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Thermodynamics of Ketone + Amine Mixtures. Part VIII. Molar Excess Enthalpies at 298.15 K for *n*-Alkanone + Aniline or + *N*-Methylaniline Systems

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ABSTRACT: Molar excess enthalpies, H_m^E, at 298.15 K and atmospheric pressure have been determined over the entire mole fraction range, using a Tian-Calvet microcalorimeter, for the systems: propanone, 2-butanone, 2-pentanone, or 2-heptanone + aniline or + *N*-methylaniline. The negative H_m^E values obtained for all of the mixtures reveal that interactions between unlike molecules are prevalent. Aniline solutions show more negative H_m^E values due to stronger amine-ketone interactions compared to those in *N*-methylaniline solutions. In addition, H_m^E increases with the size of the alkanone, which may be interpreted in terms of a weakening of interactions between unlike molecules. H_m^E varies somewhat differently in *N*-methylaniline solutions, probably due to the positive contribution to H_m^E is here more important. Molar excess internal energies at constant volume, U_{Vm}^E, have been determined from the present H_m^E data using our previous measurements on molar excess volumes, compressibilities, and isothermal expansion coefficients. U_{Vm}^E behaves similarly to H_m^E. U_{Vm}^E values are much higher than those of H_m^E, which remarks the importance of the contribution of the equation of state term to H_m^E.

INTRODUCTION

As part of our research on the thermodynamic properties of ketone + amine mixtures, we report in this work molar excess enthalpies, H_m^E, at 298.15 K and atmospheric pressure for the systems: propanone, 2-butanone, 2-pentanone, or 2-heptanone + aniline or + *N*-methylaniline. Previously, we have determined densities, ρ, molar excess volumes, V_m^E, excess adiabatic compressibilities, κ_S^E, and excess thermal expansion coefficients, α_p^E, for these solutions.[1−4] These data have been used to compute the corresponding excess molar internal energies at constant volume, U_{Vm}^E. Knowledge of the amine−carbonyl interactions is essential for a better understanding of amide solutions, and of peptide bonds,[5] and is necessary as a first step to investigate complex molecules of biological interest.[6]

EXPERIMENTAL SECTION

Materials. Propanone (≥ 0.995), 2-butanone (≥ 0.995), 2-pentanone (≥ 0.99), and *N*-methylaniline (≥ 0.98) were from Fluka; 2-heptanone (≥ 0.99) was from Sigma-Aldrich and aniline (≥ 0.995) from Riedel de Haën. The chemicals were used without further purification (purities expressed in mass fraction). The ρ values of the pure liquids are in good agreement with those from the literature (Table 1). Density measurements were carried out using a vibrating-tube densimeter and a sound analyzer, Anton Paar model DSA-5000. The uncertainty for the ρ values is $\pm\ 1\cdot 10^{-2}$ kg·m^{-3}, while the corresponding precision is $\pm\ 1\cdot 10^{-3}$ kg·m^{-3}.

Apparatus and Procedure. Binary mixtures were prepared by mass. All weighings were corrected for buoyancy effects, and the error on the final mole fraction is estimated to be less than ± 0.0001. The conversion to molar quantities was based on the relative atomic mass table of 1996 issued by the International Union of Pure and Applied Chemistry (IUPAC).[7]

Table 1. Densities, ρ, of Pure Compounds at 298.15 K

compound	ρ/g·cm^{-3}	
	exp.	lit.
propanone	0.785094	0.78443;[23] 0.7845;[24] 0.7844[25,26] 0.78455[27]
2-butanone	0.799969	0.7997;[25,26] 0.79992[27]
2-pentanone	0.801522	0.8015;[25] 0.80142[28]
2-heptanone	0.811162	0.8111;[29] 0.81123[25]
aniline	1.017457	1.01710;[30] 1.01744;[31] 1.01741[32]
N-methylaniline	0.982237	0.98206[31]

