Supporting Information

Efficient Cross-Coupling of Aryl Chlorides with Arylzinc Reagents Catalyzed by Amido Pincer Complexes of Nickel

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Experimental procedures and spectral data

General

All reactions were performed under nitrogen atmosphere using standard Schlenk and vacuum line techniques. THF and Et_2O were distilled under nitrogen over sodium/benzophenone and degassed prior to use. NMP was dried over 4A molecular sieve, fractionally distilled under reduced pressure and stored under nitrogen atmosphere. $LiBu^n$ and $CDCl_3$ was purchased from Acros Organics and used as received. 2-Furyllithium and 2,6-dimethoxyphenyllithium were prepared from furan or 1,3-dimethoxybenzene by direct lithiation using $LiBu^n$.[1,2] PhLi, *p*-MeC_6H_4Li, *o*-MeC_6H_4Li and *p*-$Me_2NC_6H_4Li$ were prepared from corresponding aryl bromides and Li according to literature.[3] NMR spectra were recorded on a Bruker av300 spectrometer at ambient temperature. The chemical shifts of the ^1H and ^{13}C NMR spectra were referenced to internal solvent resonances or TMS. HRMS was determined on a Agilent6890/Micromass GCT-MS spectrometer.

Reaction of PhZnCl with *p*-$Me_2NC_6H_4Cl$ catalyzed by complexes 1a–4

1. Preparation of a solution of PhZnCl in THF

Phenyllithium prepared from PhBr (2 g, 12.8 mmol) and Li (0.2 g, 28.5 mmol) in Et_2O (10 mL) was added dropwise to a stirred solution of $ZnCl_2$ (1.73 g, 12.9 mmol) in THF (15 mL) at 0 °C. The mixture was warmed to room temperature and stirred for 1 h. Solvents were removed under reduced pressure and the residue was dissolved in 25.6 mL of THF to afford a THF solution of PhZnCl (0.5 M).

2. Reaction of PhZnCl with *p*-$Me_2NC_6H_4Cl$ catalyzed by complex **1b** (entry 2, Table 1)

A Schlenk tube was charged *p*-$Me_2NC_6H_4Cl$ (0.0778 g, 0.5 mmol), NMP (1.5 mL) and complex **1b** (0.0029 g, 0.005 mmol). To the stirred mixture PhZnCl solution (1.5 mL, 0.5 M

solution in THF, 0.75 mmol) was added by syringe. Then the reaction mixture was stirred in a 70 °C oil bath for 30 h. The solution was cooled to room temperature, and water (10 mL) and several drops of hydrochloric acid were successively added. The resulting mixture was extracted with Et_2O (10 mL × 3), and the extracts were combined, washed with water (10 mL × 2), dried ($MgSO_4$), and concentrated. The residue was charged to column chromatography (silica gel), eluted with a mixture of CH_3COOEt and petroleum ether (1:20), and collected the composition including reaction product and unreacted starting materials. Solvents were removed by rotary evaporation and the residue was determined by ^1H NMR spectrum. The conversion of *p*-$Me_2NC_6H_4Cl$ was calculated based on the integration ratio of the NMR signals of NMe_2 groups of the unreacted *p*-$Me_2NC_6H_4Cl$ and the coupling product in the mixture.

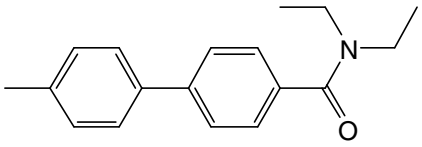
Reaction of PhZnCl with *p*-$Me_2NC_6H_4Cl$ catalyzed by each of complexes **1a** and **2a**–**4** followed the same procedure.

Reaction of PhZnCl with *p*-$Et_2NC(O)C_6H_4Cl$ catalyzed by complex 4 (entry 9, Table 2)

A Schlenk tube was charged *p*-$Et_2NC(O)C_6H_4Cl$ (0.106 g, 0.5 mmol), NMP (1.5 mL) and a solution of complex **4** in THF (0.085 mL, prepared by dissolving 0.0091 g of **4** in 20 mL of THF). To the stirred mixture PhZnCl solution (1.5 mL, 0.5 M solution in THF, 0.75 mmol) was added by syringe. Then the reaction mixture was stirred in a 70 °C oil bath for 10 h. The solution was cooled to room temperature, and water (10 mL) and several drops of hydrochloric acid were successively added. The resulting mixture was extracted with Et_2O (10 mL × 3), and the extracts were combined, washed with water (10 mL × 2), dried ($MgSO_4$), and concentrated. The residue was purified by column chromatography (silica gel, eluted using 10 % CH_3COOEt in petroleum ether) to afford colorless oil, which solidified after several days (0.1256 g, 99 %).

