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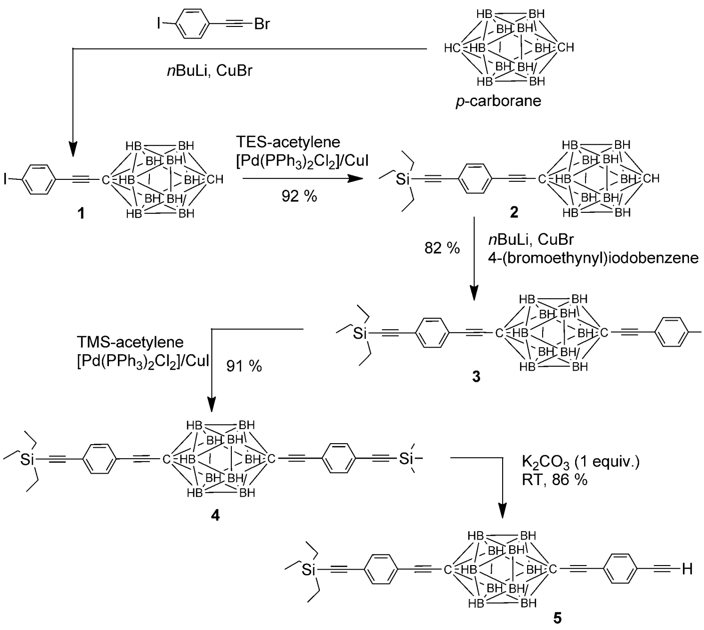
# Unsymmetrical *p*-Carborane Backbone as a Linker for Donor–Acceptor Dyads

**Delphine Hablot,[a] Alexandra Sutter,[a] Pascal Retailleau,[b] and Raymond Ziessel*[a]**

*Dedicated to Dr. Hubert le Bozec on the occasion of his 60th birthday*

The chemistry of polyhedral boron hydrides has been widely investigated[1] and *closo*-boranes in particular are known to show high kinetic stability favouring various applications. Such applications range from those of BNCT (boron neutron-capture therapy) in medicine[2] to those in the synthesis of superacids.[3] Partial substitution of boron by carbon gives rise to the related carboranes, which, in functionalised forms, are important building blocks for the engineering of soft matter,[4] polymers,[5] nonlinear materials,[6] rigid rods[7] and self-assembled molecular structures.[8] Carboranes of the *closo* form, such as 1,12-dicarbadecaborane ($C_2B_{10}H_{12}$ in Scheme 1) are geometrically rigid species with an electronic structure making them optically transparent and redox inactive.[1,9] However, most methods currently available for the synthesis of derivatives of this carborane provide only symmetically disubstituted species, typically those bearing either two alkyne functions,[10] two pyridines,[8] two phosphanes[8] or two 4-iodophenyl[11,12] residues. In the last case, metallation of the *para*-carborane and transformation to its cuprate at low temperature, and subsequent reaction with 4-(bromoethynyl)iodobenzene provides a homo disubstituted derivative with the functional groups at the vertices of the carborane cluster.[10,12] Although conversion of such a species to an unsymmetrical derivative is possible, this requires statistical cross-coupling reactions that limit the isolated yields, produce disubstituted derivatives that are useless and consume the precious starting material.[12] Note that monosubstituted carboranes are available but hetero-disubstituted carboranes are elusive and have not been obtained in a controlled process mostly because of the reactivity of the first functional group towards nucleophiles.[13] Thus, the present challenge was seen as that of functionalizing *closo*-1,12-dicarbadecaborane **1** with two orthogonal functions in a procedure of minimum length.



Scheme 1. Synthesis of the unsymmetrically functionalized *p*-carborane.

To this end, we first explored the synthesis of **3** by using *closo*-1,12-(4-ethynyliodophenyl)dicarbadecaborane **1** as the starting material. Compound **3** was prepared by a three-step procedure as outlined in Scheme 1. The choice of triethylsilylacetylene (TES-acetylene) was based on its inertness towards *n*BuLi or cuprates.[14] Indeed the pivotal compound **3** was obtained in 82 % yield by formation of the cuprate at the unsubstituted carborane C atom followed by reaction with 4-(bromoethynyl)iodobenzene. Subsequent transformation to the terminal alkyne required cross-coupling with trimethylsilylacetylene (TMS-acetylene) and selective removal of the TMS protecting group, leading to **5** in excellent yield (Scheme 1). Alternative routes using acetylene gas[15] or propargyl alcohol do not give the pivotal derivative **5** in acceptable yields.

