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Optical spectroscopy of the adducts of europium tris(dipivaloylmethanate) with derivatives of 1,10-phenanthroline

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Abstract

Spectroscopic investigations of the mutual influence of inequivalent ligands were undertaken using complex adducts of europium tris(dipyvaloylmethanate) with derivatives of 1,10-phenanthroline, $Eu(DPM)_3 \cdot Ph$. The choice of a series of related $Eu(DPM)_3 \cdot Ph$ compounds provided the opportunity to study the dependence of the spectroscopic characteristics on the value of the effective charges at the atoms in the nearest surroundings of Eu^{3+} which, in turn, depends on steric factors and the donor–acceptor properties of the ligands. It was shown that steric factors are as significant as donor–acceptor properties of the ligands in the creation of the complexes. The changes in the strength and symmetry of the crystal field were evaluated from the Stark splittings of the Eu^{3+} transitions in luminescence spectra. Crystal field parameters were calculated. Two subgroups of compounds with similar spectra were distinguished. The relative efficiency of the excitation of Eu^{3+} luminescence through the ligand absorption bands and the relative luminance of the compounds were investigated. Changes of the ligand vibration frequencies and the strength of the electron–phonon interaction with various ligands were studied by IR and vibronic spectra.

Keywords: Crystal and ligand fields; Electron–phonon interaction; Luminescence

1. Introduction

In the past few years numerous investigations have been carried out on different europium β-diketonates with donor ligands [1–11]. The present work reports the results of spectroscopic investigations of the mutual influence of inequivalent ligands in complex adducts of europium tris(dipyvaloylmethanate) ($Eu(DPM)_3$) with derivatives of 1,10-phenanthroline (Ph) of formula $Eu(DPM)_3 \cdot Ph$. The DPM radicals exhibit appreciable donor properties (Taft's constant σ^* for two identical radicals $-C(CH_3)_3$ is -0.4), and these radicals have a comparatively large size. The variation of the substituents in the 1,10-phenanthroline molecule determines the donor–acceptor properties of the ligand, changing the distribution of the electronic density in the molecule as well as the effective charges on the nitrogen atoms. This leads to changes in the strength of the bonds of Ln^{3+} with ligands and to variations of the metal–ligand distances. However, steric factors (form and dimensions of the ligands) can prevail and these characteristics will not be as dependent on the ligand donor–acceptor properties as expected. Hindering the coordination of one kind of ligand, steric factors can promote the coordination of another kind of ligand.

The choice of a series of related $Eu(DPM)_3 \cdot Ph$ compounds provides the opportunity of tracing the changes in the spectroscopic characteristics of the materials, which are associated with the values of the effective charge at the atoms in the nearest surroundings of Eu^{3+} and with the distribution of the electronic density in the ligands. The changes in the strength and symmetry of the crystal field is derived from Stark splittings of the electronic levels of Eu^{3+} and the relative intensity of the electronic transitions. From luminescence excitation spectra the relative efficiency of the excitation of Eu^{3+} through the absorption bands of the ligands can be determined. The changes of the ligand vibration frequencies and the strength of the electron–phonon interaction can then be investigated. The data obtained are compared with the corresponding characteristics of other β-diketonates as well as with X-ray data.

2. Experimental

A series of adducts of europium tris(dipyvaloylmetha-

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nate) with derivatives of 1,10-phenanthroline of formula $Eu(DPM)_3 \cdot Ph$ was synthesised. Ph is 3,4,7,8-tetramethyl-phenanthroline (Tmphen), 5-methyl-phenanthroline (Mphen), phenanthroline (Phen), 5-phenyl-phenanthroline (Phphen), 4,7-diphenyl-phenanthroline (Dphphen) and 5-nitro-phenanthroline (Nphen). Phenanthroline derivatives are ordered in such a way that their acceptor properties increase. Taft's constants for radicals are: $\sigma^*(CH_3) = 0$, $\sigma^*(H) = +0.49$, $\sigma^*(C_6H_5) = +0.6$ and $\sigma^*(NO_2) = +3.9$. The compounds were obtained by mixing ethanol or ethanol–water–acetone solutions of $Eu(DPM)_3$ with an ethanol solution of heterocyclic diimine. Mixing of the chloroform solutions of both reagents [12] was also used in synthesis.

It is known that the structure of the $Eu(DPM)_3$ precursor compound consists of noncentrosymmetric dimers [13]. Two europium ions are connected with oxygen bridges, thus each Eu^{3+} has a coordination number of seven. The coordination polyhedron of the lanthanide ion is a distorted trigonal prism. The structure of adducts $Eu(DPM)_3 \cdot Ph$ is described in Ref. [12] and is analogous to that of other lanthanide β-diketonates with heterocyclic diimines [14–16]. The coordination polyhedron of Eu^{3+} in these compounds is a distorted square antiprism, formed by six oxygen and two nitrogen atoms (coordination number eight).

