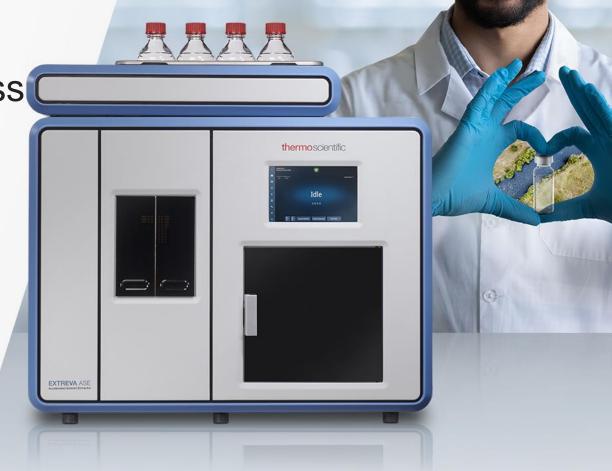
#### **Thermo Fisher** s c | e N T | F | C

## From sample to vial in one seamless ( operation with EXTREVAASE<sup>™</sup>

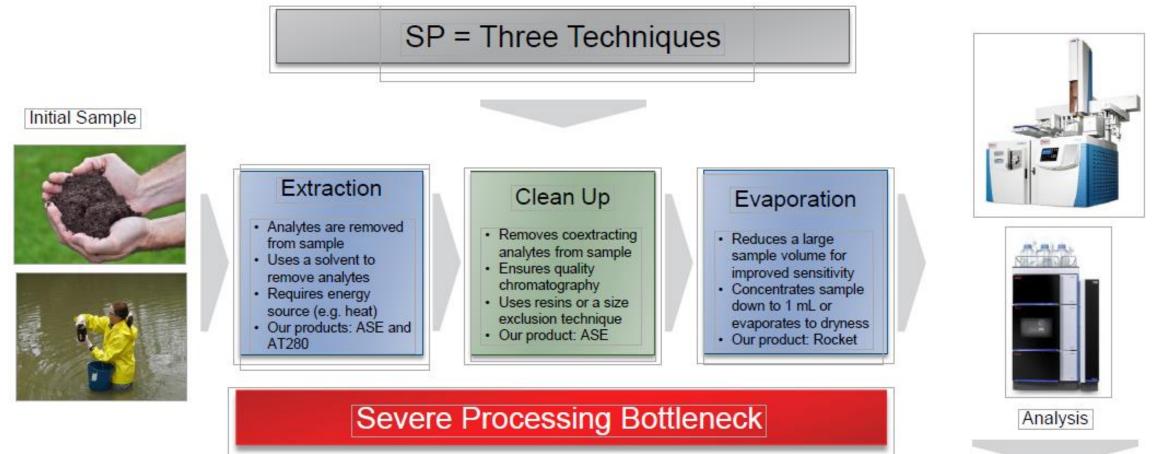
#### **Chris Shevlin**

Scientific Affairs Manager Thermo Fisher Scientific chris.shevlin@thermofisher.com

The world leader in serving science



## The sample preparation workflow



- 2/3 of processing time spent preparing samples
- >80% of all laboratory error occurs within these steps



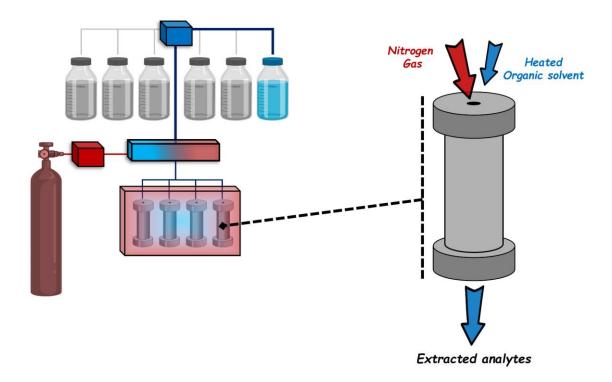
Data Recording and Reporting

## Sample prep issues

The source of many problems

Not cost effective	Uses large volume of solvents and takes a long time (4 to 48 hours)	Limits sample throughput and turnaround times, solvent usage drives up cost
Error prone	A vast majority of errors in a lab occur during sample prep	Rerunning and resampling drive up costs, accreditation risk when running PT samples
Good results need good sample prep	Errors may not be realized until data processing shown as poor recoveries and high reproducibility	Rerunning and resampling drive up costs, accreditation risk when running PT samples
Inefficient use of labor	Manual processes are laborious and tedious	Personnel could be better utilized. Labs don't realize how much productivity they are losing.
Personnel management has significant costs	Technicians are not always available. Many of these labs see a high staff turnover.	Limits productivity. Training new staff greatly impacts costs.

