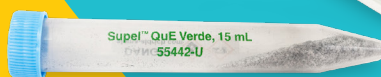
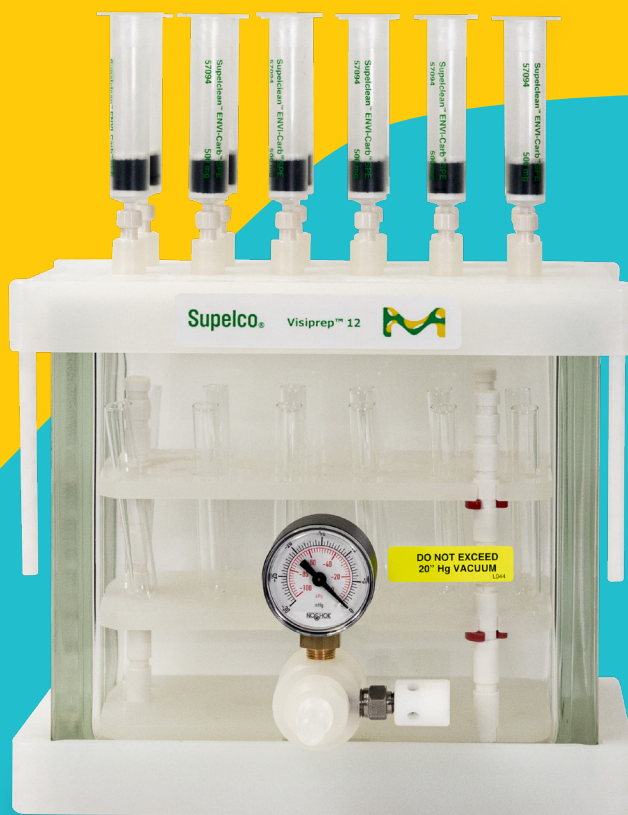


MERCK

# Solid Phase Extraction Products

Improve Sensitivity,  
Increase Throughput  
and Ensure Reliability



The Life Science business of Merck operates as MilliporeSigma in the U.S. and Canada.

**Supelco**<sup>®</sup>  
Analytical Products

# The Importance of SPE

Solid phase extraction is a form of digital (on/off) chromatography designed to extract, partition and/or adsorb one or more components from a liquid phase (sample) onto stationary phase (sorbent or resin). Over the last twenty five years, SPE has become the most powerful technique available for rapid and selective sample preparation (prep) prior to analytical chromatography.

SPE extends a chromatographic system's lifetime and improves qualitative and quantitative analysis. Also, by changing an analyte of interest's original matrix environment to a simpler matrix more suitable for subsequent analysis, the demand placed on an analytical instrument is considerably lessened.

**Figure 1. Urine sample without and with cleanup**



For more applications and application details, visit [SigmaAldrich.com/spe](https://www.sigmaaldrich.com/spe)

## Use SPE for Samples that:

- Require cleanup, trace enrichment/concentration or purification
- Contain particulate matter causing system clogging and high back-pressure
- Contain components that cause high background, misleading peaks and/or poor sensitivity
- Require sample matrix or solvent exchange

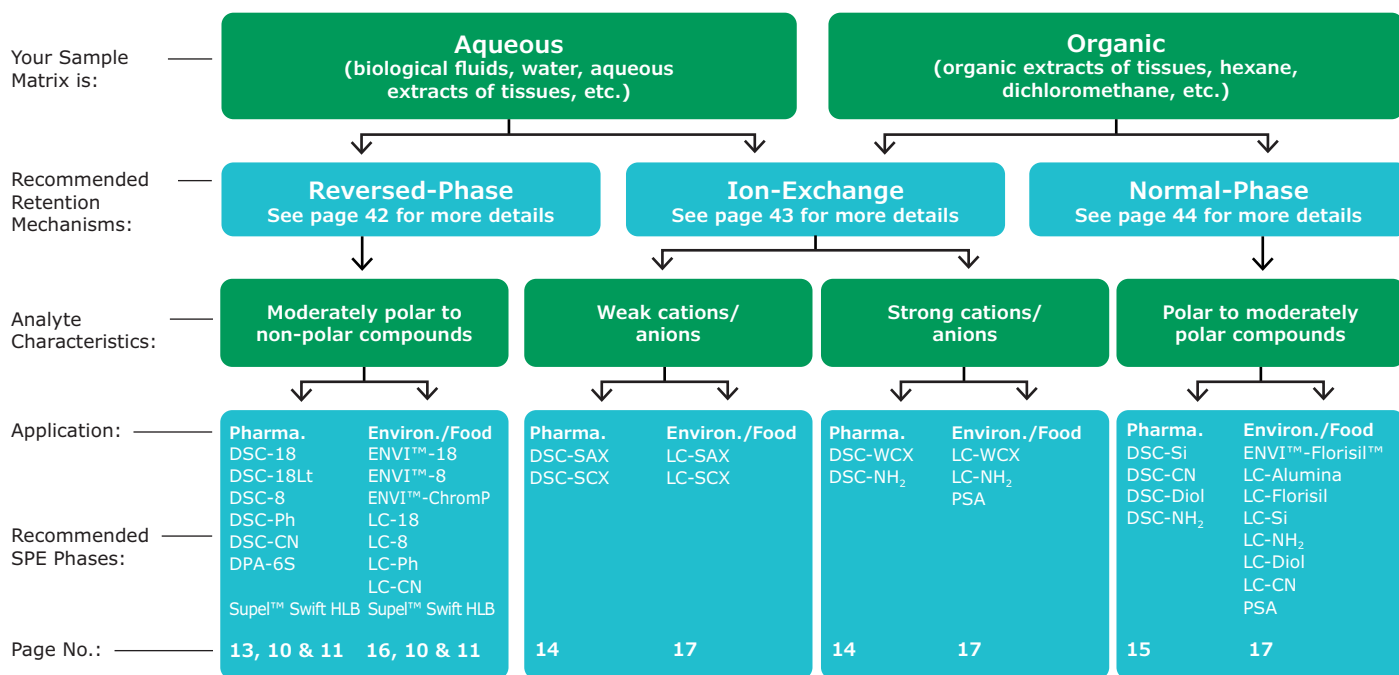
## Benefits of SPE:

- Switch sample matrices to a form more compatible with chromatographic analyses
- Concentrate analytes for increased sensitivity
- Remove interferences to simplify chromatography and improve quantitation
- Protect the analytical column from contaminants

## Common SPE Applications:

- Pharmaceutical compounds and metabolites in biological fluids
- Drugs of abuse in biological fluids
- Environmental pollutants in drinking and wastewater
- Pesticides, antibiotics or mycotoxins in food/agricultural matrices
- Desalting of proteins and peptides
- Fractionation of lipids
- Water and fat soluble vitamins

# SPE Phase Selection Quick Look-Up Guide



## Supelco® SPE Specialty Phases

Application	Field/ Application	Product	Page
Phospholipid removal/enrichment	Ph	HybridSPE®-Phospholipid	7-8
Phospholipid removal in a pipette tip format	Ph	HybridSPE® DPX Tips	9
Adsorption of polar compounds from aqueous or methanolic solution	G, E, Ph	Discovery® DPA-6S	13
Isolation of basic compounds from biological fluids	Ph, G	Discovery® DSC-MCAX	14
SPE filter discs (EPA 500 methods)	E	Supelclean™ ENVI™-18 and -8 DSK SPE Disks	16
SPE filter discs (EPA 500 methods)	E	Empore™ SPE Disks	23
Desalting proteins/peptides and other macromolecules	B	Supelclean™ LC-4 (wide pore)	16
Removal or isolation of polar compounds from organic matrices	E	Dual Layer Florisil®/Na <sub>2</sub> SO <sub>4</sub>	17
Solid-liquid extraction (SLE)	Ph, F, E, G	EXTrelut® NT	20-22
PFAS Testing	F, E	Supelclean™ ENVI-WAX™ and ENVI™-Chrom P, QuEChERS	24
Nitrosamines in water (EPA Method 521)	E	Supelclean™ Coconut Charcoal	26
Polar compounds in water	E	Supelclean™ ENVI-Carb™ Plus	26
PCBs from transformer/waste oils	E	Supelclean™ Sulfoxide	26
Pesticide residue analysis	F	Supelclean™ ENVI-Carb™	27
Pesticide residue analysis	F	Multi-layer Supelclean™ SPE Products	27
Pesticide residue analysis	F	Supel™ Sphere Carbon/NH <sub>2</sub>	29
Pesticide residue analysis from dry commodities (tea, spices, etc.)	F	Supelclean™ Ultra	28
Pesticide residue analysis - QuEChERS	F	Supel™ QuE Z-Sep, Z-Sep/C18, Z-Sep+, and Verde	30-33
Non-polar POP analysis in edible oils	F	Supelclean™ EZ-POP NP	34
FAMES (cis/trans) analysis	F	Discovery® Ag-Ion	35

Key: Ph = Pharmaceutical/Drugs; F = Food ; E = Environmental; B = Biological macromolecules; G = General

# SPE Bed Weight Quick Look-Up Guide

## Choosing the Right Bed Weight and Tube Size

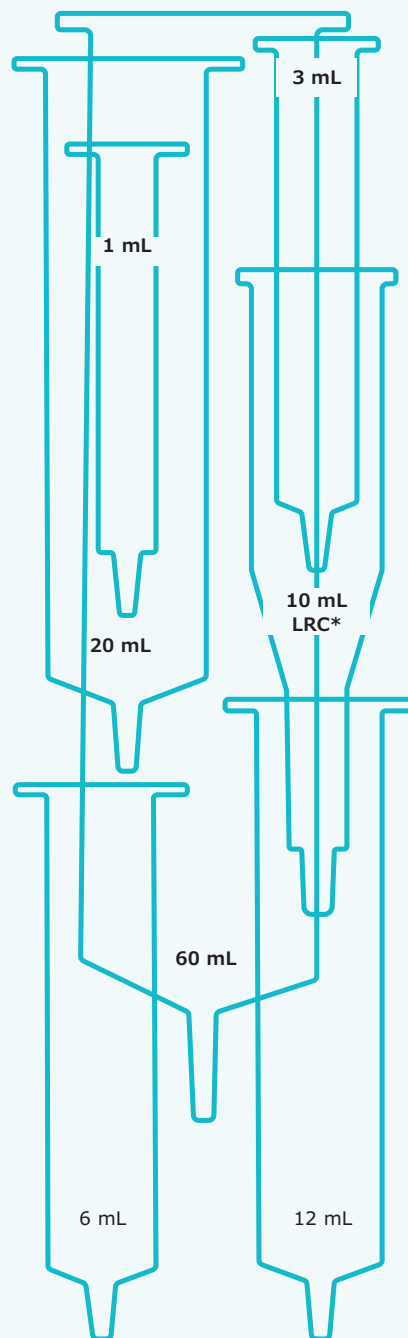
General guidelines for choosing the appropriate SPE tube size and bed weight configuration are listed in this table. Optimal method parameters and hardware/bed weight dimensions should be determined during method optimization and troubleshooting.

Bed Weight	Tube Volume	Minimum Elution Vol.	Bed Capacity*
50-100 mg	1 mL	100-200 $\mu$ L	2.5-10 mg
500 mg	3 mL	1-3 mL	25-100 mg
0.5-1 g	6 mL	2-6 mL	25-100 mg
2 g	12 mL	10-20 mL	0.1-0.2 g
5 g	20 mL	20-40 mL	1.25-2.5 g
10 g	60 mL	40-100 mL	0.5-1 g

\* This value depends on the analyte and sample matrix. As a rule of thumb, the bed capacity can be estimated with  $\sim$ 5% of the bed weight.

- Smaller tube dimensions (1 mL) contain smaller bed weights. Smaller bed weights allow for reduced elution volumes which can be beneficial for sensitive analyses, and when further processing is required (e.g., evaporation).
- 3 mL SPE tubes are the most common size dimension.
- 6 mL SPE tubes should be used when one or more steps in the SPE process require volumes greater than 3 mL. 6 mL tubes also contain larger bed weights (up to 1 g) which offers greater capacity, and can be beneficial when extracting difficult to retain compounds.
- 12, 20 and 60 mL tubes contain larger bed weights and head space volume which offer greater capacity. This allows researchers to use SPE as a purification or modified LPLC/Flash technique.
- The 10 mL LRC (large reservoir cartridges) are ideal for preparing larger sample volumes with smaller bed weights (25-100 mg). The packed section has the same diameter like a 1 mL tube.

Figure 2. Most common SPE hardware: Polypropylene SPE tubes with PE Frit



\* LRC: Large Reservoir Column

### Interested in trying a free sample?

Some of our SPE and QuEChERS products are offered in a no-cost sample pack; just fill out the form here:

[SigmaAldrich.com/SPEsample](https://www.sigmaaldrich.com/SPEsample)

## SPE Tubes and Specialty Hardware

### Additional Tubes and SPE Configurations

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Glass SPE Tubes with PTFE and SS Frits (pg. 35)



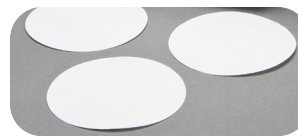
Common in environmental analysis to reduce leachables from PP hardware and PE frits

Reversible SPE Tubes (pg. 26 and 36)



Reverse SPE tubes prior to elution to minimize elution volume for strongly retained compounds

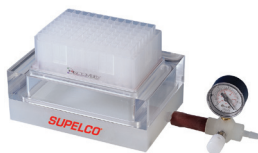
SPE Disks (pg. 16 and 23)



Allows for faster flow rates for processing large volume samples.

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Discovery® SPE 96-Well Plates (pg. 13-15)



For high throughput sample preparation

Supel™ QuE (Dispersive SPE) for QuEChERS (pg. 30-33)



Salt and sorbent vials for dispersive SPE

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## Custom Capabilities

Supelco® Analytical Products offer custom manufacturing services so you can optimize your sample processing procedure to the parameters dictated by your sample prep objectives. If there is a certain permutation of phase chemistry, bed weight and hardware configuration you require that is not listed within our standard product line, visit

[SigmaAldrich.com/customspe](https://www.sigmaaldrich.com/customspe)

# SPE Accessories

## SPE Manifolds

**Visiprep™ DL and Standard Vacuum Manifold (pg. 37)**



DL uses disposable liners that prevent cross-contamination

**PlatePrep Vacuum Manifold (pg. 40)**



For 96-well SPE  
Useful for stacking SPE tubes

**Visiprep™ 5-Port Flask Manifold (pg. 37)**



Collects the SPE eluate in round flasks for easy rotary evaporation

**ENVI-Disk™ Holder (pg. 41)**



Used with 47 mm SPE disks

**Preppy™ Vacuum Manifold (pg. 38)**



Most economical

**Visi™-1 Single SPE Tube Processor (pg. 37)**



For processing very few  
SPE samples

## SPE Manifold Accessories

**Visiprep™ Large Volume Sampler (pg. 38)**



For processing larger  
sample volumes

**Visidry™ Drying Attachment (pg. 38)**



For drying SPE tubes or  
evaporating SPE eluate

**SPE Tube Adapters and Large Volume Reservoirs (pg. 35)**



Useful for stacking SPE tubes or  
processing SPE tubes via luer  
syringe; increasing tube volume

**Trap Kit and Vacuum Gauge Bleed Valve (pg. 39)**



Additional vacuum accessories

# HybridSPE® Technology

## Simultaneous protein and phospholipid removal

HybridSPE® technology combines the simplicity of protein precipitation with the selectivity of solid phase extraction (SPE) for the targeted removal of phospholipids in biological plasma/serum (**Figure 3**). The technology utilizes a zirconia-coated particle, and exhibits selective affinity towards phospholipids while remaining non-selective towards a range of basic, acidic and neutral compounds. The phospholipid retention mechanism is based on a selective Lewis acid-base interaction between the proprietary zirconia ions (functionally bonded to the HybridSPE® stationary phase) and the phosphate moiety present in all phospholipids (**Figure 4**).



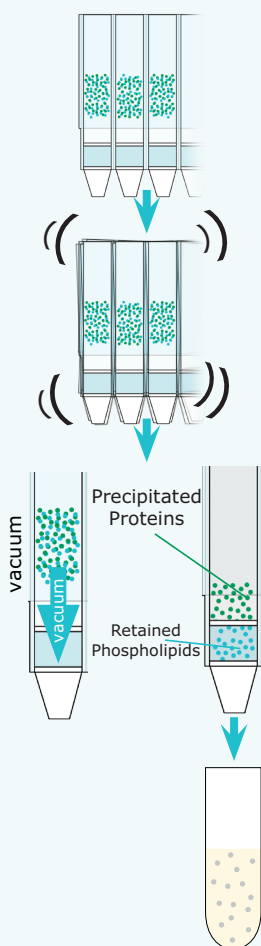
**Figure 3. HybridSPE® “In-well” Method**

1. Precipitate Proteins by adding 100  $\mu$ L plasma or serum to the HybridSPE® plate followed by 300  $\mu$ L 1% formic acid in acetonitrile. Add I.S. as necessary.

2. Mix by vortexing/shaking the HybridSPE® plate or by aspirating/dispensing with 0.5-1 mL pipette tip (e.g., TOMTEC Quadra liquid handler).

3. Apply vacuum. The packed-bed filter/frit assembly acts as a depth filter for the concurrent physical removal of precipitated proteins and chemical removal of phospholipids. Small molecules (e.g., pharma compounds and metabolites) pass through unretained.

4. Resulting filtrate/eluante is free of proteins and phospholipids and ready for immediate LC-MS/MS analysis; or it can be evaporated and reconstituted as necessary prior to analysis.



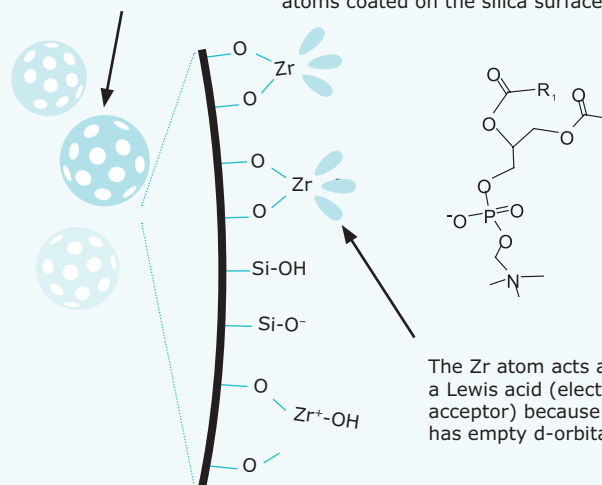
## Features and Benefits

- Merges both protein precipitation and SPE
- Offers the simplicity of protein precipitation
- Selectively removes phospholipids via Lewis acid-base interactions
- 2-3 step generic procedure
- Typically >98% removal of phospholipids and precipitated proteins
- Minimal to no method development required
  - 96-well or individual cartridge format
  - Dispersive 96-well tip format (DPX) for high throughput automation

**Figure 4. Lewis Acid-Base Interactions Between HybridSPE® Zirconia atoms and Phospholipids**

Proprietary HybridSPE® Zirconia Coated Silica

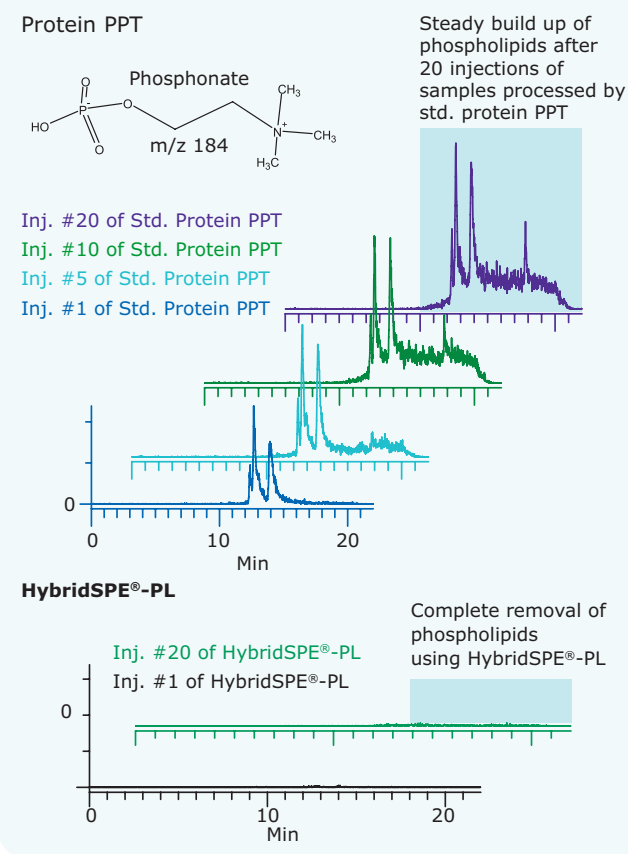
The phosphate moiety of phospholipids is a strong Lewis base (electron donor) that interacts with Zr atoms coated on the silica surface.



## LC Accumulation of Phospholipids

With advances in LC-MS technology, many analysts are decreasing LC run time by incorporating ballistic gradients and sub-2  $\mu\text{m}$  HPLC column particles. When coupled with standard protein precipitation (e.g., plasma:acetonitrile, 1:3 v/v), ballistic gradients are often inadequate at purging the column of phospholipids. As a result, phospholipids can build on the column (**Figure 5**), potentially change LC retention & selectivity, and elute uncontrollably downstream in an injection run sequence causing unpredictable ion-suppression effects and poor reproducibility. **Figure 5** compares a series of reversed-phase gradient LC-MS injections after standard protein PPT with HybridSPE® in which m/z 184 (phosphonate moiety of phospholipids) is monitored. Unlike traditional protein PPT techniques that use centrifugation or simple filtration to remove precipitated proteins, HybridSPE® 96-well plates contain a series of filters that allow users to concurrently remove proteins and phospholipids reducing LC column back pressure buildup commonly observed with standard PPT only, in particular for sub-2  $\mu\text{m}$  HPLC columns that are more prone to clogging than larger particle size columns (2.7 - 5.0  $\mu\text{m}$ ) (**Figure 5**).

