Vapor-liquid equilibrium measurements of the binary mixtures nitrogen + acetone and oxygen + acetone

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Abstract

Vapor-liquid equilibrium data of the binary mixtures nitrogen + acetone and of oxygen + acetone are measured and compared to available experimental data from the literature. The saturated liquid line is determined for both systems at specified temperature and liquid phase composition in a high-pressure view cell with a synthetic method. For nitrogen + acetone, eight isotherms between 223 and 400 K up to a pressure of 12 MPa are measured. For oxygen + acetone, two isotherms at 253 and 283 K up to a pressure of 0.75 MPa are measured. Thereby, the maximum content of the gaseous component in the saturated liquid phase is 0.06 mol/mol (nitrogen) and 0.006 mol/mol (oxygen), respectively. Based on this data, the Henry's law constant is calculated. In addition, the saturated vapor line of nitrogen + acetone is studied at specified temperature and pressure with an analytical method. Three isotherms between 303 and 343 K up to a pressure of 1.8 MPa are measured. All present data are compared to the available experimental data. Finally, the Peng-Robinson equation of state with the quadratic mixing rule and the Huron-Vidal mixing rule is adjusted to the present experimental data for both systems.

Keywords: Henry's law constant; gas solubility; vapor-liquid equilibrium; acetone; nitrogen; oxygen

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1 Introduction

The present work is motivated by a cooperation with the Collaborative Research Center Transregio 75 "Droplet Dynamics Under Extreme Ambient Conditions" (SFB-TRR75),[1] which is funded by the Deutsche Forschungsgemeinschaft (DFG). The aim of the SFB-TRR75 is to investigate droplets that are injected into trans- or supercritical gases, a scenario that is present in many technical processes, but is little understood. Examples are rocket combustion systems and future high pressure fuel combustion systems. In the SFB-TRR75, the evaporation of a single acetone droplet in a defined gaseous environment of nitrogen and oxygen is investigated by experiment and numerical computational fluid dynamics simulation. Knowledge on the phase behavior and other thermodynamic properties for the binary systems nitrogen + acetone and oxygen + acetone is crucial to undertake this task.

For the system nitrogen + acetone, some experimental data on fluid phase coexistence are available in the literature. The vapor-liquid equilibrium (VLE) at finite mole fractions was only measured by Jabloniec et al.[2] along two isotherms (303 and 363 K) up to a pressure of 3 MPa. In addition, Henry's law constant data in the temperature range between 195 and 363 K were published by Just,[3] Horiuti,[4] Kretschmer et al.,[5] Nitta et al.,[6] Vosmansky and Dohnal,[7] Tsuji et al.[8] and Jabloniec et al.[2] for nitrogen in acetone and by Levi,[9] Fischer and Pfleiderer,[10] Finlayson,[11] Horiuti,[4] Kretschmer et al.,[5] Schlaepfer et al.,[12] Sinn et al.,[13] Naumenko,[14] Bub and Hillebrand,[15] Tsuji et al.[8] and Luehring and Schumpe[16] for oxygen in acetone.

A new apparatus was constructed to measure the solubility of both gases in liquid acetone, i.e. the course of the saturated liquid line was determined. For nitrogen + acetone, eight isotherms between 223 and 400 K up to a pressure of 12 MPa were studied. These data were compared to experimental literature data. For oxygen + acetone, two isotherms at 253 and 283 K up to a pressure of 0.75 MPa were measured. For both systems, the Henry's law constant was calculated on basis of the present experimental data. Furthermore, the saturated vapor line for nitrogen + acetone was measured at three isotherms (303, 323 and 343 K) with an apparatus which was set up in prior work.[17] In order to aggregate the present experimental results for further applications, the Peng-Robinson equation of state (EOS)[18] was parameterized accordingly, considering two mixing rules that are based on one-fluid-theory, i.e. the quadratic mixing rule[19] and the Huron-Vidal mixing rule.[21]

2 Materials

Nitrogen 5.0 (volume fraction 0.99999) and oxygen 4.8 (volume fraction 0.99998) were obtained from Air Liquide. Acetone with a purity > 99.9 % was purchased from Merck and degassed under vacuum.

