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Rapid analysis of organochlorine pesticides (OCPs) in soil samples using the EXTREVA ASE accelerated solvent extractor

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Keywords

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Goal

To develop a method for the determination of organochlorine pesticides (OCPs) in soil samples using the Thermo Scientific™ EXTREVA™ ASE™ Accelerated Solvent Extractor.

Introduction

Organochlorine pesticides (OCPs) are a class of synthetic chlorinated hydrocarbon derivatives historically utilized to protect crops, livestock, structures, and domestic spaces against insect-related damage. The diverse structural and chemical characteristics of organochlorides result in a wide spectrum of nomenclature, properties, and applications. The persistence of OCPs in environmental and food systems has been recognized as a significant health hazard to humans and animals.[1] Consequently, following the Stockholm Convention in 2001, use of several organochlorine insecticides was either banned or severely restricted, particularly within the European Union and the United States, where their application in agriculture is no longer authorized.[2]

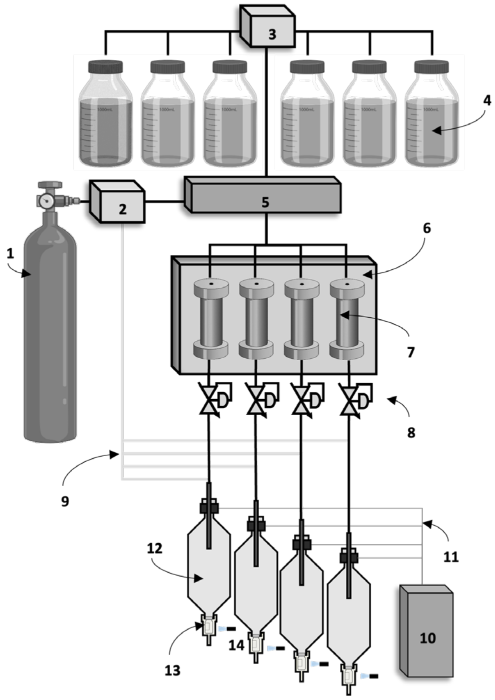
The quantification of OCPs in complex matrices, such as soil, sludge, and food, relies on a series of critical operations: solvent extraction to isolate analytes, filtration through sodium sulfate to remove moisture and particulates, evaporation to concentrate the compounds, and final volume adjustment to ensure consistency across analytical batches.[3,4] These steps are central to established workflows based on solvent extraction via Soxhlet extraction (US EPA Method 3540),[5] via sonication (US EPA Method 3550),[6] or via microwave-assisted extraction (MAE; US EPA Method 3546),[7] all of which demand significant analyst involvement. Furthermore, the complexity of sample preparation for OCP quantitation is closely tied to the analytical platform in use for detection. While cost-effective and compliant tools like gas chromatography (GC) paired with either electron capture detectors or single quadrupole MS are widely used, their lower selectivity requires precise execution of each preparatory step to ensure reliable and reproducible results.[8]

To address the labor-intensive nature of these workflows, the primary objective of new extraction methods is to reduce manual effort and shorten turnaround time (TAT)—the interval from sample preparation to instrument-ready extract. TAT is a critical metric for laboratories aiming to deliver fast and dependable results. Medium to large-scale labs often process up to 100 samples in a standard 8-hour shift, making workflow efficiency essential. Thus, emerging analytical workflows for OCPs must enable preparation of up to 24 samples per instrument in approximately four hours, helping labs meet growing demands without compromising analytical integrity.

Accelerated solvent extraction (ASE; US EPA Method 3545A)[9] is an automated extraction technique developed in the late 1990s that significantly reduced solvent usage and extraction time for nonvolatile and semi-volatile organic compounds from solids when compared to traditional methods such as Soxhlet and MAE.[2] Despite the advantages of this system, extractions are performed one at a time and require an operator to transition the extract from the extraction phase to the evaporation phase. The operator must manually control the evaporation and reconstitution processes to achieve an accurate final extract volume for precise quantitation of the target analytes.