#### **Gas assisted dynamic extraction**

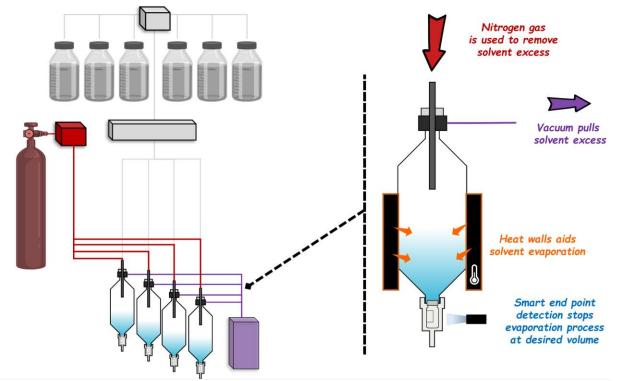


 Co-use of pressurized gas during the pressurized liquid extraction process, and the continuous replenishment of the gas-solvent mixture

Thermol

- Solvent is added for a short period of time
- Gas follows for the second period of time
- Pressure and flow rate can be precisely controlled
- Segmented alternating flow of solvent and gas follows at a user determined flow rate
- During the entire extraction, the cell is heated and held at high pressure

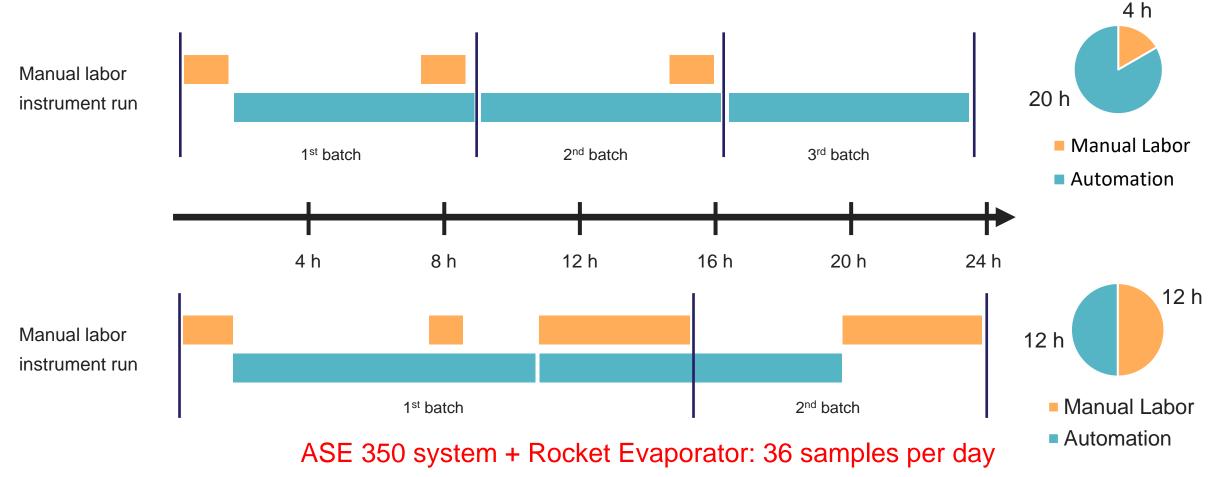
#### **Concentration with fully automated end-point detection**



- Automated end-point detection using machine learning, combined with image sensor and backlighting, solves the issue of monitoring evaporation in real-time
- Machine learning is employed to train the instrument to stop at the desired level, ensuring precise and automated end-point detection
- Eliminates the need for constant monitoring of evaporation, enabling **true walk-away sample preparation**

## New technology advantages

Based on 10 mL cell extraction



#### EXTREVA ASE system: 48 samples per day

## **EXTREVA ASE system - Key benefits**





**Combined footprint** Extraction and evapo



Increased laboratory productivity

Streamlined sample extraction

concentration

Combines sample extraction, clean-up, and

Parallel extraction of up to 4 samples at once, reduces the cost per sample

Extraction and evaporation system in one platform



**Complete workflow from sample to vial** True walk-away technology

Increased productivity, better consistency, and cost savings

## **EXTREVA ASE system – How it helps you**

#### Sample prep pain points

- Complicated workflows
- 2 Sample throughput
- Labor intensive workflows
- 4 Risk of errors
- High costs labor and solvent usage
- Target analyte recovery and reproducibility
- Sample tracking

