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

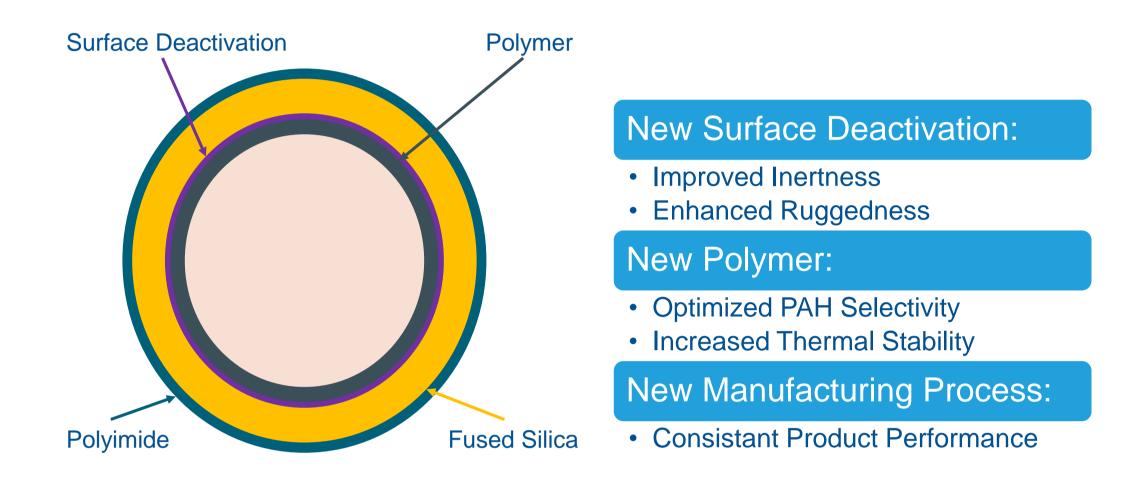
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Outline

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.

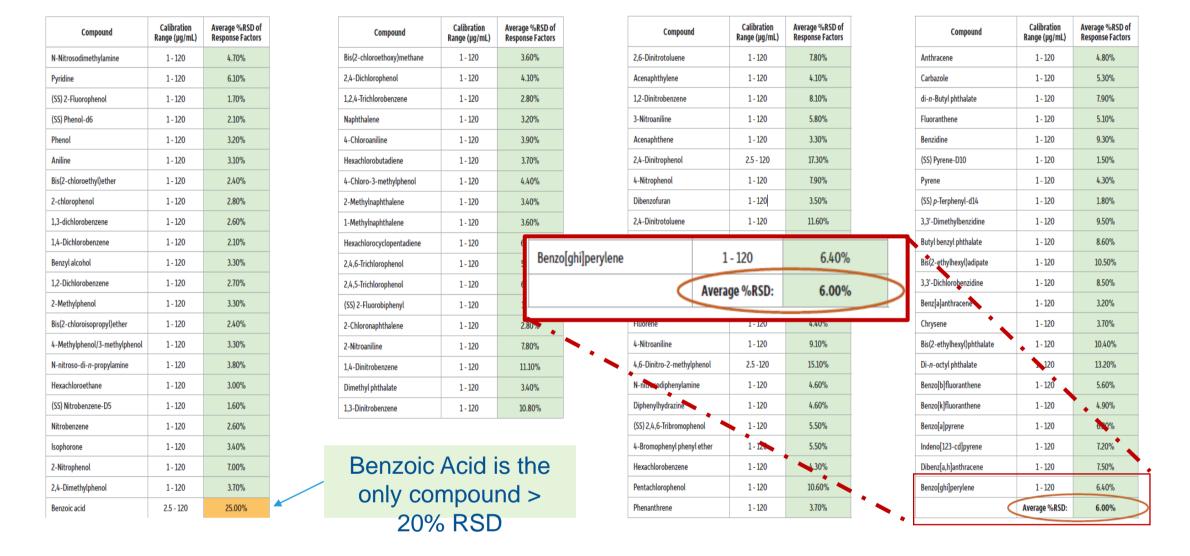
This presentation will review the details of the lifetime study results using contaminated extracts and will highlight the advantages of this column using a new deactivation and stationary phase.

