Hyperfine Interact
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Observation of the double optical-gamma resonance in Mössbauer spectra of ^{151}Eu in EuP_5O_{14} single crystal

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Abstract A theoretical analysis has been made and calculation procedures have been developed for describing the hyperfine structure of ^{151}Eu Mössbauer spectra and the effect of double optical-gamma resonance (DOGR) in the presence of hyperfine quadrupole interaction with an arbitrary symmetry of the electric field gradient tensor. An experimental setup was designed for DOGR-effect observation, incorporating the pumping argon laser and the tunable dye laser combined with the Mössbauer spectrometer on a common platform. The Mössbauer absorption spectra of $^{151}Eu^{3+}$ nuclei in single crystals of europium pentaphosphate, EuP_5O_{14}, have been measured at T = 5 K and 80 K under the absence and presence of optical pumping tuned to the $^7F_0-{}^5D_0$ electronic transition at the 578 nm wavelength. A simultaneous analysis of these spectra in terms of the spin Hamiltonian of hyperfine quadrupole interaction has allowed us to evaluate the DOGR-effect magnitude with the population of the excited ($^5D_0$) electronic state under optical pumping of about 10 %.

Keywords Mössbauer spectroscopy · Double optical-gamma resonance · Hyperfine quadrupole interaction

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1 Introduction

The well-known techniques such as ESR, NMR and Mössbauer spectroscopy allow us to study electronic and nuclear spin subsystems separately. On the other hands, the double resonance methods such as electron-nuclear double resonance (ENDOR) and gamma-magnetic resonance (GMR) allow us to study the interaction between electron and nuclear subsystems in the ground and excited nuclear states, respectively. A new technique, ESR-Mössbauer spectroscopy—double gamma-electron resonance (DGER), has been experimentally demonstrated at the Russian Research Center Kurchatov Institute [1]. It is particularly interesting to realize experimentally another double technique: double optical-gamma resonance (DOGR), combining the advantages of the most precise tools in modern physics—laser and Mössbauer spectroscopy. Up to now the feasibility of DOGR was considered mainly from the theoretical viewpoint [2–4]. It was shown that optical laser radiation can be used for nuclear transition control in the field of gamma-ray energy which inevitably must affect the shape of the Mössbauer absorption spectrum. Depending on the laser pumping power level, the following effects were predicted: changes in the population of hyperfine sublevels and nuclear polarization, modifications of the isomer shift for the Mössbauer transition, Rabi-splitting of Mössbauer spectra [5, 6].

The main objective of this study is a direct experiment to demonstrate a transformation of Mössbauer spectral shape under the influence of resonance laser pumping. Experiments on laser control of nuclear transitions may open up new vistas both in the development of the method itself and for accumulation of the experimental data for practicable implementation of a gamma-laser. We have performed the DOGR experiment on the ^{151}Eu nuclei in self-activated crystals of europium pentaphosphate, EuP_5O_{14}, where Eu^{3+} is not an impurity ion but makes a part of the matrix, and the ^{151}Eu isotope fraction in the natural mixture is 48 %. It ensures an essentially stronger resonance absorption effect as compared to that in diamagnetic crystals with impurity Mössbauer atoms [6]. Besides, a very narrow $^7F_0-{}^5D_0$electronic transition occurs on the 578 nm wavelength in this compound and it can be excited efficiently by the tunable dye laser. Therefore, $^{151}Eu^{3+}$ has the most promising characteristics both for optical and Mössbauer parts of the experiment.

A DOGR experiment is conceptually fairly simple. First, it is necessary to collect Mössbauer spectra on a sample in the absence of resonance laser field, and then to collect modified spectra in its presence. The difference in these spectra will represent the required DOGR-effect which then must be theoretically analyzed. From the physical point of view, the laser radiation effect on the sample under study leads to excitation of atomic electronic levels. Then the energy of hyperfine interaction of the nucleus with surrounding electrons as measured in the experiment is naturally defined both by nuclear parameters and the state of the electron shell. Thus, a change in the population of atomic levels under optical pumping must result in a transformation of Mössbauer absorption spectrum according with hyperfine parameters for the ground and excited electron states [4–10].

