THE NEW HALO® 1.5



**NEW DIMENSION • INCREASED SENSITIVITY • SOLVENT SAVINGS** 

TAKING SEPARATIONS TO A NEW DIMENSION

"I want more **sensitivity** with my conventional UHPLC system..."

"I need an **easy to use** microflow solution..."

"How can I get **more performance** from my LC and LCMS systems?"

> "We have a goal to reduce solvent consumption..."

**INNOVATION YOU CAN TRUST - PERFORMANCE YOU CAN RELY ON** 

## THE NEW HALO<sup>®</sup> 1.5 – DEFINING A NEW DIMENSION IN CHROMATOGRAPHY

The adoption of UHPLC instrumentation pushed LC separations to a new level of speed and resolution. In the years since, chromatographers continue to push separation limits in response to increasing challenges. How can I improve my separation in a faster time, or with less solvent consumption, increased sensitivity, or all of the above? While the specific drivers may be different, the overall goal is the same – 'I need more, but without sacrificing anything'.

In 2006 Advanced Materials Technology broke from convention by introducing Fused-Core® particle technology to the market. These little HALO® particles delivered high-speed, high-performance separations without the consequence of high back pressures allowing scientists to adapt their HPLC systems to rival the UHPLC systems.

Now in 2022 AMT breaks new ground again with a 1.5 mm internal diameter HALO<sup>®</sup> column to push the boundaries of adopted UHPLC systems. Founded on all of the benefits of Fused-Core<sup>®</sup> particles, the HALO<sup>®</sup> 1.5 delivers increased sensitivity and reduced solvent consumption, allowing scientists to experience the benefits of capillary columns without the pains of specialized microflow systems.



### **DELIVERING MORE PERFORMANCE**

More Sensitivity from conventional UHPLC Systems

 $\checkmark$ 

Higher Ionization Efficiencies from LCMS systems

Reduced Solvent Consumption compared to 2.1 mm ID columns

Easy to Implement microflow solution

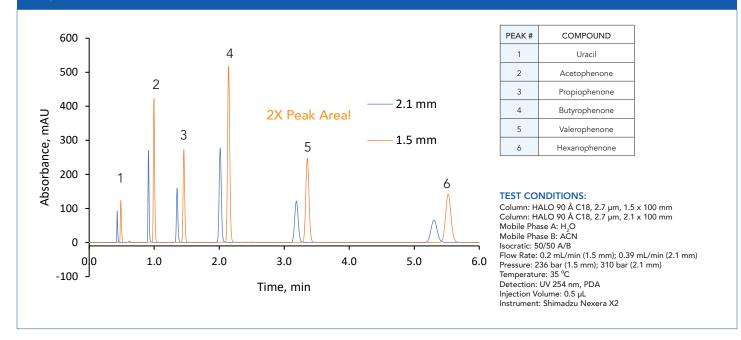
## **MORE SENSITIVITY**

## "I want more sensitivity with my conventional UHPLC system"

Through newly designed specialized manufactured fluidic hardware, the new HALO<sup>®</sup> 1.5 delivers increased signal response in comparison to 2.1 mm ID columns and demonstrates higher efficiencies compared to 1 mm ID columns on optimized UHPLC systems.

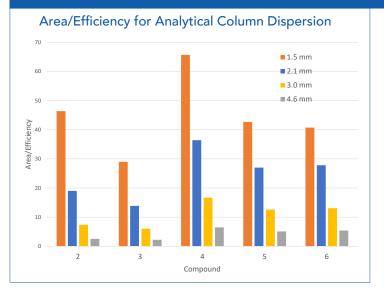
### ISOCRATIC SEPARATION OF ALKYLPHENONES SHOWS AVERAGE OF 2X GREATER AREA USING 1.5 MM COLUMN

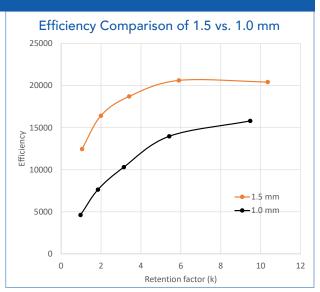
Using a 1.5 mm ID column compared to a 2.1 mm ID column, the peak areas are doubled and the sensitivity is improved using the same injection volume on both columns.



### DEMONSTRATION OF IMPROVED EFFICIENCY WITH HALO® 1.5

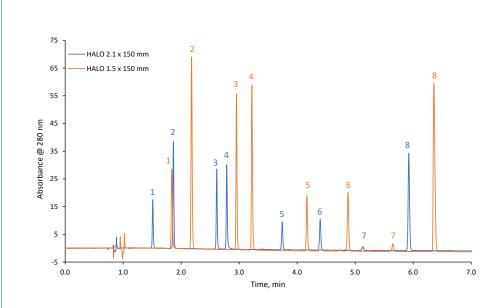
A mixture of alkylphenones was run on 1.0, 1.5, 2.1, 3.0, and 4.6 mm ID columns. The area/efficiency of the 1.5 mm ID column outperforms all of the analytical ID columns. While the 1.0 mm ID should perform better than the 1.5 mm ID, even on an optimized UHPLC system the dispersion is too great for a 1.0 mm ID column, thus causing lower efficiencies at all retention factors.