# How the EXTREVA ASE system solves issues



Accomplishes extraction and concentration without user interaction



- Extracts and concentrates 4 samples in parallel
- 3 Co for
  - Completes the entire process without the need for intervention



- Performs method consistently without outside distractions
- Reduces the required hands-on time and solvent used for extraction
- 6

5

Brings a higher level of consistency and accuracy with automation compared to manual prep



Records sample prep parameters by 2D barcode reading

#### Cost of errors - %RSD, reruns, lost samples

## **Applications Data** Organochlorine pesticides (OCPs)

## **OCPs – EXTREVA ASE system vs ASE 350 system**

#### EXTREVA ASE system extraction

Extraction	
Cell type	Stainless steel
Cell size	10 mL and 100 mL
Oven temperature	100 °C
Purge time	45 s (10 mL cell); 180 s (100 mL cell)
Nitrogen flow	10 mL/min per channel
(gas assisted extraction)	
Cell fill volume	50%
Solvent flow rate	1.1 mL/min (10 mL cell);
	0.75 mL/min (100 mL cell)
Extraction solvent	Acetone-Hexane (1:1)
Extraction volume	~26 mL (10 mL cell);
	~70 mL (100 mL cell)
Extraction time	~15 min (10 mL cell);
(four samples)	~20 min (100 mL cell)
Rinse	Prerun, 10 mL,
	Acetone-Hexane (1:1)
Concentration	
Mode	Fixed volume
Collection bottle	100 mL vial assembly
Final fixed volume	1 mL
Rinse solvent	Hexane, 1.6 mL
Evaporation temperature	40 °C
Nitrogen flow rate	50 mL/min per channel
Vacuum	8 psi (414 torr/551 mbar)

#### ASE 350 system extraction (40 and 60 mL cells)

#### **Extraction Conditions**

Temperature:	100 °C		
Pressure:	1500 psi*		
Heatup Time:	5 min		
Static Time:	5 min		
Flush Volume:	60%		
Purge Time:	100 s		
Static Cycles:	1–2		
Total Extraction Time:	14–18 min per sample		

#### The EXTREVA ASE system extracts 4 samples

in the same amount of time as a single extraction instrument

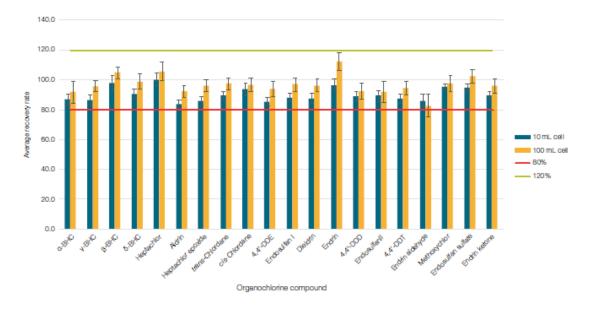
#### **OCPs**



#### Determination of organochlorine pesticides (OCPs) in soils using the EXTREVA ASE Accelerated Solvent Extractor and GC-ECD

Compound	Average recovery (%) (10 mL cell, n = 12)	RSD	Average recovery (%) (100 mL cell, n = 12)	RSD
a-BHC	86.7	3.7	91.7	7.6
γ-BHC	86.4	3.3	95.6	3.9
β-BHC	97.8	5.1	104.8	3.9
δ-BHC	90.6	3.5	98.9	4.8
Heptachlor	100.2	4.4	105.6	6.1
Aldrin	83.9	2.8	92.3	3.9
Heptachlor epoxide	85.8	2.8	96.0	4.1
trans-Chlordane	89.3	2.7	97.4	3.9
cis-Chlordane	93.5	4.0	96.7	4.6
4,4'-DDE	85.5	2.9	93.9	5.0
Endosulfan I	87.9	2.7	96.9	4.5
Dieldrin	87.4	3.4	96.3	4.6
Endrin	96.2	4.3	112.3	5.8
4,4'-DDD	89.1	3.2	92.3	5.1
Endosulfan II	89.3	3.5	91.7	7.2
4,4'-DDT	87.3	3.1	94.7	4.7
Endrin aldehyde	86.1	4.0	82.5	7.6
Methoxychlor	95.4	1.9	97.7	5.2
Endosulfan sulfate	94.6	2.5	102.3	4.7
Endrin ketone	89.5	2.4	95.9	5.0

# Average recoveries and reproducibility show excellent performance



All %RSD <10% and recoveries 82 – 106% (General acceptance <20% and 70 to 130%)

