



Dioxin and PCB determination in foodstuffs

SpeedExtractor E-914

Pressurized solvent extraction for the determination of dioxins and PCBs in dairy, egg, pork and fish

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The determination of dioxins and PCBs in foodstuffs is performed to monitor the potential exposure to humans. A reliable and fast method is introduced below. Proficiency test samples were extracted with the SpeedExtractor E-914, followed by an automated clean-up using a MIURA system and analyzed by GC-HRMS. The determined levels of dioxins, furans, dioxin-like and non-dioxin-like PCBs correspond to the assigned values.

1. Introduction

"Dioxins" is the umbrella term for polychlorinated dibenzop-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs). PCB's have been produced in large volumes for industrial applications. Some PCBs are classified as "dioxin-like" and they should be considered in the overall dioxin toxicity. Dioxins and PCBs are lipophilic and accumulate in human and animal tissues. Therefore, high fat foods from animals such as meat, eggs, milk and derived products are at a greater risk of contamination.

Both, dioxins and PCBs are persistant organic polluants and as such are banned by the Stockholm convention.

2. Experimental

Equipment: SpeedExtractor E-914, Rotavapor[®], MIURA GO-HT system, GC-HRMS

Samples: Milk powder, milk fat, egg powder, pork and herring. All samples were from EU-RL and Norwegian Institute of Public Health proficiency tests.

Determination: The samples were freeze-dried, then added directly into the extraction cells. The pressurized solvent extraction was done with the E-914, using the parameters specified in Table 1. The extraction of milk powder was done following the parameters given in the EN method [1].

Table 1: Extraction method SpeedExtractor E-914 for milk fat, egg powder, pork and herring.

Parameter	Value
Temperature	120 °C
Pressure	100 bar
Solvent	70 % Toluene: 30 % Acetone
Cells	40 mL
Vials	250 mL round bottom flasks
Cycles	3
Heat-up	4 min / 1 min / 1 min
Hold	5 min
Discharge	3 min
Flush with solvent	0 min
Flush with gas	5 min
Total extraction time	53 min

After extraction, the extracts were evaporated to dryness using a Rotavapor[®] and the fat content was calculated.

The extract was then redissolved and cleaned-up using an automated clean-up instrument (GO-HT4, MIURA). After clean-up, the extracts were analyzed for dioxins, dioxin-like PCBs and non-dioxin-like PCBs using GC-HRMS (Agilent 7980A coupled to JEOL 800D).

3. Results

The determined WHO-TEQ 2005 values for the sum of the dioxins and the dioxin-like PCBs are shown in Figure 1.

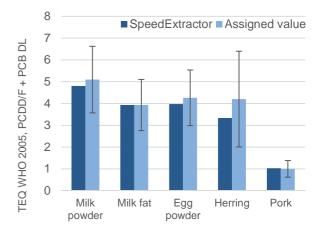


Figure 1: Determined TEQ for the sum of the dioxins and dioxinlike PCBs in proficiency test samples compared to the assigned values in [pg/g fat] for all samples except herring, where it is [pg/g fresh weight]. The error bars correspond to the concentration range with a z-score between +2 and -2. Mean values, n=4.

In all samples, 17 dioxin and furan congeners, 12 dioxinlike PCBs and 6 non-dioxin like PCBs were quantified. Zscores associated with the determined values were < 2 (data shown in the application note).

4. Conclusion

The determined WHO-TEQ values were all within an absolute value with a z-score of < 1. The presented procedure for dioxin determination using Pressurized Solvent Extraction with the SpeedExtractor E-914 is a fast, reliable method for the determination of dioxins and PCBs.

5. References

[1] EN 16215:2012 Animal feeding stuffs - Determination of dioxins and dioxin-like PCBs by GC-HRMS and of indicator PCBs by GC/HRMS.

For more detailed information and safety considerations please refer to the Application Note No. 205/2015.

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