Practical Experiences of Implementing POPs Methods using Orbitrap™ GC-MS

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Servei de Química
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York, 9 May 2017

CSB Consorci Sanitari de Barcelona

Agència de Salut Pública

Summary

- Laboratori Agència de Salut Pública de Barcelona
- Orbitrap™ technology
- Brominated Flame Retardants: PBDEs
- Our experience at the Lab
- Polychloronaphtalenes
- Non-dl-Polychlorobiphenyls

Laboratori Agència de Salut Pública

Food Safety

Regulations

Accreditation

The official control labs should:

- help to ensure food safety
- fulfill regulations
- work under accreditation ISO/IEC 17025

Regulations

COMMISSION DECISION

of 12 August 2002

implementing Council Directive 96/23/EC concerning the performance of analytical methods and the interpretation of results

(notified under document number C(2002) 3044)

(Text with EEA relevance)

(2002/657/EC)

INSTRUMENTATION

Table 5

The relationship between a range of classes of mass fragment and identification points earned

MS technique	Identification points earned per ion
Low resolution mass spectrometry (LR)	1,0
LR-MS^n precursor ion	1,0
LR-MS^n transition products	1,5
HRMS	2,0
HR- MS^n precursor ion	2,0
HR-MS^n transition products	2,5

VALIDATION

Model-independent and model-dependent performance parameters

Validation		
Model-independent performance parameters	Model-dependent performance parameters	
Common performance characteristics (3.1.1)	Conventional validation approach (3.1.2)	In-house validation approach (3.1.3)
Specificity	Recovery	Recovery
Trueness	Repeatability	Repeatability
Ruggedness: minor changes	Within-laboratory reproducibility	Within-laboratory reproducibility
Stability	Reproducibility	Reproducibility
	Decision limit (CCα)	Decision limit (CCα)
	Detection capability (CCβ)	Detection capability (CCβ)
	Calibration curves	Calibration curve
	Ruggedness: major changes	Ruggedness

CODEX ALIMENTARIUS COMMISSION E

Food and Agriculture Organization of the United Nations

World Health Organization

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Agenda Item 1 **CX/RVDF 12/20/1**

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON RESIDUES OF VETERINARY DRUGS IN FOODS

Twentieth Session

San Juan, Puerto Rico, 7-11 May 2012

The session will be held at the Hotel Sheraton Puerto Rico,
200 Convention Boulevard, San Juan, Puerto Rico
from Monday, 7 May at 10.00 hours to Friday, 11 May 2012

Guidance document on analytical quality control and method validation procedures for pesticides residues analysis in food and feed.

SANTE/11945/2015

Supersedes

SANCO/12571/2013

Implemented by 01/01/2016

6.4.2017 EN Official Journal of the European Union L 92/9

COMMISSION REGULATION (EU) 2017/644

of 5 April 2017

laying down methods of sampling and analysis for the control of levels of dioxins, dioxin-like PCBs and non-dioxin-like PCBs in certain foodstuffs and repealing Regulation (EU) No 589/2014

Laboratori Agència de Salut Pública

Food Safety

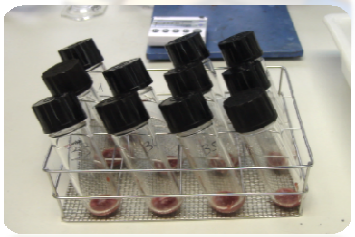
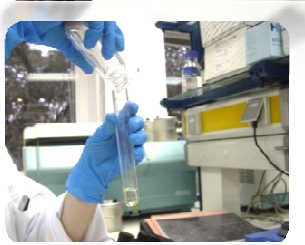
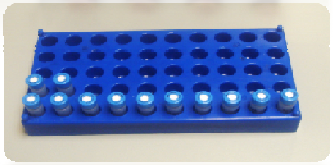
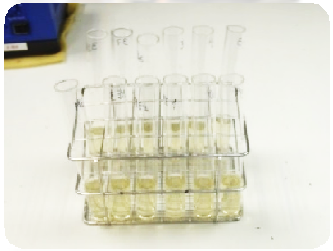




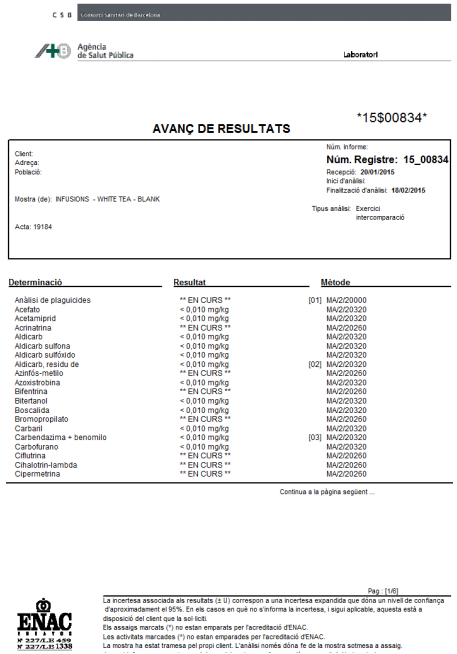










ISO International Organization for Standardization

ENAC Entidad Nacional de Acreditación

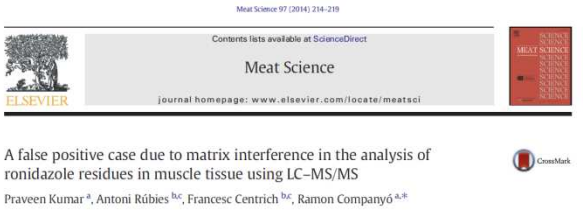
Accreditation

Methods based on Orbitrap™ at LASPB

Analytical methods already in use:

ANALYSIS OF VETERINARY DRUGS:

- Antibiotics
- Hormones
- Chloramphenicol
- Beta agonists
- Nitrofurans
- Nonsteroidal anti-inflammatory drugs
- …..


Meat Science 97 (2014) 214–219

Contents lists available at ScienceDirect

Meat Science

journal homepage: www.elsevier.com/locate/meatsci

A false positive case due to matrix interference in the analysis of ronidazole residues in muscle tissue using LC–MS/MS

Praveen Kumar [a], Antoni Rúbies [b,c], Francesc Centrich [b,c], Ramon Companyó [a,*]


Analytica Chimica Acta 780 (2013) 65–73

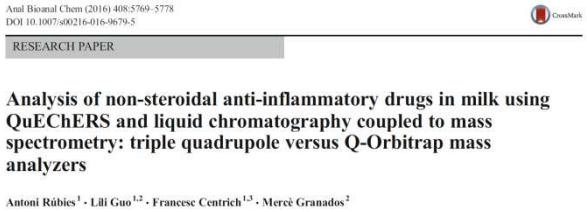
Contents lists available at SciVerse ScienceDirect

Analytica Chimica Acta

journal homepage: www.elsevier.com/locate/aca

Targeted analysis with benchtop quadrupole–orbitrap hybrid mass spectrometer: Application to determination of synthetic hormones in animal urine

Praveen Kumar [a], Antoni Rúbies [b,c], Francesc Centrich [b,c], Mercè Granados [a], Nuria Cortés-Francisco [d], Josep Caixach [d], Ramon Companyó [a,*]


Anal Bioanal Chem (2016) 408:5769–5778

DOI 10.1007/s00216-016-9679-5

RESEARCH PAPER

Analysis of non-steroidal anti-inflammatory drugs in milk using QuEChERS and liquid chromatography coupled to mass spectrometry: triple quadrupole versus Q-Orbitrap mass analyzers

Antoni Rúbies [1] · Lili Guo [1,2] · Francesc Centrich [1,3] · Mercè Granados [2]

ANALYSIS OF MARINE BIOTOXINS


Journal of Chromatography A, 1386 (2015) 62–73

Contents lists available at ScienceDirect

Journal of Chromatography A

journal homepage: www.elsevier.com/locate/chroma

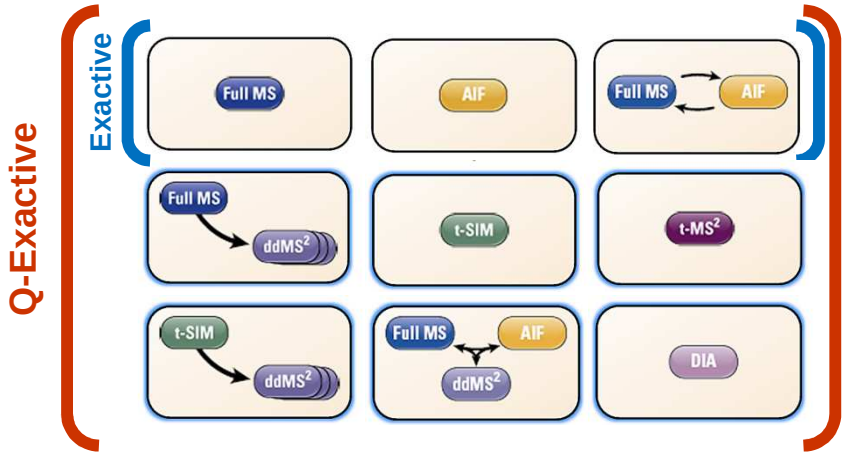
New method for the analysis of lipophilic marine biotoxins in fresh and canned bivalves by liquid chromatography coupled to high resolution mass spectrometry: A quick, easy, cheap, efficient, rugged, safe approach

A. Rúbies [a,c,*], E. Muñoz [a,c], D. Gibert [b], N. Cortés-Francisco [a,d], M. Granados [b], J. Caixach [d], F. Centrich [a,c]

ANALYSIS OF CONTAMINANTS FROM FOOD PACKAGING

ANALYSIS OF PROCESS CONTAMINANTS IN FOODSTUFF

New instrumentation: Orbitrap™ GC-MS



* Installed 14/12/2015

Pesticides

Brominated Flame Retardants: PBDEs

Polychloronaphtalenes (PCNs)

Polychlorobiphenyls (PCBs)

Thermo Scientific Q Exactive GC system

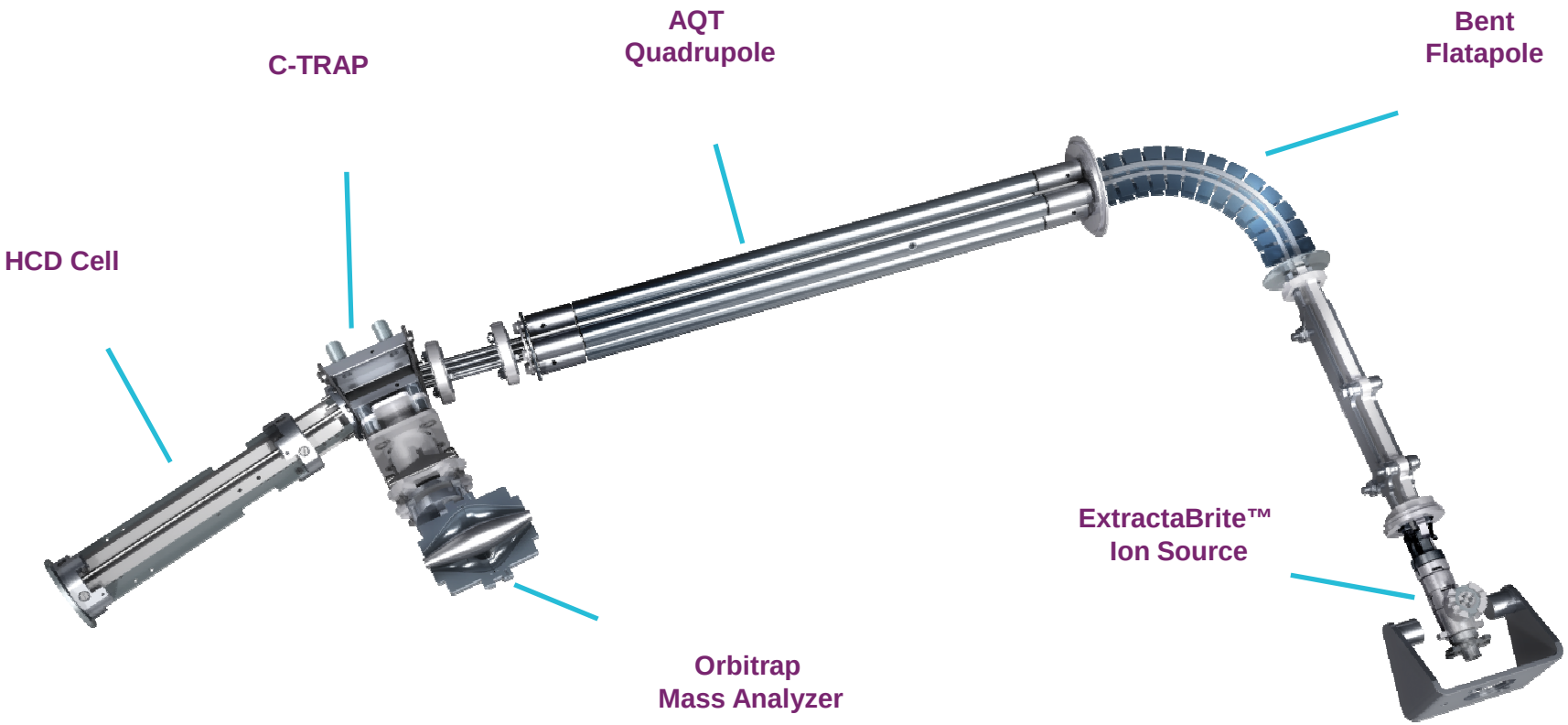
Unprecedented Depth in Analysis

RP 120,000 (FWHM @ *m/z* 200)

