Application Note

Dioxins, Furans, and PCBs in Peanut Butter Samples Processed with Automated Extraction and Clean Up

Introduction

Polychlorinated dibenzo-p-dioxins (PCDDs), furans (PCDFs) and biphenyls (PCBs) are a group of highly toxic compounds. Due to their lipophilic nature, these analytes bioaccumulate in adipose tissue and end up in food supplies, such as fish, meats, oils, and poultry. For this reason, the U.S. FDA and EU have established strict regulations for the monitoring of food products for human consumption.

Routine analysis of these compounds uses US EPA methods 1613 and 1668. Traditionally sample processing has involved multi-day Soxhlet extraction and manual sample clean up using column chromatography. As an alternative to obtain faster and more reliable data, these various steps have been automated. This application note describes the automated Pressurized Liquid Extraction (PLE) and automated open column chromatography clean up (PowerPrep) of peanut butter.

Instrumentation

- FMS, Inc. PLE®
- FMS, Inc. PowerPrep®
- FMS, Inc. SuperVap® 6 Concentrator
- FMS, Inc. SuperVap® Vial Concentrator
- FMS, Inc. 250 mL concentrator tubes (1 mL termination)
- Thermo Trace GC Ultra with high res magnetic sector DFS Thermo mass spec

Consumables

- FMS, Inc. Jumbo Acidified Silica column
- FMS, Inc. Classical Acid-Base-Neutral column
- FMS, Inc. Basic Alumina column
- FMS, Inc. Carbon-Celite column
- Fisher Optima® Dichloromethane
- Fisher Optima® Hexane
- Fisher Optima ® Toluene
- CIL EDF-8999 Method 1613 ^{13}C PCDD/F Stock Solution
- CIL EDF-5999 ^{13}C PCDD/F Recovery Standard
- CIL EC-4995 ^{13}C PCB Internal Isotope Dilution Standard who-12 PCB and 170/180
- CIL EO-5275 ^{13}C PCB Recovery Standard

PLE

- 5 g of sample mixed with 10 g inert Hydro-matrix® and spiked with surrogates
- Sample placed in extraction cell
- Capped with disposable Teflon end caps
- Heated with 50% Dichloromethane/50% Hexane for 20 min at 120 ^{o}C and 1500 psi
- 20 min cool down
- Nitrogen flush to transfer analytes and extract to 250 mL collection tubes

SuperVap Concentration

- Pre-heat temperature: 45 ^{o}C
- Pre-heat time: 15 min
- Heat in Sensor mode: 45 ^{o}C
- Nitrogen Pressure: 6-8 psi
- Solvent exchange to hexane

PowerPrep Clean Up

- Modified 25-step program
- Install jumbo silica, classical ABN, alumina and carbon/celite columns
- Mixes used are hexane, 10%/90% dichloromethane/hexane, dichloro-methane, and toluene

FMS
Fluid Management Systems

■ Run conditioning steps with columns in place
■ Load sample (in hexane)
■ Elute silica with 150 mLs hexane (waste)
■ Elute alumina with 70 mLs 10%/90% DCM/ hexane (collect as F1)
■ Elute alumina onto carbon with 120 mLs dichloromethane (waste)
■ Elute carbon with 75 mLs toluene (collect as F2)
■ All PCBs are eluted in F1 and all PCDD/Fs in F2

SuperVap step (above)

Vial Evaporator
■ Reduce sample to 10 uL final volume under 1-1.5 psi nitrogen at 25 ^{o}C

Table with native peanut butter values and ^{13}C-labeled recoveries.

	native pg/g	**recoveries %**
2378-T4CDF	< 0.10	80%
2378-T4CDD	< 0.10	89%
12378-P5CDF	< 0.50	92%
23478-P5CDF	< 0.50	78%
12378-P5CDD	< 0.50	83%
123478-H6CDF	< 0.50	84%
123678-H6CDF	< 0.50	75%
234678-H6CDF	< 0.50	69%
123789-H6CDF	< 0.50	86%
123478-H6CDD	< 0.50	88%
123678-H6CDD	< 0.50	72%
123789-H6CDD	< 0.50	
1234678-H7CDF	< 0.50	78%
1234789-H7CDF	< 0.50	96%
1234678-H7CDD	< 0.50	82%
OCDF	< 1.00	
OCDD	< 1.00	93%

Table with native peanut butter values and ^{13}C-labeled recoveries.

		native pg/g	recoveries %
33'44'-T4CB	77	3.13	72%
344'5-T4CB	81	< 0.40	73%
233'44'-P5CB	105	3.25	68%
2344'5-P5CB	114	< 0.40	71%
23'44'5-P5CB	118	6.73	67%
2'344'5-P5CB	123	< 0.40	67%
33'44'5-P5CB	126	< 0.40	76%
233'44'5-H6CB	156	0.48	65%
233'44'5'-H6CB	157	0.15	59%
23'44'55'-H6CB	167	1.21	65%
33'44'55'-H6CB	169	< 0.40	69%
233'44'55'-H7CB	170	< 0.40	59%
22'344'55'-H7CB	180	0.96	58%
233'44'55'-H7CB	189	< 0.40	64%

Conclusions

No PCDD/Fs were found in the peanut butter with detection limits reported in the Table. PCBs concentrations were also low. Excellent recoveries of the labeled ^{13}C isotope dilutions standards were seen. The results show the versatility of the automated method. With extraction times of ~ 60 min and sample clean up taking only a few hours, same-day analysis of peanut butter and other food stuffs is now possible.

PowerPrep, PLE, and Concentrator

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