SOLUTIONS BY LCTech

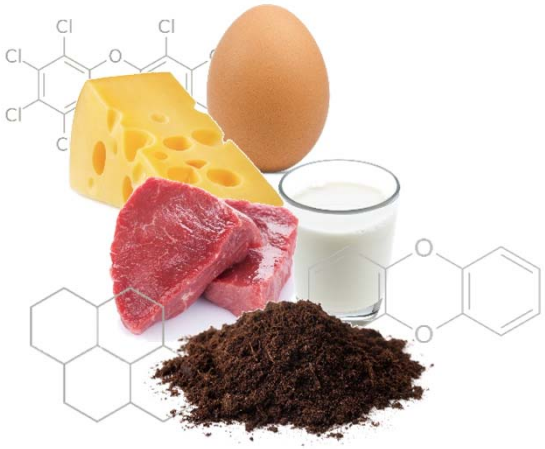
Streamlining the Sample Preparation workflow for POP Analysis

Angelika Köpf, LCTech GmbH

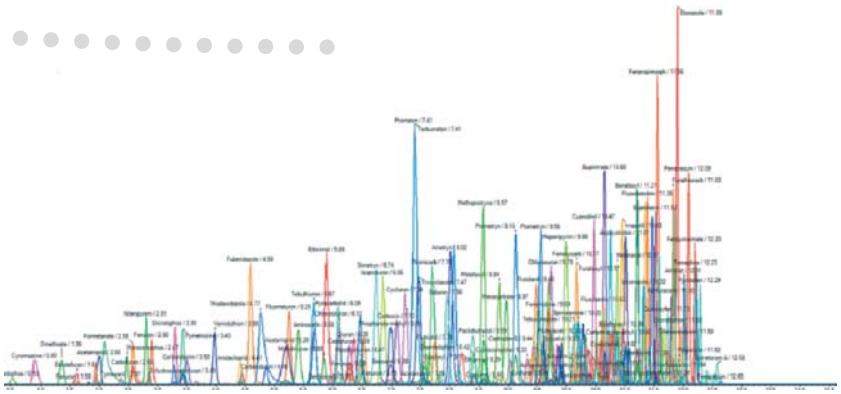
LCTech GmbH

Experts in Sample Preparation since 1998

SOLUTIONS BY LCTech

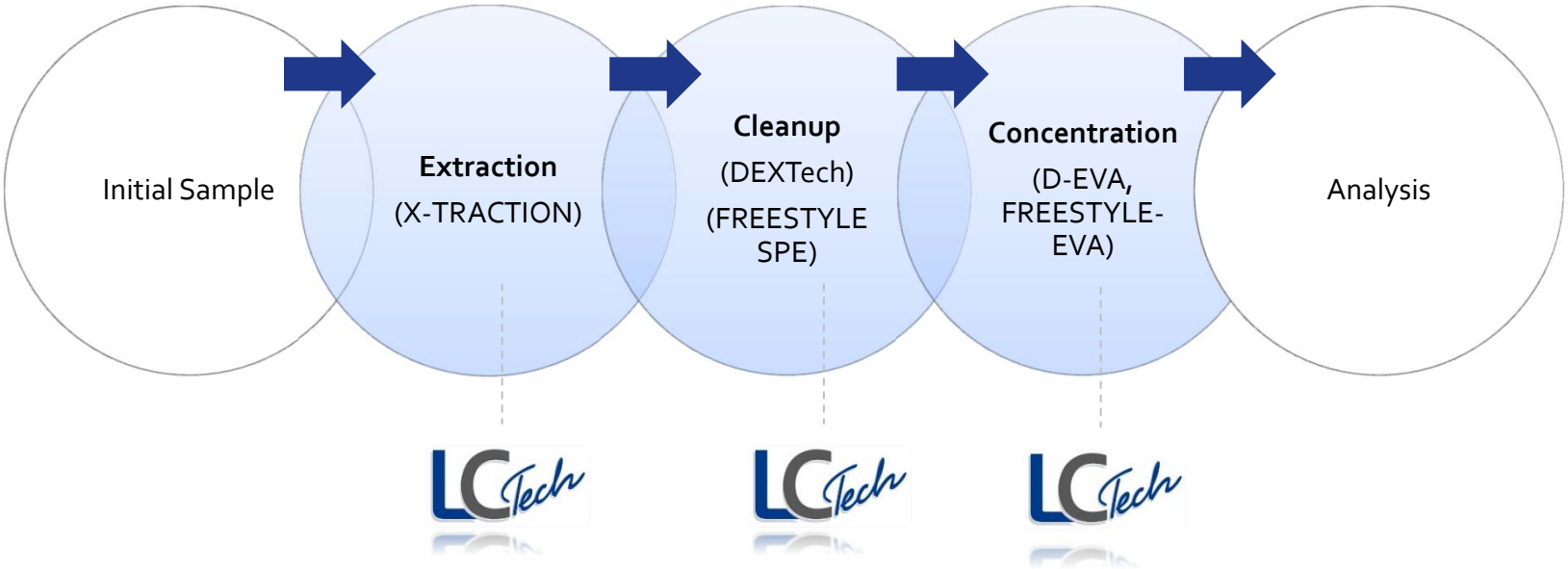


From Sample to Chromatography



Streamlining Sample prep Workflow in POP Analysis

Typical Sample Preparation Workflow



Streamlining Sample prep Workflow in POP Analysis

Common Challenges in sample prep of POP Analysis

SOLUTIONS BY LC Tech

- Reproducible and efficient Extraction, to avoid downtime
- Some analytes should not be evaporated to dryness as they get lost
- Tedious cleanup based on SPE, which is error prone from column preparation to proper fractionation
- Every transfer step can cause errors
- High cost on consumables and solvents
- Avoid blind values and Cross Contamination

Streamlining Sample prep Workflow in POP Analysis

Solution by LCTech – this years Example Dioxin/PCB

SOLUTIONS BY LCTech

Sampling

Extraction

Concentration with D-EVA

-

time-controlled

Clean-Up with DEXTech systems

Concentration with D-EVA

-

final volume-controlled

Analysis

Streamlining Sample prep Workflow in POP Analysis

X-TRACTION for Extraction

SOLUTIONS BY LC Tech

Benchtop unit – no hood
Pre-programmed – easy to use
Operation by touchscreen (reports)

Easy to install

1 unit – 1 sample – up to 6 units

Streamlining Sample prep Workflow in POP Analysis

Expandable Design

SOLUTIONS BY LC Tech

Throughput with runtime 15 min + 3 min change = 18 min = 3,3 per unit / hour
