Solid Phase Extraction (SPE)
Discovery Products

Solid Phase Extraction Products

Designed to meet the exacting requirements of pharmaceutical and clinical analysis, Discovery SPE products are ideal for all application areas including: Food & Beverage, Environmental, Petrochemical, Argiculture, Consumer Products and more...

The multitude of phase chemistries and hardware configurations available within the Discovery SPE line offer the comprehensive level of selection and flexibility required to handle today's increasingly complex and diverse sample prep challenges.

Each Discovery SPE product includes an extensive Certificate of Analysis ensuring optimal performance and reproducible properties for each Discovery product shipped from Supelco.

Discovery SPE allows you to:

- Achieve greater and more reproducible recoveries for diverse compounds from difficult sample matrices
- Removes endogenous sample interference for improved accuracy and sensitivity
- Concentrate target analytes for increased sensitivity
- Protects analytical instrument from unwanted sample matrix components

Discovery SPE offers the quality and performance you need to bridge the sample prep gap between sample collection and analysis.

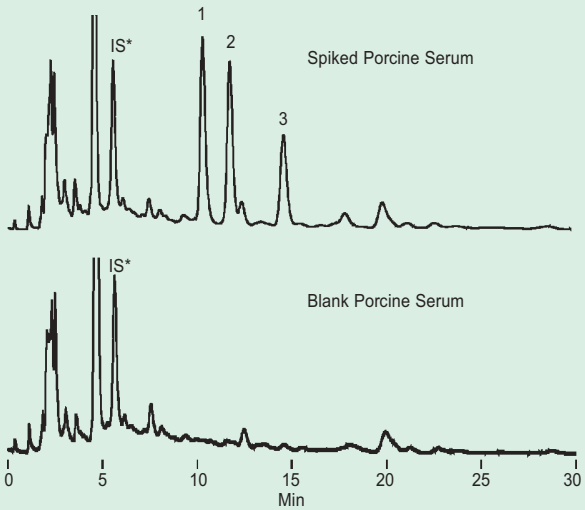
Discovery SPE Products:

- Developed, tested and quality controlled for pharmaceutical and clinical applications
- Twelve different phase chemistries ranging from polymerically bonded C18 to polyamide adsorbents
- Available in 96-well plate configurations for high throughput parallel processing
- Available in Buchner Funnel configurations for easier scalability (combinatorial chemistry clean-up)
- Ultra clean phases for highly sensitive analyses
- Narrower pore size distribution for improved extraction selectivity
- Acid washed to reduce metal chelating activity
- Consistent particle size and specific surface area coverage to ensure reproducible recoveries
- Low fines (<12µm) content to minimize injection port fouling

PROPERTIES

Base Silica:	Irregular shape, acid washed
Mean Particle Size:	50µm
Mean Pore Diameter:	70Å
Total Pore Volume:	0.9cm³/g
Specific Surface Area:	480m²/g
Endcapped:	Yes

Extraction of Anti-Ulcer Compounds from Porcine Serum using Discovery DSC-18 SPE

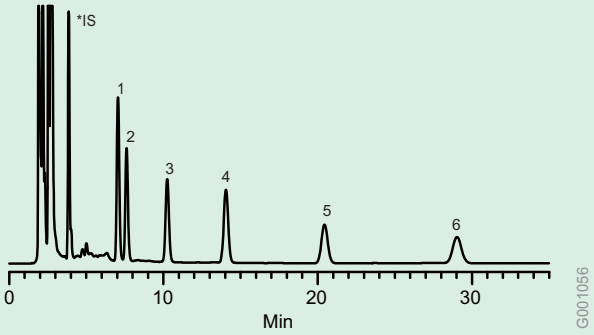


Compound	Concentration (µg/mL)	%Recovery ± RSD (n = 6)
1. Ranitidine	0.25	92.5 ± 5.4
	0.50	95.5 ± 5.1
2. Cimetidine	0.25	94.5 ± 5.2
	0.50	98.2 ± 3.2
3. Nizatidine	0.25	97.0 ± 7.0
	0.50	94.8 ± 3.4

Analyzed with a Discovery C18 HPLC Column, 15cm x 4.6mm ID, 5µm particles

* IS = Famotidine (internal standard).

Barbiturates from serum, using 500mg/3mL Discovery DSC-18Lt SPE tubes and Zymark RapidTrace SPE Workstation.



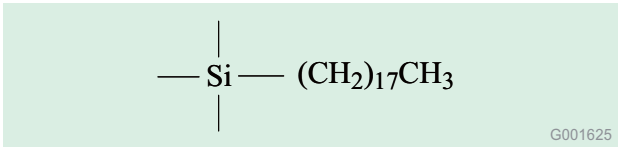
Analyzed with a Discovery C18 HPLC column, 15cm x 4.6mm ID, 5µm particles.

Efficiency of Recovery

Compound	Concentration (µg/mL)	%Recovery	%RSD (n=6)
1. Phenobarbital	0.5	96.2	±1.6
	1.0	94.9	±1.7
2. Aprobarbital	0.5	98.5	±2.1
	1.0	100.8	±0.8
3. Butabarbital	0.5	97.2	±1.9
	1.0	98.7	±1.8
4. Mephobarbital	0.5	99.7	±2.4
	1.0	101.0	±2.0
5. Pentobarbital	0.5	96.4	±1.7
	1.0	96.4	±1.9
6. Secobarbital	0.5	98.2	±1.7
	1.0	97.7	±1.8

* IS = Barbital (internal standard).

Discovery DSC-18 SPE Products



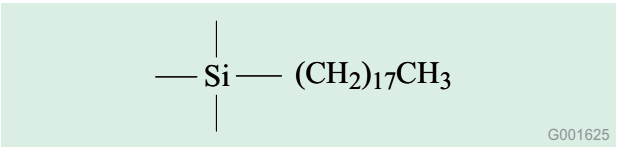
Retention Mechanism:Reversed-phase

Sample Matrix Compatibility: Aqueous solutions (biological fluids, water)

- 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
- Can also be used for desalting aqueous matrices
- Beneficial for extracting structurally diverse analytes from the same sample

DESCRIPTION	QTY.	CAT. NO.	PRICE
SPE TUBES			
50mg/1mL	108	52601-U	
100mg/1mL	108	52602-U	
500mg/3mL	54	52603-U	
500mg/6mL	30	52604-U	
1g/6mL	30	52606-U	
2g/12mL	20	52607-U	
5g/20mL	20	52608-U	
10g/60mL	16	52609-U	
SPE 96-WELL PLATES			
100mg/well	1	575603-U	
50mg/well	1	575602-U	
25mg/well	1	575601-U	
BULK PACKING			
Bulk packing	100g	52600-U	

Discovery DSC-18Lt SPE Products



Retention Mechanism:Reversed-phase

Sample Matrix Compatibility: Aqueous solutions (biological fluids, water)

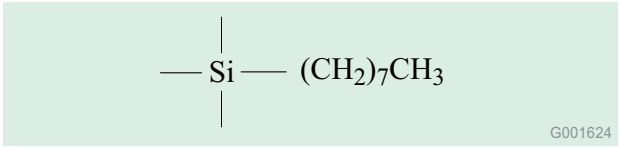
- Monomerically bonded, octadecyl (11%C), endcapped
- Increased retention for moderately polar hydrophobic molecules
- Used to elute very large hydrophobic molecules that are too strongly retained on DSC-18.
- Offers opportunity to differentiate between drug metabolites in bioanalysis applications
-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

DESCRIPTION	QTY.	CAT. NO.	PRICE
SPE TUBES			
50mg/1mL	108	52610-U	
100mg/1mL	108	52611-U	
500mg/3mL	54	52613-U	
500mg/6mL	30	52615-U	
1g/6mL	30	52616-U	
2g/12mL	20	52618-U	
5g/20mL	20	52621-U	
10g/60mL	16	52622-U	
SPE 96-WELL PLATES			
100mg/well	1	575606-U	
50mg/well	1	575605-U	
25mg/well	1	575604-U	
BULK PACKING			
Bulk packing	100g	52623-U	

Note: Unless stated otherwise, tubes are polypropylene. Frits are polyethylene with 20µm pores.

Solid Phase Extraction (SPE)
Discovery SPE Tubes

Discovery DSC-8 SPE Products



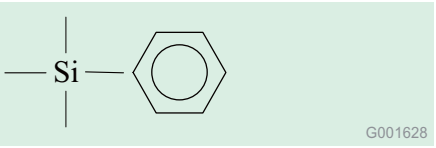
Retention Mechanism:Reversed-phase

Sample Matrix Compatibility: Aqueous solutions (biological fluids, water)

- 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
- Inorganic buffers of sufficient ionic strength may be used for elution

DESCRIPTION	QTY.	CAT. NO.	PRICE
SPE TUBES			
50mg/1mL	108	52703-U	
100mg/1mL	108	52707-U	
500mg/3mL	54	52713-U	
500mg/6mL	30	52714-U	
1g/6mL	30	52716-U	
2g/12mL	20	52717-U	
5g/20mL	20	52718-U	
10g/60mL	16	52722-U	
SPE 96-WELL PLATES			
100mg/well	1	575627-U	
50mg/well	1	575628-U	
25mg/well	1	575629-U	
BULK PACKING			
Bulk packing	100g	57223-U	

Discovery DSC-Ph SPE Products



Retention Mechanism:Reversed-phase

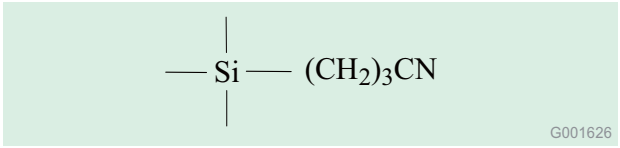
Sample Matrix Compatibility: Aqueous solutions (biological fluids, water)

- Monomerically bonded, phenyl (7%C), endcapped
- Similar in polarity to DSC-8; however, electron dense aromatic ring offers unique selectivity and retention
- Offers improved retention of conjugated ring structures over aliphatic functional groups.

DESCRIPTION	QTY.	CAT. NO.	PRICE
SPE TUBES			
50mg/1mL	108	52723-U	
100mg/1mL	108	52725-U	
500mg/3mL	54	52727-U	
500mg/6mL	30	52728-U	
1g/6mL	30	52731-U	
SPE 96-WELL PLATES			
100mg/well	1	575630-U	
50mg/well	1	575631-U	
25mg/well	1	575632-U	
BULK PACKING			
Bulk packing	100g	57227-U	

Note: Unless stated otherwise, tubes are polypropylene. Frits are polyethylene with 20µm pores.

Discovery DSC-CN SPE Products



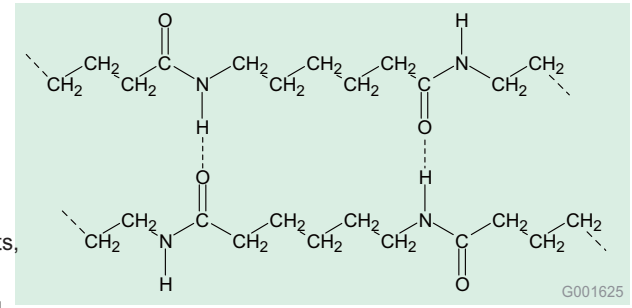
Retention Mechanism:Reversed-phase or Normal phase

Sample Matrix Compatibility: Aqueous solutions (biological fluids, water) when used in reversed-phase; or organic solvents, oils, and lipids when used in normal phase

- Monomerically bonded, cyanopropyl (7%C), endcapped
- Can be used in 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 in normal phase (organic matrices such as hexane or oils)
- Allows for the rapid release of very polar molecules irreversibly retained on very polar sorbents

DESCRIPTION	QTY.	CAT. NO.	PRICE
SPE TUBES			
50mg/1mL	108	52693-U	
100mg/1mL	108	52694-U	
500mg/3mL	54	52695-U	
500mg/6mL	30	52696-U	
1g/6mL	30	52697-U	
2g/12mL	20	52698-U	
5g/20mL	20	52699-U	
10g/60mL	16	52700-U	
SPE 96-WELL PLATES			
100mg/well	1	575624-U	
50mg/well	1	575625-U	
25mg/well	1	575626-U	
BULK PACKING			
Bulk packing	100g	57222-U	

Discovery DPA-6S SPE Products



Retention Mechanism:Reversed-phase

Sample Matrix Compatibility: Aqueous or methanolic solutions

- Polyamide Resin: Particle Size: 50-160µm, Surf pH: 4.5-7.5, Density: 0.2-0.3cm³/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 and nitroaromatic compounds
- Irreversibly retains quinones

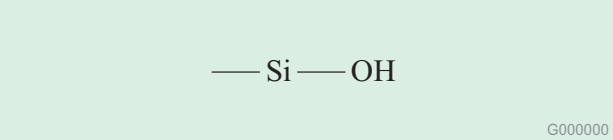
DESCRIPTION	QTY.	CAT. NO.	PRICE
SPE TUBES			
50mg/1mL	108	52624-U	
250mg/3mL	54	52625-U	
250mg/6mL	30	52626-U	
500mg/6mL	30	52627-U	
1g/12mL	20	52629-U	
2g/20mL	20	52631-U	
5g/60mL	16	52632-U	
BULK PACKING			
Bulk packing	50g	52633-U	
BUCHNER FUNNELS			
110mm ID x 66mm H; 50g/800mL	1	52634-U	

Note: Unless stated otherwise, tubes are polypropylene. Frits are polyethylene with 20µm pores.

Solid Phase Extraction (SPE)

Discovery SPE Tubes

Discovery DSC-Si SPE Products



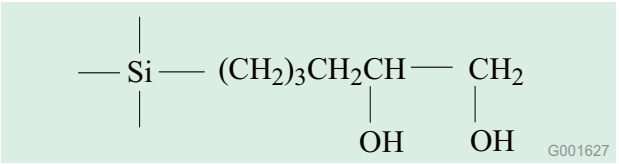
Retention Mechanism:Normal phase

Sample Matrix Compatibility: Organic solvents, oils, and lipids

- Unbonded acid washed silica sorbent ideal for normal phase SPE and other modified flash techniques
- Often used to separate or remove structurally similar molecules through successive elutions with increasingly polar solutions
- The most polar normal phase sorbent available
- Excellent capacity for purifying solution phase combinatorial chemistry reactions when removing target molecules from reaction by-products and excess reagents
- Available in Büchner Funnel configurations for easy scala

DESCRIPTION	QTY.	CAT. NO.	PRICE
SPE TUBES			
50mg/1mL	108	52652-U	
100mg/1mL	108	52653-U	
500mg/3mL	54	52654-U	
500mg/6mL	30	52655-U	
1g/6mL	30	52656-U	
2g/12mL	20	52657-U	
5g/20mL	20	52658-U	
10g/60mL	16	52659-U	
SPE 96-WELL PLATES			
100mg/well	1	575609-U	
50mg/well	1	575608-U	
25mg/well	1	575607-U	
BULK PACKING			
Bulk Packing	100g	52651-U	
BUCHNER FUNNELS			
50mmID x 30mmH; 12.5g	6	52591-U	
70mmID x 40mmH; 25g	6	52592-U	
90mmH x 48mmH; 50g	6	52593-U	
110mmID x 66mmH; 100g	6	52594-U	

Discovery DSC-Diol SPE Products



Retention Mechanism:Normal phase

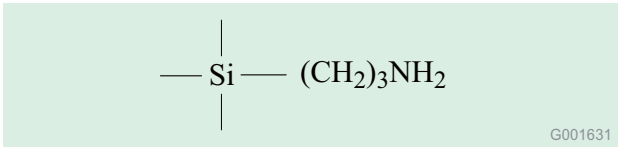
Sample Matrix Compatibility: Organic solvents, oils, and lipids

- 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 facilitates strong hydrogen bonding
- Excellent selectivity when extracting structurally similar molecules

DESCRIPTION	QTY.	CAT. NO.	PRICE
SPE TUBES			
50mg/1mL	108	52747-U	
100mg/1mL	108	52748-U	
500mg/3mL	54	52751-U	
500mg/6mL	30	52752-U	
1g/6mL	30	52753-U	
SPE 96-WELL PLATES			
100mg/well	1	575636-U	
50mg/well	1	575637-U	
25mg/well	1	575638-U	
BULK PACKING			
Bulk packing	100g	57229-U	

Note: Unless stated otherwise, tubes are polypropylene. Frits are polyethylene with 20µm pores.

Discovery DSC-NH_2 SPE Products



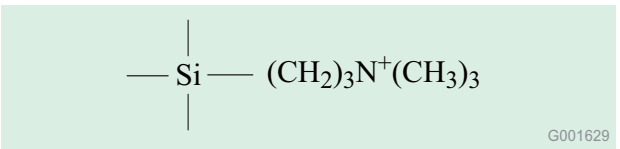
Retention Mechanism:Normal phase or Anion-exchange

Sample Matrix Compatibility: Organic or aqueous solutions

- 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
- Ion exchange capacity is ~ 0.43 meq/g.
- Allows the rapid release of very strong anions such as sulfonic acids that may be retained irreversibly by strong anion exchangers
- Can be used in some reversed-phase applications (due to ethyl spacer); however, it is predominately used as an ion-exch or normal phase sorbent due to its polar nature

DESCRIPTION	QTY.	CAT. NO.	PRICE
SPE TUBES			
50mg/1mL	108	52635-U	
100mg/1mL	108	52636-U	
500mg/3mL	54	52637-U	
500mg/6mL	30	52638-U	
1g/6mL	30	52640-U	
2g/12mL	20	52641-U	
5g/20mL	20	52642-U	
10g/60mL	16	52644-U	
SPE 96-WELL PLATES			
100mg/well	1	575615-U	
50mg/well	1	575616-U	
25mg/well	1	575617-U	
BULK PACKING			
Bulk packing	100g	57212-U	

Discovery DSC-SAX SPE Products



Retention Mechanism:Anion-exchange

Sample Matrix Compatibility: Organic or aqueous solutions

- A polymerically bonded quarternary amine that remains positively charged at all pH levels
- Counter ion is Cl^-
- Ion exchange capacity is ~ 0.14 meq/g
- 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

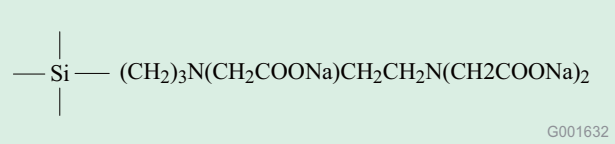
DESCRIPTION	QTY.	CAT. NO.	PRICE
SPE TUBES			
50mg/1mL	108	52661-U	
100mg/1mL	108	52662-U	
500mg/3mL	54	52664-U	
500mg/6mL	30	52665-U	
1g/6mL	30	52666-U	
2g/12mL	20	52667-U	
5g/20mL	20	52668-U	
10g/60mL	16	52669-U	
SPE 96-WELL PLATES			
100mg/well	1	575618-U	
50mg/well	1	575619-U	
25mg/well	1	575620-U	
BULK PACKING			
Bulk packing	100g	57214-U	

Note: Unless stated otherwise, tubes are polypropylene. Frits are polyethylene with 20µm pores.

Solid Phase Extraction (SPE)

Discovery SPE Tubes

Discovery DSC-WCX SPE Products



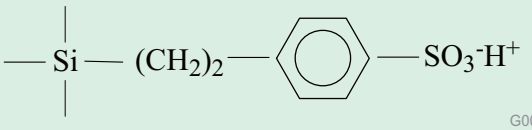
Retention Mechanism:Cation exchange

Sample Matrix Compatibility: Organic or aqueous solutions

- A polymerically bonded, carboxy propyl phase with a pKa of 4.8
- Counter ion is Na
- Ion exchange capacity is ~ 0.15 meq/g
- 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

DESCRIPTION	QTY.	CAT. NO.	PRICE
SPE TUBES			
50mg/1mL	108	52737-U	
100mg/1mL	108	52739-U	
500mg/3mL	54	52741-U	
500mg/6mL	30	52742-U	
1g/6mL	30	52743-U	
2g/12mL	20	52744-U	
5g/20mL	20	52745-U	
10g/60mL	16	52746-U	
SPE 96-WELL PLATES			
100mg/well	1	575633-U	
50mg/well	1	575634-U	
25mg/well	1	575635-U	
BULK PACKING			
Bulk packing	100g	57228-U	

Discovery DSC-SCX SPE Products



Retention Mechanism:Cation exchange

Sample Matrix Compatibility: Organic or aqueous solutions

- A polymerically bonded, benzene sulfonic acid functional group, pKa (<1.0)
- Counter ion is H
- Silica support allows for use with very organic solvents (no shrinking/swelling)
- Excellent capacity (0.8meq/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

DESCRIPTION	QTY.	CAT. NO.	PRICE
SPE TUBES			
50mg/1mL	108	52684-U	
100mg/1mL	108	52685-U	
500mg/3mL	54	52686-U	
500mg/6mL	30	52688-U	
1g/6mL	30	52689-U	
2g/12mL	20	52690-U	
5g/20mL	20	52691-U	
10g/60mL	16	52692-U	
SPE 96-WELL PLATES			
100mg/well	1	575621-U	
50mg/well	1	575622-U	
25mg/well	1	575623-U	
BULK PACKING			
Bulk packing	100g	57221-U	

Note: Unless stated otherwise, tubes are polypropylene. Frits are polyethylene with 20µm pores.

Solid Phase Extraction (SPE)
Supelclean ENVI SPE Tubes and Disks

Supelclean ENVI SPE Products:

- Developed, highly tested, and quality controlled for environmental applications
- Seven different phase chemistries ranging from our unique ENVI-Carb carbon adsorbents to ENVI-18 DSKs – reversed phase SPE membranes for large volume water samples
- Available in glass tubes, Teflon and stainless steel frit configurations for EPA compliance
- Ultra clean phases for highly sensitive analyses
- Documented applications in compliance to standardized EPA methodology
- Consistent particle size and specific surface area to ensure reproducible recoveries

PROPERTIES	
Base Silica:	Irregular shape, acid washed
Mean Particle Size:	45µm
Mean Pore Diameter:	60Å
Total Pore Volume:	0.8cm³/g
Specific Surface Area:	475m²/g
Endcapped:	Yes

Supelclean ENVI-18 SPE Products

Retention Mechanism:Reversed-phase

Sample Matrix Compatibility:Aqueous solutions (drinking, ground, waste water)

- Polymerically bonded, octadecyl (17%C), endcapped
- Excellent for cleaning, extracting and concentrating pollutants from aqueous environmental samples
- Higher 17%C loading for increased binding capacities and higher recoveries
- Higher carbon loading also offers greater resistance to extreme pH conditions
- Used for extracting herbicides, fungicides, and pesticides from waste material

DESCRIPTION	QTY.	CAT. NO.	PRICE
SPE TUBES			
100mg/1mL	108	57062	
500mg/3mL	54	57063	
500mg/6mL	30	57064	
1g/6mL	30	505706	
2g/12mL	20	57114	
5g/20mL	20	57137	
10g/60mL	16	57138	
BULK PACKING			
Bulk packing	100g	57219	

Supelclean ENVI-8 SPE Products

Retention Mechanism:Reversed-phase

Sample Matrix Compatibility:Aqueous solutions (drinking, ground, waste water)

- High 14%C loading for increased binding capacities and higher recoveries
- Higher carbon loading also offers greater resistance to extreme pH conditions
- Excellent for cleaning, extracting and concentrating pollutants from aqueous environmental samples
- Used for extracting herbicides, fungicides, and pesticides from waste material

DESCRIPTION	QTY.	CAT. NO.	PRICE
SPE TUBES			
100mg/1mL	108	57230-U	
500mg/3mL	54	57231	
500mg/6mL	30	57232	
1g/6mL	30	57233	
5g/20mL	20	57139	
10g/60mL	16	57140-U	
SPE TUBES (GLASS TUBES; TEFLON FRITS)			
500mg/3mL	27	57106	
500mg/6mL	20	57107	

Supelclean ENVI-18 & ENVI-8 DSK SPE Discs

The SPE membrane equivalents of ENVI-18 and ENVI-8 packed bed SPE sorbents

Retention Mechanism:Reversed-phase

Sample Matrix Compatibility:Aqueous solutions (drinking water)

- Porous glass fiber membranes embedded with C18 or C8 modified silica particles.
- Provides faster flow rates and exhibits less clogging than Teflon discs for the extraction of organic contaminants from drinking water samples
- Typical applications include polynuclear aromatic hydrocarbons (PAHs), polychlorinated biphenyls (PCBs), phthalates, semivolatile organics, paraquat and diquat, pesticides and herbicides

DESCRIPTION	QTY.	CAT. NO.	PRICE
ENVI-18DSK SPE DISKS			
47mm Diam.	24	57171	
90mm Diam.	12	57170-U	
ENVI-8DSK SPE DISKS			
47mm Diam.	24	57172	

Note: Unless stated otherwise, tubes are polypropylene. Frits are polyethylene with 20µm pores.

Solid Phase Extraction (SPE)

Supelclean ENVI SPE Tubes

Supelclean ENVI-Carb SPE Products

Graphitized Non-Porous Carbon

Retention Mechanism:Reversed-phase

Sample Matrix Compatibility: Aqueous solutions (drinking, ground, waste water)

- 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 vegetable

DESCRIPTION	QTY.	CAT. NO.	PRICE
ENVI-CARB (SURF. AREA 100MG; 120/400 MESH)			
250mg/3mL	54	57088	
250mg/6mL	30	57092	
500mg/6mL	30	57094	
1g/12mL	20	57127-U	
2g/12mL	20	57128	
5g/20mL	20	57129	
10g/60mL	16	57130	
Bulk Packing	50g	57210-U	
ENVI-CARB C (SURF. AREA 10MG; 80/100 MESH)			
1g/12mL	20	57149	
ENVI-CARB X (SURF. AREA 250MG; 120/400 MESH)			
Bulk Packing	50g	10439-U	
ENVI-CARB Y (SURF. AREA 25MG; 120/400 MESH)			
Bulk Packing	50g	10464-U	

Supelclean ENVI-Chrom P SPE Products

Styrene/divinyl benzene co-polymer

Retention Mechanism:Reversed-phase or Adsorption

Sample Matrix Compatibility: Aqueous solutions

- Particle Size: 80-160µm; Spherical Shape; Pore Size: 110-175Å; Surface Area: 900m²/g
- Highly crosslinked, neutral, specially cleaned styrene-divinylbenzene resin used to retain hydrophobic compounds with some hydrophilic functionality under reversed phase conditions
- Highly resistant to extreme pH conditions
- Typical applications include aromatics and phenolic compounds from aqueous sample matrices
- Used for priority pollutant phenols from aqueous samples

DESCRIPTION	QTY.	CAT. NO.	PRICE
ENVI-CHROM P SPE TUBES (GLASS TUBES; TEFLON FRITS)			
100mg/1mL	108	57143	
250mg/3mL	54	57224	
250mg/6mL	30	57225-U	
500mg/6mL	30	57226	
ENVI-CHROM P BULK PACKING			
Bulk packing	50g	57217	

Supelclean ENVI-Florisil

Magnesium Silicate

Retention Mechanism:Normal phase or Adsorption

Sample Matrix Compatibility: Organic solutions

- Mesh: 100/120; Available with Teflon 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 to polar compounds from nonpolar matrices under normal phase conditions
- Typical applications include alcohols, aldehydes, amines, herbicides, pesticides, PCBs, ketones, nitro compounds, organic acids, and phenols

DESCRIPTION	QTY.	CAT. NO.	PRICE
ENVI-FLORISIL			
500mg/3mL, Teflon	54	57058	
500mg/6mL, SS	30	57046	
1g/6mL, SS	30	57053	

Note: Unless stated otherwise, tubes are polypropylene. Frits are polyethylene with 20µm pores.

Reversed Phase Supelclean SPE Tubes

Extract nonpolar to moderately polar analytes from aqueous samples.

DESCRIPTION	QTY.	CAT. NO.	PRICE
LC-18 (OCTADECYL, ~10% C, ENDCAPPED)			
100mg/1mL	108	504270	
500mg/3mL	54	57012	
500mg/6mL	30	57054	
1g/6mL	30	505471	
2g/12mL	20	57117	
5g/20mL	20	57135-U	
10g/60mL	16	57136	
Bulk Packing	100g	57202	
LC-8 (OCTYL, ~7% C, ENDCAPPED)			
100mg/1mL	108	504157	
500mg/3mL	54	505145	
500mg/6mL	30	57052	
Bulk Packing	100g	57201	
LC-4 (BUTYLDIMETHYL, 500Å PORES, ENDCAPPED)			
500mg/3mL	54	57089	
LC-PH (PHENYL, ~5.5% C, ENDCAPPED)			
100mg/1mL	108	504599	
500mg/3mL	54	505269	
HISEP (HYDROPHOBIC SITES SHIELDED BY A HYDROPHILIC SURFACE; FOR PROTEIN EXCLUSION)			
500mg/3mL	54	57076-U	

Normal Phase Supelclean SPE Tubes

Extract moderately polar to polar analytes from nonaqueous samples.

DESCRIPTION	QTY.	CAT. NO.	PRICE
LC-CN (CYANOPROPYL, ~7% C, ENDCAPPED)			
100mg/1mL	108	504386	
500mg/3mL	54	57013	
500mg/6mL	30	57056	
5g/20mL	20	57141	
10g/60mL	16	57142	
LC-NH_2 (AMINOPROPYL, ~5% C)			
100mg/1mL	108	504483	
500mg/3mL	54	57014	
Bulk Packing	100g	57205	
LC-DIOL (DIOL, ~7% C)			
100mg/1mL	108	504718	
500mg/3mL	54	57016	

Adsorption Supelclean SPE Tubes

No bonded phase; extract polar analytes from nonpolar samples (LCP-Si, LC-Florisil, LC-Alumina).

DESCRIPTION	QTY.	CAT. NO.	PRICE
LC-SI (SILICA GEL)			
100mg/1mL	108	504041	
500mg/3mL	54	505048	
500mg/6mL	30	505374	
1g/6mL	30	57051	
2g/12mL	20	57116	
5g/20mL	20	57133	
10g/60mL	16	57134	
Bulk Packing	100g	57200	
LC-FLORISIL (MAGNESIUM SILICATE, 100/120 MESH)			
1g/6mL	30	57057	
2g/12mL	20	57115	
5g/20mL	20	57131	
10g/60mL	16	57132	
Bulk Packing	100g	57209	
LC-ALUMINA-N (ALUMINA FOR NEUTRAL pH (~6.5) BROCKMANN ACT. I, 60/325 MESH)			
1g/3mL	54	57086	
2g/6mL	30	57087	
Bulk Packing	100g	57208	
LC-ALUMINA-A (ALUMINA FOR ACIDIC pH (~5) BROCKMANN ACT. I, 60/325 MESH)			
1g/3mL	54	57082-U	
2g/6mL	30	57083-U	
Bulk Packing	100g	57206	
LC-ALUMINA-B (ALUMINA FOR BASIC pH (~8.5) BROCKMANN ACT. I, 60/325 MESH)			
1g/3mL	54	57084	
2g/6mL	30	57085	

Ion Exchange Supelclean SPE Tubes

Interaction based on ionic attraction.

DESCRIPTION	QTY.	CAT. NO.	PRICE
LC-SAX (QUATERNARY AMINE, Cl^- COUNTERION)			
100mg/1mL	108	504815	
500mg/3mL	54	57017	
Bulk Packing	100g	57203	
LC-SCX (ALIPHATIC SULFONIC ACID, Na^+ COUNTERION)			
100mg/1mL	108	504920	
500mg/3mL	54	57018	
Bulk Packing	100g	57204	
LC-WCX (CARBOXYLIC ACID, Na^+ COUNTERION)			
100mg/1mL	108	505595	
500mg/3mL	54	57061	

Note: Unless stated otherwise, tubes are polypropylene. Frits are polyethylene with 20µm pores.

Solid Phase Extraction (SPE)

Method Development Kits, Discovery 96-Well Plates

Supelclean SPE Method Development Kits

KIT:	KIT A	KIT B	KIT C	KIT RP-3	KIT NP-3	KIT IX-1	KIT IX-3
Packing	Sorbent Qty./Tube Size						
LC-Si	500mg/3mL	100mg/1mL	500mg/6mL 1g/6mL		500mg/3mL		
LC-8	500mg/3mL	100mg/1mL	500mg/6mL	500mg/3mL			
LC-18	500mg/3mL	100mg/1mL 1g/6mL	500mg/6mL	500mg/3mL			
LC-CN	500mg/3mL	100mg/1mL	500mg/6mL	500mg/3mL		100mg/1mL	500mg/3mL
LC-Diol	500mg/3mL	100mg/1mL			500mg/3mL		
LC-NH_2	500mg/3mL	100mg/1mL			500mg/3mL	100mg/1mL	500mg/3mL
LC-Ph	500mg/3mL	100mg/1mL		500mg/3mL			
LC-SAX	500mg/3mL	100mg/1mL				100mg/1mL	500mg/3mL
LC-SCX	500mg/3mL	100mg/1mL				100mg/1mL	500mg/3mL
LC-WCX	500mg/3mL	100mg/1mL				100mg/1mL	500mg/3mL
LC-Alumina-A			2g/6mL		1g/3mL		
LC-Alumina-B			2g/6mL		1g/3mL		
LC-Alumina-N			2g/6mL		1g/3mL		
LC-Florisil			1g/6mL				
QTY. EA. TUBE:	6	12	3	12	6	24	12
CAT. NO.:	57019	57009-U	57075-U	57071	57074-U	57072	57073
PRICE:							

Discovery 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.

DESCRIPTION	QTY.	CAT. NO.	PRICE
DSC-18 SPE 96-WELL PLATES			
100mg/well	1	575603-U	
50mg/well	1	575602-U	
25mg/well	1	575601-U	
DSC-18LT SPE 96-WELL PLATES			
100mg/well	1	575606-U	
50mg/well	1	575605-U	
25mg/well	1	575604-U	
DSC-8 SPE 96-WELL PLATES			
100mg/well	1	575627-U	
50mg/well	1	575628-U	
25mg/well	1	575629-U	
DSC PH SPE 96-WELL PLATES			
100mg/well	1	575630-U	
50mg/well	1	575631-U	
25mg/well	1	575632-U	
DSC-CN SPE 96-WELL PLATES			
100mg/well	1	575624-U	
50mg/well	1	575625-U	
25mg/well	1	575626-U	
DSC-SI SPE 96-WELL PLATES			
100mg/well	1	575609-U	
50mg/well	1	575608-U	
25mg/well	1	575607-U	

DESCRIPTION	QTY.	CAT. NO.	PRICE
DSC-DIOL SPE 96-WELL PLATES			
100mg/well	1	575636-U	
50mg/well	1	575637-U	
25mg/well	1	575638-U	
DSC-NH_2 SPE 96-WELL PLATES			
100mg/well	1	575615-U	
50mg/well	1	575616-U	
25mg/well	1	575617-U	
DSC-SAX SPE 96-WELL PLATES			
100mg/well	1	575618-U	
50mg/well	1	575619-U	
25mg/well	1	575620-U	
DSC-WCX SPE 96-WELL PLATES			
100mg/well	1	575633-U	
50mg/well	1	575634-U	
25mg/well	1	575635-U	
DSC-SCX SPE 96-WELL PLATES			
100mg/well	1	575621-U	
50mg/well	1	575622-U	
25mg/well	1	575623-U	
DSC-PS/DVB SPE 96-WELL PLATES			
50mg/well	1	575611-U	
25mg/well	1	575610-U	

Solid Phase Combinatorial Chemistry

In recent years, advances in combinatorial chemistry (CombiChem) have made a tremendous impact on the pharmaceutical industry by dramatically accelerating the drug discovery process. However, for each synthesis a purification step is required to remove the target molecule from reaction by-products and excess reagents. Because many reactions contain polar to moderately polar reagents, by-products, and products that can be selectively extracted with normal phase SPE, modified flash techniques utilizing silica packed SPE hardware have become a routine procedure for purifying solution-phase combinatorial reactions.

Discovery SPE products offer combinatorial chemists an excellent opportunity for developing a simple and standardized high throughput purification method for their combinatorial libraries.

In normal phase SPE, polar compounds are retained or adsorbed onto the sorbent via polar-polar interactions when loaded in the presence of an organic sample matrix. Provided that the products, by-products, and reagents display varying polarities, choosing solvents with increasing polarity will allow for sequential elution of key compounds. In most combinatorial flash purification techniques, compounds not of interest are retained on the stationary phase. The products are then collected for analysis in the load flow through, or if weakly adsorbed, they can be selectively removed with a subsequent wash step.

Many combinatorial chemistry labs are synthesizing and characterizing extensive drug libraries. Chemists are therefore employing modified flash chromatography techniques in a 96-well SPE format for the purpose of sample clean-up and baseline impurity removal. In many combinatorial chemistry labs, capacity is a primary concern for such applications. In our studies, we have determined the binding capacity of 4-Fluoro-3-nitrobenzoic acid when loaded into a DSC-Si SPE 96-well plate (100mg/well). Our results show that ~12.5mg of the Fluoro compound can be loaded onto 100mg DSC-Si before beakthrough occurs. Breakthrough determination was analyzed via HPLC analysis (see Table A).

Table A. Binding Capacity of 4-Fluoro-3-Nitrobenzoic acid on DSC-Si (100mg/well)

LOAD AMOUNT*	BREAKTHROUGH AMOUNT
2.5mg	No Breakthrough
5.0mg	No Breakthrough
10.0mg	No Breakthrough
12.5mg	No Breakthrough
15.0mg	0.10 % Breakthrough Occured

* Sample Matrix in 200µL Methylene Chloride
n = 3 for each load amount.

RELATED INFORMATION

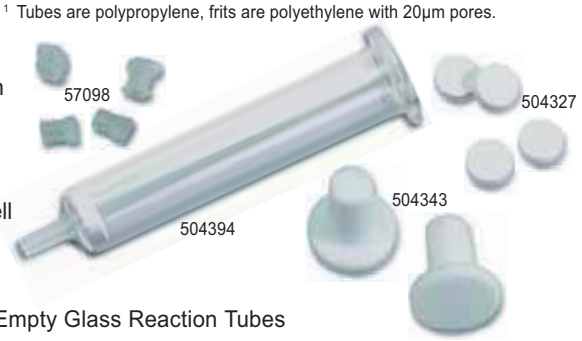
For more information on Combinatorial Chemistry request the following free technical literature.

No.	Title
EDZ	Sigma-Aldrich Combinatorial Chemistry Handbook
DGQ	Aldrich Polymer Products CD - Catalog and Reference Guide

Discovery DCS-Si SPE Products

DESCRIPTION	QTY.	CAT. NO.	PRICE
SPE TUBES			
50mg/1mL	108	52652-U	
100mg/1mL	108	52653-U	
500mg/3mL	54	52654-U	
500mg/6mL	30	52655-U	
1g/6mL	30	52656-U	
2g/12mL	20	52657-U	
5g/20mL	20	52658-U	
10g/60mL	16	52659-U	
SPE-96 WELL PLATES			
100mg/well	1	575609-U	
50mg/well	1	575608-U	
25mg/well	1	575607-U	
BULK PACKING			
Bulk Packing	100g	52651-U	
BÜCHNER FUNNELS			
50mmID x 30mmH; 12.5g	6	52591-U	
70mmID x 40mmH; 25g	6	52592-U	
90mmH x 48mmH; 50g	6	52593-U	
110mmID x 66mmH; 100g	6	52594-U	

[1] Tubes are polypropylene, frits are polyethylene with 20µm pores.



Empty Glass Reaction Tubes

Inert glass tubes, Teflon frits and Teflon closures

- Reduce interferences and contamination of your reaction mixtures
- Resistant to aggressive solvents and chemical solutions
- High flow frit porosity allows for gravity or rapid vacuum rinsing

DESCRIPTION	QTY.	CAT. NO.	PRICE
6mL glass tubes, Teflon frits	24	504394	
Teflon Tube adapters with port	24	504335	
6mL solid Teflon caps	24	504343	
Male luer plugs, PP	12	504351	
Female luer plugs, PP	12	57098	
Replacement Teflon frits for 6mL glass tubes	60	504327	

Combigel XE-305 Support

Our version of Amberlite XE-305. A proprietary, underivatized, polystyrene resin with unique swelling properties that make it ideal for solid phase combinatorial chemistry reactions.

