



Thermo Scientific

Dionex CES 300

Product Manual

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August 2012

PRODUCT MANUAL

FOR THE

ANION CAPILLARY ELECTROLYTIC SUPPRESSOR 300

(Dionex ACES 300, P/N 072052)

CATION CAPILLARY ELECTROLYTIC SUPPRESSOR 300

(Dionex CCES 300, P/N 072053)

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SECTION 1 – INTRODUCTION

1.1. Capillary Electrolytic Suppressor

The Thermo Scientific Dionex CES Capillary Electrolytic Suppressors are optimized for eluent flow rates typically seen in capillary systems (5–30 $\mu\text{L}/\text{min}$). When used for anion analysis, the Thermo Scientific Dionex ACES Anion Capillary Electrolytic Suppressor converts highly conductive hydroxide-based eluents into pure water, thus reducing their conductivity on a conductivity detector. While suppressing the eluent, the Dionex ACES™ 300 also converts the analytes into their more conductive hydronium (acid) form, thus increasing their sensitivity under conductivity detection. Likewise, when used for cation analysis, the Thermo Scientific Dionex CCES Cation Capillary Electrolytic Suppressor converts highly conductive methanesulfonic acid (MSA) eluents into pure water; simultaneously, the analytes are converted to their more conductive hydroxide form, increasing their sensitivity.

As shown in Figure 1, the Dionex CES™ suppressor uses a three chamber design to minimize dead volume while maximizing suppression capacity and reducing noise. The eluent chamber is comprised of an ion-exchange capillary tubing embedded in a bed of ion-exchange resin that facilitates the efficient exchange of the eluent counter-ions for regenerant ions.

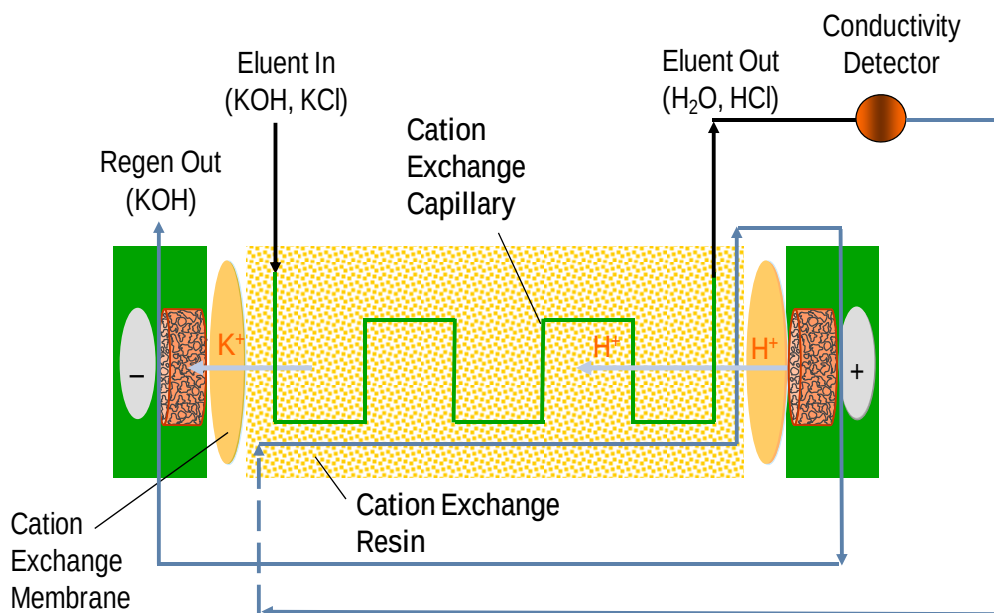


Figure 1
Dionex ACES 300 Suppressor Operated in the AutoSuppression® Recycle Mode

There are two electrode chambers that are separated from the eluent chamber by ion-exchange membranes. The deionized water stream, after passing through the ion-exchange bed in the eluent chamber, passes through the cathode and anode chambers serially. When current is passed through the electrodes, the regenerant ions are generated in the first electrode chamber; these ions migrate into the eluent chamber via an electric field, maintaining the ion exchange bed in the eluent chamber in the regenerated form. After co-ions exchange from the eluent ion exchange capillary, the co-ions are pushed out of the eluent chamber via the electric field into the second electrode chamber. Finally, these co-ions are neutralized by the ions generated in the second electrode chamber. The eluent suppression process is illustrated for Anion Suppressor in Figure 2 and for Cation Suppressor in Figure 3.

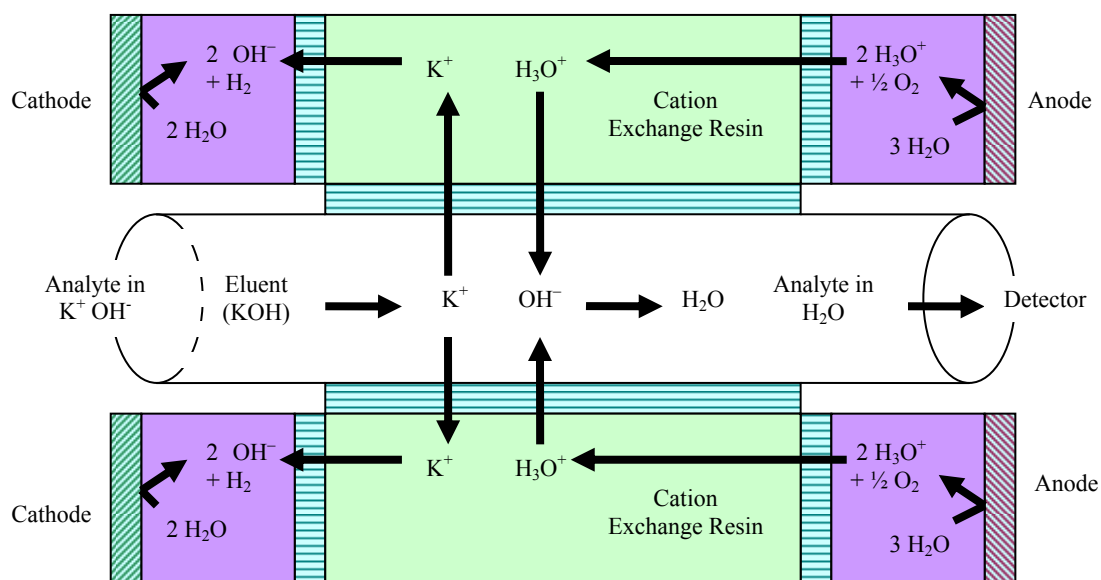


Figure 2
AutoSuppression with the Dionex ACES 300.