A standard Calvet-type microcalorimeter, equipped with a batch mixing cell with a small (< 2 %) gas phase, was used to determine H_m^E over the entire mole fraction range. Measurements were carried out at 298.15 K and atmospheric pressure. Before mixing, one liquid was maintained in the cell, and the another liquid was maintained in a syringe; both the cell and the syringe are kept in a calorimetric chamber. When baseline was steady, the quantified amount of pure reagent in cell was mixed with that in a syringe by batch injecting. In comparison with previous applications,[8] some important improvements have been developed: (i) the temperature is controlled within ± 0.0006 K using an analogical output through a Eurotherm 2416 controller connected to a power supplier Agilent 6644A; (ii) the initial glass mixing cells have been replaced by ones made of stainless steel, designed by us. For the test of the calorimeter, our H_m^E measurements were compared to those available in the literature for the systems cyclohexane + benzene and cyclohexane

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Table 2. Molar Excess Enthalpies, $H^{\mathrm{E}}_{\mathrm{m}}$, at 298.15 K and Atmospheric Pressure for *n*-Alkanone (1) + Aniline (2) or + *N*-Methylaniline (2) Mixtures

x_1	$H^{\mathrm{E}}_{\mathrm{m}}$/J·mol^{-1}
2-Propanone (1) + Aniline (2)	
0.1130	−427
0.2145	−746
0.3052	−965
0.4107	−1165.
0.5014	−1243
0.5082	−1245
0.6015	−1219
0.6991	−1076
0.8045	−816.
0.9002	−480
2-Butanone (1) + Aniline (2)	
0.1156	−378
0.1565	−499
0.2099	−648.
0.3121	−879
0.4116	−1075
0.5106	−1173
0.6086	−1171
0.7009	−1071
0.8064	−814
0.9018	−469
2-Pentanone (1) + Aniline (2)	
0.1113	−293
0.2052	−511
0.3080	−766
0.4073	−926
0.5067	−1027
0.5088	−1029
0.6049	−1049
0.6088	−1051
0.7108	−940
0.7979	−737
0.8995	−432
2-Heptanone (1) + Aniline (2)	
0.1124	−131
0.2179	−294
0.3082	−449
0.4052	−588
0.4523	−647
0.5096	−691
0.5554	−724
0.6024	−744
0.6026	−740
0.7034	−699
0.7934	−565
0.8479	−471
0.8481	−472
0.8972	−346
Propanone (1) + *N*-Methylaniline (2)	
0.1286	−335
0.2316	−500
0.3191	−598
0.4089	−666
0.5086	−672
0.6075	−622
0.7097	−525
0.8055	−381
0.9012	−204
2-Butanone (1) + *N*-Methylaniline (2)	
0.1361	−356
0.2283	−540
0.3187	−676
0.4093	−781
0.5090	−816
0.6091	−779
0.7055	−672
0.8028	−506
0.9030	−280
2-Pentanone (1) + *N*-Methylaniline (2)	
0.1297	−332
0.2222	−528
0.3239	−693
0.4060	−793
0.5083	−837
0.6057	−807
0.7044	−707
0.8058	−529
0.9031	−292
2-Heptanone (1) + *N*-Methylaniline (2)	
0.1219	−248
0.2133	−415
0.3078	−578
0.4076	−691
0.5029	−741
0.6064	−728
0.7063	−641
0.8037	−489
0.8975	−284

+ 2,2,4-trimethylpentane. Our results differ by less than 1 % from those of the literature[9,10] near $x_1 = 0.5$.

RESULTS AND DISCUSSION

Table 2 lists values of the measured $H^{\mathrm{E}}_{\mathrm{m}}$ vs x_1, the mole fraction of the *n*-alkanone. Our previous data[1−4] on $V^{\mathrm{E}}_{\mathrm{m}}$, κ_S, and α_p for these mixtures have been used to compute $U^{\mathrm{E}}_{\mathrm{Vm}}$ (Table 3) from the equation:[11,12]

$$U^{\mathrm{E}}_{\mathrm{Vm}} = H^{\mathrm{E}}_{\mathrm{m}} - \frac{\alpha_{\mathrm{p}}}{\kappa_{\mathrm{T}}} T V^{\mathrm{E}}_{\mathrm{m}} \qquad (1)$$

