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Pesticides Analysis Focused on Sample Preparation by QUECHERS

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Technical Sales Manager GCMS
Emerging Markets

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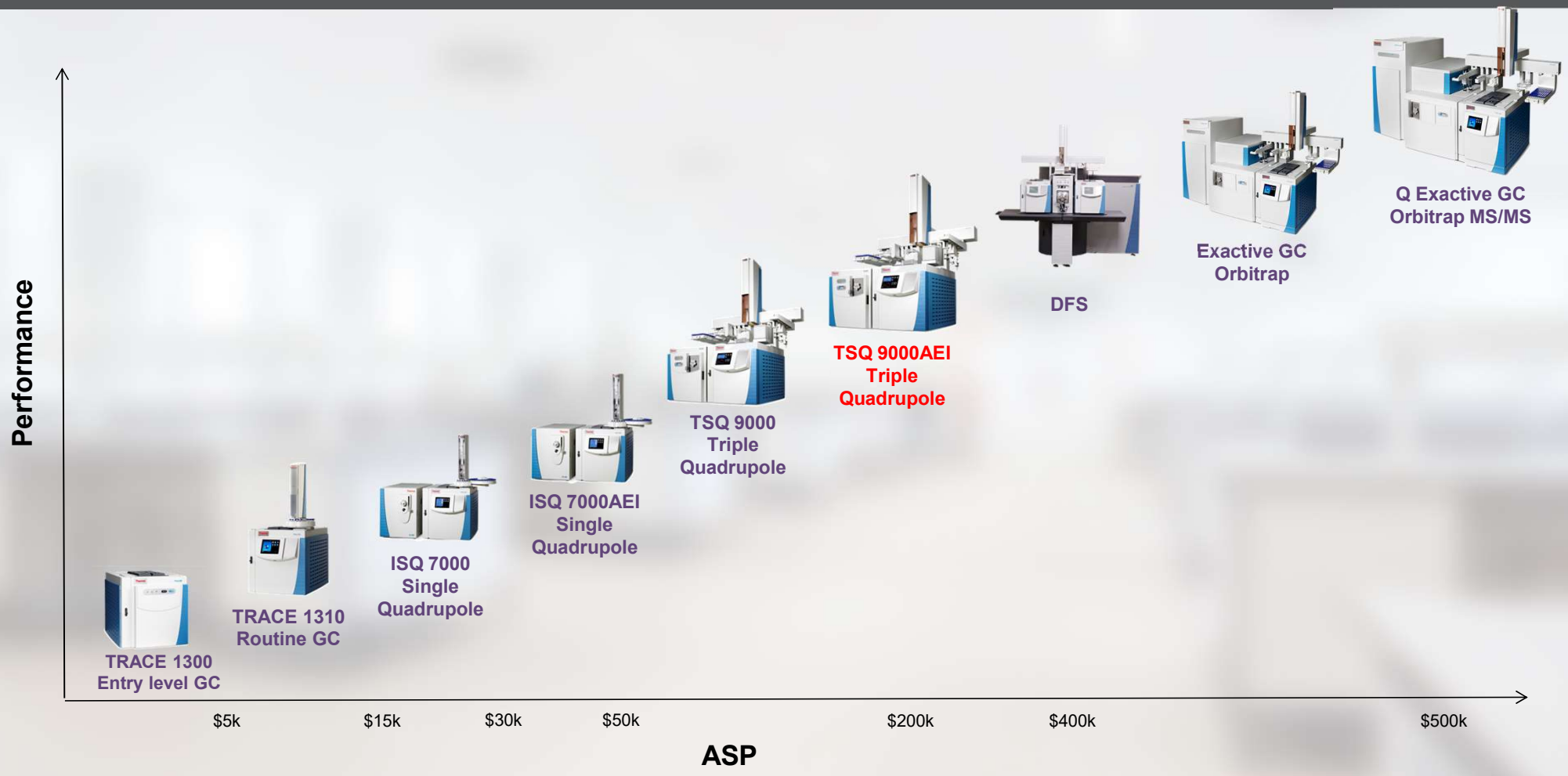
Now silent…….

You are muted
The session is going to be recorded

GC&GCMS - 2020 NEW Product Pipeline



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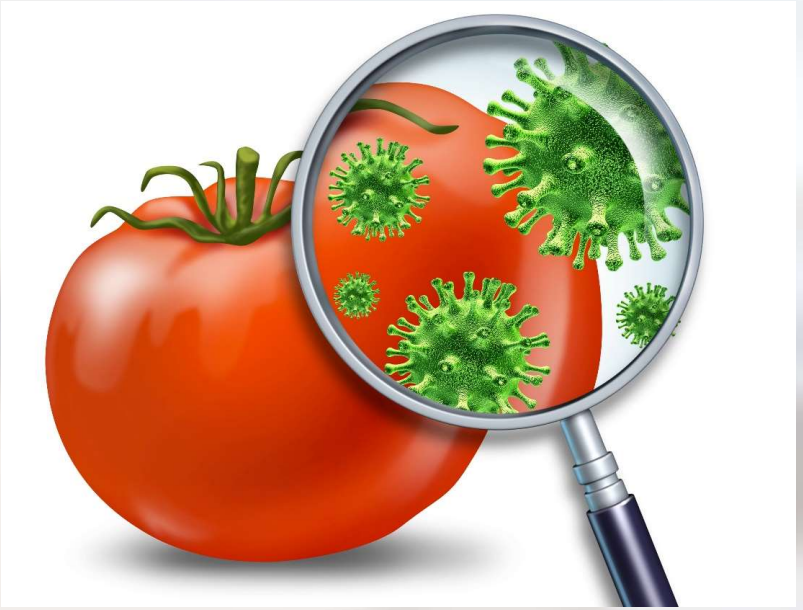
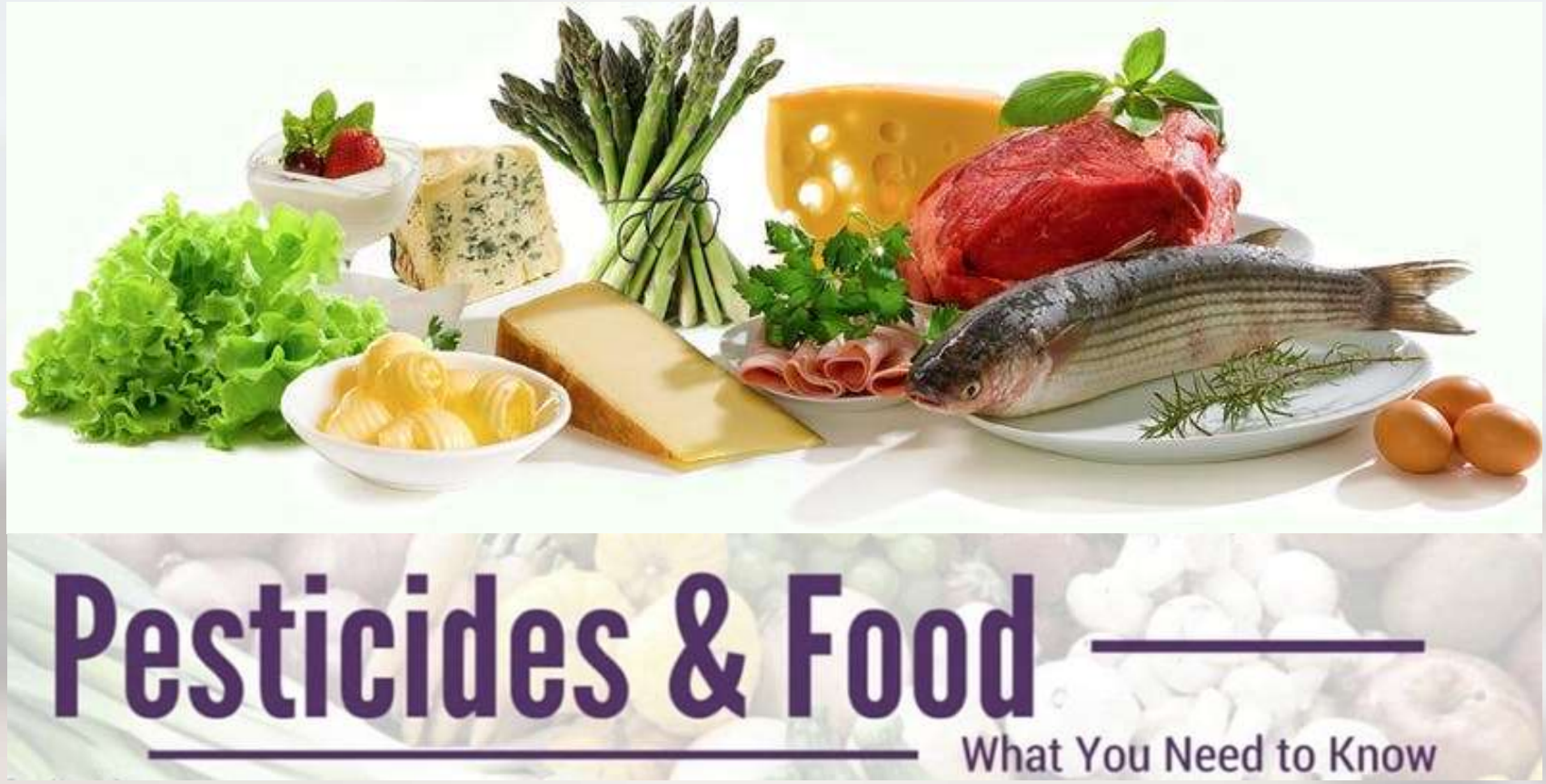
Agenda

- Pesticides & Food: What You Need to Know
- Description
- Workflows
- Sample preparation Pesticide Analysis
- Pesticide Analysis by TSQ 9000 AEI
- Results
- Summary



Pesticides & Food: What You Need to Know



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Pesticide Chemistry

The term "**-cide**" comes from the Latin word "**to kill**."

- **Organophosphate**
- **Carbamate**
- **Organochlorine**
- **Pyrethroid**
- **Sulfonylurea**
- **Biopesticides**

- **Herbicides**
- **Bactericides**
- **Fungicides**
- **Insecticides**
- **etc...**

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TSQ 9000 GC-MS/MS– Flexible purchase options which are scalable in the field

IDL

* *SRM Instrument detection limit* ***verified at installation*** *(5 fg OFN injected n=8, 99% confidence)*

< 4 fg	< 2 fg		< 0.4 fg
				
TSQ9K-MTNOVPI ExtractaBrite EI 240l pump	**TSQ9K-NOVPI ExtractaBrite EI 300l pump**	**TSQ9K-VPI NeverVent ExtractaBrite EI 300l pump**	**TSQ9K-VPICI NeverVent ExtractaBrite EI & CI 300l pump**	**TSQ9K-AEI Advanced EI 300l pump**
Most accesible entry from SQ>TQ	Best price to performance ratio	High-throughput solution with NeverVent Technology	High-throughput EI/CI solution with NeverVent technology	Ultra high perfromance and robustness with Advanced EI technology

Full field upgrade path for TSQ 9000 configurations

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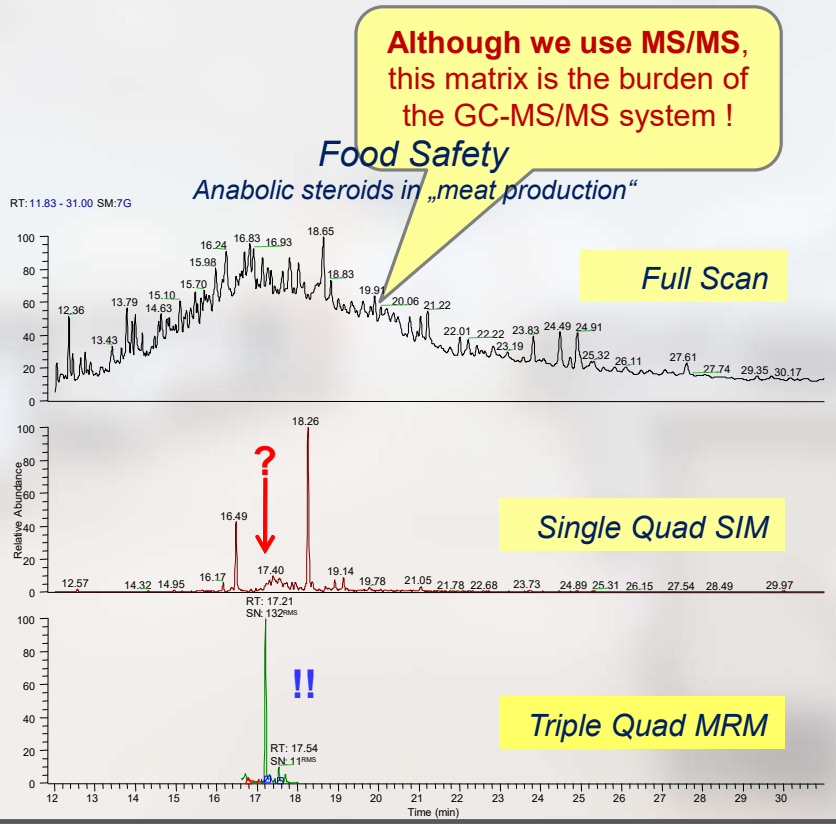
Outline

- Why do we need MS/MS?
- How do I deal with the sample matrix?
  - Eliminating the sample matrix increases efficiency and productivity
  - Chromatography - PTV Backflush
  - Mass Spectrometer – SRM and H-SRM
- MS/MS for positive identification
- Method set-up for productivity
  - Timed SRM method - Unique way to maximize sensitivity and productivity
- The POPs excellence center

Strong Analytical Challenges for GC-MS/MS

- Multi-methods => More compounds - Shorter sample prep
- Increased Burden to the GCMS-System
  - On injection system and analytical column
  - On mass spectrometer
- Excellent System Performance
  - Keeping high sensitivity
  - Keeping high precision
  - Keeping high productivity
- **Solutions are Addressing**
  1. **GC Injection:**
     - PTV BKF operation
  2. **Mass Spectrometer:**
     - Matrix elimination



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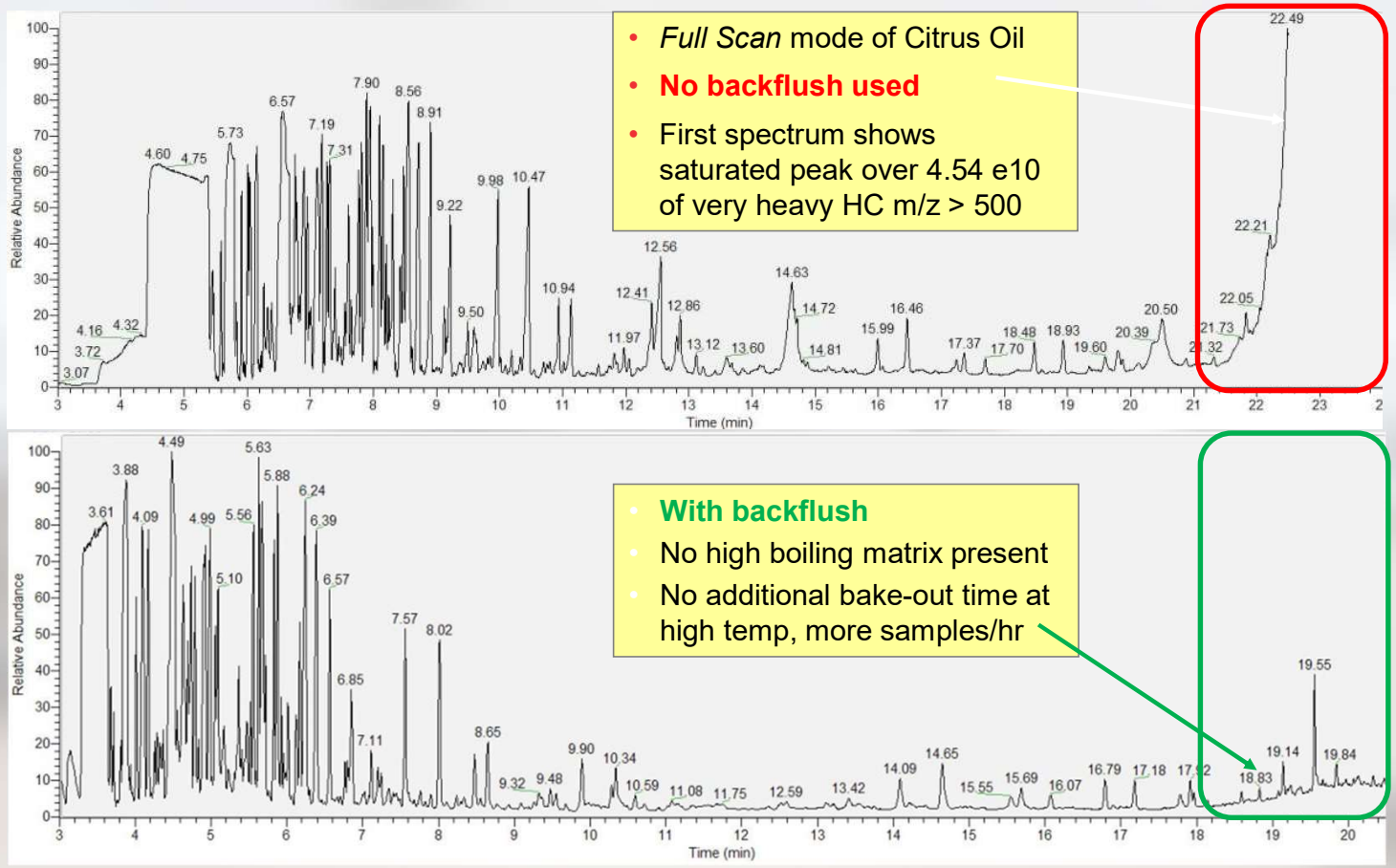
Analysis of Citrus Oils for Pesticides



