ThermoFisher SCIENTIFIC

Capillary IC – A New Platform for High Throughput or High Resolution Separations of Ionic Compounds

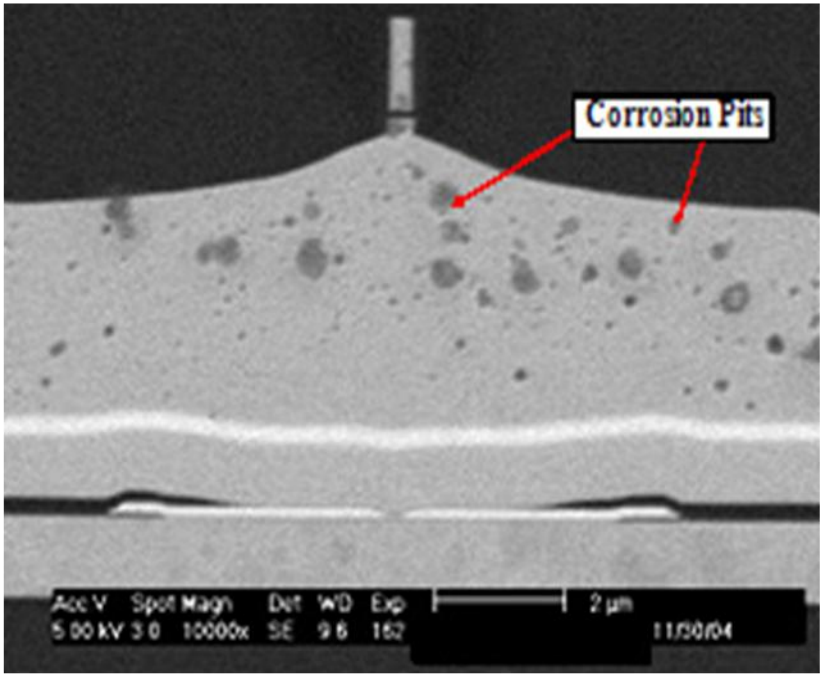
Peter Bodsky, Field Marketing

Flow Accelerated Corrosion (FAC) in PWR Power Plant Piping

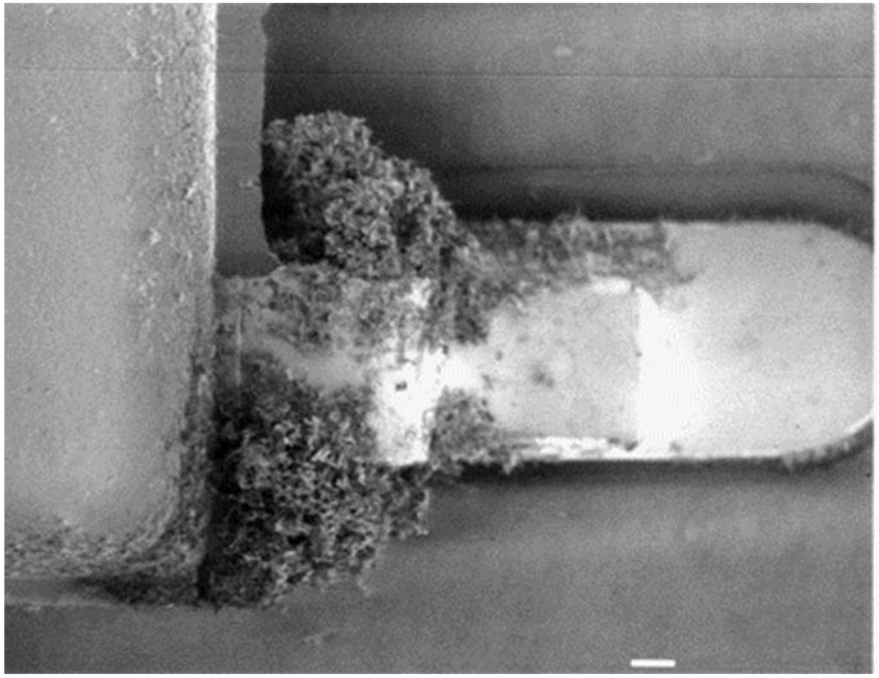
National Association of Corrosion Engineers
www.nace.org

Corrosion in the PWB and Device Industry

Corrosion Pitting in GMR Read/Write Head



Dendritic Growth in Device Lead



Foresite Laboratories
www.residues.com/picture_library.html

Outline

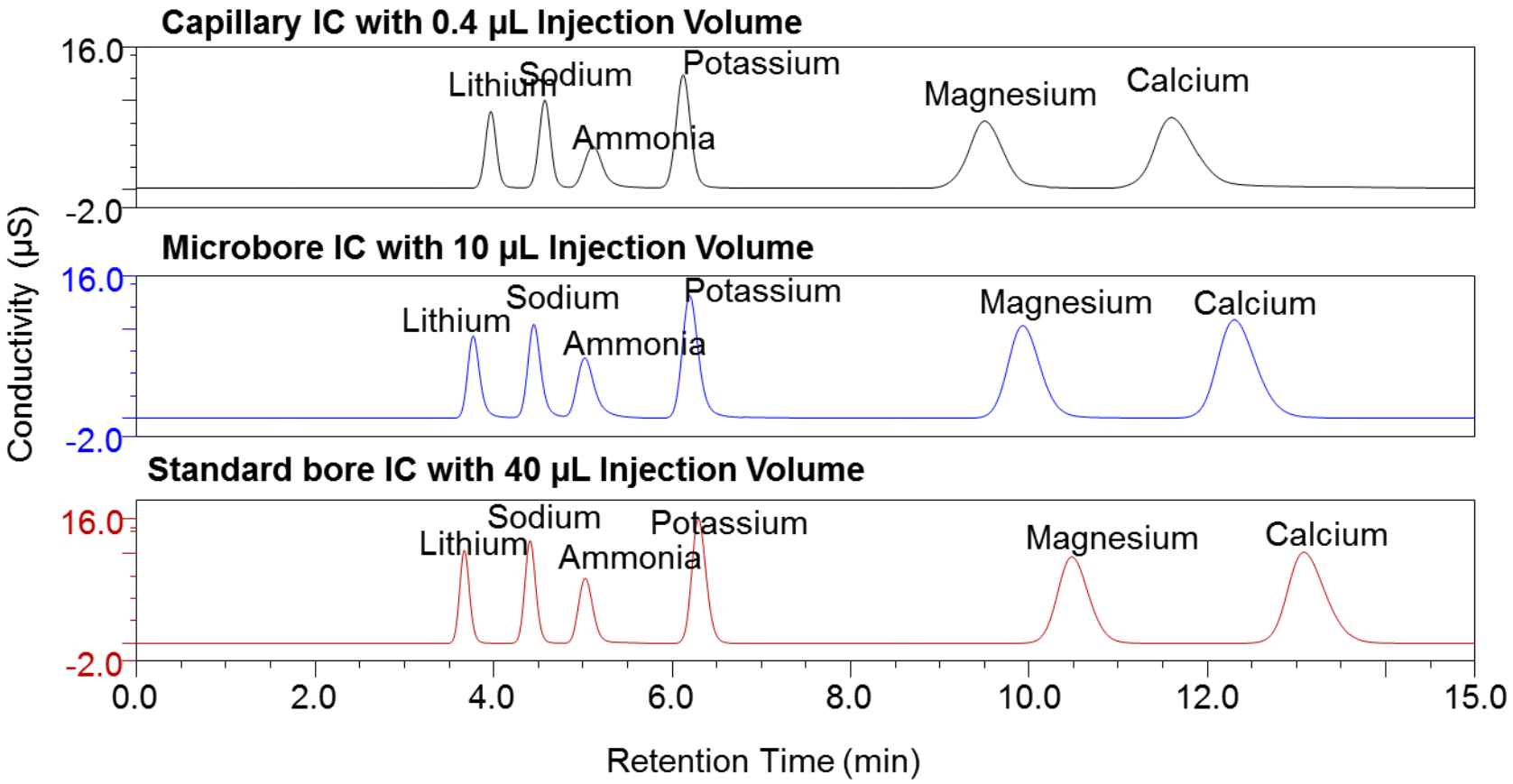
- Introduction to capillary IC and high pressure capillary IC
- Advantages of high pressure capillary IC
- Increased sensitivity for trace analysis
- Microliter volumes for large loop direct injections
- Microliter volumes for concentration
- Conclusions

