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X-ray scattering study of the critical exponent η in argon

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A new technique for evaluating the critical exponent η, which governs the rate of decay of the pair-correlation function with distance at the critical temperature, is described. In this method, η is obtained from plots of the reciprocal of the intensity as a function of the square of the scattering angle in the inner part of the small-angle scattering curve. The new procedure has the advantage of making use of the data at scattering angles at which the intensity is relatively high and thus is quite insensitive to effects which may distort the scattering curves. For argon, the value $\eta = 0.03 \pm 0.02$ is obtained. The analysis of the scattering data also provides evidence for the need for corrections to the limiting scaling behavior which fluids exhibit very near the critical point.

I. INTRODUCTION

Although the results from small-angle x-ray scattering studies[1-3] of the equilibrium properties of argon near its liquid-vapor critical point agree within the experimental uncertainty with the predictions of the scaling theories[4,5] which have been very useful for describing the properties of fluids and other systems in the critical region, recent experimental investigations[6-10] have shown that corrections to the limiting behavior given by the scaling theories are larger than was expected previously.

These new results suggested the advisability of re-examining the small-angle x-ray scattering from argon in the critical region, in order to look for the effects of corrections to scaling. As these corrections do not have a large effect on the scattering curves, it is not surprising that the need for corrections was not evident in previous analyses of the scattering data in the critical region. Since our earlier data were not in a form convenient for observing the effects of corrections to scaling, we have re-examined some of our more recent small-angle x-ray scattering curves from argon in the critical region. Because of the experience acquired in our first scattering studies, the precision of the new curves is higher than was possible previously. Some results from the new analysis are reported below.

As we explain in Sec. II, we have developed a new method for using the small-angle scattering data to evaluate the critical exponent η, which describes the rate of decay of the pair-correlation function with distance[11] at the critical temperature T_c. This new technique for calculating η has the advantage of using scattering data from the inner part of the scattering curve, where, for a fluid near its critical point, the small-angle scattering is most intense. The scattering data therefore are recorded under conditions for which any effects which may distort the scattering curves can be expected to be small. (The inner part of the scattering curve is defined to be the interval of scattering angles for which $q\xi$ is not large, where $q = 4\pi\lambda^{-1}\sin(\theta/2)$, λ is the x-ray wavelength, θ is the scattering angle, and ξ is the long-range correlation length.)

Our calculations also have shown that the value of η computed from the scattering curves by our new method is quite sensitive to corrections to scaling, so that these corrections produce a much larger relative change in η than in other quantities obtained from the scattering data. Our new technique therefore has the advantages both of evaluating η under especially favorable conditions and of providing a particularly sensitive test for corrections to scaling.

From our analysis of the scattering curves, we have found that $\eta = 0.07 \pm 0.01$ if corrections to scaling are neglected, while if allowance is made for these corrections, $\eta = 0.03 \pm 0.02$. (The uncertainties in quantities determined from our scattering curves are the standard deviations calculated in our analysis of the data.) The exponent η obtained without corrections to scaling thus is appreciably greater than the values $\eta = 0.056 \pm 0.008$ and $\eta = 0.031 \pm 0.011$ calculated by high-temperature expansions[12] and renormalization-group methods,[13,14] respectively.

Thus, besides providing a way to determine η under very favorable conditions, our new technique also shows that the exponent computed from our scattering curves is in agreement with the theoretical calculations *only* when allowance is made for corrections to scaling.

II. CALCULATION OF η FROM THE ORNSTEIN-ZERNIKE PLOTS

The Ornstein-Zernike approximation[15] for the scattered intensity $I(q)$ in the critical region can be written

$$I(q)=\frac{I_{00}(1+t)}{1+q^2\xi^2}\chi\,, \tag{1}$$

where

$$t=|T-T_c|/T_c\,,$$

T is the absolute temperature, T_c is the critical temperature, I_{00} is a constant which depends on the experimental conditions,

$$\chi=(\rho/\rho_c)^2K_TP_c\,,$$

ρ is the density of the fluid, ρ_c is its critical density, K_T is the isothermal compressibility, and P_c is the critical pressure.

