

Excess Molar Volumes and Viscosities of Binary Mixtures of 4-Methylpyridine with Methanol, Ethanol, Propan-1-ol, Propan-2-ol, Butan-2-ol, and 2-Methylpropan-2-ol at 298.15 K and Atmospheric Pressure

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New experimental data on densities and viscosities for the systems 4-methylpyridine + methanol, + ethanol, + propan-1-ol, + propan-2-ol, + butan-2-ol, and + 2-methylpropan-2-ol are presented at 298.15 K and ambient pressure using a vibrating tube densimeter and an Ubbelohde viscometer. The results are discussed qualitatively in terms of the association of the mixture components caused by chainlike self-association of the alcohol molecules and cross-association between alcohol and 4-methylpyridine.

1. Introduction

Thermodynamic properties of mixtures containing alcohols and components having strong proton acceptor groups such as amines or pyridine and their derivatives are of interest in the study of molecular fluids where hydrogen bonding is the prevailing contribution to the intermolecular interaction. It is well-known that alcohol molecules associate mainly by building up linear chains. The extent of this association depends in a specific way on the concentration of the alcohol in the liquid state, on temperature, and also on pressure. In mixtures of alcohols with nonpolar organic compounds such as hydrocarbons the thermodynamic excess properties G^E , H^E , and V^E show a characteristic behavior governed by the fact that hydrogen bondings are broken upon mixing. This can be explained by suitable statistical thermodynamic models which account for the chainlike association such as the ERAS model (Heintz, 1985; Heintz et al., 1986a; Costas et al., 1987; Kaur et al., 1991; Bender and Heintz, 1993). Of special interest are mixtures of alcohols with amines and related substances since the OH group of the alcohol molecules forms a strong hydrogen bond with the nitrogen atom of the amine upon mixing which overcompensates for the energy needed for disrupting the OH...O bonding of the alcohol, resulting in very negative values of H^E and V^E . These effects can also be described by the ERAS model in a quantitative way which allows one to gain some insight into the intermolecular energetics and fluid structure of these kinds of associating mixtures (Funke et al., 1989; Reimann and Heintz, 1991; Bender et al., 1991; Mohren and Heintz, 1997; Heintz et al., 1998; Heintz and Papaioannou, 1998). Mixtures of alcohols with pyridine and its derivatives such as 4-methylpyridine present a class of mixtures where similar properties are expected as in alcohol + amine mixtures. Besides measurements of activity coefficients and the heats of mixing, the molar excess volume is an interesting property. In this respect also viscosities of alcohol + 4-methylpyridine mixtures give valuable information. The degree of association in these kinds of mixtures contributes to the viscosity. The higher the average chain

Table 1. Densities and Dynamic Viscosities of Pure Liquids at 298.15 K and 1 bar and Comparison with Data from Literature

component	$\rho/(\text{kg m}^{-3})$ exptl	$\eta/(\text{mPa s})$		source
		exptl	lit.	
γ -picoline	950.024	0.8600	0.8628 0.8369	Blasco et al., 1993 Lafuente et al., 1994
methanol	786.433	0.5547	0.5530 0.558 0.553	Orge et al., 1997 Cea et al., 1995 Canosa et al., 1998
ethanol	785.140	1.0969	0.550 1.1050 1.105	Sastry et al., 1998 Orge et al., 1997 Canosa et al., 1998
propan-1-ol	799.624	1.9429	1.943 1.943 1.951 1.953	TRC Tables, 1994, 1996 Sastry et al., 1998 Pal and Kumar, 1998 Shan and Ashfour, 1999
propan-2-ol	781.024	2.0766	2.098 2.0436 2.082	Canosa et al., 1998 TRC Tables, 1994, 1996 Haase and Tillmann, 1995
<i>sec</i> -butanol	804.486	2.8571	2.998 3.084 3.0596	TRC Tables, 1994, 1996 Cea et al., 1995 Lafuente et al., 1994
<i>tert</i> -butanol	781.626	4.3719	n. a.	

length of the associated molecules in the mixture is, the higher will be the viscosity.

A series of experimental work on viscosities and partly on excess volumes of mixtures of alcohols with hydrocarbons and alcohols with pyridine exists already in the literature (e.g., Canosa et al. (1998), Sastry and Valand (1998), Pal and Kumar (1998), Dewan et al. (1991), Singh et al. (1980), Kowalski and Orszagh (1975), Nakanishi and Shirai (1970)).

