

The GLPC column preparation has been described (2, 3). Several columns from 10 to 24 in. in length were used. The packings were Chromosorb HP 60/80 mesh, Chromosorb w HP 90/100 mesh, and 40/50 mesh Anakrom C-22 acid-washed firebrick. The Chromosorb packing showed practically no tailing for the polar solutes.

The three predominant retention mechanisms that can occur in a GLPC column are (1) partitioning of the solute between the gas and the liquid phase, (2) solute adsorption at the gas-liquid interface, and (3) solute adsorption on the surface of the packing. The last two retention mechanisms need to be eliminated in order to measure bulk solution thermodynamic properties. Solute adsorption on the surface of the packing can be avoided by using columns with at least 15-20% liquid loading on deactivated solid supports (5). Surface effects in this work are avoided by using an inactive packing, high liquid loading (20-30%), and small but finite samples and extrapolating to zero sample size.

The chemicals used are presented in Table I. Because chromatography is a separation technique, extremely pure solute samples are not required. The purity of the solvent, *n*-octane, was checked by GC. Table II is a comparison of GLPC values obtained with literature values. The GLPC results for each of the three temperatures are listed in Tables III-V. The accuracy of the activity coefficients is believed to be within 2.4%. The relative error in the *K* value was estimated to be  $\pm 1.04\%$ .

For each solute in Tables III-V,  $V_g^\infty$ ,  $K_A^\infty$ ,  $p_A^0$ ,  $\phi_A^0$ , the Poynting term,  $\phi_A$ , and  $\gamma_A^\infty$  are listed. The more directly de-

termined  $V_g^\infty$  and  $K_A^\infty$  values are given so that, if better data on the other terms become available, the activity coefficient can be readily reevaluated. The terms  $\phi_A^0$  and  $\phi_A$  are estimated by using second virial coefficients. The computer programs for the data reduction calculations are given elsewhere (3).

#### Acknowledgment

Yonghui Liu repeated the experiments and confirmed the results at 55 °C for *n*-pentane, *n*-hexane, carbon tetrachloride, and benzene.

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## Viscosity Studies of Some Binary Liquid Systems

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Viscosity and density data are presented for six binary solvent mixtures at 20 °C over the entire range of composition. The experimental data are compared with literature values and fitted to several empirical relations proposed by McAllister, Heric, and Auslander and also to a polynomial equation using a multiparametric curve-fitting program. Further, an attempt has been made to test the validity of these relations based on the experimental data.

#### Introduction

In recent years there has been renewed interest in the study of thermodynamic, hydrodynamic, and optical properties of binary liquid mixtures (1-8). Among the various hydrodynamic properties, the literature dealing with systematic studies of the viscosity of mixtures is rather limited. A number of empirical relations (9-15) have been proposed to predict the viscosity of a mixture from that of pure components. In an attempt to test the validity of these relations, we have undertaken to measure the viscosity of six solvent mixtures comprising

benzene (B), cyclohexane (CH), ethyl acetate (EA), and carbon tetrachloride (CCL). These binary data were fitted to the empirical relations of McAllister (11), Heric (15), and Auslander (13) and also to a polynomial equation using a multiparametric curve-fitting program.

#### Theories of Liquid Viscosity

Several theories have been put forth relating the viscosity of pure components to their mixtures. They are as follows.

**Eyring's Theory (9,10).** One of the earliest theories of liquid viscosity due to Eyring and co-workers may be written as

$$\eta = (hN/M) \exp(\Delta G^*/RT) \quad (1)$$

Here,  $\eta$  is viscosity,  $M$  is the molecular weight,  $T$  is the absolute temperature, and  $h$ ,  $N$ , and  $R$  are Planck's constant, Avogadro's number, and the gas constant, respectively.  $\Delta G^*$  represents the free energy of activation for viscous flow. The excess free energy of activation,  $\Delta G^{*E}$ , is given by the difference between the free energy of activation of the mixture and the free energy of activation of the ideal mixture. Thus, from eq 1

$$\Delta G^{*E} = RT(\ln \eta \bar{M} - X_1 \ln \eta_1 M_1 - X_2 \ln \eta_2 M_2) \quad (2)$$