Enhanced SVOC Performance



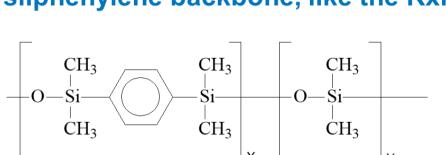
Inertness = Linear Calibrations

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

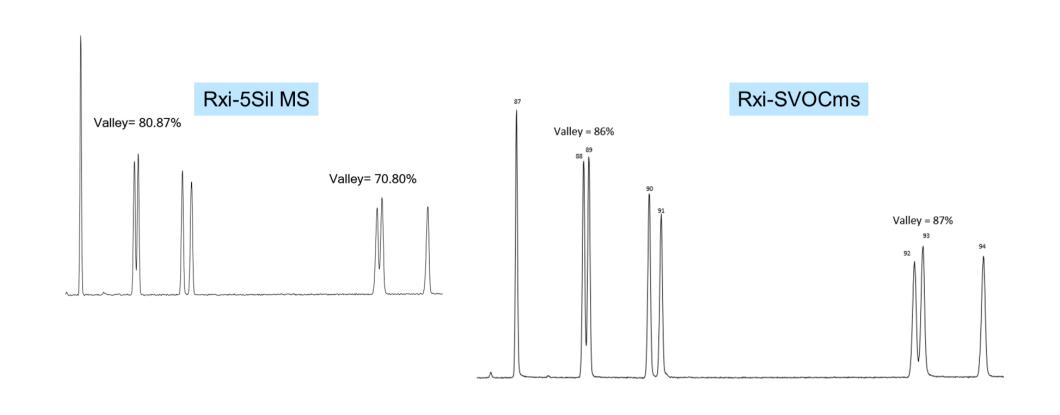
Similar phase chemistries DB-UI 8270D for Semivolatiles **ZB-Semivolatiles**

SLB-5ms Semivolatiles Other Silphenylene type phases Rxi-5Sil MS DB-5ms

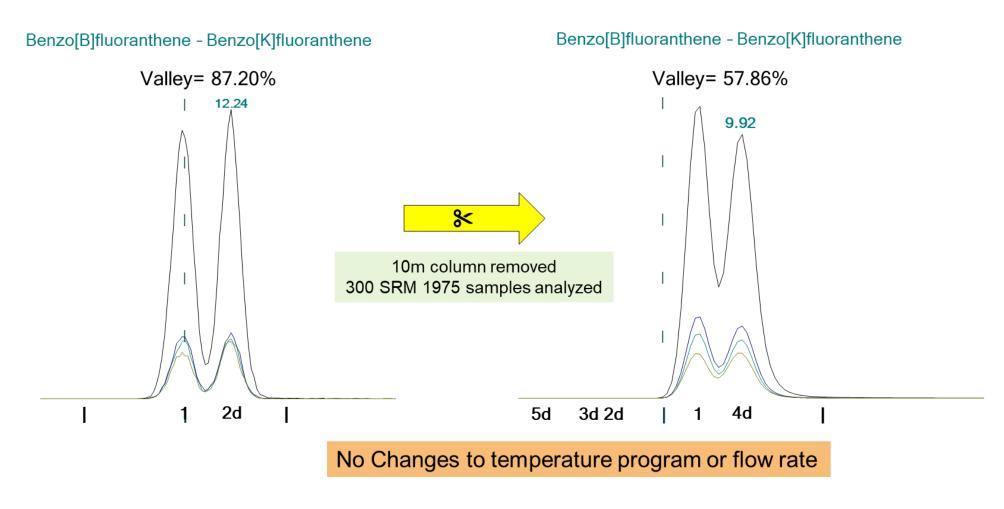
DB-5ms UI VF-ms BPX-5 ZB-5ms CP-Sil 8 CB LB/MS Optima-5ms PE-5ms

