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Volumetric Properties and Viscosities of Binary Mixtures of *N*,*N*-Dimethylformamide with Methanol and Ethanol in the Temperature Range (293.15 to 333.15) K

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Densities and viscosities of the binary mixtures of methanol + DMF and ethanol + DMF have been measured over several temperatures at atmospheric pressure. The measurements were carried out over the whole range of compositions, using a vibrating-tube density meter and an Ubbelohde viscometer. The densities are used to calculate excess molar volumes for each of the systems. The experimental values of excess molar volumes have been compared with those reported by other authors available in the literature. The Redlich–Kister equation was fitted to excess molar volumes, $V^{\rm E}$, and viscosities, η .

Introduction

N,*N*-Dimethylformamide (DMF) has a large dipole moment and a high dielectric constant;¹ therefore, it may work as an aprotic protophilic solvent used in the synthesis of pharmaceuticals, in agricultural chemistry, and as a solvent for polymers. Alkanols are polar liquids, strongly self-associated by hydrogen bonding. From the liquid theoretical viewpoint, it will be interesting to explore the thermodynamic results and transport properties of the addition of the amide carbonyl group mixtures with alcohols to obtain some information on the molecular interactions.

A survey of the literature shows that very few measurements have been made on the densities and viscosities for these binary mixtures. Bai et al.^{2,3} reported excess molar volumes for binary and ternary mixtures of (*N*,*N*-dimethylformamide + methanol + water) at 298.15 K, as well as of (*N*,*N*-dimethylformamide + ethanol + water) at the temperature 298.15 K. Zielkiewicz⁴ reported the excess molar volumes in (*N*,*N*-dimethylformamide + methanol + water) at 313.15 K. Iloukhani and Rostami⁵ gave the data of excess molar volumes for *N*,*N*-dimethylformamide with 1-alkanol at 303.15 K. However, there are no reports on excess molar volumes for binary mixtures of methanol + DMF and ethanol + DMF at other temperatures, and especially no viscosity data on these mixtures were previously reported in the literature.

In this work, the densities and viscosities for binary mixtures methanol + DMF and ethanol + DMF were measured at (293.15 to 333.15) K and atmospheric pressure. The results were used to calculate excess molar volumes. The Redlich–Kister equation was fitted to experimental data.

Experimental Section

Materials. The DMF, methanol, and ethanol were purchased from Tianjin Chemical Co. All chemicals are analytical reagent grade. Prior to use, they were stored over molecular sieves type 4 Å to remove traces of water. Before measurements, pure liquids were degassed ultrasonically. The mass fraction purities

Table 1.	Comparison of Experimental and Literature	Values of
Densities,	ρ , and Viscosities, η , for Pure Compounds	

		$\rho/g \cdot cm^{-3}$		η/m•Pas	
liquid	T/K	exptl	lit.	exptl	lit.
DMF	298.15	0.94421	0.9445 ⁶	0.808	0.8036
			0.9442^{7}		0.799^{7}
			0.94398		0.801^{8}
			0.94387^9		0.802^{9}
	303.15	0.93945	0.9395 ⁸	0.760	0.756^{6}
			0.9398^{6}		0.754^{8}
					0.752^{10}
	313.15	0.92986	0.9302^{6}	0.675	0.673^{6}
			0.9298^{11}		0.664^{10}
methanol	293.15	0.79151	0.79154^{12}	0.587	0.582^{12}
	298.15	0.78682	0.7866^{13}	0.547	0.549^{13}
			0.78663^{15}		
			0.7881^{14}		0.5482^{14}
	303.15	0.78206	0.78191^{16}	0.510	0.512^{17}
			0.781915		0.504^{15}
	313.15	0.77272	0.7725^{15}	0.447	0.448^{12}
			0.77260^{12}		0.44717
	323.15	0.76322	0.762715	0.394	0.400^{15}
ethanol	293.15	0.78985	0.7882417	1.222	1.19217
	298.15	0.78562	0.785313	1.109	1.077^{13}
			0.7852515		1.0915
			15		1.112^{14}
	303.15	0.78131	0.780815	1.009	0.98619
			0.7810^{18}		19
	313.15	0.77256	0.772618	0.840	0.82318

tested by GLC were as follows: DMF (> 0.998), methanol (> 0.994), ethanol (> 0.995). The purity of the solvents was also ascertained by comparing their densities and viscosities with the corresponding literature values in Table 1. Our results obtained are in good agreement with those listed in the literature.