Crystal-structure determinations were performed for **1** and **3** (Figures 1 and 2). Both molecules have an essentially cylindrical shape and carborane **3** is approximately twice as long (at ca. 24 Å) as carborane **1**. The carborane units in

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[a] D. Hablot, A. Sutter, Dr. R. Ziessel
Laboratoire de Chimie Moléculaire et Spectroscopies Avancées (LCOSA)
Ecole Européenne de Chimie, Polymères et Matériaux
CNRS, 25 rue Becquerel, 67087 Strasbourg Cedex 02 (France)
E-mail: ziessel@unistra.fr

[b] Dr. P. Retailleau
Laboratoire de Cristallochimie
ICSN—CNRS, Bât 27-1 avenue de la Terrasse
91198 Gif-sur-Yvette, Cedex (France)

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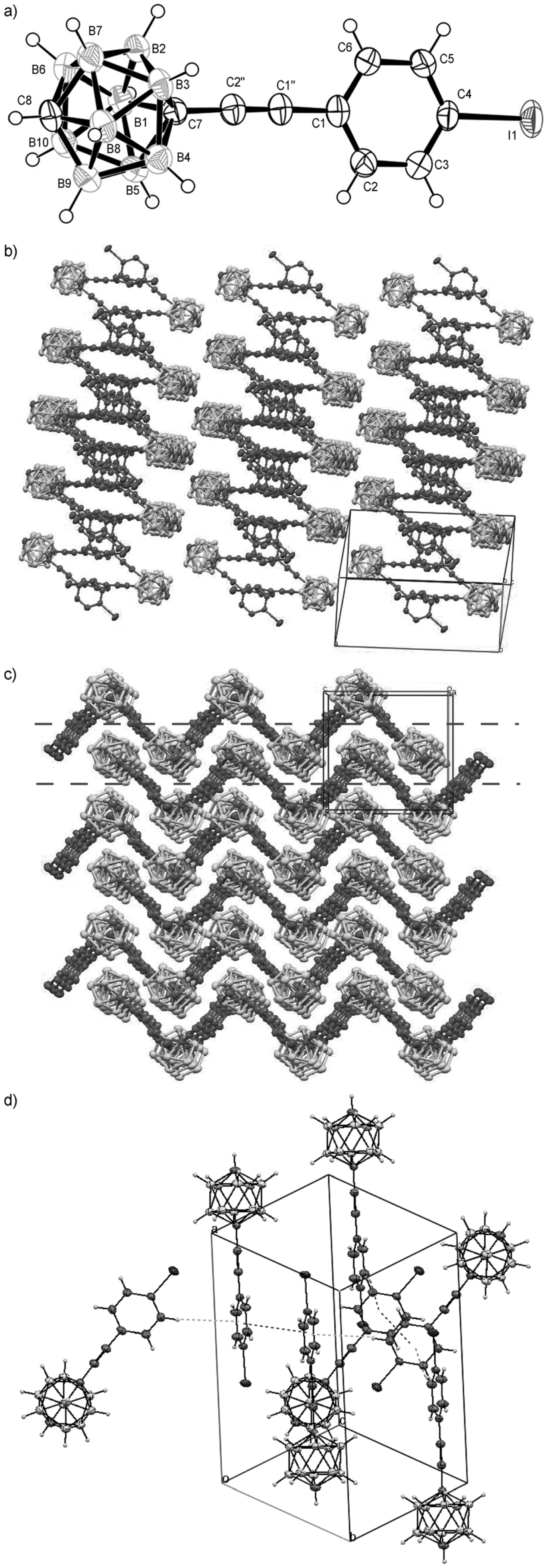


Figure 1. a) ORTEP view of carborane **1**. Displacement ellipsoids are drawn at the 50% probability level. b) Projection of the layered structure of the carborane **1** in the (01$\bar{1}$) plane. c) Lattice structure down the *a* axis. d) Close-up of intermolecular interactions (dotted lines), viewed down the [0$\bar{1}$1] direction.

both are almost identical; superposition of one on the other results in an overall r.m.s. deviation of 0.12 Å. Extension of **1** through two additional ethynyl linkers slightly increases the overall deviation from linearity by about 10° (supplementary angle of I1-C11-Si1). The two phenyl rings of **3** are nearly coplanar (dihedral angle 18.4°), with their mean plane orthogonal to the *bc* plane, which contains the long molecular axis.

Both compounds crystallize in the monoclinic space group $P2_1/c$. The lattice of **1** is layered and consists of carborane bilayers parallel to the *bc* plane (Figure 1b). The nuclei are not situated on the glide mirrors and the iodophenylethynyl groups are arranged between the bilayers in a herring-bone fashion viewed down the *a* axis, the phenyl rings being in general positions, with a dihedral angle of 71.3° between those at x, $^1/_2$-y, z- $^1/_2$ (Figure 1c). Along the [011] and [0$\bar{1}$1] directions, the inversion-related rings stack parallel with an interplanar distance of 3.407(2) Å but with an offset of 2.171 Å and are further sandwiched by C–H···aryl (centroid) interactions ($d = 3.616$ Å and $\measuredangle = 172.6°$; Figure 1d).

