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t-Butyl Biphenylation of *o*-Dibromoarenes: A Route to Soluble Polycyclic Aromatic Hydrocarbons

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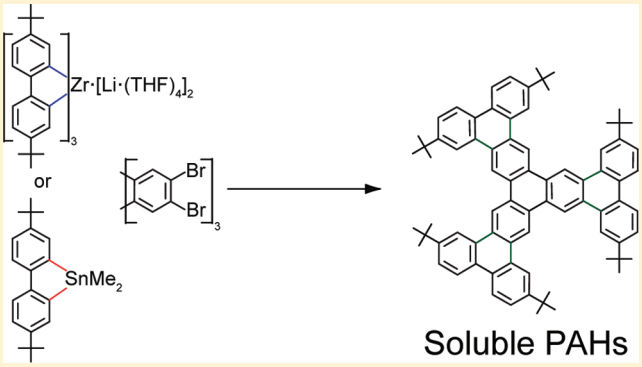
Supporting Information

ABSTRACT: Large, soluble polycyclic aromatic hydrocarbons (PAHs) have been synthesized using Zr-mediated and Stille-type biphenylation reactions. Both the Zr and Stille methodologies have been adopted to incorporate *tert*-butyl substituents, permitting the direct synthesis of alkylated PAHs that are much more soluble than their unsubstituted analogues. To demonstrate the utility of these methods and the importance of solubilizing functionality, several large PAHs were synthesized and crystallographically characterized. The scope of the Zr-mediated and Stille methodologies is shown to be complementary. The Stille methodology often gives higher yields but is ineffective for the introduction of strain and failed with some polybrominated arenes. In these difficult cases, the zirconium methodology is effective, albeit in low yields.

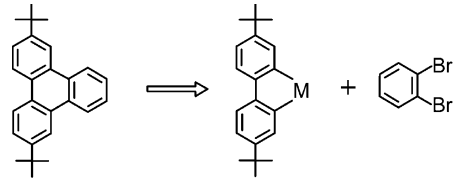


INTRODUCTION

Polycyclic aromatic hydrocarbons (PAHs) have played an important role in supramolecular chemistry and molecular electronics.[1] There are numerous examples of aryl−aryl bond-forming reactions to synthesize PAHs.[2] However, coupling reactions for the direct synthesis of triphenylenes from 1,2-dihaloarenes are limited. The recently developed biphenylation of 1,2-dihaloarenes, by Scott[3] and Nagao[4] using a palladium-catalyzed double Stille coupling and by our group using a Zr-mediated coupling,[5] provides direct routes to the triphenylene core. Itami has recently reported an effective Suzuki coupling/oxidative CH activation sequence to prepare the triphenylene core.[6]

We have modified the Zr-mediated and Stille coupling biphenylation reactions to incorporate solublizing *t*-butyl substituents (Scheme 1) and then applied these methods to the synthesis of the PAH cores hexabenzotetracene **7**, supertriphenylene **5**, and the triphenylene-based triptycene **9**. The *t*-butyl substituents render these large PAHs freely soluble in common solvents. Single crystals suitable for X-ray diffraction could be grown for **7**, **7a**, and **5**, permitting the first crystal structures of these large PAH cores. The core of **9** has been reported previously.[7] In the course of this work, we observed that in easy cases, the Stille methodology gives better yields than the Zr methodology, but the Zr methodology works in strained systems and with some polybrominated arenes, where the Stille methodology fails.

Scheme 1. Disconnection of Triphenylene



RESULTS AND DISCUSSION

2,2′-Dibromo-4,4′-di(*t*-butyl) biphenyl **1** was easily prepared on a 20 g scale by Friedel−Crafts alkylation[8] followed by electrophilic bromination.[9] Dilithiation of **1** followed by trapping with zirconocene dichloride gave tris(4,4′-di-*t*-butyl-2,2′-biphenyldiyl)zirconate **2**.[10] Likewise, dilithiation of **1** followed by trapping with trimethyltin chloride gave 9,9-dimethyl-4,4′-di(*t*-butyl)-9-stannafluorene **3** (Scheme 2).[4]

Supertriphenylene **5**, whose PAH core has been previously reported,[11,12] was prepared using both the Zr-mediated and Stille methodology. Treatment of hexabromotriphenylene[11] **4** with zirconofluorene **2** gave *tert*-butyl-substituted supertriphenylene **5** in 4% yield. The analogous Stille reaction using stannafluorene **3** gave supertriphenylene **5** in 28% yield (Scheme 3).