According to (1), $[I(q)]^{-1}$ is a linear function of $(q\xi)^2$. The scattering data can therefore be conveniently analyzed by an "Ornstein-Zernike plot," in which the reciprocal of the scattered intensity is plotted as a function of the square of the scattering angle. (In the small-angle region, q is essentially proportional to the scattering angle.)

The scaling theories state that near the critical point, for $T>T_c$ and $\rho=\rho_c$ (i.e., on the critical isochore above T_c),

$$\chi=\Gamma t^{-\gamma} \tag{2}$$

and

$$\xi=\xi_0 t^{-\nu}\,, \tag{3}$$

where γ, ξ_0, ν, and Γ are constants, and also that

$$\gamma=(2-\eta)\nu\,. \tag{4}$$

When these relations hold, that is, when corrections to scaling can be neglected, with the approximation $1+t\approx 1$, Eq. (1) can be written as

$$[I(q)]^{-1}=\frac{t^{\gamma}}{I_{00}\Gamma}+\frac{\xi_0^2t^{-\eta\nu}}{I_{00}\Gamma}q^2\,. \tag{5}$$

Equation (5) states that the slope

$$S=\frac{\xi_0^2t^{-\eta\nu}}{I_{00}\Gamma} \tag{6}$$

of the Ornstein-Zernike plot is proportional to $t^{-\eta\nu}$. Since $\log S$ is a linear function of $\log t$, $\eta\nu$ can be calculated from the slope of a plot of $\log S$ as a function of $\log t$. As the uncertainty in ν is much smaller than that in η, after $\eta\nu$ has been computed from the experimental values of S obtained from the scattering curve, the theoretically calculated value[14] $\nu=0.63$ can be used to obtain η without an appreciable increase in the uncertainty. As far as we know, the temperature dependence of the slope S of the Ornstein-Zernike plots has never been used previously to evaluate the critical exponent η.

III. CORRECTIONS TO SCALING

As (6) does not allow for any corrections to scaling, we have estimated these corrections by use of the approximate scattering equations which Chang *et al.*[10] obtained from Wegner's calculations.[16] The resulting scattering equation, which is valid when $q\xi$ is not large and which includes approximate corrections for scaling, can be written as

$$I(q)\approx I_{00}(1+t)\Gamma t^{-\gamma}\left(\frac{1}{1+q^2\xi^2}+\frac{\Gamma_1t^{\Delta_1}}{1+D_0q^2\xi^2}\right), \tag{7}$$

where $\xi=\xi_0t^{-\nu}$, as in (3), and D_0 and Δ_1 are universal constants for all systems of the same universality class, while the constant Γ_1 is system dependent. For $q=0$, (7) must be consistent with the expression[17]

$$\chi=\Gamma t^{-\gamma}(1+at^{\Delta_1}+\cdots) \tag{8}$$

giving the corrections to scaling for χ_0. The system-dependent constant a in (8) thus is equal to Γ_1. Equation (7) can be rearranged to give the expression

$$[I(h)]^{-1}=\frac{1}{I_{00}(1+t)\Gamma t^{-\gamma}}\times\left(\frac{1}{1+\Gamma_1t^{\Delta_1}}+\frac{1+D_0\Gamma_1t^{\Delta_1}}{(1+\Gamma_1t^{\Delta_1})^2}q^2\xi^2-E(q\xi)\right), \tag{9}$$

where

$$E(x)=\frac{(D_0-1)^2\Gamma_1t^{\Delta_1}}{(1+\Gamma_1t^{\Delta_1})^2[1+\Gamma_1t^{\Delta_1}+(D_0+\Gamma_1t^{\Delta_1})x^2]}x^4\,.$$

When $E(q\xi)$ and terms proportional to powers of $t^{1/2}$ higher than one are neglected, with the approximate value[13] $\Delta_1\approx\frac{1}{2}$ and with (8) and the scaling equation (4), (9) can be written as

$$[I(q)]^{-1}=\frac{t^{\gamma}}{I_{00}\Gamma[1+\Gamma_1t^{1/2}]}+S_c(t)q^2\,, \tag{10}$$

where

$$S_c(t)=At^{-\eta\nu}/(1+bt^{1/2})\,,\quad A=\xi_0^2/I_{00}\Gamma\,, \tag{11}$$

and

$$b=(2-D_0)\Gamma_1\,.$$

According to (10), $[I(q)]^{-1}$ is a linear function of q^2 even when corrections to scaling are not negligible.