For compound **3**, the molecules are also layered in the *bc* plane in a herring-bone fashion, the triethylsilane group lying over the iodophenyl of a neighbouring molecule with a 70° angle of attack (Figure 2b). In this crystal structure, the carborane nuclei are isolated from each other by surrounding triethylsilane groups and phenyl groups.

As photoactive modules to be attached, we chose a borondipyrromethene (Bodipy; **A** and/or **B**) for its $\lambda_{abs}/\lambda_{em}$ at 645/659 nm,[16] a subphthalocyanine (SubPc; **C**) for its $\lambda_{abs}/\lambda_{em}$ at 565/574 nm,[17] or a diketopyrrolopyrrole (DPP; **D**) for its $\lambda_{abs}/\lambda_{em}$ at 483/547 nm.[18] These choices were based on the potential to provoke energy transfer by a resonance mechanism[19] due to overlap of the emission and absorption properties of each module.

Our immediate target became the linking of the first fluorophore **B** to the carborane **3** under conventional Pd catalysis. The TES-protected dye **6a** was isolated in excellent yields and deprotection was straightforward in the presence of KF under polar conditions (Scheme 2). Cross-coupling of **6b** to a second fluorophore such as **C** or **D** afforded the mixed dyes **7** and **8** in excellent yields.

As expected from earlier work on photoactive molecular-scale wires, a key objective for such artificial systems is to determine the efficiency of unidirectional information transfer in the donor–acceptor system as a function of the length of the spacing unit.[20] Here, with a carborane spacer, the situation is highly interesting due to the inability to transfer information (in the form of an electron, a hole or a charge) through the cluster. Thus, an important aspect of the present work was our utilization of the chemistry developed to extend the length of the carborane spacer by formation of a

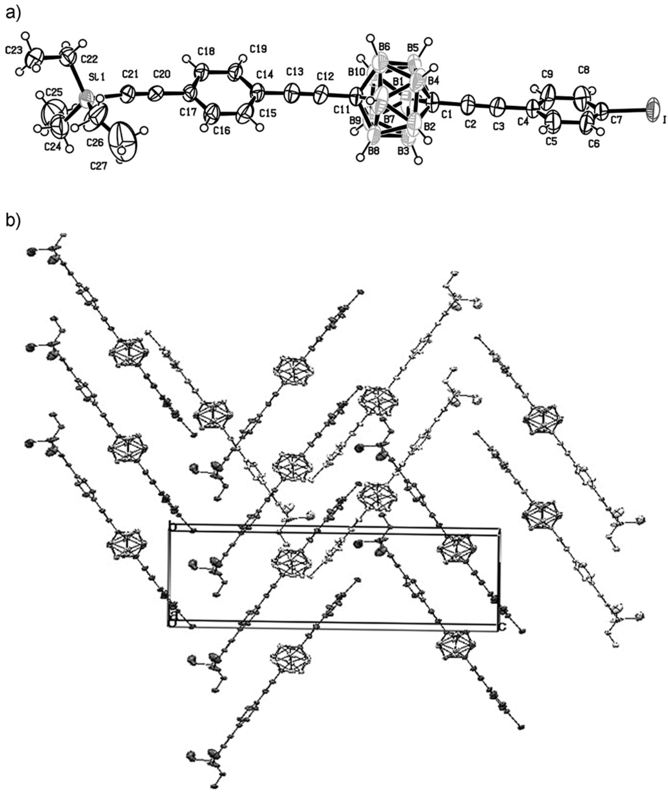
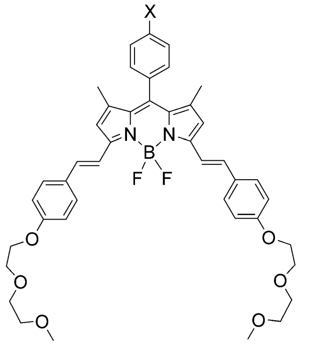
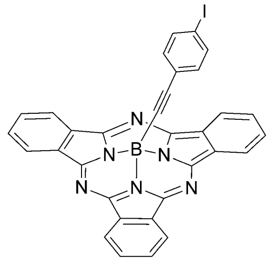


Figure 2. a) ORTEP view of carborane **3**. Displacement ellipsoids are drawn at the 50 % probability level. b) Projection of the lattice structure down the *a* axis.

