

DEXTech Heat for Difficult Matrices

Cleaning Palm Fatty Acid Distillate (PFAD) and Other Difficult Matrices for PCDD/F & PCB Analysis

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Introduction

Palm and palm kernel oil as well as many of their by-products like palm fatty acid distillates (PFAD), or palm stearin are used nowadays in a variety of products. You can find them in many processed foods, feed supplements, cosmetics or biofuels. In order to ensure the quality and safety palm oils and its derived products they are tested for a variety of contamination like PCDD/F as well as dioxin like and non-dioxin like PCBs.

PFADs and other palm oil derivatives contain high amounts of free fatty acids with high melting points like palmitic acid or stearic acid. This often leads to a low solubility of these substances in n-hexane at room temperature and can make the clean-up of the dioxin and PCB analysis quite tricky with clogged columns and low recoveries.

The DEXTech Heat system was developed especially for matrices with a high melting point but of course can be used for standard fat/oil samples as well. The system has three different heated zones, the heated sample holder, the heated sample loop and some heated tubes to ensure that the sample stays liquefied during the whole clean-up process. These features ensure an easy handling of difficult matrices as well as an increase in the clean-up performance.



“ The quantitative transfer of PFAD from the sample vial onto the clean-up columns is very critical and too often the reason for minor results and bad recoveries. The DEXTech Heat system overcomes this challenge with a heated sample holder that keeps the sample liquefied during the whole clean-up process as well as automated rinsing of the sample vial, which ensures the quantitative transfer of the sample. ”

Thomas Kerkemeier, Dioxin Specialist at LCTech GmbH

Sample Preparation Clean-up

To prepare the sample, melt the fat / oil sample at about 80 °C and weigh 3 g of the liquefied sample into the sample vial. Add 1 mL of toluene, 1 mL of methanol and the labelled compound standards (¹³C₁₂ quantification-standards) to the sample and fill up the vial to an end volume of 17 mL with n-hexane. Place the sample vial in the heated sample vial holder to keep the sample liquefied.

The maximum fat/oil content in a DEXTech Heat sample is 3 g for samples with a high melting point and 5 g for standard fat/oil samples.

Start the clean-up process.



After the clean-up the sample fractions have been evaporation down to near dryness with the automated evaporation system D-EVA. All samples and solvent blanks are analyzed with a DFS HRMS from Thermo Fisher Scientific. The PCB fraction 1 is injected in SSL mode onto a 60m HT8 PCB capillary column from Trajan and the PCDD/F fraction is injected in PTV split less mode onto a 60m RTX Dioxin2 capillary column from Restek.

Matrices that have been Tested

- PFAD: Palm Fatty Acid Distillates
- HSFA: High saturated fatty acids (Feed additive)
- PALMAC® 80-16 fatty acids (min 80 % palmitic acid) (Feed additive)
- Palm Stearin

Proficiency Test Materials

- Palm Fatty Acid Distillate (1701-PF), EURL Freiburg
- Feed Fat (1301-FF), EURL Freiburg