DESCRIPTION	QTY.	CAT. NO.	PRICE
Bulk Packing	50g	502537B	

Solid Phase Extraction (SPE)

Custom Products, Hardware Configuration

Custom SPE products

Supelco's line of SPE products comprised of an array of sorbents, resins and hardware configurations including polypropylene tu glass tubes, 96-well plates, Büchner funnels, and various positive pressure cartridges. Scattered throughout our standard SPE line you'll see the availability of these various SPE devices at varying degrees. Supelco offers custom manufacturing services you can optimize your sample processing procedure to the parameters dictated by your sample prep objectives. If there's a cert permutation of phase chemistry, bed weight and hardware configuration you require that's 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, pl contact our Order Processing representatives:

Telephone: 800-247-6628, 814-359-3441

Fax: 800-447-3044, 814-359-3044

Email: supelco@sial.com

Polypropylene SPE Tubes

Standard Design Supelco's standard Discovery and Supelclean SPE tubes are comprised primarily of straight-walled serelogical grade polypropylene syringe barrels. Each of the 20+ available bonded phases and resins are available in an array of bed weights and volumes ranging from 1, 3, 6, 12, 20, and 60mL.

Flangeless Design Flangeless (tabless/wingless) 1 and 3mL SPE tubes that can be eluted directly into 96-well collection plates, using the Gilson Nebula Series SPE 215 System.

Reversible Design Our reversible SPE tubes allows for both forward and reverse flow capabilities offering great utility in trace enrichment applications. The tubes consist of a female luer inlet and a male luer outlet. Reversible tubes are available in 0.5, 1, and 2mL configurations.

Glass Tubes

Inert glass tubes (3 & 6mL) are available for preparations that demand high purity extracts and increased solvent compatibility.

Teflon and Stainless Steel Frits

Use Teflon or stainless steel frits when solvent compatibility and tube cleanliness are of concern. Stainless steel frits are not available with glass SPE tubes.

Discovery SPE 96-Well Plates

Process up to 96 samples at once using Discovery SPE 96-Well Plates. The well plates are a one-piece 2mL polypropylene square well design which will fit most standard well plate manifolds. Available bed weights include 25, 50, & 100mg/well. The well plates are compatible with most robotic and automated liquid handling systems:

- TomTec Quadra 96
- Packard Multi-Probe
- Gilson SPE 215
- Beckman Biomek

Büchner Funnels

Our Büchner funnels are sturdy two piece polypropylene units offering excellent chemical resistance, making them invaluable tools for large scale pharmaceutical preparations. The upper half of the Büchner funnels come pre-packed with the Supelco resin or bonded phase of your choice. Holding the packed bed in place are two polyethylene frits layered tight with a thermally welded retaining ring. Available Büchner funnel dimensions and bed weights include:

- 55mmID x 30mmH, 12.5g
- 90mmID x 48mmH, 50g
- 70mmID x 40mmH, 25g
- 110mmID x 66mmH, 100g

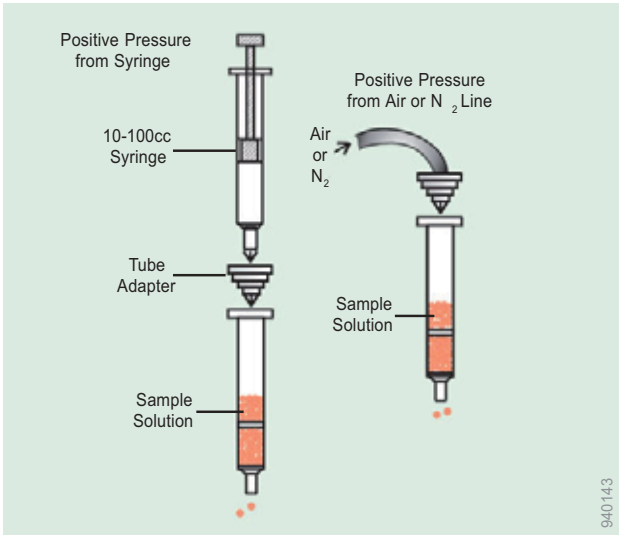
Rezorian Cartridges

Our disposable Rezorian Luer-Lock syringe-tip cartridges are fast and convenient for isolating, purifying, and concentrating molecules from a variety of sample matrices. Use where positive pressure is preferred. Rezorian cartridges, pre-packed with the Supelco bonded-phase or resin of your choice, are available in 1 & 5mL configurations.

Polypropylene SPE Tube Components

These components can be used for packing your own SPE material.

DESCRIPTION	QTY.	CAT. NO.	PRICE
EMPTY POLYPROPYLENE SPE TUBES WITH POLYETHYLENE FRITS (20µm PORE SIZE)			
1mL	108	57023	
3mL	54	57024	
6mL	30	57026	
12mL	20	57176	
20mL	20	57177	
60mL	16	57178	
EMPTY POLYPROPYLENE SPE TUBES (NO FRITS)			
1mL	108	57240-U	
3mL	54	57241	
6mL	30	57242	
12mL	20	57179	
20mL	12	57021	
60mL	12	57022	
POLYETHYLENE FRITS (20µm PORE SIZE)			
For 1mL Tubes	216	57244	
For 3mL Tubes	108	57180-U	
For 6mL Tubes	60	57181	
For 12mL Tubes	40	57182-U	
For 20mL Tubes	40	57183	
For 60mL Tubes	32	57184	
STAINLESS STEEL FRITS (20µm PORE SIZE)			
For 6mL Tubes	60	57246-U	
TEFLON FRITS (20µm PORE SIZE)			
For 1mL Tubes	216	57185	
For 3mL Tubes	108	57186	
For 6mL Tubes	60	57187	
For 12mL Tubes	40	57188	
For 20mL Tubes	40	57189	
For 60mL Tubes	32	57190-U	
CAPS FOR POLYPROPYLENE SPE TUBES (ENCLOSES TOP OF SPE TUBES)			
For 1mL Tubes	108	52171-U	
For 3mL Tubes	54	52172-U	
For 6mL Tubes	30	52173-U	
For 12mL Tubes	20	52174-U	
For 20mL Tubes	20	52175-U	
For 60mL Tubes	20	52176-U	
MALE & FEMALE LUER PLUGS (SEALS LUER OUTLETS ON SPE TUBES)			
Male Luer Plugs	12	504351	
Female Luer Plugs	12	57098	

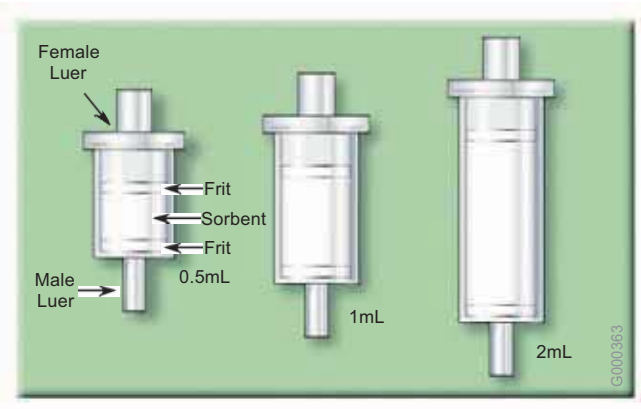


Tube Adapters

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/N_2 line).

DESCRIPTION	QTY.	CAT. NO.	PRICE
SPE TUBE ADAPTERS FOR POLYPROPYLENE TUBES			
For 1, 3, 6mL tubes	12	57020-U	
For 12, 20, 60mL tubes	6	57267	
AUTOTRACE SPE TUBE ADAPTERS*			
For 3mL Tubes	6	57123	
For 6mL Tubes	6	57126	

* Allows SPE tubes to be used with AutoTrace Automated Systems

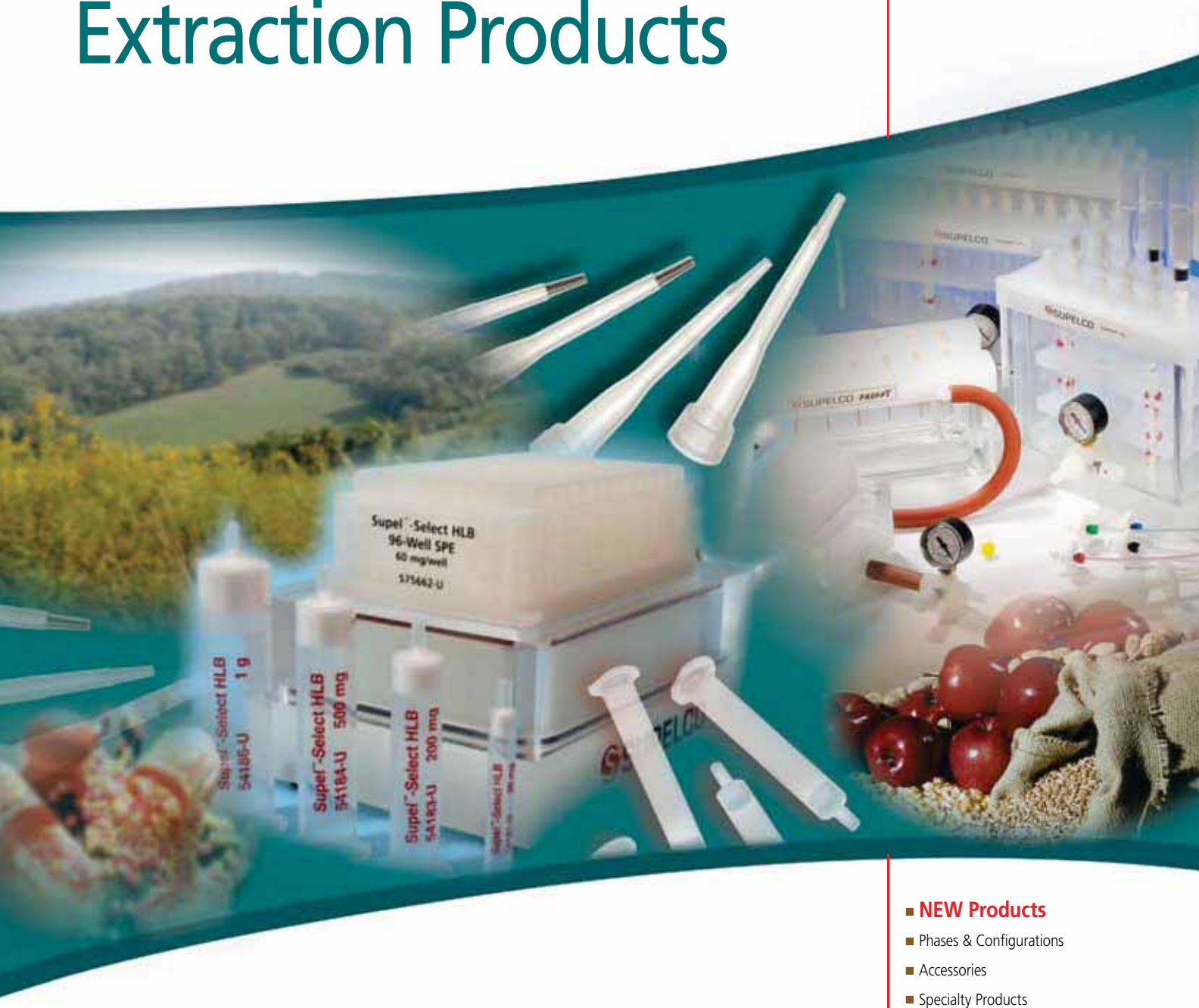


Empty Reversible SPE Tubes

Our reversible SPE tubes provide good utility in trace enrichment applications by permitting forward and reverse flows. These tubes consist of a female luer inlet and a male luer outlet, and are constructed of polypropylene. Reversible tubes are available in 0.5, 1, and 2mL configurations with maximum bed wieghts of 175, 350, and 700mg respectively. Tubes are available pre-packed with the Supelco bonded-phase or resin of your choice through our custom service (see previous page).

Supelco Solid Phase Extraction Products

SUPELCO Analytical



- **NEW Products**
- Phases & Configurations
- Accessories
- Specialty Products
- Quick Look-up Guides
- Useful Tips

Achieve Your Sample Prep Objectives

SIGMA-ALDRICH®

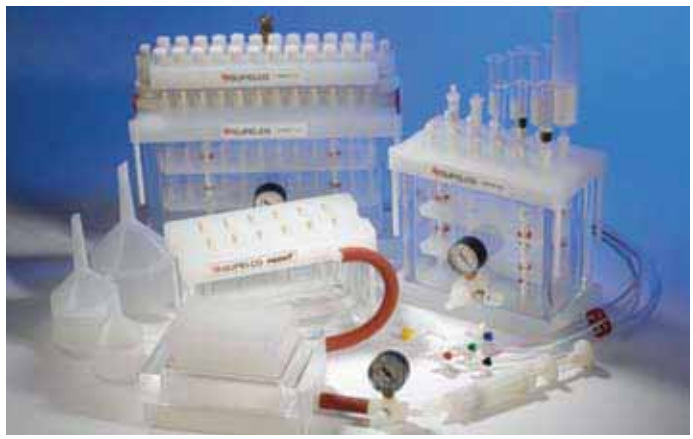
A Brief History of Supelco Solid Phase Extraction (SPE)

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

Beginning 2007, the emphasis for Supelco Sample Prep R&D has been innovation. For example, MIP Technologies AB collaborated with Supelco to introduce SupelMIP™ SPE - Molecularly Imprinted Polymer Technology. HybridSPE™ -Precipitation was developed for phospholipid and protein removal.

In addition, Supelco was among the first to introduce a dispersive SPE (QuEChERS) product line for multi-residue pesticide analysis.



P001015

20+ Years Ago

Supelclean & Supelclean ENVI

- Original pioneers of commercially available SPE Products
- Referenced in 100s of publications
- Developed, tested, and quality controlled for environmental applications
- Available in glass tubes, disk formats, and PTFE frits
- Unique chemistries such as ENVI-Carb™
- Documented applications in compliance to standardized EPA 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 and Büchner funnel configurations
- Ultra-clean phases for highly sensitive analyses

Present

An Era of Innovative SPE

- SupelMIP SPE – Molecularly Imprinted Polymers for extreme selectivity
- HybridSPE-Precipitation for quick and easy phospholipid and protein removal
- Supelclean Sulfoxide SPE for PCB analysis
- Dispersive SPE for multi-residue pesticide analysis
- Supel™-Select HLB SPE – our newest line of hydrophilic polymer SPE phases
- … and more!

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)

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:

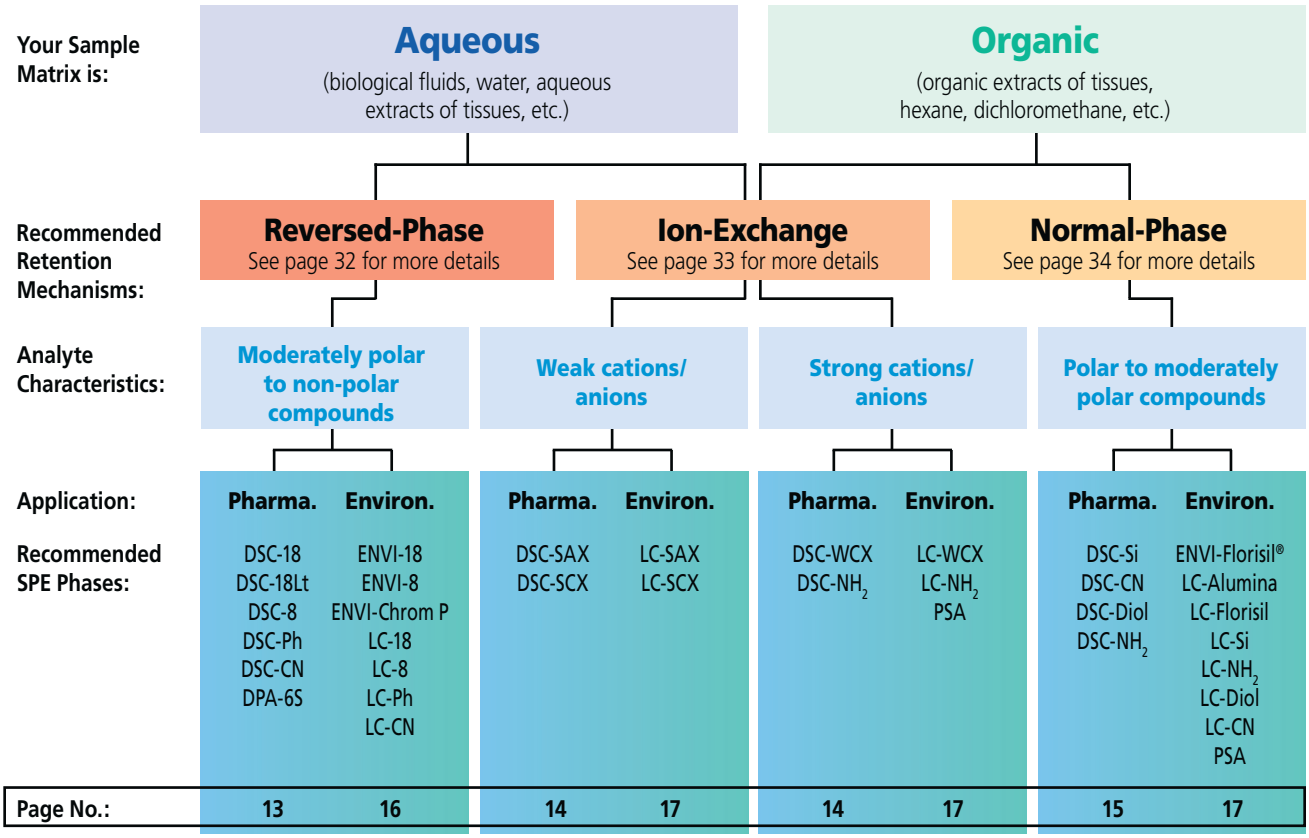
- Switch sample matrices to a form more compatible with chromatographic analyses
- Concentrate analytes for increased sensitivity
- Remove interferences to simplify chromatography and improve quantitation
- Protect the analytical column from contaminants

Common SPE Applications:

- Pharmaceutical compounds and metabolites in biological fluids
- Drugs of abuse in biological fluids
- Environmental pollutants in drinking and wastewater
- 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/spe* or refer to the current Supelco catalog.

Table of Contents:

SPE Phase Selection Quick Look-Up Guide



Supelco SPE Specialty Phases:

Phase Description	Field/Applic.	Page	Description
HybridSPE-Precipitation	Ph	8	Combines the simplicity of protein precipitation with the selectivity of SPE for the targeted removal of proteins andphospholipids in biological samples
Supel-Select HLB	Ph, G, F	9	Hydrophilic modified styrene based polymer for the broad range extraction of diverse analytes from aqueous samples
Empore SPE	Ph, E, G	10	SPE particles enmeshed in a PTFE membrane. Allows for faster flow-rates, smaller solvent consumption, and reduced elution volumes.
SupelMIP SPE	Ph, F, E	12	Molecularly imprinted polymers for the highly selective extraction of trace analytes in difficult sample matrices
Discovery DSC-MCAX	Ph, G	14	Mixed-mode cation exchange for superior selectivity/sample cleanup when extracting basic compounds (most pharmaceuticals) from biological fluids (e.g., plasma, urine, etc.)
Discovery DPA-6S	G, E, Ph	13	Polyamide resin that adsorbs polar compounds containing multi –OH and –COOH groups. Useful for extracting polyphenolics and other natural compounds (e.g., flavanoids, chlorophyll, humic acid, etc.) from plant extracts.
Polymer SAX Rezorian™ Cartridge / Polymer SCX Reversible Tube	G	21	Strong cation and anion exchanger on a styrene base particle. Offers much higher ion-exchange capacity than silica based ion-exchangers.
Supelclean ENVI-18 and –8 DSK SPE Disks	E	16	Provides fast flow rates for processing large volumes of water samples (≥0.5 L). Used in EPA 500 series methods – Drinking Water.
Dual Layer Florisil/Na_2SO_4	E	17	For total petroleum hydrocarbon index according to European Method EN9377-2
Supelclean Coconut Charcoal	E	22	Configured for EPA Method 521 – Nitrosamines in Water
Supelclean ENVI-Carb Plus	E	22	Spherical carbon particles packed in a reversible tube for the extraction of highly polar compounds from water
Supelclean Sulfoxide	E	22	Developed for the highly selective extraction of PCBs from transformer and waste oil
EPA 8290 SPE Tubes	E	22	Multi-layer SPE tubes configured for EPA Method 8290 - PCDDs and PCDFs by HRGC/HRMS
Discovery Ag-Ion	F	24	Silver Ion SPE for the fractionation of cis-trans isomers and other FAMEs
Supelclean ENVI-Carb	F	23	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	F	23	Developed to provide superior cleanup when conducting multi-residue pesticide analysis in food/agricultural (ENVI-Carb, SAX, PSA, NH_2) matrices
Supelclean LC-4 (wide pore)	B	16	Used for desalting proteins/peptides and other macromolecules

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

SPE Bed Weight Quick Look-Up Guide

Choosing the Right Bed Weight and Tube Size

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

Bed Weight	Tube Volume	Minimum Elution Vol.	Bed Capacity*
50-100 mg	1 mL	100-200 µL	2.5-10 mg
500 mg	3 mL	1-3 mL	25-100 mg
0.5-1 g	6 mL	2-6 mL	25-100 mg
2 g 5 g 10 g	12 mL 20 mL 60 mL	10-20 mL 20-40 mL 40-100 mL	0.1-0.2 g 1.25-2.5 g 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.
- The 10 mL LRC (large reservoir cartridges) are ideal for preparing larger sample volumes with smaller bed weights (25-100 mg). The packed section has the same diameter like a 1 mL tube.

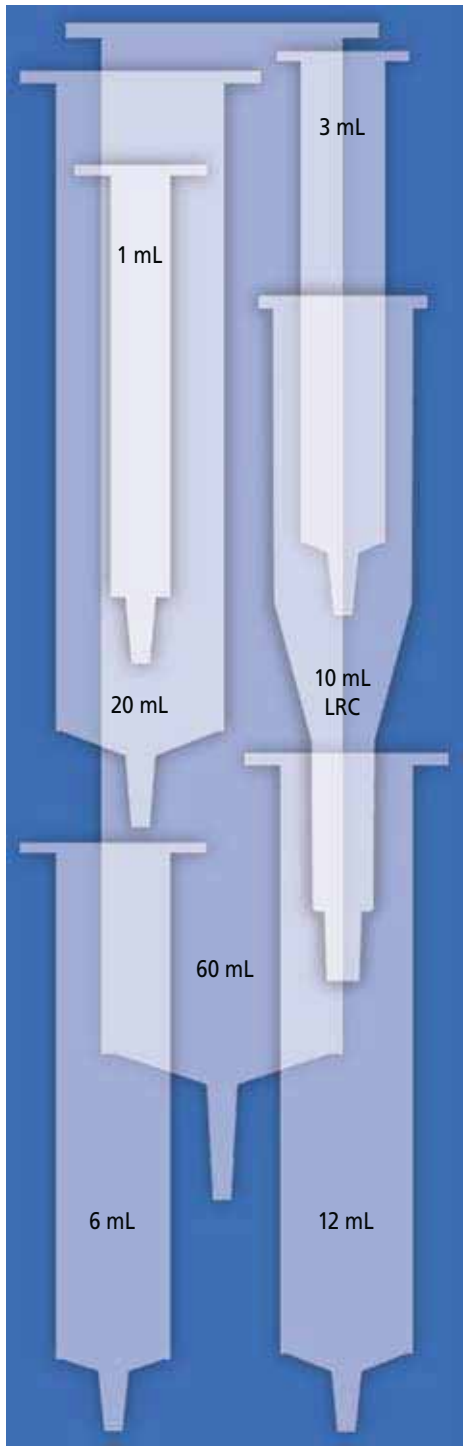
FREE SPE MultiPaks for Method Development

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

Available SPE MultiPaks

- HybridSPE-PPT
- Supel-Select HLB
- Supel-Tips
- SupelMIP
- Dispersive SPE (dSPE)
- Discovery Reversed-Phase
- Discovery Normal-Phase
- Discovery Ion-Exchange
- Discovery DSC-MCAX (Mixed-Mode Cation Exchange)
- Discovery DPA-6S (Polyamide)
- Supelclean ENVI-Carb (Graphitized Carbon)
- Discovery Ag-Ion
- Supelclean Dual Layer (for multi-residual pesticide analysis)
- Supelclean PSA

Most common SPE hardware:
Polypropylene SPE tubes with PE Frit



Actual size of SPE tubes

To learn more about SPE MultiPaks, or to request a FREE SPE MultiPak sample, please visit sigma-aldrich.com/spe or contact Technical Service at 800-359-3041/814-359-3041.

SPE Tubes and Specialty Hardware Quick Look-Up Guide

Additional Tubes & Cartridges

Glass SPE Tubes with PTFE & SS Frits (pg. 25 and 26)

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

VersaPure® PrePacked Büchner Funnels (21 and 26)

Used for large scale purification (medicinal chemistry purification)

Reversible SPE Tubes (pg. 21, 22, and 26)

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

Supel-Tips SPE (pg. 20)

Ideal for the micro-extraction of small & biological macromolecules

Rezorian Cartridges (pg. 21 and 26)

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

Discovery SPE 96-Well Plates (pg. 19 and 20)

For high throughput sample preparation

Dispersive SPE (pg. 24)

Custom pre-washed salt sorbent vials for dispersive SPE

Custom Capabilities

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

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 & Standard Vacuum Manifold (pg. 27)

Uses disposable liners that prevent cross-contamination

Visiprep 5-Port Flask Manifold (pg. 27)

Collects the SPE eluate in round flasks for easy rotary evaporation

Preppy™ Vacuum Manifold (pg. 28)

Most economical

PlatePrep Vacuum Manifold (pg. 30)

For 96-well SPE
Useful for stacking SPE tubes,

ENVI-Disk™ Holder (pg. 31)

Used with 47 mm SPE disks
Simple racks for using SPE

Visi-1™ Single SPE Tube Processor (pg. 25)

For processing very few SPE samples

Visiprep Large Volume Sampler (pg. 28)

For processing larger sample volumes

Visidry™ Drying Attachment& (pg. 28)

For drying SPE tubes or evaporating SPE eluate

Large Volume Reservoirs & Tube Adapters (pg. 25)

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

SPE Elution Rack (pg. 28)

Simple racks for using SPE under gravity flow

KNF Laboport® Vacuum Pumps (inquire)

Provides vacuum source for vacuum manifolds

Trap Kit and Vacuum Gauge Bleed Valve (pg. 29)

Additional vacuum accessories

NEW! HybridSPE - Precipitation Technology

Winner of the SelectScience.net Scientists' Choice Award for Best New Separations Product in 2008

HybridSPE-Precipitation (HybridSPE-PPT) combines the simplicity of protein precipitation with the selectivity of solid phase extraction (SPE) for the targeted removal of phospholipids in biological plasma/serum (Figure 1). The technology utilizes a zirconia-coated particle, and exhibits selective affinity towards phospholipids while remaining non-selective towards a range of basic, acidic, and neutral compounds. The phospholipid retention mechanism is based on a highly selective Lewis acid-base interaction between the proprietary zirconia ions (functionally bonded to the HybridSPE stationary phase) and the phosphate moiety consistent with all phospholipids (Figure 2).

Features & Benefits:

- Merges both protein precipitation & SPE
  - Offers the simplicity and generic nature of protein precipitation
  - Selectivity approaches SPE via the targeted removal of phospholipids
- 2-3 step generic procedure
- 100% removal of phospholipids and precipitated proteins
- Minimal to no method development required
- Available in 96-well and 1 mL cartridge dimensions

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

1) **Precipitate Proteins** by adding 100 µL plasma or serum to the HybridSPE-PPT plate followed by 300 µL 1% formic acid in acetonitrile. Add I.S. as necessary.

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

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

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

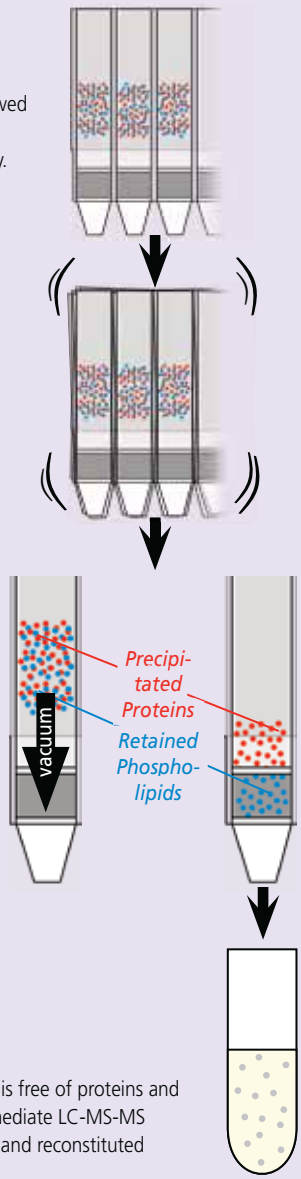
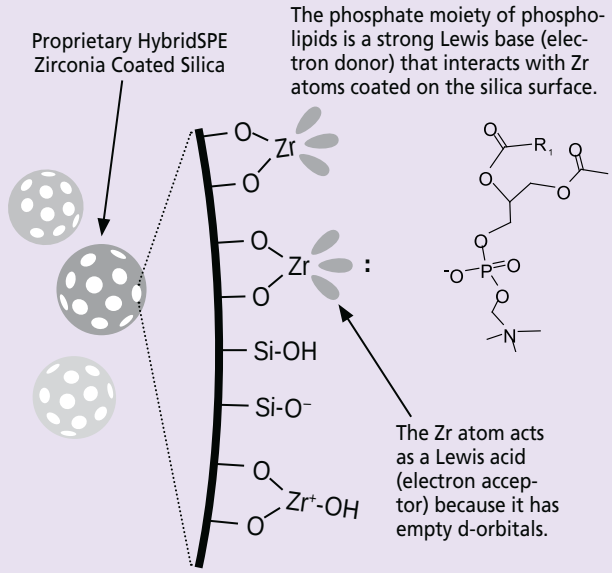


Figure 2. Lewis Acid-Base Interactions Between HybridSPE Zirconia Ions and Phospholipids



Description	Qty.	Cat. No.
HybridSPE-PPT Products		
96-well Plate, 50 mg/well	1	575656-U
1 mL Cartridge, 30 mg/well	100	55261-U
Related Products		
96-well Protein Precipitation Filter Plate	1	55263-U
Supelco PlatePrep Vacuum Manifold	1	57192-U
96 Square/Deep Well Collection Plate, 0.35 mL, PP	50	575651-U
96 Square/Deep Well Collection Plate, 0.5 mL, PP	50	575652-U
96 Square/Deep Well Collection Plate, 2 mL, PP	50	575653-U
96 Square Well Pierceable Cap Mats	25	575656-U

sigma-aldrich.com/hybridspe-ppt

NEW! Supel-Select HLB SPE

Sample Prep Performance at the Price you Desire

Supelco Supel-Select HLB SPE is a hydrophilic modified styrene-based polymer developed for the solid phase extraction of a highly broad range of compounds from aqueous samples. The retention mechanism is predominately based on reversed-phase interaction. However, because the phase is hydrophilic modified, the phase is also selective for more polar compounds (HLB: Hydrophilic Lipohilic Balance). Examples of more polar compounds that are retained and recovered on Supel-Select HLB include (but not limited to): pyridoxine (logPo/w -0.56), riboflavin (logPo/w -2.02), biotin (logPo/w 0.11).

Features & Benefits:

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

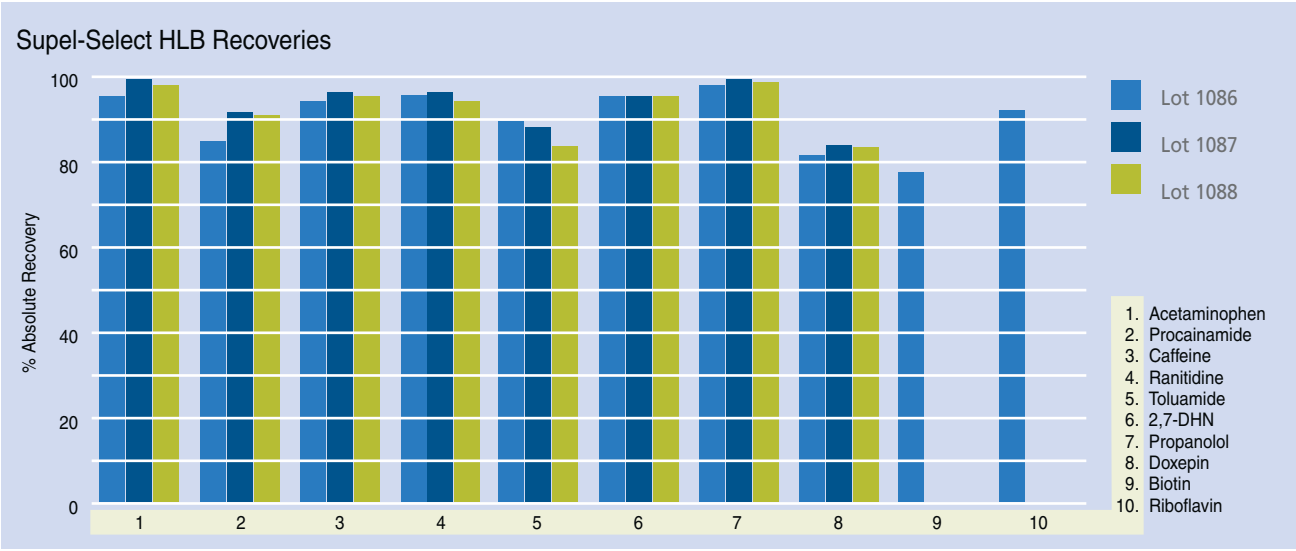
P001336

Phase Chemistry:	Hydrophilic modified styrene polymer
pH Compatibility:	0-14
Particle Size:	55-60 µm
MS Suitable:	Yes
Surface Area:	400-410 m^2/g
Pore Volume:	0.88 mL/g
Pore Size:	87 Å

High & Reproducible Recoveries

Supel-Select HLB SPE allows users to extract a broad range of compounds using a single sorbent and generic methodology.

Analyte recovery was high across all the compounds tested, and results were highly reproducible across three production lots.

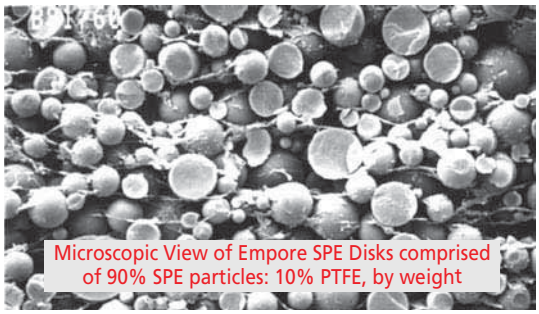


Description	Qty/Pk.	Cat. No.
Supel-Select HLB SPE		
30 mg/1 mL	100	54181-U
60 mg/3 mL	50	54182-U
200 mg/6 mL	30	54183-U
500 mg/12 mL	20	54184-U
1 g/20 mL	20	54186-U
Supel-Select HLB 96-well SPE		
10 mg/ well	1	Inquire
30 mg /well	1	575661-U
60 mg/ well	1	575662-U

sigma-aldrich.com/supel-select

NEW! Empore Solid Phase Extraction (SPE) Products

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



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

E001071

Save Time & Money with Empore SPE

Reduced SPE bed mass = Reduced SPE solvent & elution volumes

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

Dense & uniform extraction medium = NO SPE channeling/voiding

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

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

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

Available Formats:

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

E001070

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

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

E001068

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

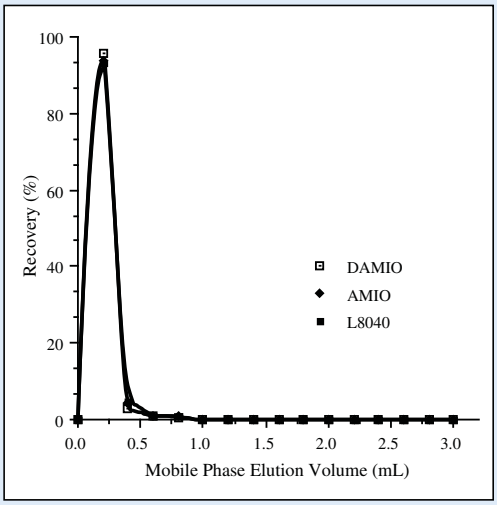
The Empore SPE cartridge line is packed with a PTFE membrane enmeshed with SPE particles. Layered above the SPE membrane is a polypropylene pre-filter to prevent particulates from reaching the underlying membrane. The dense particle packing and uniform distribution within the Empore membrane offers outstanding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

E001069

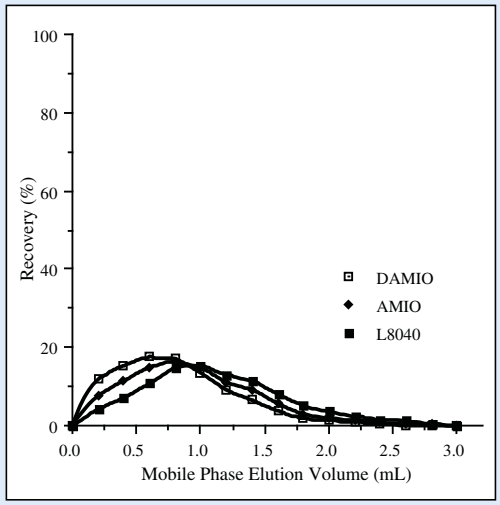
Recovery, Precision, & Elution Volume Profile of Empore SPE

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

SPE Empore Membrane (4 mm/1 mL)



SPE Packed Column (100 mg/1 mL)



	Precision (between-run, n = 15)						Recovery (at 300 µg/mL)	Sensitivity (lowest limit of quantitation)
	Mean µg/mL	SD µg/mL	CV %	Mean µg/mL	SD µg/mL	CV %		
AMIO	0.415	0.015	3.7	3.06	0.094	3.1	92-95%	0.05 µg/mL
DAMIO	0.412	0.013	3.3	3.06	0.096	3.2	90-93%	0.05 µg/mL

SD = standard deviation
CV = coefficient of variation

Empore Solid Phase Microextraction Products

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

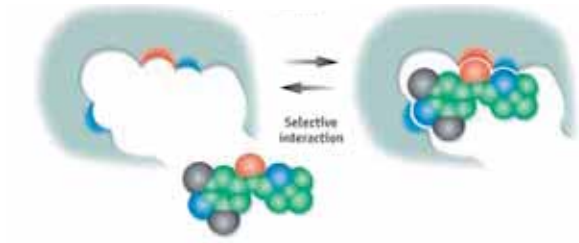
sigma-aldrich.com/empore

NEW! SupelMIP SPE – Molecularly Imprinted Polymers

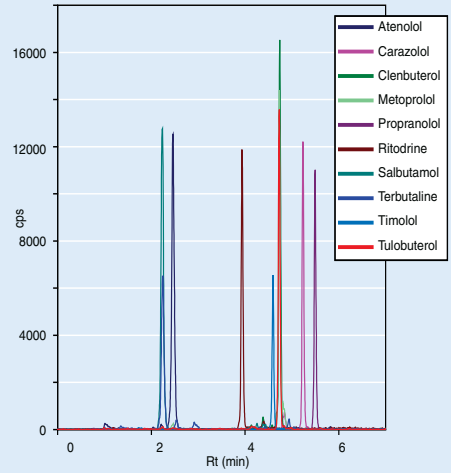
SupelMIP SPE phases were developed by MIP Technologies AB, which is one of the leading authorities and commercial pioneers of molecularly imprinted polymers for process scale separations, analytical chromatography, and sample preparation.

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

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



ß-agonists and ß-blockers (1 ng/mL spike) in Urine and Wastewater using SupelMIP SPE (53223-U)



Note: Clenbuterol and Tulobuterol were spiked at the levels of 0.1 ng/mL.

