T. TSUBOI[1]
H. MURAYAMA[2]
A. PENZKOFER[2, ✉]

Energy transfer in a thin film of TPD fluorescent molecules doped with PtOEP and Ir(ppy)₃ phosphorescent molecules

[1]Kyoto Sangyo University, Faculty of Engineering, Kamigamo, Kita-ku, Kyoto 603-8555, Japan
[2]Institut II—Experimentelle und Angewandte Physik, Universität Regensburg, Universitätsstrasse 31, 93053 Regensburg, Germany

ABSTRACT A thin film of triphenylamine dimer, N,N′-bis(3-methylphenyl)-N,N′-bis(phenyl)-benzidine (TPD), doped with *fac* tris(2-phenylpyridine) iridium (Ir(ppy)₃) and platinum octaethyl porphine (PtOEP) is characterized by photoluminescence emission measurements at several excitation wavelengths and photoluminescence excitation measurements at relevant emission wavelengths in the temperature range from 10 K to room temperature. The investigated film is a phosphorescent OLED material with singlet absorbing host (TPD) and triplet emitting guests [Ir(ppy)₃ and PtOEP]. At short wavelength excitation simultaneous triple band emission from singlet TPD, triplet Ir(ppy)₃ and TPD, and from triplet PtOEP is observed. Förster-type singlet-singlet state energy transfer from TPD to Ir(ppy)₃ and PtOEP, intra-component intersystem crossing, and Dexter-type triplet-triplet energy transfer between the substituents are studied.

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1 Introduction

Organic phosphorescent materials are used to improve the electroluminescence (EL) quantum efficiency of organic light emitting diodes (OLEDs) [1–8]. Several transition metal complexes like *fac* tris(2-phenylpyridine) iridium (Ir(ppy)₃) [9–23] and platinum octaethyl porphine (PtOEP) [24–29] show a high quantum efficiency in OLED devices [2–4]. These phosphorescent materials are doped into host materials to avoid self-quenching of photoluminescence in neat films [1, 30]. Special organic host materials like N,N′-bis(3-methylphenyl)-N,N′-bis(phenyl)-benzidine (TPD) [31–40] enhance the emission efficiency by excitation transfer from host molecules to guest molecules such as Ir(ppy)₃ and PtOEP [2–4, 41]. The study of the influence of the host material on the emission of the phosphorescent guest materials and the

study of the mutual interaction of the phosphorescent guest materials give important information on the development of phosphorescent OLEDs.

The structural formulae of TPD, Ir(ppy)₃, and PtOEP are shown in Fig. 1. A scheme of the lowest energy levels of TPD, Ir(ppy)₃, and PtOEP is shown in Fig. 2. The energy levels are taken from [2–4]. The lowest triplet state of TPD locates at lower energy (≈0.08 eV) than that of Ir(ppy)₃, while it
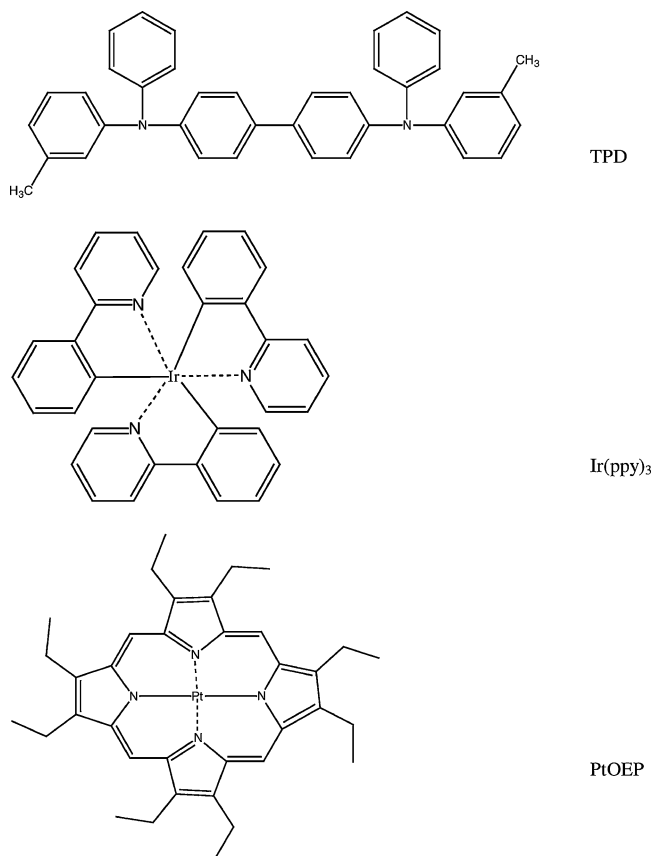
FIGURE 1 Structural formulae of N,N′-diphenyl-bis(3-methylphenyl)-biphenyl-4,4′-diamine (TPD, molar mass $M = 516.67$ g mol^{-1}), *fac*-tris(2-phenylpyridyl)Ir(III) [Ir(ppy)₃] ($M = 654.79$ g mol^{-1}), and 2,3,78,12,13,17,18-octaethyl-21H,23H-porphine platinum(II) [PtOEP] ($M = 727.84$ g mol^{-1})

