Table 2. Biological Activity of the Copolymers

Copolymer	ID_{50} (μg/ml)[a]		
	3LL[b]	B16[c]	MEF[d]
(1)	45.8	39.6	16.9
(2)	822	610	–
(4)	276	1141	227
(5)	1047	1700	804
DIVEMA[e]	2504	1511	765

[a]ID_{50} was defined as the concentration which reduced absorbance by 50% of control untreated wells in the MTT assay. All results represent the average of 8 wells. [b]Lewis lung carcinoma originated from C57BL/6 mouse. [c]Malignant melanoma originated from C57BL/6 mouse. [d]Mouse embryo fibroblast from C57BL/6 mouse. [e]An alternating copolymer of divinyl ether and maleic anhydride (1:2).

mers obtained have an alternating sequence between TAG and MA. The number-average molecular weight (Mn) of the copolymers were found to be low (Table 1). This is attributable to the chain transfer reaction which generally occurs in the radical polymerization of dihydropyran derivatives[3].

The hydrolyses of (3) were accomplished under different conditions as shown in Scheme 1. These reactions were monitored by IR and NMR spectra where peaks at 1825 cm^{-1} for cyclic anhydride and at 1.95 ppm for acetyl protons disappeared while a peak at 1730 cm^{-1} for carboxyl group emerged. The polymers (4) and (5) are soluble in DMF, DMSO, methanol and water, and insoluble in acetone, THF, ethyl acetate and other nonpolar solvents.

The biological activity of these copolymers were measured by MTT method[5] and ID_{50}-values against tumor cells (B16, 3LL) and normal cells are given in Table 2. The cytotoxicities of the copolymers *in vitro* are found to be low in comparison with those of the polymer (1) containing one acetoxyl group on THP ring[6], but higher than that of DIVEMA[7], an alternating copolymer of divinyl ether and maleic anhydride (1:2), which is known to exhibit a high antitumor activity. Studies on their anticancer effect *in vivo* are currently in progress.

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Dual Capillary Column System for the Qualitative Gas Chromatography: 1. Comparison Between Split and Splitless Injection Modes

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A dual capillary column system is described for the simultaneous measurement of retention index (RI) and area ratio (AR) values of each peak on two capillary columns of different polarity, DB-5 & DB-1701. Both capillary columns were connected to a common splitless injector *via* a deactivated fused-silica capillary tubing of 1 m length and a 'Y' splitter, the dead volume effect of which was found to be negligible. RI and AR were measured with high reproducibility(≤0.05% RSD) and with high accuracy (<10% RE), respectively. When applied to the test samples of the organic acid mixture, each acid was positively identified by the combined computer RI library search-AR comparison.

Introduction

With the advent of high resolution fused silica capillary columns and modern high performance gas chromatographs, gas chromatography (GC) which is primarily a separation technique, is now implemented into routine laboratory qualitative analysis of samples such as essential oils, organic acids, pollutants, and drugs[1-12]. Temperature programmed retention index (RI) system is most conveniently used as criteria for the identification of GC peaks without resorting to gas chromatography-mass spectrometry (GC-MS).

Confidence in the peak identification is greatly enhanced

by matching characteristic pairs of RI values of peaks measured on the columns of different polarity[2,8]. This has required painstaking separate analysis of samples on each column. Currently, the dual channel analysis system[4,5] provides a practical solution for the simultaneous sample run on two capillary columns. In this system two columns are connected to a common injector port, and a single sample injection permits recording two chromatograms from each column simultaneously. The overall analysis time is reduced considerably.

The concept that quantitative measures of corresponding peaks on each column should be in good agreement can be used in confirming peak assignments derived from RI matches[12]. The accurate quantitative comparison of corresponding peaks requires the use of splitless or on-column injection technique which can introduce samples into columns without discrimination, unlike the split injection mode[13-15]. The most common injection technique used for RI measurement is, however, the split injection mode.

We have been working on the rapid profiling of organic acids in our laboratory[9]. The present work was undertaken to investigate dual capillary column system in the splitless injection mode for the RI library peak identification supplemented by AR comparison.

Experimental

Materials. All organic acids tested and triethylamine (TEA) were pruchased from commercial vendors such as Sigma (St. Louis, MO, USA) and Aldrich (Milwaukee, WI, USA). The silylation agent, N-methyl-N-(tert-butyldimethylsilyl)trifluoro-acetamide (MTBSTFA) is available from Pierce (Rockford, IL, USA). All the other solvents and chemicals were of analytical grade. Polarity test and capillary sample mixtures were supplied from Supelco (Bellefonte, PA, USA) and Hewlett-Packard (Avondale, PA, USA), respectively.

Instrumentation. A Hewlett-Packard model 5890A equipped with split/splitless capillary inlet system, two flame ionization detectors (FIDs), a 3392A integrator, a HP 5895A GC Chemstation, and a Think Jet printer (Hewlett-Packard, Avondale, PA, USA) was used for this study. The two FID signals were processed simultaneously in dual channel mode by the GC Chemstation. DB-5 and DB-1701 fused silica capillary columns (J & W Scientific, Rancho, Cordova, CA, USA) were of 30 m×0.25 mm I.D. and 0.241 μm film thickness. For dual capillary column system, a deactivated fused silica tubing (1 m×0.25 mm I.D.) was connected to an injector and then to each capillary column *via* a Chromfit 'Y' (Unimetrics, Shorewood, IL, USA). In the split injection mode, the injector liner was packed with silane treated glass wool, and the split ratio and injector temperature were set at 30:1 and 280℃, respectively. In the splitless injection mode, the injector liner was empty, purge delay time was 42 sec, and the temperature was 220℃. FIDs were maintained at 300℃. Nitrogen carrier flow rates were adjusted to 0.84-0.90 ml/min. Polarity test mixture was run in split mode isothermally at 110℃. For the analysis of capillary sample mixture, the oven temperature was held initially at 100℃ for 5 min, then programmed to 180℃ at a rate of 10℃/min in split mode, and initially at 60℃ for 2 min, then programmed to 180℃ at a rate of 5℃/min in splitless mode. The acid test mixture and blind samples were run in splitless mode at the oven temperature of 60℃ initially, then programmed to 280℃ at a rate of 4℃/min. Every sample was run in triplicate mode.

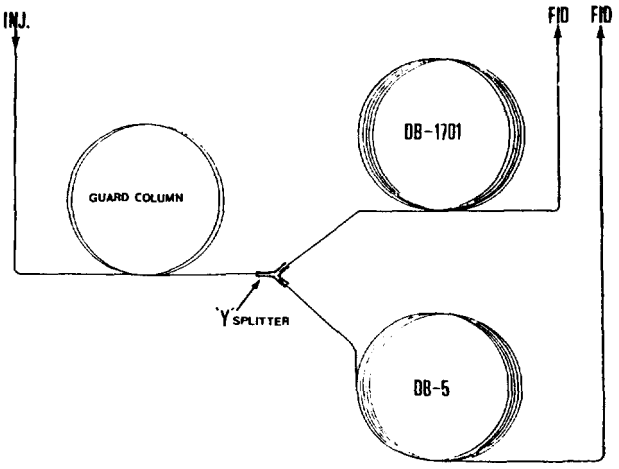

Figure 1. Schematic diagram of a dual capillary column system.

Preparation of Acid Test Mixture and Blind Samples. Prior to GC analysis, samples containing 7-10 different acids after adding palmityl methylester as an internal standard were subjected to silylation to form tert-butyldimethylsilyl (TBDMS) derivatives as described in elsewhere[16].

RI Library Searching. *Via* Chemstation BASIC programs, programmed retention indices of sample peaks in each channel were calculated by linear interpolation between the retention times of adjacent hydrocarbon standards (C_8 to C_{30} in isooctane) co-injected with samples. And they were compared with the database of a reference RI library for matches to aid in identifying the unknown peaks as described previously[9]. For the further confirmation of the assigned peaks, area ratios of corresponding peaks on each column were compared. In this case, acceptable maximum percent relative error(% RE) for agreement was limited to 10%[9].

Results and Discussion

In our previous report[9], we verified the utility of the computer RI library searching for the peak identifications of organic acids based on two sets of retention indices measured through separate runs on DB-5 and DB-1701 capillary columns. For the simultaneous sample analysis on two columns with a single injection, we prepared a dual capillary column system as illustrated in Figure 1. The connections between the fused silica capillary columns and a 1 m long guard column of equal inner diameter were quickly made by hand-pressing each end into a Y-splitter (a zero dead volume 3-way union made of glass). A leaktight seal between the connections was achieved when heated above 200℃, because the polyimide outer coating of the columns fused to the glass seal. The carrier gas flow rates through the DB-5 and DB-1701 columns were similar. Therfore, sample vapors were splitted at Y-splitter onto the columns at almost equal ratio and the FIDs exhibited virtually equivalent sensitivity.

The overall column performance after the connections was evaluated in the split injection mode with a polarity test mixture containing chemically active compounds and nonactive hydrocarbons. Figure 2 shows a typical chromatogram

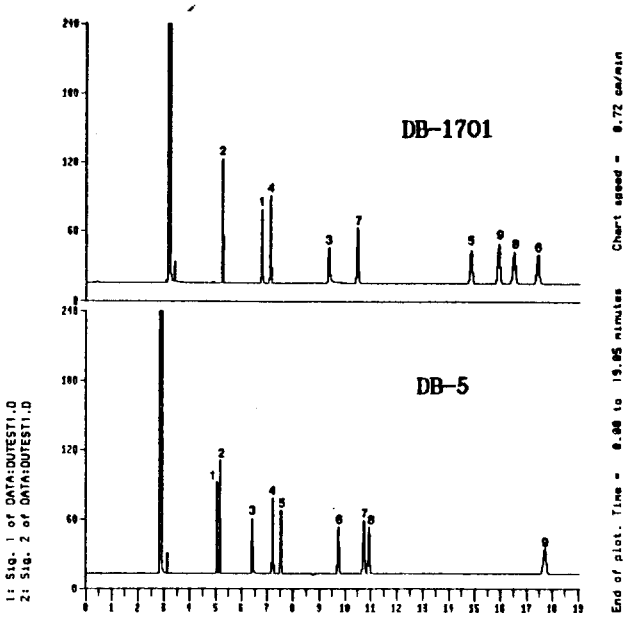

Figure 2. Dual channel chromatograms of a polarity test mixture in split injection mode. GC conditions are in the text. Peak identification: 1=2-octanone; 2=decane; 3=1-octanol; 4-undecane; 5=2,6-DMP; 6=2,6-DMA; 7=dodecane; 8=naphthalene; 9=tridecane.

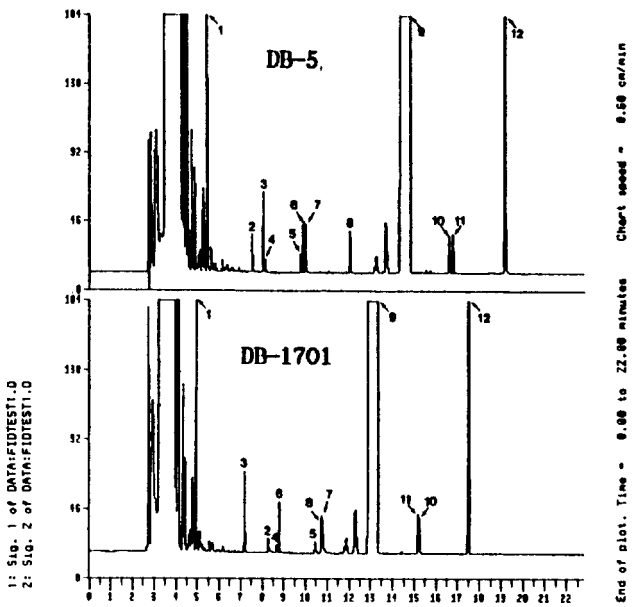

Figure 3. Dual channel chromatograms of a capillary sample mixture in splitless injection mode. GC conditions are in the text. Peak identification: 1=nonane; 2=1-octanol; 3=undecane; 4=nonanal; 5=2-decanone; 6=dodecane; 7=naphthalene; 8=tridecane; 9=tetradecane; 10=pentadecene; 11=pentadecane; 12=hexadecane.

of the mixture on DB-5 and DB-1701, and the results of the test are given in Table 1. Each compound exhibits excellent peak shape. The column efficiency and inertness were maintained, indicating no discernable dead volumn or activity effects[17,18] due to the connections. The guard column serves as a useful maintenance tool for preventing the buildup of nonvolatile material in the analytical columns of high cost. And it also serves as a retention gap for the splitless injection[19,20].

