CONffIDENCE: Contaminants in food and feed: Inexpensive detection for control of exposure

Work package WP1a – Persistent Organic Pollutants (POPs)

Implementation of GC×GC-TOFMS for the simultaneous determination of PCBs, PBDEs and PAHs in environmental samples

INSTITUTE OF CHEMICAL TECHNOLOGY PRAGUE

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Introduction

- Polychlorinated biphenyls (PCBs), polybrominated diphenylethers (PBDEs), and polycyclic aromatic hydrocarbons (PAHs) represent the major groups of ubiquitous environmental pollutants that might be transferred into human food chains.
- Since PCBs, PBDEs, and PAHs belong, according to EFSA, among food contaminants that should be monitored, the quick, rugged, sensitive and inexpensive analytical method is currently required.[1]
- Comprehensive two-dimensional gas chromatography (GC×GC) coupled to time-of-flight mass spectrometry (TOFMS) represents a powerful tool for simultaneous determination of different types of contaminants that considerably increase the separation efficiency of GC analysis.[2,3]

MAIN GOALS OF THE CONffIDENCE PROJECT

- To develop and validated a simplified sample preparation strategy for the simultaneous determination of a wide range of contaminants in food and feed focused on fish, fish feed and cereal based baby food.
- To implement a GC×GC-TOFMS for the determination of PCBs, PBDEs and PAHs in food and feed in a single run.

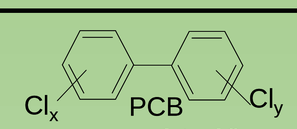
Aim of the study

- To develop and optimize the GC×GC-TOFMS method for the simultaneous determination of PCBs, PBDEs and PAHs to obtain the best chromatographic resolution and detection limits for all target analytes
- To test several chromatographic capillary column combinations with different polarities – BPX-5, BPX-50 and Rxi-17Sil-ms in the 1st dimension and BPX-50, Rt-LC-35 and HT-8 in 2nd dimension
- To optimize a programmable temperature vaporization (PTV) injection technique.

Target analytes

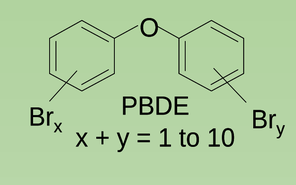
Dioxin-like polychlorinated biphenyls (PCBs)

- Non-*ortho* congeners #77, 81, 126, 169
- Mono-*ortho* congeners #105, 114, 118, 123, 156, 157, 167, 189



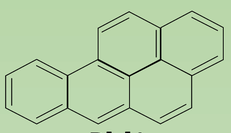
Brominated flame retardants (BFRs)

- Polybrominated diphenylethers congener (PBDEs) #28, 47, 99, 100, 153, 154, 183
- Hexabromocyclododecane (HBCD)
- Polybrominated biphenyl (PBB): congener #153



Polycyclic aromatic hydrocarbons (PAHs)

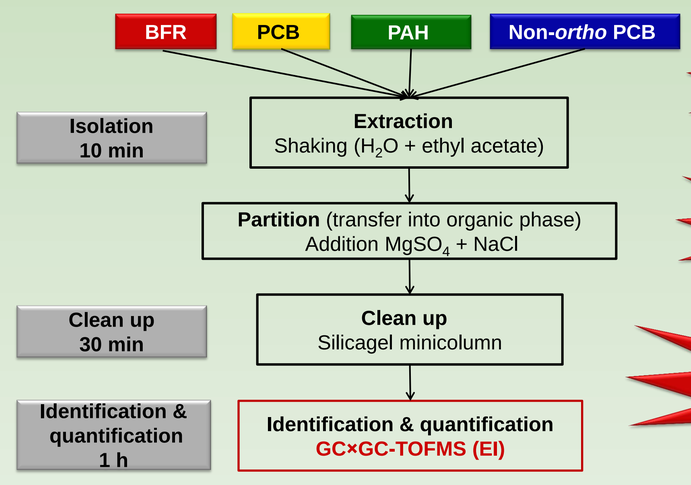
Benz(a)anthracene – BaA
Benzo(a)pyrene – BaP
Benzo(b)fluoranthene – BbFA
Benzo(c)fluoren – BcFL
Benzo(j)fluoranthene – BjFA
Benzo(k)fluoranthene – BkFA
Benzo(g,h,i)perylene – BghiP
Chrysene – CHR
Cyclopenta(c,d)pyrene – CPP
Dibenz(a,h)anthracene – DBahA
Dibenzo(a,e)pyren – DBaeP
Dibenzo(a,h)-pyrene – DBahP
Dibenzo(a,i)-pyrene – DBaiP
Dibenzo(a,l)pyrene – DBalP
Indeno(1,2,3-cd)pyrene – IP
5-Methylchrysene – 5 MC

