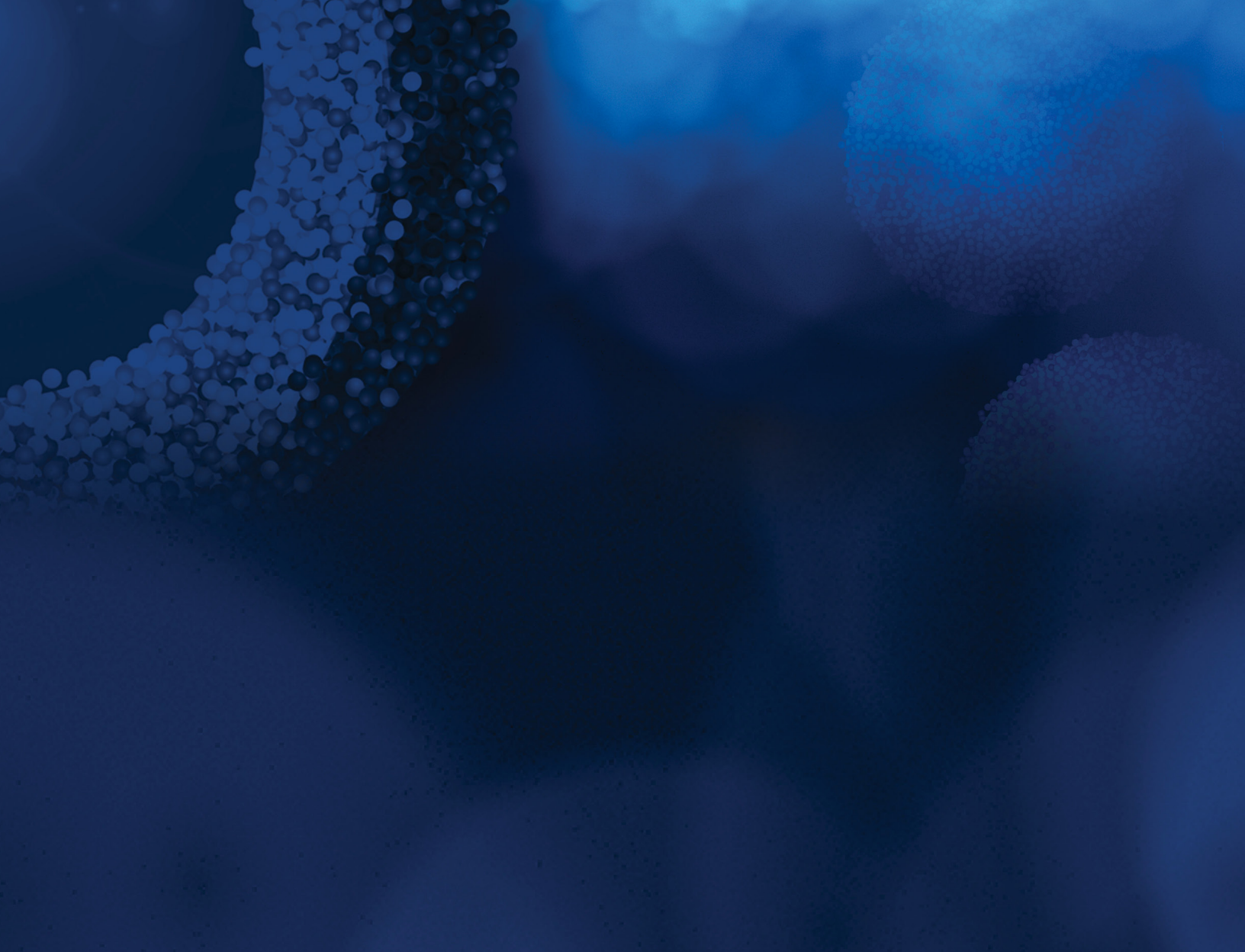
HALO®

PIONEERING FUSED-CORE® TECHNOLOGY SINCE 2005

THE MAKING OF HALO® COLUMNS AND ADVANCED MATERIALS TECHNOLOGY'S COMMITMENT TO QUALITY

谱之道
GO TO CHROM



DISCOVER MORE WITH FUSED-CORE®

Since our founding in 2005, Advanced Materials Technology has been focused on one mission – Improving the presentation of the sample to the detector. Using our novel Fused-Core® particle design, we have challenged conventional wisdom and engineered innovative solutions for the separations community. Our quality procedures and practices are integrated into every HALO® column delivered ensuring your success – every time.

AMT QUALITY POLICY

AMT is committed to providing world-class innovative products that uniquely fill the growing needs of small molecule and large molecule separation scientists. We take pride in delivering products that exceed customers' expectations on quality and delivery time and collaborate to break down any barriers that would prevent an exceptional customer experience. We continually strive to improve our organization to stay focused on safety, quality, and cost.

We are an ISO 9001:2015 Certified Company.

