Effect Of Polar Component(1-Propanol) On The Relative Volatility Of The Binary System *N*-Hexane - Benzene

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Abstract:

Vapor-liquid equilibrium data are presented for the binary systems *n*-hexane - 1-propanol, benzene - 1-propanol and *n*-hexane – benzene at 760 mm of mercury pressure. In addition ternary data are presented at selected compositions with respect to the 1-propanol in the 1-propanol, benzene, *n*-hexane system at 760 mmHg. The results indicate the relative volatility of *n*-hexane relative to benzene increases appreciably with addition of 1-propanol.

Keywords: Vapor-Liquid Equilibria, Relative volatility, Polar component.

Introduction

The rapid growth of the petrochemical industry has led to the wide application of extractive distillation as a means of separating closely boiling compounds. One of the problems in the field of extractive distillation is to find a quantitative method of assessing solvents, in terms of the physical properties of the constituents, in order to select the most efficient solvent for a particular process. The aim of the present study was to determine experimentally the effect of polar components on the relative volatility of binary systems. The binary system studied in this work was composed of *n*-hexane and benzene. These hydrocarbons are difficult to separate because of closeness of boiling points. 1-propanol was used as a solvent. Vapor-Liquid Equilibria of the binary systems *n*-hexane – benzene, *n*-hexane - 1-propanol, benzene - 1-propanol, and of the ternary system *n*-hexane – benzene-1-propanol was determined at 760 mm of mercury absolute, using a modified Colburn recirculating still. The change in relative volatility of *n*-hexane relative to benzene, in the presence of 1-propanol, was calculated.

Experimental Section Chemicals

n-hexane, 99 mole % (min.) grade, was obtained from Phillips Petroleum Co., the reagent grade benzene was obtained from Merck and Co., and the Baker analyzed reagent grade 1-propanol was obtained from Baker Chemical Co. Table 1 compares the literature (Marc, 1998) and experimental values of physical properties of these materials.

Table 1 Properties of Materials

Physical Property	n-Hexane		Benzene		1-Propanol	
	Experimental	Literature	Experimental	Literature	Experimental	Literature
Refractive Index n_D^{30}	1.36996	1.36949	1.49469	1.49460	1.38146	1.38160
Density, ρ^{30} g/cm^{-3}	0.65043	0.65026	0.86839	0.86829	0.7962	0.7960
Boiling point, 760 mmHg, ^{o}C	68.8	68.74	80.1	80.1	97.25	97.29
Antoine Constants, where $log\,P = A – [B / (C + t)]$, P = mmHg, t = ^{o}C						
A	6.87773		6.89745		7.99733	
B	1171.53		1206.35		1569.70	
C	224.366		220.237		209.5	

Apparatus

A modified Colburn recirculating equilibrium still Fig 1 was used to obtain the vapor-liquid equilibrium data. The modifications and the general procedure have been reported in (Colburn, A.P., 1984). The mixture is introduced into the equilibrium apparatus via the filler tube of the storage vessel, where the liquid mixture in the reservoir is heated to boiling by a housing heating mantle. The circulation caused by vapor bubbles ensures even heating and mixing, where the heating mantle ensures that the number of vapor bubbles remains constant and that the mixture is heated uniformly. The ascending vapor bubbles thoroughly mix the entire volume of liquid, thus preventing a concentration gradient from forming and the boiling liquid from over-heating.

When the vapor bubbles (A) pass the funnel-shaped constriction of the Cotrell pump they carry a large quantity of non-vaporized liquid (B) to the Phase divider (phase separator). Here, the vapor-liquid mixture pours over the thermocouple protection tube. The splashguard which becomes wider higher up, prevents liquid splashes from being vaporized, which would complicate establishment of stationary equilibrium. To prevent partial condensation on the glass walls of the phase divider (phase separator) the equilibrium apparatus is equipped with an evacuated glass mantle consisting of two panes. In the phase divider the vapor phase (A) and the liquid phase (B) are separated. The liquid phase (B) drains off laterally to the sampling port at which it can be sampled through the septum of the screw cap without having to open the apparatus. Sampling via a glass syringe also prevents contamination of the sample. To condense the vapor residues present in the liquid phase (B), the outlet flows into a small vessel, on which a Liebig cooler is mounted.