Key Features & Benefits:

- Achieve lower detection limits through superior selectivity
- Reduce ion-suppression
- Save time and reduce cost via robust and rapid methodology
- Minimal to no method development required
- Stable at broad pH ranges and high temperatures
- Stringent quality control conditions

SupelMIP Phases & Methods available for:

- **PAHs** (polyaromatic hydrocarbons) in edible oils
- **Nitroimidazoles** in milk, eggs, and other food matrices
- **Non-steroidal anti-inflammatory drugs** (NSAIDs) in wastewater and other sample matrices
- **Fluoroquinolones** in bovine kidney, honey, and milk
- **Amphetamine** and related compounds in urine
- **Chloramphenicol** in milk, plasma, honey, urine, and shrimp/prawns
- **NNAL** (4-(N-nitrosomethylamino)-1-(3-pyridyl)-1-butanol) in urine
- **TSNAs** (Tobacco Specific Nitrosamines) in urine and tobacco
- **ß-agonists** and **ß-blockers** in tissue, urine, and wastewater
- **Clenbuterol** in urine
- **Triazines** in water
- **Riboflavin** in milk

sigma-aldrich.com/supelmip

Analyte	Lower Limit of Quantitation (ng/mL, ppb, or µg/kg) 1 mL Horse Urine	10 mL Wastewater
Atenolol	0.1	0.01
Carazolol	0.1	0.01
Metoprolol	0.1	0.01
Propranolol	0.1	0.01
Timolol	0.1	0.01
Clenbuterol	0.02	0.002
Ritodrine	0.05	0.005
Salbutamol	0.1	0.01
Terbutaline	0.2	0.02
Tulobuterol	0.005	0.0005

SupelMIP SPE	25 mg/3 mL pk 50	25 mg/10 mL (LRC)▲ pk 50
PAHs	52773-U●	—
Nitroimidazoles	52734-U	—
NSAIDs	52769-U	—
Fluoroquinolones	53269-U	—
Amphetamines	53228-U	—
Clenbuterol	—	53201-U
Beta-agonists (class selective)	53225-U	53202-U
Beta-blockers (class selective)	53213-U	53218-U
Full Beta-receptors (beta-agonists and beta-blockers)	53223-U	53224-U
Chloramphenicol	53209-U	53210-U
NNAL (4-(methylnitrosamino)-1-(3-pyridyl)-1-butanol)	53203-U	53206-U
TSNAs (4 tobacco specific nitrosamines: NNK, NNN, NAB, NAT)	53222-U●	53221-U■
Riboflavin (vitamin B2)	—	53207-U
Triazines (class selective)	—	53208-U

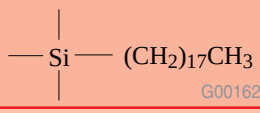
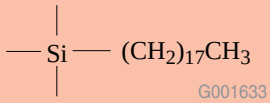
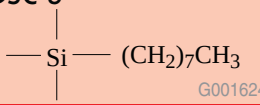
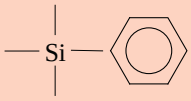
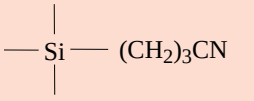
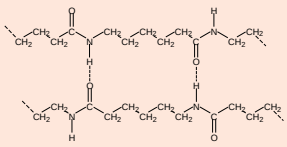
▲ LRC = large reservoir cartridge
● 50 mg/3 mL, pk 50
■ 50 mg/10 mL (LRC), pk 50

Discovery SPE

Reversed-Phase

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

For Discovery silica specifications, see pg. 2. For general guidelines on reversed-phase SPE, see pg. 32.

DSC-18  G001625	• 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	• 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	• 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	• 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	• 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	• 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
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
Bulk packing	100 g	52600-U	52623-U	57223-U	57227-U	57222-U	[10] 52633-U

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

Discovery SPE

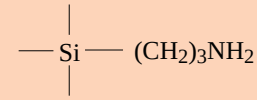
Ion-Exchange & Mixed-Mode

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. 33.

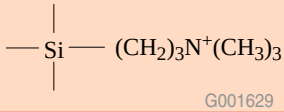
DSC-NH$_2$



G001631

- 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

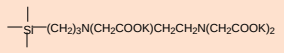
DSC-SAX



G001629

- A polymerically bonded quarternary amine that remains charged at all pH levels
- Commonly used when extracting weaker cations (e.g., carboxylic acids) that may not bind strongly enough to weaker anion-exchangers
- Selectivity can be modified by changing the counter ion with the appropriate buffer during conditionin
- Counter ion Cl^-

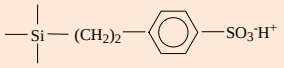
DSC-WCX



G001632

- A polymerically bonded carboxy propyl phase with a K^+ 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

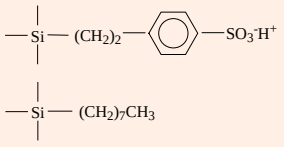
DSC-SCX



G001630

- 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

- Packed bed contains both octyl (C8) and benzene sulfonic acid (SCX) bondings. (H^+ as counterion)
- Developed for superior selectivity/sample cleanup when isolating basic compounds from biological fluids
- Dual retention mechanisms broadens retention for a range of neutral, basic, acidic and zwitterionic compounds
- Greater ion-exchange capacity for isolating polar basic and zwitterionic compounds
- Can be used to fractionate basic/zwitterionic compounds from acidic and neutral compounds

Discovery Ion-Exchange SPE Products

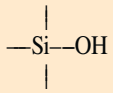
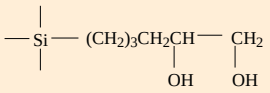
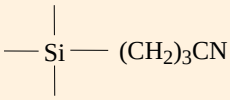
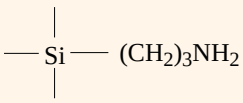
Description	Qty./Pk	DSC-NH$_2$	DSC-SAX	DSC-WCX	DSC-SCX	DSC-MCAX
Discovery SPE Tubes						
50 mg/1 mL	108	52635-U	52661-U	52737-U	52684-U	52781-U
100 mg/1 mL	108	52636-U	52662-U	52739-U	52685-U	52782-U
500 mg/3 mL	54	52637-U	52664-U	52741-U	52686-U	52783-U
500 mg/6 mL	30	52638-U	52665-U	52742-U	52688-U	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	–
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
Bulk packing	100 g	57212-U	57214-U	57228-U	57221-U	–

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. 34.

DSC-Si 	• Unbonded acid washed silica sorbent ideal for normal-phase SPE and other modified flash techniques • Considered the most polar normal-phase sorbent available • Excellent capacity for purifying solution phase CombiChem reactions when removing target molecules from reaction by-products and excess reagents • Available in Büchner Funnel configurations for easy scalability
DSC-Diol  G001627	• 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  G001626	• Monomerically bonded, cyanopropyl (7% C), endcapped • Can behave as either reversed-phase or normal-phase • Ideal for very hydrophobic analytes that may be irreversibly retained on more hydrophobic sorbents such as DSC-18 • Less retentive than DSC-Si or DSC-Diol when used as normal-phase (organic matrices such as hexane or oils) • Allows for the rapid release of very polar molecules irreversibly retained on very polar sorbents
DSC-NH$_2$  G001631	• 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-CN	DSC-Si	DSC-Diol	DSC-NH$_2$
Discovery SPE Tubes					
50 mg/1 mL	108	52652-U	52652-U	52747-U	52635-U
100 mg/1 mL	108	52653-U	52653-U	52748-U	52636-U
500 mg/3 mL	54	52654-U	52654-U	52751-U	52637-U
500 mg/6 mL	30	52655-U	52655-U	52752-U	52638-U
1 g/6 mL	30	52656-U	52656-U	52753-U	52640-U
2 g/12 mL	20	52657-U	52657-U	Custom	52641-U
5 g/20 mL	20	52658-U	52658-U	Custom	52642-U
10 g/60 mL	16	52659-U	52659-U	Custom	52644-U
Discovery SPE 96-Well Plates					
100 mg/well	1	575609-U	575609-U	575636-U	575615-U
50 mg/well	1	575608-U	575608-U	575637-U	575616-U
25 mg/well	1	575607-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
Bulk packing	100 g	52651-U	52651-U	57229-U	57212-U

For a complete list of available Büchner funnels, see page 21.

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. 32.

ENVI-18	• 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	• 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	• Monomerically bonded, octadecyl (10% C), endcapped • For reversed-phase extraction of nonpolar to moderately polar compounds.
ENVI-8	• Available in glass tubes with PTFE frits • High 14% C loading for increased binding capacities and higher recoveries • Higher carbon loading also offers greater resistance to extreme pH conditions • Excellent for cleaning, extracting & concentrating pollutants from aqueous environmental samples
LC-8	• Monomerically bonded, octyl (7% C), endcapped
ENVI-Chrom P *(polystyrene divinylbenzene)*	• Styrene/divinylbenzene co-polymer resin: Particle Size: 80-160 µm; Spherical Shape; Pore Size: 110-175 Å; Surface Area: 900 m^2/g • Highly crosslinked, neutral, specially cleaned styrene-divinylbenzene resin used to retain hydrophobic compounds with some hydrophilic functionality under the reversed-phase mechanism • Highly resistant to extreme pH conditions • Typical applications include aromatic and phenolic compounds from aqueous sample matrices • Used for priority pollutant phenols from aqueous samples
ENVI-Carb & ENVI-Carb II *(graphitized carbon black)*	• Surface Area: 100 m^2/g, Particle Size: 100/400 mesh (ENVI-Carb-II: 100/140 mesh) • Extreme affinity for organic polar and non-polar compounds from both non-polar and polar matrices when used under reversed-phase conditions • Carbon surface comprised of hexagonal ring structures, interconnected and layered into graphitic sheets • Non-porous nature of the carbon phase allows for rapid processing, adsorption does not require analyte dispersion into solid phase pores • Independent investigators have found ENVI-Carb extremely useful for the rapid sample preparation of over 200 pesticides from various matrices including ground water, fruits, and vegetables (publication T196900)
LC-4 (Wide Pore)	• Butyldimethyl, wide pore (500 Å), endcapped • Larger pore size to accommodate larger macromolecules (e.g., proteins and peptides) • Commonly used for desalting proteins and peptides in aqueous samples
Hisep™	• Hydrophobic sites shielded by a hydrophilic surface for protein exclusion during sample load • Hydrophobicity similar to C8
LC-Ph	• Monomerically bonded, phenyl (5.5% C), endcapped
LC-CN	• Monomerically bonded, cyanopropyl (7.5% C), endcapped

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

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. 33 & 34.

LC-SAX	• A strong anion exchanger with pKa of 10.1 and 10.9 • Quarternary amine, Cl^- counter-ion
LC-SCX	• Aliphatic sulfonic acid, Na^+ counter-ion, endcapped
LC-WCX	• Carboxylic acid, Na^+ counter-ion
LC-NH_2	• Monomerically bonded, aminopropyl (5% C)
PSA	• Polymerically bonded, ethylenediamine-N-propyl phase that contains both primary and secondary amines
ENVI-Florisil	• Magnesium silicate, mesh: 100/200, available with PTFE or stainless steel frits • Tested for US Environmental Protection Agency (EPA) Contract Laboratory Program (CLP) statement of work for pesticides • Highly polar material that strongly adsorbs polar compounds from non-polar matrices under normal-phase conditions • Typical applications include alcohols, aldehydes, amines, herbicides, pesticides, PCBs, ketones, nitro compounds, organic acids, and phenols
Dual Layer Florisil/Na_2SO_4	• Dual layer glass SPE tube that contains Na_2SO_4 (upper layer) and Florisil (magnesium silicate; lower layer) separated and packed with PTFE frits • Florisil, activated, size- 60/100 mesh (150-200 mm), Na_2SO_4 Purity- 99.99 %, Density- 2.68 g/mL • Excellent for removing/isolating polar compounds from organic matrices • Na_2SO_4 layer aids in removing aqueous sample residues that may hinder Florisil performance and/or subsequent GC analysis • Suitable for the determination of the hydrocarbon oil index in water (surface, waste, and sewage treatment plants) by GC-FID analysis according to European Standard EN ISO 9377-2:2000 (enclosed in the Extraction Kit for EN ISO 9377-2 Cat.No. 68172) • 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, only suitable for PP SPE tubes) and Visiprep SPE Vacuum Manifolds for processing larger volume samples
LC-Florisil	• Magnesium silicate, mesh: 100/120
LC-Alumina A, N, & B	• Alumina-A for acidic pH (~5) • Alumina-N for neutral pH (~6.5) • Alumina-B for basic pH (~8.5) • Brockman Activation I for all Alumina SPE products, mesh: 60/325
LC-CN	• Monomerically bonded, cyanopropyl (7% C), endcapped
LC-Si	• Silica gel
LC-Diol	• Monomerically bonded, Diol (7% C), endcapped

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

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 57108[1]		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						
	Hisep		57076-U						
	LC-Ph	504599	505269						
	LC-CN	504386	57013	57056			57141	57142	
	LC-Diol	504718	57016						
Normal-Phase	ENVI-Florisil		57058[2]	57046[3]	57053[3] 54095[1]				
	Dual Layer Florisil/ Na_2SO_4				52582-U[1,9] 54116-U[2,9]				
	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
	LC-Si	504041	505048	505374	57051 54335-U[1]	57116	57133	57134	57200
Ion Exch.	$LC-NH_2$	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 next page.

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
0.5 g/0.5 g/20 mL, pk 20	54217-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/$LC-NH_2$	
0.5 g/0.5 g/3 mL, pk 20	54332-U
0.5 g/0.5 g/20 mL, pk 20	54216-U
0.5 g/0.5 g/20 mL, pk 300	54024-U
0.5 g/0.5 g/6 mL, pk 30	54035-U
ENVI-Carb/NH_2/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/Na_2SO_4	
Glass tubes, PTFE frits, 2 g/2 g/6 mL, pk 48	52582-U
2 g/2 g/6 mL, PP, pk 48	54116-U

SPE Method Development Kits

Supelclean SPE Method Development Kits

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

Supelclean SPE Method Development Kits

SPE Method Development Kit	Kit A	Kit B	Kit C	Kit NP-3	Kit IX-3
Supelclean Packing			**Sorbent Qty./Tube Size**		
LC-Si	500 mg/3 mL	100 mg/1 mL	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**

For more information about FREE SPE SAMPLES, see pg. 5.

96-Well SPE Method Development Plate

Supelco 96-Well SPE method development plates contain an assortment of SPE phase chemistries ideally suited for method development. The mix of phase chemistries contained within this 96-well SPE plate allows researchers to screen for analyte retention, recovery, and selectivity when achieving one's sample prep objectives.

Description	Qty.	Cat. No.
96-well SPE Method Development Plate		
BAN, 25 mg/well (configured for extracting basic, acidic and neutral compounds)	1	577522-U

	1	2	3	4	5	6	7	8	9	10	11	12
A	Discovery DSC-PS/DVB (polystyrene divinyl benzene)[1]											
B	Discovery DSC-18 (tC18)[1]											
C	Discovery DSC-8 (C8)[1]											
D	Discovery DSC-CN (cyanopropyl)[1]											
E	Discovery DSC-MCAX (mixed-mode cation exchange)[1,2]											
F	Discovery DSC-WCX (weak cation exchange)[2]											
G	Discovery DSC-SAX (strong anion exchange)[3]											
H	Discovery DSC-NH_2 (aminopropyl weak anion exchange)[3]											

[1] Reversed-phase; [2] Cation-exchange; [3] Anion-exchange

Specialty Products for Pharmaceutical Analysis

Supelco SPE 96-well Plates


P001248

Supelco SPE 96-well plates answer the challenge of high throughput sample prep for pharmaceutical bioanalysis. These plates are packed with our high quality Discovery SPE line, Supel-Select HLB phase (see pg. 9), and our new and innovative HybridSPE-Precipitation technology (see pg. 8). The uniform flow dynamics inherent with well plate technology offers a high level of reproducibility and throughput while maintaining excellent recoveries for increased sensitivity.

96-Well Plate Specifications:

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

Supelco SPE 96-well Plates

Phase	25 mg/well	50 mg/well	100 mg/well
HybridSPE-PPT	–	575656-U	–
Supel-Select HLB	–	575661-U◆	575662-U▼
Discovery DSC-18	575601-U	575602-U	575603-U
Discovery DSC-18Lt	575604-U	575605-U	575606-U
Discovery DSC-MCAX	575639-U	575640-U	575641-U
Discovery DSC-8	–	575628-U	575627-U
Discovery DSC-Ph	575632-U	575631-U	575630-U
Discovery DSC-CN	575626-U	575625-U	575624-U
Discovery DSC-PS/DVB	575610-U	575611-U	–
Discovery DSC-Si	575607-U	575608-U	575642-U▲ 575609-U
Discovery DSC-Diol	575638-U	575637-U	575636-U
Discovery DSC-NH_2	575617-U	575616-U	575615-U
Discovery DSC-SAX	575620-U	575619-U	575618-U
Discovery DSC-WCX	575635-U	575634-U	575633-U
Discovery DSC-SCX	575623-U	575622-U	575621-U

▲PE bottom frit (5 µm porosity)
◆Actual bed weight = 30 mg/well
▼Actual bed weight = 60 mg/well

Supel-Tips SPE – Microscale Extraction

The Supel-Tips SPE product line is designed for the microscale extraction, concentration, and recovery of small molecules and biological macromolecules. These 10 µL pipette tips containing a sorbent bed bonded at the working end of the tip using an inert high-purity adhesive. The bed acts as a solid phase extraction medium to adsorb molecules of interest from the sample matrix. Subsequently, the concentrated and desalted analytes are eluted for downstream analysis.

Supel-Tips Offer:

- Superior recovery
- Exceptional binding capacity and enhanced affinity
- Excellent sorbent bed stability for cleaner samples
- Fast and effective analyte retention/elution


P001231

sigma-aldrich.com/pipette-tips

Supel-Tips

Description	Supel-Tip C18	Supel-Tip Carbon	Supel-Tip Zr	Supel-Tip Ti
Pipette Tip	10 µL, PP	10 µL, PP	10 µL, PP	10 µL, PP
Application	peptides and proteins	Oligosaccharides and other sugar containing **macromolecules**	Phosphopeptides and other phosphate **containing molecules**	Phosphopeptides and other **phosphate containing molecules**
Adsorbent	C18	Carbon	Zirconia-silica composite	Titania-silica composite
Particle Size	50-60 µm	50-60 µm	50-60 µm	50-60 µm
Pore Size	200 Å	175 Å	300 Å	300 Å
Capacity (per tip)	Insulin, Chain B, Oxidized – 17 µg; ß-amyloid – 17 µg; Bradykinin, Fragment 1-7 – 7.6 µg	Maltohexose – 10.2 µg; Glycopeptide (mol. wt. 1300-3500) - > 10 µg	MPP1- 1 µg	MPP1- 1 µg
Cat. No. (pk. 96)	TPSC18	54227-U	54266-U	54263-U

Specialty Products for Purification

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

Description	I.D. x H:	12.5 g (45 mL) - pk 6 55 mm x 30 mm	25 g (90 mL) - pk 6 70 mm x 40 mm	50 g (174 mL) - pk 3 90 mm x 48 mm	100 g (410 mL) - pk 3 110 mm x 66 mm
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

Polymer SAX Rezorian Cartridge

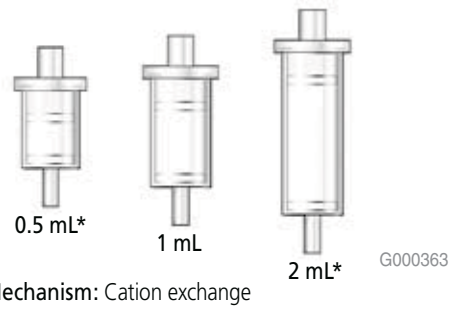
Retention Mechanism: Anion exchange

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

*Available as custom only.

Specialty Products for Environmental Analysis

Supelclean Coconut Charcoal SPE Tube

9940374

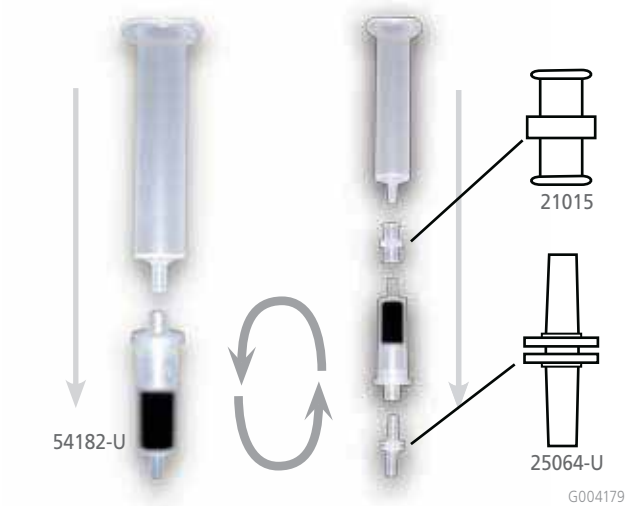
- Developed specifically for EPA Method 521 – Nitrosamines in Drinking Water
- Activated coconut charcoal stationary phase – particle size: 80/120 mesh
- Quality controlled for low fines and nitrosamine recovery

Description	Qty.	Cat. No.
Supelclean Coconut Charcoal SPE Tube, 2 g/6 mL	30	57144-U
Female Luer Coupler	20	2105
Male Luer Coupler	20	25064-U

Supelclean ENVI-Carb Plus Reversible SPE

- Spherical carbon particles (carbon mol sieve) developed for the SPE of highly polar compounds from aqueous samples as drinking or ground water
- Offers extreme affinity to organic polar and non-polar compounds from both non-polar and polar matrices when used under reversed-phase conditions.
- Strong high surface spherical particles which are less friable (fines) than traditional graphitized carbon blacks
- Examples of highly polar compounds recovered:
  - Acephate (LogPo/w: -0.85)
  - Phenol (LogPo/w: 1.51)
  - 1,4-dioxane (LogPo/w: -0.27)
  - Oxamyl (LogPo/w: -1.2)
- When used in conjunction with an SPE vacuum manifold, a male luer coupler (25064-U), female luer coupler (21015) and empty SPE tube(s) are required but not included.

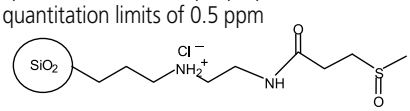
Description	Qty.	Cat. No.
Supelclean ENVI-Carb Plus Reversible SPE Tube, 0.4 g/1 mL	30	54182-U
Female Luer Coupler	20	21015
Male Luer Coupler	20	25064-U



G004179

Supelclean Sulfoxide SPE

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



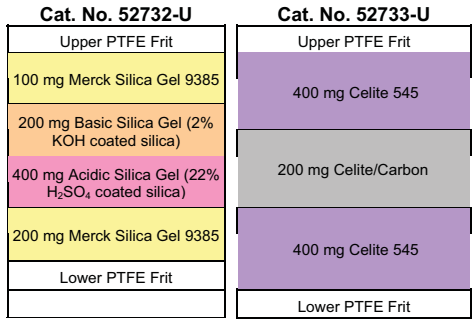
G004219

P001240

Description	Qty.	Cat. No.
Supelclean Sulfoxide Glass SPE Tube, 6 g/20 mL	5	55252-U
Supelclean Sulfoxide SPE, 3 g/6 mL	30	55253-U
Supelclean Sulfoxide, Bulk	100 g	55254-U
Empty Glass SPE Tube (17 mm I.D. x 137 mm L) with PE frit, 20 mL, with PE frit, luer cap, and screw-top cap	5	55255-U
Frit Insertion Tool for 20 mL Glass SPE tube	1	55257-U
Disposable PTFE liners	100	57059
Large volume reservoir (25 mL) for 6 mL SPE tubes, PP	30	54258-U
Large volume reservoir (25 mL) for 6 mL SPE tubes, PTFE	3	54259-U

Multi-Layer SPE Tubes for EPA Method 8290

Configured for EPA Method 8290 – PCDDs and PCDFs by HRGC/HRMS



The sample cleanup employed in EPA Method 8290 requires a series of hand-packed glass chromatography steps involving:

1. A multi-layer silica gel glass column
2. A sodium sulfate/alumina glass column
3. A multi-layer celite 545-activated carbon glass column

Cat. No. 52732-U can be used in place of the required multi-layer silica gel glass column, and Cat. No. 52733-U can be used in place of the required multi-layer celite 545-activated carbon glass column.

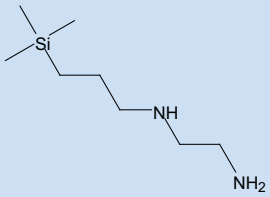
Note: The bed weights packed into these SPE tubes are smaller than what is described in EPA Method 8290. Therefore, to use these SPE tubes, sample volumes need to be scaled down accordingly.

Description	Qty.	Cat. No.
Multi-layer Silica Gel SPE Tube, glass, 6 mL	30	52732-U
Multi-layer Celite/Activated Carbon SPE Tube, glass, 6 mL	30	52733-U

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/NH_2 SPE products are also available. Please see pg. 18 for a listing.

ENVI-Carb-II/PSA  G002462	• Dual layer SPE tube that contains both Supelclean ENVI-Carb-II (upper layer) and PSA (lower layer) SPE sorbents (separated by PE frit) • Developed to offer superior cleanup when conducting multi-residue pesticide analysis in food (e.g., fruits, vegetables, etc.) • ENVI-Carb-II a graphitized non-porous carbon (100/140 mesh, surface area 100 m^2/g) that has a strong affinity towards planar molecules, and has been quality controlled specifically for the isolation/removal of pigments (e.g., chlorophyll and carotinoids) and sterols commonly present in fruits, vegetables, and other natural products • Supelclean PSA is a polymerically bonded, ethylenediamine-N-propyl phase that contains both primary and secondary amines • Supelclean PSA has a strong affinity and high capacity for fatty acids, organic acids, and some polar pigments and sugars • Tested for superior cleanliness using GC-FID and GC-MS
ENVI-Carb-III/SAX/PSA	• Tri-layer SPE tube that contains Supelclean ENVI-Carb-II (upper layer), SAX (middle layer) and PSA (lower layer) SPE sorbents (separated by PE frit) • Developed to offer superior cleanup when conducting multi-residue pesticide analysis in food (e.g., fruits, vegetables, etc.) • ENVI-Carb-II is a graphitized non-porous carbon (100/140 mesh, surface area 100 m^2/g) that has a strong affinity towards planar molecules, and has been quality controlled specifically for the isolation/removal of pigments (e.g., chlorophyll and carotinoids) and sterols commonly present in fruits, vegetables and other natural products • Supelclean PSA has a strong affinity and high capacity for fatty acids, organic acids, and some polar pigments and sugars • Supelclean SAX offers additional ion-exchange capacity for removing matrix components that may induce ion-suppression or enhancement during GC analysis
SAX/PSA	• Dual layer SPE tube that contains both Supelclean SAX (upper layer) and PSA (lower layer) SPE sorbents (separated by PE frit) • Supelclean SAX is a quarternary amine, Cl^- counter-ion • Supelclean PSA is a polymerically bonded, ethylenediamine-N-propyl phase that contains both primary and secondary amines • Ideal for removing matrix components (fatty acids, organic acids, polar pigments, and some sugars) when conducting multi-residue pesticide analysis in foods • In compliance with Luke and Luke II methods that use SPE to reduce matrix induced ion-suppression and enhancement when conducting GC analysis of pesticides in food
ENVI-Carb	• Surface Area: 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
PSA  G002460	• 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 (NH_2) in terms of selectivity, but has a much higher capacity due to presence of secondary amine (0.98-1.05 meq/g) • Strong affinity and high capacity for removing fatty acids, organic acids, and some polar pigments and sugars when conducting multi-residue pesticide analysis in foods • Has been shown to significantly reduce matrix-enhancement effects encountered during the GC analysis of food products • Bidendate nature of ligands allow for chelation

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

Specialty Products for Pesticide & FAME Analysis

Dispersive SPE (dSPE)

Dispersive SPE (dSPE), often referred to as the "QuEChERS" method (Quick, Easy, Cheap, Effective, Rugged, and Safe), is an emerging sample prep technique that is becoming increasingly popular in the area of multi-residue pesticide analysis in food and agricultural products.

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

Supelco now offers a Comprehensive Dispersive SPE Customization Service. To learn more visit sigma-aldrich.com/quechers

Dispersive SPE Products now Available for:

- *50 mL centrifuge tubes*
- *2 mL microcentrifuge tubes*
- *Glass vials*
- *A wide range for Sigma-Aldrich/Supelco salts, buffering agents, and SPE phases*

Supelco now carries a line of vials and centrifuge tubes containing pre-determined amounts of salts and SPE sorbents to support the most common method configurations used today.

Dispersive SPE (dSPE) Products

Description	Qty.	Cat. No.
Packed in 12 mL Greiner Centrifuge Tube (Greiner Cat. No. 163270)		
Citrate Extraction Tube	50	55227-U
4 g magnesium sulfate **(Cat. No. 63135)**		
1 g sodium chloride **(Cat. No. 71379)**		
0.5 g sodium citrate dibasic sesquihydrate **(Cat. No. 71635)**		
1 g sodium citrate tribasic dihydrate **(Cat. No. 32320)**		
Mg_2SO_4 Extraction Tube	50	55234-U
6 g magnesium sulfate **(Cat. No. 63135)**		
1.5 g sodium acetate **(Cat. No. 24,124-5)**		
PSA SPE Cleanup Tube 1	50	55228-U
900 mg magnesium sulfate **(Cat. No. 63135)**		
150 mg Supelclean PSA **(Cat. No. 52738-U)**		
PSA/C18 SPE Cleanup Tube 1	50	55229-U
900 mg magnesium sulfate **(Cat. No. 63135)**		
150 mg Supelclean PSA **(Cat. No. 52738-U)**		
150 mg Discovery DSC-18 **(Cat. No. 52600-U)**		
PSA/ENVI-Carb SPE Cleanup Tube 1	50	55230-U
900 mg magnesium sulfate **(Cat. No. 63135)**		
150 mg Supelclean PSA **(Cat. No. 52738-U)**		
15 mg Supelclean ENVI-Carb **(Cat. No. 57210-U)**		
PSA/ENVI-Carb SPE Cleanup Tube 2	50	55233-U
900 mg magnesium sulfate **(Cat. No. 63135)**		
150 mg Supelclean PSA **(Cat. No. 52738-U)**		
45 mg Supelclean ENVI-Carb **(Cat. No. 57210-U)**		

sigma-aldrich.com/quechers

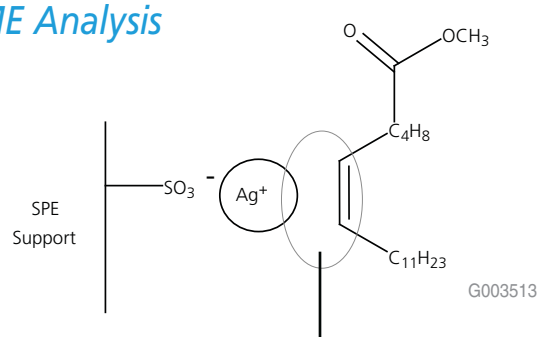
Discovery Ag-Ion SPE Tubes for cis/trans FAME Analysis

Retention Mechanism: Normal-phase

Sample Matrix Compatibility: Organic solvents, oils, and lipids

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

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



Charge-transfer complex between Ag^+ and unsaturated bond

Miscellaneous Specialty Products & SPE Accessories

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 pthalates and plasticizers
- Hygroscopic adsorbents (e.g. Florisil) can be easily heat treated/ activated (e.g., 105-120 °C oven, overnight) prior to use.

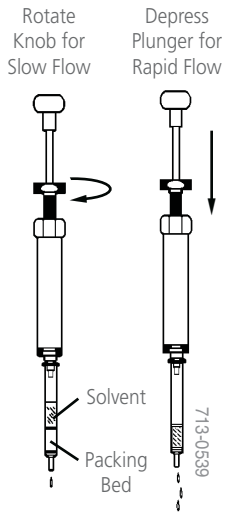
Description	Qty.	Cat. No.
Supelclean ENVI-18 SPE Tube		
glass hardware, PTFE frit, bed wt. 500 mg, vol. 6 mL	30	54331-U
Supelclean ENVI-8 SPE Tube		
glass hardware, PTFE frit, bed wt. 500 mg, vol. 3 mL	27	57106
glass hardware, PTFE frit, bed wt. 500 mg, vol. 6 mL	20	57107
Supelclean LC-Florisil SPE Tube		
glass hardware, PTFE frit, bed wt. 500 mg, vol. 6 mL	30	54333-U
glass hardware, PTFE frit, bed wt. 1 g, vol. 6 mL	30	54334-U
Supelclean LC-Si SPE Tube		
glass hardware, PTFE frit, bed wt. 1 g, vol. 6 mL	30	54335-U
Dual Layer Florisil/Na_2SO_4 SPE Tube		
bed A 2 g (Na_2SO_4), bed B 2 g (Florisil), vol. 6 mL	48	52582-U

Single SPE Tube Processor

Visi-1 processor - two rates of flow control

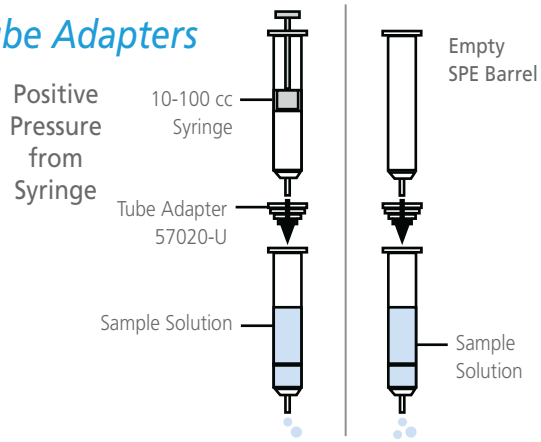
Our Visi-1 Single SPE Tube Processor provides precise flow control through a single 1 mL, 3 mL, or 6 mL SPE tube. There is no faster, more convenient, or more reliable method for processing one or a few samples.

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



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

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/N_2 line).

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

Large Volume SPE Reservoirs

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

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

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

SPE Accessories

Empty SPE Hardware & 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		54220-U (pk 100)	54221-U (pk 100)	54222-U (pk 100)	54223-U (pk 100)	57118-U	57119-U
Empty PP SPE Tube (no frits)		57240-U	57241	57242	57179	57021 (Qty. 12)	57022 (Qty. 12)
Empty Glass SPE Tubes with PTFE Frits, 20 µm porosity		–	–	504394*	–	–	–
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*	–	–	–
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	–

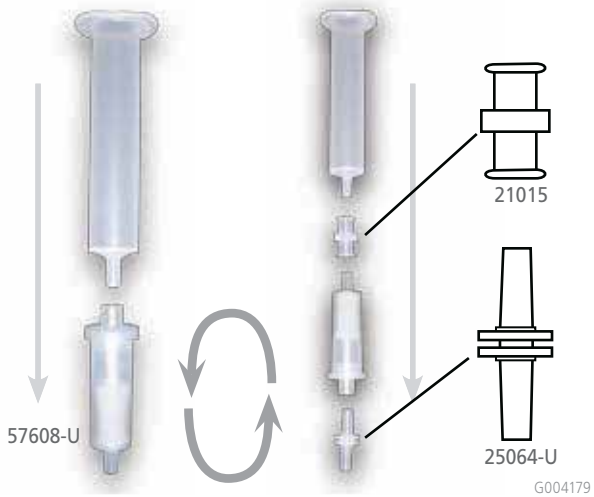
PP = Polypropylene; PTFE = Polytetrafluoroethylene; SS = Stainless steel; PE = Polyethylene

* Qty. of 24

Miscellaneous SPE Hardware & Accessories

Description	Qty.	Cat. No.
Empty Reversible SPE Tube, non-flourous PP, w/PE frits		
0.5 mL	50	57602-U
1.0 mL	50	57607-U
2.0 mL	50	57608-U
Empty Flangeless PP SPE Tubes w/PE Frits, 20 µm porosity		
1 mL	108	Inquire
3 mL	54	Inquire
6 mL	30	Inquire
Empty PP Rezorian Tube Kit w/PE Frits, luer plugs and caps		
1.0 mL	50	57609-U
5.0 mL	50	57613-U
Empty 96-well SPE Plates		
2 mL deep square well, w/PE frits	1	Inquire
1.25 mL round well, w/PE frits	1	Inquire
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 female luer fitting)	12	504351
Female Luer Coupler	20	21015
Male Luer Coupler	20	25064-U



SPE Accessories - Vacuum Manifolds

Visiprep & Visiprep DL SPE Vacuum Manifolds

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

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.

12-Port Visiprep DL Vacuum Manifold (57044)

Features & Benefits for both DL and standard models:

- Screw-type valves for SPE port for precise flow control
- Glass basin will not dissolve, fog, or discolor when exposed to solvents
- Legs on stand-alone cover allows user to easily rest cover on work surface when removed from vacuum manifold
- Screw type solvent resistant vacuum bleed gauge and valve offer better sealing and vacuum control. Valve takes ¼" 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)

Visiprep 5-Port Flask Manifold

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

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

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

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

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

P001063

P001072

SPE Processing Accessories

Supelco Preppy Vacuum Manifold

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

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

Description	Cat. No.
Preppy Vacuum Manifold	
12-Port Model	57160-U
Preppy Replacement Parts	
Cover with flow control valves and solvent needle guides	57158-U
Collection Vessel Racks	
For 2 or 4 mL vials	57159-U
For 15 or 40 mL vials	57162-U
Accessories	
Vacuum Gauge/Bleed Valve Assembly	57161-U

Visidry Drying Attachment

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

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

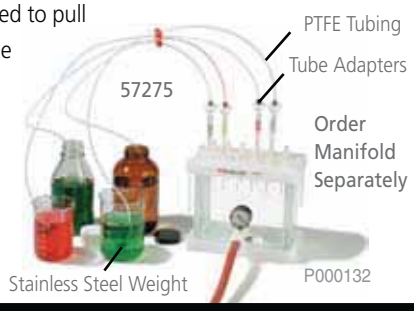
Description	Qty.	Cat. No.
Visidry Drying Attachment		
12-Port Model	1	57100-U
24-Port Model	1	57124
Replacement Parts for Visidry Drying Attachment		
Control Knobs	2	57095
Retaining "C" Clips	2	57096
Female Luer Plugs	12	57098

Replacement SPE Tube Adapters (57020-U) listed on p. 20.

VisiPrep Large Volume Samplers

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

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



Description	Qty.	Cat. No.
Visiprep Large Volume Sampler		
for 12 mL, 20 mL, or 60 mL SPE Tubes (3 adapters)[1]	1	57272
for 3 mL or 6 mL SPE Tubes (4 adapters)	1	57275
Replacement Parts		
1/8" PTFE Tubes, color-coded	4	57276
Nuts and Ferrules, color-coded	4	57277
Stainless Steel Weights	4	57278
Tube Adapters, 1/4-28 threads		
For 3 mL or 6 mL Tubes	4	57273-U
For 12 mL, 20 mL, or 60 mL Tubes	3	57274-U

[1]Also, 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

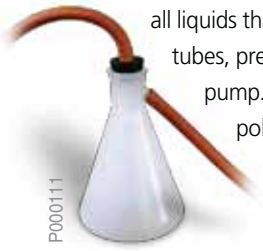
Replacement Parts and Optional Components for Visiprep Manifolds

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

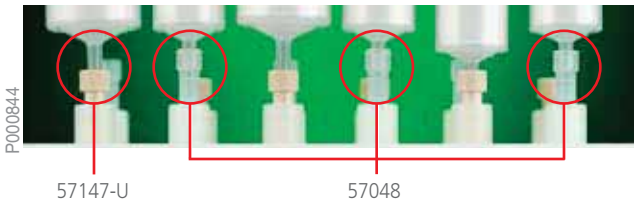
Install in-line for control of vacuum.

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

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.

P001252

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

Starter Kit Includes:

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

P000845

P001251

96-well SPE Plate

Acrylic Clear Top

96 Square Well Collection Plate or Disposable Reservoir/Waste Tray

Polypropylene Base (57194-U)

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

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.

P000116

The unit consists of a 1-liter sample funnel, a threaded screw clamp, a PTFE disk support, and a PTFE filter base/adapter with a vacuum attachment fitting. The filter base fits onto any standard 1-liter flask that has a 40/35 tapered ground glass neck. Use 25 x 250 mm test tubes to collect disk eluates. The flask and collection tubes are not included with the holder, but can be purchased separately.

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

[1] Order separately – not included with holder.

ENVI-Disk Holder Manifold

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


P000115

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


P000101

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

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

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

SPE Methodology & Useful Tips

Reversed-Phase SPE

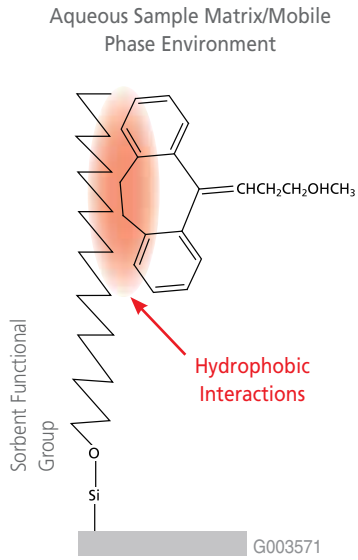
Retention Mechanism: **Non-polar or hydrophobic interactions**
- Van der Waals or dispersion forces

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



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

1. Sample Pre-treatment

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

When an analyte is in its neutral form, it becomes more hydrophobic and retention strengthens under reversed-phase conditions. Adjusting the sample pH to 2 pH units above or below the compound's 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.

