



Transformation of ${}^5I_8 \rightarrow {}^5F_5$ Ho absorption band in $\text{Ho}_{0.75}\text{Nd}_{0.25}\text{Fe}_3(\text{BO}_3)_4$ crystal during spin-reorientation transition

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ABSTRACT

Polarized absorption spectra of crystal $\text{Ho}_{0.75}\text{Nd}_{0.25}\text{Fe}_3(\text{BO}_3)_4$ in the region of the ${}^5I_8 \rightarrow {}^5F_5$ absorption band of Ho ions were investigated depending on temperature in the range of 5–20 K. Absorption spectra are decomposed into components of the Lorentz form. The spin-reorientation phase transition was detected at 6.9 K. The observed line position shifts during the reorientation transition were different for different lines, which indicated different local magnetic anisotropy of the Ho ion in its excited states. Significant changes in absorption line intensities as a function of temperature were observed during the reorientation transition and above. These were explained as a consequence of the change in even components of the local crystal field in excited states. These components mix the closely spaced $4f$ states. Distortion of local symmetry in the ground state was detected at one of the electron transitions.

1. Introduction

Ferroborate $\text{Ho}_{0.75}\text{Nd}_{0.25}\text{Fe}_3(\text{BO}_3)_4$ crystallizes into a trigonal structure with space symmetry $R32$ and undergoes a phase transition with a decrease in symmetry to $P3_121$ (D_3^4) with a decrease in temperature [1, 2]. There is a structural phase transition of the displacement type with a soft structural mode in the low-temperature phase at a temperature of $T_C = 203$ K [2]. In $\text{HoFe}_3(\text{BO}_3)_4$ such a transition occurs at 360 K [3]. Ho^{3+} ions occupy positions of the same type with symmetry D_3 in phase $R32$ and C_2 positions in low-temperature phase $P3_121$. Raman scattering spectra were studied in the works [2,4,5]. Crystal $\text{Ho}_{0.75}\text{Nd}_{0.25}\text{Fe}_3(\text{BO}_3)_4$ refers to multiferroics as well as other holmium containing ferrobates [1]. Below 32 K it becomes an easy-plane antiferromagnet [1]. In $\text{HoFe}_3(\text{BO}_3)_4$ $T_N = 38$ K [6], that testifies to a decrease of the Fe–Fe exchange interaction in $\text{Ho}_{0.75}\text{Nd}_{0.25}\text{Fe}_3(\text{BO}_3)_4$ as a result of change of the crystal structure. Magnetoelectric properties have been studied in detail only in crystals $\text{HoFe}_3(\text{BO}_3)_4$, $\text{Ho}_{0.25}\text{Nd}_{0.75}\text{Fe}_3(\text{BO}_3)_4$ and $\text{Ho}_{0.5}\text{Nd}_{0.5}\text{Fe}_3(\text{BO}_3)_4$ [7,8].

A large number of works are devoted to the optical spectra of ferrobates. Let's limit ourselves to crystal $\text{HoFe}_3(\text{BO}_3)_4$. In particular, work [3] investigated high-resolution infrared spectra as a function of temperature. Polarized absorption spectra of single crystal $\text{HoFe}_3(\text{BO}_3)_4$ in the range 8500–24500 cm^{-1} were studied as a function of

temperature, starting from 2 K, in Ref. [9]. Noticeable changes in the absorption spectra of $\text{HoFe}_3(\text{BO}_3)_4$ in the temperature region of the reorientation phase transition were observed in Ref. [9]. Change of absorption spectra of $\text{HoFe}_3(\text{BO}_3)_4$ in excited 5F_5 state of Ho^{3+} ions depending on magnetic field was studied in work [10]. The magnetic linear dichroism of crystal $\text{HoFe}_3(\text{BO}_3)_4$ as a function of the magnetic field was studied in Ref. [11]. High resolution optical spectra and neutron diffraction of crystal $\text{HoFe}_3(\text{BO}_3)_4$ were studied in Ref. [12]. (Comments on some statements of this work will be presented in the main part of this article).