### GRADIENT SEPARATION OF OTC COUGH AND COLD MEDICINES

With extracolumn dispersion minimized, the 1.5 mm ID column shows taller peaks compared to 2.1 mm ID column providing greatest benefit for minor components.



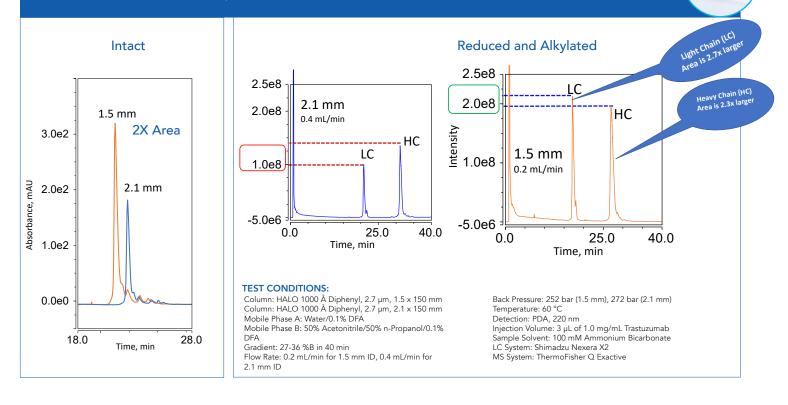
PEAK # COMPOUND 1 Phenylephrine 2 Acetaminophen Caffeine 3 4 Doxylamine 5 Guiafenesin 6 Aspirin 7 Salicylic Acid 8 Dextromethorphan

#### **TEST CONDITIONS:**

Mobile Phase A: Water/0.15% TFA Mobile Phase B: ACN/0.1% TFA Gradient: 5-50 %B in 8 min Flow Rate: 0.2 mL/min for 1.5 mm 0.4 mL/min for 2.1 mm Pressure: 425 bar/1.5 mm 470 bar/2.1 mm Temperature: 35 °C Injection Volume: 0.5 µL Detection: UV 280 nm, PDA Instrument: Shimadzu Nexera X2

### 1.5 MM ID BENEFITS FOR INTACT AND REDUCED AND ALKYLATED MAB CHARACTERIZATION

The HALO 1000 Å Diphenyl in 1.5 mm ID shows double the area for intact trastuzumab compared to the same separation run on a 2.1 mm ID HALO 1000 Å Diphenyl column. Greater than double the area is observed when used for the separation of reduced and alkylated trastuzumab using MS detection.





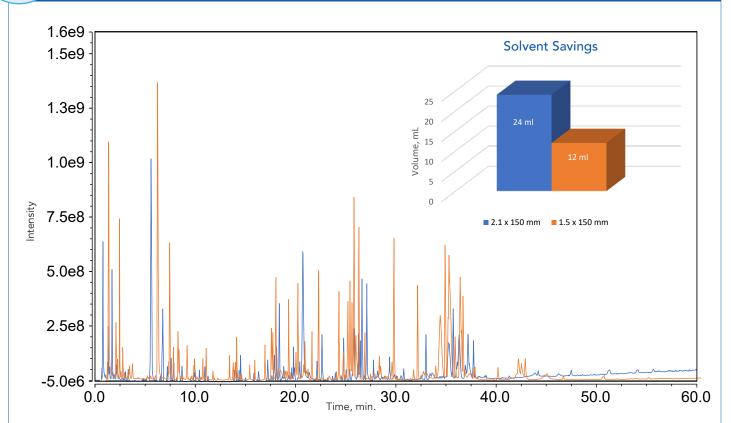
## **SOLVENT REDUCTION**

The costs associated with solvents continue to grow from both a purchase price perspective and price of waste removal. In step with efforts to decrease the amount of environmental impact generated from hazardous chemicals, companies are seeking solutions to go greener. A decrease in column ID is

### PEPTIDE MAP SOLVENT SAVINGS USING HALO 160 Å ES-C18 IN 1.5 MM ID



When a 1.5 mm ID column is used in comparison to a 2.1 mm ID column for a peptide map of trastuzumab, 50% of the solvent is saved since the 1.5 mm ID column is run at 0.2 mL/min compared to 0.4 mL/min for the 2.1 mm ID column. For a 60 minute analysis, only 12 mL of solvent is used vs. 24 mL of solvent for the 2.1 mm ID column. Not only is solvent saved, but the cost of waste disposal is also reduced.