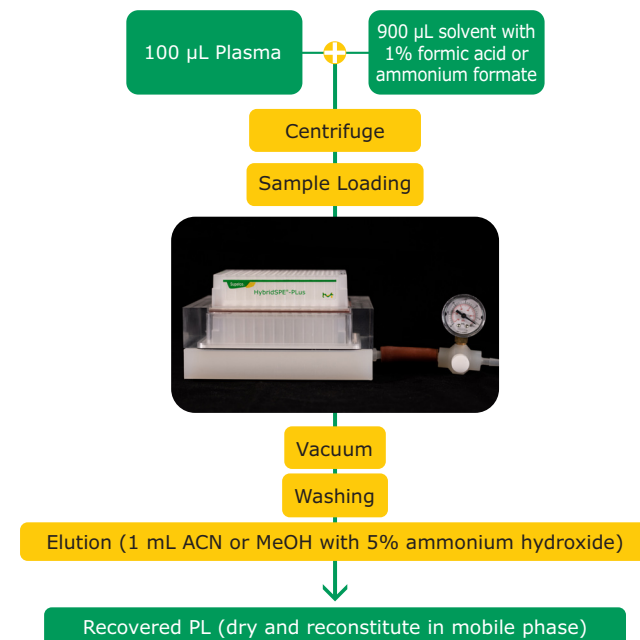
**Figure 5.** Gradient RP LC-MS of Blank Plasma Samples Prepared by Standard Protein PPT vs. HybridSPE®



## Phospholipid Enrichment Using HybridSPE® Technology

Although HybridSPE® is typically used to remove phospholipid interferences in biological samples, the same Lewis acid-base interactions that selectively remove phospholipids can also be used to recover phospholipids for analysis and phospholipid profiling. Phospholipids retained on the sorbent can be easily eluted with a strong basic solution, such as ammonium hydroxide. The bind and elute process of phospholipid enrichment is demonstrated in the flow chart below.

**Figure 6.** Experimental flow chart of the recovery of phospholipids from rabbit plasma



Description	Qty.	Cat. No.
<b>Well Plates</b>		
HybridSPE®-PLus 96-well Plate, 50 mg/well	1	575659-U
	20	575673-U
HybridSPE®-PL, Small Vol. 96-well Plate, 15 mg/well	1	52794-U
	20	52798-U
<b>SPE Cartridges</b>		
HybridSPE®-PL Ultra Cartridge, 30 mg/1 mL	100	55269-U
HybridSPE®-PL Cartridge, 30 mg/1 mL	100	55261-U
	200	55276-U
HybridSPE®-PL Cartridge, 500 mg/6 mL	30	55267-U
<b>Plate Accessories</b>		
96 Round/Deep Well Collection Plate, PP for HybridSPE®-PLus	60	Z717266
96 Well-Plate Pre-cut Sealing Films	100	Z721581
PlatePrep Vacuum Manifold	1	57192-U
96-well Protein Precipitation Filter Plate (for offline protein precipitation)	1	55263-U

For more information, visit [SigmaAldrich.com/hybridspe](https://SigmaAldrich.com/hybridspe)



# Automated SPE with HybridSPE® DPX Tips

## Extraction in Seconds

DPX stands for Dispersive Pipette Extraction. HybridSPE® DPX Tips are pipette tips that incorporate loosely contained HybridSPE® sorbent material that is mixed with the sample solution when aspirated to accomplish solid phase extraction. HybridSPE® technology is a simple and generic sample prep platform designed for the gross level removal of endogenous phospholipid interferences from biological plasma and serum prior to LC-MS or LC-MS/MS analysis (see page 8).

In this simple technique, biological plasma or serum is first subjected to protein precipitation via the addition and mixing of acidified acetonitrile. Precipitated proteins are then removed by centrifugation and the resulting supernatant is extracted using the HybridSPE® DPX tip which acts as a chemical filter that specifically targets the removal of endogenous sample phospholipids.

The phospholipid retention mechanism is based on a highly selective Lewis acid-base interaction between the proprietary zirconia ions functionally bonded to the HybridSPE® stationary phase and the phosphate moiety consistent with all phospholipids. The resulting eluent is ready for immediate LC-MS or LC-MS/MS analysis.

### What size tips do I need?

HybridSPE®-PL Sample and PPT Agent Guidelines		
	30 mg tips	50 mg tips
Plasma/serum	30-100 µL	100-300 µL
Precipitating agent	90-300 µL	300-900 µL

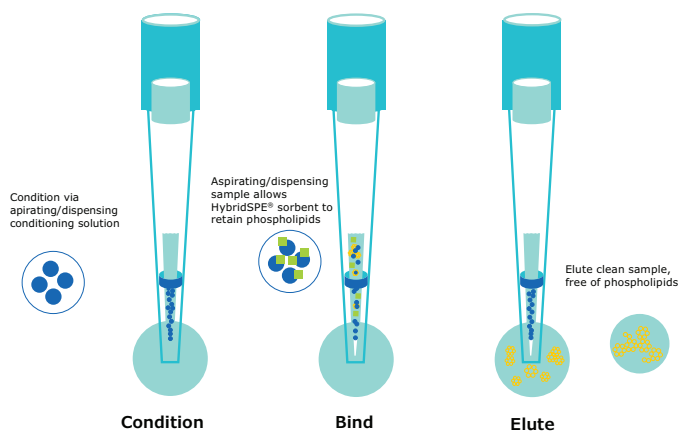


Figure 7. Typical workflow for HybridSPE® DPX tips



### The unique mixing technique employed provides numerous advantages:

- Minimal elution solvent volumes
- Rapid extraction times (less than 3 min. per sample/wellplate)
- High extraction efficiencies
- Easy to perform extractions
- Lower costs
- Higher throughput
- Minimal training required
- Environmentally friendly

Description	Qty.	Cat. No.
HybridSPE® DPX tip, 30 mg, Tecan® 200 µL	96	52973-U
HybridSPE® DPX tip, 50 mg, Tecan® 1 mL	96	52974-U
HybridSPE® DPX tip, 30 mg, Hamilton® 300 µL	96	52977-U
HybridSPE® DPX tip, 50 mg, Hamilton® 1 mL	96	52978-U
HybridSPE® DPX tip, 30 mg, Integra 300 µL	96	52979-U
HybridSPE® DPX tip, 50 mg, Integra 1250 µL	96	52980-U
HybridSPE® DPX tip, 30 mg, Universal 1 mL	96	52981-U
HybridSPE® DPX tip, 50 mg, Universal 1 mL	96	52982-U

We also offer the DPX dispersive tip format in our Supel™ Swift HLB phase chemistry.

# Supel™ Swift HLB SPE

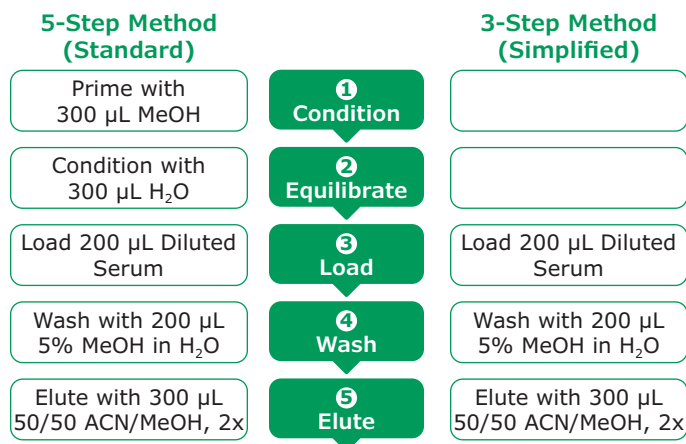
Supel™ Swift HLB SPE is a polymeric stationary phase for solid phase extraction prior to instrumental analysis. It has both hydrophilic and lipophilic functional groups for the extraction of a broad range of compounds from aqueous samples. It retains analytes having different polarities and Log P values due to its hydrophilic and lipophilic balance (HLB) property. Benefits of Supel™ Swift HLB SPE cartridges include:

- Suitable to the generic methodology
- Wide applicability
- Ideal for LC-MS and other workflows



## The possibility of 3-step SPE

Supel™ Swift HLB SPE cartridges can reduce the number of steps in the solid phase extraction of your analyte from 5 to 3. You can directly load your sample onto the Supel™ Swift HLB SPE cartridge bed and potentially eliminate the need for cumbersome pre-conditioning steps. This feature of the Supel™ Swift HLB SPE cartridges reduces the number of errors in sample processing and simplifies sample preparation.



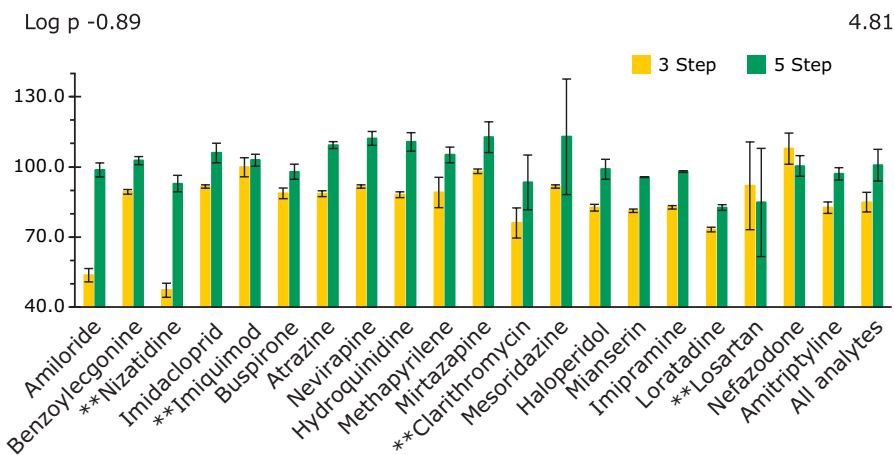
**Figure 8.** General processing of samples (serum 1:1 diluted) with Supel™ Swift HLB cartridges (30 mg/1 mL) using a 5-step method and a 3-step method

## Excellent recovery for a wide range of compounds having different polarities and Log P values

Supel™ Swift HLB SPE cartridges offers good recovery for a wide range of compounds and polarities.

**Figure 9** presents absolute recoveries of compounds ranging in log P from -0.9 to 4.8 using Supel™ Swift HLB SPE cartridges with both the 3-Step and 5-Step methods from plasma.

All-in-all, the 5-Step method shows better recoveries as compared to the 3-Step process. In the 5-Step process, all twenty analytes had recoveries between 80% and 120%. However, eighty percent of the analytes still showed recoveries in the 80% to 120% range by the 3-Step process.



**Figure 9:** Summary of Recovery for the 3- and 5-Step Process using Supel™ Swift HLB SPE cartridges. Analytes are ordered by increasing log P values.

Description	Cat. No.
Supel™ Swift HLB SPE Tubes weight 200 mg (bed), volume 6 mL, pk of 30 ea	57491-U
Supel™ Swift HLB SPE Tubes weight 60 mg (bed), volume 3 mL, pk of 54 ea	57492-U
Supel™ Swift HLB SPE Tubes weight 30 mg (bed), volume 1 mL, pk of 108 ea	57493-U
Supel™ Swift HLB 96-well SPE 10 mg / well, Pk. 1	57495-U
Supel™ Swift HLB 96-well SPE 30 mg / well, Pk. 1	57494-U

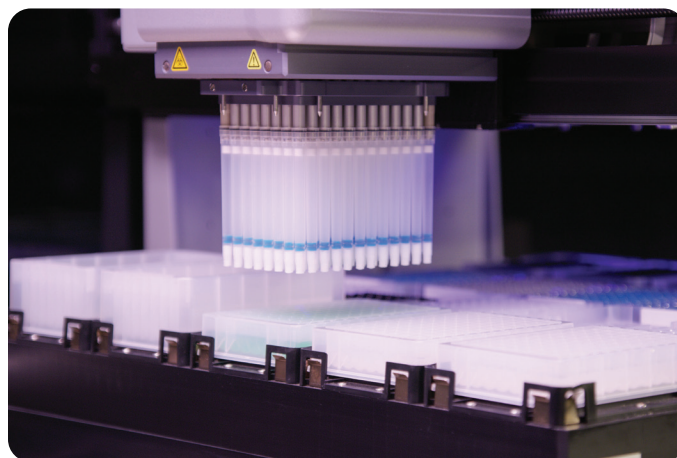
For more information visit: [SigmaAldrich.com/SupelSwiftHLB](https://SigmaAldrich.com/SupelSwiftHLB)

# Supel™ Swift HLB DPX Tips

## Automated SPE for Extraction in Seconds

DPX stands for Dispersive Pipette Extraction, a patented technology that introduces the benefits of solid phase extraction into a revolutionary, easy-to-use pipette tip. This device is unique from all other SPE devices because adsorbent is loosely contained within the tip. The Supel™ Swift Hydrophilic-Lipophilic Balanced (HLB) adsorbent within these tips was specifically developed to provide retention and cleanup of both polar and non-polar compounds from aqueous samples.

The Supel™ Swift HLB DPX Tips solution offers an automation amenable, rapid technique that can offer many advantages, including reduction of solvent and sample volumes, increased throughput, and reduction of labor and workflow costs.



### Typical workflow for Supel™ Swift HLB DPX Tips

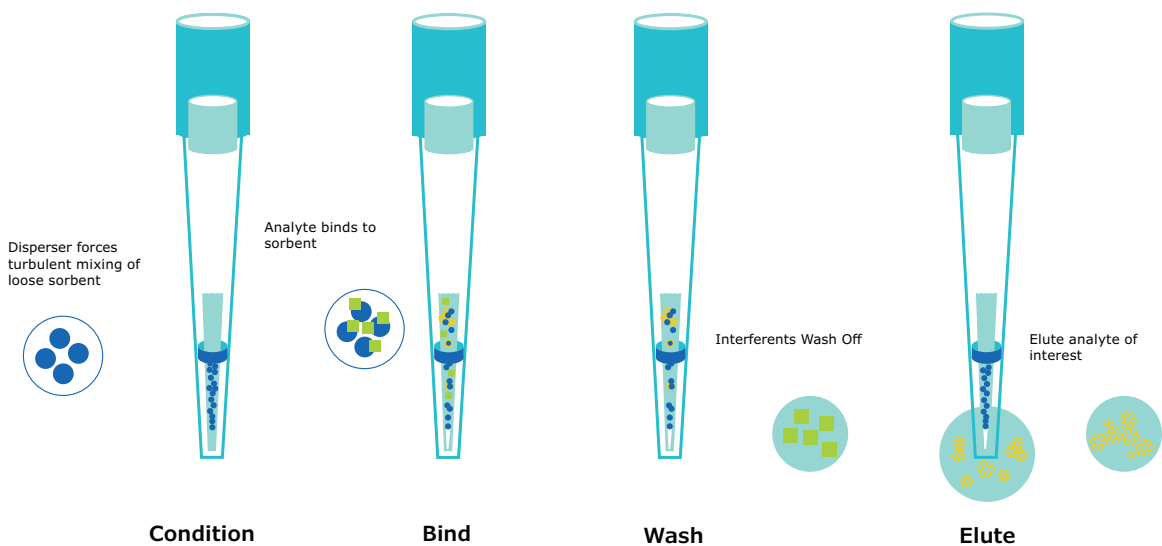


Figure 10: Typical workflow for Supel™ Swift HLB DPX Tips

In one application, the Supel™ Swift HLB DPX Tips were used to extract 13 opioid drugs from urine using a Hamilton® STARlet automation platform for cleanup followed by LC-MS/MS analysis. The automated extraction method can process multiple samples simultaneously in under 10 minutes thereby minimizing within-run sample variability and maximizing throughput.

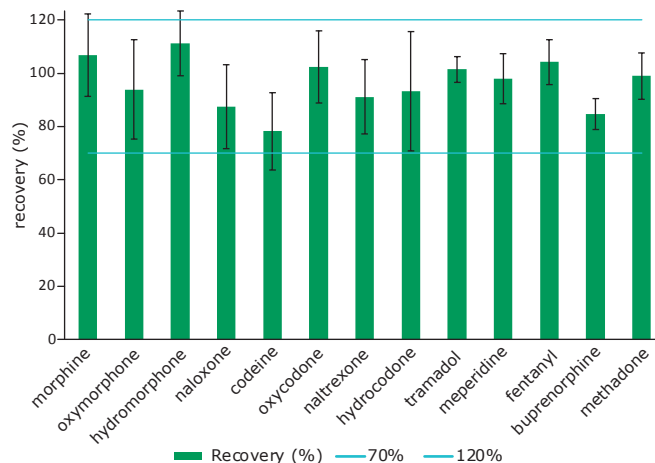


Figure 11. Recoveries for 13 opioid drugs extracted using Supel™ Swift HLB DPX Tips.

Good recovery values were achieved for all compounds between 84-111%. Relative Standard Deviations (%RSDs) were calculated using 8 replicate extractions and were under 11.2% for all compounds.

This DPX HLB method can process multiple samples in under 10 minutes allowing for a fast, automated, and high throughput workflow. The method is robust, linear, and provides the necessary sensitivity to meet most laboratories' needs.

Description	Cat. No
Supel™ Swift HLB DPX 5 mg Hamilton® 1 mL	52984-U
Supel™ Swift HLB DPX 5 mg Universal 1 mL	52989-U
Supel™ Swift HLB DPX 10 mg Hamilton® 1 mL	52992-U
Supel™ Swift HLB DPX 10 mg Universal 1 mL	52995-U
Supel™ Swift HLB DPX 20 mg Hamilton® 1 mL	52997-U
Supel™ Swift HLB DPX 20 mg Universal 1 mL	52999-U
Supel™ Swift HLB DPX® 3 mg Hamilton® Microelution 300 µL	53001-U

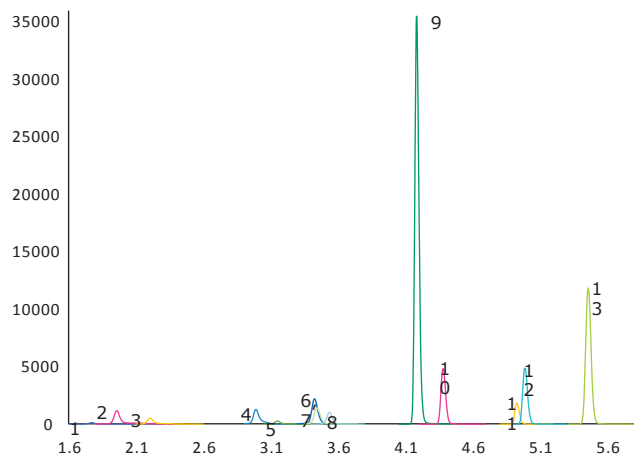


Figure 12. Chromatographic separation for 13 opioid drugs

<b>Column:</b>	Ascentis® Express Phenyl-Hexyl column 10 cm x 2.1 mm, 2.7 µm (53336-U)
<b>Mobile phase A:</b>	water with 0.1% formic acid
<b>Mobile phase B:</b>	methanol with 0.1% formic acid
<b>Column Temp:</b>	30 °C
<b>Inj. Vol:</b>	5 µL
<b>Flow Rate:</b>	0.4 mL/min
<b>Gradient:</b>	5 to 20% B in 2.25 mins; to 60% B in 2.25 mins; held for 1.5 mins; to 95% B in 0.1 mins; held for 1.4 mins; reset to 5% for 3.4 mins to re-equilibrate.

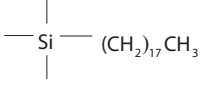
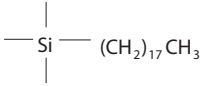
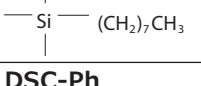
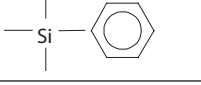
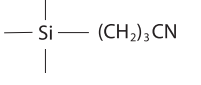
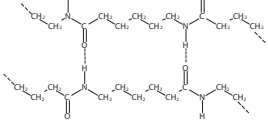
# Discovery® SPE

## Reversed-Phase

Discovery® reversed-phase SPE products are specifically developed, tested and quality controlled for pharmaceutical and clinical applications. Experience greater and more reproducible recoveries for the quick and effective extraction, isolation and concentration

of pharmaceuticals from biological fluids and other aqueous sample matrices.

For Discovery® silica specifications, see page 13. For general guidelines on reversed-phase SPE, see page 49.

