Application Note

Automated Extraction and Clean Up of Crab Meat: Dioxins and Furans Using FMS, Inc. PowerPrep® and PLE® via EPA Method 1613

Introduction

Polychlorinated dibenzo-p-dioxins (PCDDs) and furans (PCDFs) have been reported in crab meat from around the world, including in crabs from the St Louis Bay and China. The seventeen laterally chlorinated dioxins and furans are generally considered the most toxic. Analysis of crab meat is therefore important because of public health considerations. US EPA method 1613 is the standard for these analyses.

Traditionally processing of materials such as crab meat 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 open column chromatography clean up (PowerPrep®) of crab meat.

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
- Method 1613 $^{13}C_{12}$ spiking standards

PLE®

- 10 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: 55 ^{o}C
- Pre-heat time: 15 min
- Heat in Sensor mode: 55 ^{o}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
- Uses hexane, dichloromethane and toluene
- Condition columns with hexane (60 mL)
- Load sample
- Elute silica/alumina with 160 mL hexane
- Elute alumina with 70 mL dichloromethane

FMS
Fluid Management Systems

■ 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 ^{o}C

Table with native crab meat values and ^{13}C-labeled recoveries.

	native pg/g	recoveries %
2378-T4CDF	0.05	89%
2378-T4CDD	nd	92%
12378-P5CDF	0.13	94%
23478-P5CDF	0.10	92%
12378-P5CDD	0.13	95%
123478-H6CDF	0.09	92%
123678-H6CDF	0.09	90%
234678-H6CDF	0.08	87%
123789-H6CDF	0.10	93%
123478-H6CDD	0.06	94%
123678-H6CDD	0.08	92%
123789-H6CDD	0.08	
1234678-H7CDF	nd	94%
1234789-H7CDF	nd	95%
1234678-H7CDD	0.09	94%
OCDF	0.06	
OCDD	0.35	93%

Conclusions

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

PowerPrep®, PLE®, and SuperVap® Concentrator

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