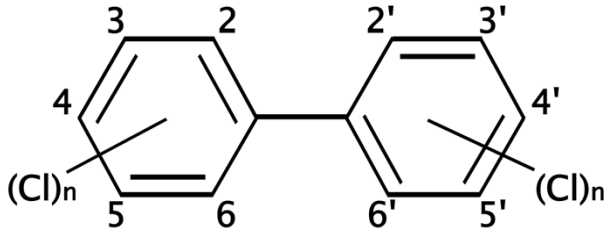
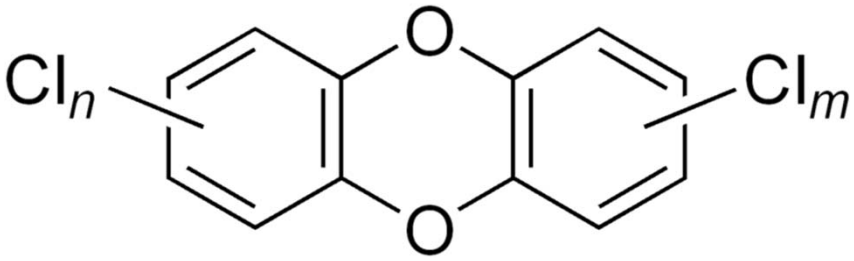
Automation of the sample purification method for the analysis of all 209 polychlorinated biphenyls and dioxins

Guillaume ten Dam
DSP-Systems, Darwinstraat 7A, 6718XR Ede, The Netherlands





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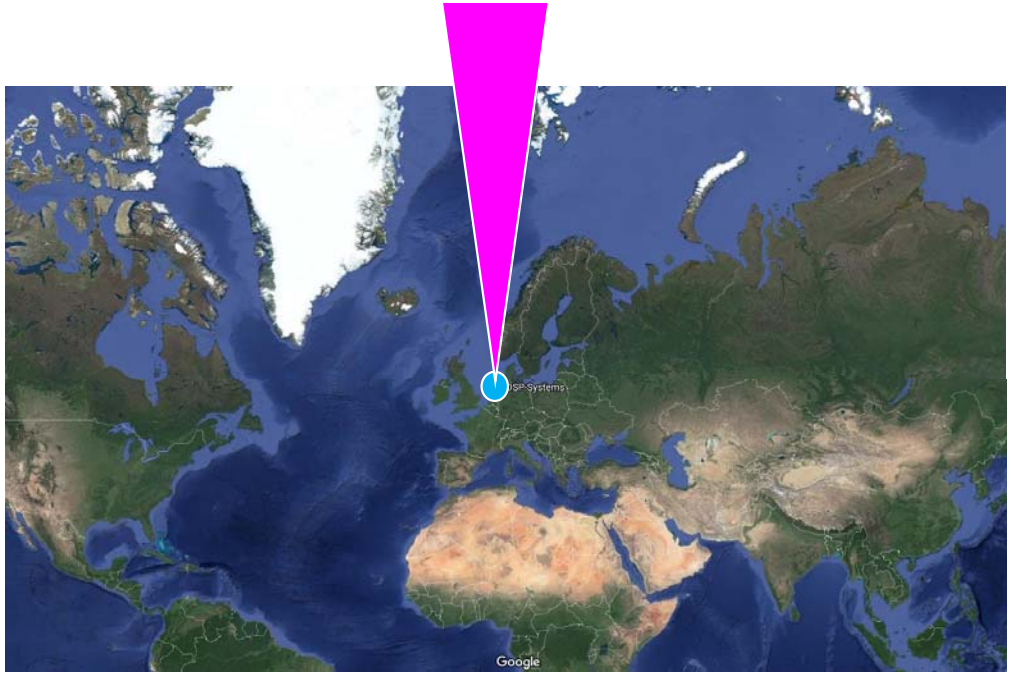
Dioxin Sample Preparation



Wim Traag

- Founder DSP-Systems
- Dioxin, PCB and POP analysis ≥ 40 years
- Collaborator in numerous international projects on chemical analysis

Chris van Wakeren

- Specialist in automated sample preparation ≥ 10 years
- Sales and technical expert

Guillaume ten Dam

- Dioxin, PCB and POP analysis ≥ 10 years
- Participated in EU workgroups

Automation of the sample purification method for the analysis of all 209 polychlorinated biphenyls and dioxins

Approach – Published and experimental

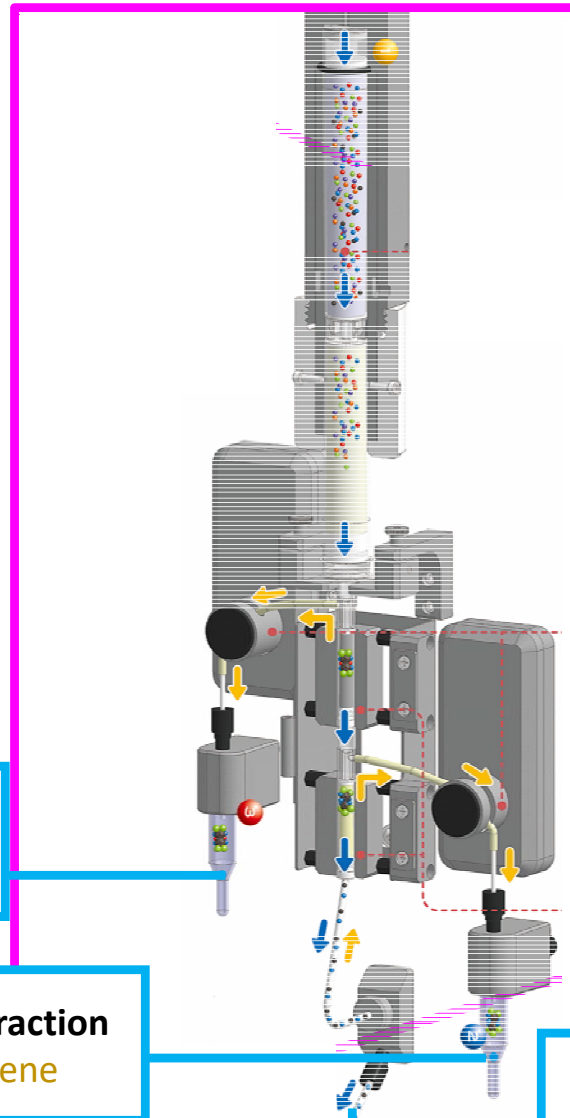
Marchand P. et al., Organohalogen Compounds Vol. 76, 546-549 (2014)
A new and highly innovative automatic purification system evaluated for dioxins and PCBs

Hayward D. G. et al., Chemosphere 256 (2020) 127023
New approach for removing co-extracted lipids before mass spectrometry measurement of persistent of organic pollutants (POPs)in foods

Markesteijn J., Thermo Fisher Scientific user meeting at Dioxin2019
Are You Being Served? The Benefits of DualData Acquisition in a Routine Dioxin Lab

ten dam G. et al., Journal of Chromatography A, Volume 1477, 16 December 2016, Pages 76-90
The performance of atmospheric pressure gas chromatography–tandem mass spectrometry compared to gas chromatography–high resolution mass spectrometry for the analysis of polychlorinated dioxins and polychlorinated biphenyls in food and feed samples

Design - Goal



Dioxin & no-PCB fraction
in approx. 1.5 ml toluene

EN 16215:2012

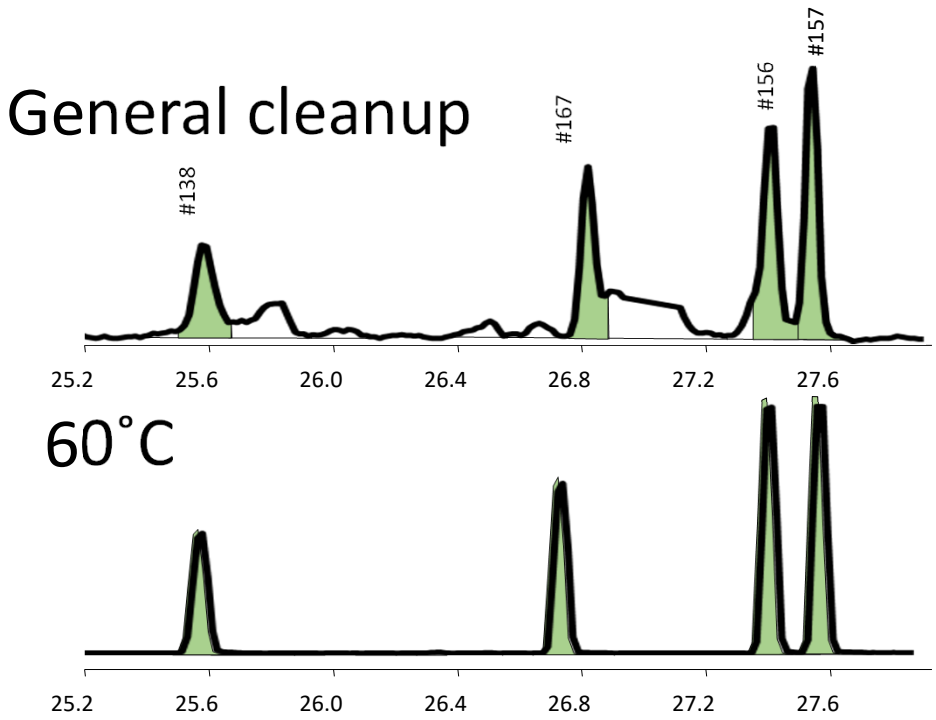
mo-PCB & marker PCB fraction
in approx. 1.5 ml toluene