3 Apparatus and measurement procedure

Figure 1 shows the newly constructed apparatus for the measurement of the gas solubility, which is based on the equipment by Rumpf and Maurer.[22,23] A thermostated high-pressure view cell with a volume of 28.8 cm^3 was filled in the first step with the gaseous component (nitrogen or oxygen) only. The mass of the gaseous component was obtained volumetrically. Therefore, the temperature and pressure were measured and the density was determined via the EOS by Span et al.[24] for nitrogen or the EOS by Schmidt and Wagner[25] for oxygen. In the second step, the solvent (acetone) was added into the cell with a high pressure screw compressor until the gas was completely dissolved. The associated equilibration process was supported by a magnetic stirrer. Subsequently, small amounts of the liquid mixture were withdrawn from the view cell to reduce the pressure until the formation of small gas bubbles in the mixture indicated the saturated liquid state point. By comparing the positions of the screw compressor before and after the experiment, the volume of the injected solvent was determined and its mass in the view cell was calculated via the EOS by Lemmon and Span[26] for acetone. Note that these three EOS[24–26] are highly accurate multiparameter models that can be regarded as best practice for these pure fluids.

To achieve a constant temperature in all functional units, the apparatus was surrounded by an insulated chamber with an additional thermostat cycle. The temperature in the cell was measured by two calibrated platinum resistance thermometers with a basic resistance of 100 Ω (Pt100). Furthermore, one additional Pt100 thermometer was attached to the screw compressor. To calibrate these thermometers, a more precise platinum resistance thermometer with a basic resistance of 25 Ω was used. The temperature measuring error was about ± 0.04 K.

Four calibrated pressure transducers (model Super TJE, Honeywell test & measurement, measuring ranges: 6.8 and 20 MPa for measurements with nitrogen and 0.2 and 1.2 MPa for oxygen) were used to determine the pressure of the pure gaseous solute and the pressure of the liquid mixture. A deadweight tester (series 5201, Desgranges & Huot) was employed to calibrate the transducers. The accuracy of these transducers is specified to be 0.05 % of their respective full measuring scale.

Experimental data on the saturated vapor line were generated with an apparatus which was built in prior work as described by Gremer et al.[17]

4 Results and discussion

4.1 Nitrogen + acetone

The VLE of the system nitrogen + acetone was measured at temperatures from 223 to 400 K and pressures of up to 12 MPa with respect to the saturated liquid line, cf. Figure 2. The numerical values are listed in Table 1. The saturated vapor line was examined at temperatures from 303 to 343 K and pressures of up to 1.8 MPa, cf. Figure 3. Experimental VLE data at finite mole fractions and also Henry's law constant data can be found in the literature for this mixture. The present results are discussed first on the basis of isothermal pressure-composition phase diagrams. To correlate the present data, the Peng-Robinson EOS[18] with the quadratic mixing rule[19] and the Huron-Vidal mixing rule[21] was used, cf. section 5. The Peng-Robinson EOS was fitted to the entire present experimental data set for the saturated liquid line simultaneously. However, for better visibility, only the results of the Huron-Vidal mixing rule are shown graphically.

The VLE at 303 and 363 K at pressures of up to 3 MPa are presented in Figure 4, where the experimental data of Jabloniec et al.[2] are shown for comparison. The mean deviation of the saturation pressure measured in this work with respect to the Peng-Robinson EOS is less than 2 % for both isotherms. The data points measured by Jabloniec et al.[2] are in line with the Peng-Robinson EOS at 363 K. But at 303 K they exhibit a steeper slope. The mean deviation of the pressure reported by Jabloniec et al.[2] with respect to the Peng-Robinson EOS is around 1.5 % at 363 K and 14 % at 303 K.

A larger temperature and pressure range of the saturated liquid line is presented in Figure 2. An almost linear relation between the mole fraction of the solute and the vapor pressure can be identified. Lower temperatures exhibit a slightly convex shape of the saturated liquid line. With rising temperature, the solubility of nitrogen in acetone is increased. It is also noticeable that the Peng-Robinson EOS agrees very well with the present data for all regarded temperatures.

The present results can also be assessed on the basis of the Henry's law constant, cf. Figure 5. To calculate the Henry's law constant on basis of the present experimental data, an extrapolation procedure at constant temperature as described by Merker et al.[27] was used

$$H_i = \lim_{p \to p^{\mathrm{s}}_{\mathrm{Ac}}} \frac{f_i}{x_i}, \tag{1}$$

where

$$f_i = p \cdot y_i \cdot \varphi_i. \tag{2}$$

f_i and φ_i are the fugacity and the fugacity coefficient of the solute in the mixture, respectively. x_i and y_i are the mole fractions of solute in the liquid and the vapor phase, respectively. The calculated Henry's law constant is listed in Table 3. Henry's law constant data for the mixture nitrogen + acetone were found in several literature sources, cf. Figure 5. The mean deviation of the present Henry's law constant and the data from the literature with respect to the straight line shown in Figure 5 is 2 % and 0.8 %, respectively, which is well within the uncertainty of the present experiments. Only the measurements of Just,[3] which were published 110 years ago, and of Jabloniec et al.[2] at 303 K differ from the remaining ones. For this temperature, Jabloniec et al.[2] reported a Henry's law constant which is 18 % above the straight line. At 363 K, the data of Jabloniec et al.[2] agree much better with the remaining data. Note that this was also observed for the VLE data at finite mole fractions by Jabloniec et al.[2]