The magnetic, magnetoelectric and structural properties of pure holmium ferrobate $\text{HoFe}_3(\text{BO}_3)_4$ [3,6,7] and mixed ferrobates with additives Nd [1,2,7,8] differ markedly. The same should be true for optical properties. Therefore, studies of the optical properties of mixed ferrobates are essential. The first study of optical absorption spectra of holmium in crystal $\text{Ho}_{0.75}\text{Nd}_{0.25}\text{Fe}_3(\text{BO}_3)_4$ was carried out in Ref. [13] and Judd-Ofelt parameters were determined. The study of the optical properties of crystal $\text{Ho}_{0.75}\text{Nd}_{0.25}\text{Fe}_3(\text{BO}_3)_4$ is only at the very beginning. Here we present the first study of changes in the absorption spectrum of crystal $\text{Ho}_{0.75}\text{Nd}_{0.25}\text{Fe}_3(\text{BO}_3)_4$ as a function of temperature in the magnetic spin-reorientation phase transition region. These changes reflect changes in the interaction of Ho ions with the environment during electronic transitions and as a result of spin-reorientation phase

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transition.

2. Experimental details

Crystal $\text{Ho}_{0.75}\text{Nd}_{0.25}\text{Fe}_3(\text{BO}_3)_4$ is grown from a melt solution based on $\text{Bi}_2\text{Mo}_3\text{O}_{12}$ [2,14]. The stoichiometric composition of the grown crystals was determined by the ratio of rare earth oxides used in the synthesis. The measurement samples were prepared as ~ 0.2 mm thick plates with a crystallographic axis C_3 lying in the plane of the sample.

Absorption spectra were measured using an automated spectrophotometer based on a 2 \AA/mm linear dispersion DFS-8 diffraction spectrometer. The optical width of the slit was 0.2 \AA . Absorption spectra are obtained by propagating light perpendicular to the C_3 axis of the crystal for a light electric vector \vec{E} of a parallel (π spectrum) and perpendicular (σ spectrum) C_3 axis of the crystal. The sample was placed in optical flow helium cryostat LH-320-MAG. Temperature adjustment and stabilization was done with the LakeShore Model 325 temperature controller using the CX-1050-CU-HT-1.4 L temperature sensor.

3. Results and discussion

The polarized absorption spectra of the $^5I_8 \rightarrow ^5F_5$ transition were measured in the range of 5–20 K (see Figs. 1 and 2 for examples). The decomposition of spectra into separate Lorentz lines and their identification in the approximation of symmetry D_3 are shown in figures. A detailed study of the position of the D7 line depending on temperature (Fig. 3) made it possible to detect a spin-reorientation phase transition at $T_R = 6.9$ K. Spin-reorientation phase transition in $\text{HoFe}_3(\text{BO}_3)_4$ occurs at 4.7 K [3,6]. The spin-reorientation transition in the crystal occurs as a result of the competition of local magnetic anisotropies of the exchange-coupled ions of Ho and Fe, which have different signs and temperature dependencies. Therefore ratio of these ions has direct influence on the temperature of the spin-reorientation phase transition. Changing spectra with temperature (Fig. 3, insert) shows that two magnetic phases coexist in a certain temperature range. In particular, spectra at 6.8 and 6.9 K clearly contain spectra at 6.7 and 7 K. Thus, Figs. 1 and 2 show spectra below and above T_R .