Itami recently reported the synthesis of the **7a**, the first example of the hexabenzotetracene core, by their Suzuki coupling/oxidative CH activation sequence.[6] We report the synthesis of hexabenzotetracenes **7** and **7a** using the Zr-mediated coupling. The reaction of 4,5,9,10-tetrabromo-2,7-

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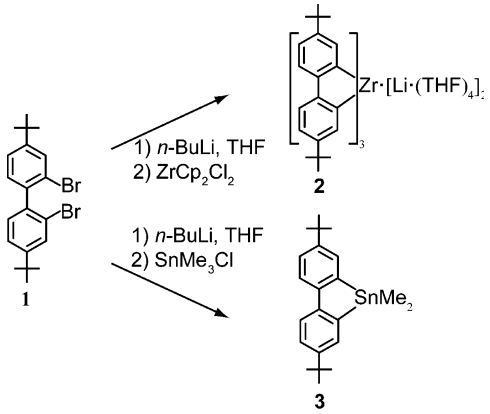
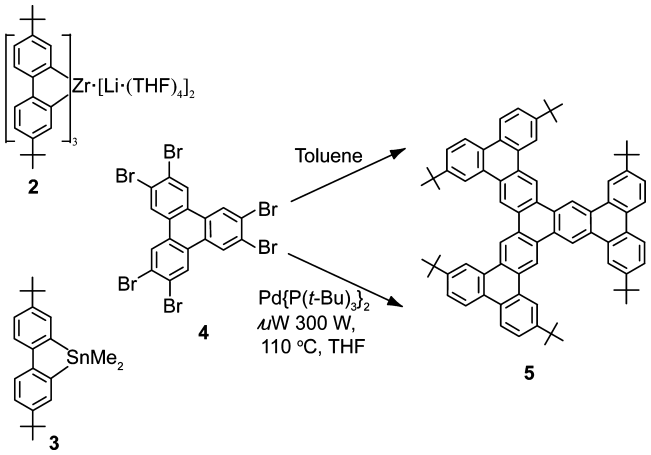
Scheme 2. Synthesis of Zirconofluorene 2 and Stannafluorene 3

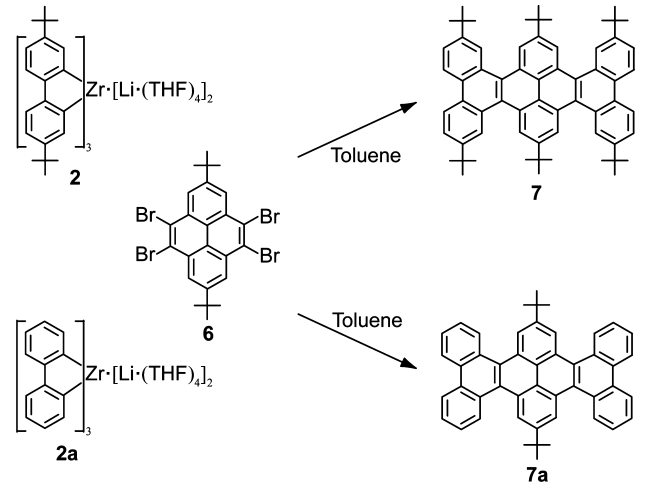


Scheme 3. Synthesis of Supertriphenylene 5



di(*t*-butyl)pyrene[13] **6** with zirconofluorene **2** gave **7** in 10% yield and the reaction of **6** with zirconofluorene **2a**[10] gave di(*t*-butyl)hexabenzotetracene **7a** in 25% yield (Scheme 4).

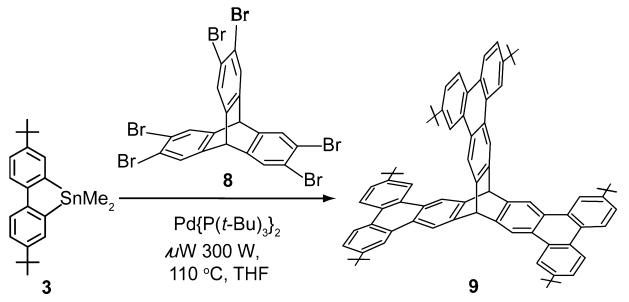
Scheme 4. Synthesis of Hexabenzotetracenes 7 and 7a



For reasons that remain unclear, the Stille conditions failed on some polybrominated arenes. All attempts to prepare the hexabenzotetracene core using Stille methods failed. Likewise, the Stille methodology failed to give [a,c,h,j]-tetrabenzoanthracenes from 1,2,4,5-tetrabromobenzene or 1,2,4,5-tetrabromo-*p*-xylene. This contrasts with the Zr-mediated coupling, which gives tetrabenzoanthracenes from tetrabromobenzene and tetrabromoxylene.[5]

In some cases, the Stille method gives better yield. Hexabromotriptycene **8** couples with the stannafluorene **3** to give triptycene **9** in 28% yield, but the Zr methodology gives **9** in only 9% yield (Scheme 5).[7]

Scheme 5. Synthesis of Triptycene 9



These *t*-butylated reagents offer many advantages over their nonalkylated analogues. The zirconofluorene **2** is more stable than its unsubstituted analogue **2a** and can be stored for months in a freezer under nitrogen atmosphere. More importantly, the *t*-butyl groups make the products soluble and tractable. All PAHs reported here are freely soluble in common organic solvents.

