

Application

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Analysis of Solvents in Industrial Atmospheres by Capillary GC

Similar structures and boiling points among airborne solvents monitored in the workplace often require two GC columns that offer differing selectivities to separate and identify the solvents collected. The highly polar SUPELCOWAX 10 capillary column resolves many industrial solvents based on polarity. In contrast, the nonpolar PTE-5 capillary column resolves solvents by boiling point. The shifts in retention times resulting from the complementary separation mechanisms of the two columns can aid analysts in identifying and quantifying solvents in complex mixtures.

Key Words:

- industrial hygiene ● solvents ● SUPELCOWAX 10
- capillary columns ● PTE-5 capillary columns

OSHA regulations require the monitoring of solvent concentrations in industrial workplace atmospheres to minimize health risks to workers. The OSHA-specified procedures normally use adsorbent sampling tubes to collect and concentrate the analytes. The analytes are solvent-desorbed from the tube and analyzed using gas chromatography. Similar structures and boiling points among the solvents often require two GC columns that offer differing separation mechanisms or selectivities to separate and identify the solvents collected.

Some analysts split the sample injection to the two columns to allow simultaneous detection and analysis with two detectors. This technique can be used when a client of an environmental lab requests a complete analysis of all possible solvents present on the adsorbent tube. Other analysts use one column, confirming the analysis on a second column only when coelutions are suspected, based on the known solvent use at an industrial site.

SUPELCOWAX™ 10 and PTE™-5 capillary columns provide complementary separations of solvent mixtures. The SUPELCOWAX 10 capillary column offers a highly polar, bonded phase that resolves many industrial solvents based on polarity (Figure A). The PTE-5 capillary column has a nonpolar, poly(5% diphenyl/95% dimethylsiloxane) phase that resolves solvents approximately by boiling point (Figure B, Table 1). If you know the boiling point of a compound, you can use the chromatogram to estimate its retention time on a PTE-5 column. (Some polar compounds may elute out of boiling point order due to their interaction with the phase.)

The SUPELCOWAX 10 column provides greater retention of extremely polar compounds (e.g., alcohols) compared to the PTE-5 column, as shown by the methanol shift (peak 11). Polar compounds, which can tail or be poorly resolved on other phases, are better resolved and display better peak symmetry on the

SUPELCOWAX 10 column. The SUPELCOWAX 10 column also provides baseline separation of the o-, p-, and m-xylanes, and nearly baseline separation of the p- and m-cresols.

We recommend the combined use of the PTE-5 and SUPELCOWAX 10 columns for the most reliable identification and quantitation of solvents in complex mixtures.

