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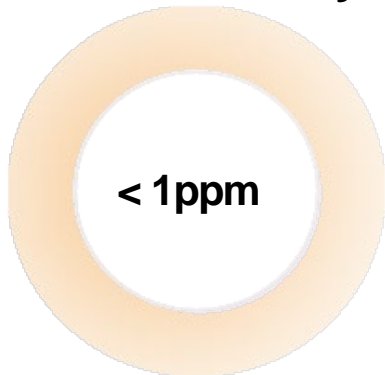


Recent developments in the analysis of pesticides and contaminants in food using LC- and GC-Orbitrap Technology

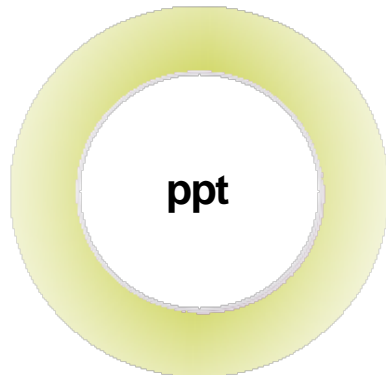
Richard Fussell & Charles Yang

Orbitrap GC-MS Family

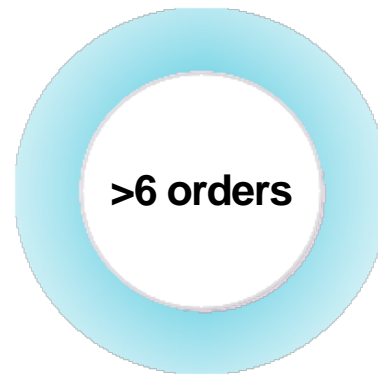
Mass Accuracy



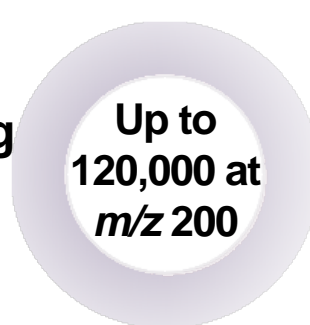
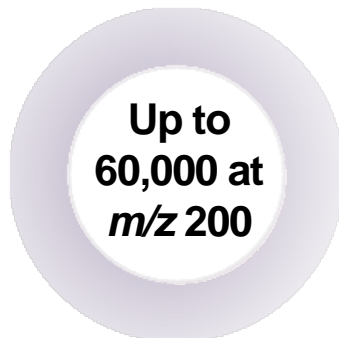
Sensitivity



Dynamic Range



Resolving Power



**2016: Exactive (Resolving Power
EI (VeV)/CI; Full-scan; Timed-SIM**

**2015 Q Exactive GC-MSEI /CI
EI(VEV)/CI Full scan, Timed SIM & MS/MS capability**



thermoscientific

APPLICATION NOTE 10584

The quantitative power of high-resolution GC-Orbitrap mass spectrometry for the analysis of pesticides and PCBs in food

Authors

Dominic Roberts,¹ Jim Garvey,²
Richard Law,¹ and Paul Silcock¹

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Runcorn, United Kingdom

²Department of Agriculture, Food
and the Marine, County Kildare,
Ireland

Goal

To demonstrate the quantitative performance of the Thermo Scientific™
Exactive™ GC Orbitrap™ mass spectrometer for the analysis of GC-amenable
pesticides and PCBs in grape and onion samples.

Introduction

The accurate and reliable determination of pesticide residues and
polychlorinated biphenyls (PCBs) in food is challenging because of the
large number of compounds and diversity of sample types involved.
The sensitivity requirements for these compounds are also demanding

Conclusions

- Limits of detection of < 2 µg/kg for 92 of the 95 compounds
- Blind analysis of a grape and onion sample showed reliable detection and accurate quantitation of spiked compounds.

Jim Garvey at NPRW 2019

- Method was successfully validated for more than 93% of the 167 pesticides & PCBs.
- The screening capability of the technology has been successfully tested with excellent results for proficiency tests

App Note 10527

EI and PCI information leads to confident chemical formulas to be proposed for molecular ions and fragments

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APPLICATION NOTE

Characterizing unknowns in food packaging using GC Orbitrap Mass Spectrometry

Authors: Dominic Roberts¹, Jesus Varela², Yves-Alexis Hammel² and Paul Silcock¹

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²Nestle, Lausanne, Switzerland

Key Words

Food packaging, Q Exactive GC, Orbitrap mass spectrometry, unknown identification, structural elucidation, food safety

Introduction

Packaging is an essential element of a safe food supply chain, with its main purpose to preserve the food it covers



manufacturers monitor and understand the health risk associated with packaging and take steps to minimize the

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Identification of non-intentionally added substances in food packaging nano films by gas and liquid chromatography coupled to orbitrap mass spectrometry



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^c Institute for Polymers, Composites and Biomaterials (IPCB), Consiglio Nazionale delle Ricerche (CNR), Via Campi Flegrei 34 Olivetti, 80078 Pozzuoli, Italy

“HRAMS has shown to be **a useful analytical approach** for the elucidation of NIAS based on the accurate mass measurement in fullscan and fragmentation modes using the Q-Orbitrap-MS analyser

.....
the combination of accurate mass matching and explaining the ions observed in the spectrum provides **a fast and confident route to the identification of unknown compounds**”

.....A.R. Fernández-Alba

Application Note 10585



Authors

Kerstin Krätschmer,¹ Cristian Cojocariu,² Alexander Schächtele,¹ Paul Silcock,² and Rainer Malisch¹

¹European Union Reference Laboratory (EURL) for Dioxins and PCBs in Feed and Food, Freiburg, Germany

²Thermo Fisher Scientific, Runcorn, UK

Goal

To demonstrate the quantitative performance of the Thermo Scientific™ Q Exactive™ GC Orbitrap™ mass spectrometer for the analysis of short- and medium-chained chlorinated paraffins in salmon samples.

