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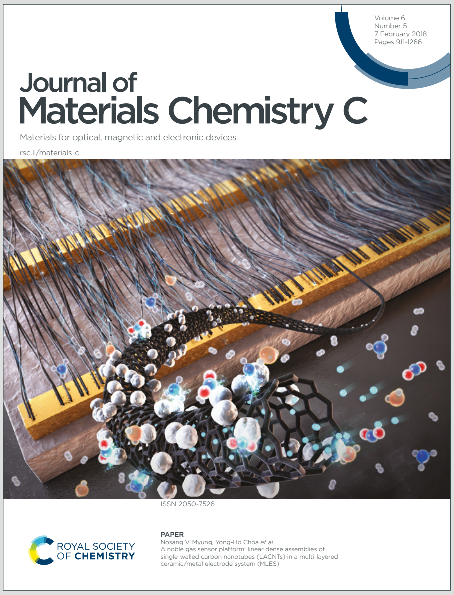

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COMMUNICATION

Room-temperature phosphorescence from a purely organic tetraphenylmethane derivative with formyl groups in both solution and crystalline states †

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Room-temperature phosphorescence of most purely organic compounds can hardly be observed in their solutions. Here, we report a tetraphenylmethane derivative with four aldehyde groups, which exhibites clear blue phosphorescence in its solution at room temperature. Furthermore, its crystal displays a nearly 7-fold higher phosphorescence quantum yield as compared to the solution.

Room-temperature phosphorescence (RTP) materials have received considerable attention recently owing to their promising applications in varying fields, including sensing,[1, 2] imaging,[3-5] organic light-emitting diodes,[6-8] information security,[9-12] etc. Organometallic complexes containing Ir, Pt, Os and other heavy metals, have so far been extensively investigated as bright phosphorescent emitters in both solution and solid states due to their strong spin-orbit coupling (SOC) and efficient intersystem crossing (ISC) promoted by these heavy metal atoms.[13, 14] However, most of metal-free organic materials are not efficient phosphors at room temperature due to weak SOC and slow ISC process, and non-radiative decay pathways of triplet excitons can be significant. Therefore, phosphorescence emission of purely organic phosphors at room temperature is usually observed in relative rigid environments constructed through the adsorption of phosphors onto solid substrates, the crystallization of phosphors,[15-19] and the embedding phosphors into rigid hosts such as polymer matrices,[1, 20, 21] steroidal molecules,[8, 22, 23] macrocycle cavities,[24-26] and hydrogen-bonded organic frameworks,[27] etc., which may suppress non-radiative dissipations of triplet excitons. Although great efforts have been devoted toward the development of purely organic phosphors in non-rigid solution states, only a limited number of purely organic RTP materials exhibit measurable phosphorescence in their solution states.[28-35]At the same time, solution phosphorescence has the potential for applications in biosensing and imaging, oxygen sensing and ion detection.[36-38]

As one of the earliest purely organic phosphors, tetraphenylmethane(TPM) with a highly steric and symmetric aromatic structure has been found to emit both ultraviolet fluorescence and green RTP in its crystal state.[39, 40] However, only ultraviolet fluorescence can be observed in its solution states. Aldehyde groups have been widely used to construct purely organic RTP materials, which can effectively enhance SOC and ISC.[18, 33] We herein report a versatile TPM derivative with four aldehyde groups, tetra(4-formylphenyl) methane (TPM-4CHO), which can emit RTP in both solution and crystalline states. In contrast, tetra(4-bromophenyl) methane (TPM-4Br) containing four heavy bromine atoms just shows ultraviolet fluorescence in its solution. Both X-ray structure analysis and theoretical calculations also reveal the importance of the aldehyde groups on the phosphorescent properties of TPM-4CHO.

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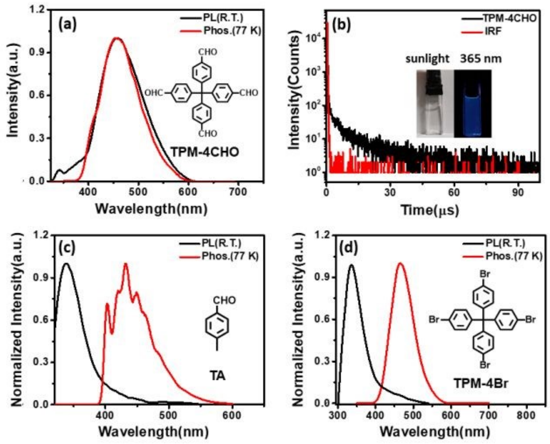

Fig. 1 Steady-state spectra (room temperature in deareated THF) and delay spectra (77 K, delay time: 10 ms) of (a)TPM-4CHO, (c)TA and (d)TPM-4Br in THF. (b)Lifetime decay curve of TPM-4CHO at room temperature in deareated THF and instrument response function(IRF). Insets in b show photographs of TPM-4CHO upon sunlight (left) and under 365 nm UV-light (right) in deareated THF at room temperature.

