

Supelco Solid Phase Extraction Products



*Phases and
Configurations*

Accessories

*Specialty
Products*

*Quick Look-Up
Guides*

Useful Tips



Achieve Your Sample Prep Objectives



Supelco Solid Phase Extraction (SPE)

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

In 1992, with the focus on environmental, food/ agrochemical, and industrial analyses, we improved and extended the line further to include Supelclean ENVI- SPE products.

In 1998, we introduced the Discovery® SPE line for pharmaceutical analysis.



P001015

20+ Years Ago

Present

Supelclean SPE

- Among original pioneers of commercially available SPE products
- Time-tested technology
- Referenced in 100s of journal publications

Supelclean ENVI SPE

- Developed, tested, and quality controlled for environmental applications
- Over 10 different phase chemistries ranging from our unique ENVI-Carb to ENVI-18 DSK Disk SPE
- Available in glass tubes, PTFE and stainless steel frits for EPA compliance
- Documented applications in compliance to standardized environmental methods

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 configurations for high throughput processing
- Available in Büchner Funnel configurations for large scale purification
- Ultra-clean phases for highly sensitive analyses
- Acid washed to reduce metal chelating activity
- Narrower pore size distribution and low fines
- Consistent particle size and surface area coverage to ensure reproducible recoveries

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 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)

For more sample preparation and purification products, like flash chromatography, please visit our web site, sigma-aldrich.com/supelco or refer to the current Supelco catalog.

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, improves qualitative and quantitative analysis, and 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.



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 waste water
- Pesticides and antibiotics in food/agricultural matrices
- Desalting of proteins and peptides
- Fractionation of lipids
- Water and fat soluble vitamins
- For more applications and application details, please visit our web site, sigma-aldrich.com/supelco or refer to the current Supelco catalog.

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SPE Phase Selection

Quick Look-Up Guide

Your Sample Matrix is:	Aqueous (biological fluids, water, aqueous extracts of tissues, etc.)		Organic (organic extracts of tissues, hexane, dichloromethane, etc.)					
	Reversed-Phase See page 24 for more details		Ion-Exchange See page 25 for more details		Normal-Phase See page 26 for more details			
	Moderately polar to non-polar compounds		Strong cations/ anions		Weak cations/ anions		Polar to moderately polar compounds	
	Pharma. Environ.		Pharma. Environ.		Pharma. Environ.		Pharma. Environ.	
Recommended SPE Phases:	DSC-18 DSC-18Lt DSC-8 DSC-Ph DSC-CN DPA-6S	ENVI™-18 ENVI-8 ENVI-Chrom P LC-18 LC-8 LC-Ph LC-CN	DSC-SAX DSC-SCX	LC-SAX LC-SCX	DSC-WCX DSC-NH ₂	LC-WCX LC-NH ₂ PSA	DSC-Si DSC-CN DSC-Diol DSC-NH ₂	ENVI-Florisil® LC-Alumina LC-Florisil LC-Si LC-NH ₂ LC-Diol LC-CN PSA
Page No.:	8	11	9	12	9	12	10	12

Supelco SPE Specialty Phases:

Phase Description	Page No.	Application
Discovery DSC-MCAX	9	Mixed-mode cation exchange for superior selectivity/sample cleanup when extracting basic compounds (most pharmaceuticals) from biological fluids (e.g., plasma, urine, etc.)
Discovery Ag-Ion	17	Silver Ion SPE for the fractionation of cis-trans isomers and other FAMES
Polymer SAX Rezorian™ Cartridge Polymer SCX Reversible Tube	17	Strong cation and anion exchanger on a styrene base particle. Offers much higher ion-exchange capacity than silica based ion-exchangers.
Discovery DPA-6S	8	Polyamide resin that adsorbs polar compounds containing multi -OH and -COOH groups. Useful for extracting polyphenolics and other natural compounds (e.g., flavenoids, chlorophyll, humic acid, etc.) from plant extracts.
Supelclean ENVI-18 and -8 DSK SPE Disks	11	Provides fast flow fraters for processing large volumes of water samples (≥ 0.5 L). Used in EPA 500 series methods – Drinking Water.
Supelclean ENVI-Carb	11	Extreme affinity for polar compound in aqueous samples and water miscible organic extracts. Commonly used in pesticide analysis of food samples.
Multi-layer Supelclean SPE Products (ENVI-Carb™, SAX, PSA, NH₂)	13	Developed to provide superior cleanup when conducting multi-residue pesticide analysis in food/agricultural matrices
Supelclean LC-4 (wide pore)	11	Used for desalting proteins/peptides and other macromolecules
Dual Layer Florisil/Na₂SO₄	12	For total petroleum hydrocarbon index according to European Method EN9377-2

SPE Bed Weight Quick Look-Up Guide

Choosing the Right Bed Weight and Tube Size

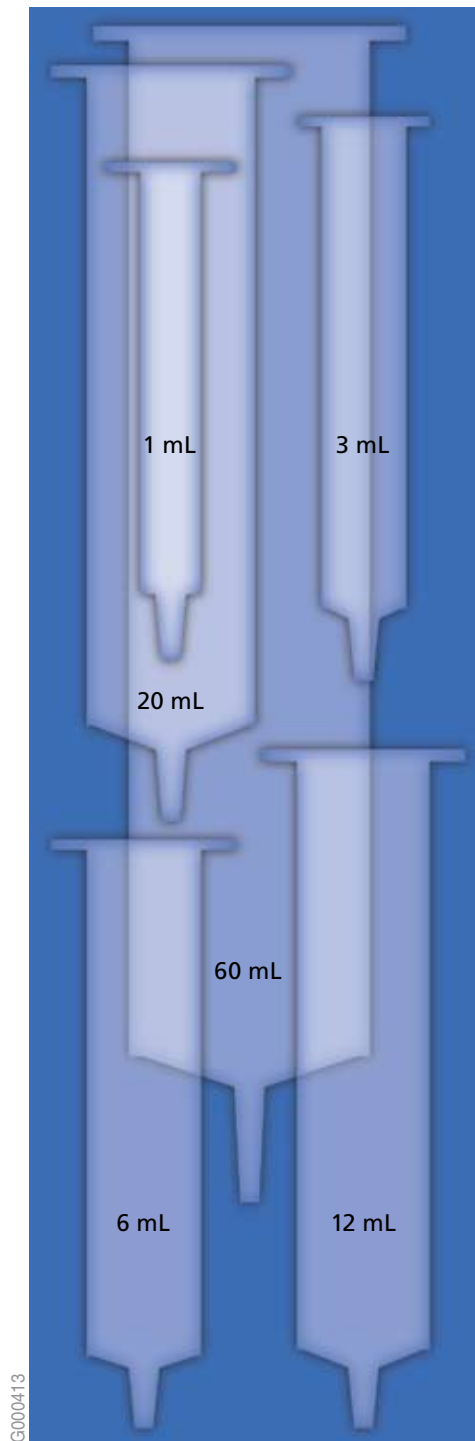
General guidelines for choosing the appropriate SPE tube size and bed weight configuration are listed in this table. Optimal method parameters and hardware/ bed weight dimensions should be determined during method optimization and troubleshooting.

