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Synthesis, Photophysical Properties, and Field-Effect Characteristics of (Ethynylphenyl)benzimidazole-Decorated Anthracene and Perylene Bisimide Derivatives

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A series of anthracene and perylene bisimide derivatives with electron-withdrawing benzimidazole substituents has been designed and prepared. Detailed studies on the electrochemical and photophysical properties as well as the field-effect mobilities of these new compounds were explored. The incorporation of electron-withdrawing benzimidazole groups lowered the LUMO levels in both anthracene and perylene bisimide derivatives compared to those of the parent compounds. Strong emission was observed for all anthracene derivatives, but only weak emission was observed for perylene bisimide derivatives. The anthracene derivatives showed typical p-type semiconducting character, even when the derivatives were substituted with electron-withdrawing benzimidazole groups at the 9- and 10-positions, which apparently does not lower the LUMO levels to transform them into electron-transporting molecules. Perylene bisimide derivatives displayed typical n-channel semiconducting properties with low threshold voltages and electron mobilities of ca. $5.2 \times 10^{-5}\ \mathrm{cm^2\,V^{-1}\,s^{-1}}$.

Introduction

Great attention has been paid to developments in the field of organic materials for field-effect transistors during the past decade or two.[1] Such devices have potential applications in the switching element in flat panel displays and smart cards. In general, organic field-effect transistors (OFET) can be fabricated at low cost if they can be deposited from solution, as this enables the easy fabrication over large areas and on flexible substrates.[2] Despite rapid development in recent years, the quest for more effective organic semiconductors that possess charge carrier mobilities comparable to those of their inorganic counterparts still requires more effort. In particular, p-type semiconducting materials have shown great progress in recent years. Pentacene, for example, has shown one of the highest OFET mobilities and nearly reached the intrinsic transport limit of organic single crystals.[3] On the other hand, the development of n-type OFETs has generally lagged behind p-type OFETs, except for a few prominent examples.[4] The major reasons for the relatively slow progress of n-type semiconducting materials are that they are air- and/or moisture-sensitive, they lack volatility for efficient film growth, and they are somewhat difficult to synthesize.[5] Indeed, both kinds of semiconductors are required for the fabrication of complementary integrated circuits, bipolar transistors, or organic p/n junctions. The development of new materials with high electron affinities for n-channel semiconductors has been an area of intensive research since the first report of n-type OFETs based on metallophthalocyanines.[6] Many electron-withdrawing substituents such as perfluoroalkyl,[7] perfluorophenyl,[8] imide,[9] cyano,[10] and fluoro[11] groups have been incorporated into aromatic π-conjugated cores to show desired n-type semiconducting properties.

Anthracene belongs to the class of acenes, but it is more stable than larger acenes.[12] The derivatives of anthracene have generally shown typical p-channel FET mobilities.[12,13] The large π-conjugation in the anthracene core has been proposed to enhance electron delocalization and promote charge carrier mobility in the resulting FET devices. On the other hand, perylene bisimide (PBI) derivatives are promising candidates for applications in molecular electronic devices.[14] A number of perylene bisimide derivatives has shown impressive field-effect mobilities with n-type semiconducting character due to the electron-deficient nature of the perylene bisimide core.[2a,15] It is also possible to convert typical electron-rich molecules to n-type semiconducting materials with appropriate incorporation of

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electron-withdrawing groups to lower the LUMO levels for effective electron transport. It has been demonstrated that pentacene, as the benchmark p-channel compound, can be transformed into an n-type charge carrier by its substitution with electron-withdrawing groups.[11a] The principles of our molecular design in the current study are based on (1) the rigid core structures of anthracene and PBI with extended π-conjugation to facilitate efficient π-electron delocalization and possible intermolecular π–π stacking, (2) incorporation of electron-withdrawing benzimidazole segments to depress the LUMO levels for effective electron transport and air stability by energetically stabilizing the induced electrons during charge transport,[10c] and (3) alkyl side chains in the benzimidazole groups to increase solubility for easy solution-processed fabrication without disrupting π-conjugation in structural frameworks and to provide potential intrusion of oxygen and moisture into the active layer of the resulting devices.[16] Herein, we report the synthesis, electrochemical, and photophysical properties and field-effect transistor characteristics of a series of anthracene and PBI derivatives for n-type semiconductors by direct functionalization of the anthracene and PBI cores with electron-withdrawing (ethynylphenyl)benzimidazole segments.

