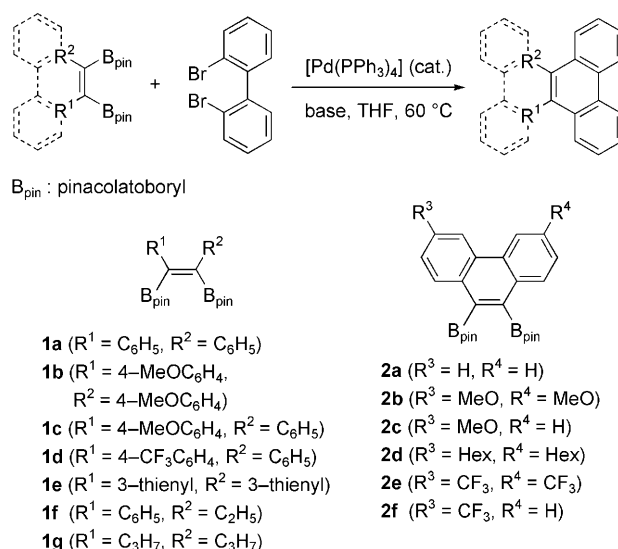


Palladium-Catalyzed Annulation of *vic*-Bis(pinacolatoboryl)alkenes and -phenanthrenes with 2,2'-Dibromobiaryls: Facile Synthesis of Functionalized Phenanthrenes and Dibenzo[*g,p*]chrysenes**

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Because of the progress in organic electronics, such as light-emitting diodes, field-effect transistors, and solar cells, there has been extensive research on polycyclic aromatic hydrocarbons (PAHs).^[1] Efficient syntheses of PAHs bearing the appropriate functional groups to control optical and electronic properties is important, and different structural motifs are essential for the exploration and improvement of materials derived from PAHs. Conventional synthetic methods for making functionalized PAHs include electrophilic or nucleophilic substitutions,^[2] cross-coupling reactions,^[3] and directed-metalation,^[4] in which the introduction of the functional groups and the substitution patterns are strongly dependent on the reactivity of the parent PAHs framework. Alternatively, formation of the aromatic ring by transition-metal-catalyzed annulation of functionalized precursors provides a more flexible and versatile synthesis for functionalized PAHs by overcoming the drawbacks of the conventional synthetic approach.^[5,6]

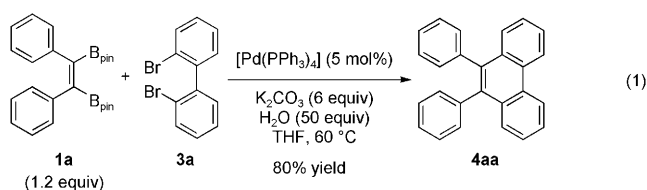
Since the cross-coupling reaction of organoboron compounds (Suzuki–Miyaura coupling) is useful for stereospecific C_{sp²–C_{sp² bond formation and is compatible with a broad range of functional groups,^[7] we focused our attention on *vic*-diborylalkenes^[8] as potential precursors for our annulation approach.^[9] Various *vic*-diborylated alkenes have been converted into multisubstituted acyclic alkenes by stepwise cross-coupling reactions with two organic halides,^[10] whereas the double cross-coupling reaction of *vic*-diborylated compounds with aromatic dihalides as a synthetic method for functionalized PAHs remains unexplored.^[11–13] We report herein a palladium-catalyzed double cross-coupling reaction of *vic*-diborylated alkenes (**1**) and phenanthrenes (**2**) with 2,2'-dibromobiaryl compounds. The annulation provides a convenient synthesis of a variety of phenanthrenes and dibenzo[*g,p*]chrysenes, as well as dithienobenzenes and}}



Scheme 1. Palladium-catalyzed double cross-coupling reaction of *vic*-diborylated alkenes (**1**) and phenanthrenes (**2**) with 2,2'-dibromobiaryl compounds.

triphenylene[1,2-*b*:4,3-*b'*]dithiophene, in good to excellent yields (Scheme 1).

Initially, we chose (*Z*)-1,2-bis(pinacolatoboryl)stilbene (**1a**) as a model *vic*-diborylalkene and 2,2'-dibromobiphenyl (**3a**) as a coupling partner [Eq. (1)]. Initial attempts revealed



that protodeborylation of **1a** was a major side reaction, presumably resulting from the steric hindrance of both **1a** and **3a**. After screening various reaction conditions,^[14] the desired 9,10-diphenylphenanthrene (**4aa**)^[15] was isolated in 80% yield using [Pd(PPh₃)₄] (5 mol%), K₂CO₃ (6 equiv), and H₂O (50 equiv) in THF at 60 °C for 24 hours. The presence of water was crucial for the reaction.^[16] The optimized conditions were applicable to gram-scale preparation of the annulated rings,^[17] and neither oligomers nor polymers were detected.

