Large Volume Injection of Pesticides Using Low Pressure Gas Chromatography

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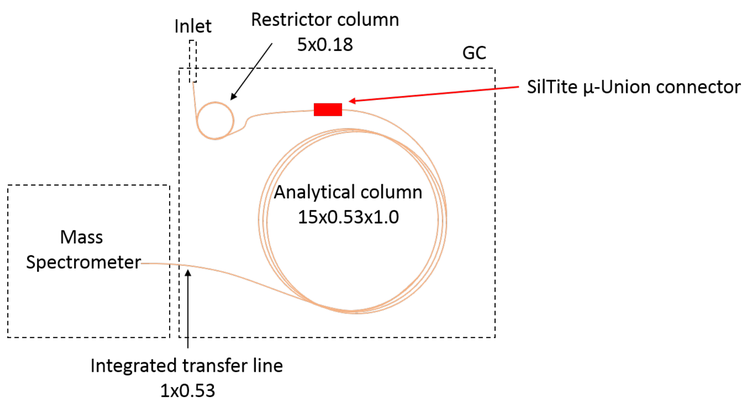
Introduction

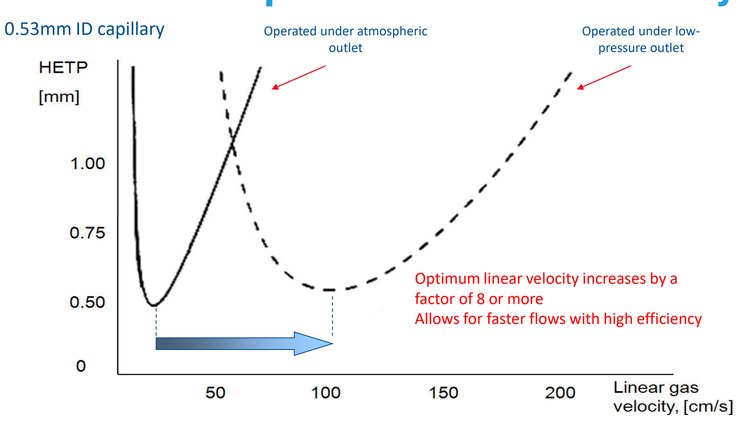
Concurrent Solvent Recondensation Large Sample Volume splitless injection (CSR-LVSI, or LVI) is a sample technique that overcomes the limitation of the maximum injection volume to 1 – 2 µL valid for classical splitless injection. Low Pressure Gas Chromatography is a novel technique that had been successfully 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 µL 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

