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Paintable Room Temperature Phosphorescent Liquid Formulations of Alkylated Bromonaphthalimide

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Abstract: Organic phosphors have been widely explored with an understanding that crystalline molecular ordering is a requisite for enhanced intersystem crossing. In this context, we explore the room temperature phosphorescence features of a solvent-free organic liquid phosphor in air. Given alkyl chain substitution varied the physical states of bromonaphthalimides, phosphorescence remained unaltered for the solvent-free liquid in air. As the first report, a solvent-free liquid of a long swallow tailed bromonaphthalimide exhibits room temperature phosphorescence in air. Doping of the phosphor with carbonyl guests resulted in enhanced phosphorescence and hence a large area paintable phosphorescent liquid composite with improved lifetime and quantum yield is developed.

Metal-free organic materials exhibiting room temperature phosphorescence (RTP) found useful in various applications.^[1,2] Easy synthetic protocols, structural tunability and air stability urge the use of organic phosphors.^[1-6] In recent years, organic small molecules decorated with various functional moieties such as halogens, boronate ester, carbonyl group etc.^[3] have been explored in RTP materials. These structural modifications eventually resulted in efficient intersystem crossing towards stable phosphorescence. However, organic phosphors mostly exhibit excellent RTP in crystalline state, than in the solution state.^[4] A strong control over the nonradiative decay in the rigid crystalline molecular packing supports intersystem crossing. Moreover, it reduces the triplet quenching by placing the molecules at finite distances. In short, RTP in organic phosphors is mainly controlled by the intermolecular interactions and overall organization of molecules in the crystal. In addition, an efficient

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triplet emission is established with the help of several other methods such as doping with molecules containing heavy atoms, matrix assisted isolation using polymers, micelles or cavitants.^[5,6] The availability of a support medium rigidifies the emitter and in turn strengthens the radiative relaxation process. Despite all the developments, still many key points to be addressed in the design of efficient organic phosphors. Moreover, a challenging problem related to the processability of these crystalline phosphors still persists. In this context, a new processable soft material called functional molecular liquid has been introduced as a replacement for solid luminescent materials.^[7-9] Here the viscous medium supports luminescence quantum yield via suppressing the nonradiative decay.^[8e] Given organic phosphors have been mostly tested in the crystalline form under inert conditions, RTP liquids have never been explored.

Naphthalimides are a class of rhylene dyes, which have been widely exploited in self-assembly, light harvesting, sensors, organic electronics etc.^[10] Here we have chosen two alkylated 4bromo-1,8-naphthlimides showing RTP in both crystalline and liquid states for a narrow singlet-triplet energy gap (ΔE_{ST}) that is essential for π - π * transitions and a heavy atom effect, which populate the triplet excitons by spin-orbit coupling.^[6b,11] These advantages found effective in delivering RTP not only in crystalline solid but in a new solvent-free liquid as well. Alkylated 4-bromo-1,8-naphthlimides 1 and 2 (Figure 1a) are synthesized and characterized by ¹H, ¹³C NMR and MALDI-TOF MS. Photophysical characterization of 1 and 2 are provided in Figure 1b, S1-S3 and Table S1. The melting (T_m) and glass transition (T_g) temperatures of **1** and **2** are determined by differential scanning calorimetry (DSC) (Figure 1c, S4). 2 shows $T_{q, offset}$ of -38.7 °C, which is different from the solid to isotropic state phase transition of **1** (101.8 °C). The high T_m of **1** and low $T_{g, offset}$ of **2**, allow 1 and 2 to exist as a crystal and a solvent-free liquid, respectively (inset; Figure 1c, S5). The melting transition of 1 originates from the favourable crystallization facilitated by the molecular skeleton of the alkyl chains whereas the random orientation of long aliphatic chains prevents crystallization of 2 and hence it remains as a RT solvent-free liquid. Thermogravimetric analysis (TGA) indicates that 2 extends stability to higher temperatures than 1 and 5% mass loss occurs at 267 °C and 212 °C, respectively (Figure 1d). The presence of large halo at around $2e = 20^{\circ}$ in X-ray diffraction (XRD) pattern of 2 corresponds to branched alkyl chains in the molten state (Figure 1e).^[8] In addition, the small and broad halo at around 2e $= 4^{\circ}$ might be the random distance among the aromatic moieties. The amorphous features seen in 2 is distinctly different from the reflection peaks of relatively ordered solid 1 (Figure 1e). It points to the direct effect of alkyl modification in tuning the physical

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characteristics of molecules. As a result, **1** forms needle-like crystal and **2** stays as RT liquid upon evaporation from CH_2Cl_2 solution (Figure S5-S7, inset; Figure 1c).



Figure 1. a) Chemical structure of **1** and **2**. b) Absorption and steady state emission spectra of **1** and **2** in CH₂Cl₂ solution ($C = 1 \times 10^{-5}$ M, I = 1 cm, $\lambda_{ex} = 342 \text{ nm}$). c) DSC thermograms of **1** and **2** at a scanning rate of 10 °C/min; insets show the photographs of **1** and **2**. d) TGA and e) XRD pattern of **1** and **2**.

