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Nickel-Catalyzed Cross-Coupling of Aryl Phosphates with Arylboronic Acids

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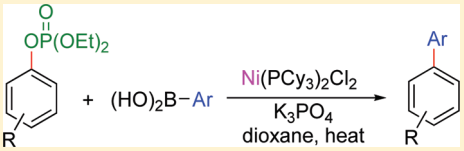
Supporting Information

ABSTRACT: The Suzuki−Miyaura cross-coupling of aryl phosphates using $Ni(PCy_3)_2Cl_2$ as an inexpensive, bench-stable catalyst is described. Broad substrate scope and high efficiency are demonstrated by the syntheses of more than 40 biaryls and by constructing complex organic molecules. The poor reactivity of aryl phosphates relative to aryl halides is successfully employed to construct polyarenes by selective cross-coupling using Pd and Ni catalysts.



Transition-metal-catalyzed cross-coupling reactions have become a cornerstone of modern synthetic chemistry.[1] Among these reactions, the Suzuki−Miyaura coupling is characterized by its mild reaction conditions, exceptionally broad functional-group tolerance, and the use of nontoxic organoboron nucleophiles.[2,3] Recent advances have significantly broadened the scope of the reaction by the use of previously unreactive substrates such as aryl chlorides,[4,5] fluorides,[6] and nitriles,[7] as well as aryl ethers,[8,9] carbonates,[9−12] carbamates,[13−15] sulfamates,[13] and phosphonium salts[16] to participate efficiently. Moreover, as a result of the intense research into the reactivity and stability of various catalyst systems, it is now possible to conduct such reactions using low catalyst loadings[17,18] and at room temperature.[19]

In addition to the above substrates, vinyl phosphates were known to undergo coupling with arylboronic acids.[20−24] Very recently, Han and co-workers have achieved the cross-coupling of phosphoramides with arylboronic acids.[25] However, the Suzuki−Miyaura coupling of aryl phosphates has not been realized up to now and has been shown to be extremely difficult.[25−27] To the best of our knowledge, metal-mediated cross-coupling involving an aryl phosphate has only been reported for a few cases. All of them employed Grignard or organoaluminum reagents as nucleophiles.[28−30] Our ongoing interest in phosphate reactivity[31−33] and in nickel- or cobalt-catalyzed cross-coupling reactions[34−36] involving arylboronic acids as substrates has led us to explore the possibility of the reaction of aryl phosphates with arylboronic acids using nickel complexes as catalysts. Herein, we describe a nickel-catalyzed cross-coupling of aryl phosphates with arylboronic acids to give various unsymmetrical biaryls. The method provides a method for the activation of the aryl−O bond and the transformation of a phenol derivative into an unsymmetrical biaryl.

We began our study by examining the reaction of 2-naphthyl phosphates **1a** with phenylboronic acid **2a**. Our initial experiments showed that Pd(0) and Pd(II) complexes were ineffective for the coupling of 2-naphthyl phosphates with phenylboronic acid. We then focused on using more reactive Ni-based catalysts, because several studies have revealed that Ni-based complexes were able to activate the C−O bonds of alkenyl phosphates,[20−24,37] aryl and benzyl ethers,[8,9,38−40] esters,[10−15,25] and salts.[41] Thus, a number of Ni-based complexes have been screened, and the results are summarized in Table 1.

$Ni(dppe)Cl_2$, $Ni(dppp)Cl_2$, $Ni(dppb)Cl_2$ and $Ni(dppf)Cl_2$, and $Ni(PPh_3)_2Cl_2$ showed poor catalytic activity, giving low yields of coupling product. Among them, $Ni(dppf)Cl_2$ was most efficient, providing **3a** in 70% yield. To improve the yield further, we used $Ni(PCy_3)_2Cl_2$ as the catalyst for the coupling reaction. Gratifyingly, the use of this complex further improved the yield to 86% at 80 °C and 93% at 110 °C (entries 6, 7). Control experiments showed that $NiCl_2$ alone afforded product **3a** in 35% yield, and PCy_3 alone was ineffective for the coupling reaction. Among the solvents and bases screened, dioxane and K_3PO_4 were the best solvent and base, respectively (entries 6−13), for the catalytic reaction. The effect of water on the cross-coupling reaction was also studied, and the results show that the addition of water to the reaction led to the decrease of the yield of **3a**.

With the optimized conditions in hand, we examined the scope of this coupling reaction first by varying the aryl group in aryl phosphates **1** (Table 2). To our satisfaction, several substituted naphthyl phosphates coupled efficiently with phenylboronic acid **2a** to provide the corresponding biaryl products in excellent isolated yields (>88%). The coupling reaction of substituted phenyl phosphates with **2a** also proceeded smoothly,

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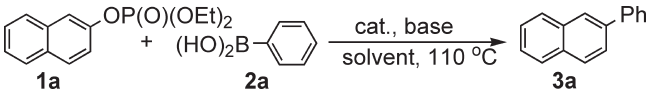
Table 1. Optimization of Reaction Conditions[a]