Results and Discussion

Synthesis, Crystal Structure, and Electrochemical Properties

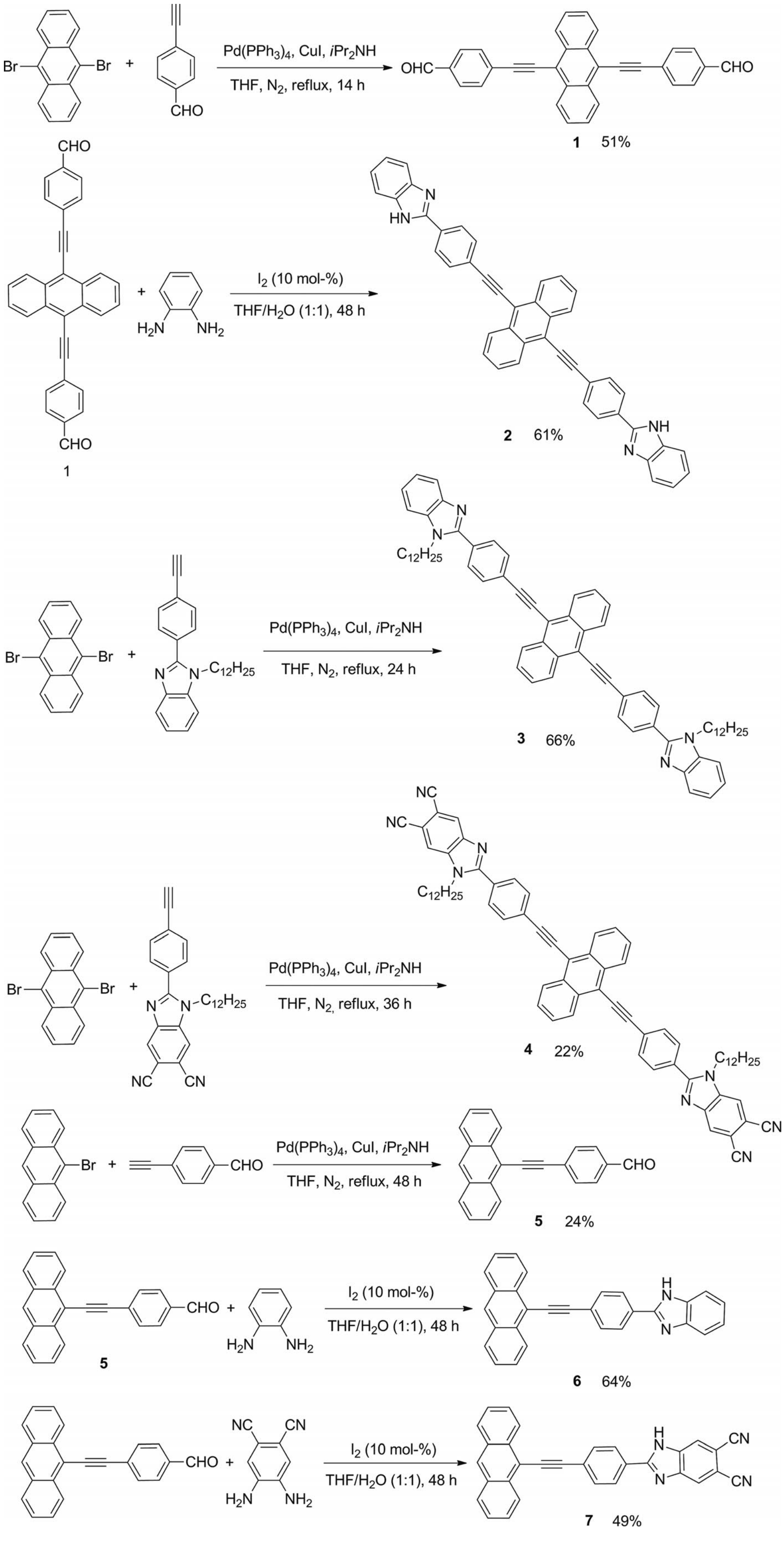
The anthracene and perylene bisimide derivatives investigated in this work were synthesized according to the procedures in Schemes 1 and 2, respectively. Anthracene derivatives **1**–**7** were obtained by palladium-catalyzed Sonogashira coupling of 9-bromoanthracene or 9,10-dibromoanthracene with the corresponding electron-withdrawing (ethynylphenyl)benzimidazoles. The analytically pure products were isolated by flash column chromatography.

The PBI derivatives were synthesized by direct functionalization of the PBI core at the bay positions. We chose the core PBI structure with branched alkyl chains attached to the imide nitrogen atoms to provide sufficient solubility for solution-processed semiconductors for OFETs. It is also known that PBI derivatives bearing branched alkyl chains are prone to organize into lamellar arrangements that are potentially advantageous for OFETs. Dibromoperylene bisimide derivative **8** was synthesized in two steps from perylene bis(anhydride) in a total yield of 69%. Subsequently, Sonogashira coupling of compound **8** with the corresponding benzimidazole-derived phenyl acetylene afforded compounds **9**, **11**, and **12**. Attempts to prepare **10** through direct palladium-catalyzed Sonogashira coupling of **8** with 2-(4-ethynylphenyl)-1*H*-benzo[*d*]imidazole were unsuccessful.[17] Instead, compound **10** was prepared by condensation reaction between **9** and *o*-phenylenediamine. All new compounds synthesized were fully characterized by ^{1}H NMR spectroscopy, high-resolution mass spectrometry, and elemental analysis. They are highly thermally stable with decomposition temperatures ranging from 247 °C (for **5**) to 464 °C (for **1**) by thermogravimetric analysis accounting for 5% mass loss as the thermolysis threshold. The high thermal stabilities of these compounds allow thin film preparation over a broad range of annealing temperatures.

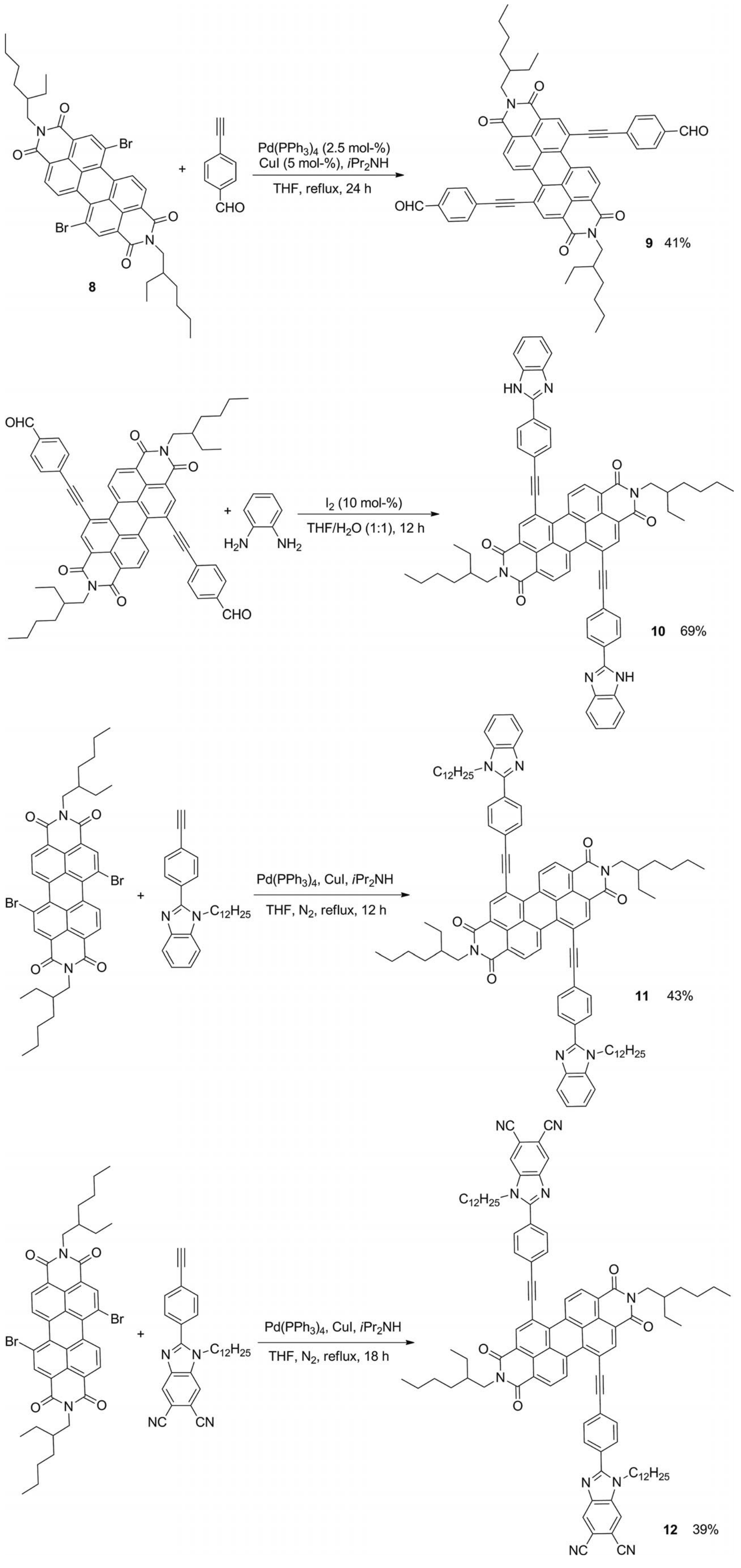
Yellow single crystals of compound **1** were grown by slow diffusion of hexane into a chloroform solution, and the crystal structure determined by X-ray diffraction is shown in Figure 1; the crystal and refinement parameters are collected in Table S1 (Supporting Information). Compound **1** crystallizes in the $P2_1/c$ space group. The angle between the central anthracene and peripheral ethynylbenzaldehyde is 19.2°. The molecules adopt herringbone packing in the crystal, similar to that of anthracene.[1] There is no significant π–π overlap in the crystal structure, but there are strong C–H···π interactions between the anthracene and adjacent benzaldehyde with a distance of 3.06 Å.

