



Thermo Scientific

Dionex CRD Carbonate Removal Device

Product Manual

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Product Manual

for

Dionex CRD Carbonate Removal Device 180

CRD 180, 0.4 mm (P/N 079960)

Dionex CRD Carbonate Removal Device 200

CRD 200, 4 mm (P/N 062983)

CRD 200, 2 mm (P/N 062986)

CRD 200, 0.4 mm (P/N 072054)

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Revision 06, June, 2013, Rebranded for Thermo Scientific. Added CRD 180.

Safety and Special Notices

Make sure you follow the precautionary statements presented in this guide. The safety and other special notices appear in boxes.

Safety and special notices include the following:



SAFETY

Indicates a potentially hazardous situation which, if not avoided, could result in death or serious injury.



WARNING

Indicates a potentially hazardous situation which, if not avoided, could result in damage to equipment.



CAUTION

Indicates a potentially hazardous situation which, if not avoided, may result in minor or moderate injury. Also used to identify a situation or practice that may seriously damage the instrument, but will not cause injury.



NOTE

Indicates information of general interest.

IMPORTANT

Highlights information necessary to prevent damage to software, loss of data, or invalid test results; or might contain information that is critical for optimal performance of the system.

Tip

Highlights helpful information that can make a task easier.

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1. Introduction

The Thermo Scientific™ Dionex™ Carbonate Removal Device 180 and 200 (Dionex CRD™ 180 and CRD 200) is a membrane-based module that transports CO₂ from the suppressed eluent stream into the outer regenerant waste stream for removal (Figure 1). Typically the Dionex CRD 180 and CRD 200 is installed between the suppressor and the detector cell (e.g. conductivity cell). This placement reduces the carbonate peak contributed by the sample during anion analysis by suppressed anion chromatography with hydroxide eluents. Alternatively the Dionex CRD 180 and CRD 200 can be installed between the autosampler and injection valve when the sample is acidic, e.g. carbonated beverages. This placement reduces the carbonate peak contributed by the sample and eliminates out gassing of CO₂.

**NOTE**

The Dionex CRD 180 and CRD 200 (Capillary) are not compatible with placement between the autosampler and injection valve. The Dionex CRD 200 (4 mm) is recommended.

For optimal performance, Thermo Scientific recommends using the Dionex CRD 180 and CRD 200 with a Reagent-Free Ion Chromatography (RFIC™) system comprised of a Dionex Ion Chromatograph (IC) system equipped with an Eluent Generator Cartridge (EGC), Continuously Regenerated Anion Trap Column (CR-ATC), and a hydroxide eluent compatible column (such as IonSwift MAX-100, IonPac AS11, AS15, AS16, AS17, AS18, AS19, AS20, or AS21). The Dionex CRD 200 can also be used with RFIC systems pursuing borate chemistry with AS4A, AS14, or AS14A chemistries. The Dionex CRD 200 can also be operated under a vacuum mode of operation and this is recommended for samples such as ammoniated waters.

Figure 1 Dionex CRD 200 Basic Plumbing

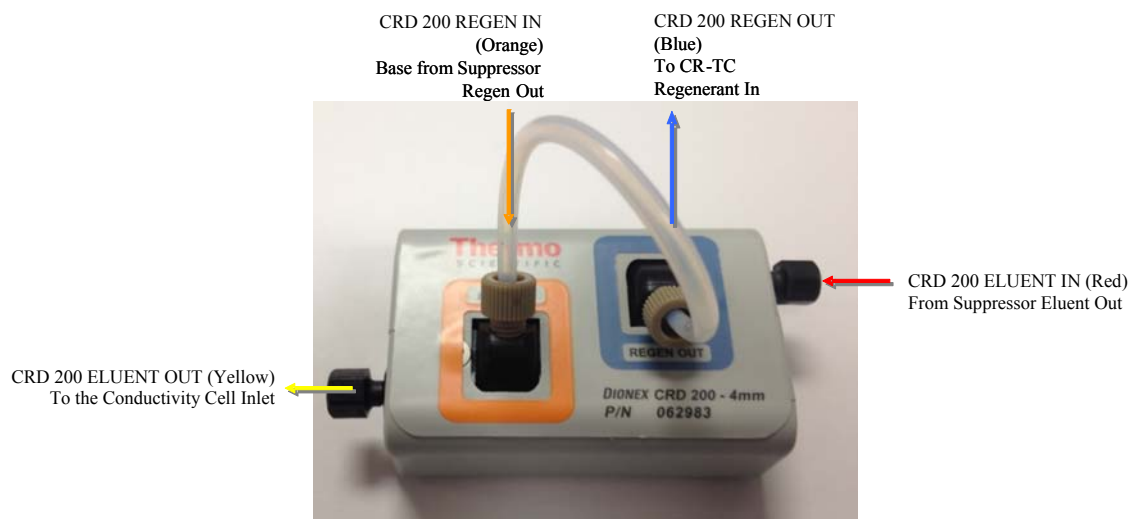
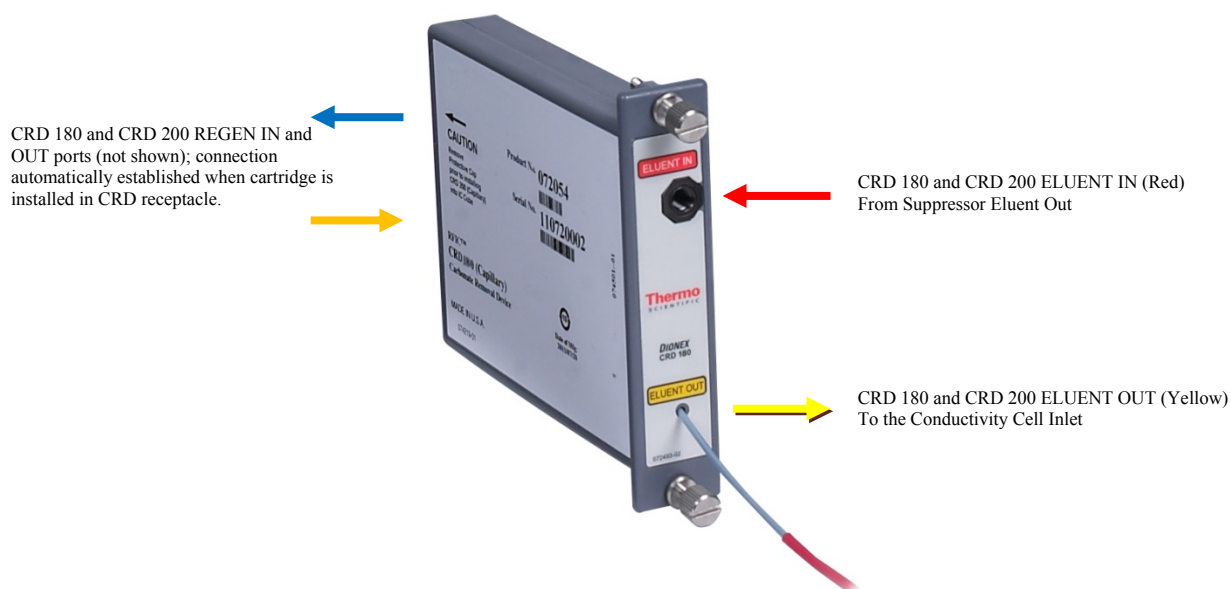


