Dialkyl-14*H*-benzo[4,5]isoquino[2,3-*a*]perimidin-14-one-3,4,10,11tetracarboxylic diimides: A New Family of *n*-Type Organic Semiconductors

Minliang Zhu,^[a, b] Ji Zhang,^[a, b] Gui Yu,^{*[a]} Huajie Chen,^[a, b] Jianyao Huang,^[a, b] and Yunqi Liu^{*[a]}

In recent years, significant research efforts have been devoted to developing high-performance organic semiconductors due to their potential applications in flexible and lowcost organic optoelectronic devices, such as organic lightemitting diodes, organic photovoltaic cells, organic fieldeffect transistors (OFETs), and organic complementary circuits.^[1] For realizing these applications, high-performance pand n-type semiconductors should be developed simultaneously. Although excellent p-type materials are widely available, the development of high-performance n-type materials is still a great challenge.

Arylenediimide derivatives are important n-type semiconductors for applications in organic optoelectronic devices.^[2] These materials have suitable energy levels of the lowest unoccupied molecular orbitals (LUMOs), linear molecular structures, and highly planar π -conjugated systems. Research on core-substituted arylenediimides has demonstrated that the introduction of electron-withdrawing groups into the π conjugated cores is an effective way to achieve excellent electron-transporting properties.^[3] Another strategy is to use a new conjugated core to design high-performance *n*-type materials. To date, however, arylenediimide derivatives with asymmetric cores have not been reported. Compared with perylene, the asymmetric 14H-benzo[4,5]isoquino[2,3-a]perimidin-14-one (BIPO) has a larger π -conjugated system and stronger electron-withdrawing ability; thus, it could exhibit a different electronic structure and may be a promising conjugated core to build novel arylenediimide derivatives. Additionally, two different N-substituents could readily be introduced into the diimide molecules to tune the molecular self-organization and intermolecular interactions.

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Based on these considerations, we designed and synthesized a series of asymmetric arylenediimide derivatives, dialkyl-14*H*-benzo[4,5]isoquino[2,3-*a*]perimidin-14-one-

3,4,10,11-tetracarboxylic diimides (**BIPO-DI**). The highly π conjugated *N*-heterocyclic planar **BIPO** core^[4] would likely have a variety of intermolecular interactions, such as van der Waals interactions and heteroatom–heteroatom interactions, which should facilitate charge carrier transport.^[5] Electron-deficient heterocyclic systems could also produce intermolecular hydrogen bond interactions and low LUMO energy levels.^[6] Herein, we studied the optical, electrochemical, and field-effect performances of the compounds **BIPO-DI**.

The route taken to synthesize the compounds **BIPO-DI** is shown in Scheme 1. 4-Nitro-1,8-naphthalic anhydride (1) and 4,5-dinitro-1,8-naphthalic anhydride (2) were obtained according to previous reports.^[7a,b] *N*-Octylnaphthalene-1,8dicarboxyanhydride-4,5-dicarboximide (7) was synthesized with a yield of 64% through the reaction of 1,4,5,8-naphthalenetetracarboxylic acid dianhydride with octan-1-amine in a mixture of water/propan-1-ol (1:1 ν/ν) under a nitrogen atmosphere.^[7e] Another intermediate, *N*-2-hexyldecylnaphtha-



Scheme 1. Synthetic routes for BIPO-DI.

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lene-1,8-dicarboxyanhydride-4,5-dicarboximide (8) was prepared through various methods. Using a reaction similar to the method described for 7 gave a fairly low yield of 8. When solvents such as ethanol, acetic acid, and N-methylpyrrolidone were used, only trace amounts of 8 were obtained. When 1,4,5,8-naphthalenetetracarboxylic acid dianhydride and 2-hexyldecan-1-amine were heated together in dimethylformamide (DMF), the corresponding diimide was obtained as the major product. When the solution of 2-hexyldecan-1-amine in DMF was added very slowly (about one drop every three seconds) into the solution of 1,4,5,8-naphthalenetetracarboxylic acid dianhydride in anhydrous DMF at 140°C, compound 8 was obtained with a yield of 36%. The obtained anhydride 7 or 8 was then condensed with diamine 5 or 6 in 1-butanol to give deep purple dialkyl-14Hbenzo[4,5]isoquino[2,3-a]perimidin-14-one-3,4,10,11-tetracarboxylic diimides (BIPO-DI). Using ethanol or acetic acid as the solvent in the reaction led to a decrease in the conversion. The three final products were purified by column chromatography on silica gel using dichloromethane/ethyl acetate (5:1 v/v) as an eluent and their chemical structures were determined by NMR spectroscopy, mass spectroscopy, and elemental analysis (see the Experimental Section and the Supporting Information). The three final compounds are readily soluble in common organic solvents such as chloroform, dichloromethane, and toluene.

