



Tips and Tricks IC Maintenance

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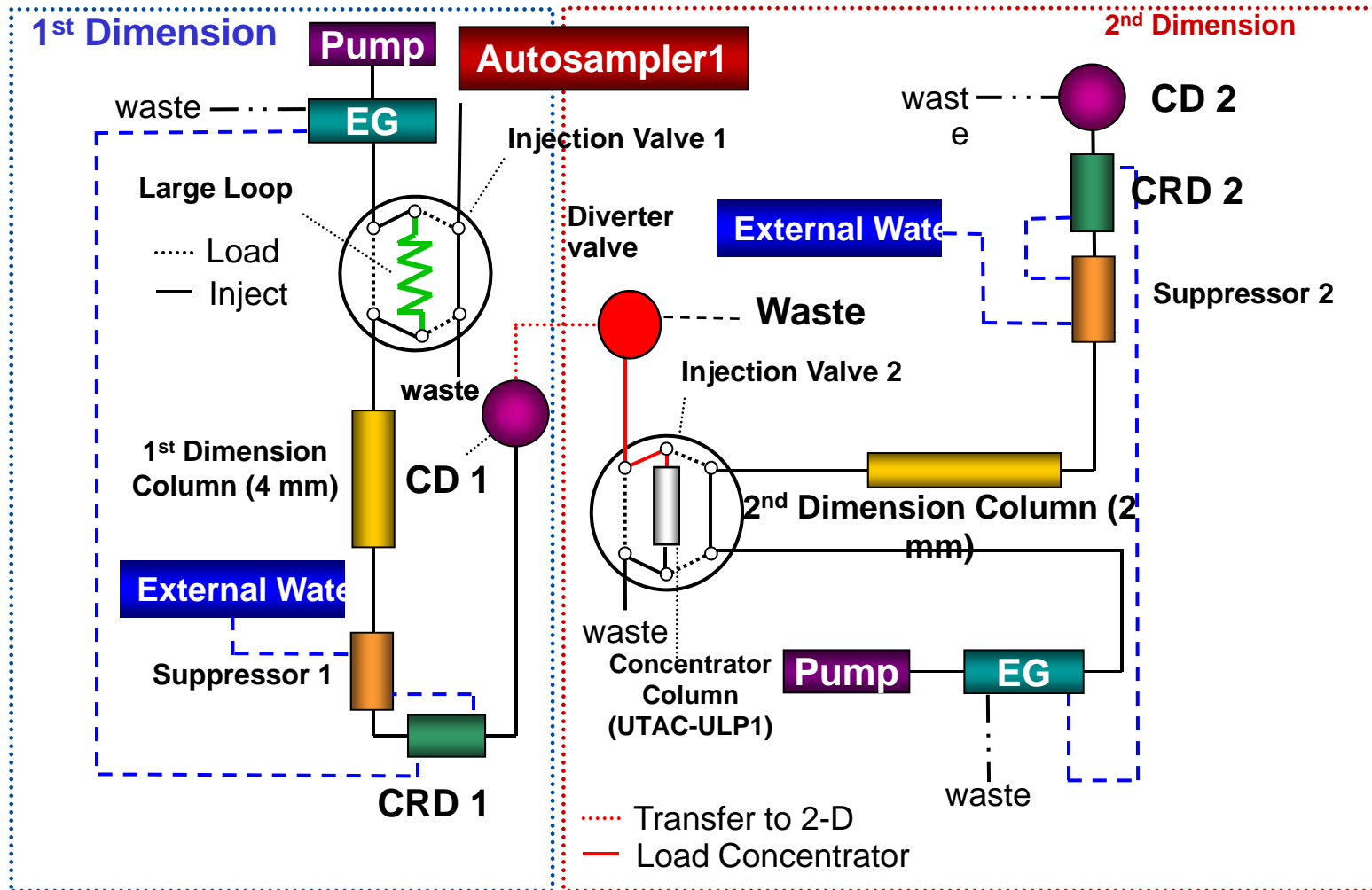
Outline

