



The Food and Environment
Research Agency

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Multi-residue determination of pesticides in food using the Thermo Scientific TSQ 8000 GC-MS/MS system

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Presentation outline



- Fera at a glance
- Collaboration with Thermo Scientific Pesticides Centre of Excellence
- Some examples of GC-MS/MS applications
- Summary of our experience with Thermo Scientific GC-MS/MS systems

Fera at a glance

- Established in 2009



Centenary 1914 – 2014 '100 Years of Science Solutions'

- NRL for Fruits & Vegetables, Cereals, SRMs
- Analyse 50,000 samples pa
- (estimate ~10,000 samples for pesticides in 2014-15)
- LC-MS/MS, GC-MS/MS
- LC-TOF & QTOF, GC-QTOF



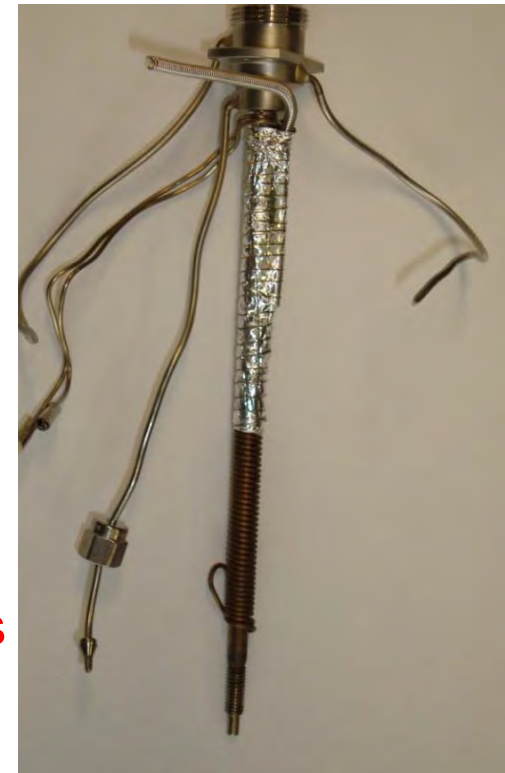
GC- MS/MS Requirements



- High sensitivity to streamline methods but still achieve low detection limits
- High through-put of 'dirty' QuEChERS extracts (ACN) 24/7
- Service back-up
- Maintenance schedules to reduce instrument downtime
 - TSQ Quantum XLS, installed 8th March 2011
 - TSQ 8000, 2013 & access to TSQ 8000 Evo 2014

Long term robustness of TSQ Quantum XLS performance

- Performance record over a period of 1 year
- Analysed ~ 200 batches of samples
- ~ 6000 matrix injections (PTV-backflush)
- Kidney, liver, fish, meat, crustaceans, milk
- Solvent extraction and HPGPC clean-up
- Response does vary but is always sufficient
- System operated every day, except for 2 occasions requiring service engineer
- **Surveillance team satisfied - purchased 3 instruments**



Routine maintenance strategy

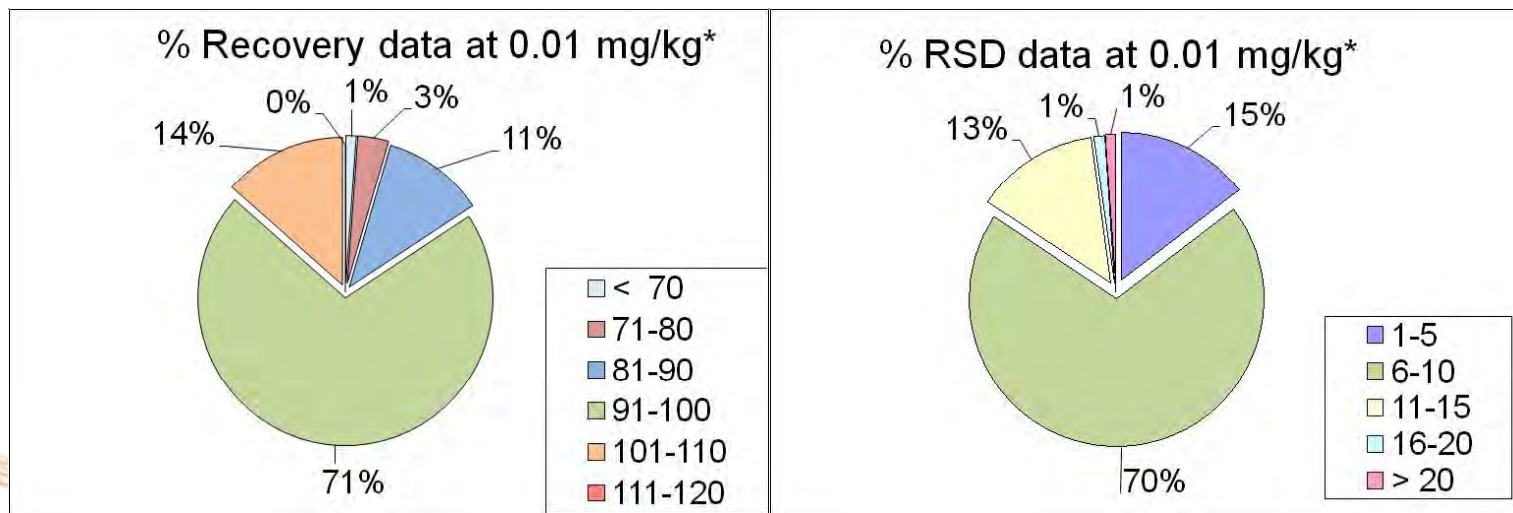
- Septum, liner and ion volume replaced daily
- Baffled liners and ion volumes cleaned and re-used
- Capillary column – replaced ~ 6 months
- Retention gap replaced ~1-2 months
- Source cleaned ~ once per month
- QO cleaned at PM visit



