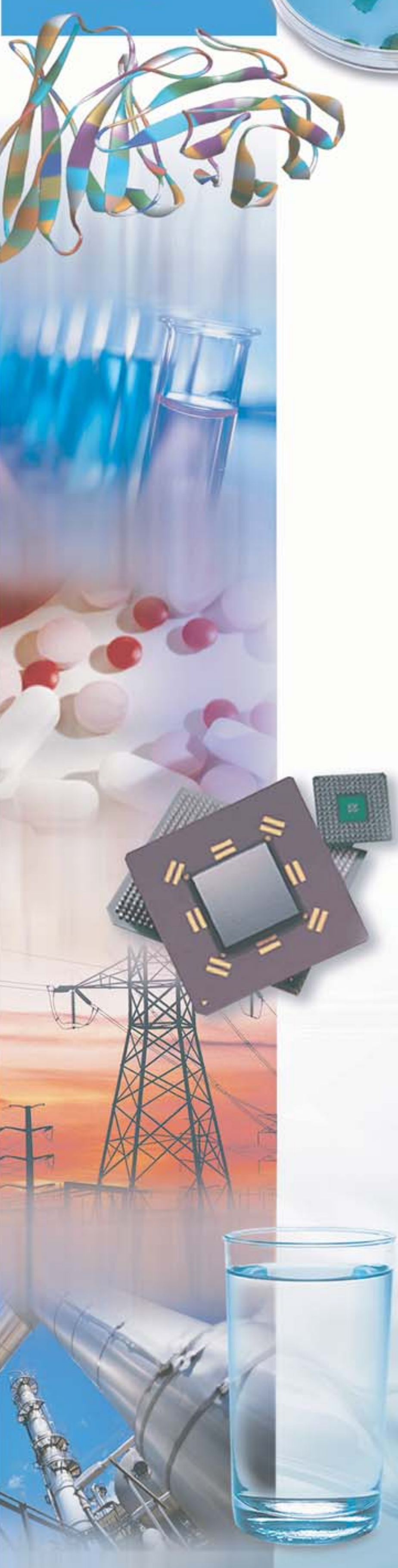

PRODUCT MANUAL

SRN™ 300
ASRN™ 300
CSRN™ 300

Now sold under the Thermo Scientific brand

Thermo SCIENTIFIC

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DIONEX

IC | HPLC | MS | EXTRACTION | PROCESS | AUTOMATION

PRODUCT MANUAL

for the

ANION SELF-REGENERATING NEUTRALIZER 300

ASRN 300 (4-mm), P/N 067526

CATION SELF-REGENERATING NEUTRALIZER 300

CSRN 300 (4-mm), P/N 067525

Document No. 034962
Revision 07
February 2009

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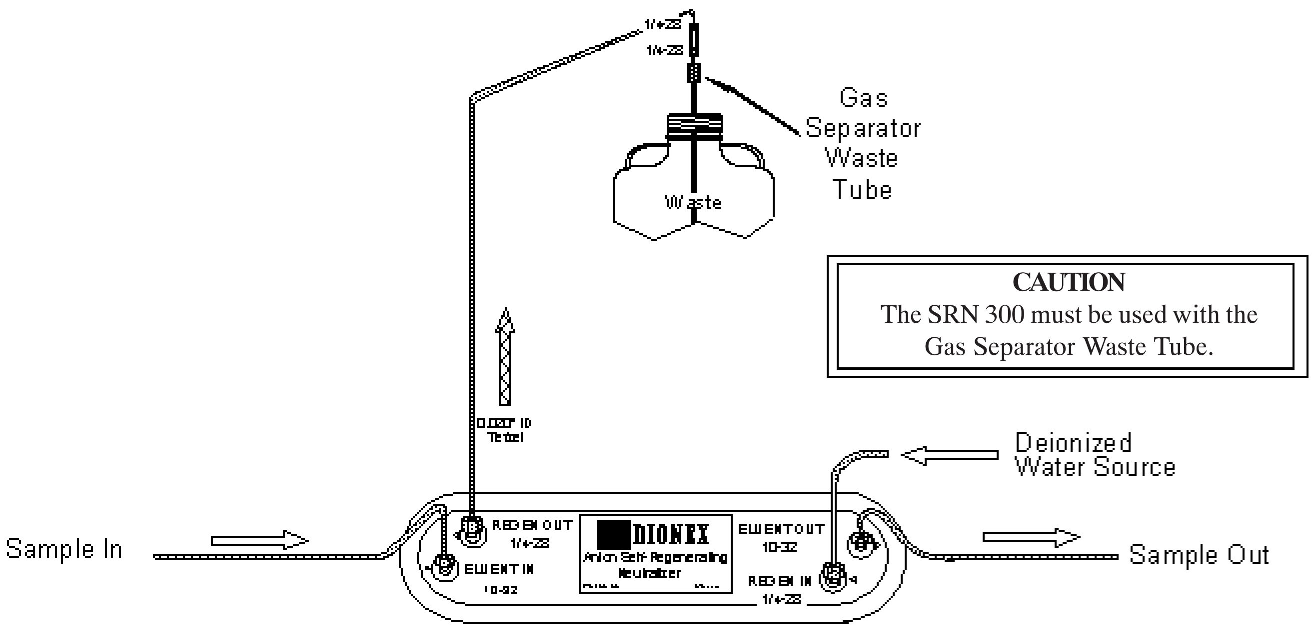
SECTION 1 - INTRODUCTION

The Dionex Anion or Cation Self-Regenerating Neutralizer (SRN 300) replaces the SRN-II neutralizer product line. The SRN 300 is designed with exterior hardware changes that allow the neutralizer to be more pressure tolerant than previous generation neutralizer devices.

1.1 The Self -Regenerating Neutralizer (SRN 300)



CAUTION
The SRN 300 must be used with the
Gas Separator Waste Tube.

Figure 1
The Self-Regenerating Neutralizer Plumbing Diagram

The Dionex Anion or Cation Self-Regenerating Neutralizer (SRN 300) is a membrane-based high capacity electrolytic device specially designed for neutralizing basic or acidic matrices. Additionally, the SRN 300 neutralizer is composed of solvent cleaned components to facilitate a low level of blank during analysis. By neutralizing the matrix ions, the SRN 300 facilitates trace analysis of anionic or cationic analytes. For example, analysis of trace anions in concentrated sodium hydroxide is possible after neutralizing the base with the ASRN 300 neutralizer.

Assistance is available for any problem that may be encountered during the shipment or operation of Dionex instrumentation and columns through the Dionex North America Technical Call Center at 1-800-DIONEX-0 (1-800-346-6390) or through any of the Dionex Offices listed in "Dionex Worldwide Offices" on the Dionex Reference Library CD-ROM.

The SRN 300 is comprised of a sample pretreatment chamber that is separated from two regenerant chambers by two ion exchange membranes. The sample and the regenerant chambers have ion exchange gasketed screens that define the flow channels. Electrodes are placed along the length of the regenerant channel for electrolysis reactions. The sample is flowed into the sample chamber in one direction while the regenerant is flowed into the regenerant chamber in the opposite direction.

When an electrical potential is applied across the electrodes, water from the regenerant channels is electrolyzed, supplying regenerant hydronium ions (H_3O^+) in ASRN 300 or hydroxide ions (OH^-) in CSRN 300 for the sample neutralization reaction. The membranes allow these hydronium or hydroxide ions to pass into the sample neutralization chamber, resulting in the conversion of the matrix ions of the sample to a weakly ionized form. At the same time, sample counter ions (cations in bases or anions in acids) are passed simultaneously into the regenerant chamber to maintain charge balance.

NOTE
The ports marked ELUENT IN and ELUENT OUT are the ports to be used for the sample and sample carrier solution stream.

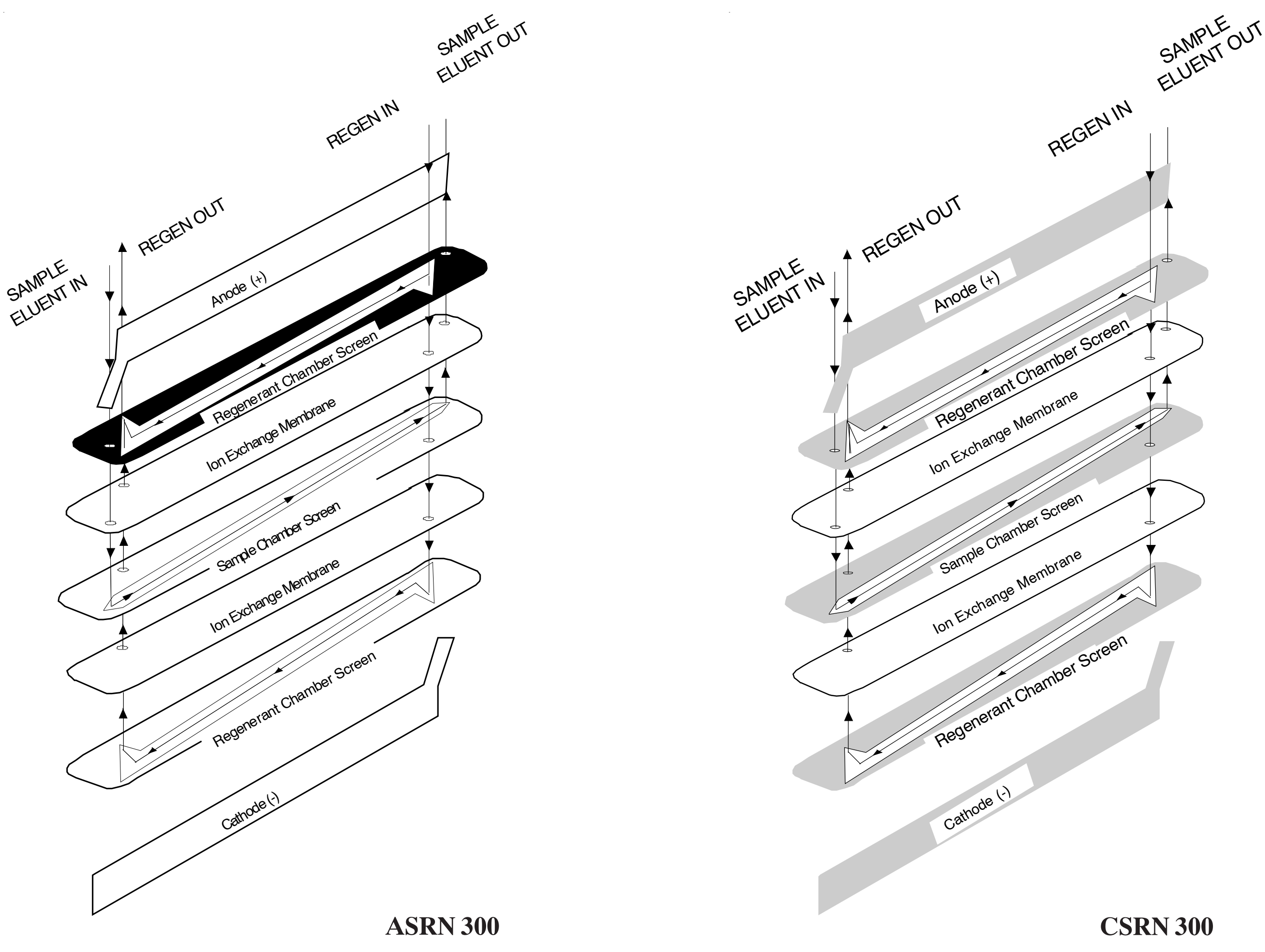
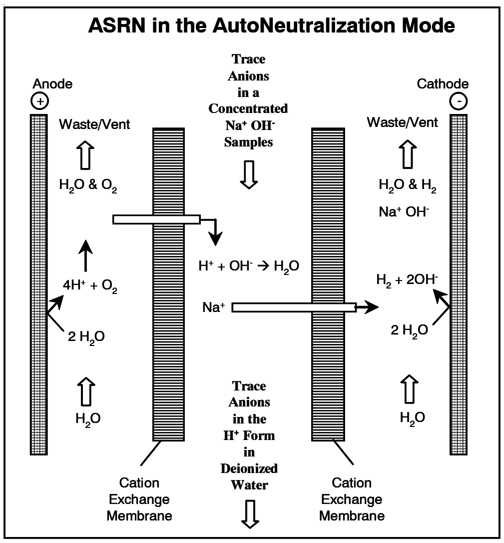


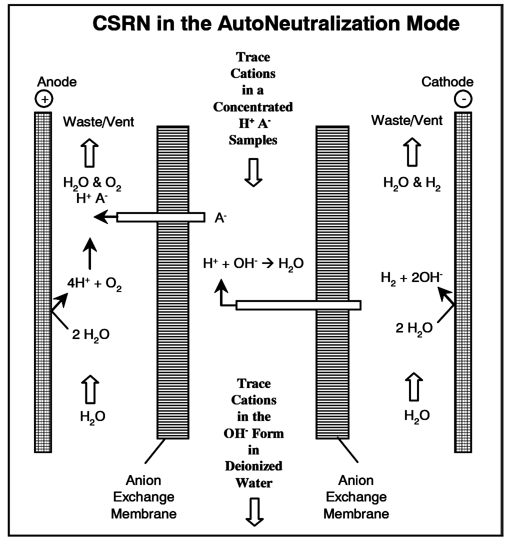
Figure 2
Electrode, Membrane, and Screen Configuration in the Self-Regenerating Neutralizer (SRN 300)

1.2 Neutralization Theory

The Self-Regenerating Neutralizer (SRN 300) requires a constant DI water flow of 3 to 5 mL/min through the regenerant chambers. The regenerant water can be delivered from a pressurized bottle or a pump.