EI/CI; Full-scan, Timed-SIM

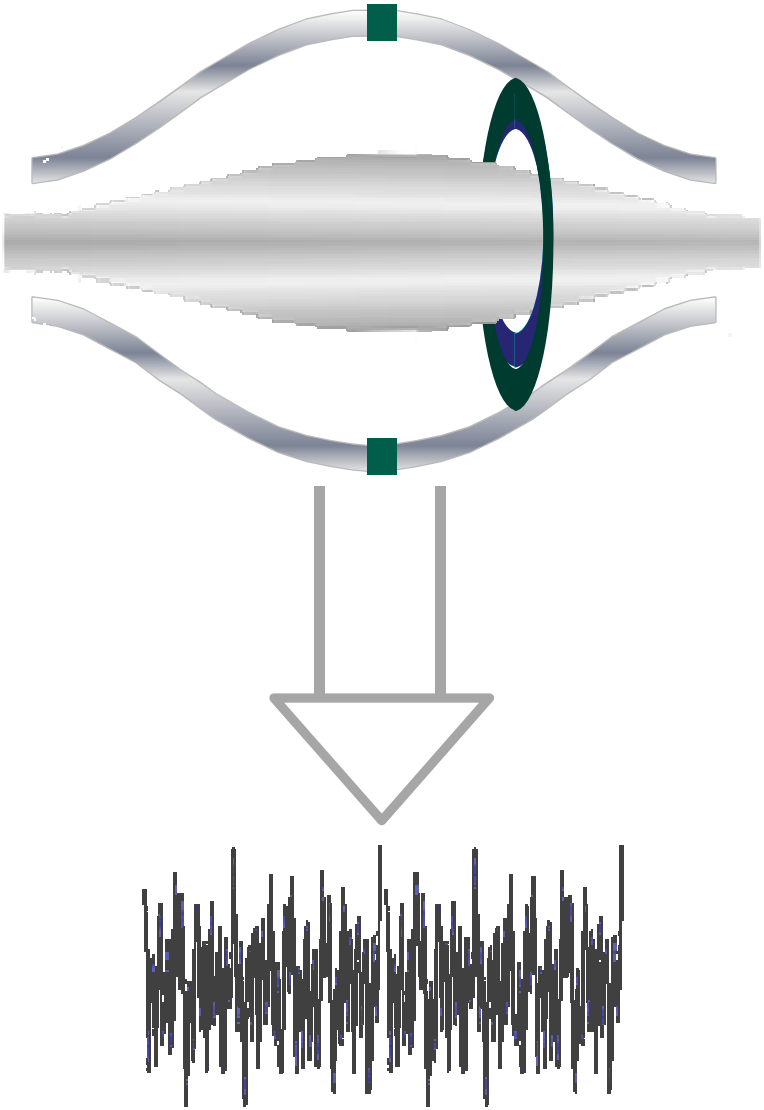
MS/MS capability

Bringing GC and Orbitrap Technology Together



* Courtesy of Thermo Fisher Scientific

Orbitrap Mass Analyzer



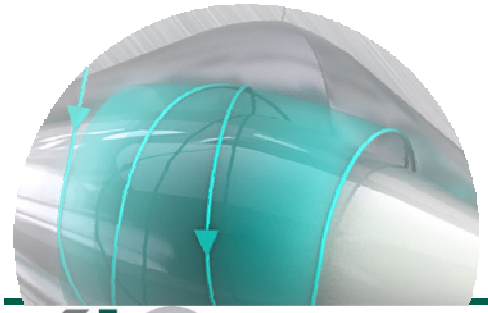
$$\omega = \sqrt{\frac{k}{m/z}}$$

- Ions injected into the Orbitrap are trapped in an electrostatic field
- Each ion oscillates axially with a frequency that is proportional to its mass
- An image current of these oscillations is measured using a split outer electrode
- This image is then converted to a mass spectrum using Fourier transform
- The longer a signal (transient) is measured, the higher the resolution

* Courtesy of Thermo Fisher Scientific

FTMS Resolution vs. Scan Rate

Resolution at *m/z* 200	Transient length, (ms)	Scan Rate, Hz
15 000	32	22
30 000	64	14
60 000	128	7
120 000	256	3.5



* Courtesy of Thermo Fisher Scientific

Brominated Flame Retardants

COMMISSION RECOMMENDATION

of 3 March 2014

5.3.2014 EN Official Journal of the European Union L 65/39

COMMISSION RECOMMENDATION

of 3 March 2014

on the monitoring of traces of brominated flame retardants in food

(Text with EEA relevance)

(2014/118/EU)

(1) Brominated flame retardants are organobromine compounds which are applied to products in order to inhibit or slow down the ignition of combustible materials in case of fire. They are commonly used in a wide range of consumer goods for example electronics, cars, furniture and construction materials to reduce the flammability of the product. Brominated flame retardants can leach out or evaporate from the products in which they were used. Since consumer goods are discharged at the end of their life, these substances have over time contaminated the environment and the food chain.

(2) However, many brominated flame retardants are persistent, bioaccumulative, and toxic to both humans and the environment. They are suspected of causing neurobehavioral effects and endocrine disruption and they have been found in biota in the environment.

(3) Therefore, the Commission asked the European Food Safety Authority (EFSA) to prepare a scientific opinion on the risks to public health related to the presence of brominated flame retardants in food.

(4) The Scientific Panel on Contaminants in Food of EFSA adopted six scientific opinions [1] on different classes of brominated flame retardants between September 2010 and September 2012.

[1] EFSA Panel on Conteaminanes in the Food Chain (CONTAM): Scientific Opinion on Polybrominated Biphenyls (PBBs) in Food. *EFSA Journal* 2010; 8(10):1789. [151 pp.]. doi:10.2903/j.efsa.2010.1789.
Scientific Opinion on Polybrominated Diphenyl Ethers (PBDEs) in Food. *EFSA Journal* 2011; 9(5):2156. [274 pp.] doi:10.2903/j.efsa.2011.2156.
Scientific Opinion on Hexabromocyclododecanes (HBCDDs) in Food. *EFSA Journal* 2011; 9(7):2296. [118 pp.] doi:10.2903/j.efsa.2011.2296.
Scientific Opinion on Tetrabromobisphenol A (TBBPA) and its derivatives in food. *EFSA Journal* 2011; 9(12):2477. [61 pp.] doi:10.2903/j.efsa.2011.2477.
Scientific Opinion on Brominated Flame Reardanes (BFRs) in Food: Brominated Phenols and their Derivaives. *EFSA Journal* 2012; 10(4):2634. [42 pp.] doi:10.2903/j.efsa.2012.2634.
Scientific Opinion on Emerging and Novel Brominated Flame Reardanes (BFRs) in Food. *EFSA Journal* 2012; 10(10):2908. [125 pp.] doi:10.2903/j.efsa.2012.2908.

dation as regards the monitoring of animal feed could follow in 2015,

HAS ADOPTED THIS RECOMMENDATION:

1. Member States should perform monitoring on the presence of brominated flame retardants in food, during the years 2014 and 2015. The monitoring should include a wide variety of individual foodstuffs reflecting consumption habits in order to give an accurate estimation of exposure and different food commodities should be included for the different classes of brominated flame retardants

2. Member States should follow the sampling procedures laid down in Annex II to Commission Regulation (EU) No 252/2012 [2] in order to ensure that the samples are representative of the sampled lot.

3. Member States should carry out analysis of the different classes of brominated flame retardants in order to detect the presence of the following substances in the respective food commodities:

(a) for the class of polybrominated diphenyl ethers (PBDEs): 2,2′,4-tribromodiphenyl ether (BDE-28, CAS No 41318-75-6); 2,2′,4,4′-tetrabromodiphenyl ether (BDE-47, CAS No 5436-43-1); 2,2′,4,5′-tetrabromodiphenyl ether (BDE-49, CAS No 243982-82-3); 2,2′,4,4′,5-pentabromodiphenyl ether (BDE-99, CAS No 60348-60-9); 2,2′,4,4′,6-pentabromodiphenyl ether (BDE-100, CAS No 189084-64-8); 2,2,3,4,4,5-hexabromodiphenyl ether (BDE-138, CAS No 67888-98-6); 2,2′,4,4′,5,5′-hexabromodiphenyl ether (BDE-153, CAS No 68631-49-2); 2,2′,4,4′,5,6′-hexabromodiphenyl ether (BDE-154, CAS No 207122-15-4); 2,2′,3,4,4′,5′,6-heptabromodiphenyl ether (BDE-183, CAS No 207122-16-5) and 2,2′,3,3′,4,4′,5,5′,6,6′-decabromodiphenyl ether (BDE-209, CAS No 1163-19-5), in eggs and egg products, milk and dairy products, meat and meat products, animal and vegetable fats and oils, fish and other seafood, products for specific nutritional uses, and food for infants and small children, using analytical methods with a limit of quantification of 0,01 ng/g wet weight or lower;

(b) for the class of hexabromocyclododecanes (HBCDDs): (+/−)-α-HBCD (1,2,5,6,9,10-hexabromo-(1R,2R,5S,6R,9R,10S)-rel-cyclododecane, CAS No 134237-50-6); (+/−)-β-HBCD (1,2,5,6,9,10-hexabromo-(1R,2S,5R,6R,9R,10S)-rel-cyclododecane, CAS No 134237-51-7) and (+/−)-γ-HBCD (1,2,5,6,9,10-hexabromo-(1R,2R,5R,6S,9S, 10R)-rel-cyclododecane, CAS No 134237-52-8) in fish and other seafood, meat and meat products, milk and dairy products, eggs and egg products, as well as infant and follow-up formula. The analytical methods used for the determination of HBCDDs include the determination of stereoisomers and should have a limit of quantification of 0,01 ng/g wet weight or lower;

(c) for the class of tetrabromobisphenol A and its derivatives: tetrabromobisphenol A (TBBPA, CAS No 79-94-7) and possibly TBBPA bismethyl ether (TBBPA-bME, CAS No 70156-79-5); TBBPA bis(2-hydroxyethyl) ether (TBBPA-bOHEE, CAS No 4162-45-2); TBBPA bisallyl ether (TBBPA-bAE, CAS No 25327-89-3); Tetrabromobisphenol A bis(glycidyl ether) (TBBPA-bGE, CAS No 3072-84-2) and TBBPA bis(2,3-dibromopropyl)ether (TBBPA-bDiBPrE, CAS No 21850-44-2) in fish and other seafood, meat and meat products, milk and dairy products, and eggs and egg products. The analytical methods used for the determination of tetrabromobisphenol A and its derivatives should have a limit of quantification of 0,1 ng/g wet weight or lower;

(d) for the class of brominated phenols and their derivatives: 2,4,6-tribromophenol (2,4,6-TBP, CAS No 118-79-6); 2,4-dibromophenol (2,4-DBP, CAS No 615-58-7); 4-bromophenol (4-BP, CAS No 106-41-2); 2,6-dibromophenol (2,6-DBP, CAS No 608-33-3); tetrabrominated bisphenol S (TBBPS, CAS No 39635-79-5); tetrabromobisphenol S bismethyl ether (TBBPS-BME, CAS No 70156-79-5) in fish and other seafood. The analytical methods used for the determination of brominated phenols and their derivatives should have a limit of quantification of 0,1 ng/g wet weight or lower;

(e) for the emerging and novel brominated flame retardants: tris(2,3-dibromopropyl) phosphate (TDBPP, CAS No 126-72-7); N,N′-ethylenebis(tetrabromophthalimide) (EBTEBPI, CAS No 32588-76-4); hexabromocyclodecane (HBCYD, CAS No 25495-98-1); bis(2-ethylhexyl) tetrabromophthalate (BEH-TEBP, CAS No 26040-51-7); 2-ethylhexyl 2,3,4,5-tetrabromobenzoate (EH-TBB, CAS No 183658-27-7) and dibromoneopentyl glycol (DBNPG, CAS No 3296-90-0) in fish and other seafood, meat and meat products (including edible offal), animal and vegetable fats and oils, milk and dairy products, eggs and egg products and food for infants and small children. The analytical methods used for the determination of emerging and novel brominated flame retardants should have a limit of quantification of 1 ng/g wet weight or lower.

4. Member States should carry out the analysis of brominated flame retardants in accordance with Annex III to Regulation (EC) No 882/2004 of the European Parliament and of the Council [1] using a method of analysis that has been proven to generate reliable results.

5. Member States should provide, on a regular basis to EFSA, the monitoring data expressed on whole weight basis or fat basis with the information and in the electronic reporting format as set out by EFSA for compilation into one database. They should include available data obtained from previous years by using a method of analysis that has been proven to generate reliable results in order to monitor trends in exposure.

Done at Brussels, 3 March 2014.

For the Commission

Tonio BORG

Member of the Commission

[2] Commission Regulation (EU) No 252/2012 of 21 March 2012 laying down methods of sampling and analysis for the official control of levels of dioxins, dioxin-like PCBs and non-dioxin-like PCBs in certain foodstuffs and repealing Regulation (EC) No 1883/2006 (OJ L 84, 23.3.2012, p. 1).

[1] Regulation (EC) No 882/2004 of the European Parliament and of the Council of 29 April 2004 on official controls performed to ensure the verification of compliance with feed and food law, animal health and animal welfare rules (OJ L 165, 30.4.2004, p. 1).