The precision of split and splitless injection modes was tested in measurements of RI and AR using a capillary sample mixture. The mixture was diluted by a factor of 30 in isooctane for the splitless injection. Figure 3 shows typical dual channel chromatograms obtained from the simultaneous analysis on two columns in a single splitless injection. RI values were measured reproducibly with relative standard deviation of 0.01-0.05% in both injection modes as seen in Table 2-1 and 2-2, proving that the splitless injection can be used in RI measurements.

The overall precision in AR measurement was, however, lower in the split mode compared with the splitless mode as listed Table 3-1 and 3-2. Comparison ratio, Q (ratio of the area ratio of corresponding peak on DB-5 to the area ratio on DB-1701) was calculated to check the accuracy in AR measurement. When the true value of Q is assumed to be unity, % relative error (% RE) in the split mode were shown to be much higher than those in the splitless mode

Table 1. Chromatographic Performance Parameters

No.	Name	DB-5				DB-1701			
		t_R[c]	W_h[d]	n/meter[e]	I[f]	t_R	W_h	n/meter	I
1	2-Octanone	5.028	0.027			6.737	0.038		
2	Decane	5.159	0.028			5.221	0.028		
3	1-Octanol	6.401	0.041			9.315	0.062		
4	Undecane	7.191	0.043			7.070	0.041		
5	2,6-DMP[a]	7.509	0.043		1.39	14.805	0.082		1.24
					(1.30)[g]				(1.33)[g]
6	2,6-DMA[b]	9.729	0.056			17.388	0.092		
7	Dodecane	10.701	0.064			10.417	0.062		
8	Naphthalene	10.911	0.064			16.458	0.096		
9	Tridecane	17.689	0.106	5142.61		15.874	0.084	6594.82	
				(3619.47)[g]				(4430.78)[g]	

[a]2,6-Dimethyl phenol, [b]2,6-Dimetyl aniline, [c]restention time (the means of triplicate runs), [d]peak width at half hight (the means of triplicate runs), [e]theoretical plates/meter (the mean of triplicate runs), [f]column inertness (peak hight ratio of 2,6-DMP to 2,6-DMA) (the means of triplicate runs), [g]values before connection to guard column.

Table 2-1. Retention Index Reproducibility in the Split and Splitless Injection Modes on DB-5

(A) Split Mode

Compound	Run Number 1	2	3	Mean RI± SD (% RSD)
1-Octanol	1067.59	1067.87	1067.92	1067.79± 0.15 (<0.01)
Nonanal	1106.40	1106.72	1106.59	1106.57± 0.13 (0.01)
2-Decanone	1193.32	1194.52	1194.05	1193.96± 0.49 (0.04)
Naphthalene	1207.36	1207.67	1207.30	1207.44± 0.16 (0.01)
Pentadecene	1492.82	1492.83	1492.82	1492.82± 0.01 (<0.01)

(B) Splitless Mode

Compound	Run Number 1	2	3	Mean RI± SD (% RSD)
1-Octanol	1065.74	1065.90	1064.81	1065.48± 0.48 (0.02)
Nonanal	1106.40	1106.40	1105.60	1106.13± 0.38 (0.01)
2-Decanone	1194.40	1194.40	1193.96	1194.25± 0.21 (0.03)
Naphthalene	1211.28	1211.28	1210.60	1211.06± 0.32 (0.02)
Pentadecene	1491.07	1491.07	1491.10	1491.08± 0.01 (0.01)

Table 2-2. Retention Index Reproducibility in the Split and Splitless Injection Modes on DB-1701

(A) Split Mode

Compound	Run Number 1	2	3	Mean RI± SD (% RSD)
1-Octanol	1173.20	1173.04	1173.52	1173.25± 0.20 (0.02)
Nonanal	1191.03	1190.93	1191.24	1191.07± 0.13 (0.01)
2-Decanone	1286.02	1286.52	1285.92	1286.15± 0.26 (0.03)
Naphthalene	1308.92	1308.42	1308.52	1308.62± 0.22 (0.02)
Pentadecene	1502.71	1502.52	1502.43	1502.55± 0.12 (<0.01)

(B) Splitless Mode

Compound	Run Number 1	2	3	Mean RI± SD (% RSD)
1-Octanol	1170.43	1170.43	1170.69	1170.52± 0.12 (0.01)
Nonanal	1194.78	1194.78	1194.83	1194.80± 0.02 (<0.01)
2-Decanone	1289.60	1290.32	1290.32	1290.08± 0.34 (0.03)
Naphthalene	1320.81	1321.50	1321.50	1321.27± 0.33 (0.02)
Pentadecene	1502.62	1501.05	1502.60	1502.09± 0.74 (0.05)

as shown in Table 3-3. The splitless injection appears to provide the more accurate quantitative AR comparison as expected. Therfore, we replaced the RI values in our previous RI library[9] with those measured in the splitless injection mode.

With several test mixtures containing known organic acids, the present dual capillary column system in splitless injection mode was tested for its usefulness for the qualitative peak identification based on the combined RI library search-AR comparison. Figure 4 shows typical dual chromatograms of an acid test mixture. RI calculation and library search procedures[9] were performed as presented in the following retention index report (Table 4-1).

Each peak was assigned as the acid giving highest match

Table 3-1. Area Ratio Reproducibility in the Split and Splitless Injection Modes on DB-5

(A) Split Mode

Compound	Run Number 1	2	3	Mean AR± SD (% RSD)
1-Octanol	93.84	88.59	94.80	92.41± 2.73 (2.95)
Nonanal	14.79	10.52	15.30	13.54± 2.14 (15.80)
2-Decanone	15.42	17.02	12.43	14.96± 1.90 (12.70)
Naphthalene	110.57	107.94	107.22	108.58± 1.44 (1.33)
Pentadecene	73.64	80.61	82.05	78.77± 3.67 (4.66)

(B) Splitless Mode

Compound	Run Number 1	2	3	Mean AR± SD (% RSD)
1-Octanol	43.09	40.67	37.91	40.56± 2.12 (5.23)
Nonanal	46.90	48.10	46.92	47.31± 0.56 (1.18)
2-Decanone	32.50	32.23	32.50	32.41± 0.13 (0.40)
Naphthalene	73.01	73.16	73.01	73.06± 0.07 (0.10)
Pentadecene	90.54	87.30	90.50	89.45± 1.52 (1.70)

I.S. = n-Tridecane.

Table 3-2. Area Ratio Reproducibility in the Split and Splitless Injection Modes on DB-1701

(A) Split Mode

Compound	Run Number 1	2	3	Mean AR± SD (% RSD)
1-Octanol	77.21	80.54	72.34	76.70± 3.37 (4.39)
Nonanal	10.31	12.54	10.58	11.14± 0.99 (8.89)
2-Decanone	32.24	30.58	30.42	31.08± 0.82 (2.64)
Naphthalene	74.79	75.42	73.53	74.58± 0.79 (1.06)
Pentadecene	51.21	55.42	50.35	52.33± 2.22 (4.24)

(B) Splitless Mode

Compound	Run Number 1	2	3	Mean AR± SD (% RSD)
1-Octanol	36.82	41.47	39.83	39.37± 1.93 (4.90)
Nonanal	44.89	44.78	44.78	44.82± 0.05 (0.11)
2-Decanone	29.74	29.95	30.47	30.05± 0.31 (1.03)
Naphthalene	67.18	67.35	68.80	67.78± 0.73 (1.08)
Pentadecene	92.44	92.62	92.40	92.49± 0.10 (0.11)

I.S. = n-Tridecane.

Table 3-3. Comparison of Peak Area Ratios of Corresponding Peaks on DB-5 and DB-1701

Compound	Split Mode Q[a]	% RE[b]	Splitless Mode Q	% RE
1-Octanol	1.205	20.5	1.030	3.0
Nonanal	1.215	21.5	1.056	5.6
2-Decanone	0.481	51.9	1.079	7.9
Naphthalene	1.456	45.6	1.078	7.8
Pentadecene	1.505	50.5	0.967	3.3

[a] $AR_{DB\text{-}5}/AR_{DB\text{-}1701}$ [b] $|1-Q| \times 100$.

