Inorganic Chemistry Communications 113 (2020) 107814

Contents lists available at ScienceDirect

Inorganic Chemistry Communications

journal homepage: www.elsevier.com/locate/inoche

Color-tunable white-light of binary *tris-β*-diketonate-(Dy^{3+}, $Gd^{3+}{}_x$) complexes' blend under single wavelength excitation

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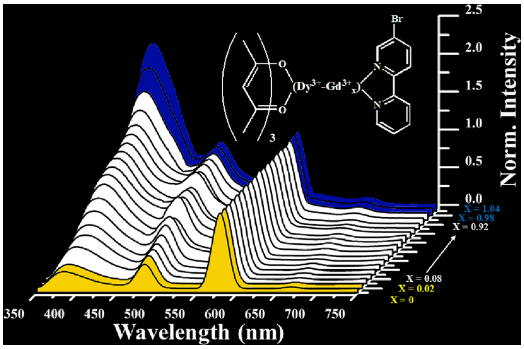
GRAPHICAL ABSTRACT

Based on the Dy^{3+}-centered yellow-light and the ligands-based blue-light of the *tris-β*-diketonate-(Dy^{3+}, $Gd^{3+}{}_x$)-mixed complex [Ln(acac)$_3$(5-Br-2,2′-bpy)] (Ln^{3+} = Dy^{3+}, $Gd^{3+}{}_x$), the stoichiometrically dichromatic integration gives the color-tunable white-light in solution or solid-state.



ARTICLE INFO

Keywords:
Tris-β-diketonate-(Dy^{3+}, $Gd^{3+}{}_x$) complex
Dichromatic white-light
Absorption
Fluorescence titrations

ABSTRACT

Based on the Dy^{3+}-centered yellow-light and the ligands-based blue-light of the iso-structural two complexes [Ln(acac)$_3$(5-Br-2,2′-bpy)] (Ln^{3+} = Dy^{3+} (**2**) or Gd^{3+} (**3**); **Hacac** = acetylacetone, **5-Br-2,2′-bpy** = 5-bromo-2,2′-bipyridine), respectively, the stoichiometric fluorescence titrations of their *tris-β*-diketonate-(Dy^{3+}, $Gd^{3+}{}_x$)-mixed complex, show that it is capable of the smooth color-tuning (yellow- to white- and to blue-light) under single wavelength excitation. Moreover, through the dichromatic integration, the binary *tris-β*-diketonate-(Dy^{3+}, $Gd^{3+}{}_x$) complex exhibits the straightforward white-light in solid-state.

1. Introduction

Contributing from the receptive "antenna" effect [1], the Dy^{3+}-centered characteristic emissions ($^4F_{5/2} \rightarrow {}^6H_{J/2}$; J = 9, 11, 13 or 15) [2] can be sensitized through the Laporte- and spin-allowed ligand-centered transition ($^0S \rightarrow {}^1S$) followed by the inter-system crossing (ISC; $^1S \rightarrow {}^1T$) and the $^1T \rightarrow Dy^{3+*}$ transfer for one specific organo-Dy^{3+} chromophore. Especially in consideration of its two prominent yellow-light (578 nm; $^4F_{5/2} \rightarrow {}^6H_{13/2}$) and blue-light (484 nm; $^4F_{5/2} \rightarrow {}^6H_{15/2}$) line-like emission bands within the visible region characteristic of ideal color-primary components toward straightforward white-light, concerted efforts have been devoted to white-light-emissive organo-

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https://doi.org/10.1016/j.inoche.2020.107814
Received 14 January 2020; Received in revised form 23 January 2020; Accepted 24 January 2020