Waste
approx. 90 ml hexane

Recoveries Dioxin and DL-PCB - *EN 16215:2012*

Advantages

- Fractions of 1.5 ml in a GC-vial
- Low solvent consumption (<100 ml)
- No Dichloromethane needed
- Purification finished within 80 min
- No cross-contamination
- No exposure to chemicals

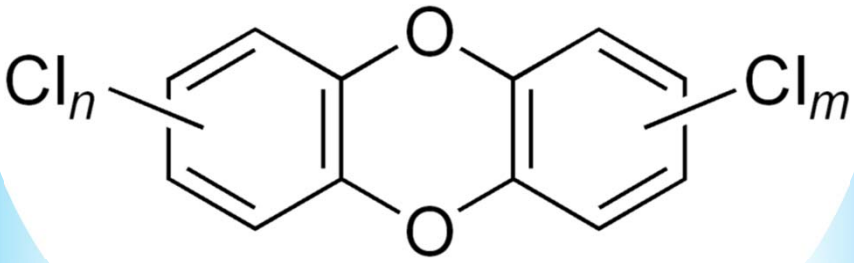


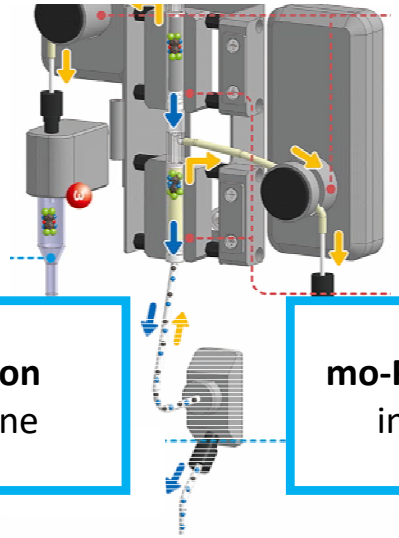
WHO Dioxin & DL-PCB and marker PCB

Type Size[mm]		***Standard*** ***20φ***
Sample		Food 3 g fat
Purification		$AgNO_3$-Silica gel
		H_2SO_4-Silica gel
Concentration		Carbon
		Alumina
Elution	To waste	90mL Hexane
	DXN fraction	1.5 mL Toluene
	PCB fraction	1.5 mL Toluene
Run time		80 min

ten Dam et al., NEMC 2020, A Next Generation Automated Purification System for the Analysis of Dioxins and Associated POPs

So what about all 209PCB?
EPA method 1618





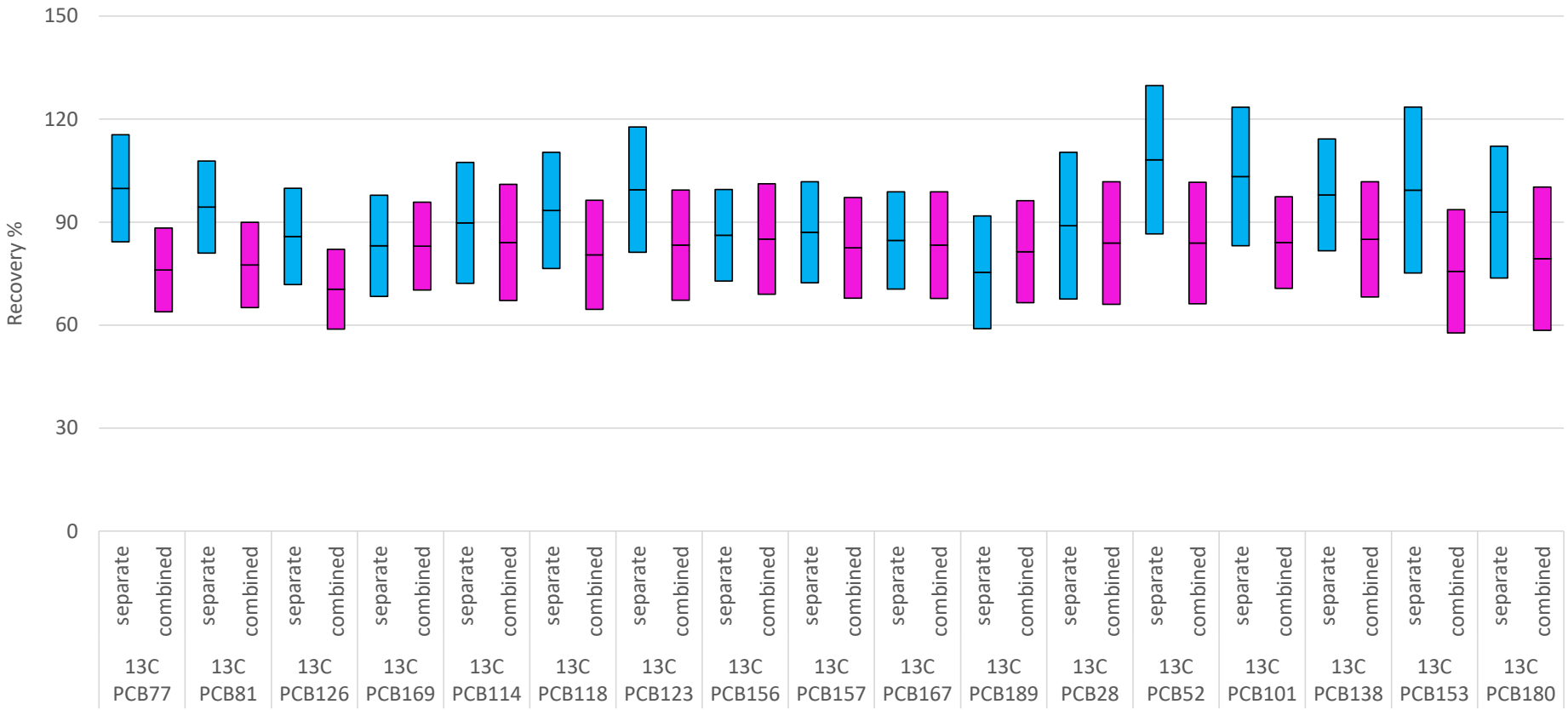
Dioxin & no-PCB fraction
in approx. 1.5 ml toluene

mo-PCB & marker PCB fraction
in approx. 1.5 ml toluene

Dioxin & 209PCB?

Recovery ^{13}C DL-PCB and ^{13}C NDL-PCB in samples

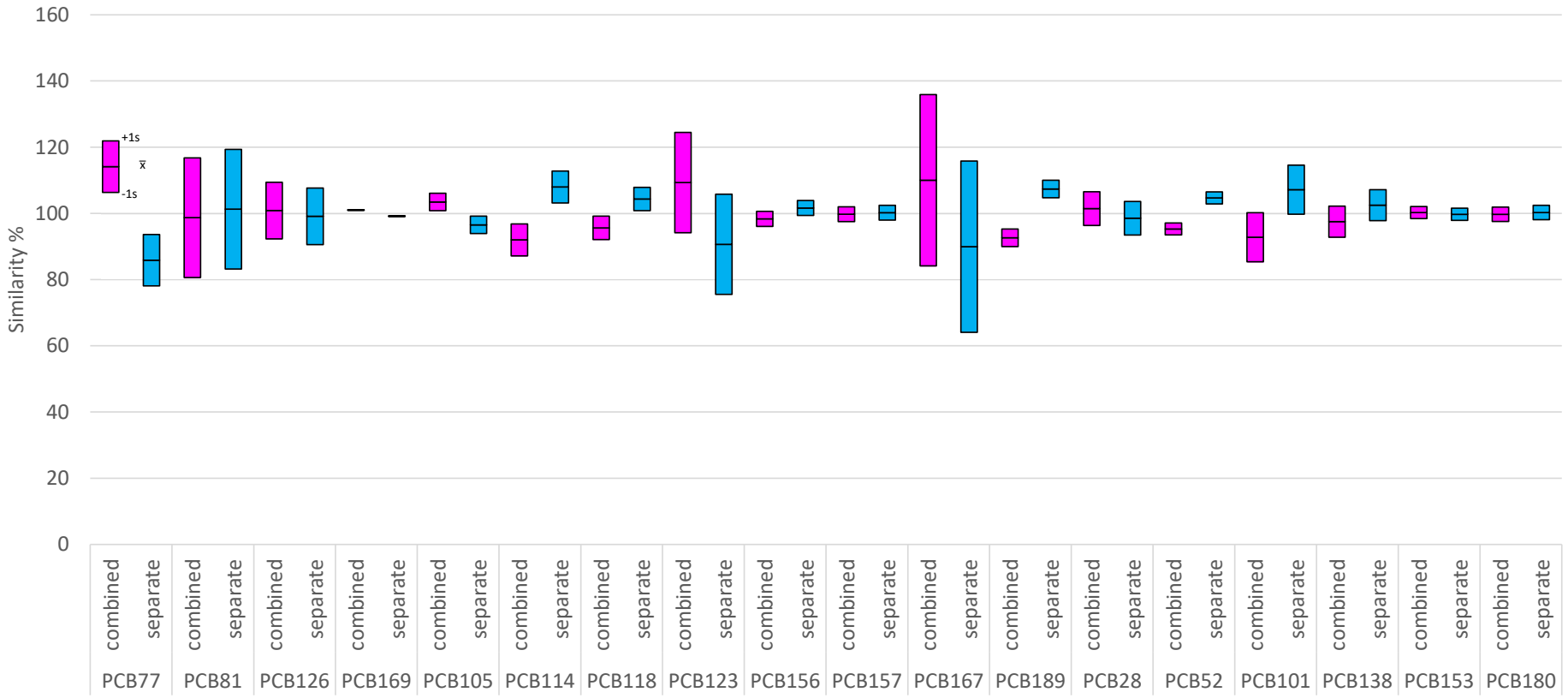
conventional 2 run analysis (blue) vs single run analysis (pink)



ten Dam et al., DSP-Systems Application note 2019:03, Performance study of the Determination of DL-PCBs and NDL-PCBs in one single GC-HRMS measurement using a Miura GO-xHT for Sample Purification (Part I; Food and Feed)

