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Optimized System for Pulsed Injections and Backflushing in GC/MS Analysis of Pesticides with Acetonitrile Solvent

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Introduction

Acetonitrile is known to be a challenging solvent for splitless injections. Temperature-programmed injection with a multimode inlet is the best way of introducing QuEChERS extracts with acetonitrile solvent onto the GC column.¹

For the configurations limited to the split/splitless inlet (S/SL), improvements for splitless injection of acetonitrile with an S/SL inlet have been identified:

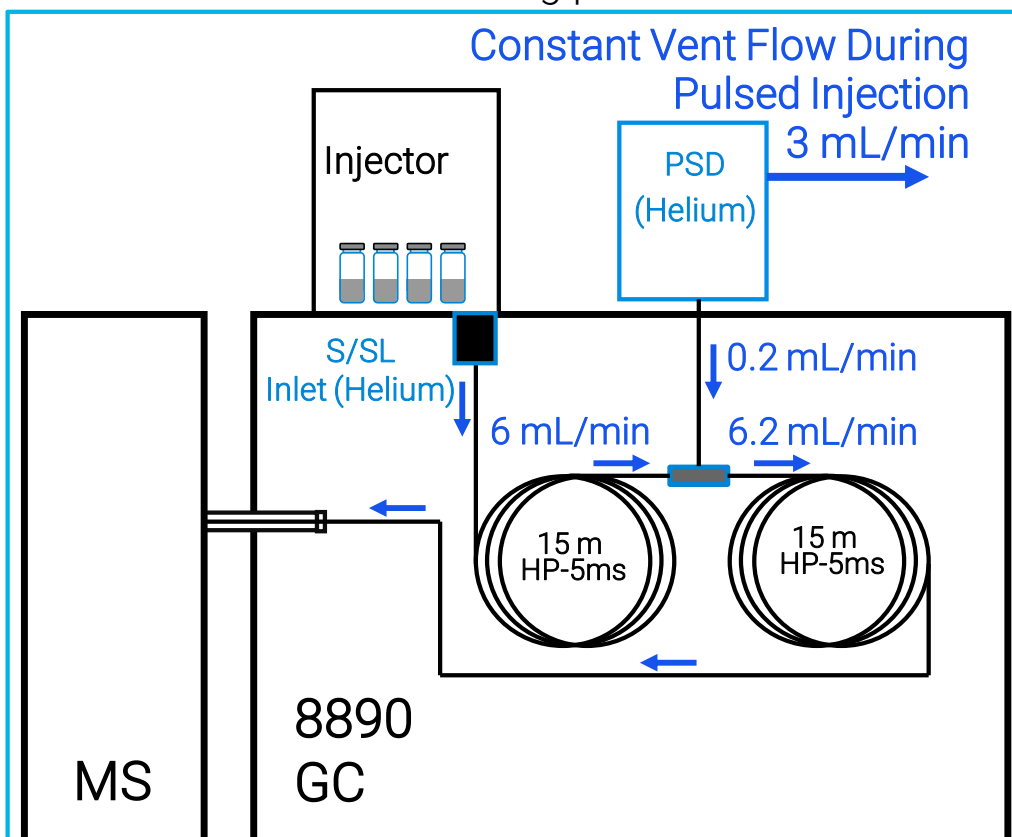
- Pulsed splitless injection provides effective transfer of the analytes from the inlet to the column minimizing the residence time
- Choice of the Universal Low Pressure Drop liner and adjustment of the initial oven temperature allows for hot pulsed splitless injection with acetonitrile

Unfortunately, one of the most widely used sample preparation techniques for pesticide residue analysis in food products results in acetonitrile being the GC injection solvent.

Experimental

Several GC/MSD and GC/MS/MS systems with multimode and split/splitless inlets were used to develop and evaluate the method.

- Two 15 m x 0.25 mm x 0.25 μm , 5% phenyl (polysiloxane) columns were used
- A purged union coupled with an electronic pressure control module (PSD) provided the mid-column backflush capability to reduce column maintenance by removing heavy matrix components from the head of the column during post-run



Experimental, cont.

Liner type, injection volume, and initial oven temperature were the key parameters to be optimized for successful use of acetonitrile as the injection solvent.