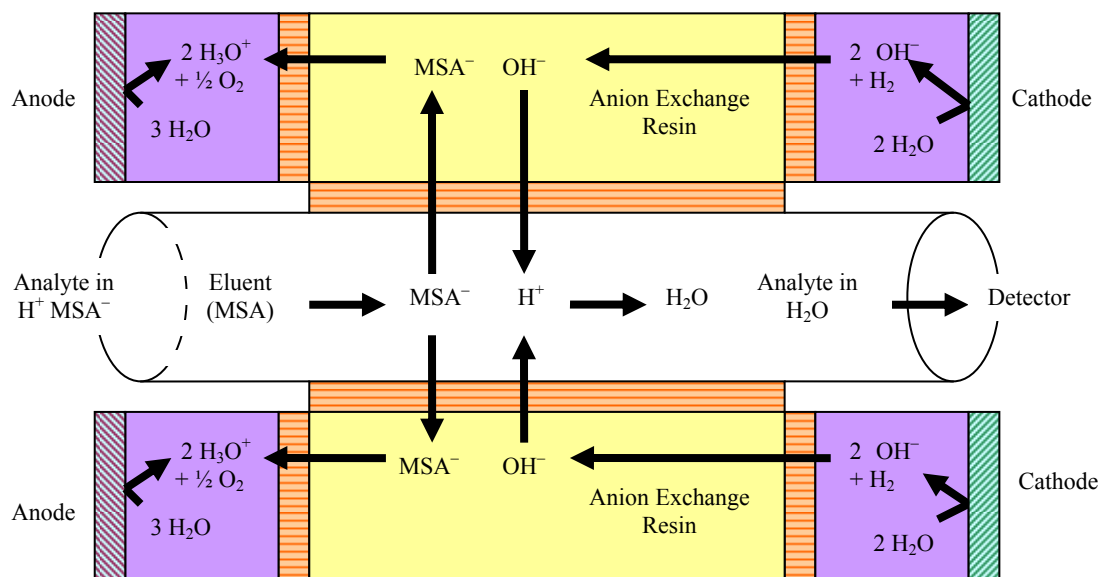


Figure 3
AutoSuppression with the Dionex CCES 300.

Dionex CES 300 suppressors are designed to operate in an Thermo Scientific Dionex ICS-5000 capillary IC system. The use of Dionex CES 300 suppressors in other IC systems is not recommended,

1.2. Overview of Suppression Modes

Two basic modes of suppression can be performed with the Capillary Electrolytic Suppressor (Dionex CES 300):

- AutoSuppression Recycle Mode
- AutoSuppression External Water Mode

AutoSuppression Recycle Mode: In AutoSuppression, the Dionex CES 300 uses detector cell effluent to generate the regenerant for eluent suppression. There are two modes of AutoSuppression operation. The most simple and recommended mode of operation is the AutoSuppression Recycle Mode. In this mode of operation, eluent flows from the eluent outlet of the suppressor into the conductivity cell and is then recycled back through the Dionex CES 300 regenerant chambers. This eliminates the need for an external source of regenerant water but limits the regenerant flow rate to the eluent flow rate. The AutoSuppression Recycle Mode is the most common mode of operation and is recommended for aqueous eluents.

AutoSuppression External Water Mode: This mode incorporates an external source of deionized water flowing through the regenerant chambers. The detector cell effluent is directed to waste. This requires the installation of pressurized bottle system or additional pump to provide an external source of water. With this configuration the regenerant flow rate is not limited to the eluent flow rate. The AutoSuppression External Water mode is primarily recommended for eluents containing solvent (up to 40%), samples that have high solvent or transition metal content, or for interfacing to destructive detectors such as MS detectors.

1.3. Mode of Operation Selection

The Dionex CES 300 mode of operation depends mainly on your eluent or sample composition, or your type of detection. The eluent and sample compatibilities are shown in

Table 1. For example, eluents containing organic solvents are not compatible with the AutoSuppression Recycle Mode. The AutoSuppression External Water Mode should be used instead.

Eluent / Sample Composition	Recycle Mode	External Water Mode
Aqueous Eluents	Yes	Yes
Eluent Containing Organic Solvents	No	Yes (Up to 40%)
Samples Containing Organic Solvents	Not Recommended	Yes
High Transition Metal Content	Not Recommended	Yes

Table 1
Eluent and Sample Composition and Suppression Mode Compatibility.

1.3.1. AutoSuppression Recycle Mode

The AutoSuppression Recycle Mode uses the neutralized conductivity cell effluent as the source of water for the regenerant chamber water. This is the preferred method of operation for the Dionex CES 300. The advantage of this mode of operation is simplicity and ease of use. This mode reliably provides AutoSuppression for most suppressed conductivity applications using solvent-free eluents. As the eluent passes through the Dionex CES 300, it is neutralized to its weakly ionized form. After passing through the conductivity cell, the effluent is redirected to the regenerant inlet on the Dionex CES 300, thus supplying it with a source of water containing minute amounts of diluted analyte. The amount of water flowing through the regenerant chambers is therefore limited to the eluent flow rate. See Section 3.4 for complete operating instructions.

1.3.2. AutoSuppression External Water Mode

The AutoSuppression External Water Mode is used for any application requiring organic solvents in the eluent or sample, or when the sample is heavily contaminated with transition metals. This mode uses a constant source of deionized water from a pressurized bottle or other source of deionized water that delivers 0.01 to 0.05 mL/min. The amount of water flowing through the regenerant chambers is therefore independent of the eluent flow rate. The AutoSuppression External Water Mode eliminates the potential for build-up of contaminating ions resulting from the oxidation of solvents. It also prevents re-entry of transition metals into the regenerant chambers where they may precipitate. Any analysis performed using the AutoSuppression Recycle Mode can also be performed using the AutoSuppression External Water Mode. See Section 3.5 for complete operating instructions.