The electrochemical properties of anthracene and PBI derivatives were characterized by cyclic voltammetry in DMF or CH_2Cl_2 containing 0.1 M Bu_4NPF_6 as the supporting electrolyte. Table 1 summarizes the potentials quoted relative to the ferrocenium/ferrocene couple. The anthracene derivatives exhibit one or two reversible reduction waves and one irreversible oxidation wave for bis[(ethynylphenyl)benzimidazole] compounds **2**–**4**, whereas no oxidation wave could be observed for mono[(ethynylphenyl)benzimidazole] compounds **6** and **7** within the solvent window. Assuming that the Koopman's theorem holds ($EA^{red} \approx -LUMO^{red}$),[18] the LUMO levels can be estimated from the first half-wave reduction potentials by taking the ferrocene energy level to be –4.8 eV below the vacuum level.[19] The converted energy levels are also summarized in Table 1 for comparison. The LUMO levels in anthracene derivatives range from –2.96 to –3.49 eV, which are lower than the LUMO energy of parent 9,10-bis(phenylethynyl)anthracene (ε_{LUMO} = –2.92 eV),[20] indicating that the electron-withdrawing effect of the aldehyde and benzimidazole groups played a part in lowering the LUMO energies. Indeed, the LUMO energies of anthracene derivatives **1**–**7** are comparable to some fluorinated or chlorinated anthracene derivatives, indicating that the electronic effects of fluoride and chloride substitution of the benzimidazole are similar.[21]

The first reduction potentials of the PBI derivatives range from –0.83 V (for **10**) to –1.01 V (for **11**). PBI derivatives **9**–**12** can be easily reduced by ≈90–270 mV due to the electron-withdrawing benzimidazole substituents when compared to parent alkyl-substituted PBIs (E_{red} = –1.10 V).[22] The LUMO levels of compounds **9**–**12** are comparable to some halo-substituted PBI derivatives.[11a,11b,23] The benzimidazole substituents effectively lower the LUMO energy of **10** to –3.97 eV. The introduction of alkyl groups on the benzimidazoles not only significantly increases the solubility of compound **11** but also provides hydrophobic side chains to prevent the intrusion of oxygen and moisture into the active layer of the resulting field-effect transistors. The drawback of these alkyl groups is to slightly raise the LUMO energy up to –3.79 eV (for



Scheme 1. Synthetic procedures for anthracene derivatives.



Scheme 2. Synthetic procedures for perylene bisimide derivatives.

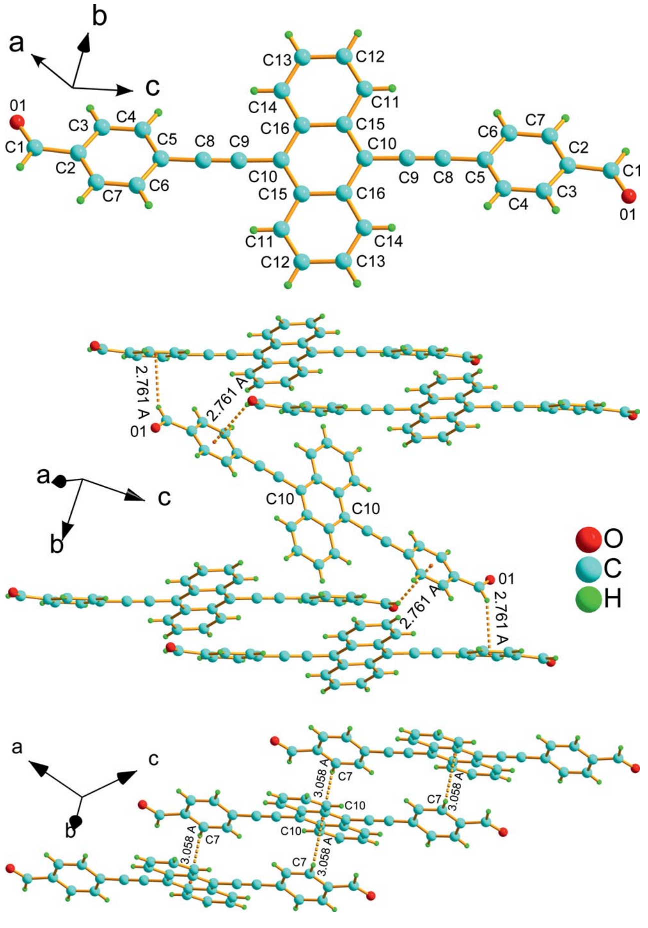


Figure 1. Crystal structure of **1** (top) showing notable C–H···π (middle) and π–π (bottom) interactions in the crystal packing.

