

DEXTech Goes Green

Now Available: Dichloromethane Free Method to fractionate your Dioxins and PCB on all DEXTech Pure, Plus, 16 and Heat

By its properties, Dichloromethane (DCM) is solvent of choice in many extraction and clean-up methods, also for PCBs and PCDD/Fs. Unfortunately DCM is toxic, risks serious health effects and is considered as likely carcinogenic. Therefore, authorities might request to avoid it best possible in the laboratories and more laboratories try to identify DCM free applications to reduce the exposure to their staff.

Following this development we have revised the proven Alox Plus method for this demand successfully and can replace the n-hexane/DCM (50 %/50 %) mixture in the method with a mixture of ethyl acetate/toluene (99 %/1 %).

Easy Change to the New Method

In order to use this new method, technically you just have to replace the solvent supply bottle with the DCM/n-hexane mixture against the ethyl acetate/toluene (99 %/1 %) mixture. That's all! Purge your solvent line and you are ready to start your next sample with the DCM free method.

The DCM-free Alox Plus method has all the same advantages of our DCM method Alox Plus:

The method is:

- **Fast**
50 minutes for up to 5 g fat
35 minutes for up to 1 g fat
- **Applicable for up to 5 g fat per sample**
- **Has low fraction volumes**

Fraction 1 (ndl-PCBs and mono-ortho PCBs) 24 mL ethyl acetate/toluene (99 %/1 %)

Fraction 2 (non-ortho PCBs and PCDD/Fs) 10 mL toluene like before

- **Reliable**



In the following, results from different oil samples cleaned with the new DCM free Alox Plus method are shown.

Method of Sample Preparation / Clean-up

The ethyl acetate/toluene (99 %/1 %) mixture was connected to the DEXTech instrument and the system was purged. 3 g of oil sample was spike with the labelled compound standard ($^{13}\text{C}_{12}$ quantifications standard). 1 mL toluene was added and the sample was filled up to 10 mL with n-hexane. Start the Alox Plus method as preconfigured in the system.

