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Efficient Synthesis of Symmetrically and Unsymmetrically Substituted Hexaphenylbenzene Analogues by Suzuki–Miyaura Coupling Reactions

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Abstract: A series of symmetrically and unsymmetrically substituted hexaphenylbenzene (HPB) analogues were efficiently synthesized by using a sterically hindered Suzuki–Miyaura cross-coupling reaction of arylboronic acids with 1,4-diiodo-2,3,5,6-tetraarylbenzenes under our optimized reaction conditions. The 1,4-diiodo-2,3,5,6-tetraarylbenzenes can be readily prepared by using a one-pot Hart reaction. Oxidative cyclodehydrogenation of the dibromo derivative with $FeCl_3$ gave the corresponding hexabenzocoronene (HBC) derivative in good yield.

Keywords: aromatic compounds · C–C coupling · cyclodehydrogenation · hexabenzocoronene · hexaphenylbenzene

Introduction

The preparation of hexaphenylbenzene (HPB) analogues is an important synthetic task, as these aromatic compounds have been widely applied in organic synthetic chemistry and in materials science.[1] Of particular importance is that HPBs are direct precursors of hexabenzocoronene (HBC) derivatives, which are potential candidates for organic semiconducting materials in field-effect transistors (FETs), hole-conducting layers in photovoltaic devices, light-emitting diodes (LEDs), and molecular wires in nanoscale molecular electronics.[2] Several distinct approaches have been reported for the preparation of HPBs, such as the transition-metal-catalyzed cyclotrimerization of diphenylacetylenes or the Diels–Alder cycloaddition between tetraphenylcyclopentadienones and substituted diphenylacetylenes.[3] However, these methods suffer from limitations, such as harsh reaction conditions or multistep synthetic pathways. Moreover, unsymmetrically substituted starting materials afford a mixture of regioisomeric products, usually with little or no selectivity. The regioselective synthesis of unsymmetrically substituted HPBs is thus difficult when using the existing protocols.[4] The ever-increasing complexity of target molecules and the constant need for functional-group tolerance make the development of novel methods for the straightforward, mild, and efficient preparation of HPBs a timely challenge.[5] Herein, we report a new approach for the synthesis of symmetrically and unsymmetrically substituted HPBs by Hart and sterically hindered Suzuki–Miyaura cross-coupling reactions (Scheme 1). Further oxidative cyclodehydrogenation with $FeCl_3$ of selected HPBs resulted in the corresponding HBC or some unexpected products.

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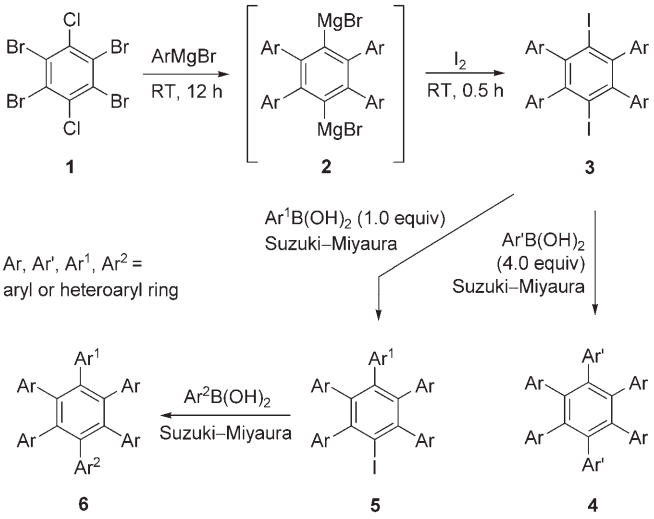
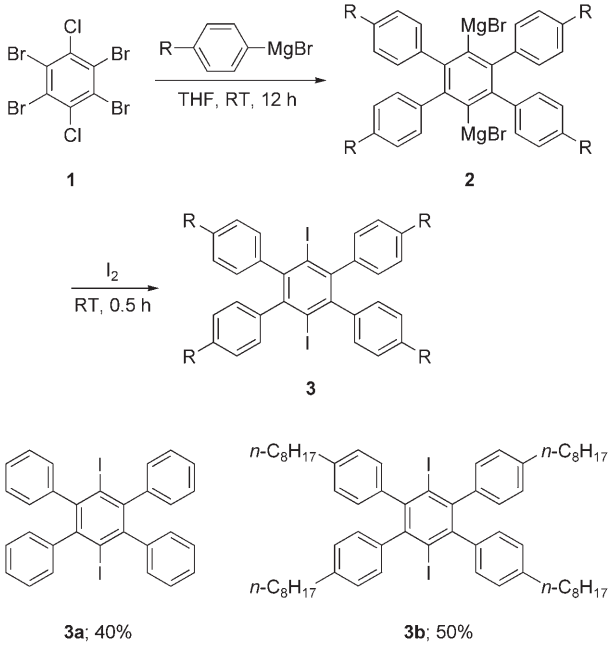
Results and Discussion

Treatment of 1,2,4,5-tetrabromo-3,6-dichlorobenzene (**1**) with various arylmagnesium bromides in THF at room temperature for 12 h led to the formation of dimagnesium intermediate **2** (Scheme 2).[6] Its direct reaction with iodine provided diiodobenzenes **3** in 40–60% yields. The observed correlation between the yield and the length of the alkyl side chain (H vs. C_8H_{17} vs. $C_{12}H_{25}$) may be a consequence of an enhancement of the solubility of intermediate **2** with increasing chain length.

With the compounds **3** in hand, our attention was refocused on the subsequent coupling reactions. The Suzuki–Miyaura coupling was chosen because it is one of the most powerful biaryl C–C bond-forming transformations available to synthetic organic chemists. This transformation enjoys a broad scope and wide functional-group tolerance.[7] However, in the case of sterically hindered aryl or alkenyl halides, limited success has been realized and special ligands and harsh reaction conditions are often required.[8] Pioneering work by Buchwald and co-workers afforded a general protocol for the sterically hindered Suzuki–Miyaura cross-coupling reaction, in which a novel and readily available phosphine, 2-dicyclohexylphosphino-2′,6′-dimethoxybiphenyl



Scheme 1. Synthesis of symmetrically and unsymmetrically substituted HPBs by Hart and Suzuki–Miyaura coupling reactions.

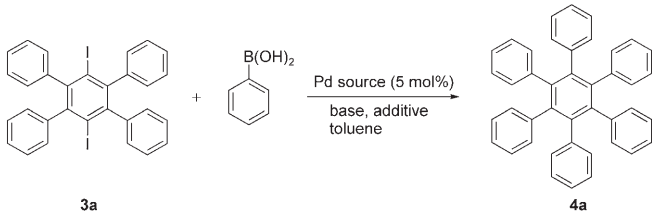


Scheme 2. One-pot procedure for the synthesis of 1,4-diiodo-2,3,5,6-tetraarylbenzenes **3**.

(S-phos), was used as a ligand in conjuction with $[Pd_2(dba)_3]$ (dba = dibenzylideneacetone).[9] Owing to the sterically hindered nature of the tetraaryl-substituted diiodobenzenes **3**, we employed Buchwald's catalyst system for the initial reaction. The 65% yield was very encouraging (Table 1, entry 1), but left room for further improvement.[9a,b] Thus, some optimization work was conducted by using compound **3a** and phenylboronic acid as the substrates. The results are summarized in Table 1.