During the synthesis of some $Eu(DPM)_3 \cdot Ph$ compounds a photochemical reaction was observed, as reported previously [17]. A two-stage photochemical reaction in a solution of europium acetate or other salt with 1,10-phenanthroline was observed in the latter report. In the second, reversible stage of the reaction, an intense green coloration of the reaction products was observed. This photochemical reaction hindered formation of the complexes, in particular $Eu(DPM)_3 \cdot Nphen$.

The luminescence, excitation of luminescence as well as IR absorption spectra of $Eu(DPM)_3 \cdot Ph$ were recorded. The luminescence and excitation of luminescence spectra were measured on a SLM Aminco SPF 500 spectrofluorimeter and on an ISP-51 spectrograph at 77 and 300 K. IR spectra in the region 70–4000 cm^{-1} were recorded using Brucker FS 88 FTIR and UR-20 spectrometers. UV absorption spectra of the phenanthroline derivatives and of some of the adducts in ethanol solution were obtained with a Specord UV–VIS at 300 K.

3. Results and discussion

3.1. Luminescence spectra

Let us consider the luminescence spectra presented in Figs. 1 and 2. Excessive numbers of Stark components in the electronic transitions of Eu^{3+}, in particular in $^5D_0$–$^7F_1$, in spectra of $Eu(DPM)_3$ were detected (Fig. 1). This is due to the nonequivalence of Eu^{3+} centres in the dimer, similar to the case of other dimers [18–22]. The maximum number of Stark components of the electronic transitions in $Eu(DPM)_3 \cdot Ph$ (Fig. 2) points to a low (rhombic) symmetry of the charge arrangement around Eu^{3+}. The features of the Stark splitting in luminescence spectra of the compounds from the $Eu(DPM)_3 \cdot Ph$ family are the same and are like the spectral pattern of the more general series of compounds $Eu(\beta)_3 \cdot Ph$, with β-β-diketones [8,9], so the structures of these compounds are isomorphous. No monotonous changes of the spectra (due to variation of the donor–acceptor properties from Tmphen to Nphen) were observed. Judging from the spectra, adducts with the

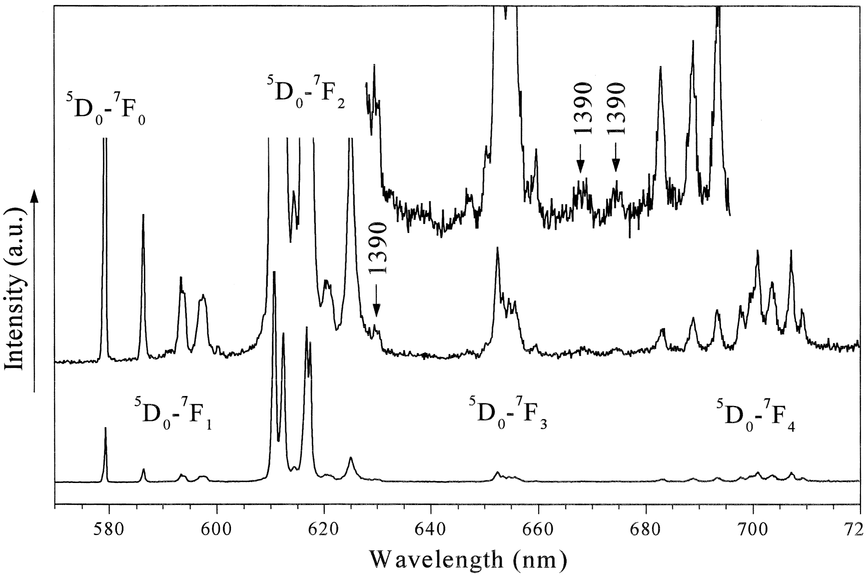

Fig. 1. Luminescence spectra of $Eu(DPM)_3$ at 77 K.

