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Ultra-high-pressure ion chromatography with suppressed conductivity detection at 70 MPa using columns packed with 2.5 µm particles

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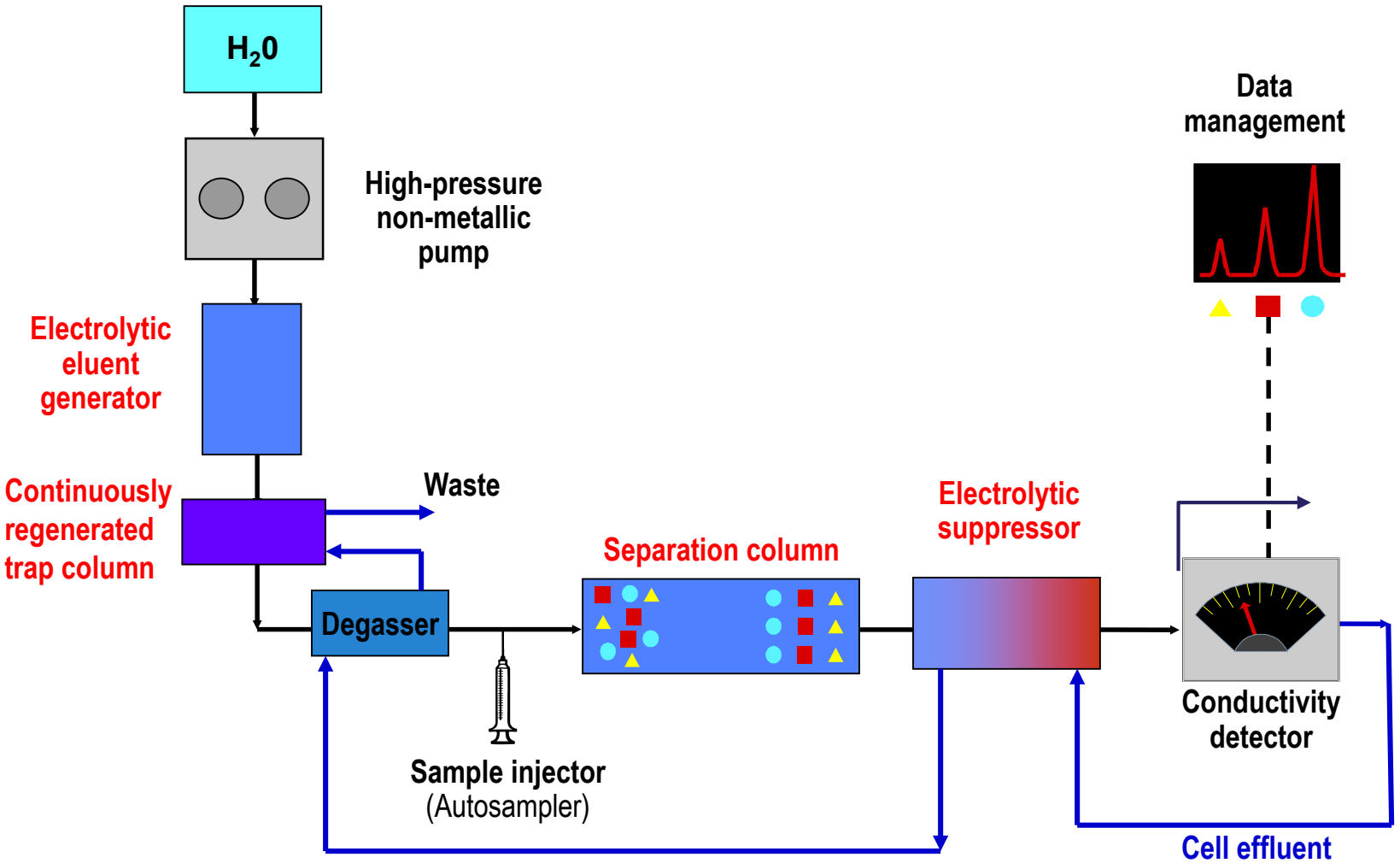
Reagent-Free™ Ion Chromatography System – IC without Manual Eluent Preparation



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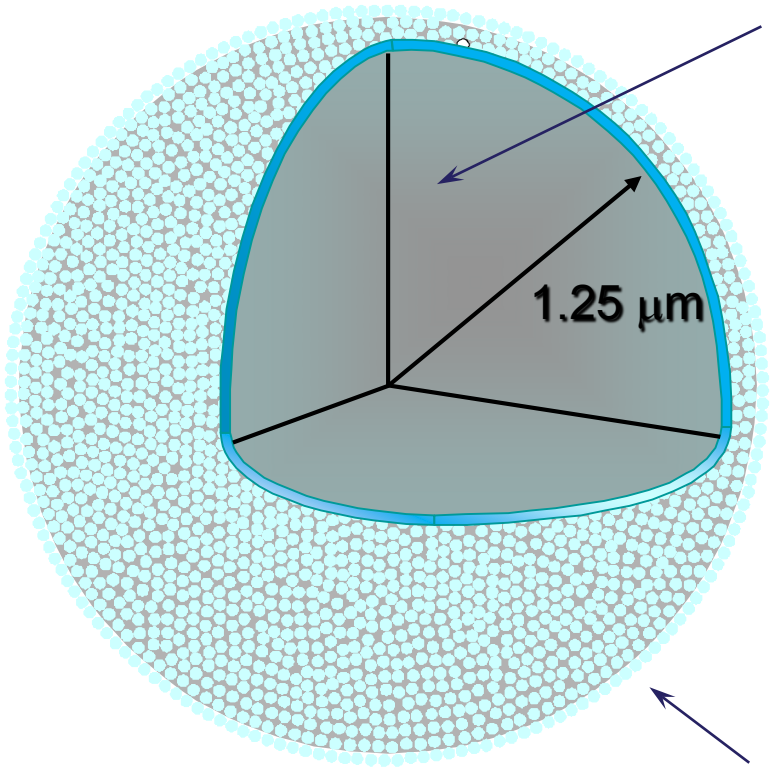
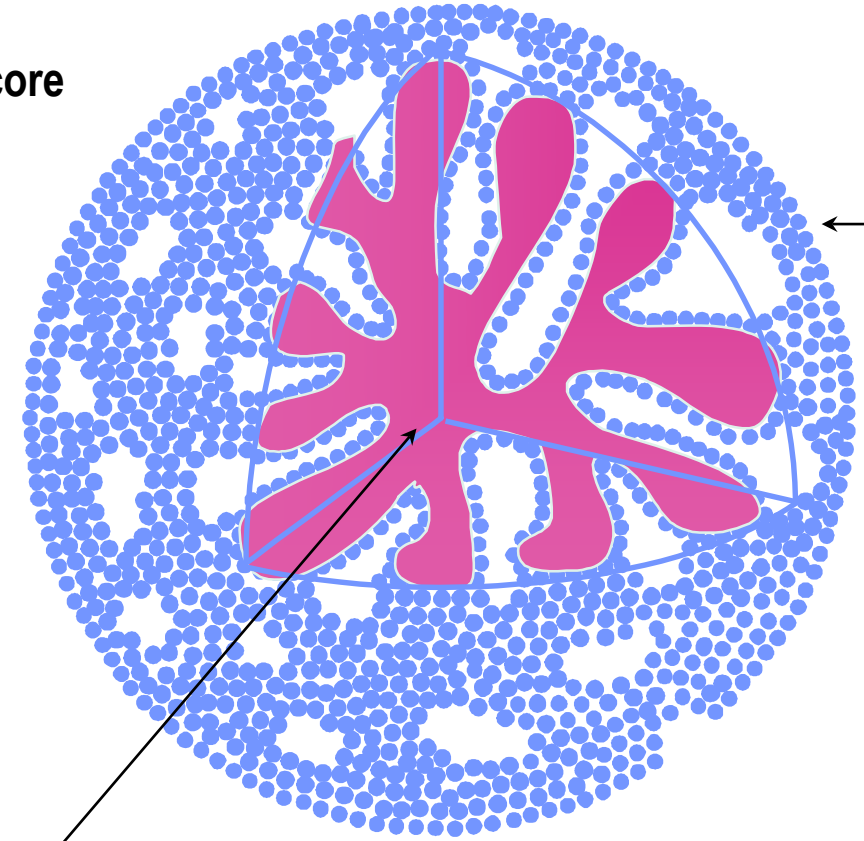
Resin Structure Schematics Thermo Scientific™ Dionex™ IonPac™ Columns

