Peter Bodsky

Field Marketing Manager
Thermo Fisher Scientific
Peter.Bodsky@ThermoFisher.com

Biography:
Peter is the Field Marketing Manager for Thermo Fisher Scientific - Dionex Products including the IC, LC, and ASE product lines. In his tenure with Dionex, he has held various management positions including Global Technical Support, North American Field Service Management, Senior IC Product Management, and Regional Field Sales. Peter earned his Bachelor's Degree in Chemistry from the University of Washington and immediately began work in the food industry (American Maize Products) where he developed methods for the larger scale production and purification of High Fructose Corn Syrup for use in beverages and food products. The tools used in these pilot scale projects included HPLC, IC, NIR, and GC. Dionex Ion Chromatography was used extensively in this work for qualification and quantification of the carbohydrate components of the sweetener product. Peter joined Dionex in 1986 and is based out of Sunnyvale, California.

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

Executive Summary:

There has been increasing interest in the development of capillary ion chromatography (IC) systems and methods for determination of ionic species. The practice of ion chromatography in capillary format offers a number of advantages. Because the eluent consumption is very low, capillary IC systems can be operated continuously and thus are always on and always ready for analysis. Capillary IC systems offer improved compatibility with applications where amount of sample is limited. Capillary IC systems provide improved performance for determination of target analyses at trace levels. The use of capillary columns can improve separation efficiency and/or speed. The operation of capillary IC systems at low flow rates improves the system compatibility with a mass spectrometer. In addition, the use of capillary separation columns opens the door for the possibility of offering new selectivity for difficult applications using new columns packed with stationary phases which are more costly and difficult to prepare.

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

IPC Midwest
August, 2012

Outline

- Introduction to Capillary IC
  - The dimension of scale
  - Top values
- Impact on trace analysis
- Higher back pressure tolerance of Capillary IC
  - Impact on sample throughput
  - Impact on chromatographic resolution
- Conclusions

The Most Important Values of Capillary IC

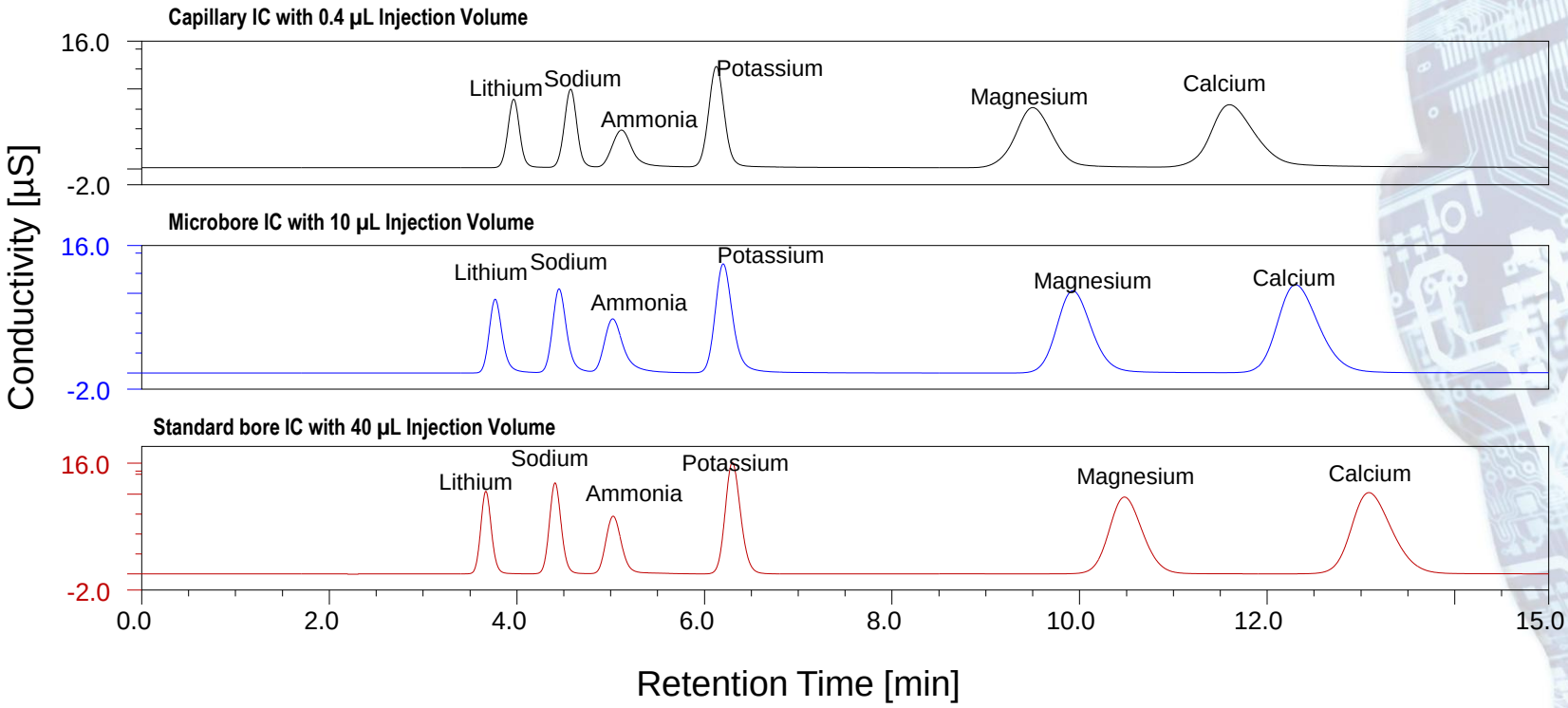
- "**IC on Demand**"
  - Permanent availability of the system
  - Higher laboratory productivity, reduced equilibration/start-up time
  - Less/fewer calibration runs
  - Isocratic and gradient elution with RFIC
- **Higher mass sensitivity**
  - High sensitivity with less sample volume
  - 100-fold increase in absolute sensitivity in comparison 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
  - 18 months lifetime of the EG cartridges
  - Operates on just 5.25 L of DI water per year

Capillary IC – The Dimension of Scale

	Analytical		Capillary
Column I.D.	4 mm		0.4 mm
Flow Rate	1.0 mL/min		10 μL/min
Injection Volume	25 μL		0.4 μL
Eluent Consumption / Waste Generated	43.2 L/month		0.432 L/month
EGC Lifetime (@75 mM)	28 Days		18 months
Mass Detection Limits	7000 fg		70 fg