The excited multiplet 5F_5 is split in the cubic crystal field, and then in the D_3 symmetry field as follows:

$$^5F_5 (J = 5) \rightarrow E + 2T_1 + T_2 \rightarrow E + 2(A_2 + E) + (A_1 + E) \quad (1)$$

Identification of the absorption spectra of $\text{HoFe}_3(\text{BO}_3)_4$ has already been made in work [9] on these spectra. It was shown that in the first

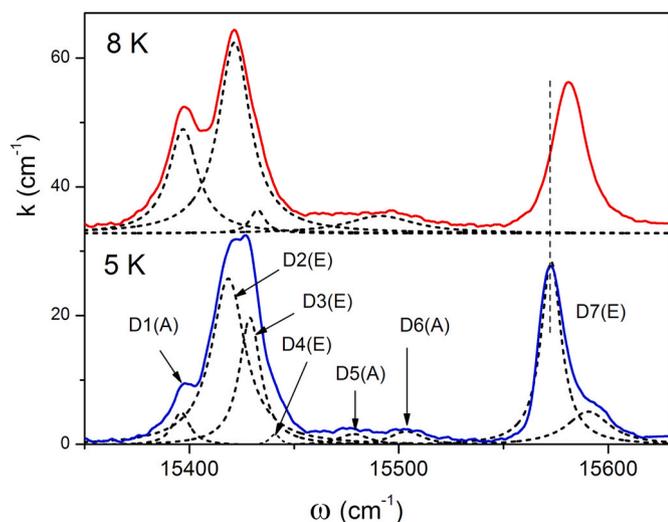


Fig. 1. π -polarized absorption spectra below (5 K) and above (8 K) temperature of the spin-reorientation transition.

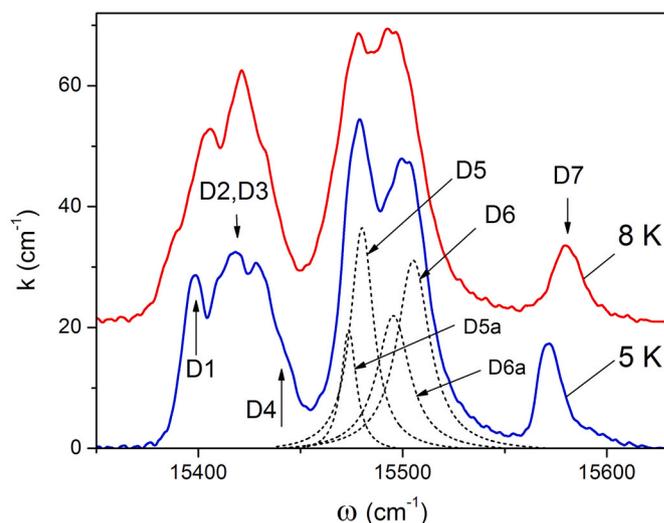


Fig. 2. σ -polarized absorption spectra below (5 K) and above (8 K) temperature of the spin-reorientation transition.

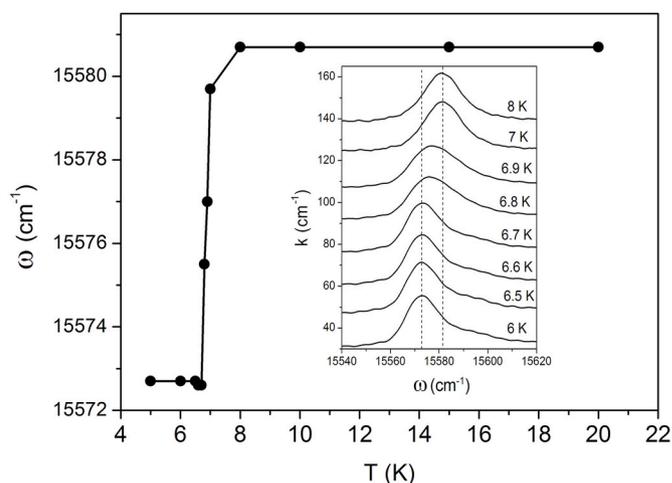


Fig. 3. Temperature dependence of π -polarized D7 line position. Insert: temperature transformation of the π -polarized D7 line spectrum.