To overcome persistent limitations in sample preparation workflows, in 2022, Thermo Fisher Scientific introduced the EXTREVA ASE system, a fully integrated instrument that automates both extraction and evaporation (Figure 1). The system combines gas-assisted dynamic solvent extraction (GA-dASE), parallel ASE, and endpoint level detection (Figure 1; objects 5, 6, and 14) to deliver a streamlined, end-to-end process with minimal user input. During operation, heated solvents, selectable from six reservoirs (Figure 1, object 4), are delivered with nitrogen gas into stainless-steel sample cells (Figure 1, object 7) housed in a temperature-controlled oven (Figure 1, object 6). The system operates at approximately 200 psi (~14 bar), regulated by a back pressure valve (Figure 1, object 8), initiating GA-dASE. Once the pressure threshold is reached, excess extract is routed through dedicated channels into collection bottles fitted with 2 mL vial adaptors (Figure 1, object 12). The EXTREVA ASE system supports simultaneous extraction of up to four solid or semi-solid samples using a multi-cell oven and enables batch processing of up to 16 samples via four independent trays. Each tray holds four sample cells, allowing sequential extraction across the batch. Following extraction, the system transitions directly into automated evaporation, using controlled heating, vacuum (Figure 1, object 10), and gas flow across the container surface to efficiently remove excess solvent. Endpoint level sensing (Figure 1, object 14) ensures precise volume control, allowing samples to be evaporated to dryness or concentrated to a defined volume, ready for downstream GC or LC analysis. When compared to ASE, Soxhlet, and MAE, the EXTREVA ASE system significantly reduces labor needs and increases operator productivity.[10–13]

Despite excellent performance, we identified several further improvements to reduce batch TAT when using the EXTREVA ASE system. By executing multiple instrument initial preparation steps simultaneously, the time it took to start the extraction process could be reduced drastically. Furthermore, optimization of the extraction conditions under dynamic gas assistance allowed for exhaustive recoveries with significant extraction solvent volume reduction. This improvement facilitated shorter sequence completion times, as there was less solvent to evaporate. To improve method speed, evaporation hardware was also enhanced to quicken the enrichment process. A more powerful mass flow controller was used in this new version of the EXTREVA ASE system, allowing for higher gas flow rates during the evaporation step. Similarly, improvements to end-point level sensing were implemented for faster monitoring and, consequently, accurate extract volume even when performing faster evaporations. Herein, we present the results of the determination of 20 OCPs in soil samples, showing excellent extraction recoveries, minimal carry-over in blank samples after extracting a heavily concentrated sample, and reproducibility of short TATs (<4 h for 24 samples per instrument).



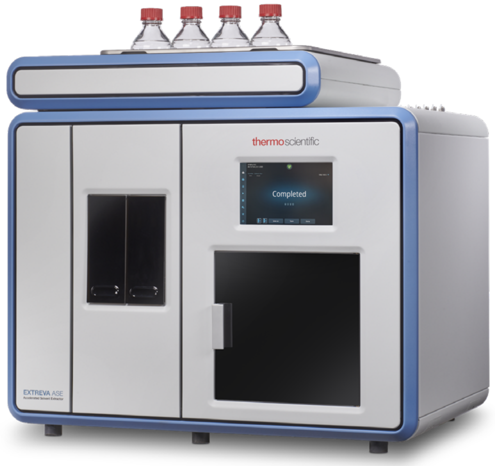


Figure 1. Schematic representation of GA-dASE apparatus. List of parts as follows: 1. Nitrogen cylinder. 2. Gas manifold. 3. Solvent pump and mixing valves. 4. Solvent bottles. 5. Gas/liquid manifold and mixing chamber. 6. Sample cells-oven. 7. Sample cells. 8. Back pressure valve. 9. Evaporation line. 10. Vacuum pump. 11. Vacuum path. 12. Evaporation bottle with adaptor for 2 mL vial. 13. Vial for GC/LC analysis. 14. Level sensing system for unattended endpoint evaporation.