TPM-4CHO was obtained by a one-step *n*-butyllithium reaction (Scheme S1, ESI†).[41] The structure and purity were confirmed by ^{1}H-NMR, ^{13}C-NMR and gas chromatography (GC) spectrometry in Fig. S1 and S2 (ESI†). In tetrahydrofuran (THF) deareated with argon, TPM-4CHO is a colorless transparent solution in daylight (Fig. 1b, inset and Fig. S5, ESI†). When excited at 365 nm, TPM-4CHO emits clear blue light with the emission peak at 455 nm in deareated THF (Fig. 1a and 1b). It is noted that delayed emission spectra of TPM-4CHO in THF at room temperature and at 77 K are almost the same and also located at 455 nm with the emission lifetime of 5.13 μs at room temperature in deareated THF (Fig. 1a, 1b and Fig. S6, ESI†), which indicate that TPM-4CHO exhibits blue RTP in the deareated THF solution. The phosphorescence quantum yield (Φ_P) of TPM-4CHO in the deareated THF is 0.3%. The phosphorescent peak decreases gradually as the concentration lowered, accompanying with the appearance of ultraviolet emission bands with nanosecond scale lifetime, which are ascribed to fluorescence (Fig. S7, Table S1, ESI†). At the same time, dynamic light scattering (DLS) measurement shows that no aggregates are observed even in high concentration solutions, indicating that the phosphorescence emission of TPM-4CHO in the true solution state (Fig. S8, ESI†). TPM-4CHO has similar RTP emission in common organic solvents such as toluene, THF, dichloromethane, and acetone etc (Fig. S9, ESI†). In contrast, *p*-tolualdehyde(TA), the segment of TPM-4CHO, only exhibits a fluorescent emission peak at 338 nm with the lifetime of 0.38 ns at room temperature, which is significantly blue-shifted relative to its phosphorescence spectrum at 77 K (Fig. 1c). By replacing the aldehyde groups of TPM-4CHO with four heavy bromine atoms, TPM-4Br also only emits fluorescence with the emission peak at 336 nm and the lifetime of 0.71 ns at room temperature (Fig. 1d), just like TPM, which just has fluorescence emission in solution.[40] These results suggest that the combination of the TPM skeleton with the aldehyde groups may be responsible for the solution-state RTP emission of TPM-4CHO.

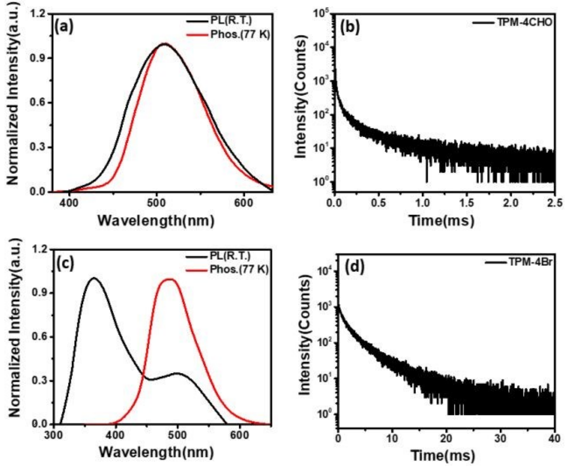

Fig. 2 (a) Steady-state spectra (room temperature), delay spectra (77 K, delay time: 50 ms) and (b) Lifetime decay curve of TPM-4CHO in crystal at room temperature. (c) Steady-state spectra (room temperature), delay spectra (77 K) and (d) Lifetime decay curve of TPM-4Br in crystal at room temperature.

The photophysical properties of TPM-4CHO crystal are investigated. The emission of TPM-4CHO crystal peaks at 509 nm at room temperature which is corresponding to its phosphorescent emission at 77 K (Fig. 2a). The crystal emission lifetime is 61.67 μs (Fig. 2b). The same emission at room temperature and 77 K and long lifetime indicate that TPM-4CHO crystal also display phosphorescent emission at room temperature. Comparing with TPM-4CHO in solution, TPM-4CHO crystal exhibits 54 nm red-shifted phosphorescent emission with the longer lifetime. Especially, the quantum efficiency in crystal increases to 2.3%, which is 7 times higher than that in solution. In contrast, TPM-4Br crystal displays two emission peaks at 361 nm and 498 nm with the lifetime of 0.36 ns

