GC-MSMS 200+ Multi-residue Pesticide Screening Workflow – Comparison of Conventional 30 m Column and LPGC Kit

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Abstract & Introduction

Throughput is one of the most important parameters in the lab. While there are numerous ways to increase the speed of analysis, the low-pressure gas chromatography (LPGC) is unique in going towards short, wider bore column rather than short and narrower one. Coupling this type of column with a narrow guard column with a restrictor allows a normal head pressure at the inlet, while the analytical column is operated under near-vacuum conditions. The low pressure inside the wide-bore column shifts the optimum linear velocity about a factor 7 higher, which allows for faster analysis without a total loss of efficiency. The wider ID and thicker film provide also higher capacity, robustness and inertness. To demonstrate the technique, spinach was spiked with over 200 pesticides at two levels, 100 ppb and 10 ppb and analysis on conventional column (Rxi-5MS column, 30 m x 0.25 mm x 0.25 µm) was compared to the analysis on the LPGC column set.

Sample preparation and analytes

Sample preparation: Spinach was extracted using the QuEChERS methodology, namely: 15 g of homogenized spinach was spiked with internal standard (triphenyl phosphate) and calibration solution . It was mixed with 15 mL of ACN (1% acetic acid) and AOAC salt packet (6 g $MgSO_4$, 1.5 g NaOAc) in 50 mL centrifuge tube. The centrifuge tube was shaken for 1 min after addition of ACN and after the salt addition. Supernatant was removed after centrifugation. Further cleanup was performed by adding extract to the dSPE sorbent vials (150 mg $MgSO_4$, 50 mg PSA, 50 mg C18-EC, 50 mg GCB). These were also briefly shaken immediately after addition to prevent clumping and subsequently vortexed for 30 seconds. Extracts were analyzed immediately.

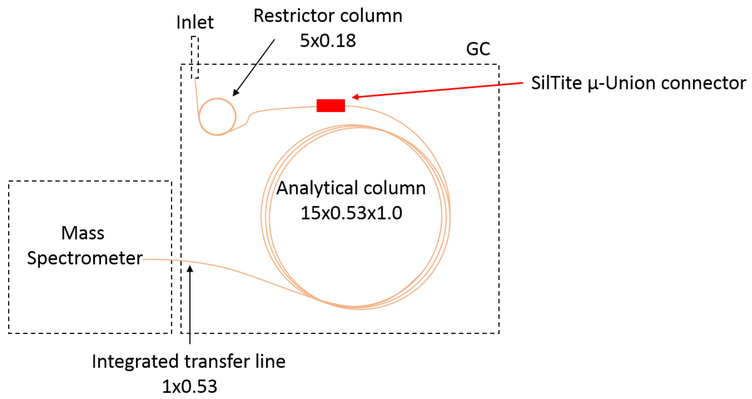
Analytes: Pesticide multiresidue mix was used (203 components) including:

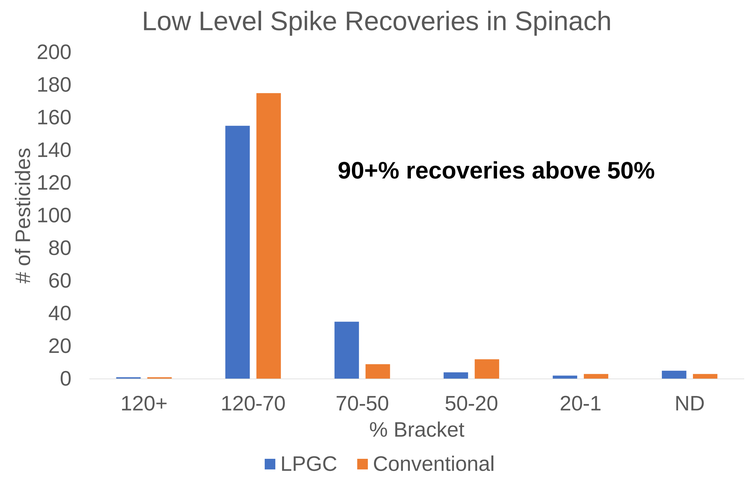
- Organophosphorus compounds
- Organochlorine Compounds
- Organonitrogen Compounds
- Synthetic Pyrethroid Compounds
- Herbicide Methyl Esters

