Recent advances in sample preparation for POPs

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Sample preparation challenges

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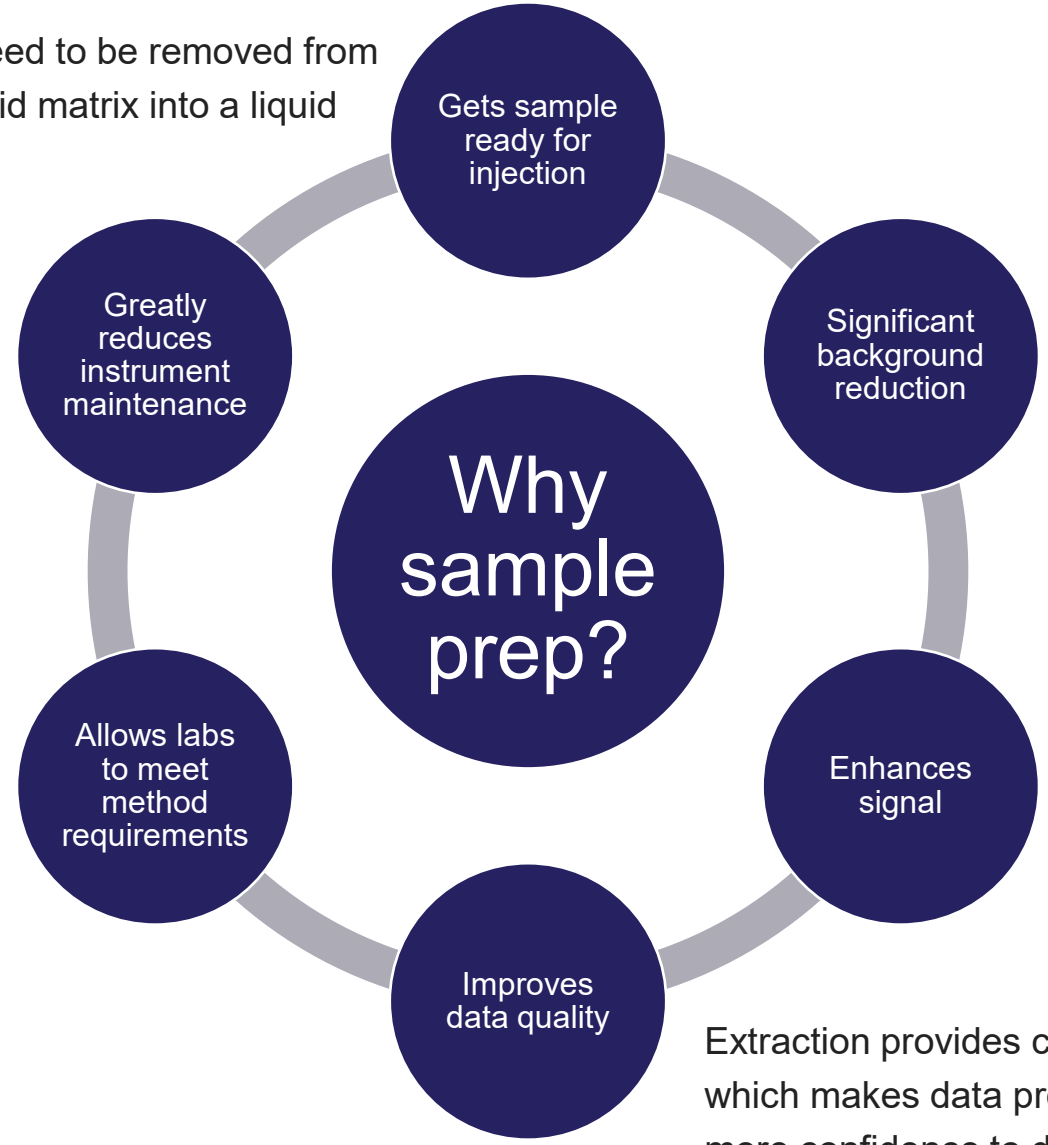
Sample prep is critical

