

Excess Molar Enthalpies and Excess Molar Volumes for Mixtures of 1,3-Dimethyl-2-imidazolidinone and an Alkanol at $T = 298.15$ K

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Excess molar enthalpies, H_m^E , and excess molar volumes, V_m^E , of the mixtures {(1,3-dimethyl-2-imidazolidinone (DMI) + methanol, ethanol, 1-propanol, 2-propanol, or 1-butanol)} for the whole range of compositions have been measured at $T = 298.15$ K. The excess molar enthalpy values ranged from a minimum of -410 J mol $^{-1}$ for the mixture (DMI + methanol) to a maximum of $+650$ J mol $^{-1}$ for the mixture (DMI + 2-propanol). The excess molar volumes are negative for all the mixtures studied and ranged from -0.83 cm 3 mol $^{-1}$ to -0.20 cm 3 mol $^{-1}$. The Redlich–Kister polynomial was used to correlate both the excess molar enthalpy and excess molar volume data. The NRTL and UNIQUAC models were used to correlate the enthalpy of mixing data. Both the NRTL and UNIQUAC models correlate the data equally well. The results are discussed in terms of the ease of hydrogen bond formation between the proton of the alkanol and the O atom of 1,3-dimethyl-2-imidazolidinone and are compared with related systems found in the literature.

Introduction

Recently, Živný et al.¹ and Awwad et al.² published excess molar enthalpy and volume measurements, respectively, on the mixture [(*N*-methylpyrrolidone (NMP) + methanol)]. The compound NMP is a cyclic amide (a cyclic urea compound) and is an ideal substitute solvent for the carcinogenic hexamethylphosphoric triamide,³ which is used in the paper and electrochemical industries. It is an important solvent in extractive distillation and its solvent extractive properties have been investigated.⁴ These cyclic amides form an interesting group of compounds as they are important in the preparation of polyamides,¹ peptides, polypeptides, and other life chemicals. In this work a related compound, 1,3-dimethyl-2-imidazolidinone (DMI), an *N,N*-dialkylated cyclic amide (a cyclic urea), was investigated and the excess molar enthalpies and the excess molar volumes for (DMI + an alkanol) were measured at 298.15 K and 1 atm of pressure. The alkanols were methanol, ethanol, 1-propanol, 2-propanol, or 1-butanol.

The experimental results were interpreted in terms of hydrogen bond formation or disruption between the proton of the alkanol and the O atom of the carbonyl of the DMI molecule. The Redlich–Kister polynomial⁵ was used to correlate the excess molar enthalpy and excess molar volume data and the NRTL⁶ and UNIQUAC⁷ models were used to correlate the excess molar enthalpy data.

Experimental Section

The chemicals that were used, their suppliers, and their purity are listed in Table 1. The compounds methanol and ethanol were dried with calcium hydride, and 1-propanol, 2-propanol, and 1-butanol were dried with anhydrous potassium carbonate. All the alkanols were stored under 4-Å molecular sieves. The purity of the alkanols was determined by chromatography and found to be greater than 99 mol %. The amount of water present in the alkanols was determined by Karl–Fischer titration and was less than 0.1%. DMI was obtained from Acros and had a purity

greater than 98 mol %. DMI was not purified further because attempts to purify DMI by distillation resulted in the discarding of large quantities of DMI to produce very small amounts of pure compound. As DMI is an expensive solvent, purification was abandoned and the solvent was used as supplied. The liquids were each degassed before use.

A Thermometric flow microcalorimeter 2277, driven by two LKB peristaltic pumps, was used to determine the excess molar enthalpies, H_m^E , at a constant temperature (298.15 K). The calibration of the instrument and the experimental procedure was detailed previously.^{8,9} The excess molar volumes, V_m^E , were calculated from measured densities of the pure liquids and the mixtures using an Anton Paar DMA 601 vibrating tube density meter thermostated at the temperature (298.15 \pm 0.01) K as previously reported.¹⁰

The performance of the calorimeter was checked by measuring H_m^E over the whole composition range for the test mixture (cyclohexane + hexane). The maximum difference between our results and the literature results¹¹ was less than 1.5% at $x = 0.5$. The estimated precision for the excess molar enthalpy measurements is 0.4 J mol $^{-1}$.

Dry air and Milli-Q water were used to calibrate the densitometer. The estimated precision for the excess volume measurements is 0.005 cm 3 mol $^{-1}$.