Introduction

The coupling of gas chromatography (GC) to high-resolution mass spectrometry (HRMS) using Orbitrap™ technology opens a broad spectrum of possible applications in environmental and food/feed analysis. Although known for several decades and widely used as plasticizers or flame retardants¹ short-chain chlorinated paraffins (SCCPs) have been only recently

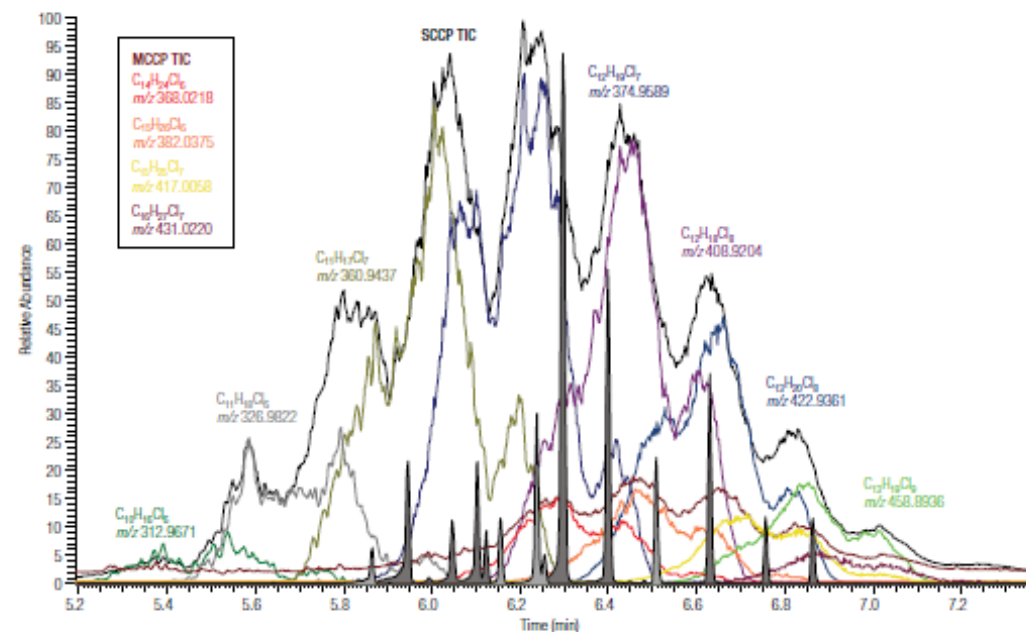
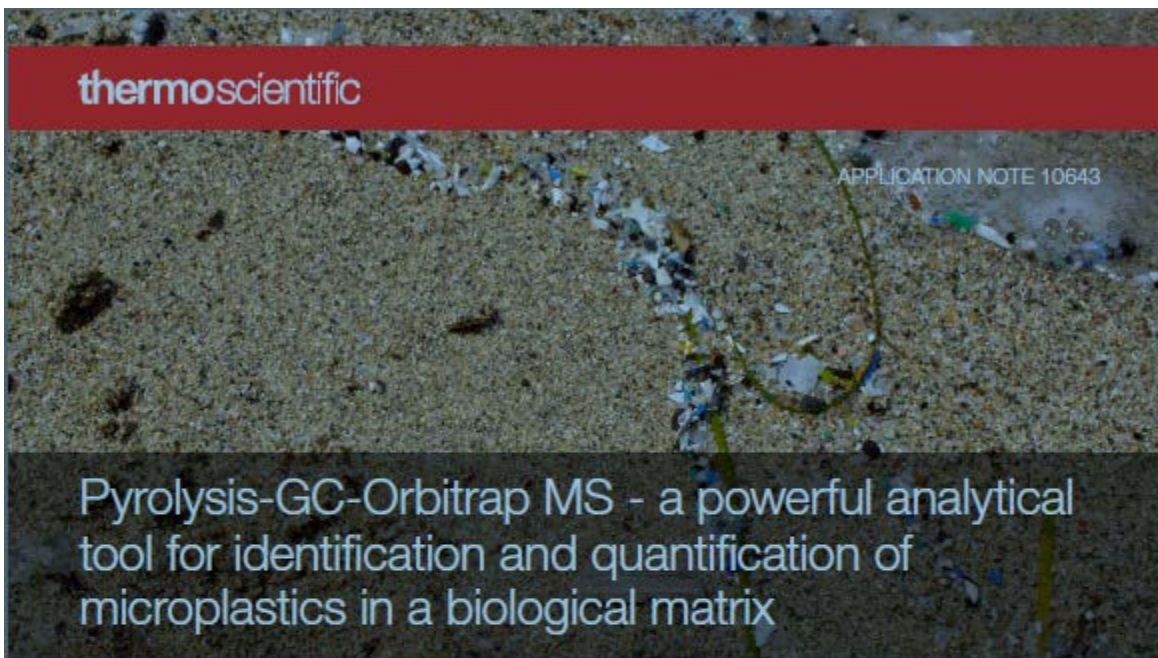


Figure 1. Overlaid chromatograms of SCCP, MCCP, and PCB standards with added extracted ion chromatograms of selected CP homologues measured with the Q Exactive GC Orbitrap GC-MS/MS system.

Kerstin Krätschmer

“Orbitrap GC, with high resolution allows selective separation of CP groups which is a pre-requisite to good quantiation.....”

GC Orbitrap: Helping to Identify and Quantify Microplastics



Authors

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Ørjan Bjørøy², Wibke Peters³,
Cristian Cojocariu⁴, and Tanja Kögel²

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Germany

²Institute of Marine Research,
Bergen, Norway

³S+H Analytik, Mönchengladbach,
Germany

⁴Thermo Fisher Scientific, Runcorn,
United Kingdom

Goal

The purpose of the experiments described in this work was to assess the applicability of pyrolysis-gas chromatography-Orbitrap™ mass spectrometry for the qualitative and quantitative analysis of plastic polymers in complex biological matrices.

