APPLICATION NOTE AN0047

SOLUTIONS BY LC Tech

Fast and Flexible Automated Sample extraction of PCDD/Fs and PCBs with X-TRACTION for Food

Content

1. Introduction

Persistent organic pollutants (POPs) are highly toxic and persistent substances, which accumulate in the environment and pose risks on health. Since 2001 they are regulated by the Stockholm Convention and monitored accordingly.

By this, the analysis of POPs got increasingly important, leading to a huge growth in sample numbers and the need for standardized yet fast, automated and low-cost methods and instruments at the same time.

Additionally, today's laboratories require instruments, where global regulations and changing methods can be implemented easily into their own processes. The workflow to analyze POPs include extraction, clean-up, evaporation steps and the final analysis.

To complete our existing automated sample preparation portfolio, we have launched a new extraction system called X-Traction by LCTech in 2021.

Pressurized Fluid Extraction (PFE) is a sample extraction method that employs liquid solvents at elevated temperatures and pressures to prepare samples for analysis. While commonly known extraction system are using high pressure (100 - 150 bar) for their process, our newly introduced extraction system works with low pressure (max. 17 bar; LPFE (Low Pressure Fluid Extraction). Working in low-pressure range is sufficient for an excellent extraction efficiency with decreased wear-and-tear of instrument parts, higher longevity and a safe handling. The ease of use is further increased by the unique extraction cell-cover-lid locking mechanism.

This system can be upgraded from 1 to 6 devices, which are able to operate either sequentially or in parallel, with a different protocol on each device.

The system features fast extraction times, easy handling, no cross-contamination and high reproducibility. It can be used for the extraction of Dioxin and PCBs acc. to US EPA method 3545A, extractions test for other POPs like PBDES, PCNs, PFOS etc. are ongoing.

In this application note the extraction for Dioxin and PCB in different food samples will be described.

2. Material and Methods

- X-TRACTION, LCTech GmbH
  - Extraction cell, 75 mL (nominal volume)
  - Glass fiber filter (37 mm diameter)
  - SST Frits
  - Result vials (60 mL; 250 mL)
- DEXTech Pure or DEXTech Heat or DEXTech 16, LCTech GmbH
  - Acidic silica gel column
  - Alumina column
  - Carbon column
- D-EVA, Martin Christ enhanced by LCTech GmbH
  - Centrifuge vials
  - Temperature sensor
- DFS HRMS, Thermo Fisher Scientific
  - SSL-injector, HT8-PCB, 60 m, 0.25 µm film, 0.25 mm ID, Trajan
  - PTV-injector, RTX-Dioxin2, 60 m, 0.25 µm film, 0.25 mm ID, Restek
- Standard Solutions
  - EPA1613-LCS, ISS and CSS, Wellington Laboratories
  - EPA1613-PAR/Stock, Wellington Laboratories
  - PCB-LCS-H, ISS-H and CSS-H, Wellington Laboratories
  - PCB-Stock-A20, Wellington Laboratories
  - EDF-5526, Recovery Standard, CIL
  - EDF-5525-100x Internal Standard, CIL

- Solvents
  - n-Hexane, picograde
  - Cyclohexane, picograde
  - Toluene picograde
  - Dichloromethane, picograde
- Drying agent
  - Sodium polyacrylate (Sigma Aldrich)

3. Filling of Extraction Cell

1. First put in reusable FEP O-ring into both lids:

NOTE: Ensure that the FEP O-ring is stable within the lid and does not fall out.

2. Put on the frit (SST) onto one end of the extraction cell:

NOTE: Keep sealing surfaces clean! In case of dirt or grains, use a clean brush and remove dirt carefully before putting on the frit again.