Summary of validation data (1 μ L splitless) for QuEChERS acetonitrile extracts



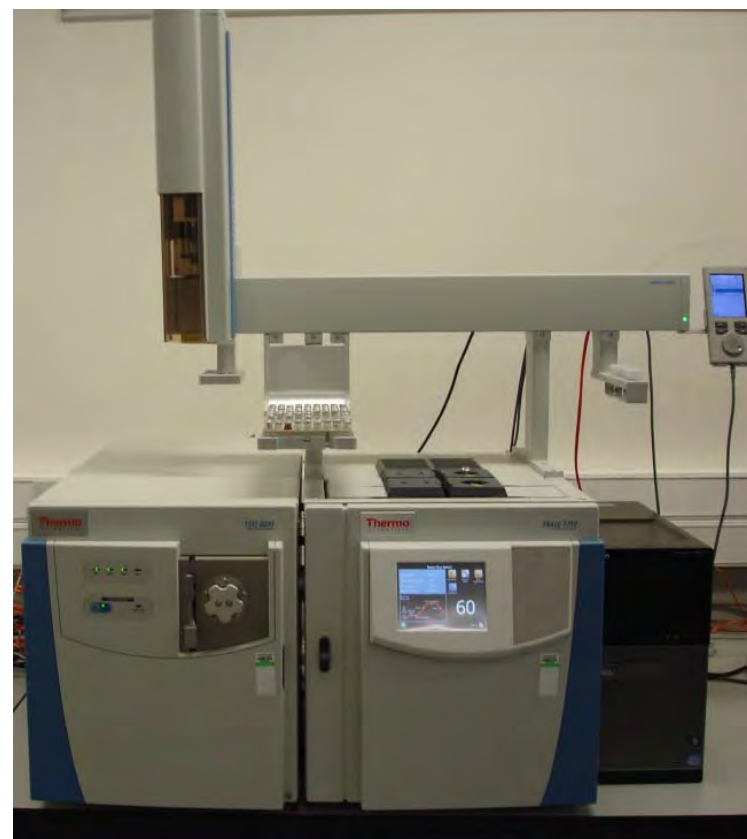
QuEChERS extraction (citrate buffer/d-PSA) of fruit preserve



Except chlorothalonil, recovery and % RSD data for the other 95 compounds met EU DG SANCO validation criteria

Why the TSQ 8000 MS/MS with TRACE™ 1310 GC) ?

- Smaller footprint
- 13 AMP power requirement
- Modular –easily change injectors, add detectors
- New source design for reduced maintenance
- *Is performance compromised?*



QuEChERS Acetonitrile extracts: compatibility with GC?



- High expansion coefficient-limited injection volume

Consider

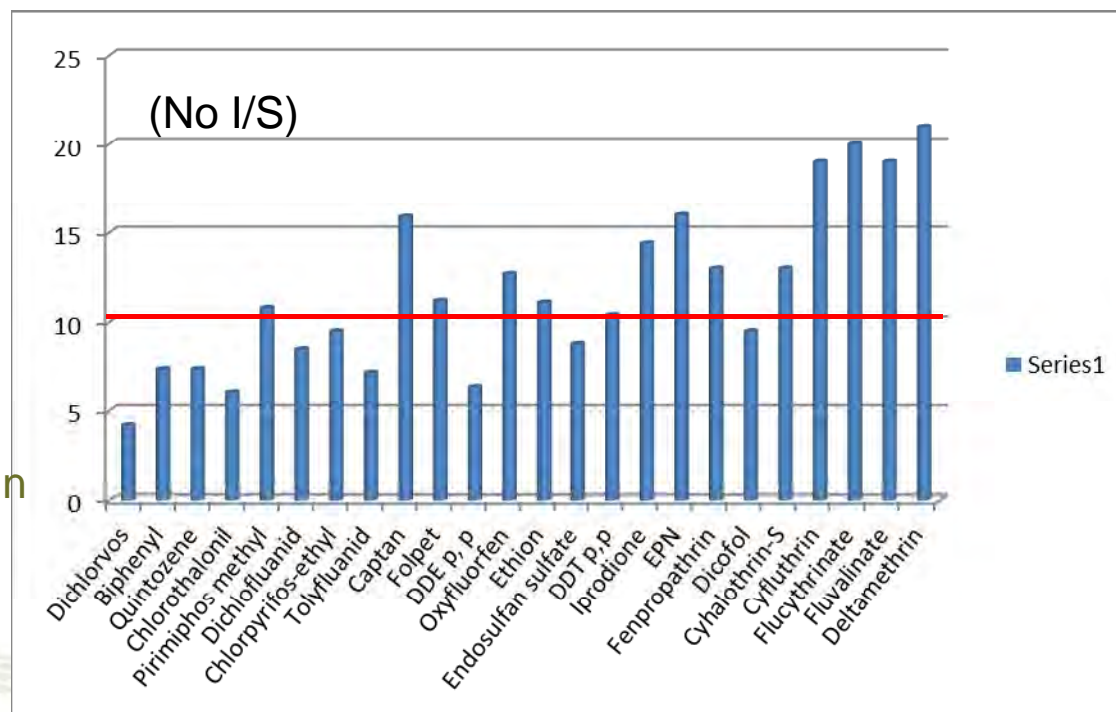
- (1) Offline solvent exchange - requires care to avoid losses of volatile or unstable pesticides
 - (2) LVI with higher capacity liners or liner packing materials (potential for adsorption of pesticides)?
- but,
- (3a) Will repeated injection of acetonitrile extracts lead to a decrease in column efficiency?
 - (3b) How quickly will matrix extracts contaminate the GC-MS system

