Extraction method for PCDD/F and PCB analysis in consumer products using pressurized fluid extraction

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DIOXIN 22
10.9–14.2022 | New Orleans, LA

Different consumer product matrices

products and raw material

- Textile
- Nonwoven
- Diaper
- …

Pictures: different consumer product samples

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Aim of study

Establish method using X-Traction from LCTech for different consumer product samples at GALAB Laboratories

- One method for all samples matrices

- LOQ → low ng/kg range (2,3,7,8-TCDD → 0.05 ng/kg)

- QC performance criteria → based on US-EPA 1613, 1668 and EU-regulations for food/feed; like recovery rates for internal ^{13}C-labelled standards

Picture by LCTech

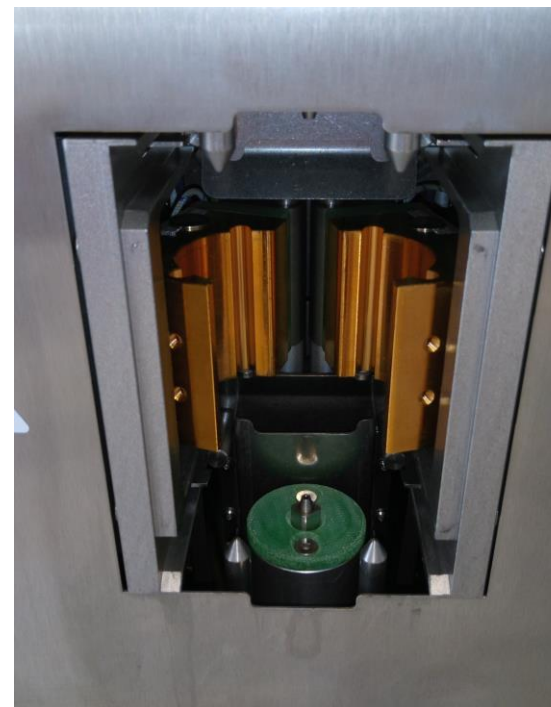DIOXIN 22
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LCTech X-Traction

Picture: sample cell

Picture: cell holder

Picture: oven

Picture: X-Traction at GALAB

Influence in extraction efficiency : solvent, temperature (pressure) and time

Starting point

Inhouse method, X-Traction application notes and experience

n-hexane method

- 3 cycles / 5 minutes

- 40mL per cycle

- Extraction temperature set at 85°C

- Natural contaminated samples

Analyte	Textile sample		
	reference value	value	recovery
	ng/kg	ng/kg	
2,3,7,8-TCDF	0.771	<LOQ	0%
1,2,3,7,8-PeCDF	0.486	<LOQ	0%
2,3,4,7,8-PeCDF	0.344	<LOQ	0%
1,2,3,4,7,8-HexCDF	3.862	0.068	2%
1,2,3,6,7,8-HexCDF	0.357	<LOQ	0%
2,3,4,6,7,8-HexCDF	0.191	<LOQ	0%
1,2,3,7,8,9-HexCDF	1.783	<LOQ	0%
1,2,3,6,7,8-HexCDD	0.557	0.049	9%
1,2,3,7,8,9-HexCDD	0.446	<LOQ	0%
1,2,3,4,6,7,8-HepCDF	1.236	<LOQ	0%
1,2,3,4,7,8,9-HepCDF	4.134	0.478	12%
1,2,3,4,6,7,8-HepCDD	10.692	<LOQ	0%
OCDD	22.719	1.068	5%
OCDF	1.117	<LOQ	0%
PCB 77	5.000	0.042	1%
PCB 81	0.273	<LOQ	0%
PCB 126	0.683	0.263	39%
PCB 169	1.389	0.084	6%
	Mean:		4%

Result tables n-hexane and n-hexane/toluene 1:1

n-hexane method:

Matrix	Mean Recovery	(min)	(max)
Nonwoven	88%	80%	95%
Diaper	185%	150%	220%
Textile	4%	0%	39%

n-hexane/toluene 1:1 method:

Matrix	Mean Recovery	(min)	(max)
Nonwoven	106%	96%	118%
Diaper	247%	150%	343%
Textile	35%	8%	97%
Ref. Material	79%	28%	214%

Extraction efficiency not enough

Result table pure toluene and toluene/acetone 7:3

toluene method:

Matrix	Mean Recovery	(min)	(max)
Nonwoven	n.a.	n.a.	n.a.
Diaper	n.a.	n.a.	n.a.
Textile	93%	55%	146%
Ref. Material	138%	89%	240%

toluene/acetone 7:3 method:

Matrix	Mean Recovery	(min)	(max)
Nonwoven	n.a.	n.a.	n.a.
Diaper	113%	-	-
Textile	123%	88%	209%
Ref. Material	142%	91%	267%

Both not working for nonwoven

Nonwoven and diaper sample blocked the system → no extraction possible → another solvent mixture

Result table toluene/isopropanol 7:3 v/v

toluene/isopropanol method:

40mL per cycle, 3 cycles, 5 minutes holding time, 105°C

Matrix	Mean Recovery	(min)	(max)
Nonwoven	104%	93%	123%
Textile	74%	17%	191%
Ref. Material	146%	80%	330%

Is working for all tested matrices → acceptable extraction efficiency compared to inhouse reference method

Summary

Solvent	Extraction efficiency	Matrix restriction
n-hexane	Lowest	Non
n-hexane/toluene 1:1 (v/v)	Low	Non
toluene	Very good	Yes
toluene/acetone 7:3 (v/v)	Very good	Yes
toluene/isopropanol 7:3 (v/v)	Good	Non

Compromise method for consumer products: "toluene/isopropanol 7:3 v/v"

1g of sample mixed with bulk material

40mL per cycle, 3 cycles, 5 minutes, 105°C

Outlook

Standardized methods for consumer products

Single methods for different matrices, if "true" results are necessary

More control of consumer product to avoid unwilling production of PCDD/F and PCB

Thanks for your interest!

Questions?

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