

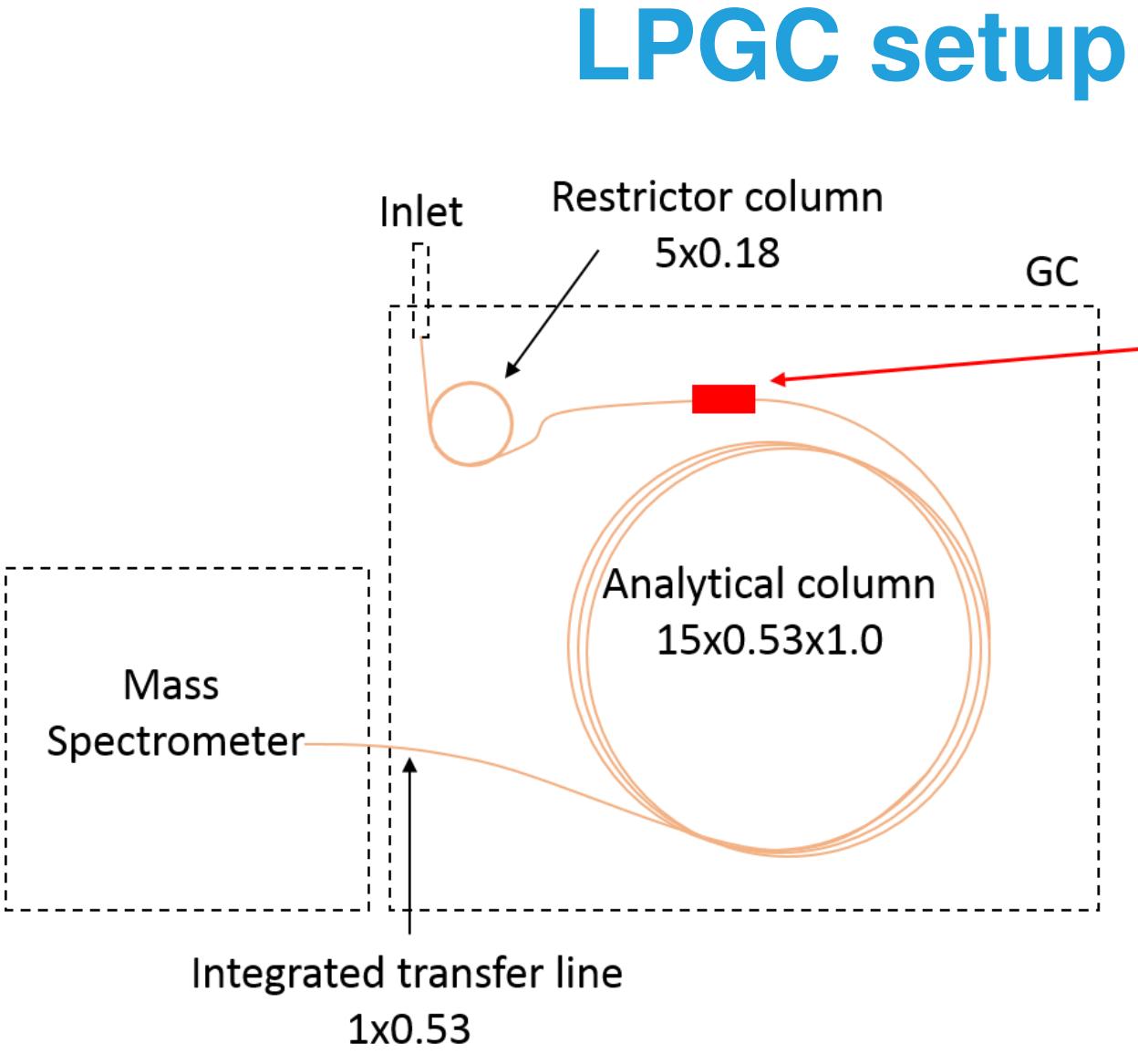
Large Volume Injection of Pesticides Using Low Pressure Gas Chromatography

