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Planar SPE coupled to flow injection TOFMS analysis – a rapid pesticide screening tool

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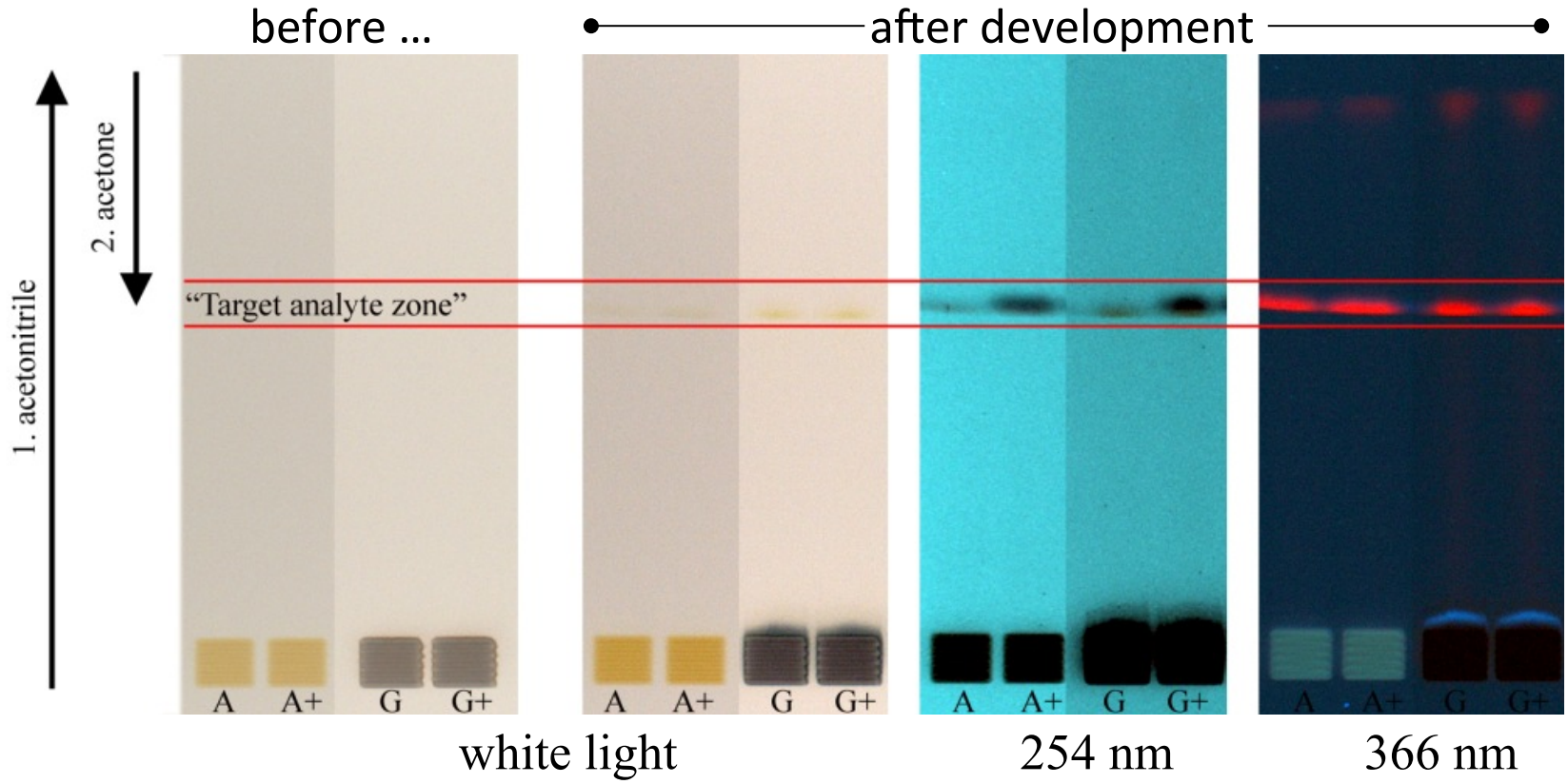
Planar solid phase extraction

- a new cleanup concept in multi-residue analysis of pesticides

- Application of QuEChERS extracts (50 µL) onto TLC amino plates
- Twofold development