The experimental data for the saturated vapor line are presented in Figure 3. These results can only be compared to the Peng-Robinson EOS, because no other experimental data are available in this temperature range. Schlichting et al.[20] covered lower temperatures between 251.8 and 273 K. It is noticeable that the agreement between the Peng-Robinson EOS with the Huron-Vidal mixing rule and the present experimental data with a mean deviation of only 0.01 MPa is best at 323 K. At 303 K (mean deviation of 0.16 MPa) and especially at 343 K (mean deviation of 0.20 MPa), the present measurements exhibit a higher vapor pressure than the Peng-Robinson EOS. Nevertheless, the curvature of the saturated vapor line is in good agreement.

4.2 Oxygen + acetone

The measurement of the system oxygen + acetone required various safety precautions. Especially the handling of pure oxygen upon filling of the cell and adding acetone was critical. In atmospheres of pure oxygen some substances (e.g. lubricants) tend to self-ignite, therefore all tubes and valves were repeatedly cleaned. Protruding burrs at the tubes were removed to avoid pressure surges. For the lubrication of the valves, a dedicated material was selected, which is certified for the use with pure oxygen. To avoid the arcing of a electrostatic charge, every component of the apparatus was grounded. In order to reduce the impact of an explosion, which would lead to a steep pressure rise, only small pressures of up to 0.8 MPa were realized during the measurement. Furthermore, only low temperatures from 253 to 283 K were studied.

The experimental results for the saturated liquid line at 253 and 283 K are presented in an isothermal pressure-composition phase diagram, cf. Figure 6 and Table 5. To correlate the present data, the Peng-Robinson EOS was used, cf. section 5, and fitted to the entire present experimental data set for the saturated liquid line simultaneously. It is hard to distinguish the differences between the present temperatures because only a small pressure range was examined. At 253 K, the experimental data agree well with the Peng-Robinson EOS using the Huron-Vidal mixing rule, especially for lower pressures. At 283 K, the Peng-Robinson EOS shows a slightly steeper slope than the experimental data.

The Henry's law constant of oxygen in acetone from the literature exhibit a large scatter compared with the predominantly consistent data published for nitrogen. For example at 298.15 K, the deviation between the lowest and the highest reported value is 67 MPa. The Henry's law constant of oxygen calculated on basis of the present experimental data at 253 and 283 K with eq. (1), is located in the upper part of the range of previously reported values, cf. Figure 5.

5 Peng-Robinson equation of state

To correlate the experimental data generated in the present work, the Peng-Robinson EOS[18] was used

$$p = \frac{RT}{v-b} - \frac{a}{v \cdot (v+b) + b \cdot (v-b)}. \tag{3}$$

The substance specific parameters a and b were specified as

$$a = (0.45724 \cdot \frac{R^2 {T_c}^2}{p_c}) \cdot [1 + (0.37464 + 1.54226 \cdot \omega - 0.26992 \cdot \omega^2) \cdot (1 - \sqrt{\frac{T}{T_c}})]^2, \tag{4}$$

and

$$b = 0.07780 \cdot \frac{RT_c}{p_c}. \tag{5}$$

Therein, R is the ideal gas constant, v the molar volume, T_c the critical temperature, p_c the critical pressure and ω the acentric factor.

5.1 Quadratic mixing rule

In order to describe mixtures with the Peng-Robinson EOS, the quadratic Van der Waals one-fluid mixing rule[19] was assumed. It states that the pure substance parameters a and b have to be replaced by

$$a_m = \sum_i \sum_j x_i x_j a_{ij}, \tag{6}$$

and

$$b_m = \sum_i x_i b_i. \tag{7}$$

Therein, a_{ij} is defined by

$$a_{ij} = \sqrt{a_i a_j} \cdot (1 - k_{ij}), \tag{8}$$

where k_{ij} is an adjustable binary parameter to correlate experimental data.

The Peng-Robinson parameters for both systems are listed in Tables 4 and 5. To consider the ternary mixture nitrogen + oxygen + acetone, the binary parameter for the subsystem nitrogen + oxygen from prior work of our group[29] can be assumed.