<b>DSC-18</b> 	<ul style="list-style-type: none"> <li>• Polymerically bonded, octadecyl (18% C), endcapped</li> <li>• Higher 18% C loading for increased binding capacities and higher recoveries</li> <li>• The least selective phase: retains most organic analytes from aqueous matrices</li> <li>• Beneficial for extracting numerous analytes diverse in structure from the same sample</li> </ul>
<b>DSC-18Lt</b> 	<ul style="list-style-type: none"> <li>• Monomerically bonded, octadecyl (11% C), endcapped</li> <li>• Increased retention for moderately polar hydrophobic molecules</li> <li>• Used to elute very large hydrophobic molecules that are too strongly retained on DSC-18. Use this less retentive phase for the rapid release of hydrophobic compounds using weaker organic solvents at lower volumes</li> </ul>
<b>DSC-8</b> 	<ul style="list-style-type: none"> <li>• Monomerically bonded, octyl (9% C), endcapped; lower carbon content than DSC-18Lt</li> <li>• Used to elute very large hydrophobic molecules too strongly retained on DSC-18 or DSC-18Lt</li> <li>• Use this less retentive phase for the rapid release of hydrophobic molecules using weaker organic solvents at lower volumes</li> </ul>
<b>DSC-Ph</b> 	<ul style="list-style-type: none"> <li>• Monomerically bonded, phenyl (7% C), endcapped</li> <li>• Similar in polarity to DSC-8; however, electron dense aromatic ring offers some unique selectivity and retention</li> </ul>
<b>DSC-CN</b> 	<ul style="list-style-type: none"> <li>• Monomerically bonded, cyanopropyl (7% C), endcapped</li> <li>• Can behave as either reversed-phase or normal-phase</li> <li>• Ideal for very hydrophobic analytes that may be irreversibly retained on more hydrophobic sorbents such as DSC-18</li> <li>• Less retentive than DSC-Si or DSC-Diol when used as normal phase (organic matrices such as hexane or oils)</li> <li>• Allows for the rapid release of very polar molecules irreversibly retained on very polar sorbents</li> </ul>
<b>DPA-6S</b> 	<ul style="list-style-type: none"> <li>• Polyamide Resin: Particle Size: 50-160 µm, Surf pH: 4.5-7.5, Density: 0.2-0.3 cm<sup>3</sup>/g, Water Content: &lt;5%</li> <li>• Used to adsorb polar compounds (-OH groups, esp. phenolic compounds) from aqueous or methanolic solutions under the reversed-phase mechanism through strong hydrogen bonding between compound hydroxyl groups and amide groups of the resin</li> <li>• Useful for extracting tannins, chlorophyll, humic acid, pharmacologically active terpenoids, flavonoids, gallic acid, catechol A, protocatechuic acid and phloroglucinol</li> <li>• Also useful for extracting aromatic carboxylic acids, nitroaromatic compounds and irreversibly retains quinones</li> </ul>

### Discovery® Reversed-Phase SPE Products

Description	Qty.	DSC-18	DSC-18Lt	DSC-8	DSC-Ph	DSC-CN	DPA-6S
<b>Discovery® SPE Tubes</b>							
50 mg/1 mL	108	52601-U	Custom	52703-U	Custom	Custom	52624-U
100 mg/1 mL	108	52602-U	Custom	Custom	Custom	Custom	Custom
500 mg/3 mL	54	52603-U	52613-U	52713-U	52727-U	52695-U	<sup>1</sup> 52625-U
500 mg/6 mL	30	52604-U	52615-U	52714-U	52728-U	52696-U	<sup>2</sup> 52626-U
1 g/6 mL	30	52606-U	52616-U	52716-U	Custom	52697-U	<sup>3</sup> 52627-U
2 g/12 mL	20	52607-U	Custom	Custom	Custom	Custom	Custom
5 g/20 mL	20	52608-U	Custom	Custom	Custom	Custom	<sup>4</sup> 52631-U
10 g/60 mL	16	52609-U	Custom	Custom	Custom	Custom	Custom
<b>Discovery® SPE 96-Well Plates</b>							
100 mg/well	1	575603-U	Custom	Custom	Custom	Custom	Custom
50 mg/well	1	Custom	Custom	Custom	Custom	Custom	Custom
25 mg/well	1	575601-U	Custom	Custom	Custom	Custom	Custom
<b>Bulk Packing</b>							
	100 g	52600-U					

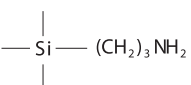
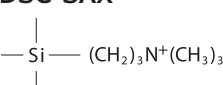

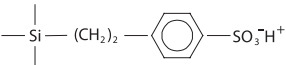
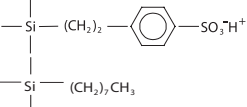
<sup>1</sup> 250 mg/3 mL, <sup>2</sup> 250 mg/6 mL, <sup>3</sup> 500 mg/6 mL, <sup>4</sup> 2 g/20 mL

## Ion-Exchange and Mixed-Mode

Discovery® ion-exchange SPE products are specifically developed, tested and quality controlled for pharmaceutical and clinical applications. The Discovery® ion-exchange product line offers excellent selectivity towards charged molecular species enabling the user to extract, isolate, purify and concentrate charged ionizable pharmaceuticals (basic or acidic) from both polar and non-polar sample matrices.

Use mixed-mode SPE (e.g., Discovery® DSC-MCAX) for superior cleanup and selectivity when extracting basic pharmaceutical compounds from biological matrices such as plasma and urine.

For Discovery® silica specifications, see page 13. For general guidelines on ion-exchange and mixed-mode SPE, see page 50.

<p><b>DSC-NH<sub>2</sub></b></p> 	<ul style="list-style-type: none"> <li>• Polymerically bonded aminopropyl phase that is very polar in nature (hydrogen bonding) allowing for both normal-phase and ion-exchange applications</li> <li>• A weak anion exchanger with a pK<sub>a</sub> of 9.8. At pH 7.8 or below, the functional groups are positively charged</li> <li>• Allows the rapid release of very strong anions such as sulfonic acids that may be retained irreversibly on SAX</li> <li>• Can be used in some reversed-phase applications (due to ethyl spacer); however, it is predominately used as an ion-exchanger or normal-phase sorbent due to its polar nature</li> </ul>
<p><b>DSC-SAX</b></p> 	<ul style="list-style-type: none"> <li>• A polymerically bonded quarternary amine that remains positively charged at all pH levels</li> <li>• Strong anion ion exchanger, commonly used when extracting weaker cations (e.g., carboxylic acids) that may not bind strongly enough to weaker anion exchangers</li> <li>• Selectivity can be modified by changing the counter ion with the appropriate buffer during conditioning</li> <li>• Counter ion is Cl<sup>-</sup></li> </ul>
<p><b>DSC-WCX</b></p> 	<ul style="list-style-type: none"> <li>• A polymerically bonded carboxy propyl phase with a K<sup>+</sup> counter ion and a pK<sub>a</sub> of 4.8</li> <li>• Its weak cation exchange properties carries a negative charge at pH 6.8 or above</li> <li>• A pH of 2.8 or below neutralizes this phase for easier elution of strong cationic analytes that are neutralized only at extreme basic conditions</li> <li>• Typically used when dealing with very strong cationic (high pK<sub>a</sub>) compounds that may be irreversibly retained on strong cation exchangers</li> </ul>
<p><b>DSC-SCX</b></p> 	<ul style="list-style-type: none"> <li>• A polymerically bonded, benzene sulfonic acid functional group with a H<sup>+</sup> counter ion that is a strong cation exchanger due to its very low pK<sub>a</sub> (&lt;1.0)</li> <li>• Silica support allows for use with all common organic solvents (no shrinking/swelling)</li> <li>• Excellent capacity (0.8 meq/g) for cleaning up solution phase combinatorial chemistry reactions (removing target molecules from reaction by-products and excess reagents)</li> <li>• The presence of the benzene ring offers some mixed-mode capabilities (hydrophobic interactions) that should be considered when extracting cations from aqueous matrices</li> </ul>
<p><b>DSC-MCAX</b></p> 	<ul style="list-style-type: none"> <li>• Packed bed contains both octyl (C8) and benzene sulfonic acid (SCX) bondings. (H<sup>+</sup> as counterion)</li> <li>• Developed for superior selectivity/sample cleanup when isolating basic compounds from biological fluids</li> <li>• Dual retention mechanisms broadens retention for a range of neutral, basic, acidic and zwitterionic compounds</li> <li>• Greater ion-exchange capacity for isolating polar basic and zwitterionic compounds</li> <li>• Can be used to fractionate basic/zwitterionic compounds from acidic and neutral compounds</li> </ul>

### Discovery® Ion-Exchange SPE Products

Description	Qty.	DSC-NH <sub>2</sub>	DSC-SAX	DSC-WCX	DSC-SCX	DSC-MCAX
<b>Discovery® SPE Tubes</b>						
50 mg/1 mL	108	Custom	52661-U	52737-U	Custom	52781-U
100 mg/1 mL	108	52636-U	Custom	52739-U	52685-U	Custom
500 mg/3 mL	54	52637-U	52664-U	Custom	52686-U	52783-U <sup>1</sup>
500 mg/6 mL	30	52638-U	52665-U	Custom	52688-U	52784-U <sup>2</sup>
1 g/6 mL	30	52640-U	52666-U	Custom	52689-U	52788-U, 52786-U <sup>3</sup>
2 g/12 mL	20	52641-U	Custom	Custom	52690-U	—
5 g/20 mL	20	Custom	Custom	Custom	52691-U	—
10 g/60 mL	16	Custom	Custom	Custom	52692-U	—
<b>Discovery® SPE 96-Well Plates</b>						
100 mg/well	1	575615-U	Custom	Custom	Custom	Custom
50 mg/well	1	Custom	Custom	Custom	Custom	Custom
25 mg/well	1	Custom	Custom	Custom	Custom	Custom
<b>Bulk Packing</b>						
	100 g	57212-U	Custom	57228-U	57221-U	—

<sup>1</sup> 3 mL/100 mg, pk 54, <sup>2</sup> 300 mg/3 mL, pk 54, <sup>3</sup> 300 mg/6 mL, pk 30

## Normal-Phase

Discovery® normal-phase SPE products are specifically developed, tested and quality controlled for normal phase pharmaceutical applications and other modified flash techniques. The Discovery® normal phase product line enables you to quickly and effectively extract, isolate, purify and concentrate polar compounds from non-polar solutions. Its highly selective properties allow

the user to separate or remove structurally similar molecules through successive wash/elutions with increasingly polar solutions.

For Discovery® silica specifications, see page 2.  
For general guidelines on normal-phase SPE, see page 51.

<b>DSC-Si</b> $\begin{array}{c}   \\ \text{—Si—OH} \\   \end{array}$	<ul style="list-style-type: none"> <li>• Unbonded acid washed silica sorbent ideal for normal-phase SPE and other modified flash techniques</li> <li>• Considered the most polar normal-phase sorbent available</li> <li>• Excellent capacity for purifying solution phase CombiChem reactions when removing target molecules from reaction by-products and excess reagents</li> </ul>
<b>DSC-Diol</b> $\begin{array}{c}   \\ \text{—Si—}(\text{CH}_2)_3\text{CH}_2\text{CH}(\text{OH})\text{CH}_2 \\   \end{array}$	<ul style="list-style-type: none"> <li>• Polymerically bonded, 2,3-Dihydroxypropoxypropyl (7% C)</li> <li>• Polar sorbent most commonly used for normal-phase applications (polar extractions from non-polar matrices)</li> <li>• The sorbent's dihydroxy groups facilitate strong hydrogen bonding</li> <li>• Excellent selectivity when extracting structurally similar molecules</li> </ul>
<b>DSC-CN</b> $\begin{array}{c}   \\ \text{—Si—}(\text{CH}_2)_3\text{CN} \\   \end{array}$	<ul style="list-style-type: none"> <li>• Monomerically bonded, cyanopropyl (7% C), endcapped</li> <li>• Can behave as either reversed-phase or normal-phase</li> <li>• Ideal for very hydrophobic analytes that may be irreversibly retained on more hydrophobic sorbents such as DSC-18</li> <li>• Less retentive than DSC-Si or DSC-Diol when used as normal-phase (organic matrices such as hexane or oils)</li> <li>• Allows for the rapid release of very polar molecules irreversibly retained on very polar sorbents</li> </ul>
<b>DSC-NH<sub>2</sub></b> $\begin{array}{c}   \\ \text{—Si—}(\text{CH}_2)_3\text{NH}_2 \\   \end{array}$	<ul style="list-style-type: none"> <li>• Polymerically bonded, aminopropyl phase that is very polar in nature (hydrogen bonding) allowing for both normal-phase and ion-exchange applications</li> <li>• A weak anion exchanger with a pKa of 9.8. At pH 7.8 or below, the functional groups are positively charged</li> <li>• Allows the rapid release of very strong anions such as sulfonic acids that may be retained irreversibly on SAX (a quarternary amine sorbent that is always positively charged)</li> <li>• Can be used in some reversed-phase applications (due to ethyl spacer); however, it is predominately used as an ion-exchanger or normal-phase sorbent due to its polar nature</li> </ul>

### Discovery® Normal-Phase SPE Products

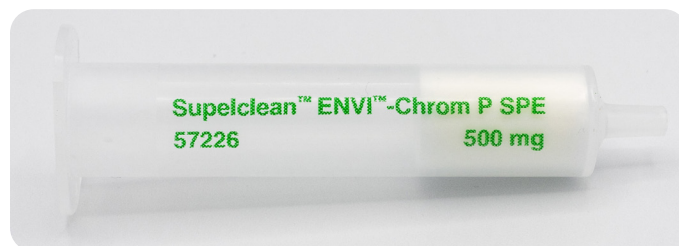
Description	Qty.	DSC-CN	DSC-Si	DSC-Diol	DSC-NH <sub>2</sub>
<b>Discovery® SPE Tubes</b>					
50 mg/1 mL	108	52693-U	52652-U	Custom	52635-U
100 mg/1 mL	108	52694-U	52653-U	Custom	52636-U
500 mg/3 mL	54	52695-U	52654-U	52751-U	52637-U
500 mg/6 mL	30	52696-U	52655-U	52752-U	52638-U
1 g/6 mL	30	52697-U	52656-U	52753-U	52640-U
2 g/12 mL	20	Custom	52657-U	Custom	52641-U
5 g/20 mL	20	Custom	52658-U	Custom	Custom
10 g/60 mL	16	Custom	52659-U	Custom	Custom
<b>Discovery® SPE 96-Well Plates</b>					
100 mg/well	1	Custom	Custom	Custom	575615-U
50 mg/well	1	Custom	575608-U	Custom	Custom
25 mg/well	1	Custom	Custom	Custom	Custom
<b>Bulk Packing</b>					
	100 g	Custom	Custom	Custom	57212-U

# Supelclean™ and Supelclean™ ENVI™ SPE

## Reversed-Phase

The Supelclean™ SPE line represents one of our original brands. It is referenced in hundreds of journal publications and validated in methods such as EPA 500 series (drinking water) and SW-846 methods (solid waste).

For Supelclean™ silica specifications, see the table below. For general guidelines on reversed-phase SPE, see page 49.



<b>LC-18</b>	<ul style="list-style-type: none"> <li>• Monomerically bonded, octadecyl (10% C), endcapped</li> <li>• For reversed-phase extraction of nonpolar to moderately polar compounds.</li> <li>• pH range 2-8</li> </ul>
<b>LC-8</b>	<ul style="list-style-type: none"> <li>• Monomerically bonded, octyl (7% C), endcapped</li> </ul>
<b>LC-4 (Wide Pore)</b>	<ul style="list-style-type: none"> <li>• Butyldimethyl, wide pore (500 Å), endcapped</li> <li>• Larger pore size to accommodate larger macromolecules (e.g., proteins and peptides)</li> <li>• Commonly used for desalting proteins and peptides in aqueous samples</li> </ul>
<b>LC-Ph</b>	<ul style="list-style-type: none"> <li>• Monomerically bonded, phenyl (5.5% C), endcapped</li> </ul>
<b>LC-CN</b>	<ul style="list-style-type: none"> <li>• Monomerically bonded, cyanopropyl (7% C), endcapped</li> </ul>
<b>ENVI™-18</b>	<ul style="list-style-type: none"> <li>• Polymerically bonded, octadecyl (17% C), endcapped</li> <li>• Excellent for cleaning, extracting and concentrating pollutants from aqueous environmental samples</li> <li>• Higher 17% C loading for increased binding capacities and higher recoveries</li> <li>• Higher carbon loading also offers greater resistance to extreme pH conditions</li> <li>• Typical applications include herbicides, fungicides, pesticides and other aqueous hazardous waste materials</li> <li>• Ideal for EPA 500 series including 525.1 and 508.1</li> </ul>
<b>ENVI™-18 DSK and ENVI™-8 DSK SPE Disks</b>	<ul style="list-style-type: none"> <li>• The SPE membrane equivalents of ENVI™-18 and ENVI™-8 packed bed SPE sorbents</li> <li>• Porous glass fiber membranes embedded with C18 or C8 silica particles</li> <li>• Provides faster flow rates and exhibits less clogging than PTFE discs for the extraction of organic contaminants from drinking water</li> <li>• Typical applications include PAHs, PCBs, phthalates, semivolatile organics, paraquat and diquat, pesticides and herbicides</li> <li>• Ideal for EPA 500 series including 525.1 and 508.1</li> </ul>
<b>ENVI™-8</b>	<ul style="list-style-type: none"> <li>• Available in glass tubes with PTFE frits</li> <li>• High 14% C loading for increased binding capacities and higher recoveries</li> <li>• Higher carbon loading also offers greater resistance to extreme pH conditions</li> <li>• Excellent for cleaning, extracting and concentrating pollutants from aqueous environmental samples</li> </ul>
<b>ENVI™-Chrom P (polystyrene divinylbenzene)</b>	<ul style="list-style-type: none"> <li>• Styrene/divinylbenzene co-polymer resin: Particle Size: 80-160 µm; Spherical Shape; Pore Size: 110-175 Å; Surface Area: 900 m<sup>2</sup>/g</li> <li>• Highly crosslinked, neutral, specially cleaned styrene-divinylbenzene resin used to retain hydrophobic compounds with some hydrophilic functionality under the reversed-phase mechanism</li> <li>• Highly resistant to extreme pH conditions</li> <li>• Typical applications include aromatic and phenolic compounds from aqueous sample matrices</li> <li>• Used for priority pollutant phenols from aqueous samples</li> </ul>
<b>ENVI-Carb™ and ENVI-Carb™ II (graphitized carbon black)</b>	<ul style="list-style-type: none"> <li>• Surface Area: 120 m<sup>2</sup>/g, Particle Size: 100/400 mesh (ENVI-Carb™-II: 120/140 mesh)</li> <li>• Extreme affinity for organic polar and non-polar compounds from both non-polar and polar matrices when used under reversed-phase conditions</li> <li>• Carbon surface comprised of hexagonal ring structures, interconnected and layered into graphitic sheets</li> <li>• Non-porous nature of the carbon phase allows for rapid processing, adsorption does not require analyte dispersion into solid phase pores</li> <li>• Independent investigators have found ENVI-Carb™ extremely useful for the rapid sample preparation of over 200 pesticides from various matrices including ground water, fruits and vegetables (see publication T196900 on our web site)</li> </ul>

For available configurations and part numbers, please see page 26.



## Ion-Exchange and Normal-Phase

The Supelclean™ SPE line represents one of the original brands to be introduced into the market place. It is referenced in hundreds of journal publications and validated in a variety of methods spanning environmental applications to the food and beverage industry. The Supelclean™ ENVI™ line was developed

and optimized for numerous environmental methods, including EPA 500 series (drinking water methods) and a number of SW-846 methods (solid waste).

For Supelclean™ silica specifications, see page 16. For general guidelines on ion-exchange and normal-phase SPE, see pages 50 and 51.

<b>LC-SAX</b>	<ul style="list-style-type: none"> <li>• A strong anion exchanger</li> <li>• Quarternary amine, Cl<sup>-</sup> counter-ion</li> </ul>
<b>LC-SCX</b>	<ul style="list-style-type: none"> <li>• Aliphatic sulfonic acid, Na<sup>+</sup> counter-ion, endcapped</li> </ul>
<b>LC-WCX</b>	<ul style="list-style-type: none"> <li>• Carboxylic acid, Na<sup>+</sup> counter-ion</li> </ul>
<b>LC-NH<sub>2</sub></b>	<ul style="list-style-type: none"> <li>• Monomerically bonded, aminopropyl (5% C)</li> </ul>
<b>PSA</b>	<ul style="list-style-type: none"> <li>• Polymerically bonded, ethylenediamine-N-propyl phase that contains both primary and secondary amines with pKa of 10.1 and 10.9</li> </ul>
<b>ENVI™-Florasil™</b>	<ul style="list-style-type: none"> <li>• Magnesium silicate, mesh: 100/200, available with PTFE or stainless steel frits</li> <li>• Tested for US Environmental Protection Agency (EPA) Contract Laboratory Program (CLP) statement of work for pesticides</li> <li>• Highly polar material that strongly adsorbs polar compounds from non-polar matrices under normal-phase conditions</li> <li>• Typical applications include alcohols, aldehydes, amines, herbicides, pesticides, PCBs, ketones, nitro compounds, organic acids and phenols</li> </ul>
<b>Dual Layer Florasil®/Na<sub>2</sub>SO<sub>4</sub></b>	<ul style="list-style-type: none"> <li>• Dual layer SPE Tube (available as glass or PP) that contains Na<sub>2</sub>SO<sub>4</sub> (upper layer) and Florasil® (magnesium silicate; lower layer) separated and packed with PTFE frits</li> <li>• Florasil®, activated, size- 60/100 mesh (150-200 μm), Na<sub>2</sub>SO<sub>4</sub> Purity- 99.99 %, Density- 2.68 g/mL</li> <li>• Excellent for removing/isolating polar compounds from organic matrices</li> <li>• Na<sub>2</sub>SO<sub>4</sub> layer aids in removing aqueous sample residues that may hinder Florasil® performance and/or subsequent GC analysis</li> <li>• Suitable for the determination of the hydrocarbon oil index in water (surface, waste and sewage treatment plants) by GC/FID analysis according to European Standard EN ISO 9377-2:2000 (enclosed in the Extraction Kit for EN ISO 9377-2 Cat. No. 68172)</li> <li>• Use in conjunction with Visiprep™ Large Volume Sampler (Cat. No.57275, only suitable for the PP version with PE frits 54116-U) and Visiprep™ SPE Vacuum Manifolds for processing larger volume samples</li> </ul>
<b>LC-Florasil®</b>	<ul style="list-style-type: none"> <li>• Magnesium silicate, mesh: 100/120</li> </ul>
<b>LC-Alumina A, N, and B</b>	<ul style="list-style-type: none"> <li>• Alumina-A for acidic pH (~5)</li> <li>• Alumina-N for neutral pH (~6.5)</li> <li>• Alumina-B for basic pH (~8.5)</li> <li>• Brockman Activation I for all Alumina SPE products, mesh: 60/325</li> </ul>
<b>LC-CN</b>	<ul style="list-style-type: none"> <li>• Monomerically bonded, cyanopropyl (7% C), endcapped</li> </ul>
<b>LC-Si</b>	<ul style="list-style-type: none"> <li>• Silica gel</li> </ul>
<b>LC-Diol</b>	<ul style="list-style-type: none"> <li>• Monomerically bonded, Diol (7% C), endcapped</li> </ul>

For available configurations and part numbers, please see page 18.

All SPE tubes listed consist of polypropylene hardware and PE frits unless noted otherwise. Color coded footnotes denote differences in hardware, package size or bed weight from the standard configuration.

