Measurement of Activity Coefficients at Infinite Dilution Using Gas−Liquid Chromatography. 9. Results for Various Solutes with the Stationary Phases 2-Pyrrolidone and *N*-Methylformamide

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The selection of the most suitable selective solvent for separation processes such as extraction or extractive distillation plays an important role in the economical design. Since the largest deviation from ideality is observed at infinite dilution, limiting activity coefficients (γ^∞) provide a useful tool for the optimal choice of the selective solvent (entrainer). Therefore, activity coefficients at infinite dilution have been measured for 31 solutes (alkanes, alkenes, cyclic hydrocarbons, aromatic hydrocarbons, alcohols, ketones, ethers, and halocarbons) in the solvents 2-pyrrolidone and *N*-methylformamide. The measurements were carried out with the help of gas−liquid chromatography (GLC) at four temperatures, (30, 40, 50, and 60) °C. The γ^∞ values are compared with published data. Furthermore, the observed temperature dependence of the limiting activity coefficients is confirmed using excess enthalpy data. To verify the application as an entrainer, selectivity and capacity of the investigated solvents are compared to those of similar structured solvents such as *N*-methyl-2-pyrrolidone and *N*,*N*-dimethylformamide.

Introduction

Values of limiting activity coefficients have important practical and direct applications for industrial problems, since activity coefficients at infinite dilution can directly be used for the selection of selective solvents for extractive distillation or extraction. To be able to estimate the selectivity of an entrainer, it is often sufficient to know the separation factor at infinite dilution (Gmehling and Brehm, 1996). In the highly dilute range, the ratio of the activity coefficients of the components to be separated is denoted as the selectivity S_{12}^∞.

$$S_{12}^\infty = \gamma_1^\infty/\gamma_2^\infty \quad (1)$$

To limit the number of theoretical stages required, the selectivity should be far from unity. Assuming that 2 is the component to be extracted with the selective solvent, selectivities greater than 1 are desired. Apart from the selectivity, the capacity of the solvent has to be considered (Hradetzky et al., 1989). The capacity describes the solubility of the component to be extracted in the solvent; i.e., small values for the activity coefficient indicate a high solubility. In the case of infinite dilution the capacity k_1^∞ can be defined as

$$k_1^\infty = 1/\gamma_1^\infty \quad (2)$$

A typical application of extractive distillation or liquid−liquid extraction is the separation of aromatics from aliphatics. In most cases capacity and selectivity counteract; i.e., high values for the capacity are accompanied by low values for the selectivity. Therefore, an economic and effective separation needs a solvent that combines a sufficient capacity with a high selectivity. Furthermore, there are many other aspects that have to be considered (high thermal stability, low corrosiveness, low vapor pressure, low price, etc.), but they are beyond the scope of this paper.

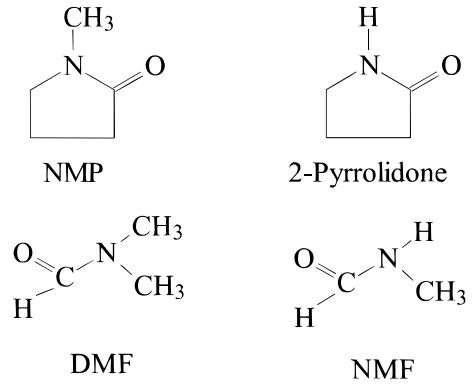

Figure 1. Molecular structures of *N*-methyl-2-pyrrolidone (NMP), 2-pyrrolidone, *N*,*N*-dimethylformamide (DMF), and *N*-methylformamide (NMF).

The investigated solvents were chosen because of their limited database for γ^∞ and since similar structured components are used as selective solvents in commercial extraction processes. Except for the methylated nitrogen atom, 2-pyrrolidone has the same structure as *N*-methyl-2-pyrrolidone (NMP). The structure of *N*-methylformamide (NMF) differs from that of *N*,*N*-dimethylformamide (DMF) only by the grade of methylation of the nitrogen atom (see Figure 1).