The enhanced solubility of the *t*-butylated PAHs also makes it somewhat easier to grow crystals for X-ray diffraction studies. After extended efforts, we were able to grow crystals of **7**, **7a**, and **5**. Triptycene **9** resisted all efforts at crystallization, but the X-ray structure of its core has been reported.[7]

The crystal structure of supertriphenylene **5** was determined for the first time (Figure 1). One of the blades is bent away downward. The least-squares plane of its 12 distal aromatic carbon atoms forms an angle of 18° with respect to the least squares plane of the other 24 distal aromatic carbon atoms.

The hexabenzotetracene core, which also has not been previously reported, has four cove regions[14] and is highly twisted. Indeed, four [4]helicene units can be identified in the structure. The central pyrene moiety in hexabenzotetracene **7a** forms angles of 32° and 30° (least-squares planes of all atoms in the moieties) with the terminal blades, which is comparable to the related compound tetrabenzonaphthacene.[15] The PAH core of **7** is essentially the same as **7a** (Figure 2).

The benzene disolvate of **7a** packs in the rhombohedral space group $R\bar{3}$ (Figure 3). The asymmetric unit contains one molecule of **7a** and two benzene molecules. The packing is determined by eight CH−π interactions between molecules of **7a**. One benzene solvate bridges two molecules of **7a** by CH−π interactions. The other benzene solvate lacks any close contacts and only fills space. π-Stacking is absent within the crystal packing. Small channels parallel to the *c*-axis are partially unfilled, with voids accounting for 1.5% of the crystal volume.

The diethyl ether monosolvate of **7** packs in space group $P2_1/c$ (Figure 4). The asymmetric unit contains one molecule of **7** and one diethyl ether molecule. Lamella of **7**, held together by four CH−π interactions per molecule, present only *t*-Bu groups to the adjacent lamella. Again, π-stacking is absent. These two crystal structures demonstrate how curvature and the presence of bulky *t*-butyl groups preclude the herringbone

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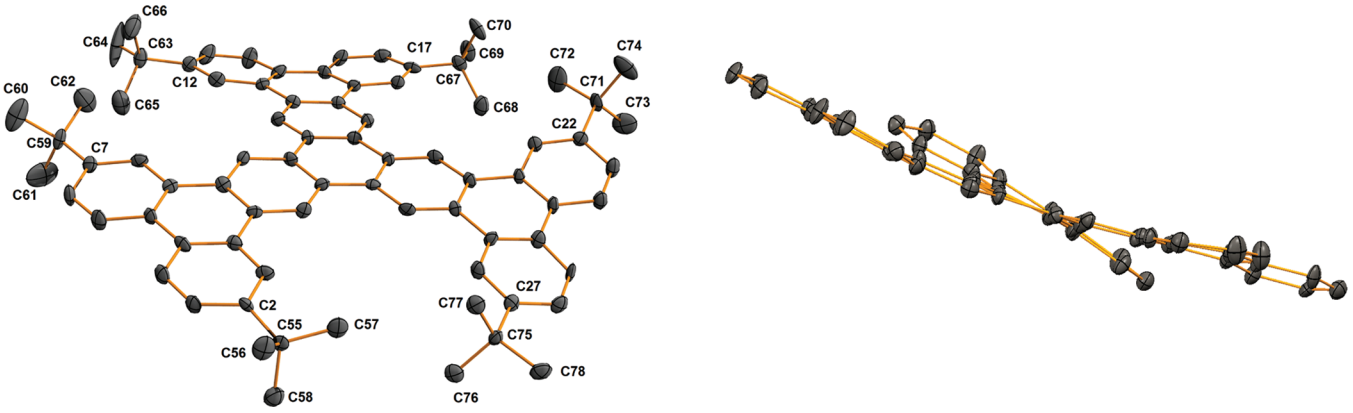

Figure 1. X-ray crystal structure of **5**. Hydrogen atoms (left and right) and *t*-butyl groups (right) omitted for clarity.

