/N016387) with the 3 M HNO3 solution. Screw the syringe into the luer adapter.

6. Turn off the DX-120 main power switch.

7. Inject 5 mL HNO3 through the cell.

8. After two minutes, push the remaining 5 mL of solution through the cell. Wait two minutes.

9. Fill the syringe with 10 mL of deionized water. Inject the water through the cell.

10. Turn on the DX-120 main power switch, calibrate the cell constant.

Calibrating the Cell Constant

Calibrate the cell after installing a new cell or after cleaning the cell electrodes. The cell does not require routine calibration.

1. Disconnect the pump eluent line from port 2 of the injection valve.

2. Disconnect the line between the suppressor outlet and the cell or DS4 inlet at the suppressor. In a dual-column system, disconnect the cell inlet line from port 8 on the column switching valve.

3. Connect the eluent line from the pump directly to the inlet of the cell or DS4, using a union (P/N 024447).

4. Pump 0.001 M KCl calibration solution through the cell at 2.0mL/min. After 5 minutes, reduce the flow rate to the value typically used during analysis and pump for an additional minute. Conductivity is slightly flow-rate sensitive, so select the flow rate used in the majority of your applications.

5. Set the temperature compensation to 1.7% (SW5-5, on; SW5-4 and 6, off). This temperature compensation value can be used for most applications. When measuring absolute conductivity, determine the optimal value for each batch of calibration solution.

6. Disconnect the cell cable from the cell connector on the edge of the electronics card.

7. Press the **Auto Offset** button. With the cell disconnected, this is the zero conductivity point. Press **Offset Cond** to display the offset conductivity.

8. Reconnect the cell cable.

9. Locate the Cell Calibration potentiometer, above the cell cable connector on the edge of the electronics card. Continue pumping calibration solution through the cell. Use the tuning screwdriver (P/N 035617) from the Ship Kit to adjust the potentiometer until the display value equals 147 ± 2 mS.

10. Replace the container of 0.001 M KCl calibration solution with a container of deionized water. Pump deionized water through the liquid lines at 2.0 mL/min for at least 5 minutes to flush the calibration solution from the system.

11. Reconnect the pump to the injection valve.

12. In a single-column system, reconnect the liquid line from the cell or DS4 inlet to the suppressor outlet. In a dual-column system, reconnect the line from the cell inlet to the column switching valve.

13. Reset the temperature compensation to the optimal value for the eluent in use (SW5-4, 5, 6). The cell is now calibrated and ready for operation.

Calibrating the Pump Flow Rate

1. Connect an 11 MPa (1600 psi) backpressure device to the pump.

2. Turn on the pump and set the flow rate to 1.2 mL/min.

3. Pump deionized water through the system for at least 15 minutes to allow the system to equilibrate.

4. Collect the water into a tared beaker for 5 minutes. Weigh the collected water. It should be 6.00 ±0.05 g.

5. If the collected water is not within the above range, locate the Pump Flow Setting Adjust potentiometer on the electronics card . Continue pumping deionized water through the cell. Use the tuning screwdriver (P/N 035617) from the Ship Kit to adjust the potentiometer until the flow rate display corresponds to the weight of the collected water. For example, if you collected 5.5 g, adjust the flow rate to 1.1 mL/min.

Cleaning a Pump Check Valves

If bubbles or particles become trapped in the check valves, backward flow may occur. If this happens, pressure may vary.

If these symptoms occur, re-prime the pump, since the problem may be a bubble trapped in the pump head or check valve. If the symptoms persist, clean the check valve using the following procedure.

1. Remove the pump head assembly.
2. Squirt cleaning solution (such as isopropyl alcohol, dilute acid, or dilute base) into the check valve
3. Lay the entire assembly flat at the bottom of as beaker.
4. Cover the assembly with cleaning solution.
5. Sonicate for 10-15 minutes.
6. Rinse the assembly thoroughly with deionized water.

Because of high operating pressures, the check valve components are packed very tightly. Therefore, the components cannot be dissembled, cleaned, and reassembled. If the cleaning procedure given fails to solve the problem, replace the check valves.

Inlet Check Valve (yellow cap)	**P/N 038273**
Outlet Check Valve (green cap)	**P/N 038272 (1/4-28)**
Outlet Check Valve (green cap)	**P/N 042761 (10-32)**

Replacing a Pump Piston Seal

A damaged piston seal allows leakage around the piston at the head mounting plate or around the base of the pump head. Flow rates will be low and erratic.

1. Turn off the main power switch on the DX-120 and disconnect the main power cord.

2. Disconnect the tube fittings from the inlet and outlet check valves.

3. Hold the head firmly against the pump housing to compensate for the spring loading, and remove the two nuts.

CAUTION

Lateral motion when disengaging the head from the piston can break the piston.

4. Slowly release the head, allowing it to separate from the housing. **CAREFULLY** disengage the head from the sapphire piston by pulling the head straight off and away from the mounting guides. Be especially careful not to snap the piston if the internal spring is stuck to the piston guide.

5. Place the head, front end down, on a clean work area. Lift the piston guide and back-up washer off, exposing the seal.

