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N-Heterocyclic Carbene Derived Nickel–Pincer Complexes: Efficient and Applicable Catalysts for Suzuki–Miyaura Coupling Reactions of Aryl/Alkenyl Tosylates and Mesylates

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Catalytic activities of NHC-derived nickel–pincer complexes for the Suzuki–Miyaura coupling reactions of aryl/alkenyl tosylates and mesylates are described. In the presence of a catalytic amount of nickelacycle **1a**, a wide array of tosylates and mesylates reacted with several aryl- and alkenylboronic acids to afford the coupling products, generally in high yields. Fine tuning of the reaction conditions for each class of electrophiles was achieved only by choosing the appropriate reaction medium (DME for tosylates, dioxane for mesylates).

Introduction

Palladium- and nickel-catalyzed cross-coupling reactions are one of the most powerful methods for the assembly of molecules such as natural products, pharmaceuticals, and functional chemicals.[1] In this area, aryl/alkenyl triflates, which are prepared from phenols or carbonyl compounds, have often been employed as electrophiles in place of aryl/alkenyl halides.[2] However, triflating reagents such as Tf_2O and $PhNTf_2$ are relatively expensive, and triflates themselves are sometimes unstable in air and moisture. Aryl/alkenyl tosylates and mesylates have recently been regarded as important alternatives to the above-mentioned traditional electrophiles. They are easily prepared from inexpensive and readily available starting materials, easier to handle crystalline solids, and more stable than the corresponding triflates. Despite their considerably inert leaving-group activity, development of the catalyst systems, which enable coupling reactions of tosylates and mesylates to be performed, has recently attracted much attention. Indeed, several examples have realized their use in transition-metal-catalyzed C–C and C–N bond-forming coupling reactions.[3–6] For example, Percec reported the Suzuki–Miyaura coupling reaction of aryl mesylates in the presence of a $NiCl_2$(dppe) catalyst.[4b] The room temperature Suzuki–Miyaura coupling reaction of aryl tosylates was also achieved by Hu by employing a $Ni(cod)_2/PCy_3$ catalyst system.[3c,3o] However, there has been only a limited number of precedents for their practical use in cross-coupling processes.[7]

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We recently reported the synthesis and evaluation of the catalytic activities of N-heterocyclic carbene (NHC)-derived nickel(II)–pincer complexes **1a**–**e** (Figure 1), which turned out to be excellent catalysts for C–C bond-forming coupling reactions.[8,9] These nickel complexes, readily prepared from inexpensive, commercially available materials, exhibit high stability to both air and moisture, making them highly applicable catalysts. Herein, we disclose that our nickel–pincer complexes efficiently catalyze the Suzuki–Miyaura coupling processes of a range of aryl/alkenyl tosylates and mesylates with aryl/alkenylboronic acids.[10]

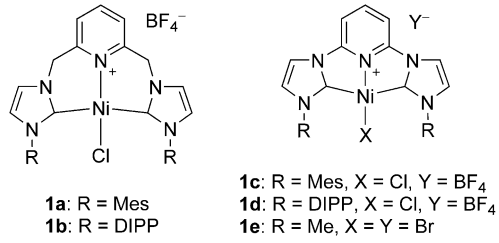


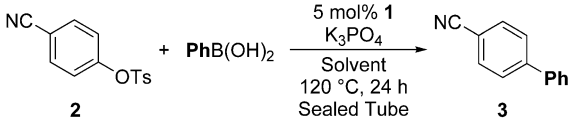
Figure 1. Nickel(II)–pincer complexes (Mes = mesityl, DIPP = 2,6-diisopropylphenyl).

Results and Discussion

Initial studies focused on the reaction of 4-(*p*-toluenesulfonyloxy)benzonitrile (**2**) with phenylboronic acid in the presence of nickel–pincer complex **1a** (5 mol-%) and K_3PO_4 (2 equiv.) to optimize the reaction conditions (Table 1). Whereas solvents such as DMSO, DMF, dichloroethane, and dioxane were not effective for this process (Table 1, Entries 1–4), the use of toluene, acetonitrile, and THF provided moderate to good yields (Table 1, Entries 5–7). DME proved to be the best solvent for this reaction, producing

coupling product **3** in excellent yield (95%; Table 1, Entry 8). K_3PO_4 was also crucial for efficient conversion.[11] Decreasing either the reaction temperature or the catalyst loading led to poorer results (Table 1, Entries 9 and 10). Complex **1b**, which has two fused six-membered rings, similar to **1a**, also showed high performance for this process, although a longer reaction time was necessary to reach completion of the reaction (Table 1, Entry 11). Interestingly, complexes **1c**–**e**, possessing the five-membered metallacycle backbone, were not as active as **1a** and **1b**. This result is contrary to the previous observation[8a] in which reactions of aryl *halides* proceeded faster in the presence of **1c** and **1d** than in the presence of **1a** and **1b**, plausibly indicating that the rate-determining step during the catalytic cycle would be different for the reactions of aryl *tosylates* than for aryl *halides*. The precise reaction mechanism remains to be elucidated.

Table 1. Optimization of reaction conditions for coupling of aryl tosylate **2** with $PhB(OH)_2$.[a]



2 **3**

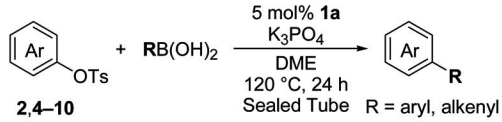
Entry	**1**	Solvent	Yield [%][b,c]
1	**1a**	DMSO	11 (39)
2	**1a**	DMF	37 (41)
3	**1a**	$ClCH_2CH_2Cl$	43 (48)
4	**1a**	dioxane	40 (47)
5	**1a**	toluene	58 (26)
6	**1a**	CH_3CN	63 (29)
7	**1a**	THF	69 (25)
8	**1a**	DME	95
9[d]	**1a**	DME	70 (26)
10[e]	**1a**	DME	59 (33)
11[f]	**1b**	DME	84
12	**1c**	DME	37 (49)
13	**1d**	DME	37 (52)
14	**1e**	DME	33 (55)

[a] Reagents: 4-(*p*-toluenesulfonyloxy)benzonitrile (0.10 mmol), **Ph**$B(OH)_2$ (0.30 mmol), **1** (0.0050 mmol), K_3PO_4 (0.20 mmol), and solvent (1 mL) in a sealed tube. [b] Isolated yield. [c] Figure in parentheses is the recovery yield of **2**. [d] 100 °C. [e] 1 mol-% of **1a** was used. [f] 48 h.

The substrate scope of this system was next examined by using various aryl tosylates and boronic acids. Under the optimal conditions identified, electron-poor aryl tosylates **2** and **4** were treated with a range of aryl- and alkenylboronic acids in the presence of **1a** (5 mol-%), and the corresponding coupled products **11**–**20** were obtained in good to high yields (Table 2, Entries 1–10). In contrast, electron-rich or sterically hindered tosylates **5**–**7** remained as poorly reactive substrates for this process (Table 2, Entries 11–13). The combinations of aryl tosylates **8**–**10**, derived from naphthols and 3-hydroxypyridine, with boronic acids were also effective for the synthesis of biaryl and styrene compounds **24**–**36** (Table 2, Entries 14–26).

Alkenyl tosylates turned out to be more reactive than aryl tosylates in our catalytic system. Thus, activated alkenyl tosylates **37**–**41** were efficiently coupled with arylboronic acids in the presence of a catalytic amount of **1a**, and 4-arylated coumarins **42**–**48**, 2-pyranones **49**–**51**, and 2-quinolones **52**–**54** were successfully obtained (Table 3).[12–14]

Table 2. Coupling of various aryl tosylates with aryl/alkenylboronic acids.[a]



2,4–10 **11–36**

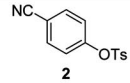
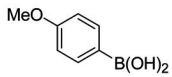
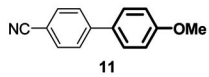
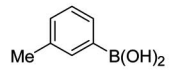
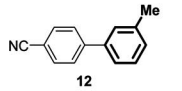
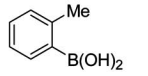
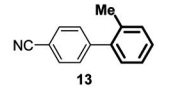
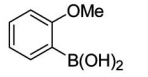
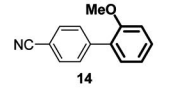
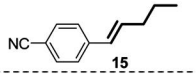
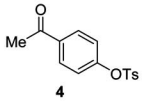
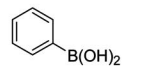
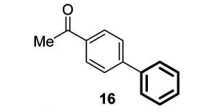
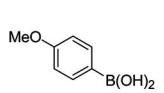
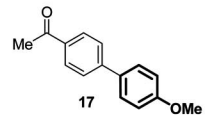
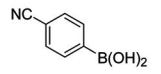
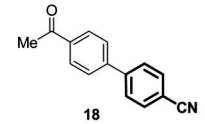
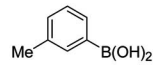
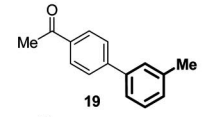
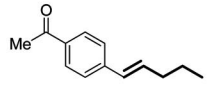
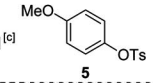
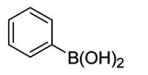
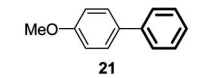
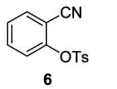
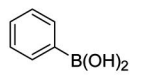
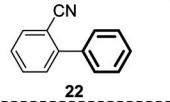
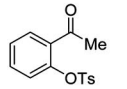
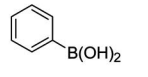
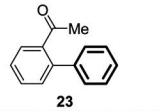
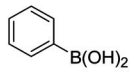
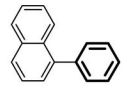
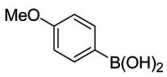
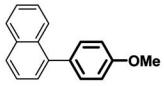
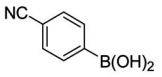
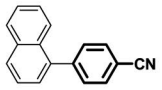
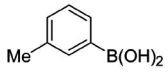
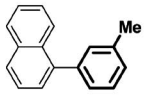
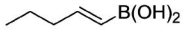
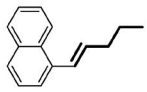
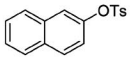
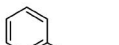
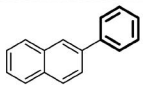
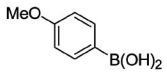
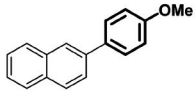
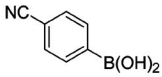
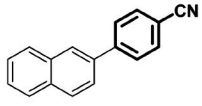
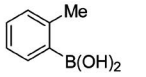
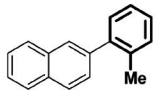
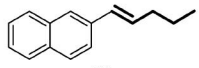
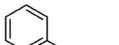
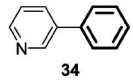
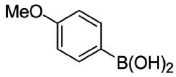
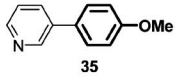
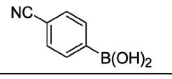
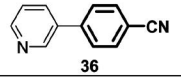
Entry	Ar–OTs	$RB(OH)_2$	Product	Yield [%][b]
1	 **2**		 **11**	55
2			 **12**	59
3			 **13**	52
4			 **14**	58
5			 **15**	74
6	 **4**		 **16**	78
7			 **17**	71
8[c]			 **18**	48
9			 **19**	79
10			 **20**	69
11[c]	 **5**		 **21**	20
12[c]	 **6**		 **22**	17
13	 **7**		 **23**	9