The vapor (A) flows through the phase divider and a lateral outlet to the Dimroth cooler where it condenses. The condensate (A) drips down in a curved tube, which also terminates at a sampling port. The liquid (B) and condensate reflux (A), each arms fitted with a siphon, flow into a common tube. This enables remixing of the two phases before they are returned to the reservoir. The tube extends into the reservoir and ends inside it with its opening facing upward. The liquid rising in the middle of the reservoir draws the returned sample out of the tube and heats it.

Temperature were measured using a Copper – Constantan thermocouple and a Type K Leeds & Northrup potentiometer. A Bausch & Lomb precision refractometer was used to measure the refractive index of the vapor and liquid samples using a sodium D line light source. A Cottrell boiling point apparatus was used to check the purity of the compounds and to calibrate the equilibrium still thermocouple. The accuracy of the refractometer was tested by the test pieces supplied by Bausch & Lomb Inc. Pressure was measured to within ± 0.5 mm of mercury using a calibrated mercury manometer.
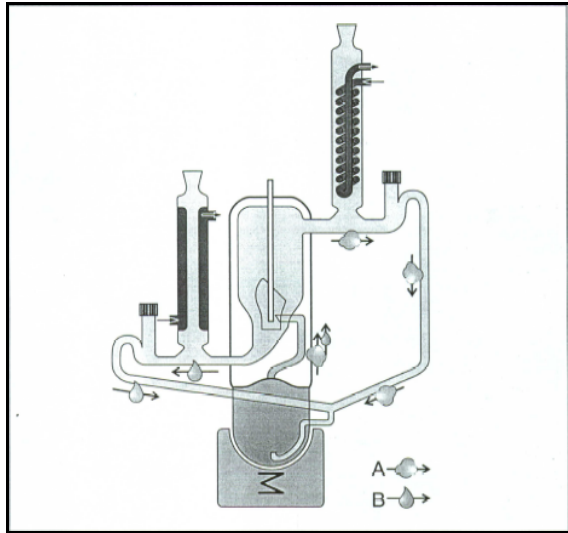
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Fig.1. Flow diagram for equilibrium apparatus.

Procedure

The procedures for determining vapor-liquid equilibrium data for the three binary systems were essentially those described below. For each of the binary hydrocarbon systems, refractive index calibration curves were obtained with samples of 12 to 15 different known concentrations at 30 ^{o}C. The compositions of vapor and liquid samples were read from the calibration curves. In the case the ternary system, three mixtures of n-hexane – benzene in the mole ratios of 25 to 75, 50 to 50, and 75 to 25 were used. 1-Propanol was added to each of the above mixtures to give equivalent mole fractions of 0.33, 0.50, 0.67, 0.75, and 0.80 in the ternary mixture. These mixtures were subjected to equilibrium distillation in the modified Colburn recirculating still and the vapor and liquid samples were obtained. The 1-propanol in these samples was extracted with water. The hydrocarbon layer was dried overnight by adding crystals of Drierite, which removed any traces of water remaining in the hydrocarbon mixture. The composition of the added agent-free samples was determined in the refractometer maintained at 30 ^{o}C.

Discussion Of Results

Experimental activity coefficients for the components in the binary mixture were calculated by the following equation (Prausnitz et.al., 1980).

$$\gamma_i = \frac{\phi_i \, y_i \, P_T}{x_i \, P_i} \tag{1}$$

The fugacity coefficient ϕ_i, was calculated using the PR equation of state, which have the form (Sytryjeck et.al., 1986):

$$\ln \alpha = m\left[1 - \frac{T}{T_c}\right] + n\left[1 - \sqrt{\frac{T}{T_c}}\right]^2 \tag{2}$$

Where m and n are two empirical factors for each pure component, their values given in Table 2 (Sytryjeck et.al., 1986).

