Manifestation of non-centrality effect in the EPR spectrum of Fe$^{3+}$ ion in the polycrystalline materials

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Abstract. In a number of works [1-6], the study of the electron paramagnetic resonance (EPR) spectrum of polycrystalline non-organic and organic matters revealed presence of EPR spectrum of impurity Fe$^{3+}$ ion which has essentially different forms at helium and room temperatures. At the helium temperature intensity of the resonance line with g-value close to $g_1 \approx 4.3$ prevails (Line 1 of the low temperature spectrum). The resonance line with $g \approx 4.3$ corresponds to magnetic center with spin $S = 5/2$ by the spin Hamiltonian parameter $D >> \hbar \nu$. The resonance line with $g \approx 2.0$ (Line 2 of the high temperature spectrum) prevails at the room temperatures. The resonance line with $g_2 \approx 2.0$ corresponds to magnetic center with spin $S = 5/2$ by the spin Hamiltonian parameter $D << \hbar \nu$. The transition from the low- to high-temperature spectrum is gradual and it is accompanied by a redistribution of the absorption intensity. The observed properties of the temperature dependence of the EPR spectrum are characteristic of systems with a multiminimum potential. The described effect of change of integral intensity of low-temperature and high-temperature EPR spectrum of Fe$^{3+}$ ion is investigated in substances Na[FeO$_6$(C$_{10}$H$_8$N)$_3$], C$_{21}$H$_{14}$Br$_4$O$_5$S, C$_{25}$H$_{30}$ClN$_3$, Al$_2$[Si$_2$O$_5$](OH)$_4$, polyaniline and poly paraphenylene.

1. Introduction
This work represents the review of results of researches of EPR spectrum of Fe$^{3+}$ ion in a number polycrystalline organic substances and kaolinite [1-6]. Feature of the given researches is the experimental detection of unusual structure of EPR spectrum of Fe$^{3+}$ ion in polycrystalline substances and unusual temperature dependence of integral intensity of resonance lines of EPR spectrum.

In works [1-6] EPR spectrum of Fe$^{3+}$ ion is investigated in an interval of temperatures from temperature liquid Helium up to room temperature in substances Na[FeO$_6$(C$_{10}$H$_8$N)$_3$], C$_{21}$H$_{14}$Br$_4$O$_5$S, C$_{25}$H$_{30}$ClN$_3$, Al$_2$[Si$_2$O$_5$](OH)$_4$, polyaniline and poly paraphenylene.

In all investigated substances EPR spectrum represents a superposition of two resonance lines, intensity of which behave opposite with change of temperature. At temperature decreases the intensity of one line increases, and intensity of the other decreases. One of the resonance lines dominates at low (helium) temperatures, and the other at room temperatures. The first line was determined as low-temperature (LT) component of spectrum, and the second one as high-temperature (HT) component. The change of temperature results in redistribution of intensity of absorption between LT and HT components of EPR spectrum. The similar behavior of EPR spectrum testifies unusual dynamics of molecular environment of Fe$^{3+}$ ion.
The research of these dynamic transitions has independent significance, because systems which showing such properties are systems with multiminimum potential. The systems with multiminimum potential can have various physical nature, but the most investigated system with the multiminimum potential is the Jahn-Teller ion (divalent copper ion in an octahedral environment) [7].

Fe\(^{3+}\) ion is S-ion and consequently the display of Jahn-Teller effect is impossible. It is more probable that properties of Fe\(^{3+}\) ion revealed in works [1-6] are associated with display of an off centre effect [8].

The off centre features appear in EPR spectrum in a number of characteristics: 1) presence LT and HT components of EPR spectrum, 2) redistribution of intensity between LT and HT components at temperature change.

2. Experimental results

The research of EPR spectrum was performed with microwave frequency close to \(\nu \approx 9.3\, \text{GHz}\) in temperatures interval \(T = 4.2 \div 300\, \text{K}\).

In the investigated substances the resonance lines of EPR spectrum of Fe\(^{3+}\) ion have significant width. In this work for efficient analysis of the contributions EPR spectra are submitted in the form of absorption line, instead of common form of a derivative of an absorption line.