Introduction

Plastics are synthetic organic polymers, commercially introduced on a large scale starting in the 1950s. Single-use plastics (grocery bags, food packaging, bottles, utensils) are persistent pollutants making up approximately 40% of beach litter¹. This litter eventually ends up in the marine environment, with an estimated 8 million metric tons of plastic waste entering the oceans worldwide every year². Most plastics have a very long degradation time, and for a timespan up to centuries they end up as macro-, micro- and

Collaboration with the Institute of Marine Research , Bergen Norway

“The Exactive GC Orbitrap GC-MS system in combination with pyrolysis has proven to be a very promising analytical technique that opens new possibilities with respect to the analysis of microplastic polymers in biological matrices



Multi-Shot Pyrolyzer (Frontier EGA/PY-3030D) with Auto-Shot Sampler (AS-1020E) coupled to an Exactive GC Orbitrap mass spectrometer



ASMS 2016
* BioPharma Option

ASMS 2019

ASMS 2017

ASMS 2014

HUPO 2013

ASMS 2011

SOFT 2014



**Orbitrap
Exploris 480**

Q Exactive HF-X*

Q Exactive HF*

Q Exactive Plus*

Q Exactive

Q Exactive Focus

*Ultimate
Performance for
rapid and deep
analysis of data
quality*

*Maximum
Performance for
rapid and deep
analysis – Superior
data quality*

*High-end Omics
workhorse -
Productivity is
key*

*Workhorse for
"Omics" lab &
Small molecule
Structure ID*

*Method
developmt for
Screening / Entry
level*

*Quanfirmation for
rapid screening
and quantitation*

Customers:

Customers:

Customers:
Core labs
performing routine
Peptide ID and
Quan

Customers:
Metabolomics labs,
Pharma, Met ID

Customers:
Small molecule
CROs, small
universities, EFS,
Forensic Customers

Customers:
Small molecule
CROs, small
universities, EFS,
Forensic Customers

**High End core labs in
Academia performing
higher demanding
Peptide ID and Quan
Analysis**

**High End core labs
performing
demanding Peptide
ID and Quan
Analysis**

VALUE

PERFORMANCE



ThermoFisher
S C I E N T I F I C

See the Pesticides You've Been Missing with a Thermo Scientific Orbitrap ID-X Tribrid Mass Spectrometer and AcquireX

Charles Yang (Anastasia Kalli, PhD)
Senior Marketing Specialist, Thermo Fisher Scientific

Challenges in Pesticide Analysis

- Large and diverse number of pesticide residues
- Wide variety of sample matrices
- Maximum residue levels requirements
- Interfering matrix and solvent/matrix background ions
- False-positive and false-negative identifications
- Presence of unexpected pesticides



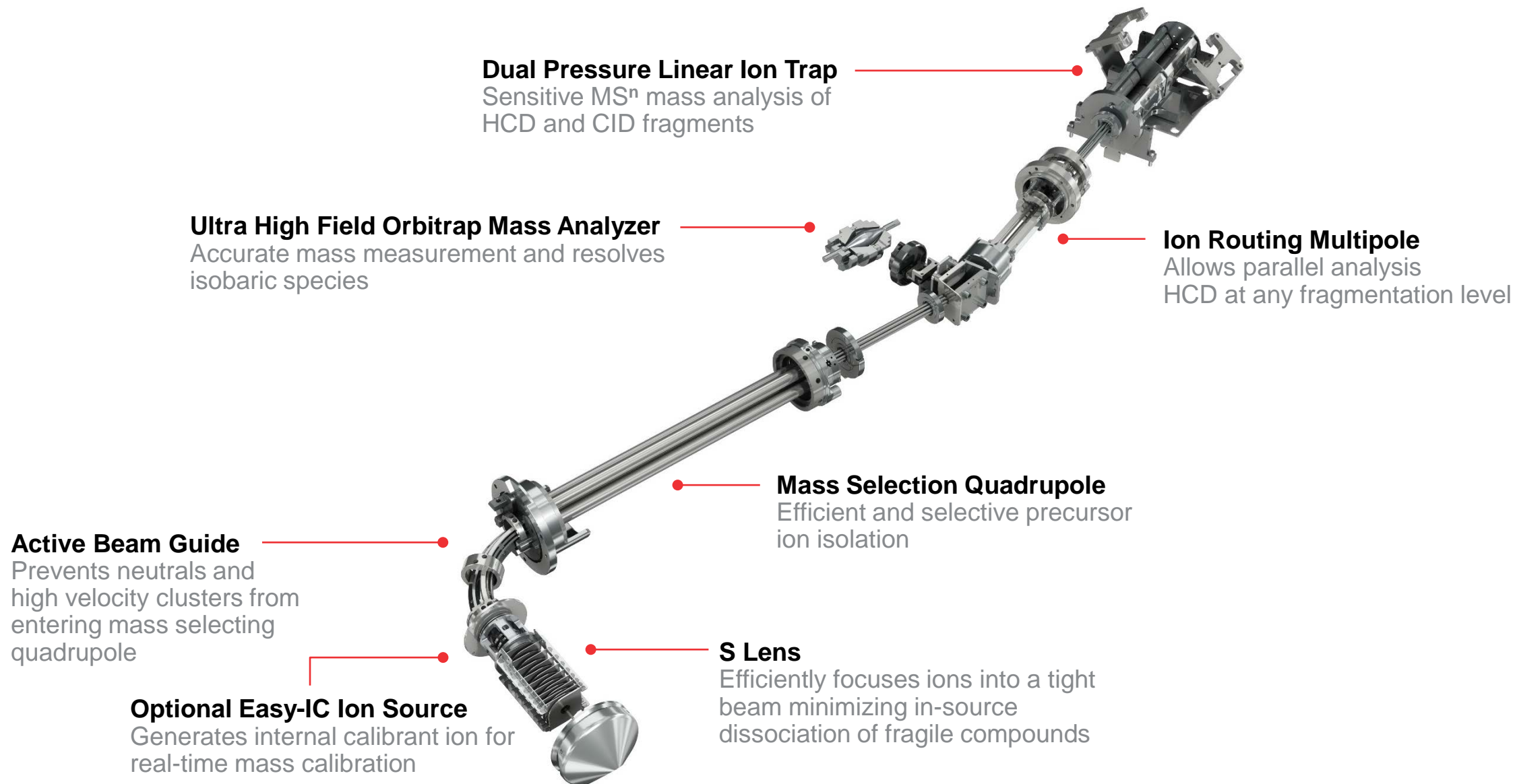
Orbitrap ID-X Tribrid Mass Spectrometer



**Thermo Scientific™ Orbitrap ID-X™
Tribrid™ Mass Spectrometer System**

- ❑ Excellent mass accuracy and resolution to minimize interferences and increase confidence in identifications
- ❑ Streamlined calibrations with improved mass calibration for ions at $m/z < 200$
- ❑ AcquireX™ for automatic background exclusion to increase productivity
- ❑ AcquireX™ for improved analyte identification

Thermo Scientific Orbitrap ID-X Tribrid Mass Spectrometer





Matrices

- Strawberry
- Baby Food (mix of bananas, apples and strawberries)

