DOI: 10.1002/chem.201000026

Synthesis, Characterization, and Bipolar Transporting Behavior of a New Twisted Polycyclic Aromatic Hydrocarbon: 1',4'-Diphenyl-naphtho-(2'.3':1.2)pyrene-6'-nitro-7'-methyl Carboxylate

Qichun Zhang,^{*[a]} Yoga Divayana,^[b] Jinchong Xiao,^[a] Zhijuan Wang,^[a] Edward R. T. Tiekink,^[a, e] Hieu M. Doung,^[c] Hua Zhang,^[a] Freddy Boey,^[a] Xiao Wei Sun,^{*[b]} and Fred Wudl^{*[c, d]}

Abstract: An asymmetric twistacene, 1',4'-diphenyl-naphtho-(2'.3':1.2)-pyrene-6'nitro-7'-methyl carboxylate (tetracene **2**), was synthesized by using benzyne-trapping chemistry. Its structure, determined by X-ray crystallography, confirmed that this material has a twisted topology with torsion angles as high as 23.8(3)°. Organic light-emitting devices using tetracene **2** as either charge-transporting materials or emitters have been fabricated. The results indicate that this material has bipolar transporting behavior in these devices.

Keywords: arenes • light-emitting device • polycycles • polycyclic aromatic hydrocarbons • semiconductors • synthesis design

Introduction

Polycyclic aromatic hydrocarbons (PAHs),^[1–3] also known as "pieces of graphene", have generated strong interest both in fundamental science underlying synthetic challenge, structure–property correlations, and stacking features in solid states as well as in potential technological applications^[4–6] such as light-emitting diodes, field-effect transistors, and photovoltaic devices.^[7–9] In addition, these materials are amenable to the fine tuning of their electrical and optical properties by proper molecular design. One member of the PAH family, pyrene (**1**), is of particular interest because it



has the fundamental framework found in many PAHs, carbon nanotubes, graphenes, and polycyclic heteroaromatics. Accordingly, **1** has been widely employed as a molecular scaffold to construct larger PAHs and heteroaromatics. Recent reports have already demonstrated that pyrene-

- [a] Prof. Q. Zhang, Dr. J. Xiao, Dr. Z. Wang, Prof. E. R. T. Tiekink, Prof. H. Zhang, Prof. F. Boey School of Materials Science Engineering Nanyang Technological University 50 Nanyang Avenue, Singapore 639798 (Singapore) Fax: (+65) 67909081 E-mail: qczhang@ntu.edu.sg
 [b] Dr. Y. Divayana, Prof. X. W. Sun
- School of Electronical and Electronic Engineering Nanyang Technological University 50 Nanyang Avenue, Singapore 639798 (Singapore) Fax: (+65)67933318 E-mail: exwsun@ntu.edu.sg
- [c] Dr. H. M. Doung, Prof. F. Wudl Department of Chemistry and Biochemistry University of California, Los Angeles, CA 90095 (USA)
- [d] Prof. F. Wudl
 Mitsubishi Chemical Centre for Advanced Materials
 Department of Chemistry and Biochemistry and
 Centre for Polymers and Organic Solids
 University of California, Santa Barbara, CA 93106 (USA)
 Fax: (+1)805-8934120
 E-mail: wudl@chem.ucsb.edu
- [e] Prof. E. R. T. Tiekink Current Address: Department of Chemistry University of Malaya, 50603 Kuala Lumpur (Malaysia)
- Supporting information for this article is available on the WWW under http://dx.doi.org/10.1002/chem.201000026.