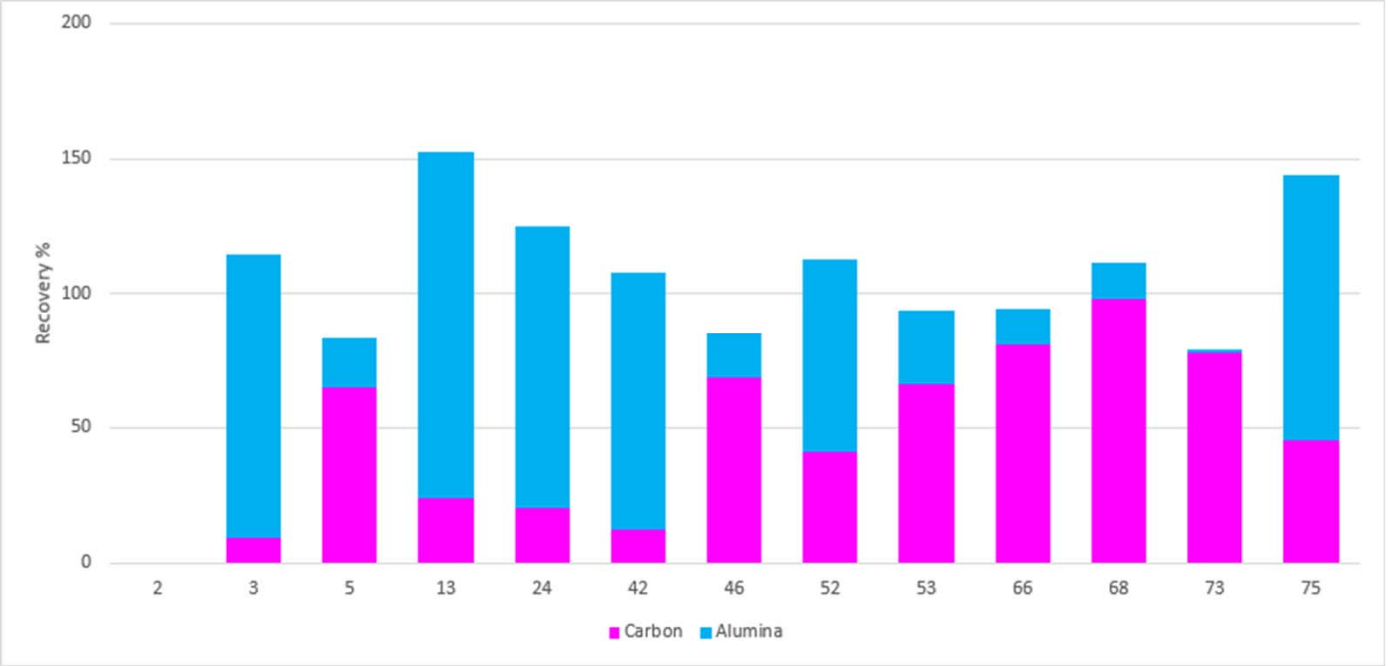
Similarity DL-PCB and NDL-PCB

single run analysis (pink) vs conventional 2 run analysis (blue)

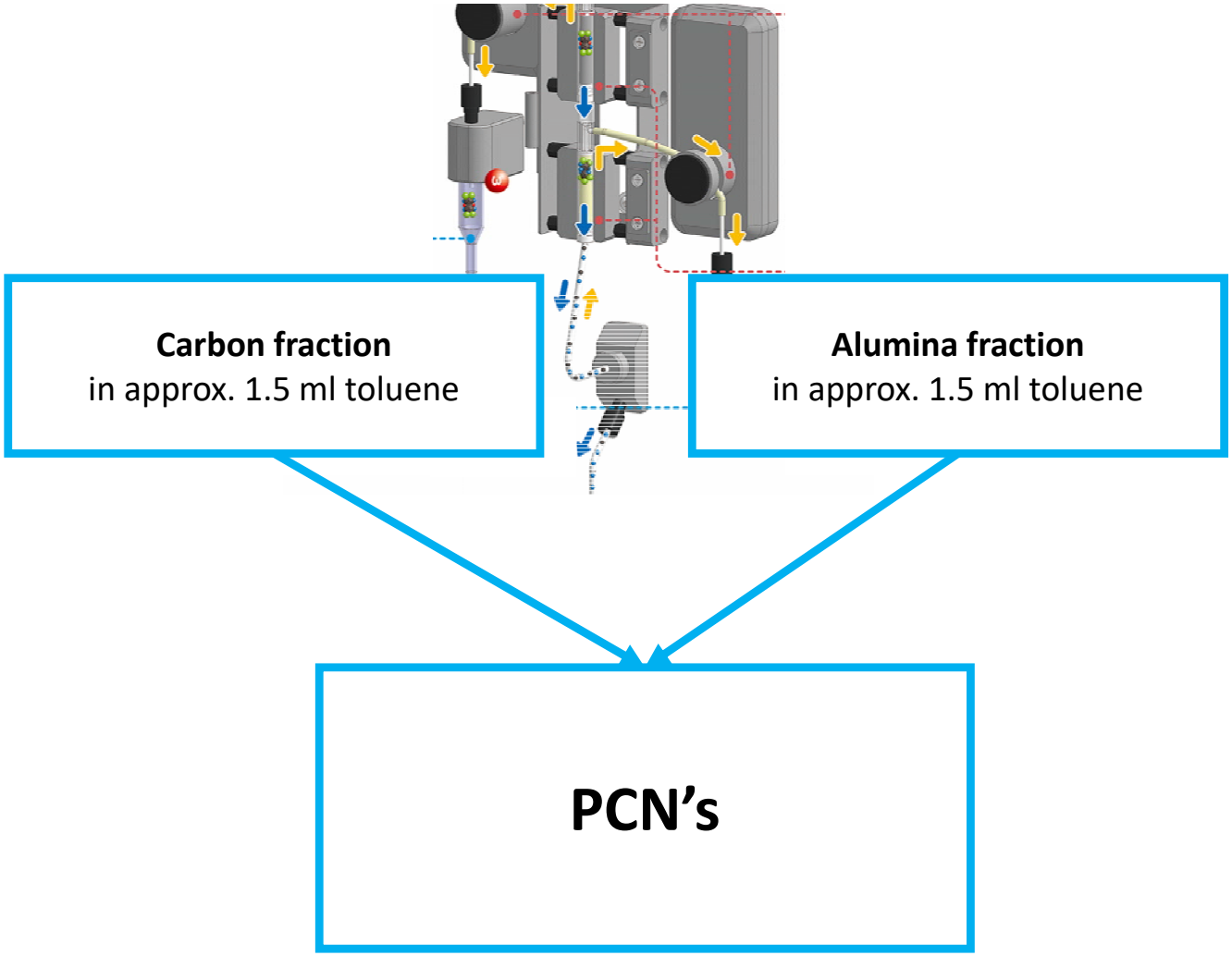


ten Dam et al., DSP-Systems Application note 2019:03, Performance study of the Determination of DL-PCBs and NDL-PCBs in one single GC-HRMS measurement using a Miura GO-xHT for Sample Purification (Part I; Food and Feed)

Experience with lower chlorinated congeners Polychlorinated naphthalene's



ten Dam et al., DSP-Systems application note 2018:12, Feasibility study for the Determination of Polychlorinated Naphthalenes in Food and Feed samples



