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Temperature dependence of luminescence of terbium complexes with different substituents in N-heterocyclic ligands

© *K.D. Shmelkov, A.V. Kharcheva, N.E. Borisova, A.V. Ivanov, S.V. Patsaeva*

Lomonosov Moscow State University, Moscow, Russia

e-mail: vinchester3030@mail.ru

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The temperature dependence of the spectral luminescent characteristics of four terbium complexes with 2, 2′-bipyridyldicarboxanilides, containing different substituents in phenyl ring, has been studied. The measurements were carried out in the temperature range from 302 to 329 K. The dependences of the luminescence quantum yield and integral intensity on temperature were obtained for the studied compounds. Asymmetry ratios were calculated. It was found that the quantum yield and integral luminescence intensity of the complexes decrease with increasing temperature, and the coefficients of temperature sensitivity of these characteristics were calculated. The maximum relative temperature sensitivity of the integral luminescence intensity was observed for the complex with a ligand without a substituent, it was (9.0±1.0)%/K at a temperature of 309 K. Some of the studied complexes can potentially be used as optical thermometers

Keywords: luminescence, terbium, REE complexes, temperature sensitivity, organic ligands, optical thermometer.

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1. Introduction

The study of new luminescent compounds is an important task of modern optics and spectroscopy. Rare earth element (REE) coordination compounds are interesting objects of study due to their narrow spectral lines, significant Stokes shift, long luminescence lifetime and high luminescence intensity. Such compounds are used for addressing a number of tasks [1–6]. Therefore, the study of terbium complexes based on 2, 2′-bipyridyldicarboxylic acids is an urgent task.

The luminescent characteristics of such complexes (e.g., quantum yield, luminescence intensity, lifetime, or asymmetry RATIO) can be sensitive to temperature, making the complexes promising in practical applications, such as optical thermometers [7–11]. For example, europium complexes were studied in Ref. [9] in the temperature range of 293−373 K. The luminescence lifetime of europium was used as a temperature-dependent parameter. The lifetime decreased as the temperature increased. The mean sensitivity of this parameter was 5 μs/K, and the relative sensitivity was 0.9%/K.

Organic complexes of terbium and europium were created and used for development of thermosensitive polymer paints in Ref. [10]. The studies were carried out in the temperature range of 273−343 K. The luminescence lifetime of terbium and europium complexes in the polymer decreased with the increase of the temperature, and the maximum sensitivity was found for one of the terbium complexes and was 13.8 μs/K, which corresponds to a relative sensitivity of 0.67%/K.

Conjugates of ytterbium-neodymium and ytterbium-erbium complexes were studied in Ref. [11] in the temperature range of 295−395 K. The ratios of luminescence bands of different ions were used as a temperature-dependent parameter. The ratio of Yb bands (at wavelengths 980 and 1060 nm) was used for the {Yb−Nd} complex. The ratio of Yb and Er bands (at wavelengths 980 and 1460 nm) was used for the {Yb−Er} complex. This ratio decreased for the {Yb−Nd} complex with the increase of temperature, and it increased for the {Yb−Er} complex. The maximum relative sensitivity of the temperature parameter was 0.45%/K for the first complex and it was 3%/K for the second complex.

Like many REE ions, terbium ions have a small extinction coefficient when excited by visible or UV light [12]. The antenna effect is often used to increase the luminescence intensity [13,14]. A chelating ligand with a high extinction coefficient is attached to the REE ion, resulting in the formation of a complex with high absorption. The ligand not only plays the role of an antenna, but also shields the REE ion from the solvent, reducing the probability of radiation-free deactivation of the excited states of the complex [15]. The excitation radiation is absorbed by the organic part (ligand) in such complexes, putting the complex into the first singlet state IFx8xxE (less often into the second). Next, the complex can have the first triplet state T_1. The energy from the triplet state (more rarely from the singlet state) occupies one of the resonance levels of the REE ion, from which luminescence is further observed [16,17].

REE complexes with organic ligands can have high luminescence intensity with large quantum yield and have long lifetime and high stability, and some of them are soluble in organic solvents or in water (depending on the type of ligand) [18–22].

