

One Step Extraction, Cleanup and Concentration of Chlorinated Pesticides in Raw Coffee Beans



Introduction

Organochlorine pesticides are man-made chemicals once used widely in agriculture and for mosquito control. Beginning in the 1960's, public awareness of their toxicity began to increase and by the mid 1970's many organochlorine pesticides were banned in the United States. Though some are still registered for use in the United States, today they are used mainly in developing nations.

Classified as persistent organic pollutants, organochlorine pesticides exhibit a wide range of toxic effects including reproductive and neurotoxicity as well as liver and kidney failure. In animals, they have been shown to cause cancer in these organs.

Due to their persistent nature and toxicity, monitoring food sources for the presence of organochlorine pesticides is a growing concern for both food manufacturers and consumers. Foods are typically difficult matrices to extract and analyze. The following procedure describes a rapid and efficient method for the extraction, extract cleanup and concentration of organochlorine pesticides from raw coffee beans.

Instrumentation and Consumables

- FMS, Inc. Pressurized Liquid Extractor (PLE)
- FMS, Inc. SuperVap™ Direct-to-vial Concentration system
- FMS, Inc. direct-to-vial concentrator tubes
- 6 gram InCell Florisil® PLE end cap columns
- Agilent 7890A GC with μ ECD

PLE Program

1. The Cells are filled with hexane
2. Cells pressurized to 1500 PSI
3. Cells heated to 120 °C
4. 120 °C held for 10 minutes
5. Cells cooled and depressurized.
6. The Cells are flushed with 80% of the cell volume hexane.
7. The remaining solvent is purged out of the cells using N₂ directly to concentration tubes

SuperVap Concentrator

1. Pre-heat temp: 40 °C
2. Pre-heat time: 30 minutes
3. Heat in Sensor mode: 40 °C
4. Nitrogen Pressure: 15 PSI
5. Sensor End Point Detection

Procedure: Sample Prep and Extraction

The green coffee beans were finely processed until no visible clumps remained. The coffee bean sample was weighed out in seven replicates of five grams per sample.

The sample portions were then mixed with Varian Hydro Matrix.

10 gram samples were transferred to 100 mL cells equipped with 6 gram Florisil® InCell cleanup end cap columns.

Five samples were spiked, each with 0.1 μ g/mL of surrogate spiking solution and pesticide spike. Two sample portions were only spiked with a surrogate solution to establish any background levels in the sample matrix.

The Cells were capped and loaded onto PLE. The extraction is initiated.

The extracts automatically transfer to the SuperVap concentrator and concentration begins.

The Extract is removed from the SuperVap Concentrator with Direct-to-Vial tubes and transferred to Agilent GC for analysis.



Figure 1: FMS Pressurized Liquid Extractor and SuperVap systems.



Results

Table 1: Mean recoveries and standard deviations from the five extracts after analysis.

Compound	AVG Rec	STD
TCMX	104.8%	4.6
Alpha-BHC	92.0%	1.5
Beta-BHC	89.8%	1.1
Gamma-BHC	89.2%	1.6
Delta-BHC	109.5%	2.7
Heptachlor	87.0%	4.8
Aldrin	95.8%	15.6
Heptachlor Epoxide	81.8%	1.3
Gamma-Chlordane	76.7%	1.5
Endosulfan I	79.9%	1.2
Alpha-Chlordane	78.7%	1.6
Dieldrin	75.4%	1
4,4"-DDE	74.7%	1.5
Endrin	135.0%	3.2
Endosulfan II	81.2%	1.5
4,4'-DDD	92.1%	9.4
Endrin Aldehyde	77.1%	1.8
Endosulfan Sulfate	98.8%	2.5
4,4'-DDT	83.4%	23.9
Endrin Ketone	80.8%	4.6
Methoxychlor	91.2%	17.9
Deca-PCB	93.6%	10.4

Conclusions

Analysis of the PLE data on the GC demonstrates excellent reproducibility, consistent recoveries and from a traditionally difficult sample matrix. Adding to the efficiency of the entire sample prep process was the use of the InCell extraction cleanup end cap column. The InCell column eliminates the need to perform post cleanup and a second concentration step. Eliminating the second concentration step dramatically reduces the risk of lower analyte recovery of volatile compounds. With the addition of the Direct-to-Vial concentration tube, no sample transfer is necessary. This allowed the sample to be extracted and cleaned up by the PLE system and then sent automatically to the SuperVap™ Concentrator system where the final extract is concentrated directly to a vial for final GC analysis.

For more information contact FMS:
 FMS Inc.
 580 Pleasant Street
 Watertown, MA 02472
 Phone: 617-393-2396
 Fax: 617-393-0194
 Email: onlineinfo@fms-inc.com
 Web site: fmsenvironmental.com