Dionex Ion Pac AS17



Dionex IonPac AS16



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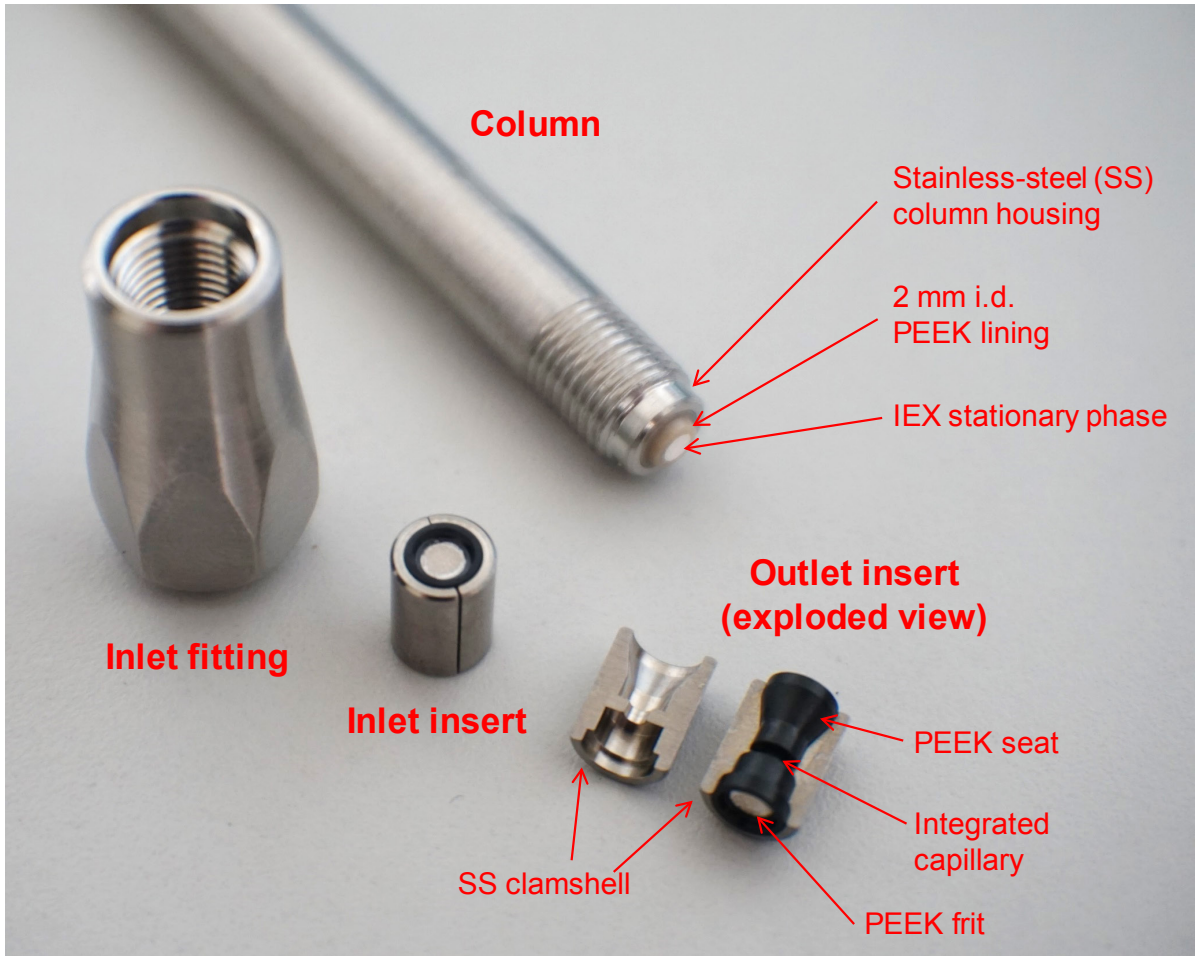
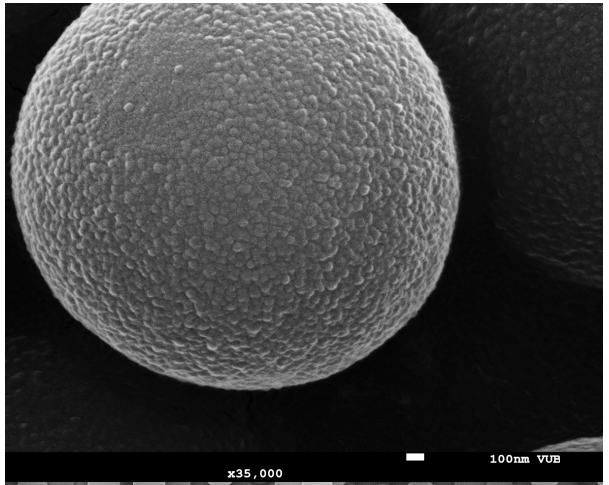
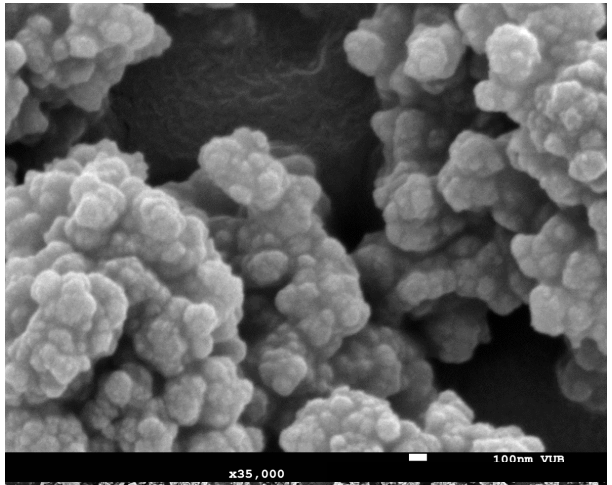
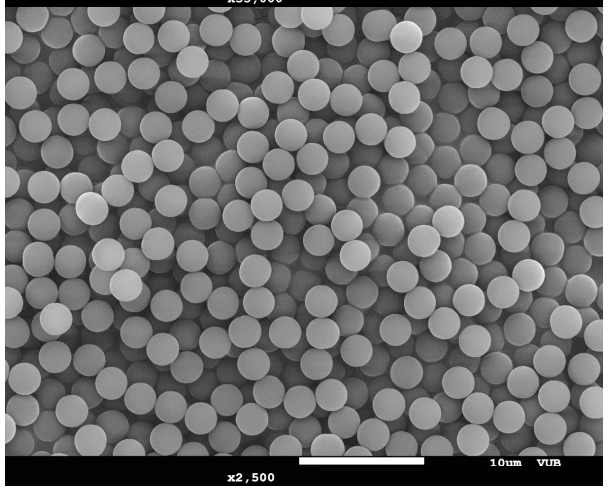
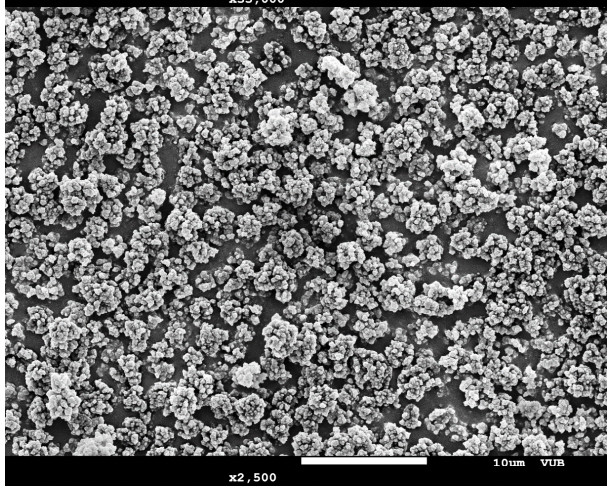
Column Hardware and Media











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System Configuration

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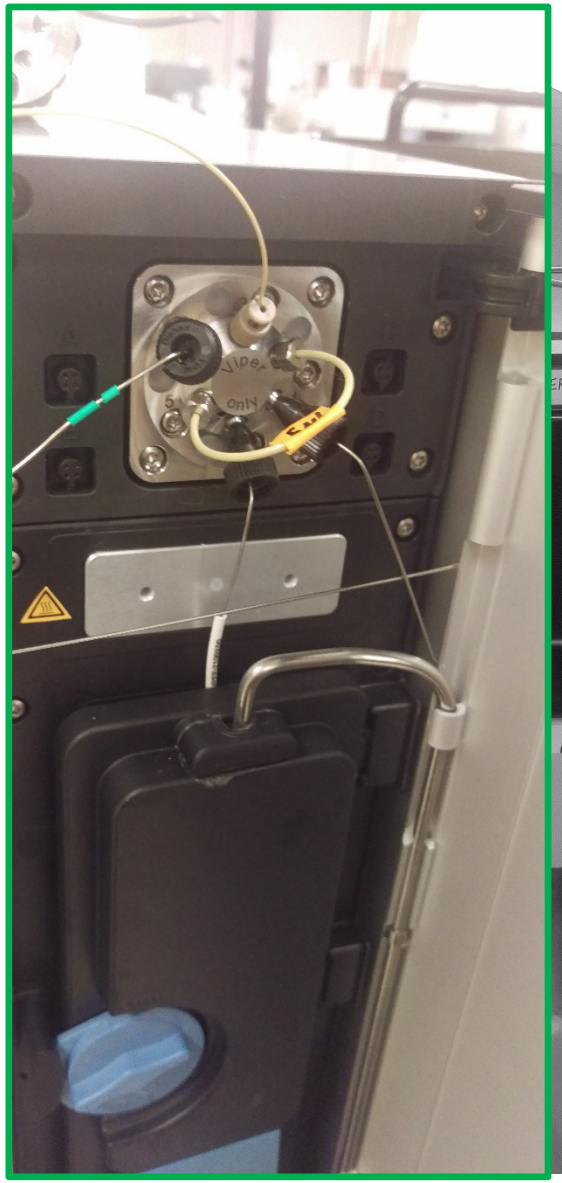
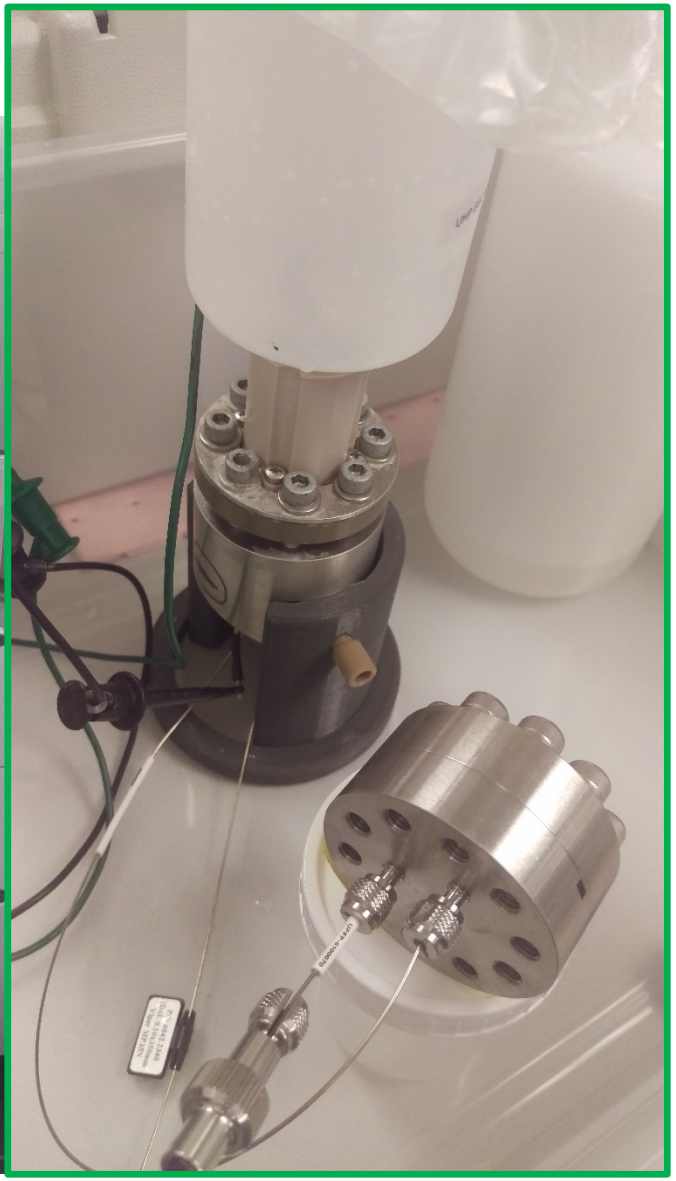
System Configuration





