

LETTER

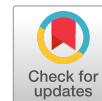
Thioxanthone-containing blue thermally activated delayed fluorescent emitter

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Thioxanthone-containing blue thermally activated delayed fluorescent emitter

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Accelerating reverse intersystem crossing (RISC) without sacrificing fast radiative decay is effective in suppressing efficiency roll-off (eRO) in thermally activated delayed fluorescence (TADF)-based organic light-emitting diodes. We here report a TADF emitter, CC-TXO-I, combining a bicarbazole donor (CC) and a sulfur-containing acceptor (TXO). The CC is used to accelerate radiative decay via moderate donor-acceptor torsion angle, and the TXO is expected to provide fast RISC by the heavy-atom effect. We realized very large rate constants of RISC (k_{RISC}) of $\sim 10^7 \text{ s}^{-1}$. Both k_{RISC} and rate constants of radiative decay of CC-TXO-I increased with increasing doping concentration, resulting in improved eROs.

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Organic light-emitting diodes (OLEDs) have several attractive characteristics such as high luminous efficiency, low power consumption, high color contrast, light weight, flexibility, and eco-friendliness.^{1–5)} To further improve the efficiency of noble-metal-free OLEDs, thermally activated delayed fluorescence (TADF) emitters have been actively investigated as new-generation luminescent materials.^{6–8)} Conventional fluorescent emitters can convert only singlet excitons into light, resulting in low internal quantum efficiency (IQE) of 25% at the maximum owing to spin statistics.⁹⁾ In contrast, TADF emitters can achieve IQE of 100% because they can convert not only singlet excitons but also triplet excitons into light via a reverse intersystem crossing (RISC) process from a triplet state (T_n , typically T_1) to an excited singlet state (S_m , typically S_1) (n and m are natural numbers with ≥ 1).⁶⁾ Actually, some TADF emitters showed an IQE of $\approx 100\%$.^{10–12)}

Nevertheless, TADF-based OLEDs have a serious problem of efficiency roll-off (eRO) in the high luminance region.^{9,13)} This is because triplet excitons generated on TADF emitters have relatively long lifetimes, which allow the quenching processes such as triplet-triplet annihilation (TTA) and singlet-triplet annihilation (STA).^{14,15)} Therefore, shortening an exciton lifetime can be an effective approach to reduce an exciton density in the emitting layer and then suppress eRO. A short exciton lifetime can be realized by fast RISC and fast radiative decay processes. When the transition between the highest occupied molecular orbital (HOMO) and the lowest unoccupied molecular orbital (LUMO) mainly contributes to the electronic configuration of S_1 , a large spatial overlap between the HOMO and LUMO leads to a large rate constant of radiative decay (k_r).^{16,17)} Besides, according to Fermi's golden rule, a rate constant of RISC (k_{RISC}) can be expressed as¹⁸⁾

$$k_{\text{RISC}} \propto |\langle S | \hat{H}_{\text{SOC}} | T \rangle|^2 \exp\left(\frac{-\Delta E_{\text{ST}}}{k_{\text{B}}T}\right), \quad (1)$$

where $|\langle S | \hat{H}_{\text{SOC}} | T \rangle|$, ΔE_{ST} , k_{B} , and T are the spin-orbit coupling matrix element value (SOCMEV), the energy gap between S_1 and T_1 , the Boltzmann constant, and temperature, respectively. This equation indicates that large SOCMEVs and small ΔE_{ST} are effective for fast RISC. Small ΔE_{ST} can be realized by a small spatial HOMO-LUMO overlap and it

is challenging to reconcile the large k_{RISC} and large k_r from the perspective of a HOMO-LUMO overlap.¹⁹⁾

