Automatization and Optimization of the Sample Preparation Workflow for certain *Persistent Organic Pollutants* (POPs)

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Introduction

In this poster, the automated workflow for the sample preparation of certain POPs like PCDD/Fs, PCBs, PCNs, and PBDEs will be shown. This includes the extraction from food/feed and environmental samples with pressurized fluid extraction (PFE), the automated multi-column clean-up with the DEXTech instrument as well as the concentration of the extracts simultaneously with the D-EVA system (see Figure 1).

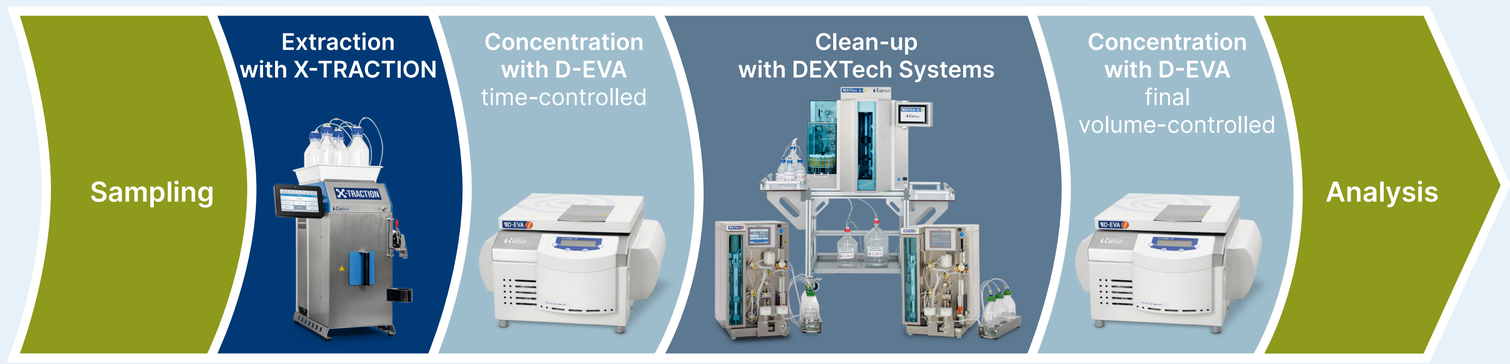


Fig. 1: Dioxin Workflow

Material & Methods

Extraction:

Fig. 2: Extraction cell

For all extractions LCTech used the X-TRACTION System, working with low pressure to max 17 bar. Working in low-pressure range is sufficient for an excellent extraction efficiency with decreased wear-and-tear of instrument parts, higher longevity and a safer handling due to the unique extraction cell-cover-lid locking mechanism (see Fig. 1 and 2). Depending on the matrix, 2 - 15 g sample has been weight in, mixed with sodium polyarcylat. With default methods, toluene for environmental samples, toluene/acetone (70/30 v/v) for feed samples and cyclohexane/toluene (50/50 v/v) at 100 - 150 °C, only 2-3 cycles á 15 min are needed for the extraction.

Clean-up:

The extracts were evaporated to 1 ml and filled up with n-hexane to 10 mL. The clean-up is done with an prepacked acidic silica column (fat capacity 5 g), an alumina column and a carbon column on a DEXTech Heat. There are several default methods available: the Alox Pure Method collects the ndl-PCBs, mono-ortho PCBs and non- ortho-PCBs in the first fraction and the PCDD/Fs and PCNs in the second. If a separation of the non-ortho PCBs is needed, the Alox Plus Method can be used, to collect the ndl-PCBs, mono-ortho PCBs and PBDEs in fraction 1 and the non-ortho-PCBs, PCDD/Fs and PCNs in the 2nd fraction. The volume of fraction 1 = 24 mL and fraction 2 = 10 mL. Depending on the method chosen the clean-up will take between 26 and 74 min.

