

Fast and Flexible Automated Sample Extraction of PCDD/Fs and PCBs with *X-TRACTION*[®] for Environmental Samples





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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 environmental 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
 - Toluene picograde
 - Dichloromethane, picograde
- **Drying Agent**
 - Sodium polyacrylate (Sigma Aldrich)
- **Certified reference materials**
 - BCR 536 (European Commission, Joint Research Centre)
 - BCR 677 (European Commission, Joint Research Centre)



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 Environmental Samples

4.1 Sample Preparation

For the tests, certified reference materials (BCR 536: PCB in harbour sediment; BCR 677: PCDD/F in sewage sludge) were used and extracted according to US EPA 3545A.

Homogenised environmental samples (1 – 2 g; particle size < 1 mm) 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.

If the sample is already dry, with a very small particles size, glass powder can be added to increase the accessibility of solvent to the matrix. The mixed sample is filled into the extraction cell. For further details, please refer to the manual.

4.2 Extraction Conditions

Method Name		Environmental samples		N°	6
◀ back					
Cell type:	<input checked="" type="checkbox"/> 75 mL			Cycles	1
	Volume [mL]	Flow rate [mL/min]			
Fill (top)	20	30	Port 4: Toluene	▼	
Fill (bottom)	20	30	Port 4: Toluene	▼	
Heating [°C]	150		Duration [min]	5	
Rinsing	10	30	Port 4: Toluene	▼	
Nitrogen [min]	0.5				
save					

The extraction conditions for the environmental 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 with a DEXTech Plus instrument, evaporated and analysed for PCDD/Fs and PCB by HR-GCMS.



4.3 Results for Environmental Samples

To show the efficiency of the X-TRACTION® several samples (certified reference material) have been extracted and analyzed for PCDD/F (BCR 677) and PCB (BCR 536).

The results are shown in table 1 and table 2 below and cover the complete workflow (extraction, clean-up, evaporation and measurement).

4.3.1 BCR – 536 – PCB in Harbour Sediment

Table 1 shows the results of different PCB congeners of 4 independent extractions of certified reference material (BCR 536, PCB in harbour sediment). As table 1 shows, the accuracy for the PCB extractions is quite good, as can be seen in the recoveries that range between 85% and 113%.

Table 1 BCR – 536 – PCB in harbour sediment

Native [µg/kg]	Mean n=4 [µg/kg]	RDS [%]	Cert. Value [µg/kg]	Recovery [%]
PCB-#28	47.3	10.0	44.0	108
PCB-#52	42.9	16.5	38.0	113
PCB-#101	42.9	11.5	44.0	98
PCB-#118	23.0	14.9	27.5	84
PCB-#105	3.0	13.2	3.5	87
PCB-#153	53.3	12	50.0	107
PCB-#138	25.7	10.2	27.0	95
PCB-#156	2.7	11.5	3.0	90
PCB-#180	21.4	14.3	22.4	96
PCB-#128	5.3	11.5	5.4	98
PCB-#170	18.1	17.4	17.2	105
PCB-#163	13.0	11.2	13.4	97



Image 1 LCTech Laboratory, Obertaufkirchen



4.3.2 BCR – 677 - PCDD/F in Sewage Sludge

In table 2, the PCDD/F results of 5 independent extractions of certified reference material (BCR 677, PCDD/F in sewage sludge) are presented. Again the results show good recoveries between 74% and 131% indicating a good accuracy for the extracted samples.