Note that the results of the quadratic mixing rule are not shown in the figures for visibility reasons, because they are very similar to the results of the Huron-Vidal mixing rule.

5.2 Huron-Vidal mixing rule

Another option is the g^E mixing rule from Huron and Vidal.[21] Here, the parameter b_m can be described with eq. (7) and the parameter a_m is written as

$$a_m = b_m \left(\sum_i \frac{a_i}{b_i} x_i - \frac{g_\infty^E}{\Lambda} \right) , \tag{9}$$

where Λ is

$$\Lambda = \frac{1}{2\sqrt{2}} \cdot \ln \left(\frac{2+\sqrt{2}}{2-\sqrt{2}} \right) , \tag{10}$$

if the Peng-Robinson EOS is used. The excess Gibbs free energy at infinite pressure g_∞^E can be calculated with an appropriate g^E model. In the present work, the UNIQUAC model[28] was used for this task, which requires the two binary interaction parameters l_{ij} and l_{ji}. For the system nitrogen + acetone, the g^E mixing rule from Huron and Vidal yields somewhat better results then the quadratic mixing rule. For oxygen + acetone, no significant differences can be identified, given that the experimental database is much smaller. The adjusted binary parameters for both mixing rules are listed in Table 5.

6 Conclusion

New experimental data on fluid phase coexistence of the binary mixtures nitrogen + acetone and oxygen + acetone were reported. On the basis of this information, Henry's law constant data were calculated for both systems. For the system nitrogen + acetone, the present experimental data show a good agreement with most data from the literature. It should be noted that the Henry's law constant at 303 K, as reported recently by Jabloniec et al.,[2] is significantly higher than the remaining data. The same deviation can be seen in the course of the saturated liquid line at 303 K. For the system oxygen + acetone, the Henry's law constant was determined with the same method and a similar accuracy as for the system nitrogen + acetone. The present data are in the range of previously reported experimental results. Furthermore, the Peng-Robinson EOS with the quadratic mixing rule and the Huron-Vidal mixing rule was adjusted to the new experimental data points of the saturated liquid line for both binary systems.

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Table 1: Experimental vapor-liquid equilibrium data along the saturated liquid line and along the saturated vapor line of the mixture nitrogen + acetone generated in this work. The numbers in parentheses indicate the uncertainty in the last digits.

T^a K	p^b MPa	x_{N2} mol/mol	y_{N2} mol/mol
223.2	9.68	0.032(2)	
223.2	2.97	0.011(1)	
223.2	3.79	0.014(1)	
243.1	7.13	0.026(2)	
273.1	7.70	0.033(2)	
303.2	0.39		0.887(5)
303.2	0.43		0.902(5)
303.2	0.45		0.904(5)
303.2	0.56		0.925(5)
303.2	0.61		0.929(5)
303.2	0.61		0.931(5)
303.2	0.76		0.943(5)
303.2	1.20		0.960(5)
303.2	2.09	0.011(1)	
303.1	2.44	0.012(1)	
303.2	4.64	0.024(1)	
303.2	6.95	0.035(2)	
303.2	8.70	0.044(3)	
303.1	10.35	0.051(3)	
303.2	11.07	0.054(3)	
303.2	11.99	0.058(4)	
323.2	0.90		0.897(5)
323.0	1.03		0.909(5)
323.2	1.10		0.916(5)
323.0	1.25		0.926(5)
323.2	1.34		0.930(5)
323.2	2.00	0.011(1)	
323.2	2.35	0.013(1)	
323.2	4.38	0.025(2)	
323.2	6.65	0.037(2)	
323.2	8.15	0.045(3)	
323.2	9.73	0.053(3)	
323.2	10.22	0.056(3)	
323.2	11.14	0.060(4)	
343.0	1.58		0.869(5)
343.0	1.68		0.879(5)
343.0	1.78		0.882(5)
343.2	1.98	0.011(1)	
343.2	4.19	0.025(2)	
343.1	6.22	0.037(2)	
343.1	7.71	0.047(3)	
343.2	9.07	0.056(3)	
343.2	10.38	0.063(4)	
363.2	2.00	0.012(1)	
363.1	2.43	0.015(1)	
363.2	4.08	0.026(2)	
363.2	8.63	0.058(4)	
400.0	2.65	0.016(1)	
400.0	3.62	0.024(2)	

[a] The uncertainty in terms of temperature is 0.04 K. [b] The uncertainty in terms pressure is 0.01 MPa.