Reaction of other arylzinc reagents with aryl chlorides catalyzed by **3b** or **4** followed the same procedure. Colors, melting points (for solid products), and spectral data of the compounds obtained was listed as follows:

1. *N*,*N*-Diethyl-4'-methylbiphenyl-4-carboxamide



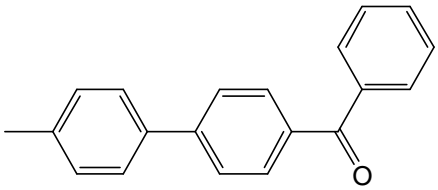
White solid, m.p. 68–70 °C.

^{1}H NMR ($CDCl_3$): δ 1.05 (b, 3H, CH_3), 1.12 (b, 3H, CH_3), 2.28 (s, 3H, Me), 3.21 (b, 2H, CH_2), 3.44 (b, 2H, CH_2), 7.14 (d, *J* = 7.8 Hz, 2H, C_6H_4), 7.32 (d, *J* = 8.1 Hz, 2H, C_6H_4), 7.37 (d, *J* = 7.8 Hz, 2H, C_6H_4), 7.48 (d, *J* = 8.1 Hz, 2H, C_6H_4).

^{13}C NMR ($CDCl_3$): δ 12.95, 14.24, 21.05, 39.41, 43.49, 126.80, 126.87, 129.53, 135.51, 137.37, 137.46, 141.94, 171.30.

HRMS: calcd. for $C_{18}H_{21}NO$ (M^+): 267.1623; found: 267.1615.

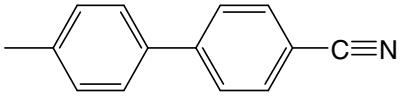
2. (4'-Methylbiphenyl-4-yl)(phenyl)methanone



White solid. m.p. 129–130 °C (lit.[4] 129–130 °C).

^{1}H NMR ($CDCl_3$): δ 2.34 (s, 3H, CH_3), 7.22 (d, *J* = 7.8 Hz, 2H, Ar), 7.40–7.55 (m, 5H, Ar), 7.61 (d, *J* = 8.4 Hz, 2H, Ar), 7.76 (d, *J* = 7.2 Hz, 2H, Ar), 7.81 (d, *J* = 8.4 Hz, 2H, Ar).

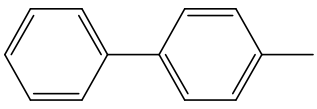
3. 4'-Methylbiphenyl-4-carbonitrile



White solid. m.p. 110–111 °C (lit.[5] 110–111 °C).

^{1}H NMR ($CDCl_3$): δ 2.42 (s, 3H, CH_3), 7.29 (d, *J* = 7.2 Hz, 2H, C_6H_4), 7.49 (d, *J* = 7.2 Hz, 2H, C_6H_4), 7.65–7.72 (m, 4H, C_6H_4).

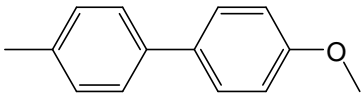
4. 4-Methylbiphenyl



White solid. m.p. 48–49 °C (lit.[6] 49–50 °C).

^{1}H NMR ($CDCl_3$): δ 2.54 (s, 3H, CH3), 7.37 (d, *J* = 7.5 Hz, 2H, Ar), 7.44 (t, *J* = 7.5 Hz, 1H, Ar), 7.55 (t, *J* = 7.5 Hz, 2H, Ar), 7.62 (d, *J* = 8.1 Hz, 2H, Ar), 7.70 (d, *J* = 8.1 Hz, 2H, Ar).

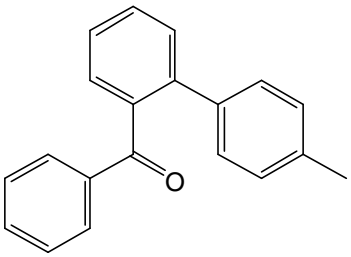
5. 4-Methoxy-4'-methylbiphenyl



White solid. m.p. 111−112 °C (lit.[7] 109 °C).