© 2010 Wiley-VCH Verlag GmbH & Co. KGaA, Weinheim

Chem. Eur. J. 2010, 16, 7422-7426

based materials possess excellent photoluminescence efficiency, high carrier mobility, and enhanced hole-injecting ability in organic light-emitting devices (OLEDs).^[10-12]

A key limitation for PAHs application in OLEDs is their concentration-quenching effect due to strong π -stacking interactions.^[13] One possible solution is to twist the PAHs, creating a more sterically hindered π system and, in effect, decrease $\pi \cdots \pi$ attractions. These topologically interesting PAHs systems have been studied in great detail by Pascal.^[14] The basic strategy used to twist PAHs is to introduce bulky substituents on their conjugated framework. Interestingly, twisting PAHs has little effect on their characteristic spectroscopic and electronic properties when compared to their unsubstituted planar parent structures. Therefore, these twisted π systems provide an attractive molecular toolset for application in OLEDs. In fact, an efficient white-light-emitting diode based on a novel "twistacene" has been demonstrated by Wudl and Yang.^[15] Herein, we reported the synthesis of a novel twistacene homologue and its bipolar electronic properties.

Results and Discussion

Synthesis of materials: 1',4'-Diphenyl-naphtho-(2',3':1,2)pyrene-6'-nitro-7'-methyl carboxylate (tetracene 2) was prepared through benzyne-trapping chemistry of bisbenzyne precursor 4 by using dienenone 3 (Scheme 1). The single-



Scheme 1. The synthetic route to tetracene 2.

crystal X-ray structural analysis of tetracene 2 shows the anticipated twisted topology in the dibenzotetracene PAH scaffold. Taking advantage of this twist feature, we fabricated several OLEDs using tetracene 2 to study its electronic transport properties.

Tetracene 2 was prepared through a [4+2] cycloaddition involving in-situ aryne (benzyne formation) as a dienophile and pyrano-diphenylcyclopentadienone (3) as a diene (Scheme 1). The intermediate benzyne precursor 4 was synthesized from a commercially available aminoterephthalic dimethyl ester in four steps. To avoid the nucleophilic displacement of the nitro group by hydroxyl group, the benzyne precursor 4 was introduced slowly as a 1,2-dichloroethane (DCE) suspension to an awaiting cyclopentadieneone 3 with isoamyl nitrite in refluxing DCE. These conditions afforded the target tetracene 2 in 45% without any evidence of phenol formation. **Crystal structure**: The molecular structure of tetracene **2** with atomic labeling is illustrated in Figure 1 (top) and crystal data are given in Table $1.^{[16a]}$ Immediately evident from the side-on view of the crystal packing of tetracene **2**



Figure 1. Top: Molecular structure of tetracene **2** showing the crystallographic numbering scheme. Bottom: View along the C_{phenyl} -C9···C12– C_{phenyl} axis highlighting the buckled nature of the carbon atom framework and the twists in the terminal phenyl, -C(=O)OMe, and -NO₂ residues.

Table 1. Crystal data and structure refinement for tetracene 2.

5	
formula	$C_{38}H_{23}NO_4$
$M_{\rm r}$	557.60
<i>T</i> [K]	250
λ [Å]	0.71073
crystal system	triclinic
space group	$P\bar{1}$
a [Å]	9.0847(2)
b [Å]	11.5552(2)
c [Å]	13.9742(3)
	78.850(1)
β[°]	73.090(1)
γ [°]	84.950(1)
$V[Å^3]$	1376.24(5)
Z	2
$\rho_{\rm calcd} [{\rm gcm^{-3}}]$	1.346
$\mu [\mathrm{mm}^{-1}]$	0.087
F(000)	580
crystal size [mm]	$0.30 \times 0.23 \times 0.22$
2θ range [°]	3.6-51.8
index ranges	$-10 \le h \le 10$
	$-13 \le k \le 13$
	$-15 \le l \le 16$
completeness	98.8 %
max/min transmission	0.990/0.9550
final R indices $[I > 2\sigma(I)]$	R1 = 0.047, wR2 = 0.120
R indices (all data)	R1 = 0.069, wR2 = 0.137

