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Development of a fast and simple method for extraction of Polychlorobiphenyls ethers (PCBs) in meat and GC/MS analysis.

Emma Sanna^{1,2}, Alyssa Bouville^{1,2}, Laurent Debrauwer^{1,2}, Erwan Engel³, Frédéric Mercier³, Jérémy Ratel³ and Sylvie Chevolleau^{1,2*}

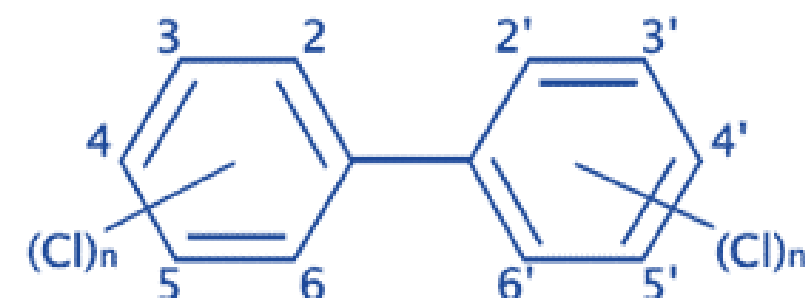
¹ Toxalim (Research Center in Food Toxicology), INRAE, ENVT, INP-Purpan, Université de Toulouse, F-31027 Toulouse, France

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Introduction

Polychlorinated biphenyls (PCBs) are synthetic molecules widely used in various industrial sectors in France during the 20th century. These persistent organic pollutants (POPs), are now present in the human diet and bioaccumulate in fatty tissues of living organisms. Their toxicity for humans and environment is well known.



Current sample preparation methods for PCBs analysis in meat (e.g. Soxhlet, microwave-assisted extraction, ASE) are often long and tedious.

The objective of this work, carried out in the frame of the SENTINEL ANR program (ANR-19-CE21-0011), was to develop a simple and fast method using QuEChERS to overcome traditional PCBs extraction methods from meat, allowing to improve the GC/MS analysis throughput and to reduce solvent consumption.

Sentinel project strategy

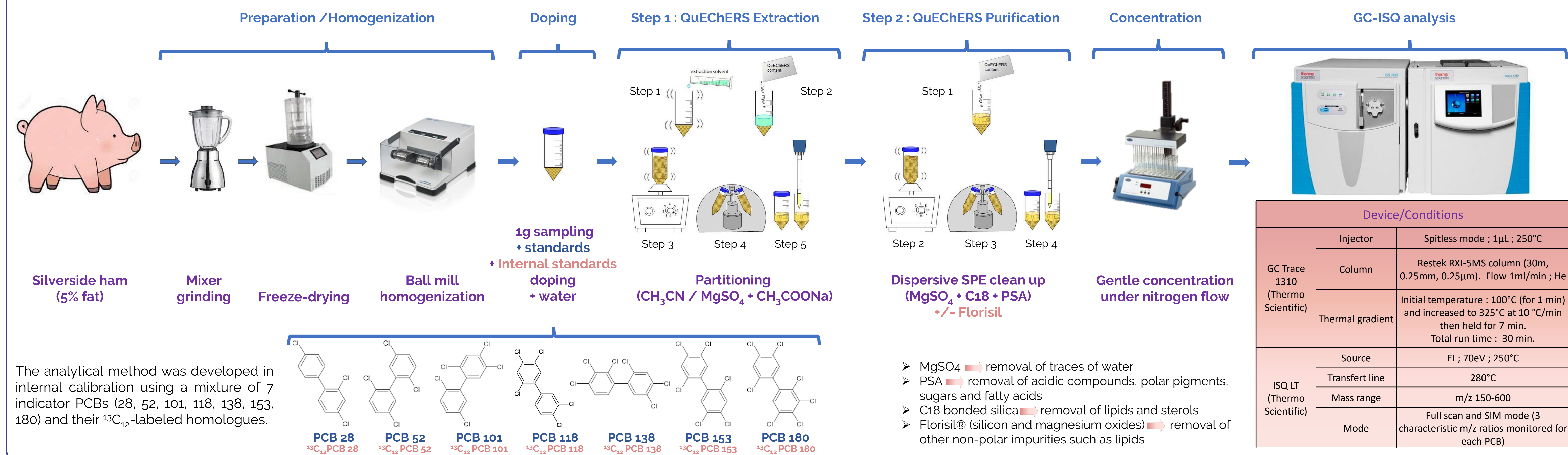
Objective: To strengthen the current food chemical safety monitoring system by developing high-throughput, sensitive and cost-effective screening tools for PCBs surveillance in meat

