

LeadScape Microflow workstation (LS1/ Zirconium/Optiflow/6500)



Planning/Current State:

LeadScape will bind to Zirconium method editor panel to set up LC gradient
.zrm method will then be treated as a 'starter method'

Zirconium software will be used directly to create 'settings' file (.zrs file)
.zrs file will be imported into LeadScape method

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Zirconium Method Editor

Configuration Parameters:

- Pump Type: Zirconium Ultra
- Description: [Empty text box]
- Continuous Flow Mode: Auto Initialize
- CL Flow Control: Auto Start
- Target Initial Pressure: [Empty text box] bar
- B Start Delay: 0 s
- Solvent A: H20(auto)
- Solvent B: ACN
- Compartment Temp.: off °C
- Column Temperature: off °C
- Equilibration Time: 30 s
- Max. Ready Time: 3000 s
- Total Volume: 515.000 uL
- Left Volume A: 890.50 ul (66.0%)
- Left Volume B: 1294.50 ul (95.9%)
- Ready Output: none
- Initialize Input: none
- Start Input: DI1
- Enforce Refill Input: none
- Refill after max. runs: unlimited

Method Run Sequence Table:

Run Time [s]	Flow [uL/min]	%A	%B	Const. Pressure	TE1	TE2	TE3	TE4	PO1	PO2
0	10	90	10							
1	10	90	10							
5	10	90	10							
50	10	10	90							
55	10	10	90							
60	10	90	10							

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Current
Control/testing
panel in
LeadScape

