# Resolving Industrial Solvent Mixtures on Packed and Capillary GC Columns

*Nonpolar SPB-1 and polar SUPELCOWAX 10 capillary columns separate complex mixtures of most commonly used solvents with good resolution. Resolution on 0.53mm ID columns is similar to that on 0.25mm ID or 0.32mm ID columns, but 0.53mm ID columns accept up to 2000ng of each analyte, compared to about 500ng for 0.32mm ID columns. Thus, these columns are useful for analyses of samples containing both concentrated and dilute components. Alternatively, packed GC columns offer various combinations of selectivity and capacity. 80/120 Carbopack B/3% SP-1500 and 60/80 Carbopack B/1% SP-1510, respectively, separate 27 and 22 of 32 widely used solvents.*

**Key Words**

● solvents ● industrial solvents ● water pollutants ● priority pollutants ● alcohols ● ketones ● esters ● aromatics ● chlorinated hydrocarbons

## Introduction

Because many different solvents are used in industrial processes, there are many different situations requiring GC analysis. The appropriate column must be chosen from among nonpolar and polar capillary columns, or from several packed columns. The advantages of specific columns are described here.

## Solvent Analyses On Capillary Columns

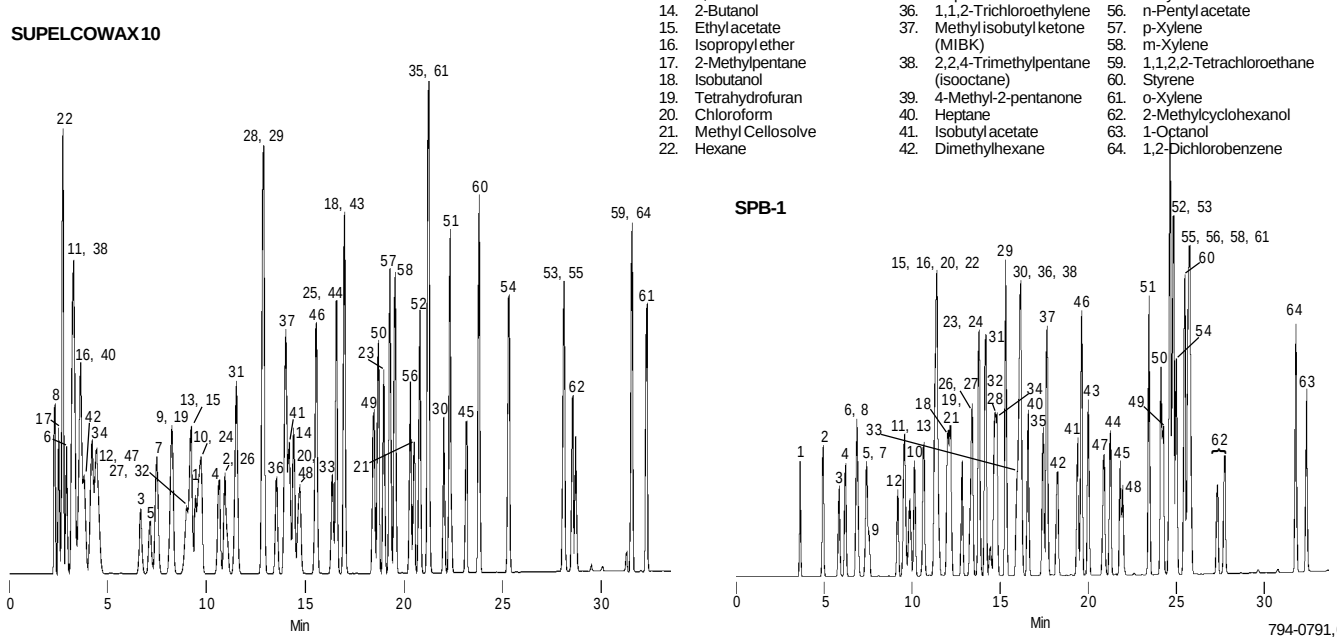
Both nonpolar SPB™-1 (bonded SE-30) and polar SUPELCOWAX™ 10 (bonded CARBOWAX® PEG 20M) capillary columns resolve most commonly used solvents, but with somewhat different selectivity. To determine which phase is appropriate for your sample, compare the performance of these columns with the same solvent mixture (Figure A).

Figure A also shows that complex solvent mixtures can be resolved on a 0.53mm capillary column, as well as on a 0.25mm or 0.32mm ID column. Sample resolution is almost the same, as a comparison

**Figure A. SUPELCOWAX 10 and SPB-1 Capillary Columns Provide Alternative Selectivities for Solvents**

| | |
|---|---|
| Column: | **SUPELCOWAX 10, 60m X 0.53mm ID, 2.0µm film** |
| Cat. No: | **25376** |
| Column: | **SPB-1, 60m x 0.53mm ID, 5.0µm film** |
| Cat. No.: | **25349** |
| Oven: | 40°C (5 min) to 220°C at 5°C/min |
| Carrier: | helium, 5mL/min |
| Det.: | FID, 300°C |
| Inj.: | 0.2-0.3µL, split injection, 250°C |

1. Methanol
2. Ethanol
3. Acetone
4. Isopropanol
5. Methyl acetate
6. Ethyl ether
7. Methylene chloride
8. Pentane
9. trans-1,2-Dichloroethylene
10. Methyl ethyl ketone (MEK)
11. Methyl t-butyl ether (MTBE)
12. 1,1-Dichloroethylene
13. 1,1-Dichloroethane
14. 2-Butanol
15. Ethyl acetate
16. Isopropyl ether
17. 2-Methylpentane
18. Isobutanol
19. Tetrahydrofuran
20. Chloroform
21. Methyl Cellosolve
22. Hexane
23. 1-Butanol
24. Isopropyl acetate
25. 1,2-Dichloroethane
26. 1-Chlorobutane
27. 1,1,1-Trichloroethane
28. 2-Pentanone
29. 3-Pentanone
30. Ethyl Cellosolve
31. Benzene
32. Carbon tetrachloride
33. 1,4-Dioxane
34. Cyclohexane
35. Isopentanol
36. 1,1,2-Trichloroethylene
37. Methyl isobutyl ketone (MIBK)
38. 2,2,4-Trimethylpentane (isooctane)
39. 4-Methyl-2-pentanone
40. Heptane
41. Isobutyl acetate
42. Dimethylhexane
43. 2-Hexanone
44. n-Butyl acetate
45. 2-Methoxyethyl acetate
46. Toluene
47. 2,2,5-Trimethylhexane
48. 1,1,2,2-Tetrachloroethylene
49. Isopentyl acetate
50. 4-Heptanone
51. Chlorobenzene
52. 2-Heptanone
53. Cyclohexanol
54. Cyclohexanone
55. Butyl Cellosolve
56. n-Pentyl acetate
57. p-Xylene
58. m-Xylene
59. 1,1,2,2-Tetrachloroethane
60. Styrene
61. o-Xylene
62. 2-Methylcyclohexanol
63. 1-Octanol
64. 1,2-Dichlorobenzene