Table 2. *(Continued)*

Entry	Ar–OTs	$RB(OH)_2$	Product	Yield [%][b]
14	 **8**		 **24**	73
15			 **25**	66
16[c]			 **26**	91
17			 **27**	72
18[c]			 **28**	49
19	 **9**		 **29**	76
20			 **30**	65
21[c]			 **31**	51
22[c]			 **32**	57
23[c]			 **33**	55
24	 **10**		 **34**	69
25[c]			 **35**	86
26[c]			 **36**	83

[a] Reagents: aryl tosylate (0.10 mmol), $RB(OH)_2$ (0.30 mmol), **1a** (0.0050 mmol), K_3PO_4 (0.30 mmol), and DME (1 mL) in a sealed tube. [b] Isolated yield. [c] 48 h.

The capability of nickel–pincer complexes to participate in for cross-coupling reactions of aryl/alkenyl tosylates encouraged us to use these catalysts in the reactions of aryl mesylates, which are less reactive but more atom-economical substrates. The reaction of mesylate **55** with phenylboronic acid was first carried out under the optimized reaction conditions developed for aryl tosylates, resulting in a low yield (Table 4, Entry 1). Subsequent screening of the solvents revealed that the use of dioxane greatly enhanced

Table 3. Coupling of activated alkenyl tosylates with arylboronic acids.[a]

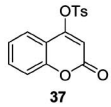
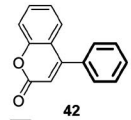
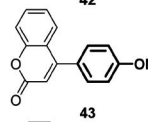
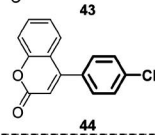
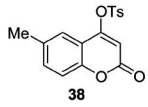
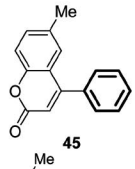
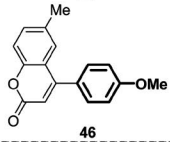
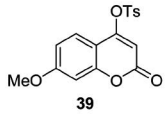
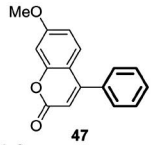
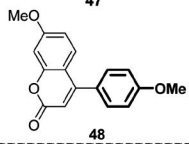
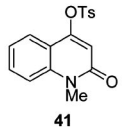
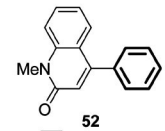
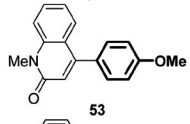
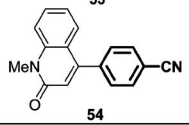
Entry	Alkenyl–OTs	$ArB(OH)_2$[b] / Conditions	Product	Yield [%][c]
1	 **37**	**A** 100 °C, 12 h	 **42**	73
2		**B** 120 °C, 2 h	 **43**	90
3		**C** 120 °C, 3 h	 **44**	44
4	 **38**	**A** 120 °C, 3 h	 **45**	62
5		**B** 120 °C, 4 h	 **46**	65
6	 **39**	**A** 120 °C, 3 h	 **47**	52
7		**B** 120 °C, 4 h	 **48**	76
8	**40**	**A** 100 °C, 12 h	**49**	84
9		**B** 120 °C, 5 h	**50**	87
10		**C** 120 °C, 16 h	**51**	86

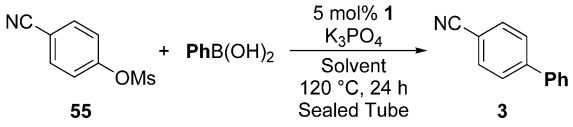
Table 3. *(Continued)*

Entry	Alkenyl–OTs	**Ar**B(OH)$_2$[b] / Conditions	Product	Yield [%][c]
11[d]	 **41**	**A** 100 °C, 16 h	 **52**	87
12[d]		**B** 80 °C, 36 h	 **53**	77
13		**C** 100 °C, 16 h	 **54**	86

[a] Reagents: alkenyl tosylate (0.10 mmol), **Ar**B(OH)$_2$ (0.30 mmol), **1a** (0.0050 mmol), K_3PO_4 (0.20 mmol), and DME (1 mL) in a sealed tube. [b] **Ar**B(OH)$_2$; **A**: **Ar** = Ph, **B**: **Ar** = *p*-MeOC$_6$H$_4$, **C**: **Ar** = *p*-NCC$_6$H$_4$. [c] Isolated yield. [d] 0.15 mmol of **Ar**B(OH)$_2$ was used.

the yield, giving coupling product **3** in fairly good yield (Table 4, Entry 8). Here again, complexes **1a** and **1b** exhibited higher catalytic activity than complexes **1c**, **1d**, and **1e** (Table 4, Entries 8 and 9 vs. 10–12).

Table 4. Optimization of reaction conditions for the coupling of aryl mesylate **55** with PhB(OH)$_2$.[a]

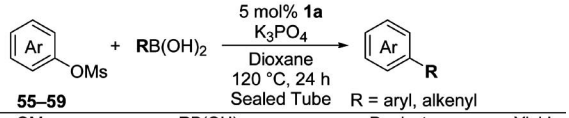


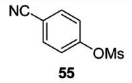
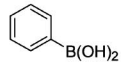
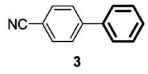
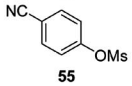
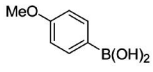
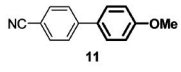
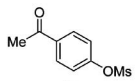
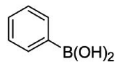
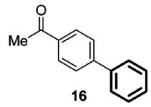
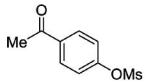
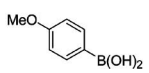
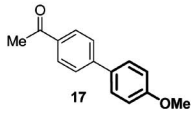
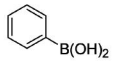
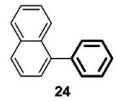
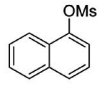
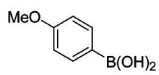
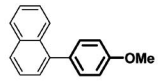
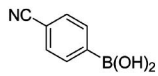
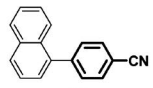
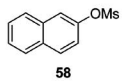
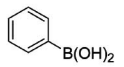
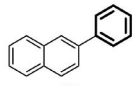
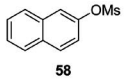
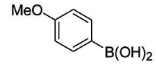
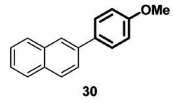
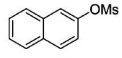
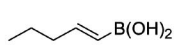
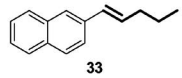
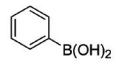
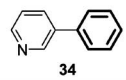
Entry	**1**	Solvent	Yield [%][b,c]
1	**1a**	DME	8 (86)
2	**1a**	THF	10 (75)
3	**1a**	DMSO	0 (79)
4	**1a**	DMF	trace (82)
5	**1a**	$ClCH_2CH_2Cl$	40 (44)
6	**1a**	CH_3CN	16 (73)
7	**1a**	toluene	38 (56)
8	**1a**	dioxane	63 (23)
9	**1b**	dioxane	56 (24)
10	**1c**	dioxane	38 (46)
11	**1d**	dioxane	25 (55)
12	**1e**	dioxane	28 (58)

[a] Reagents: 4-(methanesulfonyloxy)benzonitrile (0.10 mmol), **Ph**B(OH)$_2$ (0.30 mmol), **1** (0.0050 mmol), K_3PO_4 (0.20 mmol), and solvent (1 mL) in a sealed tube. [b] Isolated yield. [c] Figure in parentheses is the recovery yield of **55**.

Several aryl mesylates **55**–**59** were also treated with aryl/alkenylboronic acids in the presence of nickel–pincer complex **1a** to furnish the corresponding coupling compounds in moderate to good yields (Table 5).

Table 5. Coupling of aryl mesylates with aryl/alkenylboronic acids.[a]



Entry	Ar–OMs	**R**B(OH)$_2$	Product	Yield [%][b]
1	 **55**		 **3**	63
2	 **55**		 **11**	24
3	 **56**		 **16**	46
4	 **56**		 **17**	38
5	 **57**		 **24**	83
6	 **57**		 **25**	59
7	 **57**		 **26**	30
8	 **58**		 **29**	73
9	 **58**		 **30**	69
10	 **58**		 **33**	28
11	 **59**		 **34**	36

[a] Reagents: an aryl tosylate (0.10 mmol), **R**B(OH)$_2$ (0.30 mmol), **1a** (0.0050 mmol), K_3PO_4 (0.30 mmol), and DME (1 mL) in a sealed tube. [b] Isolated yield.

Conclusions

In summary, we developed a method for the Suzuki–Miyaura coupling reactions of aryl/alkenyl tosylates and mesylates by using NHC-derived nickel(II)–pincer complexes. Suitable substrates include both aryl and alkenyl tosylates with various substituents in this system. They are efficiently cross-coupled with an array of aryl/alkenylboronic acids, especially in the presence of catalyst **1a**, producing the corresponding coupling products generally in high yields. More importantly, reactions employing aryl mesylates as electrophiles – a more challenging process – also

successfully proceeded only by changing the solvent of the optimized conditions developed for tosylates. High catalytic activity of nickel–pincer complexes as well as their facile preparation would make this methodology an attractive addition to the repertoire of strategies for the catalytic activation of tosylates and mesylates. Further studies involving elucidation of the precise reaction mechanism and application of the catalysts to a range of cross-coupling reactions of aryl/alkenyl sulfonates are underway.