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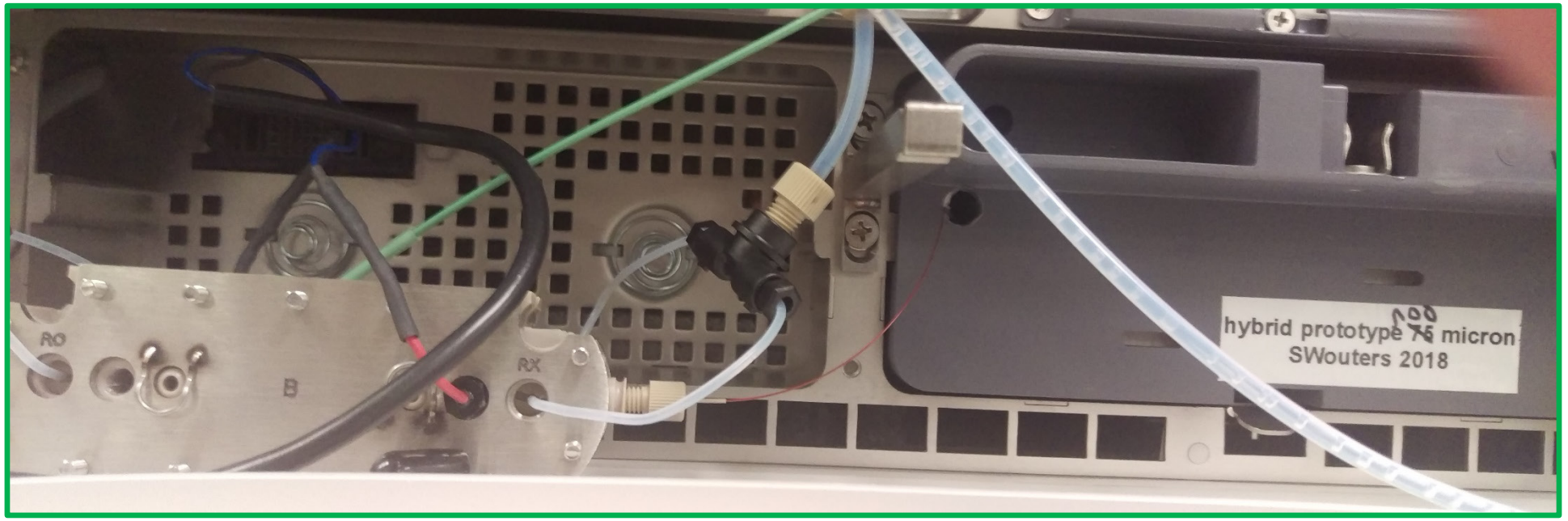
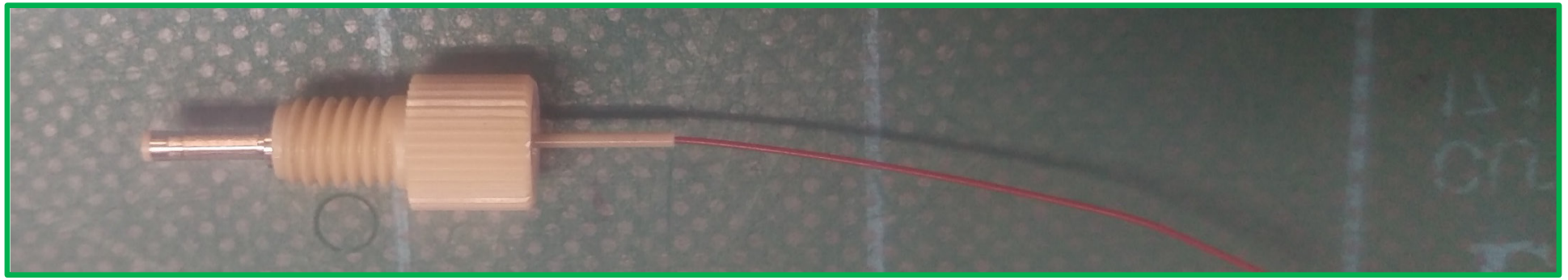
System Optimization





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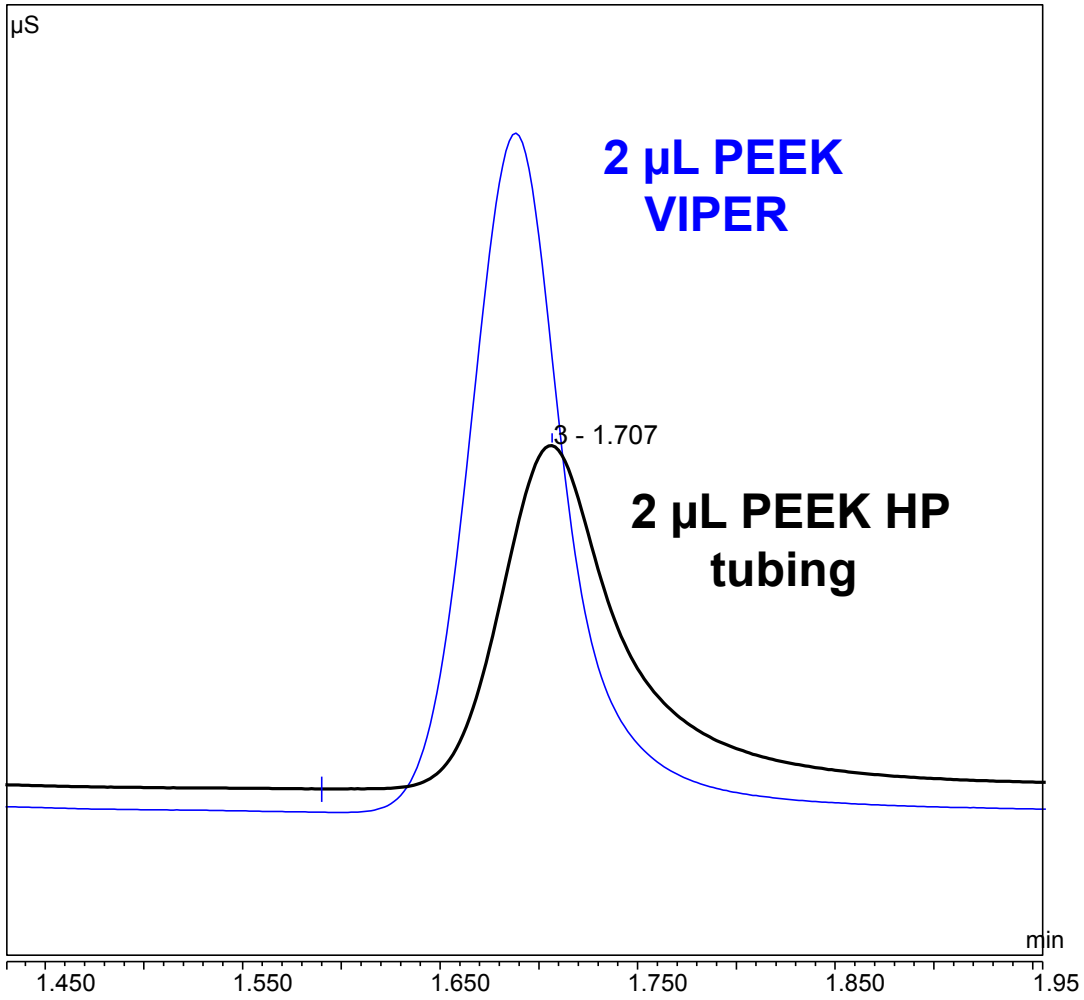
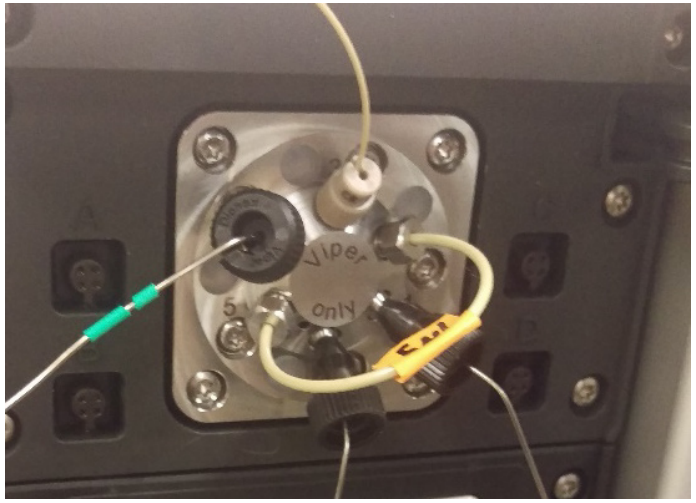
Injection Optimization





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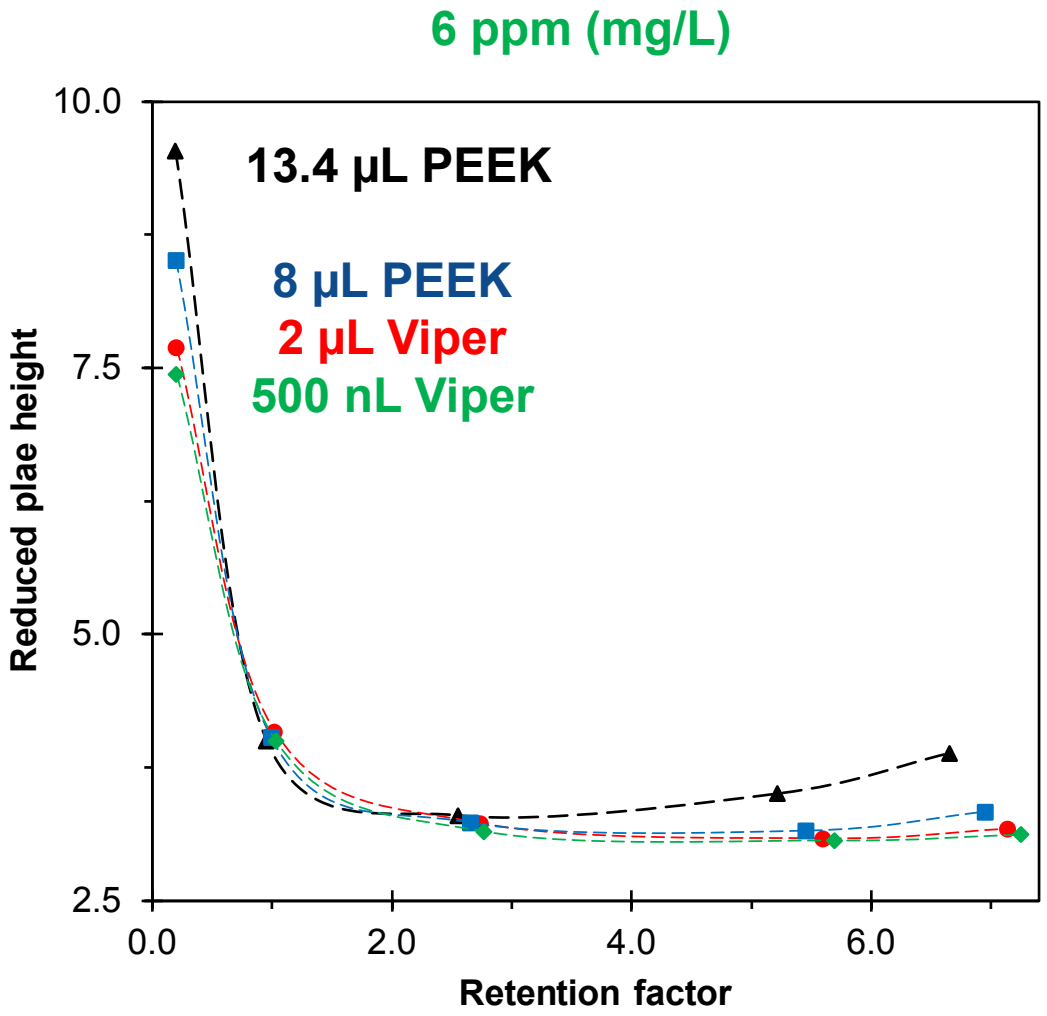
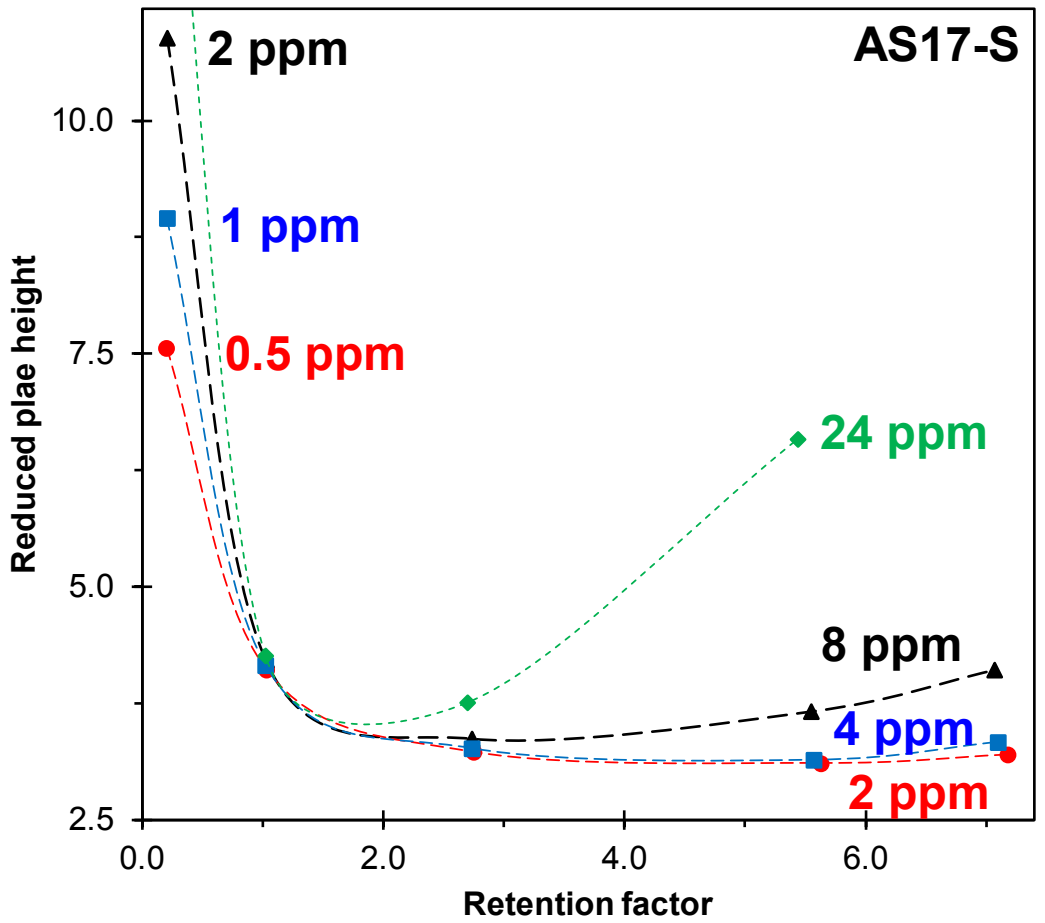
Injection Optimization