Table 2. m and n (PR) factors equation of state

compound	PR – EOS	
	m	n
1-Propanol	1.1505	0.8075
Hexane	0.7939	0.4116
Benzene	0.6671	0.4723

$$a_{ij} = \left[1 - k_{a_{ij}} + \left(k_{a_{ij}} - k_{a_{ji}}\right)x_i\right]a_{ij}^o \tag{3}$$

$$a_{ij}^o = \sqrt{(a_i \alpha_i)(a_j \alpha_j)} \quad , \qquad A = \sum_i \sum_j x_i x_j a_{ij} \tag{4}$$

$$b_{ij} = \left[1 - k_{b_{ij}} + \left(k_{b_{ij}} - k_{b_{ji}}\right)x_i\right]\left(\frac{b_i^{1/3} + b_j^{1/3}}{2}\right)^3 \tag{5}$$

$$B = \sum_i x_i B_i \tag{6}$$

$$\ln \Phi_i = \frac{B'_i}{b}(Z-1) - \ln(Z-B) - \frac{A}{2.828B}\left[\frac{2}{a}\sum_j x_j a_{ij} - \frac{B'_i}{b}\right]\ln\left(\frac{Z+2.414B}{Z-0.414B}\right) \tag{7}$$

Where

$$B'_i = \sum_{j=1}^n x_j \left(b_{ij} + b_{ji}\right) - \sum_{j=1}^n \sum_{k=1}^n \left[x_j^2 x_k \left(k_{b_{jk}} - k_{b_{kj}}\right)\left(\frac{b_j^{1/3} + b_k^{1/3}}{2}\right)^3\right]$$
$$+ x_i \sum_{j=1}^n \left[x_j \left(k_{b_{ij}} - k_{b_{ji}}\right)\left(\frac{b_i^{1/3} + b_j^{1/3}}{2}\right)^3\right] - b \tag{8}$$

The vapor -liquid equilibrium data for the three binaries are reported in Tables 3 to 5 and shown graphically in Figures 2 to 7. Our results are compared to literature data (Chen, S. et.al., 2003)

Table 3 Vapor–liquid equilibrium data for Hexane–Benzene system at 760 mmHg.

Temp. °C	Experimental				Calculated	
	x_H	y_H	γ_H	γ_B	γ_H	γ_B
77.6	0.073	0.140	1.46	1.00	1.53	1.00
75.1	0.172	0.268	1.28	1.03	1.36	1.02
73.4	0.268	0.376	1.22	1.05	1.25	1.05
72.3	0.372	0.460	1.12	1.11	1.16	1.08
70.9	0.462	0.540	1.09	1.15	1.11	1.12
70.1	0.585	0.644	1.06	1.18	1.06	1.18
69.4	0.692	0.725	1.03	1.26	1.03	1.24
69.1	0.792	0.807	1.01	1.32	1.01	1.29
69.0	0.828	0.838	1.00	1.34	1.01	1.32
68.9	0.883	0.888	1.00	1.35	1.00	1.35
68.8	0.947	0.950	1.00	1.36	1.00	1.39
68.8	0.962	0.964	1.00	1.36	1.00	1.40

Table 4 Vapor-liquid equilibrium data for Hexane–1-propanol system at 760 mmHg.

Temp. °C	Experimental				Calculated	
	x_H	y_H	γ_H	γ_{1-P}	γ_H	γ_{1-P}
89.6	0.024	0.256	6.04	1.03	6.17	1.00
82.3	0.060	0.490	5.46	1.01	5.25	1.01
74.6	0.144	0.662	3.67	1.01	3.74	1.05
71.9	0.236	0.728	2.81	1.02	2.73	1.13
71.2	0.262	0.716	2.53	1.15	2.52	1.16
70.7	0.370	0.760	1.97	1.20	1.90	1.34
68.4	0.476	0.786	1.67	1.39	1.53	1.55
67.7	0.620	0.800	1.33	1.85	1.32	2.01
67.0	0.752	0.836	1.18	2.41	1.09	2.63
66.4	0.784	0.856	1.18	2.49	1.06	2.82
66.2	0.904	0.916	1.10	3.30	1.01	3.71
65.8	0.954	0.952	1.10	3.85	1.00	4.17
67.2	0.975	0.97	1.05	4.32	1.00	4.40

Table 5 Vapor-liquid equilibrium data for Benzene –1-propanol system at 760 mmHg.

