In recent years, there has been a growing interest in purely organic materials showing ultralong room-temperature phosphorescence with lifetimes in the range of seconds. Still, the longest known phosphorescence lifetimes are only achieved with crystalline systems so far. Here, a rational design of a completely new family of halogen-free organic luminescent derivatives in amorphous matrices, displaying both conventional fluorescence and phosphorescence is reported. Hydrogen bonding between the newly developed emitters and an ethylene-vinyl alcohol copolymer (Exceval) matrix, which efficiently suppresses vibrational dissipation, enables bright long-lived phosphorescence with lifetimes up to 2.6 s at around 480 nm. The importance of the chosen matrix is shown as well as the implementation in an organic programmable luminescent tag.

Materials exhibiting persistent luminescence with long emission lifetimes have attracted the interest of researchers in various fields such as molecular sensing, organic light-emitting diodes, bioimaging, displays, or anticounterfeiting.[1-16] A long phosphorescence lifetime is a key advantage over conventional fluorescence for read-out of images since the detection window is maximized. If used as safety features, a long afterglow assures long visibility in the dark. Also in bioimaging, if only low intensity is achieved, it could help to increase the detection probability. There is an extensive number of inorganic and metal–organic complexes showing long-lived emission. However, high cost and toxicity, high preparation temperatures in case of inorganic material, as well as limited availability are severe drawbacks. Special attention has recently been paid to purely organic materials[17-19] showing long-lived room temperature phosphorescence (RTP). Due to the inefficient intersystem crossing (ISC) based on weak spin–orbit coupling and the high nonradiative decay rate of the triplet excitons as well as quenching factors like oxygen, different strategies to achieve RTP have been developed in recent years. Among those, host–guest doping,[20-23] H-aggregation,[24-28] supramolecular frameworks,[24] polymers,[29-38] carbon dots[39-41] and small molecules[42] can be found. In any case, the emitting molecules must be embedded in a rigid environment, be it a crystalline structure or a polymer host, causing a confinement of molecular motion. So far, the organic materials with the longest phosphorescence lifetimes of 2.45 s,[27] 2.50 s,[45] and 2.62 s[46] relate to crystals, whereas amorphous materials show lifetimes of up to around 1 s.[20] Still, the long-term stability of such crystalline structures remains unreliable and the preparation of thin films from these materials persists challenging. Therefore, we focused on another strategy, the embedding of the emitter in a rigid polymer matrix. Quite a number of groups have reported RTP in such systems, using host–guest and/or guest–guest interactions. Kwon et al. as well as Su et al. used a poly(vinyl) alcohol matrix to rigidify the system and showed that hydrogen bonds formed between the emitter and the host lead to the appearance or enhancement of an already existing phosphorescence.[4,47] They present three types of intermolecular hydrogen bonds—guest–host, guest–polymer, and polymer–polymer. These bonds support the ISC, confine the vibration of the guest molecules, and inhibit the diffusional motion thus enhancing the phosphorescence.

Our group just recently reported the ultralong room-temperature phosphorescence (URTP) behavior of entirely transparent and smooth films containing the emitter embedded in a rigid PMMA (poly(methyl methacrylate)) matrix, covered with an oxygen-barrier layer (Exceval, an ethylene-vinyl alcohol copolymer). These functional films can be used as programmable luminescent tags (PLTs) under ambient conditions.[48,49] The films are prepared in two steps. The emitter is dissolved in
anisole, mixed with PMMA, and spin coated onto quartz substrates. Afterward, an oxygen-barrier layer is added, which leads to the ability for their use in an aerated environment. Since the oxygen barrier is water soluble, it was natural to develop a water-soluble emitter to be directly mixed into the oxygen barrier, enhancing the phosphorescence lifetime by forming hydrogen bonds with the polymer. Thus, a well-known emitter, tetraphenylbenzidine (TPB), was modified with four carboxylic acid moieties to lead to \( N,N,N',N'-\)tetrakis(4-carboxyphenyl)benzidine which shows, mixed into the oxygen barrier, a longer phosphorescence lifetime compared to the same molecule embedded in PMMA.\(^{[30]}\) Additionally to hydrogen bonds, the nature of substituents in the molecular structure of the emitter can play a significant role in the increase of the phosphorescence lifetime. Further, we recently reported that the origin of phosphorescence for TPB is the biphenyl motif.\(^{[31]}\) Based on this, our group pursued several approaches with different side groups and substituents to extend the phosphorescence lifetime even more, where the presented one turned out to be most efficient.