Experimental Section

General Methods: ^{1}H NMR spectra were recorded with a JEOL JNM-AL400 (400 MHz) spectrometer by using tetramethylsilane (TMS) as an internal standard. Chemical shifts (δ) are given from TMS (δ =0 ppm) and coupling constants are expressed in Hertz [Hz]. The following abbreviations are used: s = singlet, d = doublet, t = triplet, q = quartet, sext. = sextet, dd = doublet of doublets, dt = doublet of triplets, td = triplet of doublets, ddd, = doublet of doublets of doublets, and m = multiplet. ^{13}C NMR spectra were recorded with a JEOL JNM-AL400 (100 MHz) spectrometer and chemical shifts (δ) are given from $^{13}CDCl_3$ (δ =77.0 ppm). Mass spectra and high-resolution mass spectra were measured with JEOL JMS-DX303 and MS-AX500 instruments, respectively. IR spectra were recorded with a Shimadzu FTIR-8400. Melting points were measured with a Yazawa micro melting point apparatus and are uncorrected. Suzuki–Miyaura coupling reactions were carried out under an argon atmosphere. Nickel(II)–pincer complexes **1a**–**e** were prepared according to previously established procedure.[8a,8c] K_3PO_4 was dried while heating at 150 °C. All other chemicals, including anhydrous solvents, were purchased from commercial suppliers and used as received.

Typical Procedure for Sulfonylation of Phenols [4-(*p*-Toluenesulfonyloxy)benzonitrile (2)]: To a solution of 4-cyanophenol (0.50 g, 4.2 mmol) in CH_2Cl_2 (20 mL)/pyridine (5 mL) was added TsCl (1.2 g, 6.3 mmol) at 0 °C. After warming to room temperature, the mixture was stirred for 12–48 h. The reaction mixture was treated with saturated aqueous NH_4Cl (15 mL) followed by extraction with $CHCl_3$ (15 mL × 3). The combined extracts were washed with 3 N HCl aq. (15 mL × 2) and saturated aqueous NaCl (15 mL × 2), and the solvent was removed under reduced pressure. The residue was purified by silica gel column chromatography using hexane/AcOEt (4:1) as an eluent to obtain a colorless solid (1.0 g, 87%). The solid was recrystallized from hexane/AcOEt to obtain colorless plates.

4-(*p*-Toluenesulfonyloxy)benzonitrile (2): 87% (1.0 g) from 4-cyanophenol (0.50 g, 4.2 mmol). Colorless plates; m.p. 88–89 °C (hexane/AcOEt, ref.[15] 90.4–90.9 °C). IR (film): $\tilde{\nu}$ = 1204, 1377, 1497, 1599, 2232 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.47 (s, 3 H), 7.14 (d, J = 8.6 Hz, 2 H), 7.35 (d, J = 8.2 Hz, 2 H), 7.61 (d, J = 8.6 Hz, 2 H), 7.72 (d, J = 8.2 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.7, 111.2, 117.7, 123.4, 128.4, 130.0, 131.8, 133.9, 146.1, 152.5 ppm. MS (EI): m/z (%) = 273 (24) [M$^+$], 155 (100), 119 (2), 91 (75). HRMS: calcd. for $C_{14}H_{11}NO_3S$ 273.0460; found 273.0444.

4-(*p*-Toluenesulfonyloxy)acetophenone (4): 72% (0.56 g) from 4-acetylphenol (0.50 g, 3.7 mmol). Colorless needles; m.p. 69–70 °C (hexane/AcOEt, ref.[3c] 67–69 °C). IR (film): $\tilde{\nu}$ = 1200, 1377, 1497, 1597, 1686 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.45 (s, 3 H), 2.57 (s, 3 H), 7.09 (d, J = 8.6 Hz, 2 H), 7.32 (d, J = 8.4 Hz, 2 H), 7.72 (d, J = 8.4 Hz, 2 H), 7.90 (d, J = 8.6 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.7, 26.6, 122.5, 128.5, 129.9, 130.0, 132.2, 135.7, 145.7, 153.0, 196.6 ppm. MS (EI): m/z (%) = 290 (65) [M$^+$], 275 (15), 155 (100), 91 (78). HRMS: calcd. for $C_{15}H_{14}O_4S$ 290.0613; found 290.0601.

4-(*p*-Toluenesulfonyloxy)anisole (5): 92% (1.0 g) from 4-methoxyphenol (0.50 g, 4.0 mmol). Colorless needles; m.p. 69–70 °C (hexane/AcOEt, ref.[3c] 69–71 °C). IR (film): $\tilde{\nu}$ = 1094, 1196, 1371, 1502, 1597 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.45 (s, 3 H), 3.76 (s, 3 H), 6.76 (d, J = 10.0 Hz, 2 H), 6.88 (dt, J = 10.0 Hz, 2 H), 7.30 (d, J = 8.4 Hz, 2 H), 7.69 (d, J = 8.4 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.7, 55.5, 114.5, 123.3, 128.6, 129.7, 132.4, 143.1, 145.2, 158.2 ppm. MS (EI): m/z (%) = 278 (44) [M$^+$], 155 (2), 123 (100), 91 (5). HRMS: calcd. for $C_{14}H_{14}O_4S$ 278.0613; found 278.0596.

2-(*p*-Toluenesulfonyloxy)benzonitrile (6): 68% (0.77 g) from 2-cyanophenol (0.50 g, 4.2 mmol). Colorless plates; m.p. 88–90 °C (hexane/AcOEt, ref.[15] 88–89 °C). IR (film): $\tilde{\nu}$ = 1194, 1383, 1485, 1599, 2235 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.46 (s, 3 H), 7.35–7.39 (m, 3 H), 7.50 (dd, J = 8.6, 0.8 Hz, 1 H), 7.58 (dd, J = 7.8, 1.3 Hz, 1 H), 7.63 (ddd, J = 8.6, 7.8, 1.3 Hz, 1 H), 7.82 (d, J = 8.8 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.7, 107.7, 114.4, 123.7, 127.3, 128.7, 130.0, 131.5, 133.7, 134.2, 146.3, 150.2 ppm. MS (EI): m/z (%) = 273 (21) [M$^+$], 155 (72), 91 (100). HRMS: calcd. for $C_{14}H_{11}NO_3S$ 273.0460; found 273.0457.

2-(*p*-Toluenesulfonyloxy)acetophenone (7): 80% (0.85 g) from 2-acetylphenol (0.50 g, 3.7 mmol). Colorless plates; m.p. 95–96 °C (hexane/AcOEt, ref.[16] 97–98 °C). IR (film): $\tilde{\nu}$ = 1198, 1373, 1479, 1601, 1693 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.46 (s, 3 H), 2.51 (s, 3 H), 7.10 (d, J = 8.4 Hz, 1 H), 7.31–7.35 (m, 3 H), 7.43 (ddd, J = 8.4, 8.0, 1.1 Hz, 1 H), 7.64 (dd, J = 8.4, 2.0 Hz, 1 H), 7.68 (d, J = 8.0 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.7, 30.4, 123.3, 127.2, 128.5, 129.9, 130.1, 132.0, 132.8, 133.8, 145.9, 147.1, 197.8 ppm. MS (EI): m/z (%) = 290 (2) [M$^+$], 275 (6), 155 (56), 121 (49), 91 (100). HRMS: calcd. for $C_{15}H_{14}O_4S$ 290.0613; found 290.0617.

1-(*p*-Toluenesulfonyloxy)naphthalene (8): 94% (0.97 g) from 1-naphthol (0.50 g, 3.5 mmol). Colorless plates; m.p. 89–91 °C (hexane/AcOEt, ref.[17] 90–92 °C). IR (film): $\tilde{\nu}$ = 1190, 1373, 1597 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.41 (s, 3 H), 7.21 (d, J = 8.0 Hz, 1 H), 7.27 (d, J = 8.4 Hz, 2 H), 7.36 (t, J = 8.0 Hz, 1 H), 7.40–7.48 (m, 2 H), 7.73 (d, J = 8.4 Hz, 1 H), 7.77–7.81 (m, 3 H), 7.90 (d, J = 8.4 Hz, 1 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.7, 118.4, 121.8, 125.1, 126.65, 126.68, 127.0, 127.3, 127.7, 128.5, 129.8, 132.9, 134.7, 145.4, 145.8 ppm. MS (EI): m/z (%) = 298 (100) [M$^+$], 155 (23), 143 (96), 115 (21), 91 (15). HRMS: calcd. for $C_{17}H_{14}O_3S$ 298.0664; found 298.0649.

2-(*p*-Toluenesulfonyloxy)naphthalene (9): 65% (0.67 g) from 2-naphthol (0.50 g, 3.5 mmol). Colorless needles; m.p. 119–120 °C (hexane/AcOEt, ref.[3c] 122–124 °C). IR (film): $\tilde{\nu}$ = 1190, 1377, 1595 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.44 (s, 3 H), 7.10 (dd, J = 9.0, 2.2 Hz, 1 H), 7.30 (d, J = 8.4 Hz, 2 H), 7.47–7.50 (m, 3 H), 7.72–7.76 (m, 4 H), 7.80–7.82 (m, 1 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.7, 119.9, 121.2, 126.3, 126.8, 127.7, 127.9, 128.6, 129.7, 129.8, 131.9, 132.5, 133.5, 145.3, 147.2 ppm. MS (EI): m/z (%) = 298 (100) [M$^+$], 155 (46), 143 (54), 115 (39), 91 (31). HRMS: calcd. for $C_{17}H_{14}O_3S$ 298.0664; found 298.0648.

