Spin-flip and nuclear quadrupole interactions in the rare-earth orthochromites $GdCrO_3$

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(Submitted 21 January 1983)
Zh. Eksp. Teor. Fiz. **85**, 670–677 (August 1983)

A nuclear-magnetic-resonance investigation of the anisotropy of the magnetic hyperfine and nuclear quadrupole interactions in rare-earth orthochromites is carried out in the vicinity of the order-order type magnetic phase transition, with $GdCrO_3$ as the example. It is shown that the nuclear quadrupole interactions, along with the magnetic anisotropies of the hyperfine interactions, contribute to the splitting of the NMR lines of ^{53}Cr in the transition region. The Γ_2–Γ_4 transition in $GdCrO_3$ is interpreted as an anomalously narrow (width <0.1 K) second-order phase transition. The asymmetry of the quadrupole splitting of the NMR spectrum of ^{53}Cr in the Γ_4 phase is ascribed to effects of second order in the nuclear quadrupole interactions. Quantitative data are obtained on the components of the electric field gradient tensor.

PACS numbers: 76.60.Gv, 75.30.Kz, 75.30.Gw

By virtue of the local character of the information obtained, nuclear resonance methods, such as nuclear magnetic resonance (NMR) or nuclear gamma resonance (NGR), are undoubtedly of interest not only for the study of various hyperfine interactions, but also in the investigations of various distinguishing features of magnetically ordered crystals, and in particular spin-flip processes.

NMR investigation of such transitions in rare-earth orthoferrite has led to observation of an interesting effect, the violation of magnetic equivalence of Fe^{3+} ions from different sublattices in the transition region, as manifest by two values of the local field at ^{57}Fe nuclei.[1]

Even more interesting possibilities are provided by the rare-earth orthochromites $RCrO_3$. For the nuclei $^{53}Cr(I=3/2)$, in contrast to $^{57}Fe(I=1/2)$, nuclear quadrupole interactions take place in the ground state alongside the magnetic hyperfine interactions. This offers an opportunity of obtaining more extensive information from the NMR spectra in the spin-flip transition region.

Just as the orthoferrites $RFeO_3$, the orthochromites $RCrO_3$ have a distorted perovskite structure.[2] The magnetic ordering of their d-sublattice corresponds to three types of magnetic sublattice configuration[3]: $\Gamma_1(A_x, G_y, C_z)$, $\Gamma_2(F_x, C_y, G_z)$, $\Gamma_4(G_x, A_y, F_z)$, with ferromagnetism basis vectors **F** and antiferromagnetism vectors **G**, **C**, and **A**, wherein $F, C, A \ll G$.

1. FEATURES OF NUCLEAR QUADRUPOLE INTERACTIONS IN $RCrO_3$

The effective Hamiltonian of the nuclear subsystem in the presence of a local magnetic field and of quadrupole interactions, with account taken of the small anisotropy of the magnetic hyperfine interactions (HFI) will be expressed, in a coordinate frame in which the z axis is directed along the local field, in the form

$$H=-g_n\beta_n H_{л}^{(0)} I_z - g_n\beta_n \frac{a}{H_{л}^{(0)}}\Big(\sum_{ij} a_{ij}G_iG_j\Big) I_z + \frac{eQ}{4I(2I-1)}\Big(\sum_{ij} V_{ij}G_iG_j\Big)[3I_z^2-I(I+1)]; \qquad (1)$$

here $\mathbf{H}_L^0$ is the main isotropic part of the local field, with $\mathbf{H}_L^0 = a\mathbf{G}$, where $|a| = |\mathbf{H}_L^0|$ and **G** is the antiferromagnetism vector (we consider the largest "antiferomagnetic" contribution, inasmuch as in orthochromites we have $F, C, A \sim 10^{-2}G$, just as in orthoferrites[4]); the sum in the second term is the anisotropic part of H_L:

$$h_i = \sum_{i>j} a_{ij}G_j; \qquad (2)$$

g_n and β_n are respectively the nuclear g factor and the nuclear magneton, Q is the quadrupole moment of the nucleus, and V_{ij} are the components of the electric-field-gradient tensor. The signs of the tensor components a_{ij} and V_{ij} (see Tables I and II) can in general be different for different positions of the ^{53}Cr nuclei. It can be easily seen that the relation of the signs is such that in the configurations $\Gamma_1, \Gamma_2, \Gamma_4$, the parameters of the spin Hamiltonian (1) are the same for all four positions of the nuclei, i.e., in pure magnetic configurations the ^{53}Cr nuclei in the orthochromites are equivalent in the sense of both the local fields and the electric field gradients.

