Development of a Semivolatile Column Optimized for the Analysis of Hazardous Waste

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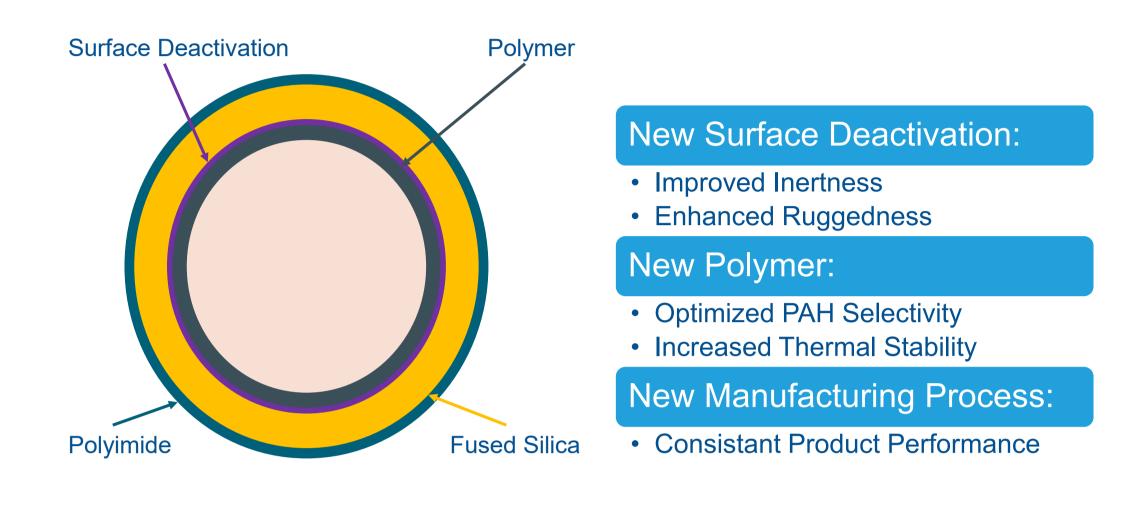
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Outline

Analyzing basic, neutral, and acidic semi-volatile environmental pollutants such as those found in US EPA 8270 at increasingly lower levels of detection puts demands on the entire analytical system. One of the greatest challenges is maintaining system performance following repeated injections of highly contaminated samples. The goal is to develop a column tuned for PAH selectivity while maintaining the same elution profile labs expect from other silarylene "5-type" columns along with improved lifetime.

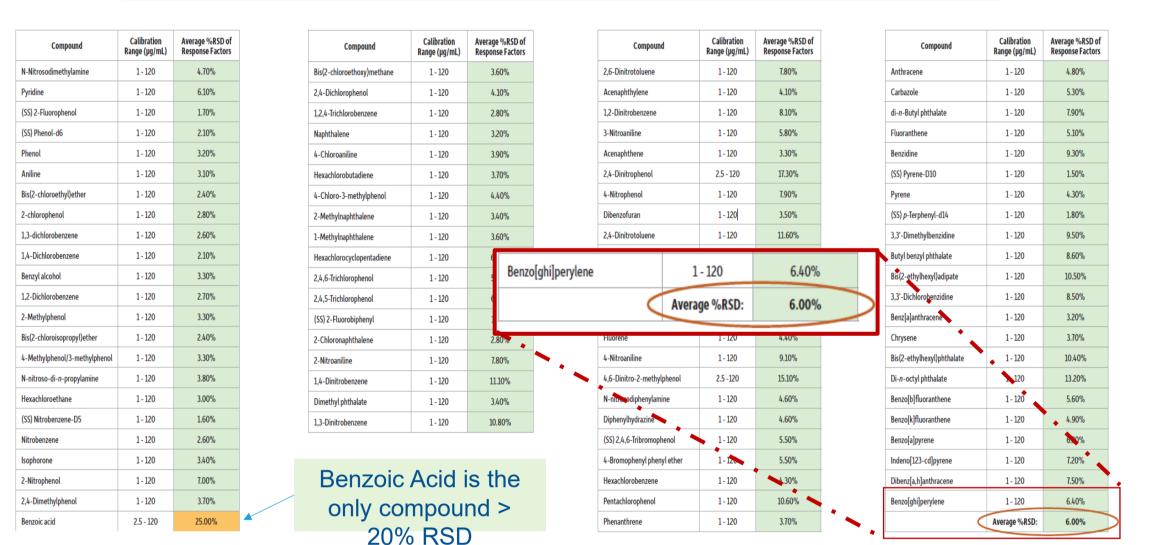
This presentation will review the details of the lifetime study results using contaminated extracts and will highlight the advantages of this column using a new deactivation and stationary phase.

