The effect of pressure on the spectrum and spin-lattice relaxation of Ce^{3+} ions in europium and yttrium ethylsulfates

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The effect of hydrostatic pressure on the characteristics of the ground state of the Ce^{3+} impurity ion in two ethylsulfates has been investigated. A strong decrease in the spin-lattice relaxation rate is observed and is due to an increase in the energy of the first excited doublet on compressing the crystal. Good agreement is found between the experimental results and the theory of spin-lattice relaxation in the case of Orbach-Aminov processes, but there is a discrepancy for Raman processes. An anomaly induced by pressure is observed in the temperature dependence of the relaxation rate in europium ethylsulfate.

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1. INTRODUCTION

Ethylsulfate compounds with cerium ion impurity have been of interest for a long time because of a number of unusual properties which appear both in the EPR spectrum[1] and in spin-lattice relaxation.[2] In addition, an anomalous behavior of the specific heat of cerium ethylsulfate has been reported[3] and also a large proportion of non-dipolar interaction between nearest cerium ions in this compound.[4,5] The variety of properties is attributed to the existence of a closely spaced excited state.

The present work is devoted to an experimental study of the spectrum and spin-lattice relaxation of the Ce^{3+} ion in two crystals under hydrostatic pressure: yttrium ethylsulfate (YETS) and europium ethylsulfate (EuETS). An attractive feature of these experiments is the possibility, by means of pressure, to vary continuously the parameters which characterize both the spectrum and the spin-lattice relaxation. It is known from previous work[6–8] that the state of the ion can be changed by pressure. Conclusions about their interrelation can be drawn from the functional dependence of the parameters (such as the g-factor, relaxation time, energy of the excited state) on pressure. It becomes possible to verify some theoretical conclusions, since the existence of an experimentally observed dependence is always more informative than a single point. Finally, the results provide a basis for analyzing other properties of ethylsulfates with cerium impurity ions under pressure.

2. EXPERIMENTAL RESULTS

A. Experimental technique. Specimens.

The measurements of EPR spectra and spin-lattice relaxation of ceirum ions introduced into EuETS and YETS lattices were carried out with two spectrometers with different wavelengths: $\lambda = 3$ cm and $\lambda = 0.8$ cm, fitted with attachments for measurements at high pressures.

High hydrostatic pressures were produced in beryllium bronze piston-plus-cylinder type vessels. A kerosene + oil mixture, with various proportions of oil and kerosene, were used as the pressure transmitting medium. The EPR spectral lines broadened very weakly, indicating that the medium still acted hydrostatically at the low temperatures necessary for measurements on rare-earth ions. The error in pressure measurement was not more than 3%. Leucosapphire resonators in the H_{111} mode were used.[9] The specimens studied under pressure had dimensions: at $\lambda = 3$ cm a diameter $d = 5$ mm, thickness $l = 3$ mm and at $\lambda = 0.8$ cm $d = 3$ mm and $l = 1.5$ mm. The spin-lattice relaxation time was measured by a pulse saturation method. The temperature was controlled between 4.2 and 1.5 K by pumping helium vapor.

The crystallographic properties of the ethylsulfate $Ln(C_2H_5SO_4)_3 \cdot 9H_2O$ have been thoroughly studied.[10–12] The paramagnetic ion is surrounded by nine water molecules with local symmetry C_{3h}. The crystals were grown from the saturated solution by lowering the temperature. Specimens with various cerium ion densities from 0.1 to 3% were used. Values of the elastic constants and compressibilities[6] were used to estimate the change of the crystal volume and the velocity of sound. For reference, the density of the ethylsulfate is $\rho = 1.84\ \mathrm{g\cdot cm^{-3}}$, the compressibility is $\sigma_v = 7.6\times10^{-6}\ \mathrm{bar^{-1}}$ at 300 K, $\sigma_v = 6.3\times10^{-6}\ \mathrm{bar^{-1}}$ at 4.2 K, and the velocity of sound is $v = 1.85\times10^5\ \mathrm{cm\cdot s^{-1}}$.

