Application Note

A Quick Screen Method for the Extraction, Cleanup and Concentration of Toxaphene in Fish via EPA Method 8081b

Introduction

Prior to it being banned in the U. S. in 1986, toxaphene was widely used throughout the southeastern United States as a pesticide on cotton and soybean crops. Classified as a group 2B carcinogen, it affects the lungs, nervous system and kidneys. Sufficient exposure can be fatal. Despite these dangers, toxaphene is still used today for treating crops outside the United States.

Toxaphene exists as a mixture of roughly 200 organic compounds formed by the chlorination of camphene, which results in a chlorine content of approximately 70%. Because of its persistent nature and the ease with which it enters biological organisms, ingestion of toxaphene through human food sources is a continuing concern since its ban.

Due to a complex and lengthy sample clean-up process, the extraction and analysis of toxaphene via EPA Method 8081b can be a daunting process for laboratories. The purpose of this study was to develop a one-step toxaphene extraction and clean-up process from fish tissue that delivers reproducible results.

Instrumentation

- FMS, Inc. PLE® (Pressurized Liquid Extractor) system
- FMS, Inc. SuperVap® Concentrator system
- FMS, Direct-to-Vial concentrator tubes
- 2.5 and 5 gram InCell acid (30%) silica gel PLE end caps
- Agilent 7890A GC with µECD

PLE Program

1. Cells are filled with 2:1 Cyclohexane/Pentane
2. Cells are pressurized to 1500 PSI
3. Cells are heated to 120 ºC and held for 15 minutes
4. Cells are cooled and depressurized.
5. Cells are flushed with 80% of cell volume.
6. The remaining solvent purged out of cells with N_2

SuperVap Concentrator

1. Pre-heat temp: 40 ºC
2. Pre-heat time: 15 minutes
3. Heat in Sensor mode: 40 ºC
4. Nitrogen Pressure: 10 PSI

Procedure

Sample Preparation and Extraction

Salmon tissue was ground up until no visible clumps remained.

It was then weighed out in duplicate portions of both 2.5 gram samples and 5 gram samples.

The sample portions were then mixed with Hydromatrix®.

2.5 gram samples were transferred to 40 mL PLE extraction cells equipped with 2.5 gram InCell acidified silica gel end caps. 5 gram samples with transferred to 100 mL Cells equipped with 6 gram acidified silica gel end caps.

The samples are spiked with .1 µg/mL surrogate spiking solution and 4 µg/mL toxaphene spiking solution. Two sample portions were left un-spiked with toxaphene to establish background for sample matrix.

The Cells were capped and loaded onto PLE system. Extraction program initiated.

Extracts automatically transferred to Power Vap system and concentration begun.

Extract removed from SuperVap® system (1mL) and transferred to Agilent GC for analysis.

FMS, Inc. PLE® and SuperVap® Concentrator Systems

FMS
Fluid Management Systems

Results

Table 1: Results of 2.5 gram and 5 gram samples

Compound	2.5 gram Avg. Conc.	Sample % rec.
TCMX	27.8 µg/kg	70%
Decachlorobiphenyl	34.8 µg/kg	87%
Toxaphenne	121.2 µg/kg	76%

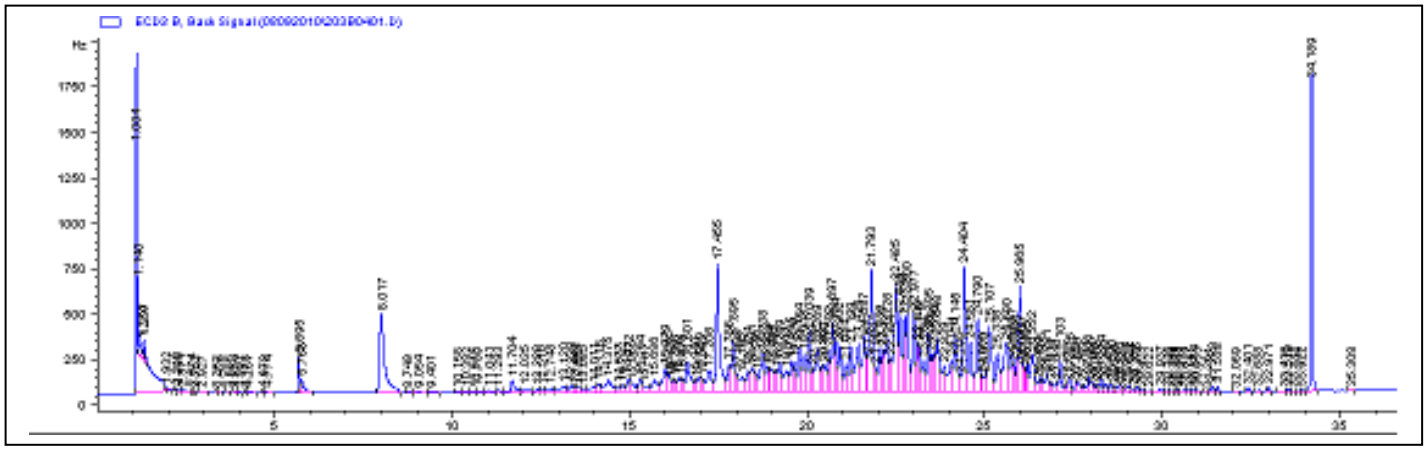
Compound	5 gram Avg. Conc.	Sample % rec.
TCMX	12.8 µg/kg	72%
Decachlorobiphenyl	18 µg/kg	74.5%
Toxaphenne	57.62 µg/kg	98.5%

Conclusions

Analysis of the sample data indicates good recoveries for technical toxaphene using the InCell cleanup for fish tissue samples of both 2.5 and 5 grams. The relative low sample RPDs for the toxaphene and decachlorobiphenyl also indicate excellent reproducibility. A higher RPD was observed for the TCMX recovery in the 5 gram sample due to the higher volatility of the compound and some concluded loss during the longer evaporation time.

The use of the PLE system with the InCell cleanup and the SuperVap® Direct-to-Vial concentration system consistently delivered reproducible, one-step extraction, clean-up and concentration for what is traditionally a three-step process. This new method substantially reduced the time of the traditional method. Samples were ready for same-day analysis and used substantially less lab reagents, which saves money.

Figure 1. Shows the chromatogram of a 2.5 gram sample runs on µECD detector.



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