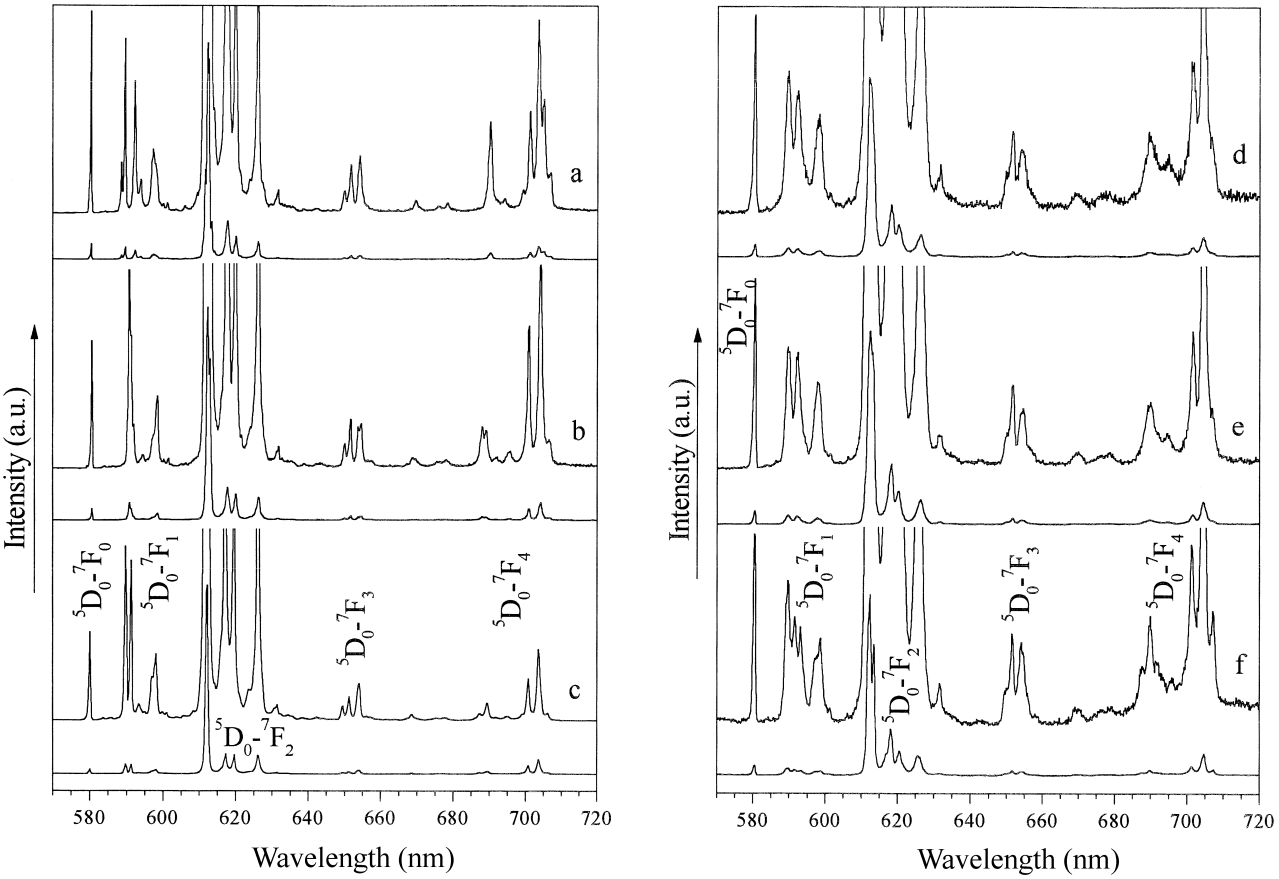

Fig. 2. Luminescence spectra of $Eu(DPM)_3 \cdot$Tmphen (a), $Eu(DPM)_3 \cdot$Mphen (b), $Eu(DPM)_3 \cdot$Phen (c), $Eu(DPM)_3 \cdot$Phphen (d), $Eu(DPM)_3 \cdot$Dphphen (e) and $Eu(DPM)_3 \cdot$Nphen (f) at 77 K.

relatively small molecules Mphen and Phen form a separate subgroup (Fig. 2b,c) with minimum distortions of the symmetry of the Eu^{3+} surroundings. In $Eu(DPM)_3 \cdot$Mphen the Eu–N bonds are strengthened slightly because of the increase of the donor properties of the heterocyclic diimine and, correspondingly, the Eu–O are weakened. The spectra of adducts containing phenyl derivatives of phenanthroline demonstrate that these compounds form another separate subgroup (Fig. 2d,e). These spectra have Stark splittings and an intensity distribution unlike those of the first subgroup and the lines are broadened. The latter effect probably correlates with the variety of positions of the phenyl rings in relation to the plane of the three condensed rings of the phenanthroline molecule. $Eu(DPM)_3 \cdot$Nphen, having two luminescence centres (Fig. 2f), belongs to the same group. The compound $Eu(DPM)_3 \cdot$Tmphen also has two inequivalent metal cation sites (Fig. 2a). The spectra of these two Eu^{3+} cations reminds us somewhat of the spectra of europium in compounds of two different subgroups with stronger and weaker Eu–O bonds. At 300 K the bonds equalize and the compound can be classified as belonging to the second subgroup.