Table 1. Elution Order Comparison

SUPELCOWAX 10 Column	PTE-5 Column
1. Hexane	11. Methanol (64.5°C)*
U Unknown	3. Acetone (56°C)
2. 1,1-Dichloroethylene	14. Isopropanol (82°C)
3. Acetone	2. 1,1-Dichloroethylene (32°C)
4. Methyl acetate	4. Methyl acetate (54°C)
5. trans-1,2-Dichloroethylene	15. Methylene chloride (40°C)
6. Tetrahydrofuran	5. trans-1,2-Dichloroethylene (48°C)
7. Carbon tetrachloride	U Unknown
8. 1,1,1-Trichloroethane	9. 1,1-Dichloroethane (57°C)
9. 1,1-Dichloroethane	13. Methyl ethyl ketone (79.6°C)
10. Ethyl acetate	1. n-Hexane (69°C)
11. Methanol	23. 2-Butanol (99.5°C)
12. Isopropyl acetate	10. Ethyl acetate (77°C)
13. Methyl ethyl ketone	21. Chloroform (61°C)
14. Isopropanol	29. Isobutanol (107°C)
15. Methylene chloride	6. Tetrahydrofuran (66°C)
16. Benzene	34. Methyl Cellosolve® (124.5°C)
17. Propyl acetate	8. 1,1,1-Trichloroethane (74°C)
18. Trichloroethylene	26. 1,2-Dichloroethane (84°C)
19. Methyl isobutyl ketone	16. Benzene (80°C)
20. Isobutyl acetate	33. 1-Butanol (118°C)
21. Chloroform	12. Isopropyl acetate (89°C)
22. Tetrachloroethylene	7. Carbon tetrachloride (77°C)
23. 2-Butanol	18. Trichloroethylene (87°C)
24. Toluene	25. 1,4-Dioxane (101°C)
25. 1,4-Dioxane	39. Cellosolve (136°C)
26. 1,2-Dichloroethane	17. Propyl acetate (102°C)
27. n-Butyl acetate	37. Isoamyl alcohol (113°C)
28. 2-Hexanone (MBK)	19. Methyl isobutyl ketone (116°C)
29. Isobutanol	24. Toluene (111°C)
30. Isoamyl acetate	20. Isobutyl acetate (117°C)
31. p-Xylene	42. N,N-Dimethylformamide (153°C)
32. m-Xylene	28. 2-Hexanone (MBK) (127 °C)
33. 1-Butanol	22. Tetrachloroethylene (121°C)
34. Methyl Cellosolve	27. n-Butyl acetate (126°C)
35. Amyl acetate	38. Chlorobenzene (132°C)
36. o-Xylene	31. m-Xylene (139°C)
37. Isoamyl alcohol	32. p-Xylene (139°C)
38. Chlorobenzene	30. Isoamyl acetate (142°C)
39. Cellosolve	43. Cyclohexanol (161°C)
40. Styrene	40. Styrene (145°C)
41. Cyclohexanone	41. Cyclohexanone (157°C)
42. N,N-Dimethylformamide	36. o-Xylene (144°C)
43. Cyclohexanol	44. Butyl Cellosolve (171°C)
44. Butyl Cellosolve	35. Amyl acetate (148°C)
45. 2-Methylcyclohexanol	47. 1,1,2,2-Tetrachloroethane (147°C)
46. 1,2-Dichlorobenzene	45. 2-Methylcyclohexanol (165°C)
47. 1,1,2,2-Tetrachloroethane	46. 1,2-Dichlorobenzene (172°C)
48. o-Cresol	48. o-Cresol (191°C)
49. p-Cresol	49. p-Cresol (202°C)
50. m-Cresol	50. m-Cresol (203°C)

* Boiling points

Figure A. Industrial Solvents on a SUPELCOWAX 10 Column

Column: SUPELCOWAX 10, 30m x 0.53mm ID, 1.0 μ m film
 Cat. No.: 25301-U
 Oven: 40°C (5 min) to 200°C at 5°C/min
 Carrier: helium, 5mL/min
 Det.: FID
 Inj.: 1 μ L mixed solvents, split 50:1