Even though many RTP organic molecules have been exploited with the advantage of crystalline assembly, till date, no RTP in a solvent-free liquid has been described in literature. Zhang et al., have reported phosphorescence features of 1poly(methyl methacrylate) (PMMA) films in vacuum, but no detailed study on tunable luminescence features of 1 is available.[11b] Hence to begin with, we measured the phosphorescence of 1 and 2 in 2-methyltetrahydrofuran (MTHF) solution (Figure S8) and in neat (Figure 2a) and found that both derivatives are phosphorescence active under tested conditions. When solid 1 is excited at 345 nm, phosphorescence spectrum exhibited two peaks with $\lambda_{\rm max}$ around 546 nm and 580 nm (Figure 2a). Compared to 1, 2 showed a red shifted peak with $\lambda_{\rm max}$ around 594 nm (Figure 2a), which is identical to that of MTHF solution and neat form at -196 °C (Figure S8, S9).^[12] It supports the molecular design of 2 lacking crystalline ordering to exhibit RTP. Aa a direct evidence for the control of molecular structure on photophysical properties, the bulky alkyl substituent on 2 doesn't allow to form excimer even at low temperature (Figure S9, S10). 1 exhibited a reversible tunable luminescence consists of phosphorescence, excimer and monomer emissions, demonstrating a luminescence based temperature thermometer for low temperature monitoring (Figure S10). Phosphorescence lifetime of 1 and 2 were found to be 6.2, 5.7 ms at 25 °C and 319, 210 ms at -196 °C, respectively, (Figure 2b, S11, S12). Here we have demonstrated RTP in a solvent-free liquid, for the first time, without the assistance of very strong intermolecular interactions,

which normally suppress the nonradiative decay processes. Generally, the lack of molecular ordering in noncrystalline materials weakens intersystem crossing. However, in the case of **2**, the viscous nature plays a key role in controlling the nonradiative decay processes^[8e] and promotes the triplet emission. The complex viscosity of **2** was found to be 0.79 Pa·s at 1 rads⁻¹, which is in the range of reported organic liquids (Figure S13).^[8] Sada et al., have recently demonstrated the high fluorescence quantum yield of tetraphenylethene based solvent-free liquids through controlled nonradiative decay by the virtue of inherent viscous medium.^[8e]



Figure 2. a) Normalized phosphorescence spectra of solid 1 and solvent-free liquid 2 at 25 °C in air (λ_{ex} = 345 nm). b) Phosphorescence lifetime decay profile of neat 2 recorded at 25 °C (λ_{ex} = 345 nm and λ_{mon} = 594 nm). c) HOMO and LUMO of 2 and d) energy level diagram showing ΔE_{ST} for 1 and 2 from DFT calculations.

To get a deeper understanding of the phosphorescence from an amorphous solvent-free liquid, luminescence features of 2 is monitored in the presence of PMMA (Figure S14). Phosphorescence measurements of 2-PMMA mixture clearly point out the requisite of Br...O interaction to exhibit RTP in air. The reduced nonradiative decay due to rigidification of the molecule by wrapping with polymer is not favouring intersystem crossing. Monomer emission (350-500 nm) dominates over phosphorescence (550-650 nm) even at -196 °C (Figure S14). Moreover, 2-PMMA thin film also exhibits major contribution from the monomer emission at 25 °C. The presence of PMMA isolates the molecules and hence narrows down the possible intermolecular interaction. Even though 2 exists as a RT liquid, the presence of weak Br...O interaction cannot be ruled out and is supported by the broad UV-Vis absorption of the molecule in neat form (Figure S2). Phosphorescence studies of 2-PMMA conclude the requisite of available Br...O interaction to support RTP in 2.

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Single crystal X-ray analysis enables to study the molecular arrangements of 1, which crystallizes from ethanol in the triclinic space group P-1 (CCDC: 1873219) (Figure S15). A selfassembled molecular arrangement is anticipated to reduce nonradiative decay, additionally provides charge-transfer character through halogen, hydrogen bonding and leads to efficient intersystem crossing.^[11b] However, 2 remain as a free flowing RT liquid, devoid of strong molecular ordering. It has to be noted that the steady decrease in crystallinity from 1 to 2 imparted only a marginal effect on phosphorescence of 2 (Figure 2a, 2b). Phosphorescence of amorphous 1 prepared by mechanical grinding retained similar phosphorescence features, but with decreased intensity (Figure S16). This control experiment shows that crystallinity is not highly required whereas minimum intermolecular interactions as in the case of 2, which enhance the spin orbit coupling might result in efficient intersystem crossing (Figure S16).

