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Measurement of Activity Coefficients at Infinite Dilution Using Gas−Liquid Chromatography. 12. Results for Various Solutes with the Stationary Phases *N*-Ethylacetamide, *N,N*-Diethylacetamide, Diethylphthalate, and Glutaronitrile

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Using gas−liquid chromatography (GLC), activity coefficients at infinite dilution have been measured for 28 solutes (alkanes, alkenes, cyclic hydrocarbons, aromatic hydrocarbons, alcohols, ketones, ethers, aldehydes, esters, and halocarbons) in the solvents *N*-ethylacetamide, *N,N*-diethylacetamide, diethylphthalate, and glutaronitrile. The measurements were carried out in a temperature range between 303.15 K and 333.15 K. The obtained γ^∞ values were compared with published data and the observed temperature dependence of the limiting activity coefficients was confirmed using excess enthalpy data. Furthermore, the experimental data are compared with the results of modified UNIFAC (Dortmund).

Introduction

The activity coefficient at infinite dilution (limiting activity coefficient) represents an important property, which is used in particular for the selection of selective solvents (e.g., for extraction and extractive distillation) and for the reliable design of thermal separation processes. The removal of the last traces of impurities causes the largest separation effort (Gmehling and Brehm, 1996). To avoid an oversizing of the column (e.g., distillation column), which would lead to an increase of the investment and operating costs, reliable information about the separation factor at infinite dilution is required. Furthermore, more reliable g^E model or group interaction parameters can be obtained by fitting the parameters simultaneously to VLE, H^E, and limiting activity coefficients.

For the measurement of activity coefficients at infinite dilution (γ^∞) several methods are employed (Gmehling et al., 1994): the retention time method (gas−liquid chromatography), ebulliometry, static methods, and the dilutor technique, which can also be employed to determine γ^∞ values in solvent mixtures (Krummen et al., 2000).

The aim of the experimental measurements was to investigate solvents for which no or only little information is available. The following solvents were used as stationary phases: (1) *N*-ethylacetamide; (2) *N,N*-diethylacetamide; (3) diethylphthalate (softener in PVC); (4) glutaronitrile.

Experimental Procedure

For all measurements Chromosorb P-AW-DMCS 60/80 mesh (acid-washed dimethyldichlorosilane-treated Chromosorb) was used as the solid support for the stationary phase. The coating of the predried carrier material with solvent was carried out with methanol (solubilizer) in a rotary evaporator. After the solubilizer was removed, the column (length, 200 mm; inner diameter, 4.1 mm) was carefully filled with the coated solid support. A scheme of the homemade gas chromatograph used for these investigations, the detailed description of the measurement procedure, and the most important equations for evaluation purposes together with the theoretical explanations are given by Knoop et al. (1989) and Weidlich et al. (1987).

The solvents used were of a purity greater than 99.8 mass % (GC analysis of peak areas, CP-Wax 52 column, temperature of 150 °C, FID detector) and a water content smaller than 100 ppm (Karl Fischer titration). γ^∞-data for 28 solutes (alkanes, alkenes, cycloalkanes, aromatic hydrocarbons, ketones, ethers, aldehydes, and alcohols) in the four solvents were measured in a temperature range between 303.15 K and 333.15 K. Since GLC itself is a separation technique, the results are not influenced by small solute impurities, and therefore the solutes were used without further purification.

To check if solvent losses occurred during the measurements, the liquid loading was determined before and after the measurement gravimetrically. Under the stated conditions the pure uncoated Chromosorb suffers from a loss of mass due to the removal of strongly adsorbed water, which has been taken into account, too. With the use of presaturators the loss of solvent was kept to a minimum. The maximum solvent loss was about 8.5 mass % over a period of about 8 h and was taken into account assuming linear solvent loss during the isothermal measurements. Furthermore, the experimental conditions (gas flow, solvent loss, etc.) were checked by measuring the retention time of a reference substance (hexane) in regular intervals.

