GC-MS/MS Pesticide Analyser Workflow Updated and Enhanced By Powerful Thermo Scientific[™] Chromeleon[™] 7.2 CDS Software

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Introduction

Triple quadrupole mass spectrometers such as the Thermo Scientific™ TSQ™ 8000 Evo GC-MS/MS system have gained popularity over their single quadrupole counterparts because of their high selectivity and lower detection limits, especially in complex matrices such as those encountered in pesticide analysis in food. In this poster we present results of GC-MS/MS analysis of pesticides using timed-Selected Reaction Monitoring (t-SRM). The t-SRM optimized dwell times combined with the Enhanced Velocity Optics (EvoCell collision cell) present in the TSQ 8000 Evo enables us to monitor multiple transitions per analyte for a more confident identification without compromising quantitation sensitivity. The results we show were obtained using Thermo Scientific[™] Dionex[™] Chromeleon[™] 7.2 CDS software, which combines powerful data analysis capability with easy pesticide analysis method creation. The Chromeleon software pesticide analyzer database contains retention times and transitions for over 1000 pesticides and other compounds of environmental interest. Historically, developing MS/MS transitions for compounds used to be arduous and time-consuming process prone to operator error, but this no longer is the case. This poster highlights the power of AutoSRM which is a tool for developing and optimizing transitions for compounds that are not yet present in the database with simple user interaction and high degree of confidence in the results.

Method

Hardware- Thermo Scientific TSQ 8000 Evo GC-MS/MS with TRACE $^{\rm TM}$ 1310 GC coupled with Al/AS 1310 autosampler.

Software- Thermo Scientific Dionex Chromeleon 7.2 CDS software

Sample- QuEChERS extracted lettuce matrix in 1:1 Ethyl acetate/cyclohexane spiked with Restek[®] GC Multiresidue Pesticide kit (Cat.# 32562) containing nine vials of standards (#1 to #9).

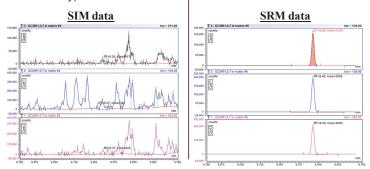
Methodology- The injector, column and the GC and MS run conditions are same as that recommended in the Thermo Scientific[™] TSQ[™] 8000 Evo Pesticide Analyzer guide. The Pesticide analyzer also contains a compound database (CDB) having 1001 compounds with expected retention time (for the given conditions) as well as fully optimized SRM transitions for at least 3 ions per compound.

FIGURE 1. Full scan chromatogram of 10 pg/µL GC MR #1,5,7 spiked in lettuce matrix. We see a number of extracted lettuce compounds that could possibly interfere with pesticides.



Why Triple Quad (SRM) not Single Quad (SIM)?

Below is an example of Etridiazol (at 10 $pg/\mu L$) SIM data and SRM data. We see no matrix interferences with SRM data (high selectivity).





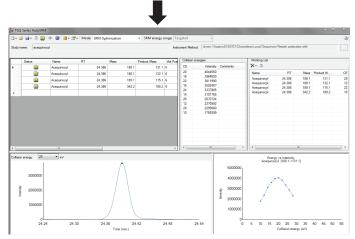


Chromeleon Pesticide Analyzer Workflow

Select compounds from the Compound Database (CDB)

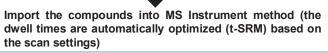
Data Sc						
ampau	nd Data Store Path: C:\User	s\amit.gujar\Documents\P	esticide analyzer\TSQ8000 F	esticides. En	Contaminants, PC	Browse
) rag a c	column header here to group by:	that column.				
	Name	Experiment Type	Category	Ionization	Chemical Formula	CA
•	1,2,3,4-Tetrachlorobenzene	SRM	SVOC	EI	C6H2Cl4	634-66-2
•	1.2,3,5-Tetrachlorobenzene	SRM	SVOC	EI	C6H2C14	634-90-2
•	1.2.3-Trichlorobenzene	SRM	SVOC	EI	C6H3CI3	87-61-6
•	1,2,4-Trichlorobenzene	SRM	SVOC	EI	C6H3CI3	120-82-1
•	1,2-Dichlorobenzene	SRM	SVOC	EI	C6H4C12	95-50-1
•	1.2-Dinitrobenzene	SRM	SVOC	EI	C6H4N2O4	528-29-0
•	1.3.5-Tribromobenzene	SRM	SVOC	EI	C6H3Br3	626-39-1
•	1,3,5-Trichlorobenzene	SRM	SVOC	EI	C6H3CI3	108-70-3
•	1,3-Dichlorobenzene	SRM	SVOC	EI	C6H4CI2	541-73-1
•	1.3-Dinitrobenzene	SRM	SVOC	EI	C6H4N2O4	99-65-0
•	1.4-Dichlorobenzene	SRM	SVOC	EI	C6H4C12	106-46-7
•	1,4-Dichlorobenzene-d4	SRM	Internal Standard (SVOC)	EI	C6CI2D4	3855-82-
•	1,4-Dinitrobenzene	SRM	SVOC	EI	C6H4N2O4	100-25-4
•	1-Methylnophtholene	SRM	PAH	EI	C11H10	90-12-0
•	2,3,4,6-Tetrachlorophenol	SRM	SVOC	EI	C6H2CI4O	58-90-2
	A DE C Terresteres illes	0014	Disad days and doat	F 1	00100140	2401.00
Select	All Unselect All Exp	and All Collapse Al			Import	Cancel

