應用手冊

A New Scientific Information System for Efficient Pesticide Residue Screening at Regulatory Limits in Food Samples

Waters Corporation

This is an Application Brief and does not contain a detailed Experimental section.

Abstract

This application brief demonstrates to accurately and efficiently identify and quantify pesticide residues present at MRL concentrations in a mandarin sample extract. Here we show results generated using the new UNIFI Scientific Information System.

Benefits

The Pesticide Screening Application Solution has been custom built to confidently report and quantify pesticide residues using a streamlined workflow.

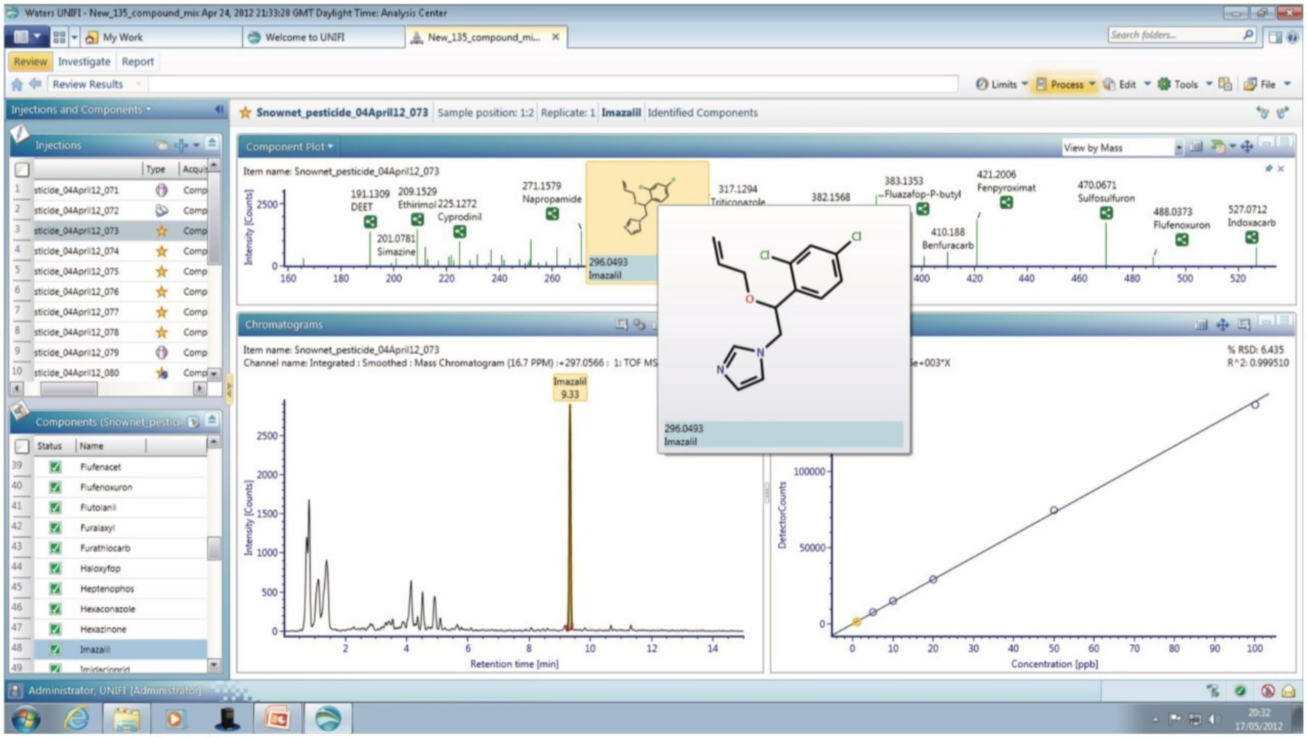
Introduction

Current trends regarding pesticide use indicate that more than 500 compounds are routinely used on a global basis with different countries having varying regulations concerning licensing and legislative limits. With increasing global trade there is a requirement for multi-residue screening strategies capable of efficiently detecting residue violations to ensure consumer safety. Using the Waters Pesticide Screening Application Solution, analysts can meet these challenges in both food and environmental matrices. UNIFI is a revolutionary new scientific information system that enables scientists to test and report more samples for an increasing number of residues and contaminants. UNIFI captures complex UPLC-MS^E data in a single platform and then rapidly processes the data to confirm and quantify the presence of pesticide residues. The Xevo G2-S QTof has high mass accuracy, exceeding the performance criteria specified in SANCO guidelines[1] of 5 ppm. Using a scientific library, that includes molecular formulae, fragment ion and retention time information, we were able to confidently detect low concentrations of pesticide residues following a simple extraction protocol.