www.chemeurj.org

(Figure 1, bottom) is the presence of significant twist to the dibenzotetracene π system in the molecule. There is also significant puckering in each of the three endocyclic six-membered rings. Thus, the "hinge" atoms C6, C11, and C10 have the greatest deviations of 0.053(2) (C3-C8 ring), 0.107(2) (C6, C7, C9–C12) and 0.132(2) Å (C10, C11, C25, C30–C32) from their respective least-squares planes. Similar distortions, although not as great, are evident in the 24-atom tricyclic systems, that is, the C25 (C25-C30 ring), C31 (C29-C31-C36-C38) and C31 (C31-C36) atoms lie 0.026(2), 0.034(2) and -0.041(2) Å out of their respective least-squares plane. A quantitative measure of the twisting in the molecule is found in the magnitudes of the torsion angles involving the C6-C7, C10-C11, and C30-C31 "hinges", which vary from a low of 6.7(3)° for the C25-C30-C31-C32 torsion angle to a high of 23.8(3)° for C32-C10-C11-C25, indicating the greatest twist occurs around the C10-C11 bond. The molecule adopts a distinctively curved motif, which is aptly quantified in terms of the dihedral angle formed between the terminal C3-C8 and C29-C31/C36-C38 six-membered rings of 35.23(11)°.

The ester and the terminal phenyl rings in tetracene **2** is twisted out of the plane of the central fused ring system as manifested in the C6-C12-C13-C14 and C7-C9-C19-C24 torsion angles of -66.9(3) and $-71.3(2)^\circ$, respectively. As seen from Figure 1 (bottom), the phenyl rings are orientated in opposite directions. Each of the ester and nitro groups are twisted out of the plane of the aromatic ring to which they are connected as seen in the O1-C2-C3-C4 and O3-N1-C4-C5 torsion angles of 47.8(3) and 30.2(3)°, respectively.

The most prominent intermolecular interactions stabilizing the crystal packing are of the type C–H···O and C– H··· π . As anticipated, the closet interactions in the solid state are greater than 4.0 Å due to the twisting of the macromolecular framework. The C–H···O contact occurs between the phenyl-C18-H and the carbonyl-O1^[16b] to form supramolecular chains along the *a* direction (Figure 2, top). The C–H··· π contacts occur between centrosymmetrically related molecules through the phenyl-C14-H and the ring centroid of the C25–C30 ring to form dimeric aggregates (Figure 2, bottom) with the consequence that the supramolecular chains mediated by the C–H···O contacts are linked into double chains.^[16c]

The UV/Vis absorption and fluorescence spectrum of tetracene **2** were recorded at room temperature in methylene chloride (DCM) solvent. As shown in Figure 3, tetracene **2** $(1 \times 10^{-5} \text{ M})$ displays three absorption bands at 294, 347, and 366 nm. It exhibits the emission maxima at 564 nm with excitation at 366 nm. The spectra of **2** thin film prepared on glass slice showed broad absorption from 370 to 440 nm (Figure S1 in the Supporting Information). Fluorescence spectra of **2** film on glass slice displayed the bands at 467 and 527 nm. The quantum yield (Φ_f) of tetracene **2** in DCM was measured as $\Phi_f = 45\%$ (9,10-diphenylanthrance as standard (100%) at 25°C).

To test the carrier-transport capability of tetracene **2**, three different organic light-emitting diode (OLED) devices



Figure 2. Top: Supramolecular chain formation through C–H···O interactions (shown as dashed lines). Bottom: Loose association between centrosymmetric molecules mediated by C–H··· π interactions (dashed lines).



Figure 3. UV/Vis and fluorescence spectra of tetracene 2 in DCM.

were fabricated: 1) ITO/NPB (50 nm)/2(50 nm)/Mg:Ag, 2) ITO/2(50 nm)/Alq₃ (50 nm)/Mg:Ag, and 3) ITO/NPB (50 nm)/Alq₃ (50 nm/Mg:Ag (standard device), in which ITO is indium-tin-oxide; NPB is N,N'-bis(naphthalen-1-yl)-N,N'-bis(phenyl)benzidine, a common hole-transporting layer, and Alq₃ is tris(8-hydroxy-quinolinato) aluminium, a common electron-transporting layer. In device 1, tetracene 2 acts as the electron-transporting layer, while it becomes hole-transporting carrier in device 2. Figure 4 (top) shows the current density for the corresponding devices. It can be seen that there is no significant difference between devices 1 and 2, which shows that tetracene 2 transports both electron and hole equally well. However, compared to the conventional carrier transporting materials (device 3), tetracene 2 shows a relatively low carrier-transport capability. Inset of Figure 4 (top) shows the emission spectrum of the corresponding devices. Emission from device 1 comes from tetracene 2, while the emission from device 2 is very similar to found for standard device 3, which comes from Alq₃. In terms of external quantum efficiency (EQE), both devices that utilize tetracene 2 as the carrier-transporting layer show poor performance. The standard device reaches EQE of