^{1}H NMR ($CDCl_3$): δ 2.39 (s, 3H, CH_3), 3.85 (s, 3H, OCH_3), 6.97 (d, *J* = 8.7 Hz, 2H, C_6H_4), 7.23 (d, *J* = 8.1 Hz, 2H, C_6H_4), 7.45 (d, *J* = 8.1 Hz, 2H, C_6H_4), 7.52 (d, *J* = 8.7 Hz, 2H, C_6H_4).

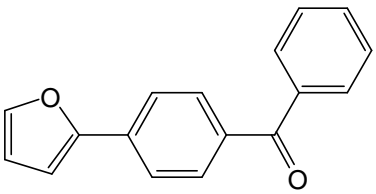
6. (4'-Methylbiphenyl-2-yl)(phenyl)methanone



White solid. m.p. 79–80 °C (lit.[8] 79–80 °C).

^{1}H NMR ($CDCl_3$): δ 2.17 (s, 3H, CH_3), 6.93 (d, *J* = 8.1 Hz, 2H, Ar), 7.08 (d, *J* = 8.1 Hz, 2H, Ar), 7.21 (t, *J* = 7.8 Hz, 2H, Ar), 7.33–7.41 (m, 4H, Ar), 7.46–7.51 (m, 1H, Ar), 7.59 (d, *J* = 7.5 Hz, 2H, Ar).

7. (4-(Furan-2-yl)phenyl)(phenyl)methanone



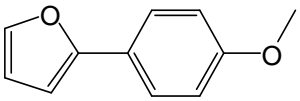
Yellowish solid. m.p. 80–82 °C.

^{1}H NMR ($CDCl_3$): δ 6.51 (dd, *J* = 1.5, 3.3 Hz, 1H, furyl), 6.81(d, *J* = 3 Hz, 1H, furyl), 7.49 (t, *J* = 7.2 Hz, 2H, Ar), 7.53 (d, *J* = 1.5 Hz, 1H, furyl), 7.56–7.61 (m, 1H, Ar), 7.75–7.87 (m, 6H, Ar).

^{13}C NMR ($CDCl_3$): δ 107.48, 112.18, 123.42, 128.39, 130.00, 130.90, 132.39, 134.52, 135.98, 137.88, 143.34, 152.98, 196.04.

HRMS: calcd. for $C_{17}H_{12}O_2$ (M^+): 248.0837; found: 248.0839.

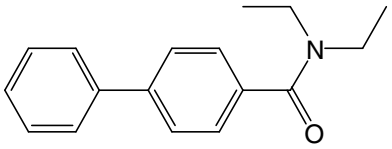
8. 2-(4-Methoxyphenyl)furan



White solid. m.p. 54–55 °C (lit.[9] 54–55 °C).

^{1}H NMR ($CDCl_3$): δ 3.83 (s, 3H, CH_3), 6.45 (dd, *J* = 1.8, 3.3 Hz, 1H, furyl), 6.52 (d, *J* = 3.3 Hz, 1H, furyl), 6.93 (d, *J* = 8.8 Hz, 2H, C_6H_4), 7.43 (b, 1H, furyl), 7.61 (d, *J* = 8.8 Hz, 2H, C_6H_4).

9. *N*,*N*-Diethylbiphenyl-4-carboxamide



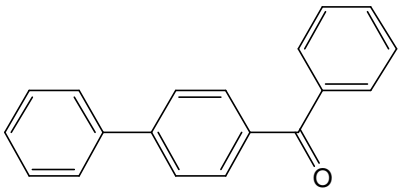
White solid. m.p. 58–60 °C.

^{1}H NMR ($CDCl_3$): δ 1.17–1.28 (m, 6H, CH_3), 3.33 (b, 2H, CH_2), 3.56 (b, 2H, CH_2), 7.34–7.39 (m, 1H, Ar), 7.43–7.48 (m, 4H, Ar), 7.58–7.63 (m, 4H, Ar).

^{13}C NMR ($CDCl_3$): δ 13.01, 14.22, 39.42, 43.48, 126.88, 127.10, 127.67, 128.85, 135.97, 140.33, 142.03, 171.20.

HRMS: calcd. for $C_{17}H_{19}NO$ (M^+): 253.1467; found: 253.1460.