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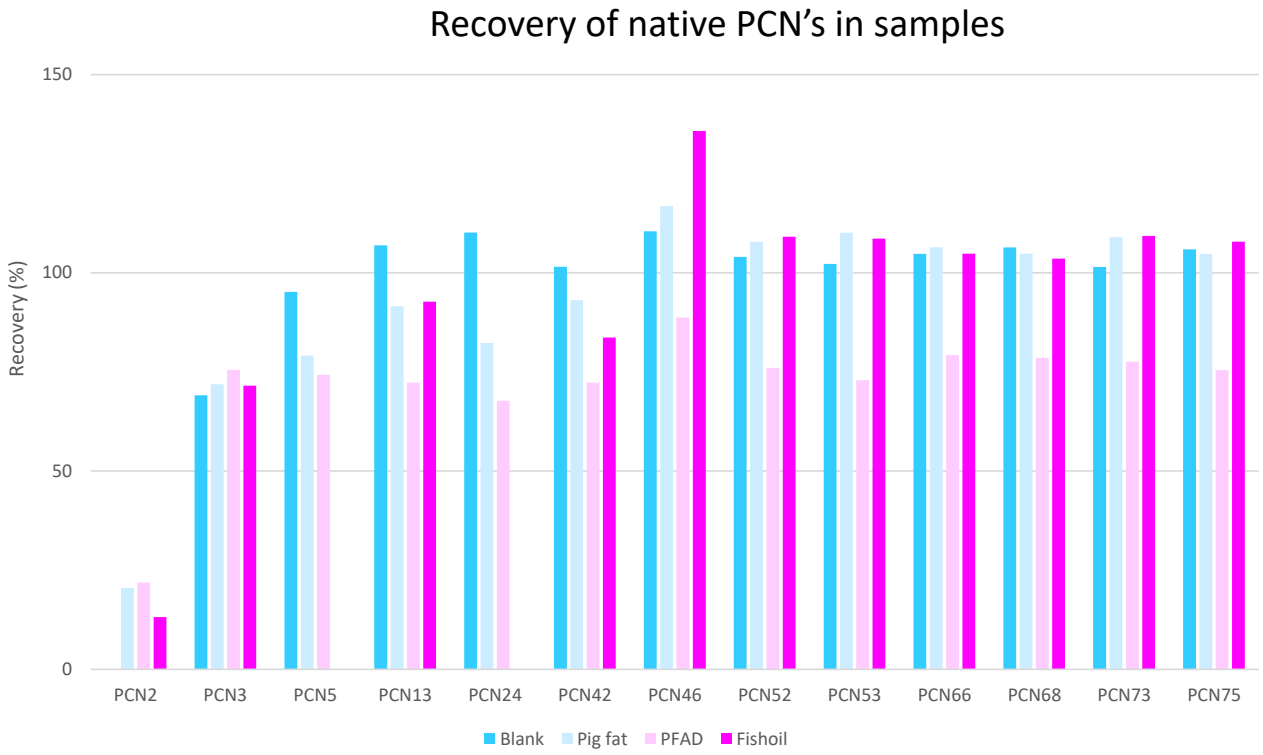
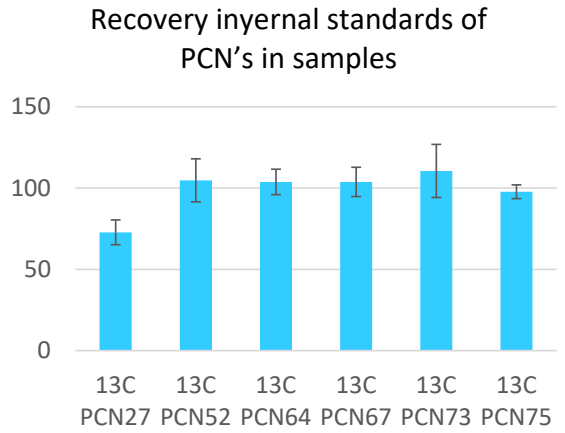
Experience with lower chlorinated congeners Polychlorinated naphthalene's





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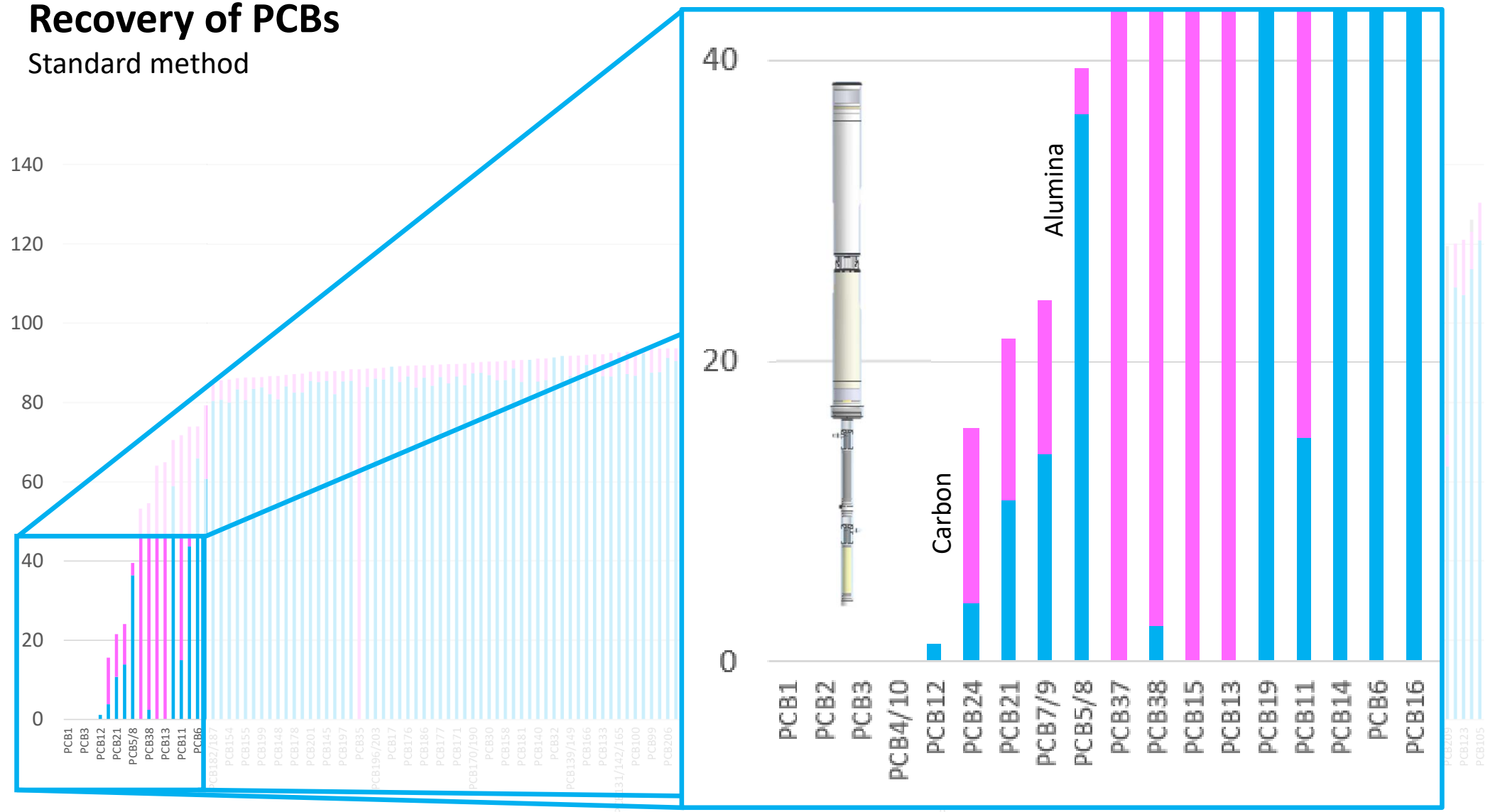
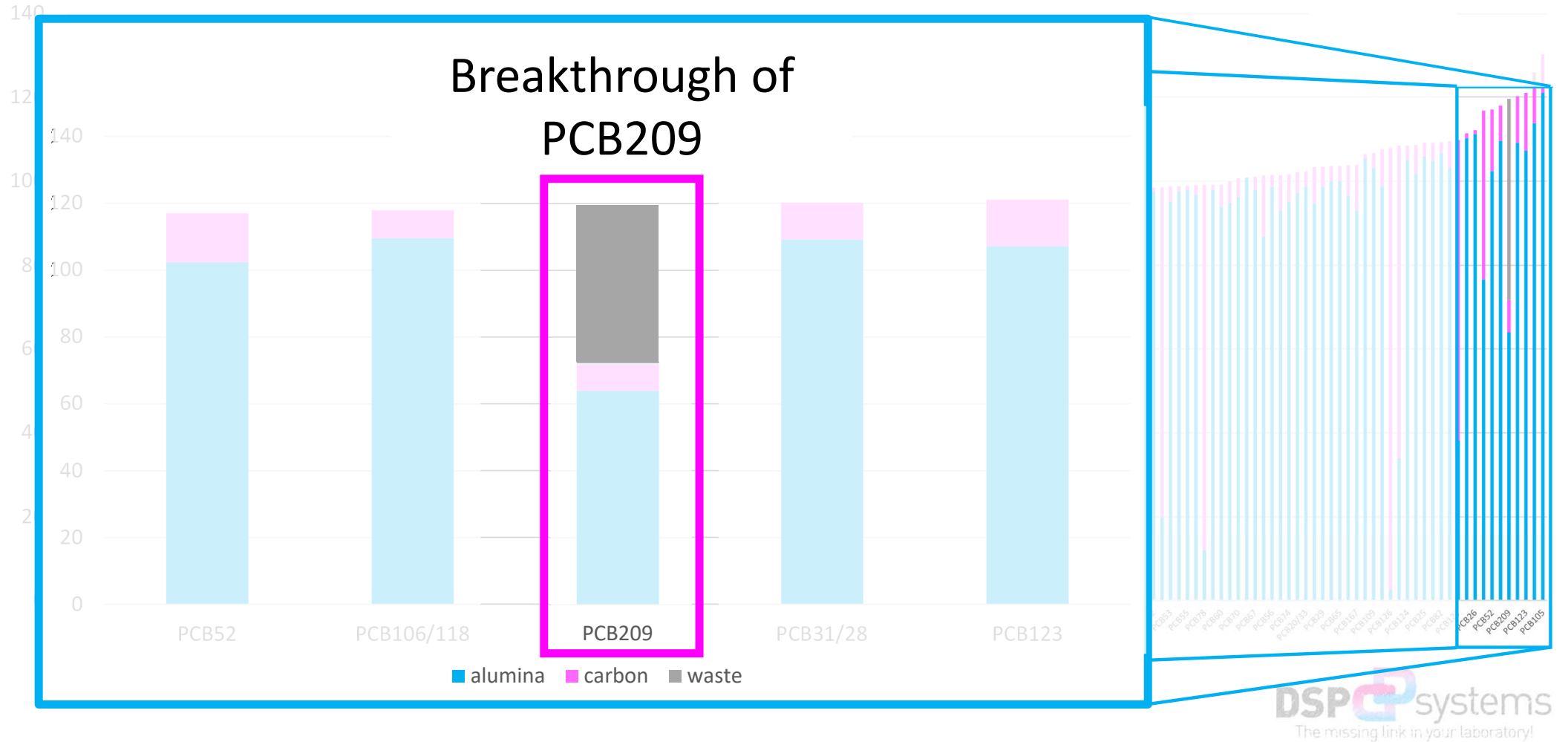
Recovery of PCBs