Equation 1 relates the activity coefficient of the solute at infinite dilution to the measured specific net retention volume (V_g^0) at 0 °C,

$$\gamma_i^\infty = \frac{273.15R}{V_g^0 P_i^s \varphi_i^s M_L} \tag{1}$$

where R is the general gas constant, M_L the molar mass of

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Table 1. Experimental Activity Coefficients at Infinite Dilution γ^∞ for Various Solutes in the Solvent *N*-Ethylacetamide

solvent: *N*-ethylacetamide (NEA)

solute	γ^∞ 30.0 °C	40.0 °C	50.0 °C	60.0 °C
pentane	7.55	7.35	7.23	7.03
hexane	9.23	8.95	8.79	8.51
heptane	11.4	11.0	10.7	10.3
octane	13.8	13.3	12.9	12.5
1-pentene	5.13	5.06	5.01	4.97
1-hexene	6.25	6.17	6.10	6.03
1-octene	9.06	9.00	8.97	8.89
cyclohexane	6.27	6.12	6.02	5.89
cyclohexene	4.56	4.51	4.48	4.44
benzene	2.43	2.42	2.44	2.43
toluene	3.12	3.12	3.12	3.13
methanol	0.661	0.664	0.671	0.673
ethanol	0.777	0.778	0.782	0.784
2-propanol	0.848	0.842	0.839	0.835
diethyl ether	3.12	3.09	3.09	3.08
diisopropyl ether	4.79		4.75	4.72
methyl *tert*-butyl ether	3.22	3.22	3.21	3.21
ethyl *tert*-butyl ether	4.31	4.31	4.32	4.32
methyl *tert*-amyl ether	3.89	3.87	3.85	3.83
tetrahydrofurane	1.73	1.71	1.71	1.70
acetone	2.07	1.98	1.93	1.87
2-butanone	2.08	2.02	1.99	1.94
2-pentanone	2.31	2.25	2.21	2.17
acetaldehyde	2.13	2.02	1.98	1.91
vinyl acetate	2.64	2.60	2.58	2.54
dichloromethane	0.866	0.905	0.954	0.992
chloroform	0.478	0.527	0.588	0.645

Table 2. Experimental Activity Coefficients at Infinite Dilution γ^∞ for Various Solutes in the Solvent *N,N*-Diethylacetamide

solvent: *N,N*-diethylacetamide (DEA)

solute	γ^∞ 30.0 °C	40.0 °C	50.0 °C	60.0 °C
pentane	4.33	4.08	3.94	3.74
hexane	5.02	4.70	4.45	4.21
heptane	5.76	5.35	5.06	4.74
octane	6.64	6.14	5.75	5.32
1-pentene	2.79	2.71	2.64	2.56
1-hexene	3.20	3.08	2.97	2.88
1-octene	4.22	4.04	3.86	3.68
cyclohexane	3.74	3.53	3.33	3.16
cyclohexene	2.50	2.43	2.35	2.29
benzene	0.948	0.971	0.990	1.01
toluene	1.20	1.20	1.20	1.20
methanol	0.418	0.434	0.446	0.460
ethanol	0.501	0.510	0.518	0.524
2-propanol	0.541	0.543	0.543	0.543
diethyl ether	1.89	1.86	1.83	1.81
diisopropyl ether	2.65	2.60	2.55	2.50
methyl *tert*-butyl ether	1.90	1.88	1.88	1.86
ethyl *tert*-butyl ether	2.49	2.45	2.41	2.37
methyl *tert*-amyl ether	2.14	2.11	2.07	2.05
tetrahydrofurane	1.11	1.11	1.12	1.12
acetone	1.03	1.03	1.04	1.04
2-butanone	1.01	1.02	1.03	1.04
2-pentanone	1.06	1.07	1.09	1.09
acetaldehyde	0.944	0.951	0.962	0.966
vinyl acetate	1.09	1.11	1.13	1.13
dichloromethane	0.260	0.281	0.311	0.329
chloroform	0.129	0.148	0.172	0.188

the solvent, and P_i^s the saturation vapor pressure of the solute calculated using Antoine constants taken from the Dortmund Data Bank (DDB). The saturation fugacity coefficient of the solute, φ_i^s, is calculated with the help of the Soave−Redlich−Kwong equation of state following Gmehling and Kolbe (1992). The required critical data and acentric factors are also taken from the DDB.