## **OCPs – Carryover and degradation tests**

#### EXTREVA ASE system

#### Carry Over Test

OCP	Average recovery % (10 mL, n = 4)	RSD %	Average carryover % (10 mL, n = 4)
a-BHC	81.7	7.9	0.00
γ-BHC	83.1	6.5	0.19
β-BHC	93.9	5.7	0.07
δ-BHC	89.6	5.0	0.09
Heptachlor	90.1	7.0	0.33
Aldrin	86.9	6.9	0.00
Heptachlor epoxide	92.6	5.7	0.01
trans-Chlordane	92.9	5.0	0.00
cis-Chlordane	93.5	5.6	0.05
4,4'-DDE	86.6	5.8	0.06
Endosulfan I	90.6	5.1	0.00
Dieldrin	94.4	4.8	0.01
Endrin	102.2	4.3	0.02
4,4'-DDD	91.0	3.9	0.00
Endosulfan II	89.8	4.0	0.43
4,4'-DDT	91.7	3.8	0.02
Endrin aldehyde	83.8	5.1	0.03
Methoxychlor	98.6	4.4	0.14
Endosulfan sulfate	97.5	3.5	0.03
Endrin ketone	95.0	3.6	0.03

## The EXTREVA ASE system yields very little carryover from high spike sample

#### EXTREVA ASE system Thermal Degradation Test

	Average breakdown (%)		
Extraction temperature	Endrin	DDT	
100 °C	4.0	1.5	
150 °C	3.2	1.0	

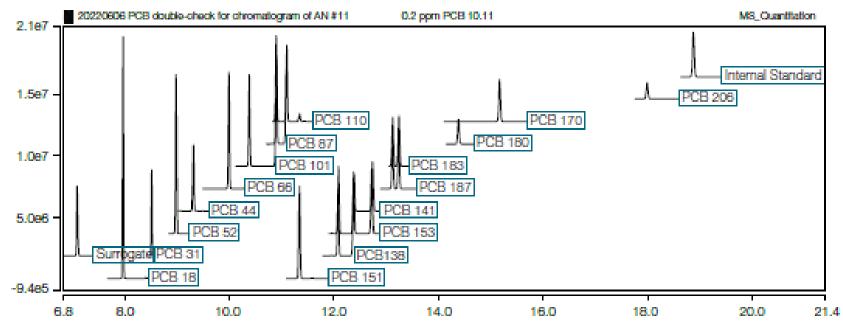
- Breakdown percentages are below recommended 15%
- For endrin, 3.1% breakdown occurred in the GC inlet
- The EXTREVA ASE system has little significant effect on the breakdown

## **Applications Data** Polychlorinated biphenyls (PCBs)

## Instrument conditions and chromatography

Extraction	
Cell type	Stainless steel
Cell size	10 mL and 100 mL
Oven temperature	100 °C
Purge time	45 s (10 mL cell) 180 s (100 mL cell)
Nitrogen flow (gas assisted extraction)	10 mL/min per channel
Cell fill volume	50%
Solvent flow rate	1.6 mL/min (10 mL cell) 0.35 mL/min (10 mL cell) 0.75 mL/min (100 mL cell)
Extraction solvent	Hexane
Extraction volume	<ul> <li>-26 mL (10 mL cell, flow rate 1.6 mL/min)</li> <li>-15 mL (10 mL cell, flow rate 0.35 mL/min)</li> <li>-70 mL (100 ml cell, flow rate 0.75 mL/min)</li> </ul>
Pre-run rinse	10 mL, Hexane
Extraction time (four samples)	-10 min (10 mL cell, flow rate 1.6 mL/min) -15 min (10 mL cell, flow rate 0.35 mL/min -20 min (100 mL cell, flow rate 0.75 mL/m

Concentration	
Mode	Fixed volume
Collection bottle	100 mL vial assembly
Final fixed volume	1 mL
Rinse solvent	Hexane, 1.6 mL
Evaporation temperature	40 °C
Nitrogen flow rate	50 mL/min per channel
Vacuum	8 psi (414 torr/551 mbar)



**Thermo Fisher** 

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## **PCBs – recovery and reproducibility**