- 1/ Increase the efficiency of regulatory inspections by health authorities
- 2/ Facilitate industrial self-control tools
- 3/ Monitor chemical contaminants at sub-regulatory levels

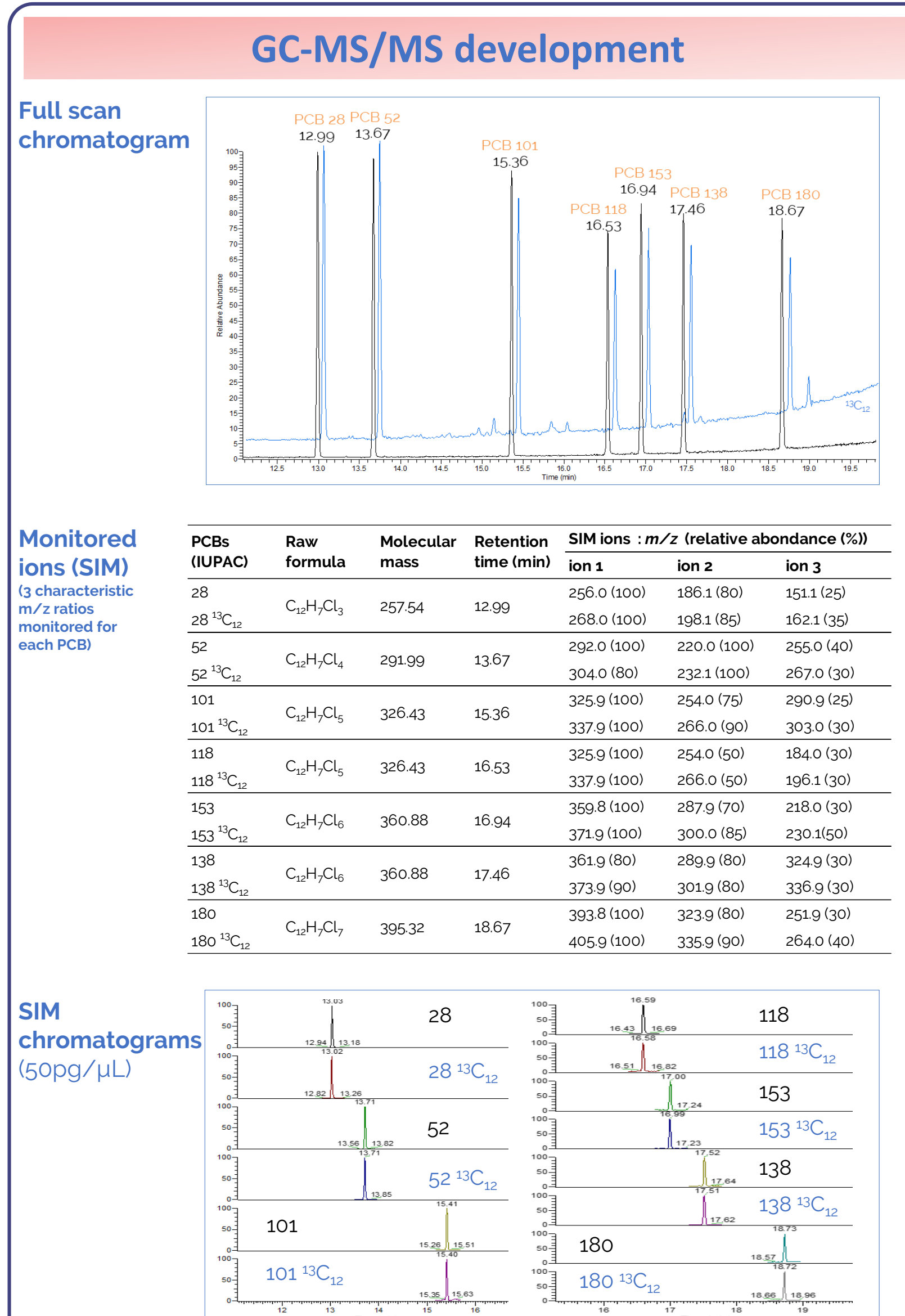


Better understanding of contaminations, better prevention of chemical risks, better understanding of the risks associated with chronic low-dose exposures

