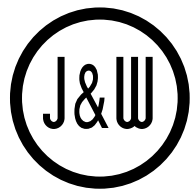


# Retention Index Characterization of Flavor, Fragrance and Many Other Compounds on DB-1 and DB-XLB

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## **Introduction**

In the quest for superior chromatography and better detection limits, novel GC stationary phases have been developed in order to push the analytical envelope. In recent years, low bleed stationary phases have been developed in order to improve compound sensitivity and detection limits, especially at higher temperatures. One of these novel polymers is DB-XLB, which has approximately 12% phenyl character. It utilizes second generation arylene polymer technology to achieve extremely low bleed levels. This increase in polymer stability allows a standard narrow bore capillary to be run up to 360°C during a temperature program while still maintaining a low bleed level. This polymer is unique, therefore its selective properties are not as well documented as some of the more standard phases, such as 100% methyl or 5% phenyl/95% methyl polysiloxanes. By characterizing the phase, the behavior of compounds on the phase may be better predicted. One way to characterize the phase is to compare its selectivity against a well-documented column such as DB-1.

A method of measuring column selectivity is by using retention indexing. Retention index (I) is the measure of the retentiveness of a compound relative to straight chain hydrocarbons under a given set of chromatographic conditions. Two equations are commonly used to calculate “I”:

**For Isothermal Conditions**

$$I = 100y + 100(z - y) \left[ \frac{\log t_r'(x) - \log t_r'(y)}{\log t_r'(z) - \log t_r'(y)} \right]$$

**For Linear Temperature Program Conditions**

$$I_T = 100y + 100 \left[ \frac{t_r(x) - t_r(y)}{t_r(z) - t_r(y)} \right]$$

*t'* = adjusted retention time

*t* = retention time

*x* = compound of interest

*y* = normal alkane with *y* carbon atoms eluting before compound *x*

*z* = normal alkane with *z* carbon atoms eluting after compound *x*

As an example, n-hexane has an “I” value of 600 and n-heptane an “I” value of 700. A known or unknown compound with an “I” value of 650 elutes between n-hexane and n-heptane. A retention index value gives an indication of where the compound of interest elutes relative to straight-chain hydrocarbons. Knowing this information, “I” values can be used to evaluate the elution of a mixture of compounds on a column for a given set of conditions. It can be determined from the closeness of the “I” values whether or not the mixture is likely to be resolvable on the chosen column.

Retention index values may also be used to compare retention indices on different columns. By doing this you can get a better idea of what column might be best suited for a specific analyte mixture. If a confirmation column needs to be selected, retention index values can be used to find a column that has the appropriate selectivity for the analytes as well as a different selectivity than the primary column.

The purpose of this study was to inject several different compounds to determine the nature of DB-XLB and its selective properties. This information will be useful in determining how different functionalities effect the elution of compounds on the phase. DB-XLB was evaluated using DB-1 as a benchmark.

## Experimental

The two columns compared in this retention index evaluation were DB-1, 100% polydimethylsiloxane, and DB-XLB, a commercially available proprietary low-bleed phase. Both columns had the dimensions 30m x 0.25mm x 0.25mm and were manufactured by J&W Scientific. All chromatographic analyses were performed via a single injection using a dual column set-up. This assembly was built by connecting one end of each column to the arms of a glass press-fit “Y” union. Approximately 5 meters of 0.53mm I.D. deactivated fused silica was connected to the remaining press-fit junction. The free end of the deactivated fused silica was installed in the GC injector port and the free ends of the columns were installed in separate FID’s on the same GC system.

The standards were analyzed with a Hewlett Packard model 5890 gas chromatograph equipped with an Optic 2 Programmable Injector (Ai Cambridge Ltd.). The programmable injector was used as a normal split/splitless inlet, run in split mode, 1:50 split.

