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 derivates 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.

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 RTXDioxin2 capillary column from Restek.

**Matrices that have been Tested**

- PFAD: Palm Fatty Acid Destillates
- 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](image)

![Figure 2: 13C- PCB recoveries](image)

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

<table>
<thead>
<tr>
<th>PFAD</th>
<th>DEXTech Heat</th>
<th>Assigned Value</th>
<th>z-score</th>
</tr>
</thead>
<tbody>
<tr>
<td>2,3,7,8-TCDF</td>
<td>0.587</td>
<td>0.565</td>
<td>0.2</td>
</tr>
<tr>
<td>1,2,3,7,8-PeCDF</td>
<td>0.260</td>
<td>0.227</td>
<td>0.7</td>
</tr>
<tr>
<td>2,3,4,7,8-PeCDF</td>
<td>0.387</td>
<td>0.324</td>
<td>1.0</td>
</tr>
<tr>
<td>1,2,3,6,7,8-HxCDF</td>
<td>0.19</td>
<td>0.17</td>
<td>0.7</td>
</tr>
<tr>
<td>2,3,4,6,7,8-HxCDF</td>
<td>0.15</td>
<td>0.16</td>
<td>0.2</td>
</tr>
<tr>
<td>1,2,3,7,8,9-HxCDF</td>
<td>0.0711</td>
<td>(0.0712)</td>
<td>*</td>
</tr>
<tr>
<td>1,2,3,4,6,7,8-HpCDF</td>
<td>0.579</td>
<td>0.606</td>
<td>0.2</td>
</tr>
<tr>
<td>1,2,3,3,7,8,9-HpCDF</td>
<td>0.065</td>
<td>(0.06)</td>
<td>*</td>
</tr>
<tr>
<td>1,2,3,7,8,9,9-OCD</td>
<td>0.21</td>
<td>0.24</td>
<td>0.6</td>
</tr>
<tr>
<td>2,3,7,8-TCDD</td>
<td>0.156</td>
<td>0.129</td>
<td>1.0</td>
</tr>
<tr>
<td>1,2,3,7,8-PeCDD</td>
<td>0.19</td>
<td>0.23</td>
<td>0.9</td>
</tr>
<tr>
<td>1,2,3,7,8,9-HxCDD</td>
<td>0.162</td>
<td>0.155</td>
<td>0.2</td>
</tr>
<tr>
<td>1,2,3,6,7,8-HxCDD</td>
<td>0.209</td>
<td>0.254</td>
<td>0.9</td>
</tr>
<tr>
<td>1,2,3,4,6,7,8-HxCDD</td>
<td>0.720</td>
<td>0.619</td>
<td>0.8</td>
</tr>
<tr>
<td>1,2,3,6,7,8,9-HxCDD</td>
<td>3.98</td>
<td>3.95</td>
<td>0.0</td>
</tr>
<tr>
<td>1,2,3,4,6,7,8,9-OCD</td>
<td>232</td>
<td>194</td>
<td>1.0</td>
</tr>
</tbody>
</table>

PCB 28 366 338 0.4
PCB 52 252 278 0.5
PCB 77 17.4 17.8 0.1
PCB 81 0.755 0.843 0.5
PCB 101 272 375 1.4
PCB 123 6.52 7.26 0.5
PCB 118 384 392 0.1
PCB 114 11.0 9.22 0.9
PCB 126 523 516 0.1
PCB 153 810 784 0.2
PCB 138 567 541 0.2
PCB 167 24.4 23.8 0.1
PCB 156 42.9 43.9 0.5
PCB 156 8.2 10.8 1.2
PCB 169 0.785 0.751 0.2
PCB 180 257 217 0.9
PCB 189 4.03 4.21 0.2

* 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

1. DEXTech Heat
2. Rotational-vacuum-concentrator D-EVAporation
3. Rotor with 48 positions
   - Angle rotor, 48 positions
   - Sensor for 15 mL vial
4. Rotor with 24 positions
   - Angle rotor, 24 positions
   - Sensor for 24 mL vial
5. Universal acidic silica gel column
6. Alumina column
7. Carbon column

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