Enhanced SVOC Performance



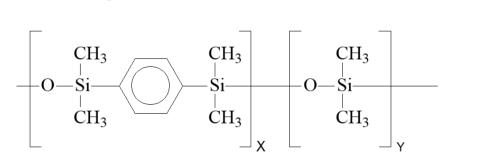
Inertness = Linear Calibrations

Average %RSDs for 6 Rxi-SVOCms columns (16623) calibrated from 1 ng/μL to 120 ng/μL



New Polymer

The polymer used for the Rxi-SVOCms has a silphenylene backbone, like the Rxi-5Sil MS



Application specific formulation changes optimized the PAH separations and improved thermal stability

New maximum temperature of 340C

Designed to be a drop-in replacement for the DB-5ms and DB-5ms UI

DB-UI 8270D for Semivolatiles
ZB-Semivolatiles
SLB-5ms Semivolatiles

Other Silphenylene type phases

Rxi-5Sil MS

DB-5ms

DB-5ms UI

VF-ms

BPX-5

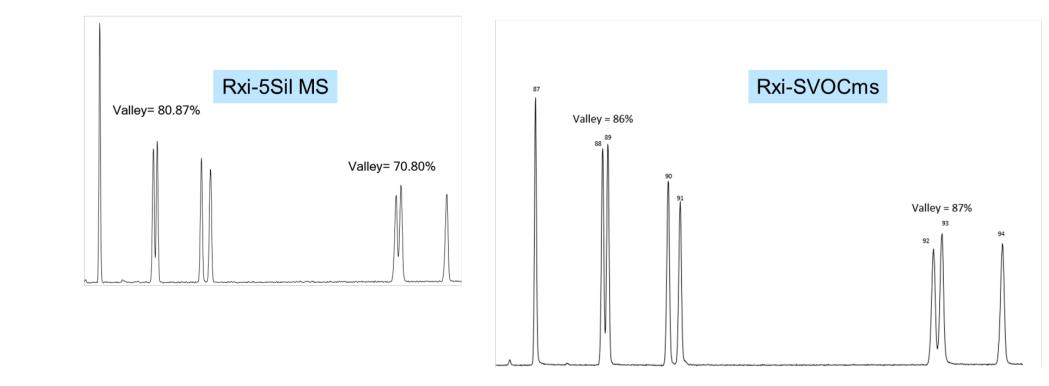
ZB-5ms

CP-Sil 8 CB LB/MS

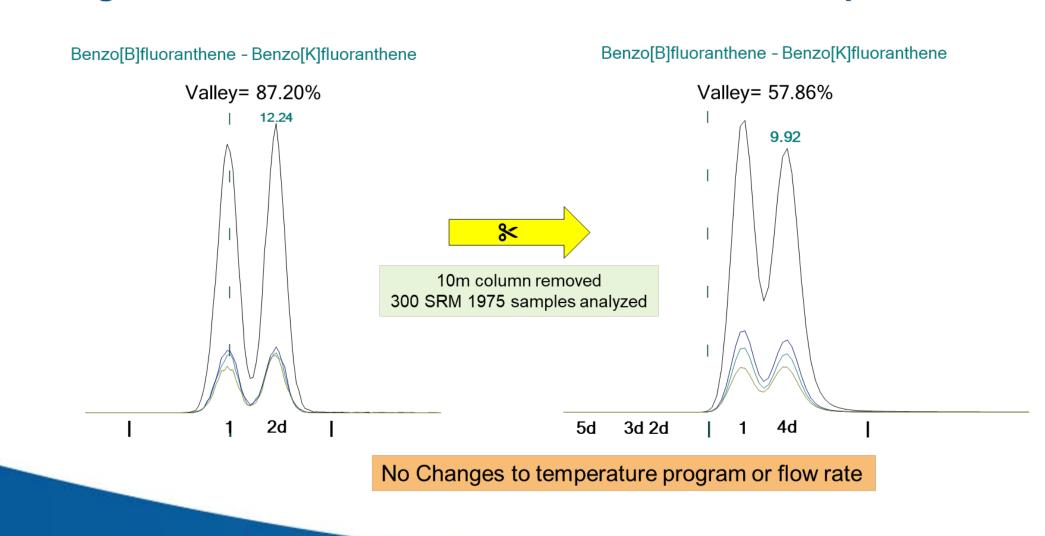
Optima-5ms

Optimized PAH Selectivity

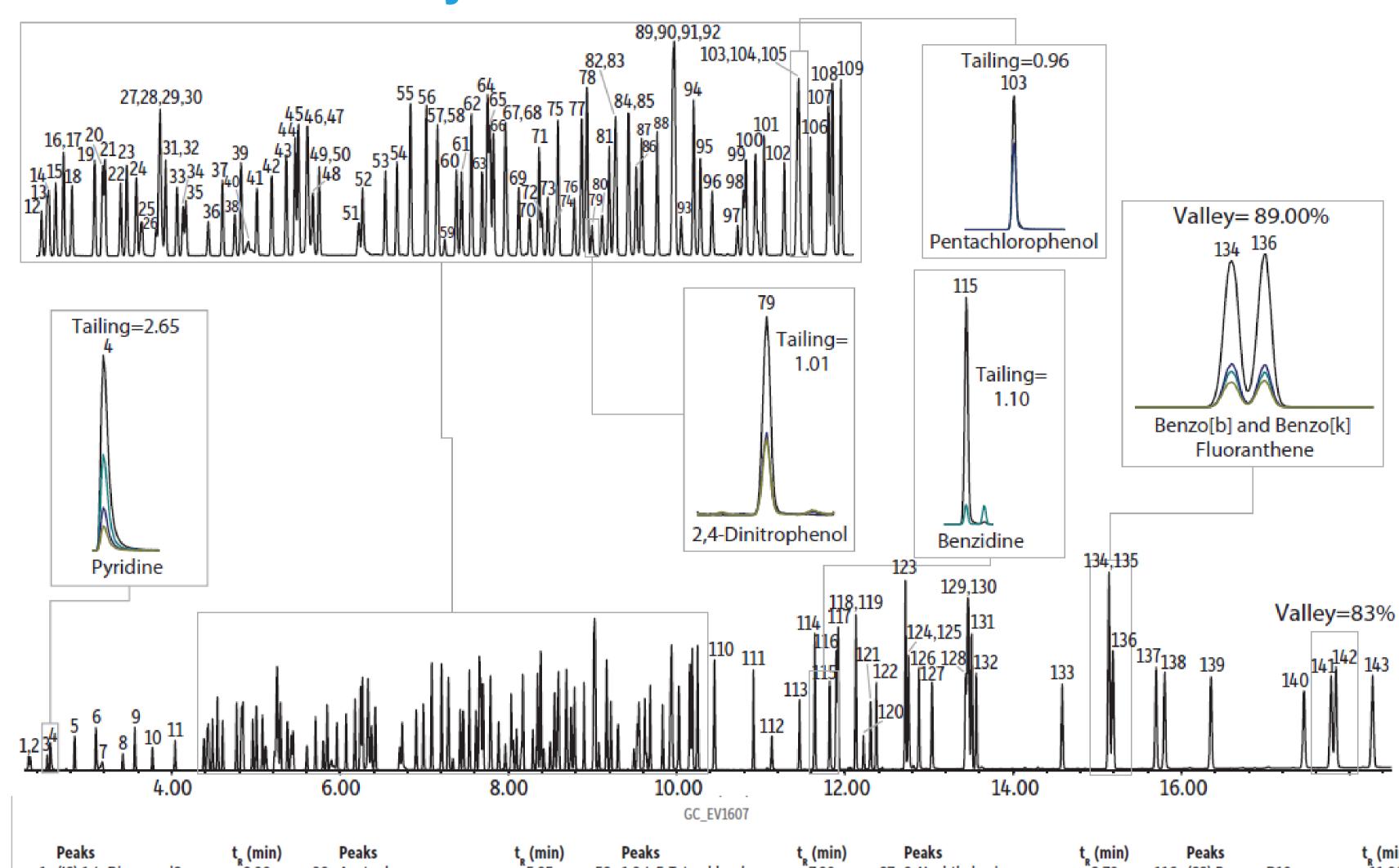
Improved Benzofluoranthene and Indeno[123-cd]pyrene – Dibenz[ah]anthracene Separations on the Rxi-SVOCms



