MS Data Processing With Full Flexibility Of Universal Processing Software.

Frans Schoutsen¹, Barbara van Cann¹, Vincent Jespers¹, Michal Godula² ¹Thermo Fisher Scientific, Breda, The Netherlands, ²Thermo Fisher Scientific, Prague, Czech Republic

Overview

Liquid Chromatography (LC) in combination with triple quadrupole Mass Spectrometry (MS) is more and more a common analysis technique in routine laboratories. Especially when using MS/MS selected reaction monitoring (SRM) for a large number of components data handling can be a bottle neck. This poster will show MS data processing of pesticides in water samples using an already established chromatography data system which is now also capable of processing large quantities of MS data.

Introduction

FIGURE 3. SRM Acquisition Table.

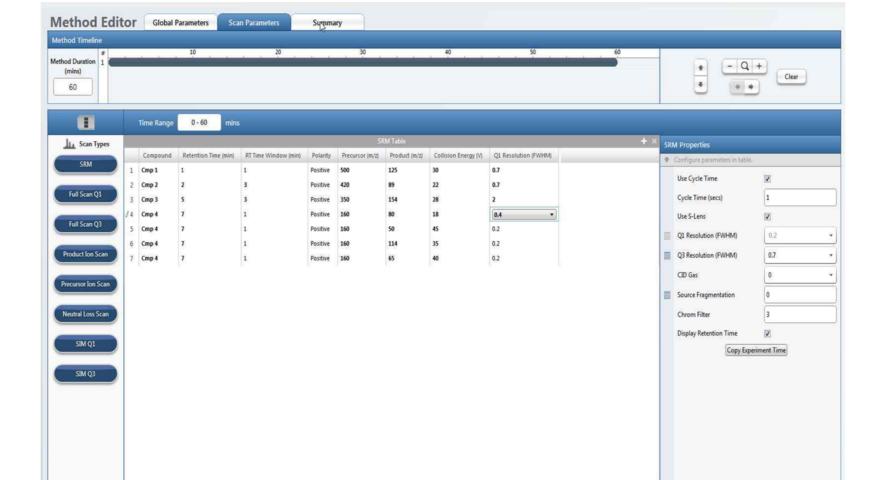
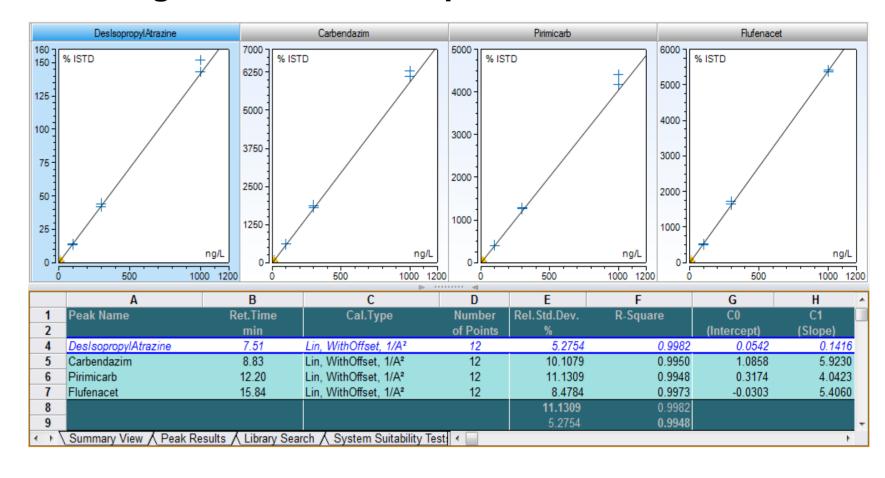


FIGURE 6. Calibration plots of selected components, including calibration curve parameters.



In routine practice, screening of pesticide residues in a large variety of fruit and vegetable samples is carried out by a number of different analytical techniques, LC-MS/MS being one of the routine approach. When considering the throughput of the lab, data processing is an important part of the total workflow of any multi-method covering up to hundreds of compounds.

Traditional data analysis with triple quadrupole MS/MS analysis is based on evaluation of multiple SRM experiments whereby a number of known contaminants is measured and quantified using available standards. With increasing number of compounds to be screened, samples to be measured, and data to be reported as fast as possible, data analysis can be optimized and automated.

Methods

Blanks, calibration standards, QC standards and unknown samples are injected via conventional reversed phase chromatography with a gradient elution, coupled to the latest state of the art Triple Quadrupole MS.

Liquid Chromatography

Thermo Scientific[™] Dionex[™] UltiMate[™] 3000 RSLC system.

