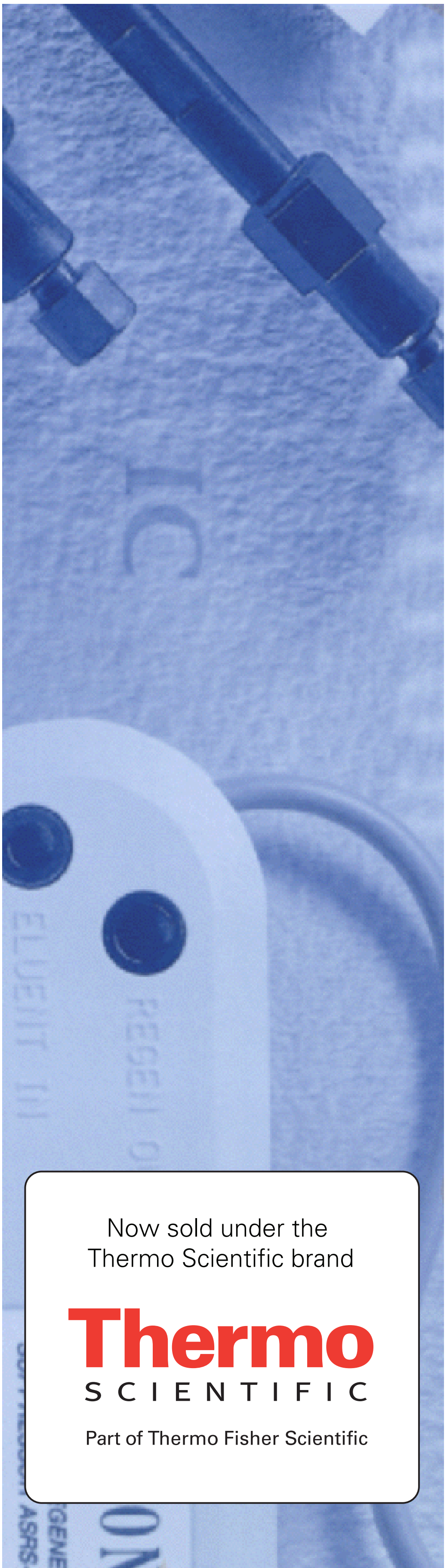
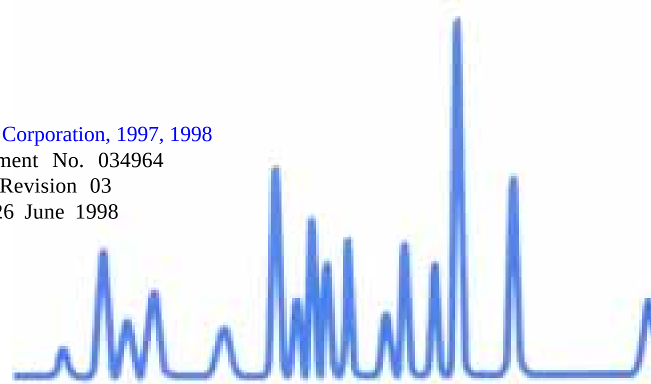


INSTALLATION INSTRUCTIONS
and
TROUBLESHOOTING GUIDE

for the

CATION SELF-REGENERATING NEUTRALIZER-I 4mm
(CSRN-I (4mm), P/N 046025)



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SECTION 1 - INTRODUCTION TO SELF-REGENERATING AUTONEUTRALIZATION FOR TRACE CATION ANALYSIS IN CONCENTRATED REAGENTS

The DIONEX Cation Self-Regenerating Neutralizer (CSRN-I) is a high capacity membrane-based electrolytic neutralization device. When used in the SP10 AutoNeutralizer, its design permits on-line neutralization of concentrated acids for the determination of trace levels of cationic species. The CSRN-I uses the electrolysis of deionized water to produce hydroxide ions which serve to neutralize the acidic portion of the sample.