3-(*p*-Toluenesulfonyloxy)pyridine (10): 43% (0.57 g) from 3-hydroxypyridine (0.50 g, 5.3 mmol). Colorless plates; m.p. 73–75 °C (hexane/AcOEt, ref.[5n] 72–73 °C). IR (film): $\tilde{\nu}$ = 1200, 1367, 1475, 1593 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.45 (s, 3 H), 7.29 (dd, J = 8.5, 4.8 Hz, 1 H), 7.34 (d, J = 8.2 Hz, 2 H), 7.46 (ddd, J

= 8.5, 2.6, 1.6 Hz, 1 H), 7.71 (d, *J* = 8.2 Hz, 2 H), 8.16 (d, *J* = 2.6 Hz, 1 H), 8.50 (dd, *J* = 4.8, 1.6 Hz, 1 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.7, 124.1, 128.5, 130.0, 130.1, 131.7, 144.0, 146.0, 146.4, 148.2 ppm. MS (EI): *m*/*z* (%) = 249 (57) [M$^+$], 155 (100), 91 (79). HRMS: calcd. for $C_{12}H_{11}NO_3S$ 249.0460; found 249.0442.

4-Methanesulfonyloxybenzonitrile (55): 96% (0.80 g) from 4-cyanophenol (0.50 g, 4.2 mmol). Colorless plates; m.p. 92–93 °C (hexane/AcOEt, ref.[18] 89–90 °C). IR (film): $\tilde{\nu}$ = 1153, 1202, 1362, 1499, 1601, 2233 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.23 (s, 3 H), 7.42 (d, *J* = 9.2 Hz, 2 H), 7.75 (d, *J* = 9.2 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 38.2, 111.5, 117.6, 123.0, 134.2, 151.9 ppm. MS (EI): *m*/*z* (%) = 197 (50) [M$^+$], 119 (100), 79 (24). HRMS: calcd. for $C_8H_7NO_3S$ 197.0146; found 197.0128.

4-Methanesulfonyloxyacetophenone (56): 97% (0.78 g) from 4-acetylphenol (0.50 g, 3.7 mmol). Colorless plates; m.p. 68–69 °C (hexane/AcOEt, ref.[18] 71–72 °C). IR (film): $\tilde{\nu}$ = 1157, 1175, 1205, 1375, 1501, 1597, 1682 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.61 (s, 3 H), 3.20 (s, 3 H), 7.38 (d, *J* = 8.8 Hz, 2 H), 8.03 (d, *J* = 8.8 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 26.6, 37.9, 122.0, 130.4, 135.9, 152.4, 196.4 ppm. MS (EI): *m*/*z* (%) = 214 (47) [M$^+$], 199 (100), 121 (57), 79 (4). HRMS: calcd. for $C_9H_{10}O_4S$ 214.0300; found 214.0269.

1-Methanesulfonyloxynaphthalene (57): 92% (0.76 g) from 1-naphthol (0.50 g, 3.5 mmol). Colorless plates; m.p. 35–36 °C (hexane/AcOEt). IR (film): $\tilde{\nu}$ = 1151, 1180, 1221, 1367, 1507, 1599 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.20 (s, 3 H), 7.46 (t, *J* = 8.0 Hz, 1 H), 7.51–7.61 (m, 3 H), 7.80 (d, *J* = 8.4 Hz, 1 H), 7.88 (d, *J* = 8.0 Hz, 1 H), 8.13 (d, *J* = 8.0 Hz, 1 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 37.9, 118.3, 121.4, 125.3, 126.9, 127.0, 127.2, 127.3, 128.0, 134.9, 145.3 ppm. MS (EI): *m*/*z* (%) = 222 (31) [M$^+$], 143 (71), 115 (100). HRMS: calcd. for $C_{11}H_{10}O_3S$ 222.0351; found 222.0340.

2-Methanesulfonyloxynaphthalene (58): 74% (0.61 g) from 2-naphthol (0.50 g, 3.5 mmol). Colorless prisms; m.p. 101–102 °C (hexane/AcOEt, ref.[19] 103.5–104.5 °C). IR (film): $\tilde{\nu}$ = 1177, 1209, 1364, 1508 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.13 (s, 3 H), 7.41 (dd, *J* = 9.0, 2.6 Hz, 1 H), 7.50–7.57 (m, 2 H), 7.76 (d, *J* = 2.0 Hz, 1 H), 7.84–7.91 (m, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 37.4, 119.4, 120.8, 126.6, 127.1, 127.8, 127.9, 130.3, 132.1, 133.6, 146.8 ppm. MS (EI): *m*/*z* (%) = 222 (32) [M$^+$], 143 (40), 115 (100). HRMS: calcd. for $C_{11}H_{10}O_3S$ 222.0351; found 222.0341.

3-Methanesulfonyloxypyridine (59): 51% (0.46 g) from 3-hydroxypyridine (0.50 g, 5.3 mmol). Colorless needles; m.p. 57–58 °C (hexane/AcOEt, ref.[20] 60 °C). IR (film): $\tilde{\nu}$ = 1178, 1202, 1377, 1479, 1576, 1587, 3421 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.22 (s, 3 H), 7.40 (dd, *J* = 8.5, 4.8 Hz, 1 H), 7.68 (dt, *J* = 8.5, 1.4 Hz, 1 H), 8.59–8.60 (m, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 37.8, 124.4, 129.7, 143.6, 146.0, 148.5 ppm. MS (EI): *m*/*z* (%) = 173 (88) [M$^+$]. HRMS: calcd. for $C_6H_7NO_3S$ 173.0147; found 173.0110.

Typical Procedure for Tosylation of Enols [4-(*p*-Toluenesulfonyloxy)-coumarin (37)]: A mixture of 4-hydroxycoumarin (0.50 g, 3.1 mmol), TsCl (0.70 g, 3.7 mmol), Et_3N (0.37 g, 3.7 mmol), and CH_2Cl_2 (15 mL) was stirred for 0.5 h at room temperature. The solvent was removed under reduced pressure, and the residue was purified by silica gel column chromatography (hexane/AcOEt, 4:1) to obtain a colorless solid (0.92 g, 94%). The solid was recrystallized from hexane/AcOEt to obtain colorless plates.

4-(*p*-Toluenesulfonyloxy)coumarin (37): 94% (0.92 g) from 4-hydroxycoumarin (0.50 g, 3.1 mmol). Colorless plates; m.p. 110–111 °C (hexane/AcOEt). IR (film): $\tilde{\nu}$ = 1069, 1194, 1371, 1489, 1607, 1628, 1732 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.47 (s, 3 H), 6.31 (s, 1 H), 7.25–729 (m, 1 H), 7.31 (d, *J* = 8.0 Hz, 1 H), 7.39 (d, *J* = 8.2 Hz, 2 H), 7.57 (t, *J* = 8.0 Hz, 1 H), 7.64 (d, *J* = 8.0 Hz, 1 H), 7.90 (d, *J* = 8.2 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.9, 103.6, 114.9, 116.9, 123.1, 124.5, 128.4, 130.3, 131.6, 133.2, 146.8, 153.4, 157.7, 160.6 ppm. MS (EI): *m*/*z* (%) = 316 (24) [M$^+$], 252 (69), 155 (100), 132 (20), 91 (64). HRMS: calcd. for $C_{16}H_{12}O_5S$ 316.0405; found 316.0387.

6-Methyl-4-(*p*-toluenesulfonyloxy)coumarin (38): 70% (0.39 g) from 4-hydroxy-6-methylcoumarin (0.30 g, 1.7 mmol). Colorless prisms; m.p. 149–151 °C (hexane/AcOEt). IR (film): $\tilde{\nu}$ = 1061, 1200, 1364, 1489, 1597, 1630, 1732 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.37 (s, 3 H), 2.47 (s, 3 H), 6.26 (s, 1 H), 7.19 (d, *J* = 8.4 Hz, 1 H), 7.35–7.41 (m, 4 H), 7.90 (d, *J* = 8.8 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 20.8, 21.7, 103.5, 114.6, 116.6, 122.7, 128.4, 130.3, 131.8, 134.2, 134.4, 146.8, 151.6, 157.8, 160.9 ppm. MS (EI): *m*/*z* (%) = 330 (100) [M$^+$], 266 (55), 212 (11), 176 (10), 155 (100), 132 (22), 91 (74). HRMS: calcd. for $C_{17}H_{14}O_5S$ 330.0562; found 330.0548.

7-Methoxy-4-(*p*-toluenesulfonyloxy)coumarin (39): Quantitative yield (0.54 g) from 4-hydroxy-7-methoxycoumarin (0.30 g, 1.6 mmol). Colorless prisms; m.p. 95–97 °C (hexane/AcOEt). IR (film): $\tilde{\nu}$ = 1067, 1192, 1211, 1379, 1510, 1618, 1728 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.47 (s, 3 H), 3.86 (s, 3 H), 6.12 (s, 1 H), 6.78 (d, *J* = 2.5 Hz, 1 H), 6.82 (dd, *J* = 8.9, 2.5 Hz, 1 H), 7.39 (d, *J* = 8.2 Hz, 2 H), 7.53 (d, *J* = 8.9 Hz, 1 H), 7.89 (d, *J* = 8.2 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.8, 55.8, 100.6, 100.8, 108.2, 112.8, 124.3, 128.4, 130.3, 131.8, 146.7, 155.5, 158.2, 161.3, 163.9 ppm. MS (EI): *m*/*z* (%) = 346 (97) [M$^+$], 282 (97), 155 (86), 132 (100), 91 (79). HRMS: calcd. for $C_{17}H_{14}O_6S$ 346.0511; found 346.0479.

6-Methyl-4-(*p*-toluenesulfonyloxy)-2-pyranone (40): 94% (1.0 g) from 4-hydroxy-6-methyl-2-pyranone (0.50 g, 4.0 mmol). Colorless prisms; m.p. 100–102 °C (hexane/AcOEt, ref.[21] 101.2–102.0 °C). IR (film): $\tilde{\nu}$ = 1180, 1194, 1387, 1570, 1641, 1740 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.24 (s, 3 H), 2.48 (s, 3 H), 5.81 (s, 1 H), 6.00 (s, 1 H), 7.39 (d, *J* = 8.3 Hz, 2 H), 7.82 (d, *J* = 8.3 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 20.1, 21.8, 100.68, 100.73, 128.4, 130.3, 131.7, 146.7, 161.9, 162.8, 164.2 ppm. MS (EI): *m*/*z* (%) = 280 (34) [M$^+$], 167 (16), 155 (100), 132 (96), 91 (87). HRMS: calcd. for $C_{13}H_{12}O_5S$ 280.0405; found 280.0399.