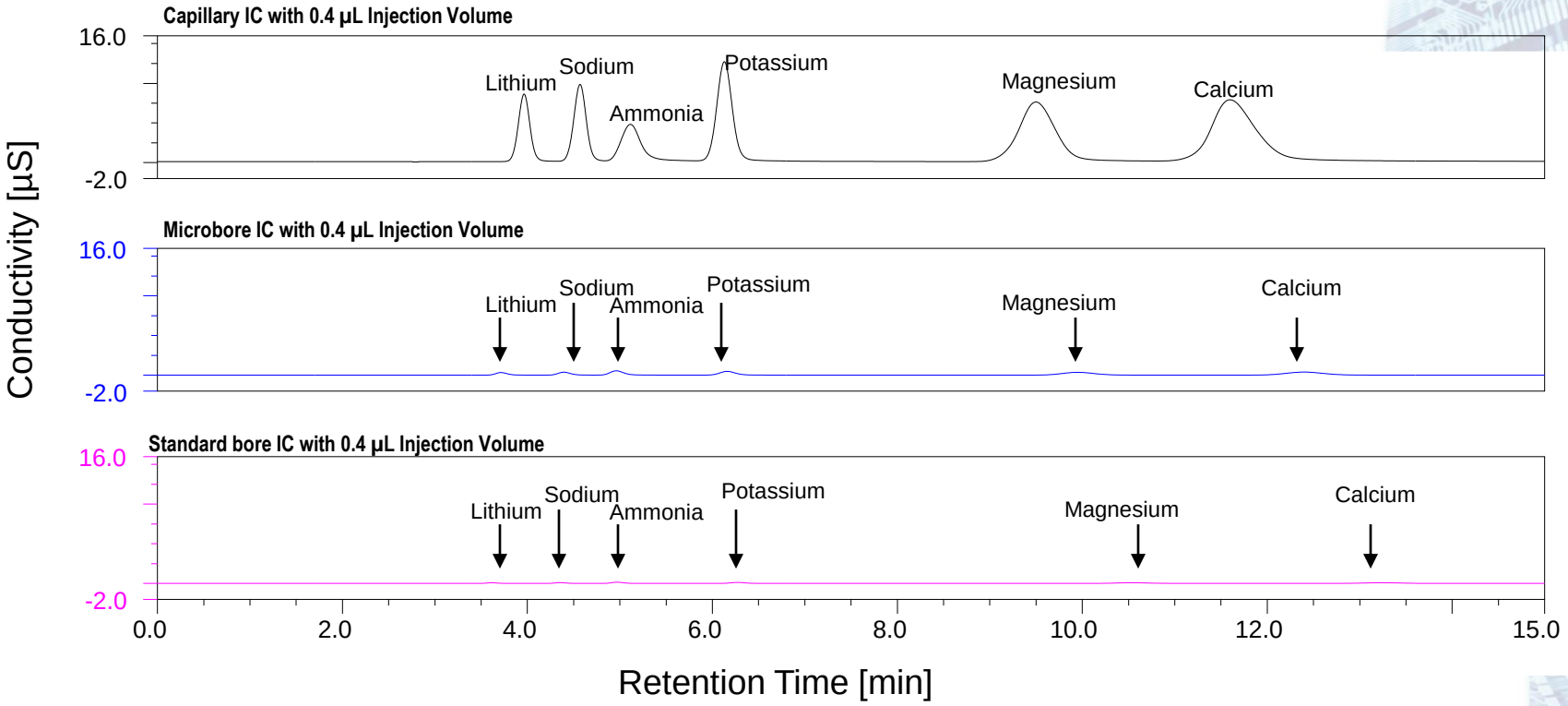
Capillary IC - Dimension of Scale

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



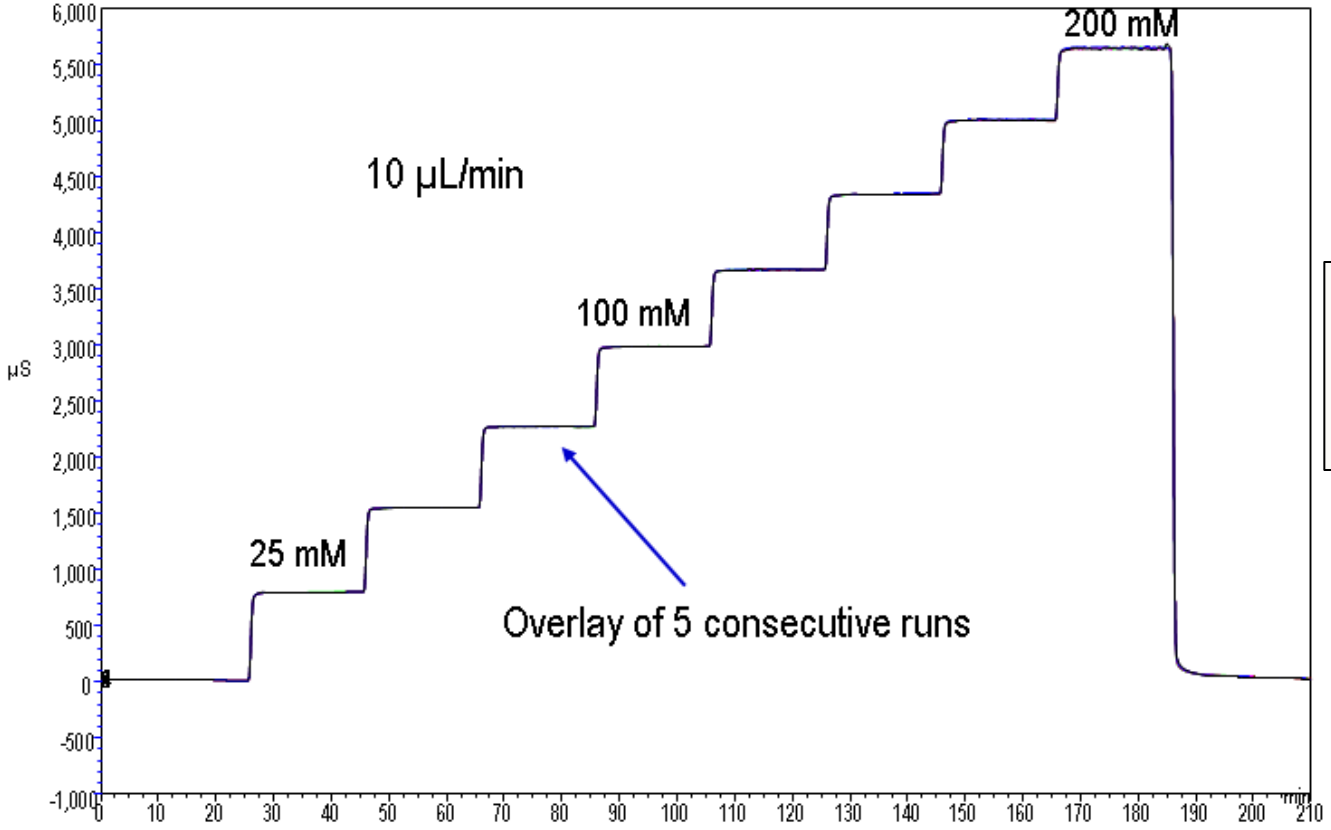
Capillary IC - Dimension of Scale

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



Electrolytically Formed Gradients

- EG step gradient – accuracy up to 200 mmol/L in capillary mode



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

Capillary Technology – The IC Cube™

CRD 200

Guard and Separation Column

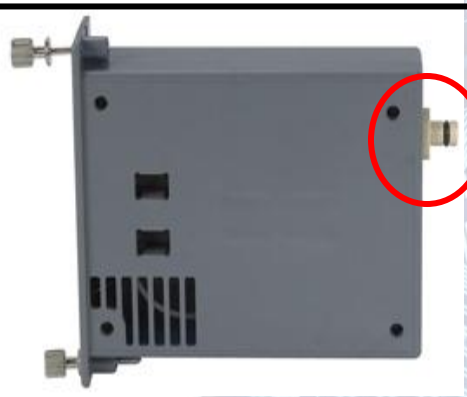
3.5”

Suppressor

Injection Valve

EG Degas

6.5”



Side view of Cap Suppressor

Fewer Connections

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

Change an IC Cube with all consumables - 6 connections!