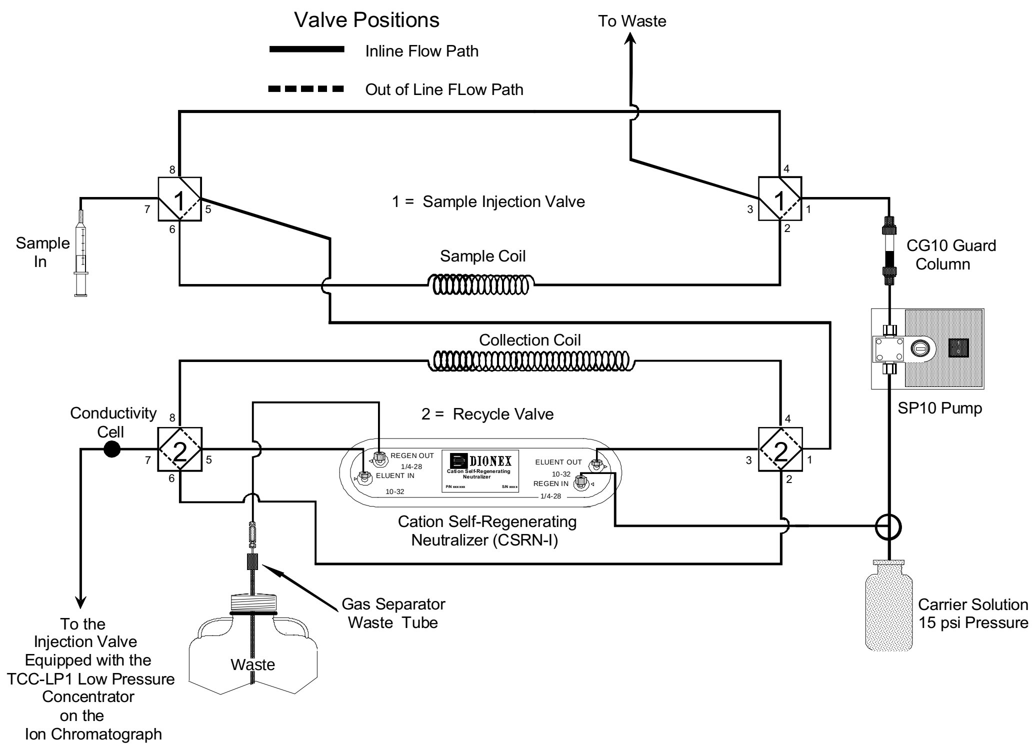

Figure 1

The SP10 AutoNeutralizer with the Cation Self-Regenerating Neutralizer

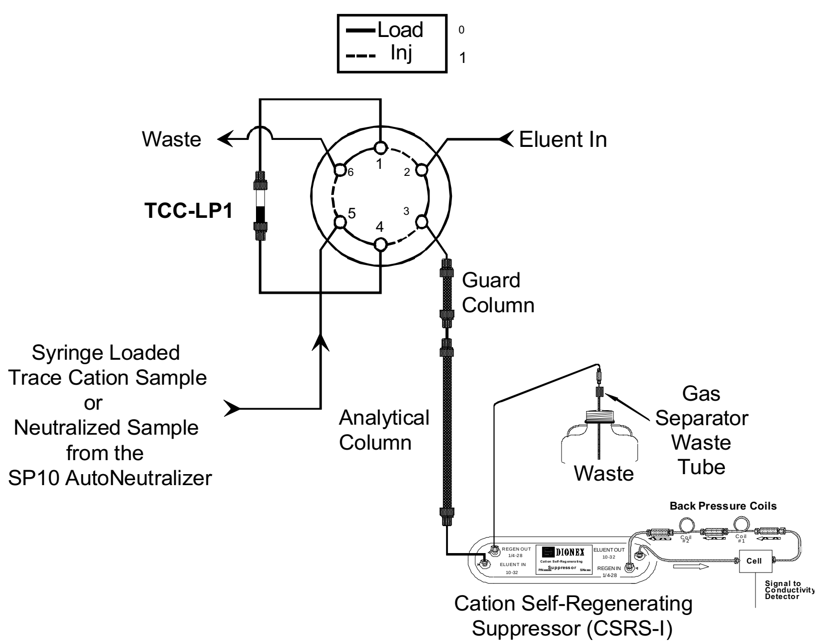

Figure 2

Ion Chromatograph Equipped with the TCC-LP1 Low Pressure Concentrator

Simultaneously, the counter ions in the bulk matrix are removed at the chamber containing the other electrode, resulting in a neutralized solution containing only the trace level cationic species of interest.

NOTE

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

Initially, a concentrated acid sample is loaded into the sample coil of the SP10 AutoNeutralizer with a syringe (see Figure 1, "The SP10 AutoNeutralizer with the Cation Self-Regenerating Neutralizer"). When **Valve #1, "The Sample Injection Valve"**, is thrown, the pump pushes the concentrated acid sample through the CSRN-I where neutralization begins.

As the sample leaves the CSRN-I it passes through the conductivity cell in the SP10 AutoNeutralizer which determines if neutralization is complete. Common acids that have been diluted 25% or more that pass through the CSRN-I at low flow rates $\leq$ 0.5 mL/min can be completely neutralized after one pass through the CSRN-I. After complete neutralization as indicated by the SP10 AutoNeutralizer conductivity cell, the sample passes out of the SP10 AutoNeutralizer and into the injection valve of any Ion Chromatograph equipped with suppressed conductivity detection and a **Low Pressure Trace Cation TCC-LP1 Concentrator Column** (P/N 046027).

For more concentrated acids or faster flow rates, multiple passes through the CSRN-I will be required to complete neutralization. These samples are pumped through the CSRN-I and when the Collection Coil is filled, **Valve #2, "The Recycle Valve"**, is thrown, This routes the sample back through the CSRN-I for additional neutralization. The sample can be recycled through the CSRN-I as many times as necessary to achieve complete neutralization. When neutralization is complete, as indicated by the SP10 AutoNeutralizer conductivity meter, Valve #2, "The Recycle Valve", is thrown back to the one-pass position and the neutralized sample passes out of the SP10 AutoNeutralizer and into the injection valve of any Ion Chromatograph equipped with suppressed conductivity detection and a Low Pressure Trace Cation TCC-LP1 Concentrator.

After complete neutralization by the CSRN-I, the sample stream contains trace level cations in deionized water. The SP10 AutoNeutralizer pump pushes the neutralized sample into the injection valve of any Ion Chromatograph equipped with suppressed conductivity detection and a Low Pressure Trace Cation Concentrator (TCC-LP1). Trace cations in the sample are concentrated on the TCC-LP1 and then analyzed. For details on the concentration process using the TCC-LP1, see the Installation Instructions and Troubleshooting Guide for the Low Pressure Trace Cation TCC-LP1 Concentrator, (Document No. 034973).

1.2 THE SELF-REGENERATING NEUTRALIZER (CSRN-I)

The Cation Self-Regenerating Neutralizer (CSRN-I) provides high capacity neutralization. The ability of the CSRN-I to completely neutralize strongly acidic samples significantly expands the capabilities of cation exchange Ion Chromatography in the area of trace cation analysis of these matrices.

The CSRN-I includes two regenerant compartments and one sample compartment separated by ion exchange membranes. A regenerant flow channel and a sample flow channel are defined on opposite sides of the membrane. The sample flow is in a direction that is countercurrent to the regenerant flow.

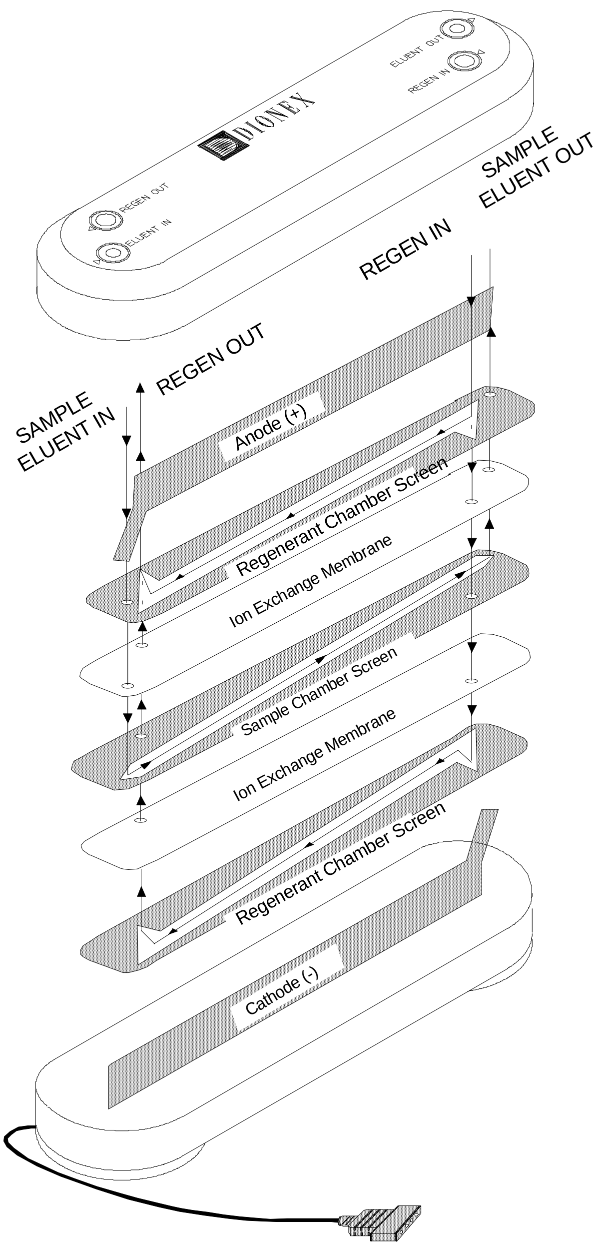

Figure 3

Electrode, Membrane and Screen Configuration in the Cation Self-Regenerating Neutralizer (CSRN-I)

Electrodes are placed along the length of the regenerant channels. When an electrical potential is applied across the electrodes, water from the regenerant channels is electrolyzed, supplying regenerant hydroxide ions (OH^-) for the sample neutralization reaction. The membrane passes these hydroxide ions into the sample neutralization chamber resulting in the conversion of the electrolyte of the sample to a weakly ionized form. Sample anions are simultaneously passed 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.