The Most Important Values of Capillary IC

- **"IC on Demand"**
  - Permanent availability of the system
  - Higher laboratory productivity, reduced equilibration/start-up time
  - Less/fewer calibration runs
  - Isocratic and gradient elution with RFIC™ technology
- **Higher mass sensitivity**
  - High sensitivity with less sample volume
  - 100-fold increase in absolute sensitivity as compared to 4 mm systems
  - IC × IC (2D-IC) – detection limits in the ppt range with only 1 mL of sample
- **Lower cost of ownership**
  - Lower eluent consumption, less waste, 5.25 L/yr of DI water
  - 18 months lifetime of the EG cartridges

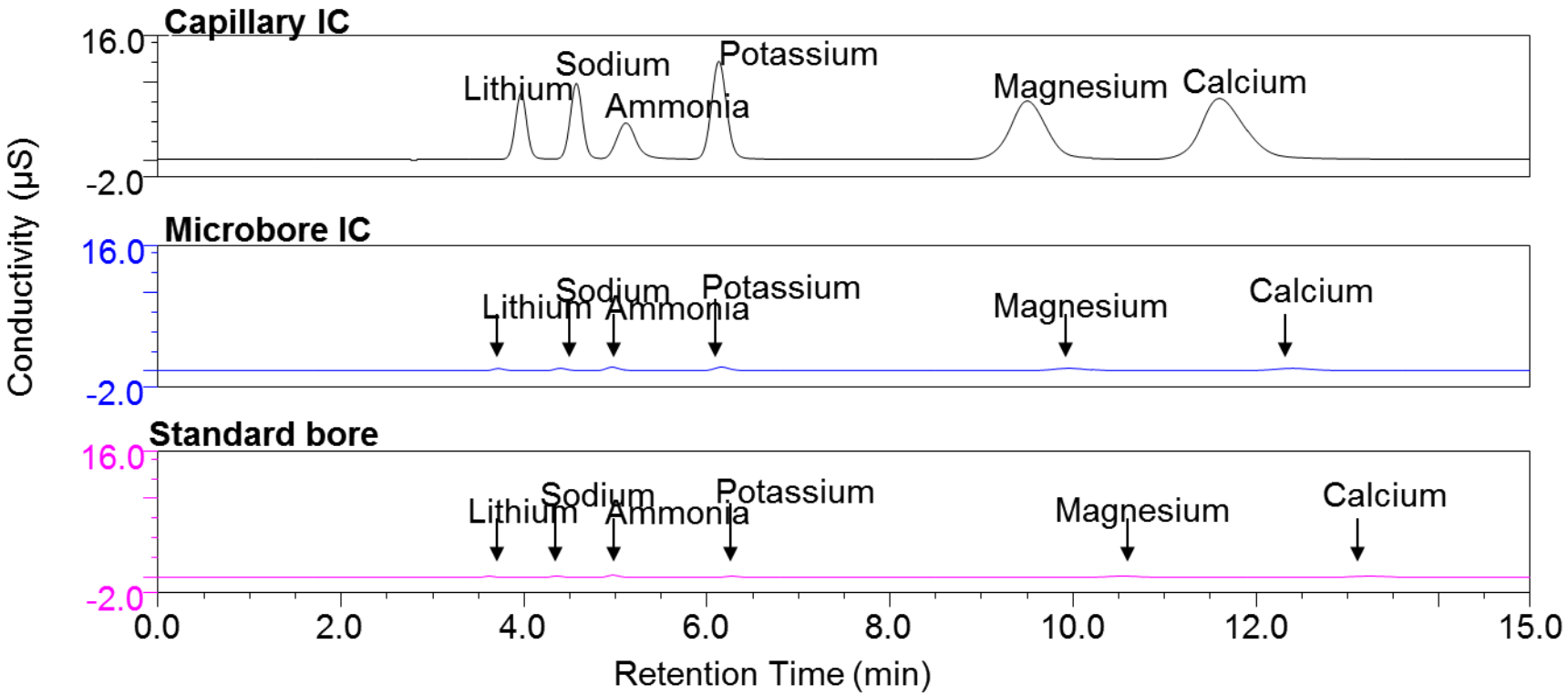
Capillary IC - Dimension of Scale

Overlay of chromatograms from 4 mm, 2 mm and 0.4 mm – all with optimum injection volume



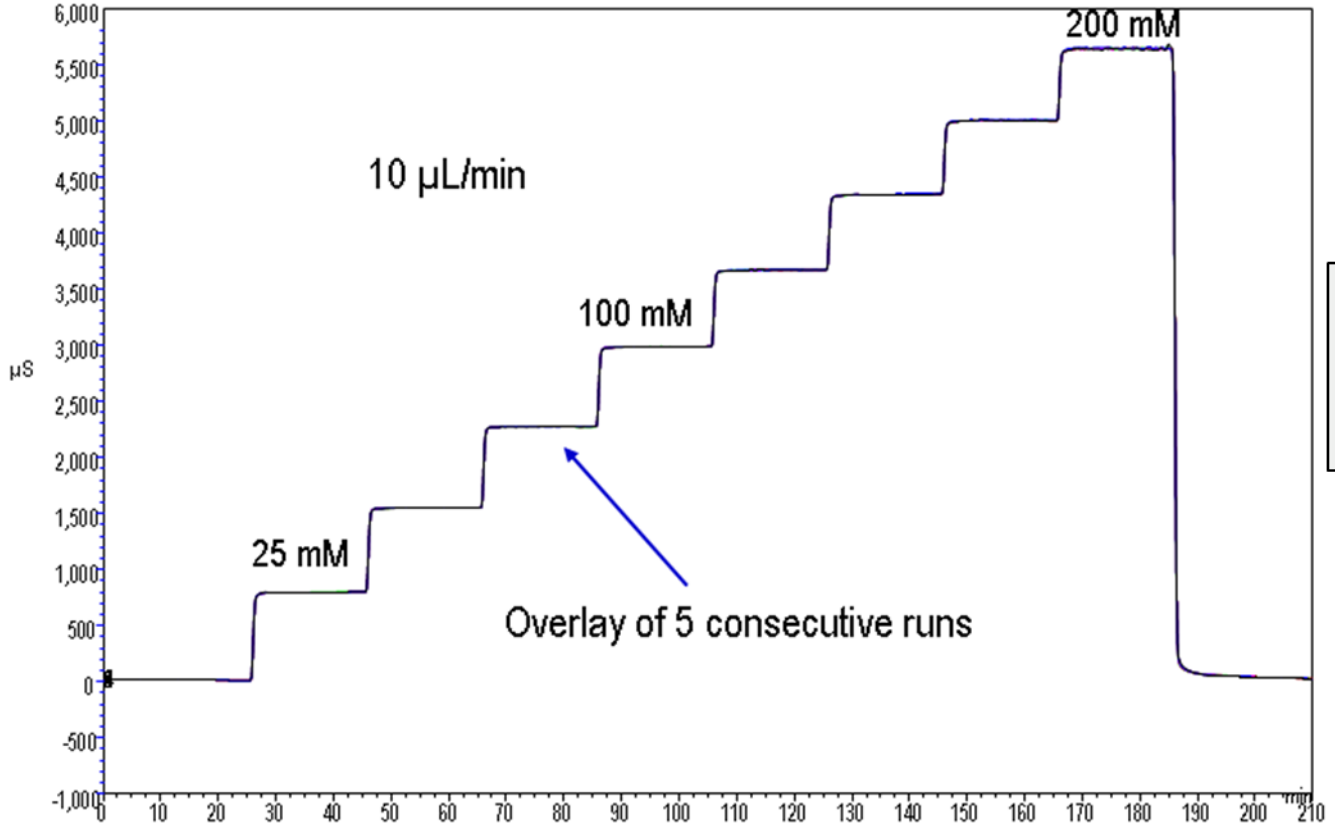
Capillary IC - Dimension of Scale

Overlay of chromatograms from 4 mm, 2 mm and 0.4 mm – all with same injection volume of 0.4 µL



Electrolytically Formed Gradients

- **EG step gradient – accuracy up to 200 mM in capillary mode**



$$[\text{Eluent}] \propto \frac{\text{Current}}{\text{Flow rate}}$$

What is High Pressure IC (HPIC™) ?