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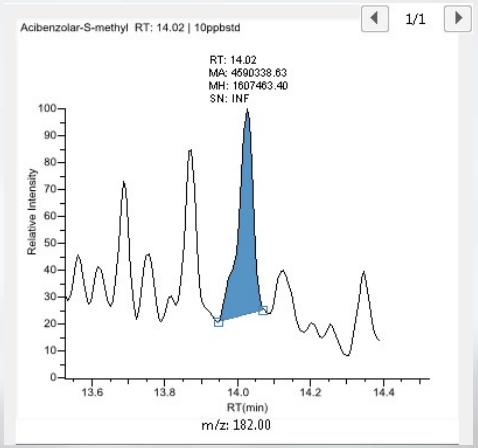
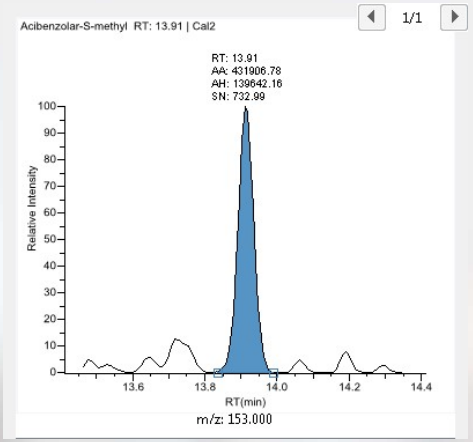
GC-MS/MS – What's so special?

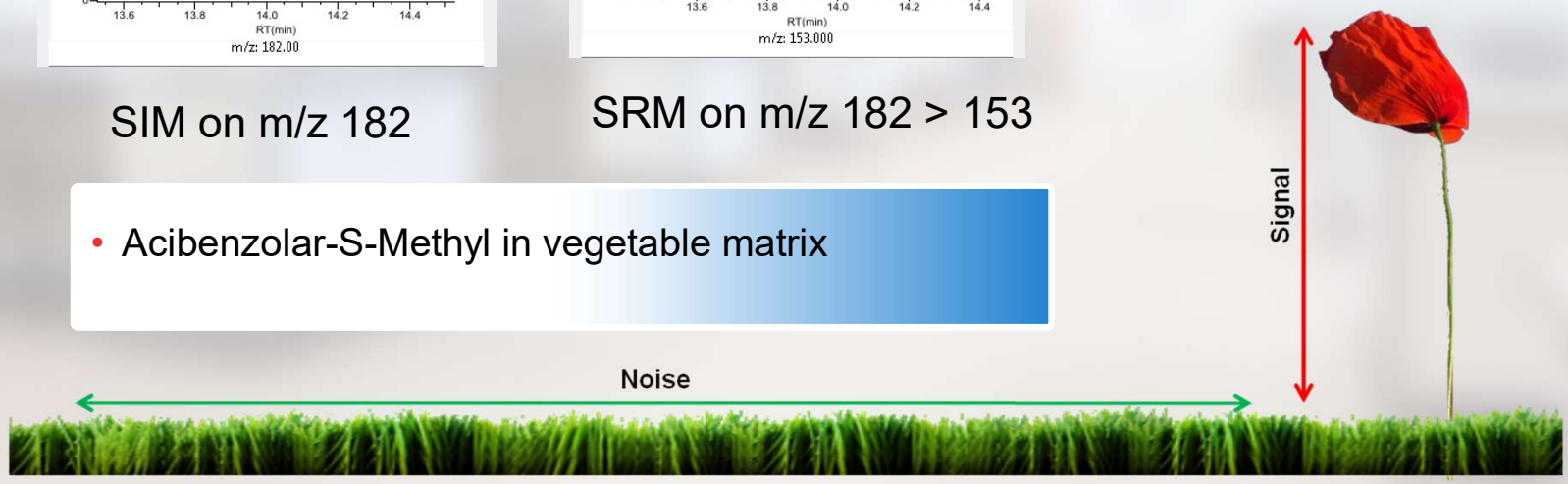


SIM on m/z 182



SRM on m/z 182 > 153

- Acibenzolar-S-Methyl in vegetable matrix



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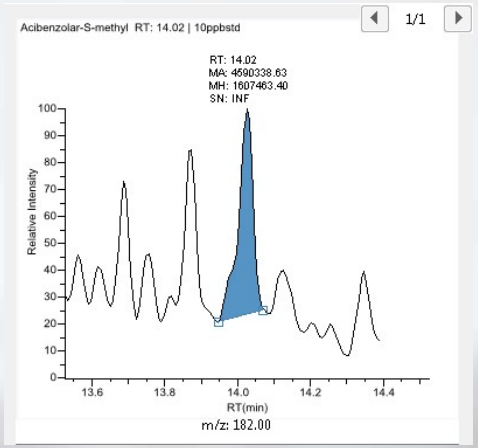
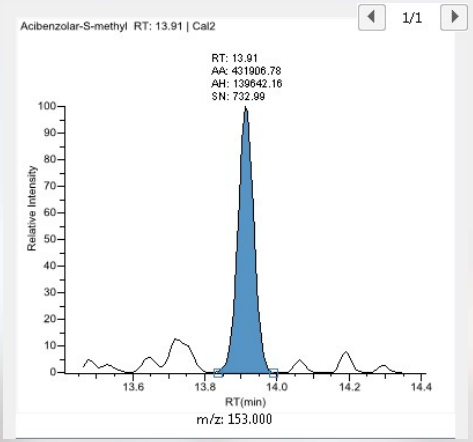
GC-MS/MS – What's so special?



SIM on m/z 182



SRM on m/z 182 > 153

Extra dimension of MS to drive selectivity

- Acibenzolar-S-Methyl in vegetable matrix

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Why triple quadrupole GC-MS/MS?

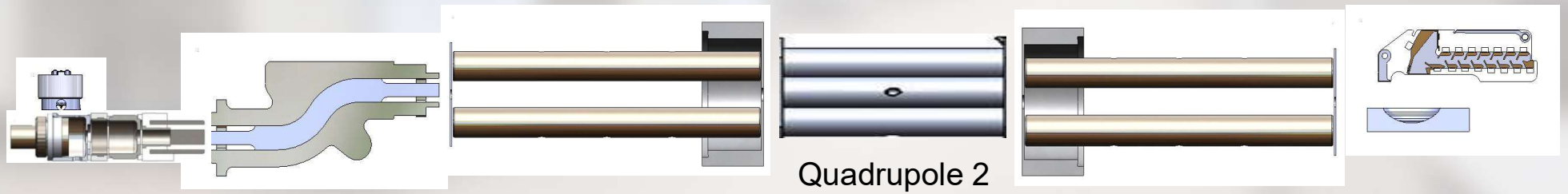
Single Quadrupole GC-MS

MS

Ion Source | Ion Transfer Optics | Quadrupole | EM Detector

Triple Quadrupole GC-MS/MS

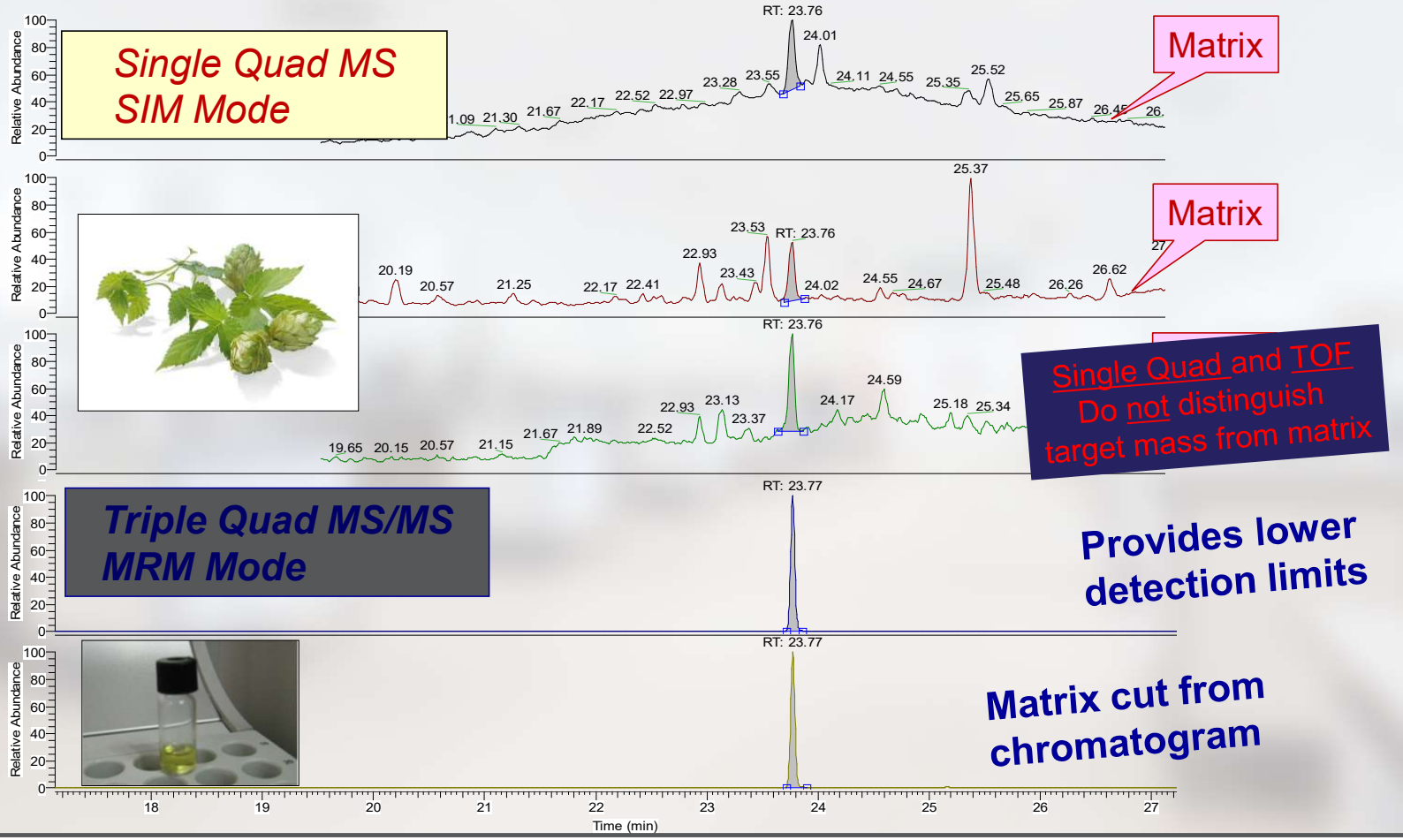
MS | MS | MS



Ion Source | Ion Transfer Optics | Quadrupole 1 (Q1) | Quadrupole 2 (Q2) Collision Cell | Quadrupole 3 (Q3) | EM Detector

Heart of MS/MS

Quinoxyfen in Hops - Using SIM and MS/MS



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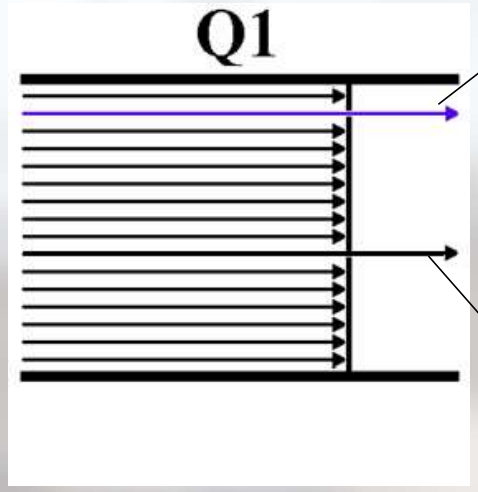
Step 1: Precursor Ion Selection

Q1 = mass selective



Selected analyte precursor ion

Matrix ion with same mass number

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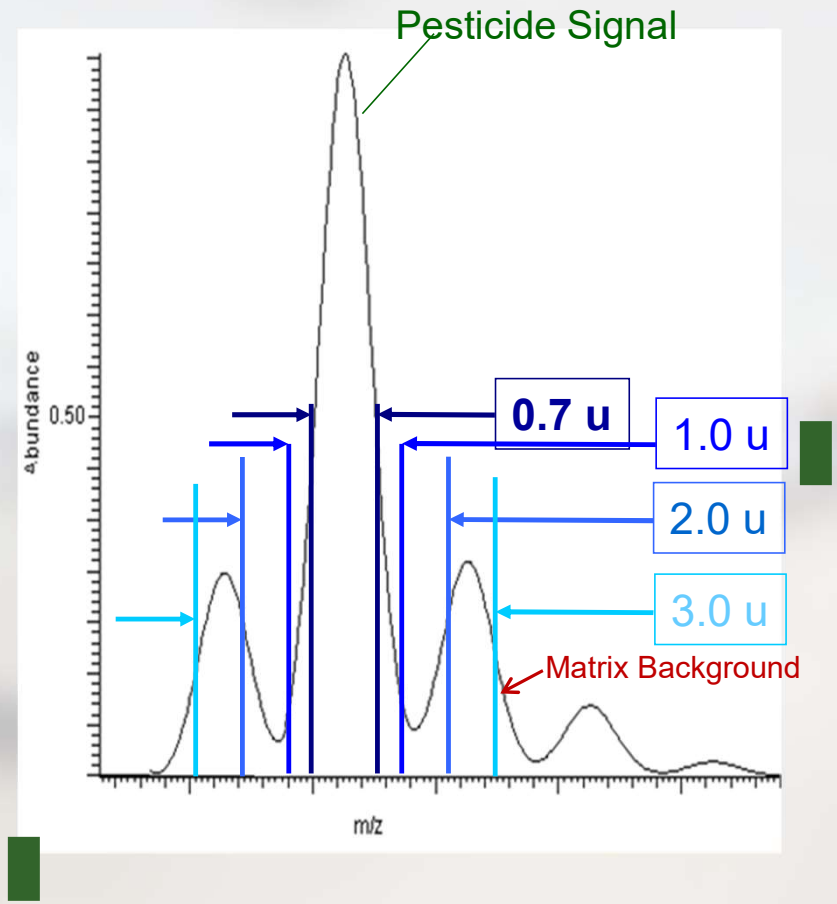
More Selectivity for More Sensitivity

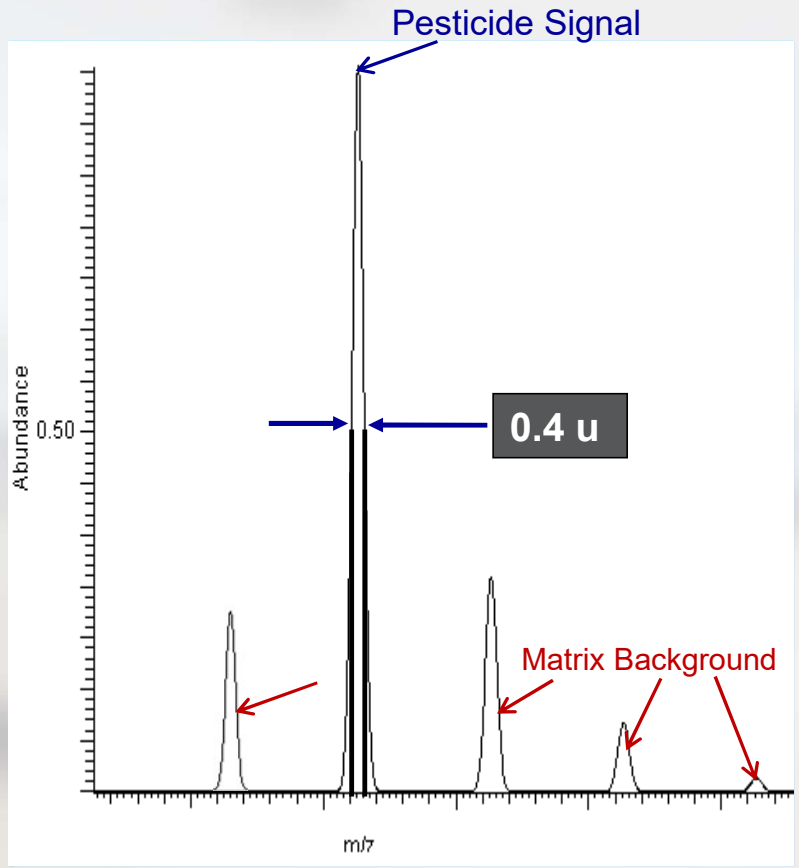


Other technologies use a wide Q1 mass window with a multiple of unit resolution.