Dy^{3+} materials promising for optoelectronic devices [3] and sensing platforms [4]. In principle, through the effective sensitization and the subsequently complete energy transfer for one specific organo-Dy^{3+} complex, one feasible strategy to direct white-light seems highly attractive after the simultaneous emissions of both Dy^{3+}-centered blue-light (λ_{em} = 478 nm; $^4F_9 \rightarrow {}^6H_{15/2}$ magnetic dipole transition) and yellow-light (λ_{em} = 572 nm; $^4F_9 \rightarrow {}^6H_{13/2}$ electric dipole transition) with a comparable intensity [5]. Nonetheless, due to the ligands-field adjustment [6] just *hyper*-sensitive to the electric-dipole-governed transition while insensitive to the magnetic dipole transition, the dominated yellow-light as a universal basis, renders the Dy^{3+}-exclusive white-light greatly challenging [7] to one certain organo-Dy^{3+} complex. Meanwhile, the dichromatic-integration just with the two Dy^{3+}-centered sharp visible emissions, does not cover the broad 400–700 nm spectral region, also suffering from the inferior color-rendering property [8]. By contrast, through the ligands-based residual blue-light color-compensated with the Dy^{3+}-dominated yellow-light, single-molecule dichromatic white-light [9] could be smoothly approached for the specific organo-Dy^{3+} complex. However, this alternative strategy still suffers from an intrinsic obstacle of having unsatisfactory white-light efficiency ($\Phi < 5\%$) caused by the partial energy transfer.

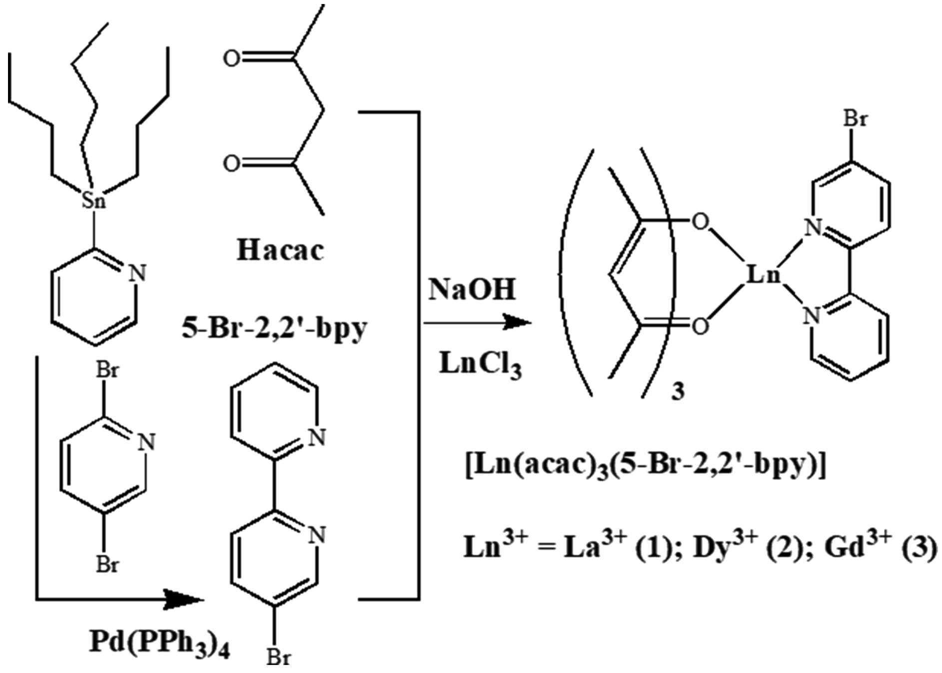
Convincingly, the circumvention of such problem can rely on (Dy, Gd)-[10], (Dy, Eu)-[11], (Dy, Eu, Gd)-[12], (Dy, Sm, Gd)-[13], (Dy, La, Sm)-[14], (Dy, Eu, Tb)-blended [15] metal-organic frameworks (MOFs) or coordination polymers, where in dependence on a precise control of the Ln^{3+}-mixed stoichiometry, different color-compensatory contributions could be well balanced for efficient and high-quality white-lights. However, despite their potential applications [3a] for white light-emitting diodes (WLEDs), the detrimental deficiency of their processing (vacuum-deposition or solution-process) inability, limits the utilization in flexible white organic/polymer light-emitting diodes (WOLEDs/WPLEDs) [3b–3d]. As a matter of fact, from the perspective of film-forming capability necessary, the Ln^{3+}-mixed complexes' blending toward direct white-light, does conceptually take effect on WOLEDs/WPLEDs. Herein, in light of the color-tunable white-light of (Eu, Tb)-mixed complexes [16] disadvantageous of unmanageable Tb^{3+}-to-Eu^{3+} energy transfer, the (Dy, Gd) complexes' blending should be more worthy of motivation, because the patent absence of Dy^{3+}-to-Gd^{3+} energy transfer is unambiguous, due to the extremely higher energy level of Gd^{3+} ion [17]. Moreover, based on the yellow-plus-blue integration, one certain (Dy^{3+}, $Gd^{3+}{}_x$) complexes' blending is also capable of direct white-light. Interestingly, with the judicious adjustment of the (Dy^{3+}, $Gd^{3+}{}_x$)-mixed ratio, the dichromatic white-light endowed from the two isomeric Ln^{3+}-complexes, can be easily realized under single wavelength excitation.