Data Analysis

Data analysis was performed with Thermo Scientific[™] Dionex[™] Chromeleon[™] Chromatography Data System (CDS) software.

Results

Data Evaluation

All data evaluation is performed in the Chromatography Studio (Figure 4). Chromeleon CDS operates with the principle of Dynamic Data Processing. This ensures the selections and changes made are instantly reflected in the data and results. For example, when selecting one or more components and/or injections in the navigation pane (Figure 4A), the component traces (Figure 4B) and corresponding results (Figure 4C) are shown. All changes are automatically applied to all injections in the sequence.

FIGURE 4. Chromatography Studio showing Navigation Pane (A), MS Component Traces (B) and Interactive Results Tables (C).

The amounts in all standards and samples were calculated based on the calibration results. The recovery of the standards compared to the theoretical amount (Figure 7).

FIGURE 7. Overview of confirmation ratios and actual and theoretical amounts of flufenacet in all calibration and QC standards.

Stand	ard Recovery	Flufenacet					
No. Injection Name		Conf. Ratio	Amount	Theoretical Amount	Deviation		
		%	ng/L	ng/L	%		
2	Cal Std 1	0.83	0.94	1.00	-5.91		
3	Cal Std 1	0.78	1.07	1.00	6.86		
4	Cal Std 2	0.84	2.80	3.00	-6.74		
5	Cal Std 2	0.74	3.10	3.00	3.48		
6	Cal Std 3	0.74	9.85	10.00	-1.49		
7	Cal Std 3	0.72	10.33	10.00	3.31		
8	Cal Std 4	0.80	98.22	100.00	-1.78		
9	Cal Std 4	0.81	95.12	100.00	-4.88		
10	Cal Std 5	0.79	318.86	300.00	6.29		
11	Cal Std 5	0.80	304.52	300.00	1.51		
12	Cal Std 6	0.78	992.79	1000.00	-0.72		
13	Cal Std 6	0.78	1000.75	1000.00	0.07		
14	QC	0.81	28.75	30.00	-4.17		
15	QC	0.80	29.93	30.00	-0.22		
16	QC	0.81	30.91	30.00	3.05		

FIGURE 8. Calculated ion ratios, amounts and repeatability in Sample 1.