The lifetime of excited electron state $^5D_0$ in europium pentaphosphate crystals being fairly long of about 5 ms at room temperature [11–14]. Just this circumstance allows one to control the population of excited electronic state $^5D_0$ by optical pumping that must result in a transformation of the Mössbauer absorption spectra of ^{151}Eu nuclei with nuclear spins $I_g = 5/2$ and $I_e = 7/2$ in the ground (g) and excited

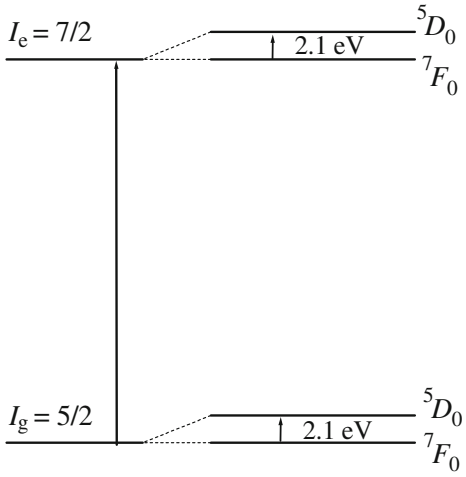

Fig. 1 Schematic of $^{151}Eu^{3+}$ electron-nuclear levels in the absence of hyperfine interaction

(e) states, accordingly. A pattern of $^{151}Eu^{3+}$ electron-nuclear levels in the absence of hyperfine interaction is shown in Fig. 1.

2 Hyperfine quadrupole interaction with an arbitrary symmetry of the EFG tensor for the ^{151}Eu nuclei

A hyperfine structure of $^{151}Eu^{3+}$ ion spectra in diamagnetic crystals is due mainly to an interaction of a nuclear quadrupole moment with an electric field gradient (EFG) caused by noncubic local environment of europium atoms. In this case, the Mössbauer absorption spectra can be described in terms of the Hamiltonians of hyperfine nuclear quadrupole interaction in the ground and excited states:

$$\hat{H}_q^{(\mathrm{g,e})} = q_{\mathrm{g,e}} \left[\hat{I}_z^2 - \frac{1}{3} I\,(I+1) + \frac{\eta}{3} \left(\hat{I}_x^2 - \hat{I}_y^2 \right) \right], \tag{1}$$

where $\hat{I}_i^{(\mathrm{g,e})}$ are the operators of nuclear spin projections on the direction of principal axes of the EFG tensor,

$$q_{\mathrm{g,e}} = \frac{3eQ_{\mathrm{g,e}}V_{zz}}{4I_{\mathrm{g,e}}\left(2I_{\mathrm{g,e}} - 1\right)} \tag{2}$$

are quadrupole interaction constants, e is the electron charge, $Q_{\mathrm{g,e}}$ are the nuclear quadrupole moments in the ground and excited states, V_{zz} is the largest (in the absolute value) of the main values of the EFG tensor,

$$\eta = \frac{V_{yy} - V_{xx}}{V_{zz}} \tag{3}$$

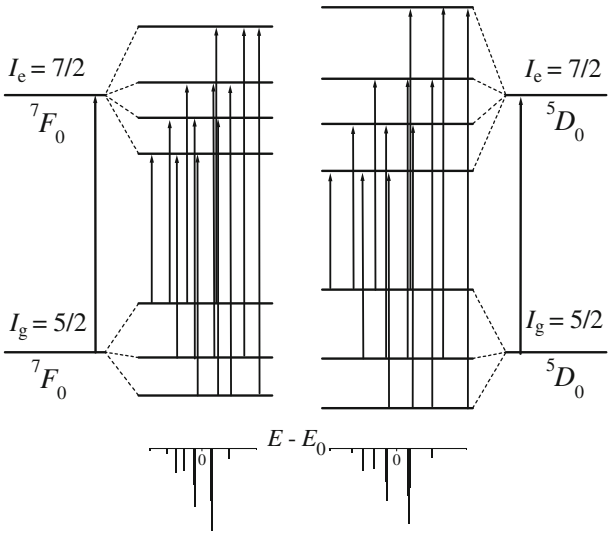

Fig. 2 Schematic of energy-level splitting of the ^{151}Eu nucleus in the ground and excited states in europium pentaphosphate and transitions between them for the ground ($^7F_0$) and excited ($^5D_0$) electronic states. Below the respective diagrams of energy spectra are given

is the asymmetry parameter of the EFG tensor. Let us note that the values of quadrupole shifts of the levels are proportional to the quantity q_g, since, according to expression (2),

$$q_e = q_g \frac{Q_e I_g (2I_g - 1)}{Q_g I_e (2I_e - 1)}. \tag{4}$$

In particular, for the ^{151}Eu nuclei:

$$q_e \approx 0.675 q_g. \tag{5}$$

In this case, the ground state is split into three levels, and the excited state—into four levels.

