

# Large Volume Injection of Pesticides Using Low Pressure Gas Chromatography

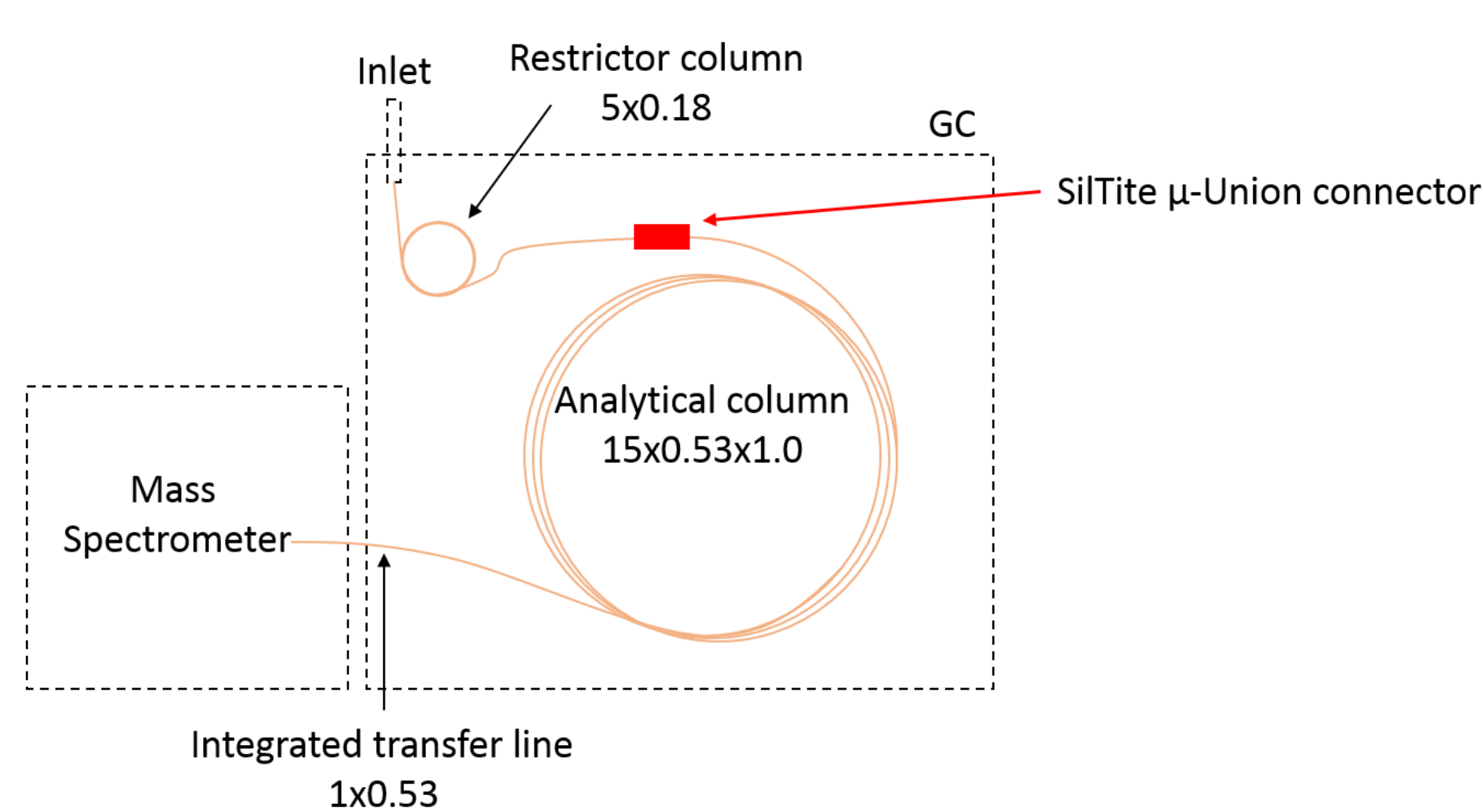
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## Introduction

