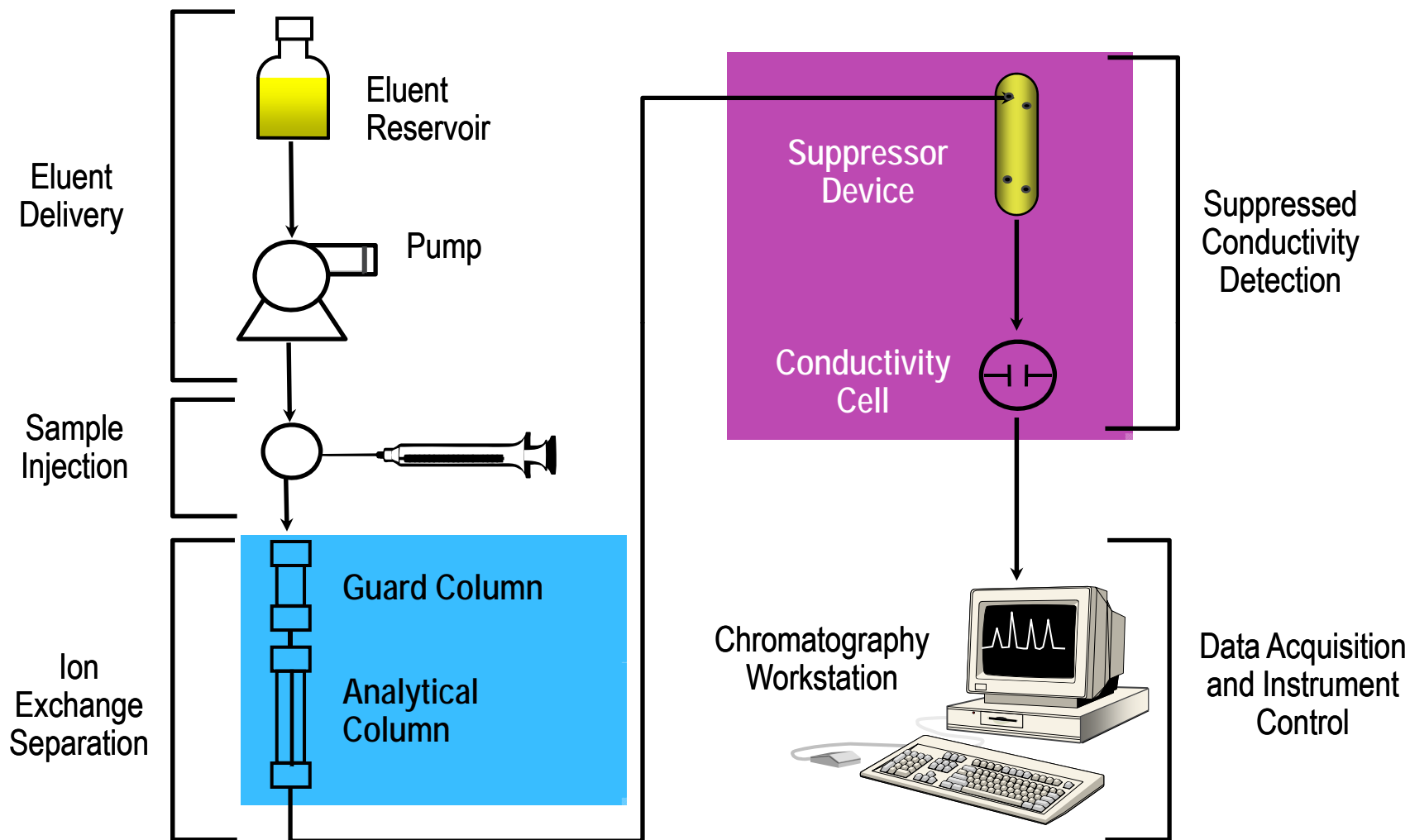




Basic IC Operation
Tips and Tricks

General IC System Configuration



Eluents

- Acts as the mobile phase – the analyte's means of transport.
 - The eluent and solute must be compatible in terms of solubility, oxidation, reduction.
 - The eluent must also be compatible with all other system components, specifically the column and suppressor (e.g. pH, solvents).
- Typical eluents used in IC for common anions and cations analysis
 - Anions
 - Hydroxide selective column: KOH, NaOH
 - Carbonate selective column: Carbonate, Carbonate/Bicarbonate
 - Cations
 - MSA, sulfuric acid

Eluent Preparation

- All standards, eluents, regenerants, and reagents should be prepared with 18.2 MΩ (or better) deionized, filtered water.
- Prepare solutions from reagent grade (HPLC) chemicals with a label analysis of 99% purity or greater.
- Filter particulates from all solutions, including samples (IC certified filter)
- Eluents should be thoroughly degassed prior to introduction into the chromatography system. This practice will help prevent air bubbles from forming in the pump or detector cell, and minimizes carbonate contamination for hydroxide eluent.

Preparation of Sodium Hydroxide Eluent

Non-Capillary IC Systems ONLY

The presence of carbon dioxide in the water and eluent concentrate can lead to the formation of carbonate which may appear as a peak in the chromatogram, affect retention, or lead to high background.