- The four aspects of an analysis
 - Application
 - Hardware
 - Samples
 - Data interpretation
- Isolating issues to these four aspects
- Logical approach to problem identification and resolution
- Routine Maintenance
- Questions

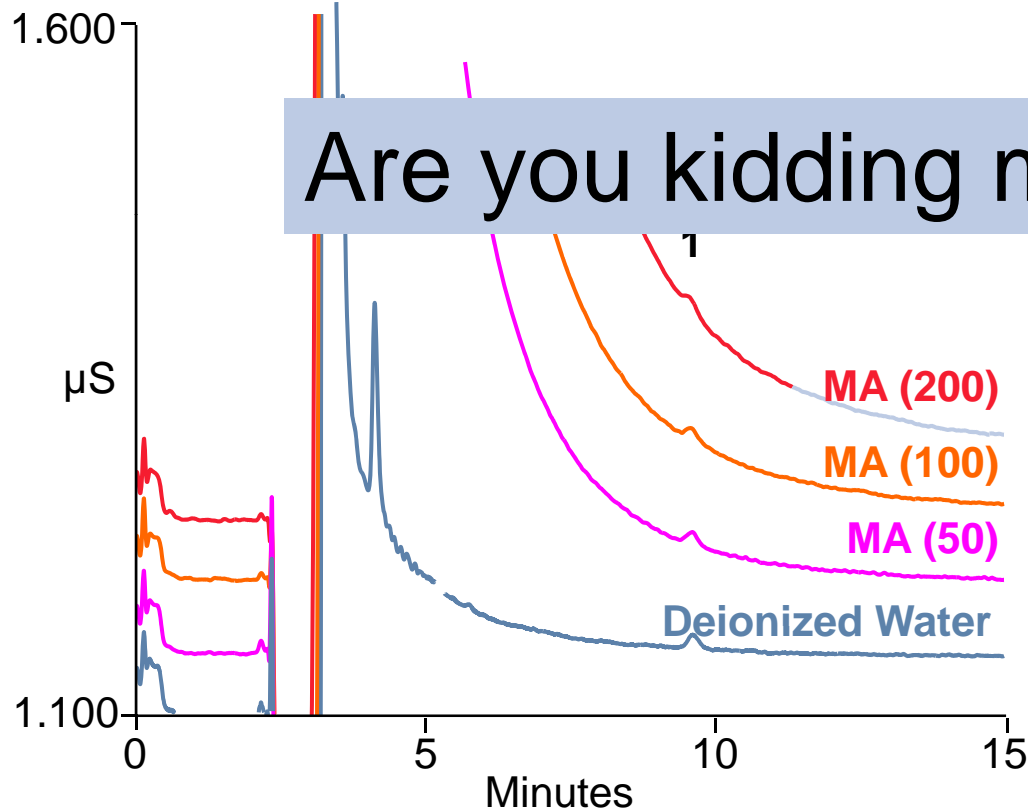
Applications can be complex.....

- Single/Multiple-Pass Setup
- Matrix Elimination Ion Chromatography
- Electrochemical Array Detection
- Chelation IC
- Amino Acid Analysis
- Multidimensional IC
- IC-Mass Spec
- IC-ICP-Mass Spec
- Autoprep
- Auto-neutralization
- Autodilution
- Hyphenated, hydrophobic interaction, split-injected, triple-quad, two-dimensional, ion-pairing, stop-flow, dual column, capillary, nano, ion-exclusion,
-If I have a problem, where do I start!

Hardware can be complex.....



We are seeing lower and lower levels!



Are you kidding me? PPQ?

Column: IonPac® AG16, AS16, 4 mm

Eluent: 65 mM KOH

Eluent Source: ICS-2000 EG with

Flow Rate: 1.2 mL/min

Injection Loop: 1000 µL

Detection: ASRS® ULTRA II,
AutoSuppression®,
external water mode, 193

mA

Samples: MA(x) = X mg/L each Cl⁻,
SO₄²⁻, CO₃²⁻

Peaks: 1. Perchlorate 1 µg/L

Sample Matrices can be complex.....