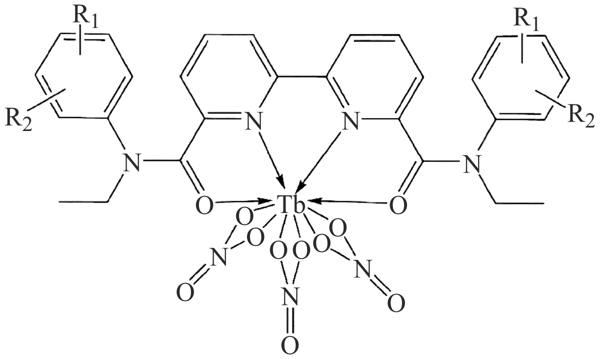

Figure 1. Structure of terbium complexes with a ligand based on 2, 2′-bipyridyl-6, 6′-dicarboxylic acid. Complex Bipy H Tb — $R_1 = R_2 = H$; Bipy 2F Tb — $R_1 = 2F$, $R_2 = H$; Bipy 3F Tb — $R_1 = 3F$, $R_2 = H$; Bipy 4F Tb — $R_1 = 4F$, $R_2 = H$.

Trivalent terbium ions have narrow spectral lines in the green wavelength range with the most intense luminescence in the spectral range 540–555 nm.

The purpose of this paper is to study the dependence of luminescence characteristics (luminescence quantum yield, asymmetry RATIO, integral luminescence intensity) of terbium complexes with different structure of alkyl substituent in ligands on temperature in the range from 302 to 329 K.

2. Objects and methods of study

The temperature dependence of the luminescence characteristics of solutions of four terbium complexes designated Bipy H Tb, Bipy 2F Tb, Bipy 3F Tb, and Bipy 4F Tb in acetonitrile is studied in this paper. These complexes can potentially be used as optical thermometers. The complexes with ligand based on 2, 2′-bipyridyl-6, 6′-dicarboxylic acid differed by substituents in the phenyl group, their structure is shown in Fig. 1.

A suspension of the complex was added to acetonitrile to prepare the solutions and the sample tube was placed for 5 min in a DADI968 ultrasonic bath. The solution was then transferred to a cuvette and the required amount of acetonitrile was added, bringing the concentration of the complex to $1 \cdot 10^{-5}$ mol/l.

Absorption, luminescence emission and excitation spectra were recorded during the study. Absorption spectra were measured using SOLAR PB2201 spectrophotometer (Belarus), and luminescence emission and excitation spectra — were measured using SOLAR CM2203 spectrofluorimeter (Belarus) with the excitation by 320 nm light. Measurements were performed in the temperature range from 302 to 329 K. A quartz cuvette with an optical path length of 10 mm was used. A 90-degree setup was used to obtain the luminescence excitation and emission spectra. The excitation radiation was not polarized. A thermostatically controlled cuvette compartment in both spectral instruments was used to maintain the required temperature of the test solution.

The acquired luminescence excitation and emission spectra were corrected for the effect of the internal filter [23] using the following formula:

$$I = I_0 \cdot 10^{\left(\frac{D_{ex}+D_{em}}{2}\right)},$$

where I_0 — the recorded luminescence intensity of the solution, D_{ex} and D_{em} — the optical densities of the solution at the luminescence excitation and emission wavelengths in a cuvette with an optical path length of 1 cm.

The luminescence quantum yield was determined using the reference dye method. As a reference standard, we used a solution of the europium complex (with a ligand based on 2, 2′-bipyridyl-6, 6′-dicarboxylic acid featuring a pyridyl substituent at the fourth position) at a concentration of $1 \cdot 10^{-5}$ mol/L in acetonitrile, for which the luminescence quantum yield had been previously measured (14%) [18]. Luminescence quantum yield of the studied complexes was calculated using the formula

$$\Phi = \Phi_{et}\frac{K}{K_{et}} = \Phi_{et}\frac{D_{et}}{D}\frac{I}{I_{et}},$$

where Φ and Φ_{et} — luminescence quantum yields of the investigated sample and reference, K and K_{et} — ratio I to D and I_{et} to D_{et} (I and I_{et} — integral luminescence intensities of solutions of the test and reference samples, D and D_{et} — optical densities of the test and reference samples at the excitation wavelength).

