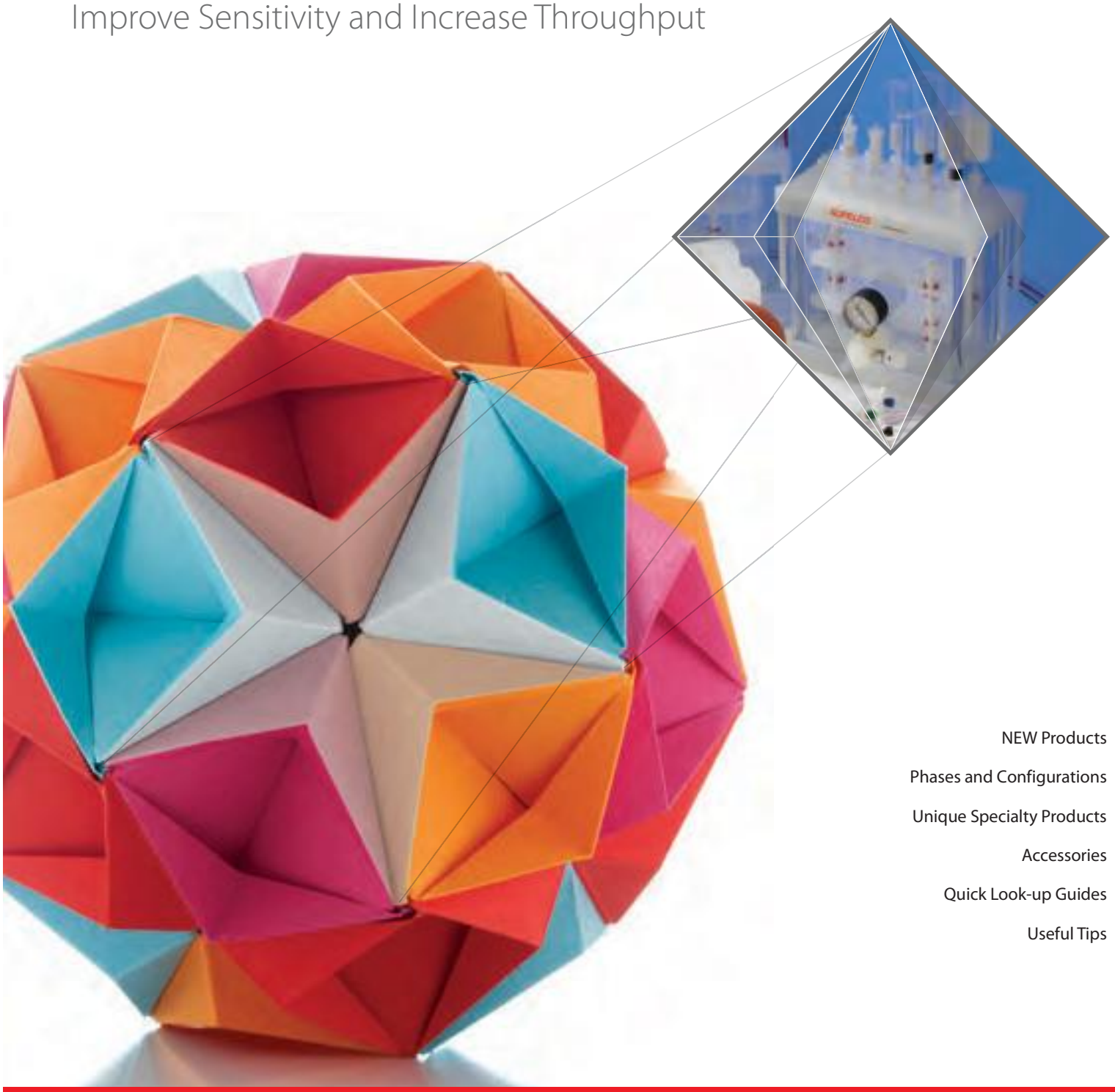
SUPELCO®
Solutions within.™

Solid Phase Extraction Products

Improve Sensitivity and Increase Throughput



NEW Products

Phases and Configurations

Unique Specialty Products

Accessories

Quick Look-up Guides

Useful Tips

SIGMA-ALDRICH®

A Brief History of Supelco Solid Phase Extraction (SPE)

Supelco®, the Analytical and Chromatography Division of Sigma-Aldrich®, first introduced SPE technology in 1985 under the Supelclean™ brand name. Shortly thereafter, we introduced our Visiprep™ Vacuum Manifold system.

With the focus on environmental, food/agrochemical and industrial analyses in 1992, we improved and extended the line further to include Supelclean ENVI™ - SPE products. In 1998 we introduced the Discovery® SPE line for pharmaceutical analysis.

Since 2007, the emphasis for Supelco Sample Prep R&D has been innovation. SupelMIP® SPE - Molecularly Imprinted Polymer Technology was introduced for the highly specific extraction of challenging analytes in difficult matrices. HybridSPE®- Phospholipid was developed for phospholipid and protein removal, as well as phospholipid enrichment.

In addition, Supelco was among the first to introduce a dispersive SPE (QuEChERS) product line for multi-residue pesticide analysis. Recently, the new Z-Sep sorbents have been added to the Supel™ QuE (dispersive SPE) family for lipid removal in difficult matrices.

20+ Years Ago

Supelclean and Supelclean ENVI

- Original pioneers of commercially available SPE Products
- Referenced in 100s of publications
- Developed, tested and quality controlled for environmental applications
- Also available in glass tubes and disk formats
- Unique chemistries such as ENVI-Carb™
- Documented applications in compliance to standardized EPA methods

Supelclean Specifications

Base Silica:	Irregular shape, acid washed for Supelclean ENVI
Mean Particle Size:	45 µm
Mean Pore Diam.:	60 Å
Tot. Pore Vol.:	0.8 cm^3/g
Specific Surf. Area:	475 m^2/g
Endcapped:	Yes (unless otherwise noted)
Frit:	Polyethylene (PE), 20 µm porosity (unless otherwise noted)

Discovery SPE

- Developed, tested and quality controlled for pharmaceutical and clinical applications
- Over 12 different phase chemistries ranging from mixed-mode SPE to polyamide adsorbents
- Available in 96-well and cartridge configurations
- Ultra-clean phases for highly sensitive analyses

Discovery Specifications

Base Silica:	Irregular shape, acid washed
Mean Particle Size:	50 µm
Mean Pore Diam.:	70 Å
Tot. Pore Vol.:	0.9 cm^3/g
Specific Surf. Area:	480 m^2/g
Endcapped:	Yes (unless otherwise noted)
Frit:	Polyethylene (PE), 20 µm porosity (unless otherwise noted)

Present

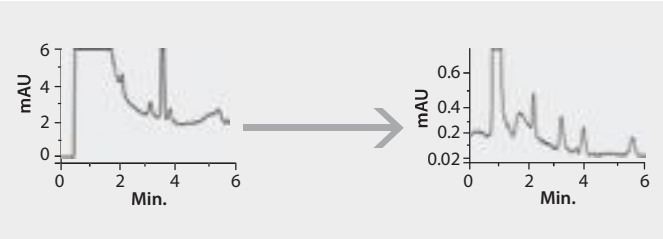
An Era of Innovative SPE

- SupelMIP SPE – Molecularly Imprinted Polymers for extreme selectivity
- HybridSPE-Phospholipid for quick and easy phospholipid and protein removal or phospholipid enrichment
- Supelclean Sulfoxide SPE for PCB analysis
- Supel QuE (dispersive SPE) for multi-residue pesticide analysis using the QuEChERS method
  - Z-Sep, Z-Sep/C18 and Z-Sep+ sorbents for lipid and pigment removal
- Supel-Select SPE – hydrophilic polymer SPE phases
- Supel Sphere Carbon/NH_2 for cleanup prior to pesticide residue analysis in half the time
- Supel Tox for fast and simple cleanup for mycotoxin analysis
- Supelclean EZ-POP NP for cleanup of edible oils prior to non-polar compound analysis

The Importance of SPE

Solid phase extraction is a form of digital (step-wise) 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 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.



For more applications and application details, visit
sigma-aldrich.com/spe

Use SPE for Samples that:

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

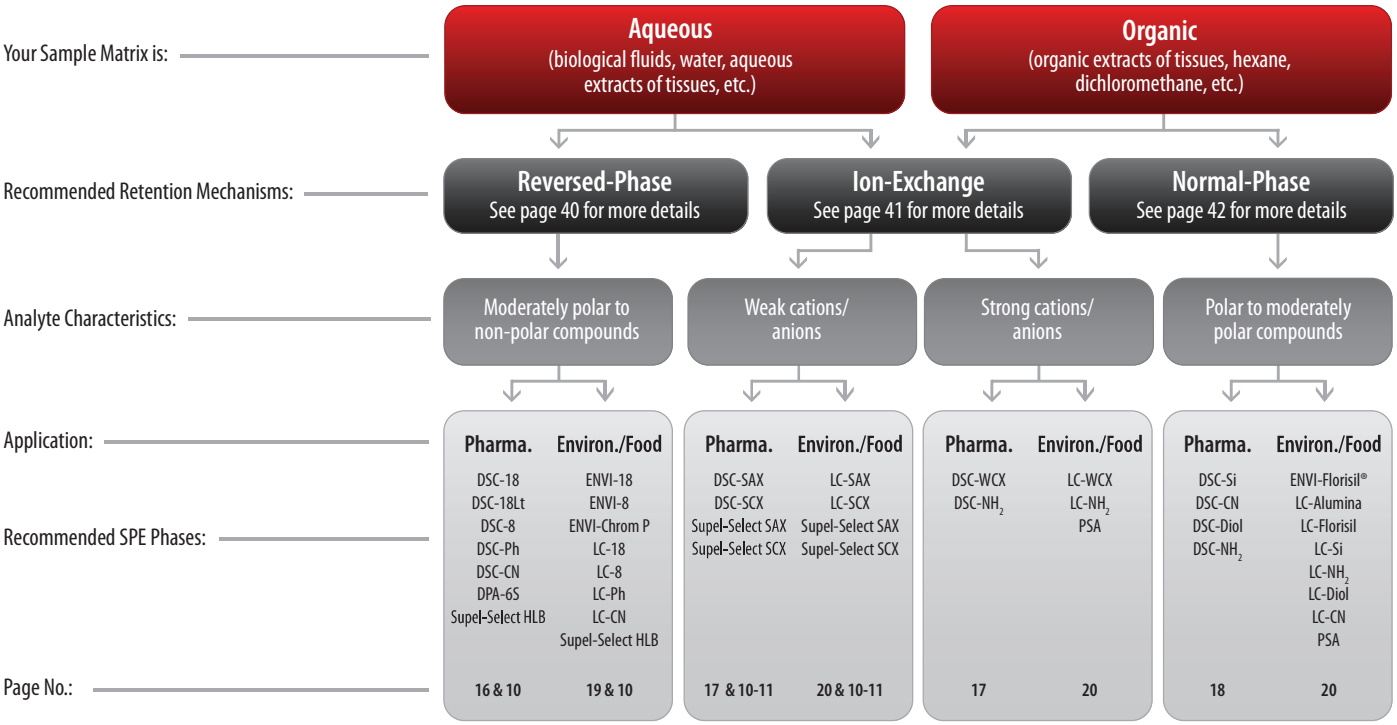
Common SPE Applications:

- 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

New and Featured Products

Phase	Page	Description
HybridSPE-Phospholipid	8	Combines the simplicity of protein precipitation with the selectivity of SPE for the targeted removal of proteins and phospholipids in biological samples.
Supel-Select HLB, SAX, SCX	10	Hydrophilic polymer for extraction of a broad range of diverse analytes from aqueous samples.
NEW! Supel Sphere Carbon/NH_2	26	Provides faster, more consistent flow for pesticide residue analysis.
NEW! Supel QuE Z-Sep Sorbents	27	Enhance sample cleanup for complex matrices by removing more fat and color from sample extracts than traditional phases for QuEChERS methods.
NEW! Supel Tox	30	Removes interferences associated with mycotoxin analysis.
NEW! Supelclean EZ-POP NP	32	Removes oily matrix interferences for the analysis of lipophilic persistent organic pollutants (POPs)

SPE Phase Selection Quick Look-Up Guide



Supelco SPE Specialty Phases

Application	Field/Application	Product	Page
Phospholipid removal/enrichment	Ph	HybridSPE-Phospholipid	8
Extraction of broad range of diverse analytes from aqueous samples	Ph, G, F	Supel-Select HLB, SAX, and SCX	10
SPE filter discs (EPA 500 methods)	Ph, E, G	Empore SPE	12
Molecularly Imprinted Polymer SPE	Ph, F, E	SupelMIP SPE	14
Adsorption of polar compounds from aqueous or methanolic solution	G, E, Ph	Discovery DPA-6S	16
Isolation of basic compounds from biological fluids	Ph, G	Discovery DSC-MCAX	17
SPE filter discs (EPA 500 methods)	E	Supelclean ENVI-18 and -8 DSK SPE Disks	19
Desalting proteins/peptides and other macromolecules	B	Supelclean LC-4 (wide pore)	19
Removal or isolation of polar compounds from organic matrices	E	Dual Layer Florisil/Na_2SO_4	20
Ion exchange in organic or aqueous solutions	G	Polymer SAX Rezorian Cartridge/Polymer SCX Reversible Tube	23
Nitrosamines in water (EPA Method 521)	E	Supelclean Coconut Charcoal	24
Polar compounds in water	E	Supelclean ENVI-Carb Plus	24
PCBs from transformer/waste oils	E	Supelclean Sulfoxide	24
Pesticide residue analysis	F	Supelclean ENVI-Carb	25
Pesticide residue analysis	F	Multi-layer Supelclean SPE Products	25
Pesticide residue analysis	F	NEW! Supel Sphere Carbon/NH_2	26
Pesticide residue analysis - QuEChERS	F	NEW! Supel QuE Z-Sep, Z-Sep/C18, and Z-Sep+	27
Mycotoxin analysis	F	NEW! Supel Tox Cartridges	30
FAMEs (cis/trans) analysis	F	Discovery Ag-Ion	33
Non-polar POP analysis in edible oils	F	NEW! Supelclean EZ-POP NP	32

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 µL	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 ~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.

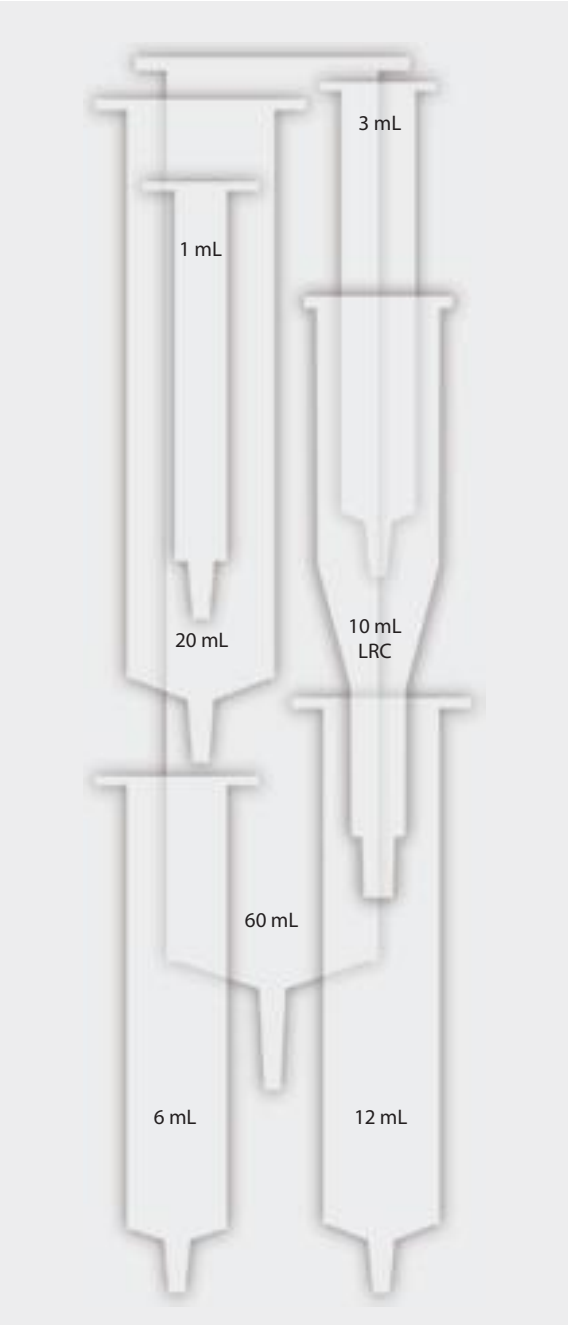
FREE SPE MultiPaks for Method Development

SPE MultiPaks consist of an assortment of SPE phase chemistries and tube dimensions ideally suited for method development. The mix of phase chemistries available in these MultiPaks allows you to screen for optimal retention and selectivity required to achieve your sample prep objectives.

Available SPE MultiPaks

- HybridSPE-Phospholipid
- Supel-Select HLB, SAX and SCX
- SupelMIP
  - NEW! Patulin
  - NEW! Aminoglycosides
  - NEW! Bisphenol A (BPA)
- Supel QuE (dispersive SPE, dSPE)
  - NEW! Z-Sep, Z-Sep/C18, Z-Sep+
  - NEW! 15 mL Shaker-Compatible Tubes
- Discovery Reversed-Phase
- Discovery Normal-Phase
- Discovery Ion-Exchange
- Discovery DSC-MCAX (Mixed-Mode Cation Exchange)
- Discovery DPA-6S (Polyamide)
- Supelclean ENVI-Carb (Graphitized Carbon)
- Discovery Ag-Ion
- Supelclean Dual Layer (for multi-residual pesticide analysis)
- Supelclean PSA
- NEW! Supel Sphere
- NEW! Supel Tox
- Supelclean Sulfoxide
- NEW! Supelclean EZ-POP NP

Most common SPE hardware: Polypropylene SPE tubes with PE Frit



To learn more about SPE MultiPaks or to request a FREE SPE MultiPak sample, visit
sigma-aldrich.com/spe

SPE Tubes and Specialty Hardware Quick Look-Up Guide

Additional Tubes and SPE Configurations

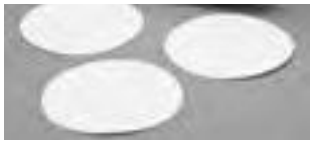
Glass SPE Tubes with PTFE and SS Frits (pg. 33 and 34)

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

Reversible SPE Tubes (pg. 23, 24, and 34)

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

SPE Disks (pg. 12: Empore, pg. 19: ENVI-8 and ENVI-18 DSK)



Allows for faster flow rates for processing large volume samples.

Rezorian Cartridges (pg. 23 and 34)

Luer-Lock® cartridges for positive pressure applications. Can also be used with vacuum manifold with proper luer connectors

Discovery SPE 96-Well Plates (pg. 23)

For high throughput sample preparation

Supel QuE (Dispersive SPE) for QuEChERS (pg. 27-29)

Salt and sorbent vials for dispersive SPE

Custom Capabilities

Supelco offers 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/custom-analytical**

Flangeless SPE Tubes (custom - inquire)

Accommodate robotic liquid vials handling systems (e.g. Gilson SPE 215™ System)

SPE Accessories Quick Look-Up Guide

SPE Manifolds

Visiprep™ DL and Standard Vacuum Manifold (pg. 35)

Uses disposable liners that prevent cross-contamination

Visiprep 5-Port Flask Manifold (pg. 35)

Collects the SPE eluate in round flasks for easy rotary evaporation

Preppy™ Vacuum Manifold (pg. 36)

Most economical

PlatePrep Vacuum Manifold (pg. 38)

For 96-well SPE
Useful for stacking SPE tubes

ENVI-Disk™ Holder (pg. 39)

Used with 47 mm SPE disks

Visi-1™ Single SPE Tube Processor (pg. 35)

For processing very few SPE samples

SPE Manifold Accessories

Visiprep Large Volume Sampler (pg. 36)

For processing larger sample volumes

Visidry™ Drying Attachment (pg. 36)

For drying SPE tubes or evaporating SPE eluate

Large Volume Reservoirs and Tube Adapters (pg. 33)

Useful for stacking SPE tubes, increasing headspace volume, or processing SPE tubes via luer syringe

SPE Elution Rack (pg. 36)

Simple racks for using SPE under gravity flow

KNF Laboport® Vacuum Pumps (inquire)

Provides vacuum source for vacuum manifolds

Trap Kit and Vacuum Gauge Bleed Valve (pg. 37)

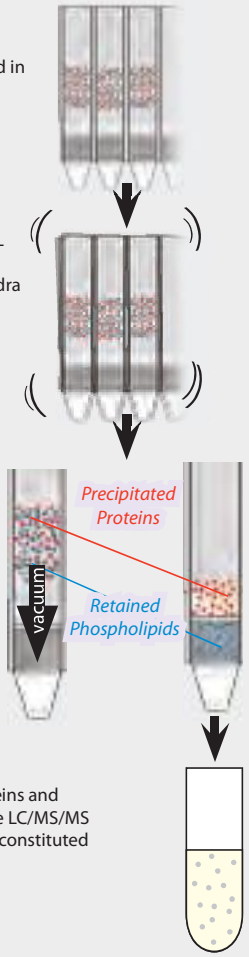
Additional vacuum accessories

HybridSPE-Phospholipid Technology

HybridSPE-Phospholipid (HybridSPE-PL) 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 1)**. 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 highly 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 2)**.

Figure 1. HybridSPE-PL "In-well" Method

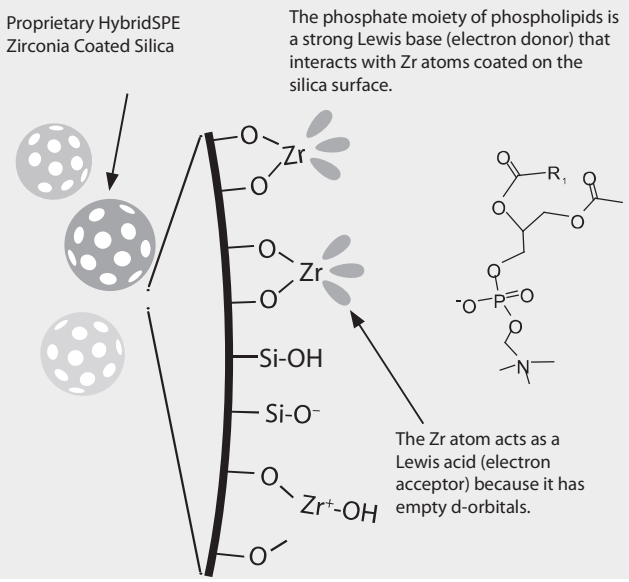
1. **Precipitate Proteins** by adding 100 µL plasma or serum to the HybridSPE-PL plate followed by 300 µL 1% formic acid in acetonitrile. Add I.S. as necessary.
2. **Mix** by vortexing/shaking HybridSPE-PL plate or by aspirating/dispensing with 0.5-1 mL pipette tip (e.g., TOMTEC Quadra liquid handler)
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/eluate** 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

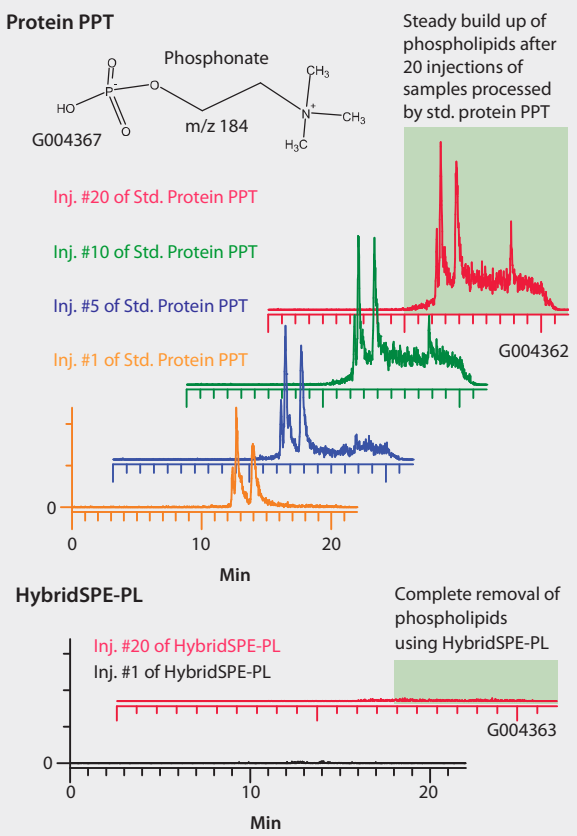
Figure 2. Lewis Acid-Base Interactions Between HybridSPE Zirconia Ions and Phospholipids



LC Accumulation of Phospholipids

With advances in LC/MS technology, many analysts are decreasing LC run time by incorporating ballistic gradients and sub-2 µm 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 3)**, potentially change LC retention selectivity, and elute uncontrollably downstream in an injection run sequence causing unpredictable ion-suppression effects and poor reproducibility. **Figure 3** compares a series of reversed-phase gradient LC/MS injections after standard protein PPT with HybridSPE-PL in which m/z 184 (phosphonate moiety of phospholipids) is monitored. Unlike traditional protein PPT techniques that use centrifugation to remove precipitated proteins, HybridSPE-PL 96-well plates contain a series of filters that allow users to concurrently remove proteins and phospholipids reducing LC column backpressure buildup, in particular for sub-2 µm HPLC columns that are more prone to clogging than larger particle size columns (2.7 - 5.0 µm) **(Figure 3)**.

Figure 3. Gradient RP LC/MS of Blank Plasma Samples Prepared by Standard Protein PPT vs. HybridSPE-PL



For more information, visit
sigma-aldrich.com/hybridspe-pl

Phospholipid Enrichment Using HybridSPE-Phospholipid Technology

Although HybridSPE-Phospholipid 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.

Experimental Flow Chart of Recovery of Phospholipids from Rabbit Plasma

Description	Qty.	Cat. No.
Well Plates		
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
HybridSPE-PLus 96-Well Plate Essentials Kit (contains: 96-well Plate, 50 mg/well, 1 cap mat , sealing film, and collection plate)	1	52818-U
SPE Cartridges		
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
Plate Accessories		
Round Well Cap Mat, Pierceable for HybridSPE-PLus	50	575680-U
96 Round/Deep Well Collection Plate, PP for HybridSPE-PLus	60	Z717266
96 Well-Plate Pre-cut Sealing Films	100	Z721581
Supelco PlatePrep Vacuum Manifold	1	57192-U
96-well Protein Precipitation Filter Plate (for offline protein precipitation)	1	55263-U

Supel-Select Polymeric SPE

Features and Benefits

- Extract and recover a very broad range of compounds from aqueous samples
- Reduce ion-suppression
- Amenable to generic methodology
- Resistant to overdrying for greater reproducibility
- Low UV and MS extractables
- Stringent production and QC guidelines
- Greater capacity for smaller elution volumes

HLB and Ion-Exchange Phases for a Wide Range of Applications and pH Conditions

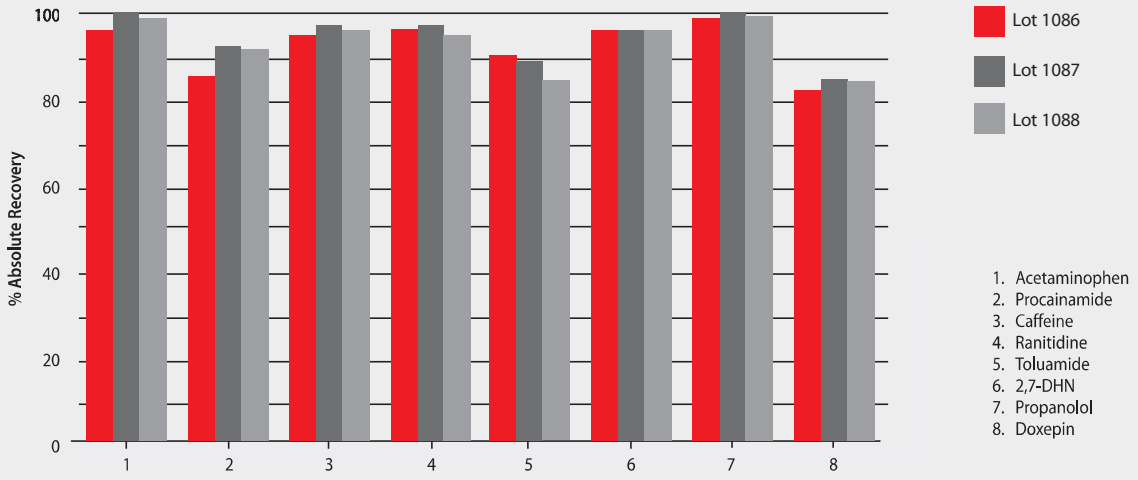
Supel-Select SPE phases are ideal for the solid phase extraction of a broad range of compounds from aqueous samples. While reversed-phase interactions dominate retention on the Supel-Select HLB, and the retention mechanisms of the Supel-Select SAX and SCX are predominately based on ion-exchange, the hydrophilic modifications of the styrene-based polymer backbone allow for retention and recovery of more polar compounds.

HLB Phase Chemistry:	Hydrophilic modified styrene polymer
SAX Phase Chemistry:	Quaternary amine functionalized hydrophilic modified styrene polymer; counter ion Cl-
SCX Phase Chemistry:	Sulfonic acid functionalized hydrophilic modified styrene polymer; counter ion H+
pH Compatibility:	0-14
Particle Size:	50-70 µm
MS Suitable:	Yes
Surface Area:	160-420 m^2/g
Pore Volume:	0.8-1.2 mL/g
Pore Size:	80-200 Å

High and Reproducible Recoveries

The hydrophilic, lipophilic balanced Supel-Select HLB SPE allows users to extract a broad range of compounds using a single sorbent and generic methodology. Analyte recovery was high across all the compounds tested, and results were highly reproducible across three production lots.

Supel-Select HLB Recoveries

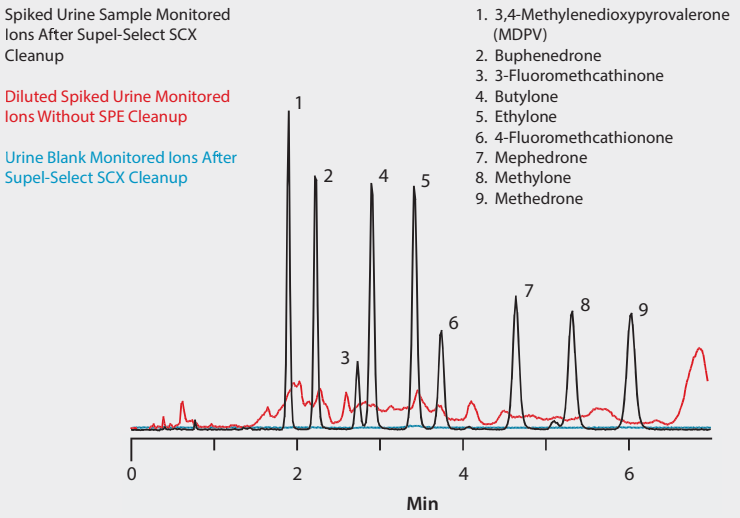


Application: Isolation and LC/MS Characterization of Illicit Bath Salts in Urine

The analysis of bath salts from urine samples is demonstrated using polymeric SPE sample preparation, followed by hydrophilic interaction liquid chromatography (HILIC) analysis with TOF-MS detection. Supel-Select SCX SPE is used for the processing and sample cleanup of the urine samples. The selective retention of the bath salts on the SCX cartridge is based upon the ion exchange mechanism between the anion functionality of the SCX and the basic functionality of the bath salts. The strong ionic interaction with the analytes enables high organic wash solvents to be used for displacement of the endogenous matrix, while maintaining retention of the analytes. Elution of the bath salts is achieved with the addition of a basic organic solvent. This approach results in a very clean sample.

The figure below illustrates the monitored bath salt ions in a spiked urine sample after SPE cleanup (black), in a diluted spiked urine sample without cleanup (red) and in a urine blank after SCX cleanup (blue). Notice the chromatogram containing the bath salts in the spiked urine sample after SPE cleanup contains no interfering peaks. Therefore, the effectiveness of the SCX cleanup is demonstrated.

sample/matrix:	1 mL urine spiked to 100 ng/mL of bath salt mixture
SPE tube:	Supel-Select SCX, 30 mg/1 mL (54240-U)
conditioning:	1 mL 1% formic acid in acetonitrile, then 1 mL water
sample addition:	1 mL spiked urine
washing:	1 mL water, 1 mL 1% formic acid in acetonitrile, 1 mL water
elution:	2 mL 10% ammonium hydroxide in acetonitrile
eluate post-treatment:	thoroughly mix via vortex agitation, evaporate 1 mL aliquot to dryness, reconstitute in 100 µL water:methanol
column:	Ascentis® Express HILIC (Si), 10 cm x 2.1 mm I.D., 2.7 µm (53939-U)
mobile phase:	(A) 5 mM ammonium formate acetonitrile; (B) 5 mM ammonium formate water; (98:2, A:B)
flow rate:	0.6 mL/min
pressure:	127 bar
column temp:	35 °C
detector:	MS, ESI+, 100-1000 m/z
injection:	1 µL
sample:	200 ng/mL in acetonitrile



Description	Qty.	Cat. No.
Supel-Select HLB 96-well SPE		
10 mg/ well	1	Inquire
30 mg /well	1	575661-U
60 mg/ well	1	575662-U
Supel-Select SAX 96-well SPE		
10 mg/well	1	Inquire
30 mg/well	1	575660-U
60 mg/well	1	575663-U
Supel-Select SCX 96-well SPE		
10 mg/well	1	Inquire
30 mg/well	1	575664-U
60 mg/well	1	575665-U
Supel-Select HLB SPE		
30 mg/1 mL	100	54181-U
60 mg/3 mL	50	54182-U
200 mg/6 mL	30	54183-U
500 mg/12 mL	20	54184-U
1 g/20 mL	20	54186-U

Description	Qty.	Cat. No.
Supel-Select SAX SPE		
30 mg/1 mL	100	54231-U
60 mg/3 mL	50	54233-U
200 mg/6 mL	30	54235-U
500 mg/12 mL	20	54236-U
1 g/20 mL	20	54237-U
Supel-Select SCX SPE		
30 mg/1 mL	100	54240-U
60 mg/3 mL	50	54241-U
200 mg/6 mL	30	54242-U
500 mg/12 mL	20	54243-U
1 g/20 mL	20	54245-U

For more information, visit
sigma-aldrich.com/supel-select

Empore Membrane SPE Products

Empore™ membrane SPE technology is comprised of SPE particles tightly enmeshed within a network of inert PTFE fibrils. The SPE-membrane fabrication process results in a very dense and uniform extraction medium that offers distinct advantages over traditional sorbent/packed-bed SPE products. Empore SPE technology allows for smaller bed weights, shorter analyte to pore diffusion paths and more efficient extractions.

Microscopic View of Empore SPE Disks comprised of 90% SPE particles: 10% PTFE, by weight

Save Time and Money with Empore SPE

Reduced SPE bed mass = Reduced SPE solvent and elution volumes

- Minimizes SPE eluate evaporation time
- Potentially allows for direct injection of the SPE eluate

Dense and uniform extraction medium = NO SPE channeling/voiding

- Efficient mass-transfer kinetics allow for faster flow rates
- Eliminate SPE fines improving column and instrument life

Comparison of Empore Cartridges to Traditional SPE Cartridges

Cartridge Dimension	Bed Vol.	Conditioning[1]	Elution[2]
Empore 7 mm (12 mg)/ 3 mL cartridge	50 µL	200-250 µL	100-150 µL
Traditional 500 mg/ 6 mL packed bed	60 µL	2400-3000 µL	1200-1800 µL
Traditional 100 mg/ 1 mL packed bed	120 µL	480-600 µL	240-360 µL

[1] Conditioning typically requires 4-5 x bed volumes
[2] Elution typically requires 2-3 x bed volumes

Available Formats

The Empore 96-well line is ideal for high throughput SPE allowing users to process up to 96 samples in parallel. The unique Empore technology has of a series of polypropylene (PP) pre-filters that are layered on top of the SPE disk. The PP pre-filter acts as a depth filter that provides faster flow rates and reduces the risk of clogging.

- Reduced elution volume (<100 µL) allows for direct injection or reduced eluate evaporation
- Faster flow rates without risk of recovery and reproducibility loss
- Proprietary pre-filter reduces risk of clogging
- Luer tip collar eliminates potential cross-contamination

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).

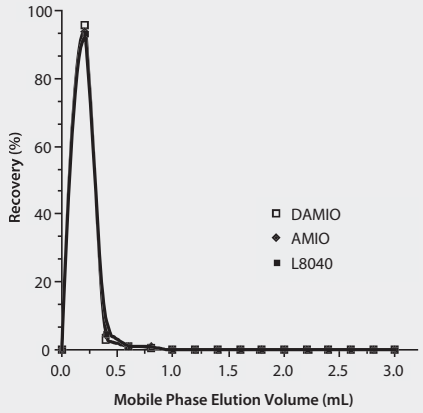
- Amenable to dozens of EPA and related environmental methods
- Developed for the efficient extraction of pollutants in large volume water samples

Empore SPE cartridges are packed with a PTFE membrane enmeshed with SPE particles. Layered above the SPE membrane is a polypropylene pre-filter to prevent particulates from reaching the underlying membrane. The dense particle packing and uniform distribution within the Empore membrane offers outstanding extraction efficiency and reproducibility.

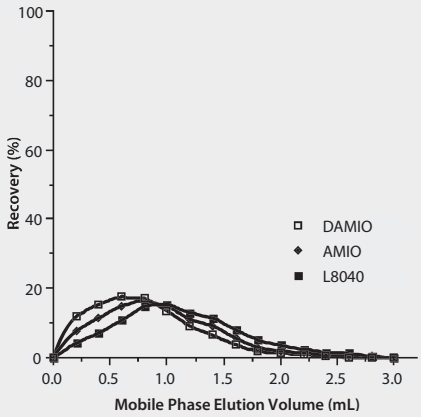
Recovery, Precision, and Elution Volume Profile of Empore SPE

Antiarrhythmic drug amiodarone (AMIO) and its metabolite, desethyl-amiodarone (DAMIO), were extracted from 250 µL of serum using reversed-phase SPE. Elution volume profiles for both the Empore and traditional packed SPE approaches are compared below. Only 0.5 mL of mobile phase elution volume was required for complete analyte elution using Empore SPE. In contrast, the traditional SPE packed column required over 2 mL to recover the analytes of interest.

SPE Empore Membrane (4 mm/1 mL)



SPE Packed Column (100 mg/1 mL)



Precision (between-run, n = 15)					
	Mean µg/mL	Standard Deviation µg/mL	Coefficient of Variation %	Recovery (at 300 µg/mL)	Sensitivity (lowest limit of quantitation)
AMIO	0.415	0.015	3.7	92-95%	0.05 µg/mL
DAMIO	0.412	0.013	3.3	90-93%	0.05 µg/mL

Description	Dimension	Qty.	Cat. No.
Cartridges			
Empore C18-SD (Standard Density)	4 mm/1 mL	100	66871-U
Empore C18-SD (Standard Density)	7 mm/3 mL	50	66872-U
Empore C18-SD (Standard Density)	10 mm/6 mL	30	66873-U
Empore UR-SD (Universal Resin)	7 mm/3 mL	50	66874-U
96-well			
Empore C18	5.5 mm/1.2 mL well	1	66875-U
Empore UR (Universal Resin)	5.5 mm/1.2 mL well	1	66877-U
Empore MPC (Mixed Phase Cation)	5.5 mm/1.2 mL well	1	66876-U
Empore Filter Plate	5.5 mm/1.2 mL well	1	66878-U
Disks			
Empore C18 Octadecyl	47 mm	20	66883-U
Empore C8 Octyl	47 mm	20	66882-U
Empore Oil and Grease	47 mm	20	66887-U
Empore Oil and Grease	90 mm	10	66898-U
Empore Styrene Divinyl Benzene (SDB-RPS)	47 mm	20	66886-U
Empore Styrene Divinyl Benzene (SDB-XC)	47 mm	20	66884-U
Empore Cation	47 mm	20	66889-U
Empore Anion-SR	47 mm	20	66888-U
Empore Chelating	47 mm	20	66894-U
Empore Carbon	47 mm	20	66896-U
Accessories			
Empore 96-well Vacuum Manifold	—	1	66879-U
Empore Filter Aid 400	—	1	66897-U
Empore Sealing Tape for 96-well	—	10 pads (25 sheets/pad)	66881-U

SupelMIP SPE – Molecularly Imprinted Polymers

Features and Benefits

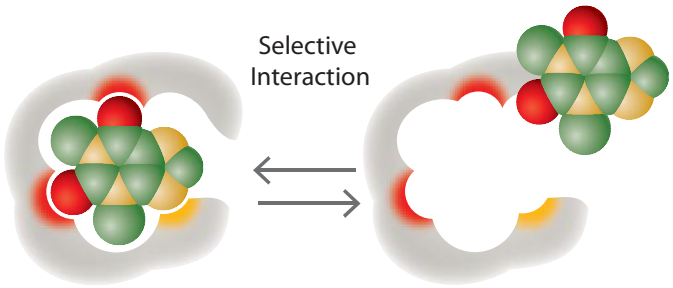
- Achieve lower detection limits through superior selectivity
- Reduce a ion-suppression
- Minimal to no method development required, giving reduced sample prep time
- Stable at broad pH ranges and high temperatures

The SupelMIP SPE line consists of highly cross-linked polymers that are engineered to extract a single analyte of interest or a class of structurally related analytes with an extremely high degree of selectivity. This is possible because selectivity is introduced during MIP synthesis in which a template molecule, designed to mimic the analyte, guides the formation of specific cavities or imprints that are sterically and chemically complementary to the analyte(s) of interest.

By careful design of the imprinting site, either by molecular modeling, experimental design or screening methods, the binding cavities can be engineered to offer multiple interaction points (ion-exchange, reversed-phase with polymer backbone, and hydrogen bonding) with the analyte(s) of interest. The MIP binding site is both chemically and sterically complementary to the analyte(s) of interest. This leads to a stronger interaction between the solid phase and the analyte(s). As a consequence, harsher wash conditions can be tolerated during SPE methodology, resulting in cleaner extracts. Because extraction selectivity is significantly improved, lower background is observed allowing analysts to achieve lower limits of detection.

SupelMIP Phases and Methods available for:

- **PAHs** (polycyclic aromatic hydrocarbons) in edible oils
- **Nitroimidazoles** in milk, eggs, and other food matrices
- **Non-steroidal anti-inflammatory drugs** (NSAIDs) in wastewater and other sample matrices
- **Fluoroquinolones** in bovine kidney, honey, and milk
- **Chloramphenicol** in milk, plasma, honey, urine, and shrimp/prawns
- **NNAL** 4-(Methylnitrosamino)-1-(3-pyridyl)-1-butanol in urine
- **TSNAs** (Tobacco Specific Nitrosamines) in urine and tobacco
- **ß-agonists and ß-blockers** in tissue, urine, and wastewater
- **Clenbuterol** in urine
- **Riboflavin** in milk
- **Patulin** in fruit matrices
- **Aminoglycosides** in animal tissue, cell culture, and honey
- **Bisphenol A** (BPA) in broth or mik-based matrices



Application: Highly Selective Sample Preparation for the Analysis of Aminoglycoside Antibiotics in Pork Muscle

This study utilizes the unique extraction capabilities of MIPs to successfully quantitate ten aminoglycosides by LC/MS/MS at 100 ng/g (400 ng/g for neomycin) with recoveries ≥ 70%. The SPE cleanup procedure, using SupelMIP SPE-Aminoglycosides, as well as the HPLC analysis, using an Ascentis® Express C18 HPLC column, is described in the condition section of **Figure 5**. Quantitation was performed using matrix-matched calibration standards, ranging from concentrations of 10 ng/mL to 1000 ng/mL.

Figure 5 depicts chromatograms of the analytes in pork muscle extracts. Recoveries for the 10 aminoglycosides are given in **Figure 4**. Most of the analyte recoveries were ≥ 70%, except for neomycin and tobramycin. Low recoveries for neomycin and tobramycin may be attributed to stronger binding of the analytes to the MIP sorbent due to the presence of several amino groups.

Figure 4. Aminoglycoside Recoveries in Pork Muscle

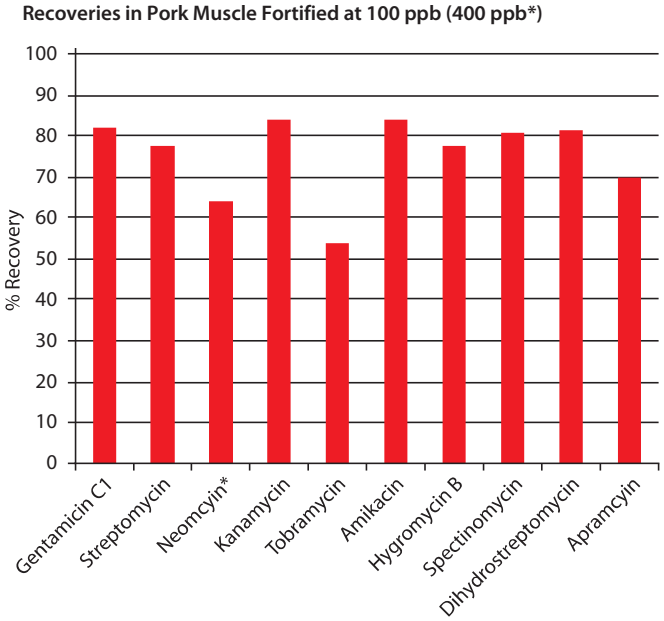
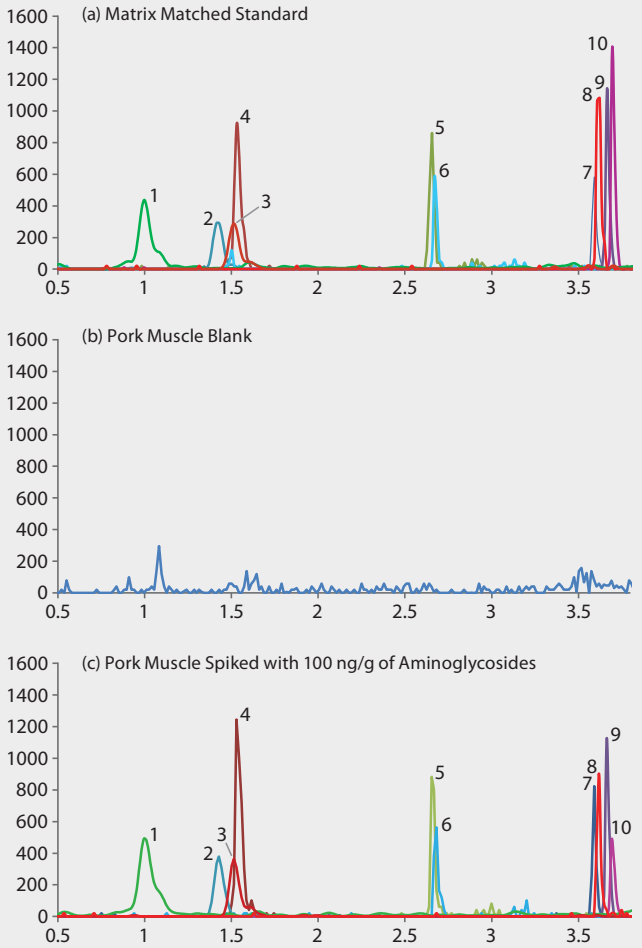


Figure 5. LC/MS/MS Analysis of Aminoglycosides after SupelMIP SPE Cleanup

sample/matrix: 3 mL of pork extract (For additional information regarding this application, refer to an article from Supelco Reporter 32.2 available at **sigma-aldrich.com/supelmip**)
SPE tube/cartridge: SupelMIP SPE – Aminoglycosides, 50 mg/3 mL (52777-U)
conditioning: 1 mL of methanol, then 1 mL of 50 mM potassium phosphate in water (pH = 7.8)
sample addition: 3 mL of pork extract
washing: 3 mL of water, followed by drying with slight vacuum for 10 seconds
washing: 1 mL of 50:50 dichloromethane:methanol (v/v), followed by drying with slight vacuum for 10 seconds
elution: 1 mL of 1% formic acid containing 5 mM heptafluorobutyric acid (HFBA) in 80:20 water:acetonitrile (v/v)
eluate post-treatment: thoroughly mix via vortex agitation, and transfer to polypropylene HPLC vials
column: Ascentis Express C18, 10 cm x 2.1 mm I.D., 2.7 µm (53823-U)
mobile phase: (A) 5mM heptafluorobutyric in water; (B) 5 mM heptafluorobutyric in acetonitrile
gradient: 20 to 90% B in 3.0 min; held at 90% B for 1 min; 90 to 20% B in 0.1 min; held at 20% B for 5.9 min
flow rate: 0.4 mL/min
column temp.: 40 °C
detector: MS/MS, ESI(+), MRM
injection: 10 µL

Analyte	Precursor	Product
Gentamicin C1	478.1	157.2
Streptomycin	582.1	263.2
Neomycin	615.0	161.1
Kanamycin	485.2	163.1
Tobramycin	468.1	163.1
Amikacin	586.2	163.1
Hygromycin B	528.1	177.1
Spectinomycin	351.1	333.1
Dihydrostreptomycin	584.2	263.1
Apramycin	540.2	217.1

1. Spectinomycin
2. Hygromycin B
3. Streptomycin
4. Dihydrostreptomycin
5. Amikacin
6. Kanamycin
7. Apramycin
8. Tobramycin
9. Gentamicin C1
10. Neomycin



Description	25 mg/3 mL pk 50	50 mg/3 mL pk 50	100 mg/3 mL pk 50	25 mg/10 mL [1](LRC) pk 50	96-well plates
PAHs (Polycyclic Aromatic Hydrocarbons)	—	52773-U	—	—	—
Nitroimidazoles	52734-U	—	—	—	—
NSAIDs	52769-U	—	—	—	—
Fluoroquinolones	53269-U	—	—	—	—
Clenbuterol	—	—	—	53201-U	—
β-agonists (class selective)	53225-U	—	—	53202-U	—
Full β-receptors (β-agonists and β-blockers)	53224-U	—	—	53223-U	—
Chloramphenicol	53209-U	—	—	53210-U	—
NNAL (4-(methylnitrosamino)-1-(3-pyridyl)-1-butanol)	53203-U	—	—	53206-U	53255-U
TSNAs (4 tobacco specific nitrosamines: NNK, NNN, NAB, NAT)	—	53222-U	—	[2] 53221-U	—
Riboflavin (vitamin B2)	—	—	—	53207-U	—
Aminoglycosides	—	52777-U	—	—	—
Patulin	—	—	52776-U	—	—
Bisphenol A (BPA)	—	—	52775-U, [3]54277-U	—	—

[1] LRC = large reservoir cartridge [2] 50 mg/10 mL (LRC), pk 50 [3] 100 mg/6 mL, pk 50

For more information, visit
sigma-aldrich.com/supelmip

SUPELCO®
Solutions within.™

Discovery SPE

Reversed-Phase

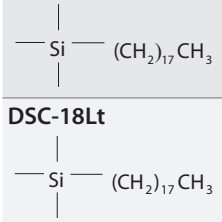
Discovery reversed-phase SPE products are specifically developed, tested and quality controlled for pharmaceutical and clinical applications. Experience greater and more reproducible recoveries for the quick and effective extraction, isolation and concentration of pharmaceuticals from biological fluids and other aqueous sample matrices.

For Discovery silica specifications, see page 2.
For general guidelines on reversed-phase SPE, see page 40.

DSC-18

- Polymerically bonded, octadecyl (18% C), endcapped
- Higher 18% C loading for increased binding capacities and higher recoveries
- The least selective phase: retains most organic analytes from aqueous matrices
- Beneficial for extracting numerous analytes diverse in structure from the same sample

DSC-18Lt

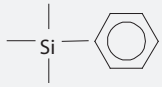


- Monomerically bonded, octadecyl (11% C), endcapped
- Increased retention for moderately polar hydrophobic molecules
- Used to elute very large hydrophobic molecules that are too strongly retained on DSC-18. Use this less retentive phase for the rapid release of hydrophobic compounds using weaker organic solvents at lower volumes

DSC-8

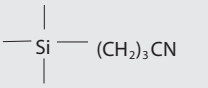
- Monomerically bonded, octyl (9% C), endcapped; lower carbon content than DSC-18Lt
- Used to elute very large hydrophobic molecules too strongly retained on DSC-18 or DSC-18Lt
- Use this less retentive phase for the rapid release of hydrophobic molecules using weaker organic solvents at lower volumes

DSC-Ph



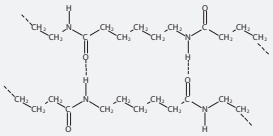
- Monomerically bonded, phenyl (7% C), endcapped
- Similar in polarity to DSC-8; however, electron dense aromatic ring offers some unique selectivity and retention

DSC-CN



- Monomerically bonded, cyanopropyl (7% C), endcapped
- Can behave as either reversed-phase or normal-phase
- Ideal for very hydrophobic analytes that may be irreversibly retained on more hydrophobic sorbents such as DSC-18
- Less retentive than DSC-Si or DSC-Diol when used as normal phase (organic matrices such as hexane or oils)
- Allows for the rapid release of very polar molecules irreversibly retained on very polar sorbents

DPA-6S



- Polyamide Resin: Particle Size: 50-160 µm, Surf pH: 4.5-7.5, Density: 0.2-0.3 cm^3/g, Water Content: <5%
- 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
- Useful for extracting tannins, chlorophyll, humic acid, pharmacologically active terpenoids, flavanoids, gallic acid, catechol A, protocatechuic acid and phloroglucinol
- Also useful for extracting aromatic carboxylic acids, nitroaromatic compounds and irreversibly retains quinones

Discovery Reversed-Phase SPE Products

Description	Qty.	DSC-18	DSC-18Lt	DSC-8	DSC-Ph	DSC-CN	DPA-6S
Discovery SPE Tubes							
50 mg/1 mL	108	52601-U	52610-U	52703-U	52723-U	52693-U	52624-U
100 mg/1 mL	108	52602-U	52611-U	52707-U	52725-U	52694-U	
500 mg/3 mL	54	52603-U	52613-U	52713-U	52727-U	52695-U	[1] 52625-U
500 mg/6 mL	30	52604-U	52615-U	52714-U	52728-U	52696-U	[2] 52626-U
1 g/6 mL	30	52606-U	52616-U	52716-U	52731-U	52697-U	[3] 52627-U
2 g/12 mL	20	52607-U	52618-U	52717-U	Custom	52698-U	[4] 52629-U
5 g/20 mL	20	52608-U	52621-U	52718-U	Custom	52699-U	[5] 52631-U
10 g/60 mL	16	52609-U	52622-U	52722-U	Custom	52700-U	[6] 52632-U
Discovery SPE 96-Well Plates							
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
Bulk Packing							
	100 g	52600-U	52623-U	57223-U	57227-U	57222-U	[7] 52633-U

[1] 250 mg/3 mL, [2] 250 mg/6 mL, [3] 500 mg/6 mL, [4] 1 g/12 mL, [5] 2 g/20 mL, [6] 5 g/60 mL, [7] 50 g

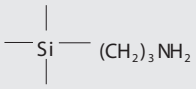
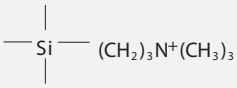
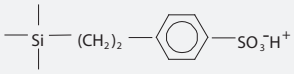
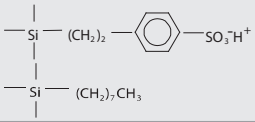
Discovery SPE

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 2.
For general guidelines on ion-exchange and mixed-mode SPE, see page 41.

DSC-NH_2 	• Polymerically bonded, aminopropyl phase that is very polar in nature (hydrogen bonding) allowing for both normal-phase and ion-exchange applications • A weak anion exchanger with a pK_a of 9.8. At pH 7.8 or below, the functional groups are positively charged • 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) • 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
DSC-SAX 	• A polymerically bonded quarternary amine that remains charged at all pH levels • Commonly used when extracting weaker cations (e.g., carboxylic acids) that may not bind strongly enough to weaker anion exchangers • Selectivity can be modified by changing the counter ion with the appropriate buffer during conditionin • Counter ion Cl^-
DSC-WCX 	• A polymerically bonded carboxy propyl phase with a K^+ counter ion and a pK_a of 4.8 • Its weak cation exchange properties carries a negative charge at pH 6.8 or above • 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 • Typically used when dealing with very strong cationic (high pK_a) compounds that may be irreversibly retained on strong cation exchangers
DSC-SCX 	• A polymerically bonded, benzene sulfonic acid functional group with a H^+ counter ion that is a strong cation exchanger due to its very low pK_a (<1.0) • Silica support allows for use with very organic solvents (no shrinking/swelling) • Excellent capacity (0.8 meq/g) for cleaning up solution phase combinatorial chemistry reactions (removing target molecules from reaction by-products and excess reagents) • The presence of the benzene ring offers some mixed-mode capabilities (hydrophobic interactions) that should be considered when extracting cations from aqueous matrices
DSC-MCAX 	• Packed bed contains both octyl (C8) and benzene sulfonic acid (SCX) bondings. (H^+ as counterion) • Developed for superior selectivity/sample cleanup when isolating basic compounds from biological fluids • Dual retention mechanisms broadens retention for a range of neutral, basic, acidic and zwitterionic compounds • Greater ion-exchange capacity for isolating polar basic and zwitterionic compounds • Can be used to fractionate basic/zwitterionic compounds from acidic and neutral compounds

Discovery Ion-Exchange SPE Products

Description	Qty.	DSC-NH_2	DSC-SAX	DSC-WCX	DSC-SCX	DSC-MCAX
Discovery SPE Tubes						
50 mg/1 mL	108	52635-U	52661-U	52737-U	52684-U	52781-U
100 mg/1 mL	108	52636-U	52662-U	52739-U	52685-U	52782-U
500 mg/3 mL	54	52637-U	52664-U	52741-U	52686-U	52783-U[1]
500 mg/6 mL	30	52638-U	52665-U	52742-U	52688-U	52784-U[2]
1 g/6 mL	30	52640-U	52666-U	52743-U	52689-U	52788-U, 52786-U[3]
2 g/12 mL	20	52641-U	52667-U	52744-U	52690-U	—
5 g/20 mL	20	52642-U	52668-U	52745-U	52691-U	—
10 g/60 mL	16	52644-U	52669-U	52746-U	52692-U	—
Discovery SPE 96-Well Plates						
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
Bulk Packing						
	100 g	57212-U	57214-U	57228-U	57221-U	—

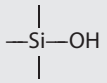
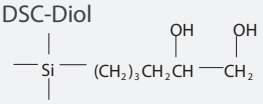
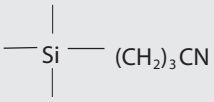
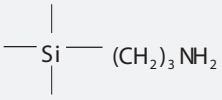
[1] 3 mL/100 mg, pk 54, [2] 300 mg/3 mL, pk 54, [3] 300 mg/6 mL, pk 30

Discovery SPE

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 42.

DSC-Si 	• Unbonded acid washed silica sorbent ideal for normal-phase SPE and other modified flash techniques • Considered the most polar normal-phase sorbent available • Excellent capacity for purifying solution phase CombiChem reactions when removing target molecules from reaction by-products and excess reagents • Available in Büchner funnel configurations for easy scalability
DSC-Diol 	• Polymerically bonded, 2,3-Dihydroxypropoxypropyl (7% C) • Polar sorbent most commonly used for normal-phase applications (polar extractions from non-polar matrices) • The sorbent's dihydroxy groups facilitate strong hydrogen bonding • Excellent selectivity when extracting structurally similar molecules
DSC-CN 	• Monomerically bonded, cyanopropyl (7% C), endcapped • Can behave as either reversed-phase or normal-phase • Ideal for very hydrophobic analytes that may be irreversibly retained on more hydrophobic sorbents such as DSC-18 • Less retentive than DSC-Si or DSC-Diol when used as normal-phase (organic matrices such as hexane or oils) • Allows for the rapid release of very polar molecules irreversibly retained on very polar sorbents
DSC-NH_2 	• Polymerically bonded, aminopropyl phase that is very polar in nature (hydrogen bonding) allowing for both normal-phase and ion-exchange applications • A weak anion exchanger with a pK_a of 9.8. At pH 7.8 or below, the functional groups are positively charged • 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) • 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

Discovery Normal-Phase SPE Products

Description	Qty.	DSC-CN	DSC-Si	DSC-Diol	DSC-NH_2
Discovery SPE Tubes					
50 mg/1 mL	108	52693-U	52652-U	52747-U	52635-U
100 mg/1 mL	108	52694-U	52653-U	52748-U	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	52698-U	52657-U	Custom	52641-U
5 g/20 mL	20	52699-U	52658-U	Custom	52642-U
10 g/60 mL	16	52700-U	52659-U	Custom	52644-U
Discovery SPE 96-Well Plates					
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
Bulk Packing					
	100 g	57222-U	52651-U	57229-U	57212-U

Supelclean and Supelclean ENVI

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 page 2.
For general guidelines on reversed-phase SPE, see page 40.

LC-18	• Monomerically bonded, octadecyl (10% C), endcapped • For reversed-phase extraction of nonpolar to moderately polar compounds. • pH range 2-8
LC-8	• Monomerically bonded, octyl (7% C), endcapped
LC-4 (Wide Pore)	• Butyldimethyl, wide pore (500 Å), endcapped • Larger pore size to accommodate larger macromolecules (e.g., proteins and peptides) • Commonly used for desalting proteins and peptides in aqueous samples
LC-Ph	• Monomerically bonded, phenyl (5.5% C), endcapped
LC-CN	• Monomerically bonded, cyanopropyl (7% C), endcapped
Hisep™	• Hydrophobic sites shielded by a hydrophilic surface for protein exclusion during sample load • Hydrophobicity similar to C8
ENVI-18	• Polymerically bonded, octadecyl (17% C), endcapped • Excellent for cleaning, extracting and concentrating pollutants from aqueous environmental samples • Higher 17% C loading for increased binding capacities and higher recoveries • Higher carbon loading also offers greater resistance to extreme pH conditions • Typical applications include herbicides, fungicides, pesticides and other aqueous hazardous waste materials • Ideal for EPA 500 series including 525.1 and 508.1
ENVI-18 and ENVI-8 DSK SPE Disks	• The SPE membrane equivalents of ENVI-18 and ENVI-8 packed bed SPE sorbents • Porous glass fiber membranes embedded with C18 or C8 silica particles • Provides faster flow rates and exhibits less clogging than PTFE discs for the extraction of organic contaminants from drinking water • Typical applications include PAHs, PCBs, phthalates, semivolatile organics, paraquat and diquat, pesticides and herbicides • Ideal for EPA 500 series including 525.1 and 508.1
ENVI-8	• Available in glass tubes with PTFE frits • High 14% C loading for increased binding capacities and higher recoveries • Higher carbon loading also offers greater resistance to extreme pH conditions • Excellent for cleaning, extracting and concentrating pollutants from aqueous environmental samples
ENVI-Chrom P (polystyrene divinylbenzene)	• Styrene/divinylbenzene co-polymer resin: Particle Size: 80-160 µm; Spherical Shape; Pore Size: 110-175 Å; Surface Area: 900 m^2/g • Highly crosslinked, neutral, specially cleaned styrene-divinylbenzene resin used to retain hydrophobic compounds with some hydrophilic functionality under the reversed-phase mechanism • Highly resistant to extreme pH conditions • Typical applications include aromatic and phenolic compounds from aqueous sample matrices • Used for priority pollutant phenols from aqueous samples
ENVI-Carb and ENVI-Carb II (graphitized carbon black)	• Surface Area: 120 m^2/g, Particle Size: 100/400 mesh (ENVI-Carb-II: 120/140 mesh) • Extreme affinity for organic polar and non-polar compounds from both non-polar and polar matrices when used under reversed-phase conditions • Carbon surface comprised of hexagonal ring structures, interconnected and layered into graphitic sheets • Non-porous nature of the carbon phase allows for rapid processing, adsorption does not require analyte dispersion into solid phase pores • 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)

For available configurations and part numbers, please see page 21.

Supelclean and Supelclean ENVI

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 2.
For general guidelines on ion-exchange and normal-phase SPE, see pages 41 and 42.