- Acetonitrile and isooctane were used for pesticide standard preparation, while the QuEChERS sample extracts were in acetonitrile
- A pulsed pressure of 50 psi was shown to maximize analyte transfer from inlet onto the column
- Single taper and low pressure-drop type GC liners were tested for pulsed splitless injection

UI Splitless Liner 5190-2293



UI Universal Low Pressure Drop Liner 5190-2295

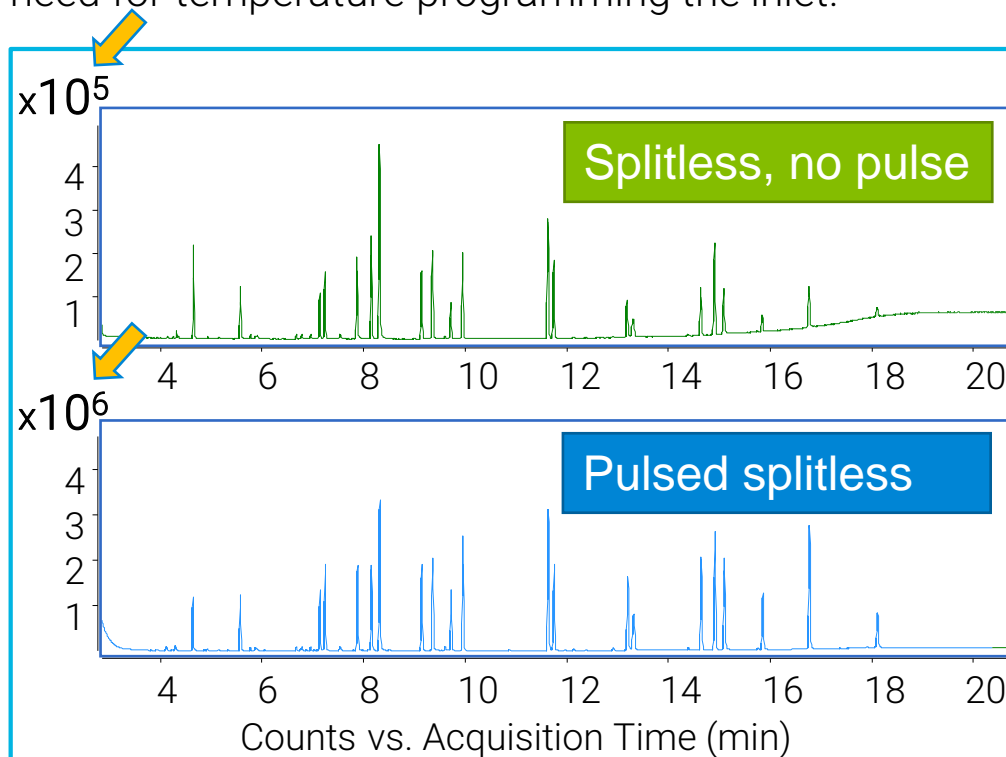


Single taper and low pressure-drop type GC liners.

