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Multiphase Equilibrium Behavior of the Mixture Ethane + Methanol + 1-Decanol

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The liquid-liquid-vapor (LLV) phase equilibria of the ternary mixture ethane + methanol + 1-decanol were experimentally studied by using a visual cell (stoichiometric) technique with pressure discrimination of ±0.01 bar, achieved by coupling a dead-weight gauge to the cell pressure transducer. The pressure, phase compositions, and molar volumes of the two liquid phases of the ternary LLV system are reported at 295.15 K. Also presented are LLV phase equilibrium data for the binary mixture ethane + methanol. The ternary system has two constituent binary mixtures that exhibit LLV partial miscibility. The portion of the three-phase region of the ternary system examined is bounded by these binary LLV loci and from above by an upper critical end point (L-L=V) locus and from below by a lower critical end point (L=L-V) locus.

Introduction

During the past several years, our group has been engaged in the study of liquid-liquid-vapor (LLV) phase equilibria in well-defined systems such as carbon dioxide + hydrocarbon, ethane + hydrocarbon, and nitrous oxide + hydrocarbon binary mixtures, where the hydrocarbon has frequently been one of the members of the homologous series of *n*-paraffins or *n*-alkylbenzenes. The goals of these studies were to map out the patterns of the multiphase equilibria of these prototype mixtures in thermodynamic phase space and to generate phase equilibrium data that would support the prediction of phase equilibria within and near regions of LLV immiscibility. With the studies of these essentially nonpolar systems as a point of reference, we have undertaken a series of studies of the multiphase equilibria of binary mixtures composed of both polar and nonpolar species. Recent studies have reported the LLV immiscibility behavior of certain members of the homologous series of ethane + 1-alkanol (*1*), carbon dioxide + 1-alkanol (*2*), and nitrous oxide + 1-alkanol (*3*) binary mixtures. Of these binary mixture homologous series, only the ethane-containing system does not have a lower 1-alkanol limit on LLV immiscibility. The LLV behavior of this series at the higher carbon numbers is similar to those of the other homologous series of nitrous oxide + 1-alkanol and carbon dioxide + 1-alkanol mixtures, as well as for the several *n*-paraffin solute series that have been studied. (Teja et al. (*4*) have recently pointed out that the properties of the 1-alkanols converge to those of the corresponding alkanes as their chain lengths increase.) However, following a decrease in the extent of the LLV immiscibility loci down to 1-butanol in the ethane + 1-alkanol homologous series of binary mixtures, there is an increase in LLV immiscibility with lower carbon number down to methanol (*1*). Apparently, at the lower 1-alkanol carbon numbers in these ethane-rich mixtures, the solvent-free concentration of the hydroxyl groups increases to the point that it offsets the decreasing size of the hydrocarbon solutes, in turn promoting LLV immiscibility.

Of particular interest here, on the basis of this observation, is the LLV behavior of the ternary mixture ethane + methanol + 1-decanol. The binary constituent subsystems ethane + methanol and ethane + 1-decanol both exhibit sizable LLV extent. We examine herein whether ternary mixtures with methanol + 1-decanol solute ratios having proportions of carbon number-to-hydroxyl groups in the solute-rich L_1 phase characteristic of 1-propanol or 1-butanol will correspondingly display significantly reduced LLV extent (i.e., a maximum in the lower critical end point, or L=L-V, temperature function) as does the binary mixture ethane + 1-butanol in the homologous series. Ternary systems containing a gas solvent and two relatively nonvolatile solutes of differing molecular nature with each being immiscible with the solvent gas have been studied by Miller and Luks (*5*) and Kohn's group (*6*-*10*). Their LLV regions were generally found to be narrow surfaces located close to and just below the solvent gas vapor pressure curve when projected onto pressure-temperature coordinates.

