PRODUCT MANUAL

Carbonate Removal Device
CRD 300

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PRODUCT MANUAL

FOR

CARBONATE REMOVAL DEVICE 300

CRD 300 (4 mm) (P/N 064637)
CRD 300 (2 mm) (P/N 064638)

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1. INTRODUCTION

The Carbonate Removal Device 200 and 300 (CRD 200 and CRD 300) are membrane-based modules that transport CO_2 from the eluent stream into the outer regenerant waste stream for removal (Figure 1). Typically the CRD 200 or CRD 300 is installed between the suppressor and the detector cell (e.g. conductivity cell). For the CRD 200 this placement reduces the carbonate peak contributed by the sample during anion analysis by suppressed anion chromatography with hydroxide eluents. For the CRD 300 this placement reduces the background conductivity contributed by the suppressed carbonate eluent when used with carbonate or carbonate / bicarbonate eluents.

CRD 200 Recommendation

For optimal performance, Dionex recommends using the CRD 200 with a Reagent-Free Ion Chromatography (RFIC™) system comprised of a Dionex Ion Chromatograph (IC) system equipped with an Eluent Generator (EluGen®), Continuously Regenerated Anion Trap Column (CR-ATC), and a hydroxide eluent compatible column (such as IonPac AS11, AS15, AS16, AS17, AS18, AS19, AS20, or AS21). The CRD 200 can be used with RFIC systems pursuing borate chemistry with AS4A, AS14, or AS14A chemistries. The CRD 200 can also be used under vacuum mode of operation for the removal of ammonia from ammoniated waters. For CRD 200 installation, refer to the CRD 200 manual.

CRD 300 Recommendation

For optimal performance, Dionex recommends using the CRD 300 with a system comprised of a Dionex Ion Chromatograph (IC) system equipped with a carbonate eluent compatible column (such as IonPac AS4A-SC, AS9-SC, AS9-HC, AS12A, AS14, AS14A, AS22, or AS23).

FIGURE 1
CRD 300 Basic Plumbing

Assistance is available for any problem during the shipment or operation of Dionex instrumentation, columns, and consumables 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.

1.1. CRD 300 Design and Operation

The CRD 300 consists of a gas permeable membrane with a silicone coating that is selective to CO_2 (Figure 2). In a typical system the CRD 300 is plumbed between the suppressor module and the cell. In the CRD chemical regeneration mode the regenerant channel that encloses the CRD 300 membrane is flushed with a solution of 200 mM NaOH which aids removal of the CO_2 as carbonate. In the vacuum regeneration mode the regenerant channel is evacuated with a vacuum pump, a bleed tube is used to bleed a small amount of air into the regenerant channel to aid the removal of gaseous carbon dioxide.

Suppression of the carbonate or carbonate/bicarbonate eluent results in conversion to carbonic acid. Carbonic acid is in equilibrium with carbon dioxide gas dissolved in water. The CRD 300 removes the carbonic acid as CO_2 from the suppressed eluent.

$$\mathbf{H_2CO_3} \quad \Longleftrightarrow \quad \mathbf{H_2O + CO_2}$$

EQUATION 1
CARBONIC ACID EQUILIBRIUM

As the CO_2 is removed by the CRD 300 membrane, the equilibrium shifts to the right, thereby facilitating further removal of the CO_2 and reducing the net carbonic acid concentration. Additionally, in CRD chemical regeneration mode the basic environment in the regenerant channel allows quick conversion of the removed CO_2 to carbonate anion. In vacuum regeneration mode the reduced partial pressure of carbon dioxide facilitates fast removal of CO_2. The net result of the above steps is a reduction of the background conductivity resulting from carbonic acid.

The CRD 300 can remove high levels of carbonic acid from the eluent stream as well as the peak/dip resulting from the void and the CO_2/carbonate in the sample. It should be noted that high levels of CO_2/carbonate in the sample may affect chromatographic peak shapes and recovery.

Table 1
Typical Background Conductivity under Different Carbonate Concentrations

Carbonate Concentration	Typical Background Conductivity
9.0 mM	< 2.0 µS/cm
15.0 mM	< 3.0 µS/cm

The typical background conductivity under different carbonate concentrations is shown in Table 1. The background can be lower if lower concentration of carbonate is used as the eluent.

If a chemical suppressor is used, higher background conductivity may be encountered, due to the leakage of the regenerant.

Regeneration Recommendation

The base used for CO_2 removal needs to be replaced every month or if the background conductivity begins to drift to unacceptable levels.

