응용 자료

Maintaining Mass Accuracy for High Concentration Residue Violations During an HRMS Screening Experiment Using the Waters Pesticide Screening Application Solution with UNIFI

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

Abstract

This application brief demonstrates the ability to maintain mass accuracy on high-level violations for compounds of interest when using the Waters Pesticide Screening Application Solution (PSAS) with UNIFI.

Benefits

The Waters Pesticide Screening Application Solution with UNIFI ensures that mass accuracy is maintained across all peaks, even at high concentrations. This removes the risk of false negatives associated with the presence of high concentration pesticide violations in food and environmental samples.

Introduction

Multi-residue pesticide analysis is challenging due to the low limits of detection required in a diverse range of food commodities. As there are currently well over 1000 pesticides in use, laboratories are under increasing pressure to broaden the range of pesticides determined. The use of high resolution, accurate mass Time-of-flight (Tof) MS shows great potential for this type of analysis for several reasons. In full spectrum mode, Tof instruments are not limited by the number of compounds that can be analyzed in a single run in the way that tandem quadrupoles are. With Tof MS the number of compounds that can be screened is not dependent on the duty cycle of the instrument, but on the chemical compatibility with the extraction and analysis methods. Using a non-targeted, non-data dependant approach to data acquisition (MS^E) allows the user to collect a comprehensive dataset that can be used to screen for a large target list of compounds as well as unexpected compounds. The data can be fully interrogated at a later date for emerging compounds of interest that were not included in the initial screen. In addition to reaching low level MRLs, an HRMS screening must be able to identify residues present at high concentrations since a considerable number of violations in fruits and vegetables are likely to be in the mg/kg range. These high level violations may cause detector saturation which in some cases, can cause a shift in mass accuracy beyond the threshold set for identification.

Data presented in this application brief demonstrates the ability to maintain mass accuracy on high concentration violations for compounds of interest in a screening experiment.

Results and Discussion

A dilution series of the Waters Pesticide Screening Mix (PSM), that contains 20 pesticides of varying polarities and ionizing ability, was prepared in locally filtered tap water. An extension loop on the ACQUITY UPLC I-Class System enabled large volume (100 µL) injections of 9 points ranging from 250 ng/L to 1 mg/L. This resulted in an on-column amount ranging from 0.025 ng to 100 ng.

Tracking mass accuracy across all injections is greatly simplified in the UNIFI Scientific Information System. Customizable views allow the user to display identification details for a specific component across all injections. Figure 1 shows the summary plot available in UNIFI with a line graph depicting the mass accuracy of the compound metolachlor across all injections from 250 ng/L (250 ppt) to 1 mg/L (1,000,000 ppt). Even with an on-column concentration amount of 100 ng, excellent mass accuracy is maintained allowing the software to make a target match within the 5 ppm tolerance set in the analysis method. This level on column would equate to a 10 µL-injection of a 10 mg/L (or 10 ppm) solution, far above the linear dynamic range expected in LC-MS analyses.