Materials and Methods



Results and Discussion



Parameters tested	7 PCB method performance																																								
Selectivity (n=3)	In progress. Numerous interferences from the matrix and QuEChERS salts																																								
Carry-over (n=3)	No carry-over in blanks (solvent) at the highest calibration point																																								
Sensitivity LOD (S/N>3), LOQ (S/N>10) (n=5) (pg/µL)	<table border="1"> <thead> <tr> <th>PCB</th> <th>LOD solvent</th> <th>LOQ solvent</th> <th>LOD matrix</th> <th>LOQ matrix</th> </tr> </thead> <tbody> <tr> <td>PCB 28</td> <td>0.1</td> <td>0.1</td> <td>0.25</td> <td>0.25</td> </tr> <tr> <td>PCB 52</td> <td>0.1</td> <td>0.1</td> <td>1-5</td> <td>1-5</td> </tr> <tr> <td>PCB 101</td> <td>0.1</td> <td>0.1</td> <td>0.5</td> <td>1</td> </tr> <tr> <td>PCB 118</td> <td>0.25</td> <td>0.25</td> <td>0.5</td> <td>1</td> </tr> <tr> <td>PCB 138</td> <td>0.1</td> <td>0.25</td> <td>0.5</td> <td>1</td> </tr> <tr> <td>PCB 153</td> <td>0.25</td> <td>0.25</td> <td>1</td> <td>10.5-1</td> </tr> <tr> <td>PCB 180</td> <td>0.25</td> <td>0.25</td> <td>1</td> <td>10.5-1</td> </tr> </tbody> </table>	PCB	LOD solvent	LOQ solvent	LOD matrix	LOQ matrix	PCB 28	0.1	0.1	0.25	0.25	PCB 52	0.1	0.1	1-5	1-5	PCB 101	0.1	0.1	0.5	1	PCB 118	0.25	0.25	0.5	1	PCB 138	0.1	0.25	0.5	1	PCB 153	0.25	0.25	1	10.5-1	PCB 180	0.25	0.25	1	10.5-1
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Calibration range 7 points: 0.5, 1, 5, 10, 50, 100, 500 pg/µL and IS 50 pg/µL (n=3)	<table border="1"> <thead> <tr> <th>Parameter</th> <th>Value</th> </tr> </thead> <tbody> <tr> <td>Model solvent</td> <td>Linear or Quadratic according to PCB</td> </tr> <tr> <td>Model matrix</td> <td>In progress</td> </tr> <tr> <td>Weighting solvent</td> <td>1/X</td> </tr> <tr> <td>Weighting matrix</td> <td>In progress</td> </tr> <tr> <td>R² solvent</td> <td>> 0.9996 for all PCB</td> </tr> <tr> <td>R² matrix</td> <td>In progress</td> </tr> </tbody> </table>	Parameter	Value	Model solvent	Linear or Quadratic according to PCB	Model matrix	In progress	Weighting solvent	1/X	Weighting matrix	In progress	R ² solvent	> 0.9996 for all PCB	R ² matrix	In progress																										
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Matrix effect Extract blank matrix spiked with 3 QC levels: 25, 50, 100 pg/µL (n=3)	Strong effect: ME>+50%																																								
Recovery rate Blank matrix spiked with 3 QC levels: 25, 50, 100 pg/µL (n=3)	[41-79 %] Improved with Florisil: [67-86%]																																								

> Blank matrix: Silverside ham



Conclusion

- Main QuEChERS parameters (solvents, salt concentrations, contact time, temperature...) have been tested for the extraction step. Several conditions have also been tested for the purification part using dSPE and dSPE + Florisil. Alternative tests are currently conducted using LLE (Hexane, AcOEt) for extraction or using SPE for purification, in order to reduce interfering materials and improve the performance of the analytical method.
- Once validated this method will be transposed to high resolution mass spectrometric detection enabling to strengthen the current food chemical safety monitoring system with the aim to use this method for PCBs determination at sub-regulatory levels by further gaining both detection selectivity and sensitivity.

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