

## Choosing an SPE Product

These are the typical steps when you have to choose the proper SPE product for your application:

### 1. Characterize your sample.

The polarity of your analyte vs. the matrix, the charged functional groups, molecular weight, solubility influence how the analyte is retained by the SPE bed.

### 2. Choice of Retention Strategy

Two basic methods exist for the sample treatment

- Select the proper packing to retain the target analyte. The contaminants are washed off and the target analyte is elute for further analyses.
- Select the proper packing to retain the contaminant. The target analyte directly pass through the SPE column.

### 3. Choose the Proper Packing

According with the previous points you need to select the proper packing to obtain the highest recovery and cleanest extract.

- Reversed-Phase silica based materials are hydrophobic and retain moderately polar to non-polar interferences. Or retain non-polar contaminants while the polar compounds pass through the bed.
- Normal-Phase silica based materials are hydrophilic and retain polar compounds from non-polar matrix. Or retain polar contaminants while the non-polar compounds pass through the bed.
- Ion-Exchange Resins retain charged compounds and/or remove ionic interferences.

### 4. Optimization of the Conditions

Choose the bed size and most appropriate solvents.

- Too big packing bed results in incomplete elution; too small bed results in incomplete retention.
- Consider the solvent strength versus the packing material. The solvent of the conditioning step should not act as eluting solvent. Buffers should be used to control ionization of charged compounds.
- The washing solvents should not be strong enough to elute the target analyte. It should only removes the retained interferences.
- Elution solvents should be strong enough to fully elute an analyte in a small volume (<2mL)

## SPE Method Development

Commonly an SPE Method Development contains 4 Steps :

### Step 1 : Condition the SPE Column

Two Substeps compose the conditioning step :

- activation of the sorbent ligands
- equilibration of the sorbent bed.

### Step 2 : Load the sample

In this step the sample is applied to the SPE column. Flow Rate and Matrix are optimized to retain the analyte of interest quantitatively.

### Step 3 : Wash the SPE Column

You need to choose the proper solvent to elutes the impurities and retain the target analytes. Often the second conditioning solvent is a good choice of washing.

### Step 4 : Elute the Analyte

The elution step should remove all target analytes with the minimum solvent to maximize the sensitivity. A combination of solvents is someti-

General SPE Method Development Procedures				
Packing	Step 1 - Conditioning	Step 2 - Loading	Step 3 - Washing	Step 4 - Eluting
<b>Reversed-Phase</b> Mechanism : retain moderately polar to non-polar compounds from a polar sample matrix	MeOH followed by Water	Process sample at 1-5mL/min Flow	Water or Water:MeOH (95:5)	MeOH or Acetonitrile. To break secondary interaction may need to add a strong acid or base.
<b>Normal-Phase</b> Mechanism : retain polar compounds from a non-polar sample matrix	IPA followed by Hexane	Process sample at 1-5mL/min Flow	Hexane or Hexane:IPA (98:2)	IPA, Ethyl Acetate, Acetone, Hexane:IPA (50:50)
<b>Ion-Exchange</b> Mechanism : retain charged compounds (negative/anionic or positive/cationic)	MeOH:Water (50:50) followed by Low Ionic strength (0.1M) Buffer	Process sample at ≤1mL/min Flow. Ion Exchange kinetics are slower than RP or NP	MeOH:Low Ionic Strength (0.1M) Buffer (10:90)	High Ionic Strength (0.5M-1.0M) Buffer or pH modifier to uncharge the analyte. Add organic if need to break hydrophobic interactions.

Recommended Guideline			
Bed Size / Sorbent Capacity	Condition Volume (4 Bed Volume)	Wash Volume (6 Bed Volume)	Minimum Elution Volume (3 Bed Volume)
50mg / 2.5mg	0.30mL	0.45mL	0.23mL
100mg / 5mg	0.60mL	0.90mL	1.80mL
200mg / 10mg	1.20mL	1.80mL	0.90mL
500mg / 25mg	3.00mL	4.50mL	2.25mL
1000mg / 50mg	6.00mL	9.00mL	4.50mL
2000mg / 100mg	12.00mL	18.00mL	9.00mL
5000mg / 250mg	30.00mL	45.00mL	22.50mL
10000mg / 500mg	60.00mL	90.00mL	45.00mL

The above table is for estimation purpose only. Must optimize for each application

### Technical Tip !

#### Sorbent Bed

Typically 150µL for every 150mg Sorbent

#### Retention Capacity

It describe the total amount that the sorbent will bind, including all the compounds retained, target analytes and contaminants.