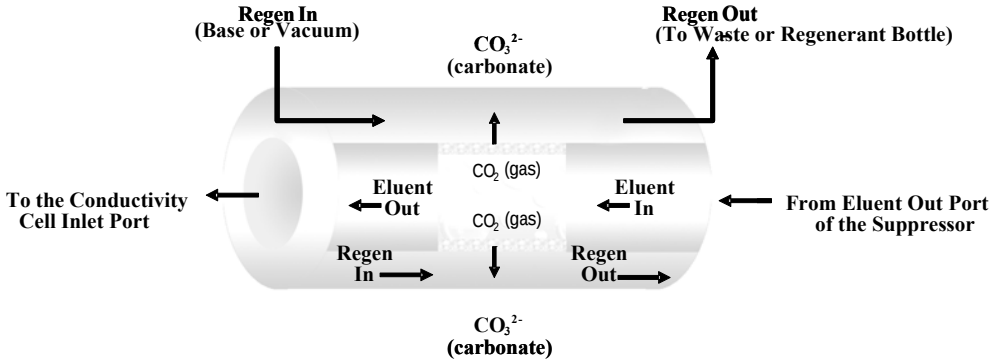


FIGURE 2
CRD 300 Design and Operation

1.2. CRD 300 Configuration

The CRD 300 is available in both 2-mm and 4-mm formats for use with 2, 3, 4, and 5-mm Ion chromatography columns. The 4-mm CRD 300 has a typical delay volume of 225 µL and is recommended for 4 or 5-mm column applications and for installation between the sampler and injection valve. The 2-mm CRD 300 has a typical delay volume of 80 µL and is recommended for 2 or 3-mm column applications, it is not recommended for installation between the sampler and injection valve.

2. GETTING STARTED

The CRD 300 must be handled with care to ensure proper operation. Fittings only need to be finger tightened.

WARNING

Do not attempt to disassemble the CRD 300; it may result in irreversible damage.

2.1. CRD 300 Quickstart

2.1.1. Hydrating the CRD 300

Step 1.

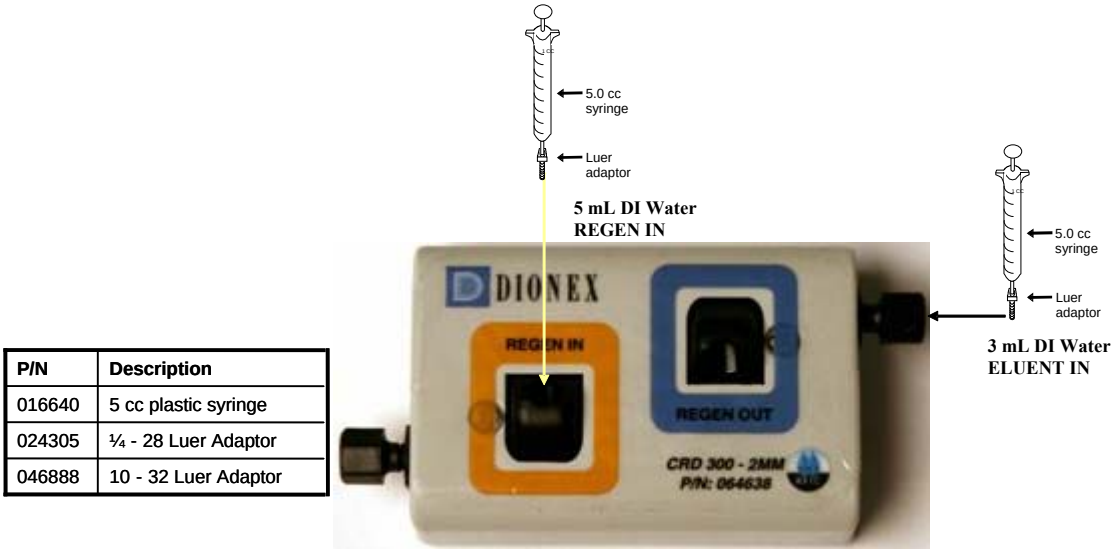
Using a 5 cc disposable plastic syringe (P/N 016640) and the 10-32 Luer adaptor (P/N 046888), push approximately 3 mL of degassed DI water through the ELUENT IN port. Using a 5 cc disposable plastic syringe (P/N 016640) and the 1/4-28 Luer adaptor (P/N 024305), push 5 mL of degassed DI water through the REGEN IN port (Figure 3).