Results

The experimental H_m^E results are given in Table 2 and plotted in Figure 1. The V_m^E results are given in Table 3 and plotted in Figure 2. The Redlich–Kister equation was fitted to the experimental data of both H_m^E and V_m^E by the method of unweighted least squares:

$$H_m^E = x(1-x) \sum_{i=0}^4 A_i(1-2x)^i \quad (1)$$

$$V_m^E = x(1-x) \sum_{i=0}^4 B_i(1-2x)^i \quad (2)$$

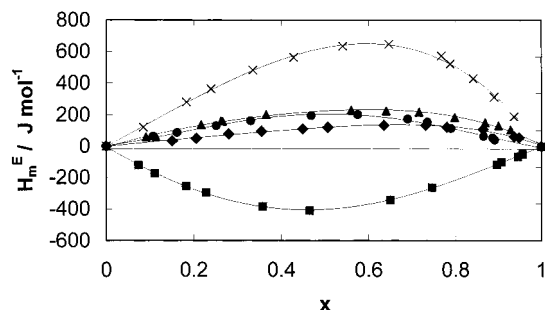
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Table 1. Details of Chemicals: Suppliers, Purity, Experimental and Literature Densities, and Surface Parameter

compound	supplier	purity	density ^(exp)	density ^(lit)	surface parameter
			g cm ⁻³	g cm ⁻³	
1,3-dimethyl-2-imidazolidinone	Acros	99.8	1.0518	1.0519	1.904
methanol	BDH Chemicals	>99.8	0.7863	0.7876	1.432
ethanol	Riedel-de Haën	>99.8	0.7849	0.7859	1.972
1-propanol	Riedel-de Haën	>99	0.7996	0.8004	2.512
2-propanol	Merck	>99.7	0.7812	0.7823	2.508
1-butanol	BDH Chemicals	>99	0.8057	0.8105	3.128

Table 2. Excess Molar Enthalpies, H_m^E , Data for the Mixtures $\{xC_nH_{2n+1}OH + (1-x)C_5H_{10}N_2O\}$ at 298.15 K (ΔH_m^E Values Were the Difference between the Experimental Data and Eq 1)

x	$\frac{H_m^E}{\text{J mol}^{-1}}$	$\frac{\Delta H_m^E}{\text{J mol}^{-1}}$	x	$\frac{H_m^E}{\text{J mol}^{-1}}$	$\frac{\Delta H_m^E}{\text{J mol}^{-1}}$	x	$\frac{H_m^E}{\text{J mol}^{-1}}$	$\frac{\Delta H_m^E}{\text{J mol}^{-1}}$	x	$\frac{H_m^E}{\text{J mol}^{-1}}$	$\frac{\Delta H_m^E}{\text{J mol}^{-1}}$	x	$\frac{H_m^E}{\text{J mol}^{-1}}$	$\frac{\Delta H_m^E}{\text{J mol}^{-1}}$
CH ₃ OH			C ₂ H ₅ OH			C ₃ H ₇ OH			CH ₃ CHOHCH ₃			C ₄ H ₉ OH		
0.0731	-120.7	1.8	0.1507	34.8	0.4	0.0906	59.78	0.0000	0.0	0.0	0.0000	0.0	0.0	0.0
0.1100	-173.4	-1.2	0.2055	50.9	-2.1	0.1133	61.1	-7.7	0.0844	121.3	0.2	0.1068	63.2	1.5
0.1816	-255.3	-2.0	0.2796	79.1	2.7	0.2166	137.0	3.5	0.1829	280.1	-1.2	0.1613	87.5	-2.1
0.2277	-295.1	1.8	0.3547	96.4	0.5	0.2620	159.7	0.9	0.2392	364.1	0.8	0.2513	130.3	0.0
0.3577	-384.1	0.3	0.4489	111.1	-2.5	0.2653	159.7	-0.8	0.3348	481.1	2.1	0.3299	161.9	1.8
0.4649	-410.3	-0.6	0.5067	121.0	-0.5	0.3650	201.6	-1.5	0.4282	562.3	-2.8	0.4687	195.1	-1.6
0.6494	-342.1	0.3	0.6353	135.2	2.8	0.5600	230.0	-1.2	0.5410	633.8	0.2	0.5752	202.2	0.2
0.7459	-263.1	-0.2	0.7314	134.3	1.8	0.6401	225.5	0.2	0.6464	647.1	1.3	0.6898	174.3	0.1
0.8956	-115.5	-1.2	0.7803	122.9	-4.98	0.7157	217.8	5.3	0.7674	559.5	4.2	0.7349	155.3	3.0
0.9067	-100.5	2.3	0.8639	107.5	1.9	0.7983	183.2	-5.3	0.7878	520.9	-4.8	0.7900	114.6	-3.7
0.9451	-64.9	-2.8	0.9369	62.0	-1.3	0.8689	150.7	-2.2	0.8408	427.6	-0.6	0.8651	63.9	-0.3
0.9564	-48.2	1.6	0.9478	56.1	1.7	0.9000	132.0	2.1	0.8896	312.3	-1.0	0.8861	55.1	5.9
1.0000	0.00	0.0	1.0000	0.0	0.0	0.9280	105.5	2.2	0.9361	188.5	2.5	0.8908	41.5	-4.5