2. Condition/Equilibration

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

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

3. Sample Load

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

4. Wash

Sample interferences are often co-retained with compounds of interest during sample load. A wash step is necessary to elute interferences without prematurely eluting compounds of interest. 5-20% methanol in water or sample pre-treatment buffer is typical for wash solvents.

5. Elution

Disrupt hydrophobic interactions between the analyte and sorbent functional groups with an organic solvent or solvent combination of sufficient non-polar character. Example elution solvents are 1-2 volumes of methanol or acetonitrile.

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

6. Eluate Post-treatment

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:

1. 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.).

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

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

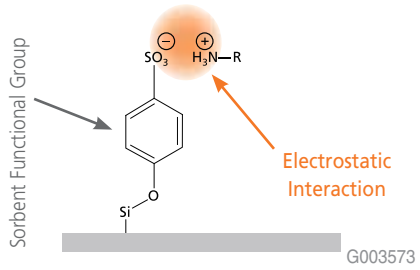
4. Reduce bed weight to minimize elution volume.

5. Increase bed weight to retain more polar compounds

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

7. A pre-conditioning solvent such as dichloromethane (or solvent used for elution) can be used before conditioning to remove any impurities on the SPE tube that can interfere with subsequent analysis.

SPE Methodology & 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:

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

For Anion Exchangers:

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

To change to a higher selective ion, pass 2-5 bed volumes of 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 (< 0.1M)

- 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 (> 1M); 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

1. 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.

2. Condition/Equilibration

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

3. Sample Load

Apply sample (from step 1) at a consistent and reduced flow rate of ~1-2 drops/second to ensure optimal retention. Mass transfer kinetics of ion-exchange SPE are slower than reversed-phase and normal-phase. Reduced flow rate is critical for consistent recovery.

4. Wash

Adequate control of pH and ionic strength should be maintained to prevent premature elution of the analytes of interest. Use buffer of appropriate pH (e.g. buffer used in sample pre-treatment) to remove polar interferences. More hydrophobic interferences can be removed using up to 100% methanol diluted in sample pre-treatment buffer.

5. Elution

Elute at a consistent and reduced flow rate of ~1-2 drops/second to ensure optimal compound desorption. The most common elution strategy is by pH manipulation. Also, most ion-exchangers exhibit some mixed-mode behavior. Addition of organic modifier is necessary to disrupt secondary reversed-phase interactions.

Examples:

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

Other elution strategies:

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

6. Eluate Post-treatment

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

SPE Methodology & Useful Tips

Normal-Phase SPE

Retention Mechanism: **Polar Interactions**
- Hydrogen bonding, pi-pi, dipole-dipole, and induced dipole-dipole

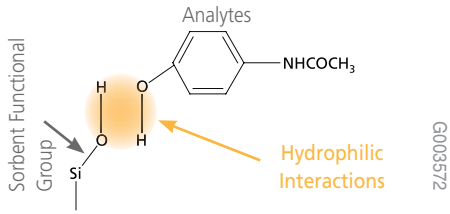
Sample Matrix: **Non-polar samples**
- Organic extracts of solids
- Very non-polar solvents
- Fatty oils, hydrocarbons

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

Non-polar sample matrix/
mobile phase environment



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

1. Sample Pre-treatment

Liquid samples should be initially extracted or diluted with a non-polar solvent such as hexane or a chlorinated solvent. Soil, sediment, and other solid samples are initially extracted (soxhlet or sonication) with a non-polar solvent, and concentrated prior to SPE cleanup. Aqueous residues in the sample can reduce normal-phase retention. It may be necessary to further dry the organic extract with sodium sulfate or magnesium sulfate prior to SPE.

2. Condition/Equilibration

Condition and equilibriate with 2-3 tube volumes of a non-polar solvent similar or identical to sample matrix resulting from sample pre-treatment.

3. Sample Load

Apply sample (from step 1) at a consistent and reduced flow rate of ~1-2 drops/second to ensure optimal retention. The compounds should be a non-polar solvent (e.g., hexane) for optimal retention. Note that methanol and acetonitrile are often used as elution solvents in normal-phase SPE, and will often not promote compound retention during sample load.

4. Wash

Sample interferences are often co-retained with compounds of interest during sample load. A wash step is necessary to elute interferences without prematurely eluting compounds of interest. In normal-phase SPE, 1-2 tube volumes of solvent used in sample pre-treatment and conditioning can be used during wash.

5. Elution

Disrupt polar interactions with a solvent or solvent/buffer mixture more polar than both the sample and wash solutions. Typical elution solvents include water miscible organic solvents such as acetone, acetonitrile, methanol, and isopropanol. Eluting with increasingly polar solvents or solvent mixtures in succession can also fractionate multiple compound classes. See "Common Normal-Phase Solvents" table for assistance.

6. Eluate Post-treatment

Normal-phase SPE is often followed by GC analysis, and therefore requires a volatile sample matrix prior to injection. Use sodium sulfate or magnesium sample to remove residual moisture. Further SPE eluate concentration may also be necessary prior to analysis.

Common Normal-Phase Solvents

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	
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	Promotes Normal-Phase Elution
Acetic acid	>0.73	

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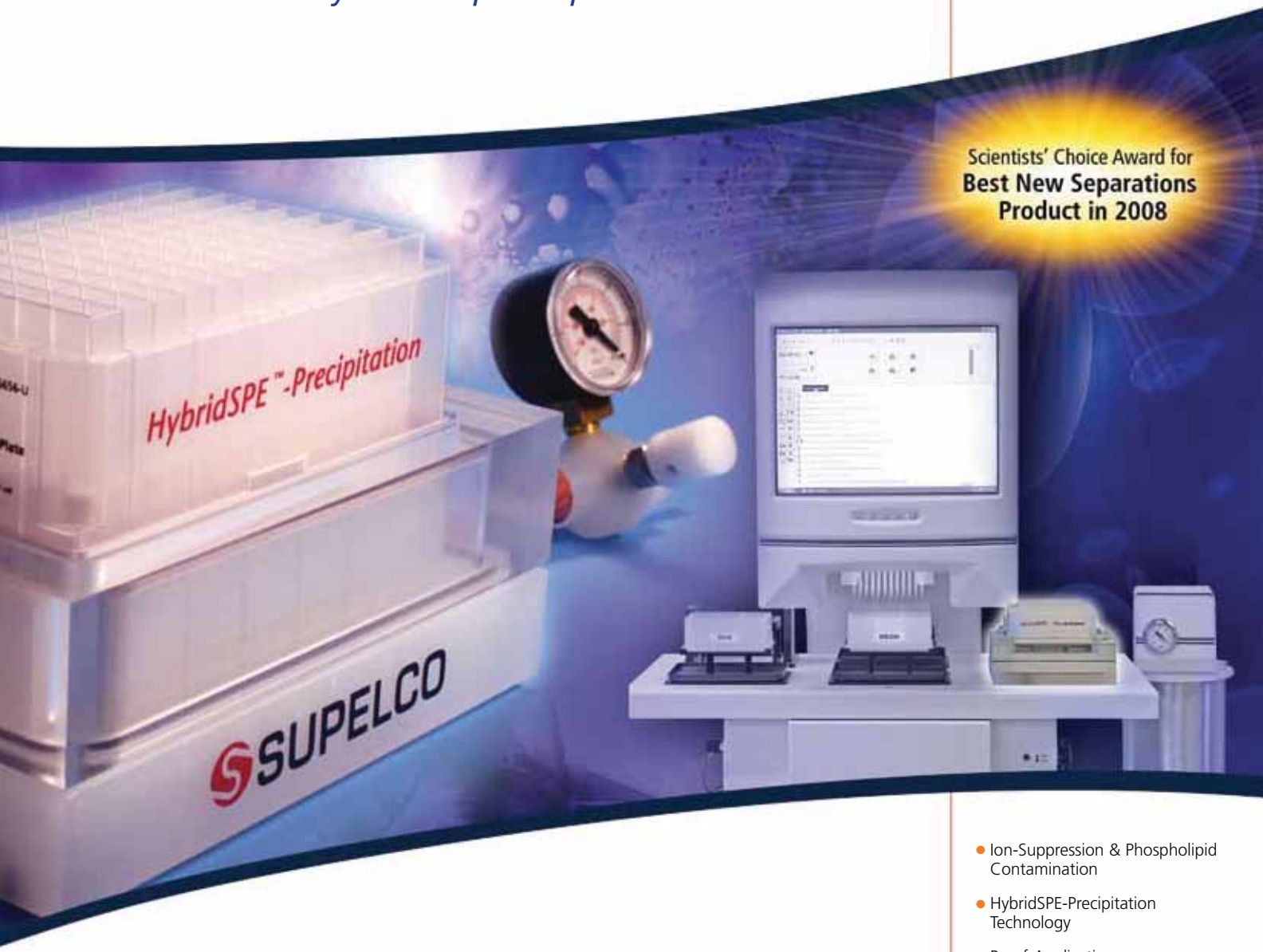
HybridSPE™-Precipitation Technology

Bridging the Gap between Simplicity and Selectivity in Pharma Bioanalytical Sample Prep



- Ion-Suppression & Phospholipid Contamination
- HybridSPE-Precipitation Technology
- Proof Applications
- Method Development: Recovery Optimization
- Troubleshooting & FAQs

Ion-Suppression & Phospholipid Contamination

In pharmaceutical bioanalysis, researchers develop and run various assays to quantitate drugs, pharmaceutical candidates, and their metabolites in biological fluids such as serum or plasma. The data resulting from these assays are used to help determine the pharmacodynamic and pharmacokinetic properties as well as the toxic and therapeutic concentrations of existing and emerging pharmaceutical compounds. Although advances in LC-MS technology have reaped overwhelming benefits in terms of increased throughput and sensitivity, good sample preparation continues to be a critical component of bioanalysis.

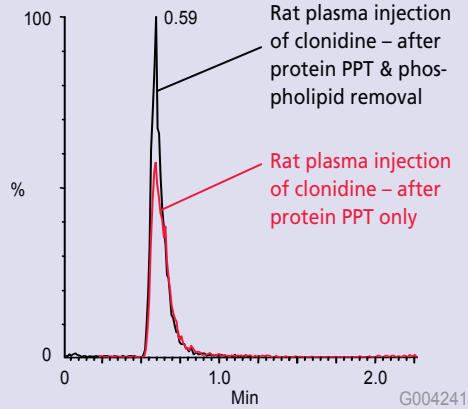
Phospholipids & Ion-Suppression

Excessive background from endogenous matrix components has always been a greater concern in bioanalysis, and has become paramount with decreasing analytical run times. In bioanalytical mass spectrometry, the issue of excessive background contributes to the growing problem of ion-suppression.

Ion-suppression is caused by one or more interfering components or species that co-elute with analyte(s) of interest during LC-MS analysis and manifests itself as a loss of analyte response. These co-eluting species can affect droplet formation or ionize concurrently resulting in an erroneous decrease (suppression) or increase (enhancement) in signal-response. The end result is poor assay reproducibility, accuracy, and sensitivity. Such deleterious effects are often most notable at the lower limits of quantiation (LLOQ).

One of the major causes of ion-suppression in bioanalysis is the presence of phospholipids during LC-MS analysis in the positive ion electrospray mode (+ESI). Phospholipids are prevalent in extremely high concentrations in blood based biological fluids (~1 mg/mL); and represent the second largest lipid component in biological matrices after triglycerides. Figure 1 compares the LC-MS chromatograms of two clonidine spiked rat plasma samples processed by standard protein precipitation alone and protein precipitation followed by phospholipid removal. When standard protein precipitation was employed, severe signal suppression of clonidine was evident. In contrast, by removing phospholipid interferences prior to analysis, response for clonidine was nearly doubled.

Figure 1. Phospholipid Effect on Ionization of Clonidine



Why are Phospholipids Difficult to Remove During Sample Prep?

When processing plasma/serum samples for LC-MS, the three most common sample prep techniques are protein precipitation (protein ppt), liquid-liquid extraction (LLE), and solid phase extraction (SPE). Each technique offers unique advantages that are considered during the method development process. For example, protein ppt methods are simple (2-3 steps), fast, and often require no method development. However, the technique offers minimal selectivity as it only removes gross levels of protein from the sample prior to analysis. In contrast, SPE offers significant benefits in terms of selectivity/sample cleanup, but the technique requires moderate to extensive levels of expertise and time for adequate method development. In addition, SPE often requires multiple steps (5-8) resulting in increased assay time.

Due to the nature of phospholipids, none of the mainstream sample prep techniques (protein ppt, LLE, and SPE) offer the proper selectivity to distinguish phospholipids from analytes of interest. Figure 2 describes this phenomenon.

Figure 2. Phospholipids Co-Extract with Analytes of Interest when Using Traditional Sample Prep Techniques

Protein PPT – No phospholipid removal

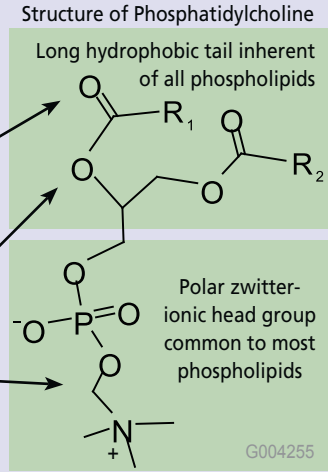
- Will only remove gross levels of proteins

LLE – No phospholipid removal

- Phospholipids will co-extract with analytes due to hydrophobic tail

SPE – Nominal phospholipid removal

- Reversed-phase – phospholipids will co-extract with analytes due to hydrophobic tail
- Mixed-mode/ion-exchange – phospholipids will co-extract with analytes due to zwitterionic polar head group



HybridSPE-Precipitation Technology

How does HybridSPE-PPT Work?

HybridSPE-PPT technology is a simple and generic sample prep platform designed for the gross level removal of endogenous protein and phospholipid interferences from biological plasma and serum prior to LC-MS or LC-MS-MS analysis. Biological plasma or serum is first subjected to protein precipitation in which biological plasma/serum is first added to the 96-well plate followed by acidified acetonitrile (precipitation agent). An upper PTFE frit impedes premature flow of the sample before vacuum application. After a brief mixing/vortexing step to facilitate protein ppt, vacuum is applied to the 96-well plate. The 96-well version contains a series of low porosity hydrophobic filters/frits. The packed-bed filter/frit assembly acts as a depth filter facilitating the concurrent removal of both phospholipids and precipitated proteins during the extraction process. After the protein-precipitated sample passes through the HybridSPE-PPT device, the resulting eluent is ready for immediate LC-MS or LC-MS-MS analysis, see Figure 3 for the recommended method.

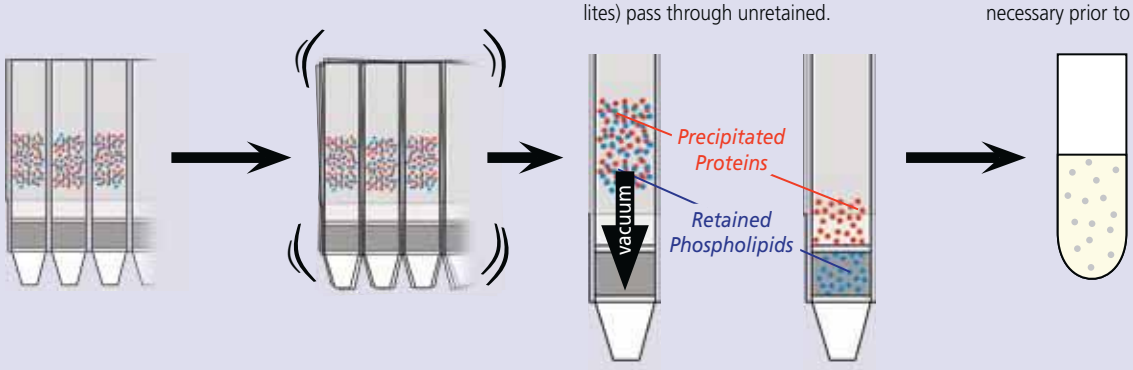
Figure 3. "In-well" Precipitation Procedure using HybridSPE-PPT 96-well Format

1) Precipitate Proteins by adding 100 µL plasma or serum to the HybridSPE-PPT plate followed by 300 µL 1% formic acid in acetonitrile. Add I.S. as necessary.

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

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

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



Note: The 1 mL HybridSPE-PPT cartridge version does not contain the necessary filters to remove precipitated proteins. As a result, protein precipitation must be conducted outside the cartridge followed by a centrifugation or filtration step prior to HybridSPE processing. The resulting supernatant/filtrate is then loaded onto HybridSPE-PPT cartridge for phospholipid removal.

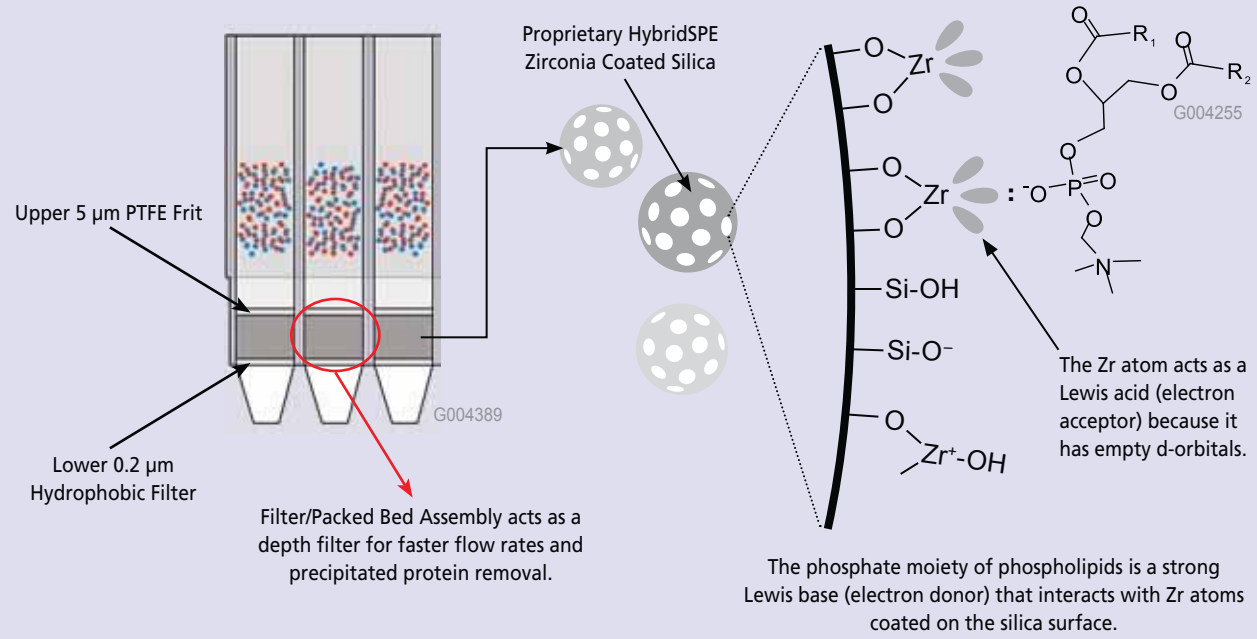
HybridSPE-PPT Feature	HybridSPE-PPT Benefit
2-3 Step Procedure (like standard protein ppt)	Save Time & Increase Throughput
Generic Methodology	Save Time & Headache during Method Development
100% Phospholipid & Precipitated Protein Removal	Reduce Ion-Suppression& Improve Assay Sensitivity
Fast Flow Rates	Robust Methodology & Faster Sample Prep
Available in Cartridge & 96-well Dimensions	Amenable to Automation
Patent Pending Technology	New and Unique Approach to Sample Prep
Low Price (less expensive than traditional SPE)	Save Money While Improving Data Quality

Abbreviation Glossary:
I.S. - Internal Standard; **LLE** - Liquid Liquid Extraction; **LLOQ** - Lower Limit of Quantitation; **PPT** - Precipitation; **SPE** - Solid Phase Extraction

How are Phospholipids Removed?

Once the plasma/serum sample is subjected to protein precipitation via the addition of acetonitrile (containing 1% formic acid), it is passed through the HybridSPE-PPT packed bed. The packed bed consists of proprietary zirconia coated silica particles. The zirconia sites exhibit Lewis acid (electron acceptor) properties that will interact strongly with Lewis bases (electron donor). Phospholipids structurally consist of a polar head group (zwitterionic phosphonate moiety) and a large hydrophobic tail (two fatty acyl groups that are hydrophobic). The phosphate group inherent with all phospholipids acts as a very strong Lewis base that will interact strongly with zirconia atoms functionalized on the particle surface (Figure 4). Formic acid is a critical modifier used in the precipitation agent to improve the recovery of many analytes of interest (e.g., acidic compounds). It plays a critical role in preventing analyte retention without affecting phospholipid retention/removal.

Figure 4. HybridSPE-PT 96-well Schematic and Phospholipid Retention Mechanism



Ordering Information

Featured Products

Description	Qty.	Cat. No.
HybridSPE-Precipitation 96-well Plate, 50 mg/well	1	575656-U
HybridSPE-Precipitation 96-well Plate for Small Volume Samples, 15 mg/well	1	52794-U
HybridSPE-Precipitation Cartridge, 30 mg/1 mL	100	**55261-U**
HybridSPE-Precipitation Cartridge, 500 mg/6 mL	30	**55267-U**

Related Products

Description	Qty.	Cat. No.
96-well Protein Precipitation Filter Plate	1	**55263-U**
Supelco PlatePrep Vacuum Manifold	1	**57192-U**
96 Square/Deep Well Collection Plates, 0.35 mL, PP	50	**575651-U**
96 Square/Deep Well Collection Plates, 1 mL, PP	50	**575652-U**
96 Square/Deep Well Collection Plates, 2 mL, PP	50	**575653-U**
96 Square Well Pierceable Cap Mats	25	**575655-U**

Comparison of Sample Prep Techniques

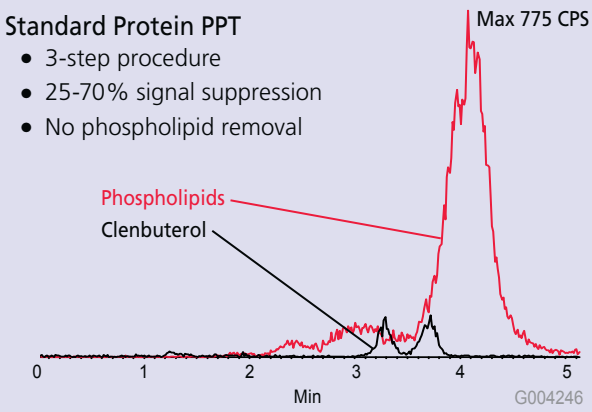
HybridSPE-PPT combines the simplicity of protein precipitation with the selectivity of SPE targeting the specific removal of both phospholipids and precipitated proteins from biological plasma. Both phospholipids and protein represent the two largest contributors of ion-suppression in pharmaceutical bioanalysis. If adequately removed prior to LC-MS, analysts can more easily and quickly achieve targeted LLOQ for most applications. Figure 5 compares standard protein PPT, SPE, and HybridSPE-PPT for the extraction of clenbuterol from plasma.

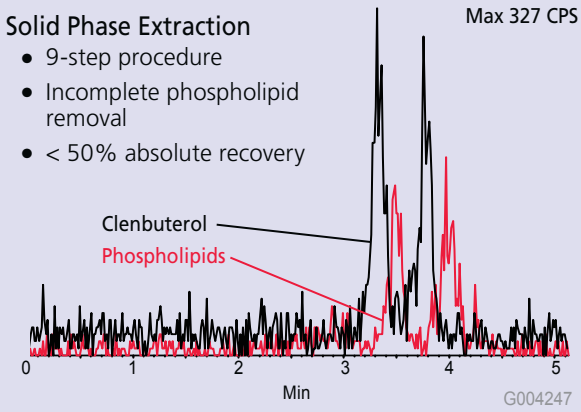
	Std. Protein PPT	HybridSPE-PPT	Solid Phase Extraction
Number of Steps	2-3	2-3	5-8
Time Required	< 10 min.	<10 min.	30-45 min.
Method Development	Generic/Minimal	Generic/Minimal	Moderate/Extensive
Sample Cleanup & Reduction of Ion-Suppression	Minimal	Moderate to High	Moderate to High
Phospholipid Removal	NONE	High	Moderate
Recovery	High	Moderate to High	Moderate to High

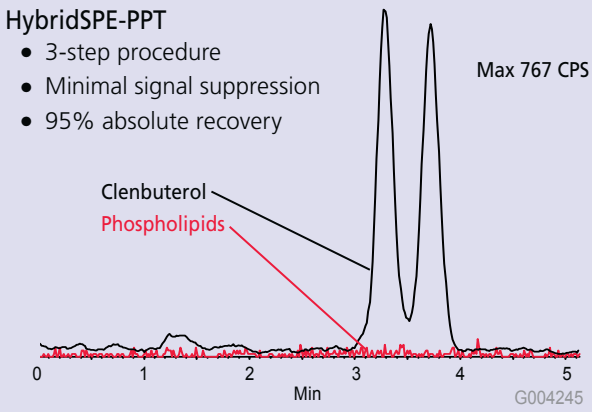
Green = Advantage **Red = Disadvantage**

Figure 5. Comparative Extraction of 10 ng/mL Clenbuterol (R(-) and S(+) enantiomers) in Rat Plasma

column: CHIROBIOTIC T, 10 cm x 2.1 mm, 5 µm particles
mobile phase: 10 mM ammonium formate:methanol
flow: 300 µL/min.
temp.: 30°C
detector: MS-MRM







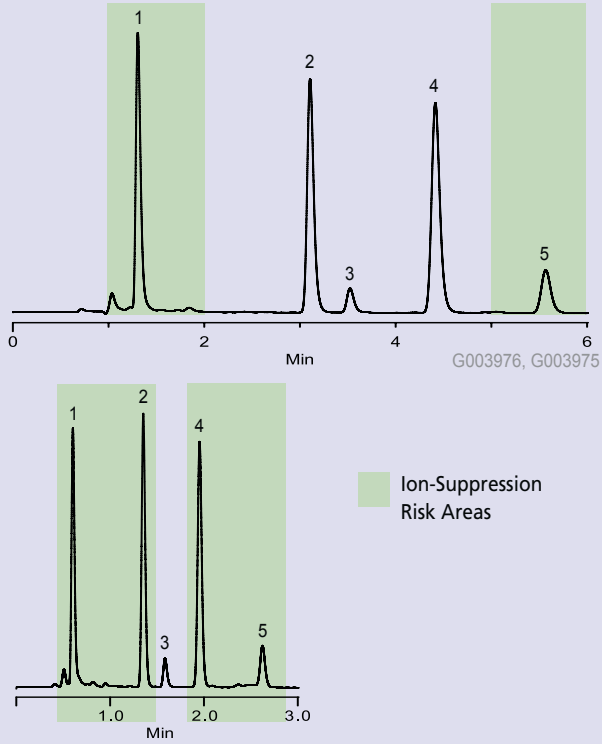
Did you know...?

HybridSPE-PPT won the SelectScience.net Scientists' Choice Award for Best New Separations Product in 2008. The award was based on an independent poll of over 25,000 scientists within the SelectScience.net community. The award was announced at PITTCON® 2009 held in Chicago, IL.

Improve LC-MS Run Time/Performance & Reduce Risk of Ion-Suppression

When analyzing biological samples via reversed-phase LC-MS, ion-suppression often occurs in the early (e.g., salts) and late regions (e.g., phospholipids) of a chromatogram. However, as analysts strive for faster run times (≤ 3 min.), resolving analytes of interest from interfering matrix components becomes a greater challenge (Figure 6).

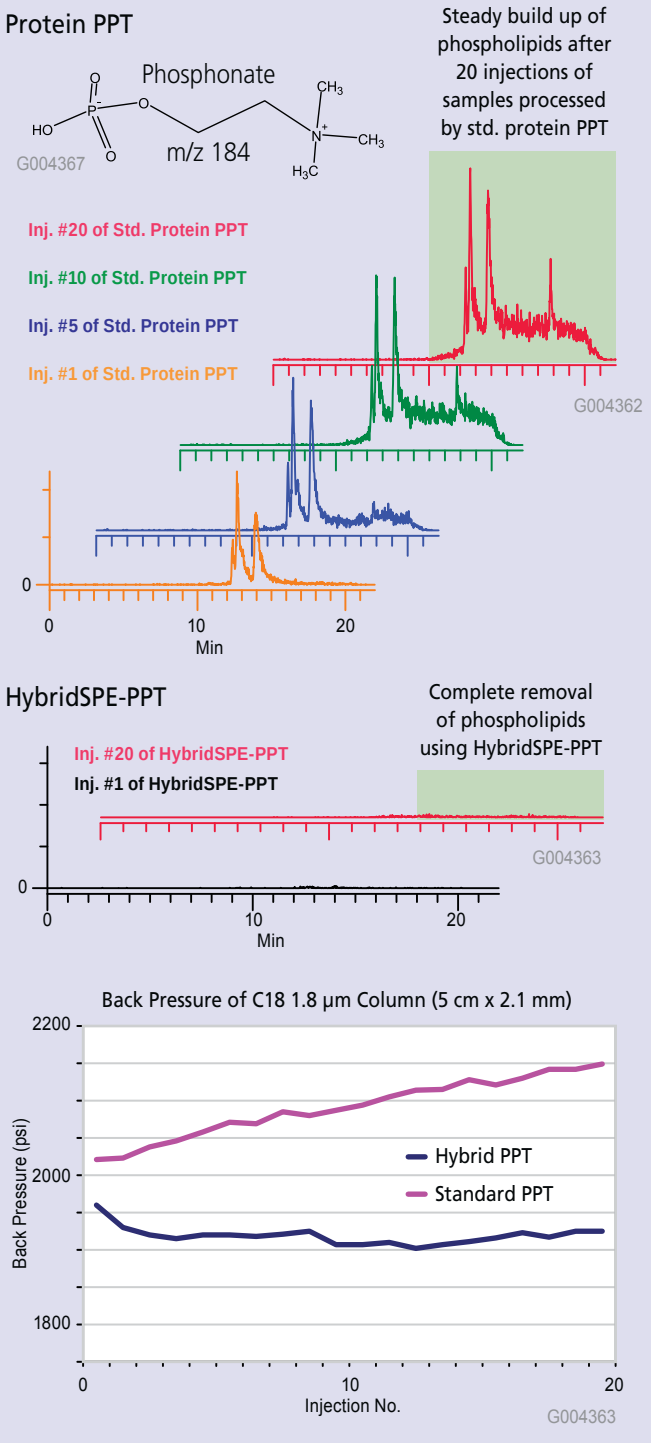
Figure 6. Decreasing LC Run Time Increases Risk of Ion-Suppression



LC Accumulation of Phospholipids

With advances in LC-MS technology, many analysts are decreasing LC run time by incorporating ballistic gradients and sub-2 µm HPLC column particles. When coupled with standard protein ppt (e.g., plasma:acetonitrile, 1:3 v/v), ballistic gradients are often inadequate at purging the column of phospholipids. As a result, phospholipids can build on the column, potentially change LC retention selectivity, and elute uncontrollably downstream in an injection run sequence causing unpredictable ion-suppression effects and poor reproducibility. Figure 7 compares a series of reversed-phase gradient LC-MS injections after standard protein ppt with HybridSPE-PPT in which m/z 184 (phosphonate moiety of phospholipids) is monitored. In addition, sub-2 µm HPLC columns are more prone to clogging than larger particle size columns (2.7-5.0 µm). Unlike traditional protein ppt techniques that use centrifugation to remove precipitated proteins, HybridSPE-PPT 96-well plates contain a series of filters that allows users to concurrently remove proteins and phospholipids reducing LC column backpressure (Figure 7).

Figure 7. Gradient RP LC-MS of Blank Plasma Samples Prepared by Standard Protein PPT vs. HybridSPE-PPT



Reduce Analytical Run Time Using HybridSPE-PPT

When processing plasma samples using standard protein ppt, contaminating phospholipids are strongly retained and do not elute off the column after 10 min. Therefore, shorter run times (e.g., < 5 min.) pose a great risk to on-column phospholipid accumulation/contamination. Gradient conditions are often required to elute phospholipids in a reasonable time frame (Figure 8). Because HybridSPE-PPT depletes phospholipids from the sample, it is not necessary to run long gradient conditions to "wash-off" contaminating phospholipids. We demonstrate the utility of combining HybridSPE-PPT and Ascentis column technology in Figure 9 in which less than 90 second run time was achieved under isocratic conditions. Because HybridSPE-PPT was employed during sample prep, the risk of phospholipid ion-suppression and on-column accumulation was eliminated.

Figure 8. Phospholipid Contamination from Standard Protein PPT Requires Increased Gradient Run Time (> 10 min.)

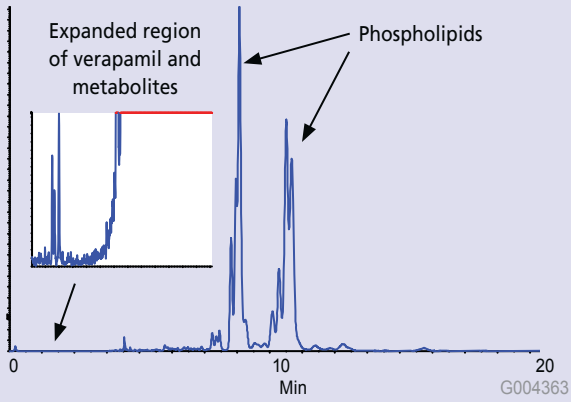
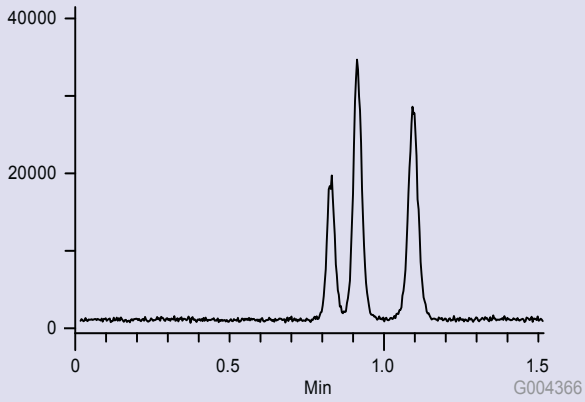


Figure 9. Less than 90 Sec. Run Time Achieved Using Ascentis Express C18 (isocratic) and HybridSPE-PPT for Verapamil and Metabolites in Rat Plasma



From Sample Prep to LC-MS Analysis…

The Perfect Complement of Speed, Selectivity, and Sensitivity for Pharma Bioanalysis

- Increase Sample Prep & LC-MS Speed
- Decrease Sample Prep Method Development Time
- Increase Sensitivity by Reducing Ion-suppression & Increasing LC Efficiency

HybridSPE-PPT Technology

- 2-3 Step generic procedure
- Reduce ion-suppression through phospholipid & protein removal
- Minimal to no method development
- Available in 96-well & 1 mL cartridges

Ascentis® Express HPLC with Fused-Core™ Technology

- Half the back-pressure of sub-2 µm particles
- Twice the efficiency of 3 µm particles
- Increased column ruggedness
- Available in 6 phases for small molecule and peptides

HybridSPE-PPT Method Development: Recovery Optimization

The HybridSPE-PPT stationary phase is comprised of a proprietary patent pending zirconia bonded silica particle. Phospholipids are retained/removed from the sample through a highly selectivity Lewis acid-base interaction between the phosphate group (Lewis base), inherent of all phospholipids, and Zr atoms bonded to the HybridSPE silica surface.

The precipitation agents recommended for HybridSPE-PPT is comprised of two parts:

1) an aqueous miscible organic solvent (e.g., MeCN or MeOH) that denatures protein into a precipitant
2) a modifier (formic acid, ammonium formate, or citric acid) that inhibits analyte(s) of interest from co-retaining with phospholipids on the Zr-Si HybridSPE stationary phase, potentially resulting in low recovery

Relative Retention Strength of Lewis Bases to Zirconia

Lewis Base	Relative Retention Strength on Zirconia
Hydroxide	Strongest
Phosphate	
Fluoride	
Citrate	
Sulfate	
Acetate	
Formate	
Chloride	Weakest

When developing new HybridSPE-PPT methods, there are three precipitation agents that we recommend for researchers to screen when optimizing analyte recovery.

96-well Primary Method (Formic Acid Modifier):

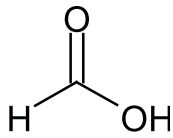
Recommended for most applications (basic, neutral, acidic compounds) – amenable for most (80%) applications

1. To each well, add 100 µL plasma followed by 300 µL **1% formic acid in MeCN**
2. Mix the sample well (e.g., vortex for 1 min.)
3. Apply vacuum and collect the resulting eluent for LC-MS analysis

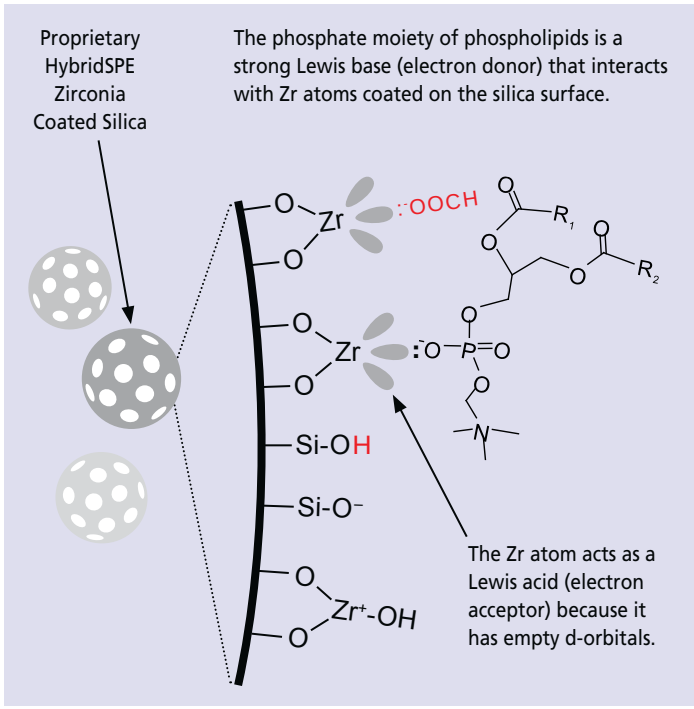
If low recovery is observed, proceed to Secondary Procedures. An alternative option is to screen both the primary and secondary procedures concurrently during initial method development.

