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Emerging Interest in GC-Triple Quadrupole Mass Spectrometry for Environmental Analysis

National Environmental Monitoring Conference

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Presentation Overview

- What are the analytical strengths of GC/QqQ?

- Example applications demonstrating strengths
  - Organotins in water
  - PAH in rubber
  - Dioxins in food and feed

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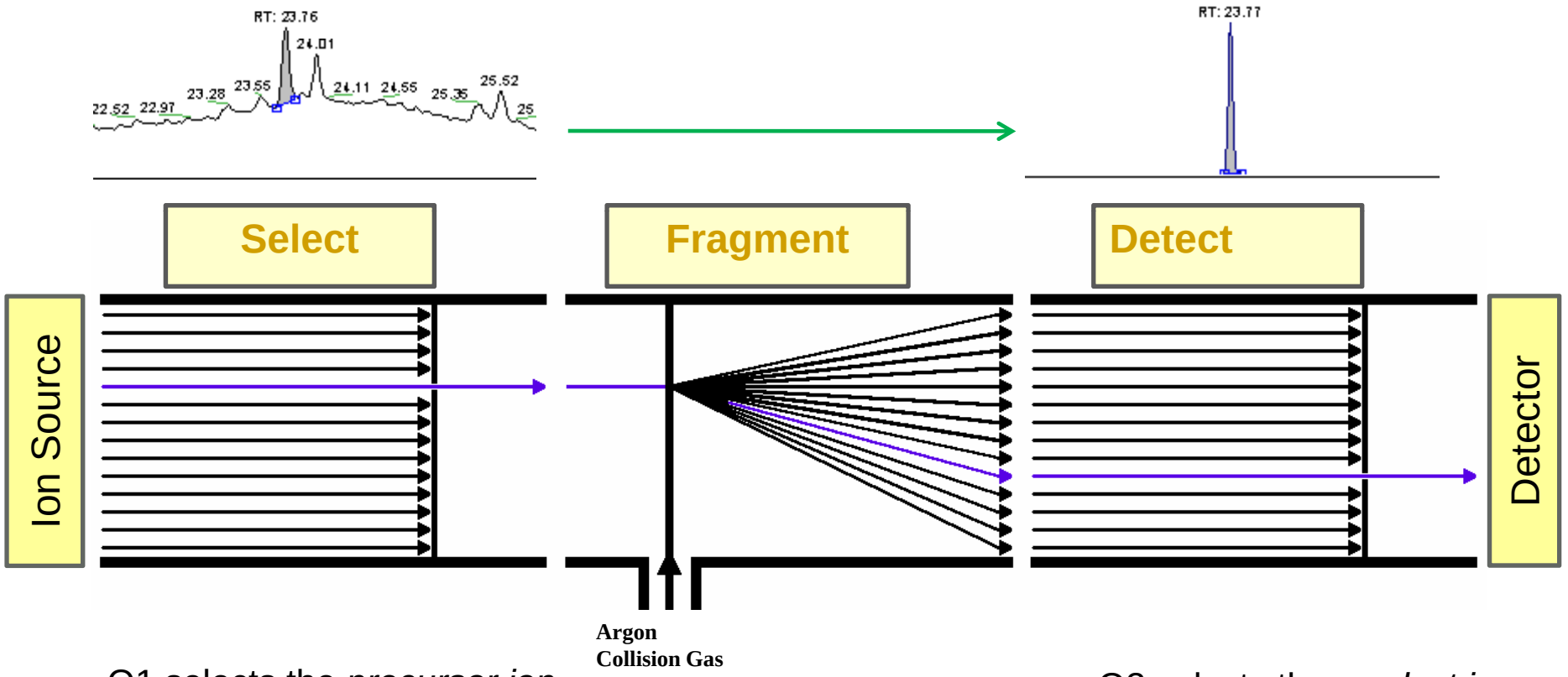
Analytical Strengths of GC Triple Quadrupole

Triple Quadrupole MS – Principle of Operation

Quantitation of target compounds in matrix samples



Q1 selects the *precursor ion*
***TCDD ion: m/z* 319.90**

Q3 selects the *product ion*
fragments to *m/z* 256.90

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Analytical Strengths of Triple Quadrupole in SRM

Analytical Strength	Analytical Reason	Application Benefit
High sensitivity	SRM has a high duty cycle	Lowered LOQ's/LOD's
High selectivity	MS/MS eliminates most isobaric interferants	Lowered LOQ's/LOD's Less sample prep High speed of analysis
High specificity	MS/MS eliminates most isobaric interferants	Less false positives

SRM process offers opportunity for lowered LOQs, reduced sample prep, higher speed of analysis, and less risk of false positives

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Organotins in Drinking Water

The world leader in serving science

Application Challenge and Approach

- Lowered limits set by EU Water Directive
  - Maximum allowable concentration – 1.5 ng/L (ppt)
  - Maximum annual average – 0.2 ng/L (ppt)