Thermal gravimetric analyses (Figure S1 in the Supporting Information) show a 5% weight loss at 394, 414, and 435°C for DO-BIPO-DI, OHD-BIPO-DI, and DHD-BIPO-DI, respectively, thus verifying high thermal stabilities of the compounds. The absorption spectra of the three compounds in *n*-hexane and thin films are shown in Figure 1. The onsets of the UV/Vis absorption for the BIPO-DI in n-hexane are at about 690 nm, which is red shifted to those of pervlene tetracarboxylic diimides.^[8] They also constitute a 100 nm redshift to the **BIPO** core (14*H*-perimidino[2,1-a]benz[de]isoquinolin-14-one, also known as Solvent Red 179; absorption onset at 600 nm).^[4] The absorption spectra of the **BIPO-DI** show two pronounced peaks in the range of 530-580 nm and a shoulder around 630 nm. The three compounds with the different side-chains exhibit an obvious effect of substituents on the two pronounced absorption peaks. A difference in the absorption spectra caused by an alkyl chain has been reported for pervlene diimides.^[9,10] In the absorption spectra of the thin films, DO-BIPO-DI shows an obvious red shift compared with OHD-BIPO-DI and DHD-BIPO-DI, which indicates a stronger molecular interaction of DO-BIPO-DI molecules in the aggregated state. Compared with the spectra determined in solution, the three BIPO-DI derivatives show an obvious red shift in the absorption peaks as thin films. This result is generally consistent with an enhanced molecular interaction in the thin films.^[11] The observed spectral shift of the compound BIPO-DI in different solvents (Figure 2 and Figure S2 in the Supporting Information) is not directly dependent on the solvent polarity because some specific solvent-molecule interaction also occurs in different solvents.^[9,12] For the compound **DO-BIPO-DI**, the nonpolar solvent toluene shows a stronger interaction than the polar solvent ethanol. With regard to **DHD-BIPO-DI**, ethanol has



Figure 1. UV/Vis absorption spectra of the **BIPO-DI** compounds in (a) *n*-hexane solution (10^{-6} M) and (b) thin films.



Figure 2. UV/Vis absorption spectra of (a) **DO-BIPO-DI** and (b) **DHD-BIPO-DI** in different solvents (10^{-5} M) .

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Table 1. Reduction potentials and the LUMO/HOMO energy levels of the BIPO-DI.

Compound	$E_{\rm red1} [{\rm eV}]$	$E_{\rm red2} [{\rm eV}]$	$E_{\rm red1}^{\rm onset} [{\rm eV}]$	$E_{\rm HOMO} [{\rm eV}]$	$E_{\rm LUMO} [{\rm eV}]$	$E_{\rm gap} [{\rm eV}]$
DO-BIPO-DI	-0.83	-1.24	-0.55	-5.57	-3.85	1.72
OHD-BIPO-DI	-0.78	-1.21	-0.59	-5.55	-3.81	1.74
DHD-BIPO-DI	-0.82	-1.21	-0.60	-5.54	-3.80	1.74

an obvious polarization effect. As a branched alkyl chain has been introduced into the building block in this compound, the stronger vibration in the molecular backbone probably leads to a stronger interaction be-

tween the molecules and the polar solvent. Furthermore, steric hindrance due to the branched alkyl chain causes a weaker π -conjugated interaction between the **BIPO-DI** core and toluene. The red shift observed in toluene is relatively small for the compounds OHD-BIPO-DI and DHD-BIPO-DI, thus implying a weak interaction with the nonpolar solvent. The photoluminescent spectra of the three **BIPO-DI** compounds in CH₂Cl₂ have nearly the same shape under excitation at the absorption band of 350 nm (see Figure S3 in the Supporting Information). At the same time, DHD-BIPO-DI exhibits new emission peaks at 500 and 530 nm, which indicates a weak intermolecular interaction of this compound caused by strong steric hindrance.^[13] Using different excitation wavelengths ranging from 330 to 380 nm, the three **BIPO-DI** compounds display spectra of the same shape and same emission peaks, thereby indicating that the emission originated from the lowest excimer-like excited state.[13]