Table 1 presents the empiric crystal field parameters (CFP) (B^k_q in Wybourne notation [23]) calculated from experimental data for the energies of the Stark splittings of the $^7F_1$–$^7F_4$ levels of Eu^{3+} in a crystal field assuming C_{2v} point symmetry of the investigated compounds and using the root mean square criterion. The root mean square deviation of the calculated data for the Stark splitting from experimental data was not more than 10 cm^{-1}. In the cases

Table 1
Crystal field parameters (cm^{-1}), 77 K

Compound	B^2_0	B^2_2	B^4_0	B^4_2	B^4_4	B^6_0	B^6_2	B^6_4	B^6_6
$Eu(DPM)_3 \cdot$Tmphen	655	124	−1314	−815	384	−14	−320	−72	70
$Eu(DPM)_3 \cdot$Mphen	720	18	−1296	−788	524	100	−128	−15	−51
$Eu(DPM)_3 \cdot$Phen	701	94	−1222	−770	382	335	348	−347	220
$Eu(DPM)_3 \cdot$Phphen	612	115	−1485	−683	570	297	−179	−143	82
$Eu(DPM)_3 \cdot$Dphphen	589	89	−1579	−750	541	108	−241	−217	56
$Eu(DPM)_3 \cdot$Nphen	702	217	−1183	−532	652	−25	−245	−678	100

of adducts with Tmphen and Nphen, Eu^{3+} luminescence centres with more intense Stark components were observed. CFP can be used to monitor the crystal field with variation of the ligand. This variation mainly changes the value of the nonaxial (rhombic) component of the crystal field harmonics of the second order. This value correlates with the sizes of the substituents and reaches a minimum for compounds with methyl-phenanthroline and a maximum for compounds with more bulky phenyl and tetramethyl substituents.

Recently, we studied the spectra of the adducts of many β-diketonates with phenanthroline derivatives [8,9] and found that the spectra of $Eu(DPM)_3 \cdot Phen$ and $Eu(TTFA)_3 \cdot Nphen$ looked very similar. Weakening of the bond with acceptor Nphen promotes strengthening of the bonds of europium with TTFA ($\sigma^*(CF_3) = +2.6$, $\sigma^*(C_4H_3S) = +1.35$) and that makes the effective charge distribution on atoms of the nearest surroundings of Eu^{3+} in $Eu(TTFA)_3 \cdot Nphen$ similar to that of $Eu(DPM)_3 \cdot Phen$, where the Eu–O bonds are relatively strong due to the donor properties of DPM, and the Eu–N bonds are relatively weak.

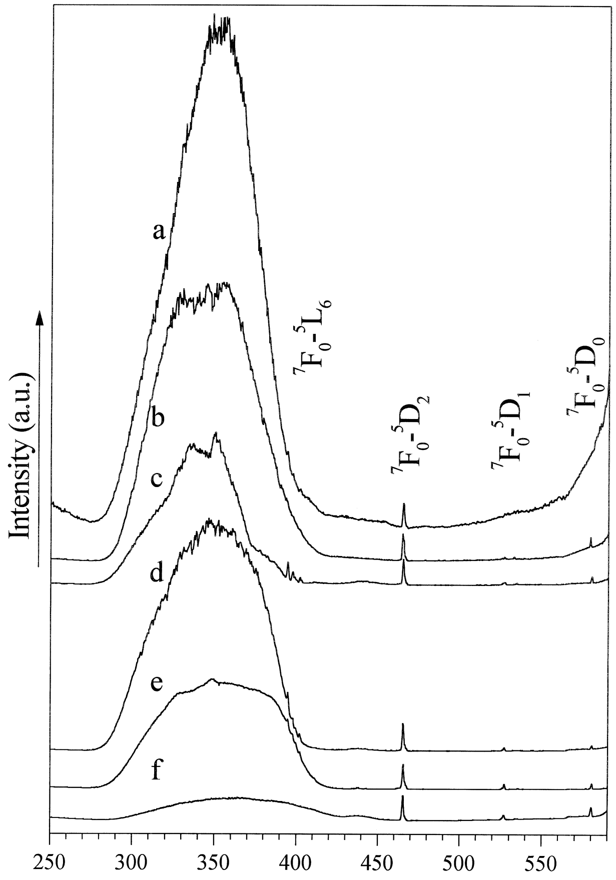

Fig. 3. Luminescence excitation spectra of $Eu(DPM)_3 \cdot Phphen$ (a), $Eu(DPM)_3 \cdot Dphphen$ (b), $Eu(DPM)_3 \cdot Tmphen$ (c), $Eu(DPM)_3 \cdot Mphen$ (d), $Eu(DPM)_3 \cdot Phen$ (e) and $Eu(DPM)_3 \cdot Nphen$ (f) at 77 K.

3.2. Luminescence excitation spectra

Excitation spectra of $Eu(DPM)_3 \cdot Ph$ are shown in Fig. 3. Let us try to analyse the changes in these spectra with variation of the phenanthroline radical. They exhibit narrow lines related to f–f transitions of Eu^{3+} and a wide band due to ligand transitions. The intensity of the short-wavelength region (<350 nm) of the ligand absorption band is filtered out by the glass walls of the sample ampoule. In the $Eu(DPM)_3$ excitation spectrum, in addition to the intense absorption band in the region about 290 nm, there are weak bands at about 380 and 400 nm. In this region of the $Eu(DPM)_3$ solution absorption spectrum, two long-wavelength bands, at 350 nm and a very weak one at 400 nm, are observed [24]. As these absorption bands are absent in the spectrum of the corresponding terbium compound, they were attributed by the authors to ligand-to-Eu charge transfer bands.

