Defining Robustness of Pesticide Analysis in *Cannabis* Matrices

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KEY FINDINGS

- The SCIEX Triple Quad 6500+ system demonstrates robustness with reproducible performance in matrix across a large sample set of 830 samples without cleaning or maintenance of the system
- Due to the highly complex and challenging matrix a decrease in sensitivity over time is expected
- IS corrected data can fail to capture changes in sensitivity over time
- It is important to consider the ion ratio reproducibility in addition to the raw peak area reproducibility to assess instrumental performance

MATERIALS AND METHODS

Sample preparation: A 1:10 dilution was performed using 5 g of homogenized *Cannabis* flower extracted in 50 mL of 0.1% formic acid in acetonitrile. Extracts were winterized at -20ºC for 2 hours before filtration with 0.2 µm PTFE syringe filters.

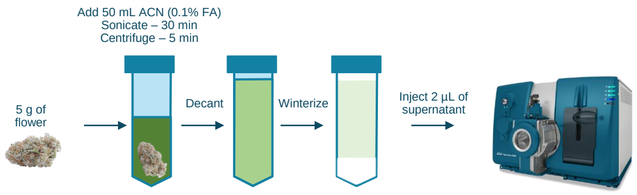

Figure 1. Sample preparation approach for a variety of Cannabis food matrices and flower

LC-MS/MS: A 5-minute gradient was used to inject *Cannabis* flower matrix repeatedly for analysis. An analytical 20-minute gradient representing a typical analysis strategy was injected every 10th sample for comparative analysis. The analytical column used was a Phenomenex 3 µm Luna Omega Polar C18 (3x150 mm) and chromatographic separation was achieved using 5mM ammonium formate with 0.1% formic acid in water and methanol.

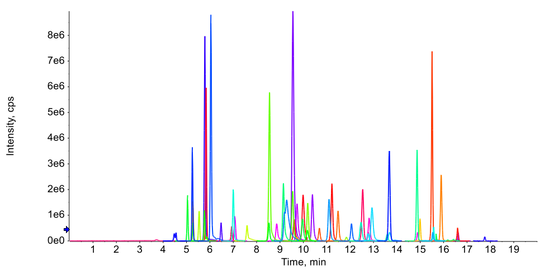

Figure 2. Example of 20-min gradient in *Cannabis* flower matrix.

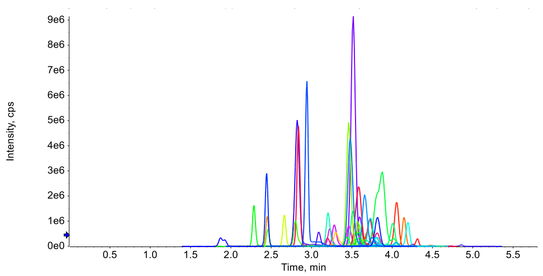

Figure 3. Example of 5-min gradient in *Cannabis* flower matrix.

RESULTS

IS Corrected Area vs. Raw Area over 830 injections of *Cannabis* flow matrix

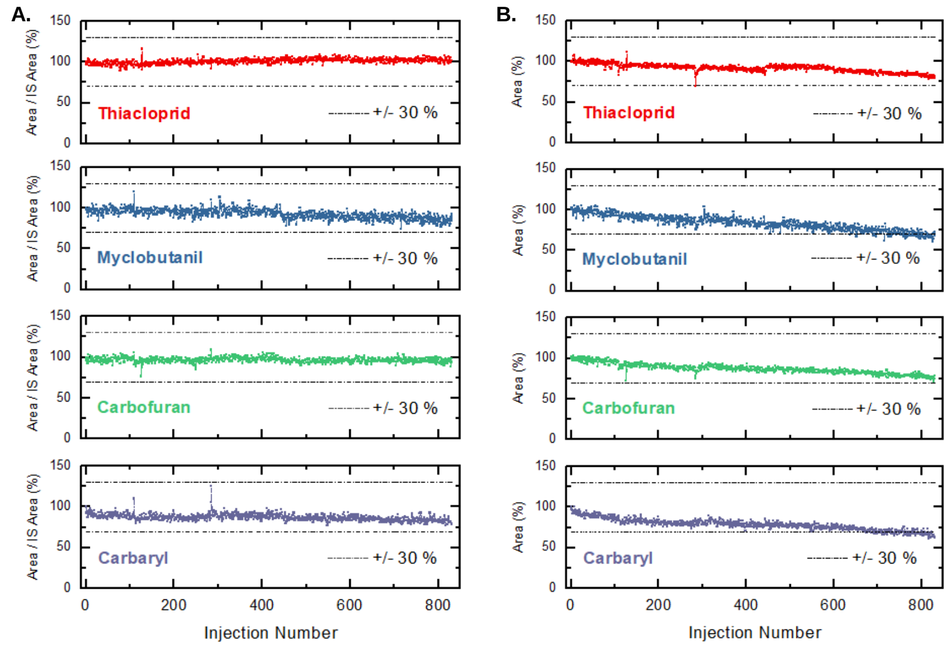

Figure 4. 830 replicate injections of *Cannabis* flower matrix. IS-corrected (A) and raw peak area (B)