The asymmetry RATIO R can be calculated using the first two bands of the luminescence emission spectrum of the terbium complex. The following formula is used [24]:

$$R = \frac{I(^5D_4 \rightarrow ^7F_5)}{I(^5D_4 \rightarrow ^7F_6)},$$

where $I(^5D_4 \rightarrow {}^7F_5)$ and $I(^5D_4 \rightarrow {}^7F_6)$ are the integral peak intensities corresponding to transitions to levels $^7F_5$ and $^7F_6$.

3. Experimental results

3.1. Absorption spectra

The absorption spectra of all complexes have a band in the range 310–330 nm, which corresponds to the absorption of light by the ligand [25]. It has been found that as the temperature of the solution increases, the optical density decreases, but this change does not exceed 13% of the initial value in the entire temperature range. The shape of the absorption spectrum was almost identical for different complexes, and the optical density at a wavelength of 320 nm differed by no more than 12% from the maximum recorded value (Fig. 2).

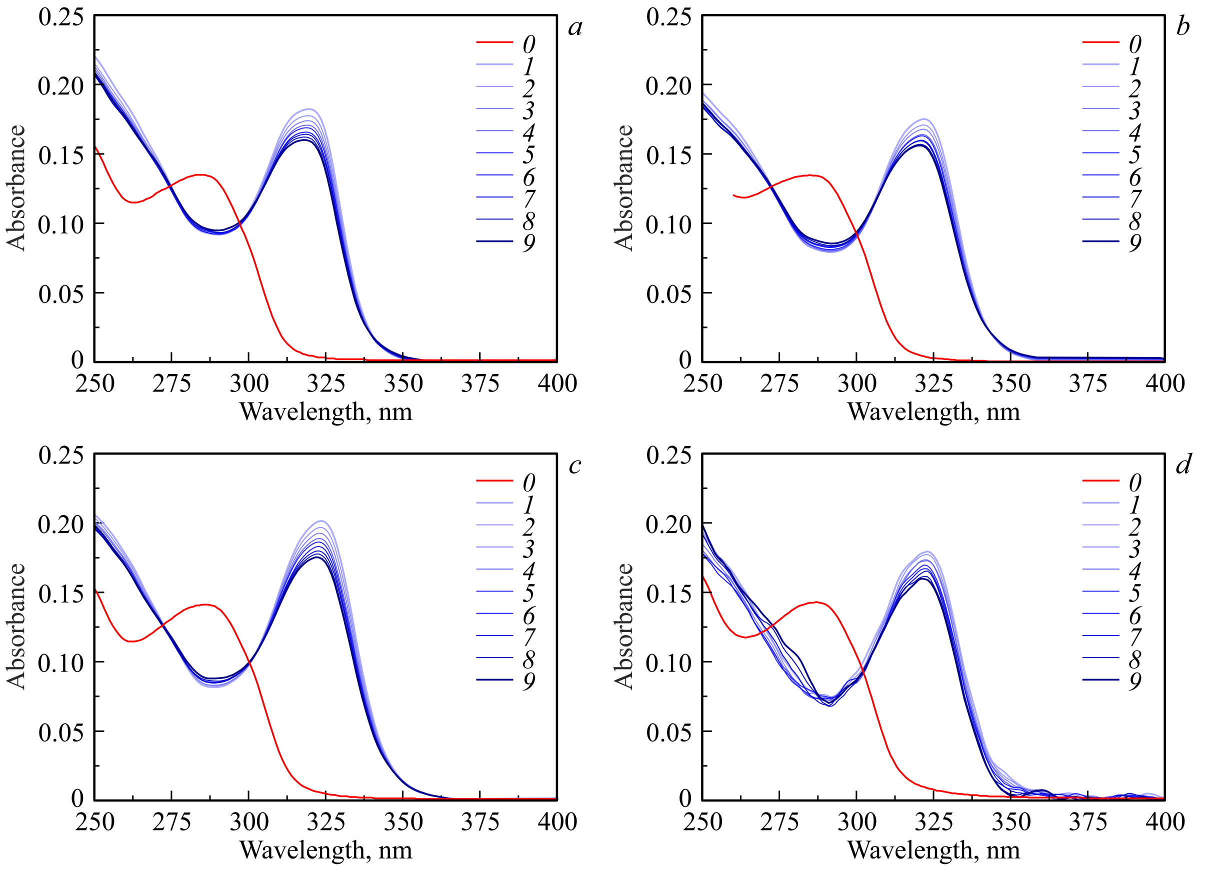

Figure 2. Absorption spectra of solutions of complexes: *a* — Bipy 2F Tb, *b* — Bipy 3F Tb, *c* — Bipy 4F Tb, *d* — Bipy H Tb in acetonitrile at a temperature *1* — 302 K; *2* — 306 K; *3* — 310 K; *4* — 313 K; *5* — 315 K; *6* — 318 K; *7* — 322 K; *8* — 325 K; *9* — 329 K. Curve *0* — ligand absorption.

3.2. Luminescence emission spectra

The luminescence emission spectra of all four studied terbium complexes exhibit similar shapes and contain characteristic peaks corresponding to transitions from the $^5D_4$ level to lower-lying energy levels ^7F_J (where $J = 6–3$), with maxima at