NOTE

Step 1 can be accomplished by installing the CRD 300 in the system and connecting the ELUENT OUT port to the REGEN IN port on the CRD by using suitable tubing and pumping 5 mL of deionized water through the CRD ELUENT IN port. In the above step, it is recommended to bypass the guard and analytical columns.

Step 2.

Allow the CRD 300 to sit for approximately 10 minutes to fully hydrate the CRD 300 membrane.



P/N	Description
016640	5 cc plastic syringe
024305	¼ - 28 Luer Adaptor
046888	10 - 32 Luer Adaptor

FIGURE 3
Hydrating the CRD 300

2.1.2. Backpressure Instructions

The total backpressure to the suppressor eluent channel should be less than 60 psi. This includes the CRD 300, the cell, and the backpressure coil. Trim the backpressure coil if required to achieve <60 psi total backpressure. Refer to Section 3.4 for backpressure measurement instructions.

WARNING

Total backpressure amounts exceeding >100 psi may cause irreversible damage to the CRD 300.

3. INSTALLATION

3.1. Installing the CRD 300 between the suppressor and the detection cell

NOTE

Always minimize tubing lengths for obtaining efficient peaks. For 4-mm or 5-mm applications (standard bore format) use 0.010" ID x 1/16" OD Black tubing. For 2-mm applications (microbore format) or 3-mm applications, use 0.005" ID x 1/16" OD Red tubing.

WARNING

Never operate the CRD 300 with an SRS ULTRA II, MMS III or earlier suppressor. The CRD 300 is intended for operation with the SRS 300, MMS 300 or later suppressors.

1. The CRD 300 is installed on the top of the SRS module. The CRD 300 is shipped with self-adhesive Velcro tape that is used to attach the device to the SRS module.

2. To install, remove the suppressor from the Chromatography Module and remove all connections to the suppressor with the possible exception of the ELUENT IN line coming from the column.

3. Remove the backing tape from the self-adhesive Velcro on CRD 300 (Figure 4) and push it firmly into place on the suppressor as shown in Figure 5. The CRD 300 can be easily removed or reinstalled by simply pulling the device on and off the SRS.

FIGURE 4
Velcro tape on rear of CRD 300

FIGURE 5
CRD 300 Mounted on SRS 300.

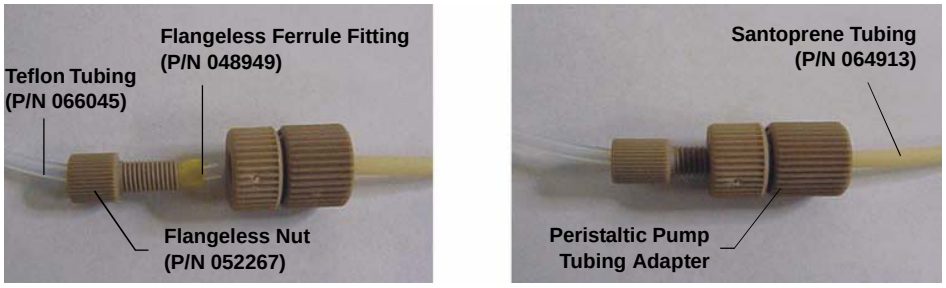
3.2. Plumbing the CRD 300 between the suppressor and the detection cell with CRD chemical regeneration

NOTE

For 4-mm or 5-mm applications (standard bore format) use 0.010" ID x 1/16" OD Black tubing. For 2-mm or 3-mm applications (microbore format) use 0.005" ID x 1/16" OD Red tubing. Connecting the wrong tubing dimensions for a given application may damage the CRD 300 and suppressor modules.

1. The CRD 300 is installed on the top of the SRS module as shown in Figure 5.
2. To begin plumbing of the CRD 300, refer to the plumbing schematic as shown in Figure 6.
   a) Turn off the pump and the Self-Regenerating Suppressor (SRS) before making any connections.
   b) Remove the plugs from the CRD 300 ports.
   c) Connect an approximately 2 inch length of 1/16" OD Black (4mm application) or Red (2mm application) tubing from the ELUENT OUT port of the suppressor to the ELUENT IN port of the CRD 300 (Red label).
   d) Connect an approximately 6 inch length of 1/16" OD Black or Red tubing from the conductivity cell inlet port to the ELUENT OUT port of the CRD 300 (Yellow label).
   e) Assemble the tubing adapter for the peristaltic pump, using the following figures.