ASRN 300



CSRN 300

Figure 3
AutoNeutralization of Concentrated Acid/Base with the Self-Regenerating Neutralizer (SRN 300)

As shown in Figure 3, when a potential is applied across the two electrodes, the water regenerant undergoes electrolysis forming hydronium ions at the anode and hydroxide ions at the cathode along with electrolytic gases at the two electrodes. In the ASRN 300, due to the applied potential and the presence of cation exchange materials, hydronium is transported from the anode towards the cathode and enters the sample chamber. At the same time, the matrix cations in the sample move towards the cathode and enter the cathode chamber. The matrix cations combine with the hydroxide ions at the the cathode forming a base and are removed out of the regenerant channel in a direction counter current to the sample flow direction. The net effect of this transport of ions is neutralization of the sample matrix ions to a weakly ionized form while the sample anions are typically converted to a strongly ionized form. The sample anions are now in a weakly dissociated matrix such as water and are ready for analysis.

In the CSRN 300, due to the applied potential and the presence of anion exchange materials, hydroxide is transported from the cathode towards the anode and enters the sample chamber. At the same time, the matrix anions in the sample move towards the anode and enter the anode chamber. The matrix anions combine with the hydronium ions at the anode forming an acid and are removed out of the regenerant channel in a direction counter current to the sample flow direction. The net effect of this transport of ions is neutralization of the sample matrix ions to a weakly ionized form while the sample cations are typically converted to a strongly ionized form. The sample cations are now in a weakly dissociated matrix such as water and are ready for analysis.

1.3 Trace Ion Analysis After Neutralization

After neutralization, the sample can be fed into any Dionex Ion Chromatography system equipped with suppressed conductivity detection and operated in the concentration mode. It is important that only the low pressure concentrator columns be used to protect the SRN 300 from damage.

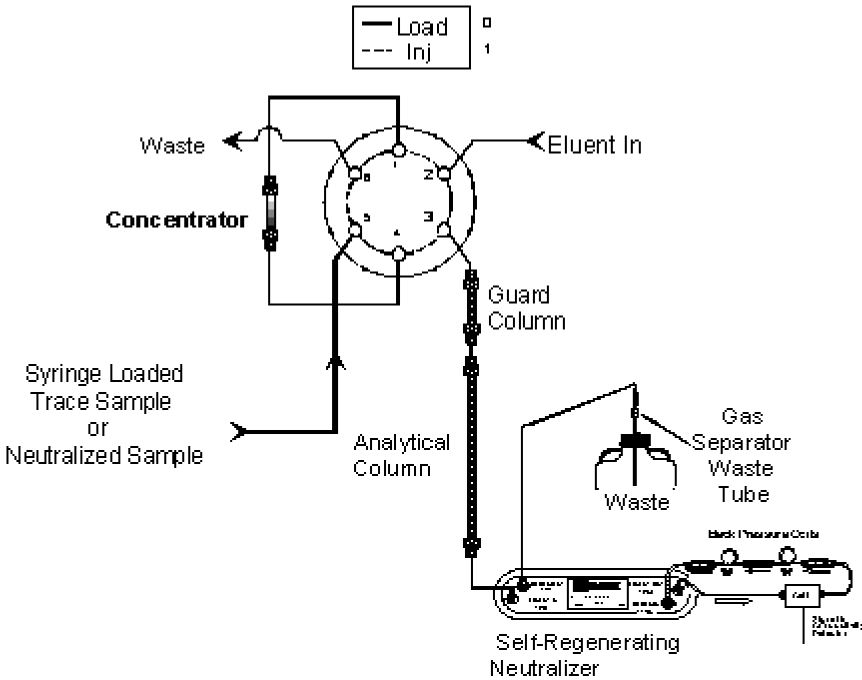


Figure 4
Ion Chromatography Equipped with the Low Pressure Concentrator

Use only the Low Pressure Trace Anion Concentrator (TAC-LP1) Column (P/N 046026) with the ASRN 300. Use of other concentrator columns, unless designated as low pressure, may damage the ASRN 300! For detailed information on the theory and operation of the Low Pressure Trace Anion Concentrator (TAC-LP1) Column, consult the Product Manual for the Low Pressure Trace Anion Concentrator TAC-LP1 Column (Document No. 034792).

Use only the Low Pressure Trace Cation Concentrator (TCC-LP1) Column (P/N 046207) with the CSRN 300. Use of other concentrator columns, unless designated as low pressure, may damage the CSRN 300! For detailed information on the theory and operation of the Low Pressure Trace Cation Concentrator (TCC-LP1) Column, consult the Product Manual for the Low Pressure Trace Cation Concentrator TCC-LP1 Column, (Document No. 034793).

1.3.1 Selection of Guard and Analytical Columns for Trace Anion Separation

Depending on your specific application, any anion exchange guard and analytical column set can be used for trace anion analysis.

1.3.2 Selection of the Suppressor for Anion Determination

Use the Anion Self-Regenerating Suppressor 300 (4-mm, P/N 061561). For detailed information on the theory and operation of the ASRS 300, see the Product Manual for the Anion Self-Regenerating Suppressor (Document No. 031956).

1.3.3 Selection of Guard and Analytical Columns for Trace Cation Separation

The TCC-LP1 is used only with carboxylated columns. Depending on your specific application, any carboxylated cation exchange guard and analytical column set can be used.

1.3.4 Selection of the Suppressor for Cation Determination

Use the Cation Self-Regenerating Suppressor 300 (4-mm, P/N 061563). For detailed information on the theory and operation of the CSRS 300, see the Product Manual for the Cation Self-Regenerating Suppressor (Document No. 031956).

1.4 Shipment and Storage

1.4.1 Shipment

CAUTION ***The Self-Regenerating Neutralizers (ASRN 300 and CSRN 300) contain components that are heat sensitive. The neutralizer should not be subjected to temperatures above 40°C during shipment, storage or operation.***

1.4.2 Storage

CAUTION ***Ensure the neutralizer is stored in a temperature controlled environment away from direct exposure to sunlight or other sources of heat. Do not store the neutralizer in a non-temperature controlled environment where temperatures in excess of 40°C are commonly experienced, such as a parked car, a tool shed, or a lab-bench in close proximity to an open window with direct sunlight.***

SECTION 2 - INSTALLATION

2.1 Installation of the Self-Regenerating Neutralizer on ICS-2500 or Equivalent Systems

For detailed information on installation of the Self-Regenerating Neutralizer (SRN 300) on ICS-2500 or equivalent systems, refer to Dionex Application Note 93 and 94.

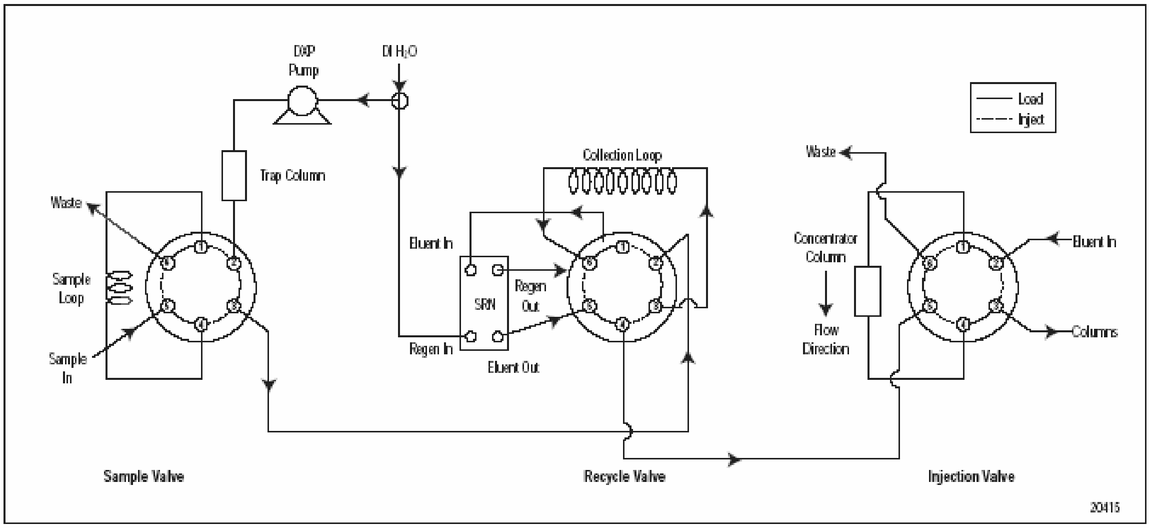


Figure 5
The Self-Regenerating Neutralizer (SRN 300) Plumbing Diagram

2.1.1 Sample Liquid Line Connections for Self-Regenerating Neutralizer (SRN 300)

A. Install the Self-Regenerating Neutralizer (SRN 300) in the LC30 or equivalent chromatography module.

B. Connect the eluent line from Port #1 on the Recycle Valve to the **ELUENT IN** port of the SRN 300.

C. Connect the **ELUENT OUT** port of the SRN 300 to Port #5 on the Recycle Valve.

CAUTION
Back pressures over 150 psi after the SRN 300can cause irreversible damage!

2.1.2 Regenerant Liquid Line Connections forSelf-Regenerating Neutralizer (SRN 300)

The **SP10 AutoNeutralizer Ship Kit** (P/N 047950) contains all of the components needed to install and operate the SRN 300 with a pressurized water reservoir. See Section 2.5 “Important System Assemblies” for important assemblies.

A. Make the following air line connections:

1. Locate the pieces of tinted 1/8" OD plastic tubing (P/N 030089) supplied in the Ship Kit.
2. Push the end of one piece of 1/8" OD tubing over the barbed fitting of the regulator. Connect the other end of the tubing to the source of air pressure.
3. Push one end of the second piece of 1/8" OD tubing over the other barbed fitting of the regulator. Push the other end of this tubing over the barbed fitting (P/N 030077) in the pressure inlet of the plastic reservoir. (see Figure 7, “Configuration of the Pressurized Water Reservoir and the Gas Separator Waste Tube with the Self-Regenerating Neutralizer”).

B. Make the following liquid line connections:

1. See Figure 7, “Configuration of the Pressurized Water Reservoir and the Gas Separator Waste Tube with the Self-Regenerating Neutralizer.”
2. Use a coupler (P/N 039056) to connect one end of the 30" tubing assembly (P/N 035727) that comes in the SP10 AutoNeutralization Ship Kit to the water reservoir. Connect the other end of this tubing to the **REGEN IN** port of the Self-Regenerating Neutralizer.
3. Using a coupler (P/N 039056) and a 1/8" OD piece of tubing (P/N 035728) from the SP10 AutoNeutralization Ship Kit, connect one end of this line to the **REGEN OUT** port of the SRN 300 and then connect the other end of the line to the **Gas Separator Waste Tube.**
4. Fill the reservoir with water. Make sure that the O-ring is inside the cap of the reservoir before screwing the cap onto the reservoir. Screw the cap onto the reservoir tightly and place the reservoir near the instrument.