Screening alternative palladium sources revealed the superiority of $[Pd(PPh_3)_4]$ over other Pd precusors (Table 1, entries 2–5). The effect of the base was also examined, and K_2CO_3 was found to be the most effective (Table 1, entries 5–8). Furthermore, solid K_2CO_3 (Table 1, entry 6; 94%) was more effective than aqueous 2 M K_2CO_3 (Table 1, entry 9; 70%). Increasing the reaction temperature from 100 °C to 120 °C had a detrimental effect on the reaction, and several by-products, such as mono-coupled and deiodinated compounds, were observed (Table 1, entry 10). Interestingly, the introduction of the phase-transfer catalyst aliquat 336 (2 mol%) was found to accelerate the reaction rate, and the desired product was obtained in an excellent yield of 95% after a reaction period of 12 h (Table 1, entry 11). Other phase-transfer catalysts, such as Bu_4NBr, were found not to promote this reaction (Table 1, entry 12).

Table 1. Optimization of reaction conditions for the sterically hindered Suzuki–Miyaura coupling reaction.



Entry	Catalyst system	T [°C]	t [h]	Base	Yield [%][a]
1	$[Pd_2(dba)_3]$ + S-phos	100	24	K_3PO_4	65
2	$[PdCl_2(PPh_3)_2]$	100	24	K_3PO_4	30
3	$[PdCl_2(dppf)]$	100	24	K_3PO_4	56
4	$[Pd(OAc)_2]$ + PPh_3	100	24	K_3PO_4	–[b]
5	$[Pd(PPh_3)_4]$	100	24	K_3PO_4	86
6	$[Pd(PPh_3)_4]$	100	24	K_2CO_3	94
7	$[Pd(PPh_3)_4]$	100	24	Cs_2CO_3	85
8	$[Pd(PPh_3)_4]$	100	24	$Ba(OH)_2 \cdot 8H_2O$	–[b]
9	$[Pd(PPh_3)_4]$	100	24	K_2CO_3	70[c]
10	$[Pd(PPh_3)_4]$	120	12	K_2CO_3	56
11	$[Pd(PPh_3)_4]$ + aliquat 336	100	12	K_2CO_3	95
12	$[Pd(PPh_3)_4]$ + Bu_4NBr	100	12	K_2CO_3	45

[a] Yield of isolated, analytically pure compound. [b] No desired product was detected. [c] K_2CO_3 was used as a 2 M aqueous solution.

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To explore the scope of this method (Table 2), the Suzuki–Miyaura coupling reaction of diiodobenzenes **3a–c** with a variety of arylboronic acids was performed under the optimized reaction conditions (Table 2). The presence and/or length of the alkyl side chain had no influence on the outcome of the reaction. Compounds **3a–c** reacted with phenylboronic acid to give compounds **4a–c** in essentially the same yield (92–95%) (Table 2, entries 1–3).[10] The electronic nature of the substituted phenylboronic acids also had little effect on the yield of the coupling, and thus HPBs with both electron-donating and electron-withdrawing groups could be synthesized in good to excellent yields (Table 2, entries 4–10). For the coupling with 4-bromophenylboronic acid, a lower temperature (80°C) and longer time (18 h) were necessary for the achievement of chemoselectivity between the hindered aryl iodides and the non-hindered aryl bromide functionality of the 4-bromophenylboronic acid. Under these conditions, HPB **4g** was prepared in 91% yield. Moreover, the reaction could be performed on a synthetically useful scale. For example, compounds **4f** and **4h** were prepared on a gram scale in 81% and 91% yields, respectively. Variation of the position of the substituent on the phenylboronic acids did not significantly affect the reaction, and the corresponding HPB analogues were formed in 74–98% yield (Table 2, entries 8–10). Remarkably, even the sterically hindered 2-methoxyphenylboronic acid readily participated in the formation of HPB analogue **4j**, and two rotamers were obtained after column purification (Table 2, entry 10). Heteroaromatic boronic acids also reacted well and this was demonstrated by the efficient formation of compounds **4k** and **4l** by this protocol (Table 2, entries 11–12).

The Suzuki–Miyaura coupling reaction is not only an expedient approach to symmetrically substituted HPBs with a 1,4-Ar^1-2,3,5,6-Ar^2 pattern, but also to unsymmetrically substituted ones with a 1-Ar^1-2,3,5,6-Ar^2-4-Ar^3 pattern. Thus, treatment of **3a** with various arylboronic acids (1.05 equiv) at 80°C for 12 h provided mono-coupled products of type **5** in moderate yields (Scheme 3). Under these conditions, less than 5% of the bis-coupled product was obtained. The lower reaction temperatures and shorter reaction times are essential for the selectivity (higher reaction temperature and longer reaction time resulted in relatively greater amounts of the bis-coupled product).

Compounds of type **5** are very useful HPB precursors as they open the door for unsymmetrically substituted HPB derivatives by sequential Suzuki–Miyaura reactions (Scheme 4). Thus treatment of **5a** and **5b** with aryl and heteroaryl boronic acids under our standard reaction conditions provided the unsymmetrically substituted HPBs **6a–d** in 85–93% yield, respectively. The heteroaryl-substituted iodobenzene **5c** also reacted well with 3-cyanophenylboronic acid, furnishing **6e** in 86% yield.

As mentioned at the beginning, HPBs are useful precursors for the synthesis of hexabenzocoronene (HBC) derivatives. Thus, the oxidative cyclodehydrogenation of the HPB **4g** with $FeCl_3$ as an oxidant in a mixture of CH_3NO_2 and CH_2Cl_2 at room temperature for 30 min afforded fused

Table 2. Preparation of symmetrically substituted HPBs **4** by sterically hindered Suzuki–Miyaura coupling reaction.

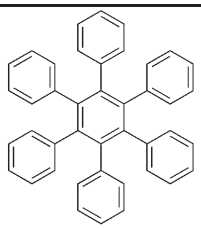
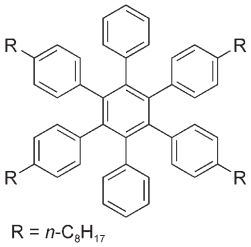
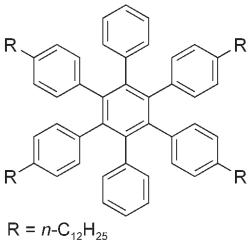
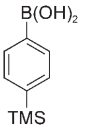
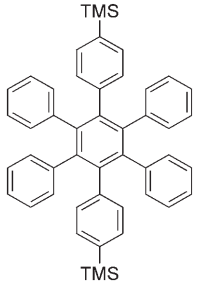
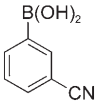
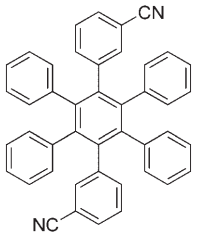
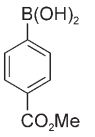
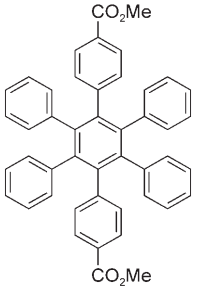
Entry	**3**	$ArB(OH)_2$	**4**	Yield [%][a]
1	**3a**		 **4a**	95
2	**3b**		 R = *n*-C_8H_{17} **4b**	93
3	**3c**		 R = *n*-$C_{12}H_{25}$ **4c**	92
4	**3a**		 **4d**	93
5	**3a**		 **4e**	87
6	**3a**		 **4f**	85, 81[b]

Table 2. (Continued)