Stoichiometric techniques are capable of measuring the molar volumes and compositions of all the fluid phases if very precise pressure measurements are made. This precision is required due to the rapid change of the phase properties over the narrow LLV pressure range at a fixed temperature. An earlier ternary study (*5*) coupled a differential pressure transducer with a dead-weight gauge to permit pressure measurements to a precision of ±0.007 bar. In this present study we found that it was adequate to calibrate the pressure transducer at the end of each run over the narrow pressure range of interest for that run (typically, 0.15 bar) to achieve a precision of no worse than ±0.01 bar, a magnitude more precise than we normally achieve in our phase equilibrium studies using a transducer alone. In this present study, this added precision was sufficient to give reproducible results.

The LLV phase equilibria of the ternary mixture ethane + methanol + 1-decanol was studied by using a visual cell

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("stoichiometric") experimental technique. We first experimentally mapped out the boundaries of the LLV region in the pressure-temperature space and then found the compositions and molar volumes of the two liquid phases along the 295.15 K isotherm in the ternary LLV region. The vapor phase was virtually pure ethane at 295.15 K.

We also report the temperature, pressure, phase compositions, and molar volumes of the two liquid phases along the LLV locus of the binary mixture ethane + methanol. An earlier study of the same binary mixture was performed by Ma and Kohn (*11*) between -40 and +100 °C, a broader temperature range than reported herein.

Experimental Apparatus and Procedure

A detailed description of the experimental apparatus is given in an earlier paper by Fall and Luks (*12*). The procedure for performing LLV studies was described in another paper by Fall et al. (*13*). These descriptions have been recently updated by Jangkamolkulchai and Luks (*14*). The experimental apparatus employs a stoichiometric approach wherein known amounts of methanol and 1-decanol are added to a volumetrically calibrated visual (glass) equilibrium cell. The total volume of the cell is typically 7-9 cm^3. The cell vapor space is thorougly flushed with ethane gas, after which measured amounts of the ethane gas are added to the cell from a high-pressure bomb. The cell contents are brought to equilibrium by a magnetically actuated steel ball stirrer mechanism. With the use of mass balances, the compositions and molar volumes of the three phases can be determined from the three "conjugate" measurements at a given temperature and pressure.

For a binary system, such as ethane + methanol, the mass balance calculations are based on a set of conjugate measurements in which each phase is respectively dominant in terms of its volumetric fraction. Specifically, L_1-, L_2- and V-dominant runs are made at a given temperature, a procedure that enables quantitative assessment of the presence of methanol in the vapor phase as well as in the liquid phases. (If one assumes that the solute is nonvolatile, the need for a V-dominant run is eliminated.)

The results of the binary studies of ethane + methanol (this study) and ethane + 1-decanol (*1*) indicate that the vapor phase of the ternary system contains negligible amounts of two alkanols at the temperature 295.15 K; thus, the vapor phase was assumed to be pure ethane gas, and its density was calculated (*15*) rather than measured.

The visual cell temperature was measured with a Pt-resistance thermometer to an estimated accuracy of ±0.02 K. For the binary study, pressures were measured to ±0.07 bar with pressure transducers that were frequently calibrated with a dead-weight gauge, while, for the ternary study, pressures were measured with a precision of at least ±0.01 bar with pressure transducers calibrated with a dead-weight gauge after each run over the appropriate pressure range, typically ±0.15 bar in breadth. Phase volumes in the calibrated visual cell were determined with the aid of a cathetometer to an accuracy of ±0.005 cm^3.

Materials

The *methanol* and 1-decanol used were purchased from Aldrich Chemical Co., both with a stated purity of 99+%, with H_2O contents of less than 0.005%. Chromatographic analysis indicated purities consistent with these stated purities. No further purification of the solutes was performed. During its use, the methanol was accessed by syringe through a barrier designed to protect it from hydroscopic contamination.

The ethane was obtained from the Matheson Co. as a Research Purity Grade gas with a minimum rating of 99.97 mol %. The primary impurity was ethylene, with smaller amounts of nitrogen and hydrogen being present as well. The ethane was first transferred to an initially evacuated storage bomb as a liquid at about 0 °C. In this two-phase condition, the vapor phase was vented to remove whatever light gas impurities might be present. Light gas impurities would have a more significant effect on the vapor pressure of ethane-rich phase equilibrium systems. Samples of the remaining ethane gas were liquefied in the visual cell at 30.00 °C, and the difference between the bubble point and dew point was found to be about 0.1 bar; the vapor pressure at this temperature was within ±0.07 bar of the data reported by Douslin and Harrison (*15*).

