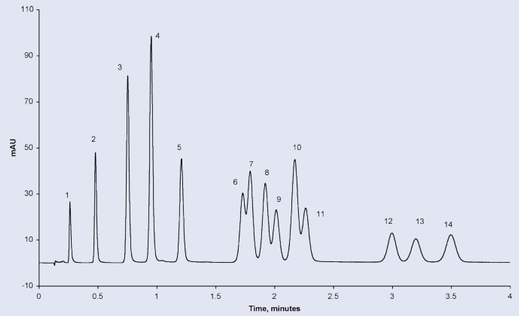
Separation of Explosives

PEAKS

1. HMX
2. RDX
3. 1,3,5-trinitrobenzene
4. 1,3-dinitrobenzene
5. tetryl
6. nitrobenzene
7. 2,4,6-trinitrotoluene
8. 4-amino-2,6-dinitrotoluene
9. 2-amino-4,6-dinitrotoluene
10. 2,6-dinitrotoluene
11. 2,4-dinitrotoluene
12. 2-nitrotoluene
13. 4-nitrotoluene
14. 3-nitrotoluene



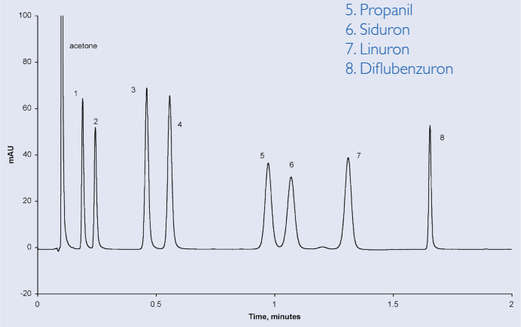
Column: 2.1 x 50 mm HALO C18 (Fused-Core Particles)
Mobile Phase: 27% Methanol / 73% Water Temperature: 40 °C
Flowrate: 0.85 ml/min Detector: UV @ 254 nm
Pressure: 310 bar Instrument: Agilent 1100

Separation of Herbicides

Time	%B
0.00	35%
0.50	35%
2.00	65%

PEAKS

1. Tebuthiron
2. Thiazuron
3. Fluometuron
4. Diuron
5. Propanil
6. Siduron
7. Linuron
8. Diflubenzuron



Column: 2.1 x 50 mm HALO C18 (Fused-Core Particles)
Flowrate: 1.0 ml/min
Detector: UV @ 245 nm Temperature: Ambient
Instrument: Agilent 1100 Pressure: 370 bar (initial), 295 bar (final)
Mobile Phase: gradient (A= H2O, B= ACN)

Conclusion

- Fused-Core particle technology has led to the production of HPLC columns with plate numbers, N, much higher than what would normally be expected from the packing particle size. In fact, columns packed with 2.7 µm Fused-Core particles rival the efficiency and separation speed of columns packed with sub-2 µm totally porous particles, but operate at a small fraction of the back pressure.
- The short, 0.5 µm, diffusion path of the Fused-Core particle's porous "halo" provides superior mass transfer kinetics and better performance at high mobile phase velocities. This characteristic is especially useful when separating larger size molecules.
- The narrow particle size distribution and high density of Fused-Core particles facilitate the packing of columns that are extremely rugged. Plus, columns packed with 2.7 µm Fused-Core particles can use 2 µm porosity inlet frits, the same porosity frit typically used on columns packed with 5 µm particles. This makes these columns less susceptible to the plugging problems that are so evident with other fast HPLC columns, especially sub-2 µm columns, and adds to their reliability and ease of use.

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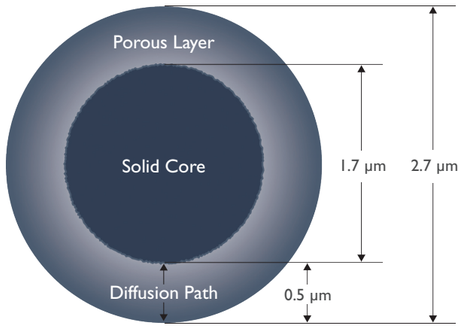
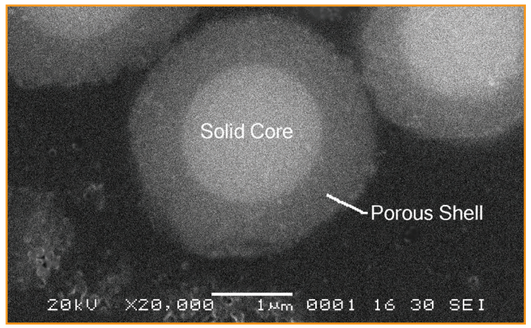
TECHNICAL REPORT

New Fused-Core® Particle Technology for Very Fast HPLC Separations

Fused-Core particle technology was specially developed to facilitate hyper-fast HPLC separations using typical HPLC systems as well as to make faster separations and higher sample throughput more rugged and reliable than what is currently possible with "sub-2 µm" packed columns. As the name implies, Fused-Core particles are manufactured by fusing a porous silica layer onto a solid silica particle. The short diffusion path created by this porous "halo" reduces axial dispersion of solutes and minimizes peak broadening. This advantage becomes very useful when separating larger solute molecules and operating at faster mobile phase velocities.