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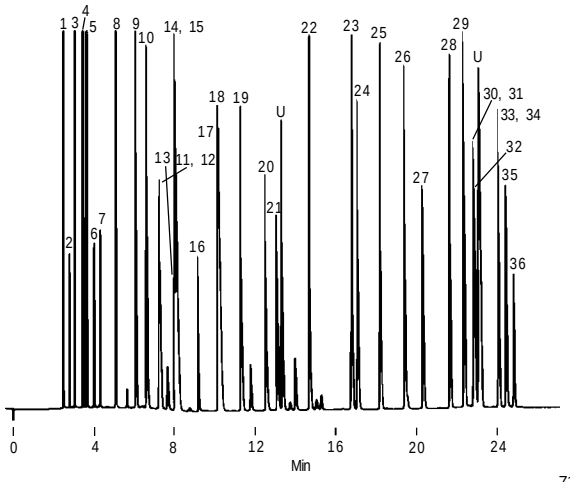
## Figure B. Common Solvents on an SPB-1 Column

Column: **SPB-1, 30m x 0.32mm ID, 1.0µm film**
Cat. No.: **24045-U**
Oven: 30°C (8 min) to 125°C at 4°C/min, hold 5 min
Carrier: helium, 25cm/sec (set at 30°C)
Det.: FID, 250°C
Inj.: 0.2µL approximately equal proportions of each component, split 200:1, 250°C

1. Methanol
2. Methyl formate
3. Ethanol
4. Acetone
5. Isopropyl alcohol
6. Ethyl formate
7. Methylene chloride
8. n-Propyl alcohol
9. Methyl ethyl ketone
10. sec-Butyl alcohol
11. Ethyl acetate
12. Chloroform
13. Tetrahydrofuran
14. Methyl Cellosolve
15. Isobutyl alcohol
16. 1,1,1-Trichloroethane
17. Isopropyl acetate
18. n-Butyl alcohol
19. 2-Nitropropane
20. 1,4-Dioxane
21. Ethyl Cellosolve
U. Unknown
22. Methyl isobutyl ketone
23. Toluene
24. Isobutyl acetate
25. Mesityl oxide
26. n-Butyl acetate
27. Diacetone alcohol
28. 5-Methyl-2-hexanone
29. Ethylbenzene
30. m-Xylene
31. p-Xylene
32. Cyclohexanone
U. Unknown
33. o-Xylene
34. Cellosolve acetate
35. Butyl Cellosolve
36. Isoamyl acetate

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of the chromatogram for the SPB-1 column in Figure A and the similar mixture in Figure B will reveal, but sample capacity for a 0.53mm ID column is far greater than for a narrow bore column – 2000ng per analyte, versus 500ng for a 0.32mm ID column (or 20,000ng per component for a packed column). Consequently, a 0.53mm ID column will resolve solvent mixtures with both concentrated and dilute components, such as a primary solvent containing trace contaminants. When you dilute such a sample to avoid overloading a 0.25mm or 0.32mm column with the concentrated component, the trace components can be difficult to detect or quantify. The large capacity of a 0.53mm ID column eliminates the need to dilute concentrated components. Furthermore, concentrated solvents will not damage the SPB-1 or SUPELCOWAX 10 bonded phase – you can introduce samples onto the column by split, splitless, or on-column injection.

A 0.53mm ID capillary column can be installed in instruments designed for packed columns and used with packed column injection and detection devices, as easily as a packed column. They will accept high carrier gas flow rates without significant loss of sample resolution (Figure C). When used with packed column flow rates, a 0.53mm column's efficiency is about equal to that of a packed column. An analysis comparable to that from the packed column, however, requires significantly less time. When maximum efficiency is needed, the flow rate must be reduced toward the ideal rate of 2.5mL/minute (helium), and make-up gas must be supplied to the detector. Our injector and detector conversion kits enable you to convert a packed column system to accept 0.53mm ID capillary columns simply, inexpensively, and reversibly. For more about how to incorporate wide bore capillary columns into a packed column system, refer to our catalog.

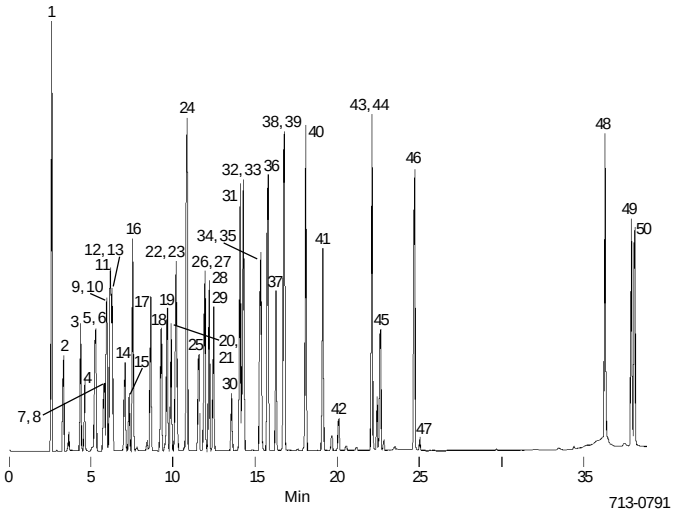
### Solvent Analyses on Packed Columns

Many industrial solvents can be separated on either 80/120 Carbopack™ B/3% SP-1500 or 60/80 Carbopack B/1% SP™-1510 packing. Although both of these packings resolve complex mixtures well, the Carbopack B/SP-1500 packing resolves more solvents from a single mixture. Under appropriate conditions, this packing separates 27 of 32 widely used solvents (Figure D). Minimal peak tailing by the low molecular weight alcohols confirms that the packing is chemically inert. The narrow, symmetrical, late eluting peaks (e.g., ethylbenzene) indicate good column

## Figure C. A 0.53mm ID Capillary Column Can Be Used at Relatively High Flow Rates

Column: **SUPELCOWAX 10, 30m x 0.53mm ID, 1.0µm film**
Cat. No.: **25301-U**
Oven: 40°C (5 min) to 200°C at 5°C/min
Carrier: helium, 5mL/min
Det.: FID, 250°C
Inj.: 1µL mixed solvents, split (50:1), 250°C