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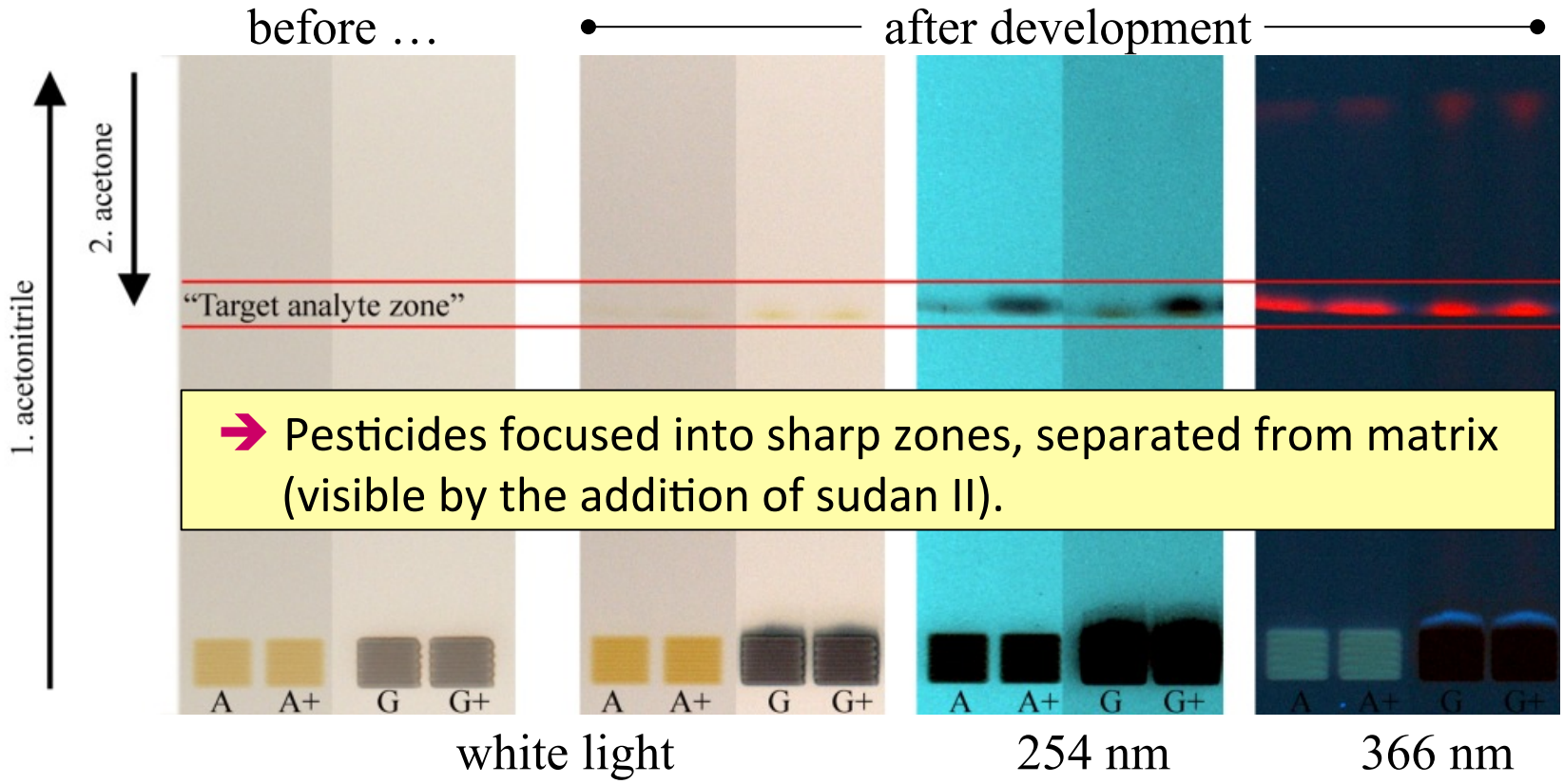

➔ Pesticides focused into sharp zones, separated from matrix (visible by the addition of sudan II).

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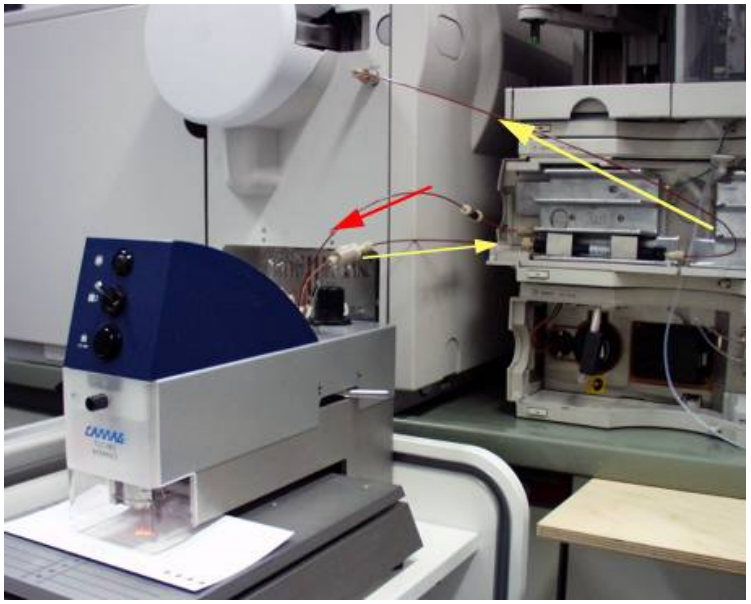
TLC-LC/MS



Extraction of target zones (acetonitrile/ammonium formate)

- on-line coupling to LC/MS column (Chromolith RP-18)
- elution into autosampler vials (LC/MS or GC/MS)

➔ Very clean, matrix-free extracts, no matrix effects!