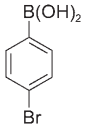
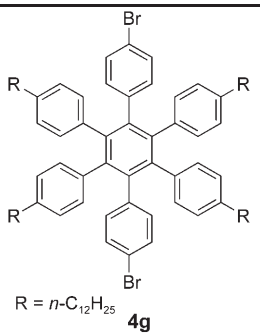
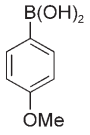
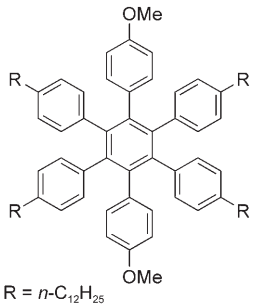
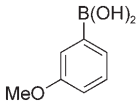
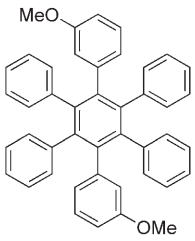
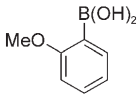
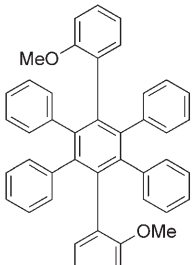
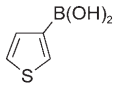
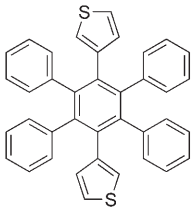
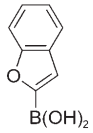
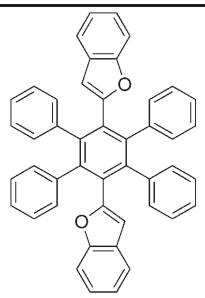
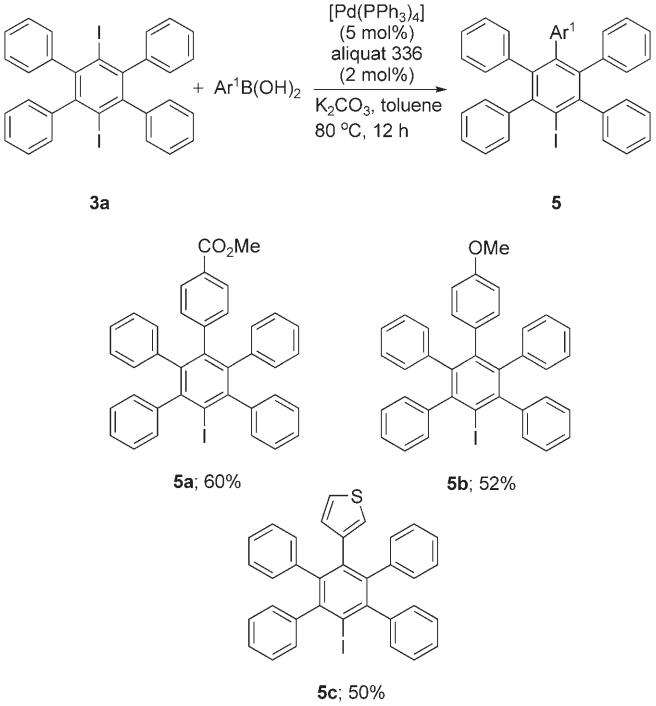
Entry	3	$ArB(OH)_2$	4	Yield [%][a]
7	**3c**		 R = *n*-$C_{12}H_{25}$ **4g**	91
8	**3c**		 R = *n*-$C_{12}H_{25}$ **4h**	
9	**3a**		 **4i**	94
10	**3a**		 **4j**	74[c]
11	**3a**		 **4k**	88

Table 2. (Continued)

Entry	3	$ArB(OH)_2$	4	Yield [%][a]
12	**3a**		 **4l**	76

[a] Yield of isolated, analytically pure compound. [b] Yield of a multigram-scale reaction. [c] Total yield of two rotamers.



Scheme 3. Synthesis of pentaaryl iodobenzenes of type **5** by a selective Suzuki–Miyaura coupling reaction.

HBC derivative **7a** in 85% yield after chromatographic purification (Scheme 5). Interestingly, cyclodehydrogenation of the HPB **4h** under the same reaction conditions unexpectedly provided the *meta*-dimethoxy HBC **7b** along with bisspirocyclic dienone **8**.[11]

Conclusions

In summary, we have developed a new and efficient method for the synthesis of HPB analogues in excellent yields by the Suzuki–Miyaura coupling reaction. The tetraphenyl-substituted diiodobenzenes of type **3** are highly sterically hin-

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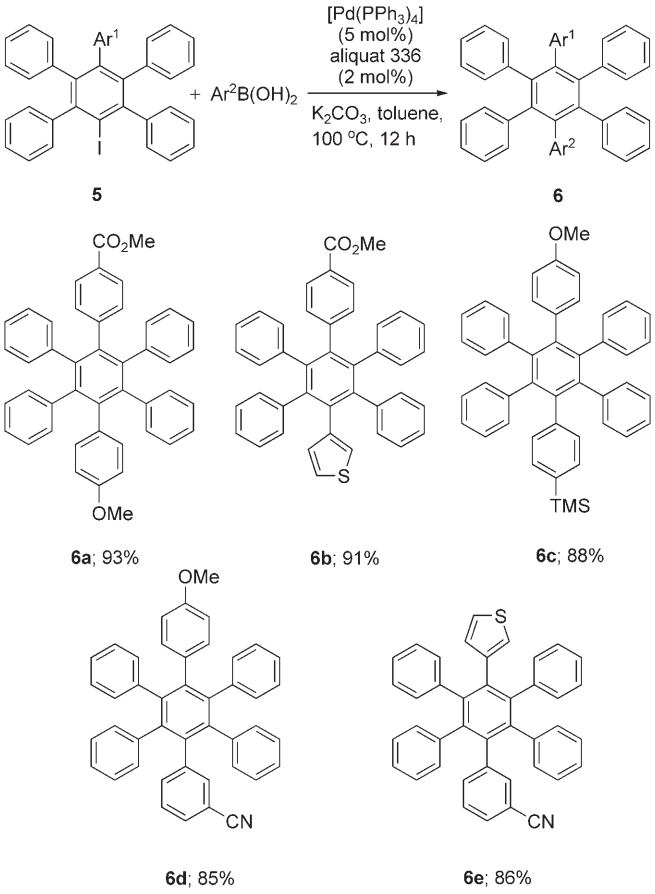
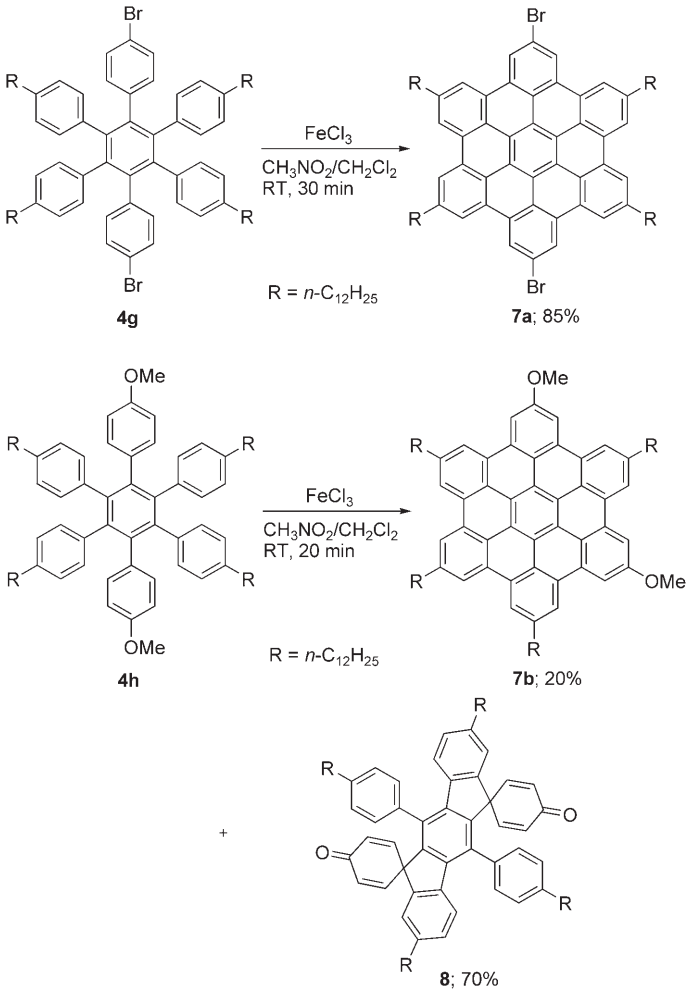


Scheme 4. Synthesis of unsymmetrical HPB analogues by Suzuki–Miyaura coupling reaction.