1. Hexane
2. 1,1-Dichloroethylene
3. Acetone
4. Methyl acetate
5. trans-1,2-Dichloroethylene
6. Tetrahydrofuran
7. Carbon tetrachloride
8. 1,1,1-Trichloroethane
9. 1,1-Dichloroethane
10. Ethyl acetate
11. Methanol
12. Isopropanol acetate
13. Methyl ethyl ketone
14. Isopropanol
15. Methylene chloride
16. Benzene
17. Propyl acetate
18. Trichloroethylene
19. Methyl isobutyl ketone
20. Isobutyl acetate
21. Chloroform
22. Tetrachloroethylene
23. 2-Butanol
24. Toluene
25. 1,4-Dioxane
26. 1,2-Dichloroethane
27. n-Butyl acetate
28. 2-Hexanone (MBK)
29. Isobutanol
30. Isoamyl acetate
31. p-Xylene
32. m-Xylene
33. 1-Butanol
34. Methyl Cellosolve
35. Amyl acetate
36. o-Xylene
37. Isoamyl alcohol
38. Chlorobenzene
39. Cellosolve
40. Styrene
41. Cyclohexanone
42. N,N-Dimethylformamide
43. Cyclohexanol
44. Butyl Cellosolve
45. 2-Methylcyclohexanol
46. 1,2-Dichlorobenzene
47. 1,1,2,2-Tetrachloroethane
48. o-Cresol
49. p-Cresol
50. m-Cresol

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efficiency. At k = 12 (methyl ethyl ketone), the efficiency of a Carbopack B/3% SP-1500 column is 600-700 theoretical plates per foot.

A column that resolves the compounds in Figure D can be very useful to solvent analysts. For example, Cellosolves® are widely used as water-soluble solvents and as nonchlorinated replacements for chlorinated solvents, but these compounds are not resolved on all columns used for solvent analyses. A Carbopack B/3% SP-1500 column separates Cellosolves from each other and from the other components of the complex test mixture. Similarly, this column resolves o-, m-, and p-xylene which, like the Cellosolves, do not separate on most general purpose solvent columns.

A Carbopack B/3% SP-1500 column also separates solvents well under conditions that minimize analysis time. In Figure D, 27 of 32 solvents were resolved in 44 minutes by temperature programming the column at 4°C/minute. When the temperature programming rate was increased to 8°C/minute, analysis time was reduced to 25 minutes and 24 of 32 solvents were resolved (Table 1). Thus, you can reduce solvent analysis time significantly with only a slight sacrifice in separation.

A Carbopack B/3% SP-1500 column separates many compounds not shown in Figure D. A number of chlorinated compounds, and other materials, are no longer used as solvents, but still must be monitored by industrial hygienists. Some of these compounds, and their retention times, are listed in Table 1. This packing also is useful for analyzing volatile priority pollutants in wastewater (Table 1). The elution order for these pollutants on Carbopack B/3% SP-1500 differs from that on 60/80 Carbopack B/1% SP-1000, the primary column used for this analysis (1). By using a Carbopack B/3% SP-1500 column as a backup, you can quantify cis-1,3-dichloropropene, tetrachloroethylene, and tetrachloroethane. These pollutants are not isolated on the SP-1500 packing, when compared to the SP-1000 packing, but they coelute with different compounds.

The other Carbopack B packing, 60/80 Carbopack B/1% SP-1510, separates 22 of the 32 solvents in the test mixture (Figure E). Peaks are nearly symmetrical, even for the xylenes and other late-eluting compounds. At k = 15 (methyl ethyl ketone) column efficiency is 500-600 theoretical plates per foot. Separation on Carbopack B/1% SP-1510 is not quite as good as on Carbopack B/3% SP-1500, but the two packings differ in selectivity. Consequently, you can use a Carbopack B/1% SP-1510 column to resolve solvents not separated on Carbopack B/3% SP-1500, or to confirm results obtained on the latter column.

Either Carbopack B packing offers distinct advantages when compared to other packings used for general solvents analyses. For example, solvent resolution is better on Carbopack B/3% SP-1500 (Figure D) than on a widely used packing, Carbopack C/0.1% SP-1000 (Figure F). Overall, solvent resolution is comparable for Carbopack B/1% SP-1510 (Figure E) and the Carbopack C packing (Figure F). Both Carbopack B packings have more than twice the sample capacity of the Carbopack C material: a 10' x 1/8" column of either Carbopack B packing can accept up to 65µg of each sample component, but a comparable column of the Carbopack C packing can accept only 30µg of each component (Figure G).*

* Sample capacity was determined by progressively increasing the amount of a nonpolar test probe (4% pentane in methylene chloride) injected onto each column, and measuring peak asymmetry at 10% of peak height. The point at which peak asymmetry sharply increased was defined as the maximum sample capacity. Peaks with an asymmetry range of 0.90-1.10 were considered symmetrical.

## Table 1. 80/120 Carbopack B/3% SP-1500 Column Monitors Many Solvents and Pollutants

**Industrial Solvents**

| Solvent | $t_R$ (min) 4°C/min temp. rise | $t_R$ (min) 8°C/min temp. rise |
|---|---|---|
| Methanol | 2.7 | 1.8 |
| Methyl formate | 4.2 | 2.7 |
| Ethanol | 5.1 | 2.9 |
| Acetone | 7.3 | 4.1 |
| Methylene chloride | 7.6 | 4.3 |
| Isopropyl alcohol | 8.6 | 4.6 |
| Ethyl formate | 9.3 | 5.0 |
| Methyl acetate | 9.3 | 5.0 |
| n-Propyl alcohol | 10.5 | 5.4 |
| Tetrahydrofuran (THF) | 13.5 | 7.1 |
| Methyl ethyl ketone (MEK) | 14.2 | 7.4 |
| Methyl Cellosolve | 15.4 | 7.8 |
| sec-Butyl alcohol | 15.8 | 8.0 |
| Ethyl acetate | 16.2 | 8.2 |
| Isobutyl alcohol | 16.6 | 8.3 |
| 2-Nitropropane | 18.3 | 9.3 |
| n-Butyl alcohol | 18.6 | 9.8 |
| Ethyl Cellosolve | 21.9 | 11.0 |
| n-Propyl acetate | 24.2 | 12.2 |
| sec-Butyl acetate | 28.9 | 14.6 |
| Cyclohexanone | 29.9 | 15.4 |
| Isobutyl acetate | 30.6 | 15.4 |
| n-Butyl acetate | 32.2 | 16.2 |
| Toluene | 32.2 | 16.3 |
| Mesityl oxide | 32.2 | 16.3 |
| Cellosolve acetate | 36.4 | 18.6 |
| Butyl Cellosolve | 37.1 | 19.1 |
| Isoamyl acetate | 37.8 | 19.5 |
| Ethylbenzene | 38.5 | 20.1 |
| m-Xylene | 41.7 | 22.5 |
| p-Xylene | 42.8 | 23.5 |
| o-Xylene | 43.3 | 24.0 |