- **What it is:**
  - Continuous Operation at 5000 psi with a Capillary IC System

- **What it can do:**
  - Enables use of longer columns and/or higher flow rates

  - Longer columns => more efficient separations
  - Smaller particle size (4 µm) columns
  - Higher flow rates => faster analysis

High Pressure Capabilities

Why Do We Need Faster Separations?

- Make laboratories more productive
- Save laboratories time and solvents
- Provide faster answers to analytical questions
- Improve LODs and LOQs
- Rush samples

Faster separations are as accurate and precise as conventional methods!

Thermo Scientific™ Dionex™ ICS-5000+ HPIC System

Universal HPIC System

High-Pressure Ion Chromatography

- Continuous operation up to 5000 psi
- High pressure capable with both analytical and capillary systems
- Increased productivity with fast run times
- Improved separations and higher resolution with 4 µm particle columns

IC Cube™ Cartridge

HPIC - High Resolution, Fast Analyses

Dionex ICS-4000 Capillary HPIC System

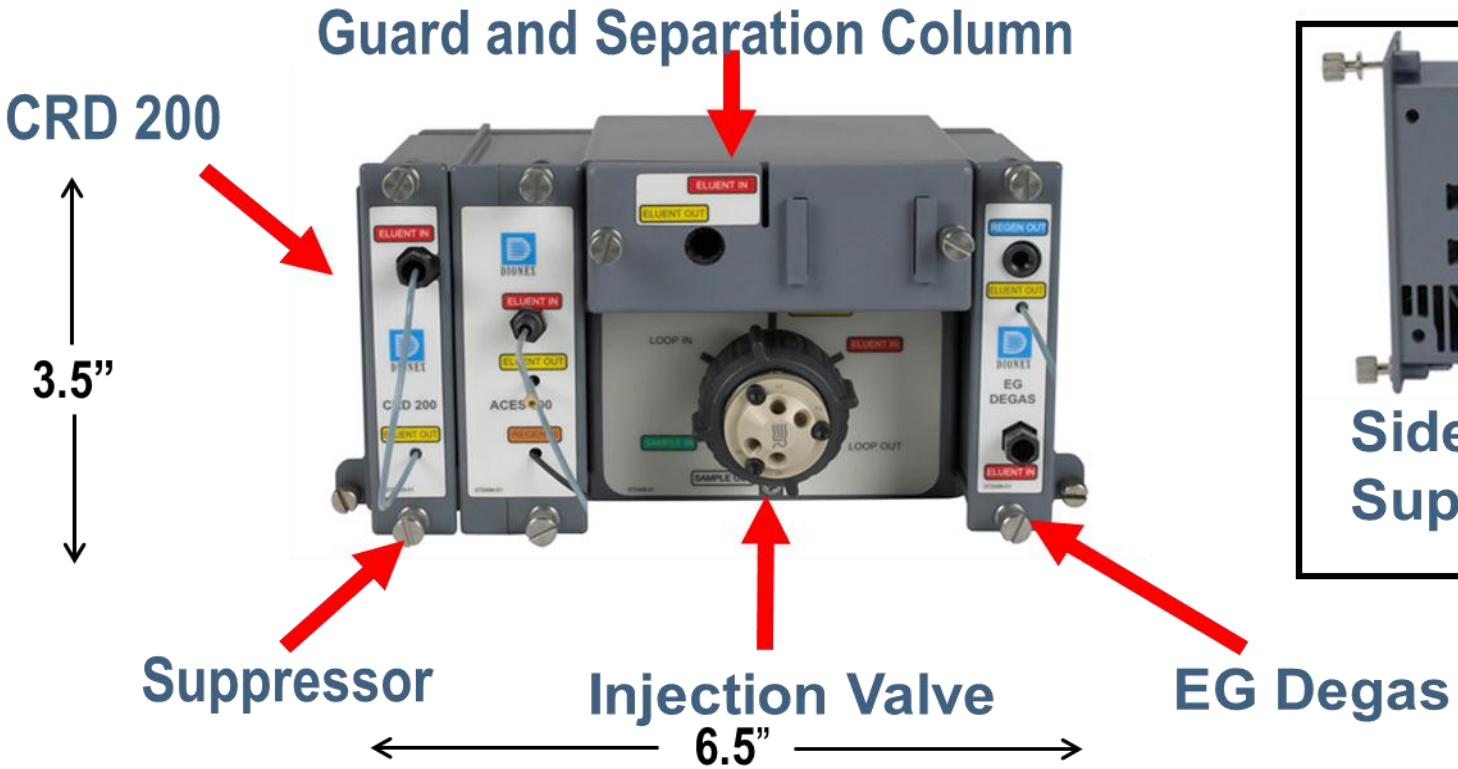
Dedicated Capillary HPIC

- New level of resolution and speed
- Delivering best in class sensitivity
- Simplifies workflows
- Increases analytical efficiency and productivity
- Small footprint
- Electrochemical, Conductivity, or Charge detection

IC Cube Cartridge

HPIC - High Resolution, Fast Analyses

Capillary Technology – The Dionex IC Cube



Dual Analytical	Dual Capillary
60 Fluidic Connections	**26 Fluidic Connections**

IC Cube – Columns and Column Compartment

Separator

Guard

Typical Workflow: Traditional IC

Dionex IonPac AS19 Gradient Analysis

Eluent Preparation

Total Time = 1 Hour

Start-up:
Equilibration:

Total Time = 1 Hour

Run 3 Standards:

Total Time = 1 h 30 min

Run 5 Samples:

Total Time = 2 h 30 min

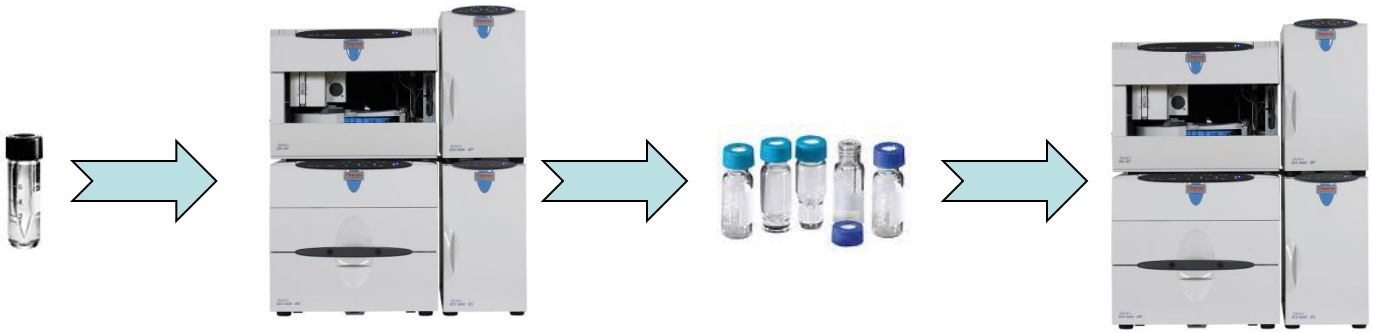
Results

Total Analysis Time:

Total Time = 6 hours

Typical Workflow: Capillary IC

Always On – Always Ready



Run Check Standard:

Total Time = 30 min

Run 5 Samples:

Total Time = 2 h 30 min

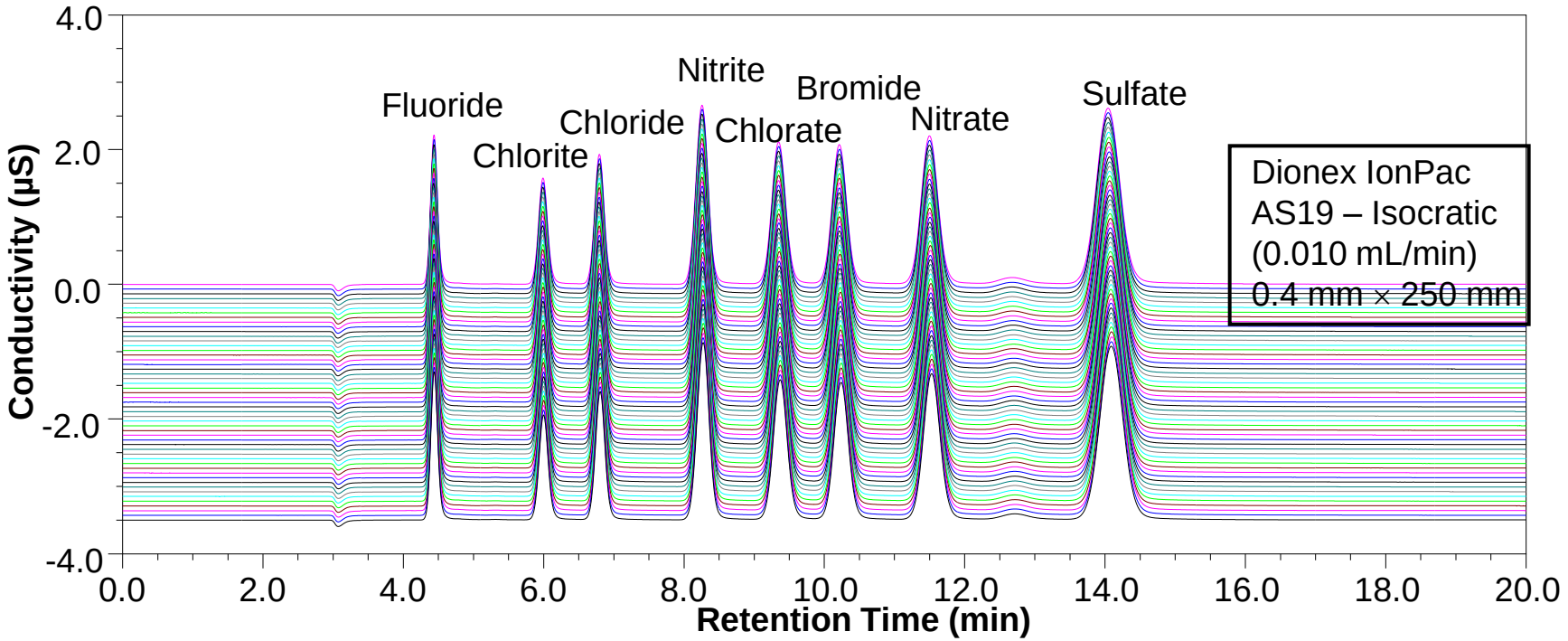
Results

Total Analysis Time:

Total Time = 3 hours

"IC on Demand"

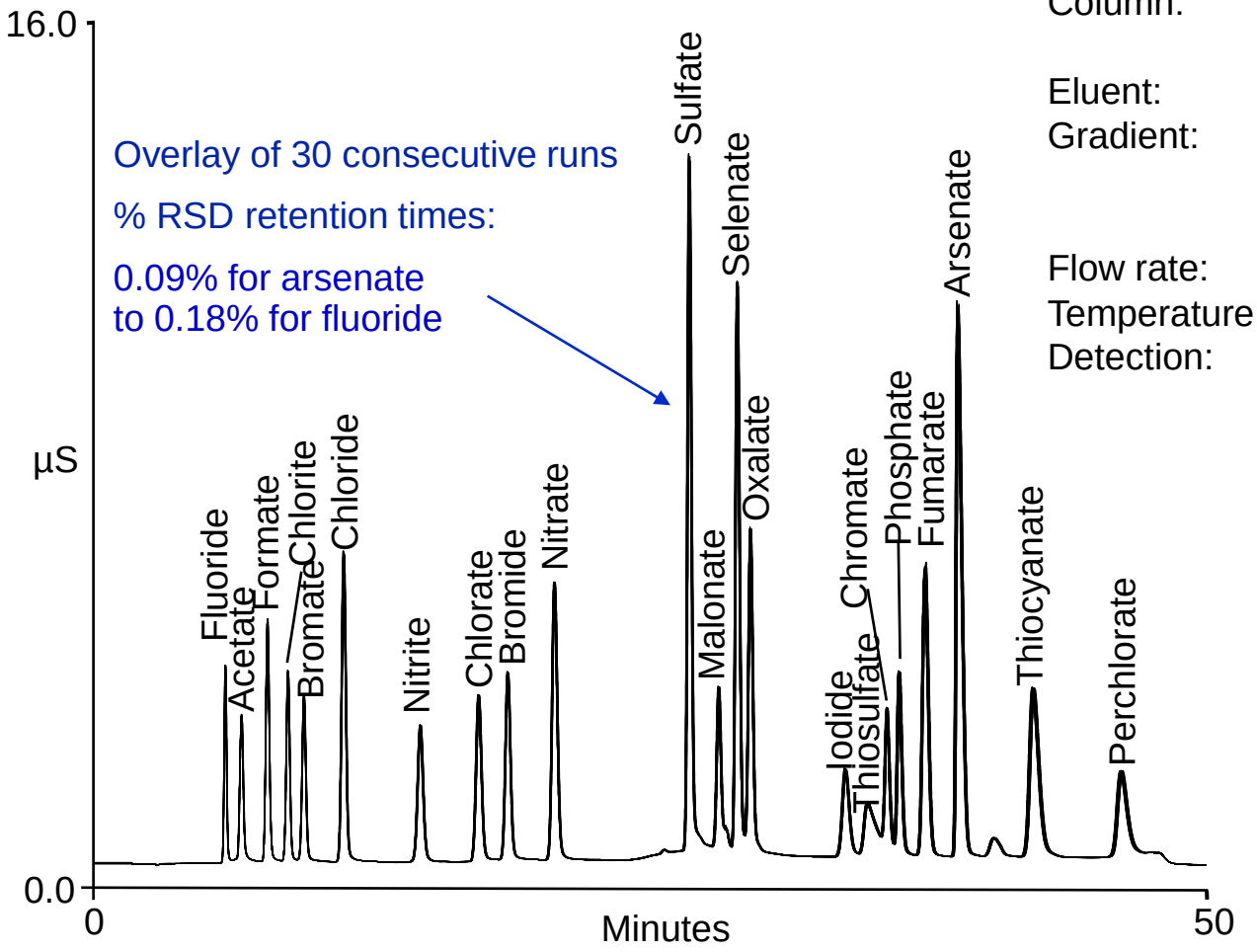
Capillary IC Value: Best Reproducibility



	Fluoride	Chlorite	Chloride	Nitrite	Chlorate	Bromide	Nitrate	Sulfate
RT (% RSD)	0.048	0.045	0.037	0.030	0.023	0.024	0.021	0.026
Peak Area (% RSD)	0.287	0.363	0.367	0.328	0.349	0.359	0.354	0.287

Overlay of 50 Consecutive Analysis

Gradient Separation of 22 Anions



Column:	Dionex IonPac AS19 250 mm × 0.4 mm
Eluent:	EGC-KOH capillary cartridge)
Gradient:	10 mM (0 to 10 min), 10–52 mM (10 to 42 min), 52–70 mM (42 to 45 min)
Flow rate:	0.010 mL/min
Temperature:	30 ° C
Detection:	Suppressed conductivity, Dionex ACES 300, recycle

Trace Analysis with Capillary IC

- Smaller sample volumes required for same sensitivity
- A 10-µL injection onto a 0.4 mm i.d. column is equivalent to a 1000-µL injection onto a 4 mm i.d. column

- Measurements possible at lower concentrations than before
- Suitable for weakly contaminated samples

