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Elimination of adsorption effects of polarity parameters determined by inverse gas chromatography

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Abstract

A procedure for the elimination of adsorption contributions to retention parameters is presented and discussed. It is shown that the corrected retention volume may be used in the calculation of polarity parameters from their basic equations. The influence of the elimination procedure on corrected polarity parameters is presented and discussed.

1. Introduction

Gas chromatography is not only the most widespread analytical method used for determination of components of complex volatile mixtures, but may also be applied as a method for the physico-chemical characterization of different systems. Inverse gas chromatography (IGC) in particular has become a reliable, fast and accurate method for the investigation of polymers, synthetic fibers, surfactant mixtures and organic and inorganic fillers. In this technique the examined material, e.g., molten surfactant, is placed in a chromatographic column and its properties are studied by injecting test solutes on to the column. The retention times and/or peak elution profiles of carefully selected standard solutes are used to estimate the interactions between the solute and stationary phase. On the basis of the column contents one may divide

inverse gas chromatography into inverse gas-liquid and inverse gas-solid chromatography.

Surface-active agents of different types have been examined as stationary phases in GLC experiments [1–10] and characterized in terms of their polarity. Poole and Poole [11] defined this as the capacity of a solvent for various intermolecular interactions. They indicated also that particular attention must be paid to the influence of interfacial adsorption, at both the gas-support and gas-liquid interfaces. There are at least three ways in which a test solute might interact with a column packing: (i) partitioning between the gas and liquid phases, (ii) adsorption at the gas-liquid interface and (iii) adsorption at the liquid-solid interface.

The well known Berezkin equation [12,13] represents the relationship between the net retention volume (V_N) and different increments of the retention mechanism:

$$V_N = K_L V_L + K_{GL} S_{GL} + K_{LS} S_{LS} \quad (1)$$

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where K_L is the partition coefficient, V_L is the volume of the liquid phase and K_{GL} and K_{LS} are the adsorption coefficients at the gas–liquid and liquid–solid interfaces, respectively.

Poole and Poole [11] used K_L estimated from Eq. 1 to calculate the polarity and selectivity parameters of stationary phases. However, in doing so, they had to redefine the well known Kováts equation for the retention index. Other polarity parameters used in the characterization of surfactants [1–10] were defined in terms describing only the partitioning process of the analyte between the stationary phase and the carrier gas.

Physico-chemical (also polarity) parameters may only be properly determined if the influence of adsorption processes at the interfaces are eliminated or minimized. Adsorption effects are usually minimized by the application of a non-active support, careful chemical deactivation of the support surface and/or the use of a high liquid loading. However, the methods of adsorption elimination are not completely satisfactory. Poole and Poole [11] showed that even at a high stationary liquid phase loading both adsorption processes are significant contributors to the retention mechanism. Therefore, we decided to evaluate a procedure of data reduction which permits the isolation of the increment of retention volume corresponding only to the partition mechanism, i.e., bulk retention. We therefore eliminated retention volume contributions corresponding to adsorption at both the gas–liquid and liquid–solid interfaces. Hence the corrected retention volume may be used directly to calculate polarity parameters from appropriate equations. The influence of the retention volume corrections on the values of the polarity parameters is also discussed.

2. Experimental

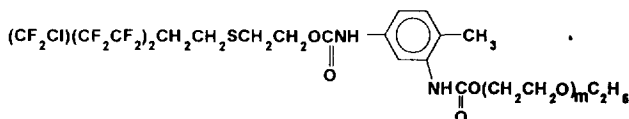
2.1. Materials

Two groups of fluorinated oxyethylene derivatives were used as liquid stationary phases in IGC experiments. The synthesis and surface

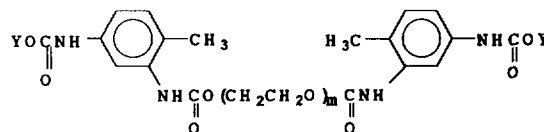
Table 1
Molecular masses of investigated liquid phases in group I

No.	Compound	Molecular mass (g mol)
1	R _{n1} M340	936.5
2	R _{n1} M456	1052.5
3	R _{n1} M666	1262.5
4	R _{n1} M1005	1601.5
5	R _{n1} M1500	2096.5
6	R _{n1} M2250	2846.5
7	R _{n1} M2000	3115.0

properties of these surfactants have been described earlier [10]. The general formulae of these compounds are as follows:



and



where $\text{Y} = \text{CF}_3\text{CF}(\text{CF}_2\text{Cl})(\text{CF}_2\text{CF}_2)\text{CH}_2\text{CH}_2\text{SCH}_2\text{CH}_2$. Their molecular masses are presented in Tables 1 and 2, where R_{n1}M340 and R_{n2}M1500 denote structures having one or two CF₂CF₂ groups in the hydrophobic part of the surfactant molecule and the molecular masses of the oligooxyethylene chain are 340 and 1500, respectively.