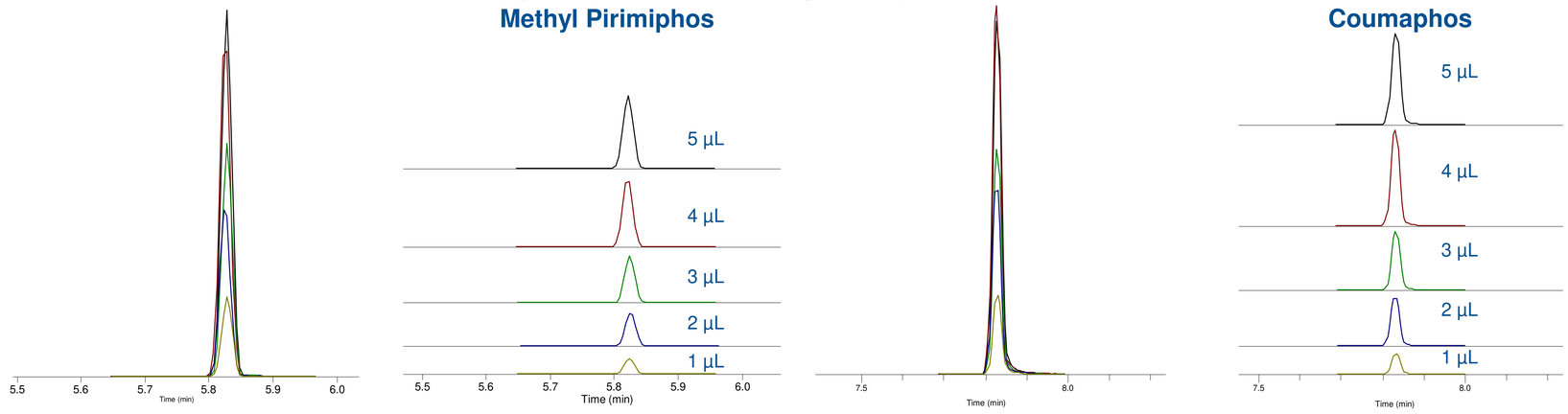
Large Volume Injections

- 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

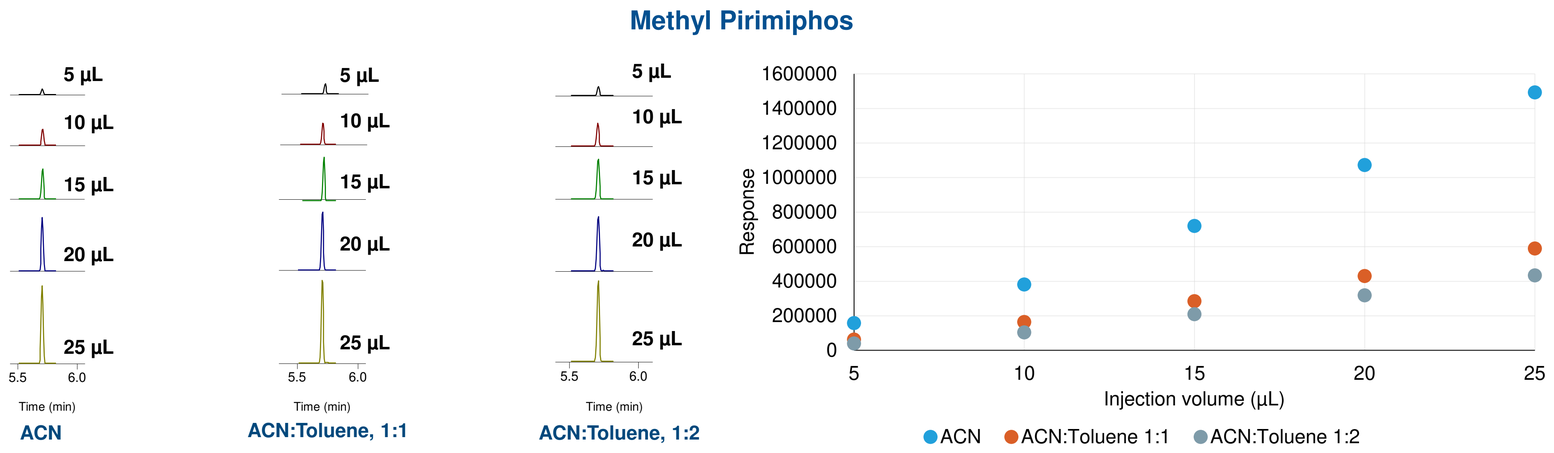
- Lower detection limits
- Saves time in sample prep
- Eliminate need for expensive PTV

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

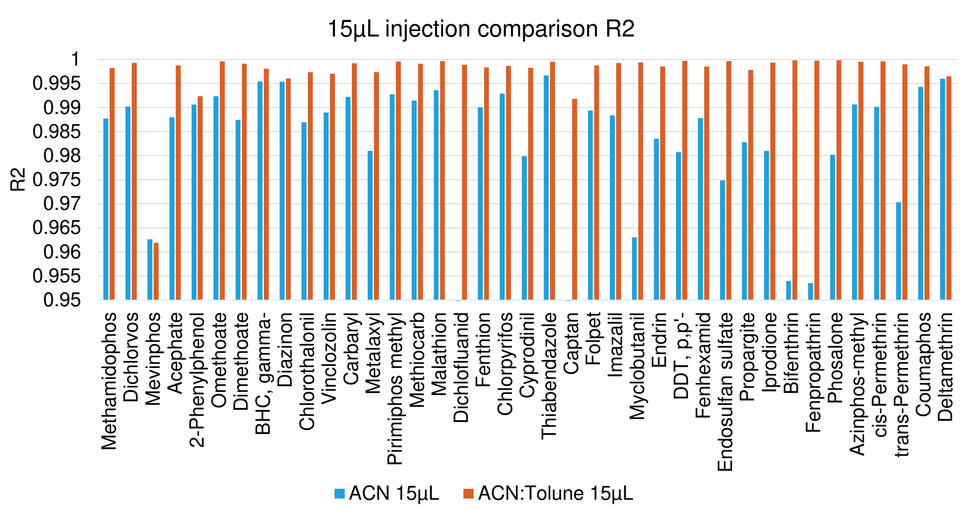
Comparison of 1-5 µL Injection in ACN

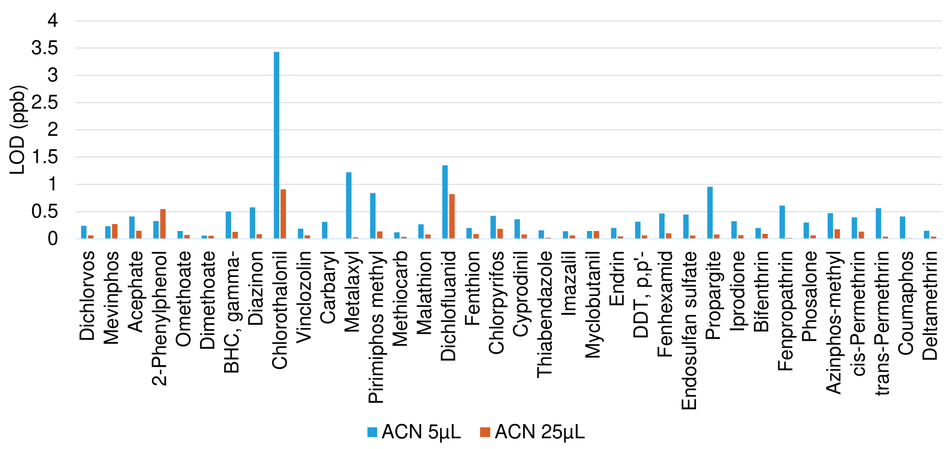


Comparison of 5-25 µL Injection in ACN and ACN:Toluene



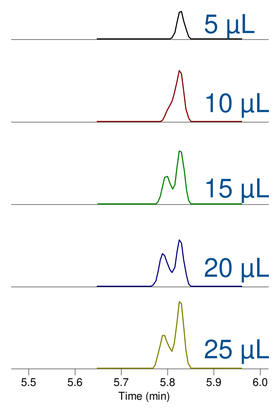
Calibration Comparison





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 µL 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