Table 2: Experimental vapor-liquid equilibrium data along the saturated liquid line of the mixture oxygen + acetone generated in this work. The numbers in parentheses indicate the uncertainty in the last digits.

T^a K	p^b MPa	x_{O2} mol/mol
253.1	0.108	0.0007(1)
253.1	0.380	0.0026(2)
253.1	0.562	0.0040(2)
253.1	0.731	0.0053(3)
283.1	0.121	0.0007(1)
283.2	0.395	0.0028(2)
283.2	0.582	0.0042(3)
283.1	0.753	0.0056(3)

[a] The uncertainty in terms of temperature is 0.04 K. [b] The uncertainty in terms of pressure is 0.0006 MPa.

Table 3: Henry's law constant of nitrogen in acetone and of oxygen in acetone calculated on basis of the experimental data generated in this work. The numbers in parentheses indicate the uncertainty in the last digits.

T K	H_{N2} MPa	H_{O2} MPa
223	252(16)	
253		150(9)
283		143(9)
303	187(12)	
323	171(11)	
343	157(10)	
363	141 (9)	

Table 4: Pure substance parameters of the Peng-Robinson equation of state.[30]

Fluid	T_c K	p_c MPa	ω
Nitrogen	126.2	3.394	0.040
Oxygen	154.6	5.046	0.021
Acetone	508.1	4.701	0.309

Table 5: Binary parameters of the Peng-Robinson equation of state for the quadratic (k_{ij}) and the Huron-Vidal (l_{ij}, l_{ji}) mixing rule. The parameter for nitrogen + oxygen was taken from prior work.[29]

Mixture	k_{ij}	l_{ij}	l_{ji}
Nitrogen + Acetone	0.2086	241.91	926.52
Oxygen + Acetone	0.2908	488.96	889.29
Nitrogen + Oxygen	-0.0119	-	-

Figure 1: Schematic of the new apparatus that was employed for the present gas solubility measurements. VX indicates a valve, TW-VX a three-way valve, TX a thermometer and PX a pressure transducer.

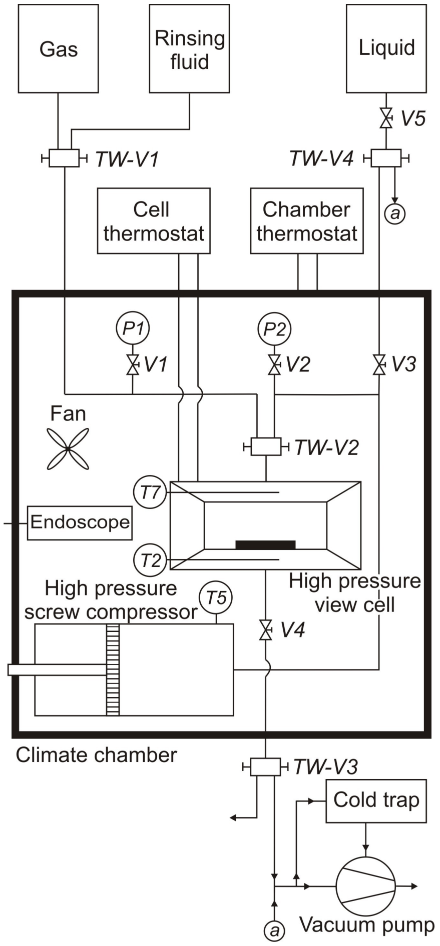

Figure 2: Saturated liquid line of nitrogen + acetone: ♦, ◊ experimental data, this work; —, - - - Peng-Robinson equation of state (Huron-Vidal mixing rule). The solid lines show the same isotherms as the full symbols.

