

## Viscosity anomaly near the critical point in nitrobenzene + alkane binary systems

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The viscosity near the critical point in nitrobenzene+hexane and nitrobenzene+heptane binary systems was studied by examining the viscosity values for critical mixtures at a variable temperature as obtained with a falling-ball viscometer. The regular part of the viscosity of the critical mixtures was calculated by interpolating measurements made at noncritical concentrations. Because viscosity anomaly studies must be conducted at zero shear, a method allowing the estimation of the effective shear for this type of viscometer was developed with a view to introducing the corrections required. This methodology was used to determine the critical exponent for the viscosity anomaly in nitrobenzene+hexane and nitrobenzene+heptane systems, which were found to be  $0.0422 \pm 0.0004$  and  $0.0432 \pm 0.0013$ , respectively, very consistent with the accepted value: 0.043.

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### I. INTRODUCTION

Some physical properties of binary mixtures deviate from their regular behavior near a liquid-liquid critical point. These anomalies take place in both equilibrium properties such as heat capacity and density [1–3] and transport properties such as viscosity [4,5] or thermal conductivity [6] and originate from long-range fluctuations near the critical point. The dynamic viscosity  $\eta$  in the vicinity of the critical temperature for a mixture of critical composition exhibits a weak divergence given by the power law of the form [7,8],

$$\eta = \eta' (Q_0 \xi)^{z_\eta}, \quad (1)$$

where  $\eta'$  is the regular viscosity of the sample,  $Q_0$  is the system-specific critical amplitude of viscosity,  $\xi$  is the correlation length of local fluctuations, and  $z_\eta$  is the universal critical exponent, with theoretical value  $z_\eta = 0.068$  [9]. Equation (1) represents the asymptotic behavior of the viscosity near the critical temperature. Far away from  $T_C$ , the viscosity should be approach the regular viscosity  $\eta'$ .

In the critical region, the correlation length obeys the potential law,

$$\xi = \xi_0 t^{-\nu}, \quad (2)$$

$\xi_0$  being the amplitude of the correlation length,  $t$  the reduced temperature ( $t = |T - T_C|/T_C$ ), and  $\nu$  a universal critical exponent with theoretical value  $\nu = 0.630$ .

The determination of the behavior of the shear viscosity of a mixture of critical composition is based on the next equation, which is obtained by combining Eqs. (1) and (2):

$$\eta = \eta' (Q_0 \xi_0)^{z_\eta} t^{-\nu z_\eta} = \eta' C_C t^{-\phi} \quad (3)$$

with  $C_C = (Q_0 \xi_0)^{z_\eta}$  and  $\phi = \nu z_\eta$  the critical exponent for the viscosity anomaly.

The most accurate theoretical calculations have determined a value for this exponent of  $\phi = 0.043$  [9]. Experimentally,  $\phi$  has also been determined using different techniques [5,10,11], some of them based on direct measurements of the viscosity of the liquid [5,10]. The most accurate experimental values [11,12] for  $\phi$  are quite consistent with its theoretical value. Nevertheless, the estimation of a single value for

this exponent in both purely theoretical treatments and analysis of the experimental data is yet a controversial subject [7–9,13–18].

Viscosity measurements are usually made with capillary or oscillating body viscometers. In the analysis of the data, corrections have to be made for the possible dependence of the viscosity on the shear gradient and on the frequency of the experimental probe [8], which is particularly important as the critical point is approached. These viscometers allow a theoretical treatment to be applied in order to derive the expressions required for shear [8] and frequency corrections [10].

In this work, the critical behavior of the dynamic viscosity of nitrobenzene+hexane and nitrobenzene+heptane systems was examined as a function of temperature in order to determine the critical exponents and compare them with the accepted value. To this end, a falling-ball viscometer was used at atmospheric pressure to obtain experimental values and a theoretical-experimental treatment employed to calculate the effective shear for the data. The results thus obtained were corrected according to Oxtoby's theory [19,20]. The regular contribution to the viscosity of the sample of critical composition was determined by interpolating the measured values for samples of noncritical composition. This allowed the experimental critical exponent for the viscosity anomaly to be determined. This method was developed by using the 2-butoxyethanol+water system, which has been analyzed for a wide variety of properties [21–23] and studied with different types of viscometers [8,24].

