



Low Flow, High Gains—Using Microflow LC Techniques for Environmental Applications

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INTRODUCTION

- Liquid chromatography (LC) has been applied to a wide range of environmental samples; combining this with tandem mass spectrometry (MS/MS) allows for highly sensitive and accurate measurements
- Electrospray ionization (ESI) is susceptible to matrix effects, including ion suppression
- Microflow LC has been shown to achieve sensitivity gains and operates at significantly lower flow rates (up to 100x lower) compared to traditional analytical high-performance LC systems. These systems operate at flow rates in the range 1–200 µL/min and the droplets created have a diameter of only a few microns; generating more ions and minimizing ion suppression effects.
- We compare microflow LC and analytical flow LC for the analysis of 69 frequently analyzed pesticides was performed. Both methods use the same SCIEX QTRAP 6500+ LC-MS/MS System. Sensitivity gains of up to 240x were observed for selected pesticides. The sensitivity gains from microflow LC compared to analytical flow LC enabled simpler sample preparation procedures without sacrificing limits of quantitation (LOQs) while also reducing solvent consumption for better sustainability.

ANALYSIS DETAILS

Microflow versus Analytical Flow: Stationary phase kept consistent

Luna Omega 3 µm Polar C18 100 Å 100 mm length

Analytical flow 800 µL/min on a 4.6 mm ID Column

Microflow: 15 µl/min on a 0.5 mm ID column

Mass Spectrometric Analysis:

Compound parameters like collision energy (CE) were kept consistent between both analytical flow and microflow LC analysis.

Case Study :Analysis of pesticides in 13 varieties of beer. Samples were sonicated for 10 minutes to remove carbonation, followed by direct LC-MS/MS injection.

Parameter	High flow	Microflow
Curtain Gas (CUR)	45 psi	20 psi
Ionspray Voltage (IS)	4500 V	4000 V
Heater Temperature (TEM)	300 ° C	200 ° C
Gas 1	60 psi	15 psi
Gas 2	50 psi	60 psi

Table 1: Comparing the source conditions used for analytical flow using the IonDrive Turbo V™ Ion Source vs. microflow using the Optiflow Turbo V™ Ion Source for pesticide analysis

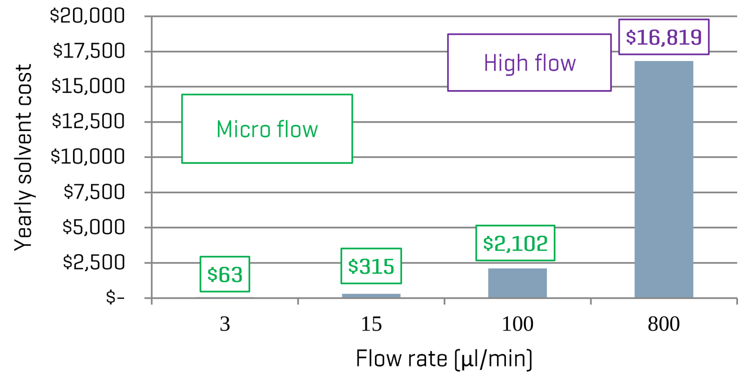

Figure 1: Yearly solvent cost of micro flow versus high flow assuming 8 min run times and $240 USD for 4L bottle of solvent