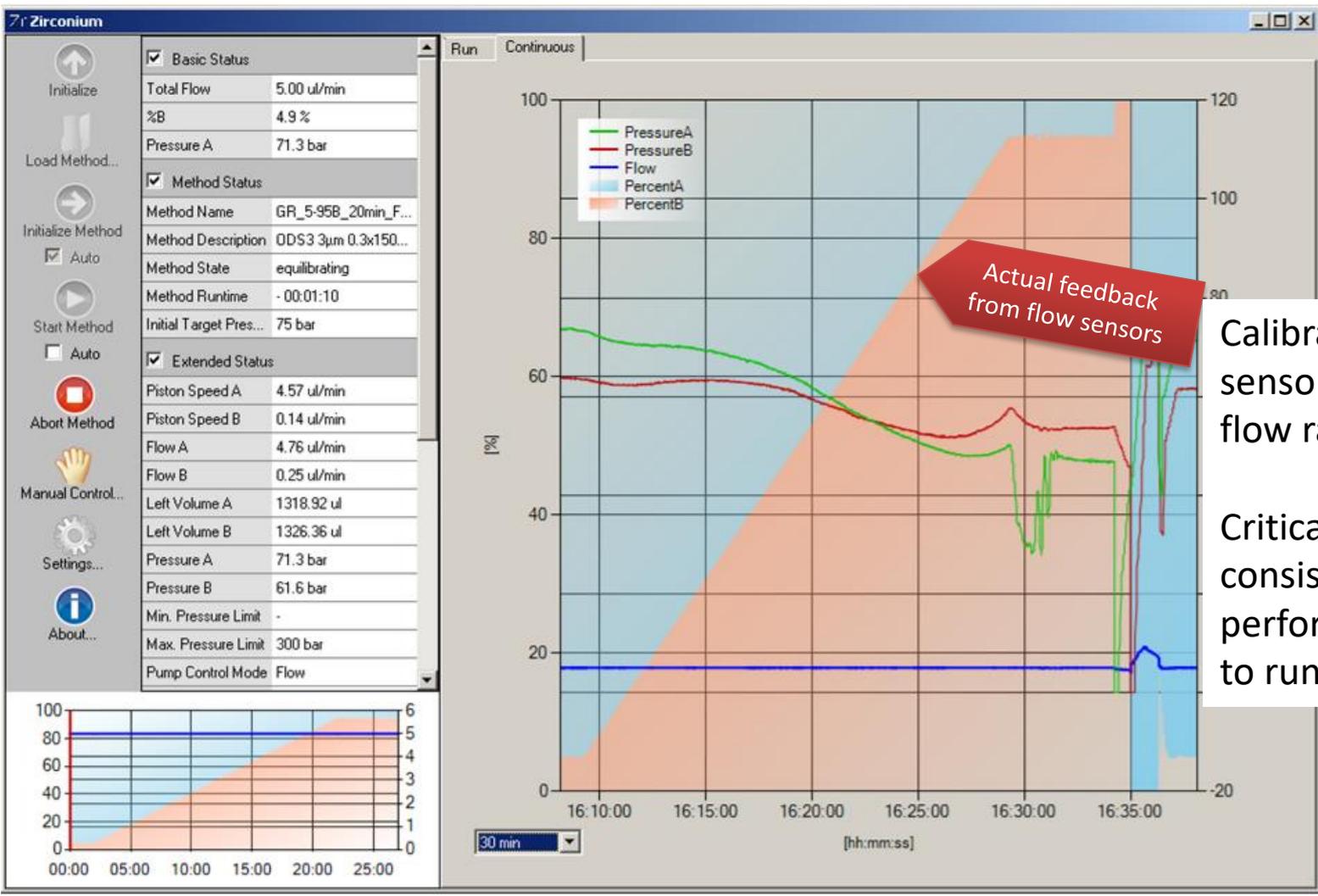
Import zrm file
Import zrs file

The screenshot displays the 'ZirCon - Test2.vi' software interface. At the top, there is a menu bar with 'File', 'Edit', 'View', 'Project', 'Operate', 'Tools', 'Window', and 'Help'. Below the menu bar are several control panels:

- transport**: A panel with a 'Serial' transport type, a 'connectionString' field set to 'COM8', and 'Connect' and 'Disconnect' buttons.
- Pump**: A panel with two file selection buttons (indicated by red arrows from the text 'Import zrm file' and 'Import zrs file'), 'Load Method', 'Load Settings', 'Initialize Method', and 'Abort Method' buttons. It also displays 'Method State' and 'Status' as 'NoMethod'.
- Pump Flow State**: A panel showing 'Time Stamp' (3:09:15.512 PM 10/12/2018), 'Flow Rate' (0 ml/min), '% B' (0), and 'Pressure' (0 psi). It includes 'Acknowledge' buttons for 'Device', 'Channel A', 'Method', and 'Channel B'.
- Flow Rate Graph**: A graph with 'Flow Rate [ml/min]' on the left y-axis (ranging from -1.0 to 1.0) and '% B' on the right y-axis (ranging from -100 to 0). The x-axis is labeled '0' and '1023'. The graph shows a blue line at 0.0 ml/min and a red line at 0% B. A legend at the bottom of the graph shows checkboxes for 'Flow', '%B', and 'Pressure'.
- Volume Remain**: A panel with 'Aspirate A', 'Dispense A', 'Aspirate B', and 'Dispense B' buttons.

At the bottom of the window, there is a 'Tab Control' label.