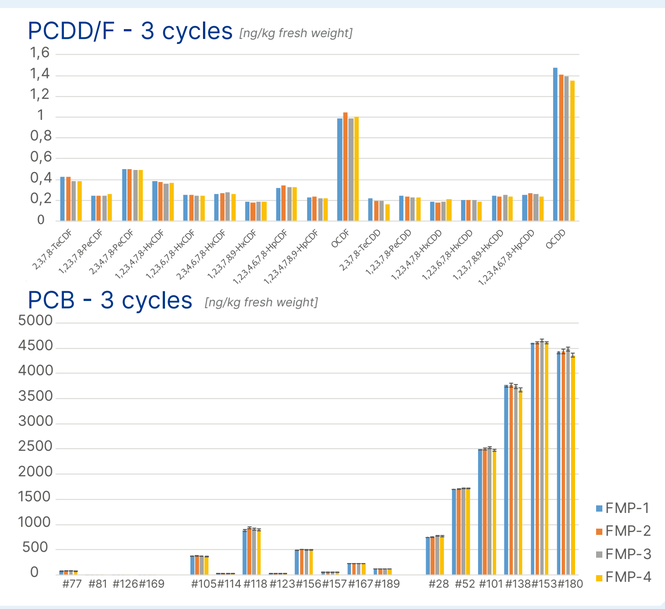
Evaporation:

To overcome the risk to loose Analytes in this critical step of Evaporation before injection, LCTech has implemented a special Vacuum-centrifuge named D-EVA. The D-EVA uses temperature, vacuum and centrifugation to evaporate the different fractions down to small final volumes. Evaporating simultaneously 26 samples of 10 mL of toluene down to 30-100 µL or 11 samples of 24 mL of n-hexane/DCM down to avg. 300 µL without supervision, grants a high sample throughput. The special sensor, developed by LCTech, prevents uncontrolled evaporation to dryness by immediately shutting off the heating and releasing the vacuum when the evaporation is done.

Results: Extraction and Clean-Up of PCDD/Fs and PCBs in Food/Feed

X-TRACTION (3 Cycles) and DEXTech Clean-up								Soxhlet + DEXTech Clean-up		
FMP-1	FMP-2	FMP-3	FMP-4	Mean	SD	RSD	[%] to Soxhlet	Soxhlet n=9	SD	RSD
[ng/kg] Fresh weight					[%]	[%]		[ng/kg] Fresh weight	[%]	[%]
Total (PCDD/F-WHO-TEQ 2005) Upper Bound										
0.843	0.810	0.794	0.762	0.802	0.029	3.6	99	0.807	0.044	5.5
Total PCB-WHO-TEQ 2005 Upper Bound										
0.608	0.617	0.609	0.591	0.606	0.009	1.6	91	0.666	0.024	3.7
Total PCDD/F-PCB-WHO-TEQ 2005 Upper Bound										
1.45	1.43	1.40	1.43	1.43	0.016	1.2	97	1.47	0.040	2.6

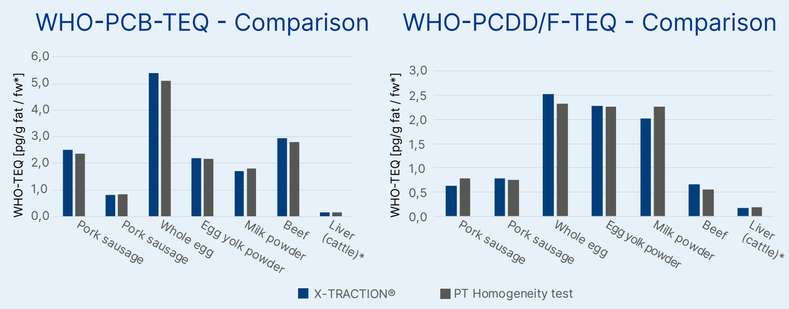
Figure 3 shows a high accuracy and precision for the TEQs of a feed quality control sample (FMP) extracted in direct comparison with the LCTech X-TRACTION (n=4) and the classical Soxhlet method (n=9). The clean-up was performed with a DEXTech Pure.



The detailed results for all congeners of the 4 extracts from the X-TRACTION® can be seen in Figure 4 and 5. The RSD for the PCDD/F of 1,2 %-9,8 % and 0,3 %-2,6 % for the PCBs showing the excellent precision of the method.