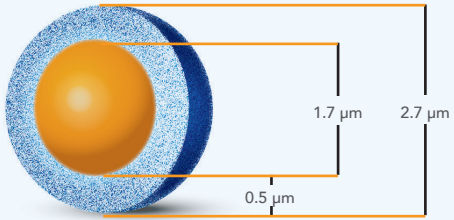
We pledge to have dynamic leadership promoting a culture of excellence embedded in every employee.

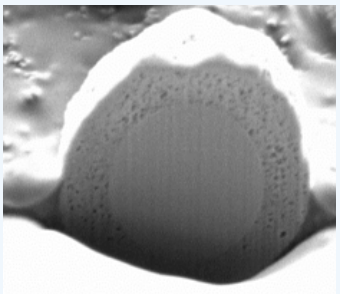
INTRODUCTION

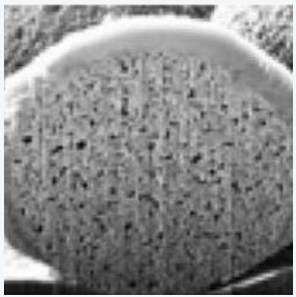
WHAT IS FUSED-CORE® TECHNOLOGY?

Nearly sixty years ago, Horvath and Lipsky published their groundbreaking paper in which they described their pellicular particles which were made of glass particles coated with a thin layer of ion-exchange resin. This paper inspired Jack Kirkland, then working for the DuPont Company, to develop the first superficially porous silica particles with solid cores and a porous crust (30 µm Zipax®, 1969). The introduction of superficially porous particles enabled liquid chromatography to begin its development into the major analytical tool it has become. This type of particle was soon displaced (early 1970s) by the development of small-particle, fully porous silica particles which dominated HPLC column technology thereafter. Superficially porous particles were reintroduced in 2006 as a major advance in HPLC particle technology when Kirkland, Langlois, and DeStefano, at Advanced Materials Technology, commercialized the first sub-3µm Fused-Core® silica particle under the brand name HALO®. This particle consists of a solid silica core surrounded by a thin porous shell that is heat-sintered to the core, hence the name Fused-Core®. Fused-Core® columns can be operated on both HPLC and UHPLC systems with superior performance.

HALO® 2.7 µm







ADVANTAGES OF FUSED-CORE® TECHNOLOGY

The advantage of superficially porous particles (SPPs) over fully porous particles (FPPs) is best shown by comparing scanning electron micrographs of cross-sections of the particles in the figure on previous page. The diffusion path for sample molecules into and out of the particles isshorter for the SPP due to the presence of the impermeable solid core leading to faster mass transfer. The classical van Deemter characterizations of FPP columns and a SPP column in the figure to the right demonstrates the effect of mobile phase velocity on column performance. Columnperformance is determined by measuring the width of peaks eluting from the column. This measurement of width can be converted into the number of theoretical plates (N).

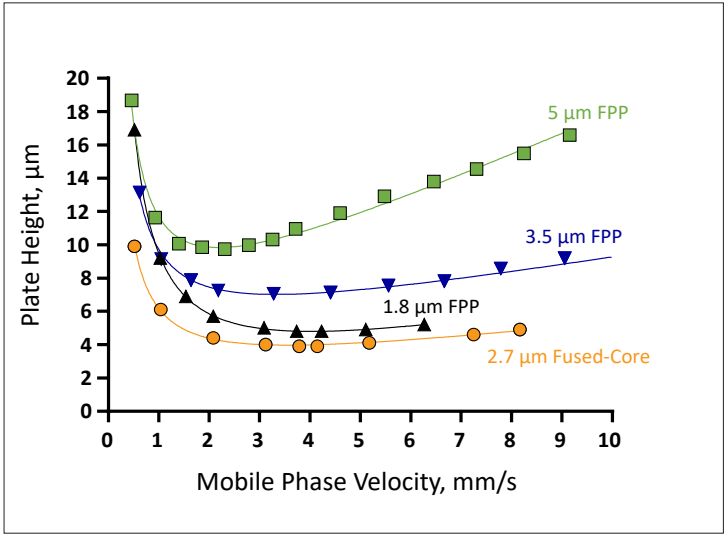
$$N = 5.54 (t_R/w_{1/2})^2$$

where t_R = peak retention time, $w_{1/2}$ = peak width at half height

The greater the number of theoretical plates the more resolving power in the column. The plate count in columns can then be converted to the Height Equivalent to a Theoretical Plate (H) by dividing the column length (in microns) by the number of theoretical plates. The Plate Height (H) is comprised of three terms that contribute to the van Deemter curve:

$$H = A + B/\mu + C\mu$$

where the A term is affected by how well the column is packed (eddy diffusion term), the B term is a measure of longitudinal diffusion and is positively affected by the presence of the solid core (25-30% smaller B-term for SPP), and C is the mass transfer term that is smaller for SPP resulting in a flatter curve as mobile phase velocity (μ) is increased. The 2.7 μm Fused-Core® column has a clear advantage as depicted in the van Deemter plot, outperforming even the sub-2-μm FPP column.