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

* This value depends on the analyte and sample matrix. As a rule of thumb, the bed capacity can be estimated with ~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 1g) 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.

Most common SPE hardware:
Polypropylene SPE tubes with PE Frit



Actual size of SPE tubes

TRADEMARKS

AutoTrace - Caliper Technologies Corp.; Celite - Celite Corp.; Discovery, ENVI, ENVI-Carb, ENVI-Disk, Hisep, Preppy, Rezorian, Sigma-Aldrich, Supelclean, Supelco, VersaPure, Visi-1, Visidry, Visiprep - Sigma-Aldrich Co.; DOWEX - Dow Chemical Co.; Florisil - U.S. Silica Company; Gilson SPE 215 - Gilson; Laboport - KNF Neuberger GmbH; Luer-Lock - Becton-Dickinson & Co.; Multi-Probe - Hewlett-Packard Corp.; Quadra 96 - TomTec, Inc.

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SPE Bed Weight

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SPE Tubes and Specialty Hardware Quick Look-Up Guide

Additional Tubes & Cartridges

Glass SPE Tubes and PTFE & SS Frits (pg. 17-18)



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

VersaPure® PrePacked Büchner Funnels (pg. 15)



Used for large scale purification (medicinal chemistry purification)

Reversible SPE Tubes (pg. 17-18)



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

Rezorian Cartridges (pg. 17-18)



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

Discovery SPE 96-Well Plates (pg. 15)



For high throughput sample preparation

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, please inquire.

To request a price quote or inquire on the feasibility of Supelco manufacturing a custom SPE product, please contact our Order Processing & Technical Service representatives:

Flangeless SPE Tubes (custom - inquire)



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

Dispersive SPE (custom - inquire)



Custom pre-weighed salt sorbent vials for dispersive SPE

Order Processing: Phone: 800-247-6628/814-359-3441

Fax: 800-447-3044/814-359-5459

email: supelco@sial.com

Technical Service: Phone: 800-359-3041/814-359-3041

Fax: 800-359-3044/814-359-5468

email: techservice@sial.com

For US only. All other countries, please contact your local Sigma-Aldrich office or distributor.

SPE Accessories

Quick Look-Up Guide

Visiprep™ DL Vacuum Manifold
(pg. 19)



Uses disposable liners that prevent cross-contamination

Visiprep Standard Vacuum Manifold
(pg. 19)



Basic model

Preppy™ Vacuum Manifold
(pg. 19)



Most economical

PlatePrep Vacuum Manifold
(pg. 22)



For 96-well SPE

ENVI-Disk™ Holder
(pg. 23)



Used with 47 mm SPE disks

Trap Kit and Vacuum Gauge Bleed Valve
(pg. 21)



Additional vacuum accessories

Visi-1™ Single SPE Tube Processor
(pg. 19)



For processing very few SPE samples

SPE Tube Adapters
(pg. 20)



Useful for stacking SPE tubes, or processing SPE tubes via luer syringe

SPE Elution Rack
(pg. 20)



Simple racks for using SPE under gravity flow

KNF Laboport® Vacuum Pumps (inquire)



Provides vacuum source for vacuum manifolds

Visiprep™ Large Volume Sampler
(pg. 20)



For processing larger sample volumes

Visidry™ Drying Attachment
(pg. 20)



For drying SPE tubes or evaporating SPE eluate

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SPE Accessories

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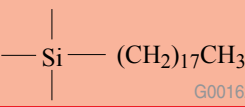
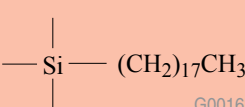
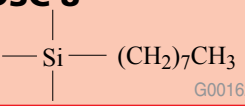
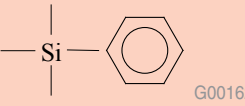
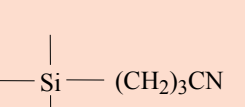

Discovery SPE

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 extrac-

tion, isolation, and concentration of pharmaceuticals from biological fluids and other aqueous sample matrices.

For Discovery silica specifications, see pg. 2. For general guidelines on reversed-phase SPE, see pg. 24.