| Temp. °C	Experimental				Calculated	
	x_B	y_B	γ_B	γ_{1-P}	γ_B	γ_{1-P}
92.8	0.049	0.142	1.99	1.07	2.34	1.00
88.4	0.104	0.296	2.22	1.12	2.21	1.01
84.8	0.180	0.436	2.10	1.14	2.04	1.02
82.1	0.254	0.530	1.97	1.17	1.88	1.04
79.7	0.398	0.622	1.62	1.33	1.61	1.13
77.4	0.504	0.680	1.47	1.46	1.44	1.24
76.5	0.642	0.728	1.27	1.78	1.25	1.49
76.2	0.764	0.774	1.15	2.31	1.12	1.94
76.1	0.792	0.776	1.11	2.59	1.10	2.11
76.3	0.834	0.812	1.10	2.70	1.06	2.39
76.9	0.916	0.864	1.04	3.75	1.02	3.24
78.2	0.956	0.916	1.01	4.16	1.01	3.89
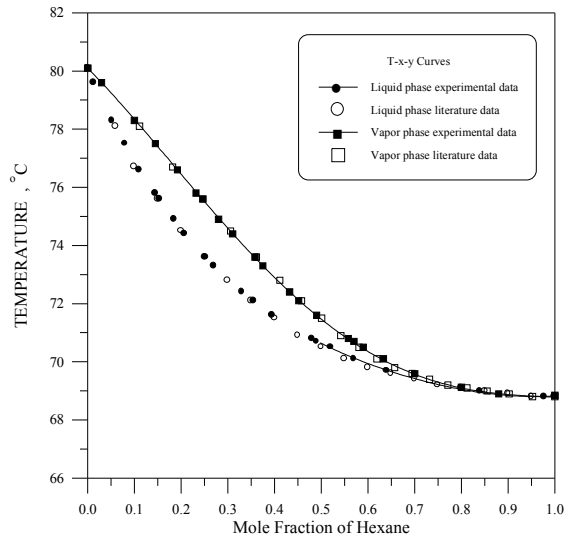
Fig.2. Boiling Point - Composition Curve for Hexane – Benzene at 760 mmHg.
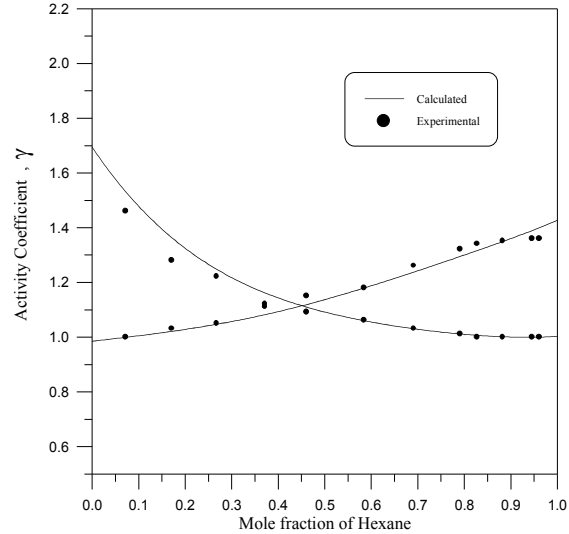

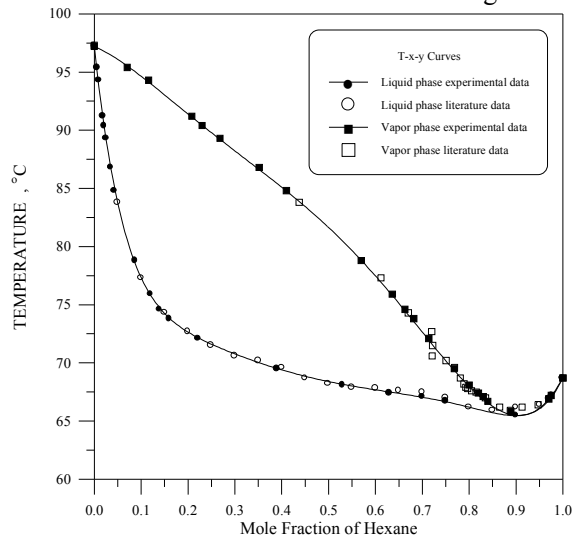Fig.3. Activity Coefficient - Composition Curve for Hexane – Benzene at 760 mmHg.