In this work we report on measurements of the molar excess volume and mixture viscosities of 4-methylpyridine (γ -picoline) + methanol, + ethanol, + propan-1-ol, + propan-2-ol, + butan-2-ol (*sec*-butanol), and + 2-methylpropan-2-ol (*tert*-butanol) at 298.15 K.

2. Experimental Section

The liquids used were supplied by Merck/Germany (methanol, ethanol), Fluka/Germany (γ -picoline, propan-2-ol), and Reanal/Hungary (propan-1-ol, *sec*-butanol, *tert*-butanol). GLC analysis of the pure liquids showed the

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Table 2. Densities ρ , viscosities η , Differences of Logarithm Viscosities $\Delta \ln \eta$, and Excess Molar Volumes V^E for Binary Mixtures at 298.15 K and Atmospheric Pressure

mole fraction	$\rho/(\text{kg m}^{-3})$	$\eta/(\text{mPa s})$	$\Delta \ln \eta$	$V^E/(\text{10}^{-6} \text{ m}^3 \text{ mol}^{-1})$	mole fraction	$\rho/(\text{kg m}^{-3})$	$\eta/(\text{mPa s})$	$\Delta \ln \eta$	$V^E/(\text{10}^{-6} \text{ m}^3 \text{ mol}^{-1})$
γ -Picoline (x) + Methanol ($1 - x$)									
0.0000	950.07	0.8600	0.0000	0.0000	0.0000	891.19	0.7764	0.1752	-0.6450
0.1671	940.45	0.8666	0.0809	-0.2771	0.7977	856.49	0.7033	0.1486	-0.4822
0.2760	932.75	0.8458	0.1044	-0.4425	0.8892	829.46	0.6526	0.1138	-0.2893
0.3035	930.58	0.8547	0.1269	-0.4818	0.9670	800.82	0.5799	0.0299	-0.0942
0.4194	919.96	0.8291	0.1473	-0.6121	1.0000	786.66	0.5547	0.0000	0.0000
0.5171	908.54	0.8161	0.1744	-0.6611					
γ -Picoline (x) + Ethanol ($1 - x$)									
0.0000	950.07	0.8600	0.0000	0.0000	0.5813	880.40	0.9345	-0.0583	-0.4416
0.1190	938.93	0.8780	-0.0083	-0.1187	0.7893	840.70	0.9902	-0.0511	-0.3746
0.2179	928.74	0.8832	-0.0264	-0.2157	0.8494	826.59	1.0152	-0.0408	-0.2933
0.3335	915.58	0.8963	-0.0398	-0.3260	0.9031	813.22	1.0407	-0.0290	-0.2321
0.4260	903.61	0.9084	-0.0489	-0.3846	1.0000	785.14	1.0969	0.0000	0.0000
γ -Picoline (x) + Propan-1-ol ($1 - x$)									
0.0000	950.07	0.8600	0.0000	0.0000	0.7017	856.93	1.3270	-0.1381	-0.3339
0.0910	940.22	0.9024	-0.0260	-0.0877	0.7646	845.95	1.4054	-0.1319	-0.2908
0.2095	926.73	0.9381	-0.0838	-0.2039	0.8291	834.25	1.5203	-0.1059	-0.2438
0.3419	910.24	1.0073	-0.1206	-0.2934	0.8670	827.00	1.5913	-0.0913	-0.2004
0.4177	900.16	1.0715	-0.1205	-0.3325	1.0000	799.84	1.9429	0.0000	0.0000
0.5662	878.67	1.1683	-0.1550	-0.3612					
γ -Picoline (x) + Propan-2-ol ($1 - x$)									
0.0000	0.95007	0.8600	0.0000	0.0000	0.5207	0.87404	1.0485	-0.2609	-0.1658
0.0886	0.93849	0.8731	-0.0629	-0.0392	0.6685	0.84822	1.1808	-0.2722	-0.1579
0.1711	0.92725	0.8916	-0.1148	-0.0725	0.8095	0.82132	1.4048	-0.2229	-0.1094
0.2569	0.91511	0.9174	-0.1619	-0.1077	0.8831	0.80637	1.5985	-0.1586	-0.0730
0.4424	0.88686	0.9998	-0.2394	-0.1567	1.0000	0.78126	2.0766	0.0000	0.0000
γ -Picoline (x) + <i>sec</i> -butanol ($1 - x$)									
0.0000	950.07	0.8600	0.0000	0.0000	0.4574	886.20	1.1860	-0.2277	-0.0407
0.0808	938.54	0.8992	-0.0524	0.0469	0.5582	871.69	1.3277	-0.2360	-0.0583
0.1812	924.54	0.9547	-0.1131	0.0533	0.6284	861.43	1.4445	-0.2359	-0.0647
0.2378	916.73	0.9923	-0.1424	0.0412	0.7034	850.32	1.6084	-0.2184	-0.0651
0.3187	905.59	1.0473	-0.1856	0.0103	0.8528	827.69	2.0916	-0.1352	-0.0493
0.3221	905.09	1.0520	-0.1853	0.0111	0.9272	816.17	2.4459	-0.0680	-0.0302
0.3999	894.28	1.1307	-0.2066	-0.0203	1.0000	804.69	2.8571	0.0000	0.0000
γ -Picoline (x) + <i>tert</i> -Butanol ($1 - x$)									
0.0000	950.07	0.8600	0.0000	0.0000	0.6060	850.48	1.6414	-0.3389	-0.1121
0.0724	938.02	0.8901	-0.0833	0.0248	0.6484	843.41	1.7774	-0.3283	-0.1266
0.2166	914.50	0.9887	-0.2127	0.0097	0.7197	831.42	2.0849	-0.2847	-0.1459
0.2902	902.51	1.0564	-0.2662	-0.0117	0.8000	817.76	2.5375	-0.2188	-0.1579
0.3416	894.17	1.1216	-0.2898	-0.0313	0.8603	807.30	2.9587	-0.1633	-0.1487
0.5070	866.92	1.3865	-0.3468	-0.0804	1.0000	781.86	4.3719	0.0000	0.0000