DSC-18  G001625	<ul style="list-style-type: none"> • 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  G001633	<ul style="list-style-type: none"> • 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  G001624	<ul style="list-style-type: none"> • 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  G001628	<ul style="list-style-type: none"> • 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  G001626	<ul style="list-style-type: none"> • 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  G001195	<ul style="list-style-type: none"> • Polyamide Resin: Particle Size: 50-160 µm, Surf pH: 4.5-7.5, Density: 0.2-0.3 cm³/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./Pk	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	⁴ 52625-U
500 mg/6 mL	30	52604-U	52615-U	52714-U	52728-U	52696-U	⁵ 52626-U
1 g/6 mL	30	52606-U	52616-U	52716-U	52731-U	52697-U	⁶ 52627-U
2 g/12 mL	20	52607-U	52618-U	52717-U	Custom	52698-U	⁷ 52629-U
5 g/20 mL	20	52608-U	52621-U	52718-U	Custom	52699-U	⁸ 52631-U
10 g/60 mL	16	52609-U	52622-U	52722-U	Custom	52700-U	⁹ 52632-U
Bulk packing	100 g	52600-U	52623-U	52723-U	52727-U	52722-U	¹⁰ 52633-U
Discovery SPE 96-Well Plates							
100 mg/well	1	575603-U	575606-U	575627-U	575630-U	575624-U	Custom
50 mg/well	1	575602-U	575605-U	575628-U	575631-U	575625-U	Custom
25 mg/well	1	575601-U	575604-U	575629-U	575632-U	575626-U	Custom

⁴ 250mg/3mL, ⁵ 250mg/6mL, ⁶ 500mg/6mL, ⁷ 1g/12mL, ⁸ 2g/20mL, ⁹ 5g/60mL, ¹⁰ 50g

For a complete list of available Discovery Büchner funnels, see page 15.

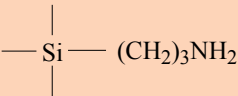
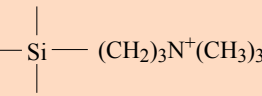
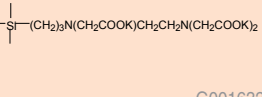
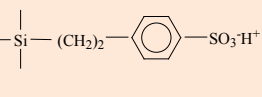
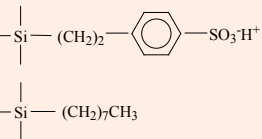
Discovery SPE

Ion-Exchange & 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 pg. 2. For general guidelines on ion-exchange & mixed-mode SPE, see pg. 25.

DSC-NH₂  G001631	<ul style="list-style-type: none"> • 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 pKa 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  G001629	<ul style="list-style-type: none"> • 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 conditioning
DSC-WCX  G001632	<ul style="list-style-type: none"> • A polymerically bonded carboxy propyl phase with a Na counter ion and a pKa 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 pKa) compounds that may be irreversibly retained on strong cation exchangers
DSC-SCX  G001630	<ul style="list-style-type: none"> • A polymerically bonded, benzene sulfonic acid functional group with a H+ counter ion that is a strong cation exchanger due to its very low pKa (<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  G001630, G001624	<ul style="list-style-type: none"> • Packed bed contains both octyl (C8) and benzene sulfonic acid (SCX) bondings • 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./Pk	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
500 mg/6 mL	30	52638-U	52665-U	52742-U	52688-U	52784-U
1 g/6 mL	30	52640-U	52666-U	52743-U	52689-U	52786-U
2 g/12 mL	20	52641-U	52667-U	52744-U	52690-U	52788-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	-
Bulk packing	100 g	57212-U	57214-U	57228-U	57221-U	-
Discovery SPE 96-Well Plates						
100 mg/well	1	575615-U	575618-U	575633-U	575621-U	575641-U
50 mg/well	1	575616-U	575619-U	575634-U	575622-U	575640-U
25 mg/well	1	575617-U	575620-U	575635-U	575623-U	575639-U

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Discovery SPE

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 pg. 2. For general guidelines on normal-phase SPE, see pg. 26.

DSC-Si —Si—OH	<ul style="list-style-type: none"> 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 $\text{—Si—(CH}_2\text{)}_3\text{CH}_2\text{CH—CH}_2$ OH OH G001627	<ul style="list-style-type: none"> 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 $\text{—Si—(CH}_2\text{)}_3\text{CN}$ G001626	<ul style="list-style-type: none"> 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-NH₂ $\text{—Si—(CH}_2\text{)}_3\text{NH}_2$ G001631	<ul style="list-style-type: none"> 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 pKa 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./Pk	DSC-Si	DSC-Diol	DSC-CN	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
Bulk packing	100 g	57222-U	52651-U	57229-U	57212-U
Discovery SPE 96-Well Plates					
100 mg/well	1	575624-U	575609-U	575636-U	575615-U
50 mg/well	1	575625-U	575608-U	575637-U	575616-U
25 mg/well	1	575626-U	575607-U	575638-U	575617-U
Discovery Büchner Funnels					
12.5 g, 55 mm ID x 30 mm H	6	Custom	52591-U	Custom	Custom
25 g, 70 mm ID x 40 mm H	6	Custom	52592-U	Custom	Custom
50 g, 90 mm ID x 48 mm H	6	Custom	52593-U	Custom	Custom
100 g, 110 mm ID x 66 mm H	6	Custom	52594-U	Custom	Custom

For a complete list of available Büchner funnels, see page 15.

Supelclean & Supelclean ENVI

Reversed-Phase

The Supelclean SPE line represents one of our original brands and 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 pg. 2. For general guidelines on reversed-phase SPE, see pg. 24.

ENVI-18	<ul style="list-style-type: none"> • Polymerically bonded, octadecyl (17% C), endcapped • Excellent for cleaning, extracting & 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 & ENVI-8 DSK SPE Disks	<ul style="list-style-type: none"> • 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
LC-18	<ul style="list-style-type: none"> • Monomerically bonded, octadecyl (10% C), endcapped • For reversed-phase extraction of nonpolar to moderately polar compounds.
ENVI-8	<ul style="list-style-type: none"> • 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 & concentrating pollutants from aqueous environmental samples
LC-8	<ul style="list-style-type: none"> • Monomerically bonded, octyl (7% C), endcapped
ENVI-Chrom P (polystyrene divinylbenzene)	<ul style="list-style-type: none"> • Styrene/divinylbenzene co-polymer resin: Particle Size: 80-160 μm; Spherical Shape; Pore Size: 110-175 \AA; 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 (graphitized carbon black)	<ul style="list-style-type: none"> • Surface Area: 100 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
LC-4 (Wide Pore)	<ul style="list-style-type: none"> • Butyldimethyl, wide pore (500 \AA), endcapped • Larger pore size to accommodate larger macromolecules (e.g., proteins and peptides) • Commonly used for desalting proteins and peptides in aqueous samples
Hisep™	<ul style="list-style-type: none"> • Hydrophobic sites shielded by a hydrophilic surface for protein exclusion during sample load • Hydrophobicity similar to C8
LC-Ph	<ul style="list-style-type: none"> • Monomerically bonded, phenyl (5.5% C), endcapped
LC-CN	<ul style="list-style-type: none"> • Monomerically bonded, cyanopropyl (7.5% C), endcapped

For part numbers, please see page 13

The Complete Line of SPE Products

technical service: 800-359-3041 (US and Canada only) / 814-359-3041

ordering: 800-247-6628 (US only) / 814-359-3441

Supelclean

SUPELCO

Supelclean & Supelclean ENVI

Ion-Exchange & 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 & beverage industry. The Supelclean ENVI reversed-phase 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, please see pg. 2. For general guidelines on ion-exchange & normal-phase SPE, see pgs. 25 & 26.