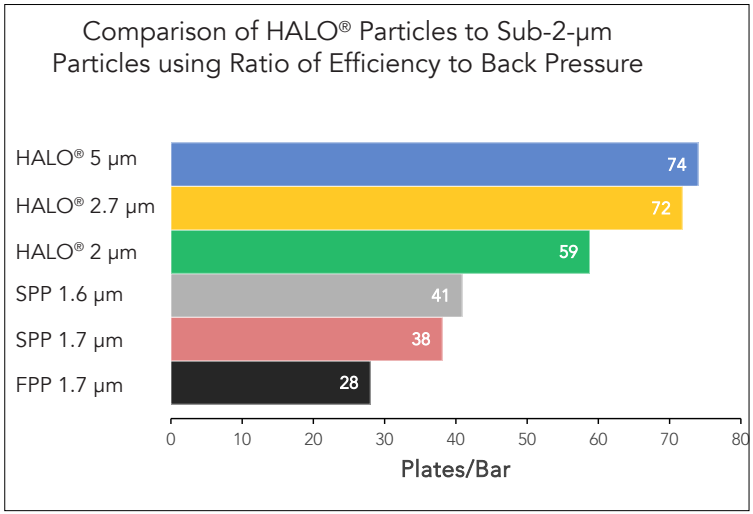


OTHER ADVANTAGES OF HALO® COLUMN TECHNOLOGY

PLATES/BAR, KINETIC PLOTS, PEAK CAPACITY

Another way to compare column performance between different types of columns is to measure the ratio of the number of theoretical plates and required back pressure (in bar) that are obtained under a fixed set of operating conditions (plates/bar). Example performance measurements for three HALO® and three competitor 2.1 x 50 mm SPP and FPP columns, run at 0.5 mL/min with 50/50 acetonitrile/water at 35°C are shown in the figure below.

All of the HALO® columns provide more theoretical plates per unit of pressure than any of the competitor sub-2-micron SPP and FPP columns.

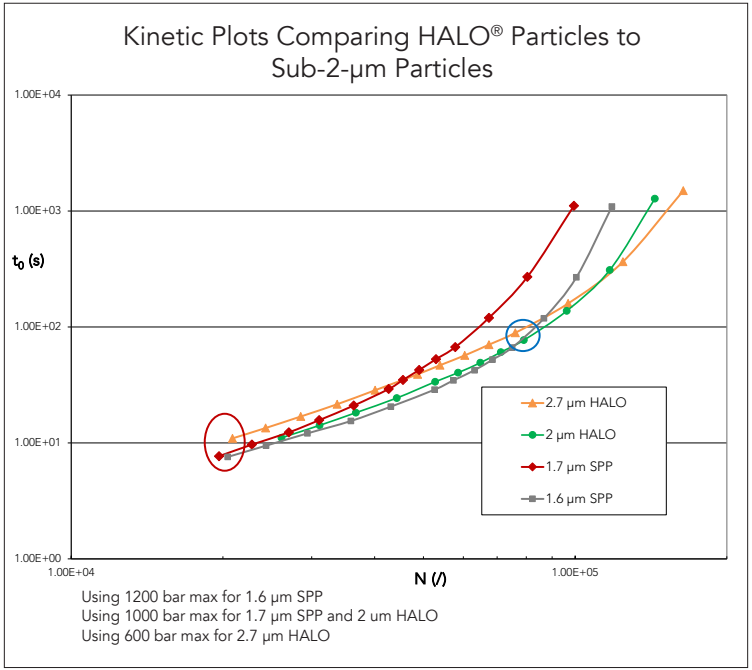


KINETIC PLOTS – HOLD UP TIME AND PRESSURE

Kinetic plots can aid the analyst to compare different columns packed with different particles at different column lengths by plotting the hold-up time as a function of the column efficiency (J. Sep. Sci. 2011, 34, 877–887). Practical decisions on the final dimensions are often limited to lengths between 3 – 15 cm. Generally, the shortest columns are ideal for high throughput analyses, and the longer length columns ideal for separations that prioritize separation efficiency and can afford longer analysis times/ larger hold up volume. The kinetic plot illustrates the same columns depicted in the previous figure (both axes in log scale). These plots can be interpreted a number of ways and are critical to demonstrate key factors important for method development.

One case scenario may be to aid particle size selection when developing an assay for speed – hence a small hold up time with the shortest column format must be selected. Looking at the graph (p. 6) in this specific region highlighted in red - The HALO® 2.7 µm particle in a shorter column length is a sensible column choice as it does not require the dedicated UHPLC system.

Another case scenario highlighted in blue – are for separations that can afford relatively longer analysis times: the HALO® 2 µm column in a longer length column format affords more plates at a longer length; the HALO® 2.7 µm column at a smaller column length is a sensible option at 600 bar.