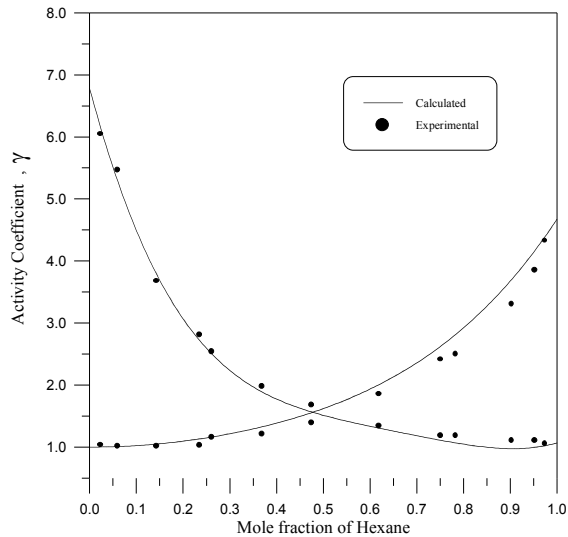Fig.4. Boiling Point - Composition Curve for Hexane – 1- Propanol at 760 mmHg.

Fig.5. Activity Coefficient - Composition Curve for Hexane – 1- Propanol at 760 mmHg.
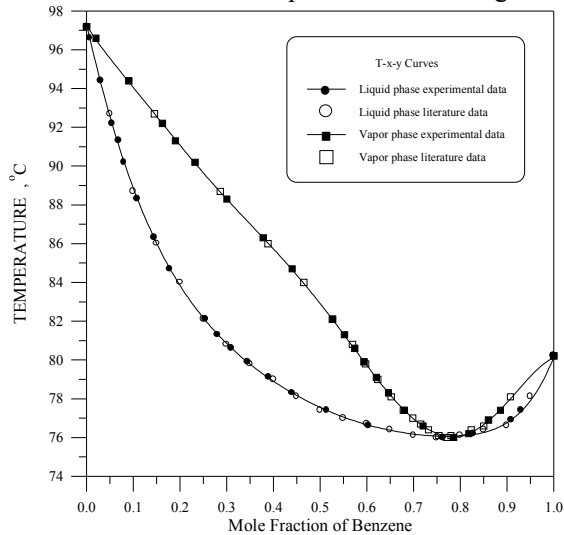

Fig.6. Boiling Point - Composition Curve for Benzene – 1- Propanol at 760 mmHg.
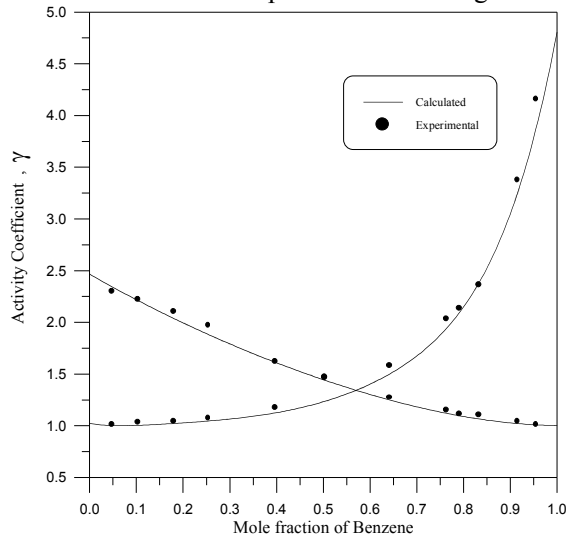

Fig.7. Activity Coefficient - Composition Curve for Benzene – 1- Propanol at 760 mmHg.

