Inverse gas chromatography in the examination of organic compounds
Polarity and solubility parameters of isoquinoline derivatives

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Abstract

Inverse gas chromatography (IGC) has been used in the characterization of isoquinoline derivatives. Physicochemical properties of these organic compounds were represented by the use of polarity (retention index, polarity index, coefficient ρ), the solubility parameter (δ_2), its increments corresponding to dispersive (δ_d), polar (δ_p) and hydrogen bonding properties (δ_h), as well as the corrected solubility parameter (δ_T). Relations between the structure of the examined isoquinolines and their properties are presented and discussed. The influence of the stationary liquid phase content and the temperature of the IGC experiment on the determined parameters is presented. © 1998 Elsevier Science B.V.

Keywords: Inverse gas chromatography; Solubility parameter; Polarity parameters; Isoquinolines

1. Introduction

The isoquinoline skeleton is found abundantly in the plant world and is widely incorporated into medicinally important compounds [1]. The frequent occurrence of the isoquinoline nucleus in alkaloids and in some physiologically active compounds has led to considerable interest in the synthesis of isoquinolines. The synthesis methods of isoquinoline systems were summarized and reviewed elsewhere [1,2].

Isoquinoline and quinoline derivatives were reported as stationary phases, which have been used for separating, e.g., hexanes, C_1–C_7 alkanes and cyclopentanes, for separation of the mixture of benzene and thiophene, benzene–cyclohexane, heptane–methylcyclohexane, different aromatic compounds and isomeric dialkyl sulfide [2].

Quinoline and isoquinoline derivatives were also separated in the chromatographic column with the use of e.g. Apiezon, SE-30, Reoplex 400 and poly-(ethylene glycol) PEG 400 as liquid stationary phases [2]. It was evidenced that there is not much difference in the influence of the single factors on the chromatographic behavior of isoquinoline derivatives. The values of relative retention (r) for the compounds uncomplicated by steric hindrance depended mainly on basicity constants and to a small degree on dipole moments. A methyl group next to a

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heteroatom causes a significant reduction in *r*. An interaction between a heterocycle and a stationary phase, was effected via the heteroatom, the screening of which reduced retention parameters. Hence the donor–acceptor type of interaction, involving participation of the unshared electron pair of a nitrogen atom predominated in this system. It was presented that the dipole–dipole interaction as well as dispersion factors were likely to be of subordinate importance. The contribution of the π systems of the bases to the interaction between a polar phase and solution was found to be only second-order. Compounds without steric hindrance to the interaction between a lone nitrogen electron pair and a stationary liquid were eluted in order of boiling point.

Isoquinoline derivatives are not yet characterized by means of inverse gas chromatography (IGC). This chromatographic technique was earlier used in the determination of the properties of surfactants and extractants [3–16]. Most often the properties of the examined material placed in the gas chromatographic column are discussed with respect to their polarity. The term "polarity" is very general and according to Poole and Poole [17] refers to the capacity of a solvent for various intermolecular interactions. Commercial stationary phases are usually characterized by McReynolds' constants [18] calculated in the standard way using the retention indices of benzene, butan-2-ol, pentan-2-one, pyridine and 1-nitropropane. The sum of the values for these test solutes is represented by

$$\sum_{i=1}^{5} \Delta I_i.$$

The polarity index (*PI*) and coefficient ρ were also used as, so-called, empirical polarity parameters. The advantages and restrictions in using these parameters and their use in quantitative structure–activity relationships were discussed [3,4].

The other useful parameter in characterization of organic species is the solubility parameter, δ_2, introduced by Hildebrand and defined as the square root of the cohesive energy ratio. The procedure of DiPaola-Baranyi and Guillet is most often used for estimation of δ_2 for non-volatile species [5–8,14–16,19–26]:

$$\frac{\delta_1^2}{RT} - \frac{\chi^\infty}{V_1^0} = \frac{2\delta_2}{RT}\,\delta_1\left(\frac{\delta_2^2}{RT} + \frac{\chi_s^\infty}{V_1^0}\right) \qquad (1)$$

where δ_1, and δ_2 are solubility parameters of the testing solute and the stationary phase, respectively; χ^∞ and, χ_s^∞ are the Flory–Huggins interaction parameter and its entropic component, respectively; V_1^0 is molar volume of testing solute, *R* is the gas constant and *T* is the temperature.