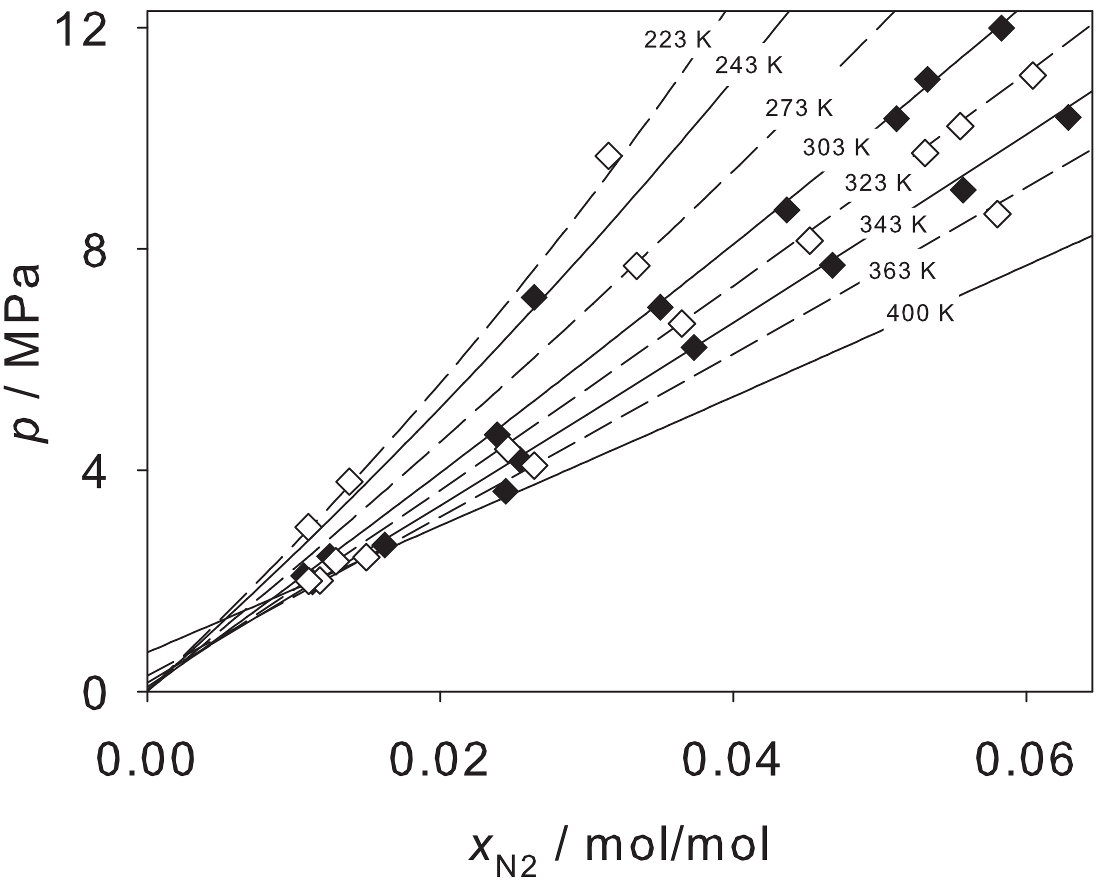

Figure 3: Saturated vapor line of nitrogen + acetone: ♦, ◊ experimental data, this work; —, - - - Peng-Robinson equation of state (Huron-Vidal mixing rule).