Table 2 BCR – 677 - PCDD/F in sewage sludge

Native [pg/g]	Mean n=4 [ng/kg]	RDS [%]	Cert. Value [ng/kg]	Recovery [%]
2,3,7,8-TCDF	41	12.2	45	91
1,2,3,7,8-PeCDF	23	3.7	24.8	93
2,3,4,7,8-PeCDF	16	5.2	16.9	95
1,2,3,4,7,8-HxCDF	13	7.3	14.5	90
1,2,3,6,7,8-HxCDF	5.7	11.1	6.1	93
2,3,4,6,7,8-HxCDF	6.4	6.5	5.6	114
1,2,3,7,8,9-HxCDF	1.1	5.3	0.8	131
1,2,3,4,6,7,8-HpCDF	59.1	10.7	61.6	96
1,2,3,4,7,8,9-HpCDF	4.7	15.9	6.3	74
1,2,3,4,6,7,8,9-OCDF	158	15.2	177	89
2,3,7,8-TCDD	1.6	12.3	1.5	103
1,2,3,7,8-PeCDD	4	14.2	4.1	97
1,2,3,4,7,8-HxCDD	nd	nd	nd	nd
1,2,3,6,7,8-HxCDD	239	13.2	235	102
1,2,3,7,8,9-HxCDD	73.6	12.2	79	93
1,2,3,4,6,7,8-HpCDD	3221	14.9	3500	92
1,2,3,4,6,7,8,9-OCDD	12921	14.1	12700	102



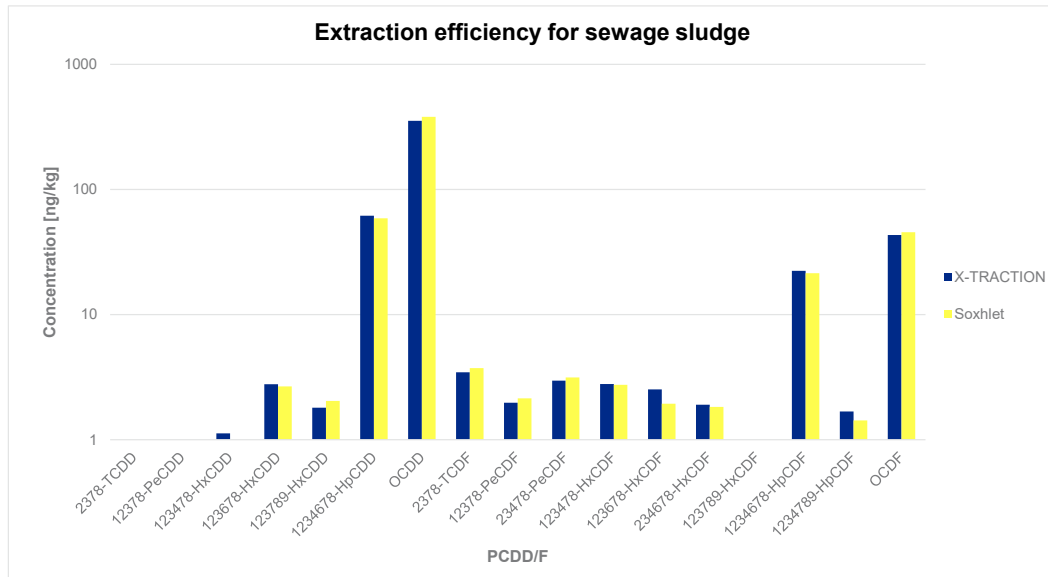
Image 2 LCTech Laboratory, Obertaufkirchen

As shown in the figures above, the recoveries (74 – 131 %) of native PCB and Dioxins are quite good, in comparison to the assigned values of the reference materials.