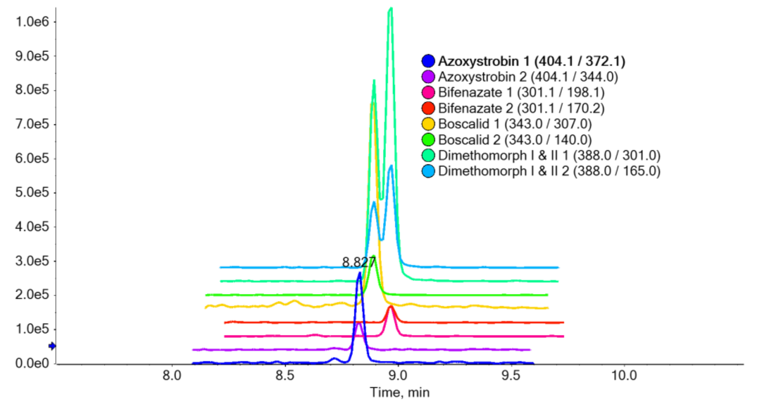

Figure 2: XIC of selected pesticides observed in beer

USING MICROFLOW LC COUPLED TO MS ALLOWS FOR SENSITIVITY GAINS UP TO 240X WHILE REDUCING SOLVENT CONSUMPTION

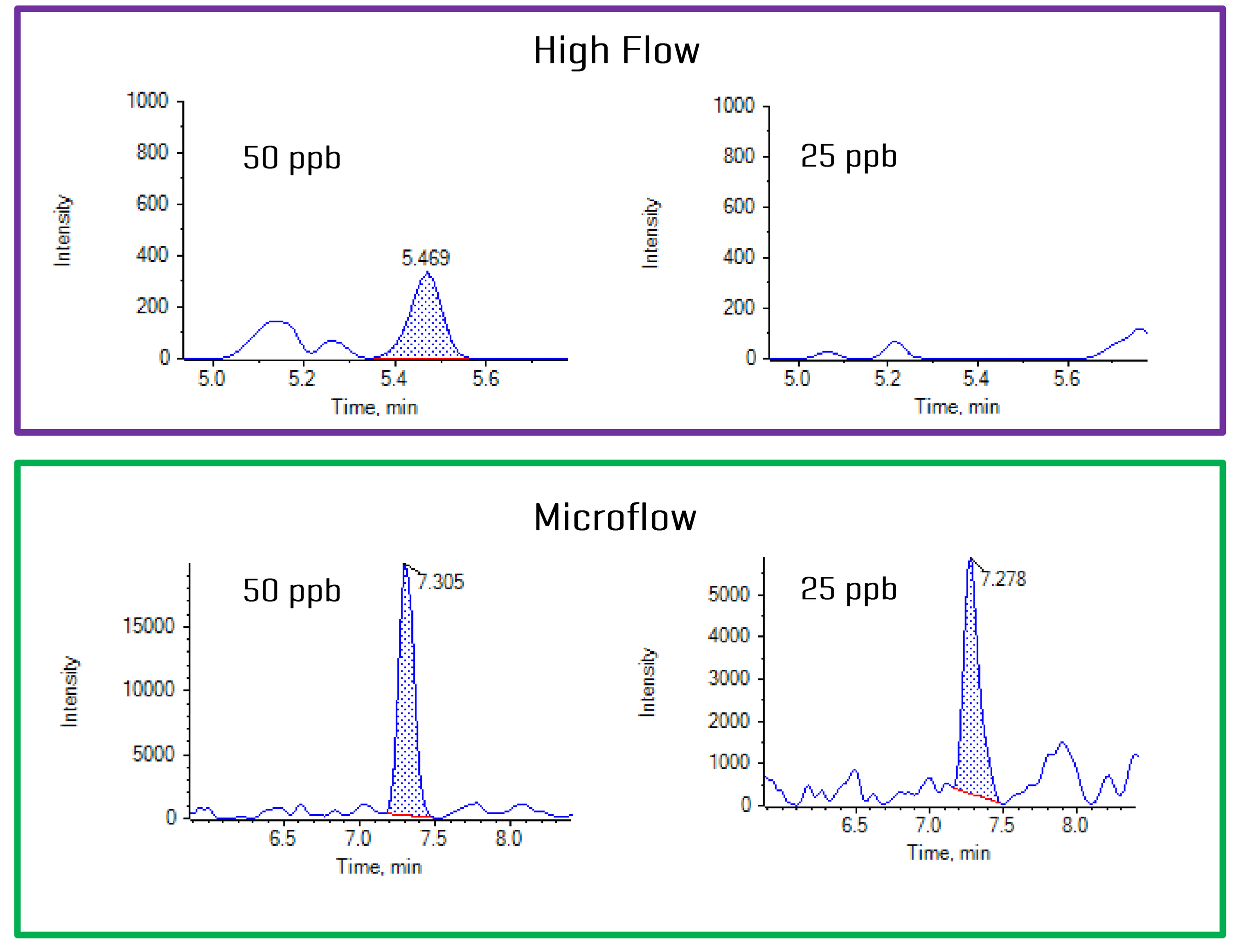