C. Oellig, W. Schwack, J. Chromatogr. A (2011) 1218, 6540-6547

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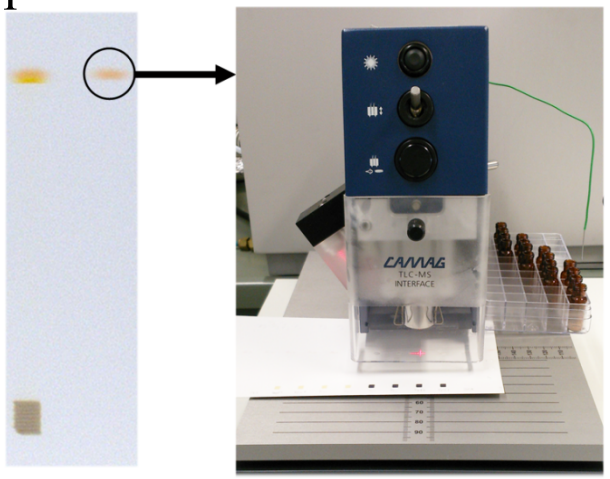
pSPE–µL-FIA–TOFMS

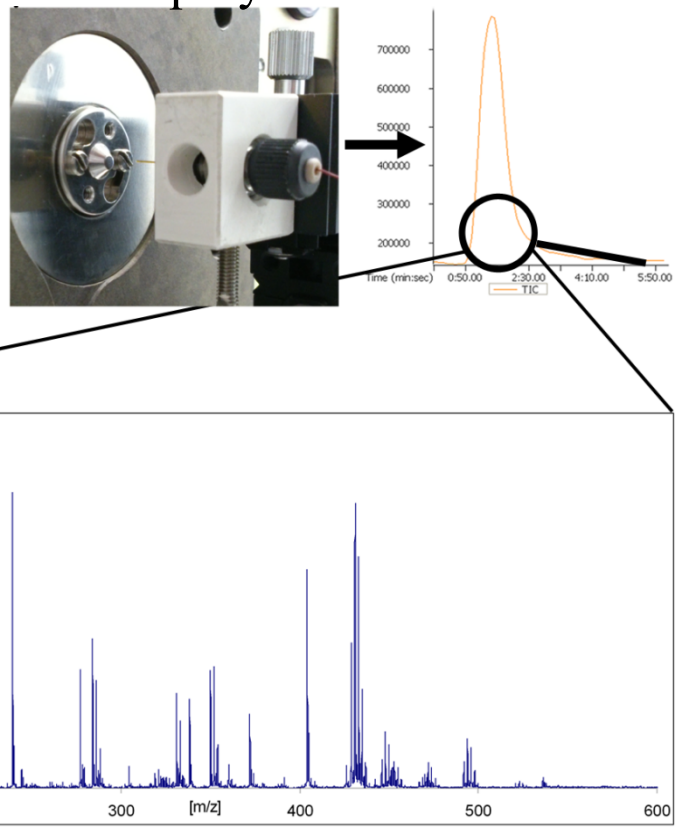
pSPE

TLC-MS



µL-FIA

nano-spray TOFMS



C. Oellig, W. Schwack, J. Chromatogr. A (2014) 1351, 1-11

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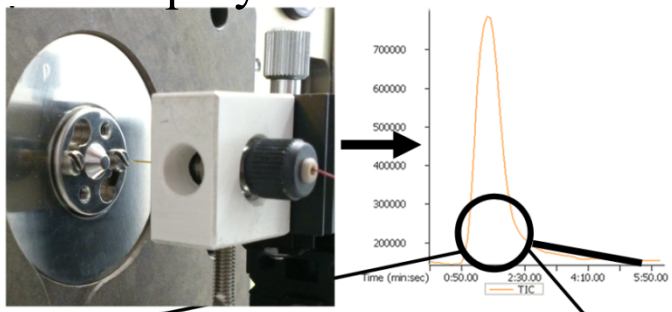
pSPE–µL-FIA–TOFMS

