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TECHNICAL ARTICLE

MARKET FOCUSED - environmental

Analysis of Volatile Compounds

on Various Phases

Volatile organic compounds (VOC) are amongst the most common contaminants in water supplies in industrialised countries. These contaminants enter the ground water from a variety of sources such as leakage's from underground tanks to industrial solvents used in septic system cleaners. They have a variety of harmful health concerns varying from affecting the central nervous system to carcinogenic properties. For this reason there are a number of US Environmental Protection Agency (EPA) methods (502.2 and 624 among others) set up to monitor these common contaminants in drinking and waste water supply.

ANALYSIS

Accurate identification and quantitation of all 60 compounds and internal standards of the US EPA Method 502.2 and the 31 compounds and internal standards in the US EPA Method 624 is a challenging task while still maintaining a high sample throughput. Most volatile columns available on the market today are thick film, high-bleed, low temperature columns. With maximum temperatures of 220-240°C, the flexibility of these columns to perform higher boiling point analyses is limited. This may lead to higher boiling contaminants being left on the column which may affect reproducibility from injection to injection resulting in a loss of productivity.

CAPILLARY COLUMN CHOICE

There is a wide choice of capillary column for the analysis of volatile organic compounds. Thick film columns are generally considered a necessity for dealing with the extremely volatile nature of components such as gases and low boiling liquids. As such, film thicknesses usually start at the 1.0µm and can be anywhere up to 3.0µm thick. This causes longer analysis times due to the extended elution times required to elute higher boiling components such as naphthalene and trichlorobenzene. Many of these methods have had columns designed specifically to perform these analyses such as the SGE cyanopropylphenyl BPX624 column. Part of this design process is to obtain the correct phase ratios for such analyses. Phase ratios reflect the ability of a column to retain solutes. A lower phase ratio will retain lower boiling solvents more efficiently than that of the higher phase ratios, which tend to have a lower capacity and are more suitable for higher boiling component analyses. Phase ratios tend to range from 40-500 with 250 being a general-purpose type of column. The phase ratio can be determined by the equation below:

β = internal diameter / 4 x stationary phase film thickness

The BPX624 column has been designed with the US EPA 502.2 and 624 methods in mind and with a phase ratio of 44 this column gives superior separation of the various volatile components (Figures 1 and 4). With an upper temperature limit of 280/290°C it has by far the highest thermal stability of any specifically designed volatiles column on the market. The thick film 5% phenyl BPX5 column from SGE with a phase ratio of 62 is also an excellent choice for the analysis of volatile mixtures. With an upper temperature limit of 360°C, the BPX5 column gives excellent flexibility in the range of analyses that can be performed on this column (Figures 2 and 5). The elution order changes also make this an excellent confirmation column to standard volatile columns such as BPX624. The new generation SolGel-WAX column from SGE has an almost completely different elution order to the previous two columns making this column the perfect confirmation column to both the BPX624 and BPX5. The thin film on this SolGel-WAX column gives a phase ratio of 250 which doesn't affect the excellent separation capabilities of the extremely volatile gases and low boiling point components of the US EPA 502.2 and 624 mixes. With a maximum temperature limit of 300°C it can easily handle the temperature requirements for these two methods (Figures 3 and 6).

FIGURE 1

Note the exceptional separation of the various components on the BPX624 column. The excellent peak shape indicates a high degree of inertness of the column making this the first choice for the analysis of the volatile components in water.