Figure 3: Comparison of captan at 25 and 50 ppb for analytical versus microflow

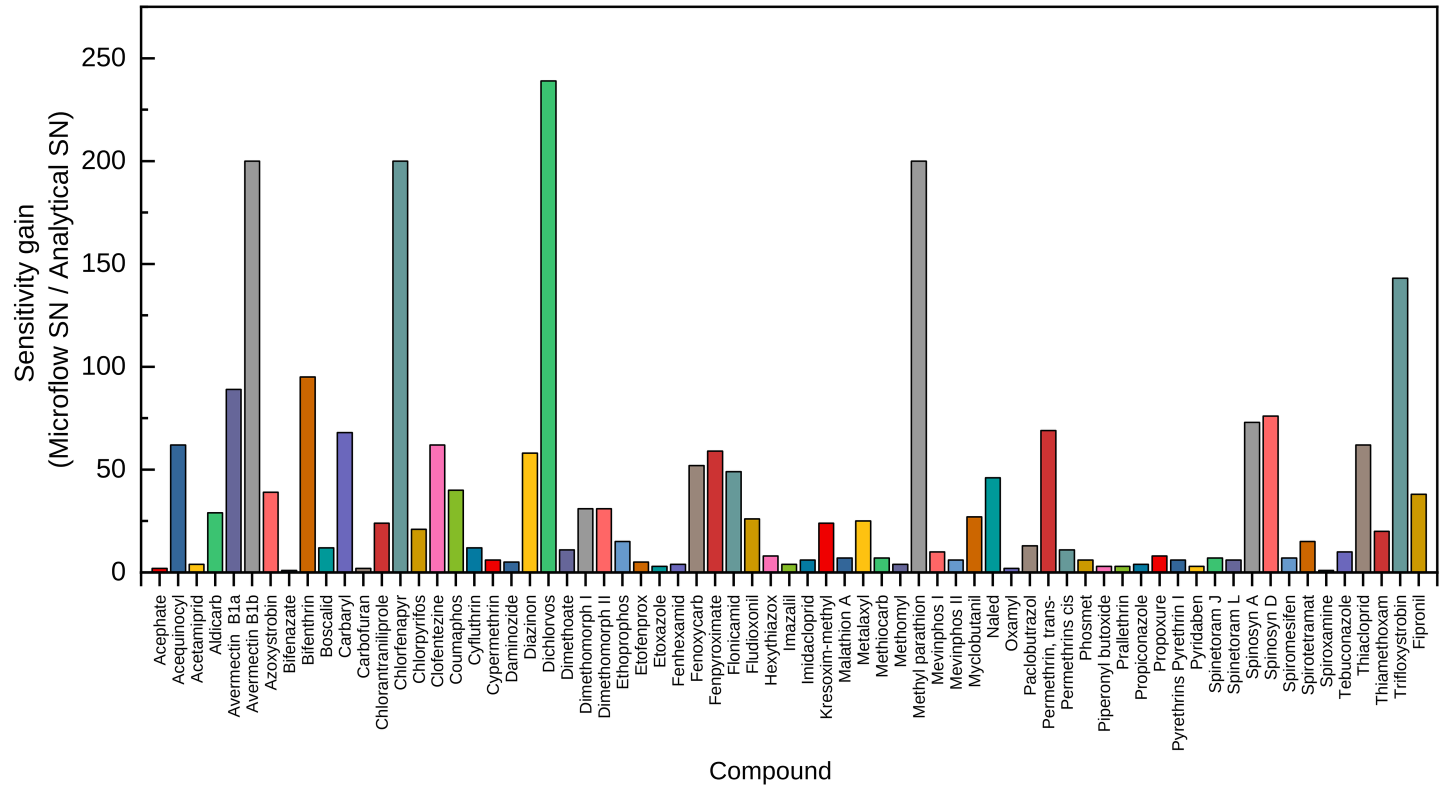

Figure 4: Sensitivity gains from microflow versus analytical flow for over 60 pesticides. Over 50% of the compounds have a gain of 10x or more.