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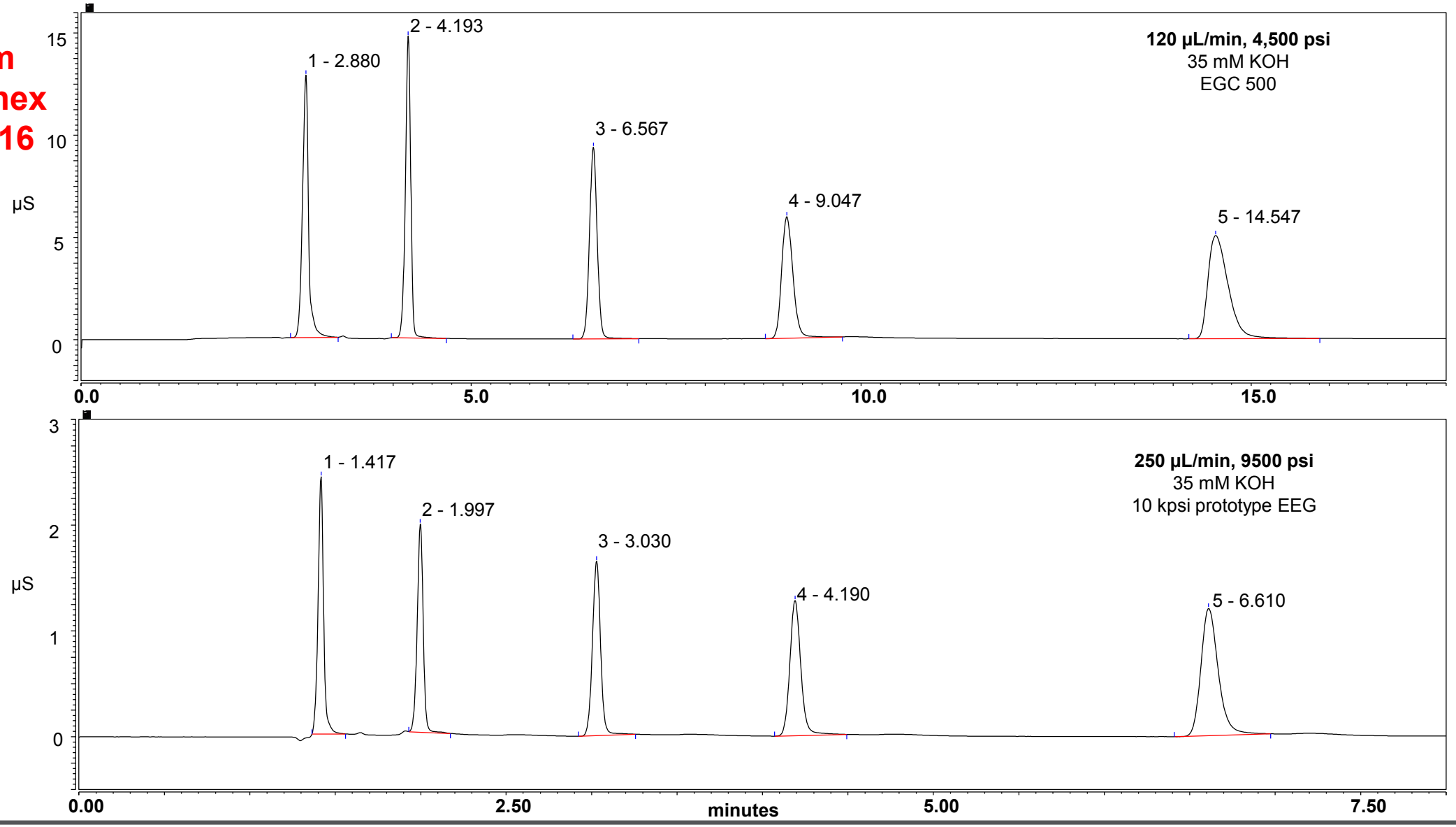
Performance of 2.5 µm Dionex IonPac AS16 Prototype

2x150 mm
2.5 µm Dionex
IonPac AS16



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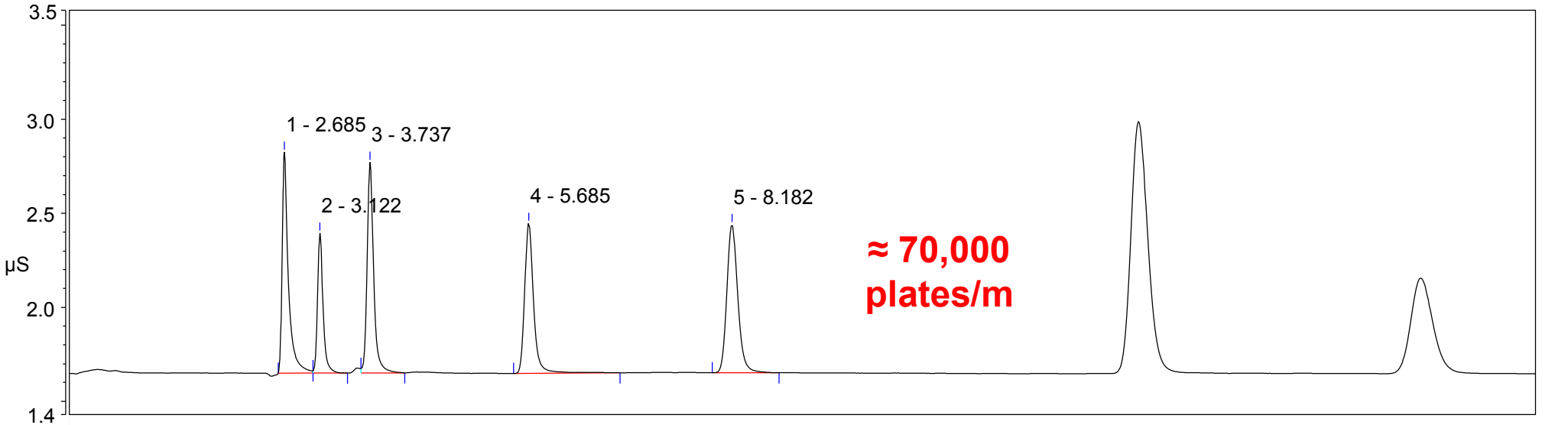
Performance Gains with Particle Size Reduction

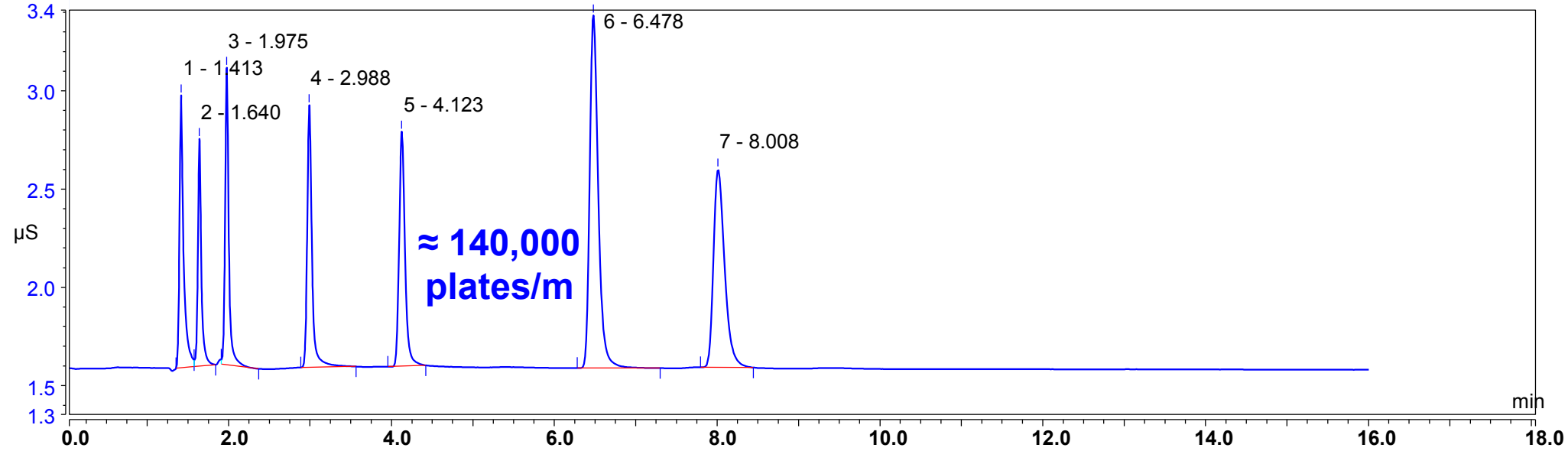
Commercial 2x250 mm 4 µm Dionex IonPac AS16