Results and Discussion

Mandarin extracts were kindly supplied by WIV-ISP for this study. The chromatographic separation was performed using an ACQUITY UPLC I-Class System with a water/methanol gradient. The data were collected using the Xevo G2-S QTof in MS^E mode, where both precursor ions and fragment ions are acquired in a single injection. Matrix-matched standards were prepared with 116 pesticides to generate a calibration series equivalent to 0.001 to 0.1 mg kg^{-1} in mandarin. Over 100 pesticides were spiked into a blank mandarin extract and a non-targeted analysis was performed against a subset of 479 pesticides from the scientific library.

Processing simultaneously acquired accurate mass precursor and fragment ion (MS^E) data using UNIFI enabled the confirmation of pesticide residues with low false positive rates (≤5%). The enhanced quantitative performance of Tof technology, shown in Figure 1, is clearly demonstrated by the calibration series for imazalil. A correlation coefficient (R^2) of 0.999 was obtained along with a limit of detection of 0.001 mg kg^{-1} in mandarin. Additional functionality provided by UNIFI, including a total component summary, component details, and component plots are also shown. The component information can be easily accessed and displayed in a variety of user-defined views, providing the scientist with added confidence in the results.



	Type	Acqui
1 sticide_04April12_071		Comp
2 sticide_04April12_072		Comp
3 sticide_04April12_073	☆	Comp
4 sticide_04April12_074	☆	Comp
5 sticide_04April12_075	☆	Comp
6 sticide_04April12_076	☆	Comp
7 sticide_04April12_077	☆	Comp
8 sticide_04April12_078	☆	Comp
9 sticide_04April12_079		Comp
10 sticide_04April12_080	☆	Comp

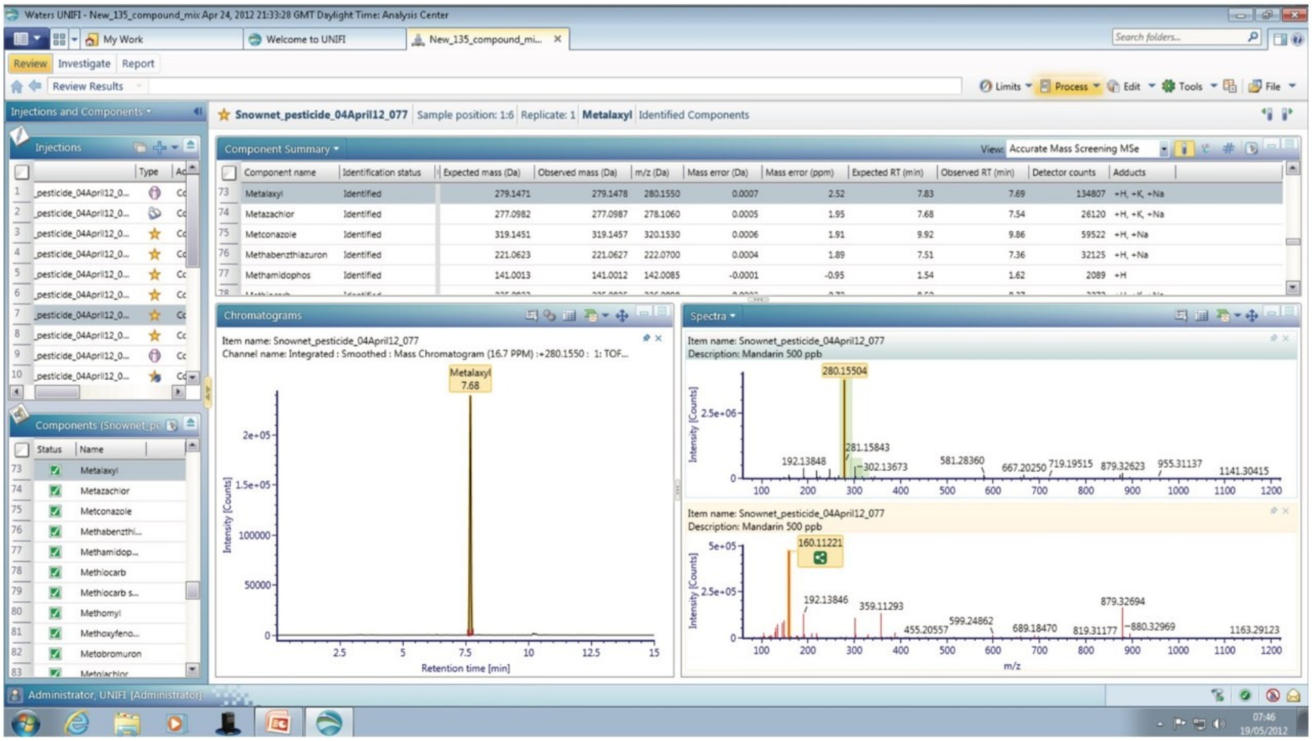
	Status	Name
39	☑	Flufenacet
40	☑	Flufenoxuron
41	☑	Flutolanil
42	☑	Furalaxyl
43	☑	Furathiocarb
44	☑	Haloxyfop
45	☑	Heptenophos
46	☑	Hexaconazole
47	☑	Hexazinone
48	☑	Imazalil
49	☑	Imidacloprid