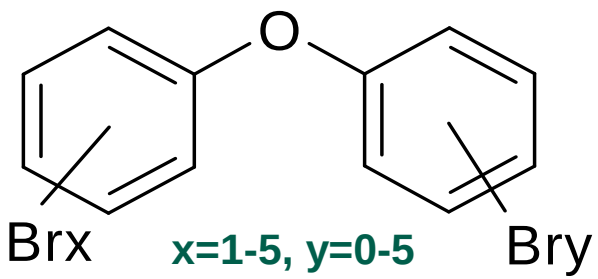
Polybrominated diphenyl ethers (PBDEs)

5.3.2014 EN Official Journal of the European Union L 65/39

COMMISSION RECOMMENDATION
of 3 March 2014
on the monitoring of traces of brominated flame retardants in food
(Text with EEA relevance)
(2014/118/EU)

(a) Polybrominated diphenyl ethers (PBDEs)

BDE – 28
BDE – 47
BDE – 49
BDE – 99
BDE – 100
BDE – 138
BDE – 153
BDE – 154
BDE – 183
BDE – 209

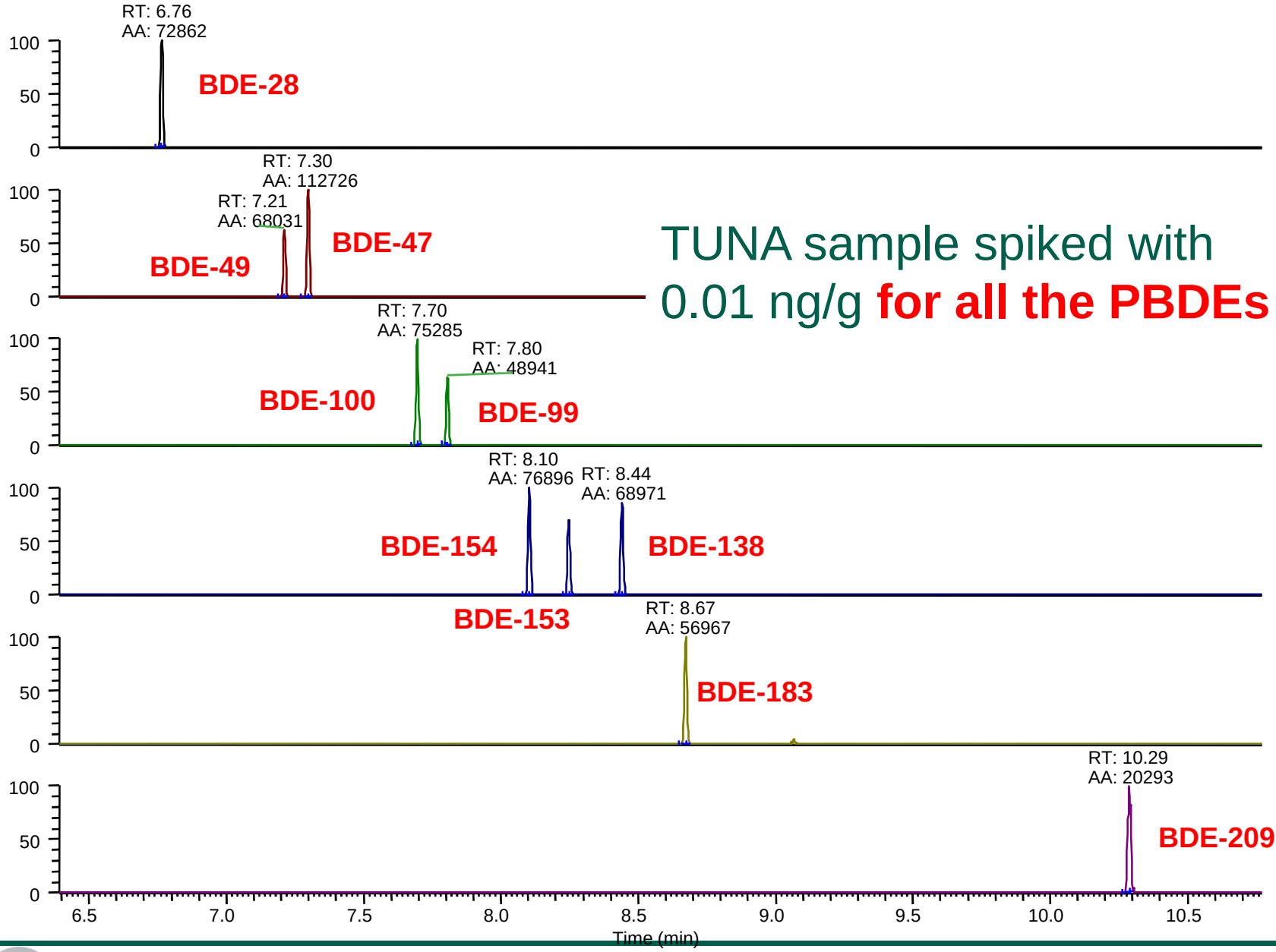


eggs and egg products
milk and dairy products
meat and meat products
animal and vegetable fats and oils
fish and other seafood
products for specific nutritional uses
food for infants and small children

Analytical methods with a limit of quantification of 0,01 ng/g wet weight or lower

10 ppt in the sample

Polybrominated diphenyl ethers (PBDEs)



TUNA sample spiked with 0.01 ng/g **for all the PBDEs**

HRMS approach

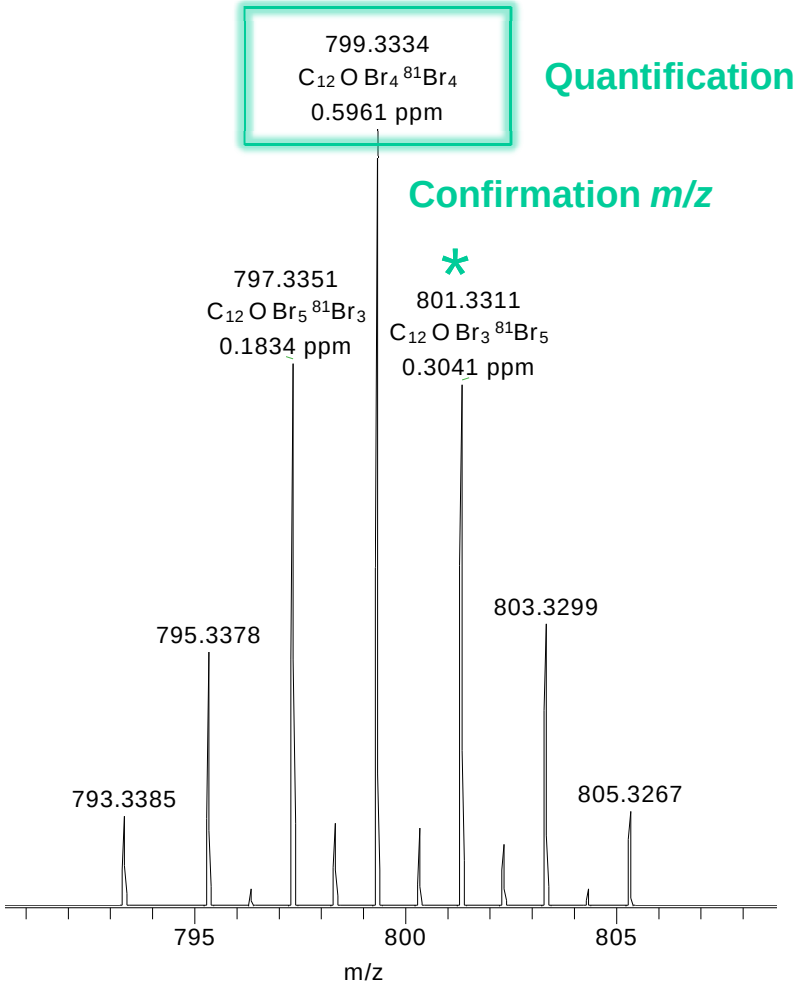
EPA United States Environmental Protection Agency

Method 1614A
Brominated Diphenyl Ethers in Water, Soil, Sediment, and Tissue by HRGC/HRMS

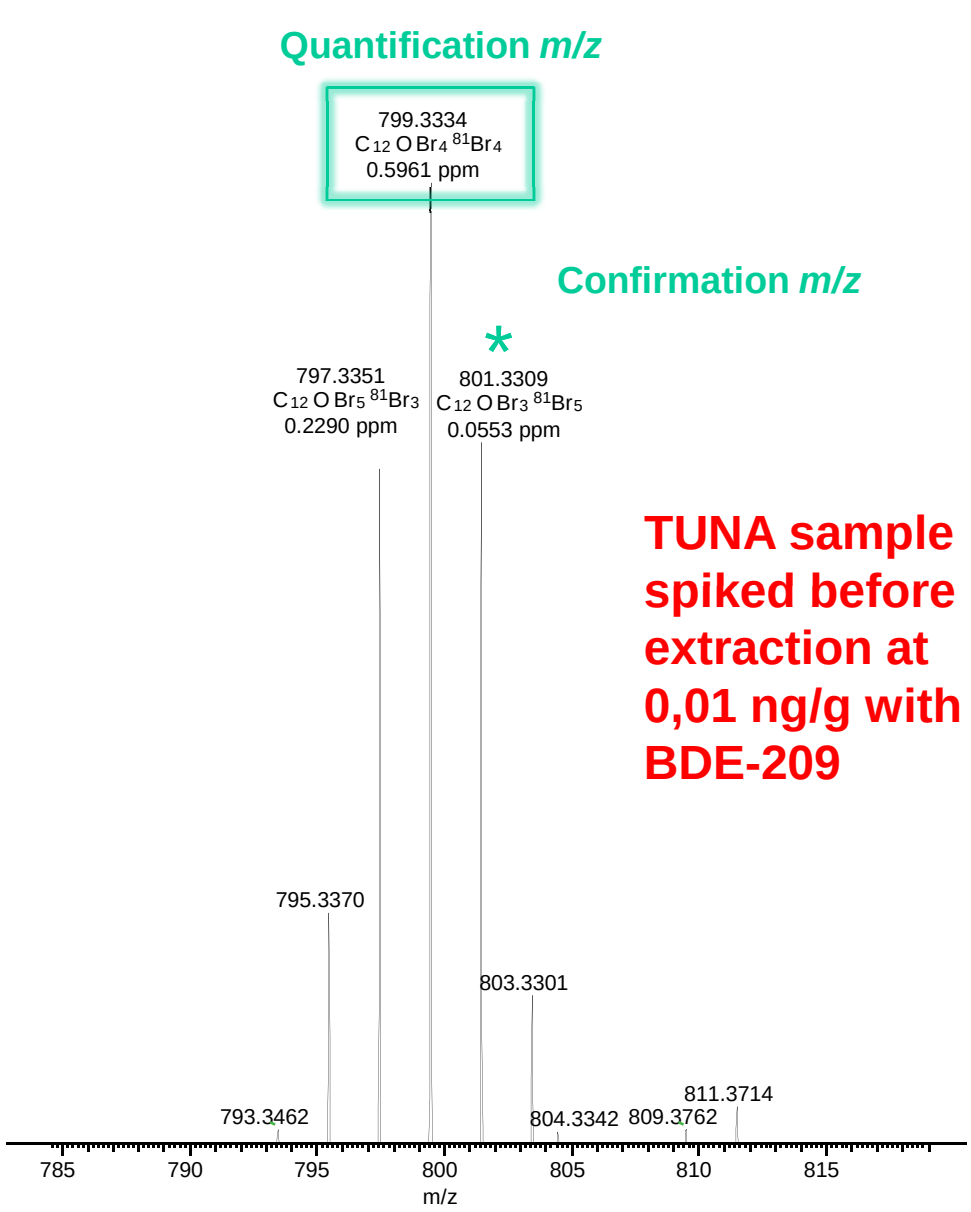
May 2010

2.3 After cleanup, the extract is concentrated to 20 µL and labeled injection internal standards are added. An aliquot of the extract is injected into the gas chromatograph (GC). The analytes are separated by the **GC and detected by a high-resolution (≥5000) mass spectrometer. Two exact m/z's are monitored at each level of bromination (LOB)** throughout a pre-determined retention time window.

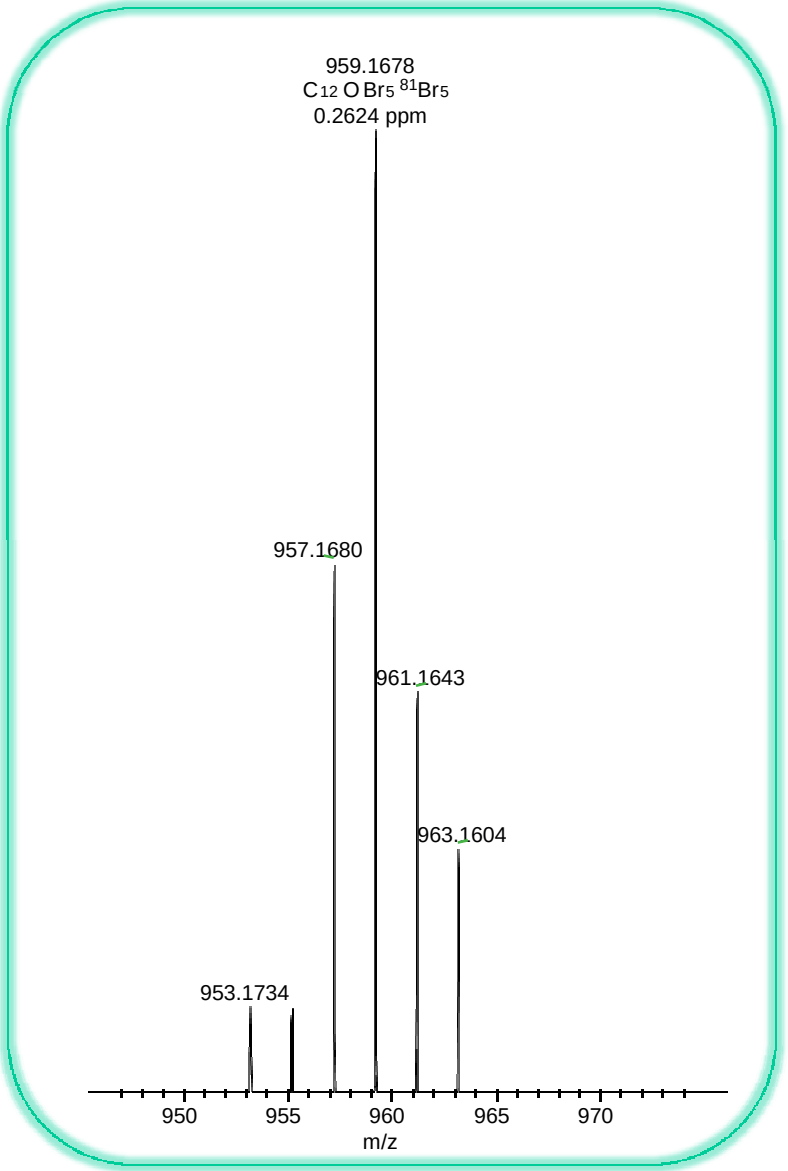
With the new HRMS instruments we are capable of acquiring the full isotopic pattern without losing sensitivity and working at R: 30,000 (FWHM, *m/z* 200) or higher.