As shown in Scheme 1, the N,N'-chelate ancillary ligand **5-Br-2,2′-bpy** was synthesized in the yield of 67% from the well-established Stille coupling reaction between 2,5-dibromopyridine and 2-(tributylstannyl) pyridine in the presence of $Pd(PPh_3)_4$ (0) as the literature [18]. Further through the one-pot reaction of $LnCl_3{\cdot}6H_2O$ (Ln = La, Dy, or Gd), the N,N'-chelate ancillary ligand **5-Br-2,2′-bpy**and the *β*-diketone ligand **Hacac** treated with an equimolar amount of anhydrous NaOH, the series of binary *tris*-*β*-diketonate-Ln^{3+} complexes [$Ln(acac)_3$(5-Br-2,2′-bpy)] (Ln = La (**1**); Dy (**2**); or Gd (**3**)) were self-assembled in receptive yields of 68–75%, respectively.

All of the three complexes **1**–**3**, soluble in common organic solvents, were well characterized by EA, FT-IR, ^{1}H NMR and ESI-MS. Especially in the ^{1}H NMR spectrum of the *anti*-ferromagnetic [$La(acac)_3$(5-Br-2,2′-bpy)] (**1**), the combined proton resonances (δ = 8.82–1.73 ppm) of both the deprotonated ligand $(\mathbf{acac})^-$ and the ancillary ligand **5-Br-2,2′-bpy** are observed with a stipulated molar ratio of 3:1. Meanwhile, the presence of the typically enolic $-CH{=}C$ proton singlet peak at δ = 5.19 ppm of the $(\mathbf{acac})^-$ ligand should be resulted from the La^{3+}-coordination. Moreover, based on the ESI-MS results of the complexes **1**–**3**, a strong mass peak at *m/z* 672.02 for [$La(acac)_3$(5-Br-2,2′-bpy)] (**1**), 695.91 for [$Dy(acac)_3$(5-Br-2,2′-bpy)] (**2**)or 691.04 for [Gd$(acac)_3$(5-Br-2,2′-bpy)] (**3**), can be definitely assigned to the major species $[M-H]^+$, respectively. These observations confirm that each binary *tris*-*β*-diketonate-Ln^{3+} species of the three iso-structural complexes **1**–**3**, can keep stable in the respective solution.

