



high performance silica products

from **RESTEK**

 CHROMalytic +61(0)3 9762 2034	Australian Distributors Importers & Manufacturers www.chromtech.net.au
 ECHnology Pty Ltd	
Website NEW : www.chromalytic.com.au E-mail : info@chromtech.net.au Tel: 03 9762 2034 . . . in AUSTRALIA	

Chromatographic Silica: Background

Introduced in the early 1970s, the first commercial HPLC silica particles were relatively large (10-15µm mean diameter), irregularly shaped, and, by today's standards, chromatographically inefficient. In the late 1970s and early '80s, spherical silica began to appear on the market. Spherical particles pack much more uniformly, and generally have higher surface areas than irregular particles. These characteristics translate into more efficient separations (i.e., more theoretical plates). The first spherical particles also were in the 10µm range, but over the years particle size has been reduced to the current standard, 5µm, and use of 3µm and smaller particles has been steadily increasing.

With the movement from irregular silica to small, high purity spherical silica, many US HPLC column manufacturers ceded silica manufacture to Europe and Asia. Only a handful of US companies now manufacture chromatographic grade silica, including companies who manufacture silica solely for their own use. Even fewer US silica manufacturers offer their materials to the wider HPLC column manufacturing community.

Now, Restek offers two types of spherical silica, available as bulk silica or bonded phases. Pinnacle II™, a Type A silica, is similar to spherical silica materials such as Hypersil® ODS. Pinnacle™ DB silica is a highly base deactivated material that is suitable for a wide range of analytes, including basic compounds. It performs very similarly to Hypersil® BDS material.

Modern Chromatographic Silica

Critical parameters for superior chromatographic materials include narrow particle size and pore size distributions, low metals content, particle stability under pressure and, of course, chromatographic performance. Mechanical strength is a primary concern. Without rugged material it is difficult to pack an efficient column, and column lifetime can be short. If the silica cannot withstand typical packing pressures of up to 12,000 psi, particles can be crushed, creating 'fines'. Fines can increase system back-pressure, crushing additional particles and further degrading performance. Particle collapse also can create voids within the packed bed in HPLC columns; this can lead to split or tailing peaks in the chromatogram.

How important is metals content?

Metal ions in a silica particle weaken the particle and negatively affect chromatography, particularly for basic analytes. Basic compounds can interact with the electron-accepting metal ions, causing peak tailing. Some silica manufacturers overcome these problems - temporarily - by annealing the metals into the framework of the particles. But, as the particles age, metal ions are re-exposed. In contrast, Restek's silica manufacturing processes remove metal impurities from the particles, so Pinnacle II™ and Pinnacle™ DB silicas do not require annealing. They provide consistently symmetric peaks for bases as the column ages, and thus offer a longer column lifetime.

Manufacturing Scale: Smaller is Better

At Restek, we manufacture chromatographic grade silica in small (kilogram) batches. We believe a small batch size imparts several advantages:

- Ability to optimize the process based on the physical properties of the raw materials. This creates a more uniform product from batch to batch.
- Ability to set very tight specifications on physical properties. Out-of-specification materials are discarded, never sold.
- Ability to quickly create silica with unique physical properties, to meet customers' specific performance needs. Our stock silicas have 110Å (Pinnacle II™) or 140Å (Pinnacle™ DB) pores, but to date we have made materials ranging from a 50Å, high surface area silica to a 300Å, wide pore silica.

Quality Control

Every lot of Restek silica is put through extensive quality testing before it is released. Parameters tested include:

- Metals content
- Particle size distribution
- Pore size distribution
- Stability under pressure
- Chromatographic performance

Typical physical properties of 5µm Pinnacle II™ and Pinnacle™ DB Silica are given in Table 1.

If the material meets all specifications, a small amount is bonded with an octadecylsilyl (C18) phase, the material is packed in an HPLC column, and chromatographic performance is tested. Most testing is conducted in our ISO registered facility; metals content is determined by an independent laboratory.

Table 1. Typical physical properties of Restek HPLC silica.

Parameters	Pinnacle II™ Silica	Pinnacle™ DB Silica
Median Particle Size	5.0 ± 1.5µm	5.0 ± 1.5µm
Surface Area	~180 m ² /g	~140 m ² /g
Mean Pore Size	~110 Å	~140 Å
Mean Pore Volume	~0.60	~0.70

Restek HPLC Columns and Accessories

General-purpose and special-purpose HPLC columns and guard columns, plus instrument parts, innovative tools, other accessories, and many example chromatograms from Restek columns. Request lit. cat.# 59241A-INT (114 pages).