Sample Preparation

- QuEChERS extraction

Chromatographic Separation

- Thermo Scientific™ Vanquish™ Flex UHPLC system
- Thermo Scientific™ Accucore™ C18 aQ column

Detection

- Orbitrap ID-X Tribrid mass spectrometer
- AcquireX intelligent data acquisition workflow

Data Analysis

- Thermo Scientific™ TraceFinder™ software
- mzCloud™

AcquireX Workflow for Background Exclusion

Matrix Blank Injection

Exclusion List Generation

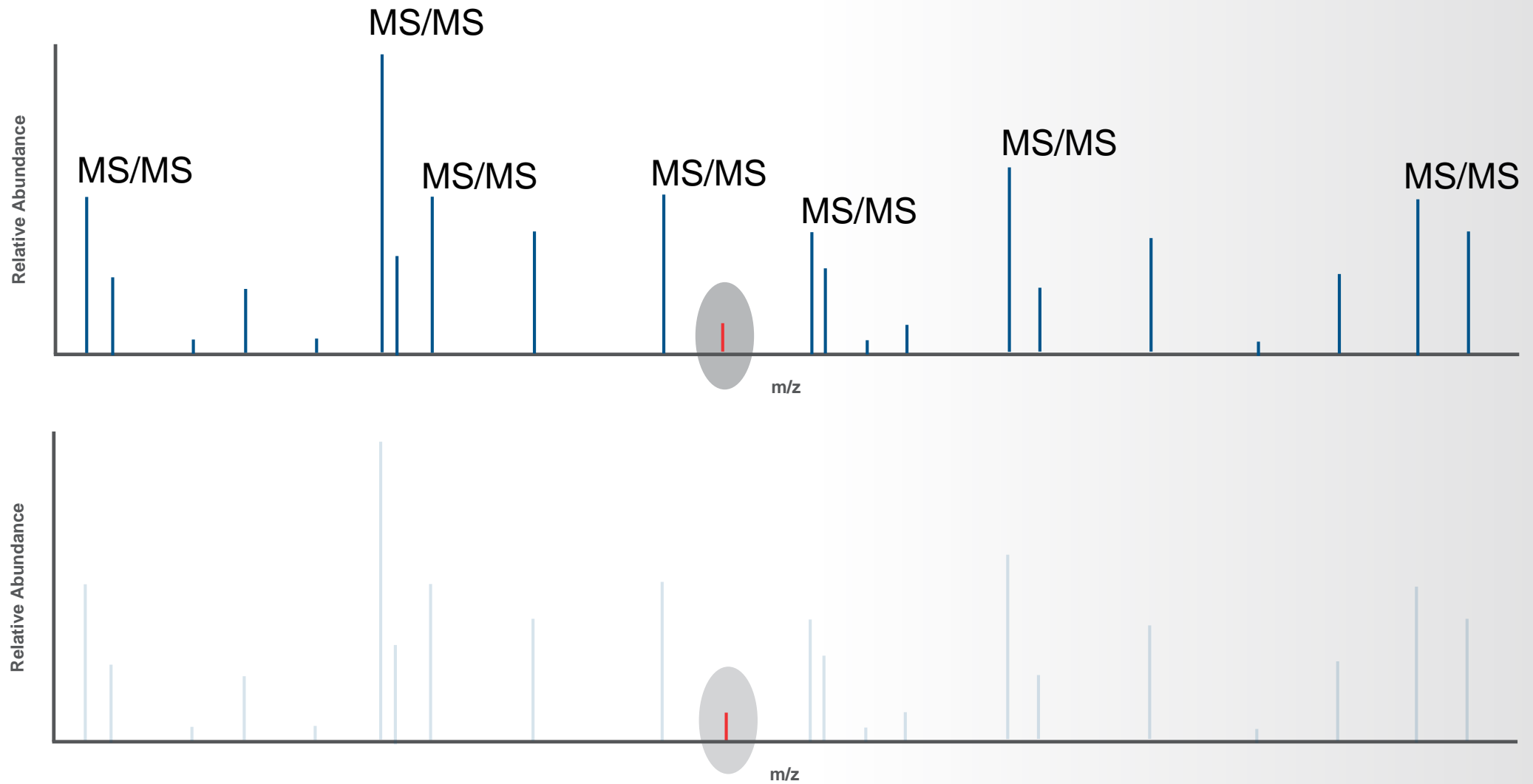
Instrument Method Update with Exclusion list