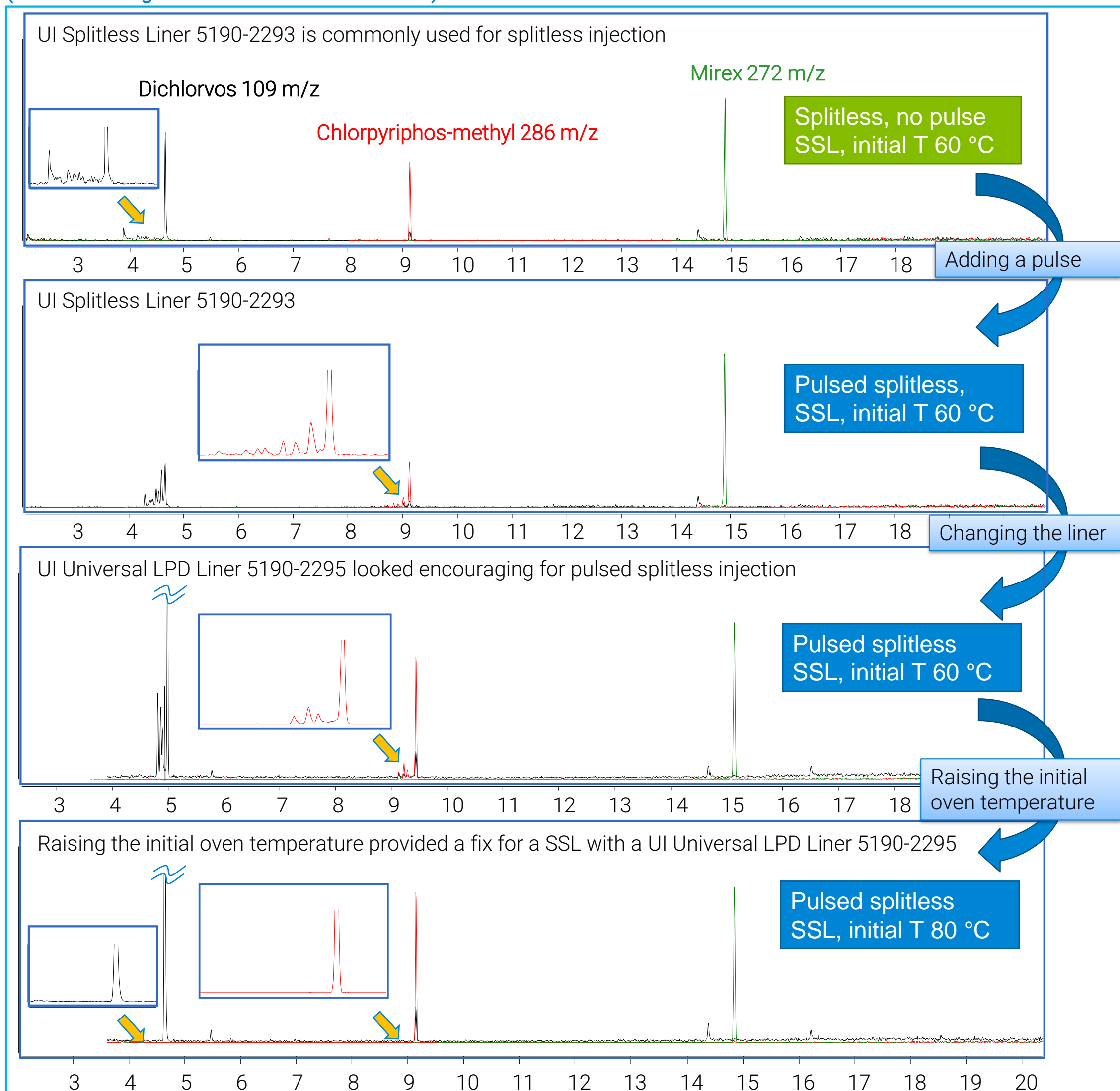
Results

Response Increase with Pulsed Splitless

Pulsed splitless injection allows for the effective transfer of high boiling and active compounds when using hot splitless injection (280 °C), reducing the need for temperature-programming the inlet.



Hot splitless injection of pesticide standards in acetonitrile with S/SL and a commonly used UI splitless liner: (the chromatograms are normalized to mirex)



With the commonly used single taper type liners, excellent chromatographic performance is achieved with isooctane, however, acetonitrile results in peak distortion. The LPD liner was found to resolve the problem with SSL inlet allowing for 1 μ L injection volume. The initial oven temperature had to be increased from 60 to 80 °C with SSL, adjusting the initial hold time to 1.5 min allows for maintaining the retention times with retention time-locking.

