



PAL System μ SPE

Info and parts

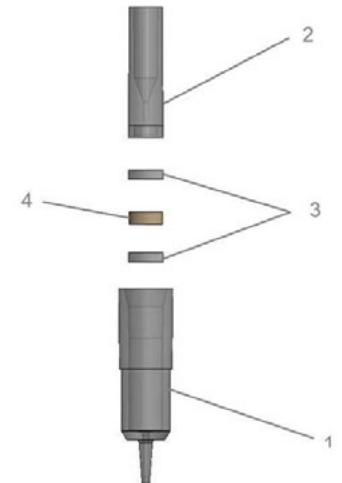
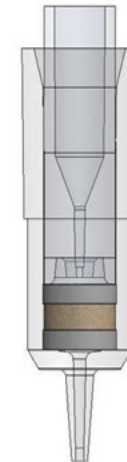
PAL SYSTEM
Ingenious sample handling

PAL System μ SPE – New cartridge

Key features of new PAL System μ SPE cartridge

The outer part (1) provides a higher capacity and flexible volume for filter disks (3) and the sorbent material (4). The bottom outlet is designed to penetrate pre-slit septa and to deliver directly to LC injection ports. The inner plunger (2) provides critical functionality with the compression of the sorbent/filter layers, and a precise needle guide for safe and upright transport.

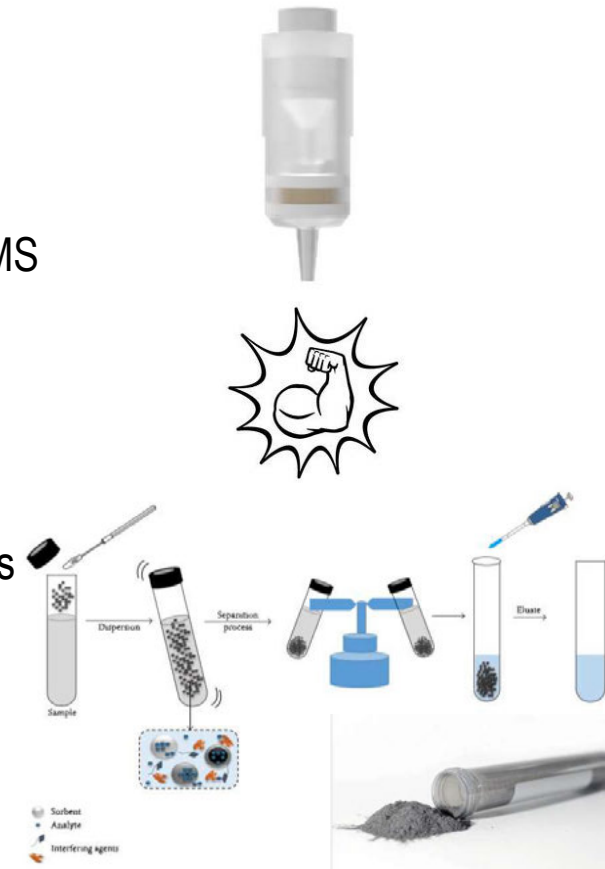
- For all PAL3 RTC Systems
- Novel septum-less cartridge design using CTC patented sealing technology – **Gauge 22 flat point style syringes required**
- High pressure rating (up to 15bar)
- Sorbent Capacity: 10 to 100 mg packing possible (depending on density)
- Chemically inert materials used and free from leachable
- Designed to penetrate pre-slit septa of vials
- Designed to elute directly into PAL System liquid injection ports
- Quality control steps for each cartridge during manufacturing guarantee highest reproducibility (batch to batch and batch internal variations are minimized).
- **SW: Supported by Chronos 5.5.0 and PAL Method Composer (PMC) 1.5 for FW 4.x**
- **Custom Scripts for QuEChERS workflow for FW 2.x, 3.x and 4.x available**



PAL System μ SPE – New cartridge

Benefits of μ SPE to dSPE

- Shorter clean-up time compared to dSPE
- Less maintenance and higher productivity due to significantly better clean-up performance compared to dSPE
- Automated clean-up and injection or offline clean-up to serve multiple LCMS and GCMS systems
- Significantly less solvent consumption
- Significantly less time to process generated data
- No evaporation needed to avoid uncontrolled decomposition and loss of analytes
- Precisely controlled loading, washing and elution by adjustable flow rates and volumes
- Automation friendly solutions
- Proven technology and developed with Key Opinion Leaders