Sample Analysis with Updated Method to Exclude Background Ions Fragmentation

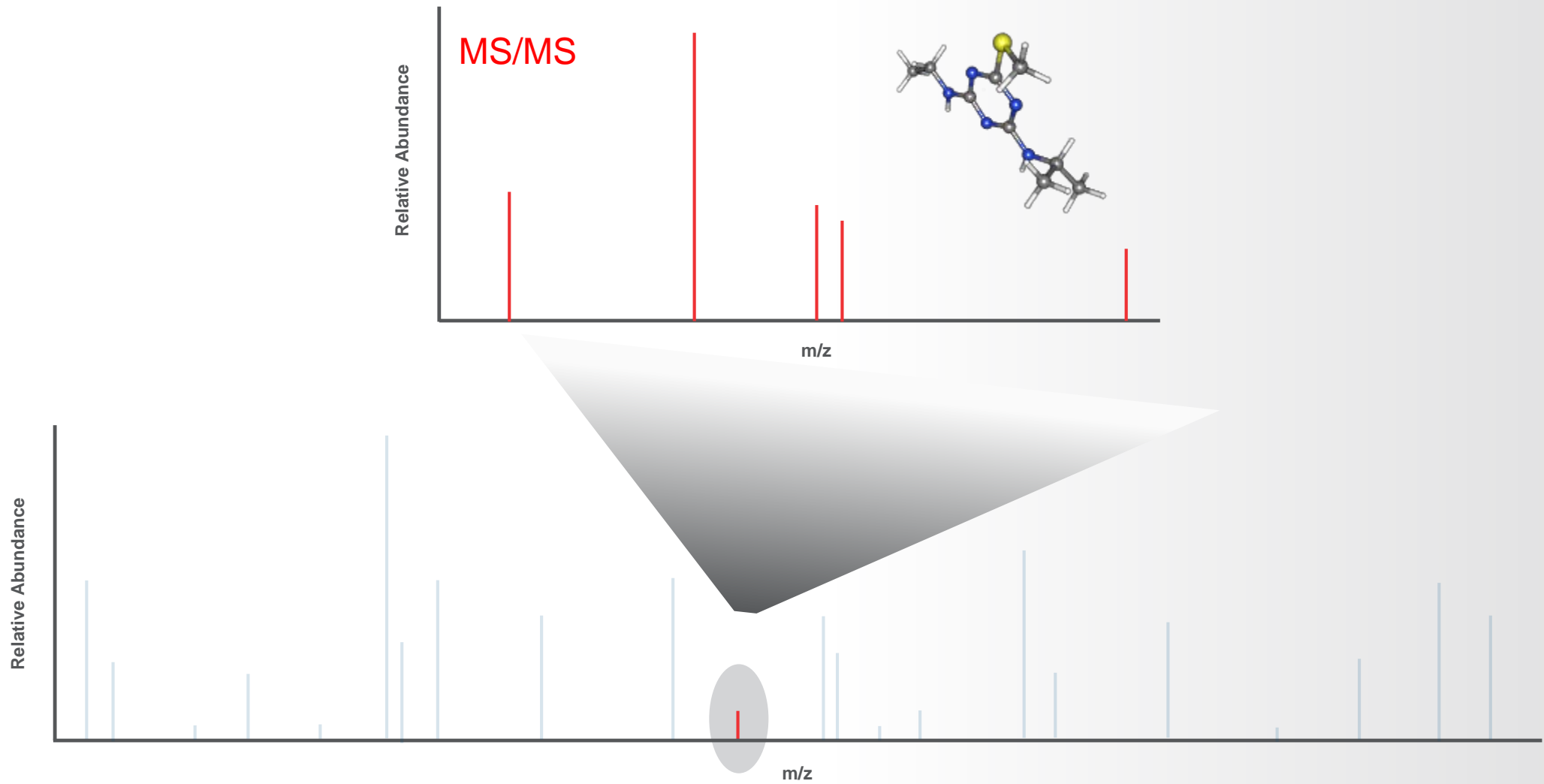


AcquireX Workflow Allows Intelligent Data Acquisition

Background Ions During Data Dependent Acquisition

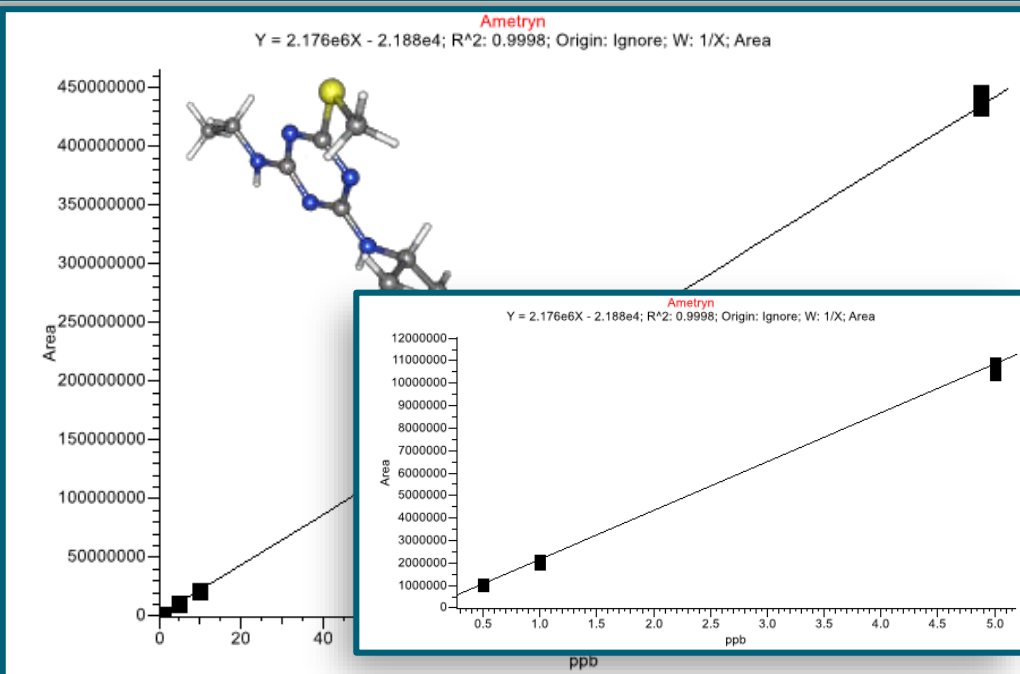


Background Ions During Data Dependent Acquisition



Pesticide Residues Quantitation (0.5 – 200ppb) –Ametryn and Ofurace

Calibration Range Ametryn

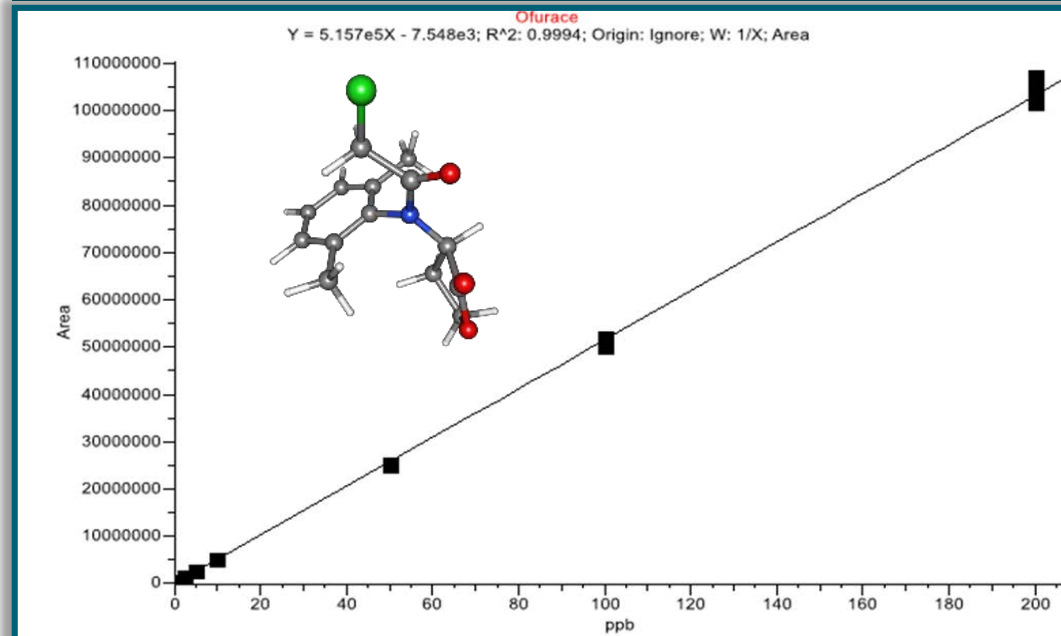


LOQ at 0.5 ppb



	% Diff	% CV	% RSD
Injection 1	-0.07	4.48	4.38
Injection 2	-6.27	4.48	4.38
Injection 3	-8.01	4.48	4.38