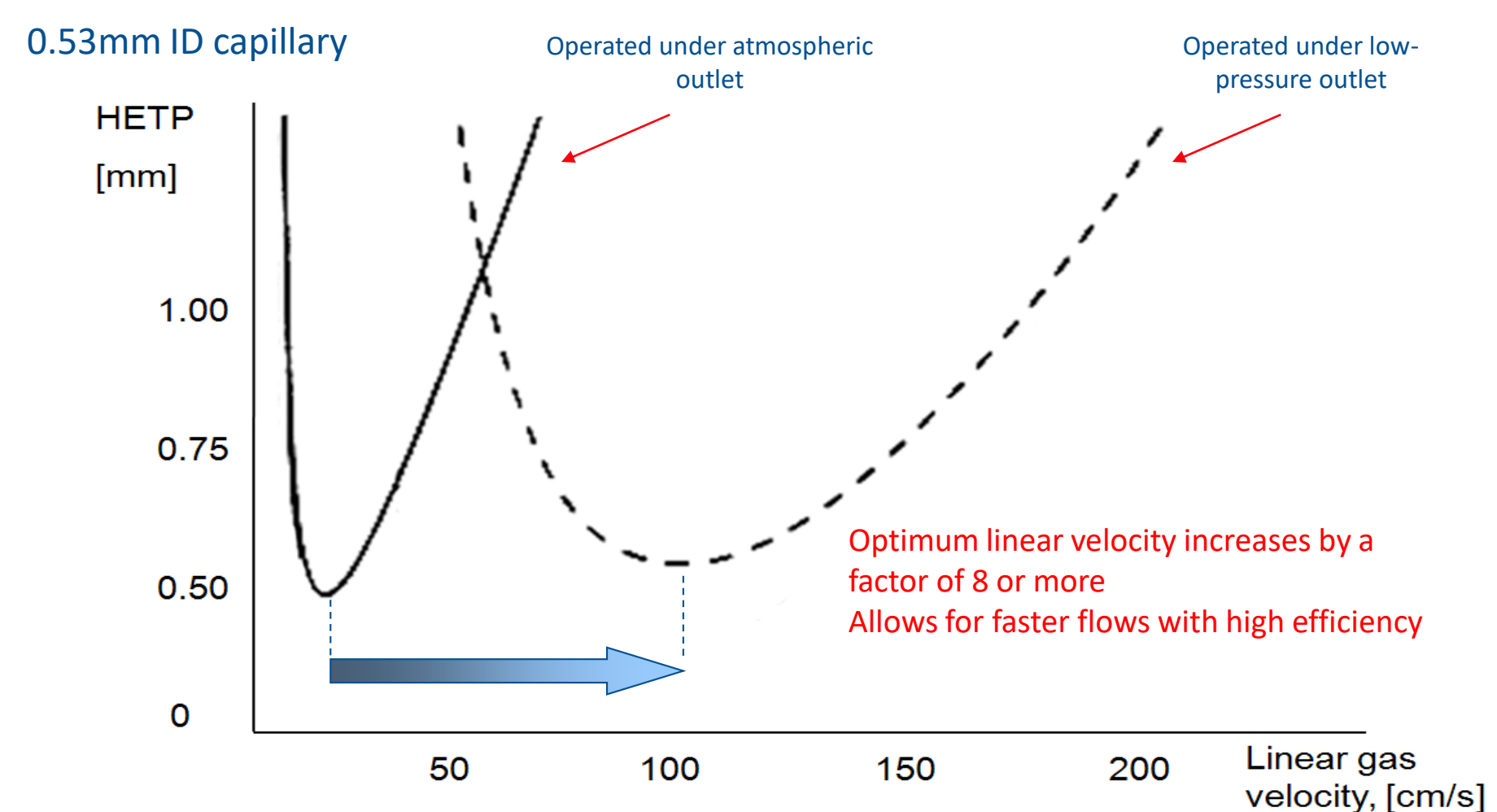
Concurrent Solvent Recondensation Large Sample Volume splitless injection (CSR-LVSI, or LVI) is a sample technique that overcomes the limitation of the maximum injection volume to 1 – 2  $\mu\text{L}$  valid for classical splitless injection. Low Pressure Gas Chromatography is a novel technique that 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  $\mu\text{L}$  for peak shapes and the relationship between the peak area and injection volume was established.