The data were correlated by the NRTL activity coefficient equations (Chen, S. et.al., 2003).

$$\ln \gamma_1 = x_2^2 \left[\tau_{21} \left(\frac{G_{21}}{x_1 + x_2 G_{21}} \right)^2 + \frac{G_{12} \tau_{12}}{(x_2 + x_1 G_{12})^2} \right] \quad (9)$$

$$\ln \gamma_2 = x_1^2 \left[\tau_{12} \left(\frac{G_{12}}{x_2 + x_1 G_{12}} \right)^2 + \frac{G_{21} \tau_{21}}{(x_1 + x_2 G_{21})^2} \right] \quad (10)$$

$$G_{12} = \exp(-\alpha \, \tau_{12}) \quad (11)$$

$$G_{21} = \exp(-\alpha \, \tau_{21}) \quad (12)$$

The values of the constants in the correlation were evaluated by using the *maximum likelihood principle* method providing a mathematical and computational guarantee of global optimality in parameter estimation that provides the best fit to measured data. The objective function in nonlinear parameter estimation problems is given below (Anderson et.al., 1978; Prausnitz et.al., 1980):

$$S = \sum_{i=1}^{M} \left\{ \frac{(P_i^c - P_i^e)^2}{\sigma_P^2} + \frac{(T_i^c - T_i^e)^2}{\sigma_T^2} + \sum_{j=1}^{N} \frac{(x_{j_i}^c - x_{j_i}^e)^2}{\sigma_x^2} + \frac{(y_{j_i}^c - y_{j_i}^e)^2}{\sigma_y^2} \right\} \quad (13)$$

Where the superscripts c and e indicate calculated and experimental values, respectively, the σ^2 are the estimated variances of the corresponding variables, and the sum is taken over all *M* experimental data, and *N* is the number of compounds in the mixtures. The standard deviation assumed were (Marc et.al., 1998; Lu et.al., 1989):

σ_P = 0.5 mmHg
σ_T = 0.1 $^{\circ}$C
σ_x = 0.001 mole fraction
σ_y = 0.005 mole fraction

A listing of optimized interaction parameters for NRTL activity coefficient model studied is shown in Table 6 for all binary systems.

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Table 6 Optimized interaction parameters for binary systems.

System	τ_{12}	τ_{21}	α
Hexane – Benzene	466.2	269.3	0.292
Hexane – 1-Propanol	822.7	174.8	0.412
Benzene- 1-Propanol	296.9	212.6	0.405

The values of activity coefficients calculated by NRTL equations are also given in Table 3 to 5. A defined deviation between the calculated and experimented activity coefficients was evaluated by the formula (Anderson et.al., 1978):

$$\omega = \frac{\sum k^2 - (\sum k)^2\, n^{1/2}}{(n-1)} \qquad (14)$$

k = deviation of experimental activity
    coefficients values from calculated values.
n = number of experimental points
ω = defined deviation of k from a mean value
    of k

The deviations are as follows:

H – B		H – 1-P		B – 1-P	
H	B	H	1-P	B	1-P
0.029	0.023	0.101	0.137	0.112	0.188

Maximum and minimum values of the activity coefficients were calculated to show the limits of the experimental deviations. The refractometer error for the *n*-hexane – benzene and benzene - 1-propanol system was within 0.001 mole fraction and for the *n*-hexane - 1-propanol system, 0.005 mole fraction. Pressure errors were within ± 0.5 mm of Hg and temperature errors were within ± 0.1 $^{\circ}$C.

$$\gamma_{max} = \frac{(y_1 + 0.001)(P_T + 0.5)}{(x_1 - 0.001)(P_1\, at\ t\ - 0.1\,^{o}C)} \qquad (15)$$

$$\gamma_{min} = \frac{(y_1 - 0.001)(P_T - 0.5)}{(x_1 + 0.001)(P_1\, at\ t\ + 0.1\,^{o}C)} \qquad (16)$$

The defined deviation between the experimental activity coefficients and the γ_{max} and γ_{min} values calculated by equation 14, in the range of $x = 0.15$ to $x = 0.85$, are as follows:

H – B		H – 1-P		B – 1-P	
H	B	H	1-P	B	1-P
$\omega_{max\ \gamma}$ 0.022	0.006	0.034	0.048	0.009	0.013
$\omega_{min\ \gamma}$ 0.006	0.016	0.026	0.053	0.005	0.012

The defined deviation over the full range will be larger because of the influence of the high error in the end values of γ's. It may be seen from Table 3 to 5 that the deviation between the calculated (Equation 9 to 12) and experimental values of γ's is maximum in the middle range and this deviation depends on the type of equation applied for calculation the γ's.