LC-SAX	<ul style="list-style-type: none"> • A strong anion exchanger with pKa of 10.1 and 10.9 • Quarternary amine, Cl⁻ counter-ion
LC-SCX	<ul style="list-style-type: none"> • Aliphatic sulfonic acid, Na⁺ counter-ion, endcapped
LC-WCX	<ul style="list-style-type: none"> • Carboxylic acid, Na⁺ counter-ion
LC-NH₂	<ul style="list-style-type: none"> • Monomerically bonded, aminopropyl (5% C)
PSA	<ul style="list-style-type: none"> • Polymerically bonded, ethylenediamine-N-propyl phase that contains both primary and secondary amines
ENVI-Florisil	<ul style="list-style-type: none"> • 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/Na₂SO₄	<ul style="list-style-type: none"> • Dual layer glass SPE tube that contains Na₂SO₄ (upper layer) and Florisil (magnesium silicate; lower layer) separated and packed with PTFE frits • Florisil particle size- 60/100 mesh (150-200 μm), Na₂SO₄ Purity- 99.99 %, Density- 2.68 g/mL • Excellent for removing/isolating polar compounds from organic matrices • Na₂SO₄ 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 • Glass SPE hardware allows user to reactivate Florisil through heating at 140 °C, 16 hours • Use in conjunction with Visiprep Large Volume Sampler (Cat. No. 57275) and Visiprep SPE Vacuum Manifolds for processing larger volume samples
LC-Florisil	<ul style="list-style-type: none"> • Magnesium silicate, mesh: 100/120
LC-Alumina A LC-Alumina N LC-Alumina B	<ul style="list-style-type: none"> • Alumina-A for acidic pH (~5) • Alumina-N for neutral pH (~6.5) • Alumina-B for basic pH (~8.5) • Brockman Act. I for all Alumina SPE products, mesh: 60/325
LC-CN	<ul style="list-style-type: none"> • Monomerically bonded, cyanopropyl (7% C), endcapped
LC-Si	<ul style="list-style-type: none"> • Silica gel
LC-Diol	<ul style="list-style-type: none"> • Monomerically bonded, Diol (7% C), endcapped

For part numbers, please see page 13

Supelclean & 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 54331-U ¹	505706	57114	57137	57138	57219
	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 57106 ¹	57232 57107 ¹	57233		57139	57140-U	
	LC-8	504157	505145	57052					57201
	ENVI-Chrom P	57143	57224 ⁵	57226 57225-U ⁷					57217 ¹¹
	ENVI-Carb	57109-U	57088 ⁵	57094 57092 ⁷		57128 57127-U ¹⁰	57129	57130	57210-U ¹¹
	ENVI-Carb C, mesh 80/100					57149 ¹⁰			
	LC-4 (Wide Pore)		57089						
Normal-Phase	Hisep		57076-U						
	LC-Ph	504599	505269						
	LC-CN	504386	57013	57056			57141	57142	
	LC-Diol	50478	57016						
	ENVI-Florisil		57058 ²	57046 ³	57053 ³				
	Dual Layer Florisil/ Na ₂ SO ₄				52582-U ^{1,9} 54116-U ⁹				
	LC-Florisil			54333-U ¹	57057 54334-U ¹	57115	57131	57132	57209
	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
Ion Exch.	LC-Si	504041	505048	505374	57051 54335-U ¹	57116	57133	57134	57200
	LC-NH ₂	504483	57014	54059-U					57205
	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 pg. 16.

Multi-Layer SPE

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

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

Description	Cat. No.
ENVI-Carb/LC-NH₂	
0.5 g/0.5 g/3 mL, pk 20	54332-U
0.5 g/0.5 g/6 mL, pk 30	54035-U
ENVI-Carb/NH₂/Silica	
0.5 g/0.4 g/0.6 g/12 mL, pk 20	54034-U
0.5 g/0.4 g/0.6 g/20 mL, pk 20	54036-U
Dual Layer Florisil/Na₂SO₄	
glass tubes, PTFE frits, 2 g/2 g/6 mL, pk 48	52582-U
2 g/2 g/6 mL, pk 48	54116-U

technical service: 800-359-3041 (US and Canada only) / 814-359-3041

ordering: 800-247-6628 (US only) / 814-359-3441

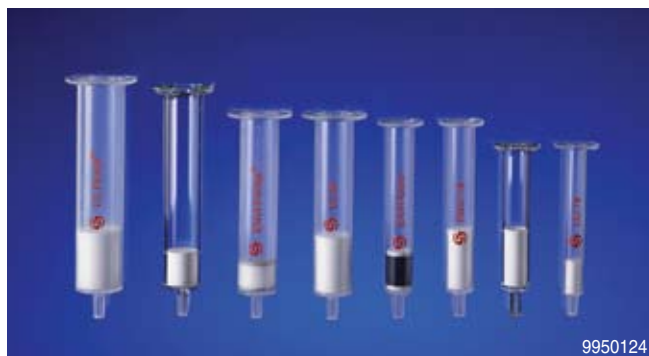
SUPELCO

Supelclean

SPE Method Development Kits

Supelclean SPE Method Development Kits

Supelclean SPE Method Development Kits consist of an assortment of SPE phase chemistries and cartridge configura-



tions 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.