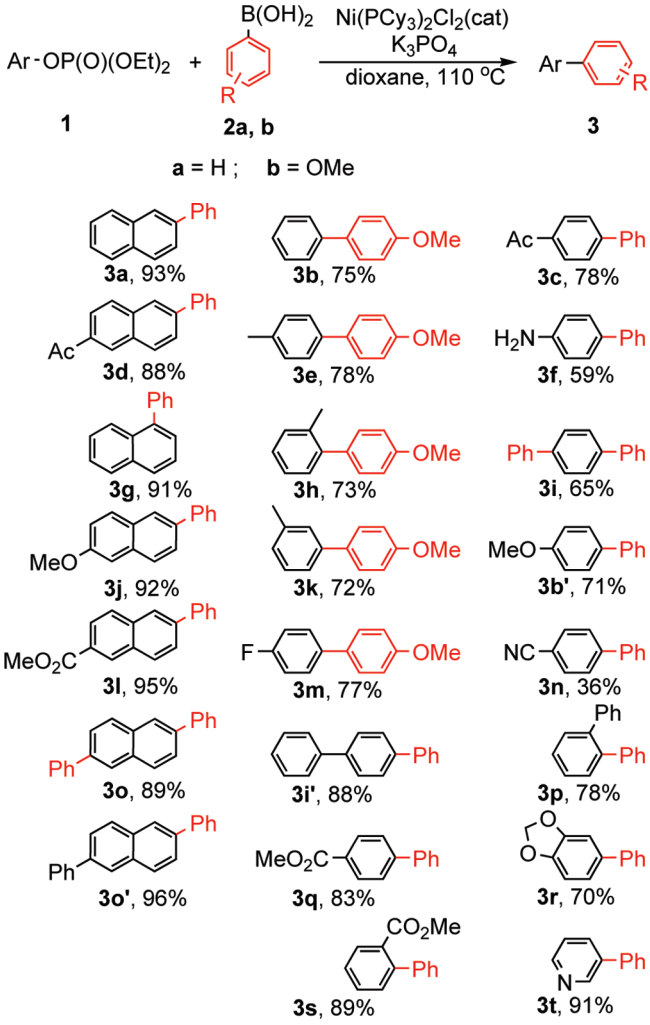
entry	catalyst	base	T (°C)	solvent	yield (%)
1	$Ni(dppe)Cl_2$	K_3PO_4	110	dioxane	38
2	$Ni(dppp)Cl_2$	K_3PO_4	110	dioxane	42
3	$Ni(dppb)Cl_2$	K_3PO_4	110	dioxane	44
4	$Ni(dppf)Cl_2$	K_3PO_4	110	dioxane	70
5	$Ni(PPh_3)_2Cl_2$	K_3PO_4	110	dioxane	20
6	**$Ni(PCy_3)_2Cl_2$**	**K_3PO_4**	**110**	**dioxane**	**93**
7	$Ni(PCy_3)_2Cl_2$	K_3PO_4	80	dioxane	86
8	$Ni(PCy_3)_2Cl_2$	K_3PO_4	80	THF	88
9	$Ni(PCy_3)_2Cl_2$	K_3PO_4	110	toluene	85
10	$Ni(PCy_3)_2Cl_2$	KOH	110	dioxane	80
11	$Ni(PCy_3)_2Cl_2$	K_2CO_3	110	dioxane	82
12	$Ni(PCy_3)_2Cl_2$	$KOBu^t$	110	dioxane	0
13	$Ni(PCy_3)_2Cl_2$	Cs_2CO_3	110	dioxane	tr

[a] Reaction conditions: 2-naphthyl diethyl phosphate (0.500 mmol), metal complex (0.050 mmol), $PhB(OH)_2$ (0.750 mmol), base (2.25 mmol), solvent (2 mL), 24 h.

affording the corresponding biaryl products in moderate to good yields (59−91%). Both electron-withdrawing and -donating groups on the aryl ring of phosphates are compatible with the reaction. For diphosphates, double arylation with **2a** was observed, providing the corresponding disubstituted products in one pot in good yields (**3i** and **3o**). Furthermore, the reactions of *ortho*-substituted phenyl phosphates including 1-naphthyl, *o*-methyl, *o*-methoxycarbonyl, and *o*-phenylphenyl phosphates proceeded smoothly to afford the corresponding coupling products in good to excellent yields. The results indicate that steric hindrance imparted by *ortho*-substitution of phenyl phosphates does not greatly affect the product yields. Finally, 3-pyridyl phosphate was also successfully employed in the coupling reaction with **2a** under the standard conditions to give 3-phenylpyridine in 91% yield (**3t**). However, the 4-cyanophenyl phosphate derivative was aberrant, giving a low yield of the corresponding product (**3n**). This is likely due to the cross-coupling at the cyano group[7] or addition to the cyano group by phenylboronic acid,[42] resulting in a mixture of products.

We next turned to the scope of arylboronic acid **2** used for the cross-coupling reaction. As shown in Table 3, arylboronic acids with electron-neutral, electron-rich, and electron-deficient substituents react with 2-naphthyl phosphate **1a** to give the expected 2-arylnaphthalenes in good to excellent yields. *Ortho*-substituted arylboronic acids appear less reactive, affording the coupling products in moderate yields (**4d**, **4o**). Various functional groups such as methoxy and fluoro groups on the aryl ring of **2** are compatible with the reaction, providing an opportunity for further functionalization of the products. It is noteworthy that 3-aminophenylboronic acid coupled successfully with **1a** to give product **4l** in an 82% yield without the need for a protecting group on nitrogen.[43] In addition, a heteroarylboronic acid also reacted smoothly with **1a** to give product **4m** in 63% yield. Finally, 4-acetylphenylboronic acid also underwent cross-coupling with **1a**, albeit in low yield (**4j**). The reaction suffered from the

Table 2. Cross-Coupling of Various Aryl Phosphates 1 with 2a or 2b[a]



[a] Reaction conditions: aryl phosphates (0.50 mmol), $Ni(PCy_3)_2Cl_2$ (0.050 mmol), $ArB(OH)_2$ (0.75 mmol), K_3PO_4 (2.25 mmol), dioxane (2.0 mL), 110 °C, 24 h.

formation of side product acetophenone as a result of protodeboronation of 4-acetylphenylboronic acid.[44]

