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Mono-functionalized dibenzo[g,p]-chrysenes for cascade energy transfer

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ABSTRACT

3-Bromodibenzo[g,p]chrysene has been synthesized in a one-pot reaction using phenyliodine(III) bis(trifluoroacetate) (PIFA) as an oxidant and a Lewis acid. The bromo function was easily converted to the cyano, alkyne, and formyl derivatives. Bodipy dyes have either been constructed from the chrysene–aldehyde or by cross coupling reactions with the chrysene ethynyl derivatives. The linking of diketopyrrolopyrrole (DPP) fragments was realized using the chrysene–ethynyl compound or with the in situ prepared chrysene–borolane derivatives. The novel chrysene dyes are highly fluorescent in apolar solvents and were used as input energy in intramolecular energy transfer processes with covalently linked DPP or Bodipy dyes. All mixed dyes are redox active and each module is clearly identified by its redox behavior. Little electronic interaction is evidenced whatever the nature of the dyes and spacer.

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Polycyclic aromatic hydrocarbons (PAHs) are molecules of considerable importance for emerging technologies concerning molecular electronics, optoelectronics, energy conversion devices, and various other forms of nanotechnology.^{1–3} Commonly encountered examples include triphenylenes composed of four fused benzene rings,⁴ corannulenes with five fused benzene rings,⁵ coronenes made from seven benzene rings,⁶ and hexa-*peri*-benzocoronenes (HBC) consisting of thirteen fused six membered rings.⁷ Derivatives of HBC with long alkyl chains self-organize in discrete columnar mesophases which have been used in organic solar cells.⁸ HBC derivatives with various patterns of substituents have proved valuable for the synthesis of large graphene-like sheets of particular symmetries,⁹ although in most cases the construction of these edifices required several synthetic steps involving multiple C–C bond formation.

Of other PAHs, dibenzophenanthrenes and dibenzo[g,p]chrysenes, with, respectively, five and six benzene rings, are of particular interest because several examples are known where a functional group suited for postfunctionalization is present.¹⁰ Their optical properties (e.g., high fluorescence in solution, well defined and sharp optical transition in the near-UV)⁹ are similar to those of units such as fluorene, pyrene, and perylene which we have used previously¹¹ as input-energy centers in multi-chromophoric dyes to promote internal energy transfer, to concentrate photons, and to obtain large virtual Stokes shifts.¹² Such small, planar PAH groups are disadvantageous in that they induce low solubility in polar solvents, while twisted PAH groups such as dibenzochrysenes provide improved solubility but are usually rather difficult to functionalize. While it has been recently found that unsubstituted or tetrasubstituted chrysene molecules can be prepared from tetra-arylethylenes using DDQ as an oxidant,¹⁰ introduction of a bromo substituent favors the formation of phenanthrene derivatives at the expense of chrysene because of the high oxidation potentials of the bromo-tetraarylethylene substrates. A variety of oxidants (FeCl₃, CuCl₂/AlCl₃, MoCl₅, SbCl₅, hv/l₂···) can be used in place of DDQ to catalyze aryl-aryl coupling reactions.¹³

We previously succeeded in using phenyliodine(III) bis(trifluoroacetate) (PIFA) in conjunction with a Lewis acid to promote C– C coupling of Bodipy monomers.^{14,15} PIFA has also been used to prepare meso–meso–linked linear arrays of porphyrins,¹⁶ and fused diporphyrin scaffoldings,¹⁷ in the presence of a Lewis acid or a fluoroalcohol as solvent.¹⁸ Thus, we were attracted to the use of PIFA as an oxidant for substituted tetraarylethylene derivatives and describe herein its novel application in the preparation of 3-bromodibenzo[*g*,*p*]chrysene in a one pot reaction. This compound was of interest for the preparation of a series of derivatives suitable for the introduction of various complex substituents leading to possible applications.