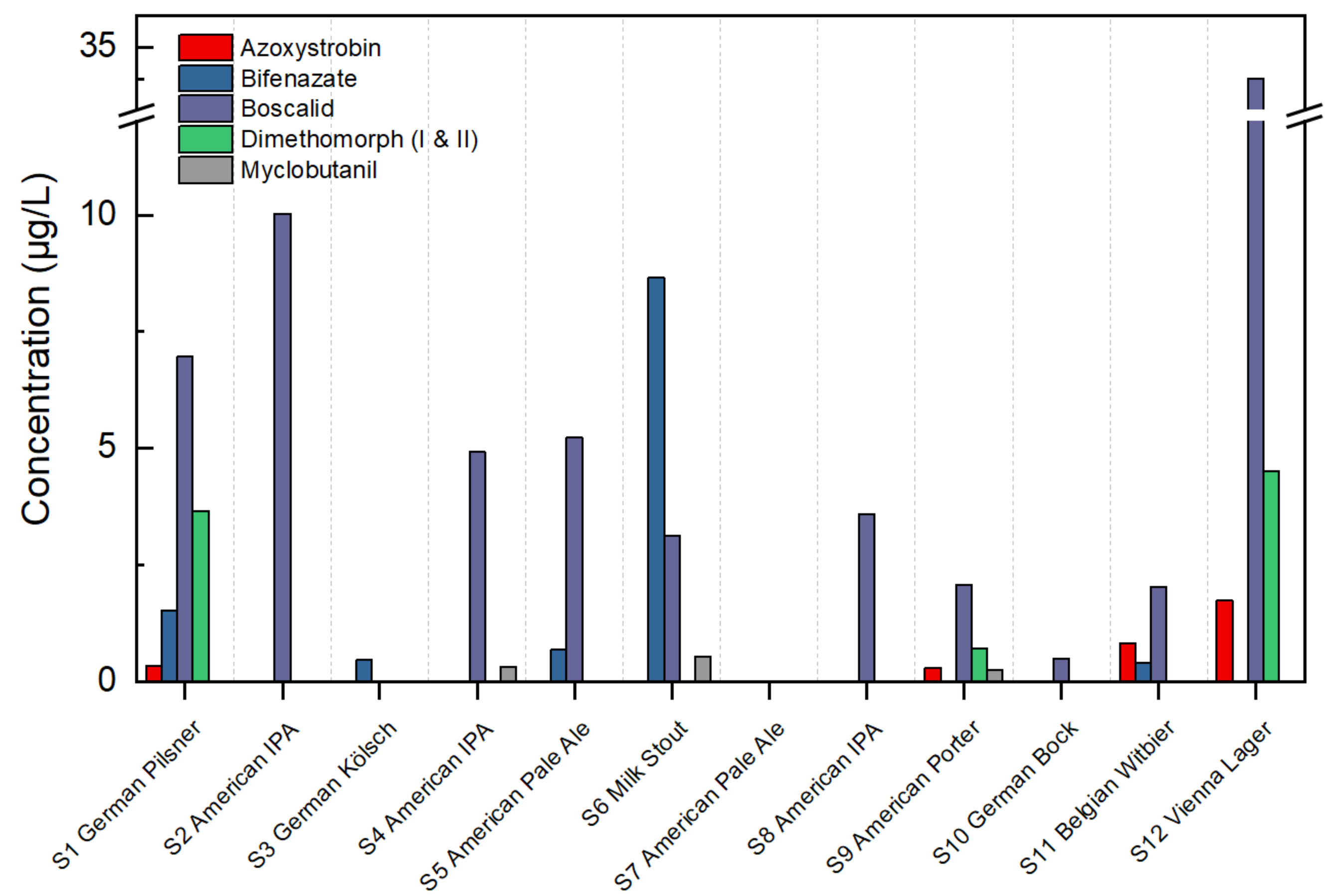

Figure 5: Of the targeted 69-panel of pesticides we detected five pesticides - azoxystrobin, bifenazate, boscalid, dimethomorph and myclobutanil. Research has shown that these pesticides can survive the brewing process due to their thermal stability and low log P values