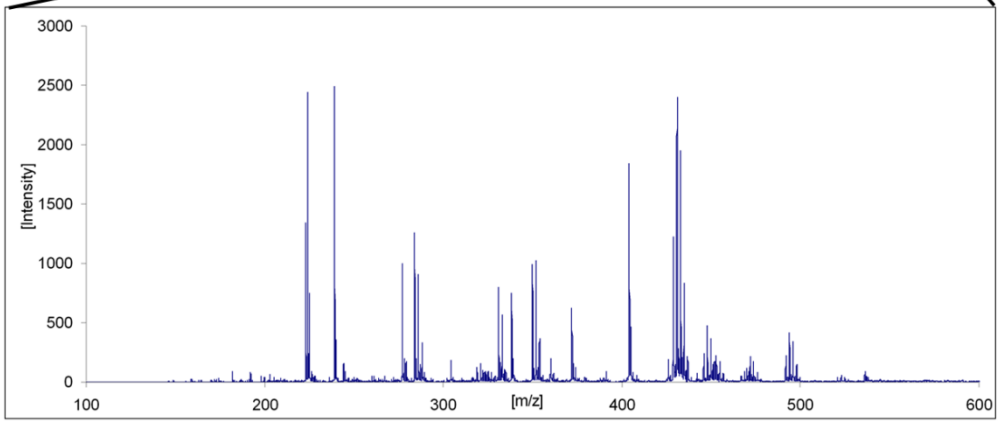
pSPE

TLC-MS

µL-FIA

nano-spray TOFMS





- No chromatography, all pesticides in a single FIA peak.
- Whole sample information in a single mass spectrum.

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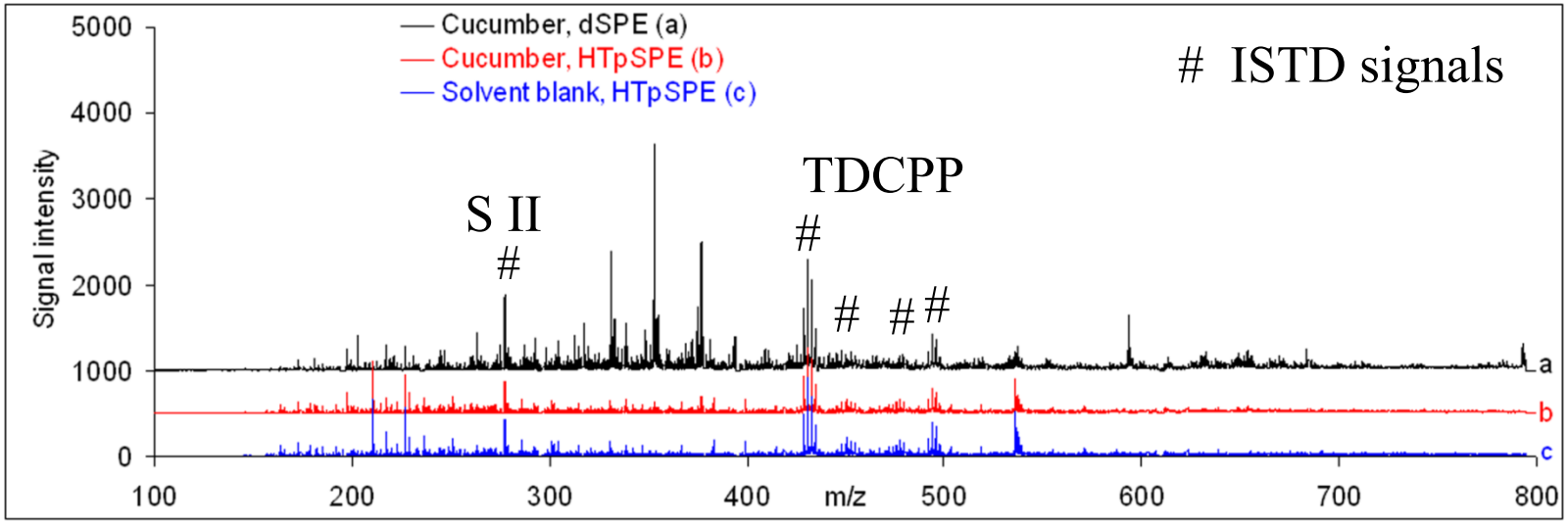
Example cucumber extracts



