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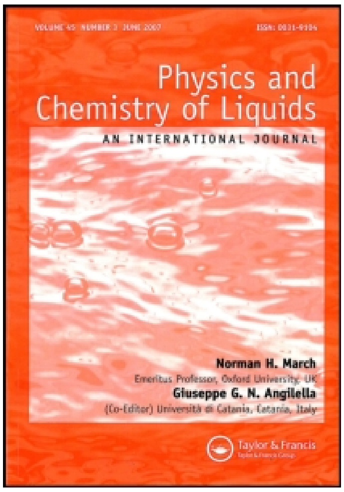

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Dew Points of Binary Methane + Water: Measurement and Correlation

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DEW POINTS OF BINARY METHANE + WATER: MEASUREMENT AND CORRELATION

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Experimental measurements of dew points for four methane + water mixtures between 2.6×10^5 Pa and 100.4×10^5 Pa and the temperature range from 225.5 K to 272.7 K were carried out. The experimental results were analysed in terms of both an equation of state model and an excess function-equation of state method, which reproduced the experimental data within AAD from 1.8 to 3.1 K and from 0.9 to 1.8 K, respectively.

Keywords: Dew point; Equation of state; Excess function

INTRODUCTION

Natural gas transported through a gas pipeline can contain different levels of water. Because of this, there is a risk of undesired formation of ice or hydrates and the corrosion of pipes or blockages during transport of natural gas. In order to understand the influence of water on the vapour–liquid equilibrium of natural gases, the system constituted

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by water and methane, the majority component of natural gas, is studied in the present work.

A water dew point generation experimental apparatus was built and commissioned [1]. The results on four methane + water mixtures between 2.6×10^5 Pa and 100.4×10^5 Pa, temperatures from 225.5 K to 272.7 K and water contents of $12.4 \times 10^{-6}\,\mathrm{kg\,m^{-3}}$ (n) to $51.2 \times 10^{-6}\,\mathrm{kg\,m^{-3}}$ (n) are presented here.

The experimental results obtained on the binary system were analysed in terms of both an equation of state (EOS) model and an excess function-equation of state (EOS) method, which reproduced the experimental data within AAD from 1.8 to 3.1 K and from 0.9 to 1.8 K, respectively.

EXPERIMENTAL

The dew point range from 2.6×10^5 to 100.4×10^5 Pa and temperatures from 225.5 to 272.7 K. Methane was supplied by Air Liquide with the specified purity of 99.95% and was used without treatment.

The dew point generation apparatus used for our experimental data collecting was built in previous works [1]. The experimental method for this work is based on the generation of saturated gases with water by condensation of this compound in two successive temperature-controlled condensers with continuous gas flow at specified pressures.

After controlled expansion, the gas is saturated with water vapour by flowing through liquid water in an isolated saturator held at laboratory temperature. The temperature controlled of water condensation is then achieved in two successive stainless steel condensers. The first condenser temperature is set to a value lying between ambient and the temperature of the second condenser. Doing so, the quantity of liquid collected into the second condenser is minimised. The concentration of water in the gas is measured at the outlet of the dew point generation system, using a Karl Fischer titration, according to the standard method [2] at atmospheric pressure. By doing so, the water content reference value of the gaseous phase is obtained.

The dew point values of the methane + water mixtures are measured by means of a chilled mirror instrument [3]. The chilled mirror instrument input pressure is set using a regulator valve, when the apparatus reaches a stable value of dew temperature, both pressure and temperature are recorded. In this way, the values of the temperature and pressure of the dew point curve of the mixture generated are obtained.

To analyse the content of water and to carry out the dew point measurements the following instrumentation is used:

- Mitsubishi CA 06 Karl Fischer Titrator. Coupled with an Elster wet gasmeter Type Gr. 00, E51, 0.2% accuracy.
- MBW Dew Point Instrument Mod. DP3-D-HP-K2. The cooling of the mirror is achieved by a cascaded Peltier elements and the dew point mirror temperature is opto-electronically controlled.
- Pressure Transmitter. With a maximum error of 0.1% in the calibrated range.

Prior to this study of methane + water dew points, the performance of both analytical methods and experimental procedures was determined. The uncertainty of the water dew point measurements, given by the supplier of the MBW Dew Point Instrument was of ± 0.2 K for dew point temperatures from 273.2 to 228.2 K and of ± 0.3 K for dew point temperature between 228.2 and 198.2 K.

