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Pascal Théveneau, Alain Valtz, Christophe Coquelet. Vapor Liquid Equilibrium Data for the Furan-Toluene Binary System between 313.02 and 352.99 K. *Journal of Chemical and Engineering Data*, 2017, 62, pp.1168-1172. 10.1021/acs.jced.6b00424 . hal-01531268

HAL Id: hal-01531268

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Submitted on 1 Jun 2017

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Vapor liquid equilibrium data for the Furan - Toluene binary system between 313.02 and 352.99 K

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Short communication

Abstract

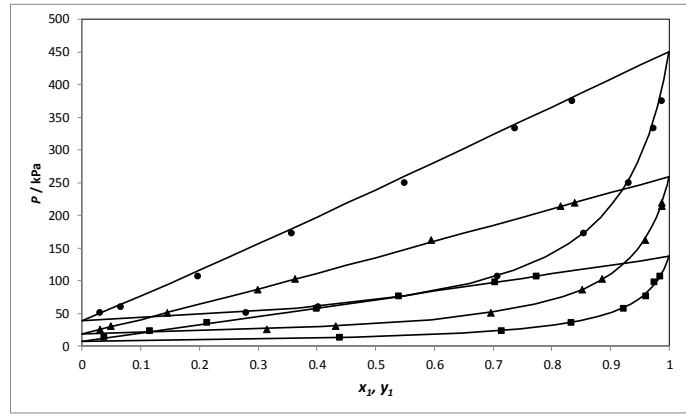
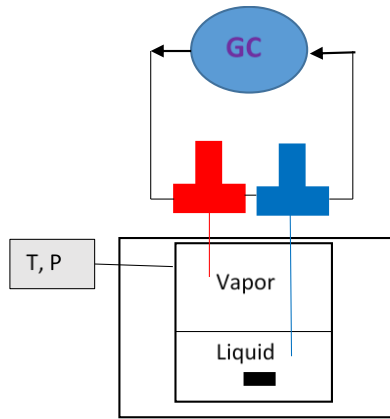
The study presents p - T - x - y phase equilibria measurements of an oxygenated compound (furan) and one of its potential solvent of extraction (toluene). The experimental technique of the equipment recently developed is based on a “static analytic” method. Measurements were done at three temperatures (313.02, 333.02 and 352.99 K) and pressures from 13 up to 375 kPa. The experimental data measured were successfully correlated, using a “gamma phi” approach, with the NRTL activity coefficient model.

Keywords: furan, toluene, static analytic method, low pressure, NRTL

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Graphical TOC JCED January 2017



1. Introduction

Chemical and processes engineering require fluid thermodynamic models and accurate knowledge of phase diagrams. In effect, their knowledge is important for the design of efficient separation units like distillation and extraction¹. A thermodynamic model allows prediction of thermophysical properties like densities, speed of sound and evaluation of phase diagrams. For example, phase diagrams can be used to predict distillation lines or residue curves for process separation. This step is very important for the design of separation units and to evaluate the difficulty of the separation. Successful design and simulation of separation units by process simulation software rely on accurate phase equilibrium computations using various thermodynamic models (equations of state and/or model of solution) that generally contain several empirical parameters. In the common engineering practice, the values of these parameters are adjusted, if possible, on different sets of experimental data such as limiting activity coefficient, heat of mixing, vapor–liquid or liquid–liquid equilibria (VLE, LLE, etc.), of the systems of interest. Measurements of accurate experimental data require efficient equipment. Recently, Zhang et al.² have developed a new equipment for data acquisition of equilibrium properties at low pressure from 50 mbar to 10 bar using a “static-analytic method”. In their paper, it has been applied to investigate vapour liquid equilibrium properties of systems containing organic sulfur compounds. New sampling mechanisms were combined with ROLSI® capillary samplers³ to achieve on-line sampling for both vapor and liquid phases. Phase samples were directly sent to a gas chromatograph (GC) for composition analysis.

In this paper, new experimental data of the Furan + Toluene binary system are presented. Furan is an interesting molecule. It can be used as an intermediate in order to produce new molecules for pharmaceutical applications, for example. Also, it is possible to extract furan from bio-oil, produced from wood pyrolysis. Toluene can be a suitable extraction solvent. NRTL activity coefficient model⁴, based on a “gamma-phi” approach, is used to correlate experimental data. This system was previously studied by Nala et al.⁵ at atmospheric pressure using an ebulliometer. In this paper, we also propose to compare the results obtained using both techniques.

2. Experimental

2.1. Materials

The sources and the qualities of the used chemicals, as certified by the manufacturers, are presented in Table 1. Indices of refraction at the sodium D-line (n_D) are also measured and compared with supplier values. Considering the high level of purity of these chemicals, no further purification or pretreatment were performed.