IC Cube – Columns and Column Compartment

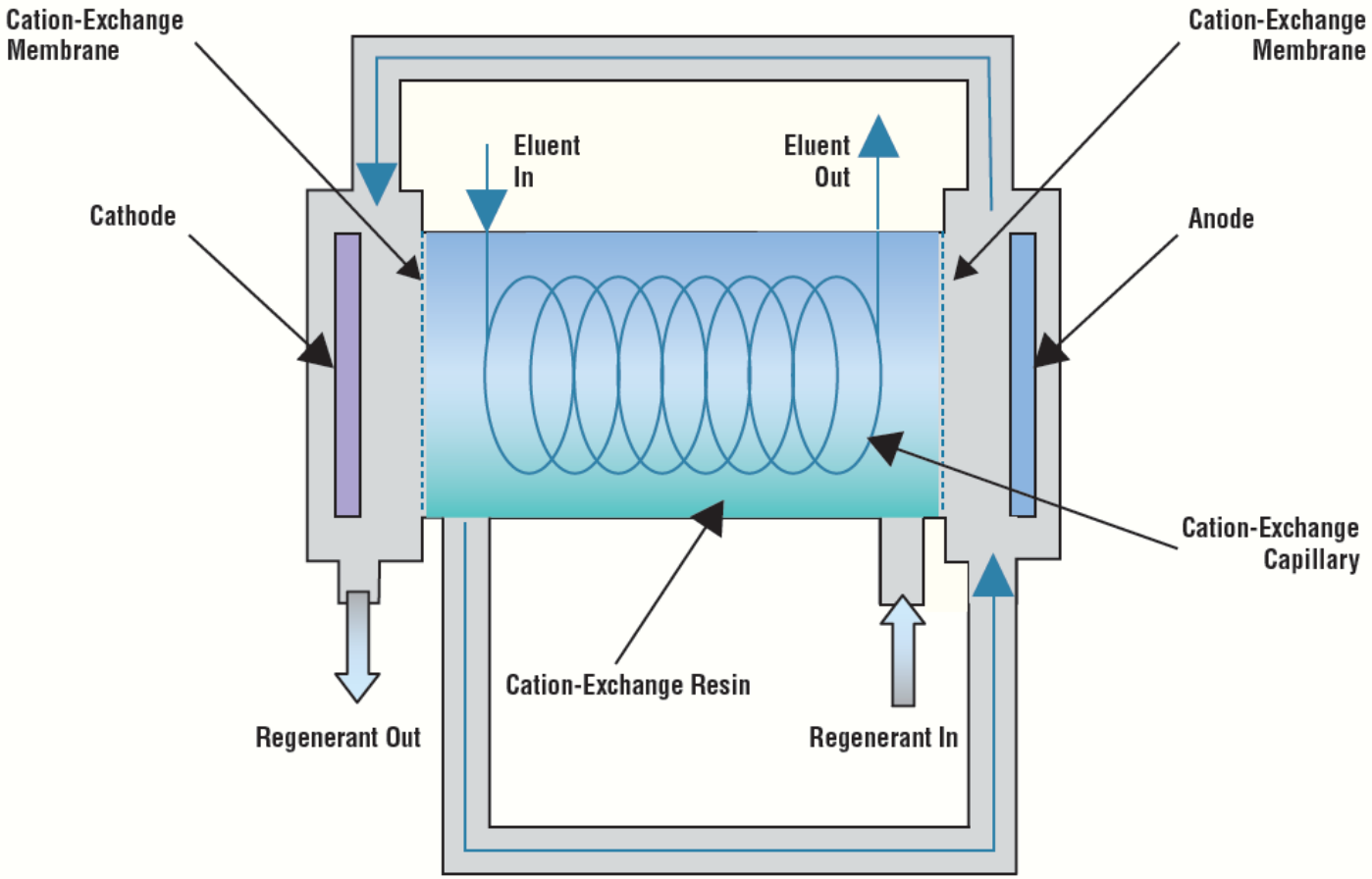
Separator

Guard

Capillary Electrolytic Suppressor

- Uses an ion exchange membrane capillary
- Regenerant channel is filled with cation exchange resin to enhance capacity
- Suppressor is cooled to enhance performance

Anion Capillary Electrolytic Suppressor



Capillary Conductivity Detector

- Volume Minimized (20 nL) Flow Cell
- Chromeleon auto-detects Capillary vs. Standard CD Cell
- Specifications for temperature stability, linearity, maximum pressure and sampling rate are unchanged from a Standard CD Cell.

Typical Workflow: Traditional IC

(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

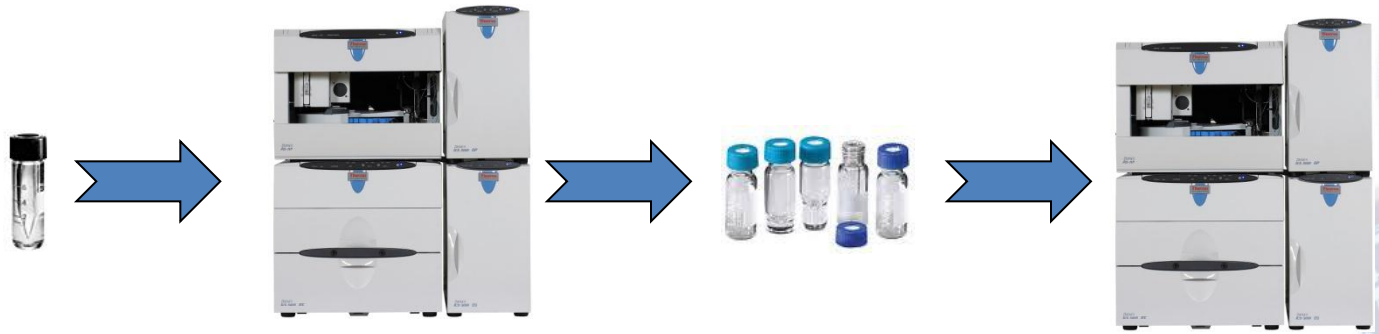
Results

Total Analysis Time:

Total Time = 6 hours

Typical Workflow: Capillary IC

Always On – Always Ready (IonPac AS19 Gradient Analysis)



Run Check Standard:

Total Time = 30 min

Run 5 Samples:

Total Time = 2 h 30 min

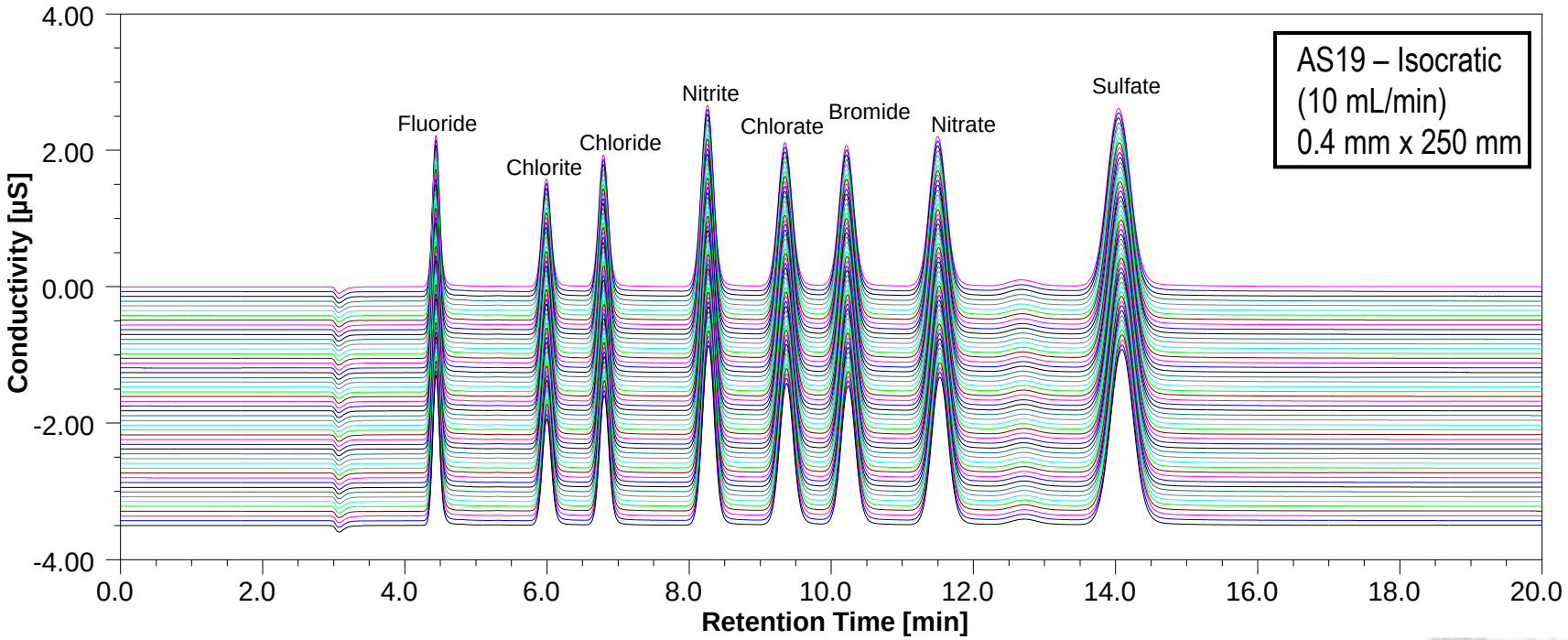
Results

Total Analysis Time:

Total Time = 3 hours

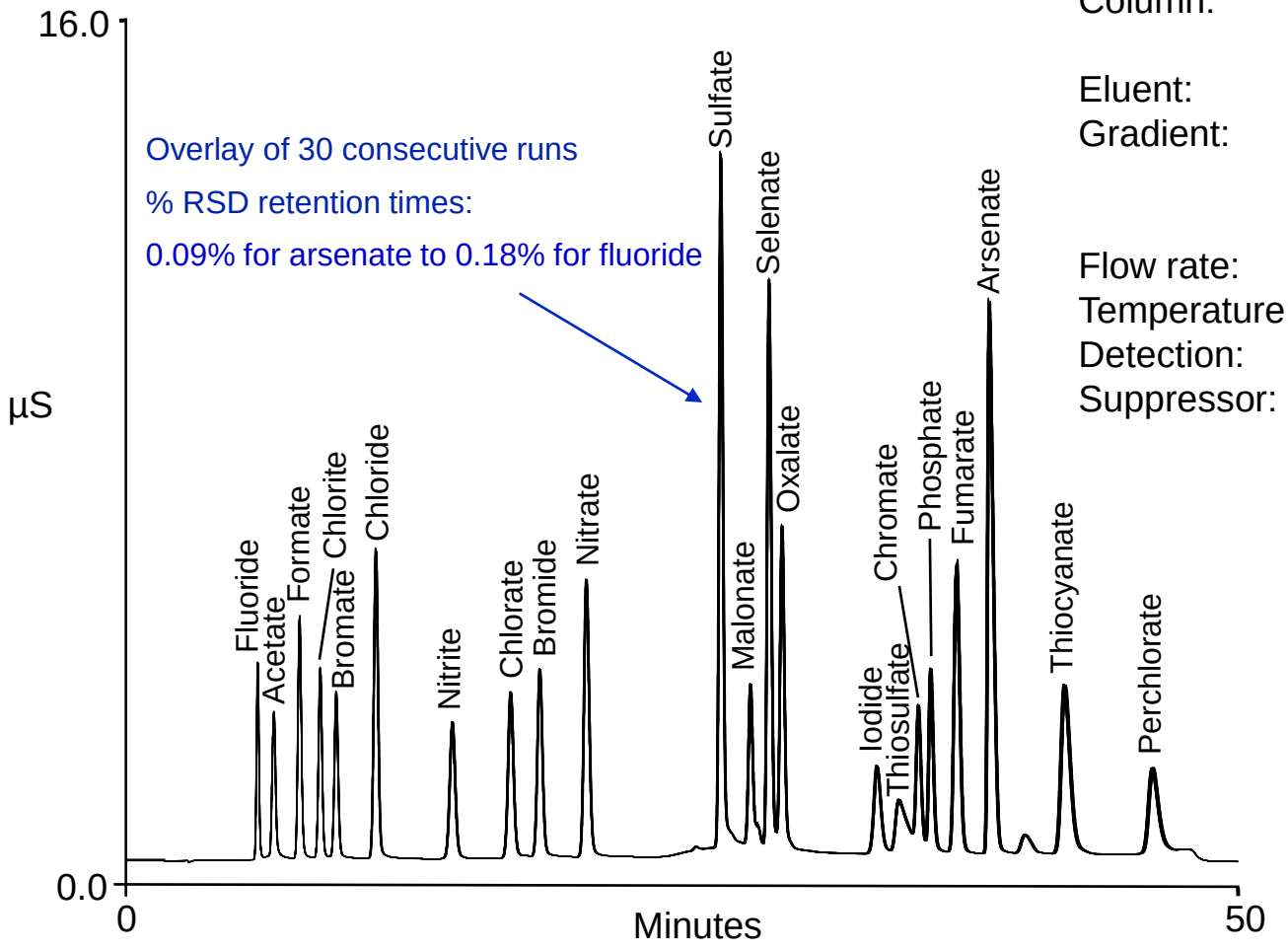
"IC on Demand"

Capillary IC Value: Best Results



	Fluoride	Chlorite	Chloride	Nitrite	Chlorate	Bromide	Nitrate	Sulfate
Retention Time (% 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

Separation of 22 Anions on a Capillary IonPac AS19 Column



Column:	IonPac AS19 (250 mm x 0.4 mm ID)
Eluent:	KOH (EG)
Gradient:	10 mmol/L (0 to 10 min), 10-52 mmol/L (10 to 42 min), 52-70 mmol/L (42 to 45 min)
Flow rate:	10 µL/min
Temperature:	30 °C
Detection:	Suppressed conductivity
Suppressor:	ACES 300

IMPACT ON TRACE ANALYSIS

Trace Analysis with Capillary IC

- **Large volume direct injection or pre-concentration**
  - A 10 µL injection onto a 0.4 mm ID column is equivalent to a
    1000 µL injection onto a 4 mm ID column
  - Loading a 250 µL sample onto a capillary can be accomplished with an AS-AP autosampler in a shorter time than loading a 25 mL sample onto a conventional concentrator with an AS-HV
  - Suitable for weakly contaminated samples

Trace Analysis with Capillary IC

- **Large volume direct injection or pre-concentration**

Column Dimension	volume (mL)	Factor	Injection Volume
4 x 250 mm	3.14	1	1000 uL
3 x 250 mm	**1.76625**	**1.777778**	**560 uL**
2 x 250 mm	0.785	4	250 uL
0.4 x 250 mm	0.0314	100	10 uL

Column Dimension	Volume (mL)	Factor	Injection Volume
4 x 250 mm	3.14	1	1000 uL
3 x 150 mm	**1.05975**	**2.962963**	340 uL
2 x 250 mm	0.785	4	250 uL
0.4 x 250 mm	0.0314	100	10 uL

*All Injection Volumes are relative to a 4-mm column (i.e. 1000 uL injection on 4 mm = 10 uL on a 0.4 mm or a 340 uL on a 3x150 mm)

**This can also be applied relative to a 3 or 2 mm to determine the volume for a cap system

***For example, the factor between a 3 x 150 mm and a 0.4 mm is 34, that is a 1000 uL on a 3x150 mm is approx 30 uL on a cap system

Capillary IC – Values: Small Sample Sizes

Limited Volumes:

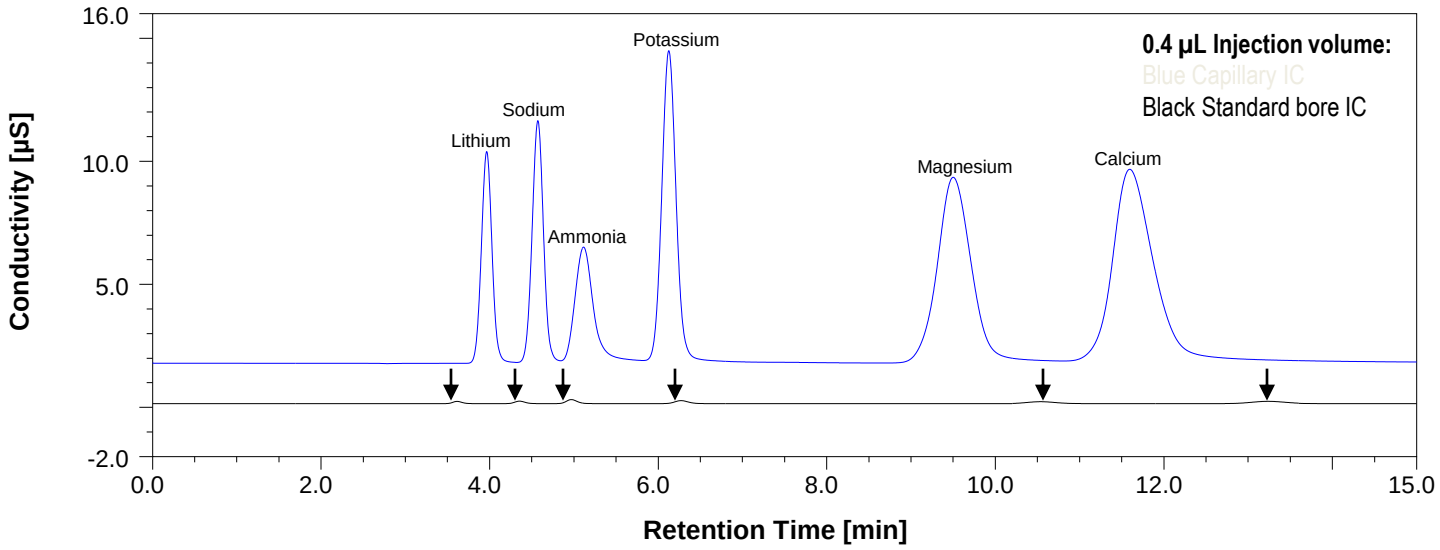
- Precious/Valuable samples (Metabolomics)
- Corrosion detection

Safety:

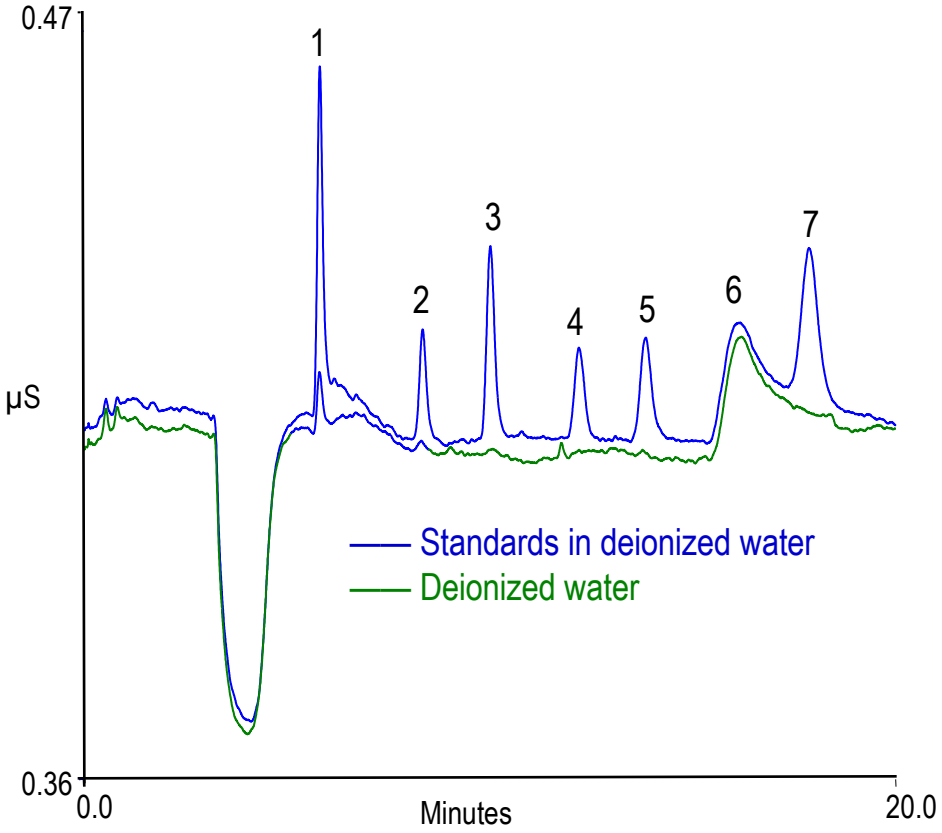
- Toxic/Dangerous samples

Reduced Waste Cost:

- Less overall waste to dispose of…. i.e. Radioactive waste.



Separation of Inorganic Anions at Trace Concentrations On IonPac® AS19 Capillary Column with 10-µL Injection



Column: IonPac® AS19 column (0.4 x 250 mm)

Eluent Source: Capillary EGC-KOH cartridge

Eluent: 20 mM KOH

Flow Rate: 10 µL/min

Temperature: 30 °C

Suppressor: Electrolytic capillary anion suppressor used in conjunction with a capillary CRD

Detection: Suppressed conductivity

Injection Volume: 10 µL

Peak	Concentration (ppb)
1. Fluoride	0.2
2. Chloride	0.3
3. Nitrite	1.0
4. Bromide	1.0
5. Nitrate	1.0
6. Carbonate	–
7. Sulfate	1.0

HPIC

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

Faster separations are as accurate and precise as conventional methods!

What is High Pressure IC (HPIC), and What Can It Do?

- 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

HPIC

High Pressure Ion Chromatography:

Uses small particle size columns (4 µm):

- High efficiency separations to resolve more peaks in one run with the same run time

Or

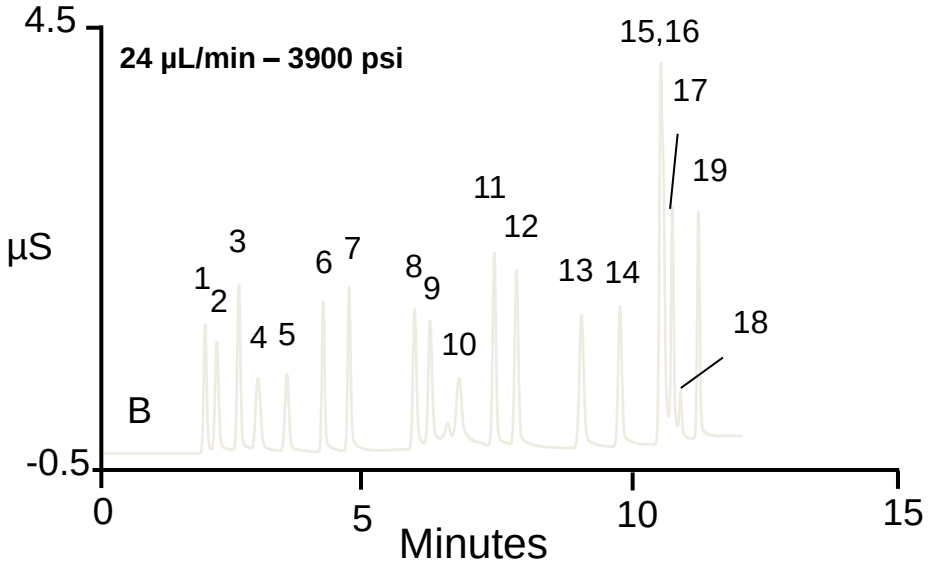
- Maintain chromatographic efficiency with same peak resolution but shorter analysis time

High Pressure Capabilities of Capillary IC

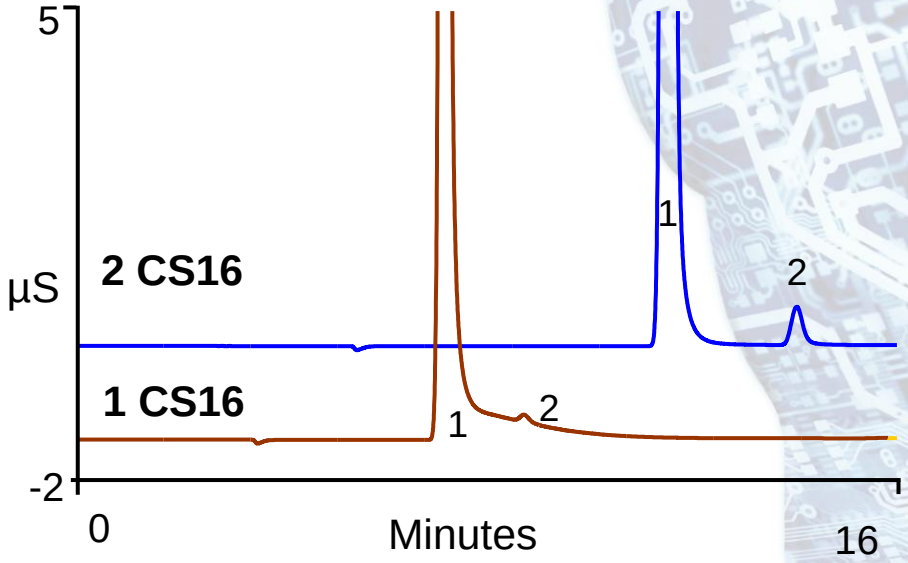
Expanded Capability:

- 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)