- Approach
  - Extraction
    - 400 ml of water at pH5
    - Extraction with pentane
    - Evaporated to 400 µl
    - Derivatized with sodium tetra-ethyl borate

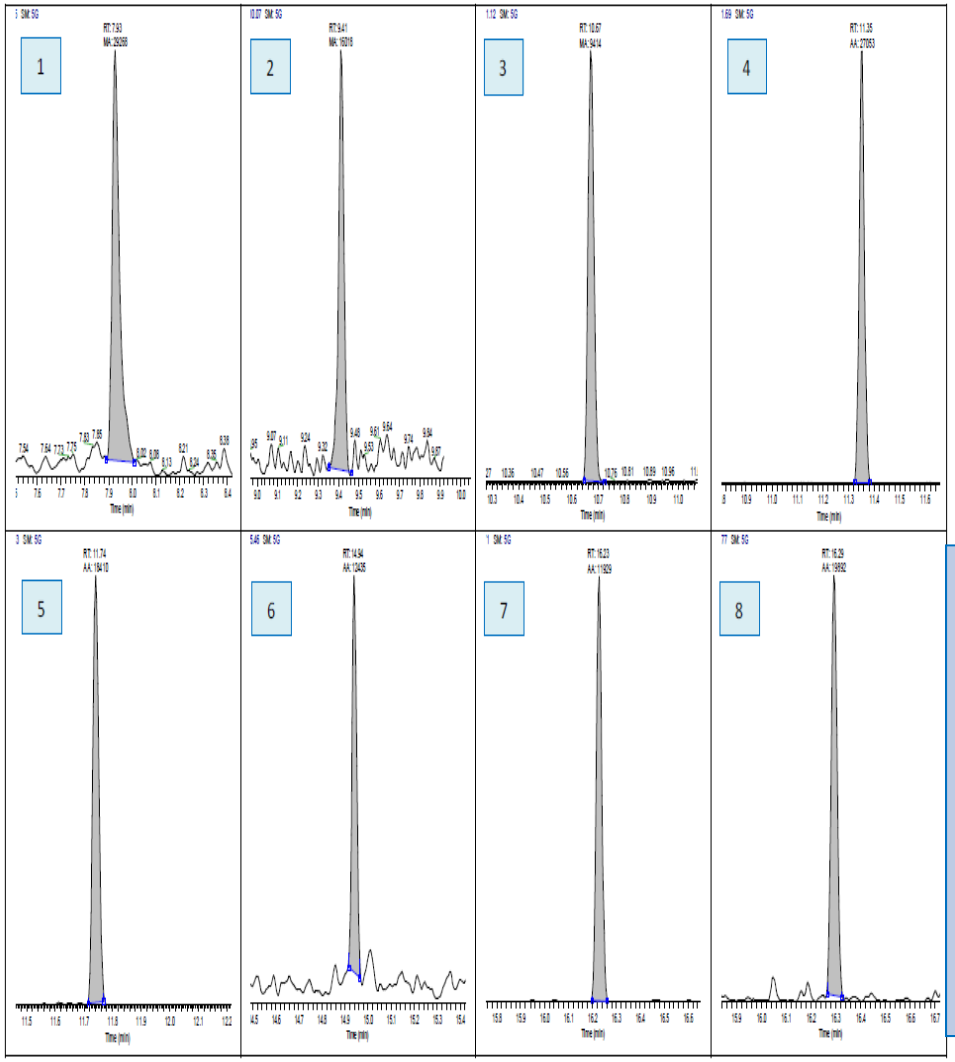
  - 3 µL injected into TSQ Quantum XLS, GC triple quadrupole

Customer Limits of Detection Using SIM

Compound	LOD Using SIM
Monobutyltin	10 ng/L
Dibutyltin	5 ng/L
Tributyltin	5 ng/L
Triphenyltin	1 ng/L

Greater than order of magnitude improvement in detection limits needed

Organotins at 0.5 ng/L



	Compound	Retention time	Transition
1	Monobutyltin	7.93	235.08>178.95
2	Dibutyltin	9.42	261.03>205.03
3	Tributyltin	10.67	289.09>176.95
4	Monooctyltin	11.35	289.09>176.95
5	Tetrabutyltin	11.74	289.09>176.95
6	Dioctyltin	14.94	261.03>148.98
7	Triphenyltin	16.23	351.15>196.94
8	Tricyclohexyltin	16.29	233.08>150.98

