# Impact of Using 1.5 mm Column ID and Various Stationary Phases on LC/MS Pesticide Screening Analysis

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## Background

- Pesticide screening analysis is used for testing various matrices, including food, soil, water, and cannabis
- Comparison of retention times and peak shapes from 3 different superficially porous particle alkyl stationary phases:
  - HALO® C18, HALO® LPH-C18, and HALO® AQ-C18
- Investigation on how changing from a 2.1 mm ID column to a 1.5 mm ID column impacts the results

## Methods

Sample: Oregon Pesticide Mixture 10x Action Limit 2-20 µg/mL in ACN from LGC (DRE-GA09000244AL)

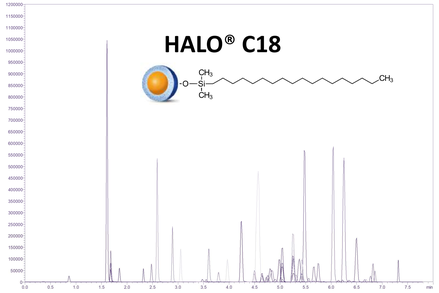
Columns: HALO 90 Å C18, 2.7 µm, 2.1 x 50 mm (92812-402)
HALO 90 Å LPH-C18, 2.7 µm, 2.1 x 50 mm (92822-416)
HALO 90 Å AQ-C18, 2.7 µm, 2.1 x 50 mm (92812-422)
HALO 90 Å AQ-C18, 2.7 µm, 1.5 x 50 mm (9281X-422)
Competitor SPP 100 Å 100% Aqueous Compatible Phase, 2.6 µm, 2.1 x 50 mm

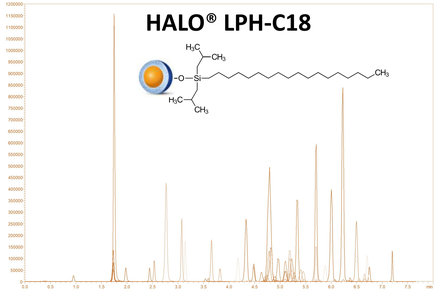
Mobile Phase A: Water with 5 mM ammonium formate & 0.1% Formic Acid
Mobile Phase B: Methanol with 5 mM ammonium formate & 0.1% Formic Acid

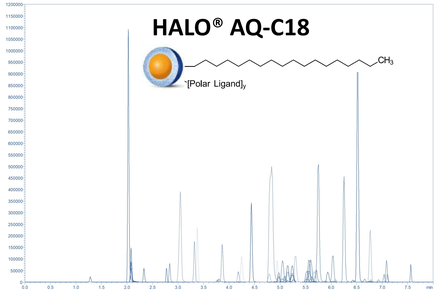
Gradient 1: 0-100% B in 7 min, hold at 100% for 1 min, then ramp to 0% B in 0.5 min, hold at 0% B for 3.5 min (AQ-C18, SPP 100% Aqueous Compatible Phase)
Gradient 2: 5-100% B in 6.65 min, hold at 100% for 1.35 min, then ramp to 0% B in 0.5 min, hold at 0% B for 3.5 min (C18, LPH-C18)

Flow Rate: 0.5 mL/min (2.1 mm) or 0.25 mL/min (1.5 mm)
Temperature: 35 °C
Detection: + ESI MS/MS for all except fludioxinil and fipronil
Injection Volume: 1 µL of 0.4 – 4 µg/mL
Sample Solvent: 80/20 Mobile Phase A/ACN
MS System: Shimadzu 8040 triple quadrupole
LC System: Shimadzu Nexera X2
Nebulizer Gas Flow: 2 L/min
DL Temperature: 250 °C
Heat Block Temperature: 400 °C
Drying Gas Flow: 15 L/min
Column outlet to Ground: AMT MarvelXACT™ PEEKsil™ 75 µm ID x 600 mm
Ground to Source: AMT MarvelXACT™ PEEKsil™ 75 µm ID x 150 mm

## Stationary Phase Screening: Which C18 Should I Use?




| Name | m/z | AQ-C18 Retention Time | LPH-C18 Retention Time | C18 Retention Time |
|---|---|---|---|---|
| Daminozide | 161.1000>143.1000 | 0.48 | 0.38 | 0.34 |
| Acephate | 184.1000>143.1000 | 1.28 | 0.95 | 0.86 |
| Oxamyl | 237.1000>72.1000 | 2.03 | 1.75 | 1.61 |
| Flonicamid | 230.1000>203.1000 | 2.08 | 1.73 | 1.68 |
| Methomyl | 163.1000>88.1000 | 2.12 | 1.83 | 1.70 |
| Thiamethoxam | 292.1000>132.1000 | 2.33 | 1.98 | 1.85 |
| Imidacloprid | 256.1000>175.1000 | 2.77 | 2.44 | 2.32 |
| Dimethoate | 230.1000>125.1000 | 2.84 | 2.53 | 2.48 |
| Acetamiprid | 223.1000>126.1000 | 3.05 | 2.77 | 2.59 |
| Aldicarb | 208.1000>89.1000 | 3.32 | 3.14 | 3.05 |
| Thiacloprid | 253.1000>126.1000 | 3.38 | 3.07 | 2.89 |
| Dichlorvos | 221.1000>109.1000 | 3.77 | 3.54 | 3.48 |
| Propoxur | 210.1000>111.1000 | 3.80 | 3.59 | 3.55 |
| Carbofuran | 222.1000>165.1000 | 3.87 | 3.66 | 3.60 |
| Carbaryl | 202.1000>145.1000 | 4.18 | 3.82 | 3.79 |
| Imazalil | 297.1000>159.1000 | 4.24 | 4.17 | 3.97 |
| Metalaxyl | 280.2000>220.1000 | 4.44 | 4.33 | 4.24 |
| Naled | 381.1000>127.1000 | 4.61 | 4.35 | 4.33 |
| Parathion-methyl | 264.2000>232.1000 | 4.67 | 4.55 | 4.51 |
| Chlorantraniliprole | 481.9000>283.9000 | 4.79 | 4.48 | 4.49 |
| Spiroxamine | 298.1000>144.1000 | 4.83 | 4.79 | 4.57 |
| Phosmet | 318.1000>160.1000 | 4.94 | 4.48 | 4.50 |
| Methiocarb | 226.1000>121.1000 | 4.97 | 4.63 | 4.64 |
| Azoxystrobin | 404.1000>344.1000 | 4.97 | 4.70 | 4.64 |
| Paclobutrazol | 294.1000>70.2000 | 5.05 | 4.79 | 4.80 |
| Boscalid | 343.1000>307.1000 | 5.09 | 4.74 | 4.76 |
| Fludioxinil | 246.6000>125.9000 | 5.14 | 4.70 | 4.73 |
| Malathion | 331.1000>99.1000 | 5.15 | 4.82 | 4.84 |
| Myclobutanil | 289.1000>69.9000 | 5.15 | 4.90 | 4.90 |
| Pyrethrin I | 329.1000>161.1000 | 5.18 | 6.30 | 6.32 |
| Spirotetramat | 374.1000>330.1000 | 5.24 | 5.10 | 5.04 |
| Ethoprophos | 243.1000>130.9000 | 5.26 | 5.10 | 5.04 |
| Bifenazate | 301.1000>170.2000 | 5.30 | 4.96 | 4.98 |
| Fipronil | 434.9000>330.8000 | 5.52 | 5.19 | 5.26 |
| Fenoxycarb | 302.1000>116.1000 | 5.57 | 5.23 | 5.25 |
| Tebuconazole | 308.2000>70.1000 | 5.60 | 5.34 | 5.37 |
| Kresoxim-methyl | 314.1000>116.1000 | 5.62 | 5.28 | 5.31 |
| Propiconazole | 342.1000>69.1000 | 5.67 | 5.46 | 5.42 |
| Diazinon | 305.1000>169.1000 | 5.71 | 5.41 | 5.43 |
| Spinosad A | 732.6000>142.1000 | 5.75 | 5.70 | 5.48 |
| MGK 264 | 276.1000>210.1000 | 5.89 | 5.64 | 5.66 |
| Prallethrin | 301.2000>133.1000 | 5.91 | 5.70 | 5.69 |
| Spinosad D | 746.5000>142.1000 | 5.93 | 5.88 | 5.66 |
| Pyrethrin II | 373.2000>161.1000 | 6.01 | 5.79 | 5.75 |
| Chlorfenapyr | 409.2000>59.0000 | 6.03 | 5.70 | 5.75 |
| Trifloxystrobin | 409.1000>186.1000 | 6.03 | 5.71 | 5.75 |
| Clofentezine | 303.1000>138.1000 | 6.09 | 5.47 | 5.56 |
| Piperonyl butoxide | 356.2000>177.1000 | 6.25 | 6.01 | 6.04 |
| Hexythiazox | 353.1000>228.1000 | 6.43 | 6.07 | 6.14 |
| Chlorpyrifos | 351.9000>124.9000 | 6.45 | 6.04 | 6.15 |
| Spiromesifen | 388.2000>273.1000 | 6.46 | 6.24 | 6.22 |
| Etoxazole | 360.2000>141.1000 | 6.52 | 6.23 | 6.25 |
| Fenpyroximate | 422.2000>366.1000 | 6.66 | 6.37 | 6.35 |
| Cyfluthrin | 451.1000>191.1000 | 6.70 | 6.29 | 6.38 |
| Cypermethrin | 433.1000>191.0000 | 6.75 | 6.36 | 6.44 |
| Pyridaben | 365.2000>147.0000 | 6.77 | 6.50 | 6.49 |
| Abamectin | 890.7000>305.3000 | 6.78 | 6.66 | 6.68 |
| Permethrin | 408.1000>183.1000 | 7.04 | 6.66 | 6.77 |
| Etofenprox | 394.3000>177.1000 | 7.09 | 6.70 | 6.82 |
| Bifenthrin | 440.1000>166.2000 | 7.10 | 6.75 | 6.86 |
| Acequinocyl | 343.2000>189.1000 | 7.56 | 7.20 | 7.31 |

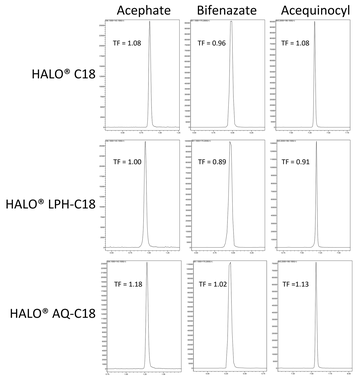
C18: Best for wide range of polarities
Endcapped

LPH-C18: Designed for low pH mobile phases
Not endcapped

AQ-C18: 100% aqueous compatible phase
Endcapped
More retention for polar compounds

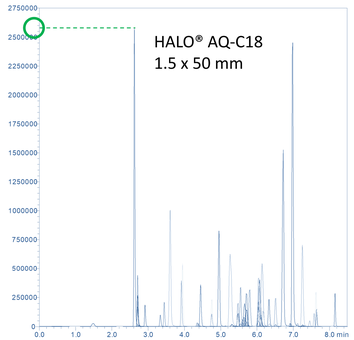
### Peak Statistics

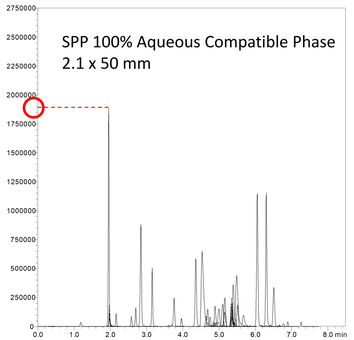
- Retention Order: AQ-C18 > LPH-C18> C18
- Peak Widths: average of 0.04 ± 0.01 for all
- Peak Tailing: AQ-C18 > C18 > LPH-C18




Based on these results, the AQ-C18 column was selected for investigation in the 1.5 mm ID

## HALO® AQ-C18 1.5 mm ID Compared to SPP 2.1 mm ID




- 1 µL of sample was injected on both the HALO® AQ-C18 1.5 mm ID column and the SPP 100% Aqueous Compatible Phase in 2.1 mm ID
- Area for the 61 MRMs that were monitored was an average of 2 times larger on the HALO® 1.5 mm ID column compared to the competitor 2.1 mm ID column
- Source conditions were kept the same for both columns for equivalency comparisons so some compounds, namely MGK 264, cyfluthrin, bifenthrin, and acequinocyl experienced greater signal enhancement than others

## Conclusions

- HALO® AQ-C18 gave the best compromise of retention time (highest) and peak shape (1.08 on average) across all of the compounds in the Oregon pesticide mix
- Injecting the same amount of sample on a 1.5 mm ID column as a 2.1 mm ID column leads to an increased response for enhanced peak interrogation and area as well as a 50% reduction of the mobile phase consumed
- Using 1.5 mm ID columns for MS analysis is a good alternative for those that do not want to move to micro flow or capillary systems, but still want signal enhancement and mobile phase savings