Flow Controller Performance

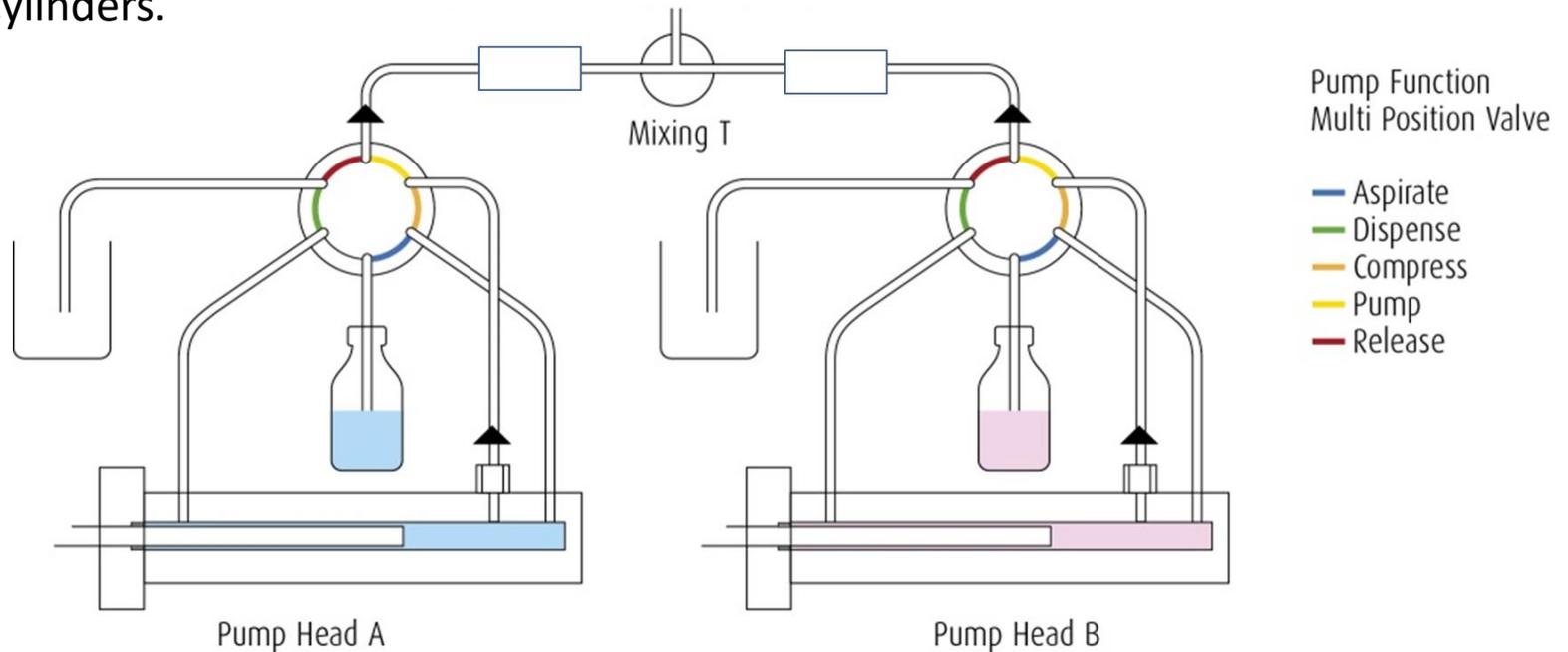


Calibrating the flow sensor is important at flow rate $20 \mu\text{L}/\text{min}$

Critical for obtaining consistent LC performance from run to run

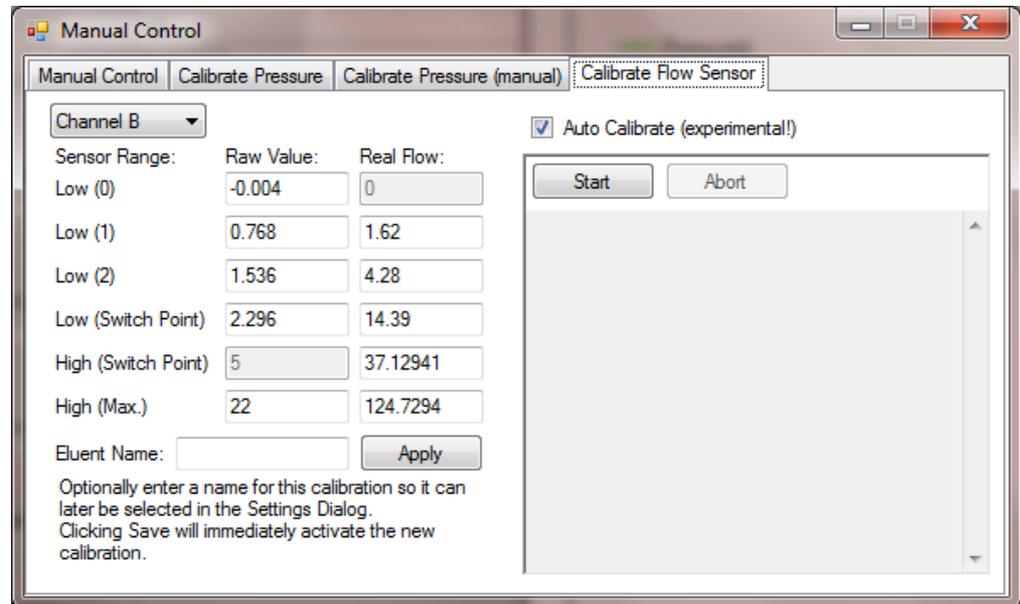
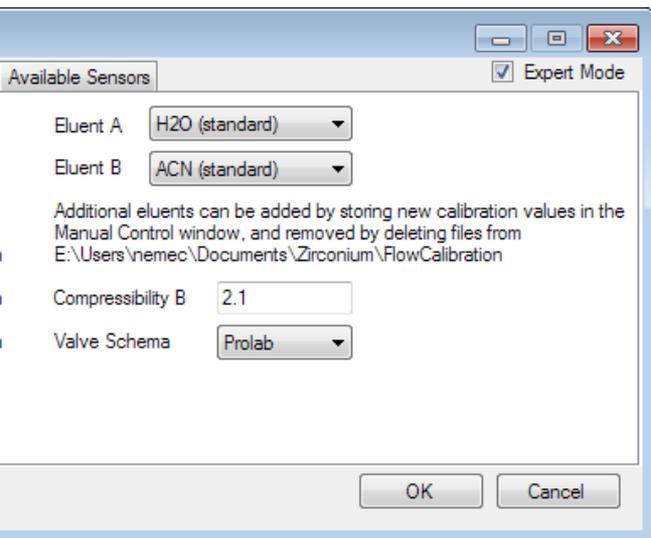
Creating .zrs file (calibrating the flow sensor)

