Application Note

The Analysis of Chlorinated Dioxins, Difurans and Polychlorinated Biphenyls in Edible Oils

Introduction

The Dioxin family consists of 210 compounds, of which 17 contain the 2,3,7,8 pattern of Chlorination. These 2,3,7,8 containing compounds are of extreme human health concern due to their high level of toxicity. Similarly, 12 of the 209 polychlorinated biphenyls have also been identified as human toxins.

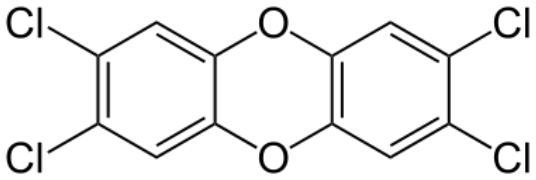

Figure #1, 2,3,7,8-TCDD structure

Due to their lipophilic nature, these analytes bio accumulate in adipose tissues and end up in food supplies. For this reason, the U.S. FDA and EU have established strict regulations for the monitoring of food products for human consumption, in particular edible oils.

Manual extractions of oils can be a time consuming procedure often delaying lab turnaround times. By automating the process, food oil samples can be reliably processed with routine 24 hour turnaround times. The following procedure utilizes the FMS PowerPrep® automated sample clean-up system to deliver this process.

Instrumentation

- FMS, Inc. PowerPrep®
- FMS, Inc. SuperVap® Concentrator
- FMS, Inc. 200ml concentrator tubes
- Thermo Trace Ultra GC with DFS HRMS
- Restek Dioxin 2, 60m GC column

Consumables

- FMS, Inc. High Capacity ABN Silica columns
- FMS, Inc. Basic Alumina columns
- FMS, Inc. Carbon columns
- Fisher Optima* Toluene
- Fisher Optima* n-Hexane
- Fisher Optima* Methylene Chloride
- Cambridge Isotopes EDF-9999, EPA 1613 calibration Standards
- Cambridge Isotopes EDF-8999, EPA 1613 Labeled Surrogate
- Cambridge Isotopes EDF-5999, EPA 1613 Recovery Standard
- Cambridge Isotopes EDF-6999, Labeled Clean-up Standard
- Cambridge Isotopes EDF-7999, EPA 1613 PAR
- Cambridge Isotopes EC-5045, WHO PCB +170&180 Clean-up STD
- Cambridge Isotopes EC-5000-1/2X, WHO PCB +170&180 Native STD
- Cambridge Isotopes EO-5275, Labeled Injection Internal Stock

FMS
Fluid Management Systems

Figure 1 PowerPrep Multicolumn Clean up system is expandable from 1 to 6 modules and goes directly to the SuperVap Concentrator.

Analyte	Mean	Dev	Blk Conc.
2,3,7,8-TCDD	78	8.6	<.1 pg/g
1,2,3,7,8-PeCDD	81	11.6	<.5 pg/g
1,2,3,4,7,8-HxCDD	81	11.3	<.5 pg/g
1,2,3,6,7,8-HxCDD	77	9.4	<.5 pg/g
1,2,3,7,8,9-HxCDD	NA	NA	<.5 pg/g
1,2,3,4,6,7,8-HpCDD	75	7.1	<.5 pg/g
OCDD	70	3.6	<1 pg/g
2,3,7,8-TCDF	70	8.5	<.1 pg/g
1,2,3,7,8-PeCDF	83	13.5	<.5 pg/g
2,3,4,7,8-PeCDF	81	10.7	<.5 pg/g
1,2,3,4,7,8-HxCDF	70	7.1	<.5 pg/g
1,2,3,6,7,8-HxCDF	62	3.6	<.5 pg/g
1,2,3,7,8,9-HxCDF	66	6.9	<.5 pg/g
2,3,4,6,7,8-HxCDF	71	10.0	<.5 pg/g
1,2,3,4,6,7,8-HpCDF	73	5.0	<.5 pg/g
1,2,3,4,7,8,9-HpCDF	85.3	9.0	<.5 pg/g
OCDF	NA	NA	<1 pg/g
PCB-77	73	14.9	<.5 pg/g
PCB-81	64	11.0	<.5 pg/g
PCB-105	75	15.2	<.5 pg/g
PCB-114	73	11.4	<.5 pg/g
PCB-118	73	8.5	<.5 pg/g
PCB-123	72	8.0	<.5 pg/g
PCB-126	88	19.7	<.5 pg/g
PCB-156	63	7.4	<.5 pg/g
PCB-157	53	8.7	<.5 pg/g
PCB-167	63	6.1	<.5 pg/g
PCB-169	75	10.4	<.5 pg/g
PCB-170	79	9.4	<.5 pg/g
PCB-180	77	14.2	<.5 pg/g
PCB-189	80	9.8	<.5 pg/g

Table #1. Mean recoveries and deviations for labeled compounds; Concentration of blank.

Procedure

Sample Prep

- Various Oil matrices obtained (Lard, Olive Oil, Corn Oil, Cod Oil, Red Palm Oil, Unrefined Pumpkin Oil, Unrefined Vegetable Oil)
- Aliquots of 5 gram samples are spiked with 13C labeled surrogate standards
- Samples are diluted into n-Hexane and drawn up into a gas tight syringe

PowerPrep

- Columns conditioned
- Samples loaded onto Power Prep
- Samples loaded across ABN silica columns in n-Hexane and eluted onto Alumina columns
- Alumina columns eluted onto carbon columns using Methylene Chloride. Methylene Chloride eluate collected in SuperVap as fractions #1
- Carbon column back eluted with Toluene and collected in SuperVap as Fraction #2

SuperVap

- Preheat temp: 20 minutes at 60 °C
- Evap mode w/Sensor temp: 60 °C
- Nitrogen Pressure: 10 PSI

Conclusions

Analysis of the 6 matrices processed yielded acceptable recoveries for all analytes with standard deviations below 20%. Analysis of an n-Hexane blank sample resulted in no detectable target analytes measured within the calibration range of each respective compound.

With a total processing time of less than 2.5 hours, the FMS PowerPrep® and SuperVap® Concentrator delivers an efficient, totally automated sample prep process for edible oils.

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