To obtain the precision of the analysis of water content, repeated analysis of water content of a standard nitrogen + water mixture prepared by Air Liquide were carried out. The measured values were equal to the standard water content within a rejecting percentage of 0.05% [4]. The absolute average deviation was of $0.5 \times 10^{-6}\,\mathrm{kg\,m^{-3}(n)}$ for a mean value of water content of $59.0 \times 10^{-6}\,\mathrm{kg\,m^{-3}(n)}$.

In order to evaluate the precision of water dew point generation, repeated generation of methane + water were carried out, and the water content and the dew point curve were measured. The results obtained in the performance evaluation are the following:

- For water content: the absolute average deviation was of $1.0 \times 10^{-6}\,\mathrm{kg\,m^{-3}(n)}$ for a mean water content of $19.6 \times 10^{-6}\,\mathrm{kg\,m^{-3}(n)}$.
- For dew point pressure: the relative average deviation was 3% on a range from 2.1×10^5 Pa to 71.1×10^5 Pa.
- For dew point temperature: the absolute average deviation was 0.4 K on a range from 225.8 K to 259.1 K.

Reference conditions for volume are 273.15 K and 1.01325×10^5 Pa. The test was achieved on a water dew point of 72×10^5 Pa and 258.1 K in pure methane.

RESULTS

The water amounts in the vapour phase and the dew point curves of the mixture generated with the moisture generation system were determined and the results of the experiments are collected in Table I.

Theory

Introduction

Equations of state such as Santis–Breedveld–Prausnitz EOS [5], Nakamura–Breedveld–Prausnitz EOS [6], Peng–Robinson EOS [7] and Robinson–Peng–Ng [8] yield good results in calculation of water dew point of natural gases at higher temperatures than those of the natural gas pipeline network.

In this work a model based on a modified Peng–Robinson EOS [9] is used in order to obtain a good description of water vapour pressure on both ice and liquid water. This equation allows predicting properly the water dew point curve in the usual temperature and pressure ranges of natural gas in transmission through pipeline.

On the other hand, classical models such as UNIFAC [10], DISQUAC [11] or modified UNIFAC [12] allow predicting the vapour–liquid equilibrium under low pressure for systems which contain a polar component but are not available for high pressure calculations. We use, in this work, an excess function-EOS method developed by Péneloux *et al.* [13] founded on the zeroth order approximation of Guggenheim's model. For the EOS based models available experimental binary systems data are required to obtain the interaction parameters. In the excess function-EOS models, they are calculated by means of a group contribution method what makes it useful to predict the dew point curves of real natural gases, provided that, not always binary experimental data for all the components of the so-called C6+ fraction exist. Moreover, this work is part of a research which aims to study the influence of the presence of methanol, as an additive of nat-

TABLE I Experimental dew points temperatures and pressures for {methane + $\bar{\rho}_{\text{water}}$} mixtures

T K	P 10^5 Pa	T K	P 10^5 Pa
$\bar{\rho}_{\text{water}} = 12.4 \times 10^{-6}\,\text{kg m}^{-3}$(n)		$\bar{\rho}_{\text{water}} = 18.1 \times 10^{-6}\,\text{kg m}^{-3}$(n)	
225.5	2.9	232.0	3.6
230.2	5.2	235.4	5.4
237.0	9.9	241.6	10.6
241.1	15.1	244.8	15.0
244.0	20.0	247.5	20.1
246.1	24.8	250.1	25.4
248.1	30.1	252.5	29.9
249.5	35.4	254.0	35.2
250.7	40.5	255.3	40.3
251.9	45.6	256.5	46.0
252.7	49.9	257.4	50.1
253.6	54.5	258.4	55.1
254.2	58.9	259.2	59.9
255.1	64.3	259.7	65.1
255.8	70.0	260.5	69.9
256.4	74.6	261.2	76.4
256.8	79.7	261.7	80.2
257.5	85.2	262.1	84.3
257.8	91.4	262.6	90.0
258.1	96.0	263.0	95.1
258.5	100.3	263.4	100.0
$\bar{\rho}_{\text{water}} = 31.4 \times 10^{-6}\,\text{kg m}^{-3}$(n)		$\bar{\rho}_{\text{water}} = 51.2 \times 10^{-6}\,\text{kg m}^{-3}$(n)	
233.0	2.6	239.6	3.1
239.4	5.2	244.8	5.3
245.4	10.1	251.1	10.1
249.2	14.8	255.2	15.1
252.2	20.3	257.9	20.1
254.2	24.9	260.3	26.0
256.0	30.4	261.9	30.4
257.3	35.3	263.5	35.7
258.7	39.6	264.5	40.1
260.3	45.1	265.7	45.6
261.6	50.7	266.1	49.9
262.9	56.3	267.2	55.4
263.8	60.7	268.0	60.0
263.9	65.5	268.7	64.7
264.4	69.0	269.4	69.9
265.2	75.1	270.1	74.9
265.9	80.4	270.7	80.0
266.4	85.3	271.3	85.3
267.1	91.2	271.8	90.0
267.6	96.2	272.2	95.1
268.0	100.1	272.1	100.4