Restek Silica Products

Pinnacle II™ Silica

Pinnacle II™ porous spherical silica is suitable for a wide range of analytical applications. It is a 'Type A' material, similar in physical characteristics to industry standard materials such as Hypersil® ODS. It has a moderate surface area (approximately 180m²/g) and its surface metals content is lower than that of Hypersil® ODS (Figure 1). Example applications for Pinnacle II™ silica-based columns are shown in Figures 2 and 3. Highly consistent performance, lot to lot, makes Pinnacle II™ materials an excellent first choice for routine applications involving neutral to slightly acidic compounds.

Figure 1. Physical and chemical parameters of Pinnacle II™ silica.

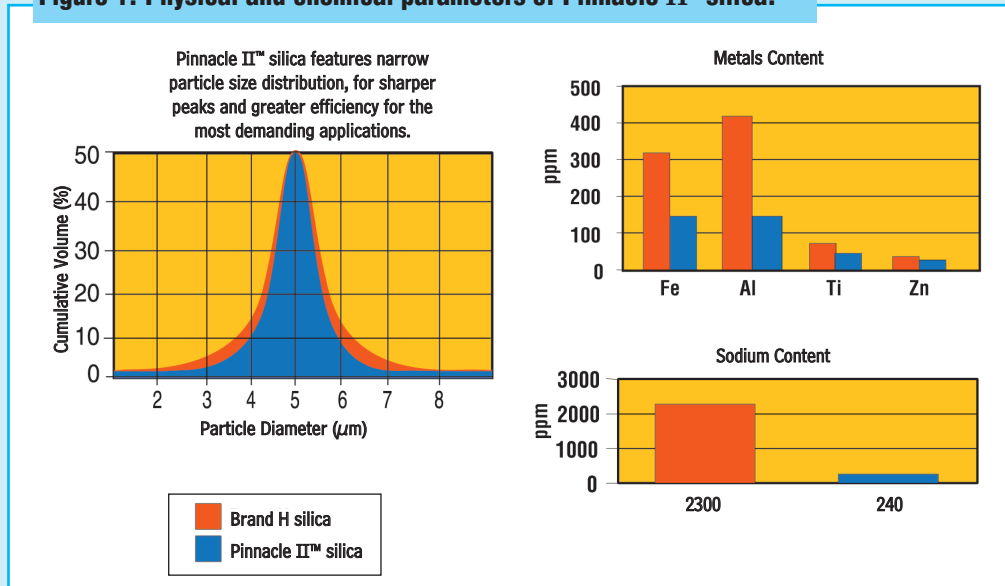


Figure 2. Capsaicinoids: rapid heat level assay for habenero nuggets on Pinnacle II™ C18.

Column: Pinnacle II™ C18
Cat. #: 9214565
Dimensions: 150 x 4.6mm
Particle size: 5µm
Pore size: 110Å

Sample:
Inj.: 10µL
Conc.: 4000µg/mL habenero pepper nugget extract
Solvent: ethanol

Conditions:
Mobile phase: A: 1% acetic acid in water (v/v)
 B: acetonitrile

Time (min.):	% B
0	50
2	50
9	80
12	80
13	50

Flow: 1.0mL/min.
Temp.: ambient
Det.: UV @ 280nm

Peak List:
 1. nordihydrocapsaicin
 2. capsaicin
 3. dihydrocapsaicin

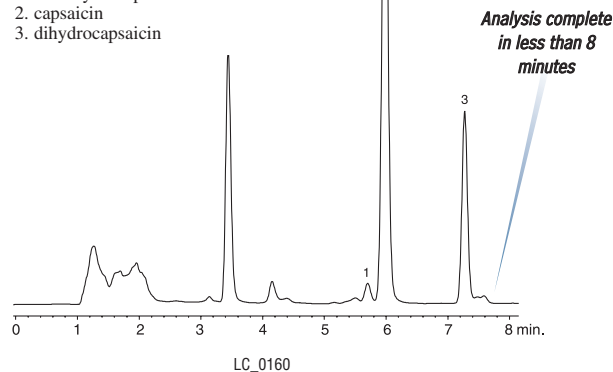


Figure 3. Corticosteroids on a Pinnacle II™ Cyano column – an excellent alternative to C18 columns.