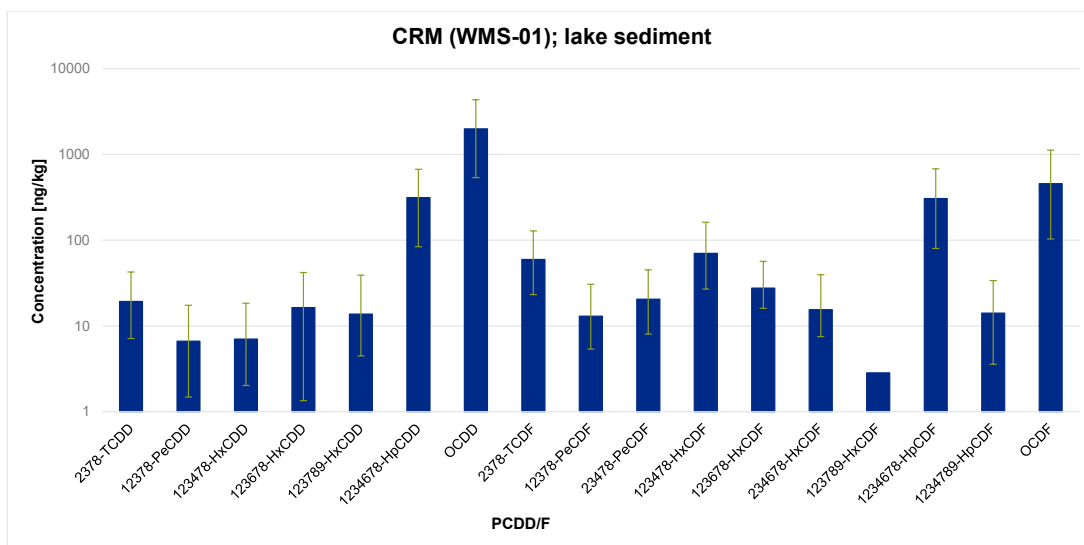
4.3.3 Exemplary Customer Data

This figure shows a comparison between the X-TRACTION® and Soxhlet extraction efficiency of real customer samples processed according to their daily routine laboratory procedure. It shows each PCDD/F in sewage sludge, after extraction, clean-up and evaporation.



As shown above, the efficiency and accuracy of X-TRACTION® extraction are completely comparable to classical Soxhlet extraction, but is much faster (45 minutes instead of several hours) and with significantly less solvent consumption.

This figure shows PCDD/F results of extractions of certified reference material (WMS-01, PCDD/F in lake sediment) are presented. The green lines show the accepted reference value limits. Also these extractions were processed from a customer according to their daily routine laboratory procedure including extraction, clean-up and evaporation.



As shown above, all reference values are obtained perfectly within the accepted reference value limits, with a method that lasts not longer than 15 minutes.



5. Appendix

Dioxin Workflow → From *Sample* to *Chromatography*

2 X-TRACTION® For extraction



3 D-EVA Concentration For the sensor controlled evaporation of extracts



4 DEXTech Product Family Sample preparation for all types of PCB and dioxin analysis

DEXTech Columns Universal and SMART



5 D-EVA Concentration For the sensor controlled evaporation to a few µL



1 Sample Preparation

- Sample intake / weigh in 5 to 10 g
- Homogenization with PAA / Hydromatrix
- Transfer into the extraction cell

2 Extraction: X-TRACTION®

- Sample intake
- Each cycle 20 min inclusive selfcleaning
- Up to 6 modules/extractions in parallel

Collection in

- 60 mL, 120 mL – bottle
- 140 mL – Centrifuge tubes for D-EVA (max. 90 mL capacity)

3 Evaporation: D-EVA

- Up to 11 × 90 mL in parallel without supervision
- Final volume approx. 1 mL
- n-hexane within approx. 40 min
- Toluene within approx. 60 min
- Manual transfer without rinsing to 15 mL Vials
- Add clean-up standard solution (n-Hex/Tol)
- Fill up to approx. 10 mL with n-hexane

4 Clean-up: DEXTech Pure / Plus and 16

- Fraction 1 in 40 mL centrifuge tubes containing PCB in 24 mL n-hexane/DCM
- Fraction 2 in 15 mL centrifuge tubes containing PCDD/F in 10 mL toluene

5 Evaporation: D-EVA

- Up to 23 x PCB or 26 x PCDD/F in parallel without supervision
- PCB within approx. 30 min to approx. 200-300 µL
- PCDD/F within approx. 40 min to 30-100 µL
- Manual transfer without rinsing to GC Vials with Insert
- Blowing down with nitrogen if necessary
- Add syringe standard solution

6 Ready for Analysis

- 6 samples in parallel
- within 5 hours
- manual handling approx. 15 min



6. Conclusion

In summary, the results shown above prove 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.

7. 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.

8. 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



Any Questions?
Do not hesitate to contact us: