

One-pot Synthesis of 2-Substituted Benzo[*b*]furan via Pd/Tetrphosphine

Catalyzed Coupling of 2-Halophenols and Alkynes

Rong Zhou,^a Wei Wang,^a Zhi-jie Jiang,^a Kun Wang,^a Xue-li Zheng,^a Hai-yan Fu,^a Hua Chen^a and Rui-xiang Li^{a*}

^a Key lab of Green Chemistry and Technology, Ministry of Education; Sichuan University, Chengdu, China.

Fax: 86-28-85412904; E-mail: liruixiang@scu.edu.cn

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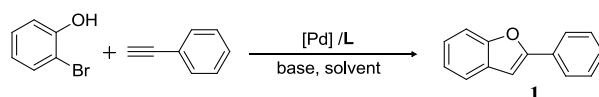
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NMR spectra of compounds

General Remarks: All chemicals were purchased from commercial suppliers. Except for some liquid reagents being sensitive to light and moisture (DMA, DMF, toluene, 1,4-dioxane, *o*-xylene, methanol and alcohol) was redistilled prior to use, there is no further treatment. ¹H NMR and ¹³C NMR spectra were recorded on a Bruker AV II-400 MHz. Mass Spectra were measured with Waters Q-TOF Premier mass spectrometer (USA). All products were isolated by short chromatography on a silica gel (300-400 mesh) using petroleum ether (60-90 °C), unless otherwise noted. **L** was prepared according to our previous work. Compounds described in the literature were characterized by ¹H NMR and ¹³C NMR spectroscopy and compared to the reported data.

General Procedure for the Synthesis of 2-Substituted Benzo[*b*]furan: Cs₂CO₃ (325 mg, 1 mmol), 2-halophenol (0.5 mmol), terminal alkynes (0.6 mmol) and degassed DMF (2 mL) were added successively into a dried Schlenk tube with a magnetic bar under nitrogen. Then DMF (0.05 mL) solution of tetraphosphine **L** (0.0005 mmol) and [Pd(η³-C₃H₅)Cl]₂ (0.00025 mmol) was added into the mixture. The reaction was performed at 130 °C. At the end of reaction, the solution was cooled to room temperature and water (3 mL) was added into it. The mixture solution was extracted with ethyl acetate (3×3 mL). The organic layer was dried over MgSO₄, filtered and purified with silica gel chromatography (petroleum ether) to give a corresponding product.

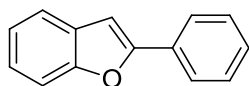
Table 1 Optimization of the reaction conditions for the synthesis of 2-bromophenol and phenylacetylene^a



Entry	Pd source	Base	Solvent	Yield (%)
1	[Pd(η ³ -C ₃ H ₅)Cl] ₂	Cs ₂ CO ₃	DMF	>99
2	Pd(OAc) ₂	Cs ₂ CO ₃	DMF	62
3	(PhCN) ₂ PdCl ₂	Cs ₂ CO ₃	DMF	58
4	Pd ₂ (dba) ₃	Cs ₂ CO ₃	DMF	70
5	Pd(COD)Cl ₂	Cs ₂ CO ₃	DMF	68
6	PdCl ₂	Cs ₂ CO ₃	DMF	71
7	[Pd(η ³ -C ₃ H ₅)Cl] ₂	Na ₂ CO ₃	DMF	55
8	[Pd(η ³ -C ₃ H ₅)Cl] ₂	K ₂ CO ₃	DMF	48
9	[Pd(η ³ -C ₃ H ₅)Cl] ₂	K ₃ PO ₄	DMF	57
10	[Pd(η ³ -C ₃ H ₅)Cl] ₂	KOH	DMF	41
11	[Pd(η ³ -C ₃ H ₅)Cl] ₂	KOtBu	DMF	28
12	[Pd(η ³ -C ₃ H ₅)Cl] ₂	LiOtBu	DMF	32
13	[Pd(η ³ -C ₃ H ₅)Cl] ₂	Et ₃ N	DMF	10
14	[Pd(η ³ -C ₃ H ₅)Cl] ₂	Cs ₂ CO ₃	DMA	50
15	[Pd(η ³ -C ₃ H ₅)Cl] ₂	Cs ₂ CO ₃	toluene	11 ^b
16	[Pd(η ³ -C ₃ H ₅)Cl] ₂	Cs ₂ CO ₃	1,4-dioxane	23 ^b
17	[Pd(η ³ -C ₃ H ₅)Cl] ₂	Cs ₂ CO ₃	<i>o</i> -xylene	33
18	[Pd(η ³ -C ₃ H ₅)Cl] ₂	Cs ₂ CO ₃	water	3 ^c
19	[Pd(η ³ -C ₃ H ₅)Cl] ₂	Cs ₂ CO ₃	DMF	75 ^d
20	[Pd(η ³ -C ₃ H ₅)Cl] ₂	Cs ₂ CO ₃	DMF	85 ^e
21	[Pd(η ³ -C ₃ H ₅)Cl] ₂	Cs ₂ CO ₃	DMF	33 ^f
22	[Pd(η ³ -C ₃ H ₅)Cl] ₂	Cs ₂ CO ₃	DMF	50 ^g
23	[Pd(η ³ -C ₃ H ₅)Cl] ₂	Cs ₂ CO ₃	DMF	69 ^h
24	[Pd(η ³ -C ₃ H ₅)Cl] ₂	Cs ₂ CO ₃	DMF	72 ⁱ