following mass fraction purities: >98% for γ -picoline, >99.8% for ethanol, >99.6% for propan-1-ol, and >99.9% for propan-2-ol and *tert*-butanol. After methanol was dried carefully, its purity was >99.5%. The same procedure applied to *sec*-butanol gave a purity > 99.1%.

The mixtures were prepared by inserting weighted amounts (accuracy ± 0.1 mg) of the pure liquid through a narrow tube into airtight, stoppered bottles using a syringe. To minimize errors caused by evaporation losses, the liquid with the higher vapor pressure was filled first into the bottle. Moreover, the bottles were almost completely filled with the liquid mixture to keep the vapor space above the liquid as small as possible. To keep the mixtures dry, all operations were carried out under nitrogen atmosphere. Nitrogen was also used to push the liquid sample from the mixture bottle into the densimeter or the viscometer, respectively.

The volume of the vapor above the liquid was small enough so that changes in the liquid mixture caused by different vaporization of the components during the filling process of the densimeter or the viscometer were negligible. The relative error of the mole fraction was less than 2×10^{-5} .

The measurement of the density of the pure components and the binary mixtures has been carried out using a

vibrating tube densimeter (Anton Paar/Austria, Model DMA 602) with a thermostated bath at 298.15 K, controlled to within ± 0.05 K. The apparatus was calibrated with deionized, doubly distilled, and degassed water and dry air at atmospheric pressure. The reproducibility of the density measurement given by the manufacturers manual (Anton Paar GmbH) is $\pm 1 \times 10^{-6} \text{ g/cm}^3$. Our estimation of the accuracy of density measured with the vibrating tube densimeter is $2 \times 10^{-5} \text{ g/cm}^3$ taking into account possible systematic errors mainly caused by the calibration procedure. Details of calibration and operational procedures are described by Haraschta (1996) and Peters (1996) and resemble the procedure in previous work (Heintz et al., 1986b).

The measurement of the kinematic viscosity ν has been carried out at atmospheric pressure with a commercial Ubbelohde viscometer (Schott Geraete GmbH/Germany, capillary no. I), inserted in a thermostated bath (Schott Geraete, Model CT 1150). The viscometer was calibrated at the measuring temperature with deionized, doubly distilled, and degassed water. The flow time was measured using an electronic stopwatch having a precision of ± 0.01 s. The Hagenbach correction was carried out by subtracting a correction value y from each time interval determined for the effluence, according to

$$\nu = k(t - y) \quad (1)$$

The value of y was interpolated from the values given in the instruction manual. Five sets of readings for the flow times were taken for each pure liquid or mixture, and the arithmetic mean was taken for the calculations of the viscosity. The accuracy of the viscosity measurement was estimated to be ± 0.002 mPa s or $\pm 0.5\%$. The measured values of ν were converted to dynamic viscosity η , applying the measured density ρ according to

$$\eta = \nu \rho \quad (2)$$

Measurements of density and viscosity were carried out at exactly the same temperature. Temperature was measured with a precision thermometer (Omega DP 95) with an accuracy of ± 0.01 K.