### II. EXPERIMENT

#### A. Materials and procedure

2-butoxyethanol (>99.8% pure), hexane (>99.5% pure), heptane (>99.5% pure), and water (>99.9% pure) were obtained from Fluka, while nitrobenzene (>99% pure) was supplied by Aldrich. Their purity was checked by gas chromatography (GC). In order to avoid the presence of moisture traces, all chemicals were stored on molecular sieves from Sigma. Also, the liquids were degassed in an ultrasonic bath prior to use. Mixtures were prepared in a nitrogen atmo-

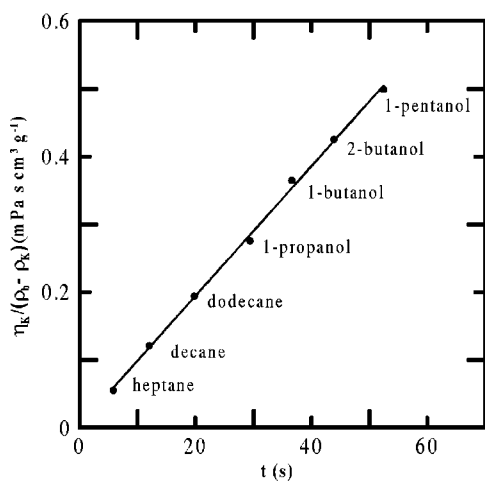


FIG. 1. Calibration of the viscometer at an angle of  $50^\circ$  and  $T = 298.15$  K.  $t$  is the rolling time,  $\rho$  the density,  $\eta$  the viscosity, and subscripts  $k$  and  $b$  refer to the unknown liquid and ball, respectively.

sphere, using a Mettler AE-240 balance accurate to within  $\pm 1 \times 10^{-5}$  g, and vigorously stirred prior to insertion into the measuring cells. Uncertainty in mole fraction is quoted in  $\pm 1 \times 10^{-4}$ . The mole fractions of 2-butoxyethanol+water, nitrobenzene+hexane, and nitrobenzene+heptane were 0.0598, 0.4275, and 0.4721, respectively. These values are consistent with previously reported critical values for these systems [8,23,25]. Because of the known influence of impurities in the mixture on the critical temperature [26],  $T_C$  was experimentally determined from turbidity measurements. Values of  $321.65 \pm 0.01$ ,  $293.55 \pm 0.01$ , and  $292.65 \pm 0.01$  K were thus obtained for the 2-butoxyethanol+water, nitrobenzene+hexane, and nitrobenzene+heptane systems, respectively; all are quite consistent with literature values [23–25,27–29].

### B. Viscosity measurements

Kinematic viscosity measurements were made with an Anton Paar AMVn falling-ball viscometer, where a gold-plated steel ball falls through a capillary tilted by a preset angle with respect to the horizontal. The measuring method was previously automated and allows a large number of data for the critical region to be obtained. The temperature was measured to within  $\pm 0.005$  K near the surface of the glass capillary by using a sensor accommodated in a thermostatic block that was controlled through the Peltier effect.

The viscosity values of the samples required the use of a nominal capillary and ball diameter of 1.6 and 1.5 mm, respectively. The measuring system was calibrated with the following liquids: heptane, decane, dodecane, 1-propanol, 1-butanol, 2-butanol, and 1-pentanol (see Fig. 1). Their density and viscosity were taken from literature [30]. The experimental dynamic viscosity was obtained from the following expression:

$$\eta = K_1 t_0 (\rho_b - \rho_k), \quad (4)$$

where  $\eta$  is the viscosity of the sample,  $K_1$  the calibration constant,  $t_0$  the time taken by the ball to fall,  $\rho_b$  the ball

TABLE I. Coefficients of the exponential fitting of the viscosity vs temperature [Eq. (5)] plots for the calculation of regular viscosity in samples of critical composition.