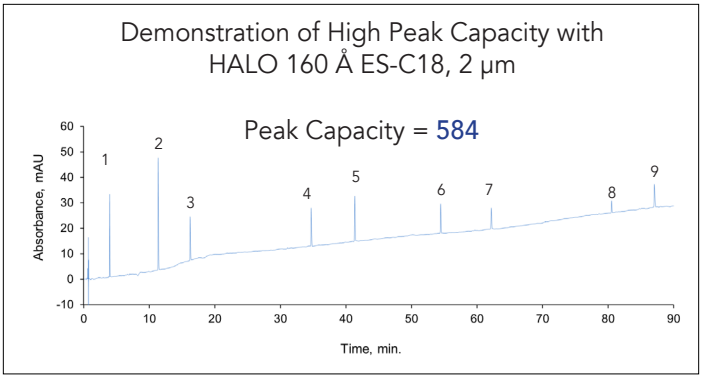


PEAK CAPACITY

Peak capacity is an important metric used to demonstrate the resolution power of the final separation strategy - tightly associated to the column(s) and how it is operated in the final method developed. It is calculated from the separation's chromatogram and the following equation:

$$Peak\ capacity = \frac{t_{R\ (last\ eluting\ peak)} - t_{R\ (first\ eluting\ peak)}}{w_{average}}$$

This number is particularly useful to gauge the separation performance of complex separations that utilize unidimensional LC (1DLC) and/or two-dimensional LC (2DLC) approaches. The following figure demonstrates the high peak capacity enabled by a HALO® column for peptide analysis.