When compound **1**, prepared as described in the literature,¹⁹ was treated with PIFA in the presence of the Lewis acid BF₃.OEt₂, the target mono-bromochrysene **3**, characterized by its mass spectrum [EI-MS m/z 406.0 (100) and 408.0 (95)], was isolated in 21% yield (Scheme 1). Under these conditions, a more polar dimeric phenanthrene compound **4** was also isolated in 11% yield and characterized by its mass spectrum [EI-MS m/z 816.0 (100) and 817.0 (60)]. Various isomeric forms other than that shown are possible for this dimer. Interestingly, such dimeric phenanthrene com-





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Scheme 1. Reagents and conditions: (i) (a) Diphenylmethane (1.2 equiv), *n*-BuLi (1.1 equiv), THF, $0 \degree C$ for 1 h; (b) 4-Bromobenzophenone (1 equiv), THF, $-78 \degree C$ for 3 h, then rt for 15 h; (c) *p*-TsOH (0.2 equiv), toluene, reflux, Dean–Stark apparatus, 3 h; (ii) BF₃·OEt₂ (20 equiv), PIFA (2.6 equiv), compd **1** (1 equiv), CH₂Cl₂, $0 \degree C$ or (iii) MsOH (large excess), PIFA (2.8 equiv), compd **1** (1 equiv), CH₂Cl₂, $0 \degree C$, 33% of 3.

pounds have also been isolated during oxidative cyclization of bis(biaryl)acetylene derivatives.²⁰ Switching from BF₃.Et₂O to large excess of methanesulfonic acid increased the formation of **3** to 33% without formation of dimer **4**.

Formation of the phenanthrene derivative 2^{10} during the course of the reaction was observed by TLC. Molecular structures were assigned by means of NMR spectroscopy (2D COSY and 2D NOESY in particular) and use of compounds **1** and **2** as reference. The spectrum of **1** shows a typical AB system for the protons of the 4-bromo substituted phenyl ($_{3}J = 8.4$ Hz), with slight shifts ($\delta = 6.88$ and 7.23 ppm) from the other aromatic proton peaks ($\delta = 7.36$ ppm). The regioselectivity (formation of a six- rather than a five-membered ring) of the oxidation leading to **2** is consistent with other observations.¹⁰ The peaks in the spectrum of **2** are shifted downfield compared to those of **1**, the bromophenyl AB system, for example, appearing at $\delta = 7.05$ and 7.39 ppm. After the two successive oxidations leading to **3**, all peaks are shifted to significantly



lower field positions (Fig. 1). The ¹H spectrum shows a diagnostic doublet (δ = 8.80 ppm, 1H, ₄*J* = 2.0 Hz) and two overlapping multiplet signals (δ = 7.58–7.71 ppm, 7H and δ = 8.48–8.69 ppm, 7H) indicative of two broadly distinct environments. Oxidation leading to five-membered rings was not observed. 2D COSY and 2D NOESY experiments confirmed the molecular structures and provided assignments of all protons (see ESI).

In considering the modification of the chrysene core to promote functionalization such as linking the module to other chromophoric units, for example borondipyrromethene (Bodipy) or diketopyrrolopyrrole (DPP), or to induce properties such as triplet formation, it may be noted that chrysene substitution has not been utilized much due to the lack of general synthetic procedures. The objectives here were therefore to develop a battery of methods suitable for introducing various types of functionalities (alkyne, cyano, formyl···).

To this end, we first explored the synthesis of derivatives **5** and **6** using well established protocols.²¹ Cross-coupling with TMSacetylene followed by deprotection under basic conditions provided the terminal alkyne **6** in 72% yield in two steps. A cyanation reaction,²² catalyzed by Pd, NaCN, and Zn enabled replacement of the bromo group in **3** by a cyano function in **7** (72% yield). Furthermore, metallation of **3** at low temperature followed by formylation with methylformate afforded the formyl derivative **8** in 60% yield (Scheme 2). A strategy based on a carboformylation reaction²³ was even more successful, reaching isolated yields of 76%.

Our next target became the construction of a Bodipy dye around the chrysene core. The Bodipy dye **9** was prepared in three steps by reaction of the chrysene carbaldehyde **8** with 2,4-dimethylpyrrole in the presence of TFA to provide the dipyrromethane intermediate, followed by DDQ oxidation to the dipyrromethene and boron complexation under basic conditions. The highly fluorescent orange-red dye was isolated in 32% yield (Scheme 3). Another pathway examined was to cross-couple the terminal alkyne **6** with a preformed Bodipy **A** (Chart 1) using Pd catalysis. This provided a quantitative yield of the dyad linking the previously unknown chrysene derivative to a well-known Bodipy dye.