## LPGC setup



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

## Large Volume Injections

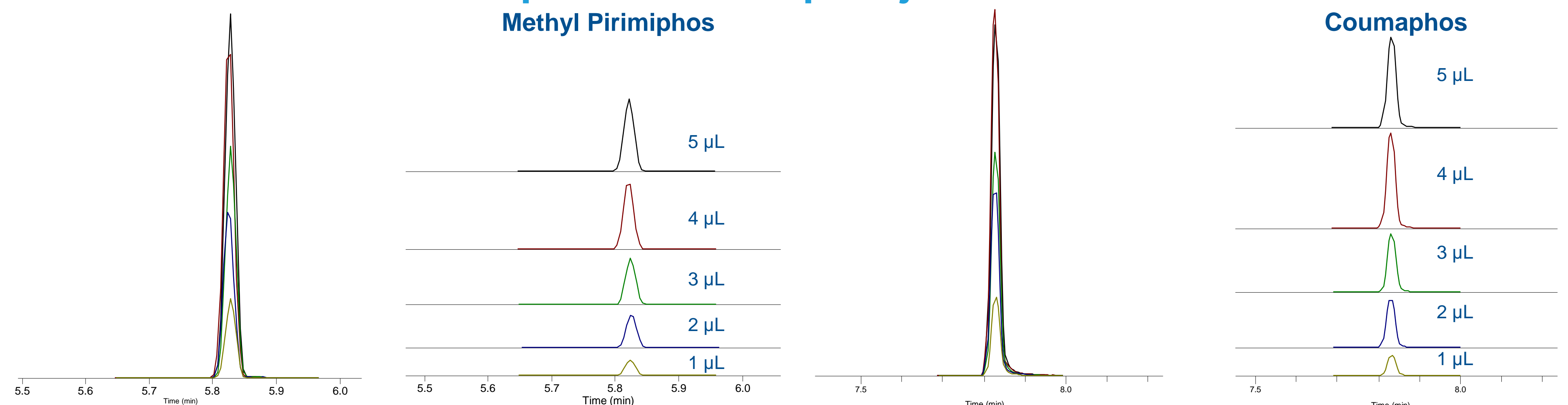
- GC oven set to temperature below the boiling point of the solvent
- Fast injection with liquid band formation
- Liquid sample is deposited on glass wool
- Pressure surge from evaporating solvent “pumps” sample into retention gap
- Most of solvent goes into retention gap