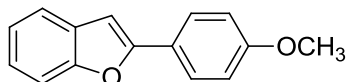
^aCondition: 2-bromophenol 0.5 mmol, phenylacetylene 0.6 mmol, solvent 2 mL, base 1 mmol, 130 °C, [Pd] 5×10⁻⁴ mmol, **L** 5×10⁻⁴ mmol, 9 h, GC yield; ^b110 °C; ^c100 °C; ^dno ligand, 24 h; ^ePPh₃, 24 h; ^fN,N,N',N'-tetra(diphenylphosphinomethyl)-1,2-ethylenediamine, 24 h; ^g2,2',6,6'-tetramethoxy-4,4'-bis(diphenylphosphino)-3,3'-bipyridine (P-Phos), 24 h; ^h1,2-bis(diphenylphosphino)ethane (dppe), 24 h; ⁱ1,4-bis(diphenylphosphino)butane (dppb), 24 h.

Characterization Data



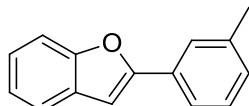
1¹H NMR (400 MHz, CDCl₃) δ: 7.87 (d, *J* = 7.3 Hz, 2H), 7.59 (d, *J* = 7.1 Hz, 1H), 7.53 (d, *J* = 7.7 Hz, 1H), 7.45 (t, *J* = 7.6 Hz, 2H), 7.36 (t, *J* = 7.1 Hz, 1H), 7.29 (t, *J* = 6.7 Hz, 1H), 7.22 (d, *J* = 6.5 Hz, 1H), 7.03 (s, 1H) ppm.

¹³C NMR (101 MHz, CDCl₃) δ 155.91, 154.89, 132.50, 130.48, 129.21, 128.78, 128.54, 128.44, 124.93, 124.25, 122.93, 120.90, 111.17, 101.30 ppm.



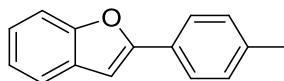
2²H NMR (400 MHz, CDCl₃) δ: 7.80 (d, *J* = 8.8 Hz, 2H), 7.55 (d, *J* = 7.2 Hz, 1H), 7.50 (d, *J* = 7.8 Hz, 1H), 7.24 – 7.18 (m, 2H), 6.98 (d, *J* = 8.8 Hz, 2H), 6.89 (s, 1H), 3.87 (s, 3H) ppm.

¹³C NMR (101 MHz, CDCl₃) δ: 159.99, 156.08, 154.70, 129.49, 126.43, 123.73, 123.36, 122.83, 120.57, 114.26, 110.99, 99.68, 55.38 ppm.



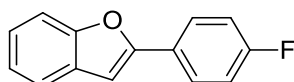
3³H NMR (400 MHz, CDCl₃) δ: 7.73 – 7.65 (m, 2H), 7.61 – 7.56 (m, 1H), 7.52 (d, *J* = 7.8 Hz, 1H), 7.34 (m, 1H), 7.31 – 7.26 (m, 1H), 7.25-7.21 (m, 1H), 7.17 (d, *J* = 7.6 Hz, 1H), 7.02 (d, *J* = 0.7 Hz, 1H), 2.43 (s, 3H) ppm.

¹³C NMR (101 MHz, CDCl₃) δ: 156.09, 154.83, 138.46, 130.37, 129.39, 129.29, 128.71, 125.53, 124.17, 122.89, 122.13, 120.86, 111.15, 101.19, 21.54 ppm.