Figures 1-4 show absorption lines of EPR spectrum of Fe\(^{3+}\) ion for organic substances Na[Fe(C\(_{10}\)H\(_{6}\)O(NO))]\(_{3}\), C\(_{2}H_{12}Br_{4}O_{5}S\), C\(_{2}H_{10}ClN_{3}\) and polyaniline for several temperatures. The absorption lines are obtained by numerical integration of experimental EPR spectra. Figure 5 shows
EPR spectrum of Fe\(^{3+}\) ion in Na[Fe(C\(_{10}\)H\(_{6}\)(NO))\(_{3}\)] at temperature \(T = 4.2\) K given in the form of an absorption line and in the form derivative simultaneously.

The analysis of EPR spectra (figures 1-4) shows that EPR spectra of the investigated substances contain two resonance lines, which are named resonance lines 1 and 2. On the ground of the g-values and character of temperature change of intensity we can draw a conclusion that these resonance lines have an essentially different physical nature. For investigated substances the g-values of resonance lines 1 and 2 are given in the table 1.

As shown in figures 1-4, the value of resonance magnetic field of lines 1 and 2 practically does not vary at change of temperature.

![Graph showing EPR spectra and resonance lines](image)

**Figure 2.** Temperature change of the form of the EPR spectrum in polyaniline. The resonance lines 1 and 2 belong to Fe\(^{3+}\) ion, and the resonance line 3 belongs to a radical.

| Substrate                          | Line 1             | Line 2             |
|-----------------------------------|--------------------|--------------------|
| Substance                         | \(g_1\) T, K       | \(g_2\) T, K       |
| Na[Fe(C\(_{10}\)H\(_{6}\)(NO))\(_{3}\)] | 4.32 \(\pm\) 0.02 | 4.2                 | 2.00 \(\pm\) 0.01 | 290  |
| Polyaniline                       | 4.22 \(\pm\) 0.03 | 4.2                 | 2.13 \(\pm\) 0.05 | 295  |
| \((C_2\text{H}_4\text{Br}_4\text{O}_5\text{S})\) | 4.39 \(\pm\) 0.03 | 4.2                 | 2.03 \(\pm\) 0.02 | 295  |
| \((C_2\text{H}_3\text{ClN}_3)\)        | 4.5 \(\pm\) 0.1    | 4.2                 | 2.15 \(\pm\) 0.15 | 185  |
| Polyparaphenylene                 | 4.21 \(\pm\) 0.05 | 4.2                 | 2.00 \(\pm\) 0.09 | 300  |
| Al\(_2\)[Si\(_3\)O\(_5\)](OH)\(_4\) | 4.13 \(\pm\) 0.16 | 4.2                 | 2.15 \(\pm\) 0.1  | 288  |
For more detailed analysis of temperature dependence of integral intensities and resonance line widths modeling of forms of resonance lines and numerical decomposition of absorption lines is carried out for all investigated substances and temperatures.

For example, figures 6 and 7 show the decomposition of lines of absorption for Na[Fe(C_{10}H_{6}(NO))_3] and Al_2[Si_2O_5](OH)_4 for two temperatures. At temperature 4.2 K intensities of a lines 1 for Na[Fe(C_{10}H_{6}(NO))_3] and Al_2[Si_2O_5](OH)_4 are maximal, and the intensities of a lines 2 are minimal. On the contrary, intensity of the resonance line 1 for Na[Fe(C_{10}H_{6}(NO))_3] is minimal at temperature 277 K (line 1 is not visible almost), and the intensity of a line 2 is maximal. Similarly to Al_2[Si_2O_5](OH)_4, intensity of the resonance line 1 is minimal, and the intensity of the line 2 is maximal
at temperature 288. The intensity and form of lines 1 and 2 were chosen on condition that there was a minimal deviation between the model line, being a superposition of lines 1 and 2, and the experimental form of the absorption line.

Figure 5. EPR spectrum in the form of the absorption curve and in the form derivative obtained experimentally.