#### Elution Volume

The Minimum Elution Volume suggested in the table on the left will offer the best sensitivity, however more solvent may be necessary, depending by the application

# GracePure™ - Solid Phase Extraction Columns

## GracePure™ Choices

GracePure™ SPE product line is suitable in pharmaceutical, agriculture, food & beverage, petrochemical and environmental application. These products deliver the selectivity and high recovery you expect from an SPE process. This guide help you to choose the appropriate sorbent, bed size, and solvent volumes to obtain a cleaner, more concentrated sample at the end of your SPE process, ready for further analyses and investigation.

## Highest Quality Control

Every part of GracePure™ SPE manufacturing process is carefully monitored. From silica production to final product, over 30 tests are performed, and the products come with a comprehensive quality assurance certificate that displays the 18 most meaningful results to the SPE user.\*

\*Applies to silica-based media.

## Component Tests

GC/FID shows that GracePure™ tubes are constructed from a highly inert grade of polypropylene to prevent extractable contamination. Polyethylene frits are thoroughly washed in organic solvent which also eliminates extractables.

## Manufacturing Control

GracePure™ SPE products are packed and assembled using custom-designed, precision equipment. Every manufacturing batch is guaranteed to have less than 2% bed weight variation and uniform flow rates.



GracePure™ Sorbent Specification - Reversed-Phase and Normal-Phase									
Packing	Support	% Carbon	End Capped	Surface Area	Average Particle Size	Pore Size	Feature	Benefits	Typical Application
C18-Max	Silica	17.1%	Yes	518 m <sup>2</sup> /g	50μ	60Å	Polymerically bonded	Highest binding capacity, best for complex samples or structurally diverse analytes.	Drugs and their metabolites in serum and plasma, pesticides
C18-Aq	Silica	12.5%	Yes	518 m <sup>2</sup> /g	50μ	60Å	Hydrophillic endcapping	100% Water-wettable C18 ideal for aqueous samples. Phase remains active even when completely dry	Desalting proteins, pharmaceuticals, hormones, pesticides, organics in water
C18-Low	Silica	6.5%	Yes	518 m <sup>2</sup> /g	50μ	60Å	Least hydrophobic	C18 phase that easily releases very hydrophobic compounds.	Surfactants, oils, antibiotics
C18-Fast	Silica	7.0%	Yes	518 m <sup>2</sup> /g	100μ	60Å	Large particle size	Process large volume (>500mL) or viscous samples with fast flow rates	Aniline, pesticides,haloethers, phthalate esters, EPA 3620, 3610
Silica	Silica	N/A	N/A	518 m <sup>2</sup> /g	50μ	60Å	Most polar phase	Able to differentiate between structurally similar compounds	Aflatoxins, pesticides,steroids, structural isomers
Amino	Silica	4.3%	No	518 m <sup>2</sup> /g	50μ	60Å	Duel retention	Retains polar compounds, or can act as a weak anion exchanger. Easily releases strong acids when SAX binds too strongly.	Carbohydrates, dyes, lipids, mycolotoxins, strong acids
Diol	Silica	N/A	No	518 m <sup>2</sup> /g	50μ	60Å	Reproducible polar bonded phase	Very polar phase that has the same benefits as silica, but wets easily and offers more reproducibility.	Alkaloids, lipids, oils, structural isomers

GracePure™ Sorbent Specification - Ion-Exchange Phase							
Packing	Support	Exchange Capacity	Counter Ion	Average Particle Size	Feature	Benefits	Typical Application
Anion-X	8% cross-linked Styrene-DVB	1.5 meq/g	Acetate Form	50μ	Tetramethyl ammonium functional group on polymer	pH range from 1–14, with excellent exchange capacity	Anionic compounds: organic acids, fatty acids
Cation-X	8% cross-linked Styrene-DVB	2.4 meq/g	Hydrogen Form	50μ	Benzene sulfonic acid functional group on polymer base material	pH range from 1–14, with excellent exchange capacity.	Cationic compounds: amines, amino acids

