

Critical behavior of the ultrasonic attenuation and velocity and shear viscosity for the binary mixture of nitrobenzene-*n*-hexane

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Ultrasonic velocity and absorption as a function of temperature, concentration, and frequency (5–25 MHz) and shear viscosity as a function of concentration and temperature are reported for the binary mixture nitrobenzene-*n*-hexane in the homogeneous phase above T_c . For the observed absorption at critical concentration and critical temperature α_c/f^2 vs $f^{-1.06}$ yields a straight line as predicted by the dynamic scaling theory of Ferrell and Bhattacharjee [Phys. Rev. A **24**, 1643 (1981)]. Also, the critical amplitudes of the thermal expansion and specific heat have been calculated using the two-scale factor universality relation. The adiabatic coupling constant g is calculated and compared to the experimental value. In addition, the experimental values of α/α_c (where α is the absorption at critical concentration above the critical temperature) for nitrobenzene-*n*-hexane are compared to the scaling function $F(\omega^*)$ and show a good agreement with the theory. Finally, the velocity for the system at the critical concentration above the critical temperature appears to decrease linearly with increasing temperature.

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INTRODUCTION

In this research, we have examined sound propagation in a nitrobenzene ($C_6H_5NO_2$) and *n*-hexane (C_6H_{14}) mixture by measuring its absorption and velocity. The shear viscosity was also measured. Several papers reporting ultrasonic measurements of this system^{1–5} cover a wide range of frequencies, but most of them are limited to the critical temperature. However, the data given by Ref. 1 can be used to calculate α/α_c for only two frequencies. In order to apply the dynamic scaling theory for the study of this system, further absorption coefficient and velocity are measured for an extended frequency range of 5–25 MHz and the temperature range of T_c to 50 °C. Also, shear viscosity measurements are made in the same temperature range. The experimental results are compared with the predictions of the dynamic scaling theory of Ferrell and Bhattacharjee.⁶ Since the necessary collateral data exist for this system, the experimental results can be compared to the theoretical predictions. According to the thermodynamic theory by Ferrell and Bhattacharjee (FB), α_c/f^2 (at critical temperature and concentration) vs $f^{-1.06}$ should yield a straight line. The slope of this line can be calculated from the theory using the collateral data. In addition, the experimental values of α/α_c at the critical mixture are compared to the theoretical scaling function $F(\omega^*)$. The adiabatic coupling constant g is calculated and compared to the experimental value. Further, the critical amplitudes of the thermal expansion and specific heat of the critical mixture are calculated using the two-scale-factor universality relationship.⁷ Finally, the background part of the thermal expansion coefficient is calculated using the linear density relation when the density is plotted against the temperature.⁸

I. THEORETICAL CONSIDERATION

The observed attenuation α_{obs}/f^2 near the critical temperature can be expressed in two terms:⁹

$$\frac{\alpha_{\text{obs}}}{f^2} = \frac{\alpha_{\text{crit}}}{f^2} + \frac{\alpha_{\text{back}}}{f^2}. \quad (1)$$

Here, α_{back} is the background term that includes absorption due to the Navier–Stokes term α_{NS} , and the radiation α_{rad} ,

$$\alpha_{\text{back}} = \alpha_{\text{NS}} + \alpha_{\text{rad}}, \quad (2)$$

where α_{NS} can be written as

$$\alpha_{\text{NS}} = \left(\frac{\omega^2}{2\rho u^3} \right) \left(\frac{4\eta}{3} + \eta_v + \frac{\dot{A}(\gamma - 1)}{C_p} \right). \quad (3)$$

Here, ρ is the mass density, u is the velocity, η is the shear viscosity, η_v is the bulk viscosity, \dot{A} is the thermal conductivity, and γ is the ratio of the heat capacities. One can neglect the contribution due to heat conduction, and radiation since $\alpha_{\text{cond}}/\alpha_{\text{visc}}$ is typically less than 10^{-3} for a nonmetallic liquid,^{9,10} $\alpha_{\text{rad}}/\alpha_{\text{cond}} \approx 10^{-2}$. In the dynamic scaling theory as well as other thermodynamic theories, the critical attenuation can be expressed as a function of the reduced frequency, $\omega^* = \omega/\omega_D$, where ω_D is a characteristic temperature-dependent relaxation rate that can be calculated in either of two ways. The first way is to use the relationship $\omega_D = 2D\xi^{-2}$, where D is the mutual mass diffusion coefficient and ξ is the correlation length.⁹ The asymptotic behavior of the diffusion coefficient can be given by the Stokes–Einstein expression

$$\omega_D = k_B T / (3\pi\eta\xi^3) = [k_B T_c / (3\pi\eta_0\xi_0^3)] t^{(3+X_\eta)\nu}, \quad (4)$$