In order to probe the phosphorescence of 2 further, a series of TD-DFT computations were carried out at the B3LYP/6-31+G(d) level of theory. The UV-Vis absorption of 2 originates from HOMO to LUMO electronic transition, where HOMO is located on the naphthalene ring (π -type) and LUMO is a delocalized p-type orbital with significant Br-C P-orbital overlap (Figure 2c, S17, Table S2, S3). On examining the relative computed energies for the singlet (S_n) and triplet (T_n) states for 2, there is a triplet state (T₂) that is nearly degenerate with the first singlet excited state (S1) (Figure 2d). Thus it appears that both the compounds have energetically well-matched states to enable efficient singlet-triplet crossing to occur; once T_2 is populated, then relaxation through the triplet manifold to the T_1 state is possible, followed by emission (Figure 2d). Here it confirms that along with the viscous medium,^[8e] a low singlettriplet energy difference (ΔE_{ST}) facilitates the intersystem crossing leading to RTP of 2.



Figure 3. a) Phosphorescence spectral changes of **2** with increasing equivalents of **A2** at RT in air; inset shows the chemical structure of carbonyl guests **A1-3**. b) Variation of phosphorescence intensity at λ_{max} of all the composites of **1** and **2** (λ_{ex} = 345 nm). c) Enhanced luminescence of RTP liquid composite **2+A2** (1:1) in air.

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Supramolecular two component assemblies are proved to be an excellent way to heighten RTP.^[13] An ample way of improving phosphorescence by mixing with carbonyl compounds such as A1-3 (Inset; Figure 3a), which has intrinsic tendency to support RTP via halogen bonding,^[14] has been adopted. A significant increment of phosphorescence intensity is observed for 2 upon mixing with increasing the equivalents of A3 and A2 at RT in air (Figure 3a-c, S18). Among all the combinations, 2+A2 (1:1) showed maximum phosphorescence enhancement (Figure 3b). The coassembly formation suppresses monomer emission of 2 and improves phosphorescence (Figure 3c, S19). A slight variation of T_{a.offset} of 2 from -38.7 °C (0 eqv.) to -33.4 °C (1.0 eqv.) upon increasing the ratio of A2 was observed (Figure 4a, S20). IR spectral variations of the composites confirm the presence of multiple halogen bonding interactions (Figure S21, Table S4). Similar phosphorescence enhancement was exhibited by 1 also in combination with A3 and A2 (Figure S22-24) and fluorescent microscope images showed the formation of luminescent crystalline rods (Figure S25).



Figure 4. a) DSC thermograms in the heating trace of RTP composite of 2 with varying ratio of A2 at a scanning rate of 10 $^{\circ}$ C/min. Variation of b) phosphorescence quantum yield and c) lifetime of 2 upon composite formation with A3 and A2. d) Photograph of large area (10x10 cm) coating of 2+A2 (1:1) RTP liquid composite in air.

Natural bond orbital analysis shows higher probability for **1** and **A2** to interact in plane mode with Br···O distance of 2.9 Å and 2 kcal/mol higher stabilization interaction energy than that of out of plane mode (Figure S26, Table S5, S6). Hence halogen bonding assisted in plane intermolecular interaction is expected to promote spin-orbit coupling to facilitate efficient triplet generation.^[4a] In addition, it will reduce vibrational losses at the carbonyl and hence activate phosphorescence emission. The advantage of carbonyl doping is reflected in enhancing both phosphorescence quantum yield and lifetime (Figure 4b, 4c, S27, S28, Table S7). However, detailed experiments have shown that no phosphorescence enhancement is observed for both **1** and **2** with **A3-1** in solution (Figure S29-33). Similarly, no variation in phosphorescence was found for **1** and **2** with **A1** (Figure S34).

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We extended the carbonyl guest to other molecules A4-15 (Scheme 2) and found that only anthraquinone A14 effectively improved phosphorescence (Figure S35, S36). Since 2 exhibited improved phosphorescence in combination with A2 (1:1), a liquid phosphor composite was developed by mixing of 2 with A2 (1:1) and this combination realised a large area (10x10 cm) paintable composite with improved RTP (Figure 4d). RTP liquid composite 2+A2 exhibit stable luminescence features, but when the ratio of A2 is high (>1), composite solidifies immediately. We strongly believe that RT liquid feature of 2 enabled to deliver a paintable RTP composite and this demonstration will be a potential alternate for the tedious and expensive processing methods of crystalline RT phosphors.

In conclusion, hitherto unknown RTP organic liquid is materialised by introducing a long branched alkyl chain on bromonaphthalimide. A comparison clearly showed that even liquefaction of bromonaphthalimide allowed the molecule to retain RTP. The suppressed nonradiative decay by available viscous medium, reduced $\Delta E_{\rm ST}$ and presence of weak Br····O halogen bonding facilitated RTP for solvent-free liquid in air. As a new strategy, paintable RTP composite with significantly improved phosphorescent quantum yield and lifetime is prepared by mixing the liquid phosphor with carbonyl guests. A relatively large area (10x10 cm²) RTP coating is realised by using the liquid composite paint. We strongly believe that our RTP liquid composite formulation will be much appreciated as this soft material exhibits the potential to make innovative changes in large area flexible lighting applications.

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Keywords: phosphorescence • organic liquids • luminescent thermometer • liquid phosphor • excimer

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A new strategy for hitherto unknown room temperature phosphorescent liquid and large area paintable formulations with enhanced phosphorescence.



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