30 per unit / 9 hour workday
180 per 6 units / 9 hour workday

Streamlining Sample prep Workflow in POP Analysis

Unique LCTech Solution – What makes it that Special

SOLUTIONS BY LCTech

- **Easiest**, **quick handling** of cells, no tools needed, intuitive and safe
- **Low cost** investment due to entry level single station (main system) and expandable design (up to five add-on systems)
- **Fast and efficient protocols**
- **Green chemistry!** Low solvent consumption in process and rinsing
- **Low cost** per sample due to **less consumables** (only one filter) and **low wear and tear**

UNIQUE

Streamlining Sample prep Workflow in POP Analysis

Quick and save handling

SOLUTIONS BY LC Tech

- ✓ **Time saving with unique easy closing by magnet**
  - ✓ **No tools needed**
  - ✓ **No risk to damage screw**
- ✓ **Besides the glass fibre filter all parts are reusable for many times**

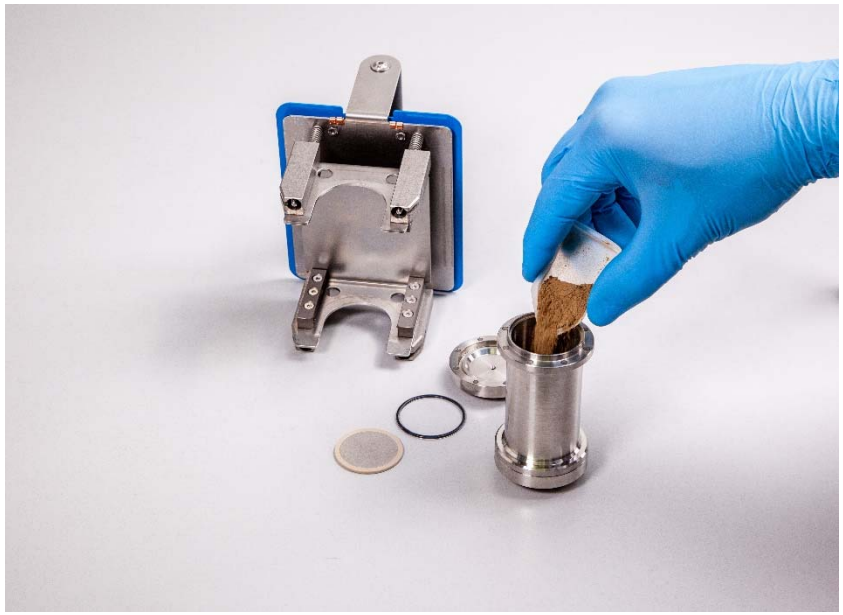
Streamlining Sample prep Workflow in POP Analysis

Efficient Extraction using special concept

SOLUTIONS BY LC Tech

- Fill in sample

- No need to overfill with expensive Hydromatrix
- No need to fill with solvent
- Universal cell size



Streamlining Sample prep Workflow in POP Analysis

Working Principle of X-TRACTION

SOLUTIONS BY LC Tech

- The extraction system works with an air gap in the cell. This allows for an extraction, without generating the high pressure, that other units pose at the sample with the same efficiency
  → **LPFE** - **L**ow **P**ressure **F**luid **E**xtraction.