Figure 2 Dionex CRD 180 and CRD 200 (Capillary) Basic Plumbing



1.1 Dionex CRD 180 and CRD 200 Design and Operation

The Dionex CRD 180 and CRD 200 consist of a gas permeable membrane with a silicone coating that is selective to CO₂ (Figure 2). In a typical system the Dionex CRD 180 or CRD 200 is plumbed between the suppressor module and the detector cell. Alternatively the Dionex CRD 200 can be installed between the autosampler and injection valve. The regenerant channel that encloses the Dionex CRD 180 and CRD 200 membrane is flushed with the suppressor waste (base) and aids removal of the CO₂ as carbonate.

Suppression of the carbonate peak results in conversion to carbonic acid. Carbonic acid is in equilibrium with carbon dioxide gas dissolved in water. The Dionex CRD 180 and CRD 200 remove the carbonate peak as CO₂ from the suppressed eluent.

Equation 1 Carbonic acid-Carbon dioxide Equilibrium



As the CO₂ peak is removed by the Dionex CRD 180 or CRD 200 membrane the equilibrium shifts to the right, thereby facilitating further removal of the CO₂ and reducing the net carbonic acid concentration. Additionally, the basic environment in the regenerant channel allows a quick conversion of the removed CO₂ to carbonate anion. The net result of the above steps is a reduction of the peak resulting from carbonate.

The Dionex CRD 200 can remove high levels of the peak resulting from carbonate; >1000 mg/L of carbonate with >90% apparent removal efficiency (Equation 2). The Dionex CRD 180 has less capacity for the removal of carbonate with >80% apparent removal efficiency. When plumbed between the suppressor and the cell it should be noted that high levels of CO₂/carbonate in the sample may affect chromatographic peak shapes and recovery. When plumbed between the autosampler and injection valve, the sample must be acidic in order to facilitate efficient carbonic acid removal.

Equation 2 Apparent Removal Efficiency

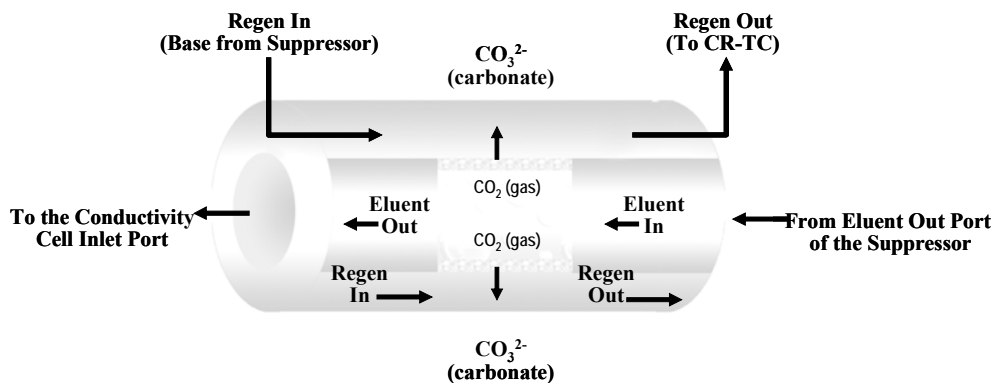
$$\text{Apparent \% Removal Efficiency} = 100 - \left[\frac{\text{Response}_{w/CRD}}{\text{Response}_{w/oCRD}} \times 100 \right]$$

Response_{w/CRD} = Peak area or height with Dionex CRD 180 or CRD 200 installed

Response_{w/oCRD} = Peak area or height without Dionex CRD 180 or CRD 200 installed

It should be noted that the true CO₂ removal efficiency is based on the chemical equilibrium outlined in (Equation 1). In (Equation 2) it shows underestimating this removal efficiency and therefore is the apparent removal efficiency. It is included here for reference purpose only. For example, using a known level of carbonate for a peak height of 23 μS/cm when the peak is reduced to 2.3 μS/cm, the true removal efficiency based on the chemical equilibrium described in (Equation 1) is calculated as 99% whereas (Equation 2) calculates this removal efficiency as 90%.