**Figure 1.** Excess enthalpy, H_m^E for $\{xC_nH_{2n+1}OH + (1-x)-C_5H_{10}N_2O\}$ at $T = 298.15$ K. Solid lines represent the Redlich-Kister fit. Key: ■, methanol; ◆, ethanol; ▲, 1-propanol; ×, 2-propanol; ●, 1-butanol.

where x is the mole fraction of the aromatic hydrocarbon.

A commercial software package, MATHCAD 6.0, was used to determine the parameters A_r and B_r .

The coefficients A_r and B_r are listed in Tables 4 and 5, respectively, with the standard deviations, σ ,

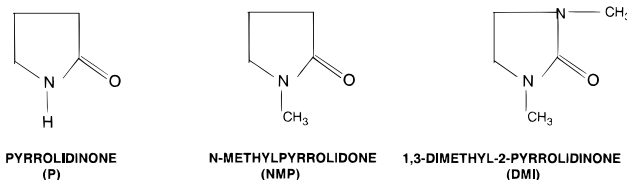
$$\sigma = \left[\frac{\sum_{i=1}^N (X_{m,\text{exp}}^E - X_{m,\text{calc}}^E)^2}{N - r} \right]^{1/2} \quad (3)$$

where X is either H_m^E or V_m^E , N is the number of experimental points, and r is the number of coefficients.

The NRTL⁶ and UNIQUAC⁷ models were tested by fitting the equations to the experimental H_m^E data. The NRTL interaction energy parameters, $g_{12} - g_{22}$ and $g_{21} - g_{11}$, together with the UNIQUAC interaction energy parameters, Δu_{12} and Δu_{21} , were calculated. The results showed that as far as correlation is concerned the Redlich-Kister equation was far superior. The standard deviations of the NRTL and UNIQUAC equations were about 5 times greater than those for the Redlich-Kister equation.

Discussion

In this work three cyclic amides are discussed. They are



The excess molar enthalpy and excess molar volume data presented in this paper are new data for the mixtures (DMI + methanol, ethanol, 1-propanol, 2-propanol, or 1-butanol).

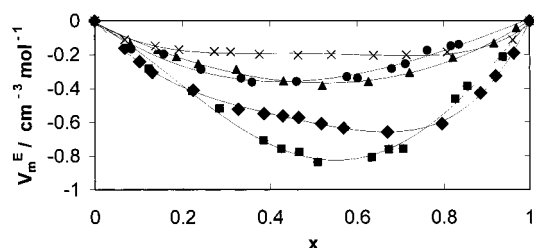
The excess molar enthalpy values were found to be dependent on the carbon number of the primary alkanol and H_m^E ($x = 0.5$) increased in the order methanol < ethanol < 1-propanol \approx 1-butanol < 2-propanol. The magnitude of the excess molar enthalpy of mixing, H_m^E , can be considered to be the result of three effects: (a) the dissociation of associated alkanol molecules (an endothermic effect), (b) decrease in dipole-dipole interactions between DMI molecules as a result of dissociation (an endothermic effect), and (c) hydrogen bond formation between the alkanol and the DMI molecules (an exothermic process).

The magnitude of the excess molar volume is likewise dependent on dissociation effects (a positive contribution) and on the hydrogen bond association between the alkanol and DMI (a negative contribution). The magnitude of the excess molar volume is furthermore complicated by a "packing effect"—the sign of which is difficult to predict.

