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Ternary Liquid−Liquid Equilibria for Mixtures of an *n*-Alkane + an Aromatic Hydrocarbon + *N*-Methyl-2-pyrrolidone at 298.2 K and 1 atm

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Tie lines and liquid−liquid equilibrium data are presented for mixtures of an *n*-alkane + toluene + *N*-methyl-2-pyrrolidone and mixtures of *n*-hexadecane + an aromatic hydrocarbon + *N*-methyl-2-pyrrolidone at 298.2 K and 1 atm pressure. *n*-Alkane refers to *n*-hexane or *n*-nonane or *n*-tetradecane or *n*-hexadecane, and an aromatic hydrocarbon refers toluene or *o*-xylene or *m*-xylene or *p*-xylene or mesitylene or ethylbenzene. *N*-methyl-2-pyrrolidone is an important solvent used in the extraction of aromatic compounds from alkane−aromatic mixtures. The data presented here complements data in the literature and gives a broader description of the phase equilibrium properties of the mixtures generated by an *n*-alkane + an aromatic hydrocarbon and *N*-methyl-2-pyrrolidone.

Introduction

An important technique in the separation of compounds is liquid−liquid extraction (Muller and Hoehfeld, 1967). This paper deals with the liquid−liquid equilibria for mixtures of (*n*-hexane or *n*-nonane or *n*-tetradecane or n-hexadecane + toluene + *N*-methyl-2-pyrrolidone) and mixtures of (hexadecane + *o*-xylene or *m*-xylene or *p*-xylene or mesitylene or ethylbenzene + NMP) at 298.2 K and 1 atm pressure. *N*-methyl-2-pyrrolidone (NMP) is a dipolar aprotic solvent used in the well-established Arosolvon process (Muller and Hoehfeld, 1967) for separating aromatic hydrocarbons from petroleum feedstock. In this work new data is presented and together with data from the literature by Al-Zayied et al. (1990) and Ferreira et al. (1984a,b), liquid−liquid equilibria for mixtures of the type defined above are summarized.

Experimental Section

Procedure. The binodal curves were determined at 298.2 K and 1 atm pressure using the cloud point method described by Letcher and Siswana (1992a). Tie lines were determined using the refractive index method described by Briggs and Comings (1943). Details of the chemicals used are given in Table 1. The purity of chemicals was determined by gas chromatography. The *n*-alkanes were used as is. The aromatic hydrocarbons were stored under 4 Å molecular sieves.

Table 1. Refractive Indices of Pure Compounds at 298.2 K

compound	n_D^{25} exptl	n_D^{25} lit.[a]	supplier	purity (%)
NMP	1.4680	1.4675	Sigma	>99
toluene	1.4940	1.4940	SAARChem	99
o-xylene	1.5018	1.5029	JANESSEN CHIMICA	99
m-xylene	1.4943	1.4946	Merck	99
p-xylene	1.4925	1.4932	JANESSEN CHIMICA	>99
mesitylene	1.4965	1.4968	Merck	98
ethylbenzene	1.4928	1.4932	ACROS	99
n-hexane	1.3725	1.3723	SAARChem	99
nonane	1.4052	1.4031	Fluka	99.8
tetradecane	1.4275	1.4269	Fluka	>99
hexadecane	1.4325	1.4325	ACROS	99

[a] Riddick et al. (1986).

Table 2. Compositions of Points on the Binodal Curve at 298.2 K and 1 atm Pressure for the Mixtures $\{x_1C_nH_{2n+2} + x_2C_6H_5CH_3 + (1 - x_1 - x_2)(C_5H_9NO)\}$