### **TEST CONDITIONS:**

Column: HALO 160 Å ES-C18, 2.7 μm, 1.5 x 150 mm Column: HALO 160 Å ES-C18, 2.7 μm, 2.1 x 150 mm Mobile Phase A: Water/0.1% DFA B: Acetonitrile/0.1% DFA Gradient: 2-50 %B in 60 min Flow Rate: 0.2 mL/min for 1.5 mm ID 0.4 mL/min for 2.1 mm ID Back Pressure: 310 bar (1.5 mm) 444 bar (2.1 mm) Temperature: 60 °C Detection: ESI + Injection Volume: 2 µL of 1.25 mg/mL Trastuzumab tryptic digest Sample Solvent: 1.5 M Guanidine HCI/0.5% Formic Acid LC System: Shimadzu Nexera X2 MS System: ThermoFisher Q Exactive

#### **MS CONDITIONS:**

Spray Voltage (kV): 3.8 Capillary temperature: 320 °C Sheath gas: 35 Aux gas: 10 RF lens: 50

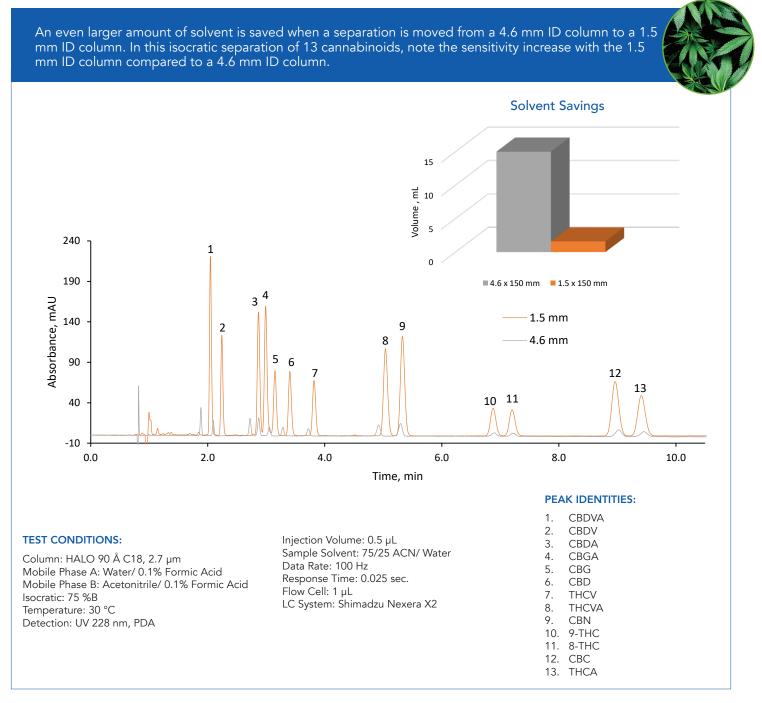


## "We have a goal to reduce solvent consumption"

optimized with lower flow rates resulting in an overall solvent savings. Moving to the HALO<sup>®</sup> 1.5 helps analysts and companies reduce their solvent consumption.

An added benefit using the HALO® 1.5 is increased sensitivity without sacrificing speed!

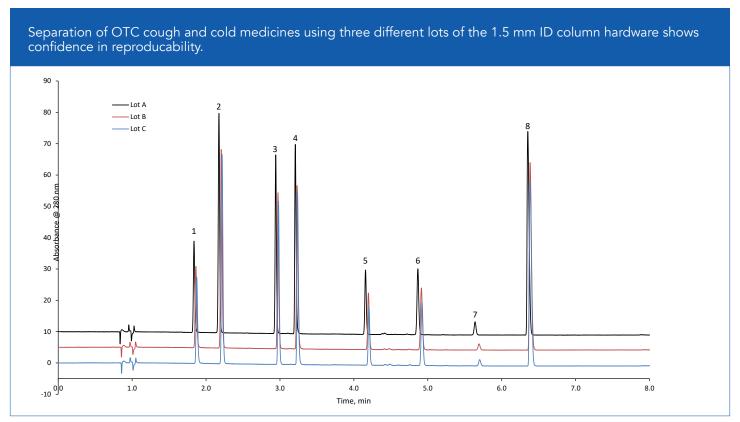
### >9X REDUCTION IN SOLVENT GOING FROM A 4.6 MM ID COLUMN TO A 1.5 MM ID COLUMN





## **STABILITY**

The new HALO<sup>®</sup> 1.5 meets the same rigorous quality standards of all HALO<sup>®</sup> products. Multi lot testing on the new hardware design and a QA report on every column produced from our ISO certified facility results in reliability you can depend on, column to column, analysis to analysis for the lifetime of the method.