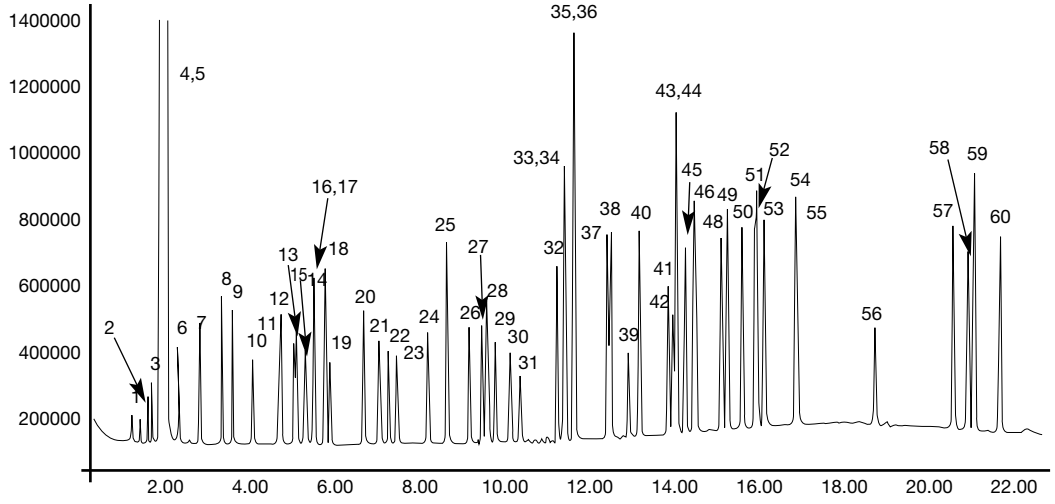
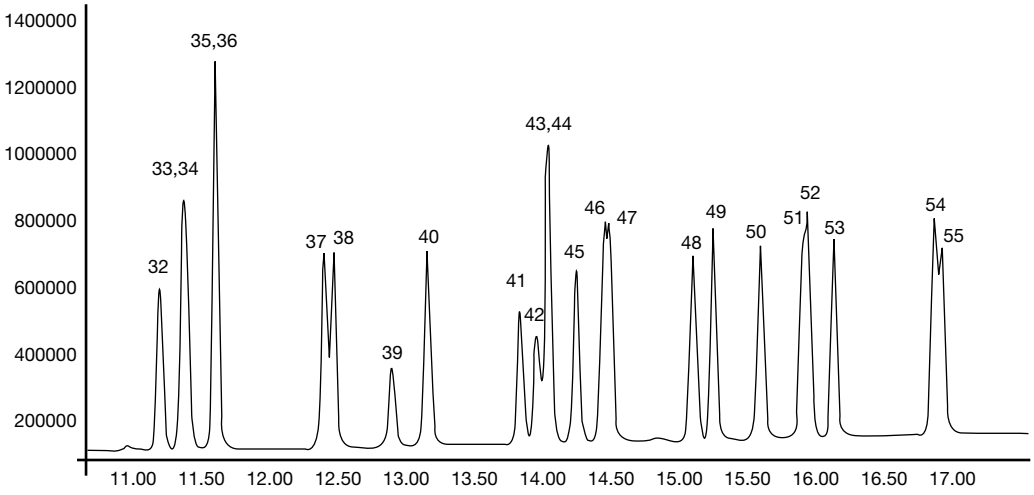


FIGURE 1A

An expanded view of the components 32 to 55 on BPX624



Phase:	**BPX624 1.4µm film**
USEPA 502.2 mix:	**200 ppm in Methanol**
Column:	**30m x 0.25 mm ID**
Initial Temp:	40°C, 0 min.
Rate 1:	6°C to 210°C
Rate 2:	15°C to 240°C
Final Temp:	240°C, 5 min.
Detector Type:	Mass Spectrometer
Carrier Gas:	He, 22.8
Carrier Gas Flow:	1.3 mL/min.
Constant Flow:	On
Injection Mode:	Split
Split Ratio:	50:1
Injection Volume:	1 µL
Injection Temperature:	250°C
Autosampler:	No
Liner Type:	4 mm ID Single Taper Liner
Liner Part Number:	092017
Column Part Number:	054860
ms-NoVent™ Part no.:	113400
HP5973 restrictor:	113409
Full scan:	25-450

COMPOUNDS

1. Dichlorodifluoromethane
2. Chloromethane
3. Vinyl chloride
4. Bromomethane
5. Chloroethane
6. Trichlorofluoromethane
7. 1,1-Dichloroethene
8. Dichloromethane
9. trans-1,2-Dichloroethene
10. 1,1-Dichloroethane
11. 2,2-Dichloropropane
12. cis-1,2-Dichloroethene
13. Bromochloromethane
14. Chloroform
15. 1,1,1-Trichloroethane
16. 1,1-Dichloropropene
17. Carbon tetrachloride
18. Benzene
19. 1,2-Dichloroethane
20. Trichloroethene
21. 1,2-Dichloropropane
22. Dibromomethane
23. Bromodichloromethane
24. cis-1,3-Dichloropropene
25. Toluene
26. trans-1,3-Dichloropropene
27. 1,1,2-Trichloroethane
28. Tetrachloroethene
29. 1,3-Dichloropropane
30. Dibromochloromethane
31. 1,2-Dibromoethane
32. Chlorobenzene
33. Ethylbenzene
34. 1,1,1,2-Tetrachloroethane
35. m-Xylene
36. p-Xylene
37. o-Xylene
38. Styrene
39. Bromoform
40. Isopropylbenzene
41. Bromobenzene
42. 1,1,2,2-Tetrachloroethane
43. 1,2,3-Trichloropropane
44. n-Propyl benzene
45. 2-Chlorotoluene
46. 1,3,5-Trimethylbenzene
47. 4-Chlorotoluene
48. tert-Butylbenzene
49. 1,2,4-Trimethylbenzene
50. sec-Butylbenzene
51. 1,3-Dichlorobenzene
52. p-Isopropyltoluene
53. 1,2-Dichlorobenzene
54. n-Butylbenzene
55. 1,4-Dichlorobenzene
56. 1,2-Dibromo-3-chloropropane
57. 1,2,4-Trichlorobenzene
58. Hexachlorobutadiene
59. Naphthalene
60. 1,2,3-Trichlorobenzene

FIGURE 2

Analysis of the 502.2 mix analysed on a thick film BPX5 column. The separation of the various components is exceptional with only 3 co-elutions in the 60 component mixture.