x_1	x_2	x_1	x_2
		C_6H_{14}	
0.174	0.000	0.503	0.130
0.237	0.058	0.707	0.100
0.282	0.089	0.785	0.081
0.289	0.097	0.842	0.041
0.387	0.127	0.919	0.000
0.434	0.130		
		C_9H_{20}	
0.079	0.000	0.464	0.208
0.102	0.104	0.645	0.172
0.220	0.166	0.783	0.109
0.290	0.216	0.931	0.000
0.307	0.208		
		$C_{14}H_{30}$	
0.021	0.000	0.345	0.348
0.031	0.098	0.462	0.310
0.052	0.190	0.593	0.255
0.082	0.274	0.708	0.175
0.169	0.333	0.818	0.095
		$C_{16}H_{34}$	
0.012	0.000	0.437	0.348
0.022	0.106	0.531	0.309
0.053	0.289	0.534	0.308
0.107	0.359	0.607	0.247
0.324	0.396	0.710	0.179
0.391	0.368	0.781	0.120
0.394	0.363	0.920	0.000

Results

The compositions of mixtures on the binodal curve are given in Table 2 and Table 3. The compositions of the conjugate phases are given in Table 4 and Table 5. The compositions of the plait points or critical points, which were determined following the method by Treybal et al. (1946), are given in Table 6 and Table 7. The ternary phase diagrams are given in Figures 1−9, where x_1 refers to mole fraction of the *n*-alkane, x_2 refers to the mole fraction of the aromatic hydrocarbon, and x_3 refers to the mole fraction of NMP. The relative solubility of toluene in NMP and an *n*-alkane is plotted in Figure 10. The relative solubility of the aromatic compound in the NMP-rich phase and in the *n*-alkane-rich phase is plotted in Figure 11. Three equations have been fitted to these data following the work of Hlavatý (1972). The coefficients A_i relate to the modified Hlavatý equation

$$x_2 = A_2x_A \ln x_A = A_1x_B \ln A_3x_Ax_B \tag{1}$$

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Table 3. Compositions of Points on the Binodal Curve at 298.2 K and 1 atm Pressure for the Mixtures $\{x_1C_{16}H_{34} + x_2C_6H_mR_{6-m} + x_3C_5H_9NO\}$

x_1	x_2	x_1	x_2
$C_6H_5CH_3$			
0.012	0.000	0.437	0.348
0.022	0.106	0.531	0.309
0.053	0.289	0.534	0.308
0.107	0.359	0.607	0.247
0.324	0.396	0.710	0.179
0.391	0.368	0.781	0.120
0.394	0.363	0.920	0.000
o-$C_6H_4(CH_3)_2$			
0.012	0.000	0.417	0.354
0.021	0.096	0.524	0.302
0.037	0.193	0.644	0.236
0.071	0.281	0.854	0.063
0.148	0.343	0.920	0.000
m-$C_6H_4(CH_3)_2$			
0.012	0.000	0.226	0.354
0.022	0.082	0.498	0.310
0.035	0.168	0.656	0.205
0.060	0.251	0.707	0.171
0.122	0.316	0.920	0.000
p-$C_6H_4(CH_3)_2$			
0.012	0.000	0.420	0.355
0.021	0.093	0.543	0.304
0.038	0.188	0.662	0.235
0.073	0.274	0.732	0.154
0.156	0.333	0.920	0.000
0.268	0.363		
1,3,5-$C_6H_3(CH_3)_3$			
0.012	0.000	0.187	0.323
0.023	0.055	0.202	0.331
0.027	0.102	0.295	0.357
0.051	0.187	0.532	0.304
0.057	0.192	0.728	0.163
0.109	0.268	0.831	0.076
0.110	0.266	0.920	0.000
$C_6H_5(CH_3CH_2)$			
0.012	0.000	0.324	0.370
0.020	0.097	0.452	0.335
0.037	0.192	0.715	0.203
0.072	0.279	0.809	0.105
0.125	0.327	0.920	0.000
0.269	0.365		

Table 4. Compositions of Conjugate Solutions x_1', x_2', x_1'', and x_2'' for $\{x_1C_nH_{2n+2} + x_2C_6H_5CH_3 + (1 - x_1 - x_2)(C_5H_9NO)\}$ at 298.15 K and 1 atm Pressure