	Fat amount	Standard deviation	RSD [%]	Fat Assigned Value [%]	RSD assigned value [%]
Milk powder n = 3	21.5	1.5	7.1	23.1	6.9
Beef n =4	6.3	0.3	5.1	6.5	3.1
Egg volk powder n = 2	52.8	0.8	1.5	55.8	5.4
Egg n = 4	8.1	0.2	2.3	8.6	5.8
Beef liver n = 3	10.1	0.4	4.0	10.8	6.5
Cod liver n = 1	44.1	-	-	46.5	5.2
Halibut n = 1	16	-	-	16.8	4.8

Figure 6 shows the results of the fat extraction with the X-TRACTION® system of different food proficiency test material. As shown, the extracted fat amount corresponds very good with the assigned values from the proficiency test. The same is also true when the assigned PCB-TEQ and PCDD/F-TEQ of the different PT-Food materials were compared with results of the X-TRACTION® and DEXTech cleaned-up samples in Figures 7+8:



Results: Clean-up of PBDEs and PCNs in Quality Control Materials

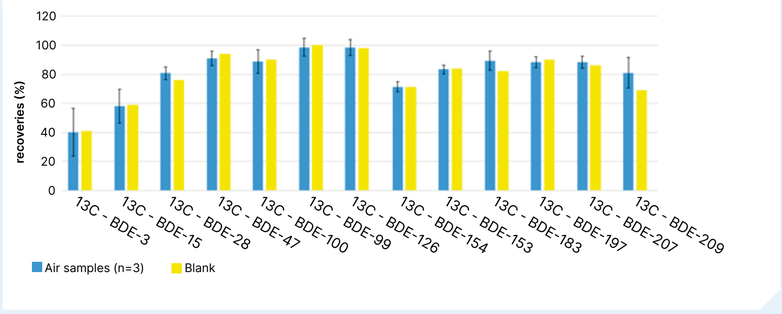


Figure 9 shows the recoveries (%) of different 13C-PBDEs standards of air and solvent blank samples that have been cleaned with a DEXTech Plus system using the Alox Plus Method. All recoveries were in a range of 40 % to 100 %.

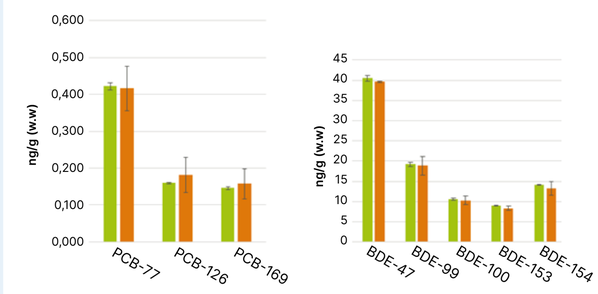


Figure 10 shows some native PCNs results of a certified standard reference material (SRM) "Organics in Whale Blubber". Like before, the results with the DEXTech clean-up were in good agreement with assigned values of the reference material.

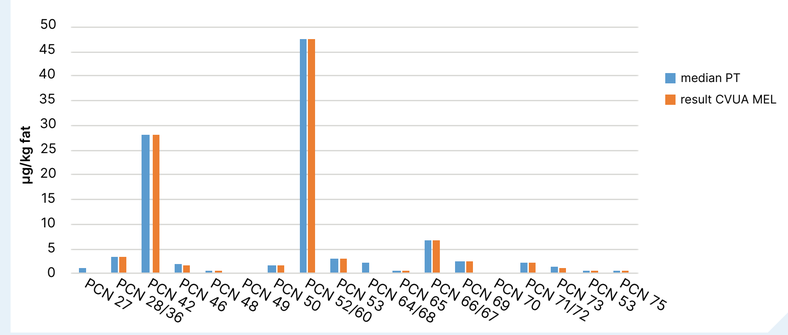


Figure 11 shows the result for the clean-up of a cod liver oil PT-material for PCNs. Again the result from the DEXTech clean-up and analysis are in very good agreement to the median results for the native PCNs of the proficiency test.

Conclusion

Together the presented data show that the LCTech instruments for the sample preparation of different POPs are a quick and reliable method for sample extraction and sample clean-up of a variety of POPs.

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