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Table I. Ratio of Molecular Radii for Six Binary Pairs at 20 °C

solvent pair	$r_2/r_1^a$	solvent pair	$r_2/r_1^a$
B (1)-CH (2)	0.937	EA (1)-B (2)	1.033
EA (1)-CH (2)	0.967	CCL (1)-B (2)	1.028
CCL (1)-CH (2)	0.963	CCL (1)-EA (2)	0.995

$$^a r_2/r_1 = (M_1 \rho_2 / M_2 \rho_1)^{1/3}$$

Here,  $\eta$  and  $\bar{M}$  are respectively the viscosity of the mixture and the average molecular weight of the components in the mixture;  $\eta_i$ ,  $X_i$ , and  $M_i$  represent the viscosity, the mole fraction, and the molecular weight of the  $i$ th component.

The theory of Eyring et al. (9, 10) has provided a foundation for numerous correlations. However, the search for satisfactory analytical correlations applicable generally, or at least to groups of chemical binary systems, has not yet produced any answer. While this effort is continuing, some semiempirical correlations have been developed with limited success.

**McAllister's Formula (11).** On the basis of the absolute reaction rate theory of Eyring et al. (9, 10), McAllister derived the following cubic relation for the kinematic viscosity of a liquid mixture:

$$\ln \eta = X_1^3 \ln \eta_1 + 3X_1^2 X_2 \ln \eta_{12} + 3X_1 X_2^2 \ln \eta_{21} + X_2^3 \ln \eta_2 + D \quad (3)$$

where

$$D = -\ln(X_1 + X_2 M_2 / M_1) + 3X_1^2 X_2 \ln(\frac{2}{3} + M_2 / 3M_1) + 3X_1 X_2^2 \ln(\frac{1}{3} + 2M_2 / 3M_1) + X_2^3 \ln(M_2 / M_1) \quad (3a)$$

It should be noted that eq 3 is applicable only if the ratio of radii of two components in the mixture is smaller than 1.5. Also, eq 3 contains two adjustable parameters, namely,  $\ln \eta_{12}$  and  $\ln \eta_{21}$ , which could be determined by a least-squares method. The term  $D$  can be easily calculated from a knowledge of the composition of the mixture and of the molecular weights of the components. The selection of a cubic equation is justified since, for each system selected in this research, the ratio of radii as shown in Table I is smaller than 1.5.

**Auslander's Formula (13).** The less complex, three-parameter Auslander equation has the following form:

$$X_1(X_1 + B_{12}X_2)(\eta - \eta_1) + A_{21}X_2(B_{21}X_1 + X_2)(\eta - \eta_2) = 0 \quad (4)$$

Here,  $A_{21}$ ,  $B_{12}$ , and  $B_{21}$  are the parameters representing binary interactions and could be calculated from a least-squares fit of the experimental data.

**Heric's Formula (15).** Heric proposed the following relation to correlate the binary viscosity data:

$$\ln \eta = X_1 \ln \eta_1 + X_2 \ln \eta_2 + X_1 \ln M_1 + X_2 \ln M_2 - \ln(X_1 M_1 + X_2 M_2) + \Delta_{12} \quad (5)$$

where  $\Delta_{12} = \alpha_{12} X_1 X_2$  is a function representing departure from a noninteracting system, and  $\alpha_{12} = \alpha_{21}$  is the interaction parameter. Heric expressed  $\alpha_{12}$  or  $\alpha_{21}$  as a linear function of composition

$$\alpha_{12} = \beta_{12}' + \beta_{12}''(X - X_2) \quad (6)$$

The coefficients  $\beta_{12}'$  and  $\beta_{12}''$  could be determined from a

weighted least-squares method.