Calibration Range Ofurace



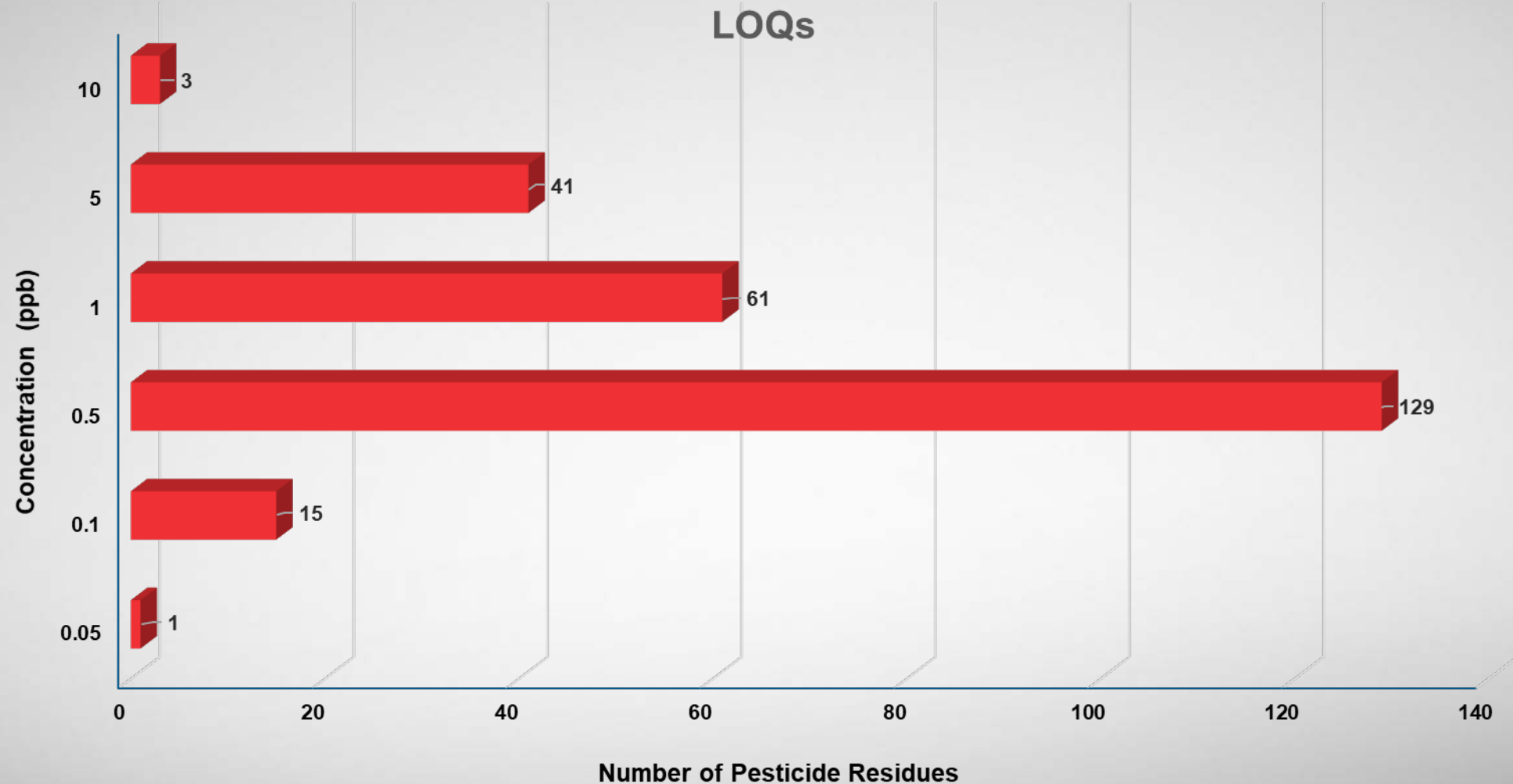
QC at 0.5 ppb



	Calculated Amount (ppb)	% Diff	% CV
Injection 1	0.507	1.37	2.47
Injection 2	0.513	2.55	2.47
Injection 3	0.490	-2.08	2.47

Confident Quantitation Excellent Reproducibility

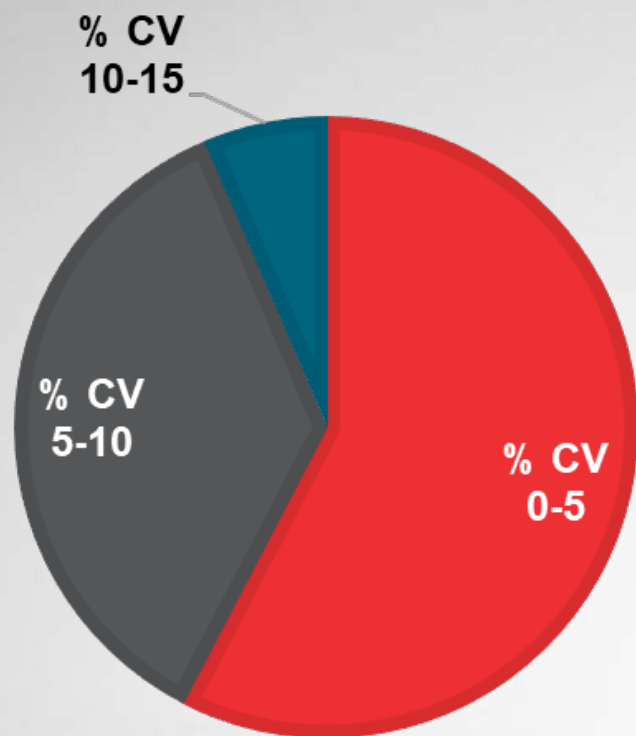
LOQ Levels in Strawberry Matrix (250 pesticides)