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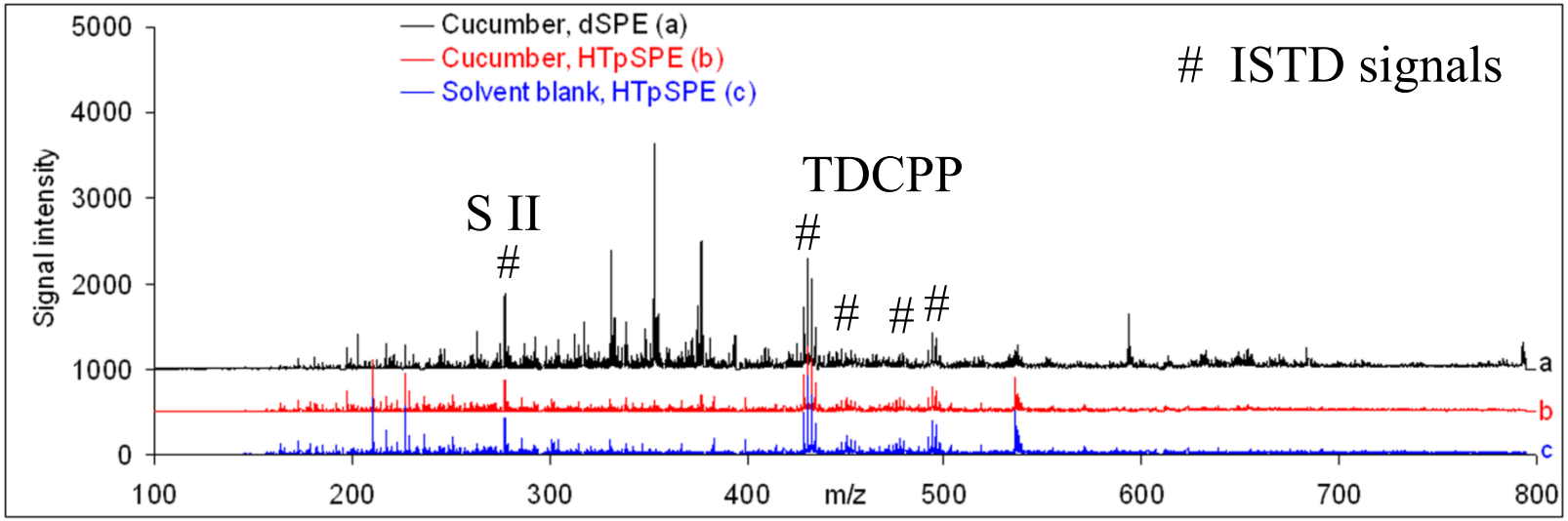
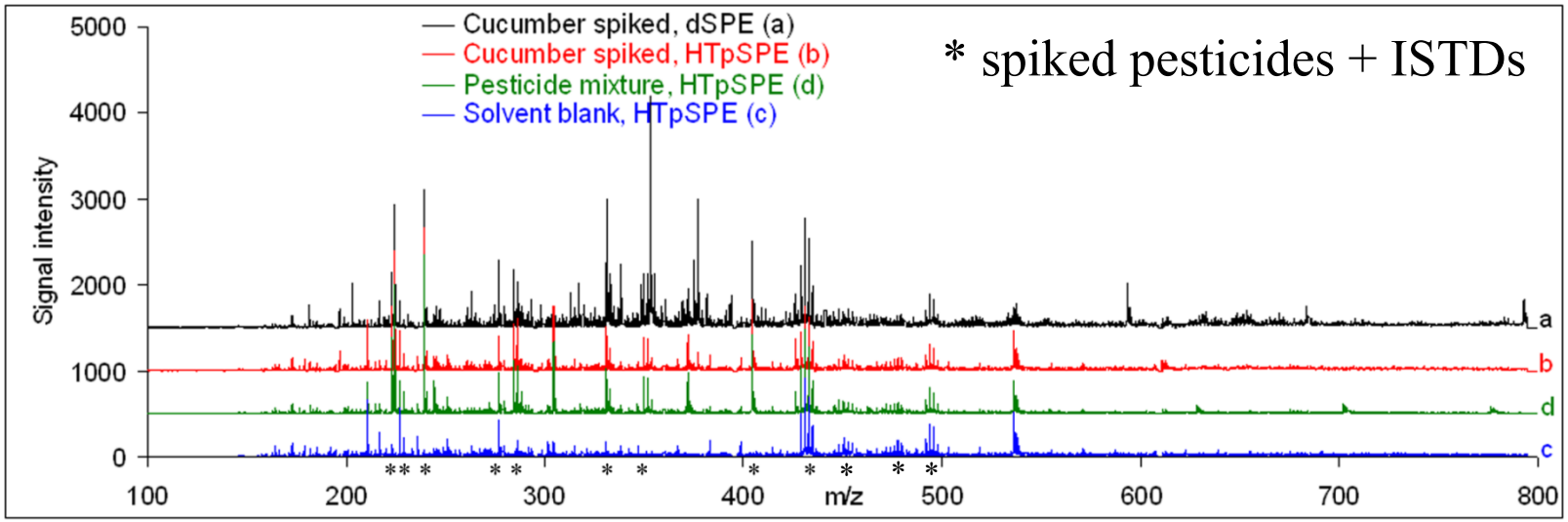
Example cucumber extracts