Table I. Temperature *T* and Pressure *P* Raw Data for the Liquid-Liquid-Vapor Boundary Loci of the Ternary Mixture Ethane + Methanol + 1-Decanol

UCEP		LCEP	
T/K	*P*/bar	*T*/K	*P*/bar
306.91[a]	49.94	275.31[a]	25.09
307.02	50.05	278.68	27.04
307.08	50.09	282.39	29.38
307.67	50.53	286.11	31.78
307.89	50.69	284.94	30.85
308.72	51.25	279.22	27.10
309.20	51.54	270.08	21.81
309.18[b]	51.34		

[a] Binary ethane + 1-decanol LLV system. [b] Binary ethane + methanol LLV system.

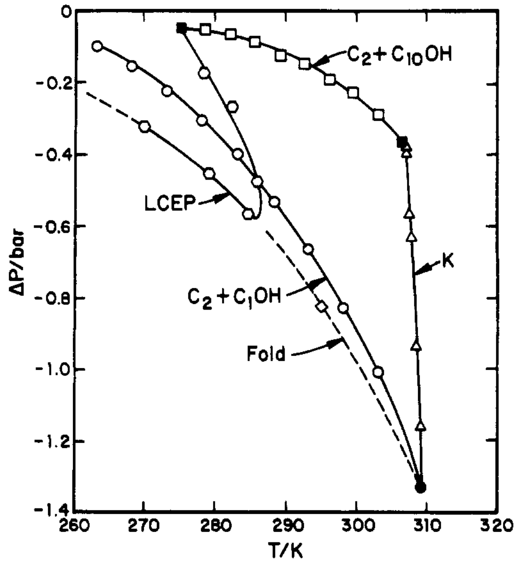

Figure 1. Liquid-liquid-vapor region of the system ethane (C_2) + methanol (C_1OH) + 1-decanol ($C_{10}OH$) in pressure-temperature space. Difference ΔP between the system pressure and the ethane vapor pressure (and its extrapolation, if necessary) as a function of temperature *T*.

Results

Table I presents pressure and temperature data for the lower critical end point (LCEP) (L=L-V) and upper critical end point (UCEP) (L-L=V) loci that bound the three-phase LLV region of the system ethane + methanol + 1-decanol. Figure 1 shows these data in "pressure difference"-temperature space, along with the constituent binary LLV loci; the pressure difference is the difference between the system pressure and the ethane vapor pressure (and its extrapolation, if necessary). The LCEP and UCEP data should be considered accurate to ±0.05 K and ±0.03 bar.

The LLV data for the ethane + methanol + 1-decanol system at 295.15 K are presented in Table II. Figure 2 is a plot of the ethane composition of the L_1 and L_2 phases versus

Table II. Liquid Phase L_2 and L_1 Compositions (Mole Fractions x) and Molar Volumes v as a Function of Pressure P for the Liquid-Liquid-Vapor Region of Ethane + Methanol + 1-Decanol at 295.15 K

L₁ phase				L₂ phase			
P/bar	$x(C_2H_6)$	$x(CH_3OH)$	v/(cm³/mol)	P/bar	$x(C_2H_6)$	$x(CH_3OH)$	v/(cm³/mol)
38.603[a]	0.3779	0.6221	52.0	38.603[a]	0.9179	0.0821	81.4
38.514	0.4021	0.5926	53.4	38.617	0.8679	0.1321	78.7
38.486	0.4342	0.5536	55.7	38.631	0.8885	0.1099	79.2
38.493	0.4770	0.5017	58.3	38.707	0.9132	0.0778	80.9
38.548	0.5552	0.4017	64.3	38.879	0.9419	0.0492	82.4
38.610	0.5968	0.3427	67.9	39.038	0.9659	0.0279	84.7
38.700	0.6295	0.2889	71.6	39.134	0.9792	0.0163	85.8
38.762	0.6519	0.2453	74.9	39.162[b]	0.9932	0.0000	88.7
38.831	0.6663	0.2112	78.1				
38.893	0.6781	0.1826	80.8				
38.920	0.6810	0.1766	81.3				
38.982	0.6929	0.1502	83.2				
39.065	0.7248	0.0714	91.0				
39.162[b]	0.7547	0.0000	97.7				

[a] Binary ethane + methanol LLV system. [b] Binary ethane + 1-decanol LLV system.