Here, we report the phosphorescence properties of a completely new family of halogen-free organic derivatives displaying, to the best of our knowledge, the first example of URTP from completely new family of halogen-free organic derivatives displaying, to the best of our knowledge, the first example of URTP from purely amorphous, noncaged and steroid free solution-processed materials with lifetimes > 2 s. We focused on 4,4′-bis(diethylphosphonomethyl)biphenyl (BDPB) and analogous derivatives (BDPB-OH, BDPB-Ac) characterized by a biphenyl core with diethylphosphonomethyl side-groups (Figure 1). Additionally, a derivative with a terphenyl core was chosen (BDPDPPT). Different synthetic routes\(^{[32-36]}\) were used to obtain the emitter BDPB, BDPB-Ac, BDPB-OH, and BDPDPPT (Figure 1). BDPB is synthesized by nucleophilic substitution, the so called Michaelis Arbusov reaction, of 4,4′-bis(bromomethyl)biphenyl (I) with triethylphosphate (PO\(\text{Et}_3\)) at reflux in high yield (95%). BDPB-Ac is synthesized in the following multi-step synthesis: A nucleophilic substitution of 1 with potassium cyanide (KCN) leads to the corresponding dinitrile, which forms the corresponding diester after esterification with H\(_2\)SO\(_4\) and ethanol at reflux. Thereafter a Wohl–Ziegler bromination with N-bromosuccinimide (NBS), azobisisobutyronitrile (AIBN), and subsequent nucleophilic substitution of the formed brominated product with P(O\(\text{Et}_3\)) leads to the target molecule BDPB-Ac with a total yield of 7.4%. BDPB-OH is synthesized by electrophilic addition of diethylphosphate (HPO\(\text{Et}_2\)) to 4,4′-biphenyldicarboxaldehyde 2 with KF/Al\(_2\)O\(_3\) at room temperature with a yield of 43%. The synthesis of BDPDPPT starts with the trimerization of 4′-methylacetophenone 3 followed by Wohl–Ziegler bromination and subsequent nucleophilic substitution of the brominated species with P(O\(\text{Et}_3\)). The total yield is 38%. Detailed procedures are described in the Supporting Information.

For the measurement of the luminescence properties, the emitters get embedded in both PMMA and Exceval, since the latter contains OH-groups and has therefore the ability to form intermolecular hydrogen bonds with the emitters, presumably leading to an increase of the phosphorescence lifetime. To obtain smooth and transparent films, the targets are dissolved in either anisole, mixed with a solution of PMMA or tetrahydrofuran (THF), mixed with a solution of Exceval. The mixture is then deposited onto quartz substrates by spin coating. The solutions of the targets in anisole and THF have a concentration of \( 17 \times 10^{-3} \text{ m} \) and are mixed into an 80 mg mL\(^{-1}\) host solution. The study of the absorption maxima of the respective targets in Exceval (Figure 2b) shows that BDPB, BDPB-Ac, and BDPDPPT have nearly the same maximum at around 265 nm. The introduction of OH-groups induces a shift of 5 nm for BDPB-OH. The large absorption bands show the possibility of excitation at 275 and 300 nm. The investigation of the emission properties shows the biluminescent behavior of our targets, i.e., the simultaneous emission of fluorescence and phosphorescence. Since PMMA itself emits by excitation at 275 nm\(^{[31]}\) (Figure 2a and Figure S12, Supporting Information), data evaluation for PMMA