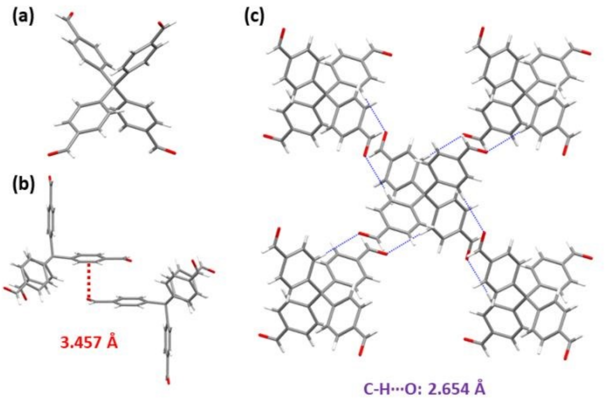

Fig. 3 Single crystal structure of TPM-4CHO. (a) Single molecular structure. (b) Intermolecular electronic coupling the aldehyde (n group) and benzene (π plane) groups in two TPM-4CHO molecules. (c) Intermolecular hydrogen bond interaction.

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and 2.26 ms, respectively. The emission band at long wavelength is consistent with the phosphorescent emission at 77 K, suggesting that both the fluorescence and phosphorescence exist in TPM-4Br crystal. However, the phosphorescence quantum efficiency of TPM-4Br is only 0.6%. As compared with TPM-4Br crystal, the single phosphorescence emission of the TPM-4CHO crystal with the higher phosphorescence quantum efficiency and the shorter phosphorescence lifetime reveals that aldehyde groups can promote ISC and phosphorescence radiation transition more effectively than heavy bromine atoms.

To gain the insight of the origin and difference of TPM-4CHO phosphorescence emission in its solution and crystal states, the single crystal of TPM-4CHO was cultivated by the solvent diffusion method in acetone and *n*-hexane (Table S2, ESI†). For one single TPM-4CHO molecule, the four benzaldehydes are connected by a sp^3-hybridized carbon, leading to the large steric hindrance between the benzene rings (Fig. 3a). In order to clearly show the steric effect of the TPM skeleton, relaxed potential surface energy scans of TPM-4CHO and TA on one of the dihedral angles are performed (Fig. S12, ESI†).[42, 43] When the dihedral angle deviates from the optimal configuration, the energy of TPM-4CHO rises rapidly, while the energy of TA hardly rises. Such a steric effect in TPM-4CHO may restrict the TPM skeleton motions and thus effectively suppress non-radiative transitions, which plays a key role in realizing RTP in solution. The benzaldehyde units between two TPM-4CHO molecules are almost parallel and the distance between the oxygen atom of aldehyde and the π-plane of the neighbouring benzene is 3.527 Å (Fig. 3b), which is analogous to that between the carbonyl (n) and carbazole (π) units of two neighboring molecules in RTP material Cz-BP reported by Chi et al.[44] Such an electronically coupling interaction of n and π units can facilitate ISC and radiation phosphorescence, leading to the higher RTP quantum efficiency of TPM-4CHO crystal comparing with TPM-4CHO solution. In its crystal, each TPM-4CHO also forms eight C-H···O=C hydrogen bonds with surrounding molecules, and the hydrogen bonds interaction distance is 2.654 Å (Fig. 3c), which is similar to the C-H···O=C interaction distance (2.673 Å) in the classic RTP benzophenone molecule.[45] The formation of hydrogen bonding network provides a rigid environment to further restrict nonradiative transition, resulting in the higher phosphorescence quantum efficiency and longer lifetime of TPM-4CHO in crystalline state than those in solution state. Additionally, compared to TPM-4Br, the formyl groups of TPM-4CHO may enhance the dipole-dipole interaction between the molecules. Therefore, TPM-4CHO shows a larger isosurface than TPM-4Br by intermolecular interaction analysis,[15, 46, 47] indicating that TPM-4CHO also has stronger intermolecular interactions (Fig. S13, ESI†), which may lead to its higher crystalline phosphorescence quantum efficiency (2.3%) than that of TPM-4Br (0.6%).