### EXCELLENT LOT TO LOT REPRODUCIBILITY WITH THE 1.5 MM ID HARDWARE

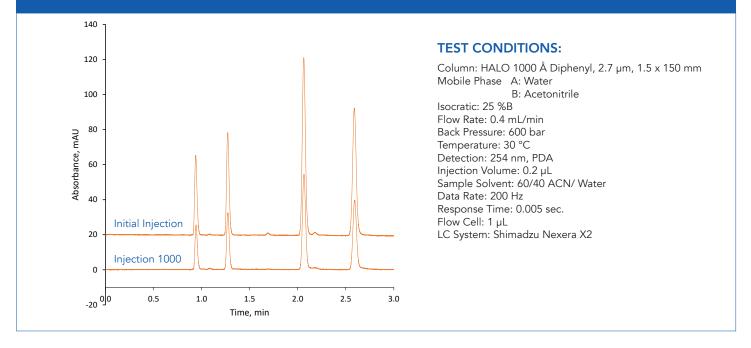
Test conditions for the above chromatogram are the same as p.3 top figure.



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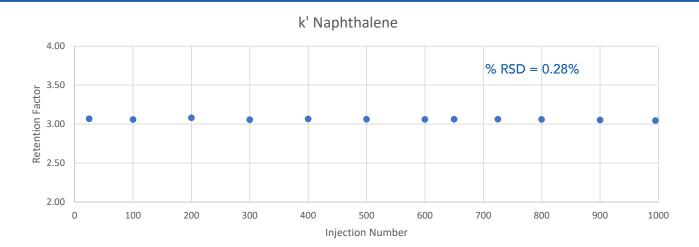
### STABILITY DEMONSTRATION WITH HALO 1000 Å DIPHENYL

The HALO 1000 Å Diphenyl in 1.5 mm ID column hardware was tested at 600 bar for 1000 injections. No loss in efficiency or retention was observed over the course of the experiment.



### 600 BAR STABILITY USING HALO 90 Å C18

A HALO 90 Å C18 1.5 mm ID column was run for 1000 injections to demonstrate the stability of the new 1.5 mm ID hardware. The retention factor of naphthalene was stable across all of the injections.



### **TEST CONDITIONS:**

Column: HALO 90 Å C18, 2.7 µm, 1.5 x 150 mm Mobile Phase A: Water B: Acetonitrile Isocratic: 60 %B Flow Rate: 0.6 mL/min Back Pressure: ~600 bar Temperature: 30 °C Detection: 254 nm, PDA

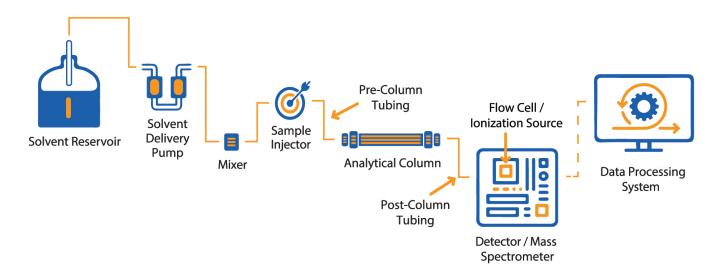
Injection Volume: 0.2 µL Sample Solvent: 60/40 ACN/ Water Data Rate: 200 Hz Response Time: 0.005 sec. Flow Cell: 1 µL LC System: Shimadzu Nexera X2



## SYSTEM OPTIMIZATION

Success with smaller ID columns requires attention to optimizing the LC system hardware for best performance. While manufacturers of UHPLC systems in general have already reduced system volumes, consideration should be taken to items like tubing and flow cells which lead to extracolumn dispersion.

Most critical when changing a gradient method from a current column to the new 1.5 mm ID column is the system dwell volume and extracolumn dispersion. The dispersion occurs post-column in the tubing leading to the detector and within the detector itself. Under isocratic conditions, the dispersion comes from both pre- and post-column tubing as well as injection volume and detector.