1.3 NEUTRALIZATION THEORY

The Cation Self-Regenerating Neutralizer (CSRN-I) requires a constant water feed provided by a constant source of deionized water from a pressurized bottle or a pump that delivers at least 3 to 5 mL/min through the regenerant chambers. Water can be delivered to the CSRN-I regenerant chambers either from a pressurized bottle or a pump to achieve sample neutralization.

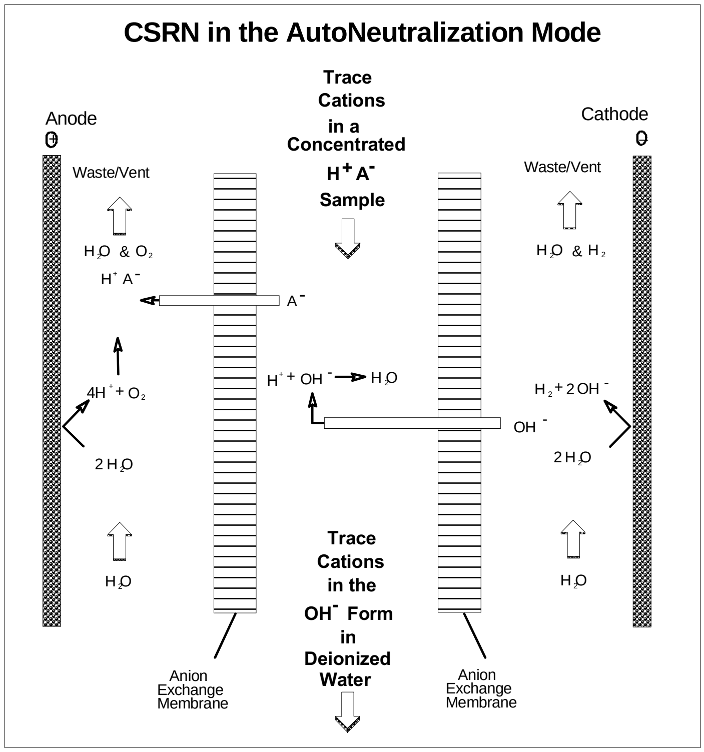

Figure 4

AutoNeutralization of Concentrated Acid with the Cation Self-Regenerating Neutralizer (CSRN-I)

The water regenerant undergoes electrolysis to form hydrogen gas and hydroxide ions in the cathode chamber while oxygen gas and hydronium ions are formed in the anode chamber. Anion exchange membranes allow hydroxide ions to move from the cathode chamber into the sample chamber to neutralize sample hydronium ions. Anions in the sample, such as sulfate, attracted by the electrical potential applied to the anode, move across the membrane into the anode chamber to maintain electronic neutrality with the hydronium ions at the electrode.

1.4 TRACE CATION CONCENTRATION

The output of the SP10 AutoNeutralizer can be fed into any DIONEX Ion Chromatograph equipped with suppressed conductivity detection and operated in the concentration mode. It is important that the Low Pressure Trace Cation Concentrator (TCC-LP1) Column (P/N 046027) be used for the concentrator to protect the CSRN-I in the SP10 AutoNeutralizer from damage. See Section 4, "Example Applications", for examples of these analyses. For detailed information on the theory and operation of the Low Pressure Trace Cation Concentrator (TCC-LP1) Column, consult the Installation Instructions and Troubleshooting Guide for the Low Pressure Trace Cation Concentrator TCC-LP1 Column, Document No. 034793.

CAUTION

Use only the Low Pressure Trace Cation Concentrator (TCC-LP1) Column (P/N 046027) with the CSRN-I. Use of other concentrator columns may damage the CSRN-I !

The TCC-LP1 is used only with carboxylated columns such as the IonPac CS12 and the IonPac CS14 Guard and Analytical Columns. The Ionpac CS12 is recommended for general purpose cation separations. If potassium is not an analyte of interest, the IonPac CS14 be used.

1.5 SELECTION OF GUARD AND ANALYTICAL COLUMNS FOR TRACE CATION SEPARATION

Depending on your specific application, any cation exchange guard and analytical column set can be used for trace cation analysis using the SP10 AutoNeutralizer.

1.6 SELECTION OF THE SUPPRESSOR FOR CATION DETERMINATION

Use the Cation Self-Regenerating Suppressor-I (4mm, P/N 043190). For detailed information on the theory and operation of the CSRS-I, see the Installation Instructions and Troubleshooting Guide for the Cation Self-Regenerating Suppressor (CSRS-I), Document No. 034651.

SECTION 2 - INSTALLATION

2.1 INSTALLATION OF THE SP10 AUTONEUTRALIZER

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

2.2 INSTALLATION OF THE CATION SELF-REGENERATING NEUTRALIZER (CSRN-I)

The Cation Self-Regenerating Neutralizer (CSRN-I) is designed to be installed and used in the SP10 AutoNeutralizer. The neutralized sample from the SP10 AutoNeutralizer can be analyzed for trace level cations by any DIONEX Ion Chromatograph (IC) equipped with a Low Pressure Trace Cation Concentrator Column (TCC-LP1), an cation exchange column set, an Cation Self-Regenerating Suppressor (CSRS-I) and suppressed conductivity detection.

The Cation Self-Regenerating Neutralizer (CSRN-I) uses water as the regenerant to achieve eluent neutralization. A carrier solution made of deionized water having a specific resistance of 10 megohm-cm or greater, is supplied to the regenerant chambers to generate hydroxide ions for neutralization. Neutralization capacity increases as the flow rate of water through the regenerant chambers increases.

The regenerant water can be supplied by either a pressurized bottle system or a pump. For most applications, the source is the sample carrier solution. The optimal regenerant flow rate (before the current is applied) is 3.0 - 5.0 mL/min. The flow rate of the regenerant (before the current is applied) can be as high as 10 - 15 mL/min. Fast flow is required to obtain maximum sweep out of the high concentrations of acid anions (e.g., SO_4^{2-}, PO_4^{3-} and F^-) that are transported through the membranes of the CSRN-I and to control temperature increases due to the heat of neutralization.