The most long-wavelength absorption bands of 1,10-phenanthroline (allowed transitions) are situated at 326 nm (n–π* transition) and at 339 nm (very weak π–π* transition) [25]. These bands practically do not shift in transition to methyl derivatives of phenanthroline and shift to the long-wavelength side by about 15–20 nm in transition to phenyl derivatives due to the lengthening of the system of conjugated π-bonds. In the nitro-phenanthroline spectrum the border of the broad absorption band of the ligand reaches 415 nm due to insertion of the nitro-group into the molecule. We observed these bands in the absorption spectra of heterocyclic diimine and its adduct solutions as well as in the excitation spectra of $Eu(NO_3)_3 \cdot Ph_2$. On coordination, as a rule, a supplementary long-wavelength shift of absorption bands by about 10–15 nm is observed.

Information on the positions of the first triplet levels of the phenanthroline derivatives can be obtained from the phosphorescence spectra of gadolinium β-diketonate adducts at 77 K. We did not have gadolinium DPM adducts, but had TTFA adducts. In particular, the short-wavelength edge of the phosphorescence band of $Gd(TFFA)_3 \cdot Phen$ lies at 485 nm, that of $Gd(TFFA)_3 \cdot Tmphen$ lies at 490 nm, and for $Gd(TFFA)_3 \cdot Nphen$, at 540 nm.

The most effective $Eu(DPM)_3 \cdot Tmphen$ excitation was obtained through the absorption bands of Tmphen at 335 and 350 nm (the excitation spectrum of $Eu(DPM)_3 \cdot Tmphen$ practically coincides with that of $Eu(NO_3)_3 \cdot Tmphen_2$). Bands of ligands in excitation spectra of Eu^{3+} widen in spectra of $Eu(DPM)_3 \cdot Mphen$ and $Eu(DPM)_3 \cdot Phen$. The increase in intensity in the 350–400 nm region is probably associated with the participation of DPM molecules in the excitation processes.

In the case of adducts of $Eu(DPM)_3$ with phenyl derivatives of phenanthroline the efficiency of the excitation through the phenyl-phenanthroline absorption bands is the highest. This can probably be attributed to the lowering of the triplet level of the phenanthroline derivatives, which makes the process of the energy transfer to the $^5D_0$ level of Eu^{3+} comparatively efficient. In the case of $Eu(DPM)_3\cdot$Nphen, excitation through the ligand bands is ineffective in comparison with excitation through the Eu^{3+} absorption bands because the position of the triplet level of Nphen is too low in comparison with the $^5D_0$ level.

Lattice expansion, weakening of bonds and increase of quenching at 300 K lowers the efficiency of the excitation through the ligands in cases of adducts with Phen, Mphen and Phphen. For adducts with Tmphen, which can have a lower rate of triplet–triplet annihilation due to substantial steric factors, the efficiency of excitation through ligands at room temperature is practically the same as at 77 K, and for the adduct with Dphphen it slightly increases. The luminance of $Eu(DPM)_3\cdot$Dphphen at 300 K, obtained with 365 nm excitation, is the highest, and that of $Eu(DPM)_3\cdot$Phen and $Eu(DPM)_3\cdot$Mphen is lower by a factor of two, and the lowest luminance (by a factor of about an order of magnitude) is exhibited by $Eu(DPM)_3\cdot$Nphen.

3.3. Vibronic interaction

Luminescence spectra recorded at a high sensitivity of registration provide an opportunity to demonstrate a series of vibronic bands (Figs. 1 and 2). Recently, we showed that vibronic sidebands of the electronic transitions in spectra of europium β-diketonates are formed mostly by β-diketone vibrations [8]. β-Diketonate vibrations are not characteristic vibrations. M–O bonds with participation of deformations of the chelate ring and of radicals give the most substantial contribution to vibrations in the region 200–700 cm^{-1}. The regions 1000–1400 cm^{-1} and 1400–1600 cm^{-1} contain vibrations of $C\overset{\cdots}{-}O$, $C\overset{\cdots}{-}C$, C–C and $C\overset{\cdots}{-}C$, $C\overset{\cdots}{-}O$, respectively (compare, for example, with the assignments of bands in the IR spectra [26–28]). In spite of a large number of studies of IR spectra, exact interpretation is still difficult due to the complex structure of the β-diketone molecules.