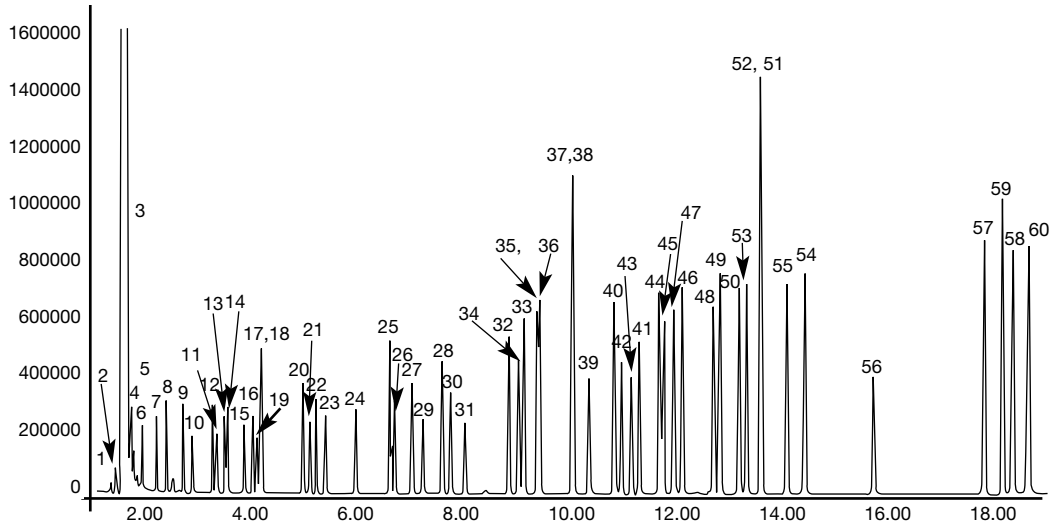
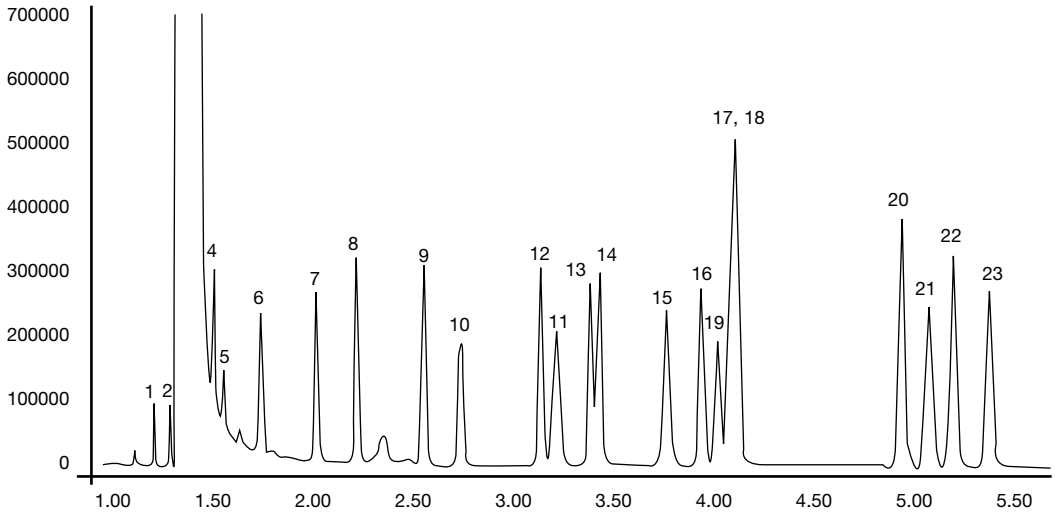


FIGURE 2A

Expanded view of comonents 1-23 of the US EPA 624 mix on BPX5.



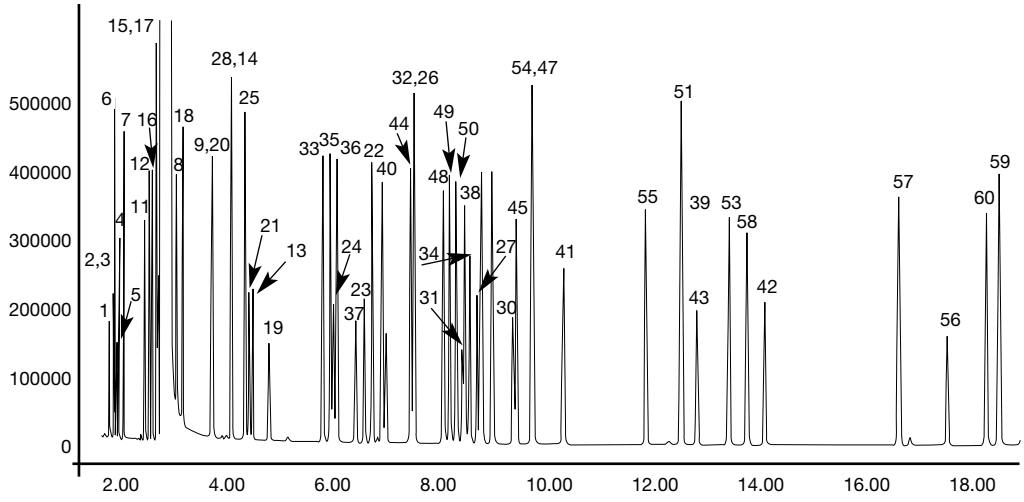
Phase:	**BPX5 1.0µm film**	Injection Mode:	Split
USEPA 502.2 mix:	**200 ppm in Methanol**	Split Ratio:	50:1
Column:	**30m x 0.25 mm ID**	Injection Volume:	1 µL
Initial Temp:	40°C, 0 min.	Injection Temperature:	250°C
Rate 1:	6°C to 210°C	Autosampler:	No
Rate 2:	15°C to 240°C	Liner Type:	4 mm ID Single Taper Liner
Final Temp:	240°C, 5 min.	**Liner Part Number:**	**092017**
Detector Type:	Mass Spectrometer	**Column Part Number:**	**054122**
Carrier Gas:	He, 22.8	ms-NoVent™ Part no.:	113400
Carrier Gas Flow:	1.3 mL/min.	HP5973 restrictor:	113409
Constant Flow:	On	Full scan:	25-450