The photo-physical properties of the binary *tris*-*β*-diketonate-Ln^{3+} complex **2**–**3** were examined in dilute MeCN solutions at room temperature or 77 K, and summarized in Figs. 1 and 1**S.** In contrast to the strong absorption bands (Fig. 1S) limited to the $\lambda_{ab} < 350$ nm range for the two (**Hacac** and **5-Br-2,2′-bpy**) free ligands, the complex **2**–**3** display the similar ligands-based (248–250 and 284–287 nm) while significantly broadened (200–400 nm) absorption spectra, in which, the lower strong one (284–287 nm) should be assigned to the ligands-based π-π*-transitions. Moreover, upon Ln^{3+}coordination, the molar absorption coefficients of the complexes **2**–**3** at the lower energy absorption peaks are almost three orders of magnitudes larger than those of the two (**Hacac** and **5-Br-2,2′-bpy**) free ligands, also indicative of their *tris*-*β*-diketonate-Ln^{3+} component with the enhanced π-conjugation effect. For the Dy^{3+}-based complex **2**, upon photo-excitation of the chromohphores at the range of 200–410 nm (λ_{ex} = 340 nm), as shown in Fig. 1, the strong Dy^{3+}-characteristic line-like emissions (484 nm ($^4F_{9/2} \rightarrow {}^6H_{15/2}$ transition), 578 nm ($^4F_{5/2} \rightarrow {}^6H_{13/2}$ transition), 629 nm ($^4F_{5/2} \rightarrow {}^6H_{11/2}$ transition) and 665 nm ($^4F_{5/2} \rightarrow {}^6H_{9/2}$ transition)) and the weak while detectable emission peaking at λ_{em} = 388 nm, are concurrently observed. For the dual-emissive complex **2** featuring a bright yellow-light with the CIE (Commission International De L'Eclairage) chromatic coordinate × = 0.400 and *y* = 0.401, the ligands-based residual (λ_{em} = 388 nm) emission should be assigned to the intra-ligands π-π* transition, and the *hyper*-sensitive peak at 578 nm from the $^4F_{5/2} \rightarrow {}^6H_{13/2}$ transition should be resulted from its low molecular symmetry [19]. Moreover, its dual-emitting nature, can further be confirmed with the lifetimes-decayed combination of the ligands-based fluorescence (τ = 1.13 ns; λ_{em} = 388 nm) and the Dy^{3+}-centered phosphorescence (τ = 3.2 μs at λ_{em} = 578 nm; $^4F_{5/2} \rightarrow {}^6H_{13/2}$ transition) from the same chromophores. As to the Gd^{3+}-based complex **3** in solution at room temperature, it displays the typically ligands-based fluorescence (λ_{em} = 394 nm, τ = 1.02 ns and Φ_{em} = 4.7%) also shown in Fig. 1, exhibiting a blue-light with the CIE chromatic coordinate × = 0.203 and *y* = 0.202. In contrast, the Gd^{3+}-centered complex **3** in solution at 77 K, shows the 0–0 transition phosphorescence (λ_{em} = 433 nm andτ = 0.98 μs; also Fig. 1), from which, the triplet (^{3}π-π*) energy level of 23095 cm^{-1} is obtained. With regard to the singlet (^{1}π-π*) energy level, it (27855 cm^{-1}) can be reasonably estimated from the lower wavelength (359 nm) of its UV–visible absorbance edge, and thus, the first energy gap ΔE^1 (^{1}π-π* - ^{3}π-π*, 4760 cm^{-1}; Fig. 2S) near to the desirable 5000 cm^{-1}, endows a relatively effective ISC process according to the Reinhoudt's empirical rule [20]. Importantly, further checking the energy level match between the ligands-based ^{3}π-π* energy and the lowest excited state $^4F_{5/2}$ (20830 cm^{-1}) of Dy^{3+} ion, the second energy gap ΔE^2 (2265 cm^{-1}; ^{3}π-π*-$^4F_{5/2}$) is beyond the ideal 2500-–4500 range ruled by the Latva's empirical rule [21], and thus, the allowed back energy transfer should be the reason to the dual emissions of the Dy^{3+}-based complex **2**. Noticeably, the absolute quantum efficiency (Φ_{em}) of the complex **2**, characteristic of the Dy^{3+}-endowed yellow-light, is up to 5.8%, which is at the top level among previously reported organo-Dy^{3+}-complexes [3b–3d,8–9], which should be mainly due to the strengthened optical absorbance and the effective suppression from the oscillator-induced quenching [22] or the non-radiative deactivation by the **5-Br-2,2′-bpy**-ancillary involvement.