**Volatile Wastewater Pollutants**

| Pollutant | $t_R$ (min) 4°C/min temp. rise |
|---|---|
| Bromochloromethane | 12.2 |
| trans-1,2-Dichloroethylene | 13.1 |
| 1,1-Dichloroethane | 13.1 |
| 1,2-Dichloroethane | 16.0 |
| Bromodichloromethane | 20.4 |
| 1,2-Dichloropropane | 22.4 |
| trans-1,3-Dichloropropene | 22.4 |
| Chlorodibromomethane | 22.4 |
| cis-1,3-Dichloropropene | 24.3 |
| Trichloroethylene | 25.7 |
| 1-Chloro-2-bromopropane | 27.5 |
| Bromoform | 31.2 |
| 1,1,2,2-Tetrachloroethylene | 33.0 |
| 1,4-Dichlorobutane | 34.4 |
| 1,1,2,2,-Tetrachloroethane | 34.4 |
| Chlorobenzene | 36.2 |

**Hazardous Solvents**

| Solvent | $t_R$(min) 4°Cmin temp. rise |
|---|---|
| Chloroform | 14.6 |
| Pentane | 15.2 |
| 1,1,1-Trichloroethane | 18.3 |
| 1,4-Dioxane | 18.6 |
| Carbon tetrachloride | 19.9 |
| Isopropyl acetate | 21.4 |
| Benzene | 21.8 |
| Hexane | 23.9 |
| 1,1,2-Trichloroethane | 24.4 |
| Methyl isobutyl ketone (MIBK) | 28.3 |
| Methyl isoamyl ketone (MIAK) | 36.4 |

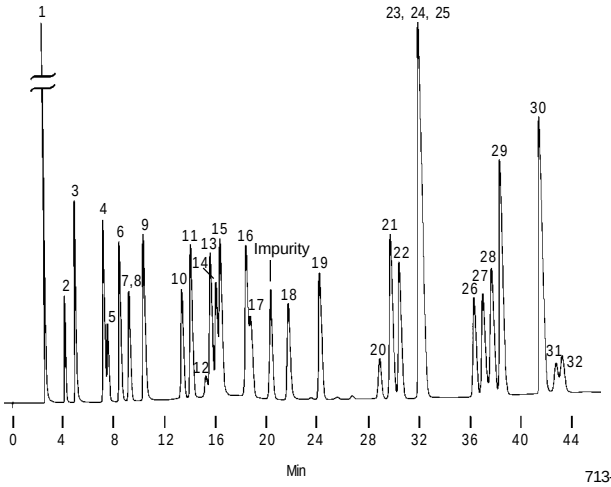
Packing: **80/120 Carbopack B/3% SP-1500**
Cat. No.: **11813** (15g bottle)
Column: 10' x 1/8" SS
Oven: 70°C to 235°C at 4°C/min or 8°C/min
Carrier: nitrogen, 20mL/min
Det.: FID
Inj.: 0.3µL (industrial and hazardous solvents) or 0.1µL (wastewater pollutants), approximately equal volumes of neat components

**Solvents in Figures D-F**

1. Methanol
2. Methyl formate
3. Ethanol
4. Acetone
5. Methylene chloride
6. Isopropyl alcohol
7. Ethyl formate
8. Methyl acetate
9. n-Propyl alcohol
10. Tetrahydrofuran (THF)
11. Methyl ethyl ketone (MEK)
12. Methyl Cellosolve
13. sec-Butyl alcohol
14. Ethyl acetate
15. Isobutyl alcohol
16. 2-Nitropropane
17. n-Butyl alcohol
18. Ethyl Cellosolve
19. n-Propyl acetate
20. sec-Butyl acetate
21. Cyclohexanone
22. Isobutyl acetate
23. n-Butyl acetate
24. Toluene
25. Mesityl oxide
26. Cellosolve acetate
27. Butyl Cellosolve
28. Isoamyl acetate
29. Ethylbenzene
30. m-Xylene
31. p-Xylene
32. o-Xylene

## Figure D. An 80/120 Carbopack B/3% SP-1500 Column Resolves Most Components of a 32 Solvent Mixture

Packing: **80/120 Carbopack B/3% SP-1500**
Cat. No.: **11813-U** (15g bottle)
Column: 10' x 1/8" SS
Oven: 70°C to 235°C at 4°C/min
Carrier: nitrogen, 20mL/min
Det.: FID
Inj.: 0.3µL, approximately equal volumes of neat components

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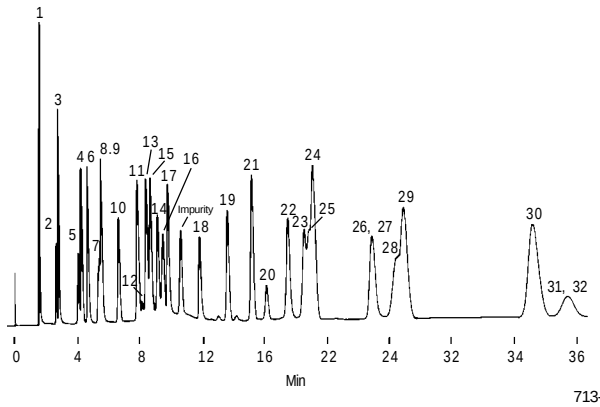
## Figure E. Selectivity for Solvents on Carbopack B/1% SP-1510 Differs from That for Carbopack B/3% SP-1500

Packing: **60/80 Carbopack B/1% SP-1510**
Cat. No.: **11809** (15g bottle)
Column: 10' x 1/8" SS
Oven: 100°C to 225°C at 8°C/min
Carrier: nitrogen, 20mL/min
Det.: FID
Inj.: 0.3µL, approximately equal volumes of neat components