TABLE I. Relative signs of the tensor components a_{ij} and of the constant a for different positions of the Cr^{3+} ion.

Position of Cr^{3+} ion	Coordinates	a, a_{ii}	a_{xy}, $_{yx}$	a_{xz}, $_{zx}$	a_{yz}, $_{zy}$
1	$(0,{}^1/_2,0)$	+	+	+	+
2	$(0,{}^1/_2,{}^1/_2)$	−	−	+	+
3	$({}^1/_2,0,{}^1/_2)$	+	−	+	−
4	$({}^1/_2,0,0)$	−	+	+	−

0038-5646/83/080390-05$04.00

TABLE II. Relative signs of the tensor components V_{ij} for different positions of the ^{53}Cr nuclei

Position of ion (nucleus)	V_{ii}	$V_{xy,\,yx}$	$V_{xz,\,zx}$	$V_{yz,\,zy}$
1	+	+	+	+
2	+	+	−	−
3	+	−	+	−
4	+	−	−	+

The situation changes in the mixed configurations Γ_{12}, Γ_{24}, Γ_{14} realized as intermediate in the region of smooth transitions. For example in the case of the spin flip $\Gamma_2 \leftrightarrow \Gamma_4$, introducing the angle θ that defines the orientation of the vector **G** relative to the c axis, we write H in the form

$$H=-g_n\beta_nH_L^{(0)}I_z+g_n\beta_n(a_{xx}\sin^2\theta+a_{zz}\cos^2\theta\pm a_{xz}\sin 2\theta)I_z$$

$$+\frac{eQ}{4I(2I-1)}(V_{xx}\sin^2\theta+V_{zz}\cos^2\theta\pm V_{xz}\sin 2\theta)$$

$$\times[3I_z^2-I(I+1)], \tag{3}$$

where the + and − signs pertain to the nuclei in positions 1, 3 and 2, 4, respectively.

The Cr^{3+} ions in orthochromites are thus nonequivalent in the transition region both magnetically and electrically. This nonequivalence manifests itself in differences both between the local magnetic fields and between the electric field gradients at nuclei from different sublattices. This phenomenon is revealed by the special character of the line splitting in the NMR spectrum of ^{53}Cr.

The energy of the transitions $|3/2M\rangle \rightarrow |3/2M'\rangle$ in the ^{53}Cr takes of the magnetic configurations Γ_2, Γ_4, Γ_{24} the form

$$\Delta E(M\rightarrow M')=g_n\beta_nH_L^{(0)}(M'-M)$$

$$\times\left[1-\frac{1}{H_L^{(0)}}(a_{xx}\sin^2\theta+a_{zz}\cos^2\theta\pm a_{xz}\sin 2\theta)\right]$$

$$+{}^1/_2(v_{xx}\sin^2\theta+v_{zz}\cos^2\theta\pm v_{xz}\sin 2\theta)(M'^2-M^2), \tag{4}$$

where we have introduced the quantities

$$v_{ij}=3eQV_{ij}/2I(2I-1)={}^1/_2eQV_{ij}.$$

Three lines with intensity ratio 3:4:3 will be observed in the NMR spectrum of the NMR in the configurations Γ_2 and Γ_4, namely: $3/2\rightarrow 1/2$; $1/2\rightarrow -1/2$; $-1/2\rightarrow -3/2$. In the region of the transition (configuration Γ_{24}) a splitting of the NMR lines will be observed. The distance between the outer lines in the NMR spectrum for each type of ^{53}Cr nucleus is determined by the quadrupole parameter

$$v_{ac}=|v_{xx}\sin^2\theta+v_{zz}\cos^2\theta\pm v_{xz}\sin 2\theta|. \tag{5}$$