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

- Discovery Reversed-Phase SPE MultiPaks
- Discovery Normal-Phase SPE MultiPaks
- Discovery Ion-Exchange SPE MultiPaks
- Discovery DSC-MCAX (Mixed-Mode Cation Exchange) SPE MultiPak
- Discovery DPA-6S (Polyamide) SPE MultiPak
- Supelclean ENVI-Carb (Graphitized Carbon) SPE MultiPak
- Discovery Ag-Ion SPE MultiPak
- Dual Layer SPE MultiPak
- Supelclean PSA SPE MultiPak

To learn more about SPE MultiPaks, or to request a free SPE MultiPak sample, please contact Technical Service at 800-359-3041/814-359-3041.

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	500 mg/6 mL 1 g/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. Ea. Tube	6	12	3	6	12
Cat. No.	57019	57009-U	57075-U	57074-U	57073

Specialty Products for Pharmaceutical Analysis

VersaPure Prepacked, Disposable Büchner Funnels

VersaPure Büchner funnels offer the convenience and capacity necessary to purify and/or filter larger scale samples and reaction mixtures. Researchers have used VersaPure Büchner Funnels for a variety of applications including: the purification of organic synthesis reactions, isolation of actives from natural products, filtration, removal of residual moisture from solvents, and more. The funnel consists of a solvent resistant two-piece semi-translucent polypropylene body. The packed bed is sandwiched between two PE frits (20 µm porosity) that are compressed in place by a heat riveted retaining ring to eliminate loose frits and minimize channeling. A 0.7 µm glass fiber membrane is placed below the bottom frit to capture any residual fines.



VersaPure Büchner Funnel

	12.5 g (45 mL) 55 mm I.D. x 30 mm H pk 6	25 g (90 mL) 70 mm I.D. x 40 mm H pk 6	50 g (174 mL) 90 mm I.D. x 48 mm H pk 3	100 g (410 mL) 110 mm I.D. x 66 mm H pk 3
Discovery DSC-Si	52591-U	52592-U	52593-U	52594-U
Merck-Si	2026-U	2027-U	2028-U	2029-U
Charcoal	2031-U	2032-U	2033-U	2034-U
Magnesium Sulfate	2037-U	2041-U	2043-U	2044-U
Celite®	2047-U	2048-U	2049-U	2064-U
Florisil	2074-U	2076-U	2077-U	2078-U
Alumina-A	2084-U	2087-U	2088-U	2089-U
Alumina-N	2091-U	2092-U	2093-U	2094-U
Alumina-B	2096-U	2097-U	2098-U	2099-U
Discovery DPA-6S	2079-U	2081-U	2082-U	2083-U (pk. 1) 52634-U
Empty Büchner Funnel	2141-U	2142-U	2143-U	2144-U

Discovery SPE 96-Well Plates



Discovery 96-well plates answer the challenge of high throughput pharmaceutical screening and analysis. The uniform flow dynamics inherent with well plate technology offers a higher level of reproducibility and throughput while maintaining excellent recoveries and increased sensitivity. These plates are packed with the same high quality phases used in our Discovery SPE line.

96-Well Plate Specifications:

- One-piece polypropylene square well design
- 2 mL sample volume
- Polyethylene frit, 20 µm porosity
- Compatible with TomTec Quadra 96®, Packard Multi-Probe®, Gilson SPE 215™, and most other 96-well automated SPE systems

Discovery 96-Well SPE Plate

Phase	25 mg/well	50 mg/well	100 mg/well
DSC-18	575601-U	575602-U	575603-U
DSC-18Lt	575604-U	575605-U	575606-U
DSC-MCAX	575639-U	575640-U	575641-U
DSC-8	—	575628-U	575627-U
DSC-Ph	575632-U	575631-U	575630-U
DSC-CN	575626-U	575625-U	575624-U
DSC-PS/DVB	575610-U	575611-U	—
DSC-Si	575607-U	575608-U	575642-U ¹ 575609-U
DSC-Diol	575638-U	575637-U	575636-U
DSC-NH ₂	575617-U	575616-U	575615-U
DSC-SAX	575620-U	575619-U	575618-U
DSC-WCX	575635-U	575634-U	575633-U
DSC-SCX	575623-U	575622-U	575621-U

¹ PE bottom frit (5 µm porosity)

technical service: 800-359-3041 (US and Canada only) / 814-359-3041


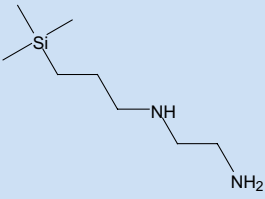
ordering: 800-247-6628 (US only) / 814-359-3441

Specialty Products for Pesticide Analysis

The Supelclean ENVI-Carb-II/PSA SPE product line consists of multi-layer SPE cartridges that were developed for superior cleanup when conducting multi-residue pesticide analysis in agricultural products (fruits, vegetables, meat, shellfish, grains, and dairy products). The technology acts as

a chemical filter in which each layer plays a specific role for removing key interferences.

Note that dual layer ENVI-Carb/NH₂ SPE products are also available.

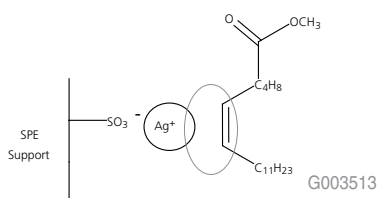
ENVI-Carb-II/PSA  G002462	<ul style="list-style-type: none"> Dual layer SPE tube that contains both Supelclean ENVI-Carb-II (upper layer) and PSA (lower layer) SPE sorbents (separated by PE frit) Developed to offer superior cleanup when conducting multi-residue pesticide analysis in food (e.g., fruits, vegetables, etc.) ENVI-Carb-II is a graphitized non-porous carbon (100/140 mesh, surface area 100 m²/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	<ul style="list-style-type: none"> 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 m²/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	<ul style="list-style-type: none"> 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	<ul style="list-style-type: none"> Surface Area: 100 m²/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  G002460	<ul style="list-style-type: none"> Polymerically bonded, ethylenediamine-N-propyl phase that contains both primary and secondary amines A weak anion exchanger with a pKa of 10.1 and 10.9 Similar to aminopropyl SPE phases (NH₂) 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

For part numbers, please see page 13

Miscellaneous Specialty Products

SPE Tubes and Cartridges

Discovery Ag-Ion SPE Tubes for FAME Analysis



Retention Mechanism: Normal-phase

Sample Matrix Compatibility: Organic solvents, oils, and lipids

- Each lot is tested and quality controlled for cis/trans FAME resolution
- 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.

