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Isobaric Vapor−Liquid−Liquid Equilibrium and Vapor−Liquid Equilibrium for the Quaternary System Water−Ethanol−Cyclohexane−Isooctane at 101.3 kPa

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Experimental isobaric vapor−liquid−liquid and vapor−liquid equilibrium data for the ternary system water (1) −cyclohexane (2) −isooctane (3) and the quaternary system water (1) −ethanol (2) −cyclohexane (3) −isooctane (4) were measured at 101.3 kPa. An all-glass, dynamic recirculating still equipped with an ultrasonic homogenizer was used to determine the VLLE. The results obtained show that the system does not present quaternary azeotropes. The point-by-point method by Wisniak for testing the thermodynamic consistency of isobaric measurements was used to test the equilibrium data.

Introduction

Systems composed of hydrocarbons, water, and ethanol are of importance in the fuel industry where ethanol + gasoline blends are widely used. Alcohol increases octane levels while also promoting more complete combustion, which reduces harmful exhaust pipe emissions. However, small amounts of water in the blend can lead to phase splitting, which in turn can cause engine trouble. For this reason, ethanol must be dehydrated prior to blending with gasoline. Currently, this is achieved by various techniques with adsorption and azeotropic distillation being the most commonly used. Heterogeneous azeotropic distillation involves adding a third component, which causes liquid−liquid phase separation. This liquid−liquid phase split provides a cheap and efficient method for crossing the distillation boundaries caused by the presence of azeotropes in the mixture.

All existing commercial processes for dehydrating ethanol by azeotropic distillation try to obtain pure ethanol. We showed recently[1] that it is possible to attain a "dry" mixture of ethanol + hydrocarbon directly while utilizing less energy. The ethanol + hydrocarbon mixture so obtained may be employed directly as gasoline. The feasibility of dehydrating ethanol to obtain a hydrocarbon−ethanol mixture using only one of the many hydrocarbons present in gasoline was assessed in previous studies.[2,3] To expand these studies to include mixtures of hydrocarbons requires knowledge of the vapor−liquid (VLE) and vapor−liquid−liquid equilibrium (VLLE) data of these water + ethanol + hydrocarbons systems.

Cyclohexane and isooctane were chosen for this purpose. Thus, experimental isobaric equilibrium data for the quaternary system water + ethanol + cyclohexane + isooctane at atmospheric pressure have been obtained.

Experimental Section

Chemicals used were the following: All the reagents were "for analysis" grade and acquired from Merck. The purity of ethanol, cyclohexane, and isooctane was higher than 0.995 (mass fraction), so no further purification was needed. The internal standard used for gas chromatography was 1-propanol. The water content (mass %) was determined by the Karl Fischer titration method, and for cyclohexane, isooctane, ethanol, and 1-propanol was around (0.004, 0.005, 0.04, and 0.07) wt %, respectively. The water used was purified using a Milli Q-Plus system.

Apparatus and procedures were as follows: An all-glass dynamic recirculating still equipped with an ultrasonic homogenizer (Braun Labsonic P) coupled to a boiling flask was used to determine the VLLE data. This commercially available apparatus (Labodest model 602) is assembled in Germany by Fischer Labor and Verfahrenstechnik and has been modified by Gomis et al.[4] The application of ultrasound to the boiling flask causes emulsification of the two liquid phases throughout the still and thus prevents the fluctuations in temperature and flow rate characteristic of two liquid phase systems. Visual observation confirmed that the emulsified state was maintained everywhere throughout the apparatus during operation. For VLE determinations, the apparatus was used without further modification since it permits good mixing and separation of the vapor and liquid phases once they achieve equilibrium. The experimental procedure of previous studies was followed.[2,3] Accordingly, only essential parts are given here.