To prevent CO_3^{2-} contamination:

- Use only low-carbonate grade 50% sodium hydroxide for eluent preparation. (No NaOH pellets!)
- It is recommended that the 50% sodium hydroxide solution be obtained in the smallest possible container, and it should **not** be used past the half-full mark.
- The water used in the preparation of an hydroxide eluent must be thoroughly degassed with helium or vacuum degassing prior to the addition of sodium hydroxide.
- After the addition of hydroxide, the eluent must be blanketed with helium.

* Refer to column manual for more information.

Reagent-Free Ion Chromatography (RFIC) Systems

- RFIC™ systems are ion chromatography systems that utilize electrolytic devices to generate (EG) or regenerate (ER) eluents in the separation processes

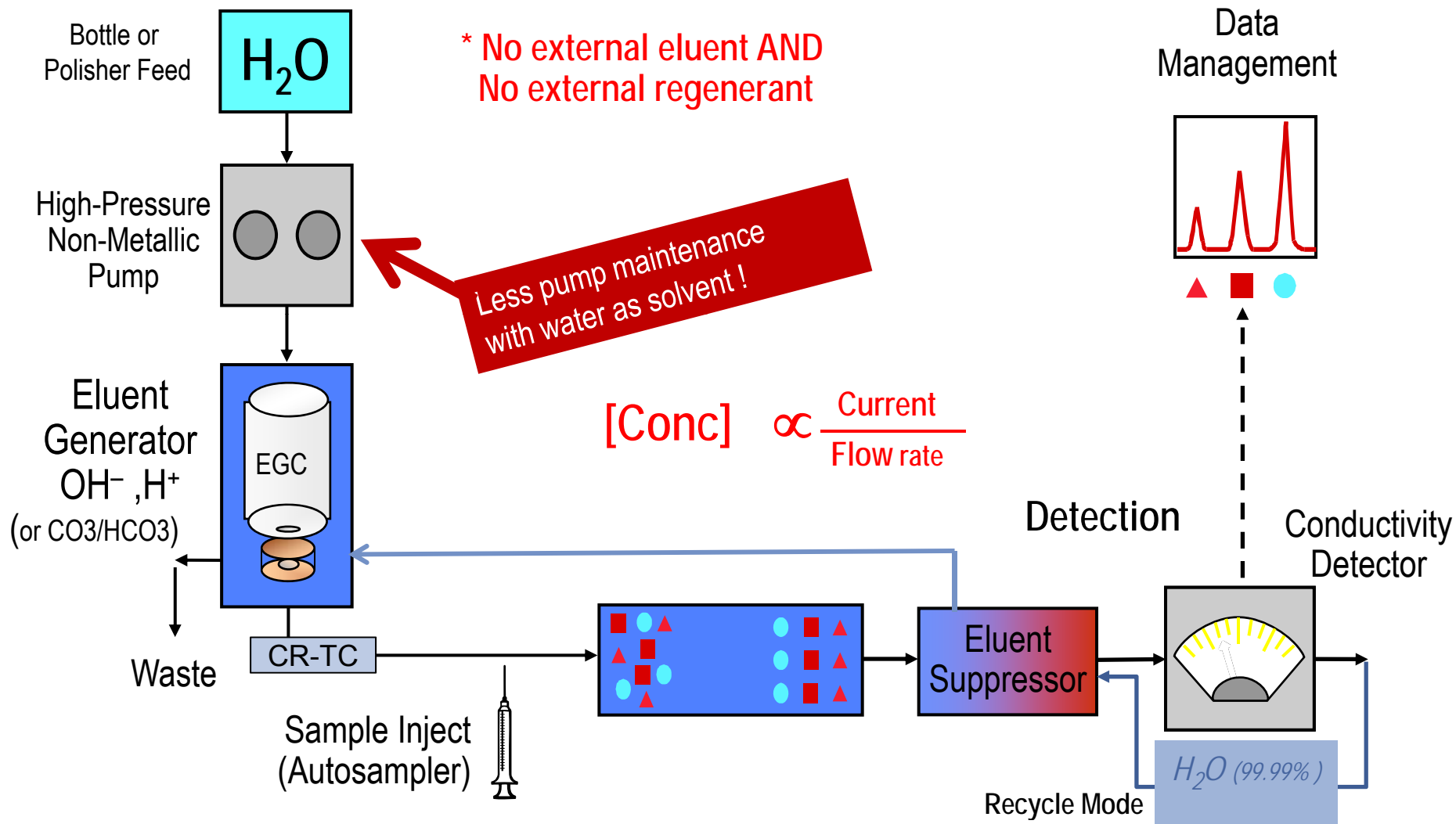
- RFIC-EG™ systems (2003)