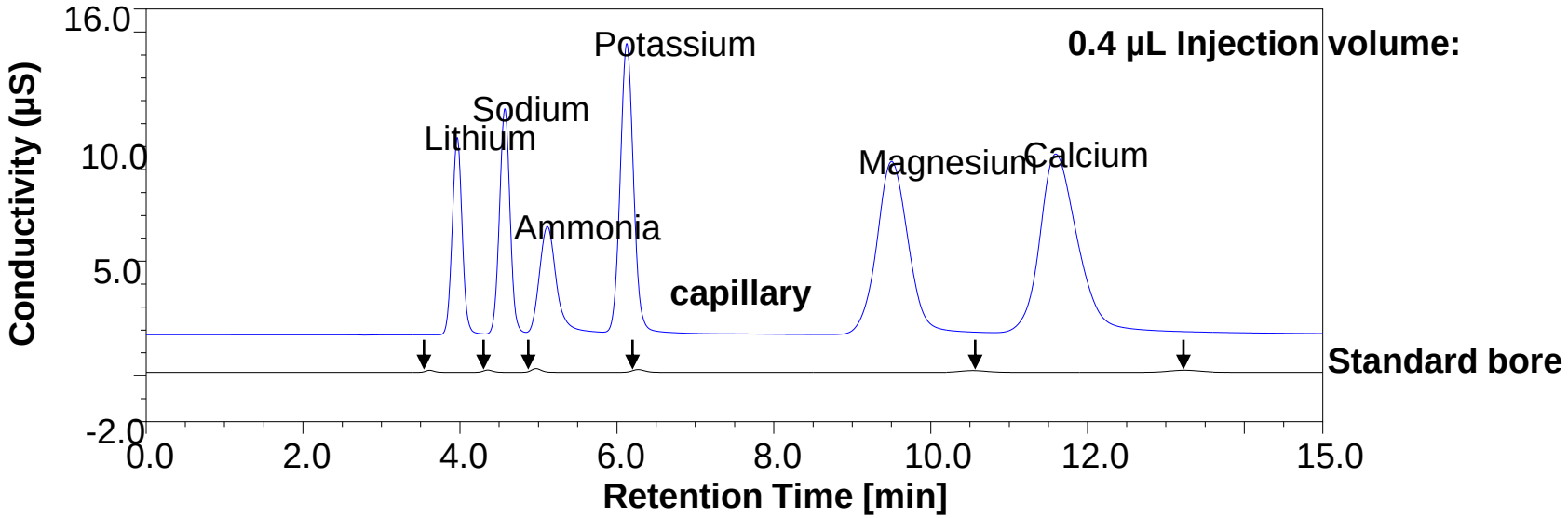
- Shorter loading time in concentration mode
  - 250-µL sample onto a capillary concentrator by a Dionex AS-AP autosampler

  Vs

  - 25-mL sample onto a conventional concentrator by a Dionex AS-HV autosampler

Capillary IC – Values: Small Sample Sizes

- Limited Volumes:
  - Precious/Valuable samples
  - Corrosion detection
- Safety:
  - Toxic/Dangerous samples
- Reduced Waste Cost:
  - Less overall waste to dispose of…. i.e. Radioactive waste

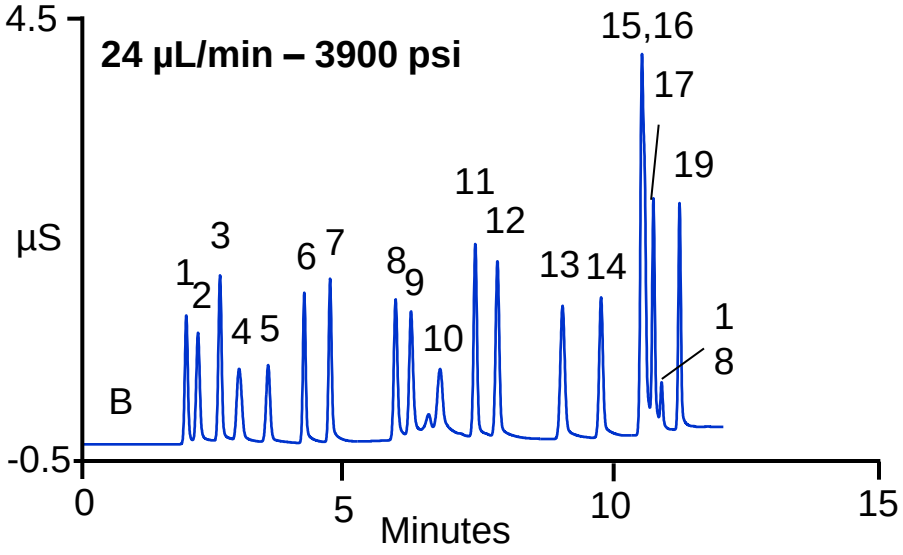


High Pressure Capabilities of Capillary IC

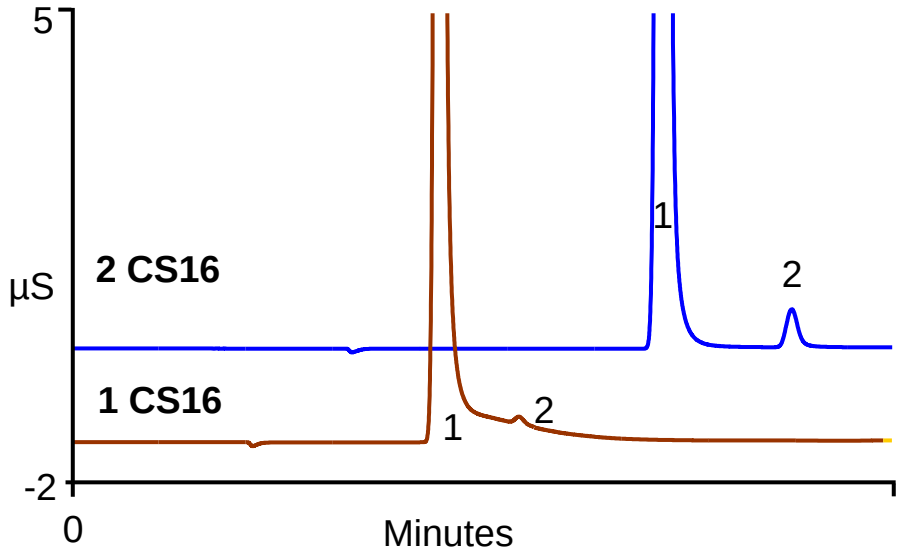
Expanded Capability:

- **Capillary IC systems can now operate at higher pressures**
  - Up to 5000 psi, in continuous operation, and with RFIC-EG
- **Faster separations with higher flow rates (left)**
- **Higher resolution with longer columns (right)**