35 mM KOH



2x150 mm 2.5 µm Dionex IonPac AS16

35 mM KOH



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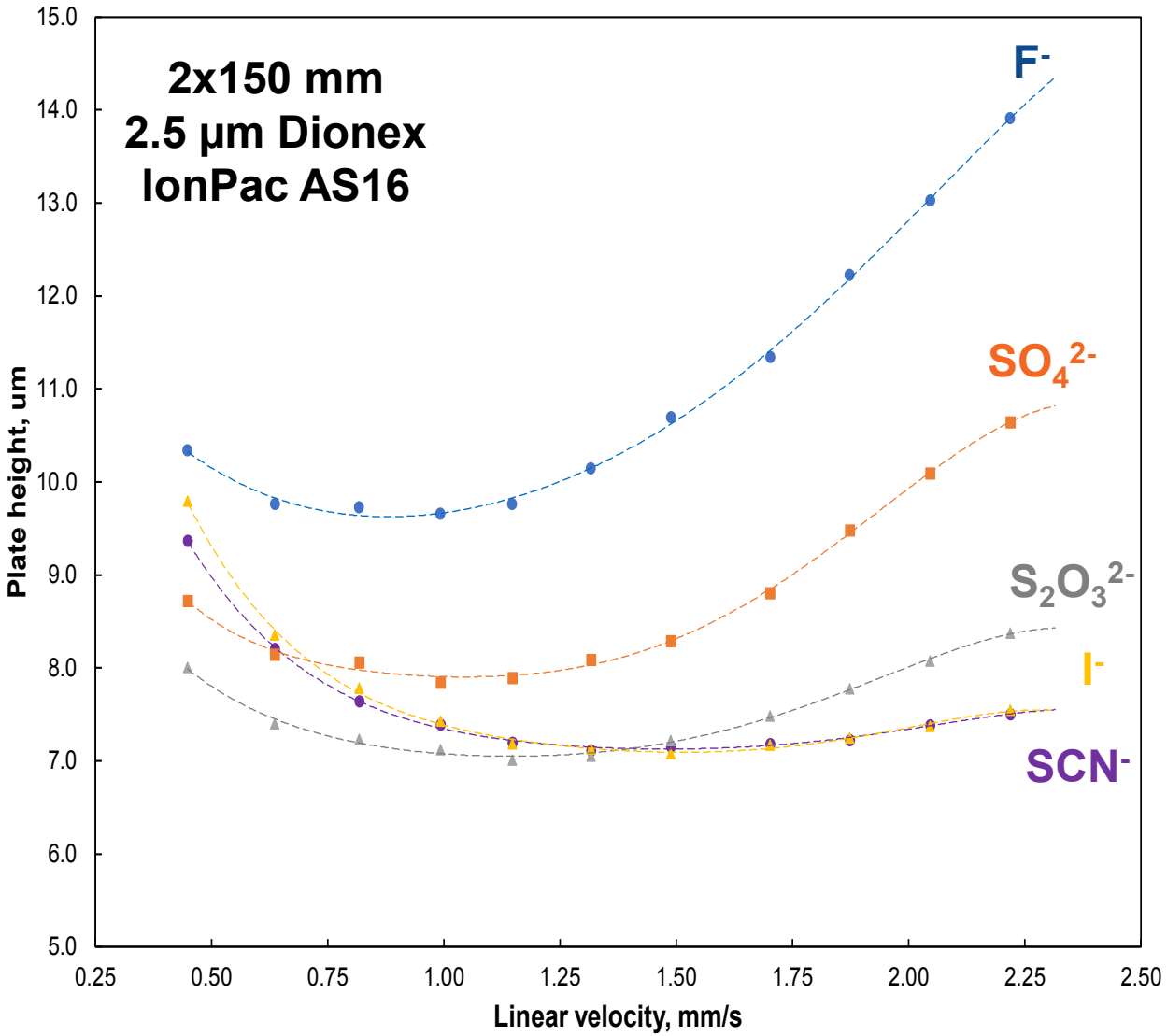
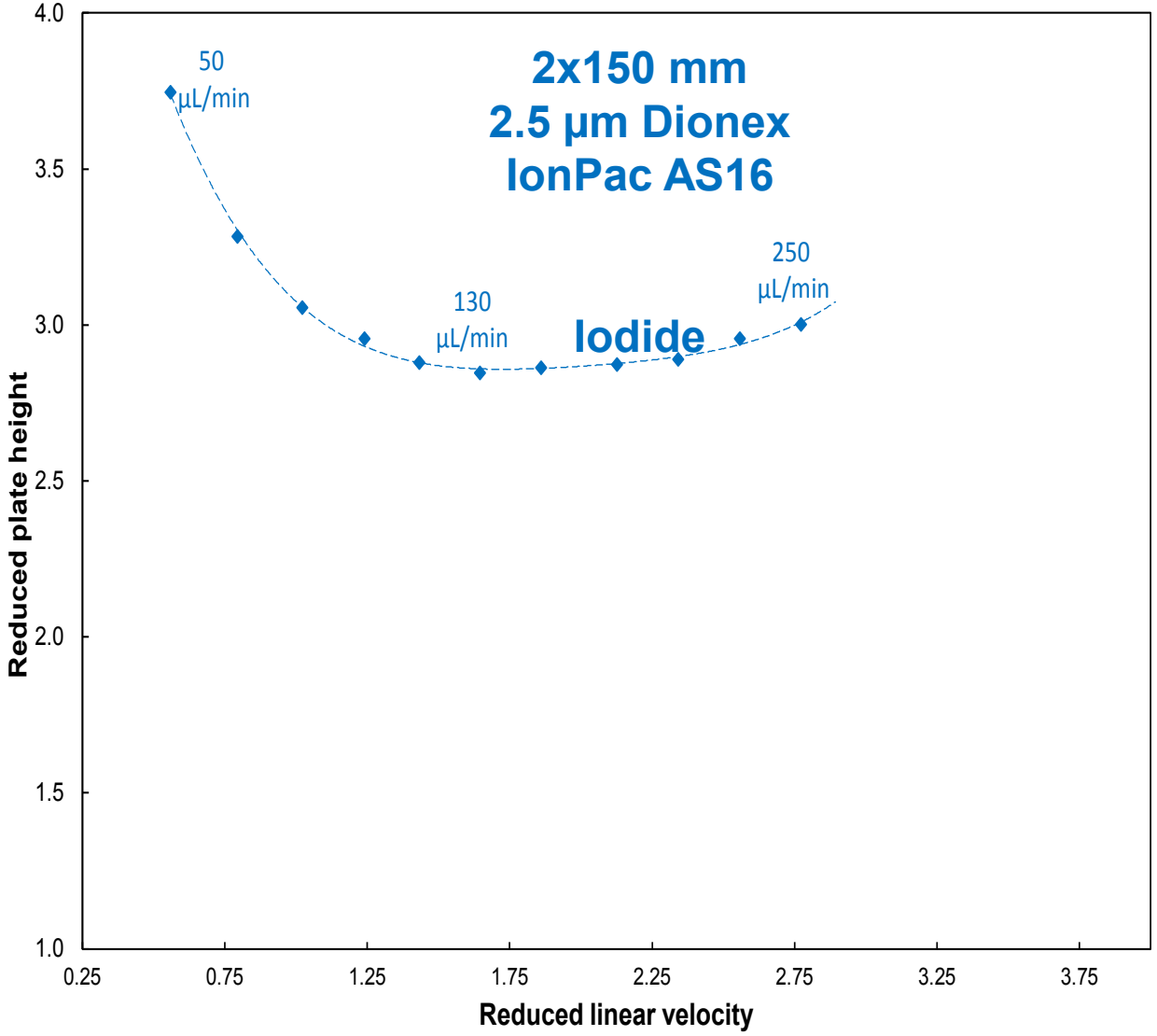
Van Deemter Analysis (Optimized System)





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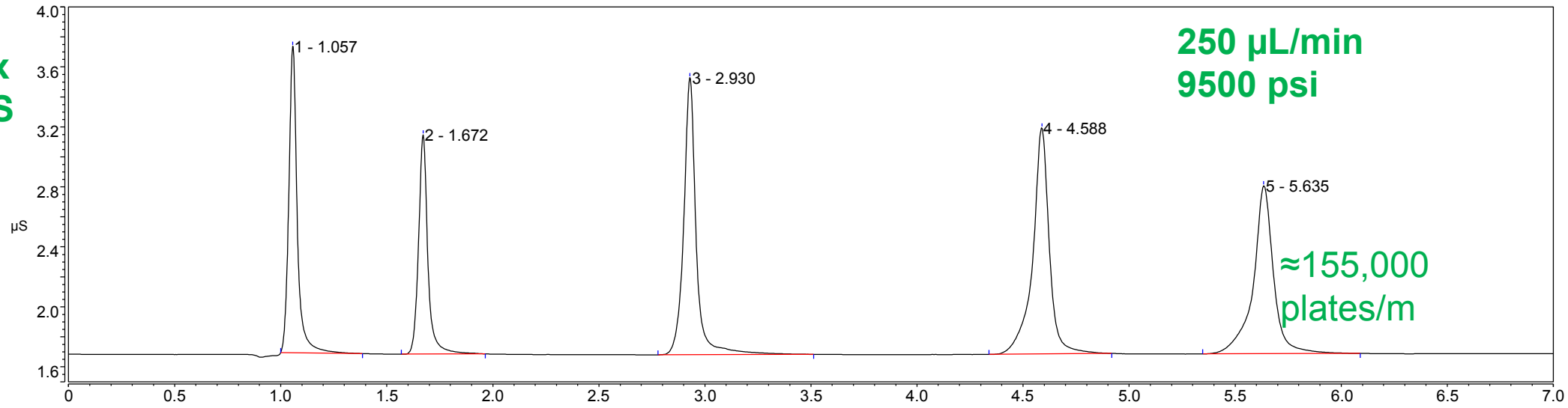
Fast IC Using Short Columns at the Kinetic Performance Limit

2x150 mm 2.5 µm Dionex IonPac AS17-S

23 mM KOH (2-mm suppressor)