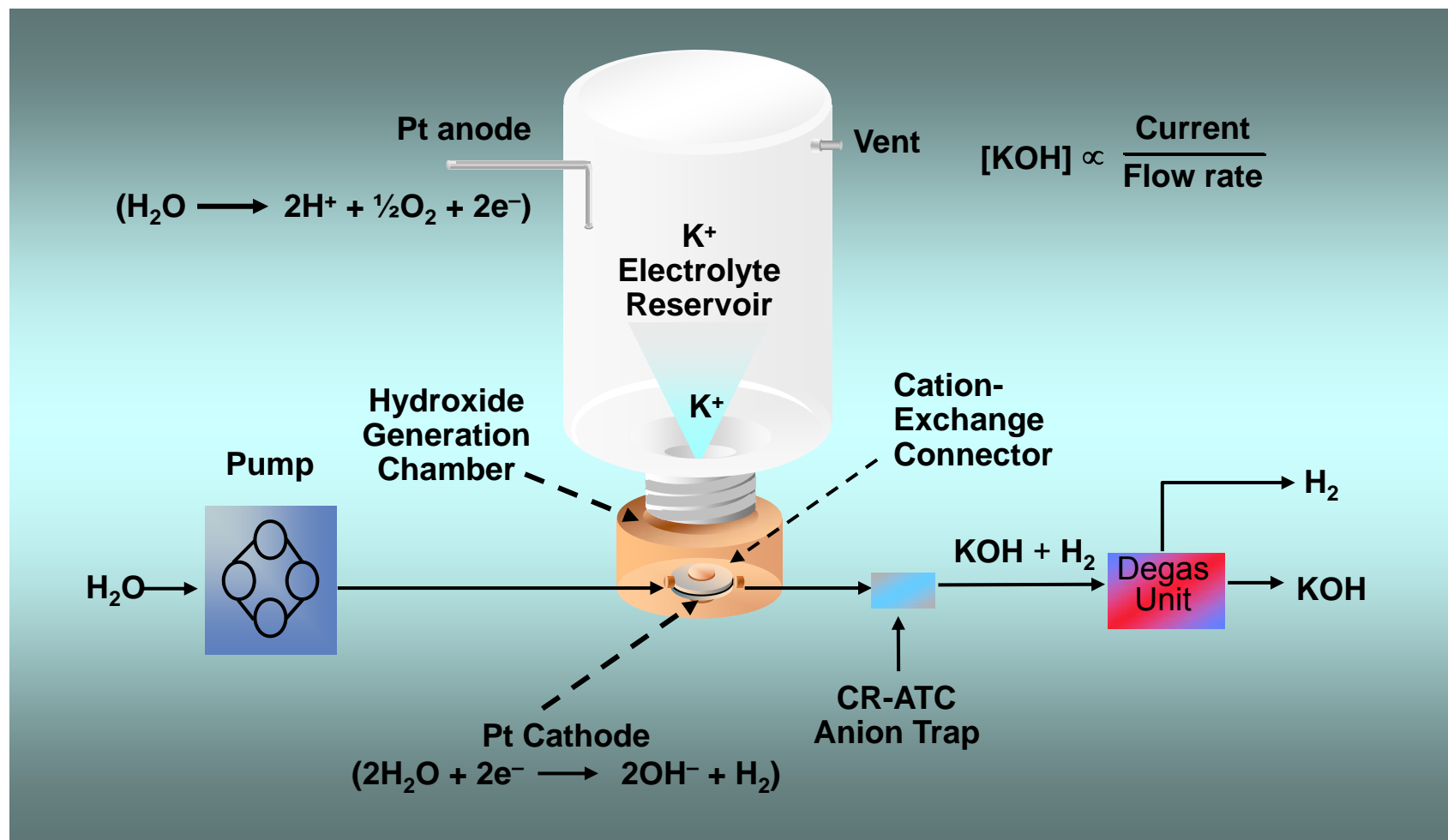
- RFIC-ER™ systems (2008)