The scope of the annulation reaction leading to functionalized phenanthrenes (**4**) is summarized in Table 1. The

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Table 1: Synthesis of functionalized phenanthrenes (**4**).^[a]

Entry	1	Dibromide 3	Product 4 (yield) ^[b]
1	1a	3b (R = Me)	4ab (85%)
2	1a	3c (R = OMe)	4ac (82%)
3	1a	3d	4ad (65%)
4	1a	3e	4ae (32%)
5	1a	3f	4af (84%)
6	1b	3a	4ba (92%)
7	1b	3d	4bd (85%)
8	1c	3a	4ca (81%)
9	1d	3a	4da (77%)
10	1e	3d	4ed (90%)
11 ^[c]	1f	3a	4fa (99%)
12 ^[d]	1g	3a	4ga (81%)
13 ^[e]	1g	3g	4gg (57%)

[a] Reaction conditions: **1** (1.2 mmol), **3** (1.0 mmol), [Pd(PPh₃)₄] (58 mg, 0.05 mmol), and K₂CO₃ (0.83 g, 6.0 mmol), H₂O (0.90 mL, 50 mmol), THF (20 mL), 60 °C, 48 h. [b] Yield of the isolated product. [c] 3 M aq K₃PO₄ (6 equiv) was used in place of K₂CO₃/H₂O. [d] 3 M aq NaOH (6 equiv) was used in place of K₂CO₃/H₂O. [e] [PdCl₂(dppf)] (5 mol%) and 3 M aq K₃PO₄ (6 equiv) was used in place of [Pd(PPh₃)₄] and K₂CO₃/H₂O, respectively. dppf = 1,1'-bis(diphenylphosphanyl)ferrocene.

tetramethyl- and tetramethoxy-substituted dibromobiphenyls **3b** and **3c** were coupled with **1a** to give **4ab** and **4ac**, respectively, in high yields (Table 1, entries 1 and 2). The reaction of 2,2'-dibromooctafluorobiphenyl (**3d**) with **1a** also

proceeded smoothly under the optimized reaction conditions to produce octafluorinated phenanthrene **4ad** in 65% yield (Table 1, entry 3). When 2,2'-dibromobinaphthyl (**3e**) and bithiophene **3f** were used with **1a**, [5]helicene **4ae** and dithieno[1,2-6:4,3-6']benzene **4af** were obtained, respectively (Table 1, entries 4 and 5). Substituted diborylstilbenes **1b–d** also underwent the annulation to give both symmetrical and unsymmetrical phenanthrenes **4ba**, **4bd**, **4ca**, and **4da** in high yields (Table 1, entries 6–9). Additionally, diboryldithienylethene **1e**, 1,2-diboryl-1-phenylbut-1-ene **1f**, and 4,5-diboryl-oct-4-ene **1g** reacted with **3** to give annulated products **4ed**, **4fa**, **4ga**, and **4gg** (Table 1, entries 10–13); a slight modification to the reaction conditions was necessary in cases of **1f** and **1g**.^[18]

Next we examined the annulation of *vic*-diborylated arenes such as **2**, which were readily prepared by photocyclization of **1** and subsequent in situ oxidation.^[14] By using the optimized conditions determined for the annulation of **1**, the reaction of **2a** with **3a** gave dibenzo[*g,p*]chrysene **5aa** in 68% yield. After re-screening the reaction conditions, 3 M aq. K₃PO₄ was found to be the best base for this system, producing **5aa** in a quantitative yield. The scope of the annulation reaction leading to functionalized dibenzo[*g,p*]chrysenes (**5**) is summarized in Table 2. Various dibenzo[*g,p*]chrysenes (**5**) substituted with electron-donating and electron-withdrawing groups are not easily accessible by previous methods,^[19] but they were isolated in moderate to good yields by using our annulation chemistry. Notably, fluorinated dibenzo[*g,p*]chrysenes **5ad**, **5bd**, **5cd**, **5dd**, and **5fd** were generally produced in high to excellent yields (Table 2, entries 3, 6, 8, 9, and 11). The annulation was also