where the correlation length and the shear viscosity are given

en by $\xi = \xi_0 t^{-\nu}$ and $\eta = \eta_0 t^{-X_\eta \nu}$, k_B is the Boltzmann's constant, and where $t = (T - T_c)/T_c$ is the reduced temperature.¹¹ This can be written simply as $\omega_D = \omega_0 t^{z\nu}$, where $z\nu = (3 + X_\eta)\nu = 1.93$. Note z , ν , and X_η are critical exponents. The characteristic relaxation rate can also be expressed as a function of the decay rate ξ of the critical concentration fluctuation and is given by

$$\omega_D = \lim_{q\xi \rightarrow 0} 2\xi / (q\xi)^2 t^{z\nu}, \quad (5)$$

where ξ is obtained from the autocorrelation function of the scattered light intensity with the corresponding wavenumber q (Ref. 12). The FB theory uses the specific volume V as a function of P and ΔT where $\Delta T = T - T_c(P)$. The critical attenuation coefficient in FB theory can be written as^{7,9}

$$\frac{\alpha_c}{f^2} = \left(\frac{\pi^2 \alpha C_{pc}}{2z\nu} \right) \left(\frac{u_c g^2}{T_c [C_p(t_f)]^2} \right) \times \left(\frac{a\omega_0}{2\pi} \right)^{\alpha/z\nu} f^{-(1 + \alpha/z\nu)}. \quad (6)$$

Here, α and C_{pc} are the critical exponent and the critical amplitude describing the leading singularity in C_p (heat capacity per gram), $C_p = C_{pc} t^{-\alpha} + C_{pb}$. Note C_{pb} is the background specific heat, $a = (\omega/\omega_0) t_f^{-z\nu}$ is a dimensionless scale factor of order unity,¹³ u_c is the adiabatic sound velocity at T_c and the experimental frequency, g is the adiabatic coupling constant, and $C_p(t_f)$ is the specific heat at a characteristic reduced temperature t_f , which can be well approximated by the t value at which $\alpha_{\text{crit.conc.}}$ for a given frequency is one-half its value at T_c (Ref. 9). Writing the total attenuation terms of α_c/f^2 yields

$$\frac{\alpha_c}{f^2} = \left[\frac{\pi^2 \alpha C_{pc}}{2z\nu} \frac{u_c g^2}{T_c [C_p(t_f)]^2} \left(\frac{\omega t_f^{z\nu}}{2\pi} \right)^{\alpha/z\nu} \right] \times f^{-1.06} + \frac{\alpha_{\text{back}}}{f^2}. \quad (7)$$

This can be expressed as

$$\alpha_c/f^2 = S f^{-1.06} + b, \quad (8)$$

where b is mainly the absorption due to viscosity which represents the frequency-independent background term of α_{obs}/f^2 at T_c that would be present in the absence of critical fluctuation. Here, S is the term in parentheses in Eq. (7). The prediction for the variation in $\alpha_{\text{crit.conc.}}$ as a function of the reduced frequency ω^* is given in the form

$$\alpha/\alpha_c = \alpha_{\text{crit},\omega,T} / \alpha_{\text{crit},\omega,T_c} = F(\omega^*). \quad (9)$$

Here, $\alpha_{\text{crit},\omega,T}$ is the critical term at temperature T and critical concentration and $\alpha_{\text{crit},\omega,T_c}$ is the critical term at the critical temperature T_c and critical concentration. There are two distinct aspects to this prediction. First, the critical attenuation ratio α/α_c should scale with respect to ω^* ; and, second, the form of the scaling function should be given by⁶

$$F(\omega^*) = (1 + \omega^{*-1/2})^{-2}. \quad (10)$$

The adiabatic coupling constant g was introduced by FB theory of the ultrasonic attenuation near the critical point. The adiabatic coupling constant is given by^{7,14}

$$g = \rho_c T_c S'_c. \quad (11)$$

Here, $S'_c = dS_c/dP$, S_c is the entropy per unit mass for the critical concentration at T_c and ρ_c is the critical density. Using the relationship

$$\left(\frac{\partial \Delta T}{\partial P} \right)_s = \left(\frac{\partial T}{\partial P} \right)_s - \frac{dT_c}{dP} = \frac{T_c S'_c}{C_p}, \quad (12)$$

g can also be written as $-g/(\rho_c C_p) = (\partial \Delta T / \partial P)_s$, or

$$g = \rho_c C_p \left[\frac{dT_c}{dP} - \left(\frac{\partial T}{\partial P} \right)_s \right]. \quad (13)$$

Using several other thermodynamic relationships, g can be expressed in a form more suitable for calculation:

$$g = \rho_c C_p \left(\frac{dT_c}{dP} - \frac{T \alpha_p}{\rho C_p} \right) \quad (14)$$

or

$$g \approx (C_{pb} \alpha_{pc} T_c / C_{pc}) - \alpha_{pb} T. \quad (15)$$

Note α_p is the isobaric thermal expansion coefficient, and α_{pc} and α_{pb} are the critical and background parts of the thermal expansion coefficient. Here, g can be positive or negative for any of the given expressions. For a system with an upper critical solution point, a positive value for g implies that spinoidal decomposition (phase separation) can be induced by a sudden pressure increase, a negative value of g means that spinoidal decomposition is induced by a sudden pressure decrease.

