SPE Method Development

SPE method development typically contains four steps:

Step 1: Condition

The conditioning step is composed of two substeps; the first activates the sorbent ligands, the second equilibrates the sorbent bed.

Step 2: Load

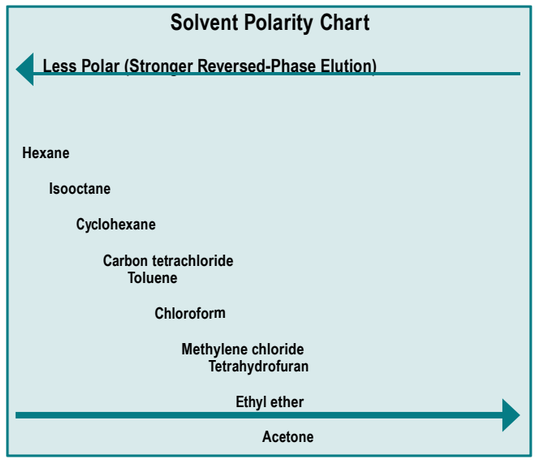
In the load step, sample is applied to the SPE device. Matrix and flow rate are optimized to quantitatively retain target analytes.

Step 3: Wash

In the wash step, choose a solvent that elutes impurities but retains target analytes. Often the second conditioning solvent is a suitable wash solvent.

Step 4: Elute

The elution step ideally removes all target analytes with minimal solvent to maximize sensitivity. Sometimes requires a combination of solvents to break both the primary and secondary interactions.



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	General Method Development Procedures			
	Step 1—Condition ~ 4 bed volumes	Step 2—Load	Step 3—Wash ~ 6 bed volumes	Step 4—Elute ~ 3 bed volumes
Reversed-Phase Extraction Procedure **Mechanism:** Bind moderately polar to non-polar compounds from a polar sample matrix.	Methanol followed by water	Process sample at a flow rate of 1–5mL/min	Water or water:methanol (95:5)	Methanol or acetonitrile. May need to add strong acid or base to organic solvent to break secondary interactions.
Normal Phase Extraction Procedure **Mechanism:** Bind polar compounds from a non-polar sample matrix.	IPA followed by hexane	Process sample at a flow rate of 1–5mL/min	Hexane or hexane:IPA (98:2)	IPA, ethyl acetate, acetone, or hexane:IPA (50:50)
Ion-Exchange Extraction Procedure **Mechanism:** Bind charged (negative/anionic or positive/cationic) compounds.	Methanol:water (50:50) followed by low ionic strength (0.1M) buffer	Apply slowly: less than or equal to 1mL/min ion exchange kinetics are slower than reversed- or normal phase	Methanol:low ionic strength (0.1M) buffer (10:90)	High ionic strength (0.5M–1.0M) buffer or modify pH such that the analyte is uncharged. May need to add organic to break hydrophobic interactions.