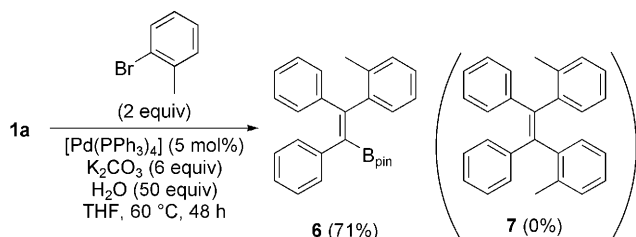
Table 2: Synthesis of functionalized dibenzo[*g,p*]chrysenes (**5**).^[a]

Entry	2	3	5	Yield [%] ^[b]
1	2a	3a	5aa	> 99
2 ^[c]	2a	3c	5ac	54
3	2a	3d	5ad	63
4	2a	3f	5af	88
5	2b	3a	5ba	64
6	2b	3d	5bd	94
7	2c	3a	5ca	61
8	2c	3d	5cd	80
9	2d	3d	5dd	82
10	2e	3c	5ec	40
11 ^[d]	2f	3d	5fd	96

[a] Reaction conditions: **2** (0.06 mmol), **3** (0.05 mmol), [Pd(PPh₃)₄] (2.9 mg, 2.5 μmol), and 3 M aq K₃PO₄ (0.1 mL, 0.3 mmol), THF (1.0 mL), 60 °C, 48 h. [b] Yield of isolated product. [c] 12 M aq K₃PO₄ (0.6 mmol) was used at 80 °C. [d] K₂CO₃ (0.3 mmol) and H₂O (2.5 mmol) were used.

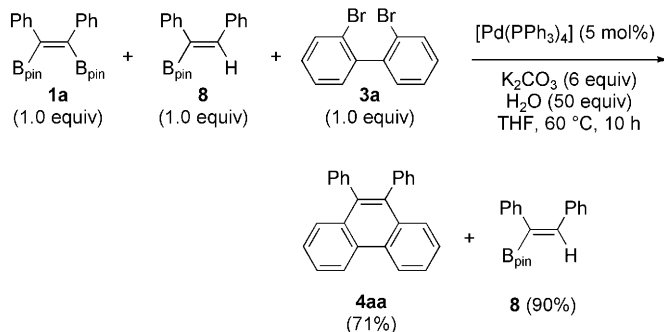
applicable to the preparation of triphenyleno[1,2-*b*:4,3-*b'*]-dithiophene (**5af**; Table 2, entry 4).

To gain mechanistic insight into the annulation reaction, we carried out the reaction of **1a** with *o*-bromotoluene (2 equiv) under the optimized conditions (Scheme 2). The only product obtained was the singly coupled product (**6**) in 71% yield; the doubly coupled product (**7**) was not observed. This result indicated that a second intermolecular transmetalation for the coupling of a second equivalent of *ortho*-substituted bromobenzene with **1a** was unfavorable. Accordingly, the use of 2,2'-dibromobiaryl compounds as the coupling partner is crucial for the success of the annulation, as it suppresses oligomerization/polymerization.



Scheme 2. Coupling reaction of **1a** with *o*-bromotoluene.

Additionally, the coupling reaction of **1a** with **3a** (1 equiv) was effected in the presence of one equivalent of pinacolatoboryl-1,2-diphenylethene (**8**) as illustrated in Scheme 3. Interestingly, **4aa** was the sole coupling product isolated



Scheme 3. Comparison of reactivity between **1a** and **8**.

in 71% yield with 90% recovery of **8**, indicating that *vic*-diborylethene was much more reactive than a monoborylethene under the reaction conditions.^[20] The higher reactivity of the *vic*-diborylated compound may play a key role in overcoming the intrinsic difficulty in the coupling reactions of sterically hindered halides with boron reagents.

In summary, we have demonstrated that the palladium-catalyzed double cross-coupling reaction of *vic*-diborylated compounds with 2,2'-dibromobiaryls provides a new annulation approach to functionalized PAHs. This methodology efficiently provides functionalized phenanthrenes and dibenzo[*g,p*]chrysenes, which have attracted attention in recent years as modules for the development of π -conjugated

materials such as luminescent polymers, thin-field effect transistors, and molecular wires.^[21] Helicene and thiophene-fused PAHs can also be synthesized easily. The results herein shed new light on the synthetic utility of *vic*-diborylated compounds in organic synthesis. The extension of the use of dihalides and diboron reagents for annulation reactions are in progress in our laboratory.

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