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 & 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/Na₂SO₄ SPE Tube		
bed A 2 g (Florisil), bed B 2 g (Na ₂ SO ₄), vol. 6 mL	48	52582-U

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	54037-U

ordering: 800-247-6628 (US only) / 814-359-3441 technical service: 800-359-3041 (US and Canada only) / 814-359-3041

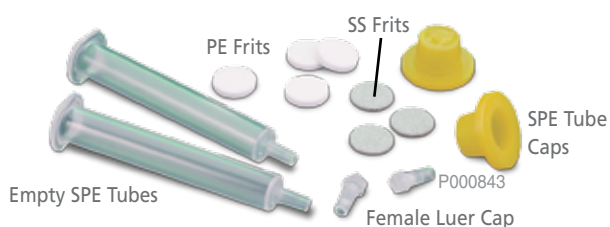
Specialty Products

SUPELCO

The Complete Line of SPE Products

SPE Accessories

Empty SPE Hardware & Components



SPE Tube Components

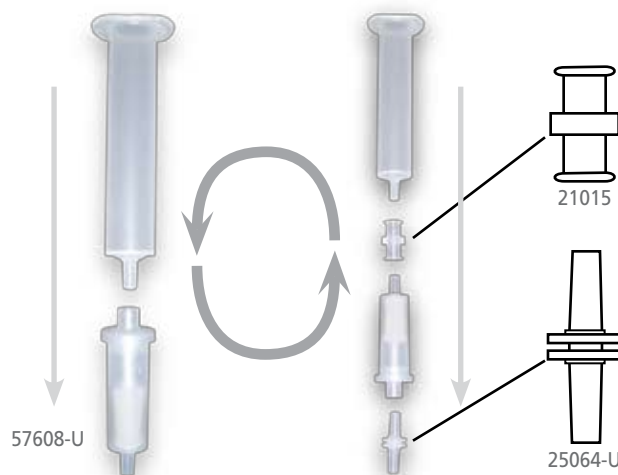
Description		1 mL	3 mL	6 mL	12 mL	20 mL	60 mL
Empty SPE Tubes with and without Frits	Qty.:	108	54	30	20	20	16
Empty PP SPE Tube with PE Frits, 20 µm porosity		57023	57024	57026	57176	57177	57178
Empty PP SPE Tube with PE Frits, 20 µm porosity – pre-fritted with bottom frit		Inquire	Inquire	Inquire	Inquire	57118-U	57119-U
Empty PP SPE Tube (no frits)		57240-U	57241	57242	57179	57021 (Qty. 12)	57022 (Qty. 12)
Empty Glass SPE Tubes with PTFE Frits, 20 µm porosity		–	–	504394 (Qty. 24)	–	–	–
SPE Tube Caps (encloses top of SPE tubes)	Qty.:	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 (Qty. 24)	–	–	–
Frits for use with SPE tubes	Qty.:	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	57189	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 & 20/60 mL tubes)							55226-U

PP = Polypropylene; PTFE = Polytetrafluoroethylene; SS = Stainless steel; PE = Polyethylene

Miscellaneous SPE Hardware & Accessories

Description	Qty.	Cat. No.
Empty Reversible SPE Tube, non-fluorous 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
Empty PP Disposable Büchner Funnels w/PE Frits		
55 mm I.D. x 30 mm H, 75 mL	6	2141-U
70 mm I.D. x 40 mm H, 165 mL	6	2142-U
90 mm I.D. x 48 mm H, 315 mL	6	2143-U
110 mm I.D. x 66 mm H, 875 mL	3	2144-U

Description	Qty.	Cat. No.
Luer Caps, Plugs, and Couplers		
Female Luer Cap, PP (caps SPE luer tips)	12	57098
Male Luer Plug, PP (plugs SPE luer tips)	12	504351
Female Luer Coupler	20	21015
Male Luer Coupler	20	25064-U



SPE Accessories

Vacuum Manifolds

Visiprep & Visiprep DL SPE

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. The liner consists of a PP luer hub that attaches to the SPE tube, and 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 & Benefits for both DL and standard models:

- Patented 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 and vacuum control. Valve takes 1/4" vacuum tubing.
- 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)

P000129

Supelco Preppy Vacuum Manifold

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

Two optional collection racks are available; one holds both 1 and 4 mL autosampler vials, and the other holds 15 (with 21 mm O.D.) or 40 (with 28 mm O.D.) mL vials. An optional vacuum gauge/bleed valve assembly can be installed to allow precise control of the vacuum used with the Preppy manifold.

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 1 or 4 mL vials	57159-U
For 15 or 20 mL vials	57162-U
Accessories	
Vacuum Gauge/Bleed Valve Assembly	57161-U



P000128

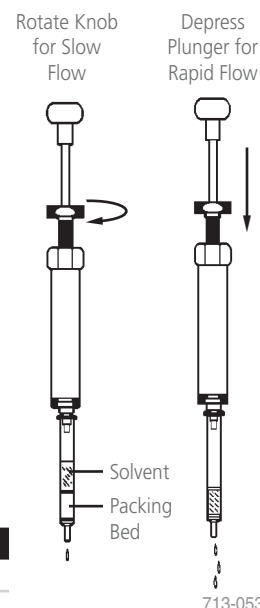
Single SPE Tube Processor

Visi-1 processor provides 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



713-0539

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ordering: 800-247-6628 (US only) / 814-359-3441