Repeatability using the baffled liner

[10ul (1/10 dilⁿ with EtAc) speed controlled solvent vent]

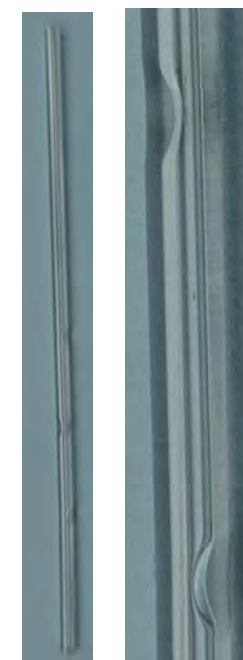
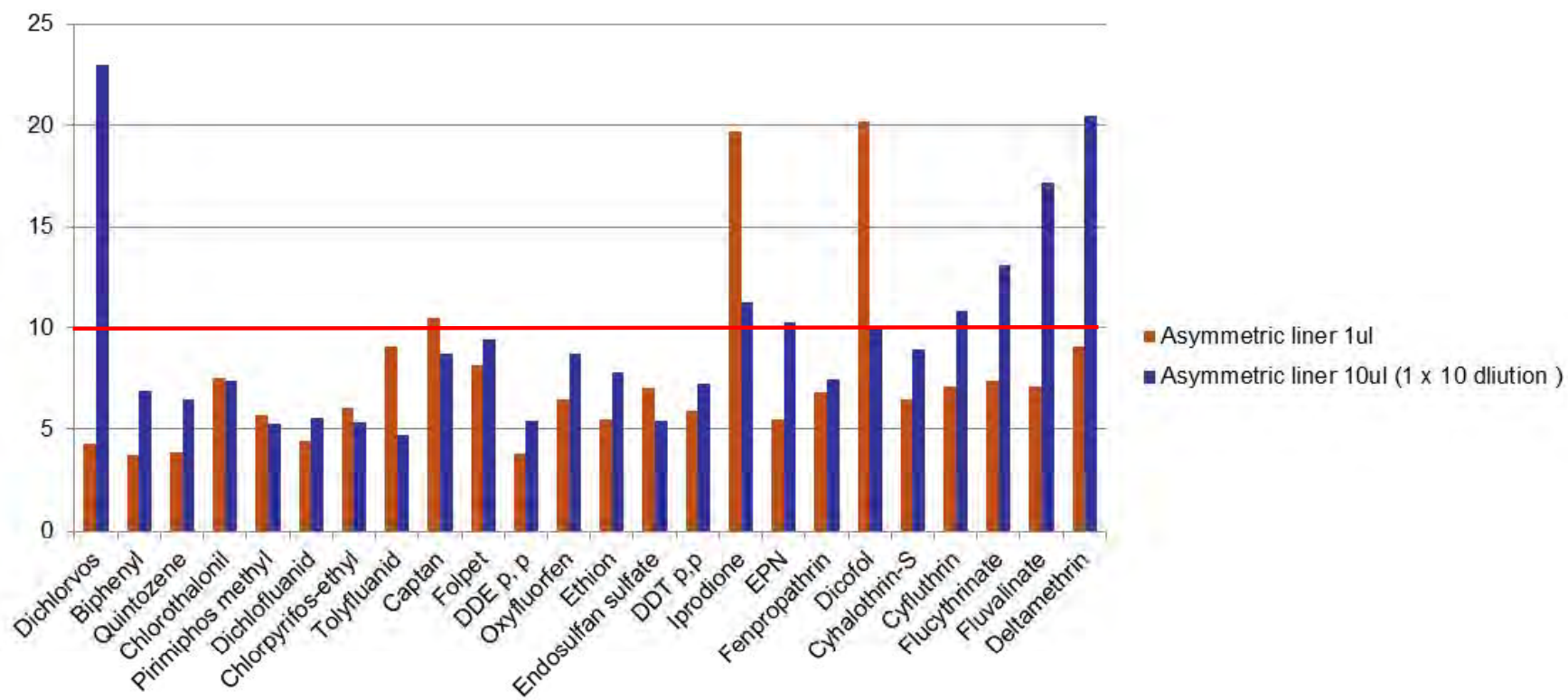
Liner: PTV Baffle Liner (Siltek), Deactivated, 2 mm ID x 2.75 mm OD x 120 mm Length