A Pt100 sensor was employed to measure the equilibrium temperatures. The probe was connected to a Presys thermometer (model ST-501) with an uncertainty of 0.01 K according to the calibration certificate (ITS-90[5]). A Fischer M101 control system was used to measure and control the pressure as well as the

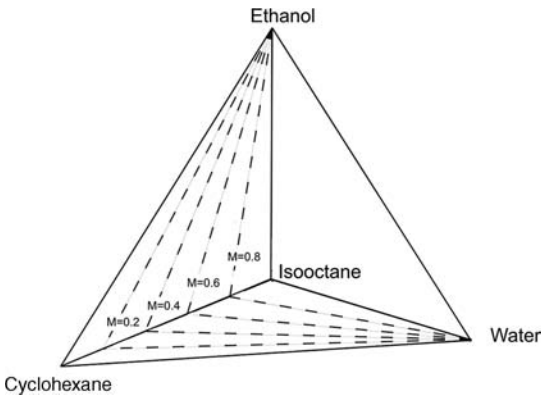

Figure 1. Tetrahedral representation. Sectional planes.

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Table 1. Vapor−Liquid−Liquid Equilibrium Data (Mole Fraction) for the Water (1) + Cyclohexane (3) + Isooctane (4) Ternary System at 101.3 kPa

aqueous			organic			vapor			
x_1	x_3	x_4	x_1	x_3	x_4	y_1	y_3	y_4	T_b/K
1.000	0.00001	0.00002	0.0010	0.707	0.292	0.326	0.588	0.087	344.25
1.000	0.00001	0.00001	0.0010	0.568	0.432	0.342	0.469	0.190	346.10
1.000	0.0001	0.00002	0.0011	0.389	0.610	0.360	0.338	0.302	348.40
1.000	0.00001	0.00005	0.0012	0.179	0.820	0.393	0.182	0.425	350.68

heating power. The pressure in the still was 101.3 kPa, measured and controlled to an accuracy of 0.1 kPa. To check that the apparatus operated properly, the boiling point of water was measured and compared to the recommended value in the bibliograpy.

Sampling was carried out in the following three different ways:

(a) Gaseous samples were injected into the GC via a UW Type, 6-port valve from Valco Instruments Co. The connecting tube walls were superheated with a resistance tape controlled by a potentiometer to ensure the vapor remained unsaturated and avoid condensation.

(b) For sampling of the liquid phase in the heterogeneous region, a small amount of the liquid coming from the separation chamber of the instrument was diverted to a tube by a solenoid valve. The tube was placed in a thermostatic bath at a temperature equal to the boiling point of the mixture. In this way, the dispersed liquid phases enter the tube and separate into two layers at their respective bubble points. A sample was taken from each layer and placed in a vial along with a small amount of 1-propanol as internal standard. Between equilibrium determinations, the drops of sample remaining in the feed line to the tube were flushed out using vacuum and external heating.

Table 2. Vapor−Liquid−Liquid Equilibrium Data (Mole Fraction) for the Water (1) + Ethanol (2) + Cyclohexane (3) + Isooctane (4) Quaternary System at 101.3 kPa