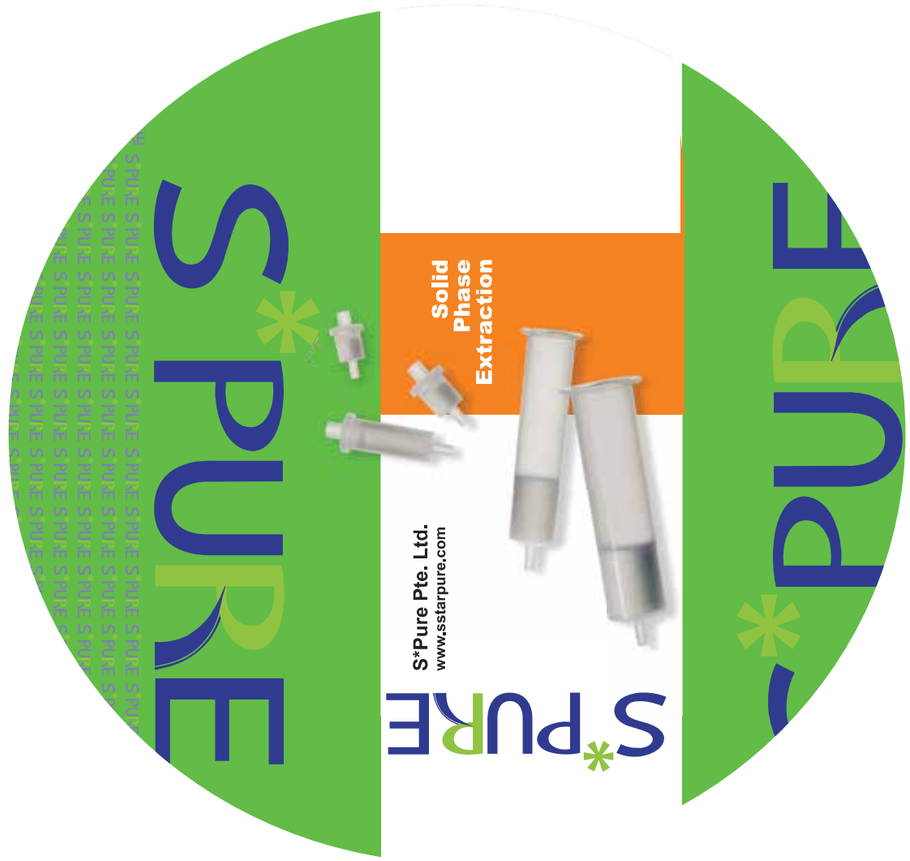
tech tips

- To calculate sorbent bed volume, use 150µL for every 100mg of sorbent.
- Retention capacity describes the total amount that an SPE sorbent will bind. This includes all compounds retained—analytes of interest as well as the contaminants.
- The minimum elution volume recommended in the bed size chart above will offer best sensitivity, but more solvent may be required depending on the application

tech tip

FOR MORE INFORMATION CONTACT

Solid Phase Extraction Essentials



www.sstarpure.com

SPE Introduction

Incorporating the highest grade of silica in the industry with over a quarter of a century's experience in making SPEs. S*Pure brings to you a highly comprehensive range of silica-based SPE products. This includes MaxiClean™, Ultra-Clean™, Extract-Clean™, Vydac®; brands synonymous with quality, reproducibility and highest recoveries.

Working with Experts in Media Production
Using a consistent and pure silica base that employs tightly controlled bonding techniques, insures predictable analyte-sorbent interactions, critical in ensuring a bonded phase with high and reproducible recoveries.

Highest Quality Control
Every part of our manufacturing process is carefully monitored. From managing our raw materials to stringent quality controls in the final product, we perform multiple quality tests, and provide a comprehensive quality assurance certificate.

Extract-Clean™ Columns

Format: SPE Columns
Sizes: 1.5, 4, 8, 15, 25, 75mL (the entire tube volume)
Summary: In production for over 25 years, with proven consistency, this is our most comprehensive SPE product line. It includes 30 media types in over 10 different bed weights. And with a complete offering of reversed normal, and specialty medias exhibiting unique retention properties, you are sure to find the packing that delivers a cleaner, more concentrated sample

Maxi-Clean™ Cartridges

Format: SPE Cartridges
Sizes: 300, 600, 900mg (media amount, not device volume)
Summary: The Maxi-Clean™ line is offered in many of the same media as the Extract-Clean™ line, but slightly paired down, with over 20 chemistries available. This lure hub cartridge device is not as prevalent in the SPE industry, and while manual processing is most common, this format offers a number of other interesting processing options, including multimedia extractions.

Ultra-Clean™ Columns

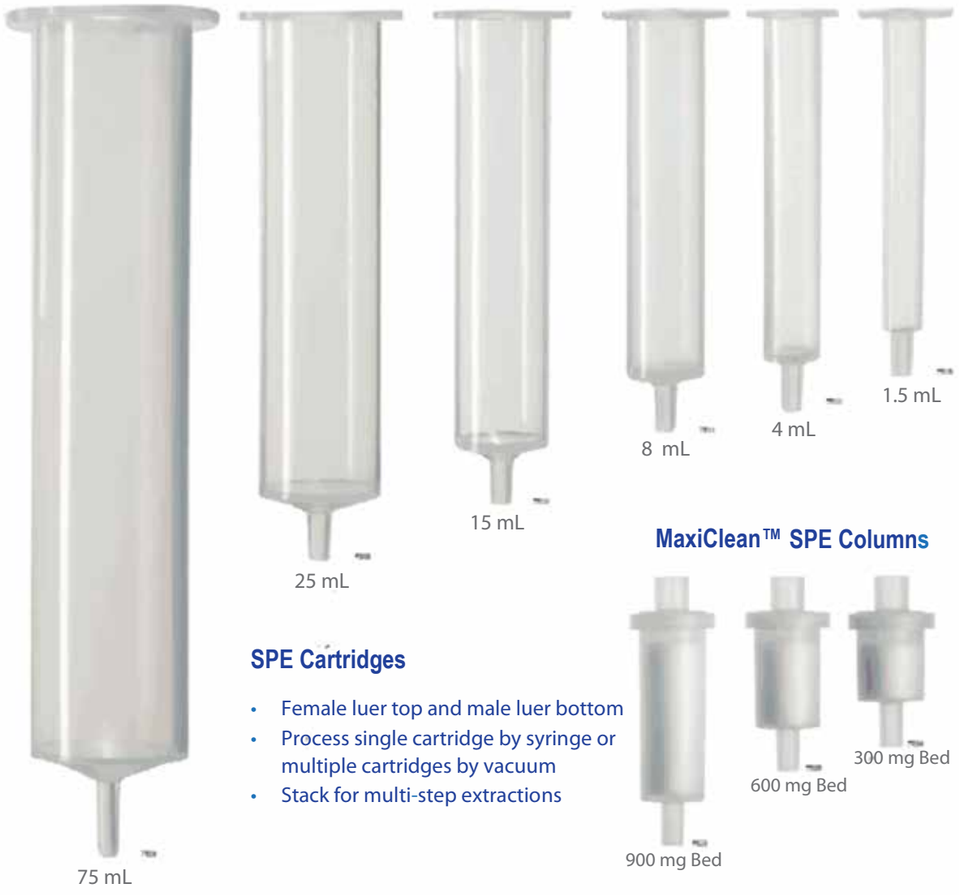
Format: SPE Columns
Sizes: 4, 8mL (the entire tube volume)
Summary: Choose this ultra-low extractable version for very sensitive applications. Nine selected media are packed into highly inert fluorinated polypropylene tubes with PTFE frits. Less expensive than glass extraction devices, this durable format offers comparable inertness without the added concern of being easily broken.