Diketopyrrolopyrrole dyes are of particular interest in that their absorption properties can be tuned over a large range and they dis-



Scheme 2. Reagents and conditions: (i) $[Pd(PPh_3)_4]$ (6 mol %), **3** (1 equiv), trimethylsilylacetylene (1.9 equiv), toluene, Et₃N, 80 °C, 15 h; (ii) K₂CO₃ (8 equiv), THF/ MeOH, 20 °C, 15 h; (iii) freshly prepared catalyst,²² **3** (1 equiv), NaCN (1.1 equiv), Zn (0.5 equiv), THF, 70 °C, 2 days; (iv) for the metallation: (a) **3** (1 equiv), *n*-BuLi (1.04 equiv), THF, -60 °C, 45 min, (b) methylformate (excess), -60 °C then rt, 3 h; for the carboformylation: $[Pd(PPh_3)_2Cl_2]$ (9 mol %), **3** (1 equiv), sodium formate (6 equiv), CO flow, DMF, 100 °C, 16 h.



Scheme 3. Reagents and conditions: (i) 2,4-dimethylpyrrole (3.1 equiv), **8** (1 equiv), TFA (1.1 equiv), CH₂Cl₂, 30 °C, 14 days; (ii) DDQ (1 equiv), 30 °C, 5 min; (iii) Et₃N (6 equiv), BF₃·OEt₂ (8 equiv), rt, 1 h; (iv) **A** (0.9 equiv) and **G** (1 equiv), [Pd(PPh₃)₄] (5 mol %), toluene, Et₃N, 65 °C, 15 h.



play outstanding charge carrier properties which have been exploited in the engineering of solar cells.²⁴ Cross linking the chrysene alkyne 6 to the mono-bromothiophene–DPP is easily realized under experimental conditions used for the Bodipy dyad described above. The chrysene–DPP dyad **11** was obtained in 69% yield after standard purification procedures. It has previously been established that the nature of the spacer linking different modules has a strong influence on the spectroscopic, electrochemical, and morphological properties of hybrid systems.²⁵ We tried to link the chrysene core to a DPP module directly by a C-C bond (compound 12 in Scheme 4). The chrysene–borolane derivative appears to be unstable and is thus very difficult to prepare, isolate, and purify. After some experimentation, we succeeded in preparing the mixed dye **12** by cross-coupling the in situ prepared borolane to the precursor **B** under standard Suzuki–Miyaura cross coupling reaction. The poor isolated yield is mostly due to the degradation of the borolane during the heating procedure. As well, the in situ formation of the trifluoroborate potassium salt²⁶ limits the efficiency of the coupling reaction.

The electronic properties of these chrysene derivatives were examined by UV absorption in deaerated cyclohexane solutions (Fig. 2). The absorption spectra exhibit large and structured bands between 220 and 320 nm with molar extinction coefficients in the



Scheme 4. Reagents and conditions: (i) **6** (1 equiv) and **B** (0.9 equiv), $[Pd(PPh_3)_4]$ (7 mol %), toluene, Et₃N, 80 °C, 15 h; (ii) (a) **2** (1 equiv), *n*-BuLi (1.1 equiv), THF, -78 °C, 5 min; (b) 2-isopropoxy-4,4,5,5-tetramethyl-1,3,2-dioxaborolane (1.2 equiv) in THF, -78 °C for 5 min then rt for 30 min; (iii) **B** (0.9 equiv), K₂CO₃ (2.5 equiv), H₂O (one drop), Pd(PPh_3)₄ (12 mol %), 80 °C, 5 days.