Installing the Peristaltic Pump Tubing Adapter



View A **View B**

Connecting the Teflon Liquid Lines

   f) Using the Peristaltic Tubing Adapters connect two pieces of 1/8" OD tubing (opaque) to the peristaltic inlet and outlet.

g) Connect the tubing from the peristaltic pump outlet to the CRD 300 Regen In port. (CRD 300 in photos has been disconnected and removed from suppressor for clarity).

h) Connect the tubing from the peristaltic pump inlet to the regenerant bottle outlet.

i) Using another piece of 1/8" OD tubing (opaque), connect the CRD 300 Regen Out to the eluent bottle inlet.
j) Reinstall the suppressor and the CRD 300 into the chromatography module.
k) Fill the regenerant bottle with 200 mM NaOH and turn on the pump by choosing the flow direction. Adjust the flow control knob to achieve 1-2 ml/min flow. The flow direction would be from the regenerant bottle to the CRD 300 (from Regen in port) and (out from Regen out port) to the bottle. If needed flip the flow direction switch to achieve the above flow direction.
l) CRD 300 installation is now complete. Before using the CRD 300 for analysis, proceed to Section 3.4, "CRD 300 Backpressure Measurement".

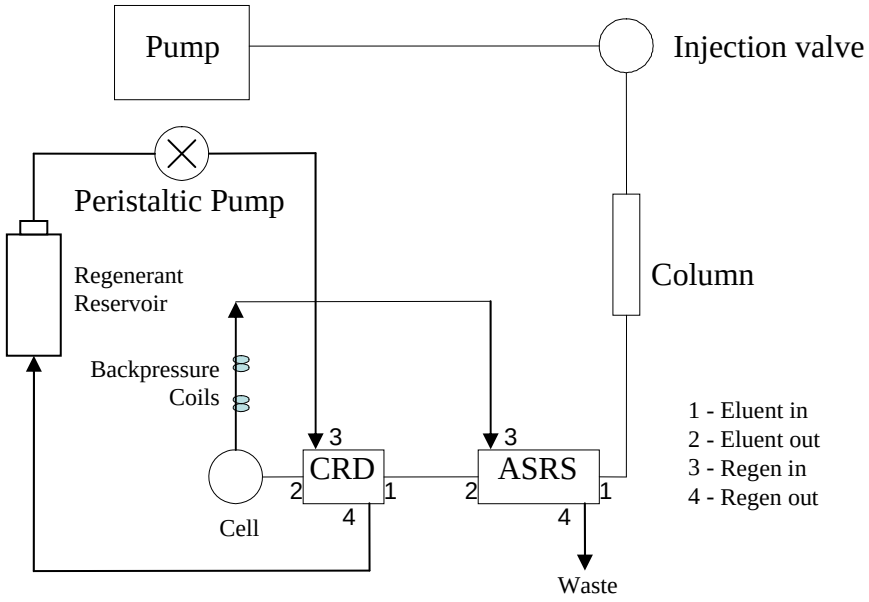
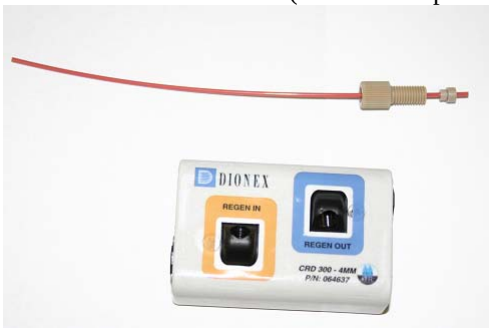


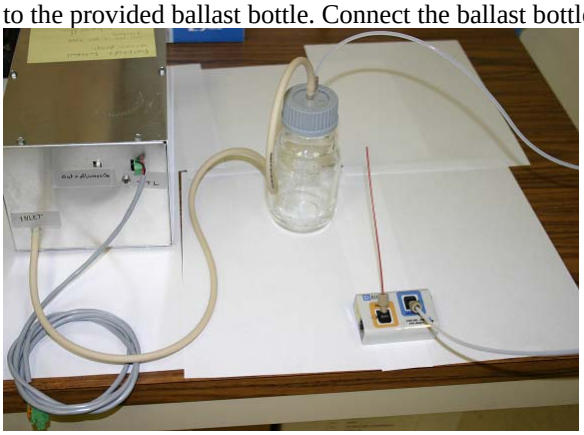
FIGURE 6
CRD 300 Chemical Regeneration Plumbing Schematic

3.3. Plumbing the CRD 300 between the suppressor and the detection cell with vacuum regeneration

1. Install the CRD 300 as described in section 3.1 and 3.2 a) through d).
2. Connect a 6” (15 cm) piece of 0.005” I.D. PEEK tubing (red) to the Regen Out port of the CRD 300. This is the bleed line for the vacuum. (CRD 300 in photos has been disconnected and removed from suppressor for clarity).