COMPOUNDS

1. Dichlorodifluoromethane
2. Chloromethane
3. Vinyl chloride
4. Bromomethane
5. Chloroethane
6. Trichlorofluoromethane
7. 1,1-Dichloroethene
8. Dichloromethane
9. trans-1,2-Dichloroethene
10. 1,1-Dichloroethane
11. 2,2-Dichloropropane
12. cis-1,2-Dichloroethene
13. Bromochloromethane
14. Chloroform
15. 1,1,1-Trichloroethane
16. 1,1-Dichloropropene
17. Carbon tetrachloride
18. Benzene
19. 1,2-Dichloroethane
20. Trichloroethene
21. 1,2-Dichloropropane
22. Dibromomethane
23. Bromodichloromethane
24. cis-1,3-Dichloropropene
25. Toluene
26. trans-1,3-Dichloropropene
27. 1,1,2-Trichloroethane
28. Tetrachloroethene
29. 1,3-Dichloropropane
30. Dibromochloromethane
31. 1,2-Dibromoethane
32. Chlorobenzene
33. Ethylbenzene
34. 1,1,1,2-Tetrachloroethane
35. m-Xylene
36. p-Xylene
37. o-Xylene
38. Styrene
39. Bromoform
40. Isopropylbenzene
41. Bromobenzene
42. 1,1,2,2-Tetrachloroethane
43. 1,2,3-Trichloropropane
44. n-Propyl benzene
45. 2-Chlorotoluene
46. 1,3,5-Trimethylbenzene
47. 4-Chlorotoluene
48. tert-Butylbenzene
49. 1,2,4-Trimethylbenzene
50. sec-Butylbenzene
51. 1,3-Dichlorobenzene
52. p-Isopropyltoluene
53. 1,2-Dichlorobenzene
54. n-Butylbenzene
55. 1,4-Dichlorobenzene
56. 1,2-Dibromo-3-chloropropane
57. 1,2,4-Trichlorobenzene
58. Hexachlorobutadiene
59. Naphthalene
60. 1,2,3-Trichlorobenzene

FIGURE 3

Analysis of the 502.2 mixed on a standard dimension SolGel-WAX column. Note the completely different elution orders of this column compared to the BPX624 and BPX5.



Phase:	**SolGel-WAX 0.25 µm film**	Injection Mode:	Split
USEPA 502.2 mix:	**200 ppm in Methanol**	Split Ratio:	50:1
Column:	**30m x 0.25 mm ID**	Injection Volume:	1 µL
Initial Temp:	40°C, 0 min.	Injection Temperature:	250°C
Rate 1:	6°C to 210°C	Autosampler:	No
Rate 2:	15°C to 240°C	Liner Type:	4 mm ID Single Taper Liner
Final Temp:	240°C, 5 min.	**Liner Part Number:**	**092017**
Detector Type:	Mass Spectrometer	**Column Part Number:**	**054796**
Carrier Gas:	He, 22.8	ms-NoVent™ Part no.:	113400
Carrier Gas Flow:	1.3 mL/min.	HP5973 restrictor:	113409
Constant Flow:	On	Full scan:	25-450

