

Exploring the application of a universal method for pesticide screening in foods using a high data acquisition speed MS/MS

ASMS 2012 WP27-574

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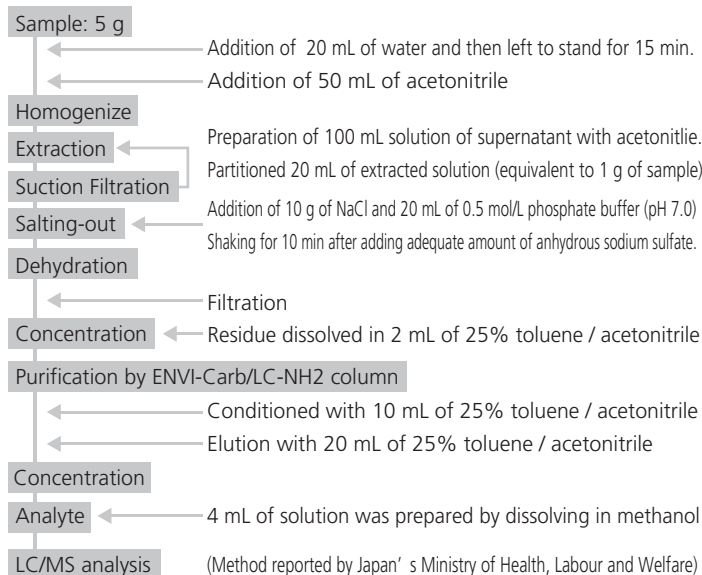
Introduction

Effective management, use, and disposal of agrochemicals, particularly pesticides, is an increasingly important health and environment issue in developing countries where economies may be heavily reliant on agriculture. The conventional approach to monitor these pesticides is to develop highly optimized triple quadrupole MRM methods to achieve the required levels of sensitivity, selectivity and

speed of analysis whilst still providing confidence in pesticide identification. In this study LCMS technology, developed for ultra-fast scanning MRM analysis, allows the possibility of a single generic 'universal' method. High speed MRM analysis and a generic parameters were used for screening 172 pesticides (344 MRM transitions) with 5 msec dwell and 1 msec pause times in food matrices.

Materials and Methods

Sample Preparation



Features of LCMS-8040

- 5 times higher sensitivity compared to LCMS-8030
- An ultra fast scan speed of 15000 u / sec.
- An ultra fast polarity switching of 15 msec.
- An ultra fast MRM transition speed of 555 ch./ sec.



Fig. 1 LCMS-8040 Triple Quadrupole Mass Spectrometer

Setting of MRM analysis & integration parameter

- In this study, no scheduling of MRM transitions was applied; thereby creating a universal generic method.

Analytical Conditions

HPLC : Nexera UHPLC system

Column : Shim-pack XR-ODSII (75 mm x 2 mmI.D., 2.2 um)
 Mobile phase : A ; 5 mM ammonium acetate – water
 B ; 5 mM ammonium acetate – methanol
 Gradient program : 30% B (0 min.) → 80% B (4 min.) → 95% B (10-15 min.) → 30% B (15.01-20 min.)
 Flow rate : 0.2 mL / min.
 Column temperature : 40°C

MS : LCMS-8040 Triple quadrupole mass spectrometer

Ionization : ESI (Positive / Negative)
 Ion spray voltage : +4.5 kV / -3.5 kV
 MRM : 344 MRM transitions (2 MRMs / compound)

FilterInstrument Parameters View (Realtime analysis)

The screenshot shows the MS Data Processing View with the following annotations:

- 167 compounds from 'Residual pesticide method package' + 5 compounds** (highlighted in a red box)
- Positive** and **Negative** ionization modes (highlighted in a blue box)
- Dwell 5 msec. Pause 1 msec. Loop time 2.058 sec.** (highlighted in a red box)
- Acquisition time set to the entire chromatographic run (0-20 min.).** (highlighted in a red box)
- 'Largest peak' selected for identification.** (highlighted in a blue box)
- MS Data Processing Parameters** window showing:
 - Identification Method: Absolute Pk
 - Peak Selection: Largest Peak
 - Retention Window: 100 s
 - Default Bandwidth: 20 min

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Results

Screening of 10 pesticides in food matrices

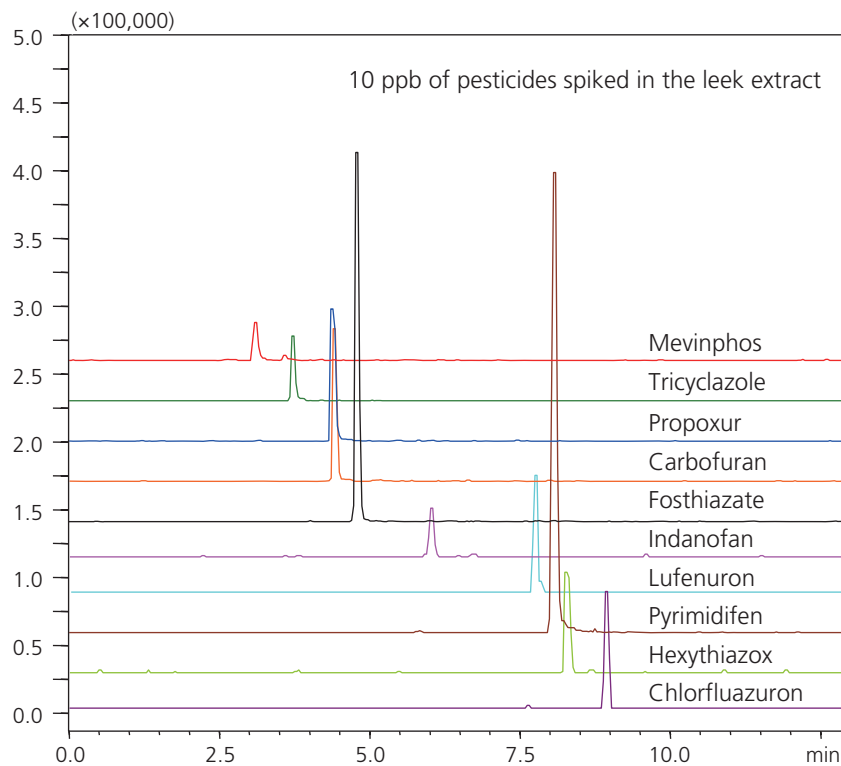


Fig. 2 MRM chromatogram of 10 pesticides

Table 2 Result of 10 pesticides screening (10 ppb spiked in each matrices)

Compounds	LeeK	Paprika	Green tea leaves
Carbofuran	✓	✓	✓
Chlorfluazuron	✓	✓	✓
Fosthiazate	✓	✓	✓
Hexythiazox	✓	✓	✓
Indanofan	✓	✓	✓
Lufenuron	✓	✓	✓
Mevinphos	✓	✓	✓
Propoxur	✓	✓	✓
Pyrimidifen	✓	✓	✓
Tricyclazole	✓	✓	✓
False positives*	8	7	10

• All peaks were automatically selected as the target compound to permit automatic identification of target analytes without retention time information.
(* Number of false positives out of 172 screened pesticides.)

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Fig. 3 Result of automatic identification (10 ppb spiked in the green tea leaves)

Conclusion

- Pesticides spiked in all matrices at 10 ppb (10 compounds) could be automatically detected using fast 5 msec MRM with 15 msec polarity switching without retention time information.



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