In consideration of the relatively higher efficiencies for the yellow-light of the Dy^{3+}-based complex **2** and the blue-emitting of the isomeric Gd^{3+}-based complex **3**, it is particular interest on their binary *tris*-*β*-diketonate-(Dy^{3+}, $Gd^{3+}{}_x$) complexes' blend toward the dichromatic white-light modulation as desirable. After adding different amounts of the Gd^{3+}-based complex **3** to the Dy^{3+}-based complex **2**, the UV–visible absorption and fluorescence titrations in MeCN solution



Scheme 1. Synthetic scheme of the ancillary ligand **5-Br-2,2′-bpy** and its binary *tris-β*-diketonate-Ln^{3+} complexes **1–3** (Ln^{3+} = La^{3+} (**1**); Ln^{3+} = Dy^{3+} (**2**) or Ln^{3+} = Gd^{3+} (**3**)).

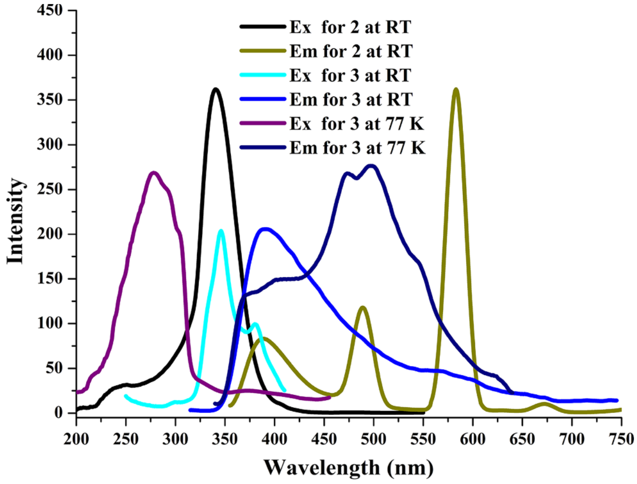


Fig. 1. The excitation and visible emission spectra of the complexes [Dy(acac)$_3$(5-Br-2,2′-bpy)] (**2**) and [Gd(acac)$_3$(5-Br-2,2′-bpy)] (**3**) in dilute MeCN solution (1 × 10^{-5} M) at room temperature or 77 K.

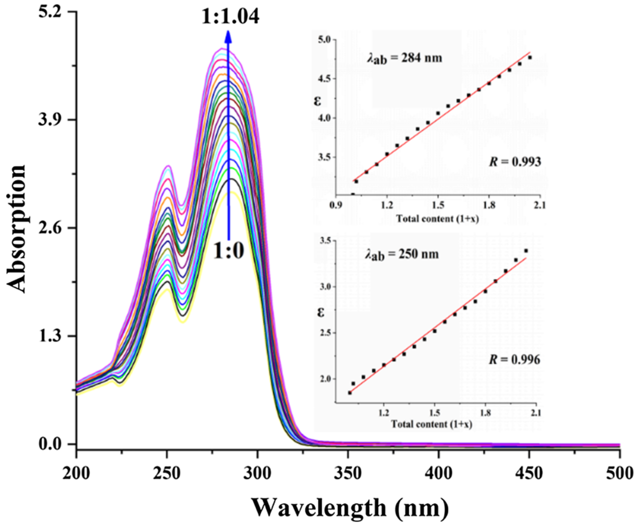


Fig. 2. The UV–visible absorption titration at room temperature of the binary *tris-β*-diketonate-Dy^{3+} complex **2** in MeCN solution with its isomeric binary *tris-β*-diketonate-Gd^{3+} complex **3** in MeCN solution.