Table 2
Molecular masses of investigated liquid phases in group II

No.	Compound	Molecular mass (g mol)
8	R _{n2} M456	1192.36
9	R _{n2} M666	1402.36
10	R _{n2} M1005	1741.36
11	R _{n2} M1500	2236.36
12	R _{n2} M2250	2986.36
13	R _{n2} M2000	3198.72

2.2. IGC experiments

The conditions for the IGC experiments were as follows: column, 1 m × 3 mm I.D.; column temperature, 70, 90, and 110°C; column loading, 5, 10 and 25% (w/w) supported on 80–120-mesh Celite; carrier gas, helium at a flow-rate of 40 ml min⁻¹; detector, flame ionization, gas chromatograph, Chrom 5 (Kovo, Czech Republic). The volatile test compounds employed were non-polar *n*-alkanes with chain lengths from C₅ to C₁₀, the aromatic hydrocarbons benzene, toluene, xylene and ethylbenzene, polar *n*-alkanols from C₁ to C₄, 2-butanone, 2-pentanone, nitropropane and pyridine. Each solute was injected five times and retention times were averaged. These mean retention times were used in further calculations of net retention volumes. All experiments were carried out for three different liquid phase loadings.

2.3. Calculations

Elimination of adsorption effects

Berezkin and co-workers [12–14] have shown that the net retention volume (V_N) is the sum of the partial retention volumes corresponding to the absorption of chromatographed test solutes in the liquid phase [$V_{N(l_i)}$] and its adsorption at all interfaces [$V_{N(s_j)}$], respectively:

$$V_N = \sum_{i=1}^m V_{N(l_i)} + \sum_{j=1}^n V_{N(s_j)} \quad (2)$$

or

$$V_N = \sum_{i=1}^m \overline{K_{L(i)}} V_{L(i)} + \sum_{j=1}^n \overline{K_{S(j)}} S_{S(j)} \quad (3)$$

Of critical importance is the selection of the component of the net retention volume corresponding to the dissolution in the liquid phase, i.e.

$$V_N^s = K_L V_L \quad (4)$$

Therefore, Eq. 1 can be rearranged into the linear relationship

$$\frac{V_N}{V_L} = \Phi \left(\frac{1}{V_L} \right) \quad (5)$$

in which the slope equals the partition coefficient, $K_L \cdot K_L$ values may be then used in Eq. 4 to calculate V_N^s . The selected increment of the net retention volume corresponding only to absorption process or free of adsorption effects may be used to calculate polarity parameters from appropriate equations.

The retention times of a series of *n*-alkanes were used to calculate the retention time corresponding to the void volume of the column [15].

Polarity parameters

The following polarity parameters were used for characterization of examined stationary phases:

(i) The retention index I of selected test solutes was calculated from the Kováts equation, expressing ΔI as the difference in retention indices of a given solute measured on the experimental liquid phase (I_p) and a reference phase, e.g., squalane (I_{np}):

$$\Delta I = I_p - I_{np} \quad (6)$$

Commercial stationary phases are often characterized by the sum of retention index differences of the first five McReynolds' [16] solutes, i.e., benzene, 1-butanol, 2-pentanone, 1-nitropropane and pyridine, $\sum_{i=1}^5 \Delta I_i$.

(ii) The polarity index, PI [17],

$$PI = 100 \log(C - 4.7) + 60 \quad (7)$$

where C is the number of carbon atoms in a hypothetical *n*-alkane having the same retention time as the alcohol test solute (methanol or ethanol).