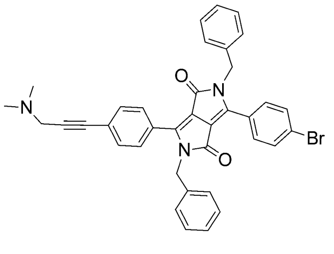


**A** (X = I)

**B'** (X = C≡CTMS)
**B** (X = C≡CH)



**C**



**D**

double carborane unit before attachment of the second fluorophore. This, we chose to do first by connecting the **C** to carborane **5**, leading to derivative **9a** in 86 % isolated yield (Scheme 3).

Subsequently, deprotection of the TES group in **9a** proved to be difficult and intense experimentation was required to reach a maximum yield of 50 %. The use of basic conditions (KOH or $K_2CO_3$) decomposes the SubPc chromophore even at room temperature. The optimal conditions eventually developed required a large excess of KF in methanol, THF, trace amounts of water and a short reaction time under mild heating (Table 1).

Table 1. Experimental conditions for the deprotection of the TES group of dye **9a**.[a]

| Solvent (ratio v/v)[b] | Base (equiv) | $t$ [h][c] | $T$ [°C][d] | Yield [%][e] |
|---|---|---|---|---|
| $CH_2Cl_2$/MeOH (1:1) | NaOH (2) | 0.5 | RT | decomp |
| THF/MeOH/$H_2O$ (3:1:$\varepsilon$) | $K_2CO_3$ (1) | 15 | RT | decomp |
| THF/MeOH/$H_2O$ (5:2:$\varepsilon$) | KF (40) | 21 | RT | 15 |
| THF/MeOH/$H_2O$ (10:3:$\varepsilon$) | KF (100) | 15 | RT | 30 |
| THF/MeOH/$H_2O$ (6:5:$\varepsilon$) | KF (400) | 6 | RT | 50 |
| THF/MeOH/$H_2O$ (6:5:$\varepsilon$) | KF (400) | 0.75 | 50 | 50 |
| THF/MeOH/$H_2O$ (1:1:$\varepsilon$) | KF (100) | 2 | 50 | 42 |
| THF/MeOH/$H_2O$ (5:4:$\varepsilon$) | KF (200) | 2 | 50 | 36 |
| $CH_2Cl_2$/MeOH (1:1) | KF (50) | 15 | RT | $<10$ |

[a] Concentration of **9a** about $4\times10^{-3}$ mol L$^{-1}$. [b] $\varepsilon$ = one drop of water. [c] Reaction time. [d] RT = room termperature. [e] decomp = decomposition.

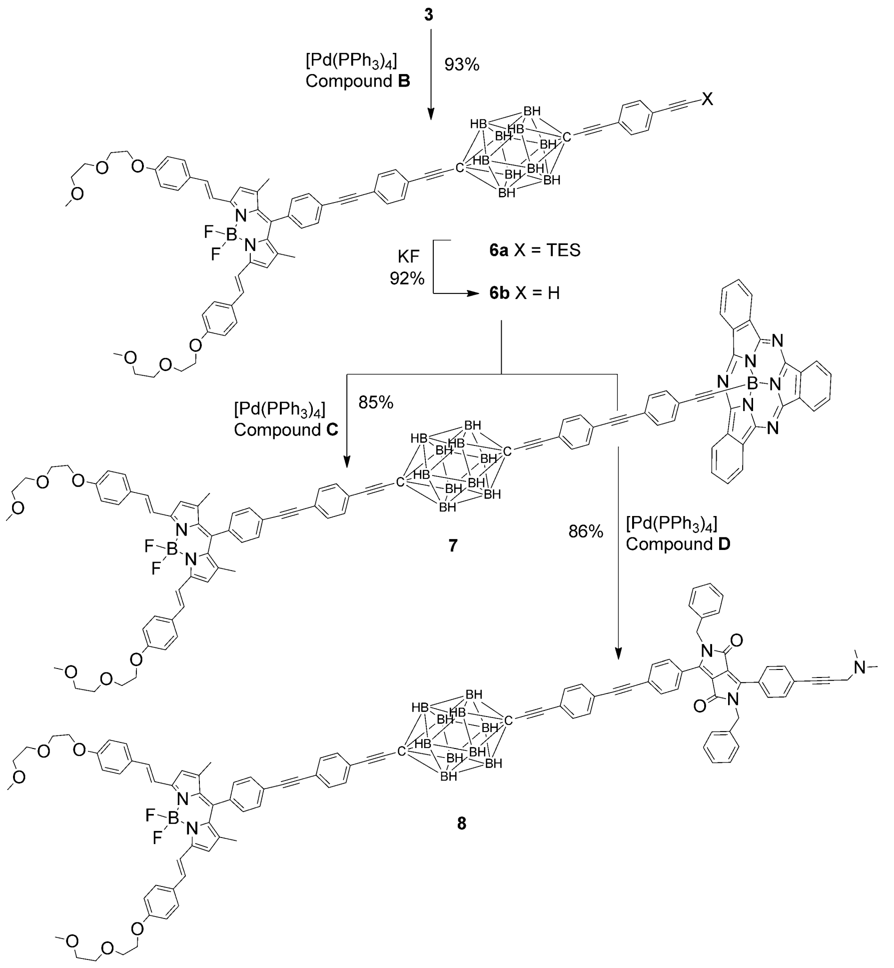
Note that switching from the SubPc to a Bodipy end-group generates a situation in which deprotection of the TES group is impossible due to the instability of the Bodipy in the presence of large quantities of KF. With compound **9b** in hand, it was easy to cross-link a second carborane residue using **3** and conventional Pd-catalyzed coupling to obtain compound **10a**. After deprotection with KF and cross-linking **10b** with **A**, the mixed dyad **11** was obtained in an acceptable overall yield (Scheme 3).