LC-SAX	• A strong anion exchanger • Quarternary amine, Cl^- counter-ion
LC-SCX	• Aliphatic sulfonic acid, Na^+ counter-ion, endcapped
LC-WCX	• Carboxylic acid, Na^+ counter-ion
LC-NH_2	• Monomerically bonded, aminopropyl (5% C)
PSA	• Polymerically bonded, ethylenediamine-N-propyl phase that contains both primary and secondary amines with pK_a of 10.1 and 10.9
ENVI-Florisil	• Magnesium silicate, mesh: 100/200, available with PTFE or stainless steel frits • Tested for US Environmental Protection Agency (EPA) Contract Laboratory Program (CLP) statement of work for pesticides • Highly polar material that strongly adsorbs polar compounds from non-polar matrices under normal-phase conditions • Typical applications include alcohols, aldehydes, amines, herbicides, pesticides, PCBs, ketones, nitro compounds, organic acids and phenols
Dual Layer Florisil/Na_2SO_4	• Dual layer SPE Tube (available as glass or PP) that contains Na_2SO_4 (upper layer) and Florisil (magnesium silicate; lower layer) separated and packed with PTFE frits • Florisil, activated, size- 60/100 mesh (150-200 mm), Na_2SO_4 Purity- 99.99 %, Density- 2.68 g/mL • Excellent for removing/isolating polar compounds from organic matrices • Na_2SO_4 layer aids in removing aqueous sample residues that may hinder Florisil performance and/or subsequent GC analysis • 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) • 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
LC-Florisil	• Magnesium silicate, mesh: 100/120
LC-Alumina A, N, and B	• Alumina-A for acidic pH (~5) • Alumina-N for neutral pH (~6.5) • Alumina-B for basic pH (~8.5) • Brockman Activation I for all Alumina SPE products, mesh: 60/325
LC-CN	• Monomerically bonded, cyanopropyl (7% C), endcapped
LC-Si	• Silica gel
LC-Diol	• Monomerically bonded, Diol (7% C), endcapped

For available configurations and part numbers, please see page 21.

Supelclean and Supelclean ENVI SPE

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	505706	57114	57137	57138	57219
				•54331-U[1]					
	ENVI-18 DSK SPE Disks			•57171[12]	•57170-U[13]				
	ENVI-8 DSK SPE Disks			•57172[12]					
	LC-18	504270	57012	57054	505471	57117	57135-U	57136	57202
	ENVI-8	57230-U	57231	57232	57233		57139	57140-U	
			•57106[1]	•57107[1]					
	LC-8	504157	505145	57052					57201
	ENVI-Chrom P	57143	•57224[5]	57226					•57217[11]
				•57225-U[7]					
	ENVI-Carb	57109-U	•57088[5]	57094		57128	57129	57130	•57210-U[11]
				•57092[7]		•57127-U[10]			
	ENVI-Carb C, mesh 80/100					•57149[10]			
	LC-4 (Wide Pore)		57089						
	Hisep		57076-U						
	LC-Ph	504599	505269						
Normal-Phase	LC-CN	504386	57013	57056			57141		
	LC-Diol	504718	57016						
	ENVI-Florisil		•57058[2]	•57046[3]	•57053[3]				
					•54095-U[1]				
	Dual Layer Florisil/ Na_2SO_4				•52582-U[1,9]				
					••54116-U[2,9]				
	LC-Florisil			•54333-U[1]	57057	57115	57131	57132	57209
					•54334-U[1]				
	LC-Alumina A		•57082-U[6]		•57083-U[8]				57026
	LC-Alumina B		•57084[6]		•57085[8]				57207
	LC-Alumina N		•57086[6]		•57087[8]				57028
	LC-Si	504041	505048	505374	57051	57116	57133	57134	57200
					•54335-U[1]				
	LC-NH_2	504483	57014	54059-U					57205
Ion Exch.	PSA		•52578-U[4]	52579-U					52738-U
	LC-SAX	504815	57017						57203
	LC-SCX	504920	57018						57204
	LC-WCX	505595	57061						

Footnotes/Color Codes

- • [1] glass SPE tubes, PTFE frits
- • [2] PP SPE tubes, PTFE frits
- • [3] PP SPE tubes, stainless steel frits
- • [4] 0.2 g/3 mL, pk 54
- • [5] 0.25 g/3 mL, pk 54
- • [6] 1 g/3 mL, pk 54
- • [7] 0.25 g/6 mL
- • [8] 2 g/6 mL, pk 30
- • [9] 2 g/2 g/6 mL, pk 48
- • [10] 1 g/12 mL, pk 20
- • [11] 50 g bulk
- • [12] 47 mm diam. disks, pk 24
- • [13] 90 mm diam. disks, pk 12

For a list of method development kits containing various phases, see next page.

Multi-Layer SPE

Developed to provide superior cleanup when conducting multi-residue pesticide analysis in food/agricultural matrices.

Description	Qty.	Cat. No.
ENVI-Carb-II/PSA		
0.3 g/0.6 g/6 mL	30	54058-U
0.5 g/0.5 g/6 mL	30	54067-U
0.5 g/0.3 g/6 mL	30	55119-U
0.5 g/0.5 g/20 mL	20	54217-U
ENVI-Carb-II/SAX/PSA		
0.5 g/0.5 g/0.5 g/12 mL	20	52574-U
SAX/PSA		
0.25 g/0.25 g/6 mL	30	52576-U
0.5 g/0.5 g/6 mL	30	52577-U

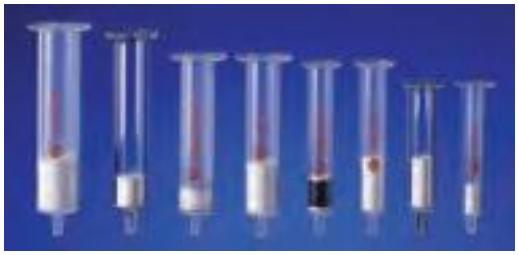
See also the new dual layer Supel Sphere products containing spherical materials on page 26.

Description	Qty.	Cat. No.
ENVI-Carb/LC-NH_2		
0.5 g/0.5 g/3 mL	20	54332-U
0.5 g/0.5 g/20 mL	20	54216-U
0.5 g/0.5 g/6 mL	300	54024-U
0.5 g/0.5 g/6 mL	30	54035-U
ENVI-Carb/NH_2/Silica		
0.5 g/0.4 g/0.6 g/12 mL	20	54034-U
0.5 g/0.4 g/0.6 g/20 mL	20	54036-U
Dual Layer Florisil/Na_2SO_4		
Glass tubes, PTFE frits, 2 g/2 g/6 mL	48	52582-U
PP tube with PE frits 2 g/2 g/6 mL	48	54116-U

SPE Method Development Kits

Supelclean SPE Method Development Kits

Supelclean SPE Method Development Kits consist of an assortment of SPE phase chemistries and cartridge configurations ideal for SPE method development. The range of phase chemistries available for each kit allows the user to profile for compound retention, elution and sample matrix selectivity.



Supelclean SPE Method Development Kits

SPE Method Development Kit	Kit A	Kit B	Kit C	Kit NP-3	Kit IX-3
Supelclean Packing			Sorbent Qty./Tube Size		
LC-Si	500 mg/3 mL	100 mg/1 mL 1 g/6 mL	500 mg/6 mL	500 mg/3 mL	
LC-8	500 mg/3 mL	100 mg/1 mL	500 mg/6 mL		
LC-18	500 mg/3 mL	100 mg/1 mL	500 mg/6 mL		
LC-CN	500 mg/3 mL	100 mg/1 mL	500 mg/6 mL		500 mg/3 mL
LC-Diol	500 mg/3 mL	100 mg/1 mL		500 mg/3 mL	
LC-NH_2	500 mg/3 mL	100 mg/1 mL		500 mg/3 mL	500 mg/3 mL
LC-Ph	500 mg/3 mL	100 mg/1 mL			
LC-SAX	500 mg/3 mL	100 mg/1 mL			500 mg/3 mL
LC-SCX	500 mg/3 mL	100 mg/1 mL			500 mg/3 mL
LC-WCX	500 mg/3 mL	100 mg/1 mL			500 mg/3 mL
LC-Alumina-A			2 g/6 mL	1 g/3 mL	
LC-Alumina-B			2 g/6 mL	1 g/3 mL	
LC-Alumina-N			2 g/6 mL	1 g/3 mL	
LC-Florisil			1 g/6 mL	–	
Qty. Tube/Kit	6	12	3	6	12
Cat. No.	57019	57009-U	57075-U	57074-U	57073

For more information about FREE SPE samples, see page 5 or go to
sigma-aldrich.com/spe-samples

Specialty Products for Pharmaceutical Analysis and Purification

Supelco SPE 96-well Plates

Supelco SPE 96-well plates answer the challenge of high throughput sample prep for pharmaceutical bioanalysis. These plates are packed with our high-quality Discovery SPE line, Supel-Select polymeric phases (see pg. 10), and our new and innovative HybridSPE-Phospholipid technology (see pg. 8). The uniform flow dynamics inherent with well plate technology offers a high level of reproducibility and throughput while maintaining excellent recoveries for increased sensitivity.

96-Well Plate Specifications

- One-piece polypropylene square well design
- 2 mL sample volume
- Polyethylene frit, 20 µm porosity (Discovery and Supel-Select HLB only)
- Compatible with TomTec Quadra 96®, Microlab® STAR, Packard Multi-Probe®, and most other 96-well automated SPE systems.

Phase	25 mg/well	50 mg/well	100 mg/well
HybridSPE-PL*	52794-U●	575656-U	
Supel-Select HLB		575661-U◆	575662-U▼
Supel-Select SAX		575660-U◆	575663-U▼
Supel-Select SCX		575664-U◆	575665-U▼
Discovery DSC-18	575601-U	Custom	575603-U
Discovery DSC-Si	Custom	575608-U	Custom
Discovery DSC-NH_2	Custom	Custom	575615-U

▲PE bottom frit (5 µm porosity)
◆Actual bed weight = 30 mg/well
▼Actual bed weight = 60 mg/well
●Actual bed weight = 15 mg/well
*See page 8 for more information. For collection plates, see page 9.

Polymer SAX Rezorian Cartridge

Retention Mechanism: Anion exchange

Sample Matrix Compatibility: Organic or aqueous samples

- A quarternary amine functional group bonded to styrene gel, 200/400 mesh (Dowex 1x8)
- Offers high capacity (3.5 meq/g) for extracting acidic compounds
- OH^- counter ion; 8% cross linking; ~42% moisture; max temp. 99 °C
- Excellent resistance to extreme pH conditions

Description	Qty.	Cat. No.
Polymer SAX Rezorian Cartridge		
Bed wt. 6 g, vol. 5 mL	10	2832-U
Bed wt. 14.4 g, vol. 13 mL	10	2833-U

Polymer SCX Reversible SPE Tube

Retention Mechanism: Cation exchange

Sample Matrix Compatibility: Organic or aqueous solutions

- A sulfonic acid functional group bonded to styrene gel, 200/400 mesh (DOWEX® 50Wx8)
- Offers high capacity (4.8 meq/g) for extracting basic compounds
- H^+ counter ion; 8% cross linking; ~54% moisture; max temp. 150 °C
- Excellent resistance to extreme pH conditions (1-14)

Description	Qty.	Cat. No.
Polymer SCX Reversible SPE Tube		
Bed wt. 700 mg, vol. 1 mL	10	Inquire**

*Available as custom only.
**Not available in Japan.

Specialty Products for 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
- Patent pending 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 Glass SPE Tube, 6 g/20 mL	5	**55252-U**
Supelclean Sulfoxide SPE, 3 g/6 mL	30	**55253-U**
Supelclean Sulfoxide, Bulk	100 g	**55254-U**
Empty Glass SPE Tube (17 mm I.D. x 137 mm L with PE frit, 20 mL, with PE frit, luer cap, and screw-top cap	5	**55255-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**

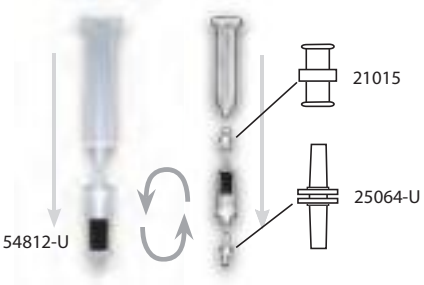
Supelclean ENVI-Carb Plus Reversible SPE for Highly Polar Compounds from Aqueous Samples

- 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)

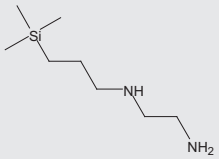
Description	Qty.	Cat. No.
Supelclean ENVI-Carb™ Plus Reversible SPE Tube, 0.4 g/1 mL	30	**54812-U**
Female Luer Coupler	20	**21015**
Male Luer Coupler	20	**25064-U**



For products for dioxin, furan, and PCB analysis, visit
sigma-aldrich.com/dioxin
or request our specialized brochure (JXB).

Specialty Products for 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. 27) 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, Supelco provides quality SPE products.

ENVI-Carb-II/PSA 	• Dual layer SPE tube that contains both Supelclean ENVI-Carb-II (upper layer) and PSA (lower layer) SPE sorbents (separated by PE frit) • Developed to offer superior cleanup when conducting multi-residue pesticide analysis in food (e.g., fruits, vegetables, etc.) • ENVI-Carb-II a graphitized non-porous carbon (100/140 mesh, surface area 100 m^2/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 • Supelclean PSA is a polymerically bonded, ethylenediamine-N-propyl phase that contains both primary and secondary amines • Supelclean PSA has a strong affinity and high capacity for fatty acids, organic acids, and some polar pigments and sugars • Tested for superior cleanliness using GC/FID and GC/MS
ENVI-Carb-II/SAX/PSA	• 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) • Developed to offer superior cleanup when conducting multi-residue pesticide analysis in food (e.g., fruits, vegetables, etc.) • ENVI-Carb-II is a graphitized non-porous carbon (100/140 mesh, surface area 100 m^2/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 • Supelclean PSA has a strong affinity and high capacity for fatty acids, organic acids, and some polar pigments and sugars • Supelclean SAX offers additional ion-exchange capacity for removing matrix components that may induce ion-suppression or enhancement during GC analysis
SAX/PSA	• Dual layer SPE tube that contains both Supelclean SAX (upper layer) and PSA (lower layer) SPE sorbents (separated by PE frit) • Supelclean SAX is a quarternary amine, Cl^- counter-ion • Supelclean PSA is a polymerically bonded, ethylenediamine-N-propyl phase that contains both primary and secondary amines • Ideal for removing matrix components (fatty acids, organic acids, polar pigments and some sugars) when conducting multi-residue pesticide analysis in foods • 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
ENVI-Carb	• Surface Area: 120 m^2/g, Particle Size:100/400 mesh • Extreme affinity for organic polar and non-polar compounds from both non-polar and polar matrices when used under reversed-phase conditions • Carbon surface comprised of hexagonal ring structures, interconnected and layered into graphitic sheets • Non-porous nature of the carbon phase allows for rapid processing, adsorption does not require analyte dispersion into solid phase pores • 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
PSA 	• Polymerically bonded, ethylenediamine-N-propyl phase that contains both primary and secondary amines • A weak anion exchanger with a pK_a of 10.1 and 10.9 • Similar to aminopropyl SPE phases (NH_2) in terms of selectivity, but has a much higher capacity due to presence of secondary amine (0.98-1.05 meq/g) • 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 • Has been shown to significantly reduce matrix-enhancement effects encountered during the GC analysis of food products • Bidendate nature of ligands allow for chelation
Supel Sphere Carbon/NH_2	• SPE tube packed entirely with spherical, non-friable particles • Improved flow characteristics and faster flow for gravity filtration • Reduced susceptibility to the formation of fines • 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 • Developed to offer superior cleanup when conducting multi-residue pesticide analysis from food • Carbon has a strong affinity toward planer molecules, and can isolate/remove pigments (eg., chlorophyll and carotinoids) and sterols commonly present in foods and natural products • Aminopropyl (NH_2) retains fatty acids, organic acids, and some polar pigments and sugars common in food matrices

For available configurations and part numbers, please see page 21.