A possible mechanism for this reaction is proposed as shown in Scheme 1. The reaction likely proceeds through a typical cross-coupling pathway[3] as follows: (1) the active catalytic species Ni(0), generated *in situ* from $Ni(PCy_3)_2Cl_2$ in the presence of arylboronic acids and K_3PO_4 undergoes oxidative addition with aryl phosphates to afford intermediate **5**;[45] (2) transmetalation of the aryl group on the activated boronic acid gives Ni(II)(Ar)(Ar′) species **6**; (3) C−C bond formation *via* reductive elimination gives the cross-coupling product and regenerates the catalytic active Ni(0) species. An alternative mechanism via Ni(I)/Ni(III) intermediates cannot be totally excluded.[46,47]

To probe the scope and utility of the phosphates cross-coupling method further, we applied the present method to the construction of complicated organic scaffolds (Scheme 2). Starting from natural product estrone, a 4-anisyl group was introduced to the compound to afford product **8** through the newly developed coupling. This illustrated a straightforward way

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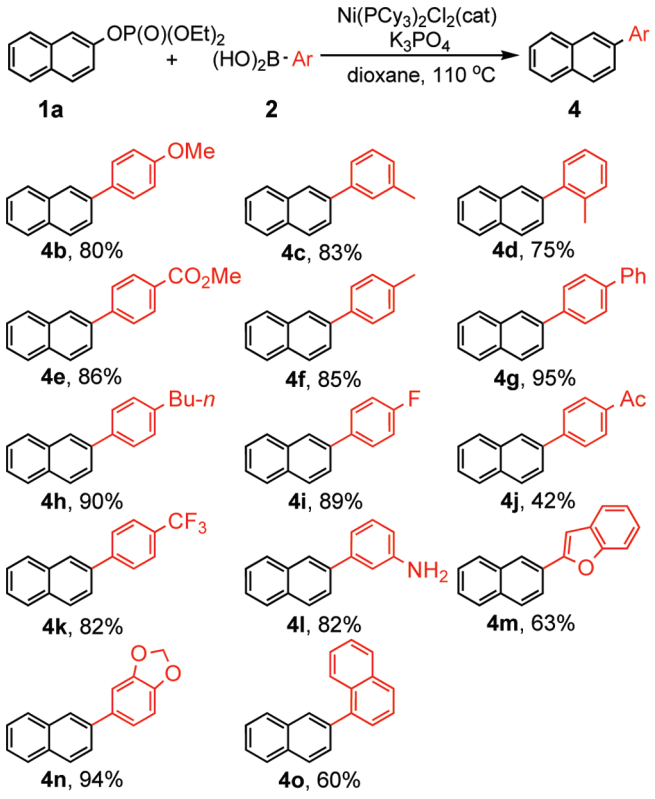
Table 3. Nickel-Catalyzed Cross-Coupling of 1a with Various Boronic Acids 2[a]

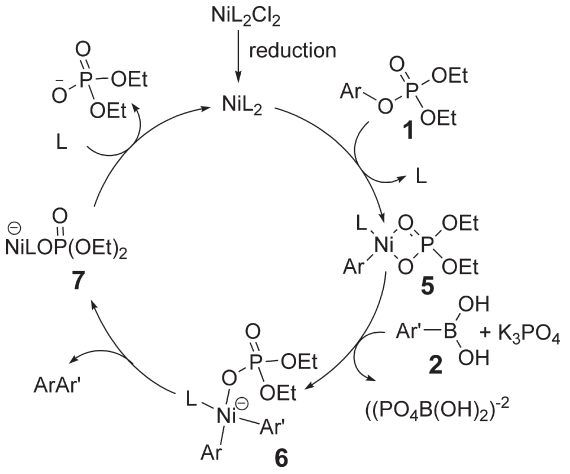


[a] Reaction conditions: phosphates **1a** (0.50 mmol), $Ni(PCy_3)_2Cl_2$ (0.050), $ArB(OH)_2$ (0.75 mmol), K_3PO_4 (2.25 mmol), dioxane (2 mL), 110 °C, 24 h.

to modify the natural product. Cross-coupling on peptides and proteins has received much attention recently because of the prospect of selective formation of a nonlabile linkage at a residue functionally orthogonal to natural amino acids.[48] Such modified proteins may then find use in a variety of biochemical and therapeutic contexts. As shown in Scheme 2, starting from L-tyrosine derivative **11**, a 4-anisyl group was introduced into **12** via our cross-coupling method. Finally, the difference in reactivity between aryl halides and phosphates has been successfully applied to the selective functionalization of substrates.[9] As revealed in Scheme 2, the cross-coupling of 6-bromo-2-naphthyl phosphate with phenylboronic acid under the standard Suzuki–Miyaura conditions, followed by the Ni-catalyzed cross-coupling of a phosphate moiety, furnished naphthalene derivative **16** containing two different aryl groups.

In summary, we have demonstrated an alternative Suzuki–Miyaura cross-coupling reaction of aryl diethyl phosphates with arylboronic acids. The Ni complex selectively cleaves the aryl–oxygen bond in the phosphate, leaving the ethyl–oxygen bond untouched to give the final unsymmetrical biaryls. Furthermore, the phosphate can be used as a protecting group and then as a leaving group for constructing biaryl and polyarene scaffolds. In view of the easy availability, moderate cost and the development of selective activation methods, aryl phosphates are expected to be extensively used in the Suzuki–Miyaura cross-coupling reaction.

Scheme 1. Proposed Mechanism



EXPERIMENTAL SECTION

General. All reactions were carried out under argon atmosphere. All the metal reagents and arylboronic acids were purchased and used without further purification. The phosphates were prepared from the phenols and diethyl phosphite in CCl_4 using Et_3N as base. $Ni(PCy_3)_2Cl_2$ was synthesized according to literature method.[10] The dry K_3PO_4 was made from $K_3PO_4 \cdot 3H_2O$ by heating under vacuum. The solvent was freshly distilled. Spectroscopy data of the known compounds matches with the data reported in the corresponding references. All new compounds were further characterized by elemental analysis or HRMS.