3. Put on the lid (equipped with FEP O-ring):

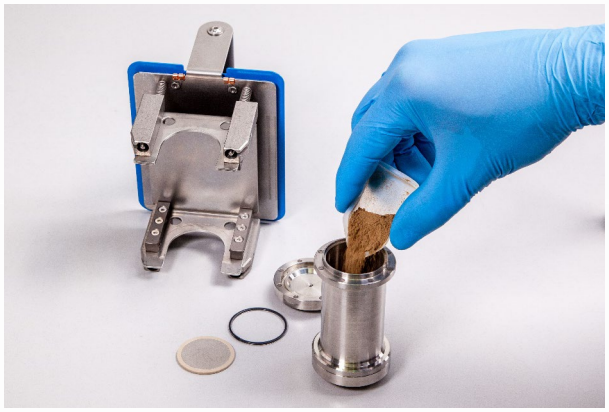
NOTE: Ensure that the lid has a solid magnetic connection to the extraction cell. We recommend turning the lid until you feel the magnetic force between the lid and the extraction cell.

4. Turn around the extraction cell and place glass fibre filter (P/N 19281) on the upper end of the extraction cell.

5. Carefully push down the glass fibre filter to the bottom of the extraction cell. Please use the plunger (P/N 19343), for the exact placement of the filter.

NOTE: Ensure that the glass fibre filter is pushed down equally on each side and has full contact to the inner diameter of the extraction cell.

6. Fill your sample. A funnel or a weighing boat is recommended to ensure accurate filling of the cell.



NOTE: Keep sealing surfaces clean! In case of dirt or grains, use a clean brush and remove dirt carefully.

NOTE: To ensure the function of the X-TRACTION system, it is mandatory to keep a minimum 2 cm air-gap between the upper end of the extraction cell and the upper end of the sample volume within the extraction cell. Please only use free-flowing, dry samples. If sample is wet or fluid, please use sodium polyacrylate until sample is free-flowing and dry. **Never use sodium sulfate as drying agent as it could lead to clogging of the capillaries!**

7. Put on the frit (SST) onto the upper end of the extraction cell.

NOTE: Keep sealing surfaces clean! In case of dirt or grains, use a clean brush and remove dirt carefully.

8. Put on the lid (equipped with FEP O-ring):

NOTE: Ensure that the lid has a solid magnetic connection to the extraction cell. We recommend turning the lid until you feel the magnetic force between the lid and the extraction cell.

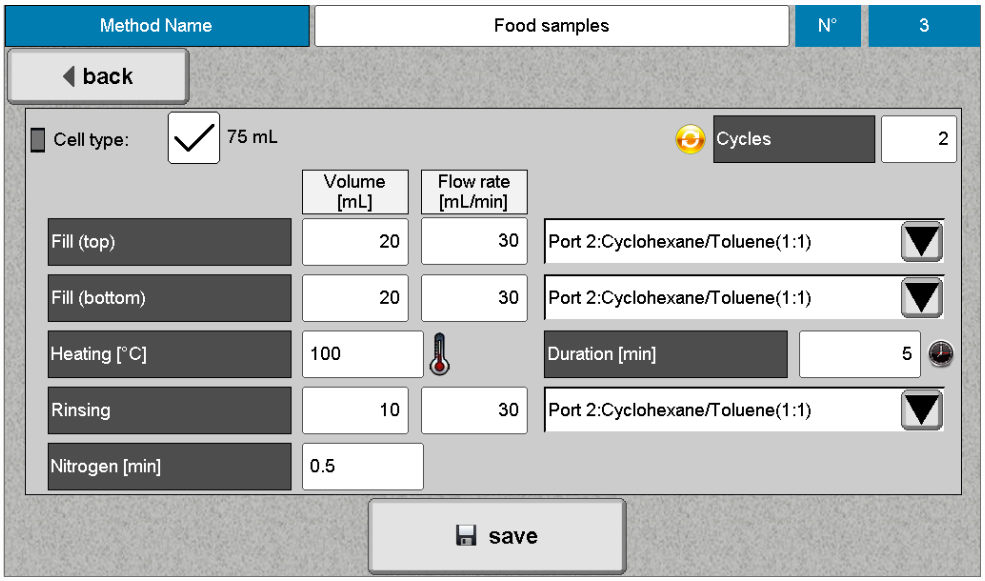
4. Extraction of PCDD/F and PCB in Food samples

4.1 Sample preparation

Homogenised food samples (7 – 15 g) have been mixed with drying agent (sodium polyacrylate) before the extraction. The amount of the drying agent depends on the water content of the samples. The ratio between sample and drying agent was between 1:0.5 and 1:1. It is important to have a dry, free-flowing sample before the extraction. Don't use sodium sulfate as drying agent, as it may lead to clogging of the tubes.