In a systematic study the limiting activity coefficients have been determined for 31 substances in two solvents (2-pyrrolidone and *N*-methylformamide) in order to obtain quantitative information for the entrainer selection. The GLC technique was chosen as the most preferable measuring technique for the planned investigations because GLC

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shows several advantages in comparison to other methods, e.g., static methods, dilutor technique, etc. (Gruber et al., 1998a). After the careful preparation of the column, the GLC method allows the measurement of a great number of γ^∞ values in a rather short time. The reliability of the γ^∞ data obtained by GLC has been confirmed by different authors (Gmehling et al., 1994) and in earlier parts of this measurement series (Measurement of Activity Coefficients at Infinite Dilution Using Gas−Liquid Chromatography: Part 1, Weidlich and Gmehling (1987); Part 2, Weidlich et al. (1987); Part 3, Knoop et al. (1989); Part 4, Schiller and Gmehling (1992); Part 5, Möllmann and Gmehling (1997); Part 6, Gruber et al. (1997); Part 7, Gruber et al. (1998a); Part 8, Gruber et al. (1998b)).

Experimental and Measurement 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 the 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. The determination of the liquid loading (i.e., the amount of stationary phase (solvent) on the inert carrier material) was carried out gravimetrically. A detailed scheme of the homemade gas chromatograph used for these investigations, the 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).

The solvents used, 2-pyrrolidone and *N*-methylformamide, were applied with a purity greater than 99.8% (GLC analysis of peak areas, CP-Wax 52 column, temperature 150 °C, FID detector) and a water content smaller than 130 ppm (Karl Fischer titration). γ^∞ values for 31 solutes (alkanes, alkenes, cyclic hydrocarbons, aromatic hydrocarbons, alcohols, ketones, ethers, and halocarbons) were measured at four temperatures, (30, 40, 50, and 60) °C. Since GLC is itself a separation technique, the results are not influenced by small solute impurities, and therefore the solutes were used without further purification.

The amount of solvent in the column has been determined gravimetrically. To check if solvent losses occurred during the measurements, the liquid loading was determined before and after the measurement. Under the stated conditions the pure uncoated Chromosorb suffers from a loss of mass owing 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. This ranged typically between 2 and 6 mass % over a period of about 6 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.

For the determination of γ^∞ the following information is required: the net retention time of the solute, the temperature, column inlet and outlet pressure, the carrier gas flow rate, and the amount of the stationary phase on the solid support. With the help of these experimentally determined parameters, the specific net retention volume (V_g^0) corrected to 0 °C is obtained. The activity coefficient at infinite dilution is then given by

$$\gamma_i^\infty = \frac{273.15R}{V_g^0 P_i^s \varphi_i^s M_L} \tag{3}$$

where R is the gas constant, M_L the molar mass of 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 φ_i^s of the solute 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. The net retention time is equal to the difference between the retention time of the solute (solute peak) and the dead time (peak caused by air that is injected together with the solute). The net retention time is a measure of the partition of the solute between the gas (mobile) phase and the stationary phase (solvent). This partitioning is a result of phase equilibrium behavior.

To examine the presence of adsorption effects the relative amount of stationary phase (liquid loading: 14−30%) and the sample volume of injected solutes (0.02−0.5 μL) were varied. Contrary to the observations in our previous investigations (Gruber et al., 1997), no adsorption effects were observed.

Table 1. Experimental Activity Coefficients at Infinite Dilution, γ^∞, for Various Solutes in the Solvent 2-Pyrrolidone

	T (K)			
solute	303.15	313.15	323.15	333.15
pentane	31.82	29.29	27.44	25.43
hexane	42.56	38.81	35.60	33.11
heptane	55.32	49.82	45.54	41.92
octane	73.33	65.83	60.27	54.98
cyclopentane	17.18	15.99	15.10	14.11
cyclohexane	24.54	22.56	20.82	19.40
methylcyclopentane	24.93	23.12	21.53	20.03
methylcyclohexane	31.89	29.39	27.29	25.38
1-pentene	16.07	15.29	14.60	13.98
1-hexene	20.25	19.37	18.47	17.73
1-octene	36.57	33.99	31.94	30.13
cyclohexene	12.53	11.82	11.15	10.63
benzene	2.809	2.825	2.843	2.854
toluene	4.004	4.036	4.063	4.087
ethylbenzene	5.639	5.600	5.558	5.516
1,2-xylene	5.291	5.234	5.198	5.154
1,3-xylene	6.034	5.918	5.815	5.707
1,4-xylene	6.073	6.010	5.928	5.865
methanol	0.744	0.752	0.759	0.766
ethanol	1.060	1.048	1.036	1.024
acetone	2.304	2.265	2.230	2.196
2-butanone	2.805	2.755	2.710	2.670
2-pentanone	3.571	3.518	3.469	3.423
diethyl ether	7.470	7.231	7.021	6.853
diisopropyl ether	14.41	13.91	13.32	12.92
methyl *tert*-amyl ether	10.70	10.32	9.939	9.674
methyl *tert*-butyl ether	8.235	8.038	7.860	7.655
ethyl *tert*-butyl ether	12.95	12.43	11.93	11.62
dichloromethane	0.821	0.863	0.906	0.951
chloroform	0.534	0.591	0.653	0.713

Results and Discussion

Activity coefficients at infinite dilution of various types of solutes were determined for 2-pyrrolidone and *N*-methylformamide at four temperatures, (30, 40, 50, and 60) °C. The results are presented in Tables 1 and 2.

The main source of error in the calculated γ^∞ values is the accuracy of the mass of the stationary phase in the

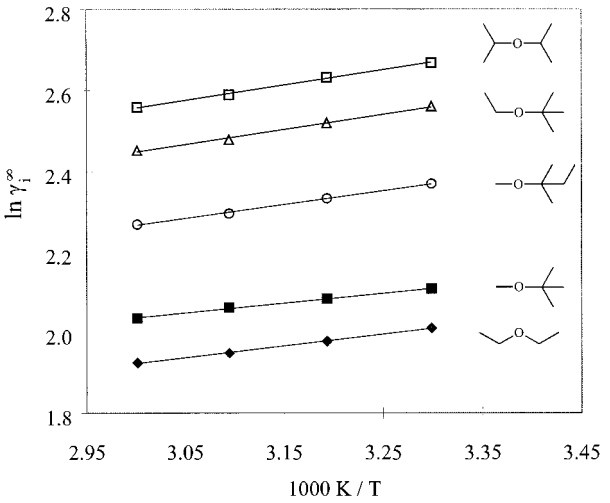

Figure 2. Experimental activity coefficients at infinite dilution γ_i^∞ for five solutes in the solvent 2-pyrrolidone as a function of temperature: (□) diisopropyl ether, (△) ethyl *tert*-butyl ether, (○) methyl *tert*-amyl ether, (■) methyl *tert*-butyl ether, (◆) diethyl ether, (—) linear regression.

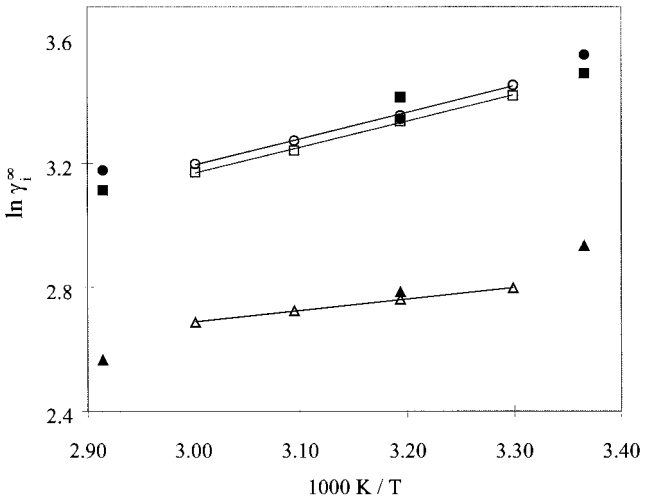

Figure 3. Activity coefficients at infinite dilution γ_i^∞ in the solvent *N*-methylformamide as a function of temperature: this work: (△) 1-pentene, (□) 2-methylbutane, (○) pentane, (—) linear regression; published values (Smiley, 1970): (▲) 1-pentene, (■) 2-methylbutane, (●) pentane.