It is not necessary that the defined deviation between calculated and experimental γ's be between $\omega_{max\ \gamma}$ and $\omega_{min\ \gamma}$ values as they are only deviations due to experimental errors.

McDermott-Ellis test method (McDermott et.al., 1965) was applied to the activity coefficient – composition data of the binaries.

According to McDermott-Ellis test method, two experimental points *a* and *b* are thermodynamically consistent if the following condition is fulfilled:

$$D < D_{max} \qquad (17)$$

The local deviation D is given by

$$D = \sum_{i=1}^{N}(x_{ia} + x_{ib})(\ln\gamma_{ib} - \ln\gamma_{ia}) \qquad (18)$$

In this method, it is recommended the use of a fixed value of 0.01 for D_{max} if the accuracy in the measurement of the vapor and the liquid mole fraction is within 0.001. The local maximum deviation, D_{max}, due to experimental errors, is not constant, and is given by

$$D_{max} = \sum_{i=1}^{N}(x_{ia} + x_{ib})\left(\frac{1}{x_{ia}} + \frac{1}{y_{ia}} + \frac{1}{x_{ib}} + \frac{1}{y_{ib}}\right)\Delta y$$

$$+ 2\sum_{i=1}^{N}|\ln\gamma_{ib} - \ln\gamma_{ia}|\Delta x + \sum_{i=1}^{N}(x_{ia} + x_{ib})\frac{\Delta P}{P}$$

$$+ \sum_{i=1}^{N}(x_{ia} + x_{ib})\left(\frac{1}{t_a} + \frac{1}{t_b}\right)\Delta t$$

$$(19)$$

Table 7 Results of Thermodynamic
Consistency test.

System	D	D_{max}
1-Propanol – Hexane	0.0243	0.026
Hexane – Benzene	0.0161	0.021
1-Propanol – Benzene	0.0274	0.029
1-Propanol – Hexane – Benzene	0.0321	0.035

In accordance with the criterion of the test the data were found to be consistent.

The experimental data for all the three binaries show that they are non–ideal in nature. (Tongberg et.al., 1992), studying the equilibrium of *n*-hexane – benzene, reported no separation obtainable at concentration above 97 mole % hexane. This is consistent with the observation made in this investigation.

The *n*-hexane – 1-propanol and benzene – 1-propanol systems evidence minimum boiling azeotropes. It is indicated by the interpolation of the data that *n*-hexane – 1-propanol form an azeotrope at 95 mole % hexane at 65.8 ^{o}C, and the benzene – 1-propanol form an azeotrope at 77.5 mole % benzene at 76 ^{o}C

The variation of the relative volatility with the concentration of the solvent in the ternary system is reported in Table 8 and shown in Figure 8.

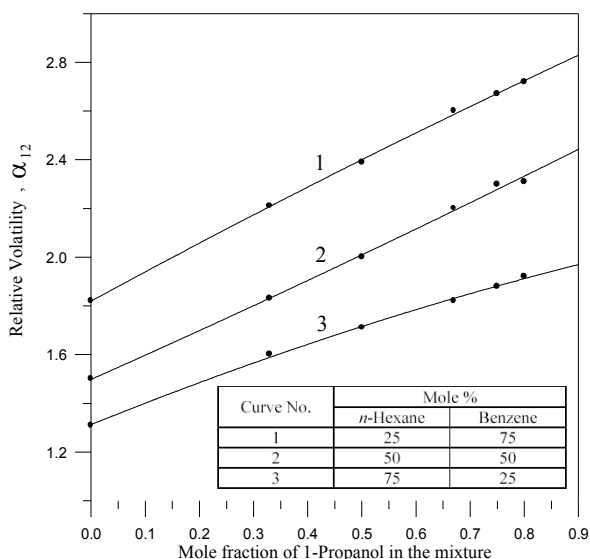| Curve No.	Mole %	
	n-Hexane	Benzene
1	25	75
2	50	50
3	75	25