✉ Fax: +49-941-9432754,
E-mail: alfons.penzkofer@physik.uni-regensburg.de
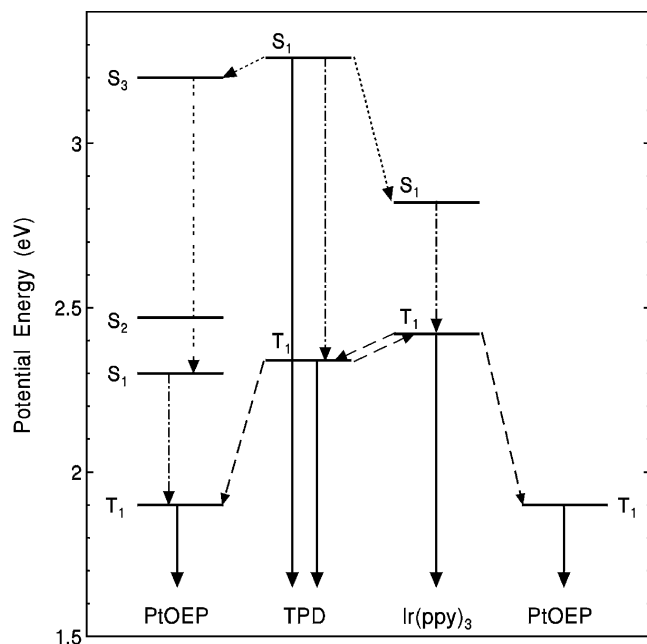
FIGURE 2 Energy level diagram and optical processes. *Solid arrows*: radiative transitions (fluorescence and phosphorescence). *Dashed arrows*: Dexter-type energy transfer. *Dotted arrows*: Förster-type energy transfer. *Triple-dotted arrow*: internal conversion. *Dash-dotted arrows*: inter-system crossing

locates at considerably higher energy (≈ 0.44 eV) than that of PtOEP. Ir(ppy)$_3$ doped in TPD forms an endothermic system for host to guest triplet excitation transfer (lowest guest level higher than lowest host level), while PtOEP in TPD presents an exothermic system for host to guest triplet excitation transfer (lowest guest level lower than lowest host level). Concerning the guest materials, Ir(ppy)$_3$ and forms an exothermic system for triplet excitation transfer from Ir(ppy)$_3$ to PtOEP.

Here we study the temperature dependence of the photoluminescence (PL) of a TPD film (host material) doped with Ir(ppy)$_3$ (endothermic triplet-triplet emitter system) and PtOEP (exothermic triplet-triplet emitter system) in order to explore the excitation transfer mechanisms towards the development of efficient OLED devices. Photoluminescence emission spectra and photoluminescence excitation spectra are analysed.

2   Experimental

A thin film of TPD (3-methyl-TPD) doped with Ir(ppy)$_3$ and PtOEP was formed by spin-coating. A solution of 20 mg/ml TPD in dichloromethane containing 4.75 wt% of Ir(ppy)$_3$ and 5 wt% of PtOEP was prepared and then spin-coated on a glass plate with a rotation speed of 1800 rpm. A film of 180 nm thickness was obtained.

Photoluminescence spectra were measured at various temperatures between 10 K and 300 K with a Spex Fluorolog-3 fluorophotometer. The excitation source was a 450 W Xe-lamp. Corrections were made for the luminescence spectrum wavelength sensitivity. Filters were used to avoid the half and second harmonics of the exciting light. Emission spectra and excitation spectra were recorded.

3   Results

3.1   *Absorption spectra*

In Fig. 3a the absorption cross-section spectra of TPD, Ir(ppy)$_3$, and Pt(OEP) are shown. They are taken from [30, 41, 42]. For wavelength $\lambda > 500$ nm only PtOEP is absorbing. In the wavelength range from 400 nm to 500 nm both PtOEP and Ir(ppy)$_3$ are absorbing, whereby the absorption of PtOEP dominates below 415 nm and above 465 nm. At short wavelengths, below 400 nm the host, TPD, and the guests, Ir(ppy)$_3$ and PtOEP, are absorbing, and the absorption cross-sections of all three components are reasonably high.