To investigate the redox properties of these new materials, cyclic voltammetry (CV) measurements were performed (Figure 3). The presence of the alkyl chain leads to a weak difference in the cyclic voltammogram, in agreement with a previous report.^[14] The potentials of the observed redox couples are listed in Table 1. For all molecules, multiple reversible reductions with no oxidations were observed, suggesting that all building blocks are substantially electron-dopable.^[15] The three compounds show almost the same onset redox potentials. The LUMO energy levels were calculated from the onset redox potentials ($E_{\rm red}^{\rm onset}$) according to the equation of $E_{\rm LUMO} = -e(E_{\rm red}^{\rm onset} + 4.4)$ eV. Their energy levels of the highest occupied molecular orbitals (HOMO) were estimated from the optical band gap and LUMO energy level to be -5.57 eV for **DO-BIPO-DI**,



Figure 3. Cyclic voltammograms of the compounds **BIPO-DI** in CH_2Cl_2 solution (2×10^{-5} M, Scan rate: 0.1 Vs⁻¹, electrolyte: 0.1 M TBAPF₆).

-5.55 eV for **OHD-BIPO-DI**, and -5.57 eV for **DHD-BIPO-DI** (Table 1).

To examine the application of these new semiconductors in OFETs, bottom-contact transistors were fabricated by vapor deposition of the compounds **BIPO-DI** on octadecyltrichlorosilane (OTS)-treated Si/SiO₂ substrates. OFET devices based on **BIPO-DI** exhibit typical *n*-channel FET characteristics (Table 2 and Figure 4). The compound **DO-BIPO-DI** exhibits an outstanding field-effect performance with a good mobility of $0.05 \text{ cm}^2 \text{V}^{-1} \text{s}^{-1}$ and a high current on/off ratio ($I_{\text{on}}/I_{\text{off}}$) of up to 10^8 . However, the OFETs based on compounds **OHD-BIPO-DI** and **DHD-BIPO-DI**

Table 2. FET characteristics of the ${\bf BIPO-DI}\xspace$ devices fabricated on OTS-treated Si/SiO2 substrates.

Compound	$\mu \ (\mathrm{cm}^2 \mathrm{V}^{-1} \mathrm{s}^{-1})^{[\mathrm{a}]}$	$I_{\rm on}/I_{\rm off}$	$V_{ m TH}[{ m V}]$
DO-BIPO-DI	0.05	10 ⁸	36
OHD-BIPO-DI	1×10^{-4}	10^{5}	13
DHD-BIPO-DI	5×10^{-3}	107	17

[a] μ = electron mobility.



Figure 4. (a) Output and (b) transfer characteristic of the **DO-BIPO-DI**based OFET devices.

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exhibit a relatively low mobility of 10^{-4} cm²V⁻¹s⁻¹ with I_{on}/I_{off} of 10^5 and a mobility of 5×10^{-3} cm²V⁻¹s⁻¹ with I_{on}/I_{off} of 10^7 , respectively. Thus, the mobility of **DO-BIPO-DI** is 2–3 orders of magnitude higher compared to those of **OHD-BIPO-DI** and **OHD-BIPO-DI**. This could be explained by a stronger molecular interaction and a better molecular orientation of **DO-BIPO-DI** in the solid state (see AFM images in Figure 5). X-ray diffraction (XRD) data provide evidence of a better molecular orientation of the compound **DO-BIPO-DI** (Figure 6).