Compounds need to be removed from solid / semi-solid matrix into a liquid

Dirty samples can foul up GC inlets, cause blockages in LC and foul up mass spec sources, causing ion suppression resulting in loss of signal

Good sample prep helps meet reproducibility recovery, and sensitivity requirements



Extraction removes many compounds which could show up on a chromatogram

Fewer compounds present in the source of the mass spec reduces competitive ionization and formation of neutrals

Extraction provides cleaner chromatograms which makes data processing easier and gives more confidence to data

The sample preparation workflow

SP = Three Techniques

Initial Sample

Extraction

- Analytes are removed from sample
- Uses a solvent to remove analytes
- Requires energy source (e.g. heat)
- Our products: ASE and AT280

Clean Up

- Removes coextracting analytes from sample
- Ensures quality chromatography
- Uses resins or a size exclusion technique
- Our product: ASE

Evaporation

- Reduces a large sample volume for improved sensitivity
- Concentrates sample down to 1 mL or evaporates to dryness
- Our product: Rocket

Analysis

Severe Processing Bottleneck

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

Data Recording and Reporting

Manual sample prep

- Time-consuming
  - Manual methods require a lot of time and constant attention
  - Setup and clean-up takes longer using manual methods
  - Sample must be manually transferred between preparation devices
- Meeting method performance requirements (recoveries and reproducibility)
  - Manual prep introduces variables that can affect the quality of the prep
- Controlling lab costs
  - More solvents used compared to automated methods
  - Increased risk of errors and resampling when manual processing samples
- Sample throughput – Manual prep takes a long time
- Sample data tracking and integrity
  - Documentation is mostly manual

Introducing the Thermo Scientific™ EXTREVA™ ASE™

Accelerated Solvent Extractor

First fully automated Sample Prep instrument on the Market.

Go from Soil Sample to GC vial in as little as 2-hours!

Parallel process 4 samples at a time, up to16 sample capacity

Extraction , Clean-up & Concentration worry free

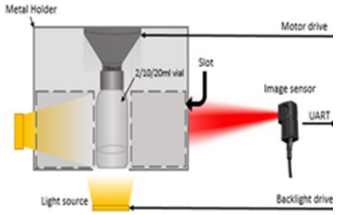
Prep Solid & Semi-solid samples for GC, GC/MS & LC/MS analysis of priority pollutants.

Extraction → Concentration → Determination → Data results

Reduce- Time Spent doing Sample Prep

Time repeating prep & analysis

Solvent use and disposal coast

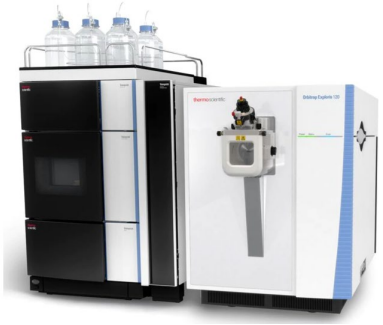


Capital Equipment for Automation of sample Prep for existing POP Analysis Workflows