Injection of 0.2 pg absolute on column

Corresponding with 0.05ng/L in water sample

New EU directive: 0.2ng/L for tributyltin and here 0.05ng/l is shown

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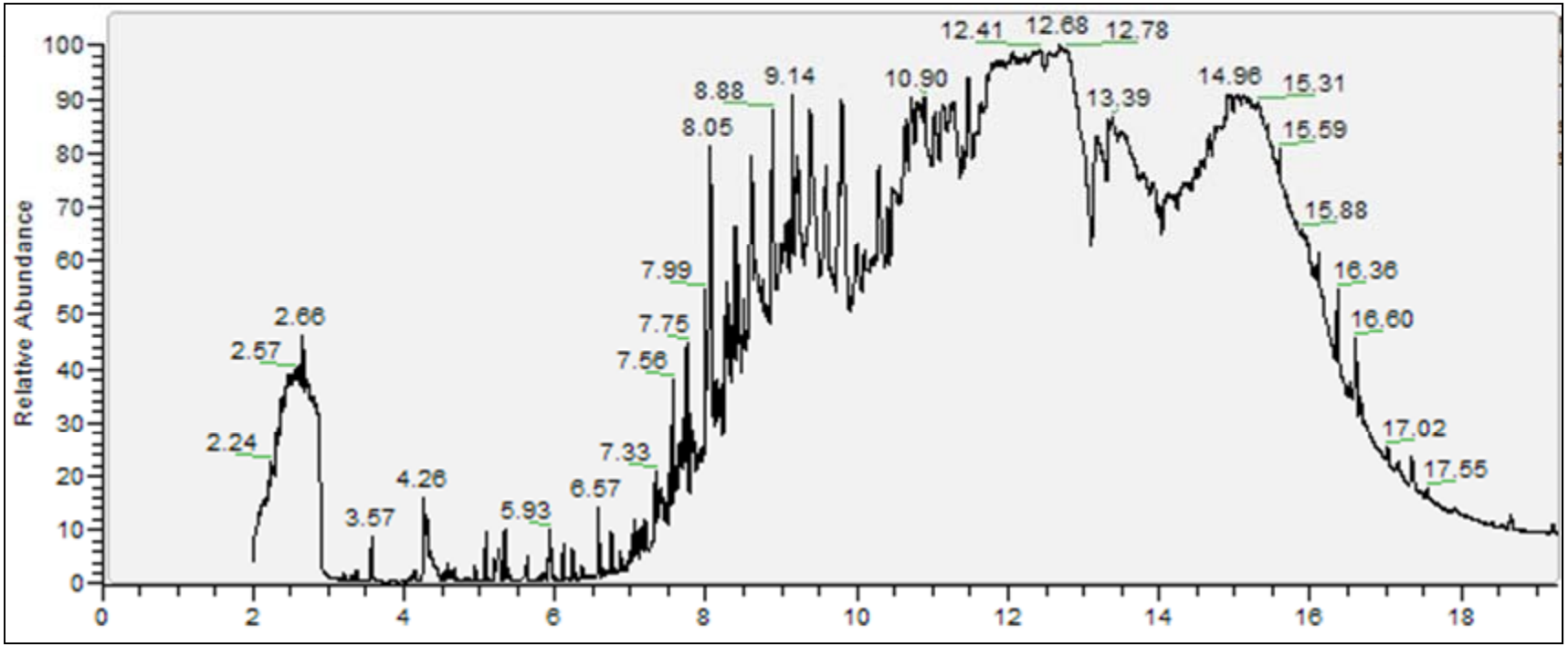
PAHs in Rubber

The Case of the Problem Sample

- Sample matrix: rubber
- Looking for PAH's that come from extender oils
- Full sample prep applied to sample



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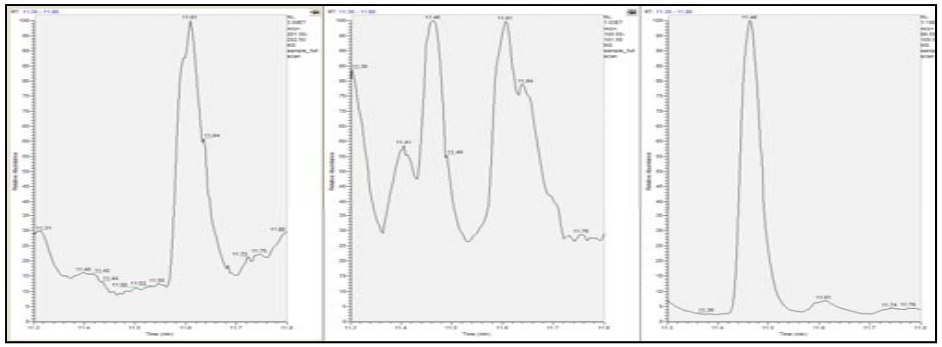
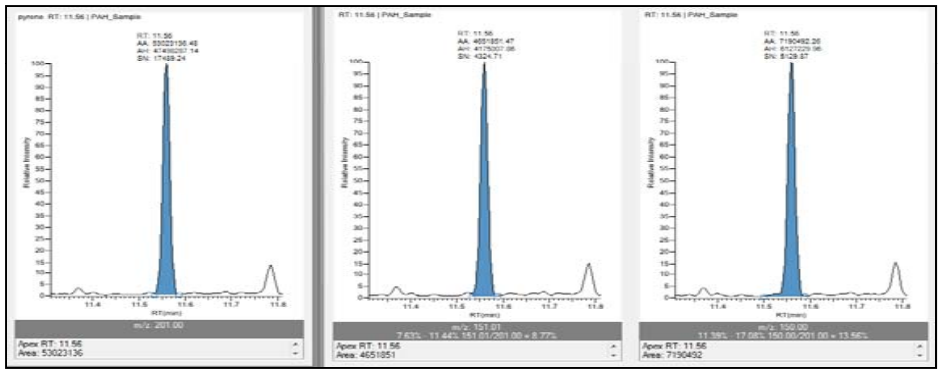
Comparison of Fullscan and SRM