[Table 1]

2.2. Apparatus and Experimental Procedure

The equipment was previously presented and described by Zhang et al.². The equilibrium cell is immersed in a thermo-regulated liquid bath (LAUDA Proline RP 3530 C). The temperature of the equilibrium cell was measured using two (Pt-100) platinum probes. The two probes were calibrated by comparison with a 25 Ω reference platinum probe (Tinsley, France). The accuracy of two probes was estimated as ± 0.02 K. Also, pressures were measured by three pressure transducers (General Electric, model UNIK 5000) with maximum absolute pressures of 0.35 bar, 1 bar and 10 bar, respectively. These pressure sensors were maintained at a constant temperature (353 K throughout this work) by means of PID regulator (FUJI, model PXE-4). The accuracy of the pressure transducers was estimated as ± 0.2 mbar after calibration. Both temperature and pressure signals were transmitted to a data acquisition unit (Agilent 34972A). Liquid and vapor samples were analyzed by means of a gas chromatograph (Perichrom, model PR-2100). A thermal conductivity detector (TCD) was calibrated and used to determine the molar composition of the two phases. The WINILAB III (Perichrom, France) data acquisition software was used for peak integration.

The calibration of the TCD was made by introducing known pure component volumes with appropriate GC syringes. The accuracies concerning the mole numbers are $\pm 5\%$ for furan and $\pm 2\%$ for toluene. The maximum standard uncertainty on liquid mole fractions was estimated about $u(x,y)=0.013$. The ZEBRON ZB-5 (5 μm , 30 m \times 0.53 mm ID) GC wide bore column is used.

The uncertainty of the mole fraction was determined after calibration of the TCD.

Equation 1 reminds how is calculated the composition of component i (ncomp is the number of component in the mixture).

$$x_i = \frac{n_i}{\sum_i^{ncomp} n_i} \quad (1)$$

Equation 2 is used for the calculation of the uncertainty of mixture composition of component i .

$$u_{cal}(x_i) = \sqrt{\sum_i^{ncomp} \left(\frac{\partial x_i}{\partial n_i} \right)^2 u^2(n_i)} \quad (2)$$

Consequently, for a binary system, one can calculate uncertainty on mole fraction x_1 by using equation 3.

$$u_{cal}(x_1) = x_1(1-x_1) \sqrt{\left(\frac{u(n_1)}{n_1} \right)^2 + \left(\frac{u(n_2)}{n_2} \right)^2} \quad (3)$$

Equation 4 reminds the combined standard temperature uncertainty on molar composition.

$$u(x_i) = \pm \sqrt{u_{calib}(x_i)^2 + u_{rep}(x_i)^2} \quad (4)$$

3. Correlation

The correlation of the experimental measurements was obtained using a “gamma phi” approach. In this work, the vapor phase is considered as an ideal phase. Eq. 5 is the DIPPR type equation used to calculate the pure component vapour pressure.

$$\ln(P_i^{sat}) = A + \frac{B}{T} + C \ln(T) + DT^E \quad (5)$$

The corresponding parameters are presented in the Table 2.

[Table 2]

The NRTL activity coefficient model is considered for the liquid phase. Eq. 6 is the expression of the excess Gibbs energy model based on NRTL model (Eqs. 7 and 8 details the calculation of the different parameters)..

$$\frac{g^E(T, p, x_i)}{RT} = \sum_{i=1}^n x_i \frac{\sum_{j=1}^n \tau_{ji} G_{ji} x_j}{\sum_{k=1}^n G_{ki} x_k} \quad (6)$$

n is the number of components in the system.

$$G_{ij} = \exp(-\alpha_{ij} \tau_{ij}) \quad (7)$$

$$\tau_{ij} = \frac{(g_{ij} - g_{ii})}{RT} \quad (8)$$

g_{ij} , g_{ji} , g_{ii} and g_{jj} are interaction energies between two molecules. It is important to note that $G_{ii} = G_{jj}$ and $\tau_{ii} = \tau_{jj} = 0$. In our case, we have considered $g_{ij} - g_{ii} = C_{ij} + C_{ij}^T (T - 273.15)$, C_{ij} and C_{ij}^T are the adjustable binary parameters. Binary interaction parameters are fitted using the Simulis ThermodynamicsTM software package (from Prosim, Toulouse, France). Eq. 9 is considered as objective function (a Flash type algorithm is also considered for the calculation of equilibrium properties).

$$F = \frac{100}{N} \left[\sum_1^N (x_{i,\text{exp}} - x_{i,\text{cal}})^2 + (y_{i,\text{exp}} - y_{i,\text{cal}})^2 \right] \quad (9)$$

N is the number of experimental data points and $z_{i,\text{exp}}$ and $z_{i,\text{cal}}$ are the experimental and calculated compositions for each phase, respectively.

4. Results and discussion

The results are presented in Table 3.

[Table 3]

We have checked the consistency of the data. For each isotherm we have determined the activity coefficient of both species using experimental data and used the classical area test (Eq. 10).

$$\sum_i x_i d \ln \gamma_i = \frac{-h^E}{RT^2} dT + \frac{v^E}{RT} dP \quad (10)$$

Densities of several mixtures of furan and toluene (Table 4) at 303.15 K were measured by Nala et al.⁵ using an Anton Paar DM5000 density meter with reported accuracy of ± 0.00001 g/cm³.

[Table 4]

We can notice that excess molar volumes are very low. Consequently, excess molar volume between these two species can be neglected.