The absorption cross-section contributions, $x_i \sigma_{a,i}$, of the film components i are displayed in Fig. 3b. x_i is the mole-fraction of component i, and $\sigma_{a,i}$ is the absorption
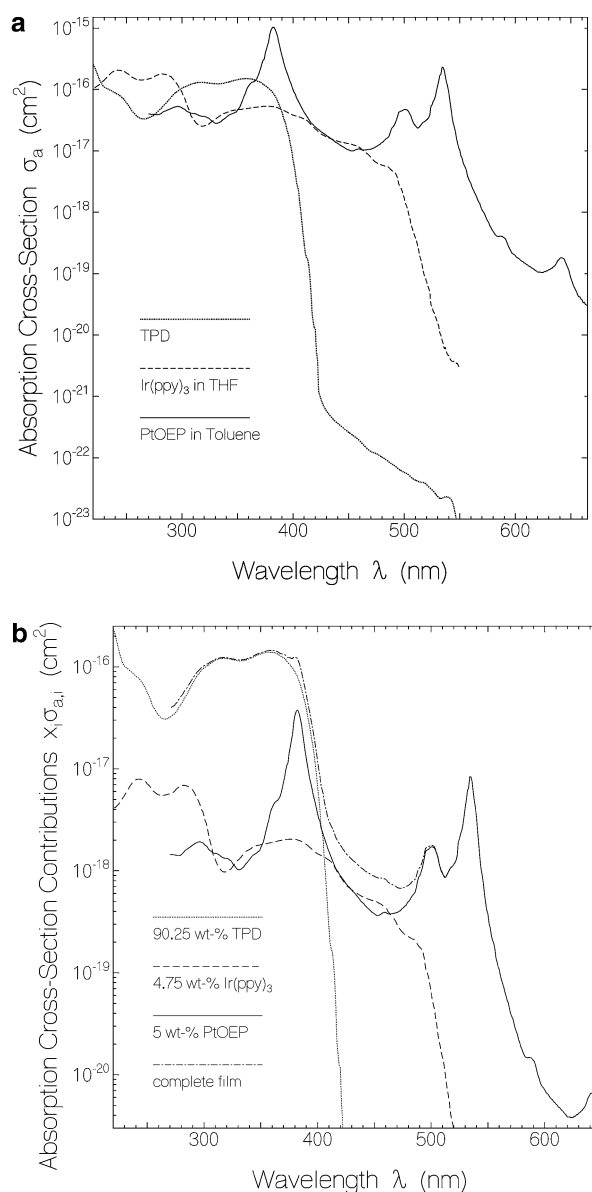
FIGURE 3 **a** Absorption cross-section spectra of TPD, Ir(ppy)$_3$, and PtOEP. **b** Absorption cross-section contribution of TPD, Ir(ppy)$_3$, and PtOEP in investigated thin film

cross-section of component i. The relation between the mole-fraction, x_i, and the weight-fraction, w_i, is given by

$$w_i = \frac{x_i M_i}{\sum_{j=1}^{n} x_j M_j} = \frac{x_i M_i}{\bar{M}}, \tag{1}$$

where M_i is the molar mass of component i, and $\bar{M}$ is the weighted molar mass. Here j runs over the three components, $1 = $ TPD, $2 = $ Ir(ppy)$_3$, and $3 = $ PtOEP ($n = 3$), and it is $x_1 + x_2 + x_3 = 1$. The applied weight fractions are $w_1 = 0.9025$ (TPD), $w_2 = 0.0475$ (Ir(ppy)$_3$), and $w_3 = 0.05$ (PtOEP). Equation (1) is solved for the mole-fractions, giving $x_1 = 0.03842$ ($M_1 = 654.79$ g mol^{-1}), $x_2 = 0.03639$ ($M_2 = 727.84$ g mol^{-1}), $x_3 = 0.9252$ ($M_3 = 516.67$ g mol^{-1}). Below 380 nm the absorption of TPD strongly dominates since 90.25 wt-% of the material belongs to TPD, in the region from 400 nm to 500 nm only Ir(ppy)$_3$ and PtOEP are absorbing, and above 500 nm only PtOEP is absorbing.

At a wavelength of $\lambda = 370$ nm a transmission of $T = 0.0845$ is measured. Using the relations, $T = \exp(-\sigma_a N d_f)$, $N = N_A \rho / \bar{M}$, and assuming a film mass density of $\rho = 1.1$ g mol^{-1}, we estimate a film thickness of $d_f \approx 180$ nm. N is the number density of molecules, N_A is the Avogadro number, $\bar{M} = 529.66$ g mol^{-1} is the weighted molar mass.