Standard method

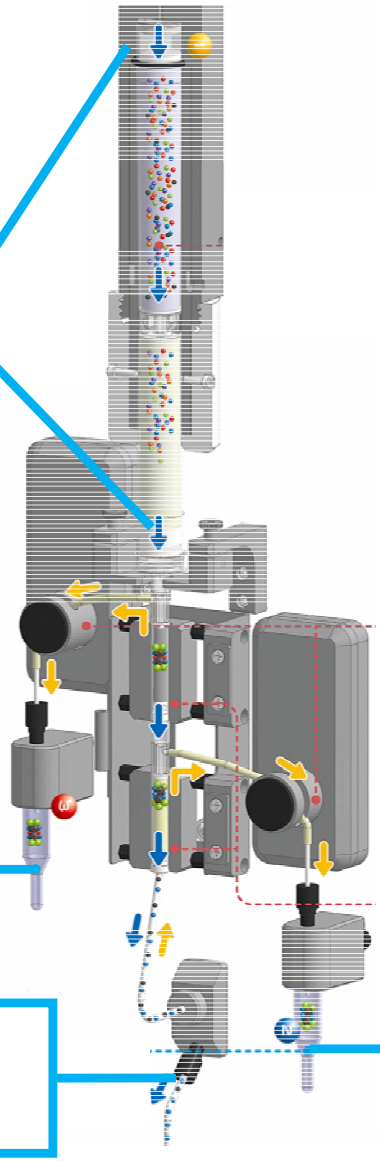


Recovery of PCBs

Standard method



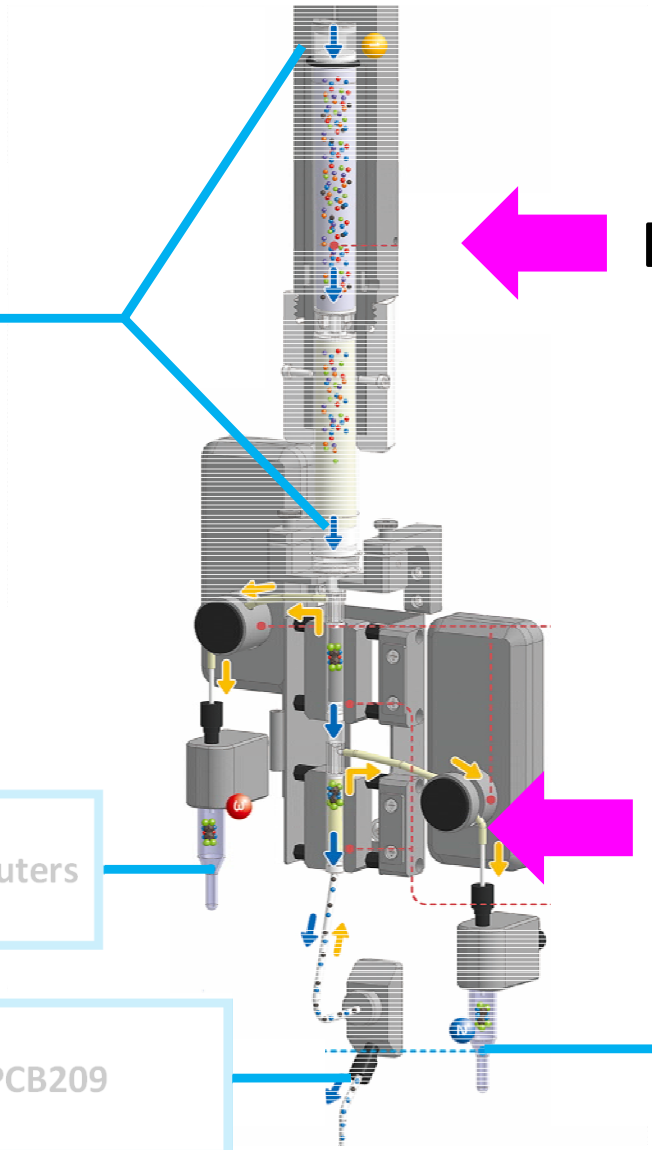
A number of lower chlorinated PCBs



no-PCB fraction and slow eluters

A fraction of PCB209

Majority of PCBs



A number of lower chlorinated PCBs

Enhance elution

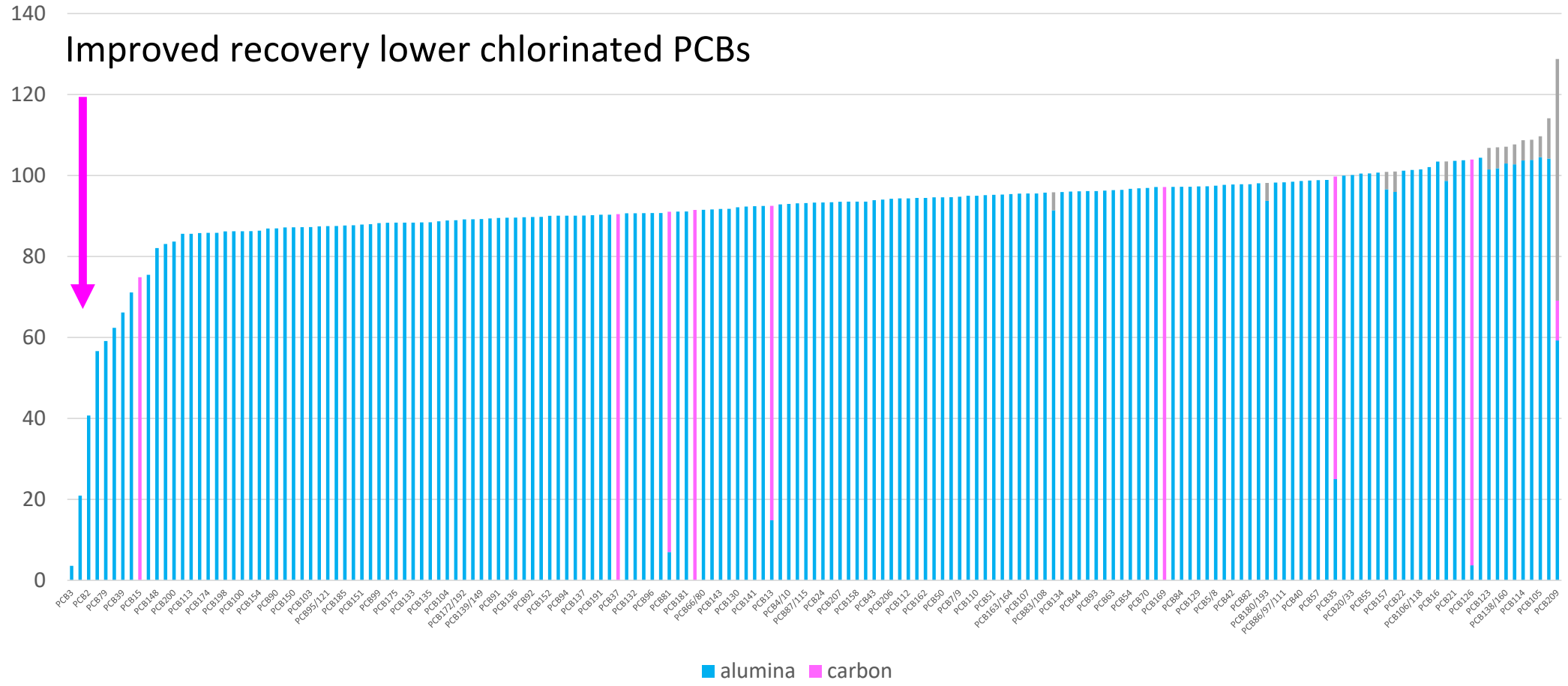
no-PCB fraction and slow eluters

Increase absorption strength

A fraction of PCB209

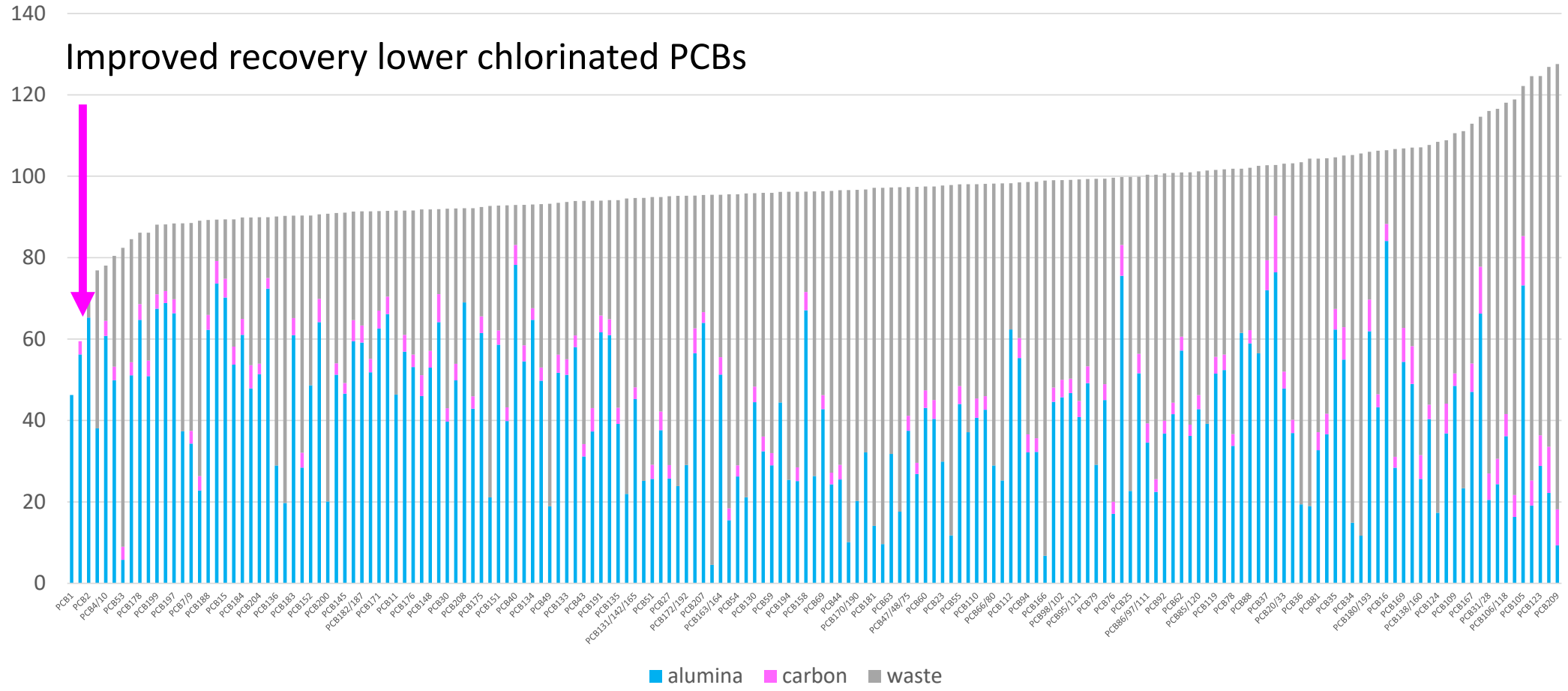
Majority of PCBs

Ethyl acetate modifier