ural gas, in the water dew point of natural gases [14]. The excess function-EOS method used in this work allows to be modified to have into account the self-association of methanol in the mixtures.

In order to evaluate these two theoretical models for the prediction of water dew points of the studied system in the temperature and pressure ranges used, a comparison between experimental and calculated values was carried out. The values of dew temperature of the vapour phase for the studied systems are calculated by means of both theoretical methods using the experimental values of pressure and composition obtained in the present work.

Description of EOS Model

The EOS model used in this work is based on a modified Peng-Robinson EOS [9] in order to obtain a good description of water vapour pressure both on ice and liquid water. The binary interaction parameters between the natural gas components and water were also obtained.

The equation of state used is the Peng–Robinson cubic equation of state [7] of the form:

$$P = \frac{RT}{v-b} - \frac{a}{v^2 + 2bv - b^2} \tag{1}$$

$$\text{with } b(T) = b(T_c) \quad \text{and} \quad a(T) = a(T_c)\alpha(T_r, \omega) \tag{2}$$

where

$$\alpha^{1/2} = 1 + \kappa(1 - T_r^{1/2}) \quad \text{and} \quad \kappa = 0.374640 + 1.54226\omega - 0.26992\omega^2 \tag{3}$$

In case of water the following equation is used [9]:

$$\alpha^{1/2} = A_0 + A_1(1 - T_r^{1/2}) + A_2(1 - T_r^{1/2})^2 + A_3(1 - T_r^{1/2})^4 \tag{4}$$

In this model, different values of A_0, A_1, A_2 and A_3 coefficients are used if the temperature is higher or lower than 273.15 K.

For calculation of parameters a and b in case of mixtures, classical mixing rules are used of the form [9]:

$$a = \sum_{i=1}^{n} x_i x_j a_{ij} \quad \text{and} \quad a_{ij} = \sqrt{a_i a_j}(1 - k_{ij}) \tag{5}$$

$$b = \sum_{i=1}^{n} x_i b_i \quad \text{with} \quad k_{ij} = k_{ji} \quad \text{and} \quad k_{ii} = k_{jj} = 0 \tag{6}$$

Due to the change of the function α for water, new binary interaction parameters for natural gas components and water were obtained [9]. In case of $CO_2 + H_2O$, $CH_4 + H_2O$ and $C_2H_6 + H_2O$, the following temperature function has to be used:

$$k_{ij}(T) = k_{ij,0} + k_{ij,1}\left(\frac{T}{273.15} - 1\right) \tag{7}$$

Description of the Excess Function-EOS Model

In order to represent the vapour–liquid equilibrium of the mixture studied, a model founded on the zeroth approximation of Guggeheim's reticular model was selected. The model satisfies two important conditions:

1. The Helmholtz energies of pure components are calculated by an equation of state.
2. The excess functions are obtained at constant packing fraction, η. That is to say the value of the packing fraction, η, for each component of the mixture is the corresponding value to the pure component. This assumption leads to:

$$\eta = \frac{b}{v} = \frac{b_i}{v_i} \quad (i = 1, \ldots, p) \tag{8}$$

The molar Helmholtz energy of a mixture, A, may be written as follows:

$$A = A^{\mathrm{id}} - RT\ln(1 - \eta) - \sum_{i=1}^{p} \frac{x_i a_i}{b_i} Q(\eta) + A^{\mathrm{E}}_{\mathrm{res}} \tag{9}$$

where A^{id} is the ideal mixture molar Helmholtz energy, a_i is the attractive parameter of i component of a translated Peng–Robinson cubic equation of state [15,16], b_i is the component i covolume, $A^{\mathrm{E}}_{\mathrm{res}}$ is the residual excess Helmholtz energy (explained in other section) and $Q(\eta)$ is expressed as follows:

$$Q(\eta) = \int_0^{\eta} \frac{Q'(\eta)}{\eta} d\eta \tag{10}$$

where

$$Q'(\eta) = \frac{\eta}{1+\gamma\eta} \quad \text{and} \quad \gamma = 2(\sqrt{2}+1) \tag{11}$$