Benzofluoranthene isomers are still sufficiently resolved after removing ten meters of from a 30 m x 0.25 mm x 0.25 µm Rxi-SVOCms



Semi-Volatile Analysis



| | 4.00 | | 6.00 8.0 | 0 | | 10.00 | 12.00 | | 14.00 | | 16 | .00 | |
|----|-------------------------------|-------------------|--|-------------------|-----|----------------------------|----------|------|---------------------------|-------------------|------|-----------------------------|---------------------|
| | | | | | | GC_EV1607 | | | | | | | |
| | Peaks t | (min) | Peaks t | (min) | | Peaks | t, (min) | | Peaks | t, (min) | | Peaks | t _e (min |
| 1 | . (IS) 1,4-Dioxane-d8 | [^] 2.30 | 30. Acetophenone | [^] 5.25 | 58. | 1,2,4,5-Tetrachlorobenzene | 7.29 | 87. | 2-Naphthalamine | [°] 8.79 | 116. | (SS) Pyrene-D10 | 11.90 |
| 2 | . 1,4-Dioxane | 2.32 | 31. 4-Nitrosomorpholine | 5.27 | 59. | Isosafrole | 7.34 | 88. | Diethyl phthalate | 8.90 | 117. | Pyrene | 11.92 |
| | . N-Nitrosodimethylamine | 2.52 | 32. o-Toluidine | 5.29 | 60. | 2,4,6-Trichlorophenol | 7.43 | 89. | Fluorene | 9.01 | 118. | (SS) p-Terphenyl-d14 | 12.13 |
| 4 | . Pyridine | 2.56 | 33. Hexachloroethane | 5.37 | 61. | 2,4,5-Trichlorophenol | 7.47 | | 4-Chlorophenyl phenyl eth | er 9.03 | 119. | Aramite-1 | 12.13 |
| 5 | . Ethyl methacrylate | 2.85 | 34. (SS) Nitrobenzene-D5 | 5.42 | 62. | (SS) 2-Fluorobiphenyl | 7.54 | 91. | 2-Methyl-5-nitroaniline | 9.03 | 120. | Aramite-2 | 12.22 |
| 6 | . 2-Picoline | 3.10 | 35. Nitrobenzene | 5.44 | 63. | Safrole | 7.62 | 92. | 4-Nitroaniline | 9.03 | 121. | Dimethylaminoazobenzene | 12.31 |
| 7 | . N-Nitrosomethylethylamine | 3.16 | 36. N-Nitrosopiperidine | 5.60 | 64. | Biphenyl | 7.65 | 93. | 4,6-Dinitro-2-methylpheno | l 9.08 | 122. | 4,4'-Dichlorobenzilate | 12.37 |
| 8 | . Methyl methanesulfonate | 3.42 | 37. Isophorone | 5.71 | 65. | 2-Chloronaphthalene | 7.67 | 94. | N-Nitrosodiphenylamine | 9.17 | 123. | 3,3'-Dimethylbenzidine | 12.72 |
| 9 | . (SS) 2-Fluorophenol | 3.56 | 38. 2-Nitrophenol | 5.80 | 66. | 1-Chloronaphthalene | 7.70 | 95. | N,N-Diphenylhydrazine | 9.22 | 124. | Butyl benzyl phthalate | 12.75 |
| 10 | . N-Nitrosodiethylamine | 3.77 | 39. 2,4-Dimethylphenol | 5.85 | 67. | Diphenyl ether | 7.79 | 96. | (SS) 2,4,6-Tribromophenol | 9.30 | 125. | Kepone | 12.77 |
| 11 | . Ethyl methanesulfonate | 4.04 | 40. Benzoic acid | 5.91 | | 2-Nitroaniline | 7.79 | 97. | 1,3,5-Trinitrobenzene | 9.49 | 126. | Bis(2-ethylhexyl) adipate | 12.88 |
| 12 | . Benzaldehyde | 4.38 | 41. Bis(2-chloroethoxy)methane | 5.96 | 69. | 1,4-Naphthoquinone | 7.88 | 98. | Diallate | 9.54 | 127. | 2-(Acetylamino)fluorene | 13.04 |
| 13 | . (SS) Phenol-d6 | 4.42 | 42. 2,4-Dichlorophenol | 6.07 | | 1,2-Dinitrobenzene | 7.97 | 99. | Phenacetin | 9.55 | 128. | 3,3'-Dichlorobenzidine | 13.43 |