Figure 3 Dionex CRD 180 and CRD 200 Design and Operation



1.2 Dionex CRD 200 Configuration

The Dionex CRD 200 is available in capillary (0.4 mm), 2 mm and 4 mm formats for use with 0.25, 0.4, 2, 3, 4, and 5 mm Ion chromatography columns. The 4 mm Dionex CRD 200 has a typical delay volume of 200 μL and is recommended for 4 or 5 mm column applications and for installation between the autosampler and injection valve. The 2 mm CRD 200 has a typical delay volume of 40 μL and is recommended for 2 or 3 mm column applications, it is not recommended for installation between the autosampler and injection valve. The capillary Dionex CRD 200 has a typical delay volume of 2 μL and is recommended for 0.25 or 0.4 mm capillary column applications; it is not recommended for installation between the autosampler and injection valve.

1.3 Dionex CRD 180 Configuration

The Dionex CRD 180 is only available in capillary (0.4 mm) format for use with 0.25 and 0.4 mm Ion chromatography columns. The CRD 180 has a typical delay volume of 1 μL and is recommended for 0.25 or 0.4 mm capillary column applications; it is not recommended for installation between the autosampler and injection valve. The Dionex CRD 180 is recommended for use with columns using 4 μm bead sizes; it is not recommended for use with columns using bead sizes greater than 4 μm .

2. Getting Started

The Dionex CRD 180 and CRD 200 must be handled with care to ensure proper operation. Fittings only need to be finger tightened.



WARNING

Do not attempt to disassemble the Dionex CRD 180 or CRD 200; it may result in irreversible damage.

2.1 Dionex CRD 180 and CRD 200 Quick Start

2.1.1 Hydrating the Dionex CRD 200 (4 mm and 2 mm versions)

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), Remove the storage tubing from the Regen OUT port and push 5 mL of degassed DI water through the REGEN OUT port (Figure 4).



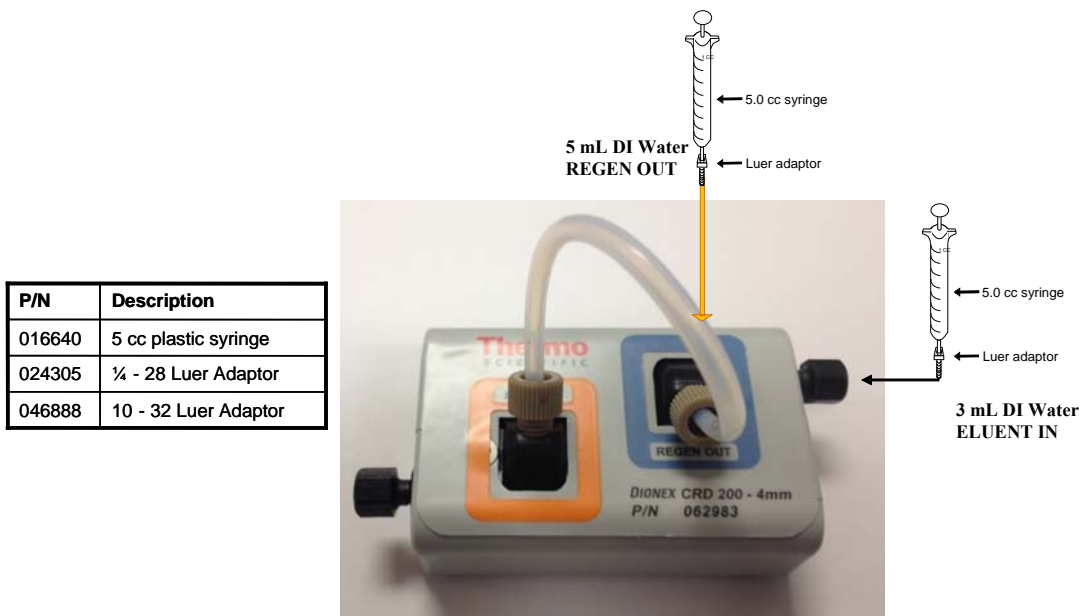
NOTE

Step 1 can be accomplished by installing the Dionex CRD 200 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 Dionex CRD 200 to sit for approximately 10 minutes to fully hydrate the Dionex CRD 200 membrane.

Figure 4 Hydrating the Dionex CRD 200



2.1.2 Hydrating the Dionex CRD 180 or CRD 200 (Capillary)

Step 1.

Using a 1 cc disposable syringe (P/N 016640) and a 10-32 Luer adapter (P/N 046888), very slowly push approximately 0.3 mL of degassed DI water through the ELUENT IN port. The entire process should take no less than one minute.

Step 2.

Install the Dionex CRD 180 or CRD 200 in the Capillary IC System.

1. Remove the plugs from the REGEN ports at the rear of the Dionex CRD 180 or CRD 200 cartridge.
2. If a dummy cartridge or used Dionex CRD 180 or CRD 200 (capillary) is installed in the CRD receptacle, loosen the thumb screws of the dummy cartridge or used device and pull it out.
3. Plug the new Dionex CRD 180 or CRD 200 into the CRD receptacle so the regenerant ports at the rear of the cartridge seat firmly.
4. Tighten the thumb screws to ensure a firm fit.