EXTREVA ASE system - Integrated analytical workflows

Extraction → Concentration → Determination → Data Results

GC based applications:
- PAHs from soil
- PCBs from soil
- OCPs from soil

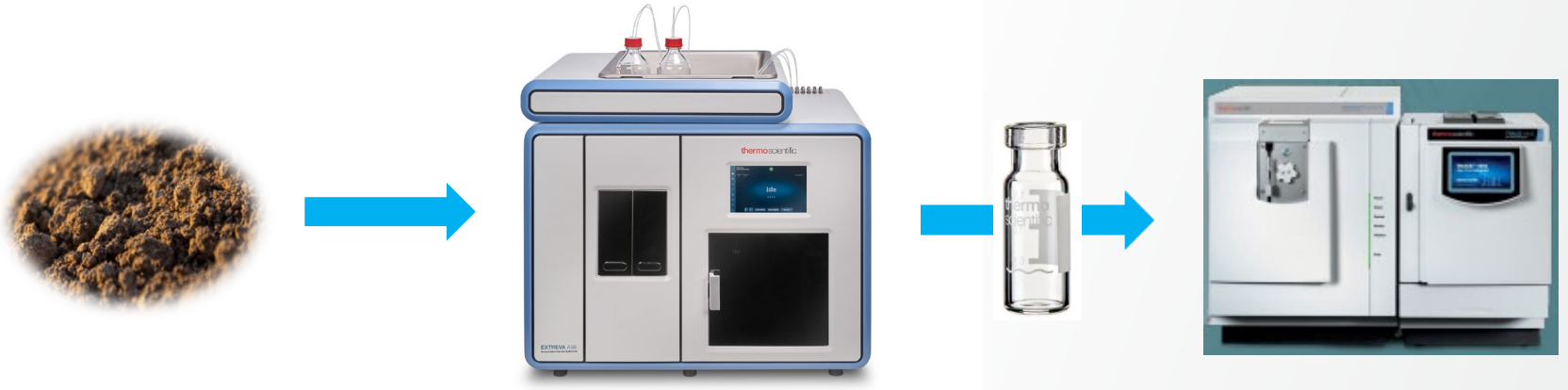


LC based applications:
- Extractables and leachables in food contact materials

Automation from sample to vial eliminates pressures of sample preparation

EXTREVA ASE system – How it impacts your lab

"I want to put my sample in one side and have it ready for analysis"



First of its kind! Start-to-finish sample prep without user intervention

Save time, increase productivity

- ✓ Parallel processing, combined automated extraction and evaporation without instrument interaction

Control cost

- ✓ New gas assisted extraction saves on solvents and extraction time
- ✓ Automation of whole process reduces errors
- ✓ Training new analysts faster and easier

Data integrity for accreditation

- ✓ 2D barcode tracking allows for full documentation of all sample conditions

Inside the EXTREVA ASE system

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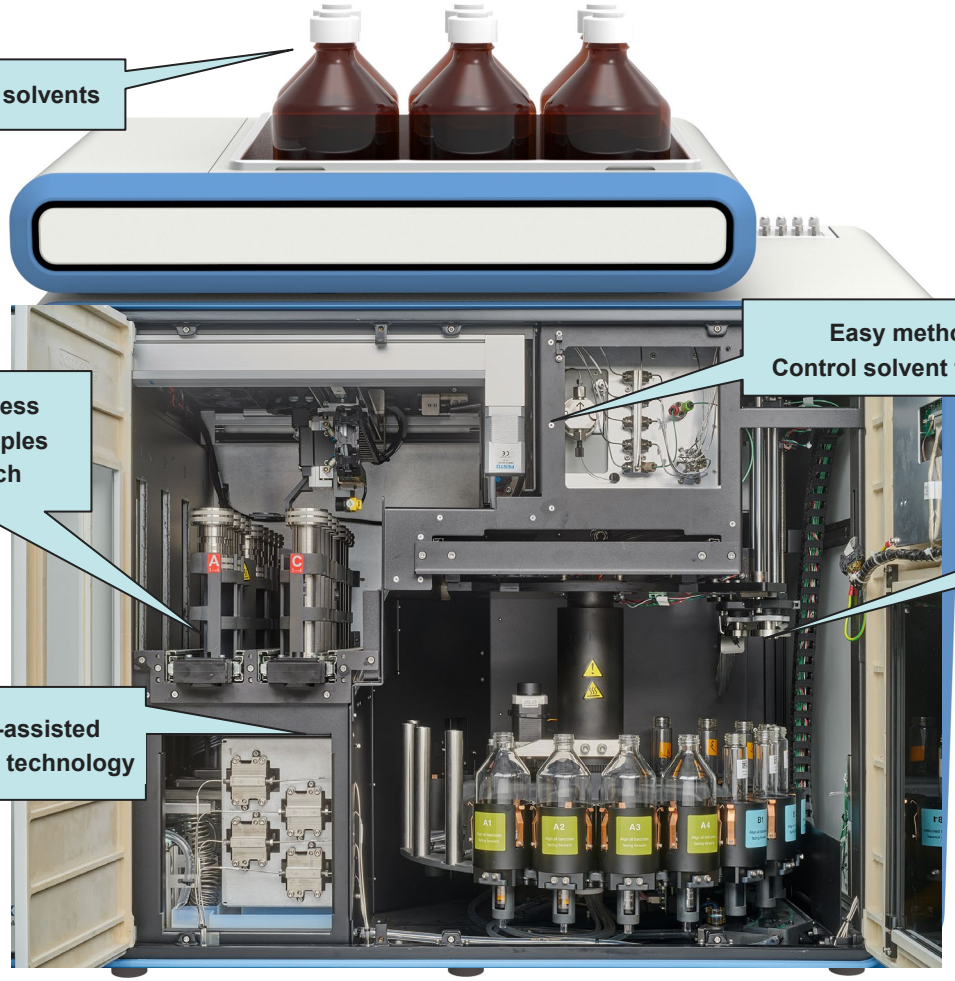
EXTREVA ASE Accelerated Solvent Extractor