Figure 4. Top: Current density of OLED devices with tetracene 2 as the electron-transporting (black squares) and hole-transporting (red circles) layers. Data for the standard device are shown for comparison (blue triangles). Inset shows the corresponding emission spectra. Bottom: Current density and luminance of devices with tetracene 2 doped in CBP with concentrations of 2% (dark squares) and 20% (orange circles). Insets i) and ii) show the current efficiencies and the emission spectra, respectively, of OLED devices with tetracene 2 utilized as dopant with different concentrations.

0.8% photons per electron, while devices 1 and 2 reach EQEs of only 0.026% and 0.018%, respectively. The low EQE may be improved by replacing the two electron-with-drawing groups (NO₂ and COOMe) with electron-donating groups, research is currently on the way.

Tetracene 2 has also been investigated as an emitter in OLEDs. Generally, a small amount of tetracene 2 was doped in a host-guest system (e.g., CBP). OLEDs with structure of ITO/NPB (50 nm)/CBP:2 (x % wt)/BCP (20 nm)/Alq₃ (20 nm)/Mg:Ag have been fabricated. Here, CBP is 4,4'-bis(carbazol-9-yl)biphenyl, a common host material and BCP is 2,9-dimethyl-4,7-diphenyl-1,10-phenanthroline, a common hole-blocking layer. The doping concentrations (x%) of tetracene 2 were 2 and 20%. Figure 4 (bottom) shows current density and luminance versus voltage of the corresponding devices. It can be seen that the voltage increases as the dopant concentration increases, which indicates that tetracene 2 traps the charges significantly. Inset i) of Figure 4 (bottom) shows the current efficiency as a function of current density of the devices. Devices with dopant concentration of 2 and 20% reach efficiency of 0.47 and 0.38 Cd A⁻¹, respectively. The emission spectrum of the corresponding devices is shown in inset ii) in the

FULL PAPER

bottom part of Figure 4b. It can be seen that the emission peak red-shifted as the doping concentration increases. The peak-shift is followed with a small drop in the EQE. It is worth mentioning that the EQE is significantly larger than that of device 1, which utilizes bulk tetracene 2 as the emitter. Utilizing tetracene 2 as a dopant proves to reduce the aggregation effect and increases the EQE.

Conclusion

In conclusion, a novel and stable tetracene 2 has been successfully synthesized and fully characterized. The twisted feature renders this type of PAH as a potential candidate for organic electronics. Although the performance of devices is not as good as we predicted, we have demonstrated the bipolar transporting ability of 2 in OLEDs. Further work to construct highly conjugated twisted compounds is under investigation.

Experimental Section

Materials: All starting materials were used as received without further purification. Compounds pyrano-diphenylcyclopentadienone (**3**) was synthesized according to the reported procedure.^[17,18]

X-ray crystal structure: Single-crystal data set was collected at 250 K on a Bruker SMART APEX II CCD fitted with graphite monochromatized $M_{O_{K\alpha}}$ radiation (λ =0.71073 Å). Data processing (APEXII^[19] and SMART^[19]) and absorption correction (SADABS^[20]) were accomplished by standard methods. The structure was solved by direct-methods using SHELXS-97^[21] and refinement (anisotropic displacement parameters, hydrogen atoms in the riding model approximation and a weighting scheme of the form $w=1/[\sigma^2(F_o^2)+(0.064P)^2+0.376P]$ for $P=(F_o^2+2F_o^2)/3$) was on F^2 by means of SHELXL-97.^[184] The crystal was weakly diffracting, a feature exacerbated by the crystal shattering at significantly reduced temperatures, and although data was collected to higher values, the crystal diffracted only to θ_{max} of 24.9°. Figure 1 was drawn with ORTEP^[22] at the 35% probability level and the remaining crystallographic figures were drawn with DIAMOND using arbitrary spheres.^[23]