Apparatus and Procedure. The densities of the pure components and their mixtures were measured with a high-precision vibrating-tube digital density meter (Density/Specific Gravity Meter DA 505, KEM, Japan) whose measurement cell temperature was controlled automatically within \pm 0.01 K of the selected value. The uncertainty in density measurements was \pm 5 · 10⁻⁵ g · cm⁻³. Density measurements were reproducible to \pm 3 · 10⁻⁵ g · cm⁻³.

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Table 2. Densities (ρ), Viscosities (η), and Excess Molar Volumes (V^{E}) for the Binary Mixtures at Different Temper-
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	ρ	η	V ^E		ρ	η	
x_1	g·cm ⁻³	mPa•s	$\overline{\text{cm}^3 \cdot \text{mol}^{-1}}$	x_1	g•cm ⁻³	mPa•s	cm ³ ·mol ⁻
1	8		(r) Methanol	+(1-r) DMF	6		
			(x_1) with the first of (x_1)	$(1 x_1)$ Divis			
0000	0.04005	0.072	T = 2	93.15 K	0.00(50	0.602	0.4070
0.0000	0.94895	0.863	0.0000	0.6000	0.88658	0.693	-0.4368
).0998	0.94181	0.835	-0.1201	0.6997	0.86936	0.665	-0.4220
).2000	0.93368	0.809	-0.2248	0.8000	0.84869	0.640	-0.3618
0.3000	0.92438	0.782	-0.3123	0.8998	0.82322	0.613	-0.2271
0.4002	0.91369	0.754	-0.3816	1.0000	0.79151	0.587	0.0000
0.5000	0.90123	0.723	-0.4249				
	0.00120	01720	011 <u>2</u> 1)	00.15 17			
			T=2	98.15 K			
0.0000	0.94421	0.808	0.0000	0.6000	0.88175	0.649	-0.4368
0.0998	0.93703	0.782	-0.1186	0.6997	0.86456	0.622	-0.4237
0.2000	0.92889	0.758	-0.2241	0.8000	0.84391	0.596	-0.3635
).3000	0.91958	0.734	-0.3121	0.8998	0.81849	0.572	-0.2292
0.4002	0.90889	0.705	-0.3826	1.0000	0.78682	0.547	0.0000
0.1002	0.89640	0.678	-0.4245	1.0000	0.70002	0.017	0.0000
	0.07040	0.070	0.4245				
			T = 3	03.15 K			
0.0000	0.93945	0.760	0.0000	0.6000	0.87684	0.608	-0.4346
).0998	0.93215	0.736	-0.1109	0.6997	0.85966	0.584	-0.4224
2000	0 92401	0.713	-0.2185	0.8000	0.83902	0 559	-0.3623
3000	0.01/70	0.688	-0.3084	0.8008	0.81364	0.53/	-0.222
2.3000	0.91470	0.000	0.3004	1.0000	0.01304	0.534	0.2283
J.4002	0.90402	0.003	-0.3812	1.0000	0.78206	0.510	0.0000
).5000	0.89148	0.636	-0.4209				
			T = 3	13.15 K			
0000	0.02086	0.675	0.0000	0.6000	0 86720	0.540	-0.4441
2.0000	0.92980	0.075	0.0000	0.0000	0.80729	0.540	0.4441
0.0998	0.92272	0.655	-0.1263	0.6997	0.85011	0.517	-0.4301
0.2000	0.91456	0.635	-0.2339	0.8000	0.82951	0.493	-0.3689
0.3000	0.90521	0.612	-0.3221	0.8998	0.80420	0.470	-0.2327