The closed-loop flow control algorithm uses feedback from two flow sensors placed right before the mixing tee in order to set the target pressures for both pump cylinders.



The temperature-based flow sensors are actually mass flow sensors, and have different calibration curves for different media (they come factory-calibrated for H₂O).

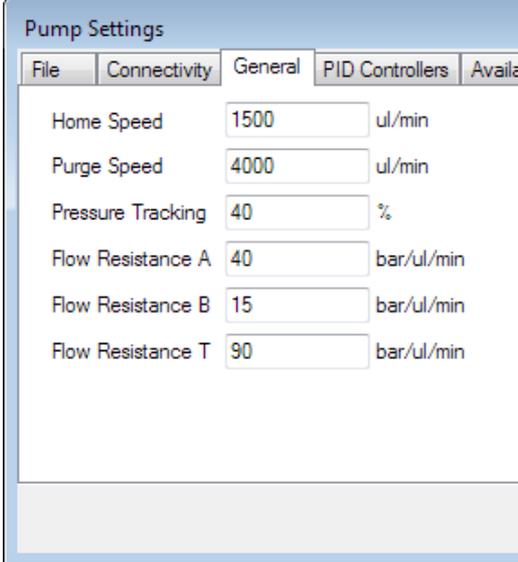
Creating .zrs file (calibrating the flow sensor)



The calibration of each sensor can be selected from a list of default curves for commonly used eluents, or individually set by comparing offset piston volume to measured value of the sensors at different calibration points.

Creating .zrs file (tuning the flow controller)

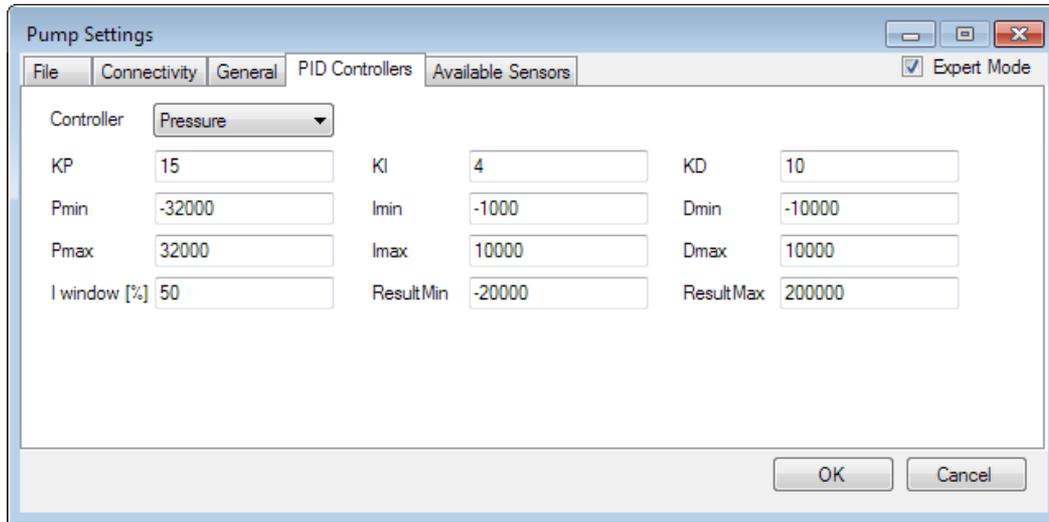
The flow controller is an algorithm which is based on both channels' current flow values and the known properties of the restriction between the sensors and the mixing tee. In order to determine given pressure changes in each iteration, it needs as configuration values the specific flow resistances on each side of the mixing tee. These depend on the installed capillary dimensions as well as on the eluent.