The H_m^E values ranged from -410 J mol⁻¹ for the (DMI + methanol) mixture to $+650$ J mol⁻¹ for the (DMI + 2-propanol) mixture. The negative H_m^E values indicate that the hydrogen bond formation between the dissimilar species predominates over the dissociation effects. This is supported by the negative excess molar volume values. As

Table 3. Excess Molar Volumes Data, V_m^E , for the Mixtures $\{xC_nH_{2n+1}OH + (1-x)C_5H_{10}N_2O\}$ at 298.15 K (ΔV_m^E Values Were the Difference between the Experimental Data and Eq 2)

x	V_m^E cm ³ mol ⁻¹	ΔV_m^E cm ³ mol ⁻¹	x	V_m^E cm ³ mol ⁻¹	ΔV_m^E cm ³ mol ⁻¹	x	V_m^E cm ³ mol ⁻¹	ΔV_m^E cm ³ mol ⁻¹
CH ₃ OH			C ₂ H ₅ OH			C ₃ H ₇ OH		
0.123	-0.282	0.001	0.068	-0.162	0.004	0.078	-0.124	-0.001
0.221	-0.414	-0.000	0.102	-0.241	0.002	0.143	-0.171	0.009
0.284	-0.517	-0.000	0.130	-0.306	-0.005	0.186	-0.213	-0.004
0.385	-0.707	-0.007	0.328	-0.524	-0.001	0.236	-0.255	-0.012
0.425	-0.757	0.007	0.386	-0.549	-0.001	0.323	-0.288	0.011
0.509	-0.835	0.008	0.429	-0.561	0.003	0.431	-0.356	0.001
0.634	-0.808	-0.004	0.466	-0.572	0.008	0.520	-0.384	-0.006
0.672	-0.759	-0.005	0.518	-0.608	-0.005	0.626	-0.359	0.001
0.826	-0.462	-0.002	0.569	-0.635	-0.008	0.721	-0.307	0.000
0.854	-0.387	-0.013	0.670	-0.658	0.005	0.821	-0.219	0.005
0.937	-0.213	-0.011	0.795	-0.609	-0.001	0.917	-0.134	-0.011
			0.885	-0.428	0.005	0.968	-0.043	0.011
			0.921	-0.327	-0.005			
CH ₃ CHOHCH ₃			C ₄ H ₉ OH					
0.069	-0.112	0.001	0.083	-0.167	-0.004			
0.138	-0.149	0.004	0.243	-0.288	0.010			
0.192	-0.172	-0.006	0.334	-0.339	-0.000			
0.273	-0.181	-0.001	0.358	-0.363	-0.015			
0.310	-0.184	0.002	0.461	-0.359	0.007			
0.376	-0.200	-0.002	0.577	-0.332	0.011			
0.465	-0.204	0.003	0.601	-0.339	-0.008			
0.541	-0.201	0.006	0.680	-0.284	-0.004			
0.638	-0.207	-0.007	0.711	-0.255	-0.001			
0.714	-0.202	-0.006	0.816	-0.149	0.006			
0.808	-0.186	0.012	0.835	-0.140	-0.003			
0.960	-0.113	-0.009						

**Figure 2.** Excess molar volumes V_m^E $\{xC_nH_{2n+1}OH + (1-x)C_5H_{10}N_2O\}$ at $T = 298.15$ K. Solid lines represent the Redlich-Kister fit. Key: ■, methanol; ◆, ethanol; ▲, 1-propanol; ×, 2-propanol; ●, 1-butanol.**Table 4.** Coefficients A_i and the Standard Deviations σ for the Excess Molar Enthalpies for the Mixtures $\{xC_nH_{2n+1}OH + (1-x)C_5H_{10}N_2O\}$ at $T = 298.15$ K

component	A_0	A_1	A_2	A_3	A_4	σ J mol ⁻¹
CH ₃ OH	-1630	-274.7	502.7	-108.5	-456.1	1.82
C ₂ H ₅ OH	482.9	-245.5	201.9	-376.5	54.8	2.60
C ₃ H ₇ OH	919.0	-143.1	71.2	-531.1	222.7	4.22
CH ₃ CHOHCH ₃	2456	-1123	646.1	243.6	-1133	2.69
C ₄ H ₉ OH	803.3	-203.6	-224.7	515.9	-284.5	3.15

Table 5. Coefficients B_i and the Standard Deviations σ for the Excess Molar Volumes for the Mixtures $\{xC_nH_{2n+1}OH + (1-x)C_5H_{10}N_2O\}$ at $T = 298.15$ K

component	B_0	B_1	B_2	B_3	B_4	σ cm ³ mol ⁻¹
CH ₃ OH	-3.353	1.201	2.864	-1.413	-3.726	0.007
C ₂ H ₅ OH	-2.377	0.903	-2.841	0.221	1.739	0.005
C ₃ H ₇ OH	-1.504	0.293	0.574	-0.478	-1.110	0.008
CH ₃ CHOHCH ₃	-0.832	-0.000	-0.147	0.432	-1.983	0.006
C ₄ H ₉ OH	-1.456	-0.243	0.456	-0.793	-0.709	0.008

the chain length of the alkanol molecule increases, the hydrogen bond interaction between the dissimilar species decreases and the overall H_m^E values become more positive.