A (mPa s)	B (K)	Temperature range (K)
0.004 ± 0.002	2-butoxyethanol+water	
	1834 ± 100	313.15–323.15
0.031 ± 0.004	nitrobenzene+hexane	
	850 ± 40	303.15–288.15
0.0152 ± 0.0008	nitrobenzene+heptane	
	1141 ± 20	303.15–288.15

density, and  $\rho_k$  the density of the sample. The measurement temperature ranges are shown in Table I. For noncritical mixtures data were obtained at 1 K intervals. The measurement range for the critical samples was identical with that for the noncritical samples; however, the measurement interval varied from 1 K at the start to 0.1 K near the critical point to 0.01 K in its immediate vicinity.

Density measurements were made with an Anton Paar DSA-48 vibrating tube densimeter, the operation of which was also previously automated [1,31]. The density meter was calibrated with water [30] and *n*-octane [32], and the temperature measured to within  $\pm 0.005$  K using a platinum resistance thermometer. The density for the binary mixtures has been fitted against temperature to a third order polynomial form. The measurement ranges for each system were identical with those used with the viscometer, both for the pure products and for the mixtures of critical and noncritical composition. The measurement interval was 1 K throughout.

## III. RESULTS AND DISCUSSION

Viscosity measurements for a sample of critical composition near its critical temperature exhibit the expected slight anomaly as the critical point is approached. Figure 2 shows the experimental dynamic viscosity results near the critical point for the studied systems. Because the 2-butoxyethanol+water system [Fig. 2(a)] is of the lower critical solution temperature type, and the regular part of the viscosity is almost in all cases decreasing with temperature, its viscosity decreases as temperature becomes closer to the critical and, when the critical region is reached, first the viscosity virtually levels off and second it increases abnormally. By contrast, the nitrobenzene+alkane [Figs. 2(b) and 2(c)] systems are of the upper critical solution temperature type; thus their viscosity increases with decreasing temperature and rises abnormally near the critical point.

### A. Determination of the regular part

Based on Eq. (3), calculating  $\phi$  entails the prior determination of the regular contribution  $\eta'$  to the viscosity of the mixture of critical composition. This was obtained by interpolating the viscosity data for mixtures of noncritical composition using a polynomial fitting of viscosity against con-

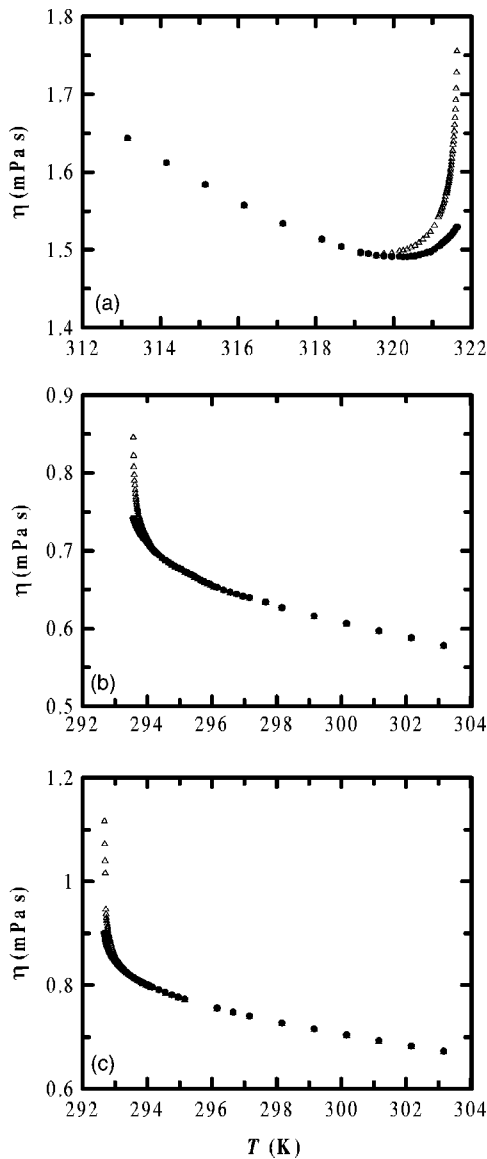


FIG. 2. Viscosity anomaly near the critical point for the sample of critical composition in the following systems: (a) 2-butoxyethanol+water, (b) nitrobenzene+hexane, and (c) nitrobenzene+heptane. (●) Experimental viscosity. (▲) Viscosity values obtained by using the zero-shear Oxtoby correction.

centration at each temperature [4]. These noncritical data are shown in Fig. 3.