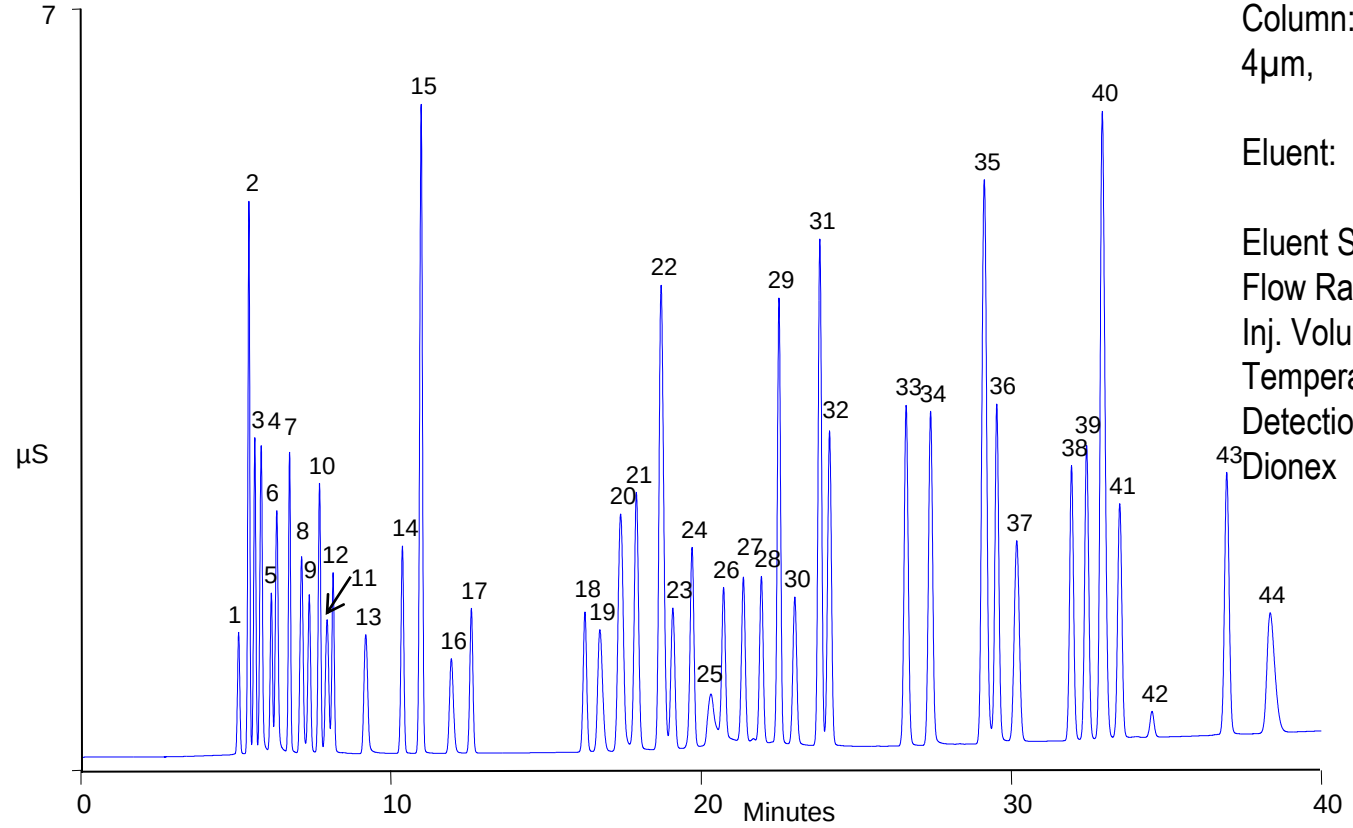
IonSwift MAX-100: 11 minutes



10,000:1 Na : Ammonia



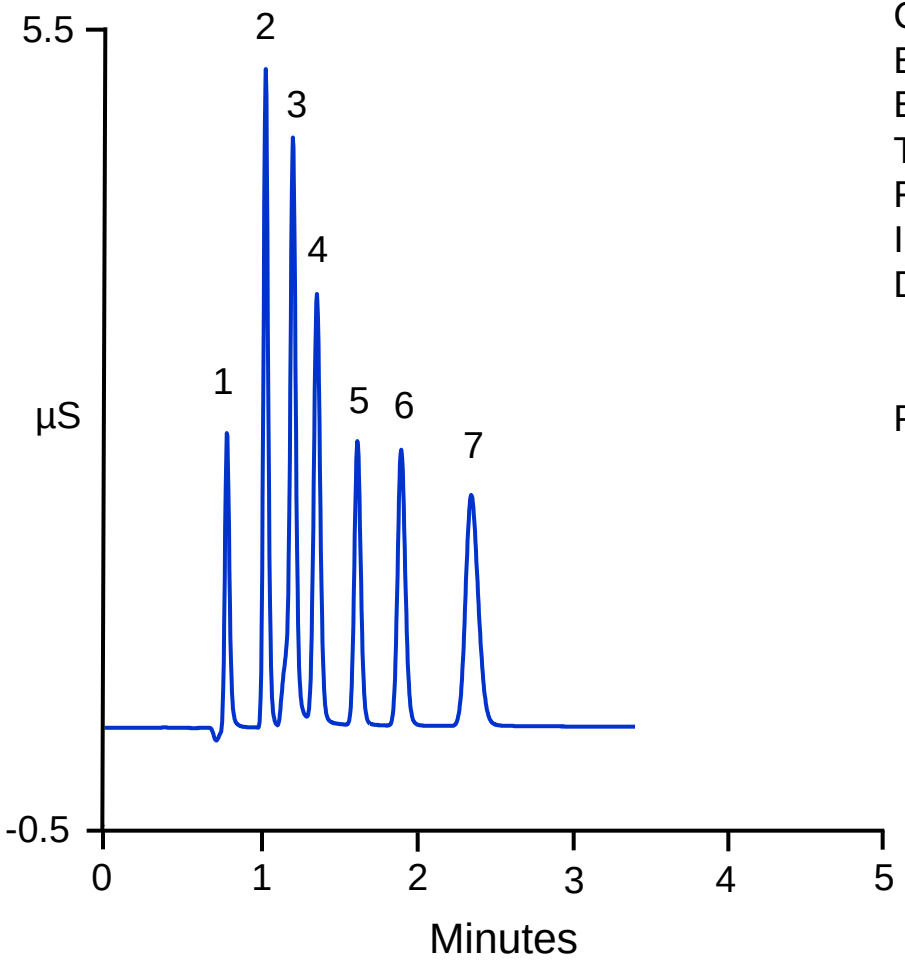
Separation of 44 Analyte Peaks!



Column:	Dionex IonPac AS11-HC-4µm, 0.4 × 250 mm
Eluent:	1–14 mM KOH in 16 min, 14–55 mM KOH in 24 min
Eluent Source:	EGC-KOH capillary cartridge
Flow Rate:	0.015 mL/min
Inj. Volume:	0.4 µL
Temperature:	30 ˚C
Detection:	Suppressed conductivity, Dionex ACES™ 300, 24 mA, recycle mode

1. Quinate
2. Fluoride
3. Lactate
4. Acetate
5. 2-hydroxybutyrate
6. Propionate
7. Formate
8. Butyrate
9. 2-hydroxyvalerate
10. Pyruvate
11. *iso*-Valerate
12. Chlorate
13. Valerate
14. Bromate
15. Chloride
16. 2-Oxovalerate
17. Nitrite
18. Ethylphosphonate
19. Trifluoroacetate
20. Azide
21. Bromide
22. Nitrate
23. Citramalate
24. Malate
25. Carbonate
26. Maleate
27. Citraconitate
28. Maleate
29. Sulfate
30. α-ketoglutarate
31. Oxalate
32. Fumarate
33. Oxaloacetic
34. Wolframate
35. Molybdate
36. Phosphate
37. Phthalate
38. Arsenate
39. Citrate
40. Chromate
41. *iso*-Citrate
42. *cis*-Aconitate
43. *trans*-Aconitate
44. Iodide

Fast Runs on the Dionex IonPac AS18-4µm Column



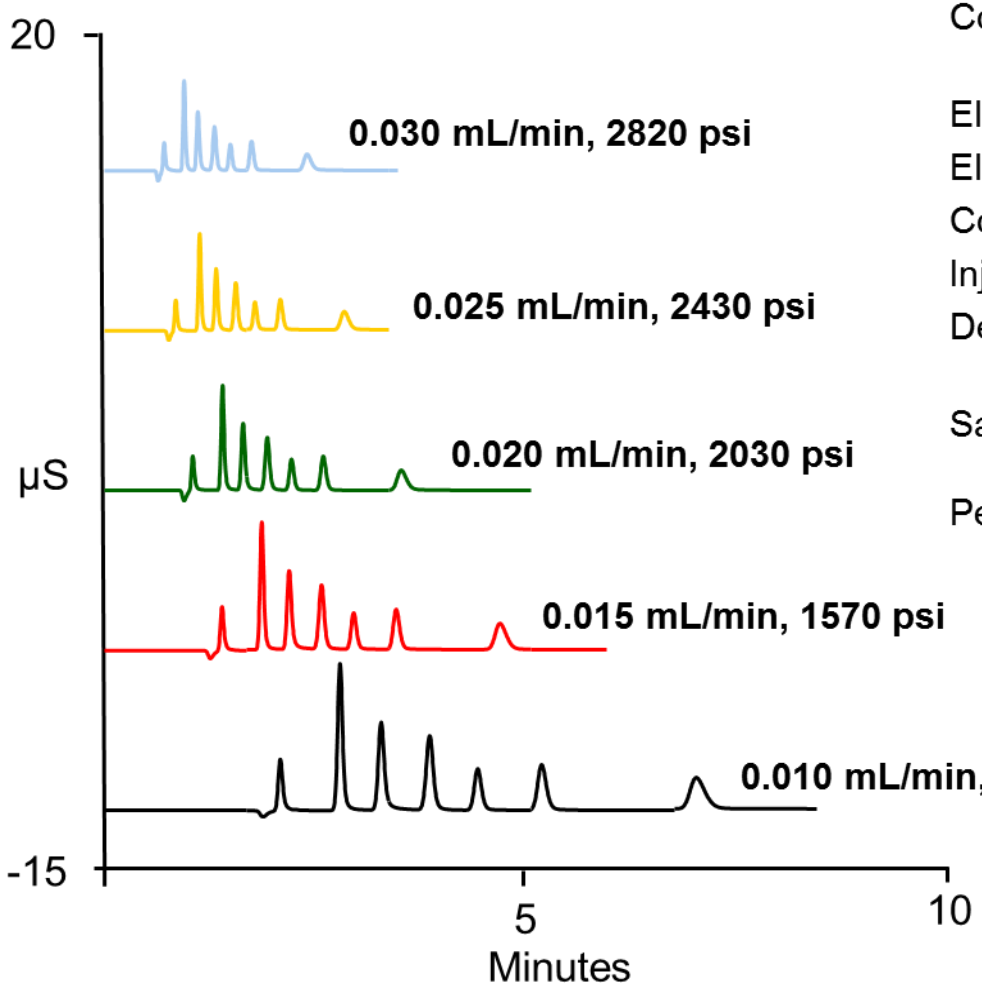
Column:	Dionex IonPac AS18-4µm, 0.4 mm
Eluent:	35 mM KOH
Eluent Source:	EGC-KOH capillary cartridge
Temperature:	30 ° C
Flow Rate:	0.030 mL/min
Inj. Volume:	0.4 µL
Detection:	Suppressed conductivity, Dionex ACES™ 300, recycle mode