The eigenvalues of Hamiltonians (1) determine splitting of nuclear energy levels in the ground and excited states and, consequently, the energy of gamma transition between the split levels, i.e., the hyperfine structure of Mössbauer spectra. In particular, from the analysis of the experimental optical data, the values of spin Hamiltonian parameters of hyperfine quadrupole interaction (1) for a ground state of ^{151}Eu nuclei in $EuP_5O_{14}$for both the electronic states have been estimated: q_g = 6.26 MHz (= 0.36 mm/s), η = 0.59 for the $^7F_0$ state and q_g = 8 MHz (= 0.46 mm/s), η = 0.62 for the $^5D_0$ state [13]. These parameters ensure the calculation of energy-level splitting of a ^{151}Eu nucleus in the ground and excited states, and also absorption spectra for both the electron states $^7F_0$ and $^5D_0$ shown in Fig. 2.

In this case, all the possible transitions between nuclear energy levels in the ground and excited states are allowed ones, and the hyperfine structure consisting of 12 components (see Fig. 2) should be observed in the ^{151}Eu absorption spectrum at $\eta \neq 0$. Since the values of the EFG tensor asymmetry parameters are similar for the electron states $^7F_0$ and $^5D_0$, the major difference in the hyperfine structure spectra for these states, as seen from this figure, is due to the magnitude of hyperfine splitting which is appreciably smaller for the $^7F_0$ state. Hence, the hyperfine structure for the exited electronic state should be resolved better than that for the ground state. This

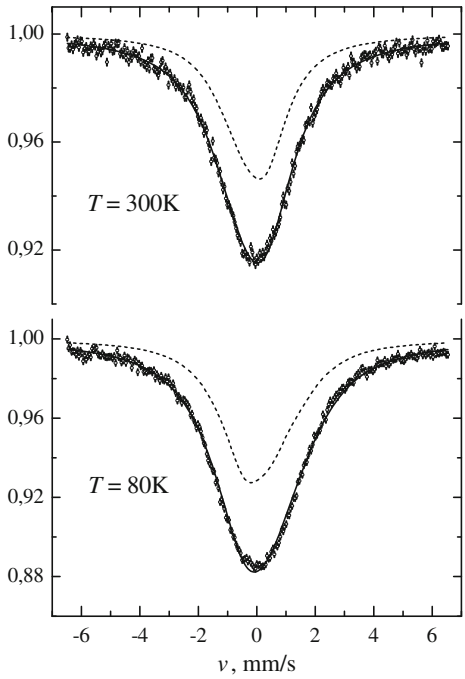

Fig. 3 Experimental Mössbauer absorption spectra of ^{151}Eu nuclei in the polycrystalline europium pentaphosphate (*dots and vertical dashes*) at room temperature (*top*) and liquid nitrogen temperature (*bottom*), and the respective absorption spectra (*solid lines*) calculated in terms of Hamiltonian (1). *Dotted lines* show the absorption spectra in the sample without regards to a convolution with the Lorentzian source line shape at a reduced by half scale

difference is confirmed by direct calculations of Mössbauer absorption spectra, and it is the primary factor for observing the DOGR-effect.

One more factor which must in the general case differentiate Mössbauer spectra of the ground and excited electron states is an isomeric chemical shift resulting from an electrostatic interaction of atomic electrons with the nucleus of finite dimensions. In the case of gamma-rays, the difference in the electrostatic energy for two isomeric levels with different nucleus dimensions must lead to a nuclear transition energy shift [6]. Calculation of respective isomer shifts from the first principles is a separate, often incompletely defined problem; however, by virtue of the fact that this parameter is integrated over the spectrum, the values of isomer shifts are definable with good accuracy from an analysis of experimental Mössbauer spectra.