3.2   *Photoluminescence emission spectra*

In Fig. 4 normalized photoluminescence spectra of a TPD thin film (fluorescence, from [42]), of Ir(ppy)$_3$ dissolved in THF (phosphorescence, from [30]) and of PtOEP in toluene (phosphorescence, own measurements) are shown for comparison with results obtained here for the TPD film doped with Ir(ppy)$_3$ and PtOEP. The fluorescence maximum of TPD is at 400 nm, and the phosphorescence peaks of Ir(ppy)$_3$ and PtOEP are at 520 nm and 650 nm, respectively.
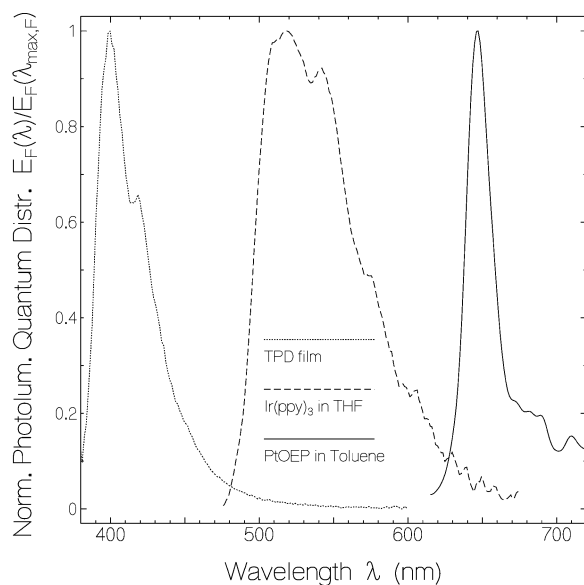

FIGURE 4 Normalized photoluminescence quantum distributions of a TPD thin film (from [7]), of Ir(ppy)$_3$ dissolved in THF (from [22]), and of PtOEP dissolved in toluene (own measurements)
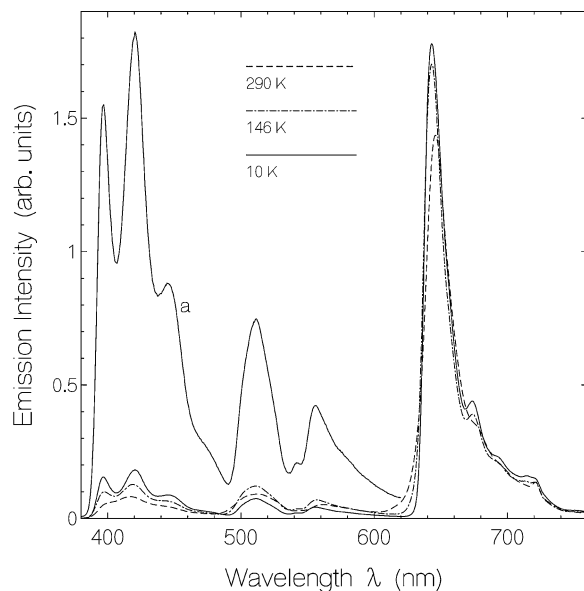
FIGURE 5 Emission spectra of investigated film excited with 360 nm light at 290 K, 146 K and 10 K. Curve a shows the 10-times enlarged spectrum of 10 K

