GAS CHROMATOGRAPHIC RETENTION INDICES OF 2-PHENYL -2-ALKYLACETONITRILES ON PACKED COLUMNS

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Abstract. *In order to determine the gas chromatografic retention indices for 2-phenyl-2-alkylacetonitriles on packed columns, a number of 2-phenyl-2-alkylacetonitriles were synthesized using the reaction of C-alkylation of phenylacetonitrile. The products were characterized by their boiling points, refractive indices, IR and NMR spectra. The gas chromatografic retention indices (I) for the synthesized nitriles were determined on packed columns (OV-17, OV-210 and OV-225) isothermally at 160, 180 and 200 ^{o}C. The isothermal data obtained on these columns were compared and a comparison of the retention index values, ΔI and dI/dT is presented. The best resolution of the investigated compounds was obtained using an OV-225 packed column.*

Key words: *2-phenyl-2-alkylacetonitriles, phase-transfer catalysis,*
gas chromatography retention indices

1. INTRODUCTION

Retention parameters, preferably Kovats indices [1,2], have become an accepted procedure for the identification of gas chromatografic peaks using isothermal retention data. The Kovats retention index [1,2] system is a logarithmic system where the logarithmic retention of a substance is interpolated between those of two standard compounds. Several homologous series of organic compounds can be used as internal standards, but n-paraffins have been used exclusively. The adjusted retention times were used to calculate the values for the retention index (1):

$$\mathbf{I}_{column}^{T} = 100 \cdot z + 100 \ \frac{\log t_{R'}(x) - \log t_{R'}(nP_z)}{\log t_{R'}(nP_{z+1}) - \log t_{R'}(nP_z)} \tag{1}$$

where **x** is the compound of interest which eluated between $\mathbf{nP_z}$ and $\mathbf{nP_{z+1}}$ n-alkanes, $\mathbf{t_R^{'}}$ is the adjusted retention time of each component and $\mathbf{nP_z}$ and $\mathbf{nP_{z+1}}$ are n-alkane standards which eluated before and after the compound of interest and consist of **z** and **z+1** carbon atoms, respectively.

The logarithmic form of Equation 1 corresponds to the logarithmic relation between the boiling point or vapor pressure and the heat of evaporation, the latter increasing linearly in a homologous series [3].

The differences between two retention index values derived from one stationary phase are defined by δI. This value is the retention index difference between two substances on the same column. The temperature dependence of the retention index is defined by the temperature gradient (dI/dT) and is given for a 10 $^{\circ}$C interval, and calculated as (the superscripts specify the temperature so that $T_1 > T_2$) (2):

$$dI/dT = \frac{I^{T_1} - I^{T_2}}{2}$$
(2)

The differences of the retention index values of one substance measured on two stationary liquid phases of different polarity are designated as δ(ΔI). The column temperature is the same in both columns (3):

$$\delta(\Delta I) = I^P - I^A$$
(3)

where: I^P - the index in the polar liquid phase
       I^A - the index in the non-polar liquid phase.

The ΔI values are defined as (e.g. OV-225 and OV-17 columns):

$$\Delta I_{225/17} = I_{OV-225} - I_{OV-17}$$

As part of our study of gas chromatografic retention indices [4], we synthesized a number of known 2-phenyl-2-alkylacetonitriles using phase-transfer reaction of C-alkylation of phenylacetonitrile in the presence of powdered potassium hydroxide as the base and tetrabutylammonium hydrogen sulfate as the catalyst in a solid-liquid system, or the 'classical' reaction of C-alkylation of phenylacetonitrile in the presence of sodium in absolute ether (Scheme 1).
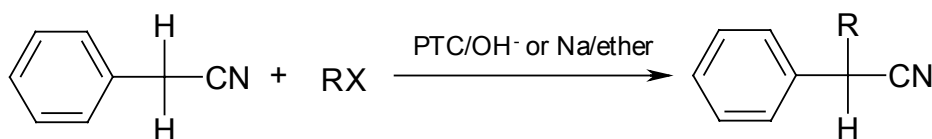
Scheme 1. Synthesis of 2-phenyl-2-alkylacetonitriles
           (R=Me, Et, n-Pr, n-Bu, n-Pe, n-He, n-Hp, n-Oc, n-No, Allyl, PhCH$_2$; X=I, Br)

The products were characterized by their boiling points, indices of refraction, IR and ^{1}H NMR spectra. The gas chromatografic retention indices (I) for the synthesized nitriles were determined on packed columns (OV-17, OV-210 and OV-225) isothermally at 160, 180 and

200 ^{o}C. The isothermal data obtained on these columns were compared and a comparison of the retention index values, ΔI and dI/dT, is presented as linear regression equations.