Reagent-Free™ IC System*



Electrolytic Generation of KOH Eluents Using an EGC-KOH Cartridge

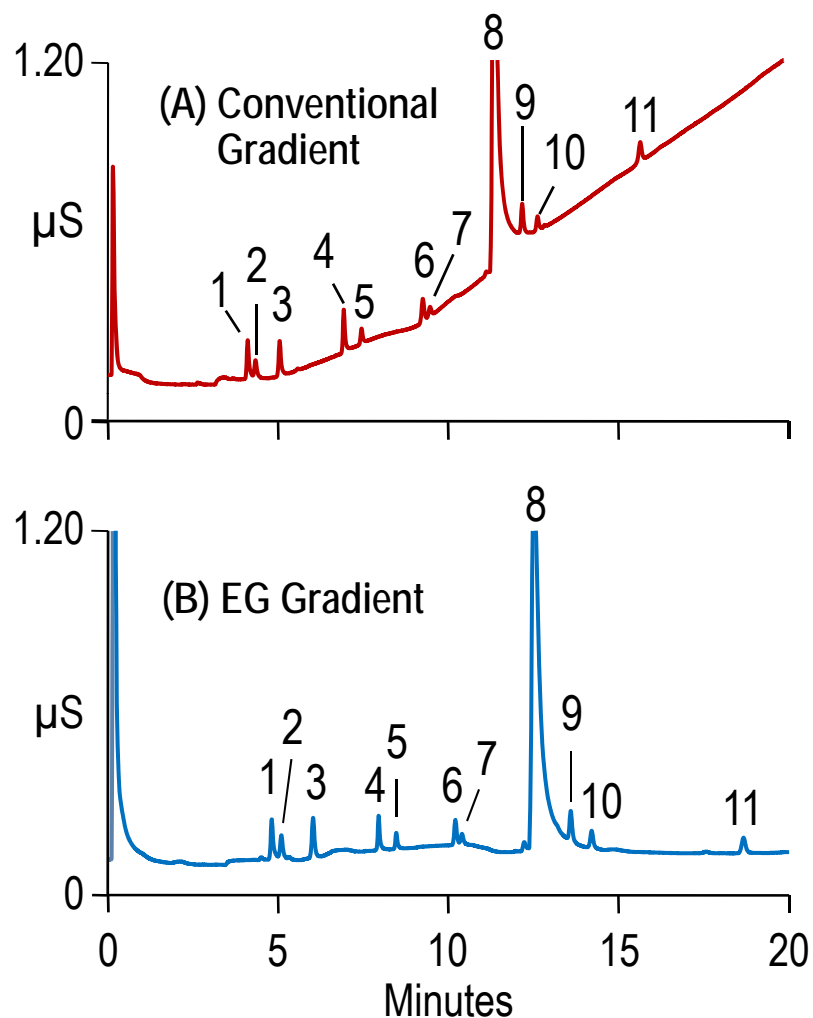


Advantages of RFIC-EG

- Eliminate the time and errors associated with manual eluent preparation.
- Save time, labor, and operating costs.
- Improve reproducibility, day-to-day, week-to-week, lab-to-lab.
- Easy to use.
- Use only DI water!
- EGC type
 - KOH, NaOH, LiOH, K₂CO₃
 - MSA



Improved Performance for Trace Anion Analysis



Column: IonPac® AG11, AS11, 2 mm

Eluent: (A) Manually prepared NaOH
 (B) KOH (EGC-KOH cartridge)
 0.5 mM to 2.5 min,
 to 5.0 mM at 6 min,
 to 26 mM at 20 min

Flow Rate: 0.5 mL/min

Inj. Vol.: 1.0 mL

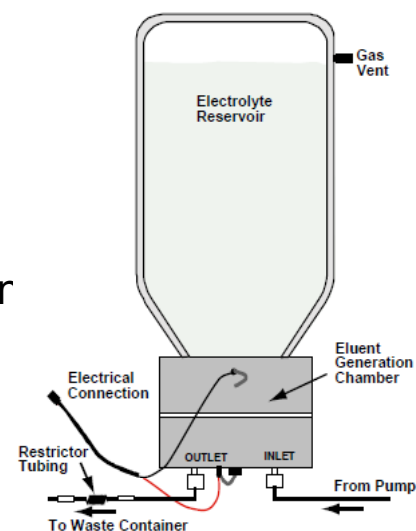
Suppressor: ASRS®, external water mode

Detection: Suppressed conductivity

Peaks:		
1.	Fluoride	0.37 µg/L (ppb)
2.	Acetate	1.0
3.	Formate	0.93
4.	Chloride	0.44
5.	Nitrite	0.27
6.	Bromide	1.0
7.	Nitrate	0.33
8.	Carbonate	–
9.	Sulfate	0.64
10.	Oxalate	0.39
11.	Phosphate	1.1

New EG Cartridge Installation

- Invert the EG Cartridge with EG Chamber downward
- Shake or tap EGC to get bubbles off of membrane
- Do **NOT** invert after bubbles have been removed
- Make sure overflow port is connected to a suitable waste container
- EGC fluid is highly caustic; use caution and clean up spills immediately!!!
- For new EGCs...
 - ICS-3000: the serial number must be entered in the CM Server Configuration
 - ICS-5000: the EG module will automatically detect and register the EGC S/N
- Ensure that the correct EGC serial number appears in the drop-down list in the Chromeleon Instrument Control Panel
- Condition a new EGC by running at 50 mM (1.0 ml/min) for 30 minutes