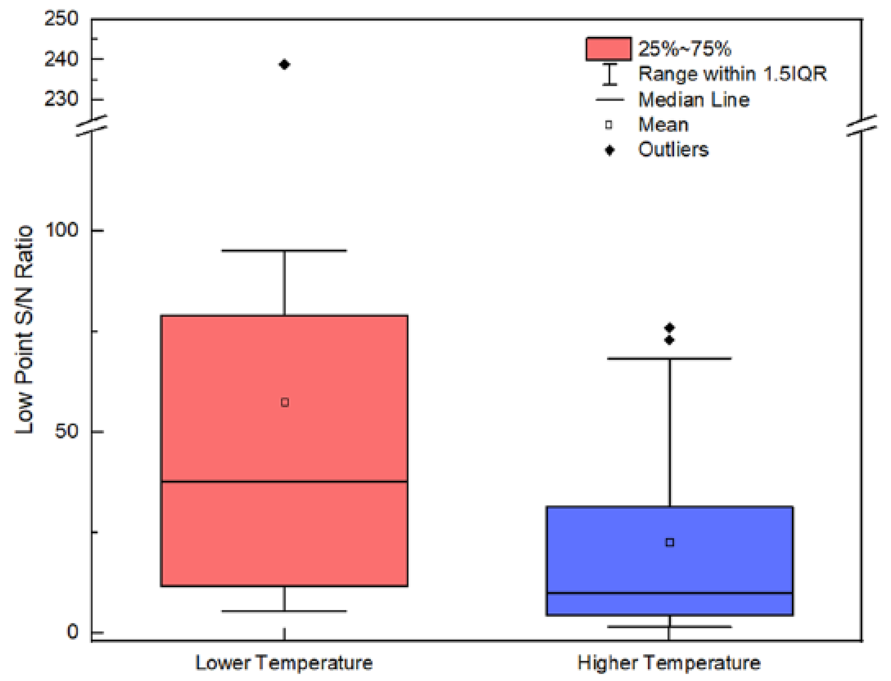

Figure 6: **Impact of source temperature on pesticide signals.** Signal to noise (S/N) ratios at the low concentration point were compared between microflow and analytical flow for the two observed classes of compounds; those that saw an increase in intensity with lower source temperatures (red) and compounds that saw an increase with higher source temperatures (blue). Compounds that preferred a lower temperature had a median S/N ratio value that was ~4x higher than the compounds that preferred higher source temperatures.

Figure 7: SCIEX M5 Microflow LC system, SCIEX Exion AE LC system and SCIEX QTRAP 6500+ system

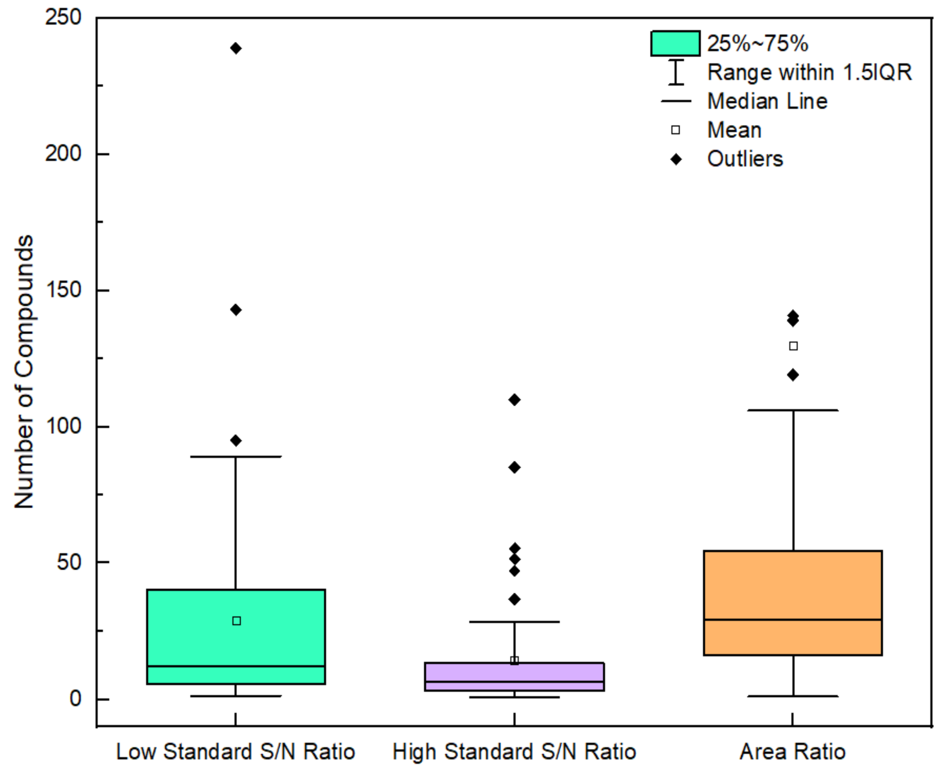

Figure 8: The sensitivity between microflow LC and traditional flow was compared by dividing the signal to noise (S/N) for the compound using the MFLC method by the signal to noise of the compound using the traditional flow method
This ratio was measured at two points; (1) the lowest point of the calibration curve in the traditional flow data, and (2) the highest point of the MFLC calibration curve

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