were examined at room temperature, respectively. As shown in Fig. 2, the UV–visible absorption titration of the binary *tris-β*-diketonate-(Dy^{3+}, Gd$^{3+}{}_x$) complexes' blending system, displays that both of the two absorbance at 250 nm and 284 nm of the Dy^{3+}-based complex **2** in solution monotonously increase with the increasing ((Dy^{3+}, Gd$^{3+}_{0.02}$) to (Dy^{3+}, Gd$^{3+}_{1.04}$)) of the concentration of the Gd^{3+}-based complex **3**. Whereas besides the absence of an isosbestic point, each absorbance is almost proportional to the (Dy^{3+}, Gd^{3+})-mixed total (1 + x) content, which strictly adhered to the Lambert-Beer law [23] with the R of 0.993 or 0.996, should be arisen from the physical coexistence of two isomeric complexes **2** and **3** without intermolecular interactions in dilute solutions. As to the fluorescence titration (λ_{ex} = 340 nm) shown Table 1S and in Fig. 3, during the amount increasing ((Dy^{3+}, Gd$^{3+}_{0.02}$) to (Dy^{3+}, Gd$^{3+}_{1.04}$)) of the Gd^{3+}-based complex **3**, its emission and the ligands' residual emission of the Dy^{3+}-based complex **2** are combined in the 350–450 nm range, which, together with the Dy^{3+}-centered characteristic emissions ($^4F_{5/2}\rightarrow{}^6H_{J/2}$; J = 9, 11, 13 or 15) are simultaneously observed and cover the broad-ranging 350–750 nm. Moreover, for the binary *tris-β*-diketonate-(Dy^{3+}, Gd$^{3+}_x$) complexes, the gradual waxing of the combined (350–450 nm) emission intensity, trades off the gradual waning of the two (572 and 478 nm) Dy^{3+}-characteristic dominated and ratio-fixed emission intensities. Interestingly, after the introduction of the Gd^{3+}-based complex **3**, their integrated colors change from yellow-light (CIE chromatic coordinates × = 0.385, y = 0.386; (Dy^{3+}, Gd$^{3+}_{0.02}$)) to white-lights (CIE chromatic coordinates × = 0.258–0.337, y = 0.250–0.324, CCTs of 3628–4153 K and CRIs of 90–92; (Dy^{3+}, Gd$^{3+}_{0.08}$) to (Dy^{3+}, Gd$^{3+}_{0.92}$)) and to blue-lights (CIE chromatic coordinates × = 0.230–0.250, y = 0.207–0.229; (Dy^{3+}, Gd$^{3+}_{0.98}$) to (Dy^{3+}, Gd$^{3+}_{1.04}$)). And during the linear shifting of the color-point beginning from the yellow-light of the Dy^{3+}-based complex **2** and ending at the blue-light of the Gd^{3+}-based

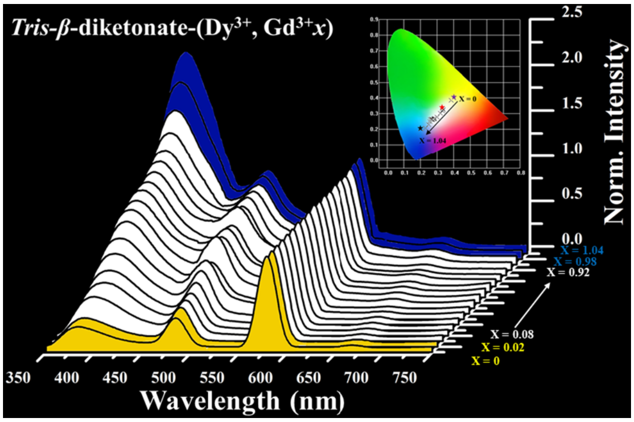


Fig. 3. The fluorescence titration upon λ_{ex} = 340 nm at room temperature of the binary *tris-β*-diketonate-Dy^{3+} complex **2** in MeCN solution with its isomeric binary *tris-β*-diketonate-Gd^{3+} complex **3** in MeCN solution.