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

Results

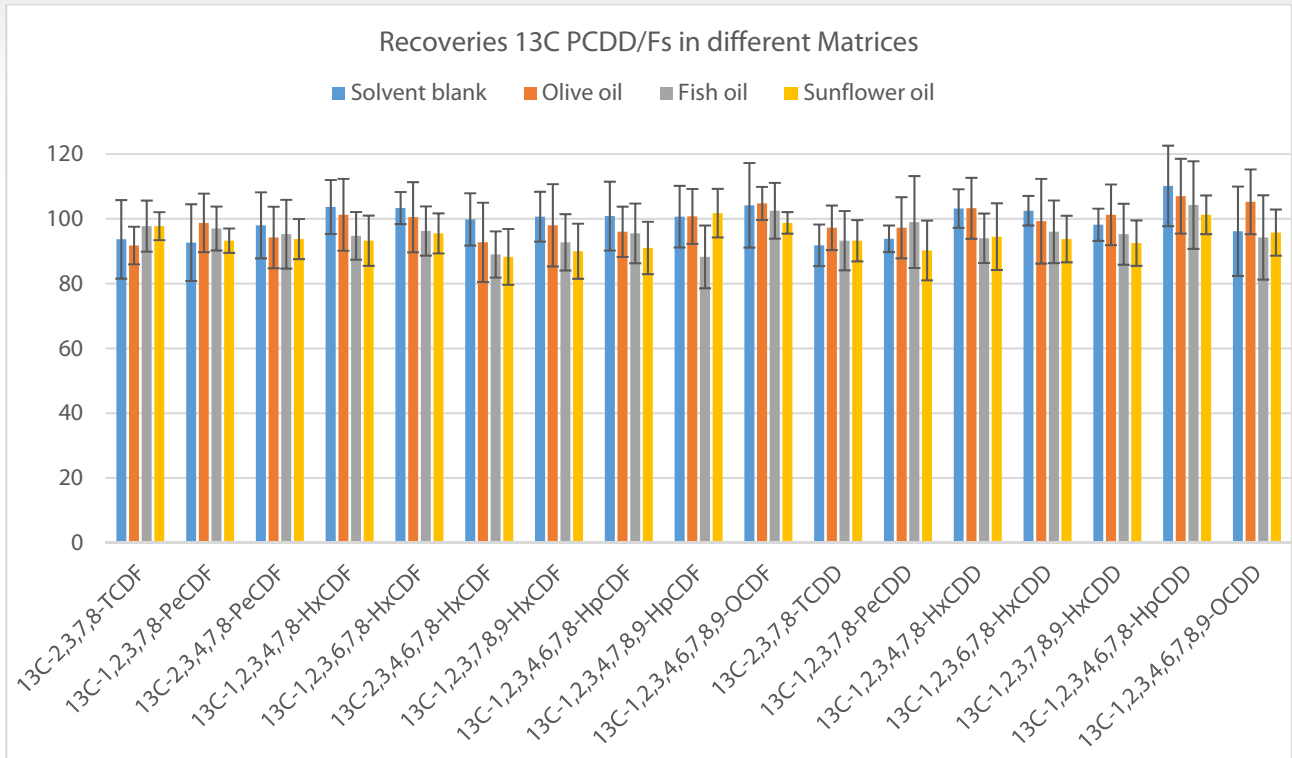


Figure 1: Shows the 13C PCDD/F recoveries for different oil samples prepared with the new DCM free method