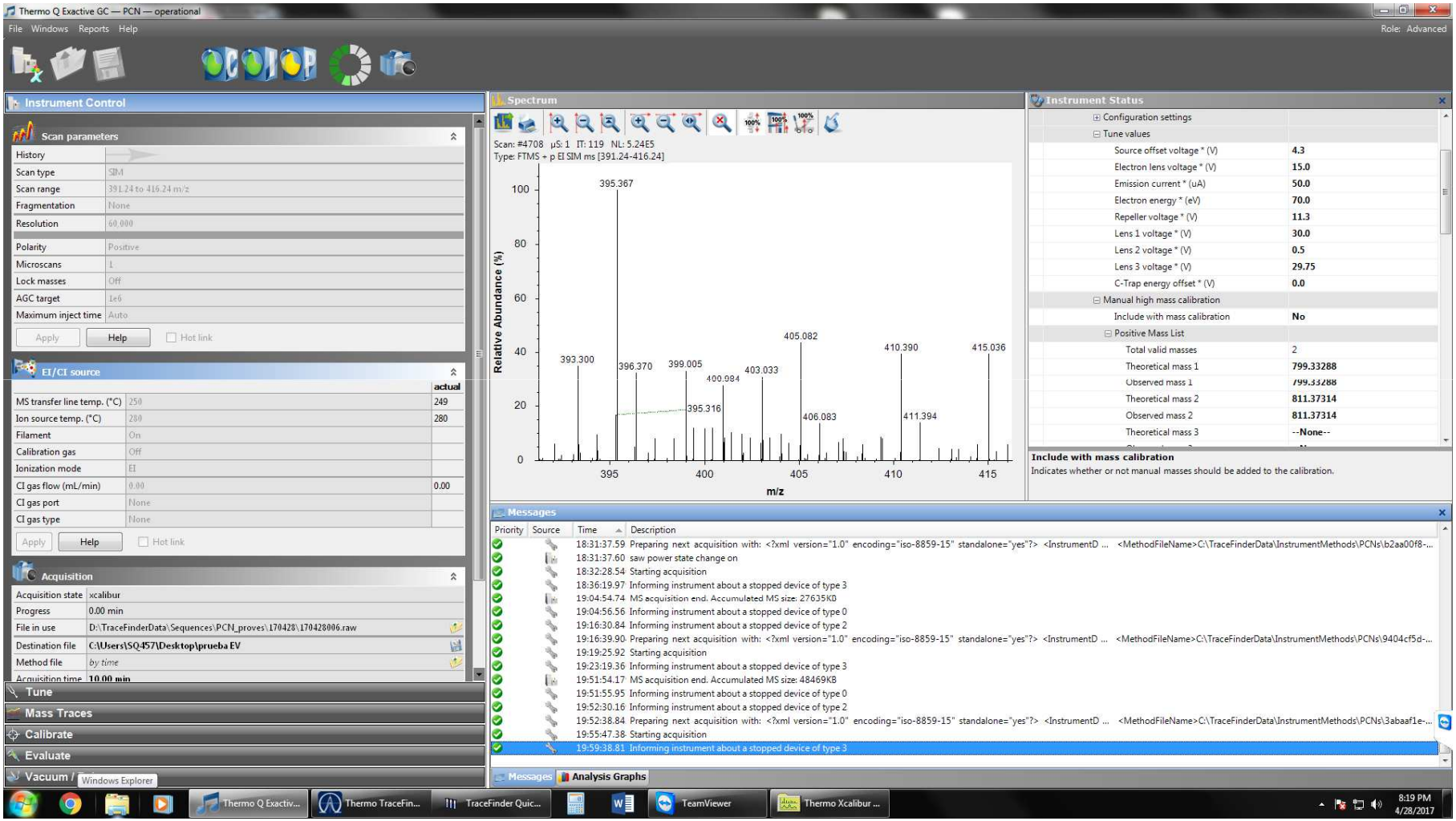
HRMS approach



TUNA sample spiked before extraction at 0,01 ng/g with BDE-209

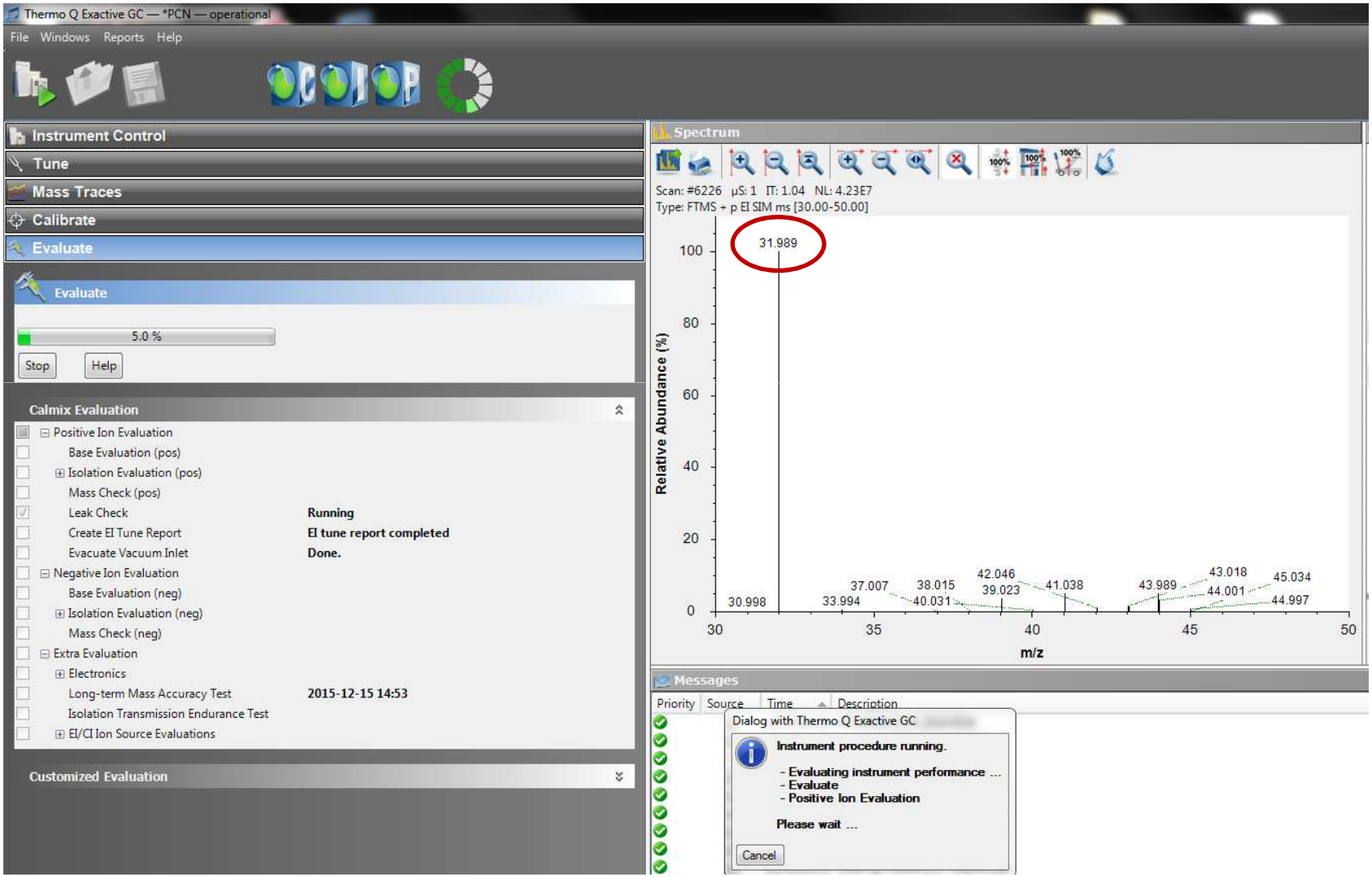


Once in the Lab: Tune Page



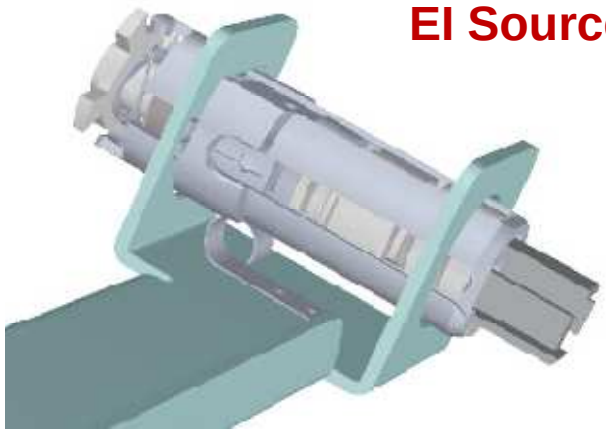
Once in the Lab: Tune Page

Leak detection: oxygen can be monitored



Optimization of EI Source Parameters

EI Source

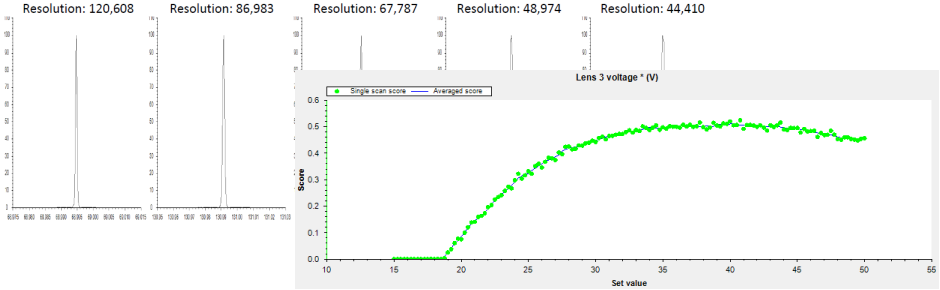


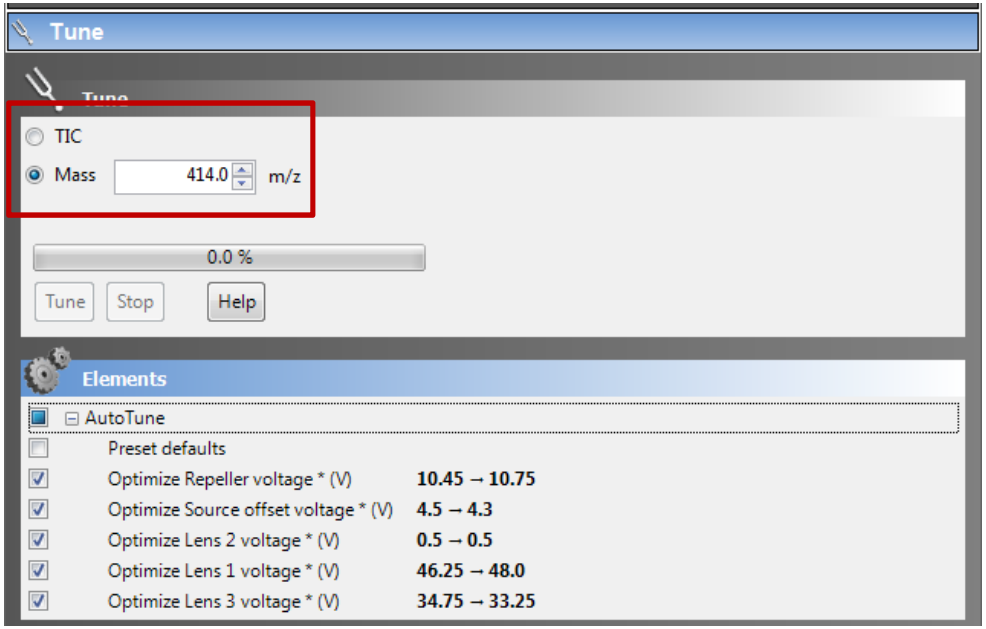
Q Exactive GC Tune Report

Thermo SCIENTIFIC

Date of Tune Report:	2017-03-02 17:03:19
Last Mass Calibration:	2017-03-02 16:42:48
Instrument Identification:	Exactive Series slot #19 (901B0E3F8F47)
Software Version:	2.8-280502/2.8.1.2806
Data System User:	PCSQUIM457\Admin(Admin)
Tune File:	C:\Xcalibur\methods\PBDE_HT.mstune

m/z 68.99471	m/z 130.99155	m/z 218.98507	m/z 413.97723	m/z 501.97093
Intensity: 1.146e+08	Intensity: 4.037e+08	Intensity: 5.004e+07	Intensity: 1.933e+07	Intensity: 8.211e+06
Accuracy: 0.67 ppm	Accuracy: 0.63 ppm	Accuracy: -0.03 ppm	Accuracy: 0.60 ppm	Accuracy: 0.68 ppm
Resolution: 120,608	Resolution: 86,983	Resolution: 67,787	Resolution: 48,974	Resolution: 44,410