As measurements have been done at constant temperature, Eq. 11 can be used for the classical area test.

$$\sum_i x_i d \ln \gamma_i = 0 \quad (11)$$

Finally, after integration of Eq. 10, we obtained the final criteria (Eq. 12) for the classical area test.

$$\int_{x_1=0}^1 \ln \frac{\gamma_1}{\gamma_2} = 0 \quad (12)$$

We have determined experimentally the activity coefficient with Eq. 13.

$$\gamma_i = \frac{Py_i}{x_i P_i^{sat}} \quad (13)$$

The uncertainty of the activity coefficients is determined considering the uncertainties of vapor and liquid compositions and uncertainty on pressure. The results are presented on Figures 1 to 3 for the different temperatures studied. We can conclude that the test is a success and so consider that the new data are consistent.

Table 5 presents adjusted NRTL activity coefficient binary interaction parameters. Figure 4 presents the three isotherms and their modelling.

[Table 5]

[Figure 1]

[Figure 2]

[Figure 3]

The performance of the model used to correlate the experimental data was evaluated by means of the following relative deviations, $BIAS_z$ and AAD_z which are expressed by Eqs. 14 and 15.

$$AAD_z = (1/N) \sum_{i=1}^N |z_{i,exp} - z_{i,cal}| \quad (14)$$

$$BIAS_z = (1/N) \sum_{i=1}^N (z_{i,exp} - z_{i,cal}) \quad (15)$$

where N is the number of experimental measurements. Results are presented in Table 6. As we can see, the data are well correlated by the model.

[Table 6]

[Figure 4]

Figure 4 reveals that an ideal Raoult's law behaviour is observed. In order to confirm this observation, limiting activity coefficient of furan in toluene were determined using a gas stripping method. The apparatus is fully described in the recent publication by Zin et al.⁶. This technique is based on the evolution of vapor phase composition when the highly diluted solute (furan) of the liquid mixture in a dilutor cell is stripped by a constant flow of inert gas (helium). Eq. 16 reminds the expression used to calculate the apparent Henry's law constant.

$$H_i = -\frac{1}{t} \ln \left(\frac{S_i}{(S_i)_{t=0}} \right) \frac{RTN}{1 - \frac{P_{solv}^{sat}}{P} + \frac{V_G}{t} \ln \left(\frac{S_i}{(S_i)_{t=0}} \right)} \quad (16)$$

where D is the carrier gas flow rate ($\text{m}^3 \cdot \text{s}^{-1}$); N is the total number of moles of solvent inside the dilutor cell; V_G (m^3) is the volume of the vapor phase inside the dilutor cell; S_i is the chromatograph solute i peak area; t (s) is the time; T (K) is the temperature inside the cell; P (101 300 Pa) is the pressure inside the cell (around 1 atm); P_{solv}^{sat} (Pa) is the saturation pressure of the solvent (i.e. toluene, see Eq. 5 and Table 2); and R ($\text{J} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$) is the ideal gas constant. Relative uncertainty concerning the apparent Henry's law constant is estimated to be within ± 5 %. The limiting activity coefficient is $\gamma_i^\infty = \frac{H_i}{P_i^{sat}}$ for furan (P_i^{sat} is the saturation pressure of the solute, see Eq. 5 and Table 2). Ideal gas law is considered for the gas phase. Results are presented in Table 7 and Figure 5. As we can see the limiting activity coefficient of furan in toluene is close to 1. It confirms the ideal solution behaviour observed.

[Table 7]

[Figure 5]

In the paper published by Nala et al.⁵ VLE data of the furan + toluene binary system were measured at atmospheric pressure. We have predicted their experimental data using our developed model. The results are presented in Table 8 and plotted on Figure 6. As we can see, data from Nala et al. are well predicted (AAD $x=0.008$, BIAS $x=0.007$, AAD $y=0.030$ and BIAS $y=0.031$). Both sets of data (from Nala et al. and the new ones presented here) are in good agreement.

[Table 8]

[Figure 6]

5. Conclusions

New vapor liquid equilibrium data of the furan + toluene binary system were measured using the new equipment developed in the CTP research group². This new equipment allows the measurement of vapor liquid equilibrium properties at low pressure using the “static analytic method”. New data measured were correlated using the NRTL activity coefficient model in a gamma phi approach. Data measured by Nala et al.⁵ are in good agreement with the ones presented here.

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Table 1: Chemical sample

Chemical name	CAS number	Source	Initial purity (weight %)	Purification method	Final purity	Analysis method	Refractive index (n_D)	
							Measured* at 20°C and 101.232 kPa	Supplier
furan	110-00-09	Acros	≥ 99	None	--	GC	1.4218	1.4197 1.4217
toluene	108-88-3	Acros	99.85	None	--	GC	1.4966	1.4968

GC: Gas Chromatograph, * with Anton Paar Abbemat 300 Refractometer (standard uncertainty: refractive index $u(n_D)=10^{-4}$ pressure (GE Druck DPI 142 Barometric Indicator) $u(p)=0.029$ kPa and temperature $u(T)=0.03^\circ\text{C}$)

Table 2: Vapor pressure correlation parameters from Simulis Thermodynamics™, Prosim, France. Range of validity: from triple point to critical point.