The injector temperature was held constant at 250°C. The GC was fitted with 2 FID detectors, at 250°C. The columns had an average linear carrier gas velocity of 35 cm/sec, measured at 40°C, using helium. The GC oven was temperature programmed from 40°C to 325°C and ramped at 3°C per minute.

A wide variety of compounds were analyzed, both aromatic and non-aromatic, with different functionalities in order to get an idea of how the selectivity of DB-XLB differs in comparison to DB-1. All test samples were diluted with acetone. Test samples were analyzed at concentrations ranging from 300 to 500 ppm, injecting 1  $\mu$ L of sample. The concentrations of the straight-chain hydrocarbon (SCH) standards ranged from 100 to 500 ppm, with the larger compounds requiring a higher concentration. 1% solutions of each SCH were made in acetone. A little hexane was added to the acetone for the larger SCH to overcome solubility problems. Ranges of less concentrated SCH solutions SCH's were made in acetone. The mixtures of SCH had carbon ranges of C<sub>7</sub>-C<sub>13</sub>, C<sub>7</sub>-C<sub>16</sub>, C<sub>14</sub>-C<sub>22</sub> and C<sub>23</sub>-C<sub>26</sub>.

Test samples and SCH's were analyzed in the following sequence. Initially, all the SCH mixtures were injected to determine the approximate retention times for each SCH under the specified conditions. A test compound was injected and its retention time noted. The appropriate SCH mix was selected to inject with the test sample. The retention times of the first and last SCH in the appropriate SCH mix needed to bracket the test sample retention time in order to be selected. An aliquot from both the test sample and the chosen SCH mix were pulled into the syringe in order to equal a total volume of 1  $\mu$ L. Data from this generated chromatogram was recorded and the retention index was calculated from this data. All data was recorded on Hewlett Packard model 3393A integrators.

## Results and Discussion

Retention data for the DB-XLB and DB-1 dual column are tabulated in Table 1. In general, DB-XLB is a more retentive phase than DB-1. DB-XLB is significantly more retentive for aromatics on the order of what would be expected for a 12% phenyl polydimethylsiloxane. The more aromatic rings a compound contains, the more DB-XLB retains the compound. However, many non-aromatic compounds are more retained on DB-XLB compared to DB-1. Alcohols, phenols, ketones, aldehydes, ethers, esters, halogenates and amines all have higher retention index values than DB-1. Unsaturated hydrocarbons are the only type of compounds more retained by DB-1.

Even some of the non-aromatic hydrocarbons evaluated have higher retention index values when analyzed on DB-XLB. Only the unsaturated, non-cyclic compounds have higher retention indices on DB-1. These compounds are identified by their negative "I Diff" values in Table 1. All the hydrocarbons that are cyclic or contain double bonds have higher retention index values on DB-XLB.

Generally speaking, the oxygen and nitrogen-containing compounds are more retained by DB-XLB, with the retention difference being even greater when additional functional groups are attached. Carbonyl-containing groups such as ketones, aldehydes and esters are retained more by DB-XLB than ether groups.

An interesting side note is the effect cyclic structures have on retention. This is observed in the hydrocarbons and more noticeably in the ketones. The straight-chained ketones are more retained by DB-XLB, but the structural change to a cyclic form significantly increases the retention index difference between the two phases.

**Table 1. Retention Index Values for DB-1 and DB-XLB - By Class****Hydrocarbons**

Compound	DB-XLB, I <sub>T</sub>	DB-1, I <sub>T</sub>	I <sub>T</sub> Difference	Functional Groups
4-methylheptane	758.0	760.4	-2.4	
2-methyl octane	858.3	860.2	-1.9	
2-methyl nonane	960.3	962.6	-2.2	
3-methyl nonane	967.2	969.0	-1.8	
5-methyl nonane	955.2	958.1	-2.9	
2,2,5-trimethylhexane	770.1	780.1	-10.0	
ethylcyclopentane	729.1	724.5	4.6	Cy
R-(+)-limonene	1036.8	1015.8	21.0	Cy, =(2)
S-(-)-limonene	1036.8	1015.8	21.0	Cy, =(2)
alpha-terpinene	1022.9	1004.2	18.7	Cy, =(2)
gamma-terpinene	1066.6	1044.0	22.6	Cy, =(2)
terpinolene	1092.6	1077.8	14.8	Cy, =(2)
alpha-phellandrene	1012.0	991.1	20.9	Cy, =(2)
1R-(+)-beta pinene	975.1	961.8	13.3	Cy>, =
1S-(-)-beta pinene	975.1	961.7	13.3	Cy>, =
1R-(+)-alpha pinene	928.7	924.2	4.5	Cy>, =
1S-(-)-alpha pinene	928.7	924.3	4.4	Cy>, =
3-carene	1011.4	999.4	12.0	Cy>, =
(+)-camphene	944.3	935.2	9.0	Cy>, =
(-)-trans-caryophyllene	1428.1	1402.9	25.2	Cy <sub>9</sub> , Cy <sub>4</sub> =(2)
trans-decahydronaphthalene	1061.5	1038.7	22.8	Cy(2)

**Aromatics**

Compound	DB-XLB, I <sub>T</sub>	DB-1, I <sub>T</sub>	I <sub>T</sub> Difference	Functional Groups
o-xylene	903.6	871.0	32.6	Ar
m-xylene	878.8	848.2	30.6	Ar
p-xylene	879.7	849.3	30.3	Ar
ethylbenzene	864.5	839.8	24.7	Ar
propylbenzene	958.5	933.5	24.9	Ar
1,3-diisopropylbenzene	1154.3	1136.2	18.1	Ar
1,4-diisopropylbenzene	1181.9	1154.4	27.5	Ar
butylbenzene	1063.5	1036.4	27.1	Ar
sec-butylbenzene	1015.5	992.1	23.3	Ar
isobutylbenzene	1013.7	989.5	24.2	Ar
tert-butylbenzene	996.3	972.4	24.0	Ar
4-tert-butyltoluene	1100.0	1072.5	27.5	Ar
cumene	927.8	906.9	21.0	Ar
mesitylene	984.2	948.9	35.4	Ar
p-cymene	1033.5	1006.8	26.8	Ar
1,3-dimethylnaphthalene	1454.4	1379.0	75.3	Ar(2)
biphenyl	1406.9	1338.4	68.5	Ar(2)
naphthalene	1203.7	1146.0	57.7	Ar(2)
phenanthrene	1833.9	1715.2	118.7	Ar(3)
pyrene	2189.2	2031.3	158.0	Ar(4)

**Alcohols**

Compound	DB-XLB, I <sub>T</sub>	DB-1, I <sub>T</sub>	I <sub>T</sub> Difference	Functional Groups
1-pentanol	771.7	744.1	27.6	OH
1-hexanol	877.0	847.9	29.0	OH
1-heptanol	981.4	951.8	29.6	OH
1-octanol	1083.1	1053.2	29.9	OH
1-nonanol	1184.3	1154.5	29.8	OH
1-decanol	1285.1	1255.2	29.9	OH
1-undecanol	1386.1	1356.0	30.1	OH
1-dodecanol	1487.6	1457.1	30.5	OH
2-hexanol	809.1	781.0	28.1	OH
2-heptanol	911.6	882.5	29.1	OH
citronellol	1240.3	1209.0	31.3	OH, =
(+/-)linalool	1111.1	1081.8	29.3	OH, =(2)
geraniol	1267.0	1232.9	34.1	OH, =(2)
(+)-menthol	1199.5	1150.2	49.3	OH, Cy
(-)-menthol	1199.1	1150.4	48.7	OH, Cy
DL-isoborneol	1167.1	1130.7	36.4	OH, Cy>
terpinen-4-ol	1197.4	1153.0	44.4	OH, Cy, =
alpha-terpineol	1215.5	1164.4	51.2	OH, Cy, =
(+/-)1,2-butanediol	849.3	812.8	36.5	OH(2)
2,3-butanediol	793.2	747.5	45.7	OH(2)
1,4-butanediol	961.4	912.4	48.9	OH(2)
2,4-dimethylphenol	1165.1	1123.0	42.1	Ar, OH
o-cresol	1064.0	1031.1	32.9	Ar, OH
m-cresol	1090.8	1053.1	37.7	Ar, OH
p-cresol	1088.1	1051.8	36.3	Ar, OH
phenol	988.6	959.6	29.0	Ar, OH
sec-phenethyl alcohol	1078.7	1027.7	51.0	Ar, OH
phenethyl alcohol	1131.3	1076.2	55.1	Ar, OH

**Ketones**

Compound	DB-XLB, I <sub>T</sub>	DB-1, I <sub>T</sub>	I <sub>T</sub> Difference	Functional Groups
2-hexanone	805.7	760.6	45.1	C=O
3-hexanone	801.5	757.4	44.2	C=O
2-methyl-3-pentanone	757.2	726.0	31.2	C=O
4-methyl-2-pentanone	747.5	716.0	31.5	C=O
2-heptanone	886.6	846.9	39.6	C=O
2-octanone	1009.3	966.0	43.3	C=O
2-nonanone	1110.4	1068.4	42.0	C=O
5-nonanone	1089.7	1051.4	38.3	C=O

## Ketones Continued

Compound	DB-XLB, I <sub>T</sub>	DB-1, I <sub>T</sub>	I Difference	Functional Groups
2,6-dimethyl-4-heptanone	982.7	951.2	31.4	C=O
5-methyl-2-hexanone	870.0	830.9	39.1	C=O
2-decanone	1211.3	1170.4	41.0	C=O
2-undecanone	1312.0	1271.7	40.3	C=O
cyclopentanone	809.9	749.6	60.2	C=O, Cy
3-methylcyclopentanone	866.6	811.9	54.7	C=O, Cy
cyclohexanone	912.6	851.7	60.9	C=O, Cy
isophorone	1153.9	1080.7	73.2	C=O, Cy, =
R-(-)-carvone	1281.0	1205.7	75.4	C=O, Cy, =(2)
acetophenone	1093.4	1025.2	68.2	Ar, C=O
benzylacetone	1272.7	1199.4	73.3	Ar, C=O

## Aldehydes

Compound	DB-XLB, I <sub>T</sub>	DB-1, I <sub>T</sub>	I Difference	Functional Groups
heptaldehyde	920.4	874.1	46.3	CHO
nonylaldehyde	1125.8	1080.2	45.6	CHO
trans-2-penten-1-al	776.1	721.4	54.7	CHO, =

## Esters

Compound	DB-XLB, I <sub>T</sub>	DB-1, I <sub>T</sub>	I <sub>T</sub> Difference	Functional Groups
(+/-)sec-butyl acetate	759.4	738.1	21.3	COO
ethyl-2-methylpentanoate	942.6	923.0	19.6	COO
ethyl-3-methylpentanoate	965.5	940.3	25.2	COO
isoamyl acetate	885.9	855.5	30.4	COO
methyl laurate	1537.6	1507.0	30.7	COO
propyl propionate	816.0	792.4	23.7	COO
cis-3-hexenyl butyrate	1196.3	1167.3	29.0	COO, =
trans-2-hexenyl acetate	1029.3	995.8	33.5	COO, =
trans-2-hexenyl propionate	1121.8	1088.7	33.0	COO, =
geranyl butyrate	1569.9	1536.9	33.0	COO, =(2)
linalyl acetate	1260.6	1239.4	21.2	COO, =(2)
gamma-nonactone	1405.2	1308.2	97.0	COO, Cy
gamma-decalactone	1510.6	1414.6	95.9	COO, Cy
gamma-undecalactone	1615.7	1521.0	94.7	COO, Cy
gamma-dodecalactone	1721.3	1627.3	94.0	COO, Cy
delta-nonactone	1646.3	1546.1	100.1	COO, Cy
delta-undecalactone	1721.4	1627.4	94.0	COO, Cy
delta-dodecalactone	1752.3	1652.8	99.5	COO, Cy
dimethyl adipate	1268.6	1208.2	60.4	COO(2)
diethyl adipate	1407.2	1353.6	53.6	COO(2)
dibutyl adipate	1786.9	1731.6	55.3	COO(2)
methyl benzoate	1115.3	1061.4	53.9	Ar, COO
propyl benzoate	1289.2	1237.2	52.0	Ar, COO
ethyl benzoate	1189.3	1138.2	51.2	Ar, COO
benzyl acetate	1184.1	1128.2	56.0	Ar, COO
bis(2-ethylhexyl)phthalate	2558.5	2499.4	59.0	Ar, COO(2)
dimethylphthalate	1484.5	1400.0	84.5	Ar, COO(2)
dibutylphthalate	1985.8	1909.1	76.7	Ar, COO(2)
dioctylphthalate	2558.8	2500.0	58.8	Ar, COO(2)
benzyl butyl phthalate	2387.4	2273.6	113.7	Ar(2), COO(2)

## Ethers

Compound	DB-XLB, I <sub>T</sub>	DB-1, I <sub>T</sub>	I <sub>T</sub> Difference	Functional Groups
pentyl ether	1080.6	1069.7	10.9	COC
eucalyptol (cineole)	1034.0	1013.0	20.9	COC, Cy>
acetal	727.4	717.3	10.1	COC(2)
anethole	1319.2	1252.4	66.8	Ar, COC, =

## Halogenated Hydrocarbons

Compound	DB-XLB, I <sub>T</sub>	DB-1, I <sub>T</sub>	I <sub>T</sub> Difference	Functional Groups
1-chlorohexane	864.8	835.9	28.9	Cl
1-chlorooctane	1074.0	1043.8	30.2	Cl
1-chlorodecane	1279.1	1249.6	29.5	Cl
1-chlorotetradecane	1689.0	1659.8	29.2	Cl
1-chlorooctadecane	2099.5	2069.8	29.7	Cl
1,3-dichloropropane	802.7	747.2	55.5	Cl(2)
1,4-dichlorobutane	927.8	867.4	60.4	Cl(2)
1,2-dichloropropane	802.6	747.3	55.3	Cl(2)
1,4-dichloro-2-butene	922.3	864.6	57.7	Cl(2), =
1,2,3-trichloropropane	942.2	878.4	63.7	Cl(3)
1,1,1,2-tetrachloroethane	860.4	821.5	38.9	Cl(4)
hexachloro-1,3-butadiene	1212.3	1196.1	16.3	Cl(6), =(2)
benzyl chloride	1029.8	977.9	52.0	Ar, Cl
chlorobenzene	852.5	821.2	31.3	Ar, Cl
2-chlorotoluene	959.8	926.1	33.7	Ar, Cl
3-chlorotoluene	966.7	927.7	39.0	Ar, Cl
4-chlorotoluene	972.4	931.0	41.3	Ar, Cl
4-chlorostyrene	1097.3	1043.7	53.5	Ar, Cl, =
1,2-dichlorobenzene	1046.7	1002.8	44.0	Ar, Cl(2)
1,3-dichlorobenzene	1020.7	975.4	45.3	Ar, Cl(2)
1,2,4-trichlorobenzene	1202.3	1141.6	60.7	Ar, Cl(3)
alpha, alpha, alpha-trichlorotoluene	1234.0	1187.4	46.6	Ar, Cl(3)
(+/-)2-bromopentane	794.3	760.8	33.5	Br
1,3-dibromopropane	965.6	906.1	59.6	Br(2)
1,8-dibromooctane	1554.9	1473.3	81.6	Br(2)
(+/-)-1,2-dibromopropane	871.1	823.0	48.1	Br(2)
1,4-dibromobutane	1101.5	1026.4	75.1	Br(2)

