Quadrupole splitting and nuclear magnetic relaxation in $ErCrO_3$ near a Morin-type phase transition

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(Submitted 20 October 1982)
Zh. Eksp. Teor. Fiz. **85**, 224–231 (July 1983)

Nuclear magnetic resonance on Cr nuclei is investigated in antiferromagnetic $ErCrO_3$ near the "easy axis–easy plane" phase transition (Morin transition). The following new phenomena are observed: a) a dependence of the NMR rate on the quadrupole multiplet number, b) an anomalous transverse magnetic relaxation whose rate cannot be measured by the standard two-pulse echo technique, and c) nonequidistant quadrupole splitting.

PACS numbers: 76.60.Es, 76.60.Gv, 75.50.Ee

We have continued the study, initiated in Refs. 1–3, of NMR near magnetic orientational phase transitions in rare-earth magnets. A distinguishing feature of the investigated samples is that the quadrupole splitting for their nuclei ^{53}Cr ($I = \frac{3}{2}$) turned out to be considerably larger than the inhomogeneous line width.[3] It was therefore possible to investigate the behavior of the nuclear magnetization by exciting only one line of the quadrupole multiplet. In contrast to the earlier paper,[3] devoted to the study of the temperature dependence of the NMR frequencies, we consider here the behavior of the quadrupole splitting in the antiferromagnetic (AF) and weakly ferromagnetic (WF) phases and of the rates of the transverse and longitudinal nuclear magnetic relaxation for each of the components of the quadrupole multiplet. To our knowledge, no such investigations were performed before.

SAMPLES AND MEASUREMENT TECHNIQUE

We investigated $ErCrO_3$ single crystals grown by spontaneous crystalization from a molten solution in with natural abundance of the ^{53}Cr isotope.

The NMR signals from the ^{53}Cr nuclei were recorded by a two-pulse spin-echo technique using a spectrometer with frequency scanning and an automatic plotter. The conditions for the formation of the echo signals corresponded to excitation of nuclear spins inside the domains. The durations of the exciting pulses were $\tau_1 = 6$ and $\tau_2 = 2$ μsec, and the pulse repetition frequency was $f = 98$ Hz. The transverse relaxation time was measured by the usual two-pulse procedure described in Refs. 1 and 4. The initial delay was chosen to be $\tau_0 = 5$ μsec. The echo amplitude was measured with a stroboscopic integrator and was also monitored on the oscilloscope screen. The temperature was maintained constant by placing the heated volume with the sample in a helium bath. The temperature instability did not exceed ± 0.01 K.

τ_1 was measured by determining the change of the amplitude of the stimulated echo, as described in Ref. 4. The delay time τ_1 between the first and second exciting pulses was chosen equal to 20 μsec, to prevent influence of the free-induction signal on the echo amplitude.

MEASUREMENT RESULTS

As shown in Refs. 5 and 6, a Morin-type transition takes place in $ErCrO_3$ at $T = 10$ K. Below 10 K the crystal is in the AF state, and above in the WF state. The NMR spectrum in the AF phase comprises three ($I = 3/2$) fully resolved equidistant lines 120 kHz wide and with quadrupole splitting $\nu_Q^{AF} = 640$ kHz. Figure 1 shows the temperature dependences of the rates of the transverse (T_2^{-1}) and longitudinal (T_1^{-1}) nuclear magnetic relaxations for all three components of the multiplet. We call attention to the following features of these dependences:

1. Both rates T_1^{-1} and T_2^{-1} increase as the Morin point is approached, in contrast to the compound $DyFe_{0.998}Co_{0.002}O_3$, in which the rate T_2^{-1} decreased.[2]

2. The rate T_2^{-1} for the central component of the multiplet increases more rapidly than for the outermost components, as a result of which the central component vanishes at $T = 7.2$ K, whereas for the outer components T_2 can be measured up to the temperature $T = 9.45$ K. The evolution of the NMR component as the Morin point is approached is shown in Fig. 2.

3. The rate T_1^{-1} is the same for all three components and is close in value to T_2^{-1} for the outer components of the multiplet.

In the WF phase the NMR signals have the following features:

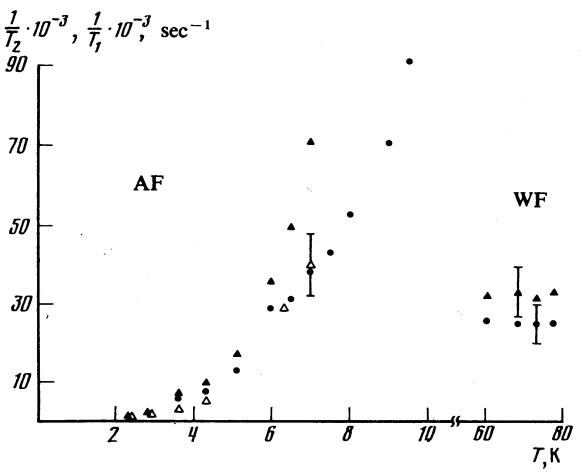

FIG. 1. Temperature dependence of the rate of the transverse nuclear magnetic relaxation for the quadrupole components of the NMR spectrum of ^{53}Cr in $ErCrO_3$ near a Morin-type transition: ●—T_2^{-1} for the outer components; ▲—T_2^{-1} for the middle component, △—T_1^{-1} for all components. In the WF phase $T_1^{-1} \approx T_2^{-1}$ for all components.

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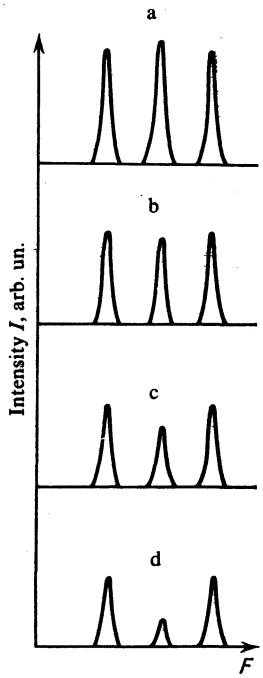

FIG. 2. Evolution of NMR spectrum of ^{53}Cr in $ErCrO_3$ as the phase transition is approached at the following temperatures (F is the frequency): *a*—$T = 4.2$ R; *b*—6.0; *c*—6.5; *d*—7.0.

1. In the temperature interval $10 \leqslant T \leqslant 37$ K no NMR signal was observed. In the interval $37 \leqslant T \leqslant 91$ K the rates T_1^{-1} and T_2^{-1} are independent of temperature within the limits of experimental accuracy.

2. At $T \geqslant 63$ K three equidistant lines are observed with a quadrupole splitting $\nu_Q^{WF} = 450$ kHz (Fig. 3). At $T < 63$ K the quadrupole splitting becomes nonequidistant (Fig. 3). Thus, at $T = 37$ K the frequency difference between the central and lower components is ≈ 325 kHz and ≈ 475 kHz, respectively.

DISCUSSION OF RESULTS

We were unable to describe the features listed above within the framework of the existing theory of NMR in magnets, and to find an explanation we had to go outside the framework of the traditional concepts.

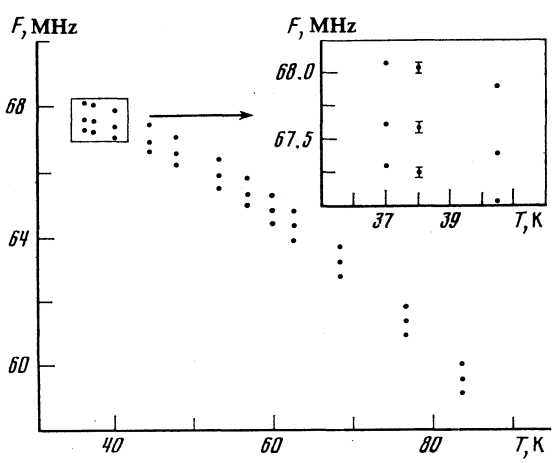

FIG. 3. Temperature dependence of the NMR frequencies of ^{53}Cr in $ErCrO_3$ in the WF phase.

1. Nonequidistance of the quadrupole splitting

Since $ErCrO_3$ is a triaxial crystal, its quadrupole interaction can be written in the form[7]

$$H_Q = {}^1/_2 h\nu_Q [I_z^2 - {}^1/_3 I(I+1) + {}^1/_6 \eta (I_+^2 + I_-^2)], \quad (1)$$

where $I_\pm = I_x \pm iI_y$; the axes x, y, and z coincide with the principal axes of the electric-field-gradient tensor; $I_{x,y,z}$ are the operators of the projections of the nuclear spin on these axes; h is Planck's constant, ν_Q is the quadrupole splitting, and η is a parameter of the order of unity and characterizes the anisotropy of the electric-field gradients in the x,y plane. The corrections to the nuclear-spin levels due to the off-diagonal terms with $I_\pm^2$ appear only in second-order perturbation theory. For the ^{53}Cr nuclei with $I = 3/2$ at an NMR frequency $\nu_n \approx 70$ MHz and $\nu_Q = 0.5$ MHz these corrections, according to our estimates, turned out to be of the order of 1 kHz, which is outside the limits of the accuracy of our measurements. The quadrupole effects are thus determined entirely by the first term of (1), and consequently the NMR frequencies corresponding to the dipole transitions $\frac{3}{2} \leftrightarrow \frac{1}{2}(\nu_{n,1/2})$, $\frac{1}{2} \leftrightarrow -\frac{1}{2}(\nu_{n,-1/2})$, and $-\frac{1}{2} \leftrightarrow -\frac{3}{2}(\nu_{n,-3/2})$ are given by the expressions

$$\nu_{n,1/2} = \nu_n - \nu_Q, \quad \nu_{n,-1/2} = \nu_n, \quad \nu_{n,-3/2} = \nu_n + \nu_Q, \quad (2)$$

which describe the equidistant quadrupole splitting.

It follows from Fig. 3 that the maximum nonequidistance of the dipole splitting reaches 150 kHz, i.e., is comparable with the inhomogeneous NMR line width. The possible cause of this equidistance is that for one of the outer components of the multiplet the maximum intensity of the signal corresponds not to the center of the NMR line, but to its right-hand edge (Fig. 4). A similar phonomenon occurs, for example for domain walls,[8] when the maximum of the signal intensity occurs on one of the edges of the NMR frequency band.

In our case the indicated shift of the signal-intensity maximum can be due to the fact that the hyperfine-field inhomogeneities, which determine the inhomogeneities of the frequency ν_n in (2), and the inhomogeneities of the quadru-

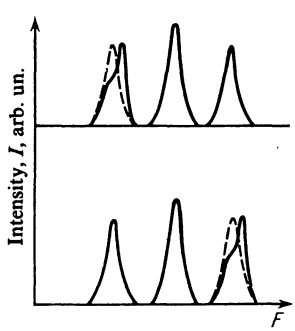

FIG. 4. Quadrupole splitting for the case when the maximum of the echo signal intensity (solid line) does not coincide with the center of the distribution in frequency (dashed); top—shift of the intensity maximum of the lower line of the quadrupole multiplet; bottom—shift of the intensity maximum of the upper line (F is the frequency).

pole splitting ν_Q have the same cause. Thus, if the frequency ν_n is written in the form

$$\nu_n=\nu_{n0}+\Delta\nu, \tag{3}$$

where ν_{n0} is the value of ν_n corresponding to the distribution center, the frequency ν_Q us given by

$$\nu_Q=\nu_{Q0}+\Delta\nu_Q(\Delta\nu), \tag{4}$$

where $\Delta\nu_Q(\Delta\nu)$ is a function of $\Delta\nu$ and its form depends on the mechanism whereby the indicated cause influences the inhomogeneities of ν_n and ν_Q. If $\Delta\nu_Q(\Delta\nu)$ can be written in the form of a series in powers of $\Delta\nu$:

$$\Delta\nu_Q(\Delta\nu)=\alpha\Delta\nu+\beta\Delta\nu^2+\dots, \tag{5}$$

we obtain from (2) and (5) for the NMR frequencies.

$$\nu_{n,1/2}^{WF}(\Delta\nu)=\nu_{n0}-\nu_{Q0}+(1-\alpha)\Delta\nu-\beta\Delta\nu^2-\dots,$$
$$\nu_{n,-1/2}^{WF}(\Delta\nu)=\nu_{n0}+\Delta\nu,$$
$$\nu_{n,-1/2}^{WF}(\Delta\nu)=\nu_{n0}+\nu_{Q0}+(1+\alpha)\Delta\nu+\beta\Delta\nu^2+\dots.$$

We recognize now that the intensity of the NMR signals at the frequency ν is proportional to the density of states at this frequency, i.e., to the number $n(\nu)$ of nuclear spins with resonant frequencies $\nu_n\approx\nu$. (For inhomogeneously broadened lines, $n(\nu)$ describes the line shape of the NMR absorption signal in the continuous-passage regime.) It can be shown (see the Appendix) that when relations (6) are satisfied the density of the states $n(\nu)$ for all three components of the multiplet can be written in the form

$$n_m(\nu)=f(\nu-\nu_{nm}^{WF}(0))\,[d\nu_{nm}^{WF}(\nu-\nu_{nm}^{WF}(0))/d(\Delta\nu)]^{-1}, \tag{7}$$

where $f(\Delta\nu)$ is the spin distribution function in the inhomogeneities of the hyperfine fields. Taking (6) into account we obtain for $n_m(\nu)$ the following expressions:

$$n_{1/2}(\nu)=f(\Delta\nu_{1/2})\,[1-\alpha-2\beta\Delta\nu_{1/2}-\dots]^{-1},\quad n_{-1/2}(\nu)=f(\Delta\nu_{-1/2}),$$
$$n_{-3/2}(\nu)=f(\Delta\nu_{-3/2})\,[1+\alpha+2\beta\Delta\nu_{-3/2}+\dots]^{-1},\quad \Delta\nu_m=\nu-\nu_{nm}^{WF}(0). \tag{8}$$

It can be seen from (8) that the maximum of $n_m(\nu)$ is determined by the maximum of $f(\Delta\nu)$ only for the central component of the multiplet $n_{-1/2}(\nu)$. For the outer components $n_{1/2}(\nu)$ and $n_{-3/2}(\nu)$ there are additional maxima at

$$\Delta\nu_\pm=\mp(1\pm\alpha)/2\beta. \tag{9}$$

If $|\Delta\nu_\pm|<\Delta\nu_0$ (where $\Delta\nu_0$ is the width of the distribution in frequency for the central component of the multiplet), the intensities of these maxima can turn out to be larger than at the center of the distribution (at $\nu=\nu_{n0}\pm\nu_{Q0}$). In particular, if such a maximum turns out to be on the right-hand edge of one of the outer lines, the NMR spectrum takes the form shown in Fig. 4.

The proposed mechanism can thus explain the quadrupole-splitting nonequidistance if it is of the order of the inhomogeneous width $\Delta\nu_0$. Unfortunately, we are as yet unable to point to the concrete microscopic mechanism that ensures a relation of the form (5).

2. Dependence of the transverse-relaxation rate on the number of the quadrupole-multiplet component.

In the analysis of this dependence we start from the known expression[9]:

$$T_2^{-1}=(2T_1)^{-1}+T_2'^{-1}, \tag{10}$$

where the term $T_2'^{-1}$ is due to interactions that make no contribution to the spin-lattice relaxation rate T_1^{-1}. It appears that the most effective of them is the indirect interaction via spin waves.[10] The corresponding value of $T_2'^{-1}$ was calculated in Ref. 11, but without allowance for the quadrupole splitting. At a large quadrupole splitting, as in our case, a contribution to the relaxation will be made by the interaction of the vibrations of nuclear spins belonging to only one line of the quadrupole multiplet. With this circumstance taken into account, the rates $T_2'^{-1}$ for the different multiplet components turn out to be different:

$$T_{2m}'^{-1}=[I(I+1)-m(m+1)]f(\nu_{n,m})\Lambda, \tag{11}$$

where for our case $I=3/2$ and m takes on values $1/2$, $-1/2$, and $-3/2$; the quantity Λ depends on the parameters of the spin-wave spectrum and on the hyperfine-interaction constant, but does not depend on m; $f(\nu_{n,m})$ is the spin distribution function in frequency and describes the NMR line shape of the corresponding quadrupole component. At the maximum, the value of $f(\nu_{nm})$ is of the order of $\Delta\nu_m^{-1}$, where $\Delta\nu_m$ is the line width and is determined for the central component ($m=-1/2$) by the inhomogeneities of the frequency ν_n (i.e., $\Delta\nu_{-1/2}=\Delta\nu_0$), while for the outer components it depends also on the inhomogeneities of the quadrupole frequency ν_Q (i.e., $\Delta\nu_{1/2}=\Delta\nu_{-3/2}=\Delta\nu_0+\Delta\nu_Q$). As a result we obtain for the ratio of the relaxation rates of the central and outer components[1)]

$$T_{2,-1/2}'^{-1}/T_{2,1/2}'^{-1}\approx{}^4/_3(\Delta\nu_0+\Delta\nu_Q)/\Delta\nu_0. \tag{12}$$

The factor 4/3 is due to the first m-dependent factor in (11). It can be seen from Fig. 1 that this factor can account for the observed difference between the rates of the central and outer components only up to 6 K. At $T=7$ K the experimental value of the ratio (12) is approximately 2.5, a fact that can be attributed within the framework of the given scheme to the faster increase of $\Delta\nu_Q$ (compared with $\Delta\nu_0$) as the phase transition point is approached.

3. Relaxation in the **WF** *phase near the Morin phase transition*

In contrast to the AF phase, we observed in the WF no drastic increase of the relaxation rates T_1^{-1} and T_2^{-1} as the phase transition was approached in the investigated temperature region (Fig. 3). Nonetheless, the NMR signal decreases near the transition temperature also from the WF-phase side, so that we could observe it only at $T>37$ K. This may possibly also be due to the increase of the transverse relaxation rate, but on account of a specific mechanism that makes no contribution to T_2 measured by the standard two-pulse echo procedure. We shall therefore call this relaxation anomalous and denote the corresponding time by T_{2a}. The existence of such an anomalous relaxation mechanism was

indicated earlier in Ref. 12, and Tsifrinovich and Krasnov[13] succeeded in describing it quantitatively.

In view of the unusual nature of the properties of the anomalous relaxation, a more detailed examination of this question is in order. The onset of the anomalous relaxation is due to the micro-inhomogeneous broadening of the NMR line (i.e., to the inhomogeneities of the hyperfine fields at nuclei with characteristic dimensions of the order of the interatomic distances in the crystal) and to the dynamic frequency shift (DFS).[9] It is known that in the absence of a DFS such a broadening leads to reversible decay of the transverse component of the nuclear magnetization, and this decay causes the damping of the free-induction signal after the exciting pulse. It turned out that the DFS prevents this decay, causing it to slow down, and most importantly, making it irreversible in time, similar to a viscous relaxation process.

The cause of this irreversibility can be easily understood by recognizing that the DFS gives rise to nuclear spin waves, or oscillations of the nuclear magnetization, whose frequencies depend on the wave vector q. Indeed, since the decay of the transverse component of the nuclear magnetization can be regarded as a transformation of homogeneous oscillations (nuclear spin waves with $q=0$) into inhomogeneous ones (nuclear spin waves with $q\neq 0$), such a transition in the presence of DFS is connected with the change of the frequency and is consequently forbidden by the energy conservation law. This process is allowed when account is taken of the influence of the thermostat, but becomes irreversible in time.

The anomalous relaxation process has a number of features that can explain qualitatively the observed behavior of the echo signals in the WF case. These features are the following.

1. The main feature of the anomalous relaxation (the impossibility of investigating it by the standard two-pulse-echo procedure) is due to the fact that the frequencies of the nuclear spin waves cease to depend on q at $q > r_{SN}^{-1}$, where r_{SN} is the radius of the Suhl-Nakamura interaction [2)] with which the existence of the DFS is connected.[9] The anomalous relaxation is thus turned off as soon as the transverse component of the nuclear magnetization vanishes in a volume with dimensions of the order of r_{SN}, since the subsequent dephasing of the nuclear spins will no longer be connected with the change of the oscillation frequency. In practice, however, it can be assumed that this turning-off takes place after the vanishing of the transverse component of the nuclear magnetic moment of the entire sample, i.e., within a time T_2^* after application of the exciting pulse. In other words, the anomalous relaxation exists only in those time intervals during which the free-induction signal exists. Since the procedure of measuring the time T_2 by using a two-pulse echo is based on measuring the dependence of the intensity of the echo signal on the delay time τ between the pulse precisely at $\tau > T_2^*$, it is impossible to measure the time T_{2a} of the anomalous relaxation by this method. In other words, with increasing rate T_{2a}^{-1} of the anomalous relaxation the echo signal will decrease, but at $\tau > T_2^*$ this decrease ceases to depend on τ, and this corresponds to the picture we observed experimentally for the WF phase.

2. The anomalous-relaxation rate T_{2a}^{-1} first increases with increasing DFS and reaches a maximum at $\nu_{DFS} = \Delta\nu_0$, where $\Delta\nu_0$ is the inhomogeneous broadening. Further increase of ν_{DFS} suppresses the inhomogeneous broadening (this is discussed in detail in Refs. 12 and 13), and therefore T_{2a}^{-1} begins to decrease. It becomes possible therefore to observe echo signals at large DFS.[13-15]

3. Since ν_{DFS} is proportional to the sample's magnetic susceptibility which increases as the transition point is approached, the rate T_{2a}^{-1} of the anomalous relaxation should also increase. However, since ν_{DFS} in the AF phase is smaller by two or three orders than in the WF phase, the influence of the anomalous relaxation is most likely not to manifest itself in the AF phase.

It can be easily seen that the foregoing features of the anomalous relaxation conform qualitatively to the observed behavior of the echo near the Morin phase transition.

APPENDIX

The state density at the frequency ν, i.e., the number ν of nuclear spins with an NMR frequency, should by definition be described by an expression of the form

$$n_m(\nu) = n_0 \int d\mathbf{r}\,\delta(\nu_{nm}(\mathbf{r}) - \nu), \tag{A.1}$$

where $\nu_{nm}(\mathbf{r})$ is the NMR frequency for the given quadrupole multiplet at the point $\mathbf{r}$, and n_0 is the number of spins per unit volume. Transforming in (A.1) from the variables $\mathbf{r}$ to new variables, one of which is the frequency inhomogeneity in (3), we obtain

$$n_m(\nu) = \int d(\Delta\nu) f(\Delta\nu)\,\delta(\nu_{nm}^{WF}(\Delta\nu) - \nu), \tag{A.2}$$

where $f(\Delta\nu)$ is the Jacobian of the transformation to the new variables, integrated over all the variables except $\Delta\nu$. The function $f(\Delta\nu)$ has the meaning of the spin distribution function over the inhomogeneities ΔH_n of the hyperfine field.

Eliminating the integration in (A.2) with the aid of a δ function and taking (6) into account, we obtain

$$n_{1/2}(\nu) = f(\Delta\nu)\,[d\nu_{n,1/2}^{WF}(\Delta\nu)/d(\Delta\nu)]^{-1}, \tag{A.3}$$

where $\Delta\nu$ are the roots of the equation

$$\beta\Delta\nu^2 + (1+\alpha)\Delta\nu + (\nu_{n0} + \nu_{Q0} - \nu) = 0. \tag{A.4}$$

Next,

$$n_{-1/2}(\nu) = f(\Delta\nu), \tag{A.5}$$

where $\Delta\nu$ is the root of the equation

$$\Delta\nu + (\nu_{n0} - \nu) = 0. \tag{A.6}$$

Finally,

$$n_{-1/2}(\nu) = f(\Delta\nu)\,[d\nu_{n,-1/2}^{WF}(\Delta\nu)/d(\Delta\nu)]^{-1}, \tag{A.7}$$

where $\Delta\nu$ are the roots of the equation

$$-\beta\Delta\nu^2 + (1-\alpha)\Delta\nu + (\nu_{n0} - \nu_{Q0} - \nu) = 0. \tag{A.8}$$

Thus, for all the components of the multiplet the quantity $\Delta\nu$

can be regarded as the detuning from the central frequency of the corresponding component

$$\Delta\nu_m=\nu-\nu_{nm}^{WF}(0); \tag{A.9}$$

and the expressions for the state densities of all three components can be written in the same form

$$n_m(\nu)=f(\Delta\nu_m)\,[d\nu_{nm}^{WF}(\Delta\nu_m)/d(\Delta\nu)]^{-1}, \tag{A.10}$$

which was indeed used to write expressions (8).

[1)]We neglect here the connection between the inhomogeneities of the hyperfine fields (3) and the inhomogeneities of the quadrupole splitting ν_Q (4), which was taken into account before for the WF phase, since no nonequidistance of the quadrupole splitting was observed experimentally in the AF phase. In this approximation, the quantities $\Delta\nu_0$ and $\Delta\nu_Q$ make an additive contribution to the resultant width of the outer components of the multiplet.

[2)]In easy-plane antiferromagnets r_{SN} reaches macroscopic dimensions of the order of $(10^3-10^4)a$, where a is the average distance between the atoms in the crystal.

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Translated by J. G. Adashko