The screenshot shows a software interface titled "Pump Settings". It has a tabbed menu with "File", "Connectivity", "General", "PID Controllers", and "Available". The "General" tab is selected. The settings are as follows:

Parameter	Value	Unit
Home Speed	1500	ul/min
Purge Speed	4000	ul/min
Pressure Tracking	40	%
Flow Resistance A	40	bar/ul/min
Flow Resistance B	15	bar/ul/min
Flow Resistance T	90	bar/ul/min

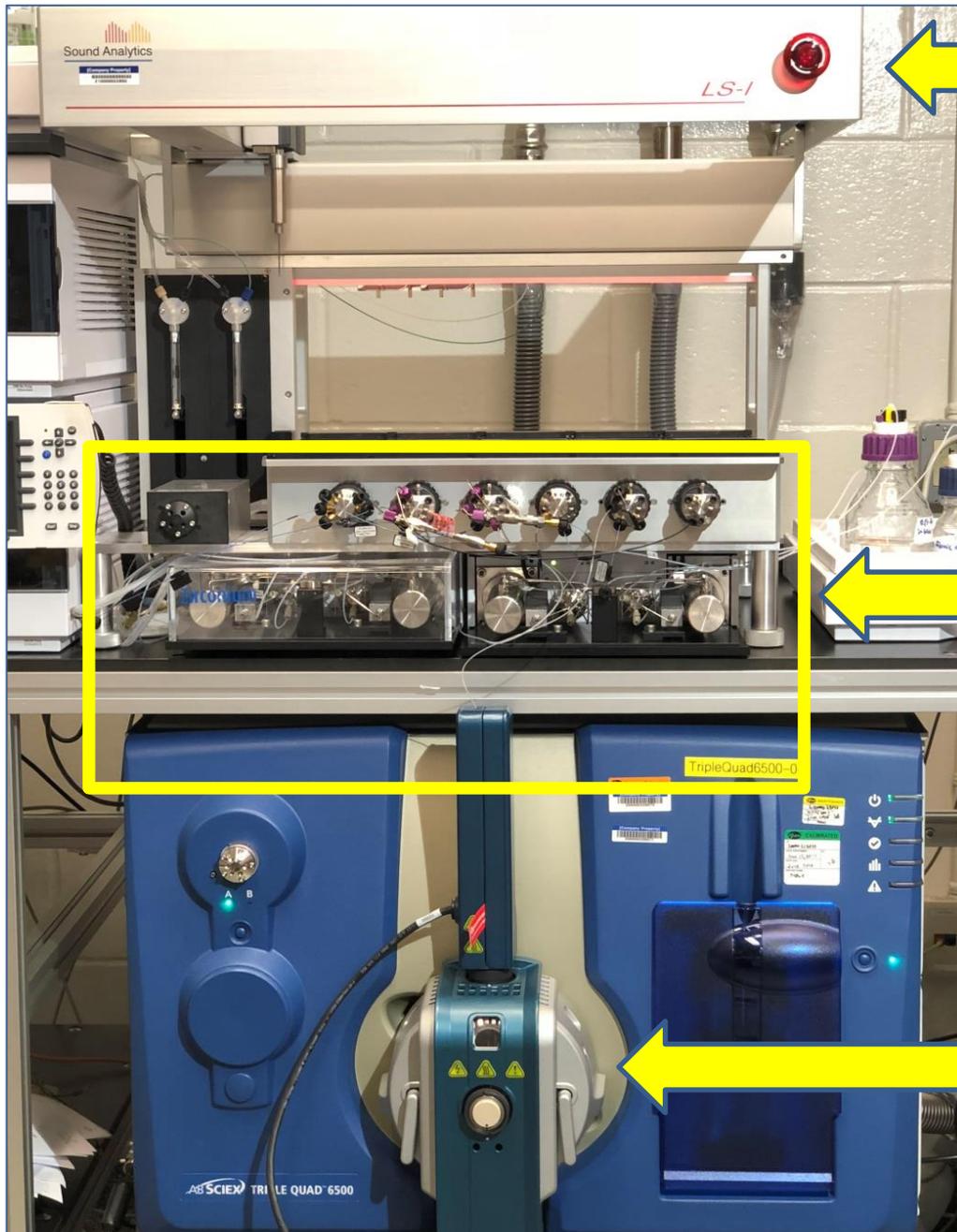
Creating .zrs file (tuning the pressure controller)



The flow of each channel is the result of the exact pressure differences between each pump head and the mixing tee. In order to control each channel's pressure, a separate pressure control algorithm is in control of the piston speed.