3 Mössbauer spectra of ^{151}Eu nuclei in a polycrystalline EuP_5O_{14} sample

At the first stage of our investigations, we employed a spectrometer operating in a uniform acceleration mode to measure Mössbauer spectra of ^{151}Eu nuclei in a polycrystalline EuP_5O_{14} sample at the room and nitrogen temperatures with the use of a 30 mCi radioactive $^{151}SmF_3$ source and a gamma-quanta scintillation detector. The experimental spectra plotted in Fig. 3 show that all the europium ions are in a trivalent state (no signal from divalent europium was detected within the attained statistical accuracy of 0.1 % in the velocity range $\pm$15 mm/s). Each spectrum looks like a slightly unsymmetrical broadened line testifying to the presence of a

quadrupole splitting. It stems from a low monoclinic symmetry of both the crystal lattice and oxygen ions in the closest environment of europium atoms.

At the initial stage, the experimental spectra were analyzed in the simplest approximation of a single Lorentzian line. As a result of the analysis, the following parameters (without regard for a hyperfine quadrupole split) were estimated to be: the isomer shifts $\delta = -0.044(5)$ and $0.033(4)$ mm/s, and the line widths $\Gamma = 3.01(2)$ and $3.53(2)$ mm/s for the spectra taken at $T = 300$ and 80 K, accordingly. (In brackets mean-root-square errors in the parameters are given.) It appeared that such spectrum characterization is quite accessible for a spectrum measured at the room temperature, and testifies first of all to the fact that a fairly large effective linewidth counterbalances the asymmetrical spectra profile in the presence of a weak quadrupole interaction (see Fig. 3). Moreover, there is an additional $\Delta\Gamma_s$ line broadening related to resonance quanta self-absorption in the radiation source. At the same time, the characterization of the experimental spectrum measured at $T = 80$ K has appeared clearly unsatisfactory, and the noticeably wider line for this spectrum as compared to that measured at room temperature evidences unambiguously in favor of the increase of the quadrupole interaction constant with temperature decreasing. This fact is not surprising by itself and it is specific to the majority of Mössbauer spectra in the presence of a hyperfine quadrupole interaction.

It is obvious that a more accurate spectra analysis is to be made within the limits of a spin Hamiltonian (1), but it is first necessary to define an additional line broadening in the source and to make a correction of the spectra for the finite absorber thickness. The analysis of both experimental spectra at $T = 80$ and 300 K in the frame of the procedures of Mössbauer spectra precision analysis DISCVER [15, 16] and effective account for absorber thicknesses [17] with a variable additional linewidth of $\Delta\Gamma_s$ in the source has allowed us to estimate the spread of the additional widening $\Delta\Gamma_s = 0.9$ (1) mm/s in the source and to carry out an accurate spectra correction with regard to the absorber thickness. The experimental spectra corrected with regard to the absorber thickness are shown in Fig. 3.

Then we analyzed the corrected spectra in terms of the spin Hamiltonian (1) and the respective analytic form of the spectrum:

$$\sigma(\omega) = \frac{\sigma_a \Gamma_0^2}{4} \sum_{i=1}^{3} \sum_{j=1}^{4} \frac{A_{ij}}{\left(\widetilde{\omega} - \omega_{ij}\right)^2 + \frac{\Gamma_0^2}{4}}. \tag{6}$$

Here σ_a is the effective absorber thickness, Γ_0 is the resonance line natural width, $\widetilde{\omega} = \omega - E_0/\hbar$, E_0 is the resonance transition energy,

$$\omega_{ij} = \frac{\left(E_j^{(e)} - E_i^{(g)}\right)}{\hbar} \tag{7}$$

$E_i^{(g,e)}$ are the eigenvalues of Hamiltonians (1), while the transition intensities A_{ij} at the resonance frequencies (7) are defined by Clebsch-Gordan coefficients and the corresponding (to the eigenvalues) wave functions of Hamiltonians (1) in the space of nuclear spin projections in the ground and excited states. In the course of experimental spectra fitting in terms of the least-square method, adjustable parameters were q_g, η and δ for each spectrum. As initial parameters it was natural to choose values of $q_g = 0.36$ mm/s and $\eta = 0.59$ for the state $^7F_0$ derived from the experimental optical data analysis [13]. The resulting calculated spectra are shown

in Fig. 3 by solid lines. For illustration purposes and more convenient comparison with model calculations, this figure also shows the absorption spectra in the sample (without convolution with the Lorentzian source line shape).