SECTION 2 – INSTALLATION

2.1. System Requirements

The Dionex CES 300 is designed to be run on a Dionex Capillary Ion Chromatography System, such as an Dionex ICS-5000 equipped with a capillary pump and IC Cube, Anion or Cation exchange column set and conductivity detection. The Dionex ICS-3000, ICS-2100, ICS-1600, and ICS-1100 and other ion chromatography systems are not equipped to operate with a Dionex CES suppressor.

2.2. Back Pressure Coils for the Dionex CES 300

Back pressure coils are not recommended for the Dionex CES 300 suppressor when operating with hydroxide or MSA eluents.

2.3. Gas Separator Waste Tube for the Dionex CES 300

The Gas Separator Waste Tube (P/N 045460) is an integral part of the Dionex IC system using an electrolytic suppressor. It separates any hydrogen gas generated in the Dionex ACES 300 during the electrolytic generation of hydronium ions (H_3O^+) or hydroxide ions (OH^-) for the Dionex CCES 300. The Gas Separator Waste Tube is used to avoid concentrating the gas in the waste container. The Gas Separator Waste Tube is shipped in one of the ShipKits of your system.



CAUTION

Do not cap the waste reservoir.



SAFETY

Minimal hydrogen gas generated by the Dionex CES 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.

2.3.1. Assembly

Assemble and install the Gas Separator Waste Tube and waste line following the steps below.

- A. Use one or two couplers (P/N 045463) to connect two or three lengths of 1/2" i.d. black polyethylene tubing (P/N 045462) depending on the waste container depth. Extend the top of the Waste Separator Tube above the top of the Waste container.
- B. Place the Gas Separator Waste Tube with the 1/8" o.d. tubing attached into the waste container. Ensure the bottom of the Gas Separator Waste Tube is resting on the floor of the waste container, the top of the device (where the white 1/8" o.d. tubing meets the black 1/2" o.d. tubing) is above the top of the container, and that both the Gas Separator Waste Tube and the waste container are open to the atmosphere.

2.4. Capillary Electrolytic Suppressor Current Calculation

For optimal performance of the Dionex CES 300, Dionex recommends the use of the following formulas to calculate the current applied to the suppressor.

For Dionex ACES 300:

$$\text{SuppressorCurrent (mA)} = ((2 * \text{Carbonate concentration (mM)} + \text{Bicarbonate concentration (mM)} + \text{Hydroxide concentration (mM)}) * \text{FlowRate (}\mu\text{L/min)} / 100) + 5 \text{ (mA)}$$

For Dionex CCES 300:

$$\text{SuppressorCurrent (mA)} = ((2 * \text{H}_2\text{SO}_4 \text{ concentration (mM)} + \text{MSA concentration (mM)}) * \text{FlowRate (}\mu\text{L/min)} / 100) + 5 \text{ (mA)}$$

Multiply by a factor of 1000 to convert the flow rate in mL/min to $\mu\text{L/min}$. For example, 0.01 mL/min = 10 $\mu\text{L/min}$.

The suppressor current for Dionex CES 300 suppressors can also be determined using CM 6.8, 7.0 or later suppressor current calculation wizard. The maximum current for Dionex CES 300 suppressor is 25 mA.

2.5. Installation

To install, remove the caps from the Dionex CES 300 regenerant ports on the rear of the cartridge and slide the Dionex CES 300 into the suppressor receptacle of the IC cube installed in a Dionex Capillary IC system. Press the Dionex CES 300 firmly to establish the electrical and regenerant connections at the rear, and then screw the thumb screws to lock the Dionex CES 300 in place.

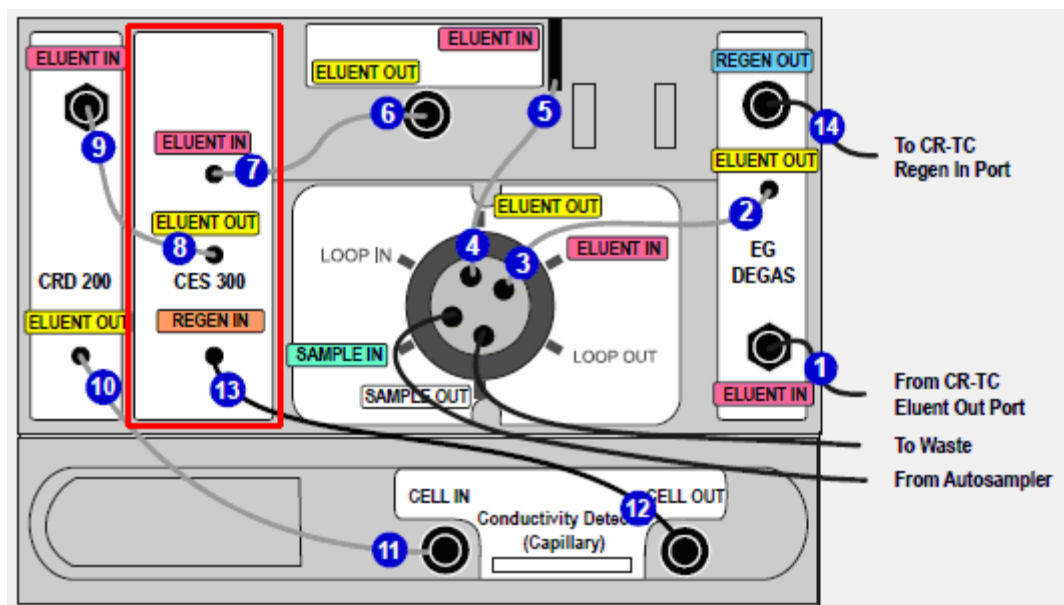


Figure 4

Placement of the Dionex CES 300 in a Dionex ICS-5000 IC Cube (Dionex CES 300 highlighted in Red) with optional Thermo Scientific Dionex CRD 200 Carbonate Removal Device.



Do not attempt to plug the Dionex CES 300 into any receptacle other than the Suppressor receptacle; damage to the regenerant ports may occur. See the system manual for details.



Dionex CES 300 suppressors are designed to operate only with Dionex ICS Capillary IC Systems equipped with IC Cubes.



It is required that the temperature of the upper compartment of the DC be maintained at 15 °C when using Dionex CES 300 suppressors in a Dionex ICS Capillary IC system.

SECTION 3 – OPERATION

This section provides instructions for the start-up and operation of the Dionex CES 300 including the selection process and suppression modes of operation.