The splitting of the line $3/2M\rightarrow 3/2M'$ will take the form

$$\delta E(M\rightarrow M')=\Delta E_{1,3}(M\rightarrow M')-\Delta E_{2,4}(M\rightarrow M')$$

$$=\left[-\Delta\frac{2a_{xz}}{H_L^{(0)}}+v_{xz}(M'^2-M^2)\right]\sin 2\theta, \tag{6}$$

or

$$\delta E\left(\frac{3}{2}\rightarrow\frac{1}{2}\right)=\left\{-\Delta\frac{2a_{xz}}{H_L^{(0)}}-2v_{xz}\right\}\sin 2\theta.$$

$$\delta E\left(\frac{1}{2}\rightarrow-\frac{1}{2}\right)=-\Delta\frac{2a_{xz}}{H_L^{(0)}}\sin 2\theta, \tag{7}$$

$$\delta E\left(-\frac{1}{2}\rightarrow-\frac{3}{2}\right)=\left\{-\Delta\frac{2a_{xz}}{H_L^{(0)}}+2v_{xz}\right\}\sin 2\theta,$$

where $\Delta=g_n\beta_nH^{(0)}$ is the energy of the transition $1/2\rightarrow -1/2$ without allowance for the small effects of the HFI anisotropy.

The splitting of the outer lines in the NMR spectrum of ^{53}Cr for orthochromites in the Γ_2–Γ_4 transition region will thus be determined by the sum or difference of the contributions of the magnetic and electric nonequivalences of the nuclei, whereas the splitting of the central line is determined only by the anisotropy of the magnetic properties of the HFI.

We have considered above the effects of nuclear quadrupole interactions in first-order perturbation-theory approximation, assuming them naturally to be small compared with the magnetic HFI. Allowance for the nuclear quadrupole interaction in second-order perturbation theory shifts the ^{53}Cr NMR lines. In the coordinate frame where the axis $z\|H_L$ we have

$$\varepsilon_1=\varepsilon_3=\frac{1}{9\Delta}(v_{yz}^2+v_{xz}^2),$$

$$\varepsilon_2=\frac{1}{9\Delta}\left[\frac{1}{2}v_{xy}^2-v_{xz}^2-v_{yz}^2+\frac{1}{4}(v_{xx}-v_{yy})^2\right], \tag{8}$$

where ε_1 and ε_3 are the shifts of the outer lines of the spectrum and ε_2 is the shift of the central line. Thus, at $\varepsilon_{1,3}\neq\varepsilon_2$ the second-order effects lead to nonequidistance in the NMR spectrum.

2. NMR SPECTRUM OF ^{53}Cr IN THE ORTHOCHROMITE GdCrO$_3$

As a system convenient for the analysis of the NMR spectrum of ^{53}Cr in the region of the Γ_4–Γ_2 transition we chose the orthochromite GdCrO$_3$. In this compound, according to Japanese data,[5,6] the Γ_4–Γ_2 transition has a smooth character in the temperature region 4–10 K. This makes it possible to track clearly the manifestations of the magnetic and electric nonequivalences of the Cr^{3+} ions and obtain abundant information on the anisotropy parameters of the HFI and of the quadrupole interactions.

Actually, however, the situation turned out to be less favorable. Figure 1 shows the NMR spectrum of ^{53}Cr in GdCrO$_3$ in the temperature region 4–10 K, obtained for single crystals with natural Cr isotope content. Both the low-temperature (LT) and the high-temperature (HT) phase contain all three lines in accord with the spin $I=3/2$ of the ^{53}Cr nucleus. The lines are 120 kHz wide and are well resolved. The frequency-determination error does not exceed ± 25 kHz. The quadrupole splitting in the LT phase is 1100 kHz and is symmetric, and in the HT phase it is asymmetric, with 510 kHz between the upper and central lines and 440 kHz between the central and lower lines. At $T=7$ K an abrupt

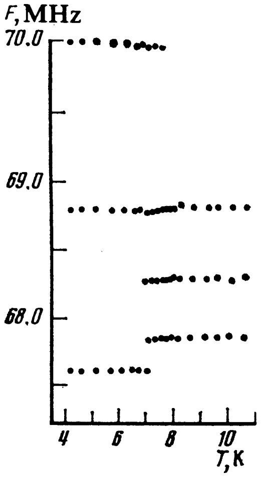

FIG. 1. Temperature dependence of the NMR frequencies of ^{53}Cr in GdCrO$_3$ in the vicinity of the phase transition.

phase transition is observed, revealed by the jump of the resonance frequencies. The phase-transition region (if it exists at all) does not exceed 0.05 K (the accuracy with which the temperature is determined).