2.2 Installation of the Self-Regenerating Neutralizer on SP10 AutoNeutralizer

For detailed information on installation of the Self-Regenerating Neutralizer (SRN 300) on SP10 AutoNeutralizer, refer to the SP10 AutoNeutralizer Operator's Manual, Document No. 034980.

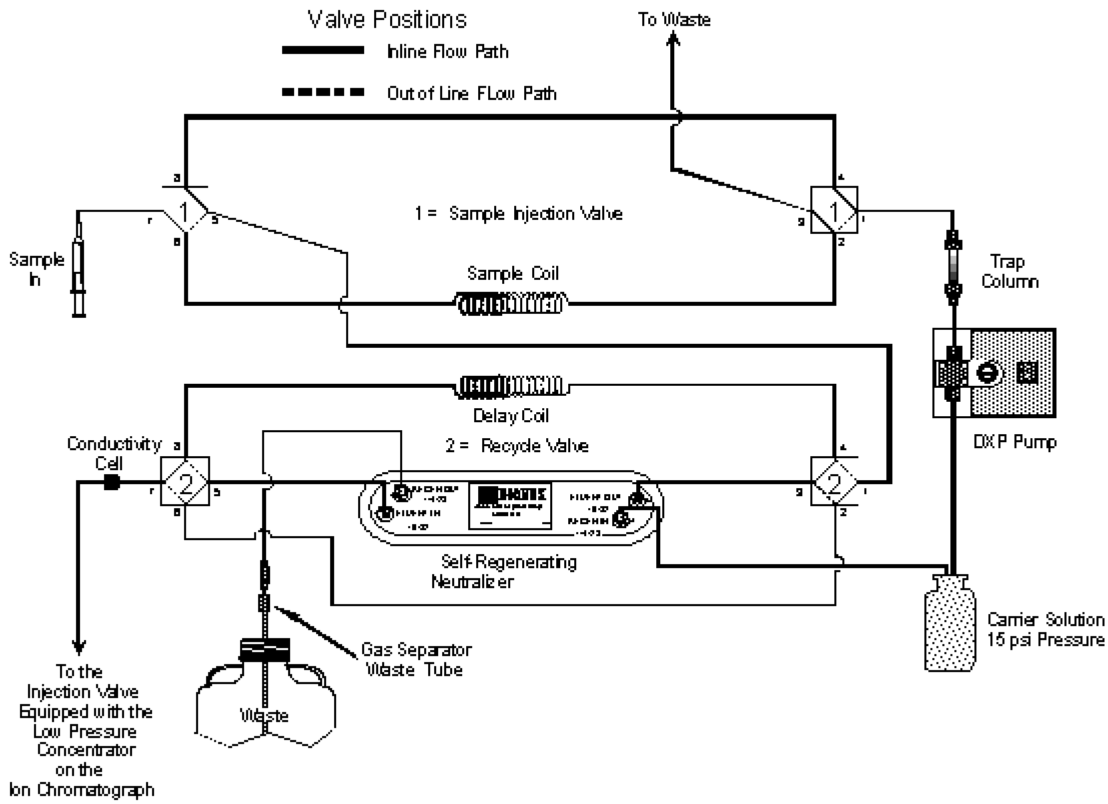


Figure 6
The Self-Regenerating Neutralizer (SRN 300) Plumbing Diagram

2.2.1 Sample Liquid Line Connections for Self-Regenerating Neutralizer (SRN 300)

A. Install the Self-Regenerating Neutralizer (SRN 300) in the SP10 AutoNeutralizer module.

B. Connect the eluent line from Port #5 on the Recycle Valve to the **ELUENT IN** port of the SRN.

C. Connect the **ELUENT OUT** port of the SRN 300 to Port #3 on the Recycle Valve.

CAUTION
Back pressures over 100 psi after the SRN 300 can cause irreversible damage!

2.2.2 Regenerant Liquid Line Connections for Self-Regenerating Neutralizer (SRN 300)

The **SP10 AutoNeutralizer Ship Kit** (P/N 047950) contains all of the components needed to install and operate the SRN with a pressurized water reservoir. See Section 2.5 “Important System Assemblies” for important assemblies.

A. Make the following air line connections:

1. Locate the pieces of tinted 1/8" OD plastic tubing (P/N 030089) supplied in the Ship Kit.

2. Push one end of one piece of 1/8" OD tubing over the barbed fitting of the regulator. Connect the other end of the tubing to the source of air pressure.

3. Push one end of the second piece of 1/8" OD tubing over the other barbed fitting of the regulator. Push the other end of this tubing over the barbed fitting (P/N 030077) in the pressure inlet of the plastic reservoir. See Figure 7, “Configuration of the Pressurized Water Reservoir and the Gas Separator Waste Tube with the Self-Regenerating Neutralizer”.

B. Make the following liquid line connections:

1. See Figure 7, “Configuration of the Pressurized Water Reservoir and the Gas Separator Waste Tube with the Self-Regenerating Neutralizer.”

2. Use a coupler (P/N 039056) to connect one end of the 30" tubing assembly (P/N 035727) that comes in the SP10 AutoNeutralizer Ship Kit to the water reservoir. Connect the other end of this tubing to the **REGEN IN** port of the Self-Regenerating Neutralizer.

3. Using a coupler (P/N 039056) and a 1/8" OD piece of tubing (P/N 035728) from the SP10 AutoNeutralizer Ship Kit, connect one end of this line to the **REGEN OUT** port of the SRN 300 and then connect the other end of the line to the **Gas Separator Waste Tube.**

4. Fill the reservoir with water. Make sure that the O-ring is inside the cap of the reservoir before screwing the cap onto the reservoir. Screw the cap onto the reservoir tightly and place the reservoir near the instrument.

2.3 The Gas Separator Waste Tube

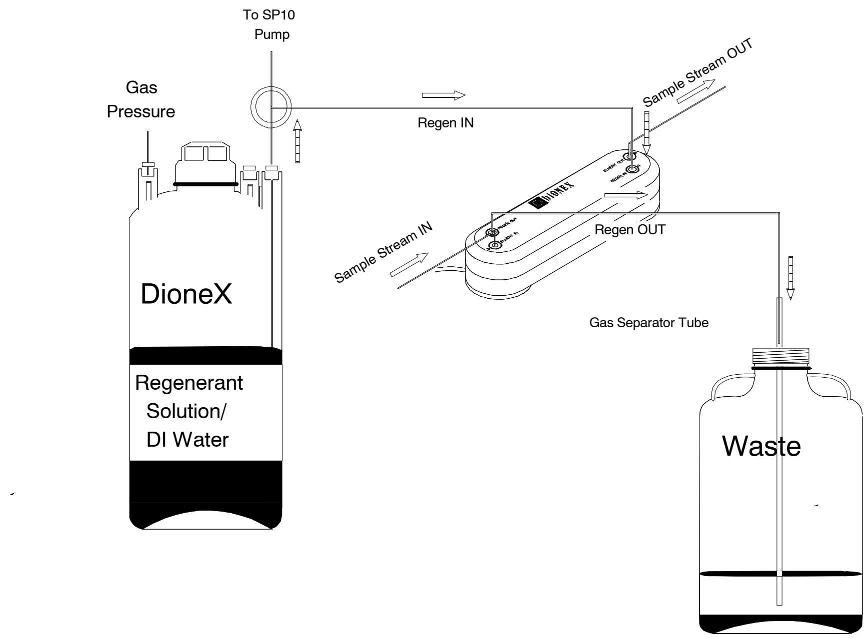


Figure 7
Configuration of the Pressurized Water Reservoir and the Gas Separator Waste Tube with the Self-Regenerating Neutralizer

The Gas Separator Waste Tube (P/N 045460) is an integral part of the Self-Regenerating Neutralizer (SRN 300) system. Its function is to ensure the separation of any gas generated in the SRN 300 during the electrolysis. The Gas Separator Waste Tube is used to avoid concentrating the gas in the waste container.

CAUTION
Do NOT cap the waste reservoir!

The very small amount of hydrogen gas generated by the SRN 300 is not dangerous unless the gas is trapped in a closed container and allowed to concentrate. The Gas Separator Waste Tube must be open to the atmosphere and not in a confined space to operate properly.

Assemble and install the **Gas Separator Waste Tube** and waste line following the steps below (see Figure 7, “Configuration of the Pressurized Water Pump and the Gas Separator Waste Tube with the Self-Regenerating Neutralizer”).

A. Use one or two couplers (P/N 045463) to connect two or three lengths of 1/2" ID black polyethylene tubing (P/N 045462) depending of the depth of your waste container. It is important that the top of the Waste Separator Tube extend above the top of the Waste container as shown in Figure 7, “Configuration of the Pressurized Water Reservoir and the Gas Separator Waste Tube with the Self-Regenerating Neutralizer.”

B. Place the Gas Separator Waste Tube with the 1/8" OD tubing attached into the waste container. Be sure that the bottom of the Gas Separator Waste Tube is resting on the floor of the waste container and the top of the device (where the white 1/8" OD tubing meets the black 1/2" OD tubing) is above the top of the container. Ensure that the Gas Separator Waste Tube and the waste container are open to the atmosphere (see Figure 7, “Configuration of the Pressurized Water Reservoir and the Gas Separator Waste Tube with the Self-Regenerating Neutralizer”).

2.4 Pump Trap Column Installation and Regeneration

2.4.1 Installation of the AG10 4-mm Guard Column

The AG10 4-mm Guard Column (P/N 043119) is used to trap anions that may be in the carrier solution which could result in high blanks. The AG10 should be regenerated prior to installation in the SP10 AutoNeutralizer (see Section 2.4.2, “Regeneration of the AG10 4-mm Guard Column”).

2.4.2 Regeneration of the AG10 4-mm Guard Column

Using a pump other than the sample carrier pump, the AG10 4-mm Guard column should be regenerated and rinsed using the following two steps:

A. Regenerate it with 0.5 M NaOH for 50 min at a flow rate of 1 mL/min.

B. Rinse it with **Type I Reagent Grade Water** (see Section 3.2.3, “Deionized Water”) for 30 min at a flow rate of 1 mL/min. If the carrier solution is different from water, equilibrate it to the carrier solution for 30 min at a flow rate of 1 mL/min.

2.4.3 Installation of the CG10 4-mm Guard Column

The CG10 4-mm Guard Column (P/N 043016) is used to trap cations that may be in the carrier solution which could result in high blanks. The CG10 should be regenerated prior to installation. (See Section 2.4.4, "Regeneration of the CG10 4-mm Guard Column".)

2.4.4 Regeneration of the CG10 4-mm Guard Column

Using a pump other than the carrier pump, the CG10 4-mm Guard column should be regenerated and rinsed using the following two steps:

A. Regenerate it with 0.5 M H_2SO_4 for 50 min at a flow rate of 1 mL/min.

B. Rinse it with **Type I Reagent Grade Water** (see Section 3.2.3, “Deionized Water”) for 30 min at a flow rate of 1 mL/min. If the carrier solution is different from water, equilibrate it to the carrier solution for 30 min at a flow rate of 1mL/min.

2.5 Important System Assemblies

Part No.	Item	
067526	**Anion Self-Regenerating Neutralizer 300 (ASRN 300)**	
or		
067525	**Cation Self-Regenerating Neutralizer 300 (CSRN 300)**	
047950	**SP10 AutoNeutralizer Ship Kit**	
	016640	5 mL Plastic Syringe
	038201	Gas Regulator Assembly
	048485	4 L Bottle Assembly
	042950	50 μL Sample Coil Assembly
	048435	2 mL Sample Collection Coil
	048436	Waste Gas Separator Tube Kit
	045460	Gas Separator Waste Tube
	038018	Pressurized Water Delivery System
	024305	Syringe Adapter, female Luer-lock, 1/4-28 threads
043199	**AG10 4-mm Guard Column**	
or		
043016	**CG10 4-mm Guard Column**	

CAUTION

The SRN 300 must be operated with the Gas Separator Waste Tube (P/N 045460)

SECTION 3 - OPERATION

For detailed information on the operation of Dionex IC systems, consult the accompanying Product Manuals.

3.1 Operation of the SP10 AutoNeutralizer

For detailed information on the operation of the SP10 AutoNeutralizer, see the SP10 AutoNeutralizer Operator's Manual, Document No. 034980.