1-Methyl-4-(*p*-toluenesulfonyloxy)-2-quinolone (41): 91% (0.85 g) from 4-hydroxy-1-methyl-2-quinolone (0.50 g, 2.9 mmol). Colorless plates; m.p. 153–155 °C (hexane/AcOEt, ref.[22] 156 °C). IR (film): $\tilde{\nu}$ = 1080, 1178, 1377, 1456, 1499, 1595, 1661 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.45 (s, 3 H), 3.66 (s, 3 H), 6.42 (s, 1 H), 7.22–7.26 (m, 1 H), 7.34–7.37 (m, 3 H), 7.60 (ddd, *J* = 8.3, 7.3, 1.4 Hz, 1 H), 7.81 (dd, *J* = 8.3, 1.4 Hz, 1 H), 7.87 (d, *J* = 8.3 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.6, 29.4, 110.5, 114.2, 116.1, 122.3, 123.6, 128.3, 130.1, 132.0, 132.1, 139.9, 146.2, 154.1, 162.0 ppm. MS (EI): *m*/*z* (%) = 329 (100) [M$^+$], 279 (22), 265 (44), 217 (13), 174 (57), 167 (33), 91 (34). HRMS: calcd. for $C_{17}H_{15}NO_4S$ 329.0722; found 329.0743.

Typical Procedure for Nickel(II)–Pincer Complex Catalyzed Suzuki–Miyaura Coupling Reaction: A mixture of **2** (27.3 mg, 0.10 mmol), $PhB(OH)_2$ (36.6 mg, 0.30 mmol), complex **1a** (3.3 mg, 0.0050 mmol), K_3PO_4 (43.0 mg, 0.20 mmol), and DME (1 mL) in a sealed tube was heated to 120 °C for 24 h. After cooling to room temperature, the reaction mixture was treated with H_2O (15 mL) followed by extraction with AcOEt (3 × 15 mL). The combined extracts were washed with saturated aqueous solution of NaCl (3 × 15 mL), and the solvent was removed under reduced pressure.

The residue was purified by silica gel column chromatography (hexane/AcOEt, 19:1) to obtain a colorless solid (17.1 mg, 95%; Table 1, Entry 8).

4-Cyanobiphenyl (3): 95% (17.1 mg) from **2** (27.3 mg, 0.10 mmol) (Table 1, Entry 8); 63% (11.3 mg) from **55** (19.7 mg, 0.10 mmol) (Table 4, Entry 8; Table 5, Entry 1). Colorless prisms; m.p. 85–86 °C (hexane, ref.[4b] 84–86 °C). IR (film): $\tilde{\nu}$ = 2228 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 7.40–7.50 (m, 3 H), 7.58 (dd, J = 7.2, 1.2 Hz, 2 H), 7.66–7.73 (m, 4 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 110.8, 118.9, 127.1, 127.6, 128.6, 129.0, 132.5, 139.0, 145.5 ppm. MS (EI): m/z (%) = 179 (100) [M^+]. HRMS: calcd. for $C_{13}H_9N$ 179.0735; found 179.0739.

4-Cyano-4′-methoxybiphenyl (11): 55% (11.5 mg) from **2** (27.3 mg, 0.10 mmol) (Table 2, Entry 1); 24% (5.0 mg) from **55** (19.7 mg, 0.10 mmol) (Table 5, Entry 2). Colorless plates; m.p. 101–102 °C (hexane/AcOEt, ref.[23] 103–104 °C). IR (film): $\tilde{\nu}$ = 1038, 1607, 2224 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 3.86 (s, 3 H), 7.00 (d, J = 8.8 Hz, 2 H), 7.53 (d, J = 8.8 Hz, 2 H), 7.63 (d, J = 8.0 Hz, 2 H), 7.69 (d, J = 8.0 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 55.4, 110.1, 114.6, 119.1, 127.1, 128.3, 131.5, 132.6, 145.2, 160.2 ppm. MS (EI): m/z (%) = 209 (100) [M^+], 194 (29), 166 (20). HRMS: calcd. for $C_{14}H_{11}NO$ 209.0841; found 209.0826.

4-Cyano-3′-methylbiphenyl (12): 59% (11.4 mg) from **2** (27.3 mg, 0.10 mmol) (Table 2, Entry 2). Colorless oil. IR (neat): $\tilde{\nu}$ = 1607, 2226 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 2.43 (s, 3 H), 7.23–7.25 (m, 1 H), 7.34–7.39 (m, 3 H), 7.67 (d, J = 8.2 Hz, 2 H), 7.71 (d, J = 8.2 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.6, 110.7, 118.9, 124.3, 127.6, 127.9, 128.9, 129.3, 132.4, 138.7, 139.1, 145.7 ppm. MS (EI): m/z (%) = 193 (100) [M^+], 178 (9). HRMS: calcd. for $C_{14}H_{11}N$ 193.0892; found 193.0907.

4-Cyano-2′-methylbiphenyl (13): 52% (10.0 mg) from **2** (27.3 mg, 0.10 mmol) (Table 2, Entry 3). Colorless prisms; m.p. 54–56 °C (hexane/AcOEt). IR (film): $\tilde{\nu}$ = 1609, 2226 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 2.25 (s, 3 H), 7.18 (d, J = 7.2 Hz, 1 H), 7.25–7.33 (m, 3 H), 7.43 (d, J = 7.6 Hz, 2 H), 7.70 (d, J = 7.6 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 20.4, 110.7, 118.9, 126.0, 128.2, 129.3, 129.9, 130.6, 131.9, 134.9, 139.9, 146.7 ppm. MS (EI): m/z (%) = 193 (100) [M^+], 178 (14). HRMS: calcd. for $C_{14}H_{11}N$ 193.0892; found 193.0904.

4-Cyano-2′-methoxybiphenyl (14): 58% (12.1 mg) from **2** (27.3 mg, 0.10 mmol) (Table 2, Entry 4). Colorless prisms; m.p. 72–74 °C (hexane/AcOEt). IR (film): $\tilde{\nu}$ = 1263, 1607, 2226 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 3.82 (s, 3 H), 6.96–7.07 (m, 2 H), 7.29 (dd, J = 7.6, 1.1 Hz, 1 H), 7.38 (ddd, J = 8.3, 7.6, 1.1 Hz, 1 H), 7.63 (d, J = 8.4 Hz, 2 H), 7.68 (d, J = 8.4 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 55.6, 110.4, 111.3, 119.1, 120.3, 121.0, 129.8, 130.1, 130.5, 131.7, 143.3, 156.2 ppm. MS (EI): m/z (%) = 209 (100) [M^+], 194 (25), 166 (9). HRMS: calcd. for $C_{14}H_{11}NO$ 209.0841; found 209.0818.

4-(*E*)-Pentenylbenzonitrile (15): 74% (12.7 mg) from **2** (27.3 mg, 0.10 mmol) (Table 2, Entry 5). Colorless oil. IR (neat): $\tilde{\nu}$ = 1651, 2224 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 0.96 (t, J = 7.2 Hz, 3 H), 1.51 (sext., J = 7.2 Hz, 2 H), 2.20–2.25 (m, 2 H), 6.33–6.42 (m, 2 H), 7.40 (d, J = 8.4 Hz, 2 H), 7.56 (d, J = 8.4 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.8, 22.3, 35.2, 109.8, 119.1, 126.3, 128.5, 132.2, 135.2, 142.3 ppm. MS (EI): m/z (%) = 171 (60) [M^+], 142 (74), 129 (100), 115 (19). HRMS: calcd. for $C_{12}H_{13}N$ 171.1047; found 171.1029.

4-Acetylbiphenyl (16): 78% (15.3 mg) from **4** (29.0 mg, 0.10 mmol) (Table 2, Entry 6); 46% (9.0 mg) from **56** (21.4 mg, 0.10 mmol) (Table 5, Entry 3). Colorless needles; m.p. 121–122 °C (hexane, ref.[4b] 117–119 °C). IR (film): $\tilde{\nu}$ = 1680 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 2.64 (s, 3 H), 7.40 (t, J = 7.8 Hz, 1 H), 7.40 (t, J = 7.8 Hz, 2 H), 7.47 (t, J = 7.8 Hz, 2 H), 7.47 (d, J = 8.4 Hz, 2 H), 8.03 (d, J = 8.4 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 26.9, 127.26, 127.31, 128.3, 128.96, 129.00, 135.9, 139.9, 145.8, 197.7 ppm. MS (EI): m/z (%) = 196 (64) [M^+], 181 (100). HRMS: calcd. for $C_{14}H_{12}O$ 196.0888; found 196.0884.

4-Acetyl-4′-methoxybiphenyl (17): 71% (16.0 mg) from **4** (29.0 mg, 0.10 mmol) (Table 2, Entry 7); 38% (8.6 mg) from **56** (21.4 mg, 0.10 mmol) (Table 5, Entry 4). Colorless plates; m.p. 152–153 °C (hexane/AcOEt, ref.[24] 154–155 °C). IR (film): $\tilde{\nu}$ = 1296, 1601, 1674 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 2.63 (s, 3 H), 3.86 (s, 3 H), 7.00 (d, J = 8.8 Hz, 2 H), 7.57 (d, J = 8.8 Hz, 2 H), 7.64 (d, J = 8.2 Hz, 2 H), 8.00 (d, J = 8.2 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 26.6, 55.4, 114.4, 126.6, 128.4, 128.9, 132.3, 135.3, 145.4, 159.9, 197.7 ppm. MS (EI): m/z (%) = 226 (94) [M^+], 211 (100), 183 (14), 168 (10). HRMS: calcd. for $C_{15}H_{14}O_2$ 226.0994; found 226.0983.

4-Acetyl-4′-cyanobiphenyl (18): 48% (10.6 mg) from **4** (29.0 mg, 0.10 mmol) (Table 2, Entry 8). Colorless prisms; m.p. 113–114 °C (hexane/AcOEt, ref.[25] 115–116 °C). IR (film): $\tilde{\nu}$ = 1603, 1684, 2226 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 2.65 (s, 3 H), 7.68 (d, J = 8.2 Hz, 2 H), 7.72 (d, J = 8.2 Hz, 2 H), 7.76 (d, J = 8.2 Hz, 2 H), 8.07 (d, J = 8.2 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 26.8, 111.9, 118.5, 127.4, 127.8, 129.0, 132.6, 136.8, 143.4, 144.2, 197.3 ppm. MS (EI): m/z (%) = 221 (56) [M^+], 206 (100), 178 (24). HRMS: calcd. for $C_{15}H_{11}NO$ 221.0841; found 221.0833.