In Figs. 1 and 2 the most intense vibronic bands, related to the $^5D_0$–$^7F_0$ and $^5D_0$–$^7F_2$ transitions in europium luminescence spectra and attributed to vibrations of the β-diketonate chelate ring with the highest contribution of $C\overset{\cdots}{-}O$ and $C\overset{\cdots}{-}C$ stretching vibrations [28], are observed at about 630–632 and 668–670, 675–680 nm (in Fig. 1 these bands are marked by arrows). The frequency of this vibration is 1390 cm^{-1} in the case of $Eu(DPM)_3$. It increases from 1370–1380 cm^{-1} for $Eu(DPM)_3\cdot$Phen to 1450 cm^{-1} for $Eu(TTFA)_3\cdot$Nphen. The relation of the

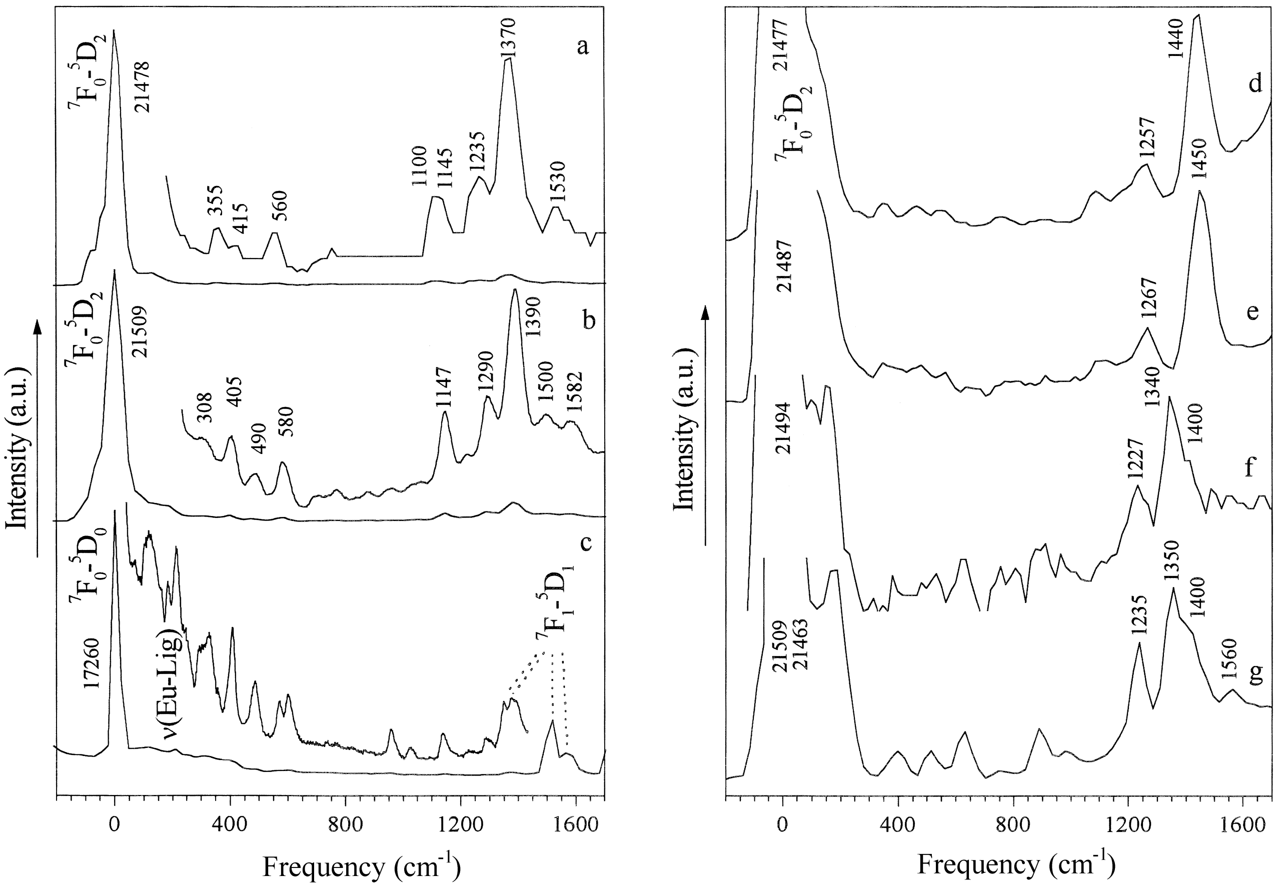

Fig. 4. Luminescence excitation spectra of $Eu(DPM)_3\cdot$Phen (a), $Eu(DPM)_3$ (b,c), $Eu(FOD)_3\cdot$Tmphen (d), $Eu(FOD)_3\cdot$Phen (e), $Eu(AA)_3\cdot$Tmphen (f) and $Eu(AA)_3\cdot 3H_2O$ (g) in the region of vibronic sidebands of the $^7F_0$–$^5D_2$ (a,b,d–g) and $^7F_0$–$^5D_0$ transitions (c) at 77 K.

intensities of the vibronic satellites in the region between the $^5D_0$–$^7F_3$ and $^5D_0$–$^7F_4$ transitions to their parent lines (the Stark components of the $^5D_0$–$^7F_2$ transition) is the same for both compounds. Therefore, the values of the electron–phonon interaction in these cases are the same.