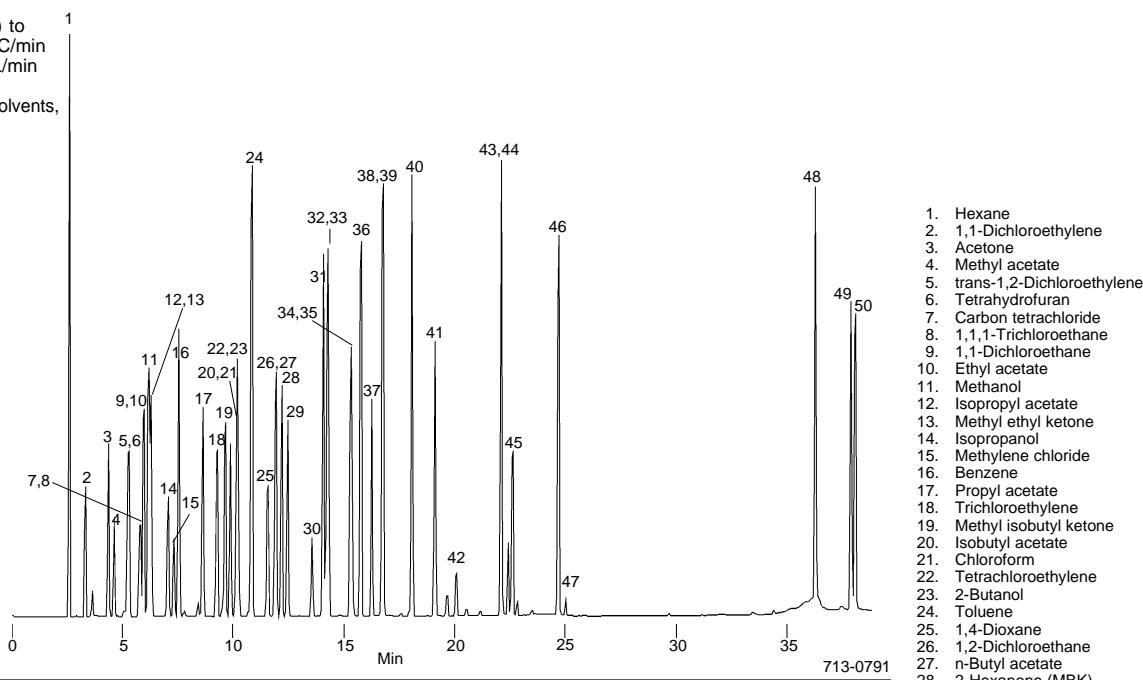
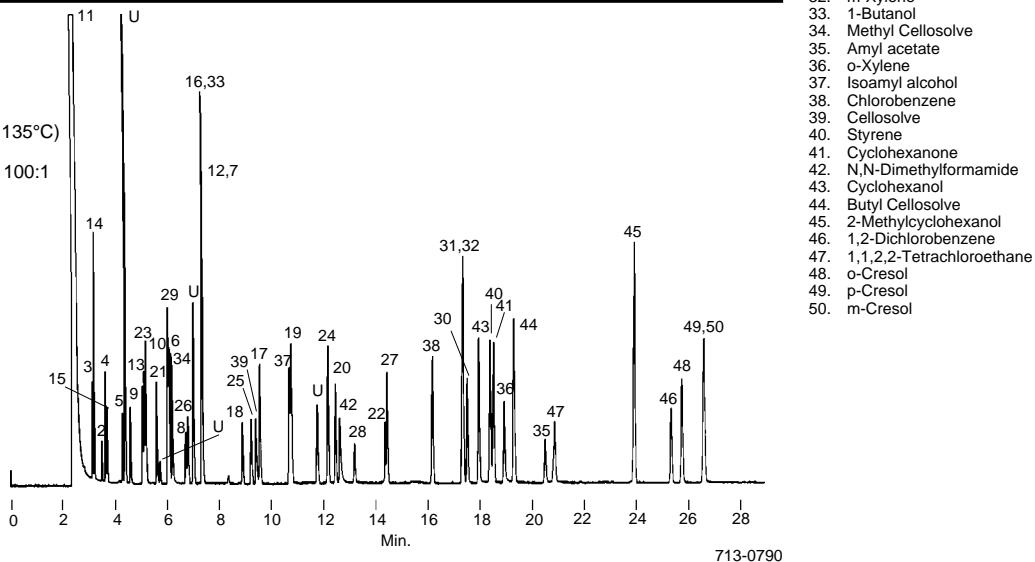


Figure B. Industrial Solvents on a PTE-5 Column

Column: PTE-5, 30m x 0.32mm ID, 1.0 μ m film
 Cat. No.: 24159
 Oven: 40°C (5 min), to 130°C at 4°C/min
 Carrier: helium, 20cm/sec (set at 135°C)
 Det.: FID
 Inj.: 1 μ L mixed solvents, split 100:1



Ordering Information:

SUPELCOWAX 10 Fused Silica Capillary Column
 30m x 0.53mm ID, 1.0 μ m film

25301-U

PTE-5 Fused Silica Capillary Column
 30m x 0.32mm ID, 1.0 μ m film

24159

See the Supelco catalog for other column dimensions.

Fused silica columns manufactured under HP US Pat. No. 4,293,415.

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