2. EXPERIMENTAL

2-Phenyl-2-alkylacetonitriles were obtained either using the phase-transfer reaction of C-alkylation of phenylacetonitrile in the presence of powdered potassium hydroxide as the base and tetrabutylammonium hydrogen sulfate as the catalyst in a solid-liquid system or the 'clasical' reaction of C-alkylation of phenylacetonitrile in the presence of sodium in absolute ether.

Procedure for phase-transfer catalyzed C-alkylation (A) [5]. Phenylacetonitrile, alkyl halide, powdered potassium hydroxide and tetrabutylammonium hydrogen sulfate were heated under gentle reflux for 8 hours. After cooling, water was added, the layers were separated and the water layer extracted (diethyl ether). The organic layers were collected and dried over sodium sulfate. The corresponding nitrile was obtained after vacuum distilation.

Procedure for 'classical' C-alkylation (B). 2-Phenyl-2-alkylacetonitriles were obtained by the procedure described by M. Rising [6] where sodium in diethyl ether is used.

The hydrocarbon standards used in this study as well as all other materials were obtained commercially from Fluka.

The IR spectra were recorded on a MB-100 Bomem FTIR spectrophotometer, in the form of KBr pellets or liquid film.

^{1}H NMR spectra were determined in deutareted chloroform (CDCl$_3$) with a Varian EM 390 instrument, using tetramethylsilane as an internal standard.

The GC analyses were performed on a Varian 1400 gas chromatograph equipped with a flame ionization detector. Data handling was provided by a Varian 4720 data system.

The packed columns were laboratory prepared using 2 m stainless steel columns, i.d. 2 mm, and commercially coated supports obtained from Varian:

1. 3 % OV-17 on Chromosorb W H/P 80/100,
2. 3 % OV-210 on Chromosorb W H/P 80/100 and
3. 3 % OV-225 on Chromosorb W H/P 80/100.

All the columns were operated under isothermal (160, 180 and 200 ^{o}C) conditions. The carrier gas was nitrogen, carrier gas flow 20 ml/min, injector temperature 250 ^{o}C, detector temperature 300 ^{o}C, attenuation 1 and range 10^{-10} amps/mV.

3. RESULTS AND DISCUSSION

In the first part of the experimental work a series of 2-phenyl-2-alkylacetonitriles were synthesized as presented in Scheme 1. The 'classical' synthesis of 2-phenyl-2-alkylacetonitriles was usually performed in such a manner that a strong basic compound such as sodium metal, sodium amide or sodium hydroxide and the corresponding alkyl halide were employed [6-10].

Nowadays 2-phenyl-2-alkylacetonitriles are obtained using the phase-transfer catalyzed reaction of C-alkylation. The reaction usually proceeds in the presence of

concentrated sodium or potassium hydroxide aqueous solution and phase-transfer catalysts [11-15].

We synthesized a number of 2-phenyl-2-alkylacetonitriles either using the phase-transfer reaction of C-alkylation of phenylacetonitrile in the presence of powdered potassium hydroxide as the base and tetrabutylammonium hydrogen sulfate as the catalyst in a solid-liquid system (Table 1) or the 'classical' reaction of C-alkylation of phenylacetonitrile in the presence of sodium in anhydrous ether (Table 2). All of the synthesized 2-phenyl-2-alkylacetonitriles are known in the literature [5-15]. As can be seen from the obtained results, better yields were obtained when phase-transfer reaction was used (e.g. when R = allyl). All the products were characterized by their IR and ^{1}H NMR data as shown in Table 3.

Table 1. Synthesis of 2-phenyl-2-alkylacetonitriles. Procedure A.

R	$PhCH_2CN$ (mol)	KOH (mol)	TBAS (mmol)	RX (mol)	b.p. (^oC/mbar)	n_D^{20}	Yield (%)
Me	0.043	0.043	0.43	MeI 0.043	109-10/2.5	1.5115	36
n-Pr	0.085	0.085	0.85	n-PrBr 0.085	120-1/2.5	1.5039	14
n-Pe	0.085	0.085	0.85	n-PeBr 0.085	136-8/2.5	1.4996	18
n-He	0.085	0.085	0.85	n-HeBr 0.085	145-6/2.5	1.4958	42
n-Hp	0.085	0.085	0.85	n-HpBr 0.085	158-60/2.5	1.4935	21
n-Oc	0.085	0.085	0.85	n-OcBr 0.085	161-3/2.5	1.4907	38
n-No	0.043	0.043	0.43	n-NoBr 0.043	166-9/2.5	1.4871	38
Allyl	0.2	0.2	2	AllylBr 0.2	133-5/3	1.5190	41

TBAS=Bu_4NHSO_4

Table 2. Synthesis of 2-phenyl-2-alkylacetonitriles. Procedure B.