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

	T (K)			
solute	303.15	313.15	323.15	333.15
pentane	31.51	28.60	26.40	24.49
hexane	41.39	38.46	36.06	33.94
heptane	57.72	52.46	47.54	44.04
octane	80.48	71.89	64.57	58.58
2-methylbutane	30.48	28.08	25.58	23.84
cyclohexane	23.88	22.51	21.23	20.05
methylcyclopentane	24.30	22.95	21.82	20.75
methylcyclohexane	31.15	29.45	27.91	26.69
1-pentene	16.40	15.82	15.24	14.68
1-hexene	22.09	21.11	20.17	19.44
1-octene	40.30	38.18	36.22	34.39
cyclohexene	14.23	13.67	13.15	12.69
benzene	5.014	4.917	4.837	4.751
toluene	7.262	7.140	6.997	6.876
ethylbenzene	9.744	9.566	9.385	9.228
1,2-xylene	9.901	9.586	9.331	9.113
1,3-xylene	10.91	10.61	10.31	10.04
1,4-xylene	10.59	10.33	10.06	9.812
methanol	1.062	1.031	0.994	0.969
ethanol	1.379	1.318	1.265	1.223
acetone	2.140	2.107	2.076	2.047
2-butanone	2.561	2.536	2.510	2.488
2-pentanone	3.212	3.186	3.164	3.141
diethyl ether	6.550	6.441	6.342	6.245
diisopropyl ether	12.32	12.08	11.89	11.70
methyl *tert*-amyl ether	9.255	9.103	8.960	8.820
methyl *tert*-butyl ether	6.821	6.771	6.720	6.670
ethyl *tert*-butyl ether	11.25	11.05	10.84	10.65
dichloromethane	1.483	1.535	1.585	1.631
chloroform	1.155	1.241	1.324	1.412

column. This error was assumed to be approximately 2%. In addition, there is a small error in the determination of the retention time (Knoop et al., 1989). 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 the γ^∞ is 3.0%.

Figure 2 shows examples of the linear relationship between the natural logarithm of the activity coefficients and the inverse absolute temperature for five different ethers in 2-pyrrolidone. The limiting activity coefficient diminishes with decreasing molecular weight (diisopropyl 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.

One of the reasons for the investigation of the solvents 2-pyrrolidone and *N*-methylformamide was the very limited database of activity coefficients at infinite dilution. Published data are only available for C_5 hydrocarbons (pentane, 2-methylbutane, and 1-pentene) in the solvent *N*-methylformamide (Smiley, 1970). Figure 3 shows that the experimental data are in good accordance with the published data.

A graph of ln γ^∞ versus the inverse absolute temperature often shows a straight line in a limited temperature interval (see Figures 2, 3 and 4). 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 this straight line (Gmehling and Kolbe, 1992).

$$\left(\frac{\partial \ln \gamma_i^\infty}{\partial \frac{1}{T}}\right)_{P,x} = \frac{H_i^{E,\infty}}{R} \tag{4}$$

Experimental H^E data can be used to confirm the observed temperature dependence. With the help of a polynomial (Redlich–Kister or SSF (*s*um of *s*ymmetrical *f*unctions)), the value of 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 (γ^∞ = 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 ($H_i^{E,\infty} > 0$). Figure 4 illustrates the temperature dependence of the limiting activity coefficient for the solutes benzene and toluene in the solvent