- Acid Mine drainage
- Oyster tissue
- Seawater
- Acid plating baths
- Urine (yuk)
- Solvents
- Tree sap
- Hydrolysates

So we have...

- Complex Applications
 - Complex Hardware Configurations
 - Extremely Low Detection Limits
 - Complex Sample Matrices
-
- So what do we do when something goes wrong?

Sometime all of these come together.....

- Customer
 - “Bob, I am having problems with my IC”
- Me
 - “What are you doing and on what system?”
- Customer
 - “I am doing low level bromate and sialic acids on a dual two-dimensional IC with post column addition, on a combination of ICS5000 and ICS2100 with a split from the conductivity and amperometry detectors going to a triple-quad mass spec with home-made source and the other part of the split routed to an ICP-MS.
- Me
 - “What is your sample matrix?”
- Customer
 - “It is a hydrolysis of oyster tissue from oysters that were raised in old mine in a combination of solvents and seawater.”
- Me
 - “Sorry , I’m off sick today!”



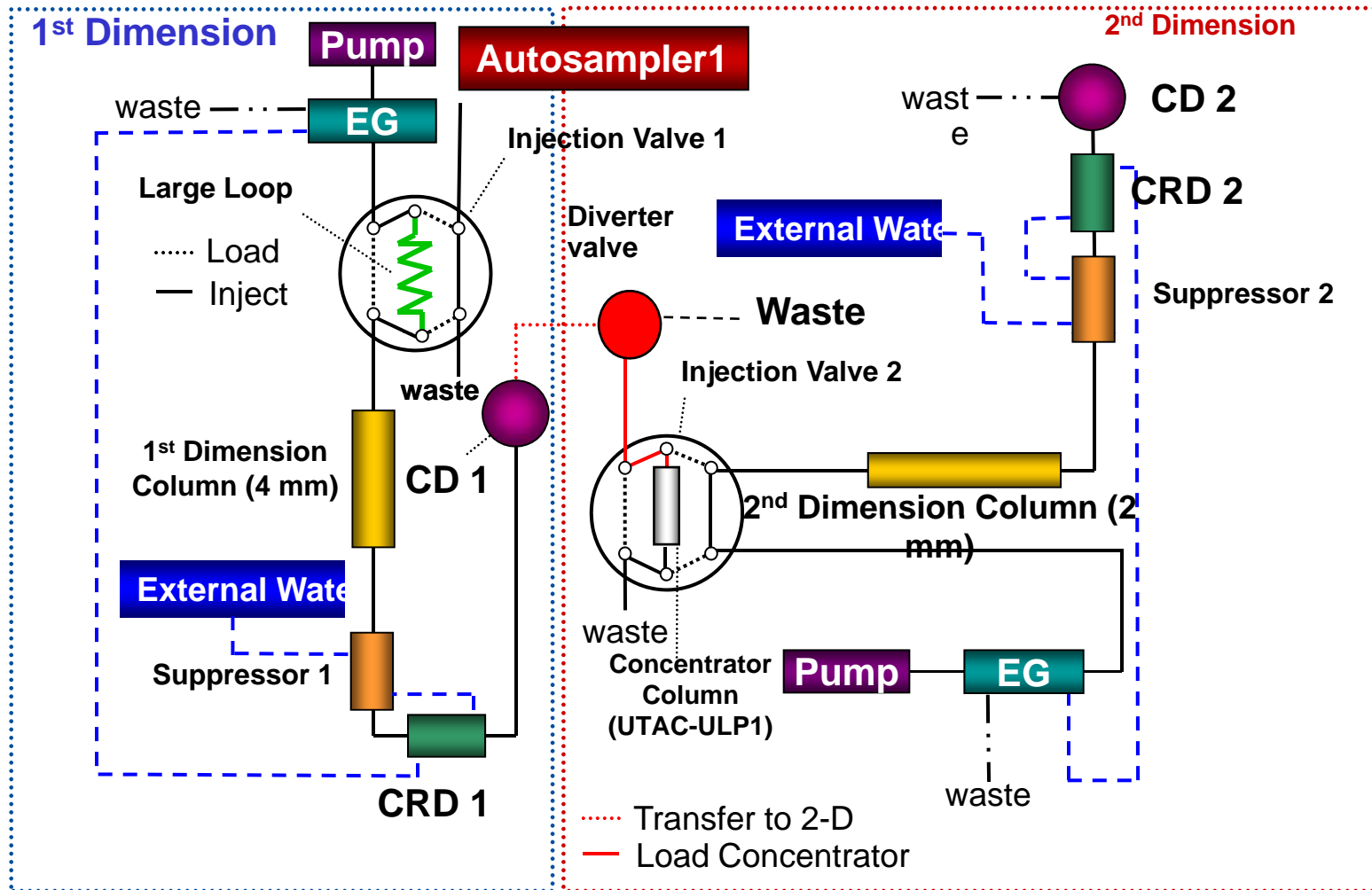
Tips and Common Sense IC Maintenance

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Hardware can be complex.....



Common Sense approach to Hardware issues

- Mobile Phase
- Pump
- Injector
- Separator
- Detector
- Data System

Hardware issues are actually simple 90% of the time

- **Mobile Phase**
 - Retention issues, Noise when accompanied by high background
- **Pump**
 - Noise (cyclic) Nothing else without obvious effects