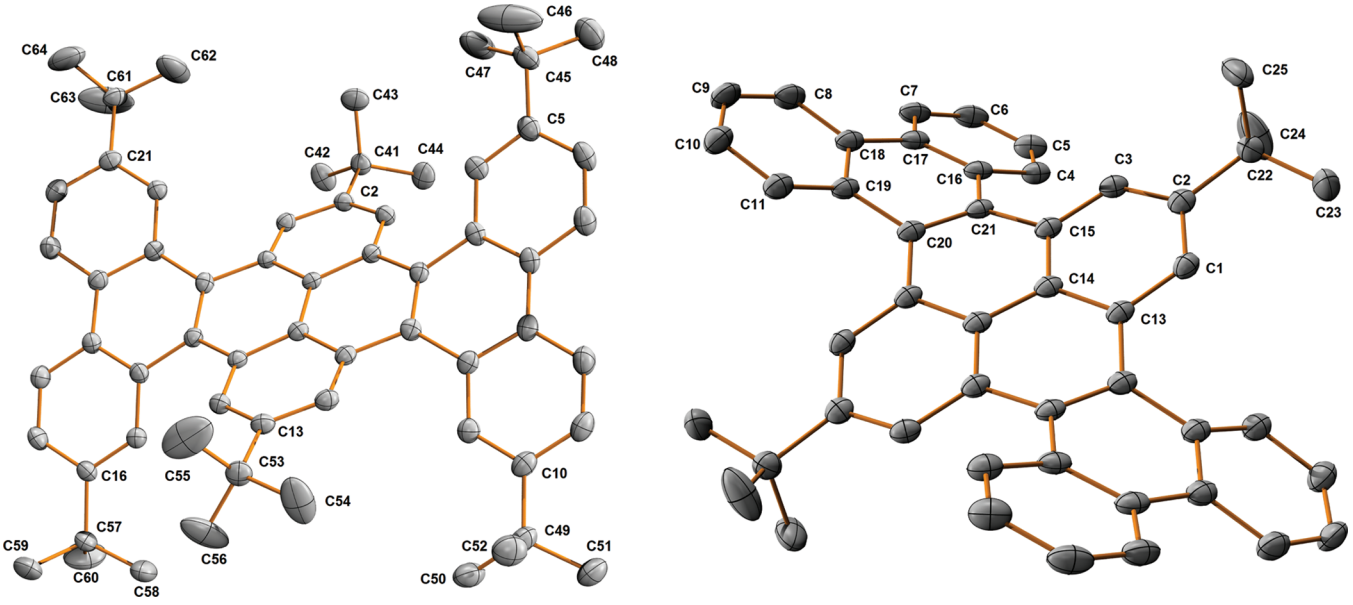

Figure 2. Crystal structures of hexabenzotetracenes **7** and **7a**.

and stacking motifs found in most PAH crystals.[16] The weak packing disfavors crystallinity and gives rise to good solubility.

The *o*-dibromobenzene monosolvate of supertriphenylene **5** packs in space group $P\bar{1}$ (Figure 5). Molecules of **5** form π-stacks that are offset along the armchair direction. The offset allows the *t*-butyl groups to nestle into the peripheral voids of the adjacent supertriphenylene core. *o*-Dibromobenzene spans the columns by donating a CBr$-\pi$ interaction on one end and a CH$-\pi$ interaction on the other end. Unlike **7** and **7a**, crystals of **5** are stabilized by partial π-stacking. This difference illustrates the importance of curvature in suppressing π-stacking. The presence of *t*-butyl groups is nonetheless sufficient to impart good solubility.

The absorbance spectrum of supertriphenylene **5** is consistent with that reported previously for the unsubstituted PAH core[11] and the dodecakis-alkoxy derivative (Figure 6).[12] The near overlap of the 392 nm absorbance peak and the 397 nm emission peak suggests their assignment to the 0–0 vibrational bands of $S_0 \rightarrow S_1$ and $S_1 \rightarrow S_0$ transitions.[17] The fine structure of the emission spectrum can be fitted to a vibrational series at ~680 cm^{-1}. The Stokes shift of 320 cm^{-1} is consistent with a rigid aromatic core whose geometry changes only slightly upon excitation.

CONCLUSIONS

The Stille cross coupling reaction was successful in the synthesis of some PAHs, but this methodology failed to afford strained PAHs. The Stille double cross coupling reaction has the advantage of higher yields than Zr-mediated coupling. The Zr-mediated coupling is effective in the synthesis of the strained PAHs though giving lower yields. Analysis of the crystal packing of **5**, **7**, and **7a** suggests that curvature and *t*-butyl groups disrupt the normal herringbone and π-stacking motifs that are normally found in large PAHs.