**Polynomial Formula.** Quite often viscosity data were also fitted to an empirical equation of the form

$$\eta = \eta_1 X_1 + \eta_2 X_2 + X_1 X_2 [a + b(X_1 - X_2) + c(X_1 - X_2)^2 + \dots] \quad (7)$$

where  $a$ ,  $b$ , and  $c$  represent the coefficients to be evaluated.

## Experimental Part

All solvents used in this work were of reagent grade and were purified by fractionating through a 5-ft column. Further purity of these samples was tested by gas chromatography. The mole percent impurity together with other physicochemical properties of pure solvents are summarized in Table II.

Solvent mixtures were prepared from 0% to 100% with an increment of 10% in each step. All of the compositions were made by weighing an appropriate volume of each solvent in the mixture. To minimize the preferential evaporation of one of the solvents in the mixture, we have selected solvent pairs in such a way that they have very close boiling points (azeotropic mixtures). In addition, while mixing the components, we have used airtight stoppered bottles. When once these mixtures were prepared, all of the measurements were made on the same day. However, keeping the mixtures overnight and re-measuring them on the next day indicated a difference of about 2-3% in both density and viscosity.

Density measurements were made by using the Anton Paar digital precision density meter, DMA 02C, made in Austria. The principle of the measurement is the variation of the frequency of a hollow oscillator when filled with liquids of different density. Since this frequency is a function of the density of the liquid, variations of the frequency (or its reciprocal, the period,  $T$ ) can be related to variations in the density of liquids. The density of an unknown liquid ( $\rho_1$ ) is measured with reference to a known standard ( $\rho_2$ ):

$$\rho_1 - \rho_2 = K(T_1^2 - T_2^2) \quad (8)$$

The instrument constant  $K$  is obtained from calibration measurements with samples of known density. The instrument constant was obtained by using air and deionized double-distilled water as known standards. The calibration was repeated for every experimental series. The instrument constant did not change significantly within the period needed to complete the density measurements. The cell compartment was maintained at  $20 \pm 0.01$  °C by a temperature controller.

Once the temperature was stabilized, the cell was washed 3 times with deionized double-distilled water followed by three aliquots of 5 mL each of dry methanol. The cell was then air-dried with the instrument pump. A sample was introduced into the cell when air gave a constant reading, which must be the same throughout the experimental series. Stabilization was considered to be achieved when at least 5-10 consecutive readings gave the same period within  $\pm 1 \times 10^{-5}$  s at a present count of  $1 \times 10^4$ . The sample was then removed, the cell dried as before followed by air reading, and another sample introduced. This procedure was repeated for all mixtures.

Viscosities of all mixtures were measured by a procedure described earlier (16), using a Fica viscomatic automatic vis-

Table II. Some Physicochemical Properties of Pure Solvents

no.	solvent	mol % impurity (obsd)	molecular wt	bp (obsd), °C	density at 20 °C, g/mL		$\eta_D$ at 20 °C	
					this work	ref 20	this work	ref 20
1	benzene	<0.02	78.115	80.2	0.8765	0.8765	1.50113	1.50110
2	cyclohexane	0.001	84.163	81.4	0.77853	0.77855	1.42668	1.42662
3	carbon tetrachloride	<0.002	153.82	76.9	1.59402	1.59404	1.46014	1.46010
4	ethyl acetate	<0.002	88.12	77.1	0.90027	0.90030	1.37220	1.37230