As to the spectrum measured at $T = 300$ K, the fitting has resulted in the values of $\delta = -0.065(6)$ mm/s, $q_g = 0.32(5)$ mm/s and $\eta = 0.6(1)$ which are in good agreement with the optical data and adequately describe the experimental Mössbauer spectrum (Fig. 3). Meanwhile, the absorption spectrum in the sample manifests clearly the asymmetry shape of the spectrum. At the same time, the results of the analysis of the experimental spectrum measured at $T = 80$ K demonstrate qualitatively different quadrupole interaction parameters with values of $\delta = 0.005(5)$ mm/s, $q_g = -0.38(3)$ mm/s and $\eta = 0.65(6)$. The nontrivial nature of this effect is the fact that the quadrupole interaction constant not only increased in magnitude with temperature decreasing but also reversed in its sign. This fact confirms the absorption spectrum in a sample (with no convolution with the source line shape) which clearly demonstrates the spectral asymmetry of the opposite sign as compared to the spectrum measured at $T = 300$ K.

The Mössbauer spectroscopy provides many instances of such behavior with an abrupt change of hyperfine quadrupole interaction parameters which, as a rule, testifies to the presence of a structural transition in the substance under study. A feasibility of such low-temperature transition in EuP_5O_{14} crystals is independently proven to be true by thorough measurements of this compound with the methods of emission and absorption optical spectroscopy [11]. Proceeding from the results of their analysis, geometrical models were suggested of local environment of the Eu^{3+} ion with the anticipated transition from a highly symmetric (D_{4d}) configuration of the Archimedes antiprism to a face-centered isosceles prism (symmetry C_{2v}) via intermediate phases. More detailed structural investigations are beyond the limits of this work, nevertheless, the discovered peculiarity, undoubtedly, is of high significance for the DOGR-effect observation since the low-temperature spectra should demonstrate better resolution (due to larger values of the quadrupole interaction constant).

4 DOGR-effect measurements in europium pentaphosphate, EuP_5O_{14}

For carrying out the DOGR experiment at low temperatures, an experimental setup was designed and assembled (Fig. 4). A platform with a cryostat, a Mössbauer velocity transducer and a gamma-quanta scintillation detector installed on it was mounted on a plate with lasers. The axes of the optical and gamma beams were adjusted so that the two beams are perpendicular to each other. The largest and most transparent EuP_5O_{14} crystals from the available batch of crystals were selected. They were cut to the thickness of about 1 mm and were polished down to the thickness of about 0.3 mm. A mosaic target of about 9×6 mm^2 in cross section from ten such crystals was mounted on an aluminum foil disk covered by a viscous liquid for measurements of optical absorption- and Mössbauer spectra. The sample was placed at an angle of 45° to both beams and was set into a copper holder of a cryostat "cold finger" on an aluminum substrate in vacuum. The distance from the source to the detector was about 10 cm and the counting rate of 21.6 keV Mössbauer gamma-quanta was of the order of $5{\cdot}10^2$ pulses/h·channel. The spectra measured with laser