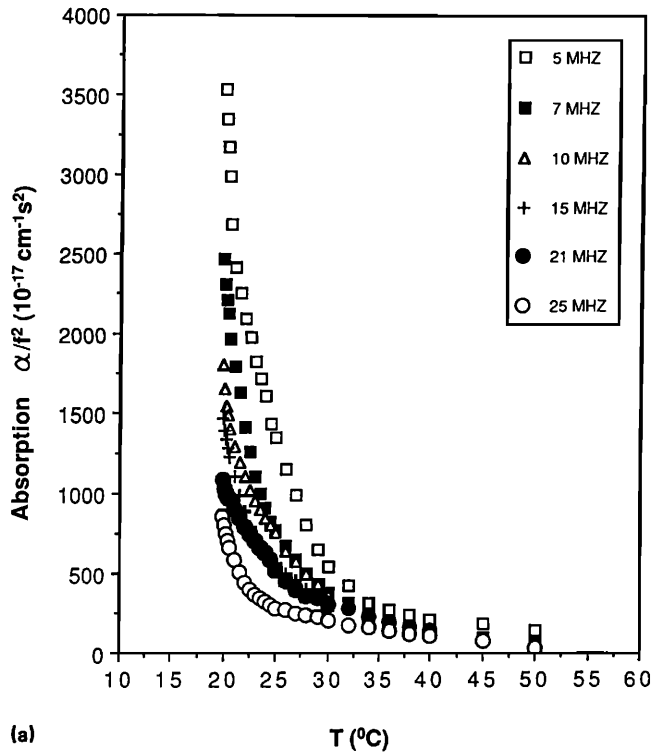
II. EXPERIMENTAL TECHNIQUE

Measurements of absorption and velocity were made using the Matec pulse-echo technique. The following Matec equipment was used: a radio frequency gated amplifier model 515, gating modulator model 5100, a doubled-tuned preamplifier model 252, a broadband receiver model 605, and the model 666 pulse comparator that generates a pulse of radio frequency which can be controlled in steps of 0.1 dB in amplitude up to a total of 61 dB. The shear viscosity was measured by using the Brookfield digital viscometer. The sample was prepared by weight with a precision of ± 0.05 mg. The nitrobenzene and *n*-hexane were of reagent quality and thus met ACS specifications. The chemicals were used without any further purification. To measure the absorption and velocity, a sample was prepared and heated until its temperature was at least 5° above the critical temperature and stirred several times to insure complete mixing. The test cell has a quartz crystal transmitter at one end and a reflector that is moved by a precision micrometer.¹⁵ The test cell was also heated above the critical temperature before the sample was added. For the sound absorption coefficient, the pulse comparator was used to measure the height of the echoes viewed on the oscilloscope. For a given number of interference peaks, the displacement of the reflector, which leads to the wavelength, is measured and used with the frequency counter to determine the velocity in the liquid.

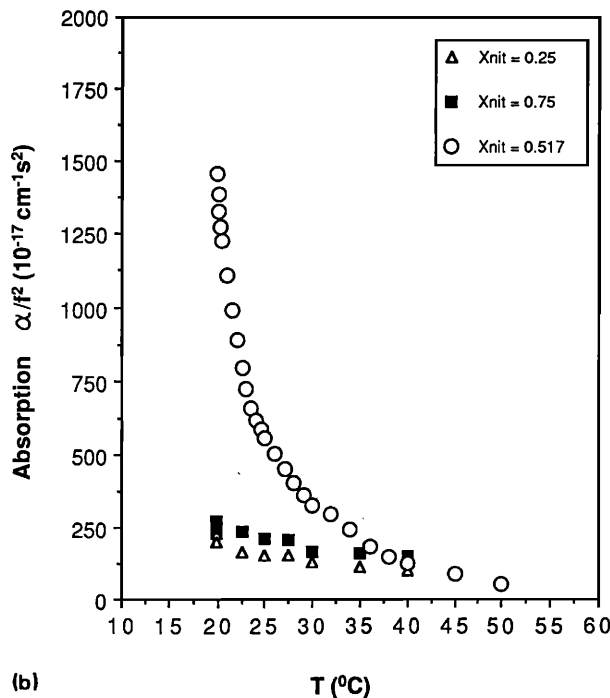
III. RESULTS AND ANALYSIS

The system of nitrobenzene and *n*-hexane has an upper critical temperature of 19.89 °C and a critical composition