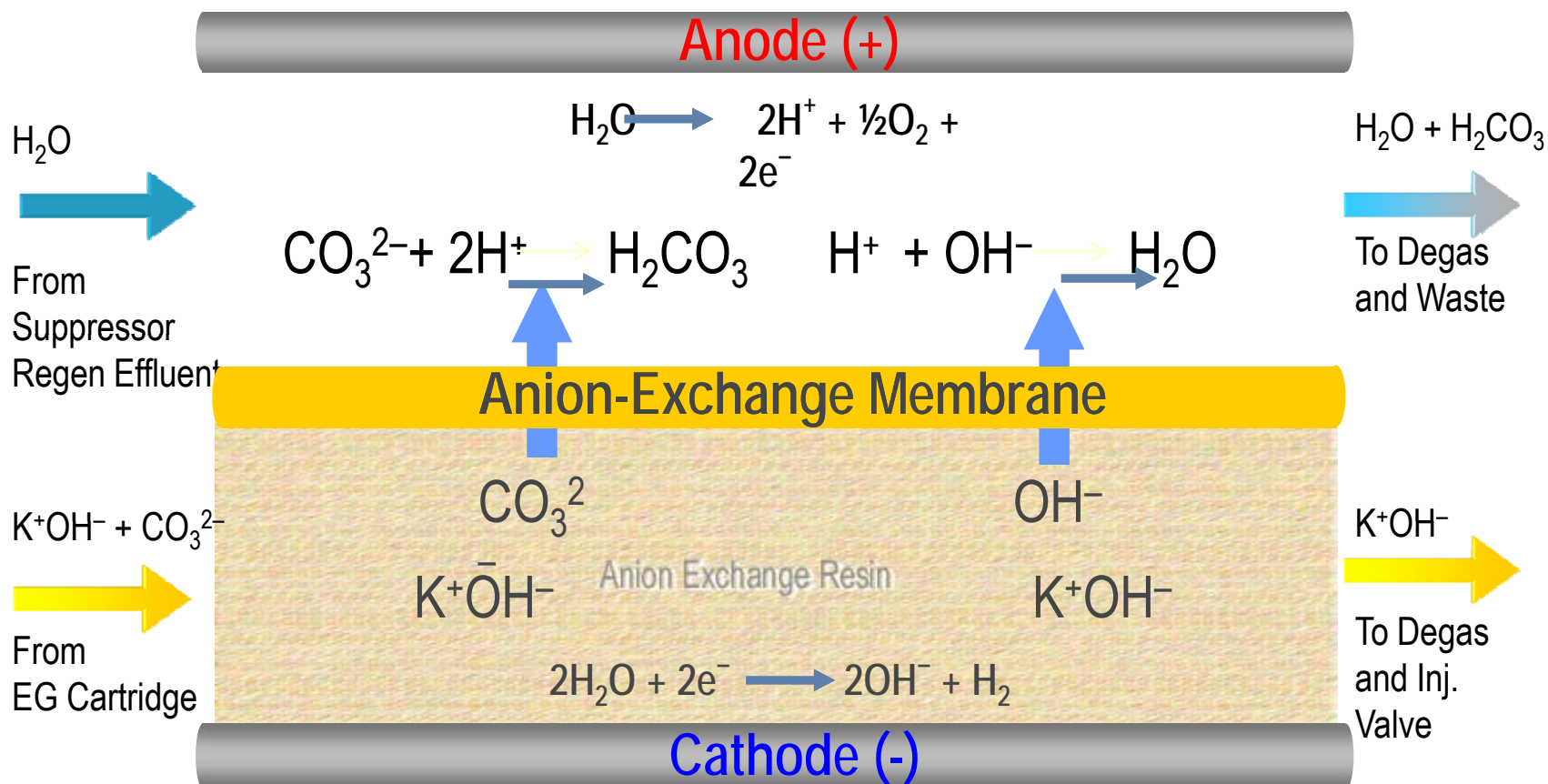


Continuously Regenerated Trap Columns

- Continuously Regenerated Trap Columns (together with the EG) remove anionic or cationic impurities from eluents and provide improved chromatographic performance over pre-mixed eluents
 - Lower baseline drift
 - Improved retention time stability
 - Improved resolution
 - Improved detection limits
- Format
 - CR-ATC: Continuously Regenerated Anion Trap Column (removal of CO_3^{2-})
 - CR-CTA: Continuously Regenerated Cation Trap Column (removal of NH_4^+)