According to Conder and Young (1978), sometimes adsorption effects have to be taken into account; e.g., adsorption at the gas−liquid interface becomes more important with increasing polarity of the solvent. Polar solutes on nonpolar stationary phases lead to adsorption at the gas−liquid interface, often accompanied by adsorption on the solid support. To examine the presence of adsorption effects, the relative amount of stationary phase (liquid loading, 15−25 mass %) and the sample volume of the injected solutes (0.02−0.5 μL) were varied, but no adsorption effects were observed.

Results and Discussion

The values of the activity coefficients at infinite dilution for the investigated solutes in the four solvents at four temperatures are listed in Tables 1−4. The main source of error in the calculation of the specific retention volume corrected to 0 °C (V_g^0) is the measurement of the mass of the stationary phase in the column (±2%). In addition, there is a small error in the determination of difference between retention time and dead time (Knoop et al., 1989). The total error in the determination of the specific retention volume is about ±2.5%. Taking into account that the Antoine constants taken from the DDB used for the calculation of the saturation vapor pressure are also subject to error, the resulting error in γ^∞ is ±3.0%. Measurements with different amounts of stationary phase or flow rates lead to results within this error.

Table 3. Experimental Activity Coefficients at Infinite Dilution γ^∞ for Various Solutes in the Solvent Diethylphthalate

solvent: diethylphthalate

solute	γ^∞ 30.0 °C	40.0 °C	50.0 °C	60.0 °C
pentane	4.05	3.81	3.67	3.50
hexane	4.84	4.53	4.32	4.09
heptane	5.74	5.33	5.07	4.76
octane	6.86	6.34	5.95	5.55
1-pentene	2.60	2.51	2.46	2.39
1-hexene	3.07	2.94	2.87	2.78
1-octene	4.27	4.03	3.89	3.72
cyclohexane	3.24	3.04	2.90	2.76
cyclohexene	2.07	1.99	1.95	1.89
benzene	0.914	0.916	0.917	0.918
toluene	1.09	1.08	1.09	1.09
methanol	2.54	2.35	2.21	2.06
ethanol	2.80	2.58	2.40	2.21
1-propanol	2.82	2.54	2.34	2.14
2-propanol	2.83	2.57	2.38	2.17
diethyl ether	1.47	1.46	1.46	1.46
diisopropyl ether	2.28	2.26	2.25	2.21
methyl *tert*-butyl ether	1.58	1.57	1.57	1.56
ethyl *tert*-butyl ether	2.10	2.09	2.07	2.05
methyl *tert*-amyl ether	1.78	1.76	1.75	1.73
tetrahydrofurane	0.778	0.786	0.799	0.806
acetone	0.939	0.937	0.941	0.941
2-butanone	0.915	0.915	0.927	0.932
2-pentanone	0.989	0.991	1.00	1.01
acetaldehyde	0.906	0.905	0.903	0.901
vinyl acetate	1.09	1.09	1.09	1.09
dichloromethane	0.508	0.519	0.533	0.539
chloroform	0.424	0.445	0.468	0.490

Figure 1 shows examples of the linear relationship between the natural logarithm of the activity coefficients and the inverse absolute temperature for five different ethers in *N,N*-diethylacetamide. The limiting activity coefficient diminishes with decreasing molecular weight (di-