(X_c) of 51.7% by weight of nitrobenzene¹ (0.428 mole fraction nitrobenzene). The ultrasonic absorption was measured for frequencies 5, 7, 10, 15, 21, and 25 MHz. The temperature of the sample was controlled within ± 0.01 °C. Figure 1(a) shows the temperature dependence of the absorption α/f^2 for the critical binary mixture of nitrobenzene and *n*-



(a)



(b)

FIG. 1. (a) Temperature dependence of the absorption α/f^2 for the critical binary mixture of nitrobenzene and *n*-hexane. (b) The absorption coefficient at $f = 15$ MHz at three different concentrations is plotted versus temperature. Note: X_{nit} is the concentration by weight of nitrobenzene.

hexane at six different frequencies. The error in the absorption measurements was less than 5%. It is observed that α_c/f^2 decreases with temperature above T_c at all frequencies studied. Figure 1(b) shows the absorption coefficient at a given frequency (15 MHz) and three different concentrations plotted versus temperature. For the noncritical mixtures of nitrobenzene and *n*-hexane some critical behavior in the absorption is still evident. The absorption coefficient at the phase separation temperature has dropped considerably when compared to the absorption coefficient of the critical concentration at the critical temperature. From Fig. 1(b), it would appear that the background absorption is approximately the same for all the concentrations. In Fig. 2, our absorption data at T_c are shown with the data of Manucharov² as a function of frequency. Manucharov has measured the ultrasonic absorption in a critical nitrobenzene and *n*-hexane solution (0.4 mole fraction nitrobenzene or 48.8% by weight of nitrobenzene). His critical separation temperature is $T_c = 20.2$ °C, and the frequency range is from 0.05–3 MHz. The velocity versus temperature at different concentrations and at $f = 15$ MHz is shown in Fig. 3. The error in the velocity measurements was less than 0.3%. A least-squares fit of the 5-MHz data at critical concentration yields a slope of -3.59 m/(s deg) and $u_c = 1209.2$ m/s. To avoid cluttering the figures excessively, the data for the other frequencies are not shown. For the 15-MHz data the slope is -3.61 m/(s deg) and $u_c = 1207.1$ m/s. Sette has measured the sound velocity in the nitrobenzene and *n*-hexane mixture.¹⁶ He took $T_c = 19.597$ °C and $X_c = 0.428$ molar fraction of nitrobenzene. Our data show a good agreement with their data. For example, at $T = 19.972$ °C and $f = 15$ MHz

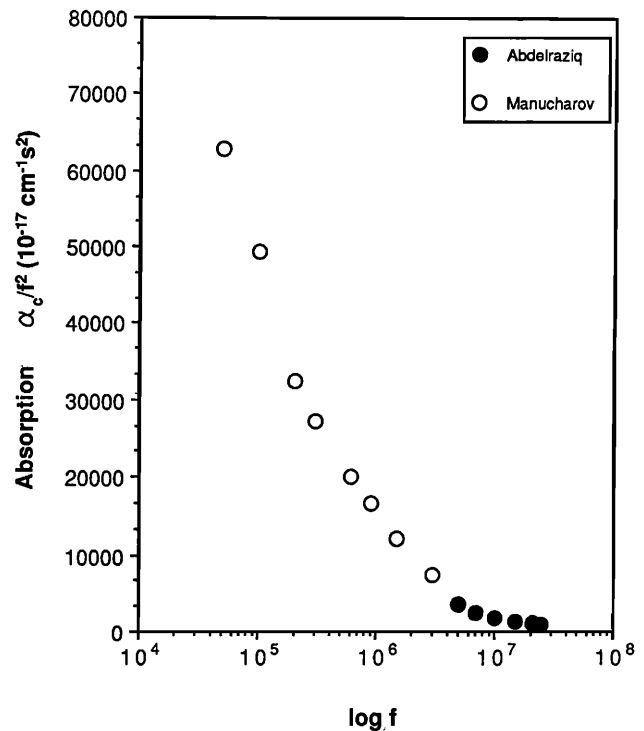


FIG. 2. Our absorption data at T_c with Manucharov's² data as function of frequency (f).