Cross Reference Tradename List						
GracePure™	Phenomenex Strata™	Waters Spe-Pak®	Supelco Discovery™ Supelclean™	Agilent Bond-Elute®	JT Baker Bakerbond™	Biotage Isolute®
C18-Max	C18-E	tC18	DSC-18	C18	Octadecyl	C18 (EC)
C18-Aq	C18-U	C18	DSC-18Lt	C18OH	Octadecyl lightload	MF C18
C18-Low	---	---	LC-18	---	---	---
C18-Fast	---	---	---	---	---	---
Silica	Si-1	Silica	DSC-Si or LC-Si	LC-Si	Silica Gel	SI
Amino	NH2	NH2	LC-NH2	NH2	Amino	NH2
Diol	---	Diol	DSC-Diol or LC-Diol	2OH	---	Diol
Anion-X	SAX	---	DSC-SAX or LC-SAX	SAX	Quaternary Amine	Sax
Cation-X	SCX	---	DSC-SCX or LC-SCX	SCX	Aromatic Sulfonic Acid	Scx

## GracePure™ SPE Columns

- Reversed-Phase Columns
- Normal-Phase Columns
- Ion-Exchange Columns

GracePure™ - Reversed-Phase Columns				
Packing	Bed Weight / Tube Volume	Qty	Old Alltech #	Part.No
C18-MAX	50mg / 1mL	100	5141484	LD0225
	100mg / 1mL	100	5138765	LD0207
	200mg / 3mL	50	5141686	LD0001
	500mg / 3mL	50	5138766	LD0208
	500mg / 6mL	30	5138767	LD0209
	1000mg / 6mL	30	5138768	LD0210
C18-Aq	500mg / 20mL	20	5141525	LD0234
	5000mg / 20mL	20	5141524	LD0233
	50mg / 1mL	100	5141486	LD0226
	100mg / 1mL	100	5138774	LD0214
	500mg / 3mL	50	5138775	LD0215
C18-Low	1000mg / 6mL	30	5138776	LD0242
	2000mg / 12mL	30	5141482	LD0224
	5000mg / 20mL	20	5141523	LD0232
	100mg / 1mL	100	5138760	LD0202
	200mg / 3mL	50	5138761	LD0203
C18-Fast	500mg / 3mL	50	5138762	LD0204
	500mg / 6mL	30	5138763	LD0205
	1000mg / 6mL	30	5138764	LD0206
C18-Fast	500mg / 3mL	50	5138758	LD0200
	1000mg / 6mL	30	5138759	LD0201
5000mg / 20mL	20	5141527	LD0235	

### Fungicides from Red Wine

#### Procedure using GracePure™ C18-Max, 500mg:

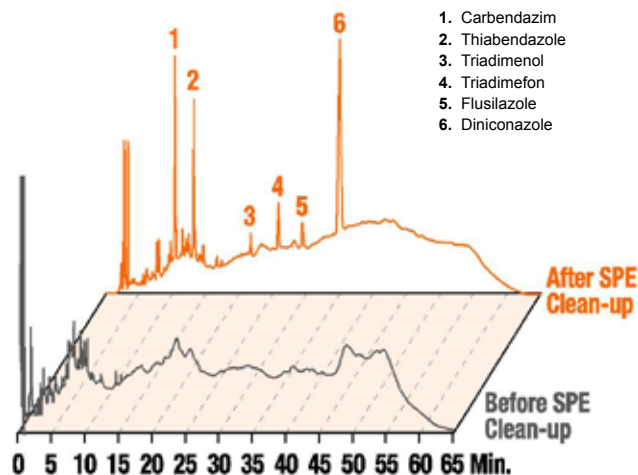
**Sample Treatment** – Add 0.167mg/mL each of carbendazim, thiabendazole, triadimenol, triadimefon, flusilazole, diniconazole into 1mL water. Combine with 1mL Beaujolais red wine.

**Conditioning** – Rinse device with 3mL methanol followed by 3mL water.

**Sample Application** – Apply 2mL red wine mixture.

**Wash** – No wash.

**Elution** – Elute with 3mL methanol.



**Column:** Alltima™ HP C18 Amide, 5µm, 250 x 4.6mm HPLC Column (Part No. BF0163)  
**Mobile Phase:** A: Water B: Acetonitrile  
**Gradient:** (Time, %B): (0,15%), (15,45%), (50,45%), (65,15%)  
**Flow Rate:** 1mL/min  
**Detector:** UV at 254nm