- Formate ion (**HCOO⁻**) is a strong enough Lewis base to inhibit most acidic compounds from interacting with Zr sites, improving recovery of acidic compounds

Fomic Acid



- **H⁺** derived from formic acid neutralizes silanol groups (Si-O- => Si-OH), minimizing potential secondary weak cation exchange (WCX) interactions of basic compounds improving analyte



96-well Secondary Method (Basic & Neutral Compounds):

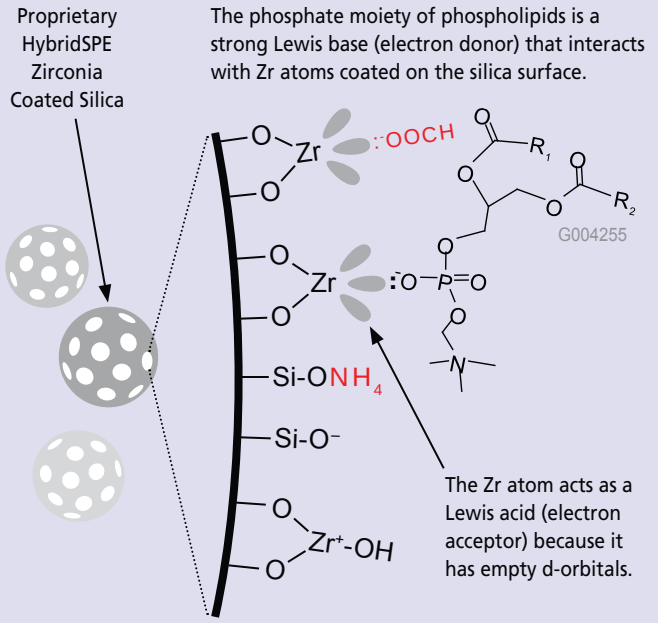
Recommended for low recovery basic & neutral compounds

1. To each well, add 100 µL plasma followed by 300 µL **1% ammonium formate in MeOH**
2. Mix the sample thoroughly (e.g., vortex for 1 min.)
3. Apply vacuum and collect the resulting eluent for LC-MS analysis

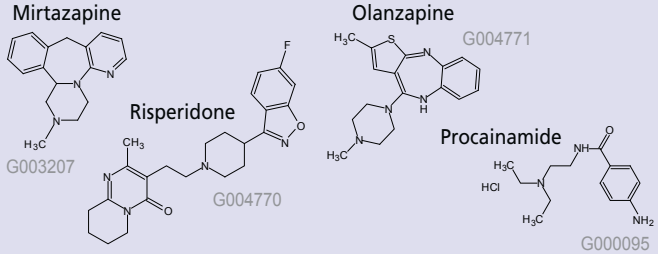
Ammonium Formate

$[NH_4]^+$ $[HCOO]^-$

- Methanol combined with ammonium formate is a powerful protein precipitation agent, providing precipitants similar in consistency to acetonitrile protein precipitation
- Formate ion (**$HCOO^-$**) is a strong enough Lewis base to inhibit most acidic compounds from interacting with Zr sites, improving recovery of acidic compounds
- **NH_4^+** is a stronger counter-ion than H^+, able to inhibit a wider range of basic compounds from interacting with exposed silanol groups



Examples of Basic Compounds that require the Secondary Method (Methanol with 1% Ammonium Formate):

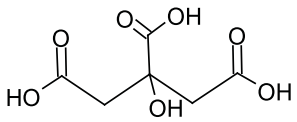


96-well Secondary Method (Chelator & Acidic Chelator Compounds):

Recommended for low recovery chelator & acidic chelator compounds

1. Condition each well with 400 µL **0.5% citric acid in acetonitrile**
2. To each well, add 100 µL plasma followed by 300 µL **0.5% citric acid in acetonitrile**
3. Apply vacuum and collect the resulting eluent for LC-MS analysis

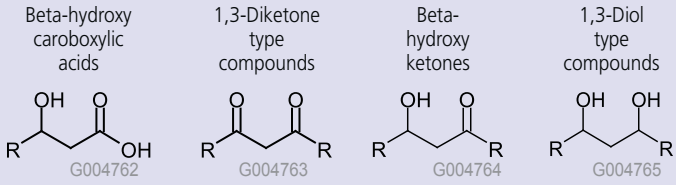
Citric Acid



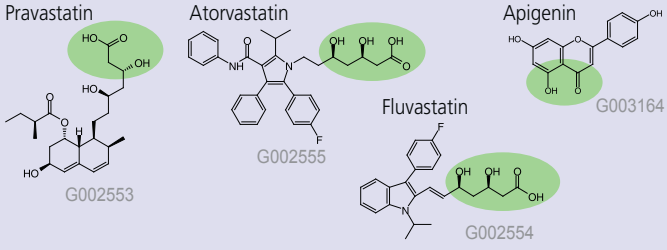
- **Citrate ion** (citric acid) is a stronger Lewis base than formate, inhibiting most chelator compounds from interacting with Zr ions on the HybridSPE stationary phase
- Note that citrate is not strong enough to disrupt phosphate (phospholipid) binding

Low Recovery Chelation Functional Groups with Example Compounds

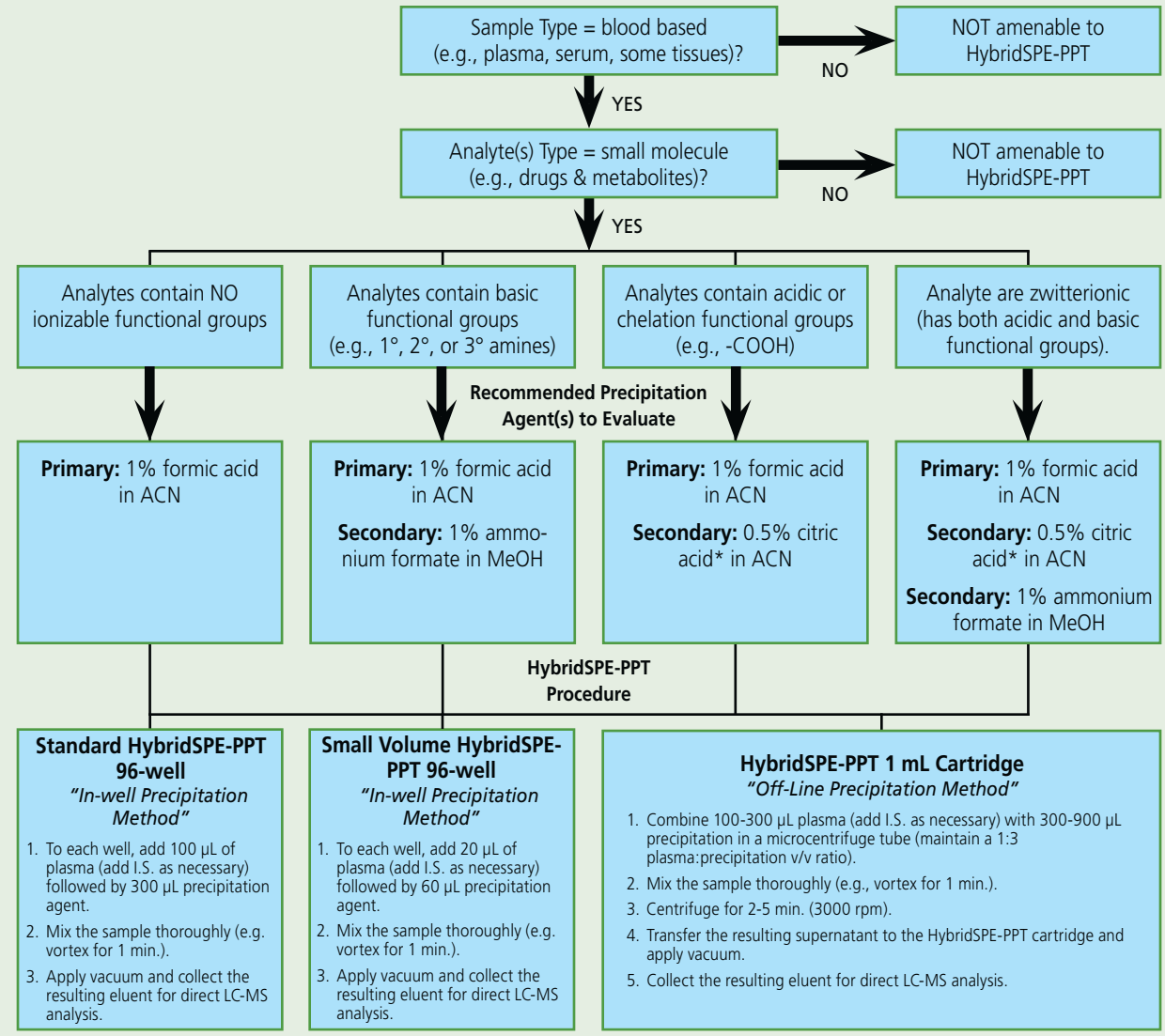
Chelation functional groups that can lead to low HybridSPE-PPT recovery and may require citric acid secondary procedure:



Example compounds with chelation functional groups:



HybridSPE-PPT Method Development (Recovery Optimization) Flow Chart



* Note: When 0.5% citric acid in ACN is employed as the precipitation agent, the HybridSPE-PPT phase (96-well or 1 mL cartridge) should be conditioned with 400 µL 0.5% citric acid in ACN prior to sample addition.

Troubleshooting & Frequently Asked Questions

Can I use HybridSPE-PPT with smaller plasma volumes (e.g., 20-40 µL plasma)?

Yes. The HybridSPE-PPT Small Volume plate (52794-U) is designed for processing plasma/serum volumes between 20-40 µL. Larger sample volumes of 100-300 µL should be processed on the standard HybridSPE-PPT plate (575656-U).

Why is acetonitrile and formic acid used as a precipitating agent in the HybridSPE-PPT method?

Acetonitrile is a commonly used protein precipitation agent when prepping plasma samples for LC-MS analysis. The resulting precipitated protein is easily filtered using the "In-well Precipitation" method and forms protein pellets easily removed when centrifugation ("Off-Line Precipitation" method – required for HybridSPE-PPT 1 mL cartridge format) is preferred.

The addition of 1-2% formic acid to the acetonitrile precipitating agent is critical because: 1) formic acid is a stronger Lewis base than most carboxyl (-COOH) groups found in acidic pharmaceutical compounds (inhibiting analyte retention on the HybridSPE phase) but not

as strong a Lewis base as the phosphate moiety found in phospholipids; and 2) the low pH environment neutralizes residual silanol activity on the silica surface, thereby eliminating secondary cation-exchange interaction with basic compounds of interest.

What if my analyte(s) of interest are not soluble in acetonitrile?

Although some analytes may not be soluble in acetonitrile, after protein precipitation, the HybridSPE eluent will consist of 75% acetonitrile (w/ formic acid) and 25% aqueous (from the biological sample). The aqueous content of the sample should provide adequate solubility prior to LC-MS analysis.

Alternatively, 1% ammonium formate in methanol may be used in place of 1% formic acid in acetonitrile. Ammonium formate in methanol provides increased solubility of polar compounds and precipitates proteins as well as acetonitrile allowing for both "Off-Line" and "In-well" precipitation methods.

Can I increase assay sensitivity by either increasing sample volume and/or concentrating (evaporation and reconstitution) of the HybridSPE eluent?

Biological sample volumes of >300 µL can be applied; however, some phospholipid breakthrough may occur with analytes of interest. 98-100% of biological phospholipids are removed when <300 µL plasma is applied to the HybridSPE phase. When increasing sample volume, be sure to increase the volume of the precipitating agent accordingly. A 1:3 (v/v) plasma: precipitating agent ratio is necessary for optimal performance.

Another strategy for increasing sensitivity is through evaporation of the HybridSPE eluent, followed by reconstitution in a smaller volume of LC-MS mobile phase. The acetonitrile portion of the HybridSPE eluent greatly aids the evaporation process. On average it takes less than 10 minutes to evaporate 300-400 µL of HybridSPE eluent under nitrogen at 37 °C.

Why can ion-suppression still be evident during LC-MS analysis after HybridSPE-PPT?

HybridSPE technology will only remove phospholipids and gross levels of precipitated protein from biological samples. Other chemical entities common to biological samples can lead to ion-suppression if not removed prior to LC-MS-MS analysis. It is important to identify the ion-suppression causing component to facilitate troubleshooting. It may be necessary to adjust chromatographic conditions to separate analytes of interest from interfering matrix components.

Examples of non-phospholipid chemicals that can lead to ion-suppression include:

- sodium citrate which is an anti-coagulant used to prepare plasma from blood
- phthalates, plasticizers and other mold release agents found in plastic ware
- polyethylene glycol which is a common dosing vehicle for many drugs
- extractables from o-rings, plastic ware, and seals used to store biological samples

Why is the resulting HybridSPE-PPT eluent lower in volume than what was applied to the HybridSPE packed bed? What are the effects of conditioning the phase?

The dead volume for the HybridSPE packed bed is ~80 µL. Also, there is an evaporation effect on the eluent when using a vacuum manifold. When applying -15 in Hg vacuum to the HybridSPE plate for 3 min. (time taken to pass the sample through the well plate), 10-20 µL of the volume of the SPE eluent can be lost due to evaporation during processing. Therefore, when processing a 400 µL precipitated sample (100 µL plasma + 300 µL precipitating agent) through the unconditioned HybridSPE phase, the resulting eluent will be a volume of ~300 µL. Although the volume is reduced during SPE processing, the final analyte concentration of the eluent does not appear to be affected. Nevertheless, addition of an I.S. is recommended prior to HybridSPE processing (which is standard for most sample prep techniques). If the analyst chooses to condition the HybridSPE phase with >80 µL of solution prior to sample addition, there could be a dilution effect. The final eluent volume will be ~80 µL greater than it should be. As a result, absolute recovery will appear lower than it actually is. If an increase in signal response is necessary during LC-MS analysis, we recommend evaporating the eluent and reconstituting in a smaller known volume of LC mobile phase prior to LC-MS analysis.

Why am I experiencing absolute recovery values > 100%?

The resulting HybridSPE eluate contains acetonitrile (volatile solvent). After the precipitated sample completely passes through the HybridSPE packed bed, the vacuum should be disengaged immediately. Further vacuum application can evaporate the eluate, thereby erroneously giving misleadingly high analyte responses during subsequent LC-MS analysis.

To learn more, visit ***sigma-aldrich.com/hybridspe-ppt***

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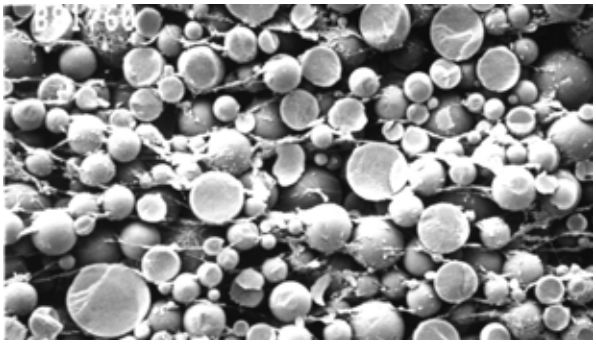
Empore® Solid Phase Extraction (SPE) Products

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



E001071

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

Save Time & Money with Empore SPE

Reduced SPE bed mass = Reduced SPE solvent & elution volumes

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

Dense & uniform extraction medium = NO SPE channeling/voiding

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

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

[1] Conditioning typically requires 4-5 x bed volumes.

[2] Elution typically requires 2-3 x bed volumes

Available Formats:

E001070

E001069

E001068

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

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

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

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

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

SUPELCO Analytical

Ordering Information:

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

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SUPELCO® Analytical

SupelMIP™ Solid Phase Extraction

Molecularly Imprinted Polymers for the Highly Selective Extraction of Trace Analytes from Complex Matrices

MIP Technologies

- **NEW** SupelMIP Phases and Applications
- Achieve Lower Detection Limits
- Reduce Time and Costs
- Improve MS-Compatibility
- No Method Development Required

Supelco Partners with MIP Technologies AB

MIP Technologies AB, Lund, Sweden, is a world leading company in the development of molecularly imprinted polymers (MIPs). The company is a pioneer in the commercial applications of MIPs, holds important patents, and maintains cutting-edge research activities in the area. The company's mission is to provide innovative products based on molecularly imprinted polymers that serve industry's needs in analytical, preparative and process scale 'selective separations'.

Supelco and MIP Technologies has entered in a collaborative agreement in which, as of December 2006, Supelco has assumed the exclusive global distribution of MIP Technologies' patent protected molecularly imprinted polymers for sample preparation, analytical, and preparative applications. The companies will collaborate on the development of new products while MIP Technologies will continue to separately develop its process scale separations business.

Supelco, Bellefonte, PA, USA MIP Technologies AB, Lund, Sweden

"With molecularly imprinted polymer technology, analysts can reach a level of sample prep extraction selectivity that could not be achieved by conventional means. With the widespread advent of mass spec technology, more and more methods are requiring lower limits of quantitation when analyzing difficult and dirty sample matrices. Improvements in selectivity during sample preparation are absolutely critical," said An Trinh, Product Manager, Supelco.

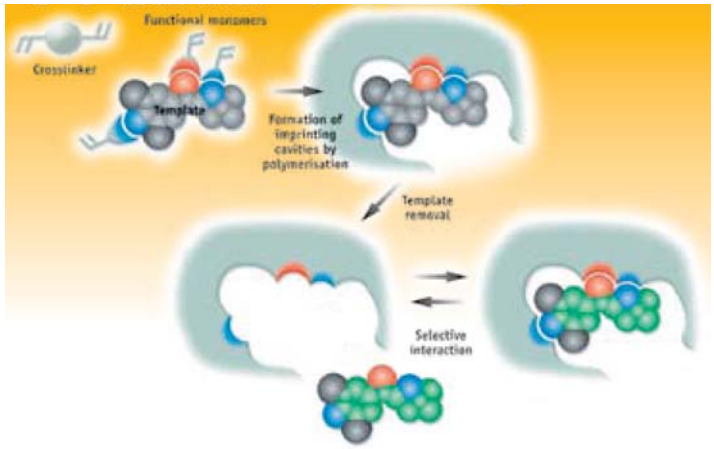
"By merging the strengths of both organizations in this collaborative effort, a new generation of innovative molecularly imprinted polymers and applications will emerge."

What are Molecularly Imprinted Polymers?

Molecularly imprinted polymers (MIPs) are a class of highly cross-linked polymer-based molecular recognition elements engineered to bind one target compound or a class of structurally related target compounds with high selectivity. Selectivity is introduced during MIP synthesis in which a template molecule, designed to mimic the analyte, guide the formation of specific cavities or imprints that are sterically and chemically complementary to the target analyte(s).

As illustrated in Figure 1, MIPs are prepared by first mixing a template molecule that consists of a structural analog of the analyte(s) of interest with one or more functional monomers. The monomers form spontaneous complexes around the template. Upon complex formation, cross-linking monomers are then added with a suitable porogen (solvent that aids in the role in pore formation) to drive polymerization. An extensive wash procedure is used to remove the template from the polymer, leaving imprints or binding sites that are sterically and chemically complementary to the template.

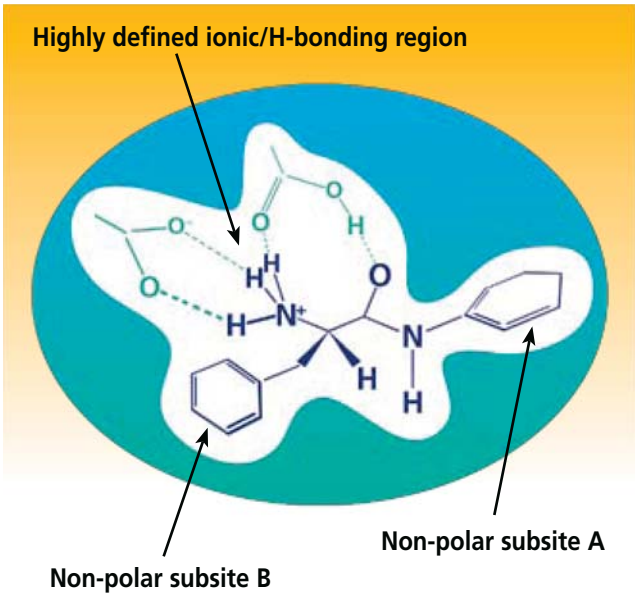
Figure 1. Formation of MIPs



SupelMIP is a trademark of Sigma-Aldrich Biotechnology LP., Inc. SupelMIPs have been developed by MIP Technologies AB.

How is Selectivity Improved Using SupelMIP SPE?

Figure 2. Visual Depiction of a Typical MIP Binding Site

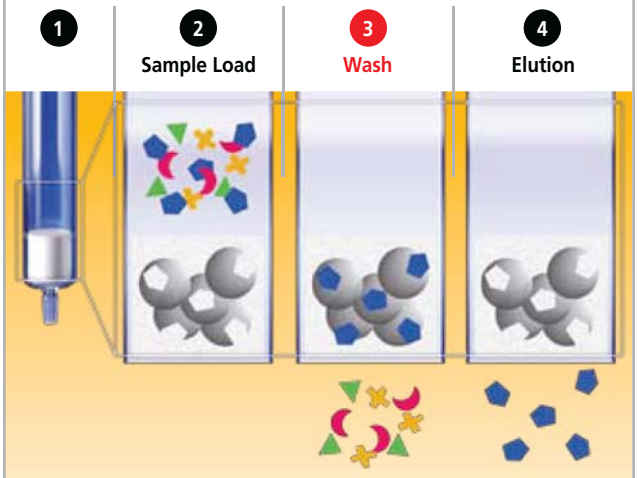


By careful design of the imprinting site, either by molecular modeling, experimental design, or screening methods, the binding cavities can be engineered to offer multiple interactions with the analyte(s) of interest (Figure 2). Multiple non-covalent interaction points (ion-exchange, reversed-phase with polymer backbone, and hydrogen bonding) between the MIP phase and analyte functional groups allow for stronger and more specific analyte retention. Improved selectivity is then introduced through the use of harsher wash conditions during sample prep methodology (Figure 3). Because extraction selectivity is significantly improved, lower background is observed allowing analysts to achieve lower detection limits relative to other less selective sample prep techniques (Table 1).

Did you know?

Did you know that SupelMIP SPE scored perfectly (z-score of 0) for the FAPAS (Food Analysis Perfomance Assessment Scheme) proficiency test for chloramphenicol in prawns? FAPAS is the largest international analytical proficiency testing program since 1990. See the press release at ***sigma-aldrich.com/supelmip*** for more information.

Figure 3. Overview of a Typical SupelMIP SPE Procedure



1. Condition and equilibrate SupelMIP SPE
2. Sample Load
3. Application of a series of vigorous wash steps that will selectively retain analyte(s) of interest but elute interfering components
4. Analyte elution

Table 1. Relative Selectivity of Various Sample Prep Techniques

Non-Selective	**Dirty Extracts**
Protein Precipitation	
Liquid-Liquid Extraction	
Hydrophobic Resin SPE	
Supported LLE	
C18-C2 Silica Based SPE	
Mixed-Mode SPE	
SupelMIP SPE	
Highly Selective	**Clean Extracts (Lower LODs/ LOQs)**

Key Features & Benefits of SupelMIP:

Achieve lower detection limits through superior selectivity	*Reduce ion-suppression*	*Save time and reduce cost via robust and rapid methodology*	*Minimal to no method development required*	*Stable at broad pH ranges and high temperatures*	*Stringent quality control conditions*

Achieving Lower Detection Limits Through Superior Selectivity

Chloramphenicol is a broad spectrum antiobiotic that has recently been determined as a causative agent of aplastic anemia and possible carcinogen in humans. Because of these health concerns, the EU, US and Canada have banned the use of chloramphenicol in food-producing animals and live stock.

In this application, the extraction of 15 ng/mL of chloramphenicol from milk using a SupelMIP SPE – Chloramphenicol cartridge was compared against a published method using a conventional hydrophilic polymer SPE phase (P.A. Guy et al. in J. Chromatogr. A 1054 (2004) 365-371). Note that unlike the SupelMIP SPE – Chloramphenicol protocol (included with the product), the conventional polymer method required a protein precipitation/filtration and three liquid-liquid extraction steps in addition to SPE cleanup. Using the SupelMIP SPE – Chloramphenicol phase, the total sample handling time is significantly reduced.

In Figure 4, we see that LC-MS signal/noise ratio for the hydrophilic polymer SPE method was double that of the SupelMIP ion-chromatograms (320-323 m/z range); and blank milk samples processed using the SupelMIP were free of interfering responses in the elution area of chloramphenicol. In Figure 5, a significantly cleaner mass spectra is observed for the SupelMIP SPE extract relative to the conventional hydrophilic polymer extract.

For additional information regarding this application, please refer to Supelco Reporter 25.1; or European Reporter, Issue 26, May 2007, available at ***sigma-aldrich.com/supelmip***

Figure 4. Chloramphenicol Spiked Milk Samples Extracted on SupelMIP SPE vs. Conventional Hydrophilic Polymer SPE

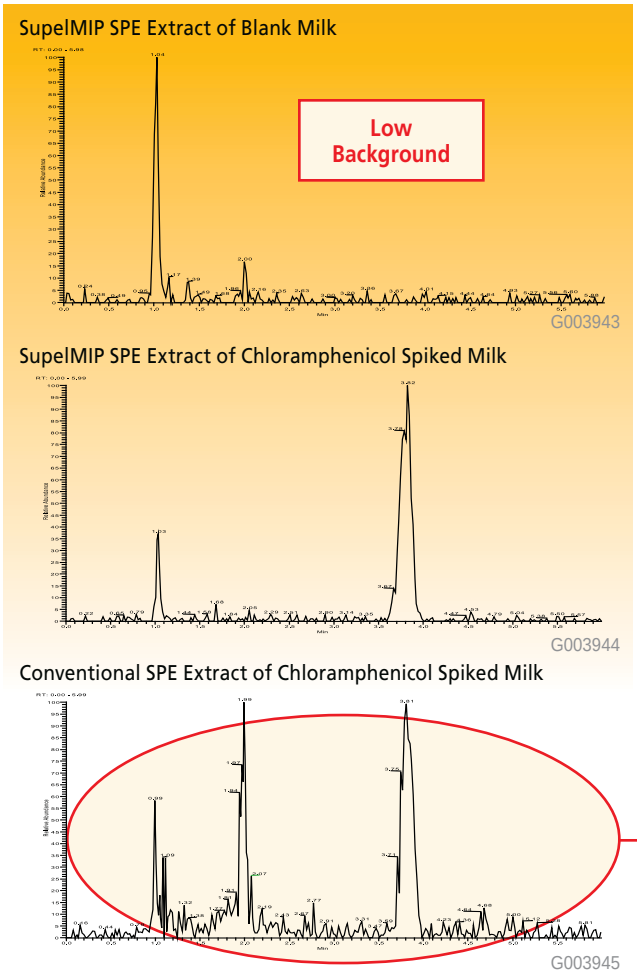
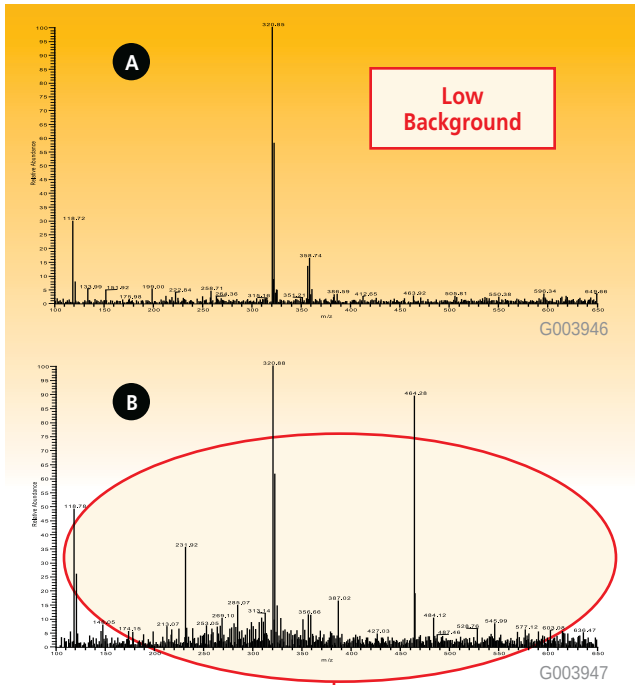


Figure 5. Mass Spectrum of Full Ion-Chromatograms (3.65- 4.00 min.) of the SupelMIP SPE Extract (A) and the Hydrophilic Polymer SPE Extract (B)



Note: Higher background observed with conventional hydrophilic polymer SPE phase

Key Features & Benefits of SupelMIP:

Achieve lower detection limits through superior selectivity	*Reduce ion-suppression*	*Save time and reduce cost via robust and rapid methodology*	*Minimal to no method development required*	*Stable at broad pH ranges and high temperatures*	*Stringent quality control conditions*

Clenbuterol is a beta-agonist known for its growth-promoting properties in which use of the drug induces significant weight gain by increasing the proportion of muscle mass to fat. Although the US Food and Drug Administration, US Department of Agricultural and European Union have banned the use of clenbuterol for humans and livestock, illegal use of the drug still readily occurs.

In this application, 0.1-1.0 ng/mL clenbuterol was extracted from human urine using a SupelMIP SPE – Clenbuterol cartridge. The SupelMIP method was compared against a published method using a conventional hydrophilic polymer SPE phase (M. Joseffson, et al., J. Chromat. A., 2006, 1120:1-12.). The extracts were analyzed via LC-MS/MS analysis.

In Figure 6, blank urine samples extracted with the SupelMIP protocol offered low background. In contrast, the conventional hydrophilic polymer SPE procedure co-extracted matrix interferences resulting in a high background response within LC elution area of clenbuterol (1-2 min.). This can potentially lead to lower assay reproducibility, accuracy and sensitivity thereby elevating lower limits of quantitation. Table 2 lists recovery values for clenbuterol using both sample prep procedures. Note that at the lowest spike levels tested (0.1 ng/mL), SupelMIP recovery was 99% whereas the hydrophilic polymer phase yielded a recovery of 8%.

For additional information regarding this application, please refer to Supelco Reporter 25.2; or European Reporter, Issue 25, March 2007, available at ***sigma-aldrich.com/supelmip***

Figure 6. Clenbuterol Spiked Urine Samples Extracted with SupelMIP SPE vs. Convention Hydrophilic Polymer SPE

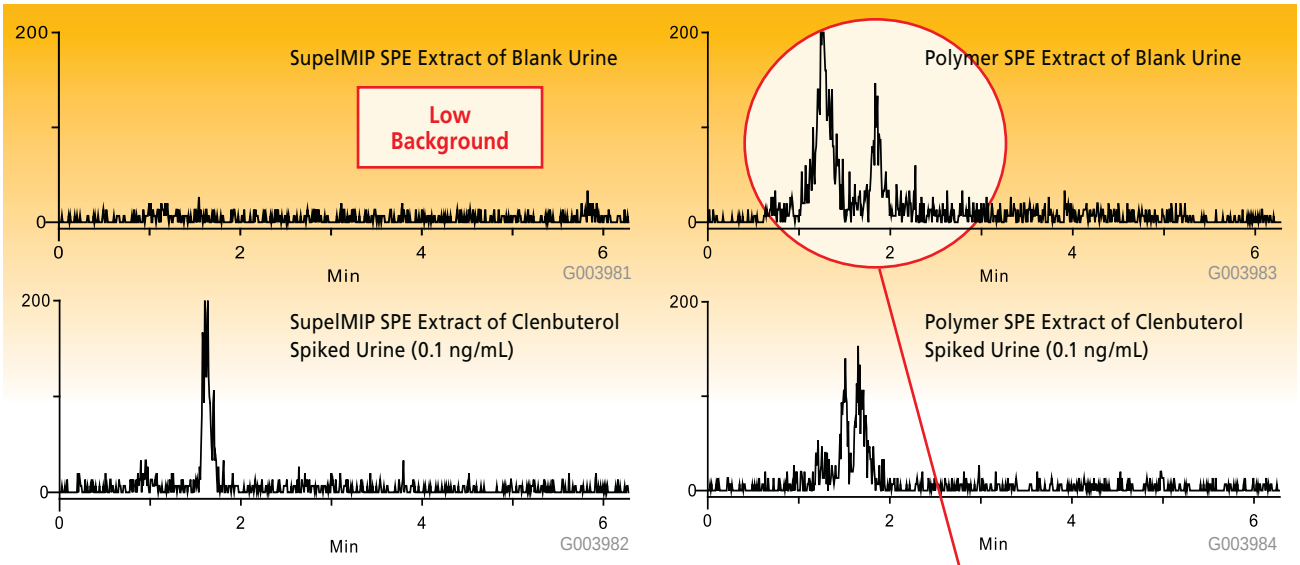


Table 2. Recovery Comparison for Clenbuterol from Urine using SupelMIP SPE and Conventional Hydrophilic Polymer SPE

	% Recovery from Urine	
Spike Level (ng/mL)	SupelMIP SPE - Clenbuterol	Hydrophilic Polymer SPE
0.1	99%	**8%**
0.5	75%	66%
1.0	75%	69%

Note: Higher background observed with conventional hydrophilic polymer SPE phase resulted in signal suppression at lower detection levels.

Key Features & Benefits of SupelMIP:

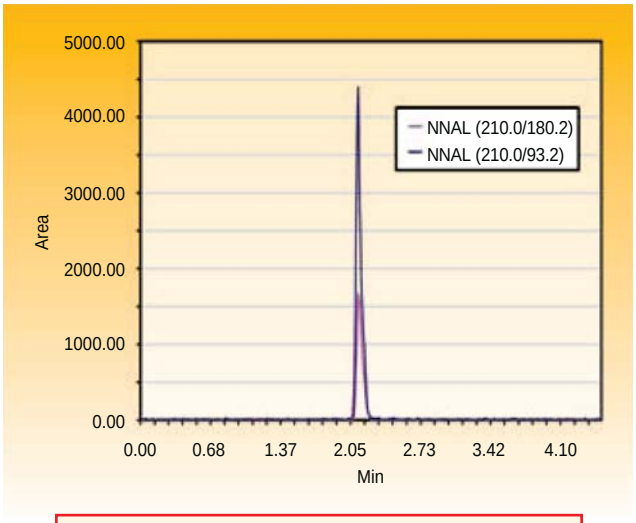
Achieve lower detection limits through superior selectivity	*Reduce ion-suppression*	*Save time and reduce cost via robust and rapid methodology*	*Minimal to no method development required*	*Stable at broad pH ranges and high temperatures*	*Stringent quality control conditions*

Tobacco Specific Nitrosamines (TSNAs) are highly carcinogenic and derived solely from tobacco products. They are generated from the fermentation, curing, and burning of tobacco. For example, NNAL is a valuable biomarker in human urine to determine exposure to second-hand smoke. Because TSNAs are often found in very low concentrations in difficult biological matrixes, a highly selective and sensitive assay is required for sample preparation and analysis.

MIP Technologies AB has developed two phases to address this issue. SupelMIP SPE – NNAL is designed for the extraction of NNAL, and SupelMIP SPE – TSNA is a class selective phase developed for the extraction of four different tobacco specific nitrosamines: NNK, NNN, NAB, and NAT.

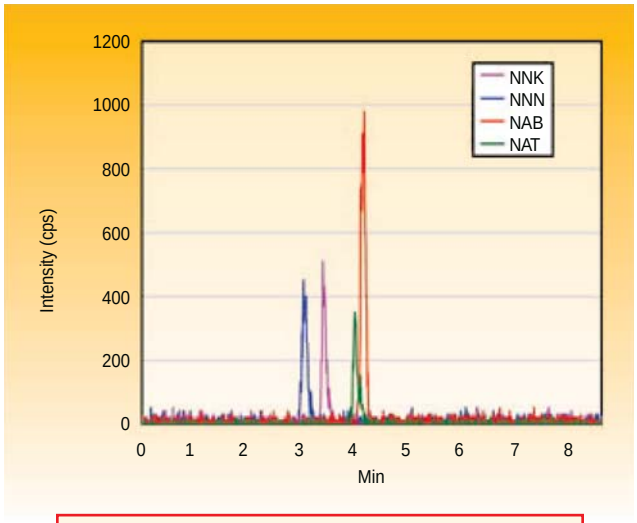
Figures 7 and 8 depict LC-MS-MS chromatograms (MRM) of SupelMIP extracts of human urine spiked with 1 ng/mL NNAL and 25 pg/mL TSNAs, respectively.

Figure 7. Representative LC-MS-MS Chromatogram (MRM) of a SupelMIP SPE – NNAL Extract of Human Urine Spiked with 1 ng/mL NNAL



SupelMIP SPE – NNAL:
- Consistent low background
- Recoveries greater than 90%
- Achieve LODs of 5 pg/mL
- Achieve LOQs of 13 pg/mL

Figure 8. Representative LC-MS-MS Chromatogram (MRM) of a SupelMIP SPE – TSNA Extract of Human Urine Spiked with 25 pg/mL TSNAs



SupelMIP SPE – TSNAs:
- Consistent low background
- Recoveries greater than 90%
- Achieve LODs of 2.5 pg/mL
- Achieve LOQs of 4 pg/mL

NNAL = 4-(methylnitrosamino)-1-(3-pyridyl)-1-butanol
NNK = 4-(methylnitrosamino)-1-(3-pyridyl)-1-butanone
NNN = N-nitrosonornicotine
NAB = N'-nitrosoanabasine
NAT = N'-nitrosoanatabine

Key Features & Benefits of SupelMIP:

Achieve lower detection limits through superior selectivity	***Reduce ion-suppression***	*Save time and reduce cost via robust and rapid methodology*	*Minimal to no method development required*	*Stable at broad pH ranges and high temperatures*	*Stringent quality control conditions*

Reduce Ion-Suppression

Ion-suppression or ion-enhancement is caused by one or more interfering components/species that co-elute with the analyte(s) of interest during LC-MS analysis. These co-eluting species can affect droplet formation or ionize concurrently resulting in an erroneous decrease (suppression) or increase (enhancement) in signal response. Ion-suppression often leads to poor assay reproducibility, accuracy, and sensitivity, and such deleterious effects are often most notable at the lower limits of quantitation.

In order to achieve adequate lower limits of quantitation when conducting trace analysis of analytes in complex matrices such as biological fluids, it is absolutely critical to procure adequate selectivity during sample preparation. By virtue of molecularly imprinted polymer technology, SupelMIP SPE offers the necessary selectivity and sample cleanup required for achieving ever-decreasing detection limits that are challenging analysts today.

Blank urine samples were extracted with SupelMIP SPE – NNAL and the resulting SPE (post-SPE) eluate was spiked with NNAL and analyzed via LC-MS-MS. The resulting chromatogram response (peak area) levels generated were compared against external standards (prepared in buffer). The results (Figure 9) show that ion-suppression was nominal (< 4% signal suppression) for the SupelMIP SPE – NNAL urine extract (post-SPE spike) relative to the external standard calibration curve.

In another study, blank urine samples were extracted with SupelMIP SPE Beta-agonists and conventional hydrophilic polymer SPE phases and the resulting SPE eluate was spiked (post-SPE) with metaproteronol at the levels of 0.5, 1, and 5 ng/mL, respectively. Figure 10 compares the response levels and linear relation of known spike concentrations vs. calculated concentrations determined from the signal responses obtained from blank urine extracts spiked post-extraction using both the SupelMIP SPE – Beta-agonist method and conventional polymer SPE method. Increased levels of ion-suppression were observed for the polymer SPE protocol relative to the SupelMIP procedure.

Figure 9. Response Comparison of NNAL Calibration Curve Generated from SupelMIP SPE – NNAL Urine Extract (post-SPE spike) vs. External Standards

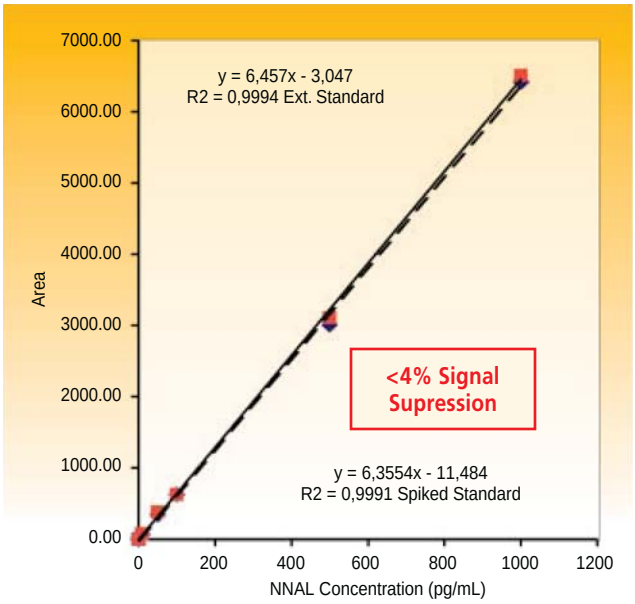
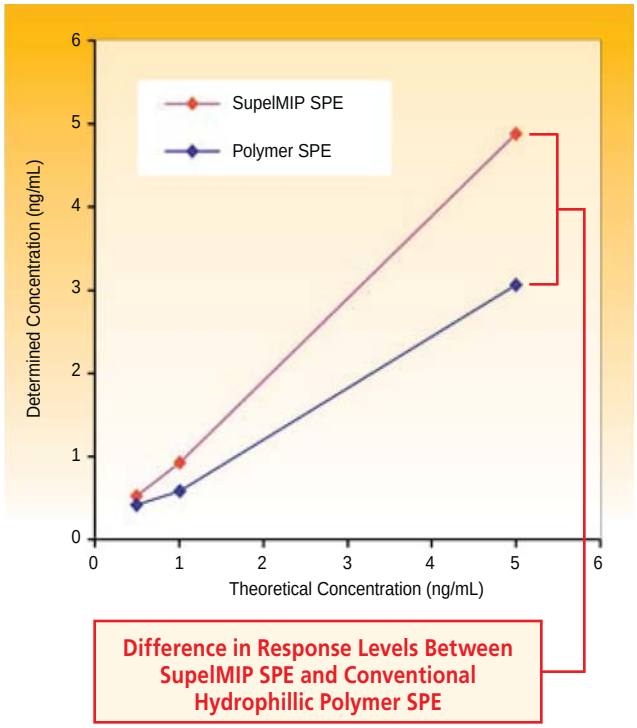


Figure 10. Known Spike Concentration vs. Determined Concentration for SupelMIP SPE Beta agonist and Polymer SPE for Metaproterenol from Urine (Post-Extraction Spike)



Key Features & Benefits of SupelMIP:

Achieve lower detection limits through superior selectivity	*Reduce ion-suppression*	***Save time and reduce cost via robust and rapid methodology***	*Minimal to no method development required*	*Stable at broad pH ranges and high temperatures*	*Stringent quality control conditions*

Save Time and Reduce Cost

Sample preparation is often the rate-limiting step within the analytical process, and can often take up to 10 times as long as the analysis in itself. It is therefore critical for analysts to develop simple, robust, and rapid extraction techniques that are selective enough to achieve sensitivity, precision, and accuracy limits required of the assay.