6. Remove the piston seal from the head. Discard the seal.

7. Remove the O-ring and back-up seal from the back-up washer.

8. Carefully push the new piston seal (P/N 035686) into the head. When properly installed, the piston seal is almost flush with the indented surface of the head.

9. Press a new O-ring (P/N 035776) into the back-up seal (P/N 036901). If necessary, also replace the O-ring. Then press the O-ring and back-up seal into the back-up washer.

10. Press the back-up washer into the head, followed by the piston guide.

11. Remove the spring, spring guide, spring retainer, and piston from the pump housing. Inspect the interior of the housing for liquid and corrosion. Clean up any spills and carefully clean any signs of corrosion from the piston and the interior of the housing. Reinstall the piston.

12. Carefully slide the pump head straight onto the mounting guides. Apply gentle pressure to push the piston through the seal. Guide the spring onto the piston guide.

CAUTION

Avoid all lateral motion when sliding the head onto the piston. Failure to slide the head straight on will damage the piston seal and back-up seal, as well as break the piston.

13. Hold the head firmly against the pump housing and replace the two nuts. Use a wrench to tighten them evenly (12 in-lb torque).

14. Reconnect the liquid line to the inlet check valve.

15. Reconnect the main power cord and turn on the main power switch.

16. Prime the pump

Replacing a Pump Piston

Continued leaking from around the pump head after replacing the piston seal indicates a scratched or broken piston.

1. Turn off the main power switch on the DX-120 and disconnect the main power cord.

2. Disconnect the tube fittings from the inlet and outlet check valves.

3. Hold the head firmly against the pump housing to compensate for the spring loading and remove the two nuts.

CAUTION

Lateral motion when disengaging the head from the piston will break the piston.

4. Slowly release the head and allow it to separate from the housing. **CAREFULLY** disengage the head from the sapphire piston by pulling the head straight off and away from the mounting guides. Be especially careful not to snap the piston if the spring is stuck to the piston guide.

5. Remove the piston guide, spring, spring guide, spring retainer, and piston by pulling them straight out, away from the pump housing.

6. If the piston is broken, replace the piston seal and the back-up seal. This will prevent pieces of broken piston from scratching the new piston assembly.

7. Remove the retainer ring (P/N 035010) from the old piston assembly and install it on the new piston assembly (P/N 036904).

8. Carefully slide the spring retainer onto the piston assembly.

9. Slide the assembled piston back into the piston housing.

10. Slide the spring over the piston, positioning it flush against the spring retainer.

11. Carefully slide the pump head straight onto the alignment rods. Gently push the piston through the seal. Guide the spring over the piston guide.

CAUTION

Avoid all lateral motion when sliding the head onto the piston. Failure to slide the head straight over the piston will damage the piston seal and back-up seal, as well as break the piston.

12. Hold the head firmly against the pump housing and replace the two nuts. Use a wrench to tighten them evenly (12 in-lb torque).

13. Reconnect the liquid lines to the check valve housing.

14. Reconnect the main power cord and turn on the main power switch.

15. Prime the pump

Replacing the Pressure Transducer Gasket and O-Ring

A damaged gasket or O-ring causes leakage around the base of the pressure transducer housing at the mounting ring. Flow rates will be low, resulting in chromatograms with excessively long retention times.

1. Press the Pump button to turn off the pump.

2. Disconnect the liquid lines between the pressure transducer housing and the check valve housings.

3. Disconnect the waste line from the pressure transducer housing.

4. Remove the output line from the pressure transducer housing.

5. While holding the housing firmly against the mounting ring, remove the four screws securing the housing to the mounting ring .

6. Gently pull the housing away from the ring, exposing the pressure transducer face.

7. Remove the temperature isolation disk, the gasket, and the O-ring from the housing.

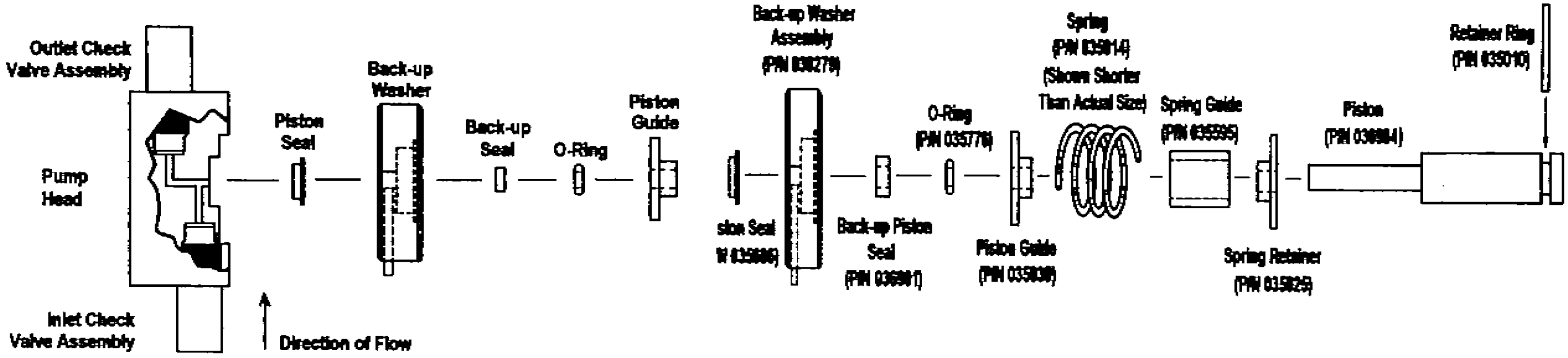
8. Install a new O-ring (P/N 041262) and gasket (P/N 035113) in the housing. Place the temperature isolation disk on top of the gasket.