### Carbamate Pesticides from Water

#### Procedure using GracePure™ C18-Fast, 500mg:

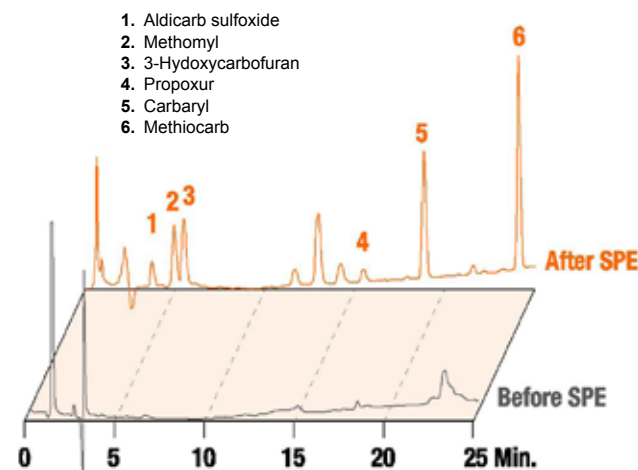
**Sample Treatment** – Spike 500mL tap water with 125µL carbamate solution for final concentration of 25ppb.

**Conditioning** – Rinse with 3mL acetonitrile:water (80:20) followed by 3mL water. Dry with vacuum.

**Sample Application** – Apply 500µL sample.

**Wash** – 2 x 3mL water.

**Elution** – Elute with 4 x 1mL acetonitrile:water (80:20)



**Column:** Platinum™ EPS C18, 5µm, 250 x 4.6mm HPLC Column (Part No. BC0001)  
**Mobile Phase:** A: DI water B: Acetonitrile  
**Gradient:** (Time, %B): (0,25), (5,25), (20,50), (25,50), (30,25)  
**Flow Rate:** 1mL/min  
**Detector:** UV at 210nm  
**Temperature:** Ambient

### GracePure™ - Normal-Phase Columns

Packing	Bed Weight / Tube Volume	Qty	Old Alltech #	Part.No
Silica	100mg / 1mL	100	5138777	LD0216
	200mg / 3mL	50	5138778	LD0217
	500mg / 3mL	50	5138779	LD0218
	500mg / 6mL	30	5138781	LD0220
	1000mg / 6mL	30	5138782	LD0221
	2000mg / 12mL	30	5138783	LD0222
Amino	5000mg / 20mL	20	5138780	LD0219
	10000mg / 60mL	16	5138784	LD0223
	500mg / 3mL	50	5138752	LD0196
Diol	1000mg / 6mL	30	5138753	LD0197
	500mg / 3mL	50	5138773	LD0213

### GracePure™ - Ion-Exchange Columns

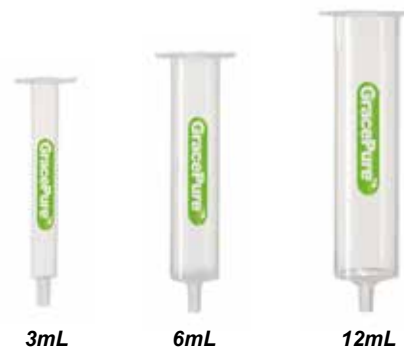
Packing	Bed Weight / Tube Volume	Qty	Old Alltech #	Part.No
Anion-X	100mg / 1mL	100	5138754	LD0198
	500mg / 3mL	50	5138755	LD0199
	1000mg / 6mL	50	5141487	LD0227
Cation-X	500mg / 6mL	100	5138769	LD0211
	500mg / 3mL	50	5138770	LD0212
	1000mg / 6mL	50	5141488	LD0228

## GracePure™ Florisil SPE Columns

Florisil® is a commonly used sorbent for the clean-up or concentration of samples in environmental analysis.

We can offer you Florisil® SPE columns with:

- Highest quality control for maximum reproducibility
- A Quality Certificate in every box
- Less than 2% bed weight variation
- Highly inert tubes and frits to prevent extractable contamination
- **At Exceptional Price**



GracePure™ Sorbent Specification - Florisil®							
Support	Endcapping	Surface Area	Particle Size	Pore Size	Feature	Benefits	Typical Application
Magnesium Silicate	No	298 m <sup>2</sup> /g	120µ	60Å	Alternate Polar Phase	Large Particle Size processes large sample sizes quickly	Environmental

GracePure™ - Florisil® SPE Columns				
Packing	Bed Weight	Qty	Old Alltech #	Part.No
Florisil®	500mg / 3mL	50	5141522	LD0231
	1000mg / 6mL	30	5141520	LD0229
	2000mg / 12mL	30	5141521	LD0230