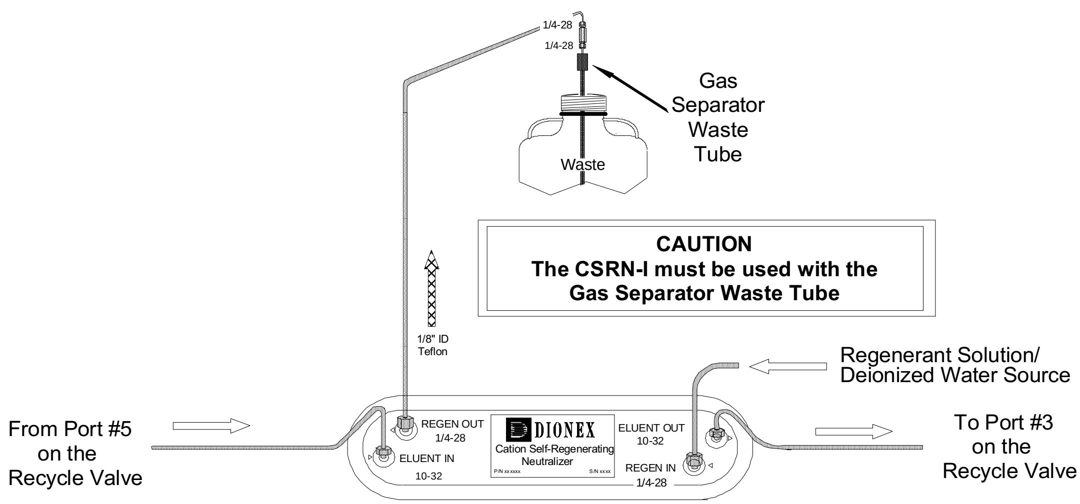

Figure 5

The Cation Self-Regenerating Neutralizer (CSRN-I) Plumbing Diagram

2.2.1 SAMPLE LIQUID LINE CONNECTIONS FOR THE CATION SELF-REGENERATING NEUTRALIZER (CSRN-I)

A. Install the Cation Self-Regenerating Neutralizer (CSRN-I) in the SP10 AutoNeutralization Module.
B. Connect the eluent line from **Port #5 on the Recycle Valve** to the **ELUENT IN Port** of the CSRN-I (see Figure 9, "The Cation Self-Regenerating Neutralizer (CSRN-I) Plumbing Diagram").

C. Connect the **ELUENT OUT Port** of the CSRN-I to **Port #3 on the Recycle Valve** (see Figure 5, "The Cation Self-Regenerating Neutralizer (CSRN-I) Plumbing Diagram").

CAUTION

BACK PRESSURES OVER 125 PSI AFTER THE CSRN-I CAN CAUSE IRREVERSIBLE DAMAGE!

2.2.2 REGENERANT LIQUID LINE CONNECTIONS FOR THE CATION SELF-REGENERATING NEUTRALIZER (CSRN-I)

The **Self-Regenerating Neutralizer (SRN) Ship Kit** (P/N 047950) contains all of the components needed to install and operate the CSRN-I with a pressurized water reservoir. See Section 2.4, "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 6, "Configuration of the Pressurized Water Reservoir and the Gas Separator Waste Tube with the Cation Self-Regenerating Neutralizer").

B. Make the following liquid line connections:

1. See Figure 6, "Configuration of the Pressurized Water Reservoir and the Gas Separator Waste Tube with the Cation 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 Installation Kit to the water reservoir. Connect the other end of this tubing to the **REGEN IN Port** of the Cation Self-Regenerating Neutralizer.

3. Using a coupler (P/N 039056) and a 1/8" OD piece of tubing (P/N 035728) from the Installation Kit, connect one end of this line to the **REGEN OUT Port** of the CSRN-I 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.

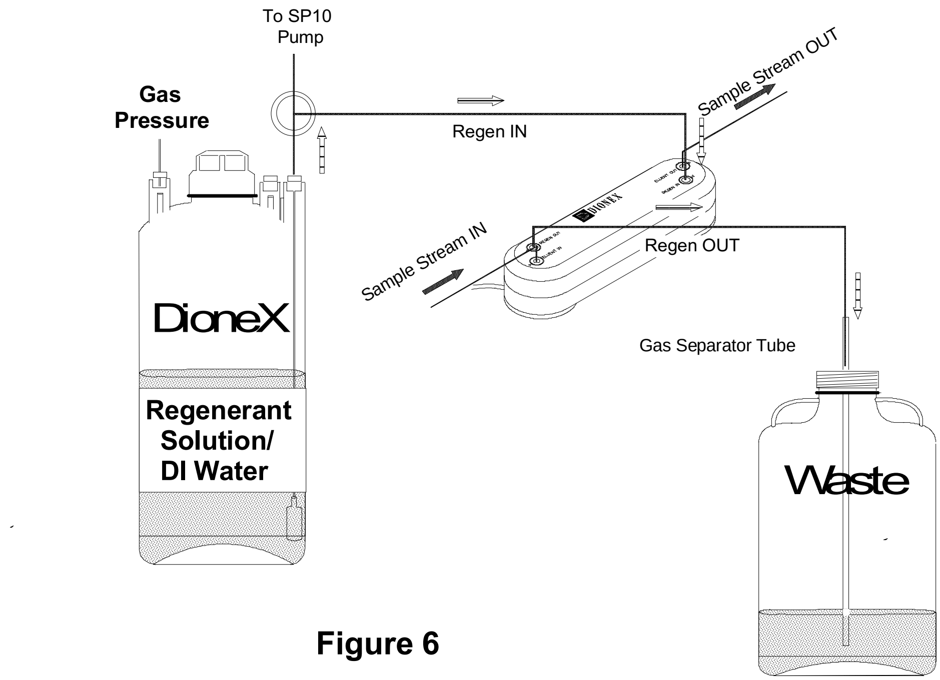

Figure 6

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

2.3 THE GAS SEPARATOR WASTE TUBE

The Gas Separator Waste Tube (P/N 045460) is an integral part of the Cation Self-Regenerating Neutralizer (CSRN-I) system. Its function is to ensure the separation of any hydrogen gas generated in the CSRN-I during the electrolytic generation of hydronium ions (H_3O^+). 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 CSRN-I 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 6, "Configuration of the Pressurized Water Pump and the Gas Separator Waste Tube with the Cation 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 6, "Configuration of the Pressurized Water Reservoir and the Gas Separator Waste Tube with the Cation 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 6, "Configuration of the Pressurized Water Reservoir and the Gas Separator Waste Tube with the Cation Self-Regenerating Neutralizer").

2.4 THE CG10 4MM GUARD COLUMN USED AS THE SP10 PUMP TRAP COLUMN

2.4.1 INSTALLATION OF THE CG10 4MM GUARD COLUMN

The CG10 4mm Guard Column (P/N 043016) is used to trap cations that may be in the carrier solution which could result in high blanks (see Figure "The SP10 AutoNeutralizer with the Cation Self-Regenerating Neutralizer"). The CG10 should be regenerated prior to installation in the SP10 AutoNeutralizer (see Section 2.4.2, "Regeneration of the CG10 4mm Guard Column").

2.4.2 REGENERATION OF THE CG10 4MM GUARD COLUMN

Using a pump other than the SP10 pump, the CG10 4mm 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 1 mL/min.

2.5 IMPORTANT SYSTEM ASSEMBLIES

046025 **Cation Self-Regenerating Neutralizer (CSRN-I)**

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

043016 **CG10 4mm Guard Column**

CAUTION

THE CSRN-I MUST BE OPERATED WITH THE GAS SEPARATOR WASTE TUBE (P/N 045460)

SECTION 3 - OPERATION

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 CSRN-I

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 CSRN-I and system components from contamination. DIONEX cannot guarantee proper CSRN-I 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 CSRN-I, 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. Currently 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 17.8 megohm-cm or greater. The water used for the **AutoNeutralization carrier solution** should have a specific resistance of 10 megohm-cm or greater. 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 CATION SELF-REGENERATING NEUTRALIZER (CSRN-I)

CAUTION

The membranes in the Cation Self-Regenerating Neutralizer (CSRN-I) must be completely hydrated to maintain liquid seals. This requirement is achieved by maintaining the regenerant cavities 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 turning on the power, install your new CSRN-I and pump water through the CSRN-I regenerant chambers until you see no more bubbles. Let the CSRN-I sit for at least 20 minutes to ensure that the membranes are fully hydrated before pumping eluent through the eluent chamber of the Neutralizer.