Jana Hepner, Jaap deZeeuw, Joe Konschnik, Hansjörg Majer

Restek Corporation, Bellefonte, PA, USA

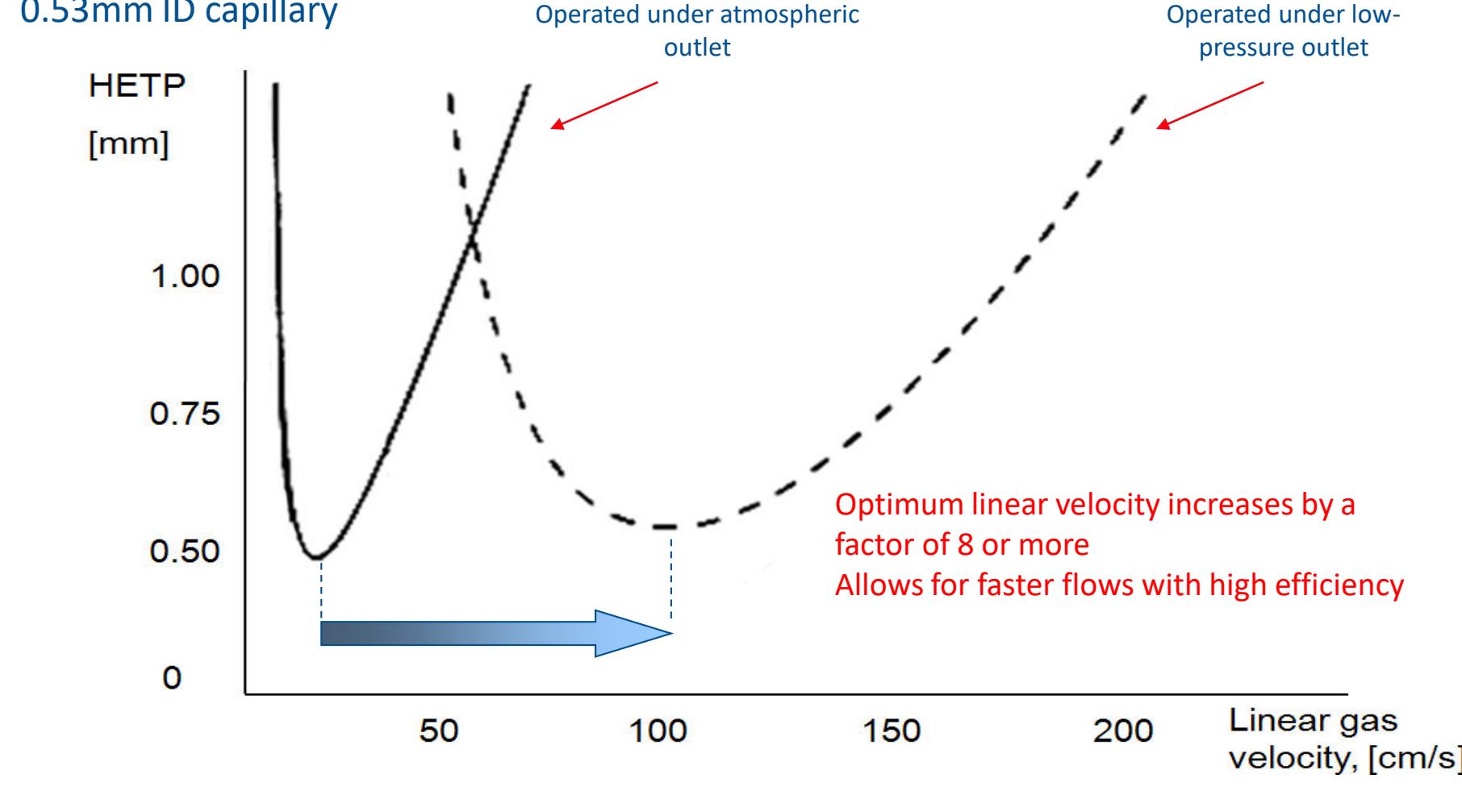
Introduction

Concurrent Solvent Recondensation Large Sample Volume splitless injection (CSR-LVSI, or LVI) is a sample technique that overcomes the limitation of the maximum injection volume to 1 – 2 μ L valid for classical splitless injection. Low Pressure Gas Chromatography is a novel technique that had been successfully used for pesticide screening and quantification. The LPGC configuration with the restrictor/guard column lends itself to the requirements of the CSR-LVSI and has a potential to improve the sensitivity and lower detection limits. Large volume injection of acetonitrile and acetonitrile – toluene samples were evaluated in range of 1 – 25 μ L for peak shapes and the relationship between the peak area and injection volume was established.



Column set is delivered pre-connected in the box
Only extra consumable needed is 0.8 mm vespel/graphite ferrule for MS transfer line

LPGC and Optimum Linear Velocity



Advantages of LPGC

Fast analysis with short 0.53 mm capillaries

- Short analysis times
- Increased sensitivity
- Higher capacity

Peak width enough for any type of MS

Lower elution temperatures

- Elution at 50-80°C lower temperatures
- Lower bleed (compared to other thick film columns)

Standard injection techniques, high volume injections

Overcoming Limitations of LPGC

Loss of theoretical plates (compared to conventional column)

