Application Note

EPA 1613 and 1668C: PCDD/Fs and PCBs Analysis in Pet Food Using Automated Extraction and Cleanup

Introduction

Screening for PCDD/Fs and PCBs in human supplies is a well established practice. Both the EU and US have extensive protocols for the testing of food for human consumption as well as additives to feed for commercial livestock. With contamination issues in pet food like the 2007 melamine incident, there is a growing concern for pet safety.

With increasing efforts to screen both finished pet foods and pet food additives, the development of reliable analytical practices is of high priority. For Persistent Organic Pollutants, the high lipid content of canned and dried pet foods make them an ideal source of dietary contamination for household pets.

Using the FMS Total Prep solution (TRP®) for the extraction and cleanup of pet food, labs can deliver extracts for same day analysis. The following outlines the procedures for processing canned dog food for PCDD/Fs and PCBs via EPA methods 1613 and 1668C.

Instrumentation

- FMS, Inc. PowerPrep®
- FMS, Inc. PLE®
- FMS, Inc. SuperVap® Concentrator
- FMS, Inc. 200ml concentrator tubes
- FMS, Inc. 40ml extraction cells w/disposable end caps
- Thermo Trace Ultra GC with DFS HRMS
- Restek Dioxin 2, 60m GC column

Consumables

- FMS, Inc. High Capacity Acidic Silica columns
- FMS, Inc. Basic Alumina columns
- FMS, Inc. Carbon columns
- Fisher Optima* Toluene
- Fisher Optima* n-Hexane
- Fisher Optima* Methylene Chloride
- Acros n-Nonane
- Fisher dodecane
- Agilent Hyrdromatrix® (diatomaceous earth)
- Restek Ottawa Sand
- 1613 and 1668C spiking and recovery standards

Sample Prep

- 3 brands of canned dog food were obtained.
- Aliquots of 10 grams were spiked with ^{13}C labeled surrogate standards
- Samples were mixed with Hydromatrix till all moisture was absorbed
- Dried samples are transferred to extraction cells.
- Remainning cell volume topped off with Ottawa Sand.

PLE

1. Extraction cells loaded onto PLE
2. Cells filled sequentially with 50% DCM in Hexane
3. Extraction cells pressurized to 1500PSI
4. Cells heated to 120 ^{o}C and held for 20 minutes
5. Cells cooled to approximately ambient temperature
6. Cells flushed with extraction solvent
7. Solvent purged from cells direct to SuperVap with Nitrogen

Super Vap

1. Preheat temp: 20 minutes at 55 °C
2. Evap mode w/Sensor temp: 55 °C
3. Nitrogen Pressure: 6-8 PSI
4. Evaporator tubes rinsed with hexane several times to ensure no sample sticks to glass walls

Power Prep

1. Columns conditioned
2. Samples loaded onto Power Prep®
3. Samples loaded across High Capacity acidic silica columns in n-Hexane and euted onto Alumina columns
4. Alumina columns eluted onto carbon columns using Methylene Chloride (collect mono- and di-ortho PCBs).
5. Elute carbon columns in reverse with toluene (collect PCDD/Fs and coplanary PCBs).

FMS
Fluid Management Systems

Super Vap

1. Preheat temp: 20 minutes at 55 °C
2. Evap mode w/Sensor temp: 55 °C
3. Nitrogen Pressure: 6-8 PSI

After extracts reach sensor termination point, extracts transferred to GC vials.
5 µl dodecane added to vials and vials transferred to SuperVap® vial Concentrator

SuperVap® Vial Concentrator

1. temp: 25 °C
2. Nitrogen Pressure: 1.5 PSI

- Vials evaporated till volume of dodecane reached.
- 5 µl recovery standard added to extract and vials transferred to Trace GC for analysis

Results

	Run #1		Run #2		Run #3		MB	
Analyte	13C Rec	Native	13C Rec	Native	13C Rec	Native	13C Rec	Native
2,3,7,8-TCDF	82	<.0178	104	0.0456	78	0.0075	90	ND
2,3,7,8-TCDD	84	0.0113	105	0.1394	77	0.0331	92	ND
1,2,3,7,8-PeCDF	67	<.0291	79	0.037	65	0.0119	74	ND
2,3,4,7,8-PeCDF	69	0.0162	84	0.0317	70	<.0088	78	ND
1,2,3,7,8-PeCDD	72	0.0373	85	0.7676	72	<.0174	79	ND
1,2,3,4,7,8-HxCDF	84	0.0192	112	<.0216	89	0.0148	96	ND
1,2,3,6,7,8-HxCDF	80	0.0148	108	0.0244	83	0.0108	91	ND
2,3,4,6,7,8-HxCDF	82	0.0174	105	0.0161	85	0.0116	94	ND
1,2,3,7,8,9-HxCDF	90	0.0088	114	0.0262	91	0.0068	99	ND
1,2,3,4,7,8-HxCDD	84	0.033	109	0.9383	87	<.0089	93	ND
1,2,3,6,7,8-HxCDD	80	0.0246	105	1.3878	85	<.0188	92	ND
1,2,3,7,8,9-HxCDD		0.0191		0.9943		0.0119		ND
1,2,3,4,6,7,8-HpCDF	67	<.0754	85	<.0754	76	0.0701	79	ND
1,2,3,4,7,8,9-HpCDF	76	ND	97	<.0142	85	0.0114	90	ND
1,2,3,4,6,7,8-HpCDD	73	0.2057	90	12.2956	81	0.0775	85	ND
OCDD	66	1.2475	75	115.7943	78	0.2291	77	0.2309
OCDF		0.0355		0.0393		<.0556		ND

Analytical results, native compounds reported in pg/g

Conclusions

Review of the 3 pet food matrices shows labeled recoveries ranging 65-109%, well within EPA1613 limits. Analysis of the Method Blank showed no background levels above the CS .1 calibration standard level except OCDD. Combining the clean background with good recoveries demonstrates the TRP process' ability to handle wet pet food of various types.

With a total process time of ~5 hours from start to finish, the TRP process enables sample turnaround for same day analysis. When factoring in the need to qualify batches for release and product delays, the value of rapid testing becomes critical.

PLE®, PowerPrep® and SuperVap® Concentrator

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