3.2 Chemical Purity Requirements for the Self-Regenerating Neutralizer SRN 300

Obtaining precise and accurate results requires eluents that are free of ionic impurities. Chemicals and deionized water used to prepare eluents must be of the purities described below. Eluents and Regenerants should have low trace impurities and low particulate levels to help protect your SRN 300 and system components from contamination. Dionex cannot guarantee proper SRN 300 performance when the quality of the chemicals and water used to prepare eluents has been compromised.

3.2.1 Inorganic Chemicals

Reagent Grade inorganic chemicals should always be used to prepare ionic eluents. Whenever possible, inorganic chemicals that meet or surpass the latest American Chemical Society standard for purity (universally accepted standard for reagents) should be used. These inorganic chemicals will detail the purity by having an actual lot analysis on each label.

3.2.2 Solvents

Since solvents may be used to cleanup the SRN 300, the solvents used must be free of ionic impurities. However, since most manufacturers of solvents do not test for ionic impurities, it is important that the highest grade of solvents available be used. Currently, several manufacturers are making ultrahigh purity solvents that are compatible for HPLC and spectrophotometric applications. These ultrahigh purity solvents will usually ensure that your chromatography is not affected by ionic impurities in the solvent. At Dionex, we have obtained consistent results using High Purity Solvents manufactured by Burdick and Jackson and Optima® Solvents by Fisher Scientific.

3.2.3 Deionized Water

The deionized water used should be **Type I Reagent Grade Water** with a specific resistance of 18.2 megohm-cm. The deionized water should be free of ionized impurities, organics, microorganisms and particulate matter larger than 0.2 μm. It is good practice to filter eluents through a 0.2 μm filter whenever possible. Bottled HPLC-Grade Water should not be used since most bottled water contains an unacceptable level of ionic impurities. Finally, thoroughly degas all deionized water prior to preparing any eluents or regenerants.

3.3 Start Up Procedure for the Self-Regenerating Neutralizer (SRN 300)

CAUTION
The membranes in the Anion Self-Regenerating Neutralizer (SRN 300) must be completely hydrated to maintain liquid seals.

This requirement is achieved by maintaining the regenerant chamber full of the appropriate regenerant solution or water, to ensure that the membranes remain properly hydrated. Occasionally, some of the regenerant solution evaporates during long term storage. Before install your new SRN 300, follow the hydration steps below.

3.3.1 Hydration of the Self-Regenerating Neutralizer (SRN 300)

A. Using a disposable plastic syringe and the 10-32 Luer adapter, push 3 ml of DI water through the **Eluent Out** port of the ASRN 300 or CSRN 300.

B. Using a disposable plastic syringe and the 1/4-28 Luer adapter, push 5 ml of DI water through the **Regen In** port of the ASRN 300 or CSRN 300.

C. Allow the neutralizer to sit for approximately 20 minutes to ensure that the membranes and the screens are fully hydrated before plumbing into the system.

3.3.2 Equilibration of the Self-Regenerating Neutralizer (SRN 300)

After the hydration, refer to Section 2 for detailed instructions on SRN 300 installation. The regenerant DI water can be supplied by either a pressurized bottle delivery system or a pump. The optimal regenerant flow rate is approximately 3 to 5 mL/min. Set the sample carrier pump flow rate at 0.5 mL/min. Turn on the power to the neutralizer and let the neutralizer equilibrate. It is important to condition and remove trace ion contamination from the Self-Regenerating Neutralizer prior to any sample analysis. For optimal performance, the background conductivity of the DI water from the neutralizer should be below 2 μS. For CSRN 300 conditioning, refer to Section 4.2 for details. For additional information, see the SP10 AutoNeutralizer Operator's Manual, Document No. 034980.

3.4 Operation Principle of Self-Regenerating Neutralizer (SRN 300)

Note
Although it is possible that in certain analyses the SRN 300 Carrier Solution and Regenerant may be different, in most analyses, the Carrier Solution and the Regenerant are both deionized water.

AutoNeutralization pretreatment coupled with Ion Chromatography involves the following steps:

A. The concentrated sample is loaded into the sample loop of the “Sample Valve” with either a syringe or an auto-sampler.

B. The sample loop is switched in-line and flushed with a stream of deionized water

C. The deionized water pushes the sample through the SRN 300 where neutralization begins.

D. Common bases or acids that have been diluted four fold or more can be completely neutralized after one pass through the SRN 300 at flow rates $\leq$ 0.5 ml/min.

E. For more concentrated bases or acids or faster flow rates, multiple passes through the SRN 300 will be required for complete neutralization.

F. The partially neutralized sample is passed down to the 2-mL collection loop on the Recycle Valve.

G The recycle valve is actuated so that that the sample flow is directed to pass through the SRN 300 a second time to completely neutralize the sample.

H. The Recycle Valve can be actuated multiple times if multiple passages are necessary for complete neutralization.

I. After complete neutralization, the sample is delivered to the injection valve of any Ion Chromatography system equipped with suppressed conductivity detection and a Low Pressure Trace Anion TAC-LP1 Concentrator Column (P/N 046026) for the ASRN 300 or a Low Pressure Trace Cation TCC-LP1 Concentrator Column (P/N 046027) for the CSRN 300.

J. The trace ions are then analyzed by the standard IC method.

For detailed information on the timing and configuration of Sample valve, Recycle Valve and Injection Valve, refer to Dionex Application Note 93 and 94 or SP10 AutoNeutralizer Operator’s Manual (Document No. 034980).

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

SECTION 4 - EXAMPLE APPLICATIONS

The following examples are presented to highlight the analysis of trace anions or cations found in the concentrated matrices that have been treated using the Self-Regenerating Neutralizer (SRN 300).

The analysis may be done on any one of a number of Dionex anion or cation exchange column sets (see Section 1.3.1 for anion exchange column sets and Section 1.3.3 for cation exchange column sets). Please refer to the Product Manual for the particular anion or cation exchange column set installed in your system for detailed operation and troubleshooting information.

Because of its low operating pressure, the Low Pressure Trace Anion Concentrator TAC-LP1 Column or the Low Pressure Trace Cation Concentrator TCC-LP1 Column must be used to concentrate the trace anions or cations in the sample neutralized by the SRN 300. The use of other concentrators may result in excessive back pressure to the SRN 300, thus damaging the SRN 300.

4.1 System Blank

4.1.1 Anion System Blank

Trace anion contamination in the deionized water used for the carrier solution, the ASRN 300 and eluent constitutes create the analytical blank. For trace analysis, the analytical blank usually determines the detection limits of the system. Anion contaminant in deionized water can be removed by installing an IonPac AG10 Guard Column between the sample carrier pump and the Sample Valve. The major source of sulfate contamination is usually from the ASRN 300. Since this device uses high capacity surface sulfonated ion exchange screens and membranes, sulfate is released from the screen and membrane surfaces, especially in a newly installed ASRN 300.

To reduce the sulfate blank, follow the SRN 300 startup procedure in Section 3.3, "Start up Procedure for the Self-Regenerating Neutralizer (SRN 300)." The sulfate blank is normally reduced to a constant level after 24 hours of operation. Typical blanks are shown in Figure 8, "IonPac AS11 Gradient Analysis of Blank, Standard, 10% NaOH Sample and spiked 10% NaOH Sample" and Figure 9, "IonPac AS12A Isocratic Analysis of Blank, Standard, 25% NaOH Sample and Spiked 25% NaOH Sample." Finally, anion contaminants in the eluent (e.g., Na_2CO_3/$NaHCO_3$ and NaOH) also contribute to the analytical blank. A choice of high purity chemicals helps to reduce the blank concentrations.

4.1.2 Cation System Blank

Trace cation contamination in the deionized water used for the carrier solution, the CSRN 300 and eluent constitutes create the analytical blank. For trace analysis, the analytical blank usually determines the detection limits of the system. Cation contaminants in deionized water can be removed by installing an IonPac CG10 Guard Column between the sample carrier pump and the Sample Valve. The major source of ammonia and amine contamination is usually from the CSRN 300. Since this device uses high capacity aminated ion exchange screens and membranes, ammonia and amines are released from the screen and membrane surfaces, especially in a newly installed CSRN 300.

To reduce the amine blank, follow the SRN 300 start up procedure in Section 3.3, "Start up Procedure for the Self-Regenerating Neutralizer (SRN 300)." The amine blank is normally reduced to a constant level after 24 hours of operation. A typical blank is shown in Figure 13A, "IonPac CS12A Blank and Standard Analysis." The use of high purity chemicals helps to reduce the blank concentrations.

4.2 CSRN 300 Conditioning

Since the CSRN 300 is a high-capacity anion exchange device which supplies the high concentration of hydroxide for acid neutralization, the hydrolysable ions such as Mg^{2+} and Ca^{2+} in hydroxide forms may be precipitated in the CSRN 300. In general, when standard cation in water solutions are employed for system calibration, it is possible that Mg^{2+} and Ca^{2+} may be precipitated due to the high hydroxide concentration. These cations are then "carried over" to the first acid run. To avoid the hydrolysis of the alkaline earth metals during standard calibration, the CSRN 300 is "conditioned" by running a complete pretreatment cycle of acid (preferably 24% sulfuric acid) prior to standard runs. Follow the CSRN 300 acid treatment procedure below prior to each standard run.

A. Confirm that the system functions properly.

B. Set "Sample Valve" to Load Position and load 24% sulfuric acid.

C. Switch the sample valve to Inject position and flush the sample loop with deionized water.

D. After the sulfuric acid passes through the neutralizer and IC injection valve (approximately 4–6 minutes with the collection coil installed or 1-2 minutes without the collection coil installed), the system is ready for standard injection.

The above steps are applied only when standard calibration is performed. However, if the standard injection is made immediately after the sample runs (acid samples), the CSRN 300 acid pretreatment is not required.

If the standard run is not started within 20 minutes after the acid calibration step, the overall process must be repeated. When multiple point calibration is performed, the CSRN 300 acid pretreatment can be made anytime after the beginning of the analytical separation.

4.3 System Calibration

The analytical blank should be incorporated into the calibration curve. One or two level standards are usually required to calibrate the Ion Chromatograph. For trace analysis, typical standard concentrations are 2 to 5 times sample concentrations. For example, for anions, if the sample contains 50 ppb each of Cl^-, NO_3^- and SO_4^{2-}, the standard calibration should not exceed 250 ppb each of these anions. For cations, if the sample contains 10 ppb each of K^+, Mg^{2+} and Ca^{2+}, the standard calibration should not exceed 50 ppb each of these cations.

4.3.1 Analysis of Acid Samples Containing High Concentrations of Transition Metals

If a sample contains transition metals in ppm (mg/L) levels, these elements may interfere with cation detection by suppressed conductivity. The additional of complexing agent such as pyridine-2,6-dicarboxylic acid (PDCA, P/N 039671) to the CS12A eluent is required to selectively remove transition metals from the eluent via the suppression system.

4.4 Detection Limits of SRN 300 Sample Pretreatment and Subsequent Ion Chromatography

Anion	ppb*
Chloride	4
Bromide	20
Chlorate	30
Nitrate	20
Phosphate	50
Sulfate	30
Oxalate	50

* Estimated values in NaOH matrix

Cation	ppb*
Lithium	0.03
Sodium	1.0
Potassium	2.0
Magnesium	0.6
Calcium	0.8

* Estimated values in H_2SO_4 matrix

4.5 Recovery Data for Trace Anions in 25% Sodium Hydroxide

Anion	25% NaOH* (ppb)	Spike (ppb)	Expected (ppb)	Found* (ppb)
Chloride	155 + 3	100	255	258 + 4
Bromide	—	100	100	99 + 8
Chlorate	—	100	100	96 + 9
Nitrate	11 + 6	100	111	119 + 13
Phosphate	106 + 25	100	206	183 + 17
Sulfate	348 + 4	200	548	553 + 11
Oxalate	134 + 28	100	234	238 + 22

* n = 8

Separation Conditions are shown in Section 4.7, “IonPac AS12A Isocratic Analysis of 25% Sodium Hydroxide.”