PAL System μ SPE – New cartridge

Benefits of the new PAL System μ SPE cartridge

- Multiple elution steps possible thanks to septum-less design
- Higher sorbents capacity for broader application ranges
- Higher productivity thanks to shorter cycle times (up to 500 μ L/ min flow rates possible)
- Minimized delay volume (20 μ L)
- Development kit for customized sorbent materials packing available



Clean-up of QuEChERS extracts with μ SPE

For GC-MS and LC-MS analysis

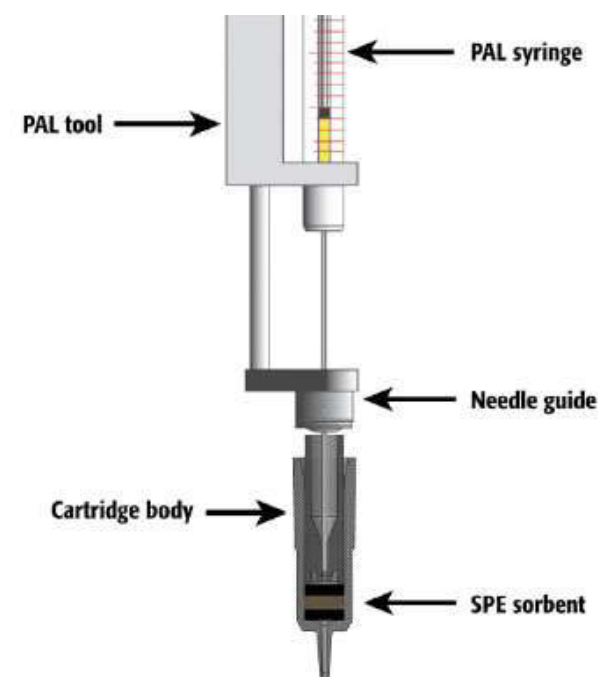
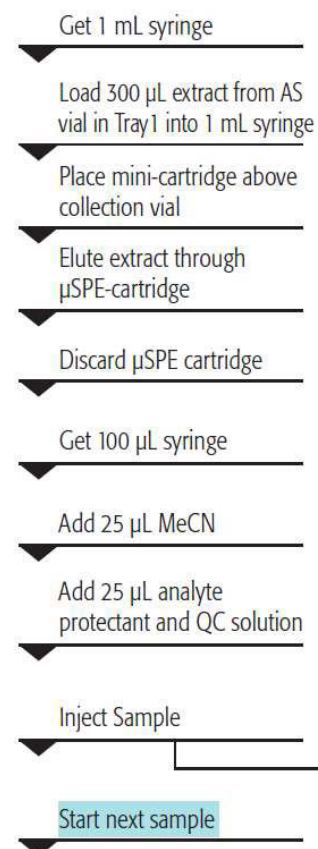
- The μ SPE clean-up achieves high quality results for diverse type of analytes and foods (apple, kiwi, carrot, kale, orange, black olive, pork loin, salmon, avocado).
- μ SPE provides better cleanup than dispersive-SPE (d-SPE) that is often incorporated in QuEChERS sample preparation.
- The automated μ SPE step takes 8 min per sample.



FOOD SAFETY



ENVIRONMENTAL



Clean-up of QuEChERS extracts with μ SPE

Publication with new PAL μ SPE by Key Opinion Leaders



Steven Lehotay; US Department of Agriculture, 2022



Evaluation of a septumless mini-cartridge for automated solid-phase extraction cleanup in gas chromatographic analysis of >250 pesticides and environmental contaminants in fatty and nonfatty foods

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latter substituting for CarbonX used in the ITSP product. The septumless μ SPE mini-cartridge employs a different gripping mechanism with the syringe needle that allows leak-free operation at higher flow rates (e.g. 10 μ L/s), whereas the ITSP design is limited to 2 μ L/s. Based on cleanup and analyte elution profiles, the extract load volume and flow rate was increased in μ SPE for QuEChERSER from 300 μ L at 2 μ L/s to 500 μ L at 5 μ L/s, which improved accuracy of results, sped the cleanup step, and obviated the need for micro-vial inserts in the receiving vials. The new design also reduced both the amount and consistency of dead (void) volumes in the mini-cartridges from 83 ± 14 μ L to 52 ± 7 μ L for 200-600 μ L load volumes.