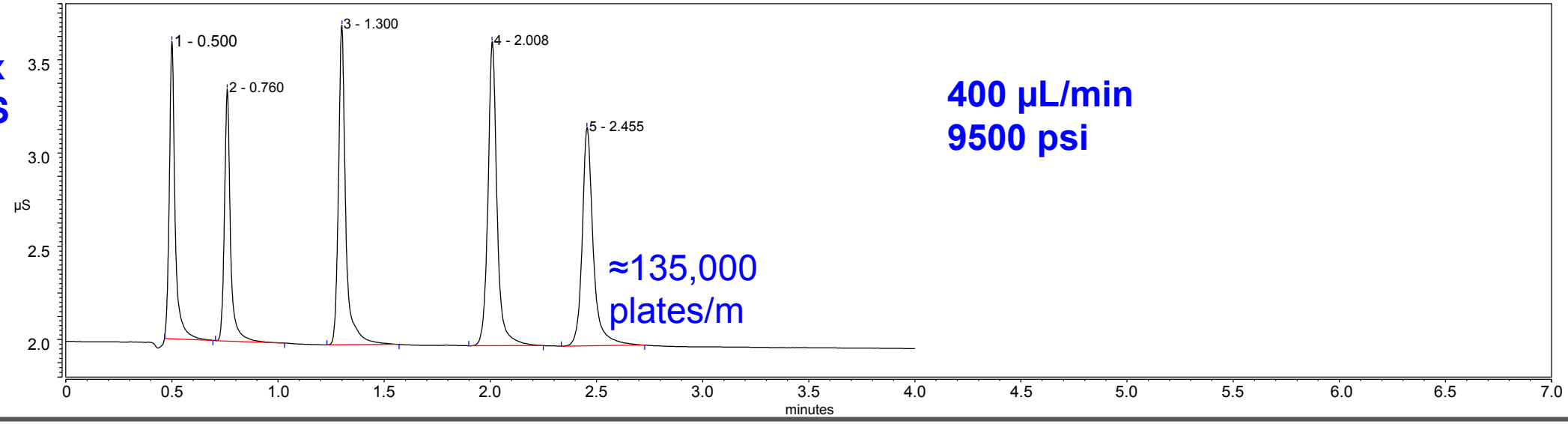


250 µL/min
9500 psi

≈155,000 plates/m

2x100 mm 2.5 µm Dionex IonPac AS17-S

23 mM KOH (2-mm suppressor)



400 µL/min
9500 psi

≈135,000 plates/m

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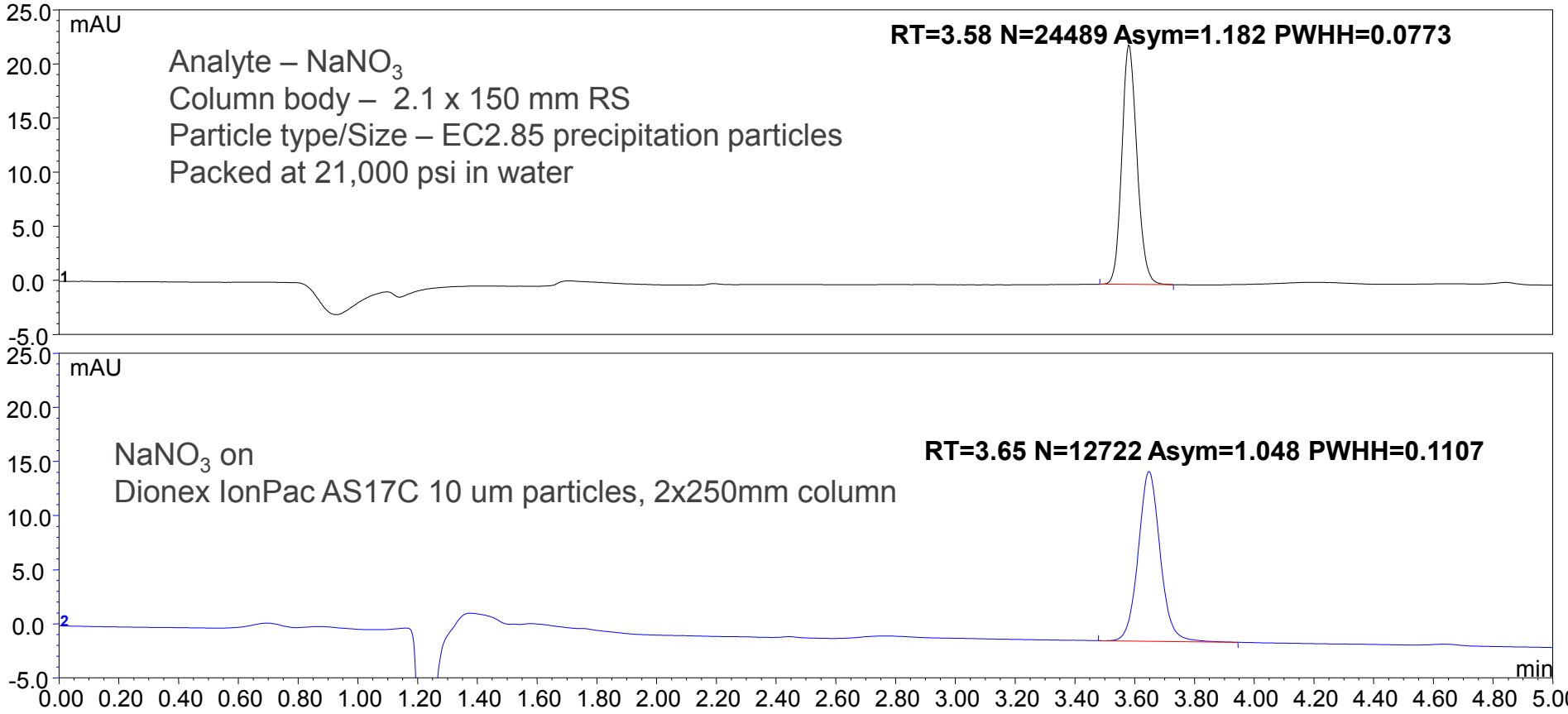
Comparison of 2.85 Micron Particles and 10 Micron Dionex IonPac AS17C Column

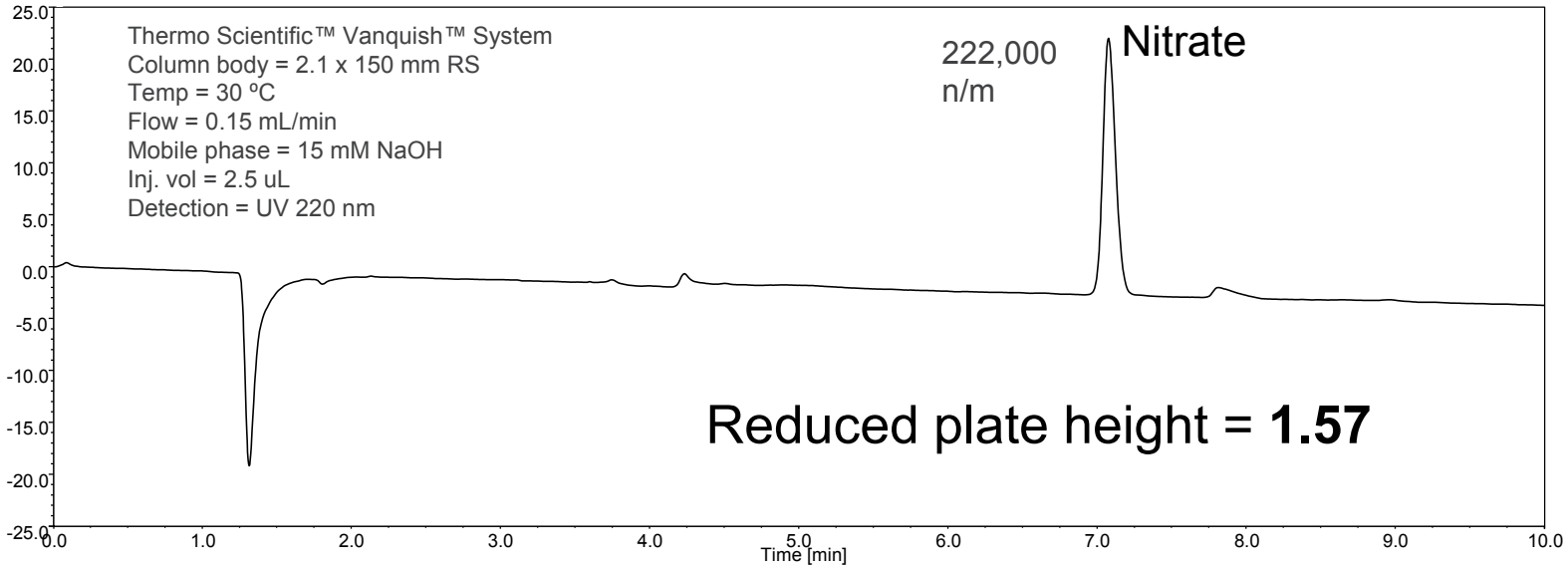


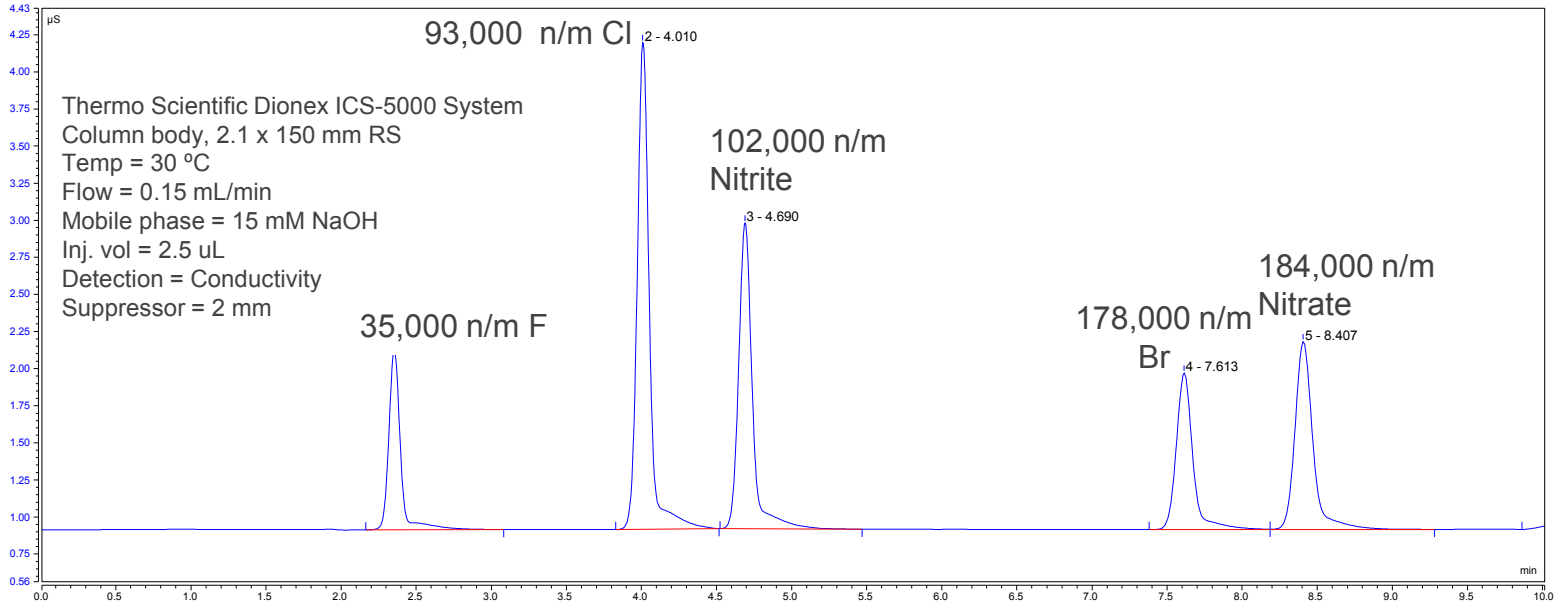
Conditions
Flow rate = 0.25 mL/min
Temp = 65 ºC
Mobile phase = 15 mM NaOH
Detection = UV 220 nm
Injection vol. = 2.5 uL