Voelkel and Janas [5,6] proposed the modification which allowed separation of increments of the solubility parameter corresponding to different solute–solvent intermolecular interactions. Their proposal was based on the earlier Price [26] report indicating the downward curvature of experimental points in Eq. (1). Increments of the solubility parameter corresponding to dispersive (δ_d), polar (δ_p) and hydrogen bonding (δ_h) interactions, were determined from Eq. (1) used separately for three groups of solutes, (i) *n*-alkanes, (ii) polar non-hydrogen bonding test solutes (aromatic hydrocarbons, ketones and nitropropane), (iii) alcohols and pyridine.

The same curvature, as reported by Price, was observed also for the series of oxyethylates [4,5,8], chemically bonded stationary phases [15] and extractants [16]. It means that the values of δ_2 for compounds having relatively low molecular mass estimated with the use of Eq. (1) were overestimated.

The aim of this work was to: (i) examine the series of isoquinoline derivatives by means of IGC; (ii) calculate polarity and solubility parameters; (iii) discuss the influence of the structure of isoquinolines on estimated physicochemical parameters; (iv) discuss the temperature dependence of the solubility parameter and its increments; (v) discuss the influence of the content of liquid stationary phase on examined polarity and solubility parameters.

2. Experimental

The following isoquinoline derivatives were used as stationary phase in IGC experiments.
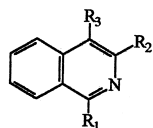
Isoquinoline (IQ)	R₁	R₂	R₃
1	phenyl	H	H
2	4-tolyl	H	H
3	4-chlorophenyl	H	H
4	phenyl	H	methyl
5	methyl	H	phenyl
6	methyl	phenyl	H
7	4-chlorophenyl	methyl	H

(Note: column headers R₁, R₂, R₃ render as R_1, R_2, R_3; structure shows positions R_1, R_2, R_3 on isoquinoline ring.)

Isoquinoline compounds (IQ-1 to IQ-5 and IQ-7) were obtained by cyclization appropriate of N-(2-phenyl-2-hydroxyethyl)amide derivatives using chlorophosphoric acids as dehydrating agents [27–29]. However, 1-methyl-3-phenylisoquinoline (IQ-6) was obtained by heating of N-(1,2-diphenyl-ethenyl)acetamide with chlorophosphoric acids at 150°C.

General procedure: the mixture (2 g of substrate and 20 ml chlorophosphoric acids) was heated at 180°C in an oil bath for 3 h and then cooled. The excess of the dehydrating agent was decomposed very carefully with 100 g of crushed ice. The mixture was boiled for 0.5 h to hydrolyze uncon-verted oxazoline. The post-cooling solution was alkalized with 30% aqueous potassium hydroxide solution and then extracted several times with ether. The combined organic extracts were washed with water dried over Na_2SO_4 and evaporated. The residue was distilled under reduced pressure to obtain the product. The yield and physical properties of the isoquinolines are summarized in Table 1.

2.1. IGC experiments

The conditions for the IGC experiments were as follows: column, 1 m×3 mm I.D.; column temperatures, 100, 110 and 120°C [for 1-(4-chlorophenyl)-3-methylisoquinoline column temperature was held at 130, 140 and 150°C due to its high melting point]; column loadings, 3, 6, 9, 12 and 25% (w/w) of the liquid phase supported on 80–120 mesh Celite (125–177 μm); carrier gas, helium at a flow-rate of 40 ml min⁻¹; flame ionization detector; gas chromato-graph, Chrom 5 (Kovo, Czech Republic) equipped with Peaksimple Chromatography Data System from SRI Instruments (Torrance, CA, USA). The volatile test compounds employed were: *n*-alkanes C_5 to C_{10}; the polar *n*-alkanols: methanol, ethanol and butan-1-ol; pentan-2-one, benzene, 1-nitropropane