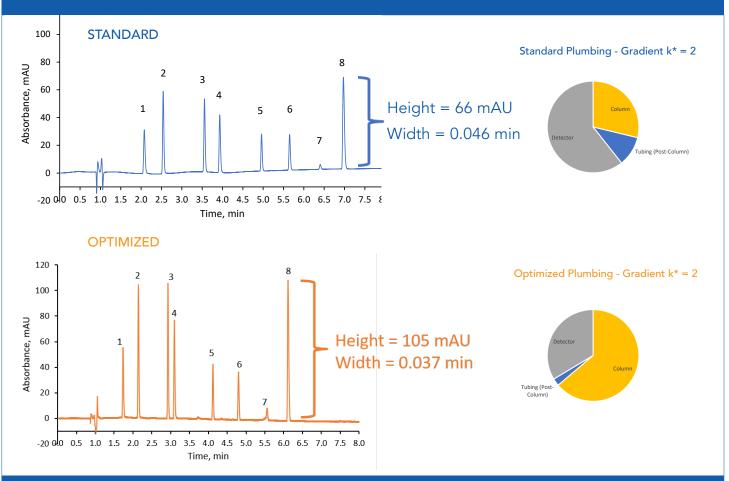
### EXAMPLE DEMONSTRATING REDUCTION OF UHPLC VOLUMES

COMPONENT	STANDARD UHPLC SYSTEM	OPTIMIZED UHPLC SYSTEM	
Mixer (µL)	100	20	
Pre-Column Tubing Volume (µL)	0.1 mm x 800 mm 6.3	75 μm x 350 mm 1.5	
Post-Column Tubing Volume (µL)	0.1 mm x 509 mm 4	60 μm x 707 mm 2	
Flow Cell (PDA) Volume (µL)	1	1	
Extracolumn Dispersion (µL²)	14	2	

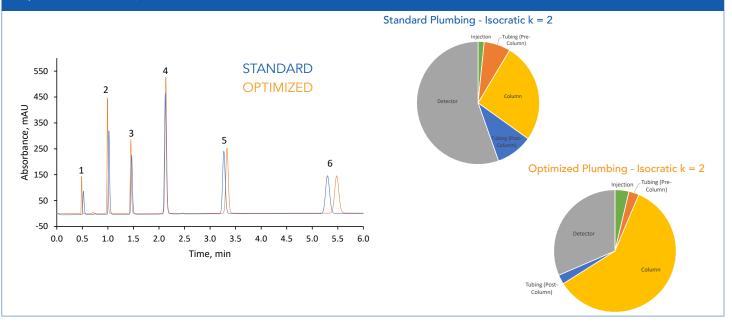


### COMPARISON OF STANDARD TO OPTIMIZED SYSTEM CONFIGURATION FOR OTC COUGH AND COLD MEDICINES

When the UHPLC is optimized, peak heights are taller and peak widths are smaller, leading to improved sensitivity. Notice how the pie wedges for the post-column tubing and the detector are reduced in size when the UHPLC system is optimized for this gradient separation.



Using isocratic conditions, the extracolumn dispersion comes from both pre- and post-column tubing, the injection volume, and the detector. In this comparison the peaks are taller and the peak efficiencies are increased with the optimized UHPLC system.





## ENSURE A PERFECT CONNECTION WITH MarvelXACT<sup>™</sup>

MarvelXACT<sup>™</sup> connection systems have been designed to eliminate the risk of under- or over-tightening with a patented torque limiting mechanism. This unique feature emits a haptic "click" feedback when it reaches the optimum torque, assuring a perfect installation every time. MarvelXACT<sup>™</sup> incorporates advanced MarvelX<sup>™</sup> Sealing Technology to deliver precise face sealing (sealing at the port bottom), which eliminates additional internal volume, and minimizes carryover risk, peak tailing, and peak broadening.

### MarvelXACT<sup>™</sup> VS. CONVENTIONAL CONED FITTINGS



EXTRA INTERNAL VOLUME

Conventional coned fittings require a ferrule in conjunction with a fitting for proper sealing. They depend on tools, to improve sealing performance, which significantly increases probablility of extra internal volume and poor chromatography results. The mechanical tightening increases wear leading to higher replacement costs.



ZERO DEAD VOLUME

MarvelXACT<sup>™</sup> fittings do not depend on ferrules. They seal with hand tightening at the bottom of the port, which significantly reduces required torque and enables many more connects and disconnects reducing wear and increasing product life. An enhanced proprietary tip design also ensures zero dead volume (ZDV) and better chromatography results.



### EXACT TIGHTENING WITH A "CLICK"

Achieve a perfect connection every time with built-in patented technology that delivers a haptic "click" when optimum torque is achieved through finger-tightening.

## MARVELXACT

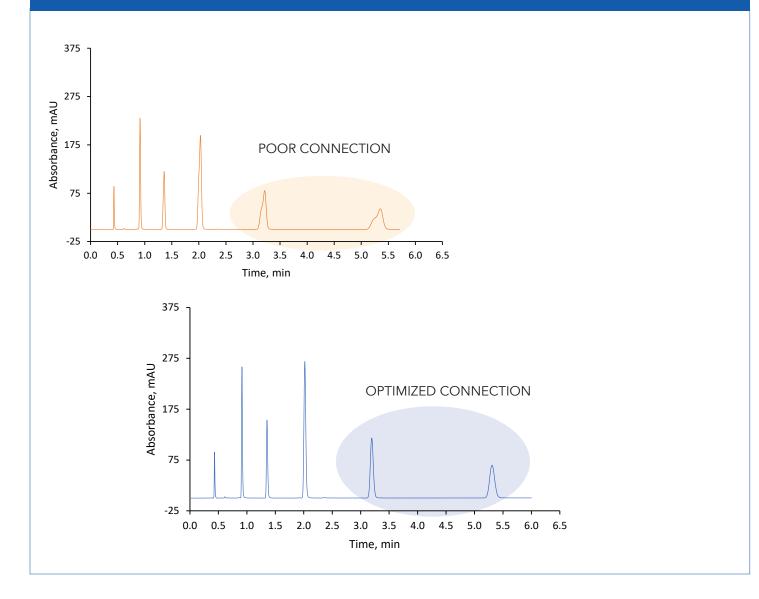
### FLEXIBLE TUBING

1/32" OD tubing prevents kinking and allows considerable flexibility to route throughout the instrument.