(iii) The coefficient ρ [18] is the relative retention of a polar test substance (methanol, ethanol) and a standard non-polar substance (*n*-hexane):

$$\rho = \frac{t'_{R(\text{EtOH})}}{t'_{R(n\text{-hexane})}} \quad (8)$$

(iv) Criterion A [19]:

$$A = \frac{t'_{R(n+1)} - t'_{R(n)}}{t'_{R(n)} - t'_{R(n-1)}} \quad (9)$$

where $t'_{R(n+1)}$, $t'_{R(n)}$ and $t'_{R(n-1)}$ denote the adjusted retention times of n -alkanes having $n+1$, n and $n-1$ carbon atoms, respectively.

(v) Partial molar excess Gibbs energy of solution per methylene group [20], defined as

$$\Delta G^E(\text{CH}_2) = \frac{1}{k} \cdot RT \ln \left[\frac{V_{gn} \times p_{i(n)}^0}{V_{g(n+k)} \times p_{i(n+k)}^0} \right] \quad (10)$$

where V_{gn} and $V_{g(n+k)}$ are specific retention volumes and $p_{i(n)}^0$ and $p_{i(n+k)}^0$ are saturated vapour pressures of two homologues having n and $n+k$ methylene groups in their molecules, respectively.

(vi) The partial molal Gibbs free energy of solution for a methylene group, $\Delta G_s^m(\text{CH}_2)$ [21], was calculated from the following equations:

$$\ln V_g = -\frac{\Delta H_s^m}{RT} + \left[\frac{\Delta S_s^m}{R} - \ln \left(\frac{1000}{273.15R} \right) \right] \quad (11)$$

$$\Delta G_s^m = \Delta H_s^m - T\Delta S_s^m \quad (12)$$

and the linear relationship between $\Delta G_s^m(\text{CH}_2)$ for the series of n -alkanes and number of carbon atoms in their molecules.

Calculation steps

The calculations of uncorrected and corrected polarity parameters were carried out according to the following scheme:

(i) five measurements were made for each test solute;

(ii) retention times were averaged and the mean value was used in the following steps;

(iii) the net retention volume was calculated for each solute;

(iv) polarity parameters were calculated from uncorrelated net retention volumes of test solutes;

(v) Eq. 5 was used and K_L values were calculated at significance level $\alpha = 0.05$;

(vi) corrected net retention volumes V_N^s were calculated from Eq. 4;

(vii) corrected polarity parameters were calcu-

lated from V_{Ni}^s values ($i =$ appropriate test solute).

3. Results and discussion

3.1. Adsorption contribution to the retention volume

The magnitude of the influence of the adsorption effect depends on the type of support, the polarity of the liquid phase and the polarity of the injected solute. To minimize this effect, a high loading of liquid phase and chemical deactivation of the support surface were used. However, as has been demonstrated [11], even in such a case the adsorption effect is not completely eliminated. This was the reason for evaluating the procedure for the isolation of the increment of retention volume corresponding only to the mechanism of partitioning of the solute between the gas and liquid phases.

As an example, K_L values for two stationary phases determined for a number of test solutes are presented in Tables 3 and 4. The K_L values were calculated from linear regression at the significance level $\alpha = 0.05$; the correlation coefficient was always higher than 0.96. Poole and co-workers [11,22] have shown that strong linearity described by Eq. 5 exists. We have assumed a universal character of their statement, which is confirmed by our results. Therefore, we limited the number of liquid phase loadings used to three. Graphical representations of Eq. 5 for two selected stationary phases and several test solutes are shown in Fig. 1. The partition coefficient K_L decreases with increase in the temperature of the experiment and increases with an increase in the molecular mass of a given test solute. According to the suggestions of Kersten et al. [22], fluorine-containing compounds may be classified as stationary phases of relatively limited selectivity (such as OV-17, OV-105, OV-225 and QF-1). The proportion of adsorption interactions for these compounds is small [23]. As expected, an increase in the content of liquid phase in the column filling decreases the propor-

Table 3
Values of the partition coefficient K_L for test solutes on $R_{n1}M340$

Test compound	Temperature (K)		
	343	363	383
Pentane	8.8	6.4	4.8
Hexane	17.6	12.4	8.1
Heptane	37.0	22.4	15.3
Octane	78.7	43.6	25.9
Nonane	165.9	83.8	47.0
Decane	348.4	162.0	83.7
Benzene	115.7	62.8	36.5
Methanol	79.7	42.6	22.7
Ethanol	110.1	55.8	29.9
1-Propanol	231.6	107.9	54.7
1-Butanol	491.5	219.5	105.9
2-Butanone	128.6	70.9	41.5
2-Pentanone	235.7	123.5	67.8
Nitropropane	685.2	317.9	159.3
Pyridine	778.7	356.5	179.1
Toluene	236.6	127.1	71.4
Xylene	504.7	187.7	128.5
Ethylbenzene	445.2	222.2	119.4