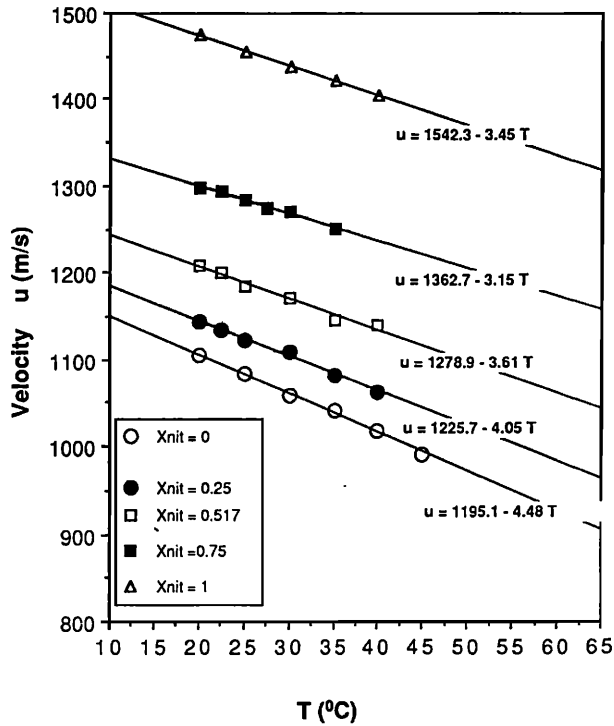


FIG. 3. The velocity versus temperature at different concentrations and at $f = 15$ MHz.

their measurement was $u = 1210.75$ m/s, and our measurement at $T = 19.95$ °C and the same frequency was 1209 m/s. In contrast to what is observed near the critical point of simple fluids, no anomaly is observed. The sound velocity, as a function of temperature at a critical concentration, can be expressed by $u = 1209.2 - 3.59(T - T_c)$ for 5 MHz and $u = 1207.1 - 3.61(T - T_c)$ in m/s for 15 MHz.

In Fig. 4, our experimental values of α/α_c with 2.56-MHz data for Tanaka and Wada¹ are shown along with $F(\omega^*)$. Our data were fit to the theoretical curve using a value of ω_0 of 1.75×10^{10} Hz which was calculated by using our shear viscosity measurements and Chen's measurements of 3.54 ± 0.15 Å for the correlation length.¹⁷ Taking our shear viscosity data and fitting to $\eta = \eta_0 t^{-0.0378}$, one obtains $\eta_0 = 0.55$ cp. The shear viscosity data for our system were also described in Ref. 17 with the expression,

$$\eta(T) = \eta'_0 \exp[E/(1+t)] t^{-y}, \quad (16)$$

where $\eta_0 = \eta'_0 \exp[E/(1+t)]$, $\eta'_0 = 1.43 \times 10^{-2}$ cp, $E = 3.58 \pm 0.04$, and $y = 0.0043 \pm 0.0003$. Using this formula one obtains $\eta_0 = 0.53$ cp. The light-scattering measurements of Chen and Tartaglia¹⁷ give a value of 3.54 ± 0.15 Å for the correlation length. Taking $T_c = 293.05$ K, a value of ω_0 can be calculated; the result is $\omega_0 = 1.8 \times 10^{10}$ Hz, where, as Sette¹⁶ has obtained, $\omega_0 = 8.01 \times 10^{10}$ Hz, which is calculated by using the quantities $D_0 = 1.65 \times 10^{-5}$ cm² s⁻¹ and $\xi_0 = 2.03$ Å, where $\omega_0 = 2D_0/\xi_0^2$. These two values differ by a factor of about 4. On the other hand our calculated value of $\omega_0 = 1.8 \times 10^{10}$ Hz is in good agreement with the value obtained by Tanaka¹ and Chen¹⁸ ($\omega_0 = 2 \times 10^{10}$ Hz).