#### 2 Methods and 2 cells

	10 mL – Method 1		10 mL – Method 2		100 mL	
Compound	Average recovery % (n=12)	RSD	Average recovery % (n=12)	RSD	Average recovery % (n=12)	RSD
PCB 18	82.8	7.4	79.2	3.8	77.0	2.6
PCB 31	88.2	7.3	81.4	4.9	80.4	2.8
PCB 52	90.4	7.8	82.5	3.7	82.0	2.5
PCB 44	93.7	6.3	87.5	4.3	82.4	2.8
PCB 66	91.6	6.2	87.6	3.1	87.7	2.6
PCB 101	92.6	5.1	86.3	5.0	86.5	2.6
PCB 87	94.3	5.2	87.1	2.6	88.7	2.6
PCB 110	95.5	5.2	86.8	2.9	86.9	2.2
PCB 151	92.9	4.0	84.6	3.3	88.9	1.9
PCB 153	91.4	3.6	90.7	6.2	92.8	2.2
PCB 141	95.6	3.0	90.7	4.0	93.4	2.3
PCB 138	99.4	4.7	94.6	4.4	94.8	2.3
PCB 187	95.4	6.8	94.8	3.5	92.9	2.5
PCB 183	94.9	6.5	89.2	4.2	92.8	2.6
PCB 180	97.4	4.6	93.7	4.4	101	4.7
PCB 170	92.7	8.1	98.0	4.4	99.8	2.0
PCB 206	85.8	8.7	93.7	4.3	97.0	2.1

## **PCBs – recovery and reproducibility**

#### 2 Methods and 2 cells



## **PCBs Carryover**

Compound	Average recovery (%) (10 mL cell, n=12)	RSD	Average carryover (%) (10 mL cell, n=12)
PCB 18	77.0	10.5	0.02
PCB 31	80.5	9.2	0.05
PCB 52	84.3	8.0	0.04
PCB 44	80.4	9.2	0.06
PCB 66	90.6	5.5	0.05
PCB 101	90.0	5.4	0.05
PCB 87	91.1	5.0	0.05
PCB 110	91.3	5.1	0.06
PCB 151	95.8	3.5	0.05
PCB 153	95.9	3.8	0.04
PCB 141	97.4	3.4	0.03
PCB 138	98.0	3.5	0.04
PCB 187	99.7	3.7	0.06
PCB 183	97.7	3.6	0.07
PCB 180	94.0	4.6	0.20
PCB 170	106	5.2	0.05
PCB 206	101	3.6	0.12

## **PCBs – Proficiency testing samples**

PT samples were purchased and run

- All 18 PCBs were detected within the PT published acceptance range
- The average of 12 sample replicates also fell within the published range and have excellent reproducibility

PCB compounds	B compounds Certified value Acceptance range		Average recov (10mL cel	-
·	µg/kg	µg/kg	Avg (n=12) µg/kg	RSD (n=12)
PCB 28	64.7 ± 17.3	12.8 to 117	60.4	4.20
PCB 52	155 ± 37	45.1 to 265	136.0	3.90
PCB 101	37.3 ± 11.5	2.6 to 71.9	38.5	4.50
PCB 81	44.3 ± 8.7	18.2 to 70.4	44.9	4.30
PCB 77	115 ± 22	48.6 to 181	107.0	4.00
PCB 123	$35.6 \pm 6.2$	16.9 to 54.3	33.7	5.10
PCB 118	120 ± 22	53.9 to 186	113.0	4.00
PCB 114	$234 \pm 50$	85.2 to 382	227.0	3.70
PCB153	147 ± 46	8.8 to 285	158.0	4.10
PCB105	$40.8 \pm 8.6$	14.9 to 66.7	40.1	4.70
PCB138	112 ± 32	17.2 to 207	118.0	4.50
PCB126	280 ± 61	97.5 to 462	270.0	3.60
PCB167	122 ± 25	47.9 to 197	123.0	4.40
PCB156	$193 \pm 38$	79.5 to 307	157.0	3.70
PCB157	216 ± 50	67.7 to 365	180.0	3.50
PCB180	146 ± 40	26.9 to 266	124.0	4.60
PCB169	66.7 ± 13.3	26.8 to 107	58.3	5.20
PCB189	123 ± 24	51.3 to 195	104.2	4.20

## **Applications Data** Organochlorine pesticides (OCPs)

## **OCPs – EXTREVA ASE system vs ASE 350 system**

#### EXTREVA ASE system extraction

Extraction	,
Cell type	Stainless steel
Cell size	10 mL and 100 mL
Oven temperature	100 °C
Purge time	45 s (10 mL cell); 180 s (100 mL cell)
Nitrogen flow	10 mL/min per channel
(gas assisted extraction)	to mermin per channel
Cell fill volume	50%
Solvent flow rate	1.1 mL/min (10 mL cell);
	0.75 mL/min (100 mL cell)
Extraction solvent	Acetone-Hexane (1:1)
Extraction volume	~26 mL (10 mL cell);
	~70 mL (100 mL cell)
Extraction time	~15 min (10 mL cell);
(four samples)	~20 min (100 mL cell)
Rinse	Prerun, 10 mL,
	Acetone-Hexane (1:1)
Concentration	
Mode	Fixed volume
Collection bottle	100 mL vial assembly
Final fixed volume	1 mL
Rinse solvent	Hexane, 1.6 mL
Evaporation temperature	40 °C
Nitrogen flow rate	50 mL/min per channel
Vacuum	8 psi (414 torr/551 mbar)