Optimized PAH Selectivity

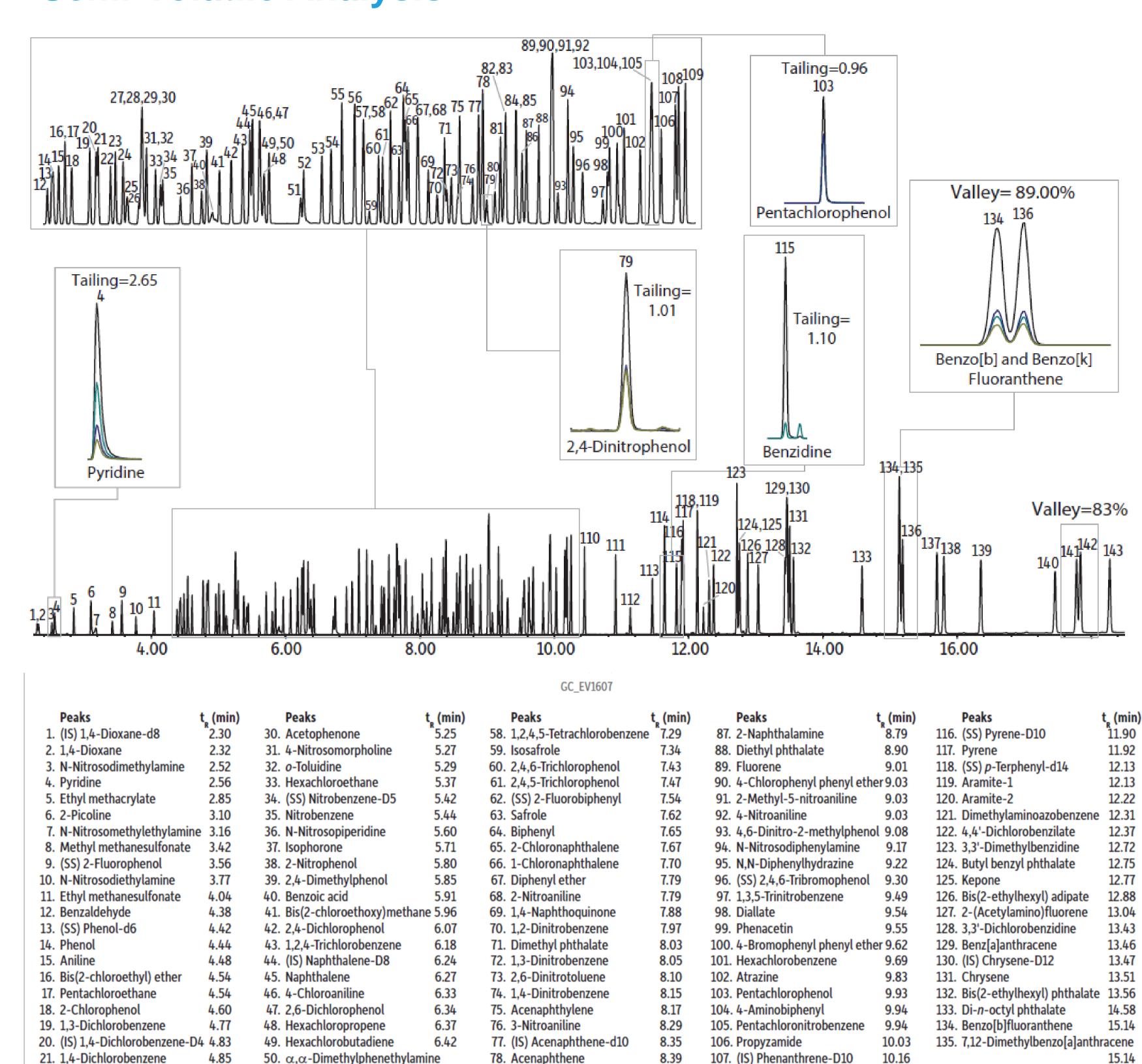
Improved Benzofluoranthene and Indeno[123-cd]pyrene – Dibenz[ah]anthracene Separations on the Rxi-SVOCms