4-Acetyl-3′-methylbiphenyl (19): 79% (16.6 mg) from **4** (29.0 mg, 0.10 mmol) (Table 2, Entry 9). Colorless oil. IR (neat): $\tilde{\nu}$ = 1684 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 2.43 (s, 3 H), 2.63 (s, 3 H), 7.33–7.37 (m, 2 H), 7.41–7.43 (m, 2 H), 7.67 (d, J = 8.6 Hz, 2 H), 8.01 (d, J = 8.6 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.6, 26.7, 124.3, 127.1, 127.9, 128.8, 128.9, 131.8, 135.7, 138.5, 139.8, 145.8, 197.6 ppm. MS (EI): m/z (%) = 210 (84) [M^+], 195 (100), 167 (12), 152 (10). HRMS: calcd. for $C_{15}H_{14}O$ 210.1045; found 210.1026.

4-(*E*)-Pentenylacetophenone (20): 69% (13.0 mg) from **4** (29.0 mg, 0.10 mmol) (Table 2, Entry 10). Colorless oil. IR (neat): $\tilde{\nu}$ = 1603, 1682 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 0.96 (t, J = 7.2 Hz, 3 H), 1.52 (sext., J = 7.2 Hz, 2 H), 2.22 (q, J = 7.2 Hz, 2 H), 2.58 (s, 3 H), 6.35–6.44 (m, 2 H), 7.41 (d, J = 8.4 Hz, 2 H), 7.88 (d, J = 8.4 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.8, 22.4, 26.6, 35.3, 125.8, 128.7, 129.0, 134.2, 135.3, 142.6, 197.5 ppm. MS (EI): m/z (%) = 188 (100) [M^+], 173 (88), 146 (19), 131 (29), 43 (36). HRMS: calcd. for $C_{13}H_{16}O$ 188.1200; found 188.1208.

4-Methoxybiphenyl (21): 20% (3.7 mg) from **5** (27.8 mg, 0.10 mmol) (Table 2, Entry 11). Colorless prisms; m.p. 86–87 °C (hexane, ref.[4b] 85–87 °C). IR (film): $\tilde{\nu}$ = 1036, 1609 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 3.83 (s, 3 H), 6.96 (d, J = 8.8 Hz, 2 H), 7.29 (t, J = 7.5 Hz, 1 H), 7.40 (t, J = 7.5 Hz, 2 H), 7.51–7.55 (m, 4 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 55.3, 114.1, 126.56, 126.64, 128.0, 128.6, 133.7, 140.7, 159.0 ppm. MS (EI): m/z (%) = 184 (100) [M^+], 169 (43), 141 (26). HRMS: calcd. for $C_{13}H_{12}O$ 184.0888; found 184.0886.

2-Cyanobiphenyl (22): 17% (3.0 mg) from **6** (27.3 mg, 0.10 mmol) (Table 2, Entry 12). Colorless oil. IR (neat): $\tilde{\nu}$ = 2224 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 7.39–7.50 (m, 5 H), 7.54–7.56 (m, 2 H), 7.62 (td, J = 7.8, 1.3 Hz, 1 H), 7.74 (dd, J = 7.8, 1.3 Hz, 1 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 111.1, 118.6, 127.4, 128.59, 128.61, 128.64, 130.0, 132.7, 133.6, 138.0, 145.4 ppm. MS (EI): m/z (%) = 179 (100) [M^+]. HRMS: calcd. for $C_{13}H_9N$ 179.0735; found 179.0725.

2-Acetylbiphenyl (23): 9% (1.8 mg) from **7** (29.0 mg, 0.10 mmol) (Table 2, Entry 13). Colorless oil. IR (neat): $\tilde{\nu}$ = 1688 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 2.00 (s, 3 H), 7.32–7.44 (m, 7 H), 7.50 (td, J = 7.5, 1.5 Hz, 1 H), 7.56 (dd, J = 7.5, 0.8 Hz, 1 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 30.5, 127.3, 127.76, 127.78, 128.6, 128.7, 130.1, 130.6, 140.4, 140.6, 140.8, 204.7 ppm. MS (EI): m/z (%) = 196 (74) [M^+], 181 (100). HRMS: calcd. for $C_{14}H_{12}O$ 196.0888; found 196.0860.

1-Phenylnaphthalene (24): 73% (14.9 mg) from **8** (29.8 mg, 0.10 mmol) (Table 2, Entry 14); 83% (16.9 mg) from **57** (22.2 mg, 0.10 mmol) (Table 5, Entry 5). Colorless oil. ^1H NMR (400 MHz, $CDCl_3$): δ = 7.39–7.53 (m, 9 H), 7.84–7.90 (m, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 125.3, 125.7, 125.92, 125.94, 126.8, 127.1, 127.5, 128.2 (2 C), 130.0, 131.5, 133.7, 140.2, 140.7 ppm. MS (EI): m/z (%) = 204 (100) [M^+]. HRMS: calcd. for $C_{16}H_{12}$ 204.0939; found 204.0921.

1-(4-Methoxyphenyl)naphthalene (25): 66% (15.4 mg) from **8** (29.8 mg, 0.10 mmol) (Table 2, Entry 15); 59% (13.8 mg) from **57** (22.2 mg, 0.10 mmol) (Table 5, Entry 6). Colorless prisms; m.p. 113–115 °C (hexane/AcOEt, ref.[26] 111–112 °C). IR (film): $\tilde{\nu}$ = 1034, 1609 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 3.88 (s, 3 H), 7.02 (d, J = 8.2 Hz, 2 H), 7.39–7.52 (m, 6 H), 7.82 (d, J = 8.2 Hz, 1 H), 7.88–7.93 (m, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 55.4, 113.7, 125.3, 125.6, 125.8, 126.0, 126.8, 127.2, 128.2, 131.0, 131.7, 133.0, 133.7, 139.8, 158.8 ppm. MS (EI): m/z (%) = 234 (100) [M^+], 219 (31). HRMS: calcd. for $C_{17}H_{14}O$ 234.1045; found 234.1028.

1-(4-Cyanophenyl)naphthalene (26): 91% (20.8 mg) from **8** (29.8 mg, 0.10 mmol) (Table 2, Entry 16); 30% (6.9 mg) from **57** (22.2 mg, 0.10 mmol) (Table 5, Entry 7). Colorless prisms; m.p. 79–81 °C (hexane/AcOEt, ref.[27] 76–77 °C). IR (film): $\tilde{\nu}$ = 1607, 2228 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 7.39 (dd, J = 7.0, 1.0 Hz, 1 H), 7.46 (t, J = 7.0 Hz, 1 H), 7.50–7.56 (m, 2 H), 7.60 (d, J = 8.4 Hz, 2 H), 7.75–7.79 (m, 3 H), 7.90–7.94 (m, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 111.1, 118.9, 125.1, 125.2, 126.1, 126.5, 126.9, 128.4, 128.7, 130.7, 130.8, 132.0, 133.7, 138.1, 145.5 ppm. MS (EI): m/z (%) = 229 (100) [M^+]. HRMS: calcd. for $C_{17}H_{11}N$ 229.0892; found 229.0896.

1-(3-Methylphenyl)naphthalene (27): 72% (15.7 mg) from **8** (29.8 mg, 0.10 mmol) (Table 2, Entry 17). Colorless oil. ^1H NMR (400 MHz, $CDCl_3$): δ = 2.43 (s, 3 H), 7.23–7.24 (m, 1 H), 7.28–7.32 (m, 2 H), 7.35–7.52 (m, 5 H), 7.84 (d, J = 8.0 Hz, 1 H), 7.88–7.91 (m, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.6, 125.3, 125.6, 125.9, 126.0, 126.7, 127.1, 127.4, 127.9, 128.0, 128.1, 130.7, 131.6, 133.7, 137.7, 140.3, 140.6 ppm. MS (EI): m/z (%) = 218 (100) [M^+], 203 (70). HRMS: calcd. for $C_{17}H_{14}$ 218.1096; found 218.1078.

1-(*E*)-Pentenylnaphthalene (28): 49% (9.6 mg) from **8** (29.8 mg, 0.10 mmol) (Table 2, Entry 18). Colorless oil. ^1H NMR (400 MHz, $CDCl_3$): δ = 1.01 (t, J = 7.3 Hz, 3 H), 1.58 (sext., J = 7.3 Hz, 2 H), 2.31 (dt, J = 7.3, 7.1 Hz, 2 H), 6.23 (dt, J = 15.6, 7.1 Hz, 1 H), 7.11 (d, J = 15.6 Hz, 1 H), 7.40–7.52 (m, 3 H), 7.55 (d, J = 7.0 Hz, 1 H), 7.73 (d, J = 7.8 Hz, 1 H), 7.82 (dd, J = 7.8, 2.4 Hz, 1 H), 8.12 (d, J = 7.8 Hz, 1 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.9, 22.7, 35.6, 123.4, 123.9, 125.5, 125.6, 125.7, 127.0, 127.1, 128.3, 131.0, 133.5, 134.2, 135.7 ppm. MS (EI): m/z (%) = 196 (69) [M^+], 167 (100), 153 (26), 128 (3). HRMS: calcd. for $C_{15}H_{16}$ 196.1252; found 196.1237.

2-Phenylnaphthalene (29): 76% (15.5 mg) from **9** (29.8 mg, 0.10 mmol) (Table 2, Entry 19); 73% (14.9 mg) from **58** (22.2 mg, 0.10 mmol) (Table 5, Entry 8). Colorless plates; m.p. 99–100 °C (hexane, ref.[28] 100–101 °C). ^1H NMR (400 MHz, $CDCl_3$): δ = 7.37 (t, J = 7.4 Hz, 1 H), 7.43–7.51 (m, 4 H), 7.71–7.75 (m, 3 H), 7.85–7.91 (m, 3 H), 8.03 (s, 1 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 125.6, 125.8, 125.9, 126.3, 127.3, 127.4, 127.6, 128.2, 128.4, 128.8, 132.6, 133.7, 138.6, 141.1 ppm. MS (EI): m/z (%) = 204 (100) [M^+]. HRMS: calcd. for $C_{16}H_{12}$ 204.0939; found 204.0923.