Here the investigated film was irradiated with light at various wavelengths, λ_{exc}, in the range from 220 nm to 620 nm at temperatures between 290 and 10 K. Results are shown for $\lambda_{exc} = 360$ nm and 530 nm. Irradiation with 220–390 nm light gave rise to three emission bands resulting from TPD, Ir(ppy)$_3$, and PtOEP emission. Irradiation with 400–480 nm light led to Ir(ppy)$_3$ and PtOEP emission, and irradiation with 490–540 nm light caused only PtOEP emission. No emission was observed by irradiation with light of wavelength longer than 550 nm.
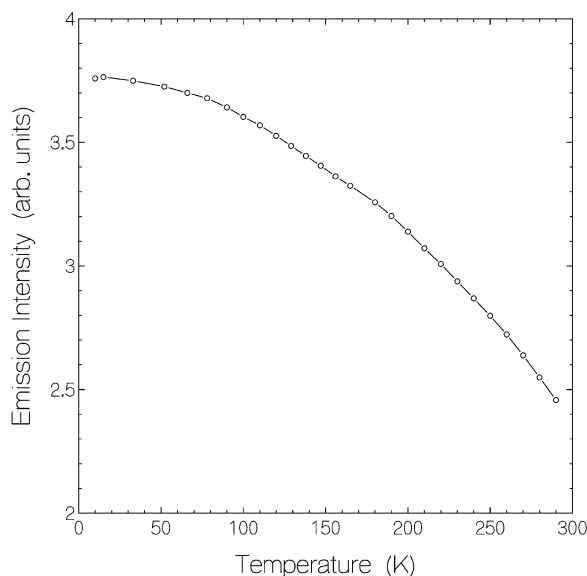
Figure 5 shows the PL spectra of the investigated film excited with UV light at 360 nm ($\approx$3.43 eV) at three temperatures (290 K, 146 K, and 10 K). The 360 nm light excites dominantly TPD (see Fig. 3b, only 1.4% of input light is directly absorbed by Ir(ppy)$_3$ and only 2.8% of input light is directly absorbed by PtOEP). Three photoluminescence bands are observed. One appears at 380–480 nm (with peaks at 396 and 419 nm and shoulders at about 442 and 470 nm at 10 K). It is due to the TPD host fluorescence. The second band, which appears at 480–620 nm (with a highest peak at 509 nm), is due to Ir(ppy)$_3$ phosphorescence and likely TPD phosphorescence. The luminescence band shape has a dip at about 535 nm which is different from the usual Ir(ppy)$_3$ emission spectrum (see e.g., [30, 43]) shown in Fig. 4. This dip is thought to be caused by re-absorption of wave-guided luminescence [44] by PtOEP, which otherwise would be scattered off and detected, since PtOEP has a high absorption cross-section at this wavelength position (Fig. 3a). The Ir(ppy)$_3$ phosphorescence is caused mainly by energy transfer from TPD to Ir(ppy)$_3$ [41] with some contribution from direct Ir(ppy)$_3$ excitation. The last band, at 630–700 nm (with peak at 640 nm), is due to PtOEP phosphorescence. It is caused by TPD to PtOEP energy transfer, Ir(ppy)$_3$ to PtOEP energy transfer, and partly by direct PtOEP excitation. The emission spectra due to TPD, Ir(ppy)$_3$, and PtOEP are consistent with previous measurements using thin films of TPD doped with Ir(ppy)$_3$, and of TPD doped with PtOEP [42, 43].
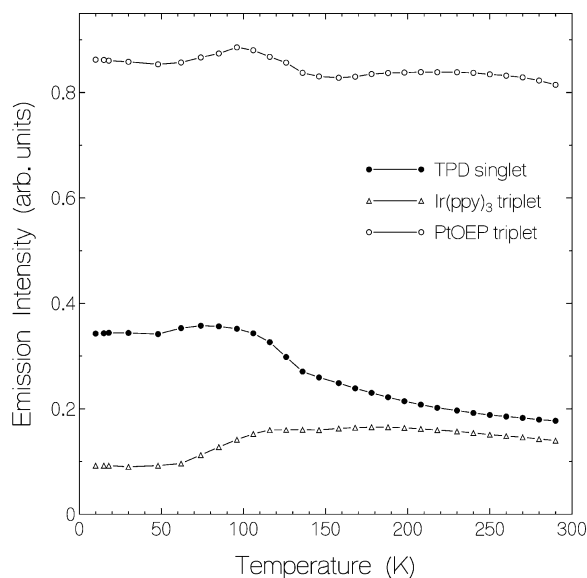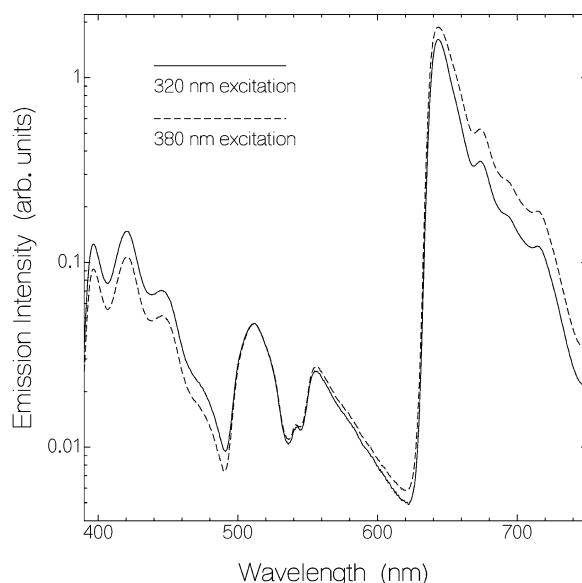
FIGURE 6 Temperature dependence of integrated intensities of the emission bands of TPD, PtOEP and Ir(ppy)$_3$ generated by 360 nm light excitation

FIGURE 8 Emission spectra of film excited with 320 and 380 nm light at 10 K

In Fig. 6 the temperature dependence of the intensities of the emission bands is shown for 360 nm excitation. The emission intensities were obtained from spectra similar to that shown in Fig. 5 by plotting them against energy (cm^{-1}) and integrating over the bands. The TPD emission intensity increases until about 70 K with decreasing temperature from 300 K and then remains approximately constant. The Ir(ppy)$_3$ intensity is nearly constant from room temperature down to 100 K and then decreases with decreasing temperature. The PtOEP emission intensity is nearly temperature independent.