Scheme 5. Oxidative cyclodehydrogenation of HPBs **4g** and **4h**.

dered aryl iodides, which to our knowledge have not been used previously in Suzuki–Miyaura coupling reactions. Our protocol provides a novel method for the efficient synthesis of symmetrically substituted HPB analogues of type **4** in the presence of various functional groups. The selectivity of this coupling reaction allows a general and expedient approach to synthesize unsymmetrically substituted HPBs of type **6**, which are very difficult to prepare by other standard methods. Oxidative cyclodehydrogenation of HPB **4g** provided the desired HBC **7a** in good yield. However, in case of **4h**, the unexpected HBC **7b** and bis-spirocyclic dienone **8** were formed under the same reaction conditions. Further applications of this convenient protocol, for example, for polymerization or for the preparation of functionalized HBC derivatives, are underway in our laboratories.

Experimental Section

General

Unless otherwise indicated, all reactions were carried out with stirring and, if air- or moisture-sensitive, in flame-dried glassware under argon. Syringes used to transfer reagents and solvents were purged with argon prior to use. Reactions were monitored by FD-MS or thin-layer chromatography (TLC). All starting materials were purchased from commercial sources and used without further purification. Yields refer to yields of isolated compounds estimated to be >95% pure as determined by ^{1}H NMR and capillary GC.

Synthesis

4-Octylphenyl magnesium bromide and 4-dodecylphenyl magnesium bromide: Magnesium turnings (200 mmol) were placed in an Ar-flushed flask, and THF (10 mL) was added. 1,2-Dibromoethane (0.1 mL) was added, and the resulting mixture was stirred at room temperature for a few minutes. After the reaction ceased, the solution was removed by cannulation, and dry THF (120 mL) was added. Then, a solution of 1-bromo-4-octylbenzene or 1-bromo-4-dodecylbenzene (150 mL) in dry THF (30 mL) was slowly added at room temperature. The reaction started within a few minutes. After addition, the reaction mixture was stirred for 12 h at room temperature. The grey solution of 4-octylphenyl magnesium bromide or 4-dodecylphenyl magnesium bromide was cannulated to another flask under argon and removed in this way from the excess magnesium.

*Typical Procedure for the Formation of Compounds of Type **3** (TP A)*

3a: A suspension of 1,4-dichloro-2,3,5,6- tetrabromobenzene (1.85 g, 4.0 mmol) in dry THF (20 mL) was added to a solution of PhMgBr (32 mmol) in dry THF (32 mL) under argon, and the resulting mixture was stirred at room temperature for 12 h. I_2 (6.35 g, 25 mmol) was added directly to the reaction mixture at 0 °C, and the reaction was stirred at room temperature for 2 h. The reaction was quenched with water, and the resulting mixture was extracted with $CHCl_3$ (3×100 mL). The combined organic layers were washed with 2 M aqueous $NaHSO_3$ solution (2×

200 mL), brine (50 mL), and water (50 mL) and dried with $MgSO_4$. After filtration of the $MgSO_4$, the solvent was removed in vacuo, and the resulting mixture was filtered. The solid was washed with a little bit of benzene and hexane to give **3a** (0.7 g). The filtrate was purified by chromatography (eluent *n*-hexane/benzene = 30:1) to give **3a** (0.3 g). The total yield is 40%. ^{1}H NMR (CD_2Cl_2, 250 MHz): δ = 7.05 ppm (m, 20H); ^{13}C NMR (CD_2Cl_2, 75 MHz): δ = 146.8, 143.6, 141.9, 130.1, 127.6, 108.9 ppm; FD-MS (8 kV): *m/z* (%): calcd: 634.3; found: 634.7 (100); HRMS (EI): calcd for $C_{30}H_{20}I_2$: 633.9654 $[M]^+$; found: 633.9629.

3b: The reaction was carried out according to *TP A* with 1,4-dichloro-2,3,5,6-tetrabromobenzene (1.85 g, 4.0 mmol), 4-octylphenyl magnesium bromide (32.0 mmol), and I_2 (6.35 g, 25.0 mmol). Standard workup and purification by flash chromatography (SiO_2, *n*-hexane/benzene = 30:1) yielded **3b** (2.2 g). Yield = 50%. ^{1}H NMR (CD_2Cl_2, 250 MHz): δ = 7.05 (d, *J* = 8.1 Hz, 8H), 6.98 (d, *J* = 8.1 Hz, 8H), 2.75 (t, *J* = 7.6 Hz, 8H), 1.47 (m, 48H), 0.96 ppm (t, *J* = 6.9 Hz, 12H); ^{13}C NMR (CD_2Cl_2, 75 MHz): δ = 146.8, 143.6, 141.9, 130.1, 127.6, 108.9, 35.9, 32.3, 31.6, 29.8, 29.7, 29.5, 23.1, 14.3 ppm; FD-MS (8 kV): *m/z* (%): calcd: 1083.1; found: 1083.2 (100); HRMS (ESI): calcd for $C_{62}H_{84}I_2$: 1082.4662 $[M]^+$; found: 1082.4663.

3c: The reaction was carried out according to *TP A* with 1,4-dichloro-2,3,5,6-tetrabromobenzene (4.16 g, 9.0 mmol), 4-dodecylphenyl magnesium bromide (72.0 mmol), and I_2 (12.7 g, 50.0 mmol). Standard workup and purification by flash chromatography (SiO_2, *n*-hexane/benzene = 30:1) yielded **3c** (7.1 g). Yield = 60%. ^{1}H NMR (CD_2Cl_2, 250 MHz): δ = 6.81 (d, *J* = 7.7 Hz, 8H), 6.75 (d, *J* = 7.6 Hz, 8H), 2.43 (t, *J* = 7.2 Hz, 8H), 1.24 (m, 80H), 0.72 (t, *J* = 6.6 Hz, 12H). ^{13}C NMR (CD_2Cl_2, 75 MHz): δ = 146.9, 143.7, 142.0, 130.2, 127.7, 109.0, 35.9, 32.4, 31.7, 30.2, 30.1, 29.9, 29.8, 29.6, 23.1, 14.3. FD-MS (8 kV): *m/z* (%): calcd: 1307.6; found: 1307.4 (100); HRMS (ESI): calcd for $C_{78}H_{116}I_2$: 1306.7166 $[M]^+$; found: 1306.7169.

*Typical Procedure for the Formation of HPB Analogues of Type **4** (TP B)*

An oven-dried 100-mL Schlenk tube equipped with a magnetic stirrer bar and a septum was charged with a suspension of 1,4-diiodo-2,3,5,6-tetraarylbenzene (**3**) (0.5 mmol, 1.0 equiv), arylboronic acid (1.5 mmol, 3.0 equiv), K_2CO_3 (10 mmol, 20 equiv), and aliquat 336 (0.01 mmol, 0.02 equiv) in toluene (15 mL). The mixture was degassed by three "freeze–pump–thaw" cycles, and then $[Pd(PPh_3)_4]$ (0.025 mmol, 0.05 equiv) was added. The resulting mixture was degassed again by three "freeze–pump–thaw" cycles. The mixture was warmed to the required temperature and stirred for the required time under argon. The reaction mixture was quenched with water and extracted with $CHCl_3$. The organic extract was washed with brine, dried over $MgSO_4$, and concentrated in vacuo. Flash chromatographic purification on silica gel furnished the desired product.