- Working in **low pressure ranges**, leads to decreased wear and tear of instrument parts, higher longevity and an easy and safe handling.

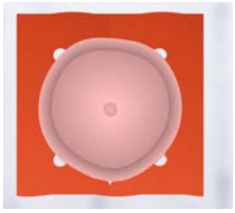
Streamlining Sample prep Workflow in POP Analysis

Efficient process by Design supporting principle

SOLUTIONS BY LC Tech

- Solvent loading

- Solvent heating



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Solution by LCTech – this years Example Dioxin/PCB

SOLUTIONS BY LCTech

Sampling

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Concentration with D-EVA

-

time-controlled

Clean-Up with DEXTech systems

Concentration with D-EVA

-

final volume-controlled

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Streamlining Sample prep Workflow in POP Analysis

Controlled and unattended Evaporation

SOLUTIONS BY LCTech

- Vacuum Centrifuge ideally for parallel processing, space saving
- Ideal Flexibility to insert containers of Extract or Fraction
- D-EVA (Dioxin-EVAporation) consists of:
  - Centrifuge esp. for Dioxins & PCB analysis with special software and Default settings
  - Cold trap
  - Rotor
  - Special LCTech sensor

Streamlining Sample prep Workflow in POP Analysis

Controlled and unattended Evaporation

SOLUTIONS BY LCTech

- Challenge:
  - To stop evaporation at defined low end volume needed for dioxin analysis
  - To avoid the impact of memory effect and "burning" of the material.

➡ **Special LCTech sensor and software** developed for automatic STOP

➡ Energy supply via light that can be stopped

Streamlining Sample prep Workflow in POP Analysis

Default methods

SOLUTIONS BY LC Tech

- Parallel processing of up to 26 toluene samples (dioxins)
- Process time: **37 min**
- Final volumes of about **30 – 100 µL or nearly dryness**

Streamlining Sample prep Workflow in POP Analysis

Solution by LCTech – this years Example Dioxin/PCB

SOLUTIONS BY LCTech

Sampling

Extraction

Concentration with D-EVA

-

time-controlled

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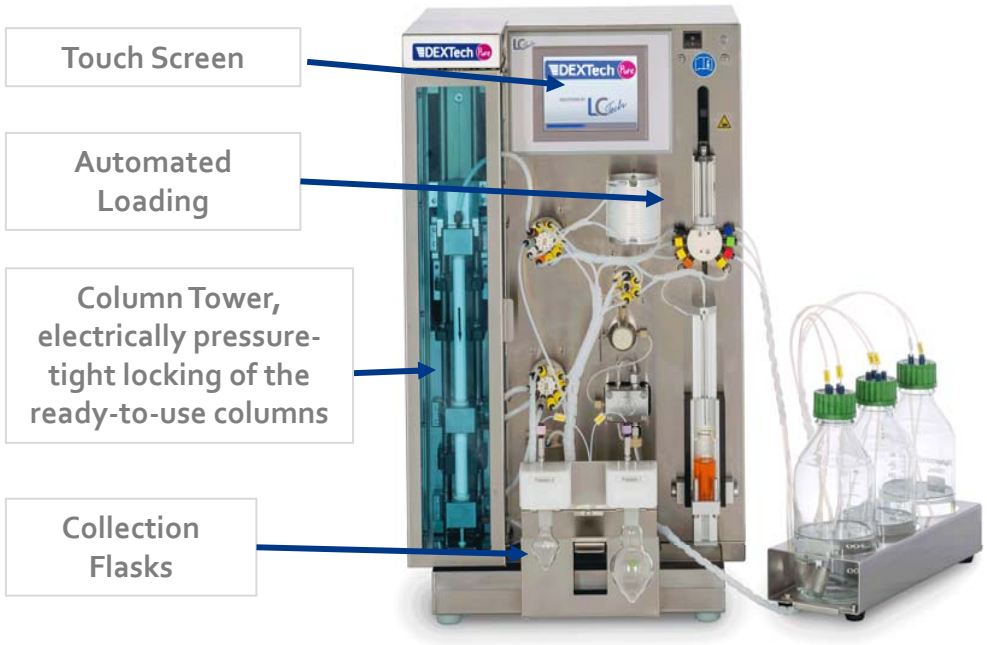
Cleanup unit DEXTech