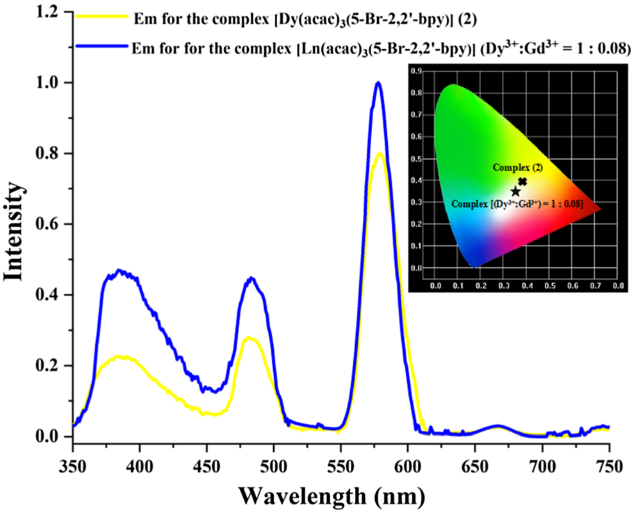


Fig. 4. The emission spectra upon λ_{ex} = 340 nm with CIE chromatic coordinates (inset) of the binary *tris-β*-diketonate-Dy^{3+} complex **2** and the binary *tris-β*-diketonate-(Dy^{3+}, Gd^{3+}_x) complex in solid-state at room temperature.

complex **3**, the best white-light point at the (Dy^{3+}, $Gd^{3+}_{0.08}$)-mixed content, enables the color-coordinate of × = 0.337, y = 0.324, the CCT of 4153 K and the CRI of 90. Noticeably, through the physical blending with an additional $^1\pi$-π^* to $^3\pi$-π^* and to Dy^{3+} radiative transition (also Fig. 2S) endowed by the Gd^{3+}-based complex **3**, the slightly increased quantum yield is up to 6.3% for the white-light-emitting *tris-β*-diketonate-(Dy^{3+}, $Gd^{3+}_{0.08}$) complex. This result is further reflected from the Dy^{3+}-centered longer phosphorescent lifetime (τ = 4.0 μs at λ_{em} = 578 nm; $^4F_{5/2} \rightarrow {}^6H_{13/2}$ transition) for *tris-β*-diketonate-(Dy^{3+}, Gd^{3+}) complex than that (τ = 3.2 μs) decayed for the Dy^{3+}-based complex **2**, also indicative of the strengthened energy transfer [24] during the dichromatic balance.

Inspiringly, motivated with the (Dy^{3+}, Gd^{3+}_x)-mixed content, the integrated color (the CIE chromatic coordinate × = 0.356, y = 0.346, the CCT of 4188 K and the CRI of 85) of the *tris-β*-diketonate-(Dy^{3+}, Gd^{3+}_x) complex at room temperature in solid-state, also falls within the white-light regime, as shown in Fig. 4, which is significantly incomparable to the yellow-light (the CIE chromatic coordinate x = 0.387, y = 0.395) of the solid-state Dy^{3+}-based complex **2**. Despite the inferior white-light performance (Φ_{em} = 4.4%) probably arisen from the aggregation-induced quenching [22], the realization of the straightforward white-light renders the binary *tris-β*-diketonate-(Dy^{3+}, $Gd^{3+}{}_x$) complexes' blending a new platform to solid-state white-light-emitting materials.

2. Conclusions

In summary, through the fluorescence titration of the yellow-light-emitting complex [$Dy(acac)_3$(5-Br-2,2′-bpy)] (**2**) with its isomeric blue-light-emissive complex [$Gd(acac)_3$(5-Br-2,2′-bpy)] (**3**), the dichromatic balance is strictly dependent on the (Dy^{3+}, $Gd^{3+}{}_x$)-mixed content, exhibiting the smoothly adjustable integration colors from yellow-lights to white-lights and blue-lights. This result renders the binary *tris-β*-diketonate-(Dy^{3+}, $Gd^{3+}{}_x$) complexes' blending a new platform to solid-state white-light-emitting materials, whose performance to be improved by the future structural optimization, is now under way.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

This work is funded by the National Natural Science Foundation (21373160, 21173165, 51704240), the Education Committee of Shaanxi Province (17JK1125), the Xi'an Science and Technology Plan Innovation Funds (2017CDWL-10, 2017CDWL-21, 2017CDWL-23, 2017CDWL-24, 2017CDWL-27, 2017CDWL-28) and the Training Program of Innovation and Entrepreneurship for Undergraduate of Xi'an University (2019058) in China.

Appendix A. Supplementary material

Supplementary data to this article can be found online at https://doi.org/10.1016/j.inoche.2020.107814.

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