Table 1
Yield, physical properties and analytical data of isoquinolines

Isoquinoline (IQ)	Yield (%)	M.p.[a] (°C)	M.p.[a] picrate (°C)	Formula	Analyses, % Found (Calcd.)		
					C	H	N
1	78	95–96	165–166	$C_{15}H_{11}N$ (205.26)	87.77 (87.73)	5.40 (5.41)	6.82 (6.86)
2	89	78–79	188–189	$C_{16}H_{13}N$ (219.29)	87.64 (87.61)	5.98 (5.97)	6.39 (6.42)
3	41	87–88	–	$C_{15}H_{10}NCl$ (239.71)	75.16 (75.22)	4.20 (4.23)	5.84 (5.77)
4	81[b]	75–76	198–199	$C_{16}H_{13}N$ (219.29)	87.64 (87.59)	5.98 (6.06)	6.39 (6.35)
5	65	79–80	222–223	$C_{16}H_{13}N$ (219.29)	87.64 (87.66)	5.98 (5.94)	6.39 (6.41)
6	71[b]	48–49	202–203	$C_{16}H_{23}N$ (219.29)	87.64 (87.62)	5.98 (5.94)	6.39 (6.43)
7	57	118–119	190–192	$C_{16}H_{12}NCl$ (253.74)	75.74 (75.71)	4.77 (4.82)	5.52 (5.54)

[a] From ethanol.
[b] at 150°C.

and pyridine. Injection volume: 0.1 μl. Each solute was injected five times and retention times were averaged. The mean retention time was used in further calculations of net retention volume. The densities of the stationary phases were measured at the temperatures of the IGC experiment with a modified picnometric procedure used earlier by Garcia-Dominguez and coworkers [30,31].

2.2. Calculations

The void volume was calculated according to the procedure proposed by Grobler and Balizs [32]. Polarity parameters: PI and coefficient ρ were calculated with the use of the retention of methanol and ethanol as the polar test probes [3,13,14]. Retention indices of the first five McReynolds' test solutes and differences of retention index on the examined phase and the reference squalane ΔI_i, as well as their sum $\Sigma^5_{\{i=1\}} \Delta I_i$ were calculated by the

usual procedure. The solubility parameter was calculated with the use of the procedure given by DiPaola-Baranyi and Guillet [19,20] and Eq. (1). Increments of δ_2 corresponding δ_d, δ_p and δ_h interactions, were calculated as described previously [5,8]

3. Results and discussion

Isoquinoline derivatives were characterized by empirical polarity parameters as the difference of the retention index on the examined phase and the reference squalane for the first five McReynolds' test solutes ΔI_i, their sum $\Sigma^5_{\{i=1\}} \Delta I_i$ (Table 2), PI and coefficient ρ (Table 3), as well as, by δ_2, increments of δ_2 corresponding to δ_d, δ_p and δ_h interactions and the corrected solubility parameter δ_T (Table 4).

Generally, the increase in polarity of the stationary phase is quantified by an increase of PI, coefficient

Table 2
Retention indices of McReynolds' test solutes for examined isoquinolines at 393 K (25%, w/w, content of liquid stationary phase)

Isoquinoline	Retention indices					
	ΔI_{Bz}	ΔI_{Bu}	ΔI_{Pe}	ΔI_{Np}	ΔI_{Py}	$\Sigma^5_{\{i=1\}} \Delta I_i$
1	191.9	347.3	272.7	394.4	330.6	1536.9
2	168.4	319.7	228.6	344.5	283.3	1344.5
3	140.4	306.8	242.0	332.8	290.7	1344.6
4[a]	173.1	330.2	236.0	350.8	292.0	1382.1
5	158.8	329.1	204.6	302.6	262.8	1257.9
6	168.7	232.9	245.0	322.2	279.6	1248.4
7[b]	149.0	254.1	168.7	284.2	252.5	1108.5

[a] 12% (w/w) ofliquid phase.
[b] at 403 K.

