

INSTALLATION INSTRUCTIONS and TROUBLESHOOTING GUIDE

for the

DISPLACEMENT CHEMICAL REGENERATION (DCR) 2-LITER KIT

(P/N 056882)

DISPLACEMENT CHEMICAL REGENERATION (DCR) 4-LITER KIT (P/N 056884)

QUICKSTART STEPS AND LINKS Click on the blue text below to get started.

1. See Section 2, "Choosing the Regenerant Concentration" for details on regenerant preparation.

2. See Section 3, "Recommended Setup" for details on installation.

3. See Section 4, "Troubleshooting" for details on troubleshooting.

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SECTION 1 - INTRODUCTION TO DISPLACEMENT CHEMICAL REGENERATION (DCR)

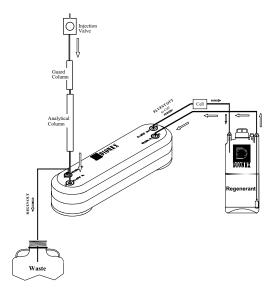


Figure 1 Displacement Chemical Regeneration (DCR) Mode with the MircoMembrane Suppressor III

Displacement Chemical Regeneration (DCR) is a new technique for dispensing regenerant in the chemical regeneration mode of operation for the MicroMembrane suppressors including MMS III suppressor devices. The DCR mode uses the effluent from the conductivity cell to drive the regenerant into the regenerant chamber of the suppressor using a closed bottle configuration. The regenerant flow is equivalent to the eluent flow. Thus, continuous suppression is achieved without the use of an external regenerant pump. DCR is recommended for all carbonate applications, many hydroxide applications and most cation applications.

DCR is recommended for use with all anion exchange columns using carbonate eluents including all 2-mm, 3-mm and 4-mm columns.

DCR is recommended for use with many anion exchange columns using hydroxide eluents including the IonPac AS11, AS15, AS16, and AS17.

DCR is recommended for use with many cation exchange applications using the IonPac CS12A, CS10, CS11, CS15, and CS16.

-NOTE -

For Anion or Cation MicroMembrane Suppressor Installation Instructions, refer the appropriate suppressor manual (AMMS III, P/N 031727 or CMMS III, P/N 031728).

Always remember that assistance is available for any problem that may be encountered during the shipment or operation of DIONEX instrumentation and columns through the DIONEX North America Technical Call Center at 1-800-DIONEX-0 (1-800-346-6390) or through any of the DIONEX Offices listed in, "DIONEX Worldwide Offices". If you feel that any of the sections in this Installation Instructions and Troubleshooting Guide are unclear, incomplete or are not needed, please write down your comments on the Reader's Comments Form at the end of this manual and return the form to us.

SECTION 2 -CHOOSING THE REGENERANT CONCENTRATION

2.1 REGENERANT CONCENTRATION OPTIMIZATION

In the DCR mode of operation, the regenerant flow rate is equivalent to the eluent flow rate. Therefore, the concentration of the regenerant needs to be optimized to achieve complete suppression and low background. The optimal regenerant concentration can be estimated for a known eluent strength by:

Anion Regenerant Concentration = (mM eluent) x 2

Cation Regenerant Concentration = $(mM \text{ eluent}) \times 5$

For example using 20 mM MSA as the eluent for cation analysis, the regenerant concentration of 100 mN tetrabutylammonium hydroxide (TBAOH) is recommended.

- NOTE ----

This recommended ratio of eluent equivalents to regenerant equivalents is a convenient starting point for optimizing the regenerant concentration. Further optimization may be done to achieve low levels of leakage and low background.

The following table summarizes recommended regenerant concentrations for various standard anion applications.

| Table 1 commended Regenerant Concentrations for Various Standard Anion Applicat | | | | | |
|---|--------------------------------------|--|--|--|--|
| | | | | | |
| AS4A, AS4A-SC, AS12A | 25 mN H ₂ SO ₄ | | | | |
| AS14, AS14A | 50 mN H ₂ SO ₄ | | | | |
| AS11, AS17 | 50 mN H ₂ SO ₄ | | | | |
| AS9-HC/AS15 | 75 mN H ₂ SO ₄ | | | | |
| | | | | | |

For 4-mm column applications, the maximum suppression capacity in the DCR mode for anion analysis is approximately 70 µequivalents/min or roughly 70 mM of hydroxide eluent at 1mL/min. For 2-mm column applications, approximately 70 mM of hydroxide eluent at 0.25 mL/min can be suppressed.

