

SOLUTIONS BY



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

## Content

1.	Introduction.....	3
2.	Material and Methods .....	4
3.	Filling of Extraction Cell.....	6
4.	Extraction of PCDD/F and PCB in environmental samples .....	10
4.1	Sample preparation .....	10
4.2	Extraction conditions .....	10
4.3	Results for environmental samples .....	11
4.3.1	BCR – 536 – PCB in Harbour Sediment.....	11
4.3.2	BCR – 677 - PCDD/F in sewage sludge .....	12
5.	Conclusion .....	12
6.	Appendix .....	13
7.	Ordering information .....	14
8.	Related Information and References.....	14

## 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 systems 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 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

## APPLICATION NOTE AN0049

- 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)

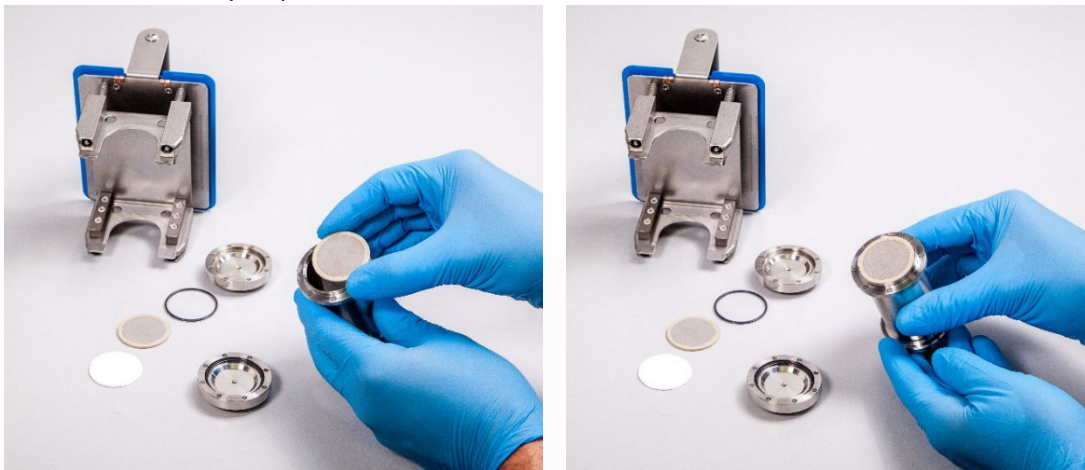
## 3. Filling of Extraction Cell

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:

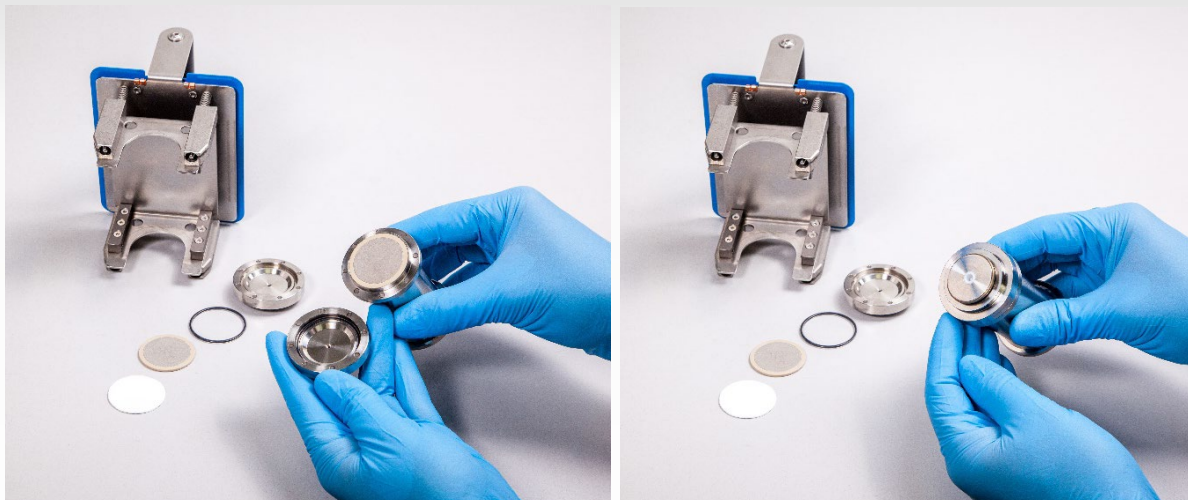


**NOTE:** Keep sealing surfaces clean! In case of dirt or grains, use a clean brush and remove dirt carefully before putting on the frit again.



