

Automated Extraction and Clean Up of Egg Yolk: Dioxins and Furans via EPA Method 1613

Introduction

Polychlorinated dibenzo-p-dioxins (PCDDs) and furans (PCDFs) have been reported in eggs and it is estimated that about 4% of daily PCDD/F intake in human diet is because of egg consumption. Because of its lipid content the PCDD/Fs are mostly concentrated in the egg yolk. The seventeen laterally chlorinated dioxins and furans are generally considered the most toxic. Analysis of eggs is therefore important because of public health considerations. US EPA method 1613 is the standard method for these analyses.

Traditionally processing of materials such as eggs for chlorinated compounds 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 column chromatography clean up (PowerPrep[®]) of eggs.

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 ¹³C₁₂ spiking and recovery standards

PLE

- 17 g of egg yolk 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: 7-10 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
- 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 egg yolk values and ^{13}C -labeled recoveries.

	native pg/g	recoveries %
2378-T4CDF	0.04	83%
2378-T4CDD	0.01	86%
12378-P5CDF	nd	93%
23478-P5CDF	nd	91%
12378-P5CDD	0.08	96%
123478-H6CDF	nd	75%
123678-H6CDF	nd	73%
234678-H6CDF	nd	71%
123789-H6CDF	nd	81%
123478-H6CDD	0.02	78%
123678-H6CDD	0.07	75%
123789-H6CDD	nd	
1234678-H7CDF	nd	82%
1234789-H7CDF	nd	89%
1234678-H7CDD	0.14	88%
OCDF	0.04	
OCDD	0.42	93%



Conclusions

Native PCDD/Fs levels were very low and excellent recoveries of the ^{13}C isotope dilution standards were observed. Egg yolks are easily and reliably processed with our combined automated extraction and clean up equipment. With extraction times of ~ 60 min and sample clean up taking only a few hours, same-day analysis of egg samples is now possible.



PowerPrep[®], PLE[®], and SuperVap[®]
Concentrator

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