3.1. Chemical Purity Requirements

Precise and accurate results require eluents free of ionic impurities. Chemicals and deionized water used to prepare eluents must be pure as described below. Low trace impurities and low particulate levels in eluents and regenerants also help protect the Dionex CES 300 and system components from contamination. Dionex CES 300 performance is not guaranteed when the quality of the chemicals and water used to prepare eluents has been compromised.

3.2. Inorganic Chemicals

Reagent Grade inorganic chemicals should always be used to prepare ionic eluents. Preferably, a lot analysis on each label will certify each chemical as meeting or surpassing the latest American Chemical Society standard for purity, a universally accepted standard for reagents.

3.2.1. Solvents

Since solvents used with the Dionex CES 300 are added to ionic eluents to modify the ion exchange process or improve sample solubility, the solvents used must be free of ionic impurities. However, since most solvent manufacturers do not test for ionic impurities, the highest grade of solvents available should be used. Currently, several manufacturers are making ultra-high purity solvents that are compatible for HPLC and spectrophotometric applications. These ultra-high purity solvents will usually ensure that your chromatography is not affected by ionic impurities in the solvent. Dionex has obtained consistent results using High Purity Solvents manufactured by Burdick and Jackson and Optima® Solvents by Fisher Scientific.

3.2.2. Deionized Water

The deionized water used to prepare eluents should be degassed Type I Reagent Grade Water with a specific resistance of 18.2 megohm-cm. The water used for the AutoSuppression External Water Mode should have a specific resistance of 18.2 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

The Dionex CES 300 is plumbed directly after the analytical column and before the carbonate removal device (if fitted) and conductivity detector cell. On the Dionex ICS-5000, the suppressor is plugged into the suppressor receptacle of the IC Cube in the DC; electrical contact and regenerant connections are established upon installation. To install, remove the caps from the Dionex CES 300 regenerant ports on the rear of the cartridge and slide the Dionex CES 300 into the receptacle. Press the Dionex CES 300 firmly to establish the regenerant connections, and then screw the thumb screws to lock the Dionex CES 300 in place. Loosen the thumb screws and pull the cartridge out to remove Dionex CES 300. Ensure the Dionex CES 300 is plumbed properly according to the selected mode of operation.



WARNING

Keep the regenerant chambers full with the appropriate regenerant solution or water. The membranes and screens in the Dionex CES 300 must be completely hydrated to maintain liquid seals and chromatographic performance.



CAUTION

The correct amount of back pressure for optimum operation is 40 psi. Back pressures over 150 psi after the Dionex CES 300 can cause irreversible damage.



CAUTION

Do not cap the waste reservoir.



To ensure proper operation of the Dionex CES suppressor, the temperature of the upper compartment of the DC module (where the IC Cube resides) must be set to 15 °C.



Hydrogen gas generated by the Dionex CES 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.

3.3.1. Hydration

Hydrating the Suppressor Regen Chamber

- A. Install the Dionex CES 300 Suppressor in the IC Cube.
- B. Install the Dionex CRD 200 or the Dionex CRD bypass cartridge in the IC Cube.
- C. Connect a piece of 24 inch x 1/10 inch x 0.0025 inch blue PEEK tubing (P/N 072203) to the outlet of the Dionex ICS-5000 Capillary Pump Pulse Damper.
- D. Connect this tubing to the union on the end of the tubing exiting the REGEN IN port of the capillary suppressor cartridge (Figure 3). (This is a temporary connection.)

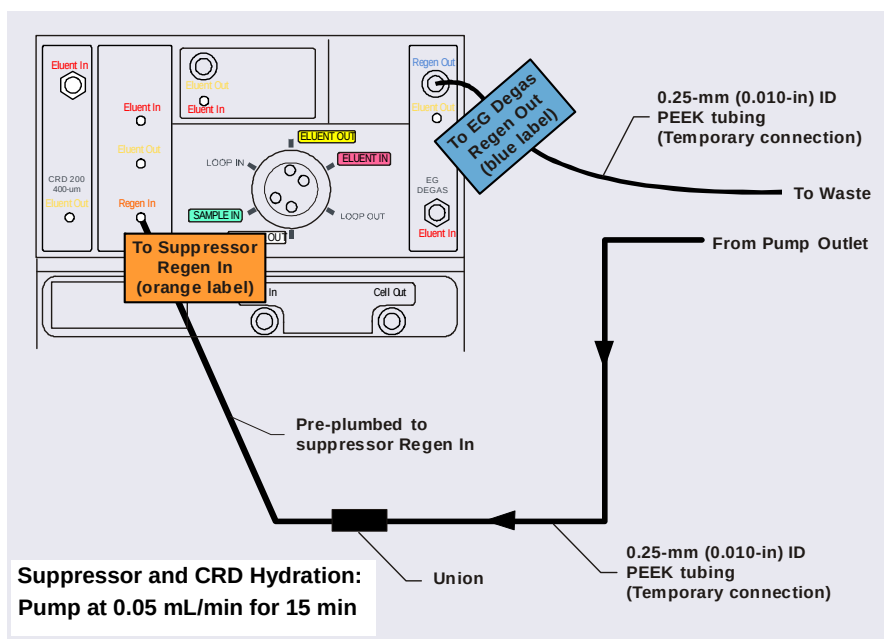


Figure 5
Flow Schematic for Hydrating the Capillary Suppressor and Dionex CRD

- E. Connect a piece of 0.25 mm (0.010 in) ID PEEK tubing to the REGEN OUT port of the capillary EG degas cartridge in the IC Cube. Direct the other end of the tubing to waste. (This is a temporary connection.)
- F. Set the pump flow rate to 0.05 mL/min and turn on the pump flow.
- G. Run the pump for 15 minutes and then turn off the pump flow. This hydrates the capillary suppressor and flushes the internal IC Cube regenerant manifold chambers.

Hydrating the Suppressor Eluent Chamber

- Install the Dionex CES 300 Suppressor in the IC Cube.
- Install the Dionex CRD 200 or the Dionex CRD bypass cartridge in the IC Cube.
- Connect a piece of 24 inch x 1/10 inch x 0.0025 inch blue PEEK tubing (P/N 072203) to the outlet of the Dionex ICS-5000 Capillary Pump Pulse Damper.
- Connect this tubing through a 10-32 union to the inlet end of the Dionex CES 300 suppressor.
- Set the pump flow rate to 0.01 mL/min and turn on the pump flow.
- Run the pump for 10 minutes and then turn off the pump flow. This hydrates the capillary suppressor.