Table 4-1. Retention Index Report on the Acid Test Mixture

No.	DB-5 Column				DB-1701 Column			
	t_R	RI	Name	(MQ)*	t_R	RI	Name	(MQ)*
1	9.706	930.00	Acetic	(9231)	9.646	980.57	Acetic	(9497)
2	29.456	1486.47	Benzoic	(9987)	28.730	1531.26	Lactic	(8590)
			Caprylic	(8297)			Caprylic	(8)
			Lactic	(7)				
3	29.583	1490.18	Lactic	(9622)	30.040	1572.83	Benzoic	(9002)
4	34.678	1652.85	α-OH iso-caproic	(9993)	33.441	1684.89	α-OH isocaproic	(9998)
5	40.935	1872.29	3-Methyl glutaric	(9777)	41.016	1960.67	3-methyl gultaric	(9642)
			5-Phenyl valeric	(5)			Glutaric	(15)
6	46.186	2077.60	Myristoleic	(9989)	45.429	2139.59	Myristoleic	(9870)
							Myristic	(9401)
7	47.055	3113.38	α-OH phenyl acetic	(8748)	47.624	2210.12	α-OH phenyl acetic	(9007)
8	50.249	2248.34	4-OH-3-methoxy phenyl acetic	(9061)	49.867	2334.07	Palmitoleic	(9993)
9	50.746	2270.54	Palmitoleic	(9938)	50.576	2362.89	4-OH-methoxy phenyl acetic	(8820)
10	57.386	2579.73	Dodecanedioic	(9504)	57.299	2689.20	Dodecanedioic	(8958)

*match quality.

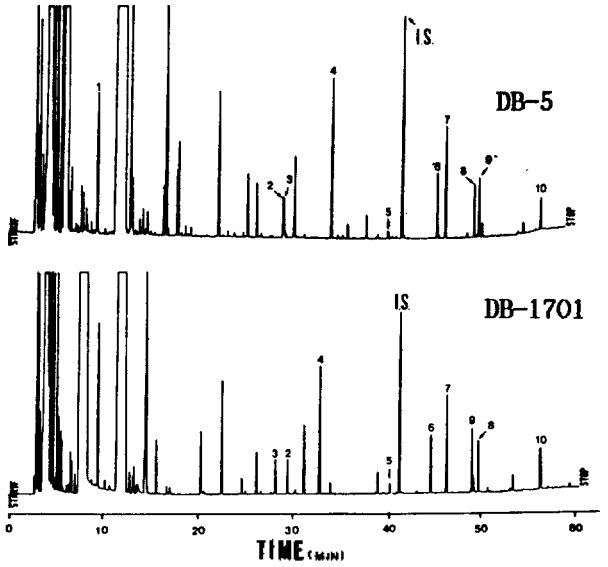

Figure 4. Dual chromatograms of an acid test mixture. GC conditions are in the text. Peak identification: 1=acetic; 2=benzoic; 3=lactic; 4=α-OH isocaproic; 5=3-methyl gulutaric; 6=myristoleic; 7=δ-OH phenyl acetic; 8=4-OH-3-methoxy phenyl acetic; 9=palmitoleic; 10=dodecanedioic.

quality (MQ) on both columns and further confirmed by % RE of AR comparison ratio, Q as listed in the confirmation report (Table 4-2). YES for AMT ? is when agreement (% RE) was within 10%. Organic acids in the test mixture were correctly confirmed by AR comparison except for the lactic acid. The reason for the high % RE of the lactic acid was found due to the coelution of an impurity peak with lactic acid on DB-5 column.

Further utility test of the present qualitative GC system was made with several acid blind samples. Two of them are well exemplified in Figure 5 and 6. Their confirmation reports are presented in Table 5 and 6 respectively. AR comparison will be particulary very useful to confirm assignme-

Table 4-2. Confirmation Report

Name	RI DB-5	RI DB-1701	AMT ?	% RE
Acetic	930.00	980.57	Yes	0.1
Benzoic	1486.47	1572.83	Yes	0.1
Lactic	1490.18	1531.26	No	18.7
α-OH isocaproic	1652.85	1684.89	Yes	4.6
3-Methyl glutaric	1872.29	1960.67	Yes	6.3
Myristoleic	2077.60	2139.59	Yes	0.3
α-OH phenyl acetic	2113.28	2210.12	Yes	2.7
4-OH-3-methoxy phenyl acetic	2248.34	2362.89	Yes	5.5
Palmitoleic	2270.54	2334.07	YES	10.0
Dodecanedioic	2579.73	2689.20	YES	10.0