B. EPR spectrum of Ce^{3+} ions

The ground state of the Ce^{3+} ion is ${}^2F_{5/2}$. The crystal field of C_{3h} symmetry splits the ground multiplet into three doublets: $|\pm\frac{1}{2}\rangle$, $|\pm\frac{3}{2}\rangle$, $|\pm\frac{5}{2}\rangle$. The crystal-field potential is fairly simple and can be written in the form[13]

$$\hat{\mathscr{H}}_{\mathrm{cr}} = \sum_{n,m} V_n{}^m = B_2{}^0 O_2{}^0 + B_4{}^0 O_4{}^0, \tag{1}$$

where O are spin operators and B_n^m are the crystal-field parameters characterizing the splitting.

For the crystals considered, the $|\pm\frac{5}{2}\rangle$ doublet is the lowest. The measured g factors, which can be uniquely identified for the given type of crystal potential, provide evidence for this.

A single absorption line of width ~ 10 G characterized by a strongly anisotropic g factor is observed. Since $g_\perp$ is small, the intensity of the signal for $H \| C_3$ is weak, about one-hundredth of that for Nd^{3+} at the same concentration. The pressure dependence of the g factors for EuETS and YETS

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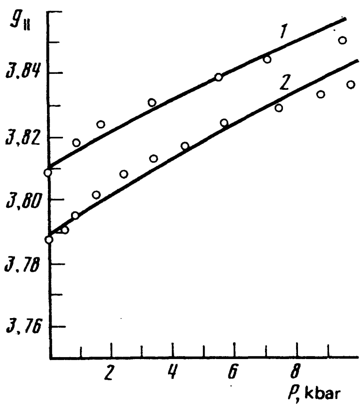

FIG. 1. Dependence of the $g_{\parallel}$ factors of the doublet $|\pm\frac{1}{2}\rangle$ on pressure for YETS (1) and EuETS (2).

crystals are shown in Fig. 1. A nonlinear increase of the g factor is observed for both crystals.

C. Spin-lattice relaxation

The temperature dependence of the relaxation time was measured at various pressures to elucidate the dominant mechanisms. The results for YETS are shown in Fig. 2 and Table I. Orbach-Aminov processes are dominant in this crystal with a contribution from Raman processes. The results of Larson and Jeffries[2] are shown as curve *l* in Fig. 2 and agree well with the present measurements at atmospheric pressure ($P=0$). As the pressure is raised the relaxation rate falls sharply, by about 100 times for $P=8.2$ kbar and $T=1.7$ K. The main cause of the observed reduction is the increase in the energy gap Δ_1 to the first excited doublet.

The relaxation rate can be written in the form[2]

$$T_1^{-1}=Be^{-\Delta_1/kT}+CT^9. \qquad (2)$$

The coefficients B and C and the distance to the excited doublet Δ_1 are shown in Table I for each pressure.

The change in Δ_1 with changing pressure is well represented by the linear dependence

$$\Delta_1=\Delta_0+KP, \qquad (3)$$

where $\Delta_0=18\ \text{cm}^{-1}$ and $K=1.2\ \text{cm}^{-1}\cdot\text{kbar}^{-1}$.

TABLE I. Experimental and theoretical quantities characterizing relaxation processes of the Ce^{3+} ion in YETS.

P, kbar	Δ_1, cm^{-1}	B, 10^9 s^{-1}	$B_{\text{th}}/30$, 10^9 s^{-1}	C, 10^{-2} s^{-1}	C_{th}, 10^{-2} s^{-1}
0	17.5	0.51	0.64	100	1.6
2.5	21	1.19	1.1	70	9.4
3.5	23	1.5	1.3	1.8	7.9
4.9	24.5	2.32	1.7	8.9	6.2
7.3	26.3	2.45	2.43	1.9	4.4
8.5	28	2.97	2.92	1.3	3.5

Similar measurements of spin-lattice relaxation were carried out on EuETS crystals. The ground state of the trivalent Eu^{3+} is a nonmagnetic singlet $^4F_0$, and the nearest magnetic state is at a distance ~300 cm^{-1} and has no effect on the ground state at helium temperature. Spin-lattice relaxation of the trivalent cerium in this crystal shows several features: a) the relaxation rate at $P=0$ is larger than in YETS, since Δ_0 is smaller than in YETS; b) Raman processes do not appear at $P=0$; c) an anomaly is observed in the temperature variation of the relaxation rate under pressure.