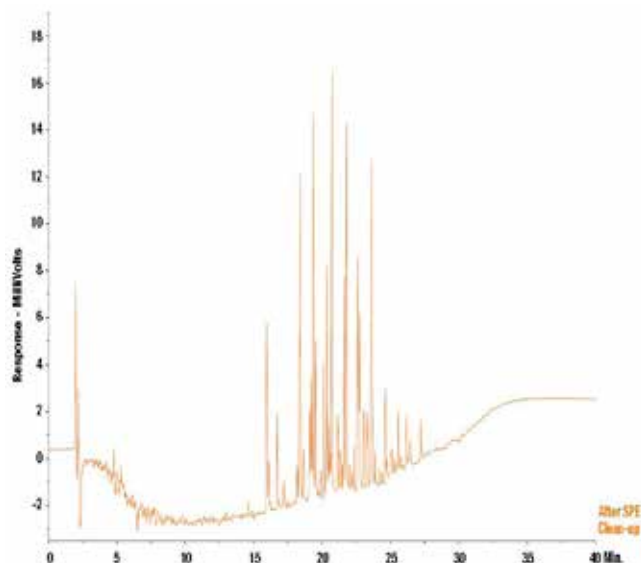
### PCBs in Transformer Oil

**SPE Column : GracePure™ Florisil® 1000mg/6mL (Part No LD0229):**

*Sample Treatment* – Dissolve 0.25g of transformer oil spiked with Aroclor 1254 (at a concentration of 50mg/kg) in 20mL of n-Hexane.

*Conditioning* – 2 x 5mL n-Hexane, making sure the column does not dry.

*Sample Application* – Attach 25mL reservoir (Part No. LA0006) with a syringe adapter and run sample through conditioned Florisil® column, aspirating all the solution from the tube. Evaporate the volume down to 4mL and analyzed by GC/ECD.



Column: AT™5ms column, 30m x 0.25mmID, 0.25µ film (Part No. PG0062)  
 Oven: 150°C (2 min) - 300°C (8 min) at 5°C/min  
 Linear Velocity: 25 cm/second  
 Flow: 0.74 mL/min  
 Split Ratio: 64:1, SGE FocusLiner with glass wool  
 Injector: 275°C

## GracePure™ Florisil® SPE Columns



### Typical applications:

#### Clean-up of pesticide residues (EPA 3620 norm):

- Chlorinated Hydrocarbons
- Organochlorine Pesticides (NF EN 12393)
- Organophosphorus pesticides
- PCBs
- Phthalate Esters
- Nitrosamines
- Nitroaromatics
- Haloethers

#### Determination of hydrocarbons in water (ISO 9377-2 norm)

#### Determination of Aflatoxins and Tricothecenes in food

#### Isolation of Antibiotics, Steroids and Alkaloids

#### Separation of Lipids

#### Purification of pharmaceuticals

#### Decolorization of oils, fats and waxes

# Solid Phase Extraction - Accessories

## Vacuum Manifolds

- 12 position, 16 position and 24 position Vacuum Manifolds available for maximum productivity
- Glass chamber for visula monitoring
- Use standard Male Luer devices

These manifolds permit consistent extraction and filtration results. Multiple sample processing with these manifolds simplifies procedures and saves time. The manifolds consist of a clear glass chamber and lid to which a vacuum is applied to draw a sample through an SPE column, cartridge, or disk.

Adjustable racks placed in the glass vacuum chamber will accommodate a variety of sample collection vessels, including test tubes, autosampler vials, volumetric flasks, and Erlenmeyer flasks. Eluants are deposited directly into the collection vessel of choice via polypropylene, optional stainless steel, or Teflon needles. Drying attachments for the 12 and 24 port manifolds will direct a flow of air or nitrogen into the collection vessels to dry eluants prior to further analysis. Drying attachments can also be connected, via adapters, to SPE columns or cartridges in order to dry the column or cartridge prior to final elusion. Optional disposable solvent resistant polypropylene liners are available for the twelve port manifolds. These waste liners greatly simplify sample preparation, solvent disposal, and clean-up



12 Position Manifold and Accessories			
Description	Qty	Old Alltech #	Part.No
<b>12 Position Manifold</b>	<b>1</b>	<b>210351</b>	<b>LA0033</b>
<i>Replacement parts</i>			
Cover, Gasket & 12 Stopcocks	1	212001	LA0034
Glass Chamber	1	213212	LA0035
Vacuum Gauge, Valve & Glass Chamber	1	212304	LA0035
Collection Rak, Legs, Clips & Post*	1	212518	LA0036
Gaskets	2	212112	LA0037
Plate - 13mm	1		LA0038
Plate - Volumetric Flask	1		LA0039
Plate - 16mm Test Tube	1		LA0040
Plate - Autosampler Vials	1		LA0041
Plate - Dimple	1		LA0042
Plate - Base	1		LA0043
Waste Container	1	210033	LA0045



12 Position Manifold



Waste Container

\* Contains 3 support posts, bottom plate, 13&16mm plates, autosampler vial plate, volumetric plate, 12 retention clips.