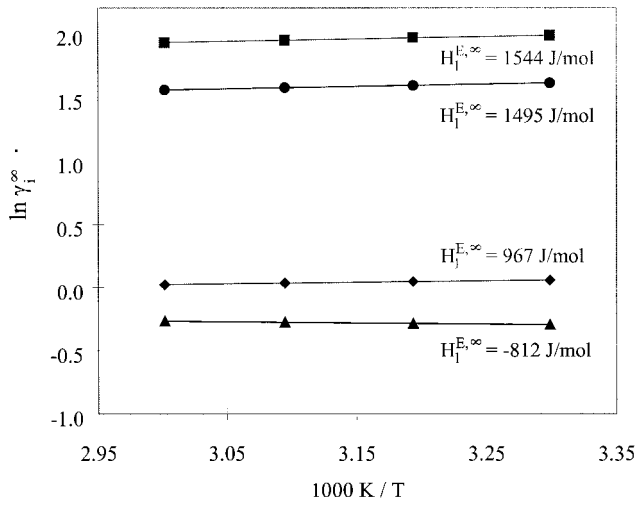

Figure 4. Temperature dependence of the limiting activity coefficients and the calculated partial molar excess enthalpies at infinite dilution $H_i^{E,\infty}$ for the systems: (■) toluene (1) in *N*-methylformamide, (●) benzene (1) in *N*-methylformamide, (◆) ethanol (1) in 2-pyrrolidone, (▲) methanol (1) in 2-pyrrolidone, (—) linear regression.

N-methylformamide and for the solutes ethanol and methanol in the solvent 2-pyrrolidone. For the system with methanol, the γ^∞ values increase with increasing temperature and a negative value of the partial molar excess enthalpy at infinite dilution can be expected. The other systems show the opposite temperature dependence of the activity coefficients, and therefore positive $H_i^{E,\infty}$ values are expected. A confirmation of the calculated $H_i^{E,\infty}$ values is shown in Figure 5 where experimental heat of mixing data (Becker and Rhensius, 1987; Mehta et al., 1997) for the same systems are shown. 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 the one side 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 possible despite the GLC experimental error (approximately 3%; Möllmann and Gmehling, 1997). On the other side, 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 γ^∞ data, however, can be seen in the correct reproduction of the direction of the temperature dependence of the limiting activity coefficients with the help of H^E measurements.

Another aim of the investigation was the selection of new potential entrainers, especially for separation of aromatics from aliphatics. Therefore, solvents with structures similar to those of already commercially used solvents were investigated. *N*-methylformamide and 2-pyrrolidone differ with their structure relative to *N*,*N*-dimethylformamide and *N*-methyl-2-pyrrolidone only by the degree of alkylation of the nitrogen atom (see Figure 1).

Regarding the selectivity at infinite dilution S_{12}^∞ (eq 1) for the typical separation of cyclohexane (1) from benzene (2), the investigated solvent *N*-methylformamide shows a higher selectivity than *N*,*N*-dimethylformamide (see Figure