The temperature dependence of the relaxation rate of the Ce^{3+} ion in EuETS (Fig. 3) under hydrostatic compression can be arbitrarily divided into three parts. In the high-temperature relaxation region Orbach-Aminov process predominates. The characteristic exponential dependence is represented a straight line as in the graph whose slope gives the distance Δ_1 to the excited state that participates in the relaxation. An increase in P leads to a linear increase in Δ_1, just as the YETS [Eq. (3)], but $\Delta_0=13.8\ \text{cm}^{-1}$ and $K=1.2\ \text{cm}^{-1}\cdot\text{kbar}^{-1}$. The rate of increase of the energy gap is thus the same as for YETS.

In the transition region the temperature dependence has an anomalous shape. On increasing the pressure above

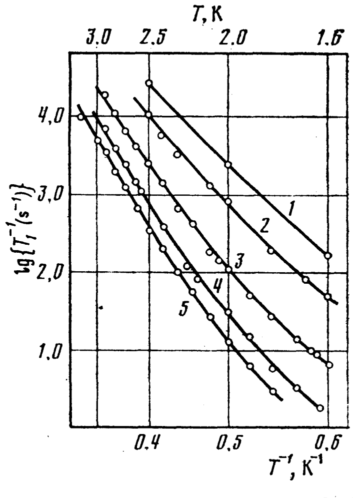

FIG. 2. Temperature dependence of relaxation rate for the Ce^{3+} ion in YETS for various pressures: 1) 0 kbar; 2) 2.5 kbar; 3) 4.9 kbar; 4) 7.3 kbar; 5) 8.5 kbar.

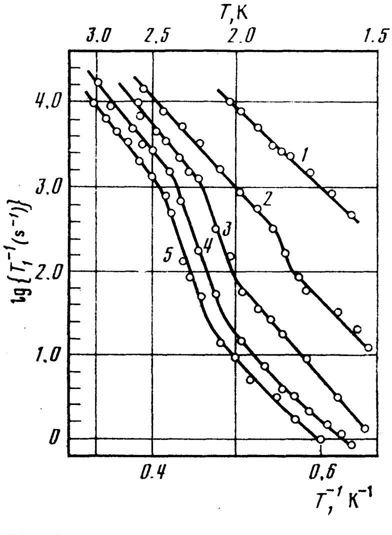

FIG. 3. Temperature dependence of relaxation rate for the Ce^{3+} ion in EuETS for various pressures: 1) 0 kbar; 2) 2 kbar; 3) 4.2 kbar; 4) 5.6 kbar; 5) 8.2 kbar.

TABLE II. Experimental and theoretical quantities characterizing relaxation processes of the Ce^{3+} ion in EuETS.

P, kbar	Δ_1, cm^{-1}	B, 10^8 s^{-1}	B_{th} /30, 10^8 s^{-1}	T_c, K
0	13.6	1.63	1.6	–
2	16	3.01	2.7	1.8
4.2	19.1	4.74	4.5	2.1
5.6	20	5.97	6	2 2
8.2	23.4	7.95	9.8	2.3

atmospheric, a sharp decrease in the relaxation rate is observed over a narrow temperature range. The temperature of the jump, T_c, determined from the center of the inflection, increases with increasing pressure (Table II).

Finally, after the anomalous jump of the rate, the Orbach-Aminov process is restored with the same slope as in the high temperature region. At the highest pressures, when the distance to the excited doublet has increased appreciably, a small departure from Orbach-Aminov processes is observed. It is possible that Raman or direct processes manifest themselves here. There were not enough experimental points for an accurate identification in this region. Values of the coefficients B and Δ_1 for various pressures are shown in Table II.

We should point out that the anomalous behavior of the relaxation was observed for crystals with different Ce^{3+} contents from 0.5 to 3%, i.e., the observed effect is independent of the amount of impurity.

3. DISCUSSION OF THE RESULTS

A. Choice of parameters

To discuss the results we shall use the crystal field theory developed to interpret EPR spectra of rare-earth ions,[13] and also conclusions from considerations of the theory of spin-lattice relaxation.[2,14,15] The parameters of the crystal field B_n^m in the Hamiltonian (1) have to be chosen in order to compare the experimental and theoretical results. Unfortunately, for the Ce^{3+} ion in ethylsulfates, there is no known energy spectrum that includes all the excited states, so that it is not possible to determine all the parameters. However, the parameters B_n^m chosen by analogy with other rare-earth ions implanted in ethylsulfates are given elsewhere.[1,2,14] We proceed in the same way and add all the results obtained in the present work.