Table III. Density (g/mL) Data for Solvent Mixtures at 20 °C

$X_2$	solvent pair					
	B (1)-CH (2)	EA (1)-CH (2)	CCL (1)-CH (2)	EA (1)-B (2)	CCL (1)-B (2)	CCL (1)-EA (2)
0.0	0.8787	0.9007	1.5940	0.9007	1.5940	1.5940
0.1	0.8669	0.8853	1.5112	0.8981	1.5227	1.5243
0.2	0.8570	0.8706	1.4298	0.8956	1.4511	1.4549
0.3	0.8441	0.8564	1.3478	0.8931	1.3793	1.3847
0.4	0.8335	0.8428	1.2661	0.8909	1.3083	1.3164
0.5	0.8230	0.8300	1.1846	0.8886	1.2370	1.2469
0.6	0.8132	0.8180	1.1036	0.8864	1.1654	1.1776
0.7	0.8038	0.8066	1.0236	0.8844	1.0943	1.1083
0.8	0.7949	0.7960	0.9413	0.8821	1.0223	1.0395
0.9	0.7864	0.7865	0.8595	0.8807	0.9511	0.9698
1.0	0.7785	0.7785	0.7785	0.8787	0.8787	0.9007

Table IV. Viscosity (cP) Data for Solvent Mixtures at 20 °C

$X_2$	solvent pair					
	B (1)-CH (2)	EA (1)-CH (2)	CCL (1)-CH (2)	EA (1)-B (2)	CCL (1)-B (2)	CCL (1)-EA (2)
0.0	0.6565	0.4508	0.9785	0.4508	0.9785	0.9785
0.1	0.6460	0.4632	0.9700	0.4656	0.9426	0.8830
0.2	0.6382	0.4703	0.9648	0.4754	0.9050	0.8095
0.3	0.6374	0.4820	0.9540	0.4862	0.8721	0.7523
0.4	0.6442	0.5033	0.9465	0.5003	0.8399	0.6968
0.5	0.6602	0.5303	0.9527	0.5170	0.8082	0.6500
0.6	0.6864	0.5673	0.9421	0.5394	0.7747	0.6061
0.7	0.7243	0.6204	0.9458	0.5616	0.7439	0.5667
0.8	0.7810	0.6921	0.9637	0.5876	0.7246	0.5276
0.9	0.8664	0.8020	0.9643	0.6179	0.6833	0.4881
1.0	0.9751	0.9751	0.9751	0.6565	0.6565	0.4508

cometer whose temperature was thermostatically controlled to within  $\pm 0.001$  °C. Viscosities were calculated from flow times which were measured in seconds for all of the systems. Flow times for water as well as for pure solvents were quite consistent during the entire period of measurement. Three independent sets of data were obtained for both density and viscosity for each composition of the mixture; the average values of these are presented in Tables III-IV. All measurements were made at 20 °C. In addition to these measurements, both density and viscosity were also measured by using a conventional pycnometer and a U-tube viscometer, respectively, at 20 °C. The data agreed within 1% for all of the mixtures as compared to automatic densitometer as well as viscometer results. However, the reported data are from the latter techniques. The reproducibility of a triplicate set of data was within  $\pm 0.2\%$  for both density and viscosity for all mixtures.

## Results and Discussion

The experimental data of viscosities are plotted in Figure 1 as a function of composition of the mixture for CCL (1)-CH (2), B (1)-CH (2), and EA (1)-CH (2) systems. Since CCL (1)-CH (2) mixtures behave like an ideal mixture, there is a slightly downward curvature from straight-line behavior. However, for the remaining two nonideal mixtures, namely, B (1)-CH (2) and EA (1)-CH (2), the curves for the dependence of viscosity on mixture composition deviate greatly from the straight line. A