1ml ethyl acetate added to a 9ml hexane standard solution

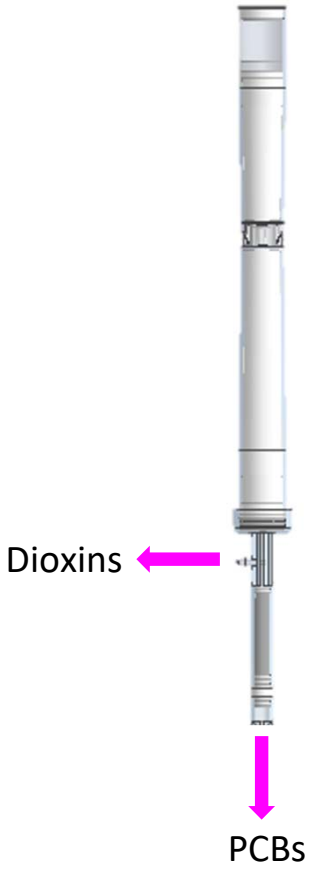
Toluene modifier



1ml modifier added to a 9ml hexane standard solution

WHO Dioxin & 209PCB – In development

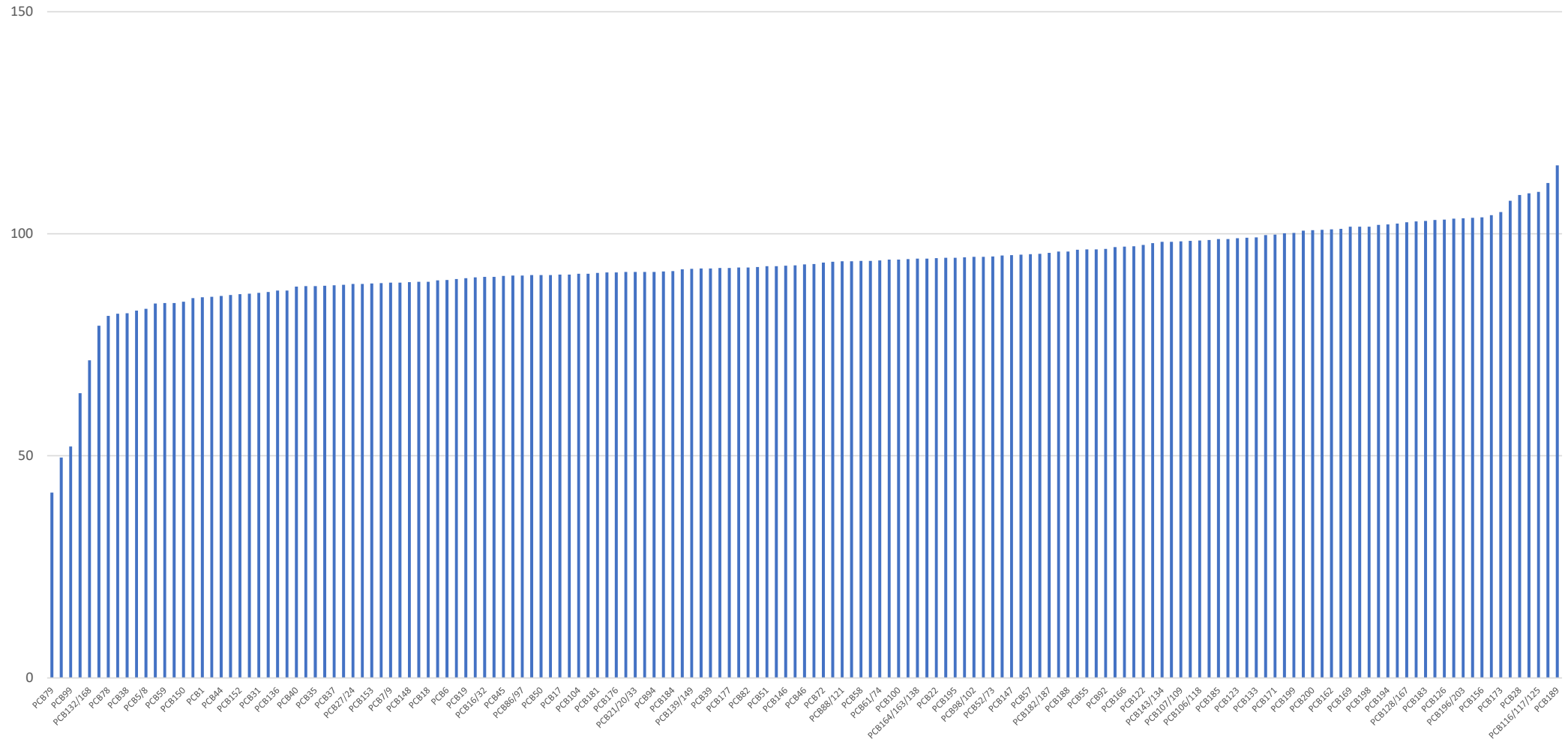
Type Size[mm]	
Sample	
Purification	
Concentration	
Elution	To waste
	DXN fraction
	PCB fraction
Run time	



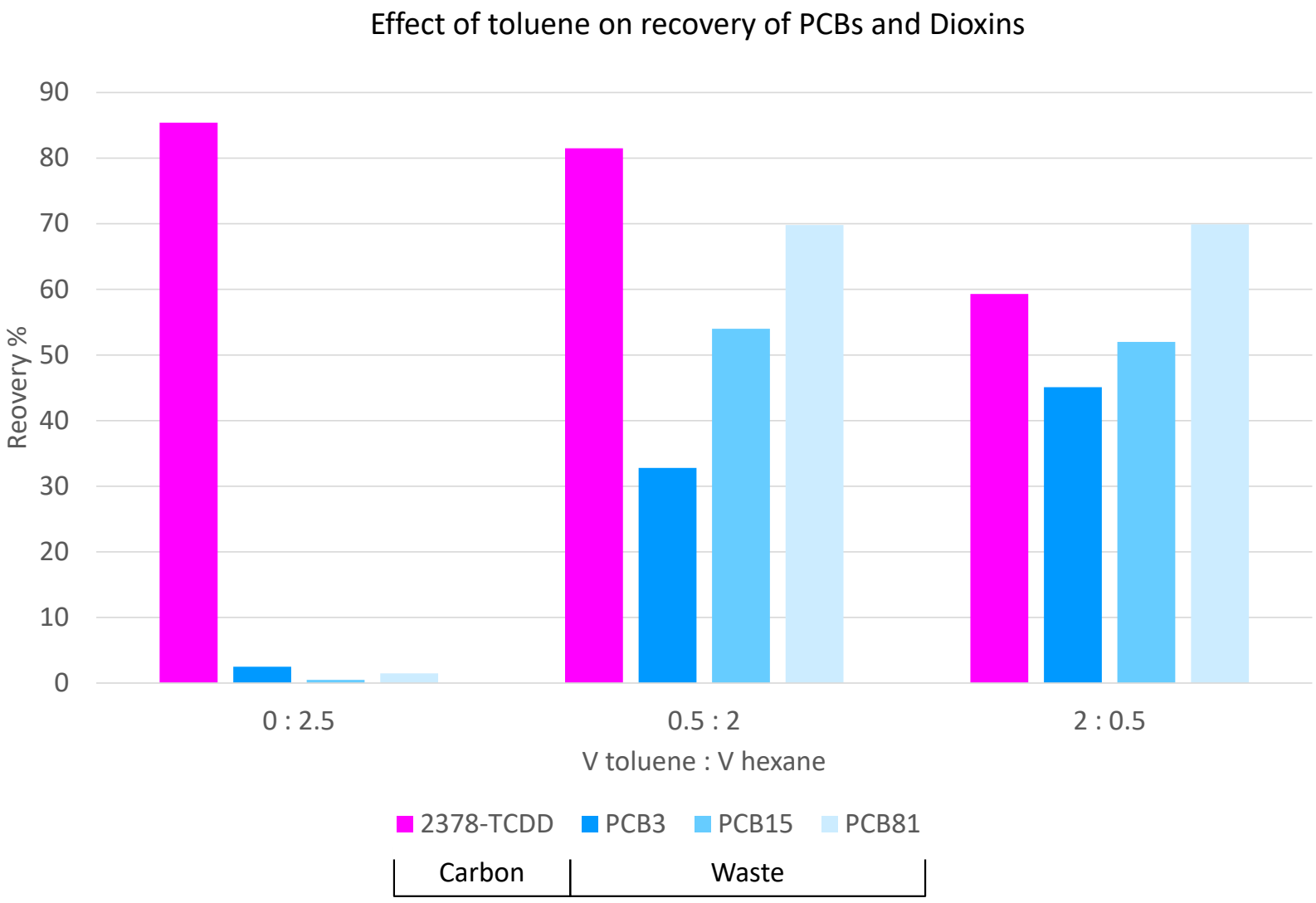
DXNmini ***18 φ***
Environmental 5 g d.m.
$AgNO_3$-Silica gel
H_2SO_4-Silica gel
Carbon
-
85mL Hexane
1.5 mL Toluene
-