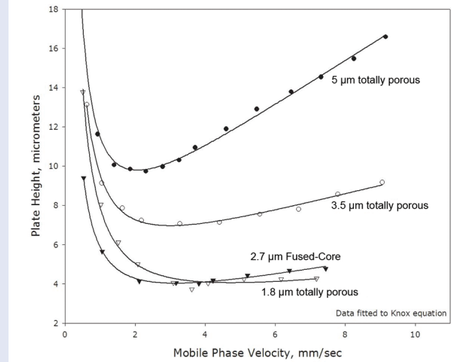
Fused-Core particle





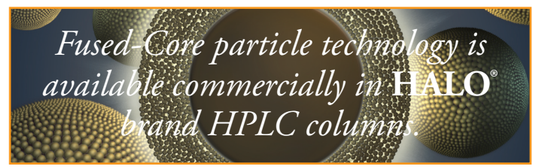
This SEM photograph of a "sliced" Fused-Core particle clearly shows the structure; a solid core surrounded by a porous "halo".

Effect of Particle Size and Type

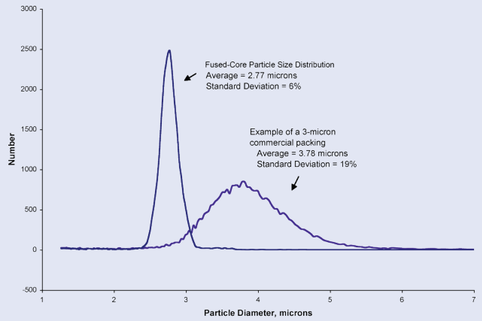


Columns 50 x 4.6 mm
Solute: naphthalene; mobile phase: 60% ACN/40% water, 24 °C

These van Deemter plots for columns packed with different size particles illustrate how efficiency increases (lower plate height) with decreasing particle size. In addition, these plots illustrate how columns packed with the smaller particles can be run at higher mobile phase velocity for faster separations while sacrificing less in efficiency. The column packed with Fused-Core particles generated efficiency much better than what would be expected from their particle size and very similar to that of a 1.8 µm totally porous particle.



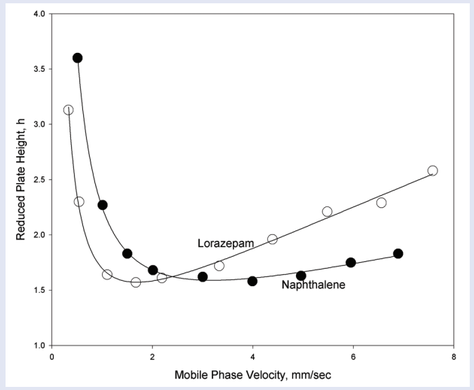
Particle Size Distribution



Average: 2.77 micrometers
Standard deviation: 5.2%

One of the distinguishing characteristics of Fused-Core particles is the unusually narrow size distribution compared to totally porous particles. This plot of particle number versus particle diameter illustrates how much more narrow the Fused-Core particle distribution is compared to typical totally porous particles.

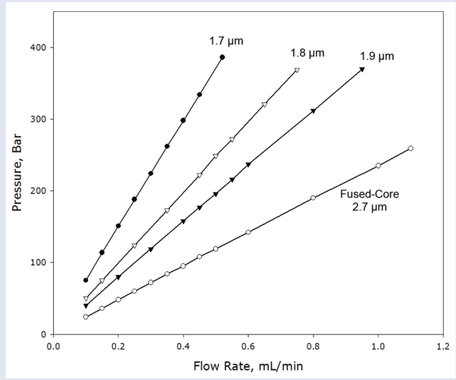
Reduced Plate Height h for Fused-Core Particles



Column: 50 x 4.6 mm, HALO C8; Temperature 24 °C
Naphthalene (MW 128); 60% ACN/40% water
Lorazepam (MW 321): 30% ACN/70% 20 mM phosphate buffer, pH 3.5

The unusually narrow particle size distribution plus the high density of Fused-Core particles, facilitate the packing of columns with remarkably good reduced plate heights, h. It has generally been assumed that a reduced plate height of 2 was the practical limit that could be achieved with packed columns and few commercial columns even achieve h values less than 2.5. The fact that columns packed with Fused-Core particles consistently have h values less than 1.7 particle diameters, as shown in this figure, indicates a breakthrough in packed column performance and explains why columns packed with Fused-Core particles rival the efficiency of columns packed with much smaller particles, i.e., 1.7 µm.