R	$PhCH_2CN$ (mol)	Na (mol)	RX (mol)	b.p. or m.p. (^oC/mbar or ^oC)	n_D^{20}	Yield (%)
Et	0.2	0.2	EtBr 0.2	111-3/3.5	1.5061	21
n-Bu	0.2	0.2	n-BuI 0.2	153-6/7	1.5150	32
Allyl	0.2	0.2	AllylBr 0.2	131-5/3	1.5191	19
$PhCH_2$	0.2	0.2	$PhCH_2Br$ 0.2	163-4/2 55-8		17*

*recristallized from ethanol

Table 3. IR and ^{1}H NMR data for the synthesized nitriles.

R	IR (cm^{-1})	^{1}H NMR (ppm)
Me	3035, 3005, 2960, 2910, 2220, 1470, 1365	1.65 (3H, d, CH$_3$), 3.75 (1H, q, CH), 7.4 (5H, s, ArH)
Et	3020, 3005, 2940, 2850, 2905, 2235, 1470, 1390, 1375	0.90 (3H, t, CH$_3$), 1.90 (2H, m, CH$_2$), 3.70 (1H, t, CH), 7.30 (5H, s, ArH)
n-Pr	3043, 3010, 2920, 2850, 2215, 1445, 695	0.95 (3H, t, CH$_3$), 1.50 (2H, m, CH$_2$-CH$_3$), 1.90 (2H, m, CH$_2$-CH), 3.77 (1H, t, CH), 7.33 (5H, s, ArH)
n-Bu	3040, 3010, 2900, 2860, 2225, 1370, 720	0.95 (3H, t, CH$_3$), 1.43 (4H, m, (CH$_2$)$_2$), 1.90 (2H, m, CH$_2$-CH), 3.80 (1H, t, CH), 7.34 (5H, s, ArH)
n-Pe	3045, 3025, 2910, 2850, 2240, 1450, 700	0.85 (3H, t, CH$_3$), 1.35 (6H, s, (CH$_2$)$_3$-CH$_3$), 1.85 (2H, m, CH$_2$-CH), 3.75 (1H, t, CH), 7.4 (5H, s, ArH)
n-He	3040, 2900, 2840, 2230, 1450, 1370, 695	0.85 (3H, t, CH$_3$), 1.3 (8H, s, (CH$_2$)$_4$-CH$_3$), 1.85 (2H, m, CH$_2$-CH), 3.75 (1H, t, CH), 7.4 (5H, s, ArH)
n-Hp	3045, 3010, 2900, 2835, 2220, 1450, 1375, 695	0.85 (3H, t, CH$_3$), 1.35 (10H, s, (CH$_2$)$_5$-CH$_3$), 1.85 (2H, m, CH$_2$-CH), 3.75 (1H, t, CH), 7.4 (5H, s, ArH)
n-Oc	3045, 3020, 2900, 2840, 2220, 1450, 1360, 695	0.85 (3H, t, CH$_3$), 1.25 (12H, s, (CH$_2$)$_6$-CH$_3$), 1.85 (2H, m, CH$_2$-CH), 3.75 (1H, t, CH), 7.35 (5H, s, ArH)
n-No	3040, 3005, 2900, 2835, 2235, 14450, 1370, 695	0.85 (3H, t, CH$_3$), 1.25 (14H, s, (CH$_2$)$_7$-CH$_3$), 1.80 (2H, m, CH$_2$-CH), 3.75 (1H, t, CH), 7.33 (5H, s, ArH)
Allyl	3070, 3045, 2990, 2920, 2225, 1460	2.58 (2H, t, CH$_2$), 3.80 (1H, t, Ph-CH), 5.13 (2H, d, CH=CH$_2$), 5.6 (1H, m, CH=CH$_2$), 7.30 (5H, s, ArH)
PhCH$_2$	3010, 2990, 2880, 2210, 1475	3.06 (2H, m, CH$_2$), 3.96 (1H, t, CH), 7.20 (10H, s, ArH)

The obtained 2-phenyl-2-alkylacetonitriles were used to obtain the retention index (I_{column}^T) values as listed in Table 4. The ΔI_{column}^T values are given in Table 5 and the temperature dependence of the index values $(dI/dT)_{column}^T$ are shown in Table 6.