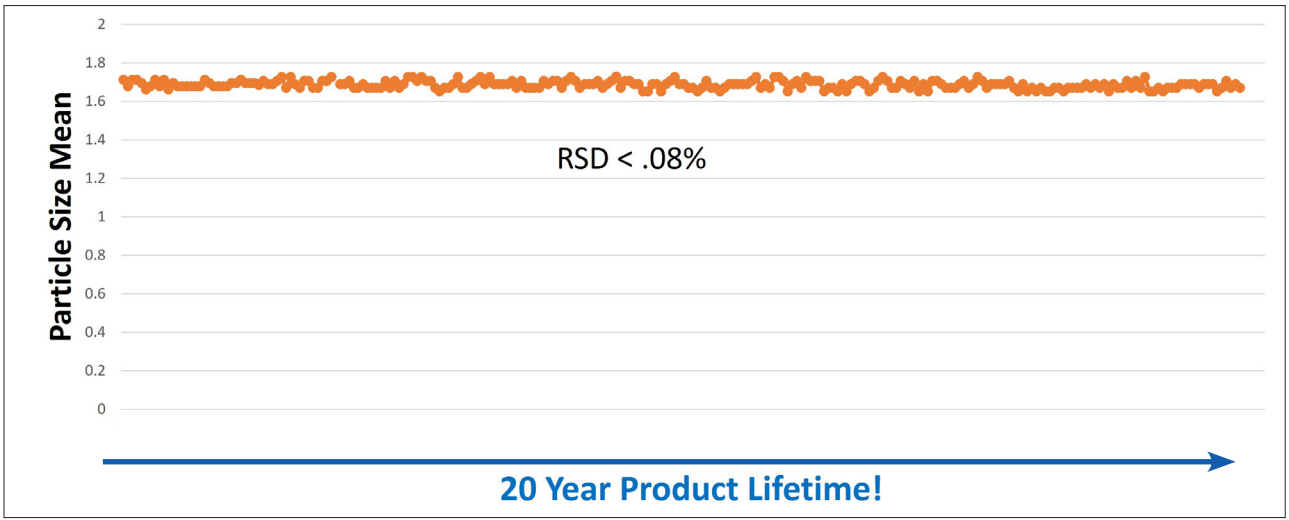


THE MAKING OF THE HALO® PARTICLE

QUALITY BY DESIGN

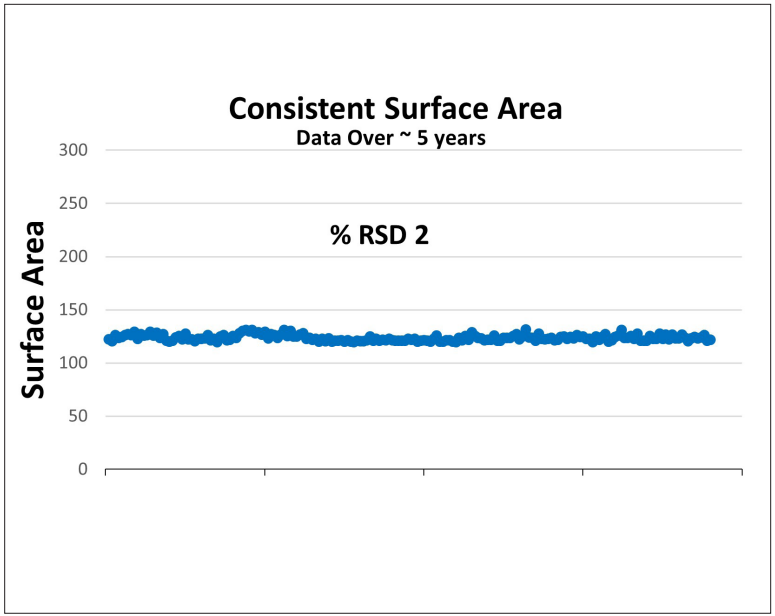
Along with the innovation that is demonstrated by HALO® products, the quality of HALO® is unsurpassed, which stems from AMT's complete control over the entire manufacturing process – from the starting high quality raw materials to the final packed column with controls and measurements built into every step. The creation of a HALO® column begins with the manufacturing of the solid silica core, which is produced using high quality raw materials. As the particle's foundation, it is essential to closely monitor the manufacture of the core. This first step of the process is characterized by more than 45 different in-process controls and 4 separate QC/QA tests. Tight tolerances are in place to guarantee that the core is made reproducibly and consistently every time. The figure demonstrates how little variation there has been in the 1.7 µm core over the span of the product lifetime. This result contributes to our commitment for customers' long-term repeatability of separations utilizing AMT powered HPLC columns.

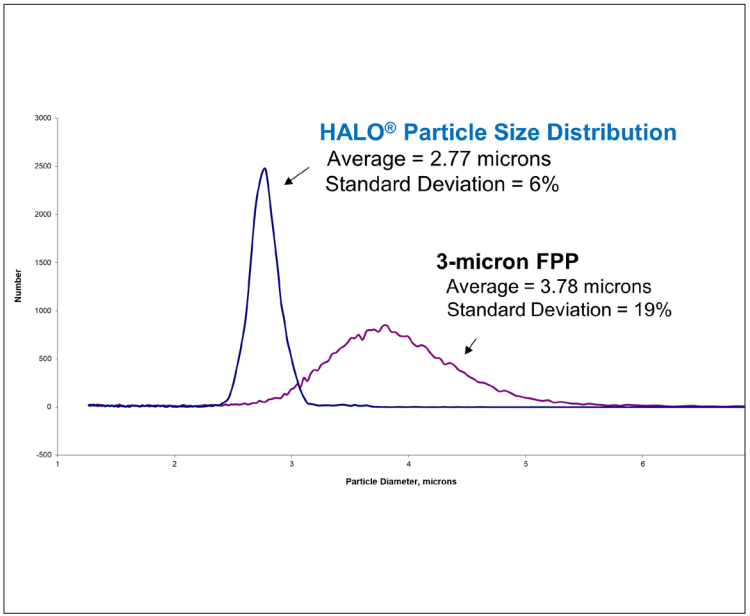
CORE CONSISTENCY



THE SHELL

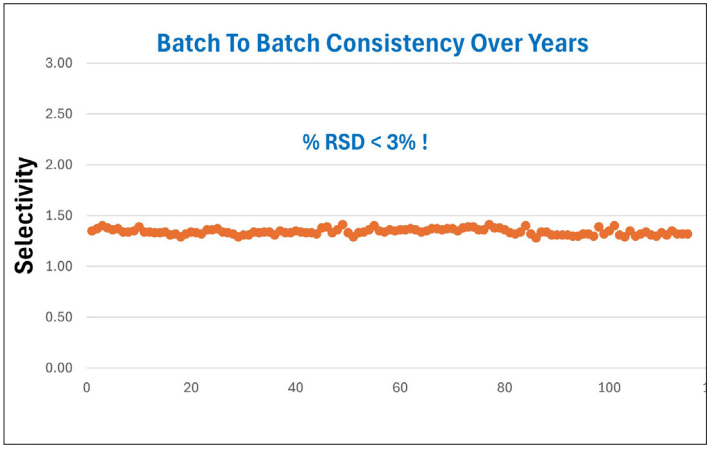
Next, the porous silica shell is added to the core. Here AMT has complete control over the pore size and shell thickness thus leading to a consistent and reliable surface area as demonstrated in the figure. This consistency in surface area from lot to lot and year after year yields excellent chromatographic reproducibility. This step consists of more than 100 in-process controls and 7 rigorous QC/QA tests proving how diligently the thickness of the porous shell is controlled and monitored. The pore size is obtained by careful selection of the nanoparticles that are used in the process. The size of the analytes of interest for the separation dictate the pore size with larger pores designed for biomolecules and smaller pores designed for small molecules. AMT has 5 different pore size products: 90 Å, 120 Å, 160 Å, 400 Å, and 1000 Å. AMT silica SPP manufacturing results in a very narrow particle size distribution that is a hallmark of HALO® column quality. This narrow particle size distribution contributes to high efficiency columns with stable chromatographic beds. An example of the particle size distribution for 2.7 µm HALO® particles compared to 3 µm fully porous particles is shown in the figure. The AMT SPP process has a standard deviation more than 3 times lower than the FPP manufacturing process.