In this study, we designed a TADF emitter by combining a bicarbazole donor and a sulfur-containing acceptor, aiming to simultaneously achieve both large k_{RISC} and large k_r . Since the heavy-atom effect is expected to increase SOCMEVs, TADF emitters have been designed containing non-metal heavy-atoms, such as sulfur,^{20–23)} selenium,^{24–26)} and halogens.^{27–29)} Among them, thioxanthone (TXO)-based emitters are especially noteworthy in terms of fast RISC.^{21,22)} For instance, MCz-TXO reported by us²²⁾ exhibited a very large k_{RISC} of $\sim 2 \times 10^8 \text{ s}^{-1}$. MCz-TXT reported by Yasuda et al.²¹⁾ showed a similarly large k_{RISC} of $1.1 \times 10^8 \text{ s}^{-1}$. Therefore, TXO was selected as the acceptor in this work to realize a large k_{RISC} .

Additionally, a moderate torsion angle between donor and acceptor is preferable to enlarge a HOMO-LUMO overlap and therefore k_r .³⁰⁾ In one example, CCX-I (Fig. S2)³¹⁾ containing 9H-3,9'-bicarbazole (CC) as a donor was determined to have a moderate torsion angle of 51° by the quantum chemical calculations because the carbazole unit contained in CC shows a relatively small steric hindrance. As a result, CCX-I was calculated to have a sizable oscillator strength between S_1 and the ground state (S_0), and experimentally exhibited the very large k_r of $5.9 \times 10^7 \text{ s}^{-1}$. On the basis of this report, we chose the CC as a donor.

On the basis of the above facts, a donor-acceptor type TADF emitter, 3-(9H-[3,9'-bicarbazol]-9-yl)-9H-thioxanthene-9-one (CC-TXO-I) [Fig. 1(a)], was designed. Quantum chemical calculations based on density functional theory (DFT) and time-dependent DFT with Tamm-Dancoff approximation were performed using the Amsterdam Density Functional program package (ADF2019.302).³²⁾ With triple zeta basis set and PBE0 functional, the geometry optimization of the ground state was performed. Using the structure, excitation energies, as well as the highest occupied natural transition orbitals (HONTOs) and lowest unoccupied natural transition orbitals (LUNTOs) of S_1 and T_n (where $n = 1, 2$), together with SOCMEVs between S_1 and T_n were calculated. The calculation results are shown in Fig. 1 and Table I. Table I also contains the results for CCX-I, which has a similar chemical structure to CC-TXO-I but the acceptor contains oxygen, not sulfur. The S_1 energy of CC-TXO-I was calculated to be 3.07 eV, which is similar to that of the blue

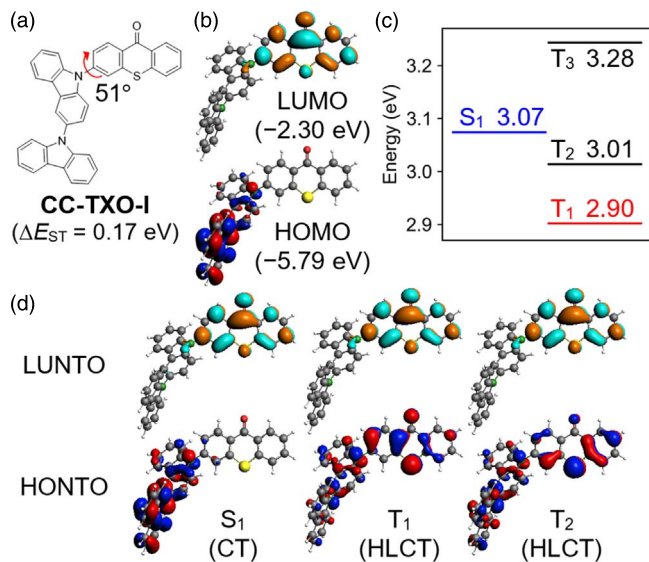


Fig. 1. (Color online) Calculation results for CC-TXO-I. (a) Chemical structure and torsion angle. (b) HOMO-LUMO distributions and energies. (c) Energy diagram. (d) HONTO-LUNTO distributions of S_1 and T_n ($n = 1, 2$).

Table I. Calculated electronic properties for CC-TXO-I and CCX-I.

Molecules	S_1 (eV) ^{a)}	$f^b)$	SOCMEV (cm^{-1})/ ΔE_{S1-Tn} (eV) ^{c)}	
			T_1	T_2
CC-TXO-I	3.07	0.1003	1.69/0.17	2.01/0.06
CCX-I ³¹⁾	3.10	0.1050	0.32/0.13	0.75/-0.20

a) The S_0 - S_1 excitation energy. b) Oscillator strength for the $S_0 \rightarrow S_1$ transition. c) The SOCMEVs and the energy difference between S_1 and T_n ($n = 1, 2$).

emitter, CCX-I (3.10 eV), predicting CC-TXO-I showed blue emission. As shown in Fig. 1(a), the torsion angle between CC and TXO was 51° . The HOMO and LUMO distributions were mostly spatially separated, but slightly overlapped as shown in Fig. 1(b). Besides, CC-TXO-I was calculated to possess a relatively small ΔE_{ST} of 0.17 eV and the large oscillator strength of 0.1003, indicating that its torsion angle is suitable for the simultaneous realization of a large k_{RISC} and large k_r . Moreover, the oscillator strength of CC-TXO-I is close to that of CCX-I (0.1050), evidencing that CC-TXO-I may show the large k_r as we designed (k_r of CCX-I is $5.9 \times 10^7 \text{ s}^{-1}$ in the doped film). Figure 1(d) shows natural transition orbitals (NTOs) for CC-TXO-I. The S_1 was attributed to a charge transfer (CT) characteristic, while T_1 and T_2 were assigned to hybridized local and charge transfer (HLCT) characteristics. In addition, large orbital coefficients were observed over the sulfur atom in HONTOs of T_1 and T_2 . As a result, notable SOCMEVs of 1.69, 2.01 cm^{-1} were calculated for $T_1 \rightarrow S_1$ and $T_2 \rightarrow S_1$ transition, respectively (Table I). Compared to SOCMEVs of CCX-I, the enhanced SOC of CC-TXO-I can be attributed to the heavy-atom effect resulting from the sulfur. A similar effect has been found in previous studies.^{24,25)} From these calculations, CC-TXO-I are expected to exhibit blue emission with large k_{RISC} and k_r .

Prior to photophysical characterizations, CC-TXO-I was synthesized and train sublimated (see detail in Supplementary Information). Figure 2 shows the fluorescence, phosphorescence, and UV-vis absorption spectra for $1 \times 10^{-4} \text{ M}$ CC-

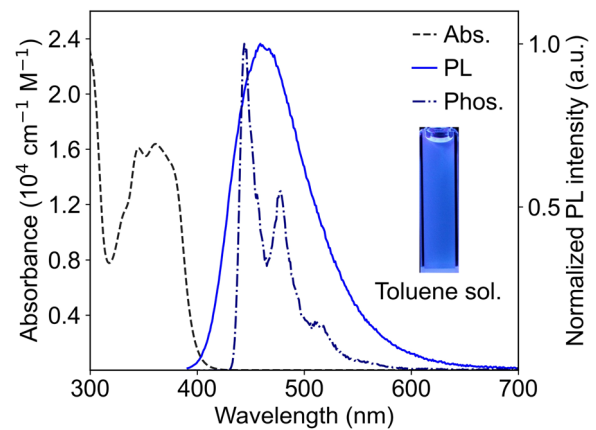


Fig. 2. (Color online) UV-vis absorption (dash line), fluorescence (solid line), and phosphorescence (dash-dot line) spectra of CC-TXO-I in oxygen-free toluene solution at a concentration of $1 \times 10^{-4} \text{ M}$. The phosphorescence spectrum was measured at 77 K and emission spectra were excited at 380 nm. A photograph of PL emission in toluene is also shown in the figure.

