NMR determination of the nonclassical critical exponents β and $\bar{\beta}$ in incommensurate Rb_2ZnCl_4

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The critical behavior below the second-order transition between the normal and the incommensurate phase of Rb_2ZnCl_4 is studied by means of quadrupolar perturbed NMR. The temperature dependences of the ^{87}Rb $m=\pm\frac{1}{2}\leftrightarrow\pm\frac{3}{2}$ satellite transitions are investigated for different Rb nuclei and different crystal orientations. It is shown that the critical exponents β and $\bar{\beta}=2-\alpha-\phi\neq 2\beta$, ϕ being the crossover exponent associated with an uniaxial perturbation, can be determined accurately. The value $\beta=0.35\pm0.01$ and $\bar{\beta}=0.83\pm0.03$ obtained are in agreement with the theoretical predictions of the three-dimensional XY model.

I. INTRODUCTION

In several compounds of the A_2BX_4 type such as K_2SeO_4, Rb_2ZnCl_4, and Rb_2ZnBr_4, transitions from a normal (N) phase to an incommensurate (IC) phase occur. The IC phase can be described by a two-component order parameter, and therefore these systems are expected to belong to the universality class of the three-dimensional XY model.[1] The critical exponent of the order parameter $\beta=0.346\pm0.002$ predicted by this model[2] has been confirmed for the substances mentioned by several techniques[3–8] including nuclear magnetic resonance (NMR) within the given respective experimental errors. According to our knowledge, the most accurate determination[7] gives the value $2\beta=0.69\pm0.01$ for Rb_2ZnCl_4. The exponents γ and ν determined from x-ray diffuse scattering[9] are in reasonable agreement with the XY model.

In contrast, we recently reported[10] unconventional critical behavior of the NMR frequency splitting of the ^{87}Rb ($I=\frac{3}{2}$) $m=\pm\frac{1}{2}\leftrightarrow\pm\frac{3}{2}$ satellite transitions in Rb_2ZnBr_4 near the N-IC phase transition. It was shown that in the IC phase for the three different cases investigated, the widths of the ^{87}Rb satellite transition frequency distributions followed a power law with a critical exponent of about 0.84. In every case the crystal was orientated with one of the crystal axes parallel to the static magnetic field $\mathbf{H}_0$, thus realizing a "quadratic case," in which until now in all models the width of the frequency distribution has been assumed to be proportional to the square of the order parameter.[11–14] Hence, the critical exponent observed was clearly in contradiction to the expected value $2\beta\approx0.70$.

In this work, new NMR measurements for the ^{87}Rb satellites in Rb_2ZnCl_4 are reported. Firstly, we will present a measurement of the temperature dependence of the ^{87}Rb satellites in a crystal orientation where a term proportional to the order parameter is dominant (the "linear case"). The effects of the IC modulation on the resultant frequency distributions are strong, thus giving rise to a high accuracy in determining the critical exponent β. The value $\beta=0.35\pm0.01$ obtained for both, Rb(1) and Rb(2), is in accordance with the theoretical predictions and the values reported previously.

Secondly, it will be shown that for crystal orientations corresponding to "quadratic cases," the critical exponent describing the temperature dependence of the ^{87}Rb satellites deviates from 2β and equals 0.83 ± 0.03 for both Rb(1) and Rb(2). This result evidences that this critical behavior is not restricted to Rb_2ZnBr_4, but is also present in the compound Rb_2ZnCl_4, thus having a general character. This behavior will be explained by an argument stressed by Bruce[15] in a general context. There can be secondary order parameters and physical properties, which a direct extension of Landau theory would require to vanish as $(T_i-T)^{2\beta}$, but which in a more careful analysis are predicted to have quite a different critical exponent. An example for this type of behavior in IC phases with a two-dimensional order parameter are the second-order diffraction satellites. The intensity of these reflections has been predicted to vanish at T_i with the critical exponent $2\bar{\beta}\approx1.69$. The value of $\bar{\beta}$ is related to the specific heat exponent α and the crossover exponent ϕ associated with an uniaxial symmetry-breaking perturbation according to $\bar{\beta}=2-\alpha-\phi$.[3,16] Because of their weakness, however, diffraction second-order satellites can only be detected far below the transition temperature T_i. This fact, together with the necessary corrections of the diffraction data for extinction, multiple and diffuse scattering, make a stringent test of the critical behavior predicted quite difficult. Measurements by means of neutron scattering in K_2SeO_4 indicated in fact some deviation from the 4β power law, but the critical exponent $2\bar{\beta}$ predicted could not be confirmed.[3]

It will be shown in the following that the arguments leading to this nontrivial critical behavior for second-order diffraction satellites can be readily extended to the second-order Fourier amplitude of the electric-field-gradient tensor (EFG) in the IC phase. In particular, its critical exponent is given by $\bar{\beta}$. Since NMR measurements on ^{87}Rb satellites allow an accurate determination of the EFG modulation[10] and of the transition temperature, this technique can examine the above-mentioned theoretical predictions for both β and $\bar{\beta}$. The results for Rb_2ZnBr_4 reported in Ref. 10 constitute the first clear experimental evidence of the correctness of the predicted $\bar{\beta}$ value. The measurements reported here for Rb_2ZnCl_4,

which determine both β and $\bar{\beta}$, confirm the theoretical model.

II. CRITICAL BEHAVIOR OF THE EFG MODULATION

The EFG modulation in an IC phase with a single modulation wave vector can be described in general by means of a Fourier series along the superspace internal coordinate:[11]

$$V(v)=V_N+\sum_n V_n\exp(inv)\ . \tag{1}$$

Assuming fast motion, only the mean value of the EFG is relevant for quadrupolar perturbed NMR. In Ref. 11 the mean value of the EFG was related by a Taylor expansion to the mean atomic displacements. Thus, the mean values V_1 and V_2 were shown to be proportional to ρ and ρ^2, respectively, ρ being the order parameter modulus. As will be shown, these results have to be corrected if fluctuations are taken into account.

Dealing with the critical behavior near T_i, the discussion can be restricted to the plane-wave limit. Then the instantaneous local order parameter, $\eta(\mathbf{T})\exp[i\varphi(\mathbf{T})]$, $\mathbf{T}$ being the label for the cells of the nondistorted structure, can be identified with the local complex amplitude of the instantaneous atomic modulation. The mean atomic displacements [see Eq. (31) in Ref. 11] are then given by substituting the complex amplitude $\eta(\mathbf{T})\exp[i\varphi(\mathbf{T})]$ by its thermal mean value $\langle\eta(\mathbf{T})\exp[i\varphi(\mathbf{T})]\rangle$, which by definition is the static order parameter $\rho\exp(i\varphi_0)$. In the plane-wave limit this mean value is space independent and its phase φ_0 is arbitrary (this phase has been taken to be zero in Ref. 11). Substituting the amplitude ρ of the order parameter by its instantaneous value $\eta(\mathbf{T})\exp[i\varphi(\mathbf{T})]$, we can then follow the same arguments as in Ref. 11 to demonstrate that the instantaneous values of the EFG Fourier amplitudes $V_1(\mathbf{T})$ and $V_2(\mathbf{T})$ are in the first approximation proportional to $\eta(\mathbf{T})\exp[i\varphi(\mathbf{T})]$ and $\eta^2(\mathbf{T})\exp[i2\varphi(\mathbf{T})]$, respectively. Consequently the corresponding Fourier amplitudes of the mean EFG $V_n\equiv\langle V_n(\mathbf{T})\rangle$ will satisfy

$$|V_1|\equiv|\langle V_1(\mathbf{T})\rangle|\propto|\langle\eta(\mathbf{T})\exp[i\varphi(\mathbf{T})]\rangle|\equiv\rho\ , \tag{2a}$$

$$|V_2|\equiv|\langle V_2(\mathbf{T})\rangle|\propto|\langle\eta^2(\mathbf{T})\exp[i2\varphi(\mathbf{T})]\rangle|\ . \tag{2b}$$

Thus, the amplitude $|V_1|$ of the first harmonic of the EFG modulation is proportional to the amplitude ρ of the static order parameter.

Defining

$$P_1(\mathbf{T})+iP_2(\mathbf{T})\equiv\eta(\mathbf{T})\exp[i\varphi(\mathbf{T})]\ ,$$

for the amplitude of the second Fourier term $|V_2|$, the following relation holds:

$$|V_2|\propto\{[\langle P_1^2(\mathbf{T})\rangle-\langle P_2^2(\mathbf{T})\rangle]^2+4\langle P_1(\mathbf{T})P_2(\mathbf{T})\rangle^2\}^{1/2}\ . \tag{3}$$

The right-hand side of (3) differs not only from ρ^2, but also from $\langle\eta^2(\mathbf{T})\rangle$ and is precisely the square root of the expression appearing in Ref. 3 for the temperature dependence of the intensity of second-order diffraction satellites. The argument developed there is therefore also valid in the present context. Thus we can expect that

$$|V_2|\propto(T_i-T)^{\bar{\beta}} \tag{4}$$

with $\bar{\beta}=2-\alpha-\phi$, α being the specific heat exponent of the d=3 XY model and ϕ the crossover exponent associated with an uniaxial symmetry-breaking perturbation of this model.[3,16,17] In the discussion so far only the first harmonic of the atomic modulation (order parameter) has been taken into account in the expansion of the EFG in terms of the atomic displacements (cf. Sec. II of Ref. 11). Nevertheless, Eq. (4) holds as well, if a second harmonic is present in the atomic modulation, since its mean amplitude also has the critical exponent $\bar{\beta}$.[3,15] This is analogous to what happens in the case of second-order diffraction satellites.[3]

Consequently, two different types of "quadratic" contributions to the quadrupolar perturbed NMR frequency modulation in IC phases have to be distinguished.

(a) The second harmonic in the frequency modulation originates in the second harmonic of the EFG. Its amplitude will then vanish at T_i following the law $(T_i-T)^{\bar{\beta}}$ with $\bar{\beta}\neq2\beta$.

(b) The second harmonic in the frequency modulation is a consequence of a second-order perturbation term of the EFG first harmonic. In that case the relevant average values of the EFG components have to be determined differently according to whether the fast or slow motion regime is realized.[18,19] Assuming fast motion, the critical behavior of the second harmonic in the frequency modulation will then be given by $(T_i-T)^{2\beta}$, while in the slow motion regime the critical exponent $\bar{\beta}$ will dominate.

Obviously, case (a) will be realized in the satellite frequency modulations, which are practically only due to first-order quadrupolar perturbation.[10] For the $m=\frac{1}{2}\leftrightarrow-\frac{1}{2}$ central transition, in general the second harmonic of the frequency modulation includes both types of contributions, (a) and (b). Their relative weight depends on the actual magnitudes of the several EFG Fourier components and the crystal orientation. Thus, a special case might be possible where the contribution of type (b) becomes dominant. This would explain the results of Nakamura *et al.*[4] These authors could fit the distribution widths of the central line in a quadratic case with an exponent $2\beta\approx0.70$, in contrast to the results of this work and of Ref. 10.

Renormalization-group techniques and series expansions give for ϕ the value 1.175 ± 0.015,[20] which taking for α the value -0.02 results in $\bar{\beta}\approx0.84$.[17] However, considering a more precise theoretical estimate of $\alpha=-0.007\pm0.006$,[2] $\bar{\beta}$ becomes 0.832 ± 0.021.

III. EXPERIMENTAL DETAILS

Crystals were available from previous investigations on Rb_2ZnCl_4.[21] The sample used in the NMR experiment had a size of about 0.6 cm^3 and was orientated by goniometric methods. The NMR equipment including special mechanical devices for adjusting the crystal's orientation with respect to the direction of the static magnetic field $\mathbf{H}_0$ was described previously.[10,21]

A specially adapted standard gas-flow temperature regulation provided a temperature stability $\Delta T \lesssim \pm 0.05$ K over the measuring period (time for scanning was 1–5 min for $T_i - T \lesssim 10$ K). The temperature was measured by a thin platinum resistance thermometer located parallel to the axis of the sample coil in a distance of about 3 mm from the sample. The temperature gradient over the sample was about 0.1 K (cf. Fig. 1). This value is much smaller than the one given in the preceding work[10] because of the following reasons: The probe head was modified to direct the gas flow around the sample in a way minimizing the temperature gradient. In addition, the temperature range investigated in this work was close to ambient temperature. In different runs temperature was lowered step by step.

IV. EXPERIMENTAL RESULTS

A. First harmonic of the EFG modulation

The upper frequency satellite transition $m = \pm\frac{1}{2} \leftrightarrow \pm\frac{3}{2}$ of the ^{87}Rb nucleus ($I = \frac{3}{2}$) in Rb_2ZnCl_4 was measured within a temperature range $0 \lesssim T_i - T \lesssim 20$ K. The orientation of the crystal was determined to be $\hat{\mathbf{c}} \perp \mathbf{H}_0$, $\angle(\hat{\mathbf{a}}, \mathbf{H}_0) = 45.2°$. Crystal axes are defined such that

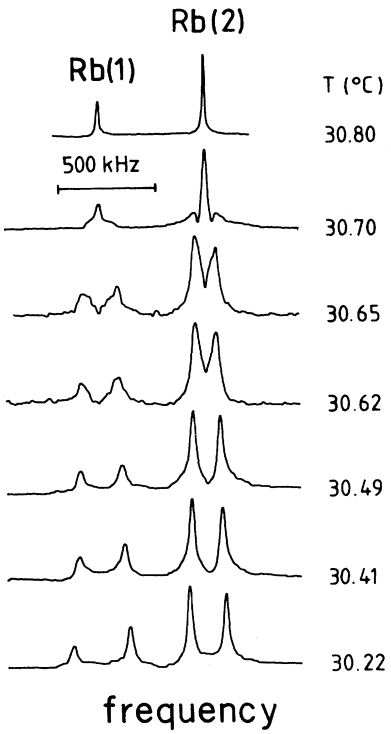

FIG. 1. Spectra of the ^{87}Rb upper frequency satellite transition in the crystal orientation $\hat{\mathbf{c}} \perp \mathbf{H}_0$, $\angle(\hat{\mathbf{a}}, \mathbf{H}_0) = 45.2°$ obtained near T_i for the two different groups of Rb nuclei Rb(1) and Rb(2). (Phase distortion was eliminated by "magnitude calculation," which gives the square root of the power spectrum. As a consequence, the well-known typical IC lineshape in absorption is changed and lines and edge singularities are broadened by a factor of about 1.6 compared to absorption spectrum.) The relative accuracy of the given temperatures is $\Delta T \lesssim \pm 0.03$ K.

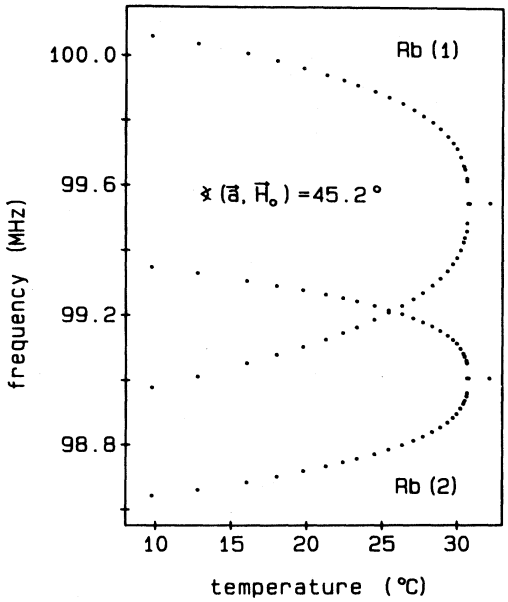

FIG. 2. Temperature dependence of the frequencies of the intensity maxima (edge singularities in the IC phase) of the ^{87}Rb spectra specified in Fig. 1.

$a > c > b$, i.e., the spacegroup of the N phase is *Pcmn*. The results are summarized in Figs. 1–3.

Figures 1 and 2 demonstrate the strong effect of the N-IC phase transition on the ^{87}Rb satellites in the given crystal orientation. Within a temperature interval of about 0.05 K (this is at the limit of our temperature resolution because of the available temperature stability and gradient) the discrete line characterizing the nonmodulated N structure splits into a frequency distribution of a

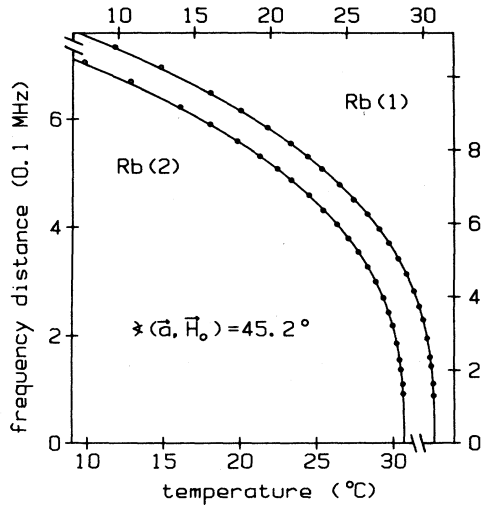

FIG. 3. Temperature dependences of the frequency distance between the edge singularities shown in Fig. 2. The fit curves correspond to a power law with $\beta = 0.35$ for both Rb(1) and Rb(2).

width of about 130 kHz for Rb(1) and 90 kHz for Rb(2), reflecting the modulation of the IC structure. The edge singularities of the frequency distribution appear to be rather broad and smeared. This smearing is reasonably attributed to the combined influence of the pronounced change of the spectra with temperature and the temperature gradient over the sample (and temperature fluctuations over the measuring period). Accordingly, the widths of the edge singularities decrease on passing to lower temperatures where the temperature dependence of the spectra is less pronounced. Because of the same reasons, the spectrum at $T=30.7\,^{\circ}\mathrm{C}$ already includes some IC contribution. In fact, an intensity comparison shows that about 40% of the crystal has transformed to the IC phase at that temperature. Therefore, for the evaluation of our experimental data, T_i is taken to be $(30.70\pm0.05)\,^{\circ}\mathrm{C}$.

Following the procedure described, for example, in Ref. 10 [cf. e.g., Eqs. (9) and (10) therein], it can be shown that in the given orientation and temperature range the frequency distance of the edge singularities is dominated by the amplitude $|V_{1xy}|$ of the first harmonic of the modulation of the corresponding EFG tensor component. In fact, using the Fourier components determined at $T\approx 10\,^{\circ}\mathrm{C}$ from measurements of the orientational dependences of the ^{87}Rb satellite transitions, the influence of the main diagonal elements V_{xx}, V_{yy} appearing in first-order quadrupolar perturbation can be calculated to be about 0.05% for Rb(1) and 0.002% for Rb(2) at $T_i-T\approx 20$ K. At higher temperatures their influence is even less. Contribution of second-order quadrupolar perturbation to the IC frequency splitting is of the order 10^{-3}%. Thus, the experimental data presented in Fig. 3 can be fitted according to Eq. (2a) yielding a critical exponent $\beta=0.35\pm0.01$ for both Rb(1) and Rb(2). Within the given accuracy, this value coincides with the value predicted by theory (see Sec. I). The given possible error includes the contribution originating from the uncertainty in determining T_i, which is one of the main contributions. The value for T_i given above has been confirmed by taking T_i as an additional fit parameter or equivalently by varying T_i in a double logarithmic plot of the experimental data in Fig. 3.

B. Second harmonic of the EFG modulation

To investigate the temperature dependence of the EFG second harmonic, the upper frequency satellite transition of the ^{87}Rb nucleus in Rb_2ZnCl_4 was also measured in a crystal orientation with $\hat{\mathbf{a}}\|\mathbf{H}_0$. There, to a very good approximation, the frequency distance between the edge singularities is proportional to the amplitude $|V_{2xx}|$ of the second harmonic of the modulation of the corresponding EFG tensor component [cf. e.g., Eqs. (16) and (17) in Ref. 10].

The frequencies obtained for the edge singularities in the IC phase and for the discrete line in the N phase (called N line henceforth), respectively, are plotted as a function of temperature in Fig. 4. Especially near T_i the widths of the frequency distributions are much smaller than in the "linear case" (Sec. IV A). Thus, close to T_i no

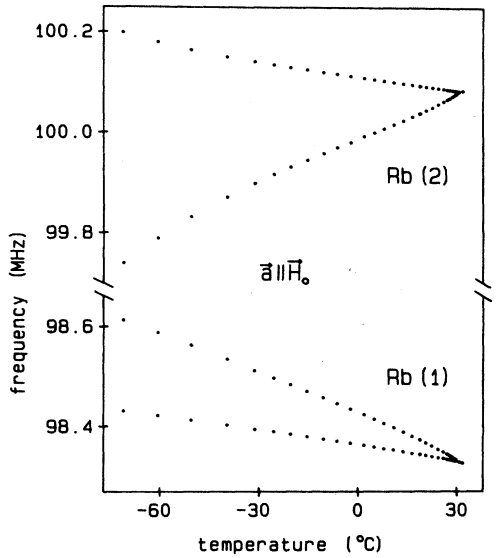

FIG. 4. Temperature dependence of the frequencies of the intensity maxima of the spectra of the ^{87}Rb upper frequency satellite transition for $\hat{\mathbf{a}}\|\mathbf{H}_0$.

splitting into edge singularities can be detected at all, but only a broadening of the N line. In order to obtain even close to T_i reliable data for the frequency distance of the edge singularities of the "ideal" frequency distribution [cf. Eq. (16) in Ref. 10], the effect of the convolution of the N line with the ideal frequency distribution was taken into account. Using power spectra like lineshapes (cf. caption of Fig. 1) for determining the edge singularity frequencies, this deconvolution procedure is only relevant in a small temperature region near T_i [$T_i-T\lesssim 1$ K for

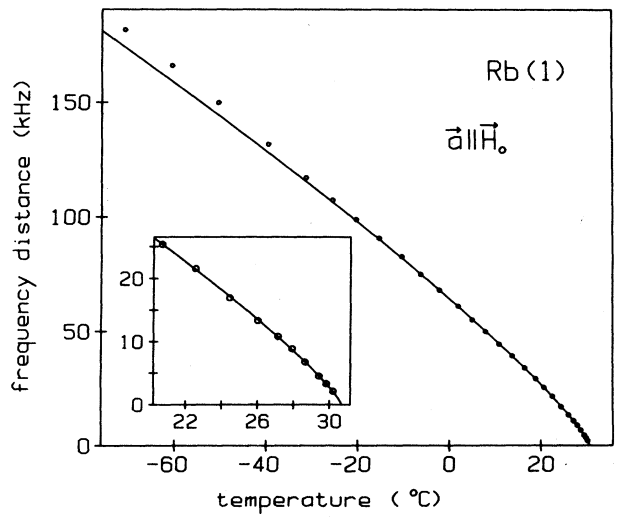

FIG. 5. Temperature dependence of the frequency distance between the edge singularities of the ideal frequency distribution of Rb(1) calculated from the data in Fig. 4 by the procedure described in the text. The inset shows the temperature range near T_i. The fit curve corresponds to a power law with $\bar{\beta}=0.83$.

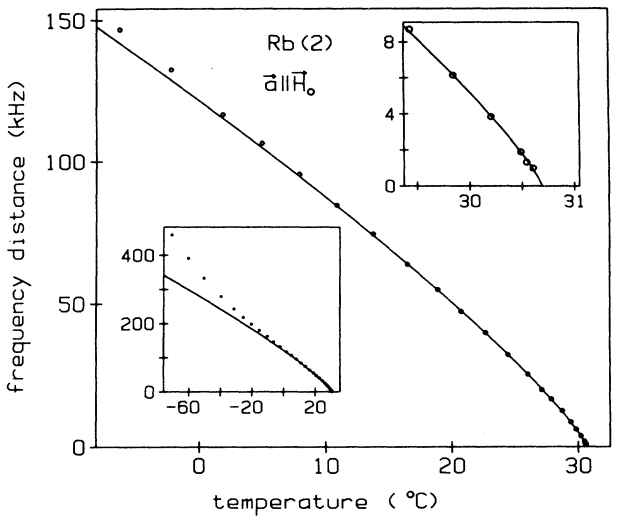

FIG. 6. As Fig. 5, but for Rb(2).

Rb(1), $T_i - T \lesssim 0.5$ K for Rb(2); such a deconvolution procedure is completely irrelevant in the linear case discussed in Sec. IV A]. The values of the frequency distances thus obtained are plotted in Figs. 5 and 6 versus temperature and are fitted according to Eq. (4). For both Rb(1) and Rb(2), the fits yield the critical exponent $\bar{\beta}=0.83$ with a tolerance of about ± 0.03 in accordance with the theoretical prediction (see Sec. II). As demonstrated especially by the upper inset of Fig. 6, the critical behavior can be followed up to temperatures close to T_i. Deviations from the power law become obvious approximately for $T < -25\,^{\circ}\mathrm{C}$ for Rb(1) and $T < 0\,^{\circ}\mathrm{C}$ for Rb(2). This does not prove that for these temperatures the power law for the order parameter is violated. Rather these deviations can also be caused by the fact that far away from T_i, Fourier terms of higher order become relevant in the EFG modulation.

Analogous experiments were done for the crystal orientation $\hat{\mathbf{b}} \| \mathbf{H}_0$ yielding the temperature dependence of $|V_{2yy}|$. The exponents $\bar{\beta}$ obtained for Rb(1) and Rb(2) coincide, within the tolerances, with the value given above. The results are also in accordance with the exponents $\bar{\beta}=0.83\pm0.02$, 0.84 ± 0.03, and 0.86 ± 0.02 determined previously for Rb_2ZnBr_4 (Ref. 10) for the three different cases investigated.

V. CONCLUDING REMARKS AND SUMMARY

In a previous work[22] (cf. also Ref. 13) the idea was introduced that in the IC phase near T_i the floating of the phase of the pinned incommensurate modulation should result in a more or less pronounced partial motional averaging of the incommensurate spectrum. This should give rise to a deviation of the temperature dependence of the width of the IC spectra from the usual power law. It has been stated[13,22] that this effect indeed becomes obvious in the ^{87}Rb central line spectra observed in Rb_2ZnCl_4. Such a deviation from the power law could not be detected in our experiments. From a physical point of view this is to be expected because [cf. Eqs. (2a) and (2b)] the relevant fluctuations are already included in the calculation of the critical temperature dependences, e.g., of the order parameter amplitude.

Sometimes attempts are made[23,24] to interpret the temperature dependences observed besides by the nonclassical critical exponents given above by applying a Landau-type theory taking into account in a suitable expansion of the free energy at least terms up to the sixth order in the order parameter. Our data, however, reveal a basic argument against the validity of Landau theory: If Landau theory strictly holds, Eqs. (2a) and (2b) can be represented as $|V_1| \propto \rho$, $|V_2| \propto \rho^2$. Consequently it should be possible to represent the data of Figs. 5 and 6 as the squares of the ones in Fig. 3 with a respective constant scale factor between them. In terms of exponents this would mean $\bar{\beta}=2\beta$. This, obviously, is not the case.

We have studied the critical behavior at the second-order N-IC phase transition at $T_i \approx 30\,^{\circ}\mathrm{C}$ of Rb_2ZnCl_4, which is a representative of the large group of A_2BX_4 compounds by investigating the temperature dependences of the distributions of the ^{87}Rb NMR satellite frequencies for different crystal orientations. By that means the critical exponents β and $\bar{\beta}$ could be determined. The results are consistent with the nonclassical exponents predicted by the $d=3$ XY model.

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