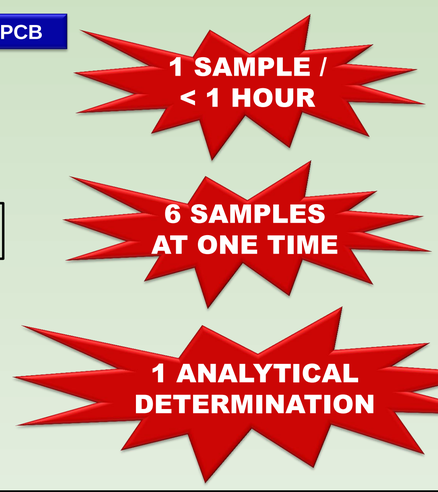


Tested matrices

- Standard reference material – SRM 1947 Lake Michigan Fish Tissue
- Standard reference material – SRM 1974b Mussel Tissue
- Non-smoked (25% fat) and smoked sprat (27% fat)

Analytical method





Results

An Agilent 6890N for comprehensive two-dimensional GC with a high speed TOFMS detector (Pegasus III, LECO Corp.)

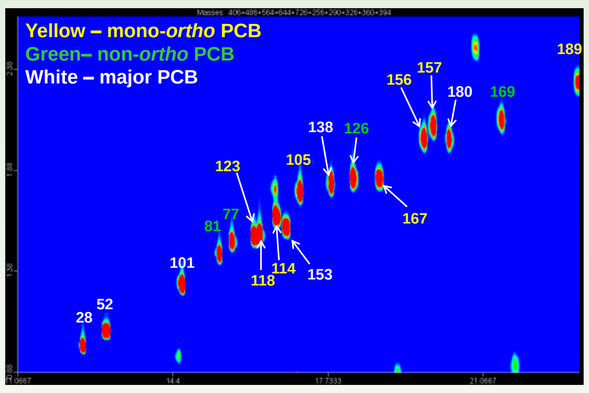


Figure 1 Separation of PCBs in standard solution (800 pg injected) on column system BPX-5 × BPX-50.

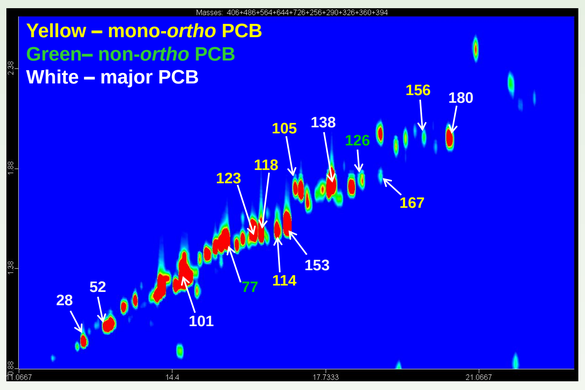


Figure 2 Separation of PCBs in standard reference material SRM1947 on column system BPX-5 × BPX-50.