It is necessary to pay attention that modeling of the form of resonance lines and decomposition of absorption lines show that absorption lines (except for resonance lines 1 and 2) contain addition non-resonance contribution as it shown in figure 6.

Figure 8 shows the temperature change of the absorption line of EPR spectrum of Fe$^{3+}$ ion in inorganic substance kaolinite (Al$_2$[Si$_2$O$_5$](OH)$_4$). On top of resonance line 2 narrow resonance line of small intensity belonging to a radical is observed. This radical is used for magnetic field calibration. It is necessary to note that in figure 8 non-resonance contribution previously was removed during the analysis of an absorption line for Al$_2$[Si$_2$O$_5$](OH)$_4$.

3. Temperature change of EPR spectra of Fe$^{3+}$ ion
The qualitative analysis of the results given in figures 1 - 4 and 8 shows, that temperature change of intensity of resonance lines 1 and 2 is different.

To implement the quantitative analysis of temperature changes of a spectrum for all investigated substances and temperatures modeling resonance lines and numerical decomposition of absorption lines has been carried out as shown in figures 6 and 7.

The analysis of temperature dependence of integral intensity of resonance lines shows that temperature dependence of a spectrum has two mechanisms. It is possible to name the first mechanism usual. It results in reduction of integral intensity of all contributions of a EPR spectrum. This reduction of intensity is described by ratio $J(T) = J_0 \tanh(h\nu/2kT)$. For example, figure 9 shows the temperature dependence of integral intensity of resonance line 1 for Al$_2$[Si$_2$O$_5$](OH)$_4$.

The second mechanism of temperature dependence is shown in redistribution of intensity between the various contributions to EPR spectrum and is defined the multiminimum potential of the magnetic centre. Figures 10 - 13 show the temperature dependences relative integral intensities of resonance lines 1 and 2, determined by ratio $J_{1R} = J_1/(J_1 + J_2)$, $J_{2R} = J_2/(J_1 + J_2) - J_0$, where $J_1$, $J_2$ are integral intensity of resonance lines 1 and 2. Parameter $J_0$ is connected to height of a potential barrier between
minima. At plotting of figures 10 – 13, parameter $J_0$ is used for mutual scaling of intensities of lines 1 and 2. These dependences describe the second mechanism of temperature change in the EPR spectrum which describes the process of the integral intensity redistribution between lines 1 and 2.

Figure 6. Decomposition of the resulting absorption line in Fe$^{3+}$ EPR spectrum in Na[Fe(C$_{10}$H$_6$ONO)$_3$] into component resonance lines 1, 2 and non-resonance absorption.

a) $T = 4.2$ K, b) $T = 277$ K.

As a result of modeling the form of resonance lines and numerical decomposition of an absorption lines on contributions not only temperature dependences integral intensities of resonance lines have been obtained, but also temperature dependences of width of resonance lines. Figure 13 shows the dependences line width of EPR spectrum of Fe$^{3+}$ ion in Al$_2$[Si$_2$O$_5$](OH)$_4$ and Na[Fe(C$_{10}$H$_6$NO)$_3$]. As shown in figure 13 width of a resonance line 1 in Al$_2$[Si$_2$O$_5$](OH)$_4$ in interval of temperatures $T = 4.2$ - 300 K changes not considerably. Width of a resonance line 2 increases with temperature decrease as for Al$_2$[Si$_2$O$_5$](OH)$_4$ and for Na[Fe(C$_{10}$H$_6$NO)$_3$]. When the temperature decrease, the width of line 2 grows exponentially. Such change of line width is not caused by relaxation mechanisms of line-broadening, and is defined by specificity of formation of a resonance line of EPR spectrum of Fe$^{3+}$ ion in polycrystalline substance in a case multiminimum potential of the magnetic centre.

4. Discussion of the results
EPR spectra, investigated in [1-6], contain two resonance lines (figures 1-4, 8). One line with the effective g-value $g_1 \approx 4.3$, and another with the effective g-value $g_2 \approx 2$. The lines of EPR spectrum
with such g-values are characteristic for Fe$^{3+}$ ions in polycrystalline substances. In [9, 10] the different reasons for simultaneous observation of resonance lines with the g-values $g \cong 2$ and $g \cong 4.3$ are considered. In [10] the analysis of results of research of EPR spectra of ions of iron in silicate glasses is carried out, and in article [9] the temperature change of EPR spectrum of Fe$^{3+}$ ions in polycrystalline substances in the presence of the off centre effect is theoretically investigated.