Table 3
Polarity Index (PI) and coefficient ρ for examined isoquinolines at 393 K (25%, w/w, content of liquid stationary phase)

Isoquinoline	PI		$\Delta PI_{EtOHH-MeOH}$	ρ		$\Delta \rho_{EtOH-MeOH}$
	MeOH	EtOH		MeOH	EtOH	
1	83.43	95.28	11.85	1.502	1.997	0.495
2	75.07	88.44	13.37	1.248	1.654	0.406
3	70.70	84.60	13.90	1.160	1.508	0.348
4[a]	65.26	78.53	13.27	1.286	1.541	0.255
5	84.22	91.96	7.74	1.471	1.786	0.315
6	52.37	76.43	24.06	0.793	1.072	0.279
7[b]	48.45	68.17	19.72	0.871	1.099	0.228

[a] 12% (w/w) of liquid phase.
[b] at 403 K.

Table 4
Solubility parameters for examined isoquinolines at 393 K (25%, w/w, content of liquid stationary phase) $[10^3 \ (\text{J m}^{-3})^{1/2}]$

Isoquinoline	δ_2	Increments of solubility parameter			δ_T
		δ_d	δ_p	δ_h	
1	16.699	12.533	4.142	5.494	14.298
2	16.306	12.798	3.551	4.690	14.086
3	16.448	12.337	4.040	5.635	14.152
4[a]	17.281	10.710	6.072	9.329	15.447
5	16.885	12.619	3.611	6.152	14.495
6	16.179	12.182	4.795	5.767	14.304
7[b]	15.771	12.368	3.504	5.099	13.829

[a] 12% (w/w) of liquid phase.
[b] at 403 K.

ρ, as well as, by the increase of the solubility parameters δ_2 and δ_T.

Values of the differences of retention index on the examined phase and the reference squalane for isoquinoline derivatives decrease when a methyl group is introduced in the *para*-position into the molecule of 1-phenylisoquinoline (IQ-1⇒IQ-2). Values of this parameter, butan-1-ol (ΔI_{Bu}) and 1-nitropropane (ΔI_{Np}) decrease when in IQ-2 the methyl group is replaced by a chlorine atom. However, this change of the molecule structure causes the slight increase in the difference of retention indices for benzene (ΔI_{Bz}), pentan-2-one (ΔI_{Pe}) and pyridine (ΔI_{Py}). Generally, the introduction of the substituent into the IQ-1 molecule decreases the polarity of the isoquinoline derivative (IQ-1 > IQ-2 > IQ-3). The same order of compounds' polarity is observed when *PI* and coefficient ρ are taken into account (Table 3). ΔI_i values decrease when a methyl group is introduced into the molecule of 1-phenylisoquinoline in the position C-4 (IQ-1⇒IQ-4). These values found for IQ-4 are higher (McReynolds' test solutes) than for isoquinoline derivatives having methyl and chlorine groups substituted to the 1-phenyl ring (IQ-2 and IQ-3). ΔI_i values for IQ-4 are also higher than those found for 4-phenyl-1-methylisoquinoline (IQ-5). It indicates that intermolecular interactions are stronger when a phenyl group is present in the position C-1 instead of an alkyl group. The change of the position of phenyl from C-4 (IQ-5) to C-3 (IQ-6) substituent causes the increase of ΔI_i values with the only exception being the parameter found for butan-1-ol.

However, liquid stationary phases in gas chromatography are most often characterized by the sum of the first five McReynolds' test solutes $\Sigma_{\{i=1\}}^5 \Delta I_i$. The highest value is observed for 1-phenylisoquinoline (IQ-1). Introduction of any substituent causes the decrease of the $\Sigma_{\{i=1\}}^5 \Delta I_i$ parameter. The examined isoquinolines may be arranged with decreasing value of $\Sigma_{\{i=1\}}^5 \Delta I_i$ as follows:

$$IQ-1 > IQ-4 > IQ-2 > IQ-3 > IQ-5 \\ > IQ-6 > IQ-7$$

1-Phenylisoquinoline derivatives exhibit lower polarity (as the liquid stationary phase) than 1-methylisoquinolines.