SPE Accessories

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SPE Processing Accessories

Visidry Drying Attachment

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

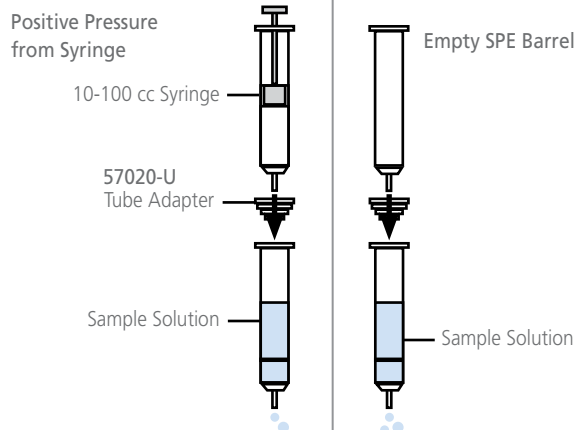


NOTE: The Visidry drying attachment cannot be used to dry 12 mL, 20 mL, or 60 mL SPE tubes.

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 below.

Tube Adapters



Tube adapters serve many purposes. They can be used to 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. Or, they can serve as an adapter for positive pressure methods (e.g. from a syringe or air/N₂ 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

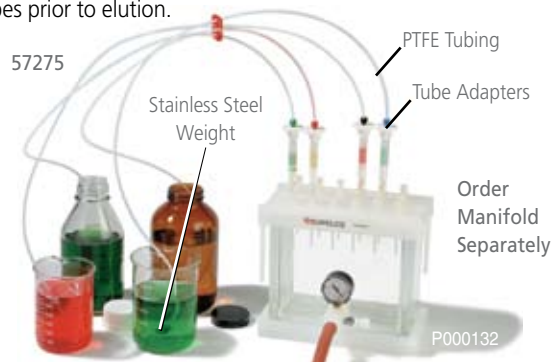
* Allows SPE tubes to be used with AutoTrace® Automated Systems

Description	Qty.	Cat. No.
SPE Tube Adapter for Glass Tubes		
PTFE, for use with 6 mL glass SPE Tube	24	504335

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.

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

¹Also, you must equip alternate manifold valves with long stem flow control knobs to accommodate 12 mL, 20 mL, or 60 mL SPE tubes.

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 & Accessories

Replacement Parts and Optional Components for Visiprep Manifolds

Description	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 (pk. of 2)	57033
Glass basin	57049
Glass basin, vacuum gauge and bleed valve ³	57034
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 (pk. of 2)	57254
Glass basin	57253
Glass basin, vacuum gauge and bleed valve ⁶	57252
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 (pk. of 24)	57146-U
Valve Stem for Visiprep/Preppy Vacuum Manifold (pk. of 24)	57147-U
Flow control valves (pk. of 2) ⁷	57032
Solvent guide needles, PTFE (pk. of 12) ^{1,8}	57047
Solvent guide needles, stainless steel (pk. of 12) ⁷	57036
Disposable liner flow control valves (pk. of 2) ⁹	57028
Liner guide needles, stainless steel (pk. of 12) ^{2,10}	57027
Disposable valve liners, PTFE (pk. of 100) ^{2,5}	57059
Vacuum gauge and bleed valve	57035-U
Retaining clips for collection racks (pk. of 12)	57041
Test tubes, 10 x 75 mm (pk. of 12) ^{1,2,8,10}	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.

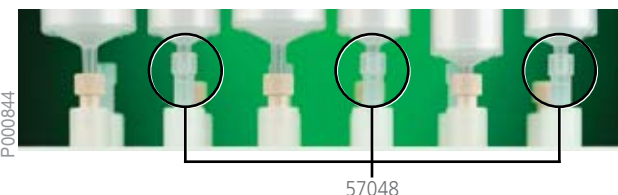


Description	Cat. No.
Vacuum Gauge / Bleed Valve Assembly	57161-U

Long Stem Flow Control Valves for Visiprep Manifolds

Equip alternate valves in your 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

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SPE Accessories

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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 Includes:

- 1 PlatePrep Vacuum Manifold
- 1 96 Sq. Well Collection Plate, 2 mL, PP
- 2 Disposable Reservoir/Waste Trays, PVC
- 1 96 Sq. Well Piercable Cap Mat
- 5 Reagent Reservoirs
- 1 Cluster Tube Rack

Description	Qty.	Cat. No.
Supelco PlatePrep Vacuum Manifold	1	57192-U
96-Well Plate Starter Kit with PlatePrep Manifold	1	575650-U

PlatePrep Vacuum Manifold Replacement Parts

Acrylic Clear Top	1	57193-U
Polypropylene Base	1	57194-U
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



96-well SPE Plate

Acrylic
Clear Top96 Square Well
Collection Plate or
Disposable Reservoir/
Waste Tray

Polypropylene Base



P000845

SPE Accessories

ENVI-Disk Accessories

ENVI-Disk Holder

Use the ENVI-Disk Holder with 47 mm ENVI-DSK SPE disks. 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/adaptor 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 47mm 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



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SPE Accessories

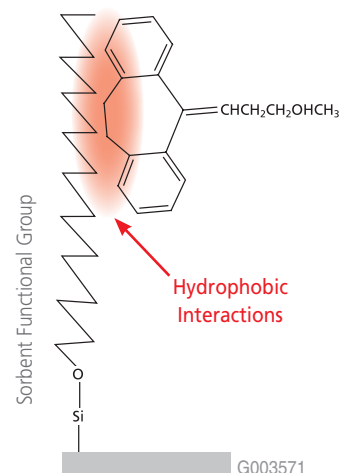
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SPE Methodology & Useful Tips

Reversed-Phase SPE

- 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

Aqueous Sample Matrix/Mobile Phase Environment



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.

Basic Steps

- 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 pKa (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.

- 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.

- Sample Load** Apply sample (from step 1) at a consistent and reduced flow rate of ~1-2 drops/second to ensure optimal retention.

- 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.

- 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.

- Eluate Post-treatment** It 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:

- Drug-protein binding should be disrupted during sample pre-treatment.