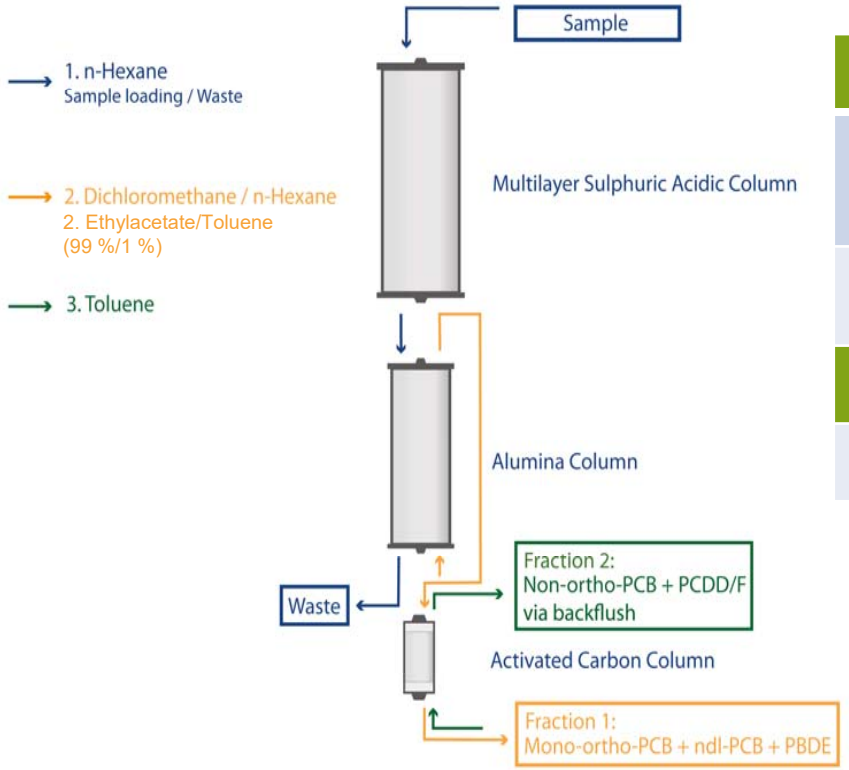
- Bench top unit, all easy accessible
- Easy setup, no fume hood
- Ready to use programms

- High quality columns
- No Cross Contamination
- Highest degree of automation
- Attractive cost for ready to use columns

Streamlining Sample prep Workflow in POP Analysis

Alox Plus Method – conventional or DCM Free

SOLUTIONS BY LC Tech

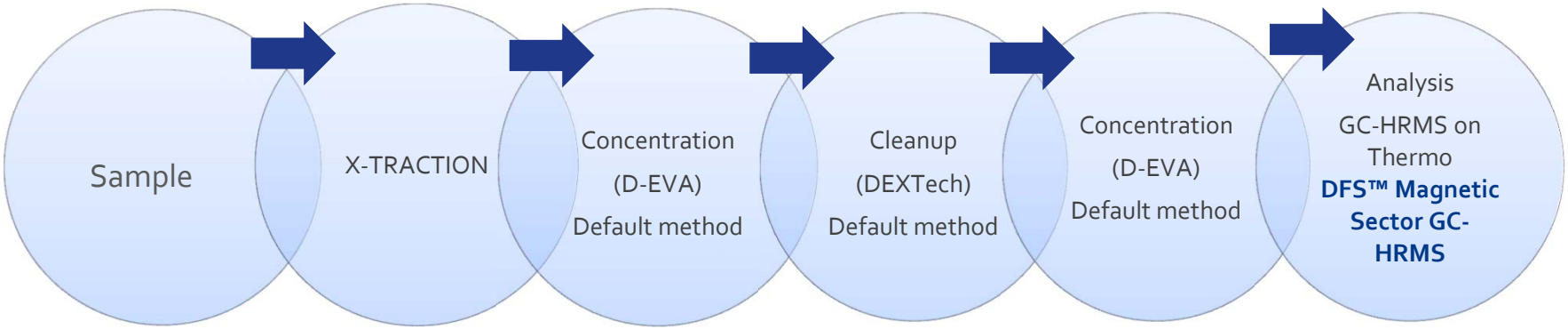


	Universal	SMART
Process Time (includes loading approx. 3 min and conditioning approx. 15 min)	50 min	34 min
Total Solvent Consumption (incl. conditioning and fractions)	230 mL	118 mL
	Fraction 1	Fraction 2
Fraction Volume	24 mL	10 mL