0.4002	0.89453	0.589	-0.3956	1.0000	0.77272	0.447	0.0000
0.5000	0.88196	0.565	-0.4333				
			<i>т</i> о	00 15 IZ			
			T = 3	23.15 K			
0.0000	0.92092	0.608	0.0000	0.6000	0.85757	0.483	-0.4257
0.0998	0.91320	0.588	-0.0852	0.6997	0.84042	0.460	-0.4187
0.2000	0.90501	0.569	-0.1990	0.8000	0.81982	0.438	-0.3609
0.3000	0.89561	0.549	-0.2914	0.8998	0.79457	0.415	-0.2288
0 4002	0 88491	0.528	-0.3712	1.0000	0.76322	0 394	0.0000
0.5000	0.87228	0.506	-0.4115	110000	0110022	01071	010000
0.0000	0.07220	0.500	0.1115				
			(x_1) Ethanol	$+ (1 - x_1) \text{ DMF}$			
			T = 2	93.15 K			
0000	0.94895	0.863	0.0000	0.6000	0 86707	0.886	-0.2210
0000	0.02010	0.005	0.0000	0.0000	0.85004	0.000	0.2217
0.0999	0.93612	0.071	-0.0983	0.7000	0.03004	0.919	-0.2017
0.2000	0.92578	0.876	-0.1624	0.8000	0.83144	0.970	-0.1568
0.3000	0.91289	0.882	-0.1972	0.9001	0.81142	1.067	-0.0902
0.4000	0.89861	0.881	-0.2203	1.0000	0.78985	1.222	0.0000
0.5001	0.88309	0.872	-0.2309				
			T - 2	08 15 K			
0000	0.04421	0 000	1 - 2	0 2000	0.96240	0.025	0.0175
0.0000	0.94421	0.808	0.0000	0.0000	0.80249	0.823	-0.21/5
1.0999	0.93340	0.819	-0.0956	0.7000	0.84549	0.852	-0.1946
).2000	0.92106	0.818	-0.1566	0.8000	0.82696	0.897	-0.1500
0.3000	0.90823	0.822	-0.1941	0.9001	0.80705	0.977	-0.0860
0.4000	0.89396	0.821	-0.2164	1.0000	0.78562	1.109	0.0000
0.5001	0.87846	0.813	-0.2251				
			<i>T</i> . 0	00.15.17			
			T=3	03.15 K			
0.0000	0.93945	0.760	0.0000	0.6000	0.85782	0.769	-0.2074
0.0999	0.92864	0.770	-0.0885	0.7000	0.84091	0.791	-0.1897
0.2000	0.91632	0.767	-0.1524	0.8000	0.82244	0.828	-0.1452
0.3000	0.90351	0.770	-0.1876	0.9001	0.80260	0.897	-0.0813
0.4000	0.88923	0.767	-0.2080	1.0000	0.78131	1.009	0.0000
0 5001	0.87376	0.759	-0.2184	1.0000	0.70101	1.007	0.0000
	0.07570	0.137	0.2107				
			T = 3	13.15 K			
0.0000	0.92986	0.675	0.0000	0.6000	0.84842	0.670	-0.1971
0.0999	0.91906	0.682	-0.0825	0.7000	0.83160	0.685	-0 1766
2000	0 90676	0.680	-0.1413	0.8000	0.81328	0.712	-0.1350
3000	0.90070	0.000	_0.1754	0.0001	0.70264	0.762	_0.1550
3.3000	0.09393	0.001	-0.1/30	1.0000	0.79304	0.702	-0.0765
J.4000	0.8/9/1	0.6/4	-0.19/5	1.0000	0.77256	0.840	0.0000
0.5001	0.86429	0.668	-0.2069				
			T - 2	23 15 K			
0000	0.00000	0.000	I = 3	23.13 K	0.02000	0.500	0.1003
0000	0.92092	0.608	0.0000	0.6000	0.83898	0.590	-0.1883
0.0999	0.90948	0.610	-0.0754	0.7000	0.82226	0.598	-0.1675