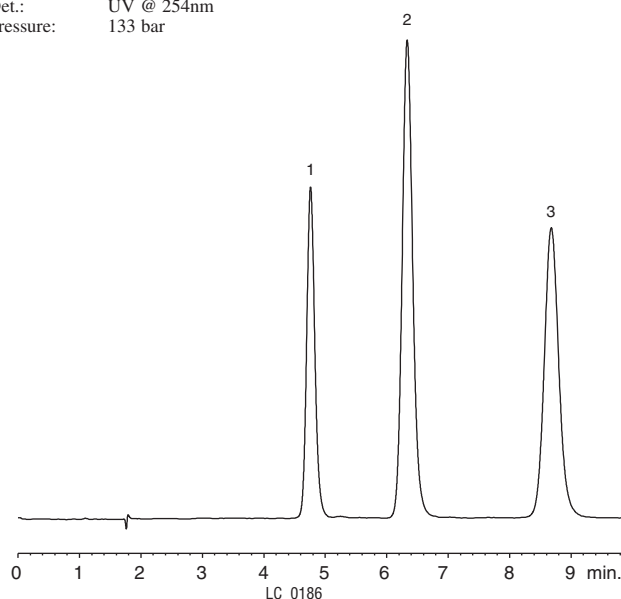
Column: Pinnacle II™ Cyano
Cat. #: 9216565
Dimensions: 150 x 4.6mm
Particle size: 5µm
Pore size: 110Å

Sample:
Inj.: 5.0µL
Solvent: methanol

Conditions:
Mobile phase: water:methanol (65:35, v/v)
Flow: 1.0mL/min.
Temp.: 30°C
Det.: UV @ 254nm
Pressure: 133 bar

Peak List:

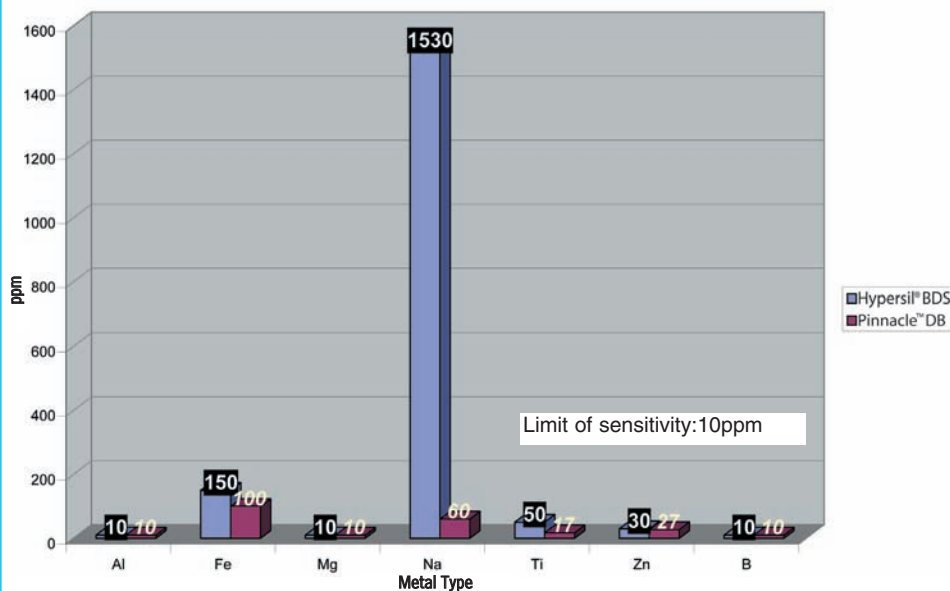
Peak	Conc. (mg/mL)
1. cortisone	0.1
2. corticosterone	0.5
3. cortisone acetate	0.7



Pinnacle™ DB Silica

Pinnacle™ DB is highly base-deactivated spherical silica. During the manufacturing process metal ions are stripped away (Figure 4) and the pore walls and particle surface are made more uniform. The resulting material has physical and performance characteristics very similar to

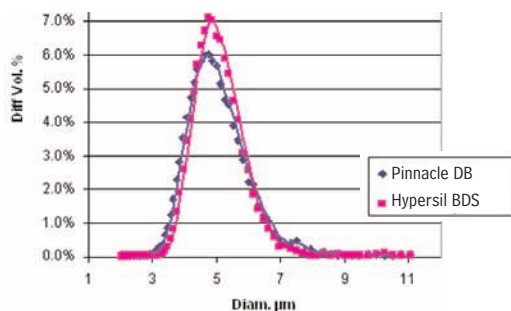
Figure 4. Consistently low metals content in Pinnacle™ DB silica ensures sharper, more symmetric peaks for basic analytes.



those of Hypersil® BDS (Figures 5 & 6), and is an excellent support for analyses of basic compounds. Elimination of residual metals from the silica greatly reduces the likelihood of peak tailing due to analyte interactions with the surface. Consequently, the need for mobile phase modifiers is significantly reduced, or eliminated, simplifying mobile phase preparation.