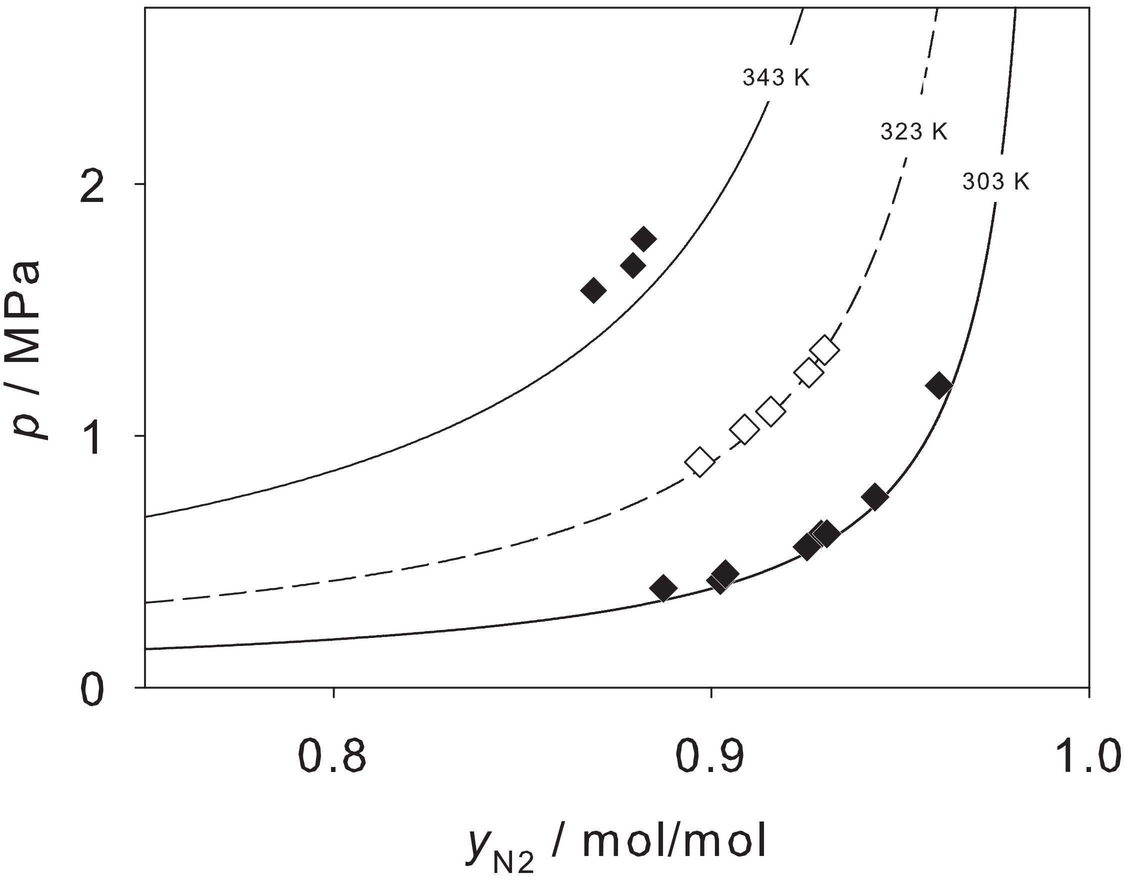

Figure 4: Saturated liquid line of nitrogen + acetone: ♦, ◊ experimental data, this work; ▲, △ experimental data, Jabloniec et al.;[2] —, - - - Peng-Robinson equation of state (Huron-Vidal mixing rule).

