

Analyzing Infant Formula for Dioxins and Furans using Pressurized Liquid Extraction and Automated Clean Up

Introduction

Because of important public health considerations for children, infant formula has been tested for Persistent Organic Pollutants (POPs) such as polychlorinated dibenzo-p-dioxins (PCDDs) and furans (PCDFs). Typically concentrations of these analytes found are lower than in human breast milk but continuous monitoring is important. This requires well equipped laboratories capable of trace level analysis work, as often PCDD/Fs amounts are at the picogram or femtogram level.

US EPA method 1613 is mostly used around the world for these analyses. Manual Soxhlet extraction and subsequent manual sample clean up using column chromatography can take a number of days and result in contamination and unreliable data. 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 infant formula.

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® Hexane

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

PLE

- 1 g of infant formula was 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 infant formula values and ¹³C-labeled recoveries.

	native pg/g	recoveries %
2378-T4CDF	nd	93%
2378-T4CDD	0.20	93%
12378-P5CDF	0.55	97%
23478-P5CDF	0.47	97%
12378-P5CDD	0.65	102%
123478-H6CDF	0.50	83%
123678-H6CDF	0.57	80%
234678-H6CDF	nd	82%
123789-H6CDF	nd	93%
123478-H6CDD	0.48	86%
123678-H6CDD	nd	84%
123789-H6CDD	nd	
1234678-H7CDF	nd	88%
1234789-H7CDF	nd	96%
1234678-H7CDD	1.58	93%
OCDF	1.17	
OCDD	75.93	85%



Conclusions

PCDD/F concentrations found in the infant formula were low with OCDD as the main analyte found. Excellent recoveries of the ^{13}C labeled internal standards were obtained between 80-100%. For ongoing monitoring of infant formula our combined PLE-PowerPrep sample processing method is very suitable, assuring both high quality reliable data and same-day analysis.



PowerPrep, PLE, and Concentrator

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