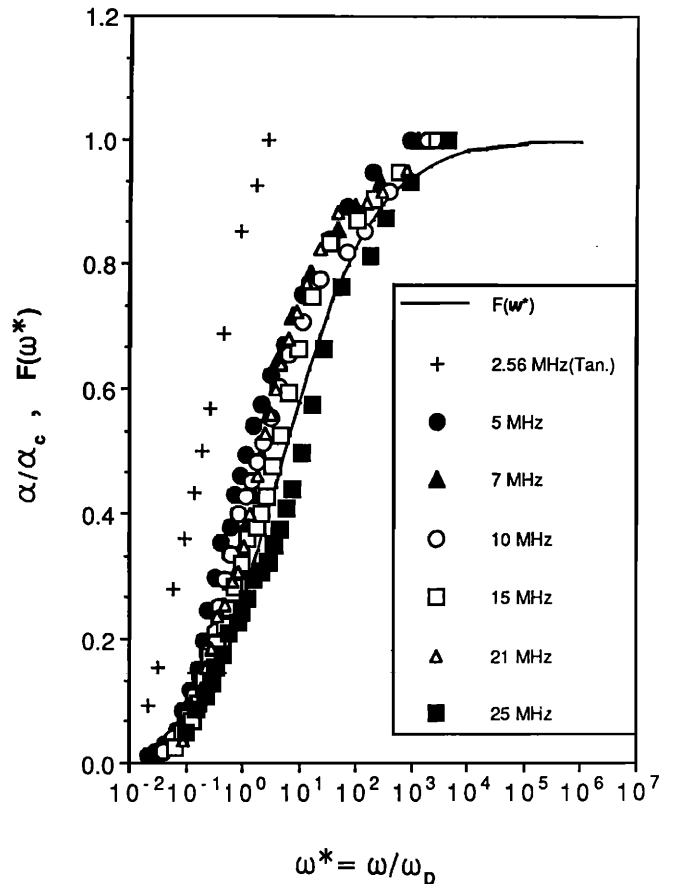


FIG. 4. A plot of α/α_c as a function of ω^* for our experimental data and the 2.56-MHz data of Tanaka and Wada¹ is shown along with the scaling function $F(\omega^*)$.

Figure 5 shows a plot of absorption α_c/f^2 at critical mixture and temperature T_c vs $f^{-1.06}$. A least-squares fit yields an experimental slope (S) of 3.94×10^{-7} cm⁻¹ s^{0.94} and an intercept of 362.3×10^{-17} cm⁻¹ s². The calculated value of S , using Eq. (9) and the calculated value of $g = -0.56$, is 2.6×10^{-7} cm⁻¹ s^{0.94}.

The density data for the critical mixture of the nitrobenzene and *n*-hexane as a function of temperature has been reported by Miyake and Izumi,⁸ and the value of the density was determined by the relation, $\rho = 0.8580 - 0.743 \times 10^{-3} T$ (°C). From their density data, we have made estimate of the slope, $(\partial \rho^{-1}/\partial T)_p$, from linear fit of ρ^{-1} at various temperatures. Using the thermal expansion coefficient $\alpha_p \equiv \rho(\partial \rho^{-1}/\partial T)_p$ and the asymptotic behavior of α_p and C_p , which can be represented by power laws of the form

$$\alpha_p = \alpha_{pc} t^{-\alpha} + \alpha_{pb}, \quad (17)$$

$$C_p = C_{pc} t^{-\alpha} + C_{pb}, \quad (18)$$

one can find the background part of the thermal expansion, where α_{pc} and C_{pc} are the critical amplitudes of thermal expansion and specific heat. Note α_{pb} and C_{pb} are the background amplitudes. Since the critical amplitudes α_{pc} and C_{pc} can be calculated from the known asymptotic behavior of the correlation length through the relationship of two-scale-factor universality.⁷

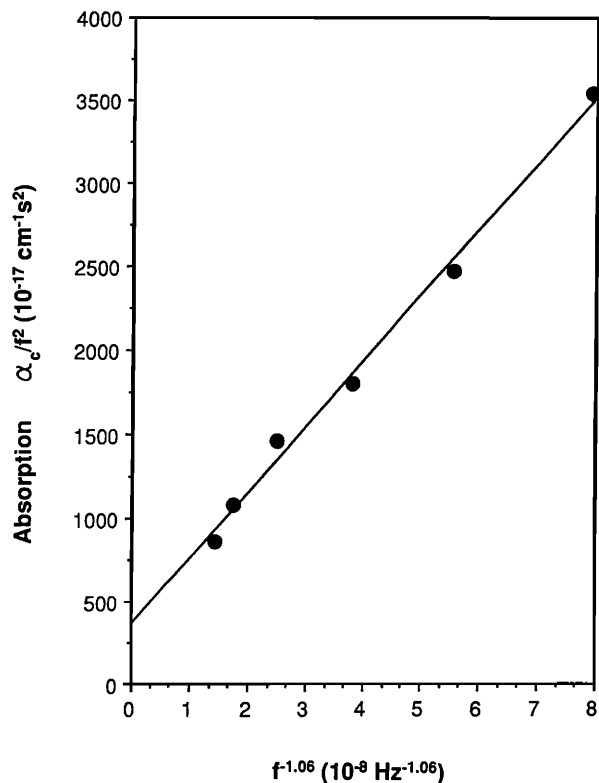


FIG. 5. The absorption α_c/f^2 at T_c and critical mixture versus $f^{-1.06}$.