- Can be mitigated by selective MS detection

Higher bleed from thicker film

- Lower elution temperatures

Greater potential for leaks

- Pre-connected, leak-free tested column set

More complicated to cut analytical column

- Less need to cut column

Need for MS instrument under vacuum

Rapid oven heating needed for optimal performance

- Either 220 V instrument or accelerator oven insert

- GC oven set to temperature below the boiling point of the solvent
- Fast injection with liquid band formation
- Liquid sample is deposited on glass wool
- Pressure surge from evaporating solvent “pumps” sample into retention gap
- Most of solvent goes into retention gap

Large Volume Injections

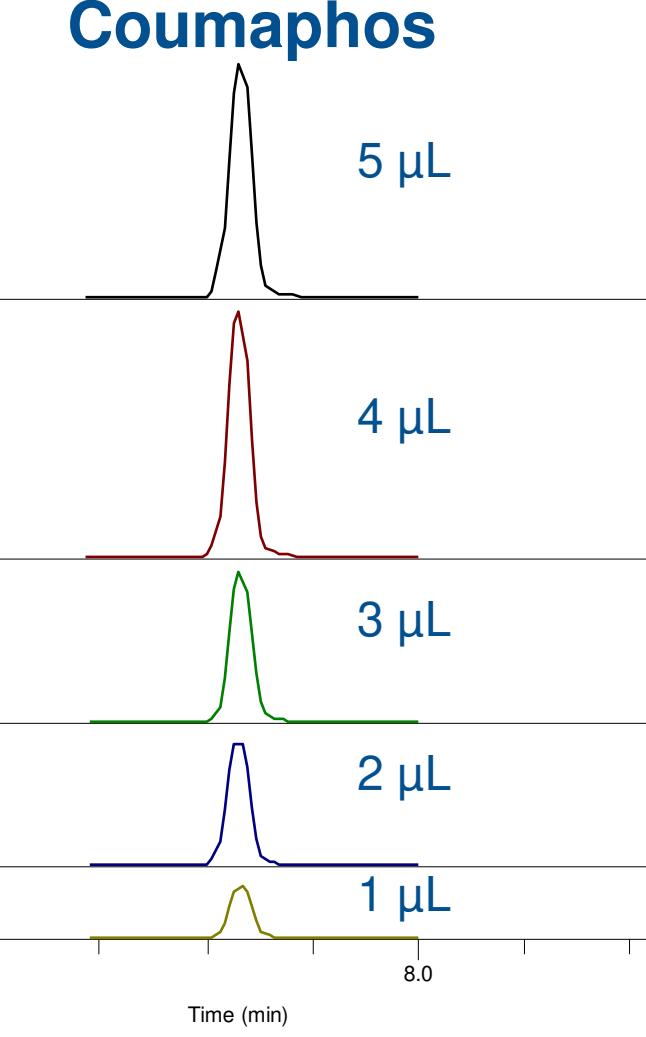
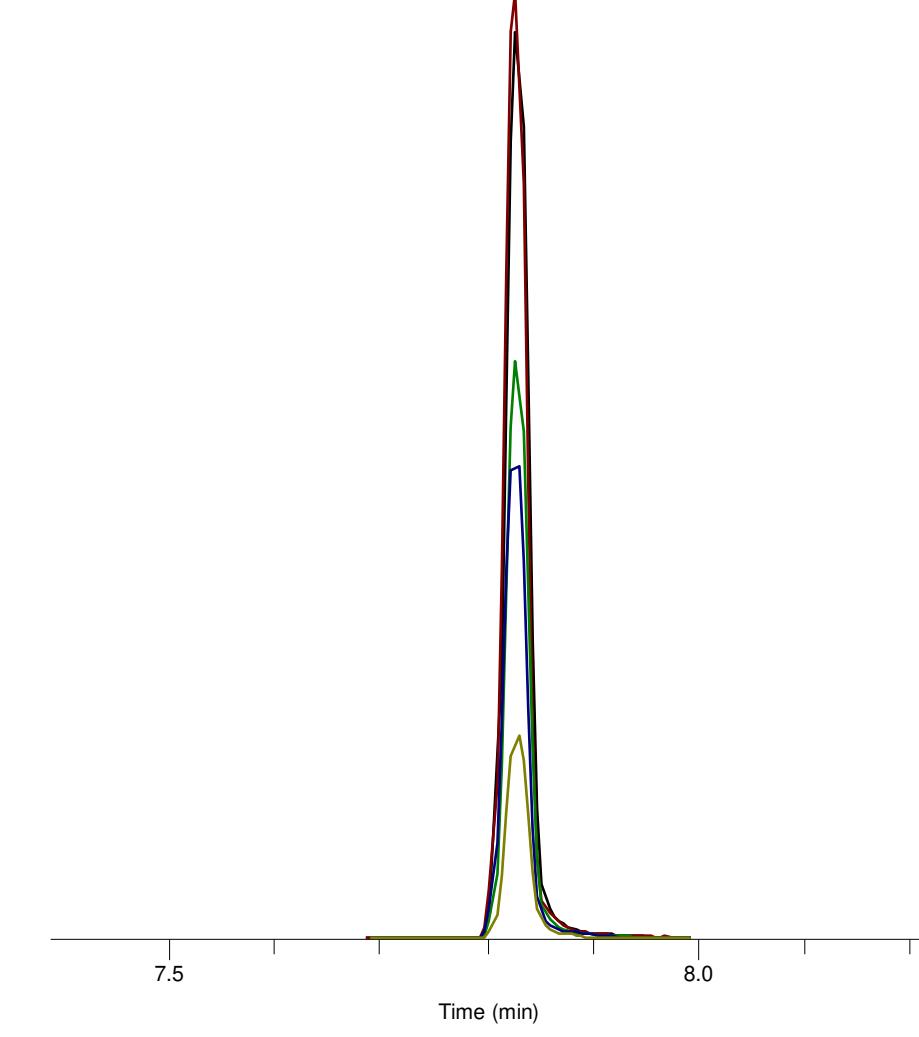
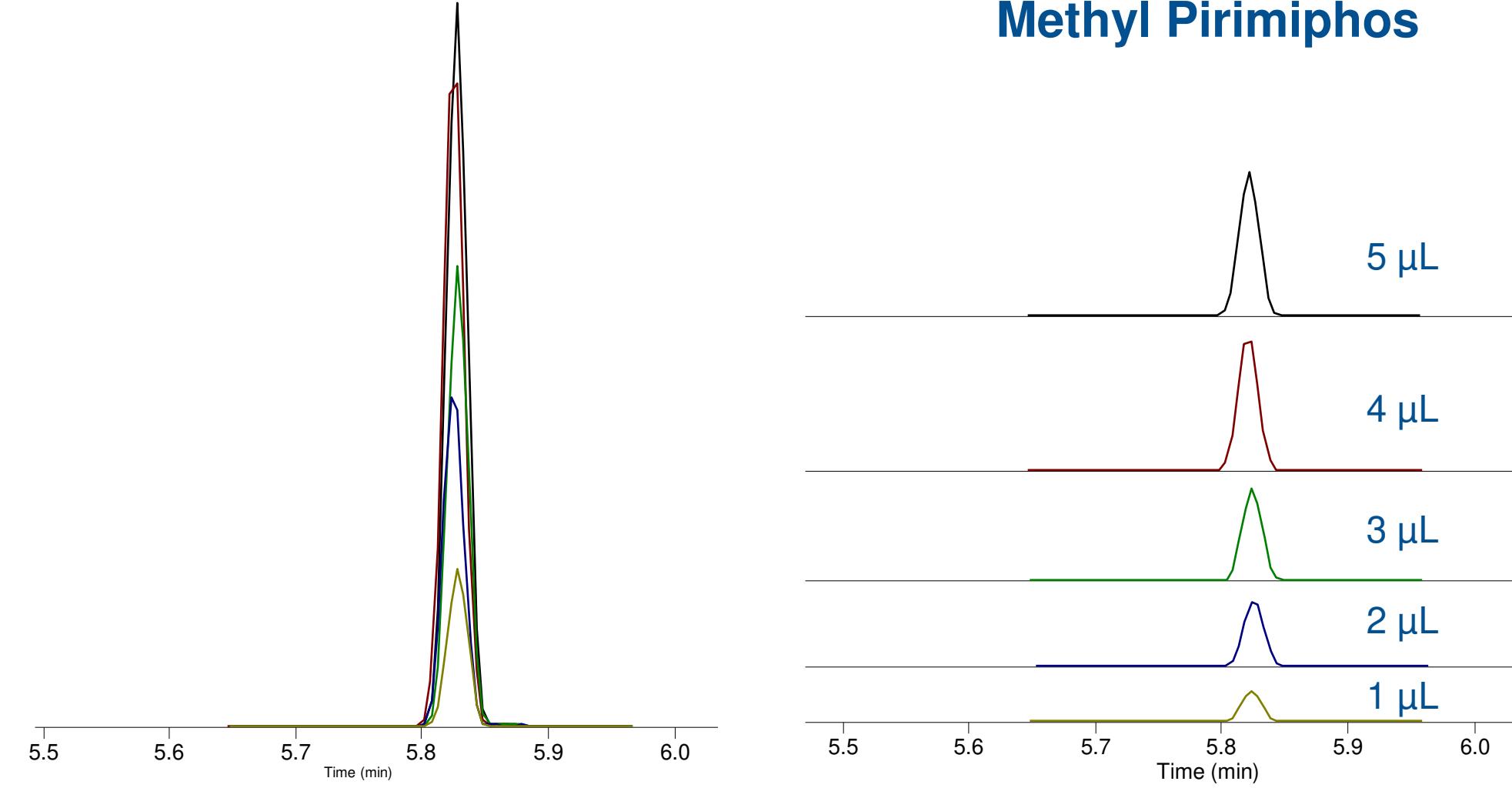
- Lower detection limits
- Saves time in sample prep
- Eliminate need for expensive PTV