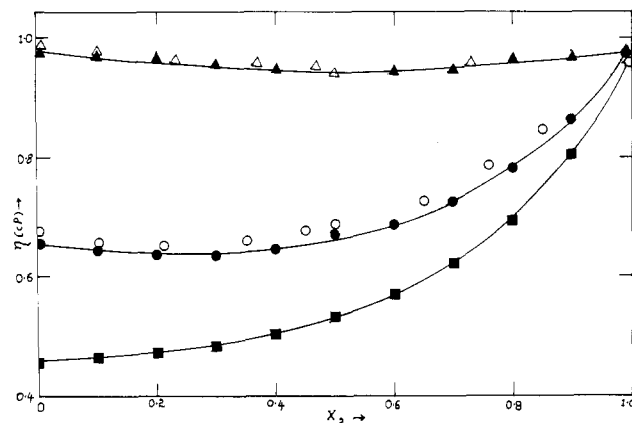


Figure 1. Dependence of viscosity (centipoise) of solvent mixtures on composition at 20 °C. Solid symbols are for the present data, and open symbols are for data from the literature: (●) B (1)-CH (2); (■) EA (1)-CH (2); (▲) CCL (1)-CH (2); (○) B (1)-CH (2) from ref 17 and 18; (△) CCL (1)-CH (2) from ref 17 and 18.

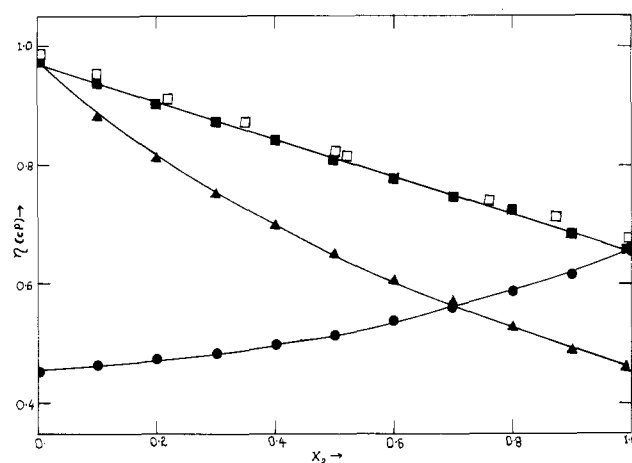


Figure 2. Dependence of viscosity (centipoise) of solvent mixtures on composition at 20 °C. Solid symbols are for the present data, and open symbols are for data from the literature: (●) EA (1)-B (2); (■) CCL (1)-B (2); (▲) CCL (1)-EA (2); (□) CCL (1)-B (2) from ref 17 and 18.

similar trend was also seen in our study (3) for the diffusion coefficients of these mixtures. Katti and Chaudhari (17) have remeasured at 25 °C the viscosities of CCL (1)-CH (2) and B (1)-CH (2) mixtures as obtained earlier by Grunberg (18). When these data were corrected to 20 °C and plotted in Figure 1 along with the present data, the agreement for both mixtures was within 3% for all compositions. For the EA (1)-CH (2) system, no literature data are available for direct comparison at this or a comparable temperature.

In Figure 2 we have presented the viscosity data for the remaining three liquid mixtures, namely, CCL (1)-B (2), CCL (1)-EA (2), and EA (1)-B (2). Here again, the data for CCL (1)-B (2) are compared with those of Katti et al. (17), and the agreement is very good. Similarly, in an extensive study, Heric

Table V. Computer Analysis of Parameters of Eq 3-7

system	McAllister formula <sup>a</sup>		Auslander formula <sup>b</sup>			Heric formula <sup>c</sup>		polynomial relation <sup>d</sup>		
	$\eta_{12}$	$\eta_{21}$	$A_{21}$	$B_{21}$	$B_{12}$	$B_{12}^I$	$B_{12}^{II}$	$a$	$b$	$c$
B (1)-CH (2)	0.6138	0.6234	0.0573	0.0000	0.1372	-0.7660	1.7000	-0.6256	0.2434	-0.0847
EA (1)-CH (2)	0.4933	0.4854	0.1494	0.9395	0.6137	-0.9180	0.4290	-0.7301	0.4770	-0.2559
CCL (1)-CH (2)	0.9613	0.9554	1809.4	1.4599	0.0796	0.0790	0.0190	-0.1288	0.0337	0.1089
EA (1)-B (2)	0.4900	0.5328	0.2250	2.2167	0.4612	-0.176	0.0920	-0.1437	0.0554	0.0164
CCL (1)-B (2)	0.8682	0.7952	0.6179	1.9231	0.5991	0.261	-0.0690	-0.0414	-0.0235	0.0189
CCL (1)-EA (2)	0.6960	0.6175	1.2628	1.5736	0.9599	0.0561	-0.2140	-0.2562	-0.1715	-0.0896

<sup>a</sup> Equation 3. <sup>b</sup> Equation 4. <sup>c</sup> Equation 5. <sup>d</sup> Equation 7.