3.4. Plumbing for the AutoSuppression Recycle Mode of Operation

The AutoSuppression Recycle Mode is the easiest method of operation. As the eluent passes through the suppressor, it is neutralized to produce its weakly ionized form. After passing through the conductivity cell, this effluent can be redirected to the regenerant inlet of the suppressor, thus supplying it with a source of water containing a small amount of diluted analyte (see Figure 6). The main advantage of this mode is its simplicity and ease of use. It is not necessary to have an external supply of water available for the suppressor.

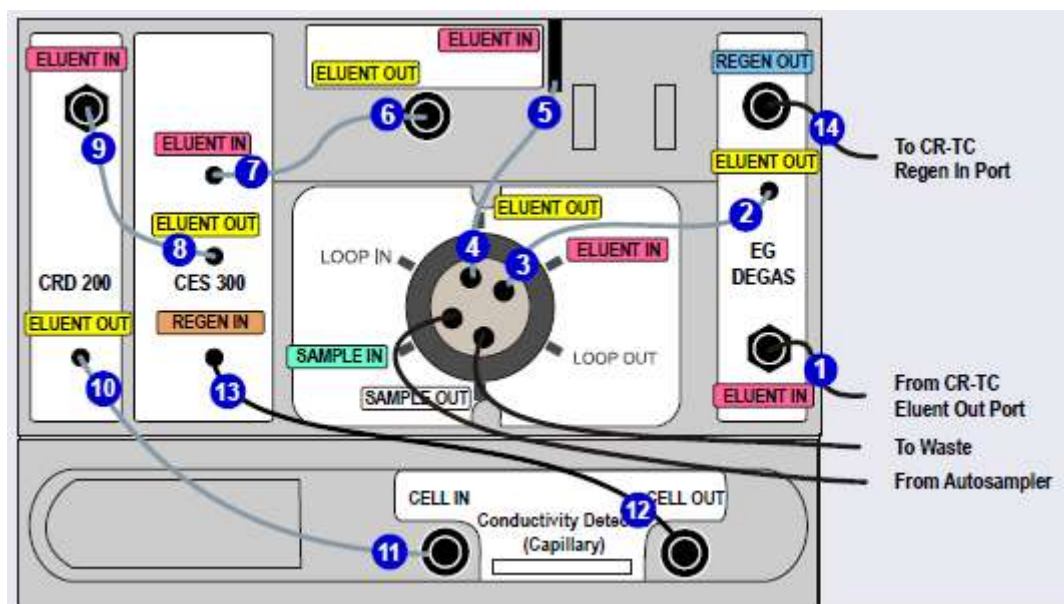


Figure 6
Schematic of Dionex ICS-5000 IC Cube in AutoSuppression Recycled Eluent Mode.
Connections 12 and 13 route cell effluent to the Dionex CES 300 Regen In.



To ensure proper operation of the Dionex CES suppressor, the temperature of the upper compartment of the DC module (where the IC Cube resides) must be set to 15 °C.



Only use the AutoSuppression Recycle Mode for eluents without organic solvents and samples without excessive metallic contaminants such as iron.

3.4.1. Eluent Flow Path Connections in the AutoSuppression Recycle Mode

Use only the provided pre-cut 0.0025" i.d. PEEK tubing (light blue) with 10-32 ferrule/bolt fittings. When connecting the fittings, make sure that the ferrule and fitting bolt are at least 2 mm (0.1 in) from the end of the tubing before you insert the tubing into the port. Do not place the ferrule and fitting bolt flush with the end of the tubing.

Figure 7 illustrates the correct and incorrect placement of the ferrule and fitting bolt on the tubing.

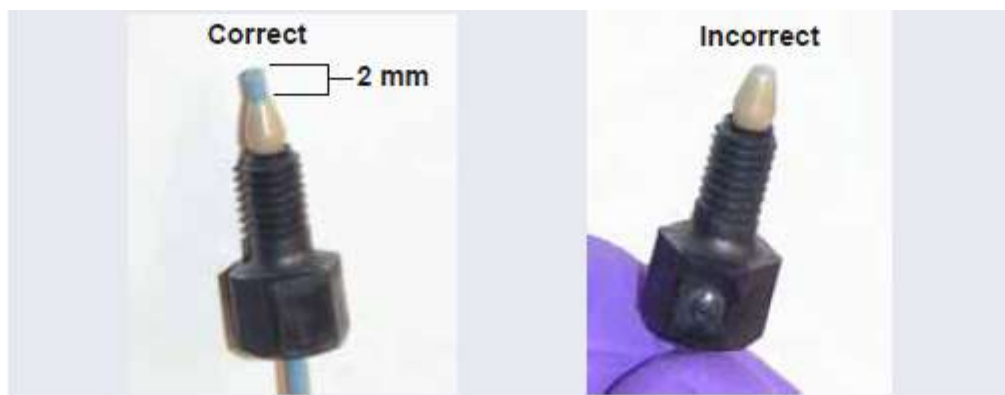


Figure 7

Correct and Incorrect Ferrule and Fitting Placement for Capillary Tubing Connections



NOTE

Always use the precut tubing included with the suppressor and other capillary devices to make connections. Do not cut or alter this tubing; the tubing is factory cut for an extremely low dead volume interface.

- A. Install the Dionex CES 300 in the Suppressor cartridge receptacle.
- B. Connect the outlet of the capillary column to the ELUENT IN of the Dionex CES 300 (Connection 7 in Figure 6).
- C. Connect the ELUENT OUT port of the Dionex CES 300 to the inlet of the Dionex CRD 200 (Connection 9 in Figure 6) if a Dionex CRD 200 is installed, or to the inlet of the conductivity cell (Connection 11 in Figure 6) if no Dionex CRD 200 is installed.
- D. Connect the REGEN IN port of the Dionex CES 300 to the outlet of the conductivity cell (Connection 12 in Figure 6) using the black (0.010") REGEN IN tubing.

3.5. Plumbing for the AutoSuppression External Water Mode Operation

Any analysis that can be performed using the AutoSuppression Recycle Mode can be done using the AutoSuppression External Water Mode. A constant source of deionized water having a specific resistance of 18.2 megohm, or greater, is supplied to the regenerant chambers to generate hydronium or hydroxide ions for neutralization.