Results

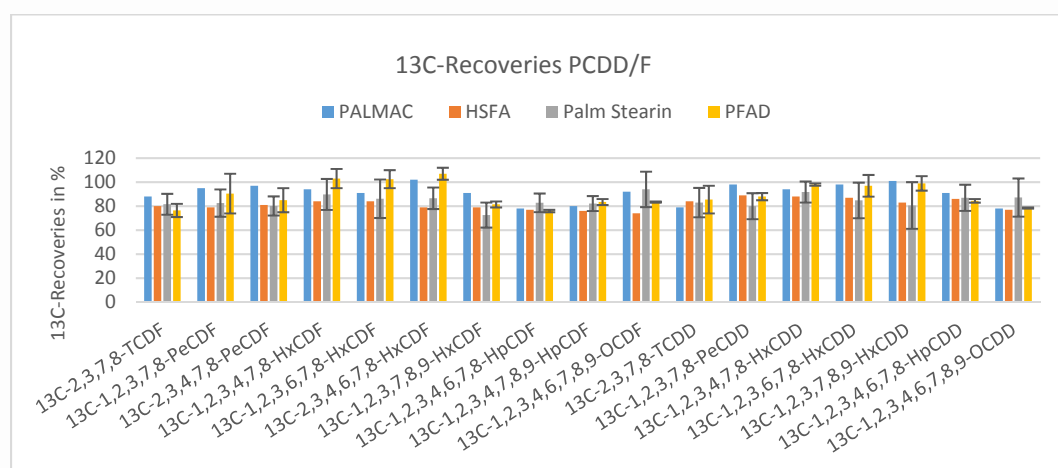


Figure 1: 13C- PCDD/F recoveries

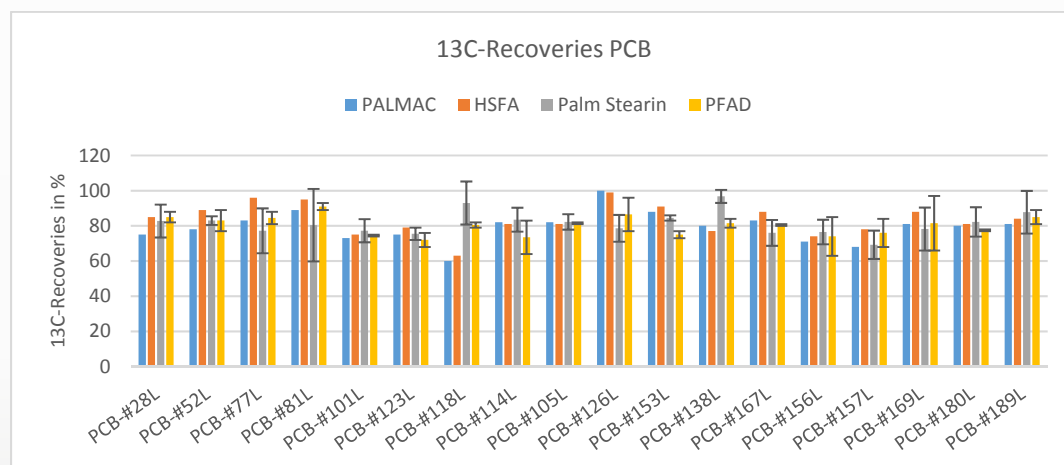


Figure 2: 13C- PCB recoveries

The efficiency of the clean-up is outstanding. There are far less interferences than normally known in a fully manual set-up with extraction and cold clean-up. This makes the interpretation of the chromatograms easier and will give very good 13C- recoveries as well as a higher signal/noise ratio for a better quantification limit.

Proficiency Test Materials

Table 1: Comparison DEXTech Heat results with the results from the EURL-proficiency test