wavelengths in the 485–500, 540–555, 580–595, and 615–625 nm regions, respectively [26,27]. However, the two peaks with wavelengths of 580–595 and 615–625 nm become indistinguishable against the background noise with the increase of the temperature in all complexes except the Bipy 2F Tb complex. The most intense lines correspond to transitions from the level $^5D_4$ to levels $^7F_5$ and $^7F_6$, which corresponds to luminescence emission with wavelengths 485–500 and 540–555 nm.

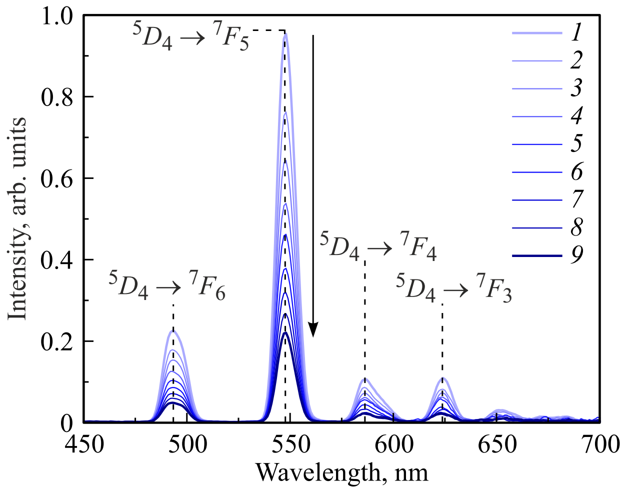

Figure 3. Luminescence emission spectra of a solution of the Bipy 2F Tb complex at temperature: *1* — 302 K; *2* — 306 K; *3* — 310 K; *4* — 313 K; *5* — 315 K; *6* — 318 K; *7* — 322 K; *8* — 325 K; *9* — 329 K.

The luminescence intensity of all complexes significantly decreased with the increase of the temperature (Fig. 3). The integral intensity of the line corresponding to the transition from the $^5D_4$ level to the $^7F_5$ level decreased by 79% for the Bipy H Tb complex, by 77%, 71%, and 65% for the Bipy 2F Tb, Bipy 3F Tb, and Bipy 4F Tb complexes. The Bipy 2F Tb complex showed high luminescence intensity against the background of other complexes. When the solution was cooled to the initial temperature, the luminescence intensity was 79% of the initial level for the Bipy H Tb complex, 98%, 89%, and 92% — for the Bipy 2F Tb, Bipy 3F Tb,

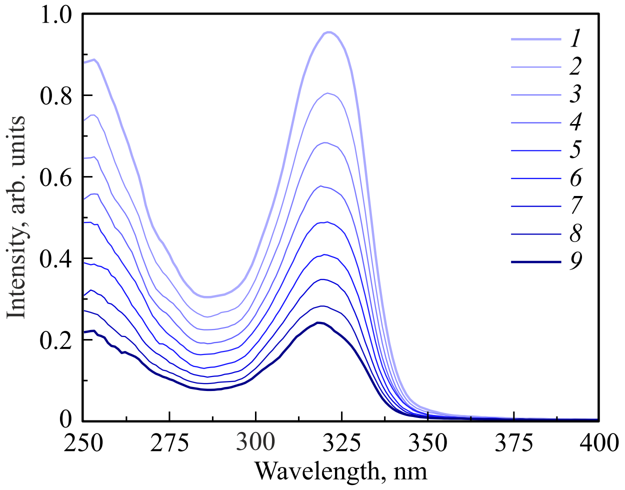

Figure 4. Luminescence excitation spectra of a solution of Bipy 2F Tb complex at temperature: *1* — 302 K; *2* — 306 K; *3* — 310 K; *4* — 313 K; *5* — 315 K; *6* — 318 K; *7* — 322 K; *8* — 325 K; *9* — 329 K.