Table 4. Experimental Activity Coefficients at Infinite Dilution γ^∞ for Various Solutes in the Solvent Glutaronitrile

solvent: glutaronitrile				
	γ^∞			
solute	30.0 °C	40.0 °C	50.0 °C	60.0 °C
1-hexene	28.4	26.1	24.4	22.7
1-octene	65.8	59.0	53.8	48.9
cyclohexane	35.8	31.6	28.9	26.4
cyclohexene	15.8	14.6	13.7	12.9
benzene	3.51	3.42	3.37	3.30
toluene	5.21	5.05	4.95	4.81
methanol	2.85	2.63	2.45	2.27
ethanol	4.12	3.72	3.40	3.12
1-propanol	5.46	4.80	4.31	3.90
2-propanol	5.26	4.68	4.26	3.85
diethyl ether	6.32	6.24	6.18	6.09
diisopropyl ether	15.8	15.4	15.0	14.6
methyl *tert*-butyl ether	6.69	6.62	6.59	6.51
ethyl *tert*-butyl ether	14.6	14.1	13.7	13.3
methyl *tert*-amyl ether	10.4	10.2	9.96	9.68
tetrahydrofurane	2.09	2.09	2.09	2.08
acetone	1.25	1.25	1.26	1.26
2-butanone	1.74	1.74	1.73	1.73
2-pentanone	2.50	2.47	2.45	2.41
acetaldehyde	1.14	1.14	1.15	1.15
vinyl acetate	2.48	2.48	2.47	2.47
dichloromethane	1.20	1.22	1.25	1.27
chloroform	1.59	1.63	1.69	1.73

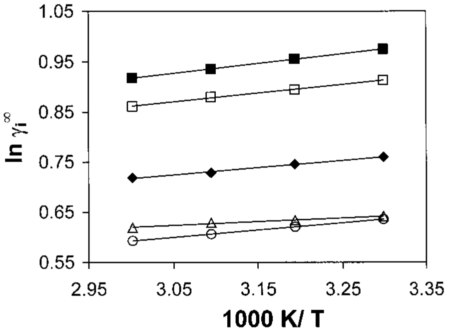

Figure 1. Experimental activity coefficients at infinite dilution γ^∞ for five solutes in the solvent *N*,*N*-diethylacetamide as a function of temperature: ■, diisopropyl ether; □, ethyl *tert*-butyl ether; ◆, methyl *tert*-amyl ether; △, methyl *tert*-butyl ether; ○, diethyl ether; —, linear regression.

isopropyl ether, ethyl *tert*-butyl ether, methyl *tert*-amyl ether: $M = 102.18$ g/mol; methyl *tert*-butyl ether: $M = 88.15$ g/mol; diethyl ether: $M = 74.12$ g/mol). A comparison of the ethers with the same molecular weight indicates that the activity coefficient is influenced by the molecular structure. With increasing symmetry of the molecule (diisopropyl ether > ethyl *tert*-butyl ether > methyl *tert*-amyl ether), a decrease in retention and therefore an increase of the γ^∞ value can be observed.

A comparison of the limiting activity coefficients of different alkenes (1-hexene and 1-octene) in the solvents *N*,*N*-diethylacetamide and *N*-ethylacetamide is shown in Figure 2. The γ^∞ values of the alkenes in the single-alkylated *N*-ethylacetamide are higher than the γ^∞ values in the double-alkylated *N*,*N*-diethylacetamide. This phenomenon can be explained with an increase of the hydrophobic character, when the nitrogen atom is alkylated twice (*N*,*N*-diethylacetamide). With an increasing degree of alkylation at the nitrogen atom, the solvent resembles more to the strongly hydrophobic solutes 1-hexene and 1-octene. This effect also explains the higher limiting activity coefficients of 1-octene compared to those of the 1-hexene. The same dependency from the chain length can be found for the alkanes.

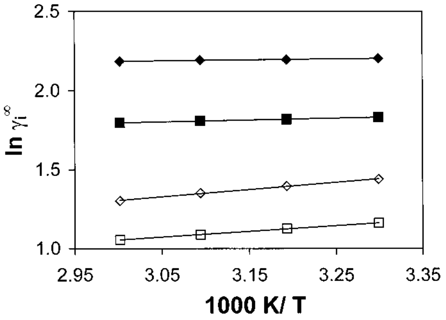

Figure 2. Experimental activity coefficients at infinite dilution γ^∞ for four solutes in the solvent *N*-ethylacetamide (NEA) and *N*,*N*-diethylacetamide (DNEA) as a function of temperature: ◆, 1-octene in NEA; ■, 1-hexene in NEA; ◇, 1-octene in DNEA; □, 1-hexene in DNEA; —, linear regression.