Streamlining Sample prep Workflow in POP Analysis

Results

SOLUTIONS BY LCTech



1 g Harbour sediment reference material	Solvent: Toluene Temperature: 150° C Holding time: 5 Min. # Cycles: 1 Solvent usage (from top, from bottom, rinsing): 50 mL Duration: Approx. 15 minutes	Up to 12 vials with 60mL Toluene Final volume approx. 1 mL in 40 mins Manual transfer to 15 mL Vials Add clean-up standard solution Fill up to approx. 10 mL with n-hexane	Alox Pure method: 50 Mins, collecting PCB in DCM/n-HEX in 24 ml Dioxin in 10ml Toluene	Default method Dioxin: 40 mins to 30µl- 100µl in 40 mins, Default method PCB: 37 mins to 300 µl transferred to vial Blow down with N2 Spike with internal standard

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Analytical Setup

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- GC-HRMS or GC-MS/MS
  - e.g. HRMS-DFS (Thermo Fisher Scientific)
  - e.g. 7010B TripleQuad (GC-MS/MS Agilent)
- PTV-Injector for PCDD/F
- SSL-Injector for PCB
- Analytical columns
  - 60 m – 0,25 mm ID – 0,25 µm df
    - PCDD/F: e.g. Restek, RTX-Dioxin2
    - PCB: e.g. SGE, HT8-PCB

Streamlining Sample prep Workflow in POP Analysis

BCR – 536 – PCB in Harbour Sediment

SOLUTIONS BY LCTech

Results after entire sample preparation and analysis workflow

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

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Streamlining Sample prep Workflow in POP Analysis

BCR – 677 - PCDD/F in sewage sludge

SOLUTIONS BY LCTech

Results after entire sample preparation and analysis workflow

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	n.d
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

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Streamlining Sample prep Workflow in POP Analysis

Excellent Precision for PCDD/F

SOLUTIONS BY LC Tech

	Extraction 1	Extraction 2	Extraction 3	Extraction 4	Mean	Deviation	RSD %
	Fresh weight ng/kg	Fresh weight ng/kg	Fresh weight ng/kg	Fresh weight ng/kg	Fresh weight ng/kg		
Polychlorinated Dibenzofurane							
2,3,7,8-TeCDF	0.4292	0.4263	0.3877	0.3862	**0.4074**	**0.0204**	**5.0**
1,2,3,7,8-PeCDF	0.2477	0.2428	0.2462	0.2615	**0.2496**	**0.0071**	**2.9**
2,3,4,7,8-PeCDF	0.4973	0.5043	0.4909	0.4892	**0.4954**	**0.0059**	**1.2**
1,2,3,4,7,8-HxCDF	0.3864	0.3791	0.3631	0.3696	**0.3746**	**0.0089**	**2.4**
1,2,3,6,7,8-HxCDF	0.2518	0.256	0.2447	0.247	**0.2499**	**0.0044**	**1.7**
2,3,4,6,7,8-HxCDF	0.2585	0.2687	0.2752	0.2619	**0.2661**	**0.0064**	**2.4**
1,2,3,7,8,9-HxCDF	0.1844	0.1819	0.1881	0.1832	**0.1844**	**0.0023**	**1.3**
1,2,3,4,6,7,8-HpCDF	0.3229	0.3448	0.3284	0.3236	**0.3299**	**0.0088**	**2.7**
1,2,3,4,7,8,9-HpCDF	0.2241	0.2323	0.2236	0.2165	**0.2241**	**0.0056**	**2.5**
OCDF Octachlordibenzofuran	0.9901	1.043	0.9885	1.001	**1.0057**	**0.0221**	**2.2**
Polychlorinated Dibenzodioxin							
2,3,7,8-TeCDD	0.2163	0.1917	0.1949	0.1634	**0.1916**	**0.0188**	**9.8**
1,2,3,7,8-PeCDD	0.247	0.2377	0.2255	0.2275	**0.2344**	**0.0086**	**3.7**
1,2,3,4,7,8-HxCDD	0.1831	0.1759	0.19	0.2084	**0.1894**	**0.0121**	**6.4**
1,2,3,6,7,8-HxCDD	0.2067	0.2032	0.2019	0.1904	**0.2006**	**0.0061**	**3.1**
1,2,3,7,8,9-HxCDD	0.2422	0.2373	0.2526	0.2351	**0.2418**	**0.0067**	**2.8**
1,2,3,4,6,7,8-HpCDD	0.2567	0.2704	0.2598	0.2401	**0.2568**	**0.0109**	**4.2**
OCDD Octachlordibenzodioxin	1.472	1.409	1.389	1.353	**1.4058**	**0.0432**	**3.1**