**CAUTION**

AutoSuppression External Water Mode is recommended for eluents with organic solvents, samples with high levels of metallic contaminants such as iron, or destructive detection methods where a source of suppressed eluent is not available.

3.5.1. Eluent Flow Path Connections for the AutoSuppression External Water Mode

Use only the provided pre-cut 0.0025" i.d. PEEK tubing (light blue) with 10-32 ferrule/bolt fittings. When connecting the fittings, make sure that the ferrule and fitting bolt are at least 2 mm (0.1 in) from the end of the tubing before you insert the tubing into the port. Do not place the ferrule and fitting bolt flush with the end of the tubing.

Figure 7 illustrates the correct and incorrect placement of the ferrule and fitting bolt on the tubing.

**NOTE**

Always use the precut tubing included with the suppressor and other capillary devices to make connections. Do not cut or alter this tubing; the tubing is factory cut for an extremely low dead volume interface.

- A. Install the Dionex CES 300 in the Suppressor cartridge receptacle.
- B. Connect the outlet of the analytical column to the ELUENT IN of the Dionex CES 300 (Connection 7 in Figure 6).
- C. Connect the ELUENT OUT port of the Dionex CES 300 to the inlet of the Dionex CRD 200 (Connection 9 in Figure 6) if a Dionex CRD 200 is installed, or to the inlet of the conductivity cell (Connection 11 in Figure 6) if no Dionex CRD 200 is installed.
- D. If using conductivity detection only, install a waste line to the conductivity cell outlet. If using a hyphenated technique such as CD-MSD, connect the inlet of the second detector to the outlet of the conductivity cell using tubing of the appropriate length and i.d. depending on your application requirements. If using a detection technique other than conductivity, connect the outlet of the Dionex CES 300 to the inlet of the detector using tubing of the appropriate length and i.d. depending on your application requirements.
- E. Connect the REGEN IN port of the Dionex CES 300 to the source of external water.
- F. Adjust the flow rate of water from the external water delivery system to the REGEN IN of the Dionex CES 300 to approximately 0.050 mL/min.

**NOTE**

To ensure the optimal performance of the Dionex CES 300 in the AutoSuppressor External Water Mode, it is recommended to use a pump that is capable of delivering deionized water accurately and reproducibly at 0.050 mL/min.

3.5.2. Using a Pressurized Bottle to provide External Water

The SRS 300 Pressurized Bottle Installation Kit (P/N 038018) contains all of the components needed to install and operate the Dionex CES 300 with a pressurized water reservoir. The kit contains the Installation Parts Kit (P/N 039055), a 25 psi regulator (P/N 038201) and a 4 liter water reservoir (P/N 039164).

- A. Make the following air line connections:
 - a. Locate the pieces of tinted 1/8" o.d. plastic tubing (P/N 030089) supplied in the Installation Parts Kit.
 - b. Push the end of one piece of 1/8" o.d. tubing over the barbed fitting of the regulator. Connect the other end of the tubing to the source of air pressure.
 - c. Push one end of the second piece of 1/8" o.d. 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.
- B. Make the following water line connection.
 - a. Use a 10-32 to 1/4-28 coupler (P/N 42806) 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 tubing of the Dionex CES 300.
- C. Fill the water source reservoir. 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 Chromatography Module.
- D. With no current applied, adjust the external water flow rate to approximately 0.050 to 0.10 mL/min by using a graduated cylinder and measuring the flow from the REGEN OUT line of the EG Degas cartridge (Connection 14 in Figure 6). The pressure applied to the reservoir can vary from 0–25 psi (the lower and upper pressure limits of the water reservoir) but the typical operating pressure is between 10–15 psi. Please note that this value is highly system dependent. After turning on the current, the external water flow rate will be less because the electrolysis of water will generate a small amount of hydrogen and oxygen gases that will be seen as bubbles from the REGEN OUT waste line. The flow rate with current turned on should be at least equal to the eluent flow rate.



SAFETY

A safety relief valve on the reservoir regulator prevents pressure greater than 25 psi from being applied to the water reservoir.

3.5.3. Using a External Pump to provide External Water

To ensure the optimal performance of Dionex CES 300 in the AutoSuppression External Water Mode, it is recommended to use a pump such as the Dionex DP pump that is capable of delivering deionized water at 0.05 mL/min accurately and consistently.

For peristaltic pump plumbing refer to the “MASTERFLEX® C/L® Peristaltic Pump Quick Start Guide” (P/N 065203). Pump the Deionized water into the REGEN IN port of the Dionex CES 300 using the peristaltic pump at 0.050 mL/min. Use a graduated cylinder and measure the flow from the REGEN OUT line of the EG Degas cartridge (Connection 14 in Figure 6).

3.6. Capillary Electrolytic Suppressor 300 (Dionex CES 300) Storage

The Dionex CES 300 is shipped with DI water as the storage solution. If the suppressor will not be used for more than one week, prepare it for storage. The membranes and resin in the Dionex CES 300 must be completely hydrated to maintain liquid seal and chromatographic performance. Plug all the ports after hydration.

3.6.1. Storage

- A. Remove the Dionex CES 300 from the IC Cube.
- B. Flush the Dionex CES 300 eluent port with deionized water for 10 minutes using the eluent delivery pump set to 0.010 mL/min with the eluent generator, CR-TC, separation column bypassed.
- C. Use the eluent delivery pump to pump deionized water through the REGEN IN port for 10 minutes at 0.05 mL/min.
- D. Use a 10-32 coupler to connect the suppressor ELUENT INLET and OUTLET.
- E. Plug the REGEN OUT port using the nylon plastic cap provided with the Dionex CES suppressor.

SECTION 4 – TROUBLESHOOTING GUIDE

The purpose of the Troubleshooting Guide is to help you solve operating problems that may arise while using the Dionex CES 300. For more information on problems that originate with the Ion Chromatograph System or the specific exchange column set in use, refer to the Troubleshooting Guide in the appropriate Product Manual. If you cannot solve the problem on your own, contact the Dionex Regional Office nearest you (see Dionex Worldwide Offices on the Reference Library CD-ROM).

4.1. Over-voltage Alarm

Higher resistance in the suppressor device typically causes an over-voltage alarm. High resistance occurs when:

- A. The suppressor is operated with higher currents than the recommended currents. The suppressor should only be operated at the current recommended for the eluent concentration and flow rate.
- B. The suppressor is exposed to contaminants such as iron, other metals or organics. Refer to the appropriate cleanup procedure and implement the recommended cleanup. In cases when the contamination is continuous, a routine cleanup step on a weekly basis is recommended as part of preventive maintenance.