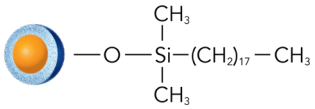


SURFACE OPTIMIZATION

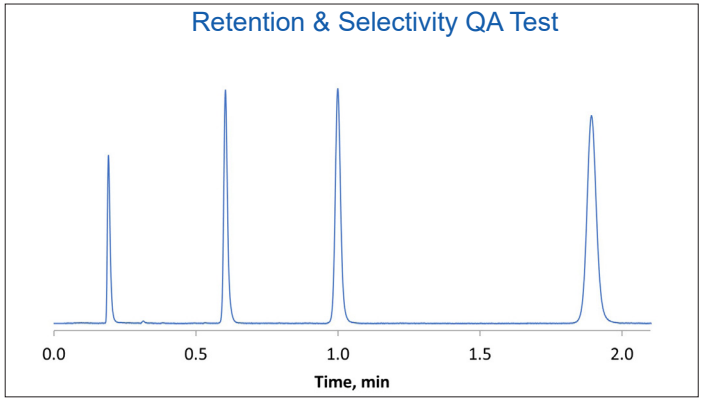
Next AMT uses a proprietary method to create uniform and consistent surface silanols on the HALO® particles. Once this step is complete the particles are ready for bonding or can be used as non-derivatized or bare silica. AMT uses a challenging chromatographic test at pH 7 with a basic analyte to confirm the uniformity of the surface silanols. The figure illustrates lot to lot consistency for several years' worth of HALO® C18 batches.



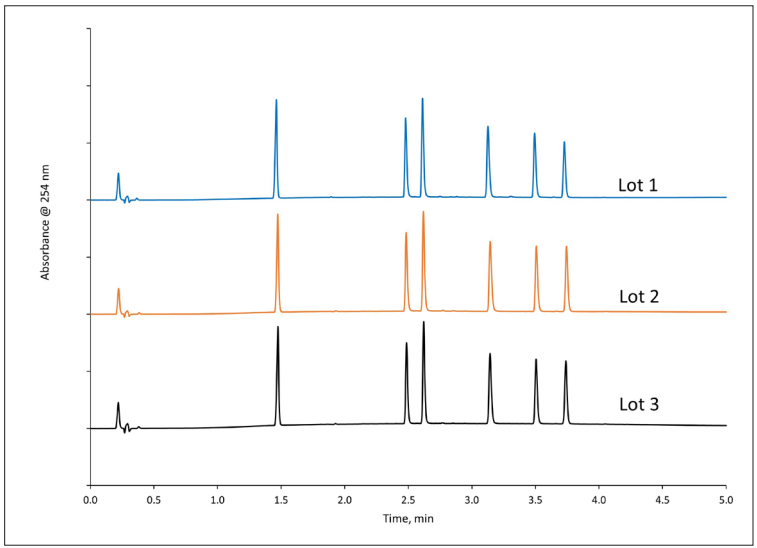
BONDING

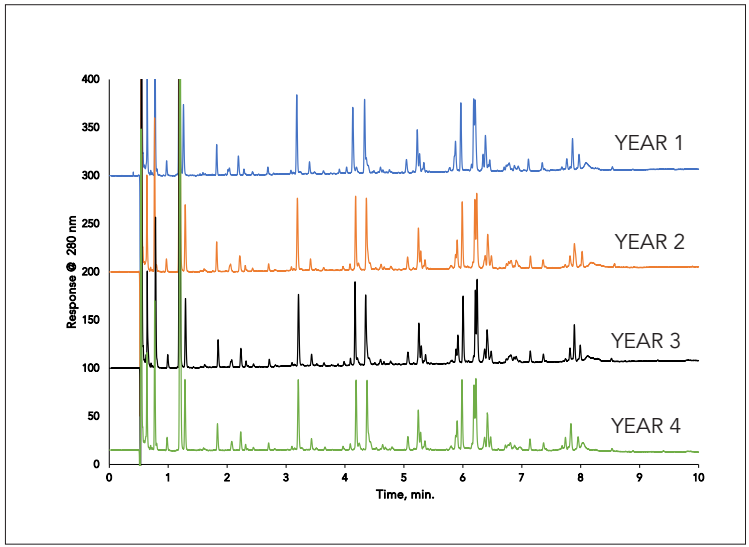


The finished HALO® particles are then ready for derivatization with the appropriate stationary phase for our small molecule, biomolecule and our application-specific columns. The bonding process consists of 25 in-process controls and 20 QC/QA tests. The figure shows an example QA test that is used for evaluating HALO® C18. This test is run to qualify that the reaction to derivatize the surface with the stationary phase has been successfully completed as demonstrated by tight specifications for retention and selectivity.