The photophysical properties of the model compounds and the carborane-substituted dyes are summarized in Table 2. The spectra of the building blocks **D**, **6a**, **9a** and **10a** are typical of DPP, Bodipy and SubPc dyes with a

Table 2. Photophysical properties measured in THF at room temperature.

| | $\lambda_{abs}$ [nm] | $\varepsilon$ [$M^{-1}$ cm$^{-1}$] | $\lambda_{em}$ [nm] | $\Phi_f$[a] | $\tau_s$ [ns] | $k_{en\phi}$ or $k_{en(\tau)}$ [s$^{-1}$] |
|---|---|---|---|---|---|---|
| **D** | 483 | 19,900 | 547 | 0.90 | 6.40 | – |
| **6a** | 645 | 127600 | 659 | 0.47 | 4.78 | – |
| **7** | 645 | 153900 | 659 | 0.47 | 4.80 | $k_{en(\phi)}$ $1.7\times10^9$ |
| | 566 | 112800 | 573 | 0.03 | 0.46 | $k_{en(\tau)}$ $1.6\times10^9$ |
| **8** | 645 | 142700 | 659 | 0.47 | 4.82 | $k_{en(\phi)}$ $1.2\times10^{10}$ |
| | 498 | 37331 | 566 | 0.01 | $<0.1$ | – |
| **9a** | 565 | 105000 | 574 | 0.11 | 1.80 | – |
| **10a** | 565 | 86400 | 574 | 0.11 | 1.78 | – |
| **11** | 645 | 127000 | 659 | 0.36 | 4.81 | $k_{en(\phi)}$ $3.2\times10^8$ |
| | 566 | 96000 | 575 | 0.07 | 1.18 | $k_{en(\tau)}$ $2.9\times10^8$ |

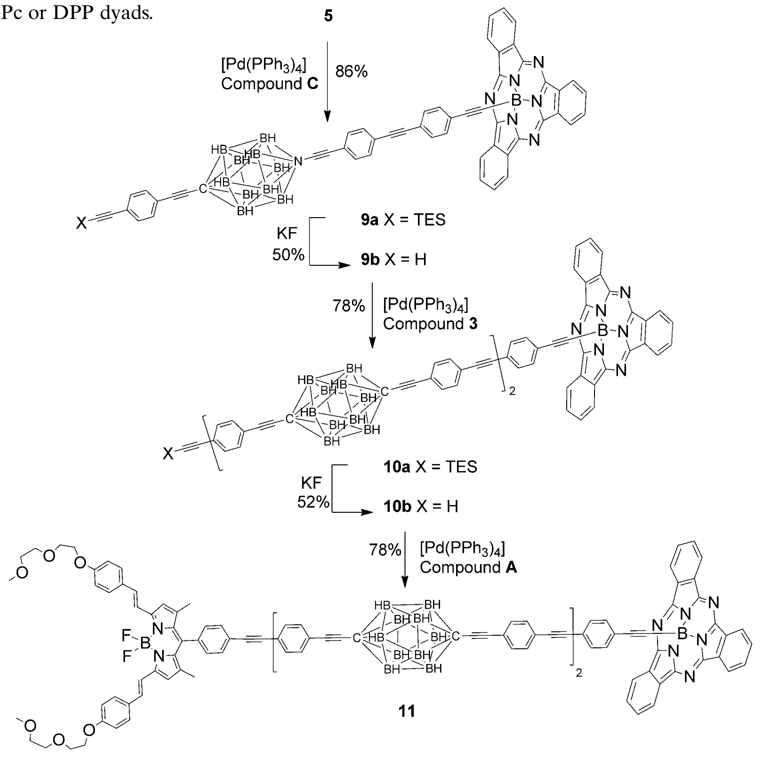
[a] Quantum yield determined in THF solution, about $5\times10^{-7}$ M at a given wavelength using as reference tetramethoxydiisoindomethenedifluoroborate ($\phi_f = 0.51$ in methanol).[22] All $\Phi_F$ are corrected for changes in refractive index. [b] Calculated using the following equations : $k_{en\phi} = [(\Phi_0/\Phi)-1]/\tau_0$, $k_{en(\tau)} = 1/\tau - 1/\tau_0$).