Voice of customer to new μ SPE cartridge

Reduced dead volumes

Improved analytical performance

Improved robustness, leak free operation

Improved speed

4. Conclusions

The new μ SPE mini-cartridge design evaluated in this study improved upon analytical performance while allowing faster cleanup with reduced chance of leaks or other common failures associated with automation. No stoppages in automation were observed

The reduced dead (void) volume of the μ SPE mini-cartridges in comparison to the ITSP design not only allowed greater load volumes but also provided excellent elution consistency. The sorbent mix combining anh. MgSO_4 , PSA, C18 and GCB continued to provide exceptional cleanup for extracts from fatty and nonfatty matrices alike. Although GCB retained some co-planar analytes, such

CTC Analytics Poster @ LAPRW Panama, 2023

Characterization of the New PAL Micro-SPE Cartridge for Pesticides Extract Clean-up

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Presented at the LAPRW 2023 in Panama
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Overview

Since more than ten years micro-SPE (μ SPE) emerged as a micro-method for sample preparation and clean-up in food safety, proteomics, forensic, environmental and analysis. Applications are wide-ranging and cover drugs, environmental contaminants, and in particular, the QuEChERS extract clean-up in multiresidue pesticide analysis. The automation of the μ SPE sample preparation steps led to the desired increase in sample throughput, uniformity of the used sorbent materials for food commodities, and the potential for the online hyphenation with GC-MS and LC-MS instrumentation.

With the increasing demand and the use of automated μ SPE workflows on PAL Systems, the requirement for extended functionality evolved due to mechanical and also analytical limitations of the initially employed ITSP micro-SPE cartridge (ITSP Solutions Inc., Hanwell, GA, USA). With the new PAL μ SPE cartridge CTC Analytics introduced a novel septumless cartridge design dedicated to an extended application range and a reliable high throughput automation. The evaluation of the new cartridge design is presented with results for pesticides analysis from leading laboratories.

Introduction

Design of the new PAL μ SPE cartridge

The novel PAL μ SPE cartridge is septumless and consists of two parts only, as shown in Fig. 1. The polymer material used is chemically inert and free from leachables. It is compatible with MeCN, EtOAc, MeOH, DCM, hexane and aqueous pH of 1 to 12. The outer dimensions are 35×2 mm in height and 8.5 mm outer diameter.

The outer shell provides a high capacity and flexible volume for sorbent materials separated by filter disks. There is a labelling option for the cartridge type for GLP purposes.

The bottom nozzle is designed to penetrate pre-seal septa of vials and also delivers directly to LC injection ports.

The inner body provides critical functionality with a predefined and reproducible compression of the sorbent/filter layers, and a precise needle guide for safe and always upright positioning. The move of cartridges on the PAL System can be achieved by syringe needle or pipette tip transport. Of key importance here is the resulting high pressure resistance and the leak-free seal against a syringe needle of gauge 22 with flat tip for increased extract load speeds and volumes. Instead of sealing through a septum, it is sealed via a constant force at a needle seat (analogous to an LC injector). This patented technology allows flow rates of up to 500 μ L/min with the CTC μ SPE cartridges.

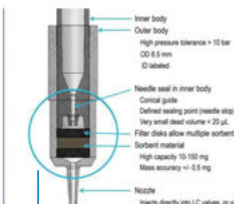


Figure 1. The novel PAL μ SPE cartridge (cross section)



Figure 2. Principle of the PAL μ SPE operation

Evaluation

The novel PAL μ SPE cartridges were evaluated in routine laboratories for pesticides analysis. A typical sorbent mixture often used for clean-up of QuEChERS extracts consists of 20 mg anh. MgSO_4 , 12 mg each of C18 and PSA, and 1 mg GCB (Carbograph-1).

Load volume and flow rate for GC-MS analysis

In the recent publication Nicolás Michlig and Steve Lehotay of the US DoA in Wyndmoor, PA, USA [1] reported about the optimization and limits of the QuEChERS extract load volume and load speed with respect to the cleanup performance.

EURL for Cereals and Feeding Stuff in Lyngby, Denmark, for several difficult food matrices [2]. Table 1 shows the achieved cleanup efficiencies in scavenging mode (% matrix removal) and the 70 to 120% recovery rates.

The custom prepared cartridges with Z-Sep and EMR sorbents gave the best results with regard to recoveries. C18 and C18 showed the poorest cleanup. For insecticide analysis the composition 12 mg C18/ 12 mg PSA/ 1 mg GCB showed the most effective cleanup efficiency.

In addition to the μ SPE cleanup the automated preparation of calibration dilutions and the addition of QC18 standards before injection have been integrated into the automated workflow achieving excellent quantitative precision.