IV. RECORDING OF DATA AND ANALYSIS OF THE SCATTERING CURVES

The scattering data discussed here were obtained with the apparatus and techniques described previously,[1] except that the new measurements were recorded with wider slits, in order to provide a greater scattered intensity. Corrections also were made for the effects of the slit widths on the scattering curves.[18]

Scattering data were recorded for a sealed argon sample with a density within about 0.1% of the critical density.[1] The data were considered to represent conditions on the critical isochore above T_c. Two sets of scattering curves, called Series I and Series II, respectively, were measured at 9 temperatures in the interval $0.12° \leq T - T_c \leq 3.53°$. The critical temperature was located[1] with a precision of $\pm 0.005°$. For our samples $T_c = 150.68 \pm 0.02$ K as measured by the techniques described in Ref. 1.

As in Ref. 1, the statistical uncertainty in a scattering measurement was set equal to $0.675N^{1/2}$, where N is the total number of counts recorded at this scattering angle. Propagation-of-error techniques[19] were then employed to estimate the uncertainties in the corrected intensities and in quantities computed from these intensities. The uncertainties given with our results are the standard deviations obtained in the propagation-of-error calculations.

For each scattering curve, we made a linear least-squares fit of $[I(q)]^{-1}$ as a function of q^2, with weights proportional to the inverse square of the statistical uncertainty in $[I(q)]^{-1}$. From each fit we obtained the slope and the intercept of the Ornstein-Zernike plot. Figure 1 shows a typical Ornstein-Zernike plot. All linear and nonlinear least-squares fits were made both by a modified Curfit program[20] and by the nonlinear least-squares fitting procedure NLIN of the Statistical Analysis System.[21] Both programs gave the same results.

Since the Ornstein-Zernike equation is only an approximation, and because there may be errors in the measured intensities, the values of the slope and intercept computed from the least-squares fit can depend on the interval of $q\xi$ used in the calculation. In order that the slopes and intercepts would be affected as little as possible by our choice of the interval of $q\xi$, after estimating ξ for each temperature from Fig. 4 of Ref. 1 and computing the largest angle θ_{m0} for which the condition $q\xi < 6$ was satisfied, we calculated the slope and intercept of an Ornstein-Zernike plot which used all intensity values for angles $\theta \leq \theta_{m0}$. We then discarded the intensity at θ_{m0} and computed the slope and intercept from all other intensities considered in the first fit. We continued this process by dropping the intensity for the largest angle used in each previous fit and computing the slope and intercept for the remaining values till

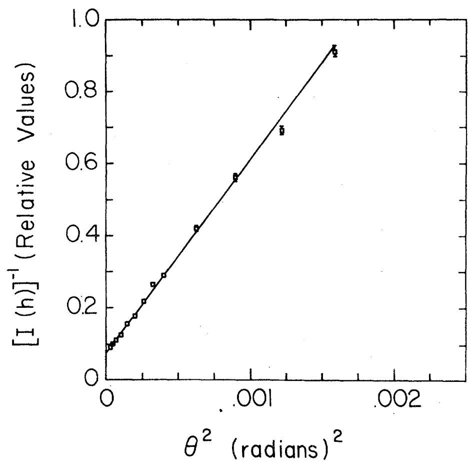

FIG. 1. The Ornstein-Zernike plot for Series I for $t = 0.0165$.

only those values for which $q\xi < 1$ remained. For each temperature we were able to find a range of $q\xi$ for which the slope of the Ornstein-Zernike plot was unaffected (within the standard deviation) by the choice of the largest scattering angle θ_m employed in the fit. We also found that when the slope was independent of the choice of θ_m, the value of the intercept was also unchanged within its standard deviation. In all analyses of quantities obtained from the scattering curves, we used the slopes and intercepts from the Ornstein-Zernike plots in which the slope was unaffected by the choice of θ_m. Calculations with (3), (4), and (5) showed that for all temperatures, when the slope did not depend on θ_m, $q\xi$ was in the interval $3 < q\xi < 5$.