COMPOUNDS

1. Dichlorodifluoromethane
2. Chloromethane
3. Vinyl chloride
4. Bromomethane
5. Chloroethane
6. Trichlorofluoromethane
7. 1,1-Dichloroethene
8. Dichloromethane
9. trans-1,2-Dichloroethene
10. 1,1-Dichloroethane
11. 2,2-Dichloropropane
12. cis-1,2-Dichloroethene
13. Bromochloromethane
14. Chloroform
15. 1,1,1-Trichloroethane
16. 1,1-Dichloropropene
17. Carbon tetrachloride
18. Benzene
19. 1,2-Dichloroethane
20. Trichloroethene
21. 1,2-Dichloropropane
22. Dibromomethane
23. Bromodichloromethane
24. cis-1,3-Dichloropropene
25. Toluene
26. trans-1,3-Dichloropropene
27. 1,1,2-Trichloroethane
28. Tetrachloroethene
29. 1,3-Dichloropropane
30. Dibromochloromethane
31. 1,2-Dibromoethane
32. Chlorobenzene
33. Ethylbenzene
34. 1,1,1,2-Tetrachloroethane
35. m-Xylene
36. p-Xylene
37. o-Xylene
38. Styrene
39. Bromoform
40. Isopropylbenzene
41. Bromobenzene
42. 1,1,2,2-Tetrachloroethane
43. 1,2,3-Trichloropropane
44. n-Propyl benzene
45. 2-Chlorotoluene
46. 1,3,5-Trimethylbenzene
47. 4-Chlorotoluene
48. tert-Butylbenzene
49. 1,2,4-Trimethylbenzene
50. sec-Butylbenzene
51. 1,3-Dichlorobenzene
52. p-Isopropyltoluene
53. 1,2-Dichlorobenzene
54. n-Butylbenzene
55. 1,4-Dichlorobenzene
56. 1,2-Dibromo-3-chloropropane
57. 1,2,4-Trichlorobenzene
58. Hexachlorobutadiene
59. Naphthalene
60. 1,2,3-Trichlorobenzene

FIGURE 3A

Expanded view of the front end of the chromatogram of the 502.2 mix. Note the excellent separation of the gases and volatile components in the front end of the chromatogram.

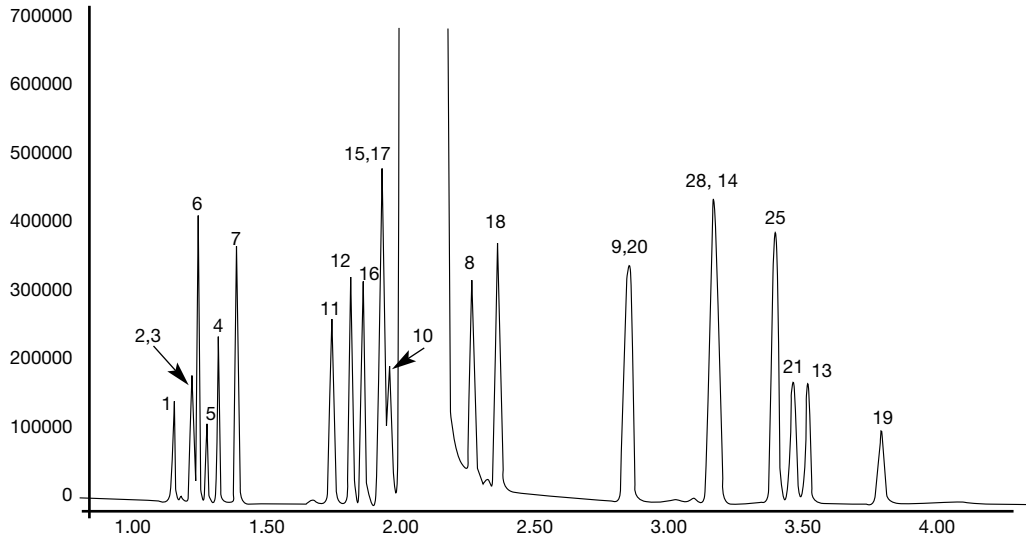


FIGURE 3B

Expanded view of the middle section of the chromatogram of the 502.2 mix on SolGel-WAX. Note the separation of the difficult to separate xylene isomers.

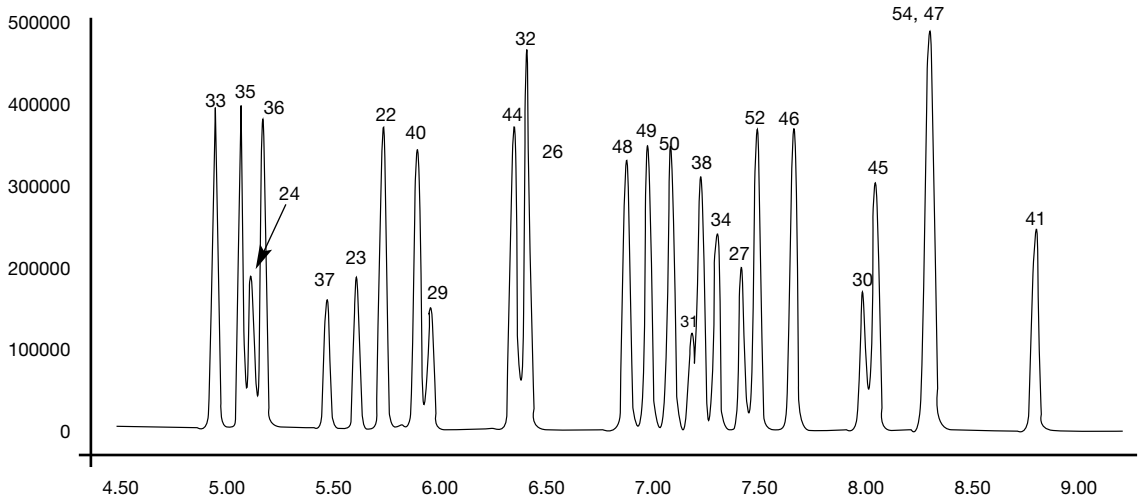
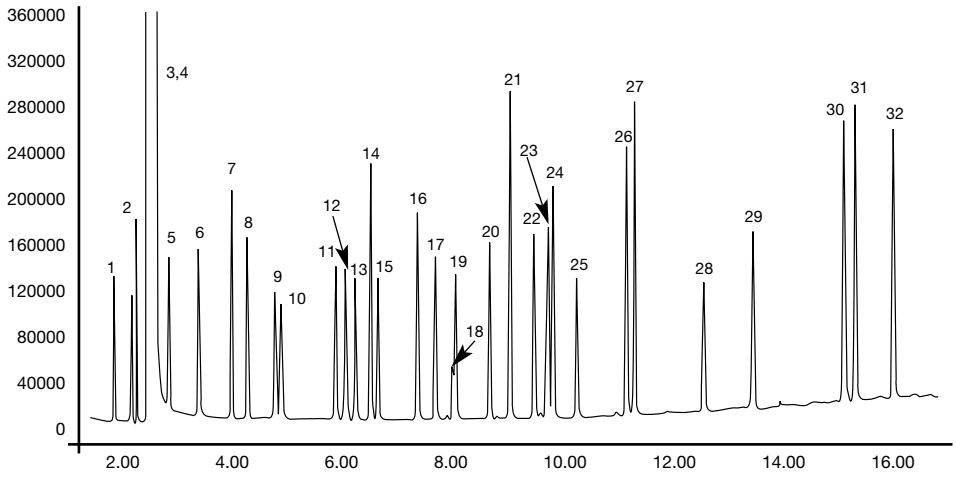