- **Injector**
 - Reproducibility of all analytes (not one directional), linearity of all analytes, contamination
- **Separator**
 - Loss of retention, resolution, peak shapes (will not improve)
- **Detector**
 - Noise (not cyclic), Low sensitivity, Reproducibility (one directional, trending)
- **Data System**
 - Its called a calibration curve, not a calibration straight line.

Application Sense Application Troubleshooting

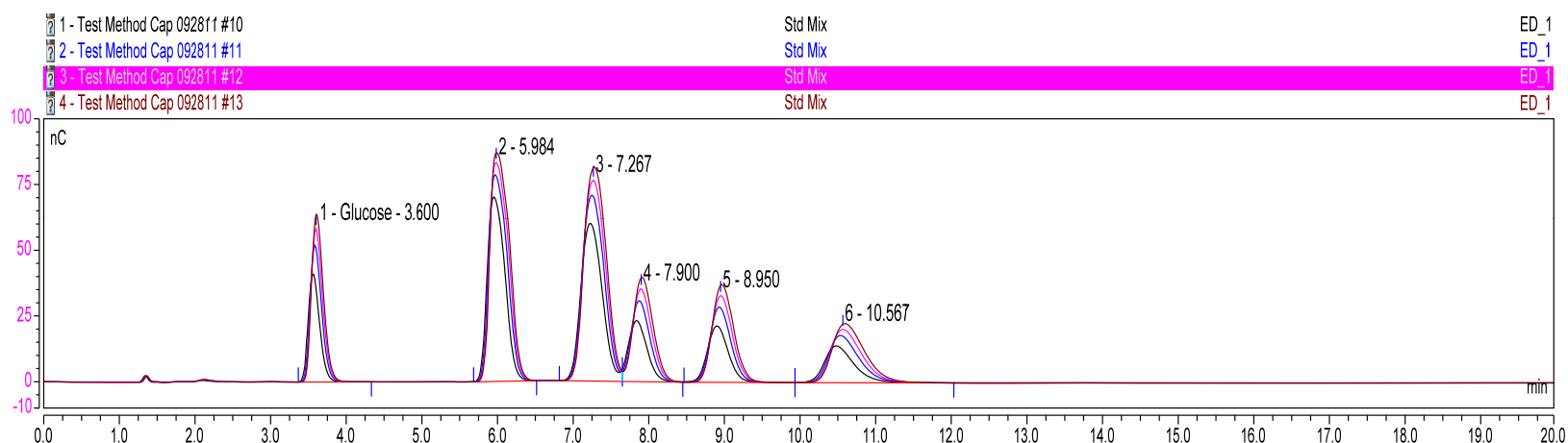
- Trace Analysis in Ion Chromatography (IC)—Current Practices
 - Three Simple Applications:
 - Single-Dimensional, Single/Multiple-Pass Setup
 - Anions in Water
 - Matrix Elimination Ion Chromatography
 - Cations in Water
 - Perchlorate Analysis
 - Carbohydrates in water
 - Bromate Analysis
 - Loop Injection
 - Haloacetic Acid Analysis
 - Syringe
 - Multidimensional 2d IC
 - Standards from a trusted third party
 - IC-Mass Spec
 - IC-IC-Mass Spec
 - It may be complex to figure out why my 0.15 ppb bromate does not look good
 - Isocratic
 - Gradients
 - It is easier to see what 1 ppm of chloride and sulfate look like on a 25 uL
 - Hopped, hydrophobic interaction, split-injected, triple-quad, two-dimensional, hydrophobic interaction, stop flow, dual column, capillary, nano, virtual column will show me if I am unsure.
 - ion exclusion
 -If I have a problem, where do I start!
 - And who is Van Deemter and why is he giving me a hard time?.

