Overcoming Challenges in Elemental Speciation Techniques

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Overview

- Plumbing Considerations
- Why LC Connections Matter
- Chromatography Examples
- ICP-MS Settings





Plumbing for Column Compartment Valve

From Column
To ICP-MS (Splitter)
ISTD Loop
ISTD from PeriPump
ISTD Return to Bottle
ISTD Loop

Position 1 – Loop Fill 1-2; 3-4; 5-6

Position 2 – ISTD Inject 1-6; 2-3; 4-5



Plumbing for Passive Seat Wash

<u>Note 1</u> – 0.5mm ID tubing (PEEK Orange) to port 4 on ALS valve for passive needle seat wash

<u>Note 2</u> – 0.12mm ID tubing (PEEK Red) to ICP-MS





ALS Valve Plumbing Diagram

From Pump
Needle Loop
Plug
From T-Splitter (0.5mm ID)
Needle Seat
To Column

Position 1 – Mainpass 1-2; 3-4; 5-6

Position 2 – Bypass 1-6; 2-3; 4-5

<u>Note:</u> When in Bypass and needle is lifted, mobile phase will backflush needle seat.



Final Plumbing Example







Why Plumbing Matters: Stainless vs. Poly Fittings

Stainless Steel:

- Agilent uses Swagelok type fittings with front and back ferrules
- Also available with a long nut

PEEK (<400 bar system pressure):

- · Connections are changed frequently
- · Connecting columns
- Pressure is less critical
- Fits on SS or PEEK tubing

Polyketone:

- · Easy, hand tightened column connection
- Used up to 600 bar (PN: 5042-8957)
- Fits on SS Tubing









Why Plumbing Matters: Types of Fittings







Swagelok

- Two piece ferrule
- Used on Agilent LCs
- Short nut
- · Also available with long nut

Parker

- One piece ferrule
- Short nut
- Very similar to Swagelok

Waters

- Longer nut
- Used on Alliance systems

Why Plumbing Matters: Types of Fittings



0.09 in.

Parker











Rheodyne





Valco





Upchurch









Leak

Peak shape problem

No dead volume















Why Plumbing Matters: Don't Forget Dispersion





Experimental – Arsenic

- Agilent 7800 x lens, MicroMist, Ni Cones
- Agilent 1260 Biolnert, Quat Pump, Well-Plate ALS, TCC 6-port Biolnert Valve

Column	Hamilton PRP X-100 SAX 250mm x 4.6mm, 10um
Mobile Phase	6mM Ammonium Phosphate / 6 mM Ammonium Nitrate / 2% MeOH / pH 6.2
Flow Rate	1.0 ml / min (isocratic)
Injection Volume	100 µl
Gas Mode	Не
Integration Time	1.0 sec
Total Run Time	~20 min



Toxicity and pKa of Arsenic Species





Experimental – Arsenic 10pg each species





Experimental – Arsenic 5,000pg each species





Experimental – Arsenic #2

- Agilent 7800 x lens, MicroMist, Ni Cones
- Agilent 1260 BioInert, Quat Pump, BioInert Multi-Sampler, TCC 6-port BioInert Valve

Column	Hamilton PRP X-100 SAX 150mm x 4.6mm, 10um
Mobile Phase	6mM Ammonium Phosphate / 6 mM Ammonium Nitrate / 2% MeOH / pH 6.2
Flow Rate	1.5 ml / min (isocratic)
Injection Volume	100 µl
Gas Mode	Не
Integration Time	1.0 sec
Total Run Time	~7 min



Experimental – Arsenic 10pg each species





Experimental – Arsenic 500pg each species





Experimental – Arsenic Comparison





Experimental – Arsenic Contamination



Watch out for contamination from the labware

- Amber glass may contain low levels of arsenic
- This includes sample vials
- Clear glass usually OK
- PFA/FEP bottles are best



Examples: Add PEEK Red Before Column





Examples: Add PEEK Blue Before Column





Examples: +0.5% HCI





Examples: +0.5% Nitric Acid





Experimental – Selenium

- Agilent 7800 x lens, MicroMist, Ni Cones
- Agilent 1260 Biolnert, Quat Pump, Well-Plate ALS, TCC 6-port Biolnert Valve

Column	Metrosep A Supp10 250mm x 4.0 mm
Mobile Phase	A – DIW / 2% Acetonitrile B – 200 mM Ammonium Nitrate / 2% ACN / pH 9.5
Flow Rate	1.0 ml / min (gradient)
Injection Volume	100 µl
Gas Mode	H ₂
Integration Time	0.8 sec
Total Run Time	~30 min



Experimental – Selenium 5pg each species





Experimental – Selenium 5,000pg each species





Experimental – Chromium

- Agilent 7800 x lens, MicroMist, Ni Cones
- Agilent 1260 Biolnert, Quat Pump, Well-Plate ALS, TCC 6-port Biolnert Valve

Column	Agilent Bio WAX 50mm x 4.6mm, 5um
Mobile Phase	100 mM Ammonium Carbonate / 5 mM EDTA
Flow Rate	1.0 ml / min (isocratic)
Injection Volume	100 µl
Gas Mode	Не
Integration Time	1.0 sec
Total Run Time	~3 min



Experimental – Chromium 3+ @ 5000pg Chromium 6+ @ 50pg





ICP-MS Instrument Settings

- Nebulizer Gas Flow Rate
- Waste Pump Rate
- Conditioning
- Detector Integration Time











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Mark Powell









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