For a related mixture (2-pyrrolidinone + an alkanol), the H_m^E curve is sigmoidal, ranging from +135 J mol⁻¹ at high methanol concentrations to -80 J mol⁻¹ at low methanol concentrations and the H_m^E curves are endothermic for the mixtures (2-pyrrolidinone + ethanol, 1-propanol, or 1-butanol).¹² Similar interactions are no doubt present in the two related sets of data.

There is evidence that DMI has parallel dipole-dipole interactions^{13,14} as reflected by the Kirkwood correlation factor ($g = 1.2$). A Kirkwood correlation factor of $g = 1$ indicates a normal polar liquid. The order of dipole-dipole interaction is DMI > NMP ($g = 1.20$ and 0.92 , respectively). The strength of the hydrogen bond is DMI > NMP > P. This order is in harmony with that expected from the magnitude of the electron density on the carbonyl oxygen.^{15,16} The groups adjacent to the carbonyl functionality determine the electron density on the carbonyl group. DMI has one methyl group attached to each of the nitrogen atoms, thereby increasing the electron density the most on the carbonyl functionality (methyl groups are electron donating).

The H_m^E ($x = 0.5$) value for the mixture (NMP + cyclohexane) is 1600 J mol⁻¹, indicating the strength of the dipole-dipole interactions of the amide group.¹ For the mixture (NMP + methanol) H_m^E ($x = 0.5$) is approximately -800 J mol⁻¹, again supporting the idea that, for this type of mixture, the hydrogen bond interaction between the dissimilar species predominates. The value of H_m^E for the (DMI + methanol) mixture is -410 J mol⁻¹, indicating that there is a greater compensation between hydrogen bonding and the decrease in dipole-dipole interactions, which will be greater than that for the (NMP + methanol) mixture. Excess molar enthalpies for NMP with higher alkanols have not been reported.

For all the mixtures studied in this work V_m^E is negative. The V_m^E values ranged from -0.83 cm³ mol⁻¹ for the mixture (DMI + methanol) to -0.20 cm³ mol⁻¹ for the mixture (DMI + 2-propanol). For the mixtures studied in this work

V_m^E ($x = 0.5$) increases in the order methanol < ethanol < 1-propanol < 1-butanol < 2-propanol, which is similar to the order reported for the H_m^E measurements. This suggests that the magnitude of the V_m^E values is due to a similar mechanism and that packing effects are either constant throughout the set of mixtures or are not important.

To place the results obtained in this work in perspective, V_m^E results for related systems in which hydrogen bonding occurs are also discussed. For the mixtures {2-pyrrolidinone (P), 1-methyl-pyrrolidinone (NMP), or 1,3-dimethylpyrrolidinone (DMI) + water} the magnitude of V_m^E reflects the difference in strength of the hydrogen bonding between the proton of a water molecule and the carbonyl oxygen in the organic molecule. The V_m^E for the mixtures {(P + water), (NMP + water), and (DMI + water)} were $-0.56 \text{ cm}^3 \text{ mol}^{-1}$, $-1.11 \text{ cm}^3 \text{ mol}^{-1}$, and $-1.33 \text{ cm}^3 \text{ mol}^{-1}$, respectively.

The V_m^E obtained for the mixtures (NMP + an alkanol) ranged from $-0.63 \text{ cm}^3 \text{ mol}^{-1}$ for the mixture (NMP + methanol) to $-0.20 \text{ cm}^3 \text{ mol}^{-1}$ for the mixture (NMP + 1-butanol).² This fits in nicely for the mixtures studied in this work, that is, $-0.83 \text{ cm}^3 \text{ mol}^{-1}$ for the mixture (DMI + methanol) to $-0.36 \text{ cm}^3 \text{ mol}^{-1}$ for the mixture (DMI + 1-butanol). Such behavior might also arise from restrictions of rotational motion when the DMI molecules are accommodated interstitially within the branched structure of *n*-alkanol multimers such as that which occurs in the mixture (*N*-formylmorpholine + an alkanol)¹⁷ and the mixture (NMP + an alkanol).²

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