Data provided by Dr. Bernsmann, Chemical and Veterinary Analytical Institute, Münsterland-Emscher-Lippe (CVUA-MEL), Muenster, Germany

Streamlining Sample prep Workflow in POP Analysis

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Excellent Precision for PCB

Quality Control Feed Material FMP

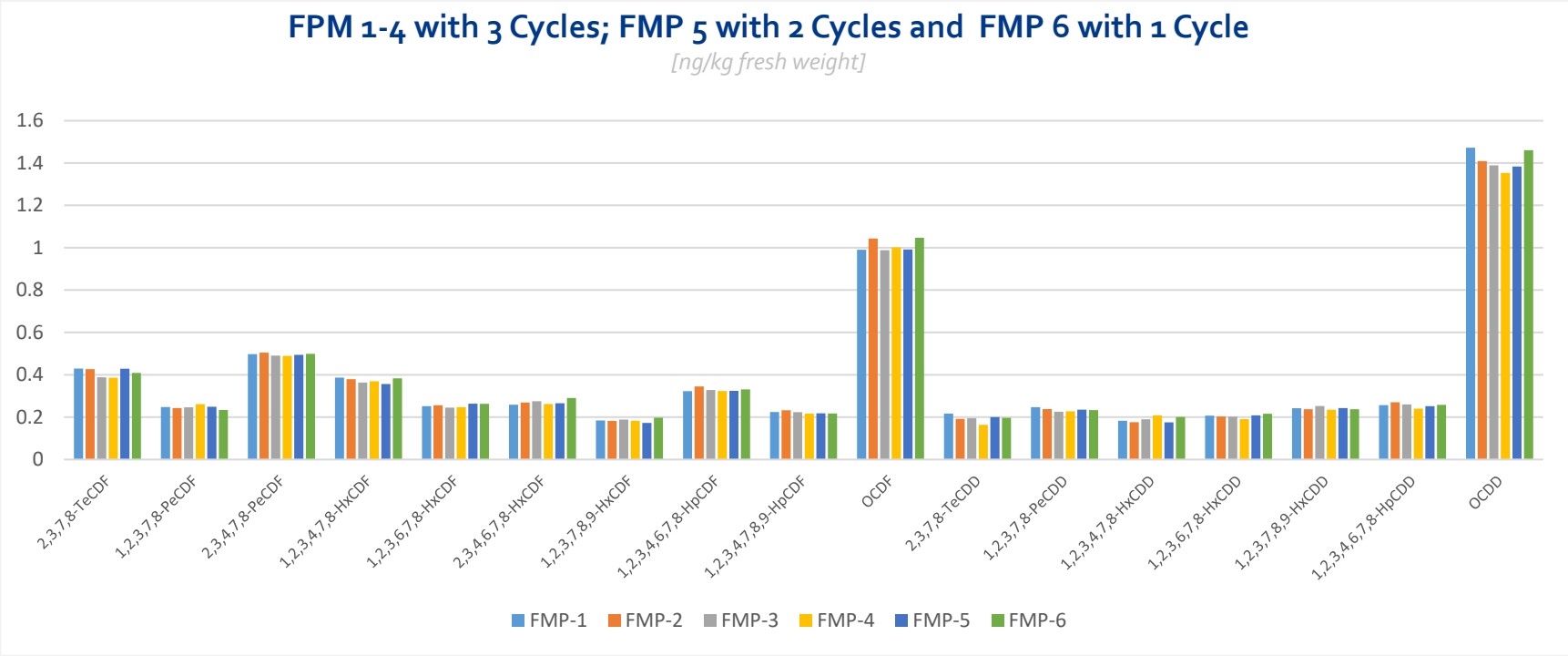
SOLUTIONS BY LC Tech

	Extraction 1	Extraction 2	Extraction 3	Extraction 4	Mean	STABWN	RSD %
	Fresh weight | ng/kg	Fresh weight | ng/kg	Fresh weight | ng/kg	Fresh weight | ng/kg	Fresh weight | ng/kg		
non-ortho PCB							
PCB 77	74.58	77.17	77.39	73.8	75.74	1.57	2.1
PCB 81	6.957	7.275	6.708	6.934	6.969	0.2019	2.9
PCB 126	4.561	4.628	4.575	4.43	4.549	0.0728	1.6
PCB 169	2.542	2.546	2.516	2.405	2.5023	0.0573	2.3
mono-ortho PCB							
PCB 105	370.4	378.8	370.9	368.1	372.1	4.0376	1.1
PCB 114	33.57	33.36	33.13	31.39	32.9	0.8643	2.6
PCB 118	886.1	934.4	905.6	899.5	906.4	17.64	1.9