The next figure below shows the lot-to-lot reproducibility for 3 different lots of HALO® Elevate C18, 2.7 µm. A mix of 1 neutral compound and 5 different bases was run using a gradient of pH 10.7 ammonium hydroxide and acetonitrile with 2.1 x 50 mm columns. The average %RSD across the retention times for all of the compounds was 0.3% demonstrating excellent lot-to-lot reproducibility.



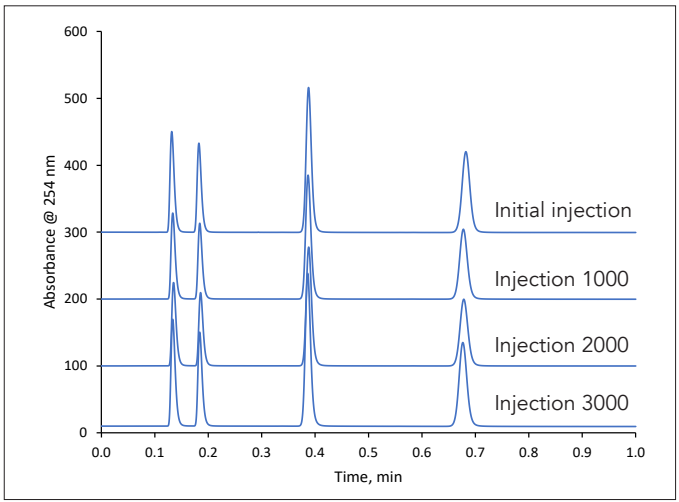


Excellent lot-to-lot reproducibility is shown spanning a period of 4 years using the HALO 160 Å ES-C18 stationary phase and a sample of adalimumab tryptic digest. Lot performance such as this is crucial for customers developing methods that will be used today, tomorrow, and years from now.

STABILITY

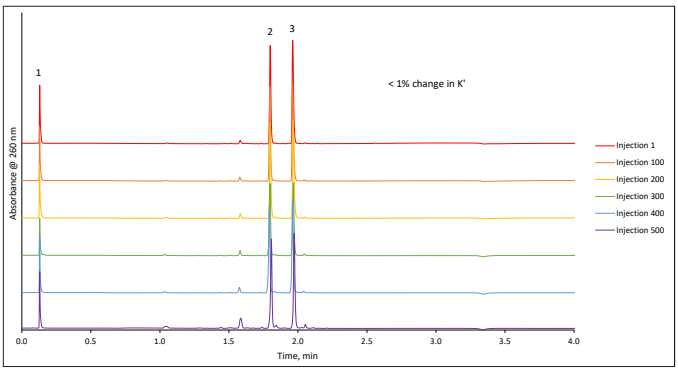
The longevity of our superficially porous technology is showcased at low pH and high temperature for 3000 injections (approximately 23,000 column volumes); the results illustrate no significant changes.

HALO® C18 Stability at Low pH and High Temperature



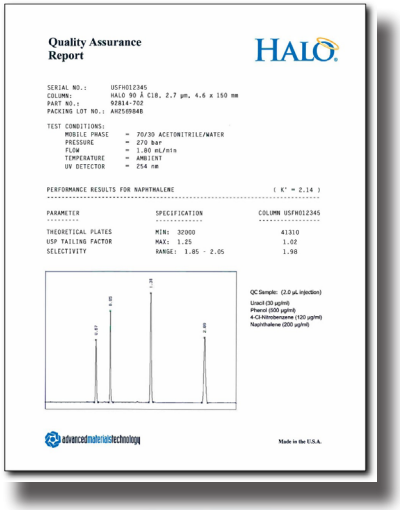
One of the newest stationary phases is Elevate C18, which is designed for operation at high pH and high temperature. The stability of Elevate C18 was tested at a challenging environment of high pH 10 and 60 °C. The column demonstrated superior performance (no loss in retention and no changes in peak shape and back pressure) for over 500 injections (>20,000 column volumes) of mobile phase consumption.

HALO® Elevate C18 Stability at High pH and High Temperature



QA/QC TESTING

After the HALO® particles are bonded and pass their qualification tests, they are ready to be loaded into columns. Using an automated process, columns are loaded to eliminate any variation in packing quality. This step includes 20 in-process controls and 6 QC/QA tests. Every column is tested and must pass tight criteria for selectivity, tailing factor, and efficiency before it is qualified as acceptable. HALO® columns have complete traceability back to the starting core lot, coated final particle lot, and bonded lot. This traceability enables excellent customer support because archive material is available for potential troubleshooting investigations.