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### CONNECTION CONSEQUENCES

When a poor connection is made to the injector, distorted peak shape and leaks may occur. In this example, the connecting tubing was not fully seated into the injector port which caused a slow leak and distorted peak shape, especially for the later eluting peaks.



### FINGER-TIGHT TO UHPLC

MarvelXACT<sup>™</sup> is truly a finger-tight connection system that has a patented torque-limiting mechanism for exact tightening every time, and seals up to 19000 psi (~1310 barr) for routine use.

### SMALL & ACCESSIBLE

Fittings are small enough to fit in tight spaces, yet allow for finger-tightening at UHPLC pressures.

### **ROBUST TIP**

Enable robust structure, superior re-usability, and minimizes chances of tip damage from connecting and disconnecting.

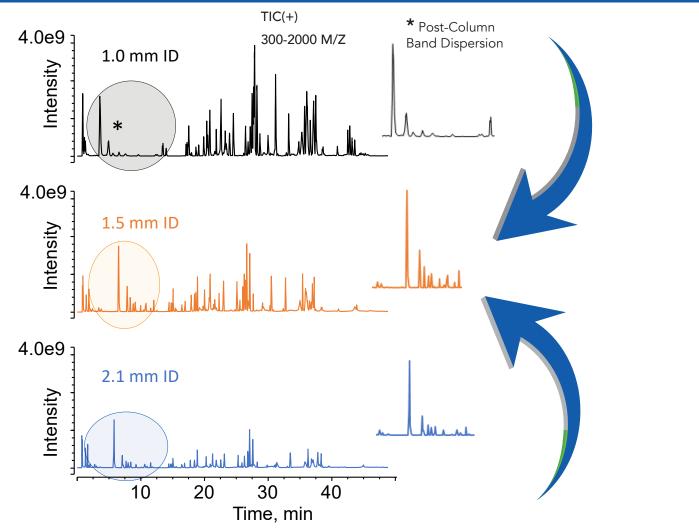




## **EXTRACOLUMN DISPERSION**

### IMPACT OF EXTRACOLUMN DISPERSION ON DIFFERENT ID COLUMNS

The 1.0 mm ID column shows broad peak shape due to the amount of extracolumn dispersion going from the column to the source of the MS. The 1.5 mm ID performs better than both the 2.1 mm ID (in terms of peak height) and the 1.0 mm ID (in terms of peak width).



### **TEST CONDITIONS:**

Column: HALO 160 Å ES-C18, 2.7  $\mu m,$  1.0 x 150 mm Column: HALO 160 Å ES-C18, 2.7  $\mu m,$  1.5 x 150 mm Column: HALO 160 Å ES-C18, 2.7  $\mu m,$  2.1 x 150 mm

Mobile Phase A: Water/0.1% DFA B: Acetonitrile/0.1% DFA Gradient: 2-50 %B in 60 min Flow Rate: 0.1 mL/min for 1.0 mm ID 0.2 mL/min for 1.5 mm ID 0.4 mL/min for 2.1 mm ID Back Pressure: 265 bar (1.0 mm) 310 bar (1.5 mm) 444 bar (2.1 mm) Temperature: 60 °C Detection: ESI + Injection Volume: 2 μL of 1.25 mg/mL Trastuzumab tryptic digest Sample Solvent: 1.5 M Guanidine HCl/0.5%Formic Acid LC System: Shimadzu Nexera X2 MS System: ThermoFisher Q Exactive

### **MS TEST CONDITIONS:**

Spray Voltage (kV): 3.8 Capillary temperature: 320 °C Sheath gas: 35 Aux gas: 10 RF lens: 50



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## **CONVERTING TO THE NEW 1.5**

### WHY SHOULD WE CARE ABOUT EXTRACOLUMN DISPERSION?

The analyte bands or peaks naturally broaden as they move through the connecting tubing in a UHPLC system. The smaller the column ID, the more this broadening could impact the results of the column. If the true efficiency of the column is the goal, then the extracolumn dispersion must be reduced. The extracolumn dispersion comes from the injector, the pre-column tubing, the heat exchanger, the post-column tubing, and the detector. For isocratic separations, all of these impact the observed efficiency. For gradient separations, only the post-column tubing and the detector impact the observed efficiency. In order to maximize what efficiency the column is capable of delivering, the extracolumn volume needs to be reduced as much as possible by using shorter length, smaller ID tubing. However, changing to smaller ID tubing will increase the pressure of the system so a compromise must be made in order to still be able to run at the flow rates needed for optimum column performance.