Table 3 describes the cost difference for the extraction of chloramphenicol using SupelMIP SPE – Chloramphenicol vs. a conventional sample prep method that utilizes a common commercially available hydrophilic polymer SPE phase (1).

Time data depicted in Table 3 courtesy of Dr. Philippe A. Guy, Nestlé Research Center, Nestec Ltd., Lausanne, Switzerland.

Total Cost and Sample Prep/Analysis Time Reduced by 75% using SupelMIP SPE - Chloramphenicol.

Table 3. Comparison of SupelMIP SPE Method and Conventional Method Using a Hydrophilic Polymer SPE Phase

SupelMIP SPE - Chloramphenicol Method Sample

Pre-Treatment:

Whole pasteurized milk (purchased from the local supermarket) was centrifuged for 15 min. at 5k rpm. The aqueous lower layer was spiked with chloramphenicol at the level of 15 ng/mL and 38 ng/mL.

SPE Procedure:

SupelMIP SPE – Chloramphenicol, 25 mg/10mL (LRC) (53210-U)

1. Condition and equilibrate MIP phase with 1 mL methanol followed by 1 mL DI water.
2. Apply 1 mL of the pre-treated milk sample to the cartridge.
3. Elute interferences using the following wash scheme:
   2 x 1 mL MS-grade water
   1 mL 5% acetonitrile in 0.5% acetic acid
   2 x 1 mL MS-grade water
   1 mL 20% acetonitrile in 1% ammonium hydroxide
   Dry SPE tubes for 5 min. under gentle vacuum
   3 x 1 mL dichloromethane
   Dry SPE tubes for 1 min. under gentle vacuum
4. Elute chloramphenicol with 2 x 1 mL methanol:acetic acid:MS-grade water (89:1:10, v/v/v)
5. Evaporate combined eluate to dryness at 50 °C under nitrogen. Reconstitute 150 µL LC mobile phase prior to LC-MS analysis.

SupelMIP SPE - Chloramphenicol
Sample Prep/Analysis Time: 1.5 hrs.
Sample Prep Analysis Cost (140 EUR/hr.) = 195 EUR
Total Cost of Extraction = 202.5 EUR (275 USD)

Published Chloramphenicol Method Using Conventional Hydrophilic Polymer SPE Phase

Sample Pre-Treatment:

5 mL of milk was spiked with 40 ng chloramphenicol. Proteins were precipitated by the addition of 15 mL 10% trichloracetic acid in water. The sample was vortexed and heated for 1 hour at 65 °C. After cooling to room temperature, the mixture was centrifuged for 15 min. at 3K rpm. The supernatant was filtered over glass wool, and the filtered was rinsed with 10 mL DI water. The pH of the filtrate was adjusted to pH 5 with 0.1 M sodium acetate.

SPE Procedure:

Conventional Hydrophilic Polymer SPE, 500 mg/12 mL

1. Condition and equilibrate SPE phase with 3 mL methanol, 4 mL DI water, and 4 mL 10 mM HCl
2. Apply the pre-treated milk extract to the cartridge.
3. Elute interferences using the following wash scheme:
   4 mL MS-grade water
   2 mL 5% methanol
   2 mL 50% methanol
4. Elute chloramphenicol with 2 mL methanol
5. Evaporate combined eluate to dryness at 50 °C under nitrogen. Reconstitute 0.4 mL DI water

Liquid-Liquid Extraction

1. Liquid-liquid extract of reconstituted eluate with 0.6 mL acetonitrile: dichloromethane (4:1, v/v).
2. Centrifuge at 7k rpm for 5 min. Transfer upper organic layer to a fresh tube.
3. Repeat steps 1 & 2 of the LLE procedure two additional times on the lower aqueous layer.
4. Combine all organic layers, evaporate to dryness at 60 °C under nitrogen. Reconstitute with 0.2 mL LC mobile phase and filter through a 0.2 µm nylon filter.

Published Chloramphenicol Method
Sample Prep/Analysis Time: 6.5 hrs.
Sample Prep Analysis Cost (140 EUR/hr.) = 845 EUR
Total Cost of Extraction = 855 EUR (1,161 USD)

Key Features & Benefits of SupelMIP:

Achieve lower detection limits through superior selectivity	*Reduce ion-suppression*	*Save time and reduce cost via robust and rapid methodology*	***Minimal to no method development required***	*Stable at broad pH ranges and high temperatures*	*Stringent quality control conditions*

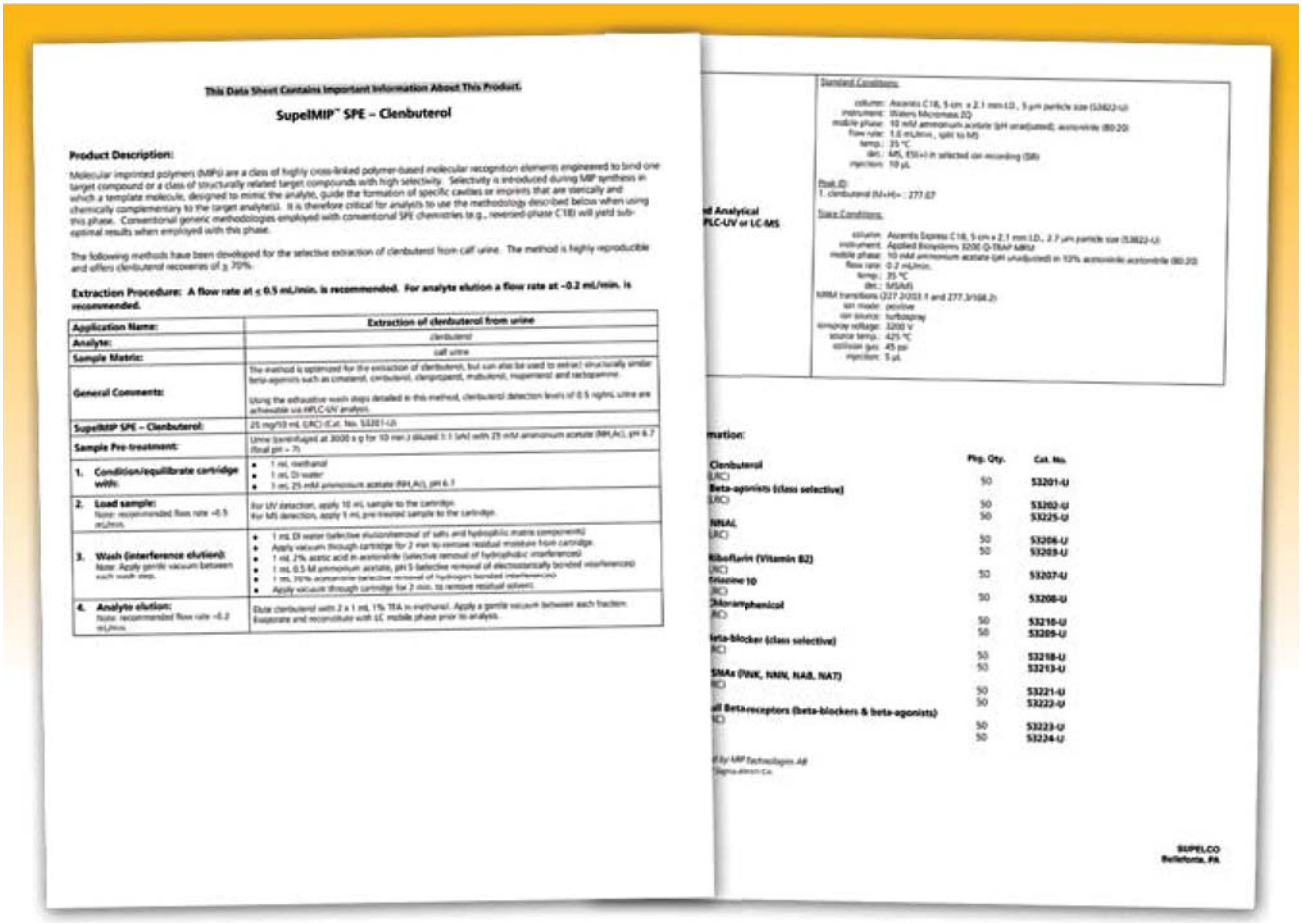
Minimal to No Method Development Required

Sample prep methods are often developed using a variety of schemes such as: referring to published methods of similar/identical applications; implementation of generic methodology; requesting support from a chromatography vendor; screening of techniques, phase chemistries, and method conditions. These approaches are often effective; however, more often than not, a sample prep method can often be frustrating and time consuming.

Unlike many traditional sample prep techniques, SupelMIP is developed and tailored for very specific applications. Therefore, each SupelMIP SPE phase comes with a detailed protocol and analytical technique for its respective application.

Figure 11 depicts a typical data/instruction sheet that is included with each SupelMIP SPE phase.

Figure 11. Example of a Typical Data/Instruction Sheet Included With Each SupelMIP SPE Phase



This Data Sheet Contains Important Information About This Product.

SupelMIP™ SPE – Clenbuterol

Product Description:

Molecular imprinted polymers (MIPs) are a class of highly cross-linked polymer-based molecular recognition elements engineered to bind one target compound or a class of structurally related target compounds with high selectivity. Selectivity is introduced during MIP synthesis in which a template molecule, designed to mimic the analyte, guide the formation of specific cavities or imprints that are sterically and chemically complementary to the target analyte(s). It is therefore critical for analysts to use the methodology described below when using this phase. Conventional generic methodologies employed with conventional SPE chemistries (e.g., reversed-phase C18) will yield sub-optimal results when employed with this phase.

The following methods have been developed for the selective extraction of clenbuterol from calf urine. The method is highly reproducible and offers clenbuterol recoveries of ≥ 70%.

Extraction Procedure: A flow rate at ≤ 0.5 mL/min. is recommended. For analyte elution a flow rate at ~0.2 mL/min. is recommended.

Application Name:	Extraction of clenbuterol from urine
Analyte:	clenbuterol
Sample Matrix:	calf urine
General Comments:	The method is optimized for the extraction of clenbuterol, but can also be used to extract structurally similar beta-agonists such as cimaterol, cimbuterol, clenproperol, mabuterol, mapenterol and rechopamine. Using the extraction wash steps detailed in this method, clenbuterol detection levels of 0.5 ng/mL urine are achievable via HPLC-UV analysis.
SupelMIP SPE – Clenbuterol:	25 mg/10 mL (LRC) (Cat. No. 53201-U)
Sample Pre-treatment:	Urine (centrifuged at 3000 x g for 10 min.) diluted 1:1 (v/v) with 25 mM ammonium acetate (NH4Ac), pH 6.7 (final pH ~ 7)
1. Condition/equilibrate cartridge with:	• 1 mL methanol • 1 mL DI water • 1 mL 25 mM ammonium acetate (NH4Ac), pH 6.7
2. Load sample: Note: recommended flow rate ~0.5 mL/min.	For UV detection, apply 10 mL sample to the cartridge. For MS detection, apply 1 mL pre-treated sample to the cartridge.
3. Wash (interference elution): Note: Apply gentle vacuum between each wash step.	• 1 mL DI water (selective elution/removal of salts and hydrophilic matrix components) • Apply vacuum through cartridge for 2 min to remove residual moisture from cartridge. • 1 mL 2% acetic acid in acetonitrile (selective removal of hydrophilic interferences) • 1 mL 0.5 M ammonium acetate, pH 5 (selective removal of electrostatically bonded interferences) • 1 mL 35% acetonitrile (selective removal of hydrogen bonded interferences) • Apply vacuum through cartridge for 2 min. to remove residual solvent.
4. Analyte elution: Note: recommended flow rate ~0.2 mL/min.	Elute clenbuterol with 2 x 1 mL 1% TFA in methanol. Apply a gentle vacuum between each fraction. Evaporate and reconstitute with LC mobile phase prior to analysis.

Key Features & Benefits of SupelMIP:

- *Achieve lower detection limits through superior selectivity*
- *Reduce ion-suppression*
- *Save time and reduce cost via robust and rapid methodology*
- *Minimal to no method development required*
- ***Stable at broad pH ranges and high temperatures***
- ***Stringent quality control conditions***

High Stability

SupelMIP SPE consists of highly cross-linked polymers that maintain stability when exposed to a broad range of organic solvents, can withstand high temperatures, and can be used over broad pH ranges, without loss of selectivity. Furthermore, they can be stored at room temperatures for prolonged periods of times. This is extremely advantageous over immunoaffinity based products.

Stringent Quality Control Conditions

SupelMIP SPE phases are manufactured by MIP Technologies AB. Each lot is subjected to stringent QC conditions to ensure low batch-to-batch variation. MIP based SPE technology is currently employed by a number of industrial and regulatory agencies for routine analysis. References from these organizations are available upon request.

Frequently Asked Questions (FAQs):

1. How is sample preparation improved using molecularly imprinted polymer SPE technology?

Because MIPs are tailor-made for individual analytes and analyte classes, analyte retention strength is increased significantly allowing for powerful wash steps within the SPE procedure. This allows for highly selective and simple extractions resulting in lower detection limits and improved MS compatibility (reduced ion-supression). Each SupelMIP phase also comes with a detailed application specific protocol simplifying the method development process which in turn saves time and cost.

2. Are sample packs available?

Yes. Sample packs are available and can be obtained through the SupelMIP website: ***sigma-aldrich.com/supelmip***
Alternatively, you can also request a sample pack by calling or emailing your local Sigma-Aldrich office and connecting with technical service.

3. There is no MIP phase for my application? How do I develop a MIP protocol for my application?

Within the SupelMIP website, ***sigma-aldrich.com/supelmip***, there is a survey where you can describe your application and needs for MIP based SPE product/procedure. Scientists from both Supelco and MIP Technologies AB will evaluate your application through a short feasibility stage. If your application is prioritized to move through feasibility, the next stages will be development and optimization. The latter two stages can often take up to 8 months; however, we are in the process of streamlining how we develop and approach new SupelMIP applications.

4. Are process scale MIP products available through Supelco?

No. Process scale MIP products are not available through Supelco. Please contact MIP Technologies AB directly by visiting *www.miptechnologies.com*

5. Can we use existing or traditional SPE protocols with SupelMIP SPE technology?

No. Existing protocols cannot be used. Every SupelMIP SPE includes a detailed extraction protocol that is analyte and matrix specific. This protocol needs to be used in order to achieve optimal retention during sample load, maximum interference removal during sample wash, and high recoveries during elution.

6. What dimensions are available for SupelMIP SPE?

Currently, our standard product consists of 25 mg bed weights (50 mg for SupelMIP SPE – TSNAs) packed in 3 mL and 10 mL LRC (large reservoir cartridges) SPE tubes. The phases can be custom packed in all other SPE hardware that Supelco offers (other SPE tube dimensions, glass SPE tubes, 96-well plates, etc.)

Ordering Information

SupelMIP SPE Cartridges	Sorbent Mass (mg)	Cartridge Volume (mL)	Cartridges per Box	Cat. No.
PAHs	25	3	50	**52773-U**
Nitroimidazoles	50	3	50	**52734-U**
NSAIDs	25	3	50	**52769-U**
NSAIDs	25	10	50	**52772-U**
Fluoroquinolones	25	3	50	**53269-U**
Amphetamines	25	3	50	**52228-U**
Clenbuterol	25	10	50	**53201-U**
Beta-agonists (class selective)	25	10	50	**53202-U**
Beta-agonists (class selective)	25	3	50	**53225-U**
Beta-blockers (class selective)	25	10	50	**53218-U**
Beta-blockers (class selective)	25	3	50	**53213-U**
Full Beta Receptor (Beta-agonists and Beta-blockers)	25	10	50	**53223-U**
Full Beta Receptor (Beta-agonists and Beta-blockers)	25	3	50	**53224-U**
Chloramphenicol	25	10	50	**53210-U**
Chloramphenicol	25	3	50	**53209-U**
NNAL (4-(methylnitrosamino)-1-(3-pyridyl)-1-butanol)	25	10	50	**53206-U**
NNAL (4-(methylnitrosamino)-1-(3-pyridyl)-1-butanol)	25	3	50	**53203-U**
TSNAs (4 different Tobacco specific Nitrosamines: NNK, NNN, NAB, NAT)	50	10	50	**53221-U**
TSNAs (4 different Tobacco specific Nitrosamines: NNK, NNN, NAB, NAT)	50	3	50	**53222-U**
Riboflavin (vitamin B2)	25	10	50	**53207-U**
Triazines (class selective)	25	10	50	**53208-U**

For a complete SupelMIP product listing, and to request a SupelMIP sample pack, visit ***sigma-aldrich.com/supelmip***

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- Amphetamine and related compounds in urine
- Chloramphenicol in milk, plasma, honey, urine, and shrimp/prawns
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- TSNAs in urine and tobacco
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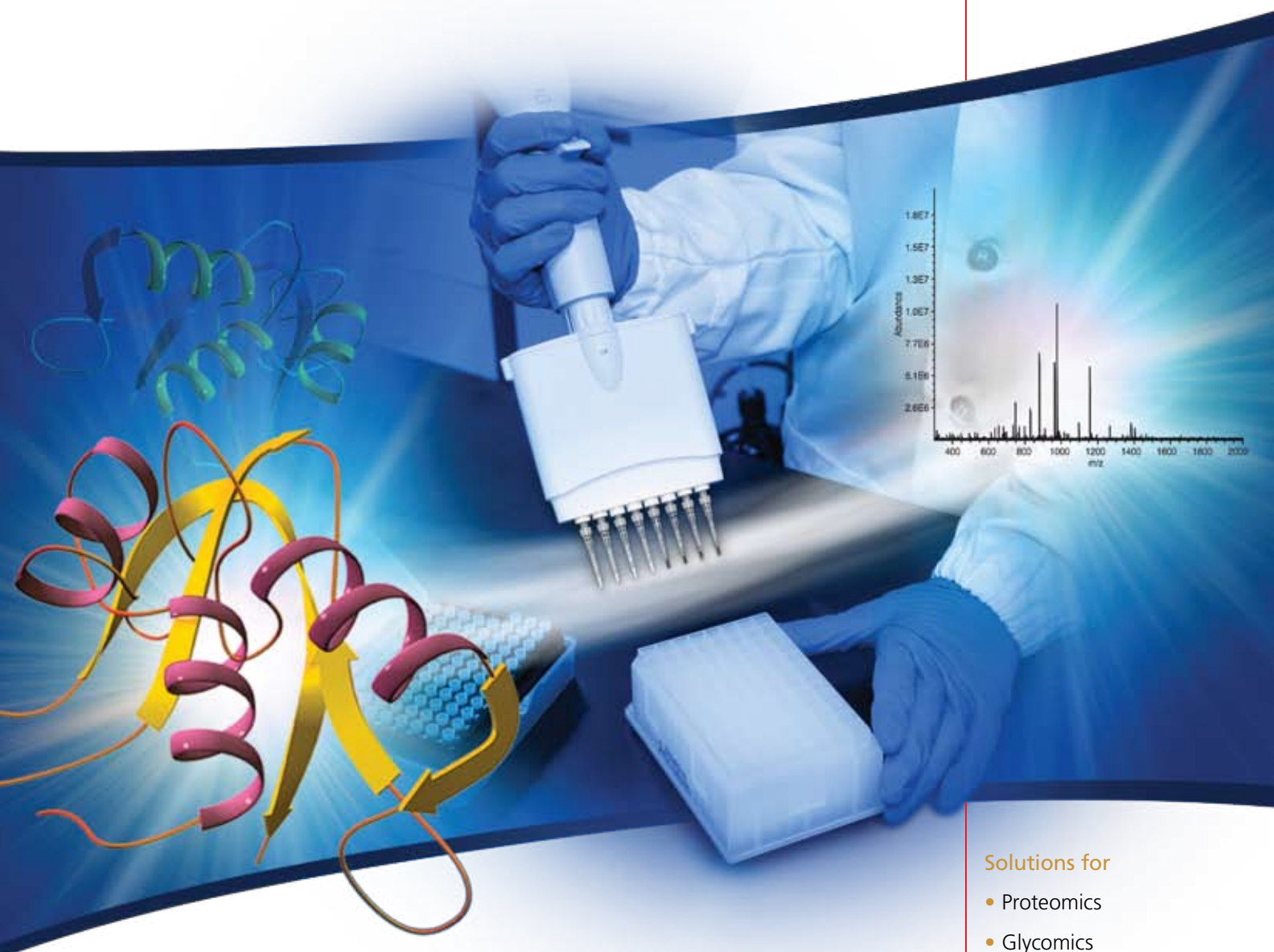
Supel™-Tips

Pipette Tips for Micro-Purification/Enrichment



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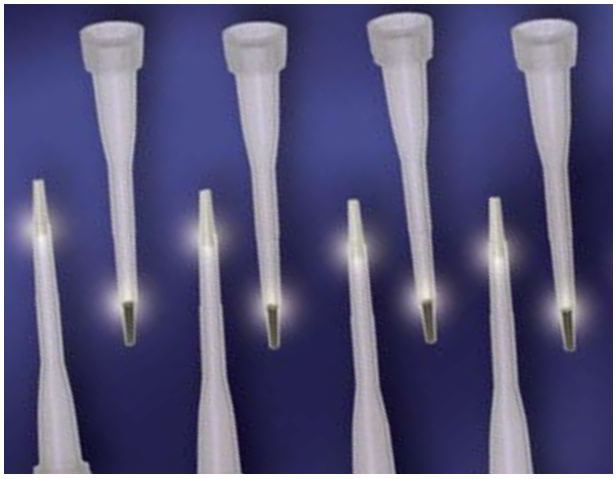
Pipette Tips



P001235

Pipette tips are convenient and useful tools for extraction, concentration, and/or purification of complex bio-molecules through hydrophobic interactions. Supelco's Supel-Tips are designed to purify and enrich femtomole to picomole quantities of desired samples for subsequent analysis and identification with mass spectrometric and/or chromatographic techniques.

Supel-Tips contain a chromatography sorbent bed bonded at the working end of the tip with a proprietary high purity adhesive. This sorbent bed acts as a solid phase extraction medium that adsorbs molecules of interest from the sample matrix. Subsequently the concentrated, desalted analytes are eluted for down-stream analysis.

Supel-Tips provide the following separation benefits:

- Superior recovery
- Exceptional binding capacity and enhanced affinity
- Excellent sorbent bed stability for cleaner samples
- Fast and effective analyte retention/elution

Applications and Suitable Products for Micro-Purification/Enrichment of:

Phosphopeptides: **Supel-Tips Zr & Supel-Tips Ti**

Proteins & Peptides: **Supel-Tips C18**

Oilgosaccharides/
Similar Molecules: **Supel-Tips Carbon**

Supel-Tips C18 Pipette Tips

Cat. No.: TPSC18

Applications: **Sample preparation for proteins and and peptide analysis**

Features:

- 10 µL polypropylene pipette tips
- C18 bonded spherical silica-based sorbent
- Sorbent particle size of 50-60 µm, pore size of 200 Å

Benefits:

- Excellent recovery and reproducibility
- Effective for achieving high capacity for trace-level peptides

Excellent Recovery

Peptide	(M+H)+ Monoisotopic	Recovery by HPLC (%)
Insulin, Chain B, Oxidized	3494	89
β-Amyloid	1446	100
Bradykinin, Fragment 1-7	757	78

Supel-Tips Carbon Pipette Tips

Cat. No.: 54227-U

Applications: **Sample preparation for analysis and identification of oligosaccharides and other macromolecules containing sugar moieties**

Features:

- 10 µL polypropylene pipette tips
- Graphitized carbon adsorbent
- Adsorbent particle size of 50-60 µm, pore size is 175 Å
- Proprietary high purity adhesive

Benefits:

- Higher binding capacity
  1. Maltahexose: 10.2 µg
  2. Glycopeptide (mol. wt.: 1300 – 3500): >10 µg
- Large sample volume of 0.5-10 µL
- Fast and effective analyte transport

NEW Tips for Phosphopeptide Enrichment

Zirconia-Silica and Titania-Silica Composite Pipette Tips

Supel-Tips Zr Pipette Tips (Cat. No.: 54266-U)
Supel-Tips Ti Pipette Tips (Cat. No.: 54263-U)

Applications: **Phosphopeptide purification for analysis and identification**

Features:

- 10 µL polypropylene micropipette tips
- Zirconia-silica or Titania-silica composite adsorbent
- Adsorbent particle size of 50-60 µm
- Adsorbent average pore size is 300 Å
- Proprietary, composite particles and adhesives

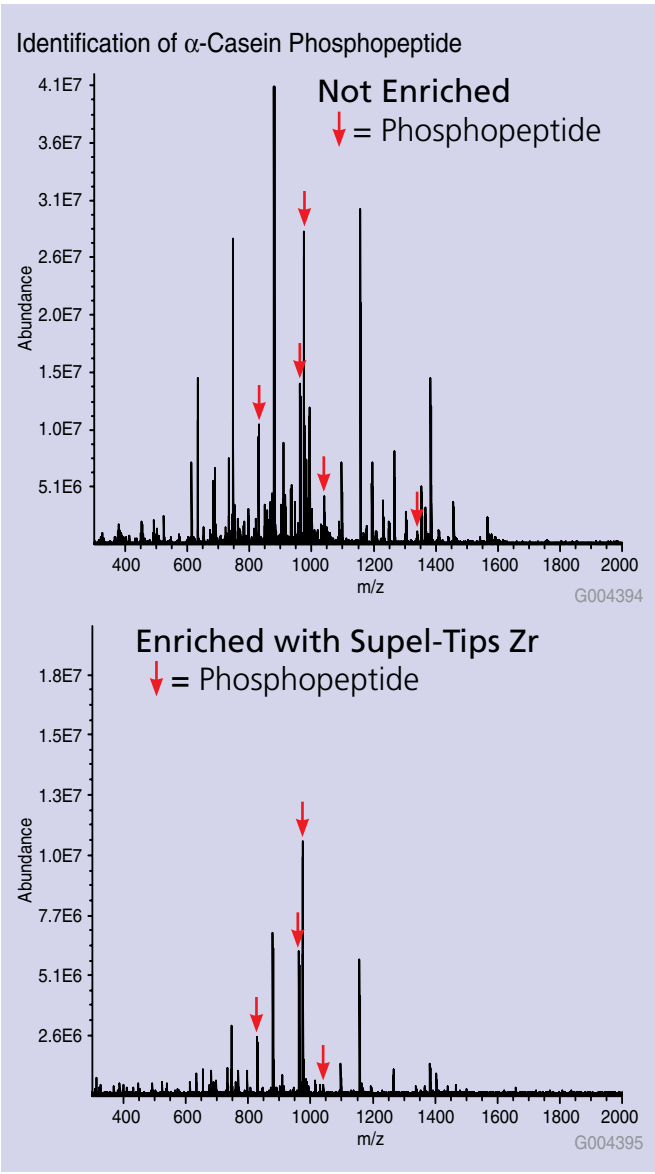
Benefits:

- Excellent recovery and reproducibility
- Effective for achieving high capacity for trace-level peptides

Identification

Phosphopeptide:
Bovine Casein, Monophosphopeptide
[M+H]+ Monoisotopic (2061)

Analysis (by MS+MS/MS)	Confidence Index %
Supel-Tips Zr	100
Supel-Tip Ti	100



Chemical Compatibility:

Supel-Tips are compatible with most organic solvents, buffers and alkaline solutions. They are not compatible with concentrated inorganic acids such as hydrochloric and nitric acids.

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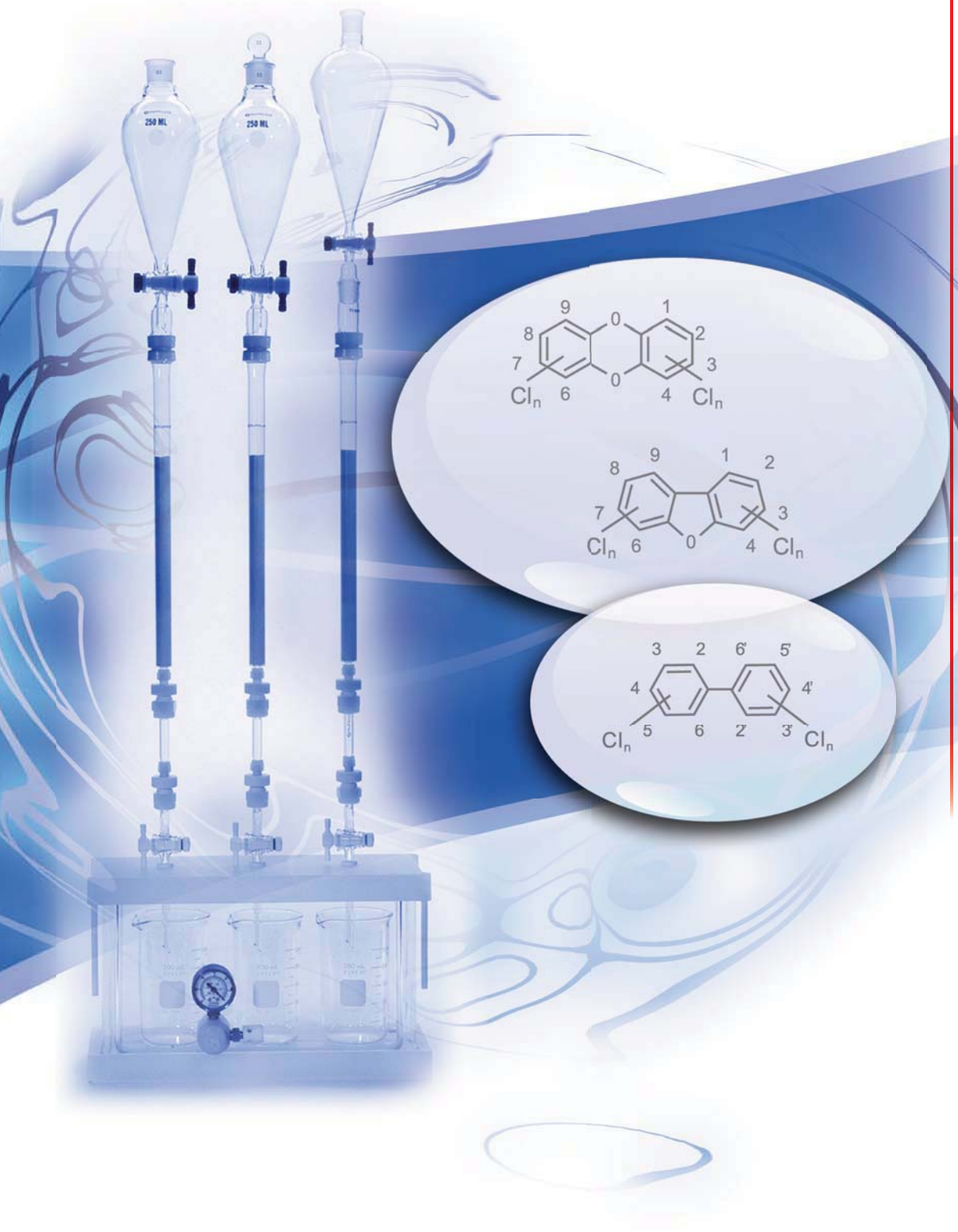


Sample Clean up

GC Analysis

Air Monitoring

Resins

Solvents

Introduction

Figure 1. Dioxin / Furan skeletal structure, can be chlorinated at any of the suitable positions on the aromatic ring

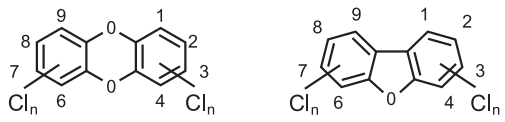
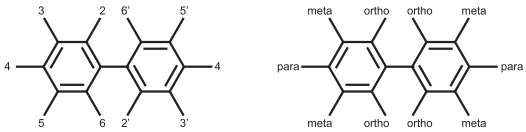


Figure 2. Biphenyl skeletal structure of PCBs



What are Dioxins?

Dioxins and PCBs belong to the group of compounds known as Persistent Organic Pollutants (POPs). They are known to bio accumulate due to their lipophilic nature and, therefore, have health implications. As a result their emission into the environment and food chain is strictly controlled. Samples that are analysed, amongst others, are foodstuffs like fish, fish feed, and stack emissions from waste incineration sites. Limits are published by the World Health Organisation (WHO) and local authorities. As a consequence, low levels of contamination have to be detected, providing a challenge to sample preparation and detection systems.

Compounds of Interest

The term 'Dioxin' covers a wide range of halogenated aromatic compounds, including polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans (PCDDs and PCDFs). These compounds are formed as a result of incomplete combustion of hydrocarbons in the presence of chlorine e.g. metal processing, domestic waste incineration, etc. They have high melting points and are stable to acids and bases; these characteristics make them very persistent in the environment. PCDD/Fs can be found in many environmental matrices such as soils, air, and water.

The basic structure of PCDD/Fs comprises two benzene rings joined by either a single (furan) or a double oxygen bridge (dioxin), see Figure 1.

There are 210 possible combinations of chlorine atoms on the skeletal structure of dioxins and furans. However, only a few congeners are considered to have significant risk to human health. The toxicity of these compounds is measured in TEF (Toxic Equivalence Factor), which is an internationally recognised calculation that weighs the toxicity of each individual congener against the most toxic compound in that family, in the case of PCDD/PCDF, this is 2,3,7,8-TCDD. The closer the ratio is to unity, the greater the toxicity of that congener. Calculation of the total toxicity of a sample is achieved by multiplying the concentrations of the individual target compounds by their respective TEFs. These values are known as TEQs (Toxic Equivalents); and the total TEQ of a sample is obtained by summing the individual TEQs.

In addition to PCDF and PCDD, some polychlorinated biphenyls (PCBs) (Figure 2) that are similar in structure and lipophilic properties as the dioxins have been identified as having similar toxic health effects. These are often referred to as non-ortho, coplanar, or dioxin like PCBs, and their TEF is also measured against 2,3,7,8-TCDD. For example, a PCB congener with a TEF of 0.01 is considered to be one hundred times less toxic than 2,3,7,8-TCDD (see Table 1).

Non-ortho PCBs are those which are not chlorinated at the ortho position, and as such are free to rotate around the single carbon carbon bond, resulting in a co-planar ('flat') configuration; PCBs that have a single ortho chlorine are also able to adopt a relatively planar arrangement; the twelve possible congeners that obey these rules are known as the WHO-12 PCBs. These compounds are monitored along with the dioxins (see Table 1).

Table 1.

IUPAC No.	Type	Structure	WHO-TEF
77	Non-ortho	3,3'4,4'-TeCB	0.0001
81		3,4,4',5-TeCB	0.0001
126		3,3',4,4',5-PeCB	0.1
169		3,3',4,4'5,5'-HxCB	0.01
105	Mono-ortho	2,3,4,4'5-PeCB	0.0005
114		2,3,4,4',5-PeCB	0.0005
118		2,3',4,4',5-PeCB	0.0001
123		2',3,4,4',5-PeCB	0.0001
156		2',3,3',4,4',5-HxCB	0.0005
157		2',3,3',4,4',5'-HxCB	0.0005
167		2,3',4,4',5,5'-HxCB	0.00001
189		2,3,3',4,4',5,5'-HpCB	0.0001

Abbreviations	
HRGC	High Resolution Gas Chromatography
HRMS	High Resolution Mass Spectrometry
PCDDs	Polychlorinated Dibenzo-p-dioxins
PCDFs	Polychlorinated Dibenzofurans
PCBs	Polychlorinated Biphenyls
TCDD	Tetrachloro Dibenzo-p-dioxin
TEQs	Toxic Equivalents
TEF	Toxic Equivalent Factor
WHO	World Health Organisation

Table 2. Limits for Environmental Samples (EU)

Sample Matrix	Measured Typical Range	Max. Conc. for Contaminated Sites	Units
Soil	<1 - 100	100.000	ng I-TEQ/kg d.m.
Sediment	<1 - 200	80.000	ng I-TEQ/kg d.m.
Air (ambient)	<1 - 100s	14.800	fg I-TEQ/m³
Air (bulk deposition)	<1 - 100s	14.800	pg I-TEQ/m³
Sewage Sludge	<1 - 200 (average 15 - 40)	1.200	ng I-TEQ/kg d.m.
Spruce/Pine Needles (biomonitors)	0.3 - 1.9	100	ng I-TEQ/kg d.m.