4.2. Small or Increasing Analyte Peak Areas

This problem is caused by running eluent through the Dionex CES 300 with the.

- A. Disconnect the line from the analytical column attached to the ELUENT IN port of the Dionex CES 300.
- B. Disconnect the line from the ELUENT OUT port of the Dionex CES 300.
- C. For the Dionex ACES 300:
 - a. Fill a 1 mL plastic syringe with 0.5 mL of 200 mN H₂SO₄.
 - b. Connect the OUTLET of the syringe to the ELUENT INLET of the Dionex ACES suppressor.
 - c. Push approximately 0.3 mL of 200 mN H₂SO₄ slowly through the ELUENT IN port of the Dionex ACES suppressor.
 - d. Use a piece of pH paper to collect a small drop of the effluent coming out of the Dionex ACES suppressor OUTLET.
 - e. Make sure that the pH paper indicates the effluent is less than pH 1.
 - f. Connect a piece of 24 inch x 1/10 inch x 0.0025 inch blue PEEK tubing (P/N 072203) to the OUTLET of the Dionex ICS-5000 capillary pump pulse damper.
 - g. Connect this tubing through a 10-32 union to the INLET end of the Dionex CES 300 suppressor.
 - h. Set the pump flow rate to 0.01 mL/min and turn on the pump flow.
 - i. Run the pump for 10 minutes and then turn off the pump flow.
- D. For the Dionex CCES 300:
 - a. Fill a 1 mL plastic syringe with 0.5 mL of 200 mN NaOH.
 - b. Connect the OUTLET of the syringe to the ELUENT INLET of the Dionex ACES suppressor.
 - c. Push approximately 0.3 mL of 200 mN NaOH slowly through the ELUENT IN port of the Dionex ACES suppressor.
 - d. Use a piece of pH paper to collect a small drop of the effluent coming out of the Dionex ACES suppressor OUTLET.
 - e. Make sure that the pH paper indicates the effluent is less than pH 3.
 - f. Connect a piece of 24 inch x 1/10 inch x 0.0025 inch blue PEEK tubing (P/N 072203) to the OUTLET of the Dionex ICS-5000 capillary pump pulse damper.
 - g. Connect this tubing through a 10-32 union to the INLET end of the Dionex CES 300 suppressor.
 - h. Set the pump flow rate to 0.01 mL/min and turn on the pump flow.
 - i. Run the pump for 10 minutes and then turn off the pump flow.
- E. Reconnect the line from analytical column to the ELUENT IN port of the Dionex CES 300 and the line from the ELUENT OUT port of the Dionex CES 300 to the ELUENT IN port of the conductivity detector cell.
- F. If you are in the Auto Suppression Recycle mode of operation, begin pumping eluent and immediately turn on the power.

- G. If you are in the Auto Suppression External water mode of operation, establish water flow through the regenerant chambers, begin pumping eluent and immediately turn on the power.

**NOTE**

Do not let the eluent flow through the Dionex CES 300 for more than a few minutes without turning on the power.

**NOTE**

The sulfuric acid in the above steps could be replaced with a non oxidizing strong acid such as Methanesulfonic acid (MSA) which is recommended when pursuing MS applications.

**NOTE**

Do not use the Analytical pump for regeneration purpose as this will contaminate the pump.

4.3. High Background Conductivity

- A. Check for eluent flow out of the Dionex CES 300 ELUENT OUT port.
- If there is no flow out of the Dionex CES 300 ELUENT OUT port, make sure that eluent is entering the Dionex CES 300 at the ELUENT IN port. If there is no flow at this point, trace the eluent flow path backward through the system to find and remove the blockage.
 - If there is flow into the Dionex CES 300 but not out, and there are no visible leaks from the suppressor, a break in the eluent capillary is probably allowing eluent to leak into the regenerant chambers. If this is the case, then the Dionex CES 300 must be replaced. The Dionex CES 300 is sealed during manufacture; attempting to open it will destroy it.
 - If there is flow from the ELUENT OUT port, but no eluent suppression, the membrane may have been contaminated. Try to restore system performance by cleaning the membrane.
- B. Check the eluent concentration to be sure that it is correct. Be sure that water of the required purity is being used as eluent (see Section 3.1, “Chemical Purity Requirements”). If the eluent concentration is high, the Dionex CES 300 may not be set up to suppress the high concentration resulting in high background conductivity. Use CM 6.8, 7.0 or later suppressor current calculator wizard to calculate the correct suppression current setting for Dionex CES 300 suppression.
- C. Contact the nearest Dionex Regional Office (see, “Dionex Worldwide Offices”) if you cannot solve the problem on your own.

**NOTE**

Do not exceed 25 mA with the Dionex CES 300 suppressor.

4.4. Drifting baseline

If the baseline drifts steadily upward, increase the current setting up to 25 mA if necessary to reduce the background conductivity. As the background conductivity decreases, the baseline drift should decrease.

**NOTE**

Do not exceed 25 mA with the Dionex CES 300 suppressor.

4.5. Noisy baseline

If the baseline is noisy (> 5 nS), it could be caused by trapped air bubbles in the cell or tubing. Burp or release the trapped bubbles by gently tapping on the cell while the fittings are slightly loosened or bleeding the tubing.

4.6. Decreased sensitivity

- A. Check for leaks throughout the system. If a fitting is leaking, tighten it carefully until the leak stops. Do not over tighten. If the Dionex CES 300 is observed to be leaking, see Section 4.8, “Liquid Leaks.” If you cannot cure the problem yourself, call the nearest Dionex Regional Office (see, “Dionex Regional Offices”) for assistance.
- B. Ensure that the injection valve is operating correctly. Refer to the valve manuals that accompany the chromatography module for troubleshooting assistance.
- C. Pursue regeneration of the suppressor as outlined in section 4.2.
- D. If sensitivity remains low, clean the suppressor membrane (see Section 5, “Suppressor Cleanup”).
- E. Check the backpressure on the cell. Verify it is not exceeding 40 psi in the current plumbing configuration and flow rate.
- F. Replace the Dionex CES 300 if cleaning the suppressor membrane does not restore sensitivity.
- G. Contact the nearest Dionex Regional Office (see, “Dionex Worldwide Offices”) if you cannot solve the problem on your own.