FIGURE 4

US EPA 624 mix on BPX 624. Note the excellent separation on BPX624. There are no co-elution's in this chromatogram.

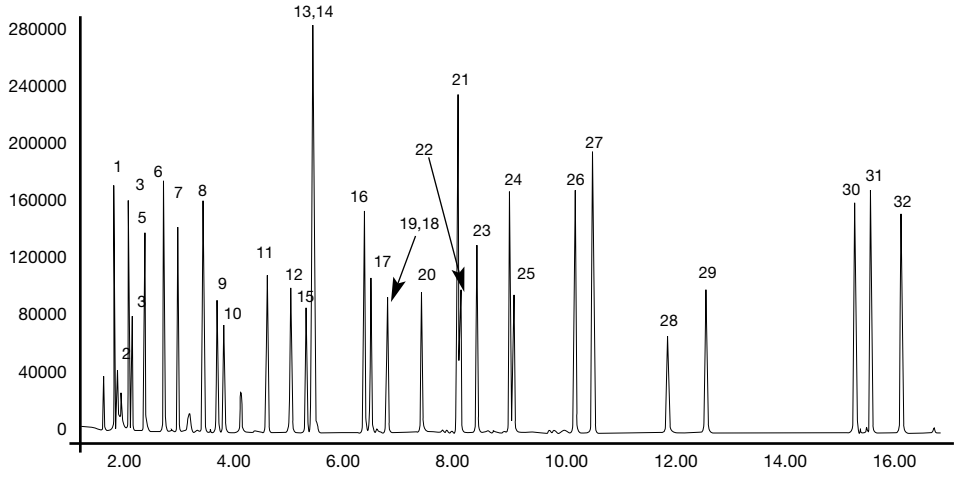


Components
1. Chloromethane
2. Vinyl chloride
3. Chloroethane
4. Bromomethane
5. Trichlorofluoromethane
6. 1,1-Dichloroethene
7. Dichloromethane
8. 1,2-Dichloroethene (trans)
9. 1,1,-Dichloroethane
10. Unknown
11. Chloroform
12. 1,1,1-Trichloroethane
13. Carbon tetrachloride
14. Benzene
15. 1,2-Dichloroethane
16. Trichloroethene
17. 1,2-Dichloropropane
18. 2-Chloroethylvinyl ether
19. Bromodichloromethane
20. 1,3-Dichloropropene (Z)
21. Toluene
22. 1,3-Dichloropropene (E)
23. 1,1,2-Trichloroethane
24. Tetrachloroethene
25. Dibromochloromethane
26. Chlorobenzene
27. Ethylbenzene
28. Bromoform
29. 1,1,2,2-Tetrachloroethane
30. 1,3-Dichlorobenzene
31. 1,4-Dichlorobenzene
32. 1,2-Dichlorobenzene

Phase:	**BPX624, 1.4µm film**	Injection Mode:	Split
Sample:	**100ppm in Hexane/Toluene**	Split Ratio:	40:1
Column:	**30m x 0.25 mm ID**	Injection Volume:	1 mL
Initial Temp:	40°C, 3 min.	Injection Temperature:	250°C
Rate 1:	8°C/min to 90°C,	Autosampler:	No
Rate 2:	6°C/min to 200°C	Liner Type:	4 mm ID Single Taper Liner
Final Temp:	200°C, 5 min.		
Detector Type:	Mass Spectrometer	**Liner Part Number:**	**092017**
Carrier Gas:	He, 25.7 psi	**Column Part Number:**	**054860**
Carrier Gas Flow:	1.8 mL/min.	ms-NoVent™ Part no.:	113400
Constant Flow:	On	HP5973 restrictor:	113409
Average Linear	Velocity:35 cm/sec at 40°C	Full Scan / SIM:	Full scan 45-450

FIGURE 5

US EPA 624 mix on BPX5. Note the slight changes in elution order to that of BPX624 making this an excellent confirmation column for this type of analysis.