TXO-I in an oxygen-free toluene. In the UV-vis absorption spectrum, absorptions of CC and TXO are observed in the region of 300–350 nm³³⁾ and 350–380 nm,³⁴⁾ respectively. Also, the absorption in the region around 400 nm could be assigned to intramolecular CT absorption. The HOMO-LUMO gap was determined to be 3.09 eV from the onset of the absorption spectra, and CC-TXO-I showed blue emission with the emission peak maximum wavelength (λ_{MAX}) of 454 nm and CIE(x, y) = (0.17, 0.18) in the fluorescence spectrum. From the onset of both fluorescence and phosphorescence spectra, the experimental ΔE_{ST} was determined to be 0.19 eV, which agreed well with the calculated ΔE_{ST} of 0.17 eV.

We prepared several CC-TXO-I-doped films with different host materials and conducted photophysical measurements. As shown in Table SI, we found that 2,8-bis(diphenylphosphineoxide)dibenzofuran (PPF) was an appropriate host for CC-TXO-I in terms of the photoluminescence (PL) quantum yield (Φ_{PL}). Table SII summarizes the doping concentration dependence of λ_{MAX} s and Φ_{PL} s for vacuum-processed CC-TXO-I:PPF doped films. 6, 15, and 20 vol% CC-TXO-I:PPF films exhibited similar Φ_{PL} s of 84%, 83%, and 84%, respectively, while the PL spectra were red-shifted with increasing the concentration, probably due to the polarity of CC-TXO-I itself. Fig. S5(a) exhibits the transient PL decays of CC-TXO-I:PPF doped films at each doping concentration. All the films showed clear delayed emission of microsecond order, indicating TADF characteristics. The rate constants of CC-TXO-I:PPF doped film were determined with the equation reported in Ref. 34 and summarized in Table II. Owing to the heavy-atom effect of sulfur in TXO, CC-TXO-I exhibited fast RISC with the k_{RISC} of $\sim 10^7 \text{ s}^{-1}$ as we designed, which is three orders of magnitude larger than that of the CCX-I doped film ($\sim 10^4 \text{ s}^{-1}$). In contrast, the CC-TXO-I-doped films showed k_r of $\sim 10^6 \text{ s}^{-1}$. In comparison with the k_r of the CCX-I doped film ($\sim 10^7 \text{ s}^{-1}$), that of CC-TXO-I was not large as expected from the calculation results, although the relatively high Φ_{PL} of 84%. Additionally, we performed temperature-dependent transient PL decay measurement for 6 vol% CC-TXO-I:PPF doped film. As shown in Fig. S6, the delayed component increased with increasing

Table II. Lifetimes and rate constants of *X* vol% CC-TXO-I:PPF doped films.

<i>X</i> (vol%)	$\tau_p^{(a)}$ (ns)	$\tau_d^{(a)}$ (μ s)	$k_r^{(b)}$ (10^6 s $^{-1}$)	$k_{nr}^{(b)}$ (10^5 s $^{-1}$)	$k_{ISC}^{(b)}$ (10^9 s $^{-1}$)	$k_{RISC}^{(b)}$ (10^7 s $^{-1}$)
6	0.8	31.8	1.6	3.0	13	2.1
15	1.3	13.9	1.8	3.5	7.4	2.5
20	1.6	8.7	2.0	3.8	6.0	3.1

a) Prompt lifetime (τ_p) and delayed lifetime (τ_d). b) Rate constants of radiative decay (k_r), nonradiative decay from S_1 (k_{nr}), intersystem crossing process (k_{ISC}), and reverse intersystem crossing process (k_{RISC}).

temperature. This result confirmed that CC-TXO-I possessed TADF property.