| 14 | . Phenol | 4.44 | 43. 1,2,4-Trichlorobenzene | 6.18 | 71. | Dimethyl phthalate | 8.03 | 100. | 4-Bromophenyl phenyl eth | er 9.62 | 129. | Benz[a]anthracene | 13.46 |
| 15 | . Aniline | 4.48 | 44. (IS) Naphthalene-D8 | 6.24 | 72. | 1,3-Dinitrobenzene | 8.05 | 101. | Hexachlorobenzene | 9.69 | 130. | (IS) Chrysene-D12 | 13.47 |
| 16 | . Bis(2-chloroethyl) ether | 4.54 | 45. Naphthalene | 6.27 | 73. | 2,6-Dinitrotoluene | 8.10 | 102. | Atrazine | 9.83 | 131. | Chrysene | 13.51 |
| 17 | . Pentachloroethane | 4.54 | 46. 4-Chloroaniline | 6.33 | 74. | 1,4-Dinitrobenzene | 8.15 | 103. | Pentachlorophenol | 9.93 | 132. | Bis(2-ethylhexyl) phthalate | 13.56 |
| 18 | . 2-Chlorophenol | 4.60 | 47. 2,6-Dichlorophenol | 6.34 | 75. | Acenaphthylene | 8.17 | 104. | 4-Aminobiphenyl | 9.94 | 133. | Di-n-octyl phthalate | 14.58 |
| 19 | . 1,3-Dichlorobenzene | 4.77 | 48. Hexachloropropene | 6.37 | 76. | 3-Nitroaniline | 8.29 | 105. | Pentachloronitrobenzene | 9.94 | 134. | Benzo[b]fluoranthene | 15.14 |
| 20 | . (IS) 1,4-Dichlorobenzene-D4 | 4.83 | 49. Hexachlorobutadiene | 6.42 | 77. | (IS) Acenaphthene-d10 | 8.35 | 106. | Propyzamide | 10.03 | 135. | 7,12-Dimethylbenzo[a]anthr | acene |
| 21 | . 1,4-Dichlorobenzene | 4.85 | α,α-Dimethylphenethylami | ne | 78. | Acenaphthene | 8.39 | 107. | (IS) Phenanthrene-D10 | 10.16 | | | 15.14 |
| 22 | . Benzyl alcohol | 4.96 | | 6.43 | 79. | 2,4-Dinitrophenol | 8.42 | 108. | Phenanthrene | 10.19 | 136. | Benzo[k]fluoranthene | 15.19 |
| 23 | . 1,2-Dichlorobenzene | 5.01 | 51. Caprolactam | 6.71 | 80. | 4-Nitrophenol | 8.50 | 109. | Anthracene | 10.25 | 137. | Benzo[a]pyrene | 15.70 |
| 24 | . 2-Methylphenol | 5.08 | 52. N-Nitroso-N-butylamine | 6.74 | 81. | Pentachlorobenzene | 8.55 | 110. | Carbazole | 10.45 | 138. | (IS) Perylene-D12 | 15.80 |
| 25 | . Bis(2-chloroisopropyl)ether | 5.12 | 53. 4-Chloro-3-methylphenol | 6.91 | 82. | 2,4-Dinitrotoluene | 8.58 | 111. | di-n-Butyl phthalate | 10.91 | 139. | 3-Methylcholanthrene | 16.35 |
| 26 | . Nitrosopyrrolidine | 5.22 | 54. Isosafrole | 6.99 | 83. | Dibenzofuran | 8.60 | 112. | 4-Nitroquinoline 1-oxide | 11.13 | 140. | Dibenz[a,j]acridine | 17.46 |
| 27 | . 4-Methylphenol | 5.24 | 55. 2-Methylnaphthalene | 7.09 | 84. | 1-Naphthalamine | 8.69 | 113. | Isodrin | 11.46 | 141. | Indeno[1,2,3-cd]pyrene | 17.78 |
| 28 | . 3-Methylphenol | 5.24 | 56. 1-Methylnaphthalene | 7.21 | 85. | 2,3,5,6-Tetrachlorophenol | 8.69 | 114. | Fluoranthene | 11.64 | 142. | Dibenz[a,h]anthracene | 17.84 |
| 29 | . N-Nitrosodi-N-propylamine | 5.25 | 57. Hexachlorocyclopentadiene | 7.28 | 86. | 2,3,4,6-Tetrachlorophenol | 8.75 | 115. | Benzidine | 11.82 | 143. | Benzo[ghi]perylene | 18.27 |
| | | | | | | | | | | | | | |