PCB 123	23.05	23.69	23.32	23.00	23.3	0.2739	1.2
PCB 156	493.1	503.8	496.7	498.8	498.1	3.8710	0.8
PCB 157	53.14	53.23	52.49	51.88	52.7	0.5455	1.0
PCB 167	229.7	224.5	224.4	223.6	225.6	2.4213	1.1
PCB 189	117.2	122.3	118.3	116.9	118.7	2.1568	1.8
ndl-PCB	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	µg/kg	
PCB 28	0.7508	0.7508	0.7764	0.7697	0.7619	0.0114	1.5
PCB 52	1.705	1.705	1.719	1.713	1.7105	0.0059	0.3
PCB 101	2.498	2.498	2.53	2.477	2.5008	0.0189	0.8
PCB 138	3.771	3.771	3.742	3.674	3.7395	0.0396	1.1
PCB 153	4.603	4.603	4.653	4.611	4.6175	0.0208	0.4
PCB 180	4.437	4.437	4.483	4.361	4.4295	0.0438	1.0

Streamlining Sample prep Workflow in POP Analysis

Exceptional Precision PCDD/F

SOLUTIONS BY LC Tech



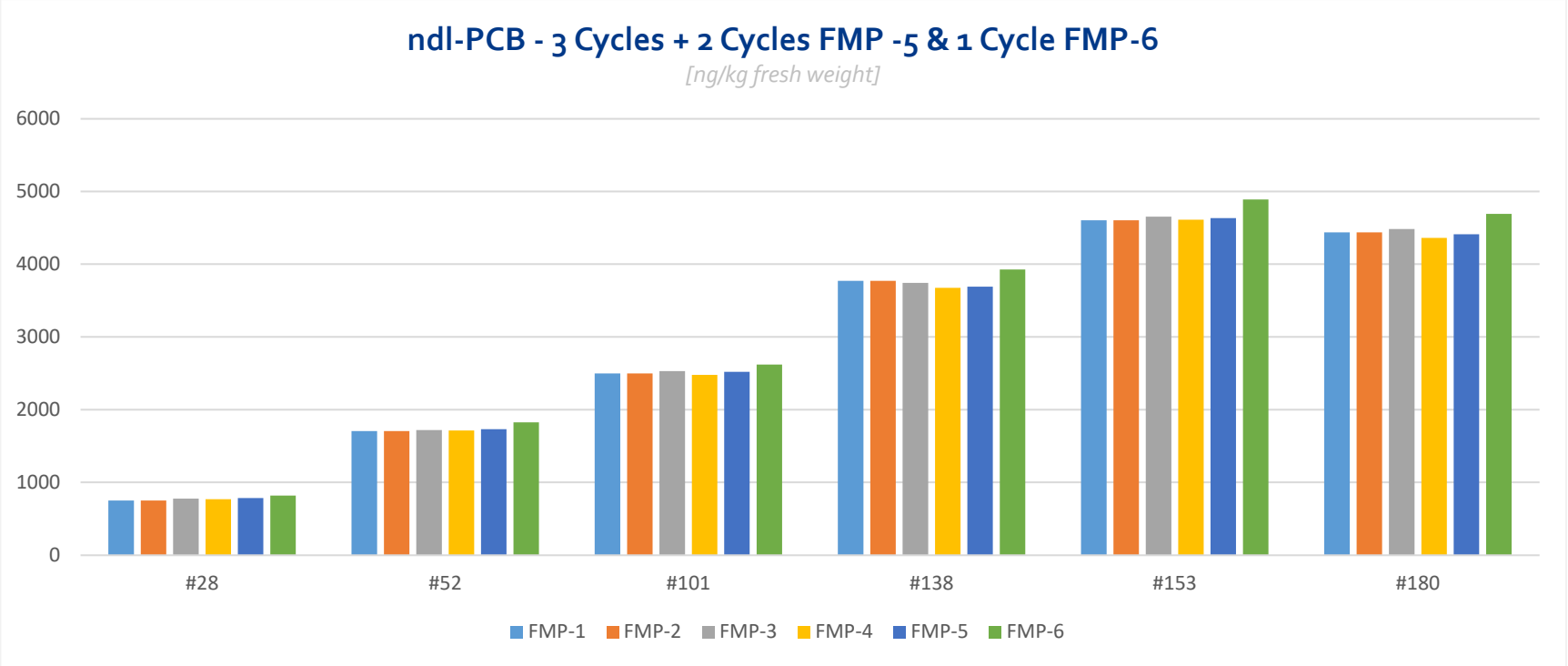
Quality Control Feed Material FMP

Data provided by Dr. Bernsmann, Chemical and Veterinary Analytical Institute, Münsterland-Emscher-Lippe (CVUA-MEL), Muenster, Germany