NMP-rich phase		n-alkane-rich phase	
x_1'	x_1'	x_1''	x_2''
C_6H_{12}			
0.211	0.042	0.870	0.030
0.234	0.061	0.843	0.045
0.245	0.069	0.831	0.052
0.305	0.100	0.767	0.080
0.320	0.106	0.754	0.086
0.344	0.113	0.731	0.095
C_9H_{20}			
0.121	0.117	0.798	0.096
0.134	0.132	0.777	0.110
0.146	0.143	0.759	0.121
0.173	0.164	0.724	0.140
0.199	0.177	0.694	0.155
0.264	0.198	0.626	0.182
$C_{14}H_{30}$			
0.025	0.063	0.784	0.120
0.026	0.078	0.751	0.146
0.028	0.100	0.703	0.180
0.033	0.132	0.638	0.225
0.039	0.158	0.585	0.256
0.066	0.234	0.463	0.323
$C_{16}H_{34}$			
0.014	0.030	0.827	0.083
0.016	0.102	0.639	0.231
0.018	0.124	0.588	0.266
0.020	0.147	0.537	0.298
0.025	0.177	0.478	0.331
0.042	0.248	0.350	0.385

Table 5. Compositions of Conjugate Solutions x_1', x_2', x_1'', and x_2'', for the Mixtures $\{x_1C_{16}H_{34} + x_2C_6H_mR_{6-m} + x_3C_5H_9NO\}$ at 298.2 K and 1 atm Pressure

x_1'	x_2'	x_1''	x_2''
$C_6H_5CH_3$			
0.014	0.030	0.827	0.083
0.016	0.102	0.639	0.231
0.018	0.124	0.588	0.266
0.020	0.147	0.537	0.298
0.025	0.177	0.478	0.331
0.042	0.248	0.350	0.385
o-$C_6H_4(CH_3)_2$			
0.013	0.024	0.878	0.040
0.014	0.045	0.840	0.076
0.016	0.068	0.802	0.109
0.019	0.100	0.744	0.158
0.027	0.150	0.656	0.224
0.032	0.174	0.615	0.252
0.055	0.241	0.499	0.319
m-$C_6H_4(CH_3)_2$			
0.015	0.045	0.827	0.080
0.017	0.065	0.787	0.112
0.019	0.080	0.758	0.135
0.020	0.094	0.732	0.155
0.027	0.134	0.655	0.209
0.034	0.164	0.599	0.245
0.035	0.167	0.593	0.249
0.056	0.228	0.486	0.307
p-$C_6H_4(CH_3)_2$			
0.014	0.039	0.841	0.064
0.016	0.070	0.787	0.114
0.029	0.153	0.647	0.227
0.038	0.185	0.592	0.264
0.040	0.192	0.581	0.271
0.073	0.260	0.458	0.333
0.106	0.297	0.386	0.357
1,3,5-$C_6H_3(CH_3)_3$			
0.019	0.052	0.785	0.121
0.022	0.070	0.743	0.156
0.030	0.106	0.657	0.220
0.035	0.126	0.611	0.251
0.037	0.131	0.600	0.257
0.060	0.193	0.462	0.326
0.065	0.203	0.441	0.333
0.096	0.253	0.335	0.355
$C_6H_5(CH_3CH_2)$			
0.013	0.058	0.826	0.095
0.014	0.069	0.807	0.113
0.015	0.084	0.783	0.135
0.018	0.116	0.730	0.181
0.020	0.131	0.705	0.201
0.028	0.174	0.631	0.255

Table 6. Compositions of Plait Points for the Mixtures $\{x_1C_nH_{2n+2} + x_2C_6H_5CH_3 + (1 - x_1 - x_2)(C_5H_9NO)\}$at 298.2 K and 1 atm Pressure

n-alkane	x_1	x_2
C_6H_{12}	0.539	0.125
C_9H_{20}	0.446	0.213
$C_{14}H_{30}$	0.202	0.347
$C_{16}H_{34}$	0.149	0.382