Table VI. Comparison of Experimental and Calculated Values of Viscosity of Ethyl Acetate (1)-Benzene (2) Mixtures at 20 °C

$X_2$	$\eta(\text{obsd})$ , cP	$\eta(\text{calcd})$ , cP			
		McAllister	poly- nomial	Auslander	Heric
0.0	0.4508	0.4508	0.4508	0.4508	0.4508
0.1	0.4656	0.4622	0.4634	0.4616	0.4635
0.2	0.4754	0.4742	0.4752	0.4736	0.4762
0.3	0.4862	0.4874	0.4876	0.4869	0.4894
0.4	0.5003	0.5020	0.5014	0.5019	0.5037
0.5	0.5170	0.5187	0.5177	0.5189	0.5197
0.6	0.5394	0.5380	0.5372	0.5384	0.5382
0.7	0.5616	0.5605	0.5605	0.5608	0.5600
0.8	0.5876	0.5871	0.5880	0.5872	0.5862
0.9	0.6179	0.6187	0.6199	0.6185	0.6179
1.0	0.6565	0.6565	0.6565	0.6565	0.6565

et al. (19) measured the viscosity data for this mixture at 25 °C, and their data (after suitable temperature correction and conversion to centipoise) appear to agree within 4% of our measurement. However, to avoid overcrowding of points, we do not show these points in Figure 2. An almost linear dependence of viscosity with composition (see Figure 2) was observed for the CCL (1)-B (2) system. On the other hand, nonlinearities were observed for the CCL (1)-EA (2) and EA (1)-B (2) systems. To our knowledge, no data on viscosities at this temperature have been studied for the latter two mixtures so that a direct comparison is not possible. However, on the basis of the comparison for our other three mixtures (where data were available from the literature), we believe that the data for mixtures containing ethyl acetate are accurate, with the scatter of points from the smooth line being less than 1%.

The density data for all six mixtures studied here have been treated rigorously in an earlier study (7). Consequently, no data treatment was attempted in this work, but the actual values are presented in Table III.

In the theoretical analysis of the data, eq 3-7 were fitted to viscosity data by using an initial estimate of the unknown parameters. The multiparametric curve-fitting program uses a nonlinear least-squares analysis in conjunction with the method of steepest descent to simultaneously vary the unknown parameters until the best fit is achieved. Using this program, we evaluated various parameters of the different relations, i.e., eq 3-7, and the results of the computer analysis are summarized in Table V for all of the systems.

From this analysis it is evident that all of the relations are, in fact, very effective in fitting the binary data. This is not surprising in view of the number of adjustable parameters in each expression and the relatively gentle variation of the data with composition. It is therefore difficult to make any general statements regarding the superiority of one relation over the other and their relative validity. However, the theoretical treatment of our data showed that the McAllister formula and the polynomial relation gave a better fit of the experimental points to well within the experimental uncertainty. The agree-

ment of the computed and experimental data was found to be within about 1% by these relations (see, for instance, Table VI for the ethyl acetate (1)-benzene (2) system). The other two formulas, namely, Auslander's and Heric's relations, gave a somewhat larger percentage error of the calculated and experimental points for the mixtures reported here.

## Conclusions

Both density and viscosity data were measured for six binary solvent mixtures. Viscosity data were compared with literature values for some of the mixtures, and the agreement was very good. The binary data were further fitted to several theoretical and empirical relations. The validity of these relations was tested on the basis of the experimental data. Accurate data on additional binary systems are needed to arrive at a quantitative understanding of the theories of liquid viscosity.

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