In order to further understand the RTP process of TPM-4CHO, the excited state energy (Fig. 4a) and SOC constants are calculated (Table S3-S4, ESI†). According to the EL-Sayed rule, when the electronic configuration of S_1 state is $^1(n, \pi^*)$ and the electronic configuration of T_1 state is $^3(\pi, \pi^*)$, or when S_1 is $^1(\pi, \pi^*)$ and T_1 is $^3(n, \pi^*)$, the ISC process will be effective.[48, 49] In a single TPM-4CHO molecule, ISC process (e. g. S_1-T_7) is from $^1(n, \pi^*)$ to $^3(\pi, \pi^*)$ and T_1 for radiation phosphorescence is a hybrid mixture of (n, π*) and (π, π*), which means that TPM-4CHO can effectively perform ISC and radiate phosphorescence (Fig. S16, ESI†).[50] For the ISC process from S_1-T_n, the maximum SOC constant is 12.35 cm^{-1} and the SOC constant of the radiation phosphorescence (from T_1 to S_0) is 73.61 cm^{-1}. These large SOC constants allow the TPM-4CHO for the efficient ISC process and phosphorescence radiation[51, 52]. Meanwhile, eight ISC channels can be achieved for a single TPM-4CHO molecule. The favourable electronic configuration, large SOC constants and eight channels for ISC may lead to room temperature phosphorescence in the solution of TPM-4CHO. For comparison, excited state energy and SOC constants for TA and TPM-4Br are also calculated (Table S7-S10, ESI†). Relative to TPM-4CHO, TA has only one possible ISC channel, which makes it difficult to achieve efficient RTP in its solution state. While for TPM-4Br, the remarkably lower SOC constants also prohibited phosphorescence radiation process in its solution at room temperature. Comparing with the TPM-4CHO monomer, the TPM-4CHO dimer has lower T_1 state energy of 3.24 eV, which is consistent with the spectral red shift of the crystalline state. The electron configuration during ISC and radiation phosphorescence of dimer is similar to that of monomer, where S_1-T_{10} is (n, π*) to (π, π*) and T_1 for radiation phosphorescence is a hybrid mixture of (n, π*) and (π, π*) (Fig. S17, ESI†). The maximum SOC constant of ISC and the SOC constant of radiation phosphorescence are 10.78 cm^{-1} and 76.73 cm^{-1}, respectively, which are comparable to those of monomer. However, the number of ISC channels of the dimer (20) is larger than that of the monomer (8), which is highly benefiting for the ISC process and may also account for the increased phosphorescence quantum efficiency in its crystalline state. As a consequence, the favourable electronic configuration, large SOC and many channels for

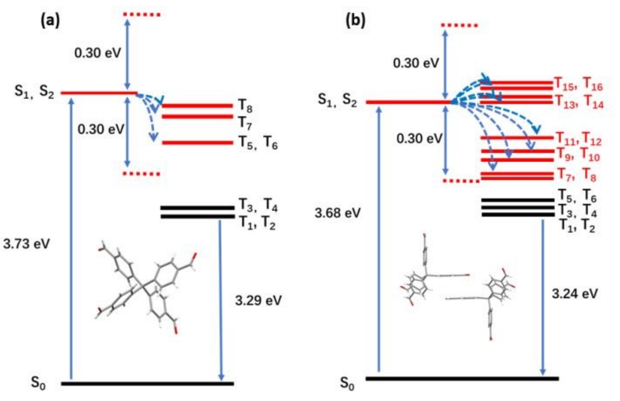

Fig. 4 Schematic diagrams showing the TD-DFT calculated energy levels of a TPM-4CHO monomer (a) and its dimer (b) at singlet (S_n) and triplet (T_n). states.

ISC result in RTP emission of TPM-4CHO in both solution and crystalline states.

In summary, we studied the optical properties of a readily prepared TPM derivative with four aldehyde groups (TPM-4CHO), which exhibits clear blue phosphorescence in common organic solvents at room temperature. Its crystal shows green phosphorescence and the quantum efficiency of its crystal is seven times as large as that of its solution. On the other hand, although tetra(4-bromophenyl) methane (TPM-4Br) containing four heavy bromine atoms also displays phosphorescence in crystalline state, only ultraviolet fluorescence could be observed in its solution. Single-crystal analysis and theoretical studies demonstrate the effects of the highly steric and symmetric TPM structure and the aldehyde groups, which may inspire new approaches in the development of new metal-free organic phosphorescent materials in both solution and crystalline states.

Conflicts of interest

There are no conflicts to declare.

Acknowledgements

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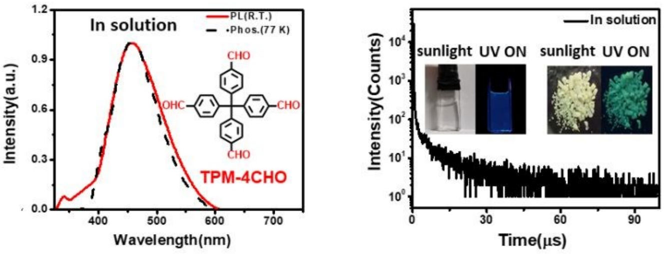

A tetraphenylmethane derivative with four aldehyde groups (TPM-4CHO) exhibits clear blue phosphorescence in common organic solvents at room temperature. Additionally, bright green phosphorescence can be observed in its crystal.