As the incident scattered intensity was not the same for the two series, a scale factor had to be calculated before the slopes and intercepts from the two series could be compared. We obtained this factor by averaging the ratio of the slopes from the two series at each temperature. After this scale factor had been used to normalize the two series of curves, we had 18 slopes and 18 intercepts which were available for studying the temperature dependence of the slope and the intercept.

With the least-squares fitting programs we calculated γ in (2) and ν and ξ_0 in (3). We were unable to detect any effects of corrections to scaling on the values of γ, ν, and ξ_0 obtained from our data, since the precision in the intercept was not high enough, and we did not have values of the slope and intercept for a sufficient number of temperatures.

We calculated γ, ν, and ξ_0 for each series and also for the set of 18 slopes and intercepts obtained by use of the normalizing factor. The re-

TABLE I. Values of γ, ν, and ξ_0.

	ξ_0	γ	ν
High-temperature expansions (Ref. 12, p. 605)		1.250 ± 0.007[a]	0.6430 ± 0.0025
Renormalization group (Ref. 14)		1.241 ± 0.004	0.630 ± 0.002
Argon-from Series I	2.06 ± 0.09 Å	1.08 ± 0.02	0.56 ± 0.01
Argon-from Series II	1.32 ± 0.14 Å	1.25 ± 0.05	0.64 ± 0.02
Argon-from the combined results	1.70 ± 0.13 Å	1.14 ± 0.04	0.59 ± 0.02

[a] Calculated from (4) and the values of γ and ν from Ref. 12.

sults of the calculations are given in Table I. The values of γ, ν, and ξ_0 from the two samples differ by more than their standard deviations, but the exponents calculated from the combined results from the two series are in fair agreement with the corresponding exponents calculated for each series and also with the quantities given in Ref. 1. We are unable to explain the reason for the different results obtained from the two series. We expect that the quantities from Series II are more reliable, as they are in best agreement with the theoretical results. As we measured only the angular distribution of the scattering and did not evaluate the scattering cross section, we could not calculate the constant Γ in (2).

With (6) and (11) and the slopes of the Ornstein-Zernike plots, we made least-squares fits to evaluate η both when corrections to scaling are neglected and also when they are considered. In the calculations we made use of the result $\nu = 0.630\pm0.002$ from the renormalization-group calculations.[14] Our calculations of η are listed in Table II. For comparison we show the values of η obtained by substituting the exponents γ and ν from Table I in (4). We also give the η values from the theoretical calculations. In Fig. 2 we show the temperature dependence of the slope and the curves obtained from the least-squares fits of (6) and (11).

The uncertainties in the γ's and ν's used in (4) are so large that this equation does little more than provide an estimate of the magnitude of η. The exponents η obtained from (6)—that is, without corrections to scaling—are much larger than the values calculated by renormalization-group

TABLE II. The critical exponent η.

		Theory	
High-temperature expansions (Ref. 12, p. 605)		0.056 ± 0.008	
Renormalization group (Ref. 14)		0.031 ± 0.011	
		Argon	
	Series I	Series II	Combined data
From (11)	0.04 ± 0.04	0.02 ± 0.02	0.03 ± 0.02
From (6)	0.07 ± 0.01	0.08 ± 0.01	0.07 ± 0.01
From (4), with γ and ν from Table I	0.07 ± 0.04	0.05 ± 0.10	0.07 ± 0.08

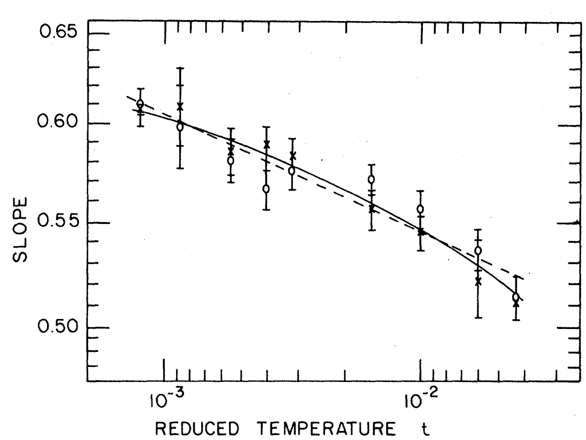

FIG. 2. Relative values of the slope $S_c(t)$ of the Ornstein-Zernike plots for several values of reduced temperature t. Circles and crosses show slopes calculated from the scattering curves of Series I and Series II, respectively, and the dashed and solid lines were obtained by least-squares fits (6) and (11), respectively, to the combined set of slopes.