4.7. System Back Pressure Increases Over Time

- A. If the increased back pressure does not affect system performance, no maintenance is necessary.
- B. Check the conductivity cell backpressure. If disconnecting the cell lowers the pressure by more than 40 psi, replace the cell or remove the blockage causing the increased pressure. Backpressure over 150 psi after the suppressor can cause irreversible damage.
- C. Find and eliminate any system blockage. Bypass the Dionex CES 300 by coupling the lines attached to the ELUENT IN and ELUENT OUT ports. If the back pressure decreases by less than 150 psi with the Dionex CES 300 out of line, a blockage in the system rather than in the Dionex CES 300 is causing the high pressure.
- D. Remove a blockage from Dionex CES 300 by reversing the eluent flow. If the back pressure decreases by more than 150 psi with the Dionex CES 300 out of line, the high pressure may be caused by a blockage in the Dionex CES 300. Reverse the direction of flow of the eluent or both the eluent and the external water through the Dionex CES 300. After the pressure drops, allow eluent, or eluent and regenerant, to flow to waste for several minutes after the pressure drops. Perform step A of Section 3.2, “Startup” and reinstall the Dionex CES 300 in the appropriate configuration.
- E. Clean the suppressor membranes if reversing the flow through the Dionex CES 300 does not decrease the pressure. (See Section 5, “Suppressor Cleanup”).
- F. Replace the Dionex CES 300 if cleaning the suppressor membrane does not reduce the pressure.
- G. Contact the nearest Dionex Regional Office (see, “Dionex Regional Offices”) if you cannot solve the problem on your own.

**NOTE**

Always use the precut tubing included with the suppressor and other capillary devices to make connections. Do not cut or alter this tubing, or add other tubing other than Dionex pre-cut tubing; the tubing is factory cut for an extremely low dead volume interface.

4.8. Liquid Leaks

If there is leakage from the Dionex CES 300, check the back pressure after the suppressor.

If the system back pressure after the suppressor is greater than 150 psi, the leaks are caused by excessive back pressure downstream from the Dionex CES 300. Find and eliminate the source of the pressure. If the Dionex CES 300 continues to leak when operated within the proper back pressure range, it must be replaced. The Dionex CES 300 will not usually recover from overpressure conditions, so ensure the source of backpressure is eliminated before installing a replacement Dionex CES 300.



CAUTION

The correct amount of back pressure for optimum operation is 40 psi. Back pressures over 150 psi after the Dionex CES 300 can cause irreversible damage.

4.9. Poor or unstable recovery of certain peaks

If one or two peaks are experiencing poor or unstable recoveries while the other peaks are stable, it could be that the current to the suppressor is set too high. It is recommended to use the recommended current (see Section 2.4) unless necessary to stabilize a drifting baseline.

4.10. Peaks and spikes in the absence of an injection

- A. Precipitation on the suppressor membrane or screens (calcium, magnesium and other metals): Follow the Metal Contaminants or Precipitates procedure from section 5.1. To prevent contaminants from reaching the suppressor, a CP1 cation polisher column (P/N 064930) can be used during anion analysis to strip cationic contaminants from the sample. Refer to the CP1 Operator's Manual for detailed instructions.
- B. Outgassing or trapped bubbles in the suppressor regenerant chambers: Ensure that the external water (if used) is degassed before use; do not pressurize the external water with air, use nitrogen or helium. Eliminate causes of excessive backpressure between the cell outlet and the regenerant inlet if recycled eluent mode is used.
- C. Excess suppressor temperature: It is important to ensure that the Dionex CES 300 is operated in an environment of 15°C. Operating the Dionex CES 300 at a higher temperature may compromise its performance.
- D. Large changes to the flow rate or applied current of the suppressor. If the current is changed, allow the suppressor a few hours to reestablish baseline stability.
- E. Operating the suppressor with power but no eluent or regenerant flow: Operation of the suppressor without flow may irreversibly damage the suppressor, depending on amount of applied current and duration. Ensure that the regenerant lines are connected and flow is established when powering on the suppressor at all times. Ensure that during external water operation that the suppressor is never operated without regenerant flow. Replace the suppressor if operation without flow is known to have occurred. Confirm that the minimum pressure limit on the pump is set to a non-zero value to ensure that the system turns off in the event of a leak.

SECTION 5 – SUPPRESSOR CLEAN-UP

This section describes routine cleanup procedures for the Capillary Electrolytic Suppressor (Dionex CES 300) in the case of contamination. Consult the Troubleshooting Guide (see Section 4, “Troubleshooting Guide”) to first determine that the system is operating properly. If the Dionex CES 300 is determined to be the source of higher than normal back pressure, higher than anticipated conductivity, decreased suppression capacity or decreased sensitivity, cleaning the membrane may restore the performance of the system. Use the following procedures to clean the membrane.

5.1. Metal Contaminants or Precipitates

Note: The suppressor voltage is a good indicator of the resistance across the suppressor. Higher resistance may indicate contamination of the suppressor.

- A. Turn off the pump and Dionex CES current.
- B. Disconnect the analytical (and guard) column(s) from the injection valve and the Dionex CES 300. Refer to the specific analytical column Product Manual for column cleanup procedures.
- C. If you are running in the AutoSuppression External Water Mode, turn off the external water and disconnect the external water line from the Dionex CES 300 REGEN IN port.
- D. Disconnect the liquid line from the Dionex CES 300 ELUENT OUT port to the cell or Dionex CRD 200 at the cell fitting and reconnect it to the REGEN IN port.
- E. If iron is not present then proceed directly to step F. If iron is present prepare a solution of 0.2 M oxalic acid. Pump this solution through the Dionex CES 300 at 0.010 – 0.030 mL/min for 30 minutes.
 - a. Do not use the Analytical pump for the clean-up purpose as this will contaminate the pump; use an auxiliary pump or pressurized container to deliver the cleaning solution.
- F. Regenerate the suppressor as outlined in 4.2.
 - a. It is recommended to allow the regenerant solution to soak in the suppressor overnight before rinsing with deionized water.
- G. Reinstall the analytical (and guard) column(s). Begin pumping eluent through the system at the flow rate required for your analysis and equilibrate the system and resume normal operation.