NOTE

Remove the plugs from the REGEN ports at the rear of the Dionex CRD 180 or CRD 200 before inserting the cartridge into the receptacle. Failure to remove the plugs may cause damage to the Capillary IC System.



NOTE

Do not attempt to plug the Dionex CRD 180 or CRD 200 (Capillary) into any receptacle other than the CRD receptacle. See the system manual for details.

Step 3.

Ensure the suppressor and EG Degas cartridges are in place; install dummy units if these devices are not available. Using a 1 cc disposable syringe and a 10-32 Luer adapter, push approximately 2 mL of degassed DI water through the REGEN IN port of the suppressor. This will flush the regenerant chambers of the suppressor, Dionex CRD 180 or CRD 200 and EG Degas cartridges simultaneously.

Step 4.

Allow the Dionex CRD 180 or CRD 200 to sit for approximately 10 minutes to fully hydrate the membrane.

2.1.3 Backpressure Instructions

The total backpressure to the suppressor eluent channel should be less than 100 psi. This includes the Dionex CRD 180 or CRD 200, the cell, and the backpressure coil. Trim the backpressure coil if required to achieve <100 psi total backpressure. Refer to Section 3.3 for backpressure measurement instructions.



WARNING

Total backpressure amounts exceeding >100 psi may cause irreversible damage to the Dionex CRD 180 or CRD 200 and the suppressor.

3. Installation

3.1 Installing the Dionex CRD 200 (4 mm and 2 mm versions) between the Suppressor and the Detection Cell

3.1.1 Mounting the Dionex CRD 200 to the Suppressor



NOTE

Always minimize tubing lengths for obtaining efficient peaks. For 4-mm or 5-mm applications (standard bore format) use 0.010" ID x 0.062" (1/16") OD Black tubing. For 2-mm applications (microbore format) or 3-mm applications, use 0.005" ID x 0.062" (1/16") OD Red tubing.

1. The Dionex CRD 200 is installed on the top of the Dionex ERS suppressor. The Dionex CRD 200 is shipped with self-adhesive Velcro tape that is used to attach the device to the Dionex ERS suppressor.
2. To install, remove the suppressor from the Chromatography Module and remove all connections to the suppressor.
3. Remove the backing tape from the self-adhesive Velcro on the Dionex CRD 200 (Figure 5) and push it firmly into place on the suppressor as shown in (Figure 6). The Dionex CRD 200 can be easily removed or reinstalled by simply pulling the device on and off the Dionex ERS suppressor.

Figure 5 Velcro tape on rear of Dionex CRD 200



Figure 6 Dionex CRD 200 Installed on Suppressor



3.1.2 Plumbing the Dionex CRD 200 between the Suppressor and the Detection Cell



NOTE

For 4-mm or 5-mm applications (standard bore format) use 0.010" ID x 0.062" (1/16") OD Black tubing. For 2-mm or 3-mm applications (microbore format) use 0.005" ID x 0.062" (1/16") OD Red tubing. Connecting the wrong tubing dimensions for a given application may damage the CRD 200 and suppressor modules.

1. The Dionex CRD 200 is installed on the top of the Dionex ERS module as shown in (Figure 6).
2. To begin plumbing of the Dionex CRD 200, refer to the plumbing schematic as shown in (Figure 7).
 - a) Turn off the pump and the Dionex ERS suppressor before making any connections.
 - b) Remove the plugs from the Dionex CRD 200 ports.
 - c) Connect an approximately 2 inch length of 0.062" (1/16") OD Black or Red tubing from the ELUENT OUT port of the suppressor to the ELUENT IN port of the CRD 200 (red label).
 - d) Connect an approximately 6 inch length of 0.062" (1/16") OD Black or Red tubing from the conductivity cell inlet port to the ELUENT OUT port of the Dionex CRD 200 (yellow label).
 - e) Connect a short piece of 1/8" OD tubing (opaque) from the backpressure coil at the cell outlet port to the REGEN IN port of the suppressor.
 - f) Connect an approximately 5 inch length of 1/8" OD tubing (opaque) from the REGEN OUT port of the suppressor to the REGEN IN port of the Dionex CRD 200 (orange label).

3 – Installation

- g) Divert the REGEN OUT port of the Dionex CRD 200 (blue label) to the waste or use it as a regenerant stream for other modules such as continuously regenerated –trap column (Dionex CR-TC) and the Eluent generator (EG) degas assembly.
- h) Reinstall the suppressor and the Dionex CRD 200 into the chromatography module. An example of a typical installation of a Dionex CRD 200 in a chromatography module is shown in (Figure 8).
- i) The Dionex CRD 200 installation is now completed. Before using the Dionex CRD 200 for analysis, proceed to Section 3.3, “Dionex CRD 200 Backpressure Measurement”.