The energy spacing Δ_1 to the first excited doublet and its pressure variation $\partial\Delta_1/\partial P$ are known from §2(C). It follows from solving the Hamiltonian of Eq. (1) that:

$$\Delta_1=1.03B_2^0+0.381B_4^0, \qquad (4)$$

$$\partial\Delta_1/\partial P=1.03\partial B_2^0/\partial P+0.381\partial B_4^0/\partial P.$$

It can be concluded from the results of work in which the changes in the parameters of the crystal field with pressure were determined for the ions Nd^{3+} (Ref. 8), Pr^{3+} (Ref. 16), and Gd^{3+} (Ref. 6) that the parameter B_2^0 changes most. Furthermore, parameter B_4^0 varies less from ion to ion.[13]

The following values of the parameters are the most suitable for making the experimental results agree with Eq. (4):

$$\text{YETS:}\quad B_2^0=35\ \text{cm}^{-1},\quad B_4^0=-42\ \text{cm}^{-1};$$

$$\text{EuETS:}\quad B_2^0=31\ \text{cm}^{-1},\quad B_4^0=-42\ \text{cm}^{-1}.$$

Since the pressure dependence of Δ_1 is the same for both crystals, we shall also take the derivatives of the parameters, $\partial B_n^m/\partial P$, to be the same for YETS and EuETS and equal to

$$\partial B_2^0/\partial P=1.3\ \text{cm}^{-1}/\text{kbar},\quad \partial B_4^0/\partial P=-0.4\ \text{cm}^{-1}/\text{kbar}.$$

The energy gap to the $|\pm\frac{3}{2}\rangle$ doublet and its variation with pressure can be calculated by using the chosen values:

$$\text{YETS:}\quad \Delta_2=88\ \text{cm}^{-1},\quad \partial\Delta_2/\partial P=1.5\ \text{cm}^{-1}/\text{kbar};$$

$$\text{EuETS:}\quad \Delta_2=85.3\ \text{cm}^{-1},\quad \partial\Delta_2/\partial P=1.5\ \text{cm}^{-1}/\text{kbar}.$$

There is, naturally, some arbitrariness in the choice of absolute values for the crystal field parameters. However, as will be seen later, the changes in the energy spacing Δ_1 with pressure, which are measured experimentally, play the largest role.

B. Change in *g* factors

According to the static model of a crystal field described by the potential (1), the g factors should not change with a change in the parameters B_n^m, since the states of the Ce^{3+} ion are a pure states. However, the observed g factors are fairly far from the theoretical ones and, moreover, change with increase in pressure. There is, consequently, a mixing of states. There are two possible explanations for this.

According to the first,[17] mixing of states $|\pm\frac{1}{2}\rangle$ and $|\pm\frac{5}{2}\rangle$ occurs because of the addition of the terms $B_n^3 O_n^3$ to the crystal potential (1).

According to the second explanation,[1] the mixing is produced by the dynamic crystal field, i.e., by those lower-symmetry terms which appear because of lattice vibrations (of the $B_n^2 O_n^2$ type).

The change in g factors on increasing Δ_1 should be much faster in the first case, since in calculating the admixture of the excited state by perturbation theory denominator contains the energy spacing Δ_1 while in the second case it contains $\Delta_1 + E_{ph}$, where E_{ph} is the energy of the phonons.

The second assumption is confirmed experimentally. One can verify that the change in the g factors is very insignificant by varying Δ_1 by means of pressure. In addition, it has been shown[18] that in direct observation of interdoublet transitions between the $|\pm\frac{1}{2}\rangle$ and $|\pm\frac{5}{2}\rangle$ states in LaETS the transitions corresponding to the second assumptions are allowed.

A simple expression for the g factor of the $|\pm\frac{5}{2}\rangle$ doublet has been obtained[19] for the Ce^{3+} ion in $LaCl_3$, which has the same symmetry as ethylsulfate

$$g=g_{5/2}\{1-a_1(1-g_{1/2}/g_{5/2})-a_2(1-g_{3/2}/g_{5/2})\}, \qquad (5)$$

where $g_{1/2}$, $g_{3/2}$, and $g_{5/2}$ are the theoretical g factors on the static crystal field model, and are for the ethysulfates respectively 1.01; 2.537; 4.138; (Ref. 1) the *a* are coefficients equal to

$$a_1=N/(\Delta_1+E_{\text{ph}})^2, \quad a_2=N/(\Delta_2+E_{\text{ph}})^2,$$

where N is a quantity determined by properties of the phonon spectrum and E_{ph} is the energy of the phonons which introduce the greatest change in g factor.