4a: Prepared according to TP B from **3a** (100 mg, 0.158 mmol, 1.0 equiv), phenylboronic acid (58 mg, 0.47 mmol, 3.0 equiv), K_2CO_3 (441 mg, 3.2 mmol, 20 equiv), aliquat 336 (1.0 mg, 3.3 μmol, 0.020 equiv), $[Pd(PPh_3)_4]$ (9.1 mg, 7.9 μmol, 0.05 equiv) at 100°C for 12 h up to give **4a** (80 mg, 95%) after workup. ^{1}H NMR (CD_2Cl_2, 250 MHz): δ = 7.05 (m, 30H). ^{13}C NMR (CD_2Cl_2, 75 MHz): δ = 140.6, 142.0, 131.4, 126.6, 125.2 ppm; FD-MS (8 kV): *m/z* (%): calcd: 534.7; found: 534.9 (100).

4b: Prepared according to TP B from **3b** (162 mg, 0.15 mmol, 1.0 equiv), phenylboronic acid (55 mg, 0.45 mmol, 3.0 equiv), K_2CO_3 (414 mg, 3.0 mmol, 20 equiv), aliquat 336 (1.0 mg, 3.0 μmol, 0.020 equiv), $[Pd(PPh_3)_4]$ (9.0 mg, 7.5 μmol, 0.050 equiv) at 100°C for 12 h to give **4b** (137 mg, 93%) after workup. ^{1}H NMR (CD_2Cl_2, 250 MHz): δ = 6.76 (m, 10H), 6.62 (d, *J* = 8.1 Hz, 8H), 6.56 (d, *J* = 8.2 Hz, 8H), 2.26 (t, *J* = 7.5 Hz, 8H), 1.14 (m, 48H), 0.79 ppm (t, *J* = 7.0 Hz, 12H); ^{13}C NMR (CD_2Cl_2, 75 MHz): δ = 141.9, 141.1, 141.0, 140.3, 138.9, 132.2, 132.0, 127.2, 127.1, 125.7, 35.9, 32.7, 31.9, 30.2, 30.1, 29.6, 23.5, 14.7 ppm; FD-MS (8 kV): *m/z* (%): calcd: 983.5; found: 984.3 (100); HRMS (EI): calcd for $C_{74}H_{95}$: 983.7434 $[M+H]^+$; found: 983.7419.

4c: Prepared according to TP B from **3c** (196 mg, 0.15 mmol, 1.0 equiv), phenylboronic acid (55 mg, 0.45 mmol, 3.0 equiv), K_2CO_3 (414 mg, 3.0 mmol, 20 equiv), aliquat 336 (1.0 mg, 3.0 μmol, 0.020 equiv), $[Pd(PPh_3)_4]$ (9.0 mg, 7.5 μmol, 0.050 equiv) at 100°C for 12 h to give **4c** (167 mg, 92%) after work-up. ^{1}H NMR (CD_2Cl_2, 250 MHz): δ = 6.76 (m, 10H), 6.62 (d, *J* = 8.0 Hz, 8H), 6.56 (d, *J* = 8.2 Hz, 8H), 2.26 (t, *J* = 7.4 Hz, 8H), 1.18 (m, 80H), 0.80 ppm (t, *J* = 6.9 Hz, 12H); ^{13}C NMR (CD_2Cl_2, 75 MHz): δ = 141.9, 141.1, 141.0, 140.3, 138.9, 132.2, 132.0, 127.2, 127.1, 125.7, 36.0, 32.7, 32.0, 30.5, 30.4, 30.3, 30.2, 29.6, 23.5, 14.7 ppm; FD-MS (8 kV): *m/z* (%): calcd: 1208.0; found: 1208.2 (100); HRMS (ESI): calcd for $C_{90}H_{126}$: 1206.9860 $[M]^+$; found: 1206.9871.

4d: Prepared according to TP B from **3a** (200 mg, 0.32 mmol, 1.0 equiv), 4-trimethylsilanylphenylboronic acid (183 mg, 0.95 mmol, 3.0 equiv), K_2CO_3 (871 mg, 6.31 mmol, 20 equiv), aliquat 336 (2.5 mg, 6.3 μmol, 0.020 equiv), $[Pd(PPh_3)_4]$ (18.2 mg, 0.0158 mmol, 0.050 equiv) at 100°C for 12 h to give **4d** (199 mg, 93%) after work-up. ^{1}H NMR ($CDCl_3$, 250 MHz): δ = 6.89 (d, *J* = 7.8 Hz, 4H), 6.73 (m, 20H), 6.69 (d, *J* = 8.0 Hz, 4H), 0.00 ppm (s, 18H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 142.2, 141.8, 141.6, 141.5, 137.7, 132.7, 132.6, 131.9, 127.7, 125.3, 0.00 ppm; FD-MS (8 kV): *m/z* (%): calcd: 679.0; found: 678.8 (100%); HRMS (ESI): calcd for $C_{48}H_{46}Si_2$: 678.3138 $[M]^+$; found: 678.3147.

4e: Prepared according to TP B from **3a** (200 mg, 0.32 mmol, 1.0 equiv), 3-cyanophenylboronic acid (140 mg, 0.95 mmol, 3.0 equiv), K_2CO_3 (871 mg, 6.31 mmol, 20 equiv), aliquat 336 (2.5 mg, 6.3 μmol, 0.020 equiv), $[Pd(PPh_3)_4]$ (18.2 mg, 0.0158 mmol, 0.050 equiv) at 100°C for 12 h to give **4e** (160 mg, 87%) after work-up. ^{1}H NMR ($CDCl_3$, 250 MHz): δ = 7.08 (s, 2H), 7.05 (t, *J* = 7.3 Hz, 2H), 6.90 (t, *J* = 7.3 Hz, 4H), 6.81 ppm (m, 22H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 142.3, 140.8, 140.0, 139.3, 135.9, 134.9, 131.6, 131.4, 129.6, 127.9, 127.4, 127.2, 126.2, 119.0, 111.2 ppm; FD-MS (8 kV): *m/z* (%): calcd: 584.2; found: 584.5 (100); HRMS (ESI): calcd for $C_{44}H_{28}N_2$: 584.2252 $[M]^+$; found: 584.2241.

4f: Prepared according to TP B from **3a** (2.54 g, 4.0 mmol, 1.0 equiv), 4-methoxycarbonylphenylboronic acid (2.16 g, 12.0 mmol, 3.0 equiv), K_2CO_3 (11.0 g, 80.0 mmol, 20 equiv), aliquat 336 (32.3 mg, 0.08 mol, 0.020 equiv), $[Pd(PPh_3)_4]$ (231 mg, 0.2 mol, 0.050 equiv) at 110°C for 24 h to give **4f** (2.11 g, 81%) after work-up. ^{1}H NMR (CD_2Cl_2, 250 MHz): δ = 7.43 (d, *J* = 8.3 Hz, 4H), 6.88 (d, *J* = 8.3 Hz, 4H), 6.79 (m, 20H), 3.66 ppm (s, 6H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 167.5, 146.5, 140.9, 132.2, 132.0, 132.0, 128.5, 127.6, 127.5, 127.4, 126.3, 52.5 ppm; FD-MS (8 kV): *m/z* (%): calcd: 650.8; found: 649.9 (100); HRMS (EI): calcd for $C_{46}H_{34}O_4$: 650.2457 $[M]^+$; found: 650.2439.