4.6 Recovery Data for Trace Cations in 24% Sulfuric Acid

Cation	24% H_2SO_4* (ppb)	Spike (ppb)	Expected (ppb)	Found* (ppb)
Lithium	---	2.0	2.0	1.993 ± 0.008
Sodium	7.4 ± 0.4	8.0	15.4	14.3 ± 0.1
Potassium	3.3 ± 0.5	20.0	23.3	22.5 ± 0.2
Magnesium	2.0 ± 0.2	10.0	12.0	12.2 ± 0.2
Calcium	10.6 ± 0.1	20.0	30.6	33 ± 1

* n = 8

Separation Conditions are shown in Section 4.7, “IonPac CS12A Isocratic Analysis of 24% Sulfuric Acid”

4.7 Anion Example Applications

4.7.1 IonPac® AS11 Gradient

A. Without Guard

Eluent 1:	Type I DI Water	Eluent Flow Rate:	2.0 mL/min
Eluent 2:	5.0 mM NaOH		
Eluent 3:	100 mM NaOH		

TIME (min)	% E1	% E2	%E3	Comments
Equilibration				
0	90	10	0	0.5 mM NaOH for 7 min
7.0	90	10	0	
Analysis				
0.0	90	10	0	0.5 mM NaOH, Inject
0.2	90	10	0	Inject Valve to Load Position
2.0	90	10	0	0.5-5.0 mM NaOH in 3 min
5.0	0	100	0	5.0-38.25 mM NaOH in 10 min
15.0	0	65	35	

B. With Guard

Eluent 1:	Type I DI Water	Eluent Flow Rate:	2.0 mL/min
Eluent 2:	5.0 mM NaOH		
Eluent 3:	100 mM NaOH		

TIME (min)	% E1	% E2	%E3	Comments
Equilibration				
0	90	10	0	0.5 mM NaOH for 7 min
7.0	90	10	0	
Analysis				
0.0	90	10	0	0.5 mM NaOH, Inject
0.2	90	10	0	Inject Valve to Load Position
2.5	90	10	0	0.5-5.0 mM NaOH in 3.5 min
6.0	0	100	0	5.0-38.25 mM NaOH in 12 min
18.0	0	65	35	

NOTE

The steps for sample neutralization can be performed while the AS11 is equilibrating with the starting eluent (90% E1/10% E2).

Seven minutes are required at the beginning of the above program for equilibration of the AS11 with E1 prior to injecting the next sample. If the system is not used continuously, that is, the run program (equilibration plus analysis) is not started exactly every 22 minutes (without AG11) or 25 minutes (with AG11), the run program can be modified to start with 2 minutes of the highest eluent concentration for regeneration and then to equilibrate with E1 for 7 minutes with the next injection 9 minutes into the program.

4.7.2 IonPac AS11 Gradient Analysis of 10% Sodium Hydroxide

Sample:	100 μL of 10% NaOH neutralized in the SP10 AutoNeutralizer
Concentrator Column:	Low Pressure Trace Anion Concentrator (TAC-LP1, P/N 046026)
Trap Column:	Anion Trap Column (ATC-1, P/N 037151)
Guard Column:	AG11 Guard Column
Analytical Column:	AS11 Analytical Column
Eluents	E1: Type I Deionized Water E2: 5.0 mM NaOH E3: 100 mM NaOH
Eluent Flow Rate:	2.0 mL/min (4-mm)
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS 300 AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS 300
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	0.5 mM NaOH: 1 μS 35 mM NaOH: 3.5 μS
Expected System Operating Back Pressure:	Without Guard: 1,600 psi (11.03 MPa) With Guard: 1,850 psi (12.75 MPa)

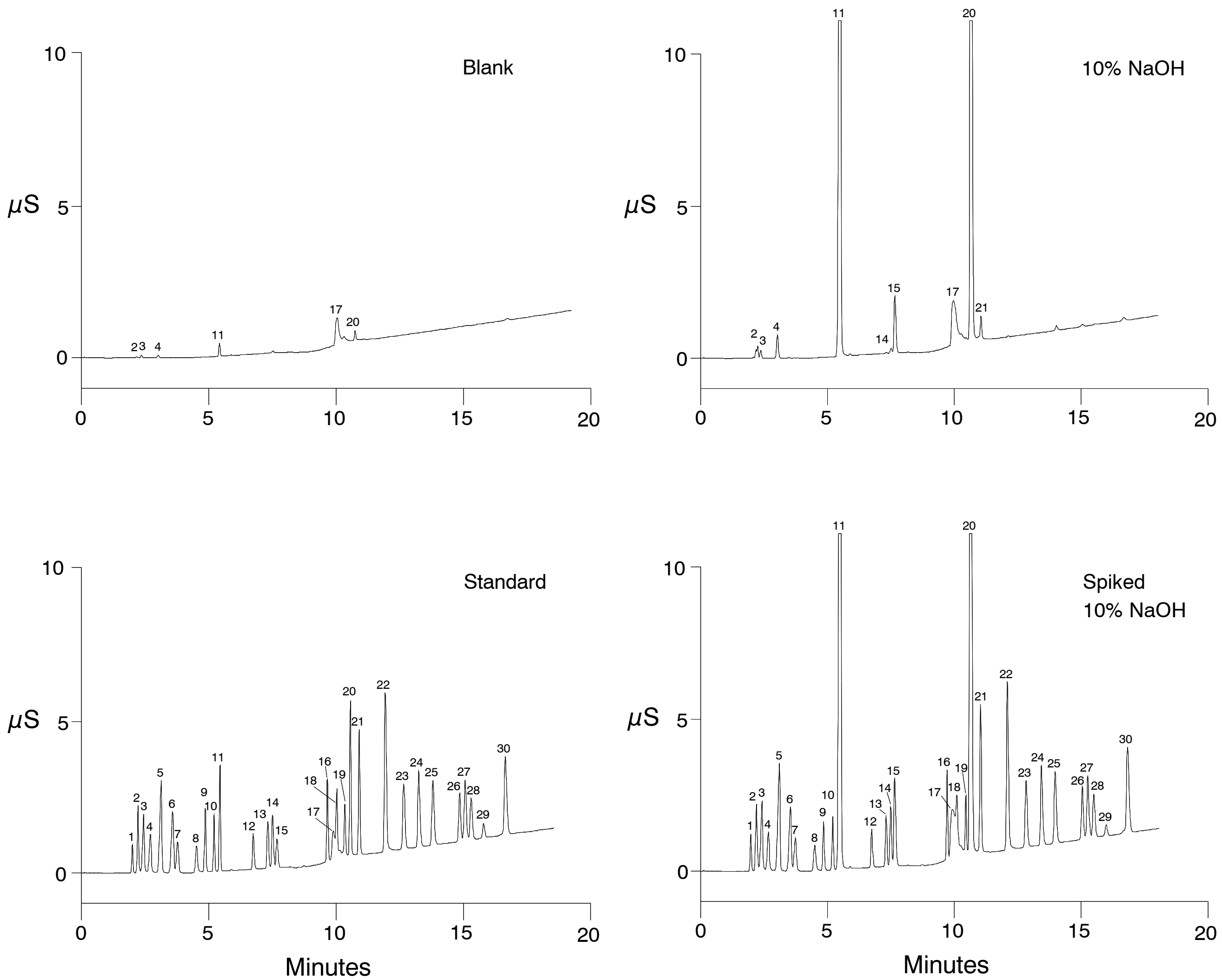


Figure 8
IonPac AS11 Gradient Analysis of Blank, Standard, 10% NaOH Sample and Spiked 10% NaOH Sample

Table 1
Recovery Data for the IonPac AS11 Analysis of 10% NaOH

	Standard Conc. (mg/L)	10% NaOH Conc. (mg/L)	Expected Spiked Value Conc. (mg/L)	10% NaOH Spiked Conc. (mg/L)
1. Quinate	1.000	ND	1.000	1.435
2. Fluoride	0.200	0.030	0.230	0.244
3. Acetate	1.000	0.081	1.081	1.301
4. Propionate	1.000	ND	1.000	1.069
5. Formate	1.000	0.213	1.213	1.216
6. Methylsulfonate	1.000	ND	1.000	1.070
7. Pyruvate	1.000	ND	1.000	1.103
8. Valerate	1.000	ND	1.000	1.009
9. Monochloroacetate	1.000	ND	1.000	0.811
10. Bromate	1.000	ND	1.000	1.036
11. Chloride	0.400	8.750	9.150	9.095
12. Trifluoroacetate	1.000	ND	1.000	1.055
13. Bromide	0.600	ND	0.600	0.640
14. Nitrate	0.600	0.048	0.648	0.657
15. Chlorate	0.600	1.178	1.778	1.870
16. Selenite	1.000	ND	1.000	0.990
17. Carbonate	---	---	---	---
18. Malonate	1.000	ND	1.000	1.303
19. Maleate	1.000	ND	1.000	1.026
20. Sulfate	1.000	9.842	10.842	10.925
21. Oxalate	1.000	0.166	1.166	1.244
22. Tungstate	2.000	ND	2.000	2.201
23. Phthalate	2.000	ND	2.000	2.086
24. Phosphate	2.000	ND	2.000	2.120
25. Chromate	2.000	ND	2.000	2.287
26. Citrate	2.000	ND	2.000	2.214
27. Tricarballylate	2.000	ND	2.000	2.098
28. Isocitrate	2.000	ND	2.000	2.112
29. cis-Aconitate	2.000	ND	2.000	1.512
30. trans-Aconitate	2.000	0.072	2.072	2.303

ND = None Detected

4.7.3 IonPac AS12A Isocratic Analysis of 25% Sodium Hydroxide

Sample:	100 μL of 25% NaOH neutralized in the SP10 AutoNeutralizer
Concentrator Column:	Low Pressure Trace Anion Concentrator (TAC-LP1, P/N 046026)
Column:	IonPac AG12A + IonPac AS12A
Eluent:	2.7 mM Na_2CO_3/0.3 mM $NaHCO_3$
Eluent Flow Rate:	1.5 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS 300 AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS 300
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	14–16 μS

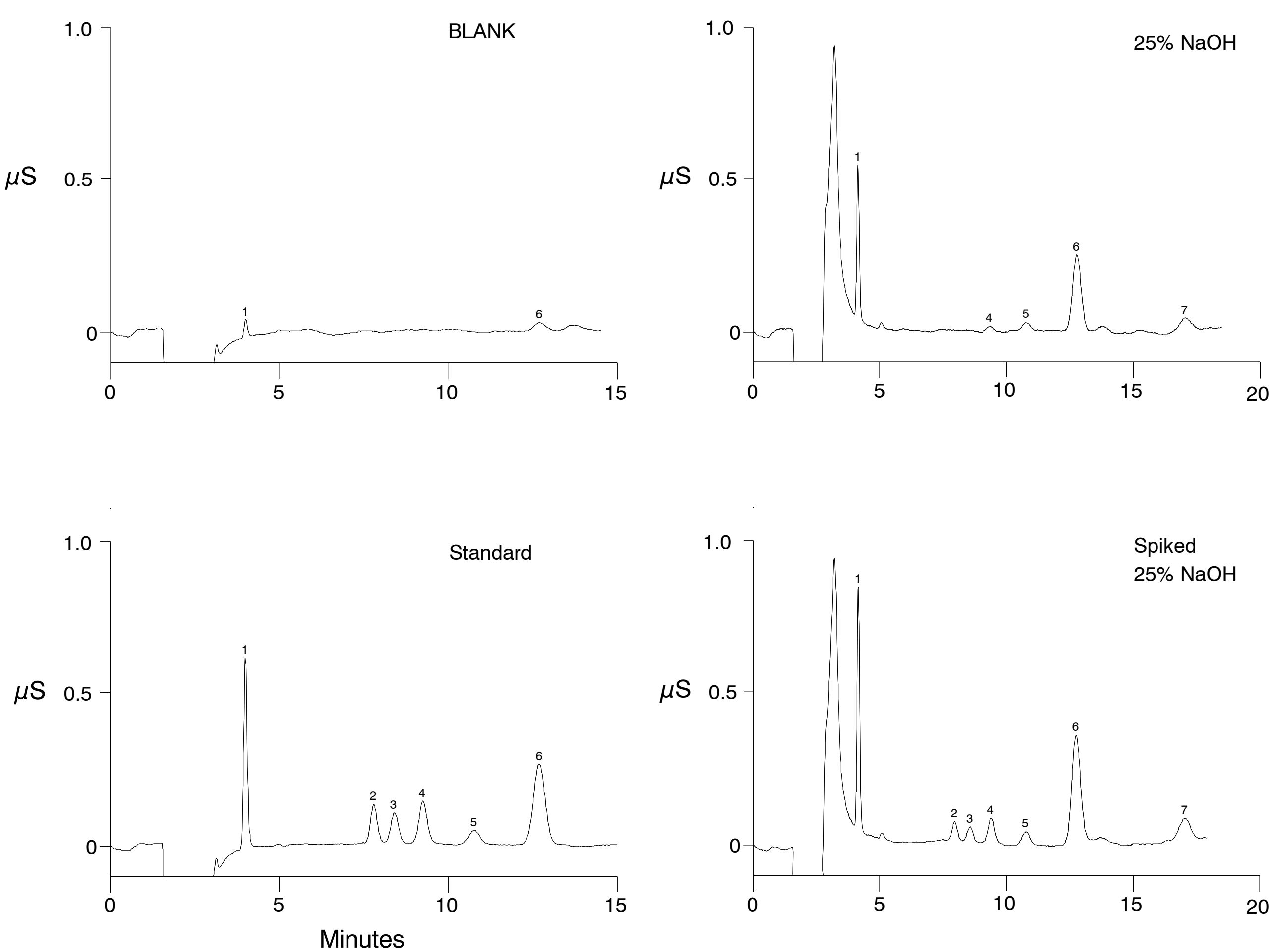


Figure 9
IonPac AS12A Isocratic Analysis of Blank, Standard, 25% NaOH Sample and Spiked 25% NaOH Sample