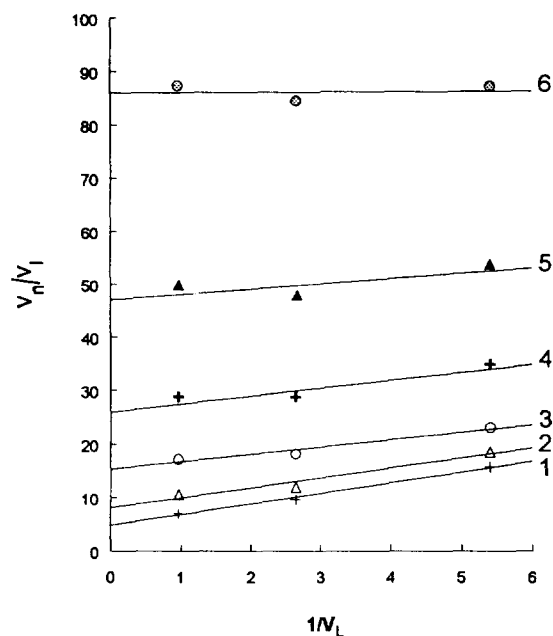


Fig. 1. Relationship $V_n/V_1 = \Phi(1/V_1)$ for $R_{n1}M340$ at 383 K for n -alkanes: 1 = n -pentane; 2 = n -hexane; 3 = n -heptane; 4 = n -octane; 5 = n -nonane; 6 = n -decane.

Table 4
Values of the partition coefficient K_L for test solutes on $R_{n2}M1005$

Test compound	Temperature (K)		
	343	363	383
Pentane	9.4	7.2	5.7
Hexane	17.8	12.2	9.2
Heptane	34.2	21.5	14.9
Octane	71.6	40.9	25.1
Nonane	146.9	76.7	44.2
Decane	300.9	145.0	75.5
Benzene	138.2	74.3	40.7
Methanol	93.5	53.4	30.2
Ethanol	119.5	60.1	33.3
1-Propanol	239.9	115.9	58.1
1-Butanol	516.1	232.0	108.5
2-Butanone	122.2	67.3	39.0
2-Pentanone	215.2	113.2	60.8
Nitropropane	809.0	369.8	178.6
Pyridine	716.6	341.5	169.5
Toluene	283.6	145.6	79.3
Xylene	558.5	271.5	138.0
Ethylbenzene	517.4	251.0	126.8

tion of adsorption effects in the retention parameter (Tables 5 and 6).

The greatest influence of adsorption effects is observed on the retention of non-polar aliphatic hydrocarbons and decreases with increase in their molecular masses. Similarly, the adsorption effect decreases with increase in molecular mass in the n -alkanol series. The adsorption contribution to the retention of polar test solutes having high molecular mass is insignificant or not observed at all. An increase in liquid phase loading significantly decreases the adsorption contribution to retention in the case of n -alkanes (Tables 5 and 6). The symbols A and R used in Tables 5 and 6 denote the adsorption and bulk retention (partition) increment to the retention volume, respectively. Therefore, $100 [A/(A + R)]$ (%) express adsorption contribution to retention. The influence of the adsorption effect is higher for two of the stationary phases, $R_{n1}M2000$ and $R_{n2}M2000$ (Table 6), owing to the screening of the oligooxyethylene chain by two large hydrophobic groups.

Table 5

Influence of liquid phase loading on the adsorption contribution $\{100[A/(A+R)]\%$ for R_{n1} M340 (at 383 K) (A and R are the adsorption and bulk retention increments to the net retention volume, respectively)