3. Results and Discussion

The experimental density and viscosity data for the pure components are presented in Table 1 and are compared there with literature data. The agreement of the viscosity data is satisfying considering the comparatively large scattering of literature data. In the case of *sec*-butanol our results are lower than literature data.

The experimental data of the densities and viscosities for the mixtures are given in Table 2.

Values for the molar excess volume V^E are also presented in Table 2. They were calculated using the molar mass M_i of the components, mole fractions x_i , and the densities ρ_i of the pure liquids and the density of the mixture ρ_{mix} according to

$$V^E = \sum_{i=1}^2 x_i M_i (\rho_{mix}^{-1} - \rho_i^{-1}) \quad (3)$$

with $i = 1$ for the solvent γ -picoline and $i = 2$ for the alcohol. The difference of the logarithm viscosities was calculated from

$$\Delta \ln \eta = \ln \eta_{mix} - \sum_{i=1}^2 x_i \ln \eta_i \quad (4)$$

and is also presented in Table 2.

Each set of values of V^E and $\Delta \ln \eta$ given in Table 2 was fitted according to the Redlich–Kister equation

$$X^E = x_1 x_2 \sum_{p=1}^m A_p (x_1 - x_2)^p \quad (5)$$

where X^E is V^E or $\Delta \ln \eta$ and A_i are coefficients of the fitting equation. In each case the coefficients were determined by a least squares procedure.

The parameters are presented in Table 3 together with the root mean square deviation σ defined by

$$\sigma = \left[\frac{\sum_{i=1}^n (y_{\text{expt}} - y_{\text{pred}})^2}{n} \right]^{1/2} \quad (6)$$

Here, n is the number of data points y_{expt} and y_{pred} are the experimentally determined or calculated values respectively.

Results are graphically presented in Figures 1 and 2. V^E is negative over the whole range of mole fraction and increases in the order of increasing size of the alcohol molecule. In case of γ -picoline + *sec*-butanol and γ -picoline + *tert*-butanol even positive V^E values at low mole fractions

Table 3. Coefficients A_i of the Redlich–Kister Equation (Equation 6) and the Standard Deviation σ for the Binary Mixtures at 298.15 K and Atmospheric Pressure^a

function	A_0	A_1	A_2	A_3	σ
γ -Picoline + Methanol					
$V^E/(\text{m}^3 \text{mol}^{-1})$	-2.628	-0.741	0.336		0.0043
$\Delta \ln \eta$	0.666	0.335	0.272		0.0068
γ -Picoline + Ethanol					
$V^E/(\text{m}^3 \text{mol}^{-1})$	-1.684	-0.826	-0.184		0.0080
$\Delta \ln \eta$	-0.222	-0.141	0.009		0.0019
γ -Picoline + Propan-1-ol					
$V^E/(\text{m}^3 \text{mol}^{-1})$	-1.425	-0.389	-0.025		0.0035
$\Delta \ln \eta$	-0.570	-0.207	-0.143		0.0060
γ -Picoline + Propan-2-ol					
$V^E/(\text{m}^3 \text{mol}^{-1})$	-0.660	-0.183	0.101	0.080	0.0011
$\Delta \ln \eta$	-1.028	-0.502	-0.255		0.0014
γ -Picoline + <i>sec</i> -Butanol					
$V^E/(\text{m}^3 \text{mol}^{-1})$	-0.189	-0.453	0.453	-0.284	0.0023
$\Delta \ln \eta$	-0.944	-0.255	0.051		0.0034
γ -Picoline + <i>tert</i> -Butanol					
$V^E/(\text{m}^3 \text{mol}^{-1})$	-0.313	-0.632	-0.388	-0.708	0.0013
$\Delta \ln \eta$	-1.391	-0.142	0.192		0.0051

^a A_i and σ have the unit $10^{-6} \text{ m}^3 \text{ mol}^{-1}$ for V^E , and are dimensionless for $\Delta \ln \eta$.