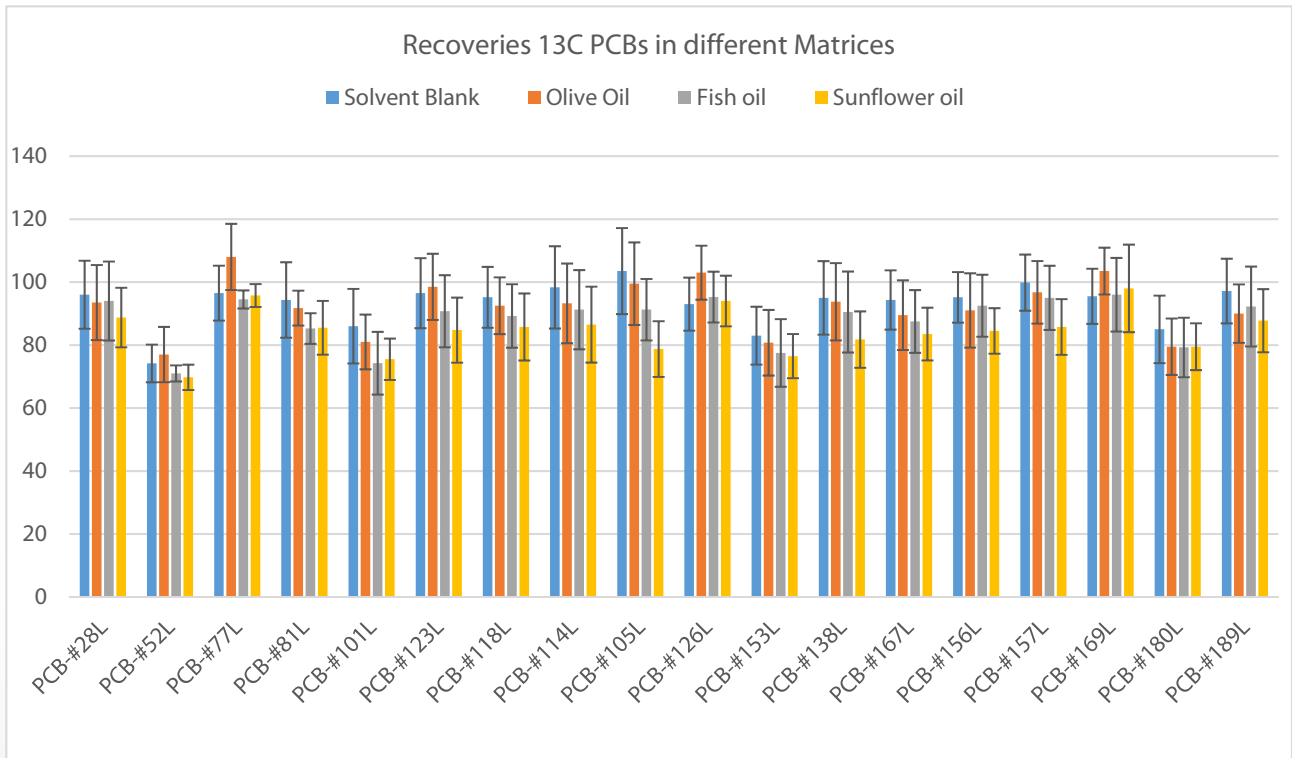


Figure 2: Shows the 13C PCB recoveries of different oil samples prepared with the new DCM free method

Table 1: Clean-up result of a Proficiency Test PFAD Material with the classical Alox Plus method (with DCM) and the new DCM free Alox Plus method.

EURL-PT-DP_1701-PF					
	assigned value	DCM Alox Plus Methode	z-score	DCM free Alox Plus Method	z-score
	[pg/g]	[pg/g]		[pg/g]	
2,3,7,8-TCDF	0,565	0,587	0,2	0,515	0,4
1,2,3,7,8-PeCDF	0,227	0,260	0,7	0,240	0,3
2,3,4,7,8-PeCDF	0,324	0,387	1,0	0,320	0,1
1,2,3,4,7,8-HxCDF	0,17	0,19	0,7	0,16	0,2
1,2,3,6,7,8-HxCDF	0,15	0,17	0,7	0,13	0,6
2,3,4,6,7,8-HxCDF	0,16	0,15	0,2	0,18	0,5
1,2,3,7,8,9-HxCDF	(0,0712)*	0,0711	*	0,0750	*
1,2,3,4,6,7,8-HpCDF	0,606	0,579	0,2	0,525	0,7
1,2,3,4,7,8,9-HpCDF	(0,06)*	0,065	*	0,040	*
1,2,3,4,6,7,8,9-OCDF	0,24	0,21	0,6	0,18	1,3
2,3,7,8-TCDD	0,129	0,156	1,0	0,105	0,9
1,2,3,7,8-PeCDD	0,23	0,19	0,9	0,24	0,1
1,2,3,4,7,8-HxCDD	0,155	0,162	0,2	0,141	0,5
1,2,3,6,7,8-HxCDD	0,254	0,209	0,9	0,180	1,5
1,2,3,7,8,9-HxCDD	0,619	0,720	0,8	0,586	0,3
1,2,3,4,6,7,8-HpCDD	3,95	3,98	0,0	3,04	1,2
1,2,3,4,6,7,8,9-OCDD	194	232	1,0	156	1,0
PCB 28	338	366	0,4	328	0,2
PCB 52	278	252	0,5	324	0,8
PCB 77	17,8	17,4	0,1	16,2	0,4
PCB 81	0,843	0,755	0,5	0,916	0,4
PCB 101	375	272	1,4	442	0,9
PCB 123	7,26	6,52	0,5	8,41	0,8
PCB 118	392	384	0,1	405	0,2
PCB 114	9,22	11,0	0,9	7,5	0,9
PCB 105	151	157	0,2	152	0,0
PCB 126	3,16	3,23	0,1	3,14	0,0
PCB 153	784	810	0,2	823	0,3
PCB 138	541	567	0,2	596	0,5
PCB 167	23,8	24,4	0,1	27,3	0,7
PCB 156	38,9	42,9	0,5	39,8	0,1
PCB 157	10,8	8,2	1,2	11,0	0,1
PCB 169	0,751	0,785	0,2	0,641	0,7
PCB 180	217	257	0,9	203	0,3
PCB 189	4,21	4,03	0,2	4,35	0,2

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

Conclusion

In summary, the results prove excellent clean-up with very good recoveries for the 13C standards and fulfill the criteria of European and US FDA regulations for the PCDD/F and PCB analysis. The PFAD results with the new DCM free Alox Plus method are in good agreement with the proficiency test result as well as with the results of the classical DCM Alox Plus method.