Test compounds	Liquid phase loading (%)		
	5	10	25
Pentane	69.5	50.2	31.5
Hexane	56.5	32.4	23.6
Heptane	33.7	15.8	13.4
Octane	25.8	6.0	10.2
Nonane	12.6	10.4	6.3
Decane	4.1	0.0	7.3
Benzene	27.3	10.9	8.7
Methanol	42.8	20.9	15.5
Ethanol	32.1	13.8	10.6
1-Propanol	12.3	2.0	4.6
1-Butanol	0.0	0.0	0.5
2-Butanone	22.5	8.8	5.2
2-Pentanone	5.6	0.0	3.9
Nitropropane	0.0	0.0	0.9
Pyridine	0.0	0.0	0.0
Toluene	5.1	0.0	2.9
Xylene	0.0	0.0	2.9
Ethylbenzene	0.0	0.0	1.9

No clear relationship (e.g. linear) was found between the adsorption contribution to the retention mechanism and the structure of the compounds examined (Fig. 2). However, in group I the highest adsorption contribution was observed for homologues having a short oligooxyethylene chain. This contribution decreases with increase in the number of ethylene oxide (EO) units up to 20–22. Further increase in the oligooxyethylene chain length causes an increase in the adsorption contribution to the retention volume of *n*-octane. The corresponding relationship for oxyethylates in group II is irregular. However, the adsorption contribution to the retention of *n*-octane on liquid phases from this group is often lower than the corresponding value for group I.

3.2. Influence of elimination of the adsorption effect on polarity parameters

The procedure for eliminating the adsorption effect was also applied in the determination

Table 6

Influence of liquid phase loading on the adsorption contribution $\{100[A/(A+R)]\%$ for R_{n2} M2000 (at 383 K) (A and R are the adsorption and bulk retention increments to the net retention volume, respectively)

Test compounds	Liquid phase loading (%)		
	5	10	25
Pentane	91.57	85.06	65.77
Hexane	80.22	68.48	40.82
Heptane	67.87	52.88	26.66
Octane	56.81	40.48	19.06
Nonane	45.47	29.15	13.66
Decane	39.81	24.91	10.95
Benzene	28.69	14.92	8.02
Methanol	34.91	21.01	9.20
Ethanol	28.14	16.03	6.98
1-Propanol	21.18	11.74	4.84
1-Butanol	17.04	9.33	3.70
2-Butanone	31.78	19.01	7.92
2-Pentanone	25.28	14.34	6.01
Nitropropane	12.55	7.53	2.10
Pyridine	14.84	9.08	2.60
Toluene	21.51	12.85	4.38
Xylene	17.23	10.27	3.14
Ethylbenzene	0.0	0.0	0.0

process of polarity parameters, i.e., the corrected net retention volumes calculated from Eqs. 6–13. The influence of the liquid phase

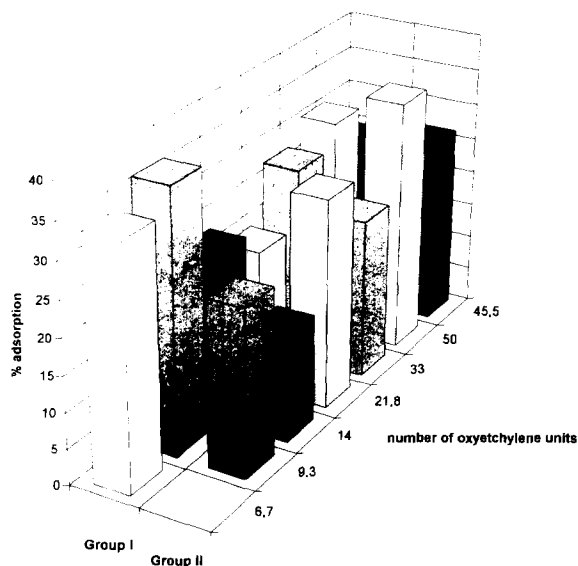


Fig. 2. Adsorption contribution to *n*-octane retention for liquid phases used at 383 K (25% liquid phase loading).

loading on the retention index of several test solutes before and after this correction is presented in Fig. 3a and b, respectively. The application of the correction procedure significantly decreases the variations in retention index with liquid phase loading.