			- F				- F		
	ρ	η	V^{\perp}		ρ	η	V^{\perp}		
x_1	g•cm ⁻³	mPa•s	$\overline{\text{cm}^3 \cdot \text{mol}^{-1}}$	<i>x</i> ₁	g•cm ⁻³	mPa•s	$\overline{\text{cm}^3 \cdot \text{mol}^{-1}}$		
T = 323.15 K									
0.2000	0.89718	0.604	-0.1276	0.8000	0.80404	0.616	-0.1236		
0.3000	0.88439	0.603	-0.1620	0.9001	0.78450	0.651	-0.0688		
0.4000	0.87017	0.596	-0.1860	1.0000	0.76367	0.705	0.0000		
0.5001	0.85481	0.592	-0.1944						
			T = 33	33.15 K					
0.0000	0.91064	0.549	0.0000	0.6000	0.82938	0.522	-0.1774		
0.0999	0.89980	0.547	-0.0676	0.7000	0.81271	0.527	-0.1576		
0.2000	0.88749	0.543	-0.1163	0.8000	0.79455	0.537	-0.1148		
0.3000	0.87473	0.540	-0.1507	0.9001	0.77520	0.559	-0.0631		
0.4000	0.86051	0.533	-0.1772	1.0000	0.75453	0.597	0.0000		
0.5001	0.84516	0.524	-0.1813						

 Table 3. Coefficients of the Redlich-Kister Equation and Standard

 Deviation for Excess Molar Volumes and Viscosity Deviations of

 Mixtures

$T(\mathbf{K})$	property	A_0	A_1	A_2	A_3	σ		
(x_1) Methanol + $(1-x_1)$ DMF								
293.15	$V^{\rm E}/{\rm cm}^3 \cdot {\rm mol}^{-1}$	-1.694	-0.625	-0.373	-0.183	0.0119		
	$\Delta \eta$ /mPa·s	-0.006	-0.050	-0.009	0.066	0.0040		
298.15	$V^{E}/cm^{3} \cdot mol^{-1}$	-1.695	-0.627	-0.378	-0.228	0.0113		
	$\Delta \eta$ /mPa·s	0.001	-0.055	-0.013	0.082	0.0040		
303.15	$V^{E}/cm^{3} \cdot mol^{-1}$	-1.689	-0.613	-0.317	-0.327	0.0146		
	$\Delta \eta$ /mPa·s	0.004	-0.036	0.000	0.041	0.0044		
313.15	$V^{E}/cm^{3} \cdot mol^{-1}$	-1.730	-0.606	-0.416	-0.226	0.0180		
	$\Delta \eta$ /mPa·s	0.016	-0.028	0.004	0.015	0.0024		
323.15	$V^{E}/cm^{3} \cdot mol^{-1}$	-1.663	-0.638	-0.155	-0.562	0.0287		
	$\Delta \eta$ /mPa•s	0.020	-0.020	-0.012	0.012	0.0022		
(x_1) Ethanol + $(1-x_1)$ DMF								
293.15	$V^{\rm E}/{\rm cm}^3 \cdot {\rm mol}^{-1}$	-0.917	-0.032	-0.207	0.142	0.0082		
	$\Delta \eta$ /mPa·s	-0.0657	-0.635	-0.238		0.0223		
298.15	$V^{E}/cm^{3} \cdot mol^{-1}$	-0.898	-0.018	-0.173	0.134	0.0030		
	$\Delta \eta$ /mPa·s	-0.562	-0.557	-0.177		0.0192		
303.15	$V^{\vec{E}}/\text{cm}^3 \cdot \text{mol}^{-1}$	-0.872	-0.009	-0.125	0.096	0.0145		
	$\Delta \eta$ /mPa·s	-0.487	-0.485	-0.124		0.018		
313.15	$V^{\vec{E}}/\text{cm}^3 \cdot \text{mol}^{-1}$	-0.823	-0.003	-0.100	0.074	0.0073		
	$\Delta \eta$ /mPa·s	-0.353	-0.358	-0.077		0.0082		
323.15	$V^{\vec{E}}/\text{cm}^3 \cdot \text{mol}^{-1}$	-0.777	-0.037	-0.035	0.134	0.0069		
	$\Delta \eta$ /mPa·s	-0.261	-0.251	-0.050		0.0084		
333.15	$V^{\dot{E}}/cm^3 \cdot mol^{-1}$	-0.734	-0.039	0.015	0.112	0.0098		
	$\Delta \eta$ /mPa•s	-0.189	-0.188	-0.035		0.0071		