The spectrum has the following characteristic features:

1) No increase of the NMR signal gain is observed at the transition point 7 K, unlike in second-order phase transitions (e.g., Ref. 1).

2) There is no pronounced region of coexistence of two phases in the transition region, unlike in the NMR investigation of a first-order transition.[7] The lower line has no coexistence region (at least to within the experimental accuracy 0.05 K). The upper line of the LT phase penetrates about 0.2 K into the LT phase region. As for the central line, nothing can be said about its coexistence region because, most likely by accident, it merges with the upper line of the HT phase.

3) In the temperature interval 7.1–7.7 K at frequencies 69.1 and 67.3 MHz, lines were observed of intensity not lower than of the fundamental lines, and apparently due to signals from nuclei within the boundaries.

3. DISCUSSION OF RESULTS

Since the spin of the Cr^{3+} ion is $S = 3/2$, the cubic anisotropy, meaning also the second magnetic-anisotropy constant of the Cr sublattice in GdCrO$_3$, vanishes. Within the framework of the usual spin-flip theory, 4 based on allowance for the temperature-dependent contribution of the R sublattice to the first anisotropy constant, we might expect in this case for GdCrO$_3$ a first-order $G_x \rightarrow G_z$ phase transition.

A more detailed theoretical analysis of the $\Gamma_4 \rightarrow \Gamma_2$ transition in GdCrO$_3$ (see the Appendix) shows that this transition takes place nevertheless via a smooth rotation of the spins, but in a very narrow temperature interval. Indeed, according to the data of Ref. 8 we have $H_c = 5.5$ kOe, meaning $\mu_{\text{in}} \approx 1.1\ \text{K}/T_c$. The experimental data of the present paper allow us to state that $T_c \gtrsim 7$ K, whence $\mu_{\text{in}} \lesssim 0.16$. Consequently, the temperature of the start of the transition differs from T_c by less than 1 K, and a good approximation is $T_c \approx 7$–8 K. To find μ_{fin} we use Eq. (A.4) for T_c and the known value of the first anisotropy constant of the Cr sublattice in YCrO$_3$, which is close in structure to GdCrO$_3$; this value is[9] $K_1 = 0.02\ \text{cm}^{-1}/\text{ion}$, yielding $H_q \approx 6.3$ kOe and $\mu_{\text{fin}} \approx 1.3\ \text{K}/T_c \approx 0.19$. So small a difference between μ_{fin} and μ_{in} (namely $\mu_{\text{fin}} - \mu_{\text{in}} = 0.2/T_c \approx 0.03$) with their absolute values small corresponds to an anomalously narrow temperature interval of the spin-flip transition: $\Delta T = T_{\text{in}} - T_{\text{fin}} \approx 0.02$ K. Such a phase transition will seem jumplike in experiment, although the main attributes of a first-order transition (the presence of a phase-coexistence region, hysteresis phenomena) will be missing.

The presented interpretation of the transition in GdCrO$_3$ is based on a simple albeit quite reasonable model. We have disregarded such facts as the presence of a weak initial splitting for the Gd^{3+} ions, due to the action of the crystal field, the existence of exchange and magnetodipole interactions between the Gd^{3+} ions, and finally the existence of a small second anisotropy constant for the Cr sublattice, connected in particular with the magnetoelastic interactions. At the same time the smallness of the anisotropy constant K_1 makes it important to take into account the shape of the sample and the inhomogeneity of the constant K_1, due in particular to the surface anisotropy. Finally, the presence of a small amount of impurity ($< 1\%$) can also influence substantially the character of the spin-flip transition. This is not surprising if it is recognized that even replacement of the Cr^{3+} by nonmagnetic ions can lead to the appearance of very strong ($\sim 10^4$–10^5 Oe) magnetic fields at the Gd^{3+} ions in accord with the theory of the gigantic influence of magnetic vacancies.[3] It is possible that it is just this influence of the shape and purity of the sample that causes the differences between the data of different workers[5,8] on the character of the transition as well as on the compensation temperature in GdCrO$_3$.

