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Critical parameters for isobutane determined by the image analysis

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Abstract

 (p, ρ, T) Measurements and visual observations of the meniscus for isobutane were carried out carefully in the critical region over the range of temperatures: $-15 \text{ mK} \leq (T - T_c) \leq 35 \text{ mK}$, and of densities: $-7.5 \text{ kg} \cdot \text{m}^{-3} \leq (\rho - \rho_c) \leq 7.5 \text{ kg} \cdot \text{m}^{-3}$ by a metal-bellows volumometer with an optical cell. Vapor pressures were also measured at T = (310, 405, 406, 407, and 407.5) K. The critical point of T_c and ρ_c was determined by the image analysis of the critical opalescence which is proposed in this study. The critical pressure p_c was determined to be the pressure measurement at the critical point. Comparisons of the critical parameters with values given in the literature are presented.

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Keywords: Critical parameters; Image analysis; Isobutane; (p, ρ, T) Measurements; Vapor pressure

1. Introduction

In previous publications [1,2] we reported measurements of thermodynamic properties of isobutane in the compressed liquid phase between the temperatures (280 and 440) K at pressures up to 200 MPa by a metal-bellows variable volumometer. In this paper, the results of (p, ρ, T) measurements and visual observations of the meniscus for isobutane in the critical region using an optical cell and a metal-bellows volumometer are presented. The critical parameters were determined by the image analysis of the critical opalescence. Comparisons of the critical parameters with values given in the literature are also reported.

2. Experimental

2.1. Materials

The isobutane was supplied by the Takachiho Chemical Industrial Co., Ltd., Ibaragi, Japan, and its volume fraction purity was specified by the supplier to be 0.9999. 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 [3,4].

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. The inner volume can change from 18 cm³ to 12 cm³. The sample density ρ 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 ± 0.8 kg \cdot m⁻³ at a 95% confidence level.

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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 volumometer was measured with a 25Ω platinum resistance thermometer (Chino: model R 800-2) and a thermometer bridge (Tinsley: type 5840) on the International Temperature Scale of 1990 (ITS-90) [5]. The resistance of the thermometer at the triple-point temperature of water was measured periodically. 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 (Raska: 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 ± 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 vapor-liquid interface of the sample and its critical opalescence were observed by a CCD camera. The pictures were monitored on a display and recorded by a video tape recorder. 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.

2.3. Determination of the critical point by the image analysis

In our previous publications [3,4] the critical point of fluids and fluid mixtures was visually determined using the pictures taken by the CCD camera as the state where the critical opalescence is observed the most intensely with disappearance of the vapor-liquid interface. In this study we introduced image analysis for the pictures into the determination of the critical point.

The picture taken by the CCD camera was a rectangle of 320×240 pix. We cut a round image of 52 pix in diameter, in which the behavior of the sample was taken, from the picture. A color image was then transformed into monochromatic image for determination of relative brightness (L_R) of each picture. The relative brightness at each point in the picture is determined in comparison with the scale on which black and white are assigned the values of 0 and 255, respectively. Figure 1(a) shows six pictures taken by the CCD camera for isobutane at a density of 228.0 kg · m⁻³ with increasing temperature by 10 mK from T = (407.870 to 407.920) K. Each picture was taken after the temperature and density were kept constant at least for 10 h. We can clearly distinguish the existence of the meniscus at the center of the picture at T = 407.870 K.



FIGURE 1. (a) Pictures of the critical opalescence for isobutane at $\rho = 228.0 \text{ kg} \cdot \text{m}^{-3}$. 1, +, T = 407.870 K; 2, ×, T = 407.880 K; 3, \bigcirc , T = 407.890 K; 4, \square , T = 407.900 K; 5, \triangle , T = 407.910 K; 6, \bullet , T = 407.920 K. (b) Distribution of the relative brightness along the vertical axis of each picture by the image analysis.

No significant differences in darkness can be found in two pictures at T = (407.880 and 407.890) K, and the meniscus can be hardly found in these pictures. The picture at T = 407.900 K is brighter than the picture at T = 407.890 K. As temperature increases, pictures at T = (407.910 and 407.920) K become brighter. Figure 1(b) shows distribution of relative brightness $(L_{\rm R})$ along the vertical axis (y) for the pictures shown in figure 1(a)by the present image analysis. The center in the picture shown in figure 1(a) is assigned the value of 0 in figure 1(b). The distribution of relative brightness at T = 407.870 K shows minimum at the center of the picture, which corresponds to the position of the meniscus shown in figure 1(a). The distribution of relative brightness at T = 407.880 K shows also minimum at the center of the picture, though it is not much difference in relative brightness from that at T = 407.890 K. Based on the distribution of relative brightness at 407.880 K, it is clear that there is a meniscus in the picture at this temperature. The distribution of relative brightness in the picture at T = 407.890 K shows maximum, and it is different from that at T = 407.880 K. The distributions of relative brightness at T = (407.900, 407.910, and 407.920) K show also maximum, and their values of relative brightness increase with temperature, which corresponds to the variation of darkness in the pictures shown in figure 1(a). It becomes easily concluded that the meniscus disappears between T = (407.880 and 407.890) K.

On the basis of these results, it may be concluded that the critical point of fluids and fluid mixtures can be determined by the present image analysis. The critical point can be determined as the state where the relative brightness (L_R) is the smallest and its distribution along the vertical axis (y) shows no position dependence. Critical pressure p_c can be determined to be the pressure measurement at the critical point of T_c and ρ_c . Download English Version:

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