9. Reposition the housing on the ring and replace the screws. Carefully tighten the screws until evenly tight (20 in-lb).

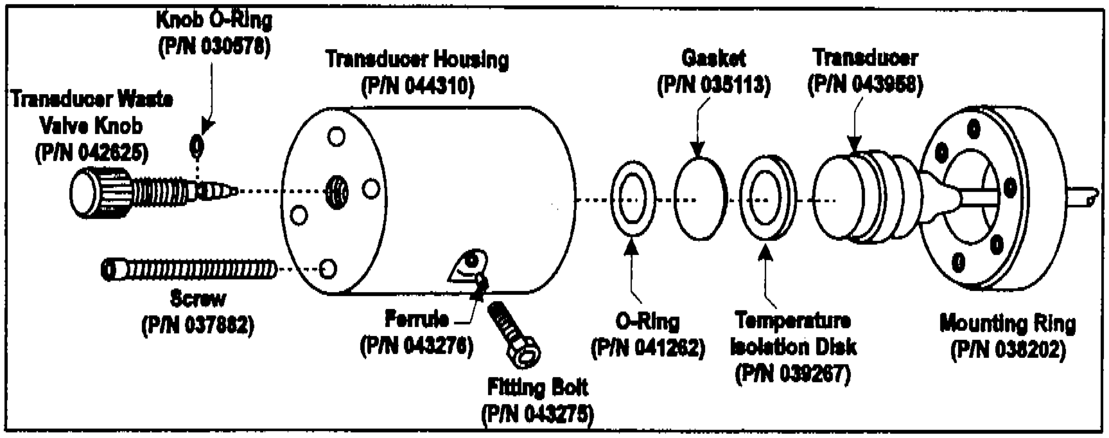
CAUTION

Overtightening or uneven tightening may damage the pressure transducer housing.

10. Reconnect the liquid lines from the check valve housings. Reconnect the outlet line and the waste line.



Pump Head and Piston Components



Pressure Transducer Assembly

Replacing the Pressure Transducer Waste Valve O-Ring

A damaged O-ring causes leakage around the base of the pressure transducer waste valve knob. Flow rates will be low, resulting in chromatograms with unusually long retention times.

1. Press the **Pump** button to turn off the pump.
2. Remove the valve from the pressure transducer housing by turning the knob counterclockwise until it comes loose from the housing .
3. Cut through the O-ring with a razor blade, being careful not to scratch the valve body. Remove the O-ring.
4. Carefully slide a new O-ring (P/N 035776) over the end of the valve and push it into the groove.
5. Wipe a very thin film of silicone grease on the O-ring.

CAUTION

Excessive grease may contaminate the eluent as it flows through the pressure transducer housing.

6. Reinstall the valve in the housing, turning the knob clockwise until the valve is seated.

CAUTION

Do not overtighten the valve knob. Overtightening the valve may damage the seat and pressure transducer housing.

SYSTEM TROUBLESHOOTING

Objectives

By the end of this section, each participant should be able to:

- Develop a daily system log
- Develop a maintenance schedule
- Develop a troubleshooting procedure
- Identify troubleshooting resources

Daily System Log

A daily system log can serve several functions including:

- establish normal operating parameters for the system
- record deviations from normal operating parameters
- provide a record of maintenance procedures

All of these functions are essential for system troubleshooting if a problem occurs.

A system log can be a dated, initialed record of:

- Chromatogram parameters
- Maintenance procedures
- Preparation of eluents /reagents
- Component operating conditions

A sample daily system log incorporating these parameters follows.

DAILY LOG

Application:

Date:	**S**	**M**	**Tu**	**W**	**Th**	**F**	**S**
System pressure							
Detector background							
Flow rate							
Post-column or regen flow rate							
Eluent prep							
Post column or regen prep							
Retention time: Analyte: Analyte: Analyte: Analyte: Analyte:							
Maintenance performed							
Comments							

Maintenance Schedule

A maintenance schedule serves two purposes:

1) a reminder of when maintenance procedures are recommended
2) a record of when maintenance procedures were performed

Although this information may appear in a daily log, a maintenance schedule will provide a centralized location for this information.

A sample of a maintenance schedule follows.

MINIMUM MAINTENANCE SCHEDULE

Application:

		Date	Initials
Monthly	Change inlet bed supports if more than a 30% increase in pressure at the pump has occurred Flush check valves with IPA **(Caution: bypass all columns)**		
6 Months	Lubricate pump Change piston seals and back-up seals Inspect port faces Inspect slider		
12 Months	Clean out integrator printer chart recorder of any paper dust Check waste lines, eluent lines, air tubing, regenerent lines, grippers. Replace damaged or worn lines Replace port faces		

Troubleshooting

Troubleshooting a system can be a very complicated task. Problems could be of a chemical, mechanical or data collection origin.