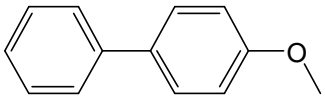
10. Biphenyl-4-yl(phenyl)methanone



White solid. m.p. 105–106 °C (lit.[10] 106 °C).

^{1}H NMR ($CDCl_3$): δ 7.42 (d, *J* = 10.2 Hz, 1H, Ar), 7.46–7.53 (m, 4H, Ar), 7.60 (d, *J* = 7.2 Hz, 1H, Ar), 7.66 (d, *J* = 7.2 Hz, 2H, Ar), 7.71 (d, *J* = 8.4 Hz, 2H, Ar), 7.83–7.86 (m, 2H, Ar), 7.90 (d, *J* = 8.1 Hz, 2H, Ar).

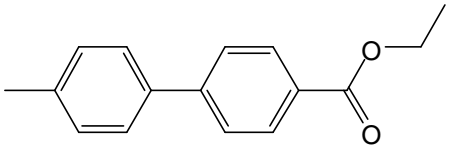
11. 4-Methoxybiphenyl



White solid. m.p. 91–92 °C (lit.[11] 91.1-92.3°C).

^{1}H NMR ($CDCl_3$): δ 3.86 (s, 3H, CH_3), 6.98 (d, *J* = 8.7 Hz, 2H, Ar), 7.31 (t, *J* = 7.2 Hz, 1H, Ar), 7.42 (t, *J* = 7.2 Hz, 2H, Ar), 7.52–7.57 (m, 4H, Ar).

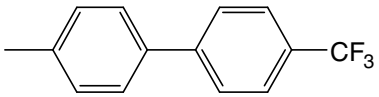
12. Ethyl 4'-methylbiphenyl-4-carboxylate



White solid. m.p. 76–77 °C (lit.[12] 80–80.5 °C).

^{1}H NMR ($CDCl_3$): δ 1.34 (t, *J* = 7.1 Hz, 3H, CH_3), 2.33 (s, 3H, CH_3), 4.32 (q, *J* = 7.1 Hz, 2H, CH_2), 7.19 (d, *J* = 8.6 Hz, 2H, Ar), 7.45 (d, *J* = 8.1 Hz, 2H, Ar), 7.56 (d, 2H, *J* = 8.5 Hz, Ar), 8.02 (d, 2H, *J* = 8.5 Hz, Ar).

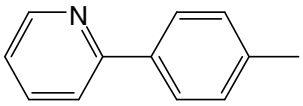
13. 4-Methyl-4'-(trifluoromethyl)biphenyl



White solid. m.p. 121 °C (lit.[13] 121 °C).

^{1}H NMR ($CDCl_3$): δ 2.42 (s, 3H, CH_3), 7.29 (d, *J* = 7.8 Hz, 2H, C_6H_4), 7.50 (d, *J* = 7.8 Hz, 2H, C_6H_4), 7.68 (s, 4H, C_6H_4).

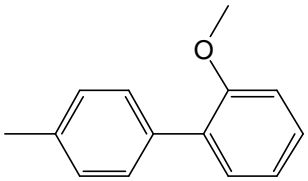
14. 2-*p*-Tolylpyridine[14]



Colorless oil.

^{1}H NMR ($CDCl_3$): δ 2.41 (s, 3H, CH_3), 7.17–7.22 (m, 1H, pyridyl), 7.28 (d, *J* = 8 Hz, 2H, Ar), 7.69–7.76 (m, 2H, pyridyl), 7.89 (d, *J* = 8.1 Hz, 2H, Ar), 8.67 (d, 1H, *J* = 4.6 Hz, pyridyl).

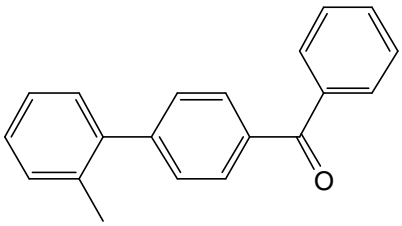
15. 2-Methoxy-4'-methylbiphenyl



White solid. m.p. 79–80 °C (lit.[15] 79–80 °C).