The choice of the type of fitting to be used in order to estimate the variation of the regular viscosity with temperature in the sample of critical composition is irrelevant to the determination of the critical exponent [33]. Thus, both an exponential function and a polynomial are appropriate for this purpose, and both should provide an accurate estimate of the regular viscosity [34]. In this work, the temperature dependence of the regular viscosity  $\eta'$  was assumed to be of the form

$$\eta' = A \exp\{B/T\}, \quad (5)$$

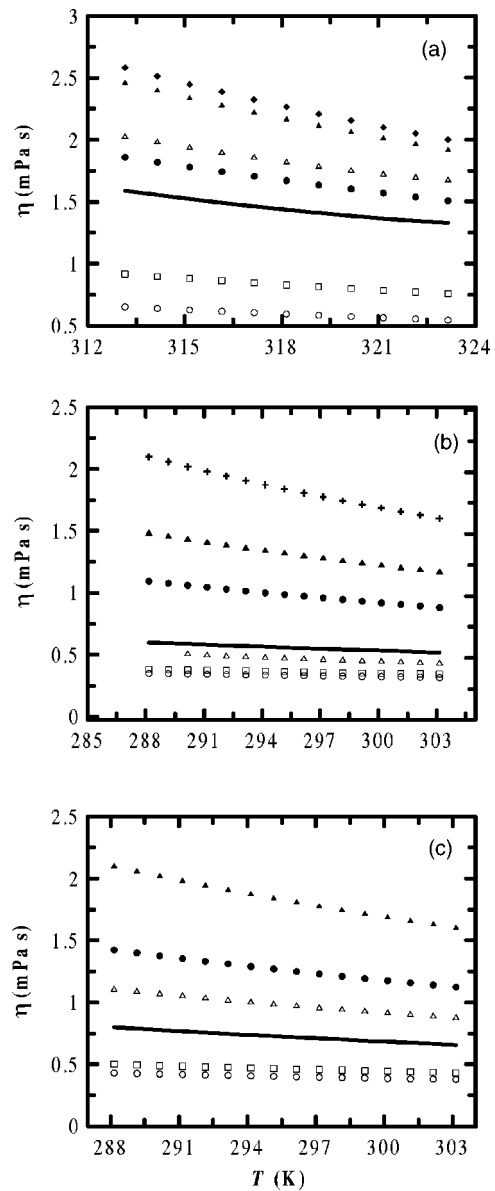


FIG. 3. Experimental viscosity values for the following systems: (a) 2-butoxyethanol+water, for noncritical mole fractions  $x = 0$  (○); 0.0199 (□); 0.0998 (△); 0.1989 (▲); 0.3460 (◆); 1 (●) and estimation of the regular viscosity for the critical mole fraction (solid line),  $x_C = 0.0598$ ; (b) nitrobenzene+hexane, for noncritical mole fractions  $x = 0$  (○); 0.1013 (□); 0.2500 (△); 0.7104 (●); 0.8457 (▲); 1 (+), and estimation of the regular viscosity for the critical mole fraction (solid line)  $x_C = 0.4275$ ; (c) nitrobenzene+heptane, for noncritical mole fractions  $x = 0$  (○); 0.1527 (□); 0.6507 (△); 0.8005 (●); 1 (▲) and estimation of the regular viscosity for the critical mole fraction (solid line)  $x_C = 0.4721$ .

where  $A$  and  $B$  are fitting parameters, and  $T$  is the temperature (in Kelvin). This equation was developed by Arrhenius [35] and usually provides an accurate prediction of the variation of the regular viscosity with the temperature [5]. The values of the fitting parameters are shown in Table I and the interpolated regular viscosity of the sample of critical composition for each system in Fig. 3.