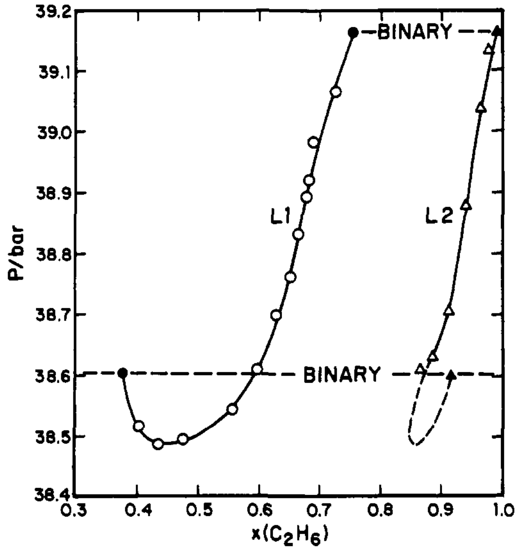

Figure 2. Ethane mole fraction $x(C_2H_6)$ as a function of pressure P for the L_1 and L_2 phases for the liquid-liquid-vapor region of ethane + methanol + 1-decanol at 295.15 K.

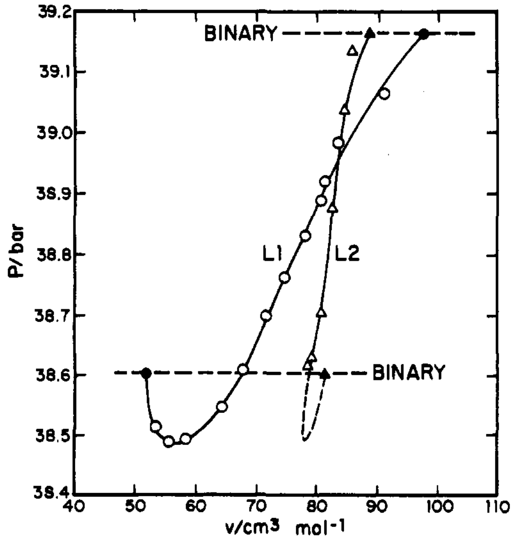

Figure 3. Molar volume v as a function of pressure P for the L_1 and L_2 phases for the liquid-liquid-vapor region of ethane + methanol + 1-decanol at 295.15 K.

Table III. Liquid Phase L_1 and L_2 Compositions on an Ethane-Free Basis as a Function of Pressure P for the Liquid-Liquid-Vapor Region of Ethane + Methanol + 1-Decanol at 295.15 K

L₁ phase			L₂ phase		
P/bar	X-(CH_3OH)	X-($C_{10}H_{21}OH$)	P/bar	X-(CH_3OH)	X-($C_{10}H_{21}OH$)
38.603[a]	1.0000	0.0000	38.603[a]	1.0000	0.0000
38.514	0.9912	0.0088	38.617	1.00[c]	0.00
38.486	0.9784	0.0216	38.631	0.9957	0.0143
38.493	0.9593	0.0407	38.707	0.9865	0.1035
38.548	0.9030	0.0970	38.879	0.8467	0.1533
38.610	0.8499	0.1501	39.038	0.8174	0.1826
38.700	0.7798	0.2202	39.134	0.7852	0.2148
38.762	0.7048	0.2952	39.162[b]	0.0000	1.0000
38.831	0.6329	0.3671			
38.893	0.5674	0.4326			
38.920	0.5535	0.4465			
38.982	0.4891	0.5109			
39.065	0.2594	0.7406			
39.162[b]	0.0000	1.0000			

[a] Binary ethane + methanol LLV system. [b] Binary ethane + 1-decanol LLV system. [c] No 1-decanol was detectable in the L_2 phase by stoichiometric means at this pressure.