Parameter	Furan	Toluene
A	74.738	76.945
B	-5417	-6729.8
C	-8.0636	-8.179
D	$7.47 \cdot 10^{-6}$	$5.3017 \cdot 10^{-6}$
E	2	2

Table 3: Vapor liquid equilibrium of furan (1) + toluene (2) binary system at 313.02, 333.02 and 352.99 K.

Experimental data					Calculated data using NRTL activity coefficient model	
<i>T</i> /K= 313.02						
<i>P</i> / kPa	<i>x</i> ₁	u(<i>x</i> ₁)	<i>y</i> ₁	u(<i>y</i> ₁)	<i>x</i> ₁	<i>y</i> ₁
13.04	0.0381	0.0023	0.4389	0.0153	0.0435	0.4251
22.88	0.1147	0.0063	0.7147	0.0127	0.1245	0.7004
35.31	0.2131	0.0104	0.8329	0.0087	0.2244	0.8284
57.18	0.3990	0.0149	0.9221	0.0045	0.3954	0.9179
75.69	0.5399	0.0154	0.9599	0.0024	0.5367	0.9530
97.56	0.7037	0.0130	0.9743	0.0016	0.7013	0.9768
106.79	0.7744	0.0109	0.9844	0.0010	0.7705	0.9839
<i>T</i> /K= 333.02						
<i>P</i> / kPa	<i>x</i> ₁	u(<i>x</i> ₁)	<i>y</i> ₁	u(<i>y</i> ₁)	<i>x</i> ₁	<i>y</i> ₁
25.81	0.0309	0.0019	0.3143	0.0134	0.0333	0.3112
30.30	0.0491	0.0029	0.4316	0.0153	0.0533	0.4254
50.81	0.1455	0.0077	0.6956	0.0132	0.1435	0.6901
86.37	0.2985	0.0130	0.8520	0.0078	0.2956	0.8505
102.86	0.3632	0.0144	0.8851	0.0063	0.3646	0.8871
162.24	0.5942	0.0150	0.9595	0.0024	0.6078	0.9565
214.10	0.8156	0.0094	0.9873	0.0008	0.8170	0.9849
219.48	0.8383	0.0084	0.9872	0.0008	0.8387	0.9871
<i>T</i> /K= 352.99						
<i>P</i> / kPa	<i>x</i> ₁	u(<i>x</i> ₁)	<i>y</i> ₁	u(<i>y</i> ₁)	<i>x</i> ₁	<i>y</i> ₁
51.15	0.0303	0.0018	0.2785	0.0125	0.0332	0.2717
60.07	0.0651	0.0038	0.4025	0.0150	0.0564	0.3946
106.52	0.1966	0.0098	0.7069	0.0129	0.1753	0.7016
172.99	0.3568	0.0143	0.8539	0.0078	0.3404	0.8534
249.73	0.5489	0.0154	0.9303	0.0040	0.5256	0.9275
333.19	0.7367	0.0121	0.9729	0.0016	0.7230	0.9687
375.06	0.8342	0.0086	0.9867	0.0008	0.8215	0.9823

U(*T*, k=2)= 0.02 K, U(*P*, k=2)= 0.02 kPa.

Table 4: Experimental and calculate excess volume ^a at 303.15 K ^b for the system furan (1) + toluene (2) from Nala et al.⁵.

x_1^c	$\rho/\text{g.cm}^{-3}$	$\rho/\text{mol.cm}^{-3}$	$v/\text{cm}^3.\text{mol}^{-1}$	$v^E/\text{cm}^3.\text{mol}^{-1}$
0	0.8610	0.00934	107.016	0.000
0.100	0.8659	0.00965	103.631	-0.017
0.199	0.8712	0.00997	100.266	-0.047
0.387	0.8815	0.01064	93.962	-0.019
0.507	0.8889	0.01112	89.931	-0.008
0.591	0.8946	0.01148	87.098	-0.011
0.761	0.9075	0.01229	81.352	-0.031
0.89	0.9184	0.01299	77.006	-0.032
1	0.9283	0.01364	73.333	0.000

^a $u(v) = 0.007 \text{ cm}^3$, ^b $u(T) = 0.01 \text{ K}$, ^c $u(x_1) = 0.001$.

Table 5: NRTL binary interaction parameters for furan (1) + toluene (2) binary system

C_{12}	C_{21}	α_{12}
/ cal.mol ⁻¹		
206.90	-240.71	0.2

Table 6: BIAS and AAD of liquid and vapor compositions for the three isotherms.

Temperature /K	BIAS _x	AAD _x	BIAS _y	AAD _y
313.02	-0.002	0.006	0.006	0.007
333.02	-0.002	0.004	0.002	0.003
352.99	0.013	0.014	0.005	0.005

Table 7: Temperature dependence of Henry's Law Constant for furan in toluene at atmospheric pressure.