Analysis Conditions

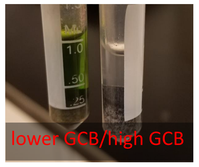
	Conventional Analysis	LPGC Analysis
Column	Rxi-5ms	LPGC Rtx-5MS
Flow	1.4 mL/min	2 mL/min
Temp program	90 °C (hold 1 min) to 330 °C at 8.5 °C/min (hold 5 min)	80 °C (hold 1 min) to 320 °C at 35 °C/min (hold 5 min)
Injection volume	2 µL split (split ratio 10:1)	
Injection temperature	250 °C	
Transferline temperature	290 °C	
Source temperature	330 °C	
Allidochlor RT (1st comp.)	**7.06 min**	**3.34 min**
Deltamethrin RT (last comp.)	**26.31 min**	**8.31 min**

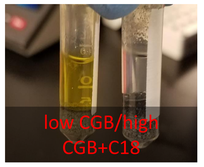
LPGC setup



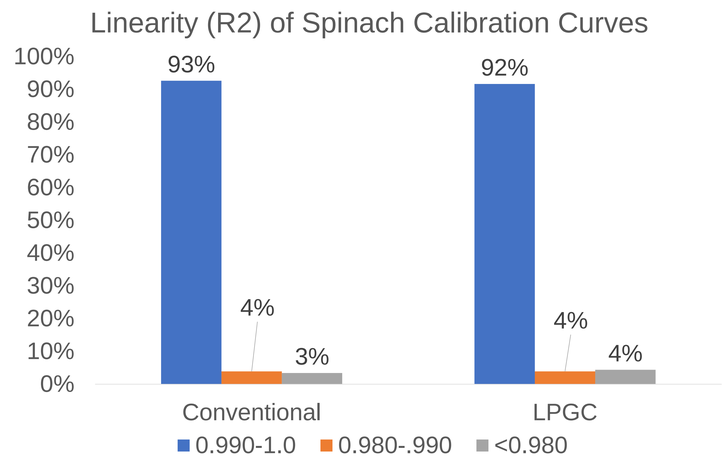


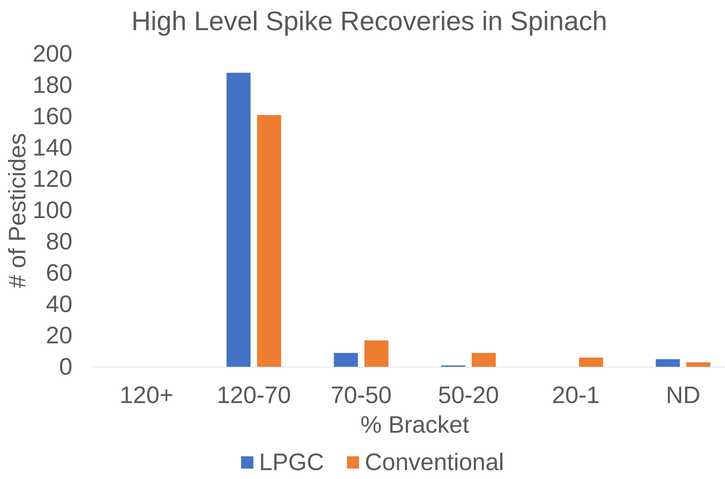
Spinach is in particular difficult commodity where the pigment cleanup affects recoveries for some pesticides, in particular planar non-polar pesticides. Therefore, recoveries lower than 70% are not usual. Interestingly, we've seen very similar recoveries for the high level spike, with better recoveries using the conventional setup for low level spikes





Linearity





Conclusions

- More than three times faster analysis with LPGC
- No decrease in linearity
- Some decrease in recoveries, especially for low level samples, however, majority residues still above 50%
- Analyte protectants were not used in this study. Their effect on the low level of recoveries will be the next phase of our study