Table 3. Molar Excess Internal Energies at Constant Volume, U^E_{Vm}, at 298.15 K and Atmospheric Pressure for *n*-Alkanone (1) + Aniline (2) or + *N*-Methylaniline (2) Mixtures

x_1	U^E_{Vm} / $J \cdot mol^{-1}$
2-Propanone (1) + Aniline (2)	
0.1088	−219
0.1978	−380
0.3039	−537
0.4114	−648
0.5035	−696
0.5501	−700
0.5922	−688
0.6993	−610
0.8082	−455
0.8951	−280
2-Butanone (1) + Aniline (2)	
0.1118	−153
0.1547	−4199
0.2038	−272
0.3064	−426
0.4089	−553
0.5026	−623
0.6019	−639
0.7048	−587
0.8030	−480
0.9019	−277
2-Pentanone (1) + Aniline (2)	
0.0948	−100
0.1497	−169
0.1939	−229
0.2993	−383
0.4058	−515
0.5005	−597
0.6055	−636
0.6963	−596
0.7943	−480
0.8890	−291
2-Heptanone (1) + Aniline (2)	
0.1115	26
0.2133	−43
0.3064	−143
0.4002	−242
0.4537	−301
0.5092	−349
0.5430	−374
0.5964	−404
0.7057	−408
0.8041	−345
0.8515	−289
0.9033	−206
Propanone (1) + *N*-Methylaniline (2)	
0.1447	−244
0.2503	−328
0.2919	−354
0.3924	−380
0.4948	−364
0.6004	−325
0.6962	−257
0.8009	−186
0.8986	−91
2-Butanone (1) + *N*-Methylaniline (2)	
0.1115	−192
0.2076	−325
0.2967	−449
0.3973	−520
0.4950	−545
0.5992	−521
0.6921	−439
0.8007	−333
0.8993	−178
2-Pentanone (1) + *N*-Methylaniline (2)	
0.1000	−160
0.1980	−308
0.3057	−441
0.4037	−525
0.5014	−565
0.6052	−550
0.6985	−489
0.7949	−378
0.8894	−225
2-Heptanone (1) + *N*-Methylaniline (2)	
0.1032	−131
0.2036	−273
0.2987	−394
0.3994	−492
0.4949	−542
0.5977	−545
0.6904	−499
0.8023	−373
0.8989	−212

In this equation, κ_T has been evaluated from the well-known relation:[13]

$$\kappa_T = \kappa_S + \frac{TV_m\alpha_p{}^2}{C_{Pm}} \qquad (2)$$

Due to the lack of experimental data, κ_T values were determined assuming that the isobaric heat capacities of the investigated mixtures are ideal.[14] The data were fitted by unweighted least-squares polynomial regression to the equation:

$$F^E_m = x_1(1 - x_1)\sum_{i=0}^{k-1} A_i(2x_1 - 1)^i \qquad (3)$$

where F_m stands for the properties cited above. The number of coefficients k used in eq 3 for each mixture was determined by