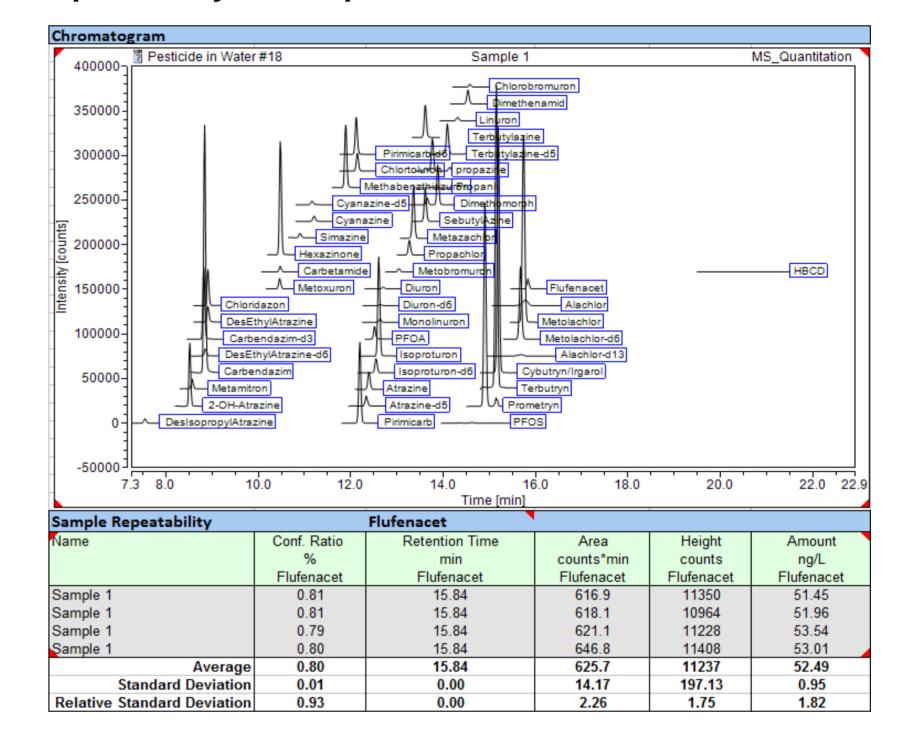


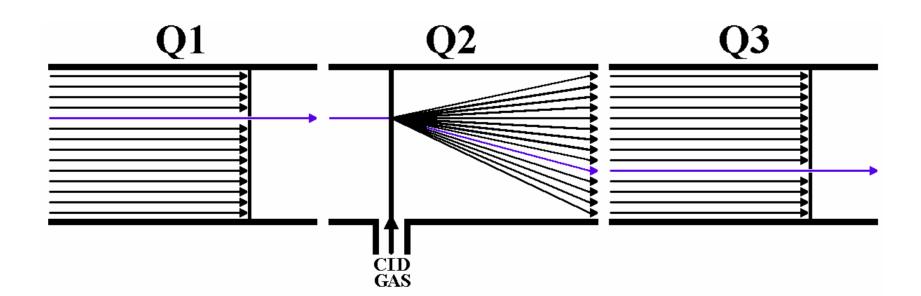
FIGURE 1. UltiMate 3000 RSLC System and TSQ Quantiva Triple Quadrupole MS.



Mass Spectrometry

MS analysis was performed with a Thermo Scientific[™] TSQ Quantiva[™] Triple Quadrupole MS with AIM Technology.

FIGURE 2. Schematic Overview of Triple Quadrupole LC-MS/MS.







Detection parameters and component settings (Figure 5) in the Processing Method are applied to all component traces.

FIGURE 5. Comprehensive Component Table, including Quantitation and Confirming peaks (A), Calibration settings (B) and Calibration levels (C)

Dete	ection MS Detection	n MS Comp	onent Table	Calibration MS S	Settings MS Library	Screening S	ST/IRC Advanced Settings	Peptide Table Compo	site Scoring						
Component Table															
Group Area Drag a column header here to group by that column. <u>Run Component Table Wizard.</u> , <u>Show Properties</u>															
#	Name	Ret.Time 🔺	Window	MS Quantitation Peak	MS Confirming Peak 1	Extraction Time	Stand.Meth.	Cal.Type	Level "01"	Level "02"	Level "03"	Level "04"	Level "05"	Level "06"	1
27	Diuron	12.700	0.400 AN	233.250 / 72.150	233.250 / 46.000	RT	Internal Diuron-d6	Lin, WithOffset, 1/A²	1.000000	3.000000	10.000000	100.000000	300.000000	1000.000000	30
28	Metobromuron	13.050	0.300 AN	259.100 / 169.993	259.100/218.148	RT	Internal Diuron-d6	Lin, WithOffset, 1/A ²	1.000000	3.000000	10.000000	100.000000	300.000000	1000.000000	30
29	Propachlor	13.300	0.300 AN	212.100 / 170.081	212.100 / 94.100	RT	Internal Diuron-d6	Lin, WithOffset, 1/A ²	1.000000	3.000000	10.000000	100.000000	300.000000	1000.000000	30
30	Metazachlor	13.370	0.300 AN	278.100 / 134.18	278.100 / 210.059	RT	Internal Diuron-d6	Lin, WithOffset, 1/A ²	1.000000	3.000000	10.000000	100.000000	300.000000	1000.000000	30
31	SebutyIAzine	13.630	0.300 AN	230.100 / 174.2 0		RT	Internal Diuron-d6	Lin, WithOffset, 1/A ²	1.000000	3.000000	10.000000	100.000000	300.000000	1000.000000	30
32	Dimethomorph	13.750	0.500 AG	388.350 / 301.066	388.350 / 165.161	RT	Internal Diuron-d6	Lin, WithOffset, 1/A ²	1.000000	3.000000	10.000000	100.000000	300.000000	1000.000000	30
33	Propanil	13.800	0.400 AN	218.000 / 162.000		RT	Internal Diuron-d6	Lin, WithOffset, 1/A ²	1.000000	3.000000	10.000000	100.000000	300.000000	1000.000000	30
34	propazine	13.800	0.400 AN	230.100 / 146.100		RT	Internal Diuron-d6	Lin, WithOffset, 1/A ²	1.000000	3.000000	10.000000	100.000000	300.000000	1000.000000	30
35	Terbutylazine-d5	14.100	0.400 AN	235.000 / 179.100		RT	ISTD Internal	Lin, WithOffset, 1/A ²	1.000000	3.000000	10.000000	100.000000	300.000000	1000.000000	30
36	Terbutylazine	14.150	0.400 AN	230.100 / 174.200		RT	Internal Terbutylazine-d5	Lin, WithOffset, 1/A ²	1.000000	3.000000	10.000000	100.000000	300.000000	1000.000000	30
37	Linuron	14.320	0.400 AN	249.000 / 160.128	249.000 / 182.060	RT	Internal Terbutylazine-d5	Lin, WithOffset, 1/A ²	1.000000	3.000000	10.000000	100.000000	300.000000	1000.000000	30
38	Dimethenamid	14.550	0.400 AN	276.100 / 168.112	276.100 / 244.106	RT	Internal Terbutylazine-d5	Lin, WithOffset, 1/A ²	1.000000	3.000000	10.000000	100.000000	300.000000	1000.000000	30
39	Chlorobromuron	14.600	0.400 AN	292.900 / 203.900	292.900 / 182.150	RT	Internal Terbutylazine-d5	Lin, WithOffset, 1/A ²	1.000000	3.000000	10.000000	100.000000	300.000000	1000.000000	30
40	PFOS	14.700	0.750 AG	499.100 / 80.100	499.100 / 99.087	RT	External	Lin, WithOffset, 1/A ²	1.000000	3.000000	10.000000	100.000000	300.000000	1000.000000	30
41	Prometryn	14.900	0.400 AN	242.100 / 158.150	242.100 / 200.250	RT	Internal Terbutylazine-d5	Lin, WithOffset, 1/A ²	1.000000	3.000000	10.000000	100.000000	300.000000	1000.000000	30
42	Terbutryn	15.200	0.400 AN	242.100 / 186.050	242.100 / 91.050	RT	Internal Terbutylazine-d5	Lin, WithOffset, 1/A ²	1.000000	3.000000	10.000000	100.000000	300.000000	1000.000000	30
43	Cybutryn/Irgarol	15.200	0.400 AN	254.200 / 198.139	254.200 / 83.136	RT	Internal Terbutylazine-d5	Lin, WithOffset, 1/A²	1.000000	3.000000	10.000000	100.000000	300.000000	1000.000000	30
44	Alachlor-d13	15.700	0.750 AN	283.250 / 251.200		RT	ISTD Internal	Lin, WithOffset, 1/A ²	1.000000	3.000000	10.000000	100.000000	300.000000	1000.000000	30
45	Metolachlor-d6	15.700	0.400 AN	290.250 / 258.150		RT	ISTD Internal	Lin, WithOffset, 1/A²	1.000000	3.000000	10.000000	100.000000	300.000000	1000.000000	30
46	Metolachlor	15.750	0.300 AN	284.100 / 252.197	284.100 / 176.141	RT	Internal Metolachlor-d6	Lin, WithOffset, 1/A²	1.000000	3.000000	10.000000	100.000000	300.000000	1000.000000	30
47	Alachlor	15.800	0.750 AN	270.100 / 238.191		RT	Internal Alachlor-d13	Lin, WithOffset, 1/A ²	1.000000	3.000000	10.000000	100.000000	300.000000	1000.000000	30
48	Flufenacet	15.850	0.400 AN	364.100 / 152.141	364.100 / 194.087	RT	Internal Alachlor-d13	Lin, WithOffset, 1/A²	1.000000	3.000000	10.000000	100.000000	300.000000	1000.000000	30
1	HRCD	20.500	1.000 AG	640 700 / 81 115	640 700 / 78 970	RT	Internal DeeFthvIAtrazine-d6	Lin WithOffeet 1//2	1.000000	3.000000	10.000000	100.000000	300.00000	1000.000000	20