Parameters to Consider when Optimizing

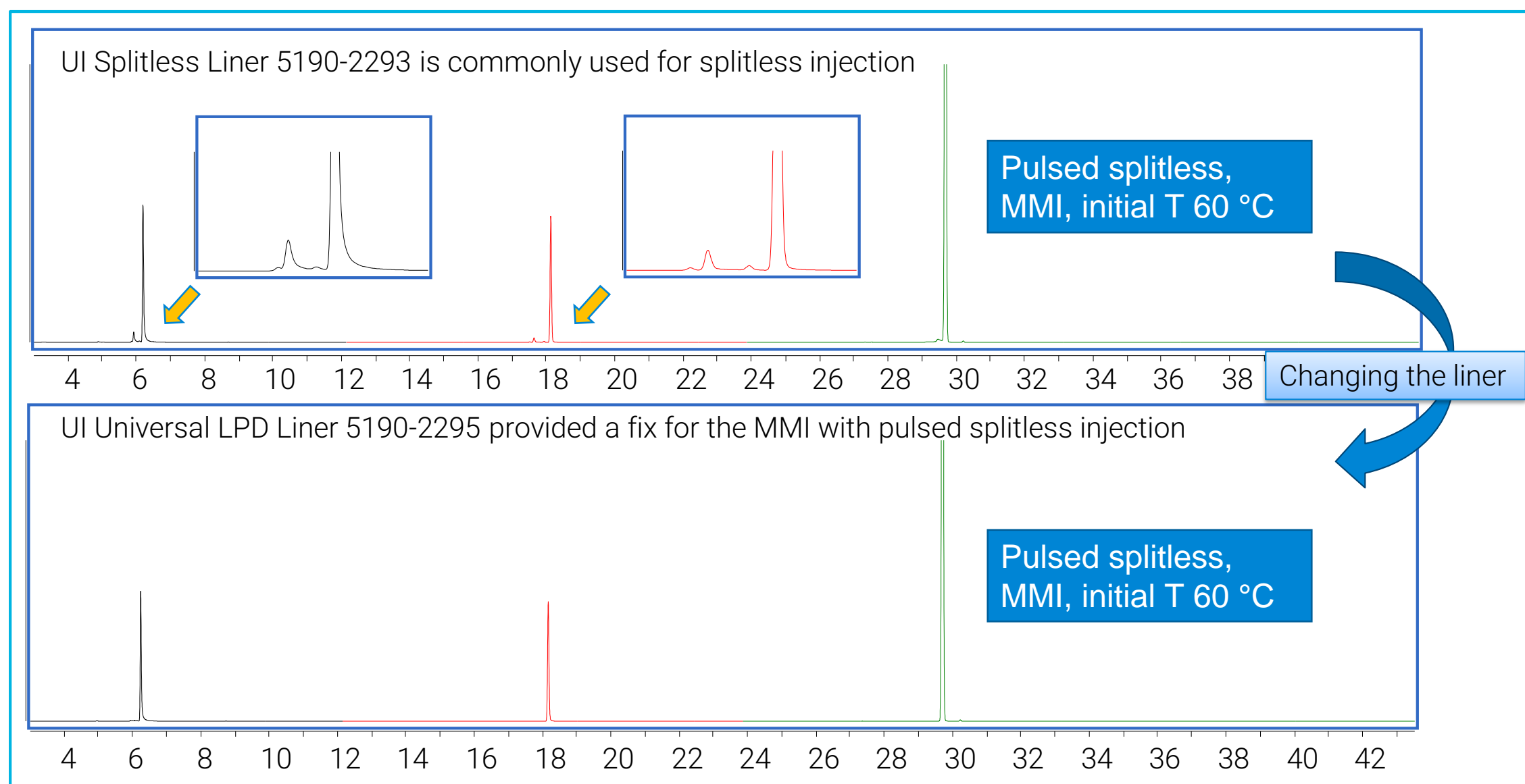
Using an MMI in pulsed hot splitless mode

- Use the UI Universal Low Pressure Drop liner for pulsed 1 μ L injections
- There is no need to adjust the initial oven temperature

Using a S/SL inlet in pulsed splitless mode

- Use the UI Universal Low Pressure Drop liner for pulsed 1 μ L injections
- The initial oven temperature may need to be increased to 80 $^{\circ}$ C

LPD liner with MMI in hot pulsed splitless mode allows for the initial oven temperature of 60 $^{\circ}$ C



Conclusions

While temperature-programming the inlet is the preferred sample introduction means, if it is not available, a hot pulsed splitless injection with either the split/splitless (SSL) or multimode inlets (MMI) can be used to advantage.

With the correct choice of parameters, the challenges caused by the solvents like acetonitrile can be minimized.

References

¹K. Mastovska. GC/MS/MS Pesticide Residue Analysis. A Reference Guide. Agilent Technologies, 2018.