Common Sense for samples

- Polymeric IC columns have a very high tolerance for tough sample matrices.
 - Being polymeric, they are tough and rugged, very much like your Tupperware™ containers at home.
 - What can damage your Tupperware™?
 - Heat and Spaghetti Sauce (color)!
- Colorful Solutions can cause damage to the separator.
- Heat can damage the separator.
 - Most common heat source is acid/base related.
- Particulates and precipitates can clog.
- High ionic strength matrices can effect the chromatography.
- If you are running PPT and inject PPM, expect a few days of issues.

Troubleshooting Example

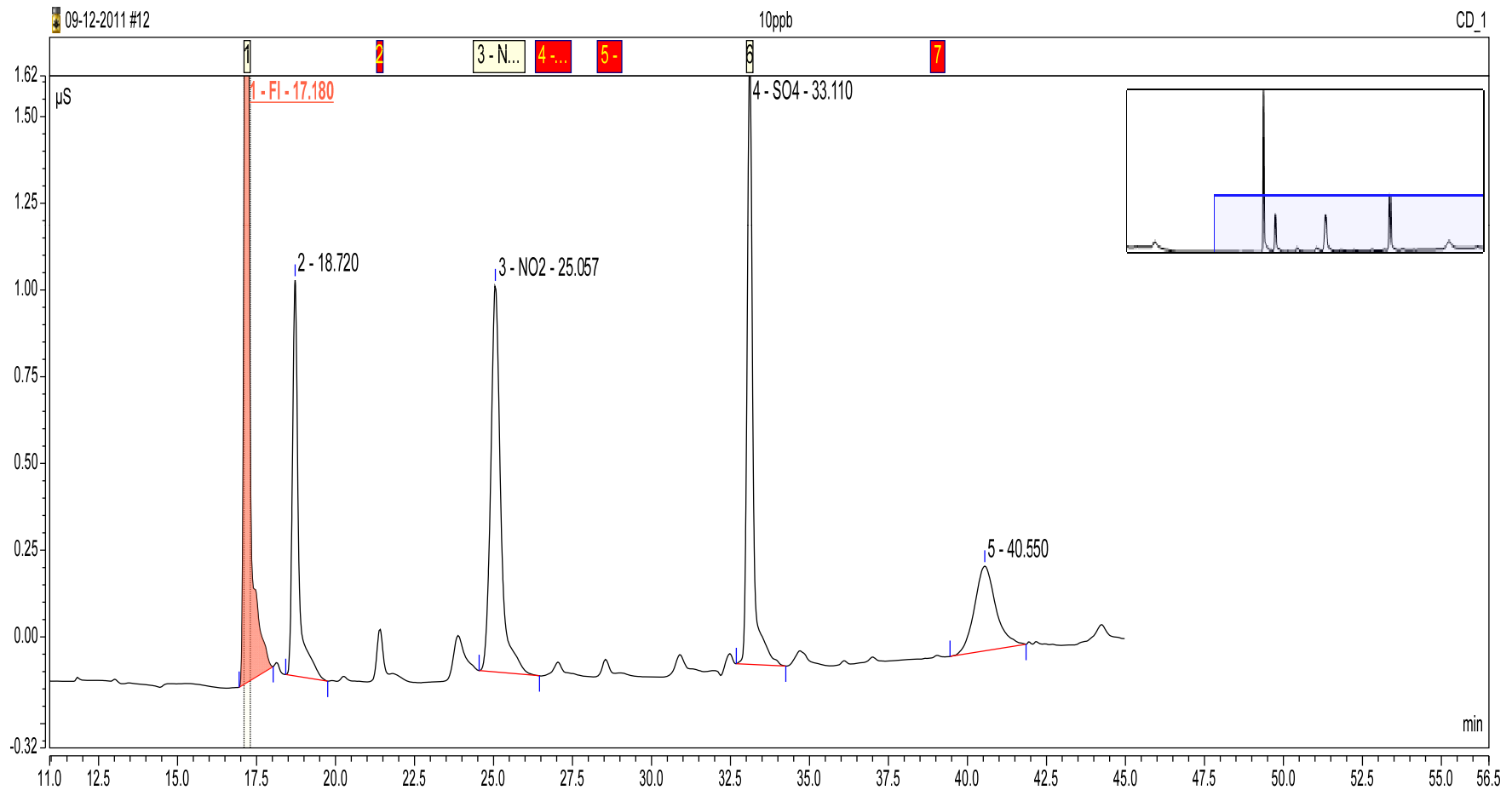
Carbohydrates on a CAP system have reproducibility issues.