EXPERIMENTAL SECTION

4,4′-Dibromo-2,2′-di(*t*-butyl)biphenyl[8] and 2,7-di(*t*-butyl)-4,5,9,10-tetrabromopyrene[9] **6** were synthesized according to the literature procedures. The reactions involving zirconofluorenes **2**, **2a**, and palladium compounds were run under strictly anhydrous conditions using the standard Schlenk techniques. ^{1}H and ^{13}C spectra were fully assigned for the PAHs **7**, **7a**, and **5** using ^{1}H–^{1}H-COSY, NOE, HMQC, and HMBC experiments. IR spectra were recorded using a total internal reflectance module having a spectral range 4000–600 cm^{-1}. Mass spectra were recorded using an atmospheric pressure photoionization (APPI) source on a time-of-flight (TOF) instrument in the positive mode. The addition of toluene promoted ionization. In

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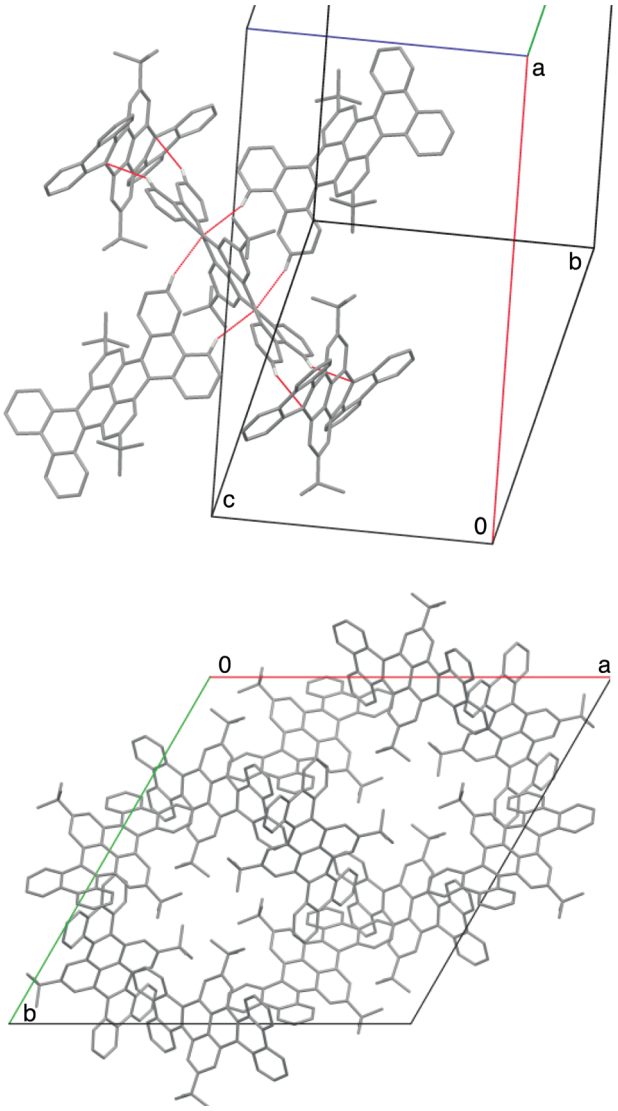

Figure 3. CH−π interactions (top) and crystal packing (bottom) in **7a**. Solvent and most hydrogen atoms are omitted.

some cases, MS were recorded using a matrix assisted laser desorption (MALDI-TOF).

Synthesis of Tris(4,4′-di(*t*-butyl)-1,1′-biphenyldiyl) Zirconate (2). 2,2′-Dibromo-4,4′-di(*t*-butyl)biphenyl **1** (19.0 g, 45.0 mmol) was dissolved in THF (90 mL) under nitrogen. This solution was cooled to −78 °C, and *n*-butyllithium (33.6 mL, 90.0 mmol) was added at a rate of 0.1 mL/min using a syringe pump. After the addition was complete, the reaction mixture was warmed to −30 °C. Then, dichlorobis(η^5-2,4-cyclopentadien-1-yl) zirconium (3.25 g, 11.0 mmol) was added, and the solution was warmed to room temperature. The resulting precipitate was filtered under nitrogen to give a first crop of **2** (3.0 g). Heptane (90 mL) was added to the filtrate to give a second crop of **2** as THF solvate (total yield = 7.0 g, 58%): ^{1}H NMR (C_6D_6) δ 8.35 (s, 6H), 7.81 (d, J = 8 Hz, 6H), 7.35 (d, J = 8 Hz, 6H), 3.18 (s, 32H), 1.48 (s, 54H), 1.26 (s, 32H); ^{13}C NMR (C_6D_6) 148.2, 137.1, 125.5, 120.5, 67.6, 34.7, 32.1, 25.4.