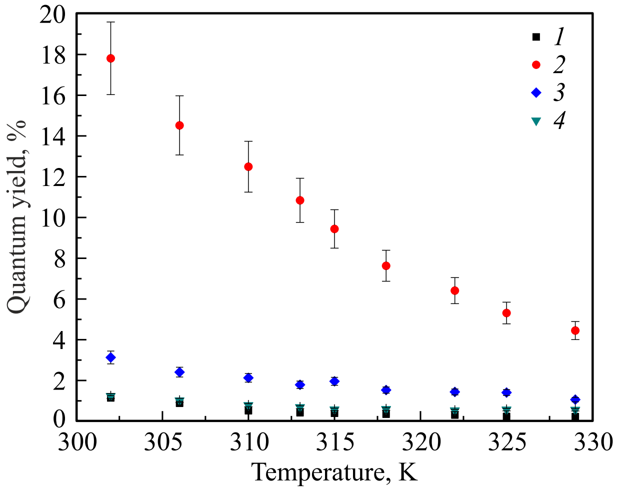

Figure 5. Dependence of luminescence quantum yield Φ of solutions of the complexes: *1* — Bipy H Tb, *2* — Bipy 2F Tb, *3* — Bipy 3F Tb and *4* — Bipy 4F Tb from temperature over the temperature range of 302–329 K.

Table 1. Relative temperature sensitivities of the quantum yield of the complexes in two temperature ranges

Complex	Temperature, K	Relative Temperature sensitivity of quantum yield, %/K
Bipy H Tb	302–315	9.0 ± 1.0
	315–329	4.0 ± 0.6
Bipy 2F Tb	302–315	4.3 ± 0.5
	315–329	5.0 ± 0.6
Bipy 3F Tb	302–315	4.7 ± 0.7
	315–329	3.6 ± 0.8
Bipy 4F Tb	302–315	5.3 ± 0.5
	315–329	0.4 ± 0.4

and Bipy 4F Tb complexes, respectively. The decrease of the luminescence intensity with the increase of the temperature is explained by the increase of the probability of back energy transfer from the resonance level $^5D_4$ of the terbium ion to the first triplet level of the ligand increases with the increase of the temperature [28].

3.3. Luminescence excitation spectra

Luminescence excitation spectra of terbium ion were recorded at a wavelength of 545 nm. They are shown in Fig. 4 for one of the complexes at different temperatures. A broad band in the excitation spectrum with a maximum at a wavelength of 320 nm is observed in all complexes. The luminescence intensity decreases with the increase of the temperature. The luminescence excitation spectra are similar in shape to each other and to the absorption spectra of the same solutions. When the temperature was reduced to the initial temperature, the luminescence intensities in the excitation spectra increased similarly to the changes of the luminescence emission spectra.

3.4. Luminescence quantum yield

The temperature dependences of the luminescence quantum yield Φ of the solutions are shown in Fig. 5. The highest quantum yield was found for the Bipy 2F Tb complex, which amounted to 17.8%. We relate this to the fact that the F-substituent at position 2 of the phenyl ring is located in close proximity to the terbium ion; presumably, it prevents acetonitrile molecules (or other possible quenchers, such as CH groups of the ligand) from penetrating into the first or second coordination spheres of terbium. The substituent in the remaining three complexes is either located further away or is not present at all (as in the case of the complex with unsubstituted ligand). It can be seen that a decrease of Φ for all Tb complexes was observed with the temperature increase. This is explained by the increase of the probability of back energy transfer from the resonance level of terbium to the ligand triplet level, and hence by the increase of the probability of radiation-free deactivation of the resonance level. Re As the temperature increased, the luminescence quantum yield of the Bipy H Tb complex decreased by 80%, that of the Bipy 2F Tb — by 75%, that of the Bipy 3F Tb — by 66%, and that of the Bipy 4F Tb — by 55%.