The mixed sample is filled into the extraction cell. For further details, please refer to the manual.

4.2 Extraction conditions



The extraction conditions for the Food samples are shown in the figure above. The parameters shown, are meant as a starting point for further method development, as results may vary depending on matrix composition. Changing the parameters (increasing number of cycles, temperature and holding time) may lead to better extraction efficiency.

The extracted samples were further processed with a DEXTech Plus or Pure instrument, evaporated and analysed for PCDD/Fs and PCB by HR-GCMS.

4.3 Results for pure fat extraction of different food matrices

	Mean % fat	Standard deviation	RSD%	Assigned value % fat	Deviation from 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 yolk 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

Table 1 Fat extraction of different food matrices

Data provided by European Union Reference Laboratory (EURL) for Halogenated POPs in Feed and Food, Freiburg, Germany

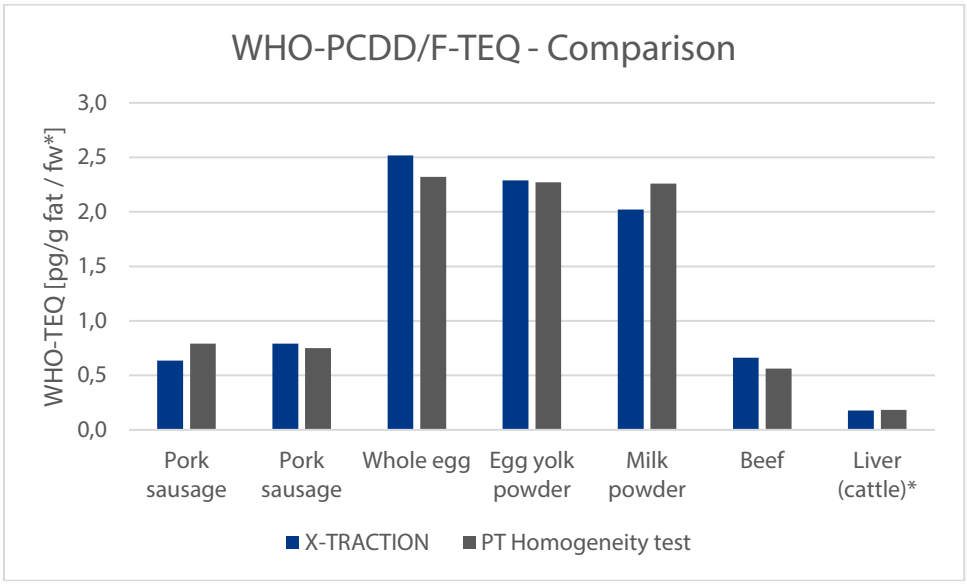
As the results show, the precision for the fat extraction of different food matrices with X-TRACTION is very good, as can be seen by the low RSD values for each matrix.

The same is true for the accuracy, shown by the low deviation of the fat content for each extracted sample in comparison to the assigned fat value. The deviation from the assigned values are well below 10%.