CR-ATC Operation Schematic

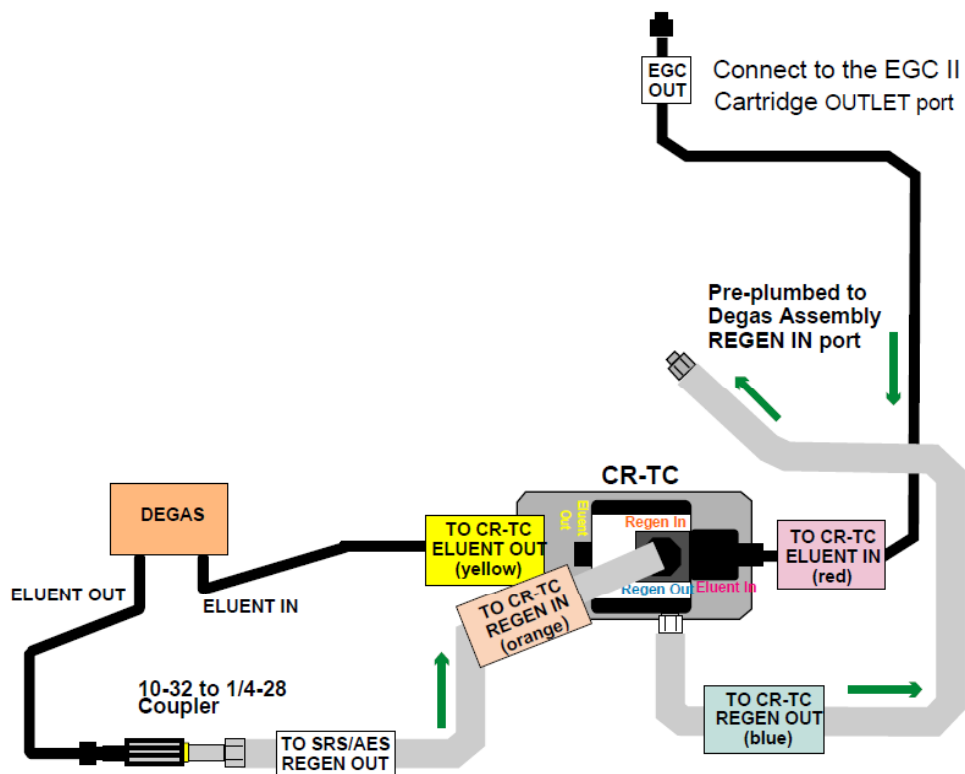


Continuously Regenerated Trap Columns

Care and Maintenance

- Always follow hydration procedure for start up
- Never turn on current without flow
- After use, re-hydrate for storage

CR-TC Hydration



CR-TC Plumbing Diagram for Hydration

Pumping DI water at flow rate of the application for at least 10minutes

Pump

- Priming
 - Make sure to prime pump to remove air bubbles from eluent lines and pump head
- Flow rate
 - Gradually increase flow rate to target for application.
- Backpressure
 - Backpressure easily monitored in Chromeleon. Log backpressure to make sure pump is sufficiently primed and system is stable.



**Conductivity Cell & Suppressor
Care & Start-up**



The world leader in serving science

Suppression Process

- Suppression converts the eluent counter ions so that the background conductance of the eluent is negligible. Decreases background conductivity.
- Also, it converts the analyte to a single, highly conductive form. Increases analyte response.
- Lastly, it removes the sample counter ions. Increases analyte sensitivity.