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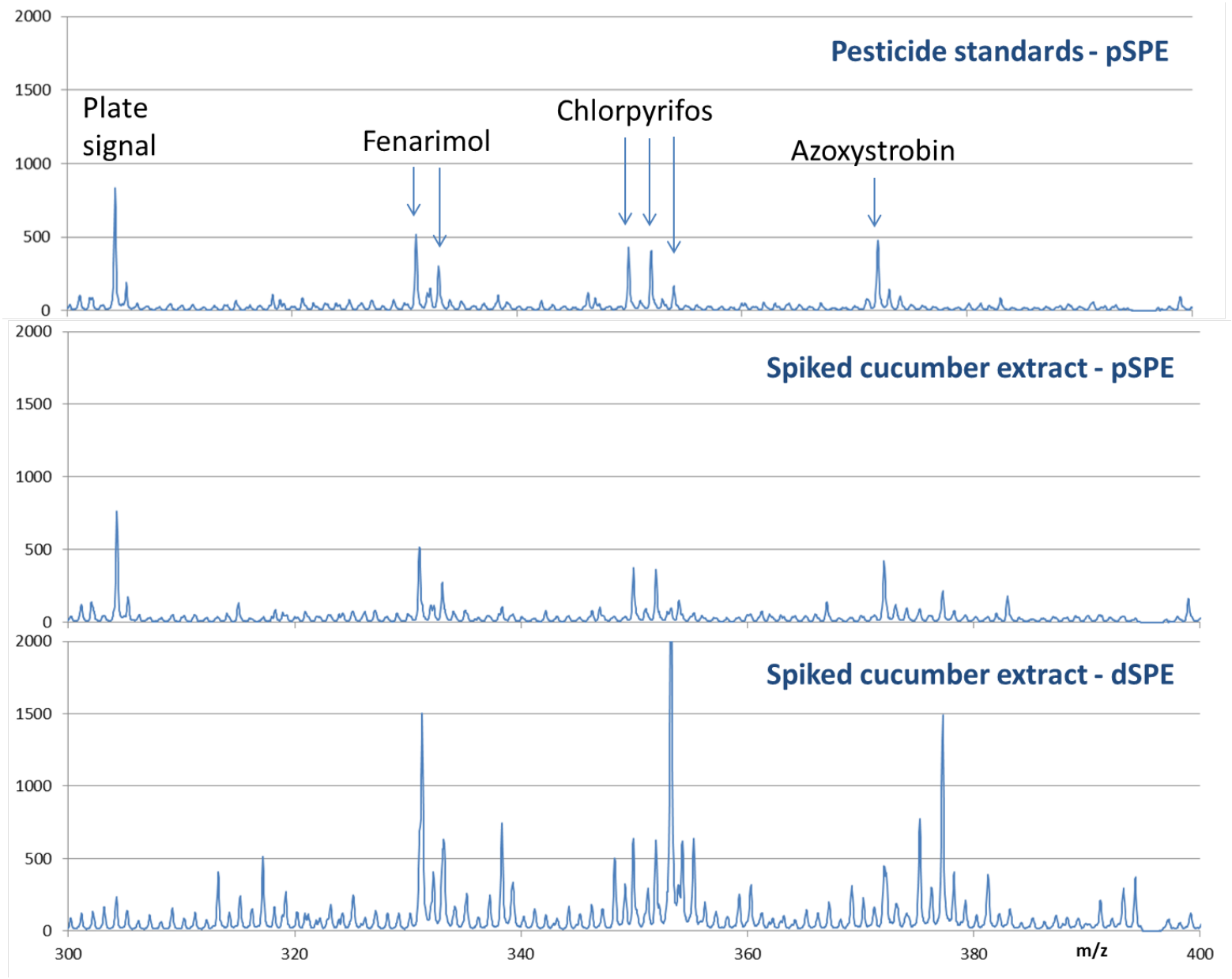
Zoom