General Procedure for the Cross-Coupling of Aryl Phosphates with Arylboronic Acids: Preparation of 2-Phenylnaphthalene (3a).[11]. An oven-dried Schlenk tube containing $Ni(PCy_3)_2Cl_2$ (0.050 mmol, 10%), K_3PO_4 (2.25 mmol), and phenylboronic acid (2a) (0.750 mmol) was evacuated and purged with argon three times. Freshly distilled dioxane (2.0 mL) and 2-naphthyl phosphate (1a) (0.500 mmol) were sequentially added to the system at room temperature. The reaction mixture was heated with stirring at 110 °C for 24 h. The mixture was cooled to room temperature and then transferred to a round-bottom flask. Silica gel (3.0 g) was added, and the solvent was removed under reduced pressure to afford a free-flowing powder. This powder was then dry-loaded onto a silica gel column and purified by flash chromatography (100% hexane) to yield the desired 2-phenylnaphthalene (94.7 mg, 93%) as a white powder. A number of products were synthesized according to this procedure and their spectral data were shown below.

*2-Phenylnaphthalene (**3a**) (CAS no. 612-94-2).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03 (d, J = 1.3 Hz, 1 H), 7.91–7.82 (m, 3 H), 7.75–7.69 (m, 3 H), 7.51–7.44 (m, 4 H), 7.26 (tt, J = 7.4, 1.2 Hz, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.1, 138.6, 133.7, 132.6, 128.8, 128.4, 128.2, 127.6, 127.4, 127.3, 126.3, 125.9, 125.8, 125.6.

*4-Methoxybiphenyl (**3b**, **3b′**) (CAS no. 613-37-6).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.56–7.51 (m, 4 H), 7.43–7.38 (m, 2 H), 7.29 (tt, J = 7.4, 1.2 Hz, 1 H), 6.97 (dt, J = 8.8, 2.2 Hz, 2 H), 3.84 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.1, 140.8, 133.8, 128.7, 128.1, 126.7, 126.6, 114.2, 55.3.

*1-(Biphenyl-4-yl)ethanone (**3c**) (CAS no. 92-91-1).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.05–8.02 (m, 2 H), 7.70–7.67 (m, 2 H), 7.64–7.62 (m, 2 H), 7.50–7.45(m, 2 H), 7.43–7.38 (m, 1 H), 2.64 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.7, 145.8, 139.9, 135.9, 128.9, 128.8, 128.2, 127.2, 127.1, 26.6.

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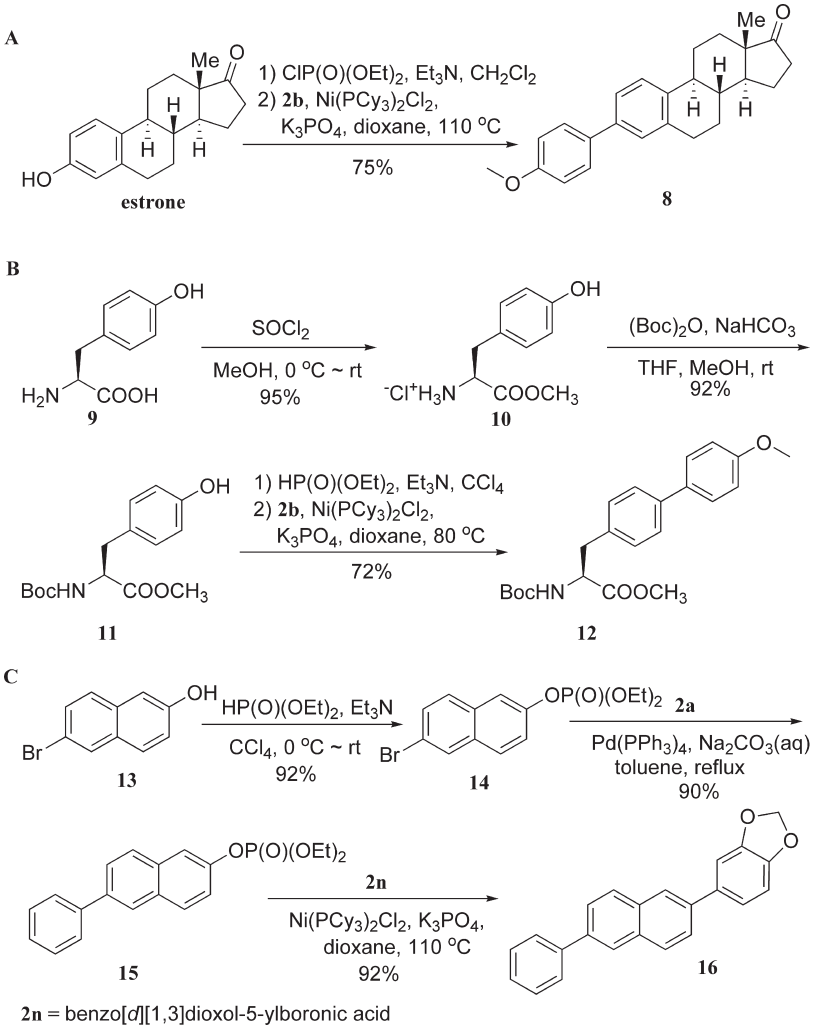
Scheme 2. Application of the Ni-Catalyzed Cross-Coupling of Aryl Phosphates with Arylboronic Acids



1-(6-Phenylnaphthalen-2-yl)ethanone (***3d***) *(CAS no. 1048964-07-3).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.41 (d, J = 0.6 Hz, 1 H), 8.00−7.95 (m, 3 H), 7.87 (d, J = 8.6 Hz, 1 H), 7.75 (dd, J = 8.5, 1.8 Hz, 1 H), 7.67-7.64 (m, 2 H), 7.45−7.41 (m, 2 H), 7.36−7.32 (m, 1 H), 2.67 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.9, 141.2, 140.5, 135.9, 134.5, 131.7, 130.0, 129.9, 129.0, 128.7, 127.9, 127.5, 126.5, 125.6, 124.4, 26.7.