Peaks:

1. Fluoride	0.2	mg/L
2. Chloride	0.5	
3. Nitrite	1.0	
4. Sulfate	1.0	
5. Bromide	1.0	
6. Nitrate	1.0	
7. Phosphate	2.0	

Speed

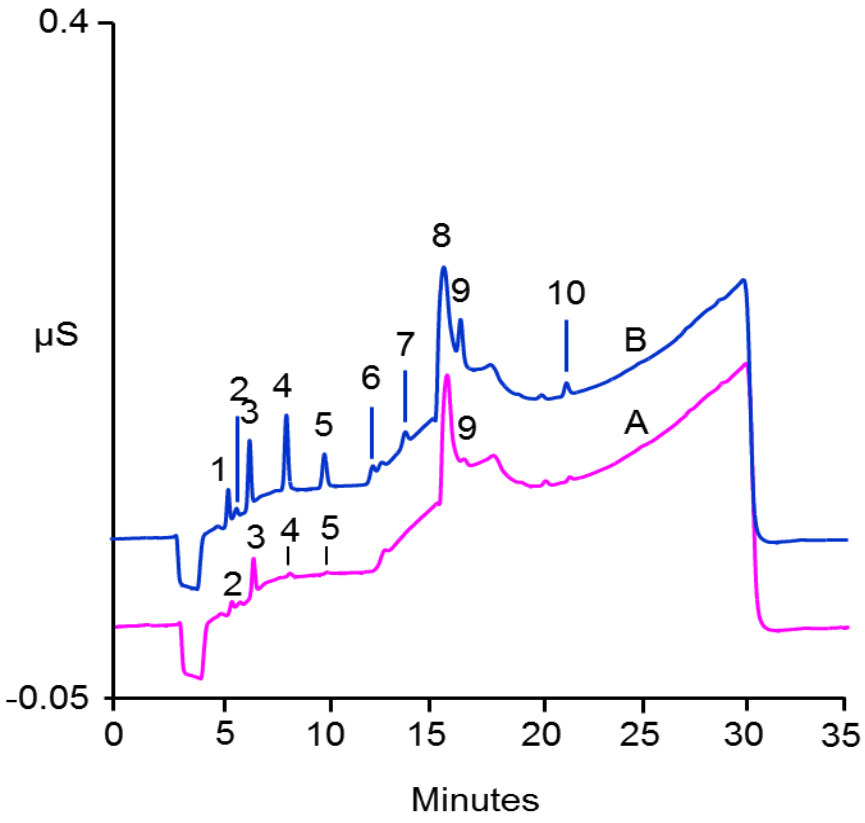
Separation of inorganic anions using a 4 µm column at different flow rates.



Column:	Thermo Scientific Dionex IonPac AS18-4µm, 0.4 × 150 mm
Eluent Source:	Dionex EGC-KOH (Capillary)
Eluent:	30 mM KOH
Col. Temp.:	30 °C
Inj. Volume:	0.4 µL
Detection:	Suppressed Conductivity, Dionex ACES 300
Sample:	Anion Standard Solution

Peaks:		
1. Fluoride	0.2	mg/L
2. Chloride	1	
3. Nitrite	1	
4. Sulfate	1	
5. Bromide	1	
6. Nitrate	1	
7. Phosphate	2	

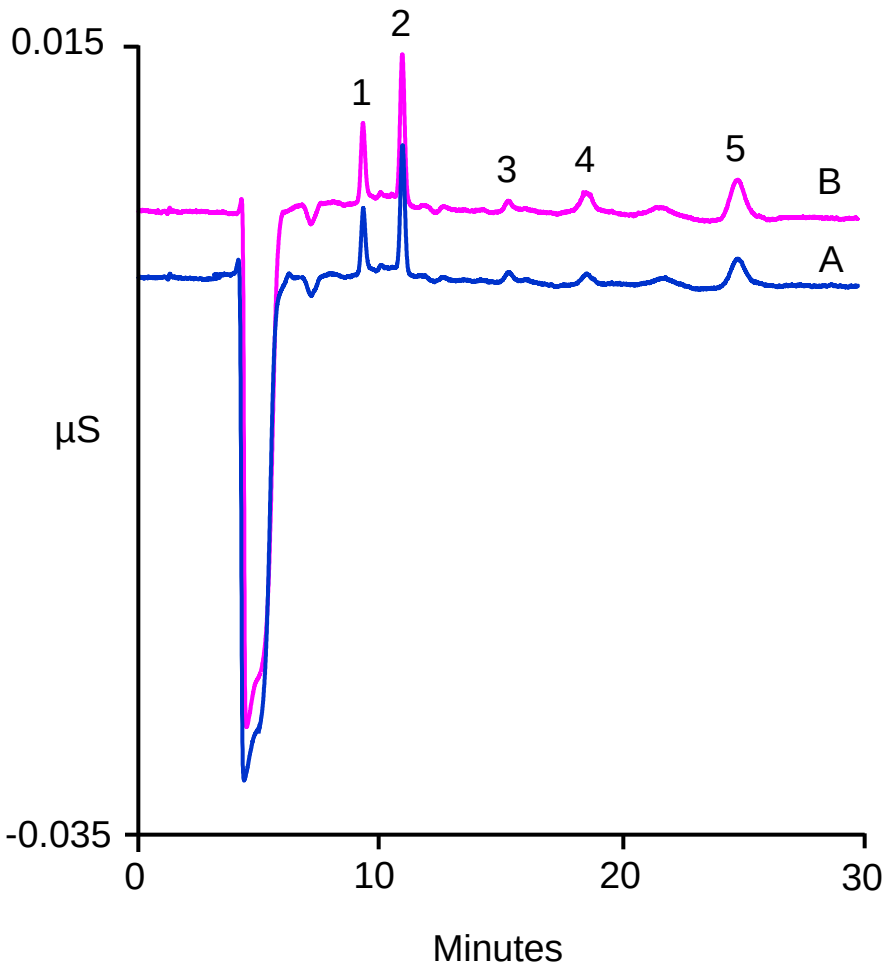
Anions - Large Loop Direct Injection of 10 µL



Column:	Dionex IonPac AG19, AS19, 0.4 x 250 mm
Eluent Source:	Dionex EGC-KOH capillary
Eluent:	14 mM KOH for 7 min, 14–45 mM (7–25 min), 14 mM (25.1–35 min)
Flow Rate:	0.010 mL/min
Col. Temp.:	30 °C
Inj. Volume:	10 µL
Detection:	Suppressed conductivity, Dionex ACES™, Dionex CRD-200 capillary
Samples:	A: Blank B: Diluted anion standard

Peaks:	A	B	
1. Fluoride	--	0.2	µg/L
2. Acetate	--	--	
3. Formate	--	--	
4. Chloride	0.045	1.0	
5. Nitrite	--	1.0	
6. Bromide	--	1.0	
7. Nitrate	--	1.0	
8. Carbonate	--	--	
9. Sulfate	0.002	1.0	
10. Phosphate	0.002	2.0	

Cations - Large Loop Direct Injection of 10 µL



Column:	Dionex IonPac™ CG16, CS16, capillary, 0.4 × 250 mm
Eluent Source:	Dionex EGC-MSA capillary
Eluent:	30 mM MSA
Flow Rate:	0.010 mL/min
Col. Temp.:	30 ° C
Inj. Volume:	10 µL
Detection:	Suppressed conductivity, Dionex CCES™, recycle mode
Samples:	Blank
Vial Septa:	**A: Teflon Single Injection** **B: Blue Septa**