	Description	0.1 g/1 mL pk 108	0.5 g/3 mL pk 54	0.5 g/6 mL pk 30	1 g/6 mL pk 30	2 g/12 mL pk 20	5 g/20 mL pk 20	10 g/60 mL pk 16	100 g bulk
Reversed-Phase	ENVI™-18	57062	57063	57064 ●54331-U <sup>1</sup>	505706	57114	57137	57138	57219
	ENVI™-18 DSK SPE Disks			●57171 <sup>12</sup>	●57170-U <sup>13</sup>				
	ENVI™-8 DSK SPE Disks			●57172 <sup>12</sup>					
	LC-18	504270	57012	57054	505471	57117	Custom	57136	57202
	ENVI™-8	Custom	57231	57232	57233		Custom	Custom	
	LC-8	Custom	●Custom	Custom					
	ENVI™-Chrom P	57143	505145	57052					57201
			●57224 <sup>5</sup>	57226					●57217 <sup>11</sup>
				●57225-U <sup>7</sup>					
	ENVI-Carb™	57109-U	●57088 <sup>5</sup>	57094		57128	Custom	57130	●57210-U <sup>11</sup>
			●57092 <sup>7</sup>		●57127-U <sup>10</sup>				
ENVI-Carb™ C, mesh 80/100					Custom				
LC-4 (Wide Pore)		57089							
LC-Ph	504599	505269							
LC-CN	504386	57013	57056			Custom			
LC-Diol	Custom	57016							
ENVI™-Florisil™		●57058 <sup>2</sup>	●57046 <sup>3</sup>	●57053 <sup>3</sup>					
				●54095-U <sup>1</sup>					
Dual Layer Florisil®/ Na <sub>2</sub> SO <sub>4</sub>				●52582-U <sup>1,9</sup>					
				●●54116-U <sup>2,9</sup>					
LC-Florisil®			●54333-U <sup>1</sup>	57057	57115	57131	57132	57209	
				●54334-U <sup>1</sup>					
LC-Alumina A		●57082-U <sup>6</sup>		●57083-U <sup>8</sup>				57026	
LC-Alumina B		●57084 <sup>6</sup>		●57085 <sup>8</sup>				Custom	
LC-Alumina N		●57086 <sup>6</sup>		●57087 <sup>8</sup>				57028	
LC-Si	504041	505048	505374	57051	57116	57133	57134	57200	
				●54335-U <sup>1</sup>					
Ion Exch.	LC-NH <sub>2</sub>	504483	57014	54059-U					57205
	PSA		●52578-U <sup>4</sup>	52579-U					52738-U
	LC-SAX	504815	57017						57203
	LC-SCX	504920	57018						Custom
	LC-WCX	505595	57061						

Footnotes/Color Codes

●<sup>1</sup> glass SPE tubes, PTFE frits

●<sup>2</sup> PP SPE tubes, PTFE frits

●<sup>3</sup> PP SPE tubes, stainless steel frits

●<sup>4</sup> 0.2 g/3 mL, pk 54

●<sup>5</sup> 0.25 g/3 mL, pk 54

●<sup>6</sup> 1 g/3 mL, pk 54

●<sup>7</sup> 0.25 g/6 mL

●<sup>8</sup> 2 g/6 mL, pk 30

●<sup>9</sup> 2 g/2 g/6 mL, pk 48

●<sup>10</sup> 1 g/12 mL, pk 20

●<sup>11</sup> 50 g bulk

●<sup>12</sup> 47 mm diam. disks, pk 24

●<sup>13</sup> 90 mm diam. disks, pk 12

## Multi-Layer SPE

Developed to provide superior cleanup when conducting multi-residue pesticide analysis in food/agricultural matrices. See also the new dual layer Supel™ Sphere products containing spherical materials on page 29.

Description	Qty.	Cat. No.
<b>ENVI-Carb™-II/PSA</b>		
0.3 g/0.6 g/6 mL	30	<b>54058-U</b>
0.5 g/0.5 g/6 mL	30	<b>54067-U</b>
0.5 g/0.3 g/6 mL	30	<b>55119-U</b>
0.5 g/0.5 g/20 mL	20	<b>54217-U</b>
<b>SAX/PSA</b>		
0.5 g/0.5 g/6 mL	30	<b>52577-U</b>

Description	Qty.	Cat. No.
<b>ENVI-Carb™/LC-NH<sub>2</sub></b>		
0.5 g/0.5 g/3 mL	20	<b>54332-U</b>
0.5 g/0.5 g/20 mL	20	<b>54216-U</b>
0.5 g/0.5 g/6 mL	300	<b>54024-U</b>
0.5 g/0.5 g/6 mL	30	<b>54035-U</b>
<b>ENVI-Carb™/NH<sub>2</sub>/Silica</b>		
0.5 g/0.4 g/0.6 g/20 mL	20	<b>54036-U</b>
<b>Dual Layer Florisil®/Na<sub>2</sub>SO<sub>4</sub></b>		
Glass tubes, PTFE frits, 2 g/2 g/6 mL	48	<b>52582-U</b>
PP tube with PE frits 2 g/2 g/6 mL	48	<b>54116-U</b>

# LiChrolut® SPE Products

## Reverse Phase, Normal Phase & Ion Exchange

The LiChrolut® SPE line also represents one of our original brands. The table below contains information about the typical applications for each LiChrolut® product. This selection guide will help you select the right product for your application needs.

Application	LiChrolut® extraction column	Typical sample matrix	Typical sample substances	Typical elution solvent
Non-polar extraction	RP-18 RP-18e (endcapped) CN	Aqueous buffer solution	Aromatic ring systems, compounds with alkyl chains	Acetonitrile, methanol, ethyl acetate
Cation exchange extraction	SCX (strong)	Methanolic/aqueous buffer with low ionic strength; 2 pH units under pK value of the sample substance	Cations: amines, pyrimidines	Aqueous buffer of high ionic strength (0.1 mol/L); 2 pH units over pK value of the sample substance
Mixed mode extraction	TSC (Tox Screening Cation)	Body fluids (not for <i>in vitro</i> )	Cationic and neutral analytes	Chloroform-acetone, NH <sub>3</sub> -ethyl-acetate or NH <sub>3</sub> -methanol
Medium polar extraction of environmental pollutants	Florisil®	Waste/ground/drinking water, soil samples	Herbicides, pesticides, PCBs, PCPs, dioxins, phenols, nitro compounds, HCHs	n-Hexane, dichloromethane

Description	Qty.	Cat. No.
<b>LiChrolut® Florisil® (150 - 250 µm)</b>		
1000 mg/6 mL	30	<b>1.19127.0001</b>
<b>LiChrolut® RP-18 (40 - 63 µm)</b>		
100 mg/1 mL	100	<b>1.19855.0001</b>
200 mg/3 mL	50	<b>1.02014.0001</b>
500 mg/3 mL	50	<b>1.02023.0001</b>
500 mg/6 mL	30	<b>1.19687.0001</b>
1000 mg/6 mL	30	<b>1.02122.0001</b>
2000 mg/6 mL	30	<b>1.19686.0001</b>

Description	Qty.	Cat. No.
<b>LiChrolut® RP-18e (40 - 63 µm)</b>		
200 mg/3 mL	50	<b>1.19847.0001</b>
500 mg/3 mL	50	<b>1.19849.0001</b>
<b>LiChrolut® SCX (40 - 63 µm)</b>		
200 mg/3 mL	50	<b>1.02016.0001</b>
500 mg/3 mL	50	<b>1.02022.0001</b>
<b>LiChrolut® TSC (40 - 63 µm)</b>		
300 mg/3 mL		<b>1.19767.0001</b>

\*glass SPE tube

# Replace Classical LLE with EXtrelut® NT

## SLE: Emulsion-Free Supported-Liquid Extractions

Classical liquid-liquid extraction (LLE) using a separation funnel is often associated with certain disadvantages: Formation of emulsion, poor phase separation, high solvent consumption, low degree of automation and high personnel costs. EXtrelut® NT simplifies liquid-liquid extraction by replacing separation funnels. Using a single step is more efficient and provides solvent, material, and time savings in comparison to classical funnel separation.

### Specifications of EXtrelut® NT

<b>Characteristics</b>	Specially processed, wide-pore diatomaceous earth with a high pore volume		
	Chemically inert Naturally occurring product		
<b>Capacity limit with aqueous sample</b>	EXtrelut® NT1	1 mL	<b>without any breakthrough</b>
	EXtrelut® NT3	3 mL	
	EXtrelut® NT20	20 mL	
<b>pH range</b>	pH 1-10		
<b>Uniform batch-to-batch quality</b>			

### Benefits of EXtrelut® NT over LLE

- Minimal solvent usage
- Simple method
- Higher sample capacity and throughput
- Emulsion free extracts
- Higher purity, suitable for trace analysis

EXtrelut® NT SLE sorbent is extremely versatile and can be used for biological samples, water analysis, food and beverage, and environmental applications. Any LLE of aqueous samples can be easily replaced with EXtrelut® NT supported liquid extraction.

With its easy-to-use working principle a higher recovery and cleaner extraction can be achieved. The aqueous sample is simply applied to the LLE of aqueous samples. It distributes itself in the form of a thin film over the chemically inert matrix and thus acts as a stationary phase.

Subsequently, elution takes place using organic solvents that are non miscible with water, solvents like e.g. diethyl ether, ethyl acetate or



halogenated hydrocarbons. All the lipophilic substances are extracted from the aqueous into the organic phase. During this process the aqueous phase remains on the stationary phase. The eluate is free from emulsions and can be evaporated for further analysis.

1 mL	3 mL	20 mL
EXtrelut® NT1	EXtrelut® NT3	EXtrelut® NT20

Maximum aqueous sample capacity

### The capacity of EXtrelut® NT pre-packed columns for aqueous samples are specified by the designation

Significantly smaller samples must be appropriately diluted. If larger volumes are applied, the columns are overloaded; water breaks through into the solvent. Elution is carried out with 2-3 times the sample volume. The liquid may simply be allowed to run through the column by gravity. The column outlet cannula regulates the solvent flow appropriately.

### Important EXtrelut® NT extraction parameters

EXtrelut® NT extraction columns	Outlet cannulae	Maximum sample volume (mL)	Waiting period (mn)	Recommended elution volume (mL)
EXtrelut® NT1	0.60 x 30 mm	1	5 - 10	6
EXtrelut® NT3	0.60 x 30 mm	3	5 - 10	15
EXtrelut® NT20	0.70 x 30 mm	20	10 - 15	40

1. In order to prevent water breaking through the sample, don't overload the column.
2. Shorter waiting times can affect the recoveries adversely.
3. The recommended sample volumes must be adhered to. Solutions of smaller volumes must be diluted to give indicated volumes.

## Application: HILIC separation of antiepileptic drugs (AEDs) in serum after EXTrelut® NT SLE

EXTrelut® NT has been used for quite some time within research, for the sample preparation of urine, whole blood, plasma, serum, gastric juice, liquor, amniotic fluid, feces, animal and plant tissue. Other applications are in the areas of environmental and residue analysis,

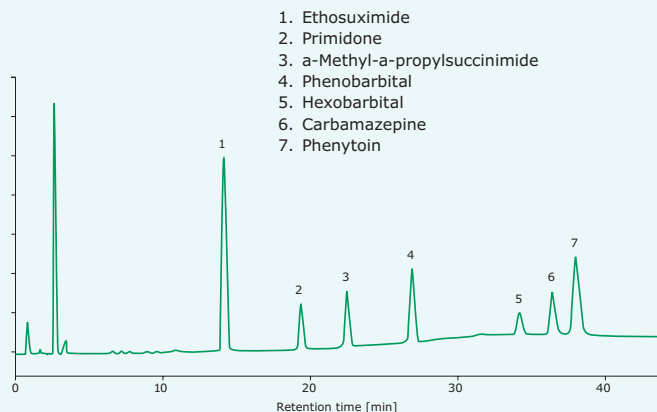
e.g. the analysis of industrial, domestic and waste water. The fractionated elution of acidic and basic substances (e.g. drugs and their metabolites) from body fluids is also possible.

**Figure 13.** HILIC Separation of Antiepileptic Drugs (AEDs) After EXTrelut® NT SLE Cleanup

HPLC: LaChrom system  
 column: LiChrospher® RP-select B (5 µm) LiChroCART® 250-4  
 mobile phase: A: Water LiChrosolv® Acetonitrile LiChrosolv® (1+1)  
 B: Water LiChrosolv®

gradient:	Time [min]	% A	% B
	0	10	90
	30	60	40
	44	60	40
	44.1	100	0
	50	100	0
	51	10	90
	75	10	90

flow: 1 mL/min  
 temperature: 30 °C  
 detection: UV 205 nm



### Determination of antiepileptic drugs (AEDs) in serum

500 µL serum  
 500 µL phosphate buffer\*



Apply in sequence onto the column

EXTrelut® NT1



Wait 8 minutes

1 mL dichloromethane / 2-propanol (9+1)



Wait 10 minutes then elute with

6 mL dichloromethane / 2-propanol (9+1)



Evaporate to dryness under nitrogen stream

Redissolve residue in 1 mL of methanol



Inject 10 µL into HPLC column

\* 17.6 g NaH<sub>2</sub>PO<sub>4</sub>, 4.5 g Na<sub>2</sub>HPO<sub>4</sub> · 2 H<sub>2</sub>O, 1.5 g NaN<sub>3</sub>, dissolve in 1 L water (pH 6.0-6.1)

### Recoveries [mean values N = 3]

Ethosuximide*	14.1 min	84 ± 7 %
Primidone	19.4 min	100 ± 2 %
a-Methyl-a-propylsuccinimide	22.5 min	Internal standard
Phenobarbital	26.9 min	96 ± 2 %
Hexobarbital	34.2 min	99 ± 2 %
Carbamazepine	36.4 min	97 ± 1 %
Phenytoin	38.0 min	100 ± 1 %

\*Ethosuximide is volatile on evaporation



EXTrelut® NT collection tube for  
 EXTrelut® NT1 and EXTrelut® NT3  
 glass columns

## EXTrelut® NT pre-packed columns

Description	Qty.	Cat. No.
EXTrelut® NT1 glass columns for 0.1 to 1 mL sample solution	100 columns	1.15094.0001
EXTrelut® NT3 glass columns for 1 to 3 mL sample solution	50 columns	1.15095.0001
EXTrelut® NT20 polyethylene columns including special outlet cannulae for up to 20 mL sample solution	25 columns	1.15096.0001

## EXTrelut® NT packing material

Description	Qty.	Cat. No.
EXTrelut® NT bulk packing for preparing large-volume columns	1 kg	1.15092.1000
EXTrelut® NT refill packs for refilling 50 EXTrelut® NT20 columns (incl. replacement filters)	50 bags	1.15093.0001

## EXTrelut® NT accessories

Description	Qty.	Cat. No.
EXTrelut® NT accessories cannulae .60/30 with Luer tip for EXTrelut® NT1 and EXTrelut® NT3	100 pieces	1.15373.0001
EXTrelut® NT collection tubes with tapered bottom and screw cap (normal capacity 15 mL) for EXTrelut® NT1 and EXTrelut® NT3	30 pieces	1.15622.0001
Replacement filter for EXTrelut® NT1 (10 mm Ø)	100 pieces	1.14236.0001
Replacement filter for EXTrelut® NT3 (15 mm Ø)	100 pieces	1.14237.0001
Replacement filter for EXTrelut® NT20 (24 mm Ø)	50 pieces	1.14567.0001



EXTrelut® NT – Packing Material

### Need help choosing? Want more information on initial setup?

Our Tech Service Team is happy to help get you started.  
Email our experts at:

[SigmaAldrich.com/support/customer-support](mailto:SigmaAldrich.com/support/customer-support)

For more information or to order, visit

[SigmaAldrich.com/EXTrelut](http://SigmaAldrich.com/EXTrelut)

# Empore™ SPE Disks

## For Large Volume Aqueous Samples

Empore™ solid phase extraction products are produced by trapping sorbent particles within an inert matrix of an engineered polymer. The resulting particle loaded membrane yields a more uniform and more densely packed particle bed than traditional loosely packed SPE products.

The Empore™ SPE disk line is the most complete line of SPE disks for extracting large volumes of aqueous samples. The product line ranges from time-tested C18 to unique phase chemistries such as carbon and the oil and grease disk. The disks are ideal for environmental analysis where 1 L sample volumes are not uncommon and provide an efficient alternative to liquid-liquid extraction (LLE).

### Empore™ SPE Disks are:

- Amenable to dozens of EPA and related environmental methods
- Developed for the efficient extraction of pollutants in large volume water samples



### Product Specifications

<b>Compositions</b>	C8, C18, Anion, Cation, MPC, SDP-RPS, SDB-XC, Chelator	≥90% sorbent particles ≤10% inert polymer matrix
	Carbon	≥80% sorbent particles ≤20% inert polymer matrix
<b>Thickness</b>	0.50±0.05 mm	
<b>SPE Flow Rate</b>	<10 min L <sup>-1</sup> DI H <sub>2</sub> O @ 25 °C @ in Hg (47 mm disk)	
<b>Particle Size</b>	12 μm (nominal) for HD, 50 μm (nominal) for SD	
<b>Solvents</b>	Compatible with all organic solvents	
<b>pH Range</b>	Silica-based sorbents	2-12 under normal conditions
	Resin-based sorbents	1-14 under normal conditions

### Empore™ Extraction Disks

Sorbent	Suggested Application	EPA Method	Disk Size (mm)	Cat. No.
C8 HD	moderately nonpolar	549.1	47	66882-U
C18 HD	highly nonpolar	506, 508.1, 525.2, 550.2, 608, 1613B	47	66883-U
SDB-XC	water soluble, moderately polar analytes	515.2, 525.3	47	66884-U
SDB-RPS	moderately nonpolar and cation exchange	--	47	66886-U
Cation-SR Exchange	metals, amines	--	47	66889-U
Anion-SR Exchange	chromium, arsenic, selenium, carboxylic acids, etc.	548.1, 552.1	47	66888-U
Oil & Grease	nonpolar, dirty samples	1664	47 90	66887-U 66898-U
Chelating	divalent metals and other cations	--	47	66894-U
Activated Carbon	water soluble and volatile organic compounds	--	47	66896-U
Filter Aid	removal of large particulates in dirty samples	--	--	66897-U

# Products for PFAS Testing

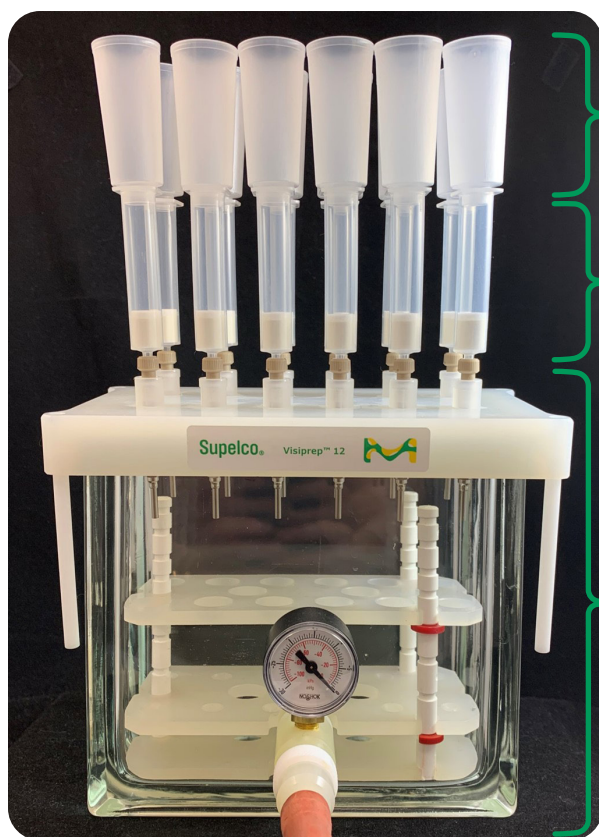
## Supelclean™ ENVI-WAX™ and ENVI™-Chrom P, QuEChERS

Perfluoroalkyl substances (PFAS) are a group of human-made Organofluorine compounds, a class of highly fluorinated substances. PFAS compounds are also commonly known as “forever chemicals” which means they do not break down in the environment like other chemicals. This persistence can result in the concentration of these compounds growing to levels that are unsafe for human exposure with possible negative health effects such as: low infant birth weights, immune system dysfunction, cancer, and thyroid hormone disruption.

### Supelclean™ SPE Cartridges

Multiple regulatory methods, such as EPA 537 and 533, detail the extraction of PFAS analytes from drinking water using SPE cartridges followed by analysis by LC/TQ. Most commonly, weak anion exchange (WAX) cartridges, such as Supelclean™ ENVI-WAX™ SPE cartridges, are used due to their ability to extract both short and long-chain PFAS analytes with good recoveries as seen in EPA 533 and ISO methods (ISO 25101 and ISO 21675).

EPA 537 uses a polystyrene divinylbenzene (PS-DVB) cartridge, such as a Supelclean™ ENVI™-Chrom P SPE cartridge, which offers high recoveries for medium and long-chain PFAS analytes.



Large Volume SPE Reservoir (Cat. No. 54258-U) for cartridge extractions

Supelclean™ ENVI™-Chrom P (for EPA 537.1) or Supelclean™ ENVI-WAX™ SPE Cartridges (for EPA 533)

### Visiprep™ Vacuum Manifolds

The Visiprep™ system contains a patented valve system that allows for precise flow control through each SPE tube via rotating, independent, screw-type valves situated in each port within the manifold cover. Visiprep™ vacuum manifolds allow you to process up to 12 (12-port version) or 24 (24-port version) PFAS samples simultaneously.

### QuEChERS Tubes and Salts

The “QuEChERS” method (Quick, Easy, Cheap, Effective, Rugged, and Safe), has emerged as a sample prep technique popular e.g. in the area of matrix rich samples, like food product. The PFAS testing in food is formalized in FDA method C-010.02. In a first step, food sample is extracted with an aqueous miscible solvent (e.g., acetonitrile) in the presence of high amounts of extraction salts to induce liquid phase separation. Upon shaking and centrifugation, an aliquot of the organic phase is subjected to further



cleanup step. The second step is facilitated by mixing bulk amounts of sorbent (e.g., Supelclean™ PSA, ENVI-Carb™, MgSO<sub>4</sub>) with the extract. After sample cleanup, the mixture is centrifuged and the resulting supernatant can, depending on the sample, either be analyzed directly or can be subjected to further minor treatment before analysis.