More matrix gets into the collision cell and severely sacrifices selectivity.

A narrow mass window of 0.7 Da selects the target precursor efficiently from the matrix

Higher Resolution for More Selectivity in Q1



H-SRM

Highly Selective SRM Mode

Less matrix gets into the collision cell – **less noise**.

Increased Q1 resolution on the TSQ Quantum **increases selectivity** for **higher S/N.**

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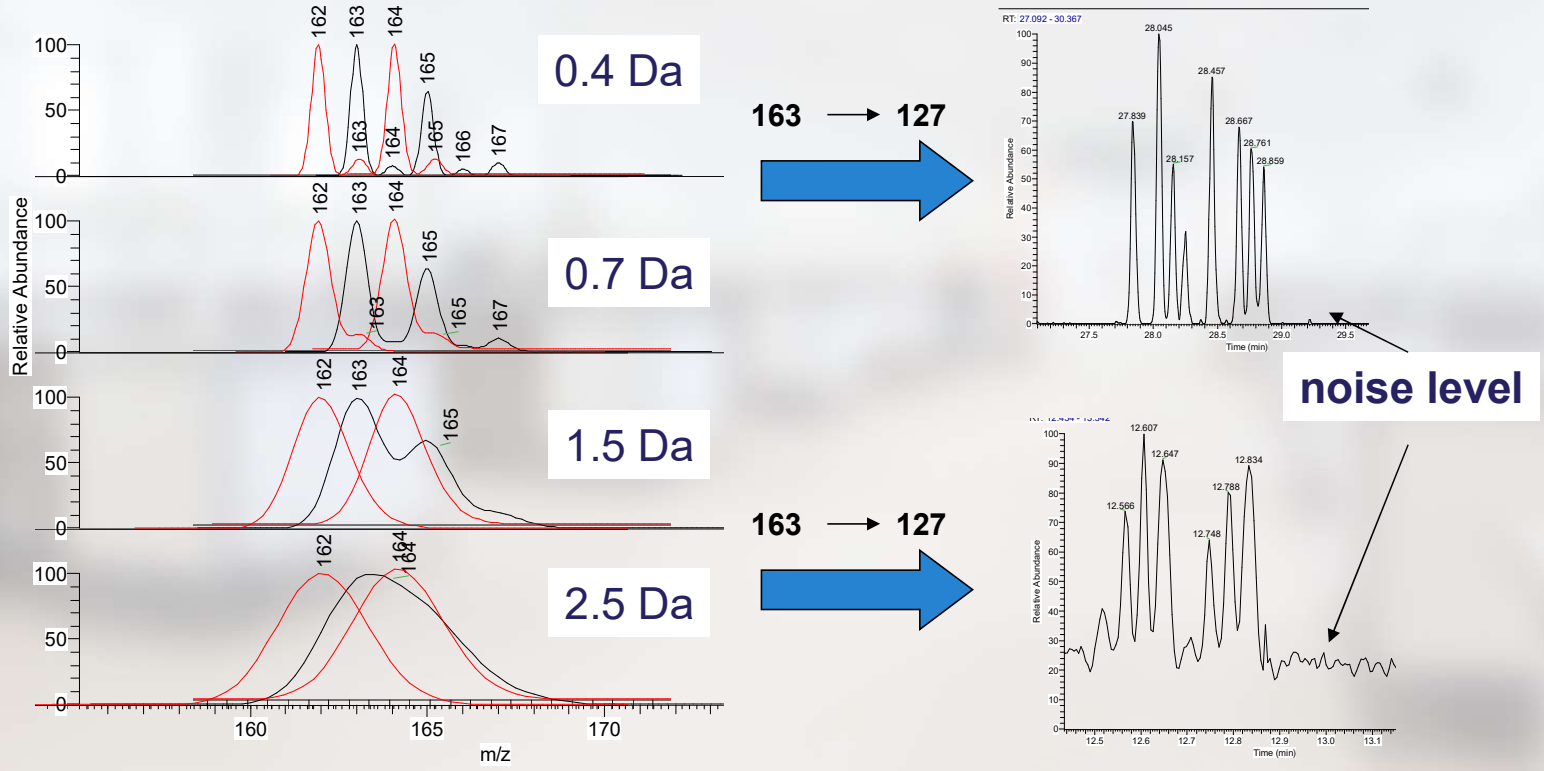
Mass resolution - FWHM of 0.4, 0.7, 1.5 and 2.5 Da

An example: 163 as one of precursor ions for cyfluthrin and cypermethrim,
162 or 164 as interference ions



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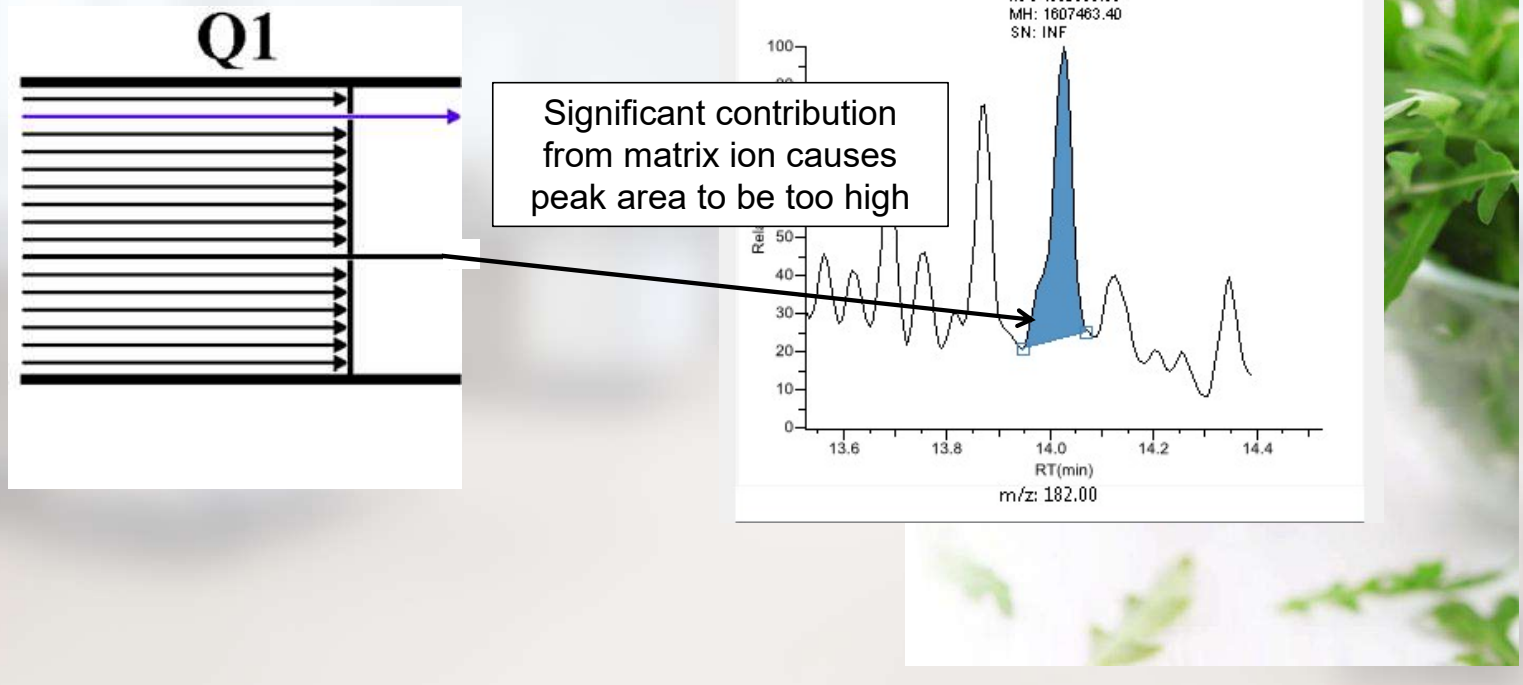
Step 1: Precursor Ion Selection

Acibenzolar-S-Methyl – 10 ppb in Lettuce



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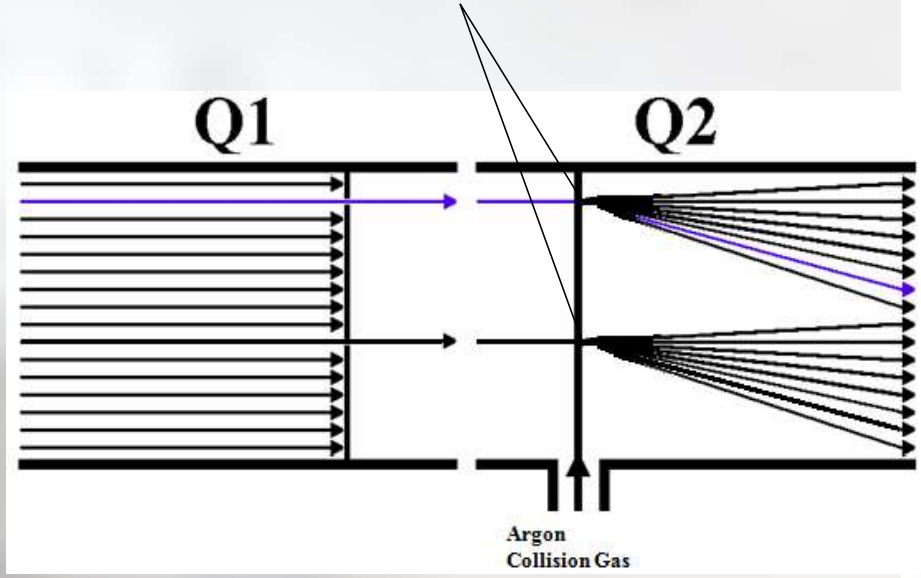
Step 2: Fragmentation in Collision Cell

Fragmentation of both analyte and matrix ion



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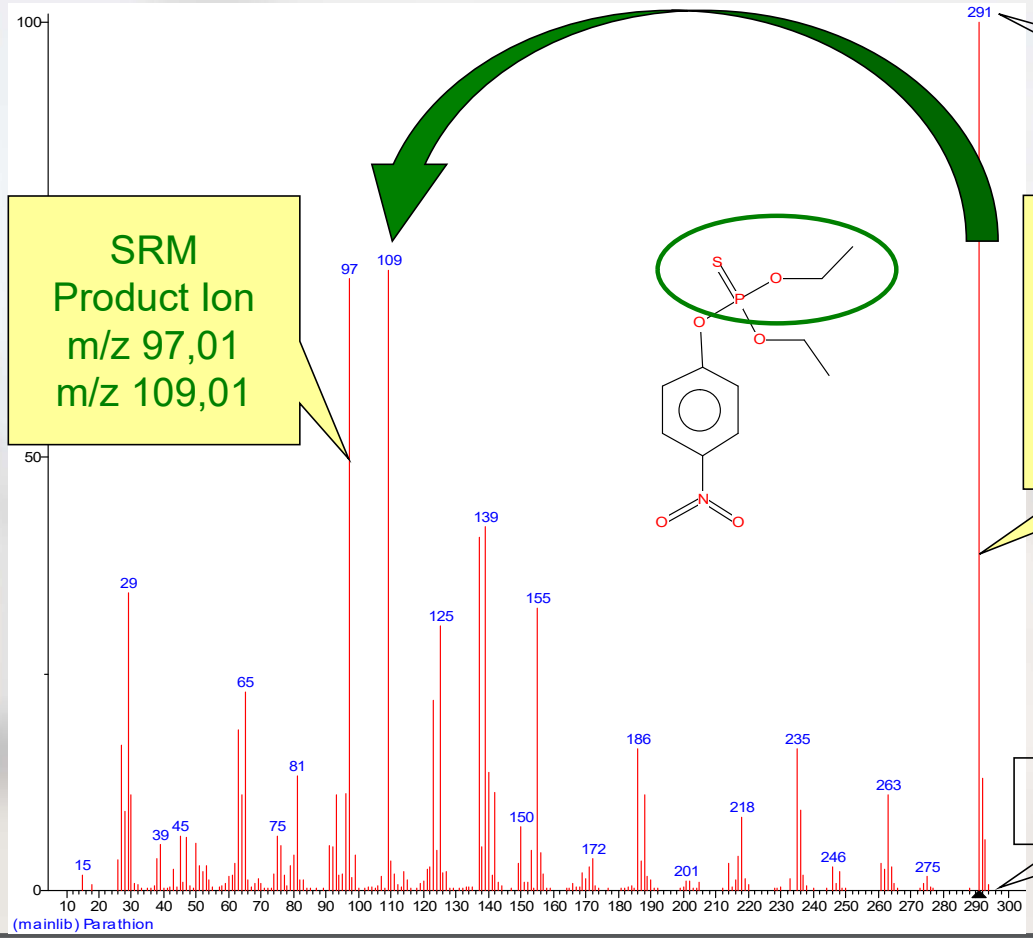
Structure Specific Selectivity – Parathion-Ethyl



SRM
Product Ion
m/z 97,01
m/z 109,01

M^+ m/z 291,03

SRM
Precursor Ion

(used for SIM in single quad methods)

NIST Library Spectrum

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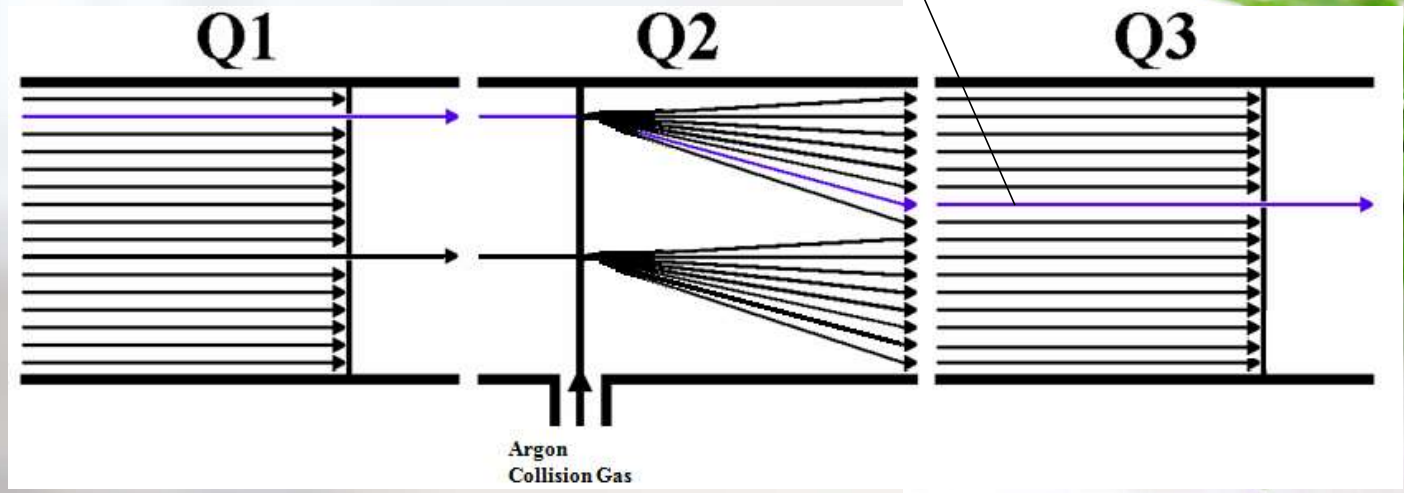
Step 3: Production Ion Selection