	aqueous				organic				vapor				
M^a	x_1	x_2	x_3	x_4	x_1	x_2	x_3	x_4	y_1	y_2	y_3	y_4	T_b/K
0.2	0.939	0.060	0.0002	0.00005	0.0011	0.010	0.756	0.232	0.244	0.168	0.510	0.078	340.01
	0.889	0.111	0.0002	0.00004	0.0016	0.014	0.695	0.290	0.221	0.219	0.484	0.076	338.61
	0.810	0.189	0.0007	0.0001	0.0024	0.036	0.749	0.212	0.203	0.257	0.467	0.073	337.65
	0.747	0.252	0.0013	0.0002	0.0030	0.042	0.739	0.216	0.198	0.271	0.457	0.074	337.33
	0.690	0.307	0.0023	0.0004	0.0052	0.058	0.706	0.231	0.191	0.283	0.451	0.074	337.14
	0.636	0.358	0.0051	0.0010	0.0046	0.066	0.688	0.241	0.185	0.294	0.446	0.076	337.01
	0.562	0.422	0.013	0.0026	0.0076	0.080	0.638	0.274	0.180	0.304	0.437	0.080	337.00
	0.528	0.452	0.018	0.0027	0.013	0.107	0.684	0.196	0.174	0.308	0.449	0.069	336.68
	0.473	0.491	0.031	0.0056	0.013	0.116	0.678	0.192	0.169	0.313	0.448	0.069	336.63
	0.421	0.528	0.042	0.0087	0.013	0.129	0.667	0.190	0.165	0.320	0.446	0.069	336.62
	0.373	0.560	0.055	0.012	0.021	0.157	0.640	0.183	0.166	0.331	0.435	0.068	336.65
	0.265	0.557	0.142	0.036	0.056	0.297	0.505	0.142	0.157	0.336	0.439	0.068	336.69
0.4	0.874	0.126	0.0001	0.00004	0.0018	0.021	0.535	0.442	0.237	0.246	0.366	0.152	339.73
	0.785	0.214	0.0004	0.0001	0.0036	0.042	0.521	0.433	0.212	0.287	0.347	0.154	338.94
	0.694	0.303	0.0020	0.0006	0.0028	0.052	0.512	0.432	0.203	0.307	0.339	0.151	338.54
	0.643	0.350	0.0048	0.0018	0.0050	0.061	0.482	0.452	0.199	0.312	0.340	0.148	338.32
	0.607	0.384	0.0069	0.0027	0.0089	0.074	0.484	0.433	0.197	0.320	0.339	0.143	338.19
	0.527	0.455	0.012	0.0052	0.0089	0.095	0.490	0.406	0.188	0.335	0.332	0.146	338.15
	0.457	0.512	0.021	0.011	0.016	0.133	0.451	0.399	0.181	0.345	0.326	0.147	338.11
	0.423	0.535	0.028	0.014	0.017	0.146	0.470	0.367	0.179	0.346	0.338	0.138	337.90
	0.367	0.575	0.036	0.022	0.036	0.197	0.420	0.346	0.173	0.355	0.333	0.139	337.93
	0.297	0.588	0.069	0.047	0.047	0.259	0.383	0.311	0.166	0.360	0.332	0.142	337.96
	0.313	0.591	0.058	0.038	0.043	0.240	0.397	0.321	0.168	0.361	0.330	0.141	337.98
0.6	0.858	0.142	0.0002	0.0002	0.0017	0.024	0.327	0.647	0.242	0.272	0.244	0.242	341.33
	0.834	0.165	0.0002	0.0002	0.0026	0.031	0.343	0.624	0.239	0.282	0.254	0.225	340.61
	0.768	0.231	0.0006	0.0005	0.0035	0.050	0.337	0.609	0.224	0.307	0.246	0.223	340.08
	0.708	0.290	0.0014	0.0010	0.0040	0.055	0.340	0.602	0.216	0.322	0.240	0.222	339.83
	0.668	0.328	0.0021	0.0016	0.0057	0.070	0.318	0.606	0.210	0.331	0.238	0.220	339.66
	0.607	0.386	0.0041	0.0032	0.0058	0.080	0.330	0.584	0.203	0.343	0.234	0.220	339.53
	0.549	0.439	0.0062	0.0054	0.0065	0.100	0.321	0.572	0.198	0.353	0.230	0.219	339.43
	0.495	0.485	0.010	0.010	0.013	0.118	0.309	0.560	0.191	0.362	0.227	0.219	339.39
	0.442	0.529	0.014	0.016	0.015	0.140	0.295	0.550	0.188	0.369	0.227	0.216	339.29
	0.373	0.571	0.024	0.032	0.022	0.183	0.274	0.521	0.180	0.381	0.223	0.217	339.31
	0.343	0.595	0.026	0.037	0.028	0.204	0.267	0.501	0.177	0.385	0.221	0.217	339.33
	0.310	0.599	0.039	0.052	0.033	0.239	0.265	0.464	0.174	0.388	0.223	0.215	339.34
	0.295	0.605	0.041	0.059	0.041	0.259	0.247	0.454	0.172	0.392	0.219	0.217	339.35
	0.259	0.602	0.056	0.083	0.054	0.319	0.227	0.401	0.168	0.397	0.219	0.216	339.38
0.8	0.898	0.101	0.0002	0.0008	0.0013	0.015	0.176	0.808	0.269	0.263	0.140	0.328	343.18
	0.853	0.146	0.0001	0.0001	0.0018	0.032	0.131	0.834	0.258	0.293	0.124	0.325	342.56
	0.828	0.171	0.0001	0.0003	0.0022	0.036	0.155	0.807	0.248	0.309	0.130	0.314	342.07
	0.737	0.261	0.0005	0.0010	0.0071	0.066	0.148	0.779	0.232	0.337	0.122	0.309	341.47
	0.689	0.309	0.0008	0.0018	0.0044	0.066	0.138	0.792	0.225	0.351	0.120	0.305	341.23
	0.609	0.385	0.0019	0.0040	0.0047	0.083	0.154	0.759	0.214	0.368	0.117	0.301	340.93
	0.570	0.421	0.0036	0.0048	0.0087	0.096	0.137	0.759	0.213	0.373	0.120	0.294	340.77
	0.540	0.449	0.0030	0.0083	0.013	0.126	0.133	0.727	0.208	0.380	0.116	0.296	340.76
	0.474	0.503	0.0070	0.016	0.013	0.143	0.125	0.719	0.201	0.391	0.107	0.301	340.73
	0.429	0.544	0.0045	0.023	0.021	0.188	0.121	0.671	0.196	0.401	0.105	0.298	340.72
	0.359	0.592	0.010	0.039	0.034	0.246	0.106	0.614	0.187	0.413	0.105	0.295	340.69
	0.314	0.613	0.014	0.059	0.034	0.246	0.106	0.614	0.181	0.421	0.102	0.295	340.72
	0.271	0.624	0.019	0.086	0.034	0.279	0.102	0.584	0.175	0.428	0.103	0.294	340.74
	0.245	0.626	0.021	0.107	0.079	0.390	0.082	0.450	0.174	0.429	0.107	0.290	340.75