Synthesis of 9,9-Dimethyl-4,4′-di(*t*-butyl)-9-stannafluorene (3). 2,2′-Dibromo-4,4′-di(*tert*-butyl) biphenyl (0.50 g, 1.2 mmol) was dissolved in THF (10 mL) under nitrogen. This solution was cooled to −78 °C, and *n*-butyllithium (1.6 mL, 2.5 mmol) was added at a rate of 0.1 mL/min using a syringe pump. This mixture was stirred for 1 h followed by the addition of trimethyltinchloride (0.70 g, 3.5 mmol). The solution was then allowed to warm to room temperature. The solvent was evaporated. The solid residue was dissolved in chloroform

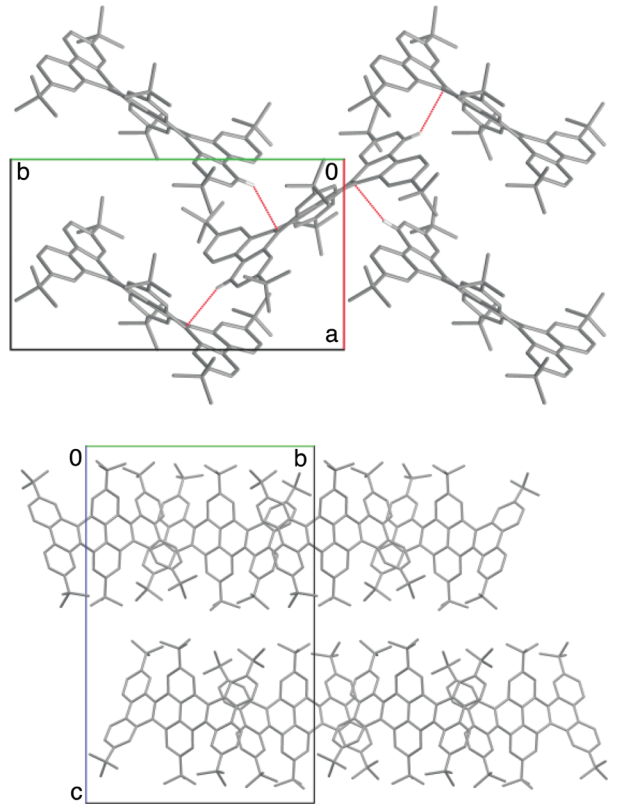

Figure 4. CH−π interactions (top) and crystal packing (bottom) in **7**. Solvent and most hydrogen atoms are omitted.

(50 mL) and passed through a small plug of florisil. The product was further purified by kugelrohr sublimation at 110 °C/60 mTorr to give **3** as white crystals (0.25 g, 54%): mp 127−129 °C; ^{1}H NMR ($CDCl_3$) δ 7.81 (d, J = 8 Hz, 2H), 7.64 (s, 2H), 7.38 (d, J = 8 Hz, 2H), 1.35 (s, 18H), 0.53 (s, 3H); ^{13}C NMR ($CDCl_3$) 149.9, 145.8, 140.7, 133.0, 126.5, 122.0, 34.9, 31.7, −8.1; IR (ATR) [cm $^{-1}$] 2958, 1455, 1390, 1253, 819, 787; HRMS (APPI-TOF) calcd for $C_{22}H_{30}Sn$ 410.1365, found 410.1335.

Synthesis of 11,22-Di(*t*-butyl)hexabenzo[a,c,fg,j,l,op]-tetracene (7a). 4,5,9,10-Tetrabromo-2,7-di(*tert*-butyl)-pyrene **6** (0.16 g, 0.25 mmol) was dissolved in toluene (150 mL) in an oven-dried Schlenk flask, and the zirconofluorene **2a** (0.85 g, 0 0.75 mmol) was added. The reaction mixture was then stirred at room temperature for 2 days. After all starting material was consumed (by APPI+/MS), the reaction was quenched with dry carbon dioxide gas. Methanol (10 mL) was added, and solvents were evaporated. The crude solids were triturated with chloroform, and the solution was passed through a plug of basic alumina. The solvent was evaporated, and product was purified with column chromatography (SiO_2, hexane/chloroform 7:3). The product was further purified by crystallization from chloroform/hexane (7:3) to give **7a** (78 mg, 25%): mp > 350 °C (darkens 260 °C); ^{1}H NMR ($CDCl_3$) δ 9.06 (s, 4H), 8.91 (d, J = 8 Hz, 8H), 8.81 (d, J = 8 Hz, 8H), 7.72 (m, 8H), 1.64 (s, 18H); ^{13}C NMR ($CDCl_3$) 148.0, 131.3, 128.7, 128.5, 128.2, 126.9, 126.9, 124.9, 124.0, 123.7, 123.0, 36.0, 32.1; UV λ_{max}/nm (hexane) (log ε) 264 (5.08), 312 (4.76), 325 (5.02), 376 (4.43), 396 (4.48); HRMS (MALDI-TOF) calcd for $C_{48}H_{38}$ 614.2974, found 614.2963; IR (ATR) [cm^{-1}] 2959, 1590, 1257, 756, 726.

