Application Note

The Analysis of Polychlorinated Dibenzo-p-dioxins and Furans in Cranberry Pulp with Automated Extraction and Reduced Solvent Volume Cleanup via EPA Method 1613

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 Acidic Silica column
- FMS, Inc. Basic Alumina column
- FMS, Inc. Carbon/Celite column
- Fisher Optima® Dichloromethane
- Fisher Optima® Hexane
- Fisher Optima® Toluene
- 1613 spiking and recovery standards

PLE®

- 10 g of cranberry pulp mixed with 10 g inert Hydromatrix®
- 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: 6-8 psi
- Solvent exchange to hexane

PowerPrep® Clean Up

- Reduced solvent volume 12-step program
- Install high capacity acidic silica, alumina and carbon/celite columns
- Solvents used are 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
- 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)

FMS
Fluid Management Systems

■ **SuperVap step** (above)

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

Table 1 with ^{13}C PCD/Fs recoveries for cranberry pulp using PowerPrep® 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 300 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 SuperVap® Concentrator

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