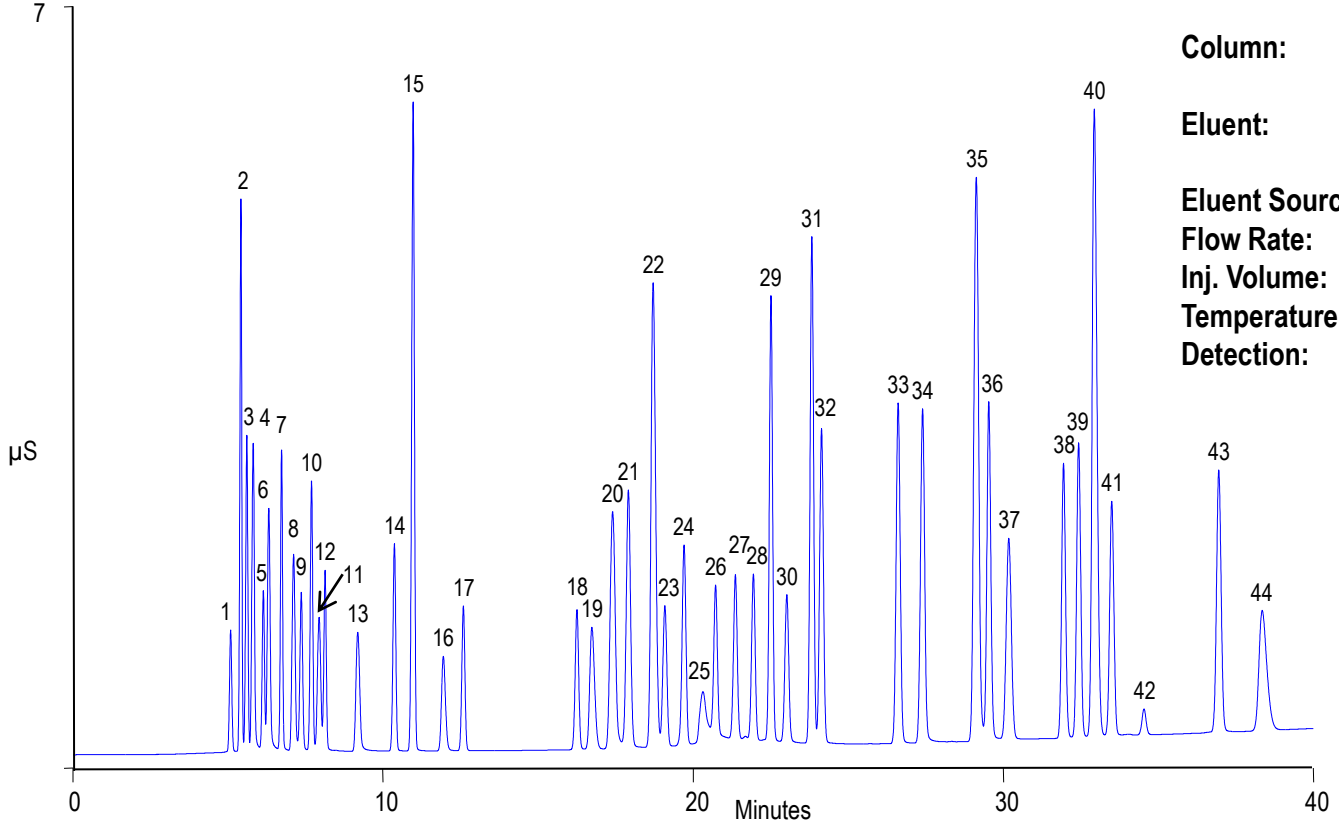
IonSwift MAX-100: 11 minutes



10000:1 Na : Ammonia



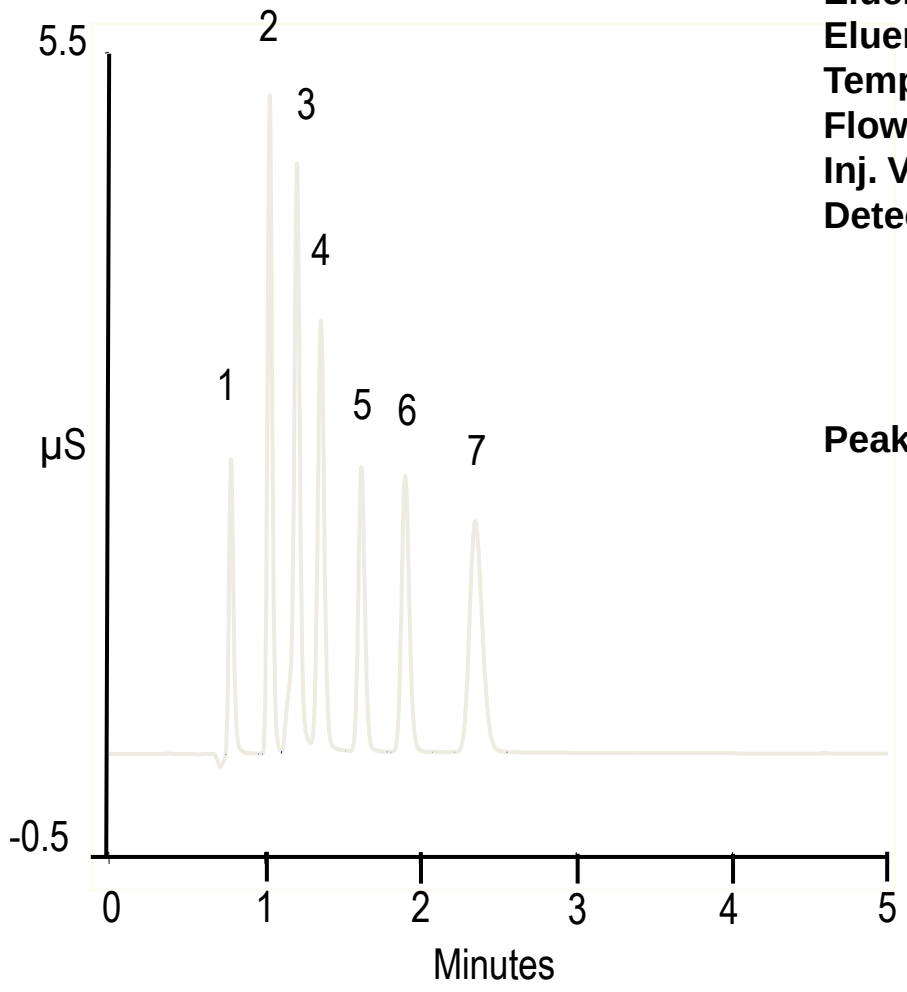
IonPac AS11-HC-4µm (0.4 × 250 mm Column)



Column: IonPac AS11-HC-4µm , capillary column
Eluent: 1-14 mM KOH in 16 min, 14-55 mM KOH in 24 min,
Eluent Source: EGC KOH capillary cartridge
Flow Rate: 15 µL/min
Inj. Volume: 0.4 µL
Temperature: 30 ˚C
Detection: Suppressed conductivity, Anion Capillary Electrolytic Suppressor ACES™ 300, 24 mA AutoSuppression recycle mode

1. Quinate
2. Fluoride
3. Lactate
4. Acetate
5. 2-hydroxybutryrate
6. Propionate
7. Formate
8. Butyrate
9. 2-hydroxyvalerate
10. Pyruvate
11. so-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 Run on the IonPac AS18-4µm Column