Streamlining Sample prep Workflow in POP Analysis

Exceptional Precision for PCB

SOLUTIONS BY LC Tech



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Streamlining Sample prep Workflow in POP Analysis

Customer Carry Over Test

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PCDD/Fs	Carry Over (%)
2378-TetraCDF	0.000999476
2378-TetraCDD	0.000933642
12378-PentaCDF	0.000894312
23478-PentaCDF	0.001000214
12378-PentaCDD	0.000927239
123478-HexaCDF	0.000897438
123678-HexaCDF	0.000799541
234678-HexaCDF	0.001123207
123478-HexaCDD	0.000950550
123678-HexaCDD	0.001042591
123789-HexaCDD	0.001110944
123789-HexaCDF	0.001051582
1234678-HeptaCDF	0.001760539
1234678-HeptaCDD	0.003740431
1234789-HeptaCDF	0.001014591
OctaCDD	0.008554827
OctaCDF	0.002093968

PCB	Carry Over (%)
PCB-28 **	0.087920007
PCB-52 **	0.057359327
PCB-101 **	0.042526266
PCB-81	0.001353596
PCB-77	0.005647445
PCB-123	0.000957830
PCB-118	0.032228256
PCB-114	0.000778819
PCB-153 **	0.061951924
PCB-105	0.016001291
PCB-138 **	0.023677053
PCB-126	0
PCB-167	0.003419919
PCB-156	0.004653067
PCB-157	0.003789838
PCB-180 **	0.017443246
PCB-169	0.004602942
PCB-189	0

For the test two certified reference materials (soil) have been used

(BCR - 529 Industrial soil and SQC068 - 50 g)

Each time the high contaminated soil sample was performed including with the DEXTech Plus system followed with a blank sample.

Streamlining Sample prep Workflow in POP Analysis

Separate Cross contamination test in D-EVA

SOLUTIONS BY LC Tech

- Centrifuge glasses in 90° and 180° orientation
- Standard 4 x highest calibration level
- No detectable signal / cross-contamination by GC-HRMS (Thermo DFS)

Streamlining Sample prep Workflow in POP Analysis

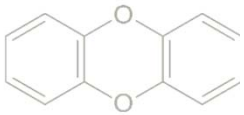
Advantages in this automated processing

SOLUTIONS BY LCTech

- Complete workflow from sample to injection, whilst still having control over each chapter
- Unmatched easy and safe handling of X-TRACTION, quick, low cost and solvent saving
- Cleanup on DEXTech brings great performance with safe operation, Time saving, solvent saving and cost saving flexibility of protocols and high quality Columns
- D-EVA saves the sensitive fractions with unique safety stop
- Design and material chosen ensure no blind values and no Cross Contamination
- All in all Robust LCTech engineering for reliable operation and low maintenance

Streamlining Sample prep Workflow in POP Analysis

Any Questions?



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Thank you for your attention!

SOLUTIONS BY LCTech

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SAMPLE PREPARATION & ANALYSIS

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X-TRACTION

EPA Compliance

SOLUTIONS BY LC Tech

Text of EPA 3534A (SW 846):

2.2 The extraction cell containing the sample is heated to the extraction temperature (see Sec. 11.8), ***pressurized with the appropriate solvent system****, and extracted for 5-10 minutes* ***(or as recommended by the instrument manufacturer).*** *Multiple extractions are 3545A - 3 Revision 1 February 2007 recommended for some groups of analytes. The solvent systems used for this procedure vary with the analytes of interest and are described in Sec. 7.7.*

6.1.2 Other system designs may be employed, provided that adequate performance can be demonstrated for the analytes and matrices of interest

*11.9.4 Optimize the conditions, as needed, according to the manufacturer's instructions****. In general, the pressure is not a critical parameter, as the purpose of pressurizing the extraction cell is to prevent the solvent from boiling at the extraction temperature and to ensure that the solvent remains in intimate contact with the sample.***

Proof on Performance data , that X-TRACTION is as efficient