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

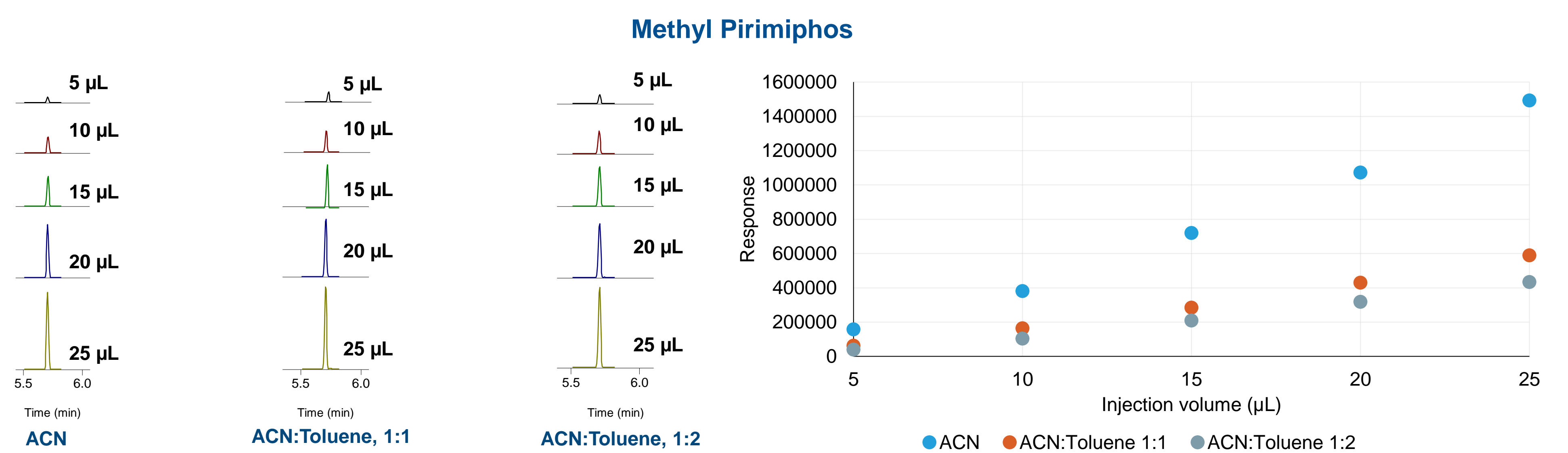
### Large volume splitless injection needs:

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

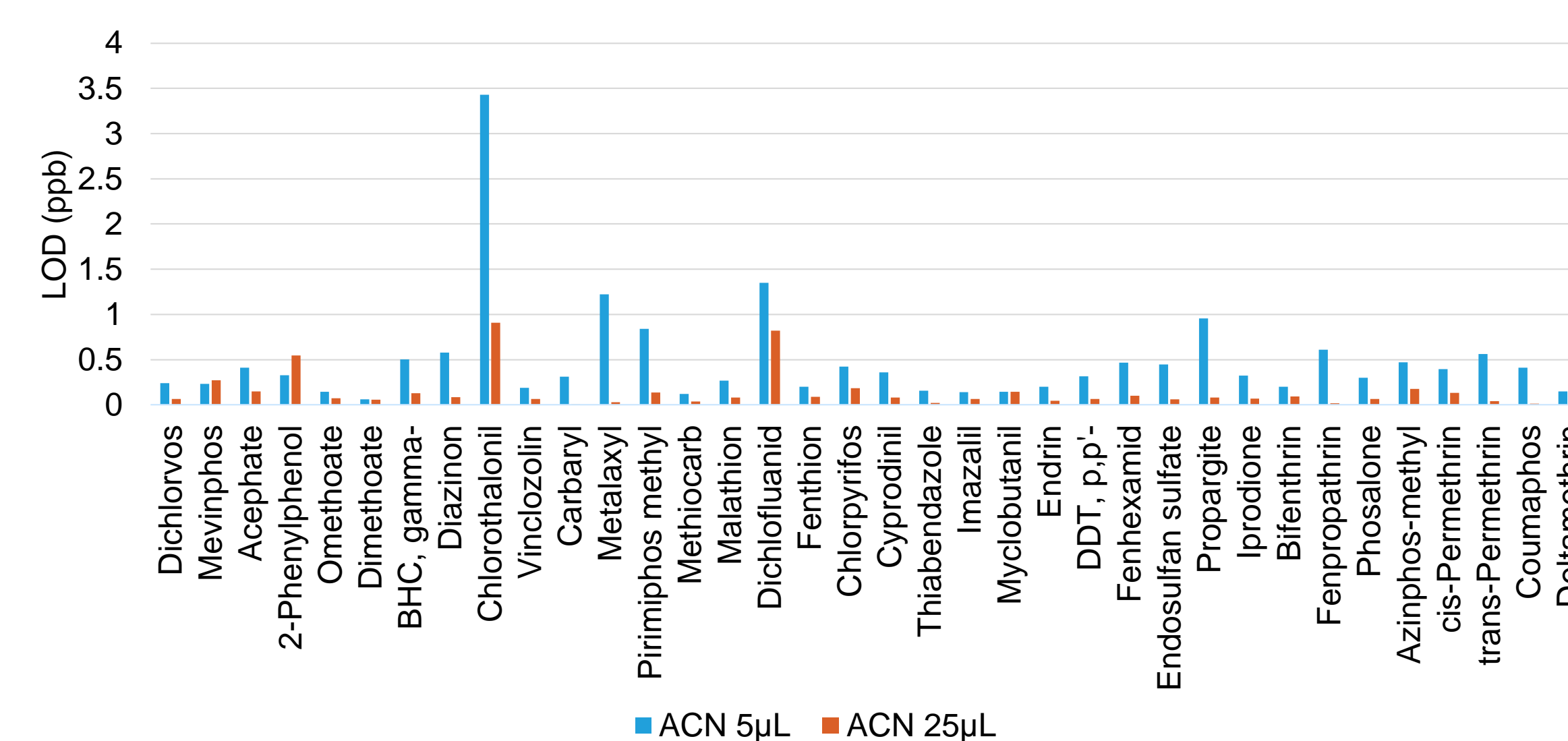
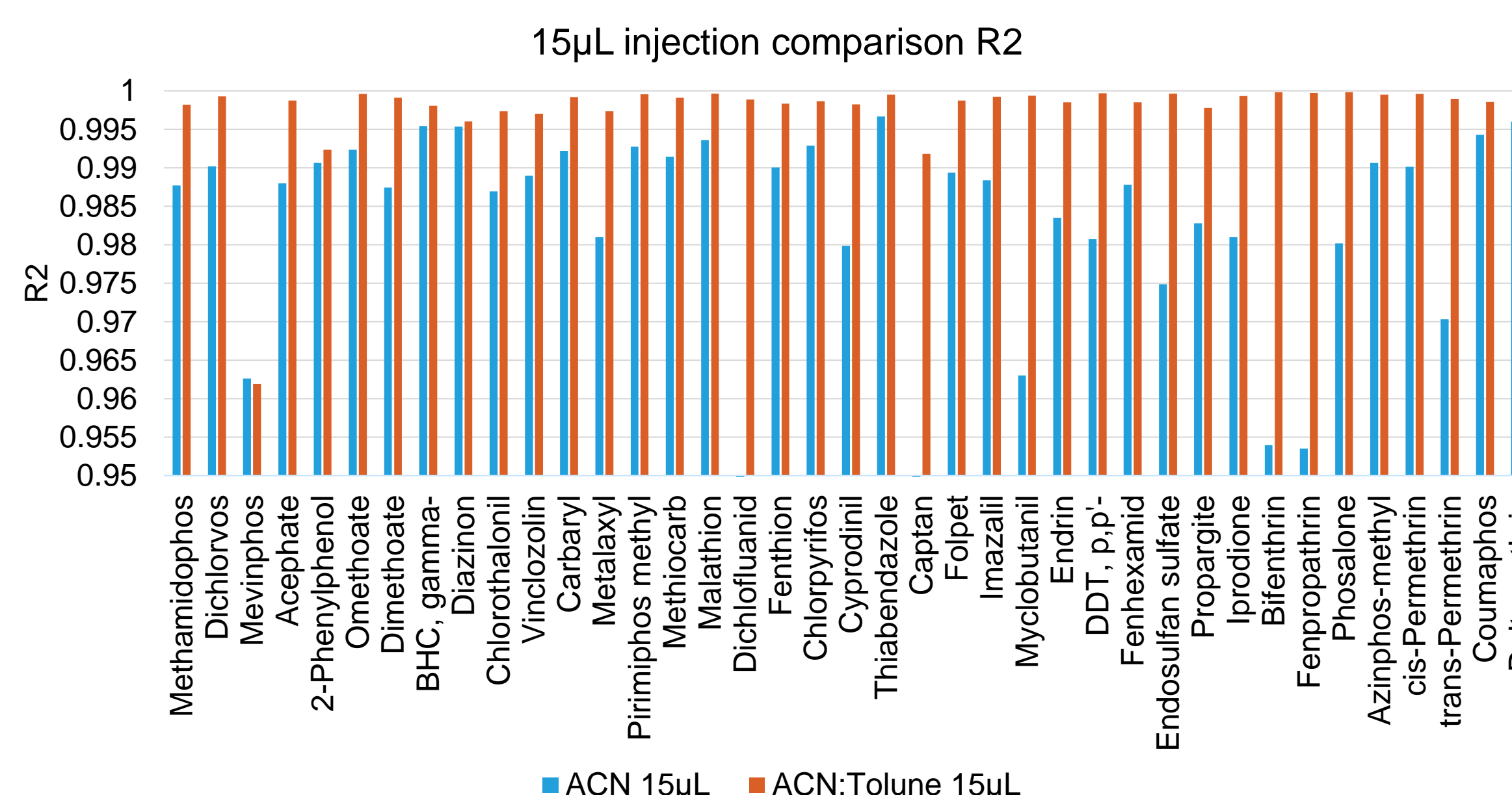
## Comparison of 1-5 $\mu\text{L}$ Injection in ACN



