HALO®

HALO® 90 Å Selectivity Kit

HALO® 90 A C18, AQ-C18, BIPHENYL

Fused-Core® Particle Technology

HALO® 90 Å UHPLC and HPLC Selectivity Kit

- HALO® 90 Å Selectivity Kit is available in 50 and 150 mm lengths with 2.1 and 4.6 mm IDs.
- The selectivity kit is comprised of three phases, with 2.7 µm Fused-Core® particles, which offer selectivity options.
- All columns in the kit provide excellent peak shape, efficiency, reproducibility and column lifetime for challenging separations.
- This kit allows you to change the selectivity of your separation with three unique bonding chemistries that provide options for selectivity adjustment and optimization.

F_s	Phase	H	S	A	B	$C_{2.8}$	$C_{7.0}$	Overall Retention	USP Type	Phase
0.00	HALO® C18	1.100	0.040	0.000	-0.050	0.050	0.04	6.1	L1	C18
11.85	HALO® AQ-C18	1.000	-0.036	0.100	-0.048	0.160	0.86	6.7	L1	C18
26.51	HALO® Biphenyl	0.708	-0.180	-0.280	0.028	0.047	0.99	3.1	L11	Phenyl

References:

1. Journal of Chromatography A, 1057 (2004) 49-57, L. R. Snyder et al.
2. www.hplccolumns.org, Results from characterization using Hydrophobic Subtraction Model procedure.

Resolution: Selectivity, Efficiency and Retention

Efficiency | Selectivity | Retention

$$R_S = \left(\frac{\sqrt{N}}{4}\right) \times \left[\frac{(\alpha-1)}{\alpha}\right] \times \left[\frac{k_2}{(1+\overline{k})}\right]$$

where

$$\overline{k} = \frac{(k_1+k_2)}{2}, \quad \alpha = \frac{k_2}{k_1} \quad \text{and} \quad N = \frac{L}{H} = \frac{L}{h \times d_p}$$

Of the three parameters in the resolution equation, selectivity (α) is, by far, the most powerful in its effect on resolution, compared to efficiency (N) and retention (k).

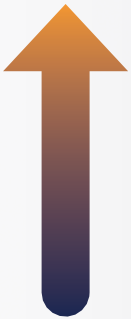
Factors That Impact Selectivity for Isocratic and Gradient RPLC Separations[2]

This is a list of parameters that affect reversed-phase selectivity (α or relative retention) for isocratic and gradient separations in HPLC and UHPLC. Column stationary phase is comparable to gradient time, percent organic modifier and gradient steepness in its ability to affect relative retention, and thus, resolution.

Isocratic Separations

- Mobile phase pH[1]
- Column stationary phase
- Organic modifier choice
- Percent organic modifier
- Column temperature
- Buffer choice
- Buffer concentration
- Additive concentration

Most Influence



Least Influence

Gradient Separations

- All parameters for isocratic separations
- Delay volume
- Gradient steepness (b=1/k*)

$$k^* = \frac{87 \times t_G \times F}{\Delta\Phi \times V_M \times S}$$

- t_G = Gradient time
- F = Flow rate
- S = Constant
- Φ = Gradient range (%B final - %B initial)
- V_M = Column volume (length, diameter)

[1]For ionizable analytes, mobile phase pH is the most powerful factor for adjusting selectivity.
[2]Adapted from "Introduction to Modern Liquid Chromatography," 3rd Edition. Snyder, Kirkland and Dolan, 2010, p.29, Wiley & Sons

HALO® C18 (Dimethyloctadecylsilane)

Analyte Interactions

- Hydrophobic

Advantages

- Excellent reproducibility
- Exceptional stability
- Impeccable performance for broad range of analyte polarities

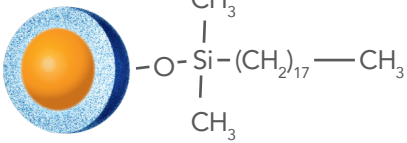
Target Analytes

- Analytes with differing hydrophobicities
- Applicable for a wide variety of different types of analytes in isocratic and gradient RPLC modes