## Large Volume Reservoirs

A large volume reservoir enables you to transfer PFAS water samples directly from any sample container to conventional solid phase extraction tubes on a Visiprep™ SPE vacuum manifold. The large volume reservoir is used in both EPA 537 and EPA 533.

## EPA 533 for Drinking Water

We tested our Supelclean ENVI-WAX™ SPE cartridges using the method outlined in EPA 533. Per EPA method 533 the recovery of the laboratory spiked blank water samples should fall in the range 70-130%, and the recovery of stable isotope surrogates should fall in the range 50-200% with reproducibility of better than 20%. **Figure 14** demonstrates the laboratory spiked UHPLC-MS water blanks where the recoveries for 25 compounds met the EPA method requirements. Recoveries of all 16 surrogates (**Figure 14**) were also within the specified method range. **Figure 14** presents %RSD for each of the 25 compounds indicating that less than 20% RSD requirement was met.

### Chromatography conditions:

<b>Column:</b>	Ascentis® Express PFAS HPLC Column, 3 µm, 15 cm x 2.1 mm (Cat. No. <b>53560-U</b> )
<b>Delay column:</b>	Ascentis® Express PFAS Delay Column, 3 µm, 5 cm x 2.1 mm (Cat. No. <b>53572-U</b> )
<b>Mobile Phase:</b>	(A) 20 mM Ammonium Acetate, (B) Methanol
<b>Gradient:</b>	

Find more about PFAS Testing Solutions at:  
[SigmaAldrich.com/PFAS](https://www.sigmaaldrich.com/PFAS)

## Supelclean™ SPE Cartridges

For EPA 533, EPA 1633, ISO 25101, and ISO 21675

Description	Cat. No.
Supelclean™ ENVI-WAX™ SPE Cartridges, 500 mg, Pk. 30	<b>54057-U</b>
Supelclean™ ENVI-WAX™ SPE Cartridges, 200 mg, Pk. 30	<b>54056-U</b>

For EPA 537 and EPA 537.1

Description	Cat. No.
Supelclean™ ENVI™-Chrom P SPE Cartridges, 500 mg, 6 mL, Pk. 30	<b>57226</b>
Supelclean™ ENVI™-Chrom P SPE Cartridges, 500 mg, 6 mL, for use with Gerstel® MPS 3, Pk. 30	<b>57239-U</b>

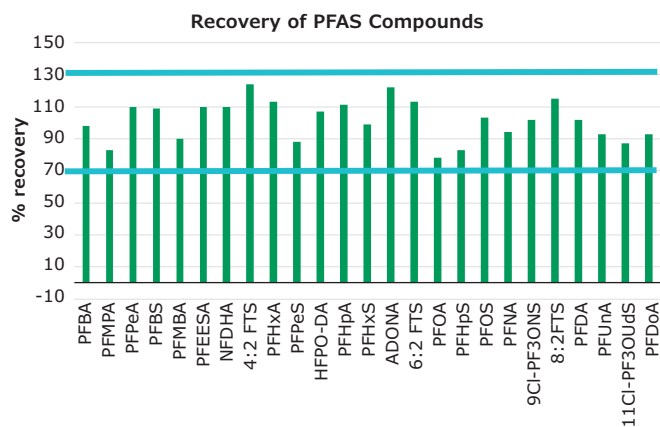


Figure 14.

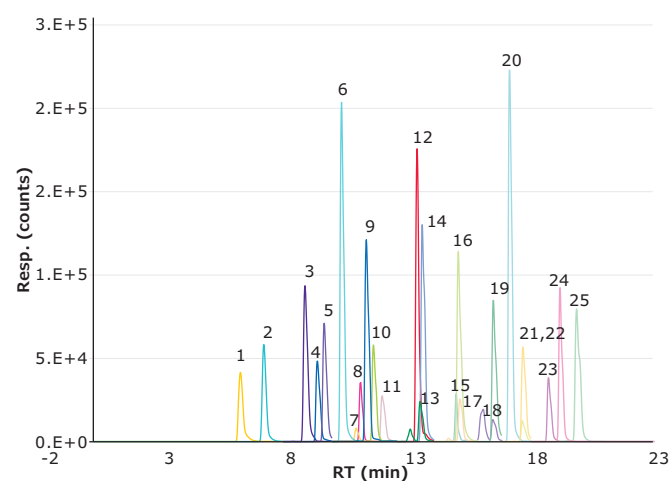


Figure 15.

## Accessories

Description	Cat. No.
Visiprep™ Solid Phase Extraction Manifold, 12-Port Model	<b>57030-U</b>
Visiprep™ Solid Phase Extraction Manifold, 24-Port Model	<b>57250-U</b>
Large Volume SPE Reservoir, Polypropylene, Pk. 30	<b>54258-U</b>

Description	Comment	Cat. No.
<b>FDA C_0.10.02</b>		
Supel™ QuE, Non-Buffered tube 2, pk of 50	Extraction salts	<b>55295-U</b>
BRAND® PP graduated centrifuge tube, screw cap	Empty tube	<b>BR114820</b>
pk of 50 Supel™ QuE PSA/ ENVI-Carb™ Tube 3, 15 mL	Clean-up tube with sorbent, 900 mg MgSO <sub>4</sub> , 300mg PSA, 150 mg ENVI-Carb	<b>55479-U</b>

## Environmental Analysis

### Supelclean™ Coconut Charcoal SPE Tube for Nitrosamines in Drinking Water

- Developed specifically for EPA Method 521 – Nitrosamines in Drinking Water
- Activated coconut charcoal stationary phase – particle size: 80/120 mesh
- Quality controlled for low fines and nitrosamine recovery

Description	Qty.	Cat. No.
Supelclean™ Coconut Charcoal SPE Tube, 2 g/6 mL	30	57144-U
Female Luer Coupler	20	21015
Male Luer Coupler	20	25064-U

### Supelclean™ Sulfoxide SPE for PCB's from Transformer, Waste and Mineral Oil

- Developed for the extraction of polychlorinated biphenyls (PCBs) from transformer, waste and mineral oil
- Silica-bonded sulfoxide (-SO) phase
- PCB retention facilitated by interaction between the SPE phase's electrophilic sulfur atom and the pi-electron cloud formed from aromatic rings inherent with PCBs
- Simple and efficient sample prep method for identifying PCBs at quantitation limits of 0.5 ppm



Description	Qty.	Cat. No.
Supelclean™ Sulfoxide SPE, 3 g/6 mL	30	55253-U
Supelclean™ Sulfoxide, Bulk, 100 g	1	55254-U
Disposable PTFE liners	100	57059
Large volume reservoir (25 mL) for 6 mL SPE tubes, PP	30	54258-U
Large volume reservoir (25 mL) for 6 mL SPE tubes, PTFE	3	54259-U

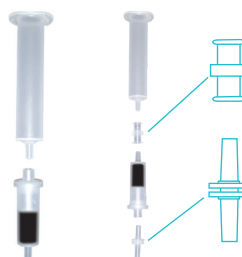
### Supelclean™ ENVI-Carb™ Plus Reversible SPE for Highly Polar Compounds from Aqueous Samples

- Spherical carbon particles (carbon mol sieve) developed for the SPE of highly polar compounds from aqueous samples as drinking or ground water
- Offers extreme affinity to organic polar and non-polar compounds from both non-polar and polar matrices when used under reversed-phase conditions
- Strong high surface spherical particles which are less friable (fines) than traditional graphitized carbon blacks
- When used in conjunction with an SPE vacuum manifold, a male luer coupler (25064-U), female luer coupler (21015) and empty SPE tube(s) are required but not included

Examples of highly polar compounds recovered

- Acephate (LogPo/w: -0.85)
- Phenol (LogPo/w: 1.51)
- 1,4-dioxane (LogPo/w: -0.27)
- Oxamyl (LogPo/w: -1.2)


Description	Qty.	Cat. No.
Supelclean™ ENVI-Carb™ Plus Reversible SPE Tube, 0.4 g/1 mL	30	54812-U
Female Luer Coupler	20	21015
Male Luer Coupler	20	25064-U



# Pesticide Analysis

Unlike typical “bind and elute” SPE practices, the modern strategy for SPE cleanup prior to routine multi-residue pesticide analysis is removal/trapping of the majority of the matrix by the sorbent phase, while the analytes of interest pass through. This results in a purified eluate. The use of packed SPE tubes, often with 2 layers of sorbent, is common. Likewise,

the “QuEChERS” approach (pg. 35) using bulk SPE materials has been incorporated into a number of methods. In all cases, the purity and the efficiency of the adsorbents used are the key to reliable and reproducible pesticide determination. With expertise in particle technology, we provide quality SPE products.

<b>Supelclean™ Ultra</b>	<ul style="list-style-type: none"> <li>• Designed for the cleanup of extracts of difficult matrices such as dry commodities (tea, spices, coffee, etc.)</li> <li>• Dual layer SPE tube contains a mixture of PSA/C18 and graphitized, spherical carbon (upper layer), and zirconia-coated silica (bottom layer)</li> <li>• PSA removes acidic interferences, C18 retains some hydrophobic interferences, and specialized carbon removes pigments while allowing for better recoveries of compounds with planar structures</li> <li>• Zirconia-coated silica (Z-Sep) removes oily residues and provides additional pigment removal</li> </ul>
<b>Supel™ Sphere Carbon/NH<sub>2</sub></b>	<ul style="list-style-type: none"> <li>• SPE tube packed entirely with spherical, non-friable particles</li> <li>• Improved flow characteristics and faster flow for gravity filtration</li> <li>• Reduced susceptibility to the formation of fines</li> <li>• Dual layer SPE tube contains both spherical carbon (upper layer) and spherical silica-aminopropyl phase (lower layer), SPE sorbents are separated by a PE frit</li> <li>• Developed to offer superior cleanup when conducting multi-residue pesticide analysis from food</li> <li>• Carbon has a strong affinity toward planar molecules, and can isolate/remove pigments (eg., chlorophyll and carotinoids) and sterols commonly present in foods and natural products</li> <li>• Aminopropyl (NH<sub>2</sub>) retains fatty acids, organic acids, and some polar pigments and sugars common in food matrices</li> </ul>
<b>ENVI-Carb™-II/ PSA</b>	 <ul style="list-style-type: none"> <li>• Dual layer SPE tube that contains both Supelclean™ ENVI-Carb™-II (upper layer) and PSA (lower layer) SPE sorbents (separated by PE frit)</li> <li>• Developed to offer superior cleanup when conducting multi-residue pesticide analysis in food (e.g., fruits, vegetables, etc.)</li> <li>• ENVI-Carb™-II is a graphitized non-porous carbon (100/140 mesh, surface area 100 m<sup>2</sup>/g) that has a strong affinity towards planar molecules, and has been quality controlled specifically for the isolation/removal of pigments (e.g., chlorophyll and carotinoids) and sterols commonly present in fruits, vegetables and other natural products</li> <li>• Supelclean™ PSA is a polymerically bonded, ethylenediamine-N-propyl phase that contains both primary and secondary amines</li> <li>• Supelclean™ PSA has a strong affinity and high capacity for fatty acids, organic acids, and some polar pigments and sugars</li> <li>• Tested for superior cleanliness using GC/FID and GC/MS</li> </ul>
<b>ENVI-Carb™-II/ SAX/PSA</b>	<ul style="list-style-type: none"> <li>• Tri-layer SPE tube that contains Supelclean™ ENVI-Carb™-II (upper layer), SAX (middle layer) and PSA (lower layer) SPE sorbents (separated by PE frit)</li> <li>• Developed to offer superior cleanup when conducting multi-residue pesticide analysis in food (e.g., fruits, vegetables, etc.)</li> <li>• ENVI-Carb™-II is a graphitized non-porous carbon (100/140 mesh, surface area 100 m<sup>2</sup>/g) that has a strong affinity towards planar molecules, and has been quality controlled specifically for the isolation/removal of pigments (e.g., chlorophyll and carotinoids) and sterols commonly present in fruits, vegetables and other natural products</li> <li>• Supelclean™ PSA has a strong affinity and high capacity for fatty acids, organic acids, and some polar pigments and sugars</li> <li>• Supelclean™ SAX offers additional ion-exchange capacity for removing matrix components that may induce ion-suppression or enhancement during GC analysis</li> </ul>
<b>SAX/PSA</b>	<ul style="list-style-type: none"> <li>• Dual layer SPE tube that contains both Supelclean™ SAX (upper layer) and PSA (lower layer) SPE sorbents (separated by PE frit)</li> <li>• Supelclean™ SAX is a quarternary amine, Cl<sup>-</sup> counter-ion</li> <li>• Supelclean™ PSA is a polymerically bonded, ethylenediamine-N-propyl phase that contains both primary and secondary amines</li> <li>• Ideal for removing matrix components (fatty acids, organic acids, polar pigments and some sugars) when conducting multi-residue pesticide analysis in foods</li> <li>• In compliance with Luke and Luke II methods that use SPE to reduce matrix induced ion-suppression and enhancement when conducting GC analysis of pesticides in food</li> </ul>
<b>ENVI-Carb™</b>	<ul style="list-style-type: none"> <li>• Surface Area: 120 m<sup>2</sup>/g, Particle Size:100/400 mesh</li> <li>• Extreme affinity for organic polar and non-polar compounds from both non-polar and polar matrices when used under reversed-phase conditions</li> <li>• Carbon surface comprised of hexagonal ring structures, interconnected and layered into graphitic sheets</li> <li>• Non-porous nature of the carbon phase allows for rapid processing, adsorption does not require analyte dispersion into solid phase pores</li> <li>• Independent investigators have found ENVI-Carb™ extremely useful for the rapid sample preparation of over 200 pesticides from various matrices including ground water, fruits and vegetables</li> </ul>
<b>PSA</b>	 <ul style="list-style-type: none"> <li>• Polymerically bonded, ethylenediamine-N-propyl phase that contains both primary and secondary amines</li> <li>• A weak anion exchanger with a pKa of 10.1 and 10.9</li> <li>• Similar to aminopropyl SPE phases (NH<sub>2</sub>) in terms of selectivity, but has a much higher capacity due to presence of secondary amine (0.98-1.05 meq/g)</li> <li>• Strong affinity and high capacity for removing fatty acids, organic acids, and some polar pigments and sugars when conducting multi-residue pesticide analysis in foods</li> <li>• Has been shown to significantly reduce matrix-enhancement effects encountered during the GC analysis of food products</li> <li>• Bidentate nature of ligands allow for chelation</li> </ul>

## Supelclean™ Ultra

Supelclean™ Ultra solid phase extraction (SPE) cartridges were designed for the cleanup of extracts of difficult matrices such as dry commodities (tea, spices, coffee, etc.) prior to pesticide residue analysis, typically performed by GC/MS/MS and LC/MS/MS. These types of samples can contain highly concentrated pigments and oils, which may not be sufficiently cleaned using a standard QuEChERS cleanup. With little solvent usage, Ultra cartridges provide a cleaner extract and improved recovery of planar pesticides over traditional SPE cartridges without the use of toluene. By removing problematic interferences, these cartridges enable analysts to achieve detection of analytes at the ppb level.

In a recent study, green tea was spiked at 5 and 50 ng/g and extracted using QuEChERS. Cleanup using a 1 mL Supelclean™ Ultra 2400 cartridge was then compared with QuEChERS cleanup using PSA/C18/GCB. The final extracts were analyzed by LC/MS/MS and GC/MS/MS. Performance of the cleanups was compared with regards to background and pesticide recoveries.

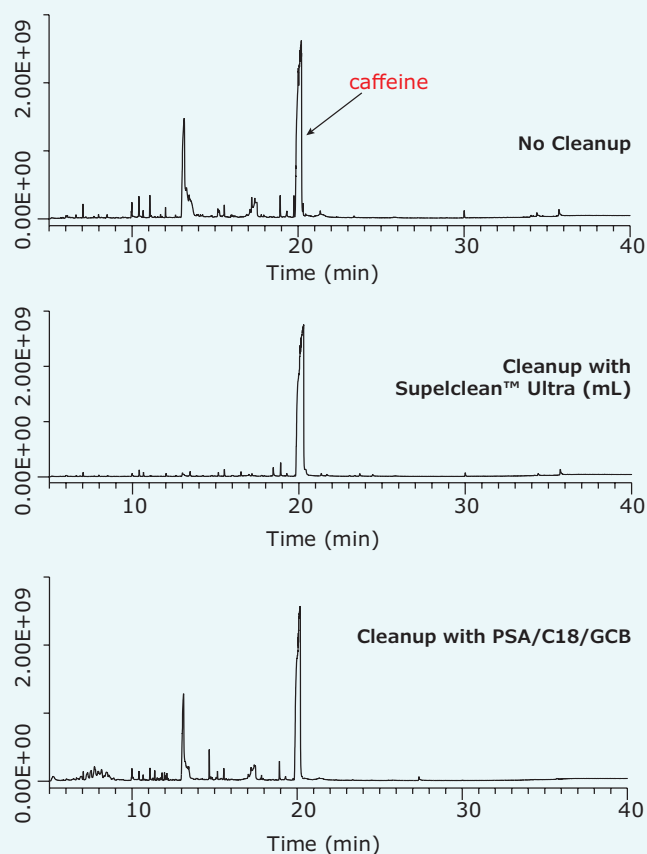
**Figure 16** shows that Supelclean™ Ultra 2400 SPE was found to provide lower background than QuEChERS cleanup using PSA/C18/GCB. This allowed for the analysis of more pesticides at lower levels. These cartridges are advantageous because they use little solvent, and do not require the use of toluene in the elution solvent to release planar pesticides.

### Supelclean™ Ultra SPE Products

Description	Qty.	Cat. No.
<b>Supelclean™ Ultra 2400 (2 beds)</b>		
120 mg PSA, C18, spherical carbon mix/100 mg Z-Sep, 1 mL	108	<b>52779-U</b>
270 mg PSA, C18, spherical carbon mix/225 mg Z-Sep, 3 mL	54	<b>54281-U</b>

For more information, visit [SigmaAldrich.com/supelcleanultra](http://SigmaAldrich.com/supelcleanultra)

**Figure 16. GC/MS Scan Analyses of Green Tea Extracts**



## Supel™ Sphere Carbon/NH<sub>2</sub>

### Features and Benefits

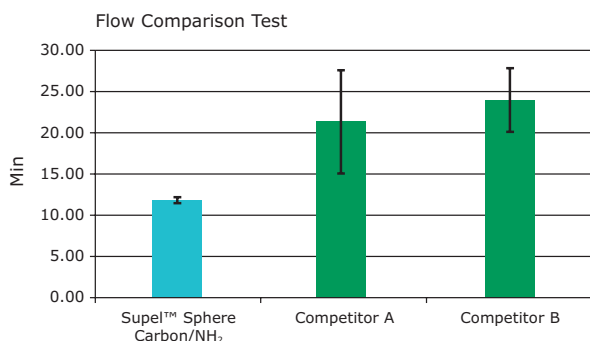
- SPE tube packed entirely with spherical, non-friable particles
- Improved flow characteristics and faster flow for gravity filtration use
- Reduced susceptibility to the formation of fines
- Carbon removes pigments and sterols, commonly present in many food and natural products
- Aminopropyl (NH<sub>2</sub>) removes organic acids, polar pigments and sugars

### Spherical SPE Materials Optimize Flow and Increase Throughput

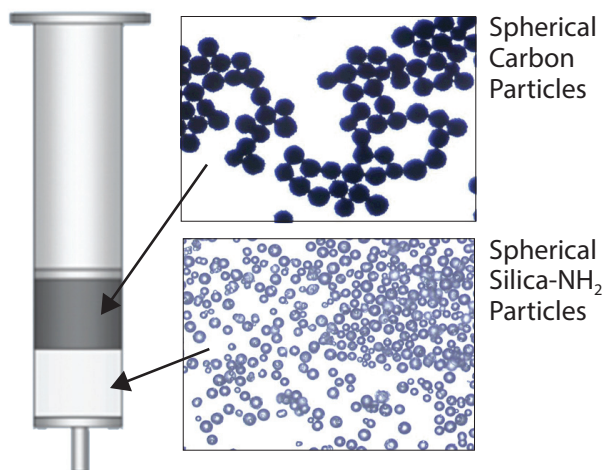
The demand for SPE cartridges with improved flow characteristics and reduced susceptibility to the formation of fines has led to the development of a family of SPE tubes packed entirely with spherical, non-friable particles. The Supel™ Sphere Carbon/NH<sub>2</sub> dual layer SPE tube contains both spherical carbon particles and spherical aminopropyl (NH<sub>2</sub>) modified silica. It was developed to offer superior flow characteristics when conducting cleanup for multi-residue pesticide analysis from food.

### Supel™ Sphere Carbon/NH<sub>2</sub> for Analysis of Pesticide Residues in Spinach

In a study comparing Supel™ Sphere Carbon/NH<sub>2</sub> with current products containing irregular materials, results illustrated that Supel™ Sphere Carbon/NH<sub>2</sub> removed as much color and background, and exhibited faster and more consistent flow than cartridges containing irregular materials, providing pesticide recovery similar to that of other dual layer SPE cartridges. Improved flow characteristics and GC/MS background is illustrated in Figures 17 and 19.

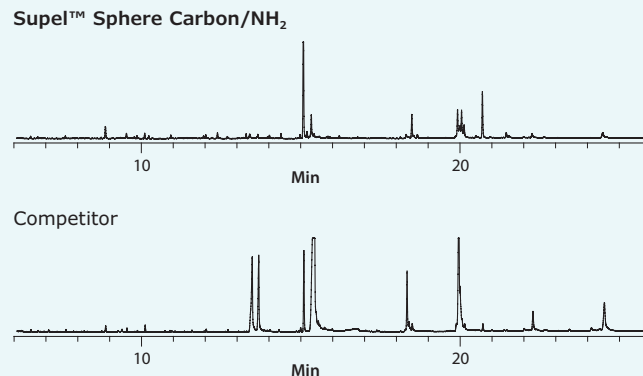


**Figure 17.** Flow Comparison Test  
Timed Gravity Elution of Solvent (25 mL) from Dual-Layer Cartridges.  
Average Flow n = 5.