Similarly, this procedure also decreases the variations in the polarity index (Fig. 4). A narrow scatter of results is observed when both methanol and ethanol are used as test solutes. As an example, PI^{EtOH} for $R_{n1}M2000$ at 365 K varies from 119.1 to 127.5 index units before correction, whereas after the application of the elimination procedure it changes from 126.9 to 127.9 index units (Table 7). Similar changes were found for the coefficient ρ . Values of both of these polarity parameters increase after the

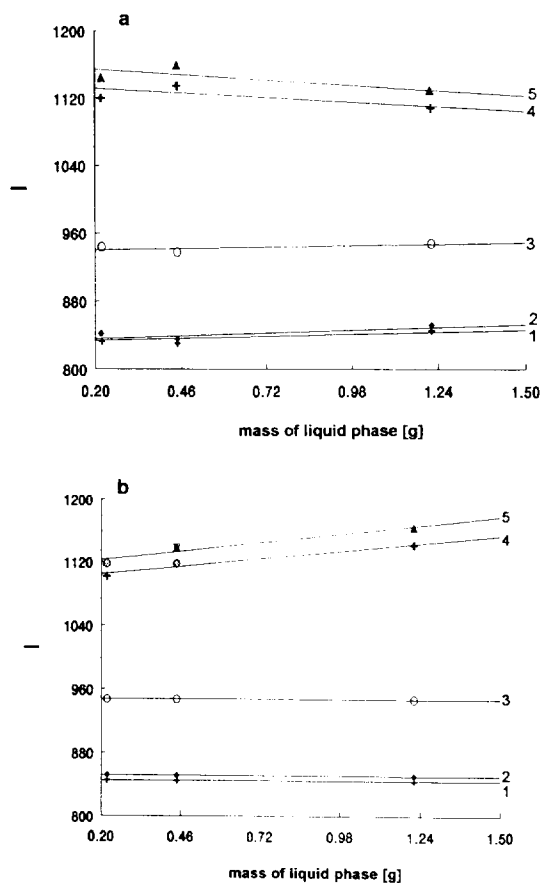


Fig. 3. Dependence of the retention index I on the liquid phase loading, (a) before and (b) after correction due to adsorption contribution for (1) benzene, (2) ethanol, (3) 2-pentanone, (4) nitropropane and pyridine.

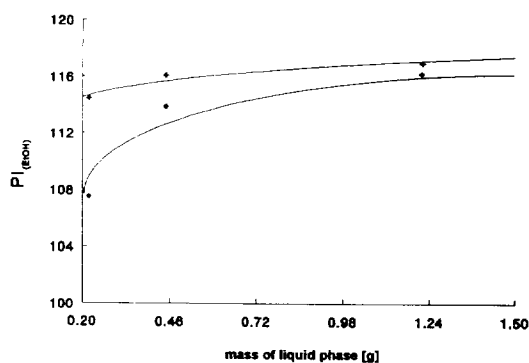


Fig. 4. Relationship between polarity index PI^{EtOH} and mass of the liquid phase ($R_{n1}M340$ at 343 K), (\blacklozenge) before and ($+$) after correction.

elimination of the adsorption contribution from retention volume of test solutes.

The most significant results were found for criterion A . The correction of the retention data for adsorption effects leads to criterion A being independent of the liquid phase loading (Table 8). Criterion A was calculated with the use of n -alkane retention data. Before correction, the net retention volumes were significantly influenced by adsorption effects, which led to different values being found for three liquid phase loadings. The use of corrected net retention volumes V_N^s gave the same criterion A values in each instance.

Thermodynamic parameters of solution were also used to characterize fluorinated oligooxyethylene derivatives. The values for the partial molar excess Gibbs function of solution per methylene group, $\Delta G^E(\text{CH}_2)$, and partial molal Gibbs function of solution, $\Delta G_s^m(\text{CH}_2)$, depend significantly on the liquid phase loading. The contribution of adsorption effects to the numerical values of these parameters is lowered after the use of the correction procedure (Figs. 5 and 6).

4. Conclusions

In the proposed method for the elimination of adsorption effects, a computational method is used to account for the bulk retention (partition) increment in retention data and the adsorption

Table 7

Values of polarity index, PI^{EtOH} , at 343 K for 5, 10 and 25% (w/w) liquid phase loadings before and after correction for adsorption contribution