Figure 7 shows the temperature dependence of the PtOEP emission intensity of the film excited with 530 nm light. The intensity increases with decreasing temperature from 290 K to 40 K and saturates below about 40 K. The same result was obtained by 480 nm excitation. A similar behavior was

FIGURE 7 Temperature dependence of the integrated emission intensity of PtOEP excited with 530 nm light

observed previously for a TPD film only doped with PtOEP [41].

In Fig. 8 the emission spectra of the film at 10 K are shown for excitation at 320 nm and 380 nm. In the case of excitation at 380nm (88.8% light absorption) the TPD emission band (400 nm to 480 nm) is slightly lower than in the case 320 nm excitation (93.3% light absorption). The effect is larger than expected from the different light absorption. The Ir(ppy)$_3$ emission band (480 nm to 620 nm) is practically the same for 380 nm and 320 nm excitation. The PtOEP emission band increases somewhat in the case of 380 nm excitation. There the absorption of PtOEP is strong and direct excitation takes place (27% and 1.1% of light absorption are due to PtOEP excitation at 380 nm and 320 nm, respectively). The observed PtOEP emission is thought to be due to direct PtOEP excitation and due to the energy transfer from TPD to PtOEP.
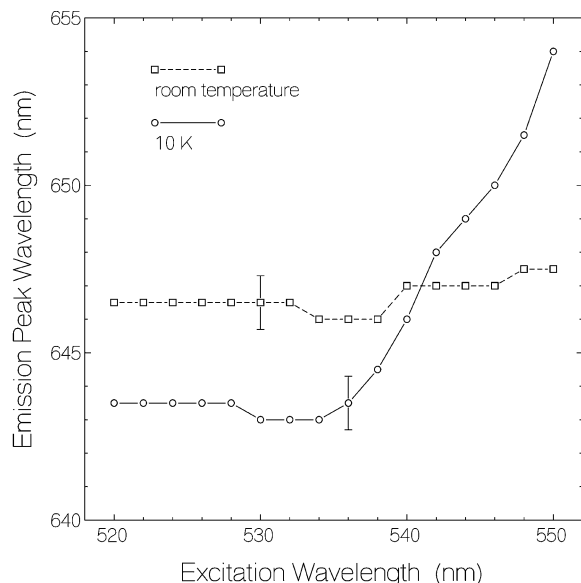
It was observed that, unlike the cases of the TPD and Ir(ppy)$_3$ emission bands, the PtOEP emission peak slightly shifts to shorter wavelength with decreasing temperature as shown in Fig. 9. This is consistent with the peak shift of the absorption band by temperature [43]. Additionally we observed that the emission peak wavelength of PtOEP depends on the excitation wavelength in the long-wavelength absorption tail at low temperature like 10 K, but no shift was observed at room temperature as shown in Fig. 10. Some frozen-in inhomogeneous distribution seems to be responsible for this behavior.

3.3   *Photoluminescence excitation spectra*

Figure 11 shows the excitation spectra for the 440 nm TPD singlet emission, the 512 nm Ir(ppy)$_3$ triplet emission, and the 645 nm PtOEP triplet emission at 10 K. Excitation bands with peaks at about 361 and 320 nm are quite similar to the absorption bands of TPD observed by previous measurements [42, 45–47]. Therefore the 361 and 320 nm excitation bands are attributed to TPD absorption.
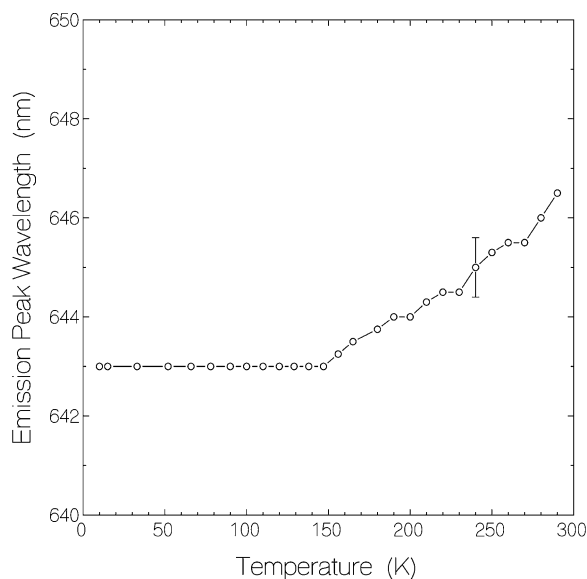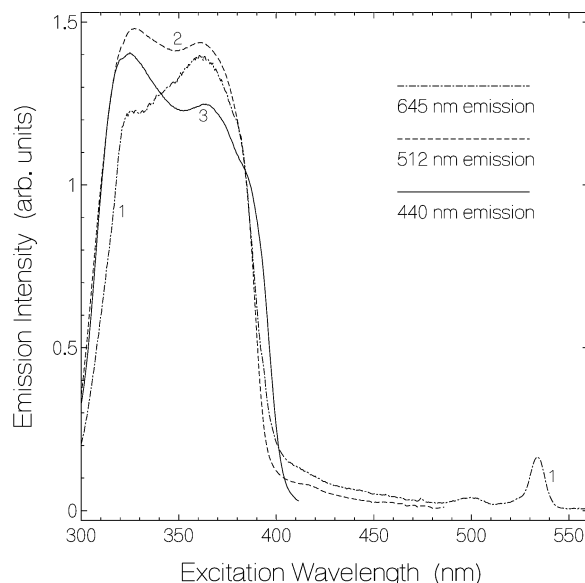
FIGURE 9 Temperature dependence of the peak wavelength of PtOEP emission band generated by 530 nm light excitation