2-(4-Methoxyphenyl)naphthalene (30): 65% (15.2 mg) from **9** (29.8 mg, 0.10 mmol) (Table 2, Entry 20); 69% (16.1 mg) from **58** (22.2 mg, 0.10 mmol) (Table 5, Entry 9). Colorless plates; m.p. 130–132 °C (hexane/AcOEt, ref.[28] 131–133 °C). IR (film): $\tilde{\nu}$ = 1283, 1607 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 3.86 (s, 3 H), 7.02 (dt, J = 8.9, 2.0 Hz, 2 H), 7.43–7.50 (m, 2 H), 7.66 (dt, J = 8.9, 2.0 Hz, 2 H), 7.71 (dd, J = 8.6, 1.8 Hz, 1 H), 7.83–7.89 (m, 3 H), 7.98 (s, 1 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 55.4, 114.3, 125.0, 125.4, 125.6, 126.2, 127.6, 128.0, 128.3, 128.4, 132.3, 133.6, 133.8, 138.2, 159.3 ppm. MS (EI): m/z (%) = 234 (100) [M^+], 219 (31), 191 (10). HRMS: calcd. for $C_{17}H_{14}O$ 234.1045; found 234.1026.

2-(4-Cyanophenyl)naphthalene (31): 51% (11.7 mg) from **9** (29.8 mg, 0.10 mmol) (Table 2, Entry 21). Colorless prisms; m.p. 148–150 °C (hexane/AcOEt). IR (film): $\tilde{\nu}$ = 2224 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 7.51–7.56 (m, 2 H), 7.70 (dd, J = 8.4, 1.6 Hz, 1 H), 7.75 (d, J = 8.4 Hz, 2 H), 7.81 (d, J = 8.4 Hz, 2 H), 7.87–7.96 (m, 3 H), 8.05 (s, 1 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 110.9, 118.9, 124.8, 126.5, 126.67, 126.70, 127.6, 127.9, 128.3, 128.9, 132.6, 133.1, 133.4, 136.3, 145.5 ppm. MS (EI): m/z (%) = 229 (100) [M^+]. HRMS: calcd. for $C_{17}H_{11}N$ 229.0892; found 229.0886.

2-(2-Methylphenyl)naphthalene (32): 57% (12.4 mg) from **9** (29.8 mg, 0.10 mmol) (Table 2, Entry 22). Colorless oil. ^1H NMR (400 MHz, $CDCl_3$): δ = 2.31 (s, 3 H), 7.27–7.33 (m, 4 H), 7.45–7.51 (m, 3 H), 7.76 (s, 1 H), 7.84–7.88 (m, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 20.6, 125.7, 125.8, 126.1, 127.3, 127.4, 127.6, 127.65, 127.69, 127.9, 129.9, 130.3, 132.2, 133.2, 135.5, 139.4, 141.8 ppm. MS (EI): m/z (%) = 218 (100) [M^+], 203 (30). HRMS: calcd. for $C_{17}H_{14}$ 229.1096; found 229.1079.

2-(*E*)-Pentenylnaphthalene (33): 55% (10.8 mg) from **9** (29.8 mg, 0.10 mmol) (Table 2, Entry 23); 28% (5.5 mg) from **58** (22.2 mg, 0.10 mmol) (Table 5, Entry 10). Colorless oil. ^1H NMR (400 MHz, $CDCl_3$): δ = 0.98 (t, J = 7.3 Hz, 3 H), 1.54 (sext., J = 7.3 Hz, 2 H), 2.24 (dt, J = 7.3, 7.1 Hz, 2 H), 6.35 (dt, J = 16.0, 7.3 Hz, 1 H), 6.54 (d, J = 16.0 Hz, 1 H), 7.37–7.45 (m, 2 H), 7.57 (d, J = 8.4 Hz, 1 H), 7.66 (s, 1 H), 7.74–7.81 (m, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 13.9, 22.7, 35.3, 123.5, 125.2, 125.3, 126.1, 127.5, 127.7, 127.9, 129.9, 131.4, 132.5, 133.6, 135.3 ppm. MS (EI): m/z (%) = 196 (77) [M^+], 167 (100), 141 (16), 128 (7). HRMS: calcd. for $C_{15}H_{16}$ 196.1252; found 196.1250.

3-Phenylpyridine (34): 69% (10.7 mg) from **10** (24.9 mg, 0.10 mmol) (Table 2, Entry 24); 36% (5.6 mg) from **59** (17.3 mg, 0.10 mmol) (Table 5, Entry 11). Pale yellow oil. IR (neat): $\tilde{\nu}$ = 3396 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 7.35–7.43 (m, 2 H), 7.48 (t, J = 7.4 Hz, 2 H), 7.58 (d, J = 7.4 Hz, 2 H), 7.87 (dt, J = 7.8, 1.9 Hz, 1 H), 8.59 (d, J = 4.0 Hz, 1 H), 8.85 (s, 1 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 123.4, 127.1, 128.0, 129.0, 134.2, 136.5, 137.7, 148.2, 148.3 ppm. MS (EI): m/z (%) = 155 (100) [M^+]. HRMS: calcd. for $C_{11}H_9N$ 155.0735; found 155.0738.

3-(4-Methoxyphenyl)pyridine (35): 86% (15.9 mg) from **10** (24.9 mg, 0.10 mmol) (Table 2, Entry 25). Colorless prisms; m.p. 60–61 °C (hexane/AcOEt, ref.[29] 60–61 °C). IR (film): $\tilde{\nu}$ = 1032, 1611, 3373 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 3.86 (s, 3 H), 7.01 (d, J = 8.2 Hz, 2 H), 7.33 (dd, J = 7.7, 4.7 Hz, 1 H), 7.52 (d, J = 8.2 Hz, 2 H), 7.83 (d, J = 7.7 Hz, 1 H), 8.54 (d, J = 4.7 Hz, 1 H),

8.81 (s, 1 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 55.4, 114.5, 123.4, 128.1, 130.2, 133.7, 136.1, 147.8, 147.9, 159.6 ppm. MS (EI): *m*/*z* (%) = 185 (100) [M$^+$], 170 (43). HRMS: calcd. for $C_{12}H_{11}NO$ 185.0841; found 185.0851.

3-(4-Cyanophenyl)pyridine (36): 83% (14.9 mg) from **10** (24.9 mg, 0.10 mmol) (Table 2, Entry 26). Colorless prisms; m.p. 94–96 °C (hexane/AcOEt, ref.[30] 95–96 °C). IR (film): $\tilde{\nu}$ = 2226, 3358 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.42 (t, *J* = 6.7 Hz, 1 H), 7.69 (d, *J* = 8.2 Hz, 2 H), 7.78 (d, *J* = 8.2 Hz, 2 H), 7.89 (d, *J* = 6.7 Hz, 1 H), 8.67 (s, 1 H), 8.86 (s, 1 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 111.9, 118.5, 123.7, 127.7, 132.8, 134.4, 134.7, 142.2, 148.1, 149.6 ppm. MS (EI): *m*/*z* (%) = 180 (100) [M$^+$]. HRMS: calcd. for $C_{13}H_8N_2$ 180.0687; found 180.0670.

4-Phenylcoumarin (42): 73% (16.2 mg) from **37** (31.6 mg, 0.10 mmol) (Table 3, Entry 1). Pale yellow oil. IR (neat): $\tilde{\nu}$ = 1180, 1604, 1724 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 6.38 (s, 1 H), 7.23 (dd, *J* = 8.4, 7.2 Hz, 1 H), 7.40–7.57 (m, 8 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 115.2, 117.3, 119.0, 124.1, 126.9, 128.4, 128.8, 129.6, 131.8, 135.1, 154.1, 155.5, 160.6 ppm. MS (EI): *m*/*z* (%) = 222 (100) [M$^+$], 194 (58), 165 (19). HRMS: calcd. for $C_{15}H_{10}O_2$ 222.0681; found 222.0665.

4-(4-Methoxyphenyl)coumarin (43): 90% (22.7 mg) from **37** (31.6 mg, 0.10 mmol) (Table 3, Entry 2). Colorless needles; m.p. 130–131 °C (hexane/AcOEt, ref.[31] 119–120 °C). IR (film): $\tilde{\nu}$ = 1180, 1248, 1510, 1605, 1726 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.89 (s, 3 H), 6.34 (s, 1 H), 7.04 (d, *J* = 8.4 Hz, 2 H), 7.23 (t, *J* = 7.6 Hz, 1 H), 7.37–7.41 (m, 3 H), 7.52–7.57 (m, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 55.4, 114.2, 114.5, 117.2, 119.0, 123.9, 126.9, 127.3, 129.8, 131.6, 154.1, 155.1, 160.7 (2 C) ppm. MS (EI): *m*/*z* (%) = 252 (100) [M$^+$], 224 (47), 209 (19). HRMS: calcd. for $C_{16}H_{12}O_3$ 252.0786; found 252.0785.

4-(4-Cyanophenyl)coumarin (44): 44% (10.9 mg) from **37** (31.6 mg, 0.10 mmol) (Table 3, Entry 3). Colorless needles; m.p. 250–251 °C (hexane/AcOEt). IR (film): $\tilde{\nu}$ = 1182, 1603, 1722, 2220 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 6.38 (s, 1 H), 7.24–7.27 (m, 1 H), 7.33 (d, *J* = 7.2 Hz, 1 H), 7.44 (d, *J* = 8.4 Hz, 1 H), 7.57–7.61 (m, 3 H), 7.84 (d, *J* = 8.4 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 113.7, 115.9, 117.6, 117.9, 118.1, 124.4, 126.2, 129.2, 132.4, 132.6, 139.6, 153.4, 154.1, 159.9 ppm. MS (EI): *m*/*z* (%) = 247 (100) [M$^+$], 219 (78), 190 (18). HRMS: calcd. for $C_{16}H_9NO_2$ 247.0633; found 247.0629.