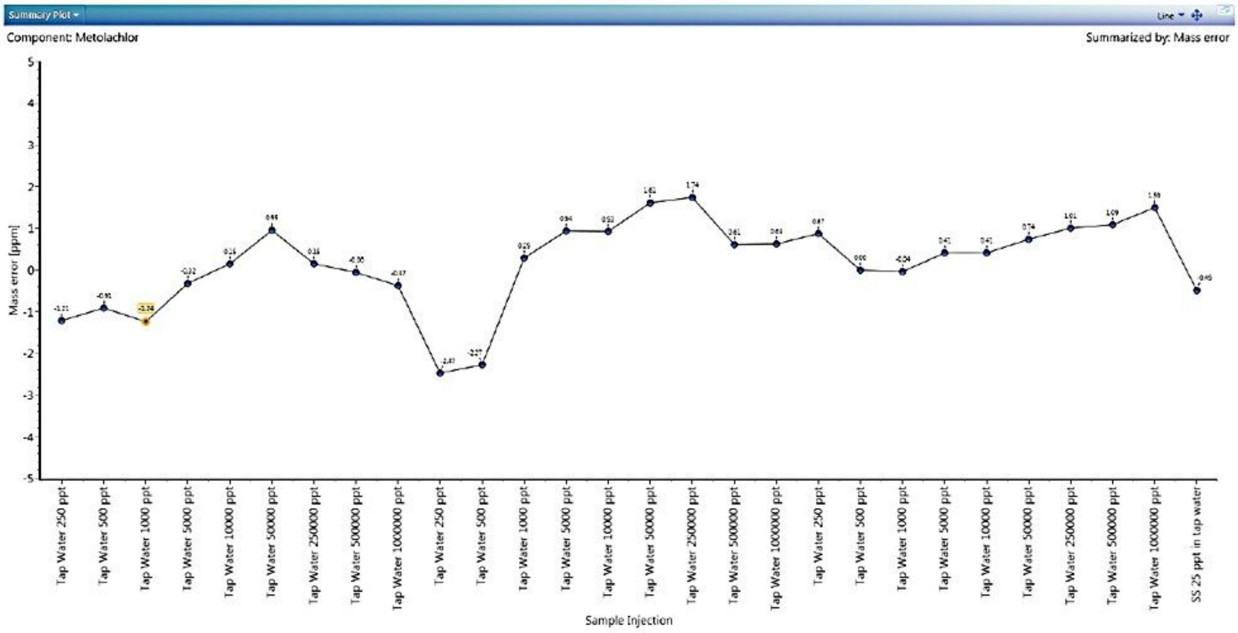
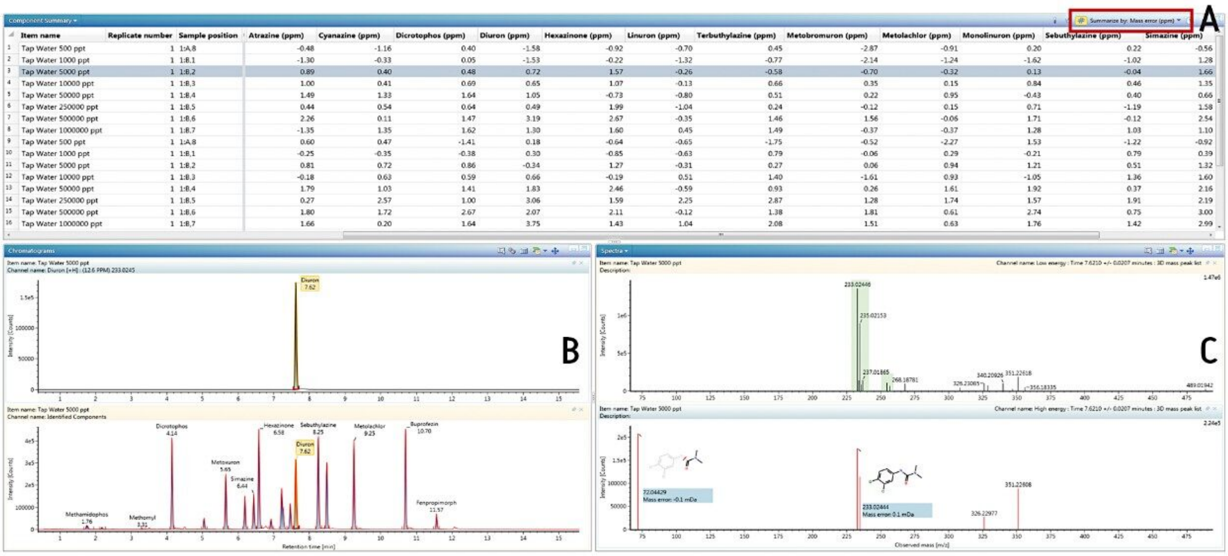


Figure 1. Screen shot of the summary plot available in UNIFI which allows the user to track any result component across all injections via a line or bar plot. The plot above summarizes the mass accuracy of an individual compound (metolachlor) across all levels of spiked tap water injections from 250 ng/L to 1 mg/L.

With a single click, analysts can invoke a pivot table within UNIFI's component summary in order to display the data obtained for all compounds in all injections. Figure 2A shows the display of mass errors across all identified components in all injections. The criteria that can be viewed across all identified components in all injections are easily changed with a click in the highlighted area of Figure 2A. Other criteria that are useful to compare across all injections include the response and observed retention time. Also shown in Figure 2 is the extracted ion chromatogram of the highlighted level for diuron (upper chromatogram in Figure 2B) and an overlay of the extracted ion chromatograms for all identified components (lower chromatogram in Figure 2B). Figure 2C shows the corresponding low energy and high energy spectra for diuron.

Maintaining mass accuracy for high-level pesticide violations in food and environmental samples avoids false negatives and gives the user confidence in the identifications returned from a routine screening experiment.