The following table summarizes recommended regenerant concentrations for various standard cation applications.

| | Table 2 | | | | | | |
|--|--|--|--|--|--|--|--|
| Recommended Regenerant Concentrations for Various Standard Cation Applications | | | | | | | |
| Cation Application | Recommended Regenerant Concentrations | | | | | | |
| CS12A, CS16 | 100 mM TBAOH | | | | | | |
| CS15 | 100 mM TBAOH | | | | | | |
| CS10, CS11 | 150 mM TBAOH | | | | | | |

The maximum suppression capacity in the DCR mode for cation analysis varies with the type of acid, the flow rate and the concentration of the regenerant used. The following table lists the maximum suppressable concentrations with appropriate regenerants for CMMS III suppressors in the DCR mode of operation.

| Suppressor | Regenerant | Eluent Flow Rate | Maximum Suppression Capacity |
|--------------|--------------|---------------------|---------------------------------|
| CMMS III 4mm | 100 mM TBAOH | 1 mL/min | 30-35 mM MSA |
| | | | 40 mM HCL |
| | | | $20 \text{ mN H}_2\text{SO}_4$ |
| | 150 mM TBAOH | 1 mL/min | 40-45 mM MSA |
| | | | 50 mM HCL |
| | | | $30 \text{ mN H}_2\text{SO}_4$ |
| CMMS III 2mm | 100 mM TBAOH | 0.5 mL/min | 35 mM MSA |
| | | | 30 mM HCL |
| | | | 15-20 mN H_2SO_4 |
| | 150 mM TBAOH | 0.5 ml/min | 40 mM MSA |
| | | | 55 mM HCL |
| | | | $30 \text{ mN H}_2\text{SO}_4$ |
| CMMS III 2mm | 100 mM TBAOH | 0.3 ml/min | 45 mM MSA |
| | | | 35 mM HCL |
| | | | $20 \text{ mN H}_2\text{SO}_4$ |
| | 150 mM TBAOH | 0.3 ml/min | 55 mM MSA |
| | | | 70 mM HCL |
| | | | $30 \text{ mN H}_2\text{SO}_4$ |

2.2 REGENERANT PREPARATION FROM CONCENTRATES

The following reagent concentrates are available for preparing regenerants in the DCR mode of operation. Note the slight variance in the regenerant concentrations has minimal impact on suppression.

| Table 4 Displacement Chemical Regeneration Reagents | | | | | |
|---|---|--|--|--|--|
| | | | | | |
| | 75 mL of 2.0 N sulfuric acid | | | | |
| P/N 057555 | 4-Pak of Anion Regenerant Concentrate | | | | |
| | 75 mL of 2.0 N sulfuric acid | | | | |
| P/N 057561 | Cation Regenerant Concentrate | | | | |
| | 100 mL of 2.06 M TBAOH | | | | |
| P/N 057556 | 4-Pak of Cation Regenerant Concentrate 100 mL of 2.06 M TBAOH | | | | |

A. The total volume of the above concentrate container can be diluted to 2L and topped off to roughly achieve 75 mN sulfuric acid regenerant solution or 100 mM TBAOH regenerant solution in the 2L container.

B. For 4L, 2 bottles of the above concentrate can be diluted to 4L and topped off to make 75 mN sulfuric acid regenerant solution or 100 mM TBAOH regenerant solution in a 4L container.

2.2.1 SULFURIC ACID REGENERANT CONCENTRATE (500 mN)

Another Sulfuric acid regenerant concentrate is P/N 039203 (200 mL of 500 mN sulfuric acid regenerant concentrate solution) designed for conventional chemical suppression applications.

In the DCR mode this concentrate can be used to prepare 25 mN and 50 mN acid solution as shown below.

- A. The total volume of the sulfuric acid concentrate (P/N 039203) can be diluted to the top in a 2L container to roughly generate 50 mN regenerant solution.
- B. 100 mL of the concentrate P/N 039203 when diluted to the top in a 2L container is sufficient to roughly generate 25 mN regenerant solution.

Use proper safety precautions in handling acids and bases.

SECTION 3 - RECOMMENDED SETUP

The following DCR setup is recommended and ensures continuous operation with minimal down time.

In the DCR mode both the eluent and regenerant are used at the same rate and hence should be replaced at the same time. This ensures that the suppressor down time is minimal. For example, if a 2L container is used for the eluent, the same capacity container should be used for the regenerant. The regenerant reservoir should be completely filled to the top of the neck on startup while the eluent is made up to the prescribed 2L volume mark. Since the regenerant volume is slightly higher than the eluent volume, > 90% of the regenerant reservoir can be used for suppression. The expended regenerant should be replaced with fresh regenerant after emptying the entire bottle to waste. Note the waste in the bottle is predominantly dilute acid for anion applications or dilute base for cation applications and should be disposed off to waste following appropriate waste disposal procedures.

3.1 DCR BOTTLE

The DCR bottle is available in both 2L and 4L capacities. The bottle has two liquid lines for making connections. A short line inserted roughly 12.5 mm ($\frac{1}{2}$ in.) into the bottle is labeled as "S". The unlabeled longer line is inserted all the way to the bottom of the bottle.

3.2 DCR BOTTLE ASSEMBLY

Fill the bottle with the desired regenerant all the way to the top of the bottle neck leaving approximately 12.5 mm ($\frac{1}{2}$ in.) from the top. Fill the rest of the bottle neck with DI water and ensure that the entire container is filled completely. Tighten the cap on the reservoir, leaving no air pockets. A small liquid leak occurring on closing the bottle should be wiped off with a wipe. Make liquid connections as outlined below.