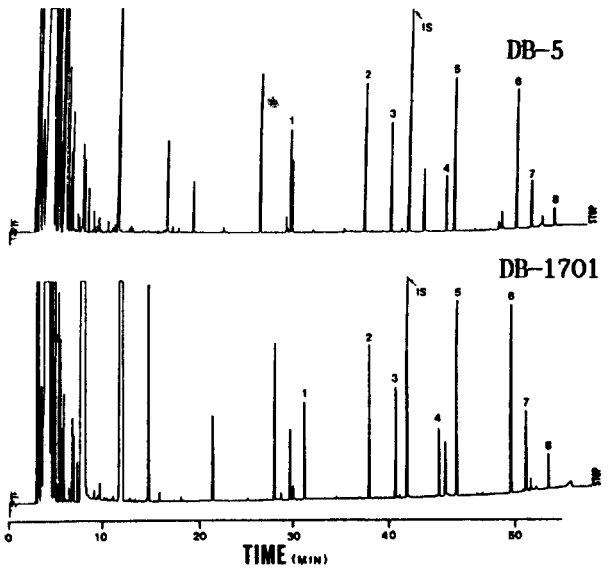

Figure 5. Dual chromatograms of acid blind sample I. GC conditions are in the text. Peak identification: 1=glycolic; 2=succinic; 3=glutaric; 4=myristic; 5=p-OH benzoic; 6=γ-resorcylic; 7=α-resorcylic; 8=p-OH phenyl lactic.

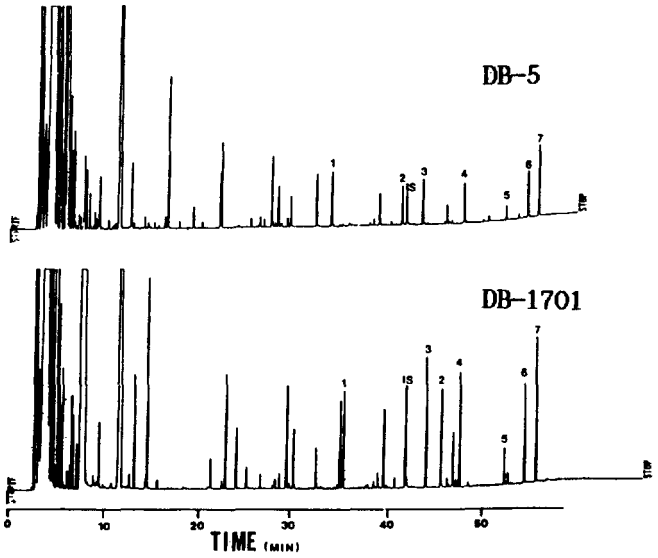

Figure 6. Dual chromatograms of acid blind sample II. GC conditions are in the text. Peak identification: 1=malonic; 2=3-methyl adipic; 3=p-amino benzoic; 4=malic; 5=tartaric; 6=α-resorcylic; 7=protocatechuic.

Table 5. Confirmation Report on Acid Blind Sample I

No.		RI		AMT ?
		DB-5	DB-1701	
1	Glycolic	1506.25	1563.63	Y
2	Succinic	1759.25	1851.77	Y
3	Glutaric	1857.18	1956.49	Y
4	Myristic	2086.96	2139.24	Y
5	p-OH benzoic	2121.77	2218.66	Y
6	γ-Resorcylic	2410.74	2484.81	Y
7	α-Resorcylic	2484.53	2562.65	Y
8	p-OH phenylactic	2602.06	2675.39	Y

Table 6. Confirmation Report on Acid Blind Sample II

No.		RI		AMT ?
		DB-5	DB-1701	
1	Malonic	1640.49	1728.23	Y
2	3-Methyladipic	1990.69	2092.55	Y
3	p-Aminobenzoic	1907.71	2121.20	Y
4	Malic	2171.33	2239.50	Y
5	Tartaric	2375.52	2461.16	Y
6	α-Resorcylic	2484.82	2562.70	Y
7	Protocatechuic	2537.57	2620.31	Y

nts derived from RI matches when two assignments are suggested by RI library search.

In conclusion, we can state that the present dual capillary column system in the splitless injection mode permits the combined RI library search-AR comparion to be implemented in routine organic analysis for the rapid positive peak identification without resorting to GC-MS.

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