approximation the polarizations of the absorption lines correspond to the selection rules in local symmetry D_3 (Table 1) on the assumption that the ground state has symmetry E . Lowering the symmetry to C_2 mainly leads to a small splitting of the absorption lines. However, recently in work [12] there was a statement that the ground state is singlet. Obviously, a singlet was implied in the approximation of symmetry D_3 , since in symmetry C_2 all states are singlet. Such a statement destroys all previous conceptions and conclusions regarding the absorption spectra and electronic structure of crystal $\text{HoFe}_3(\text{BO}_3)_4$. Therefore, we have to return to the problem of identifying the states and electronic transitions of the Ho^{3+} ion in $\text{HoFe}_3(\text{BO}_3)_4$ and further in crystal $\text{Ho}_{0.75}\text{Nd}_{0.25}\text{Fe}_3(\text{BO}_3)_4$.

Measurements of neutron diffraction in work [6], as well as in the

Table 1

Selection rules for electric dipole transitions in D_3 symmetry for integer moments.

	A_1	A_2	E
A_1	–	π	$\sigma(\alpha)$
A_2	π	–	$\sigma(\alpha)$
E	$\sigma(\alpha)$	$\sigma(\alpha)$	$\pi, \sigma(\alpha)$

discussed work [12] show that the magnetic moment of the Ho^{3+} ion in $\text{HoFe}_3(\text{BO}_3)_4$ is $\sim 5\mu_B$ at 2 K. How is it possible if the ground state is singlet, and the energy of the first excited state of the ground multiplet is 9 cm^{-1} [9]? Based on the magnetic moment of $5\mu_B$ and the exchange field Fe–Ho 25 kOe [15], the exchange splitting of the ground state $\Delta E = 11.7 \text{ cm}^{-1}$ [9] was obtained. Electronic transitions from the upper component of the exchange splitting of the ground state are observed rather rarely, since they occur with the reversal of the magnetic moment. By the way, the transition of the electron from the state with practically the specified energy was observed in $\text{HoFe}_3(\text{BO}_3)_4$ [9].

Now let's analyze the problem in terms of selection rules. Suppose the ground state is A_2 in symmetry D_3 . Then, for the electron transitions from the ground state A_2 to the multiplet (1), we obtain $4\sigma + \pi$ lines according to the selection rules of Table 1. For transitions from state A_1 , we get $4\sigma + 2\pi$ lines, and for transitions from state E: $4\pi\sigma + 3\sigma$ lines. The latter version fully corresponds to the absorption spectrum of the ${}^5I_8 \rightarrow {}^5F_5$ band of $\text{HoFe}_3(\text{BO}_3)_4$ in the easy axis state (see Ref. [10], Fig. 2), that is, in the first approximation, the spectrum is described in D_3 local symmetry with the ground state E, and the distortions C_2 are small. In particular, the D1, D5 and D6 absorption lines were σ -polarized and were therefore identified as $E \rightarrow A$ transitions. However, the D1 line shows peculiar behavior. Above the reorientation transition in $\text{HoFe}_3(\text{BO}_3)_4$, it splits and appears in the π -polarization [10], while lines D5 and D6 remain σ -polarized. Splitting of line D1 means splitting of the ground state since the excited state is singlet. This confirms the E type of ground state in the symmetry approximation D_3 . Splitting of the ground state of Ho^{3+} in $\text{HoFe}_3(\text{BO}_3)_4$ was also observed on the magnetic linear dichroism spectra of some electronic transitions [11].

In our crystal, the π -polarization of the D1 line already exists below T_R , but increases greatly at T_R (Fig. 1). The appearance of π -polarization indicates the appearance of local symmetry distortions in the ground state at the electronic transition D1, since the excited state is singlet. The described feature applies only to the electronic transition to the singlet state D1, but not to the electronic transitions to the singlet states D5 and D6, which remain practically σ -polarized both below and above T_R (Figs. 1 and 2). Thus, symmetry distortion is not static related to the entire crystal, but occurs locally only at the electronic transition D1 and occurs only in the ground state. Indeed, electronic transitions occur due to the mixing of the initial and final states by optical perturbation. As a result, both initial and final states change, and the interaction of the atom with the environment in both states changes. Therefore, local symmetry can also change in the initial state. The position of the atom, of course, does not change during the transition of the electron, but this position is not equilibrium for the changed states of the electron.