Table 7. Compositions of Plait Points for the Mixtures $\{x_1C_{16}H_{34} + x_2C_6H_mR_{6-m} + x_3C_5H_9NO\}$ at 298.2 K and 1 atm Pressure

aromatic hydrocarbon	x_1	x_2
$C_6H_5CH_3$	0.149	0.382
o-$C_6H_4(CH_3)_2$	0.215	0.383
m-$C_6H_4(CH_3)_2$	0.208	0.367
p-$C_6H_4(CH_3)_2$	0.215	0.383
1,3,5-$C_6H_3(CH_3)_3$	0.178	0.336
$C_6H_5(CH_3CH_2)$	0.217	0.362

the coefficients B_i relate to a β function equation

$$x_2 = B_2(1 - x_A)^{B1}{x_A}^{B2} \tag{2}$$

and the coefficients C_i relate to a log γ

$$x_2 = C_2(-\ln x_A)^{C1}{x_A}^{C3} \tag{3}$$

where

$$x_A = x_1 + \frac{1}{2}x_1 - x_1^\circ/(x_{11}^\circ - x_1^\circ) \tag{4}$$

$$x_B = x_{11}^\circ - x_1 - \frac{1}{2}x_2/(x_{11}^\circ - x_1^\circ) \tag{5}$$

where, x_1 refers to the mole fraction of the n-alkane and x_2 refers to the mole fraction of the aromatic hydrocarbon and x_{11}° and x_1° are the values of x_1 on the binodal curve that cuts the $x_2 = 0$ axis and have been used to summarize the

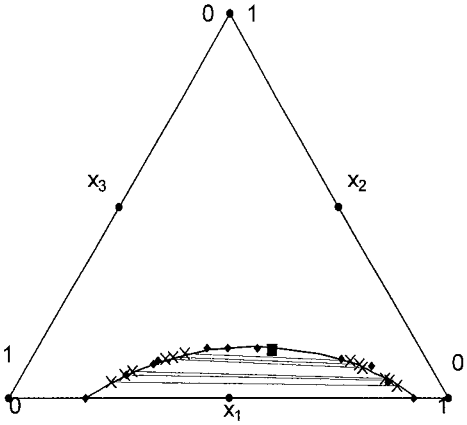

Figure 1. Binodal curve and tie lines for $x_1(C_6H_{14})$ + $x_2(C_6H_5\text{-}CH_3)$ + $x_3(C_5H_9NO)$ at 298.2 K and 1 atm pressure. Legend: ◆ = binodal curve, × = tie line and ■ = plait point determined by the Treybal method.

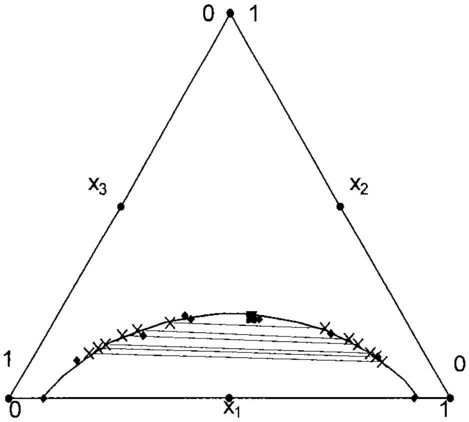

Figure 2. Binodal curve and tie lines for $x_1(C_9H_{20})$ + $x_2(C_6H_5\text{-}CH_3)$ + $x_3(C_5H_9NO)$ at 298.2 K and 1 atm pressure. Legend: ◆ = binodal curve, × = tie line, and ■ = plait point determined by the Treybal method.