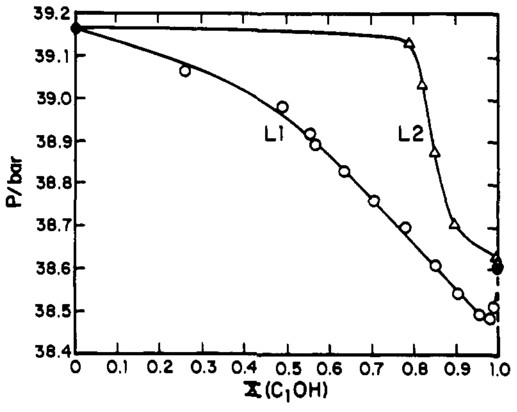

Figure 4. Methanol mole fraction on an ethane-free basis $X(C_1OH)$ as a function of pressure P for the liquid-liquid-vapor region (phases L_1 and L_2 only) of ethane + methanol + 1-decanol at 295.15 K.

pressure, while Figure 3 presents the molar volumes of the two liquid phases as a function of pressure. The ternary mole fractions are estimated to be accurate to ±0.005 and the liquid phase molar volumes to ±1 cm³/mol. These are conservative estimates, based primarily on average absolute deviation (AAD) of the raw data from smoothed curves as well as our experience at being able to generate reproducible data using the stoichiometric technique described herein. The uncertainties suggested here are slightly larger than we have normally encountered in earlier studies, which is attributable to the difficulty of taking reproducible data on this system with its rapidly changing properties over its narrow pressure range.

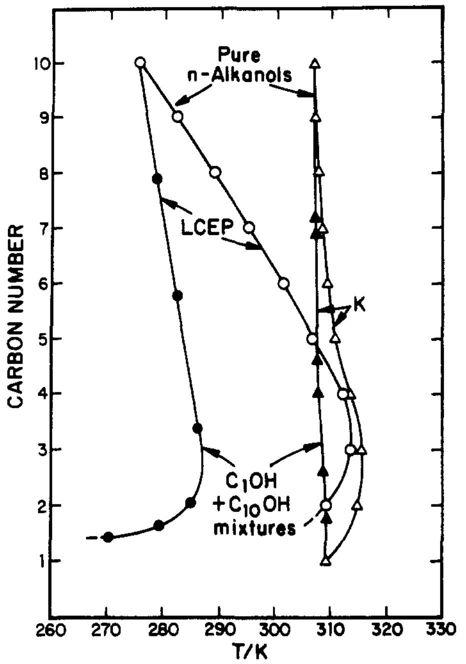

Figure 5. Ternary LLV region bounds (filled symbols) in temperature T-carbon number coordinates for certain solute molar mixture proportions of methanol + 1-decanol, compared to the LLV bounds (open symbols) of members of the homologous series of binary ethane + 1-alkanol mixtures.

Table IV. Liquid Phase L_1 and L_2 Composition and Molar Volume v for the Liquid-Liquid-Vapor Locus of the Binary System of Ethane + Methanol

		L_2 phase		L_2 phase	
T/K	P/bar	$x(C_2H_6)$	v/(cm^3/mol)	$x(C_2H_6)$	v/(cm^3/mol)
263.15	18.25	0.4275	50.6	0.8930	66.4
268.15	20.68	0.4243	51.2	0.8921	67.7
273.15	23.33	0.4193	51.5	0.8933	69.4
278.15	26.23	0.4119	51.8	0.8974	71.3
283.15	29.39	0.4039	51.8	0.8991	73.7
288.15	32.78	0.3936	51.9	0.9056	76.3
293.15	36.51	0.3822	51.9	0.9128	79.8
298.15	40.53	0.3705	51.9	0.9244	84.3
303.15	44.88	0.3602	51.7	0.9371	90.6
309.18	51.34^a	0.3476^b	51.5^b		

^{a} UCEP (L=L-V). ^{b} Values obtained by extrapolation.

Table III and Figure 4 present the methanol mole fractions *on an ethane-free basis* as a function of pressure for the ternary system. The binary data appear in Figures 2-4 as filled symbols.