T/ K	H/ MPa	γ_{∞}
293.15	0.068	1.037
298.15	0.083	1.033
303.16	0.100	1.034
308.19	0.119	1.025
313.17	0.141	1.022
318.12	0.167	1.028
323.09	0.196	1.026

$U(p, k=2)=0.06\text{kPa}$, $U(T, k=2) = 0.2\text{K}$, $u_r(H) = 0.05$

Table 8: Vapor liquid equilibrium of furan (1) + toluene (2) binary system at atmospheric pressure ($p=101.325$ kPa) from Nala et al.⁵.

Nala et al. ⁵			Calculated data using NRTL activity coefficient model	
T/K	x_1	y_1	x_1	y_1
383.78	0	0	0	0
381.66	0.007	0.068	0.008	0.068
381.96	0.010	0.076	0.007	0.058
380.20	0.013	0.111	0.014	0.111
379.11	0.017	0.154	0.019	0.143
377.88	0.029	0.214	0.024	0.178
376.20	0.036	0.283	0.031	0.223
375.47	0.037	0.289	0.034	0.242
372.75	0.045	0.361	0.046	0.311
369.97	0.068	0.441	0.060	0.376
364.81	0.098	0.560	0.087	0.485
363.12	0.106	0.577	0.096	0.517
360.26	0.125	0.637	0.113	0.568
357.97	0.136	0.665	0.128	0.607
352.97	0.168	0.733	0.162	0.681
348.26	0.207	0.792	0.199	0.742
340.60	0.289	0.867	0.270	0.823
336.59	0.326	0.888	0.314	0.857
334.07	0.353	0.901	0.345	0.877
330.13	0.414	0.927	0.397	0.903
324.67	0.481	0.945	0.483	0.934
320.78	0.552	0.958	0.554	0.952
318.02	0.610	0.966	0.610	0.963
312.33	0.758	0.981	0.748	0.982
308.67	0.869	0.989	0.854	0.991
305.98	0.958	0.995	0.944	0.997
305.00	0.995	0.996	0.979	0.999
304.55	1	1	0	1

$u(T)=0.01$ K, $u(p)=0.029$ kPa and $u_{\max}(x)=u_{\max}(y)=0.007$

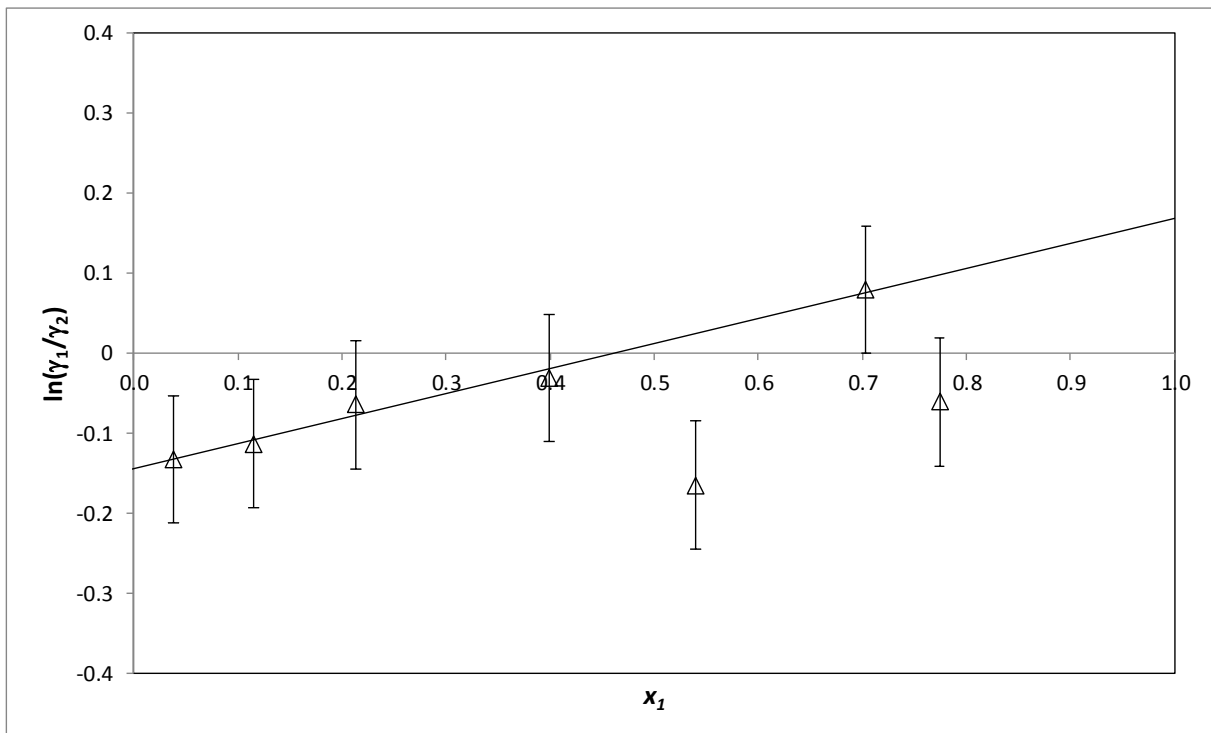


Figure 1 : Classical area test concerning the measurements of VLE of the furan (1) + toluene (2) binary system at 313.02 K. Error bar: ± 0.08