In [10] it is assumed that the spectrum consisting of two resonance lines with g-values $g \cong 2$ and $g \cong 4.3$ belongs to different nonequivalent magnetic centers of the Fe$^{3+}$ ion.

![Figure 7](image_url)

**Figure 7.** Computer resolution of the absorption line for Al$_2$[Si$_2$O$_5$(OH)$_4$. a) T = 4.2 K, b) T = 288 K.

The distinction of the centres consists in distinction of the low-symmetry components of a crystalline field acting on a magnetic ion. The line with $g \cong 2$ corresponds to the centre, low-symmetry component of crystal field of which there is less Zeeman energy. Under this condition, zero splitting parameter $D$ satisfies to a condition $D \ll \hbar \nu$, where $\hbar \nu$ quantum of a microwave field. The line with $g \cong 4.3$ corresponds to the centre, low-symmetry component of crystal field of which is much greater of Zeeman energy. Under this condition, zero splitting parameter $D$ satisfies to a condition $D \gg \hbar \nu$.

In [1-6] it is supposed that the resonance lines with the g-values $g \cong 2$ and $g \cong 4.3$ belong to various vibronic states of the same magnetic centre of Fe$^{3+}$ ion. This assumption is made on the basis of the analysis of temperature dependence of EPR spectra of substances containing noncentral Fe$^{3+}$ ions [1-6] and model off centres [9], which takes into account whole set vibronic states. This model is similar to
description of the temperature change of EPR spectrum of Jahn-Teller ion having ground orbital $^2E$-state [7, 11].

![Figure 8](image.png)

**Figure 8.** Temperature change of the form of the EPR spectrum in kaolinite $\text{Al}_2[\text{Si}_2\text{O}_5](\text{OH})_4$ in the form of the absorption curve for the temperatures $T = 4.2, 9, 26, 50, 100, 202$ and 288 K.

According to the temperature dependence of the EPR spectrum obtained in [1-6] the Fe$^{3+}$ ions are distributed over states having different values of the low-symmetry component of the crystalline field. At low temperatures, the states with the low-symmetry component possessing much more than the Zeeman energy are mainly occupied. When the temperature increases, the excited states with the low-symmetry component much less than the Zeeman energy are occupied. Since the total number of magnetic ions is constant, the transition of ions to the excited states results in the decrease of the intensity of line 1 as the temperature increased. Line 2 becomes more intense in this case (figures 1-4, 8).

We thus pay attention to the following three characteristics. First, the low-temperature states are characterized by the low-symmetry properties; while the excited states are typical of the properties of much higher symmetry. Secondly, a region exists in which the low- and high-symmetry spectra coexist. Thirdly, a decrease of the intensity of the low-symmetry spectrum when the temperature increases, and a corresponding increase of the intensity of the high-symmetry spectrum is observed. Such characteristics are inherent in the systems with the multiminimum potential.
The best studied system with the multiminimum potential is the Jahn-Teller ion of a divalent copper ion in an octahedral environment. In such systems, the temperature-induced changes in the intensity of spectra which correspond to the low- and high-symmetric states are due to changes in the population of vibronic states [7, 11]. It should be noted that Fe$^{3+}$ is an S-ion, and the appearance of the Jahn-Teller effect for this ion is hardly probable. In the present case, the ion plays the role of a paramagnetic probe enabling one to observe the unusual dynamics of the ligand environment. The coincidence of the temperature changes of the EPR spectrum of Fe$^{3+}$ in investigated substances with similar temperature changes in systems with the multiminimum potential provides grounds to propose that the magnetic center under consideration possesses similar properties.

**Figure 9.** Temperature dependence of the integral intensities of lines 1 in the EPR spectrum of Fe$^{3+}$ in kaolinite Al$_2$[Si$_2$O$_5$](OH)$_4$. The solid line describes dependence $J(T) = J_0*\tanh(h\nu/2kT)$.