^{1}H NMR ($CDCl_3$): δ 2.37 (s, 3H, CH_3), 3.78 (s, 3H, CH_3), 6.94–7.02 (m, 2H, Ar), 7.20 (d, *J* = 7.8 Hz, 2H, Ar), 7.29 (d, *J* = 7.1 Hz, 2H, Ar), 7.41 (d, *J* = 8 Hz, 2H, Ar).

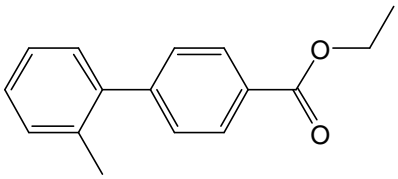
16. (2'-Methylbiphenyl-4-yl)(phenyl)methanone



White solid. m.p. 102–104 °C (lit.[16]).

^{1}H NMR ($CDCl_3$): δ 2.23 (s, 3H, CH_3), 7.17–7.22 (m, 4H, Ar), 7.37 (d, *J* = 8.4 Hz, 2H, Ar), 7.43 (t, *J* = 7.5 Hz, 2H, Ar), 7.53 (t, *J* = 7.5 Hz, 1H, Ar), 7.77–7.81 (m, 4H, Ar).

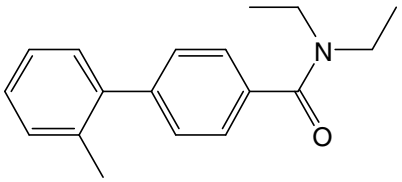
17. Ethyl 2'-methylbiphenyl-4-carboxylate[17]



Colorless oil.

^{1}H NMR ($CDCl_3$): δ 1.34 (t, *J* = 7.2 Hz, 3H, CH_3), 2.19 (s, 3H, CH_3), 4.34 (q, *J* = 7.2 Hz, 2H, CH_2), 7.13–7.21 (m, 4H, C_6H_4), 7.33 (d, *J* = 8.4 Hz, 2H, C_6H_4), 8.02 (d, *J* = 8.4 Hz, 2H, C_6H_4).

18. *N*,*N*-Diethyl-2'-methylbiphenyl-4-carboxamide



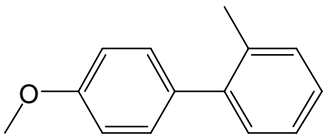
White solid. m.p. 82–84 °C.

^{1}H NMR ($CDCl_3$): δ 1.15 (b, 6H, CH_3), 2.20 (s, 3H, CH_3), 3.28 (b, 2H, CH_2), 3.50 (b, 2H, CH_2), 7.15–7.20 (m, 4H, C_6H_4), 7.27 (d, *J* = 7.8 Hz, 2H, C_6H_4), 7.36 (d, *J* = 7.8 Hz, 2H, C_6H_4).

^{13}C NMR ($CDCl_3$): δ 13.06, 14.28, 20.46, 39.35, 43.41, 125.86, 126.23, 127.57, 129.23, 129.71, 130.45, 135.30, 135.67, 141.16, 142.91, 171.29.

HRMS: calcd. for $C_{18}H_{21}NO$ (M^+): 267.1623; found: 267.1620.

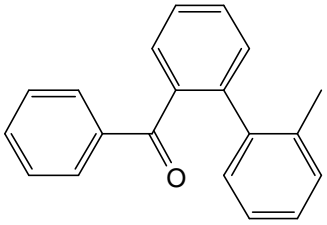
19. 4'-Methoxy-2-methylbiphenyl[18]



Colorless oil.

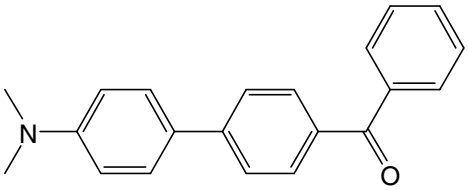
^{1}H NMR ($CDCl_3$): δ 2.27 (s, 3H, CH_3), 3.85 (s, 3H, CH_3), 6.95 (d, J = 8.4 Hz, 2H, C_6H_4), 7.21–7.26 (m, 6H, C_6H_4).

20. (2'-Methylbiphenyl-2-yl)(phenyl)methanone[19]



Colorless oil. ^{1}H NMR ($CDCl_3$): δ 2.14 (s, 3H, CH_3), 7.00–7.08 (m, 4H, Ar), 7.28–7.35 (m, 3H, Ar), 7.40–7.48 (m, 2H, Ar), 7.53–7.61 (m, 4H, Ar).