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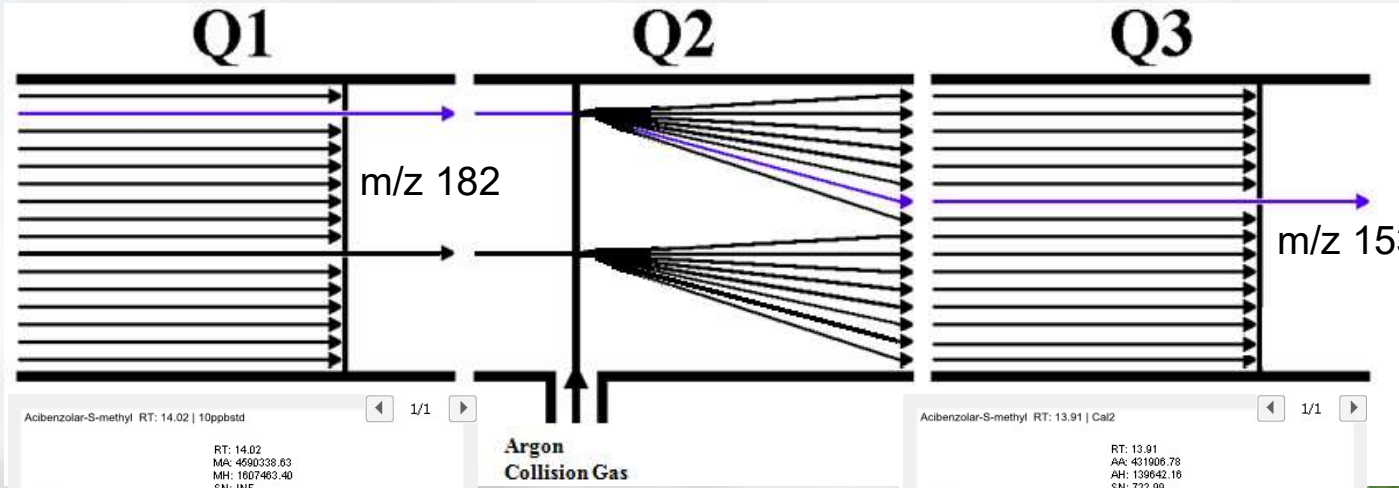
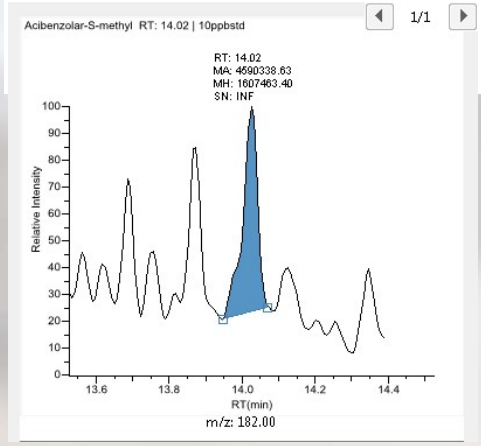
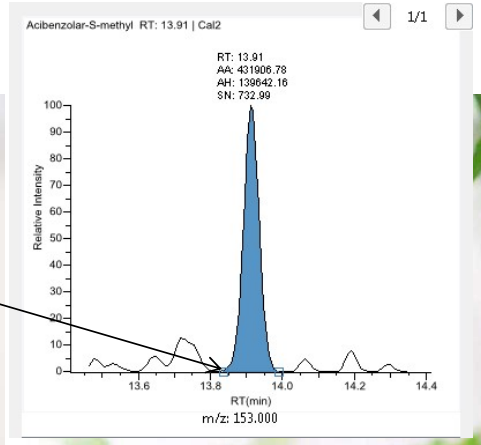
Step 3: Production Ion Selection





SIM on m/z 182

SRM removes the shoulder on from matrix interference & reduces background



SRM on m/z 182 > 153

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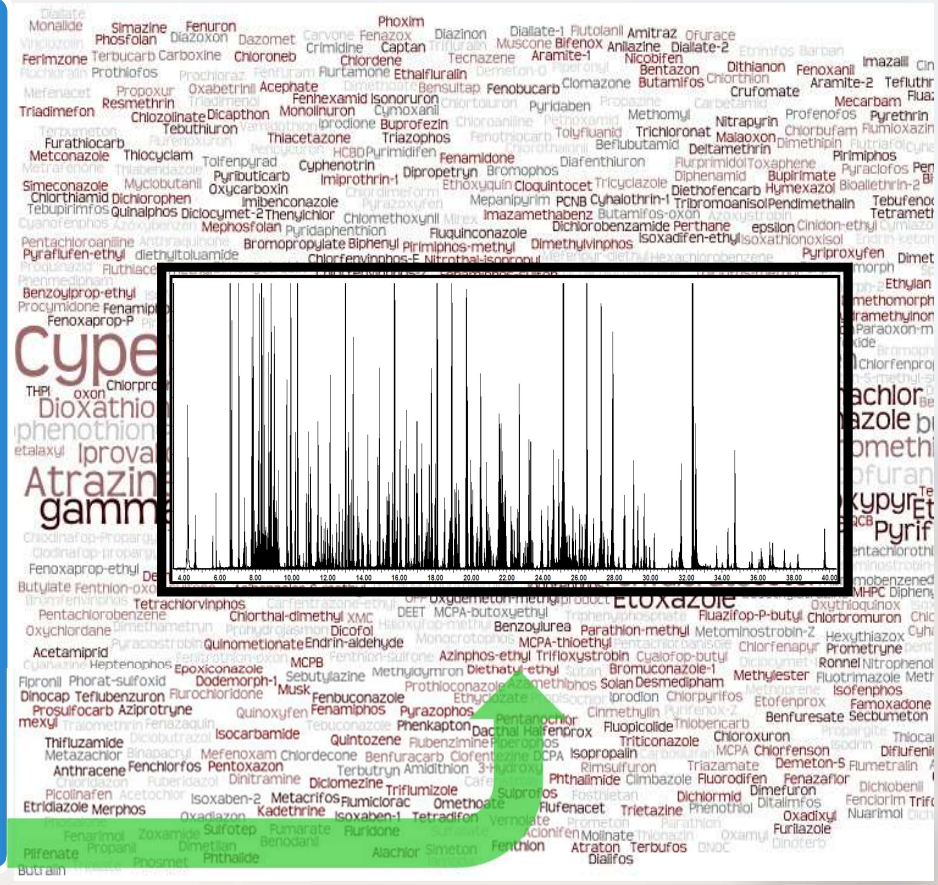
Benefits of selectivity

High selectivity

- Possibility to reduce selectivity in sample preparation.
- Reduced sample prep steps create a more generic sample prep method – more compounds & matrices
- Consolidated GC-MS methods due to high performance – buffer against requirements
- Compressed chromatography possible
- Easy peak evaluation – auto-integrators



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TSQ 9000 Pesticide Analyzer

Thermo Scientific TSQ 9000 Pesticide Analyzer

A complete pesticide method implementation, management and maintenance solution to drive unstoppable result productivity

TSQ 9000 PA designed to create powerful pesticide methods that are:

1. **Self-customized**
2. **Auto-optimized**

Powering the TSQ 9000 AEI Pesticide Analyzer

- **Preconfigured performance leading TSQ 9000 GC-MS/MS system featuring the award winning TRACE1310 GC**
- **Pre-loaded acquisition methods**
- **TraceGOLD GC Column and consumable technology**
- **Tracefinder 5.0 EFS Data Processing software**
- **1000+ Pesticide compound database (CDB) with 1500 + SRM transitions**
- **AutoSRM & timed SRM (t-SRM)**
- **Pesticide Analyzer installation guide**

Pesticide Analyzer Reference

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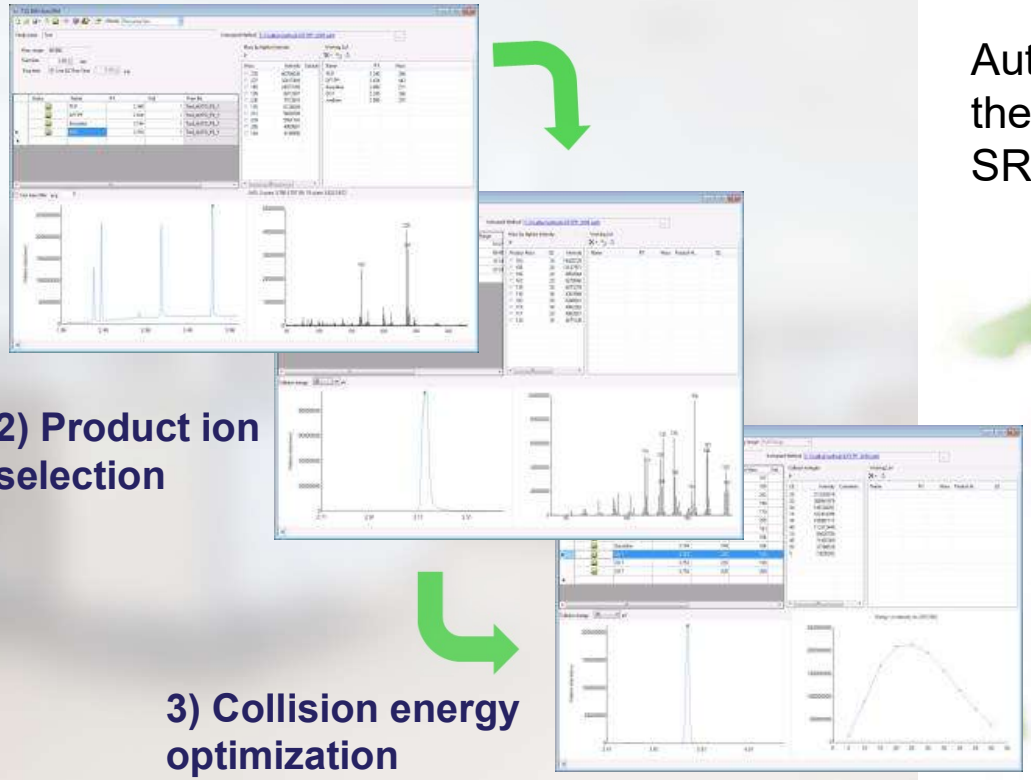
AutoSRM: Fast, Simple Route to Optimized SRM

1) Precursor ion selection



2) Product ion selection

3) Collision energy optimization

AutoSRM automates the development of SRM methodology

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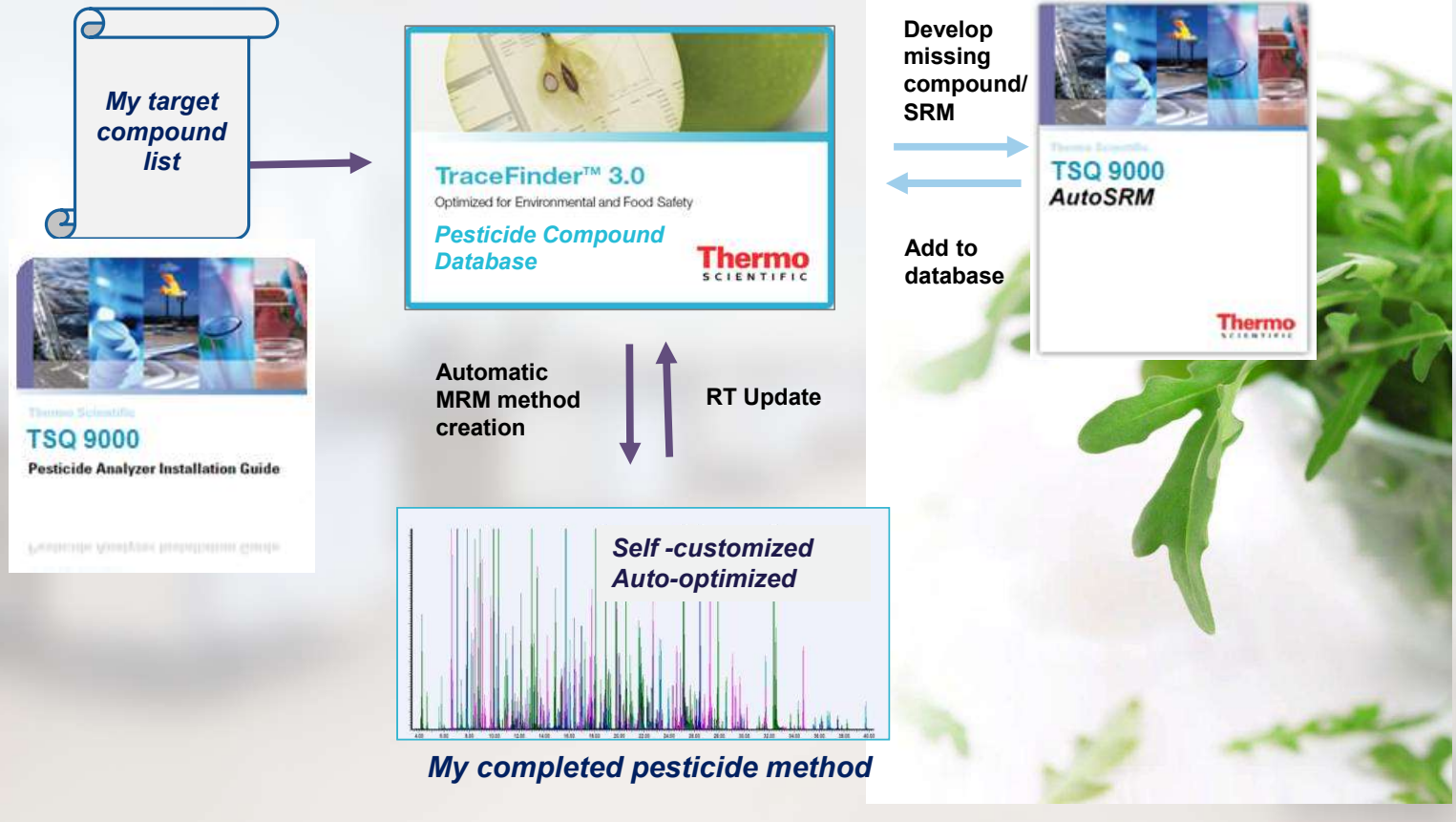
SRM Method Library

Table 27. Compounds For GCMS Positive Electron Ionization (EI) SRM Detection (Organized by Compound Name), continued

Compound	Nom Mass	Mass	M Defect	Precursor Ion (*m/z*)	Product Ion (*m/z*)	Collision Energy (eV)	Comments
Alachlor	269	269.1184	44	161.07	146.06	12	—
	—	—	44	188.08	160.07	10	
Aldrin	362	361.8760	-34	292.90	257.91	20	—
	—	—	-34	292.90	222.92	20	
	—	—	-34	264.91	229.92	26	
	—	—	-34	262.91	227.92	26	
	—	—	-34	262.91	192.93	32	
Allethrin	302	302.1883	62	367.23	213.03	25	—
	—	—	62	136.08	93.06	10	
	—	—	62	123.08	81.05	10	
Allidochlor	173	173.0609	35	134.05	56.02	15	—
	—	—	35	132.05	56.02	15	
Ametryne	227	227.1207	53	227.12	212.11	15	—
	—	—	53	227.12	170.09	10	
	—	—	53	227.12	170.09	15	
Amitraz	293	293.1894	65	293.19	162.10	10	—
	—	—	65	293.19	147.10	15	
Ancymidol	256	256.1213	47	228.11	121.06	15	—
	—	—	47	215.10	107.05	15	
Anilazine	274	273.9583	-15	238.96	142.98	23	—
	—	—	-15	177.97	142.98	9	

TSQ 9000 Pesticide Analyzer Workflow



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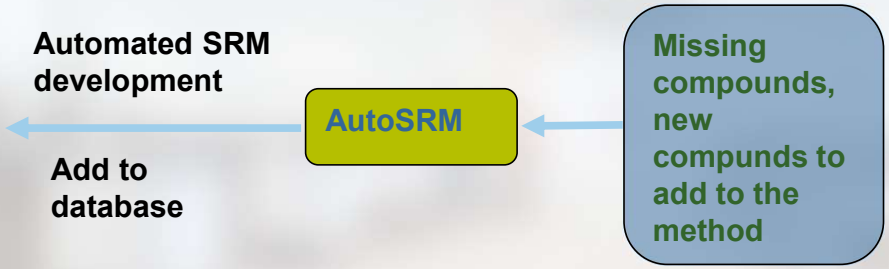
Auto-Optimized: Adding New Compounds

Step-by-step instruction make adding new compounds to the Compound Database easy.