In contrast to the above polar solutes, methanol or ethanol show a negative deviation from Raoult's law. An extension of the carbon chain leads to stronger deviations from ideality ($\gamma^\infty = 1$), both for *N*,*N*-diethylacetamide and *N*-ethylacetamide. Once again, this effect can be explained with hydrophobicity. Both acetamides show a less hydrophobic character compared with the investigated alcohols. Decreasing the chain length of the alcohols (methanol, ethanol) results in stronger solute–solvent interactions, i.e., stronger negative deviation from Raoult's law.

The choice of the optimal temperature is an important criterion for the employment of selective solvents. According to the Gibbs–Helmholtz equation, the value for the partial molar excess enthalpy at infinite dilution, $H_i^{E,\infty}$, can directly be obtained from the slope of a straight line (Gmehling and Kolbe, 1992) derived from

$$\left(\frac{\partial \ln \gamma_i^\infty}{\partial(1/T)}\right) = \frac{H_i^{E,\infty}}{R} \tag{2}$$

Experimental H^E data can be used to confirm the observed experimental temperature dependence. With the help of a polynomial (Redlich–Kister (Redlich and Kister, 1948) or SSF (sum of symmetrical functions) (Rogalski and Malanowski, 1977)) the value of the partial molar excess enthalpy at infinite dilution, $H_i^{E,\infty}$, can be calculated. For the majority of the solutes investigated the activity coefficients at infinite dilution tend toward ideal behavior ($\gamma^\infty = 1$) with increasing temperature. For components with γ^∞ values less than unity this implies an increase of the activity coefficient ($H_i^{E,\infty} < 0$) and for components with γ^∞ values greater than unity a decrease of the activity coefficient with increasing temperature ($H_i^{E,\infty} > 0$).

A confirmation of the calculated $H_i^{E,\infty}$ values is shown in Table 5, where experimental enthalpy of mixing data (Ferino et al., 1985; Novoselova et al., 1977) for the same systems (hexane in diethylphthalate, hexane in *N*,*N*-diethylacetamide, and heptane in *N*,*N*-diethylacetamide) are shown. For all systems a comparison with H^E data was performed with the help of the DDB. In all cases the temperature dependence of the limiting activity coefficient obtained by GLC is confirmed by the experimental H^E data. The accuracy of the calculated partial molar excess enthalpies at infinite dilution depends, on one hand, on the experimental determined limiting activity coefficients. If the slope of the straight line (ln γ^∞ vs 1000/T) is sufficiently large, a satisfactory to good reproduction of the value for the partial molar excess enthalpy at infinite dilution is

Table 5. Calculated Partial Molar Excess Enthalpies at Infinite Dilution $H_i^{E,\infty}$ for the Different Systems

solvents (1)	solute (2)	$H_2^{E,\infty}$ (J mol^{-1})[a]	ϑ (°C)	$H_2^{E,\infty}$ (J mol^{-1})[b]	ϑ (°C)	reference
diethylphthalate	hexane	4635	30−60	6585	25	Novoselova et al. (1977)
N,*N*-diethylacetamide	hexane	4903	30−60	3372	25	Ferino et al. (1985)
N,*N*-diethylacetamide	heptane	5420	30−60	3842	25	Ferino et al. (1985)

[a] Calculated by linear regression from experimental γ^∞ data. [b] Calculated from experimental H^E data using polynomials (Redlich−Kister, SSF).