ThermoFisher SCIENTIFIC

True walk-away automated extraction and concentration

Sample tracking with 2D barcode



Up to 6 organic solvents

Parallel process up to 16 samples in one batch

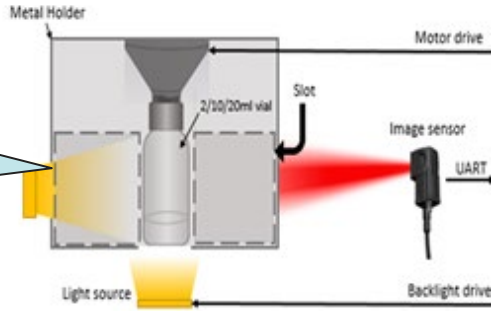
Patented gas-assisted solvent delivery technology

Easy method development. Control solvent temperature and time.

Automatic incorporation of extraction and evaporation

Powerful user interface control or Thermo Scientific™ Chromeleon™ CDS software

Evaporation end-point detection using AI technology



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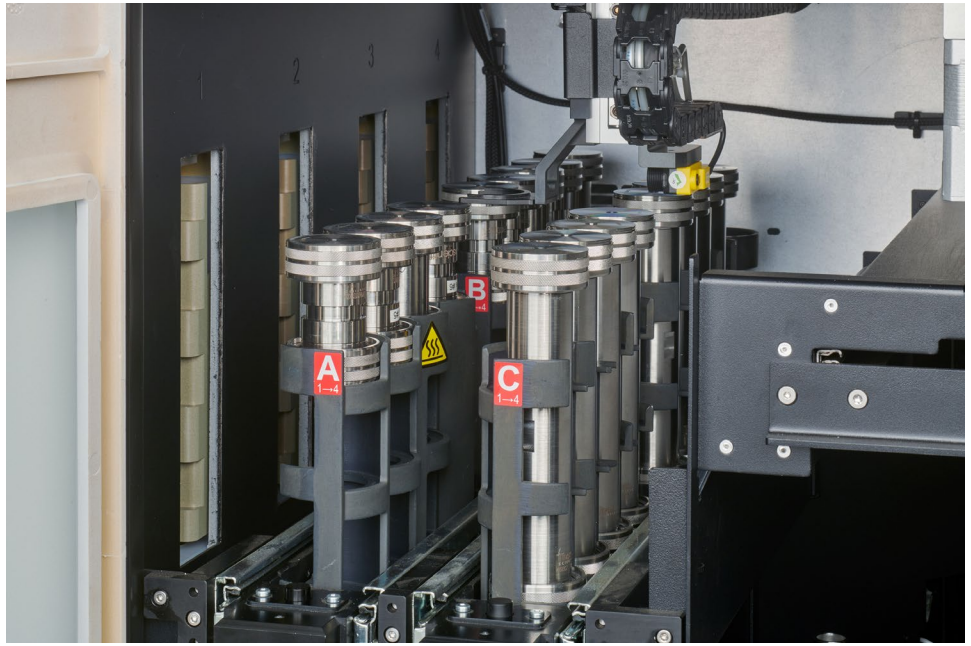
Parallel processing

The EXTREVA ASE system delivers value:

- Increases lab throughput → increases profitability
- Improves processing time → reduced risk of missing hold times and helps lab bottom line



Features:

- Parallel process of 4 samples
- In-cell clean up and moisture removal
- 16 sample autosampler – same cell size per group of 4

Extract collection and solvent evaporation

Hands free Etract evaporation:

- Needle enables N2 flow directly to the collection vessels
- Each vessel individually heated with gentle vacuum
- Combined mode helps facilitate evaporation
- Allows for true walk-away sample prep

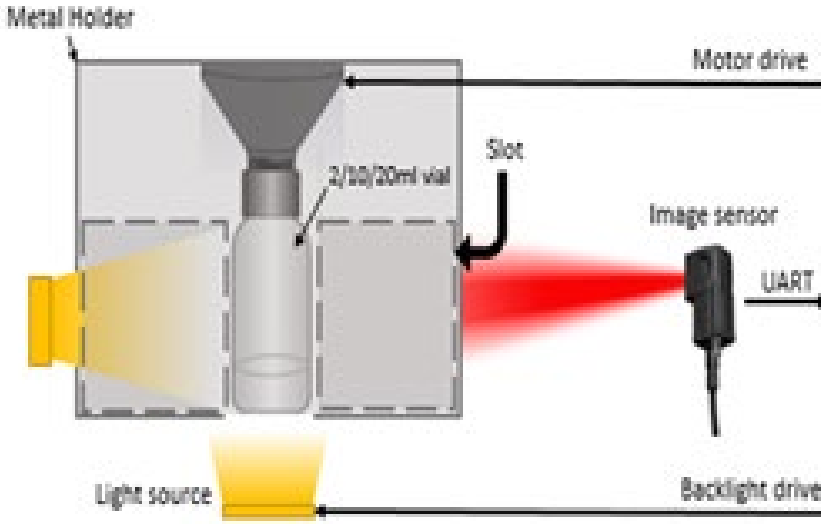
Hands free Extract collection:

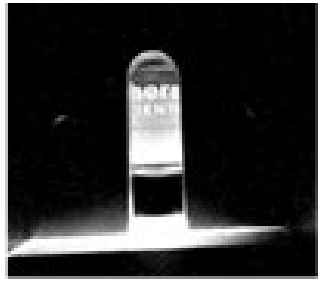
- Extracts are collected on 4 independent channels X4
- Solvent switch for compatibility with Instrument /Method.
- Use any of 3 different sized concentrators
- GC or LC vials holders are built into concentrators

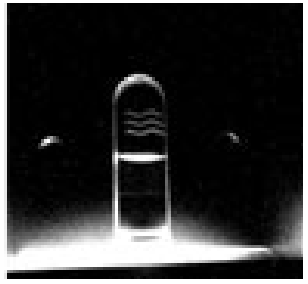
Automated end-point detection

Monitoring of evaporation no longer required

- Allows for true walk-away sample prep
- Each channel will stop at the endpoint even if the others have not reached the endpoint







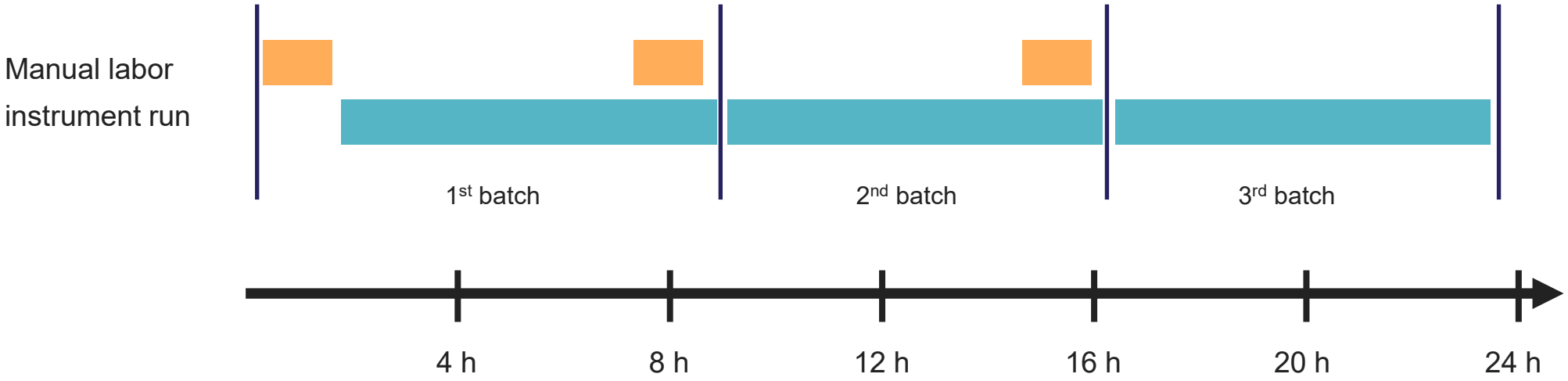
Automated end-point detection using machine learning solves this issue