For compounds not in the CDB perform AutoSRM



AutoSRM has 3 simple one-click "Auto Process" steps -

- 1. Select precursor ions (or import SIM data)
- 2. Select product ions
- 3. Choose optimum collision energy

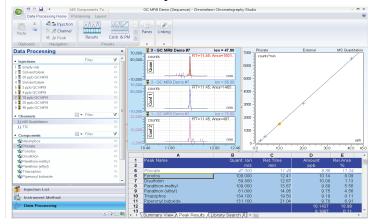


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Method Setup							
Scans							
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Name	RT	Ion Polarity	Mass	Product Mass	Collision Energy	^	Time summary Resulting total scan time: 0.300 sec
Methabenzthiazuror	n 11.21	Positive	136.1	109	18		SBM/SIM Time: 0.300 sec
Methabenzthiazuror	n 11.21	Positive	164.1	108	28		
Methabenzthiazuror	n 11.21	Positive	164.1	135.8	10		Lowest dwell time; D.DD1 sec
Bromoxynil	11.27	Positive	116.7	62	16		Window optimization
Bromoxynil	11.27	Positive	276.8	88	30		V Optimize
Bromoxynil	11.27	Positive	276.8	170	18		Desired min dwell time: 10.0 🚔 ms
Cadusafos	11.28	Positive	159	96.9	16		Desired window 0.6 🗘 mi
Cadusafos	11.28	Positive	159	130.9	8		
Cadusafos	11.28	Positive	213	89.1	12		Minimum window 0.3 🐑 mi
Promecarb	11.30	Positive	135.1	91	16		Peak width
Promecarb	11.30	Positive	150.1	91.1	24		Min. baseline peak width: 30 🛊 sec
Promecarb	11.30	Positive	150.1	135.1	10		Desired scens per peak 10 -
Tebutam	11.32	Positive	91	39	28		
Tebutam	11.32	Positive	91	65	16		SRM Resolution
Tebutam	11.32	Positive	190.1	57.1	10		Set resolution for each unique transition scan
Pencycuron	11.36	Positive	125	62.8	28		Precursor (Q1): Normal -
Pencycuron	11.36	Positive	125	89	16		Product (G3): Normal
Pencycuron	11.36	Positive	125	99	16		Acquisition options
Diallate-cis	11.36	Positive	234.1	150	18		Allow for asymmetric acquisition windows
Diallate-cis	11.36	Positive	235.8	152	18		Allow dwell time prioritization High priority m
Diallate-cis	11.36	Positive	235.8	194	12		Full scan
Phorate	11.37	Positive	75	47	8		Use full scan with mass range: 50-550
Phorate	11.37	Positive	121	65	8		Fullscen 0.083 sec Positive +

Start your sequence



Check your results



Results

Screening studies- Restek[®] GC MR #1 to #9 were spiked at 10 pg/ μ L levels in lettuce matrix. The whole Pesticide analyzer method for screening 1001 compounds was run on this sample. Out of the 203 compounds, 198 were detected at this trace level.

FIGURE 2. MS Quantitation chromatogram for 10 pg/µL of GC MR #1 to 9 spiked in lettuce extract.



Quantitative studies- Analytical performance of the instrument was determined by plotting the calibration curves and looking at repeatability at low concentrations for GC MR #9 mix. The limits of detection (LoD) were then determined by-

 $LoD = 2.998 \times Amount \ on \ column \times \% RSD$

FIGURE 3. Calibration curves for Restek® GC Multiresidue Pesticide Standard #9 in lettuce matrix; 2-40 pg/µL . The average R^2 was 0.9994.

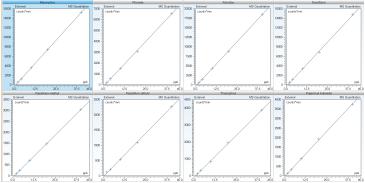
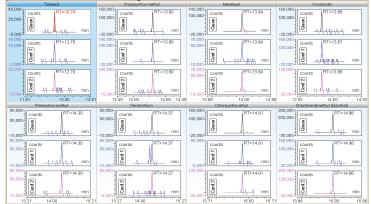


Table 1. %RSDs and LoDs for Restek[®] GC Multi-residue Pesticide Standard #9 in lettuce matrix (8 consecutive injections of 2 $pg/\mu L$)

Compound	%RSD	LoD, fg on-column		
Mevinphos	11.4	0.68		
Phorate	5.4	0.33		
Fonofos	7.3	0.44		
Disulfoton	8.6	0.51		
Parathion-methyl	8.4	0.50		
Parathion (ethyl)	10.1	0.61		
Triazophos	12.4	0.74		
Piperonyl butoxide	9.7	0.58		

A similar study was done for a 60 pesticide mix of GC MR #1,5 and 7. Shown below are selected compounds with their quantitation and confirming ions.



Conclusion

- The high selectivity offered by GC-MS/MS is critical in positively identifying pesticides in complex food matrices.
- Chromeleon 7.2 and AutoSRM provides an easy workflow for GC-MS/MS pesticide analysis with single platform for lab-wide instrument control.
- The TSQ 8000 Evo GC-MS/MS with timed-SRM and EvoCell provides superior detection limits, even in the most challenging samples.

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