Fabrication and evaluation of OLEDs: The ITO (indium-tin-oxide)-glass was cleaned by using a routine cleaning procedure, which include ultrasonication in acetone, followed by ethanol, and rinsing in de-ionized water. A clean ITO glass exhibited resistance on the order of 50 Ω per square. Before deposition, the ITO was treated by oxygen plasma at 10 Pa for 2.5 min. Evaporation of organic materials and metals was carried out under high vacuum conditions (about 2×10^{-4} Pa). Electroluminescence spectra of the fabricated devices were measured with a PR650 Spectra Scan spectrometer. Current-density/voltage characteristics were recorded by Yokogawa source measurement unit. All measurements were carried out at room temperature under ambient conditions without any encapsulation.

Acknowledgements

Q.Z. thanks the financial support from the NTU start-up grant, X.W.S. and Y.D. thanks the Singapore Millennium Foundation (SMF) for the financial support, and F.W. thanks the NSF for support.

www.chemeurj.org

CHEMISTRY

- [1] a) Y. Geerts, G. Klärner, K. Müllen in *Electronic Materials: The Oligomer Approach* (Eds.: K. Müllen, G. Wagner), Wiley-VCH, Weinheim, **1998**; Chapter 1; b) R. G. Havey, *Polycyclic Aromatic Hydrocarbons*, Wiley-VCH, New York, **1997**.
- [2] a) E. Clar, Polycyclic Hydrocarbons, Vol. 1 and 2, Academic Press, London, 1964; b) E. Clar, The Aromatic Sexet, Wiley, London, 1972;
 c) H. Hosoya, Top. Curr. Chem. 1990, 153, 255; d) M. Zander, Polycyclische Aromaten: Kohlenstoffe und Fullerene, Teubner, Stuttgart, 1995.
- [3] For recent reviews: a) R. Gleiter, B. Esser, S. C. Kornmayer, Acc. Chem. Res. 2009, 42, 1108; b) J. E. Anthony, Angew. Chem. 2008, 120, 460; Angew. Chem. Int. Ed. 2008, 47, 452; c) S. Kotha, S. Misra, S. Halder, Tetrahedron 2008, 64, 10775; d) R. A. Pascal, Jr., Chem. Rev. 2006, 106, 4809; e) Y. T. Wu, J. S. Siegel, Chem. Rev. 2006, 106, 4843; f) J. E. Anthony, Chem. Rev. 2006, 106, 5028; g) M. Bendikov, F. Wudl, D. F. Perepichka, Chem. Rev. 2004, 104, 4891.