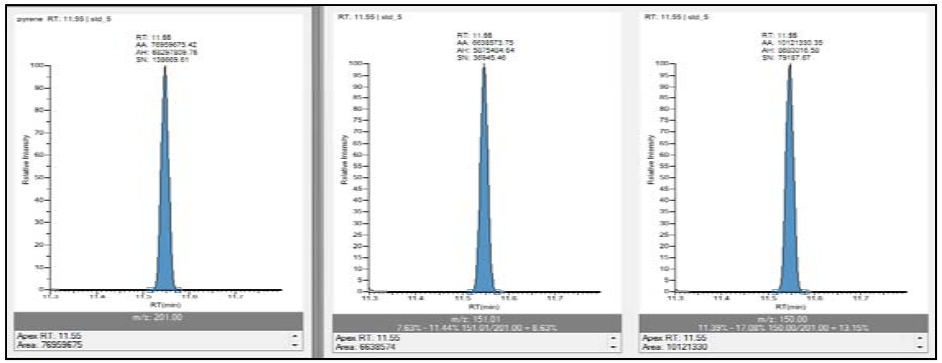
Pyrene in rubber by fullscan



Pyrene in rubber by SRM



Pyrene in Standard by SRM



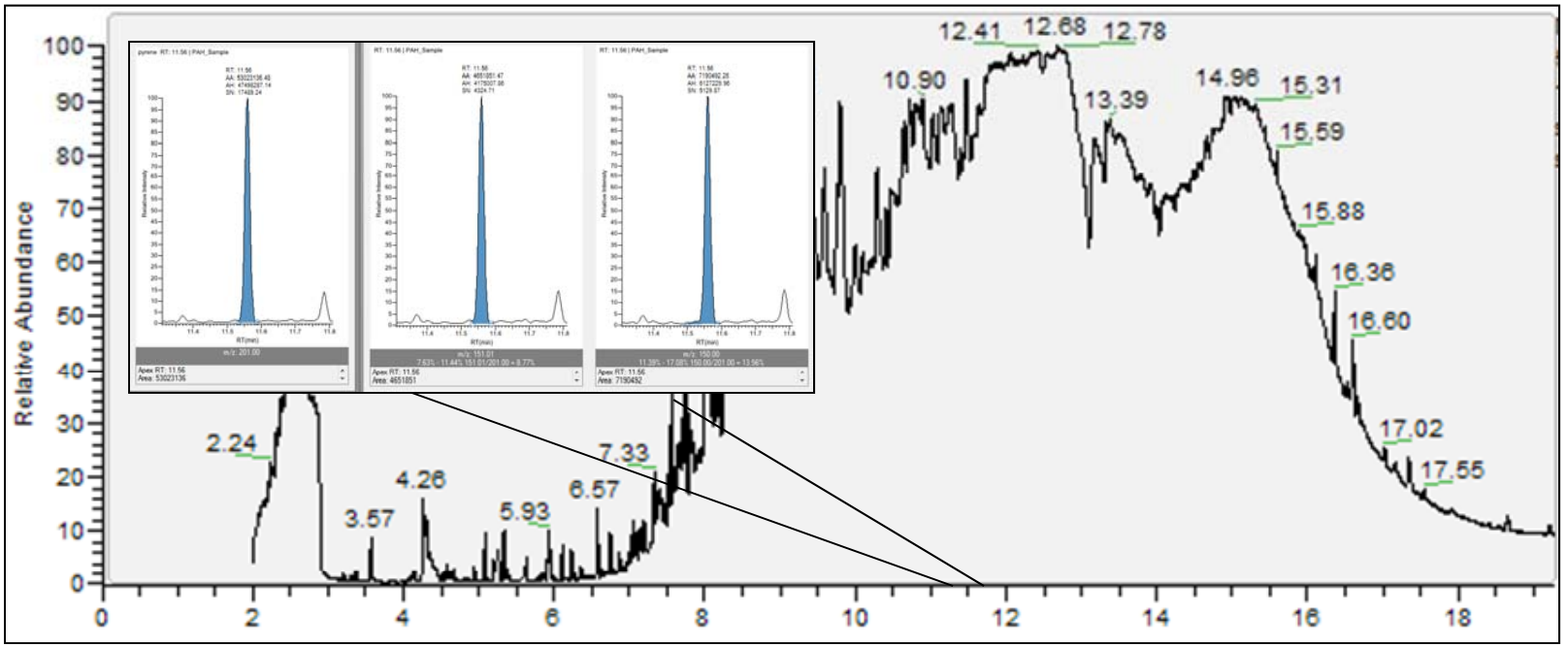
Compounds Detected – SRM vs Fullscan

Compound	SRM*	Fullscan
Naphthalene	X	X
Acenaphthylene	X	
Acenaphthene	X	
Anthracene	X	
Phenanthrene	X	
Pyrene	X	