Figure 7 CRD 200 Plumbing Schematic

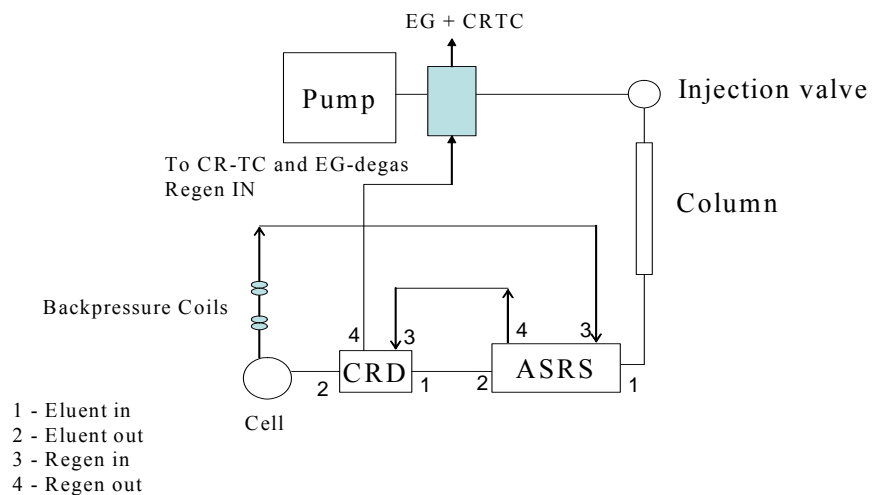


Figure 8 Dionex CRD 200 Installed in the System



3.1.3 External Water Setup

When pursuing the external water the Dionex CRD 200 is plumbed similar to what was previously described. However, in Step “e”, the fluid from the cell outlet port is diverted to waste and the suppressor REGEN IN port is plumbed to the external water source. It is recommended that the external water flow rate is

1-2 ml/min for 2 or 3-mm applications and 3-5 ml/min for 4 or 5-mm applications.

3.1.4 Chemical Regenerant Mode

If operating the suppressors in the conventional chemical regeneration mode or the Displacement Chemical Regeneration (DCR) mode the Dionex CRD 200 installation is similar to what was described previous. However, in step “e” the fluid from the cell is diverted to waste (in conventional mode) or to the DCR bottle (DCR mode). An external reservoir of 200 mM base (NaOH) is used as a regenerant in place of the suppressor effluent.

- a) Connect a 6” piece of 0.01” ID. PEEK tubing (black) to the REGEN OUT port on the Dionex CRD. Then connect a waste line using 1/8” OD (opaque) tubing and a coupler.
- b) Connect a 1/8” OD (opaque) tubing (18”) to the REGEN IN port on the Dionex CRD and then connect the other end to a reservoir containing 200mM base.
- c) Pressurize the reservoir containing 200 mM base and adjust the gas pressure to regenerate a flow of 1 to 2 ml/min through the regenerant channel of the Dionex CRD 200.

It is also possible to use a displacement approach to supply the base to the Dionex CRD 200 regenerant channel. The cell effluent will be used in this mode to displace the base from the DCR container into the Dionex CRD 200 regenerant channel.

To achieve this follow step “a” from (Section 3.1.4.) and then connect the line coming from the top of the 2 L DCR bottle (P/N 056882) to the cell out port on the cell. The line coming from the bottom of the DCR bottle should be connected to the REGEN IN port on the Dionex CRD 200. The DCR bottle should be completely filled to the top with 200mM base. In the above setup the cell effluent displaces the DCR bottle hence the contents of the DCR bottle should be replenished when the eluent container is replenished. For further information on DCR operation refer to the DCR manual.

3.1.5 Installing the Dionex CRD 200 in the Vacuum Mode of Operation

The Dionex CRD can also be operated in the vacuum mode to support specific applications such as analysis of anions in an ammoniated water sample. Operation using the base regenerant is not recommended for these samples since ammonia out gases in a basic environment and can cause anomalous baseline shifts.

To install the Dionex CRD 200 in the vacuum mode a continuous source of vacuum is needed that would generate approximately 65mm of Hg. Install the Dionex CRD 200 as described before. However, in step “e”, the fluid from the cell is diverted to waste.

- a) Connect a 6” piece of 0.005” ID. PEEK tubing (red) to the REGEN OUT port on the Dionex CRD. This is a bleed line for the vacuum.
- b) Connect a 1/8” OD (opaque) tubing (18”) to the REGEN IN port on the CRD and then connect the other end of the tubing to a vacuum source.
- c) Apply a vacuum to the regenerant channel to aid removal of volatile components such as CO₂ and NH₃.

3.1.6 Installing and Plumbing the Dionex CRD 200 (4 mm) between a Dionex AS-DV or Dionex AS40 Autosampler and Injection Valve

1. The Dionex CRD 200 may be installed on the top of the Dionex ERS module as in (Section 3.1.); alternatively the Dionex CRD 200 may be installed elsewhere in the system. The included self-adhesive Velcro can be used to attach the Dionex CRD 200 to any convenient solid surface.
2. Replace the PEEK injection tubing on the bleed valve of the Dionex AS40 auto sampler with a ~50 cm (18 in.) piece of (orange) (0.51 mm or 0.020 in. i.d.) tubing. Install the free end into the “Eluent In” port of the Dionex CRD 200.
3. Cut another ~20 cm (8 in.) piece of orange PEEK and install one end in the “Eluent Out” port of the Dionex CRD 200 and the other end into Port 5 of the injection valve.
4. Connect the regenerant waste line of the EGC degas module to the “Regen In” port of the Dionex CRD 200.
5. Connect another length of (1.6 mm or 0.063 in. i.d.) Teflon to the “Regen Out” port of the Dionex CRD 200 and direct the other end to waste.



WARNING

It is important that the flow rate of the sample through the Dionex CRD 200 does not exceed 2.0 mL/min. On a Dionex AS40 this is achieved by selecting the “Concentrate” mode. See the Dionex AS40 Operators Manual for more information.



NOTE

Application Update 153 “Fast Determinations of Phosphate and Citrate In Carbonated Beverages Using On-Line Degassing with the Carbonate Removal Device (CRD) and a Reagent-Free Ion Chromatography System” has more details on operating the Dionex CRD 200 in this mode.