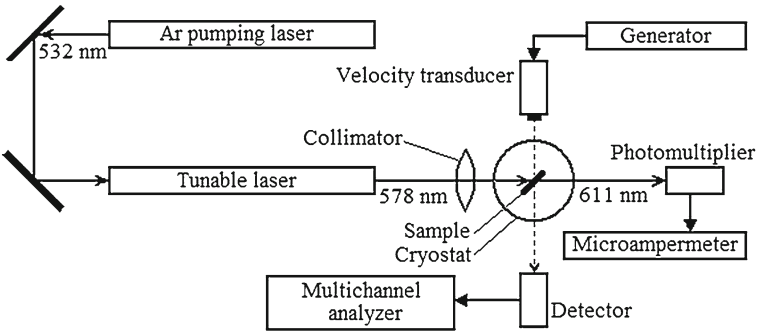

Fig. 4 Schematic diagram of the experimental set-up

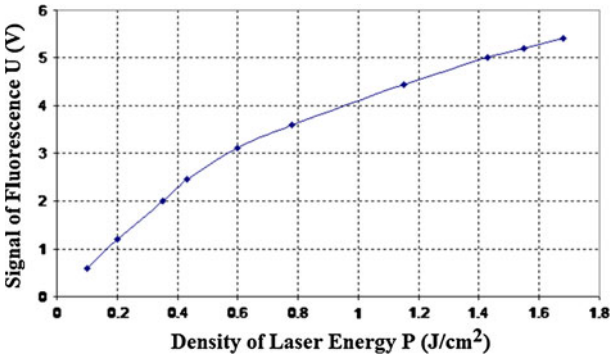

Fig. 5 Fluorescence signal dependence on the laser surface energy density (P) at $T = 80$ K. The saturation starts at P$\sim$0.4 J/cm^2

pumping at the wavelength of 578.2 nm and without laser pumping were alternately recorded in different groups of Mössbauer spectrometer memory with 4-hr duration of each mode. The net measurement time of each spectrum was 1 week. A tandem of "argon laser SP-171-18 – tunable dye-laser SP-380D" was adjusted to the absorption baseline (578.4 nm in vacuum). Manual adjustment to this line was controlled by measurements of the maximum of the optical fluorescence that had passed through a 1 mm hole aperture in aluminum foil at the wavelength of 611.5 nm as detected by a scintillation detector. The adjustment accuracy was about 3 % of the peak signal. The laser radiation output intensity for the multimode operation was $\sim$1 W/cm^2 (with the diameter of the optical and gamma beams of about 6 mm). Under these conditions, appreciable transition saturation at 80 K started already at the power level of 0.4 W/cm^2 (Fig. 5). The power absorbed by the crystal is assessed to be $\sim 10^{-2}$ W/cm^2 from the results of optical absorption spectra measurement.

The experimental absorption spectra of ^{151}Eu nuclei in the mosaic EuP_5O_{14} crystal sample measured at $T = 5$ K and 80 K in the absence and presence of optical pumping are shown in Fig. 6. Then we have performed a simultaneous analysis of each pair of the spectra taken at a given temperature within the Hamiltonians (1) and Eqs. (6) and (7) where an angular distribution function for radiation intensity for the mosaic crystal sample and a given direction of the wave vector of the gamma-ray beam has been taken into account. The latter is reduced to the averaging of the absorption spectrum over a random distribution of the x- and y-axes in the plane