4.4 Results for WHO-PCDD/F-TEQ and WHO-PCB-TEQ

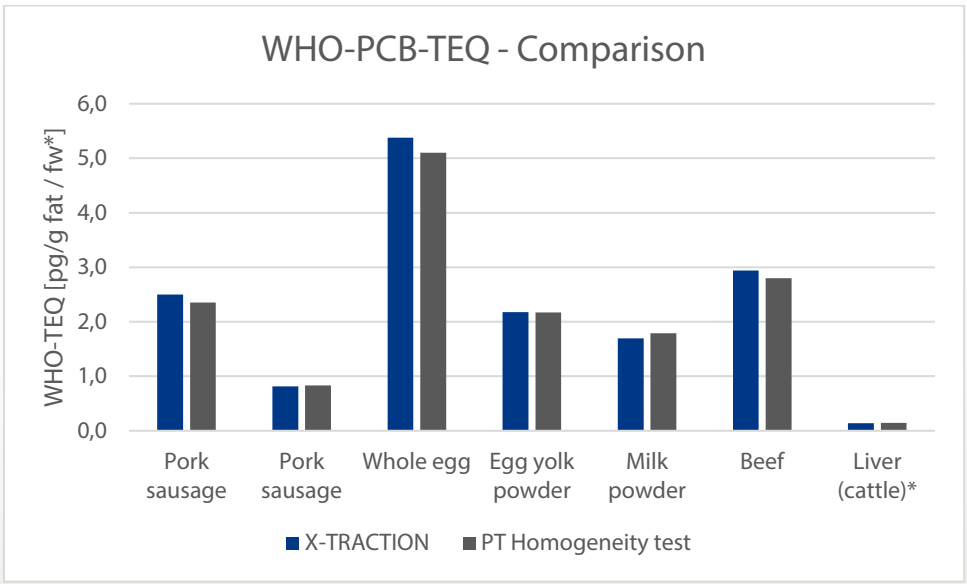
The extracted fats from chapter 2.2.2 were also analyzed for PCDD/F and PCB-TEQ. Results are shown in Figure 1 and
Figure 2 below. Figure 5 shows a WHO-PCDD/F-TEQ comparison of samples extracted with the X-TRACTION system (orange) and the assigned values of a PT homogenity test (green) of different food matrices. As seen in the figure, the results are well comparable to each other. The same is true for Figure 6, which shows the comparison of WHO-PCB-TEQ between the X-TRACTION system and the PT homogenity test.

Figure 1 WHO-PCDD/F-TEQ-Comparison



Data provided by European Union Reference Laboratory (EURL) for Halogenated POPs in Feed and Food, Freiburg, Germany

Figure 2 WHO-PCB-TEQ-Comparison



Data provided by European Union Reference Laboratory (EURL) for Halogenated POPs in Feed and Food, Freiburg, Germany

As the results show, the accuracy for the Dioxin/PCB analyses is very good, the analyzed samples are comparable to the assigned values.

5. Conclusion

In summary, the results shown above proof that the X-TRACTION system is a very flexible and reliable system for a variety of different samples (food, feed, environmental). The results show overall good precision and accuracy for the whole PCB and PCDD/F workflow, including extraction, clean-up, evaporation and analysis.
The handling of the X-TRACTION system is very easy, fast and intuitive. Especially in comparison to classical extraction methods like Soxhlet, the system offers short extraction times and low solvent consumption.