The following can be used to troubleshoot problems:

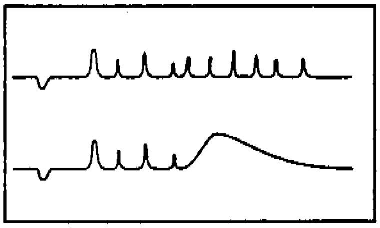
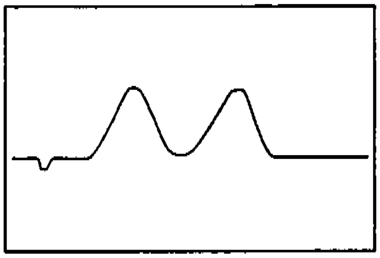
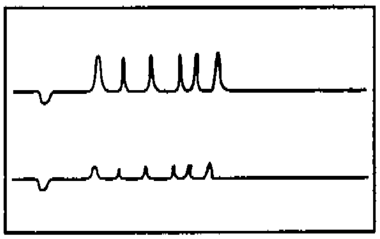
- daily log
- maintenance schedules
- operator's manuals
- troubleshooting procedure

A troubleshooting procedure gives you a systematic approach to finding and eliminating causes of problems. The following is a suggested procedure.

Troubleshooting Procedure

- Make a list of problems
- List possible causes using system records and troubleshooting guides
- Rank causes in order of likelihood
- Create a flow chart
- Check out each possible cause until the right one is isolated and corrected

ELUENT TROUBLESHOOTING CHART

Problem	Cause	Solution
Poor efficiency, strongly retained analytes do not elute 	Eluent too weak	Remake eluent
	Chemicals used in preparation not dried	Dry chemicals (in a dissicator or oven) and remake eluent
	Chemicals used in preparation are in a hydrated form	Make the appropriate adjustment for the difference in molecular weight and remake eluent
Poor efficiency, tailing peaks, longer than usual retention times 	Counter ion in eluent cannot effectively compete with analyte	Check literature for a more suitable counter ion (i.e. *p*-cyanophenate in addition to carbonate) and prepare eluent accordingly
	Column selectivity not optimized for analytes hydrophobic/hydrophilic nature	Review column selectivity parameters and select the appropriate column
Decreased sensitivity or shifting retention times in transition metals analysis	Eluent pH not properly adjusted	Adjust eluent pH
	Eluent filters plugged	Change filters
Pump will not stay primed	Eluent has not been degassed	Degas eluent

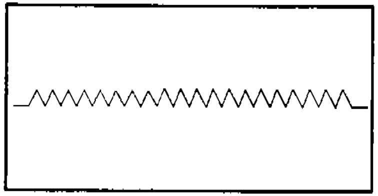
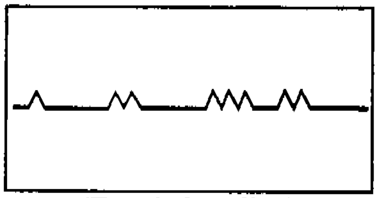
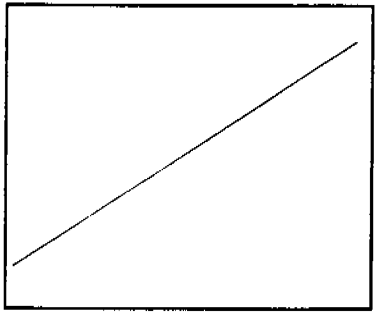
PUMP TROUBLESHOOTING CHART

Problem	**Cause**	**Solution**
Pump is difficult to prime or will not prime	No eluent is connected	Connect the eluent reservoir
	Eluent reservoir is empty	Refill the eluent reservoir
	Low pressure limit is on	Low pressure limit must be off during priming
	Liquid line connections are loose	Locate and tighten any loose connections
	Priming valve on pressure transducer is closed.	Open the valve by turning counterclockwise one full turn
Pump will not start	Power switch on pump or front panel is off	Turn on switch(es)
	High pressure limit is set too low	Set the high pressure limit at least 300-400 psi above the normal system operating pressure
	Low pressure limit is set too high	Set the low pressure limit at least 200-300 psi below the normal system operating pressure
Pressure readout is 0	Flow rate is set to zero	Select a flow rate greater than 0.0 ml
	No backpressure load	Check system for leaks

CONDUCTIVITY DETECTOR TROUBLESHOOTING CHART

Problem	Cause	Solution
No response	Cell drive is off	Turn cell on
	Output range is too high	Select a more sensitive range
	Cell drive or cell return cable is disconnected	Turn DX-120 off. Re-connect both cables. Turn DX-120 on.
Low detector output	Output range is too high	Select a more sensitive range
	Insufficient sample is injected	Increase injection size or concentration
	Detector cell is out of calibration	Clean and calibrate the cell
	Eluent/Regenerant concentration or flow rate is incorrect	Use correct parameters
	Dirty conductivity cell	Clean cell