4g: Prepared according to TP B from **3c** (196 mg, 0.15 mmol, 1.0 equiv), 4-bromophenylboronic acid (90 mg, 0.45 mmol, 3.0 equiv), K_2CO_3 (414 mg, 3.0 mmol, 20 equiv), aliquat 336 (1.0 mg, 3.0 μmol, 0.020 equiv), $[Pd(PPh_3)_4]$ (9.0 mg, 7.5 μmol, 0.050 equiv) at 80°C for 18 h to give **4g** (186 mg, 91%) after work-up. ^{1}H NMR ($CDCl_3$, 250 MHz): δ = 6.88 (d, *J* = 8.4 Hz, 4H), 6.62 (d, *J* = 8.4 Hz, 4H), 6.60 (s, 16H), 2.29 (t, *J* = 7.5 Hz, 8H), 1.18 (m, 80H), 0.80 ppm (t, *J* = 6.9 Hz, 12H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 140.7, 140.6, 140.3, 139.7, 138.0, 133.5, 131.5, 129.9, 127.1, 119.5, 35.6, 32.3, 31.6, 30.2, 30.1, 30.0, 29.9, 29.8, 29.2, 23.1, 14.3 ppm. FD-MS (8 kV): *m/z* (%): calcd: 1365.7; found:1365.6 (100).

4h: Prepared according to TP B from **3c** (2.6 g, 2.0 mmol, 1.0 equiv), 4-methoxyphenylboronic acid (0.91 g, 6.0 mmol, 3.0 equiv), K_2CO_3 (5.5 g, 40 mmol, 20 equiv), aliquat 336 (14 mg, 0.04 mol, 0.020 equiv), $[Pd(PPh_3)_4]$ (120 mg, 0.2 mmol, 0.050 equiv) at 100°C for 12 h to give **4h** (2.31 g, 91%) after work-up. ^{1}H NMR (CD_2Cl_2, 250 MHz): δ = 6.68 (m, 20H), 6.40 (d, *J* = 8.8 Hz, 4H), 3.59 (s, 6H), 2.37 (t, *J* = 6.8 Hz, 8H), 1.28 (m, 80H), 0.88 ppm (t, *J* = 6.9 Hz, 12H); ^{13}C NMR (CD_2Cl_2, 75 MHz): δ = 157.3, 140.9, 140.2, 139.8, 138.7, 133.9, 132.8, 131.6, 126.9, 112.2, 55.2, 35.6, 32.3, 31.6, 30.09, 30.06, 30.0, 29.9, 29.82, 29,76, 29.3, 23.1, 14.3 ppm; FD-MS (8 kV): *m/z* (%): calcd: 1268.1; found: 1267.6 (100); elemental analysis: calcd (%) for $C_{92}H_{130}O_2$: C 87.14, H 10.13; found: C 87.03, H 10.01.

4i: Prepared according to TP B from **3a** (200 mg, 0.32 mmol, 1.0 equiv), 3-methoxyphenylboronic acid (144 mg, 0.95 mmol, 3.0 equiv), K_2CO_3 (871 mg, 6.31 mmol, 20 equiv), aliquat 336 (2.5 mg, 6.3 μmol, 0.020 equiv), $[Pd(PPh_3)_4]$ (18.2 mg, 0.0158 mmol, 0.050 equiv) at 100°C for 12 h to give **4i** (182 mg, 94%) after work-up. ^{1}H NMR (CD_2Cl_2, 250 MHz): δ = 6.91–6.88 (m, 20H), 6.77 (t, *J* = 7.8 Hz, 2H), 6.47–6.38 (m, 6H), 3.48 ppm (s, 6H); ^{13}C NMR (CD_2Cl_2, 62.5 Hz): δ = 158.6, 142.3, 141.2, 141.1, 140.7, 140.6, 140.5, 131.7, 131.7, 131.6, 131.5, 127.8, 126.9, 125.7, 125.6, 125.6, 124.6, 124.5, 117.2, 111.9, 55.3 ppm; FD-MS (8 kV):

m/*z* (%): calcd: 594.7; found: 595.1 (100); HRMS (ESI): calcd for $C_{44}H_{35}O_2$: 595.2637 $[M+H]^+$; found: 595.2618.

4j: Prepared according to TP B from **3a** (200 mg, 0.32 mmol, 1.0 equiv), 2-methoxyphenylboronic acid (144 mg, 0.95 mmol, 3.0 equiv), K_2CO_3 (871 mg, 6.31 mmol, 20 equiv), aliquat 336 (2.5 mg, 6.3 μmol, 0.020 equiv), $[Pd(PPh_3)_4]$ (18.2 mg, 0.0158 mmol, 0.050 equiv) at 120 °C for 18 h. After chromatographic purification on silica gel, two isomers (tentatively assigned as *anti*-**4j** and *syn*-**4j**) were readily obtained. The total yield is 74% (80 mg of *anti*-**4j** and 60 mg of *syn*-**4j**).

For *anti*-**4j**: ^{1}H NMR ($CDCl_3$, 250 MHz): $\delta = 6.89$–6.74 (m, 24H), 6.43 (t, $J = 7.5$ Hz, 2H), 6.34 (d, $J = 8.1$ Hz, 2H), 3.42 ppm (s, 6H); ^{13}C NMR ($CDCl_3$, 75 MHz): $\delta = 156.9$, 141.1, 137.4, 132.8, 131.9, 131.3, 131.0, 130.4, 127.9, 126.7, 126.5, 125.6, 119.4, 109.8, 55.1 ppm FD-MS (8 kV): *m*/*z* (%): calcd: 594.7, found: 594.4 (100); HRMS (EI): calcd for $C_{44}H_{35}O_2$: 595.2637 $[M+H]^+$; found: 595.2618.

For *syn*-**4j**: ^{1}H NMR ($CDCl_3$, 250 MHz): $\delta = 6.92$–6.72 (m, 24H), 6.44 (t, $J = 8.0$ Hz, 2H), 6.32 (d, $J = 8.1$ Hz, 2H), 3.47 ppm (s, 6H); ^{13}C NMR ($CDCl_3$, 75 MHz): $\delta = 157.3$, 141.7, 137.8, 133.6, 131.8, 131.4, 131.0, 130.7, 128.4, 127.1, 126.8, 126.0, 119.8, 110.3, 55.7 ppm; FD-MS (8 kV): *m*/*z* (%): calcd: 594.7; found: 594.3 (100); HRMS (EI): calcd for $C_{44}H_{35}O_2$: 595.2637 $[M+H]^+$; found: 595.2618.

4k: Prepared according to TP B from **3a** (200 mg, 0.32 mmol, 1.0 equiv), 3-thiopheneboronic acid (122 mg, 0.95 mmol, 3.0 equiv), K_2CO_3 (871 mg, 6.31 mmol, 20 equiv), aliquat 336 (2.5 mg, 6.3 μmol, 0.020 equiv), $[Pd(PPh_3)_4]$ (18.2 mg, 0.0158 mmol, 0.050 equiv) at 100 °C for 12 h to give **4k** (152 mg, 88%) after work-up. ^{1}H NMR (CD_2Cl_2, 250 MHz): $\delta = 6.97$–6.87 (m, 20H), 6.81–6.78 (m, 2H), 6.53–6.48 ppm (m, 4H); ^{13}C NMR (CD_2Cl_2, 62.5 Hz): $\delta = 141.6$, 141.4, 141.3, 141.2, 141.0, 140.8, 140.7, 135.9, 131.7, 131.4, 131.1, 130.9, 127.2, 127.0, 127.0, 125.9, 125.8, 125.6, 125.0, 123.0 ppm; FD-MS (8 kV): *m*/*z* (%): calcd: 545.7; found: 546.1 (100); HRMS (ESI): calcd for $C_{38}H_{26}S_2Na$: 569.1395 $[M+Na]^+$; found: 569.1359.

