

Automated Extraction and Clean Up of Crab Meat: Dioxins and Furans

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. Jumbo Acidified Silica column
- FMS, Inc. Classical Acid-Base-Neutral column
- FMS, Inc. Basic Alumina column
- FMS, Inc. Carbon-Celite column
- Millipore OmniSolv® Benzene
- Fisher Optima® Dichloromethane

- Fisher Optima® Ethylacetate
- Fisher Optima® Hexane
- Fisher Optima® Toluene
- CIL EDF-8999 Method 1613 ¹³C PCDD/F Stock Solution
- CIL EDF-5999 ¹³C PCDD/F Recovery Standard

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 °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 °C
- Pre-heat time: 15 min
- Heat in Sensor mode: 45 °C
- Nitrogen Pressure: 6-8 psi
- Solvent exchange to hexane

PowerPrep Clean Up

- Standard 25-step program
- Install jumbo silica, classical ABN, alumina and carbon/celite columns
- Mixes used are hexane, 2%/98% dichloromethane/hexane, 50%/50% dichloromethane/hexane, 50%/50% ethylacetate/benzene, and toluene



- Run conditioning steps 1-13 with columns in place
- Load sample (in hexane)
- Elute silica with 150 mLs hexane (waste)
- Elute alumina with 60 mLs 2%/98% DCM/hexane (waste)
- Elute alumina with 120 mLs 50%/50% DCM/hexane (waste)
- Elute carbon with 4 mL 50%/50% ethyl-acetate/benzene (waste)
- Elute carbon with 75 mLs toluene (collect as PCDD/F fraction)

SuperVap step (above)

Vial Evaporator

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

Table with native crab meat values and ¹³C-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 Concentrator

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