11). This negative effect can be compensated by introducing cyano groups in the benzimidazoles to bring the LUMO energy back down to –3.86 eV (for **12**).

Table 1. Redox potentials (vs. Fc/Fc^+) and calculated HOMO and LUMO energies.[a]

Compd.	E_{ox} [V]	E_{red} [V]	ε_{HOMO}[c] [eV]	ε_{LUMO}[c] [eV]
1	0.77	–1.54, –1.72	–5.57	–3.25
2	0.54	–1.70	–5.34	–3.10
3[b]	0.68	–1.82	–5.48	–2.98
4[b]	0.72	–1.76	–5.52	–3.04
5		–1.84		–2.96
6		–1.36, –2.04		–3.44
7		–1.30		–3.49
9[b]		–0.96, –1.16		–3.84
10		–0.83, –1.08, –1.42		–3.97
11[b]		–1.01, –1.22		–3.79
12[b]		–0.94, –1.19		–3.86

[a] Measured in 0.1 M Bu_4NPF_6 in anhydrous DMF unless noted, scan rate of 100 mV s^{-1}, ferrocene served as internal standard. [b] Measured in anhydrous CH_2Cl_2. [c] Determined according to literature methods: $\varepsilon_{HOMO} = -4.8\ eV - E_{ox}$; $\varepsilon_{LUMO} = -4.8\ eV - E_{red}$.

Photophysical Properties

The absorption and fluorescence data of compounds **1**–**12** are collected in Table 2. Representative absorption and fluorescence spectra of compounds **3** and **11** are depicted in Figure 2. The absorption spectra of anthracene derivatives **1**–**7** show characteristic anthracene absorption features between 400 and 500 nm.[20] The absorption maxima of the bis[(ethynylphenyl)benzimidazole]-substituted compounds are redshifted by ≈50–60 nm vs. the parent anthracene and mono(ethynylphenyl)benzimidazole]-substituted compounds. The bathochromic shift is consistent with enhanced π-conjugation of the molecular backbone and the incorporation of the electron-withdrawing phenylbenzimidazole group. Compounds **1**–**7** all exhibit strong fluorescence with quantum yields higher than 0.5 and excited-state lifetimes of a few nanoseconds.

Table 2. Absorption and fluorescence properties in DMF solution.

Compd.	Absorption λ_{max} [nm] ($\varepsilon \times 10^{-3}$ [$M^{-1}\,cm^{-1}$])	Fluorescence λ_{fl} [nm][a]	Φ_{fl}[a]	τ [ns]
1	315 (18.2), 329 (24.8), 458 (32.4), 484 (34.3)	503	0.78	2.8
2	338 (14.5), 351 (14.3), 437 (sh., 10.0), 463 (13.9), 489 (13.5)	503	0.53	1.8
3	329 (38.0), 455 (38.3), 481 (40.1)	496	0.61	2.2
4	333 (40.3), 456 (39.3), 482 (41.3)	500	0.64	2.0
5	315 (13.7), 391 (14.0), 413 (23.3), 436 (22.8)	497	0.62	3.6
6	314 (8.5), 328 (13.2), 342 (14.3), 392 (9.4), 411 (14.8), 435 (13.9)	445	0.68	2.9
7	308 (22.3), 343 (21.9), 357 (25.3), 393 (20.6), 411 (32.5), 439 (31.0)	454	0.63	2.6
9	344, 358, 475, 523, 563	585	0.42	6.5
10	345 (48.2), 468 (14.4), 540 (15.8), 574 (15.6)	584	0.016	1.3
11	329 (57.4), 458 (12.7), 530 (20.1), 572 (25.1)	598	0.0077	[b]
12	334 (65.7), 429 (15.2), 450 (14.0), 530 (24.8), 568 (35.4)	595	0.057	5.8

[a] Fluorescence quantum yields in optically dilute toluene solution. The quantum yields were determined by using 9,10-diphenylanthracene in cyclohexane ($\Phi_{em} = 0.90$).[24] The molar absorptivity of compound **9** was not determined due to the extremely low solubility in DMF. [b] The lifetime (<100 ps) is too short to be measured in our instrument.

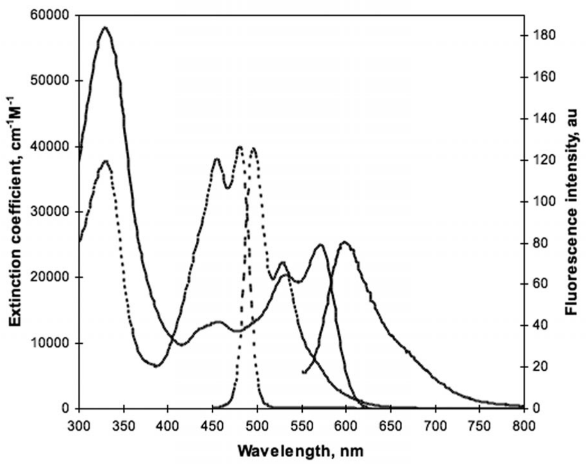


Figure 2. Absorption and fluorescence spectra of compounds **3** (dashed lines) and **11** (solid lines) measured in a DMF solution.

The absorption spectra of PBI derivatives **9**–**12** show typical spectral features of perylene bisimide chromophores in the range of 450–600 nm with a well-defined vibronic structure.[25] As shown in Figure 2, a strong absorption band near 330 nm is attributed to the π–π* transition located on the (ethynylphenyl)benzimidazole moieties. The low solubility of compound **9** precluded a precise measurement of its molar extinction coefficient in DMF. The absorption maximum of the allowed S_0–S_1 transition appears at 563 nm for **9** and is further redshifted to ≈570 nm for **10**–**12** due to the extended conjugation on benzimidazoles. The HOMOs of **10** and **11** (Figure 3) calculated by using DFT with the B3LYP/6-31+(d) basis set and the Gaussian03 program[26] show electron delocalization to the (ethynylphenyl)benzimidazole moiety but only to the ethynylphenyl moiety in the case of compound **12**, whereas the LUMOs mainly concentrate in the parent PBI moiety with very minor extension to the (ethynylphenyl)benzimidazole moieties.