[a] $M = x_4/(x_3 + x_4)$ in the initial mixture.

(c) For the homogeneous region, samples were withdrawn from the liquid coming from the separator chamber using a syringe and put into a vial along with a small amount of internal standard.

All analytical work was carried out by gas chromatography in a Shimadzu GC-14A coupled to a personal computer employing Shimadzu Labsolution GC-Solution software. Component separation was achieved in a 2 m × 3 mm column packed with Porapak Q 80/100. The oven temperature was 483.15 K. The helium flow rate was 50 $\text{mL}\cdot\text{min}^{-1}$.

Detection was carried out by various techniques, which depended upon the composition of the samples: thermal conductivity detection (TCD), for organic and aqueous samples (analysis for water, ethanol, cyclohexane, and isooctane), and flame ionization detection (FID), for aqueous samples (analysis for ethanol, cyclohexane and isooctane). The temperature of the detector was 493.15 K and the current reading on the TCD was 100 mA. The water in the organic phase was also determined by the Karl Fischer Titration method.

An internal standard was used to obtain quantitative results from the analysis of the liquid phases. For this reason, 1-propanol, which is completely miscible in water, ethanol, cyclohexane, and isooctane, was also added to the sample vials. Moreover, addition of the standard prevents phase splitting when adjusting the temperature after separation of the phases. The relative accuracy of the mole fraction measurements was approximately 2%.