Diesel Particulate Extract Injections

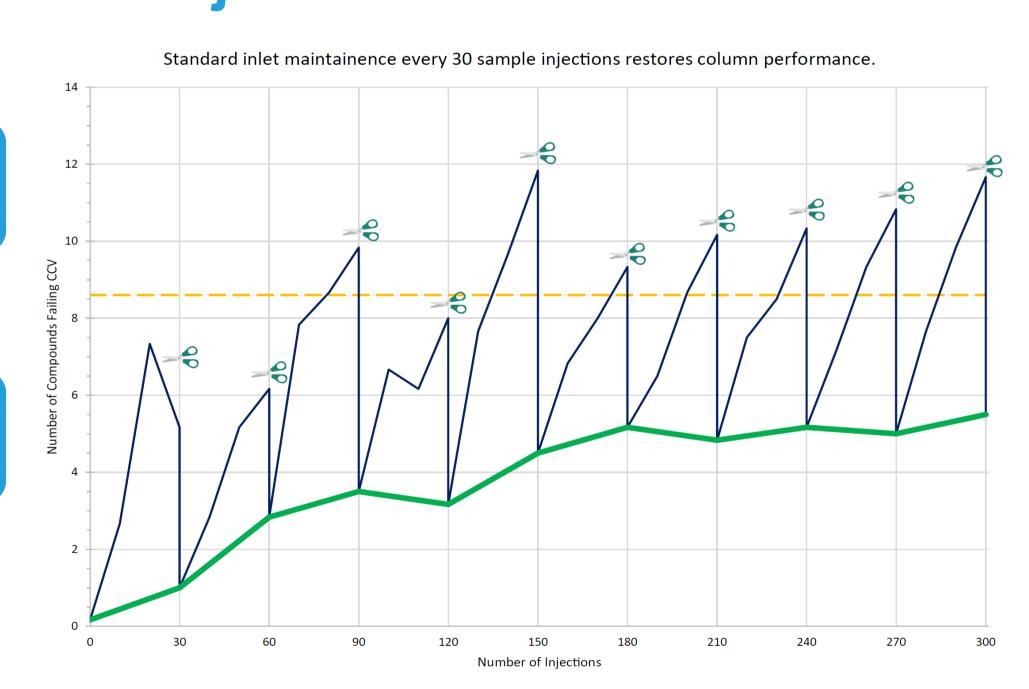
300 injections of a diesel particulate extract (NIST SRM 1975)

Calibration Verification (CCV) performed every 10 SRM injections

 Maintenance (2 loops removed, new liner, new seal) every 30 SRMinjections
 After 300 injections, CCV still

6.4% of targets analytes fall outside the ± 20% recovery window

meets expected peformance



Fast GC Method Translation Preserves Elution Profiles