#### ASE 350 system extraction

#### **Extraction Conditions**

Temperature:	100 °C	
Pressure:	1500 psi*	
Heatup Time:	5 min	
Static Time:	5 min	
Flush Volume:	60%	
Purge Time:	100 s	
Static Cycles:	1–2	
Total Extraction Time:	14–18 min per sample	

#### The EXTREVA ASE system extracts 4 samples

in the same amount of time as a single extraction instrument

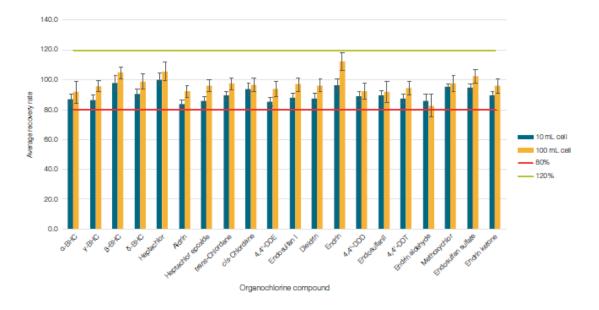
#### **OCPs**



#### Determination of organochlorine pesticides (OCPs) in soils using the EXTREVA ASE Accelerated Solvent Extractor and GC-ECD

Compound	Average recovery (%) (10 mL cell, n = 12)	RSD	Average recovery (%) (100 mL cell, n = 12)	RSD
a-BHC	86.7	3.7	91.7	7.6
γ-BHC	86.4	3.3	95.6	3.9
β-BHC	97.8	5.1	104.8	3.9
δ-BHC	90.6	3.5	98.9	4.8
Heptachlor	100.2	4.4	105.6	6.1
Aldrin	83.9	2.8	92.3	3.9
Heptachlor epoxide	85.8	2.8	96.0	4.1
trans-Chlordane	89.3	2.7	97.4	3.9
cis-Chlordane	93.5	4.0	96.7	4.6
4,4'-DDE	85.5	2.9	93.9	5.0
Endosulfan I	87.9	2.7	96.9	4.5
Dieldrin	87.4	3.4	96.3	4.6
Endrin	96.2	4.3	112.3	5.8
4,4'-DDD	89.1	3.2	92.3	5.1
Endosulfan II	89.3	3.5	91.7	7.2
4,4'-DDT	87.3	3.1	94.7	4.7
Endrin aldehyde	86.1	4.0	82.5	7.6
Methoxychlor	95.4	1.9	97.7	5.2
Endosulfan sulfate	94.6	2.5	102.3	4.7
Endrin ketone	89.5	2.4	95.9	5.0

# Average recoveries and reproducibility show excellent performance



All %RSD <10% and recoveries 82 – 106%

## **OCPs – Carryover and degradation tests**

OCP	Average recovery % (10 mL, n = 4)	RSD %	Average carryover % (10 mL, n = 4)
a-BHC	81.7	7.9	0.00
γ-BHC	83.1	6.5	0.19
β-BHC	93.9	5.7	0.07
δ-BHC	89.6	5.0	0.09
Heptachlor	90.1	7.0	0.33
Aldrin	86.9	6.9	0.00
Heptachlor epoxide	92.6	5.7	0.01
trans-Chlordane	92.9	5.0	0.00
cis-Chlordane	93.5	5.6	0.05
4,4'-DDE	86.6	5.8	0.06
Endosulfan I	90.6	5.1	0.00
Dieldrin	94.4	4.8	0.01
Endrin	102.2	4.3	0.02
4,4'-DDD	91.0	3.9	0.00
Endosulfan II	89.8	4.0	0.43
4,4'-DDT	91.7	3.8	0.02
Endrin aldehyde	83.8	5.1	0.03
Methoxychlor	98.6	4.4	0.14
Endosulfan sulfate	97.5	3.5	0.03
Endrin ketone	95.0	3.6	0.03

#### EXTREVA ASE system Thermal Degradation Test

Thermo Fi

	Average breakdown (%)			
Extraction temperature	Endrin	DDT		
100 °C	4.0	1.5		
150 °C	3.2	1.0		