TABLE III. Values of the electric-field-gradient components at the ^{53}Cr nuclei in RCrO$_3$ in the model of pointlike charges (in MHz).

RCrO$_3$	v_{zz}	v_{xx}	v_{yy}	v_{xz}	v_{yz}	v_{xy}
NdCrO$_3$	1.35	−0.32	−1.03	−1.54	−0.15	0.62
GdCrO$^*_3$	1.10	−0.47	−0.63	$v_{xz}^2+0.75$	$v_{yz}^2 \approx 2.2$	$\lvert v_{xy} \rvert \approx 3$
TbCrO$_3$	0.37	−0.40	0.03	0.66	0.40	0.37
DyCrO$_3$	0.00	−0.15	0.15	0.58	0.58	0.01
ErCrO$_3$	−0.58	−0.17	0.75	1.14	0.70	−0.48

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*These values were obtained from experiment.

The experimental conclusions of Ref. 5, which attest to a broad region of the transition (≈ 6 K) are apparently due to the use of an external magnetic field in the measurements of the magnetization; this could indeed distort the information on the character of the transition. Incidently, in Ref. 8 they drew a more realistic conclusion concerning the transition width, $\Delta T = T_1 - T_2 \sim 1$ K.

The information obtained from NMR investigations of $GdCrO_3$ on the anisotropy of the HFI and on the quadrupole interactions remains somewhat limited for lack of data directly in the spin-flip transition region. We note that the observed shift of the central line in the transition in $GdCrO_3$ (Fig. 1) is evidence of a substantial anisotropy of the HFI for the ^{53}Cr nuclei, namely $|a_{xx} - a_{zz}| \approx 0.5$ MHz.

The symmetric quadrupole splitting in the Γ_2 phase of $GdCrO_3$ attests to approximate equality of the contributions of second order to the shifts of the outer and central lines of the spectrum. In a coordinate frame where $\mathbf{H}_L \| c$ we have

$$\varepsilon_1 = \varepsilon_3 \approx \varepsilon_2,$$

which allows us to establish an important relation between the different components of the electric-field-gradient tensor:

$$2(v_{yz}^2 + v_{xz}^2) \approx {}^1/_2 v_{xy}^2 + {}^1/_4 (v_{xx} - v_{yy})^2. \tag{9}$$

In the Γ_4 phase, where $\mathbf{H}_L \| a$, the second-order shifts will be expressed in terms of tensor components v_{ij} specified in the *abc* coordinate frame in the following manner:

$$\varepsilon_1 = \varepsilon_3 = \frac{1}{9\Delta}(v_{xz}^2 + v_{xy}^2),$$

$$\varepsilon_2 = \frac{1}{9\Delta}\left\{\frac{1}{2} v_{yz}^2 - v_{xy}^2 - v_{xz}^2 + \frac{1}{4}(v_{yy} - v_{zz})^2\right\}. \tag{10}$$

If the signs of $\varepsilon_1 = \varepsilon_3$ and ε_2 in the Γ_4 phase are assumed to be different, the use of the experimental data yields

$$\varepsilon_1 - \varepsilon_2 \approx 35\,\text{kHz}.$$

From the experiment we obtain also the absolute values of the diagonal components

$$|v_{xx}| = 475\,\text{kHz}, \quad |v_{zz}| = 1100\,\text{kHz}.$$

Information on the most probable sign of these quantities can be obtained by calculating them within the framework of the model of pointlike charges. Unfortunately, for $GdCrO_3$ there are no complete crystallographic data that makes such a calculation possible. In Table II we list the calculated values of the tensor components v_{ij} for a number of orthochromites $RCrO_3$ with known crystallographic data. We have used here the values of the Sternheimer factor and of the quadrupole moment of ^{53}Cr, given in Ref. 10.