Synthesis of 3,8,11,14,19-Hexakis(*t*-butyl)hexabenzo-[a,c,fg,j,l,op]tetracene (7). 4,5,9,10-Tetrabromo-2,7-di(*t*-butyl)-pyrene **6** (0.32 g, 0.50 mmol) was dissolved in toluene (150 mL) in an oven-dried Schlenk flask, and the zirconofluorene **2** (1.5 g, 1.0 mmol) was added. The reaction mixture was then stirred at room temperature for 2 days. After all starting material was consumed (by APPI+/MS), the reaction was quenched with dry carbon dioxide gas. Methanol (10 mL) was added, and solvents were evaporated. The

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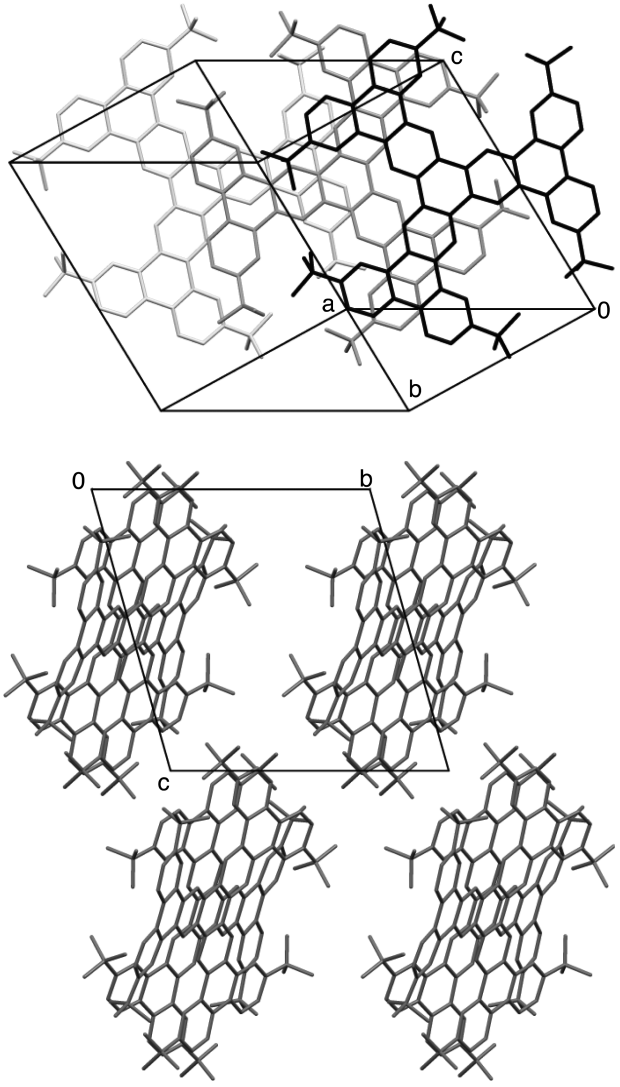

Figure 5. π-Stacking (top, shading to assist perception of the overlapping molecules) and crystal packing (bottom) in **5**.

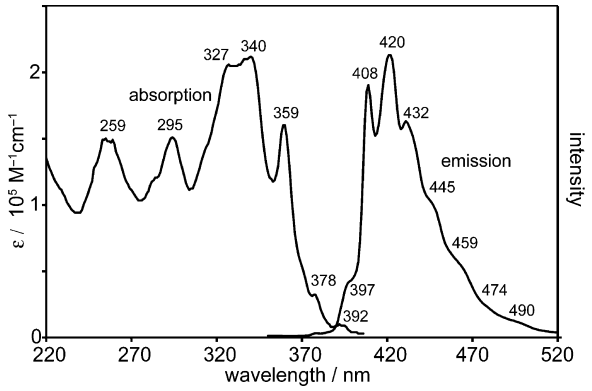

Figure 6. UV−vis spectrum (left axis) and steady-state emission spectrum (right axis) of **5**.

crude solids were dissolved in chloroform, and the solution was passed through a plug of basic alumina. The solvent was evaporated, and product was purified with column chromatography (SiO_2, hexane/chloroform 2:8). The product was further purified by normal phase SiO_2 HPLC using chloroform/hexane (2:8) to give **7** (40 mg, 9.7%): mp > 350 °C (darkens 210 °C); ^{1}H NMR ($CDCl_3$) δ 9.21 (s, 4H), 9.14 (s, 4H), 8.66 (d, J = 8 Hz, 4H), 7.70 (d, J = 8 Hz, 4H), 1.55 (s, 18H), 1.44 (s, 36H); ^{13}C NMR ($CDCl_3$) 149.3 (C_3), 148.2 (C_{22}), 129.8 ($C_{1a}{}^2$), 129.1 (C_{1a}), 129.0 ($C_{1a}{}^1$), 128.5 (C_{5a}), 124.8 (C_5), 124.8 (C_2), 123.60 (C_4), 123.0 (C_1), 122.7 ($C_{20a}{}^3$), 36.0 ($C_{22'}$), 35.4 ($C_{3'}$), 32.4 ($C_{22''}$), 31.9 ($C_{3''}$); UV λ_{max}/nm (hexane) (log ε) 272 (5.47), 330 (5.32), 378 (4.85), 398 (4.89); HRMS (APPI-TOF) calcd for $C_{64}H_{70}$ 838.5477, found 838.5468; IR (ATR) [cm^{-1}] 2953, 1609, 877, 814, 738.