Equipment and consumables

- EXTREVA ASE system (P/N B51004598)
- Thermo Scientific™ TRACE™ 1310 Gas Chromatograph with Electron Capture Detector
- Thermo Scientific™ Dionex™ Cellulose Filter (P/N 056780)
- Thermo Scientific™ Dionex™ ASE™ 10 mL stainless steel extraction cells (P/N 068087)
- Fisherbrand™ amber glass vial, 9 mm screw thread, graduated, compatible with 9 mm screw thread caps (P/N 03-377B)
- Diatomaceous Earth (DE) Dispersant for ASE, 1 kg Bottle (P/N 062819)
- Sand, Ottawa, Fisher Chemical™ (P/N S23-3)
- Sigma Aldrich™ Clean Loamy Soil (P/N CLNLOAM6-100G)

Reagents and standards

- Acetone, Optima™, for HPLC and GC, Fisher Chemical™ (P/N A929-4)
- Hexane, Optima for HPLC and GC, Fisher Chemical (P/N H303-4)
- Restek™ Organochlorine Pesticide Mix (P/N 32415)
- Restek™ Pesticide Surrogate Mix (P/N 32000)
- Restek™ Pesticide Internal Standard (P/N 32091)

Extraction, concentration, and measurement

The pesticide surrogate mix (decachlorobiphenyl and 2,4,5,6-tetrachloro-m-xylene) was diluted with hexane to produce a stock solution. Calibration standards with concentrations of 0.01, 0.02, 0.05, 0.1, and 0.2 µg/mL were prepared by diluting the stock solution with hexane. A cellulose filter was placed on top of a 10 mL cell body, and the end cap was hand-tightened. Two grams of clean loam soil were mixed in a glass beaker with an equal amount of diatomaceous earth (ASE Prep DE). The resulting mixture was carefully poured into the extraction cell and spiked with the appropriate amount of pesticide standard mix. Any empty volume was filled with DE while lightly tapping. Another cellulose filter was placed on top of the cell body, and the second end cap was hand-tightened. The DE acts as a dispersant and plays a key role in preventing sample compaction during the compression phase, ensuring efficient solvent contact with the sample.[14]

The instrument was programmed according to the conditions reported in Table 1. Before proceeding to the extraction of the samples, the system was rinsed with the extraction solvent (hexane-acetone 1:1, v/v). Hexane was used during evaporation as a rinse solvent, and 3 mL was added before the final evaporation step to rinse the collection bottle walls. After concentration, the samples were added with internal standard (25 µL of a solution of pentachloronitrobenzene with a concentration of 2 µg/mL in hexane-acetone 1:1, v/v) and analyzed by GC-Electron Capture Detector (GC-ECD). The GC-ECD conditions are summarized in Table 2.

Figure 2 shows the chromatogram of a 0.05 µg/mL OCP standard. All peaks were well-resolved (Rs >2.0), and the total analysis time is less than 27 minutes. A five-point calibration curve was used (0.01, 0.02, 0.05, 0.1, and 0.2 µg/mL). Calibration curves were created by plotting concentrations versus peak area ratios of analyte to internal standard. A linear regression was employed for quantification. The percentage errors between the measured amount and the true amount of each calibration point were less than 10% for all analytes.

Extraction and evaporation

For recovery studies for the extraction and evaporation workflows, a 250 µg/kg fortified soil sample was used. 10 mL cells were used, and the conditions are reported in Table 1. The results are summarized in Table 3 and Figure 3. All recoveries were between 80% and 121%, thus demonstrating the high extraction efficiency and the minimal loss of the most volatile compounds like naphthalene. These results met the recommended acceptance criteria of 70–130% from the US EPA for all compounds. The relative standard deviation (RSD) was below 20% for all compounds, suggesting good channel-to-channel and run-to-run reproducibility for extraction and evaporation.

Table 1. Extraction and evaporation conditions for the EXTREVA ASE system

Extraction	
Cell type	Stainless steel
Cell size	10 mL
Oven temperature	100 ˚C
Purge time	30 s
Nitrogen flow (gas-assisted extraction)	20 mL/min per channel
Cell fill volume	50%
Solvent flow rate	0.5 mL/min
Extraction solvent	Acetone-Hexane (1:1, v/v)
Extraction volume	~12.5 mL (~7.5 mL real volume)
Pre-run rinse	10 mL, Acetone-Hexane (1:1, v/v)
Extraction time (4 samples)	5 min
Enrichment	
Mode	Fixed volume
Collection bottle	250 + 2 mL vial assembly
Final fixed volume	1 mL
Rinse solvent	Hexane, 3.0 mL
Evaporation	40 ˚C
Nitrogen flow rate	200 mL/min per channel (800 mL total)
Vacuum	8 psi (420 torr)