Automates the following:

- ✓Creation of full scan, product ion scan, and SRM methods
- ✓Creation of sample sequences
- ✓Creation of data layouts for analyzing results
- ✓Selection of precursor, product and collision energies

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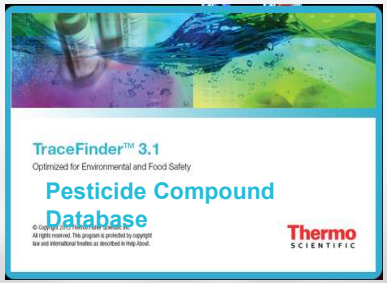
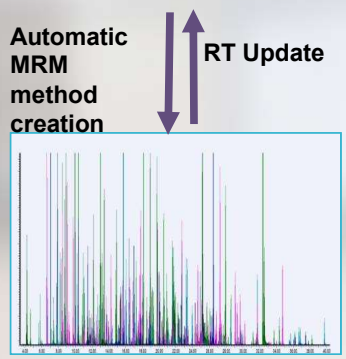
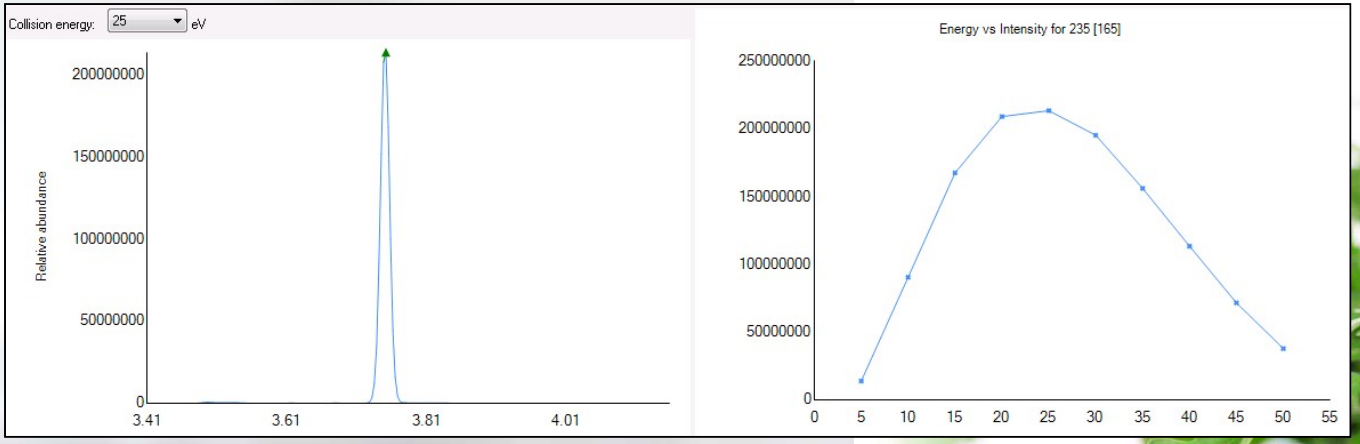
Auto-Optimized:Timed-SRM





- **Classical "segmented" SRM methods become problematic with ~100 or more compounds**
  - Difficult to find "quiet time" in TIC for segment break
  - Too many transitions at once can give unacceptable sensitivity
  - Complex calculations and dwell time decisions

Highlights of AutoSRM



End result showing optimized transition

- **Automates the following:**
  - Creation of fullscan, product ion scan and SRM methods
  - Creation of sample sequences
  - Creation of data layouts for analyzing results
  - Selection of precursor, product and collision energies

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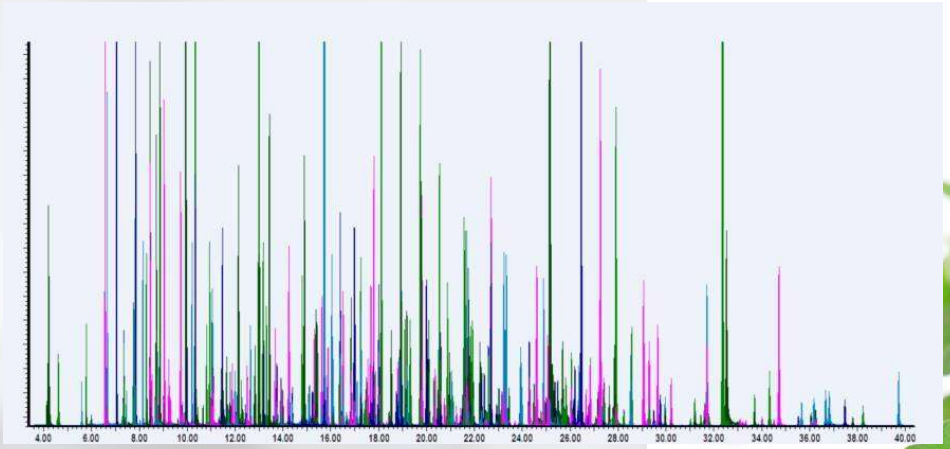
Analysis of 300 Pesticides – Segmented vs. Timed



Segmented SRM	Timed SRM
Closest compound to segment break: **5 seconds**	Closest compound to segment break: **15 seconds**
Average number of simultaneous transitions: **55**	Average number of simultaneous transitions: **15** **(4X higher dwell times- better sensitivity)**

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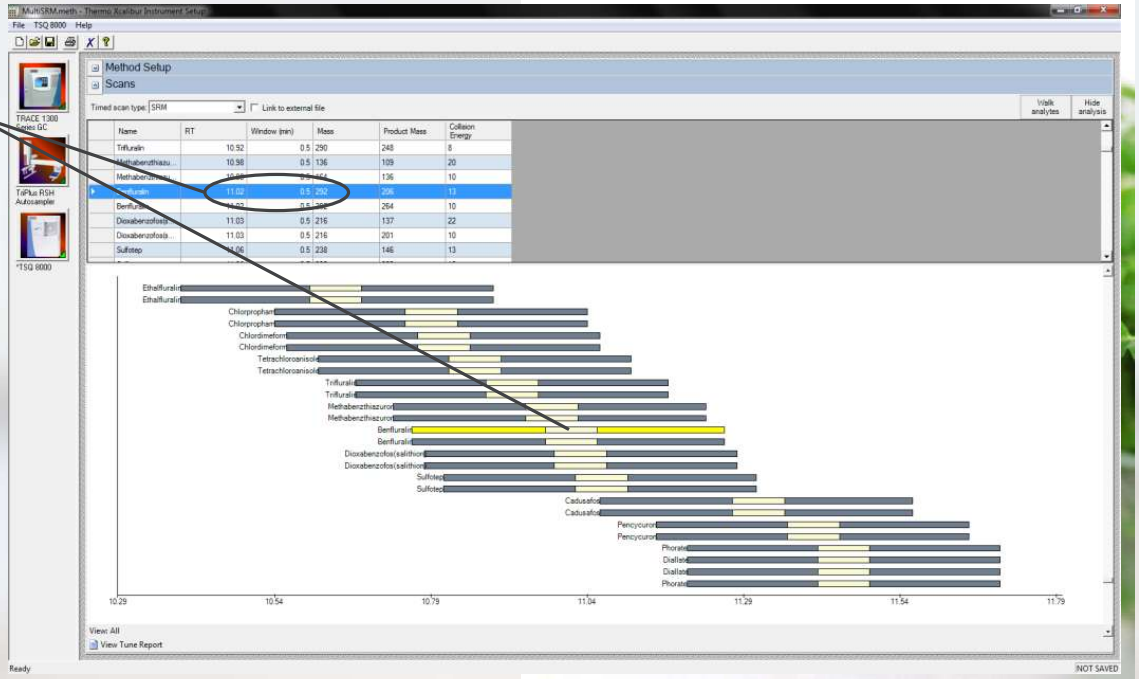
Auto-Optimised :Timed-SRM Method Overview

Acquisition windows centered around retention time



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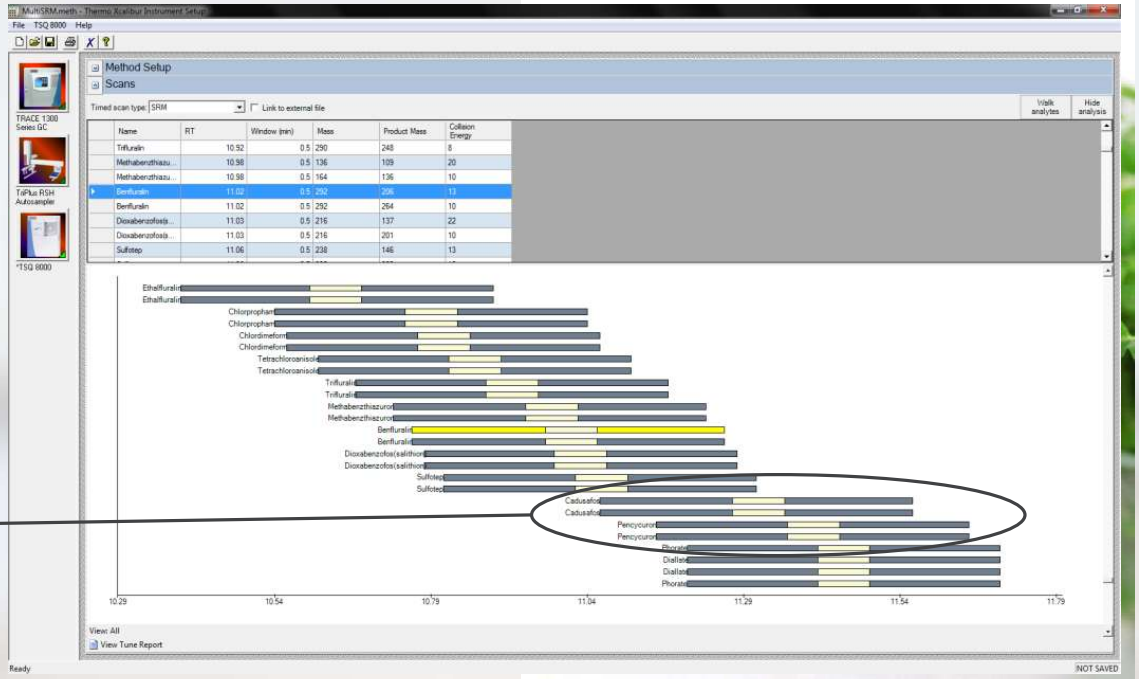
Timed-SRM Method Overview

Acquisition windows allowed to overlap



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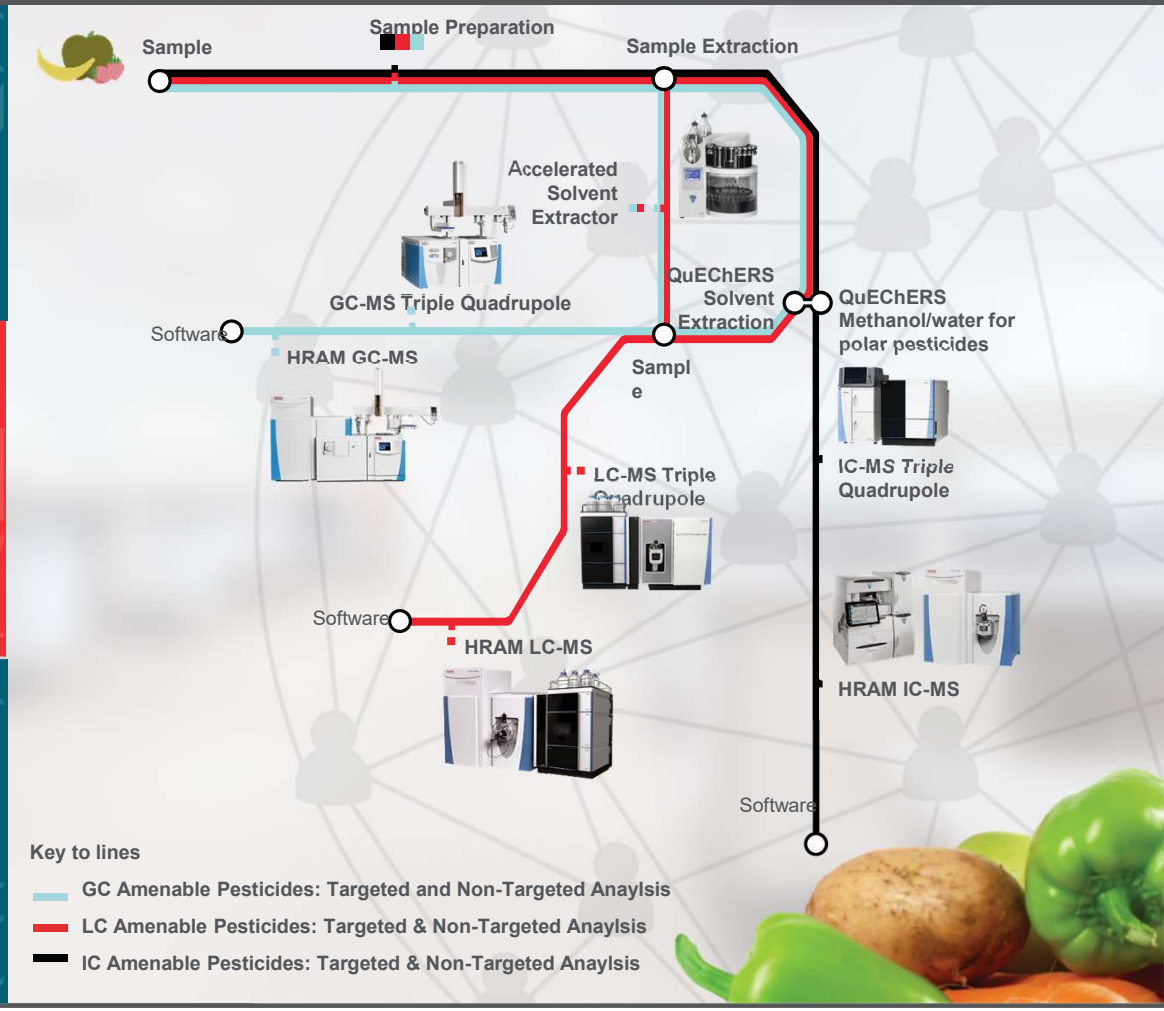
Pesticides Workflows

GC-MS/MS Pesticide Analyzer

LC-MS/MS Pesticide Explorer Collection

IC-MS/MS Anionic Pesticide Explorer



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Challenges of residue analysis

Challenges

- Sample variability (matrix)
- Compound different characteristics
- Number of samples
- Number of analytes monitored
- Low levels controlled (<10 ng/g)
- Fast response required

Goals

- Accurate, consistent data
- Keep the instruments running
- Process as many samples for as little cost as possible (cost effective)
- Remove interferences (matrix effect)

Complete Workflow Solution

Sampling → **Sample preparation** → **Sample analysis by GC/MSMS** → **Data evaluation, reporting**

- **Homogenization, sampling**

- **Extraction, clean-up, filtration, centrifugation etc...**

- **GC**
- **Detectors**
- **Consumables**

- **Advanced software solution**

Equipments and all consumables

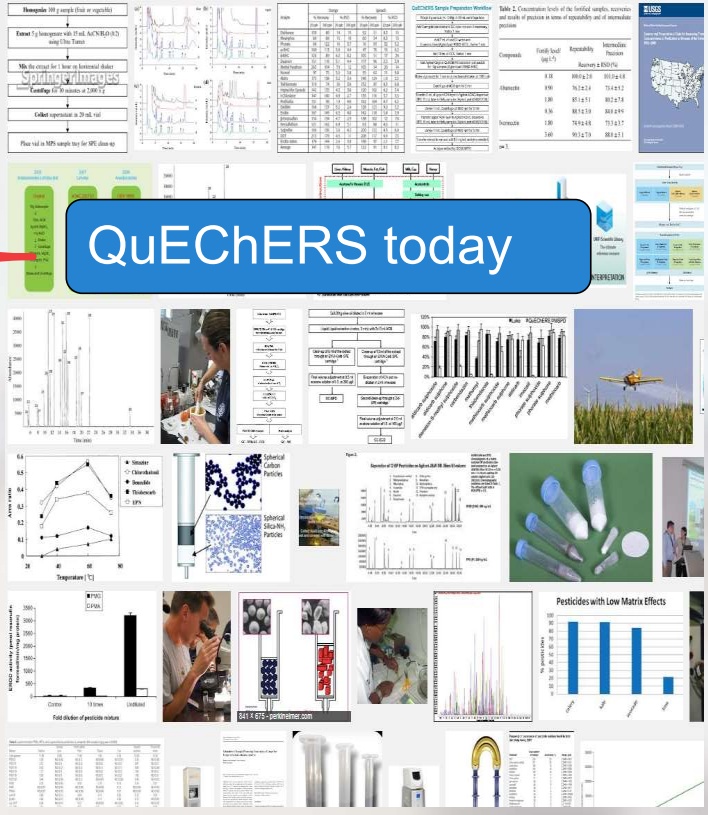
Thermo Scientific™
TSQ™ 9000 AEI
Pesticide Analyzer

ThermoFisher SCIENTIFIC

Former Pesticide Multi-Residue Method Setup

Sample preparation

- **Extraction**
  Acetonitrile, Ethyl acetate, Methanol...
- **Clean-up**
  GPC, SPE, LLE, on- and offline LC
- **Determination**
  GC, LC, GC/MS, LC/MS, GC/MS/MS, LC/MS/MS...