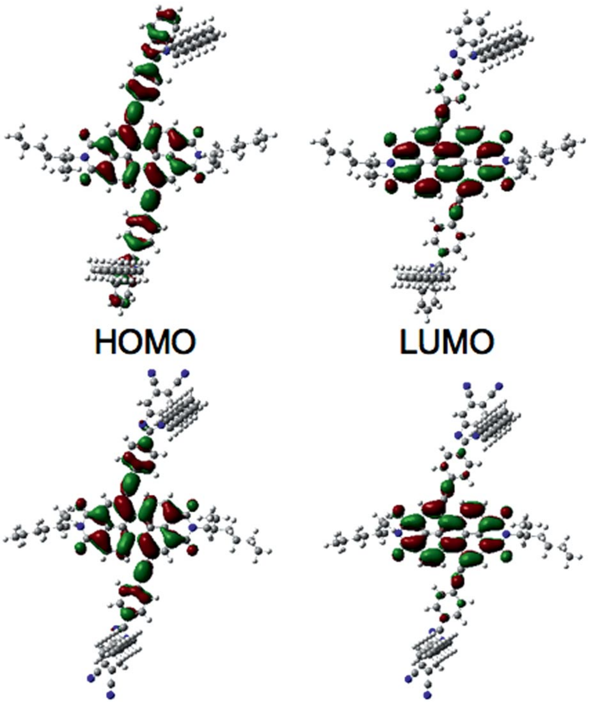


Figure 3. Pictorial presentation of the HOMOs and LUMOs of compounds **11** (top) and **12** (bottom) calculated at the B3LYP/6-31+(d) level solvated with THF.

The fluorescence maxima of compounds **9**–**12** all appeared at λ_{max} = 584–598 nm, which are mirror images relative to the respective S_0–S_1 absorption bands (see Figure 2). Unlike typical PBI derivatives that usually display strong fluorescence with quantum yields near unity, the PBI derivatives of **10**–**12** showed very weak emission with emission quantum yields lower than 0.06 and fluorescence lifetimes on the nanosecond time scale. The lack of characteristic vibrational features in the fluorescence spectra in compounds **10**–**12** implies a less-rigid structural framework than those of typical PBI derivatives that may contribute to a more efficient nonradiative relaxation to give lower quantum yields.

Thin-Film Transistor Device Characterization

The charge transport properties of these benzimidazole-substituted anthracenes and perylene bisimides were examined through their incorporation into OFET devices. OFETs with top-contact/bottom gate device structures were fabricated by spin coating on octadecyltrichlorosilane (OTS)-treated SiO_2/p-type Si substrates. All spin-coating processes were carried out under ambient conditions and the devices were then annealed at 100 °C for 30 min under an atmosphere of nitrogen. Finally, Ca/Al or Au contacts were patterned by thermal evaporation by using a shadow mask to give channel lengths of 100 µm and widths of 2 mm. FET properties were evaluated under positive or negative gate bias under an atmosphere of nitrogen to explore the majority charge carrier type and device performance. All charge mobilities and threshold voltages were calculated in the saturation regime. Table 3 summarizes the FET data, and the representative transfer and output plots for compound **11** are illustrated in Figure 4.

Table 3. Majority charge carrier types, field-effect mobilities (μ), threshold voltages (V_T), and current I_{on}/I_{off} ratios for thin films of compounds fabricated by spin-coating on OTS-treated SiO_2/p^+-Si substrates.[a]

Compd.	Polarity	μ [$cm^2 V^{-1} s^{-1}$]	I_{on}/I_{off}	V_T [V]
1	p	[b]	[b]	[b]
9	n	4.2×10^{-6}	10	9
11	n	5.2×10^{-5}	10	12
12	n	1.1×10^{-5}	10^2	6

[a] SiO_2 dielectric (300 nm, Ci = 10 nF/cm²), L = 100 µm, W = 2 mm. The mobilities were determined in the saturation regime from the slope of plots of $(I_{DS})^{1/2}$ vs. V_{GS}. [b] The current is too small to extract the values.

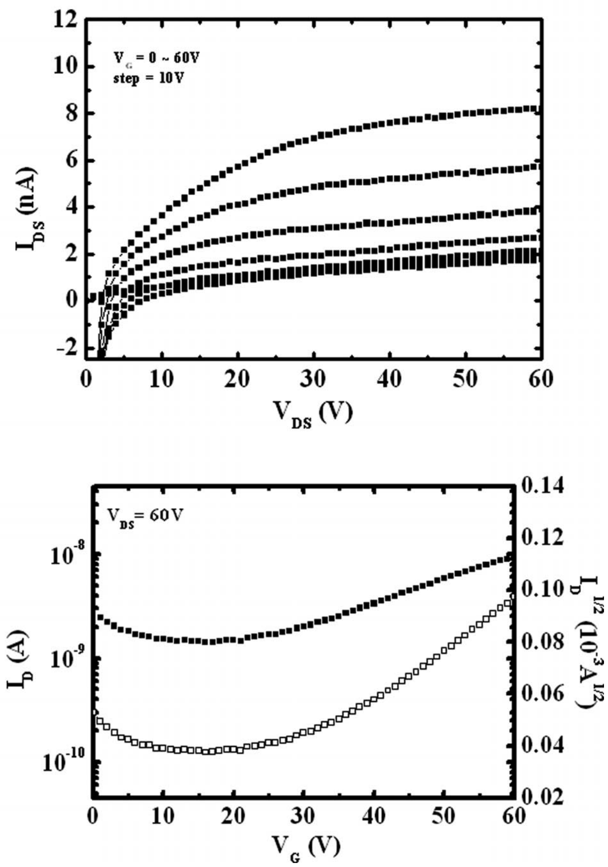


Figure 4. Output characteristic (top) and I–V transfer plots (bottom) for compound **11**.

Devices fabricated with compounds **2**–**7** were found to exhibit no FET activity. Compound **1** was found to show p-channel character, whereas compounds **9**, **11**, and **12** exhibit FET activity as n-channel semiconductors. Compound **10** was not fabricated for device measurement due to the extremely low solubility in common organic solvents. Compound **1** is a p-channel semiconductor with very weak electrical response. The attachment of electron-deficient benzimidazole groups is apparently not effective enough to lower the LUMO level to allow the electron flow within the device.