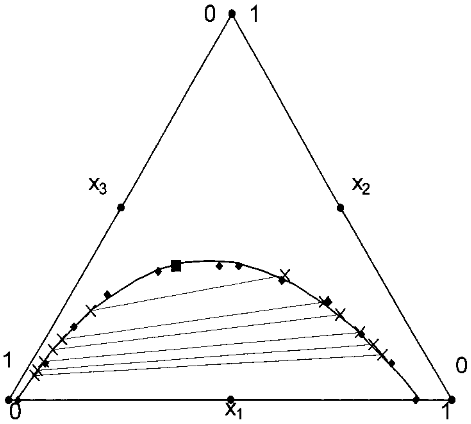

Figure 3. Binodal curve and tie lines $x_1(C_{16}H_{34})$ + $x_2(C_6H_5CH_3)$ + $x_3(C_5H_9NO)$ at 298.2 K and 1 atm pressure. Legend: ◆ = binodal curve, × = tie line, and ■ = plait point determined by the Treybal method.

binodal curve data. Letcher and co-workers (1992b) have discussed these equations previously. The coefficients A_i, B_i, and C_i are given in Table 8 and Table 9 together with the standard deviation, σ, which is defined as

$$\sigma = [\sum(x_2(\text{calc}) - x_2(\text{exp}))^2/(n - 3)]^{1/2} \tag{6}$$

where n is the number of data points and 3 is the number of coefficients.

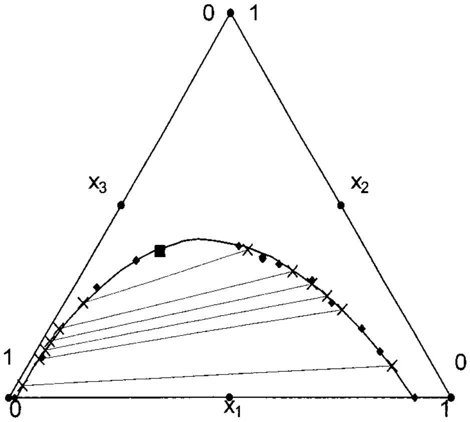

Figure 4. Binodal curve and tie lines $x_1(C_{14}H_{30})$ + $x_2(C_6H_5CH_3)$ + $x_3(C_5H_9NO)$ at 298.2 K and 1 atm pressure. Legend: ◆ = binodal curve, × = tie line, and ■ = plait point determined by the Treybal method.

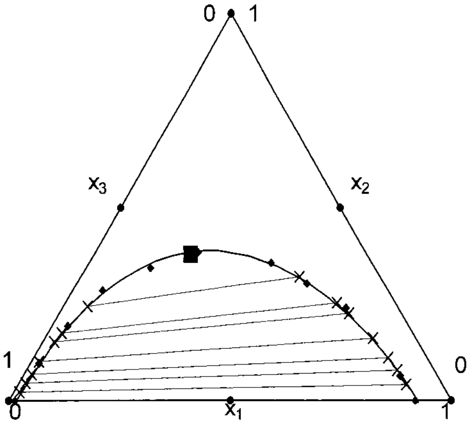

Figure 5. Binodal curve and tie lines for $x_1(C_{16}H_{34})$ + $x_2(o\text{-}C_6H_4(CH_3)_2$ + $x_3(C_5H_9NO)$ at 298.2 K and 1 atm pressure. Legend: ◆ = binodal curve, × = tie line, and ■ = plait point determined by the Treybal method.

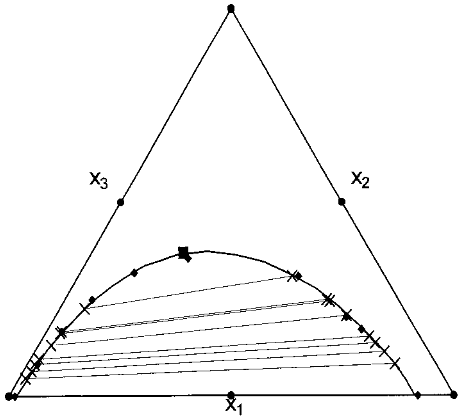

Figure 6. Binodal curve and Tie lines for $x_1(C_{16}H_{34})$ + $x_2(m\text{-}C_6H_4(CH_3)_2$ + $x_3(C_5H_9NO)$ at 298.2 K and 1 atm pressure. Legend: ◆ = binodal curve, × = tie line, and ■ = plait point determined by the Treybal method.