### B. Correction for the influence of shear

Near the critical point the shear rate produces two effects: a decrease of the shear viscosity and a delay in the phase transition. Thus, corrections in experimental viscosity values are needed [4–6,8]. A variety of corrective procedures based on extrapolation to zero shear have been reported. Chhabra *et al.* [36] reviewed the techniques involved, emphasized the lack of accuracy in the results they provide, and noted the errors made. In fact, these methods, some empirical and some theoretical, overestimate the corrected viscosity in the shear region.

At present, viscosity corrections are made according to Oxtoby's theory [19,20]. The correction to zero shear of the viscosity of a mixture of critical composition is given by

$$\eta(0) = \frac{\eta(S_{\text{eff}})}{1 - \Delta(\lambda)}, \quad (6)$$

where

$$\lambda = \frac{\eta_S^{\xi^3} S_{\text{eff}}}{k_B T}, \quad (7)$$

and

$$\Delta(\lambda) = \begin{cases} 0, & \lambda < 0.1 \\ 0.0214 + 0.0266 \log_{10} \lambda + 0.0078(\log_{10} \lambda)^2, & 0.1 < \lambda < 20, \\ \frac{8}{45\pi^2} \ln\left(\frac{\lambda}{0.45}\right), & \lambda > 20, \end{cases} \quad (8)$$

$k_B$  being the Boltzmann constant,  $T$  the temperature at which the viscosity is measured,  $\xi$  the correlation length, and  $S_{\text{eff}}$  the effective shear, and must be estimated for any method.

### C. Determination of the effective shear for a ball viscometer

Ball viscometers exhibit highly complex fluid dynamics; this has so far precluded the theoretical estimation of the effective shear involved in the measurements they provide. In this work, the problem was circumvented by experimentally determining the effective shear for a ball viscometer from the balance of the forces that cause the ball to move through the liquid. Newton's viscosity law relates the viscous force  $F$  per unit area  $A$  to the shear  $S = du/dy$  via a proportionality factor, viz., the viscosity ( $\eta$ ):

$$\frac{F}{A} = \eta \left( \frac{du}{dy} \right) = \eta S, \quad (9)$$

Therefore, the viscous force can be modeled as

$$F = \eta S_{\text{eff}} A_{\text{eff}} \quad (10)$$

where  $S_{\text{eff}}$  is the effective shear and  $A_{\text{eff}}$  the effective area.

The forces causing the ball to move include gravity ( $F_g$ ), which facilitates its displacement, and buoyancy ( $F_b$ ) and viscous force ( $F$ ), which hinder it. While the ball falls at a constant velocity, the net force on it is zero, i.e.,

$$F = F_g - F_b, \quad (11)$$

Substitution of  $F_g$  and  $F_b$  by their expressions as a function of the sphere radius ( $r$ ) the capillary tilting angle ( $\theta$ ), and the ball ( $\rho_b$ ) and fluid ( $\rho_k$ ) densities, and using Eq. (10), we obtain

$$\pi \frac{r^3}{48} g \sin \theta (\rho_b - \rho_k) = \eta S_{\text{eff}} A_{\text{eff}}. \quad (12)$$

Based on Eq. (12), obtaining  $S_{\text{eff}}$  entails the prior calculation of the effective area  $A_{\text{eff}}$  on which the viscous force acts. Such an area was determined here for the 2-butoxyethanol + water system, which is widely documented and possesses an amplitude of correlation length univocally determined using light scattering methods [8,23].

The first step involved parametrizing the effective area via a proportionality factor  $k$  (i.e.,  $A_{\text{eff}} = k \pi r^2$ ). Then,  $k$  was given different numerical values to obtain  $S_{\text{eff}}$  from Eq. (12). Once the  $S_{\text{eff}}$  values at each  $k$  value were known, the experimental non-zero-shear viscosity values near the critical point were corrected as described in Sec. III B. When the corrected viscosity value  $\eta$  at each  $k$  value was known, it was substituted together with the regular viscosity  $\eta'$  determined as described in Sec. III A, into Eq. (3) to obtain

$$\log_{10} \left( \frac{\eta}{\eta'} \right) = \log_{10} C_C - \phi \log_{10} t, \quad (13)$$

TABLE II. Critical exponent  $\phi$  obtained by using different  $k$  values with the Oxtoby correction.