3. Connect about 18” (50 cm) of 1/8” I.D. (opaque) tubing to the Regen In port of the CRD 300, and the other end to the provided ballast bottle. Connect the ballast bottle to the vacuum source.



WARNING

It is important that vacuum is not applied to the CRD 300 until eluent flow has been established. Always turn on the eluent pump first, and then the vacuum pump. The vacuum pump provided by Dionex has a TTL control that allows it to be controlled by Chromeleon. Refer to the vacuum pump manual (Document Number 065186) for more details.

3.4. CRD 300 Backpressure Measurement

The backpressure to the suppressor module needs to be measured and adjusted if needed. This will ensure optimal performance of the system. Failure to complete the following steps may result in a leaking suppressor and/or CRD 300.

a) After installing the CRD 300, measure the total system pressure (P1).
b) Remove the line from the ELUENT OUT port of the suppressor and measure the system pressure (P2).

$$P1 - P2 = P3 \leq 60$$

c) If P3 is higher than 60 psi, then trim the backpressure coil (line out of the conductivity cell) to achieve a total pressure of less than 60 psi.

WARNING ***Total backpressure exceeding >100 psi may cause irreversible damage to the CRD 300 and/or suppressor.***

NOTE ***Always use 1/8" tubing when connecting lines for the regenerant side of the Self – Regenerating Suppressor (SRS) and Carbonate Removal Device 300 (CRD 300).***

4. APPLICATIONS

The Carbonate Removal Device (CRD 300) has been optimized for reducing the baseline resulting from carbonic acid in ion chromatography systems for anion analysis using carbonate and carbonate/bicarbonate eluents.

4.1. Recommended Applications

The CRD 300 is recommended for both routine and trace level work with carbonate and carbonate/bicarbonate eluents. Reducing carbonic acid using the CRD 300 leads to improved peak sensitivity and quantification for analytes such as fluoride, acetate and formate in carbonate and carbonate/bicarbonate systems. The CRD 300 is particularly useful for analyzing ultra pure water when large-volume injections or preconcentration techniques result in large void volumes. The CRD 300 also improves sensitivity.

Some applications may not benefit from the CRD 300 module due to the following reasons.

a) The peaks of interest are large and well separated.
b) The void dip does not interfere with peaks of interest.

Under these conditions, there may be no need for a CRD 300 device.

WARNING ***The CRD 300 is designed to perform optimally with carbonate and carbonate/bicarbonate eluents only. It is not recommended for borate, hydroxide, or cation applications as it adds significant delay volume and leads to band dispersion.***

NOTE ***The CRD 300 will remove CO_2 from the carbonate background thus minimizing the void dip, but complete removal of CO_2 may not occur.***

NOTE ***Due to the added delay volume of the device, the early eluting peaks may show a decline in peak efficiency and all peaks will show a small change in retention time.***

5. OPERATION

5.1. Example Chromatogram

5.1.1. Carbonate or carbonate/bicarbonate are commonly used as eluents for the ion chromatography analysis. The example below (see Figure 7) shows the chromatograms obtained with and without CRD 300 inline. The CRD 300 is useful in reducing the background conductivity, minimizing the void, and increasing the analyte peak height and area.

Column:	IonPac® AS9-HC 4 x 250 mm	
Flow Rate:	1.0 mL/min	
Eluent:	9.0 mM Carbonate	
Suppressor:	ASRS® ULTRA II, 4 mm	
Current::	50 mA (Recycle)	
Loop:	25 µL	
Oven:	30 °C	
		Concentration
Peak:	1. Fluoride	1.0 mg/L
	2. Chloride	1.5 mg/L
	3. Nitrate	5.0 mg/L
	4. Phosphate	7.5 mg/L
	5. Sulfate	7.5 mg/L
Background:	No CRD 300	23.0 µS/cm
	with CRD 300	0.87 µS/cm