Discussion

***n-Alkane* + *Toluene* + *N-Methyl-2-pyrrolidone*.** (See Table 2 and Figures 1–4.) From the binodal curve data for *n*-hexane or *n*-nonane or n-tetradecane or *n*-hexadecane + toluene + NMP, it can be seen that an increase in the carbon chain length of the *n*-alkane results in an increase in the area of the two-phase region; i.e., the mutual solubility of the components are reduced. The tie lines show that toluene is slightly more soluble in the NMP-rich

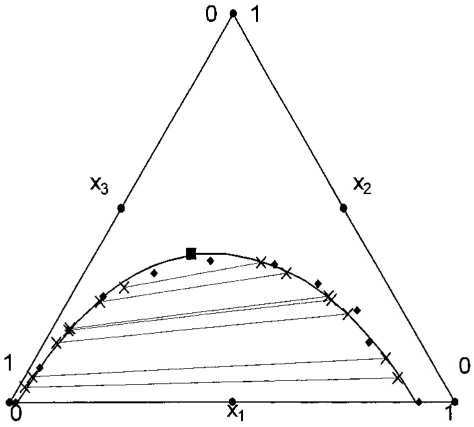

Figure 7. Binodal curve and tie lines for $x_1(C_{16}H_{34})$ + $x_2(p\text{-}C_6H_4(CH_3)_2$ + $x_3(C_5H_9NO)$ at 298.2 K and 1 atm pressure. Legend: ◆ = binodal curve, × = tie line, and ■ = plait point determined by the Treybal method.

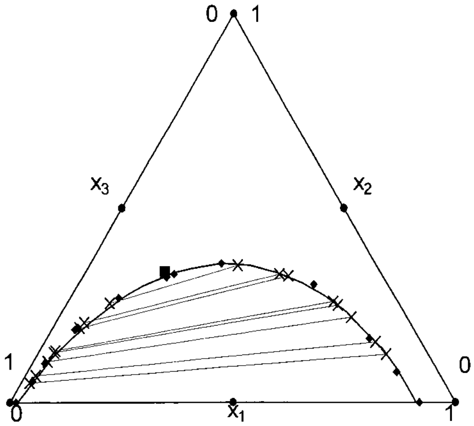

Figure 8. Binodal curve and tie lines for $x_1(C_{16}H_{34})$ + $x_2(1,3,5\text{-}C_6H_3(CH_3)_3$ + $x_3(C_5H_9NO)$ at 298.2 K and 1 atm pressure. Legend: ◆ = binodal curve, × = tie line, and ■ = plait point determined by the Treybal method.

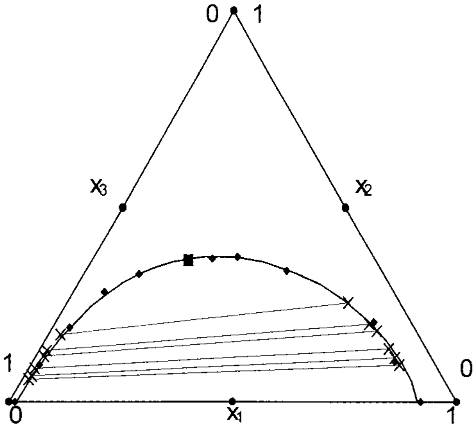

Figure 9. Binodal curve and tie lines for $x_1(C_{16}H_{34})$ + x_2 $C_6H_5CH_2CH_3$ + $x_3(C_5H_9NO)$ at 298.2 K and 1 atm pressure. Legend: ◆ = binodal curve, × = tie line, and ■ = plait point determined by the Treybal method.

phase than in the *n*-alkane-rich phase for mixtures containing *n*-hexane or *n*-nonane and the reverse is true for mixtures containing the higher *n*-alkanes, *n*-tetradecane and *n*-hexadecane. Increasing the carbon chain length of the *n*-alkane has a dramatic effect on the plait point, and it moves from a region of high *n*-alkane concentration to a region of low *n*-alkane concentration. The work presented here is in agreement to within 0.01 mole fraction with the work of Ferreira et al. (1984a,b), in which the concentration of conjugate phases was determined by gas−liquid chromatography. However, our results are not compatible with the work of Al-Zayied et al. (1990). We have checked our work many times and were not able to reproduce the shape of the phase diagram as given by Al-Zayied et al. (1990).