The drop of Φ with the increase of the temperature in the first approximation can be described by two linear sections — from 302 to 315 K and from 315 to 329 K. The relative temperature sensitivity (as the ratio of the change in

Table 2. Temperature sensitivities of the integral intensity of complexes in two temperature ranges

Complex	Temperature, K	Relative temperature sensitivity of the integral intensity, %/K
Bipy H Tb	302–315	9.0 ± 1.0
	315–329	3.6 ± 0.5
Bipy 2F Tb	302–315	4.9 ± 0.5
	315–329	5.2 ± 0.6
Bipy 3F Tb	302–315	5.5 ± 1.0
	315–329	4.9 ± 0.7
Bipy 4F Tb	302–315	5.8 ± 0.6
	315–329	0.8 ± 0.4

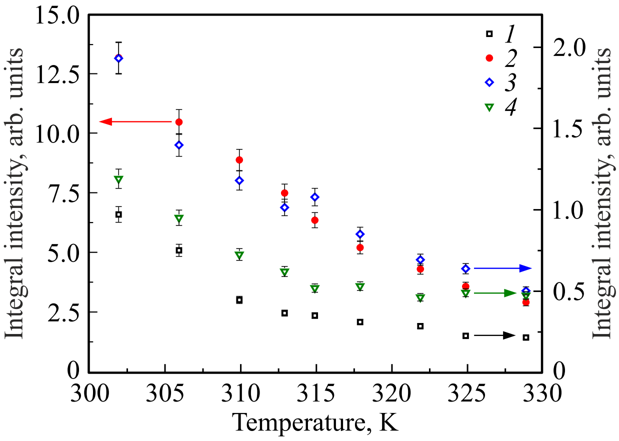

Figure 6. The dependence of the integral luminescence intensity of solutions of the complexes: *1* — Bipy H Tb, *2* — Bipy 2F Tb, *3* — Bipy 3F Tb and *4* — Bipy 4F Tb as a function of temperature in the temperature range 302–329 K. (The ordinate scale on the right is shown for the dependencies *1, 3* and *4*, the ordinate scale for the dependency *2* is shown on the left).

quantum yield to the change in temperature divided by the value of the quantum yield of the middle of the range) was calculated for each of these temperature ranges; Table 1 summarizes its values. It can be seen that the value of relative sensitivity is higher at lower temperatures for all complexes, except for the Bipy 2F Tb complex. The highest relative sensitivity of this parameter, equal to 9%/K, was observed for the complex without a substituent (Bipy H Tb).

3.5. Integral Intensity

The integral intensity was calculated from the sum of the integrals of the bands corresponding to the transitions from level $^5D_4$ to levels $^7F_6$ and $^7F_5$ in the luminescence emission spectrum. Its dependence on temperature is shown in Fig. 6.

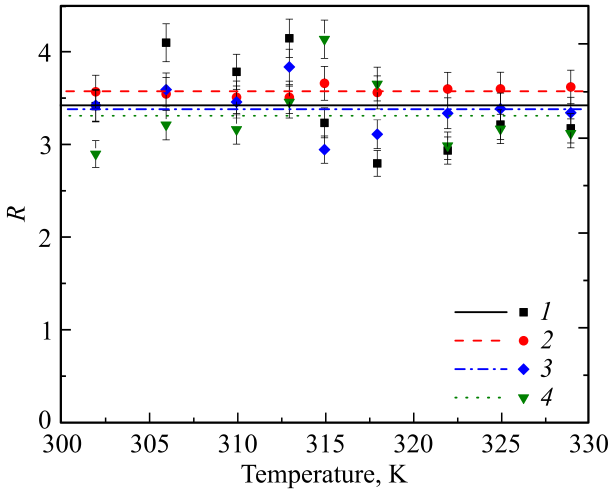

Figure 7. Dependence of the asymmetry ratio R of the complexes: *1* — Bipy H Tb, *2* — Bipy 2F Tb, *3* — Bipy 3F Tb and *4* — Bipy 4F Tb as a function of temperature in the temperature range 302–329 K. The solid line indicates the mean value of the asymmetry RATIO of the Bipy H Tb complex, the dashed — Bipy 2F Tb, the dashed — Bipy 3F Tb, and the dotted — Bipy 4F Tb.