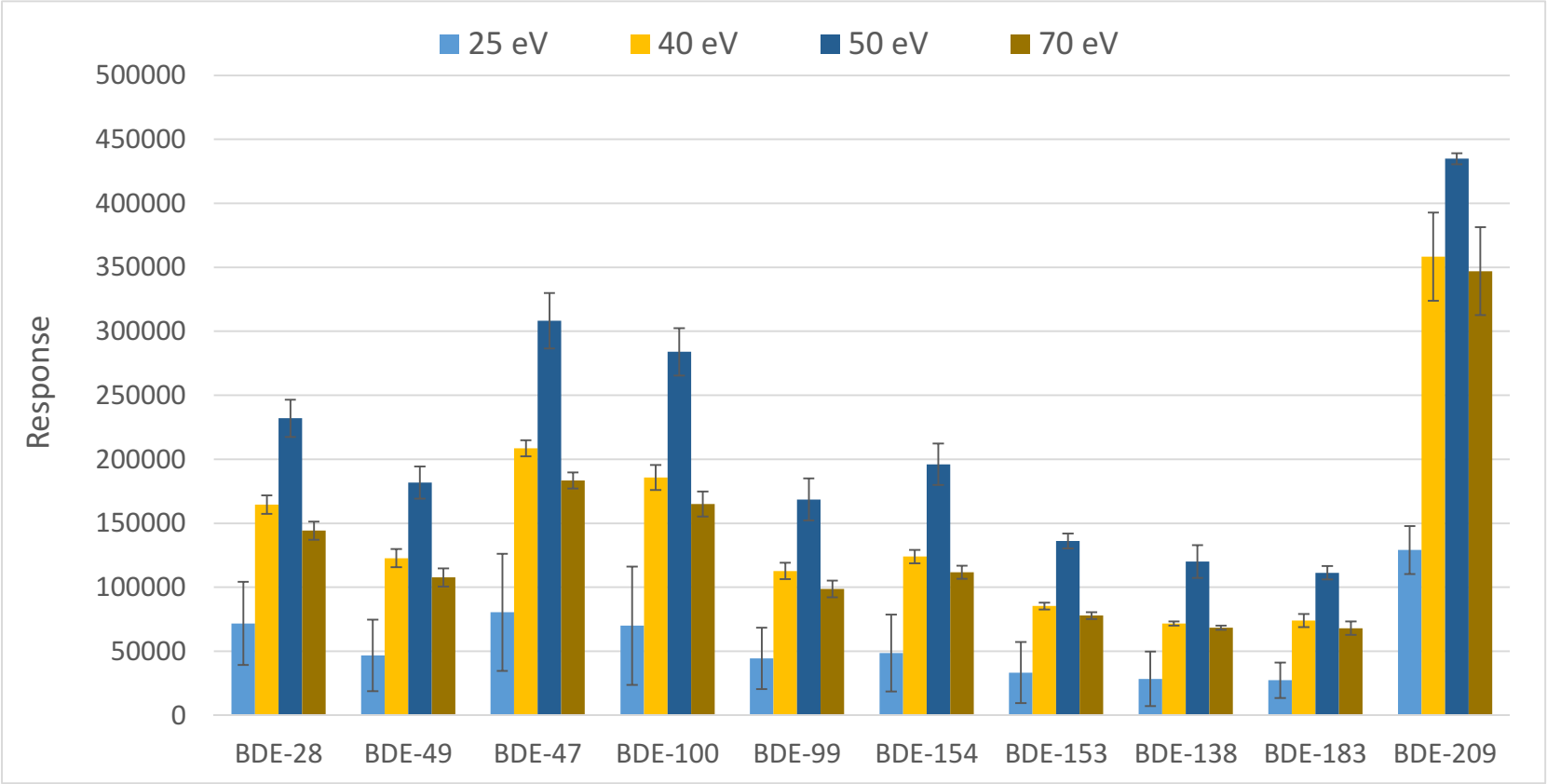




Tune values	
Source offset voltage * (V)	**3.5**
Electron lens voltage * (V)	**15.0**
Emission current * (uA)	**50.0**
Electron energy * (eV)	**50**
Repeller voltage * (V)	**12.75**
Lens 1 voltage * (V)	**36.25**
Lens 2 voltage * (V)	**0.5**
Lens 3 voltage * (V)	**27.75**
C-Trap energy offset * (V)	**0.0**

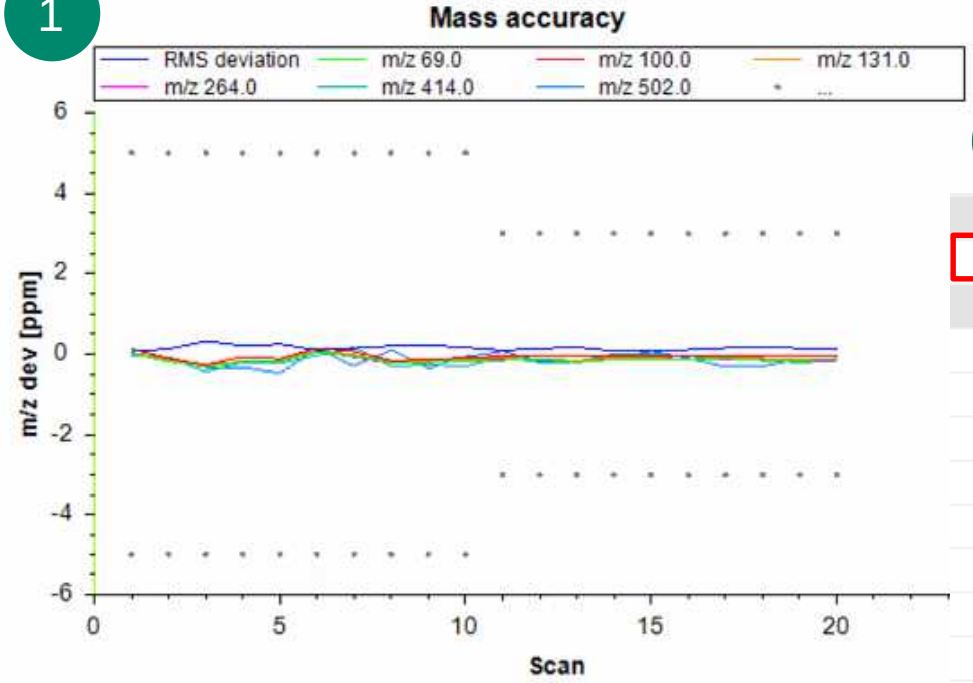
Optimization of EI Source Parameters

Tune values	
Source offset voltage * (V)	**3.5**
Electron lens voltage * (V)	**15.0**
Emission current * (uA)	**50.0**
Electron energy * (eV)	**50**
Repeller voltage * (V)	**12.75**
Lens 1 voltage * (V)	**36.25**
Lens 2 voltage * (V)	**0.5**
Lens 3 voltage * (V)	**27.75**
C-Trap energy offset * (V)	**0.0**



High Mass Calibration

1



2 **Injection of your compounds**

3

Manual high mass calibration	No valid negative masses...
Include with mass calibration	**Yes**
Positive Mass List	
Total valid masses	2
Theoretical mass 1	**799.33288**
Observed mass 1	**799.33154**
Theoretical mass 2	**811.37314**
Observed mass 2	**811.37197**
Theoretical mass 3	**--None--**
Observed mass 3	**--None--**
Theoretical mass 4	**--None--**
Observed mass 4	**--None--**
Theoretical mass 5	**--None--**
Observed mass 5	**--None--**

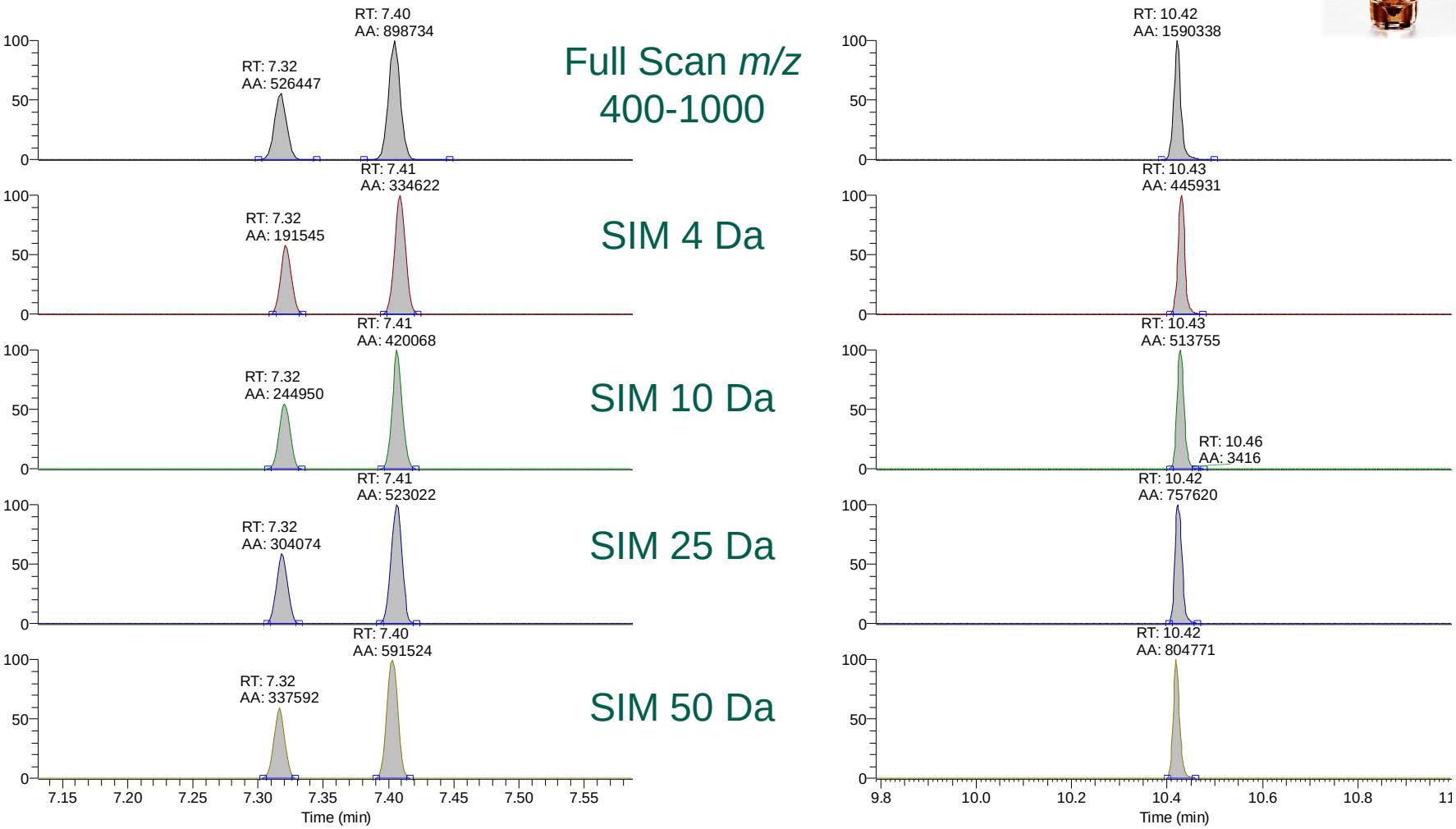
4

Manual High Mass List Added:
Theo mass: 799.33288 observed mass: 799.33154 frequency: 514.755915435
Theo mass: 811.37314 observed mass: 811.37197 frequency: 510.922291777

Procedure result: passed (rms = 0.13/0.19 ppm)