- Injection mode: PTV 10 μ L
- Carrier mode: constant flow (1.0mL)
- Inlet temperature: 60°C
- Split flow: 50 mL/min
- Splitless time: 2 min
- PTV injection time: 0.15 min
- PTV injection flow: 100 mL/min
- PTV transfer rate: 10°C/sec
- PTV transfer temperature: 300°C
- PTV –back flush- transfer time: 12 min
- PTV cleaning rate 14.5°C
- PTV cleaning temperature: 350°C
- PTV cleaning time: 20 min
- PTV cleaning flow: 50 mL/min



Repeatability using asymmetric liners

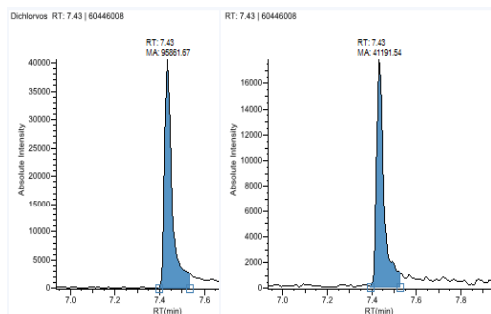
Quechers extracts of baby food (no IS)



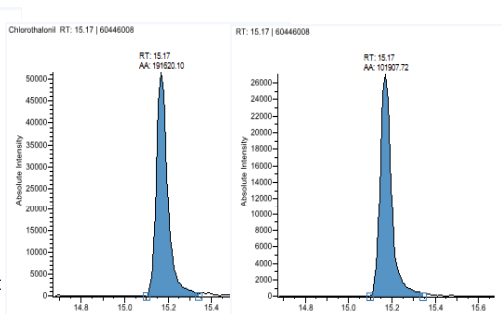
Example SRMs (10 pg on column)



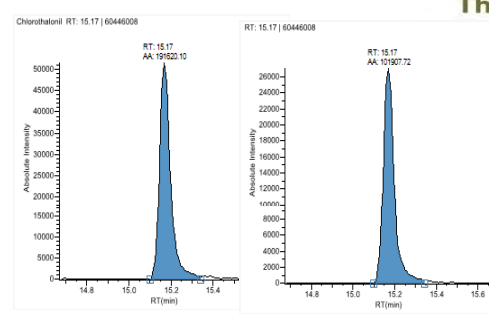
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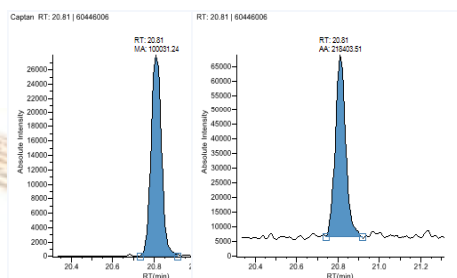
Dichlorvos
(m/z 185-93, 186.9-93)



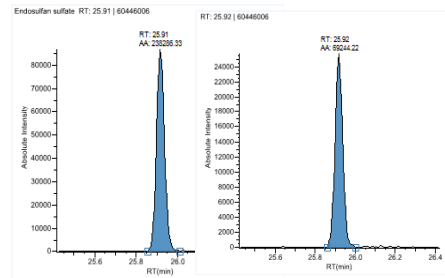
Chlorothalonil
(m/z 265.8-133, 265.8-170)



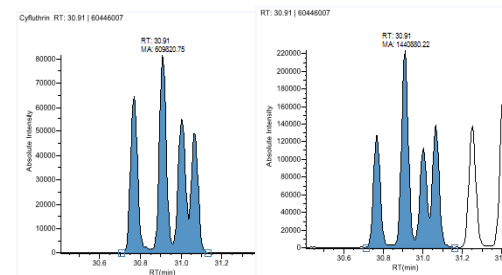
Pirmiphos-methyl
(m/z 290.1-125, 290.1-233)



Captan
(m/z 149-70, 149-105)



Endosulfan-sulphate
(m/z 271.7-236.8, 238.7-203.9)