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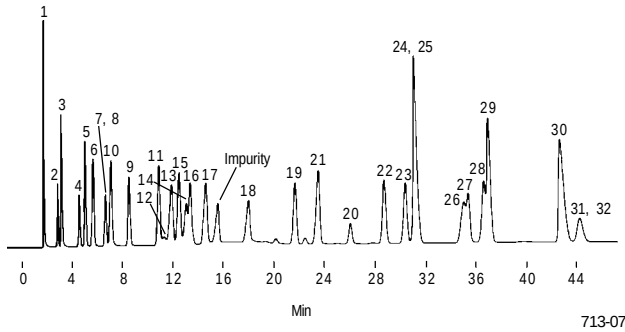
## Figure F. Industrial Solvents on a Carbopack C/0.1% SP-1000 Column

Packing: **80/100 Carbopack C/0.1% SP-1000**
Cat. No.: **11820** (15g bottle)
Column: 10' x 1/8" SS
Oven: 70°C to 225°C at 4°C/min
Carrier: nitrogen, 20mL/min
Det.: FID
Inj.: 0.3µL, 3% each component by volume (approx. 12µg each)

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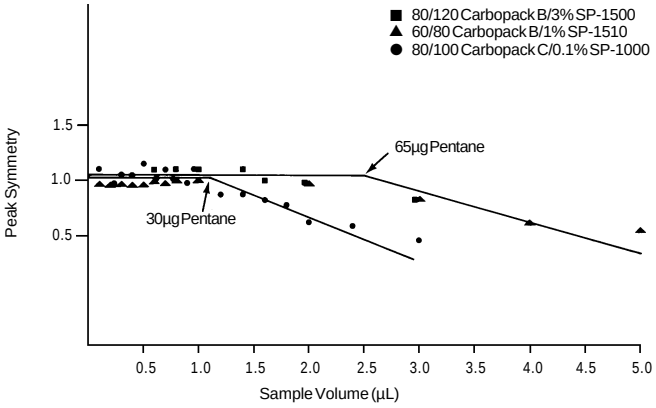
## Figure G. Sample Capacity of Carbopack B Packings Is Twice That for Carbopack C

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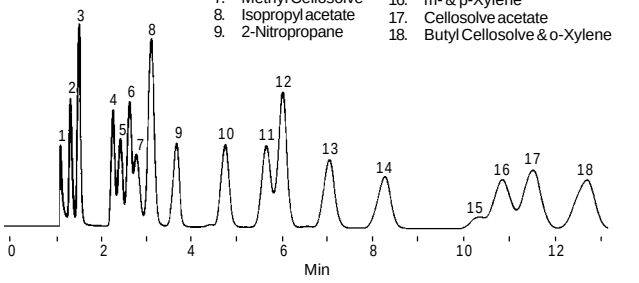
Despite the superiority of the Carbopack B packings, Carbopack C/0.1% SP-1000 is useful for separating complex mixtures of alcohols, ketones, esters, aromatics, and chlorinated hydrocarbons. Alcohol peaks do not tail. Depending on the separation needed, this packing can be used in place of the Carbopack B packings, or to confirm results obtained on them. Table 2 lists retention data for solvents on Carbopack C/0.1% SP-1000. This packing's capacity is less than the capacities of the Carbopack B packings (as discussed above), and sample volume should be restricted to 0.1-0.5µL.

SP-2100 methyl silicone is another useful phase for separating a variety of solvents, with elution order generally according to increasing boiling points. We suggest a mixed phase of 20% SP-2100 combined with 0.1% Carbowax® 1500 (to reduce tailing of alcohols) on 100/120 SUPELCOPORT™ (Figure H, Table 2). The packing is perhaps most useful for separating complex mixtures of chlorinated solvents (Figure I), or for resolving impurities from a single chlorinated solvent.

## Figure H. Solvents on a 20% SP-2100/0.1% Carbowax 1500 Column

Packing: **20% SP-2100/0.1% Carbowax 1500 on 100/120 SUPELCOPORT**
Cat. No.: **11821** (20g bottle)
Column: 10' x 1/8" SS
Oven: 100°C
Carrier: nitrogen, 20mL/min
Det.: FID
Inj.: 0.5µL, approximately equal volumes of neat components

1. Methanol
2. Ethanol
3. Acetone
4. MEK
5. Ethyl acetate
6. Isopropyl alcohol
7. Methyl Cellosolve
8. Isopropyl acetate
9. 2-Nitropropane
10. Methyl isobutyl ketone (MIBK)
11. Isobutyl acetate
12. Toluene
13. n-Butyl acetate
14. Diacetone alcohol
15. Ethylbenzene
16. m- & p-Xylene
17. Cellosolve acetate
18. Butyl Cellosolve & o-Xylene

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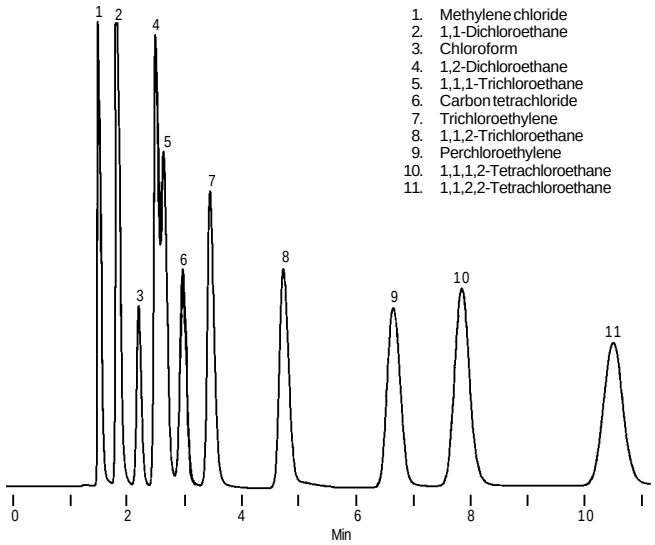
## Figure I. Chlorinated Solvents on a 20% SP-2100/0.1% Carbowax 1500 Column

Packing: **20% SP-2100/0.1% Carbowax 1500 on 100/120 SUPELCOPORT**
Cat. No.: **11821** (20g bottle)
Column: 10' x 1/8" SS
Oven: 100°C
Carrier: nitrogen, 20mL/min
Det.: FID
Inj.: 0.5µL, approximately equal volumes of neat components

1. Methylene chloride
2. 1,1-Dichloroethane
3. Chloroform
4. 1,2-Dichloroethane
5. 1,1,1-Trichloroethane
6. Carbon tetrachloride
7. Trichloroethylene
8. 1,1,2-Trichloroethane
9. Perchloroethylene
10. 1,1,1,2-Tetrachloroethane
11. 1,1,2,2-Tetrachloroethane

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Despite the inclusion of Carbowax 1500 in this packing, methanol and ethanol tail when present in low concentrations. SP-2100/Carbowax 1500 columns also are poorly suited to separating mixtures of aliphatic solvents combined with alcohols, ketones, etc. The hydrocarbons tend to be eluted along with the other components, unless the aliphatics have either lower or higher boiling points. Furthermore, the Carbowax 1500 restricts the upper temperature limit of this packing to 175°C.