methods and also are almost certainly inconsistent with the η from high-temperature expansions. On the other hand, (6) gives exponents quite near those obtained[3] when scaling corrections are neglected in the calculation of η from the part of scattering curve for which $q\xi \gg 1$. The η values from (6) also are only a little smaller than those found in a small-angle neutron scattering study[22] of neon in the critical region.

The exponent η obtained with (11) is much smaller than that calculated with (6) and is in good agreement with the value obtained by Chang *et al.*[10] and with the results from renormalization-group theory. It also is not inconsistent with the high-temperature-expansion calculations.

As the results in Table II show, the exponent η calculated from the scattering curve agrees with the results from renormalization-group and high-temperature-expansion calculations *only* when corrections to scaling are considered. If these corrections are neglected, η is clearly too large. The evaluation of η from the small-angle-scattering curves thus provides additional evidence for the need for corrections to scaling.

Moreover, our proposed technique for determining η provides a very sensitive test of the magnitude of corrections to scaling. For example, from our values of γ and ν, we were unable to estimate how much the scattered intensity was affected by corrections to scaling, since the magnitude of the change is relatively small. On the other hand, the *relative* change in η as a result of corrections to scaling is quite large, and thus the calculation of η from the scattering curves promises to be a particularly sensitive test for estimating the magnitude of corrections to scaling, as well as being an especially convenient method of evaluating η.

Table III lists the values of the constant b in (11) determined from the least-squares fits to the scattering data from each series of scattering curves and from the fit combining the results from the two series.

The η values calculated from (11) and (6) depend only on the slope of the Ornstein-Zernike plots and thus are not affected by errors in the intercept. To determine γ and ν, on the other hand, both the intercept and the slope must be known. While the standard deviations in the slopes which we calculated were of the order of 2%, the corresponding deviations in the intercepts were about 10%. It thus is not surprising that the uncertainty in the values of η obtained from (6) and (11) is less than that in the η's calculated from (4) with the exponents γ and ν from the Ornstein-Zernike plots.

TABLE III. The constant b in Eq. (11).

From the slopes from Series I	0.57 ± 0.75
From the slopes from Series II	1.10 ± 0.38
From the combined set of slopes	0.84 ± 0.43

When both the conditions $q\xi \gg 1$ and $qa \ll 1$ are satisfied, where a is the average intermolecular distance, and if corrections to scaling can be neglected, the scattering is proportional[23] to $q^{\eta-2}$. In Ref. 3, we described an investigation of the small-angle x-ray scattering from argon when $q\xi \gg 1$. Our data showed that if corrections to scaling are not applied, $\eta = 0.08$. This result, though in essential agreement with the exponent η found in a small-angle neutron-scattering study of neon,[22] was significantly larger than the theoretical values listed in Table II. In Ref. 3 we suggested that the high value of η could be the result of lack of consideration of corrections to scaling. Our most recent analysis of the scattering data from argon confirms this conclusion.

Our new procedure for determining η obtains this exponent from the quantity $\eta\nu$, while the technique employed in Ref. 3 gives the quantity $2-\eta$. Calculation of η from experimentally determined values of $(2-\eta)$ has the disadvantage that since η almost certainly is no greater than 0.06, the *relative* precision with which η can be obtained from the quantity $2-\eta$ is considerably less than the relative precision with which $2-\eta$ itself is known.