- [4] a) D. Wasserfallen, M. Kastler, W. Pisula, W. A. Hofer, Y. Fogel, Z. Wang, K. Müllen, J. Am. Chem. Soc. 2006, 128, 1334; b) M. Kastler, W. Pisula, D. Wasserfallen, T. Pakula, K. Müllen, J. Am. Chem. Soc. 2005, 127, 4286; c) D. W. Breiby, O. Bunk, W. Pisula, T. I. Solling, A. Tracz, T. Pakula, K. Müllen, M. M. Nielsen, J. Am. Chem. Soc. 2005, 127, 11288; d) W. Pisula, Z. Tomovic, C. Simpson, M. Kastler, T. Pakula, K. Müllen, Chem. Mater. 2005, 17, 4296; e) W. Pisula, Z. Tomovic, M. Stepputata, U. Kolb, T. Pakula, K. Müllen, Chem. Mater. 2005, 17, 2641.
- [5] a) Y. Yang, F. Wudl, Adv. Mater. 2009, 21, 1401; b) J. Cornil, J. L. Brédas, J. Zaumseil, H. Sirringhaus, Adv. Mater. 2007, 19, 1791; c) J. Wu, Curr. Org. Chem. 2007, 11, 1220; d) Y. H. Wijsboom, A. Patra, S. S. Ade, Y. Sheynin, M. Li, L. J. W. Shimon, M. Bendikov, Angew. Chem. 2009, 121, 5551; Angew. Chem. Int. Ed. 2009, 48, 5443; e) A. Patra, Y. H. Wijsboom, S. S. Zade, M. Li, Y. Sheynin, G. Leitus, M. Bendikov, J. Am. Chem. Soc. 2008, 130, 6734.
- [6] a) L. B. Roberson, J. Kowalik, L. M. Tolbert, C. Kloc, R. Zeis, X. Chi, R. Flemming, C. Wilkins, J. Am. Chem. Soc. 2005, 127, 3069;
 b) V. De Halleux, J. P. Calbert, P. Brocorens, J. Cornil, J. P. Declercq, J. L. Brédas, Y. Geerts, Adv. Funct. Mater. 2004, 14, 649; c) L. Schmidt-Mende, A. Fenchtenkötter, K. Müllen, E. Moons, R. H. Friend, J. D. MacKenzie, Science 2001, 293, 1119; d) C. J. Brabec, N. S. Sariciftci, J. C. Hummelen, Adv. Funct. Mater. 2001, 11, 15;
 e) A. M. Van de Craats, J. M. Warman, Adv. Mater. 2001, 13, 130;
 f) C. Y. Liu, A. Bard, J. Chemother. 2000, 12, 2353.
- [7] a) M. L. Tang, S. C. B. Mannsfeld, Y. S. Sun, H. A. Becerril, Z. Bao, J. Am. Chem. Soc. 2009, 131, 882; b) M. L. Tang, A. D. Reichardt, T. Siegrist, S. C. B. Stefan, Z. Bao, Chem. Mater. 2008, 20, 4669; c) A. Dadvand, F. Cicoira, C. Fabio, Y. Konstantin, E. S. Balenkova, R. M. Osuna, F. Rosei, V. G. Nenajdenko, D. F. Perapichka, Chem. Commun. 2008, 5354; d) M. M. Payne, S. R. Parkin, R. Sean, J. E. Anthony, J. Am. Chem. Soc. 2005, 127, 8028; e) Z. Bao, Adv. Mater. 2000, 12, 227.
- [8] a) D. Chun, Y. Cheng, F. Wudl, Angew. Chem. 2008, 120, 8508; Angew. Chem. Int. Ed. 2008, 47, 8380; b) H. Wang, C. Schaffner-Hamann, F. Marchioni, F. Wudl, Adv. Mater. 2007, 19, 558.
- [9] A. S. Paraskar, A. R. Reddy, A. Patra, Y. H. Wijsboom, O. Gidron, L. J. W. Shimon, G. Leitus, M. Bendikov, *Chem. Eur. J.* **2008**, *14*, 10639.