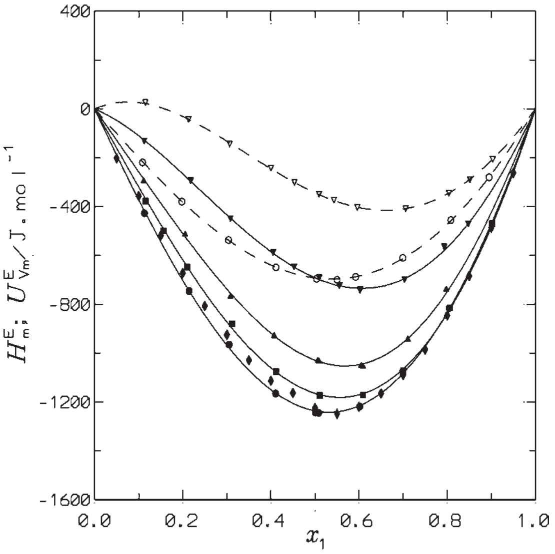

Figure 1. H_m^E and U_{Vm}^E at 298.15 K for *n*-alkanone (1) + aniline (2) mixtures. Full points, H_m^E, measurements: ●, propanone; ■, 2-butanone; ▲, 2-pentanone; ▼, 2-heptanone (this work); ◆, propanone;[16] open symbols, U_{Vm}^E data: ○, propanone; ▽, 2-heptanone (this work). Curves, H_m^E (solid lines) and U_{Vm}^E (dashed lines), indicated results obtained using eq 3 and parameters listed in Table 4.

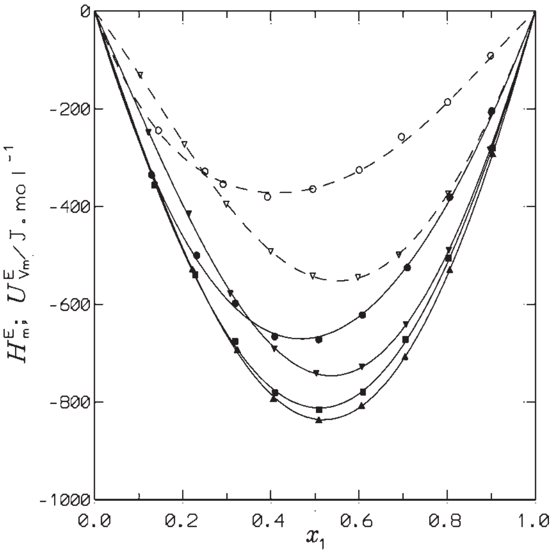

Figure 2. H_m^E and U_{Vm}^E at 298.15 K for *n*-alkanone (1) + *N*-methylaniline (2) mixtures. Full points, H_m^E measurements: ●, propanone; ■, 2-butanone; ▲, 2-pentanone; ▼, 2-heptanone; open symbols, U_{Vm}^E data: ○, propanone; ▽, 2-heptanone (this work). Curves, H_m^E (solid lines) and U_{Vm}^E (dashed lines), indicate results obtained using eq 3 and parameters listed in Table 4.

Table 4. Coefficients A_i and Standard Deviations, $\sigma(F_m^E)$ (eq 4), for the Representation of the F_m^E Property at Temperature T for *n*-Alkanone (1) + Aniline (2) or + *N*-Methylaniline (2) Systems by eq 3

	F_m^E					$\sigma(F_m^E)$
system	J·mol^{-1}	A_0	A_1	A_2	A_3	J·mol^{-1}
propanone + aniline	H_m^E	−4946	−690	400		9
	U_{Vm}^E	−2775	−450	309		2
2-butanone + aniline	H_m^E	−4660	−1150	290		10
	U_{Vm}^E	−2473	−1075	339		7
2-pentanone + aniline	H_m^E	−4115	−1340	500	300	10
	U_{Vm}^E	−2401	−1273	596	326	7
2-heptanone + aniline	H_m^E	−2767	−1570	450		6
	U_{Vm}^E	−1371	−1657	520		2
propanone + *N*-methylaniline	H_m^E	−2672	340			8
	U_{Vm}^E	−1469	592			6
2-butanone + *N*-methylaniline	H_m^E	−3248	−140	300		6
	U_{Vm}^E	−2178	−7.3	330		8
2-pentanone + *N*-methylaniline	H_m^E	−3340	−295	370		3
	U_{Vm}^E	−2255	−313	351		0.6
2-heptanone + *N*-methylaniline	H_m^E	−2966	−517	490		3
	U_{Vm}^E	−2180	−567	463		2

applying an *F*-test[15] at the 99.5 % confidence level. Table 4 lists the parameters A_i obtained in the regression, together with the standard deviations σ, defined by:

$$\sigma(F_m^E) = \left[\frac{1}{N-k}\sum(F_{m,\,cal}^E - F_{m,\,exp}^E)^2\right]^{1/2} \tag{4}$$

where N is the number of direct experimental values. Figures 1 and 2 show the data graphically. It is remarkable that our H_m^E measurements are in good agreement with those available in the literature for the propanone + aniline system.[16]