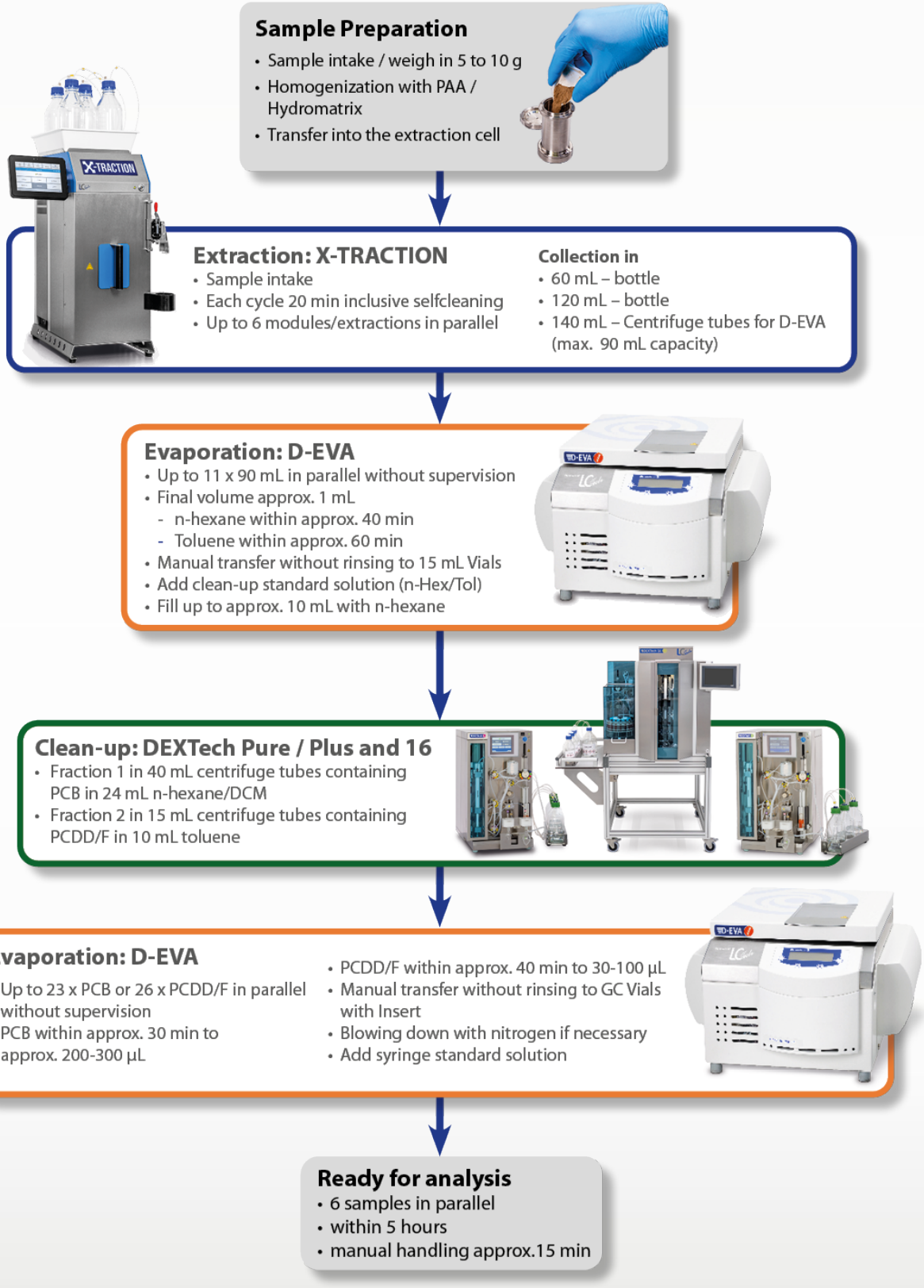
6. Acknowledgements

All tests for „Extraction of PCDD/F and PCB in **Food** samples“ were done at the European Union Reference Laboratory for halogenated POPs in Feed and Food in Freiburg.

We would like to thank the EURL for generously providing the data for this study.

7. Appendix

Dioxin Workflow



8. Ordering information

• X-TRACTION Main system	P/N	20000
• X-TRACTION Add-on-System	P/N	20001
• Extraction cell	P/N	19700
• Rack for extraction cell	P/N	19341
• Flass fiber filter (100 pcs./pck.)	P/N	19281
• Plunger for filter placement	P/N	19343

For a detailed quotation and more information about D-EVA and DEXTech products please contact LCTech.

9. Related Information and References

1. Bernsmann, T., Albrecht M., Fürst, P. (2016); Organohalogen Compounds Vol. 78, 797-799

2. Calaprice C, Calvano CD, Zambonin C, Focant JF (2015); Organohalogen Compounds Vol. 77, 733-735

3. Bernsmann, T., Albrecht M., Fürst, P. (2014); Organohalogen Compounds Vol. 76, 1281-1284

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