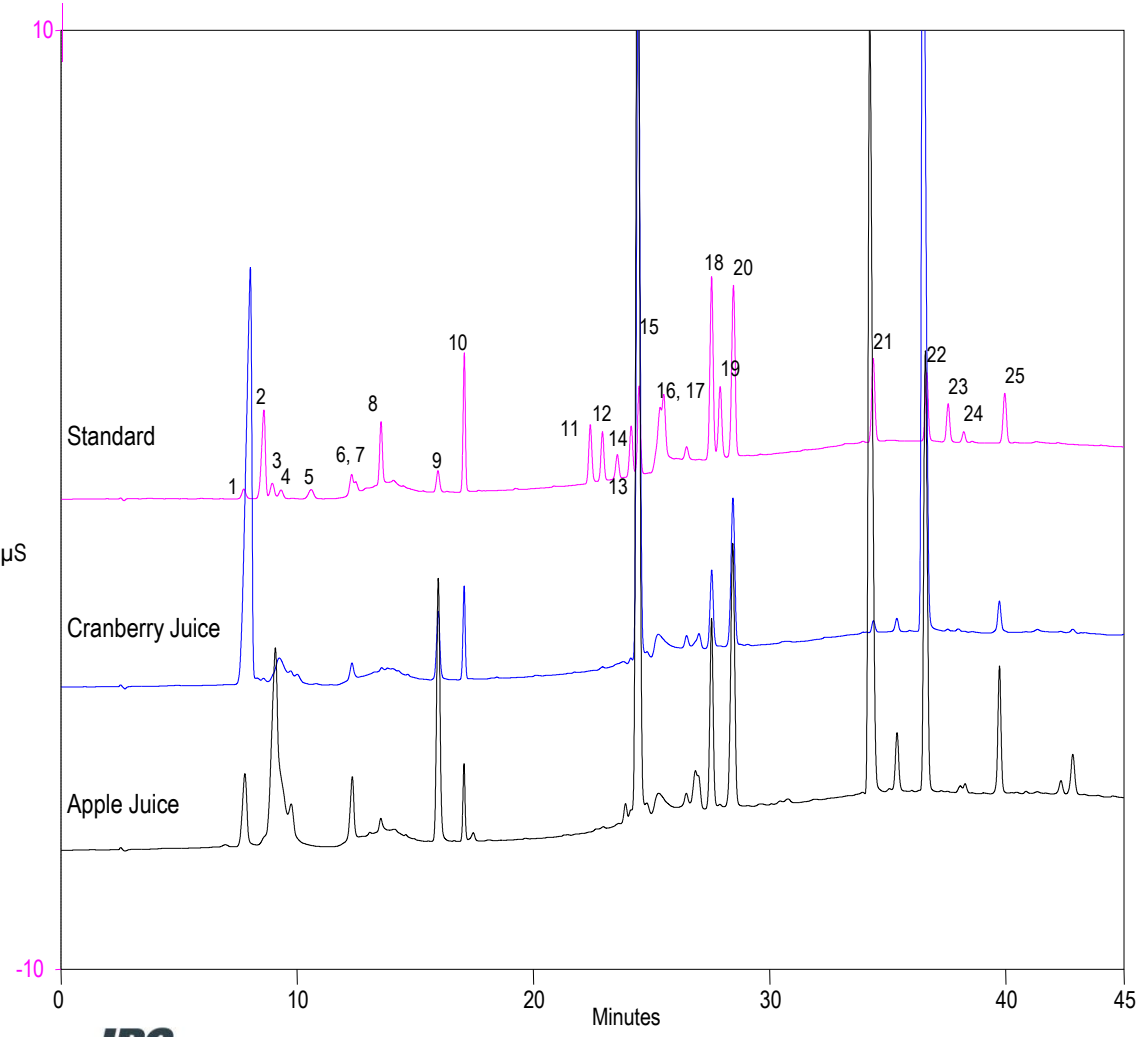


Column:	**AS18-4um Capillary Column**
Eluent:	**35 mM KOH**
Eluent Source:	**EGC-KOH capillary cartridge**
Temperature:	**30 °C**
Flow Rate:	**30 µL/min**
Inj. Volume:	**0.4 µL**
Detection:	**Suppressed conductivity, Anion Capillary Electrolytic Suppressor (ACES™ 300) AutoSuppression® recycle mode**

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**	

Analysis of Juice Samples Using Dionex IonPac AS11-HC-4µm Capillary Column and Gradient Chromatography



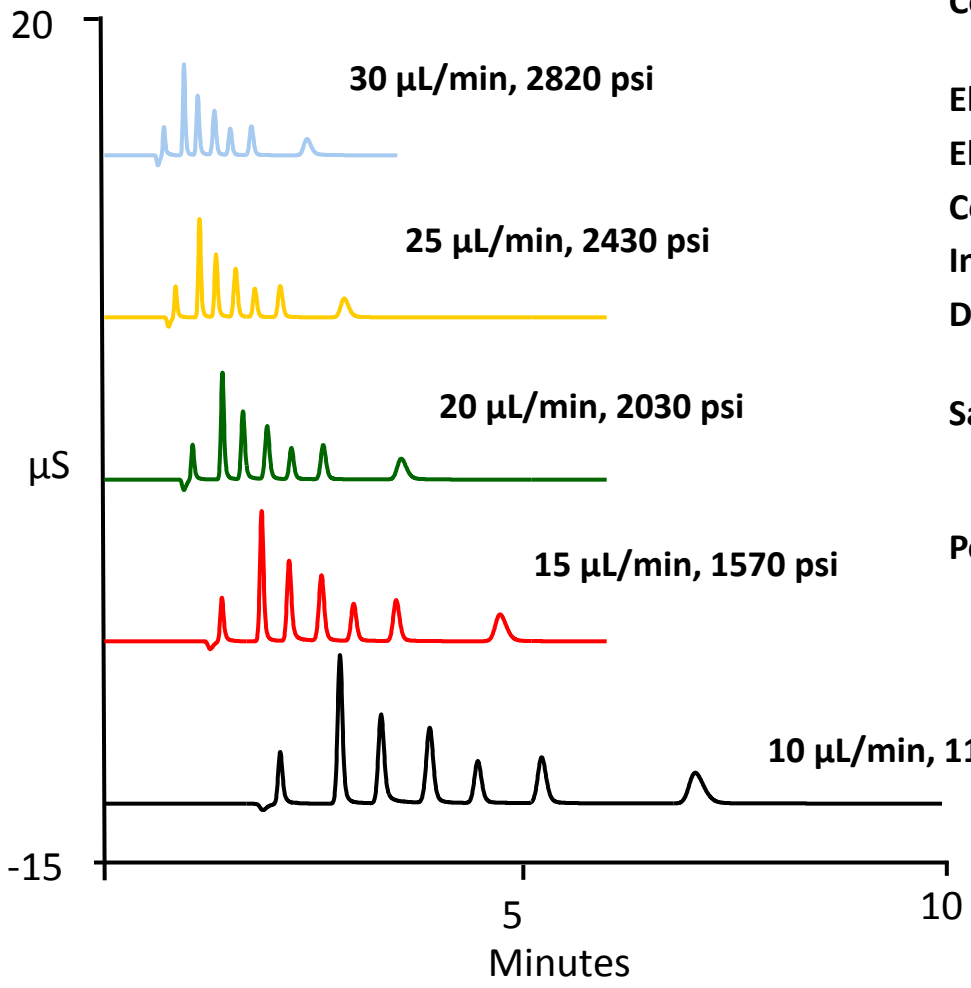
Column:	Dionex IonPac AG11-HC-4µm/ AS11-HC-4µm (0.4 × 250 mm))
Eluent +*10% Methanol :	1mM KOH for 8min, 1-15mM KOH in 10 min; 15 to 30mM KOH in 10 min 30 to 60mM KOH in 10 min
Eluent Source:	Dionex EGC III KOH capillary cartridge
Flow Rate:	0.015mL/min
Inj. Volume:	0.4µL
Temperature:	30˚C
Detection:	Suppressed Conductivity, Dionex Anion Capillary Electrolytic Suppressor (Dionex ACES 300) AutoSuppression, external water mode
Sample:	Juice with 1:50 sample dilution

Peaks (Standard):		**mg/mL**
1.	Quinate	1
2.	Fluoride	0.6
3.	Lactate	1
4.	Acetate	1
5.	Propionate	1
6.	Formate	1
7.	Butyrate	1
8.	Pyruvate	2
9.	Galacturonate	2
10.	Chloride	1
11.	Bromide	1
12.	Nitrate	1
13.	Glutarate	2
14.	Succinate	2
15.	Malate	2
16.	Carbonate	--
17.	Tartrate	2
18.	Sulfate	2
19.	Fumarate	2
20.	Oxalate	2
21.	Phosphate	3
22.	Citrate	3
23.	Isocitrate	3
24.	cis-Aconitate	--
25.	trans-Aconitate	3

* 10% Methanol was added to Di water

Speed

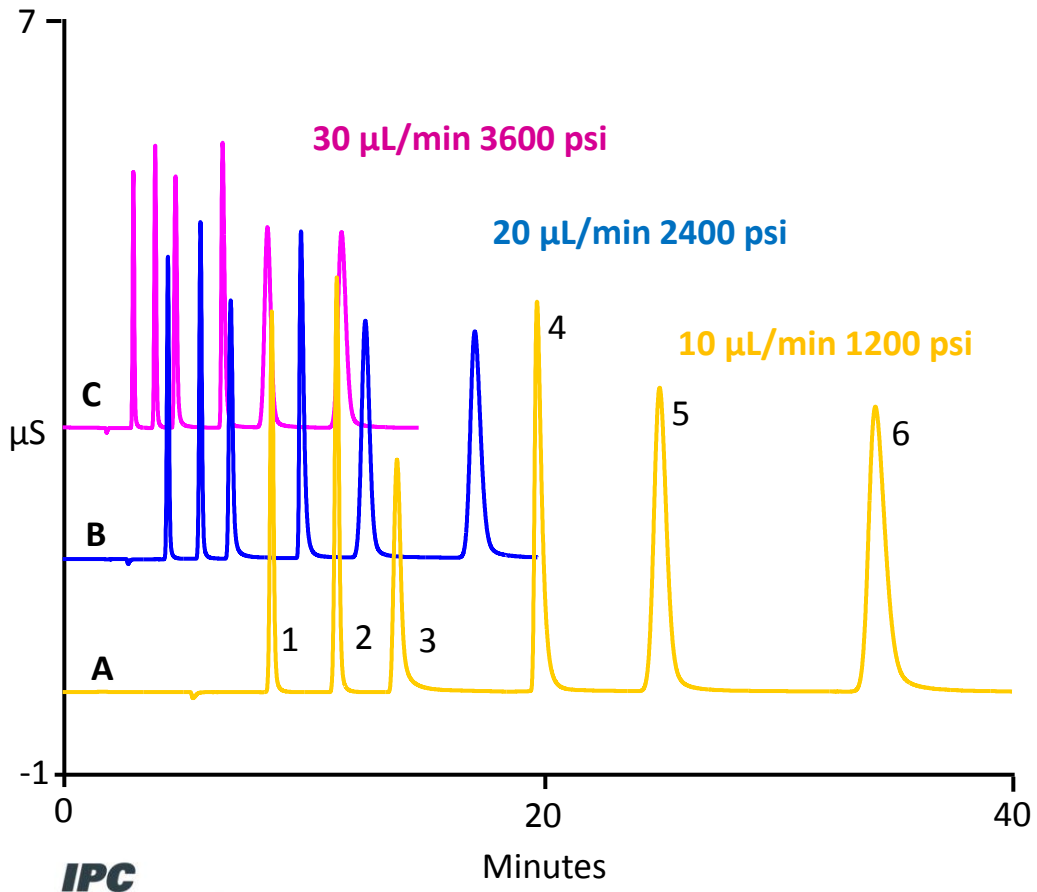
Separation of inorganic anions using a 4 µm column at different flow rates.