System	$k$	$\phi_{\text{expt}}$
2-butoxyethanol+water	0.10	0.0447±0.0008
	0.15	0.0431±0.0006
	0.20	0.0418±0.0006
	0.25	0.0409±0.0006
nitrobenzene+hexane	0.15	0.0422±0.0004
nitrobenzene+heptane	0.15	0.0432±0.0013

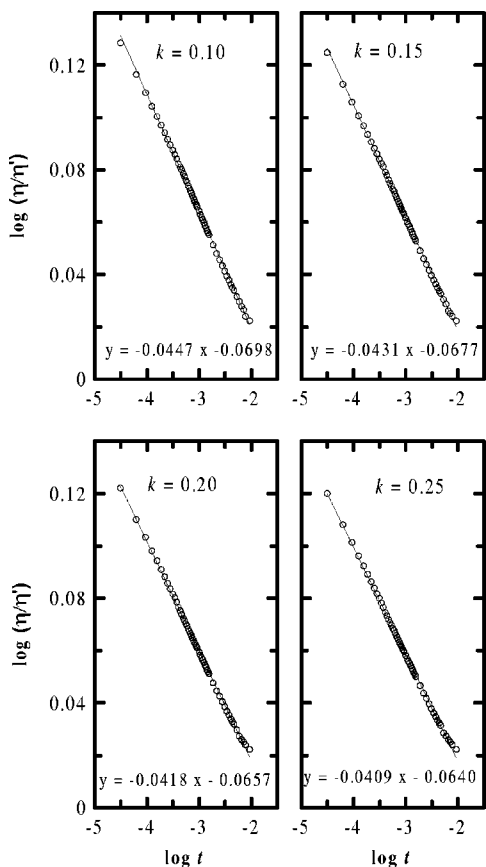


FIG. 4. Log-log plot of  $(\eta/\eta')$  against the reduced temperature at different  $k$  values for the sample of critical composition in the 2-butoxyethanol+water system.  $\eta$  values were subjected to the Oxtoby correction  $\eta' = A \exp\{B/T\}$ ;  $t = |T - T_C|/T_C$ ;  $T_C = 321.65$  K. Linear fitting of the corrected slope data (solid line)  $\phi$  to Eq. (13). The logarithms are to base 10.

In Table II the influence of the  $k$  values in the exponent  $\phi$  is shown. The linear fittings used to determine the critical exponent are shown in Fig. 4.  $k = 0.15$  was the value resulting in the best fit of the experimental critical exponent to the

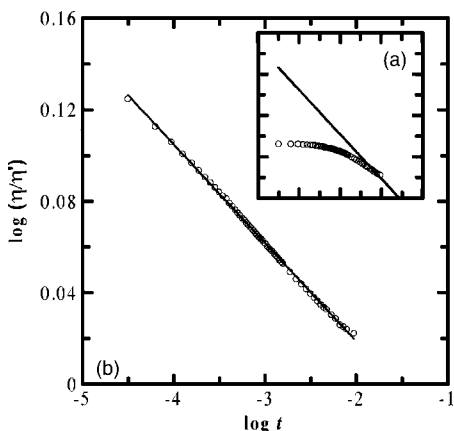


FIG. 5. Plot of  $\log_{10}(\eta/\eta')$  vs  $\log_{10} t$  (logarithmic decay) for the 2-butoxyethanol+water system for the critical mixture: (a) experimental data; (b) corrected data. Linear fitting of the corrected data (solid line). The slope of the solid line is  $\phi = 0.0431$ .