It can be seen that the value of integral intensity of all complexes decreased with the increase of the temperature. The greatest change was found in the Bipy H Tb and Bipy 2F Tb — it amounted to 78%, it reached 74% in the Bipy 3F Tb and 60% in the Bipy 4F Tb.

Table 2 shows the values of relative sensitivity of integral intensity for two temperature zones: from 302 to 315 K and from 315 to 329 K. The highest relative sensitivity of this parameter, equal to 9%K, was observed for the complex without a substituent (Bipy H Tb).

Let us compare the obtained temperature sensitivity coefficients with those available in the literature. The ratio of the luminescence intensity of europium to terbium in hydroxybenzoate complexes was used in [29] as a temperature-dependent parameter. The relative temperature sensitivity of this ratio reached a maximum value of 4%/K at a temperature of 373 K. The luminescence intensity ratio of terbium to europium in tetrakis(benzoyltrifluoroacetone) complexes in the temperature range 20–225 K was used in Ref. [30], with a relative sensitivity as high as 7.6%/K at 20 K. The same parameter was used in metal-organic frameworks (MOFs) in Ref. [31]. The relative temperature sensitivity reached 7.32%/K for one of the samples in the temperature range 293–333 K. The mixed complexes of terbium and europium with ligands based on triphenylphosphinoxide and furan-containing analog of tenoyltrifluoroacetone were studied in a wide temperature range in Ref. [32]. The ratio of the integral luminescence intensity of terbium to europium was used as a temperature-dependent parameter. The relative sensitivity of this parameter was 3%/K in the range from 180 to 320 K.

As we can see, the relative temperature sensitivity of terbium luminescence for all the terbium complexes we have studied based on 2, 2′-bipyridyldicarboxanilide (and especially for the unsubstituted Bipy H Tb complex) reaches rather high values in comparison with those known from scientific publications.

3.6. Asymmetry ratio

The asymmetry ratio R was calculated (Fig. 7). This coefficient reflects the different effects of the ligand field (including the symmetry of the ligand field and the charge density near the REE) [33–35]. No unambiguous dependence of the asymmetry ratio on temperature was found. R is 3.4 in average for all complexes and over the entire temperature range.

4. Conclusion

When studying the temperature dependence of the luminescence characteristics of four terbium complexes in acetonitrile, a significant (approximately by 55−80%) decrease of the luminescence quantum yield and integral intensity with the increase of temperature from 302 to 329 K was found. We attribute this to an increase of the probability of radiation-free deactivation of the resonance state of the terbium ion by back energy transfer from the resonance level to the triplet level of the ligand. The highest absolute luminescence quantum yield was observed for the Bipy 2F Tb complex: it amounted to 17.8% at room temperature. The quantum yield was no more than 4% for the other three complexes. The maximum relative change of the luminescence quantum yield with temperature was found for the Bipy H Tb complex: it amounted to 80%; the same complex exhibited the highest relative sensitivity of this parameter in the temperature range from 302 to 315 K: it reached rather high values (9.0 ± 1.0)%/K). The largest decrease of the integral luminescence intensity was found for Bipy H Tb and Bipy 2F Tb complexes: it amounted to 78% with the temperature increase from 302 to 329 K. The maximum temperature sensitivity of this parameter was observed for the Bipy H Tb complex in the temperature range from 302 to 315 K and amounted to (9.0 ± 1.0)%/K. The asymmetry ratio R for all the studied complexes in acetonitrile was calculated using the intensities of the two main lines in the emission spectrum of terbium; its average value is equal to 3.4. No unambiguous dependence of the value of the asymmetry ratio on temperature was found.

The Bipy 2F Tb complex has the greatest potential for the development of an optical thermometer. Its high luminescence intensity, temperature-dependent luminescence parameters, and recovery (with 2% accuracy) of the luminescence intensity upon cooling to the initial temperature make it an interesting object for further studies.

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Conflict of interest

The authors declare that they have no conflict of interest.

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