The liquid mixtures were prepared by weight using a BP210s balance accurate to within ± 0.1 mg. The average uncertainty in the compositions (mole fraction) of the mixtures was estimated to be less than ± 0.0001 . The molar excess volumes



Figure 1. Fractional deviations $\Delta V^{\text{E}} = V^{\text{E}}_{\text{exptl}} - V^{\text{E}}_{\text{calcd}}$ of the experimental excess volumes from values $V^{\text{E}}_{\text{calcd}}$ obtained with the correlation of the Redlich–Kister equation for the system methanol (1) + DMF (2) at different mole fractions, x_1 , and different temperatures T: , 293.15 K; •, 298.15 K; , 303.15 K; , 313.15 K; solid triangle pointing left, 323.15 K.



Figure 2. Fractional deviations $\Delta V^{\text{E}} = V^{\text{E}}_{\text{exptl}} - V^{\text{E}}_{\text{calcd}}$ of the experimental excess volumes from values $V^{\text{E}}_{\text{calcd}}$ obtained with the correlation of the Redlich–Kister equation for the system methanol (1) + DMF (2) at different mole fractions, x_1 , and different temperatures T: \blacksquare , 293.15 K; •, 298.15 K; \blacktriangle , 303.15 K; \blacktriangledown , 313.15 K; solid triangle pointing left, 323.15 K; solid triangle pointing right, 333.15 K.



Figure 3. Fractional deviations $\eta = \eta_{exptl} - \eta_{calcd}$ of the experimental viscosities from values η_{calcd} obtained with the correlation of eq 3 for the system methanol (1) + DMF (2) at different mole fractions, x_1 , and different temperatures T: **...**, 293.15 K; •, 298.15 K; **...**, 303.15 K; **...**, 313.15 K; solid triangle left, 323.15 K.

were calculated from composition-density data with an uncertainty better than $\pm 0.002 \text{ cm}^3 \cdot \text{mol}^{-1}$. All molar quantities were based on the IUPAC relative atomic mass table.

The viscosities of pure liquids and the mixtures were measured at atmospheric pressure and at different temperatures using an Ubbelohde suspended-level viscometer which was calibrated with doubly distilled water. The uncertainty of viscosity results was within ± 0.003 mPa·s.



Figure 4. Fractional deviations $\eta = \eta_{exptl} - \eta_{calcd}$ of the experimental viscosities from values η_{calcd} obtained with the correlation of eq 3 for the system ethanol (1) + DMF (2) at different mole fractions, x_1 , and different temperatures $T: \blacksquare$, 293.15 K; \bullet , 298.15 K; \blacktriangle , 303.15 K; \blacktriangledown , 313.15 K; solid triangle pointing left, 323.15 K; solid triangle pointing right, 333.15 K.