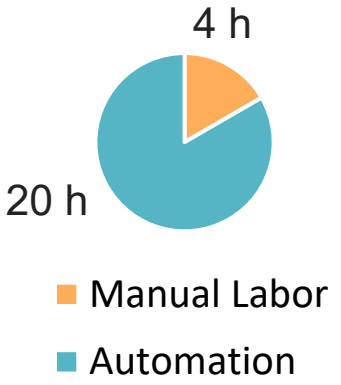
- Using an image sensor and proper backlighting allows the instrument to get a real picture of the evaporation level
- Machine learning is employed to teach the instrument to stop at the process at the desired level

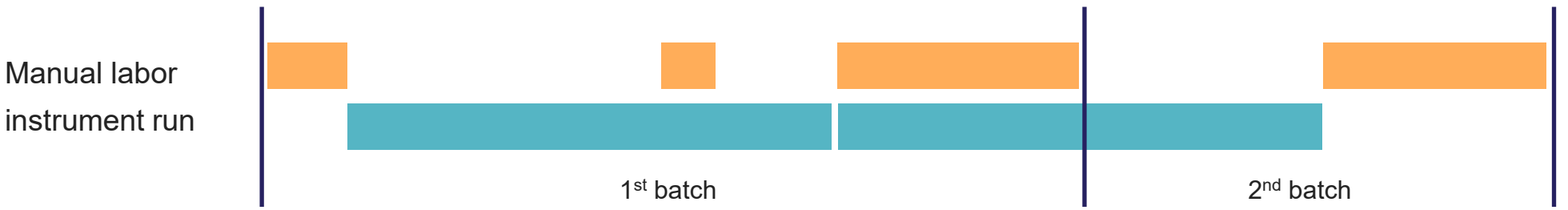
EXTREVA ASE system vs. ASE 350 system: 24-h total workflow (Extraction + Evaporation)

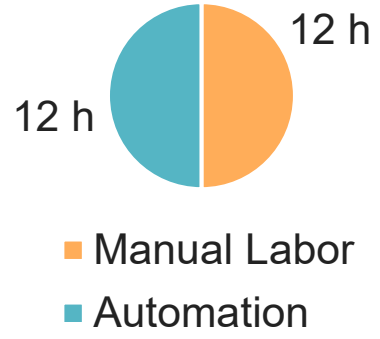
Based on 10 mL cell extraction

EXTREVA ASE system: 48 samples per day









ASE 350 system + Rocket Evaporator: 36 samples per day

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 (gas assisted extraction)	10 mL/min 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 (four samples)	~15 min (10 mL cell); ~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 & 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

TRACE™ 1310 Gas Chromatograph with Electron Capture Detector (ECD)

GC conditions	
Injector	
Injector type	Programmable Temperature Vaporizer (PTV)
Liner	Topaz liner, Split PTV, 2 mm × 2.75 mm × 120 mm
PTV ramp	75 °C to 225 °C at 5 °C/s, hold for 10 min
Injected volume	1.0 µL
GC	
Column	Rtx-CLPesticides (30 m × 0.25 mm × 0.25 µm)
Carrier gas	Helium
Flow rate	2 mL/min, constant
Oven temperature	120 °C (hold for 0.3 min), ramp to 190 °C at 4 °C/min, ramp to 300 °C at 18 °C/min (hold for 3 min)
Detector	
Detector type	Electron Capture Detector (ECD)
Detector temperature	310 °C
Makeup gas flow	15 mL/min