In summary, we developed a new family of asymmetric arylenediimides, namely dialkyl-14*H*-benzo[4,5]isoquino[2,3-a]perimidin-14-one-3,4,10,11-tetracarboxylic diimides. These compounds can be readily synthesized and structurally modified by appending different groups to the N atoms of imides. The thermal properties and electronic performance



Figure 5. Atomic force microscopy (AFM) images of the compounds on OTS-treated SiO_2 . (a) **DO-BIPO-DI**, (b) **OHD-BIPO-DI**, and (c) **DHD-BIPO-DI**.



Figure 6. XRD data of the compounds on OTS-treated SiO₂.

could be tuned through the incorporation of alkyl groups. Imide groups were found to be effective to tune energy levels and give rise to an *n*-type performance. The **DO-BIPO-DI**-based OFET devices show a good field-effect performance with an electron mobility of $0.05 \text{ cm}^2 \text{V}^{-1} \text{s}^{-1}$ and a large current on/off ratio of 10^8 .

Experimental Section

Synthesis of DO-BIPO-DI

Compounds 5 (1 g, 2.94 mmol) and 7 (1.1 g, 2.94 mmol) were dissolved in 1-butanol (80 mL). The reaction mixture was heated for 12 h at $150 \text{ }^{\circ}\text{C}$

under an N₂ atmosphere. Subsequently, the solvent was evaporated and the residue was purified by column chromatography on silica gel using a mixture of dichloromethane/ethyl acetate (5:1 ν/ν) as an eluent. Finally, the product was recrystallized from ethanol/chloroform (3:1 ν/ν) to give a purple solid (910 mg, 50% yield). M.p. 270–273 °C; ¹H NMR (CDCl₃, 400 MHz): $\delta = 0.87$ (m, 6H), 1.28–1.43 (m, 20H), 1.75 (m, 4H), 4.20 (m, 4H), 7.65 (d, J = 8.3 Hz, 1H), 8.63 (d, J = 8.3 Hz, 2H), 8.80–8.83 (m, 3H), 9.05 (d, J = 8.3 Hz, 1H), 9.12 ppm (d, J = 8.3 Hz, 1H); ¹³C NMR (CDCl₃, 75 MHz): $\delta = 14.90$, 23.38, 27.86, 27.97, 28.93, 29.79, 29.85, 29.91, 29.98, 32.43, 41.40, 41.50, 115.14, 119.89, 120.38, 122.90, 122.97, 124.45, 124.79, 125.58, 126.23, 126.28, 126.49, 126.62, 127.64, 127.79, 129.19, 129.34, 131.64, 132.50, 136.47, 143.16, 158.48, 160.73, 160.82, 160.90, 161.03 ppm; MS (MALDI-TOF) *m*/*z* [*M*⁺] calcd. for C₄₂H₄₂N₄O₅: C 73.88, H 6.2, N 8.21; found: C 74.14, H 6.02, N 8.17.

Synthesis of OHD-BIPO-DI

OHD-BIPO-DI was synthesized according to the procedure described for DO-BIPO-DI using 5 (1 g, 2.94 mmol), 8 (1.45 g, 2.94 mmol), and 1butanol (80 mL). The crude product was purified by column chromatography on silica gel using a mixture of dichloromethane/ethyl acetate (5:1 v/v) as an eluent to afford OHD-BIPO-DI as a yellow solid (800 mg, 34% yield). M.p. 241–244°C; ¹H NMR (CDCl₃, 400 MHz): $\delta = 0.86$ (m, 9H), 1.28-1.45 (m, 34H), 1.75 (m, 2H), 1.99 (m, 1H), 4.17 (m, 4H), 7.62 (d, J=8.0 Hz, 1 H), 8.59 (m, 2 H), 8.75-8.80 (m, 3 H), 9.03 (d, J=10.4 Hz, 1 H), 9.14 ppm (d, J = 10.4 Hz, 1 H); ¹³C NMR (CDCl₃, 75 MHz): $\delta =$ 14.13, 22.68, 26.44, 26.45, 27.23, 28.03, 29.29, 29.34, 29.39, 29.61, 29.75, 30.07, 31.70, 31.88, 31.91, 36.73, 40.61, 45.04, 115.90, 118.46, 118.55, 118.99, 121.35, 125.26, 125.85, 126.53, 126.58, 126.95, 127.68, 128.47, 129.33, 130.78, 131.02, 132.90, 133.43, 137.63, 142.70, 146.90, 161.06, 162.84, 162.93, 162.99, 163.23 ppm; MS (MALDI-TOF) m/z [M^+] calcd. for C50H58N4O5: 794.4; found, 794.4; elemental analysis (%) calcd for C₅₀H₅₈N₄O₅: C 75.54, H 7.35, N 7.05; found: C 75.82, H 7.25, N 6.99.

