Application Note

The Analysis of Polychlorinated Dibenzo-p-dioxins and Furans in Cranberry Pulp with Automated Extraction and Reduced Solvent Volume Clean Up

Introduction

Persistent organic pollutants (POPs) such as polychlorinated dibenzo-p-dioxins (PCDD/Fs) have been a major environmental concern for a number of decades. Also their presence in various foods has been of concern. This includes analysis of beverages for these toxic compounds. Routine analysis of PCDD/Fs follows US EPA method 1613.

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 cranberry pulp.

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 Acid-Base-Neutral Silica column
- FMS, Inc. Basic Alumina column
- FMS, Inc. Carbon/Celite column
- Fisher Optima® Dichloromethane
- Fisher Optima® Hexane
- Fisher Optima® Toluene
- Cambridge Isotope Labs (CIL) EDF-8999 Method 1613 ^{13}C PCDD/F Stock Solution
- CIL EDF-5999 ^{13}C PCDD/F Recovery Standard

PLE

- 10 g of cranberry pulp mixed with 10 g inert Hydro-matrix®
- 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: 45 ^{o}C
- Pre-heat time: 15 min
- Heat in Sensor mode: 45 ^{o}C
- Nitrogen Pressure: 6-8 psi
- Solvent exchange to hexane

PowerPrep Clean Up

- Reduced solvent volume 12-step program
- Install high capacity acid-base-neutral silica, alumina and carbon/celite columns
- Mixes used are hexane, 10%/90% dichloromethane/hexane, dichloromethane and toluene
- Spike extract from PLE with ^{13}C surrogates. Labeled recoveries over Power Prep clean up step were studied here. In most cases sample would be spiked prior to PLE extraction

FMS
Fluid Management Systems

■ Condition high capacity acid-base-neutral, alumina and carbon/celite columns with hexane
■ Load sample (in hexane)
■ Elute high capacity silica with 140 mLs hexane (waste)
■ Elute alumina with 70 mLs 10%/90% DCM/ hexane (pcb fraction goes to waste)
■ Elute alumina with 50 mLs dichloromethane to bring PCDD/Fs onto carbon/celite
■ Elute carbon/celite with 35 mLs toluene (collect as PCDD/F fraction)
■ Total volume used is 400 mLs

SuperVap step (above)

Vial Evaporator
■ Reduce sample to 10 uL final volume under 1-1.5 psi nitrogen at 25 ^{o}C

Table with ^{13}C PCD/Fs recoveries for cranberry pulp using Power Prep cleanup program

	recoveries %
2378-T4CDF	65%
2378-T4CDD	69%
12378-P5CDF	74%
23478-P5CDF	71%
12378-P5CDD	73%
123478-H6CDF	83%
123678-H6CDF	84%
234678-H6CDF	84%
123789-H6CDF	77%
123478-H6CDD	84%
123678-H6CDD	81%
1234678-H7CDF	81%
1234789-H7CDF	86%
OCDD	57%

Conclusions

As can be seen the analysis of cranberry pulp showed very good recoveries of the labeled PCDD/Fs standards across the PowerPrep cleanup step of the sample processing. With the new reduced solvent volume program only 400 mL of solvent is needed per sample for successful PCDD/Fs analysis.

Extraction, clean up and analysis by properly trained personnel can be carried out in one day, resulting in low turnaround times for large (and small) sample batches.

PowerPrep, PLE, and Concentrator

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