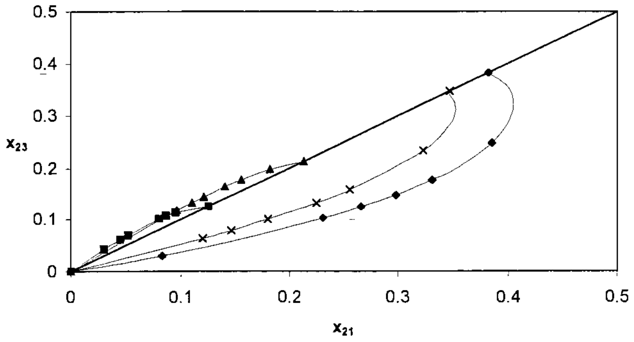

Figure 10. Relative solubility of toluene in NMP and the *n*-alkane at 298.2 K and 1 atm pressure, x_{21} versus x_{23}. Legend: ■ = *n*-hexane, ▲ = *n*-nonane, × = tetradecane, and ◆ = hexadecane

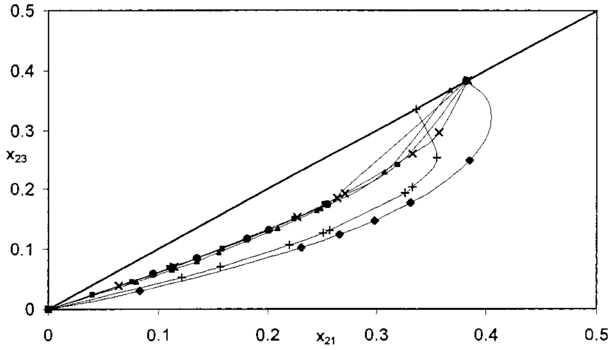

Figure 11. Relative solubility of the aromatic in NMP and in hexadecane at 298.2K and 1 atm pressure, x_{21} versus x_{23}. Legend: ◆ = $C_6H_5CH_3$, ■ = $o\text{-}C_6H_4(CH_3)_2$, ▲ = $m\text{-}C_6H_4(CH_3)_2$, × = $p\text{-}C_6H_4(CH_3)_2$, + = $(1,3,5\text{-}C_6H_3(CH_3)_3$, and ● = $C_6H_5CH_2CH_3$.

Table 8. Coefficients *A*, *B*, and *C* in Eqs 1−3 Respectively for $\{x_1C_nH_{2n+2} + x_2C_6H_5CH_3 + (1 - x_1 - x_2)(C_5H_9NO)\}$at 298.2 K and 1 atm Pressure

Hlavatý	β	log γ
	C_6H_{12}	
$A_1 = -0.080$	$B_1 = 0.436$	$C_1 = 0.407$
$A_2 = -0.139$	$B_2 = 0.818$	$C_2 = 0.790$
$A_3 = 0.232$	$B_3 = 0.884$	$C_3 = 1.188$
$\sigma = 0.005$	$\sigma = 0.005$	$\sigma = 0.005$
	C_9H_{20}	
$A_1 = -0.378$	$B_1 = 0.557$	$C_1 = 0.516$
$A_2 = -0.367$	$B_2 = 0.693$	$C_2 = 0.659$
$A_3 = -0.178$	$B_3 = 0.686$	$C_3 = 0.926$
$\sigma = 0.010$	$\sigma = 0.010$	$\sigma = 0.009$
	$C_{14}H_{30}$	
$A_1 = -0.239$	$B_1 = 1.277$	$C_1 = 1.162$
$A_2 = -0.058$	$B_2 = 0.964$	$C_2 = 0.922$
$A_3 = 1.007$	$B_3 = 0.884$	$C_3 = 1.226$
$\sigma = 0.013$	$\sigma = 0.013$	$\sigma = 0.009$
	$C_{16}H_{34}$	
$A_1 = -0.156$	$B_1 = 1.568$	$C_1 = 1.433$
$A_2 = 0.042$	$B_2 = 1.018$	$C_2 = 0.977$
$A_3 = 1.456$	$B_3 = 0.938$	$C_3 = 1.308$
$\sigma = 0.020$	$\sigma = 0.020$	$\sigma = 0.015$