Table 2
Recovery Data for the IonPac AS12A Analysis of 25% NaOH

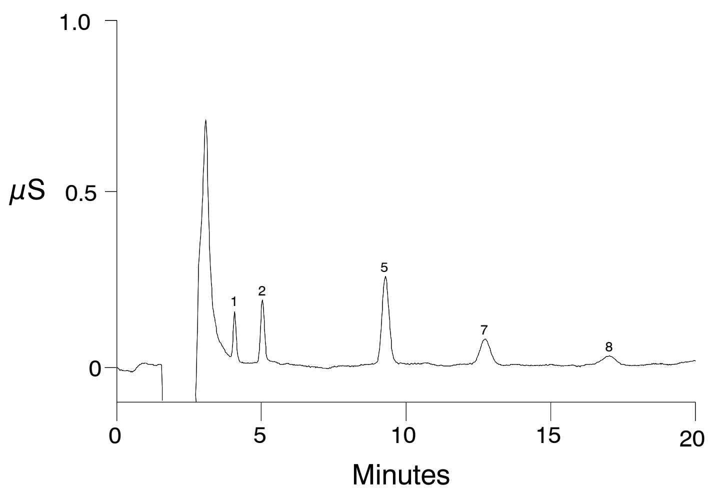
	Standard Conc. (mg/L)	Spiking Standard Conc. (mg/L)	25% NaOH Conc. (mg/L)	Expected Spiked Value Conc. (mg/L)	25% NaOH Spiked Conc. (mg/L)
1. Chloride	0.200	0.100	0.155 ± 0.003	0.255	0.258 ± 0.004
2. Bromide	0.200	0.100	ND	0.100	0.099 ± 0.008
3. Chlorate	0.200	0.100	ND	0.100	0.099 ± 0.009
4. Nitrate	0.200	0.100	0.011 ± 0.006	0.111	0.199 ± 0.013
5. Phosphate	0.200	0.100	0.106 ± 0.025	0.206	0.183 ± 0.017
6. Sulfate	0.400	0.200	0.348 ± 0.004	0.548	0.553 ± 0.011
7. Oxalate	---	0.100	0.134 ± 0.028	0.234	0.238 ± 0.022

Based on 8 runs

ND = None Detected

4.7.4 IonPac AS12A Isocratic Analysis of 20% Ammonium Hydroxide

Sample:	100 μL of 20% NH_4OH neutralized in the SP10 AutoNeutralizer
Concentrator Column:	Low Pressure Trace Anion Concentrator (TAC-LP1, P/N 046026)
Column:	IonPac AG12A + IonPac AS12A
Eluent:	2.7 mM Na_2CO_3/0.3 mM $NaHCO_3$
Eluent Flow Rate:	1.5 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS 300 AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS 300
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	14–16 μS



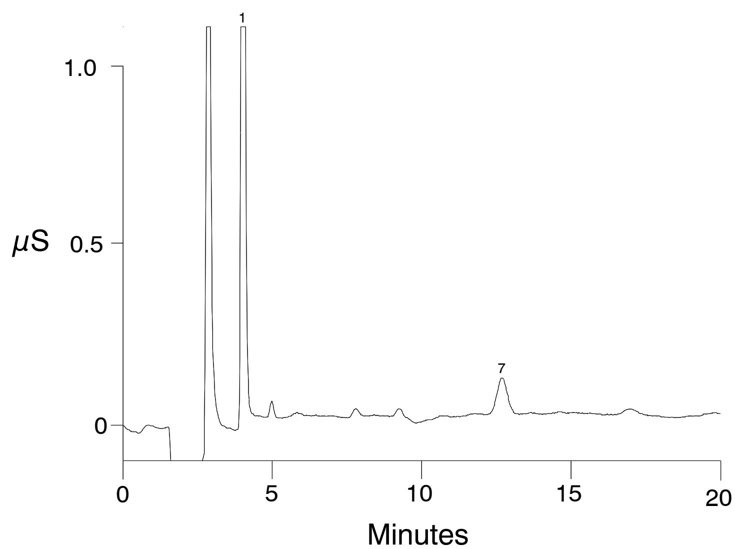
	Analyte	Conc. (mg/L)
1.	Chloride	0.0278
2.	Nitrite	ND
3.	Bromide	ND
4.	Chlorate	ND
5.	Nitrate	0.345
6.	Phosphate	ND
7.	Sulfate	0.080
8.	Oxalate	0.055

ND = None Detected

Figure 10
IonPac AS12A Analysis of 20% Ammonium Hydroxide

4.7.5 IonPac AS12A Isocratic Analysis of 25% Tetramethylammonium Hydroxide

Sample:	100 μL of 25% TMAOH neutralized in the SP10 AutoNeutralizer
Concentrator Column:	Low Pressure Trace Anion Concentrator (TAC-LP1, P/N 046026)
Column:	IonPac AG12A + IonPac AS12A
Eluent:	2.7 mM Na_2CO_3/0.3 mM $NaHCO_3$
Eluent Flow Rate:	1.5 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS 300 AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS 300
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	14–16 μS



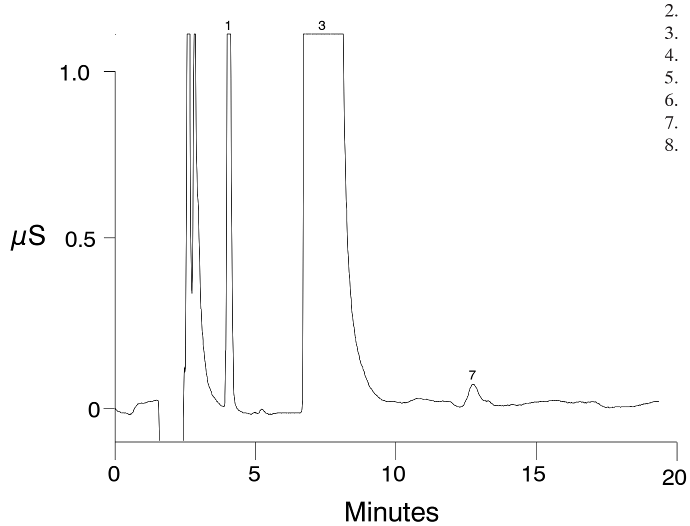
	Analyte	Conc. (mg/L)
1.	Chloride	1.917
2.	Nitrite	---
3.	Bromide	ND
4.	Chlorate	ND
5.	Nitrate	ND
6.	Phosphate	ND
7.	Sulfate	0.117
8.	Oxalate	ND

ND = None Detected

Figure 11
IonPac AS12A Analysis of 25% Tetramethylammonium Hydroxide

4.7.6 IonPac AS12A Isocratic Analysis of 25% Tetrabutylammonium Hydroxide

Sample:	100 μL of 25% TBAOH neutralized in the SP10 AutoNeutralizer
Concentrator Column:	Low Pressure Trace Anion Concentrator (TAC-LP1, P/N 046026)
Column:	IonPac AG12A + IonPac AS12A
Eluent:	2.7 mM Na_2CO_3/0.3 mM $NaHCO_3$
Eluent Flow Rate:	1.5 mL/min
SRS Suppressor:	Anion Self-Regenerating Suppressor, ASRS 300 AutoSuppression Recycle Mode
or MMS Suppressor:	Anion MicroMembrane Suppressor, AMMS 300
MMS Regenerant:	50 mN H_2SO_4
Expected Background Conductivity:	14–16 μS



	Analyte	Conc. (mg/L)
1.	Chloride	0.831
2.	Nitrite	ND
3.	Bromide	NA
4.	Chlorate	ND
5.	Nitrate	ND
6.	Phosphate	ND
7.	Sulfate	0.045
8.	Oxalate	ND

ND = None Detected
NA = Not Applicable (unable to quantify)

Figure 12
IonPac AS12A Analysis of 25% Tetrabutylammonium Hydroxide

4.8 Cation Example Applications

4.8.1 IonPac CS12A Blank and Standard Analysis

Sample:	100 µL of Standard or Blank neutralized in the CSRN 300 Neutralizer
Concentrator Column:	Low Pressure Trace Cation Concentrator (TCC-LP1, P/N 046027)
Guard Column:	CG12A Guard Column
Analytical Column:	CS12A Analytical Column
Eluent:	20 mM Methanesulfonic acid (EG50)
Eluent Flow Rate:	1.0 mL/min (4-mm)
SRS Suppressor:	Cation Self-Regenerating Suppressor, CSRS 300 4mm AutoSuppression Recycle Mode
or MMS Suppressor:	Cation MicroMembrane Suppressor CMMS 300 (4-mm)
MMS Regenerant:	100 mN TBAOH
Expected Background Conductivity:	20 mM MSA: <1 µS

Expected System Operating Back Pressure: Without Guard: 1,400 psi (9.65 MPa)
With Guard: 1,850 psi (12.75 MPa)

	Analyte	Conc.. (µg/L)
1.	Lithium	5.0
2.	Sodium	20.0
3.	Ammonium	25.0
4.	Dimethylamine	B
5.	Potassium	50.0
6.	Trimethylamine	B
7.	Magnesium	25.0
8.	Calcium	50.0

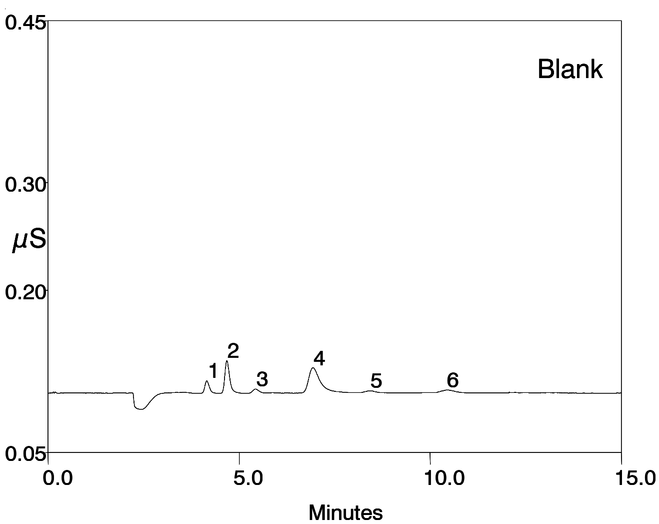


Figure 13A

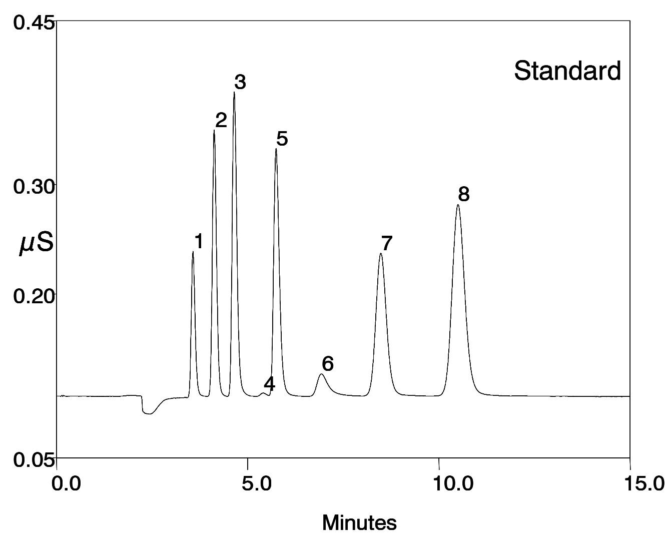


Figure 13B

Figure 13
IonPac CS12A Blank and Standard Analysis

4.8.2 IonPac CS12A Analysis of 24% Sulfuric Acid

Sample:	100 μL of 24% sulfuric acid neutralized in the CSRN 300 neutralizer
Concentrator Column:	Low Pressure Trace Cation Concentrator (TCC-LP1, P/N 046027)
Guard Column:	CG12A Guard Column
Analytical Column:	CS12A Analytical Column
Eluent:	20 mM Methanesulfonic acid (MSA)
Eluent Flow Rate:	1.0 mL/min (4-mm)
SRS Suppressor:	Cation Self-Regenerating Suppressor, CSRS 300 AutoSuppression Recycle Mode
or MMS Suppressor:	Cation MicroMembrane Suppressor CMMS 300, (2-mm) or CMMS 300I (4-mm)
MMS Regenerant:	100 mN TBAOH
Expected Background Conductivity:	20 mM MSA: 1 μS
Expected System Operating Back Pressure:	Without Guard: 1,400 psi (9.65 MPa) With Guard: 1,850 psi (12.75 MPa)