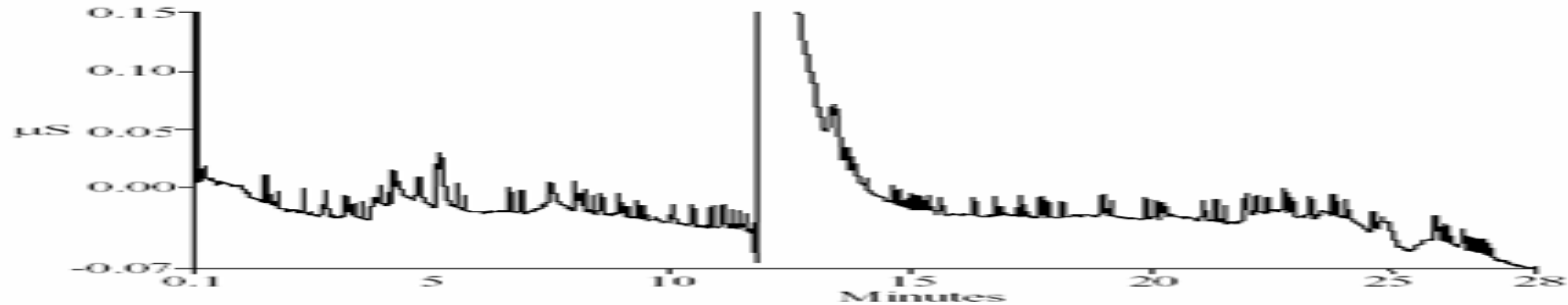
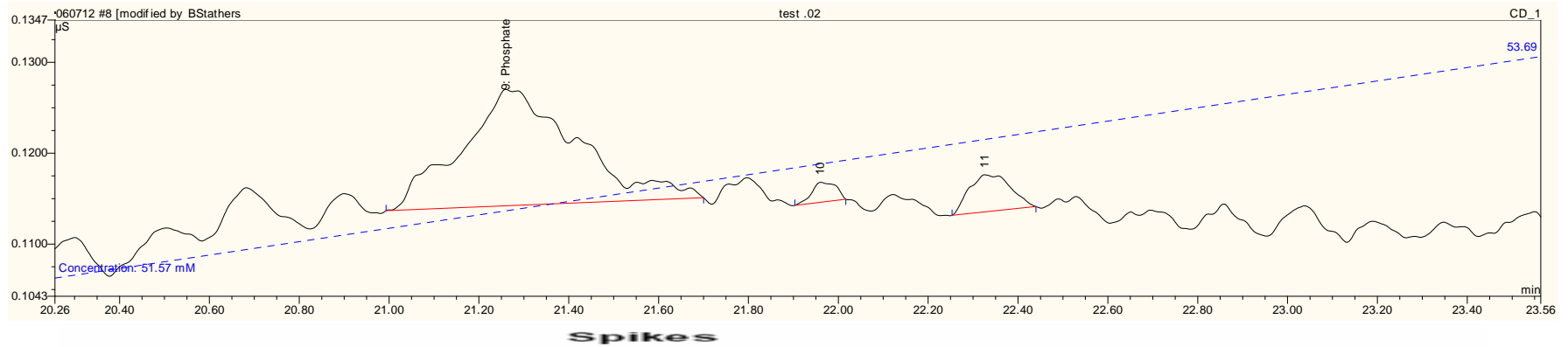
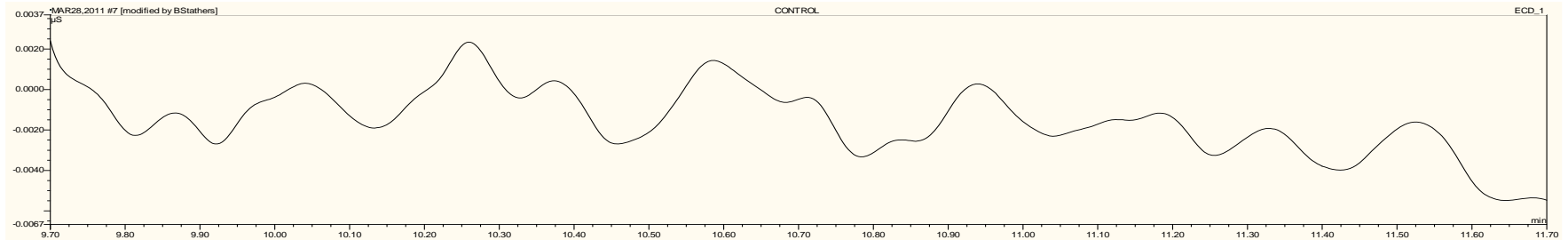
Glucose	Area	Height
Inj1	8.2064	47.06
Inj2	7.2599	41.01
Inj3	9.3525	52.01
Inj4	10.5758	58.28
Inj5	11.7355	63.72