Scheme 2. Synthesis of Bodipy–carborane-bridged SubPc or DPP dyads.

strong $\pi$–$\pi^*$ transitions in the visible region. The linkage of carborane subunits to the Bodipy or SubPc dyes does not significantly perturb the fluorescence properties. This is in line with results found with fluorescent “nanocars”,[21] and related dyads.[12] For the dyad **8**, the absorption spectrum (Figure 3b) is almost a linear combination of the absorption of **6a** (Figure 3a) and of DPP. Irradiation in the DPP absorption band at 480 nm resulted in very efficient electronic energy transfer (EET > 99 %) to the red emitter. The rate constant for this EET is about $1.2\times10^{10}\,\mathrm{s}^{-1}$, taking into account the quantum yields and the excited state lifetime of the DPP moiety (Table 1). The excitation spectrum matches the absorption spectrum fairly well over the entire absorption window (Figure 3b).

On increasing the number of carborane units in the SubPc precursors **9a** and **10a** the transitions corresponding to the SubPc group in the 450 to 570 nm region remain identical, while the absorption due to the carboraneethynylphenyl group between 250 and 355 nm increases accordingly (Figure 4a). On linking both scaffoldings to a Bodipy fragment, the absorption spectra are almost linear combinations of the absorptions of the respective modules (Figure 4b), but the emission spectra showed dual emissions at 575 and 659 nm. In case of dyad **7**, linked with one carborane unit, the residual emission at 575 nm is very weak, highlighting an almost quantitative energy-transfer process deduced from the red emitter at



Scheme 3. Synthesis of the Bodipy/SubPc dyad spaced by two carborane subunits.

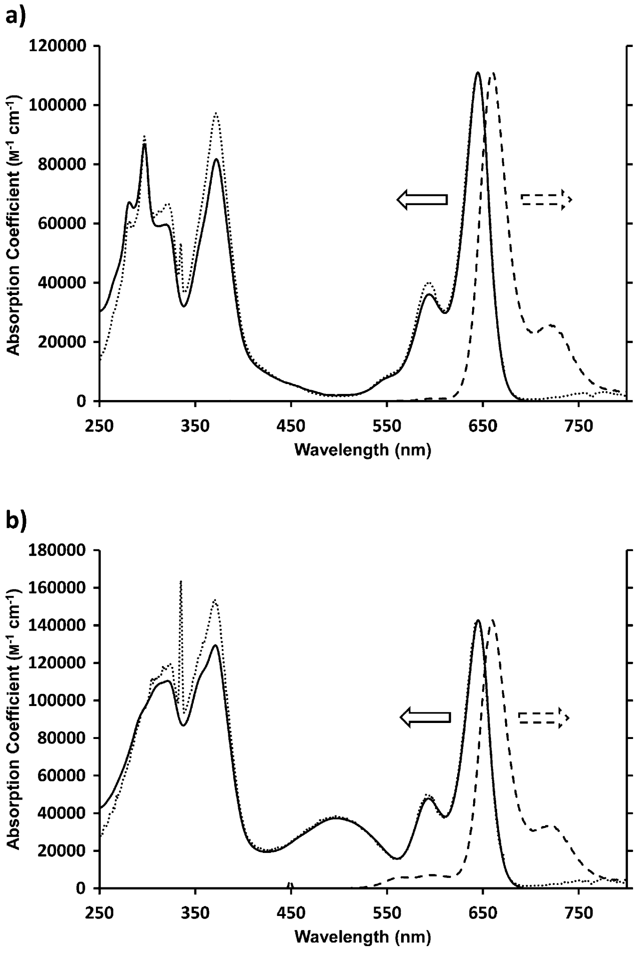


Figure 3. Absorption (black line), emission (dashed line) and excitation (dotted line) spectra of a) dye **6a** and b) dyad **8** in THF at room temperature.