It is important to condition and remove trace cation contamination from the CSRN-I prior to CSRN-I installation. To perform the start up procedure, follow steps below.

A. Connect the **ELUENT OUT Port** to the **REGEN IN Port with a liquid line having one 10-32 fitting and one 1/4-28 fitting (see, "DIONEX Liquid Line Fittings")**. Make sure that the **REGEN OUT Port** is connected to the liquid line leading to the waste container.

B. Prepare 250 mL of 1.0 M H_2SO_4 solution by dilution of analytical grade H_2SO_4 in Type I Reagent Grade Water.

C. Pump approximately 100 mL of 1.0 M H_2SO_4 through the CSRN-I at 1.0 mL/min, followed by 300 mL of Type I Reagent Grade Water.

D. If the background conductivity is below 2 µS, then the CSRN-I is ready for operation. For additional information, see the SP10 AutoNeutralizer Operator's Manual, Document No. 034980.

SECTION 4 - EXAMPLE APPLICATIONS

The following examples are presented to highlight the analysis of trace cations found in the concentrated matrices that have been treated using the SP10 AutoNeutralizer.

The analysis may be done on any one of a number of DIONEX cation exchange column sets (see Section 1.3, "Selection of Guard and Analytical Columns"). Please refer to the Installation Instructions and Troubleshooting Guide for the particular cation exchange column set installed in your system for detailed operation and troubleshooting information.

Because of its low operating pressure, the Low Pressure Trace Cation Concentrator TCC-LP1 Column must be used to concentrate the trace cations in the sample neutralized by the SP10 AutoNeutralizer. The use of other concentrators may result in sufficient back pressure to damage the CSRN-I in the SP10 AutoNeutralizer.

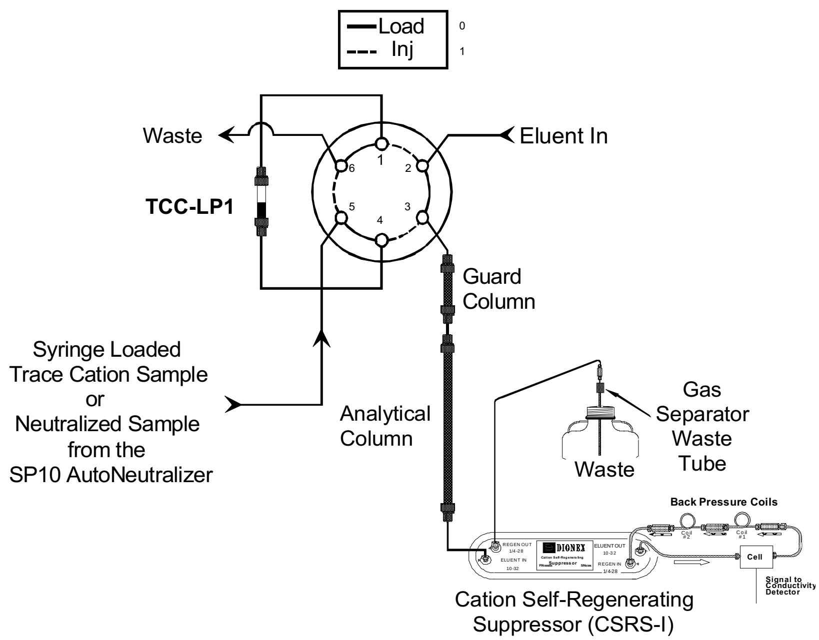

Figure 7

Ion Chromatography with Suppressed Conductivity Detection in the Concentrator Mode of Operation

4.1 SYSTEM BLANK

Trace cation contamination in the deionized water used for the carrier solution, the CSRN-I 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 SP10 Pump and the Sample Injection Valve in the SP10 AutoNeutralizer. The major source of amine contamination is usually from the CSRN-I. Since this device uses high capacity aminated ion exchange screens and membranes, amines are released from the screen and membrane surfaces, especially in a newly installed CSRN-I. To reduce the amine blank, follow the CSRN-I start-up procedure in Section 3.3, "Start-up Procedure for the Cation Self-Regenerating Neutralizer (CSRN-I)". The amine blank is normally reduced to a constant level after 24 hours of operation. A typical blank is shown in Figure 8A, "IonPac CS12 Blank and Standard Analysis". The use of high purity chemicals helps to reduce the blank concentrations.

4.2 CSRN CONDITIONING AND SYSTEM CALIBRATION

4.2.1 CSRN CONDITIONING

Since the CSRN is a high capacity anion exchange device which supplies the high concentration of hydroxide for acid neutralization, the hydrolyzable ions such as Mg^{2+} and Ca^{2+} in hydroxide forms may be precipitated in the CSRN. 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 is "conditioned" by running a complete pretreatment cycle of acid (preferably 1:4 sulfuric acid) prior to standard run. Follow the CSRN acid treatment procedure below prior to each standard run.

A. Confirm that the system conductivity is below 2 µS as indicated by the front panel conductivity meter of the SP10. Also confirm that the system is in "Local" mode and in "Load" position.

B. Inject 1:4 sulfuric acid into the sample loop and press "Inject".

C. In approximately 3.5-4.0 minutes (with the collection coil installed or 0.3 minutes without the collection coil installed), the conductivity signal will increase. Wait until the conductivity output decreases to the original value (less than 2 µS) and then press the control button to "Remote".

D. 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 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 acid pretreatment can be made anytime after the beginning of the analytical separation.

4.2.2 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, 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.

4.2.3 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 CS12 eluent is required to selectively remove transition metals from the eluent via the suppression system. An example of this can be found in Section 4.9, "IonPac CS12 Analysis of 22% Phosphoric Acid".

4.3 DETECTION LIMITS OF CSRN SAMPLE PRETREATMENT AND SUBSEQUENT ION CHROMATOGRAPHY

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

* Estimated values in H_2SO_4 matrix

4.4 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

4.5 IONPAC CS12 BLANK AND STANDARD ANALYSIS

Sample:	100 µL of Standard or Blank neutralized in the SP10 AutoNeutralizer
Concentrator Column:	Low Pressure Trace Cation Concentrator (TCC-LP1, P/N 046027)
Guard Column:	CG12 Guard Column
Analytical Column:	CS12 Analytical Column
Eluent:	19 mM Methanesulfonic acid (MSA)
Eluent Flow Rate:	1.0 mL/min (4mm)
Suppressor*:	Cation Self-Regenerating Suppressor AutoSuppression Recycle Mode
Expected Background Conductivity:	19 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)

* An Cation MicroMembrane Suppressor CMMS (2mm) or CMMS-I (4mm) may be used in place of the CSRS-I. The Regenerant is 100 mN TBAOH and the Regenerant Flow Rate is 5 mL/min.