With a view to obtaining experimental data for the entire region comprising quaternary mixtures (the region in which the liquid mixtures are heterogeneous at the bubble point as well as the one in which they are homogeneous), the liquid mixtures introduced in the vapor−liquid equilibrium apparatus at the start of every experiment were chosen to more or less lie in the planes shown in the tetrahedral diagram in Figure 1. All the points in a given plane represent mixtures of equal M, which is defined as $x_4/(x_3 + x_4)$, where x_3 and x_4 are the molar fractions of components 3 and 4, respectively.

Results and Discussion

The experimental isobaric VLLE data for the ternary system water (1) −cyclohexane (2) −isooctane (3) and the quaternary system water (1) −ethanol (2) −cyclohexane (3) −isooctane (4) at 101.3 kPa are reported in the Tables 1 and 2, respectively. VLE data for the homogeneous region of the quaternary system are shown in Table 3. The bubble point (T_b/K) and composition (mole fraction) of the liquid phases (x_i) and the vapor phases (y_i) are recorded in these tables.

Figure 2 shows the isobaric vapor−liquid−liquid equilibrium data for the ternary system. It also includes the binary heterogeneous azeotropes determined by Font et al.[2] and Verhoeye.[6] Tie-lines are shown connecting conjugate liquid phases; the vapor line is also shown. Numbers indicate the correspondence between equilibrium liquid and vapor phases. As can be seen, all the vapor phase composition points lie above the tie-lines connecting their corresponding equilibrium liquid phases. This confirms that a ternary heterogeneous azeotrope is not present.

Figure 3 shows the isobaric vapor−liquid−liquid equilibrium data for the quaternary system. It also shows the equilibrium data determined by Font et al.[2] and Gomis et al.[7] for those ternary systems comprising a pair of partially miscible compounds. The points plotted in Figure 3a represent the liquid phases; they define the bubble-point-temperature solubility surface. This surface (shaded in Figure 3a)

Table 3. Vapor−Liquid Equilibrium Data (Mole Fraction) for the Water (1) + Ethanol (2) + Cyclohexane (3) + Isooctane (4) Quaternary System at 101.3 kPa