### Special Purpose Packed Columns

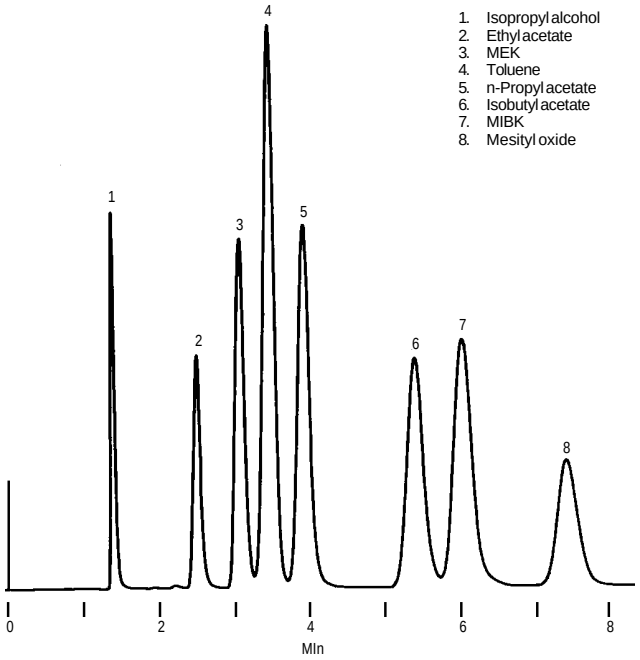
Relative to other packings, 20% SP-2401/0.1% Carbowax 1500 on 100/120 SUPELCOPORT delays the elution of ketones. For example, methyl ethyl ketone is eluted after ethyl acetate, with good resolution (Figure J). In contrast, 20% SP-2100/0.1% Carbowax 1500 barely separates this pair (Figure H). Retention times for solvents on the SP-2401 packing at 100°C are given in Table 2. At higher temperatures, isophorone is eluted in a reasonable analysis time.

When used as a stationary phase, THEED (tetrahydroxyethylenediamine) retards elution of alcohols relative to other classes of compounds. It also allows hydrocarbons to elute rapidly, compared to other compounds with similar boiling points. Figure K shows the rapid elution of heptane and octane, esters, aromatics, and finally alcohols from 15% THEED on 100/120 Chromosorb® W AW. Retention data for other compounds are listed in Table 2. We do not recommend using a silanized support with THEED because it would be extremely difficult to coat with the THEED stationary phase.

## Figure J. Solvents on a Ketone-Retarding Column

Packing: **20% SP-2401/0.1% Carbowax 1500 on 100/120 SUPELCOPORT**
Cat. No.: **11822** (20g bottle)
Column: 10' x 1/8" SS
Oven: 100°C
Carrier: nitrogen, 20mL/min
Det.: FID
Inj.: 0.1µL, approximately equal volumes of neat components

1. Isopropyl alcohol
2. Ethyl acetate
3. MEK
4. Toluene
5. n-Propyl acetate
6. Isobutyl acetate
7. MIBK
8. Mesityl oxide




797-0433

## Table 2. Retention Times for Common Solvents on Packed Column Phases ($t_R$ in min)

| Compound | Column | | | | |
|---|---|---|---|---|---|
| | 80/100 Carbopack C/ 0.1% SP-1000 | 10% SP-1000 | 20% SP-2100/ 0.1% Carbowax 1500 | 20% SP-2401/ 0.1% Carbowax 1500 | 15% THEED |
| **Alcohols** | | | | | |
| Methyl | 0.6 | 4.0 | 1.1 | 1.1 | 3.0 |
| Ethyl | 0.9 | 4.1 | 1.2 | 1.2 | 3.4 |
| Isopropyl | 1.5 | 4.2 | 1.4 | 1.4 | 3.0 |
| n-Propyl | 1.8 | 6.2 | 1.8 | 1.7 | 4.9 |
| Isobutyl | 4.0 | 7.8 | 2.5 | 2.2 | 5.4 |
| n-Butyl | 5.5 | 10.1 | 3.0 | 2.5 | 7.6 |
| Diacetone | Decomposes | 3.2 | 8.0 | 14.4 | Not eluted |
| **Cellosolves** | | | | | |
| Methyl | 3.4 | 13.1 | 2.7 | 2.8 | 12.1 |
| Ethyl | 8.6 | 15.8 | 4.0 | 3.8 | 12.3 |
| Butyl | Late | 39.4 | 12.4 | 9.7 | 27.0 |
| **Ketones** | | | | | |
| Acetone | 1.3 | 3.1 | 1.4 | 2.2 | 1.4 |
| MEK | 3.2 | 3.9 | 2.1 | 3.1 | 1.8 |
| MIBK | 21.9 | 5.9 | 4.6 | 6.1 | 2.3 |
| DIBK | Late | 12.4 | — | — | — |
| Mesityl oxide | Late | 10.8 | 6.4 | 7.6 | 4.1 |
| Cyclohexanone | — | — | 11.1 | 18.3 | Not eluted |
| Isophorone | — | — | 43.9 | — | Not eluted |
| **Ethers** | | | | | |
| Ethyl | 2.7 | 2.0 | — | — | — |
| Dioxane | 4.4 | 7.7 | — | — | — |
| **Acetates** | | | | | |
| Ethyl | 4.5 | 3.5 | 2.2 | 2.5 | 1.3 |
| Isopropyl | 9.0 | 3.6 | 3.0 | 3.0 | 1.3 |
| n-Propyl | Late | 4.8 | 3.9 | 4.0 | 1.7 |
| Isobutyl | Late | 5.7 | 5.4 | 5.5 | 1.8 |
| n-Butyl | Late | 7.2 | 6.8 | 6.5 | 2.2 |
| Cellosolve | Late | 21.7 | 11.1 | 12.0 | 6.5 |
| **Formates** | | | | | |
| Methyl | 0.9 | 2.9 | 1.2 | 1.3 | — |
| Ethyl | 1.8 | 3.3 | 1.6 | 1.8 | — |
| **Chloroalkanes** | | | | | |
| Carbon tetrachloride | 4.9 | 3.6 | — | — | — |
| 1,1,1-Trichloroethane | 4.4 | — | 3.0 | 1.7 | 1.3 |
| Methylene chloride | 1.3 | 4.1 | 1.7 | 1.0 | 1.2 |
| Trichloroethylene | 8.9 | 5.3 | — | — | — |
| Chloroform | 2.8 | 5.7 | — | — | — |
| Tetrachloroethylene | Late | 6.3 | — | — | — |
| 1,2-Dichloroethane | 3.2 | 7.2 | — | — | — |
| 1,2,2-Trichloroethane | Late | 17.8 | — | — | — |
| **Aromatics** | | | | | |
| Benzene | 9.8 | 4.6 | 3.2 | 2.2 | 1.4 |
| Toluene | Late | 6.9 | 5.8 | 3.5 | 1.9 |
| Ethylbenzene | Late | 10.0 | — | — | — |
| m- & p-Xylene | Late | 10.8 | 10.5 | 5.5 | 2.6 |
| o-Xylene | Late | 13.3 | 12.1 | 6.6 | 3.3 |
| Styrene | Late | 18.6 | — | — | — |
| **Aliphatics** | | | | | |
| n-Hexane | Late | 2.1 | 2.4 | 1.3 | 0.8 |
| n-Heptane | Late | 2.4 | 4.0 | 1.8 | 0.8 |
| Isooctane | Late | 2.2 | — | — | — |
| n-Octane | Late | 3.0 | 7.1 | 2.6 | 0.9 |
| n-Nonane | — | — | 13.0 | 3.9 | 1.1 |
| n-Decane | Late | 5.6 | 24.3 | 6.2 | 1.3 |
| **Miscellaneous** | | | | | |
| Carbon disulfide | 2.6 | 2.5 | — | — | — |
| THF | 2.4 | 3.9 | 2.7 | — | 1.6 |
| 2-Nitropropane | 4.6 | 10.2 | 3.6 | 7.0 | 3.5 |
| DMF | 8.1 | 33.5 | 5.4 | — | — |