60,000–70,000 M⁻¹ cm⁻¹ range. In all cases, a weaker absorption peak at 360 nm ($\varepsilon \approx 19000 \text{ M}^{-1} \text{ cm}^{-1}$) is evident. All these derivatives have similar energy gaps and their absorption spectra are in keeping with those of chrysene derivatives previously described.²⁷

Derivatives **5–8** are fluorescent in cyclohexane solution. A maximum of 18% quantum yield was determined for the cyano-chrysene derivative **7** under anaerobic conditions. In the case of the aldehyde derivative **8** the fluorescence is markedly weaker due to the presence of $n \rightarrow \pi *$ transition deactivating the excited state by non-radiative processes.²⁸ In most cases, the emission profile mirrors the transition lying at lower energy, which is clear evidence for a localized singlet excited state. The Stokes shifts are relatively modest, lying in the 2600 to 3200 cm⁻¹ range, but in all cases the excitation spectrum matches the absorption spectrum, excluding formation of aggregates at these solution concentrations.

The hybrid dyes bearing a Bodipy residue linked to chrysene either directly (**9**), or through a tolane spacer (**10**) exhibit absorption spectra which are almost linear combinations of the absorption bands of the individual modules (Fig. 3a and b). This indicates weak electronic interaction between both modules, a situation expected in light of the presence of the methyl groups on the Bodipy core which force the aromatic ring to be almost orthogonal to it. This arrangement has been found in several X-ray structures.²⁹ The low energy transition at 500 nm corresponds to the $S_0 \rightarrow S_1$ transition of the Bodipy fragment, while its $S_0 \rightarrow S_2$ transition is localized beneath the low energy absorption of the chrysene moiety. This is a situation extremely favorable for energy transfer, as previously established with pyrene hybrids.¹¹ Irradiation at 300 nm (**9**) and 305 nm (**10**) in the chrysene absorption band did



Figure 2. Absorption, emission, and excitation spectra for **3**, **5**, **7**, and **8** in degassed cyclohexane at 25 °C. (a) **3**: Emission $\lambda_{ex} = 352 \text{ nm}$ ($D_0 = 0.2852$). (b) **5**: Emission $\lambda_{ex} = 273 \text{ nm}$ ($D_0 = 0.1546$); excitation $\lambda_{em} = 401 \text{ nm}$. (c) **7**: Emission $\lambda_{ex} = 292 \text{ nm}$ ($D_0 = 0.5954$); excitation $\lambda_{em} = 442 \text{ nm}$. (d) **8**: Emission $\lambda_{ex} = 269 \text{ nm}$ ($D_0 = 0.3043$); excitation $\lambda_{em} = 472 \text{ nm}$.



Figure 3. Absorption, emission, and excitation spectra of dyes **9** (a) and **10** (b) in degassed cyclohexane at 25 °C. (a) **9**: Emission $\lambda_{ex} = 300 \text{ nm}$ ($D_0 = 0.2656$); excitation $\lambda_{em} = 550 \text{ nm}$. (b) **10**: Emission $\lambda_{ex} = 352 \text{ nm}$ ($D_0 = 0.1000$); excitation $\lambda_{em} = 553 \text{ nm}$.



Figure 4. Absorption, emission, and excitation spectra of dyes **11** (a) and **12** (b) in degassed cyclohexane at 25 °C. (a) **11**: Emission $\lambda_{ex} = 310 \text{ nm}$ ($D_0 = 0.3503$); excitation $\lambda_{em} = 695 \text{ nm}$. (b) **12**: Emission $\lambda_{ex} = 312 \text{ nm}$ ($D_0 = 0.3428$); excitation $\lambda_{em} = 613 \text{ nm}$.



Figure 5. Cyclic voltammograms of: (a) reference compound **A** (red trace) and chrysene–Bodipy **10** (black trace), (b) **B** (purple trace), and DPP–Chrysene **11** (black trace). Concentration of dyes 1.5×10^{-3} M in CH₂Cl₂ (0.1 M, *n*-Bu₄NPF₆), Fc⁺/Fc refers to the ferricinium/ferrocene couple used as internal reference $E_{1/2}$ (Fc⁺/Fc) = +0.38 V ($\Delta E_p = 60$ mV).

not produce any emission of the chrysene at 402/414 nm but exclusively emission of the Bodipy module [$\lambda_{em} = 515$ nm, $\phi_{em} = 0.68$, $\tau = 3.4$ ns for **9** and $\lambda_{em} = 514$ nm, $\phi_{em} = 0.56$, $\tau = 3.0$ ns for **10**] (Fig. 3). This is clear evidence that very efficient energy transfer from the chrysene to the Bodipy is occurring. An ultimate confirmation was obtained by running an excitation spectrum ($\lambda_{em} = 553$ nm); excellent agreement with the absorption spectrum is in keeping with such a situation.