Example Applications

- Pharmaceutical
- Chemical
- Clinical
- Environmental
- Cannabinoids

C18



HALO® Biphenyl (Dimethylbiphenylsilane)

Analyte Interactions

- Hydrophobic
- π-π
- Shape selectivity

Advantages

- Excellent reproducibility
- Complementary selectivity to alkylsilane stationary phases (e.g., C8 and C18)
- Enhanced retention and selectivity for aromatic compounds
- Improved retention for polar analytes

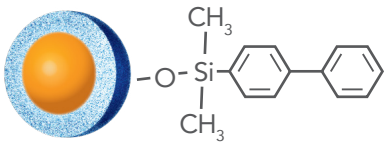
Target Analytes

- Electron-poor molecules, aromatic and other unsaturated compounds (ketones, nitriles, alkenes)

Example Applications

- Pain management panels
- Clinical samples
- Pesticides
- Heterocyclic aromatics

Biphenyl



HALO® AQ-C18 (Proprietary Ligand)

Analyte Interactions

- Hydrophobic
- Dipole-Dipole

Advantages

- Excellent reproducibility
- Compatible with 100% aqueous mobile phases in isocratic and gradient modes
- Enhanced retention for polar molecules

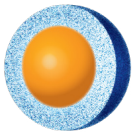
Target Analytes

- Acids, bases, polar analytes

Example Applications

- Polar organic acids
- Nucleobases
- Neurotransmitters
- Triazine pesticides

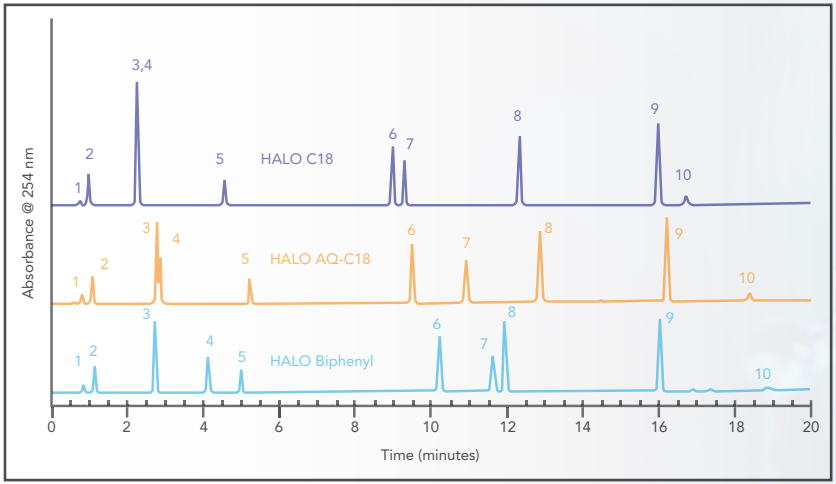
AQ-C18



$-(CH_2\text{-}(CH_2)_{16}\text{-}CH_3]_x$

$[\text{Polar Ligand}]_y$

Significant Selectivity Changes are Evident with CH_3OH



Peak Number	Compound Peak Identity	Peak Number	Compound Peak Identity
1	Uracil	6	Valerophenone
2	Resorcinol	7	trans-Stilbene
3	4-Nitrophenol	8	Biphenyl
4	Benzonitrile	9	o-Terphenyl
5	Anisole	10	Impurity

Test Conditions:

Columns: HALO® 90 Å, 2.7 µm, 4.6 x 100 mm

Solvent A: H_2O

Solvent B: Methanol

Gradient: A/B = 55/45 to 5/95 in 20 minutes

Flow Rate: 1.2 mL/minute

Pressure: ≈ 315 Bar (to start)