**Figure 18.** Supel™ Sphere Cartridge

**Column:** SLB®-5 ms, 20 m x 0.18 mm I.D., 0.36 µm (28576-U)  
**Oven:** 70 °C (2 min), 15 °C/min to 325 °C (5 min)  
**Inj. temp.:** Programmed, 60 °C (0.28 min), 600 °C/min to 325 °C (5 min)  
**Carrier gas:** helium, 1 mL/min constant  
**Detector:** MS, SIM mode  
**Injection:** 10 µL LVI, PTV solvent vent, rapid injection speed; split vent flow: 100 mL/min (5 psi) until 0.28 min, 60 mL/min at 2.78 min  
**Liner:** 4 mm I.D., split/splitless type, single taper FocusLiner™ design (wool packed)



**Figure 19.** GC/MS Comparison of Cleaned Spinach Extracts

Description	Qty.	Cat. No.
Supel™ Sphere Carbon/NH <sub>2</sub> 500 mg/500 mg, 6 mL	30	54283-U

For more information, visit  
[SigmaAldrich.com/product/supelco/54283u](https://www.sigmaaldrich.com/product/supelco/54283u)

# Supel™ QuE (Dispersive SPE) for “QuEChERS” Method

## Quick and Simple Cleanup for Pesticide Residue Analysis

The “QuEChERS” method (Quick, Easy, Cheap, Effective, Rugged, and Safe), has emerged as a sample prep technique popular in the area of multi-residue pesticide analysis in food and agricultural products, and is formalized in the EN15662:2008 and AOAC 2007.01 Method.

The “QuEChERS” method (Quick, Easy, Cheap, Effective, Rugged, and Safe), has emerged as a sample prep technique popular e.g. in the area of matrix rich samples, like food product. The PFAS testing in food is formalized in FDA method C-010.02. In a first step, food sample is extracted with an aqueous miscible solvent (e.g., acetonitrile) in the presence of high amounts of extraction salts to induce liquid phase separation. Upon shaking and centrifugation, an aliquot of the organic phase is subjected to further cleanup step. The second step is facilitated by mixing bulk amounts of sorbent (e.g., Supelclean™ PSA, ENVI-Carb™, MgSO<sub>4</sub>) with the extract. After sample cleanup, the mixture is centrifuged and the resulting supernatant can, depending on the sample, either be analyzed directly or can be subjected to further minor treatment before analysis of vials and centrifuge tubes contains pre-determined amounts of salts and SPE sorbents to support the most common method configurations used today for QuEChERS.

For more information, visit [SigmaAldrich.com/quechers](http://SigmaAldrich.com/quechers)



## Features and Benefits

- Efficient and economic sample cleanup
- Pre-weighed amounts of sorbents and salts save labor and time
- High purity reagents
- Convenient and reliable in ready-to-use 15 mL, 12 mL and 2 mL centrifuge tubes

## Supel™ QuE Z-Sep: Fat Removal in Difficult Matrices

The patent-pending zirconia-coated silica particles of Supel™ QuE Z-Sep sorbents selectively remove more fat and color from sample extracts than traditional phases for QuEChERS methods. Lipid retention is based on two synergistic interactions: the interaction between the polar group of the lipid and the proprietary bonded zirconia (Z-Sep) group of the sorbent as well as the interaction between the hydrophobic chains of the lipid and the hydrophobic group of the sorbent (either that of the C18 or Z-Sep+). Supel™ QuE Z-Sep/C18, a combination of Discovery® DSC-18 and Z-Sep particles, is recommended for samples containing <15% fat. Supel™ QuE Z-Sep+, a C18 & zirconia dual bonded silica, is recommended for cleanup of samples containing >15% fat. Supel™ QuE Z-Sep is recommended for the analysis of hydrophobic analytes in fatty matrices.

- Significantly diminishes fatty matrix interferences and various colors
- Provides more robust LC-MS and GC/MS methods by eliminating problematic matrix interferences
- Can replace C18 and PSA phases in current methods without additional method development

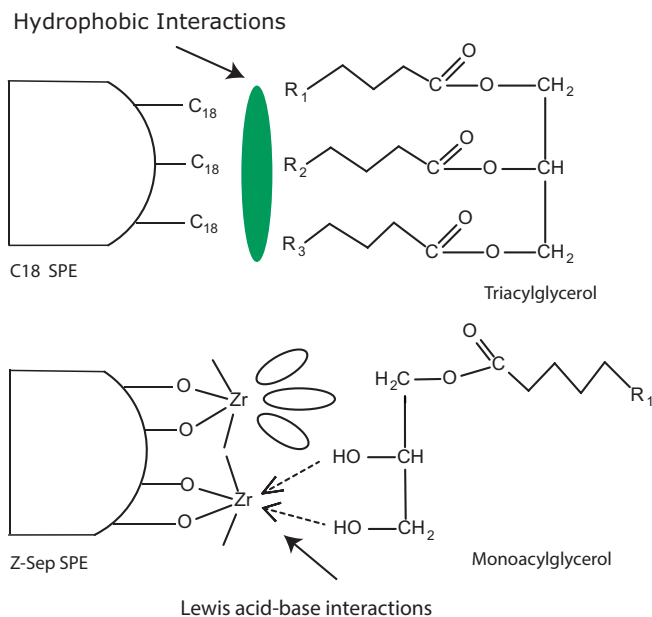


Figure 20. Interactions of Supel™ QuE Z-Sep and C18

## Analysis of Pesticides in Avocado using Z-Sep+ SPE Sorbent in QuEChERS Method for Sample Cleanup

In a recent experiment examining the cleanup of avocado extracts prior to pesticide residue analysis, the Z-Sep+ sorbent showed improved cleanup over PSA/C18, as illustrated in the bar chart below. The Z-Sep+ cleanup shows the lowest mass of remaining extractables after cleanup of 1.44 g of avocado. In addition, as shown in the graph below, Z-Sep+ showed improved analyte recovery over PSA/C18.

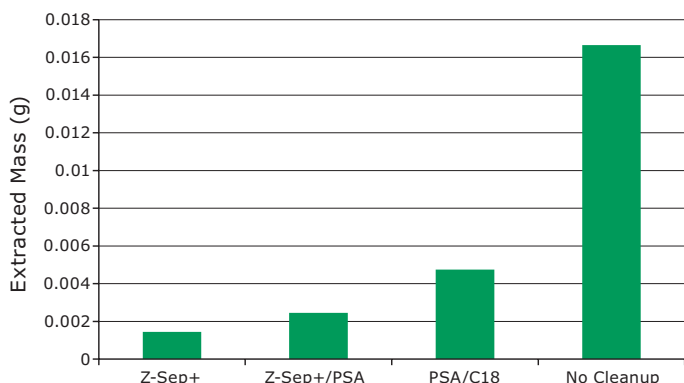


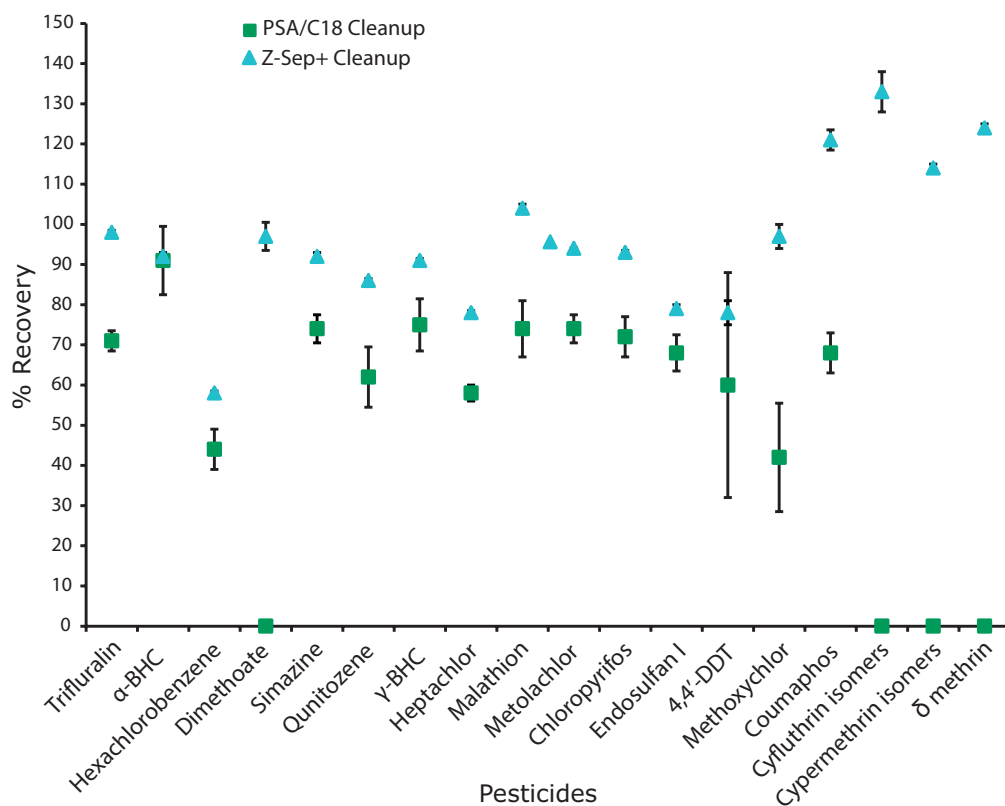
Figure 21. Total Extractables

## Supel™ QuE Verde: For Challenging Compounds in Green Matrices

Supel™ QuE Verde for QuEChERS combines a novel carbon with zirconia coated silica (Z-Sep+) to provide an optimum balance between analyte recovery and color removal. This sorbent combination has been shown to provide recoveries in the range of 70% to 120% of even the most challenging planar pesticides while maintaining >95% pigment removal in high chlorophyll matrices.

Supel™ QuE Verde is a mixture of an improved graphitized carbon black (GCB), Z-Sep+, and primary-secondary amine (PSA). The improved GCB has been optimized to balance chlorophyll removal and improve recoveries of planar pesticides. As mentioned, Z-Sep+ is a silica that is functionalized with both zirconia and C18. Zirconia will retain some fats and carotenoids, while C18 retains hydrophobic interferences. The PSA in the mix functions to remove acidic interferences. When used to clean samples containing chlorophyll, this sorbent blend will provide better recovery of planar pesticides than sorbents containing traditional GCB.

Learn more at [SigmaAldrich.com/verde](http://SigmaAldrich.com/verde)



- Z-Sep+ showed higher recovery overall.
- PSA/C18: matrix interference prevented analysis of cyfluthrin, cypermethrin and deltamethrin.
- Z-Sep+ showed better reproducibility than PSA/C18

Figure 22. Analyte Recovery of Selected Pesticides from Avocado

For more information, visit [SigmaAldrich.com/zsep](http://SigmaAldrich.com/zsep)

## Supel™ QuE Products for QuEChERS and Related Products

### Pre-Packed dSPE Tubes

Description	Qty.	Cat. No.
<b>EN15662:2008 (15 mL centrifuge tubes, shaker compatible)</b>		
Supel™ QuE PSA/C18 (EN) Tube, 15 mL 150 mg Supelclean™ PSA, 150 mg Discovery® DSC-18, 900 mg MgSO <sub>4</sub>	50	55439-U
Supel™ QuE PSA/ENVI-Carb™ (EN) Tube 1, 15 mL 150 mg Supelclean™ PSA, 15 mg Supelclean™ ENVI-Carb™, 900 mg MgSO <sub>4</sub>	50	55446-U
Supel™ QuE PSA/ENVI-Carb™ (EN) Tube 2, 15 mL 150 mg Supelclean™ PSA, 45 mg Supelclean™ ENVI-Carb™, 900 mg MgSO <sub>4</sub>	50	55464-U
<b>EN15662:2008 (12 mL centrifuge tubes)</b>		
Supel™ QuE Citrate (EN) Tube, 12 mL 4 g MgSO <sub>4</sub> , 1 g NaCl, 0.5 g NaCitrate dibasic sesquihydrate, 1 g NaCitrate tribasic dihydrate	50	55227-U
Supel™ QuE Citrate/Sodium Bicarbonate (EN) Tube, 12 mL 4 g MgSO <sub>4</sub> , 5 g NaBicarbonate, 1 g NaCl, 0.5 g NaCitrate dibasic sesquihydrate, 1 g NaCitrate tribasic dihydrate	50	55237-U
Supel™ QuE PSA (EN) Tube, 12 mL 150 mg Supelclean™ PSA, 900 mg MgSO <sub>4</sub>	50	55228-U
Supel™ QuE PSA/C18 (EN) Tube, 12 mL 150 mg Supelclean™ PSA, 150 mg Discovery® DSC-18, 900 mg MgSO <sub>4</sub>	50	55229-U
Supel™ QuE PSA/ENVI-Carb™ (EN) Tube 1, 12 mL 150 mg Supelclean™ PSA, 15 mg Supelclean™ ENVI-Carb™, 900 mg MgSO <sub>4</sub>	50	55230-U
Supel™ QuE PSA/ENVI-Carb™ (EN) Tube 2, 12 mL 150 mg Supelclean™ PSA, 45 mg Supelclean™ ENVI-Carb™, 900 mg MgSO <sub>4</sub>	50	55233-U
<b>EN15662:2008 (2 mL centrifuge tubes)</b>		
Supel™ QuE PSA (EN) Tube, 2 mL 25 mg Supelclean™ PSA, 150 mg MgSO <sub>4</sub>	100	55172-U
Supel™ QuE PSA/C18 (EN) Tube, 2 mL 25 mg Supelclean™ PSA, 25 mg Discovery® DSC-18, 150 mg MgSO <sub>4</sub>	100	55173-U
Supel™ QuE PSA/ENVI-Carb™ (EN) Tube 1, 2 mL 25 mg Supelclean™ PSA, 2.5 mg Supelclean™ ENVI-Carb™, 150 mg MgSO <sub>4</sub>	100	55174-U
Supel™ QuE PSA/ENVI-Carb™ (EN) Tube 2, 2 mL 25 mg Supelclean™ PSA, 7.5 mg Supelclean™ ENVI-Carb™, 150 mg MgSO <sub>4</sub>	100	55176-U
<b>AOAC 2007.01 (15 mL centrifuge tubes, shaker compatible)</b>		
Supel™ QuE PSA (AC) Tube, 15 mL 400 mg Supelclean™ PSA, 1200 mg MgSO <sub>4</sub>	50	55466-U
Supel™ QuE PSA/C18 (AC) Tube, 15 mL 400 mg Supelclean™ PSA, 400 mg Discovery® DSC-18, 1200 mg MgSO <sub>4</sub>	50	55470-U
Supel™ QuE PSA/C18/ENVI-Carb™ (AC) Tube 1, 15 mL 400 mg Supelclean™ PSA, 400 mg Discovery® DSC-18, 400 mg Supelclean™ ENVI-Carb™, 1200 mg MgSO <sub>4</sub>	50	55474-U

Description	Qty.	Cat. No.
<b>AOAC 2007.01 (12 mL centrifuge tubes)</b>		
Supel™ QuE Acetate (AC) Tube, 12 mL 6 g MgSO <sub>4</sub> , 1.5 g NaAcetate	50	55234-U
<b>AOAC 2007.01 (2 mL centrifuge tubes)</b>		
Supel™ QuE PSA (AC) Tube, 2 mL 50 mg Supelclean™ PSA, 150 mg MgSO <sub>4</sub>	100	55287-U
Supel™ QuE PSA/C18 (AC) Tube, 2 mL 50 mg Supelclean™ PSA, 150 mg MgSO <sub>4</sub> 50 mg Discovery® DSC-18	100	55288-U
Supel™ QuE PSA/C18/ENVI-Carb™ (AC) Tube, 2 mL 50 mg Supelclean™ PSA, 150 mg MgSO <sub>4</sub> 50 mg Discovery® DSC-18, 50 mg ENVI-Carb™	100	55289-U
Supel™ QuE PSA/ENVI-Carb™ (AC) Tube 50 mg Supelclean™ PSA, 150 mg MgSO <sub>4</sub> 50 mg ENVI-Carb™	100	Custom

<b>Specialty Products for Challenging (Fatty/Lipid containing) Matrices (2 mL centrifuge tubes)</b>		
Supel™ QuE Z-Sep Tube, 2 mL 75 mg Z-Sep	100	55411-U
Supel™ QuE Z-Sep/C18 Tube, 2 mL 20 mg Z-Sep, 50 mg Discovery® DSC-18	100	55284-U
Supel™ QuE Z-Sep+ Tube, 2 mL 75 mg Z-Sep+	100	55408-U
Supel™ QuE Verde Tube, 2 mL 60 mg Z-Sep+, 50 mg Supelclean™ PSA, 10 mg Supelclean™ ENVI-Carb™ Y, 150 mg MgSO <sub>4</sub>	100	55447-U

<b>Specialty Products for Challenging (Fatty/Lipid containing) Matrices (15 mL centrifuge tubes, shaker compatible)</b>		
Supel™ QuE Z-Sep Tube, 15 mL 500 mg Z-Sep	50	55491-U
Supel™ QuE Z-Sep/C18 Tube, 15 mL 120 mg Z-Sep, 300 mg Discovery® DSC-18	50	55506-U
Supel™ QuE Z-Sep+ Tube, 15 mL 500 mg Z-Sep+	50	55486-U
Supel™ QuE Z-Sep+/MgSO <sub>4</sub> Tube, 15 mL 300 mg Z-Sep+, 900 mg MgSO <sub>4</sub>	50	55511-U
Supel™ QuE Verde Tube, 15 mL 480 mg Z-Sep+, 400 mg Supelclean™ PSA, 80 mg Supelclean™ ENVI-Carb™ Y, 1200 mg MgSO <sub>4</sub>	50	55442-U

<b>Non-buffered extraction tubes (12 mL centrifuge tubes)</b>		
Supel™ QuE Non-Buffered Tube 1, 12 mL 4 g MgSO <sub>4</sub> , 1 g NaCl	50	55294-U
Supel™ QuE Non-Buffered Tube 2, 12 mL 6 g MgSO <sub>4</sub> , 1.5 g NaCl	50	55295-U

<b>Specialty Extraction Salts</b>		
Supel™ QuE Ammonium Sulfate Tube, 12 mL 4 g Ammonium Sulfate	1,000	54276-U

Description	Comment	Cat. No.
<b>FDA C_0.10.02</b>		
Supel™ QuE, Non-Buffered tube 2, pk of 50	Extraction salts	55295-U
BRAND® PP graduated centrifuge tube, screw cap	Empty tube	BR114820
pk of 50 Supel™ QuE PSA/ ENVI-Carb™ Tube 3, 15 mL	Clean-up tube with sorbent, 900 mg MgSO <sub>4</sub> , 300mg PSA, 150 mg ENVI-Carb	55479-U



## Bulk Adsorbents and Salts

Description	Qty.	Cat. No.
Supelclean™ PSA, bulk sorbent	100 g	<b>52738-U</b>
Supelclean™ ENVI-Carb™, bulk sorbent	50 g	<b>57210-U</b>
Discovery® DSC18, bulk sorbent	100 g	<b>52600-U</b>
Z-Sep+	20 g	<b>55299-U</b>
Z-Sep	20 g	<b>55418-U</b>
MgSO <sub>4</sub> (as cited in EN15662:2008)	var.	<b>208094</b>
Sodium citrate dibasic sesquihydrate	var.	<b>71635</b>
Sodium citrate tribasic dihydrate	var.	<b>54641</b>
Sodium chloride	var.	<b>57653</b>
Sodium acetate	var.	<b>241245</b>

QuEChERS tubes, salts, and clean-up tubes, Conform to method FDAC\_010.02

Description	Comment	Cat. No.
<b>FDA C_0.10.02</b>		
Supel™ QuE, Non-Buffered tube 2, pk of 50	Extraction salts	<b>55295-U</b>
BRAND® PP graduated centrifuge tube, screw cap	Empty tube	<b>BR114820</b>
pk of 50 Supel™ QuE PSA/ENVI-Carb™ Tube 3, 15 mL	Clean-up tube with sorbent, 900 mg MgSO <sub>4</sub> , 300mg PSA, 150 mg ENVI-Carb	<b>55479-U</b>

## QuEChERS Shakers and Accessories

Description	Qty	Cat. No.
<b>Benchmark Benchmixer™ XL Laboratory Shakers</b>		
QuEChERS Shaker and Rack Starter Kit, USA compatible plug, AC input 115 V	—	<b>55278-U</b>
QuEChERS Shaker and Rack Starter Kit, EU compatible Schuko plug, AC input 230 V	—	<b>55438-U</b>
Multi-tube Vortexer, USA compatible plug, AC input 115 V	—	<b>Z765503</b>
<b>Benchmark Benchmixer™ XL Laboratory Shaker Racks</b>		
50 mL QuEChERS Extraction Tube Shaker Rack	1	<b>55279-U</b>
15 mL QuEChERS Cleanup Tube Shaker Rack	1	<b>Z765589</b>
2 mL QuEChERS Cleanup Tube Shaker Rack	1	<b>Z765554</b>

To receive a sample of our specialty products, visit [SigmaAldrich.com/SPESample](http://SigmaAldrich.com/SPESample)

# Analytes in Edible Oils

## Supelclean™ EZ-POP NP SPE Cartridges

### Features and Benefits

- Provides simultaneous extraction of a full range of polycyclic aromatic hydrocarbons (PAHs), while removing both fatty matrix and polar interferences from oil matrices
- Produces cleaner extracts and gives better overall PAH recoveries than other SPE methods
- Easier and more versatile methodology than other SPE methods, requiring fewer steps and little to no method development
- Final extracts are GC and HPLC compatible
- Yields clean extracts which can be analyzed using any MS detector

### Simple, Effective Extraction of Lipophilic Persistent Organic Pollutants (POPs) from Oily Samples

This dual-layer SPE cartridge offers superior cleanup for the extraction of non-polar POPs, specifically heavy and light PAHs, from edible oil matrices. The top Florisil® layer retains polar functional groups such as acids and alcohols. The bottom Z-Sep/C18 layer binds fatty matrix through hydrophobic interaction as well as Lewis acid-base interactions. Fatty matrix is preferentially retained by the cartridge while non-polar POPs, are washed through using acetonitrile. The resulting extract is suitable for either GC/MS or HPLC analysis.