659 nm. When two carborane moieties are linked in dyad **11**, a non-quantitative energy-transfer process, reaching 76 % efficiency was calculated. In both cases, the excitation spectra overlap with the absorption spectra, proving that the SubPc units are involved in the energy transfer. The rates have been calculated by taking into account the quantum yields and lifetimes of the model compounds **9a** and **10a** ($k_{en(\phi)}=1.7\times10^9$ s$^{-1}$ and $k_{en(\phi)}=3.2\times10^8$ s$^{-1}$ in dyes **7** and **11**, respectively). The rates calculated from the residual lifetimes are in excellent agreement with those previously calculated from the quantum yields ($k_{en(\tau)}=1.6\times10^9$ s$^{-1}$ and $k_{en(\tau)}=2.9\times10^8$ s$^{-1}$ , respectively, in dyes **8** and **11**; Table 2).

Note that the fluorescence is insensitive to the presence of oxygen and the short lifetimes (ns) are consistent with a singlet-state emission. The measured Stokes shifts are small ($<330$ cm$^{-1}$) for the model dyes **6a**, **9a** and **10a**, but are significantly increased in the dyads, reaching 4900 cm$^{-1}$ in the best cases.

In summary, we have employed an inert *closo*-carborane spacer that has been functionalized, for the first time, at the vertices with two different functional groups of different reactivity. This strategy allows the stepwise post-linking of two

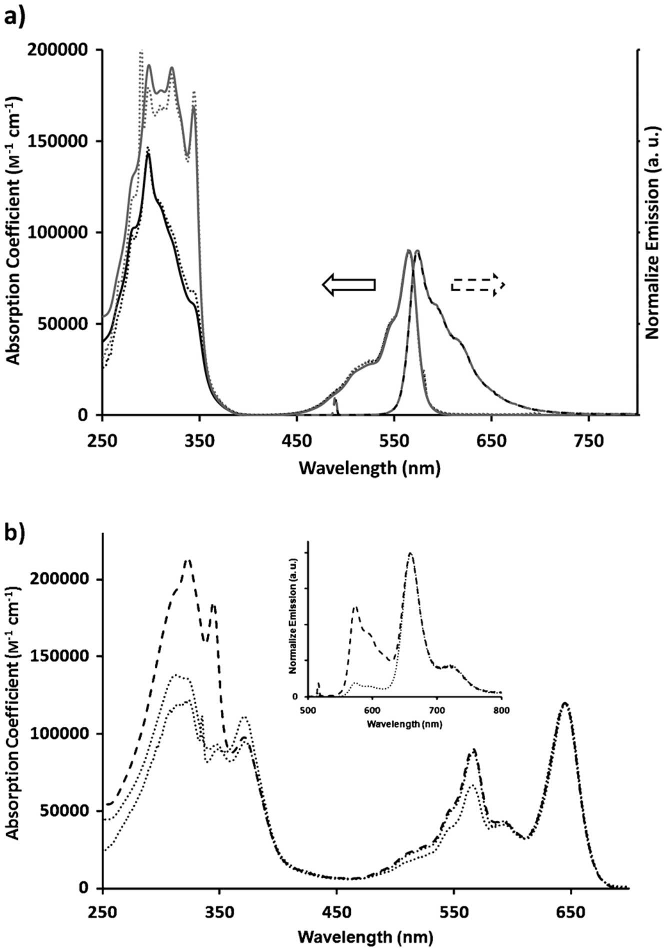


Figure 4. a) Absorption (black line), emission (dashed line) and excitation (dotted line) spectra of dyes **9a** and **10a**. b) Absorption and excitation spectra of dyad **7** (dotted line) and **11** (dashed line): inset fluorescence of **7** (dotted line) and **11** (dashed line) in THF at room temperature.

different photoactive modules displaying specific spectroscopic features suitable for efficient singlet energy transfer. This chemistry paves the way for the engineering of larger rigid systems.

## Experimental Section

**Selected data for 7**: $^1$H NMR (400 MHz, $CD_2Cl_2$): $\delta=8.80$–8.90 (4 line m, 6H), 7.89–7.99 (4 line m, 6H), 7.67 (d, $J=8.3$ Hz, 2H), 7.52–7.63 (4 line m, 6H), 7.47 (d, $J=8.6$ Hz, 2H), 7.37 (d, $J=8.3$ Hz, 2H), 7.19–7.34 (5 line m, 8H), 7.10 (d, $J=8.6$ Hz, 2H), 6.96 (d, $J=8.6$ Hz, 4H), 6.71 (d, $J=8.6$ Hz, 2H), 6.66 (s, 2H), 4.11–4.21 (3 line m, 4H), 3.79–3.88 (3 line m, 4H), 3.63–3.72 (4 line m, 4H), 3.60–1.55 (large m, 10H, BH), 3.50–3.59 (4 line m, 4H), 3.35 (s, 6H), 1.50 ppm (s, 6H); 13C NMR (100 MHz, $CD_2Cl_2$): $\delta=160.45$, 153.32, 151.09, 142.57, 137.96, 136.59, 136.12, 133.53, 132.86, 132.46, 132.32, 132.06, 131.91, 131.57, 131.41, 130.37, 130.14, 129.55, 124.34, 124.21, 123.28, 122.74, 122.49, 121.72, 121.47, 118.15, 117.47, 115.50, 91.56, 90.71, 90.34, 87.47, 80.67, 72.53, 71.26, 70.14, 68.26, 59.25, 15.20 ppm; ESI-MS *m*/*z* (%): 1620.6 (100), 1622.6 (70); elemental analysis calcd (%) for $C_{97}H_{80}B_{12}F_2N_8O_6$: C 71.85, H 4.97, N 6.91; found: C 71.56, H 4.75, N 6.71.