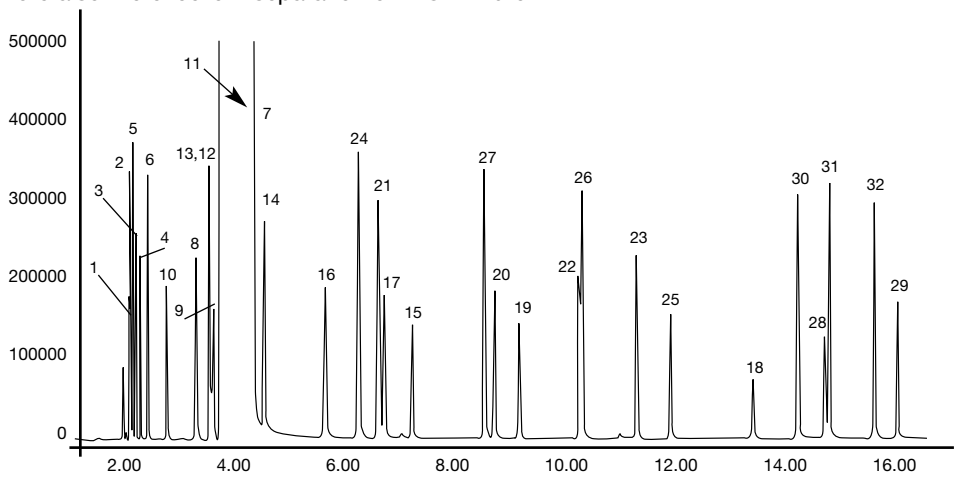


Phase:	**BPX5, 1.0µm film**	Injection Mode:	Split
Sample:	**100ppm in Hexane/Toluene**	Split Ratio:	40:1
Column:	**30m x 0.25 mm ID**	Injection Volume:	1 mL
Initial Temp:	40°C, 3 min.	Injection Temperature:	250°C
Rate 1:	8°C/min to 90°C,	Autosampler:	No
Rate 2:	6°C/min to 200°C	Liner Type:	4 mm ID Single Taper Liner
Final Temp:	200°C, 5 min.		
Detector Type:	Mass Spectrometer	**Liner Part Number:**	**092017**
Carrier Gas:	He, 25.7 psi	**Column Part Number:**	**054122**
Carrier Gas Flow:	1.8 mL/min.	ms-NoVent™ Part no.:	113400
Constant Flow:	On	HP5973 restrictor:	113409
Average Linear	Velocity:35 cm/sec at 40°C	Full Scan / SIM:	Full scan 45-450

Components
1. Chloromethane
2. Vinyl chloride
3. Chloroethane
4. Bromomethane
5. Trichlorofluoromethane
6. 1,1-Dichloroethene
7. Dichloromethane
8. 1,2-Dichloroethene (trans)
9. 1,1,-Dichloroethane
10. Unknown
11. Chloroform
12. 1,1,1-Trichloroethane
13. Carbon tetrachloride
14. Benzene
15. 1,2-Dichloroethane
16. Trichloroethene
17. 1,2-Dichloropropane
18. 2-Chloroethylvinyl ether
19. Bromodichloromethane
20. 1,3-Dichloropropene (Z)
21. Toluene
22. 1,3-Dichloropropene (E)
23. 1,1,2-Trichloroethane
24. Tetrachloroethene
25. Dibromochloromethane
26. Chlorobenzene
27. Ethylbenzene
28. Bromoform
29. 1,1,2,2-Tetrachloroethane
30. 1,3-Dichlorobenzene
31. 1,4-Dichlorobenzene
32. 1,2-Dichlorobenzene

FIGURE 6

US EPA 624 ix on SolGel WAX. Note the completely different elution order of the 624 mix when analyzed on SolGel WAX. Note also the excellent separation of this mixture



Phase:	**SolGel-WAX, 0.25µm film**	Injection Mode:	Split
Sample:	**100ppm in Hexane/Toluene**	Split Ratio:	40:1
Column:	**30m x 0.25 mm ID**	Injection Volume:	1 mL
Initial Temp:	40°C, 3 min.	Injection Temperature:	250°C
Rate 1:	8°C/min to 90°C,	Autosampler:	No
Rate 2:	6°C/min to 200°C	Liner Type:	4 mm ID Single Taper Liner
Final Temp:	200°C, 5 min.		
Detector Type:	Mass Spectrometer	**Liner Part Number:**	**092017**
Carrier Gas:	He, 25.7 psi	**Column Part Number:**	**054796**
Carrier Gas Flow:	1.8 mL/min.	ms-NoVent™ Part no.:	113400
Constant Flow:	On	HP5973 restrictor:	113409
Average Linear	Velocity:35 cm/sec at 40°C	Full Scan / SIM:	Full scan 45-450