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, Thermo Scientific 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**	

High Resolution Cation Analysis on IonPac CS16 at Different Flow Rates



Column: IonPac CS16, **2 x 250 mm x 0.5 mm ID**
Eluant: 30 mmol/L MSA (EG)
Flow rate: A: 10 µL/min
B: 20 µL/min
C: 30 µL/min
Inj. volume: 0.4 µL
Temperature: 40 ° C
Detection: Suppressed conductivity CCES 300, AutoSuppression, Recycle mode

Peaks:		
1. Lithium	0.5	mg/L
2. Sodium	2.0	
3. Ammonium	2.5	
4. Potassium	5.0	
5. Magnesium	2.5	
6. Calcium	5.0	

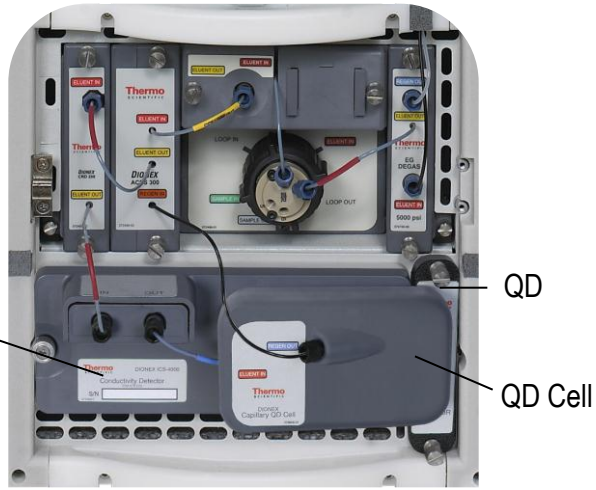
High Pressure Capillary IC - Summary

- Enables high-resolution separations and Fast IC separations using new 4 µm particle columns

- **Speed** 19 anions and organic acids in 12 minutes Common anions/cations in less than 5 minutes!
- **Simplicity** Easy resolution improvement using columns in series
  Half the connections of a traditional analytical system
- **Solutions** Convenient upgrade kits make any ICS-5000 capillary system ready for high pressure and Fast IC

What is Charge Detection?

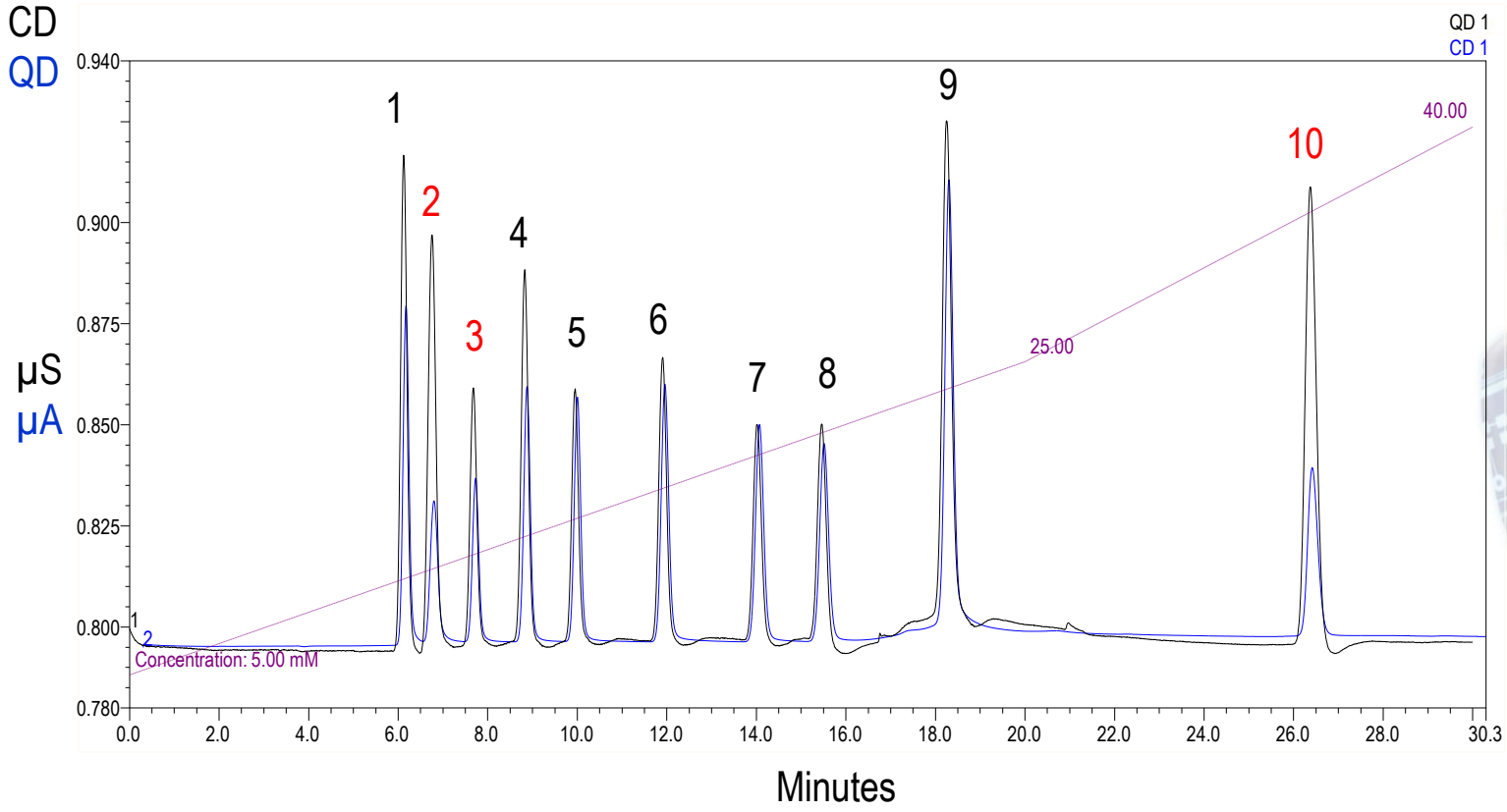
- Response is proportional to ion charge, providing universal calibration
- Cell is a membrane device similar to a CR-TC (est. 24-month lifetime)
- Weakly dissociated analytes show higher response
- Improved linearity for weakly dissociated species:
  organic acids, amines, silicate, borate
- Complements suppressed conductivity detection



Capillary QD Cell

Quantification of unknowns at low cost with universal, linear calibration

Overlay of CD and QD (Normalized to Chloride)



1. Fluoride
2. Acetate
3. Formate
4. Bromate
5. Chloride
6. Nitrite
7. Bromide
8. Nitrate
9. Sulfate
10. Phosphate

Greater response for weakly dissociated and multiply charged ions

HPIC: Feature, Benefit, Value

Feature	Benefit	Value
HPIC: Continuous operation at 5000 psi	Support for high flow rates	Faster run times, high productivity
HPIC: Continuous operation at 5000 psi	Support for small particle columns	High resolution, better separations
High pressure RFIC	No need to make eluents	Ease of use, just add water
Capillary and analytical formats	Continuous operations for capillary systems	Results on demand/Results on request

HPIC: High Resolution, Fast Analysis

Summary

- Capillary IC allows you to reduce operational costs by running a full year on 5 L of water. It is “Always Ready” allowing you to start running samples whenever you want, and continue to run them without compromise. Operation with Capillary IC is truly plug & play, making it the easy to use. With configurations that allow both capillary and analytical formats, including isocratic and gradient capabilities, it is the most flexible IC system in the world.

Take-Home Message

- New Capillary HPIC products offer High Resolution and Fast Ion Chromatography

- New Charge Detector QD
  - Brings a new level of detection simplicity to ion chromatography

Thank you!

ThermoFisher
SCIENTIFIC

The world leader in serving science