Item name: Snownet_pesticide_04April12_073

Item name: Snownet_pesticide_04April12_073
Channel name: Integrated : Smoothed : Mass Chromatogram (16.7 PPM) :+297.0566 : 1: TOF MS

Figure 1. A screenshot of the UNIFI Scientific Information System user interface. The component plot (top data panel) shows all the components detected in the mandarin extract; the calibration series for imazalil (0.001 to 0.1 mg kg^{-1}) is shown in the bottom right data panel with correlation coefficient r^2=0.997; and the extracted ion chromatogram for the imazalil calibration standard at 0.001 mg kg^{-1} can be seen in the bottom left data panel.

Analyte specific information that can be used for confirmation purposes, such as molecular structure, adduct formation, and fragmentation, can be generated from the accurate mass (≤2 ppm) data. The MS^E spectra generated for metalaxyl is shown in Figure 2. The component summary view shows all the components identified in the mandarin sample extract. The information-rich MS^E data, in combination with the UNIFI scientific library allow the pesticides to be identified based on accurate mass of both precursor and fragment ions, isotopic pattern, and retention time information. This enables pesticide residues to be confirmed with a high degree of confidence and minimizes false positives.



Snownet_pesticide_04April12_077 | Sample position: 1:6 | Replicate: 1 | **Metalaxyl** | Identified Components

Component Summary — View: Accurate Mass Screening MSe

	Component name	Identification status	Expected mass (Da)	Observed mass (Da)	m/z (Da)	Mass error (Da)	Mass error (ppm)	Expected RT (min)	Observed RT (min)	Detector counts	Adducts
73	Metalaxyl	Identified	279.1471	279.1478	280.1550	0.0007	2.52	7.83	7.69	134807	+H, +K, +Na
74	Metazachlor	Identified	277.0982	277.0987	278.1060	0.0005	1.95	7.68	7.54	26120	+H, +K, +Na
75	Metconazole	Identified	319.1451	319.1457	320.1530	0.0006	1.91	9.92	9.86	59522	+H, +Na
76	Methabenzthiazuron	Identified	221.0623	221.0627	222.0700	0.0004	1.89	7.51	7.36	32125	+H, +Na
77	Methamidophos	Identified	141.0013	141.0012	142.0085	-0.0001	-0.95	1.54	1.62	2089	+H

Item name: Snownet_pesticide_04April12_077
Channel name: Integrated : Smoothed : Mass Chromatogram (16.7 PPM) :+280.1550 : 1: TOF...

Item name: Snownet_pesticide_04April12_077
Description: Mandarin 500 ppb

Item name: Snownet_pesticide_04April12_077
Description: Mandarin 500 ppb

Figure 2. The component summary table (top data panel) shows the components identified and the associated experimental results in the mandarin sample. Illustration of MS^E simultaneously acquired precursor and fragmentation spectra for metalaxyl (bottom right data panel) and the associated extracted ion chromatogram (bottom left data panel).

Conclusion

In this technology brief we have demonstrated the functionality of the new Pesticide Screening Application Solution for routine detection, quantification, and reporting of pesticide residues at the relevant regulatory limits in complex food matrices. The UNIFI Scientific Information System uses the accurate mass of both precursor and fragment ions, isotopic pattern and intensity, and retention time information to reliably detect and confirm pesticide residues while minimizing false positive and negative results (≤5%), in accordance with EU guidelines.

References

1. Method validation and quality control procedures for pesticide residues analysis in food and feed. DOCUMENT No. SANCO/12495/2011.

Featured Products

- ACQUITY UPLC I-Class PLUS System <https://www.waters.com/134613317>
- Xevo G2-XS QTof Quadrupole Time-of-Flight Mass Spectrometry <https://www.waters.com/134798222>
- Pesticide Screening Application Solution with UNIFI <https://www.waters.com/134682906>

720004406, June 2012