Finally, we fabricated OLEDs using CC-TXO-I-doped films as emitting layers by vacuum deposition. The device structure is as follows: ITO (50 nm)/4,4'-cyclohexylidenebis[*N,N*-bis(4-methylphenyl)benzenamine] (TAPC) (60 nm)/1,3-bis(9,9-dimethylacridin-10(9*H*)-yl)benzene (mAP) (10 nm)/*X* vol% CC-TXO-I (*X* = 6, 15, 21): PPF(40 nm)/PPF (10 nm)/1,3-bis[3,5-di(pyridin-3-yl)phenyl]benzene (BmPyPhB)³⁵ (35 nm)/lithium quinolin-8-olate (LiQ) (1 nm)/Al (80 nm) as shown in Fig. 3(a). TAPC and BmPyPhB were used as a hole transport layer and an electron transport layer, respectively. mAP and PPF layers were inserted to block electrons and holes, respectively. Figures 3(b)–3(d) show external quantum efficiency (EQE)-luminance characteristics, electroluminescence (EL) spectra, and current density–voltage–luminance characteristics, respectively. The EL properties were summarized in Table III. CC-TXO-I-based OLEDs showed blue to greenish blue emission with maximum EQEs (EQE_{max}s) of 19.0%, 17.7%, and 17.2% for 6, 15, and 21 vol%, respectively. Among

them, 21 vol% device showed the smallest eRO; EQEs at 100 and 1000 cd m $^{-2}$ (EQE₁₀₀ and EQE₁₀₀₀, respectively) were 15.3% and 13.1%; the EQE₁₀₀ and EQE₁₀₀₀ dropped by 11.0% and 23.8% relative to its EQE_{max}, respectively. Although CC-TXO-I showed a very large k_{RISC} of $\sim 10^7$ s $^{-1}$, eRO was still observed. The reason can be attributed to the relatively large delayed lifetime (τ_d) in the CC-TXO-I doped films, inducing TTA and STA in the high luminance region. In fact, the larger the τ_d , the greater eRO (see Table II and Fig. 3; 6 vol% device exhibited the largest eRO). The three orders of magnitude larger k_{ISC} ($\sim 10^9$ s $^{-1}$) compared to k_r ($\sim 10^6$ s $^{-1}$) is considered to be a bottleneck in achieving eRO-free OLEDs.

In conclusion, we have developed a blue TADF emitter, CC-TXO-I, by combining CC as a donor and TXO as an acceptor. TXO was selected to enhance RISC due to the heavy-atom effect of sulfur and CC was selected to achieve a moderate HOMO-LUMO overlap. The quantum chemical calculations indicated that CC-TXO-I had large SOCMEVs of 1.69 cm $^{-1}$ between S_1 and T_1 , and 2.01 cm $^{-1}$ between S_1 and T_2 . The doped films using CC-TXO-I as a dopant

