

Extraction of PCDD/Fs and PCBs in fish using SpeedExtractor E-914

A consensus material and two additional fish samples were extracted with the SpeedExtractor E-914. After clean-up of residual fat PCDDs, PCDFs, dl-PCBs and ndl-PCBs were quantified with GC-HRMS. The results from the SpeedExtractor correspond to the consensus results. In addition, good comparability of the SpeedExtractor results to the results found by Soxhlet extraction was shown. Furthermore, good accuracy of triplicate extraction with the SpeedExtractor was demonstrated.

Introduction

Dioxins are persistent environmental pollutants. They are found throughout the world in the environment and they accumulate in the food chain, mainly in the fatty tissue of animals.

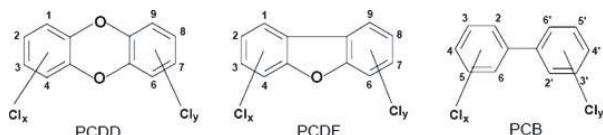


Figure 1: General formula of PCDD, PCDF and PCB

This application note describes the use of the SpeedExtractor for the extraction and determination of PCDDs, PCDFs and PCBs in fish tissue (trout and eel).

Experimental

Two fish samples (trout and eel) and a consensus material (trout) were extracted with the SpeedExtractor E-914. The results were compared to results from Soxhlet extraction and consensus results, where applicable.

About 10 g of freeze dried eel and 20 g of freeze dried trout were mixed with diatomaceous earth and extracted with the SpeedExtractor according to the parameters in table 1. A single extraction of the consensus material and a triplicate extraction of eel and trout samples were conducted afterwards. The raw extracts were concentrated and dried. The resulting fat residue was weighed to calculate the fat content of the sample. Quantification with GC-HRMS of PCDD/F and PCB was done after clean-up of the fat. Extraction, quantification and recovery standards were applied.

Table 1: Extraction parameters E-914

Temperature	100 °C
Pressure	100 bar
Solvent	Dichloromethane/ n-hexane 50:50
Cells	80 mL
Vials	240 mL
Cycles	3
Heat-up	5/1/1 min
Hold	10 min
Discharge	4 min
Flush with solvent	2 min
Flush with gas	10 min
Total extraction time	1h 25 min

Results

In figure 1 the TEQ-results are shown for three sample materials. The results represent a short summary of the good results found in general.

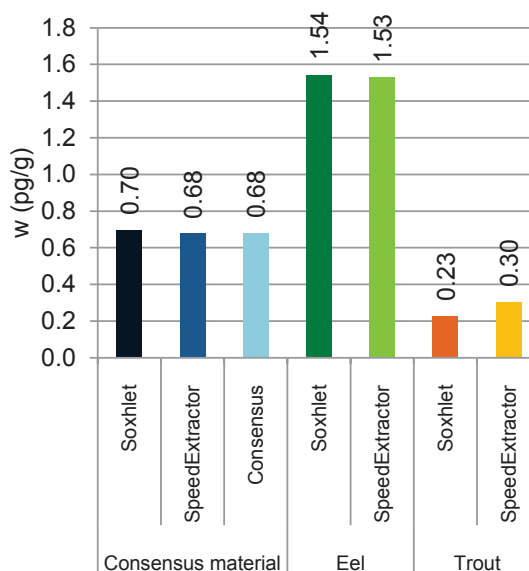


Figure 1: Median of TEQ (WHO 2005) for PCDD/Fs in consensus material. Mean of TEQ in eel and trout. Consensus material: Soxhlet n=1, SpeedExtractor n=1, consensus n=79. Eel and trout: Soxhlet n=1, SpeedExtractor n=3.

Conclusion

An excellent comparability of SpeedExtractor results with the consensus results and the Soxhlet extraction was demonstrated. The extraction with the SpeedExtractor is an excellent, reliable and fast alternative to the extraction according to Soxhlet.

Acknowledgement

We sincerely thank the analytical team of mas – münster analytical solutions gmbh from Münster, Germany.

References

Interlaboratory Comparison on POPs in Food 2010, Norwegian Institute of Public Health
AN 069/2012 – Extraction of fish using the SpeedExtractor E-914 for the determination of PCDD/F and PCB in fish