Analysis of xanthenes (basic compounds) using a Pinnacle™ DB C18 column is an example of the chromatography that can be achieved (Figure 7). For additional example applications, and direct comparisons to the performance of Hypersil® columns, request free publications *New Pinnacle™ DB HPLC Columns (lit. cat.# 59499)* and *Pinnacle™ DB HPLC Columns as Replacements for Hypersil® BDS (lit. cat.# 59742)*.

Figure 5. Equivalent particle size distribution for Pinnacle™ DB silica and Hypersil® BDS silica.



Particle Size Analysis	Pinnacle™ DB	Hypersil® BDS
Geometric Mean	4.81 ± 1.20 µm	4.89 ± 1.70 µm
Arithmetic Mean	4.89 ± 0.94 µm	4.95 ± 0.82 µm
Median	4.69 µm	4.81 µm
Mode	4.69 µm	4.79 µm
Max Diff.	6.04%	7.11%

Figure 7. Rapid analysis of xanthenes, with excellent separation of theophylline and β-hydroxyethyltheophylline, on a Pinnacle™ DB C18 column.

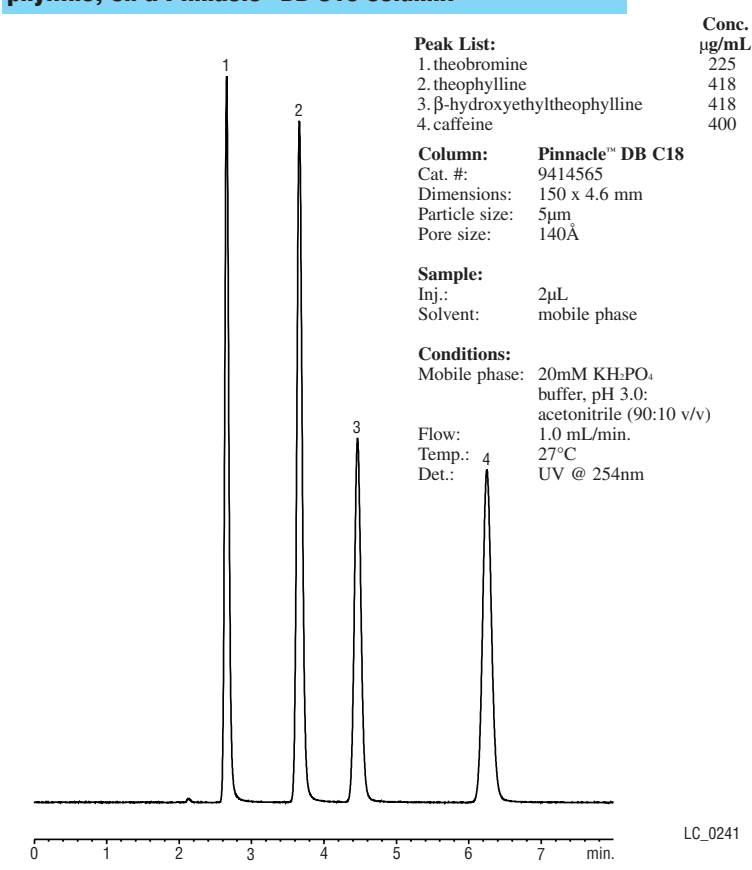
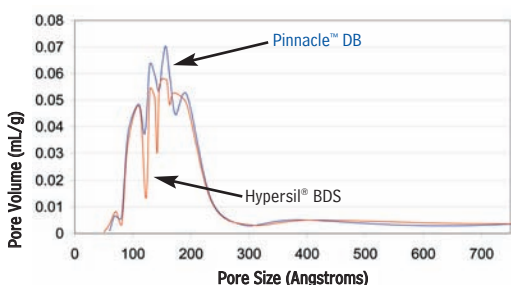


Figure 6. Pinnacle™ DB silica and Hypersil® BDS silica have remarkably equivalent pore volume distributions.