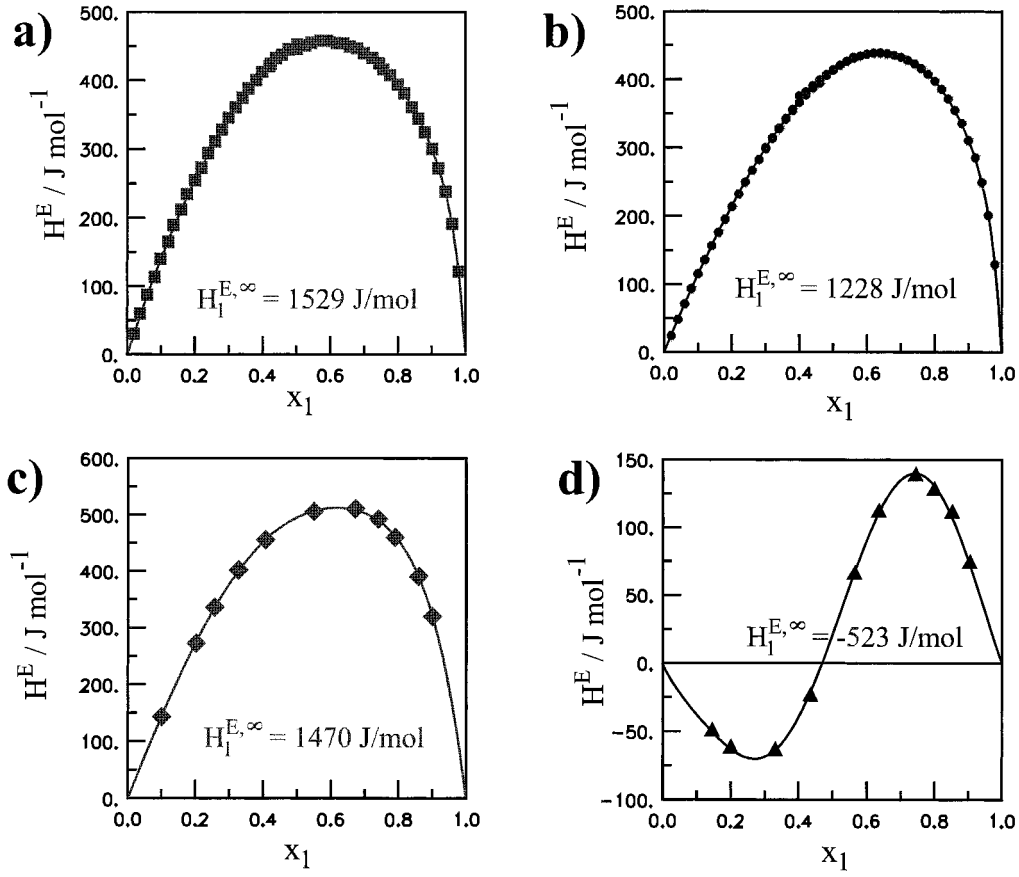

Figure 5. H^E data at different temperatures (25 °C and 30 °C) for the systems (a) (■) toluene (1) in *N*-methylformamide (Becker and Rhensius, 1987); (b) (●) benzene (1) in *N*-methylformamide (Becker and Rhensius, 1987); (c) (◆) ethanol (1) in 2-pyrrolidone (Mehta et al., 1997); (d) (▲) methanol (1) in 2-pyrrolidone (Mehta et al., 1997), (—) polynomial interpolation.

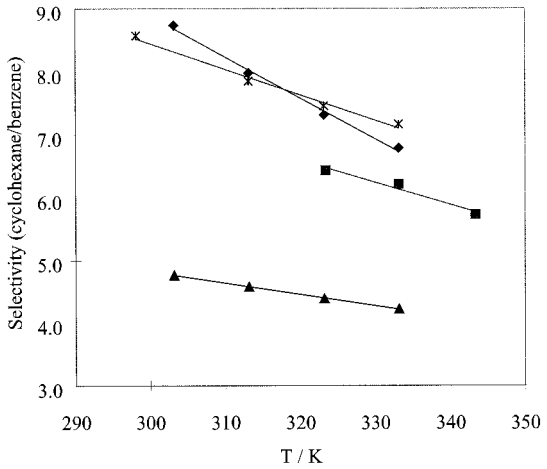

Figure 6. Selectivities at infinite dilution for four solvents at different temperatures regarding the separation of cyclohexane from benzene: (▲) in *N*-methylformamide (this work); (*) in *N*,*N*-dimethylformamide (Popescu et al., 1967); (◆) in 2-pyrrolidone (this work); (■) in *N*-methyl-2-pyrrolidone (Weidlich et al., 1987); (—) linear regression.

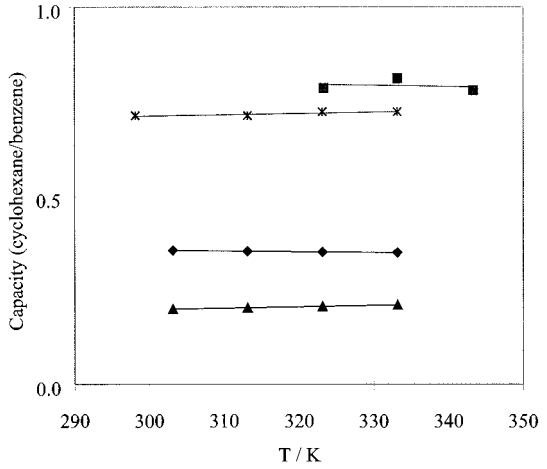

Figure 7. Capacities of benzene at infinite dilution for four solvents at different temperatures: (▲) in *N*-methylformamide (this work); (*) in *N*,*N*-dimethylformamide (Popescu et al., 1967); (◆) in 2-pyrrolidone (this work); (■) in *N*-methyl-2-pyrrolidone (Weidlich et al., 1987); (—) linear regression.