Compound	Polarity index, PI_{EtOH}					
	Before correction			After correction		
	5%	10%	25%	5%	10%	25%
R_{n1} M340	107.5	113.9	115.9	114.6	116.1	116.9
R_{n1} M456	112.3	118.9	119.8	119.3	120.5	121.1
R_{n1} M666	113.0	117.2	119.3	118.5	119.7	120.2
R_{n1} M1005	117.7	118.3	120.1	120.7	121.0	122.0
R_{n1} M1500	115.6	120.2	122.2	121.8	122.8	122.7
R_{n1} M2250	116.4	121.3	124.1	124.2	124.8	124.9
R_{n1} M2000	124.6	128.1	129.4	130.4	130.1	129.8
R_{n2} M456	107.6	112.7	116.6	115.0	116.7	118.0
R_{n2} M666	107.4	113.8	117.8	114.8	118.3	119.5
R_{n2} M1005	111.9	116.3	119.7	118.3	119.3	120.0
R_{n2} M1500	113.0	118.4	120.9	120.0	121.3	121.8
R_{n2} M2250	116.5	120.4	122.4	121.2	122.3	123.0
R_{n2} M2000	124.0	127.3	128.9	129.9	129.8	129.2

effects at gas–liquid phase and liquid phase–solid support interfaces are eliminated. This leads to the precise determination of physico-chemical parameters. Corrected retention data

may be used to calculate polarity parameters from appropriate equations. The use of a high liquid phase loading was not always effective. In the authors' opinion, the proposed procedure

Table 8

Values of Criterion A at 343 K for 5, 10 and 25% (w/w) liquid phase loadings before and after correction for adsorption contribution

Compound	Criterion A			
	Before correction			After correction ^a
	5%	10%	25%	
R_{n1} M340	2.114	2.105	2.136	2.132
R_{n1} M456	2.017	2.069	2.106	2.127
R_{n1} M666	1.986	2.034	2.124	2.150
R_{n1} M1005	2.417	2.127	2.094	2.053
R_{n1} M1500	2.051	2.027	2.098	2.091
R_{n1} M2250	2.009	2.020	2.069	2.079
R_{n1} M2000	2.018	1.987	2.007	1.995
R_{n2} M456	1.976	2.061	2.117	2.176
R_{n2} M666	2.291	2.108	2.167	2.110
R_{n2} M1005	2.102	2.103	2.065	2.073
R_{n2} M1500	2.111	2.038	2.069	2.049
R_{n2} M2250	1.966	2.043	2.072	2.108
R_{n2} M2000	2.127	2.006	2.006	1.954

^a For all liquid phase loadings.

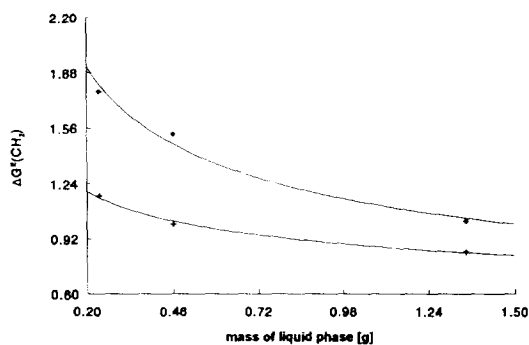


Fig. 5. Relationship between partial molar excess Gibbs function per methylene group, $\Delta G^E(\text{CH}_2)$ (kJ mol^{-1}), for n -alkanes and mass of the liquid phase (R_{n1} M340 at 343 K), (\blacklozenge) before and (+) after correction.

provides the most favourable solution of the problem. The discussed polarity parameters calculated with the use of corrected retention volumes V_N^S do not depend on the liquid phase loading, or the dependence is much lower than before correction.

The applicability of the proposed procedure for the elimination of adsorption effects was demonstrated in the calculation of polarity parameters. However, the corrected retention volume may be used in calculating any physico-chemical parameter defined in terms of the partition retention mechanism.

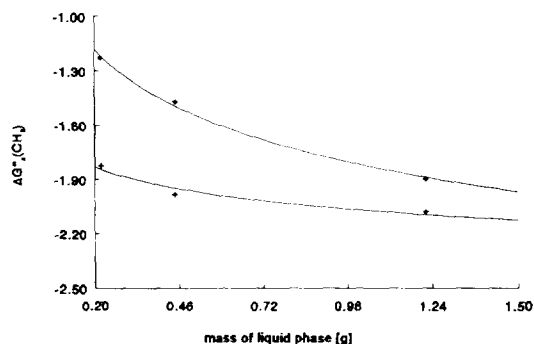


Fig. 6. Relationship between partial molal Gibbs function of solution per methylene group, $\Delta G_s^m(\text{CH}_2)$ (kJ mol^{-1}), and mass of the liquid phase (R_{n1} M340 at 343 K), (\blacklozenge) before and (+) after correction.

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