### EQUATION FOR SCALING FLOW RATE

$$F_2 = F_1 \times \frac{(\pi R_2)^2}{(\pi R_1)^2} = F_1 \times \frac{(R_2)^2}{(R_1)^2} = F_1 \times \frac{(D_2)^2}{(D_1)^2}$$

Where F =flow rate

R = radius

D = diameter

- 1 = original column
- 2 =column being changed to

	COLUMN IDS				
	4.6	3.0	2.1	1.5	1.0
FLOW RATES (mL/min)	0.96	0.41	0.20	0.10	0.045
	1.44	0.61	0.30	0.15	0.068
	1.92	0.82	0.40	0.20	0.091
	2.40	1.02	0.50	0.26	0.113
	2.88	1.22	0.60	0.31	0.136

### EXTRACOLUMN DISPERSION REFERENCES

- 1. D.R. Stoll, K. Broeckhoven, LCGC North America. 39, Issue 4 (2021) 159–166.
- 2. G. Desmet, K. Broeckhoven, TrAC Trends Anal. Chem. 119 (2019) 115619.
- 3. K. Broeckhoven, J. De Vos, and G. Desmet, LCGC Europe 30 (2017) 618–625.



## HOW TO SELECT THE BEST CONNECTORS FOR THE BEST 1.5 MM ID PERFORMANCE

- In general, use the shortest length and smallest ID tubing that will work for your system
- Are you running isocratic or gradient?
  - If isocratic, then pre-column tubing is most impactful reduce length and ID of tubing before the column
  - If gradient, then post-column tubing is most impactful reduce the length and ID of tubing after the column
- Does your system have a heat exchanger?
  - If so, consider bypassing it as long as you are able to operate your method without it
- Is there a smaller volume flow cell available for your system?
  - If so, consider switching to it

### STEPS FOR SELECTING CONNECTING TUBING

1. Measure the length in mm from the injector to the column and from the column to the detector or MS source

2. Select ID tubing and length that will fit according to the table below being mindful of your system back pressure limits:

	L(mm)	ID (µm)	volume (uL)	
	25	0.07		
	50	0.29		
	150	75	0.66	
	100	1.18		

L(mm)	ID (μm)	volume (uL)	
350	25	0.17	
	50	0.69	
	75	1.55	
	100	2.75	

L(mm)	ID (μm)	volume (uL)	
600	25	0.29	
	50	1.18	
	75	2.65	
	100	4.71	

Green = minimal extra back pressure (<15 bar)

Orange = may generate too much back pressure (>40 bar)

Red = not recommended (>100 bar)

### CONNECTION TUBING OPTIONS

DESCRIPTION	VOLUME	PART NUMBER	DESCRIPTION	VOLUME	PART NUMBER
AMT MarvelXACT™ PLS 25µm x 150mm	75 nl	PL7025150	AMT MarvelXACT™ PEEKsil™ 25µm ID x 150mm	75 nl	PS7025150
AMT MarvelXACT™ PLS 25µm x 350mm	170 nl	PL7025350	AMT MarvelXACT™ PEEKsil™ 25µm ID x 350mm	170 nl	PS7025350
AMT MarvelXACT™ PLS 25µm x 600mm	295 nl	PL7025600	AMT MarvelXACT™ PEEKsil™ 25µm ID x 600mm	295 nl	PS7025600
AMT MarvelXACT™ PLS 50µm x 150mm	295 nl	PL7050150	AMT MarvelXACT™ PEEKsil™ 50µm ID x 150mm	295 nl	PS7050150
AMT MarvelXACT™ PLS 50µm x 350mm	685 nl	PL7050350	AMT MarvelXACT™ PEEKsil™ 50µm ID x 350mm	685 nl	PS7050350
AMT MarvelXACT™ PLS 50µm x 600mm	1178 nl	PL7050600	AMT MarvelXACT™ PEEKsil™ 50µm ID x 600mm	1178 nl	PS7050600
AMT MarvelXACT™ PLS 75µm x 150mm	665 nl	PL7075150	AMT MarvelXACT™ PEEKsil™ 75µm ID x 150mm	665 nl	PS7075150
AMT MarvelXACT™ PLS 75µm x 350mm	1545 nl	PL7075350	AMT MarvelXACT™ PEEKsil™ 75µm ID x 350mm	1545 nl	PS7075350
AMT MarvelXACT™ PLS 75µm x 600mm	2650 nl	PL7075600	AMT MarvelXACT™ PEEKsil™ 75µm ID x 600mm	2650 nl	PS7075600
AMT MarvelXACT™ PLS 100µm x 150mm	1178 nl	PL7100150	AMT MarvelXACT™ PEEKsil™ 100µm ID x 150mm	1178 nl	PS7100150
AMT MarvelXACT™ PLS 100µm x 350mm	2750 nl	PL7100350	AMT MarvelXACT™ PEEKsil™ 100µm ID x 350mm	2750 nl	PS7100350
AMT MarvelXACT™ PLS 100µm x 600mm	4710 nl	PL7100600	AMT MarvelXACT™ PEEKsil™ 100µm ID x 600mm	4710 nl	PS7100600

