



# Occurrence of halogenated flame retardants in Belgian foodstuff

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## INTRODUCTION and OBJECTIVES

- ✓ Lack of data on the presence of halogenated flame retardants (HFRs) in food → EFSA indicated that it is not possible to perform an accurate risk assessment on the occurrence of HFRs in food and consequently on the exposure to HFRs via the diet.
- ✓ This project follows up the European Commission Recommendation 2014/118/EU<sup>1</sup> on the monitoring of HFRs in food.
- ✓ Due to the diverse nature of the considered HFRs, two analytical methods, based on gas chromatography in combination with electron capture negative ionization mass spectrometry (GC-ECNI/MS) and ultra-high performance liquid chromatography-tandem mass spectrometry (UHPLC-MS/MS), were developed and in-house validated<sup>2,3</sup>.
- ✓ In 2015 and 2016, 183 composite food samples, belonging to 15 different food categories, were analyzed in the frame of the project.

Target analytes		Food categories
GC-ECNI/MS	PBDEs	Liquid milk composite LC; n=13
	HBB	Dessert/sweet composite DC; n=3
	TBB	Cheese composite CHC; n=22
	BTBP	Baby-food composite BC; n=18
	TBPE	Oil/fat composite OC-FAT; n=9
	TBPH	Fish composite FC; n=51
UHPLC-MS/MS	DPs	Crustacean composite CC; n=7
	TBA	Mussel composite MC; n=3
	HBCDDs	Egg composite EGC; n=4
	TBBPA	Grain composite GRAC; n=7
	TBBPS	Potato composite POC; n=4
	BrPhs	Other food composite OTC; n=5
		Vegetable composite VEC; n=2
		Meat composite MEC; n=35

## MATERIALS and METHODS

### GC amenable compounds

- ✓ Solid-liquid extraction with 5 mL ACN:toluene (9:1, v/v)
- ✓ Clean-up performed on Florisil® and acidified silica (AS, 1 g, 5% H<sub>2</sub>SO<sub>4</sub> w/w)
- ✓ Target analysis performed with a GC-ECNI/MS, operated in SIM
- ✓ LOQs: 50 pg/g ww for TBA, 5 pg/g ww for PBDEs, 100 pg/g ww for BDE-209, 20 pg/g ww for HBB and DPs, 10 pg/g ww for BTBP, 200 pg/g ww for TBB and TBPH

### LC amenable compounds

- ✓ TBBPS: extraction with ACN:formic acid (9:1, v/v), clean-up with EMR-Lipid dSPE kit
- ✓ Other target HFRs: extraction with Hex:DCM (1:1, v/v), clean-up with AS (8 g, 44% H<sub>2</sub>SO<sub>4</sub> w/w)
- ✓ Target analysis performed with an ACQUITY UPLC system coupled to a Xevo-TQ-S mass spectrometer operated in ESI(-) mode
- ✓ LOQs from 4 pg/g ww (HBCDDs) to 8 ng/g ww (BrPhs)

## RESULTS and DISCUSSION

- ✓ Mean levels of PBDEs, TBA, TBPH, DPs, BrPhs, TBBPA, and HBCDDs (pg/g ww on lower-LB bound basis) in the analyzed food groups is represented in Fig.1; HBB, TBB, BTBP, TBBPS and 26-DBP were not detected in any analyzed food groups, and thus were excluded from the discussion.