24 Position Manifold

16 Position Manifold and Accessories			
Description	Qty	Old Alltech #	Part.No
<b>16 Position Manifold</b>	<b>1</b>		<b>LA0046</b>
<i>Replacement parts</i>			
Cover, Gasket & 16 Stopcocks	1		LA0047
Glass Chamber	1		LA0048
Vacuum Gauge, Valve & Glass Chamber	1		LA0050
Collection Rak, Legs, Clips & Post*	1		LA0051
Gaskets	2		LA0052
Plate - 13mm	1		LA0053
Plate - 16mm Test Tube	1		LA0054
Plate - Dimple	1		LA0055
Plate - Base	1		LA0056

\* Contains 3 support posts, bottom plate, 13&16mm plates, autosampler vial plate, volumetric plate, 12 retention clips.

24 Position Manifold and Accessories			
Description	Qty	Old Alltech #	Part.No
<b>24 Position Manifold</b>	<b>1</b>	<b>210224</b>	<b>LA0058</b>
<i>Replacement parts</i>			
Cover, Gasket & 24 Stopcocks	1	211224	LA0059
Glass Chamber	1	210124	LA0060
Vacuum Gauge, Valve & Glass Chamber	1	210324	LA0061
Collection Rak, Legs, Clips & Post*	1	210424	LA0062
Gaskets	2	210724	LA0063
Plate - 13mm	1		LA0064
Plate - 16mm Test Tube	1		LA0065
Plate - Dimple	1		LA0066
Plate - Base	1		LA0067

\* Contains 3 support posts, bottom plate, dimple plate, 13&16mm plates, 12 retention clips.

# Solid Phase Extraction - Accessories

## Manifold Accessories and Needles

- Replacement Parts for All Models
- Stainless Steel, Polypropylene and Teflon Needles
- Stopcocks

Manifold Accessories			
Description	Qty	Old Alltech #	Part.No
<i>Drying Attachments</i>			
12 Position Drying Attachemnt	1	212100	LA0020
16 Position Drying Attachemnt	1	212117	LA0021
24 Position Drying Attachemnt	1	212124	LA0022
<i>Stopcocks</i>			
12-16-24 Position Stopcocks	100	212112	LA0024
<i>Needles</i>			
Polypropylene Needles	24	210924	LA0025
Stainless Steel Needles	24	210824	LA0026
Teflon Needles	100	412410	LA0027
Valved Teflon Needle	25	411525	LA0028
<i>Accessories</i>			
Vacuum Gauge & Valve	1	212203	LA0029
Female Luer Fitting	2	212002	LA0030
Male Luer Fitting	2	212120	LA0031
Vacuum Manifold Plug	50	211234	LA0032



Teflon, Polypropylene and SS Needles



Stopcocks



12 Port Drying Attachment



Empty SPE Columns and Frits



Inlet Caps and Plugs

## SPE Empty Column and Frits

- Select Empty Reservoirs and Frits to Pack Your Own Custom SPE Columns

SPE Empty Column and Frits			
Description	Qty	Old Alltech #	Part.No
<i>Empty Polypropylene Columns</i>			
1.5mL	100	210001	LA0003
4.0mL	100	210104	LA0004
8.0mL	100	210208	LA0005
15.0mL	100	210315	LA0001
25.0mL	100	210425	LA0006
75.0mL	100	210575	LA0007
<i>Polyethylene Frits</i>			
For 1.5mL Column	100	211401	LA0008
For 4.0mL Column	100	211404	LA0009
For 8.0mL Column	100	211408	LA0010
For 15.0mL Column	100	211412	LA0002
For 25.0mL Column	100	211416	LA0011
For 75.0mL Column	100	211775	LA0012
<i>Caps &amp; Plugs</i>			
Inlet Caps for 1.5mL	100	222000	LA0013
Inlet Caps for 4.0mL	100	220301	LA0014
Inlet Caps for 8.0mL	100	220600	LA0015
Inlet Caps for 15.0mL	100	221200	LA0017
Inlet Caps for 25.0mL	100	221006	LA0018
Inlet Caps for 75.0mL	100	227503	LA0019
Outlet Caps for all Size	100	220710	LA0016