Troubleshooting Example

Capillary Anions – Spurious Peaks

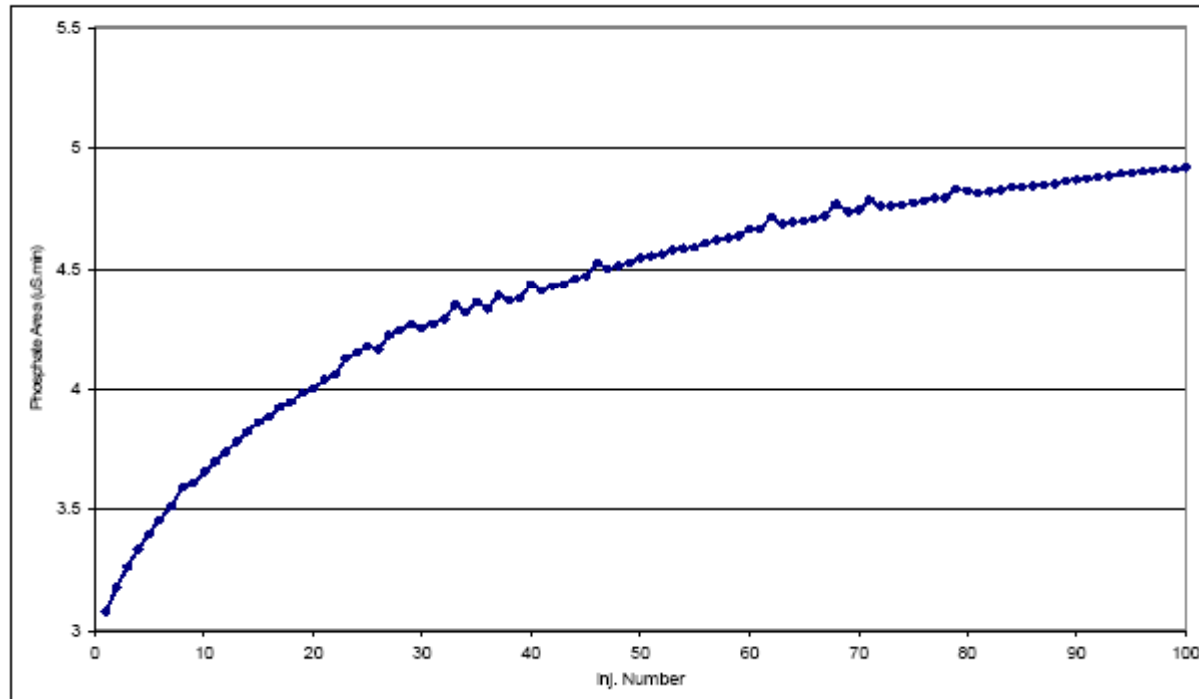


Examples of Noise



Example of a reproducibility issue.

My QC falls out high as I run throughout the day!.



Trend plot showing Increasing Peak Areas after a suppressor was operated with the power off for 20 minutes.

RFIC Scheduled Maintenance.

- CAP systems
 - Run for 18 months
 - Refill DI every 3 months
 - Do not shut them off!
 - Upper Oven at 15-20 degrees!
 - Seals and Check valves
 - Run DI water
 - Piston stroke every couple of minutes
 - Long CR-ATC, CRD, SRS life
 - Could make it 18 months
 - PdH reference (Carbo's)
 - Should last the duration
 - Shut off cell, replace working, turn on cell
 - A minute and back on
 - Do not cut your own tubing
 - Watch for voids, use proper fittings
 - Do not use seal wash



RFIC Scheduled Maintenance.

- 2mM systems
 - Rarely shut them down
 - Only long term like vacation
 - 720 mL of DI per day @ 0.5 mL/min
 - Top it off, keep it running
 - Swap bottles every 3 to 6 months
 - Seals
 - Run DI water
 - 5-10 strokes per minute.
 - Long CR-ATC, CRD, SRS life