+1ml Toluene

Recovery of all 209PCBs

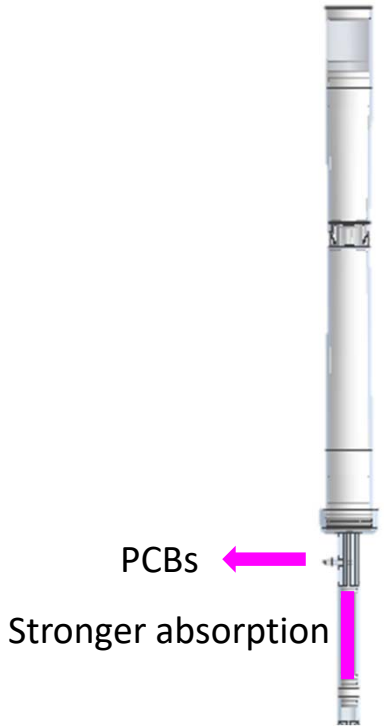


Sample bottle rinsed with 1ml toluene and this 1ml toluene added to a 18Ø column


Effect of toluene on recovery of PCBs and Dioxins
90
80
70
60
50
40
30
20
10
0
Reovery %
0 : 2.5
0.5 : 2
2 : 0.5
V toluene : V hexane
2378-TCDD
PCB3
PCB15
PCB81
Carbon
Waste

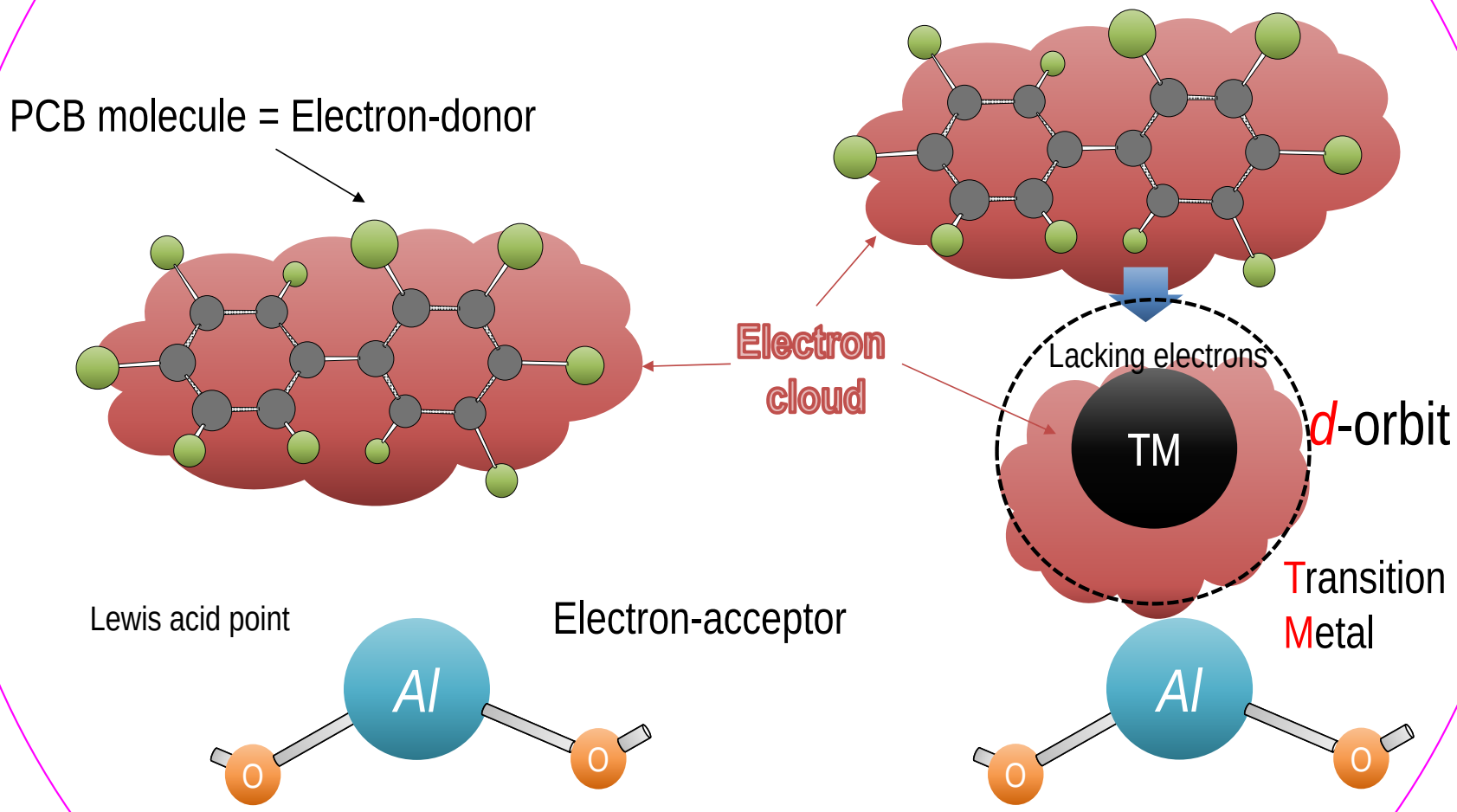
209PCB only – In development

Type Size[mm]	
Sample	
Purification	
Concentration	
Elution	To waste
	DXN fraction
	PCB fraction
Run time	



209PCB ***18 φ***
Environmental 5 g d.m.
$AgNO_3$-Silica gel
H_2SO_4-Silica gel
Alumina TM
-
-
N/A
-

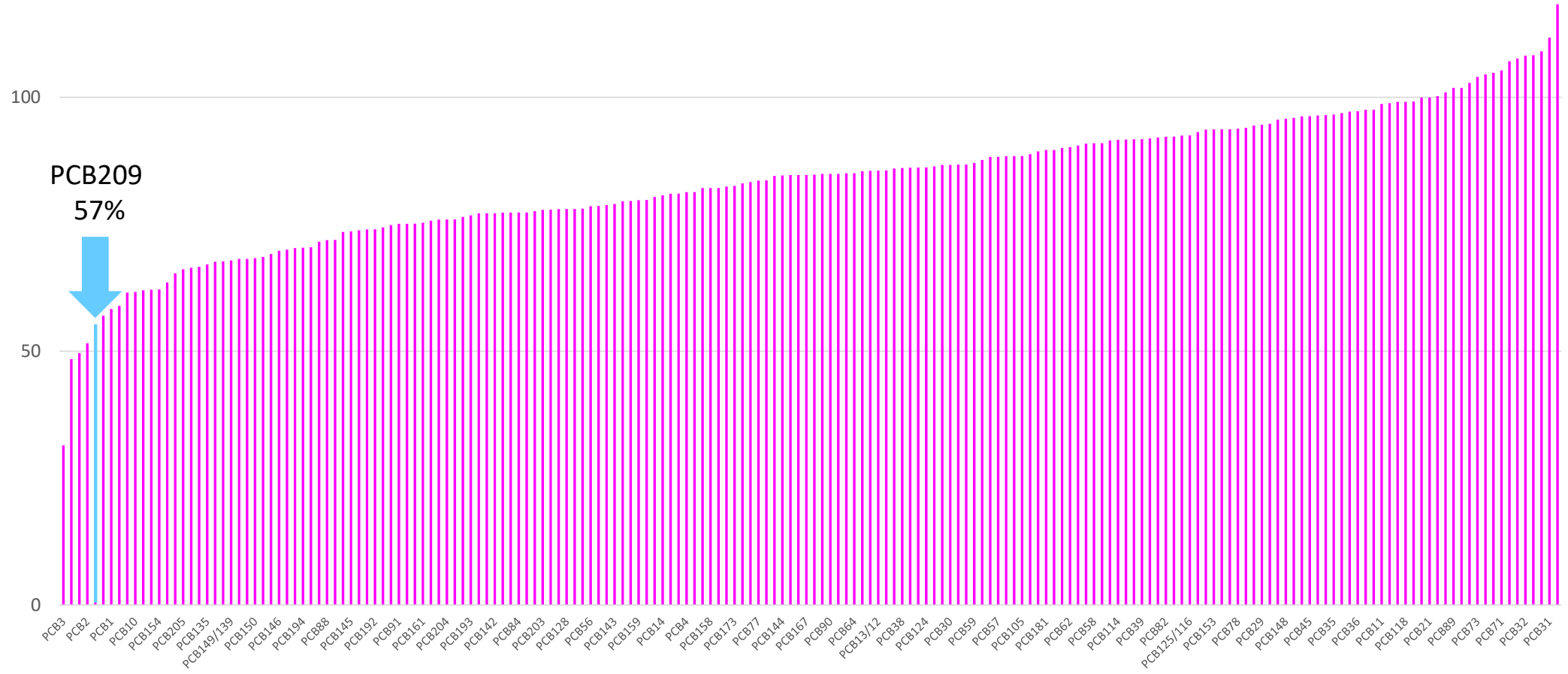
Improving absorption



Fujita et al. Dioxin2019

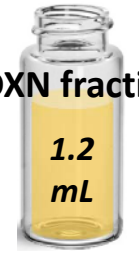
Image of Adsorption Principle of PCB Molecules on Alumina