Problem	**Cause**	**Solution**
High detector output	AUTO OFFSET is not ON	Turn AUTO OFFSET ON before making an injection
	Membrane Suppressor is not installed correctly or has failed	Install as directed in the suppressor manual or clean/replace suppressor
	Eluent/Regenerant concentration or flow rate is incorrect	Use correct parameters

Problem	**Cause**	**Solution**
Noisy baseline  (Regular pulsing)	Air trapped in cell	Remove trapped air
	Backpressure on cell too low	Install approx. 6 ft. length of 0.020 inch tubing to waste container
 (Erratic baseline)	Leak in the system	Locate and repair leak
	Eluent/Regenerant concentration or flow is incorrect	Use correct parameters
	Insufficient system equilibration	Allow to equilibrate longer, especially at high sensitivities
	Baseline has not been reset following a change in the TEMPERATURE compensation setting, causing a step-change in the baseline	Reset baseline by turning AUTO OFFSET OFF and then turning it back ON
Drifting baseline 	Insufficient system equilibration	Allow to equilibrate longer, especially at high sensitivities
	The cell thermistor cable is not attached correctly	Attach correctly and reset the detector microprocessor
	Un-optimized temperature compensation setting	Optimize the selected setting
	Leak in system	Check entire system flow-path for leaks (valve, pump column, suppressor, cell)

TROUBLESHOOTING SCENARIOS

1. After running for several months, a user noticed the fitting of the bottom of the check valve was leaking. After fixing the leak, he noticed the back pressure was very high. Describe how you would track down the high back pressure.

2. Even though a user has degassed his eluents, his pump sounds out of prime. Describe what the causes could be.

3. A user's system had run fine three months ago, and then he left it. Now, when he starts up the system, there is high back pressure. What is the probable cause.?

4. Upon injection, an analyst notices the pump pressure seems to speed up for an instant. What is the cause?

Injector switch isn't
Switching properly

TROUBLESHOOTING SCENARIOS

(con't.)

5. An analyst noticed his peak widths getting wider and less defined on his anion system. He cleaned the column with HCl and though his resolution was better, the peaks were still wider than before. Speculate to the cause.

voids in column

6. The water dip is a measure of the void volume of the system. What does this mean, and why is there an evident water dip on the anion channel, but the water dip is negligible to non-existent on the cation channel?

- what's not held on

↳ turns acid into H_2O

7. You reviewed the anion printouts since the last Autocal runs, and you noticed a sudden drop in the concentration of Cl^- and SO_4^{2-}, but the peak heights look normal. This is an indication of what?

8. You reviewed the printouts and noticed that the peak heights had decreased, and the peak shapes had broadened on all runs. What could be the cause?

Suppressor

detector ~

column ~

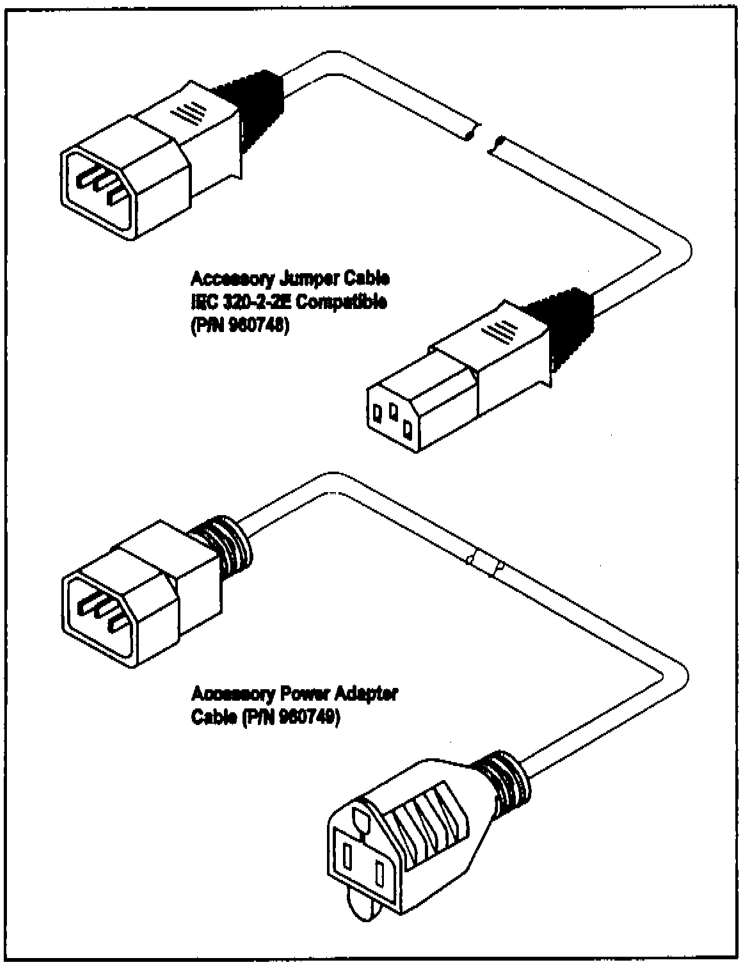
~~[illegible]~~

Switched AC Outlet Connection (Optional)

The AC power outlet provides on off control of an external accessory, such as a pre-concentration pump.
PeakNet software controls the outlet; there is no control from the DX-120.