For mixed DPP-chrysene dyes 11 and 12 the presence of the five membered thiophene rings and the bis-lactam configuration of the fused rings decrease the steric congestion around the grafted subunit, allowing some electronic interactions between the modules. This is highlighted in the absorption spectra (Fig. 4) where the absorption band of the chrysene is broadened by about 30 to 40 nm. The band of the DPP residue is also wider by about 25 nm compared to the unsubstituted starting material (reference measured in THF). Selective irradiation in the chrysene module around 310 nm does provide emission of the DPP module but also some residual emission of the chrysene at 417/444 nm $[\lambda_{\rm em} = 610 \text{ nm}, \phi_{\rm em} = 0.37, \tau = 6.1 \text{ ns} \text{ for } \mathbf{11} \text{ and } \lambda_{\rm em} = 615 \text{ nm},$ $\phi_{\rm em}$ = 0.48, τ = 7.5 ns for **12**] (Fig. 4). This weak residual emission confirms that in this case there is less efficient spectral overlap between the chrysene fragment and the DPP dye. Excitation spectra for the emission at 695 and 613 nm match well with the absorption spectra, confirming the energy transfer process and the participation of all modules chrysene/DPP in the emission process.

The redox behavior of the new dyes was investigated by cyclic voltammetry in CH₂Cl₂ solution containing 0.1 M tetra-N-butylam-

monium hexafluorophosphate (TBAPF₆) as supporting electrolyte and the results are shown in Fig. 5a and b. They confirm that each fragment in dyads 10 and 11 retains the redox activity of the separate modules.

Dyad **10** exhibits redox waves at +1.07 V ($\Delta E_p = 60 \text{ mV}$) and -1.34 V ($\Delta E_p = 60$ mV), similar to those of compound **A** (Fig. 5a). The additional reversible oxidation at +1.29 V ($\Delta E_p = 60 \text{ mV}$) is assigned to the radical cation of the chrysene residue. This potential is similar to that for the oxidation of compound 5 [+1.30 V $(\Delta E_{\rm p} = 60 \text{ mV}]$. Likewise, dyad **11** exhibited three reversible oxidation waves at +0.89 V ($\Delta E_p = 60 \text{ mV}$), +1.21 V ($\Delta E_p = 60 \text{ mV}$) and +1.44 V (ΔE_p = 60 mV), one reversible reduction at -1.15 V $(\Delta E_{\rm p} = 70 \text{ mV})$ and an irreversible additional reduction at -1.57 V(Fig. 5b). By comparison to compound **B** [+0.92 V ($\Delta E_p = 60 \text{ mV}$), +1.27 V (ΔE_p = 60 mV), -1.21 V (ΔE_p = 70 mV)] the first two oxidation waves in **11** are safely assigned to the DPP residues and the third to the chrysene moiety. The irreversible reduction at -1.57 V is probably due to the reduction of the chrysene fragment a potential close to the irreversible reduction of 5. Note that the other redox processes appear highly reversible $(i_{
m pa}/i_{
m pc}pprox 1)$ and exhibit the characteristic shape ($\Delta E_p = 60-70 \text{ mV}$) of a Nernstian, one-electron process (Fig. 5a and b).

In summary, a convergent method has been successfully developed for the preparation of 3-bromochrysene by treatment of a substituted tetraphenylethylene with hypervalent iodine derivative (PIFA). This new soluble molecule was efficiently converted to alkyne, cyano, formyl, and borolane derivatives which may be useful for the construction of sophisticated platforms. The linkage of fluorescent Bodipy or DPP modules provided evidence of very efficient intramolecular cascade energy transfer. Both spectroscopic and electrochemical measurements showed little electronic interaction in the dyads whatever the nature of the dyes and spacer. Ongoing work in our laboratory is now focused on selectively linking such chrysene modules to phosphorescent transition metal modules suitable for long lived triplet emission states.

Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.tetlet.2013. 04.073.

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