Compounds **9**, **11**, and **12** displayed n-channel properties with a positive gate voltage bias to induce the accumulation of electron carriers and current flows in the channel between source and drain electrodes. The highest mobility among the three PBI derivatives was observed for compound **11**, which showed a charge carrier mobility of 5.2×10^{-5} $cm^2 V^{-1} s^{-1}$. All three compounds gave excellent threshold voltages with the values around 10 V or less. The low V_T indicates the carrier traps in the devices were relatively few and resulted in such low threshold voltages. The I_{on}/I_{off} ratios, however, gave disappointing values of only 10–100. The low charge mobilities and I_{on}/I_{off} ratios may be attributed to the intrinsic molecular property or the lack of long-range π stacking of the molecules in the thin film morphology,[27] as implied from the lack of sharp and strong diffractions in powder X-ray diffraction spectra of compounds **9**-, **11**-, and **12**-based thin films prepared from spin-coating on Si/SiO_2 with a nominal thickness of 50 nm (see Figures S5–S7 in the Supporting Information).

Conclusions

In summary, we have prepared a series of potential n-type semiconducting materials. The attachment of electron-withdrawing benzimidazole groups to the parent anthracene or perylene bisimide lowers the LUMOs of the resulting compounds. Some of these benzimidazole-substituted perylene bisimides have measurable field-effect mobilities. The anthracene derivative showed a typical character of p-channel semiconducting properties, whereas perylene bisimide derivatives displayed expected n-channel semiconducting properties with electron mobilities of ca. 5.2×10^{-5} $cm^2 V^{-1} s^{-1}$ and low threshold voltages. Relatively easy synthetic accessibility and thermal stability of these benzimidazole-substituted anthracenes and perylene bisimides allows for further functionalization to a variety of new derivatives with potentials for understanding of charge transport properties in these materials.

Experimental Section

Materials and Methods: All chemicals are commercially available unless otherwise noted. All reactions and manipulations were carried out under a nitrogen atmosphere with the use of standard inert-atmosphere and Schlenk techniques. Solvents used for synthesis were dried by standard procedures and stored under an atmosphere of nitrogen. Compounds **8**,[28] 2-(4-ethynylphenyl)benzimidazole,[29] and 4,5-diaminophthalonitrile were synthesized according to published methods. The syntheses of 1-dodecyl-2-(4-ethynylphenyl)benzimidazole and 1-dodecyl-2-(4-ethynylphenyl)benzimidazole-5,6-dicarbonitrile are described in the Supporting Information. NMR spectra were recorded with a Bruker AMX400 spectrometer. ^1H NMR chemical shifts are reported in ppm downfield from tetramethylsilane (TMS) with the solvent resonances as internal standards. Cyclic voltammetry experiments were performed with a CHI electrochemical analyzer. All measurements were carried out at room temperature with a conventional three-electrode configuration consisting of a platinum working electrode, a platinum wire auxiliary electrode, and a nonaqueous Ag/$AgNO_3$ reference electrode.[30] The potentials were quoted against the ferrocene/ferrocenium internal standard. The solvent containing 1.0 mM of the analyte and 0.1 M tetrabutylammonium hexafluorophosphate as the supporting electrolyte in all experiments. Absorption spectra were obtained by using a Varian Cary 300 UV/Vis spectrophotometer. Emission spectra were recorded with a Fluorolog III photoluminescence spectrometer. Luminescence quantum yields in solution were calculated relative to diphenylanthracene in cyclohexane (Φ = 0.95).[24] Corrected emission spectra were used for the quantum yield measurements and all reported data. Luminescence lifetimes were determined on a time-correlated pulsed single-photon-counting instrument as reported previously.[31] The geometry and electronic properties of the compounds were calculated with the Gaussian03 program package.[26] All calculations were performed by using density functional theory (DFT) with the B3LYP/6-31G+(d) basis set.

Device Fabrication and Electrical Measurements: The devices were fabricated on a heavily doped p-type Si wafer as gate electrode with 300-nm thick layer of thermally oxidized silicon dioxide. After routine solvent cleaning, the substrates were treated with octadecyltrichlorosilane following a procedure reported previously.[32] Then, the semiconductor layer was spun over the (OTS)-treated SiO_2 surface from a 1 wt.-% ratio chloroform solution. Prior to source and drain electrodes deposition, the device was thermally annealed on a hot plate at 70 °C for a period of 30 min. Finally, layers of Ca (30 nm) and Al (100 nm) were thermally evaporated under vacuum (pressure: $<6\times10^{-6}$ Torr) through a shadow mask to form the source and drain electrodes. The typical channel length and width of the devices were 100 and 2 mm, respectively. The electrical measurements of the devices were performed by using a HP4156C semiconductor parameter analyzer. All the device fabrication and electrical measurements were carried out in a nitrogen-filled glove box.

Compound 1: To a 150-mL flask containing 4-ethynylbenzaldehyde (0.87 g, 6.7 mmol), 9,10-dibromoanthracene (1.1 g, 3.3 mmol), $Pd(PPh_3)_4$ (0.38 g, 0.33 mmol), and CuI (62 mg, 0.33 mmol) was added diisopropylamine (1 mL) and THF (60 mL). The resulting mixture was heated at 80 °C under an atmosphere of nitrogen for 14 h before evaporating all volatiles under vacuum. The crude product was purified by column chromatography (hexane/CH_2Cl_2, 1:5) and recrystallized (THF/EtOH) to afford compound **1** as a yellow solid (0.72 g, 51%). M.p. 224–226 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 10.07 (s, 2 H), 8.65–8.69 (m, 4 H), 7.97 (d, J = 8.4 Hz, 2 H), 7.91 (d, J = 8.4 Hz, 2 H), 7.66–7.70 (m, 4 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 191.6, 136.0, 132.5, 132.4, 130.0, 129.7, 127.6, 127.4, 118.7, 102.0, 90.6 ppm. HRMS (EI): calcd. for $[M]^+$ 434.1307; found 434.1302. $C_{32}H_{18}O_2\cdot0.2H_2O$ (438.08): calcd. C 87.55, H 4.12; found C 87.55, H 4.25.