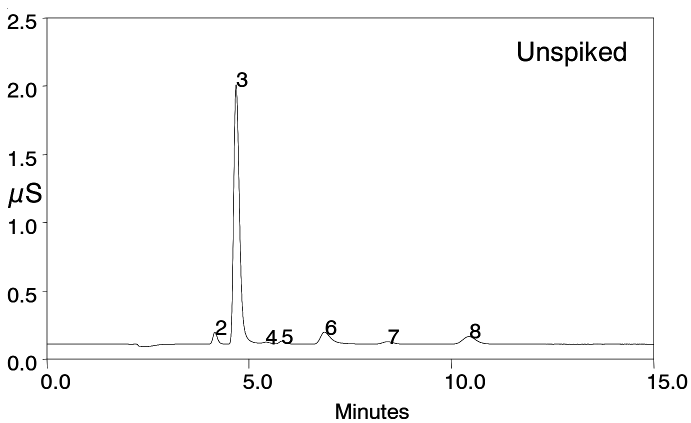


Figure 14A
IonPac CS12A Analysis of 24% H_2SO_4 Sample

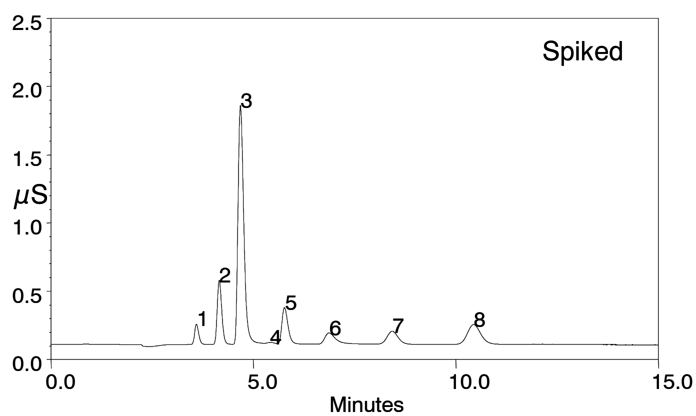


Figure 14B
IonPac CS12A Analysis of Spiked 24% H_2SO_4 Sample

Table 3
Recovery Data for the IonPac CS12A Analysis of 24% H_2SO_4

	Spiking Standard Conc. (μg/L)	24% H_2SO_4* Unspiked Value Conc. (μg/L)	Expected Spiked Value Conc. (μg/L)	24% H_2SO_4* Spiked Value Conc. (μg/L)
1. Lithium	2.0	ND	2.0	1.993 ± 0.008
2. Sodium	8.0	7.4 ± 0.4	15.4	14.3 ± 0.1
3. Ammonium	B	B	B	B
4. Dimethylamine	B	B	B	B
5. Potassium	20.0	3.3 ± 0.5	23.3	22.5 ± 0.2
6. Trimethylamine	B	B	B	B
7. Magnesium	10.0	2.0 ± 0.2	12.0	12.2 ± 0.2
8. Calcium	20.0	10.6 ± 0.1	30.6	33 ± 1

* Based on 8 runs
ND = None Detected, B = Blank

4.8.3 IonPac CS12A Analysis of 25% Acetic Acid

Sample:	100 μL of 25% acetic acid neutralized in the CSRN 300 Neutralizer
Concentrator Column:	Low Pressure Trace Cation Concentrator (TCC-LP1, P/N 046027)
Guard Column:	CG12A Guard Column
Analytical Column:	CS12A Analytical Column
Eluent:	20 mM Methanesulfonic acid (EG50)
Eluent Flow Rate:	1.0 mL/min (4-mm)
SRS Suppressor:	Cation Self-Regenerating Suppressor, CSRS 300 4mm AutoSuppression Recycle Mode
or MMS Suppressor:	Cation MicroMembrane Suppressor CMMS 300 (4-mm)
MMS Regenerant:	100 mN TBAOH
Expected Background Conductivity:	20 mM MSA: <1 μS

Expected System Operating Back Pressure: Without Guard: 1,400 psi (9.65 MPa)
With Guard: 1,850 psi (12.75 MPa)

	Analyte	Conc.. (μg/L)
1.	Sodium	86.7
2.	Ammonium	33.7
3.	Dimethylamine	B
4.	Potassium	21.6
5.	Trimethylamine	B
6.	Magnesium	2.5
7.	Calcium	133.0

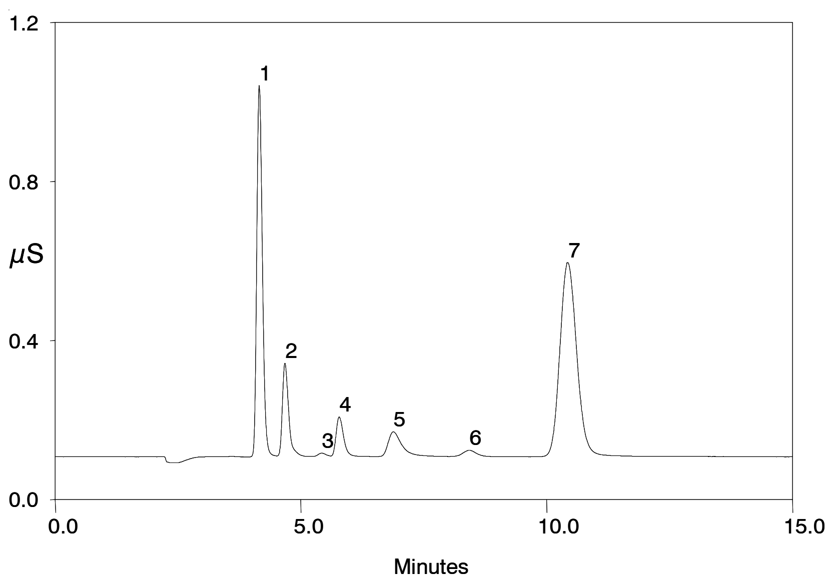


Figure 15
IonPac CS12A Analysis of 25% Acetic Acid

4.8.4 IonPac CS12A Analysis of 10% Hydrofluoric Acid

Sample:	100 µL of 10% hydroflouric acid neutralized in the CSRN 300 Neutralizer
Concentrator Column:	Low Pressure Trace Cation Concentrator (TCC-LP1, P/N 046027)
Guard Column:	CG12A Guard Column
Analytical Column:	CS12A Analytical Column
Eluent:	20 mM Methanesulfonic acid (EG50)
Eluent Flow Rate:	1.0 mL/min (4-mm)
SRS Suppressor:	Cation Self-Regenerating Suppressor, CSRS 300 4mm AutoSuppression Recycle Mode
or MMS Suppressor:	Cation MicroMembrane Suppressor CMMS 300 (4-mm)
MMS Regenerant:	100 mN TBAOH
Expected Background Conductivity:	20 mM MSA: <1 µS

Expected System Operating Back Pressure: Without Guard: 1,400 psi (9.65 MPa)
With Guard: 1,850 psi (12.75 MPa)

	Analyte	Conc. (µg/L)
1.	Lithium	0.4
2.	Sodium	66.7
3.	Ammonium	110.5
4.	Dimethylamine	B
5.	Potassium	8.4
6.	Trimethylamine	B
7.	Magnesium	15.3
8.	Calcium	59.6

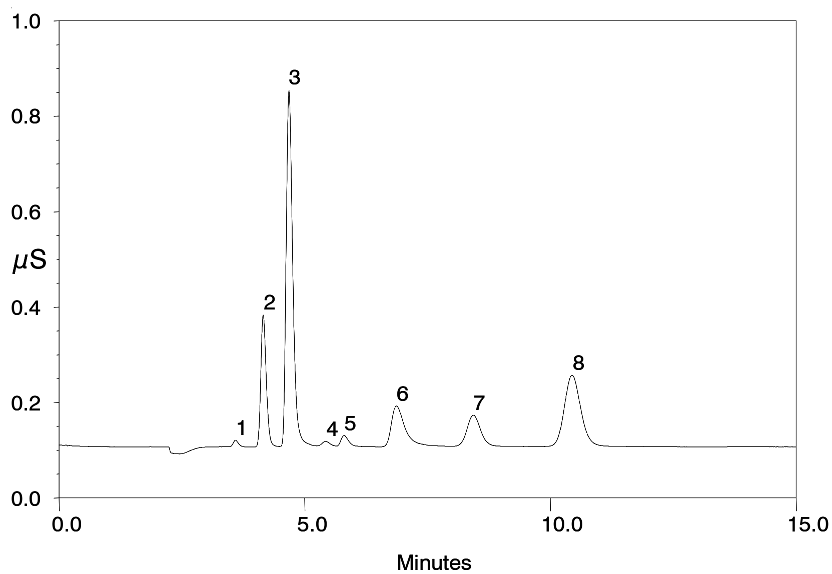


Figure 16
IonPac CS12A Analysis of 10% Hydrofluoric Acid

4.8.5 IonPac CS12A Analysis of 22% Phosphoric Acid

Sample:	100 μL of 22% phosphoric acid neutralized in the CSRN 300 Neutralizer
Concentrator Column:	Low Pressure Trace Cation Concentrator (TCC-LP1, P/N 046027)
Guard Column:	CG12A Guard Column
Analytical Column:	CS12A Analytical Column
Eluent:	20 mM Methanesulfonic acid (EG50)
Eluent Flow Rate:	1.0 mL/min (4-mm)
SRS Suppressor:	Cation Self-Regenerating Suppressor, CSRS 300 4mm AutoSuppression Recycle Mode
or MMS Suppressor:	Cation MicroMembrane Suppressor CMMS 300 (4-mm)
MMS Regenerant:	100 mN TBAOH
Expected Background Conductivity:	20 mM MSA: <1 μS

Expected System Operating Back Pressure: Without Guard: 1,400 psi (9.65 MPa)
With Guard: 1,850 psi (12.75 MPa)

	Analyte	Conc.. (μg/L)
1.	Sodium	509.1
2.	Ammonium	438.6
3.	Dimethylamine	B
4.	Potassium	25.8
5.	Trimethylamine	B
6.	Magnesium	22.4
7.	Calcium	144.1

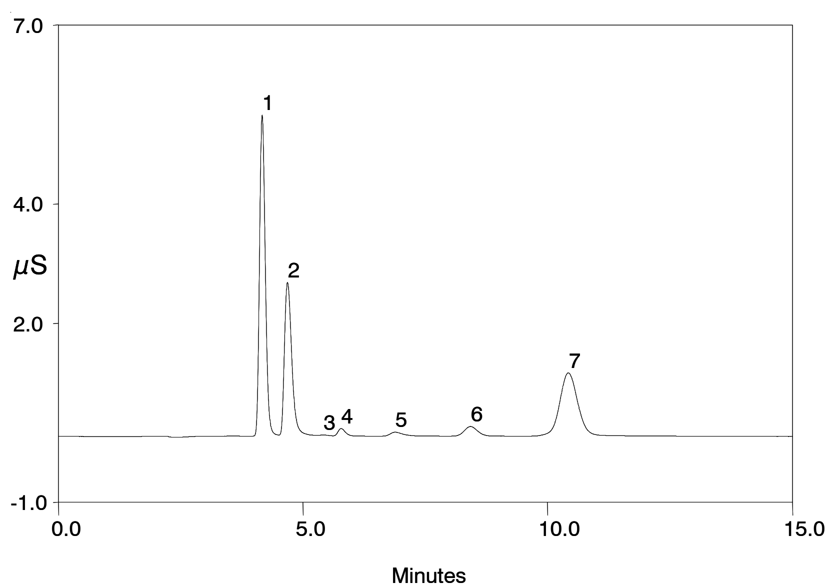


Figure 17
IonPac CS12A Analysis of 22% Phosphoric Acid

SECTION 5 - TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using the Self-Regenerating Neutralizer (SRN 300).