This is a standard so called PID controller, the parameters of which have to be empirically determined. Default values and example method settings help users quickly find the right values for their method.

LS-1 Microflow Workstation w/ Dual-Prolab Zirconium

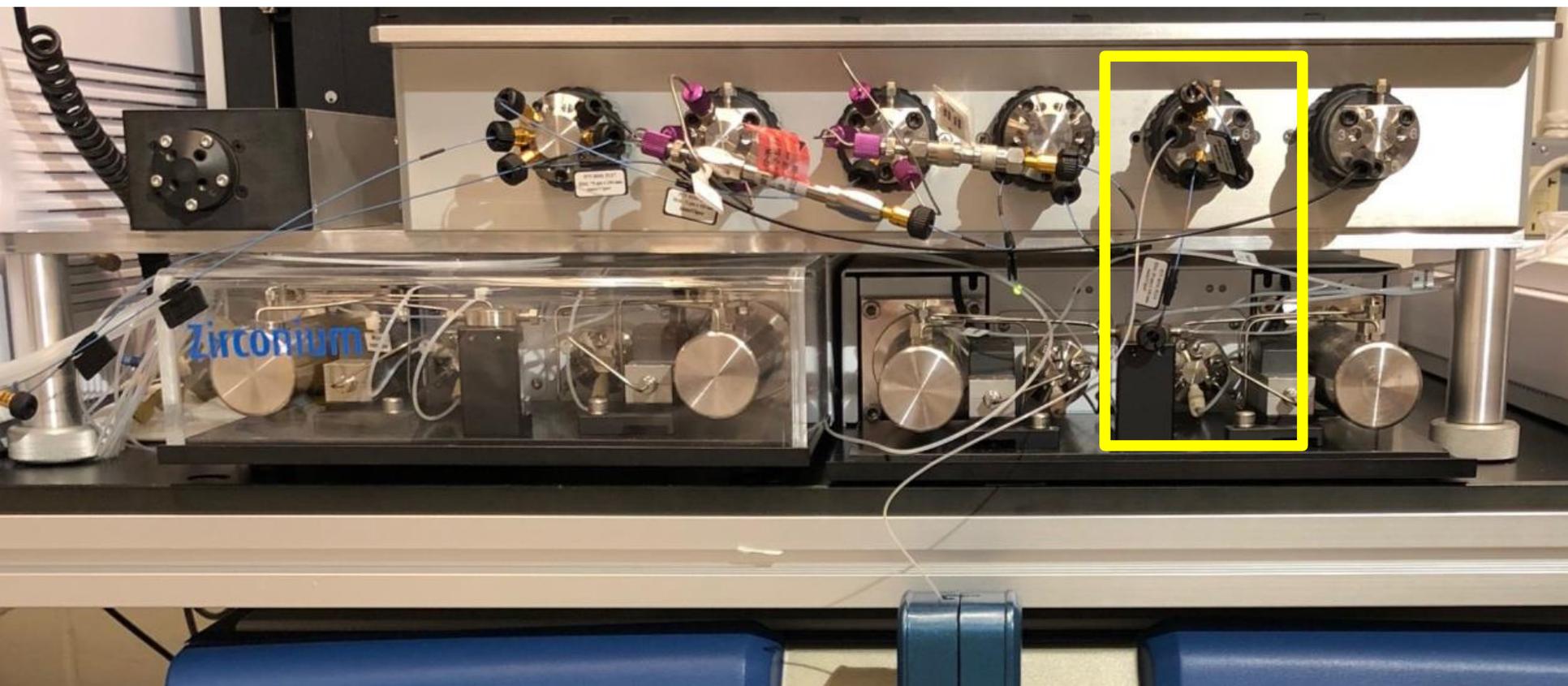


← LS-1 (Sound Analytics)

← ZIRCONIUM™ UHPLC PUMPS (PROLAB)

← OptiFlow source (Sciex)

LS-1/ Zirconium pump/ 6500 : Designed for reduced system volume, leading to exceptional performance and throughput in 1-20 $\mu\text{L}/\text{min}$ flow range