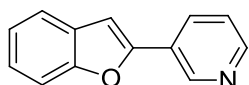
4²H NMR (400 MHz, CDCl₃) δ: 7.76 (d, *J* = 8.2 Hz, 2H), 7.57 (d, *J* = 7.3 Hz, 1H), 7.51 (d, *J* = 8.2 Hz, 1H), 7.42 (d, *J* = 8.1 Hz, 1H), 7.27 (s, 1H), 7.24 – 7.20 (m, 1H), 7.14 (d, *J* = 7.9 Hz, 1H), 6.97 (s, 1H), 2.40 (s, 3H) ppm.

¹³C NMR (101 MHz, CDCl₃) δ: 156.15, 154.72, 138.58, 132.37, 129.47, 129.20, 124.86, 123.97, 122.83, 120.72, 111.07, 100.53, 21.40 ppm.



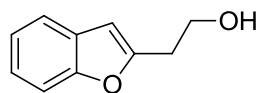
5⁴H NMR (400 MHz, CDCl₃) δ: 7.89 – 7.80 (m, 2H), 7.58 (dd, *J* = 7.6, 0.7 Hz, 1H), 7.51 (d, *J* = 8.2 Hz, 1H), 7.32 – 7.26 (m, 1H), 7.25 – 7.21 (m, 1H), 7.19 – 7.11 (m, 2H), 6.96 (s, 1H) ppm.

¹³C NMR (101 MHz, CDCl₃) δ: 164.09, 161.62, 154.99, 154.88, 129.16, 126.79, 126.71, 124.28, 123.01, 120.88, 116.00, 115.78, 111.13, 100.99 ppm.



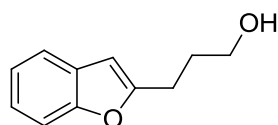
6⁶ ¹H NMR (400 MHz, CDCl₃) δ: 9.13 (s, 1H), 8.59 (d, *J* = 4.5 Hz, 1H), 8.18 (d, *J* = 8.0 Hz, 1H), 7.63 (d, *J* = 7.6 Hz, 1H), 7.55 (d, *J* = 8.1 Hz, 1H), 7.43 (dd, *J* = 7.9, 4.9 Hz, 1H), 7.34 (t, *J* = 7.2 Hz, 1H), 7.28 (d, *J* = 7.8 Hz, 1H), 7.15 (s, 1H) ppm.

¹³C NMR (101 MHz, CDCl₃) δ: 152.92, 149.29, 146.43, 131.88, 128.76, 126.64, 124.96, 123.62, 123.27, 121.22, 111.33, 102.75 ppm.



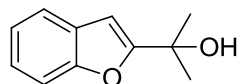
7⁷ ¹H NMR (400 MHz, CDCl₃) δ: 7.50 (d, *J* = 7.4 Hz, 1H), 7.43 (d, *J* = 7.7 Hz, 1H), 7.25 – 7.15 (m, 2H), 6.52 (s, 1H), 4.00 (t, *J* = 5.9 Hz, 2H), 3.05 (t, *J* = 6.0 Hz, 2H) ppm.

¹³C NMR (101 MHz, CDCl₃) δ: 155.94, 154.97, 128.81, 122.93, 122.36, 120.63, 110.86, 103.43, 61.00, 31.79 ppm.



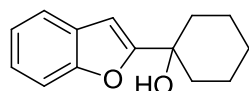
8⁸ ¹H NMR (400 MHz, CDCl₃) δ: 7.48 (d, *J* = 8.1 Hz, 1H), 7.41 (d, *J* = 7.7 Hz, 1H), 7.22 – 7.17 (m, 2H), 6.42 (s, 1H), 3.75 (t, *J* = 6.3 Hz, 2H), 2.89 (t, *J* = 7.4 Hz, 2H), 2.10 – 1.96 (m, 3H) ppm.

¹³C NMR (101 MHz, CDCl₃) δ: 160.13, 154.72, 128.93, 123.22, 122.48, 120.26, 110.75, 102.21, 61.90, 30.69, 24.84 ppm.



9⁹ ¹H NMR (400 MHz, CDCl₃) δ: 7.53 (d, *J* = 7.6 Hz, 1H), 7.46 (d, *J* = 8.0 Hz, 1H), 7.26 (t, *J* = 7.7 Hz, 1H), 7.20 (d, *J* = 7.2 Hz, 1H), 6.58 (s, 1H), 1.68 (s, 6H), 1.25 (s, 1H) ppm.

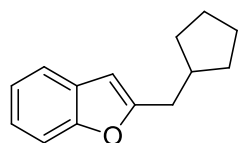
¹³C NMR (101 MHz, CDCl₃) δ: 162.99, 154.67, 128.30, 124.00, 122.72, 120.99, 111.18, 100.33, 69.33, 28.75 ppm.