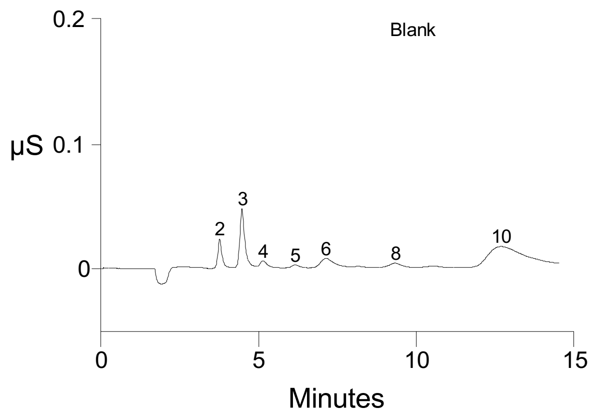

Figure 8A

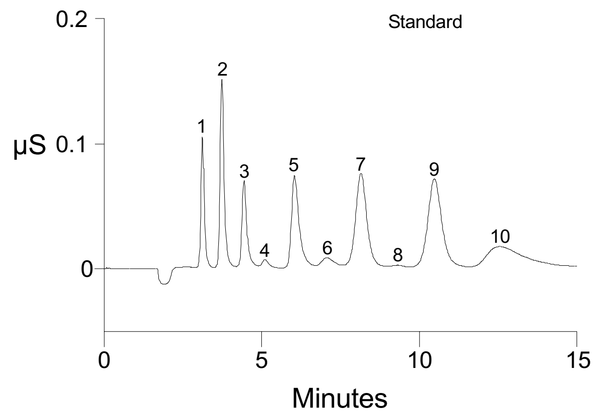

Figure 8B

IonPac CS12 Blank and Standard Analysis

Table 1

Standard/Blank Data for the IonPac CS12 Analysis

	Standard Figure 8B Conc. (µg/L)
1. Lithium	5.000
2. Sodium	20.000
3. Ammonium	B
4. Methylamine	B
5. Potassium	50.000
6. Dimethylamine	B
7. Magnesium	25.000
8. Unknown	B
9. Calcium	50.000
10. Trimethylamine	B

ND = None Detected
B = Blank

4.6 IONPAC CS12 ANALYSIS OF 24% SULFURIC ACID

Sample:	100 µL neutralized in the SP10 AutoNeutralizer
Concentrator Column:	Low Pressure Trace Cation Concentrator (TCC-LP1, P/N 046027)
Guard Column:	CG12 Guard Column
Analytical Column:	CS12 Analytical Column
Eluent:	19 mM Methanesulfonic acid (MSA)
Eluent Flow Rate:	1.0 mL/min (4mm)
Suppressor*:	Cation Self-Regenerating Suppressor AutoSuppression Recycle Mode
Expected Background Conductivity:	19 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)

* An Cation MicroMembrane Suppressor CMMS (2mm) or CMMS-I (4mm) may be used in place of the CSRS-I. The Regenerant is 100 mN TBAOH and the Regenerant Flow Rate is 5 mL/min.

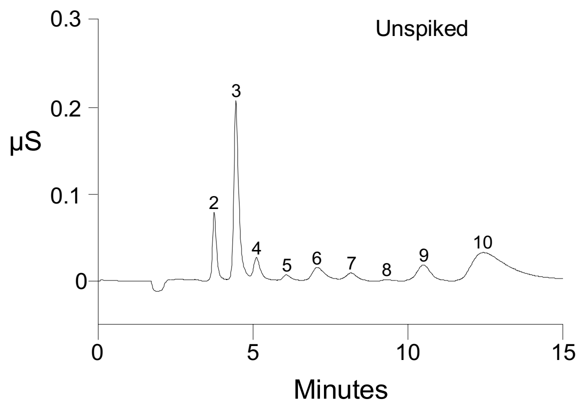

Figure 9A

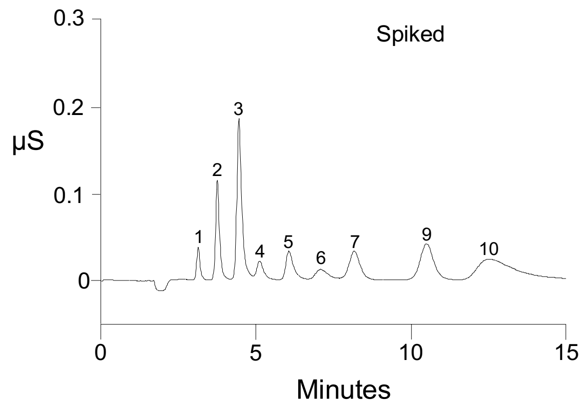

Figure 9B

IonPac CS12 Analysis of 24% H_2SO_4 Sample and Spiked 24% H_2SO_4 Sample

Table 2

Recovery Data for the IonPac CS12 Analysis of 25% 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. Methylamine	B	B	B	B
5. Potassium	20.0	3.3 ± 0.5	23.3	22.5 ± 0.2
6. Dimethylamine	B	B	B	B
7. Magnesium	10.0	2.0 ± 0.2	12.0	12.2 ± 0.2
8. Unknown	B	B	B	B
9. Calcium	20.0	10.6 ± 0.1	30.6	33 ± 1
10. Trimethylamine	B	B	B	B

*Based on 8 runs

ND = None Detected
B = Blank

4.7 IONPAC CS12 ANALYSIS OF 25% ACETIC ACID

Sample:	100 µL neutralized in the SP10 AutoNeutralizer
Concentrator Column:	Low Pressure Trace Cation Concentrator (TCC-LP1, P/N 046027)
Guard Column:	CG12 Guard Column
Analytical Column:	CS12 Analytical Column
Eluent:	19 mM Methanesulfonic acid (MSA)
Eluent Flow Rate:	1.0 mL/min (4mm)
Suppressor*:	Cation Self-Regenerating Suppressor AutoSuppression Recycle Mode
Expected Background Conductivity:	19 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)

* An Cation MicroMembrane Suppressor CMMS (2mm) or CMMS-I (4mm) may be used in place of the CSRS-I. The Regenerant is 100 mN TBAOH and the Regenerant Flow Rate is 5 mL/min.

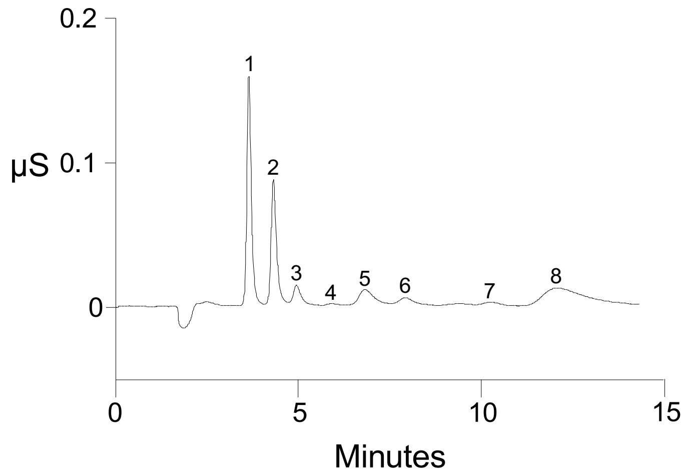

Figure 10

IonPac CS12 Analysis of 25% Acetic Acid

Table 3

Quantification Data for the IonPac CS12 Analysis of 25% Acetic Acid

	25% Acetic Acid Conc. (µg/L)
1. Sodium	21.00
2. Ammonium	NA
3. Methylamine	B
4. Potassium	0.5
5. Dimethylamine	B
6. Magnesium	2.1
7. Calcium	1.4
8. Trimethylamine	B