Vydac® Columns

Format: SPE Columns
Sizes: 1, 3mL (volume above the packing)
Summary: Ideal for extraction, concentration and cleanup of biological samples. This 300Å silica-based media has the same properties as the industry-leading Vydac® TP HPLC packing. Offered in C18 and C4, use for a variety of protein and peptide applications

Extract-Clean™ SPE Columns

- Open top tubes with male luer bottom
- Process multiple samples with vacuum manifold or automated SPE instruments
- Process individual samples manually with use of adapter and syringe



SPE Cartridges

- Female luer top and male luer bottom
- Process single cartridge by syringe or multiple cartridges by vacuum
- Stack for multi-step extractions

MaxiClean™ SPE Columns

Device Specifications

Device	Housing	Frit Material
Extract-Clean™ Columns	Polypropylene	20µm Polyethylene
Ultra-Clean™ Columns	Treated Polypropylene	10µm PTFE
Vydac® Columns	Polypropylene	Glass Fiber Filter Paper with Polyethylene Mesh Support
Maxi-Clean™ Cartridges	Polypropylene	20µm Polyethylene

Reversed-Phases (Non-Polar) Sorbent Specifications

Packing	Base	% Carbon	End-capped	Average Particle Size	Pore Size	Features	Benefits	Extract-Clean™	Ultra-Clean™	Maxi-Clean™
Prevail™ C18	Silica	11.0%	Yes	50µm	60Å	100% water wettable	Hydrophilic/hydrophobic retention. Phase remains active even when completely dry. Can omit preconditioning step.	x		x
Standard C18	Silica	6.0%	Yes	50µm	60Å	Low carbon load C18	General purpose phase.	x	x	x
High-Flow C18	Silica	8.0%	Yes	100µm	60Å	Large particle	Less flow resistance for faster flow rates of large volume sample.	x		
High-Capacity C18	Silica	17.0%	Yes	50µm	60Å	High carbon load	Maximum capacity phase.	x		x
Large Pore C18	Silica	14.0%	Yes	50µm	150Å	Larger than average pore size	Ideal for compounds >1500MW.	x		x
Octyl (C8)	Silica	4.5%	Yes	50µm	60Å	Less hydrophobic than C18	Less retention of highly hydrophobic compounds. Use when C18 is too retentive.	x	x	x
Ethyl (C2)	Silica	5.5%	Yes	50µm	60Å	Short chain functional group is less hydrophobic than C8	Less retention of highly hydrophobic compounds. Use when C8 is too retentive.	x		x
Phenyl (PH)	Silica	3.8%	Yes	50µm	60Å	Aromatic structure	Highly selective for aromatic compounds.	x		x