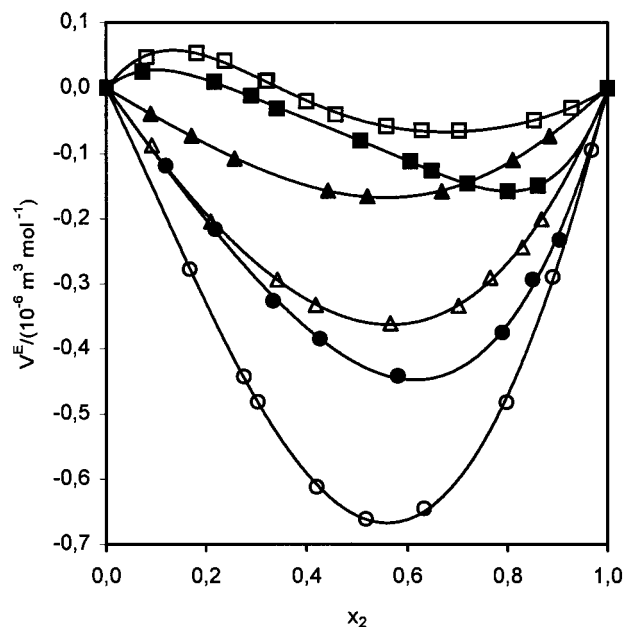


Figure 1. Molar excess volumes (V^E) of (○) γ -picoline + methanol, (●) γ -picoline + ethanol, (△) γ -picoline + propan-1-ol, (▲) γ -picoline + propan-2-ol, (□) γ -picoline + *sec*-butanol, (■) γ -picoline + *tert*-butanol at 298.15 K. x_2 is the mole fraction of alcohol. Solid lines: eq 6 with parameters from Table 3.

of the butanol molecules are observed. An interpretation of this behavior can be given using the experience made with the quantitative evaluation of alcohol + amine mixtures by the ERAS model (Bender et al., 1991; Funke et al., 1989; Heintz et al., 1998; Heintz and Papaioannou, 1998; Mohren and Heintz, 1997; Reimann and Heintz, 1991). The excess volume is determined by two main contributions. One contribution arises from free volume effects upon mixing caused by differences in the unspecific nonpolar intermolecular interactions of the two compounds. This contribution is usually small and negative. The main contribution to V^E is caused by the changes of hydrogen bondings upon mixing. The formation of a hydrogen bonding is associated with a negative reaction volume Δv^* .

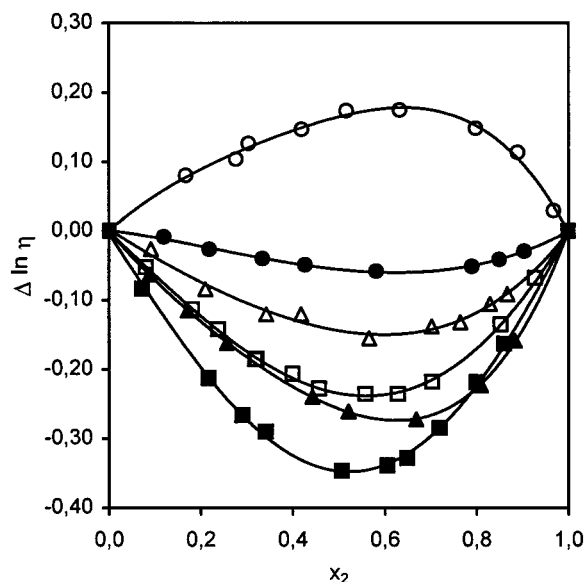


Figure 2. Differences of logarithm viscosities ($\Delta \ln \eta$) of (○) γ -picoline + methanol, (●) γ -picoline + ethanol, (△) γ -picoline + propan-1-ol, (▲) γ -picoline + propan-2-ol, (□) γ -picoline + *sec*-butanol, (■) γ -picoline + *tert*-butanol at 298.15 K. x_2 is the mole fraction of alcohol. Solid lines: eq 6 with parameters from Table 3.

Mixing alcohols with γ -picoline has the consequence that hydrogen bondings of the self-association of alcohols are broken and new hydrogen bonds $\text{OH}\cdots\text{N}$ with γ -picoline are generated. Since the bonding $\text{OH}\cdots\text{N}$ is stronger than $\text{OH}\cdots\text{O}$ and has a more negative reaction volume Δv^* the resulting excess volume reflects this balance leading to a negative contribution. This contribution depends also on the degree of cross-association between the alcohol and the γ -picoline molecule which is larger the smaller the alcohol molecule is due to steric or entropic effects which have an influence on the cross-association equilibrium constant. Also the self-association constant of the alcohol has some influence. A quantitative treatment of these effects in terms of the ERAS model will be possible as soon as experimental data of H^E and activity coefficients of the alcohol + γ -picoline mixtures are available.

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