6). In contrast to this, the selectivity for cyclic amides increases with the degree of methylation at the nitrogen S_{12}^{∞}(2-pyrrolidone) > S_{12}^{∞}(*N*-methyl-2-pyrrolidone)).

With respect to the capacity at infinite dilution (eq 2), which is a measure for the solubility of a substance in the extractive solvent, the commercially used solvents *N*,*N*-dimethylformamide and *N*-methyl-2-pyrrolidone show great advantages in comparison to the investigated solvents (see Figure 7).

To decide what solvent is the most suitable, the quantity Q_{12} is introduced (Hradetzky et al., 1989). Assuming that component 2 is the component to be extracted, the quantity at infinite dilution is defined as the product of selectivity and capacity

$$Q_{12}^{\infty} = S_{12}^{\infty} k_2^{\infty} \tag{5}$$

Regarding the typical aliphatic/aromatic separation problem cyclohexane/benzene, the quantities at infinite dilution of the investigated solvents 2-pyrrolidone and *N*-methylformamide show smaller values in comparison to the commercially used solvents *N*-methyl-2-pyrrolidone and *N*,*N*-dimethylformamide (Figure 8). Comparing the Q_{12}^{∞} values of 2-pyrrolidone with *N*-methyl-2-pyrrolidone, the values for *N*-methyl-2-pyrrolidone are a factor of 2 higher. In the case of *N*-methylformamide and *N*,*N*-dimethylformamide, the Q_{12}^{∞} values of *N*,*N*-dimethylformamide are five times higher. These data indicate that the solvents 2-pyrrolidone and *N*-methylformamide show disadvantages for the aliphatic/aromatic separation when compared with the commercially used solvents *N*-methyl-2-pyrrolidone and *N*,*N*-dimethylformamide. The degree of alkylation at the nitrogen atom of the amide group strongly influences the properties of an entrainer.

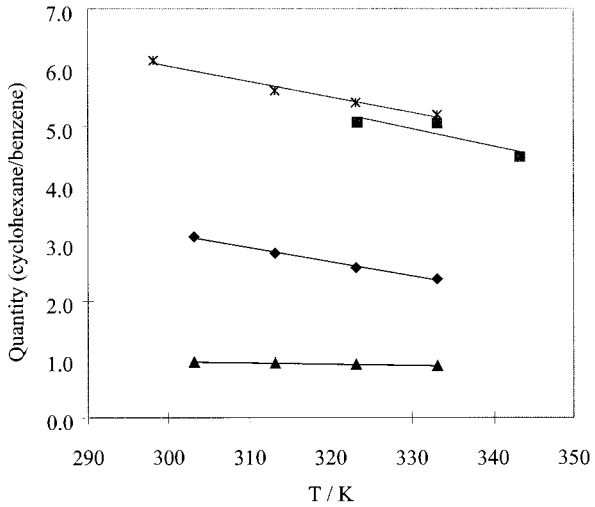

Figure 8. Quantities at infinite dilution for four solvents at different temperatures regarding the separation of cyclohexane from benzene: (▲) in *N*-methylformamide (this work); (*) in *N*,*N*-dimethylformamide (Popescu et al., 1967); (◆) in 2-pyrrolidone (this work); (■) in *N*-methyl-2-pyrrolidone (Weidlich et al., 1987); (—) linear regression.

Conclusion

Activity coefficients at infinite dilution for 31 solutes in the solvents 2-pyrrolidone and *N*-methylformamide have been measured at four different temperatures by GLC. The GLC technique was chosen owing to the possibility of a fast and reliable determination of γ^{∞} values. The reliability of the obtained data was confirmed by comparing these data with literature values. Furthermore, the temperature dependence of the limiting activity coefficients was verified using experimental H^E data. To check if the investigated solvents can be used as selective solvents for the aliphatic/aromatic separation, the data of selectivity, capacity, and the product Q_{12} at infinite dilution were compared with the values for *N*-methyl-2-pyrrolidone and *N*,*N*-dimethylformamide, which are already used as selective solvents.

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