- NOTE

Some mixing of the regenerant with the cell effluent may occur when the bottle is tilted or shaken and usually such mixing is not detrimental to the suppression process. However it is recommended that the regenerant bottle is left undisturbed after installation.

3.3 TUBING ORIENTATION IN THE BOTTLE

The tubing orientation inside the regenerant reservoir is based on the density of the cell effluent and the regenerant. For example in the case of hydroxide and carbonate eluents, the lower density cell effluent displaces the higher density sulfuric acid regenerant and hence the detector cell outlet line delivers to the top of the regenerant bottle while the regenerant line into the suppressor is the longer line from the bottom of the regenerant bottle.

3.3.1 FOR ANION APPLICATIONS (HYDROXIDE AND CARBONATE ELUENTS)

Since the suppressed effluent (water or carbonic acid) is less dense than the sulfuric acid regenerant, connect the black 0.01" I.D. tubing from the conductivity cell outlet to the coupler connected to the line labeled "S" on the bottle. In addition to serving as the connecting tubing, the black tubing also serves as the back pressure to the cell. The unlabeled line from the bottle should then be connected to the REGEN IN port of the suppressor using 1/8" TFE tubing (0.062" I.D.; 0.125" O.D.). The cell effluent will then be pumped into the top of the bottle while the regenerant is dispensed from the bottle into the suppressor regenerant chambers. The waste line from the suppressor REGEN OUT port should be 1/8"TFE tubing (0.062". I.D.; 0.125 in. O.D.) diverted to an appropriate waste container.

3.3.2 FOR CATION APPLICATIONS (METHANESULFONIC ACID, SULFURIC ACID OR HYDROCHLORIC ACID)

Since the suppressed effluent (water) is denser than the tetrabutylammonium hydroxide regenerant, connect a black 0.01 in. I.D. tubing from the conductivity cell outlet to the coupler connected to the unlabelled line on the bottle. The line labeled "S" should then be connected to the REGEN IN port of the suppressor using 1/8 in. TFE tubing (0.062 " I.D.; 0.125" O.D.). Using this configuration, the cell effluent will be pumped into the bottle of the bottle while the regenerant is dispensed from the top of the bottle into the suppressor regenerant chambers. The waste line from the suppressor REGEN OUT port should be a 1/8 in. TFE tubing (0.062 in. I.D.; 0.125 in. O.D.) diverted to an appropriate waste container.

3.3.3 FOR SOLVENT APPLICATIONS

When using solvents, the density of the solvent with respect to water determines the location of the return line from the cell. For example, when using acetonitrile in MSA eluents with TBAOH regenerant, TBAOH is denser than acetonitrile. Therefore, the return line from the regenerant bottle into the suppressor is the longer tubing from the bottom of the bottle. For eluents using methanol with hydroxide, methanol is less dense than the regenerant and the regenerant line from the bottle into the suppressor is the longer tubing.

- NOTE -

The black 0.01 in. I.D. tubing from the cell outlet is used as the back pressure for both 2-mm and 4-mm applications.

DO NOT ATTACH ANY ADDITIONAL BACKPRESSURE COILS.

Additionally, do not attach any backpressure coils or backpressure tubing/devices to the suppressor regenerant chambers. The suppressor waste should be connected using 1/8 in. I.D. tubing only.

SECTION 4 - TROUBLESHOOTING

- NOTE –

For Anion or Cation MicroMembrane Suppressor Troubleshooting Instructions, refer the appropriate suppressor manual (AMMS III, P/N 031727 or CMMS III, P/N 031728).

4.1 HIGH NOISE AND HIGH DRIFT

- A. Unusually high noise and drift may be observed when the bottle is not completely filled all the way to the top of the container. Since liquid is incompressible and gases are compressible, any void left on the top of the DCR reservoir will result in pulsation that will be transferred to the analytical run.
- B. Trapped gases in the MMS III connections or in the conductivity cell may also cause high noise. To release trapped gases simply loosen the fittings to the MMS III or the cell, one at a time with the pump on and re-tightening the fitting after a minute or so.
- C. Gases in the regenerant in the DCR bottle may also cause high noise and drift. On long term storage of the regenerant, excessive gas bubbles may be observed. Simply make fresh regenerant using vacuum degassed DI water and replace the old regenerant.

4.2 LEAKING BOTTLES

Carefully depressurize the bottle and wipe the liquid in the cap and on the threads of the bottle top. Reconnect the bottle cap and tighten firmly. If leakage does not stop this indicates a defective bottle hence replace the bottle.

4.3 SIPHONING DCR RESERVOIR

If the liquid in the DCR reservoir drains this indicates that the bottle was not firmly sealed. Another possibility is a leaking bottle. Carefully depressurize the bottle and wipe the liquid in the cap and on the threads of the bottle top. Reconnect the bottle cap and tighten firmly. If siphoning does not stop this indicates a defective bottle hence replace the bottle.