4l: Prepared according to TP B from **3a** (200 mg, 0.32 mmol, 1.0 equiv), 3-benzofuranboronic acid (155 mg, 0.95 mmol, 3.0 equiv), K_2CO_3, (871 mg, 6.31 mmol, 20 equiv), aliquat 336 (2.5 mg, 6.3 μmol, 0.020 equiv), $[Pd(PPh_3)_4]$ (18.2 mg, 0.0158 mmol, 0.050 equiv) at 100 °C for 12 h to give **4l** (147 mg, 76%) after work-up. ^{1}H NMR (CD_2Cl_2, 250 MHz): $\delta = 7.13$ (m, 4H), 6.97 (m, 10H), 6.91 (m, 4H), 6.85 (m, 10H), 5.98 ppm (s, 2H); ^{13}C NMR (CD_2Cl_2, 62.5 MHz): $\delta = 154.4$, 154.2, 142.6, 139.9, 132.3, 130.7, 128.5, 127.2, 126.4, 123.8, 122.6, 120.8, 110.8, 108.4 ppm; FD-MS (8 kV): *m*/*z* (%): calcd: 614.7; 614.6 (100); HRMS (ESI): calcd for $C_{46}H_{30}O_2$: 615.2324 $[M+H]^+$; found: 615.2306.

Typical Procedure for the Formation of Compounds of Type ***5*** *(TP C)*

An oven-dried 100-mL Schlenk tube equipped with a magnetic stirrer bar and a septum was charged with a suspension of 1,4-diiodo-2,3,5,6-tetraphenylbenzene (**3a**) (1.0 mmol, 1.0 equiv), arylboronic acid (1.0 mmol, 1.0 equiv), K_2CO_3 (20 mmol, 20 equiv), and aliquat 336 (0.02 mmol, 0.02 equiv) in toluene (20 mL). The mixture was degassed by three "freeze–pump–thaw" cycles, and $[Pd(PPh_3)_4]$ (0.05 mmol, 0.05 equiv) was then added. The resulting mixture was degassed again by three "freeze–pump–thaw" cycles. The mixture was warmed to 80 °C and stirred for the required time under argon. The reaction mixture was quenched with water and extracted with $CHCl_3$. The organic extract was dried over $MgSO_4$ and concentrated in vacuo. Flash chromatographic purification on silica gel furnished the desired product.

5a: Prepared according to TP C from **3a** (634 mg, 1.0 mmol, 1.0 equiv), 4-methoxycarbonylphenylboronic acid (122 mg, 1.0 mmol, 1.0 equiv), K_2CO_3 (2.76 g, 20 mmol, 20 equiv), aliquat 336 (8.1 mg, 0.020 mmol, 0.020 equiv), $[Pd(PPh_3)_4]$ (58 mg, 0.050 mmol, 0.050 equiv) at 80 °C for 12 h to give **5a** (386 mg, 60%) after work-up. ^{1}H NMR (CD_2Cl_2, 250 MHz): $\delta = 7.42$ (d, $J = 8.0$ Hz, 2H), 7.06 (m, 10H), 6.87 (d, $J = 8.0$ Hz, 2H), 6.76 (m, 10H), 3.66 ppm (s, 3H); ^{13}C NMR (CD_2Cl_2, 62.5 MHz): $\delta =$ 154.4, 154.2, 142.6, 139.9, 132.3, 130.7, 128.5, 127.2, 126.4, 123.8, 122.6, 120.8, 110.8, 108.4 ppm; FD-MS (8 kV): *m*/*z* (%): calcd: 642.5; found: 642.2 (100); HRMS (ESI): calcd for $C_{38}H_{27}IO_2$: 643.1134 $[M+H]^+$; found: 643.1109.

5b: Prepared according to TP C from **3a** (634 mg, 1.0 mmol, 1.0 equiv), 4-methoxyphenylboronic acid (152 mg, 1.0 mmol, 1.0 equiv), K_2CO_3 (2.76 g, 20.0 mmol, 20 equiv), aliquat 336 (8.1 mg, 0.020 mol, 0.020 equiv), $[Pd(PPh_3)_4]$ (58.0 mg, 0.050 mmol, 0.050 equiv) at 80 °C for 12 h to give **5b** (319 mg, 52%) after work-up. ^{1}H NMR ($[D_8]$THF, 250 MHz): $\delta = 7.16$ (m, 10H), 6.88 (m, 10H), 6.43 (d, $J = 8.62$ Hz, 2H), 3.58 ppm (s, 3H); ^{13}C NMR ($[D_8]$THF, 75 MHz): $\delta = 156.5$, 145.5, 145.1, 145.0, 139.8, 132.1, 131.1, 130.1, 129.6, 126.8, 126.5, 125.8, 125.7, 124.7, 111.1, 54.0 ppm; FD-MS (8 kV): *m*/*z* (%): calcd: 613.8; found: 614.1 (100); HRMS (EI): calcd for $C_{37}H_{27}IO$: 614.1107 $[M]^+$; found: 614.1111.

5c: Prepared according to TP C from **3a** (634 mg, 1.0 mmol, 1.0 equiv), 3-thiopheneboronic acid (128 mg, 1.0 mmol, 1.0 equiv), K_2CO_3 (2.76 mg, 20.0 mmol, 20 equiv), aliquat 336 (8.1 mg, 0.020 mol, 0.020 equiv), $[Pd(PPh_3)_4]$ (57.8 mg, 0.050 mol, 0.050 equiv) at 80 °C for 12 h to give **5c** (295 mg, 50%) after work-up. ^{1}H NMR ($CDCl_3$, 250 MHz): $\delta = 7.42$ (d, $J = 8.0$ Hz, 2H), 7.06 (m, 10H), 6.87 (d, $J = 8.0$ Hz, 2H), 6.76 (m, 10H), 3.66 ppm (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz): $\delta = 154.4$, 154.2, 142.6, 139.9, 132.3, 130.7, 128.5, 127.2, 126.4, 123.8, 122.6, 120.8, 110.8, 108.4 ppm; FD-MS (8 kV): *m*/*z* (%): calcd: 642.5; found: 642.2 (100), HRMS (EI): calcd for $C_{34}H_{23}IS$: 590.0565 $[M]^+$; found: 590.0563.

6a: Prepared according to TP B from **5a** (150 mg, 0.23 mmol, 1.0 equiv), 4-methoxyphenylboronic acid (52 mg, 0.35 mmol, 1.5 equiv), K_2CO_3 (644 mg, 4.7 mmol, 20 equiv), aliquat 336 (1.9 mg, 4.7 μmol, 0.020 equiv), $[Pd(PPh_3)_4]$ (13.5 mg, 0.012 μmol, 0.050 equiv) at 100 °C for 12 h to give **6a** (135 mg, 93%) after work-up. ^{1}H NMR ($[D_8]$THF, 250 MHz): $\delta = 7.60$ (d, $J = 8.2$ Hz, 2H), 7.05 (d, $J = 8.3$ Hz, 2H), 6.93 (m, 20H), 6.83 (d, $J =$ 8.6 Hz, 2H), 6.50 (d, $J = 8.7$ Hz, 2H), 3.82 ppm (s, 3H), 3.64 (s, 3H); ^{13}C NMR ($[D_8]$THF, 75 MHz): $\delta = 167.6$, 159.2, 147.8, 143.0, 142.7, 142.4, 142.2, 142.0, 141.8, 141.3, 134.2, 134.0, 133.2, 133.1, 129.4, 128.8, 128.3, 128.2, 127.0, 126.8, 113.8, 55.8, 52.5 ppm; FD-MS (8 kV): *m*/*z* (%): calcd: 621.5; found: 622.7 (100); HRMS (EI): calcd for $C_{45}H_{35}O_3$: 623.2586 $[M+H]^+$; found: 623.2568.