Source: http://ec.europa.eu/environment/dioxin/pdf/task2.pdf
d.m. = dried matter

Table 3. Limits for Foodstuff (EU)

Sample	WHO-PCDD/F-TEQ/g fat or product
Milk and milk products, including butter fat	3 pg/g fat
Hen eggs and egg products	3 pg/g fat
Liver and derived products	6 pg/g fat
Fish oil	2 pg/g fat
Fish (flesh)	4 pg/g fresh weight

Source: http://www.gafta.com/fin/findioxin.html

The limits allowed in various matrices are published by the WHO and other local authorities. Examples for European environmental levels and foodstuff limits (status Feb/2007) are given below (Tables 2. & 3.). The current EU Commission Maximum Levels for dioxins are contained in Commission Regulation 466/2001, amended by Council Regulation 2375/2001. This was implemented in July 2002 and became effective February 2003. These are general guidelines, actual limits and measured concentrations can vary from country to country.

http://ec.europa.eu/environment/dioxin/download.htm

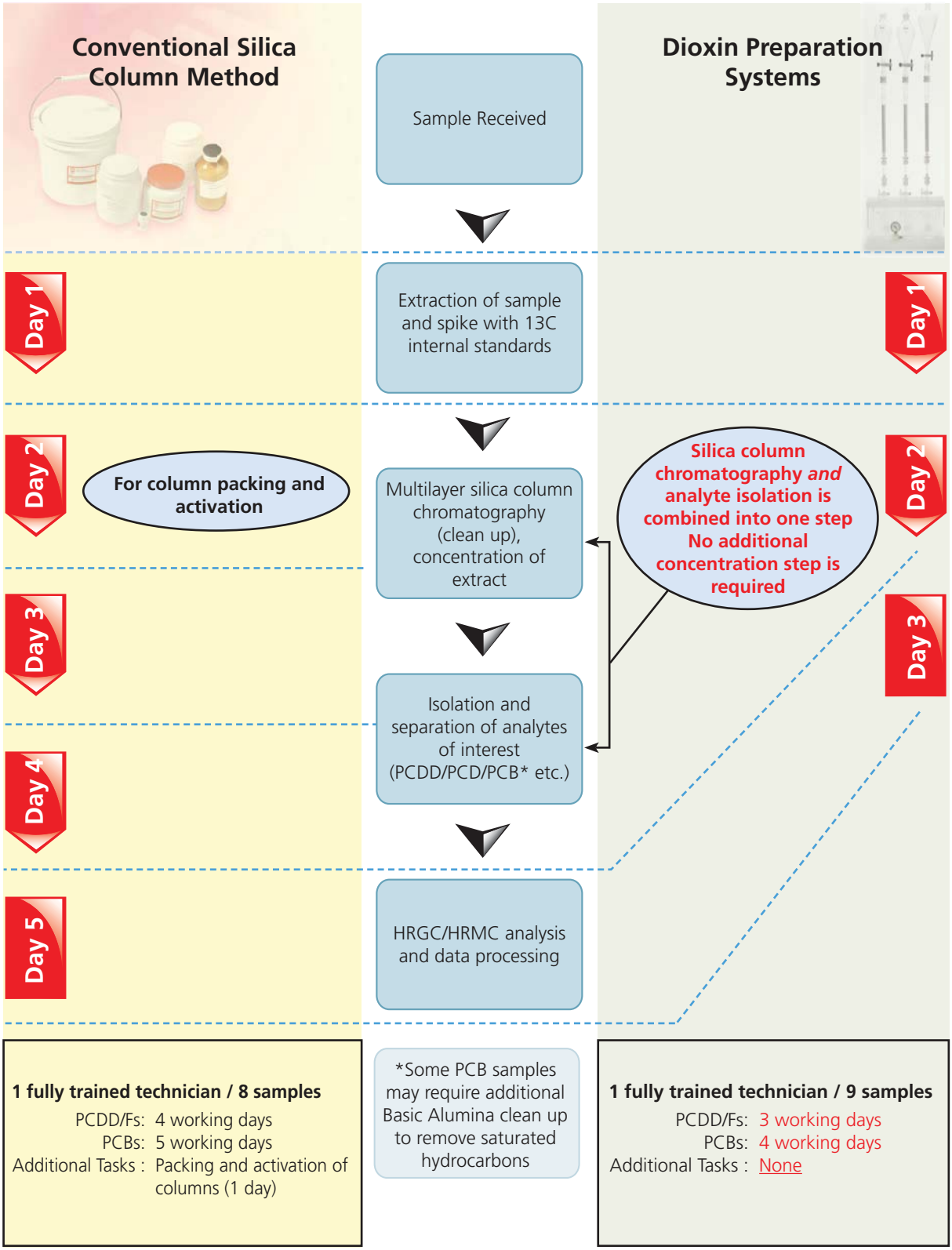
Contents

Accredited Methods

Several accredited methods for sample collection, clean up and analysis exist. These include (also see p.13):

Accredited Methods		
	EPA Method 1613b	Tetra- through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS
	EPA Method 1668	Chlorinated Biphenyl Congeners in Water, Soil, Sediment, and Tissue by HRGC/HRMS
	EPA Method 8290A	Polychlorinated Dibenzodioxins (PCDDs) and Polychlorinated Dibenzofurans (PCDFs) by High-Resolution Gas Chromatograph/High-Resolution Mass Spectrometry (HRGC/HRMS)
	EN 1948-1	Stationary source emissions. Determination of the mass concentration of PCDDs/PCDFs and dioxin-like PCBs. Sampling of PCDDs.PCDFs
	EN 1948-2	Stationary source emissions. Determination of the mass concentration of PCDDs/PCDFs and dioxin-like PCBs. Extraction and clean-up
	EN 1948-3	Stationary source emissions. Determination of the mass concentration of PCDDs/ PCDFs and dioxin-like PCBs. Identification and quantitation, sample collection collection and clean up general information
	JSA JIS K 0311:2005	Method for determination of tetra- through octachlorodibenzo-p-dioxins, tetra- through octachlorodibenzofurans and dioxin-like polychlorinated biphenyls in stationary source emissions
	JSA JIS K 0312	Method for determination of tetra- through octachlorodibenzo-p-dioxins, tetra- through octachlorodibenzofurans and dioxin-like polychlorinated biphenyls in industrial water and waste water limits

Sample Preparation for Dioxin and PCB Analysis: Conventional vs. Supelco System



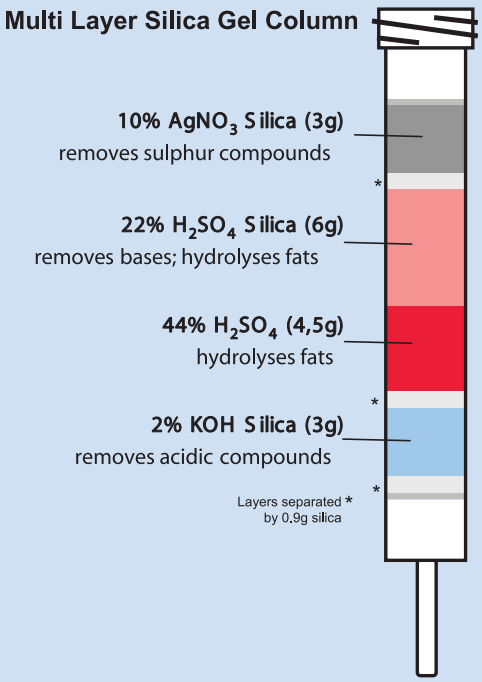
The Supelco Dioxin Prep System

The Supelco Dioxin Prep system provides a highly efficient means of extracting and isolating dioxins, furans, and PCBs from stack gases, wastewater, soil, food, blood, and milk. The prep system design reduces solvent usage, decreases sample preparation time by 1-2 days, and results in extraction recoveries greater than 85%.

The convenient multi-layer silica gel column is key to the extraction process; seven layers of treated silica oxidize, reduce, and separate polar interferences. The modular glassware and hardware design makes it convenient for analysts to select only a few pieces or the entire prep system for their extraction needs. A vacuum adapter and a vacuum manifold provide the option of running a single sample or multiple samples at one time, using vacuum or gravity feed.

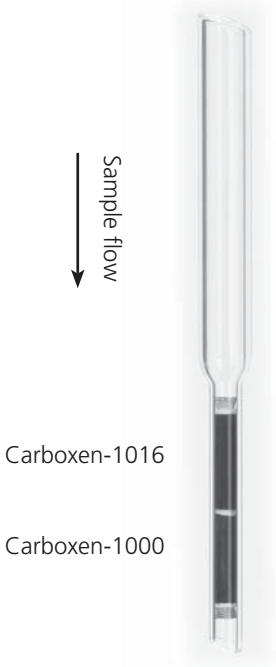
Multi-layer Silica Gel Dioxin Column

Potential chromatographic interferences are removed from the sample as it migrates through the several layers of treated silica gel. The silver nitrate treated layer removes sulphur-containing compounds; whilst two sulphuric acid treated layers oxidise sample lipids and remove any basic analytes. The potassium hydroxide treated layer removes any acidic sample components. Dioxins, furans, and PCBs pass through the silica column unretained. The column design includes an elongated tapered end that slips inside the dual-layer carbon reversible tube or Florisil micro column, preventing leakage of solvent and sample as well as contamination of/by the PTFE fittings. For very dirty samples, bulk treated silica gels and empty glass tubes are available to customise packings to meet individual sample needs.



Dual-layer Carbon Reversible Tube

Originally developed for the Japanese market in accordance with JIS method K-0311 and K-0312, a unique dual-layer carbon reversible tube isolates and concentrates the non-ortho PCBs, dioxins, and furans with a minimum of hexane and toluene. Isolation and separation is based on the two layers of carbon having different affinities for such compounds. Carboxen-1016 provides a low surface area (75 m^2/g), whilst Carboxen-1000 has a high surface area (1200 m^2/g). The combination of the two Carboxen™ layers isolate the dioxins, furans, and non-ortho PCBs. Any aliphatic hydrocarbons and the remaining PCBs present in the sample pass completely through the carbon tube into a waste fraction. The carbon tube is then removed and flushed in reverse direction with toluene to collect the dioxins, furans, and non-ortho PCBs.



Dual-Layer Reversible Carbon Column

Dioxin Prep System - Florisil Version

Micro-column & ampoulised Florisil®
(28309-U & 48924-U)
to be packed just before use

Figure 3. Elution efficiency with hexane followed by toluene for Dual-Layer Reversible Carbon Tube - Recoveries of dioxins, furans and PCBs

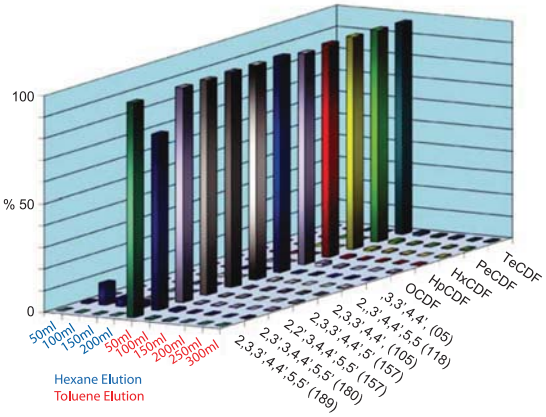
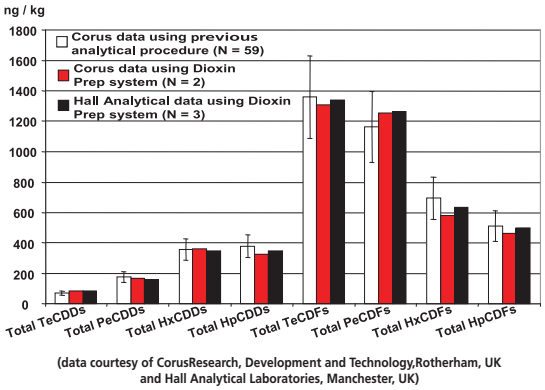


Figure 4. Dioxin extraction from waste ESP# dust using the Dual-Layer Reversible Carbon Tube System and comparison to previously used method (#electrostatic percipitator)



(data courtesy of CorusResearch, Development and Technology,Rotherham, UK and Hall Analytical Laboratories, Manchester, UK)

In 1998, the World Health Organization identified 12 polychlorinated biphenyls (PCBs) that exhibit dioxin-like activities. These WHO-12 PCBs are now included as part of the overall dioxin concentration and should be systematically investigated in industrial emissions. The original Dioxin Sample Prep System (Multi-layer Silica Gel Dioxin Column plus Dual-layer Carbon Reversible Tube) is ideal for the rapid cleanup and isolation of polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans (PCDD/Fs). However the extraction of PCBs can prove more challenging requiring multiple fractionation steps. As not all the WHO-12 PCBs are non-ortho, some of these compounds of interest will not be retained. These will pass through the carbon tube directly into the waste fraction, resulting in a split of the PCBs into two fractions. To address this issue a "Dioxin Prep System–Florisil Version" was developed in collaboration with Corus Research, Development and Technology, Rotherham, UK, and Hall Analytical Laboratories, Manchester, UK.

In this new system, the Dual-Layer Carbon Reversible Tube is replaced with a micro-column (reversible tube) packed with Pre-Activated Florisil. As the sample extract passes through the multilayer silica gel column and onto the Florisil micro-column, the relatively weak retention of all the PCBs means they can be easily eluted with n-hexane and/or n-hexane/dichloromethane mixtures. The subsequent PCB fraction contains all PCBs and can be further treated by basic alumina clean-up to remove any saturated hydrocarbons before analyzed by GC/MS. Further elution of the Florisil micro-column with dichloromethane is used to collect the PCDD/F fraction. As a result, the new "Dioxin Prep System–Florisil Version" can rapidly separate PCBs from PCDD/Fs prior to analysis for simpler quantitative determination.

For convenience, ampoulised Pre-Activated Florisil is available for use with the Dioxin Prep System. The Florisil ampoule is snapped open and emptied into an empty micro-column (reversible tube), 6.35/10 mm O.D. before sample clean up is performed.

How does it compare? - Extraction Recoveries

The multi-layer silica gel column in series with the dual-layer carbon reversible tube provides extraction recoveries of 85% or better with less than 200 mL of toluene as illustrated in Figure 3. Recoveries of $^{13}C_{12}$ internal standards ranged from 65% to 95% [n = 3; RSD from 10% to 20%] for the dual layer carbon system. Figure 4 shows how the recoveries compare to previous used method. An overview on recoveries with the Florisil system is shown below.

Challenged with a variety of matrices, the Dioxin Prep System -Florisil Version has demonstrated the ability to clean up sample extracts for dioxin (and PCB) analysis from an array of certified reference materials and inter-calibration samples including sediments WMS-01 and DX-3, fish tissue WMF-01, and intercalibration samples from Orebro University, Sweden (2004 and 2005).

A selection of the results is shown in Figure 5 and Tables 4 & 5*. The Dioxin Prep System showed good recoveries and RSDs for Dioxins and PCBs. Recoveries of $^{13}C_{12}$ internal standards ranged from 80% to 87% [n=4; RSD from 13% to 16%).

For more information or extraction recoveries on additional dioxins, furans, and PCBs, please e-mail the Technical Service department at Europe: EurTechServ@sial.com or USA: techservice@sial.com

Selected Dioxin and PCB concentrations in WMF-01 reference freeze-dried fish tissue and DX-3 certified reference sediments determined using the Supelco Dioxin Prep System-Florisil Version.

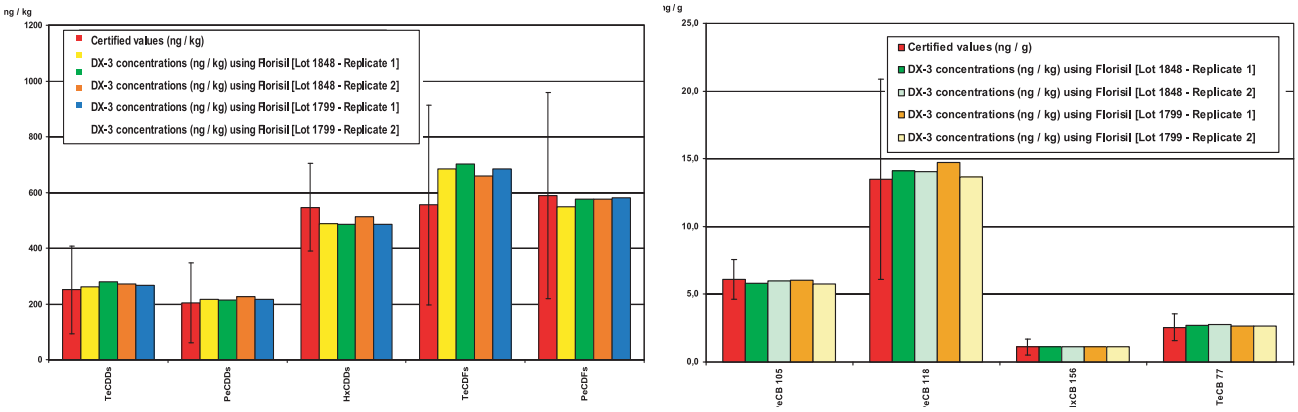
Table 4. Recovery of selected dioxins in reference materials*

Matrix	DX-3 (Sediment)			WMF-01 (Fish Tissue)		
Dioxin/Furan	Certified value (SD) [ng/kg]	Average for n=4 (SD) [ng/kg]	% Recovery	Certified value (SD) [ng/kg]	Average for n=4 (SD) [ng/kg]	% Recovery
2,3,7,8-TeCDD	121 (43)	120 (4.3)	99,2	13.1 (4.4)	12.1 (0.4)	92,4
1,2,3,7,8-PeCDF	35 (17)	36.8 (1.7)	102,2	1.53 (1.4)	0.89 (0.73)	58,2
1,2,3,6,7,8-HxCDD	60 (18)	51 (1.8)	85,7	0.88 (0.4)	0.72 (0.23)	81,8
1,2,3,4,7,8,9-HpCDF	98 (39)	105 (5.9)	107,2	0.4 (0.4)	1.0 (1.9)	250,0
OCDD	3'067 (888)	3'349 (223)	109,2	5.055 (5.1)	2.01 (0.88)	40,1

Table 5. Extraction results for selected dioxins and PCBs with two Florisil lots*

Matrix	DX-3 (Sediment)			WMF-01 (Fish Tissue)		
PCB	Certified value (SD) [ng/kg]	Average for n=4 (SD) [ng/kg]	% Recovery	Certified value (SD) [ng/kg]	Average for n=4 (SD) [ng/kg]	% Recovery
TeCB 77	2.56 (0.99)	2.69 (0.05)	105,1	2'233 (720)	2'293 (22)	102,7
PeCB 105	6.097 (1.467)	5.88 (0.13)	96,4	49'050 (14200)	54'077 (1829)	110,2
PeCB 118	13.48 (7.4)	14.14 (0.43)	104,9	130'100 (32500)	141'535 (1170)	108,8
HxCB 169	0.01 (0.01)	0.018 (0.01)	128,6	76 (30)	78 (2.6)	103,8
HpCB 189	0.185 (0.13)	0.192 (0.01)	103,8	2'016 (611)	2'155 (44)	06,9

Figure 5. Extraction results for selected dioxins and PCBs with two Florisil lots*



*data provided by Corus Research, Development and Technology, Rotherham, UK

Acknowledgements:

We wish to thank Koji Takayanaet al. from Kawaju Techno Service Corporation and Masaaki Maeokaet al. from the Japan Quality Assurance Organization (JQA) for their involvement in the development and evaluation of the Dioxin Prep System applying the Dual-Layer Carbon Reversible Tube.

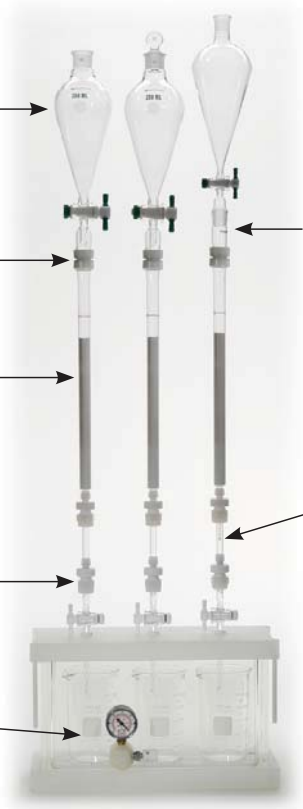
We wish to thank Eric Aries from Corus Research, Development and Technology and Nicholas Ordsmith from Hall Analytical Manchester, UK for their involvement in the development of the Florisil Version of the Dioxin Sample Prep System.

Sample Clean up

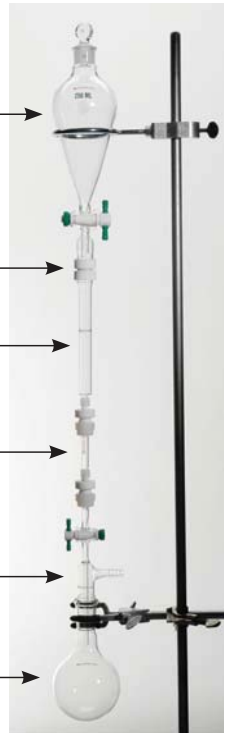
Feature	Advantage	Benefit
Pre packed silica tubes	Reduced additional analysis tasks e.g. silica pre treatment, activation and tube packing	Reduces health & safety implications of small particulate inhalation and exposure to acid and silver treated reagents
Developed in accordance with JIS methods and adapted to EU and EPA methods	Applicable to a wide range of matrices (fat content ~1.5g max per silica column)	Minimise the quantity of lab equipment required to cover a range of samples
Simple and easy to use	Low capital investment and ongoing consumable costs	Excellent option for both start up and established laboratories
Small system footprint	Fume hood space dedicated to laboratory equipment is decreased	Increases the available space for other laboratory and sample preparation tasks
Parallel sample preparation	Sample preparation (including multilayer column preparation) time is reduced by at least two days vs. conventional silica tube method	Increased sample throughput = quicker reporting times
Multiple treated silica layers	Superior clean up of potential interferences	More accurate GC or LC analysis and interpretation

Ordering Information

Vacuum Manifold System



Single Sample Station



Ordering Information

System Components

Dioxin Sample Preparation Kit
Kit includes all glassware and connectors.
Note: Requires, but does not include, Multi-Layer Silica Gel Dioxin Column (28397-U) and Dual-Layer Carbon Reversible Tubes (28399-U) for "Standard Version", and Pre-Activated Florisil (48924-U) and Empty Micro-Column (Reversible Tube) (28309-U) for "Florisil Version"

	1 ea	**28423-U**

Required Consumables for Standard Version

Description	Pkg	Cat. No.
Multi-Layer Silica Gel Dioxin Column O.D. 6.35 mm × length 35 cm	5 ea	**28397-U**
Dual-Layer Carbon Reversible Tube (Micro-Column), O.D. 6.35/10 mm	10 ea	**28399-U**

Required Consumables for Florisil Version

Description	Pkg	Cat. No.
Multi-Layer Silica Gel Dioxin Column O.D. 6.35 mm × length 35 cm	5 ea	**28397-U**
Pre-Activated Florisil®, ampulized, 1 g, particle size 60/100 mesh	10 ea	**48924-U**
Empty Glass Micro-Column (Reversible Tube), O.D. 6.35/10 mm	10 ea	**28309-U**

Replacement Kit Parts

Instruction sheets delivered with the Dioxin Sample Prep System include details and descriptions of the following replacement parts.

Glassware

Description	Pkg	Cat. No.
Dioxin Vacuum Manifold	1 ea	**28403-U**
Vacuum Adapter, I.D. 10 mm	1 ea	**28408-U**
Top Flask with Stopcock, volume 250 mL, neck 24 mm	1 ea	**28449-U**
Empty Dioxin Column, O.D. 6.35 mm × length 35 cm	5 ea	**28404-U**
Syringe Luer Adapter, I.D. 10 mm	3 ea	**28405-U**
Collection Flask/Beaker, flat bottom, volume 300 mL	3 ea	**21269-U**
Long Stem Stopcock, I.D. 10 mm	3 ea	**28425-U**

Connectors

Description.	Pkg	Cat. No
6.35 mm/6.35 mm Union, PTFE	3 ea	**28411-U**
6.35 mm/10 mm Reducing Union, PTFE	3 ea	**28398-U**
10 mm/10 mm Union, PTFE	3 ea	**28412-U**
24 mm/24 mm Polypropylene Viton Connector	6 ea	**28432-U**

Optional components (not included with kit)

Description.	Pkg	Cat. No
Clear Seal Top Flask Adapter, neck 24 mm	3 ea	**21002-U**
Short Stem Stop Cock, I.D. 10 mm	3 ea	**28402-U**
Empty Dioxin Column, I.D. 6.35/10 mm × length 20 cm, to be used with 6.35/ 10mm Reducing Union (Cat. No.28398-U)	5 ea	**28409-U**

Bulk Media (Silica Gels/Sodium Sulfate/Alumina)

The same treated silica gels found in the pre packed multi-layer silica gel columns are available in bulk packages. These materials are useful for customizing your own columns to more efficiently clean very dirty samples, or to prepare shorter columns when samples are relatively clean, e.g. drinking water.

Description.	Pkg	Cat. No
10% $AgNO_3$ Coated Silica Gel	100 g	**21319-U**
44% H_2SO_4 Coated Silica Gel	100 g	**21334-U**
22% H_2SO_4 Coated Silica Gel	100 g	**21341-U**
2% KOH Coated Silica Gel	100 g	**21318-U**
Washed Silica Gel	250 g	**21342-U**
Sodium sulfate, ACS reagent, anhydrous, ≥99.0%, granular	500 g & 2.5 kg	**17876**
Alumina, Basic Type WB-5	250 g & 1 kg	**A1647**

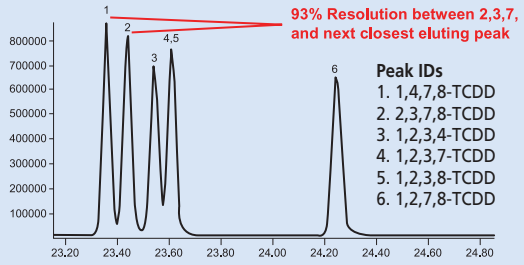
For solvents specifically developed for this analysis see p.15

Related Information

Custom Tubes

If you have a **special multi-layer column** need, or require other packing materials in micro columns, please contact our technical service for support and further assistance.

GC Columns suitable for Dioxin and PCB analysis



column: SP-2331, 60 m x 0.25 mm I.D., 0.20 µm (24104-U)
oven: 170 °C (1 min.), 8 °C/min. to 265 °C
inj.: 250 °C
MSD interface: 265 °C
scan range: SIM
carrier gas: helium, 37 cm/sec constant
injection: 1 µL, splitless (1 min.)
liner: 4 mm I.D., single taper
sample: 1.5 µg/mL TCDD standard in dodecane

SP™-2331

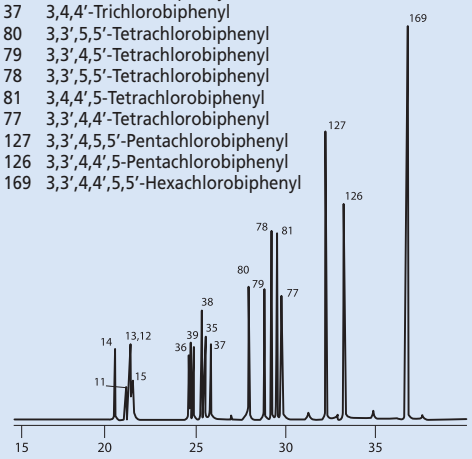
A highly polar cyanosilicone stationary phase, specially tested for analyses of TCDD isomers. The phase is stabilized, providing a maximum temperature slightly higher than nonbonded cyanosilicone phases, such as SP-2330.

Temp. Limits: subambient to 275°C
Phase: Propriatary, stabilised

I.D. (mm)	Length (m)	d_f (µm)	Beta Value	Cat. No.
0.25	30	0.20	313	**24257**
	60	0.20	313	**24104-U**
0.32	60	0.20	400	**24105-U**

PCB IUPAC No.

14	3,5-Dichlorobiphenyl
11	3,3′-Dichlorobiphenyl
13	3,4′-Dichlorobiphenyl
12	3,4-Dichlorobiphenyl
15	4,4′-Dichlorobiphenyl
36	3,3′,5-Trichlorobiphenyl
39	3,4′,5-Trichlorobiphenyl
38	3, 4,5-Trichlorobiphenyl
35	3,3′,4-Trichlorobiphenyl
37	3,4,4′-Trichlorobiphenyl
80	3,3′,5,5′-Tetrachlorobiphenyl
79	3,3′,4,5′-Tetrachlorobiphenyl
78	3,3′,5,5′-Tetrachlorobiphenyl
81	3,4,4′,5-Tetrachlorobiphenyl
77	3,3′,4,4′-Tetrachlorobiphenyl
127	3,3′,4,5,5′-Pentachlorobiphenyl
126	3,3′,4,4′,5-Pentachlorobiphenyl
169	3,3′,4,4′,5,5′-Hexachlorobiphenyl



column: SPB-Octyl, 60 m x 0.25 mm I.D., 0.25 µm (24219-U)
oven: 150 °C (4 min.), 6 °C/min. to 320 °C (16 min.)
inj: 250 °C
det.: ECD, 340 °C
carrier gas: helium, 20 cm/sec at 200 °C
injection: 1 µL, splitless (45 sec)
sample: PCB mixture in isooctane, each analyte at 50-200 ng/mL

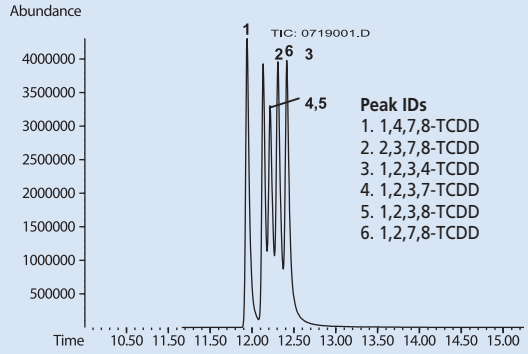
SPB™-Octyl

The polarity of SPB-Octyl approaches that of squalane and is substantially less polar than that of the widely used nonpolar methyl silicone phase. Because this column offers unique selectivity compared to nonpolar and intermediate polarity columns, we recommend SPB-Octyl columns for conformational analyses of PCB-containing samples. Operating Conditions: Chemically compatible with water and other injection solvents. Sensitive to strong inorganic acids and bases. Columns can be rinsed.

Temp. Limits: -60°C to 280°C (isothermal)
McReynolds Nos.: x′y′z′u′s′= 3 14 11 12 11
Phase: bonded; poly (50% n-octyl/50% methylsiloxane)

I.D. (mm)	Length (m)	d_f (µm)	Beta Value	Cat. No.
0.25	30	0.25	250	**24218-U**
	60	0.25	250	**24219-U**
	30	1.00	63	**24232**
	60	1.00	63	**24233-U**
0.53	60	3.00	44	**25398**

TCDD Isomers



column: SLB-5ms, 30 m x 0.25 mm I.D., 0.25 µm (28471-U)
oven: 170 °C (1 min.), 8 °C/min to 270 °C (10 min.)
inj: 250 °C
MSD interface: 270 °C
scan range: SIM, m/z=320, 322, 324
carrier gas: helium, 37 cm/sec., constant
injection: 1 µL, splitless (1 min.)
liner: 4 mm I.D., single taper
sample: TCDD standard, 1500 ppb in n-dodecane

SLB™-5MS

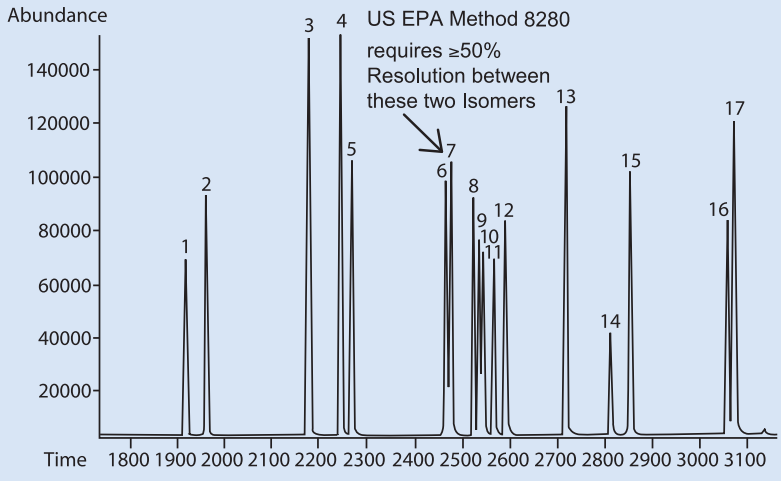
Supelco Low Bleed-5ms columns are designed for GC-MS and GC analysts who require a low bleed, inert, durable, and consistent capillary column for routine and trace analyses. SLB-5ms provides consistently lower bleed, lower detection limits, shorter analysis times, easier mass spectral identification, and less instrument downtime. The low phenyl content provides a boiling point elution order with a slight increase in selectivity, especially for aromatic compounds. The low bleed characteristics, inertness, and durable nature of the SLB™-5ms make it the column of choice for US EPA Methodologies such as environmental semivolatiles by GC-MS and pesticides/PCBs by GC-ECD as well as for dioxins/furans with HRGC/HRMS.

This column meets USP G27 and G36 requirements.
Operating Conditions: Chemically compatible with water and other injection solvents. Sensitive to strong inorganic acids and bases. Columns can be rinsed.

Temp. Limits: 0.10 -0.32 mm I.D.: -60 °C to 340 °C (isothermal)
0.10 -0.32 mm I.D.: -60 °C to 360 °C (programmable)
0.53 mm I.D.: -60 °C to 330 °C (isothermal)
0.53 mm I.D.: -60 °C to 340 °C (programmable)

Phase: bonded and highly crosslinked; silphenylene polymer virtually equivalent in polarity to 5% phenyl polymethylsiloxane

2,3,7,8-substituted PCDDs (Isomers)

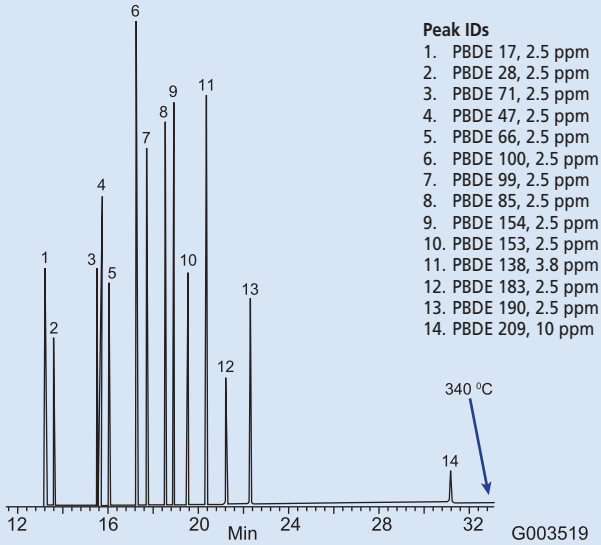


column: SLB-5ms, 30 m x 0.25 mm I.D., 0.25 µm (28471-U)
oven: 150 °C (1 min.), 5 °C/min. to 325 °C (2 min.)
inj.: 250 °C
MSD interface: 325 °C
scan range: SIM
carrier gas: helium, 37 cm/sec constant
injection: 1 µL, splitless (1 min.)
liner: 4 mm I.D., single taper
sample: 17 component 2,3,7,8-substituted dioxin standard, 100-500 ppb in n-nonane

SLB-5ms (cont.)

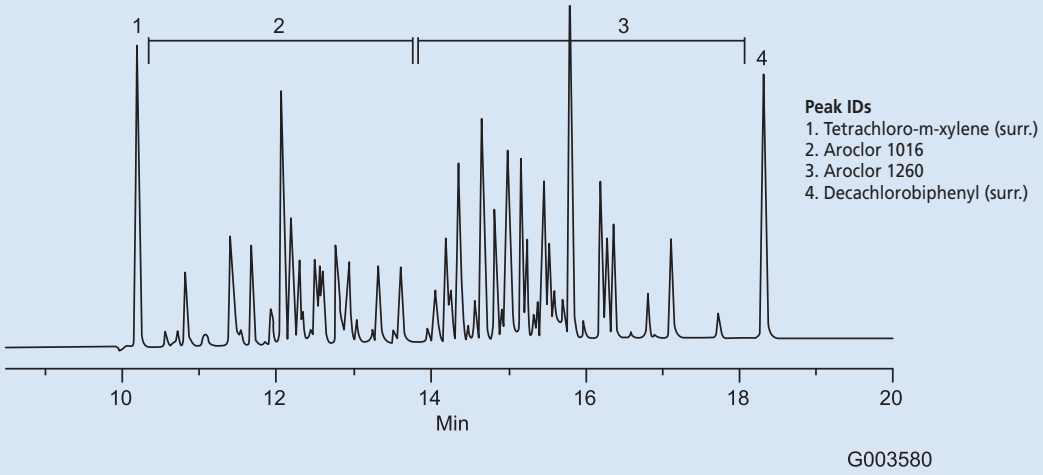
Length (m)	d_f (µm)	Beta	Cat. No.
0.10 mm ID Fused Silica			
10	0.10	250	**28465-U**
15	0.10	250	**28466-U**
0.18 mm ID Fused Silica			
20	0.18	250	**28564-U**
12	0.30	150	**28566-U**
30	0.30	150	**28575-U**
20	0.36	125	**28576-U**
0.20 mm ID Fused Silica			
30	0.20	250	**28513-U**
0.25 mm ID Fused Silica			
30	0.10	625	**28467-U**
15	0.25	250	**28469-U**
30	0.25	250	**28471-U**
60	0.25	250	**28472-U**
15	0.50	125	**28577-U**
30	0.50	125	**28473-U**
60	0.50	125	**28474-U**
30	1.0	63	**28476-U**
0.32 mm ID Fused Silica			
15	0.25	320	**28557-U**
30	0.25	320	**28482-U**
30	0.32	250	**28532-U**
15	0.50	160	**28597-U**
30	0.50	160	**28484-U**
30	1.0	80	**28487-U**
0.53 mm ID Fused Silica			
15	0.50	265	**28542-U**
30	0.50	265	**28541-U**
30	1.0	132	**28559-U**

Polybrominated diphenylethers (PBDEs) by GC-MS



column: SLB-5ms, 30 m x 0.25 mm I.D., 0.25 µm (28471-U)
oven: 125 °C (1 min.), 10 °C/min. to 340 °C (15 min.)
inj.: 300 °C
MSD interface: 340 °C
scan range: SIM
carrier gas: helium, 1.5 mL/min., constant
injection: 1 µL, pulsed splitless (30 psi until 0.2 min.)
liner: 4 mm I.D., single taper
sample: 14 component PBDE standard, 2.5-10 µg/mL in n-nonane

PCB/Aroclor's



column: SLB-5ms, 30 m x 0.25 mm I.D., 0.25 µm (28471-U)
oven: 100 °C (2 min.), 15 °C/min to 330 °C (3 min.)
inj.: 250 °C
det.: ECD, 330 °C
carrier gas: helium, 25 cm/sec, constant
injection: 2 µL, splitless (0.75 min.)
liner: 4 mm I.D., single taper
sample: Aroclor standard mix 1 (46846-U) diluted to 500 ppb / 50 ppb (Aroclors / surrogates) in n-hexane

Air Monitoring

Methods on air sampling of Dioxins/Furans & PCBs (*sampling media reference*)

European Method

EN1948-1 – Stationary source emissions. Determination of the mass concentration of PCDDs/PCDFs and dioxin-like PCBs -Sampling (*Orbo-1000, Orbo-2000, XAD-2 / Supelpak-2*)

US Methods

EPA TO-9A – Polychlorinated, Polybrominated and Brominated Chlorinated Dibenzo-p-Dioxins and Dibenzofurans in Ambient Air (*ORBO-2000*)

EPA 0023A – PCDD / PCDF emissions from stationary sources (*Supelpak-2*)

CARB 428 – PCDD / PCDF and PCBs emissions from stationary sources (*Supelpak-2*)

NIOSH 5503 – PCBs (*ORBO-60*)

EPA TO-10A – Pesticides/PCBs In Ambient Air Using Low Volume PUF Sampling (*ORBO-1000*)

EPA TO-4A – Pesticides / PCBs in Ambient Air Using High Volume PUF Sampling (*ORBO-2000*)

ASTM D4861-05 – Pesticides and PCBs in Air (*ORBO-1000*)

Dioxins /Furans and PCB are also sampled in ambient and indoor air as well as in emissions of industrial sites and waste incinerations. Supelco offers a range of purified sampling media and samplers suitable for this type of analysis. Common adsorbent materials are polyurethane foam (PUF) and Amberlite® XAD-2 (see below for details).

Samplers packed with PUF allow due to the low back pressure created by the foam for high flow rates (up to 5 L/min e.g. ORBO™-1000). For the ORBO-1000 PUF sampler, a filter cartridge assembly with a quartz filter is also available. This enables the simultaneous sampling of both the gas and particulate fractions. The ORBO-1500 and –2500 also have purified XAD-2 as an adsorbent (between two PUF plugs).

ORBO-1000 with installed filter cartridge
(Cat.Nos. 20557 & 21031)

Ordering Information

Description	Dimension O.D. x L	Pkg	Cat.No.
PUF sampler (product selection*)			
ORBO-1000	Assembled Cartridge - 22 mm x 7.8 cm PUF plug in glass	3 ea	20557
ORBO-1000	22 mm x 7.8 cm precleaned PUF plugs	3 ea	20600-U
ORBO-1500	Assembled Cartridge - 22 mm x 30 mm PUF, 1.5 g XAD-2, 22 mm x 30 mm PUF in glass holder	3 ea	22133-U
Filter cartridge for ORBO-1000		1 ea	21031
Replacement Quartz filter for ORBO-1000		10 ea	21038
ORBO-2000	Assembled Cartridge - 6 cm x 7.6 cm PUF in Glassholder	1 ea	20037
ORBO-2000	6 cm x 7.6 cm precleaned PUF replacement plugs	1 ea	20038
ORBO-2500	Assembled Cartridge - 6 cm x 5 cm PUF, 10 g XAD-2, 6 cm x 5 cm PUF	3 ea	21235-U
Other air sampling tubes & filter*			
ORBO-60 6 mm x 70 mm, Florisil (30/45) 100/50 mg		50 ea	20351
Boro silicate Glass Fiber Filter (Binder free) 13 mm OD, 1 µm pores		500 ea	23376
Empty Filter cassette 13 mm, 2-piece, with washer (Swinney Filter Holder)		5 ea	23367

* For more details on the above products or on other air monitoring media and equipment please refer to the Supelco catalogue or the Sigma-Aldrich web site.

Resins

Amberlite® XAD-2 & purified versions

Amberlite® XAD-2 is a polyaromatic (styrene-divinylbenzene) adsorbent resin commonly used for adsorbing hydrophobic compounds up to MW 20,000: phenols, organic removal, surfactants, aroma compounds, antibiotic recovery. It is one of the most used adsorbents for dioxin/furan sampling. The nonionic macroreticular resin that adsorbs and releases analytes through hydrophobic and polar interactions is usually used under isocratic conditions.

For cleaned US EPA versions, see Supelpak-2, Supelpak-2B and Supelpak-2SV below.