The absorption spectra of crystal $\text{Ho}_{0.75}\text{Nd}_{0.25}\text{Fe}_3(\text{BO}_3)_4$ were decomposed into components of the Lorentzian form (Figs. 1 and 2). In σ -polarized spectra, some splitting of electronic transitions in the region of singlet states D5 and D6 are observed (Fig. 2), but their π -polarized intensity is very small both before and after the spin-reorientation transition (Figs. 1 and 2), which indicates the preservation of local symmetry close to D_3 in the ground state. Therefore, said splitting is of a different nature. Apparently, D5a and D6a lines are due to transitions from excited sublevels of the ground multiplet.

Temperature relationships of absorption line intensities are shown in Fig. 4a and b. The present experiment showed that the overall intensity of band D varies very slightly with temperature. This means that the odd part of the crystal field that resolves electronic f - f transitions also weakly changes. All electronic transitions discussed occur from the ground state. Therefore, their intensities should gradually decrease with increasing temperature due to a decrease in the population of the ground state. On the contrary, the intensities of individual absorption lines vary significantly in different directions depending on temperature, especially during the spin-reorientation transition (Fig. 4a and b). This observation indicates distortions of the even part of the crystal field in excited electronic states. The distortions mix the near $4f$ electronic states having the same parity and change the probabilities of the

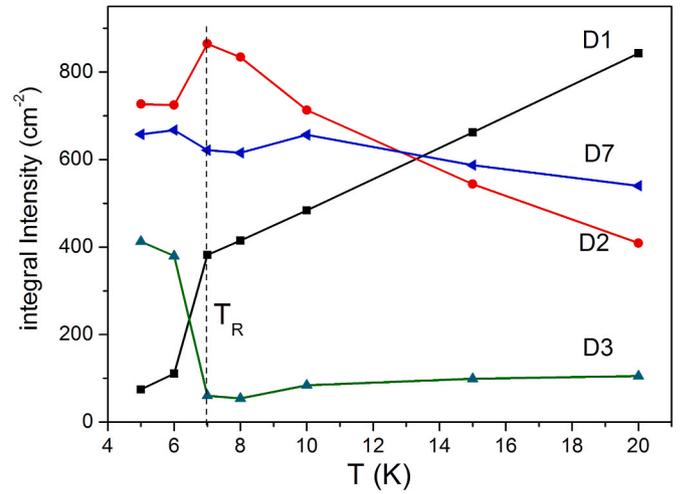


Fig. 4a. Integral intensities of absorption lines in π polarization.

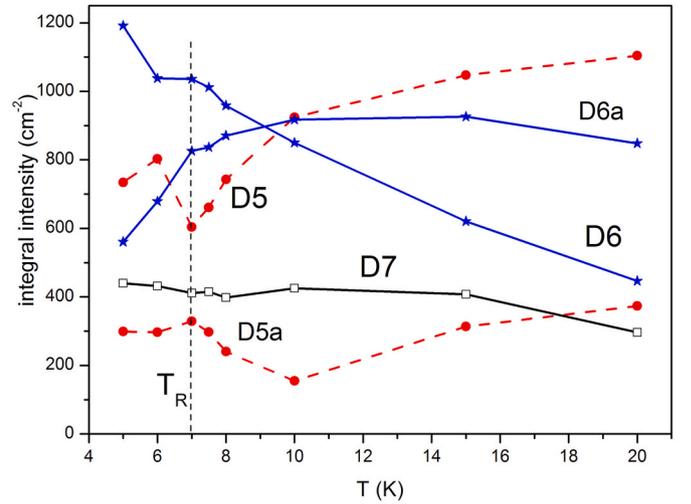


Fig. 4b. Integral intensities of absorption lines in σ polarization.