Figure 5 shows the LLV occurrence in temperature-carbon number coordinates for certain ethane + 1-alkanol homologous series of mixtures (*1*); the filled symbols are plotted at carbon numbers appropriate for ethane-free mixtures of methanol and 1-decanol for the ternary system in the L_1 phase.

Table IV presents raw data for the LLV locus of the mixture ethane + methanol, including compositions and molar volumes of each of the liquid phases and the vapor phase. Figures 6 and 7 show the L_1 and L_2 compositions and molar volumes plotted versus temperature. The mole fractions reported for this binary system should be good to ±0.001, and the molar volumes should be good to ±0.5 cm^3/mol. These are conservative estimates based on the average absolute deviations (AAD) of the raw data from the smoothed curves shown in the figures. The AAD's for the ethane compositions in both liquid phases for the different LLV studies were less than 0.001 while the corresponding AAD's for the molar volumes ranged from 0.1 to 0.5 cm^3/mol. Our ethane + methanol binary data in Table I compare favorably with that of Ma and Kohn (*11*) for the L_1 phase but are slightly higher in composition for the L_2 phase. Ma and Kohn did not present LLV molar volume data.

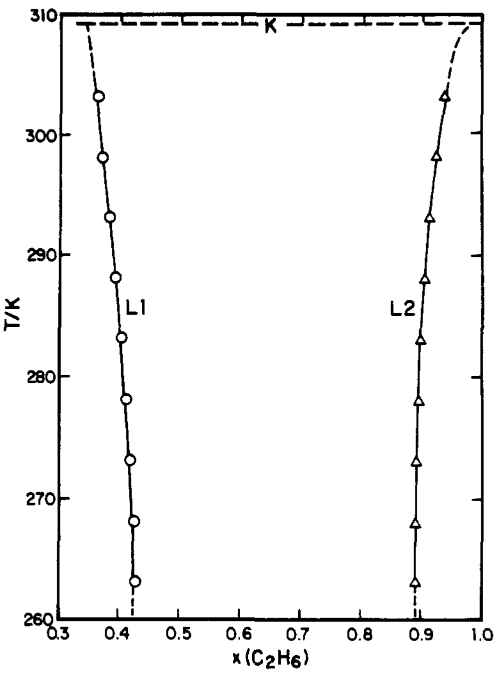

Figure 6. Ethane mole fraction $x(C_2H_6)$ as a function of temperature T for the L_1 and L_2 phases for the liquid-liquid-vapor region of the binary mixture ethane + methanol.

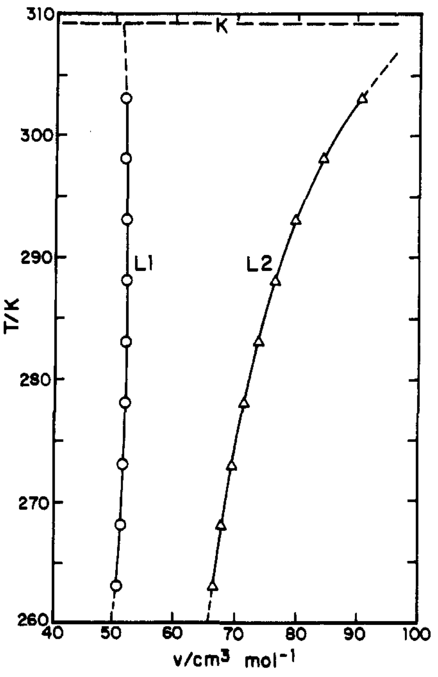

Figure 7. Molar volume v as a function of temperature T for the L_1 and L_2 phases for the liquid-liquid-vapor region of the binary mixture ethane + methanol.

Discussion

In this study, we identified the boundaries of the LLV surface of the ternary system ethane + methanol + 1-decanol (Figure 1). The surface extends between the binary LLV locus of ethane + 1-decanol and the binary LLV locus of ethane + methanol. The LCEP locus exhibits a prominent temperature maximum (Figure 5). The extremum in temperature indicates that the polar-nonpolar interaction becomes more predominant as methanol concentrations increase. The LCEP behavior seen in this ternary system mimics the LCEP behavior of the binary homologous series of ethane + 1-alkanol mixtures from carbon numbers 1 to 10.