Taking into account the position of $GdCrO_3$ in the orthochromite series and the regularity in the variation of the calculated values of v_{ij} in the $RCrO_3$ series, which is approximately observed in Table II. We specify the following signs: $v_{xx} \approx +0.5$ MHz, $v_{zz} \approx -1.1$ MHz, meaning thus $v_{yy} = +0.6$. From (9) and (10) we then obtain

$$10 v_{xz}^2 + 7.5 v_{yz}^2 = 22\,\text{MHz}^2 \quad \text{and} \quad |v_{xy}| \simeq 3\,\text{MHz}.$$

whereas the first of these relations does not contradict the ordering of the values of v_{xz} and v_{yz} in the $RCrO_3$ series, our value of the component v_{xy} is substantially larger than than predicted within the simple pointlike-charge model. The possible reason is the appreciable contribution made to the electric field gradient at the ^{53}Cr nucleus by the covalency and the Cr^{3+}–O^{2-} overlap.

4. CONCLUSIONS

The nuclear quadrupole interactions contribute alongside the anisotopic magnetic HFI to the splitting of the NMR lines of ^{55}Cr in orthochromites in the spin-flip transition region. The physical nature of this phenomenon is connected with manifestation, in the transition region, of both magnetic and electric nonequivalence of nuclei from different sublattices.

NMR Investigation of the orthochromite $GdCrO_3$ has shown that in contrast to the conclusions of earlier studies, the $\Gamma_4 \leftrightarrow \Gamma_2$ transition in $GdCrO_3$ is realized in a very narrow temperature interval, < 0.1 K. A theoretical analysis of the $\Gamma_4 \leftrightarrow \Gamma_2$ transition in $GdCrO_3$ allows us to interpret it as an anomalously narrow second-order phase transition.

The asymmetry of the quadrupole spectrum of the NMR spectrum of ^{53}Cr in the Γ_4 phase of $GdCrO_3$ is due to effects of second order in the nuclear quadrupole interactions. An analysis of the experimental data with account taken of the components of the electric field gradient, calculated within the framework of the pointlike model, yielded quantitative information on all the components of this tensor.

APPENDIX

With the second anisotropy constant of the Cr sublattice ($S_{Cr} = 3/2$ equal to zero, the free anisotropy energy of $GdCrO_3$ in the *ac* plane can be represented in the form

$$\Phi_{\text{an}} = K_1 \cos 2\theta - kT \ln \sum_{m=-J}^{+J} \exp\left(-\frac{g\mu_B m H(\theta)}{kT}\right), \tag{A.1}$$

$$H(\theta) = [H_c^2 \sin^2\theta + H_a^2 \cos^2\theta]^{1/2},$$

where $H(\theta)$ is the effective field produced at the S ions of Gd^{3+} by the Cr sublattice; $J = 7/2$ is the spin of the Gd^{3+} ion.

The condition that Φ_{an} be a minimum yields several possible states of the system: 1) $\sin 2\theta = 0$, i.e., $\theta = 0$ or π, or else $\pm\pi/2$—pure Γ_2 and Γ_4 configurations. 2) The angle θ is determined from the equation

$$\mu = B_J\left(\frac{3J}{J+1}\frac{\mu}{\tau}\right), \tag{A.2}$$

where B_J is a Brillouin function and

$$\mu = -\frac{4K_1 H(\theta)}{g\mu_B J(H_c^2 - H_a^2)}, \quad \tau = \frac{T}{T_c},$$

$$T_c = -\frac{g^2\mu_B^2 J(J+1)(H_c^2 - H_a^2)}{12kK_1} \tag{A.3}$$

with $H_c = H(\pi/2)$ and $H_a = H(0)$. This corresponds to the spin-flip region. The equation for the determination of $\theta(T)$ in the transition region takes the form of the equation, known from molecular-field theory, for the reduced magnetization of a ferromagnet.

The beginning and end of the transition are determined from Eq. (A.2) at

$$\mu_{\text{in}} = \frac{g\mu_B H_c(J+1)}{3kT_c}, \quad \mu_{\text{fin}} = \frac{g\mu_B H_a(J+1)}{3kT_c}. \tag{A.4}$$

Here, just as in (A.1), H_c and H_a are the effective fields for the Gd^{3+} ion in the phases Γ_4 and Γ_2, respectively.

In practice the transition region in $GdCrO_3$ is best analyzed within the framework of the model presented graphically. It suffices to find the points corresponding to μ_{in} and μ_{fin} on the plot of the solution of Eq. (A.2).

The abscissas of these points will correspond to the temperatures of the beginning and the end of the spin flip.

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Translated by J. G. Adashko