We obtained the following values by minimizing expression (5) using the experimental values:

$$\text{YETS:}\quad N=1700\ (\text{cm}^{-1})^2,\ E_{\text{ph}}=124\ \text{cm}^{-1};$$

$$\text{EuETS:}\quad N=1550\ (\text{cm}^{-1})^2,\ E_{\text{ph}}=116\ \text{cm}^{-1}.$$

The greatest contribution to the difference between the g factors and the theoretical values thus comes from phonons of frequency $\sim 120\ \text{cm}^{-1}$, which agrees well with Wong and Erath,[20] who gave the vibronic spectrum of an ethylsulfate with Pr^{3+} and Nd^{3+} impurity. In fact, the greatest intensity in the vibronic spectrum appears in the region from 100 to 140 cm^{-1}. The solid lines in Fig. 1 show the change in g factors according to Eq. (5).

C. Spin-lattice relaxation

The relaxation characteristics of rare-earth ions have been considered in greatest detail for ethylsulfates[2,15,14] both theoretically and experimentally. We compare the theoretical calculations with experiments at the parameters change produced by hydrostatic pressure.

We shall use the analytical expression of Larson and Jeffries.[2] The relaxation rate for Orbach-Aminov processes is

$$T_1^{-1}=\frac{3}{2\pi\rho v^5\hbar}\left(\frac{\Delta_1}{\hbar}\right)^3$$

$$\times 2\,\frac{\sum\limits_{n,m}|\langle a|v_n^m|c\rangle|^2\sum\limits_{n,m}|\langle c|v_n^m|b\rangle|^2}{\sum\limits_{n,m}|\langle a|v_n^m|c\rangle|^2+\sum\limits_{n,m}|\langle c|v_n^m|b\rangle|^2}\,e^{-\Delta_1/kT}, \tag{6}$$

where ρ is the density, v the velocity of sound, Δ_1 the energy distance to the first excited state, and T is the temperature; $|a\rangle$ and $|b\rangle$ are the wave functions of the lower doublet, $|c\rangle$ and $|d\rangle$ are the wave functions of the first excited doublet, v_n^m are the crystal-field dynamic parameters determined by Larson and Jeffries.[2] Taking account of all matrix elements contained in Eq. (6), making the necessary transformations and in doing so neglecting small terms, Eq. (6) can be transformed into the simple form:

$$T_1^{-1}=1.9\cdot 10^3(\Delta_1)^3B_4^0e^{-\Delta_1/kT}=Be^{-\Delta_1/kT}. \tag{7}$$

Δ_1 and B_4^0 are expressed in cm^{-1}, T in Kelvin, T_1^{-1} in s^{-1}. The calculated values of the coefficient B, divided by 30, are shown for both crystals in Tables I and II.

Comparison with the experimental values shows that the theory describes sufficiently well the change in relaxation rate with pressure. It should be emphasized that the agreement is in the pressure dependence and not in the absolute values. As can be seen from Eq. (7), the Δ_1^3 dependence plays the main role, since this quantity changes the most strongly. Experiment confirms this dependence fairly accurately.

It is interesting to note that the calculated value of the coefficient B is 30 times larger than the experimental value for both crystals. This indicates that the disagreement does not come about by chance but from some shortcomings of the theory. Confirmation of the Δ_1^3 dependence in the experiments excludes the possibility of proposing a phonon bottleneck in Orback-Aminov processes even at large concentrations of $\sim 3\%$. It should be remembered that a Δ_1^3 dependence was not observed in experiments on the cerium ion in lanthanum magnesium nitrate.[21]

The relaxation rate for Raman processes is expressed in the following way:

$$T_1^{-1}=\frac{9!\,\hbar}{\pi^3\rho^2v^{10}}\left(\frac{k}{\hbar}\right)^9\sum\frac{1}{\Delta_i^4}$$

$$\times\sum|\langle a|v_n^m|i\rangle|^2\sum|\langle i|v_n^m|b\rangle|^2T^9 \tag{8}$$

(k is Boltzmann's constant and the other symbols have the same meaning as in the previous case). Summation is over all excited states $|i\rangle$. Since Δ_i^4 occurs in the denominator, the contribution from the second excited level is small and we shall neglect it.