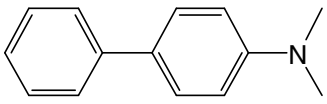
21. (4'-(Dimethylamino)biphenyl-4-yl)(phenyl)methanone



Yellow solid. m.p. 182–183 °C (lit.[20] 184.5 °C).

^{1}H NMR ($CDCl_3$): δ 3.03 (s, 6H, CH_3), 6.82 (d, J = 8.9 Hz, 2H, Ar), 7.49 (t, J = 7.7 Hz, 2H, Ar), 7.57–7.60 (m, 3H, Ar), 7.67 (d, 2H, J = 8.3 Hz, Ar), 7.81–7.88 (m, 4H, Ar).

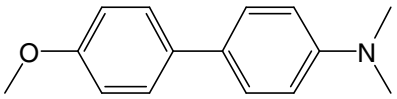
22. *N*,*N*-Dimethylbiphenyl-4-amine



White solid. m.p. 121–122 °C (lit.[21] 121–122 °C).

^{1}H NMR ($CDCl_3$): δ 2.92 (s, 6H, CH_3), 6.74 (d, J = 8.7 Hz, 2H, Ar), 7.15–7.20 (m, 1H, Ar), 7.32 (t, J = 7.5 Hz, 2H, Ar), 7.43 (d, J = 8.7 Hz, 2H, Ar), 7.49 (d, J = 7.5 Hz, 2H, Ar).

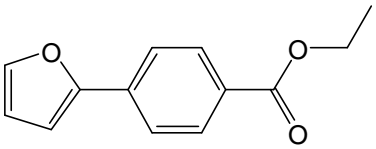
23. 4'-Methoxy-*N*,*N*-dimethylbiphenyl-4-amine



White solid. m.p. 151–152 °C (lit.[22] 150.5–152.3 °C).

^{1}H NMR ($CDCl_3$): δ 2.98 (s, 6H, CH_3), 3.84 (s, 3H, CH_3), 6.80 (d, *J* = 8.7 Hz, 2H, Ar), 6.94 (d, *J* = 8.7 Hz, 2H, Ar), 7.46 (t, *J* = 8.1 Hz, 4H, Ar).

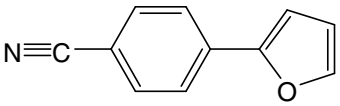
24. Ethyl 4-(furan-2-yl)benzoate[9]



Yellowish oil.

^{1}H NMR ($CDCl_3$): δ 1.40 (t, *J* = 7.2 Hz, 3H, CH_3), 4.38 (q, *J* = 7.2 Hz, 2H, CH_2), 6.48–6.52 (m, 1H, furyl), 6.78 (d, *J* = 3.3 Hz, 1H, furyl), 7.52 (s, 1H, furyl), 7.72 (d, *J* = 8.1 Hz, 2H, C_6H_4), 8.06 (d, *J* = 8.1 Hz, 2H, C_6H_4).

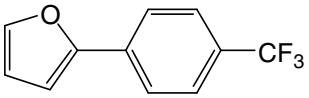
25. 4-(Furan-2-yl)benzonitrile



White solid. m.p. 65–66 °C (lit.[23] 65–66 °C).

^{1}H NMR ($CDCl_3$): δ 6.53 (dd, *J* = 1.8, 3.3 Hz, 1H, furyl), 6.81 (d, *J* = 3.3 Hz, 1H, furyl), 7.54 (d, *J* = 1.8 Hz, 1H, furyl), 7.65 (d, *J* = 8.4 Hz, 2H, Ar), 7.75 (d, *J* = 8.4 Hz, 2H, Ar).

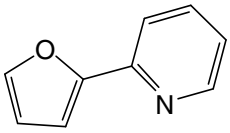
26. 2-(4-(Trifluoromethyl)phenyl)furan



White solid. m.p. 88–89 °C (lit.[9] 88–89 °C).

^{1}H NMR ($CDCl_3$): δ 6.51 (dd, *J* = 1.8, 3.3 Hz, 1H, furyl), 6.77 (d, *J* = 3.3 Hz, 1H, furyl), 7.52 (d, 1H, furyl), 7.63 (d, *J* = 8.4 Hz, 2H, C_6H_4), 7.76 (d, *J* = 8.4 Hz, 2H, C_6H_4).