Compound 2: To a 100-mL flask containing **1** (50 mg, 0.12 mmol), *o*-phenylene diamine (27 mg, 0.25 mmol), and I_2 (3 mg, 11.8 µmol) was added THF/H_2O (30:30 mL). The resulting mixture was stirred in the dark for 3 d before evaporating all volatiles under vacuum. The residue was extracted with CH_2Cl_2 and water. The organic layer was collected, dried with $MgSO_4$, and concentrated to dryness. The crude solid was purified by column chromatography (MeOH/CH_2Cl_2, 1:10). The obtained dark brown solid was further sublimed at 330 °C to afford pure **2** as a brown solid (45 mg, 61%). M.p. 261–264 °C. ^{1}H NMR (400 MHz, [D$_6$]DMSO): δ = 13.12 (s, 2 H), 8.76–8.78 (m, 4 H), 8.36 (d, J = 8.4 Hz, 4 H), 8.10 (d, J = 8.0 Hz, 4 H), 7.85–7.87 (m, 4 H), 7.65 (m, 4 H), 7.24–7.26 (m, 4 H) ppm. ^{13}C NMR (125 MHz, [D$_6$]DMSO): δ = 150.3, 143.9, 135.1, 132.2, 131.4, 130.6, 127.9, 126.8, 126.7, 123.2, 122.9, 121.9, 119.0, 117.6, 111.4, 102.7, 87.6 ppm. HRMS (ESI): calcd. for $[M + H]^+$ 611.2236; found 611.2245.

Compound 3: To a 100-mL flask containing 1-dodecyl-2-(4-ethynylphenyl)benzimidazole (0.32 g, 0.83 mmol), 9,10-dibromoanthracene (140 mg, 0.41 mmol), $Pd(PPh_3)_4$ (24 mg, 0.02 mmol), and CuI (8 mg, 0.04 mmol) was added diisopropylamine (0.5 mL) and THF (60 mL). The resulting mixture was heated at 80 °C under an atmosphere of nitrogen for 24 h before evaporating all volatiles under vacuum. The crude product was purified by column chromatography (hexane, then hexane/CH_2Cl_2, 3:1). The obtained solid was re-dissolved in a minimum amount of THF and precipitated with EtOH (95%, 500 mL) to afford compound **3** as a yellowish green solid (0.23 g, 59%). M.p. 115–118 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.71–8.73 (m, 4 H), 7.93 (d, J = 8.0 Hz, 4 H), 7.82–7.84 (m, 6 H), 7.68 (br. s, 4 H), 7.43 (d, J = 5.0 Hz, 2 H), 7.32–7.34 (m, 4 H), 4.28 (t, J = 7.2 Hz, 4 H), 1.85 (m, 4 H), 1.21–1.25 (m, 36 H), 0.82 (t, J = 6.0 Hz, 6 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 152.9, 143.1, 135.8, 132.2, 132.0, 130.8, 129.5, 127.2, 127.1, 124.8, 123.0, 122.6, 120.1, 118.5, 110.2, 102.0, 88.4, 44.9, 31.9, 29.8, 29.6, 29.5, 29.4, 29.3, 29.0, 26.7, 22.6, 14.1 ppm. HRMS (FAB): calcd. for $[M + H]^+$ 947.5992; found 947.5987.

Compound 4: A procedure similar to that outlined for the synthesis of compound **3** was employed. The starting material was changed from 1-dodecyl-2-(4-ethynylphenyl)benzimidazole to 1-dodecyl-2-(4-ethynylphenyl)benzimidazole-5,6-dicarbonitrile. The desired product was isolated as a bright orange solid (80 mg, 22%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.72–8.69 (m, 4 H), 8.24 (s, 2 H), 7.99 (d, J = 7.6 Hz, 4 H), 7.86 (s, 2 H), 7.84 (d, J = 8.0 Hz, 4 H), 7.71–7.68 (m, 4 H), 4.35 (m, 4 H), 1.83 (m, 4 H), 1.26–1.17 (m, 36 H), 0.82 (t, J = 6.4 Hz, 6 H) ppm. ^{13}C NMR was not acquired due to low solubility. HRMS (FAB): calcd. for $[M + H]^+$ 1047.5802; found 1047.5807.

Compound 5: A procedure similar to that outlined for the synthesis of compound **1** was employed. The starting material was changed from 9,10-dibromoanthracene to 9-bromoanthracene. The desired product was isolated as a golden yellow solid (0.85 g, 24%). M.p. 136–139 °C. ^{1}H NMR (300 MHz, $CDCl_3$): δ = 10.1 (s, 2 H), 8.61 (d, J = 8.7 Hz, 2 H), 8.48 (s, 1 H), 8.03 (d, J = 8.4 Hz, 2 H), 7.93 (d, J = 8.4 Hz, 4 H), 7.62 (m, 2 H), 7.54 (m, 2 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ = 191.3, 135.4, 132.8, 131.9, 131.1, 129.8, 129.7, 128.8, 128.7, 126.9, 126.4, 125.8, 116.2, 99.8, 90.5 ppm. HRMS (EI): calcd. for $[M + Na]^+$ 306.1045; found 306.1049.

Compound 6: A procedure similar to that outlined for the synthesis of compound **2** was employed. The starting material was changed from **1** to **5**. The desired product was isolated as a yellow solid (45 mg, 64%). M.p. 180–184 °C. ^{1}H NMR (400 MHz, [D$_6$]acetone): δ = 12.1 (s, 1 H), 8.74 (d, J = 8.8 Hz, 2 H), 8.68 (s, 1 H), 8.38 (d, J = 8.0 Hz, 2 H), 8.18 (d, J = 8.4 Hz, 2 H), 8.03 (d, J = 8.4 Hz, 2 H), 7.70–7.74 (m, 3 H), 7.60–7.64 (m, 2 H), 7.56 (m, 1 H), 7.25 (m, 2 H) ppm. ^{13}C NMR (100 MHz, [D$_6$]acetone): δ = 151.7, 145.6, 136.3, 133.6, 133.1, 132.4, 131.7, 130.0, 129.4, 128.2, 127.8, 127.4, 127.0, 125.5, 124.0, 123.0, 120.5, 117.5, 112.2, 101.6, 88.7 ppm. HRMS (APCI): calcd. for $[M + H]^+$ 395.1548; found 395.1552.

Compound 7: A procedure similar to that outlined for the synthesis of compound **2** was employed. The starting materials were changed from **1** and *o*-phenylene diamine to **5** and 4,5-diaminophthalonitrile. The desired product was isolated as a yellowish green solid (70 mg, 49%). M.p. 176–178 °C. ^{1}H NMR (400 MHz, [D$_6$]DMSO): δ = 14.2 (s, 1 H), 8.76 (s, 1 H), 8.65 (d, J = 8.8 Hz, 2 H), 8.55 (s, 1 H), 8.41–8.39 (m, 3 H), 8.21 (d, J = 8.4 Hz, 2 H), 8.12 (d, J = 8.0 Hz, 2 H), 7.77–7.74 (m, 2 H), 7.67–7.63 (m, 2 H) ppm. ^{13}C NMR (100 MHz, [D$_6$]DMSO, 328 K): δ = 156.1, 132.0, 131.8, 130.5, 128.7, 128.5, 128.2, 127.5, 127.3, 125.8, 125.7, 125.2, 116.7, 115.2, 107.0, 100.0, 88.3 ppm. HRMS (FAB): calcd. for $[M + H]^+$ 445.1453; found 445.1456.