4-Methoxy-4′-methylbiphenyl (***3e***) *(CAS no. 53040-92-9).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53−7.50 (m, 2 H), 7.47−7.44 (m, 2 H), 7.23 (d, J = 8.4 Hz, 1 H), 7.00−6.95 (m, 2 H), 3.85 (s, 3 H), 2.39 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.9, 138.0, 136.3, 133.8, 129.4, 127.9, 126.6, 114.2, 55.3, 21.0.

4-Aminobiphenyl (***3f***) *(CAS no. 92-67-1).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54−7.52 (m, 2 H), 7.43−7.36 (m, 4 H), 7.28−7.23 (m, 1 H), 6.78−6.72 (m, 2 H), 3.70 (s, 2 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.8, 141.1, 131.5, 128.6, 128.0, 126.4, 126.2, 115.4.

1-Phenylnaphthalene (***3g***) *(CAS no. 605-02-7).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00−7.96 (m, 2 H), 7.92 (d, J = 8.2 Hz, 1 H), 7.61−7.47 (m, 9 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.8, 140.3, 133.8, 131.6, 130.1, 128.3, 127.6, 127.2, 126.9, 126.0, 125.8, 125.4.

4-Methoxy-2-methylbiphenyl (***3h***) *(CAS no. 92495-54-0).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.20−7.13(m, 6 H), 6.90−6.86 (m, 2 H), 3.78 (s, 3 H), 2.20 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.5, 141.6, 135.5, 134.4, 130.3, 130.2, 129.9, 127.0, 125.7, 113.5, 55.3, 20.5.

4-Phenylbiphenyl (***3i***, ***3i′***) *(CAS no. 92-94-4).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.67(s, 4 H), 7.65−7.62 (m, 4 H), 7.47−7.43 (m, 4 H), 7.35 (tt, J = 7.4, 1.2 Hz, 2 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.7, 140.1, 128.8, 127.5, 127.3, 127.0.

2-Methoxy-6-phenylnaphthalene (***3j***) *(CAS no. 59115-43-4).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96 (d, J = 1.6 Hz, 1 H), 7.78 (dd, J = 8.6, 5.0 Hz, 2 H), 7.71−7.67 (m, 3 H), 7.48−7.43 (m, 2 H), 7.36−7.32 (m, 1 H), 7.18−7.14 (m, 2 H), 3.91 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 157.8, 141.2, 136.4, 133.8, 129.7, 129.2, 128.8, 127.2, 127.2, 127.0, 126.0, 125.6, 119.1, 105.6, 55.3.

4-Methoxy-3-methylbiphenyl (***3k***) *(CAS no. 17171-17-4).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.55−7.51 (m, 2 H), 7.38−7.30 (m, 3 H), 7.14−7.13 (m, 1 H), 7.00−6.96 (m, 2 H), 3.86 (s, 3 H), 2.42 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.1, 140.8, 138.3, 133.9, 128.6, 128.1, 127.6, 127.4, 123.8, 114.1, 55.3, 21.5.

Methyl 6-Phenyl-2-naphthoate (***3l***) *(CAS no. 904688-59-1).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.56 (s, 1 H), 8.03−8.00 (m, 2 H), 7.95 (d, J = 8.5 Hz, 1 H),7.86 (d, J = 8.6 Hz, 1 H), 7.74 (dd, J = 8.5, 1.5 Hz, 1 H), 7.66 (d, J = 7.6 Hz, 2 H), 7.43 (t, J = 7.6 Hz, 2 H), 7.34 (t, J = 7.3 Hz, 1 H), 3.92 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.2, 141.0, 140.6, 135.8, 131.6, 130.8, 129.8, 128.9, 128.4, 127.8, 127.5, 127.4, 126.4, 125.7, 125.6, 52.2.

4-Fluoro-4′-methoxybiphenyl (***3m***) *(CAS no. 450-39-5).* ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.51−7.44 (m, 4 H), 7.12−7.06 (m, 2 H), 6.96

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(dt, J = 8.8, 2.6 Hz, 2 H), 3.84 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.1 (d, J = 245.5 Hz), 159.1, 136.9 (d, J = 3.1 Hz), 132.8, 128.2 (d, J = 7.9 Hz), 128.0, 115.5 (d, J = 21.4 Hz), 114.3, 55.3.

4-Cyanobiphenyl (**3n**) *(CAS no. 2920-38-9).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.67−7.59 (m, 4 H), 7.53−7.50(m, 2 H), 7.43−7.39 (m, 2 H), 7.37−7.33 (m, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.7, 139.2, 132.6, 129.1, 128.6, 127.7, 127.2, 118.9, 110.9.

2,6-Diphenylnaphthalene (**3o**, **3o′**) *(CAS no. 60706-24-3).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.07 (d, J = 1.9 Hz, 2 H), 7.97(d, J = 8.5 Hz, 2 H), 7.79−7.77 (dd, J = 8.5, 1.8 Hz, 2 H), 7.76−7.73(m, 4 H), 7.52−7.47 (m, 4 H), 7.41−7.37 (tt, J = 7.4, 1.2 Hz, 2 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.1, 138.7, 132.9, 128.9, 128.7, 127.4, 126.0, 125.5.

o-Terphenyl (**3p**) *(CAS no. 84-15-1).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.51−7.45 (m, 4H), 7.29−7.23 (m, 6H), 7.22−7.18 (m, 4H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.5, 140.6, 130.6, 129.9, 127.8, 127.4, 126.4.