 - Annually seals and check valves
 - Do not use seal wash



RFIC Scheduled Maintenance.

- 4mM systems
 - Shut down as convenient
 - 1440 mL of DI per day @ 1.0 mL/min
 - Swap bottles every 3 to 6 months
 - Sunlight and water = Bacteria
 - Seals
 - Run DI water
 - Pump works harder
 - Long CR-ATC, CRD, SRS life
 - Annually seals and check valves
 - If 8-10 hours a day
 - Twice a year if 24-7
 - Do not use seal wash



Hand-made mobile phase Scheduled Maintenance.

- 4mM systems
 - Shut down as convenient
 - 1440 mL of DI per day @ 1.0 mL/min
 - Swap bottles every 3 to 6 months
 - Sunlight and water = Bacteria
 - Seals
 - Salts cause piston etching
 - Use the seal wash
 - Monitor the seal wash
 - Segregate seal wash if using two different chemistries.
 - Long CR-ATC, CRD, SRS life
 - Annually seals and check valves
 - If 8-10 hours a day
 - Twice a year if 24-7



Autosampler Scheduled Maintenance.

- Annually
 - Needle
 - Needle Port
 - Buffer tubing
 - Rotor/Stator

- Always use plastic vials for IC
 - No Glass
- Always use split septa
 - It is a PEEK needle



Questions.

- Thank you!