The Ship Kit includes two accessory power cords. Choose the cord appropriate for the module under control.

- The accessory jumper cable (P/N 960748) is for modules with IEC 320 connectors.
- The accessory power adapter cable (P/N 096749) is for modules with NEMA 5-15 connectors (the 3-prong American line cord plug).



Accessory Cables

DX LAN Card Installation (Optional)

For the DX-120 to communicate with PeakNet software, a DX LAN interface card (P/N 050225) must be installed in the DX-120. If you order a DX-120 with the DX LAN option, the card was installed at the factory. Follow the installation procedure below if you order a DX LAN at a later time.

IMPORTANT

STATIC- The DX-120 electronics are not user-serviceable and the DX LAN interface card should be installed by qualified personnel only. Observe standard anti-static procedures when installing the interface card or handling the electronics card.

CAUTION

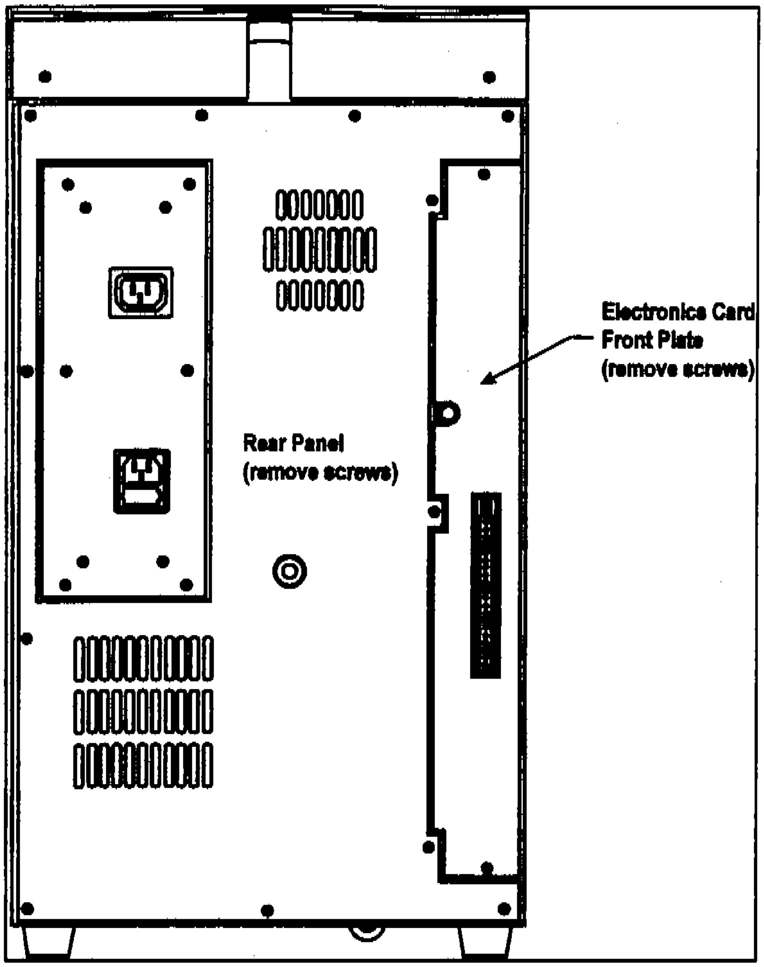
To prevent damage to the DX-120, turn off the main power and unplug the power cord before installing the interface card. Turning off only the front panel power switch is not sufficient.

1. Disconnect all cables from the electronics card at the front left of the main compartment.

2. Disconnect the gas supply line from the rear panel and unplug any TTL connectors.

3. Remove the screws from the rear panel and remove the rear panel from the DX-120. Remove the screws from the electronics card front plate. Inside the DX-120 rear compartment, disconnect all cables from the electronics card. Note the connector location for each cable.