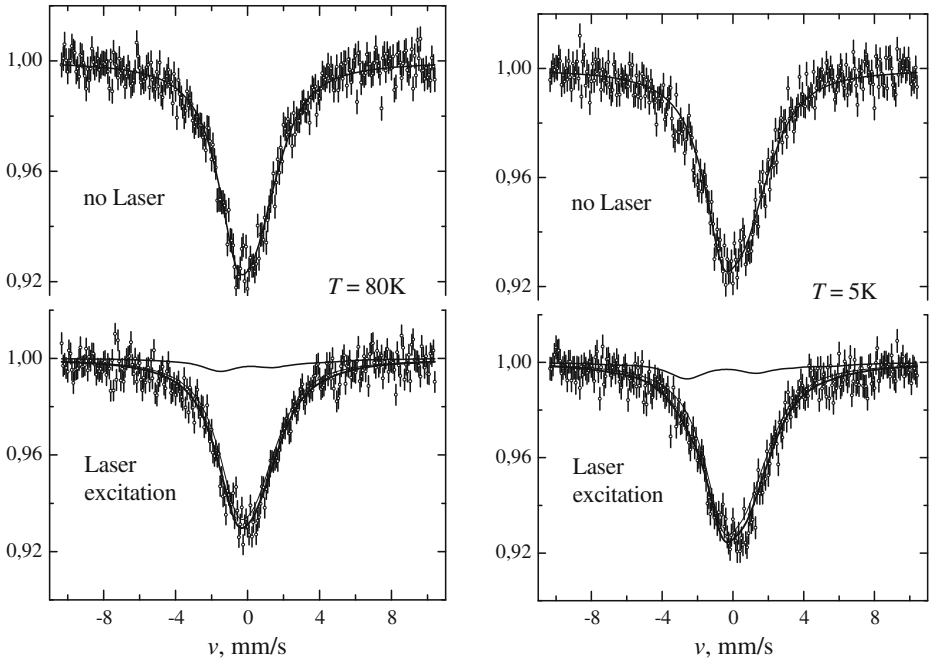

Fig. 6 Experimental ^{151}Eu Mössbauer absorption spectra of the europium pentaphosphate monomosaic measured at $T = 80$ K (*left*) and 5 K (*right*) in the absence (*top*) and presence (*bottom*) of optical pumping. Also plotted are the resulting and partial absorption spectra for the ground ($^7F_0$) and excited ($^5D_0$) electron states (*solid lines*)

of the monoclinic mosaic sample with the z-axes normal to the sample plane, which results in the following expressions for the transition intensities (see, e.g., [18, 19]:

$$A_{ij} = \sum_{\zeta} \sum_{\substack{m_g m_e \\ \widetilde{m}_i \widetilde{m}_j}} C_\zeta \, V^{(\zeta)+}_{\widetilde{m}_i \widetilde{m}_j} \langle \widetilde{m}_i \mid m_g \rangle \langle m_e \mid \widetilde{m}_j \rangle \, V^{(\zeta)}_{m_e m_g}. \tag{8}$$

Here, m_g and m_e are the nuclear spin projections onto the hyperfine field direction for the ground (g) and excited (e) nuclear states, $\widetilde{m}_i$ and $\widetilde{m}_j$ are the nuclear spin projections onto the quantization axes for which the operators representing the Hamiltonians (1) are diagonal, $V^{(\zeta)}_{m_e m_g}$ are the matrix elements for the operator of interaction of the gamma-quantum with polarization ζ and the nucleus, the sum over random polarizations in our case is reduced to that over $\zeta = x, y, z$ with the coefficients

$$C_z = \frac{1}{2} \sin^2 \theta_\gamma, \, C_{x,y} = \frac{1}{4} \left(1 + \cos^2 \theta_\gamma\right), \tag{9}$$

where θ_γ is the angle between the gamma-ray beam direction and the normal to the sample's surface.

Each pair of the spectra taken at a given temperature has been analyzed within the least-square method with variable parameters q_g and η of the hyperfine quadrupole interaction for the ground electronic state, $^7F_0$, whose contribution is present in both the spectra, and the additional contribution from the excited state, $^5D_0$, in the

spectrum measured under laser pumping. We also varied isomer shifts δ for each partial spectrum. As a result, the following values of the parameters were obtained at $T = 5$ K and 80 K: $\delta = 0.06(3)$ and $-0.05(2)$ mm/s, $q_g - 0.49(2)$ and $-0.40(2)$ mm/s, $\eta = 0.6(2)$ and 0.6(2) for the $^7F_0$ state, and $\delta = 0.05(4)$ and $-0.04(4)$ mm/s, $q_g - 1.6(3)$ and $-1.2(5)$ mm/s, $\eta = 0.6(3)$ and 0.6(3) for the $^5D_0$ state which ensures a correct characterization of the experimental spectra (Fig. 6). The partial contribution (spectral area) of the excited state $^5D_0$ in the spectrum measured under conditions of laser pumping was evaluated to be $C_{D0} = 0.11(2)$ and 0.10(3) at $T = 5$ K and 80 K which is a direct evidence of the DOGR-effect observation in our experiment. It should be noted that the analysis of the dependence of fluorescence signal at the 611.5 nm wavelength on the surface density of the P pulse energy with the start of saturation at $P \sim 0.4$ J/cm^2, as shown in Fig. 5, is also indicative of the fact that level $^5D_0$ population at $P \approx 1$ W/cm^2 is approximately 10 %.

Thus, the qualitative correlation of the theoretically anticipated and experimentally observed evolution of the Mössbauer spectrum profile under the laser excitation effect and the quantitative coincidence of the data of optical and Mössbauer measurements of about 10 %-population of the exited atomic state testify to the observation of a double optical-gamma resonance effect in our experiment.

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