## APPLICATION NOTE AN0049

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



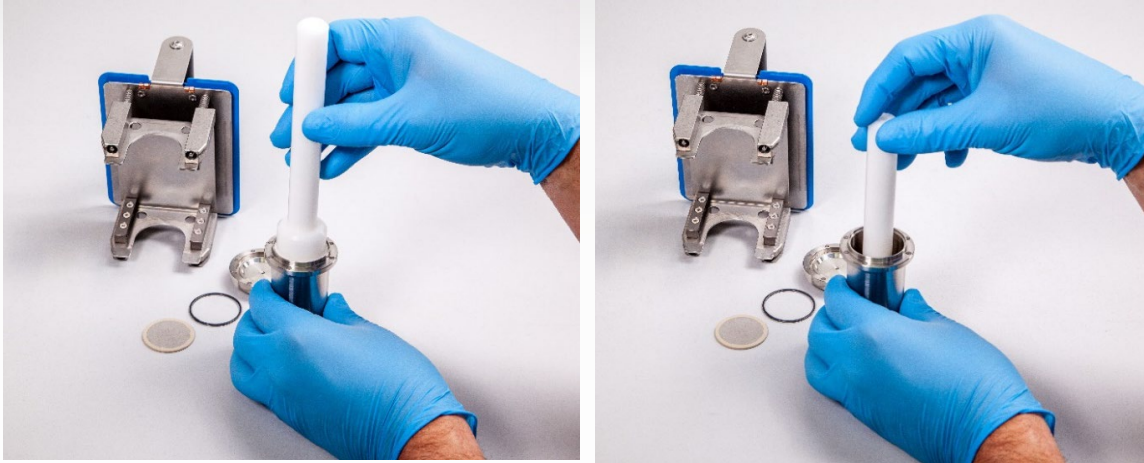
**NOTE:** Ensure that the lid has a solid magnetic connection to the extraction cell. We recommend turning the lid until you feel the magnetic force between the lid and the extraction cell.

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



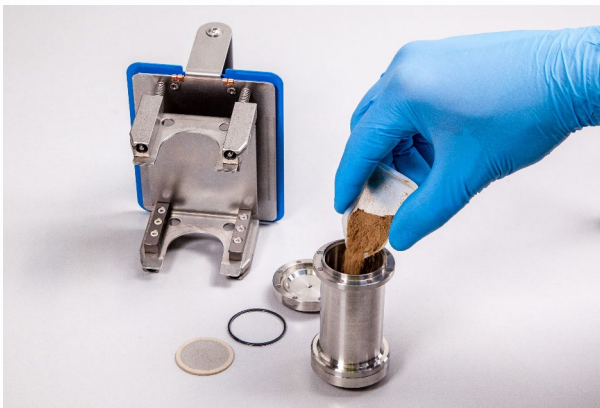
## APPLICATION NOTE AN0049

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.

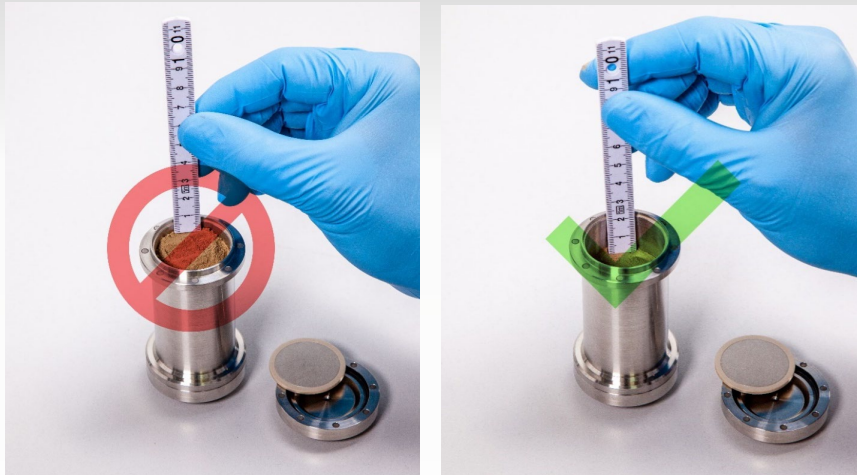
6. Fill your sample. A funnel or a weighing boat is recommended to ensure accurate filling of the cell.