	Item name	Replicate number	Sample position	Atrazine (ppm)	Cyanazine (ppm)	Dicrotophos (ppm)	Diuron (ppm)	Hexazinone (ppm)	Linuron (ppm)	Terbuthylazine (ppm)	Metobromuron (ppm)	Metolachlor (ppm)	Monolinuron (ppm)	Sebuthylazine (ppm)	Simazine (ppm)
1	Tap Water 500 ppt	1	1:A,8	-0.48	-1.16	0.40	-1.58	-0.92	-0.70	0.45	-2.87	-0.91	0.20	0.22	-0.56
2	Tap Water 1000 ppt	1	1:B,1	-1.30	-0.33	0.05	-1.53	-0.22	-1.32	-0.77	-2.14	-1.24	-1.62	-1.02	1.28
3	Tap Water 5000 ppt	1	1:B,2	0.89	0.40	0.48	0.72	1.57	-0.26	-0.58	-0.70	-0.32	0.13	-0.04	1.66
4	Tap Water 10000 ppt	1	1:B,3	1.00	0.41	0.69	0.65	1.07	-0.13	0.66	0.35	0.15	0.84	0.46	1.35
5	Tap Water 50000 ppt	1	1:B,4	1.49	1.33	1.64	1.05	-0.73	-0.80	0.51	0.22	0.95	-0.43	0.40	0.66
6	Tap Water 250000 ppt	1	1:B,5	0.44	0.54	0.64	0.49	1.99	-1.04	0.24	-0.12	0.15	0.71	-1.19	1.58
7	Tap Water 500000 ppt	1	1:B,6	2.26	0.11	1.47	3.19	2.67	-0.35	1.46	1.56	-0.06	1.71	-0.12	2.54
8	Tap Water 1000000 ppt	1	1:B,7	-1.35	1.35	1.62	1.30	1.60	0.45	1.49	-0.37	-0.37	1.28	1.03	1.10
9	Tap Water 500 ppt	1	1:A,8	0.60	0.47	-1.41	0.18	-0.64	-0.65	-1.75	-0.52	-2.27	1.53	-1.22	-0.92
10	Tap Water 1000 ppt	1	1:B,1	-0.25	-0.35	-0.38	0.30	-0.85	-0.63	0.79	-0.06	0.29	-0.21	0.79	0.39
11	Tap Water 5000 ppt	1	1:B,2	0.81	0.72	0.86	-0.34	1.27	-0.31	0.27	0.06	0.94	1.21	0.51	1.32
12	Tap Water 10000 ppt	1	1:B,3	-0.18	0.63	0.59	0.66	-0.19	0.51	1.40	-1.61	0.93	-1.05	1.36	1.60
13	Tap Water 50000 ppt	1	1:B,4	1.79	1.03	1.41	1.83	2.46	-0.59	0.93	0.26	1.61	1.92	0.37	2.16
14	Tap Water 250000 ppt	1	1:B,5	0.27	2.57	1.00	3.06	1.59	2.25	2.87	1.28	1.74	1.57	1.91	2.19
15	Tap Water 500000 ppt	1	1:B,6	1.80	1.72	2.67	2.07	2.11	-0.12	1.38	1.81	0.61	2.74	0.75	3.00
16	Tap Water 1000000 ppt	1	1:B,7	1.66	0.20	1.64	3.75	1.43	1.04	2.08	1.51	0.63	1.76	1.42	2.99

Figure 2. Screen shot showing the results from injection of increasing concentrations of the Waters pesticide screening mix (PSM) spiked in locally filtered tap water. The component summary section (2A) shows a table displaying the mass accuracy (in ppm) of 15 compounds over replicate injections of nine calibration levels. The parameter displayed can easily be changed to another parameter (for example the observed retention time) by clicking in the area highlighted in the red box. The chromatogram section (2B) shows an XIC for all compounds identified from the PSM as well as an XIC for the highlighted compound diuron. Low energy and high energy spectra for the selected component are displayed in the spectra window (2C).

Conclusion

The example presented demonstrates the preservation of mass accuracy, even for compounds present at concentrations well beyond the linear dynamic range of MS systems. This is critical to ensure that high concentration level violations of compounds are not missed in a screening experiment.

Featured Products

Pesticide Screening Application Solution with UNIFI <https://www.waters.com/134682906>

ACQUITY UPLC I-Class PLUS System <https://www.waters.com/134613317>

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