

# The Analysis of Polychlorinated Biphenyls in Coffee using Automated Extraction and Reduced Solvent Volume Clean Up via EPA Method 1668C

## Introduction

Persistent organic pollutants (POPs) such as polychlorinated biphenyls (PCBs) have been a major environmental and food product concern for a number of decades. Hence, the US FDA has carried out monitoring of various food stuffs for PCBs. Routine analysis of PCBs follows US EPA method 1668C.

Traditionally sample processing has involved multi-day Soxhlet extraction and manual 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<sup>®</sup>) and automated open column chromatography clean up (PowerPrep<sup>®</sup>) of ground coffee beans.

## Instrumentation

- FMS, Inc. PLE<sup>®</sup>
- FMS, Inc. PowerPrep<sup>®</sup>
- FMS, Inc. SuperVap<sup>®</sup> 6 Concentrator
- FMS, Inc. SuperVap<sup>®</sup> 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
- Fisher Optima<sup>®</sup> Dichloromethane
- Fisher Optima<sup>®</sup> Hexane

- 1668C spiking and recovery standards

## PLE

- 10 g of ground coffee mixed with 10 g inert Hydromatrix<sup>®</sup>
- 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: 55 °C
- Pre-heat time: 15 min
- Heat in Sensor mode: 55 °C
- Nitrogen Pressure: 6-8 psi
- Solvent exchange to hexane

## PowerPrep Clean Up

- Reduced solvent volume 7-step program
- Install high capacity acidic silica and alumina columns
- Mixes used are hexane and 10%/90% dichloromethane/hexane
- Spike extract from PLE with <sup>13</sup>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 high capacity acidic and alumina columns with 60 mLs hexane
- Load sample (in hexane)
- Elute high capacity silica with 160 mLs hexane (waste)
- Elute alumina with 70 mLs 10%/90% DCM/hexane (collect as pcb fraction)
- Total volume used is 300 mLs

**SuperVap step** (above)

**Vial Evaporator**

- Reduce sample to 10  $\mu$ L final volume under 1-1.5 psi nitrogen at 25  $^{\circ}$ C

*Table 1 with  $^{13}$ C PCB recoveries for coffee using Power Prep cleanup program*

|                 |     | recoveries<br>% |
|-----------------|-----|-----------------|
| 33'44'-T4CB     | 77  | 77%             |
| 344'5-T4CB      | 81  | 79%             |
| 233'44'-P5CB    | 105 | 71%             |
| 2344'5-P5CB     | 114 | 76%             |
| 23'44'5-P5CB    | 118 | 74%             |
| 2'344'5-P5CB    | 123 | 73%             |
| 33'44'5-P5CB    | 126 | 81%             |
| 233'44'5-H6CB   | 156 | 75%             |
| 233'44'5'-H6CB  | 157 | 76%             |
| 23'44'55'-H6CB  | 167 | 74%             |
| 33'44'55'-H6CB  | 169 | 82%             |
| 233'44'55'-H7CB | 170 | 71%             |
| 22'344'55'-H7CB | 180 | 75%             |
| 233'44'55'-H7CB | 189 | 78%             |



### Conclusions

As can be seen the analysis of ground coffee beans showed very good recoveries of the labeled  $^{13}\text{C}$  PCB standards across the Power Prep clean up step of the sample processing. With the new reduced solvent volume program only 300 mL of solvent is needed per sample for successful PCB 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<sup>®</sup>, PLE<sup>®</sup>, and SuperVap<sup>®</sup>  
Concentrator

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