Suppression

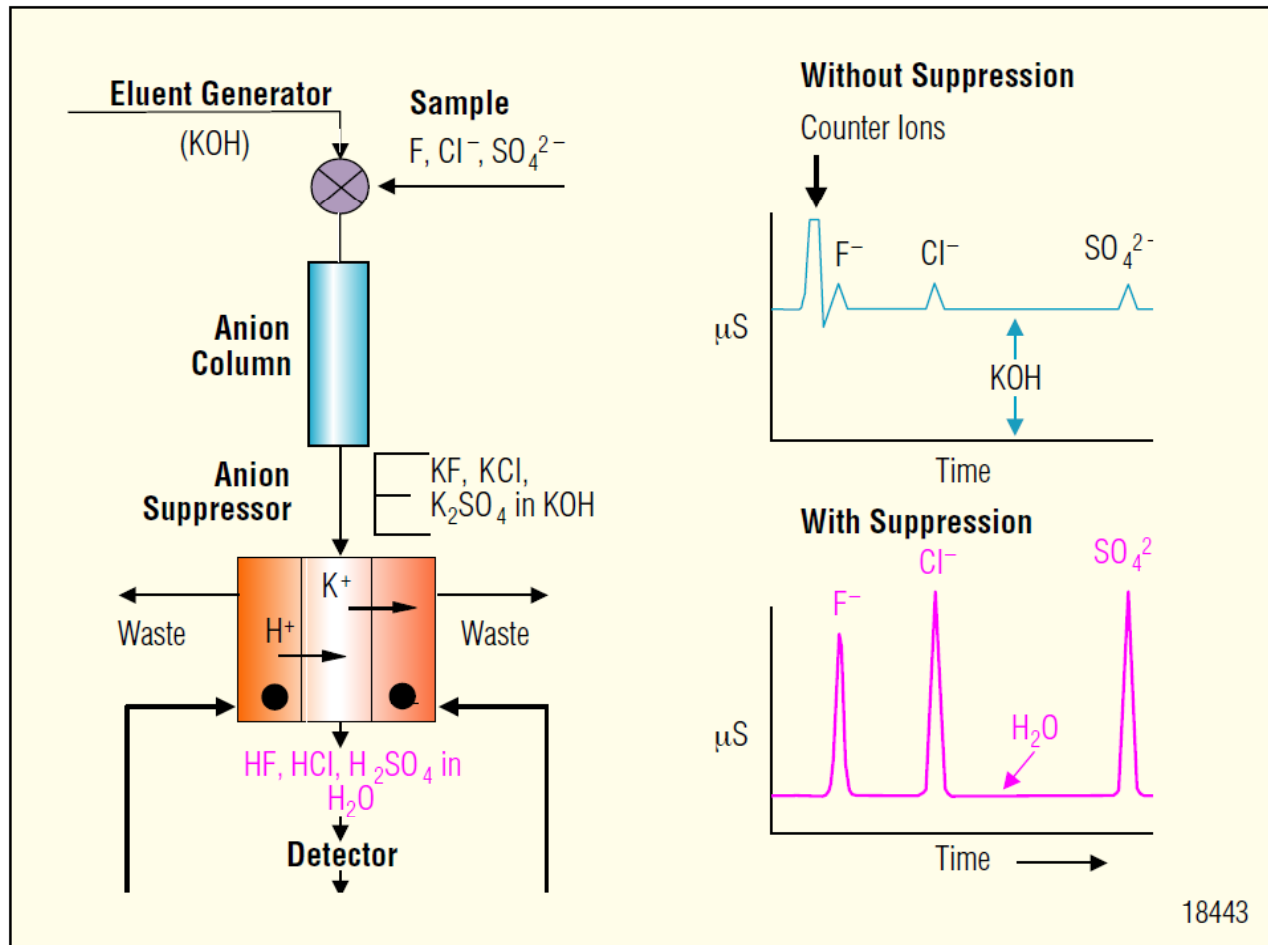
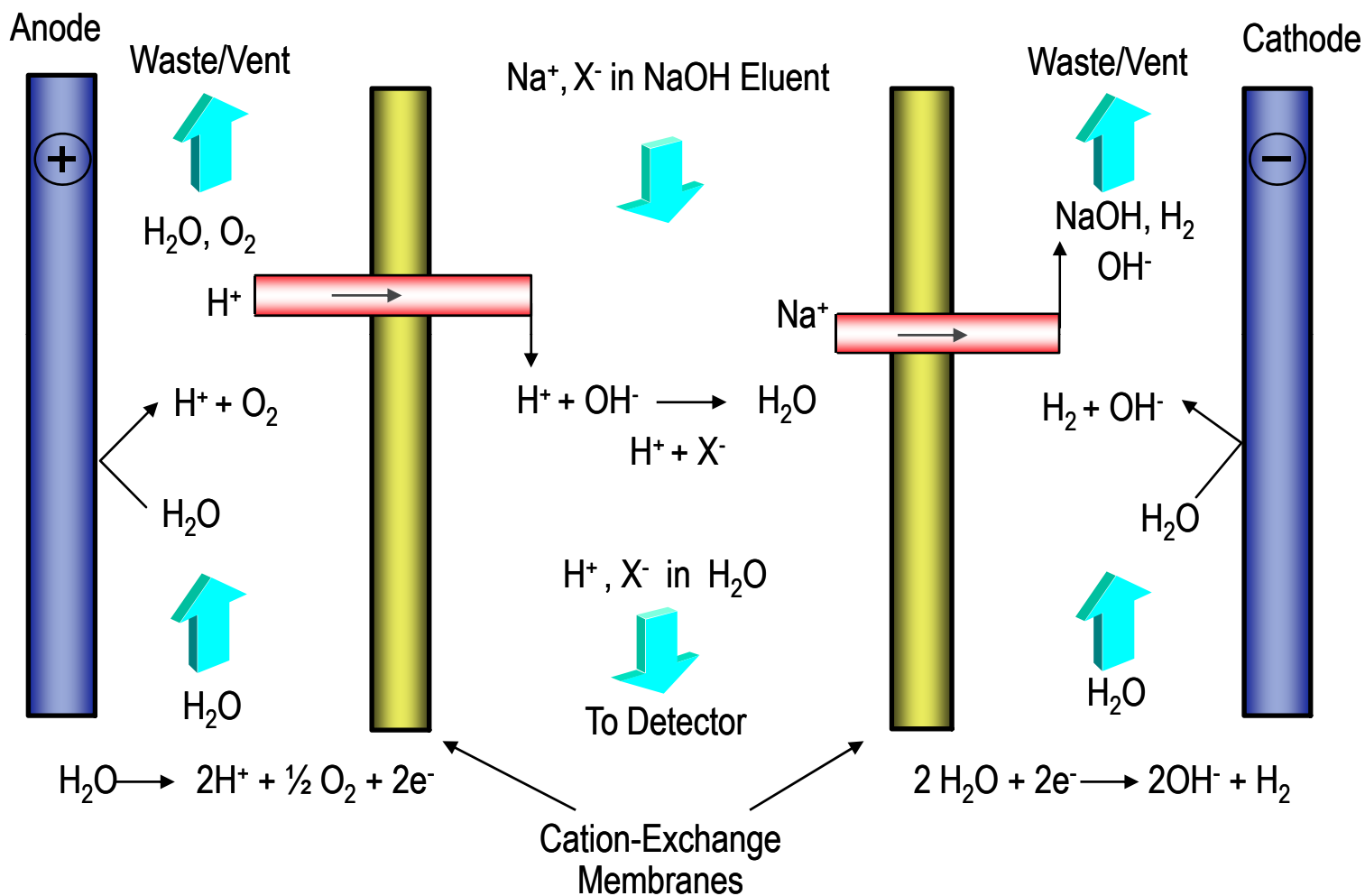


Diagram of eluent suppression for anion chromatography.