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**Figure 1.** Synthesis of aromatic phosphonates BDPB, BDPB-Ac, BDPB-OH, and BDPDPPT. a) PO\(\text{Et}_3\), reflux, 2 h, 95%, b) KCN, H\(_2\)O, ethanol, reflux, 12 h, 37%, c) H\(_2\)SO\(_4\), ethanol, reflux, 12 h, 35%, d) NBS, AIBN, benzene, reflux, 60%, e) PO\(\text{Et}_3\), reflux, 2 h, 95%, f) HPO\(\text{Et}_2\), KF, Al\(_2\)O\(_3\), r.t., 12 h, 43%, g) SiCl\(_4\), EtOH, –20 °C to reflux, 50%, h) NBS, AIBN, benzene, reflux, 79%, and i) PO\(\text{Et}_3\), reflux, 2 h, 97%.
samples will only be performed with an excitation wavelength $\lambda_{\text{exc}} = 300$ nm. The position of the fluorescence and phosphorescence peaks of the four targets are nearly unaffected by excitation wavelength and matrix (Table 1). Since the fluorescence is completely in the UV range, it is barely visible and, in case of Exceval samples, the phosphorescence is the only emission visible for the human observer. The in-depth study of the phosphorescence shows that all targets display a spectrum characterized by three well-resolved vibronic sub-bands. The structure of the phosphorescence band represents the vibrational structure of the molecular ground state. The separations between the vibronic features of 32 nm (1469 cm$^{-1}$) and 25 nm (1019 cm$^{-1}$) therefore match very well with peaks in the infrared spectra of the emitter (Figure S13, Supporting Information). The most prominent band is centered around 485 nm. The substituents at the biphenyl core strongly influence the phosphorescence lifetimes. In a PMMA matrix, excited at 300 nm, BDPB-OH has the shortest lifetime (750 ms) followed by BDPB-Ac (1740 ms), BDPB (1880 ms), and BDPDPPT (2150 ms). The phosphonate groups make the molecules more hydrophilic, though not water-soluble under ambient conditions. This offers the opportunity of building strong hydrogen bonds with the surrounding matrix. Mixed into Exceval, the phosphonate targets have therefore phosphorescence lifetimes that are up to 400 ms longer compared to PMMA as matrix material. Excitation of the Exceval samples at 275 nm further increases the lifetime (see Table 1). Additionally, the emitters have been embedded in polystyrene (PS). The fluorescence and phosphorescence peak positions remain the same compared to PMMA but the phosphorescence lifetimes drastically increase to less than a quarter of the values (Table 1 and Figure S26, Supporting Information). The reason for these different lifetimes is revealed by examining the chemical structure of the host molecules. Whereas PMMA and Exceval exhibit OH-groups—PMMA by tautomerization of the methyl ester groups, Exceval by the nature of its structure—PS does not. The ability to form hydrogen bonds is therefore not given for PS but for PMMA and even more for Exceval.

Figure 2. Thin-film emission data showing BDPDPPT (green), BDPB-Ac (yellow), BDPB-OH (blue), and BDPB (red). a) Absorption spectra of pure PMMA and Exceval, b) absorption spectra of targets embedded in Exceval, c) phosphorescence decay in PMMA, nitrogen atmosphere, $\lambda_{\text{exc}} = 300$ nm, d) emission spectra under aerated (light color) and nitrogen atmosphere (dark color), e) delayed spectra showing only the phosphorescence, Exceval, aerated atmosphere, $\lambda_{\text{exc}} = 300$ nm, and f) corresponding phosphorescence decays.
Not only the amorphous films but also the crystals themselves show URTP. To assure high emission intensity, pure crystals of BDPB-OH, BDPB-Ac, and BDPB have been immersed into an Exceval solution, drop-casted onto quartz substrates, and measured under aerated atmosphere after drying. The phosphorescence lifetimes (Table 1) are higher than in the PMMA:guest system but do not reach the ones in the amorphous Exceval:guest films. Nevertheless, embedded in Exceval, BDPB-Ac and BDPB crystals show impressively long lifetimes of 2020 and 2150 ms, respectively.