Fig. 4 shows the vibronic sidebands of the $^7F_0$–$^5D_0$ and $^7F_0$–$^5D_2$ transitions in the excitation spectra of Eu(DPM)$_3$, as well as the vibronic sidebands of the $^7F_0$–$^5D_2$ transition in spectra of Eu(DPM)$_3$·Phen, Eu(FOD)$_3$·Tmphen, Eu(FOD)$_3$·Phen, Eu(AA)$_3$·Tmphen and Eu(AA)$_3$·3H$_2$O (FOD, heptafluorodimethyloctanedione; AA, acetylacetone). In the spectrum of Eu(DPM)$_3$·Phen most of the vibronic satellites are shifted by 20–30 cm^{-1} towards low frequencies in comparison with the spectrum of Eu(DPM)$_3$. The strongest band shifts from 1390 to 1370 cm^{-1}. An analogous picture is observed in the case of acetylacetone compounds in the transition from Eu(AA)$_3$·3H$_2$O to Eu(AA)$_3$·Phen and Eu(AA)$_3$·Tmphen, where this band shifts from 1350 to 1340 cm^{-1}. The decrease of the vibration frequency at transition from dipyvaloylmethanates to acetylacetonates and from tris-β-diketonates to their adducts with Ph demonstrates a decrease of the π-electronic density in the chelate ring. As mentioned earlier, the maximum shift of this strong band, to 1450 cm^{-1}, is observed in the transition to fluorinated β-diketonates. In detailed studies of the behaviour of vibration frequencies one should take into consideration not only the change in the π-electronic density in the chelate ring, but also the changes of the effective atomic mass taking part in the vibrational motion [27]. The change in electronic density in the chelate ring results in a change of the Eu–O bond strength. Therefore, the different values of the band shifts in europium acetylacetonates and dipivaloylmethanates are in accordance with the X-ray data. The Eu–O distance in Eu(AA)$_3$·Phen (2.397 Å) [14] is smaller than the corresponding distance in Eu(AA)$_3$·3H$_2$O (2.43 Å) [29] by 0.03 Å. In Eu(DPM)$_3$·Phen and Pr(DPM)$_3$, these distances are different by 0.08 Å (2.339 Å [12] and 2.445 Å [13]).

The intensity distributions of the vibronic sidebands of the $^7F_0$–$^5D_0$ and $^7F_0$–$^5D_2$ transitions in the spectra of Eu(DPM)$_3$ are different due to the influence of *J*-selection rules (Fig. 4c,b). Vibronic satellites related to the $^7F_0$–$^5D_2$ transition and corresponding to vibration with a frequency of 1390 cm^{-1} have the highest intensities, which could point to the fact that these satellites are due to symmetric vibrations, analogous to the case of the spectra of europium compounds containing flat trigonal anions [30], where the symmetric stretching vibration of the anion exhibits the highest intensity.