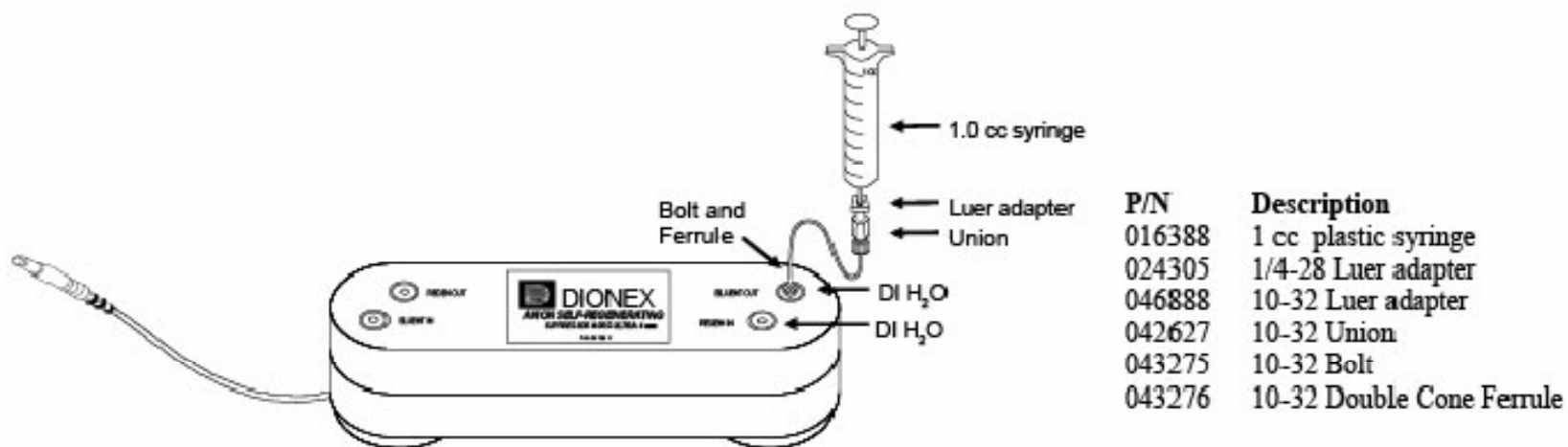
Anion Self-Regeneration System in AutoSuppression Mode



Suppressor Quick Start

Non-Capillary IC Systems ONLY

- Hydrating the suppressor ensures the ion exchange membranes are in a swollen form for proper operation.
- Push 3 mL of DI water through the ELUENT OUT port.
- Push 5 mL of DI water through the REGEN IN port.
- Allow the suppressor to sit for 20 minutes to ensure complete hydration.



Suppressor Care

- Always hydrate SRS prior to installation
- SRS suppressors should only be stored in DI Water
- Refer to the corresponding suppressor manuals for cleaning instructions

Conductivity Cell Care

- Conductivity cells require very little maintenance.
- Periodically...
 - check the liquid line connections for leaks
 - clean up any spills
 - remove the eluent with DI water and plug the cell-in and cell-out when no in use
- Air bubbles in the cell can cause pulsations of the baseline, random noise, and low readings.

NOTE: As important as it is to maintain backpressure on the cell, it is also necessary to keep from over-pressuring the suppressor. Cell backpressure should be maintained in the 40 - 100 psi range.

Column Care

Please Do...	Please Do NOT...
<ul style="list-style-type: none">• Store columns in Eluent• Plug columns when not in use• Clean with 10x stronger Eluent when needed• Protect columns from impact• Set pressure limits in Chromeleon• Use column manual and Dionex Customer Care Group for assistance. (1-800-DIONEX-0)	<ul style="list-style-type: none">• Store columns in plain water or harsh solvents• Leave columns open to air• Exceed manual's recommendations• Drop columns on floor or down stairs• Exceed maximum pressure specifications• Guess

Shutdown Procedures (Non-RFIC-EG Systems)

Short term

- When using an eluent of moderate to high salt concentration, rinse the fluid system with DI water for 30 minutes.
- Turn off the pump, suppressor power, and detectors.
- Leave the system power on.

Shutdown Procedures (Standard & Microbore IC)

Prolonged shutdown (> 1 week)

1. Prepare the columns for storage.
2. Remove the columns from the system and plug the ends to keep the column material from drying out.
3. Remove the EG cartridge, plug both ports, and store appropriately.
4. Remove the CR-TC, hydrate it, and plug all four ports.
5. Remove the suppressor, hydrate it, and plug the four ports to avoid drying out.
6. Couple the column inlet and column outlet lines with a union and flush the system with deionized water for 30 minutes.
7. Turn power off to all components.
8. Turn system power off.

Daily System and Column Logs

- Establishes the normal operating parameters of the system and the column
- Documents deviations from typical performance
- Alerts the user to potential problems and their causes, before they become catastrophic failures

Daily Log (Example)

Application: _____ Column Type: _____ Column S/N: _____

Week: _____	Sun	Mon	Tue	Wed	Thu	Fri	Sat
System pressure (psi)							
Signal background (μ S)							
Retention times (minutes)							
Analyte: _____							
Analyte: _____							
Analyte: _____							
Analyte: _____							
Analyte: _____							
Resolution Critical Pair							
Maintenance Performed							
Other Remarks							

Questions



ThermoFisher
S C I E N T I F I C