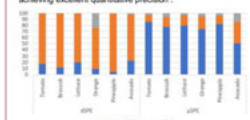
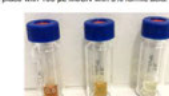


Figure 3. LC-MS matrix effects observed with dSPE and μ SPE cleanup [2]

Matrix effect reduction for LC-MS analysis

The reduction of matrix effects in LC-MS using dSPE and μ SPE and the cleanup performance for polar compounds in QuEChERS extracts from tomato, orange, rice, avocado and black tea matrices were compared by Lorena Mancano Sánchez and Florencia Javiel et al. at the EURL for Pesticide Residues in Fruit & Vegetable in Almería, Spain [3].

The PAL μ SPE cartridges comprised 45 mg MgSO_4 /PSA/C18/ Carbograph-1 (20/12/12/1), 100 μ L of MeCN conditioned the cartridge before the QuEChERS extract load of 200 μ L. Elution took place with 100 μ L MeCN with 5% formic acid.



Patented sealing,
Requires Gauge 22, FL needles

Clean-up of QuEChERS extracts with μ SPE

Publication with new PAL μ SPE by Key Opinion Leaders

Mette Erecius Poulsen; National Food Institute Technical University DK, 2021



Evaluation of the automated micro-solid phase extraction clean-up system for the analysis of pesticide residues in cereals by gas chromatography-Orbitrap mass spectrometry

Elena Hakme^a, Mette Erecius Poulsen
^aNational Food Institute, Technical University of Denmark, Søborg, Denmark

4. Conclusion

The main benefit of μ -SPE is the increase in laboratory productivity and sample throughput, with an associated reduction of labor. The best strategy for accurate pesticide determination and quantitation is the use of semi-procedural matrix calibration. The automated μ -SPE system could be used as a standalone system, or it could be coupled to a high-sensitivity analytical instrument. In the latter case, the addition of some features, such as a thermo-

PAL SYSTEM
Ingenious sample handling

Voice of customer to new μ SPE

- Increase productivity – shorter cleanup time
- Robustness – consistent data,
- Robustness - reduced qualitative and quantitative errors
- Flexible (standalone or in line)
- One method for all, simplify multiresidue methods
- Full automation

Amadeo Fernandez-Alba; European Union Reference Lab – University of Almeria, 2023



Evaluation of automated clean-up for large scope pesticide multiresidue analysis by liquid chromatography coupled to mass spectrometry

Lorena Manzano Sánchez, Florencia Jesús, Carmen Ferrer, M. Mar Gómez-Ramos, Amadeo Fernández-Alba^a
^aEuropean Union Reference Laboratory for Pesticide Residues in Fruits & Vegetables, Agrifood Campus of International Excellence (ceiA3), Ctra. Sacramento S/N, University of Almeria, La Cañada de San Urbano, Almería 04120, Spain

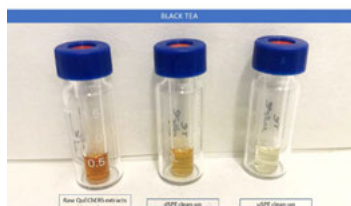
In general, the μ SPE method provides recoveries that are very similar to those obtained with manual clean-up because the extraction step is the same for both approaches. Of great inter-

fective at avoiding inconvenient trapping processes. In conclusion, automatic μ SPE avoids the qualitative and quantitative errors that are produced when dSPE is applied.



4. Conclusions

The use of automated μ SPE clean-up reduces the laboratory workflow and allows increased sample throughput in routine analysis by 30%. Moreover, as only a single clean-up is employed equally for all commodities, a simplify multiresidue method is obtained with the important benefits with method application. Very high homogeneity is typically obtained in the calibration curves avoiding typical quantitation errors. Instrument maintenance is



Each row, from left to right: raw QuEChERS extracts without clean-up, QC extract (dispersive clean-up) and μ SPE extract (columnar).

Agilent Technologies, ASMS Poster, 2022



PAL System μ SPE – New cartridge

QuEChERS Cleanup Workflow



QuEChERS follows the scavenging mode: Matrix is retained, and analytes eluted.







[Movie PAL \$\mu\$ SPE QuEChERS Cleanup](#)

μ SPE has two typical modes of operation.