The EXTREVA ASE system extracts 4 samples in the same time as a single extraction instrument

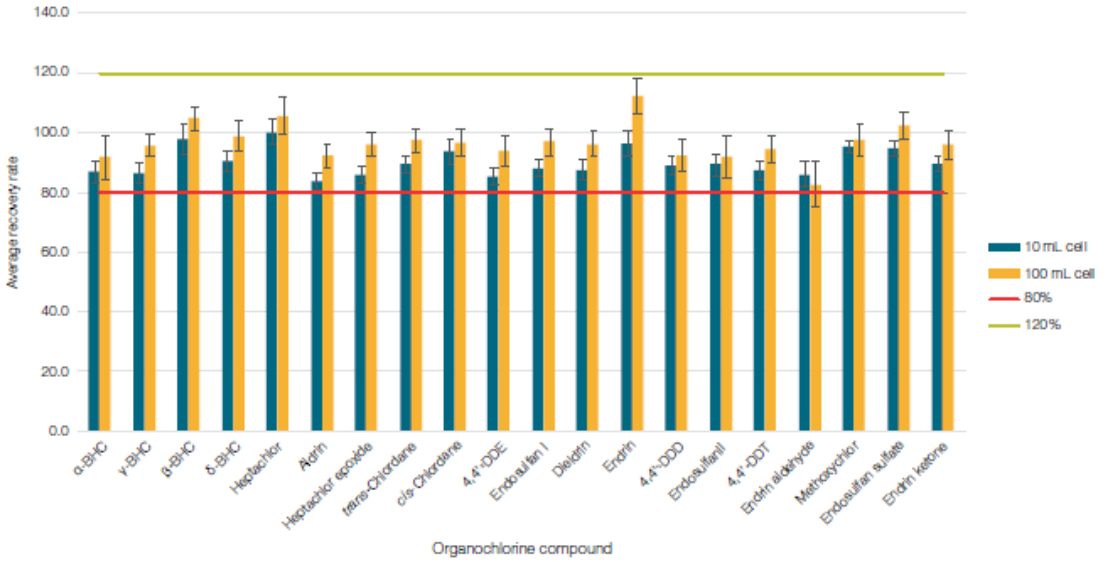
OCPs

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

Application Note AN001054

Compound	Average recovery (%) (10 mL cell, n = 12)	RSD	Average recovery (%) (100 mL cell, n = 12)	RSD
α-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%

(EPA acceptance <20% and 70 to 130%)

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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)
α-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

Polycyclic aromatic hydrocarbons (PAHs)

PAHs – 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 (gas assisted extraction)	10 mL/min per channel
Cell fill volume	50%
Solvent flow rate	1.6 mL/min (10 mL cell); 1.1 mL/min (10 mL cell); 0.75 mL/min (100 mL cell)
Extraction solvent	Acetone-methylene chloride (1:1, v/v)
Extraction volume	~26 mL (10 mL cell); ~70 mL (100 mL cell)
Pre-run rinse	10 mL, acetone-methylene chloride (1:1, v/v)
Extraction time (four samples)	~10–15 min (10 mL cell); ~20 min (100 mL cell)
Concentration	
Mode	Fixed volume
Collection bottle	100 mL vial assembly
Final fixed volume	1 mL
Rinse solvent	Acetone-methylene chloride (1:1, v/v), 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

Accelerated Solvent Extraction Conditions	
Solvent:	Methylene chloride/acetone (1:1 v/v)
Temperature:	100 °C
Static Extraction Time	5 min
Number of Static Cycles:	2
Purge Volume:	60%
Purge Time:	90 sec
Extraction Cell Size:	34 mL stainless steel
Filters:	Cellulose (30 mm)
Total Extraction Time per Sample:	20 min
Total Solvent Volume per Sample:	40 mL
Sample Size:	10 g