LS-1/ Zirconium: Drive down system volume to increase throughput



System volume

Pre-column

Pump to inj valve: 20 μ x 150mm (0.047 μ L)

Sample loop: 50 μ x 150mm (0.29 μ L)

Inj valve to column: 20 μ x 550mm (0.173 μ L)

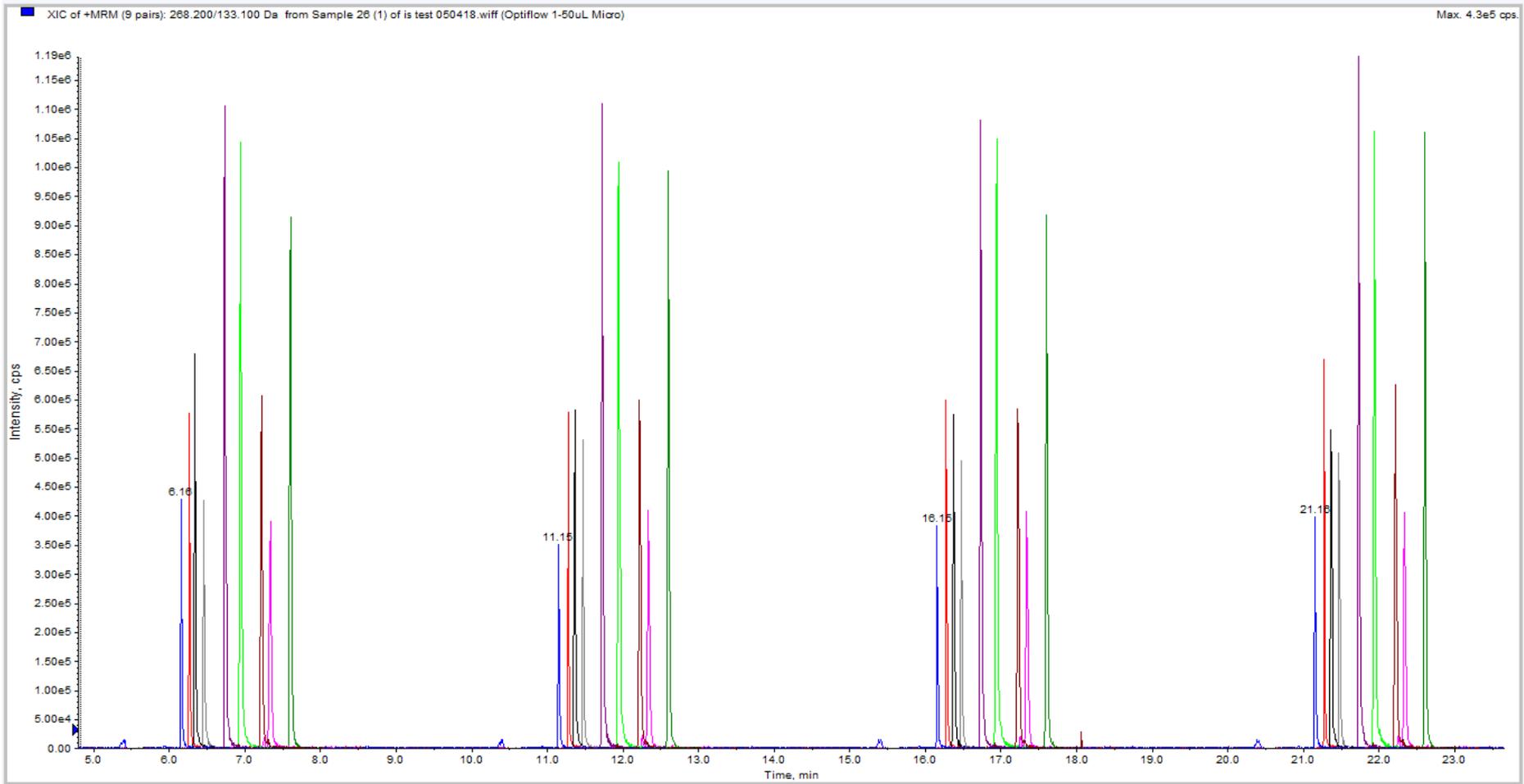
Total Pre-column Volume (including sample loop): 0.51 μ L

Post-column

SCIEX SteadySpray electrode, 25 μ x 200mm (0.1 μ L)