Methyl 4-Biphenylcarboxylate (**3q**) *(CAS no. 720-75-2).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.04−8.01(m, 2 H), 7.59−7.52 (m, 4 H), 7.40−7.36 (m, 2 H), 7.33−7.29 (m, 1 H), 3.86 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.0, 145.6, 140.0, 130.1, 128.9, 128.1, 127.2, 127.0, 52.1.

5-Phenylbenzo[d][1, 3]dioxole (**3r**) *(CAS no. 24382-05-6).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.52−7.49 (m, 2 H), 7.41−7.37 (m, 2 H), 7.32−7.28 (m, 1 H), 7.07−7.04 (m, 2 H), 6.87 (dd, J = 7.9, 0.5 Hz, 1 H), 5.98 (s, 2 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.1, 147.0, 140.9, 135.6, 128.7, 126.9, 126.8, 120.6, 108.5, 107.7, 101.1.

Methyl 2-Biphenylcarboxylate (**3s**) *(CAS no. 16605-99-5).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.82 (dd, J = 7.7, 1.3 Hz, 1 H), 7.52 (td, J = 7.5, 1.4 Hz, 1 H), 7.42−7.29 (m, 7 H), 3.62 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 169.1, 142.4, 141.3, 131.2, 130.8, 130.7, 129.7, 128.3, 128.0, 127.2, 127.1, 51.9.

3-Phenylpyridine (**3t**) *(CAS no. 1008-88-4).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.85 (d, J = 1.8 Hz, 1 H), 8.59 (dd, J = 4.8, 1.5 Hz, 1 H), 7.87 (dt, J = 7.9, 2.0 Hz 1 H), 7.59−7.56 (m, 2 H), 7.49−7.45 (m, 2 H), 7.42−7.34 (m, 2 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.4, 148.3, 137.8, 136.6, 134.3, 129.0, 128.1, 127.1, 123.5.

2-(4-Methoxyphenyl)naphthalene (**4b**) *(CAS no. 59115-45-6).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.96 (s, 1 H), 7.87−7.81 (m, 3 H), 7.69 (dd, J = 8.5, 1.8 Hz, 1 H), 7.65−7.61(dt, J = 8.8, 2.6 Hz, 2 H), 7.45 (dd, J = 6.9, 1.5 Hz, 2 H), 7.00(dt, J = 8.8, 2.6 Hz, 2 H), 3.82 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 159.2, 138.1, 133.7, 133.6, 132.3, 128.4, 128.3, 128.0, 127.6, 126.2, 125.6, 125.4, 125.0, 114.3, 55.3.

2-m-Tolylnaphthalene (**4c**) *(CAS no. 36821-15-5).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.01 (d, J = 1.3 Hz, 1 H), 7.89−7.82 (m, 3 H), 7.72 (dd, J = 8.5, 1.9 Hz, 1 H), 7.52−7.42 (m, 4 H), 7.35 (t, J = 7.6 Hz, 1 H), 7.17 (d, J = 6.6 Hz, 1 H), 2.43 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.1, 138.7, 138.4, 133.7, 132.6, 128.7, 128.3, 128.2, 128.1, 128.0, 127.6, 126.2, 125.8, 125.7, 125.6, 124.5, 21.6.

2-o-Tolylnaphthalene (**4d**) *(CAS no. 66778-24-3).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.87−7.83 (m, 3 H), 7.76 (d, J = 0.6 Hz, 1 H), 7.51−7.45 (m, 3 H), 7.33−7.24 (m, 4 H), 2.30 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 141.8, 139.5, 135.5, 133.3, 132.3, 130.3, 130.0, 128.0, 127.8, 127.7, 127.6, 127.5, 127.4, 126.1, 125.9, 125.8, 20.5.

Methyl 4-(Naphthalen-2-yl)benzoate (**4e**) *(CAS no. 205823-29-6).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.15−8.12 (m, 2 H), 8.07 (d, J = 1.3 Hz, 1 H), 7.93- 7.84 (m, 3 H), 7.79−7.72 (m, 3 H), 7.53−7.47 (m, 2 H), 3.94 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.0, 145.5, 137.2, 133.5, 132.9, 130.1, 128.9, 128.6, 128.3, 127.6, 127.2, 126.5, 126.4, 126.3, 125.2, 52.1.

2-p-Tolylnaphthalene (**4f**) *(CAS no. 59115-49-0).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00 (s, 1 H), 7.88−7.82 (m, 3 H), 7.72 (dd, J = 8.5, 1.7 Hz, 1 H), 7.61 (d, J = 8.1 Hz, 2 H), 7.49−7.42 (m, 2 H), 7.27 (d, J = 8.1 Hz, 2 H), 2.40 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.5, 138.2, 137.1, 133.7, 132.5, 129.6, 128.3, 128.1, 127.6, 127.2, 126.2, 125.7, 125.5, 125.4, 21.1.

2-(4-Phenylphenyl)naphthalene (**4g**) *(CAS no. 68862-02-2).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.09 (d, J = 1.1 Hz, 1 H), 7.94−7.86 (m, 3 H), 7.83−7.78 (m, 3 H), 7.74−7.71 (m, 2 H), 7.68−7.65 (m, 2 H), 7.53−7.45 (m, 4 H), 7.39−7.35(m, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.7, 140.2, 140.0, 138.0, 133.7, 132.7, 128.8, 128.5, 128.2, 127.7, 127.6, 127.5, 127.4, 127.1, 126.3, 126.0, 125.7, 125.4.

2-(4-Butylphenyl)naphthalene (**4h**) *(CAS no. 1075754-29-8).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.02 (d, J = 1.3 Hz, 1 H), 7.89−7.83 (m, 3 H), 7.73 (dd, J = 8.5, 1.8 Hz, 1 H), 7.65−7.62 (m, 2 H), 7.50−7.43 (m, 2 H), 7.28 (d, J = 8.2 Hz, 2 H), 2.67 (t, J = 7.8 Hz, 2 H), 1.65 (m, 2 H), 1.40 (m, 2 H), 0.95 (t, J = 7.3 Hz, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 142.2, 138.5, 138.4, 133.7, 132.5, 128.9, 128.3, 128.1, 127.6, 127.2, 126.2, 125.7, 125.6, 125.4, 35.3, 33.6, 22.4, 14.0.