* All compounds confirmed with retention times and ion ratios (2)

Benefits of Triple Quad for the Analysis

- Less sample prep required
- Ability to measure “worst case” samples
- Greater confidence in confirmation



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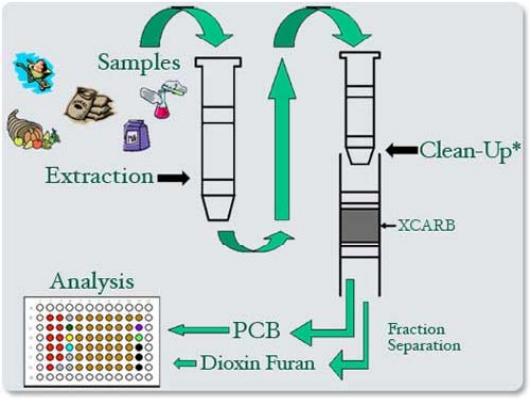
Dioxins in Food and Feed

Why GC-Triple Quad for Dioxins Analysis?

- Screening
  - Higher quality of screening results
    - TEQs can be calculated from results of screen [1]
    - Congener specific - Results depend on PCDD/F and PCB congener profile
  - Improvement of screening sensitivity for false negative results below 1%
    - Could result in a improved specificity, thus affecting the overall cost. [2]

- Confirmation
  - Lower upfront cost
  - More versitile approach



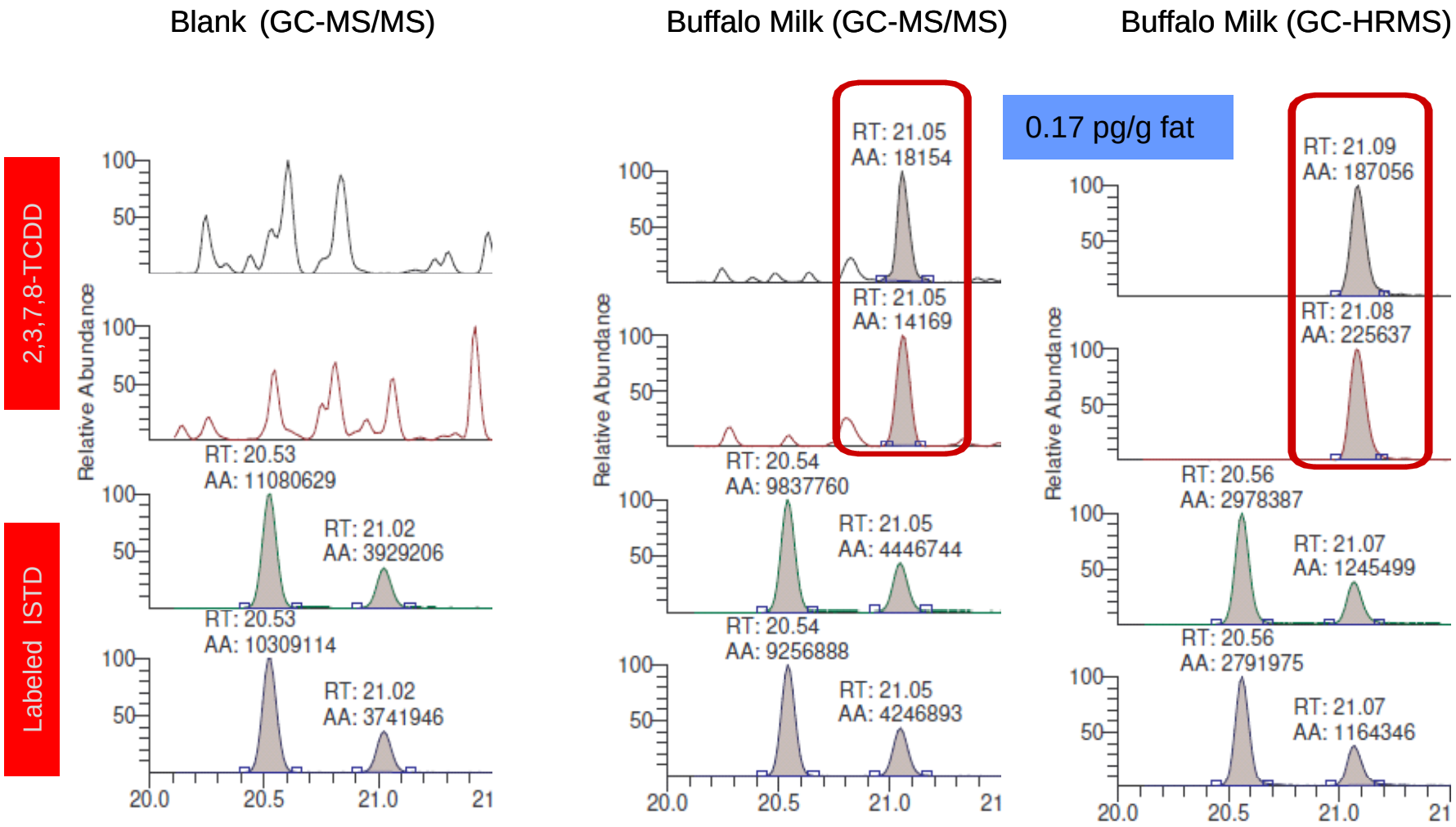
- References:
  - [1] Kotz, Malisch 2010, Dioxon Conference
  - [2] Fochi, Brambilla 2008