## Comparison of 5-25 $\mu\text{L}$ Injection in ACN and ACN:Toluene

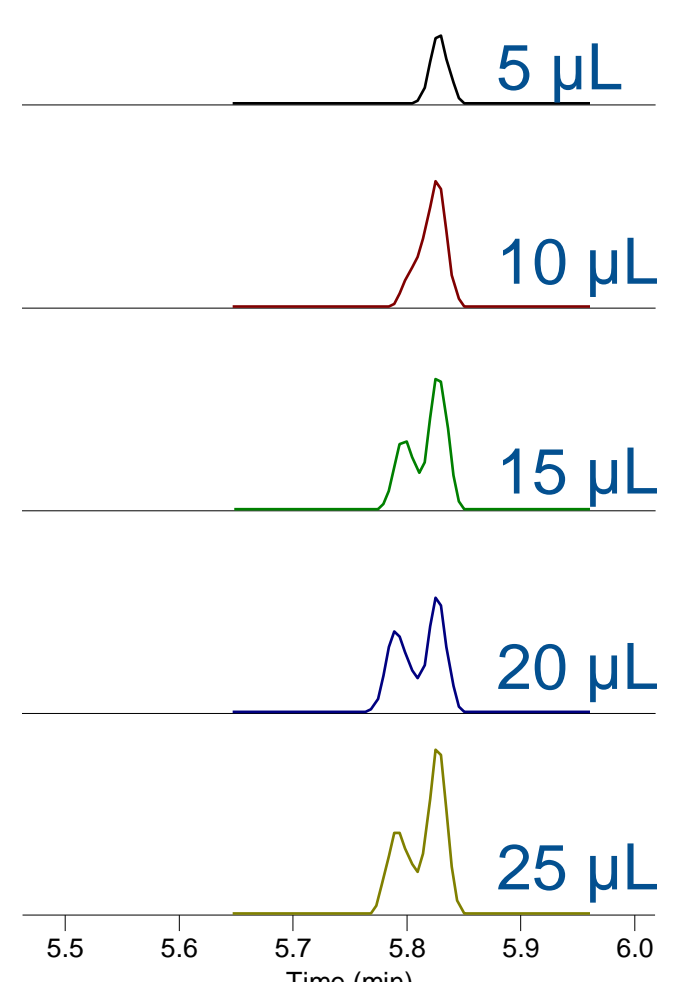


## Calibration Comparison



## 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  $\mu\text{L}$  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