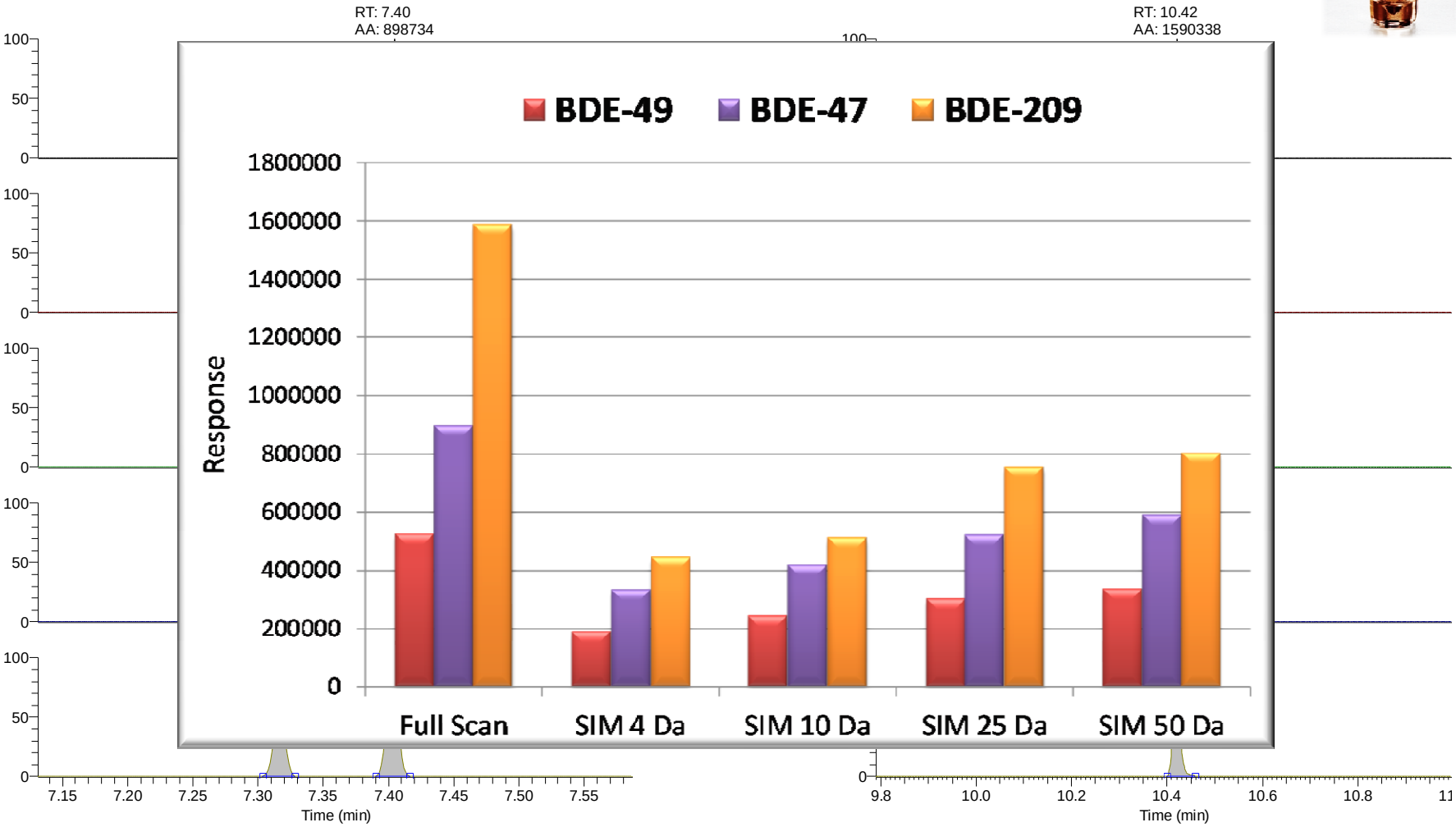
Acquisition modes

0.5 µg/L PBDE standard



Acquisition modes

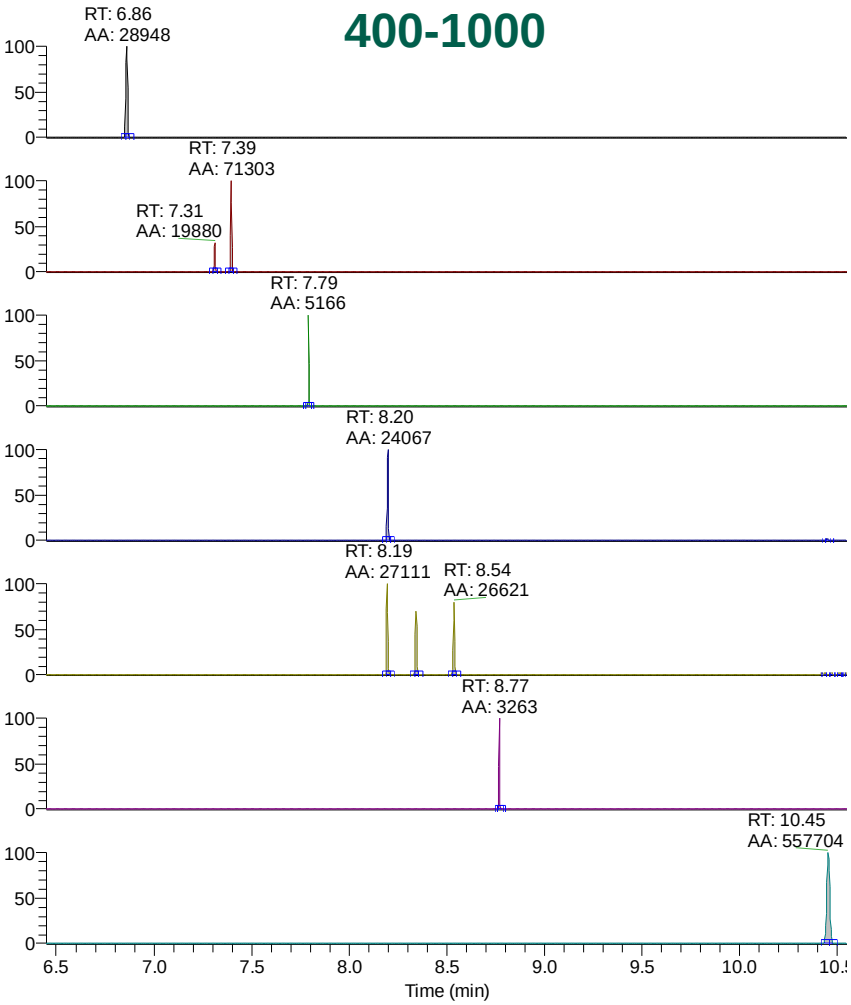
0.5 µg/L PBDE standard



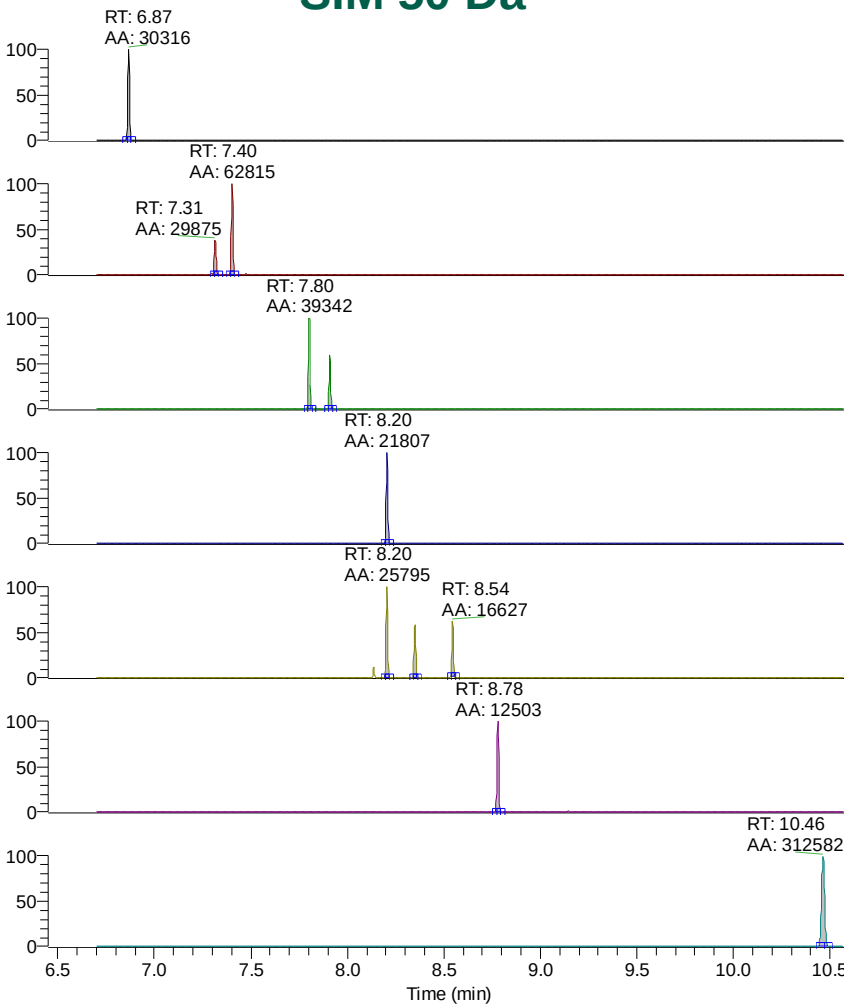
Acquisition modes

TUNA sample spiked with 0,01 ng/g

Full Scan *m/z* 400-1000



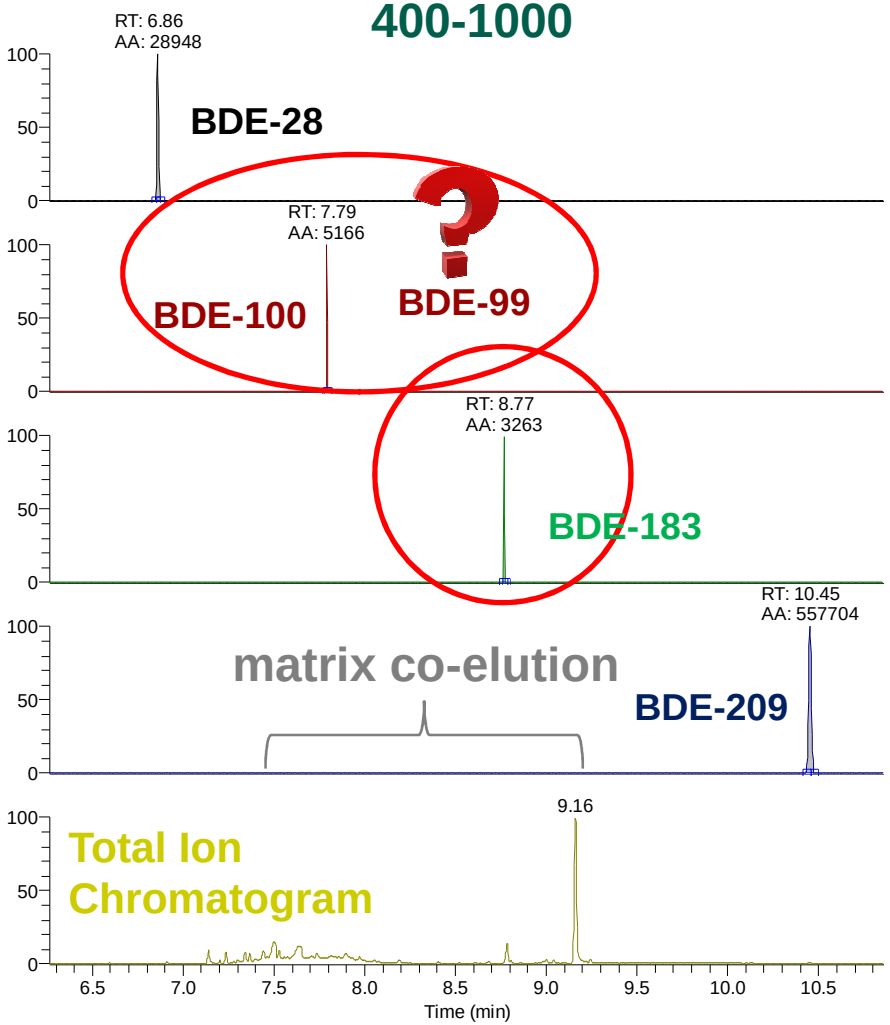
SIM 50 Da



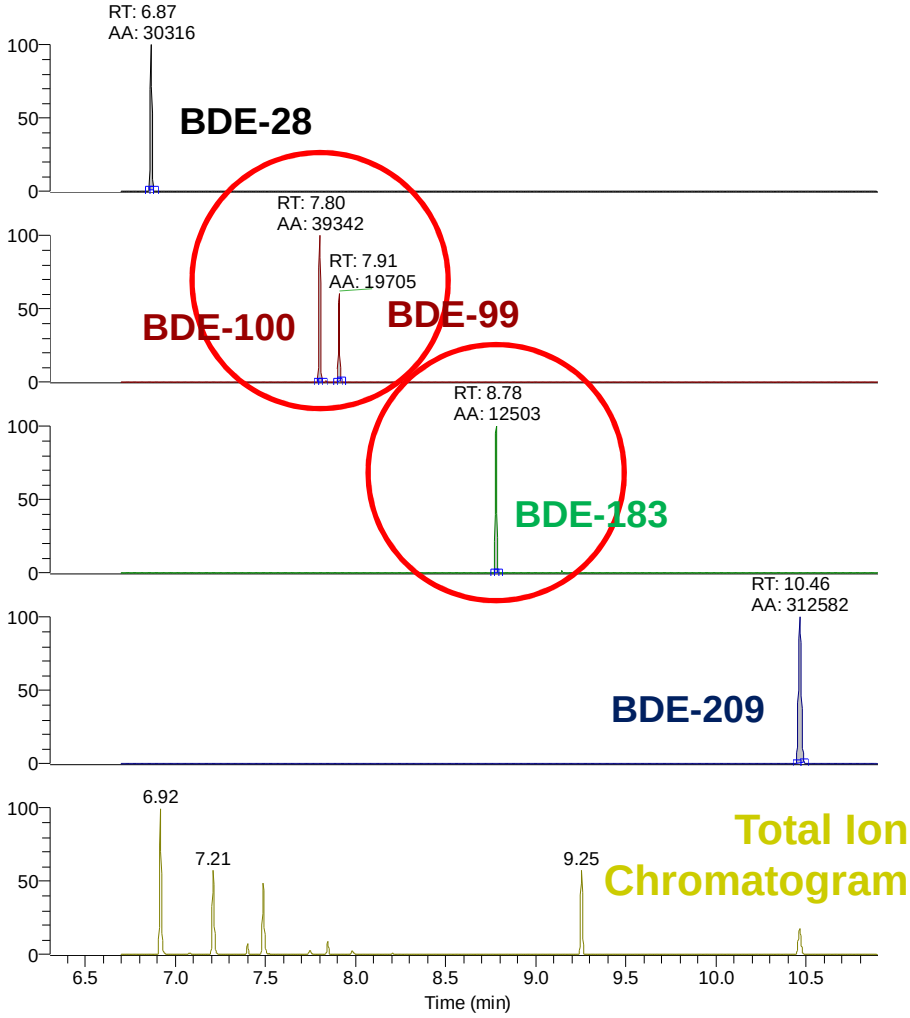
Acquisition modes

TUNA sample spiked with 0,01 ng/g

Full Scan *m/z* 400-1000



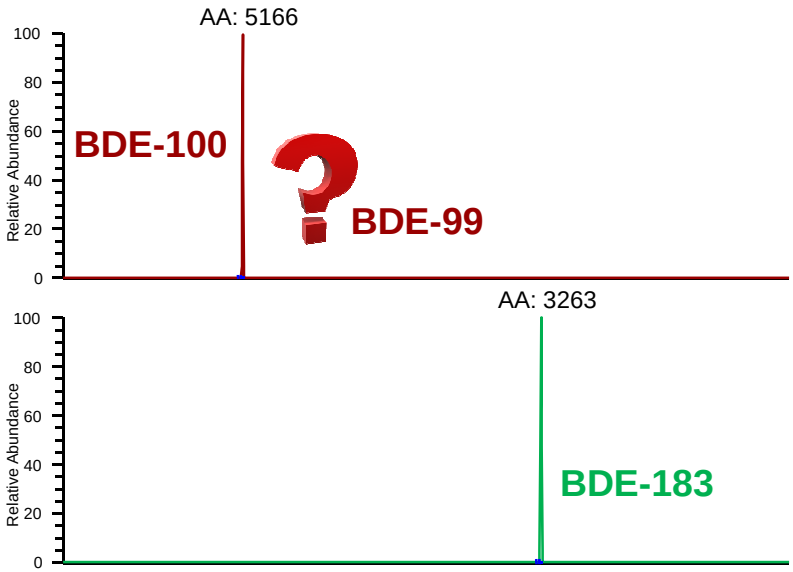
SIM 50 Da



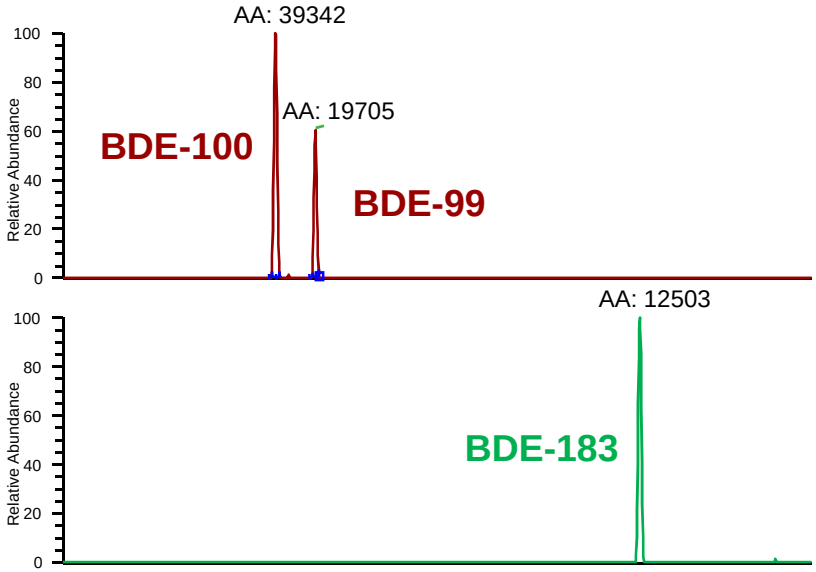
Acquisition modes

TUNA sample spiked with 0,01 ng/g

Full Scan *m/z* 400-1000



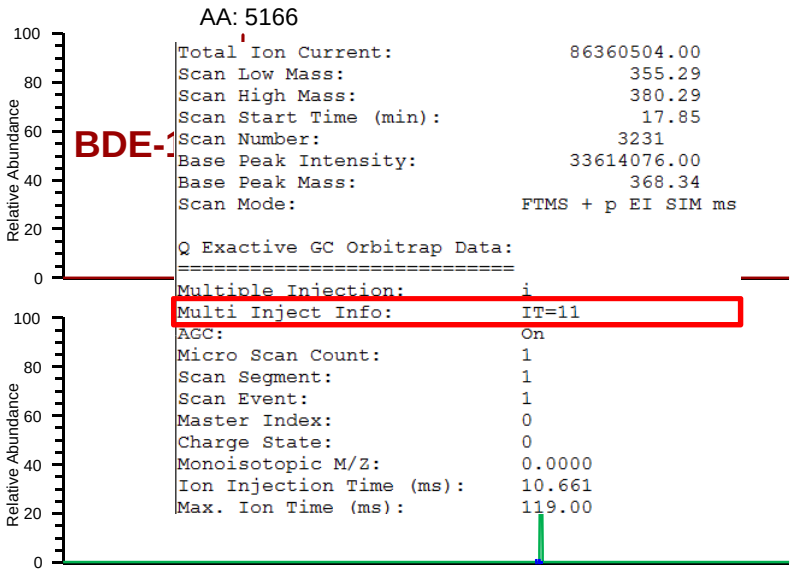
SIM 50 Da



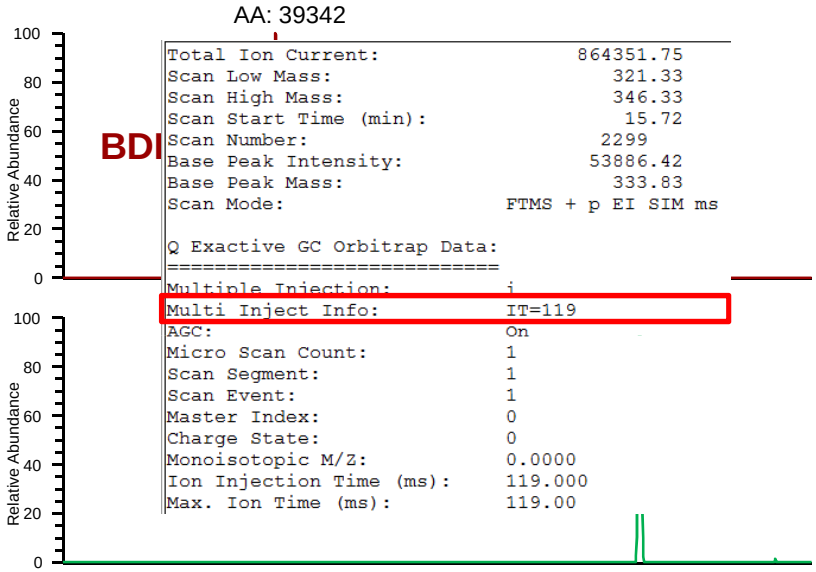
Acquisition modes

TUNA sample spiked with 0,01 ng/g

Full Scan *m/z* 400-1000



SIM 50 Da



Ion Time: 11 ms

Ion Time: 119 ms

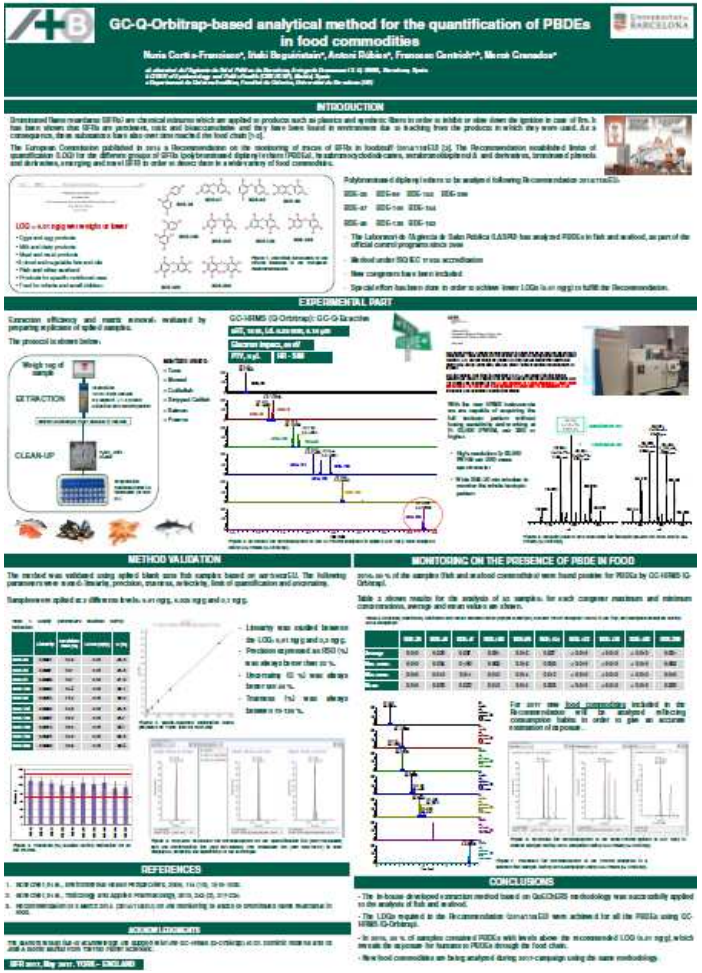
C- trap saturation due to matrix is overcome using the Q!

Polybrominated diphenyl ethers (PBDEs)

- PTV injection