corresponding transitions. Indeed, it can be seen from Fig. 4a that the intensities of the close lines D1, D2 and D3 in the π -polarization change the most strongly, which indicates the continued distortion of the lattice above T_R , especially at the electronic transition D1. A similar phenomenon was observed in crystal $\text{HoFe}_3(\text{BO}_3)_4$ at electronic bands ${}^5I_8 \rightarrow {}^5F_2$, 5F_3 [16], but depending on the magnetic field. Work [17] shows the possibility of allowance of symmetrically forbidden electronic transitions in rare-earth ions by the exchange interaction iron - rare-earth element. However, we are dealing with symmetry allowed electronic transitions, and, moreover, their intensities do not correlate with the magnetization of holmium (see Fig. 4a and b).

Denote the difference in magnetic energy of Ho between the states $\mathbf{M}_{\text{Ho}}||ab$ and $\mathbf{M}_{\text{Ho}}||c$ (where \mathbf{M}_{Ho} is the average magnetic moment of the sublattice) as:

$$\Delta E = E(ab) - E(c). \quad (2)$$

The $\mathbf{M}_{\text{Ho}}||c$ state is energetically favorable for the Ho ion. The magnetic anisotropy energy of holmium (2) can have different values in the ground and excited states (Fig. 5). Electronic transitions occur between $\mathbf{M}_{\text{Ho}}||ab$ states below the reorientation transition (ω_1) and between $\mathbf{M}_{\text{Ho}}||ab$ states above the reorientation transition (ω_2) (Fig. 5). The change in these energies is determined by the change in the energy of the absorption line above and below the spin-reorientation transition:

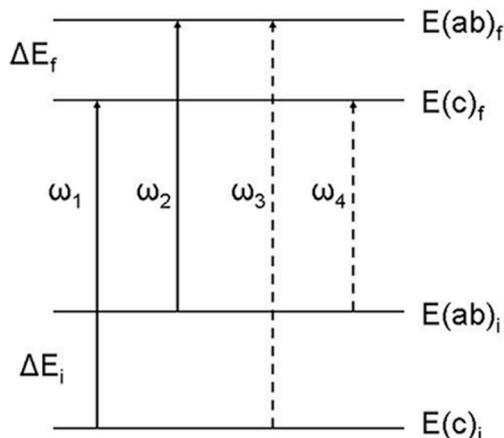


Fig. 5. Diagram of electron transitions before and after the spin-reorientation transition.

$$\Delta\omega = \omega_2 - \omega_1 = \Delta E_f - \Delta E_i, \quad (3)$$

where i and f denote the initial and final states (Fig. 5). The ω_3 and ω_4 transitions correspond to a change in the magnetic state of Ho in the electronic transition, and their probabilities are very small. The measured $\Delta\omega$ values are shown in Table 2. They characterize the energy differences of magnetic anisotropy of Ho ion in the ground and excited electronic states. It can be seen from Table 2 that these parameters significantly depend on the excited state of the electron. In $\text{HoFe}_3(\text{BO}_3)_4$ $\Delta\omega = +6.2 \text{ cm}^{-1}$ for line D1 and $+10.3 \text{ cm}^{-1}$ for line D7 [9], what appreciably differs from that in $\text{Ho}_{0.75}\text{Nd}_{0.25}\text{Fe}_3(\text{BO}_3)_4$ (Table 2). In $\text{HoFe}_3(\text{BO}_3)_4$ also distance between D1 and D7 lines differs from that in $\text{Ho}_{0.75}\text{Nd}_{0.25}\text{Fe}_3(\text{BO}_3)_4$. In particular, at 5 K it is equal to $15,164 \text{ cm}^{-1}$ in $\text{HoFe}_3(\text{BO}_3)_4$ and to $15,177 \text{ cm}^{-1}$ in $\text{Ho}_{0.75}\text{Nd}_{0.25}\text{Fe}_3(\text{BO}_3)_4$, that testifies to different crystal fields in Ho ion position.