Compound 9: A procedure similar to that outlined for the synthesis of compound **1** was employed. The starting material was changed from 9,10-dibromoanthracene to compound **8**. The desired product was isolated as a dark purple solid (0.14 g, 41%). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 10.1 (s, 2 H), 9.98 (d, J = 8.0 Hz, 2 H), 8.87 (s, 2 H), 8.72 (d, J = 8.0 Hz, 2 H), 7.96 (d, J = 8.0 Hz, 4 H), 7.77 (d, J = 8.0 Hz, 4 H), 4.16 (m, 4 H), 2.00–1.96 (m, 4 H), 1.41–1.23 (m, 14 H), 0.97–0.84 (m, 12 H) ppm. ^{13}C NMR spectrum was not acquired due to low solubility. HRMS (MALDI): calcd. for $[M + H]^+$ 871.3747; found 871.3758.

Compound 10: A procedure similar to that outlined for the synthesis of compound **2** was employed. The starting material was changed from **1** to **8**. After removal of all the volatiles under vacuum, the residue was repeatedly washed with CH_2Cl_2 and ethyl acetate to afford a dark purple solid (0.25 g, 69%). M.p. 232–235 °C. The ^{1}H NMR and ^{13}C NMR spectra were not obtained due to extremely low solubility. HRMS (MALDI): calcd. for $[M + H]^+$ 1047.4601; found 1047.4598.

Compound 11: A procedure similar to that outlined for the synthesis of compound **3** was employed. The starting material was changed from 9,10-dibromoanthracene to compound **8**. The desired product was isolated as a dark purple solid (0.20 g, 44%). M.p. 196–199 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 10.1 (d, J = 8.0 Hz, 2 H), 8.91 (s, 2 H), 8.76 (d, J = 8.4 Hz, 2 H), 7.78–7.86 (m, 10 H), 7.42 (m, 2 H), 7.32 (m, 2 H), 4.27 (t, J = 8.0 Hz, 4 H), 4.17 (m, 4 H), 1.85–1.97 (m, 8 H), 1.20–1.43 (m, 50 H), 0.96 (t, J = 7.2 Hz, 6 H), 0.89 (t, J = 6.0 Hz, 6 H), 0.80 (t, J = 6.8 Hz, 6 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 163.4, 163.0, 152.2, 143.0, 137.6, 135.9, 133.7, 133.4, 132.2, 131.8, 130.4, 129.9, 127.5, 127.3, 127.2, 123.4, 123.2, 123.1, 122.8, 122.1, 120.1, 119.8, 110.4, 98.6, 92.6, 45.1, 44.5, 38.1, 32.0, 31.0, 30.0, 29.8, 29.7, 29.6, 29.5, 29.3, 28.9, 26.9, 24.3, 23.2, 22.8, 14.3, 14.2, 10.8 ppm. HRMS (FAB): calcd. for $[M + H]^+$ 1383.8354; found 1383.8367. $C_{94}H_{106}N_6O_4 \cdot H_2O$ (1401.90): C 80.53, H 7.76, N 5.99; found C 80.41, H 7.76, N 5.77.

Compound 12: A procedure similar to that outlined for the synthesis of compound **4** was employed. The starting material was changed from 9,10-dibromoanthracene to compound **8**. The desired product was isolated as a dark purple solid (30 mg, 39%). M.p. 261–264 °C. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 10.1 (d, J = 8.0 Hz, 2 H), 8.95 (s, 2 H), 8.78 (d, J = 8.4 Hz, 2 H), 8.24 (s, 2 H), 7.88 (s, 2 H), 7.86 (s, 4 H), 4.36 (t, J = 8.0 Hz, 4 H), 4.18 (m, 4 H), 1.86–1.97 (m, 8 H), 1.21–1.41 (m, 50 H), 0.96 (t, J = 7.2 Hz, 6 H), 0.89 (t, J = 7.2 Hz, 6 H), 0.80 (t, J = 6.4 Hz, 6 H) ppm. ^{13}C NMR (125 MHz, $CDCl_3$): δ = 163.8, 163.4, 158.1, 145.3, 137.9, 134.6, 134.3, 132.7, 131.0, 130.0, 129.8, 128.2, 127.9, 126.8, 125.4, 123.6, 122.7, 119.8, 117.0, 116.6, 116.5, 109.5, 109.2, 97.1, 93.5, 46.1, 44.7, 38.3, 32.1, 31.1, 30.3, 29.8, 29.7, 29.6, 29.5, 29.2, 29.0, 26.9, 24.4, 23.3, 22.9, 14.3, 14.3, 10.9 ppm. HRMS (MALDI): calcd. for $[M + H]^+$ 1484.9680; found 1484.8197.

CCDC-862005 (for **1**) contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Supporting Information (see footnote on the first page of this article): Synthetic procedures, HOMO and LUMO of compounds **9** and **10**, output characteristic and I-V transfer plots for compounds **9** and **12**, XRD spectra, ^{1}H NMR and ^{13}C NMR spectra.

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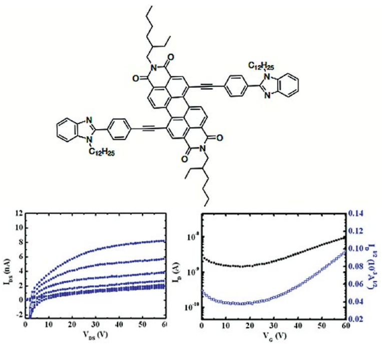
Optoelectronic Materials

The synthesis and photophysical studies of a series of anthracene and perylene bisimide derivatives containing an electron-deficient (ethynylphenyl)benzimidazole moiety are presented. Some of these molecules display n-channel semiconducting properties.



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Synthesis, Photophysical Properties, and Field-Effect Characteristics of (Ethynylphenyl)benzimidazole-Decorated Anthracene and Perylene Bisimide Derivatives

Keywords: Polycycles / Arenes / Conjugation / Photophysics / Semiconductors