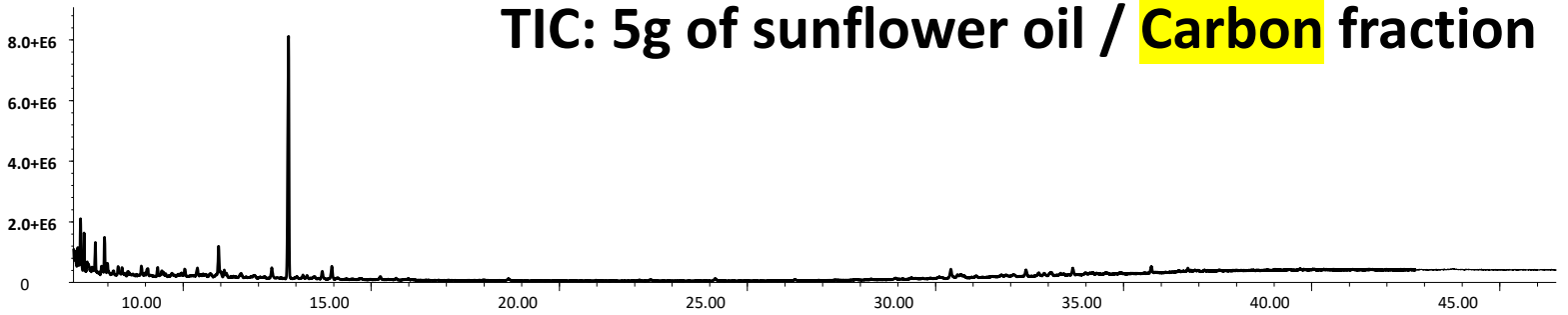
Recovery of PCBs from using a alumina oxide TM concentration column

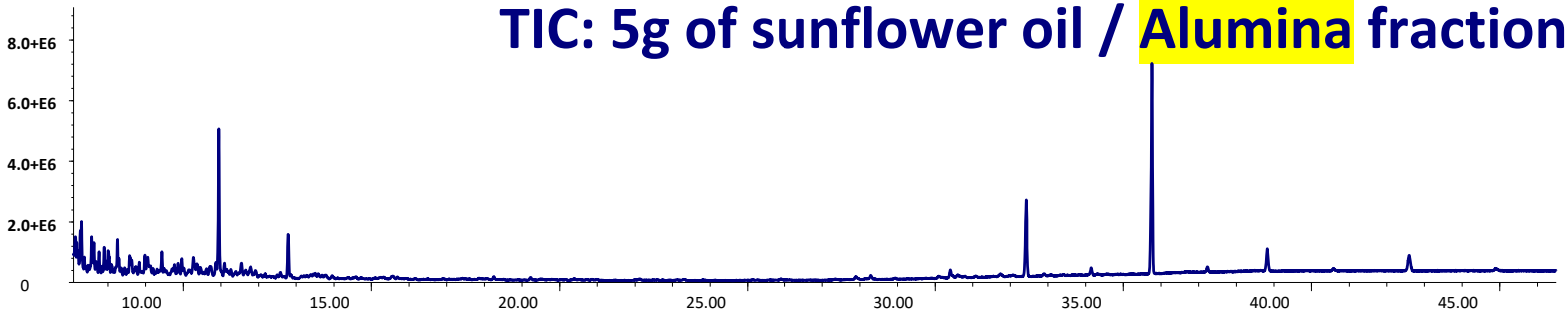


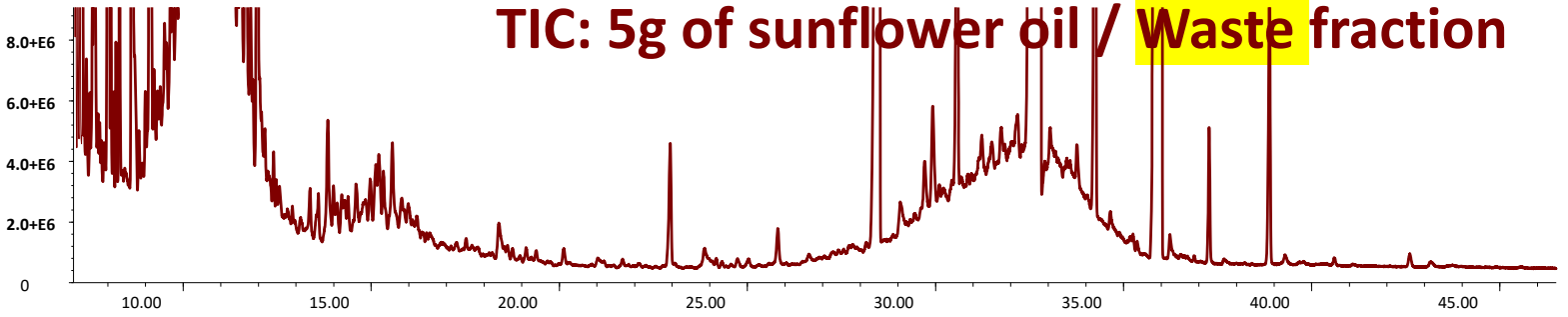
Fujita et al. Dioxin2019

Advantages of fractionation









Fujita et al. Dioxin2019

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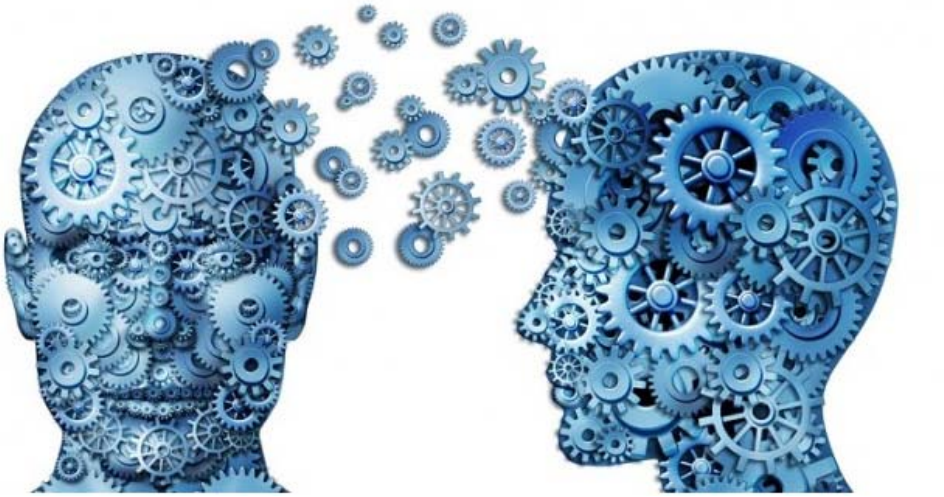
Chemosphere

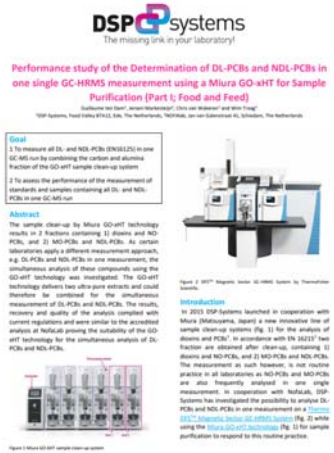
New approach for removing co-extracted lipids before mass spectrometry measurement of persistent of organic pollutants (POPs) in foods

Douglas G. Hayward

Questions?



- Hayward D. G. et al, Chemosphere Volume 256, October 2020, 127023, New approach for removing co-extracted lipids before mass spectrometry measurement of persistent organic pollutants (POPs) in foods
- Fujita et al. Organohalogen Compounds (2016), Simulteneous analysis of dioxins, PCBs, and PBDEs with a fully automated sample preparation system (II: validation)
- Marchand P. et al., Organohalogen Compounds Vol. 76, 546-549 (2014), A new highly innovative automatic purification system evaluated for Dioxins and PCBs.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Performance study of the Determination of DL-PCBs and NDL-PCBs in one single GC-HRMS measurement using a Miura GO-xHT for Sample Purification (Part I; Food and Feed)

- 2019:11 - Comparison of available MS Systems for the Analysis of Dioxins and PCBs
- 2019:03 - Single injection analysis of PCBs and Dioxins in food and feed using GO-HT sample purification
- 2018:12 - Feasibility study on Polychlorinated Naphthalenes (PCNs) analysis using GO-HT sample purification
- 2017: Simultaneous Analysis for Dioxins, PCBS and PBDES Part III
- 2016: Simultaneous Analysis of Dioxins, PCBs and PBDEs Part II
- 2015: Simultaneous Analysis of Dioxins, PCBs and PBDEs Part I

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