The technique used in Ref. 3 for determining η requires both that $q\xi \gg 1$ and that $qa \ll 1$. To test the degree to which the determination of η was affected by the fact that the latter condition may not have been satisfied, in Ref. 3 we calculated a factor which gave the first correction for the fact that qa was not negligible. We found that the estimated correction produced a change of about 0.01 in the calculated value of η. The change thus was of the same magnitude as the uncertainty in η. The correction will be even less important when η is evaluated from (6) or (11), since these equations use intensity values for scattering angles at which the correction for the effects of qa is smaller than for the conditions of Ref. 3, and also because (6) and (11) employ scattering data for which $q\xi$ is not large and for which the scattered intensity thus is higher than the intensities used in Ref. 3.

As the first corrections to scaling are usually proportional to t^{Δ_1}, Eq. (11) can be expected to give the first correction to scaling even if the details of the scattering theory developed by Chang *et al.*[10] do not apply to our data.

The quantity χ_r^2 provides a measure of how well a theoretical expression fits the results of an experiment.[24] [There is no relation between χ_r^2 and the reduced susceptibility χ introduced in (8).] The values of χ_r^2 obtained from our least-squares fits of (11) are only slightly smaller than when η is obtained from (6). For example, for the series combining the results from the two samples, χ_r^2 was 0.67 and 0.84 for the analysis with (11) and (6), respectively.

The strongest evidence which our latest analysis of the scattering data from argon can provide for the necessity for corrections to scaling is the fact that without these corrections, the values of η which are calculated from the scattering curves are clearly larger than the results from high-temperature-expansion and renormalization-group methods.

We would like to emphasize that Eqs. (5) and (10), and thus also (6) and (11), are based on the validity of the scaling relation (4). This dependence should be kept in mind, even though all available evidence indicates that (4) is valid.

The exponents γ and ν which we obtained for argon agree within the estimated uncertainty with the values $\gamma = 1.20 \pm 0.02$ and $\eta = 0.045 \pm 0.010$ which Anisimov *et al.* recently determined[25] by analysis of the light scattering data from a nitroethane-hexane critical mixture.

The quantity $-E(q\xi)/[I_{00}(1+t)\Gamma t^{-\gamma}]$ in (9) is the major part of the error caused by approximating (7) by (10). Quantitative estimates of this error are difficult because of the somewhat complex form of $E(x)$ and because as yet no information about the value of D_0 is available. At least tentatively, however, it is perhaps not unreasonable to expect that D_0 is positive and has a magnitude not large compared to one. Under these conditions, for small x the magnitude of the coefficient of x^4 in $E(x)$ will be primarily be determined by the factor $\Gamma_1 t^{\Delta_1}$, which has been assumed to be small. When x is large, $E(x)$ is proportional to x^2, and in the large-x limit, (9) is again a linear function of x^2, but with a different slope from that in (10). The difference in the slopes of the large-x and small-x limits is proportional to t^{Δ_1} and thus is small.

Since (9) thus can be approximated by nearly the same linear function of $(q\xi)^2$ for large and small $q\xi$, the approximate equation (10) can be expected to be useful for all $q\xi$ for which the data can be analyzed by an Ornstein-Zernike plot.

In the analysis of our data, we used intensities only for scattering angles for which the slope of the Ornstein-Zernike plots was essentially independent of the choice of θ_m. The fact that we were able to find conditions for which the slope of the Ornstein-Zernike plots did not depend on θ_m provides evidence that (10) can be used with our data.

As (7) itself is only approximate, attempts to find a better approximation to (9) than (10) are probably not worthwhile.

An equation like (10) for the first corrections to scaling is reasonable from quite general considerations. Since the first corrections to scaling are usually proportional to t^{Δ_1}, and since both terms in (10) can be obtained by adding these corrections to the two terms in (5), an equation like (10) can be obtained intuitively, without the use of a detailed scattering theory like that developed by Chang *et al.*[10]

The use of (11) to determine η from the slope of the Ornstein-Zernike plots provides a simple, convenient, and relatively precise method, never suggested previously, for evaluating the critical exponent η from small-angle scattering curves. The technique has the advantage of using scattering data near the central maximum of the scattering curve, where the intensity is near its maximum value and thus is quite insensitive to distortions of the scattering curve produced by the apparatus and also to effects which may not be negligible for large values of $q\xi$.

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