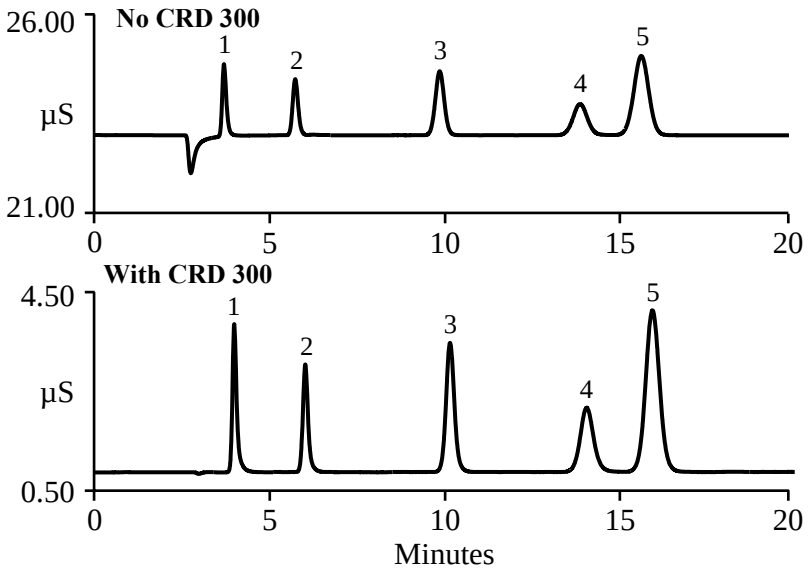


FIGURE 7
Removing CO_2 from Suppressed Carbonate Eluent

6. TROUBLESHOOTING

WARNING

High backpressure may cause irreversible damage to the CRD 300 and suppressors. The total pressure from the cell, the CRD 300, and the backpressure coil should not exceed 100 psi.

6.1. Leakage Due to High Backpressure

a) High backpressure will cause irreversible damage to the CRD 300.
b) Measure the backpressure to the suppressor (i.e. cell + CRD 300 + the backpressure coil) following the instructions in Section 3.4. The backpressure should not exceed 100 psi. Trim the backpressure coil, if needed.
c) Check the waste lines and ensure these do not contribute to the overall backpressure. Replace and trim tubings if needed.
d) When operating with a pump, ensure that the external regenerant flow is 1 – 2 mL/min for 2-mm and 3-mm applications and 3 - 5 mL/min for 4-mm and 5-mm applications. Lower the external water flow rate if recommended rates were exceeded.

6.2. Poor CO_2 Removal Efficiency

a) Ensure that the CRD 300 is installed correctly with regenerant (base) flowing into the regenerant channel or air bleeding through the regenerant channel to the vacuum pump.
b) Check for leaks and reconnect.

6.3. No Peaks

a) Ensure that the CRD 300 is connected as outlined in the plumbing instructions.
b) Check for leaks and reconnect.
c) CRD 300 may have an internal leak due to damage; replace the CRD 300.

6.4. High Noise in the Baseline and on Top of the Peaks

a) Release any trapped gases in the cell, suppressor, or the CRD 300 by systematically opening the lines from:
   - ELUENT OUT port of the suppressor.
   - The ELUENT IN and ELUENT OUT ports of the CRD 300.
   - The cell outlet port.

6.5. Negative Dips Before and/or After All the Analyte Peaks

a) The tubing leading into or exiting the CRD 300 may not be cut straight. Recut the ends of the tubing ensuring the cut is perpendicular to the tubing and the finish is smooth and flat.
b) Backpressure on CRD 300 is too high. Trim or remove backpressure coils to reduce backpressure.
c) The CRD 300 may have an internal leak due to damage. Replace the CRD 300 if a leak is detected. Ensure the suppressor is fully regenerated by pursuing a sulfuric acid treatment.
d) Suppressor may not be fully regenerated. Chemically regenerate the suppressor according to the instructions given in the suppressor user's guide.

6.6. How to Check for CRD 300 Leaks

a) Visually inspect for liquid drops or leakage at the CRD 300 eluent and regen ports. Reconnect the lines and tighten the fittings if needed. Do not overtighten as damage may occur.
b) Disconnect the lines from the regen ports of the CRD 300 and, with the eluent flowing in the eluent channel of the CRD 300, observe for fluid flow out of the regen channel. A continuous flow of liquid from the regen ports of the CRD 300 indicates an internal leak. Replace the CRD 300.

Assistance is available for any problem during the shipment or operation of Dionex instrumentation, columns, and consumables 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.