#### The EXTREVA ASE system yields

very little carryover from high spike sample

## **Applications Data** Combined dioxins and furans/PCBs



23 chris.shevlin@thermofisher.com NEMC Aug 2023

## **Test Summary: Dioxin/Furan; PCB** → *soil*

2378-TCDD 12378-PeCDD 123478-HxCDD 123678-HxCDD 123789-HxCDD 1234678-HpCDD OCDD

2378-TCDF

12378-PeCDF

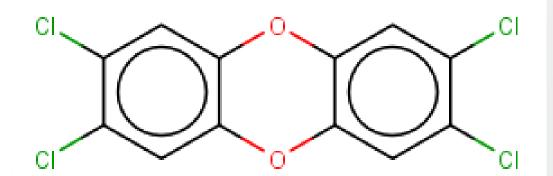
23478-PeCDF

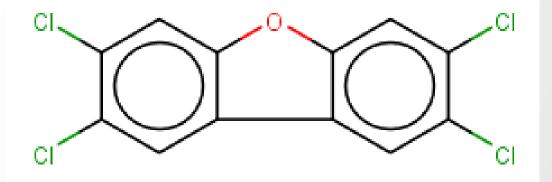
Analysis of

- 7 dioxins
- 10 furans

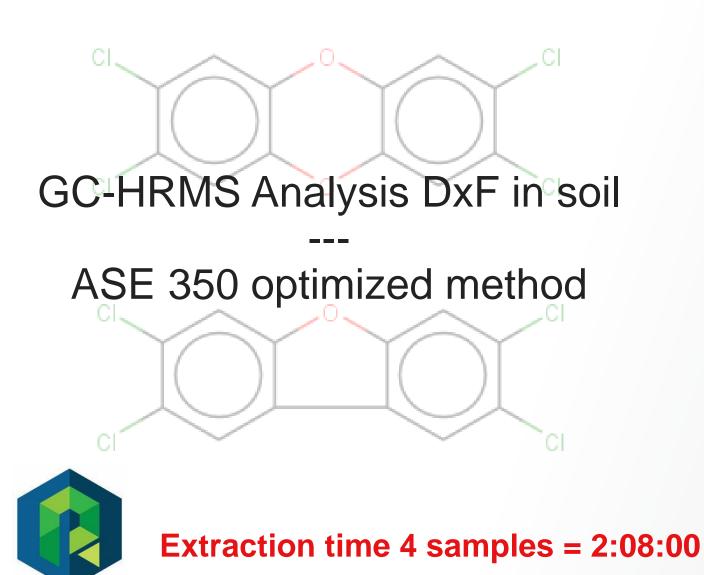


123478-HxCDF 123678-HxCDF 234678-HxCDF 123789-HxCDF 1234678-HpCDF 1234789-HpCDF 0CDF





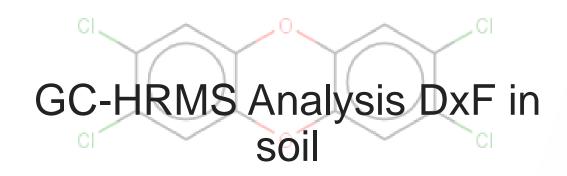
## **Dioxins and furans in soil – ASE 350**

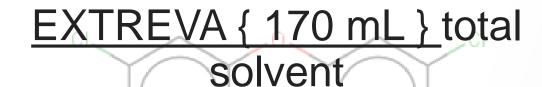


INTERNAL STANDARDS	% REC	STDE V	RSD
2378-TCDD	50	5.57	11.2%
12378-PeCDD	68	5.32	7.8%
123478-HxCDD	72	4.55	6.3%
123678-HxCDD	105	10.02	9.6%
1234678-HpCDD	72	10.86	15.1%
OCDD	40	2.83	7.1%
2378-TCDF	44	5.44	12.4%
12378-PeCDF	51	0.82	1.6%
23478-PeCDF	60	6.45	10.8%
123478-HxCDF	68	8.29	12.2%
123678-HxCDF	95	6.90	7.2%
234678-HxCDF	94	9.00	9.5%
123789-HxCDF	86	4.03	4.7%
1234678-HpCDF	93	7.63	8.2%
1234789-HpCDF	74	8.46	11.5%