3.2 Installing the Dionex CRD 180 or CRD 200 (Capillary) between the suppressor and conductivity cell.

3.2.1 Mounting the Dionex CRD 180 or CRD 200 (Capillary) in the IC System.

The Dionex CRD 180 and CRD 200 (Capillary) are a cartridge design and are plugged into their receptacle in the Capillary system, for example the IC Cube in a Dionex ICS-5000⁺ DC. If the Dionex CRD 200 is not already installed:

1. Remove the plugs from the REGEN ports at the rear of the Dionex CRD 180 or CRD 200 cartridge.
2. If a dummy cartridge or used Dionex CRD 180 or CRD 200 (Capillary) is installed in the CRD 200 receptacle, loosen the thumb screws of the dummy cartridge or used device and pull it out.
3. Plug the new Dionex CRD 180 or CRD 200 into the CRD receptacle so the regenerant ports at the rear of the cartridge seat correctly.
4. Tighten the thumb screws to ensure a firm fit.

3.2.2 Plumbing the Dionex CRD 180 or CRD 200 between the suppressor and the detection cell.



NOTE

Always use the precut tubing included with the suppressor and Dionex CRD 180 or CRD 200 (Capillary) to make connections. Do not cut or alter this tubing; the tubing is factory cut for an extremely low dead volume interface.

1. The Dionex CRD 180 or CRD 200 should be installed in the IC System.
2. To begin plumbing the Dionex CRD 180 or CRD 200, refer to the plumbing schematic shown in Figure 6.
3. Turn off the pump and Dionex ACES 300 before making any connections.
4. Remove the plugs from the Dionex CRD 180 or CRD 200 ports.
5. Connect the Outlet tubing from the Dionex ACES 300 to the ELUENT IN port of the Dionex CRD 180 or CRD 200 (Red label).
6. Connect the Outlet tubing from the Dionex CRD 180 or CRD 200 (Yellow label) to the conductivity cell inlet.
7. Connect the suppressor REGEN IN line to the outlet of the conductivity cell.
8. Dionex CRD 180 or CRD 200 installation is now complete. Regenerant connections are established when the Dionex CRD 180 or CRD 200 is plugged into its receptacle in the capillary system.

3.2.3 External Water setup

When using the external water mode of operation for the suppressor, the Dionex CRD 180 or CRD 200 (Capillary) is plumbed similar to what was previously described. However in step 7 the fluid from the conductivity cell outlet is diverted to waste and the suppressor REGEN IN line is plumbed to an external water source. It is recommended that the external water mode be set to 0.1 mL/min.

3.3 Dionex CRD 180 or CRD 200 Backpressure Measurement

The backpressure to the suppressor module may need to be measured and adjusted. This will ensure optimal performance of the system. Failure to complete the following steps may result in a leaking suppressor and/or Dionex CRD 80 or CRD 200.

- a) After installing the Dionex CRD 180 or CRD 200, measure the total system pressure (P1).
- b) Remove the line from the ELUENT OUT port of the suppressor and measure the system pressure (P2).

$$P1 - P2 = P3 \leq 100$$

- c) If P3 is higher than 100 psi, then trim the backpressure coil (line out of the conductivity cell) to achieve a total pressure of less than 100 psi.



WARNING

Total backpressure exceeding >100 psi may cause irreversibly damage to the Dionex CRD 180 or CRD 200 and/or suppressor.



NOTE

Always use 1/8" tubing when connecting lines for the regenerant side of the Dionex ERS 500 Suppressor or Dionex Atlas Electrolytic Suppressor (ERS/AES), Dionex Carbonate Removal Device 200 (CRD 200), Dionex Continuously Regenerated – Trap Column (CR-TC), and Eluent Generator (EG) Degas modules.

4. Applications

The Dionex Carbonate Removal Device (CRD 180 or CRD 200) has been optimized for removing the peak resulting from carbonate (as CO₂) in ion chromatography systems for anion analysis using Eluent Generation modules.

4.1 Recommended Applications

The Dionex CRD 180 and CRD 200 are recommended for both routine and trace level work with hydroxide and borate eluents when the presence of CO₂/carbonate from the sample interferes with the anion analytes. By simply being exposed to carbon dioxide in the air, samples can become contaminated with carbonate. In some samples, depending on the column and separation conditions, the presence of high levels of carbonate originating from dissolved carbon dioxide interferes with the accurate determination of analytes of interest, such as sulfate and nitrite. Under the above conditions, minimizing carbonate using the Dionex CRD 180 or CRD 200 leads to improved peak integration and quantification for analytes such as sulfate and nitrite in hydroxide RFIC systems. The Dionex CRD 180 and CRD 200 are particularly useful for analyzing drinking water, groundwater, wastewater, ultra-pure-water, and caustic solutions where carbonate is a major component in the samples, especially when pursuing large-volume injections or preconcentration techniques.

The Dionex CRD 180 is a version of the CRD that has reduced internal volume. This facilitates operation in conjunction with 4 µm resin packed capillary columns without unacceptable loss of peak efficiency. However the Dionex CRD 180 has less capacity for the removal of carbonate and is therefore not recommended for use with columns packed with resin larger than 4 µm. The Dionex CRD 180 is available only in capillary format. Microbore and standard-bore systems using 4 µm resin packed columns should use the Dionex CRD 200.