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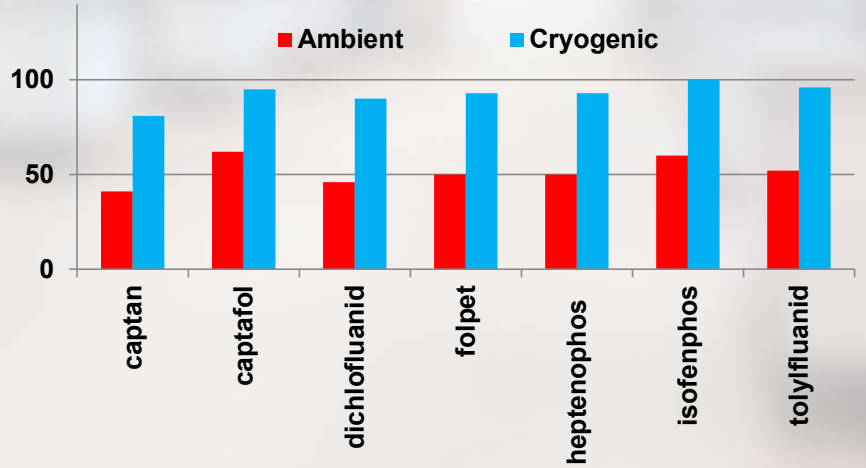
Sample Preparation

Sampling

- Losses of a number of pesticides during sample processing at ambient temperatures may occur – solidify with solid CO_2

Cryogenic Homogenization



Fussell et al. (2007) Food Additives & Contam., 24:1247-1256

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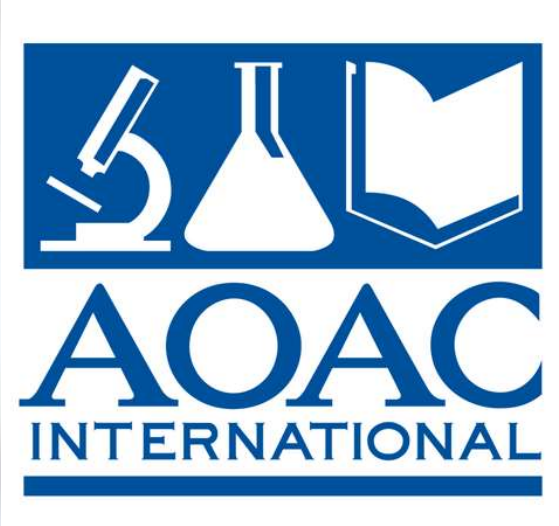
Sample Preparation
1.Extraction

QuEChERS methods



unbuffered

pH 4.8

pH 5.0 – 5.5

Original QuEChERS method (unbuffered)

original QuEChERS method

unbuffered

Quick, **E**asy, **Ch**eap, **E**ffective, **R**ugged, and **S**afe

Introduced in 2002 : European Pesticide Residues Workshop (EWPR), Rome, Book of Abstracts, 2002 Anastassiades M., Lehotay S.J., et al., Quick, Easy, Cheap, Effective, Rugged and Safe (QuEChERS) approach for the determination of pesticide residues,

Published in 2003: 2] Anastassiades M., Lehotay S.J., Stajnbaher D., Schenck F.J., Fast and easy multiresidue method employing acetonitrile extraction/partitioning and "dispersive solid-phase extraction" for the determination of pesticide residues in produce, J. AOAC Int., 2003, 86(2), 412-31, PMID: 12723926

•Validated in 2005, with subsequent modification in 2007

Unbuffered method have a negative effect on few pH-dependent pesticides

The original QuEChERS version included no pH control (unbuffered)

4g $MgSO_4$, 1g NaCl

- ✓ **Rapid** (8 samples in less than 30 min)
- ✓ **Simple** (no laborious steps, minimal sources of errors)
- ✓ **Cheap** (ca. 1 € per sample for the sample preparation)
- ✓ **Low Solvent Consumption** (10 mL acetonitrile)
- ✓ **Practically No Glassware Needed**
- ✓ **Wide Pesticide Range** (polars, pH-dependent compounds)
- ✓ **Extract in Acetonitrile** (GC- and LC-amenable)

Quick, **E**asy, **Ch**eap, **E**ffective, **R**ugged, **S**afe

Association of Official Analytical Chemists(AOAC) pH dependent

AOAC Official Method 2007.01
Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate

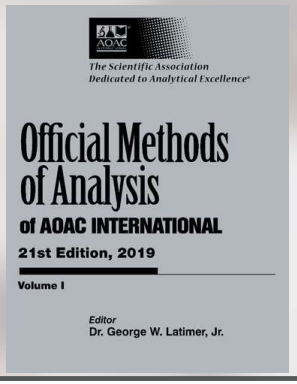
S.J. Lehotay, K. Mastovska, A.R. Lightfield, J. AOAC Int. 88 (2005), 615-629 & 60A

Buffered method

Relatively strong Acetate buffer pH 4.8 is used for low pH susceptible compounds . Low pH samples such as orange juice (pH~3.5) also need pH adjustment during extraction to efficiently extract pesticides of a range of polarities

pH 4.8

6 g $MgSO_4$, 1.5 g Na Acetate



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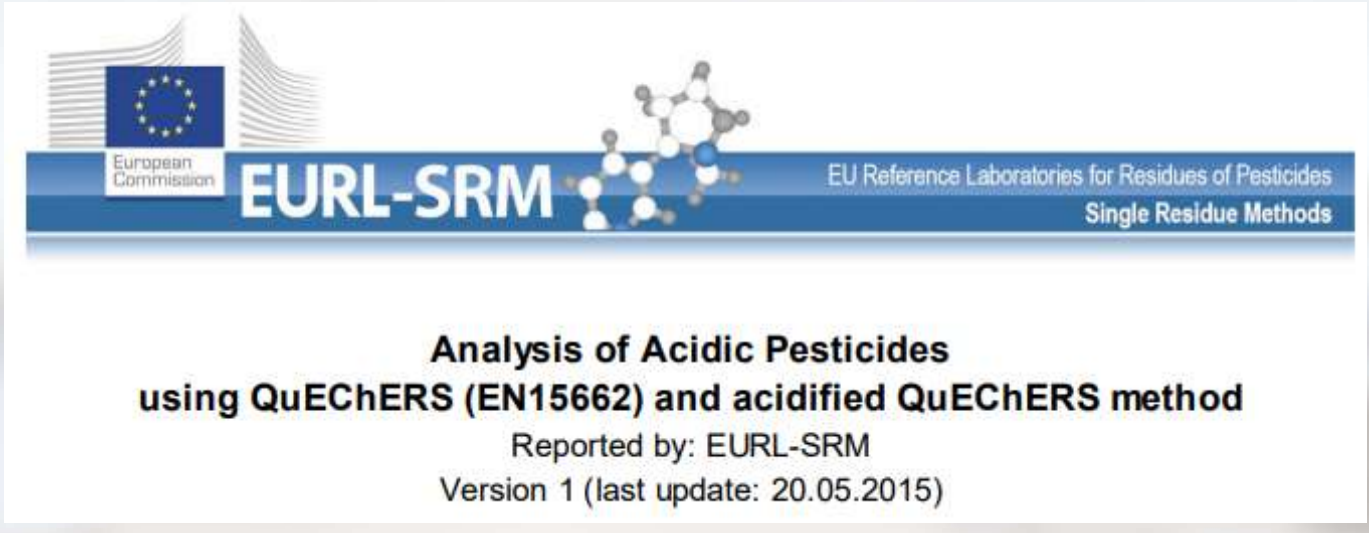
European Committee for Standardization (EN 15662) pH dependent

M. Anastassiades, E. Scherbaum, B. Tasdelen, D. Stajnbaher, in: H. Ohkawa, H. Miyagawa, P. W. Lee (Eds.), Crop Protection,Public Health, Environmental Safety, Wiley-VCH, Weinheim, Germany, 2007, p.439.



pH 5.0 – 5.5

4 g MgSO4, 1 g NaCl, 1 g NaCitrate ($Na_3C_6H_5O_7$), 0.5 g disodium citrate sesquihydrate ($HOC(COOH)(CH_2COONa)_2 \cdot 1.5H_2O$)

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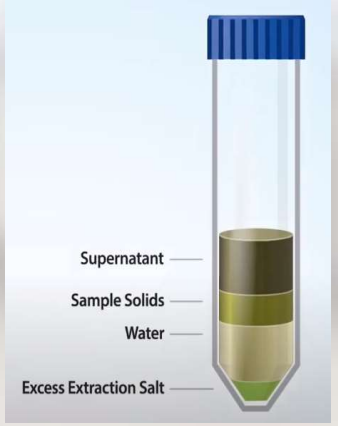
Sample Preparation
2.Clean-up

Cleaning step - Dispersive Solid Phase Extraction (d-SPE)

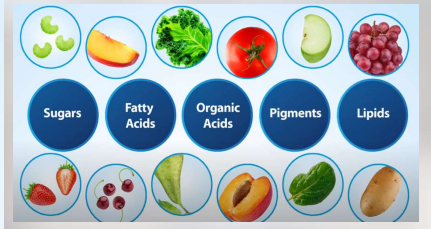
I. Sample Homogenization (cryogenic & wetted if required)

II. Extraction / partitioning & ACN & salts (Origin, AOAC, EN)

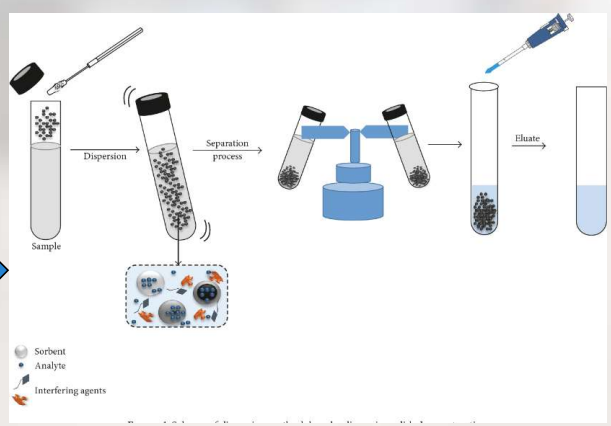
III. Cleaning process Dispersive Solid Phase Extraction (d-SPE)
I + II steps



Matrix interferences
co-extracts



III. Step Dispersive Solid Phase Extraction (d-SPE)



Criteria for right cleaning process d-SPE

1. Size of d-SPE tube
2. What sorbent is inside of the tube
3. How much of sorbent is inside of the tube

$MgSO_4$

PSA primary, secondary, amine

C_{18} end capped silica coated with C_{18} sorbent

GSB graphitized carbon black

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d-SPE Sorbents

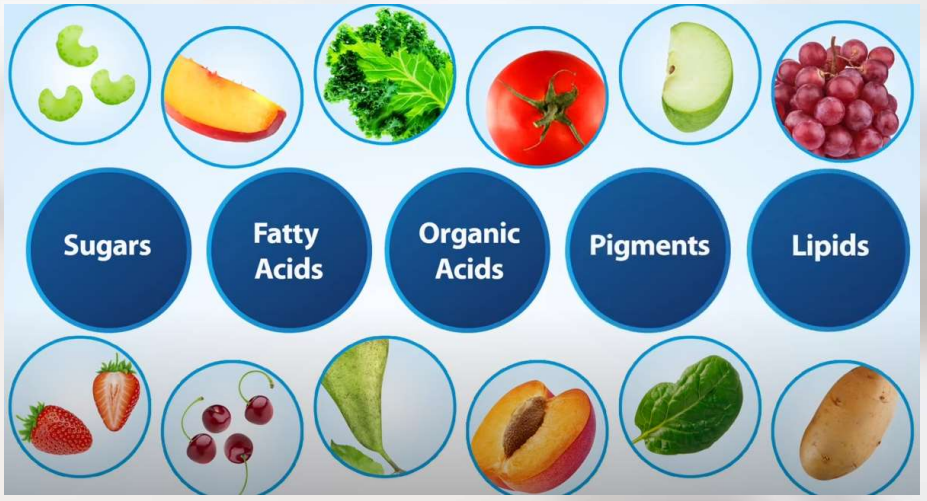
MgSO4 — Residual water removal after extraction

PSA
Primary Secondary Amine — Polar matrix components, Sugar, Organic Acids, Fatty acids, Sterols

C18
end capped silica coated with C18 sorbent — Non polar-fat & waxes

GSB
graphitized carbon black — Pigment, colour

Al_2O_3 — Removal of lipophilic compounds



Enhanced Matrix Removal - Lipids

EMR-Lipid Mechanism –Size exclusion and hydrophobic interaction.

The use of freezing-out after ethyl acetate salt-out extraction can remove the high lipid content in the co-extracted matrix

GC/MS/MS to analyze food samples with high fat content.

Sorbent

- The materials selective hydrophobic interactions increase.
-Suspension of nanoparticles (high surface area).
-Rapidly interacts with straight chain, “lipid-like” functional groups.

What are Lipids?

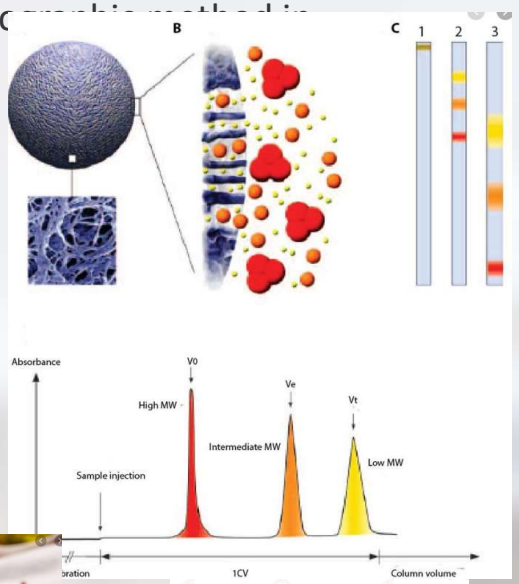
A class of naturally occurring hydrocarbon containing compounds commonly known as fats and oils (butter, vegetable oil, **cholesterol** and other **steroids**, **waxes**, phospholipids, and fat-soluble vitamins. The common characteristic of all of these compounds is that they are essentially insoluble in water, yet soluble in one or more organic solvents)

Enhanced Matrix Removal-Lipid

Gel Permeation-Chromatography (GPC)

GPC is the separation of molecules according to molecular size when using an organic solvent as the mobile phase

SEC Size-exclusion chromatography, also known as molecular sieve chromatography, is a chromatographic method in which molecules in solution are separated by their size, and in some cases molecular weight.



The QuEChERS method, for example, can bring coextractives such as fatty acids, sugars, and carotinoids[12] into the solution along with the analytes of interest which can often interfere with the chromatography. Thus, GPC remains one of the most effective cleanup methods for complex samples because it removes the matrix entirely rather than try to work around it

Some challenges with high lipid content in which lipophilic pesticides may remain in the fatty layer even after the extraction.

GPC is especially true for highly colored samples or those with high fat content.