HETP (EC2.85) = 6.13 µm
Reduced plate height = 2.15

HETP (Dionex IonPac AS17C) = 19.65 µm
Reduced plate height = 1.97

UV Detection and Conductivity Detection at 30 ºC





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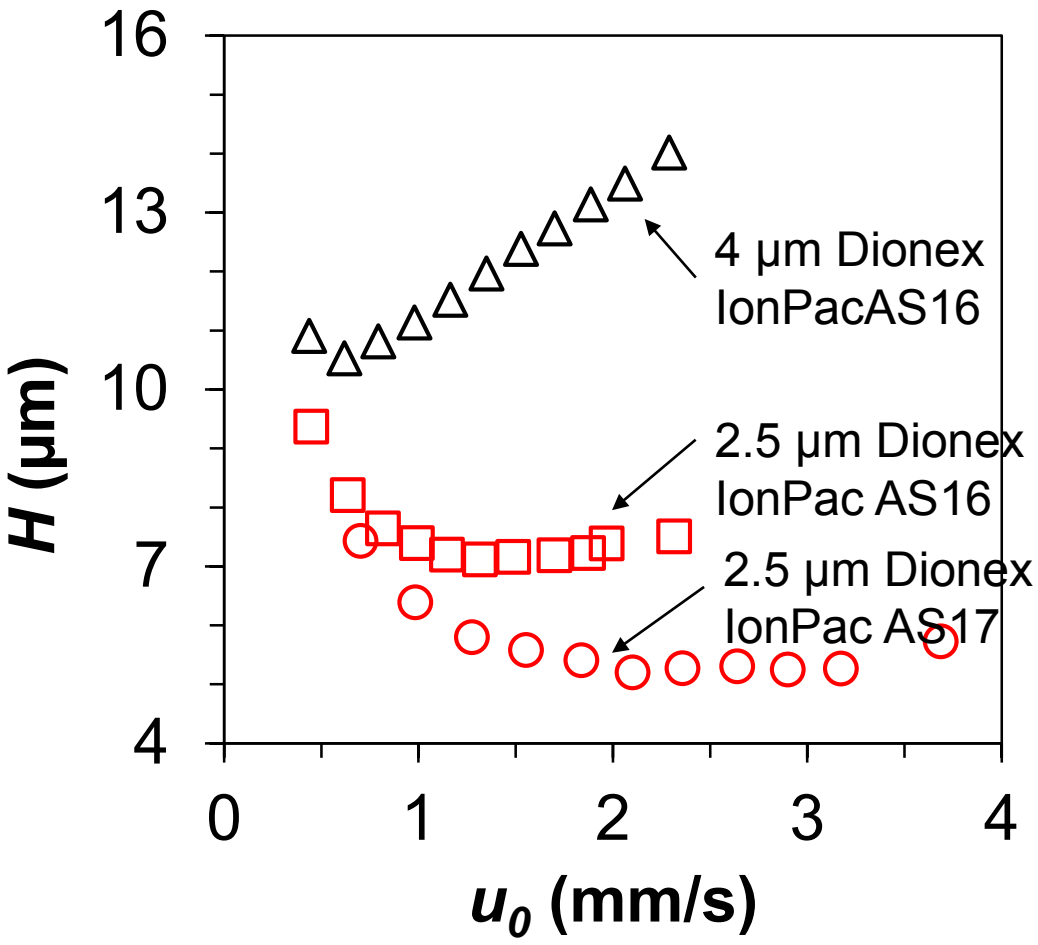
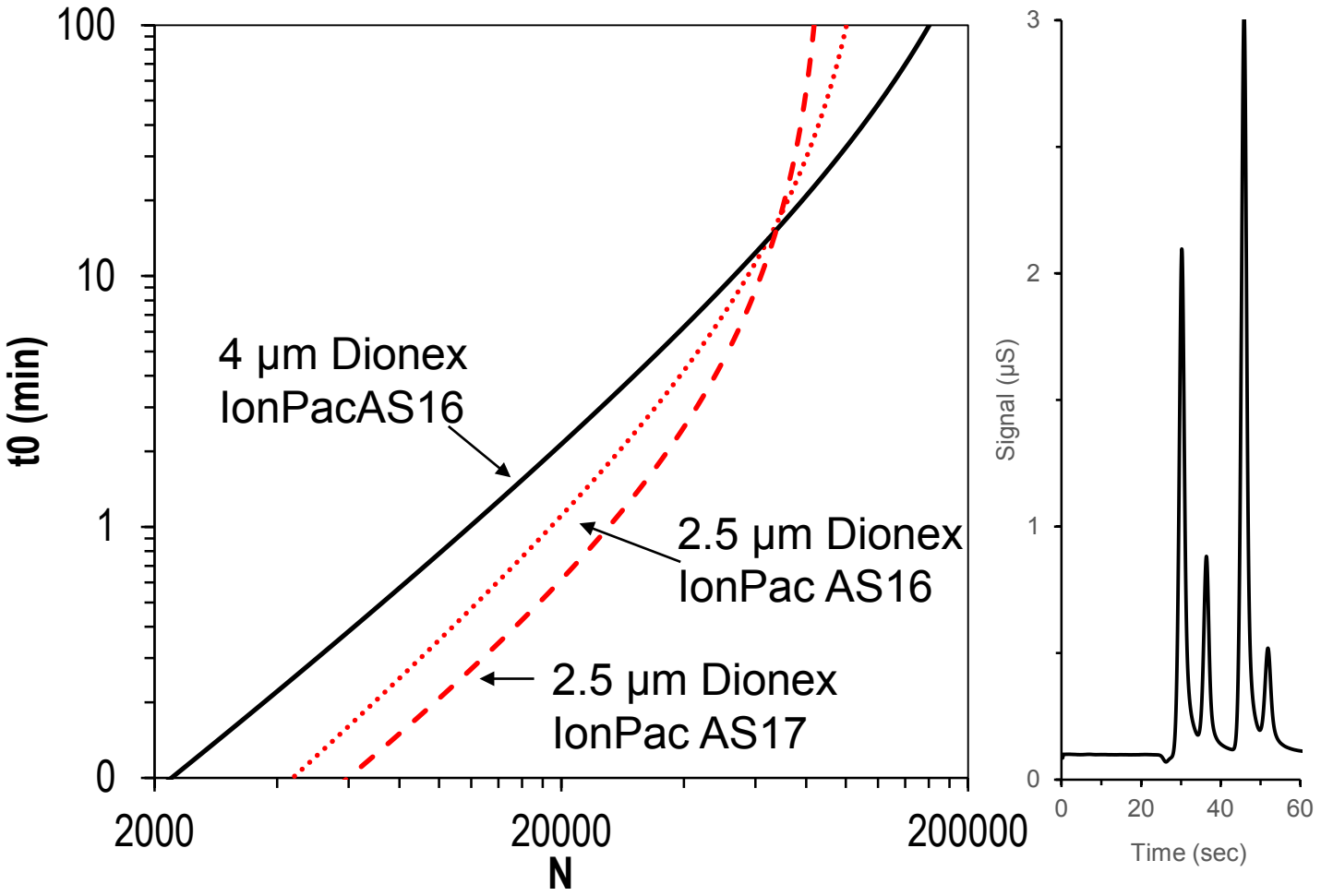
van Deemter and Kinetic Plots





