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J. Chem. Thermodynamics 40 (2008) 208-211

Critical parameters for propane determined by the image analysis

Y. Honda, T. Sato, M. Uematsu \*

Center for Multiscale Mechanics and Mechanical Systems, Keio University, Hiyoshi 3-14-1, Kohoku-ku, Yokohama 223-8522, Japan

Received 21 May 2007; received in revised form 28 June 2007; accepted 29 June 2007 Available online 13 July 2007

### Abstract

The  $(p, \rho, T)$  measurements and visual observations of the meniscus for propane were carried out carefully in the critical region over the range of temperatures:  $-60 \text{ mK} \leq (T - T_c) \leq 40 \text{ mK}$  and of densities:  $-4 \text{ kg} \cdot \text{m}^{-3} \leq (\rho - \rho_c) \leq 6 \text{ kg} \cdot \text{m}^{-3}$  by a metal-bellows volumometer with an optical cell. Vapour pressures were also measured at T = (320.000, 343.132, 369.000, and 369.625) K. The critical point of  $T_c$ ,  $\rho_c$ , and  $\rho_c$  was determined by the image analysis of the critical opalescence. Comparisons of the critical parameters with values given in the literature are presented. © 2007 Elsevier Ltd. All rights reserved.

Keywords: Critical parameters; Image analysis; Propane;  $(p, \rho, T)$  Measurements; Vapour pressure

## 1. Introduction

In previous publications [1,2], we reported measurements of thermodynamic properties of propane in the compressed liquid phase between the temperatures (280 and 400) K at pressures up to 200 MPa by a metal-bellows variable volumometer. In this paper, the results of vapour pressure measurements,  $(p, \rho, T)$  measurements, and visual observations of the meniscus for propane in the critical region using an optical cell and a metal-bellows volumometer are presented. The critical parameters were determined by the image analysis [3] of the critical opalescence. Comparisons of the critical parameters with values given in the literature [4–13] are also reported.

## 2. Experimental

# 2.1. Materials

The propane was supplied by the Takachiho Chemical Industrial Co., Ltd., Ibaragi, Japan, and its mol-fraction purity was specified by the supplier to be 0.99997. The sample to be loaded was degassed five times by freeze-thaw cycling with liquid nitrogen before loading. The sample loaded in this study is supplied by the same cylinder as the previous work [1,2] used.

### 2.2. Apparatus and experimental procedure

The measurements were carried out using an optical cell and a metal-bellows volumometer in a pressure vessel. The apparatus and experimental procedures have been described in detail in our previous publications [14,15].

A sample of known mass was loaded into the optical cell and the metal-bellows volumometer. Nitrogen gas from a pressure-measurement system was supplied to the outside of the bellows in the pressure vessel to compress or expand the bellows. The inner volume of the optical cell and the volumometer as well as its variation with the bellows displacement, temperature, and pressure were calibrated with the known density of water [16]. The inner volume can change from 18 cm<sup>3</sup> to 12 cm<sup>3</sup>. The sample density  $\rho$  was calculated from the mass of the sample loaded and the inner volume of the optical cell and the volumometer. The uncertainty in the resulting density measurements was estimated to be less than  $\pm 0.8 \text{ kg} \cdot \text{m}^{-3}$  at a 95% confidence level.

The optical cell and the volumometer were immersed in the thermostatted bath filled with silicon oil. The temperature that was detected at the well drilled in the body of the

<sup>\*</sup> Corresponding author. Tel.: +81 45 566 1497; fax: +81 45 566 1495. E-mail address: uematsu@mech.keio.ac.jp (M. Uematsu).

<sup>0021-9614/\$ -</sup> see front matter @ 2007 Elsevier Ltd. All rights reserved. doi:10.1016/j.jct.2007.06.021

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volumometer was measured with a  $25 \Omega$  platinum resistance thermometer (Tinsley: 5187SASS) and a thermometer bridge (Tinsley: type 5840) on ITS-90. The resistance of the thermometer at the triple-point temperature of water was measured periodically. The temperature in the bath was controlled within  $\pm 5$  mK. The uncertainty in the temperature measurements was estimated to be less than  $\pm 7 \text{ mK}$  at a 95% confidence level. The pressure of the nitrogen gas was measured with an air-piston pressure gauge (Ruska: model 2465-753). The pressure of the sample was obtained by subtracting the difference between the internal and external pressures of the bellows from the pressure of the nitrogen gas outside the bellows. This pressure difference was calibrated as a function of the bellows displacement, temperature, and pressure. The uncertainty in the pressure measurements was estimated to be less than  $\pm 1.6$  kPa at a 95% confidence level.

Through the 8 mm diameter windows, which were set on two sides of the optical cell, the meniscus at the (vapour + liquid) interface of the sample and its critical opalescence were observed by a CCD camera (Elmo: UN411). The pictures were monitored on a display and recorded by the personal computer used. Each window was sealed by a synthetic sapphire, whose dimensions were 12 mm in thickness and 16 mm in diameter. The height of the space where the sample was loaded in the optical cell and volumometer was designed to be as small as possible. Two windows were also set on two sides of the thermostatted oil bath.

## 3. Results and discussion

We measured the vapour pressure for propane at T = (320.000, 343.132, 369.000, and 369.625) K with different positions of the meniscus to confirm no impurity effect. The mean values of these measurements are given in table 1. Comparison of the present vapour pressure measurements with the experimental data by Miyamoto and Uematsu [1] and Thomas and Harrison [8] is also given in table 1. The present values are in good agreement with those data within the present uncertainties, although the pressure difference between the present values and the experimental data by Thomas and Harrison are 1.12 kPa at T = 343.132 K.

The  $(p, \rho, T)$  measurements were carried out in the region of T = (369.950 to 370.050) K and of  $\rho = (218.0 \text{ to } 1000 \text{ cm})$ 

Results and comparison	of the vapour pressur	re measurements for	r propane

<i>T</i> /K	<i>p</i> <sup>s</sup> /MPa			ε/kPa <sup>a</sup>
	This work	Miyamoto and Uematsu [1]	Thomas and Harrison [8]	
320.000	1.6002	1.5998		0.4
343.132	2.5873			
343.15 <sup>b</sup>			2.58618	1.12
369.000	4.1843	4.1844		-0.1
369.625	4.2316			
369.65 <sup>b</sup>			4.23160	0.00

<sup>a</sup> Pressure deviation of the literature data from values of this work.

<sup>b</sup> Temperature is based on IPTS-68.



FIGURE 1. The  $(p, \rho, T)$  measurements and visual observations of the meniscus for propane at T = (369.950 to 370.050) K and at  $\rho = (218.0 \text{ to } 228.0) \text{ kg} \cdot \text{m}^{-3}$ . \* pictures observed with meniscus.

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