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

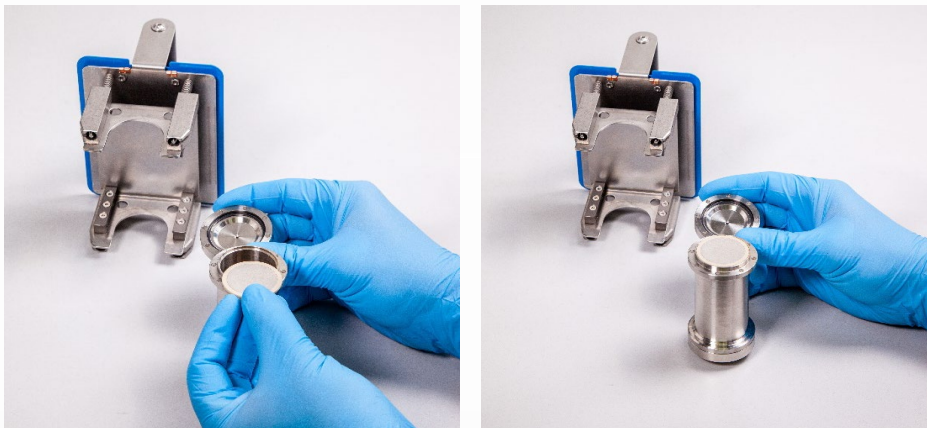


## APPLICATION NOTE AN0049



**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 2 and Table 3 below and cover the complete workflow (extraction, clean-up, evaporation and measurement)."

### 4.3.1 BCR – 536 – PCB in Harbour Sediment

Table 2 shows the results of different PCB congeners of 4 independent extractions of certified reference material (BCR 536, PCB in harbour sediment).

As Table 2 shows, the accuracy for the PCB extractions is quite good, as can be seen in the recoveries that range between 85% and 113%.

native [µg/kg]	mean n=4 [µg/kg]	cert. Value [µg/kg]	recovery [%]
PCB-#28	47.3	44.0	108
PCB-#52	42.9	38.0	113
PCB-#101	42.9	44.0	98
PCB-#118	23.0	27.5	84
PCB-#105	3.0	3.5	87
PCB-#153	53.3	50.0	107
PCB-#138	25.7	27.0	95
PCB-#156	2.7	3.0	90
PCB-#180	21.4	22.4	96
PCB-#128	5.3	5.4	98
PCB-#170	18.1	17.2	105
PCB-#163	13.0	13.4	97



Image 1 LCTech Laboratory, Obertaufkirchen

Table 1 BCR – 536 – PCB in Harbour Sediment

## 4.3.2 BCR – 677 - PCDD/F in sewage sludge

In table 3, 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.

native [pg/g]	mean n=5 [ng/kg]	cert. Value [ng/kg]	recovery [%]
2,3,7,8-TCDF	41	45	91
1,2,3,7,8-PeCDF	23	24.8	93
2,3,4,7,8-PeCDF	16	16.9	95
1,2,3,4,7,8-HxCDF	13	14.5	90
1,2,3,6,7,8-HxCDF	5.7	6.1	93
2,3,4,6,7,8-HxCDF	6.4	5.6	114
1,2,3,7,8,9-HxCDF	1.1	0.8	131
1,2,3,4,6,7,8-HpCDF	59.1	61.6	96
1,2,3,4,7,8,9-HpCDF	4.7	6.3	74
1,2,3,4,6,7,8,9-OCDF	158	177	89
2,3,7,8-TCDD	1.6	1.5	103
1,2,3,7,8-PeCDD	4	4.1	97
1,2,3,4,7,8-HxCDD	nd	nd	nd
1,2,3,6,7,8-HxCDD	239	235	102
1,2,3,7,8,9-HxCDD	73.6	79	93
1,2,3,4,6,7,8-HpCDD	3221	3500	92
1,2,3,4,6,7,8,9-OCDD	12921	12700	102



Image 2 LCTech Laboratory, Obertaufkirchen

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

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.