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



79. 2.4-Dinitrophenol

81. Pentachlorobenzene

85. 2,3,5,6-Tetrachlorophenol

86. 2,3,4,6-Tetrachlorophenol

82. 2.4-Dinitrotoluene

80. 4-Nitrophenol

83. Dibenzofuran

84. 1-Naphthalamine

Diesel Particulate Extract Injections

57. Hexachlorocyclopentadiene 7.28

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

4.96

5.01

5.24

51. Caprolactam

54. Isosafrole

52. N-Nitroso-N-butylamine

55. 2-Methylnaphthalene

53. 4-Chloro-3-methylphenol

21. 1,4-Dichlorobenzene

23. 1,2-Dichlorobenzene

25. Bis(2-chloroisopropyl)ether 5.12

29. N-Nitrosodi-N-propylamine 5.25

22. Benzyl alcohol

24. 2-Methylphenol

26. Nitrosopyrrolidine

27. 4-Methylphenol

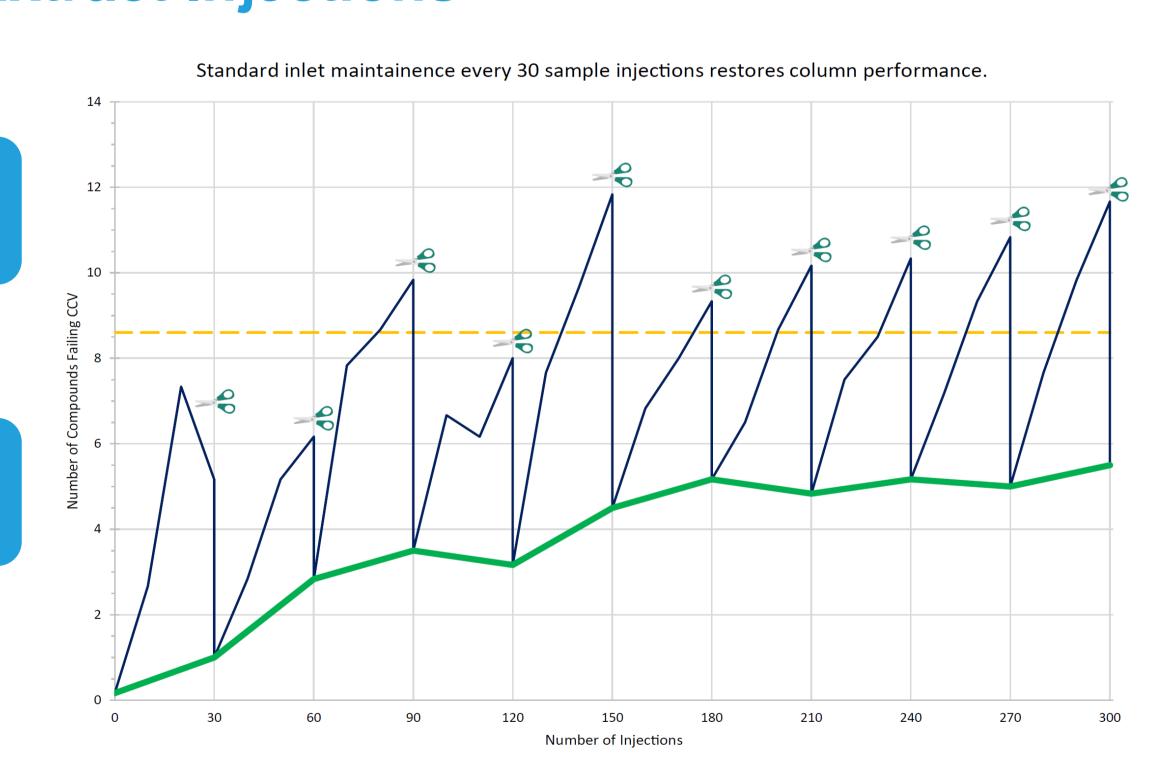
28. 3-Methylphenol

- Calibration Verification (CCV) performed
- every 10 SRM injections Maintenance (2 loops removed, new
- liner, new seal) every 30 SRMinjections

After 300 injections, CCV still meets expected peformance targets

• 6.4% of targets analytes fall outside the

± 20% recovery window



108. Phenanthrene

114. Fluoranthene

115. Benzidine

111. di-n-Butyl phthalate

112. 4-Nitroquinoline 1-oxide

109. Anthracene

110. Carbazole

113. Isodrin

136. Benzo[k]fluoranthene

139. 3-Methylcholanthrene

141. Indeno[1,2,3-cd]pyrene

142. Dibenz[a,h]anthracene

18.27

143. Benzo[ghi]perylene

137. Benzo[a]pyrene

138. (IS) Perylene-D12

140. Dibenz[a,j]acridine

Fast GC Method Translation Preserves Elution Profiles