	liquid				vapor				
M^a	x_1	x_2	x_3	x_4	y_1	y_2	y_3	y_4	T_b/K
0.2	0.146	0.459	0.315	0.080	0.151	0.337	0.445	0.067	336.73
	0.120	0.386	0.394	0.100	0.153	0.335	0.444	0.067	336.70
	0.074	0.583	0.271	0.072	0.079	0.409	0.443	0.069	337.91
	0.094	0.681	0.178	0.047	0.079	0.425	0.426	0.071	338.22
	0.203	0.594	0.161	0.042	0.139	0.354	0.433	0.075	337.12
	0.280	0.678	0.030	0.011	0.151	0.377	0.392	0.079	337.80
	0.323	0.660	0.012	0.005	0.157	0.411	0.350	0.082	339.19
	0.115	0.816	0.053	0.016	0.079	0.552	0.304	0.064	341.40
	0.078	0.863	0.046	0.014	0.058	0.666	0.226	0.050	343.25
	0.164	0.800	0.027	0.009	0.113	0.599	0.234	0.055	343.80
	0.293	0.678	0.022	0.007	0.166	0.495	0.272	0.067	342.13
0.4	0.169	0.498	0.193	0.140	0.155	0.375	0.326	0.144	338.04
	0.139	0.576	0.162	0.123	0.129	0.408	0.316	0.147	338.65
	0.117	0.605	0.157	0.122	0.113	0.433	0.304	0.150	339.04
	0.094	0.658	0.138	0.109	0.087	0.466	0.298	0.149	339.50
	0.078	0.736	0.103	0.084	0.065	0.514	0.274	0.147	340.50
	0.164	0.662	0.098	0.077	0.127	0.432	0.288	0.154	339.09
	0.234	0.636	0.076	0.053	0.148	0.418	0.277	0.157	339.12
	0.286	0.612	0.059	0.043	0.160	0.384	0.290	0.166	338.70
	0.245	0.736	0.010	0.009	0.162	0.591	0.147	0.101	344.60
	0.138	0.781	0.045	0.036	0.100	0.542	0.228	0.131	341.85
	0.074	0.874	0.028	0.024	0.049	0.673	0.176	0.101	344.46
	0.120	0.837	0.024	0.019	0.086	0.660	0.158	0.096	344.88
	0.082	0.314	0.356	0.248	0.155	0.367	0.341	0.137	337.88
	0.074	0.432	0.290	0.204	0.114	0.412	0.334	0.139	338.55
	0.069	0.543	0.229	0.159	0.083	0.448	0.328	0.141	339.08
0.6	0.054	0.448	0.178	0.321	0.096	0.469	0.219	0.216	340.29
	0.062	0.628	0.104	0.206	0.070	0.507	0.208	0.215	340.86
	0.161	0.591	0.088	0.160	0.148	0.428	0.204	0.220	339.79
	0.156	0.705	0.047	0.092	0.130	0.595	0.106	0.169	340.68
	0.167	0.775	0.020	0.039	0.126	0.577	0.116	0.182	343.61
	0.291	0.654	0.019	0.037	0.177	0.501	0.116	0.206	343.03
	0.340	0.634	0.009	0.016	0.193	0.527	0.095	0.185	343.37
	0.113	0.582	0.114	0.191	0.116	0.458	0.211	0.215	340.10
	0.180	0.598	0.080	0.142	0.148	0.426	0.204	0.222	339.78
	0.087	0.733	0.069	0.111	0.071	0.533	0.190	0.206	341.24
	0.080	0.797	0.049	0.074	0.058	0.591	0.162	0.189	342.50
	0.071	0.866	0.024	0.039	0.047	0.691	0.116	0.146	344.92
	0.217	0.680	0.041	0.062	0.140	0.471	0.172	0.217	341.02
0.8	0.233	0.695	0.000	0.072	0.158	0.450	0.100	0.292	341.92
	0.068	0.442	0.090	0.400	0.128	0.477	0.114	0.282	341.14
	0.054	0.568	0.066	0.311	0.078	0.530	0.107	0.285	341.82
	0.049	0.649	0.053	0.249	0.059	0.556	0.103	0.282	342.20
	0.058	0.704	0.044	0.193	0.063	0.565	0.095	0.278	342.35
	0.060	0.778	0.030	0.132	0.055	0.598	0.084	0.263	343.01
	0.052	0.858	0.016	0.074	0.044	0.677	0.062	0.217	344.61
	0.133	0.779	0.014	0.074	0.108	0.569	0.071	0.251	343.01
	0.149	0.706	0.024	0.121	0.127	0.503	0.089	0.281	341.68
	0.044	0.426	0.102	0.428	0.091	0.509	0.114	0.286	341.71
	0.035	0.541	0.079	0.344	0.060	0.545	0.109	0.285	342.16
	0.170	0.574	0.048	0.208	0.160	0.446	0.103	0.290	340.80
	0.157	0.620	0.042	0.181	0.146	0.467	0.098	0.288	343.13

[a] $M = x_4/(x_3 + x_4)$ in the initial mixture.

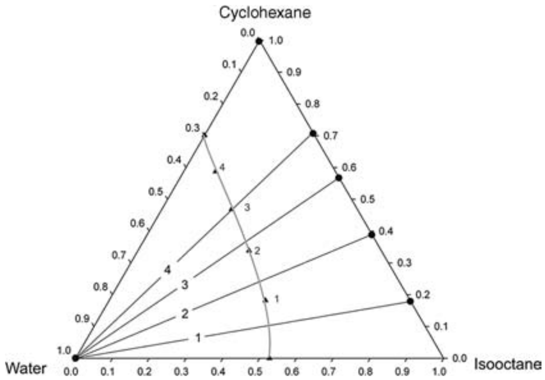

Figure 2. VLLE (mole fraction) diagram for the water (1) + cyclohexane (3) + isooctane (4) ternary system at 101.3 kPa. ●, liquid phase; ▲, vapor phase; −, tie line.