Specialty Products for Pesticide Analysis

Supel Sphere Carbon/NH_2

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_2) removes organic acids, polar pigments and sugars

Spherical SPE Materials Optimize Flow and Increase Throughput

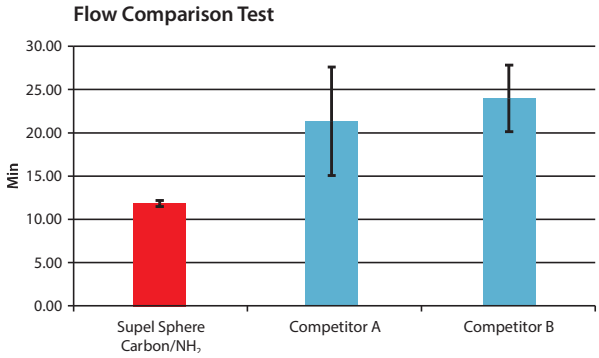
The demand for SPE cartridges with improved flow characteristics and reduced susceptibility to the formation of fines has led to the development of a family of SPE tubes packed entirely with spherical, non-friable particles. The Supel Sphere Carbon/NH_2 dual layer SPE tube contains both spherical carbon particles and spherical aminopropyl (NH_2) modified silica. It was developed to offer superior flow characteristics when conducting cleanup for multi-residue pesticide analysis from food.

Supel Sphere Carbon/NH_2 for Analysis of Pesticide Residues in Spinach

In a study comparing Supel Sphere Carbon/NH_2 with current products containing irregular materials, results illustrated that Supel Sphere Carbon/NH_2 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 6 and 7.

Figure 6. Flow Comparison Test

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



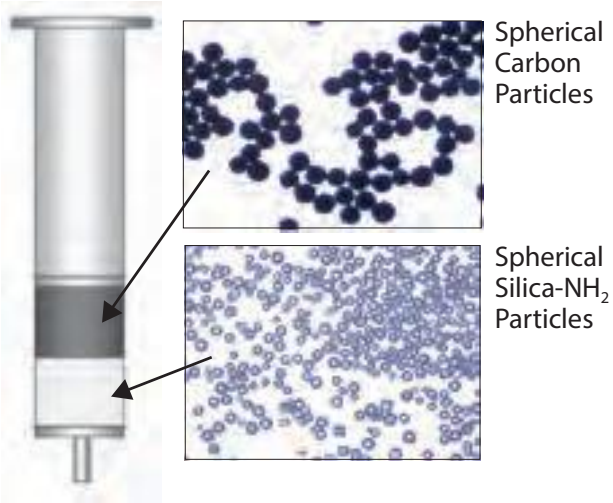
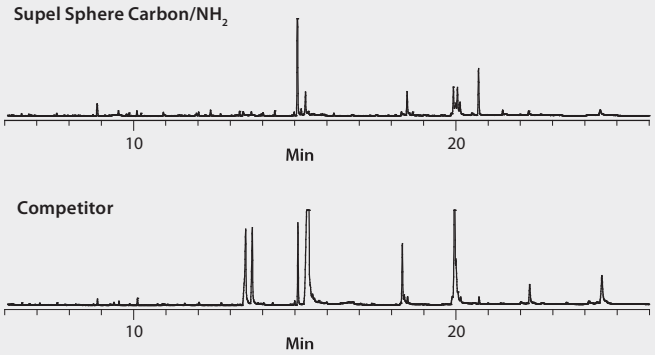


Figure 7. GC/MS Comparison of Cleaned Spinach Extracts

column:	SLB®-5ms, 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)



Description	Qty.	Cat. No.
Supel Sphere Carbon/NH_2 500 mg/500 mg, 6 mL	30	54283-U

For more information, visit
sigma-aldrich.com/supelsphere

Specialty Products for Pesticide Analysis

Supel QuE (Dispersive SPE) for "QuEChERS" Method

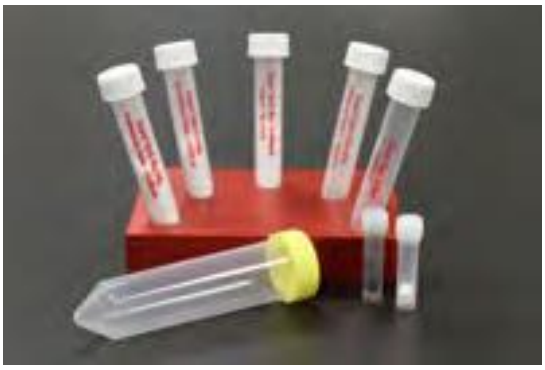
Quick and Simple Cleanup for Pesticide Residue Analysis

The "QuEChERS" method (**Qu**ick, **E**asy, **Ch**eap, **E**ffective, **R**ugged, and **S**afe), 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.

In QuEChERS methodology, food/agricultural samples are first extracted with an aqueous miscible solvent (e.g., acetonitrile) in the presence of high amounts of salts (e.g., sodium chloride and magnesium sulfate) and/or buffering agents (e.g., citrate) to induce liquid phase separation and stabilize acid and base labile pesticides, respectively. Upon shaking and centrifugation, an aliquot of the organic phase is subjected to further cleanup using SPE. Unlike traditional methods using SPE tubes, in QuEChERS methodology, cleanup is facilitated by mixing bulk amounts of SPE (e.g., Supelclean PSA, ENVI-Carb, and/or Discovery DSC-18) with the extract. After sample cleanup, the mixture is centrifuged and the resulting supernatant can either be analyzed directly or can be subjected to further minor treatment before analysis.

The Supel™ QuE line 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
sigma-aldrich.com/quechers



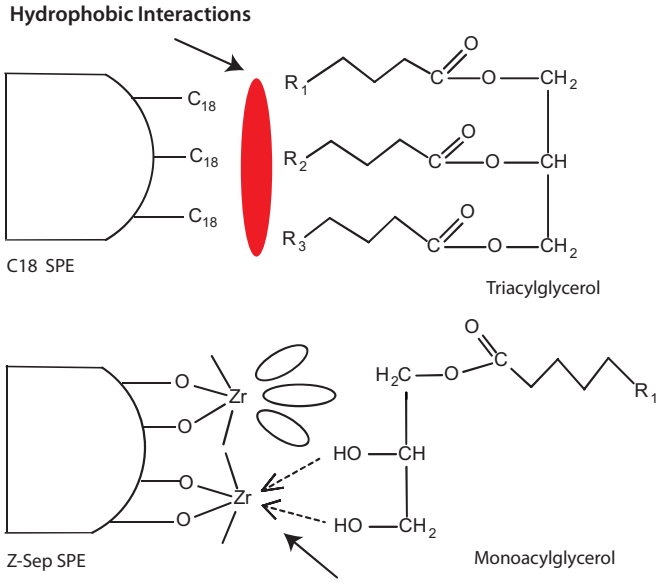
Features and Benefits

- Efficient and economic sample cleanup
- Pre-weighed amounts of sorbents and salts save labor and time
- High purity reagents from Sigma-Aldrich
- 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 synergetic interactions: the interaction between the polar group of the lipid and the proprietary bonded ion-exchange 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+, C18 and Z-Sep dual bonded to 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.

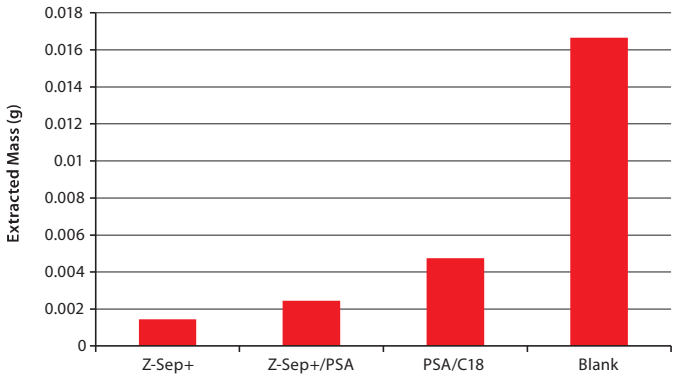
- Significantly diminishes fatty matrix interferences and various colors
- Provides more robust LC/MS and GC/MS methods by eliminating problematic matrix interferences
- Can replace C18 and PSA phases in current methods without additional method development



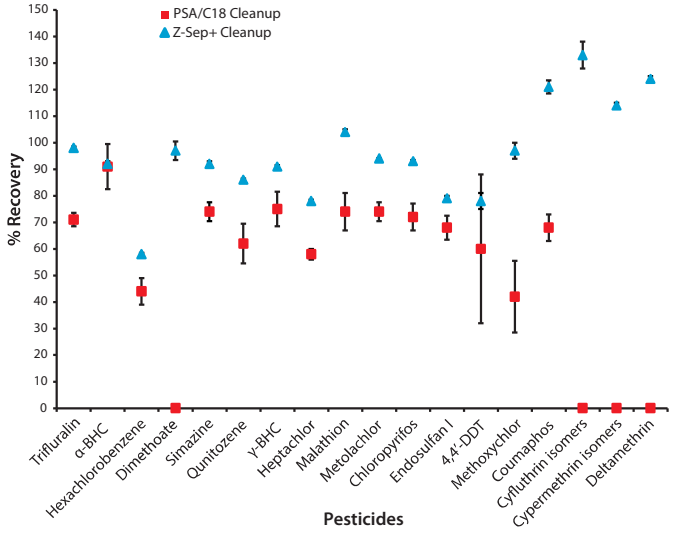
Analysis of Pesticides in Avocado using Z-Sep+ SPE Sorbent in QuEChERS Method for Sample Cleanup

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.

Total Extractables



Analyte Recovery of Selected Pesticides from Avocado



For more information, visit
sigma-aldrich.com/zsep

Supel QuE Products for QuEChERS and Related Products

Pre-Packed dSPE Tubes

Description	Qty.	Cat. No.
EN15662:2008 (15 mL centrifuge tubes, shaker compatible)		
Supel™ QuE PSA (EN) Tube, 15 mL 150 mg Supelclean PSA, 900 mg $MgSO_4$	50	55437-U
Supel QuE PSA/C18 (EN) Tube, 15 mL 150 mg Supelclean PSA, 150 mg Discovery DSC-18, 900 mg $MgSO_4$	50	55439-U
Supel QuE PSA/ENVI-Carb (EN) Tube 1, 15 mL 150 mg Supelclean PSA, 15 mg Supelclean ENVI-Carb, 900 mg $MgSO_4$	50	55446-U
Supel QuE PSA/ENVI-Carb (EN) Tube 2, 15 mL 150 mg Supelclean PSA, 45 mg Supelclean ENVI-Carb, 900 mg $MgSO_4$	50	55464-U
EN15662:2008 (12 mL centrifuge tubes)		
Supel QuE Citrate (EN) Tube 4 g $MgSO_4$, 1 g NaCl, 0.5 g NaCitrate dibasic sesquihydrate, 1 g NaCitrate tribasic dihydrate	50	55227-U
Supel QuE Citrate/Sodium Bicarbonate (EN) Tube 4 g $MgSO_4$, 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 150 mg Supelclean PSA, 900 mg $MgSO_4$	50	55228-U
Supel QuE PSA/C18 (EN) Tube 150 mg Supelclean PSA, 150 mg Discovery DSC-18, 900 mg $MgSO_4$	50	55229-U
Supel QuE PSA/ENVI-Carb (EN) Tube 1 150 mg Supelclean PSA, 15 mg Supelclean ENVI-Carb, 900 mg $MgSO_4$	50	55230-U
Supel QuE PSA/ENVI-Carb (EN) Tube 2 150 mg Supelclean PSA, 45 mg Supelclean ENVI-Carb, 900 mg $MgSO_4$	50	55233-U
EN15662:2008 (2 mL centrifuge tubes)		
Supel QuE PSA (EN) Tube, 2 mL 25 mg Supelclean PSA, 150 mg $MgSO_4$	100	55172-U
Supel QuE PSA/C18 (EN) Tube, 2 mL 25 mg Supelclean PSA, 25 mg Discovery DSC-18, 150 mg $MgSO_4$	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 $MgSO_4$	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 $MgSO_4$	100	55176-U

(continued on next page)

Description	Qty.	Cat. No.
AOAC 2007.01 (15 mL centrifuge tubes, shaker compatible)		
Supel QuE PSA (AC) Tube, 15 mL 400 mg Supelclean PSA, 1200 mg $MgSO_4$	50	55466-U
Supel QuE PSA/C18 (AC) Tube, 15 mL 400 mg Supelclean PSA, 400 mg Discovery DSC-18, 1200 mg $MgSO_4$	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 $MgSO_4$	50	55474-U
AOAC 2007.01 (12 mL centrifuge tubes)		
Supel QuE Acetate (AC) Tube 6 g $MgSO_4$, 1.5 g NaAcetate	50	55234-U
Supel QuE PSA (AC) Tube 400 mg Supelclean PSA, 1200 mg $MgSO_4$	50	55282-U
Supel QuE PSA/C18 (AC) Tube 400 mg Supelclean PSA, 1200 mg $MgSO_4$, 400 mg Discovery DSC-18	50	55283-U
Supel QuE PSA/C18/ENVI-Carb (AC) Tube 400 mg Supelclean PSA, 1200 mg $MgSO_4$ 400 mg Discovery DSC-18, 400 mg ENVI-Carb	50	55286-U
AOAC 2007.01 (2 mL centrifuge tubes)		
Supel QuE PSA (AC) Tube, 2 mL 50 mg Supelclean PSA, 150 mg $MgSO_4$	100	55287-U
Supel QuE PSA/C18 (AC) Tube, 2 mL 50 mg Supelclean PSA, 150 mg $MgSO_4$ 50 mg Discovery DSC-18	100	55288-U
Supel QuE PSA/C18/ENVI-Carb (AC) Tube, 2 mL 50 mg Supelclean PSA, 150 mg $MgSO_4$ 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 $MgSO_4$ 50 mg ENVI-Carb	100	Custom
Specialty Products for Challenging (Fatty/Lipid containing) Matrices (2 mL centrifuge tubes)		
Supel QuE Z-Sep Tube 75 mg Z-Sep	100	55411-U
Supel QuE Z-Sep/$MgSO_4$ Tube 50 mg Z-Sep, 150 mg $MgSO_4$	100	55417-U
Supel QuE Z-Sep/C18 Tube 20 mg Z-Sep, 50 mg Discovery DSC-18	100	55284-U
Supel QuE Z-Sep+ Tube 75 mg Z-Sep+	100	55408-U
Supel QuE Z-Sep+/$MgSO_4$ Tube 50 mg Z-Sep+,150 mg $MgSO_4$	100	55414-U
Specialty Products for Challenging (Fatty/Lipid containing) Matrices (15 mL centrifuge tubes, shaker compatible)		
Supel QuE Z-Sep Tube, 15 mL 500 mg Z-Sep	50	55491-U
Supel QuE Z-Sep/$MgSO_4$ Tube, 15 mL 300 mg Z-Sep, 900 mg $MgSO_4$	50	55503-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+/$MgSO_4$ Tube, 15 mL 300 mg Z-Sep+, 900 mg $MgSO_4$	50	55511-U

Description	Qty.	Cat. No.
Specialty Products for Challenging (Fatty/Lipid containing) Matrices (12 mL centrifuge tubes)		
Supel QuE Z-Sep Tube 500 mg Z-Sep	50	55403-U
Supel QuE Z-Sep/$MgSO_4$ Tube 300 mg Z-Sep, 900 mg $MgSO_4$	50	55407-U
Supel QuE Z-Sep/C18 Tube 120 mg Z-Sep, 300 mg Discovery DSC-18	50	55401-U
Supel QuE Z-Sep+ Tube 500 mg Z-Sep+	50	55296-U
Supel QuE Z-Sep+/$MgSO_4$ Tube 300 mg Z-Sep+,900 mg $MgSO_4$	50	55406-U
Non-buffered extraction tubes (12 mL centrifuge tubes)		
Supel QuE Non-Buffered Tube 1 4 g $MgSO_4$, 1 g NaCl	50	55294-U
Supel QuE Non-Buffered Tube 2 6 g $MgSO_4$, 1.5 g NaCl	50	55295-U
Specialty Extraction Salts		
Supel QuE Ammonium Sulfate Tube 12 mL) 4 g Ammonium Sulfate	1,000	54276-U
Empty Extraction Tubes (50 mL)		
50 mL empty Extraction Centrifuge Tubes	50	55248-U