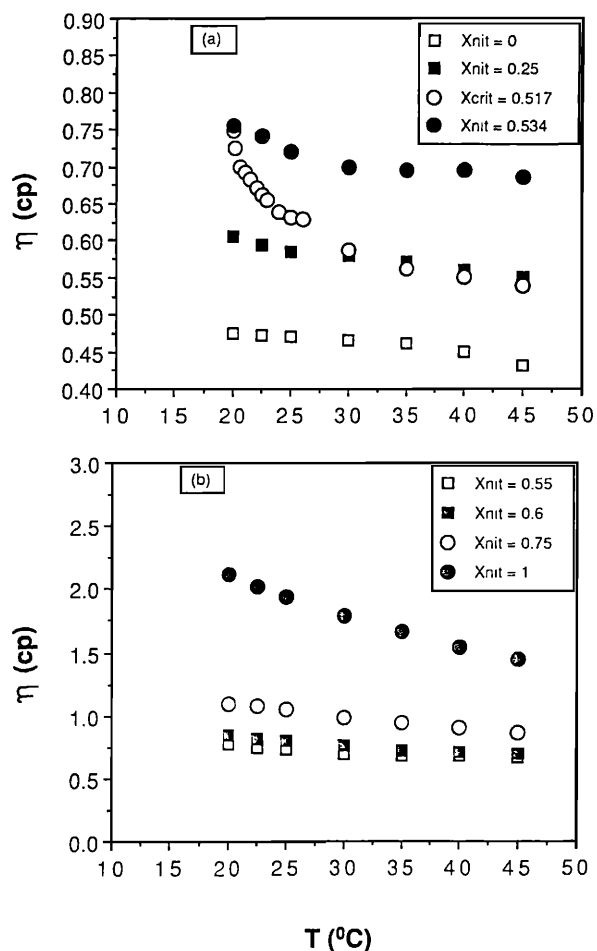


FIG. 6. (a) and (b) Temperature dependence of shear viscosity η above T_c at different concentrations and the critical concentration $X_c = 0.517$ by weight of nitrobenzene.

$$\xi_0 [\alpha_p \rho_c C_{pc} / k_B]^{1/3} = \xi_0 [\alpha_p T_c \alpha_{pc} / (k_B T'_c)]^{1/3} = 0.270.$$

Here, k_B designates Boltzmann's constant and ξ_0 is the amplitude in the power law $\xi = \xi_0 t^{-\alpha}$ for the correlation length ξ . From the relationship of two-scale-factor universality and the value of α_p as deduced from the density as a function of ΔT , the values of the thermal expansion and specific heat are represented by

$$\alpha_p = 0.312 \times 10^{-4} t^{-\alpha} + 8.5 \times 10^{-4} \text{ K}^{-1},$$

$$C_p = 0.066 \times 10^7 t^{-\alpha} + 1.9 \times 10^7 \text{ erg/(g K)}.$$

Having determined the asymptotic behavior of the thermal expansion coefficient and the specific heat, the coefficient $(\partial T / \partial P)_s$ at temperature T can be calculated using the form $(\partial T / \partial P)_s = T \alpha_p / (\rho C_p)$. For example, at $T = 25^\circ\text{C}$, $(\partial T / \partial P)_s = 1.59 \times 10^{-8} \text{ K cm}^2/\text{dyn}$. The coefficient dT_c/dp can also be calculated; $T'_c = dT_c/dp = T_c \alpha_{pc} / (\rho_c C_{pc}) = -1.64 \times 10^{-8} \text{ K cm}^2/\text{dyn}$ which is the same value given by Timmermans.¹⁹

The thermodynamic quantities, ρ_c , dT_c/dp , α_p , C_p , and absorption data enable us to determine the adiabatic coupling constant g for nitrobenzene and n -hexane critical mixture. In particular, from Eq. (14), we obtain $g = -0.56$. An experimental value of $g = -0.69 \pm 0.015$ can be found from the experimental slope of α_c/f^2 vs $f^{-1.06}$.

Figure 6(a) and (b) shows the temperature dependence of the measured shear viscosity η (cp) above T_c at different concentrations with the critical concentration. Plots of the logarithms of the viscosity versus the reciprocal of the abso-

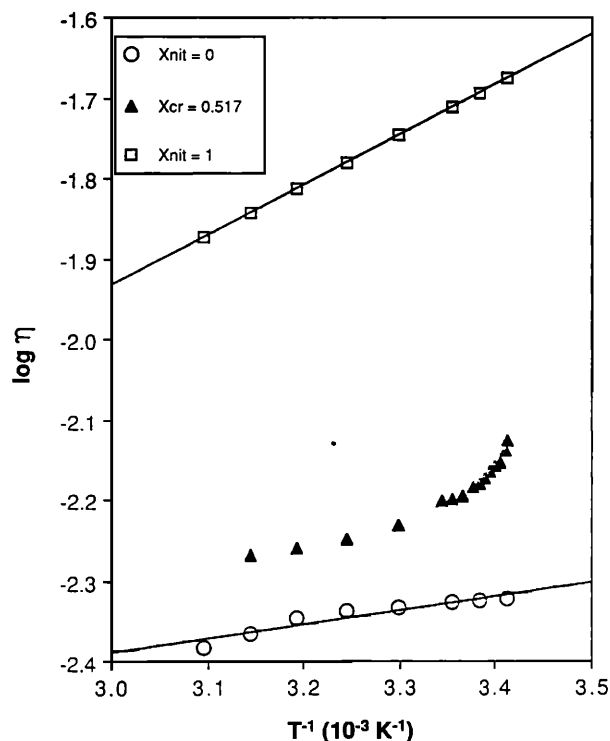


FIG. 7. Plot of the logarithms of the viscosity versus the reciprocal of the absolute temperature at concentrations $X_{nit} = 0$, $X_{nit} = 1$, and $X_c = 0.517$ by weight of nitrobenzene.