Table 6. Comparison of Experimental γ^∞ Data with Predicted γ^∞ Values (mod. UNIFAC (Do))

		γ^∞			
solvent	solute	30 °C	40 °C	50 °C	60 °C
glutaronitrile	toluene	5.21	5.05	4.95	4.81
		(5.48)	(5.38)	(5.29)	(5.21)
	benzene	3.51	3.42	3.37	3.30
		(3.92)	(3.86)	(3.81)	(3.76)
	ethanol	4.12	3.72	3.40	3.12
		(5.98)	(5.45)	(4.99)	(4.60)
diethylphthalate	toluene	1.09	1.08	1.09	1.09
		(1.13)	(1.12)	(1.11)	(1.10)
	acetone	0.939	0.937	0.941	0.941
		(0.92)	(0.93)	(0.94)	(0.94)
	hexane	4.84	4.53	4.32	4.09
		(5.25)	(4.97)	(4.70)	(4.45)
	methanol	2.54	2.35	2.21	2.06
		(2.92)	(2.69)	(2.48)	(2.30)

[a] () give the predicted γ^∞ values using mod. UNIFAC (Do).

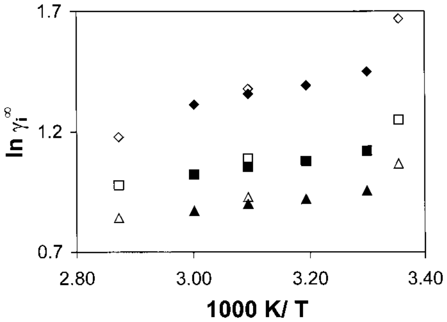

Figure 3. Experimental activity coefficients at infinite dilution γ^∞ for three solutes in the solvent diethylphthalate as a function of temperature. This work: ◆, 1-octene; ■, 1-hexene; △, 1-pentene. Published values: ◇, 1-octene; □, 1-hexene; △, 1-pentene (Alessi et al., 1975).

possible despite the GLC experimental error (≈3%; Möllmann and Gmehling, 1997). On the other hand, enough H^E data points in the diluted range $x_1 \rightarrow 0$ (small solute concentration) are required in order to obtain a good polynomial interpolation. A good criterion for the reliability of the temperature dependence of the limiting activity coefficients γ^∞ is the correct sign and the approximate value of the partial molar excess enthalpies ($H_i^{E,\infty}$) at infinite dilution.

In Table 6 limiting activity coefficients for the systems methanol, acetone, toluene, and hexane in diethylphthalate and benzene, toluene, and methanol in glutaronitrile are compared with predicted γ^∞ values of the group contribution method mod. UNIFAC (Do) (Gmehling et al., 1993; Gmehling et al., 1998). It can be seen that the experimental data are in good agreement with the predicted data. This underlines the importance of mod. UNIFAC (Do) as a software tool for the selection of selective solvents.

Figure 3 shows examples of the linear relationship between the natural logarithm of the activity coefficient and the inverse absolute temperature for selected solutes in diethylphthalate. The figure additionally contains γ^∞ data taken from the literature which were obtained by GLC (Alessi et al., 1975). The results presented here show a good agreement of our experimental values with the published data.

Conclusion

Activity coefficients at infinite dilution for 28 solutes in the solvents *N*-ethylacetamide, *N*,*N*-diethylacetamide, diethylphthalate, and glutaronitrile have been measured at different temperatures with the help of GLC. This technique has been chosen due to the possibility of a fast and reliable determination of γ^∞ values.

The selection of the solvents focused thereby on solvents for which so far no or only few experimental data were available.

The accuracy of the group contribution method mod. UNIFAC (DO) was proven by comparing experimental γ^∞ data with calculated values. This underlines the applicability of the model as a predictive method for the synthesis and design of thermal separation processes.

To estimate the influence of the degree of substitution at the nitrogen atom on the γ^∞ values and thus on the selectivity or capacity, investigations for the solvents *N*-ethylacetamide and *N*,*N*-diethylacetamide were performed.

To check the quality of the measured data, a comparison with available H^E data and a comparison with published data was executed.

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