The density and viscosity values and those from the literature for all the pure compounds are listed in Table 1. The details of the methods and techniques used to determine densities and viscosities have been described previously.^{20,21}

Result and Discussion

Excess volumes and viscosity deviations were calculated from our measurements according to the following equations:²⁰

$$V^{\rm E} = \frac{x_1 M_1 + x_2 M_2}{\rho} - \frac{x_1 M_1}{\rho_1} - \frac{x_2 M_2}{\rho_2} \tag{1}$$

where x_1 and x_2 are mole fractions; M_1 and M_2 are the molar masses; and ρ_1 and ρ_2 are the densities of pure components 1 and 2, respectively. Quantities without subscripts refer to the mixture.

The Redlich-Kister equation²¹ is fitted to the experimental excess volumes, V^{E} , for each binary mixture.

$$V^{\rm E} = x_1(1-x_1)\sum_{i=0}^n A_i(2x_1-1)^i$$
(2)

where A_i are adjustable parameters. The viscosities are correlated with the equation

$$\eta = x_1 \eta_1 + (1 - x_1) \eta_2 + x_1 (1 - x_1) \sum_{i=0}^n A_i (2x_1 - 1)^i \quad (3)$$

where η is the viscosity of mixtures and η_1 and η_2 are the viscosities of components 1 and 2, respectively.

In each case, the optimum number of coefficients A_i was determined from an examination of the variation of the standard derivation

$$\sigma(Y) = \left[\sum \left(Y_{\text{calcd}} - Y_{\text{exptl}}\right)^2 / (n-m)\right]^{\frac{1}{2}}$$
(4)

where n is the total number of experimental values and m is the number of parameters.

The densities, viscosities, and excess molar volume data are presented in Table 2. Table 3 lists the values of the parameters A_i together with the standard deviations.

The relative deviations of the excess molar volumes and viscosity were calculated by the equation

$$RD\% = \frac{(y_{exptl} - y_{calcd}) \cdot 100}{y_{exptl}}$$
(5)

where $y = V^{E}$ or η ; y_{expt1} are the experimental values; and y_{calcd} are calculated values obtained with eq 1 and eq 3, respectively. The average absolute deviations were calculated by

$$AAD\% = \frac{\sum |(y_{exptl} - y_{calcd}) \cdot 100/y_{exptl}|}{n} = \frac{\sum |\Delta y|}{n} \quad (6)$$

The relative deviation of the excess molar volumes and viscosity at different mole fraction and temperatures for systems of methanol and ethanol with DMF are shown in Figure 1 to Figure 4, respectively. It can be seen from the figures that the relative deviation values of the excess molar volumes for these two binary systems are within ± 2.5 %, and the values for viscosities are within ± 1.0 %.

The $V^{\rm E}$ values for mixtures of methanol + DMF and ethanol + DMF obtained in this work compare with those reported by Wang et al.^{2,3} and Zielkiewicz⁴ for these mixtures. For the system of methanol with DMF, the absolute average deviation of the experimental excess molar volumes from values obtained with the correlation of the Redlich–Kister equation given by Wang et al. and Zielkiewicz under 9 mole fractions at 293.15 K is 12.6 % and 2.1 %, respectively. For systems of ethanol with DMF, the absolute average deviation between experimental values with the values given by Wang et al.³ at 313.15 K is 20.5 %. Our results are different from those of Iloukhani and Rostami⁵ which are positive at 313.15 K.

From Table 2, it can be seen that V^{E} values are negative for these two binary mixtures over the entire range of compositions. The viscosities decrease with mole fraction of DMF.

Conclusions

Densities and viscosities of the binary mixtures of methanol + DMF and ethanol + DMF have been experimentally measured over several temperatures at atmospheric pressure. The excess molar volume and viscosity were correlated using the Redlich–Kister polynomial equation, and the coefficients and the standard deviation values are given. The excess molar volumes for these two binary systems were negative over the whole composition range and at all temperatures.

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