**Figure 10.** Temperature dependence of the relative integral intensities of lines 1 and 2 in kaolinite Al$_2$[Si$_2$O$_5$](OH)$_4$.
Figure 11. Temperature dependence of the relative integral intensities of lines 1 and 2 in bromide cresol green C_{21}H_{14}Br_{4}O_{5}S.

Figure 12. Temperature dependence of the relative integral intensities of lines 1 and 2 in poly paraphenylene.

Figure 13. Temperature dependence of the width of resonance lines in kaolinite $\text{Al}_2[\text{Si}_2\text{O}_5](\text{OH})_4$ and $\text{Na}[\text{Fe}(\text{C}_{10}\text{H}_6(\text{NO}))_3]$.

Figure 14. a) Potential surface form of the magnetic center and b) dependence of the zero splitting parameter on vibronic states energy.

In Figure 14а for visibility representation of structure and vibronic states properties, the form of a potential surface with two potential holes is schematically given. The quantity of holes in different substances can be different. For example, as shown in article [3] in poly-paraphenylene the magnetic $\text{Fe}^{3+}$ ion has two potential holes, and as theoretically shown in article [8] non-central ions in cubic crystals can have six or eight potential holes. In Figure 14а the parameters $E_0$ and $R_0$ designate height of a barrier dividing potential holes and spontaneous shift of $\text{Fe}^{3+}$ ion from a point of symmetry of a molecular environment.

It is convenient to divide whole set vibronic states into three parts according to their properties. By analogy with [7] these groups of states it is possible to name under barrier, about barrier and above
barrier states. In figure 14а the area about barrier states is allocated with a grey background. The magnetic ion in under barrier state is actually located in one of potential holes and for this reason its molecular environment is deformed much more strongly, than the environment of an ion, which is in any above barrier state. About a barrier states have intermediate properties. The properties about barrier states strongly depend on energy of excitation and vary from isotropic properties of above barrier states up to strongly anisotropic properties of under barrier states.

The distortion of an environment results in occurrence of low-symmetry component of the crystalline field working on a magnetic ion. This low-symmetry component of the crystalline field determines zero splitting parameter D, which corresponds with given vibronic state. Different vibronic states have the different values of zero splitting parameter D. Figure 14b schematically shows the dependence of parameter D on activation energy of vibronic states. The maximal value of parameter D₀ corresponds with the ground vibronic state. For above barrier vibronic states parameter D is equal to zero.

Figure 15. Structure of the energy states of the spin multiplet S=5/2 and a form of the EPR spectrum at T=4.2K in kaolinite.

In article [9] is shown, that at the given value of microwave field quantum hv the line with the g-value \( g \approx 4.3 \) is observed in a EPR spectrum of a polycrystalline sample under condition, if zero splitting parameter of ground vibronic state fulfils condition \( D₀ \geq hv \). This condition less rigid than condition \( D₀ >> \cdot hv \), given in [10]. For an Fe³⁺ ion the condition \( D₀ = hv \) defines border \( E₁ \) between under barrier and about barrier vibronic states.

In article [9] is shown that a resonance line with the g-value \( g \approx 2 \) in EPR spectrum of a polycrystalline sample is observed under condition \( D₀ \leq 0.26 \cdot hv \). For the Fe³⁺ ion the condition \( D₀ = 0.26 \cdot hv \) defines border \( E₂ \) between about barrier and above barrier vibronic states.

It should be noted that the three different states of the Fe³⁺ magnetic ion are observed experimentally. In the first low-symmetry state, the parameter of spin Hamiltonian \( D >> hv = 0.3 \text{ cm}^{-1} \). The presence of line 1 testifies to the existence of such a state.

The third state is highly symmetric and it is defined by the spin Hamiltonian parameter \( D << D₀ < hv \). The form of line 2 at \( T \approx 300 \text{ K} \) corresponds to this state.
The second state is intermediate on degree of molecular environment distortion. In kaolinite at \(T = 4.2\) K analyses of form of resonance line 2 allow to estimate the spin Hamiltonian parameter \(D \approx 0.09\) cm\(^{-1}\) (\(D < h\nu\)) (Fig.15).