Synthesis of DHD-BIPO-DI

DHD-BIPO-DI was synthesized according to the procedure described for **BIPO-DI** using **6** (1 g, 2.22 mmol), **8** (1.1 g, 2.22 mmol), and 1-butanol (80 mL). The crude product was purified by column chromatography on silica gel using a mixture of dichloromethane/ethyl acetate (5:1 ν/ν) as an eluent to yield **DHD-BIPO-DI** as a yellow solid (710 mg, 35% yield). M.p. 140–143°C; ¹H NMR (CDCl₃, 400 MHz): δ =0.84 (m, 12H), 1.24–1.40 (m, 48H), 2.00 (m, 2H), 4.12 (m, 2H), 7.63 (d, *J*=8.0 Hz, 1H), 8.59 (m, 2H), 8.77–8.81 (m, 3H), 9.03 (d, *J*=10.4 Hz, 1H), 9.14 ppm (d, *J*=10.4 Hz, 1H); ¹³C NMR (CDCl₃, 75 MHz): δ =14.30, 22.69, 26.44, 26.47, 26.54, 27.44, 29.34, 29.58, 29.61, 29.71, 29.74, 29.78, 30.02, 30.07, 31.71, 31.91, 36.66, 116.03, 118.54, 118.80, 119.09, 126.04, 126.74, 127.03, 127.96, 128.69, 130.99, 131.16, 133.07, 142.91, 161.21, 163.13, 163.16, 163.49, 163.78 ppm; MS (MALDI-TOF) *m*/z [*M*⁺] calcd. for C₅₈H₇₄N₄O₅: Offset offset offset off offset offs

Fabrication of OFET Devices

OFET devices with a bottom-gate/bottom-contact configuration were fabricated on a highly n-doped silicon wafer with a thermally oxidized 300 nm-thick SiO₂ as a gate dielectric layer. Source and drain electrodes made of gold were pre-prepared on the SiO2/Si substrates. The channel length and width are 0.04 and 1.4 mm, respectively. Before the deposition of organic semiconductors, OTS treatment was performed on the SiO₂ gate dielectrics under vacuum to form an OTS self-assembled monolayer. Organic thin films were then deposited onto the OTS-modified SiO₂ surface under vacuum. AFM measurements were carried out using a Nanoscope V instrument. XRD of thin films was performed in the reflection mode at 40 kV and 200 mA with CuKa radiation using a 2 kW Rigaku Xray diffractometer. The electrical characteristics of the OFET devices were recorded at room temperature inside a glove box filled with nitrogen gas by using a Keithley 4200 SCS semiconductor parameter analyzer. The mobility of the devices was calculated in the saturation regime using the following equation:

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 $I_{\rm DS} = (W/2L) C_{\rm i} \mu (V_{\rm GS} - V_{\rm TH})^2$

where W and L denote the channel width and length, respectively, C_i is the insulator capacitance per unit area, and $V_{\rm GS}$ and $V_{\rm TH}$ are the gate voltage and threshold voltage, respectively.

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Organic Semiconductors

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Dialkyl-14*H*-benzo[4,5]isoquino[2,3*a*]perimidin-14-one-3,4,10,11-tetracarboxylic diimides: A New Family of *n*-Type Organic Semiconductors



Starting a family: A novel family of asymmetric arylenediimides, dialkyl-14*H*-benzo[4,5]isoquino[2,3-*a*]perimidin-14-one-3,4,10,11-tetracarboxylic diimides (**BIPO-DI**), was successfully designed and synthesized. The effects of different alkyl substitution on the structures were characterized by UV/ Vis absorption spectrocopy and cyclic voltammetry. Field-effect transistors based on di-*n*-octyl **BIPO-DI** thin films show an electron mobility of $0.05 \text{ cm}^2 \text{V}^{-1} \text{s}^{-1}$ with a high current on/ off ratio of 10^8 .

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