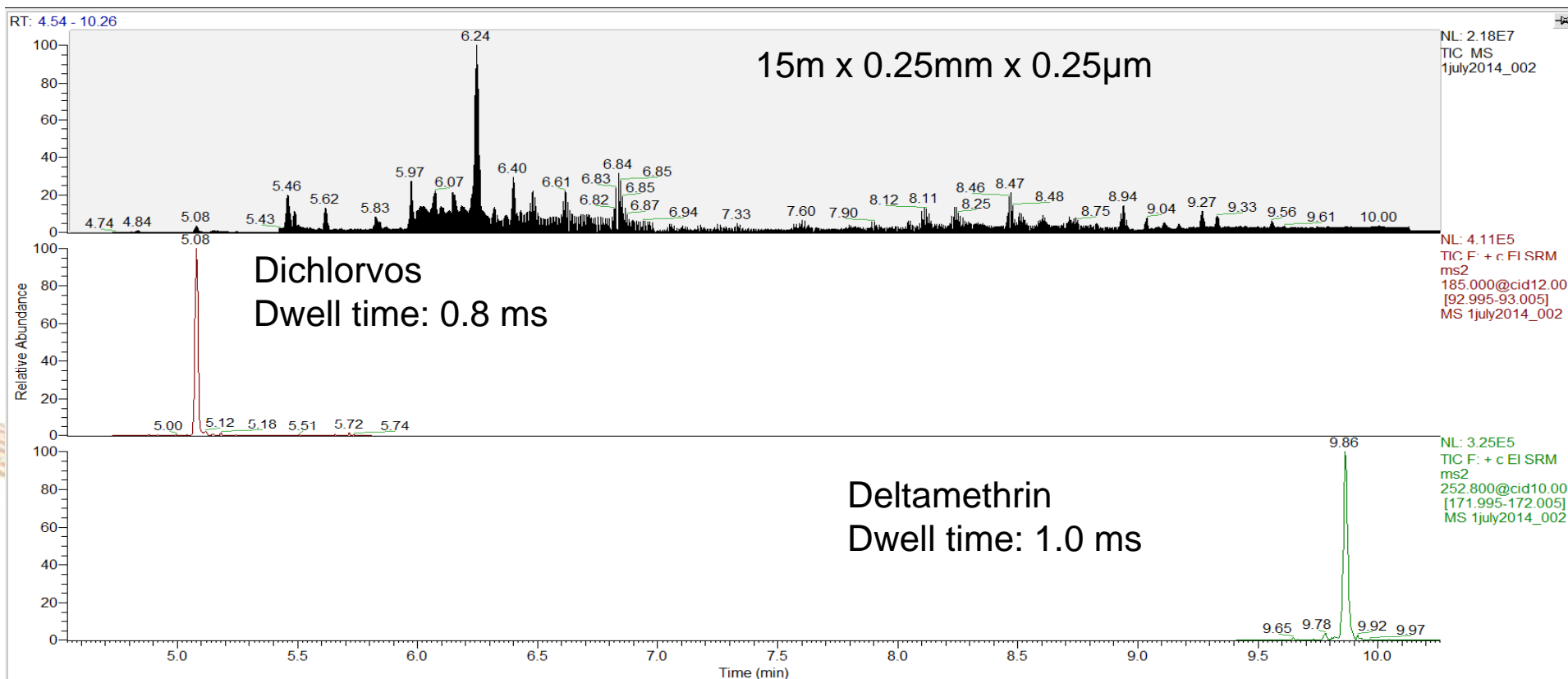
Cyfluthrin
(m/z 226.0-206.1, 163.0-127)

Further work :TSQ 8000 Evo GC-MS/MS

- Latest introduction June 2014
- EvoCell for fast transition speeds with high sensitivity
- How can you apply this to multi-residue analysis?

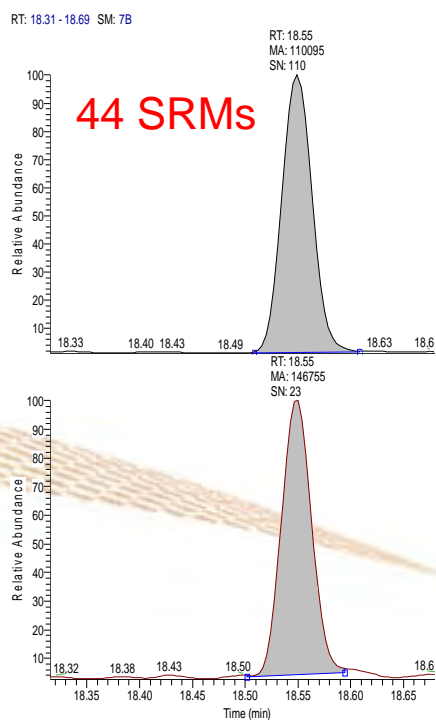


Fast analysis <10 mins (~150 pesticides) 8 SRMs/ Compound

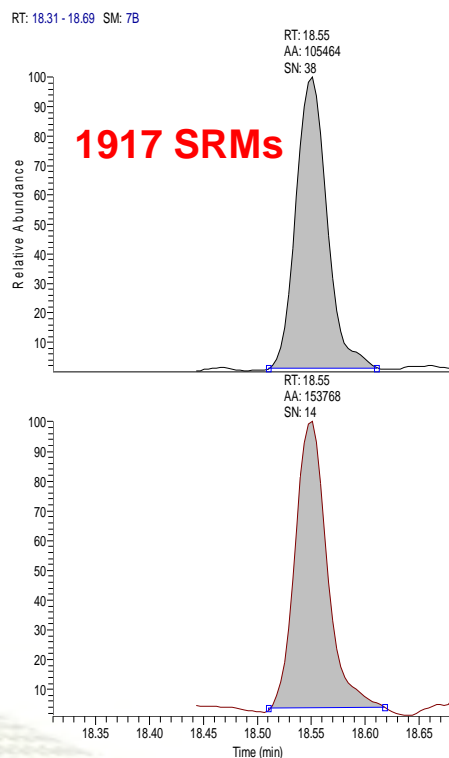


Fast acquisition: does it still allow good sensitivity?