Table 4. Retention index values for 2-phenyl-2-alkylacetonitriles on three packed columns measured at three different temperatures.

R	Column								
	OV-17			OV-210			OV-225		
	160 ^{o}C	180 ^{o}C	200 ^{o}C	160 ^{o}C	180 ^{o}C	200 ^{o}C	160 ^{o}C	180 ^{o}C	200 ^{o}C
H	1430.72	1449.10	1457.67	1574.95	1622.11	1682.31	1624.95	1644.09	1658.60
Me	1443.56	1459.86	1467.92	1613.61	1664.78	1715.86	1663.02	1678.35	1693.43
Et	1477.89	1491.68	1501.91	1647.28	1702.01	1761.67	1685.56	1702.21	1717.34
n-Pr	1567.95	1584.89	1590.23	1742.93	1792.93	1853.68	1771.12	1786.14	1801.19
n-Bu	1665.12	1679.83	1690.79	1813.35	1881.18	1941.19	1846.07	1860.39	1875.03
n-Pe	1787.08	1799.81	1810.34	1951.64	2000.36	2061.85	1977.18	1992.13	2007.45
n-He	1889.08	1904.32	1913.71	2056.73	2109.25	2169.17	2084.43	2101.13	2116.75
n-Hp	1995.93	2012.12	2017.93	2157.99	2209.92	2269.02	2179.54	2194.68	2209.66
n-Oc	2098.23	2113.83	2124.44	2260.29	2312.63	2372.63	2253.77	2270.18	2285.25
n-No	2190.25	2209.58	2218.19	2352.80	2404.80	2465.95	2343.13	2358.89	2375.95
Allyl	1584.05	1598.45	1608.57	1744.82	1794.80	1847.51	1755.99	1778.88	1794.27
PhCH$_2$	2097.03	2116.21	2120.54	2267.03	2316.32	2373.26	2232.27	2252.03	2273.31

Table 5. ΔI values for 2-phenyl-2-alkylacetonitriles on three packed columns
measured at three different temperatures.

R	Temperature (°C)								
	160			180			200		
	$\Delta I_{210/17}$	$\Delta I_{225/17}$	$\Delta I_{225/210}$	$\Delta I_{210/17}$	$\Delta I_{225/17}$	$\Delta I_{225/210}$	$\Delta I_{210/17}$	$\Delta I_{225/17}$	$\Delta I_{225/210}$
H	144.23	194.23	50.00	173.01	194.99	21.98	224.64	200.93	-23.71
Me	170.05	219.46	49.41	204.92	218.49	13.57	247.94	225.51	-22.43
Et	169.39	207.67	38.28	210.33	210.53	0.20	259.76	215.43	-44.33
n-Pr	174.95	203.17	28.19	208.04	201.25	-6.79	263.45	210.96	-52.49
n-Bu	166.23	180.95	14.72	201.35	180.56	-20.79	250.40	184.24	-66.16
n-Pe	166.56	192.10	25.54	200.55	192.32	-8.23	251.59	197.11	-54.40
n-He	167.65	195.35	27.70	204.93	196.81	-8.12	255.46	203.04	-52.42
n-Hp	162.06	183.61	21.55	197.80	182.56	-15.24	251.09	191.73	-59.36
n-Oc	162.06	155.54	-6.52	198.80	156.35	-42.45	248.19	160.81	-87.38
n-No	162.55	152.88	-9.67	195.22	150.31	-44.91	247.78	156.76	-91.02
Allyl	160.77	171.94	11.17	196.35	180.43	-15.92	238.94	185.70	-53.24
PhCH₂	170.00	135.24	-34.76	200.11	135.82	-64.29	252.72	152.77	-99.95

Table 6. Retention index values for 2-phenyl-2-alkylacetonitriles on three packed
columns measured at 170 and 190 °C (retention index temperature increments).