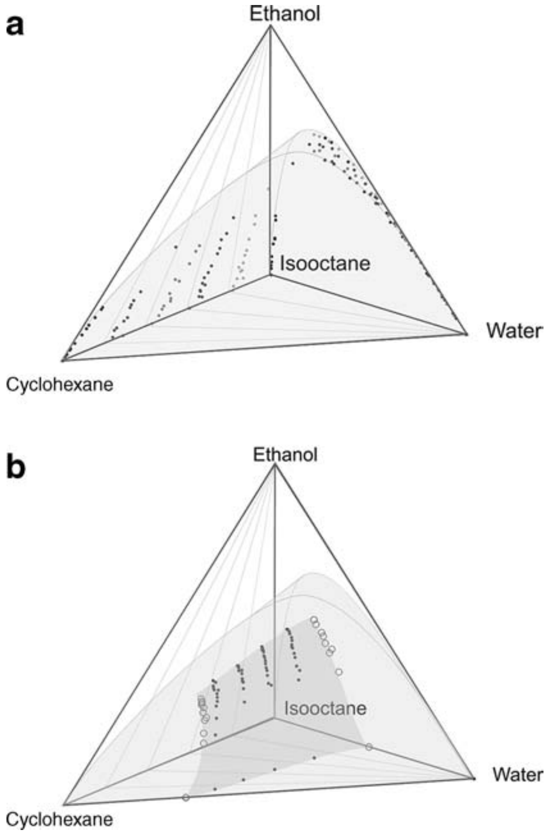

Figure 3. (a) VLLE data (mole fraction) for the quaternary system water (1) −ethanol (2) −cyclohexane (3) −isooctane (4) at 101.3 kPa. ●, liquid phase. (b) VLLE data (mole fraction) for the quaternary system water (1) −ethanol (2) −cyclohexane (3) −isooctane (4) at 101.3 kPa. ●, vapor phase.

envelopes the region in which the liquid mixtures are heterogeneous at the bubble point temperature. It occupies most of the tetrahedron as a result of the high immiscibility of organic compounds 3 and 4 in water. The points plotted in Figure 3b represent the compositions of the vapor phases in equilibrium with the heterogeneous liquid mixtures. They also define a surface, whose area has been shaded using a dark color. This surface is completely inside the heterogeneous liquid mixture region.

Figure 4 shows (for every sectional plane of the tetrahedron) pseudoternary representations of the tie-lines connecting the conjugated liquid phases and the vapor phases in equilibrium with the liquid phases. Here, numbers have also been used to indicate the correspondence between equilibrium liquid and vapor phases.

The VLLE data for the quaternary mixtures show that the tie-lines that connect the liquid phases more or less lie in the same sectional planes as those from which the initial mixtures were chosen. On the other hand, the equilibrium vapor phases occur outside these planes and always on the side favoring mixtures richer in cyclohexane. This confirms that a quaternary heterogeneous azeotrope is not present either.

PRO-VLE 2.0 was used to apply the test to the equilibrium data of the ternary system.[8] Since this program does not permit applying the test to mixtures of more than three components, a program was developed in Excel that would make it possible to apply the Wisniak[9] test to equilibrium data of quaternary systems. All the *L*/*W* values are between 0.97 and 1.00. The test did not reveal any significant inconsistency in the data. Vapor pressures for the four