2-(4-Fluorophenyl)naphthalene (**4i**) *(CAS no. 28396-55-6).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94 (d, J = 1.3 Hz, 1 H), 7.87−7.80 (m, 3 H), 7.65−7.59 (m, 3 H), 7.49− 7.43 (m, 2 H), 7.17−7.09 (m, 2 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 163.7, 161.3, 137.5, 137.2, 137.1, 133.6, 132.5, 128.9, 128.8, 128.5, 128.1, 127.6, 126.4, 126.0, 125.6, 125.4, 115.8, 115.6.

1-(4-(Naphthalen-2-yl)phenyl)ethanone (**4j**) *(CAS no. 150988-77-5).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.08−8.04(m, 3 H), 7.94−7.85 (m, 3 H), 7.81−7.78 (m, 2 H), 7.74 (dd, J = 8.5, 1.9 Hz, 1 H), 7.54−7.48(m, 2 H), 2.64 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.7, 145.6, 137.1, 135.8, 133.5, 133.0, 129.0, 128.7, 128.3, 127.7, 127.4, 126.5, 126.4, 126.3, 125.1, 26.6.

2-(4-(Trifluoromethyl)phenyl)naphthalene (**4k**) *(CAS no. 460743-71-9).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.03 (d, J = 1.4 Hz, 1 H), 7.93−7.84 (m, 3 H), 7.78 (d, J = 8.2 Hz, 2 H), 7.72−7.68 (m, 3 H), 7.54−7.48 (m, 2 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.6, 137.0, 133.5, 133.0, 129.5, 129.2, 128.7, 128.3, 127.7, 127.6, 126.6, 126.5, 126.3, 125.8, 125.7, 125.6, 125.2, 123.0.

3-(Naphthalen-2-yl)benzenamine (**4l**) *(CAS no. 176034-11-0).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.00 (d, J = 1.5 Hz, 1 H), 7.89−7.81 (m, 3 H), 7.70 (dd, J = 8.5, 1.8 Hz, 1 H), 7.50−7.43 (m, 2 H), 7.24 (dd, J = 14.9, 7.1 Hz, 1 H), 7.11 (ddd, J = 7.7, 1.6, 1.0 Hz, 1 H), 7.01 (t, J = 1.9 Hz, 1 H), 6.68 (ddd, J = 7.9, 2.3, 0.9 Hz, 1 H), 3.72 (s, 2 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.8, 142.3, 138.7, 133.6, 132.6, 129.7, 128.2, 128.1, 127.6, 126.2, 125.8, 125.7, 125.6, 117.9, 114.2, 114.1.

2-(Naphthalen-2-yl)benzofuran (**4m**) *(CAS no. 26870-25-7).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.36 (d, J = 0.5 Hz, 1 H), 7.91−7.81 (m, 4 H), 7.60−7.54 (m, 2 H), 7.52−7.45 (m, 2 H), 7.30 (td, J = 7.3, 1.5 Hz, 1 H), 7.24 (td, J = 7.3, 1.1 Hz, 1 H), 7.11 (d, J = 0.8, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 155.9, 155.0, 133.4, 133.3, 129.3, 128.5, 128.4, 127.8, 127.7, 126.6, 126.4, 124.4, 123.8, 123.0, 122.8, 120.9, 111.2, 101.9.

5-(Naphthalen-2-yl)benzo[d][1, 3]dioxole (**4n**) *(CAS no. 228715-55-7).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.97 (d, J = 1.3 Hz, 1 H), 7.91−7.85 (m, 3 H), 7.69 (dd, J = 8.5, 1.9 Hz, 1 H), 7.54−7.46 (m, 2 H), 7.23−7.20 (m, 2 H), 6.95 (dd, J = 7.9, 0.5 Hz, 1 H), 6.03 (s, 2 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.2, 147.2, 138.2, 135.5, 133.7, 132.4, 128.4, 128.1, 127.6, 126.3, 125.8, 125.5, 125.3, 120.9, 108.6, 107.9, 101.2.

1-(Naphthalen-2-yl)naphthalene (**4o**) *(CAS no. 4325-74-0).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.94−7.86 (m, 7 H), 7.62 (dd, J = 8.3, 1.8 Hz, 1 H), 7.56−7.46 (m, 5 H), 7.43−7.38 (m, 1 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.2, 138.3, 133.8, 133.4, 132.6, 131.8, 128.7, 128.5, 128.3, 128.1, 127.7, 127.6, 127.2, 126.3, 126.1, 126.0, 125.8, 125.4.

(8R,9S*,13S*,14S*)-3-(4-Methoxyphenyl)-13-methyl-7,8,9,11,12, 13,15,16-octahydro-6H-cyclopenta[a]phenanthren-17(14H)-one* (**8**) *(CAS no. 1019777-25-3).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.53−7.49 (m, 2 H), 7.35 (d, J = 1.2 Hz, 2 H), 7.29 (s, 1 H), 6.98−6.94 (m, 2 H), 3.84 (s, 3 H), 2.98 (dd, J = 8.7, 4.0 Hz, 2 H), 2.55−2.44 (m, 2 H), 2.34 (td, J = 11.1, 4.1 Hz, 1 H), 2.18−2.03(m, 3 H), 2.00−1.96 (m, 1 H), 1.67−1.45 (m, 6 H), 0.92 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 220.8, 159.0, 138.4, 138.3, 136.8, 133.6, 128.0, 127.3, 125.8, 124.2, 114.1, 55.3, 50.5, 48.0, 44.4, 38.2, 35.8, 31.6, 29.7, 29.5, 26.6, 25.8, 21.6, 13.9. IR (film): 2928, 2864, 1737, 1608, 1518, 1493, 1468, 1455, 1441, 1282, 1245, 1174, 1025, 822 cm^{-1}. $[\alpha]^{25}_D$ +135.2 (c 1.0, $CHCl_3$) {lit.[49] $[\alpha]^{20}_D$ +135.0 (c 0.99, $CHCl_3$)}.