R	dI/dT					
	OV-17		OV-210		OV-225	
	170 °C	190 °C	170 °C	190 °C	170 °C	190 °C
H	9.19	4.28	23.58	30.10	9.57	7.25
Me	8.15	4.03	25.58	25.54	7.66	7.54
Et	6.89	5.11	27.36	29.83	8.32	7.56
n-Pr	8.47	2.67	25.0	30.37	7.52	7.52
n-Bu	7.35	5.48	24.91	30.00	7.16	7.32
n-Pe	7.36	5.26	24.36	30.74	7.47	7.66
n-He	7.62	4.69	26.26	29.96	8.35	7.82
n-Hp	8.09	2.90	25.95	29.55	7.57	7.49
n-Oc	7.80	5.30	26.17	30.00	8.20	7.53
n-No	9.66	4.30	26.0	30.58	8.36	7.53
Allyl	7.20	5.06	24.99	26.35	11.44	7.69
PhCH₂	9.59	2.16	24.64	28.47	9.88	10.64

The obtained data can be presented by the following linear regression equations:

$$I^{160}_{OV\text{-}17} = 160.03 + 1.0027 \cdot I^{160}_{OV\text{-}210} \qquad r = 0.9997 \qquad (4)$$

$$I^{160}_{OV\text{-}17} = 307.63 + 0.9294 \cdot I^{160}_{OV\text{-}225} \qquad r = 0.9987 \qquad (5)$$

$$I^{180}_{OV\text{-}17} = 192.40 + 1.0041 \cdot I^{180}_{OV\text{-}210} \qquad r = 0.9995 \qquad (6)$$

$$I^{180}_{OV\text{-}17} = 314.41 + 0.9266 \cdot I^{180}_{OV\text{-}225} \qquad r = 0.9989 \qquad (7)$$

$$I^{200}_{OV\text{-}17} = 233.31 + 1.0091 \cdot I^{200}_{OV\text{-}210} \qquad r = 0.9995 \qquad (8)$$

$$I^{200}_{OV\text{-}17} = 317.13 + 0.9293 \cdot I^{200}_{OV\text{-}225} \qquad r = 0.9989 \qquad (9)$$

The retention index values of the investigated nitriles increase with increasing temperature on all three columns (Tables 4 and 5), as well as with the polarity of the liquid phase, but the change of the retention index value with increasing temperature is not constant:

$$I^{180}_{OV-17} - I^{160}_{OV-17} = 13.79 \div 19.36 \tag{10}$$

$$I^{200}_{OV-17} - I^{180}_{OV-17} = 4.33 \div 10.96 \tag{11}$$

$$I^{180}_{OV-210} - I^{160}_{OV-210} = 47.46 \div 56.26 \tag{12}$$

$$I^{200}_{OV-210} - I^{180}_{OV-210} = 51.08 \div 61.49 \tag{13}$$

$$I^{180}_{OV-225} - I^{160}_{OV-225} = 14.35 \div 22.89 \tag{14}$$

$$I^{200}_{OV-225} - I^{180}_{OV-225} = 14.64 \div 21.25 \tag{15}$$

The negative values of ΔI for the OV-225 and OV-210 columns at 200 ^{o}C are caused by the high polarity of the OV-225 and OV-210 columns. The best resolution of the investigated nitriles was obtained on an OV-17 column. Due to the wide range of the boiling points of the investigated compounds, the higher homologues are in the liquid state in the columns at 160 and 180 ^{o}C, instead of in the gas phase producing different values of ΔI for the OV-225 and OV-210 columns in comparison to the OV-17 column.

On the basis of equations 4-9 it is possible to calculate the retention index ($I^{iso, column}$) values from the literature data for the OV-17, OV-210 and OV-225 columns, with an accuracy better than 2.1 i.u.

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Abbreviations.

Me	methyl
Et	ethyl
n-Pr	n-propyl
n-Bu	n-butyl
n-Pe	n-pentyl
n-He	n-hexyl
n-Hp	n-heptyl
n-Oc	n-octyl
n-No	n-nonyl
TBAS	tetrabutylammonium hydrogen sulfate

GASNO HROMATOGRAFSKI RETENCIONI INDEKSI
2-FENIL-2-ALKILACETONITRILA
NA PAKOVANIM KOLONAMA

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2-Fenil-2-acetonitrili su sintetizovani koristeći reakciju C-alkilovanja fenilacetonitrila i okarakterisani temperaturama ključanja, indeksima refrakcije, infracrvenim i NMR spektrima u cilju određivanja gasno hromatografskih retencionih indeksa. Gasno hromatografski retencioni indeksi su određeni na pakovanim kolonama (OV-17, OV-210 i OV-225) izotermno na 160, 180 i 200 ^{o}C, izračunate su ΔI i dI/dT vrednosti.

Ključne reči: *2-fenil-2-alkilacetonitrili, međufazna kataliza, gasno hromatografski retencioni indeksi*