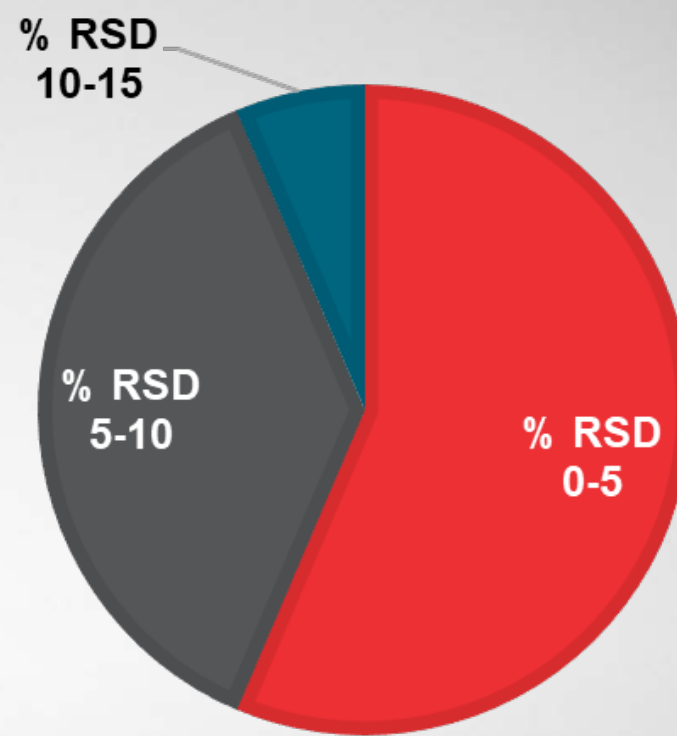
LOQ Levels at or below 1 ppb for 82% for all Pesticide Residues Tested