109 ms dwell time



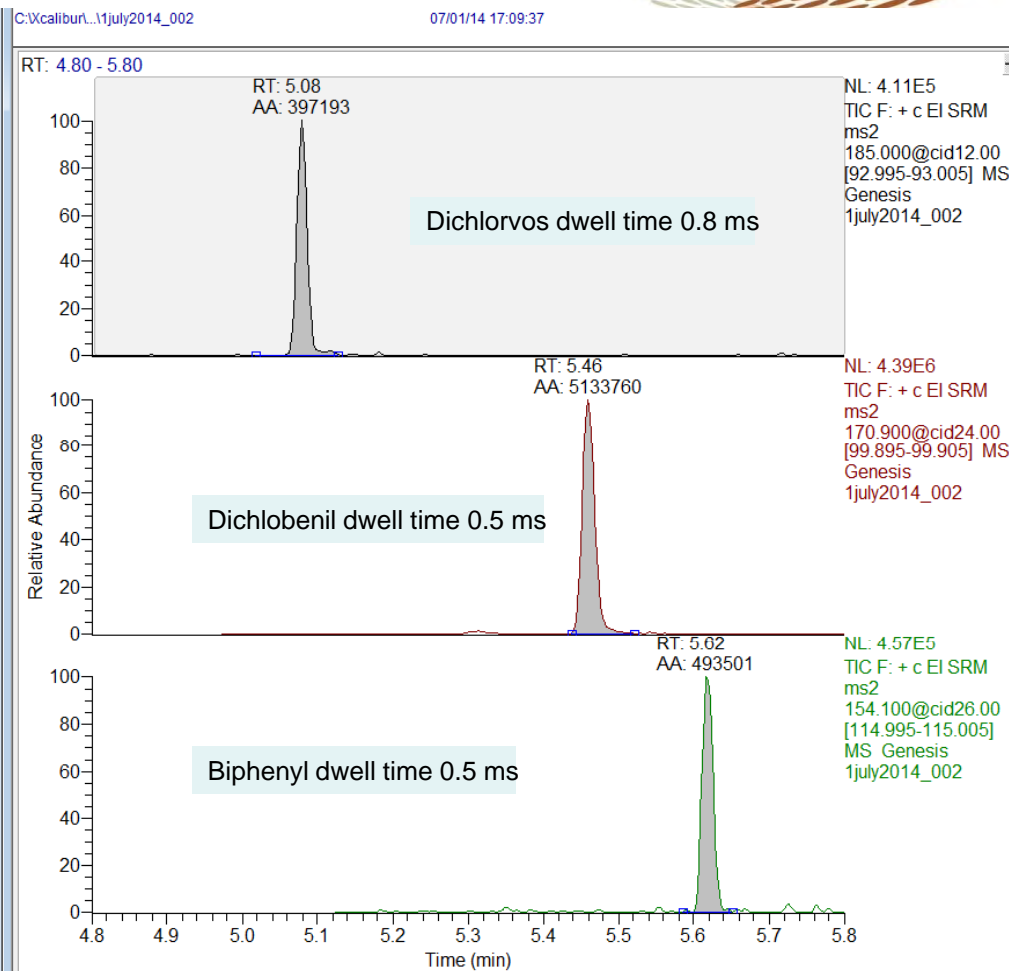
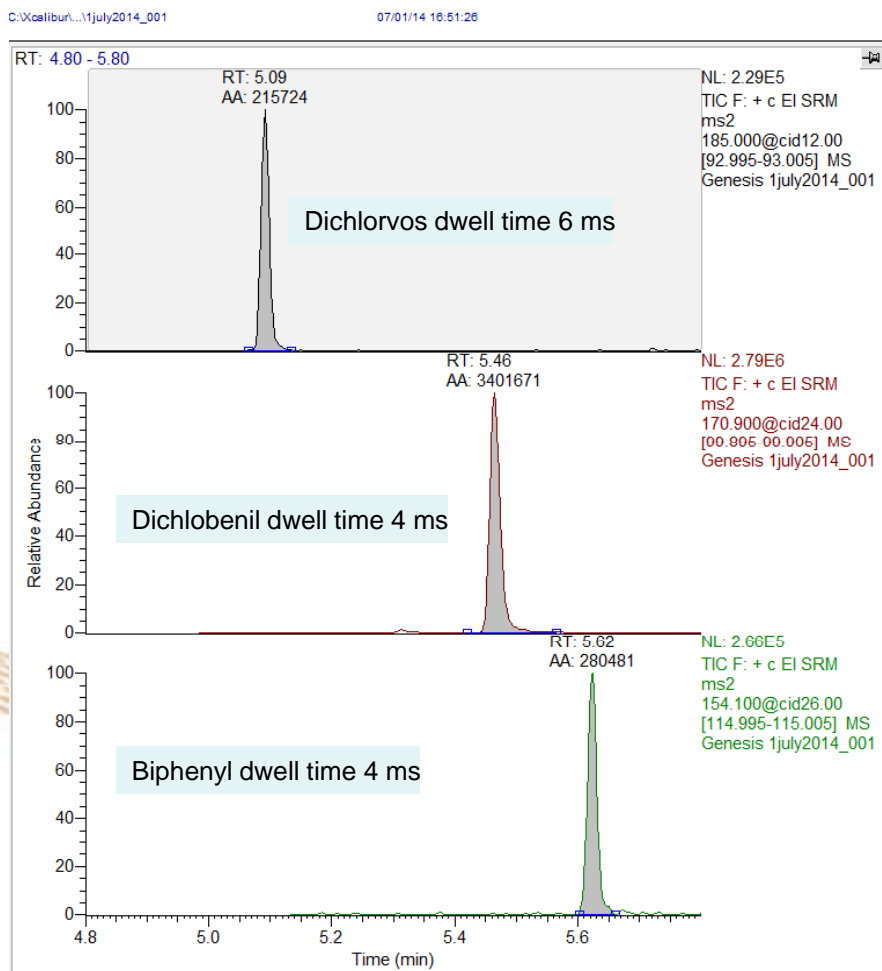
1.4 ms dwell time