Fig 8 Effect of 1-propanol on relative volatility of binary system *n*-hexane – benzene at 760 mmHg

Table 8 Variation of relative volatility with solvent concentration for ternary system
n-hexane – benzene- 1-propanol at 760 mmHg Pressure

Binary mixture		Mole fraction 1-propanol in mixture	Temperature ^{o}C	Mole fraction 1-propanol free basis				$\alpha(H/B)$
x'_H	x'_B	x_{1-P}		x_H	y_H	x_B	y_B	
0.25	0.75	0.00	74.1	0.21	0.326	0.79	0.674	1.82
		0.33	27.0	0.193	0.346	0.807	0.654	2.21
		0.50	74.3	0.172	0.332	0.828	0.668	2.39
		0.67	78.7	0.156	0.325	0.884	0.675	2.60
		0.75	82.3	0.154	0.327	0.846	0.673	2.67
		0.80	84.8	1.36	0.300	0.864	0.709	2.72
0.50	0.50	0.00	71.2	0.428	0.528	0.572	0.472	1.50
		0.33	67.5	0.424	0.574	0.576	0.426	1.83
		0.50	70.2	0.400	0.572	0.600	0.423	2.00
		0.67	75.2	0.396	0.593	0.606	0.407	2.23
		0.75	80.3	0.375	0.580	0.625	0.429	2.30
		0.80	83.0	0.368	0.574	0.632	0.426	2.31
0.75	0.25	0.00	69.4	0.684	0.724	0.316	0.276	1.31
		0.33	66.3	0.681	0.776	0.324	0.224	1.63
		0.50	68.2	0.632	0.744	0.378	0.256	1.71
		0.67	72.8	0.658	0.776	0.342	0.224	1.80
		0.75	78.2	0.635	0.766	0.365	0.234	1.88
		0.80	80.7	0.605	0.746	0.395	0.251	1.92

x'= Solvent free basis

Conclusion

The data show that the greatest change of relative volatility is obtained at higher concentration of the solvent. As the vapor and liquid samples were extracted with water, it was ensured by laboratory tests that the solvent–free hydrocarbon concentration did not change because of the different solubilities of the hydrocarbons in water. Also, it was found that the drying agent, Drierite, had no selective absorption capacity for the hydrocarbon mixture involved.

Nomenclature

a_i　　Combining rule coefficient
a_{ij}　　Mixing rule coefficient
a_{ij}^O　　Mixing rule coefficient
A　　Cohesion parameter of cubic equation of state
b_i　　Combining rule coefficient
B　　Covolume term (parameter) of the cubic equation of state
B_i　　Combining rule coefficient
$k_{a_{ij}}$　　Interaction coefficient of equation of state
$k_{b_{ij}}$　　Interaction coefficient of equation of state
m　　Temperature dependent correlation parameter
n　　Temperature dependent correlation parameter
n_D　　Refractive index
P　　Equilibrium pressure of the system, MPa
P_i　　Vapor pressure of pure component i MPa
T　　Temperature, K
T_C　　Critical temperature, K
x_i　　Mole fraction of component i in liquid phase
y_i　　Mole fraction of component i in vapor phase
z　　Compressibility factor

Greek Litters

α　　Temperature dependency of the attractive term of equation of state
α　　Relative volatility
γ　　Activity coefficient
σ　　Standard deviation
ρ　　Density
σ^2　　Estimated variance
Φ　　Fugacity coefficient

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تأثير المركب القطبي (١-بروبانول) على التطايرية النسبية للنظام الثنائي هكسان – بنزين

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الخلاصة :

تم عرض بيانات إتزان بخار سائل للأنظمة الثنائية هكسان – ١بروبانول، بنزين – ١بروبانول و هكسان –بنزين في ضغط بالاطمادابالقى ذلك تم عرض بيانات النظام الثلاثي لتراكيز مختارة بالنسبة إلى١بروبانول في نظام ١-بروبانول، بنزين، هكسان في ضغط ٧٦٠ ملم زئبقالنتائج أشارت الى إنه التطايرية النسبية للهكسان بالنسبة الى البنزين قد إزدادت بصورة ملموسة عند إضافه ١-بروبانول.

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