In the high-field part of resonance line 2 of the EPR spectrum of figure 8 (\(T = 4.2, 9, 26\) K), one can see the maxima rather well. The maxima are typical of the EPR spectrum which is averaged over directions of the magnetic field. Figure 15 shows the structure of energy levels of the multiplet \(S=5/2\) and the EPR spectrum for \(T = 4.2\) K in kaolinite. The resulting form of line 2 averaged over magnetic field directions has 5 maxima. The central maximum corresponds to transition \(1/2 \longleftrightarrow -1/2\). The other maxima correspond to another four transitions and appear as a background of the averaged line 2. The two high-field maxima correspond to transitions \(-1/2 \longleftrightarrow -3/2, -3/2 \longleftrightarrow -5/2\). The two close maxima corresponding to transitions \(-5/2 \longleftrightarrow -3/2\) and \(3/2 \longleftrightarrow 1/2\) are near the field \(H \approx 1\) kOe.

The maxima on the high-field part of curve 2 permit one to estimate the parameter of the zero splitting \(D = 0.09\) cm\(^{-1}\) (\(D < h\nu\)).

Modeling the form of a absorption line of EPR spectrum that was presented in [9], shows that magnetic dipole transitions in polycrystalline substances result in occurrence only resonance lines 1 and 2. Non-resonance contribution experimentally observed and shown in figure 6 is not connected with magnetic dipole transitions.

The possible reason for this absorption is electric dipole transitions between vibronic states. Noncentral ions in under barrier and about barrier vibronic states have the electrical dipole moment \(d\) not equal to zero. For this reason these ions can interact with a microwave electrical field. In antinode of microwave electrical field the interaction \(-d \cdot E\) is several orders more than interaction \(-g \cdot \mu_B \cdot (S \cdot H)\) in antinode of microwave magnetic field. For this reason even the small electrical field, which affects a part of a sample near antinode of microwave magnetic field, can result in microwave field absorption greater than absorption caused by a microwave magnetic field.

5. Conclusions

Within the framework of the proposed model of the excited-state population, it is possible to explain the character of the temperature dependence of the integral intensity of the EPR spectrum resonance lines, the width of resonance for line 2.

At helium temperature, magnetic centers are "frozen" at the bottom of the potential well (or wells) which correspond to the largest value of the low-symmetry component of the crystalline field. Simultaneously, the anisotropy of the crystalline electrical field is maximum, and therefore line 1 is the most intense. As the temperature is increased, a number of magnetic centers pass to the excited vibronic states. The excited states of magnetic centers are less anisotropic than the ground states. The resonance line 2 of the EPR spectrum results from the population of weakly anisotropic excited states (figures 1-4, 8). Increasing the number of magnetic centers in the excited states leads to a decrease of the number of magnetic centers in the ground state. It is the cause of the increase of the intensity of resonance line 1 when the temperature is raised.

The unusual temperature dependence of the width of resonance line 2 (figure 13) is due to the change in distribution of magnetic centers over the excited states.

At high temperatures, the greater part of the magnetic centers is found in the excited states for which \(D << h\nu\). The EPR spectrum of these centers is characterized by the g value of the transition \(1/2 \longleftrightarrow -1/2\). The orientational averaging of the rest of transitions does not broaden the resonance line.

At low temperatures, a major portion of the magnetic centers is in states close to the ground state. The lower the excitation energy, the higher the value of the low-symmetry component of the crystalline field and the farther the resonance lines of transitions \(\pm 5/2 \longleftrightarrow \pm 3/2\) are located from the line of the transition \(1/2 \longleftrightarrow -1/2\). As a result of the orientational averaging over directions of the axes of symmetry, the contribution of the transitions \(\pm 5/2 \longleftrightarrow \pm 3/2\) and \(\pm 3/2 \longleftrightarrow \pm 1/2\) at low temperatures will result in the broadening of resonance line 2 (figure 15).
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