6-Methyl-4-phenylcoumarin (45): 62% (14.6 mg) from **38** (33.0 mg, 0.10 mmol) (Table 3, Entry 4). Colorless oil. IR (neat): $\tilde{\nu}$ = 1180, 1616, 1724 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.33 (s, 3 H), 6.35 (s, 1 H), 7.24–7.25 (m, 1 H), 7.30 (d, *J* = 8.6 Hz, 1 H), 7.35 (dd, *J* = 8.6, 1.6 Hz, 1 H), 7.43–7.45 (m, 2 H), 7.52–7.54 (m, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 21.0, 115.1, 117.0, 118.6, 126.6, 128.3, 128.8, 129.5, 132.8, 133.8, 135.3, 152.2, 155.5, 160.8 ppm. MS (EI): *m*/*z* (%) = 236 (100) [M$^+$], 208 (41). HRMS: calcd. for $C_{16}H_{12}O_2$ 236.0837; found 236.0831.

4-(4-Methoxyphenyl)-6-methylcoumarin (46): 65% (17.3 mg) from **38** (33.0 mg, 0.10 mmol) (Table 3, Entry 5). Colorless prisms; m.p. 128–130 °C (hexane/ AcOEt, ref.[32] 132–134 °C). IR (film): $\tilde{\nu}$ = 1178, 1248, 1512, 1607, 1724 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.35 (s, 3 H), 3.90 (s, 3 H), 6.32 (s, 1 H), 7.05 (d, *J* = 8.6 Hz, 2 H), 7.26–7.36 (m, 3 H), 7.41 (d, *J* = 8.6 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 20.9, 55.4, 114.3, 114.7, 117.1, 118.9, 126.7, 127.6, 129.9, 132.8, 133.7, 152.4, 155.3, 160.8, 161.1 ppm. MS (EI): *m*/*z* (%) = 266 (100) [M$^+$], 238 (50), 223 (21), 195 (5), 160 (15), 132 (6). HRMS: calcd. for $C_{17}H_{14}O_3$ 266.0943; found 266.0948.

7-Methoxy-4-phenylcoumarin (47): 52% (13.1 mg) from **39** (34.6 mg, 0.10 mmol) (Table 3, Entry 6). Colorless prisms; m.p. 107–109 °C (hexane/AcOEt, ref.[33] 110–111 °C). IR (film): $\tilde{\nu}$ = 1150, 1281, 1508, 1609, 1726 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.88 (s, 3 H), 6.21 (s, 1 H), 6.79 (dd, *J* = 8.8, 2.6 Hz, 1 H), 6.89 (d, *J* = 2.6 Hz, 1 H), 7.38 (d, *J* = 8.8 Hz, 1 H), 7.42–7.44 (m, 2 H), 7.49–7.52 (m, 3 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 55.8, 101.1, 111.8, 112.3, 112.5, 127.9, 128.3, 128.7, 129.5, 135.5, 155.7, 155.9, 161.1, 162.7 ppm. MS (EI): *m*/*z* (%) = 252 (100) [M$^+$], 224 (49), 209 (22). HRMS: calcd. for $C_{16}H_{12}O_3$ 252.0786; found 252.0820.

7-Methoxy-4-(4-methoxyphenyl)coumarin (48): 76% (21.4 mg) from **39** (34.6 mg, 0.10 mmol) (Table 3, Entry 7). Colorless needles; m.p. 159–160 °C (hexane/AcOEt). IR (film): $\tilde{\nu}$ = 1150, 1248, 1512, 1611, 1726 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.885 (s, 3 H), 3.887 (s, 3 H), 6.19 (s, 1 H), 6.80 (dd, *J* = 9.0, 2.6 Hz, 1 H), 6.89 (d, *J* = 2.6 Hz, 1 H), 7.03 (d, *J* = 8.6 Hz, 2 H), 7.40 (d, *J* = 8.6 Hz, 2 H), 7.45 (d, *J* = 9.0 Hz, 1 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 55.4, 55.8, 101.1, 111.4, 112.2, 112.7, 114.3, 128.0, 128.4, 129.8, 130.3, 155.5, 156.1, 161.3, 162.7 ppm. MS (EI): *m*/*z* (%) = 282 (100) [M$^+$], 254 (80), 239 (30), 211 (8). HRMS: calcd. for $C_{17}H_{14}O_4$ 282.0892; found 282.0887.

6-Methyl-4-phenyl-2-pyranone (49): 84% (15.6 mg) from **40** (28.0 mg, 0.10 mmol) (Table 3, Entry 8). Colorless plates; m.p. 89–90 °C (hexane/AcOEt, ref.[34] 89–91 °C). IR (film): $\tilde{\nu}$ = 1140, 1547, 1636, 1711 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.32 (s, 3 H), 6.31 (s, 1 H), 6.35 (s, 1 H), 7.47–7.48 (m, 3 H), 7.56–7.57 (m, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 20.1, 103.4, 108.1, 126.6, 129.1, 130.5, 135.8, 155.5, 162.1, 163.4 ppm. MS (EI): *m*/*z* (%) = 186 (78) [M$^+$], 158 (100), 129 (20), 115 (19). HRMS: calcd. for $C_{12}H_{10}O_2$ 186.0681; found 186.0684.

4-(4-Methoxyphenyl)-6-methyl-2-pyranone (50): 87% (18.8 mg) from **40** (28.0 mg, 0.10 mmol) (Table 3, Entry 9). Colorless needles; m.p. 109–111 °C (hexane/AcOEt, ref.[35] 114–115 °C). IR (film): $\tilde{\nu}$ = 1186, 1254, 1545, 1607, 1638, 1705 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.31 (s, 3 H), 3.86 (s, 3 H), 6.29 (s, 1 H), 6.30 (s, 1 H), 6.98 (d, *J* = 9.0 Hz, 2 H), 7.54 (d, *J* = 9.0 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 20.1, 55.4, 103.1, 106.4, 114.6, 127.8, 128.1, 154.7, 161.7, 161.8, 163.6 ppm. MS (EI): *m*/*z* (%) = 216 (95) [M$^+$], 188 (100), 173 (31), 145 (14). HRMS: calcd. for $C_{13}H_{12}O_3$ 216.0786; found 216.0748.

4-(4-Cyanophenyl)-6-methyl-2-pyranone (51): 86% (18.1 mg) from **40** (28.0 mg, 0.10 mmol) (Table 3, Entry 10). Colorless plates; m.p. 182–184 °C (hexane/AcOEt). IR (film): $\tilde{\nu}$ = 1545, 1638, 1719, 2228 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.35 (s, 3 H), 6.26 (s, 1 H), 6.36 (s, 1 H), 7.67 (d, *J* = 8.4 Hz, 2 H), 7.78 (d, *J* = 8.4 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 20.2, 102.8, 109.7, 114.2, 117.9, 127.3, 132.9, 140.3, 153.4, 162.5, 163.1 ppm. MS (EI): *m*/*z* (%) = 211 (55) [M$^+$], 183 (100). HRMS: calcd. for $C_{13}H_9NO_2$ 211.0633; found 211.0620.

1-Methyl-4-phenyl-2-quinolone (52): 87% (20.4 mg) from **41** (32.9 mg, 0.10 mmol) (Table 3, Entry 11). Colorless plates; m.p. 147–148 °C (hexane/AcOEt, ref.[13d] 146–148 °C). IR (film): $\tilde{\nu}$ = 1379, 1452, 1587, 1647, 1653 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.78 (s, 3 H), 6.69 (s, 1 H), 7.17 (t, *J* = 7.6 Hz, 1 H), 7.41–7.60 (m, 8 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 29.5, 114.4, 120.4, 121.1, 121.9, 127.6, 128.5, 128.6, 128.8, 130.6, 136.9, 140.1, 150.8, 161.8 ppm. MS (EI): *m*/*z* (%) = 235 (100) [M$^+$], 207 (14). HRMS: calcd. for $C_{16}H_{13}NO_2$ 235.0997; found 235.0998.

4-(4-Methoxyphenyl)-1-methyl-2-quinolone (53): 77% (20.4 mg) from **41** (32.9 mg, 0.10 mmol) (Table 3, Entry 12). Colorless needles; m.p. 116–117 °C (hexane/AcOEt). IR (film): $\tilde{\nu}$ = 1178, 1250, 1379, 1454, 1512, 1587, 1587, 1607, 1645, 1657, 3368 cm^{-1}. ^{1}H

NMR (400 MHz, $CDCl_3$): δ = 3.78 (s, 3 H), 3.89 (s, 3 H), 6.67 (s, 1 H), 7.02 (d, J = 8.8 Hz, 2 H), 7.18 (dd, J = 8.0, 7.6 Hz, 1 H), 7.37 (d, J = 8.8 Hz, 2 H), 7.43 (d, J = 8.4 Hz, 1 H), 7.56–7.63 (m, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 29.4, 55.4, 114.0, 114.4, 120.7, 121.0, 121.8, 127.7, 129.4, 130.2, 130.6, 140.4, 150.7, 160.0, 162.1 ppm. MS (EI): *m/z* (%) = 265 (100) [M^+], 222 (16). HRMS: calcd. for $C_{17}H_{15}NO_2$ 265.1103; found 265.1091.

4-(4-Cyanophenyl)-1-methyl-2-quinolone (54): 86% (22.4 mg) from **41** (32.9 mg, 0.10 mmol) (Table 3, Entry 13). Colorless needles; m.p. 185–186 °C (hexane/AcOEt). IR (film): $\tilde{\nu}$ = 1379, 1454, 1587, 1605, 1659, 2230 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$): δ = 3.79 (s, 3 H), 6.66 (s, 1 H), 7.20 (dd, J = 7.8, 7.4 Hz, 1 H), 7.39 (d, J = 7.8 Hz, 1 H), 7.47 (d, J = 8.7 Hz, 1 H), 7.55 (d, J = 8.0 Hz, 2 H), 7.62 (dd, J = 8.7, 7.4 Hz, 1 H), 7.81 (d, J = 8.0 Hz, 2 H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 29.5, 112.7, 114.7, 118.2, 119.5, 121.6, 122.2, 127.0, 129.7, 131.1, 132.4, 140.3, 141.7, 148.9, 161.4 ppm. MS (EI): *m/z* (%) = 260 (100) [M^+], 232 (47), 190 (23). HRMS: calcd. for $C_{17}H_{12}N_2O$ 260.0950; found 260.0951.

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