NA = Not Available
B = Blank

4.8 IONPAC CS12 ANALYSIS OF 12% HYDROFLUORIC ACID

Sample:	100 µL neutralized in the SP10 AutoNeutralizer
Concentrator Column:	Low Pressure Trace Cation Concentrator (TCC-LP1, P/N 046027)
Guard Column:	CG12 Guard Column
Analytical Column:	CS12 Analytical Column
Eluent:	19 mM Methanesulfonic acid (MSA)
Eluent Flow Rate:	1.0 mL/min (4mm)
Suppressor*:	Cation Self-Regenerating Suppressor AutoSuppression Recycle Mode
Expected Background Conductivity:	19 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)

* An Cation MicroMembrane Suppressor CMMS (2mm) or CMMS-I (4mm) may be used in place of the CSRS-I. The Regenerant is 100 mN TBAOH and the Regenerant Flow Rate is 5 mL/min.

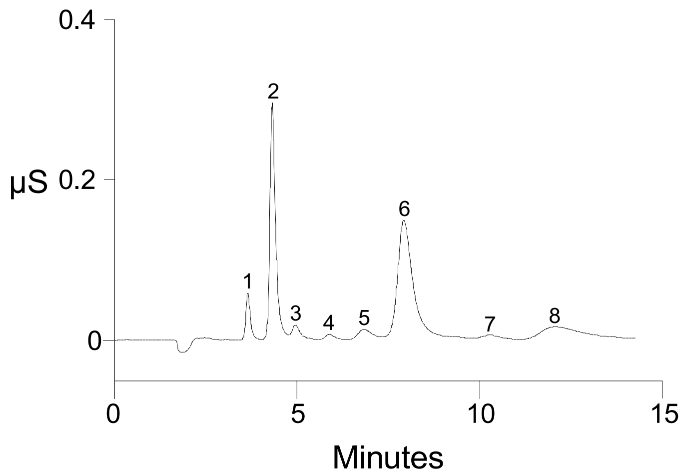

Figure 11

IonPac CS12 Analysis of 12% Hydrofluoric Acid

Table 4

Quantification Data for the IonPac CS12 Analysis of 12% Hydrofluoric Acid

	12% HF Conc. (µg/L)
1. Sodium	8.0
2. Ammonium	NA
3. Methylamine	B
4. Potassium	5.0
5. Dimethylamine	B
6. Magnesium	69
7. Calcium	4.5
8. Trimethylamine	B

NA = Not Available
B = Blank

4.9 IONPAC CS12 ANALYSIS OF 22% PHOSPHORIC ACID

Sample:	100 µL neutralized in the SP10 AutoNeutralizer
Concentrator Column:	Low Pressure Trace Cation Concentrator (TCC-LP1, P/N 046027)
Guard Column:	CG12 Guard Column
Analytical Column:	CS12 Analytical Column
Eluent:	Figure 12A: 19 mM Methanesulfonic acid (MSA) Figure 12B: 9 mM Methanesulfonic acid (MSA)/ 0.5 mM Pyridine-2,6-dicarboxylic acid (PDCA)
Eluent Flow Rate:	1.0 mL/min (4mm)
Suppressor*:	Cation Self-Regenerating Suppressor AutoSuppression Recycle Mode
Expected Background Conductivity:	19 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)

* An Cation MicroMembrane Suppressor CMMS (2mm) or CMMS-I (4mm) may be used in place of the CSRS-I. The Regenerant is 100 mN TBAOH and the Regenerant Flow Rate is 5 mL/min.

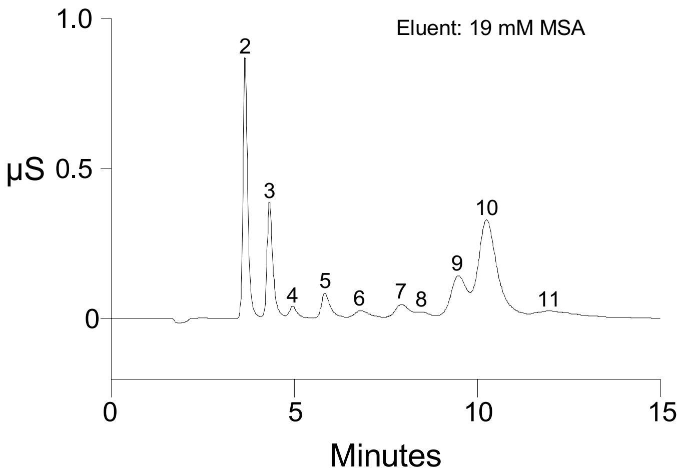

Figure 12A

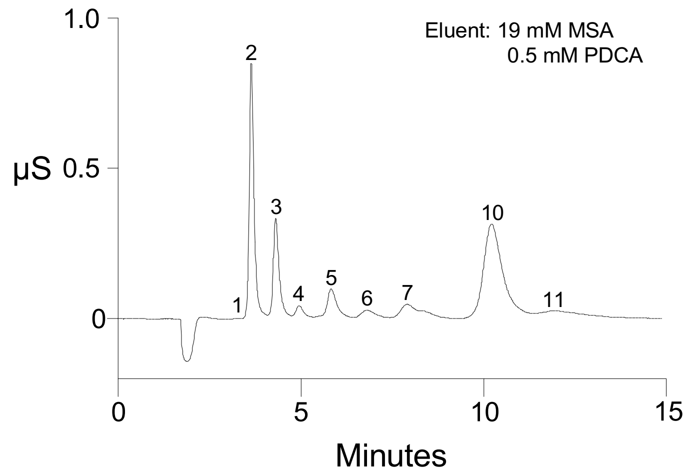

Figure 12B

IonPac CS12 Analysis of 22% Phosphoric Acid

Table 5

Quantification Data for the IonPac CS12 Analysis of 22% Phosphoric Acid

		22% H_2PO_4 Figure 12A 19 mM MSA Conc. (µg/L)	**22% H_2PO_4 Figure 12B 19 mM MSA / 0.5 mM PDCA** Conc. µg/L)
1.	Lithium	ND	0.2
2.	Sodium	150	150
3.	Ammonium	NA	NA
4.	Methylamine	B	B
5.	Potassium	60	80
6.	Dimethylamine	B	B
7.	Magnesium	19	19
8.	Manganese	ND	ND
9.	Transition Metals	NA	ND
10.	Calcium	316	305
11.	Trimethylamine	B	B

ND = None Detected
NA = Not Available
B = Blank

SECTION 5 - TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using the Cation Self-Regenerating Neutralizer (CSRN-I). The SP10 AutoNeutralizer has 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 (Document No. 034980, "SP10 AutoNeutralizer Operator's Manual"), the Ion Chromatograph or the specific cation exchange column set in use, refer to the Troubleshooting Guide in the appropriate Installation Manual. If you cannot solve the problem on your own, contact the DIONEX Regional Office nearest you (see, "DIONEX Worldwide Offices").

5.1 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).