80/100 Carbopack C/0.1% SP-1000: 6' x 1/8" SS column, 20mL/min flow rate (nitrogen). 10% SP-1000 on 80/100 SUPELCOPORT: 20' x 1/8" SS column, 30mL/min flow rate. Other packings: 10' x 1/8" SS columns, 20mL/min flow rate. Oven (all columns): 100°C

## Monitoring Water in Solvents on a Packed Column

Determining the amount of water in a sample by GC is difficult. With many columns, the water peak will tail severely. The degree of tailing is influenced by a variety of factors, including the nature of the column packing and the composition of the column tubing and of the instrument inlet. Diatomite supports cause tailing, even when acid washed and dimethylchlorosilane treated. However, water does not tail on porous polymers (e.g., Chromosorb 101) because these supports are polar in nature.

Water in solvents can be quantified rapidly with Chromosorb 101 packed in a short metal tube. The analysis of water in toluene in Figure L was performed on a GC equipped with a thermal conductivity detector. The Chromosorb 101 column also can be used to separate some solvent mixtures.

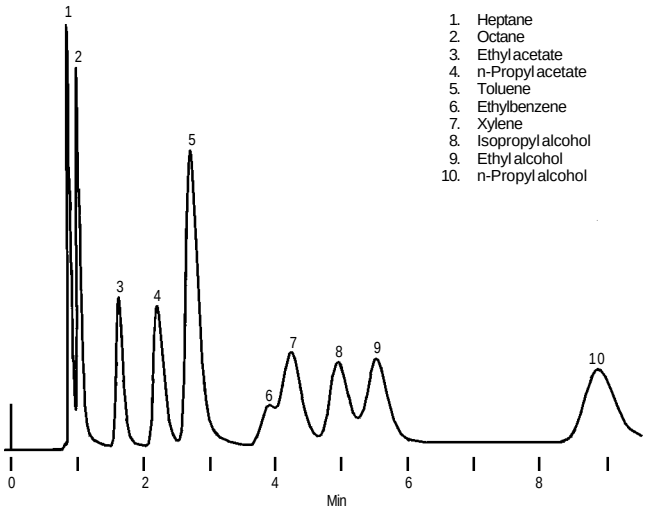
Metal column tubing has been blamed for water peak tailing, but this depends on both the quality of the tubing and the concentration of water in the sample. We have been able to detect as little as 0.01% water in solvents, using columns made with our chromatographic quality stainless steel tubing. Some analysts prefer using glass or Teflon® tubing to obtain a symmetric water peak. The overall efficiency of a glass column is higher than that of stainless steel, but efficiency of Teflon columns is poor.

A metal inlet in an instrument can be highly adsorptive and thus cause tailing. Avoid this by using a PureCol™ glass insert in the inlet or by injecting the sample directly onto the column.

A calibration curve for water can be obtained by injecting onto the column equal volumes of several water standards. Plot peak height versus water concentration (Figure M). The calibration mixtures should be prepared from a water-free solvent and dried with a good drying agent, such as Molecular Sieve 5A. The syringe used to inject the sample also should be water-free. For details on an internal standard procedure for water, see Hogan, *et al.* (2). References 3-5 also deal with determining water in samples.

## Figure K. Solvents on an Alcohol-Retarding Column

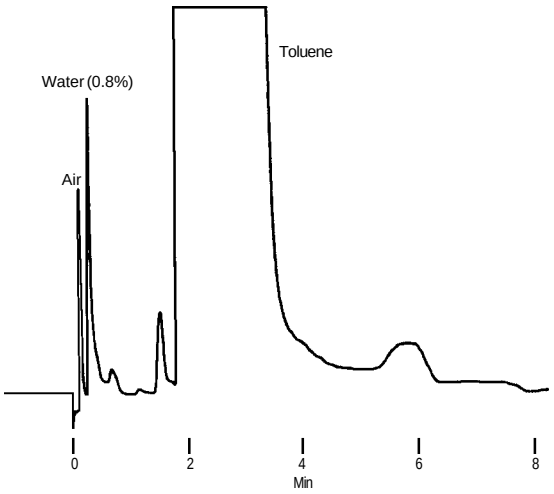
Packing: **15% THEED on 100/120 Chromosorb W AW**
Cat. No.: **11823** (20g bottle)
Column: 10' x 1/8" SS
Oven: 80°C
Carrier: nitrogen, 20mL/min
Det.: FID
Inj.: 0.5µL, approximately equal volumes of neat components