Going from two to three phosphonate groups, the phosphorescence lifetime in PMMA rises from already very long 1880 ms (BDPB) to 2150 ms (BDPDPPt). In Exceval, it is further increased to an impressively long lifetime of 2570 ms, up to 2620 ms when excited at 275 nm. First experiments suggest that a further increase in the number of phosphonate groups does not increase the lifetime any more (see the Supporting Information, cf. sixfold aromatic phosphonate). Figure 3 shows photographs of the different molecules embedded in Exceval. After illumination at 275 nm, the light-emitting diode was turned off and the pictures were taken immediately. In the group of the twofold phosphate derivatives, the triplet lifetimes correspond with the phosphorescence intensity. BDPB-OH, which has the shortest lifetime, also exhibits the lowest intensity, whereas BDPB shows the highest intensity and the longest lifetime. Interestingly, BDPDPPt, which has the longest lifetime of all four targets, has a comparably low intensity as BDPB-OH.

Using PMMA as host and Exceval as an oxygen barrier, these samples can be used for phosphorescent information storage in PLTs, e.g. in the packaging industry or for anticounterfeiting products. In fact, since these phosphors are very wide bandgap, the phosphorescence and PLT spectral range can only be excited with rather hard UV, which is unlike from conventional UV sources used in daily life, e.g., for bill validation that is usually done with black light. Thus, this may allow for more secure use of these materials. The mechanism based on the UV light-dependent oxygen consumption has already been described in a previous report. Since commonly used printed masks cannot be applied below 300 nm, as the transparency blocks the light, a laser-cut mask made of medium-density fiberboard has been used here.

Looking at the photoluminescence quantum yield (PLQY, number of photons emitted as fraction of the number of photons absorbed) values (Table 1 and Figure S14, Supporting Information) it is noticeable that BDPB-OH has the highest value in PMMA as well as in Exceval. In PMMA, there is a trend visible. The higher the PLQY, the shorter the lifetime. This trend is less visible in Exceval samples and might therefore be a coincidence, although the BDPB-OH has as well the highest PLQY value and BDPDPPt the lowest one. However, the PLQY of the phosphorescence is not only dependent on the phosphorescence lifetime but also on the intersystem crossing rate. Due to the different morphologies of the hosts PMMA and Exceval, ISC could be influenced as well. In case of BDPB and BDPB-OH, the PLQY seems to be independent of the used host material, whereas BDPB-Ac and BDPDPPt have lower values in Exceval. Possible intermolecular interplays involved here have not been investigated yet.

To summarize, we have analyzed and compared the ultralong room-temperature phosphorescence properties of four analogous compounds dispersed into a matrix of PMMA, Exceval, and PS, respectively. The derivatives containing diethyl-phosphonomethyl groups differ on the one hand in the nature of their substituents and on the other hand in the number of these groups. We have shown that these structural differences play a critical role in the phosphorescence lifetime. Besides, the surrounding matrix also strongly influences these lifetimes. Building hydrogen bonds with the matrix increases the phosphorescence lifetime up to 2.62 s. The emission color is blue with Commission Internationale de l’Eclairage coordinates of $x = 0.21$ and $y = 0.35$. The substituents of the molecules do not play a big role concerning fluorescence and phosphorescence spectrum properties, which suggests that the main reason for the high lifetimes is the presence of diethyl-phosphonomethyl units. Going from two to three groups increases the lifetimes by around 250 ms. It was revealed that BDPB and BDPDPPt possess the most promising URTP features when embedded in host matrices containing hydroxyl groups—which have the ability to form hydrogen bonds with the emitter molecules, leading to an increase of the phosphorescence lifetime—such as PMMA and Exceval.

**Supporting Information**

Supporting Information is available from the Wiley Online Library or from the author.

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Conflict of Interest

The authors declare no conflict of interest.

Keywords

aromatic phosphonates, biluminescence, organic programmable tags, ultralong room temperature phosphorescence, UV excitation

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Figure 3. Photographs of the different targets in Exceval at ambient conditions directly after excitation at 275 nm. a) BDPB-OH, b) BDPB-Ac, c) BDPB, d) BDPDPPT, and e) delayed phosphorescent image written by masked UV illumination in a PMMA:BDPB sample covered with Exceval.

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