Synthesis of 2,7,12,17,22,27-Hexakis(*t*-butyl)hexabenzo-[a,c,k,m,u,w]trinaphthylene (5) by Zr-Mediated Coupling. Hexabromotriphenylene **4** (0.11 g, 0.15 mmol) was dissolved in toluene (100 mL) under nitrogen. The zirconofluorene **2** (1.0 g, 0.7 mmol) was added, and the reaction was stirred for 3 days at room temperature. After all the starting material was consumed (by APPI +/MS), the reaction mixture was quenched with dry carbon dioxide gas. Methanol (10 mL) was added, and solvents were evaporated. The residue was dissolved in chloroform, and the solution was passed through a small plug of basic alumina. The crude product was washed with hexane, and product was crystallized from acetone to give **5** (4.7 mg, 3.1%): mp > 350 °C (darkens 230 °C); ^{1}H NMR ($CDCl_3$) δ 10.19 (s, 6H), 9.09 (s, 6H), 8.66 (d J = 8 Hz, 6H), 7.83 (d, J = 8 Hz, 6H), 1.68 (s, 54H); ^{13}C ($CDCl_3$) 150.1 (C_3), 130.1 ($C_{30a}{}^1$), 129.6 ($C_{1a}{}^1$), 129.5 (C_{1a}), 128.3 (C_{5a}), 125.7 (C_4), 123.5 (C_5), 119.8 (C_2), 118.3 (C_1), 35.4 ($C_{3'}$), 32.2 ($C_{3''}$); UV λ_{max}/nm (hexane) (log ε) 254 (3.50), 295 (3.48), 339 (3.63), 359 (3.52); HRMS (APPI-TOF) calcd for $C_{78}H_{78}$ 1014.6104, found 1014.6175; IR (ATR) [cm $^{-1}$] 2960, 1393, 871, 810.

Synthesis of 2,7,12,17,22,27-Hexakis(*t*-butyl)hexabenzo-[a,c,k,m,u,w]trinaphthylene (5) by Stille Coupling. To a microwave tube, hexabromotriphenylene **4** (50 mg, 0.07 mmol), stannafluorene **3** (0.15 g, 0.30 mmol), and bis(tri-*t*-butylphosphine) palladium (0) (0.02 g, 0.14 mmol) were added in a glovebox. To this reaction mixture, THF (10 mL) was added. The mixture was irradiated in the microwave at 120 °C (300 W). The reaction was monitored by APPI+/MS. After all the starting material was consumed, the solvent was evaporated. The solid mixture was dissolved in chloroform, and the solution was passed through a small plug of silica. The crude product was then washed with acetone to give clean **5** (20 mg, 28%).

Reaction of 9,9-Dimethyl-4,4′-di(*t*-butyl)-9-stannafluorene 3 with Hexabromotriptycene 8. To a microwave tube, hexabromotriptycene **8** (50 mg, 0.07 mmol), stannafluorene **3** (0.15 g, 0.28 mmol), and bis(tri-*t*-butylphosphine) palladium (0) (20 mg, 0.14 mmol) were added in a glovebox. To this reaction mixture, THF (10 mL) was added. The reaction mixture was irradiated in the microwave at 120 °C (300 W). The reaction was monitored by APPI+/MS. After all the starting material was consumed (1−2 h), the solvent was evaporated. The solid mixture was dissolved in chloroform, and the solution was passed through a small plug of silica. The crude product was then washed with acetone to give clean **9** (23 mg, 28%): mp > 350 °C (darkens 210 °C); ^{1}H NMR δ 8.91 (s, 6H), 8.68 (s, 6H), 8.47 (d, J = 8 Hz, 6H), 7.64 (d, J = 8 Hz, 6H), 6.32 (s, 2H), 1.51 (s, 18H); ^{13}C NMR δ 149.4, 143.2, 129.3,128.5, 127.7, 125.0, 123.1, 119.4, 118.6, 54.5, 35.3, 31.8; UV λ_{max}/nm (log ε) 277 (3.07), sh 281; HRMS (APPI-TOF) calcd for $C_{80}H_{80}$ 1040.6260, found 1040.6225; IR (ATR) [cm $^{-1}$] 2951, 1613, 877, 812, 723.

■ ASSOCIATED CONTENT

Ⓢ Supporting Information

Crystallographic information files for **2**, **5**, **7**, and **7a** and NMR, IR, UV−vis, and fluorescence spectra. This material is available free of charge via the Internet at http://pubs.acs.org.

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■ ACKNOWLEDGMENTS

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