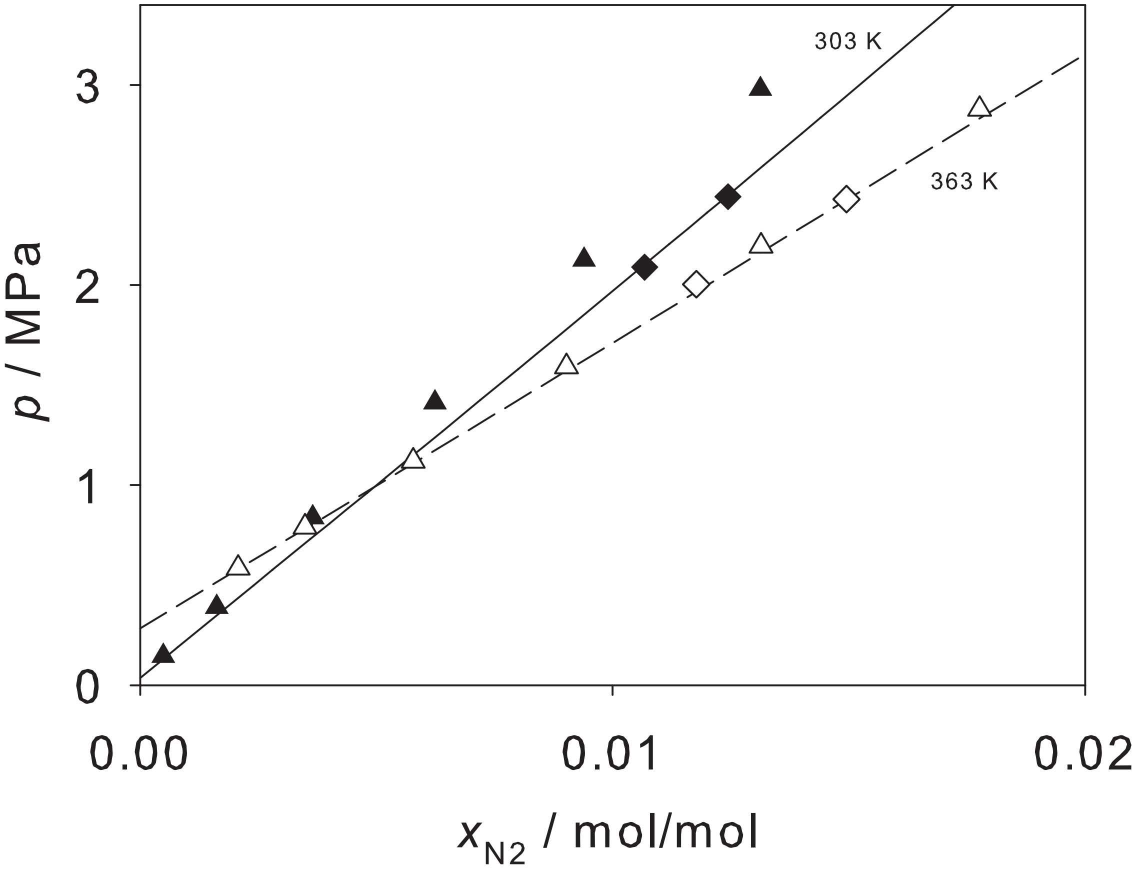

Figure 5: Henry's law constant. Nitrogen in acetone: □ this work, ▲ Jabloniec et al.,[2] • Just,[3] ★ Horiuti,[4] ▼ Kretschmer et al.,[5] ■ Nitta et. al,[6] + Vosmansky and Dohnal,[7] × Tsuji et al.;[8] Oxygen in acetone: ○ this work, + Levi,[9] ∗ Fischer and Pfleiderer,[10] ▼ Finlayson,[11] ★ Horiuti,[4] ■ Kretschmer et al.,[5] • Schlaepfer et al.,[12] ⧫ Sinn et al.,[13] - Naumenko,[14] ▲ Bub and Hillebrand,[15] | Tsuji et al.,[8] × Luehring and Schumpe;[16] — straight line. All points above the dotted line represent nitrogen data, the others represent oxygen data.

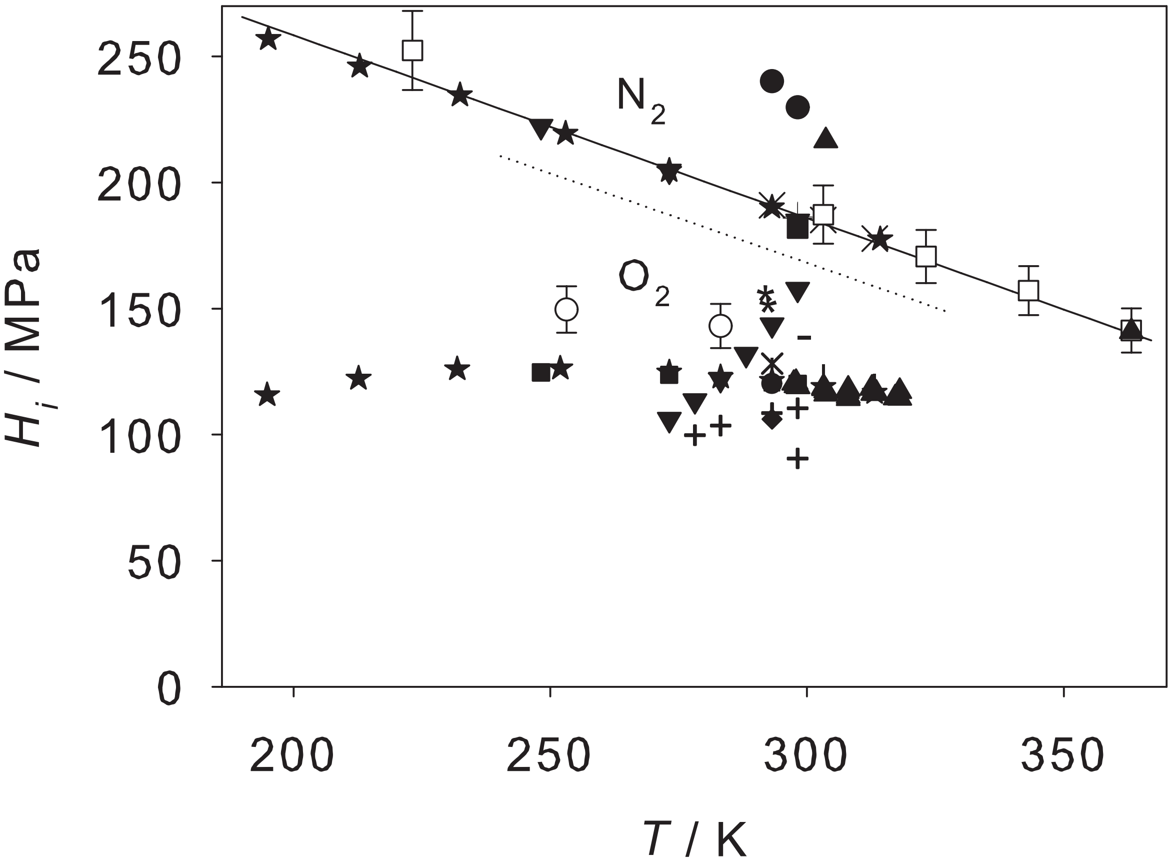

Figure 6: Saturated liquid line of oxygen + acetone: ♦, ◊ experimental data, this work; —, - - -Peng-Robinson equation of state (Huron-Vidal mixing rule). The solid line shows the same isotherm as the full symbols.