Temperature: 30 °C

Detection: 254 nm, VWD

Injection Volume: 2.0 µL

Sample Solvent: 50:50 Methanol/Acetonitrile

Response Time: 0.02 seconds

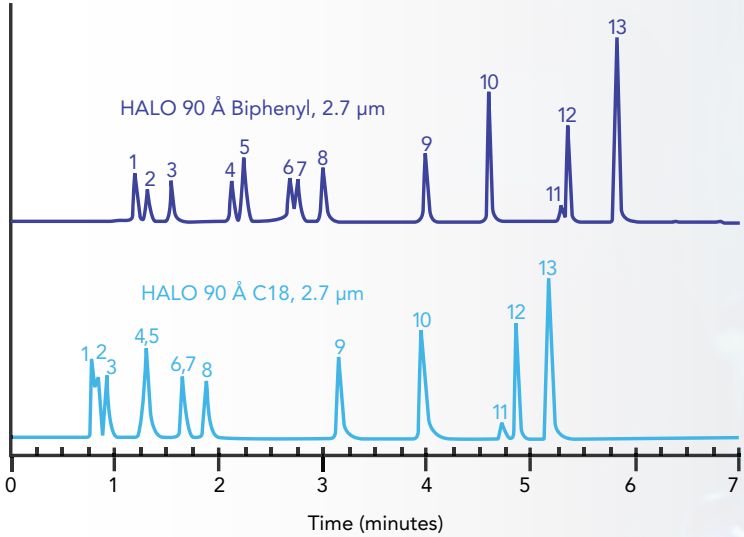
Data Rate: 25 Hz

Flow Cell: 2.5 µL, Semi-Micro

LC System: Shimadzu Prominence UFLC XR

Extracolumn Volume (ECV): 14 µL

HALO® Biphenyl Compared to HALO® C18 for Pain (Management) Panel



Test Conditions:

Column: HALO® 90 Å Biphenyl, 2.7 µm, 2.1 x 100 mm

Mobile Phase A: H_2O/0.1% Formic Acid

Mobile Phase B: ACN/0.1% Formic Acid

Gradient: 0 - 3 minutes, 10 - 20% B

3 - 3.5 minutes, 20 - 100% B

3.5 - 6 minutes, hold at 100% B

Flow Rate: 0.3 mL/minute

Temperature: 30 °C

Instrument: Agilent 1290

Dwell Volume: 0.19 mL

Injection Volume: 2 µL

Sample Solvent: H_2O: Methanol, 99:1

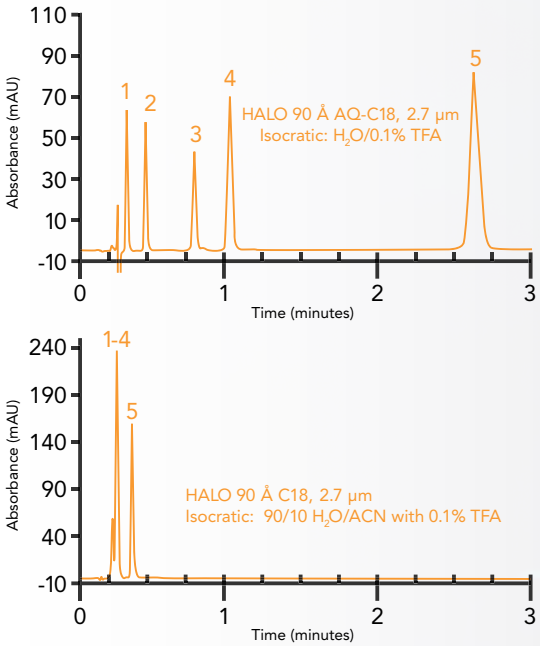
MS System: Agilent 6210 TOF

ESI: +4 kV

Peak Identities:

1. Morphine
2. Oxymorphone
3. Hydromorphone
4. Naloxone
5. Codeine
6. Naltrexone
7. Oxycodone
8. Hydrocodone
9. cis-Tramadol HCl
10. Meperidine
11. Fentanyl
12. Buprenorphine
13. (±)-Methadone

Neurotransmitters: HALO® AQ-C18 vs. HALO® C18



Test Conditions:

Columns: HALO® 90 Å, 2.7 µm, 2.1 x 50 mm

Flow Rate: 0.5 mL/minute

Temperature: 30 °C

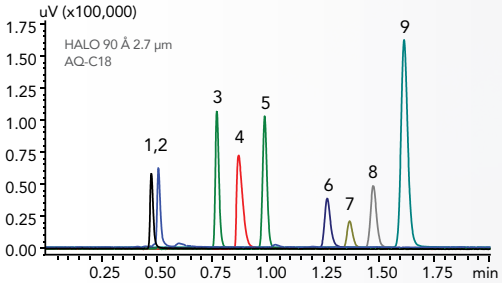
Detection: UV 210 nm, PDA

Injection Volume: 1 µL

Peak Identities:

1. Norepinephrine
2. Epinephrine
3. Dopamine
4. L-Dopa
5. Serotonin

HALO® AQ-C18 Comparison Against Competitors' SPP Columns

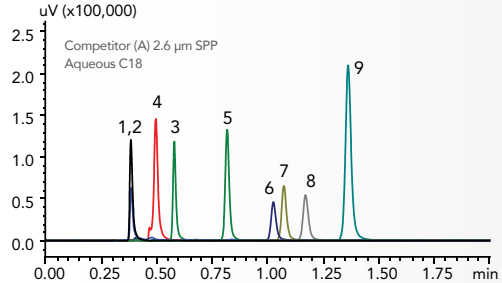


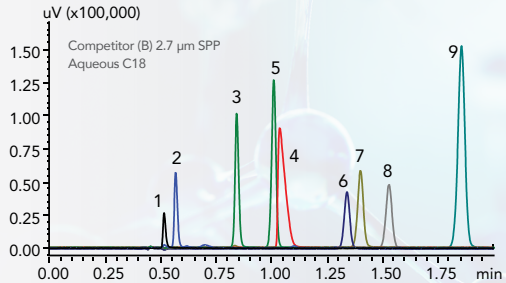
LC Conditions

Columns: 2.1 x 100 mm
Mobile Phase: 0.1% Formic Acid
Flow Rate: 0.5 mL/minute
Temperature: 35 °C
Detection: UV 254 nm
Injection: 1 µL
Instrument: Shimadzu Nexera

Peak Identities

1: 5-Amino-imidazole-4-carboxamide
2: Azepinomycin
3: Guanine (substrate)
4: 2,6-Diaminopurine
5: Uric acid
6: Xanthine (product)
7: 8-Azaxanthine
8: 8-Azaguanine
9: Allopurinol





Note: Samples degrade over time and that explains peak height differences. Also, each component was injected individually; they are not all in one mix.

HALO® 90 Å Selectivity Kits Specifications Table

	Octadecyl	Biphenyl	AQ-C18
Functional Group	Dimethyloctadecyl	Dimethylbiphenyl	Proprietary
USP Designation	L1	L11	L1
Particle Size (µm)	2.7	2.7	2.7
Pore Size (Ångstroms)	90	90	90
Carbon Load (%)	7.7	7.0	6.7
Surface Area (m^2/g)	135	135	135
Endcapped	Yes	Yes	Yes
pH Range	2-9	2-9	2-9
100% Aqueous Compatibility	No	Yes	Yes
Maximum Temperature Low pH	60	60	60
Maximum Temperature High pH	40	40	40
Maximum Pressure	600 Bar	600 Bar	600 Bar

HALO® 90 Å Selectivity Kit List Price

Column Dimensions (mm)	Phases Included	Part Number	Method Development Kit List Price	Price Until December 31st, 2018
2.1 x 50	C18 Biphenyl AQ-C18	928124KT	$1,935	$645
2.1 x 150	C18 Biphenyl AQ-C18	928127KT	$2,455	$820
4.6 x 50	C18 Biphenyl AQ-C18	928144KT	$1,935	$645
4.6 x 150	C18 Biphenyl AQ-C18	928147KT	$2,455	$820

MAC-MOD.COM
info@mac-mod.com
(800) 441-7508