### **Dioxins and furans in soil – EXTREVA ASE**

Thermo Fisher

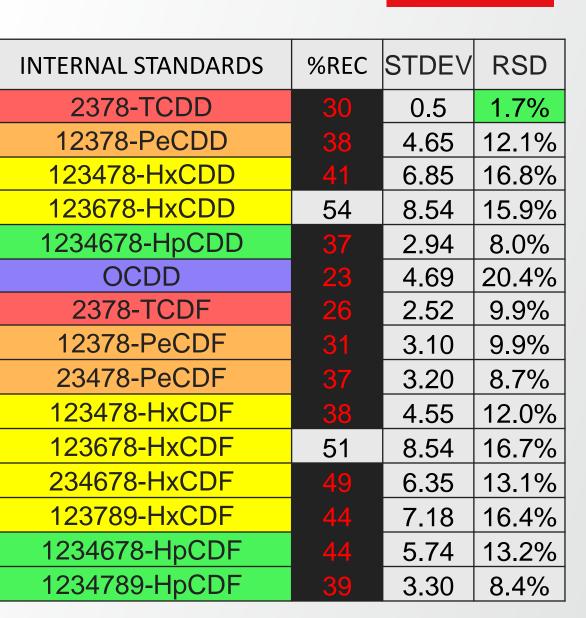






INTERNAL STANDARDS	%REC	STDE V	RSD
2378-TCDD	44	4.65	10.5%
12378-PeCDD	61	5.51	9.1%
123478-HxCDD	59	7.70	13.1%
123678-HxCDD	82	10.87	13.2%
1234678-HpCDD	57	5.97	10.4%
OCDD	35	4.65	13.5%
2378-TCDF	37	5.45	14.9%
12378-PeCDF	46	6.32	13.7%
23478-PeCDF	52	5.19	9.9%
123478-HxCDF	54	4.11	7.6%
123678-HxCDF	77	9.36	12.1%
234678-HxCDF	76	5.35	7.0%
123789-HxCDF	60	6.55	10.9%
1234678-HpCDF	72	7.27	10.1%
1234789-HpCDF	59	7.27	12.3%

## **Dioxins and furans in soil – EXTREVA ASE**



**Thermo Fisher** 

EXTREVA { 85 mL } total solvent

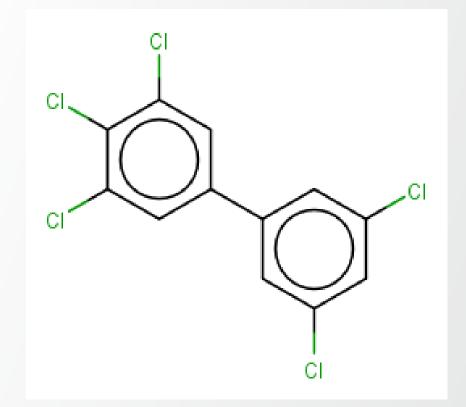
GC-HRMS Analysis DxF in soil



Extraction time 4 samples = 0:53:13

Thermo Fisher scientific

# GC-HRMS Analysis of 209 PCBs in soil





## **GC-HRAM 209 PCBs in Soil**

#### Old method with ASE 350

C13 INTERNAL STANDARD	%REC	RSD	PCB 104	32	14%	PCB 157	70	2%
PCB 1	7	33%	PCB 123	62	5%	PCB 169	66	5%
PCB 3	8	45%	PCB 118	60	6%	PCB 188	50	3%
PCB 4	13	30%	PCB 114	62	4%	PCB 189	87	7%
PCB 15	17	36%	PCB 105		3%	PCB 202	55	3%
PCB 19	20	25%	PCB 126		4%	PCB 205	74	1%
PCB 37	34	21%						
PCB 54	27	21%	PCB 155	40 '	11%	PCB 208	70	8%
PCB 81	59	10%	PCB 167	65	3%	PCB 206	61	3%
PCB 77	64	10%	PCB 156	68	7%	PCB 209	60	3%



## **GC-HRAM 209 PCBs in soil**

#### EXTREVA ASE Method

C13 INTERNAL STANDARD	%REC	RSD	PCB 104	32	14%	PCB 157	70	2%
PCB 1	7	33%	PCB 123	62	5%	PCB 169	66	5%
PCB 3	8	45%	PCB 118	60	6%	PCB 188	50	3%
PCB 4	13	30%	PCB 114	62	4%	PCB 189	87	7%
PCB 15	17	36%	PCB 105	66	3%	PCB 202	55	3%
PCB 19	20	25%	PCB 126	77	4%	PCB 205	74	1%
PCB 37	34	21%	PCB 155	40	11%	PCB 208	70	8%
PCB 54	27	21%	PCB 167	65	3%	PCB 206	61	3%
PCB 81	59	10%	PCB 156	68	7%	PCB 209	60	3%
PCB 77	64	10%						



# **Questions?**