Hereafter, we are referring to the values of the excess functions at equimolar composition and 298.15 K. The negative H_m^E values obtained (Table 2) indicate the existence of strong interactions between unlike molecules. In a previous article,[1] the strength of the H-bonds between aniline and propanone has been evaluated to be −30.52 kJ·mol^{-1}, a value which is even lower than that for the H-bonds between 1-alkanol molecules (−25.1 kJ·mol^{-1} in the ERAS model[17,18]). The large and negative V_m^E values of these systems also support the existence of strong amine−ketone interactions. Thus, V_m^E/cm^3·mol^{-1} = −1.183 and −0.727 for the propanone + aniline or + *N*-methylaniline mixtures,[1] respectively, and −0.861 and −0.509 for the 2-heptanone + aniline or + *N*-methylaniline systems.[3] We note that for a given alkanone, H_m^E(aniline) < H_m^E(*N*-methylaniline) (Table 2, Figures 1 and 2). This may be due to: (i) interactions between unlike molecules are stronger in aniline solutions; (ii) a lower number of amine−ketone interactions is created upon mixing in the case of systems containing *N*-methylaniline, as in this molecule the amine group is more sterically hindered; (iii) a higher positive contribution to H_m^E from the breaking of the ketone−ketone interactions when *N*-methylaniline is mixed with alkanones, because of the aliphatic surface of this secondary amine.

The variation of H_m^E with the chain length of the 2-alkanone is somewhat different for mixtures with aniline of *N*-methylaniline. For aniline systems, H_m^E/J·mol^{-1} changes in the sequence: −1236 (propanone) < −1165 (2-butanone) < −1029 (2-pentanone) < −692 (2-heptanone), which may be ascribed to a weakening of the interactions between unlike molecules, when the size of the ketone is increased. For *N*-methylaniline systems, H_m^E/J·mol^{-1}

varies in the order: −668 (propanone) > −712 (2-heptanone) > −812 (2-butanone) > −835 (2-pentanone). A similar trend is encountered in alkanenitrile + 2-alkanone mixtures.[19] Such H_m^E variation is probably due to the major importance of the positive contribution to H_m^E from the disruption of the alkanone-alkanone interactions, more favorably produced by the existence of the aliphatic part of *N*-methylaniline. It should be noted that in systems with heptane, $H_m^E/\mathrm{J\cdot mol^{-1}}$ changes as follows: 1704 (propanone)[20] > 1338 (2-butanone)[21] > 1135 (2-pentanone)[21] > 886 (2-heptanone).[22]

On the other hand, the large difference between H_m^E and U_{Vm}^E values (Tables 2 to 4, Figures 1 and 2) remarks on the importance of the equation of state contribution, which is related to the large and negative V_m^E of these systems[1−4] (see above). The observed trends for H_m^E are still valid for U_{Vm}^E. However, it is remarkable that the latter magnitude is practically constant from 2-butanone for *N*-methylaniline systems, which reveals that the different contributions to U_{Vm}^E are here counterbalanced.

■ CONCLUSIONS

H_m^E values at 298.15 K have been determined for the propanone, 2-butanone, 2-pentanone, or 2-heptanone + aniline or + *N*-methylaniline systems. Negative H_m^E values reveal that interactions between unlike molecules are dominant. Aniline solutions show more negative H_m^E values due to stronger amine−ketone interactions compared to those in *N*-methylaniline solutions. In addition, H_m^E increases with the size of the alkanone, which may be interpreted in terms of a weakening of interactions between unlike molecules. H_m^E varies somewhat differently in *N*-methylaniline solutions, probably because the positive contribution to H_m^E is more important here. The excess functions H_m^E and U_{Vm}^E show similar trends, although the latter is much higher, which remarks on the importance of the contribution of the equation of state term to H_m^E.

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