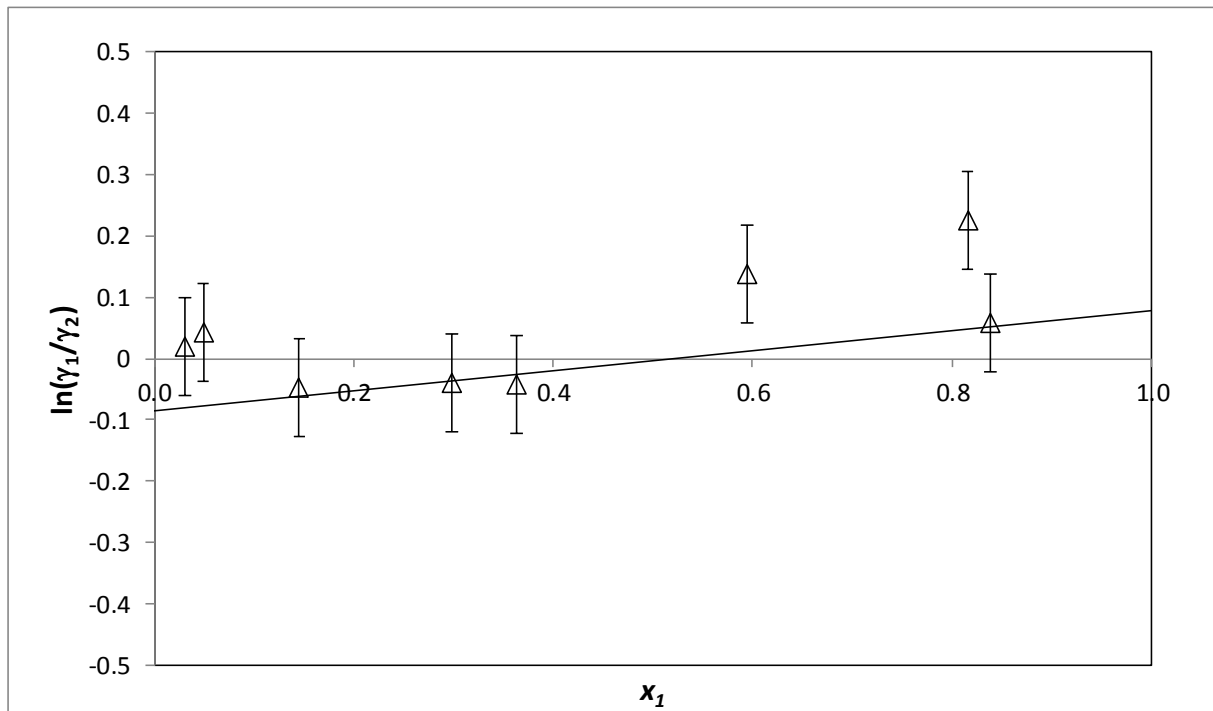


Figure 2 : Classical area test concerning the measurements of VLE of the furan (1) + toluene (2) binary system at 333.02 K. Error bar: ± 0.08