## Halogenated Hydrocarbons Continued

Compound	DB-XLB, I <sub>T</sub>	DB-1, I <sub>T</sub>	I <sub>T</sub> Difference	Functional Groups
ethylene bromide	824.9	770.9	54.0	Br(2)
bromoform	907.8	845.2	62.6	Br(3)
bromobenzene	940.4	903.9	36.5	Ar, Br
2-bromo-1-chloropropane	792.9	743.6	49.4	Cl, Br
1,2-dibromo-3-chloropropane	1113.1	1033.9	79.2	Cl, Br(2)
1,4-dibromo-1,1,2,2-tetrafluorobutane	851.0	823.9	27.1	Br(2), F(4)
1-chloro-2-fluorobenzene	871.5	835.2	36.3	Ar, Cl, F
1-bromo-4-fluorobenzene	940.1	896.9	43.2	Ar, Br, F

## Amines

Compound	DB-XLB, I <sub>T</sub>	DB-1, I <sub>T</sub>	I <sub>T</sub> Difference	Functional Groups
N,N-dimethylbenzyl amine	1048.3	1017.9	30.4	Ar, N
<i>N</i> -benzylmethyl amine	1070.5	1026.0	44.5	Ar, NH
aniline	993.5	939.2	54.3	Ar, NH
indole	1318.1	1244.3	73.8	Ar, NH, =
o-toluidine	1090.7	1032.7	58.0	Ar, NH <sub>2</sub>
2,4-dimethylaniline	1165.2	1123.1	42.1	Ar, NH <sub>2</sub>
2-aminonaphthalene	1593.8	1484.8	109.0	Ar(2), NH <sub>2</sub>
benzidine	2190.0	2018.8	171.1	Ar(2), NH <sub>2</sub> (2)

## Others

Compound	DB-XLB, I <sub>T</sub>	DB-1, I <sub>T</sub>	I <sub>T</sub> Difference	Functional Groups
2-butoxyethanol	914.5	884.4	30.1	OH, CO
furfuryl alcohol	864.1	826.1	38.0	OH, COC, Cy, 2=
1,3-dichloro-2-propanol	914.5	852.9	61.6	OH, Cl(2)
diisopropyl D-tartrate	1428.6	1385.2	43.5	OH(2), COO(2)
diisopropyl L-tartrate	1428.6	1385.2	43.4	OH(2), COO(2)
2-ethoxyethyl acetate	921.6	880.8	40.9	COO, COC
1,1-dichloroacetone	743.2	707.2	36.0	C=O, Cl(2)
2-chloroethyl ether	1013.0	945.9	67.1	COC, Cl(2)
dibromoacetonitrile	945.1	853.3	91.7	Br(2), CN
ethyl salicylate	1291.2	1236.8	54.5	Ar, OH, COO
2-chlorophenol	999.6	960.5	39.1	Ar, OH, Cl
benzoyl chloride	1097.6	1036.5	61.0	Ar, C=O, Cl
4-chlorobenzylamine	1471.1	1400.0	71.1	Ar, NH <sub>2</sub> , Cl
4-chloro-2-methylaniline	1329.6	1247.3	82.3	Ar, NH <sub>2</sub> , Cl
2,4-dichlorobenzylamine	1602.9	1532.6	70.3	Ar, NH <sub>2</sub> , Cl(2)
benzonitrile	1012.0	937.3	74.8	Ar, CN
4-chlorodiphenyl ether	1625.8	1546.5	79.2	Ar(2), COC, Cl
4-bromophenyl phenyl ether	1728.0	1639.0	88.9	Ar(2), COC, Br