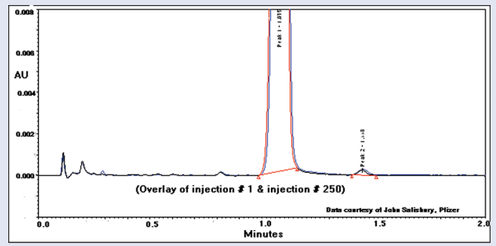
Back Pressure Plots for High-Speed Columns



Back Pressure Plots for High-Speed Columns
Columns: 50 x 2.1mm, C18; Mobile phase: 70% ACN/30% water
Temperature: 24 °C; Agilent 1100

Although columns packed with Fused-Core particles exhibit efficiencies that are far better than what you would expect from their particle size, the back pressure on these columns is exactly what you would expect from a 2.7 µm particle. This figure shows a comparison of back pressure for columns of the same dimensions packed with different size particles. This points out one of the major advantages of columns packed with Fused-Core particles: efficiency that rivals columns packed with sub-2 µm particles but operating pressure that is a small fraction of the sub-2 µm columns. Columns packed with Fused-Core particles operate within the acceptable pressure ranges of most HPLC systems, yet generate the speed and efficiency approaching that of sub-2 µm columns.

Fused-Core Particle Stability Test



Columns: 2.1 x 100 mm HALO C18
Mobile phase: 70% ACN/30% water
Flow rate: 1.6 mL/min
Column temperature: 30 °C
Column back pressure: 12264 psi
Number of injections: 250

Results, peak 1, n = 250

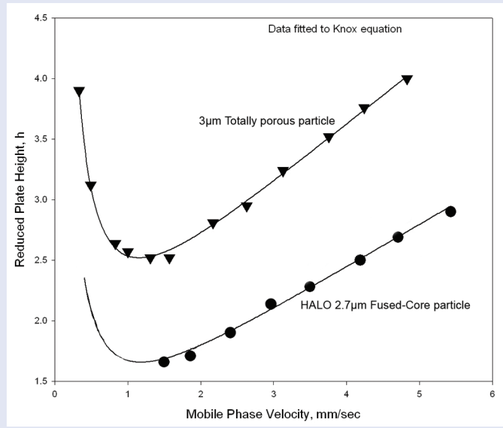
RSD, retention time: 0.06%
RSD, plates: 0.38%
RSD, tailing: 0.53%
RSD, Rs, peaks 1,2: 2.7%

Fast separations run under high flow rate and high pressure conditions can quickly degrade the performance of an HPLC column. However, columns packed with Fused-Core particles exhibit unusual stability under extreme conditions. This is illustrated in this set of comparison chromatograms where the HALO column (packed with Fused-Core particles) showed virtually no loss in performance after 250 sample injections at extremely high flow rate and pressure. Although Fused-Core particle technology was developed for the purpose of producing hyper-fast HPLC columns that could be used with most any standard HPLC system, they certainly are rugged enough to be used at the higher pressures of UPLC systems.

The following chromatograms show how complex mixtures can be separated at high speed using columns packed with Fused-Core particles. Notice that the operating pressure of all these chromatograms are well within the acceptable limits of most HPLC systems.

Although Fused-Core particles were developed for hyper-fast HPLC separations, the high peak capacity of columns packed with these particles make them a really good choice for separating complex mixtures.

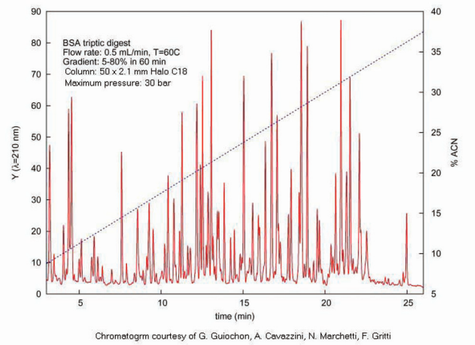
Column Comparison Study with Virginiamycin (MW=574)



Columns: 50 x 4.6 mm; Temperature: 30 °C; Agilent 1100
Mobile phase: 35% ACN/65% potassium phosphate buffer, pH 3.0

The short (0.5 µm) diffusion path offered by Fused-Core particles is particularly advantageous when separating larger size molecules. In this comparison of reduced plate heights for a column packed with 3µm totally porous particles and a column packed with 2.7µm Fused-Core particles (HALO), a solute with a molecular weight of 574 was used. The column packed with Fused-Core particles shows much superior reduced plate heights for this size molecule.

BSA triptic digest



Flow rate: 0.5 mL/min, T=60 °C Gradient: 5-80% in 60 min
Column: 50 x 2.1 mm HALO C18