

# The Analysis of Chlorinated Dioxins, Difurans and Biphenyls in Edible Oils Using Low-Cost, Green, Automated Sample Clean Up

## 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.

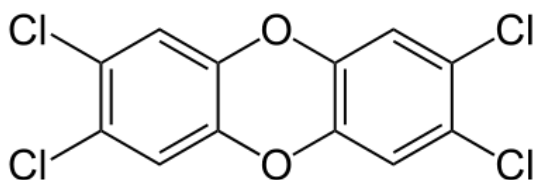


Figure #1, 2,3,7,8-TCDD structure

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 EconoPrep® automated sample clean-up system to deliver this process.

## Instrumentation

- FMS, Inc. EconoPrep®
- 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® Dichloromethane
- Fisher Optima® n-Hexane
- Fisher Optima® Toluene
- 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 EC-4995, WHO PCB +170&180 Labeled STD
- Cambridge Isotopes EC-5000-1/2X, WHO PCB +170&180 Native STD
- Cambridge Isotopes EO-5275, Labeled Injection Internal Stock



Figure 1 EconoPrep Multicolumn Clean up system is expandable from 1 to 4 modules and goes directly to the SuperVap Concentrator.

Analyte	Mean	Dev	Blk Conc.
2,3,7,8-TCDD	80	11	<.1 pg/g
1,2,3,7,8-PeCDD	67	6	<.5 pg/g
1,2,3,4,7,8-HxCDD	78	16	<.5 pg/g
1,2,3,6,7,8-HxCDD	82	7	<.5 pg/g
1,2,3,7,8,9-HxCDD	NA	NA	<.5 pg/g
1,2,3,4,6,7,8-HpCDD	82	9	<.5 pg/g
OCDD	65	10	<1 pg/g
2,3,7,8-TCDF	67	5	<.1 pg/g
1,2,3,7,8-PeCDF	69	5	<.5 pg/g
2,3,4,7,8-PeCDF	65	6	<.5 pg/g
1,2,3,4,7,8-HxCDF	72	8	<.5 pg/g
1,2,3,6,7,8-HxCDF	85	8	<.5 pg/g
1,2,3,7,8,9-HxCDF	85	6	<.5 pg/g
1,3,4,6,7,8-HxCDF	90	10	<.5 pg/g
1,2,3,4,6,7,8-HpCDF	86	8	<.5 pg/g
1,2,3,4,7,8,9-HpCDF	82	6	<.5 pg/g
OCDF	65	5	<1 pg/g
PCB-77	96	7	<.5 pg/g
PCB-81	94	5	<.5 pg/g
PCB-105	95	6	<.5 pg/g
PCB-114	92	4	<.5 pg/g
PCB-118	91	6	<.5 pg/g
PCB-123	93	7	<.5 pg/g
PCB-126	88	7	<.5 pg/g
PCB-156	101	6	<.5 pg/g
PCB-157	101	7	<.5 pg/g
PCB-167	99	5	<.5 pg/g
PCB-169	103	6	<.5 pg/g
PCB-170	104	12	<.5 pg/g
PCB-180	99	7	<.5 pg/g
PCB-189	100	11	<.5 pg/g

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

## Procedure

### Sample Prep

- Various Oil matrices obtained (Olive Oil, Corn Oil, Red Palm Canola Oil, Unrefined Pumpkin Seed Oil)
- Aliquots of 2 gram samples were spiked with  $^{13}\text{C}$  labeled surrogate standards
- Samples were diluted into n-Hexane and transferred into sample tubes

### EconoPrep

- Columns conditioned with n-Hexane
- Samples loaded onto EconoPrep
- Samples auto loaded across high capacity ABN silica columns in n-Hexane and eluted onto Alumina columns
- Alumina columns eluted with 10% Dichloromethane in Hexane to elute PCBs (collected in SuperVap tubes as Fraction # 1)
- Alumina columns eluted with Dichloromethane to transfer PCDD/Fs onto Carbon column
- Carbon columns eluted with Toluene to collect PCDD/Fs in SuperVap tubes as Fraction # 2
- Final n-Hexane push to flush out any residual PCDD/F in solvent lines into Fraction # 2

### SuperVap

- Preheat temp: 20 minutes at 50 °C (PCBs) or 55 °C (PCDD/Fs)
- Evap mode w/Sensor temp: 50 °C or 55 °C
- Nitrogen Pressure: 5-7 PSI



## Conclusions

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Analysis of the 4 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 for Sample Cleanup and Concentration of up to eight samples in parallel in less than 1.5 hours, the FMS EconoPrep® and SuperVap® Concentrator delivers an efficient, reproducible, high throughput totally automated sample prep process for edible oils.

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