## Legend

Symbol	Meaning
Ar	Aromatic Ring
Br	Bromine
CHO	Aldehyde
Cl	Chlorine
CN	Cyano
C=O	Ketone
COC	Ether
COO	Ester
Cy	Cyclic
Cy>	Bridged Ring
F	Fluorine
N	Tertiary Amine
NH	Secondary Amine
NH <sub>2</sub>	Primary Amine
OH	Hydroxyl
=	Double Bond

Halogenated hydrocarbons are also more highly retained by DB-XLB. Brominated compounds have slightly higher retention differences than chlorinated compounds. The more halogenated groups attached, the higher the retention index difference is between DB-1 and DB-XLB. This is true to the point of adding about 3 functional groups. The addition of more groups does not increase and may even decrease the difference in retention index. A more targeted analysis needs to be done to validate this observation.

The selectivity of DB-XLB is quite different from DB-1, as indicated by the retention index differences of the 186 compounds analyzed. Ideally, a confirmation column should change the elution pattern while still maintaining full resolution of all the compounds in the mixture. If most of the compounds do not move relative to each other, compound confirmation is not achieved. The difference in phase selectivity makes DB-XLB a suitable confirmation column to DB-1.

Not only does DB-XLB make a nice confirmation column to DB-1, but it is versatile and can be used for many different applications. Figure 1 demonstrates its use for a common chlorinated pesticide analysis. This analysis is run to higher temperatures where column bleed can have an effect on compound detection limits. DB-XLB can be used for lower temperature applications as well. Figure 2 shows DB-XLB used for a food and flavor application. DB-XLB is a great all-purpose phase that is useful as a primary or confirmation column and can extend compound detection limits because of its extremely low-bleeding character.