n-Hexadecane + an Aromatic Hydrocarbon + N-Methyl-2-pyrrolidone. (See Table 3 and Figures 4−10.) Figures 4−10 show that for all the systems described here, the area of the two-phase region changes little with a change in the type of aromatic compound used, and the mutual solubilities of the compounds used in these mixtures are almost independent of the type of aromatic hydrocarbon involved in the mixture. From the slope of the tie lines it can be clearly seen that the aromatic

Table 9. Coefficients *A*, *B*, and *C* in Eqs 1−3 Respectively for the Mixtures $\{x_1C_{16}H_{34} + x_2C_6H_mR_{6-m} + x_3C_5H_9NO\}$ at 298.2 K and 1 atm Pressure

Hlavatý	β	$\log \gamma$
	$C_6H_5(CH_3)$	
$A_1 = -0.156$	$B_1 = 1.568$	$C_1 = 1.433$
$A_2 = 0.042$	$B_2 = 1.018$	$C_2 = 0.977$
$A_3 = 1.456$	$B_3 = 0.938$	$C_3 = 1.308$
$\sigma = 0.020$	$\sigma = 0.019$	$\sigma = 0.015$
	o-$C_6H_4(CH_3)_2$	
$A_1 = -0.169$	$B_1 = 1.377$	$C_1 = 1.254$
$A_2 = -0.146$	$B_2 = 0.925$	$C_2 = 0.882$
$A_3 = 1.102$	$B_3 = 0.915$	$C_3 = 1.242$
$\sigma = 0.012$	$\sigma = 0.012$	$\sigma = 0.008$
	m-$C_6H_4(CH_3)_2$	
$A_1 = -0.130$	$B_1 = 1.404$	$C_1 = 1.246$
$A_2 = 0.005$	$B_2 = 0.995$	$C_2 = 0.939$
$A_3 = 1.299$	$B_3 = 0.936$	$C_3 = 1.273$
$\sigma = 0.015$	$\sigma = 0.014$	$\sigma = 0.011$
	p-$C_6H_4(CH_3)_2$	
$A_1 = -0.196$	$B_1 = 1.284$	$C_1 = 1.172$
$A_2 = -0.255$	$B_2 = 0.875$	$C_2 = 0.833$
$A_3 = -0.877$	$B_3 = 0.899$	$C_3 = 1.204$
$\sigma = 0.014$	$\sigma = 0.014$	$\sigma = 0.013$
	1,3,5-$C_6H_3(CH_3)_3$	
$A_1 = -0.073$	$B_1 = 1.299$	$C_1 = 1.178$
$A_2 = -0.184$	$B_2 = 0.908$	$C_2 = 0.865$
$A_3 = 1.069$	$B_3 = 0.957$	$C_3 = 1.273$
$\sigma = 0.011$	$\sigma = 0.011$	$\sigma = 0.011$
	$C_6H_5(CH_2CH_3)$	
$A_1 = -0.330$	$B_1 = 1.165$	$C_1 = 1.083$
$A_2 = -0.404$	$B_2 = 0.807$	$C_2 = 0.778$
$A_3 = 0.473$	$B_3 = 0.836$	$C_3 = 1.128$
$\sigma = 0.017$	$\sigma = 0.017$	$\sigma = 0.014$

compound is more soluble in the hexadecane-rich phase than in the NMP-rich phase. It can also be seen from the tie line data that as the number of methyl groups on the benzene ring increases, the solubility of the aromatic compound in the NMP-rich phase decreases and, correspondingly, increases in the hexadecane-rich phase.

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