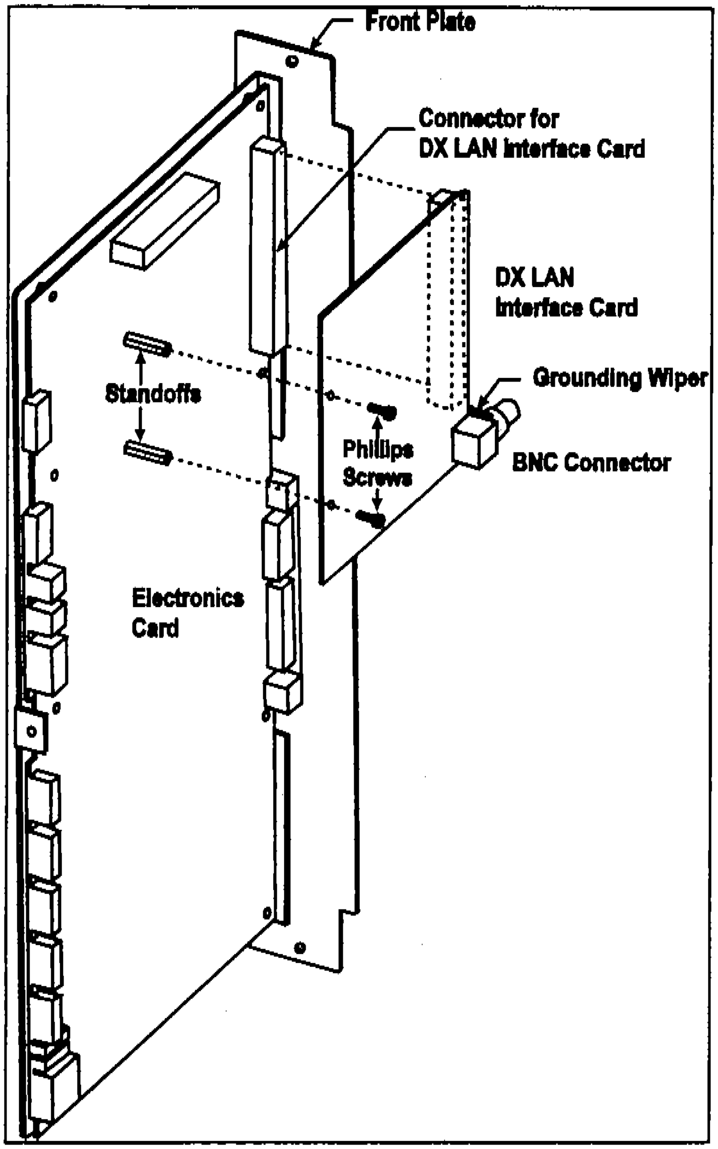
4. Pull out the electronics card far enough to allow access to the DX LAN connector on the top edge of the card.



Removing the Rear Panel

6. Align the BNC connector on the interface card with the opening on the electronics card front plate and insert the interface card connector into the connector on the electronics card. The small stainless steel grounding wipers on the BNC connector slide behind the front plate. Press firmly on the card until it mates fully with the electronics card connector.

7. Fasten the interface card to the standoffs on the electronics card with the two Phillips screws (P/N045791) supplied in the Ship Kit.



Installing the DX LAN Interface Card

8. Push the electronics card back in and replace the mounting screws. Reconnect the cables that were removed in Step 4.
9. Replace the rear panel and mounting screws.
10. Replace the gas supply line and the TTL connectors.
11. Reconnect the cables that were removed in Step1.
12. Connect the DX LAN cable.

DX LAN Cable Connection (Optional)

NOTE

For the DX-120 to communicate with PeakNet software, a DX LAN interface card (P/N 050225) must be installed in the DX-120. If you order a DX-120 with the DX LAN option, the card is installed at the factory. If you order the DX LAN at a later time, see Section for installation instructions.

Figure (Installing the DX LAN Interface Card)- illustrates the cable connections for a single DX-120 connected to a PeakNet workstation. Refer to Installing the PeakNet System for information about installing more than one DX-120 (or other DX LAN equipped instrument) on the network.

1. Connect a BNC tee connector (P/N 921914) to the DX LAN connector on the DX-120 rear panel.

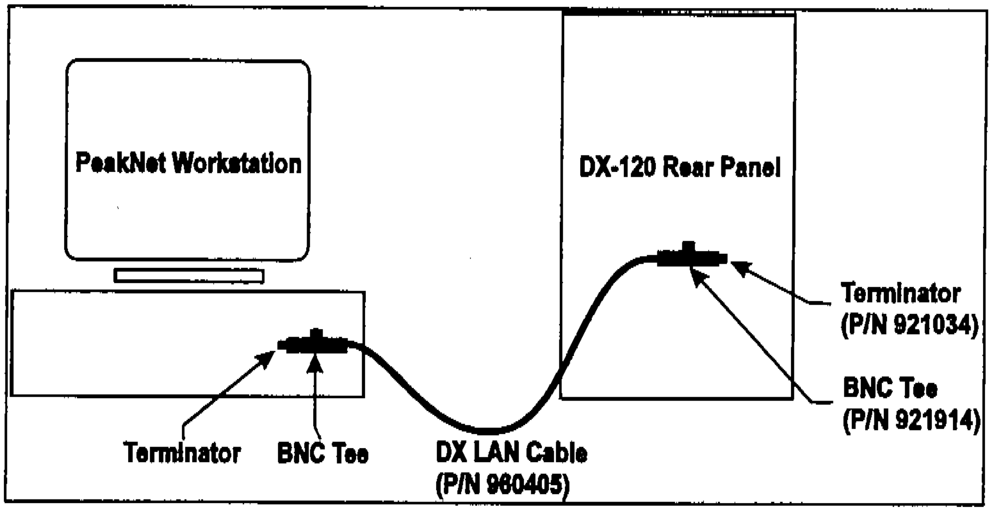
2. Plug the DX LAN cable (P/N 960405) into the tee.

3. If the DX-120 is the last (or only) instrument on the network, cap the unconnected end of the tee with a terminator plug (P/N 921034).

4. Connect the other end of the cable to the BNC tee connector on the PeakNet workstation. If no other device is to be connected, cap the unconnected end of the tee with a terminator plug.