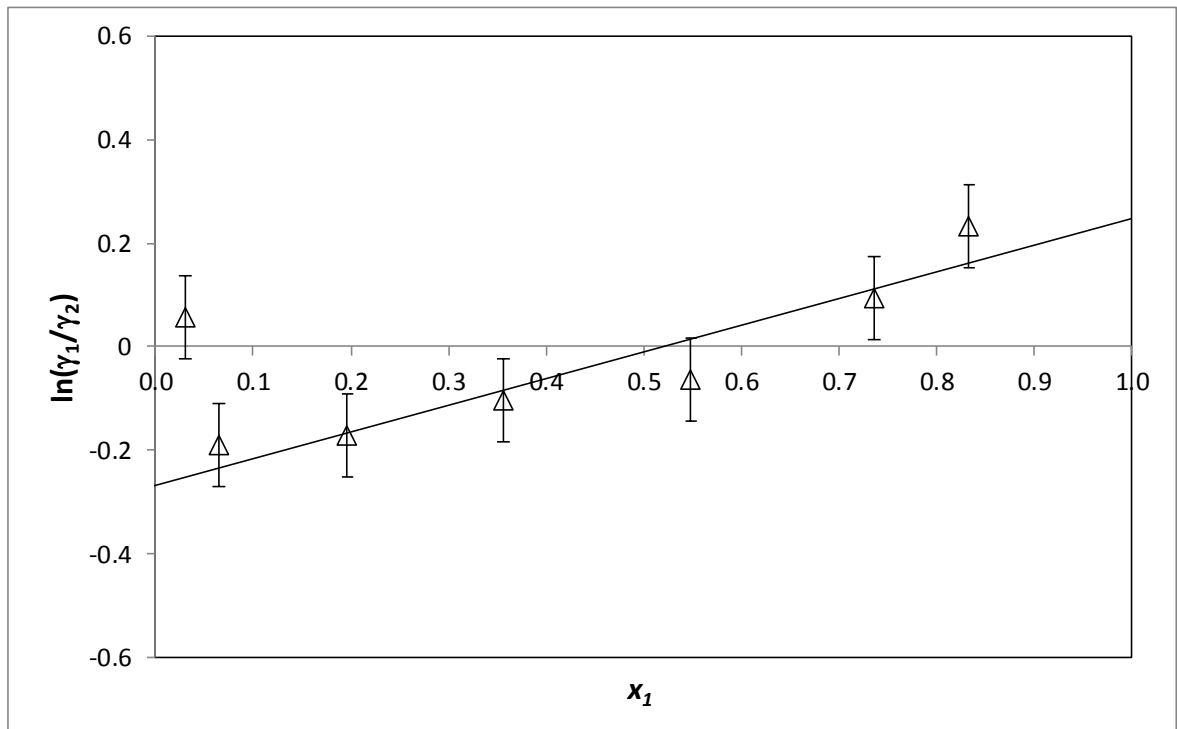


Figure 3 : Classical area test concerning the measurements of VLE of the furan (1) + toluene (2) at 352.99 K. Error bar: ± 0.08

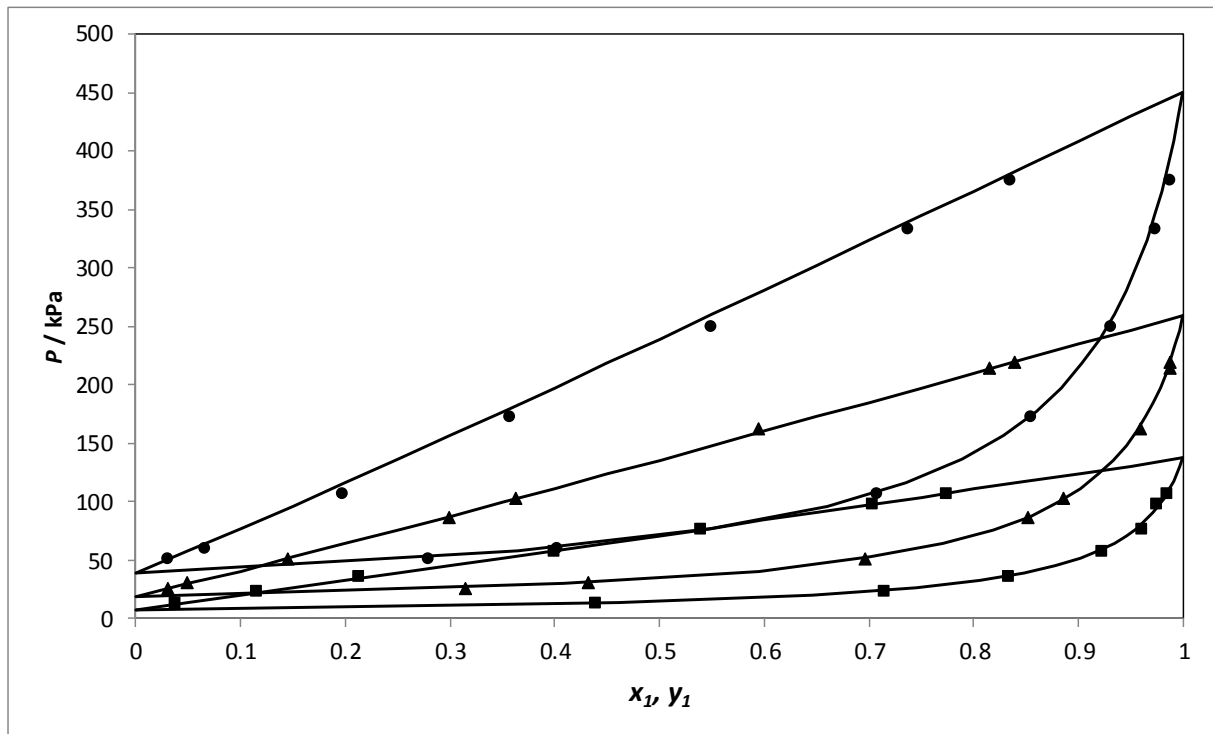


Figure 4: Vapor – Liquid equilibrium isotherms for furan (1) + toluene (2) binary system: Symbols (this work): ■: 313.02 K, ▲, 333.02 K;●: 352.99 K solid lines, calculated results using NRTL activity coefficient model.