A good example of this is the analysis of certain pesticides in meat using gas chromatography. To clean the sample prior to analysis, it is first dissolved in cyclopentane, and is then cleaned up with the GPC

Using gel permeation chromatography (GPC),olive oil minor constituents (diglycerides, free fatty acids, oxidation products, flavor compounds and dimers) can be fractionated and therefore quantified.

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Sample Analysis
3.Determination

Samples

Two sample types of baby food samples were sourced locally.

- Apple/pear/banana
- Carrot/potato

Samples were pre-spiked at 10 µg/kg with a pesticide mix containing over 200 individual pesticide residues.

Extractions were carried out following a citrate buffered QuEChERS protocol with dispersive solid phase extraction (dSPE) as per the European Committee for Standardization CEN 15662:2017 E.

GC and Injector conditions

TRACE 1310 GC System Parameters	
Injection Volume:	1 mL
Liner:	Siltek™ six baffle PTV liner (P/N 453T2120)
Inlet:	70°C
Carrier Gas:	He, 1.2 mL/min
Inlet Mode:	PTV Splitless (split flow 50mL/min after 2 min)
Column:	Thermo Scientific™ TraceGOLD™ TG-5SilMS with SafeGuard (30m x 0.25mm, 0.25µm- with 5m integrated guard column - P/N: 26096-1425)

PTV Parameters:	Rate (°C/s)	Temperature (°C)	Time (min)	Flow (mL/min)
Injection	-	70	0.10	-
Transfer	5.0	300	2.00	-
Cleaning	14.5	320	5.00	75.0

Oven Temperature Program:

Ramp	RT (min)	Rate (°C/min)	Target Temperature (°C)	Hold Time (min)
Initial	0	-	40	1.50
1	1.5	25.0	90	1.50
2	5.0	25.0	180	0.00
3	8.6	5.0	280	0.00
Final	28.6	10.0	300	5.00
Run time	35.6	-	-	-

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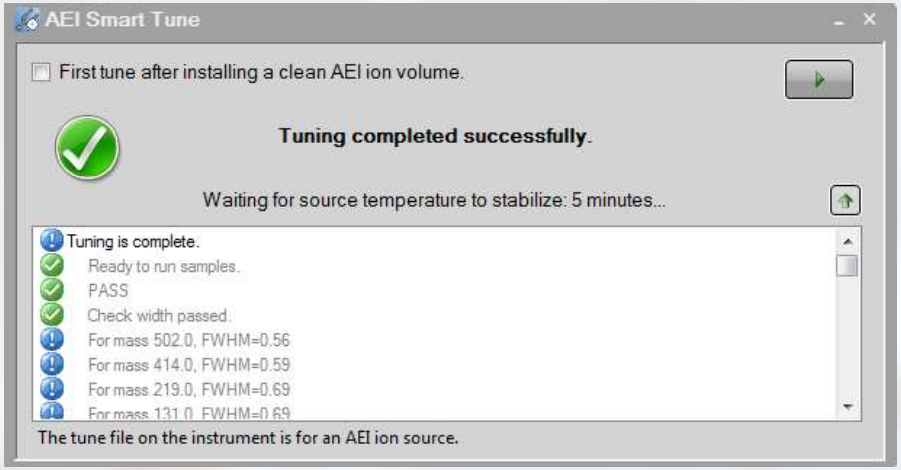
MS conditions

TSQ 9000 Mass Spectrometer Parameters	
Transfer Line:	250 °C
Ionization Type:	EI using the Thermo Scientific™ advanced electron ionisation (AEI) source
Ion Source:	320°C
Acquisition Mode:	Timed SRM, peak width 3 seconds, 10 scans per peak Detector gain factor x7
Tuning parameters:	AEI **SmartTune**
Collision gas and pressure:	Argon at 70 psi
Peak Width:	0.7 Da (both Q1 and Q3 @FWHM)

r9



selected or multiple reaction monitoring (SRM, MRM), mass resolution for precursor-ion isolation equal to or better than unit mass resolution

SANTE compliant

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Slide 55

r9 No mention of 45 EV ionisation energy
richard.fussell, 4/2/2018

Sample Preparation

Extraction

4000mg Magnesium Sulfate, 1000mg Sodium Chloride, 500mg Sodium Citrate dibasic sesquihydrate, 1000mg sodium citrate tribasic

60105-333 (50pk)

Clean up (dSPE)

Apple/banana/pear

Anhydrous Magnesium Sulfate (150mg), Primary/Secondary Amine (25mg)

60105-219 (100pk)

Carrot/potato

Anhydrous Magnesium Sulfate (150mg), Primary/Secondary Amine (25mg), Graphitized Carbon Black (25mg)

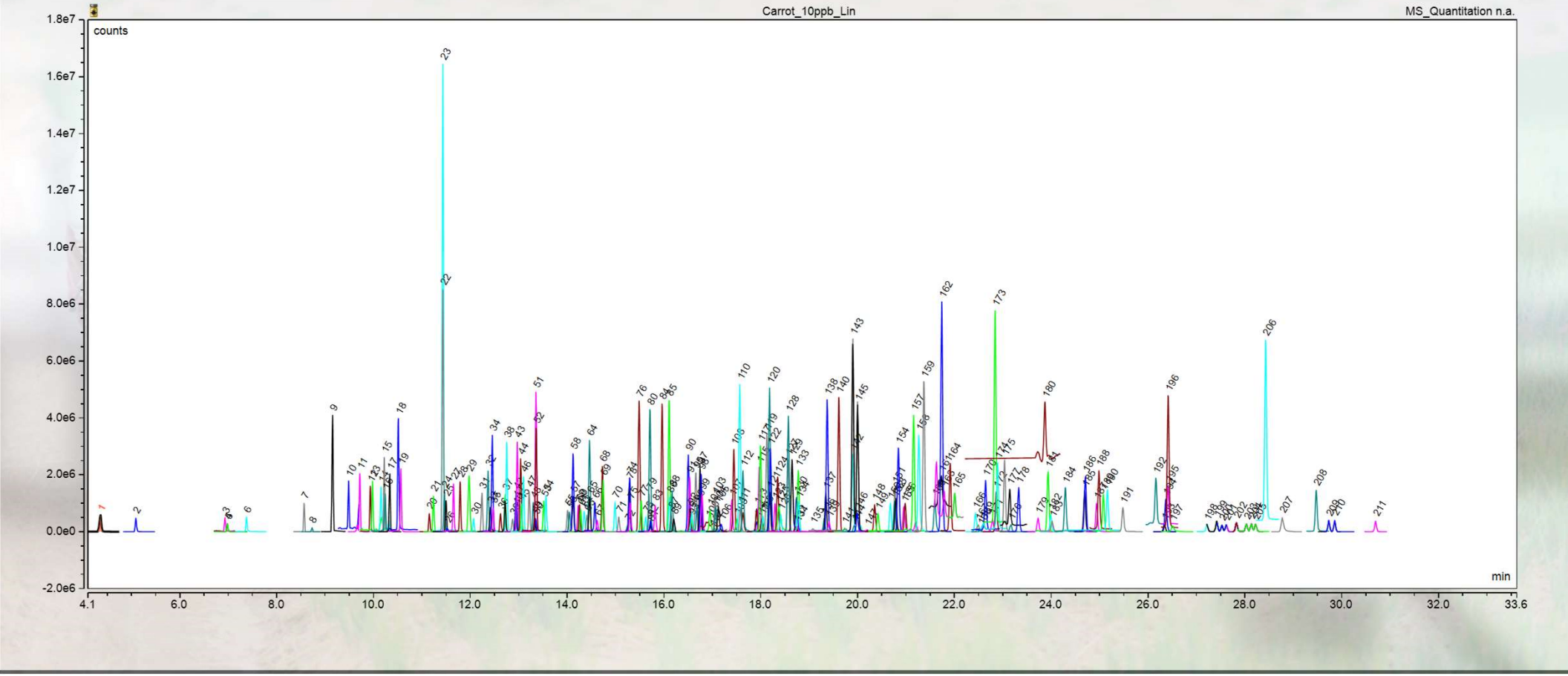
60105-221 (100pk)

Results

Pesticide recoveries were obtained from the QuEChERS extractions performed on the samples spiked before extraction (10 µg/kg). All detected compounds, at the three spiking levels in both matrices satisfied all SANTE requirements.

	Carrot 1ppb		Apple 1ppb		Carrot 2.5ppb		Apple 2.5ppb		Carrot 10ppb		Apple 10ppb	
	Mean recovery (n=6)	Precision (% RSD)	Mean recovery (n=6)	Precision (% RSD)	Mean recovery (n=6)	Precision (% RSD)	Mean recovery (n=3)	Precision (% RSD)	Mean recovery (n=6)	Precision (% RSD)	Mean recovery (n=6)	Precision (% RSD)
Min	34.1%	0.9%	27.6%	0.8%	35.8%	0.7%	27.2%	0.2%	34.1%*	0.3%	29.0%	1.0%
Max	119.6%	27.8%*	118.8%	26.3%	118.9%	29.2%†	119.5%	28.2%	120.0%	47.7%*	115.2%	16.7%
Average	100.3%	6.1%	95.2%	6.3%	95.2%	4.4%	98.7%	3.6%	96.1%	3.2%	96.9%	3.3%
No. of Pesticides detectable	202	202	202	202	208	208	209	209	210	210	210	210
No. of Pesticides within limits	197	201	197	201	203	207	206	208	206	209	208	210

Chromatography



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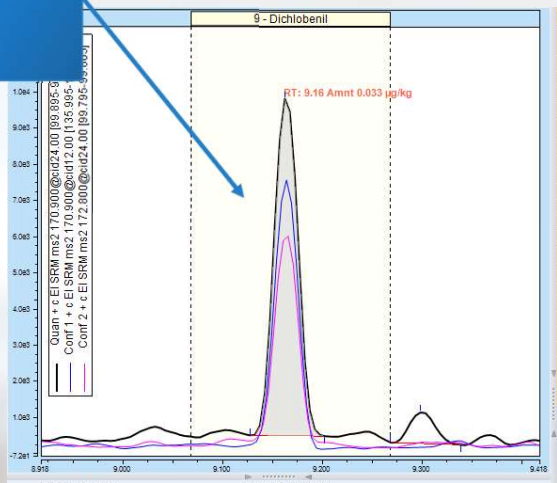
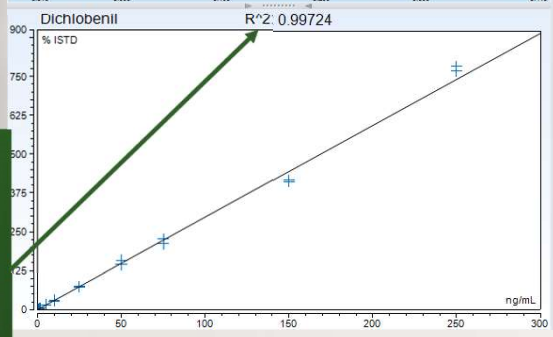
Sensitivity and linearity

Guassian peak shape - Quan SRM and two additional SRM transitions

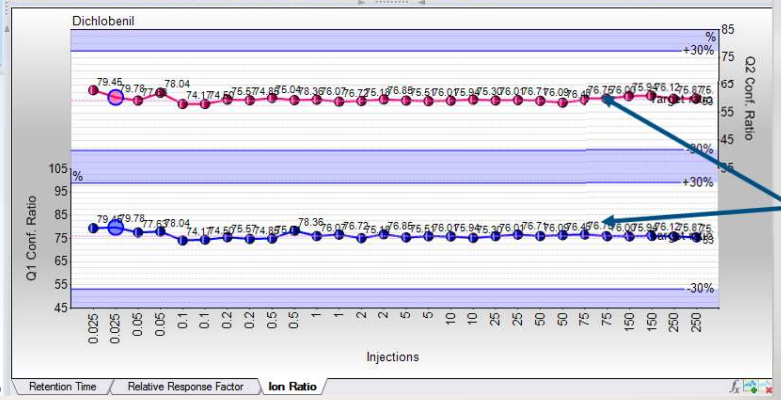
Dichlobenil at 25 fg (on column concentration)





	A	B	C	D	E	F	G	H	I
1	Inj.	Injection Name	Calibrator conc	Conf. Result Qual 1	Conf. Ratio Qual 1	Conf. Result Qual 2	Conf. Ratio Qual 2	SANTE Compliant	Relative response
2	No.	Selected Peak:	ng/mL		%		%		Factor
4			Dichlobenil	Dichlobenil	Dichlobenil	Dichlobenil	Dichlobenil	Dichlobenil	Dichlobenil
19	24	Carrot_5ppb_Lin	5.0000	Confirmed	75.51	Confirmed	59.56	Y	1.486
20	25	Carrot_5ppb_Lin	5.0000	Confirmed	76.01	Confirmed	59.35	Y	1.429
21	26	Carrot_10ppb_Lin	10.0000	Confirmed	75.94	Confirmed	59.53	Y	1.364
22	27	Carrot_10ppb_Lin	10.0000	Confirmed	75.30	Confirmed	59.88	Y	1.551
23	28	Carrot_25ppb_Lin	25.0000	Confirmed	76.01	Confirmed	59.61	Y	1.459
24	29	Carrot_25ppb_Lin	25.0000	Confirmed	76.71	Confirmed	59.73	Y	1.498
25	30	Carrot_50ppb_Lin	50.0000	Confirmed	76.09	Confirmed	59.48	Y	1.570
26	31	Carrot_50ppb_Lin	50.0000	Confirmed	76.46	Confirmed	58.80	Y	1.454
27	32	Carrot_75ppb_Lin	75.0000	Confirmed	76.75	Confirmed	59.80	Y	1.514
28	33	Carrot_75ppb_Lin	75.0000	Confirmed	76.00	Confirmed	59.80	Y	1.406
29	34	Carrot_150ppb_Lin	150.0000	Confirmed	75.94	Confirmed	60.67	Y	1.379
30	35	Carrot_150ppb_Lin	150.0000	Confirmed	76.12	Confirmed	60.85	Y	1.394
31	36	Carrot_250ppb_Lin	250.0000	Confirmed	75.87	Confirmed	59.70	Y	1.574
32	37	Carrot_250ppb_Lin	250.0000	Confirmed	75.53	Confirmed	59.77	Y	1.542
33		Mean							1.40
34		Stdev							0.14
35		RSD (%)							10.2%
36		Min			74.17		58.32		0.94
37		Max			79.78		63.27		1.57

Calibration Summary | Peak Results | System Suitability Test | Calibration



Ion ratio confirmation flagging and custom columns to show SANTE compliance

RF RSD% ~10%

Both SRM qualifier ion ratios stable across the entire calibration range

Excellent linearity with $R^2 > 0.99$
1/x weighting used throughout,

Duplicate injection per calibration point

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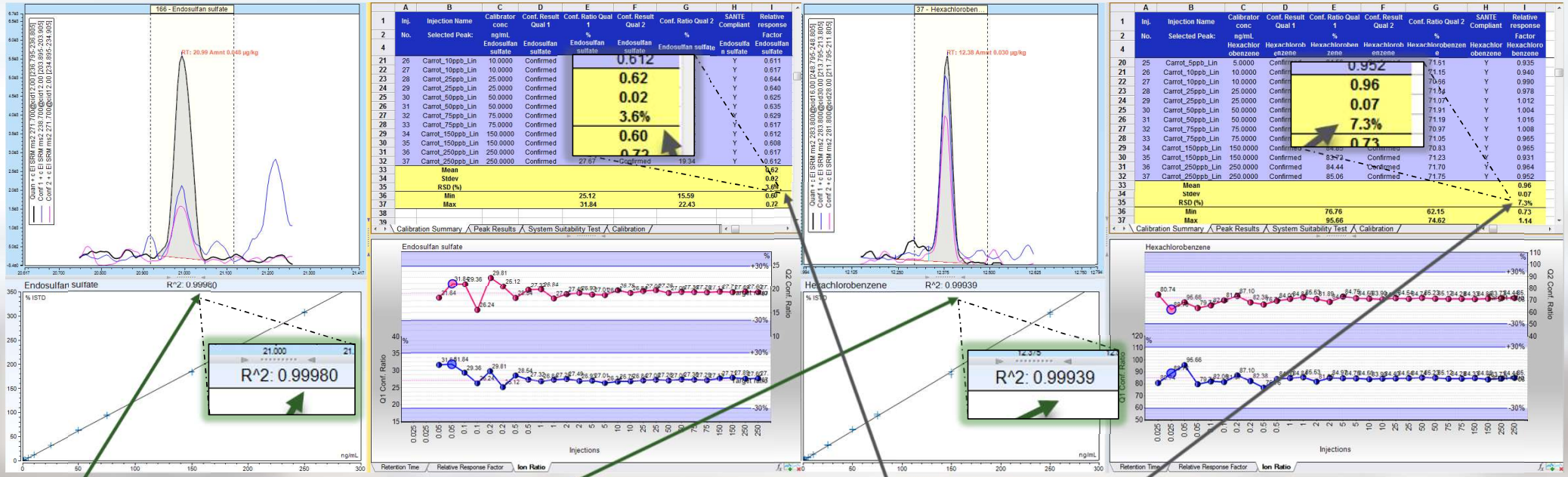
Sensitivity and linearity (continued)