Conclusion

To process the data with MS detection several parameters have to be entered depending on the experiment and settings. Here we demonstrated that by selecting the appropriate SRM transition data can be quantified on one of the SRM transitions, usually the most intense fragment. The other fragment can be used to confirm the compound detected. Calculation of concentration is as simple as with other detection techniques, where the area is compared to concentration with or without an internal standard.

The objective of the work was to demonstrate the use of Chromeleon CDS with the latest triple quadrupole. The processing of MS data in Chromeleon CDS is handled the same as conventional chromatography data. Dynamic Data Processing ensures the selections and changes made are instantly reflected in the data and results, saving huge amounts of data processing time. Data processing was complete and fast in an intuitive environment.

Parameters

Source	HESI
Polarity	Positive
ISV	3500V
Sheath gas	40 arb units
Aux	15 arb units
Vaporizer	280 °C
Transfer	350 °C

Experiment

SRM transitions Resolution Q1 and Q3 Cycle time

Quantitation and Confirmation unit mass resolution 0.4 sec

Quantitation

After injection of calibration standards, curves were calculated as defined in the processing Method (Figure 5B and C), using the Chromeleon CDS algorithm (Figure 6). Peaks (quantitation and confirming), representing the components in the SRM trace were integrated automatically, using the Cobra[™] peak detection algorithm. List all non-Thermo trademarks and registered trademarks that appear in the poster. Examples include TMT, SEQUEST, ActiveX, Eksignet, Mascot. Follow this with: All other trademarks are the property of Thermo Fisher Scientific and its subsidiaries.

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