Table 2. GC-ECD system conditions

Injector	
Injector type	Programmable temperature vaporizer (PTV)
Liner	Topaz Liner, Split PTV, 2 mm x 2.75 x 120
PTV ramp	65 ˚C to 300 ˚C at 14.5 ˚C/s, hold for 50 min
Injection mode	75 ˚C to 225 ˚C at 5 ˚C/s, hold for 10 min
Injected volume	1.0 µL
GC	
Column	Rtx-CL Pesticides (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)
Evaporation	40 ˚C
Nitrogen flow rate	200 mL/min per channel (800 mL total)
Vacuum	8 psi (420 torr)
Detector	
Detector type	Electron capture detector
Detector temperature	275 °C
Makeup gas flow	15 mL/min

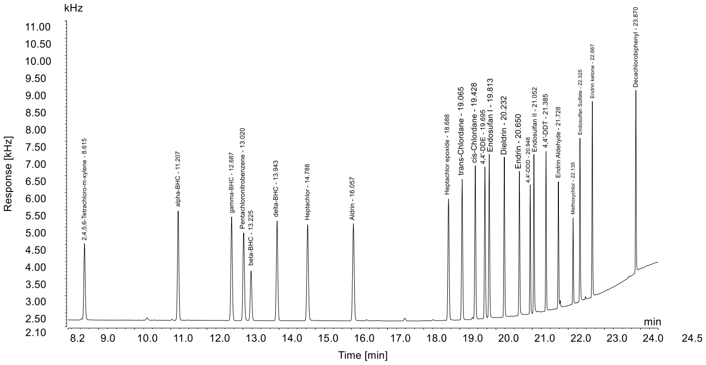


Figure 2. GC-ECD chromatogram of a 0.05 µg/mL standard solution

Carryover

Compared to other extraction methods such as MAE and Soxhlet, which typically use a higher solvent-to-sample volume ratio, the EXTREVA ASE system operates with a lower solvent volume per sample. This reduced solvent usage raises potential concerns about carryover and cross-contamination.

To investigate these risks, a heavily fortified soil sample (500 µg/kg) was extracted and concentrated under the conditions outlined in Table 1. A second extraction was then performed using a fresh cell packed with Ottawa sand under the same conditions. Between the two extractions, each flow path channel was rinsed with 10 mL of hexane. The results of the carryover test are shown in Table 4. The carryover percent was calculated by comparing the peak area ratio of the analyte between the spiked samples and the blanks. Carryover was less than 0.6% for all analytes. These results demonstrate that the rinse implemented between the extractions effectively minimized carryover and cross-contamination. Moreover, the rinse volume can be adjusted to accommodate different sample sizes, matrices, and concentrations.

Table 3. Average recoveries for the 250 µg/kg spike level

Compound	Average recovery % (10 mL cell , n = 4)	RSD %
α-BHC	80	10
γ-BHC	87	10
β-BHC	112	13
δ-BHC	104	14
Heptachlor	105	4
Aldrin	88	11
Heptachlor epoxide	106	11
trans-Chlordane	110	7
cis-Chlordane	115	10
4,4’-DDE	113	11
Endosulfan I	116	12
Dieldrin	117	6
Endrin	122	10
4,4’-DDD	113	8
Endosulfan II	120	11
4,4’-DDT	107	10
Endrin aldehyde	110	11
Methoxychlor	117	15
Endosulfan sulfate	117	13
Endrin ketone	121	11