In contrast, the volatilities of methanol and 1-decanol and its mixtures are less than that of 1-propanol or 1-butanol. Thus we do not see the pronounced maximum in the UCEP (*K* point) temperatures for the ternary mixtures that we saw with the homologous series of binary mixtures (*10*), also illustrated in Figure 5.

A "fold" in the LLV surface can be observed in Figures 1–4. This fold is associated with a pressure minimum. There is more than one three-phase state at a given temperature and pressure where the surface overlaps itself. Merrill et al. (*16*) mentioned that there was probably a (small) fold in the LLV surface of the system nitrogen + methane + *n*-butane. Recent studies by our group on carbon dioxide + ethane + *n*-tetradecylbenzene (*17*) and carbon dioxide + *n*-butylbenzene + *n*-eicosane (*5*) report more prominent LLV surface folds. Miller and Luks (*18*) mathematically analyzed these folds and drew general conclusions about species separability between the fluid phases present.

Figure 4 illustrates the shape of the envelope of consolute points at 295.15 K for the L_1 and L_2 phases, where a consolute point is the three-phase analogue of a two-phase saturation point. In L_2-dominant runs for initial methanol mole fraction loadings of the order of 0.977 or greater in our experiments, the ethane-rich phase (L_2) selectively extracted enough methanol from the L_1–solute mixture to make it difficult to obtain L_2 compositions or molar volumes in the region of the fold. These portions of the L_2 data are represented by dashed curves, which are meant to imply (only) their existence.

Glossary

AAD	average absolute deviation
K	denotes a *K* point (herein, the UCEP) where L–L=V
LCEP	lower critical end point, or L=L–V
L, L_1, L_2	liquid phases
L–L=V	a three-phase critical end point where the less dense L phase and the V phase are critically identical; also the UCEP (*K* point)
L=L–V	a three-phase critical end point where the two liquid phases are critically identical; also the LCEP
P	pressure
T	temperature
UCEP	upper critical end point, or L–L=V (herein, the *K* point)
v	molar volume of a phase
V	gas phase

Registry No. C_2H_6, 74-84-0; CH_3OH, 67-56-1; 1-decanol, 112-30-1.

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Vapor–Liquid Equilibria of the 1,1,1-Trichloroethane–1-Propanol System at 101.3-kPa Pressure

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The *T–x* data for 1,1,1-trichloroethane–1-propanol mixtures have been measured ebulliometrically at 101.3-kPa pressure. The system shows positive deviations from Raoult's law, forming a minimum boiling azeotrope. The vapor compositions have been predicted by using UNIQUAC and CLC models.

As part of a continuing study of the vapor liquid equilibria of alcohol–chlorohydrocarbon mixtures, the equilibrium temperature–composition data of 1,1,1-trichloroethane–1-propanol mixtures at 101.3-kPa pressure were measured in an all-glass ebulliometer.

Experimental Section

Purity of Chemicals. Reagent grade 1,1,1-trichloroethane, supplied by BDH Chemicals Ltd., Poole, England, and AnalaR grade 1-propanol, supplied by Renaal, Budapest, Hungary, were further purified by distilling in a laboratory packed column. The glass distillation column was of 4-cm diameter and 30-cm length and was packed with 0.4-cm-diameter and 0.7-cm-length hollow cylindrical glass packing. The fraction collected at the boiling point for each chemical was used in the study. The physical properties of the chemicals are listed in Table I, along with the literature (*1–4*) values for comparison.

Equilibrium Still. The equilibrium temperature–liquid composition data were measured in an all-glass Swietoslawski type ebulliometer of 120-cm^3 capacity that was standardized earlier by measuring the *T–x* data of the benzene–cyclohexane system at 101.3-kPa pressure and comparing the data with that reported in the literature (*5*).

The details of the experimental setup of the ebulliometer and its operation are described elsewhere (*6*, *7*). A solution of known composition was prepared by weighing on a precision

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