- 5HT, 15 m, i.d. 0.25 mm, 0.10 µm GC column
- Optimized Tune parameters
- High Resolution (≥ 30,000 FWHM *m/z* 200)
- Wide SIM: 50 *m/z* window to monitor the whole isotopic pattern
- Mass accuracy < 1ppm
- External calibration (high mass calibration)

Method validation performed in TUNA FISH

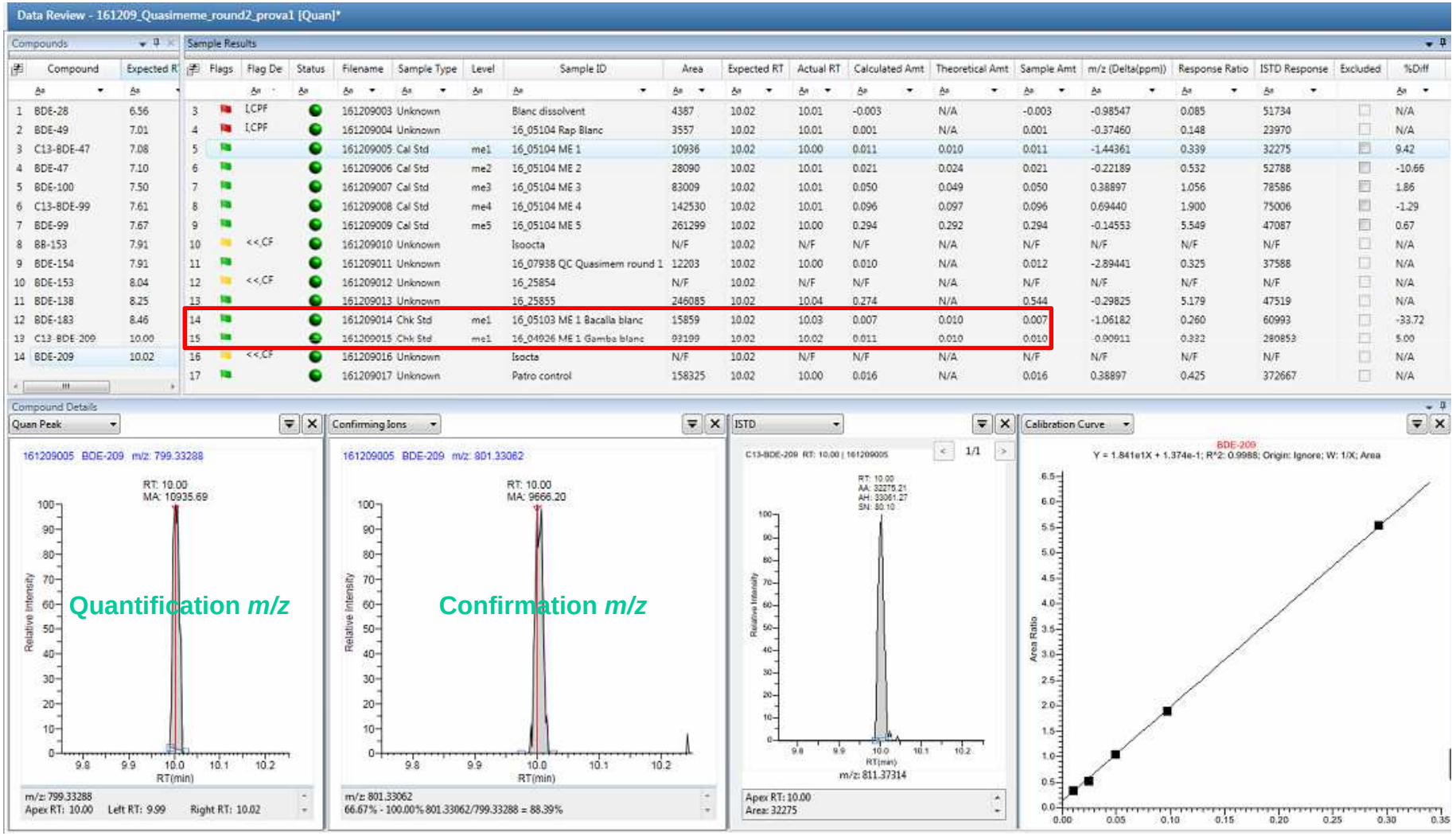
Quality Parameters **POSTER 18**



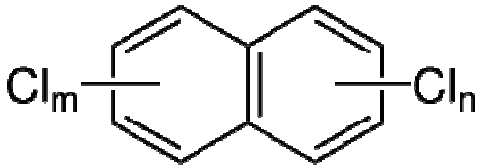
Polybrominated diphenyl ethers (PBDEs)

Monkfish extracted matrix-matched calibration curve
0.01 ng/g to 0.3 ng/g PBDEs

BDE-209



Polychloronaphtalenes (PCNs)



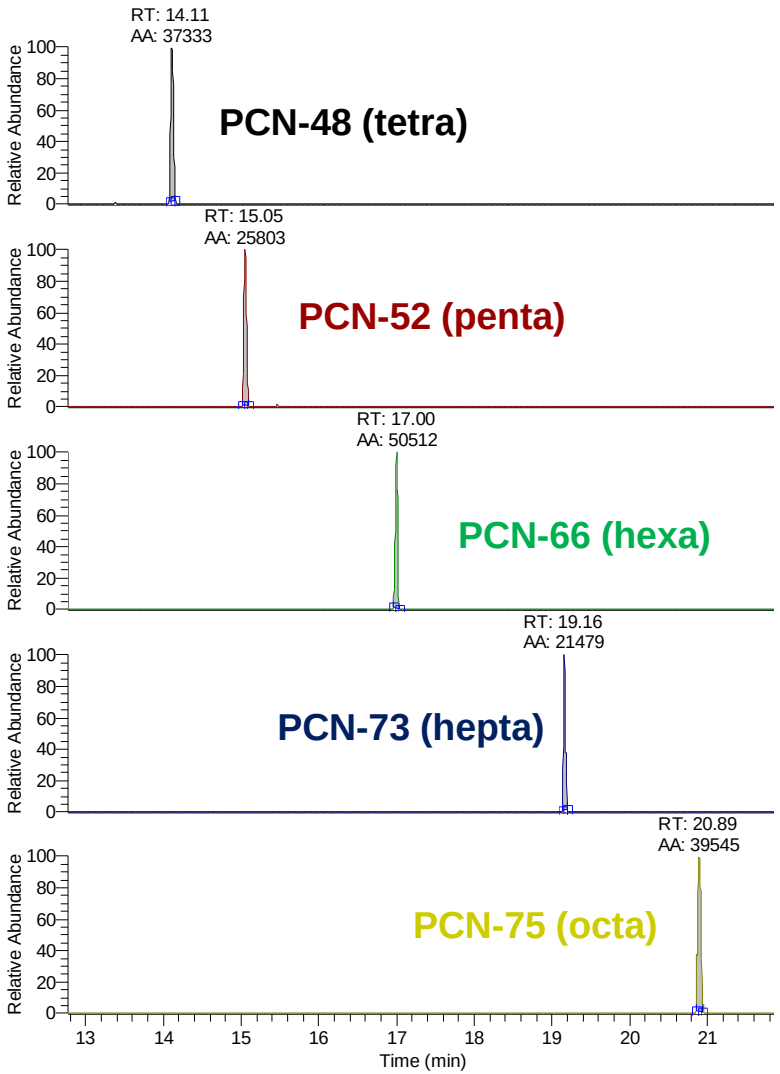
Listed under Annex A and C with specific exemptions for use

Samples analyzed

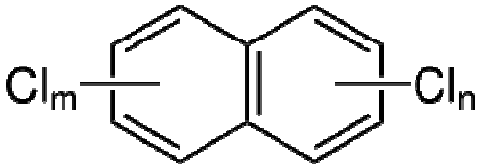
- Fish and sea products
- Eggs and egg products

Instrumental Analysis

- PTV injection
- HP-5MS, 30 m, i.d. 0.25 mm, 0,25 µm GC column
- Optimized Tune parameters
- High Resolution (≥ 60,000 FWHM *m/z* 200)
- Wide SIM: 25 *m/z* window to monitor the whole isotopic pattern
- Mass accuracy < 1ppm
- External calibration