1. Enrichment mode or
2. Scavenging mode

PAL System μ SPE – New cartridge



Portfolio and Applications

CTC part number	Description	Image
μ SPE cartridges for pesticide analysis / QuEChERS		
uSPE-GCQuE1-45-V	<p>PAL System uSPE Cartridge - GC QuEChERS sorbent mix 1 - 45mg C18 EC (12 mg), PSA (12 mg), MgSO₄ (20mg), GCB (1mg) 108 pcs type 01-05B Requires 4.x or higher*</p>	 
uSPE-GCQuE2-44-V	<p>PAL System uSPE Cartridge - GC QuEChERS sorbent mix 2 - 44mg C18 EC (12 mg), PSA (12 mg), MgSO₄ (20mg) 108 pcs type 01-19A Requires 4.x or higher*</p>	 
uSPE-GCQuE3-45-V	<p>PAL System μSPE Cartridge -GC QuEChERS sorbent mix 3 – 45mg PSA (6.43mg), MgSO₄ (38.57 mg) 108 pcs type 01-14A Requires FW 4.x or higher*</p>	 

* Scripts for FW 3.x or 2.x available on request

PAL System μ SPE – New cartridge

Portfolio and Applications





CTC part number	Description	Image
μ SPE cartridges for pesticide analysis / QuEChERS		
uSPE-LC-QuE1-30-T	PAL System μ SPE Cartridge -LC QuEChERS sorbent mix – 30mg C18 EC (21 mg), Z-Sep (8 mg), GCB (1mg) 96 pcs in blister type 01-03A Requires FW 4.x or higher*	
μ SPE cartridges for custom loads – Development Kit		
uSPE-Dev-Kit-100	PAL System uSPE Cartridge - Development Kit - without resin 100 pcs type 01-09 Requires 4.x or higher *	



* Scripts for FW 3.x or 2.x available on request

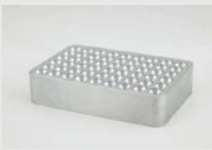

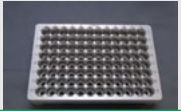


PAL System μ SPE – New cartridge

Accessories for μ SPE

CTC part number	Description	Image
μSPE hardware kits		
PAL3-HW-uSPE54	<p>PAL3 Kit for PAL System uSPE cartridges with smart syringes.</p> <p>1 pc uSPE Liquid Tool D8/57 with Needle Guide for μSPE cartridges</p> <p>2 pc Liquid smart syringe 1000μL (SF1000-57-T-22-FL), gauge 22, flat tip</p> <p>1 pc Trayholder</p> <p>2 pc VT54 rack (PAL3-Rack-VT54)</p> <p>1 pc uSPE Cartridge Tray, 54 position, aluminum</p> <p>1 pc uSPE Elution Tray Cover, 54 position aluminum</p> <p>1 pc Waste Receptacle with drain</p> <p>4 pc PAL System uSPE cartridges for initial installation, 3-point teaching</p> <p>USB stick with installation guide and scripts (FW 2.x, 3.x and 4.x)</p>	
μSPE Cartridge Kits & Vials		
Vial-1.5ND-CG-100	PAL System Vial 2CV, 1.5ml Clear Glass with Label, designed for the PAL Autosampler. 12x32mm, 1st Class Hydrolytic Glass, fits ND9 Screw Caps, Pk of 100 Pcs	
Vial-0.3ND9-PP-100	PAL System Micro-Vial 2CV, 0.3mL Clear PP, designed for the PAL Autosampler. 12x32mm, with conical insert, fits ND9 Screw Caps, Pk of 100 Pcs	
Cap-ND9-ST-SP10Sb-100	PAL System Screw Cap 2CV, designed for the PAL Autosampler. ND9, Magnetic, Gold, Silicone/PTFE/Starburst-Slitted Septa 1.0mm, Pk of 100 pcs	
Cap-ND9-PP-SP10S-100	PAL System Screw Cap 2CV, designed for the PAL Autosampler. ND9, Silicone/PTFE/I-Slitted Septa 1.0mm, Pk of 100 pcs	



PAL System μ SPE – New cartridge

CTC part number	Description	Image
μ SPE Accessories: Racks and holders		
PAL3-Vial-Lock-uSPE54	μ SPE Elution Tray Cover to lock 2 mL Vials in place, matching VT54 Rack For use with uSPE Cartridges	
PAL3-Wasterecept-uSPE	μ SPE Waste Receptacle to hold uSPE Cartridge Tray 54 or 96 positions Includes waste adapter for mounting on bottom of tray holder	
PAL3-Rack-Cart-uSPE54	Cartridge Tray to hold 54 μ SPE cartridges - also used for optional cartridge conditioning	
PAL3-Rack-Cart-uSPE96-B	Cartridge Tray to hold 96 μ SPE cartridges in blister	
PAL3-Rack-Cart-uSPE96	Cartridge Tray to hold 96 μ SPE cartridges	



NEW!

PAL3-Rack-Cart-uSPE96-9

uSPE Cartridge Tray, 96 Positions, 9mm