IMPORTANT

Do not connect the DX LAN cable directly to the BNC connectors on the PeakNet workstation or DX-120. BNC tee connectors and terminator plugs are required for correct data link/transfer



DX LAN Cable Connection

Autosampler Connections (Optional)

1. Disconnect the sample inject line frofin port 5 of the DX-120 injection valve

2. Route the outlet line from the autosampler through the opening below the injection port on the DX-120 front door. Connect the line to port 5.

3. Locate the Relay Control cable (P/N 047946) supplied with the AS40 Automated Sampler. Connect this cable's 10-pin connector to the RELAY CONTROL connector on the AS40 rear panel.

4. Remove the 2-pin connector attached to the pair of wires labeled **READY**.

5. Locate the green 5-pin connector (P/N 921273) in the DX-120 Ship Kit. Insert the violet wire from the READY pair into the last position on the connector. Use a screwdriver to tighten the locking screw. Insert the ground wire into the first position on the connector and tighten.

CAUTION

When connecting wires to the green connectors, be careful not to allow stray strands of wire to short to the adjoining terminal on the connector.

6. Plug this connector into the TTL INPUTS connector on the DX-120 rear panel. Make sure the violet wire connects to the **Inject** position and the ground wire connects to the **TTL Ground**.

7. Remove the 2-pin connector from the pair of wires labeled LOAD on the Relay Control cable.

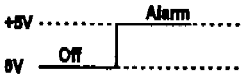
8. Locate a green 3-pin connector (P/N 921186) in the DX-120 Ship Kit. Insert the red wire from the **LOAD** pair into the last position on the connector. Use a screwdriver to tighten the locking screw. Insert the ground wire into the first position on the connector and tighten.

9. Plug this connector into the upper **TTL Outputs** connector on the DX-120 rear panel. Make sure the red wire connects to the **Inject** position and the ground wire connects to the **Ground**.

Outputs

There are two types of TTL outputs:

- Inject and Pressure Alarm outputs signal when an injection or a pressure alarm occurs. When connected to the TTL inputs of another device, such as an autosampler, these signals can trigger a response in the connected device. For example, the Inject output can start the autosampler load cycle.
- TTL Out 1 and TTL Out 2 are controlled by PeakNet software only. When connected to the TTL inputs of other devices, these outputs can control functions in the connected devices.

TTL Output Function	Logic Type	Waveform	Power-up Status	Description
Pressure Alarm	Hold		0V	Normally low. Goes to high when an alarm occurs.
Inject	Pulse Trigger		+5V	Normally pulled high. Pulses down at injection. 500 ms pulse duration.
TTL Out 1		- PeakNet Software Control Only -		
TTL Out 2		- PeakNet Software Control Only -		

TTL Outputs

Electrical

Main Power	Three voltage/frequency configurations(not user-selectable): 100Vac/50 Hz 115 Vac/60 Hz 230 Vac/50 Hz
Fuses	Two fast-blow fuses rated 5.0 A (P/N 214339)
Analog Output	1 V or 10 V, full-scale output

Environmental/Physical

Operating Temperature	10 °C to 40 °C (50 °F to 104 °F)
Operating Humidity	5% to 95% relative humidity, non-condensing
Operating Pressure	28 Mpa (4000 psi)maximum liquid path (tubing, valves, columns, etc.)
Dimensions	60 cm high x 30 cm x 40 cm deep (23 in x 12 in x 16 in)
Weight	30 kg (65 lbs)

CONTROL PANEL

Display	Liquid crystal display
Buttons	16 front panel buttons for controlling the display and operating functions.

PUMP

Type	Single-reciprocating piston with metal-free flow path using PEEK components
OperationMode	Constant volume
Flow Range	0.5 to 4.5 mL/min

PULSE DAMPER

Type	Coilded restricted tubing (PEEK), controlled compliance
Maximum Pressure	28 Mpa (4000 psi)

DETECTOR

Range	1000 μS, full-scale
Temperature Compensation	1.5, 1.7, or 1.9% per °C
Cell Drive	Pulsed, bipolar fixed frequency
Auto Offset	-999 to 999 μS
Local Operation	Front panel controls and display status of all functions
Remote Operation	Control of four functions via TTL inputs
DX Lan	Control of all functions by PeakNet Software via the DX
Operation (Optional)	LAN interface

CONDUCTIVITY CELL

DX-120	Cell body: Chemically inert polymer
Standard Cell	Electrodes 316 stainless steel
(CDM-3)	Active Volume: 1.25 μL Maximum Pressure : 2.0 Mpa (300 psi)
DX-120 High-	Note: This cell is included with the DS4 Detection stabilizer
Performance	Cell body: PEEK
Cell	Electrodes: 316 stainless steel Active Volume: 1.0 μL Maximum Pressure: 2.0 Mpa (300 psi)

DS4 DETECTION STABILIZER (Optional)

Warm-up Time	10 minutes (typical)
Temperature	High or low temperature message displayed when over or
Alarms	under the set temperature

VALVES

Injection	Two-position, six-port, electrically activated Rheodyne valve with PEEK wetted components
Column Switching (Optional)	Two-position, ten-port, electrically activated Rheodyne valve, with PEEK wetted components

DELAY VOLUME

System Total	<5 mL