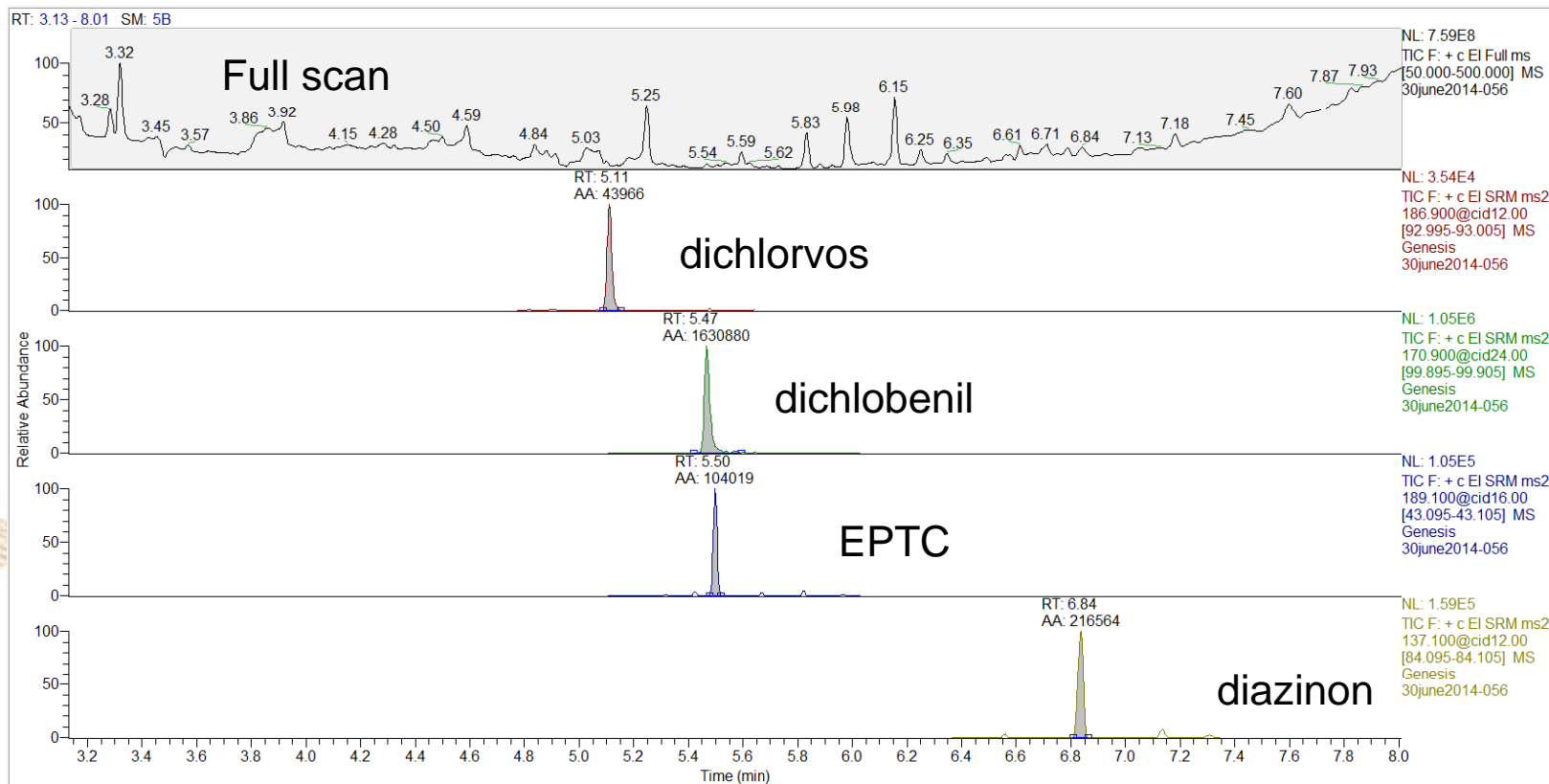
Pyraclofos
green tea 10 ng/g

2 SRMs/compound

8 SRMs/compound



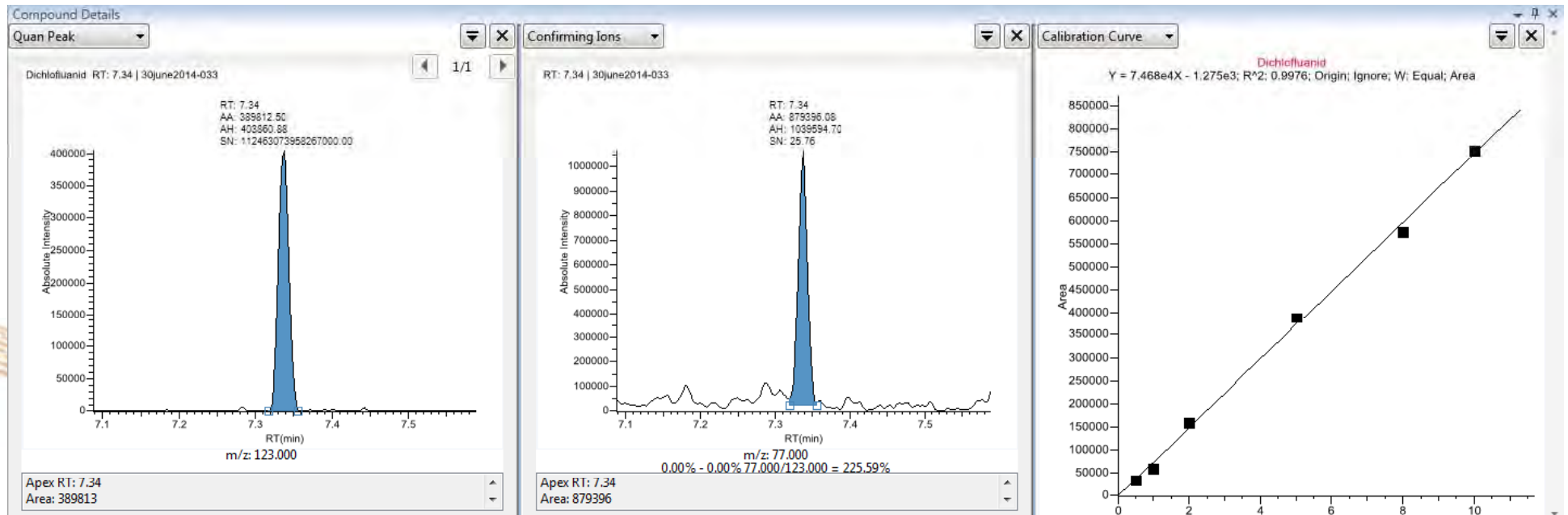
Simultaneous Full Scan & SRM (2SRMs/compound): baby food 5 ng/g



Linearity 0.5 – 10 ng/g

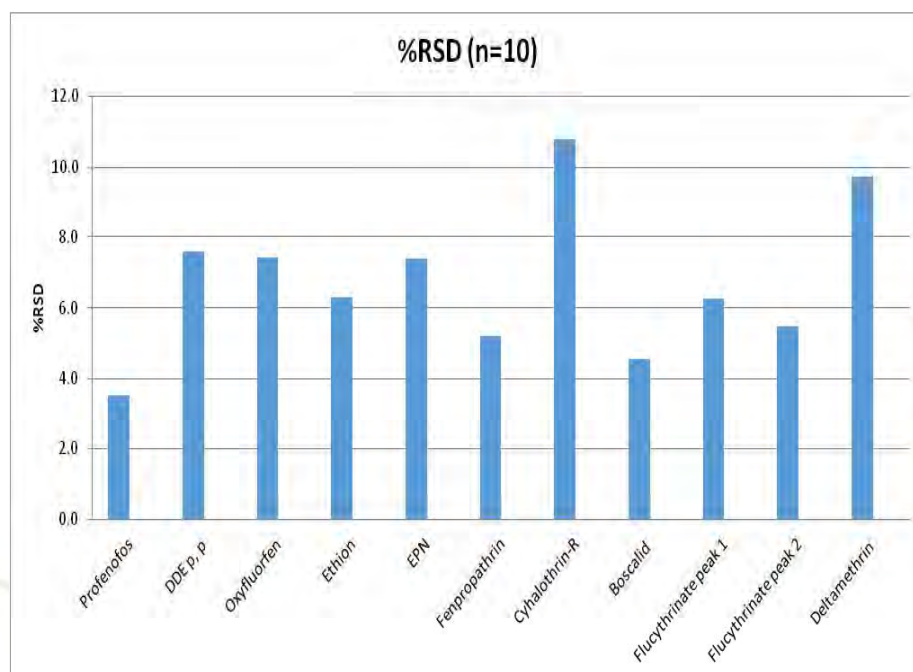


Dichlofluanid in baby food



Green Tea: repeatability at 10 ng/g

- 1917 SRM transitions acquired, 12 points /peak, timed-SRM acquisition mode



Summary



- TSQ 8000 provides excellent response – sufficient to meet the 10 ng/g level with 1g/mL crop in acetonitrile extracts.
- Less maintenance required (source clean every 2-3 months)
- Current status is <10% CV for most compounds
- Optimisation of injection parameters in progress
- TSQ 8000 Evo provides new possibilities for fast analyses and hence more productivity

Thank you for your attention



Fera user requirements for GC-MS/MS



- Data acquisition rate (No. of compounds/precision)
- Inherent response of detector (2nd transition)
 - *identification of analytes according to EU criteria*
 - *can assist streamlining of extraction and clean-up*
- Software
 - *efficiency of data processing and ease of use*
- Upgrades and flexibility
- Reliability of operation 24/7
 - *running costs*
 - *ease of maintenance*
 - *service back-up*