Pore Analysis: Pinnacle™ DB	
Surface Area:	150 (m ² /g)
Pore Size:	140 (Å)
Pore Volume:	0.66 (mL/g)

www.restekcorp.com

Bonded Phases

Pinnacle II™ silica is available with C18, C8, cyano, amino, phenyl, or PAH phases. Pinnacle™ DB is available with C18, C8, or cyano phases. We can bond other stationary phases to high-performance Restek silica, and will be happy to work with you to provide what you need.

Figure 8. Sharp, well separated peaks for SRM 870 Mix components on a Pinnacle II™ C18 column.

Column: Pinnacle II™ C18
Cat. #: 9214565
Dimensions: 150 x 4.6mm
Particle size: 5µm
Pore size: 110Å

Sample:
Inj.: 5µL
Solvent: methanol

Conditions:
Mobile phase: 5mM potassium phosphate
 pH 7:methanol (20:80 v/v)
Flow: 2.0mL/min.
Temp.: ambient
Det.: UV @ 254nm

Peak List:	Ret. time (min.)	Conc.
1. uracil	0.783	28.0µg/mL
2. toluene	1.801	1.4mg/mL
3. ethylbenzene	2.236	1.7mg/mL
4. quinizarin	4.377	94.0µg/mL
5. amitriptyline	4.624	2.8mg/mL

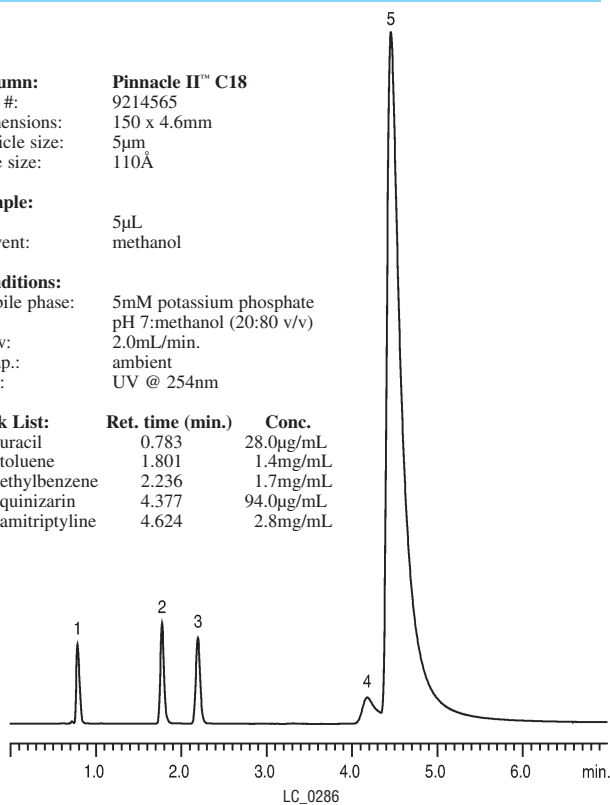


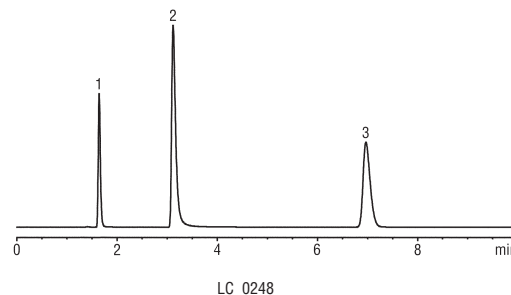
Figure 9. Test mix on a Pinnacle™ DB C18 column: rapid analysis, sharp symmetric peak for basic pyridine.

Column: Pinnacle™ DB C18
Cat. #: 9414565
Dimensions: 150 x 4.6mm
Particle size: 5µm
Pore size: 140Å

Sample:
Inj.: 5.0µL
Solvent: mobile phase

Conditions:
Mobile phase: 20mM potassium phosphate, pH 7.0:
 acetonitrile (80:20 v/v)
Flow: 1.0mL/min.
Temp.: ambient
Det.: UV @ 254nm

Peak List:	Ret. Time (min.)	Conc.
1. uracil	1.643	5.0µg/mL
2. pyridine	3.119	0.1µL/mL
3. phenol	6.969	1.86mg/mL