Large volume splitless injection needs:

- A splitless injection device
- A liner with glass wool
- A retention gap that has to be coupled

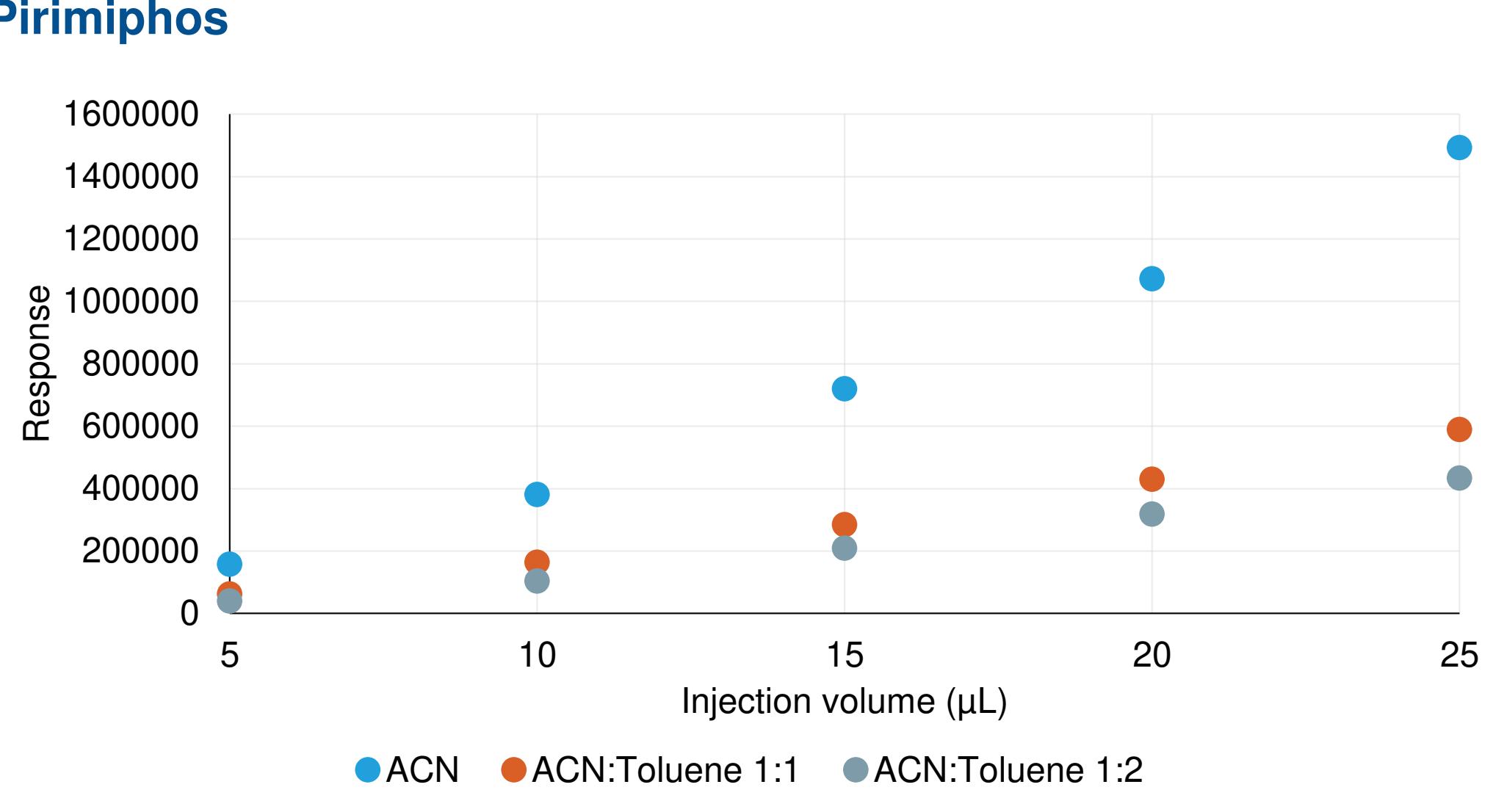
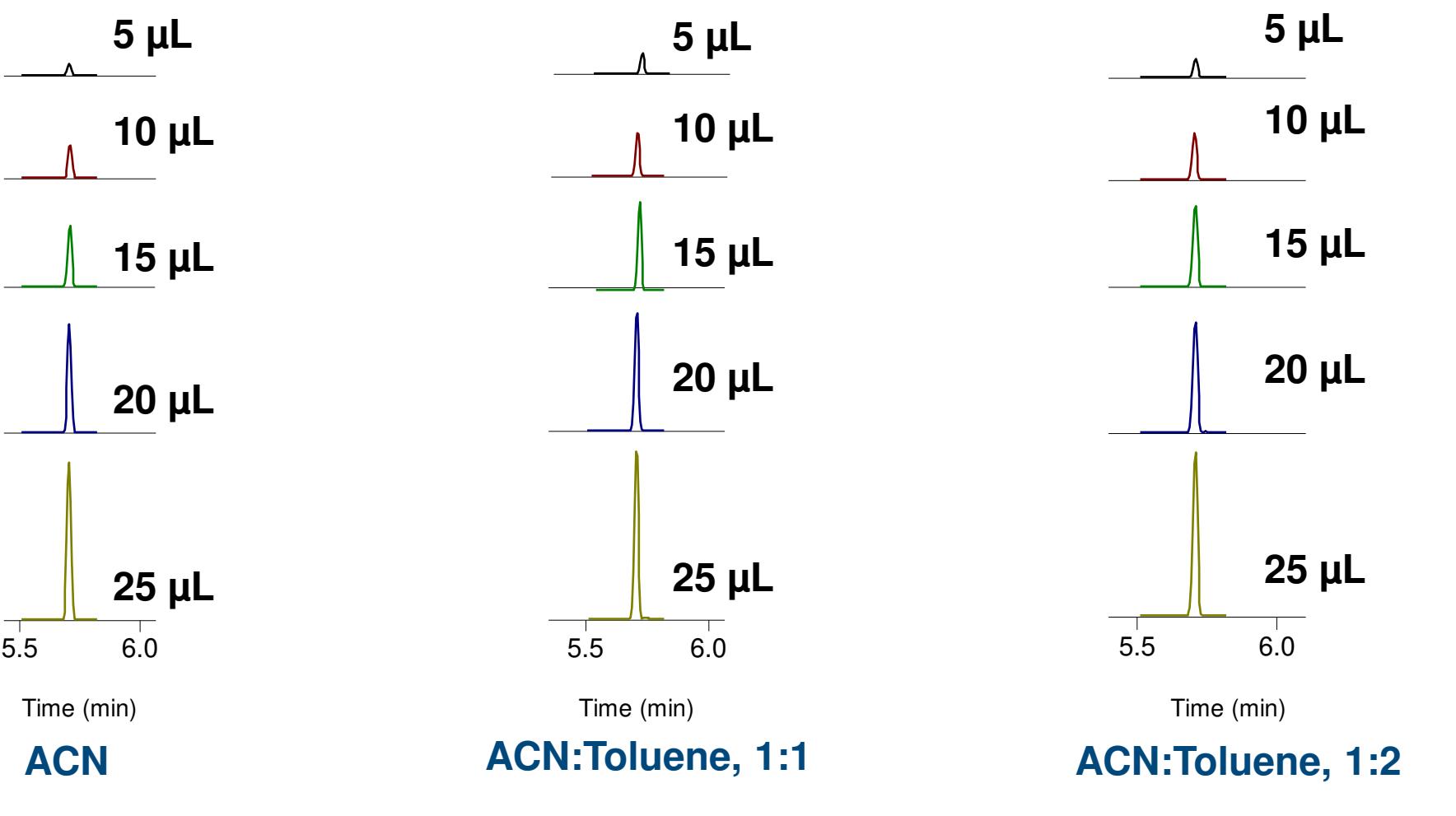
Comparison of 1-5 μ L Injection in ACN