**Figure 1.** *CLP Pesticides*

**Column:** DB-XLB  
**30 m x 0.25 mm I.D., 0.25  $\mu$ m**

**J&W P/N:** 122-1232

**Carrier:** Helium at 45 cm/sec, measured at 110°C

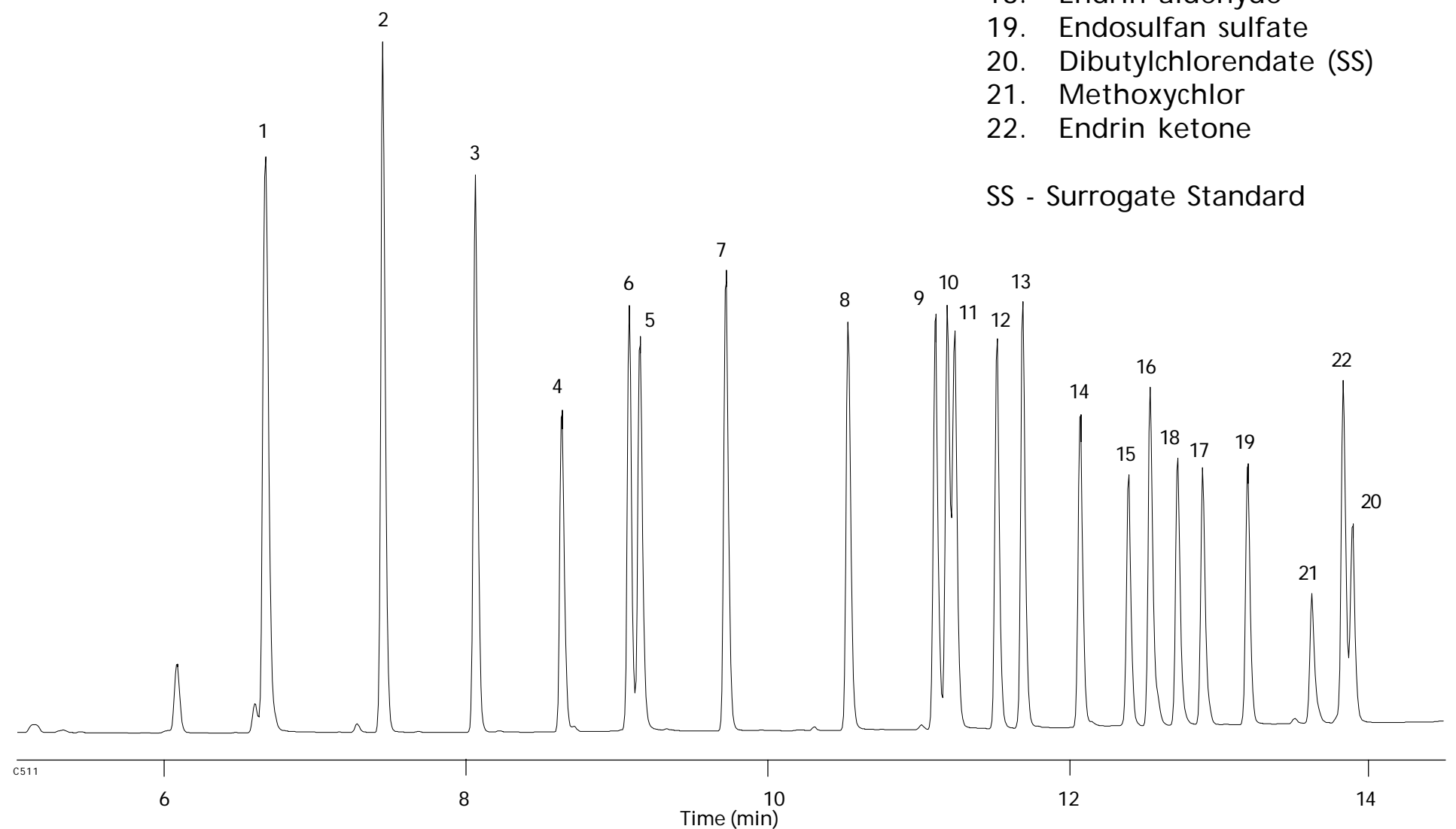
**Oven:** 110°C for 0.5 min  
 110-150°C at 25°/min  
 150-260°C at 12°/min  
 260-320°C at 15°/min  
 320°C for 2 min

**Injector:** Splitless, 250°C  
 30 sec purge activation time  
 50 pg composite 8081 standard,  
 and CLP-034 surrogate mix, Accustandard Inc.

**Detector:** ECD, 350°C  
 Nitrogen makeup gas at 40 mL/min

**Compounds**

1. Tetrachloro-m-xylene (SS)
2.  $\alpha$ -BHC
3.  $\gamma$ -BHC
4.  $\beta$ -BHC
5. Heptachlor
6.  $\delta$ -BHC
7. Aldrin
8. Heptachlor epoxide
9.  $\gamma$ -Chlordane
10.  $\alpha$ -Chlordane
11. Endosulfan I
12. *p, p'*-DDE
13. Dieldrin
14. Endrin
15. *p, p'*-DDD
16. Endosulfan II
17. *p, p'*-DDT
18. Endrin aldehyde
19. Endosulfan sulfate
20. Dibutylchlorendate (SS)
21. Methoxychlor
22. Endrin ketone

**Figure 2.** *Polymethoxyflavones in Orange Oil*

**Column:** DB™-XLB  
**30 m x 0.18 mm I.D., 0.18  $\mu$ m film**

**Carrier:** Helium, 35 cm/sec at 100°C

**Injector:** Split, 280°C,  
 Split ratio 80:1

**Oven:** 100-310°C at 8°/min  
 310°C for 10 min

**Detector:** MSD, at 310°C

**Compounds**

1. Tetra-O-methylscutellarein
2. Tangeretin
3. Sinensetin
4. Nobiletin
5. Hexamethoxyflavone
6. Heptamethoxyflavone