**Selected date for 8**: $^1$H NMR (400 MHz, $CD_2Cl_2$): $\delta = 7.78$ (d, $J = 8.1$ Hz, 2H), 7.73 (d, $J = 8.6$ Hz, 2H), 7.68 (d, $J = 7.6$ Hz, 2H), 7.43–7.64 (10 line m, 14H), 7.38 (d, $J = 8.1$ Hz, 2H), 7.22–7.35 (m, 12H), 7.18 (d, $J = 7.6$ Hz, 4H), 6.97 (d, $J = 8.6$ Hz, 4H), 6.66 (s, 2H), 4.98 (s, 4H), 4.12–4.22 (3 line m, 4H), 3.78–3.90 (3 line m, 4H), 3.63–3.72 (4 line m, 4H), 3.52–3.59 (4 line m, 4H), 3.49 (s, 2H), 3.62–1.57 (large m, 10H, BH), 3.35 (s, 6H), 2.34 (s, 6H), 1.51 ppm (s, 6H); $^{13}$C NMR (100 MHz, $CD_2Cl_2$): $\delta = 163.05$, 160.45, 153.32, 148.75, 148.40, 142.57, 138.11, 137.96, 136.59, 136.13, 133.52, 132.86, 132.48, 132.40, 132.17, 132.08, 130.13, 129.63, 129.55, 129.34, 128.36, 127.97, 127.15, 127.13, 126.97, 126.36, 124.37, 124.21, 121.88, 121.72, 118.15, 117.46, 115.50, 110.91, 110.75, 92.05, 91.58, 90.34, 87.62, 80.67, 72.53, 71.26, 70.14, 68.26, 66.85, 59.26, 49.04, 46.12, 44.48, 15.21 ppm; ESI-MS $m/z$ (%): 1675.7 (85), 1674.7 (60), 1673.7 (100); elemental analysis calcd (%) for $C_{102}H_{94}B_{11}F_2N_5O_8$: C 73.15, H 5.66, N 4.18; found: C 72.84, H 5. 43, N 3.76.

**Selected data for 11**: $^1$H NMR (400 MHz, $CD_2Cl_2$): $\delta = 8.79$–8.90 (4 line m, 6H), 7.87–7.99 (4 line m, 6H), 7.68 (d, $J = 8.3$ Hz, 2H), 7.53–7.64 (3 line m, 6H), 7.49 (d, $J = 8.3$ Hz, 2H), 7.18–7.46 (m, 18H), 7.11 (d, $J =$ 8.3 Hz, 2H), 6.97 (d, $J = 8.3$ Hz, 4H), 6.72 (d, $J = 8.3$ Hz, 2H), 6.66 (s, 2H), 4.12–4.21 (3 line m, 4H), 3.80–3.88 (3 line m, 4H), 3.64–3.72 (4 line m, 4H), 3.62–1.52 (large m, 20H, BH), 3.51–3.59 (4 line m, 4H), 3.36 (s, 6H), 1.51 ppm (s, 6H); $^{13}$C NMR (100 MHz, $CD_2Cl_2$): $\delta = 160.47$, 153.35, 151.09, 142.56, 137.96, 136.61, 136.14, 133.55, 132.87, 132.48, 132.43, 132.32, 132.09, 132.06, 131.92, 131.85, 131.58, 131.41, 130.36, 130.16, 129.56, 124.40, 124.26, 123.30, 122.76, 122.49, 121.76, 121.49, 118.18, 117.51, 115.52, 91.63, 91.46, 90.73, 90.38, 87.65, 87.50, 80.69, 72.54, 71.28, 70.16, 68.34, 68.28, 66.86, 59.27, 34.72, 26.17, 22.92, 15.22, 14.39 ppm; ESI-MS $m/z$ (%): 1987.0 (100); elemental analysis calcd (%) for $C_{117}H_{98}B_{22}F_2N_8O_6$: C 70.69, H 4.97, N 5.64; found: C 70.49, H 4.76, N 5.48.

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**Keywords:** alkynes • carboranes • donor–acceptor systems • energy transfer • fluorescence • phthalocyanines

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