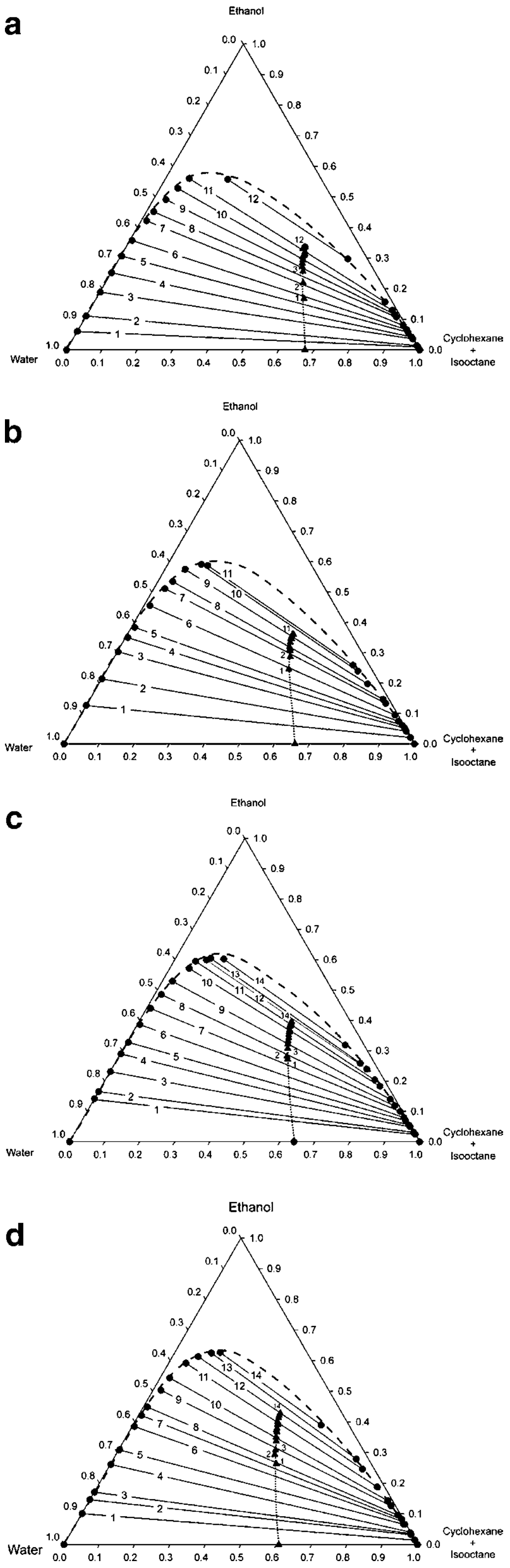

Figure 4. (a) Pseudoternary VLLE (mole fraction) representations for the water (1) + ethanol (2) + cyclohexane (3) + isooctane (4) quaternary system at 101.3 kPa at $M = 0.2$. ●, liquid phase; ▲, vapor phase; −, tie line. (b) Pseudoternary VLLE (mole fraction) representations for the water (1) + ethanol (2) + cyclohexane (3) + isooctane (4) quaternary system at 101.3 kPa at $M = 0.4$. ●, liquid phase; ▲, vapor phase; −, tie line. (c) Pseudoternary VLLE (mole fraction) representations for the water (1) + ethanol (2) + cyclohexane (3) + isooctane (4) quaternary system at 101.3 kPa at $M = 0.6$. ●, liquid phase; ▲, vapor phase; −, tie line. (d) Pseudoternary VLLE (mole fraction) representations for the water (1) + ethanol (2) + cyclohexane (3) + isooctane (4) quaternary system at 101.3 kPa at $M = 0.8$. ●, liquid phase; ▲, vapor phase; −, tie line.

Table 4. Antoine Equation Parameters[a] for the Pure Substances

compound	A	B	C	temperature range/K
water	7.196213	1730.63	−39.724	274.15−373.15
ethanol	7.237103	1592.864	−46.966	293.15−366.15
cyclohexane	5.976363	1206.47	−50.014	280.15−354.15
isooctane	5.936793	1257.84	−52.415	296.15−373.15

[a] Antoine Equation: $\log(P) = A - B/(T + C)$ with P/kPa and T/K.

components were calculated using the Antoine equation with parameters A_i, B_i, and C_i taken from the literature[10,11] and shown in Table 4.

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