6b: Prepared according to TP B from **5a** (150 mg, 0.23 mmol, 1.0 equiv), 3-thiophenephenylboronic acid (45 mg, 0.35 mmol, 1.5 equiv), K_2CO_3 (644 mg, 4.7 mmol, 20 equiv), aliquat 336 (1.9 mg, 4.7 μmol, 0.020 equiv), $[Pd(PPh_3)_4]$ (13.5 mg, 0.012 μmol, 0.050 equiv) at 100 °C for 12 h to give **6b** (127 mg, 91%) after work-up. ^{1}H NMR ($[D_8]$THF, 250 MHz): $\delta = 7.57$ (dd, $J = 8.2$ Hz, $J = 1.6$ Hz, 2H), 7.00 (dd, $J = 8.2$ Hz, $J = 2.0$ Hz, 2H), 6.93 (m, 10H), 6.88 (m, 11H), 6.59 (d, $J = 2.6$ Hz, 1H), 6.50 (d, $J = 4.9$ Hz, 1H), 3.78 ppm (s, 3H); ^{13}C NMR ($[D_8]$THF, 75 MHz): $\delta = 167.6$, 147.7, 143.2, 142.8, 142.5, 142.217, 142.1, 141.5, 137.7, 133.2, 133.1, 132.8, 132.0, 129.5, 128.9, 128.5, 128.4, 128.3, 127.1, 126.4, 124.5, 52.6 ppm; FD-MS (8 kV): *m*/*z* (%): calcd: 598.1; found: 597.8 (100); HRMS (ESI): calcd for $C_{42}H_{30}O_2S$: 599.2045 $[M+H]^+$; found: 599.2026.

6c: Prepared according to TP B from **5b** (150 mg, 0.24 mmol, 1.0 equiv), 4-trimethylsilanylphenylboronic acid (70 mg, 0.36 mmol, 1.5 equiv), K_2CO_3 (674 mg, 4.9 mmol, 20 equiv), aliquat 336 (2.0 mg, 4.9 μmol, 0.020 equiv), $[Pd(PPh_3)_4]$ (14.1 mg, 0.012 μmol, 0.050 equiv) at 100 °C for 12 h to give **6c** (137 mg, 88%) after work-up. ^{1}H NMR ($[D_8]$THF, 250 MHz): $\delta = 6.91$ (d, $J = 7.8$ Hz, 2H), 6.73 (m, 22H), 6.62 (d, $J = 8.6$ Hz, 2H), 6.29 (d, $J = 8.6$ Hz, 2H), 3.44 (s, 3H), 0.00 ppm (s, 9H); ^{13}C NMR ($[D_8]$THF, 75 MHz): $\delta = 159.4$, 143.4, 143.0, 142.8, 142.8, 142.4, 142.2, 138.1, 135.7, 134.8, 134.3, 133.4, 133.3, 132.8, 128.5, 128.4, 128.4, 128.2, 126.9, 114.0, 56.0, 0.0 ppm; FD-MS (8 kV): *m*/*z* (%): calcd: 636.2; found: 635.6 (100); HRMS (ESI): calcd for $C_{46}H_{40}OSi$: 636.2848 $[M]^+$, found: 636.2837.

6d: Prepared according to TP B from **5b** (150 mg, 0.24 mmol, 1.0 equiv), 3-cyanophenylboronic acid (53 mg, 0.36 mmol, 1.5 equiv), K_2CO_3 (674 mg, 4.9 mmol, 20 equiv), aliquat 336 (2.0 mg, 4.9 μmol, 0.020 equiv), $[Pd(PPh_3)_4]$ (14.1 mg, 0.012 μmol, 0.050 equiv) at 100 °C for 12 h to give **6d** (122 mg, 85%) after work-up. ^{1}H NMR ($[D_8]$THF, 250 MHz): $\delta = 7.22$ (s, 1H), 7.17 (d, $J = 8.0$ Hz, 2H), 7.03 (t, $J = 7.6$ Hz, 1H), 6.89 (m, 21H), 6.78 (d, $J = 8.8$ Hz, 1H), 6.45 (d, $J = 8.5$ Hz, 2H), 3.59 ppm (s, 3H); ^{13}C NMR ($[D_8]$THF, 75 MHz): $\delta = 159.2$, 144.1, 143.3, 142.8, 142.3, 140.2, 140.0, 137.3, 136.5, 134.1, 134.0, 133.1, 133.0, 133.0, 132.9, 130.6, 128.6, 128.4, 128.3, 127.2, 126.9, 119.8, 113.8, 55.7; FD-MS (8 kV): *m*/*z* (%): calcd: 589.2; found: 589.1 (100); HRMS (ESI): calcd for $C_{44}H_{31}NO$: 589.2406 $[M]^+$; found: 589.2396.

6e: Prepared according to TP B from **5c** (180 mg, 0.31 mmol, 1.0 equiv), 3-cyanophenylboronic acid (86 mg, 0.47 mmol, 1.5 equiv), K_2CO_3 (842 mg, 6.1 mmol, 20 equiv), aliquat 336 (2.5 mg, 6.1 μmol, 0.020 equiv), $[Pd(PPh_3)_4]$ (17.6 mg, 0.015 mmol, 0.050 equiv) at 100 °C for 12 h to give **6e** (148 mg, 86 %) after work-up. ^{1}H NMR ([D$_8$]THF, 250 MHz): δ = 7.20 (m, 3H), 7.06 (dd, J = 8.0 Hz, J = 1.6 Hz, 1H), 6.94 (m, 21H), 6.60 (m, 1H), 6.50 ppm (d, J = 4.9 Hz, 1H); ^{13}C NMR ([D$_8$]THF, 75 MHz): δ = 144.0, 143.4, 142.9, 142.3, 141.8, 140.3, 140.2, 137.7, 137.3, 136.4, 133.1, 132.9, 132.7, 131.9, 130.7, 129.3, 128.4, 127.3, 127.1, 126.4, 124.6, 119.7, 112.9 ppm; FD-MS (8 kV): m/z (%): calcd: 565.1; found: 564.9 (100); HRMS (ESI): calcd for $C_{41}H_{27}NS$: 566.1942 $[M+H]^+$; found: 566.1934.

7: A solution of **5g** (100 mg, 0.073 mmol, 1.0 equiv) in freshly distilled CH_2Cl_2 (100 mL) was bubbled with argon for 15 min. Then a solution of $FeCl_3$ (242 mg, 1.49 mmol, 20.4 equiv) in CH_3NO_2 (1 mL) was added slowly to the reaction solution with bubbling of argon. The resulting black solution was stirred at room temperature for 30 min with bubbling of argon, and the reaction was quenched with methanol (50 mL). The solvent was removed under reduced pressure, and the residue was purified by fast chromatography with hot toluene as eluent to give the crude product as a yellow solid. The crude product was precipitated in methanol to give the desired product **7** (84 mg, 85 %). ^{1}H NMR (50 % $CDCl_3$/CS_2, 300 MHz): δ = 7.51 (b, 4H), 7.11 (s, 8H), 2.42 (t, J = 7.4 Hz, 8H), 1.71–1.40 (m, 80H) 0.98 ppm (t, J = 6.8 Hz, 12H); ^{13}C NMR ($CDCl_3$, 75 MHz): δ = 138.8, 130.0, 127.8, 126.8, 122.6, 121.4, 121.1, 120.8, 120.2, 117.2, 116.9, 37.3, 32.8, 32.6, 30.9, 30.8, 30.7, 30.6, 30.3, 23.7, 15.0 ppm; FD-MS (8 kV): m/z (%): calcd: 1354.3; found: 1353.6 (100).

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