FIGURE 11 Excitation spectra for 440 nm emission (curve 3), 512 nm emission (curve 2), and 645 nm emission (curve 1) at 10 K

FIGURE 10 Excitation wavelength dependence of the peak wavelength of PtOEP emission band at 285 K and 10 K

Intense excitation bands are observed at these wavelengths not only for the 440 nm TPD emission but also for the 510 nm Ir(ppy)$_3$, and 645 nm PtOEP emissions. This indicates that the Ir(ppy)$_3$ and PtOEP emissions arise from the excitation of the TPD host with subsequent excitation transfer. Weak excitation bands are observed at 400–470 nm for the 512 nm emission. These weak bands are attributed to Ir(ppy)$_3$ because they correspond to the absorption bands of Ir(ppy)$_3$ ([43] and Fig. 3). The photoluminescence excitation spectrum for 645 nm emission resembles the absorption spectrum of PtOEP in the wavelength range from 400 nm to 600 nm. Therefore it seems to be caused by direct PtOEP excitation. Some excitation transfer from Ir(ppy)$_3$ to PtOEP cannot be excluded in the wavelength range from 400 nm to 500 nm.

4 Discussion

Different temperature dependences are observed for the intensities of the PtOEP, TPD and Ir(ppy)$_3$ emission bands by 360 nm light irradiation as is shown in Fig. 6.

The TPD emission intensity is nearly constant with increasing temperature from 10 K to about 100 K and then decreases at higher temperature. The non-radiative relaxation (fluorescence quenching) increases with temperature above 100 K. The temperature dependence of TPD emission intensity observed here is similar to that of a neat TPD film without any guest molecules [41]. This indicates that the temperature dependence is not strongly disturbed by doping with PtOEP and Ir(ppy)$_3$.

The emission band from 490 nm to 620 nm (Fig. 5) is likely to be composed of the phosphorescence from Ir(ppy)$_3$ and TPD (see Fig. 2). The spectrum rises with temperatures up to 150 K and then it is rather constant. The excitation light at 360 nm is dominantly absorbed by TPD. After TPD singlet excitation, it occurs intersystem crossing in the TPD host to T_1(TPD), energy transfer from TPD to Ir(ppy)$_3$, relaxation to T_1(Ir(ppy)$_3$) within the Ir(ppy)$_3$ system, and thermodynamic equilibrium between T_1(Ir(ppy)$_3$) and T_1(TPD). A Förster-type energy transfer [48] from the lowest singlet state of TPD to a singlet excited-state of Ir(ppy)$_3$[S_1(TPD) + S_0(Ir(ppy)$_3$) → S_0(TPD) + S_n(Ir(ppy)$_3$)] followed by intersystem crossing to T_1(Ir(ppy)$_3$) is expected [6, 41]. A thermodynamic equilibrium between the lowest triplet state of Ir(ppy)$_3$ and the lowest triplet state of TPD is thought to exist due to Dexter-type energy transfer [49], according to T_1(Ir(ppy)$_3$) +S_0(TPD) ↔ T_1(TPD) + S_0(Ir(ppy)$_3$) [2–4, 41]. Below 120 K the phosphorescence in the 490 nm to 620 nm region reduces with decreasing temperature because the equilibrium between lower-level TPD triplet population and higher-level Ir(ppy)$_3$ triplet population shifts towards the TPD side and the TPD phosphorescence is less efficient than the Ir(ppy)$_3$ phosphorescence [41]. With a temperature

decrease the T_1(TPD) lifetime increases (from ca. 200 μs at room temperature[2] to the order of 0.1 s at 10 K [41]), leading to population accumulation in the lowest triplet state of TPD allowing delayed Ir(ppy)$_3$ phosphorescence and long-time TPD phosphorescence.