5.1.1 CSRN-I LIQUID LEAKS

A. If there is leakage between the cover and body of the CSRN-I, carefully tighten the fittings in the **ELUENT** and **REGEN IN** and **OUT 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 CSRN-I, check the system back pressure.

   1. If the system back pressure is less than 100 psi with only the CSRN-I in-line, eluent may be flowing from the eluent chamber into the regenerant chamber. In this case the CSRN-I is defective and must be replaced. **Do not attempt to disassemble the CSRN-I and repair it yourself.**

   2. If the system back pressure is greater than 100 psi, the leaks are caused by excessive back pressure downstream from the CSRN-I. Find and eliminate the source of the pressure. The CSRN-I 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 Cation Self-Regenerating Neutralizer (CSRN-I)"). If the CSRN-I continues to leak when operated within the proper back pressure range, it must be replaced.

5.1.2 LOW RESPONSE DUE TO LOSS OF SAMPLE

A. If an CSRN-I fitting is leaking, tighten it carefully until the leak stops. **Do not overtighten.** If the CSRN-I is observed to be leaking from the center seam, see Section 5.1.1, "CSRN Liquid Leaks". If you cannot cure the problem yourself, call the nearest DIONEX Regional Office (see, "DIONEX Worldwide Offices") for assistance.

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 requies excessive recycling, clean the CSRN-I membrane (see Section 6, "Cation Self-Regenerating Neutralizer Cleanup").

D. If cleaning the CSRN-I membrane does not restore neutralization efficiency, the CSRN-I 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").

5.1.3 SYSTEM BACK PRESSURE INCREASES OVER TIME

A. **Excessive back pressure after the CSRN-I will cause it to leak. The CSRN-I cannot operate at pressures greater than 120 psi.** The main source of back pressure for the CSRN-I in normal operation is the TCC-LP1 concentrator column. It should not generate more than 70 psi at 0.5 mL/min flow rate. Periodically test the back pressure generated by the TCC-LP1 on the analytical system to ensure that it is not generating excessive back pressure. If the increased back pressure does not exceed 100 psi, no maintenance is necessary. If the TCC-LP1 generates more than 100 psi at 0.5 mL/min, change the TCC-LP1 inlet bed support assembly and clean the column (see Document No. 034973, "Installation Instructions and Troubleshooting Guide for the Low Pressure Concentrator (TCC-LP1) Column").

B. A blockage in the CSRN-I may result in increased back pressure and potentially damage it. If the back pressure generated by the CSRN-I is greater than 25 psi at a flow rate of 0.5 mL/min, the CSRN-I might have a blockage. Reverse the flow directions of the sample eluent and the external water through the CSRN-I. Connect a separate waste line to the sample eluent flowing from the ELUENT IN port of the CSRN-I. During this flushing operation, do not connect the line between the cell and the CSRN-I to avoid contamination of the cell. If this inverted operation decreases the back pressure, reconnect the CSRN-I to the cell 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.

C. If reversing the flow through the CSRN-I does not decrease the pressure, clean the CSRN-I membranes (see Section 6, "Cation Self-Regenerating Neutralizer Cleanup").

D. If cleaning the CSRN-I membranes does not reduce the pressure, the CSRN-I must be replaced.

E. 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".

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

5.2 SP10 AUTONEUTRALIZER VOLT ALARM

The CSRN-I is designed to operate at a maximum of 8.5 $\pm$ 0.5 V. The Volt Alarm will sound if the CSRN-I does not have liquid in it or if the CSRN-I cable is disconnected.

5.3 SP10 AUTONEUTRALIZER TEMP ALARM

The CSRN-I requires an internal temperature of 40 °C or less. Excessive temperature will cause the CSRN-I to leak. The temperature alarm will sound if the internal temperature of the CSRN-I exceeds 40 $\pm$ 2 °C. This can be caused by an insufficient flow of carrier solution through the CSRN-I. The required minimum flow rate of regenerant solution is 3 - 4 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 - 4 mL/min.

5.4 SP10 AUTONEUTRALIZER 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.

5.4.1 HIGH BACKGROUND CONDUCTIVITY

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

A. Try multiple passes through the CSRN-I.

B. Check for carrier flow out of the CSRN-I **ELUENT OUT Port.**

1. If there is no flow out of the CSRN-I **ELUENT OUT Port**, make sure that the carrier solution is entering the CSRN-I 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 CSRN-I but not out, and there are no visible leaks from the side seam of the CSRN-I, a break in the membrane is probably allowing sample to leak into the regenerant chambers. If this is the case, then the CSRN-I must be replaced. **The CSRN-I is sealed during manufacture. Attempting to open it will destroy it.**

DO NOT ATTEMPT TO DISASSEMBLE THE CATION 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, "Cation Self-Regenerating Neutralizer 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").

5.4.2 LOW NEUTRALIZATION CAPACITY

This problem is caused when the ion exchange sites in the CSRN-I 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. Should the sample types that you are running suddenly not neutralize with multiple passes through the CSRN-I, perform the following procedure:

A. Disconnect the sample line from **Port #5 on the Recycle Valve** to the **ELUENT IN Port** of the CSRN-I at the Recycle Valve end of the line. Direct this line to a separate waste beaker.

B. Disconnect the sample line from the **ELUENT OUT Port** of the CSRN-I to **Port #3 on the Recycle Valve** at the Port #3 end of the line and install a 10-32 to 1/4-28 union (P/N 042806) on this line.

C. Install a plastic syringe with a luer adaptor in the **ELUENT OUT Port** and inject 5 mL of 0.5 N H_2SO_4 through the CSRN-I in the reverse direction to normal flow so that the waste comes out of the **ELUENT IN Port**.

D. Reconnect the sample line from the **ELUENT IN Port** of the CSRN-I to **Port #5 on the Recycle Valve** and the sample line from the **ELUENT OUT Port** of the CSRN-I to **Port #3 on the Recycle Valve**.

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

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").

SECTION 6 - CATION SELF-REGENERATING NEUTRALIZER CLEANUP

This section describes routine cleanup procedures for the Cation Self-Regenerating Neutralizers (CSRN-I) 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 CSRN-I 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 CSRN-I power cord.

B. Disconnect the liquid line between the CSRN-I **ELUENT OUT Port** and **Port #3 on the Recycle Valve** at the Recycle Valve 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 (APM, GPM, AGP or DX-100) to a container with a solution of 0.1 M KCl in 1.0 M HCl. Pressurize the container to 5-10 psi. Pump this solution through the CSRN-I (4mm) at 1-2 mL/min for 30 minutes.

D. Flush the CSRN-I with deionized water for 10 minutes.

E. Reconnect the power cord to the CSRN-I.

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 CSRN-I power cord.

B. Disconnect the liquid line between the CSRN-I **ELUENT OUT Port** and **Port #3 on the Recycle Valve** at the Recycle Valve 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 CSRN-I cleanup. Pressurize the bottle to 5-10 psi pressure. Pump this solution through the CSRN-I at 1-2 mL/min for 30 minutes.

D. Flush the CSRN-I with deionized water for 10 minutes.

E. Reconnect the power cord to the CSRN-I.

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

Note 1, Consumables Department, 07/20/98 06:26:53 AM

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