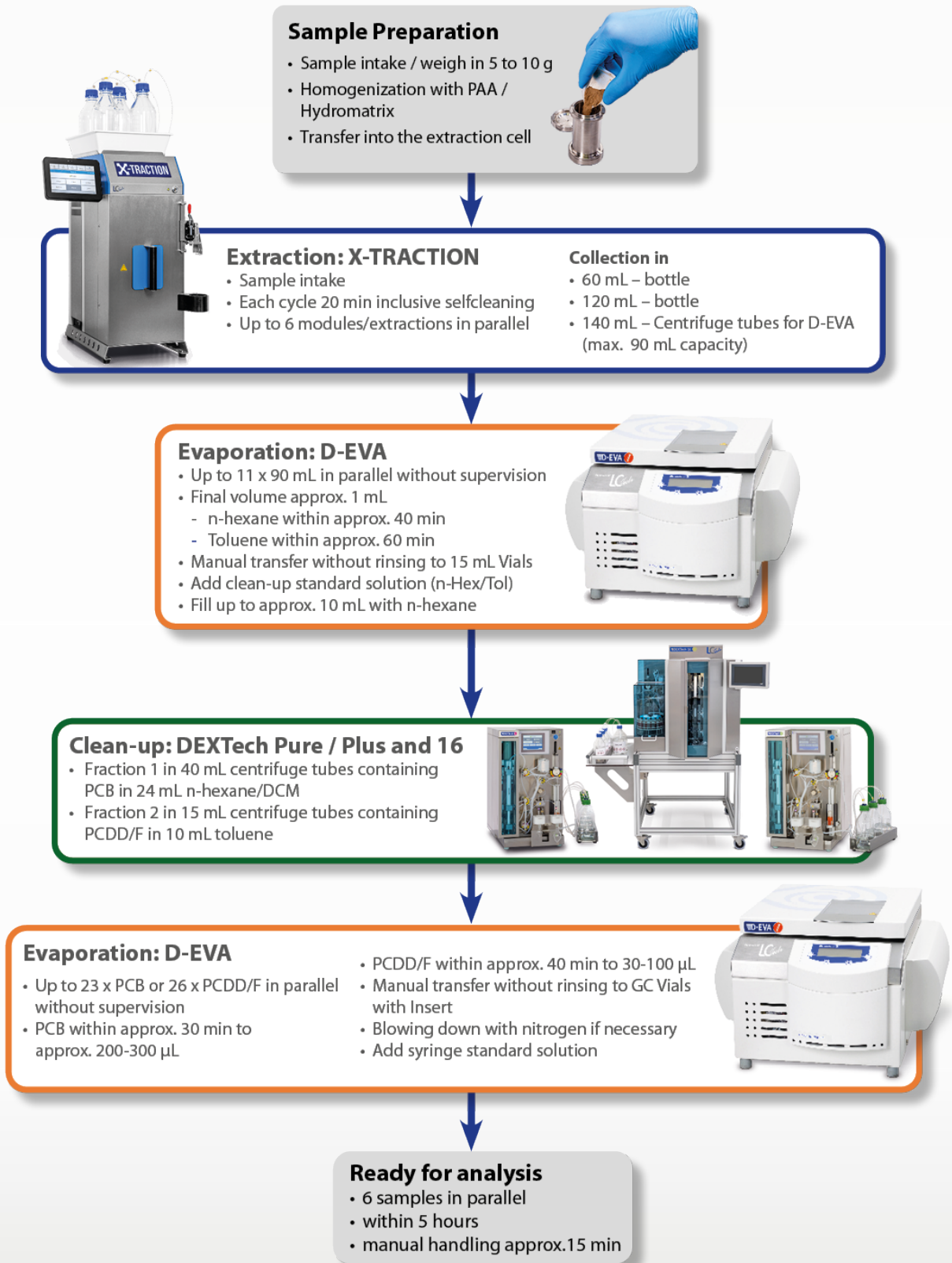
## 5. Conclusion

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. Appendix

### Dioxin Workflow



## 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



## Contact

LCTech GmbH  
Daimlerstraße 4  
84419 Obertaufkirchen  
Germany

SOLUTIONS BY



Tel.: +49 8082 2717-0  
E-Mail: [info@LCTech.de](mailto:info@LCTech.de)

[www.LCTech.de](http://www.LCTech.de)  
[www.LCTech-online.com](http://www.LCTech-online.com)



Image sources:  
© 135804379 / Julian Peters Photos – stock.adobe.com.  
© 296254257 / New Africa – stock.adobe.com.  
© 164670160 / Werner – stock.adobe.com.  
© Tobias Müller