The energy of the Ho ion in the ferroborate in the zero magnetic field is described by the Hamiltonian [15]:

$$\hat{H}(\text{Ho}) = \hat{H}_{CF} - g_J \mu_B \vec{J}_{\text{Ho}} \cdot \lambda_{fd} \vec{M}_{\text{Fe}}, \quad (4)$$

where \hat{H}_{CF} is the Hamiltonian of the crystal field on the Ho ion. Its shape corresponds to the symmetry of the local environment of the Ho ion in the considered electronic state and determines the local ionic anisotropy. g_J - Lande factor and \vec{J}_{Ho} rare-earth ion angular momentum operator; \vec{M}_{Fe} - magnetic momentum of Fe ion; $\lambda_{fd} < 0$ is a constant of antiferromagnetic interaction Ho-Fe. As it was mentioned above, the spin-reorientation transition in the crystal occurs as a result of the competition of local magnetic anisotropies of the exchange-coupled ions of Ho and Fe, which have different signs and temperature dependencies. The energy shifts $\Delta\omega$ and the behavior of the electronic transition intensities (Fig. 4) showed that the crystal field in the Ho position and, therefore, the local magnetic anisotropy change during the spin-reorientation transition. The Ho-Fe exchange energy is also different for different orientations of the magnetic moments of the Ho and Fe ions. All listed interactions depend on the electron state of the holmium, which is confirmed by non-zero and different changes in the energy of magnetic anisotropy at various electronic transitions.

4. Conclusions

Polarized absorption spectra of crystal $\text{Ho}_{0.75}\text{Nd}_{0.25}\text{Fe}_3(\text{BO}_3)_4$ in the region of the $^5I_8 \rightarrow ^5F_5$ Ho ion absorption band were measured at temperatures of 5–20 K. The spin-reorientation phase transition was detected at 6.9 K from the temperature dependence of the spectrum of one of the absorption lines. (In $\text{HoFe}_3(\text{BO}_3)_4$, this transition occurs at

Table 2
Shifts of absorption lines at the reorientation phase transition.

Line	D1	D2	D3	D5a	D5	D6a	D6	D7
$\Delta\omega \text{ (cm}^{-1}\text{)}$	+1.0	+3.3	+3.4	-2.3	-1.4	-3.2	-2.3	+8.1

4.7 K). In a certain temperature range, two magnetic phases coexist. It has been found that the shifts in the positions of the absorption lines during the reorientation transition are different between the lines. This was explained as a consequence of different magnetic anisotropy in excited states. Both line shifts and spin-reorientation transition temperatures are different in $\text{Ho}_{0.75}\text{Nd}_{0.25}\text{Fe}_3(\text{BO}_3)_4$ and $\text{HoFe}_3(\text{BO}_3)_4$. The Néel temperature and the structural phase transition temperature in these crystals are also different. In particular, in the former crystal $T_N = 32 \text{ K}$, and $T_C = 203 \text{ K}$, but in the latter crystal $T_N = 38 \text{ K}$, and $T_C = 360 \text{ K}$. Thus, substitution of Ho for Nd results in substantial changes of magnetic and structural properties of the crystal. Absorption spectra were decomposed into components of Lorentz forms. Significant changes in absorption line intensities were observed during and above the reorientation transition, while the overall absorption band intensity changed very weakly. The first indicates a change in even components of the local crystal field in excited states that mix closely spaced $4f$ states, while the odd components of the crystal field responsible for allowance of $f-f$ transitions change weakly. The appearance of π -polarization at one of the electronic transitions to the singlet state, unlike other transitions to the singlet states, indicated that at the former transition there was a distortion of local symmetry in the ground state.

Credit author statement

A.V. Malakhovskii: Formal analysis, Writing – original draft; A. L. Sukhachev: Investigation; V.V. Sokolov: Investigation; I.A. Gudim: Resources; V.R. Kuchsheva-Titova: Resources.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

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