PFAD	EURL-PT-DP_1701-PF			Feed Fat	Feed Fat (1307-FF)		
	DEXTech Heat	Assigned Value	z-score		DEXTech Heat	Assigned Value	z-score
	[pg/g]	[pg/g]			[pg/g]	[pg/g]	
2,3,7,8-TCDF	0,587	0,565	0,2	2,3,7,8-TCDF	0,334	0,306	0,5
1,2,3,7,8-PeCDF	0,260	0,227	0,7	1,2,3,7,8-PeCDF	0,150	0,132	0,7
2,3,4,7,8-PeCDF	0,387	0,324	1,0	2,3,4,7,8-PeCDF	0,168	0,139	1,1
1,2,3,4,7,8-HxCDF	0,19	0,17	0,7	1,2,3,4,7,8-HxCDF	0,155	0,142	0,5
1,2,3,6,7,8-HxCDF	0,17	0,15	0,7	1,2,3,6,7,8-HxCDF	0,0788	0,0869	0,5
2,3,4,6,7,8-HxCDF	0,15	0,16	0,2	2,3,4,6,7,8-HxCDF	0,049	(0,065)	*
1,2,3,7,8,9-HxCDF	0,0711	(0,0712)	*	1,2,3,7,8,9-HxCDF	<0,03	(0,052)	*
1,2,3,4,6,7,8-HpCDF	0,579	0,606	0,2	1,2,3,4,6,7,8-HpCDF	0,211	0,197	0,4
1,2,3,4,7,8,9-HpCDF	0,065	(0,06)	*	1,2,3,4,7,8,9-HpCDF	0,036	(0,054)	*
1,2,3,4,6,7,8,9-OCDF	0,21	0,24	0,6	1,2,3,4,6,7,8,9-OCDF	0,34	(0,33)	*
2,3,7,8-TCDD	0,156	0,129	1,0	2,3,7,8-TCDD	<0,02	(0,042)	*
1,2,3,7,8-PeCDD	0,19	0,23	0,9	1,2,3,7,8-PeCDD	0,071	(0,048)	*
1,2,3,4,7,8-HxCDD	0,162	0,155	0,2	1,2,3,4,7,8-HxCDD	0,0212	(0,044)	*
1,2,3,6,7,8-HxCDD	0,209	0,254	0,9	1,2,3,6,7,8-HxCDD	<0,03	(0,05)	*
1,2,3,7,8,9-HxCDD	0,720	0,619	0,8	1,2,3,7,8,9-HxCDD	<0,03	(0,05)	*
1,2,3,4,6,7,8-HpCDD	3,98	3,95	0,0	1,2,3,4,6,7,8-HpCDD	0,249	0,257	0,1
1,2,3,4,6,7,8,9-OCDD	232	194	1,0	1,2,3,4,6,7,8,9-OCDD	2,84	2,42	0,9
PCB 28	366	338	0,4	PCB 28	170	(230)	*
PCB 52	252	278	0,5	PCB 52	248	268	0,4
PCB 77	17,4	17,8	0,1	PCB 77	85,7	85,2	0,0
PCB 81	0,755	0,843	0,5	PCB 81	2,67	2,63	0,1
PCB 101	272	375	1,4	PCB 101	631	629	0,0
PCB 123	6,52	7,26	0,5	PCB 123	22,6	22,1	0,1
PCB 118	384	392	0,1	PCB 118	1010	990	0,1
PCB 114	11,0	9,22	0,9	PCB 114	33,0	30,1	0,5
PCB 105	157	151	0,2	PCB 105	607	546	0,6
PCB 126	3,23	3,16	0,1	PCB 126	19,6	18	0,4
PCB 153	810	784	0,2	PCB 153	820	749	0,5
PCB 138	567	541	0,2	PCB 138	1120	966	0,8
PCB 167	24,4	23,8	0,1	PCB 167	62,1	53,3	0,8
PCB 156	42,9	38,9	0,5	PCB 156	158	136	0,8
PCB 157	8,2	10,8	1,2	PCB 157	36,8	34,5	0,3
PCB 169	0,785	0,751	0,2	PCB 169	0,453	(0,515)	*
PCB 180	257	217	0,9	PCB 180	308	245	1,3
PCB 189	4,03	4,21	0,2	PCB 189	7,74	(7,01)	*

* Values in () are not assigned values, but the median of all values

The results are in good agreement with the results of the proficiency test and therefore show the excellent usability of the DEXTech system for the task.

These LCTech Products were Used for the Tests

Instruments & Columns

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|---|-----------|
| 1. DEXTech Heat | P/N 16800 |
| 2. Rotational-vacuum-concentrator D-EVApotation | P/N 16900 |
| 3. Rotor with 48 positions | |
| • Angle rotor, 48 positions | P/N 16742 |
| • Sensor for 15 mL vial | P/N 16741 |
| 4. Rotor with 24 positions | |
| • Angle rotor, 24 positions | P/N 16802 |
| • Sensor for 24 mL vial | P/N 16738 |
| 5. Universal acidic silica gel column | P/N 15068 |
| 6. Alumina column | P/N 15433 |
| 7. Carbon column | P/N 15242 |