Maximum Allowed Levels

Commission Regulation (EC) No. 1881/2006
Commission Directive 2006/13/EC)

	Maximum levels WHO-PCDD/F-TEQ
Food (Meat, liver, eggs, milk products, fats and oils, fish)	**0.75 – 6** pg/g fat **4** pg/g wet weight (fish)
Feed (Compound feed, premixtures, additives ...)	**0.75 – 6** ng/kg product (12 % moisture content)

TSQ Quantum XLS - TCDD in Buffalo Milk Samples



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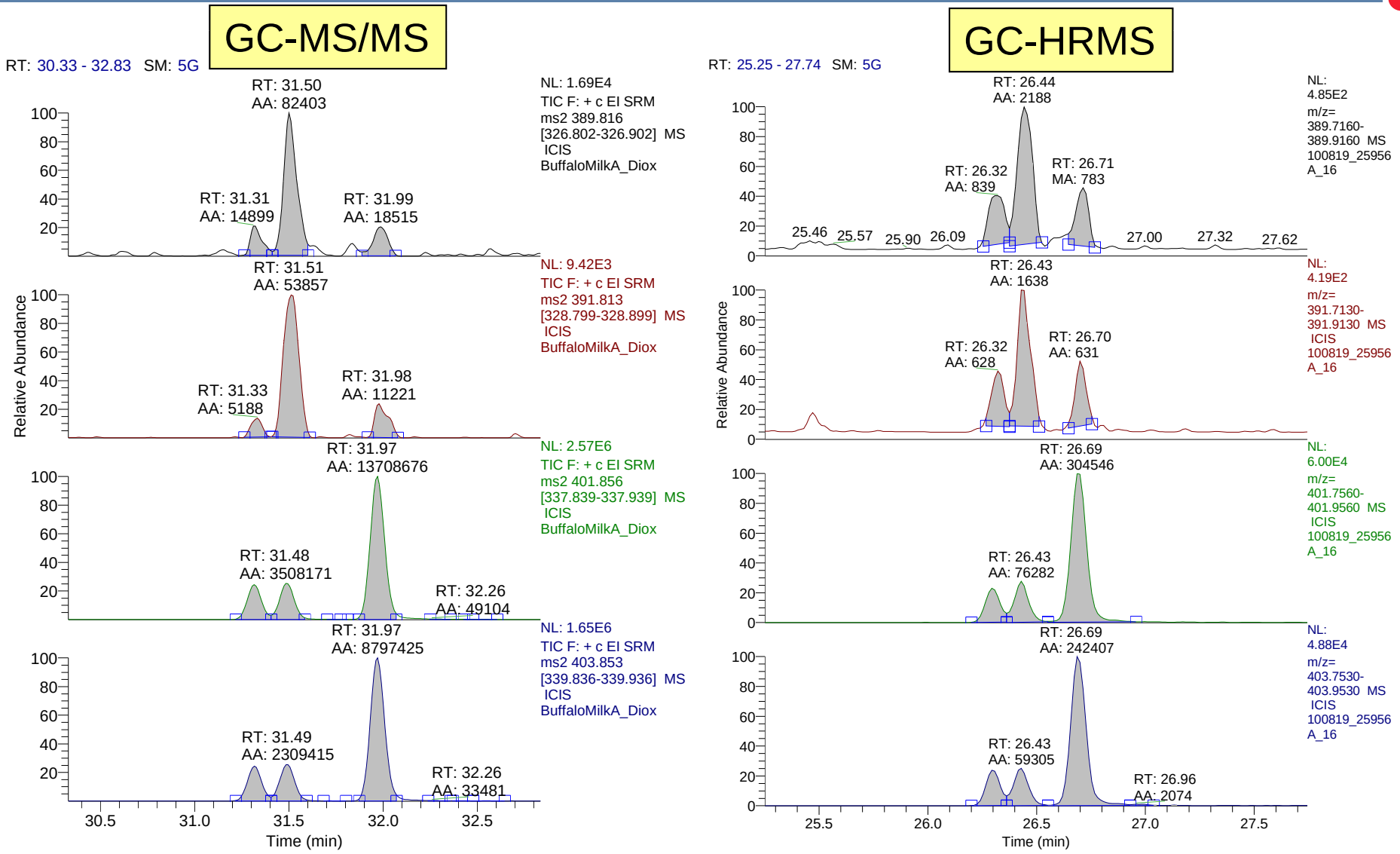
Buffalo Milk Sample, HxCDD