Surface active agents and extractants were often characterized with the use of empirical parameters: *PI* and coefficient ρ. The values of these parameters determined with the use of two polar test solutes (methanol and ethanol) are presented in Table 3. Values obtained with the use of ethanol were always higher but the difference depended on the structure of the examined compound and no general relationship was observed. A similar situation was found for coefficient ρ and also in this case differences between values of this parameter found for ethanol and methanol, $\Delta \rho_{EtOH-MeOH}$, depended on the examined isoquinoline. The most significant difference in the arrangement with the use of *PI* or coefficient ρ is the "higher" position of IQ-5. However, one should take into account that the previous order had been arranged with the use of the parameter representing the sum of different intermolecular interactions. The selection by *PI* or coefficient ρ is based on interactions of the stationary phase and a single test solute—alcohol. Both parameters are "poorer" in information than $\Sigma_{\{i=1\}}^5 \Delta I_i$. However, the same results are obtained from the arrangement according to ΔI_{Bu} and PI_{EtOH}.

Introduction of a methyl group or chlorine atom into the molecule of 1-phenylisoquinoline causes the decrease of value of δ_2 (Table 4). A significant increase of δ_2 is observed when the methyl group is placed in position C-4 of isoquinoline (IQ-4). The δ_2 value for 1-methyl-isoquinoline derivatives depends on the position of the substituent. The highest value of δ_2 is found for the 4-phenyl-derivative (IQ-5), lower for the 3-phenyl-derivative (IQ-6) and lowest

for 1-(4-chlorophenyl)-3-methylisoquinoline (IQ-7). Values of the increments of δ_2 corresponding to dispersive, polar and hydrogen bonding interactions also depend on the structure of the isoquinoline system. Introduction of the methyl substituent into the phenyl ring in the molecule of IQ-1 leads to the increase of δ_d, while the presence of a chlorine atom in the same position causes the decrease of this parameter. Substitution of a methyl group in position C-4 of IQ-1 causes the significant decrease of δ_d from 12.533 to 10.710 [10^3 (J m^{-3})$^{1/2}$]. 4-Phenyl-1-methylisoquinoline (IQ-5) exhibits higher values of δ_d than 3-phenyl-1-methylisoquinoline (IQ-6). No significant effect is observed when a methyl group is substituted in position C-3 of the IQ-3 molecule. δ_d Values for IQ-3 and IQ-7 are almost the same even if the higher temperature of the IGC experiment for IQ-7 is taken into account. Introduction of a chlorine atom into the IQ-1 molecule (IQ-1⇒IQ-3) only slightly decreases the value of δ_p. However, the substitution of the hydrogen atom by a methyl group in the phenyl ring (IQ-1⇒IQ-2) leads to a significant decrease of this parameter. The value of δ_p depends on the position of the phenyl substituent in 1-methylisoquinolines. 3-Phenylisoquinoline (IQ-6) exhibits a higher value of δ_p in comparison to the 4-phenyl derivative (IQ-5).

One should check the very high values of increments of δ_h, found for 1-phenyl-4-methylisoquinoline (IQ-4). This value is much higher than those found for IQ-1. 1-(4-Chlorophenyl)-isoquinoline exhibits a slightly higher value of δ_h than non-substituted IQ-1, Replacement of the hydrogen or chlorine atom by a methyl group in these compounds leads to the decrease of δ_h. Values of this increment of solubility parameter for 1-methylisoquinoline derivatives are higher than those for 1-phenylisoquinolines. 4-Phenyl-1-methylisoquinoline (IQ-5) is characterized by a higher value of δ_h than the 3-phenyl derivative (IQ-6).