5.1 SRN 300 Liquid Leaks

A. If there is liquid coming out of the four ports of the Self-Regenerating Neutralizer (SRN 300), carefully tighten the fittings in the **ELUENT** and **REGEN** ports. If tightening the fittings does not stop the leak, replace the fittings on the tubing.

B. If there is leakage from the side seam of the SRN 300, check the system back pressure after the SRN 300. **Excessive back pressure after the SRN 300 will cause it to leak. The SRN 300 cannot operate at pressures greater than 100 psi.** The main source of back pressure for the SRN 300 in normal operation is the TAC-LP1or the TCC-LP1 Concentrator Column and the valves. It should not generate more than 70 psi at 0.5 mL/min flow rate. If excessive back pressure is traced to the faulty valve operation or crimped lines in the SP10 AutoNeutralizer, see Document No. 034980, "SP10 AutoNeutralizer Operator's Manual."

C. If the system back pressure after SRN 300 is greater than 100 psi, the leaks are caused by the excessive back pressure downstream from the SRN 300. Find and eliminate the source of the pressure. The SRN 300 will usually recover from momentary overpressure conditions if allowed to stand approximately 20 minutes with the membranes fully hydrated (see the caution note in Section 3.3, "Start up Procedure for the Self-Regenerating Neutralizer (SRN 300)"). If the SRN 300 continues to leak when operated within the proper back pressure range, it may have been damaged and must be replaced.

D. If the system back pressure after SRN 300 is less than 100 psi, check the back pressure of SRN 300. A blockage in the SRN may result in increased back pressure and potentially damage it. If the back pressure generated by the SRN 300 is greater than 25 psi at a flow rate of 0.5 mL/min, the SRN 300 may have a blockage.

E. To remove the blockage with back flush, follow the steps below.

1. Reverse the flow directions of the sample eluent and the external water through the SRN 300.

2. Connect a separate waste line to the sample eluent flowing from the **ELUENT IN** port of the SRN 300. During this flushing operation, do not connect the line between the IC injection valve and the SRN 300 to avoid contamination of injection valve.

3. If this inverted operation decreases the back pressure, reconnect the SRN 300 to the injection valve and use it in this position.

NOTE

Be sure to reverse BOTH the sample carrier solution AND the regenerant so that they remain flowing in opposite directions to one another.

F. If reversing the flow through the SRN does not decrease the pressure, clean the SRN 300 membranes (see Section 6, "Cleanup").

G If cleaning the SRN 300 membranes does not reduce the pressure, the neutralizer may be damaged and must be replaced.

Warning

Do not attempt to disassemble the SRN 300 and repair it yourself. Call the nearest Dionex Regional Office (see, "Dionex Worldwide Offices") for assistance.

5.2 Low Sample Response

A. If the SRN 300 is observed to be leaking, see Section 5.1.1, "SRN 300 Liquid Leaks."

B. Ensure that the **Sample Injection Valve** and the **Recycle Valve** are operating correctly. Refer to the valve manuals that accompany the SP10 AutoNeutralizer for troubleshooting assistance. Be sure to check the slider port faces for damage.

C. If sample neutralization requires excessive recycling, clean the SRN 300 membrane (see Section 6, "Cleanup").

D. If cleaning the SRN 300 membrane does not restore neutralization efficiency, the SRN 300 may need to be replaced.

E. If you cannot solve the problem on your own, contact the Dionex Regional Office nearest you (see, "Dionex Worldwide Offices").

5.3 High Background Conductivity

When the SRN 300 is neutralizing a sample and removing the counter ions, the conductivity observed on the front panel of the SP10 AutoNeutralizer typically is less then 5 μS. Higher conductivity indicates that the sample has not been completed neutralized. The sample may require multiple passes through the SRN 300 or the SRN 300 may not be operating properly.

A. Try multiple passes through the SRN 300.

B. Check for carrier flow out of the SRN 300 **ELUENT OUT** port**.**

1. If there is no flow out of the SRN 300 **ELUENT OUT** port, make sure that the carrier solution is entering the SRN 300 at the **ELUENT IN** port. If there is no flow at this point, trace the carrier solution flow path backward through the system to find and remove the blockage.

2. If there is flow into the SRN 300 but not out and there are no visible leaks from the side seam of the SRN 300, then a break in the membrane is probably allowing sample to leak into the regenerant chambers. If this is the case, then the SRN 300 must be replaced. **The SRN 300 is sealed during manufacture. Attempting to open it will destroy it.**

WARNING
Do not attempt to disassemble the Self-Regenerating Neutralizer.

3. If there is flow from the **ELUENT OUT** port, but no sample neutralization, the membrane may have been contaminated. Try to restore system performance by cleaning the membrane (see Section 6, "Cleanup" or Section 5.4.2 "Low Neutralization Capacity").

C. If the background conductivity remains high, and you cannot solve the problem on your own, contact the nearest Dionex Regional Office (see, "Dionex Worldwide Offices").

5.4 Low Neutralization Capacity

For the ASRN 300, this problem is caused when the ion exchange sites in the neutralizer are converted from the hydronium form to the sodium form. They must converted back to the hydronium form for efficient operation.

For CSRN 300, this problem is caused when the ion exchange sites in the CSRN 300 are converted from the hydroxide form to a salt form such as the sulfate form. They must converted back to the hydroxide form for efficient operation.

If the sample types that you are running suddenly not neutralize with multiple passes through the SRN 300, perform the following procedure.

A. Disconnect the sample line to the **ELUENT IN** port of the SRN 300.

B. Disconnect the sample line from the **ELUENT OUT** port of the SRN 300.

C. Install a plastic syringe with a Luer adaptor in the **ELUENT OUT** port.

D. Connect a separate line from **EUENT IN** port to a waste container.

E. Inject 5.0 mL of 0.5 N H_2SO_4 through the **ASRN 300**.

OR

Inject 5.0 mL of 0.5 M NaOH through the **CSRN 300**.

D. Reconnect the sample line to the **ELUENT IN** and **ELUENT OUT** port of the SRN 300.

E. Establish water flow through the regenerant chambers, begin pumping water through sample chamber and turn on the power.

F. If the correct neutralization is not observed following two injections of a standard test solution, contact the nearest Dionex Regional Office (see, "Dionex Worldwide Offices").

5.5 System Back Pressure Increases Over Time

Periodically test the back pressure after the SRN 300 generated by the Concentrator Column on the analytical system to ensure that it is not generating excessive back pressure. **Excessive back pressure after the SRN 300 will cause it to leak. The SRN 300 cannot operate at pressures greater than 100 psi.** If the increased back pressure does not exceed 100 psi, no maintenance is necessary.

If the concentrator column generates more than 100 psi at 0.5 mL/min, change its inlet bed support assembly and clean the column (see Document No. 034972, "Product Manual for the Low Pressure Concentrator (TAC-LP1) Column" or Document No. 034973, "Product Manual for the Low Pressure Concentrator (TCC-LP1) Column").

5.6 SP10 AutoNeutralizer Alarms

The SP10 AutoNeutralizer has four (4) alarms that are associated with Self-Regenerating Neutralizer. When an alarm condition occurs, the corresponding LED indicator on the control panel lights and a buzzer sounds. For more information on problems that originate with the SP10 AutoNeutralizer, refer to the Troubleshooting Guide in Document No. 034980, "SP10 AutoNeutralizer Operator's Manual". If you cannot solve the problem on your own, contact the Dionex Regional Office nearest you (see, "Dionex Worldwide Offices").

5.6.1 SP10 AutoNeutralizer Leak Alarm

The Leak Alarm signifies that there is liquid in the drip tray in the SP10 AutoNeutralizer. If a leak warning alarm in the SP10 AutoNeutralizer occurs, check for leaks throughout the SP10 AutoNeutralizer (see Section 4, "Troubleshooting," in the SP10 AutoNeutralizer Operator's Manual, Document No. 034980).

5.6.2 SP10 AutoNeutralizer Volt Alarm

The SRN 300 is designed to operate at a maximum of 8.5 ± 0.5 V. The Volt Alarm will sound if the SRN 300 does not have liquid in it or if the SRN 300 cable is disconnected.

5.6.3 SP10 AutoNeutralizer Temp Alarm

The SRN 300 requires an internal temperature of 40°C or less. Excessive temperature will damage the SRN 300. The temperature alarm will sound if the internal temperature of the SRN 300 exceeds 40 ± 2 °C. This can be caused by an insufficient flow of carrier solution through the SRN 300. **The required minimum flow rate of regenerant solution is 3– 5 mL/min.** If the regenerant flow rate is low, adjust the air pressure on the pressurized bottle containing the carrier solution to deliver at least 3–5 mL/min.

5.6.4 SP10 AutoNeturalizer Cond. Alarm

If the conductivity of the liquid in the conductivity cell is greater than the value selected by the **Cond Alarm Set Point DIP switches** in the SP10 AutoNeutralizer (see Document No. 034980, "SP10 AutoNeutralizer Operator's Manual"), the Cond Alarm will sound. The default value is 50 μS.

SECTION 6 – CLEANUP

This section describes routine cleanup procedures for the Self-Regenerating Neutralizers (SRN 300) in the case of contamination. Consult the Troubleshooting Guide (see Section 5, "Troubleshooting Guide") to first determine that the system is operating properly. If the SRN 300 is determined to be the source of higher than normal back pressure, higher than anticipated conductivity, decreased neutralization capacity or decreased sensitivity, cleaning the membrane may restore the performance of the system. Use the following procedures to clean the membrane.

6.1 Metal Contaminants or Precipitates

A. Disconnect the SRN 300 power cord.

B. Disconnect the liquid line between the SRN 300 **ELUENT OUT** port and the Recycle Valve at the Recycle Valve end and reconnect it to the **REGEN IN** port. Ensure that the **REGEN OUT** port is connected to the waste line.

C. Connect a temporary line from the priming block or the low-pressure tee on the isocratic or gradient pump to a container with a solution of 0.1 M KCl in 1.0 M HCl or a solution of 0.2 M oxalic acid. Pressurize the container to 5–10 psi. Pump this solution through the SRN 300 at 1–2 mL/min for 30 minutes.

D. Flush the SRN 300 with deionized water for 10 minutes.

E. Reconnect the power cord to the SRN 300.

F. Begin pumping carrier solution through the system at the flow rate required for your neutralization and equilibrate the system.

6.2 Organic Contaminants

A. Disconnect the SRN 300 power cord.

B. Disconnect the liquid line between the SRN 300 **ELUENT OUT** port and the Recycle Valve at the Recycle Valve end and reconnect it to the **REGEN IN** port. Ensure that the **REGEN OUT** port is connected to the SP10 pump.

C. Connect a temporary line from the priming block on the SP10 pump to a container with a solution of freshly prepared 10% 1.0 M HCl/90% acetonitrile or methanol. HCl/acetonitrile solutions are not stable during long term storage so this cleanup solution must be made immediately before each cleanup. Pressurize the bottle to 5–10 psi pressure. Pump this solution through the ASRN 300 at 1–2 mL/min for 30 minutes.

D. Flush the SRN 300 with deionized water for 10 minutes.

E. Reconnect the power cord to the SRN 300.

F. Begin pumping carrier solution through the system at the flow rate required for your neutralization and equilibrate the system.