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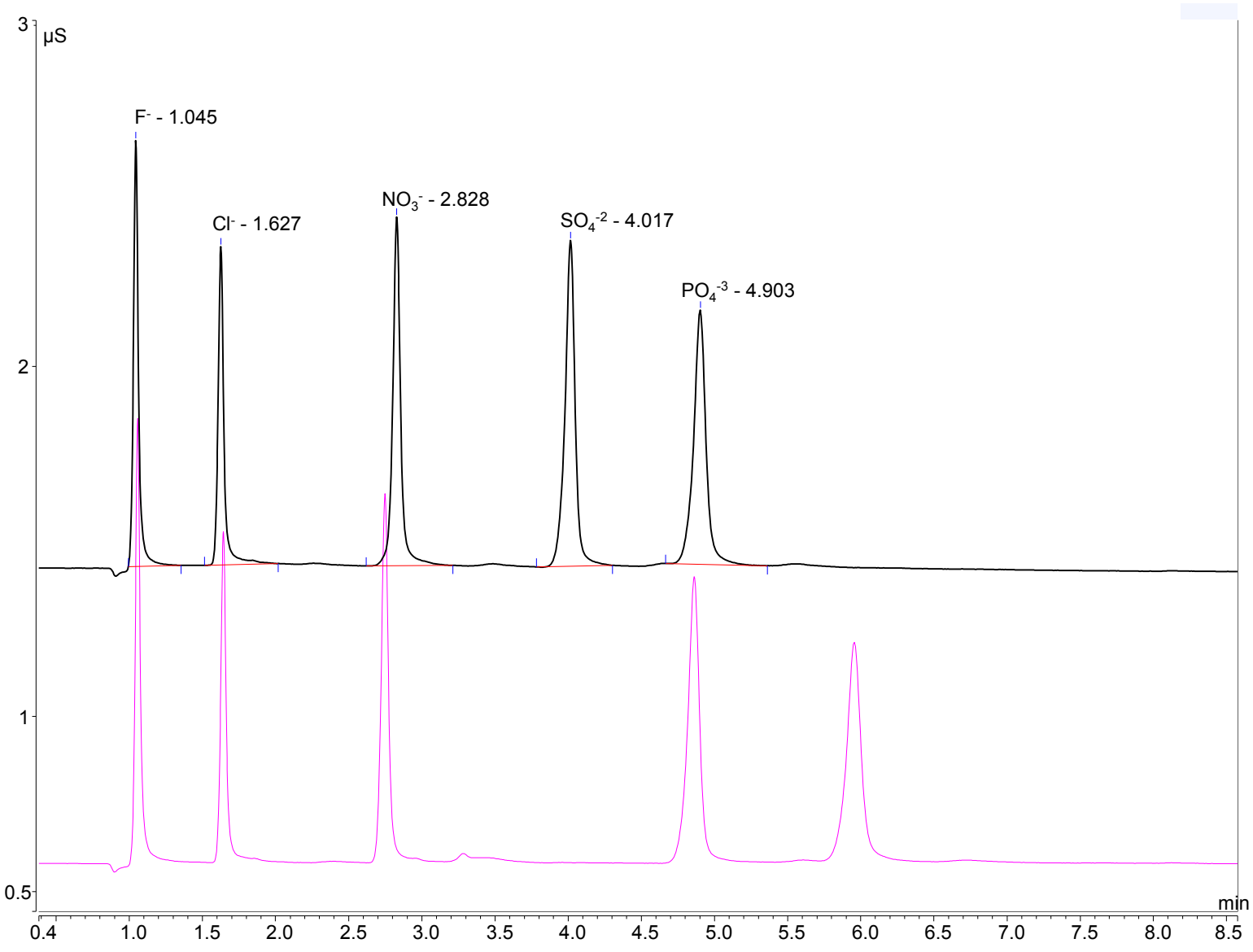
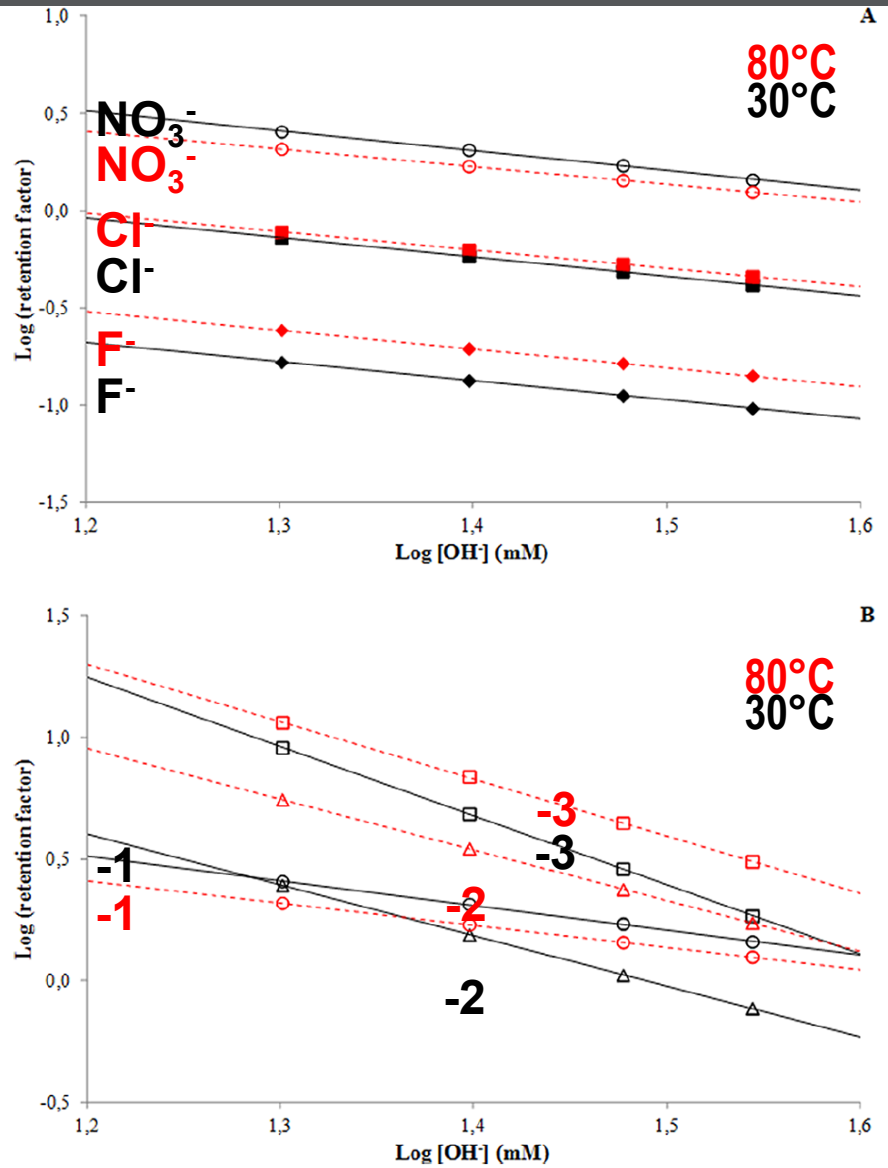
Consequences of New Operating Regimes: Heating Effects





Bert Wouters *et al.* (2014) JCA 1370

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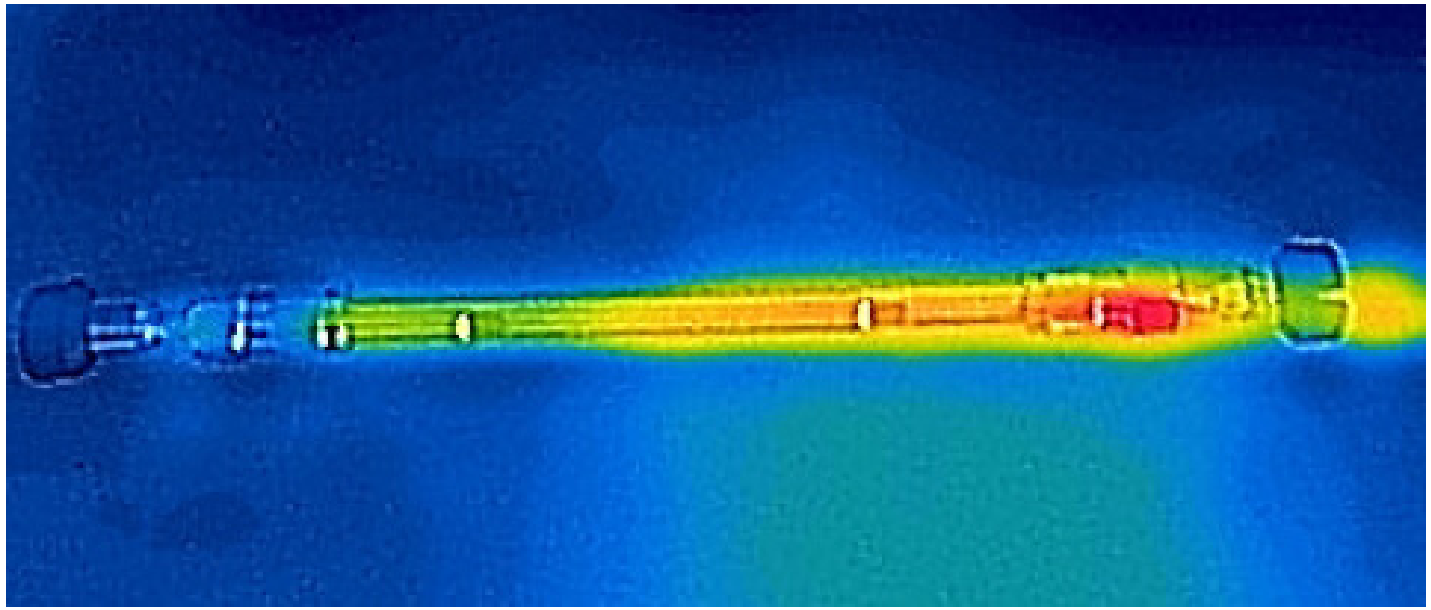
Thermal Heating Effects in Ultra-High-Pressure IC

Percolation of a liquid through columns packed with 2.5 µm particles at ultra-high pressure induces frictional heating, which in turn induced axial and radial temperature gradients over the column

- Temperature sensors connected to column end-fittings (providing quantitative information)
- Heat-mapping camera for visualization

Column inlet



Column outlet

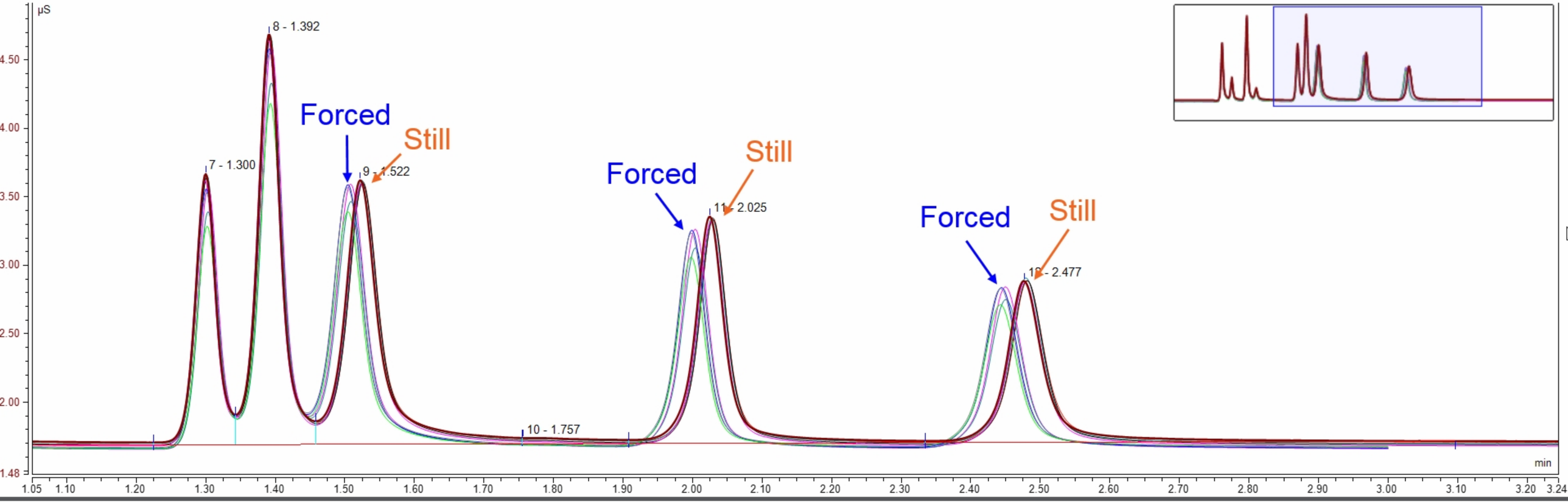
70 Mpa on SS column packed with 2.5 µm particles

Consequences of New Operating Regimes: Heating Effects

Forced @ 400 µL/min:
T_{in}= 29.4°C
T_{out}= 30.9 0C
ΔT= 1.5°C

Still @ 400 µL/min:
T_{in}= 29.1°C
T_{out}= 33.2°C
ΔT= 4.1°C

ΔT= Altered retention behavior – selectivity Affects resolution and efficiency!



Conclusions

- **2.5 µm particles versus 4 µm**
  - Efficiency doubled (plates/m)
  - 2-3 times faster (maintaining plates/column)
  - Highly efficient: enables reduction of column length
  - 100 mm column operated at kinetic performance limit: 25% loss in plates/column, but 5 times faster
- **Future requirements**
  - Ultra-low dispersion suppressor
  - Low dispersion conductivity cell
  - Column oven with still air mode
- **High pressure compatible upgrade requirements**
  - Inert pump
  - Eluent generator, degasser and electrolytic trap column
  - Injection valve and tubing
  - Column hardware