GC-MS/MS (5 µl injection PTV solv. split) → GC-HRMS 1/10 concentration



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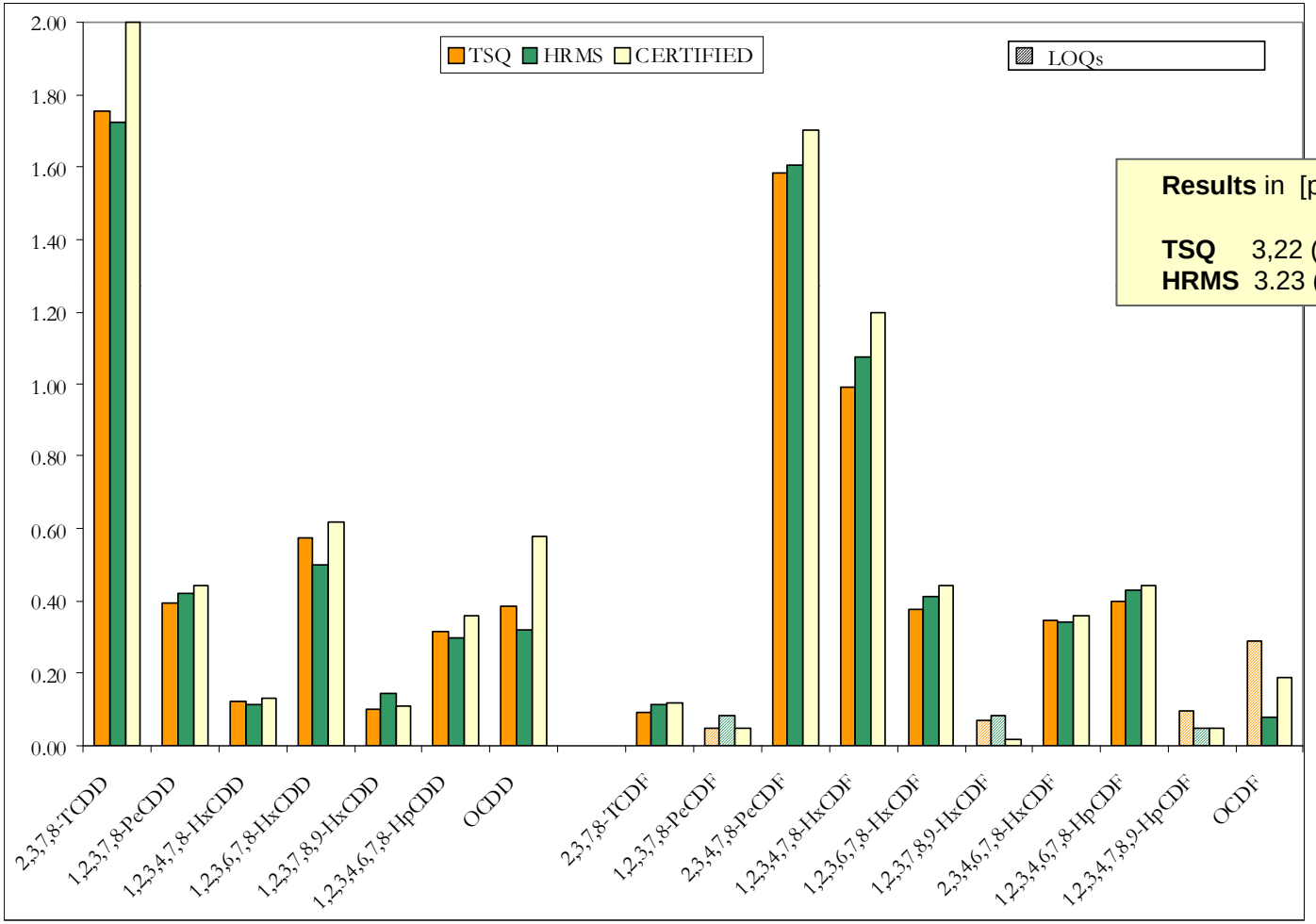
TSQ Quantum XLS vs. GC-HRMS of Certified Material



Results in [pg TEQ/g fat]

TSQ 3,22 (5% RSD)
HRMS 3.23 (2% RSD)