$A^{\mathrm{E}}_{\mathrm{res}}$ is written by means of a formalism that enables to separate the composition and packing fraction variables:

$$A^{\mathrm{E}}_{\mathrm{res}} = E(T,x)Q(\eta) \tag{12}$$

For the first term on the right hand side of Eq. (6) the following equations are used [17]:

$$E(T,x) = \frac{1}{2q_m}\left[\sum_{i=1}^{p} q_i x_i \left[\sum_{j=1}^{p} q_j x_j K_{ij}\right] + \sum_{i=1}^{p} q_i x_i \left[\sum_{j=1}^{p} q_j^{1/3} x_j L_{ji}^{1/3}\right]\right] \tag{13}$$

$$\text{with} \quad K_{ij} = \frac{E^1_{ij} + E^2_{ij}}{2} \quad \text{and} \quad L_{ij} = E^2_{ij} - E^1_{ij} \quad L_{ij} = -L_{ji} \tag{14}$$

$$q_m = \sum_{k=1}^{p} q_k x_k \quad \text{and} \quad q_k = \delta_k b_k \tag{15}$$

where the subscripts i and j are referred to the components i and j of the mixture with p components, q_i is the molecular surface of the component i and it is supposed that $q_i/q_j = (b_i/b_j)\delta$, δ being an adjustable parameter. K_{ij} and L_{ij} are two binary interaction parameters between components i and j, which depend on the terms of the interchange energy, E^1_{ij} and E^2_{ij}, calculated using a group contribution method

TABLE II Values of the group interaction parameters, ${}_1A^0_{kl}$, ${}_1B^0_{kl}$, ${}_2A^0_{kl}$ and ${}_2B^0_{kl}$, used in this work

Binary	${}_1A^0_{kl}$	${}_1B^0_{kl}$	${}_2A^0_{kl}$	${}_2B^0_{kl}$
Methane + water	1279.540 [17]	– 0.726 [17]	4279.212*	5.537*

*This work.

as follows [17]:

$$E^1_{ij} = -\frac{1}{2}\sum_{k=1}^{N}\sum_{l=1}^{N}(\alpha_{ik}-\alpha_{jk})(\alpha_{il}-\alpha_{jl})A^1_{kl}(T) \quad \text{with} \quad A^1_{kl} = {}_1A^0_{kl}\left(\frac{T^0}{T}\right)^{{}_1B^0_{kl}} \tag{16}$$

$$E^2_{ij} = -\frac{1}{2}\sum_{k=1}^{N}\sum_{l=1}^{N}(\alpha_{ik}-\alpha_{jk})(\alpha_{il}-\alpha_{jl})A^2_{kl}(T) \quad \text{with} \quad A^2_{kl} = {}_2A^0_{kl}\left(\frac{T^0}{T}\right)^{{}_2B^0_{kl}} \tag{17}$$

where ${}_1A^0_{kl}$, ${}_1B^0_{kl}$, ${}_2A^0_{kl}$ and ${}_2B^0_{kl}$ are group interaction parameters. In this work, we obtain some of these parameters using the results of methane + water system presented in this paper. The values of these parameters used in this work are presented in Table II.

DISCUSSION

The experimental dew point curves and calculated with both the EOS model and the excess function-EOS method are represented in Fig. 1. As it can be seen in Fig. 1 an increase of water content in the mixtures of the system studied leads to a displacement of the dew point to higher values of temperature and pressure.