Quality Assurance Report

HALO®

SERIAL NO.: USFH012345
COLUMN: HALO 90 Å C18, 2.7 µm, 4.6 x 150 mm
PART NO.: 92814-702
PACKING LOT NO.: AH058048

TEST CONDITIONS:
MOBILE PHASE = 70/30 ACETONITRILE/WATER
PRESSURE = 270 bar
FLOW = 1.80 mL/min
TEMPERATURE = AMBIENT
UV DETECTOR = 254 nm

PERFORMANCE RESULTS FOR NAPHTHALENE (K' = 2.14)

PARAMETER	SPECIFICATION	COLUMN USFH012345
THEORETICAL PLATES	MIN: 32000	41310
USP TAILING FACTOR	MAX: 1.25	1.02
SELECTIVITY	RANGE: 1.85 - 2.05	1.98

QC Sample: (2.0 µL injection)
Uracil (30 µg/ml)
Phenol (600 µg/ml)
4-Cl-Nitrobenzene (120 µg/ml)
Naphthalene (200 µg/ml)

advancedmaterialstechnology

Made in the U.S.A.

OUR JOURNEY ENDS WITH YOU!

The final step in the process of manufacturing a column is packaging. HALO® columns are packaged with their quality assurance reports and Care and Use documentation. All packaging materials are 100% recyclable.

HALO® columns are sold globally and ship quickly. AMT meets our 24 hour turn around time KPI 99.05% of the time!

INNOVATION OF FUSED-CORE®

2017
1000 Å Protein
First 1000 Å pore size SPP for high resolution of large protein complexes

2013
BIOCLASS Line Introduced
Protein, Peptide and Glycan solutions for biomolecule separations

2006
Original 2.7 µm SPP
Changed the perception of what is required for high efficiency separations

2005
AMT Founded
Wilmington DE, USA

INNOVATION CONTINUES

We continue our mission to build a better path to separations

2024

Innovative Particle Surface Modifications - ELEVATE, PCS

New phase chemistries launched for stability within very low to very high pH environments and with charged surfaces

2022

1.5 MM ID Column

50% less solvent usage than 2.1 mm ID and up to 2x signal intensity

2020

HALO® ENVIROCLASS

Targeted applicated solutions for PAH and PFAS analysis

COMPANY PROFILE

Advanced Materials Technology founded in 2005 has been focused on one mission – Improving the presentation of the sample to the detector. Using our novel Fused-Core® particle design, we have challenged conventional wisdom and engineered innovative solutions for the separations community.

All company operations and functions are proudly located in Wilmington, Delaware, USA.

AMT invites a company culture of diversity, respect and pride in delivering quality products. We embrace ISO 9001 standards in our work systems and daily work. We pledge to have a dynamic leadership team which promotes our culture of excellence embedded in every employee.

Joseph J. DeStefano B.S., M.S., Ph.D.

Chairman of the Board, CSO, Co-Founder

Co-founder of AMT with over 50 years of experience in silica product development, research supervision, and business management with the DuPont Company, Rockland Technologies, Hewlett Packard where he was integral in the business development of the well-known Zorbax stationary phase, and Agilent Technologies. Previously, President and co-founder of Rockland Technologies.

Dr. DeStefano holds a B.S. degree in Chemistry from University of Connecticut and Ph.D. in Analytical Chemistry from University of Delaware. At AMT he is involved in strategic planning of new products, applications development and business relationships.

Timothy J. Langlois B.S.

President, Co-Founder

Active for 25 years in silica technology development, quality assurance, and manufacturing. Prior experience as a product engineer at Rodel (now DuPont Microelectronics). Later held R&D, operations and engineering positions at Hewlett-Packard (now Agilent Technologies). Mr. Langlois holds a B.S. degree in Chemical Engineering from Lehigh University.

Mr. Langlois was involved in the start-up of AMT and instrumental in the development of HALO®. As President he is involved in strategic business decisions for AMT including R&D, operations, business development and sales initiatives.

Dr. Joseph "Jack" Kirkland

Principal Scientist, Founding Vice President, R&D

Regarded as a founding father of HPLC Jack is highly renowned for his contributions in the field of chromatography and pioneering superficially porous particle technology with >160 publications, 32 patents and 8 books. Together with Lloyd Snyder, he educated more than 5000 people in the principles and practice of HPLC by means of the first American Chemical Society short course and was a major contributor to the hydrophobic subtraction model of reversed phase column selectivity used widely today. His research interests involved HPLC method development, field flow fractionation, silica chemistry and silane bonding reactions (i.e., novel HPLC columns).

His long list of awards is too long to include, but is highlighted by the 1972 American Chemical Society Award in Chromatography, the 1982 Torbern Bergman Medal in Analytical Chemistry from the Swedish Chemical Society, DuPont's Lavoisier Medal in 1997 (DuPont's highest technical award), the the A. J. P. Martin Chromatography Award Medal in 1997, and the first Uwe Neue Award in 2013 for achievements by an industrial scientist, an honorary D.Sc. degree by Emory University in 1974 and the 2015 LCGC Lifetime Achievement in Chromatography Award.

ADDITIONAL TECHNICAL RESOURCES

Application notes, guidebooks, white papers, and technical reports can be found on our website at:

www.halocolumns.cn


谱之道
GO TO CHROM

HALO®

谱之道

GO TO CHROM

manufactured by:

advancedmaterialstechnology

halocolumns.cn

AMT25_QB_Rev_0