## HALO® 1.5 PRODUCT OFFERINGS

	ANALYTICAL COLUMNS						
MM COLUMN SIZES AND PHASES	BONDED PHASE	PARTICLE SIZE	PORE SIZE	DESCRIPTION	PART NUMBER		
H				HALO 90 A C18, 2.7 μm, 1.5 x 50 mm	9281X-402		
	C18	2.7 UM	90 Å	HALO 90 A C18, 2.7 μm, 1.5 x 100 mm	9281X-602		
Z				HALO 90 A C18, 2.7 μm, 1.5 x 150 mm	9281X-702		
S				HALO 90 Å AQ-C18, 2.7 μm, 1.5 X 50mm	9281X-422		
ZE	AQ-C18 2.7 UM 90	90 Å	HALO 90 Å AQ-C18, 2.7 μm, 1.5 X 100mm	9281X-622			
N S				HALO 90 Å AQ-C18, 2.7 μm, 1.5 X 150mm	9281X-722		
Σ				HALO 90 Å LPH-C18, 2.7 μm, 1.5 X 50mm	9282X-416		
	LPH-C18	2.7 UM	90 Å	HALO 90 Å LPH-C18, 2.7 μm, 1.5 X 100mm	9282X-616		
8				HALO 90 Å LPH-C18, 2.7 μm, 1.5 X 150mm	9282X-716		
Σ				HALO 90A PCS C18, 2.7um 1.5 mm x 50 mm	9281X-417		
	PCS C18	2.7 UM	90 Å	HALO 90A PCS C18, 2.7um 1.5 mm x 100 mm	9281X-617		
1.5				HALO 90A PCS C18, 2.7um 1.5 mm x 150 mm	9281X-717		
Ì				HALO 90 Å Penta-HILIC, 2.7 μm, 1.5 X 50mm	9281X-405		
	Penta-HILIC 2.7 UM	2.7 UM	90 Å	HALO 90 Å Penta-HILIC, 2.7 μm, 1.5 X 100mm	9281X-605		
_			HALO 90 Å Penta-HILIC, 2.7 μm, 1.5 X 150mm	9281X-705			
				HALO 160 A ES-C18, 2.7 µm, 1.5 x 50 mm	9212X-402		
	ES-C18	2.7 UM	160 Å	HALO 160 A ES-C18, 2.7 µm, 1.5 x 100 mm	9212X-602		
				HALO 160 A ES-C18, 2.7 µm, 1.5 x 150 mm	9212X-702		
				HALO 160 Å ES-C18, 2.0 μm, 1.5 X 50mm	9112X-402		
	ES-C18	2 UM	160 Å	HALO 160 Å ES-C18, 2.0 μm, 1.5 X 100mm	9112X-602		
				HALO 160 Å ES-C18, 2.0 μm, 1.5 X 150mm	9112X-702		
				HALO 160A PCS C18, 2.7um 1.5 mm x 50 mm	9211X-417		
	PCS C18	2.7 UM	160 Å	HALO 160A PCS C18, 2.7um 1.5 mm x 100 mm	9211X-617		
				HALO 160A PCS C18, 2.7um 1.5 mm x 150 mm	9211X-717		
				HALO 1000 A C4, 2.7 μm, 1.5 x 50 mm	9271X-414		
	C4	2.7 UM	1000 Å	HALO 1000 A C4, 2.7 μm, 1.5 x 100 mm	9271X-614		
				HALO 1000 A C4, 2.7 μm, 1.5 x 150 mm	9271X-714		
				HALO 1000 A Diphenyl, 2.7 μm, 1.5 x 50 mm	9271X-426		
	Diphenyl	2.7 UM	1000 Å	HALO 1000 A Diphenyl, 2.7 μm, 1.5 x 100 mm	9271X-626		
				HALO 1000 A Diphenyl, 2.7 µm, 1.5 x 150 mm	9271X-726		

ISO 9001:2015 certified QMS

# HALO

## Manufactured by:



advancedmaterialstechnology

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Made in the USA

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1.5Brochure\_Rev\_2

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