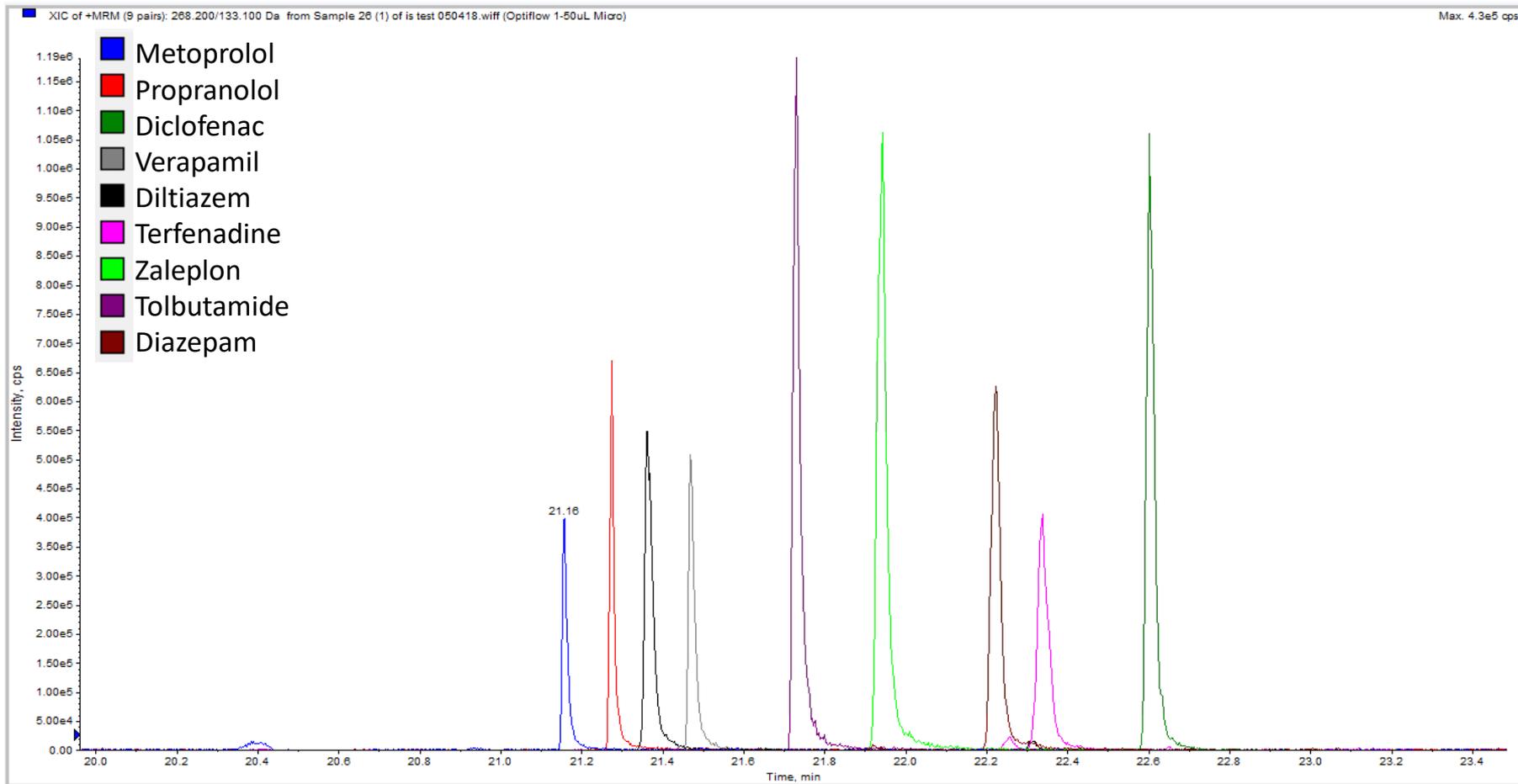
Total system volume: 0.61 μ L

LS-1/ Zirconium/Optiflow: 8 μ L/min, 5min cycle time *



*Courtesy Brendon Kapinos, Pfizer Inc.

LS-1/ Zirconium/Optiflow: 8 μ L/min, 5min cycle time*



*Courtesy Brendon Kapinos, Pfizer Inc.

Luna Omega PS
C18, 3 μ , 50X0.3mm
Phenomenex