Endosulfan sulphate at 50 fg (on column concentration)

Hexachlorobenzene at 25 fg (on column concentration)



Excellent linearity with R^2 > 0.999
1/x weighting used throughout,

Duplicate injection per calibration point

RF RSD% <10%

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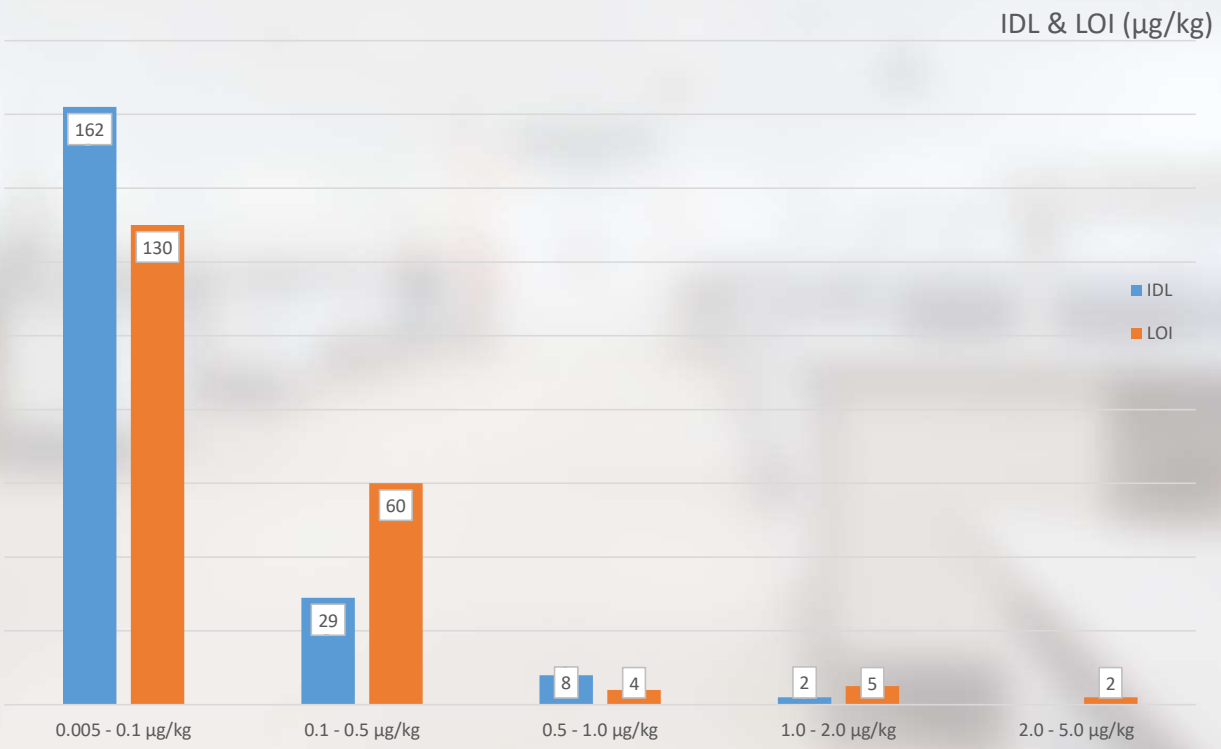
Instrumental detection limit (IDL) & Limit of identification (LOI)

IDL determined by repeatedly injecting (n=10) the 0.05, 0.2, 1 and 5 µg/kg matrix-matched standards and using the Student's-t critical values for the corresponding degrees of freedom (99% confidence).

LOI was defined as the lowest level calibration standard which satisfied all of the SANTE criteria

- Two SRM product ions
  - Ion ratio agreement with average calibration (±30% relative)
  - Ions must fully overlap



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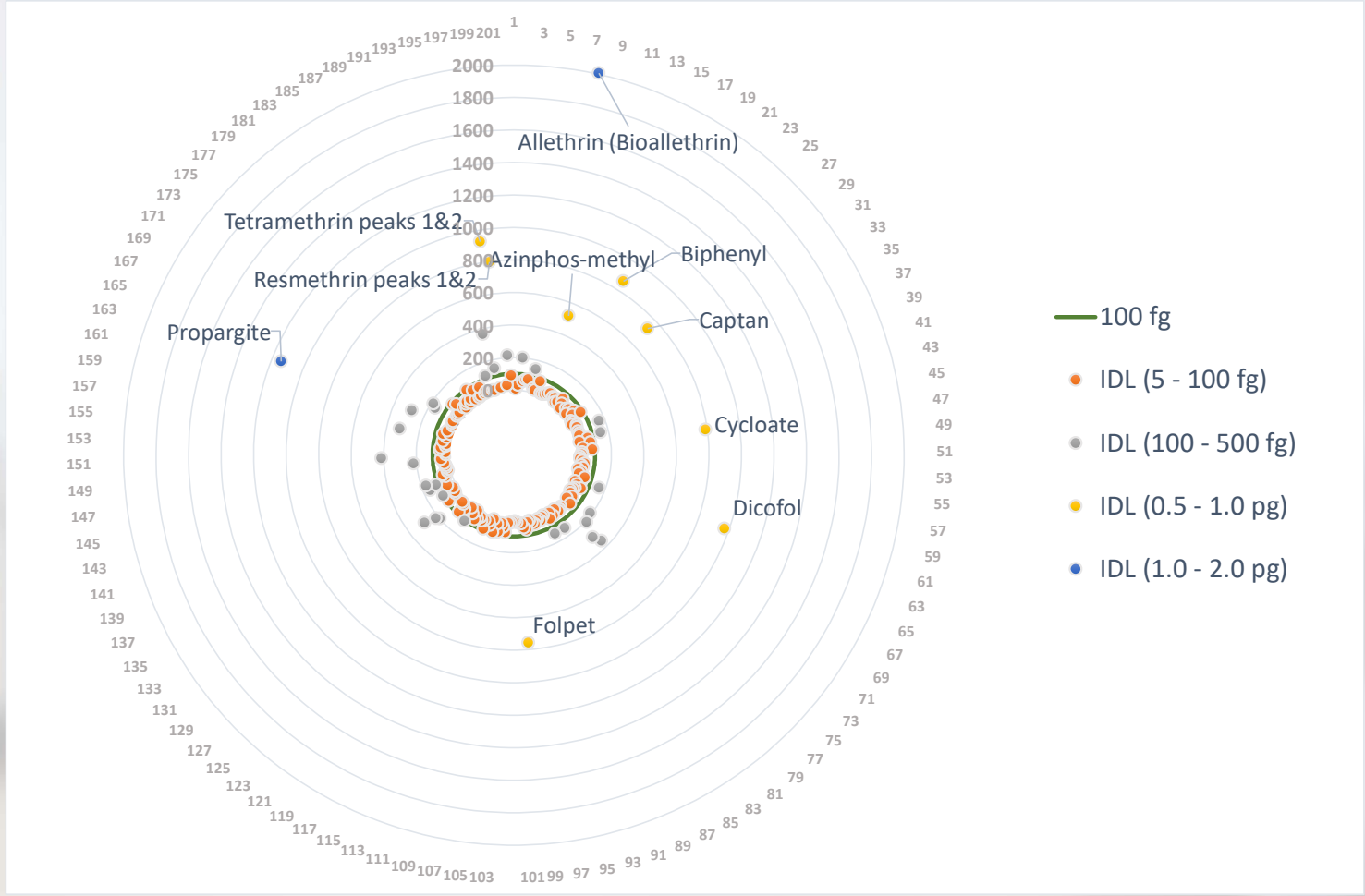

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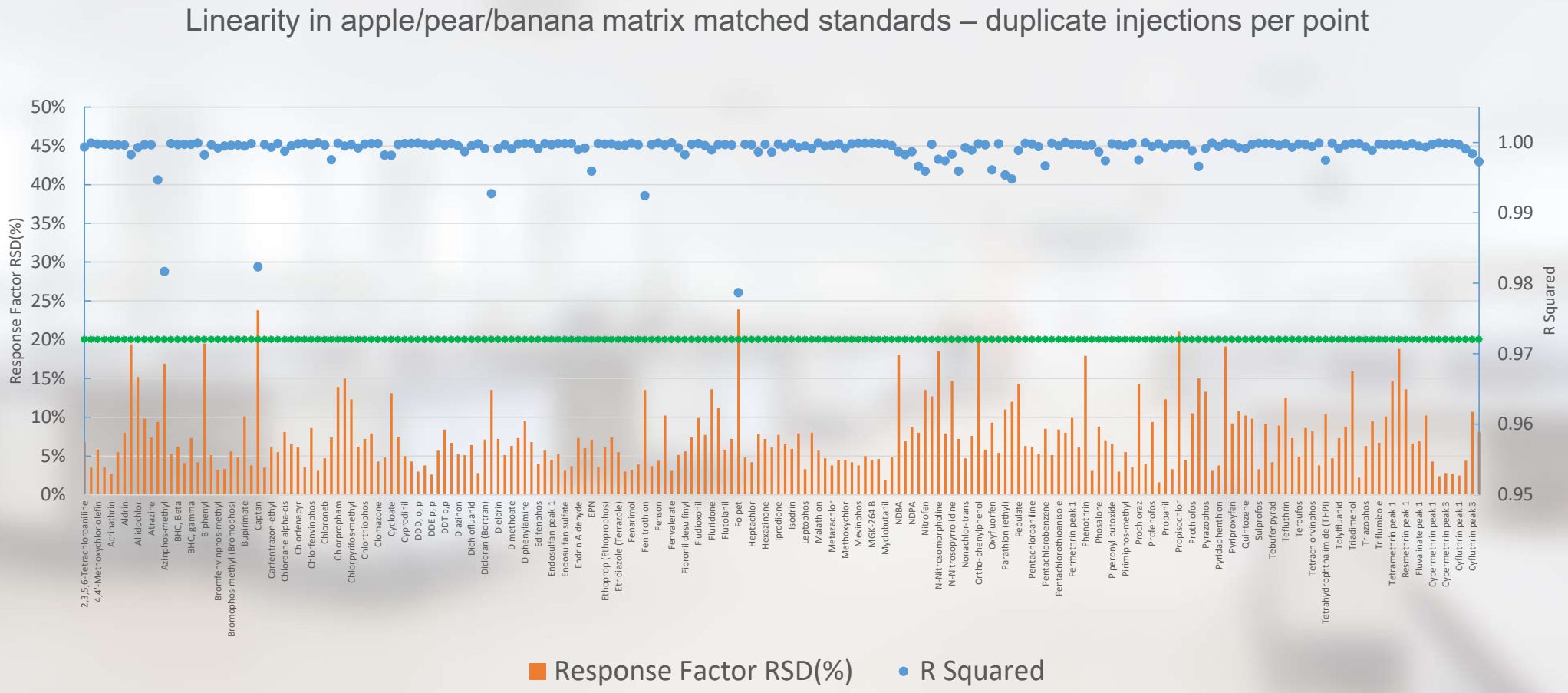
Linearity



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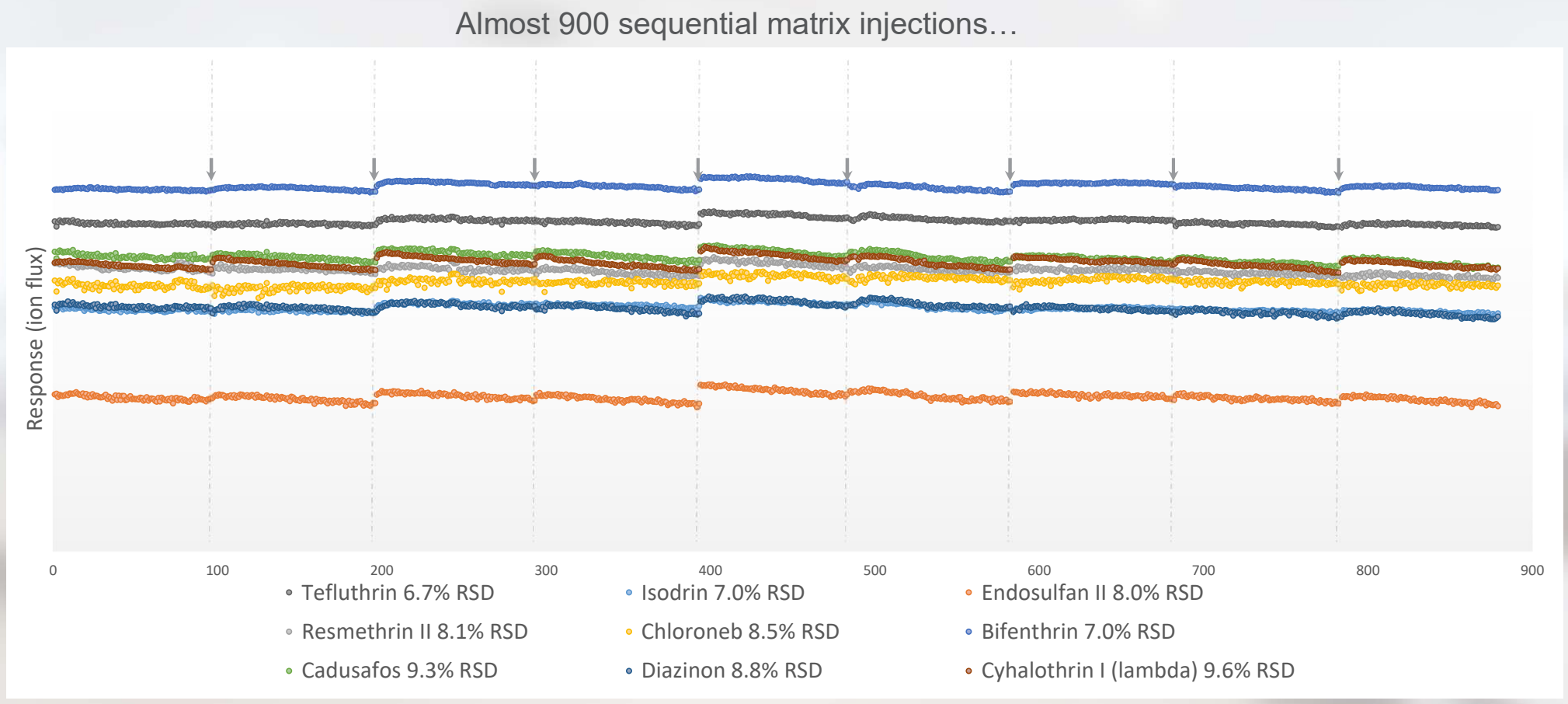
Robustness



ThermoFisher SCIENTIFIC

Software

Chromeleon™ 7.2 Chromatography Data System (CDS) software was used for instrument control, data acquisition, processing and reporting.

One-click eWorkflows™ available for simplified method, sequence creation and reporting.

Integration with Thermo Scientific™ SampleManager LIMS™, SDMS and LES to manage the complete laboratory workflow.

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Conclusions

The demonstrated stability of the advanced ion source ensures that even after hundreds of complex matrix samples, the sensitivity required is robustly maintained.

QuEChERS extraction and subsequent clean-up of over 200 pesticides from replicate analysis (n=6 each at three concentrations) of each of two sample matrices, demonstrating excellent accuracy (recovery) and precision.

Accurate, quantitative analysis of over 200 pesticides over up to 5 orders of magnitude (0.025 – 250 µg/kg), showing outstanding LODs and linear response.

Robustness displayed over ~400 consecutive injections of sample matrix (1 g/mL), with SANTE compliance at the default MRL throughout.

High sensitivity providing the real possibility to dilute the sample extract, thus limiting matrix contamination and system maintenance, leading to an increase in laboratory productivity.


Q
A