The Dionex CRD 200 (4 mm) is also recommended for the minimizing CO₂ from acidic samples prior to injection or analysis. Samples that have relatively high concentrations of CO₂ such as carbonated beverages can out-gas causing issues with reproducibility of both retention time and peak response. Trapped bubbles in the system can affect the system operation and performance and overall chromatography. It is therefore desirable to remove these bubbles from the sample prior to injection. The Dionex CRD 200 (4 mm) is particularly useful for analyzing carbonated beverages such as cola. As a sample pretreatment tool the Dionex CRD 200 (4 mm) is not recommended for basic or neutral samples since CO₂ is no longer a volatile component, rather it exists as bicarbonate and/or carbonate anion in solution.

The Dionex CRD 200 is also recommended for anion analysis in ammoniated waters. The CRD 200 is operated in the vacuum mode for the above application.

4.2 Negative Dip at the Carbonate Peak Location

A negative peak may be observed at the location where the peak resulting from carbonate is removed. It is hypothesized that the trace level of carbonic acid at the carbonate peak location consumes the trace hydroxide in the background and this lowers the background response at that location.

Some applications may not benefit from the Dionex CRD 180 or CRD 200 module due to the following reasons.

- a) Even after CO₂ removal the peaks of interest continue to co-elute with the residual CO₂ peak. If possible, it is recommended under these conditions to investigate other columns or eluent conditions that exhibit different selectivity for the peaks of interest.
- b) The presence of CO₂ does not interfere with peaks of interest. Under these conditions, there is no need for a Dionex CRD 180 or CRD 200 device.



WARNING

The Dionex CRD 180 and CRD 200 are designed to perform optimally with hydroxide and borate eluents only. It is not recommended for carbonate and/or bicarbonate, or cation applications. The CRD 300 is recommended for carbonate and/or bicarbonate eluents.



NOTE

The Dionex CRD 180 and CRD 200 will remove CO₂ from the carbonate peak thus minimizing the peak, but complete removal of CO₂ may not occur.



NOTE

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

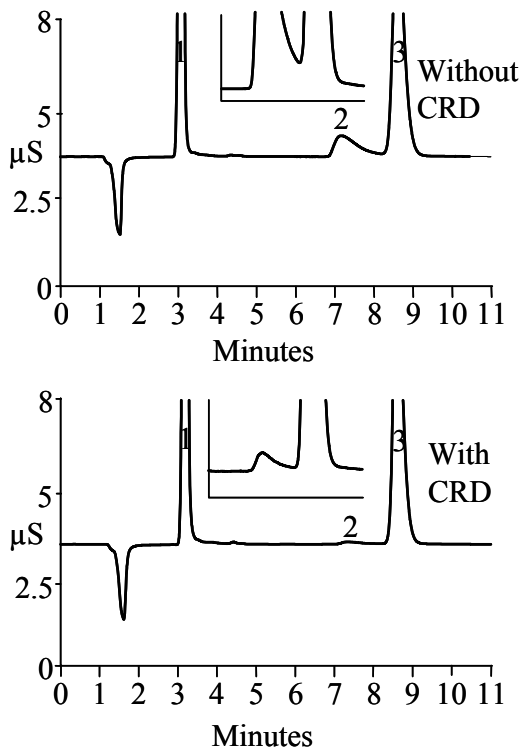
5. Operation

5.1 Example Chromatograms

Analysis using borate eluents is typically pursued in the nuclear power industry for analyzing borated waters. In the example below, the Dionex CRD 200 is useful in reducing the carbonate peak and improving the quantitation of chloride.

Figure 9 Dionex CRD 200 with AS4A Borate Chemistry

CRD Application with AS4A Borate Chemistry



Column: IonPac® AS4A/AG4A, 4 mm
 Eluent: 100 mM boric acid
 5 mM KOH (EG)
 Flow Rate: 1.5 mL/min
 Suppressor: ASRS® ULTRA II, 4 mm
 Current: 300 mA
 Oven: 30 °C

Concentrator: IonPac AG4A
 Volume: 40 mL

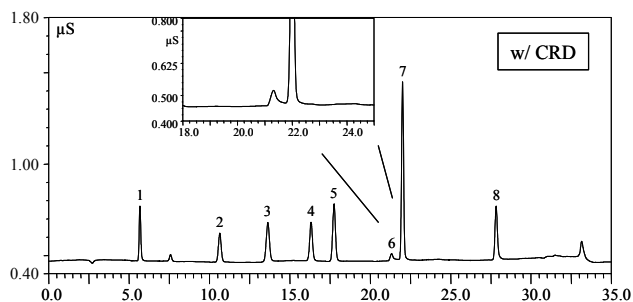
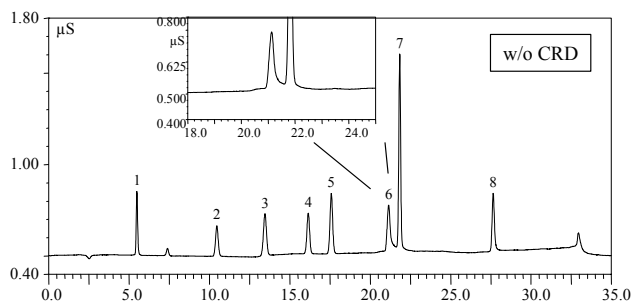
Sample:	Concentration (ppb)
Fluoride	10
Carbonate	n.a.
Chloride	10

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A typical anion application at ppb level shown below demonstrates the utility of the Dionex CRD 200 to remove CO₂ and aid in the improved quantitation of sulfate.