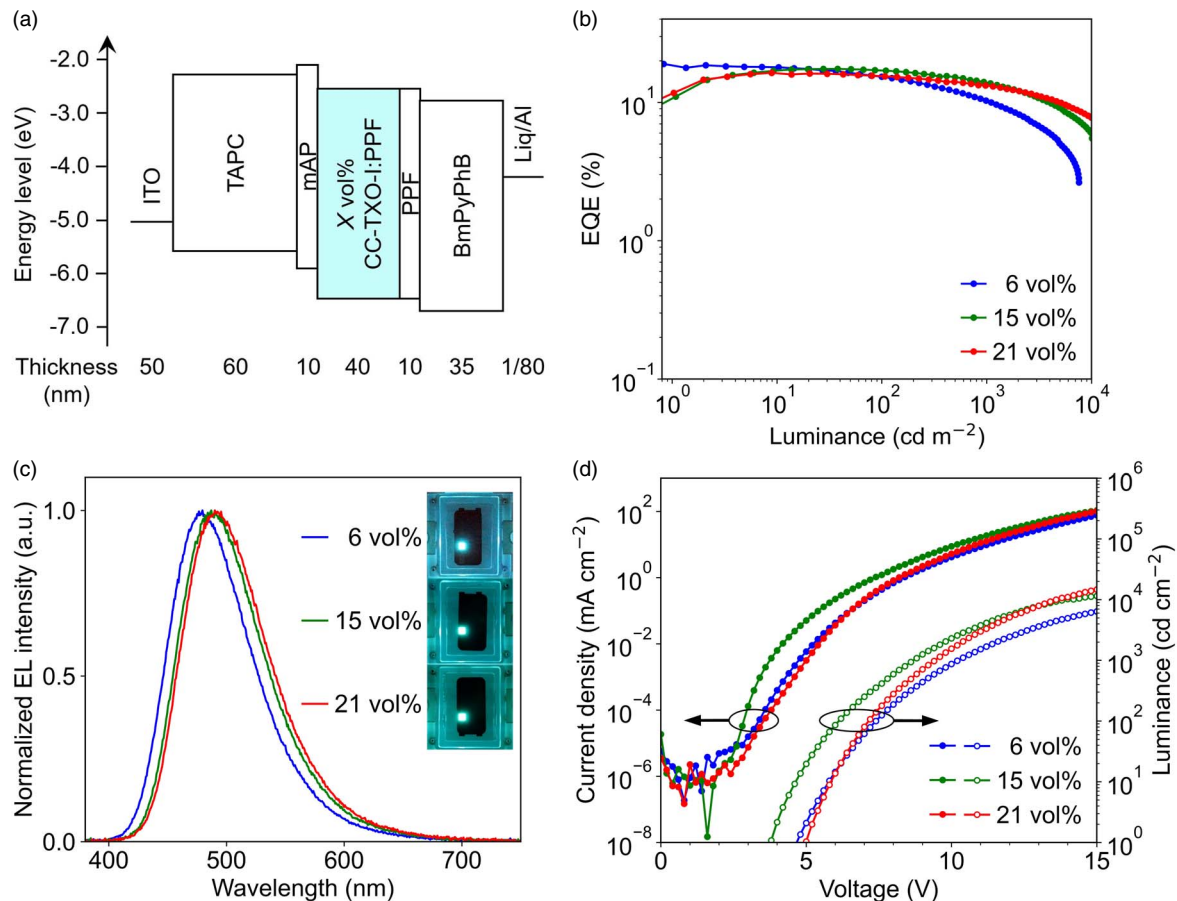


Fig. 3. (Color online) EL performances of CC-TXO-I-based OLEDs. (a) Device structure (*X* = 6, 15, or 21). (b) EQE-luminance characteristics. (c) EL spectra at 100 cd m $^{-2}$ with photographs of OLEDs. (d) Current density–voltage–luminance characteristics.

Table III. Performances of OLEDs using *X* vol% CC-TXO-I:PPF.

<i>X</i> (vol%)	CIE(<i>x</i> , <i>y</i>) ^{a)}	EQE _{max} ^{b)} (%)	EQE ₁₀₀ /EQE ₁₀₀₀ ^{c)} (%)	Roll-off ₁₀₀ /Roll-off ₁₀₀₀ ^{d)} (%)	<i>L</i> _{max} ^{e)} (cd m ⁻²)
6	(0.17, 0.28)	19.0	15.3/10.3	19.5/45.8	7592
15	(0.19, 0.36)	17.7	16.9/13.9	4.5/21.5	12 190
21	(0.20, 0.38)	17.2	15.3/13.1	11.0/23.8	17 200

a) CIE coordinates at 100 cd m⁻². b) Maximum EQE. c) EQE_Y means EQE at *Y* cd m⁻². d) Roll-off_Y = (EQE_{max} - EQE_Y)/EQE_{max}. e) Maximum luminance.

showed the TADF properties with the k_{RISC} of $\sim 10^7 \text{ s}^{-1}$, k_{r} of $\sim 10^6 \text{ s}^{-1}$, and the Φ_{PL} of 84%. The EQE_{max} of 19.0%, and the maximum luminance of 17 200 cd m⁻² were obtained for CC-TXO-I-based OLEDs at doping concentrations of 6 and 21 vol%, respectively. Both k_{RISC} and k_{r} increased with the doping concentration, which improved eROs. The doping concentration dependence suggests that accelerated RISC and accelerated radiative decay effectively realize OLEDs with reduced eROs.

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