Bulk Adsorbents and Salts

Description	Qty.	Cat. No.
Supelclean™ PSA, bulk sorbent	100 g	52738-U
Supelclean ENVI-Carb™, bulk sorbent	50 g	57210-U
Discovery DSC18, bulk sorbent	100 g	52600-U
Z-Sep+	20 g	55299-U
Z-Sep	20 g	55418-U
$MgSO_4$ (as cited in EN15662:2008)	var.	208094
Sodium citrate dibasic sesquihydrate	var.	71635
Sodium citrate tribasic dihydrate	var.	S4641
Sodium chloride	var.	71379
Sodium acetate	var.	241245

QuEChERS Shakers and Accessories

Description	Qty	Cat. No.
Benchmark Benchmixer™ XL Laboratory Shakers		
QuEChERS Shaker and Rack Starter Kit, USA compatible plug, AC input 115 V	—	55278-U
QuEChERS Shaker and Rack Starter Kit, EU compatible Schuko plug, AC input 230 V	—	55438-U
Multi-tube Vortexer, USA compatible plug, AC input 115 V	—	Z765503
Multi-tube Vortexer, EU compatible Schuko plug, AC input 230 V	—	Z765511
Benchmark Benchmixer XL Laboratory Shaker Racks		
50 mL QuEChERS Extraction Tube Shaker Rack	1	55279-U
15 mL QuEChERS Cleanup Tube Shaker Rack	1	Z765589
2 mL QuEChERS Cleanup Tube Shaker Rack	1	Z765554

Specialty Products for Mycotoxin Analysis

Supel Tox SPE Cartridges

Features and Benefits

- Removes interferences associated with mycotoxin analysis
- Basic and quick methodology requiring no additional method development
- Time associated with sample preparation is up to ten times less than that associated with immunoaffinity columns, the current industry standard
- No refrigeration required for shipping and storage

Supel Tox SPE Products

Description	Use
Supel Tox AflaZea	Cleanup of grains, feed, TMR samples, peanuts, peanut products, and aqueous solutions for detection of aflatoxin and zearalenone
Supel Tox DON	Cleanup of wheat, flour and corn for detection of deoxynivalenol (DON)
Supel Tox Tricho	Cleanup of grains and complex matrices for detection of Type A and B Trichothecenes
Supel Tox TrichoBind	Cleanup of grains and complex matrices for the detection and purification of Type A and B Trichothecenes
Supel Tox FumoniBind	Cleanup of whole grains and cereals for detection of fumonisin (B_1 and B_2)
Supel Tox OchraBind	Cleanup of whole grain and feed samples for the detection of ochratoxin A

Fast and Simple Cleanup for Mycotoxin Analysis

The need for a quick, simplistic sample cleanup approach prior to mycotoxin analysis has brought about a line of SPE cartridges that significantly decrease sample prep time, increase reproducibility, and are more user friendly as compared to the industry standard immunoaffinity columns (IAC). In addition, the Supel Tox SPE approach requires less equipment and fewer consumables, providing additional cost savings.

Table 1. Sample Cleanup Procedures Using Supel Tox AflaZea SPE Cartridges and Immunoaffinity Columns (n=3)

	Immunoaffinity	Supel Tox AflaZea SPE Cartridge
Sample Prep Time (post-extraction to pre-analysis)	• 60 minutes • 8 samples/day (if processing 1 at a time)	• 6 minutes • 80 samples/day (if processing 1 at a time)
Ease of Use	• Large volumes of liquid • Controlled drop rates • Numerous complicated steps • Additional buffer salts required • Must be refrigerated, brought to room temp before use	• Small volumes of liquid • Vacuum filtration used • Steps few and not complicated • No additional reagents required • Column does not require special storage conditions
Procedure (post-extraction to analysis)	Stage 1 (15 minutes) 1. Configure manifold for waste collection 2. Add 1 mL sample to 17 mL of phosphate buffered saline, vortex 3. Uncap/mount/drain cartridges by gravity 4. Apply reservoirs, load sample onto cartridges Stage 2 (15 minutes) 5. Rinse interferences 6. Reconfigure manifold for sample collection 7. Elute/collect sample Stage 3 (30 minutes) 8. Evaporate sample to dryness 9. Reconstitute sample and vortex 10. Transfer 0.2 mL sample to vial 11. Dilute sample and vortex Analysis	Purify and Transfer (6 minutes) 1. Configure manifold for sample collection 2. Mount cartridges 3. Load 2 mL sample 4. Elute and collect under vacuum 5. Transfer 0.2 mL sample to vial 6. Dilute sample and vortex Analysis

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Application: HPLC Analysis of Aflatoxins in Raw Peanut Paste

A comparison of sample processing time, product performance, and process simplicity associated with the use of IAC and SPE cleanup methods for the analysis of aflatoxins in peanut paste is described herein. Sample purification procedures comparing cleanup with a leading brand of IAC columns to SPE cleanup using Supel Tox AflaZea cartridges (n=3) are summarized **Table 1**. The time required for each procedure was recorded and averaged. Chromatographic analysis was performed by HPLC with florescence detection using a Discovery® C18 column and a KOBRA® electro-chemical cell for aflatoxin derivatization.

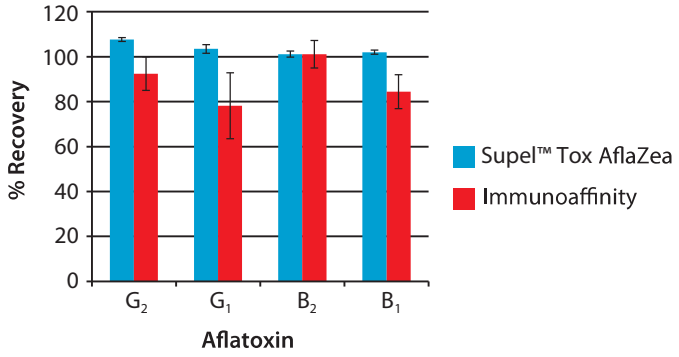
Sample Preparation (Time and Ease of Use)

As illustrated in **Table 1**, the use of the Supel Tox AflaZea SPE cartridges for sample cleanup was 10 times faster than that of the IAC columns. Use of the SPE cartridges eliminated the need for buffer solution, waste collection glassware, manifold reconfiguration, and equipment necessary to evaporate samples to dryness, making the SPE cartridges more user friendly than the IAC columns.

Analyte Recovery

The average % recoveries and %RSD values were compared for IAC and SPE purification techniques. **Figure 8** illustrates that Supel Tox AflaZea SPE cartridges gave higher analyte recoveries of B_1, G_1, B_2, and G_2 than the IAC columns used in this study. Also, as shown by the error bars, the %RSD was much lower for the SPE purification than the IAC purification, indicating that the SPE cartridges demonstrated better reproducibility than IAC for the analysis of aflatoxins in peanut paste.

Figure 8. Cleanup of Aflatoxins in Peanut Paste: Supel Tox AflaZea SPE Cartridges vs. Immunoaffinity Columns

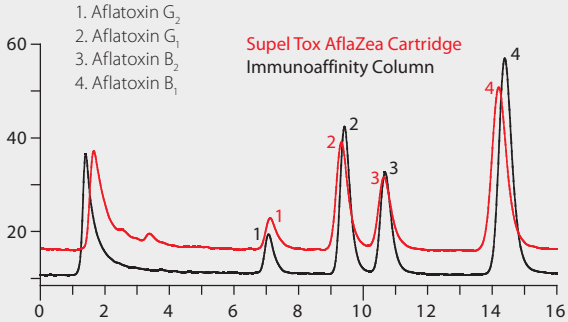


Chromatography

Figure 9 shows a comparison of SPE cleanup to IAC cleanup. Background response was negligible, and there was no significant difference in response when using SPE versus IAC methods. Therefore, the SPE method demonstrated equivalent sample cleanup performance to the IAC purification.

Figure 9. Spiked Peanut Paste Extracts After Cleanup

column: Discovery® C18, 15 cm x 2.1 mm I.D., 5 µm (50495521)
mobile phase: (A) water; (B) acetonitrile; (C) methanol; (72:12:12, A:B:C) with 0.780 g potassium bromide and 230 µL nitric acid
derivatization: KOBRA electrochemical cell
flow rate: 0.400 mL/min
column temp.: 35 °C
det.: florescence detector, excitation: 360 nm. emission: 440 nm
injection: 40 µL



Conclusion

This experiment illustrated that sample preparation using Supel Tox AflaZea SPE cartridges for cleanup was fast and simple compared to the IAC cleanup method. Because there were fewer steps needed to accomplish the SPE method, less variability was introduced into sample preparation, giving a more reproducible method. Also, the time associated with sample prep using SPE was far less than that associated with IAC, allowing for an ultimate increase in sample throughput. In addition, labware, reagents, and necessary equipment to perform sample preparation were minimal when using SPE. In this study, Supel Tox AflaZea SPE cartridges demonstrated superiority over IAC columns in terms of process simplicity, time required for sample preparation, and control of variation while maintaining the same sample cleanup performance associated with IAC purification.

Description	Qty.	Cat. No.
Supel Tox AflaZea SPE Cartridge, 6 mL	30	**55314-U**
Supel Tox DON SPE Cartridge, 6 mL	30	**55316-U**
Supel Tox Tricho SPE Cartridge, 6 mL	30	**55308-U**
Supel Tox TrichoBind SPE Cartridge, LRC	25	**55307-U**
Supel Tox FumoniBind SPE Cartridge, LRC	25	**55315-U**
Supel Tox OchraBind SPE Cartridge, LRC	25	**55318-U**

Specialty Products for 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 Persistant Organic Pollutants (POPs) from Oily Samples

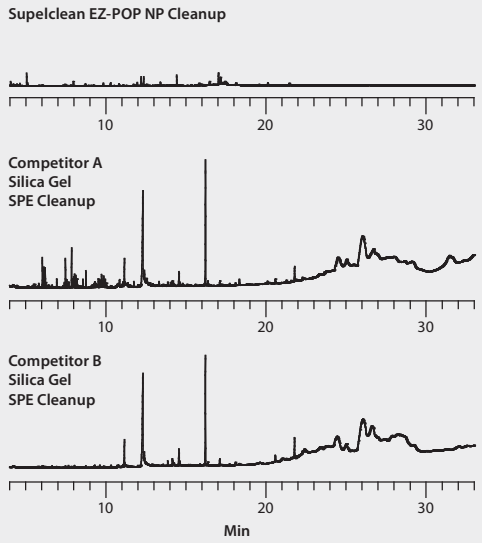
This dual-layer SPE cartridge offers superior cleanup for the extraction of non-polar POPs, specifically heavy and light PAHs, from edible oil matrices. The top Florisil layer retains polar functional groups such as acids and alcohols. The bottom Z-Sep/C18 layer binds fatty matrix through hydrophobic 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 more unwanted background than silica gel SPE, greatly decreasing the matrix effects (**Figure 10**). It produced better, more accurate, analyte recoveries than the silica gel SPE with good reproducibility (**Figure 11**). Thus, the Supelclean EZ-POP NP provides suitable matrix removal for rugged GC/MS analysis of PAHs in olive oil.

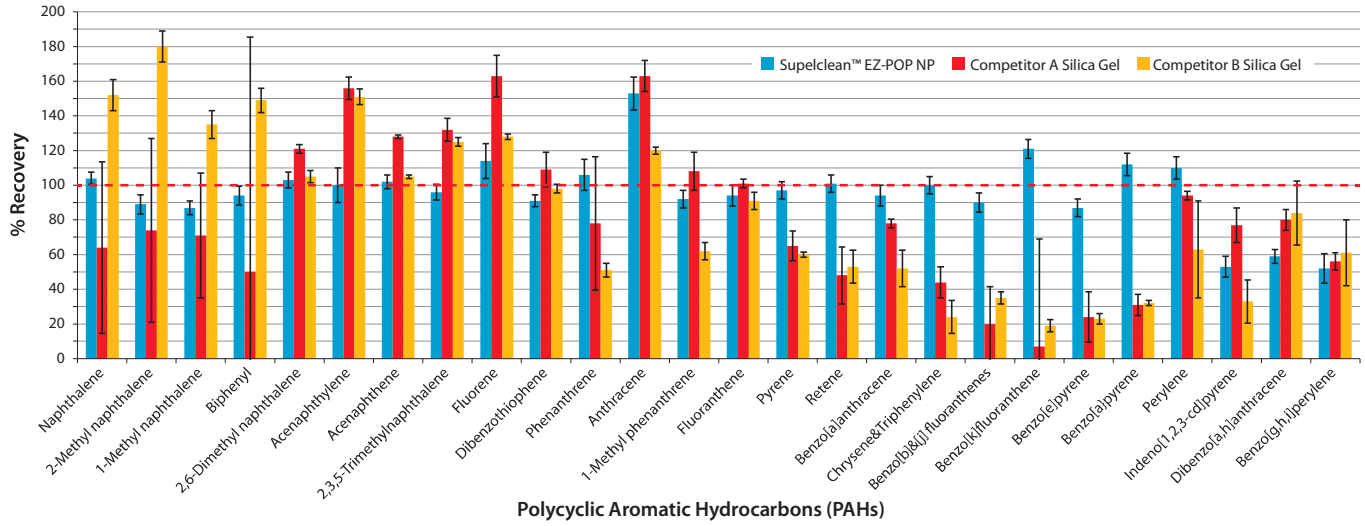
Figure 10. GC/MS Full Scan Chromatograms of Olive Oil Extract (same y axis)

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



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

Figure 11. Analyte Recovery of PAHs from Olive Oil Extract (n=3)



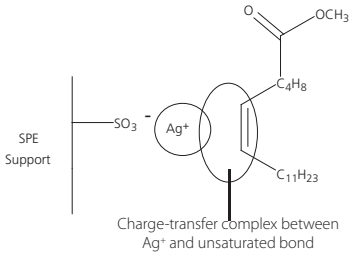
Miscellaneous Specialty Products and SPE Accessories

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



Description	Qty.	Cat. No.
750 mg/6 mL	30	54225-U
750 mg/1 mL reversible cartridge	10	54226-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.