Even if the experimental data presented here for methane + water system have been collected in the pressure–temperature gas–hydrate equilibrium region, no experimental evidence of hydrates presence, as blockages in the experimental device, has been observed. This fact can be explained as follows: The operations procedure used in this work is represented as "way 1" in Fig. 2. Following this procedure, even if the final point is located in gas–hydrate equilibrium region,

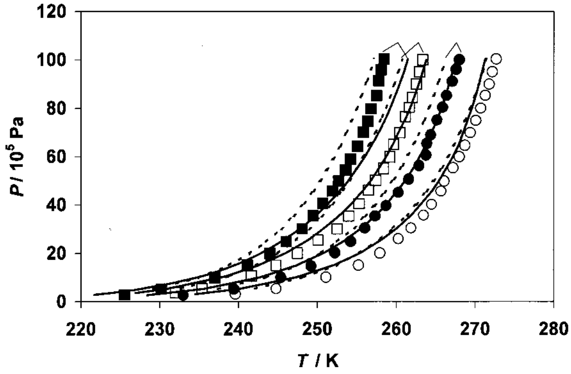

FIGURE 1 Comparison between experimental dew points curves (symbol) and calculated with the EOS model (dotted line) and with the excess function-EOS method (line) for the system {methane + $\bar{\rho}_{\text{water}}$}: ■, $\bar{\rho}_{\text{water}} = 12.4 \times 10^{-6}\,\text{kg}\,\text{m}^{-3}$; □, $\bar{\rho}_{\text{water}} = 18.1 \times 10^{-6}\,\text{kg}\,\text{m}^{-3}$(n); ●, $\bar{\rho}_{\text{water}} = 31.4 \times 10^{-6}\,\text{kg}\,\text{m}^{-3}$(n); ○, $\bar{\rho}_{\text{water}} = 51.2 \times 10^{-6}\,\text{kg}\,\text{m}^{-3}$(n).

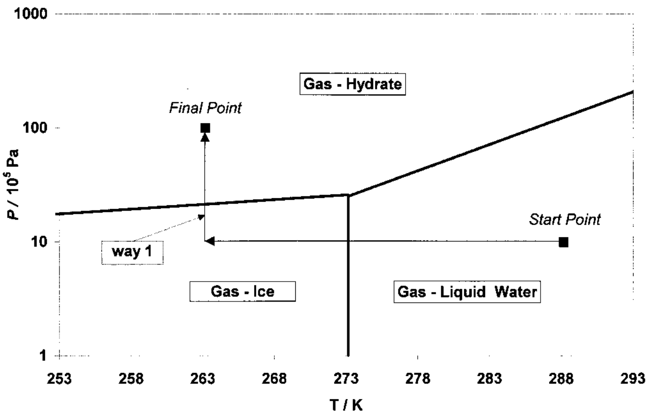

FIGURE 2 Representation of the pressure–temperature regions of gas–hydrate, gas–ice and gas–liquid water equilibria for the system {methane + $\bar{\rho}_{\text{water}}$} [18]. As "way 1" the operational procedure used in the water dew point generation apparatus is illustrated.

this equilibrium is kinetically unfavourable as contrasted with metastable ice–gas equilibrium [9]. Moreover, in case of gas–hydrates equilibrium, the theoretically calculated water contents for our system [18] are much higher (about 20% as relative average deviation value) than the experimental ones.

TABLE III Values of ADD[1] (EOS model) and AAD[2] (excess function-EOS method) and experimental ranges of dew temperatures and pressures for mixtures {methane + $\bar{\rho}_{\text{water}}$}

$\bar{\rho}_{\text{water}}$ $10^{-6}\,\text{kg}\,\text{m}^{-3}$(n)	*T range* K	*P range* 10^5 Pa	*AAD[1]* K	*AAD[2]* K
12.4	225.5–258.5	2.9–100.3	2.1	1.5
18.1	232.0–263.4	3.6–100.0	3.1	1.6
31.4	233.0–268.0	2.6–100.1	1.8	0.9
51.2	239.6–272.7	3.1–100.4	1.9	1.8

A comparison between experimental and calculated values was carried out and the results obtained for each dew point curve are presented in Table III. The results are quite good with values of AAD for dew point temperature between 1.8 K and 3.1 K with the EOS model and from 0.9 K to 1.8 K for excess function-EOS method.

In the EOS model an influence of water content in AAD values is observed. The greatest deviations occur for the mixtures with the lowest water contents. It can be due to the experimental error for the very low water content analysis. No influence of pressure and temperature has been seen in values of AAD for both theoretical models.

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APPENDIX 1

For comparison between calculated and experimental dew point temperature for each studied dew point curve we used the deviation

$$\mathrm{AAD} = \frac{1}{N}\sum_{i=1}^{N} |T_i^{\mathrm{exp}} - T_i^{\mathrm{cal}}|$$

where N is the number of dew points that constitute a dew point curve.