Values of the corrected solubility parameter δ_T are lower than those of δ_2 by approx. 2 [10^3 (J m^{-3})$^{1/2}$] but the arrangement according to the decreasing solubility parameter is similar in both cases:

$$IQ - 4 > IQ - 5 > IQ - 1 > IQ - 3 > IQ - 2$$
$$> IQ - 6 > IQ - 7 \text{ for } \delta_2$$

$$IQ - 4 > IQ - 5 > IQ - 6 \approx IQ - 1 > IQ - 3$$
$$> IQ - 2 > IQ - 7 \text{ for } \delta_T.$$

The correction of the solubility parameter causes the slight change in the above order. One should note that this arrangement is different from that based on $\sum_{\{i=1\}}^{5} \Delta I_i$ values.

All solubility parameters are estimated through the determination of the Flory–Huggins interaction parameter where besides retention data other physico-chemical properties (molar volume, density, second virial coefficient) of the stationary phase and the test solutes are taken into account. In the authors' opinion, this may be the main reason for the observed differences in isoquinolines' arrangement according to their polarity ($\sum_{\{i=1\}}^{5} \Delta I_i$) or solubility parameters. Very often the increase of the compounds' polarity measured by retention indices, PIs and/or thermodynamical polarity parameters, was accompanied by the increase of the solubility parameter, its increments and corrected solubility parameter [5,6,8,16]. However, the results presented above seem to indicate that these physicochemical parameters are not fully exchangeable. Therefore, it should be fruitful to use the solvation parameters model of Abraham [33,34].