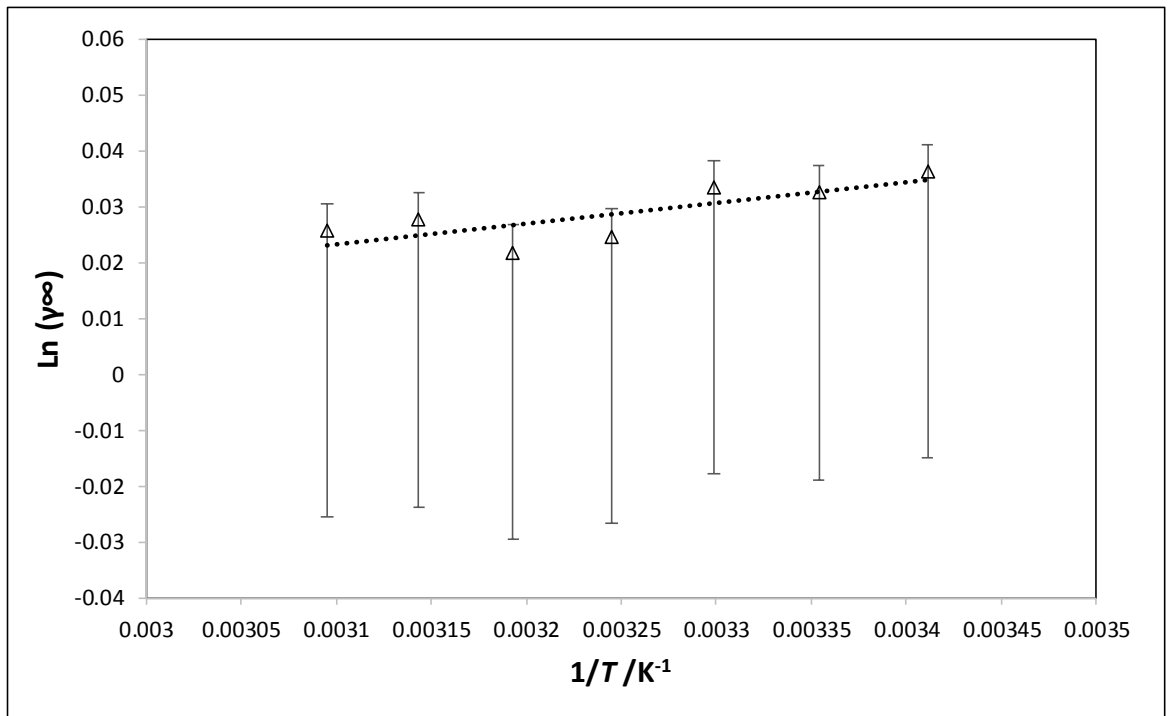


Figure 5: Logarithm of limiting activity coefficient of furan in toluene as a function of inverse temperature. Error bar is calculated by considering $u_r(H)=0.05$.

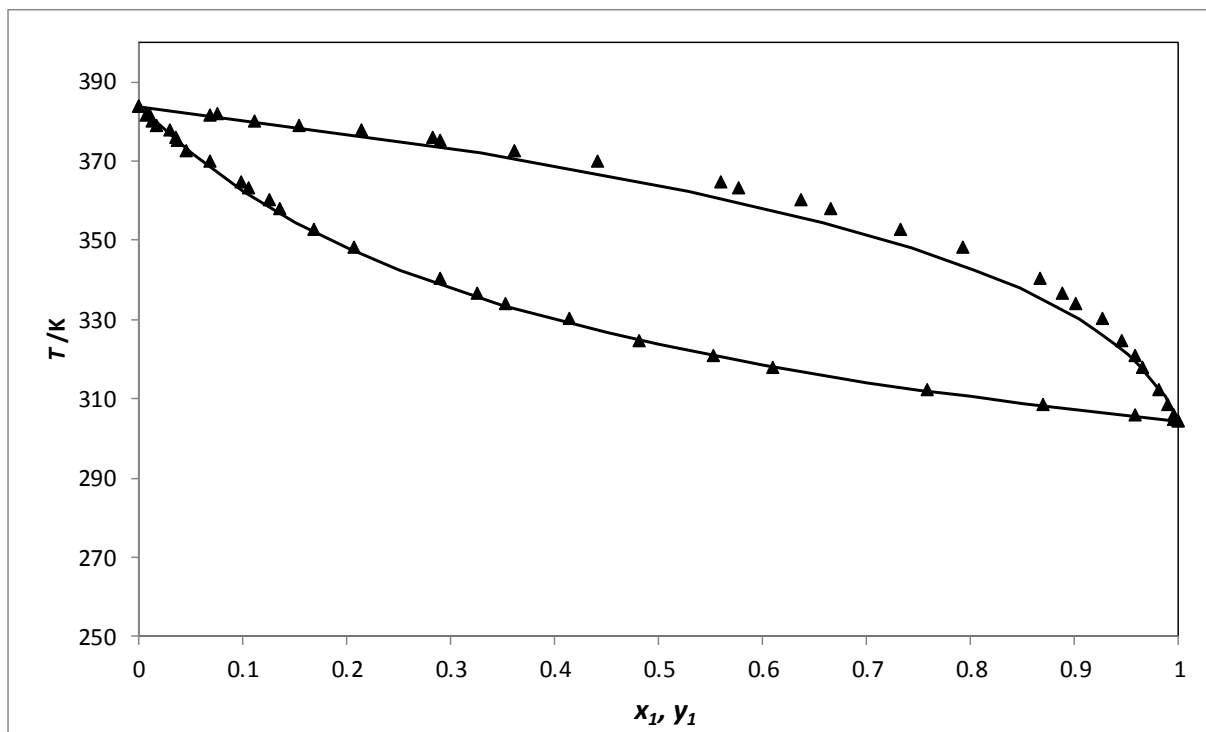


Figure 6: Vapor – Liquid equilibrium isotherm for furan (1) + toluene (2) binary system at atmospheric pressure. Solid line: prediction using our NRTL activity coefficient model. Symbol: experimental data from Nala et al.⁵.