### Application: The Analysis of PAHs in Olive Oil

The Supelclean™ EZ-POP NP was compared to two competitor silica gel SPE cartridges in terms of matrix removal and analyte recovery for the extraction of select PAHs from olive oil. The EZ-POP NP removed

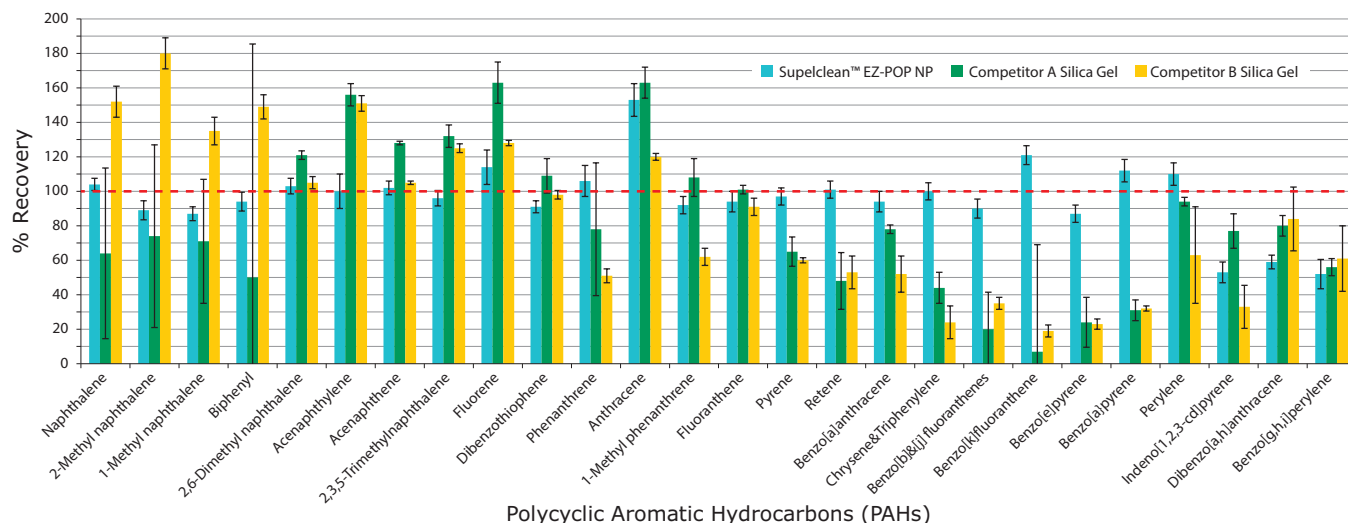
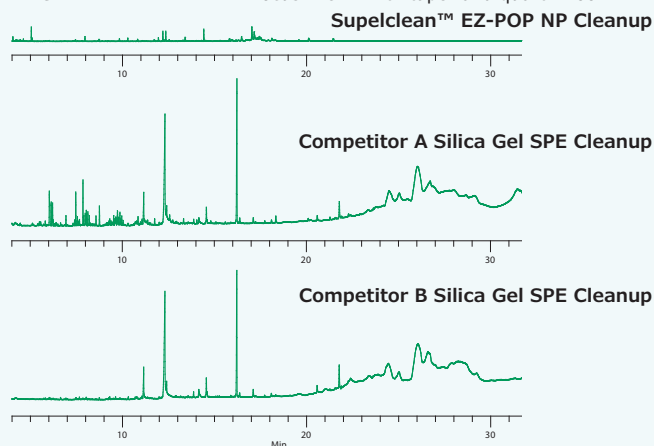


Figure 24. Analyte Recovery of PAHs from Olive Oil Extract (n=3)

Figure 23. GC/MS Full Scan Chromatograms of Olive Oil Extract (same y axis)

**Column:** SLB®-5ms, 20 m x 0.18 mm I.D., 0.18 µm (28564-U)  
**Oven:** 60 °C (1 min.), 15 °C/min. to 250 °C, 8 °C/min. to 330 °C (7 min.)  
**Inj. temp.:** 300 °C  
**Carrier gas:** helium, 1 mL/min constant flow  
**Detector:** MS  
**MSD interface:** 330 °C  
**Injection:** 1 µL, pulsed splitless (50 psi until 0.75 min, splitter open at 0.75 min.)  
**Liner:** 4 mm ID FocusLiner™ with taper and quartz wool



more unwanted background than silica gel SPE, greatly decreasing the matrix effects (Figure 23). It produced better, more accurate, analyte recoveries than the silica gel SPE with good reproducibility (Figure 24). Thus, the Supelclean™ EZ-POP NP provides suitable matrix removal for rugged GC/MS analysis of PAHs in olive oil.

Description	Qty.	Cat. No.
Supelclean™ EZ-POP NP, 2.5 g/1 mL	20	54341-U

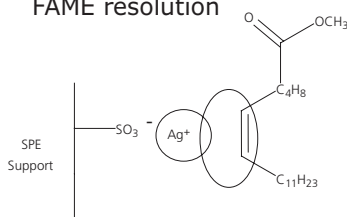
# Miscellaneous Specialty Products and SPE Accessories

## Discovery® Ag-Ion SPE Tubes for *cis/trans* FAME Analysis

**Retention Mechanism:** Normal-phase

**Sample Matrix Compatibility:** Organic solvents, oils, and lipids

- Developed for the fractionation of FAMEs based on degree of unsaturation and for the resolution of *cis/trans* isomers.
- Silver counter-ions are anchored onto a SCX support using a proprietary procedure to offer optimal resolution, performance and capacity.
- Each lot is tested and quality controlled for *cis/trans* FAME resolution



Charge-transfer complex between Ag<sup>+</sup> and unsaturated bond

Description	Qty.	Cat. No.
750 mg/6 mL	30	54225-U

## Glass SPE Tubes with PTFE Frits

A select line of our Supelclean™ SPE phase chemistries is also available in inert glass and PTFE hardware configurations.

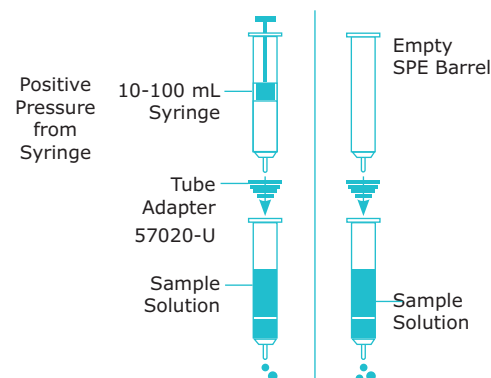


- Resistant to harsh chemicals and aggressive solvents
- Absence of leachables such as phthalates and plasticizers
- Hygroscopic adsorbents (e.g. Florisil®) can be easily heat treated/activated (e.g., 105-120 °C oven, overnight) prior to use.

Description	Qty.	Cat. No.
<b>Supelclean™ ENVI™-18 SPE Tube</b> bed wt. 500 mg, vol. 6 mL	30	54331-U
<b>Supelclean™ LC-Florisil® SPE Tube</b> bed wt. 500 mg, vol. 6 mL	30	54333-U
<b>Supelclean™ LC-Florisil® SPE Tube</b> bed wt. 1 g, vol. 6 mL	30	54334-U
<b>Supelclean™ LC-Si SPE Tube</b> bed wt. 1 g, vol. 6 mL	30	54335-U
<b>Dual Layer Florisil®/Na<sub>2</sub>SO<sub>4</sub> SPE Tube</b> bed A: 2 g (Na <sub>2</sub> SO <sub>4</sub> ), bed B: 2 g (Florisil®), vol. 6 mL	48	52582-U

## Accessories

### Tube Adapters



Tube adapters serve many functions:

- Stack one SPE tube on top of another to provide different selectivities
- A larger empty syringe barrel can be stacked on top of a smaller SPE tube to act as a larger load reservoir
- Adapter for positive pressure methods (e.g. from a syringe or air/N<sub>2</sub> line)

Description	Qty.	Cat. No.
<b>SPE Tube Adapters for Polypropylene Tubes</b>		
For 1, 3, 6 mL Tubes	12	57020-U
For 12, 20, 60 mL Tubes	6	57267
<b>AutoTrace® SPE Tube Adapters*</b>		
For 3 mL Tubes	6	57123
For 6 mL Tubes	6	57126
* Allows SPE tubes to be used with AutoTrace® Automated Systems		
<b>SPE Tube Adapter for Glass Tubes</b>		
PTFE, for use with 6 mL glass SPE Tube	24	504335

## Large Volume SPE Reservoirs

Large volume SPE reservoirs are designed to increase the headspace volume of standard polypropylene SPE tubes. Because these reservoirs are designed to connect directly to the mouth of the SPE tube, they are ideal for gravity applications where increased headspace volume is required.



The reservoirs are designed for use with 6 mL polypropylene SPE tubes and add an additional headspace volume of 25 mL.

Description	Qty.	Cat. No.
<b>Large Volume SPE Reservoir</b>		
Polypropylene	30	54258-U
PTFE	3	54259-U

# SPE Accessories

## Empty SPE Hardware and Components



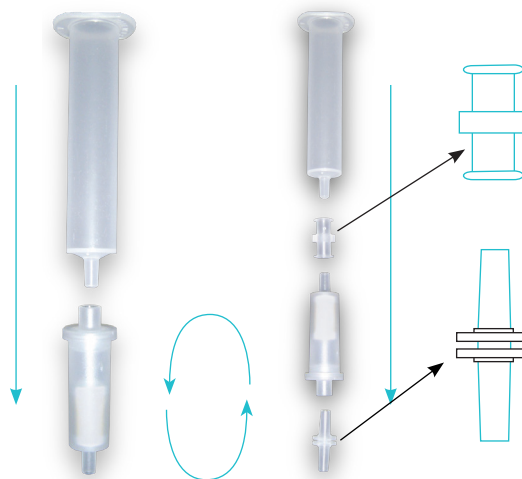
SPE Tube Components

Description		1 mL	3 mL	6 mL	12 mL	20 mL	60 mL
<b>Empty SPE Tubes with and without Frits</b>	<b>Qty.</b>	<b>108</b>	<b>54</b>	<b>30</b>	<b>20</b>	<b>20</b>	<b>16</b>
Empty PP SPE Tube with PE Frits, 20 µm porosity		57023	57024	57026	57176	57177	57178
Empty PP SPE Tube with PE Frits, 20 µm porosity – pre-fritted with bottom frit		54220-U (pk 100)	54221-U (pk 100)	54222-U (pk 100)	54223-U (pk 100)	57118-U	57119-U
Empty PP SPE Tube (no frits)		57240-U	57241	57242	57179 (pk 12)	57021 (pk 12)	57022
<b>Frits for use with SPE tubes</b>	<b>Qty.</b>	<b>216</b>	<b>108</b>	<b>60</b>	<b>40</b>	<b>40</b>	<b>32</b>
PE Frits for PP SPE tubes, 20 µm porosity		57244	57180-U	57181	57182-U	57183	57184

PP = Polypropylene; PTFE = Polytetrafluoroethylene; SS = Stainless steel; PE = Polyethylene \* Qty. of 24

## Miscellaneous SPE Hardware and Accessories

Description	Qty.	Cat. No.
<b>Empty Reversible SPE Tube, non-fluorous PP, w/PE frits</b>		
0.5 mL	50	57602-U
1.0 mL	50	57607-U
2.0 mL	50	57608-U
<b>Empty PP Rezorian Tube Kit w/PE Frits, luer plugs and caps</b>		
1.0 mL	50	57609-U
<b>Empty 96-well SPE Plates</b>		
2 mL deep square well, w/PE frits	1	Inquire
1.25 mL round well, w/PE frits	1	Inquire
<b>Luer Caps, Plugs, and Couplers</b>		
Female Luer Cap, PP (caps SPE luer tips)	12	57098
Male Luer Plug, PP (plugs female luer fitting)	12	504351
Female Luer Coupler	20	21015
Male Luer Coupler	20	25064-U



## Visiprep™ and Visiprep™ DL SPE Vacuum Manifolds

Visiprep™ SPE Vacuum Manifolds allow you to process up to 12 or up to 24 SPE tubes simultaneously. Both DL (disposable liner) and standard models are available.



12-Port Visiprep™ DL Vacuum Manifold (57044)

The Visiprep™ DL Vacuum Manifold eliminates the possibility of cross contamination when processing a new sample on the same port by employing a disposable liner that builds the complete flow path through the valve. The liner consists of a PP luer hub that attaches to the SPE tube, and a thin walled PTFE tubing that is threaded through the SPE port. This ensures that

all SPE port/valve surfaces coming in contact with the sample can be easily & conveniently replaced following each extraction.

### Features and Benefits DL and Standard Models

- Screw-type valves for each SPE port for precise flow control by just turning the attached SPE tube
- Glass basin will not dissolve, fog or discolor when exposed to solvents
- Legs on stand-alone cover allows user to easily rest cover on work surface when removed from vacuum manifold
- Screw type solvent resistant vacuum bleed gauge and valve offer better sealing
- PP collection vessel rack accommodates autosampler vials, small scintillation vials, 10 and 16 mm test tubes and 1, 2, 5, and 10 mL volumetric flasks. An optional plate for 20 mL scintillation vials is available for 24-port models.

Description	Cat. No.
<b>Visiprep™ DL Solid Phase Extraction Manifold</b>	
12-Port Model	57044
24-Port Model	57265
Disposable valve liners, PTFE, pk. of 100	57059
<b>PTFE-free Solid Phase Extraction Manifold (Coming Soon)</b>	
12-Port Model	57030-U
24-Port Model	57250-U



24-Port Visiprep™ Vacuum Manifold (57250-U)

## Visiprep™ 5-Port Flask Manifold

The Visiprep™ 5-Port Flask Vacuum Manifold enables analysts using solid phase extraction tubes to simultaneously prepare up to 5 samples.



Unlike conventional vacuum manifolds, the Visiprep™ 5-Port Flask Manifold allows users to collect their SPE eluate directly into 50 mL round or flat bottom flasks for direct rotovap evaporation. The manifold consists of a chemical resistant 5-port cover (DL or standard available), gasket, base, a glass basin, vacuum gauge and bleed valve, 5 flow control valves, 5 replaceable solvent guide needles and a base plate that supports up to five 50 mL round or flat bottom flasks. Each port on both the standard and DL Visiprep™ models are equipped with flow control valves.

Recommended Flasks: Aldrich® single-neck flask, 50 mL, joint: ST/NS 24/40

- Round Bottom (Cat. No. **Z414484**)
- Flat Bottom (Cat. No. **Z418773**)

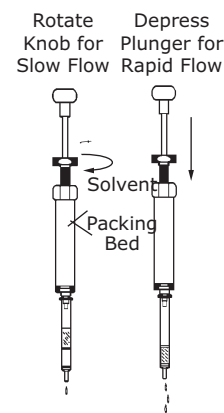
Description	Cat. No.
<b>Visiprep™ 5-Port Flask Vacuum Manifold</b>	
DL (Disposable Liner)	57101-U
Standard	57103-U

## Visi™-1 Single SPE Tube Processor

### Visi™-1 processor - two rates of flow control

Our Visi™-1 Single SPE Tube Processor provides precise flow control through a single 1 mL, 3 mL or 6 mL SPE tube. There is no faster, more convenient, or more reliable method for processing one or a few samples.

Simply fill the SPE tube with the appropriate solution and attach it to the Visi™-1 processor. Remove the tube from the processor, introduce the next solution and repeat the process.



Description	Cat. No.
Visi™-1 Single SPE Tube Processor	57080-U

## Preppy™ Vacuum Manifold

Simultaneously prepare up to 12 samples with our simplest and most economical manifold. The Preppy™ consists of a chemical-resistant cover and gasket, glass basin, vacuum release vent and 12 individual control valves with knurled tops and stainless steel solvent guide needles.

Two optional collection racks are available – one for 2 and 4 mL autosampler vials and the other for 15 (w/21 mm O.D.) or 40 (w/28 mm O.D.) mL vials. An optional vacuum gauge/bleed valve assembly can be installed to allow precise control of the vacuum.

Description	Cat. No.
<b>Preppy™ Vacuum Manifold</b>	
12-Port Model	57160-U
<b>Collection Vessel Racks</b>	
For 2 or 4 mL vials	57159-U
For 15 or 40 mL vials	57162-U
<b>Accessories</b>	
Vacuum Gauge/Bleed Valve Assembly	57161-U

Chemical  
Resistant Cover

Glass Basin



## Visidry™ Drying Attachment

Designed for our Visiprep™ Vacuum Manifold, the Visidry™ Drying Attachment (57100-U) also fits our economical Preppy™ manifold. The Visidry™ unit installs in minutes, dries up to 12 or up to 24 SPE tubes at one time and can be used with any inert gas supply. It is also useful for evaporating and concentrating recovered samples. (Gas) flow through each Visiprep™ port can be still independently adjusted.



57030-U 12-Port Model  
Order Separately

Description	Qty.	Cat. No.
<b>Visidry™ Drying Attachment</b>		
12-Port Model	1	57100-U
24-Port Model	1	57124
<b>Replacement Parts for Visidry™ Drying Attachment</b>		
Control Knobs	2	57095
Retaining "C" Clips	2	57096
Female Luer Plugs	12	57098

Replacement SPE Tube Adapters (57020-U) listed on p. 42.

**Note:** The Visidry™ drying attachment cannot be used to dry 12 mL, 20 mL, or 60 mL SPE tubes.

## Visiprep™ Large Volume Samplers

Allows for easy "hands-off" transfer of large volumes of low viscosity liquid samples directly from any sample container to conventional SPE tubes (not suitable for glass tubes).

The samplers consist of 1/8" PTFE tubing with a stainless steel weight at one end and a screw-fitted SPE tube adapter on the other end. To use the sampler, the weighted end is placed in the sample container, and the tube adapter is inserted into a pre-conditioned SPE tube. Vacuum pressure delivered from the vacuum manifold is used to pull the sample through the PTFE tubing into the SPE tube where analytes of interest are concentrated on the SPE tubes prior to elution.



Description	Qty.	Cat. No.
<b>Visiprep™ Large Volume Sampler</b>		
for 12 mL, 20 mL, or 60 mL SPE Tubes (3 adapters)	1	57272
for 3 mL or 6 mL SPE Tubes (4 adapters)	1	57275
<b>Replacement Parts</b>		
1/8" PTFE Tubes, color-coded	4	57276
Nuts and Ferrules, color-coded	4	57277
Stainless Steel Weights	4	57278
<b>Tube Adapters, 1/4-28 threads</b>		
For 3 mL or 6 mL Tubes	4	57273-U

## Vacuum Manifold Replacement Parts and Accessories

Description	Qty.	Cat. No.
<b>For 12-Port Manifold</b>		
Cover, 12 flow control valves, gasket <sup>1</sup>	-	57031-U
Cover, 12 DL flow control valves, gasket <sup>2</sup>	-	57029
Gaskets	2	57033
Collection rack (base, 3 support rods, center plate, 10 mm test tube plate, 12 retaining clips) <sup>3</sup>	-	57037
Plate for 16 mm test tubes <sup>3</sup>	-	57039
<b>For 24-Port Manifold</b>		
Cover, 24 flow control valves, gasket <sup>4</sup>	-	57251
Cover, 24 DL flow control valves, gasket <sup>5</sup>	-	57266
Gaskets	2	57254
Collection rack (base, 2 support rods, center plate, 10 mm test tube plate, 8 retaining clips) <sup>6</sup>	-	57255
Plate for 16 mm test tubes <sup>6</sup>	-	57257
Plate for 2 mL autosampler vials <sup>6</sup>	-	57258
<b>For 12-Port or 24-Port Manifold</b>		
Valve Stem for Visiprep™ DL Vacuum Manifold	24	57146-U
Valve Stem for Visiprep™/Preppy™ Vacuum Manifold	24	57147-U
Flow control valves <sup>7</sup>	2	57032
Solvent guide needles, PTFE <sup>1,8</sup>	12	57047
Solvent guide needles, stainless steel <sup>7</sup>	12	57036
Disposable valve liners for DL versions, PTFE <sup>2,5</sup>	100	57059
Disposable liner flow control valves <sup>9</sup>	2	57028
Liner guide needles, stainless steel <sup>2,10</sup>	12	57027
Vacuum gauge and bleed valve		57035-U
Retaining clips for collection racks	12	57041
Test tubes, 10 x 75 mm <sup>1,2,8,10</sup>	12	57042

<sup>1</sup> Compatible with 57030-U

<sup>2</sup> Compatible with 57044

<sup>3</sup> Compatible with 57030-U and 57044

<sup>4</sup> Compatible with 57250-U

<sup>5</sup> Compatible with 57265

<sup>6</sup> Compatible with 57250-U and 57265

<sup>7</sup> Compatible with 57030-U and 57250-U

<sup>8</sup> 2 packages included with 57250-U

<sup>9</sup> Compatible with 57044 and 57265

<sup>10</sup> 2 packages included with 57265



## Trap Kit for SPE Vacuum Manifolds

When installed between a Visiprep™ SPE vacuum manifold and the vacuum source, a SPE Vacuum



Pump Trap collects all liquids that are aspirated through the SPE tubes, preventing contamination of the vacuum pump.

The easily assembled kit contains a polypropylene filtering flask, a one-hole rubber stopper, 4" (10 cm) of polypropylene tubing and 5' (1.5 m) of red rubber vacuum hose.

Description	Cat. No.
SPE Vacuum Pump Trap Kit	57120-U

## Vacuum Gauge / Bleed Valve Assembly

Install in-line for control of vacuum.