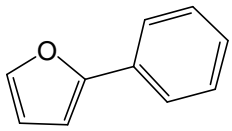
27. 2-(Furan-2-yl)pyridine[24]



Colorless oil.

^{1}H NMR ($CDCl_3$): δ 6.53 (dd, *J* = 1.5, 3.3 Hz, 1H, furyl), 7.06 (d, *J* = 3.3 Hz, 1H, furyl), 7.12–7.17 (m, 1H, pyridyl), 7.53 (s, 1H, furyl), 7.67–7.74 (m, 2H, pyridyl), 8.59 (d, *J* = 4.8 Hz, 1H, pyridyl).

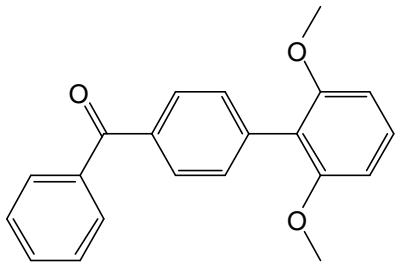
28. 2-Phenylfuran[25]



Colorless oil.

^{1}H NMR ($CDCl_3$): δ 6.50 (dd, *J* = 1.5, 3.3 Hz, 1H, furyl), 6.68 (d, *J* = 3.3 Hz, 1H, furyl), 7.28 (t, *J* = 7.5 Hz, 1H, Ar), 7.41 (t, *J* = 7.5 Hz, 2H, Ar), 7.50 (d, *J* = 1.5 Hz, 1H, furyl), 7.71 (d, *J* = 7.5 Hz, 2H, Ar).

29. (2',6'-Dimethoxybiphenyl-4-yl)(phenyl)methanone



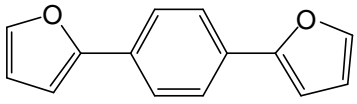
White solid. m.p. 142–144 °C.

^{1}H NMR ($CDCl_3$): δ 3.69 (s, 6H, OCH_3), 6.61 (d, *J* = 8.4 Hz, 2H, Ar), 7.25 (t, *J* = 8.4 Hz, 1H, Ar), 7.39–7.45 (m, 4H, Ar), 7.52 (t, *J* = 7.2 Hz, 1H, Ar), 7.78–7.81 (m, 4H, Ar).

^{13}C NMR ($CDCl_3$): δ 56.07, 104.42, 118.65, 128.35, 129.50, 129.76, 130.15, 131.19, 132.23, 135.76, 138.20, 139.31, 157.72, 196.71.

HRMS: calcd. for $C_{21}H_{18}O_3$ (M^+): 318.1256; found: 318.1251.

30. 1,4-Di(furan-2-yl)benzene

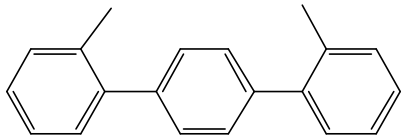


Pelter, Andrew; Rowlands, Martin; Clements, Gillian; SYNTBF; Synthesis; EN; 1; 1987; 51-53.

White solid. m.p. 148–150 °C (lit.[25] 148–150 °C).

^{1}H NMR ($CDCl_3$): δ 6.49 (dd, *J* = 1.8, 3.3 Hz, 2H, furyl), 6.68 (d, *J* = 3.3 Hz, 2H, furyl), 7.48 (d, *J* = 1.8 Hz, 2H, furyl), 7.69 (s, 4H, C_6H_4).

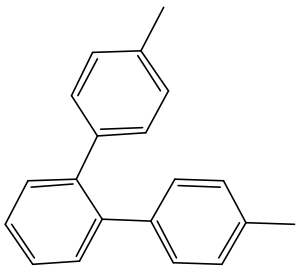
31. 1,4-Di(o-methylphenyl)benzene



White solid. m.p. 142–143 °C (lit.[26] 145.5–146 °C).

^{1}H NMR ($CDCl_3$): δ 2.27 (s, 6H, CH_3), 7.15–7.22 (m, 8H, C_6H_4), 7.28 (s, 4H, C_6H_4).

32. 1,2-Di(p-methylphenyl)benzene



White solid. m.p. 94–95 °C (lit.[27] 94–95 °C).

^{1}H NMR ($CDCl_3$): δ 2.32 (s, 6H, CH_3), 7.04 (s, 8H, C_6H_4), 7.40 (s, 4H, C_6H_4).

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