Courtesy N. Iacovella, G. Brambilla, ISS, Rome, Italy

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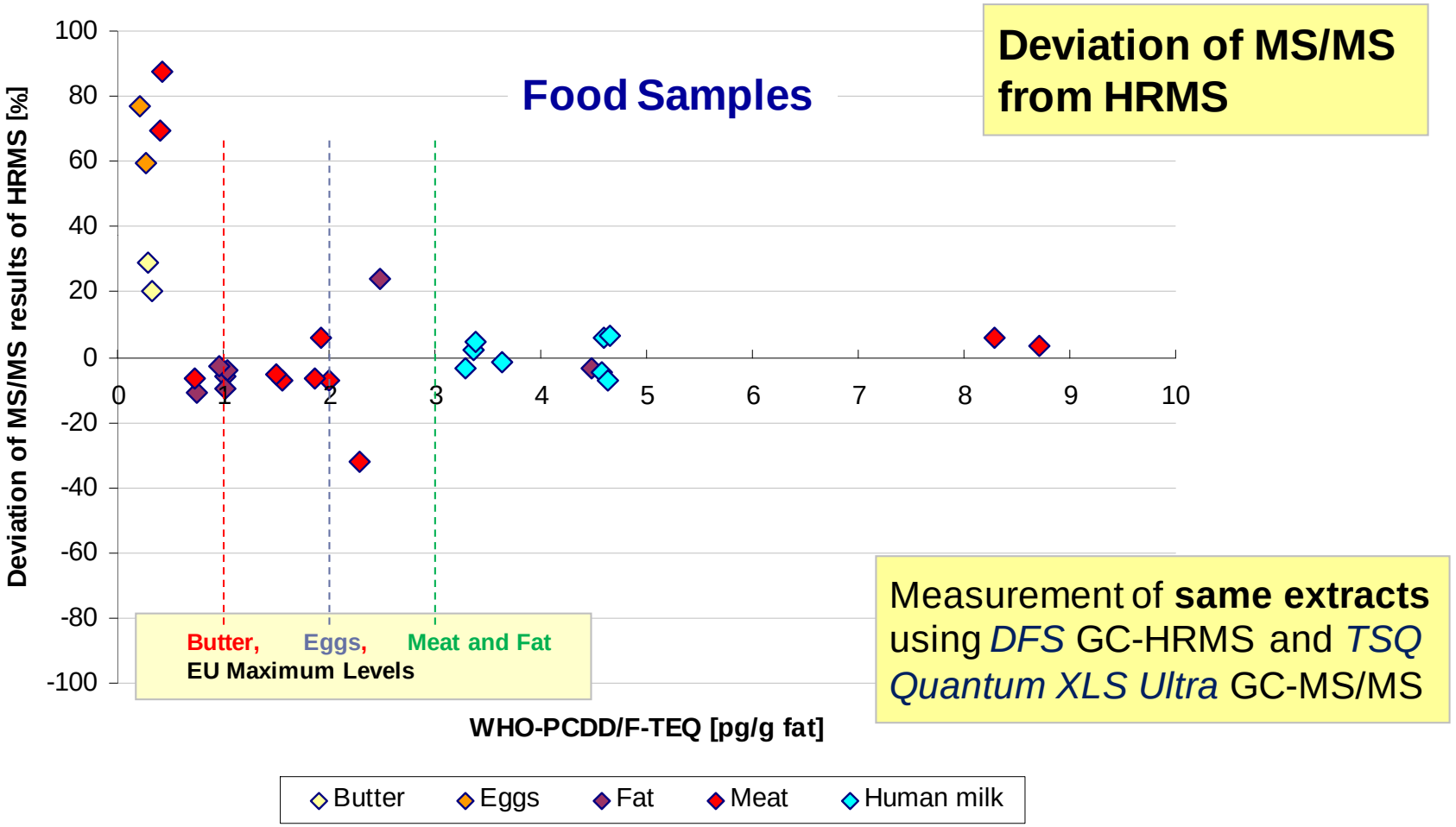
TSQ Quantum XLS Ultra – Food Dioxin Screening

- Courtesy Alexander Kotz, EURL Freiburg, Germany
  - Presented at the 6th POPs Users Meeting, Niagara-on-the-Lake, Canada, Apr 28/29, 2011



Measurement of **same extracts** using *DFS* GC-HRMS and *TSQ Quantum XLS Ultra* GC-MS/MS

Conclusion From Webinar by Dr. Kotz

- GC-MS/MS systems in principle applicable for PCDD/F and DL-PCB analysis in food and feed samples
- Sensitive enough to check for dioxins at maximum allowed levels
- GC/HRMS still preferred at lower concentration

Presenation Summary

- Laboratories are showing increasing interest in GC triple quad technology because of inherent advantages in the technology:
  - Low LOQ's from combination of high selectivity with high sensitivity
  - High specificity for more confident results

- Examples of application of technology
  - Organotins in drinking water for reduced LOQ's
  - PAH in rubber for confident detection in worst case matrix scenario
  - Dioxins in food and feed for higher sensitivity/specificity screening

Thank you very much for your attention!