Specifications of Amberlite XAD-2:

- surface area ~300 m2/g
- density 1.02 g/mL, 25 °C (true wet)
- density 1.08 g/mL, 25 °C (skeletal)
- particle size 20-60 mesh
- pore volume ~0.65 mL/g
- mean pore size 90 Å
- max. temp. 200 °C

Ordering Information

Description	Pkg	Cat. No.
Amberlite XAD-2	100 g	**20275**
	500 g	**10357**
	5 kg	**SU853005**
	10 kg	**52672-U**
	25 kg	**3025-U**
Purified Amberlite XAD-2		
Supelpak-2	100 g	**20279**
	1 kg	**21130-U**
Supelpak-2B	100 g	**13670**
Supelpak-2SV	100 g	**13673-U**
	250 g	**13682-U**
	1 kg	**13674-U**

For more details on the above products or other air monitoring media and equipment please refer to the Supelco catalogue or the Sigma-Aldrich web site under sample preparation.

Purified Amberlite® XAD-2

The purified versions of Amberlite XAD-2 from Supelco are the Supelpak™-2 Materials. These have been treated in reference to official methods or special requirements:

Supelpak™-2 – Purified Amberlite® XAD-2 that has been cleaned to meet and exceed US EPA-recommended criteria for purity, as outlined in Level I Environmental Assessment Procedures Manual. It is the best resin to use for standard air sampling methods requiring resin tested for background TCO (total chromatographic organics) level. Packaged in glass containers.

Supelpak™-2B – It has been cleaned to meet and exceed US EPA requirements for determining PCBs in water according to the Great Lakes National Program Office (GLNPO). Packaged in glass containers.

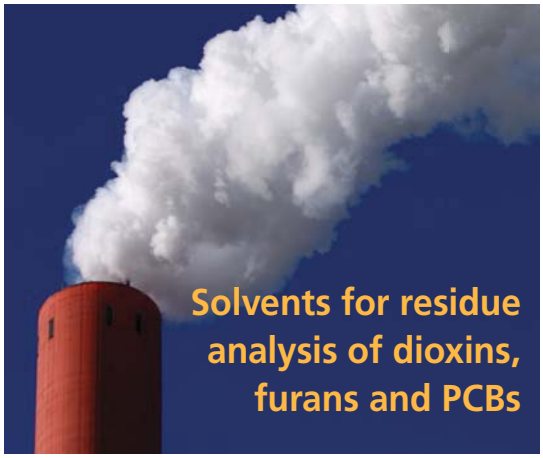
Supelpak™-2SV – Purified Amberlite® XAD-2 that has been specially cleaned and tested for optimal performance in the capturing and extraction of semivolatile organics. Packaged in glass containers.

Related Information

Custom capabilities

Often analysts are confronted with analytical needs that are deviating from what is commercially available. Therefore Sigma-Aldrich offers the possibility to custom manufacture e.g. multi-layer silica tubes, standards and special treated bulk packing and adsorbent materials like alumina, silica or certain resins like Amberlite® XAD-2. If you have a special need please contact your local Sigma-Aldrich office/Technical Service for more details on the custom capabilities for these products.

Solvents & Standards



ENVISOLV® Solvents for Dibenzo-p-dioxin/ -furan & PCB analysis

To achieve a good and reliable sample clean up results largely depends also on the used solvents that should not contribute to significant increase in the background of an analysis.

ENVISOLV® solvents are specified especially for the use in dibenzodioxin/furan analysis. They are GC-MS tested and contain less than 5 pg/l (5 ppq) of the 17 relevant dibenzodioxins and dibenzofurans listed in Table 6. Further specifications of this solvents are listed in Table 7.

Ordering Information

Description	Pkg	Cat.No.
ENVISOLV		
Acetone	2.5L & 7L	**34410**
Dichloromethane	2.5L & 7L	**34411**
Hexane	2.5L & 7L	**34412**
Toluene	2.5L & 7L	**34413**

For the availability of the pack sizes please check with your local Sigma-Aldrich office or our website **sigma-aldrich.com**

Table 6. Tested dioxins & furans

Dibenzodioxins
2,3,7,8-tetra-CDD
1,2,3,7,8-penta-CDD
1,2,3,4,7,8-hexa-CDD
1,2,3,6,7,8-hexa-CDD
1,2,3,7,8,9-hexa-CDD
1,2,3,4,6,7,8-hepta-CDD
Octa-CDD

Dibenzofurans
2,3,7,8-tetra-CDF
2,3,4,7,8-penta-CDF
1,2,3,7,8/1,2,3,4,8-penta-CDF
1,2,3,4,7,8/1,2,3,4,7,9-hexa-CDF
1,2,3,6,7,8-hexa-CDF
1,2,3,7,8,9-hexa-CDF
2,3,4,6,7,8-hexa-CDF
1,2,3,4,6,7,8-hepta-CDF
1,2,3,4,7,8,9-hepta-CDF
Octa-CDF

Table 7. Specifications of the ENVISOLV product range

	Acetone	Dichloromethane	Hexane	Toluene
Assay (GC)	min. 99.8 %	min. 99.8 %	min. 95 %	min. 99.7 %
Non-volatile matter	max. 5ppm	max. 5ppm	max. 5ppm	max. 5ppm
Water content (Karl Fischer)	max. 0.2 %	max. 0.02 %	max. 0.01 %	max. 0.02 %
Suitability for GC-MS of dioxins, furanes, PCBs	complying	complying	complying	complying

Sigma-Adrich offers a wide range of standards, reference materials and calibration solutions. Please refer to our standards catalogue & CD, or visit us on our website: www.sigma-aldrich.com/standards

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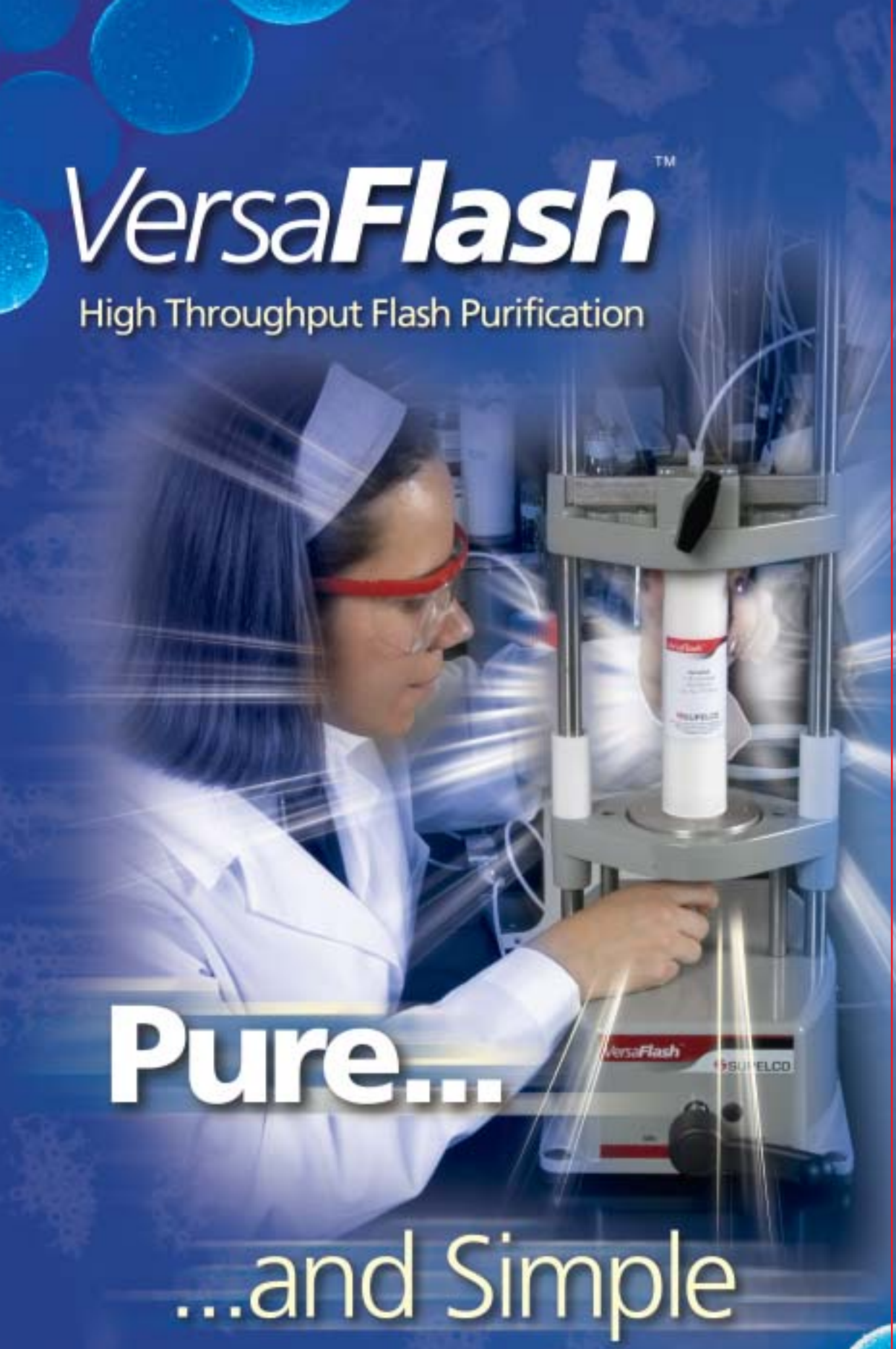
Date: July 2007;
SAMS Code: JXB

Printed on Recycled Paper —
A Worldwide Operation with a Global Commitment

VersaFlash™

High Throughput Flash Purification



Pure...

...and Simple

- VersaFlash Station
- VersaPak™ Cartridges
- VersaVac™ Sample Loading Station
- Pump
- Kits

sigma-aldrich.com/supelco

SUPELCO

Save Time

- **Fast and easy cartridge change-over –** simply turn and pull the handle on the VersaFlash station to exchange cartridges in less than 10 seconds.
- **Fast elution of desired substances –** strongly retained compounds can be eluted quickly through the unique Rev-Elution capability of the VersaFlash system.
- **Speedy scale-up to larger sample loads –** the VersaFlash station accepts VersaPak™ cartridges up to 80mm diameter. There is no need for time consuming loading and unloading of cartridge compression barrels.
- **One-step, bimodal purifications –** the exclusive capability of stacking VersaPak cartridges permits on-line, rapid, clean-up of complex synthetic and natural product mixtures prior to the final purification of the desired substance(s).
- **Rapid loading and partial separation of multiple sample mixtures –** the VersaVac™ Sample Loading Station makes it possible to conduct as many as six parallel separations prior to elution on the VersaFlash station.
- **Instant mobile phase change-over –** a switching valve provides a fast and simple means of changing mobile phase composition. As many as six solvent strengths could be configured for step gradients.

SUPELCO

Save Money

- **Pre-compressed cartridges eliminate the expense of additional compression barrels –** the patent pending pre-compression technology of all VersaPak cartridges eliminates the added expense of purchasing separate compression barrels for different cartridge dimensions.
- **All VersaPak cartridges fit on the VersaFlash station –** the entire range of VersaFlash cartridges from discovery to development scale can be used with a single VersaFlash station. No need to purchase separate systems for different sample loads.
- **Rev-Elution saves solvent costs –** the unique capability of reversing the flow through VersaPak cartridges reduces the volume of solvent needed to elute highly retained substances. This can amount to significant savings especially during the use of 80mm ID cartridges.
- **Direct sample loading eliminates the cost of cartridge inserts –** the patented end-fittings permit one-step sample loading directly onto the cartridge without the use of special pre-loaded inserts or on-line valving devices.

Improve Performance

- **Spherical particles result in low band spreading –** the spherical particles insure more uniform packing of the material and eliminate fines created when irregular shaped particles fracture under pressure leading to voids and channeling in the bed.
- **Flexible flow direction –** the unique VersaFlash system design allows you the flexibility to pump the mobile phase solvent in either direction through the station. Choose between upward flow to insure proper wetting of the cartridge bed and complete expulsion of air or downward flow similar to other systems.
- **Higher sample loading capacity** – the spherical silica used in the VersaPak cartridges packs more densely and uniformly, resulting in higher sample loading capacity and cleaner separations compared to cartridges with irregular silica.
- **Leak-resistant system design saves samples, solvents and cartridges –** the patent pending end-fitting design eliminates leakage due to broken or under-tightened fittings.
- **Unobstructed cartridges allow monitoring of the separation –** the unique cartridge design eliminates the need for compression barrels allowing for easy viewing of the solvent front and tinted bands during separation.

VersaFlash Station

The most versatile flash purification system.

The new, patent pending Supelco VersaFlash High Throughput Flash Purification (HTFP) system is the first system to offer all of the capabilities required for flash separations and the versatility to perform unique purification tasks that conventional flash systems do not allow. These capabilities permit researchers to expand beyond the limitations of contemporary flash chromatography, saving time and money while improving their separations.

Built-In Versatility

The unique design of the VersaFlash station makes it possible to:

- Scale up from 40mm ID to 80mm ID cartridges on the same system.
- Extend cartridge length by stacking two cartridges together.
- Achieve bimodal separations by stacking different phase chemistries.
- Elute strongly retained substances quickly by using the Rev-Elution technique.

Versatility Plus

The VersaFlash station can easily be connected to existing flash system hardware with either a pressurized reservoir or a pump. The outlet can also be connected to a fraction collector or optional fractioning valve.

VersaFlash Makes Cartridge Change-Out Fast and Easy

The unique, patent pending VersaPak pre-compression design eliminates the need for compression barrels making cartridge change-out fast and easy.

Installing, removing, and changing out VersaPak cartridges in the VersaFlash station is fast and easy. With the handle in the ***idle*** position and pulled out, the bottom platen is lowered, allowing ample room to move cartridges in and out. Once you have installed a cartridge, simply push the handle in, raising the lower platen, securing the cartridge in place. Then simply turn the handle toward the ***operate*** position to fully engage the cartridge and to create a leak-proof seal.

Figure 1. Fast and Easy Cartridge Change-Out

You can see the previously used cartridge sealed into the VersaFlash station with the handle in the *operate* position.

Turn the handle to the *idle* position and pull it out to remove the cartridge.

Insert a new cartridge, push the handle in and turn it toward the *operate* position to seal the new cartridge.

VersaFlash Efficiently Handles a Wide Range of Sample Sizes

Most flash chromatography systems typically limit users to a narrow sample range requiring additional hardware purchase and laborious change-out in order to work with larger sample sizes. The robust VersaPak cartridge design eliminates the need for a compression barrel making cartridge change-out fast and easy. The VersaFlash station offers you the versatility to work with discovery scale cartridges (40 x 75mm) and only requires a simple adjustment of the upper platen to work with development scale cartridges (80 x 300mm). In less than one minute you can change from the smallest cartridge to the largest cartridge giving you the flexibility you need to handle a wide range of sample sizes.

Common Sense Technology

The VersaFlash HTFP system incorporates several simple scientific principles allowing for the best separations possible, the highest cartridge capacity, and the most cost-effectiveness.

The VersaFlash employs:

- Spherical particles to optimize cartridge bed uniformity and prevent bed shifting due to irregular particle abrasion and fines.
- The use of vacuum to permit loading and partial separation in multiple cartridges.
- Secure end fittings with no threaded luer connections to leak or fracture.
- Precompression to eliminate the need for a compressed gas source, expensive and clumsy compression barrels, and cartridges stuck in compression barrels.
- Capped, flat-ended cartridges to allow for cartridge stacking and Rev-Elution.

The VersaFlash HTFP system was designed for total versatility. The VersaFlash station can even be installed to operate with existing flash apparatus. It can be used with a pump or a pressurized reservoir. Simply replace the cartridge apparatus with a VersaFlash station to gain all of the benefits of the VersaFlash system. A detector and fraction collector of choice also may be connected to the VersaFlash stand to further expand your capabilities.

Only Supelco offers the versatility of a fully integrated HTFP system and the cartridge technology to add versatility to an existing flash system.

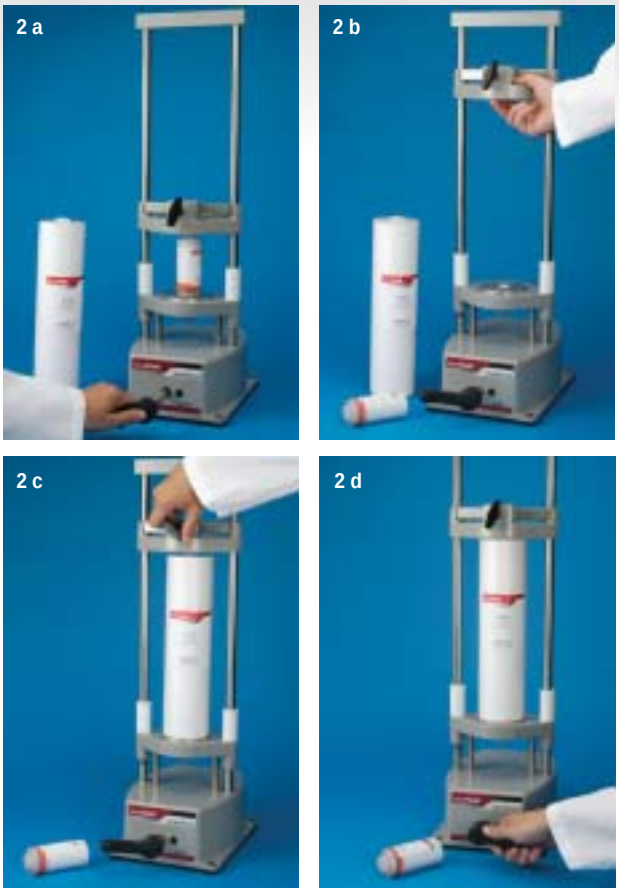
Figure 2. VersaFlash is Scalable Across the Entire Range of Cartridge Sizes Using the Exact Same Hardware

2a. To switch to the larger cartridge, turn the handle to the ***idle*** position and pull it out lowering the bottom sealing platen.

2b. Once the cartridge is removed, push the handle in and loosen the securing knob on the upper platen to raise it to the proper height to insert the 300mm cartridge.

2c. With the upper platen resting on the cartridge, tighten the securing knob.

2d. Turn the handle toward the ***operate*** position sealing it in the VersaFlash station.



VersaFlash Station Specifications

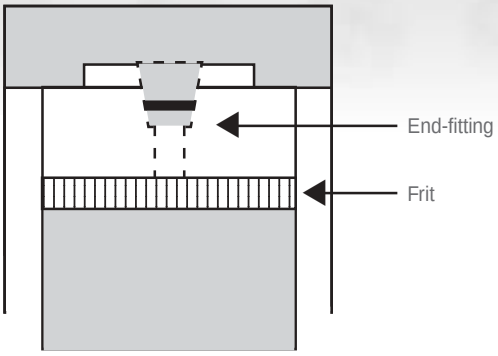
Height:	70cm (27.5 in.)
Length:	28cm (11 in.)
Width:	21cm (8 in.)
Weight:	12kg (27 lbs)

VersaPak Cartridges

A key feature of the VersaPak cartridges is the patent pending end-fitting. The symmetric end-fitting design allows for fast and easy cartridge change-out, cartridge stacking, and easy sample loading on the VersaVac vacuum manifold. There is no need for a compression apparatus or external compressed gas supply since the cartridges are pre-compressed and sealed by the end-fittings. VersaPak cartridges are tightly held in place during pressurization by the leak-resistant VersaFlash station.

Leak-Resistant Cartridge Design

The VersaPak cartridges are carefully packed and pre-compressed for optimum performance. The end-fittings are designed to fit tightly into the VersaFlash station so that leakage will not occur and the packing will stay tightly compressed. When set in the ***operate*** position, the VersaPak cartridge seats securely and withstands pressures up to 80psig/5.5 bar (40mm).



Cartridge Stacking

Another benefit of the unique design of the VersaPak cartridge is that it allows for extending cartridge length through stacking one cartridge on top of another. Two 75mm cartridges quickly become a 150mm cartridge using the cartridge stacking connector. Even cartridges of different packing materials may be stacked for complex multi-modal separations.

Figures 3a - 3c show separation of a multi-dye mixture using bimodal stacked cartridges (silica and C18).

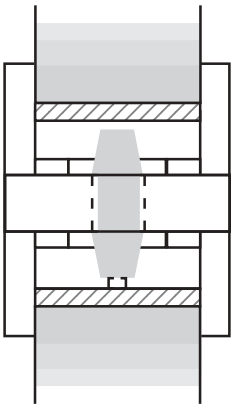


Figure 3. Efficient Separation of a Multi-Dye Mixture Using BiModal Stacked Cartridges and Rev-Elution (Silica and C18)

3a. The dye mixture (Red 40, Blue 1 and Yellow 5) is loaded and begins to separate on the cartridges.

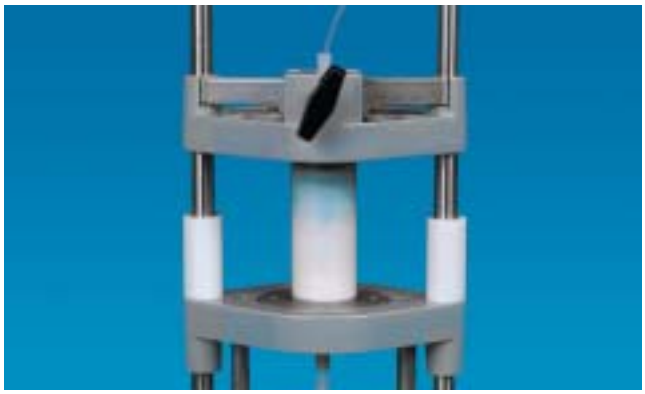
3b. The yellow 5 dye elutes first with water.

3c. The red 40 dye then elutes with water.

Rev-Elution

When desired compounds are fully retained on the cartridge, they must be eluted using a stronger mobile phase. Normally this spreads the band as it passes through the entire media bed. Also, if there are traces of sample material remaining on the cartridge, contamination of the retained component is likely to occur. Finally, excess solvent is required to move the band entirely through the cartridge. These problems are solved by Rev-Elution, or the technique of reversing the cartridge 180 degrees (Figures 3d - 3e). Consequently the retained substance can be quickly and cleanly eluted using a slightly stronger mobile phase without the need to travel through the entire cartridge bed.

3d. The C18 cartridge with the blue 1 dye at the front of the cartridge is reversed to put the band at the end of the cartridge.



3e. The blue 1 dye band elutes quickly using 100% methanol without travelling the length of the cartridge, resulting in no band spreading and less chance of contamination from residual sample components.

Sample Loading

The VersaPak cartridge provides you with a variety of ways to load samples. There are at least five ways to load a sample onto a VersaPak cartridge.

- Direct sample injection onto the cartridge (off-line).
- Vacuum aspiration onto the cartridge using the VersaVac manifold (off-line).
- Using a solid sample cartridge prior to the primary cartridge (on-line).
- Using a valve and loop injector for repetitive volume sample loading (on-line).
- Through the pump loading for large volume dilute samples (on-line).

Direct Sample Application

VersaPak cartridges can be loaded with sample material prior to insertion in the VersaFlash station. There are no valves or tubing to carry over residual material or clog with precipitated sample. Even viscous fluids and substances of low solubility with the mobile phase can be loaded directly onto the cartridge without cartridge inserts or in-line sample devices (Figure 4). This makes it possible to load the samples in a different location from the VersaFlash station where the purification will be performed. Cartridges can even be loaded hours before the separation takes place.

Figure 4. Direct Sample Loading Onto Cartridge

Multiple Sample Loading

The VersaVac loading station permits as many as six cartridges to be simultaneously loaded with sample material (Figure 5). Simply place the cartridges onto the VersaVac, pour the sample material into the funnel, and turn on the vacuum. Samples are immediately applied to the head of the cartridge and are ready for purification. The VersaVac also allows for partial separations to take place (Figure 6). By following the sample with a solvent, the sample components will begin to separate in the VersaPak cartridge. This time-saving method can be done with as many as six different samples simultaneously.

Figure 5. Loading Multiple VersaPak Cartridges

Figure 6. Partial Separation

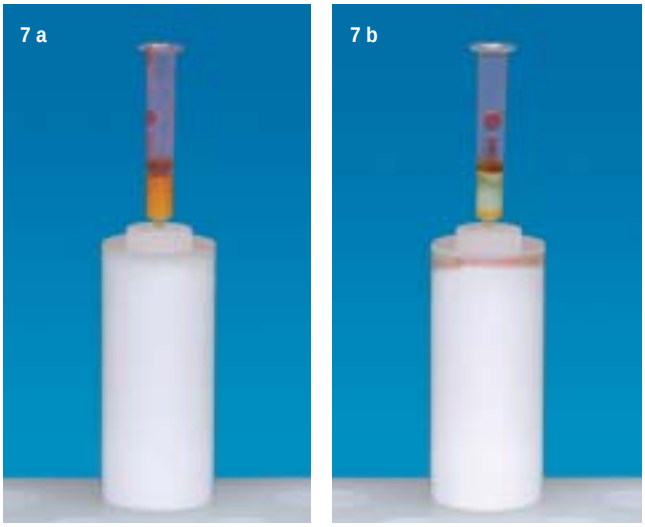
One-Step Pre-Purification

Samples often contain impurities that will not be retained by the packing media in the VersaPak. Impurities such as dissolved salts, colored material, and reaction by-products can be removed easily before the sample reaches the VersaPak. Supelco SPE tubes, in tandem with a VersaPak cartridge, will retain the contaminants while allowing the remaining components to be loaded onto the VersaPak cartridge. The VersaVac serves as a convenient device to pull the mixture through the SPE / VersaPak combo. As many as six samples can be pre-purified and loaded simultaneously.

Figure 7. Separation of a Chlorophyll/Carotenoid Mixture Using One-Step Pre-Purification

7a. Load sample on C18 cartridge using silica SPE tube for pre-purification

7b. Carotenoids were eluted into the VersaPak with methanol/dichloromethane, chlorophyll sticks on the silica (green band)



Solid Sample Cartridges

A solid sample cartridge is provided for those low solubility samples that require pre-adsorption prior to loading onto the VersaPak cartridge.

For samples that require precise volumetric measurement, an optional valve and loop injector is available, allowing a finite sample volume to be loaded on each injection.

VersaVac Sample Loading Station

The VersaVac sample loading station allows you to pre-load and partially separate multiple samples off-line. Cartridges can be placed on top of the vacuum manifold, a vacuum applied and samples added to the top of the cartridge, thereby being drawn by vacuum onto the top of the cartridge. The VersaVac gives you the flexibility to pre-load from one to six cartridges off-line while other samples are being purified, allowing you to work more efficiently.

The kit includes the VersaVac manifold, with vacuum gauge and female Luer plugs.

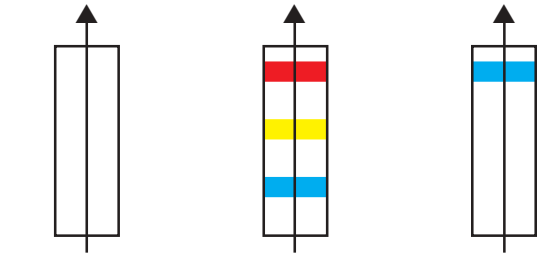
Mobile Phase Management

Supelco offers a wide range of accessories for mobile phase management for high throughput flash purification. From Teflon tubing, solvent reservoirs, and inlet filters to solvent switching valves for creating step gradients. These accessories can be combined in numerous ways to customize a solvent delivery system to meet your needs.

Gradient Formation

Complex or poorly resolved samples may require gradient elution in which the solvent concentration is increased during sample purification.

The VersaFlash can be set up for both step and continuous gradients. An optional six-position solvent select valve permits as many as six different solvent compositions to be selected during a separation. If there is a need to precondition a cartridge with a weak solvent, the solvent can be pumped into the cartridge before switching over to the primary solvent. A third solvent might be used to flush the cartridge or to elute a component following Rev-Elution as shown below.



Although continuous gradients are seldom used in HTFP, there may be times when they are needed. On such occasions, a simple gradient configuration is available. This device consists of two connected flasks and a stirring mechanism. As the weak solvent is pumped into the cartridge, the stronger solvent is drawn into the flask containing the weak solvent.

Step Gradient Accessories

A large number of accessories are available to create step or continuous gradients for use with the VersaFlash system. By combining a solvent switching valve, tubing, and multiple solvent bottles, step gradients of up to six different solvents can be created to enhance the purification parameters.

Mobile Phase Reservoir - 55060-U

Valve - 58829

Filter - 58267

VersaFlash Pump

Many flash chromatography systems utilize a pressurized stainless steel solvent reservoir to contain the solvent and force it through the flash cartridges. The VersaFlash system uses a pump, which eliminates the need for a stainless steel reservoir, thereby eliminating the following limitations:

- The level of solvent cannot be seen, often times causing incomplete separations and unknown solvent flush volumes.
- The reservoir is difficult to empty and refill any time solvent change is required.
- Solvent volume is limited by the tank capacity.
- An external gas supply is required to pressurize the tank.

Advantages of the VersaFlash piston pump include:

- Variable speed for flow rate adjustment.
- Compact pump design conserves valuable fume hood space.
- Unlimited solvent volume for uninterrupted flow.
- No external gas source required.
- Solvent changeover is fast and easy.
- Digital flow adjustment provides an easy means of repeating flow settings.

Specifications

Flow Rate:	5 to170mL/min
Pressure:	0 to 7.8 bar (100psi)
Fluid Contact Parts:	Tefzel, Ceramic
Fluid Viscosity Range:	Up to 2000cps without feed pressure
Fluid Connections:	1/4”- 28 HPLC fittings 1/4” ID compression
Pump Type:	Piston
Motor Type:	3400 rpm DC, Optically Encoded, Servo Controlled

Controls

Manual Input:	Buttons on front panel
Analog Input:	4-20 mA, or 0-5 VDC
Analog Output:	Pulsed output proportional to motor speed
Operating Temperature Range:	4° to 40°C
Operating Humidity Range:	Up to 100% humidity
Electrical Input:	110/220 VAC, 50/60 Hz, 75 Watts, Double fused
Physical Size:	5.75” W x 6.25” H x 11” D 14.6cm W x 15.9cm H x 28cm D
Weight:	5 kg (11 lb.)
Agency Certifications:	CE
Supplied Accessories:	Power Cord, Teflon tubing and fittings
Warranty:	Limited Warranty – See operation manual or ask your dealer for a copy of the warranty. Misuse, misapplication, neglect and abuse are specifically excluded. No warranty on the pump head.

Kits

VersaFlash Starter Kit

The starter kit provides you with everything that you will need to get started using the broad capabilities of the VersaFlash system for purification. From flushing solvents out of the system using the solvent flush adapter to stacking multiple cartridges using the cartridge stacking connector. The starter kit allows you to utilize the broad capabilities of the VersaFlash system.

The kit includes a 40 x 40mm cartridge stacking connector, a solvent flush connector, 1/8 in. tubing (10 ft.) and solid sample cartridges (3 large and 3 small)

Cartridge Stacking Assemblies

The cartridge stacking assemblies allow you to connect two cartridges in series to expand the sorbent capacity and/or to mix different cartridge phase chemistries to conduct bimodal separations. The connector consists of a stacking connector to connect the fluid flow paths of the two cartridges as well as a sleeve to hold the two cartridges in alignment during operation. Cartridge stacking assemblies are available in three sizes: 40 x 40mm, 40 x 80mm, and 80 x 80mm; to stack in different diameter cartridges.

Solvent Flush Connector

The solvent flush connector is used to flush solvents out of the VersaFlash station or when changing from one solvent to another without a VersaPak cartridge in the station.

Solid Sample Cartridge Kit

The solid sample cartridge kit allows you to adsorb samples onto silica or other packing materials and pack the dried sample into a solid sample cartridge and link it to the separation cartridge, allowing for direct desorption by the mobile phase. Samples that have low solubility in the mobile phase can be transferred to the head of the cartridge with no tubing or fittings to restrict or clog due to precipitation.

The kit includes 6 large (40 x 75mm) solid sample cartridges, 6 small (40 x 37.5mm) solid sample cartridges, 12 end-fittings, and 12 frits.

VersaPak Cartridge Characteristics and Loading Guidelines

- VersaPak cartridges are packed with 45 -75µm, spherical, silica based porous (70Å) particles.
- They are pre-packed and compressed for optimum loading capacity, flow performance, and mass transfer.
- Optimum loading per cartridge depends on separation conditions, length and diameter, flow rate, and temperature.

The table below is useful to determine the general operating conditions and loading limits.

Cartridge	Packing Weight (g)	Liquid Volume (mL)	Maximum Pressure (psi)	Hydrophobicity	Maximum Loading Capacity (g)
Silica, 40 x 75mm	40	60	80	Hydrophilic	4
Silica, 40 x 150mm	96	120	80	Hydrophilic	9.6
Silica, 80 x 150mm	340	490	40	Hydrophilic	34
Silica, 80 x 300mm	700	980	40	Hydrophilic	70
C18, 40 x 75mm	70	30	80	Hydrophobic	7
C18, 40 x 150mm	140	60	80	Hydrophobic	14
C18, 80 x 150mm	515	240	40	Hydrophobic	51
C18, 80 x 300mm	1050	480	40	Hydrophobic	105

Elution Volume Determination

Conversion of TLC conditions to VersaFlash operating conditions can be calculated roughly by reciprocating the rf value of the component of interest. This value converts to the number of liquid column (cartridge) volumes required for elution.

For tightly and fully retained substances, perform Rev-Elution, possibly requiring a stronger mobile phase for complete elution.

Step or continuous gradient application generally will reduce solvent volume.

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Ordering Information

SUPELCO

Description	Qty.	Cat. No.
Systems		
System I (Includes station assembly, pump, starter kit, sample loading kit)	1	**97730-U**
System II (Includes station assembly, starter kit, sample loading kit)	1	**97731-U**
Station Assembly		
Station Assembly (Includes Teflon tubing, flangeless fittings)	1	**97732-U**
Replacement Teflon Tubing, 10' x 1/8" OD x 1.5mm ID	1	**58703**
Replacement Teflon Tubing, 50' x 1/8" OD x 1.5mm ID	1	**58699**
Replacement Flangeless Fittings	5	**58686**
Replacement PEEK Seal Assemblies	2	**97748-U**
Replacement O-rings for PEEK Seal	6	**97745-U**
Pump		
Assembled Pump, 120V	1	**97734-U**
Assembled Pump, 220V	1	**97737-U**
VersaPak Silica Cartridges		
Silica Cartridge, 40mm x 75mm	12	**97704-U**
Silica Cartridge, 40mm x 75mm	96	**97705-U**
Silica Cartridge, 40mm x 150mm	6	**97706-U**
Silica Cartridge, 40mm x 150mm	48	**97707-U**
Silica Cartridge, 80mm x 150mm	2	**97708-U**
Silica Cartridge, 80mm x 150mm	12	**97709-U**
Silica Cartridge, 80mm x 300mm	1	**97710-U**
Silica Cartridge, 80mm x 300mm	6	**97711-U**
VersaPak C18 Cartridges		
C18 Cartridge, 40mm x 75mm	2	**97700-U**
C18 Cartridge, 40mm x 150mm	1	**97701-U**
C18 Cartridge, 80mm x 150mm	1	**97702-U**
C18 Cartridge, 80mm x 300mm	1	**97703-U**
Starter Kit		
Starter Kit (Includes Teflon tubing, flangeless fittings, solvent flush connector, 3 small solid sample cartridges with frits and end-fittings, 3 large solid sample cartridges with frits and end-fittings, 40mm to 40mm cartridge stacking assembly)	1	**97739-U**
Replacement Solvent Flush Connector	1	**97743-U**
Sample Loading Kit		
VersaVac Sample Loading Kit (Includes vacuum manifold, vacuum gauge / bleed valve, vacuum hose, threaded ¼-28 to male Luer adapters, female Luer plugs)	1	**97750-U**
Replacement Vacuum Gauge/Bleed Valve	1	**57161-U**
Replacement Vacuum Hose, 10' x 1" OD x 1/4" ID	1	**Z255998**
Replacement Threaded ¼-28 to Male	6	**97744-U**
Luer Adapters Replacement Female Luer Plugs	12	**57098**
Solid Sample Cartridges & Bulk Media		
Solid Sample Cartridge Kit (Includes 6 small cartridges, 6 large cartridges, frits, end-fittings)	1	**97738-U**
Small Solid Sample Cartridges, 40mm x 37.5mm*	12	**97746-U**
Large Solid Sample Cartridges, 40mm x 75mm*	12	**97747-U**
Celite 545 AW	454g	**20199-U**
Flash Silica, dried	100g	**97728-U**
Flash Silica, dried	1Kg	**97729-U**
Flash C18 Silica	100g	**97727-U**
*Includes frits and end-fittings.		
Cartridge Stacking Assemblies		
Cartridge Stacking Assembly, 40mm to 40mm**	1	**97740-U**
Cartridge Stacking Assembly, 40mm to 80mm**	1	**97741-U**
Cartridge Stacking Assembly, 80mm to 80mm**	1	**97742-U**
Replacement O-rings for Cartridge Connectors	6	**97745-U**
**Includes 1 connector and 1 sleeve.		

Description	Qty.	Cat. No.
Sample Loading Kit Accessories		
Vacuum Pump Trap Kit (Includes flask, stopper and rigid tubing, vacuum hose)	1	**57120-U**
Male Luer Couplers (For stacking cartridges)	20	**25064-U**
Disposable Male Luer Syringes, 3mL (For loading samples)	100	**Z192112**
Disposable Male Luer Syringes, 5mL	100	**Z192139**
Disposable Male Luer Syringes, 10mL	100	**Z192031**
Disposable Male Luer Syringes, 20mL	40	**Z192228**
Empty SPE Tubes, 1mL, (For use as sample/solvent reservoirs)	108	**57240-U**
Empty SPE Tube, 3mL	54	**57241**
Empty SPE Tube, 6mL	30	**57242**
Empty SPE Tube, 12mL	20	**57179**
Empty SPE Tube, 20mL	12	**57021**
Empty SPE Tube, 60mL	12	**57022**
On-Line Sample Loading Accessories		
Rheodyne Model 5020 Low Pressure Sample Injection Valve (For repetitive loading of cartridges)	1	**58814**
Mounting Bracket	1	**57460-U**
Sample Loop, 0.1mL	1	**57680-U**
Sample Loop, 1mL	1	**57683**
Sample Loop, 5mL	1	**57684**
Sample Loop, 10mL	1	**57685**
Disposable Male Luer Syringe, 20mL (For filling sample loops)	40	**Z192228**
Mobile Phase Accessories		
Rheodyne Model 5011 Low Pressure Mobile Phase Selection Valve (For switching between mobile phases)	1	**58829**
Mounting Bracket	1	**57460-U**
Mobile Phase Reservoir System, 1L (Includes 1L bottle, Teflon tubing, 2µm filter/sparger)	1	**55060-U**
Mobile Phase Reservoir System, 2L (Includes 2L bottle, Teflon tubing, 2µm filter/sparger)	1	**55061**
2µm Stainless Steel Inlet Filter	1	**58267**
10µm Slip-On Inlet Filter	1	**59277**
Solvents		
Acetonitrile 99.5+% ACS Reagent	4 x 4L	**43,755-7**
Chloroform 99.8% ACS Reagent	4 x 4L	**47,247-6**
Dichloromethane 99.9% ACS HPLC	4 x 4L	**27,056-3**
Methanol 99.8% ACS Reagent	4 x 4L	**17,933-7**
Hexane 95+% spectrophotometric grd	4 x 4L	**27,050-4**
Toluene 99.5+% ACS Reagent	4 x 4L	**17,941-8**
Ethyl Acetate 99.5+% ACS Reagent	4 x 4L	**44,350-6**
Petroleum ether ACS Reagent	4 x 4L	**18,451-9**
ACS grade Reagents		
Puriss.p.a. grade Reagents		
Acetonitrile ≥99.5% ACS Reagent, Ph. Eur. (GC)		**33019**
Chloroform 99.0-99.4% Reagent, Ph. Eur. ISO, (GC)		**32211**
Dichloromethane ≥99.9% ACS Reagent, ISO, (GC)		**32222**
Methanol ≥99.8% ACS Reagent, Ph. Eur. ISO, (GC)		**32213**
Hexane ≥99% ACS Reagent, Ph. Eur. (GC)		**32293**
Toluene ≥99.7% ACS Reagent, Ph. Eur. ISO, (GC)		**32249**
Ethyl acetate ≥99.5% ACS Reagent, Ph. Eur. ISO, (GC)		**33211**
Petroleum ether bp 60-80°C		**32248**
Petroleum ether Ph Eur high boiling bp 50-70°C		**32247**
Petroleum ether low boiling point hydrogen treated naphtha bp 30-50°C		**32246**
TLC Plates		
Glass silica TLC plate w/ fluorescent indicator		**Z12,269-6**
C18 silica gel TLC plate w/ fluorescent indicator		**Z26,548-9**

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