The expression transforms, as in the previous case, to the simpler form:

$$T_1^{-1}=4.8\cdot 10^{-3}\Delta_1^{-4}(B_4^0)^4T^9=CT^9. \tag{9}$$

Calculated and experimental values of the coefficient C are shown in Table I. It can be seen that neither the absolute values nor the form of the variation in the theoretical quantities C agree with the experimental results. In the experiments C decreases much more rapidly than follows from theory. It can be concluded from what has been said that either the departure from Orbach-Aminov processes in the low-temperature region for YETS (see Fig. 2) cannot be ascribed to Raman processes and another cause has to be sought, or the theory of Raman relaxation processes does not take account of some most essential factors.

Additional studies were carried out to elucidate the nature of the anomalous reduction in relaxation rate which occurs in EuETS under pressure. The EPR spectra of single ions and pairs of nearest-neighbor cerium ions were carefully measured with the aim of finding temperature anomalies in the region of the jump in relaxation rate. However, no changes were observed within the limits of experimental errors. Since two interacting systems, the spin system and the lattice, take part in spin-lattice relaxation, it can be proposed on the basis of the measurements that drastic changes, furthermore in a fairly narrow frequency range, occur in the spectrum of high-energy phonons interacting with the electron system of the Ce^{3+} ions in phonon resonant-fluorescence processes.

There are also no anomalies in the relaxation of the Nd^{3+} ion measured in a EuETS crystal under similar condi-

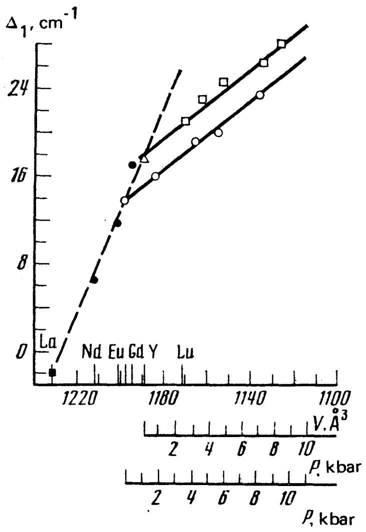

FIG. 4. Dependence of energy spacing Δ_1 in ethylsulfates on the volume of the unit cell. A pressure scale is also given for YETS and EuETS: □—YETS (present work); ○—EuETS (present work); △—Ref. 2; ●—Ref. 22; ■—Ref. 18.

tions. This confirms indirectly the assumption that a narrow frequency range undergoes change, since relaxation for the Nd^{3+} ion proceeds via a direct process in which phonons with energy 0.3 cm^{-1} participate, and a Raman processes in which the entire phonon spectrum interacts with the spin system.

In conclusion we compare the methods of homologous series and hydrostatic pressure with regards to their influence on the splitting Δ_1 in the crystal field. The summary results are shown in Fig. 4, where the energy of the first excited doublet is the ordinate and the unit cell volume of the abscissa. A pressure abscissa scale is also included. The relationship between the scales is established by using the known compressibility of the crystals.[6] Changing the volume by substituting rare-earth ions turns out to be a stronger influence on Δ_1 than pressure: $\partial\Delta_1/\partial V = 0.47$ cm^{-1} A^{-3} for substitution and 0.13 cm^{-1} A^{-3} for hydrostatic pressure. A qualitatively similar picture is observed when comparing the parameters B_2^0 for the Gd^{3+} ion under similar conditions.

4. CONCLUSIONS

From the experiments described, information has thus been obtained on the change in the chief characteristics of a paramagnetic ion under hydrostatic pressure. The energy spacing to the first excited doublet, Δ_1, and also its rate of change with pressure, were found for two crystals from relaxation measurements. Since these are the decisive parameters, the changes in both the g factors and relaxation rates with pressure could be analyzed from the results. The Δ_1^3 dependence of relaxation rate, typical of Orbach–Aminov processes, was found experimentally. The experimentally determined values of the coefficients that describe the Raman processes were markedly different from the theoretical predictions.

An anomalous decrease of the relaxation rate was found for Orbach–Aminov relaxation processes and is ascribed to changes in the phonon spectrum of the crystal.

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