- IS ratio remains consistent across many injections
- The true measure of instrument robustness must be an evaluation of the uncorrected peak area as a function of time
- Expected decrease in peak area is observed when the raw area is plotted (Figure 4B)

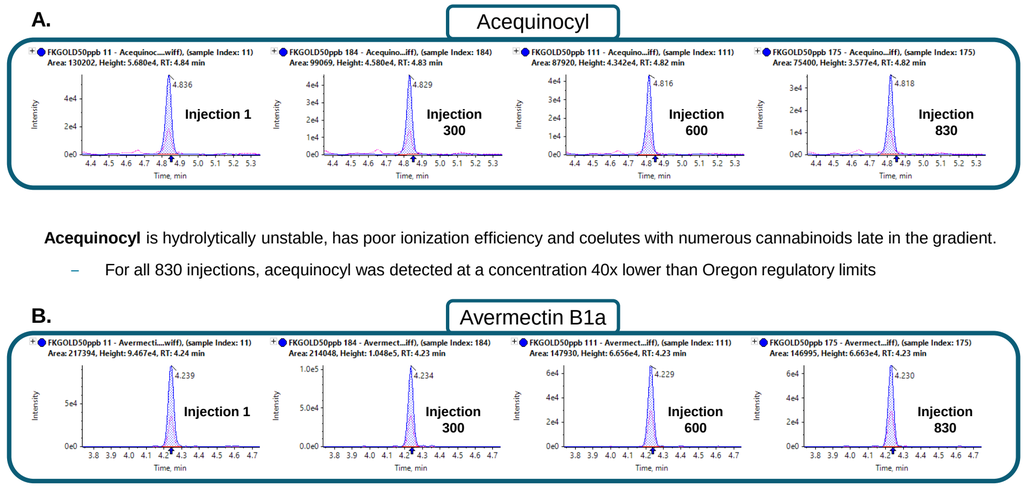

Figure 5. Stable pesticide peak areas across 830 injections at 0.05 ppm in cannabis flower. Acequinocyl (A) and Avermectin B1a, the primary component of Abamectin (B)

Acequinocyl is hydrolytically unstable, has poor ionization efficiency and coelutes with numerous cannabinoids late in the gradient.

- For all 830 injections, acequinocyl was detected at a concentration 40x lower than Oregon regulatory limits

Avermectin B1a is known for its thermal lability

- Detected at a concentration 10x lower than Oregon regulatory limits[5] after the 830 matrix injections
- Both quantifier and qualifier are clearly visible with no impact on ion ratio
- Excellent sensitivity is maintained, even under the extreme conditions employed for this robustness test

Table 1. LOQ analysis of OR List pesticides in *Cannabis* flower extracts using the Triple Quad 6500+ System from spiked matrix curve replicate injections (n=7).

Compound	LOQ (ppb)	%CV	Compound	LOQ (ppb)	%CV
*Abamectin**	0.25	13%	*Imazalil*	0.25	3%
Acephate	0.25	4%	*Imidacloprid*	0.25	6%
Acequinocyl	1	7%	*Kresoxim-methyl*	0.25	18%
Acetamiprid	0.25	4%	*Malathion ≠*	0.25	6%
Aldicarb	0.25	9%	*Metalaxyl*	0.25	2%
Azoxystrobin	0.25	5%	*Methiocarb*	0.25	3%
Bifenazate	0.25	5%	*Methomyl*	0.25	4%
Bifenthrin	0.25	13%	*MGK 264* ≠*	0.25	10%
Boscalid	1	3%	*Myclobutanil*	0.25	18%
Carbaryl	1	10%	*Naled*	0.25	17%
Carbofuran	0.25	19%	*Oxamyl*	0.25	5%
Chlorantraniliprole	0.25	13%	*Parathion Methyl ≠*	0.25	5%
Chlofenapyr	5	22%	*Permethrins**	1	19%
Chlorpyrifos	1	2%	*Phosmet*	0.25	5%
Clofentezine ≠	0.25	12%	*Piperonyl Butoxide*	0.25	6%
Cyfluthrin	5	16%	*Prallethrin ≠*	0.25	2%
Cypermethrin	2	10%	*Propiconazole*	0.25	6%
Daminozide ≠	5	4%	*Paclobutrazol*	1	8%
Diazinon	0.25	9%	*Propoxure*	0.25	3%
Dichlorvos	0.25	11%	*Pyrethrins**	0.25	18%
Dimethoate	0.25	3%	*Pyridaben*	1	2%
Ethoprophos ≠	1	6%	*Spinosad**	0.25	3%
Etofenoprox	0.25	2%	*Spiromesifen*	1	11%
Etoxazole	0.25	1%	*Spirotetramat*	0.25	7%
Fenoxycarb ≠	0.25	6%	*Spiroxamine*	0.25	6%
Fenpyroximate	1	3%	*Tebuconazole*	1	14%
Fipronil	1	14%	*Thiaclomprid*	0.25	2%
Flonicamid	0.25	2%	*Thiamethoxam*	0.25	4%
Fludioxinil	0.25	2%	*Trifloxstrobin*	0.25	4%
Hexythiazox ≠	0.25	4%			

**Analytes is based on the most abundant isomer.*
≠Analytes have %recoveries that can be improved using their deuterated internal standards.

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TRADEMARKS/LICENSING