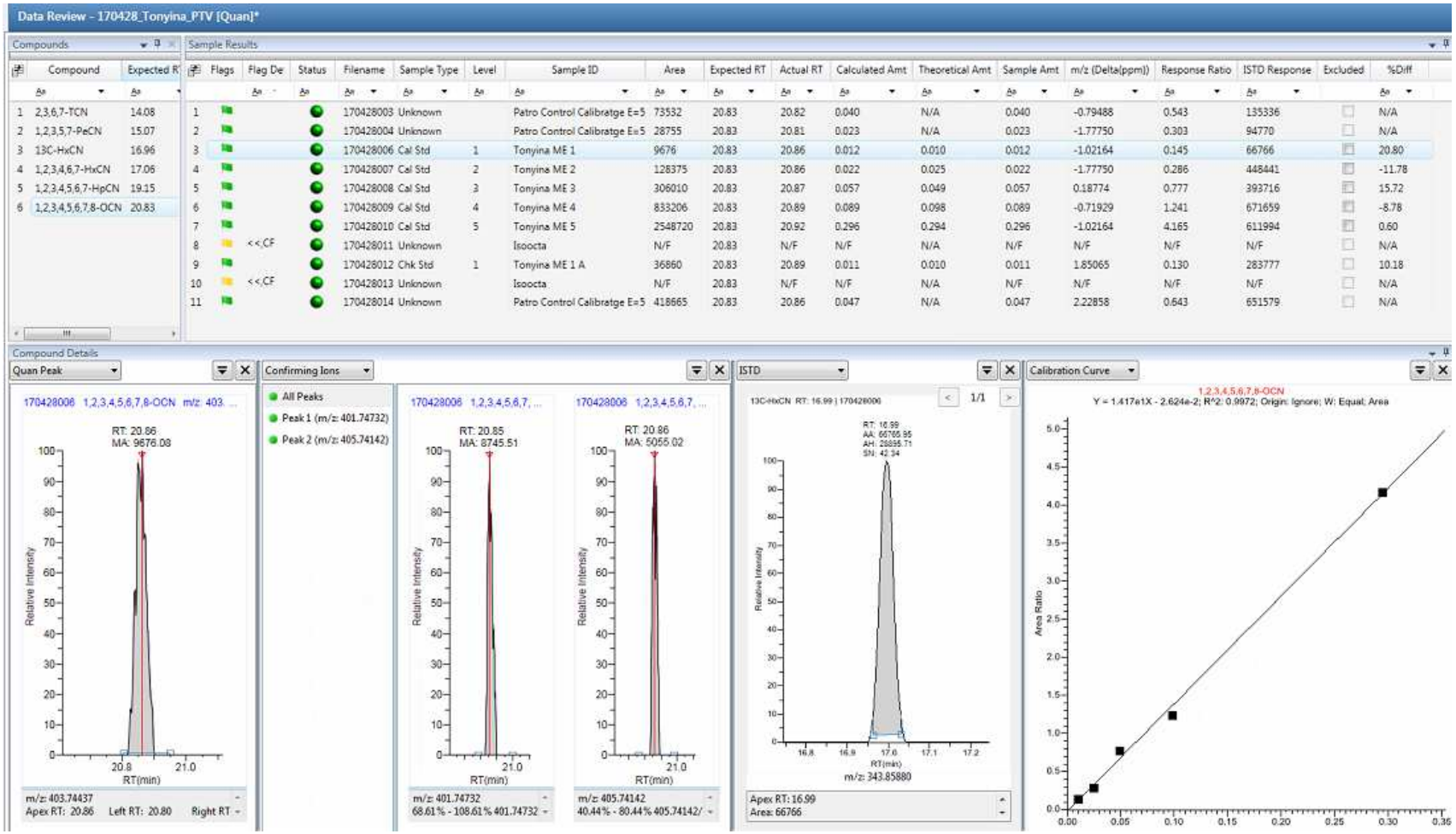
Polychloronaphtalenes (PCNs)



If m=4 and n=4

PCN-75: Octachloronaphthalene

LOQ= 0,01 ng/g wet weight



Quantification *m/z*

Confirmation *m/z*

Non-dl-Polychlorobiphenyls (n-dl-PCBs)

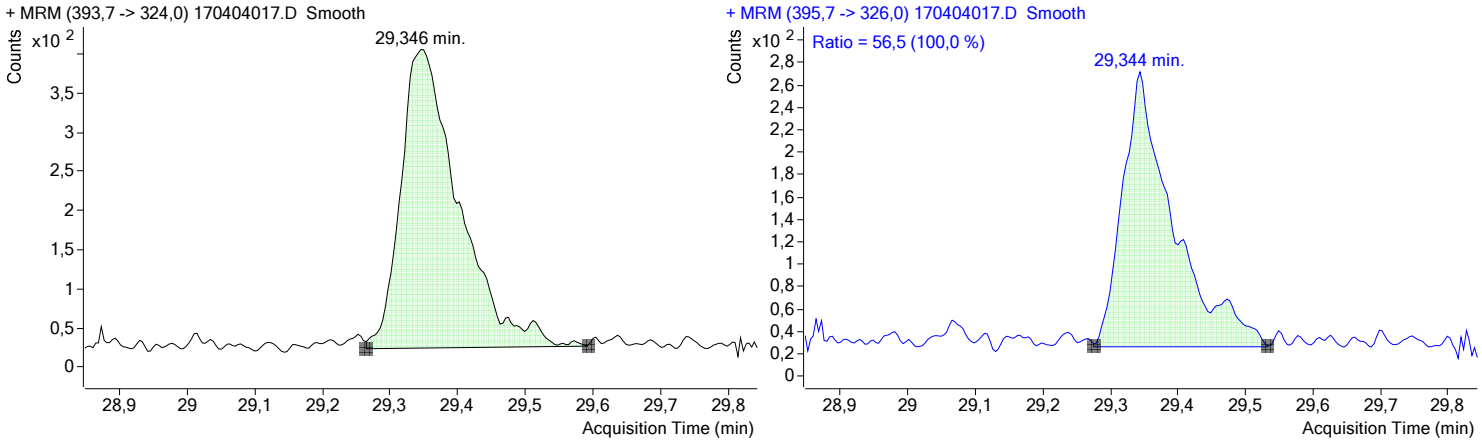
Analytical requirements for the official control of the levels of non-dioxin-like PCBs:

PCB-180

0,08 ng/g wet weight

≥ 0,100 ng/g fat

GC-MS/MS (QqQ)



Non-dl-Polychlorobiphenyls (n-dl-PCBs)

Analytical requirements for the official control of the levels of non-dioxin-like PCBs:

PCB-180

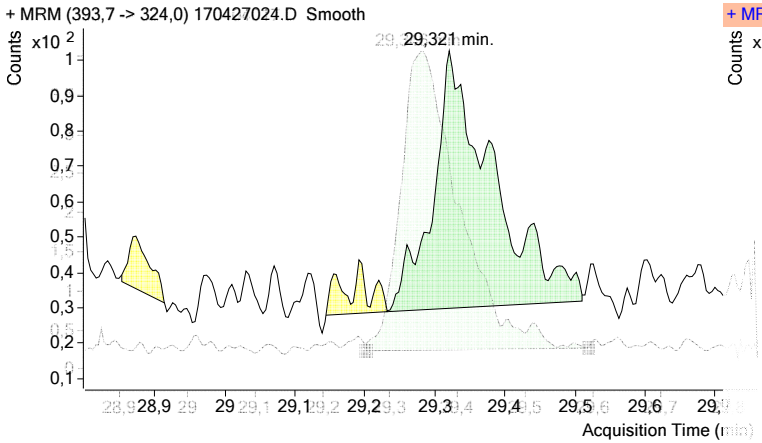
0,08 ng/g wet weight

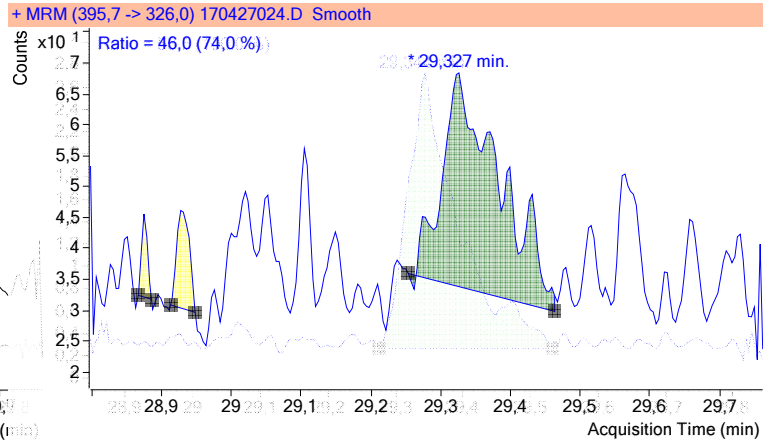
≥ 0,100 ng/g fat

0,025 ng/g wet weight

≥ 0,031 ng/g fat

GC-MS/MS (QqQ)





6.4.2017 EN Official Journal of the European Union L 92/9

COMMISSION REGULATION (EU) 2017/644

of 5 April 2017

laying down methods of sampling and analysis for the control of levels of dioxins, dioxin-like PCBs and non-dioxin-like PCBs in certain foodstuffs and repealing Regulation (EU) No 589/2014

3. **Demonstration of performance of method:**

Validation in the range of the maximum level (0,5 to 2 times the maximum level) with an acceptable coefficient of variation for repeated analysis (see requirements for intermediate precision in point 8).

4. **Limit of quantification:**

The sum of the LOQs (2) of non-dioxin-like PCBs shall not be higher than one-third of the maximum level (3).

5. **Quality control:**

Regular blank controls, analysis of spiked samples, quality control samples, participation in interlaboratory studies on relevant matrices.

Non-dl-Polychlorobiphenyls (n-dl-PCBs)

Analytical requirements for the official control of the levels of non-dioxin-like PCBs:

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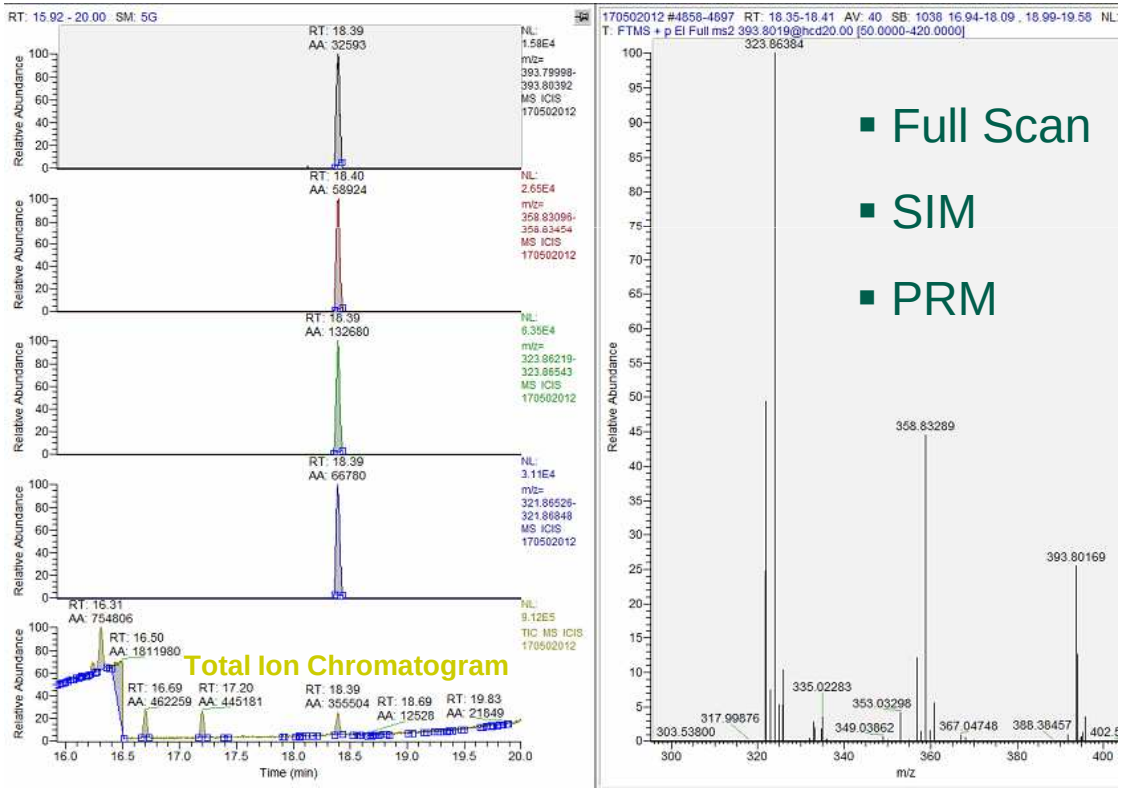
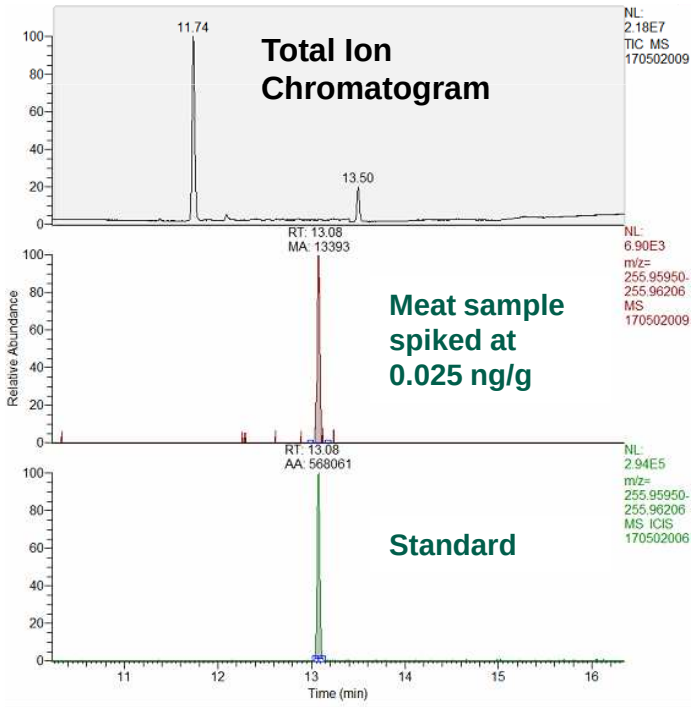
PCB-180



- Full Scan
- SIM
- PRM

PCB-28



Conclusions

GC-Orbitrap is a useful tool for control labs:

- outstanding sensitivity
- high selectivity when analyzing complex matrices
- high robustness
- helps to fulfill new Regulations with extremely low limits (e.g. PCBs, PBDEs)

But some expertise and optimization of instrumental parameters is needed.

Thanks for your attention

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Agència de Salut Pública