Components
1. Chloromethane
2. Vinyl chloride
3. Chloroethane
4. Bromomethane
5. Trichlorofluoromethane
6. 1,1-Dichloroethene
7. Dichloromethane
8. 1,2-Dichloroethene (trans)
9. 1,1,-Dichloroethane
10. Unknown
11. Chloroform
12. 1,1,1-Trichloroethane
13. Carbon tetrachloride
14. Benzene
15. 1,2-Dichloroethane
16. Trichloroethene
17. 1,2-Dichloropropane
18. 2-Chloroethylvinyl ether
19. Bromodichloromethane
20. 1,3-Dichloropropene (Z)
21. Toluene
22. 1,3-Dichloropropene (E)
23. 1,1,2-Trichloroethane
24. Tetrachloroethene
25. Dibromochloromethane
26. Chlorobenzene
27. Ethylbenzene
28. Bromoform
29. 1,1,2,2-Tetrachloroethane
30. 1,3-Dichlorobenzene
31. 1,4-Dichlorobenzene
32. 1,2-Dichlorobenzene

Ordering Information

	ID(mm) (mm)	Length (m)	Film Thickness (µm)	Temperature Limits (°C)	Part No.
BPX5	0.10	10	0.10	-40 to 360/370	054099
	0.22	12	0.25	-40 to 360/370	054112
		25	0.25	-40 to 360/370	054113
		30	0.25	-40 to 360/370	054142
		50	0.25	-40 to 360/370	054114
	0.25	15	0.25	-40 to 360/370	054100
		15	1.00	-40 to 360/370	054121
		30	0.25	-40 to 360/370	054101
		30	0.50	-40 to 360/370	0541025
		30	1.00	-40 to 360/370	054122
		60	0.25	-40 to 360/370	054102
	0.32	15	0.25	-40 to 360/370	054144
		15	1.00	-40 to 360/370	054152
		25	0.25	-40 to 360/370	054119
		25	0.50	-40 to 360/370	054125
		30	0.25	-40 to 360/370	054145
		30	0.50	-40 to 360/370	0541205
		30	1.00	-40 to 360/370	054145
		60	0.25	-40 to 360/370	054146
		60	1.00	-40 to 360/370	054154
	0.53	12	1.00	-40 to 360/370	054130
		15	1.00	-40 to 360/370	054147
		15	1.50	-40 to 350/360	0541347
		15	3.00	-40 to 350/360	054159
		25	1.00	-40 to 360/370	054131
		30	0.50	-40 to 360/370	0541345
		30	1.00	-40 to 360/370	054148
		30	1.50	-40 to 350/360	0541348
		30	3.00	-40 to 350/360	054160
		60	1.00	-40 to 360/370	054158
		60	3.00	-40 to 350/360	054164
BPX35	0.10	10	0.1	0 to 360/370	054699
	0.22	15	0.25	0 to 360/370	054713
		30	0.25	0 to 360/370	054714
	0.25	15	0.25	0 to 360/370	054700
		15	1	0 to 360/370	054703
		30	0.25	0 to 360/370	054701
		30	0.5	0 to 360/370	0547025
		30	1	0 to 360/370	054704
		60	0.25	0 to 360/370	054702
		60	1	0 to 360/370	054705
	0.32	15	0.25	0 to 360/370	054723
		15	0.5	0 to 360/370	054718
		15	1	0 to 360/370	054716
		30	0.25	0 to 360/370	054724
		30	0.5	0 to 360/370	0547158
		30	1	0 to 360/370	054717
		60	0.25	0 to 360/370	054725
	0.53	15	0.5	0 to 360/370	054734
		15	1	0 to 360/370	054736
		30	0.5	0 to 360/370	054735
		30	1	0 to 360/370	054737
BPX50	0.10	10	0.10	0 to 360/370	054740
	0.25	15	0.25	0 to 360/370	054750
		30	0.25	0 to 360/370	054751
		60	0.25	0 to 360/370	054752
	0.32	15	0.25	0 to 360/370	054760
		30	0.25	0 to 360/370	054761
	0.53	15	0.50	0 to 360/370	054770
		30	0.50	0 to 360/370	054771
		30	1.00	0 to 360/370	054772
SolGel-1ms™	0.25	30	0.25	0 to 370/380	054795
		60	0.25	0 to 370/380	054793
	0.32	30	0.25	0 to 370/380	054798
		60	0.25	0 to 370/380	054794

SUMMARY:

Analysis of the US EPA 502.2 and 624 mixes can be easily performed on BPX624, BPX5 and SolGel-WAX columns. The different degrees of polarity for each column results in different elution orders making these columns perfect confirmation columns for each of the other columns. With upper temperature limits of 280°C or greater, the added temperature limits provide much greater flexibility in the range of analyses able to be performed.

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