Product Listing

Bulk Packing Materials

Minimum order quantity: 5g

Description	cat.#
3µm Pinnacle II™	
Pinnacle II™ C8 Bulk Packing	92133
Pinnacle II™ C18 Bulk Packing	92143
Pinnacle II™ Cyano Bulk Packing	92163
Pinnacle II™ Phenyl Bulk Packing	92153
5µm Pinnacle II™	
Pinnacle II™ C8 Bulk Packing	92135
Pinnacle II™ C18 Bulk Packing	92145
Pinnacle II™ Cyano Bulk Packing	92165
Pinnacle II™ Phenyl Bulk Packing	92155
5µm Pinnacle™ DB	
Pinnacle™ DB C18 Bulk Packing	94145
Pinnacle™ DB C8 Bulk Packing	94135
Pinnacle™ DB Cyano Bulk Packing	94165

Bulk Silica Materials

Minimum order quantity: 5g

Description	cat.#
Pinnacle II™ Silica Bulk Packing, 3µm	92103
Pinnacle II™ Silica Bulk Packing, 5µm	92105
Pinnacle™ DB Silica Bulk Packing, 5µm	94105

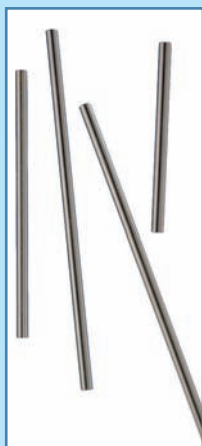
We offer a range of particle sizes, pore sizes, and bonded phases to meet your chromatographic and purification needs. If you don't see what you need in this list, please contact your local representative. We will be happy to discuss your bulk silica needs!

Product Listing

HPLC Column Tubing

Restek tubing is manufactured from fine, chromatographic-grade 316 stainless steel. It is corrosion resistant, ultrasonically cleaned and passivated. We polish the tubing and promise a burr-free cut. You can use this tubing immediately—there is no need for any additional treatment.

Length	ID	OD	qty.	cat.#
30mm	2.1mm	1/4"	ea.	25100
50mm	2.1mm	1/4"	ea.	25101
100mm	2.1mm	1/4"	ea.	25102
150mm	2.1mm	1/4"	ea.	25103
200mm	2.1mm	1/4"	ea.	25104
250mm	2.1mm	1/4"	ea.	25105
30mm	3.2mm	1/4"	ea.	25106
50mm	3.2mm	1/4"	ea.	25107
100mm	3.2mm	1/4"	ea.	25108
150mm	3.2mm	1/4"	ea.	25109
200mm	3.2mm	1/4"	ea.	25110
250mm	3.2mm	1/4"	ea.	25111
30mm	4.6mm	1/4"	ea.	25112
50mm	4.6mm	1/4"	ea.	25113
100mm	4.6mm	1/4"	ea.	25114
150mm	4.6mm	1/4"	ea.	25115
200mm	4.6mm	1/4"	ea.	25116
250mm	4.6mm	1/4"	ea.	25117



1/4-Inch HPLC Frits

Our frits are made from fine, chromatographic-grade 316 stainless steel. We offer sizes to fit most column and particle sizes. Choose the correct pore size based on the particle size of the packing material (see right).



ID	Pore Size	qty.	cat.#
4.6mm	2.0µm	10-pk.	25071
4.6mm	0.5µm	10-pk.	25072
3.2mm	2.0µm	10-pk.	25073
3.2mm	0.5µm	10-pk.	25074
2.1mm	2.0µm	10-pk.	25075
2.1mm	0.5µm	10-pk.	25076

Packing particle size	Use this pore size
3—4µm	0.5µm
5—20µm	2.0µm

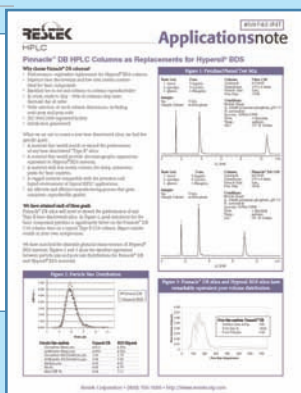
1/4-Inch Column End-Fittings

Description	qty.	cat.#
Column End-Fitting for 4.6mm Column	ea.	25077
Column End-Fitting Flat Bottom	ea.	25078



Pinnacle™ DB HPLC Columns as Replacements for Hypersil® BDS

Pinnacle™ DB base-deactivated silica will match or exceed the performance of any "type B" base-deactivated silica, and is an equivalent, effective, reliable replacement for Hypersil® BDS material. This 8-page note includes many examples of the highly equivalent performance of Pinnacle™ DB and Hypersil® BDS columns. Request lit. cat.# 59742.



2004 Restek Chromatography Products

Our 2004 catalog features innovative, high-value HPLC and GC columns and accessories. Request lit. cat.# 59854-INT.



Lit. Cat.# 59901-INT