Methyl Pirimiphos

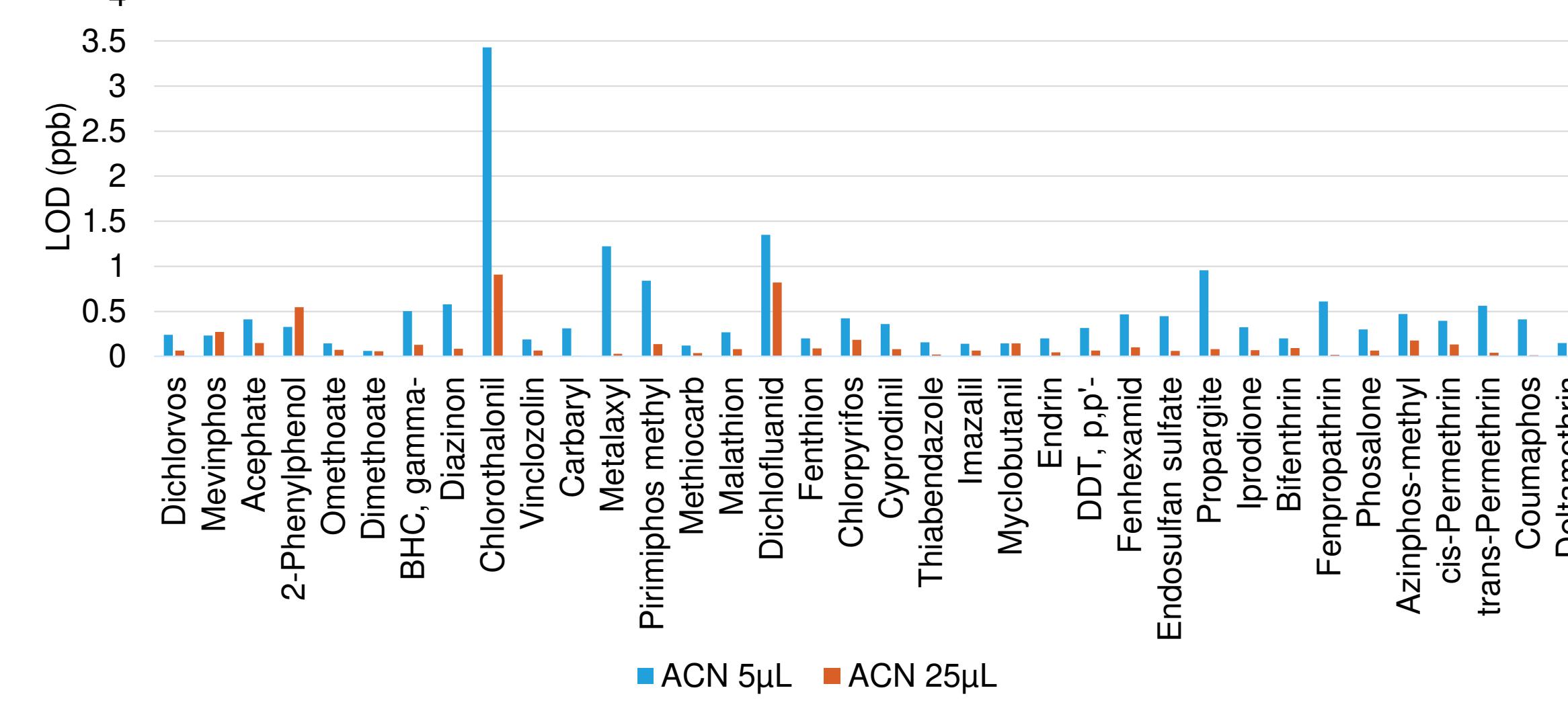
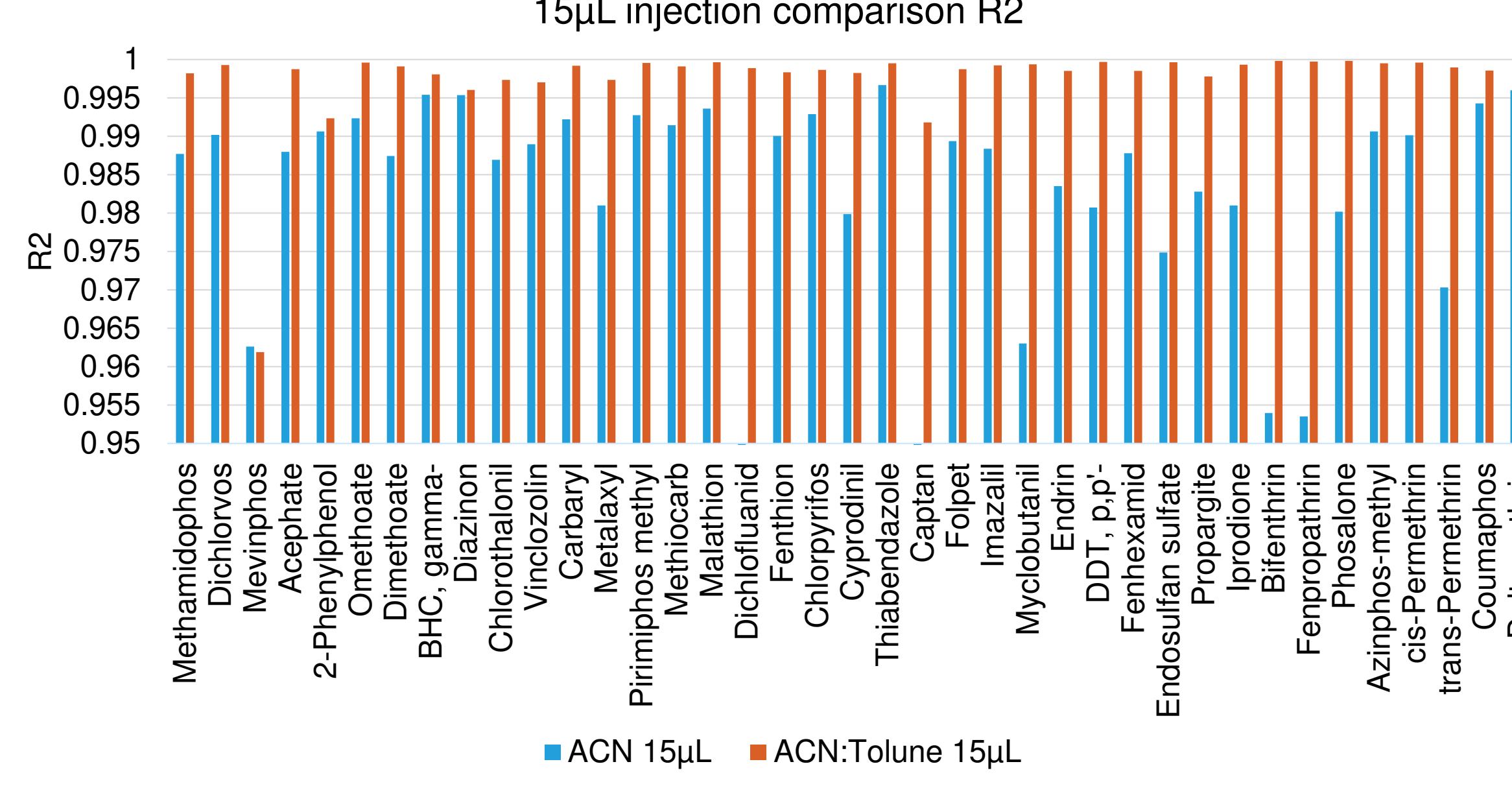