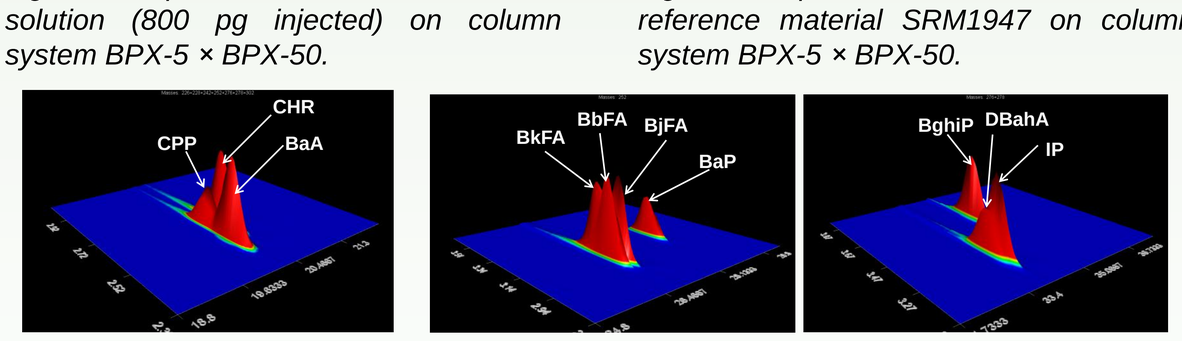


Figure 3 Separation of critical groups of PAHs on column system BPX-5 × BPX-50.

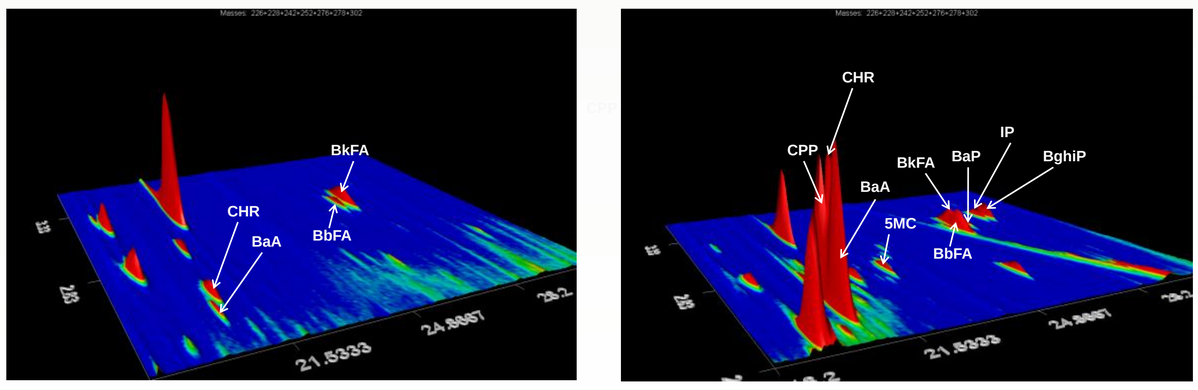


Figure 4 Separation of PAHs in non-smoked and smoked trout on column system BPX-5 × BPX-50

SEPARATION OF CRITICAL GROUPS OF PAHs

- 1st group: BaA, CPP and CHR
- 2nd group: BjFA, BkFA and BbFA
- 3rd group: DBahA, IP and BghiP

Table I Separation of critical groups of PAHs using different column systems

1st dim. 2nd dim.	BPX-5 BPX-50	BPX-5 Rt-LC-35	BPX-5 HT-8	BPX-50 BPX-5	BPX-50 HT-8	Rxi-17Sil-ms HT-8
1st group	✓	✗	✓	✗	✗	✗
2nd group	✓	✗	✗	✓	✓	✓
3rd group	✓	✗	✓	✗	✗	✗

Conclusions

- All PCBs and PBDEs were separated on all column systems except for (Rxi-17Sil-ms × HT-8) and (BPX-50 × HT-8) where PCB118 and 123 were co-eluted.
- Selection of the column system was mainly influenced by its ability to separate critical groups of PAHs.
- BaA, CPP and CHR (1st group) – best separation on BPX-5 × BPX-50.
- BjFA, BkFA and BbFA (2nd group) – best separation on BPX-50 × HT-8.
- DBahA, IP and BghiP (3rd group) – best separation on BPX-5 × HT-8.
- All PAHs were separated using column system BPX-5 × BPX-50.

References

1 http://www.efsa.europa.eu/EFSA/efsa_locale-1178620753812_1211902012171.htm (13/01/2011)
2 Borajandi L. R., Ramos J. J., Sanz J., González M. J., Ramos L.: J. Chromatogr. A, 1186 (2008), 312.
3 Korytár P., Leonards P. E. G., de Boer J., Brinkman U. A. Th.: J. Chromatogr. A, 1086 (2005), 29

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