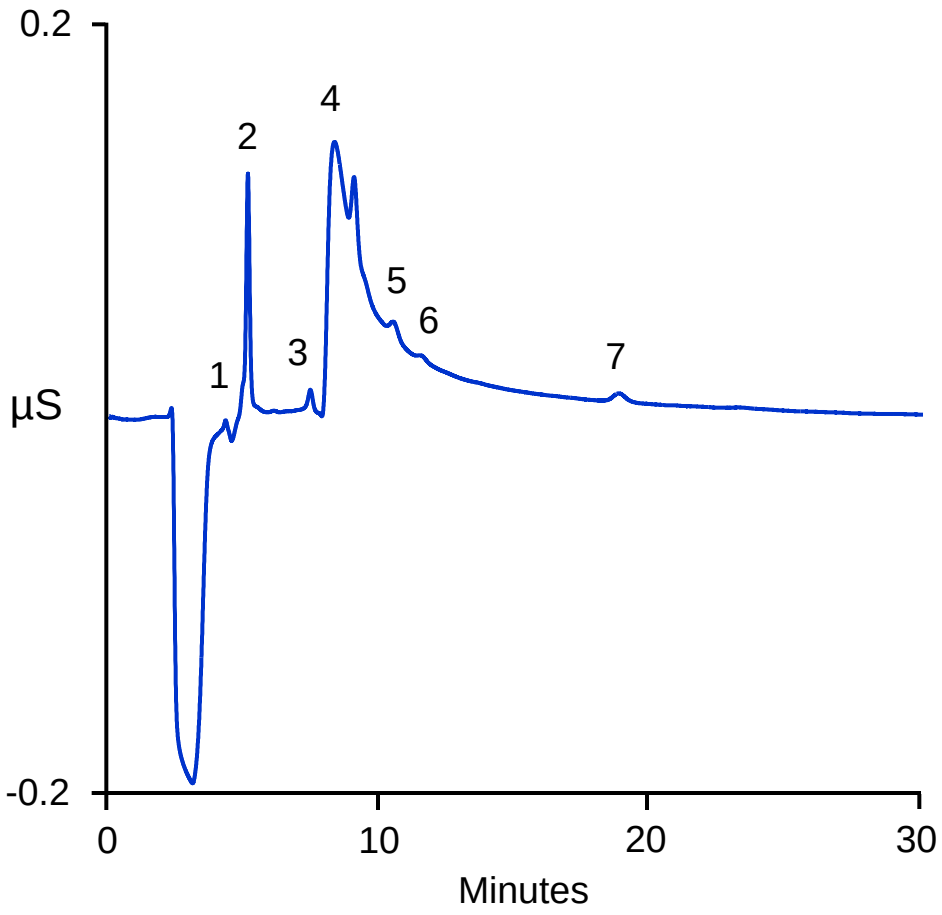
Peaks:	A	B
1. Sodium	0.057	0.068µg/L
2. Ammonium	0.21	0.32
3. Potassium	0.024	0.016
4. Magnesium	0.011	0.023
5. Calcium	0.068	0.11

Once we have clean water, how to we keep it clean?

Solution! ICW-3000 Online Water Purifier

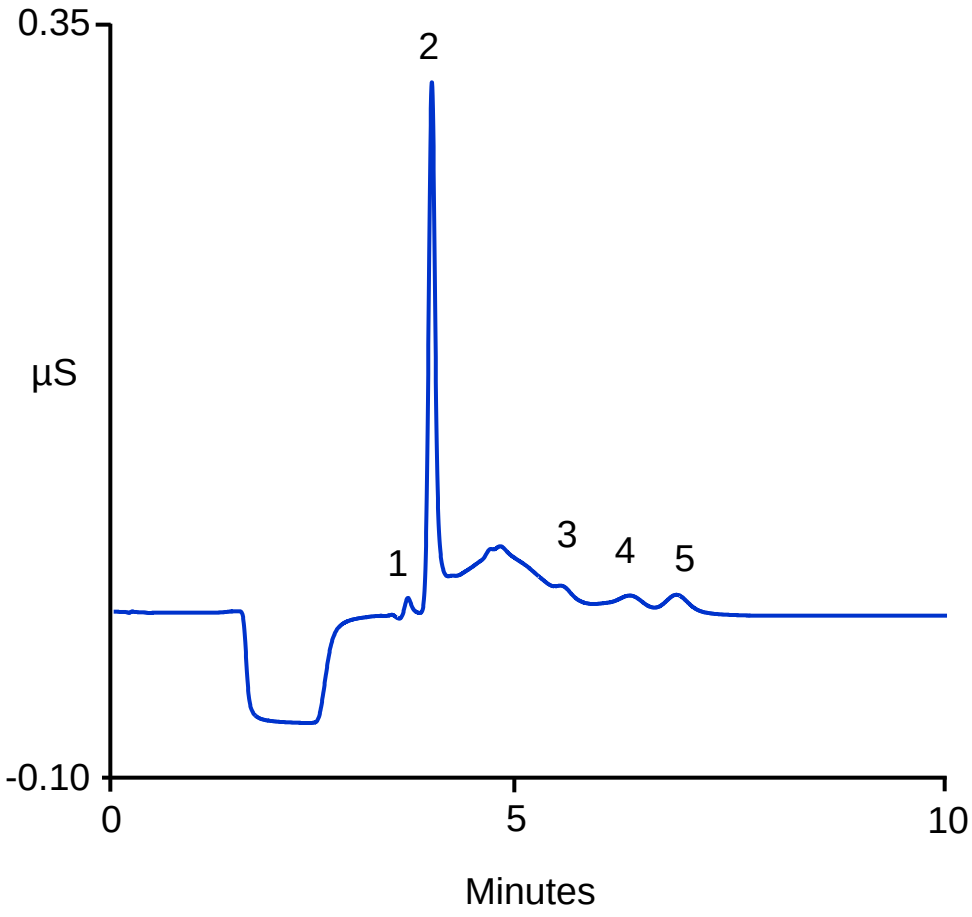
- Millipore product
- Designed specifically and only for Dionex RFIC-EG systems
- Online water supply-ports on back for Eluent and Suppressor
- Support two standard bore systems

Trace Anions by Concentrating 200 µL



Column:	Dionex IonPac AG15, AS15, 0.4 mm
Eluent Source:	Dionex EGC-KOH capillary, **ICW-3000 Online Water Purifier**
Eluent:	30 mM KOH
Flow Rate:	10 µL/min
Column Temp.:	30 ° C
Detection:	Suppressed conductivity, Dionex ACES™, **external water by ICW-3000 Online Water Purifier,** Dionex CRD 200 capillary
Inj. Volume:	200 µL
Concentrator:	IonSwift™ MAC-100
Autosampler:	**No flush container, ICW-3000**
Vial Septa:	**Teflon Single Injection "Septa"**
Peaks:	1. Fluoride 11 ng/L
	2. Acetate --
	3. Chloride 26
	4. Carbonate --
	5. Sulfate 22
	6. Oxalate --
	7. Nitrate 26

Cation Blank Using ICW-3000 Online Water Purifier



Column:	Dionex IonPac™ CG12A, CS12A, 0.4 x 250 mm
Water Source:	**ICW-3000**
Eluent Source:	Dionex EGC-MSA capillary
Eluent:	20 mM MSA
Flow Rate:	0.015 mL/min
Column Temp.:	30 ° C
Detection:	Suppressed conductivity, Dionex CCES™, **external mode by ICW-3000 Online Purifier**
Concentrator:	Dionex IonSwift™ MCC-100
Inj. Volume:	200 µL
Sample:	Blank
Autosampler:	**No flush container, ICW-3000**
Vials:	**Single Injection PTFE "Septa"**

Peaks:	1.	Sodium	< 20 ng/L
	2.	Ammonium	~100
	3.	Potassium	< 20
	4.	Magnesium	< 20
	5.	Calcium	< 20

Summary

High pressure capillary IC allows you to

- Lower operational costs – water, waste, consumables
- "Always Ready"
  - Improved system and analytical performance
  - Convenience
  - Rush samples
- Enables high-resolution separations and Fast IC separations using new 4 µm particle columns
- Expand separation capabilities
  - Smaller particle columns
  - Faster flow rates
  - Higher eluent concentrations using RFIC chemistry

Capillary IC is a quantum leap ahead!

Thank you!

ThermoFisher
SCIENTIFIC

The world leader in serving science