The emission band from 620 nm to 750 nm (Fig. 5) is due to phosphorescence from PtOEP. It is roughly temperature independent (Fig. 6). Again the 360 nm excitation light is practically only absorbed by TPD. A Förster-type energy transfer occurs from the lowest singlet state of TPD to a singlet excited-state of PtOEP[S_1(TPD) + S_0(PtOEP) → S_0(TPD) + S_n(PtOEP)] followed by intersystem crossing to T_1(PtOEP) [41]. Additional population of T_1(PtOEP) by Dexter-type energy transfer from Ir(ppy)$_3$ [T_1(Ir(ppy)$_3$) + S_0(PtOEP) → T_1(PtOEP) + S_0(Ir(ppy)$_3$)] and from TPD [T_1(TPD) + S_0(PtOEP) → T_1(PtOEP) + S_0(TPD)] is expected by the following fact: in a TPD film only doped with Ir(ppy)$_3$ and excited at 360 nm, the Ir(ppy)$_3$ phosphorescence is dominant (Fig. 7 in [41]), while here the PtOEP phosphorescence is dominant. The Ir(ppy)$_3$ phosphorescence is thought to be quenched by energy transfer to PtOEP.

As shown in Fig. 7, the emission intensity of PtOEP increases moderately with decreasing temperature when excited with 530 nm light. The non-radiative relaxation of T_1(PtOEP) increases slightly with temperature. The phosphorescence lifetime of PtOEP at room temperature is typically 110 μs [3]. Because of rising non-radiative $T_1 - S_0$ intersystem crossing with temperature generally phosphorescence emission of most compounds is only detectable at low temperature [50].

The photoluminescence excitation spectra in Fig. 11 show the absorption contributions to the selected emission bands and give information on the excited species which contribute to the emitting species either by direct excitation or excitation transfer [51]. Curve 3 shows the absorption contribution to the TPD S_1 fluorescence. It fits approximately to the TPD absorption spectrum of Fig. 3. Curve 2 gives the absorption contribution to the Ir(ppy)$_3$ and TPD T_1 phosphorescence. The excitation spectrum in the 400 nm to 500 nm region is due to direct Ir(ppy)$_3$ excitation, and the excitation spectrum below 400 nm is dominated TPD singlet excitation followed by Förster-type energy transfer and relaxation to T_1(Ir(ppy)$_3$) and T_1(TPD). Curve 1 gives the absorption contribution to the PtOEP T_1 phosphorescence. The excitation spectrum in the 500 nm to 600 nm region is due to direct PtOEP excitation, the excitation spectrum in the range from 400 nm to 500 nm is due to direct PtOEP excitation and Ir(ppy)$_3$ excitation followed by relaxation to T_1(Ir(ppy)$_3$) and then Dexter-type energy transfer from T_1(Ir(ppy)$_3$) to the triplet system of PtOEP. In the range between 400 nm and 360 nm the PtOEP excitation spectrum is caused by PtOEP absorption and TPD absorption followed by partial energy transfer from TPD to PtOEP. Below 360 nm the excitation spectrum is dominated by TPD absorption followed by energy transfer to PtOEP.

5   Conclusions

A TPD thin film doped with Ir(ppy)$_3$ and PtOEP has been characterized by photoluminescence emission spectra and excitation spectra measurements over a wide temperature range from 10 K to room temperature and comparison with the absorption behavior. Multi-band emissions and excitations have been obtained due to multiple component excitation and due to excitation transfer. The host (TPD) to guests (Ir(ppy)$_3$ and PtOEP), and guest (Ir(ppy)$_3$) to guest (PtOEP) excitation transfer due to Förster-type dipole-dipole interaction in the singlet systems and due to Dexter-type exchange interaction in the excited triplet states has been studied. Simultaneous photoluminescence emission in the wavelength range from 390 nm to 730 nm was achieved. Slightly increasing the Ir(ppy)$_3$ doping level and somewhat reducing the PtOEP doping level may result in a film with similar emission intensity of TPD, Ir(ppy)$_3$, and PtOEP giving a better white-light emission spectrum.

The reported results may be of interest in the application of the singlet emitter TPD and the triplet emitters Ir(ppy)$_3$ and PtOEP in organic light-emitting diodes.

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