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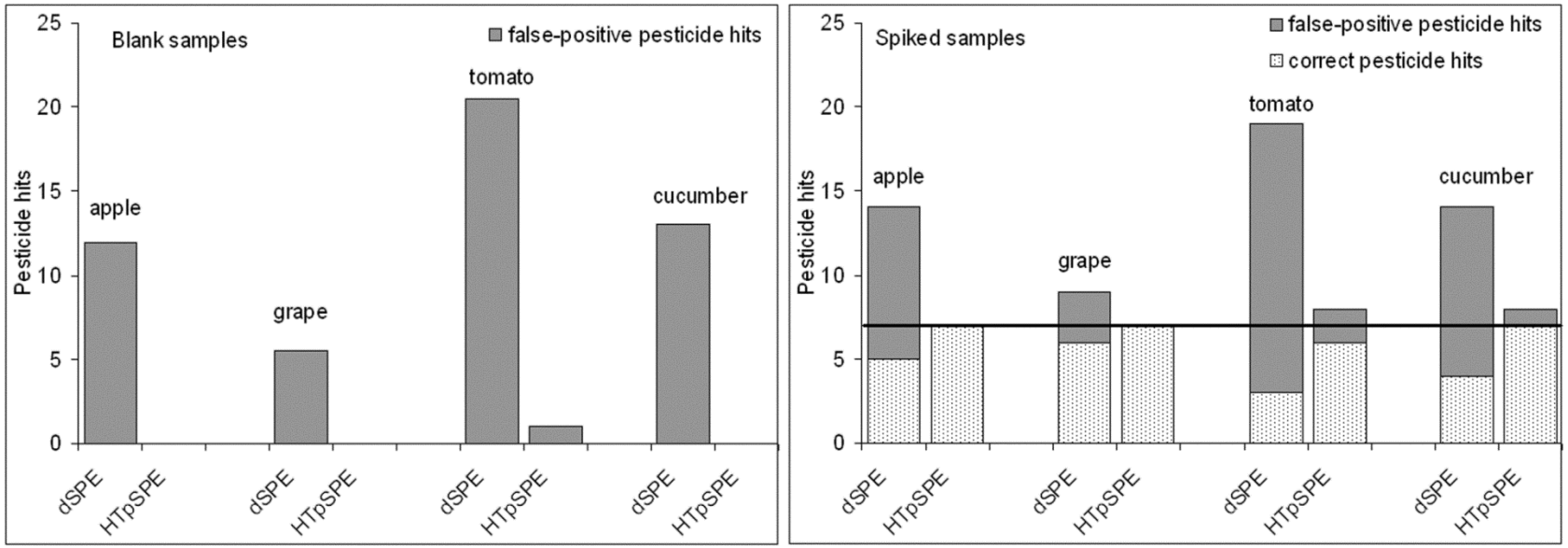
Target / Non-target screening



EXCEL Sheet 1: data base table (about 250 pesticides)

EXCEL Sheet 2: results table (TOFMS mass list)

ACCESS Query: comparison of tables
- positive correlation = target screening
- negative correlation = non-target screening

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Real samples - pSPE-FIA-TOFMS ↔ dSPE-LC-MS/MS [1)]

Sample	Pesticide	LC-MS/MS [mg/kg]	FIA-TOFMS [S/N]
Banana	Thiabendazole	**0.76**	**2004**
	Imazalil	**0.73**	**1090**
Blackberry	Cyprodinil	**0.86**	**2347**
	Lambda-cyhalothrin	0.01	n.d.
	Thiacloprid	0.01	n.d.
Currant	Cyprodinil	**0.47**	**1373**
	Boscalid	**0.37**	**124**
	Pyraclostrobin	**0.14**	**121**
	Trifloxystrobin	0.06	n.d.
Savoy cabbage	Azoxystrobin	**1.3**	**1770**
	Difenoconazole	**0.39**	**317**
	Pymetrozine	0.20	n.d.
	Cyfluthrin	0.09	n.d.
	Indoxacarb	0.03	n.d.

1) Institute Kirchhoff, Berlin

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Conclusions

- Even our low-resolution TOFMS provided exact results.
- The novel pSPE-FIA-TOFMS concept fully meets the SANCO document criteria for a qualitative screening method.
- FIA-TOFMS clearly showed the high efficiency of planar SPE.
- All residues are detected in a single peak/mass spectrum.
- A simple and rapid target screening is allowed.
- A true non-target screening is also simply enabled.
- A state-of-the art HRMS undoubtedly will improve the hit rates.

☞ Poster P-37 (experimental details)

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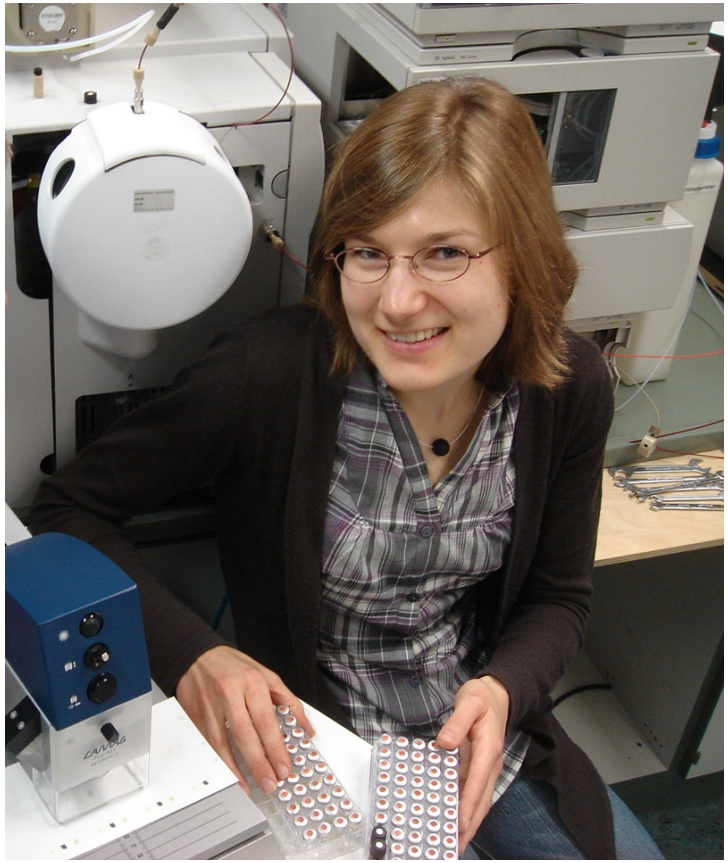
Many thanks!