- [10] a) F. Liu, L. H. Xie, C. Tang, J. Liang, Q. Q. Chen, B. Peng, W. Wei, Y. Cao, W. Huang, Org. Lett. 2009, 11, 3850; b) L. H. Xie, X. Y. Hou, Y. R. Hua, Y. Q. Huang, B. M. Zhao, F. Liu, B. Peng, W. Wei, W. Huang, Org. Lett. 2007, 9, 1619; c) C. Tang, F. Liu, Y. J. Xia, L. H. Xie, A. Wei, S. B. Li, Q. L. Fan, W. Huang, J. Mater. Chem. 2006, 16, 4074.
- [11] a) L. Turker, A. Tapan, S. Gümüs, *Polycyclic Aromat. Compd.* 2009, 29, 139; b) T. Otsubo, Y. Aso, K. J. Takimiya, *J. Mater. Chem.* 2002, 12, 2565; c) P. C. Bevilacqua, R. Kierzek, K. A. Johnson, D. H. Turner, *Science* 1992, 258, 1355.
- [12] a) H. Y. Oh, C. Lee, S. Lee, Org. Electronics 2009, 10, 163; b) U. Mitschke, P. Bauerle, J. Mater. Chem. 2000, 10, 1471.
- [13] a) M. Y. Lai, C. H. Chen, W. S. Huang, J. T. Lin, T. H. Ke, L. Y. Chen, M. H. Tsai, C. C. Wu, *Angew. Chem.* 2008, *120*, 591; *Angew. Chem. Int. Ed.* 2008, *47*, 581; b) Z. Ge, T. Hayakawa, S. Ando, M. Ueda, T. Akiike, H. Miyamoto, T. Kajita, M. Kakimoto, *Adv. Funct. Mater.* 2008, *18*, 584; c) Z. Ge, T. Hayakawa, S. Ando, M. Ueda, T. Akiike, H. Miyamoto, T. Kajita, M. Kakimoto, *Chem. Mater.* 2008, *20*, 2532.
- [14] a) J. Lu, D. M. Ho, N. J. Vogelaar, C. M. Kraml, S. Bernhard, N. Byrne, L. R. Kim, R. A. Pascal, Jr., J. Am. Chem. Soc. 2006, 128, 17043; b) J. Lu, D. M. Ho, N. J. Vogelaar, C. M. Kraml, R. A. Pascal, Jr., J. Am. Chem. Soc. 2004, 126, 11168; c) I. I. Schuster, L. Cracium, D. M. Ho, R. A. Pascal, Jr., Tetrahedron 2002, 58, 8875; d) J. Zhang, D. M. Ho, R. A. Pascal, Jr., Tetrahedron Lett. 1999, 40, 3859; e) X. Qiao, D. M. Ho, R. A. Pascal, Jr., Angew. Chem. 1997, 109, 1588; Angew. Chem. Int. Ed. Engl. 1997, 36, 1531.
- [15] a) Q. Xu, H. M. Duong, F. Wudl, Y. Yang, *Appl. Phys. Lett.* 2004, 85, 3357; b) H. M. Duong, M. Bendikov, D. Steiger, Q. Zhang, G. Sonmez, J. Yamada, F. Wudl, *Org. Lett.* 2003, 5, 4433.
- [16] a) CCDC-744864 (2) 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. Details of the intermolecular interactions; b) C18–H18···O1ⁱ=2.34 Å, C18···O1ⁱ=3.260(3) Å, with an angle subtended at H18 of 166° for symmetry operation i: 1+*x*, *y*, *z*; c) C14–H14···Cg(C25–C30)ⁱⁱ=2.72 Å, C14···Cg(C25–C30)ⁱⁱ=3.587(3) Å, with angle at H14 of 154° for symmetry operation ii: 2-*x*, 1-*y*, 1-*z*.
- [17] a) T. Kappe, D. Pocivalnik, *Heterocycles* 1983, 20, 1367; b) C. Heldmann, M. Schulze, G. Wegner, *Macromolecules* 1996, 29, 4686; c) H. M. Duong, Ph.D. Thesis (UCLA), 2003.
- [18] R. A. Pascal, Jr., W. D. McMillan, D. Van Engen, R. G. Eason, J. Am. Chem. Soc. 1987, 109, 4660.
- [19] Bruker AXS Inc. APEX2 and SAINT software, Bruker AXS Inc., Madison, Wisconsin, 2009.
- [20] G. M. Sheldrick, SADABS. University of Göttingen, Göttingen, 1996.
- [21] G. M. Sheldrick, Acta Crystallogr. Sect. A 2008, 64, 112.
- [22] L. J. Farrugia, J. Appl. Crystallogr. 1999, 32, 837.
- [23] DIAMOND, Visual Crystal Structure Information System, Version 3.1, CRYSTAL IMPACT, Postfach 1251, 53002 Bonn, Germany, 2006.

Received: January 6, 2010 Published online: May 12, 2010

7426 ·