Values of the solubility parameters were determined under isothermal conditions at three different temperatures and for changing contents of liquid stationary phase in column filling. Both parameters of the IGC experiment significantly affect the chromatographic process and estimated physicochemical characteristics. The influence of the content of stationary phase on the values of the solubility parameters is presented in Fig. 1. The most significant changes are observed for lower contents of liquid phase. The magnitude of the changes and their direction depend on the given parameter and the examined isoquinoline. However, in each case one may observe that this influence decreases for higher liquid phase contents. The influence of the liquid phase content on the solubility parameters is probably the result of the mixed retention mechanism during the IGC process. The partition coefficient – the parameter free from disturbing influences due to interfacial adsorption – may be calculated from basic chromatographic data. Unfortunately, it would be
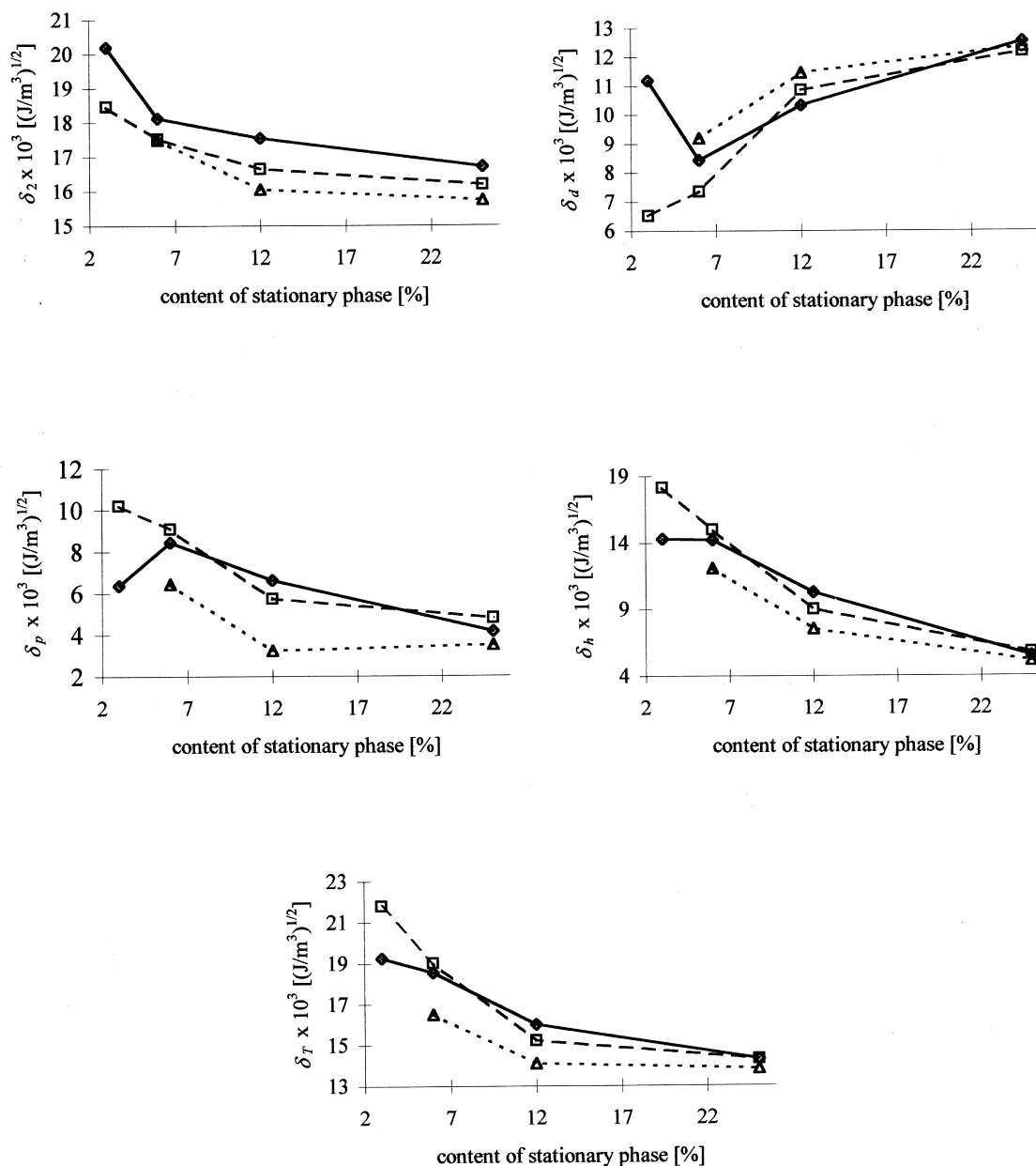
Fig. 1. Relationships between values of the solubility parameters and content of the liquid stationary phase ($\diamond$, IQ-1; $\square$, IQ-6; $\triangle$, IQ-7).

