

Dioxins, Furans, and PCBs in Peanut Butter Samples Processed with Automated Extraction and Clean Up via EPA Methods 1613 and 1668C

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 1668C. 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. High Capacity Acidified Silica column
- FMS, Inc. Basic Alumina column
- FMS, Inc. Carbon-Celite column
- Fisher Optima[®] Dichloromethane
- Fisher Optima[®] Hexane

- Fisher Optima[®] Toluene
- 1613 and 1668C spiking and recovery standards

PLE

- 5 g of sample mixed with 10 g inert Hydromatrix[®] 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 °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: 55 °C
- Pre-heat time: 15 min
- Heat in Sensor mode: 55 °C
- Nitrogen Pressure: 6-8 psi
- Solvent exchange to hexane

PowerPrep Clean Up

- Standard program
- Install high capacity acidic silica, alumina and carbon/celite columns
- Solvents used are hexane, dichloromethane and toluene
- Condition columns with hexane (60 mL)
- Load sample
- Elute silica/alumina with 160 mL hexane
- Elute alumina/carbon with 70 mL dichloromethane (collect mono- and di-ortho PCBs)
- Elute carbon in reverse direction with 60 mLs toluene (collect PCDD/Fs)



SuperVap step (above)

Vial Evaporator

- Reduce sample to 10 uL final volume under 1-1.5 psi nitrogen at 25 °C

Table 1 with native peanut butter values and ¹³C-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 2 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 SuperVap[®]

For more information contact FMS:
FMS, Inc.

580 Pleasant Street
Watertown, MA 02472

Phone: (617) 393-2396

Fax: (617) 393-0194

Email: onlineinfo@fms-inc.com

Web site: www.fms-inc.com