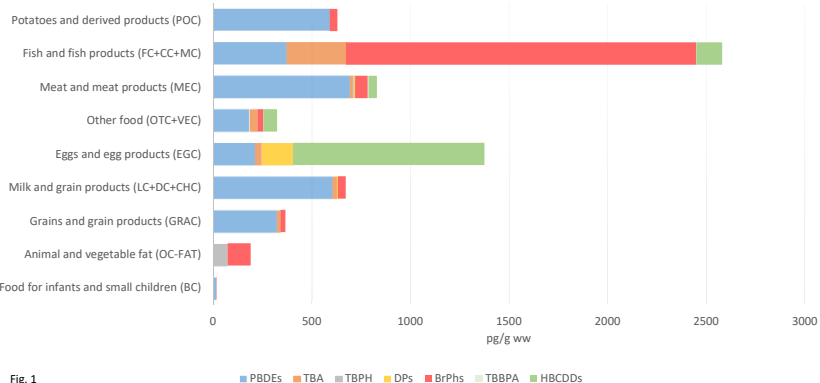


Fig. 1

- ✓ BDE-47 was the most representative BDE-congener of the mean BDE contamination in the composite fish/seafood samples, while cheese, meat, and potatoes were mainly affected by BDE-209 (95 - 100 %).
- ✓ Of the HBCDDs, a-HBCD was the most frequently detected and was most predominant in fish/seafood
- ✓ In all foodstuffs, the most frequently occurring BrPhs was 246-TBP, followed by 4-BP, and 24-DBP.
- ✓ TBA was mainly present in FC and MC categories, as expected due to its primary natural origin from the marine environment<sup>4</sup>.

- ✓ The highest number of detects was observed in the groups potatoes and fish/seafood, with 100 % and 93 % of the samples where at least one compound was quantified above the LOQ (Fig. 2b).

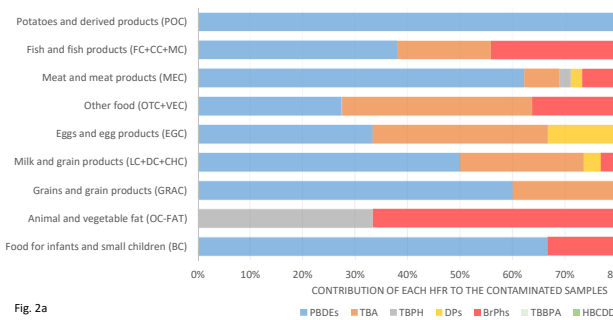


Fig. 2a

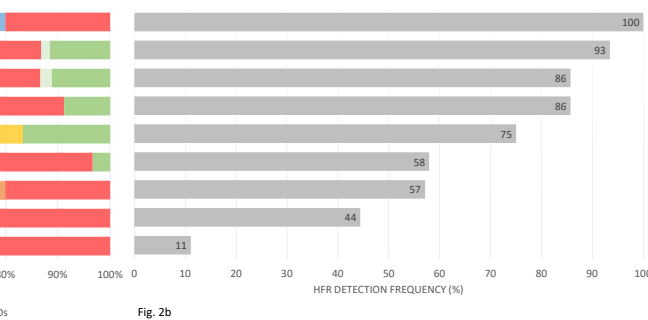


Fig. 2b

## CONCLUSION

- ✓ The present study provided occurrence data on HFRs in Belgian food, contributing to a more robust risk assessment on dietary exposure.
- ✓ Our findings confirm that certain Belgian food contain detectable concentrations of such BFRs as PBDEs, banned in the EU for several years, and HBCDDs, on the use of which restrictions have been applied.
- ✓ General low contamination of food with HFRs, but no declining trend in the PBDE and HBCDD levels compared to literature data could be clearly observed.
- ✓ It is reasonable to continue surveillance on the occurrence of the HFRs in food.
- ✓ The measured levels will be used to estimate the dietary exposure to HFRs in the Belgian population.

## ACKNOWLEDGEMENTS

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## REFERENCES

1. Commission Recommendation 2014/118/EU Off. J. Eur. Union L65, 39–40.
2. Poma G, Malarvannan G, Voorspoels S, Symons N, Malysheva SV, Van Loco J, Covaci A (2016). Food Control, 65, 168-176.
3. Malysheva SV, Gosciny S, Malarvannan G, Poma G, Andjelkovic M, Voorspoels S, Covaci A, Van Loco J. Quantitative UPLC-MS/MS method for selected brominated flame retardants in food. Submitted.
4. Bidleman, T.F. Agosta, K., Andersson, A., et al. Marine Pollution Bulletin, 2016, 112: 58-64.