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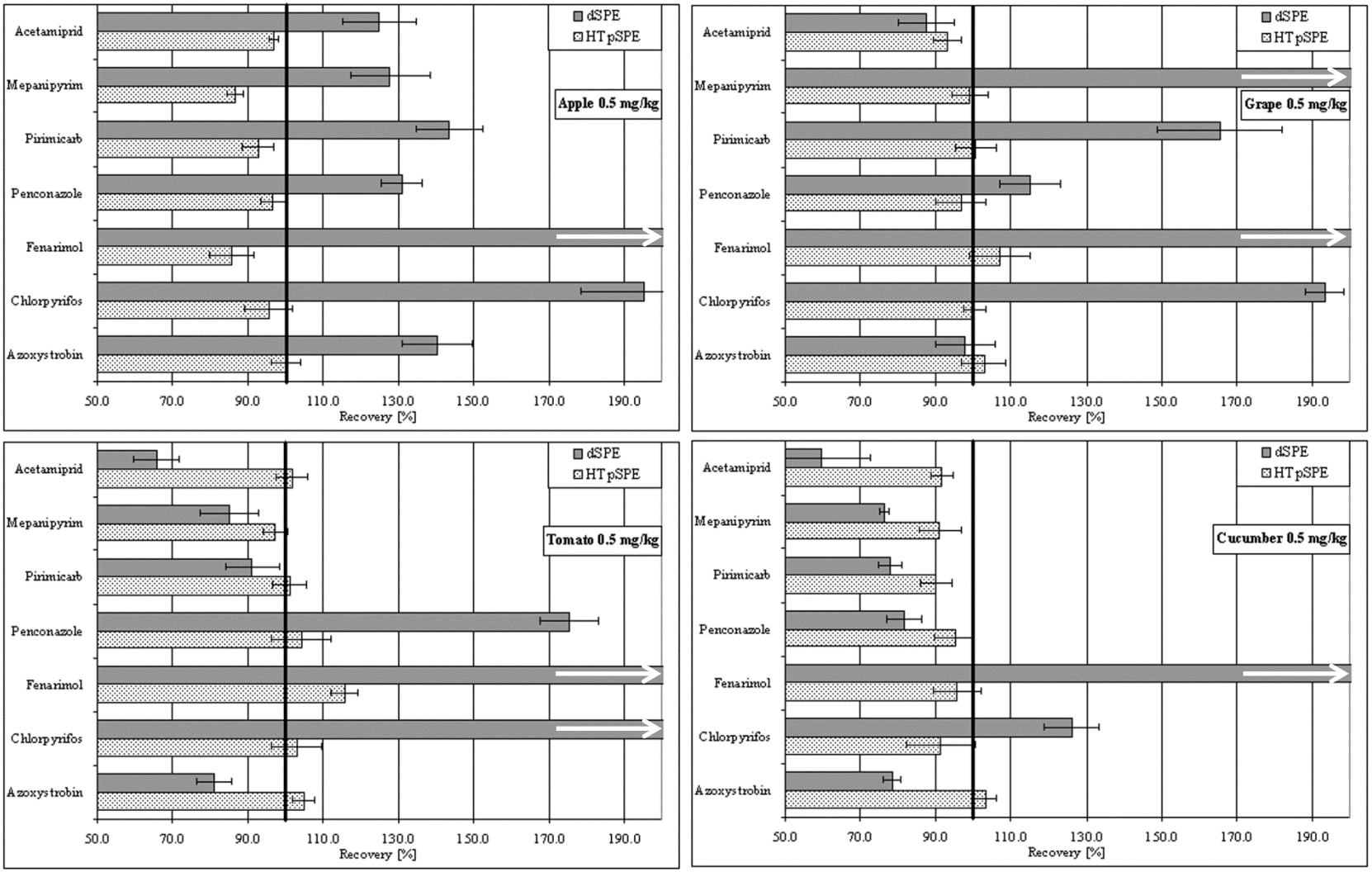
Matrix effects ↔ recoveries (n=5)



post-extraction spiking