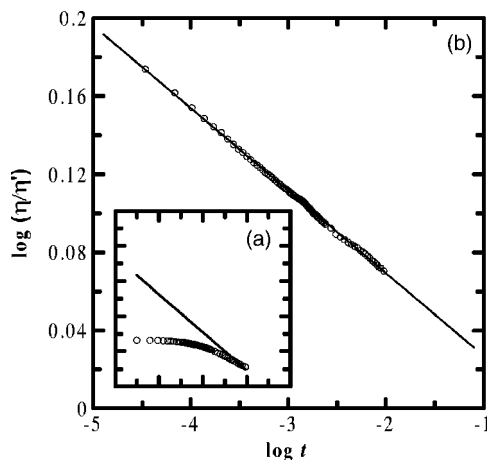


FIG. 6. Plot of  $\log_{10}(\eta/\eta')$  vs  $\log_{10} t$  (logarithmic decay) for the nitrobenzene+hexane system for the critical mixture: (a) experimental data; (b) corrected data. Linear fitting of the corrected data (solid line). The slope of the solid line is  $\phi = 0.0422$ .

accepted one ( $\phi = 0.043$ ). The uncertainty in  $k$  was  $\pm 0.02$ . Therefore, the effective shear for a falling-ball viscometer of the above-described geometry can be calculated from the following expression:

$$S_{\text{eff}} = \frac{\pi(r^3/48)g \sin \theta(\rho_b - \rho_k)}{\eta 0.15 \pi r^2}, \quad (14)$$

Figure 2(a) shows the corrected viscosity values. Figure 5, which is a plot of  $\log_{10}(\eta/\eta')$  against  $\log_{10} t$ , illustrates the influence of shear on the experimental viscosity at near-critical temperatures.

#### D. Determination of the critical exponent for nitrobenzene+alkane systems

The above-described procedure was used to obtain the exponent  $\phi$  for the nitrobenzene+hexane and nitrobenzene

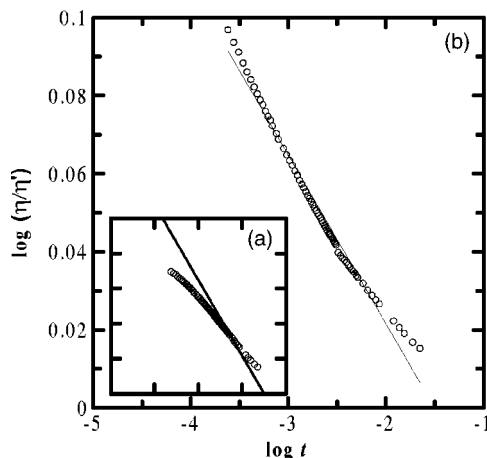


FIG. 7. Plot of  $\log_{10}(\eta/\eta')$  vs  $\log_{10} t$  (logarithmic decay) for the nitrobenzene+heptane system for the critical mixture: (a) experimental data; (b) corrected data. Linear fitting of the corrected data (solid line). The slope of the solid line is  $\phi = 0.0432$ .

+heptane systems. Once the regular viscosity  $\eta'$  was calculated, the experimental data for the samples of critical composition at a near-critical temperature were corrected using Eqs. (6)–(8) and (14). The amplitudes of the correlation length  $\xi_0$ , needed to calculate the corrected viscosities, were taken from literature, their values for the nitrobenzene+hexane and nitrobenzene+heptane systems being 0.265 [28,37,38] and 0.28 nm [39], respectively. Figures 2(b) and 2(c) illustrate the differences between the experimental and corrected viscosity values. Finally, the corrected values were fitted to Eq. (13), in order to obtain  $\phi$ . These fits are shown in Figs. 6 and 7. The experimental values of the critical exponent  $\phi$  for studied systems are shown in Table II. The obtained values for exponent  $\phi$  are in good agreement with the accepted value ( $\phi=0.043$ ).

#### IV. CONCLUSIONS

The viscosity anomaly near the liquid-liquid critical point of binary mixtures was studied by using a falling-ball vis-

cometer. This required estimating the effective shear for the viscometer. The procedure used allows viscosity corrections to be accurately estimated irrespective of the specific type of system involved. The proposed methodology was developed with the widely documented system 2-butoxyethanol+water.

The results for nitrobenzene+alkane systems reveal good consistency between the obtained critical exponent and the accepted value. This confirms the accuracy of the proposed methodology for determining the experimental critical exponent that characterizes the viscosity anomaly.

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