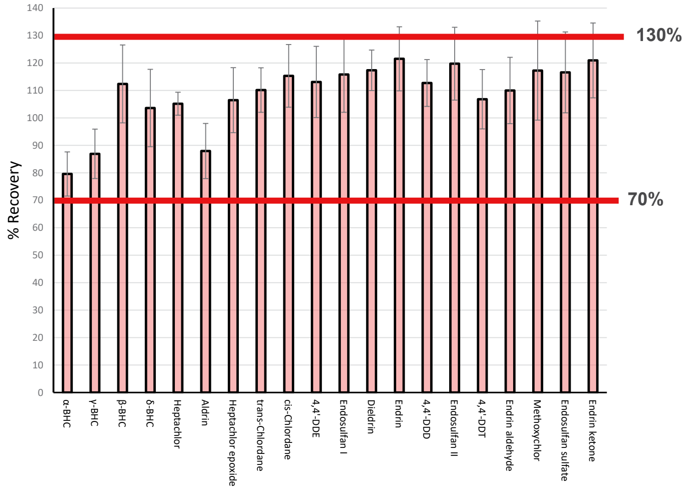


Figure 3. Average recoveries for OCPs from soil samples

Table 4. Average carryover from soil samples with high spike level

Compound	Average carryover % (10 mL cell, n = 4)
α-BHC	0.00
γ-BHC	0.00
β-BHC	0.09
δ-BHC	0.44
Heptachlor	0.00
Aldrin	0.00
Heptachlor epoxide	0.26
trans-Chlordane	0.29
cis-Chlordane	0.29
4,4'-DDE	0.25
Endosulfan I	0.32
Dieldrin	0.24
Endrin	0.31
4,4'-DDD	0.05
Endosulfan II	0.08
4,4'-DDT	0.06
Endrin aldehyde	0.06
Methoxychlor	0.00
Endosulfan sulfate	0.00
Endrin ketone	0.04

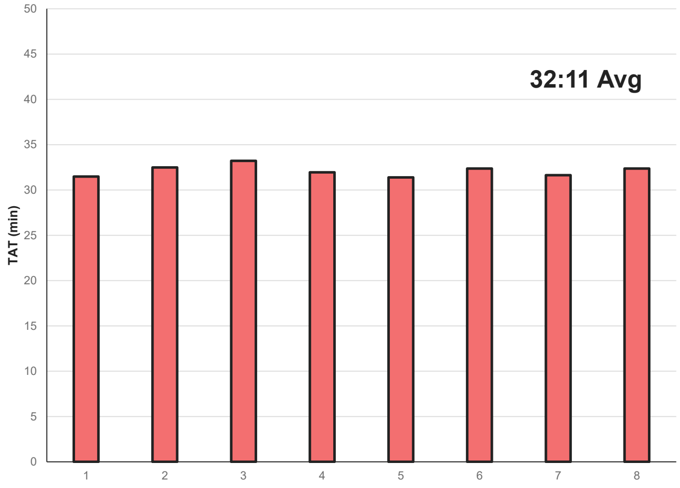


Figure 4. TAT reproducibility towards determination of OCPs in soil samples

TAT improvements

Eight replicate tests of the method have not only confirmed the precision of the process with a RSD under 2.0%, but they have also highlighted its rapidity, with an average analysis time of approximately 32 minutes (Figure 4). This rapid turnaround enables an expedited response to environmental monitoring and contamination assessments. Compared to the previous version of the instrument, the TAT has been reduced by nearly fourfold, setting a new standard for efficiency and performance.

Thermal degradation test

Because soil samples are typically extracted at elevated temperature (>100 °C), thermally labile analytes may partially degrade during the extraction phase. Out of the 20 OCP analytes used in this study, endrin and 4,4'-DDT are the least thermally stable compounds.[14] Therefore, soil samples spiked with endrin and 4,4'-DDT at 25 µg/kg were extracted to evaluate the presence of thermal degradation products. Table 5 shows the thermal degradation results when the soil sample was extracted at 100 °C and 150 °C. All breakdown percentages were well below the 15% criteria suggested by the US EPA.[3] For endrin, a 3.1% breakdown occurred in the GC injection port, as evidenced by injecting a 0.05 ppm QC standard. DDT breakdown was very low for both the soil samples and the QC standard. In summary, the newly developed gas assisted extraction method employed by the EXTREVA ASE system does not cause significant breakdown for OCP analytes during the extraction process.

Table 5. Endrin and DDT breakdown at different extraction temperatures

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

Conclusion

Offering the benefits of automation and an easy load-and-go start process, the EXTREVA ASE system saves time, reduces errors and solvent usage, enables unattended extractions, and increases analytical throughput. By refining the extraction and evaporation processes, we streamlined the analytical workflow, ensuring a more efficient approach to sample preparation and analysis. The extraction and enrichment workflow presented offers a fast and accurate analysis of 20 OCPs in less than 35 minutes with high reproducibility. The EXTREVA ASE system is a powerful tool for environmental monitoring and assessment, enabling rapid and reliable detection of OCPs in soil samples according to US EPA Method 3545A.[9]

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