Figure 10 Dionex CRD 200 with AS19 Gradient Chemistry

CRD Application with AS19 Gradient Chemistry



Conditions:

Column	AS19 4-mm
Flow rate	1.0 ml/min
Suppressor	ASRS Ultra II 4-mm
Current	300 mA
Loop	200 μl
Oven	30 °C

Time	Gradient (mM)
0	10
10	10
25	45
30	45
30.1	10
35	10

Sample	Concentration (ppb)
Fluoride	10
Chloride	15
Nitrite	50
Bromide	50
Nitrate	50
Carbonate	n/a
Sulfate	75
Phosphate	75

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6. Troubleshooting



WARNING

High backpressure may cause irreversible damage to the Dionex CRD 180, CRD 200 and suppressors. The total backpressure from the cell, the CRD, 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 Dionex CRD 180 and CRD 200.
- b) Measure the backpressure to the suppressor (i.e. cell + Dionex CRD 180 or CRD 200 + the backpressure coil) following the instructions in (Section 3.3). 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 tubing's if needed.
- d) When operating in external water mode, ensure that the external water 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 CO₂ Removal Efficiency

- a) Ensure that the Dionex CRD 180 or CRD 200 is installed correctly with suppressor waste (base) flowing into the regenerant channel.
- b) Check for leaks and reconnect.

6.3 No Peaks

- a) Ensure that the Dionex CRD 180 or CRD 200 is connected as outlined in the plumbing instructions.
- b) Check for leaks and reconnect.
- c) The Dionex CRD 180 or CRD 200 may have an internal leak due to damage; replace the device.

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

- a) Ensure that the recommended RFIC-EG system pressure is maintained (optimal operating pressure for 2 mm and 4 mm systems is 2300 psi). If needed, add the backpressure coil for raising the total system pressure by installing 0.003" ID Yellow tubing between the EG degas module and the injection valve.
- b) Release any trapped gases in the cell, suppressor, or the Dionex CRD 180 or CRD 200 by systematically opening the lines from:
 - ELUENT OUT port of the suppressor.
 - The ELUENT IN and ELUENT OUT ports of the Dionex CRD 180 or CRD 200.
 - The cell outlet port.

6.5 Negative Dips Before All the Analyte Peaks

- a) Analytical IC systems only: The tubing leading into or exiting the Dionex CRD 200 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) The suppressor may not be fully regenerated. Regenerate the suppressor using sulfuric acid, as outlined in the suppressor User's Manual.
- c) The Dionex CRD 180 or CRD 200 may have an internal leak due to damage. Replace the device if a leak is detected.

6.6 Negative Peak or Dip at the Carbonate Peak Location

- a) Refer to (Section 4.2.).

6.7 How to Check for Leaks

- a) Visually inspect for liquid drops or leakage at the Dionex CRD 180 or CRD 200 eluent and regen ports. Reconnect the lines and tighten the fittings if needed. Do not over tighten as damage may occur.
- b) Disconnect the lines from the regen ports of the Dionex CRD 200 and, with the eluent flowing in the eluent channel of the Dionex CRD 200, observe for fluid flow out of the regen channel. A continuous flow of liquid from the regen ports of the Dionex CRD 200 indicates an internal leak. Replace the device.

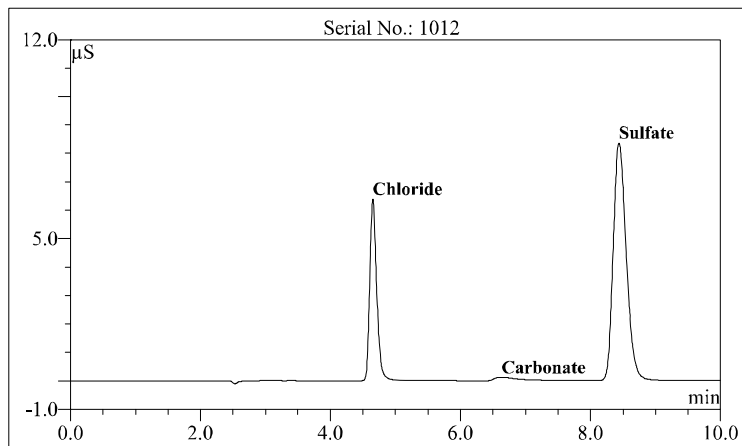
For assistance, contact Technical Support for Dionex Products. In the U.S., call 1-800-346-6390. Outside the U.S., call the nearest Thermo Fisher Scientific office.

Appendix – Quality Assurance Report (QAR)

Carbonate Removal Device
CRD 4-mm
Product No. 062983

Date: 04-Jun-05 09:49
Serial No. : 001012

Eluent: 23 mM KOH
Eluent Source: EGC II KOH Cartridge
Flow Rate: 1.00 mL/min
Temperature: 30 °C
Detection: Suppressed Conductivity using a CD25
Suppressor: Anion Self-Regenerating Suppressor (ASRS® 300, 4mm)
 AutoSuppression® Recycle Mode
Applied Current: 300 mA
Injection Volume: 20 µL
Storage Solution: Deionized Water



QA Results:

<u>Test Parameter</u>	<u>Specification</u>	<u>Results</u>
Leak	Pass/Fail	Passed
Eluent Back Pressure	Pass/Fail	Passed
Regen Back Pressure	Pass/Fail	Passed
CO2 Removal	Pass/Fail	Passed

Production Reference:

Datasource: SPD_local
 Sequence: C62983-VAL-02_CRD_4mm
 Sample No.: 63

6.60 SP3 Build 1485 (Demo-Installation)
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