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*(S)-Methyl 2-(tert-Butoxycarbonylamino)-3-(4′-methoxybiphenyl-4-yl)propanoate (**12**) (CAS no. 196395-09-2).* ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.49 (dd, J = 10.9, 8.5 Hz, 4 H), 7.17 (d, J = 8.0 Hz, 2 H), 7.01−6.91 (m, 2 H), 5.01 (d, J = 7.8 Hz, 1 H), 4.62 (dd, J = 13.5, 5.9 Hz, 1 H), 3.85 (s, 1 H), 3.74 (s, 1 H), 3.12 (qd, J = 13.8, 5.9 Hz, 1 H), 1.42 (s, 3 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 172.4, 159.12, 155.1, 139.5, 134.4, 133.3, 129.7, 128.0, 126.8, 114.2, 79.9, 55.3, 54.4, 52.2, 38.0, 28.3. IR (film): 3371, 2976, 2931, 2837, 1746, 1716, 1610, 1500, 1441, 1392, 1366, 1294, 1248, 1216, 1176, 1041, 1016, 1002, 818, 522 cm^{-1}. $[\alpha]^{25}_D$ +46.5 (c 1.0, $CHCl_3$) {lit.[50] $[\alpha]^{26}_D$ +32.99 (c 1.0, $CHCl_3$)}.

*6-Bromonaphthalen-2-yl Diethyl Phosphate (**14**).* Pale yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.98 (d, J = 1.7 Hz, 1 H), 7.73 (d, J = 8.9 Hz, 1 H), 7.67−7.65 (m, 2 H), 7.55 (dd, J = 8.8, 2.0 Hz, 1 H), 7.38 (ddd, J = 8.9, 2.4, 0.7 Hz, 1 H), 4.32−4.19 (m, 4 H), 1.36 (td, J = 7.1, 1.1 Hz, 6 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.6 (d, J_{PC} = 6.9 H z), 132.3, 131.8, 130.0, 129.7, 129.1, 128.9, 121.1 (d, J_{PC} = 5.4 Hz), 119.2, 116.4 (d, J_{PC} = 4.9 Hz), 64.7 (d, J_{PC} = 6.0 Hz), 16.1 (d, J_{PC} = 6.6 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ −6.3. IR (film): 3060, 2984, 2932, 2909, 1628, 1589, 1500, 1479, 1462, 1443, 1363, 1280, 1247, 1199, 1150, 1127, 1032, 977, 935, 881, 801, 755, 706, 649, 573, 544, 506, 476 cm^{-1}. HRMS (ESI) calcd for $C_{14}H_{16}O_4PBrNa$, 380.9867; found 380.9863.

*Diethyl 6-Phenylnaphthalen-2-yl Phosphate (**15**).* White solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.01 (d, J = 1.1 Hz, 1 H), 7.87 (dd, J = 8.8, 2.2 Hz, 2 H), 7.76 (dd, J = 8.5, 1.8 Hz, 1 H), 7.71−7.68 (m, 3 H), 7.50−7.46 (m, 2 H), 7.41−7.35 (m, 2 H), 4.33−4.20 (m, 4 H), 1.37 (td, J = 7.1, 1.1 Hz, 6 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.4 (d, J_{PC} = 7.0 Hz), 140.8, 138.2, 133.0, 131.1, 130.1, 128.8, 128.0, 127.4, 127.3, 126.4, 125.5, 120.5 (d, J_{PC} = 5.3 Hz), 116.2 (d, J_{PC} = 4.8 Hz), 64.7 (d, J_{PC} = 6.2 Hz), 16.1 (d, J_{PC} = 6.6 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ −6.2. IR (film): 3058, 2984, 1633, 1599, 1496, 1474, 1370, 1340, 1274, 1239, 1196, 1150, 1032, 977, 889, 810, 765, 755, 699, 664, 533, 477 cm^{-1}. Mp 49−50 °C. HRMS (ESI) calcd for $C_{20}H_{21}O_4PNa$, 379.1075; found 379.1075.

*5-(6-Phenylnaphthalen-2-yl)benzo[d][1,3]dioxole (**16**).* White solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.04 (d, J = 0.9 Hz, 1 H), 7.97 (d, J = 1.3 Hz, 1 H), 7.93 (dd, J = 8.5, 2.4 Hz, 2 H), 7.77−7.68 (m, 4 H), 7.49 (t, J = 7.6 Hz, 2 H), 7.38 (t, J = 7.4 Hz, 1 H), 7.21−7.19 (m, 2 H), 6.93 (d, J = 8.4 Hz, 1 H), 6.02 (s, 2 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.3, 147.2, 141.0, 138.5, 138.3, 135.4, 132.9, 132.7, 128.9, 128.7, 128.6, 127.4, 126.0, 125.9, 125.5, 125.0, 120.9, 108.7, 107.8, 101.2. IR (KBr) ν_{max}: 3053, 2917, 1595, 1512, 1487, 1472, 1438, 1316, 1254, 1240, 1102, 1047, 931, 910, 889, 858, 801, 767, 753, 692, 470 cm^{-1}. Mp 183−184 °C. $C_{23}H_{16}O_2$ (324.12) calcd C 85.16, H 4.97, found C 85.30, H 5.12.

■ ASSOCIATED CONTENT

Supporting Information. General experimental procedure and characterization details. This material is available free of charge via the Internet at http://pubs.acs.org.

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