Description	Cat. No.
Vacuum Gauge / Bleed Valve Assembly	57161-U

## Long Stem Flow Control Valves for Visiprep™ Manifolds

Equip alternate valves in your standard 12-port or 24-port Visiprep™ vacuum manifold with these long stem flow control valves if you intend to use all ports of the manifold with 12 mL, 20 mL or 60 mL tubes.

Not for use with DL manifolds.



Description	Qty.	Cat. No.
Long Stem Flow Control Valves	6	57048

## 96-Well Vacuum Manifolds

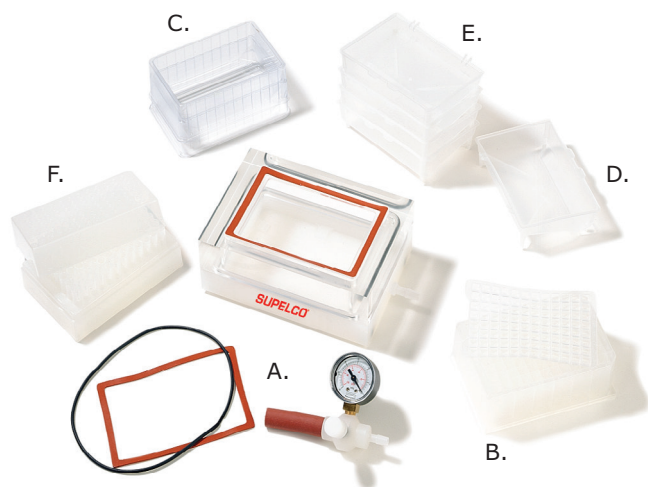
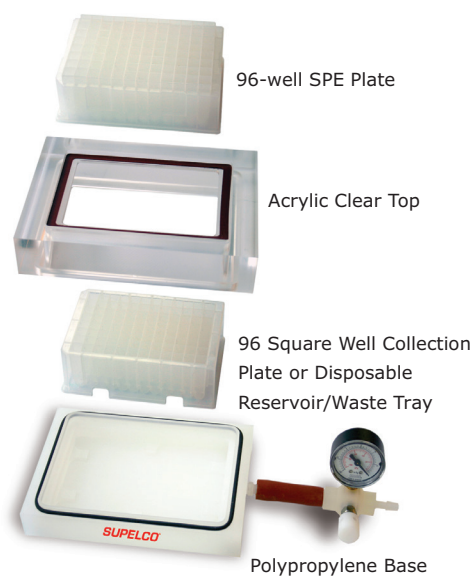
### PlatePrep Vacuum Manifold

The PlatePrep vacuum manifold consists of a clear acrylic top allowing for easier inspection of flow rates during SPE 96-well plate processing. The polypropylene base offers excellent chemical resistance while a single remote vacuum gauge/bleed valve controls flow through all the wells.

Use this compact vacuum manifold in conjunction with any of our 96-well plate offerings to process up to 96 samples concurrently. The single valve control, parallel processing capabilities and uniform flow dynamics allow for easier method development, reduce clutter and allow for greater reproducibility. Unused wells can be covered and used at a later date.

#### Starter Kit (575650-U) Includes:

- A. 1 PlatePrep Vacuum Manifold (57192-U)
- B. 1 96 Sq. Well Collection Plate, 2 mL, PP (575653-U)
- C. 2 Disposable Reservoir/Waste Trays, PVC (575654-U)
- D. 1 96 Sq. Well Pierceable Cap Mat (575655-U)
- E. 5 Reagent Reservoirs (R9259-100EA)
- F. 1 Cluster Tube Rack (CLS4410-960EA)



Description	Qty.	Cat. No.
PlatePrep Vacuum Manifold	1	57192-U
96-Well Plate Starter Kit with PlatePrep Manifold	1	575650-U
<b>PlatePrep Vacuum Manifold Replacement Parts</b>		
Gasket/Connector Replacement Kit	1	57195-U
Remote Vacuum Gauge/Bleed Valve Assembly	1	57161-U
<b>96-Well SPE Accessory Items</b>		
96 Sq. Well Collection Plates, 2 mL, PP	50	575653-U
Disposable Reservoir/Waste Tray, PVC	25	575654-U
96 Sq. Well Pierceable Cap Mats	50	575655-U
Reagent Reservoirs	100	R9259-100EA
Cluster Tube Rack	1	CLS4410-960EA



## ENVI-Disk™ Accessories

### ENVI-Disk™ Holder

Use the ENVI-Disk™ Holder with 47 mm ENVI™-DSK SPE disks (for information on ENVI™-8 and ENVI™-18 DSK SPE disks, see page 19). The unique design of the holder allows each disk to be installed and held firmly in place without wrinkling or tearing. A screw clamp provides uniform pressure on the disk and the sealing surfaces to prevent troublesome leaks – spring-loaded clamps cannot offer the sealing integrity of the ENVI-Disk™ Holder.



The unit consists of a 1-liter sample funnel, a threaded screw clamp, a PTFE disk support and a PTFE filter base/ adapter with a vacuum attachment fitting. Use 25 x 250 mm test tubes to collect disk eluates. The flask and collection tubes are not included with the holder, but can be purchased separately.

Description	Cat. No.
ENVI-Disk™ Holder	57173
Flask, 1-liter, 40/35 fitting <sup>1</sup>	Z290610-1EA
Collection Tube, 25 x 250 mm <sup>1</sup>	57175

<sup>1</sup> Order separately – not included with holder

### ENVI-Disk™ Holder Manifold

The ENVI-Disk™ Holder Manifold holds one to six ENVI-Disk™ Holders with flasks, allowing you to simultaneously extract up to six 1-liter samples. Each of the six stations is controlled through an independent flow control valve. These valves are designed to vent the flask to the atmosphere when moved from the open to the closed position. The flow rate is controlled by the needle valve on the manifold.



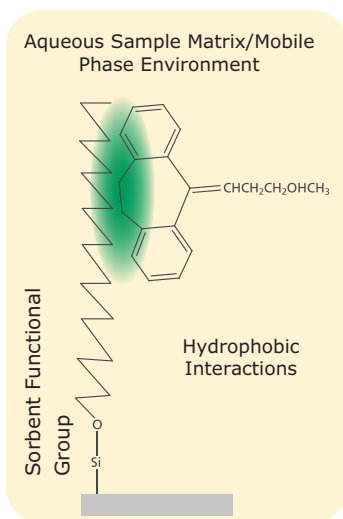
The unit includes a sturdy polymer base with six stations, six flow control valves, a needle valve, a vacuum gauge and vacuum tubing. A 1-liter glass bottle in the manifold acts as a trap to protect the vacuum source in the event of an overflow from one of the sample flasks.

Description	Cat. No.
ENVI-Disk™ Holder Manifold	57174

# SPE Methodology and Useful Tips

## Reversed-Phase SPE

Reversed-phase SPE is considered the least selective retention mechanism when compared to normal-phase or ion-exchange SPE. In other words, it may be difficult for a reversed-phase method or the bonded-chemistry to differentiate between molecules that are structurally similar. However, because reversed-phase will retain most molecules with any hydrophobic character, it is very useful for extracting analytes that are very diverse in structure within the same sample.



### Retention Mechanism: Non-polar or hydrophobic interactions

- Van der Waals or dispersion forces

**Sample Matrix:** Aqueous samples

- Biological fluids (serum, plasma, urine)
- Aqueous extracts of tissues
- Environmental water samples
- Wine, beer and other aqueous food & beverage samples

**Analyte Characteristics:** Analytes exhibiting non-polar functionalities

- Most organic analytes
- Alkyl, aromatic, alicyclic functional groups

**Elution Scheme:** Disrupt reversed-phase interaction with solvent or solvent mixtures of adequate non-polar character

- Methanol, acetonitrile, dichloromethane
- Buffer/solvent mixtures

### Common Applications

- Drugs and metabolites in biological fluids
- Environmental pollutants in water
- Pesticide and other contaminants in aqueous extracts from tissue & solids

## Basic Steps

- 1. Sample Pre-treatment** – For interference laden samples (e.g., biological fluids), dilute samples 1:1 with buffer. pH manipulation may be important when dealing with ionizable compounds. A compound's ionization state can drastically change its retention and elution characteristics on a given SPE sorbent.

When an analyte is in its neutral form, it becomes more hydrophobic and retention is strengthened under reversed-phase conditions. Adjusting the sample pH to 2 pH units above or below the compound's  $pK_a$  (depending on the functional group) will effectively neutralize or ionize the compound. When dealing with tissues and other solids, conduct a solid-liquid extraction or homogenization using a buffer. Solvents of non-polar character (including methanol and isopropanol) disrupt interaction between the compound and sorbent functional groups.

To avoid clogging, it may be necessary to centrifuge, dilute and/or pre-filter the sample prior to introducing it to the SPE phase.

- 2. Conditioning/Equilibration** – Conditioning wets or activates the bonded phases to ensure consistent interaction between the analyte and the sorbent functional groups. Reversed-phase sorbents are often conditioned with 1-2 tube volumes of a water miscible solvent such as methanol or acetonitrile.

Equilibration introduces a solution similar to the sample matrix in terms of solvent strength and pH in order to maximize retention. 1-2 tube volumes of buffer (used in sample pre-treatment) or water are good choices for reversed-phase equilibration.

- 3. Sample Load** – Apply sample (from step 1) at a consistent and reduced flow rate of  $\sim 1$ -2 drops/second to ensure optimal interaction time & retention.
- 4. Wash** – Sample interferences are often co-retained with compounds of interest during sample load. A wash step is necessary to elute interferences without prematurely eluting compounds of interest. 5-20% methanol in water or sample pre-treatment buffer are typical for wash solvents.
- 5. Elution** – Disrupt hydrophobic interactions between the analyte and sorbent functional groups with an organic solvent or solvent combination of sufficient non-polar character. Example elution solvents are 1-2 volumes of methanol or acetonitrile.

pH manipulation during elution can often improve recovery when dealing with ionizable compounds. In their ionic form, basic and acidic compounds become more polar, weakening reversed-phase interaction, possibly allowing for weaker elution solvents and/or reduced elution volumes.

**6. Eluate** – Post-treatment is often necessary to evaporate and reconstitute the SPE eluate in mobile phase prior to LC analysis. GC analysis often requires further SPE eluate concentration and/or possible matrix exchange with a more volatile solvent.

## SPE Tips

1. Drug-protein binding should be disrupted during sample pre-treatment. Strategies include:
  - 40  $\mu\text{L}$  of 2% disodium EDTA per 100  $\mu\text{L}$  mouse plasma
  - 40  $\mu\text{L}$  of 2% formic acid per 100  $\mu\text{L}$  mouse plasma
  - Other possible reagents (per 100  $\mu\text{L}$  matrix): 40  $\mu\text{L}$  of 2% TCA, 40  $\mu\text{L}$  of 2% acetic acid, 40  $\mu\text{L}$  of 2% TFA, 40  $\mu\text{L}$  of 2% phosphoric acid, or 200  $\mu\text{L}$  MeCN (protein ppt.).
  - If the SPE eluate needs to be evaporated prior to analysis, pass vacuum air through the SPE tube for  $\sim 10$  minutes prior to elution. This will remove residual moisture that may prolong evaporation.
2. Consistent and slow flow rate (1-2 drops per second) during sample load and elution will improve recovery and reproducibility.
3. Reduce bed weight to minimize elution volume.
4. Increase bed weight to retain more polar compounds
5. Concern for sorbent overdrying is only critical during methanol conditioning.
6. A pre-conditioning solvent such as dichloromethane (or solvent used for elution) can be used before conditioning to remove any impurities on the SPE tube that can interfere with subsequent analysis.

## Ion-Exchange and Mixed-Mode SPE

**Retention Mechanism: Electrostatic attraction of charged functional groups of the analyte(s) to oppositely charged functional groups on the sorbent. Combination of reversed-phase and ion-exchange for mixed-mode**

**Sample Matrix:** Aqueous or organic samples of low salt concentration ( $< 0.1$  M)

- Biological fluids
- Solution phase synthesis reactions

### Analyte Characteristics:

- Use cation-exchange for isolating basic compounds: primary, secondary, tertiary and quarternary amines
- Use anion-exchange for isolating acidic compounds: carboxylic acids, sulphonic acids and phosphates

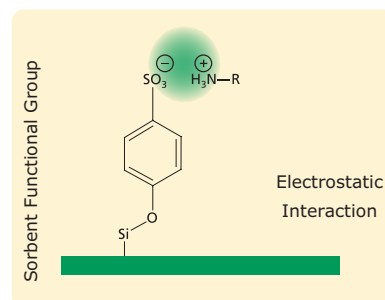
**Elution Scheme:** Electrostatic interactions disrupted via:

- pH modification to neutralize compound and/or sorbent functional groups
- Increase salt concentration ( $> 1$  M); or use a more selective counter-ion to compete for ion-exchange binding sites

## Common Applications:

- Drugs of abuse and pharmaceutical compounds in biological fluids
- Fatty acids removal in food/agricultural samples
- Cleanup of synthetic reactions
- Organic acids from urine
- Herbicides in soil

In order for electrostatic retention to occur, both analyte and sorbent functional groups must be in their ionized form. This is done through strict pH control of the sample matrix. For basic analytes, the pH should be adjusted to at least 2 pH units below the molecule's  $\text{pK}_a$ . For acidic analytes, the pH should be adjusted to at least 2 pH units above the molecule's  $\text{pK}_a$ .



To elute, the opposite is true. By adjusting the pH of the eluant to at least two pH units above or below the analytes' and/or sorbent's  $\text{pK}_a$ , one can effectively neutralize one or both functional groups; disrupting the electrostatic interaction allowing for elution to occur.

**Note:** Because the kinetic exchange processes between sample and sorbent functional groups are considerably slower for ion-exchange than for normal and reversed-phase, flow rates should be drop wise ( $\sim 1$  drop/second). One may also need to increase elution and wash volumes allowing for sufficient residence time for the mobile phase and stationary phase to interact.

## Basic Steps

1. **Sample Pre-treatment** – Salt concentration should be less than 0.1 M. Dilute sample 1:1 with buffer of appropriate pH to ensure analyte functional groups are ionized.

### Examples:

- Basic compounds: dilute with 10-25 mM buffer (e.g., potassium phosphate or ammonium acetate), pH 3-6
- Acidic compounds: dilute with 10-50 mM buffer (e.g., acetate buffer), pH 7-9

For interference laden samples (e.g. biological fluids) containing varying levels of salt concentration, use mixed-mode SPE technology.

2. **Condition/Equilibration** – If samples are in a non-polar solvent, the same solvent should be used to condition the SPE device. For aqueous samples, condition with 1-2 tube volumes of methanol or acetonitrile. Equilibrate with buffer similar/identical in pH and salt concentration to buffer used in the sample pre-treatment.

- 3. Sample Load** – Apply sample (from step 1) at a consistent and reduced flow rate of  $\sim 1$  drop/second to ensure optimal retention. Mass transfer kinetics of ion-exchange SPE are slower than reversed-phase and normal-phase. Reduced flow rate is critical for consistent recovery.
- 4. Wash** – Adequate control of pH and ionic strength should be maintained to prevent premature elution of the analytes of interest. Use buffer of appropriate pH (e.g. buffer used in sample pre-treatment) to remove polar interferences. More hydrophobic interferences can be removed using up to 100% methanol diluted in sample pre-treatment buffer.
- 5. Elution** – Elute at a consistent and reduced flow rate of  $\sim 1$  drop/second to ensure optimal compound desorption. The most common elution strategy is by pH manipulation. Also, most ion-exchangers exhibit some mixed-mode behavior. Addition of organic modifier is necessary to disrupt secondary reversed-phase interactions.

#### Examples:

- Basic compounds: elute with 2-5% ammonium hydroxide in 50-100% methanol
- Acidic compounds: elute with 2-5% acetic acid in 50-100% methanol.

#### Other elution strategies:

- Use an SPE eluate of higher salt concentration ( $> 1$  M)
- Use a more selective counter-ion to compete for ion-exchange binding sites

- 6. Eluate Post-treatment** – A number of elution strategies are available. Various elution strategies should be tested and optimized to minimize eluate post-treatment.

### Counter Ion Selectivity and Ion Exchange:

Counter ion selectivity is defined as the degree to which a counter ion is capable of competing with other counter ions for the functional group of an ion exchanger sorbent. Retention is facilitated by having a sorbent and/or sample matrix pre-equilibrated with a counter ion that is less selective than the analyte functional group (minimum competition). Analyte elution is facilitated by using buffers with counter ions more selective than analyte functional group.

#### For Cation Exchangers:

- $\text{Ca}^{2+} > \text{Mg}^{2+} > \text{K}^+ > \text{Mn}^{2+} > \text{RNH}_3^+ > \text{NH}_4^+ > \text{Na}^+ > \text{H}^+ > \text{Li}^+$

#### For Anion Exchangers:

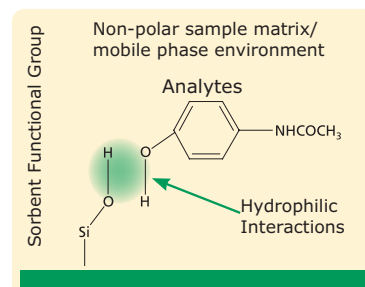
- Benzene Sulphonate  $>$  Citrate  $>$   $\text{HSO}_4^- > \text{NO}_3^- > \text{HSO}_3^- > \text{NO}_2^- > \text{Cl}^- > \text{HCO}_3^- > \text{HPO}_4^- > \text{Formate} > \text{Acetate} > \text{Propionate} > \text{F}^- > \text{OH}^-$

To change to a higher selective ion, pass 2-5 bed volumes of 1 N solution of the new counter ion through sorbent. To change to a lower selective ion, pass 5-6 bed volumes of 1 N solution of the new counter ion through sorbent.

**Note:** Number of bed volumes is dependent on how much less selective the new counter ion is than the present one on the sorbent.

### Normal-Phase SPE

In order for polar retention to occur between the sorbent and the sample, the analyte must be introduced to the SPE device in a non-polar sample or mobile phase environment. Therefore, typical sample matrices that can be employed in normal-phase SPE include hydrocarbon or fatty oils diluted in an organic solvent, hexane, isooctane, chlorinated solvents, THF, diethyl ether and ethyl acetate.



Most organic analytes exhibit some polar functionalities that can be exploited for normal-phase separation. Because many molecules exhibit polarity, each interaction can provide different levels of selectivity offering highly selective separations of compounds very similar in structure.

#### Retention Mechanism: Polar Interactions

- Hydrogen bonding, pi-pi, dipole-dipole and induced dipole-dipole

#### Sample Matrix: Non-polar samples

- Organic extracts of solids
- Very non-polar solvents
- Fatty oils, hydrocarbons

#### Analyte Characteristics: Analytes exhibiting polar functionalities

- Hydroxyl groups, carbonyls, amines, double bonds
- Hetero atoms (O, N, S, P)
- Functional groups with resonance properties

#### Elution Scheme: Polar interactions disrupted with a more polar solvent or solution

- Acetonitrile, methanol, isopropanol
- Combinations of buffer/solvent or solvent/solvent mixtures

#### Common Applications:

- Cleanup of organic extracts of soils and sludge
- Fractionation of petroleum hydrocarbons
- PCBs in transformer oil
- Isolation of compounds in cosmetics

## Basic Steps

- 1. Sample Pre-treatment** – Liquid samples should be initially extracted or diluted with a non-polar solvent such as hexane or a chlorinated solvent. Soil, sediment and other solid samples are initially extracted (soxhlet or sonication) with a non-polar solvent, and concentrated prior to SPE cleanup. Aqueous residues in the sample can reduce normal-phase retention. It may be necessary to further dry the organic extract with sodium sulfate or magnesium sulfate prior to SPE.
- 2. Condition/Equilibration** – Condition and equilibrate with 2-3 tube volumes of a non-polar solvent similar or identical to sample matrix resulting from sample pre-treatment.
- 3. Sample Load** – Apply sample (from step 1) at a consistent and reduced flow rate of ~1-2 drops/second to ensure optimal retention. The compounds should be in a non-polar solvent (e.g., hexane) for optimal retention. Note that methanol and acetonitrile are often used as elution solvents in normal-phase SPE and will often not promote compound retention during sample load.
- 4. Wash** – Sample interferences are often co-retained with compounds of interest during sample load. A wash step is necessary to elute interferences without prematurely eluting compounds of interest. In normal-phase SPE, 1-2 tube volumes of solvent used in sample pre-treatment and conditioning can be used during wash.
- 5. Elution** – Disrupt polar interactions with a solvent or solvent/buffer mixture more polar than both the sample and wash solutions. Typical elution solvents include water miscible organic solvents such as acetone, acetonitrile, methanol and isopropanol. Eluting with increasingly polar solvents or solvent mixtures in succession can also fractionate multiple compound classes. See “Common Normal-Phase Solvents” table for assistance.
- 6. Eluate Post-treatment** – Normal-phase SPE is often followed by GC analysis, and therefore requires a volatile sample matrix prior to injection. Use sodium sulfate or magnesium sample to remove residual moisture. Further SPE eluate concentration may also be necessary prior to analysis.

## Common Normal-Phase Solvents

Solvent	Elutropic ( $\epsilon^\circ$ ) or Elution Strength on Silica	
Hexane	0.00	Promotes Normal-Phase Retention
Isooctane	0.00	
Carbon tetrachloride	0.14	
Toluene	0.22	
Benzene	0.27	
<i>tert</i> -Butyl methyl ether	0.29	
Chloroform	0.31	
Methylene chloride (dichloromethane)	0.32	
Diethyl ether	0.29	
Ethyl acetate	0.43	
Tetrahydrofuran	0.35	Promotes Normal-Phase Elution
Acetone	0.45	
Acetonitrile	0.50	
40% methanol in acetonitrile	0.67	
20% methanol in diethyl ether	0.65	
20% methanol in methylene chloride	0.63	
Isopropanol	0.63	
Methanol	0.73	
Water	>0.73	
Acetic acid	>0.73	





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