Comparison of 5-25 μ L Injection in ACN and ACN:Toluene

Methyl Pirimiphos

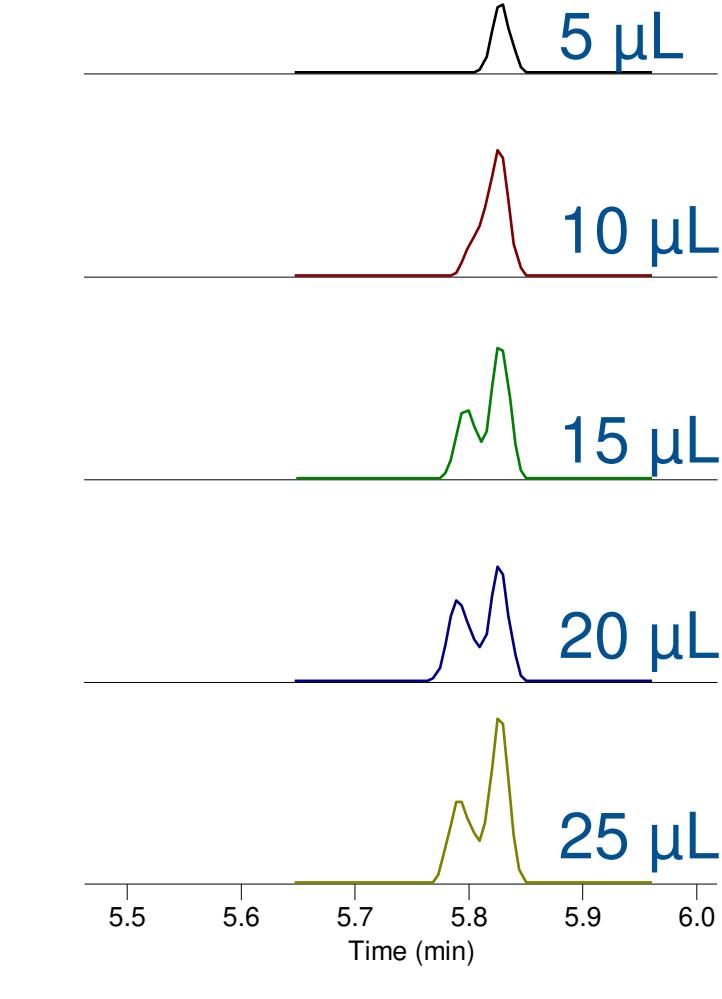


Calibration Comparison



Peak Splitting

- Adjust injection speed
- Change liner/check the wool
- Re-install the column
- Dilute with a more non-polar solvent
- Use a gas-tight syringe



Conclusions

- LPGC is a great tool for fast and robust analysis
 - Generally 3-4x faster analysis
- Large volume splitless injection is a convenient way to introduce more than 1-2 μ L sample
 - Without upgrading your instrument
- Combination of these two techniques can improve your pesticide analysis!
 - Higher sensitivity
 - Potentially easier sample prep
 - Great peak shapes at high volumes
 - Addition of toluene improves the calibration performance