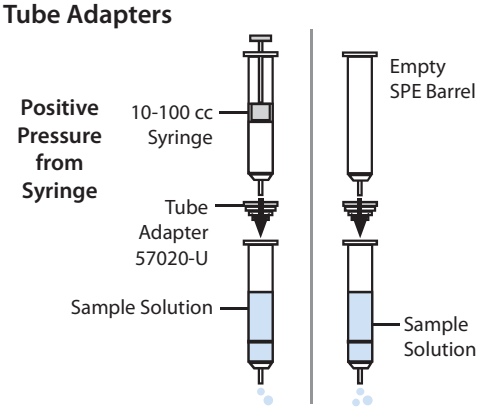
Features and Benefits

- 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.
Supelclean ENVI-18 SPE Tube		
glass hardware, PTFE frit, bed wt. 500 mg, vol. 6 mL	30	54331-U
Supelclean ENVI-8 SPE Tube		
glass hardware, PTFE frit, bed wt. 500 mg, vol. 3 mL	27	57106
glass hardware, PTFE frit, bed wt. 500 mg, vol. 6 mL	20	57107
Supelclean LC-Florisil SPE Tube		
glass hardware, PTFE frit, bed wt. 500 mg, vol. 6 mL	30	54333-U
glass hardware, PTFE frit, bed wt. 1 g, vol. 6 mL	30	54334-U
Supelclean LC-Si SPE Tube		
glass hardware, PTFE frit, bed wt. 1 g, vol. 6 mL	30	54335-U
Dual Layer Florisil/Na_2SO_4 SPE Tube		
bed A 2 g (Na_2SO_4), 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_2 line)

Description	Qty.	Cat. No.
SPE Tube Adapters for Polypropylene Tubes		
For 1, 3, 6 mL Tubes	12	57020-U
For 12, 20, 60 mL Tubes	6	57267
AutoTrace SPE Tube Adapters*		
For 3 mL Tubes	6	57123
For 6 mL Tubes	6	57126
* Allows SPE tubes to be used with AutoTrace® Automated Systems		
SPE Tube Adapter for Glass Tubes		
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.
Large Volume SPE Reservoir		
Polypropylene	30	54258-U
PTFE	3	54259-U

SPE Accessories

Empty SPE Hardware and Components

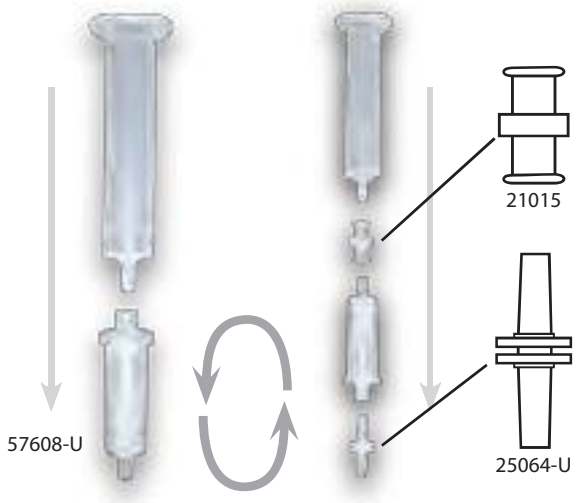
SPE Tube Components

Description	1 mL	3 mL	6 mL	12 mL	20 mL	60 mL
Empty SPE Tubes with and without Frits	108	54	30	20	20	16
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 (Qty. 12)	57021 (Qty. 12)	57022
Empty Glass SPE Tubes with PTFE Frits, 20 µm porosity	—	—	504394*	—	—	—
SPE Tube Caps (encloses top of SPE tubes)	108	54	30	20	20	20
PP cap for PP SPE tubes	52171-U	52172-U	52173-U	52174-U	52175-U	52176-U
PTFE cap for glass SPE tube	—	—	504343*	—	—	—
Frits for use with SPE tubes	216	108	60	40	40	32
PE Frits for PP SPE tubes, 20 µm porosity	57244	57180-U	57181	57182-U	57183	57184
PTFE Frits for PP SPE tubes, 20 µm porosity	57185	57186	57187	57188		57190-U
PTFE Frits for glass SPE tubes, 20 µm porosity	—	—	504327	—	—	—
SS Frit for PP SPE tubes, 20 µm porosity	—	—	57246-U	—	—	—
SPE Frit Insertion Tool						
SPE Frit Insertion Tool, pk 1	55217-U	55218-U	55219-U	55221-U	55224-U	55224-U
SPE Frit Insertion Tool Kit (includes all 5 tools for 1, 3, 6, 12 and 20/60 mL tubes)	—	—	—	—	55226-U	—

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

Miscellaneous SPE Hardware and Accessories

Description	Qty.	Cat. No.
Empty Reversible SPE Tube, non-flourous PP, w/PE frits		
0.5 mL	50	57602-U
1.0 mL	50	57607-U
2.0 mL	50	57608-U
Empty Flangeless PP SPE Tubes w/PE Frits, 20 µm porosity		
1 mL	108	Inquire
3 mL	54	Inquire
6 mL	30	Inquire
Empty PP Rezorian Tube Kit w/PE Frits, luer plugs and caps		
1.0 mL	50	57609-U
5.0 mL	50	57613-U
Empty 96-well SPE Plates		
2 mL deep square well, w/PE frits	1	Inquire
1.25 mL round well, w/PE frits	1	Inquire
Luer Caps, Plugs, and Couplers		
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



SPE Accessories – Vacuum Manifolds and Single Tube Processor

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 with 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 replaced following each extraction.

Features and Benefits DL and Standard Models

- Screw-type valves for SPE port for precise flow control
- 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.
Visiprep DL Solid Phase Extraction Manifold	
12-Port Model	57044
24-Port Model	57265
Disposable valve liners, PTFE, pk. of 100	57059
Visiprep Solid Phase Extraction Manifold	
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 Supelco 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)**

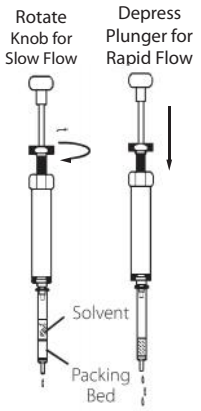
Description	Cat. No.
Visiprep 5-Port Flask Vacuum Manifold	
DL (Disposable Liner)	57101-U
Standard	57103-U
Visiprep 5-Port Vacuum Manifold Conversion Kit	
For converting 24-port model into DL 5- port flask model, includes DL 5-port lid and flask base plate	57104-U
For converting 24-port model into standard 5-port flask model, includes standard 5-port lid and flask base plate	57105-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

SPE Processing Accessories

Supelco 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.
Preppy Vacuum Manifold	
12-Port Model	57160-U
Preppy Replacement Parts	
Cover with flow control valves and solvent needle guides	57158-U
Collection Vessel Racks	
For 2 or 4 mL vials	57159-U
For 15 or 40 mL vials	57162-U
Accessories	
Vacuum Gauge/Bleed Valve Assembly	57161-U

Visidry Drying Attachment

Designed for our Visiprep Vacuum Manifold, the Visidry Drying Attachment (57100-U) also fits our economical Preppy manifold.

57030-U
12-Port Model
Order Separately

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 to each port can be independently adjusted.

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

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

Replacement SPE Tube Adapters (57020-U) listed on p. 29.

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.
Visiprep Large Volume Sampler		
for 12 mL, 20 mL, or 60 mL SPE Tubes (3 adapters)[1]	1	57272
for 3 mL or 6 mL SPE Tubes (4 adapters)	1	57275
Replacement Parts		
1/8" PTFE Tubes, color-coded	4	57276
Nuts and Ferrules, color-coded	4	57277
Stainless Steel Weights	4	57278
Tube Adapters, 1/4-28 threads		
For 3 mL or 6 mL Tubes	4	57273-U
For 12 mL, 20 mL, or 60 mL Tubes	3	57274-U

SPE Elution Rack for Gravity Feed Elution

This versatile stand-alone elution rack can be used with a variety of SPE tubes and receiving vessels, for simultaneous gravity feed extraction of up to 12 tubes. By assembling the plates in appropriate combinations, you can configure the rack to accept the following:

- 1 mL, 3 mL or 6 mL syringe barrel-type tubes
- Closed cartridge (reversible) tubes
- 5 mL or 10 mL volumetric flasks
- 2 mL or 4 mL vials
- Test tubes up to 15 mm I.D. x 10 cm

Description	Cat. No.
SPE Elution Rack	21043-U

SPE Accessories

Vacuum Manifold Replacement Parts and Accessories

Description	Qty.	Cat. No.
For 12-Port Manifold		
Cover, 12 flow control valves, gasket[1]	–	57031-U
Cover, 12 DL flow control valves, gasket[2]	–	57029
Gaskets	2	57033
Collection rack (base, 3 support rods, center plate, 10 mm test tube plate, 12 retaining clips)[3]	–	57037
Plate for 16 mm test tubes[3]	–	57039
Plate for 2 mL autosampler vials[3]	–	57040-U
Plate for 20 mL scintillation vials	–	57043
Splash guard	–	57045-U
For 24-Port Manifold		
Cover, 24 flow control valves, gasket[4]	–	57251
Cover, 24 DL flow control valves, gasket[5]	–	57266
Gaskets	2	57254
Collection rack (base, 2 support rods, center plate, 10 mm test tube plate, 8 retaining clips)[6]	–	57255
Plate for 16 mm test tubes[6]	–	57257
Plate for 2 mL autosampler vials[6]	–	57258
For 12-Port or 24-Port Manifold		
Valve Stem for Visiprep DL Vacuum Manifold	24	57146-U
Valve Stem for Visiprep/Preppy Vacuum Manifold	24	57147-U
Flow control valves[7]	2	57032
Solvent guide needles, PTFE[1,8]	12	57047
Solvent guide needles, stainless steel[7]	12	57036
Disposable valve liners for DL versions, PTFE[2,5]	100	57059
Disposable liner flow control valves[9]	2	57028
Liner guide needles, stainless steel[2,10]	12	57027
Vacuum gauge and bleed valve		57035-U
Retaining clips for collection racks	12	57041
Test tubes, 10 x 75 mm[1,2,8,10]	12	57042

[1] Compatible with **57030-U**
[2] Compatible with **57044**
[3] Compatible with **57030-U** and **57044**
[4] Compatible with **57250-U**
[5] Compatible with **57265**
[6] Compatible with **57250-U** and **57265**
[7] Compatible with **57030-U** and **57250-U**
[8] 2 packages included with **57250-U**
[9] Compatible with **57044** and **57265**
[10] 2 packages included with **57265**

Trap Kit for SPE Vacuum Manifolds

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

Long Stem Flow Control Knobs

If you have equipped your Visiprep Vacuum Manifold with long stem flow control valves, these control knobs will enable you to attach the Visidry Drying Attachment without removing the long stem valves.

NOTE: Not to be used w/24-port manifold to process 12 mL, 20 mL, or 60 mL tubes.

Description	Qty.	Cat. No.
Long Stem Flow Control Knobs	6	57093

SPE Accessories

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 a Discovery SPE 96-well plate to process up to 96 samples concurrently. The single valve control, parallel processing capabilities and uniform flow dynamics allow for easier method development, reduces 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**)

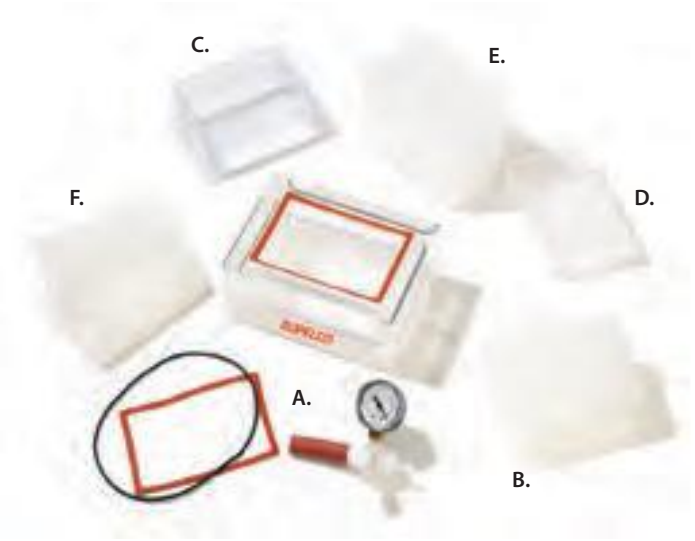
B. 1 96 Sq. Well Collection Plate, 2 mL, PP (**575653-U**)

C. 2 Disposable Reservoir/Waste Trays, PVC (**575654-U**)

D. 1 96 Sq. Well Piercable Cap Mat (**575655-U**)

E. 5 Reagent Reservoirs (**R9259-100EA**)

F. 1 Cluster Tube Rack (**CLS4410-960EA**)



Note: The PlatePrep Vacuum Manifold is not compatible with the Empore 96-well plate

Description	Qty.	Cat. No.
Supelco PlatePrep Vacuum Manifold	1	57192-U
96-Well Plate Starter Kit with PlatePrep Manifold	1	575650-U
Empore 96-well vacuum manifold	1	66879-U
PlatePrep Vacuum Manifold Replacement Parts		
Gasket/Connector Replacement Kit	1	57195-U
Remote Vacuum Gauge/Bleed Valve Assembly	1	57161-U
96-Well SPE Accessory Items		
96 Sq. Well Collection Plates, 0.35 mL, PP	50	575651-U
96 Sq. Well Collection Plates, 1 mL, PP	50	575652-U
96 Sq. Well Collection Plates, 2 mL, PP	50	575653-U
Disposable Reservoir/Waste Tray, PVC	25	575654-U
96 Sq. Well Piercable Cap Mats	50	575655-U
Reagent Reservoirs	100	R9259-100EA
Cluster Tube Rack	1	CLS4410-960EA

SPE Accessories

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. The filter base fits onto any standard 1-liter flask that has a 40/35 tapered ground glass neck. 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[1]	Z290610-1EA
Collection Tube, 25 x 250 mm[1]	57175

[1] 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

ENVI-Disk Clamp

- Eliminates leaks
- Attaches to any 34/45 tapered flask

When used with a standard 47 mm glass filtration apparatus, the ENVI-Disk Clamp creates a better seal, eliminating leaks with SPE extraction disks or when filtering HPLC mobile phase solvents.

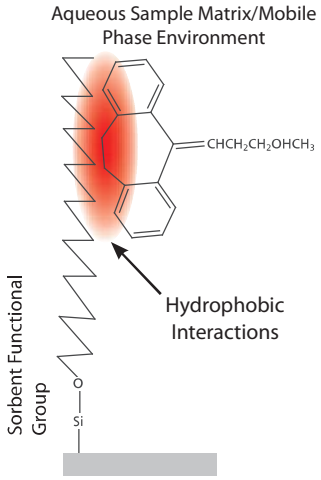
Use only with a filtration glassware funnel base that has a removable filtration stage, such as Supelco Mobile Phase Filtration Apparatus 1 **(58061)** or 2 **(58062-U)**, or with a funnel base **(58064 or 58068)**. It cannot be used with a permanent fritted glass filtration stage or stainless steel holder screen.

Description	Cat. No.
ENVI-Disk Clamp, 47 mm assembly	57260-U
Replacement PTFE stage	57261

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 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 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
- Aqueous extracts of tissues and 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 strengthens 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 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. **Condition/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 load 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 ~1-2 drops/second to ensure optimal 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 is 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 µL of 2% disodium EDTA per 100 µL mouse plasma
- 40 µL of 2% formic acid per 100 µL mouse plasma
- Other possible reagents (per 100 µL matrix): 40 µL of 2% TCA, 40 µL of 2% acetic acid, 40 µL of 2% TFA, 40 µL of 2% phosphoric acid, or 200 µL MeCN (protein ppt.).

2. If the SPE eluate needs to be evaporated prior to analysis, pass vacuum air through the SPE tube for ~10 minutes prior to elution. This will remove residual moisture that may prolong evaporation.

3. Consistent and slow flow rate (1-2 drops per second) during sample load and elution will improve recovery and reproducibility.

4. Reduce bed weight to minimize elution volume.

5. Increase bed weight to retain more polar compounds

6. Concern for sorbent overdrying is only critical during methanol conditioning.

7. 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.

SPE Methodology and Useful Tips

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

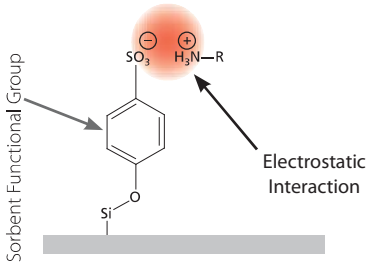
Elution Scheme: Electrostatic interactions disrupted via:

- 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 pK_a. For acidic analytes, the pH should be adjusted to at least 2 pH units above the molecule's 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 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 (~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 ~1-2 drops/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 ~1-2 drops/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:

- $Ca^{2+} > Mg^{2+} > K^+ > Mn^{2+} > RNH_3^{2+} > NH_4^+ > Na^+ > H^+ > Li^+$

For Anion Exchangers:

- Benzene Sulphonate > Citrate > $HSO_4^- > NO_3^- > HSO_3^- > NO_2^- > Cl^- > HCO_3^- > HPO_4^-$ > Formate > Acetate > Propionate > $F^- > OH^-$

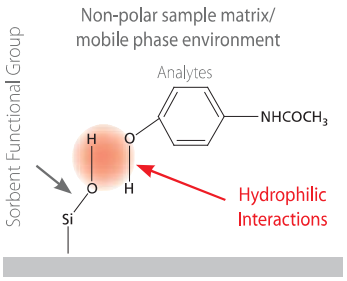
To change to a higher selective ion, pass 2-5 bed volumes of 1 N solution of the new counter ion through sorbent. To change to a lower selective ion, pass 5-6 bed volumes of 1 N solution of the new counter ion through sorbent.

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

SPE Methodology and Useful Tips

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 polar functionality, 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 equilibriate 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 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

Elutropic (e°) or Elution Strength Solvent	On Silica
Hexane	0.00
Isooctane	0.00
Carbon tetrachloride	0.14
Toluene	0.22
Benzene	0.27
tert-Butyl methyl ether	0.29
Chloroform	0.31
Methylene chloride (dichloromethane)	0.32
Diethyl ether	0.29
Ethyl acetate	0.43
Tetrahydrofuran	0.35
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

Promotes Normal-Phase Retention

Promotes Normal-Phase Elution

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