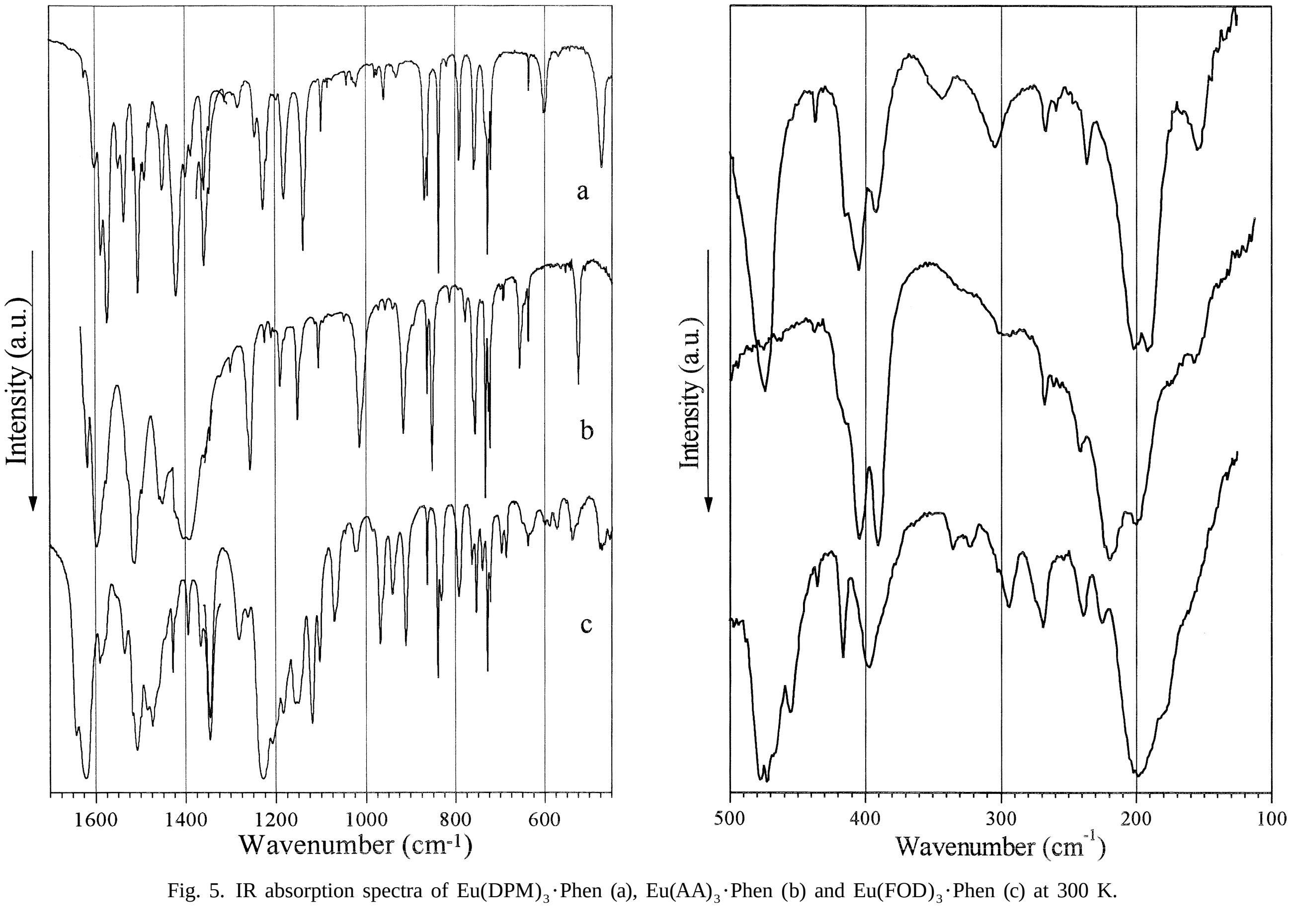

Fig. 5. IR absorption spectra of Eu(DPM)$_3$·Phen (a), Eu(AA)$_3$·Phen (b) and Eu(FOD)$_3$·Phen (c) at 300 K.

To a first-order approximation the intensity distribution of the vibronic sidebands of spectra of different europium β-diketonates is the same (Fig. 4). This allows us to study changes in the chelate ring vibration frequencies in the spectra of the investigated compounds. It is much more difficult to study these changes by IR spectra, due to the overlap of lines of the substituents (Fig. 5). Comparison of the vibronic and IR spectra helps to attribute the bands related to vibrations of the nearest surroundings of the metal ions. The shifts of the split intense IR band in the region 200 cm^{-1} correlate with the changes of mass of the β-diketones DPM, AA and FOD. The splitting of this band also correlates with X-ray data for the differences in the Eu–O and Eu–N distances. These correlations give the opportunity to attribute the IR bands in the region 190–230 cm^{-1} to vibrations of metal–ligand (Eu–O, Eu–N) bonds.

4. Conclusions

The influence of the donor–acceptor properties and of the size of the ligands on the spectroscopic characteristics of a series of $\mathrm{Eu(DPM)_3 \cdot Ph}$ compounds was investigated. The spectra of other europium β-diketonates were also studied. The dependencies of the Stark splitting of the Eu^{3+} energy levels, the efficiency of the excitation of Eu^{3+} through the ligand bands, vibration frequencies, and patterns of vibronic sidebands with variation of the phenanthroline substituent were examined. Crystal field parameters were calculated.

It was shown that steric factors are significant in determining the structure of the title compounds. From the spectra, $\mathrm{Eu(DPM)_3 \cdot Ph}$ compounds form two subgroups: with relatively small and bulky molecules of phenanthroline derivatives. The compounds with small ligands, phenanthroline and 5-methyl-phenanthroline, have the most symmetric surroundings of Eu^{3+}.

The same intensity distribution patterns of vibronic sidebands of the ${}^7F_0$–${}^5D_2$ transition in excitation spectra of different β-diketonates were demonstrated. A correlation of the vibration frequencies of the β-diketonate chelate ring with the donor–acceptor properties of radicals was observed. Vibronic spectra can be used for assignments of lines in IR and Raman spectra.

Values of the luminance of adducts were measured. $\mathrm{Eu(DPM)_3 \cdot Ph}$ compounds, containing phenyl derivatives of phenanthroline, exhibit the highest luminance.

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