The EXTREVA ASE system boosts Sample Prep productivity for PAHs

4 samples extracted in the same amount of time as one on ASE 350 system

Similar solvent usage

TRACE™ 1310 Gas Chromatograph with ISQ 7000 Single Quad Mass Spec

GC-MS conditions	
Injector	
Injector type	Programmable Temperature Vaporizer (PTV)
Liner	Thermo Scientific™ LinerGOLD™, PTV Split Liner with recessed gooseneck, 2 mm ID × 120 mm, P/N 45352070
PTV ramp	65 to 300 °C at 14.5 °C/s, hold for 50 min
Injection mode	Splitless
Splitless time	1 min
Injected volume	1.0 µL
GC	
Column	Thermo Scientific™ TRACE™ TR-5MS GC Column, 30 m × 0.25 mm × 0.25 µm
Carrier gas	Helium
Flow rate	1.2 mL/min, constant
Oven temperature	60 °C (hold for 1 min), ramp to 125 °C at 25 °C/min, ramp to 240 °C at 6 °C/min, ramp to 310 °C at 3 °C/min (hold for 4 min)
Mass spectrometer parameters	
Source temperature	275 °C
Ionization	EI
Electron energy	70 eV
Transfer line temperature	280 °C
Acquisition mode	Timed-SIM

PAHs in soil

Determination of Polycyclic aromatic hydrocarbons in soils using the EXTREVA ASE Accelerated Solvent Extractor and GC-MS

Application Note AN001106

PAH compound	Certified value	Acceptance range	Average recovery and RSD (10 mL cell, n = 12)	
	µg/kg	µg/kg	Avg (n=12) µg/kg	RSD (n=12)
Naphthalene	494 ± 38	164 to 824	362	6.76
Acenaphthylene	630 ± 38	328 to 933	490	1.58
Acenaphthene	651 ± 64	141 to 1162	502	1.25
Fluorene	157 ± 19	10.7 to 303	140	3.07
Phenanthrene	290 ± 26	65.2 to 516	283	0.58
Anthracene	612 ± 51	173 to 1051	447	2.76
Fluoranthene	333 ± 25	119 to 547	349	0.95
Pyrene	202 ± 20	35.7 to 369	240	2.21
Benzo[a]anthracene	329 ± 20	158 to 500	404	1.22
Chrysene	146 ± 12	49.8 to 241	168	4.45
Benzo[b]fluoranthene	69.9 ± 4.5	32.6 to 107	79	1.74
Benzo[k]fluoranthene	266 ± 21	95.0 to 437	251	1.41
Benzo[a]pyrene	223 ± 17	83.5 to 363	206	4.34
Indeno[1,2,3-cd]fluoranthene	88.8 ± 8.3	19.5 to 158	106	6.50
Dibenz[a,h]anthracene	193 ± 16	74.4 to 312	230	1.95
Benzo[ghi]perylene	224 ± 22	44.3 to 404	274	1.49

Compound	Average recovery (%) (10 mL cell, n = 4)	RSD	Average carryover (%) (10 mL cell, n = 4)
Naphthalene	78	2.0	0.01
Acenaphthylene	85	2.3	0.01
Acenaphthene	84	2.6	0.01
Fluorene	85	2.4	0.01
Phenanthrene	92	2.4	0.01
Anthracene	98	2.1	0.01
Fluoranthene	102	3.2	0.02
Pyrene	99	2.2	0.02
Benzo[a]anthracene	104	1.8	0.02
Chrysene	100	2.2	0.02
Benzo[b]fluoranthene	101	1.2	0.02
Benzo[k]fluoranthene	100	1.4	0.01
Benzo[a]pyrene	100	2.3	0.01
Indeno[1,2,3-cd]fluoranthene	92	2.4	0.01
Dibenz[a,h]anthracene	88	2.1	0.01
Benzo[ghi]perylene	91	2.4	0.01

Blanks after high spike samples show minimal carryover

12 replicates on certified PT samples all fall within acceptance range and have %RSDs of < 7%

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Questions?