Strategies include:

- 40 µL 2% disodium EDTA per 100 µL mouse plasma
- 40 µL 2% formic acid per 100 µL mouse plasma
- Other possible reagents (per 100 µL matrix): 40 µL 2% TCA, 40 µL 2% acetic acid, 40 µL 2% TFA, 40 µL 2% phosphoric acid, or 200 µL MeCN (protein ppt.).

- 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.

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

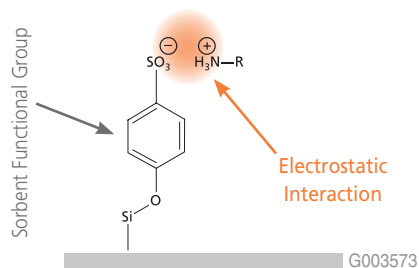
- Reduce bed weight to minimize elution volume.

- Increase bed weight to retain more polar compounds.

- Concern for sorbent over drying is only critical during methanol conditioning.

- 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 & Useful Tips



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 pKa. For acidic analytes, the pH should be adjusted to at least 2 pH units above the molecule's pKa.

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 pKa, 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.

Counter Ion Selectivity & Ion Exchange:

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

For Cation Exchangers:

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

For Anion Exchangers:

- Benzene Sulphonate $>$ Citrate $>$ $\text{HSO}_4^- > \text{NO}_3^- > \text{HSO}_3^- > \text{NO}_2^- > \text{Cl}^- > \text{HCO}_3^- > \text{HPO}_4^{2-} >$ Formate $>$ Acetate $>$ Propionate $>$ $\text{F}^- > \text{OH}^-$

To change to a higher selective ion, pass 2-5 bed volumes of 1N solution of the new counter ion through sorbent. To change to a lower selective ion, pass 5-65 bed volumes of 1N solution of the new counter ion through sorbent.

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

Ion-Exchange & 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 ($< 0.1\text{M}$)
- 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 ($> 1\text{M}$); 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

Basic Steps

- Sample Pre-treatment** Salt concentration should be less than 0.1M. 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.

- 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 sample pre-treatment.

- 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.

- 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.

- 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 ($> 1\text{M}$)
- Use a more selective counter-ion to compete for ion-exchange binding sites

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

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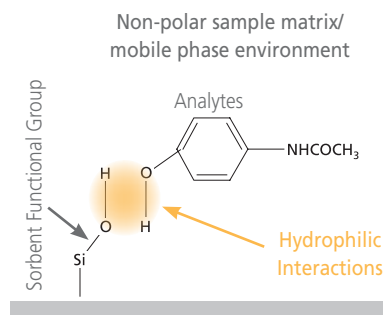
General Guidelines

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SPE Methodology & Useful Tips

Normal-Phase SPE

- 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



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.

Basic Steps

- 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.
- Condition/Equilibration** Condition and equilibrate with 2-3 tube volumes of a non-polar solvent similar or identical to sample matrix resulting from sample pre-treatment.
- Sample Load** Apply sample (from step 1) at a consistent and reduced flow rate of ~1-2 drops/second to ensure optimal retention. The compounds should be in a non-polar solvent (e.g., hexane) for optimal retention. Note that methanol and acetonitrile are often used as elution solvents in normal-phase SPE, and will often not promote compound retention during sample load.
- 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.
- 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.
- Eluate Post-treatment** Normal-phase SPE is often followed by GC analysis, and therefore requires a volatile sample matrix prior to injection. Use sodium sulfate or magnesium sample to remove residual moisture. Further SPE eluate concentration may also be necessary prior to analysis.

Common Normal-Phase Solvents:

Solvent	Elutropic (e°) or elution strength on silica	
Hexane	0.00	Promotes Normal-Phase Retention
Isooctane	0.00	
Carbon tetrachloride	0.14	
Toluene	0.22	
Benzene	0.27	
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	Promotes Normal-Phase Elution
Acetone	0.45	
Acetonitrile	0.50	
40% methanol in acetonitrile	0.67	
20% methanol in diethyl ether	0.65	
20% methanol in methylene chloride	0.63	
Isopropanol	0.63	
Methanol	0.73	
Water	>0.73	
Acetic acid	>0.73	

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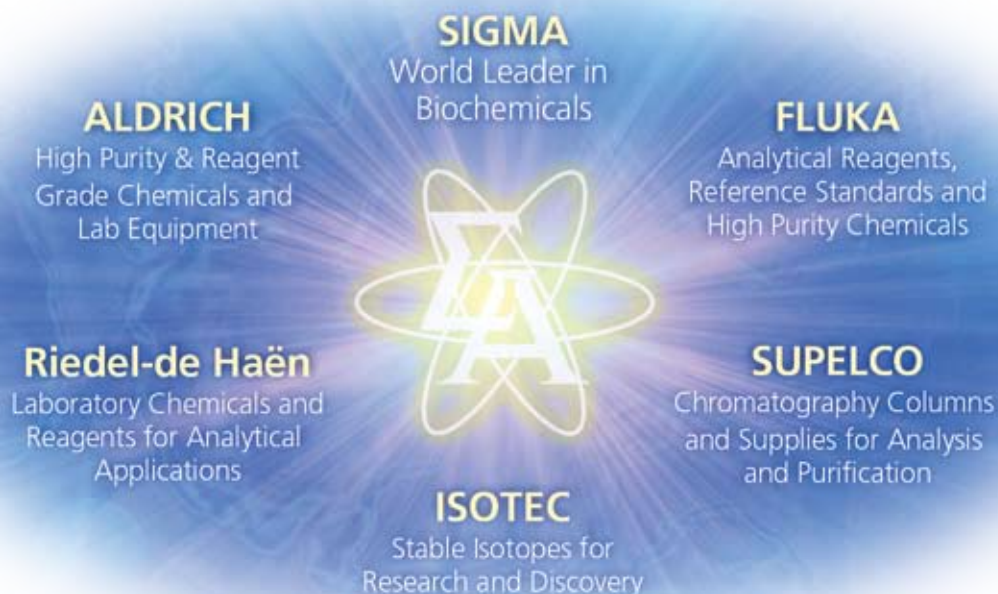
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