lute temperature at the concentration extremes and at the critical composition for the binary system are shown in Fig. 7. The background viscosity for the pure components obeys the equation, $\log \eta = A + B/T$, where $A = -3.79$ and $B = 619.6$ °K for nitrobenzene and $A = -2.92$ and $B = 175.2$ °K for *n*-hexane, where η was taken in poise. The error in the viscosity measurements was less than 0.5%. Table I shows the measured and the calculated values in this work and other references.

IV. DISCUSSION AND CONCLUSIONS

It is evident from the curves in Fig. 1 that α_c/f^2 increases as the critical temperature is approached. The lower the frequency, the more rapidly α_c/f^2 varies with the temperature. The observed absorption, for higher frequencies at the critical temperature and concentration, α_c/f^2 versus frequency fits well at the higher frequency end of the data for lower

frequencies measured by Manucharov.² Combining our shear viscosity and absorption with the density data of Miyake and Izumi,⁸ we were able to determine the asymptotic critical behavior of the thermal expansion coefficient and of the specific heat as well as the value of the adiabatic coupling constant, which appears in the dynamic scaling theory of critical sound attenuation. Our calculated value for the coefficient dT_c/dp gives very close agreement to that given by Timmermans.¹⁹ The velocity measurements show a good agreement with Sette's¹⁶ and Manucharov's² lower frequency measurements. It is shown from Fig. 1(b) that the background absorption is approximately the same for all of the measured concentrations. Our measured value of viscosity η_0 is in a good agreement with the values measured by Krivokhizha²⁰ and Chen.¹⁷ The values of α/α_c for nitrobenzene and *n*-hexane at the critical mixture are compared to the scaling function $F(\omega^*)$ and show a good agreement with the FB theory. The observed absorption of the α_c/f^2 versus $f^{-1.06}$ also yields a straight line as predicted by the FB theory. The Navier-Stokes term, which represents the background viscosity attenuation that would be present in the absence of the critical fluctuation can be neglected near the critical point. The value of the absorption due to shear viscosity term was estimated to be approximately $4 \times 10^{-17} \text{ cm}^{-1} \text{ s}^2$ compared to $\alpha_c/f^2 = 857 \times 10^{-17} \text{ cm}^{-1} \text{ s}^2$ at 15 MHz.

TABLE I. Some measured and calculated values.

Quantity	Measured	Calculated	From references
T_c (°C)			19.89 ^a
α			0.11 ^b
$z\nu$			1.9 ⁱ
γ			1.23 ^c
ξ_0 (Å)			3.54 ^c
η_0 (cp)	0.55		0.563 ^d 0.53 ^e
ω_0 (Hz)		1.75×10^{10}	8.01×10^{10e} 2×10^{10a}
D_0 (cm ² /s)		1.1×10^{-5}	1.65×10^{-5e}
ρ_c (g/cm ³)			0.8432 ^f
u_c (m/s)	1208.15		
du/dT (m K/s)	-3.6		
$(\partial\rho^{-1}/\partial t)$ (cm ³ /g)		0.3077	
$(\partial\rho^{-1}/\partial T)$ (cm ³ /gK)		1.05×10^{-3}	
α_{pc} (K ⁻¹)		0.312×10^{-4}	
α_{pb} (K ⁻¹)		8.346×10^{-4}	
C_{pc} {erg/(g K)}		0.066×10^7	
C_{pb} {erg/(g K)}			1.9×10^{7g}
dT_c/dp (K cm ² /dyne)		-1.64×10^{-8}	-1.64×10^{-8h}
$(\partial T/\partial P)_c$ (K cm ² /dyne)		1.59×10^{-8}	
At $T = 25$ °C			
S (cm ⁻¹ s ^{0.94})	3.94×10^{-7}	2.6×10^{-7}	
g	-0.69	-0.56	
A_{nit}	-3.79		
B_{nit} (K)	619.6		
A_{n-hex}	-2.92		
B_{n-hex} (K)	175.2		

^a Reference 1.

^b Reference 21.

^c Reference 17.

^d Reference 20.

^e Reference 16.

^f Reference 8.

^g Reference 2.

^h Reference 19.

ⁱ Reference 12.

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