impossible to calculate solubility parameters from the partition coefficient K_L values and redefinition of basic equations (e.g. Eq. (1)) would be required. However, the values of the partition coefficient estimated for the series of test solutes may be used in characterization of the stationary phases through Abraham's solvation parameters [33,34]. This problem will be discussed in the subsequent paper.

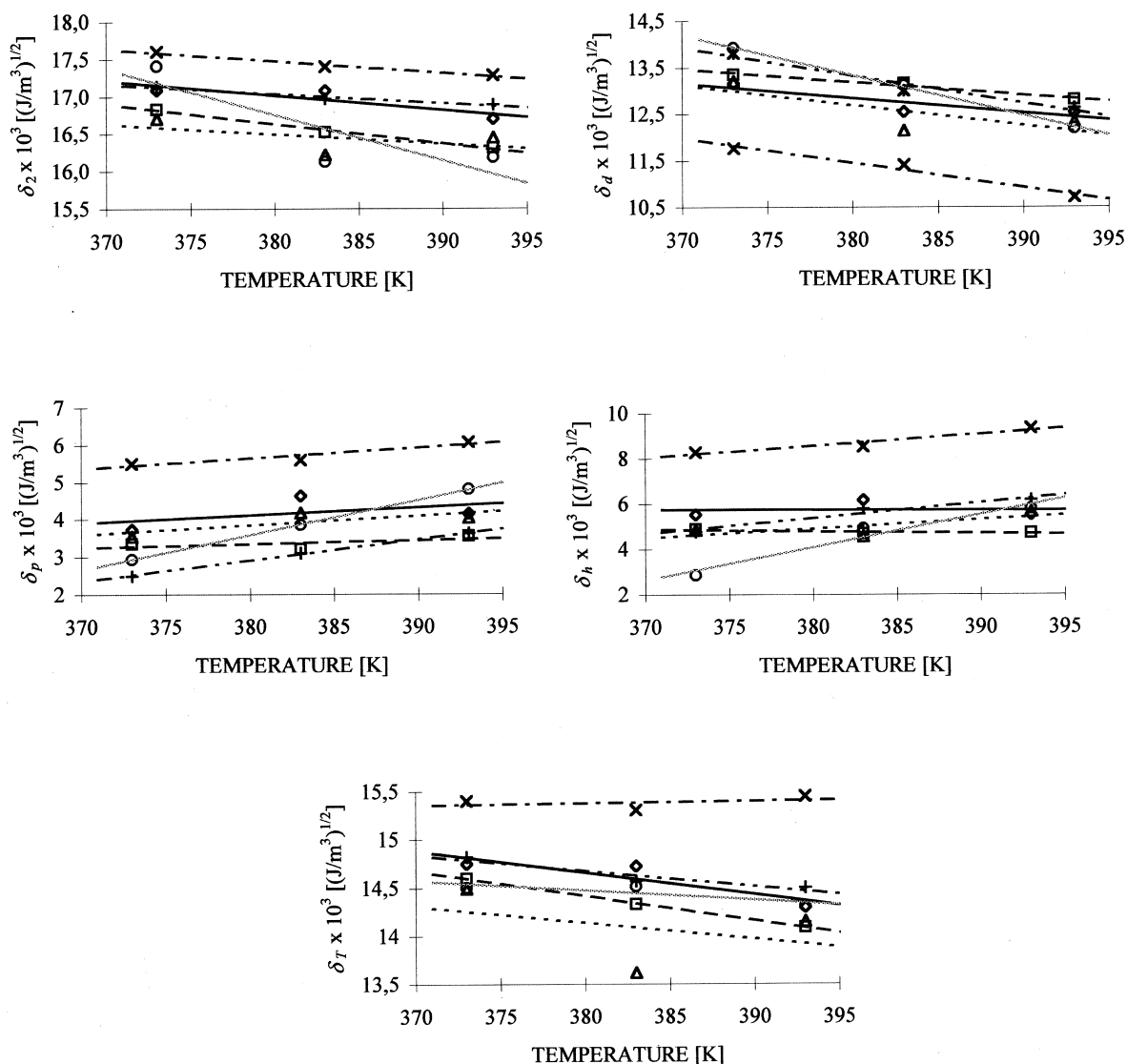The influence of the temperature of the IGC experiment is presented in Fig. 2. All solubility

Fig. 2. Relationships between values of the solubility parameters and temperature of the IGC experiment ($\diamondsuit$, IQ-1; $\square$, IQ-2; $\triangle$, IQ-3; $\times$, IQ-4; $+$, IQ-5; $\bigcirc$, IQ-6).

parameters change with the increasing temperature and the observed relationships are approximately linear. The slopes and directions of changes in parameter–temperature relationships depend on the type of solubility parameter and isoquinoline. The solubility parameter δ_2, dispersive increment δ_d and the corrected solubility parameter δ_T decrease with

the increasing temperature. However, for IQ-5 δ_T slightly increases at the higher temperature of the chromatographic experiment (from 15.404 at 373 K to 15.447 units at 393 K). The polar and hydrogen bonding increments of the solubility parameter mainly increase with the increase in temperature although the decrease of both parameters is found for IQ-2.

4. Conclusions

IGC was used in physicochemical characterization of the group of isoquinoline derivatives. IGC parameters are sensitive enough to reflect the changes in the structure of the isoquinoline system. The arrangements of examined isoquinolines according to increasing/decreasing polarity and solubility parameters are, to some extent, different reflecting other physicochemical factors, meaning – the nature of both groups of these parameters. It has been shown that the solubility parameters depend on the temperature of the IGC experiment and the content of liquid stationary phase.

Acknowledgements

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