797-0434

## Figure L. Trace Water in Toluene

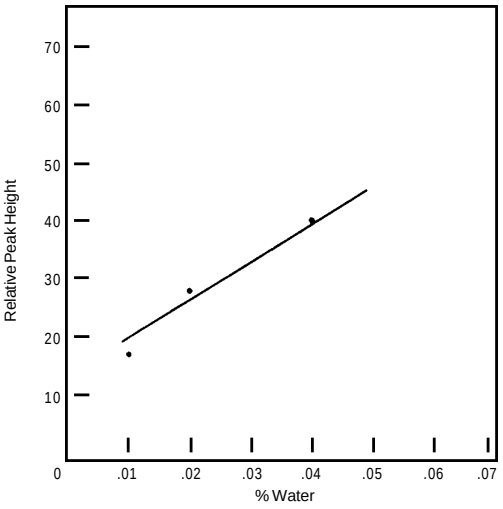
Packing: **60/80 Chromosorb 101**
Cat. No.: **20213** (50g bottle)
Column: 3' x 1/8" SS
Oven: 165°C
Carrier: helium, 20mL/min
Det.: TCD
Inj.: 0.1µL




797-0441

## Figure M. Peak Height vs. Concentration of Water in a Solvent

Packing: **60/80 Chromosorb 101**
Cat. No.: **20213** (50g bottle)
Column: 3' x 1/8" SS
Oven: 165°C
Carrier: helium, 20mL/min
Det.: TCD
Inj.: 0.1µL




797-0442


**References**

1. *Federal Register,* 44, No. 233, Dec. 3 (1979).
2. Hogan, J.M., R.A. Engel, and H.F. Stevenson, *Anal. Chem.,* 42, 249 (1970).
3. Bennett, O.F., *Anal. Chem.,* 36, 684 (1964).
4. Gvozdovich, T.N., G.S. Grinberg, L.V. Zuyeva, and Y.I. Yashin, *Petroleum Chemistry* (USSR), 12, No. 2, 120 (1972) English.
5. Hollis, O.L. and W.W. Haye, J. *Gas Chromatogr.,* 4, 235 (1966).

References not available from Supelco.

## Ordering Information:

### Stock Packed Stainless Steel Columns for Solvents Analyses

| General Config. (bend to fit most GCs) | Hewlett-Packard 5700, 5992-3 GC-MS (config. 5) | Hewlett-Packard 5880, 5890 5987, 6890 (config. A) | Perkin-Elmer 900, 3920 Sigma 1,2,3 (not on-column injection) | Perkin-Elmer 8300, 8400, 8500 8600, 8700, Auto System (not on-column injection) | Varian 3700 Vista series (FID) | Varian 3300/3400 Vista series (FID) |
|---|---|---|---|---|---|---|
| 3% SP-1500 on 80/120 Carbopack B (Cat. No. 1-1813), 10' x 1/8" column | | | | | | |
| **12592** | **12593** | **12594** | **12595-U** | **13734-U** | **12596** | **13735-U** |
| 0.1% SP-1000 on 80/100 Carbopack C (Cat. No. 1-1820), 6' x 1/8" column | | | | | | |
| **12495-U** | **12499** | **12500-U** | **12496** | **13736-U** | **12497** | **13737** |
| 10% SP-1000 on 80/100 SUPELCOPORT (Cat. No. 1-1872), 10' x 1/8" column | | | | | | |
| **12537** | **12541** | **13752-U** | **12538** | **13753** | **12539** | **13754** |
| 20% SP-2100/0.1% Carbowax 1500 on 100/120 SUPELCOPORT (Cat. No. 1-1821), 10' x 1/8" column | | | | | | |
| **12718-U** | **12740-U** | **12804-U** | **12751** | **13773** | **12773** | **13774** |

### Stock Packings for Solvents Analyses

| Description | Cat. No. |
|---|---|
| 80/120 Carbopack B/3% SP-1500, 15g | **11813-U** |
| 60/80 Carbopack B/1% SP-1510, 15g | **11809** |
| 80/100 Carbopack C/0.1% SP-1000, 15g | **11820** |
| GP 20% SP-2100/0.1% Carbowax 1500 on 100/120 SUPELCOPORT, 20g | **11821** |
| GP 20% SP-2401/0.1% Carbowax 1500 on 100/120 SUPELCOPORT, 20g | **11822** |
| 15% THEED on 100/120 Chromosorb W AW, 20g | **11823** |
| 10% SP-1000 on 80/100 SUPELCOPORT, 20g | **11872** |
| **Chromosorb 101** | |
| 60/80, 50g | **20213** |
| 80/100, 50g | **20214** |
| 100/120, 50g | **20215** |

GP – Indicates packing has been tested for specific analysis shown in this bulletin.

**Nukol™ capillary columns provide symmetrical peaks and good resolution for glycols analyses. For information about these columns, please see our catalog.**

### Capillary Columns

| Description | Cat. No. |
|---|---|
| **SPB-1** | |
| 30m x 0.25mm ID, 0.25µm film | **24028-U** |
| 60m x 0.25mm ID, 0.25µm film | **24030-U** |
| 30m x 0.32mm ID, 0.25µm film | **24044** |
| 30m x 0.32mm ID, 1.0µm film | **24045-U** |
| 60m x 0.32mm ID, 0.25µm film | **24046** |
| 15m x 0.53mm ID, 1.5µm film | **25302-U** |
| 30m x 0.53mm ID, 1.5µm film | **25303** |
| 30m x 0.53mm ID, 5.0µm film | **25345-U** |
| 60m x 0.53mm ID, 5.0µm film | **25349** |
| **SUPELCOWAX 10** | |
| 30m x 0.25mm ID, 0.25µm film | **24079** |
| 60m x 0.25mm ID, 0.25µm film | **24081** |
| 30m x 0.32mm ID, 0.25µm film | **24080-U** |
| 60m x 0.32mm ID, 0.25µm film | **24082** |
| 15m x 0.53mm ID, 1.0µm film | **25300-U** |
| 30m x 0.53mm ID, 1.0µm film | **25301-U** |
| 60m x 0.53mm ID, 2.0µm film | **25376** |

For other column lengths and film thicknesses, see our catalog.

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CARBOWAX, Cellosolve – Union Carbide Corp.

Chromosorb – Manville Corp.

Teflon – E.I. du Pont de Nemours & Co., Inc.

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

80/120 Carbopack B/3% SP-1500 was developed in cooperation with Dr. A. DiCorcia of the University of Rome.



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