% RSD and % CV per Number of Analytes at LOQ



% CV	Number of Pesticide Residues
0-5	144
5-10	90
10-15	16

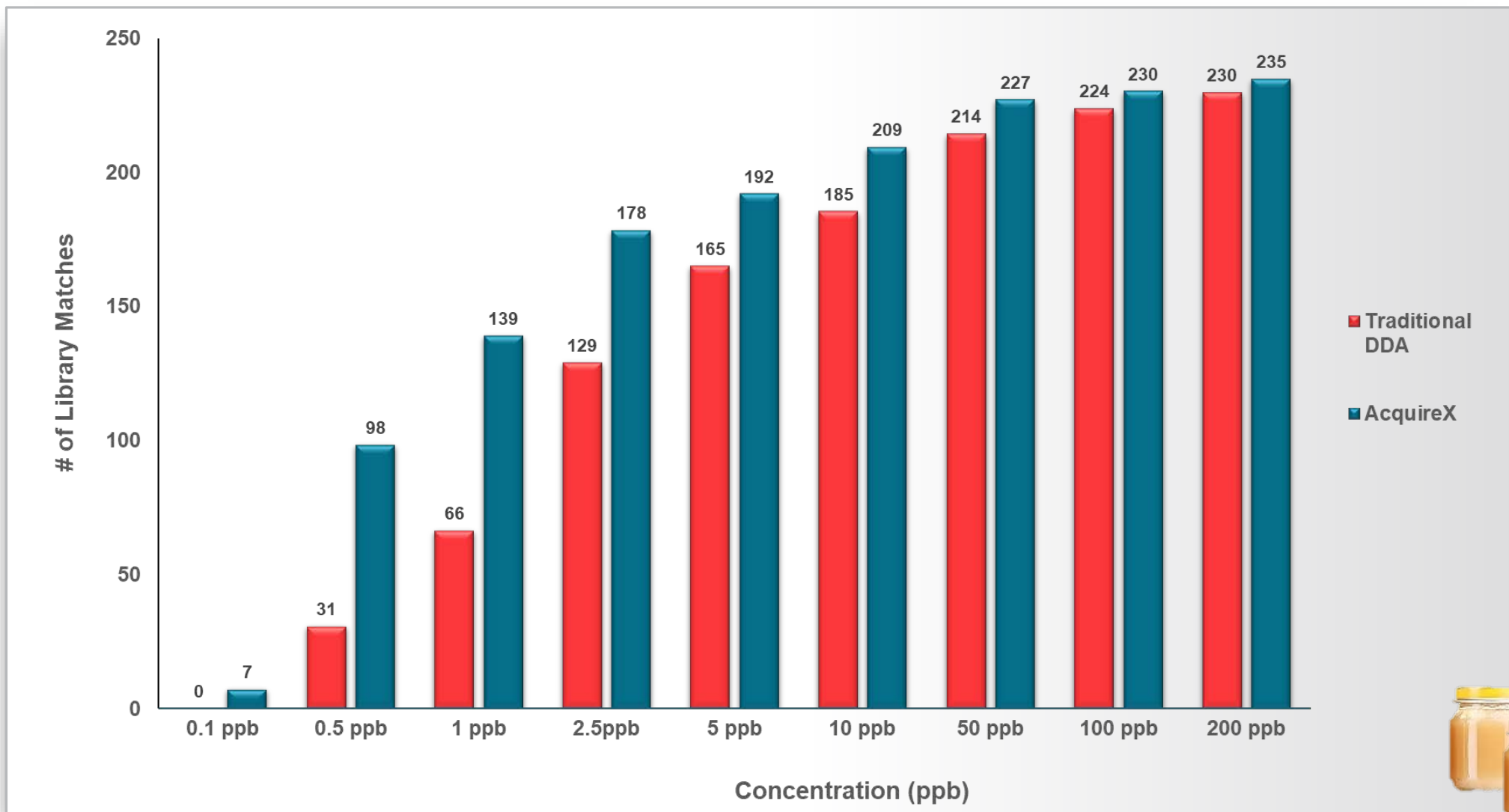


% RSD	Number of Pesticide Residues
0-5	141
5-10	93
10-15	16



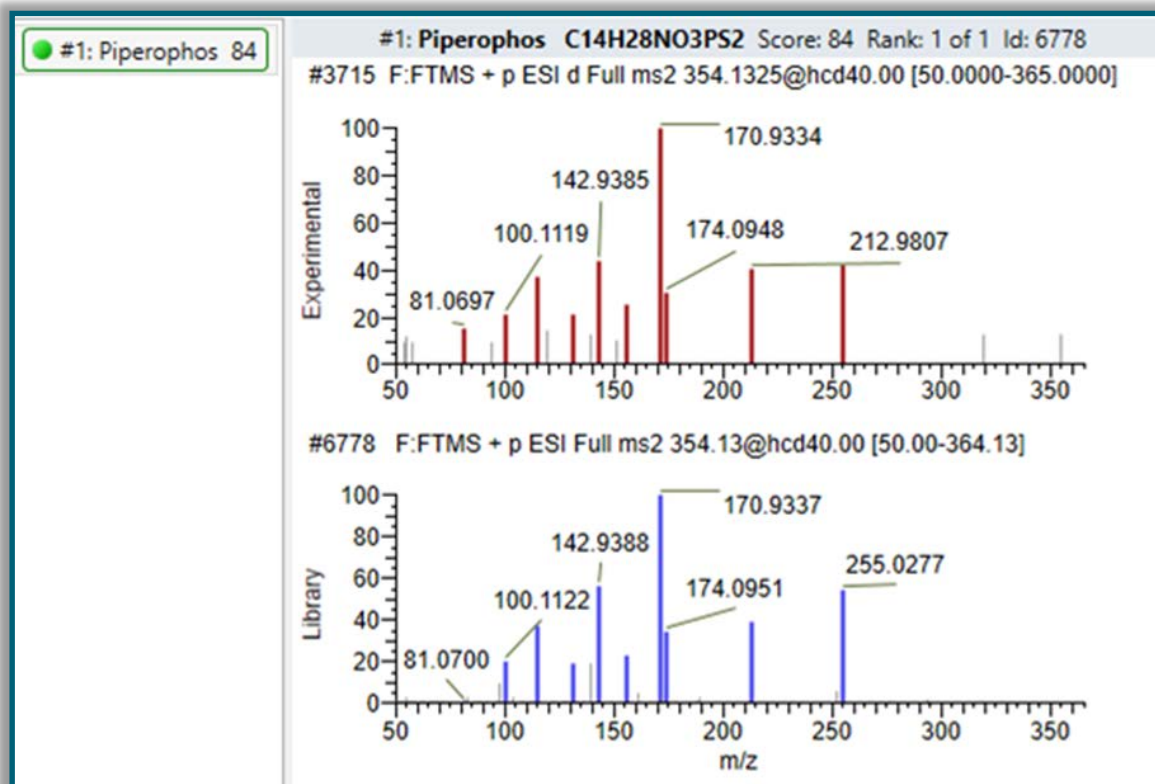
Excellent Reproducibility at LOQ Levels

Screening Results with Traditional DDA vs AcquireX Workflow

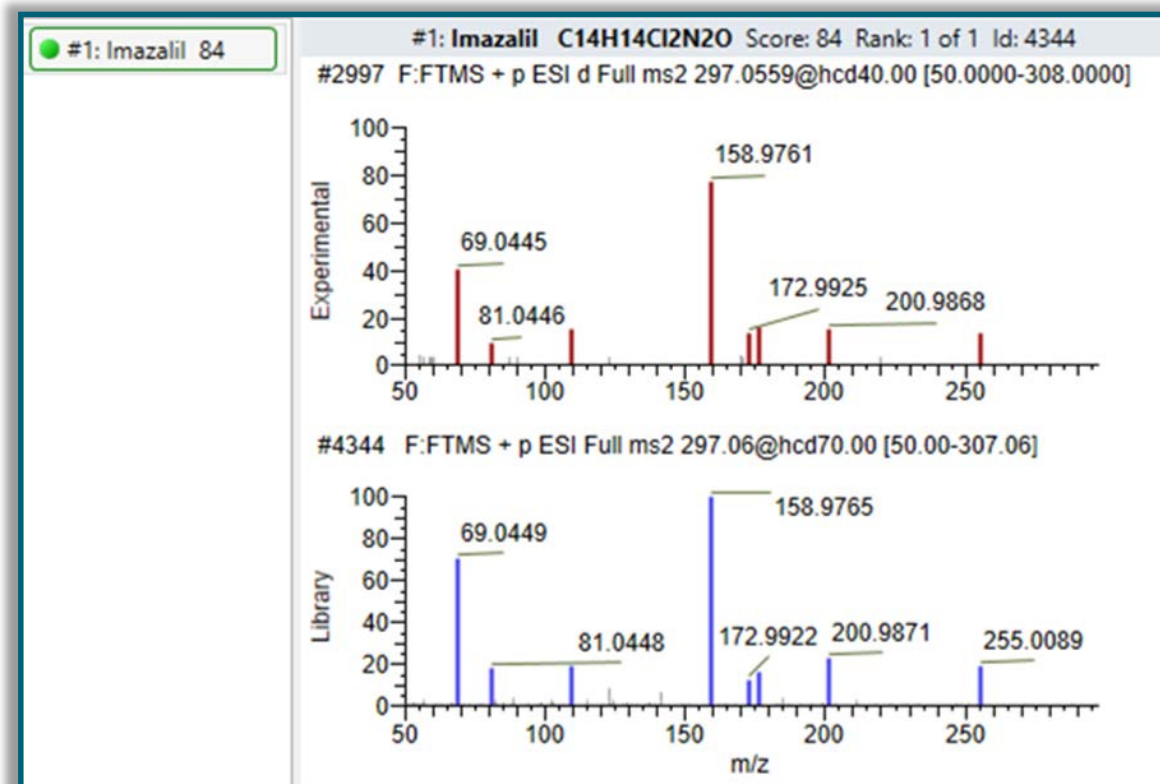


AcquireX Workflow Significantly Increases the Number of Identified Pesticides

Library Matches through mzCloud Offline Libraries



Piperophos at 0.5 ppb



Imazalil at 1.0 ppb

Confident Pesticide Identification at Low ppb Levels



Conclusions

- ❑ **The Orbitrap ID-X Tribrid mass spectrometer delivers confident, accurate and reproducible quantitation**
- ❑ **AcquireX workflow enables the generation of background exclusion list in an automated fashion to increase productivity, efficiency and quality of results**
- ❑ **AcquireX workflow significantly increases the number of identified pesticide residues at low ppb levels compared to traditional DDA**