Normal-Phases (Polar) Sorbent Specifications

Packing	Base	% Carbon	End-capped	Average Particle Size	Pore Size	Features	Benefits	Extract-Clean™	Ultra-Clean™	Maxi-Clean™
Silica (SI)	Silica	—	—	50µm	60Å	Highly polar surface	Most common polar phase.	x	x	x
Aminopropyl (NH_2)	Silica	5.0%	No	50µm	60Å	Polar phase with slight anion exchange properties	Ideal for carbohydrates or generally with analyses containing hydroxyl functional groups.	x		x
Cyanopropyl (CN)	Silica	6.0%	Yes	50µm	60Å	Unique selectivity	Can be used in normal-phase or reversed-phase modes.	x	x	x
Diol (2OH)	Silica	4.0%	No	50µm	60Å	Polar surface with minor hydrophobic retention	Wets easily and offers more reproducibility.	x		x
Florisil® (FL)	Magnesium Silicate	—	—	75 – 150µm	60Å	Highly polar surface	Referenced in many EPA methods. Ideally suited for pesticides and metals.	x	x	x
Florisil® PR (FL-PR)	Magnesium Silicate	—	—	75 – 150µm	60Å	Specifically tested for chlorinated pesticides	Ensures most inert batches suitable for highly active compounds.	x	x	x
Alumina Acidic (AL-A)	Aluminum Oxide	—	—	130µm	100Å	Alumina washed with acid surface	Increase capacity for acidic compounds.	x		x
Alumina Basic (AL-B)	Aluminum Oxide	—	—	130µm	100Å	Alumina washed with base surface	Increase capacity for basic compounds.	x		x
Alumina Neutral (AL-N)	Aluminum Oxide	—	—	130µm	100Å	Alumina washed with neutral surface	Interacts with highly aromatic compounds and neutral hydroxyls.	x		x

Specialty Packings Specifications

Packing	Base	% Carbon	End-capped	Average Particle Size	Pore Size	Features	Benefits	Extract-Clean™	Ultra-Clean™	Maxi-Clean™
DVB	100% DVB	—	—	40µm	—	100% DVB	Greater capacity than C18 for general SPE. Also free vinyl surface groups make a suitable solid-phase synthesis support.	x		
Carbograph	Graphitized Carbon	—	—	38 – 125µm	—	Graphitized Carbon	Retains polar organics in aqueous matrices. Ideally suited for acid, base-neutral extraction of pesticides and herbicides.	x	x	
Drug-Clean SB-C	Silica	—	—	50µm	60Å	Silica-based mixed mode C8/cation exchange	Ideal for drugs of abuse.	x		
Drug-Clean SB-A	Silica	—	—	50µm	60Å	Silica-based mixed mode C8/anion exchange	Ideal for drugs of abuse.	x		
Drug-Clean PB	Polymer	—	—	30µm	—	Polymer-based mixed mode C8/cation exchange	pH stable with no conditioning required. Extract acidic, neutral and basic drugs of abuse from single column.	x		

General Ion-Exchange Sorbent Specifications

Packing	Base	Counter Ion	Particle Size	Functional Group	Exchange Capacity	Retains	Applications	Extract-Clean™	Ultra-Clean™	Maxi-Clean™
SCX	Styrene-DVB	Hydrogen	50µm	Benzene Sulfonic Acid	2.0meq/mL	Cations, (+) charged compounds	Remove/concentrate basic compounds.	x	x	x
SAX	Styrene-DVB	Acetate	50µm	Tetramethyl Ammonium	1.0meq/mL	Anions, (–) charged compounds	Remove/concentrate acidic compounds.	x	x	x

Ion Chromatography Sorbent Specifications

Packing	Base	Counter Ion	Particle Size	Molecular Exclusion Limit	Exchange Capacity	Retains	Applications	Extract-Clean™	Ultra-Clean™	Maxi-Clean™
IC-OH	Styrene-DVB	Hydroxide	50µm	1000 Daltons	1.0meq/mL	Anions	Exchanges anions for hydroxide. May be used to remove or concentrate anions from sample and to increase pH of acidic samples. Removes cations that form insoluble hydroxide salts.	x		x
IC-H	Styrene-DVB	Hydronium	50µm	1000 Daltons	2.0meq/mL	Cations	Exchanges cations for H^+. May be used to remove or concentrate cations from sample and to reduce pH of basic samples.	x		x
IC-Ag	Styrene-DVB	Silver	50µm	1000 Daltons	2.0meq/mL	Chloride Iodide Bromide	Removes excess halides through formation of Ag-halide salts.	x		x
IC-Ba	Styrene-DVB	Barium	50µm	1000 Daltons	2.0meq/mL	Sulfate	Removes excess sulfate through formation of BaSO.	x		x
IC-Na	Styrene-DVB	Sodium	50µm	1000 Daltons	2.0meq/mL	Cations	Exchanges cations for Na^+. May be used to remove or retain cations from sample without changing the pH of the sample.	x		x
IC-Chelate	Styrene-DVB	Sodium	50µm	1000 Daltons	0.4meq/mL	Polyvalent metal ions	Exchanges transition metals and divalent cations for Na^+. May be used to remove or retain divalent cations and transition metals from sample.	x		x
IC-RP	Polystyrene	—	550µm	—	—	Hydrophobic components	Removes surfactants, organic acids, and other organic substances. Inorganic ions pass through.	x		x