10¹⁰ ¹H NMR (400 MHz, CDCl₃) δ: 7.54 (d, *J* = 7.4 Hz, 1H), 7.46 (d, *J* = 7.9 Hz, 1H), 7.28-7.19 (m, 2H), 6.61 (s, 1H), 2.08 (m, 2H), 1.99 – 1.90 (m, 3H), 1.85 – 1.71 (m, 3H), 1.58 (m, 4H) ppm.

¹³C NMR (101 MHz, CDCl₃) δ: 162.84, 154.48, 128.30, 123.94, 122.65, 120.96, 111.20, 101.12, 70.57, 36.45, 25.43, 22.00 ppm.

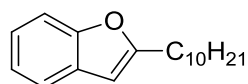
MS (ACPI): Calc'd for C₁₄H₁₆O₂, 216.1150; found, 216.02.



11¹¹ ¹H NMR (400 MHz, CDCl₃) δ 7.50 – 7.46 (m, 1H), 7.41 (d, *J* = 7.4 Hz, 1H), 7.18 (tt, *J* = 7.3, 5.7 Hz, 2H), 6.38 (s, 1H), 2.75 (d, *J* = 7.3 Hz, 2H), 2.33 – 2.26 (m, 1H), 1.82 (m, 2H), 1.69 – 1.61 (m, 3H), 1.56 (m, 3H) ppm.

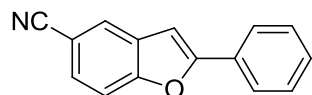
¹³C NMR (101 MHz, CDCl₃) δ: 159.41, 154.56, 122.96, 122.31, 120.13, 110.68, 102.18, 38.63, 34.54, 32.53, 25.07 ppm.

MS (ACPI): Calc'd for C₁₆H₁₅O, 220.1201; found, 220.03.



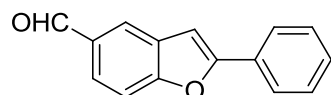
12¹⁰¹H NMR (400 MHz, CDCl₃) δ 7.47 (dd, *J* = 6.5, 2.1 Hz, 1H), 7.40 (d, *J* = 7.5 Hz, 1H), 7.22 – 7.14 (m, 2H), 6.37 (s, 1H), 2.75 (t, *J* = 7.6 Hz, 2H), 1.72 (q, *J* = 7.5, 2H), 1.33–1.21 (m, 14H), 0.88 (t, *J* = 7.0 Hz, 3H) ppm.

¹³C NMR (101 MHz, CDCl₃) δ: 158.77, 153.58, 128.00, 121.96, 121.31, 119.10, 109.66, 100.70, 30.88, 28.57, 28.52, 28.34, 28.30, 28.18, 27.43, 26.67, 21.66, 13.09 ppm.



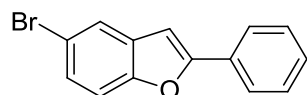
13² ¹H NMR (400 MHz, CDCl₃) δ: 7.92 (s, 1H), 7.88 (d, *J* = 7.4 Hz, 2H), 7.59 (q, *J* = 8.5 Hz, 2H), 7.49 (t, *J* = 7.4 Hz, 2H), 7.42 (t, *J* = 7.3 Hz, 1H), 7.07 (s, 1H) ppm.

¹³C NMR (101 MHz, CDCl₃) δ: 158.37, 156.47, 129.89, 129.61, 129.29, 129.00, 127.88, 125.76, 125.27, 119.50, 112.30, 106.89, 100.75 ppm.



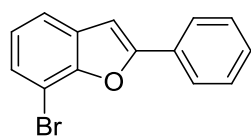
14¹¹ ¹H NMR (400 MHz, CDCl₃) δ: 10.08 (s, 1H), 8.14 (d, *J* = 1.0 Hz, 1H), 7.87 (dd, *J* = 13.5, 4.8 Hz, 3H), 7.65 (d, *J* = 8.5 Hz, 1H), 7.49 (t, *J* = 7.5 Hz, 2H), 7.42 (d, *J* = 7.3 Hz, 1H), 7.13 (s, 1H) ppm.

¹³C NMR (101 MHz, CDCl₃) δ: 191.77, 158.30, 157.96, 132.41, 129.86, 129.28, 128.94, 125.95, 125.19, 124.11, 101.52, 77.34, 77.02, 76.70 ppm.



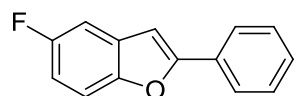
15² ¹H NMR (400 MHz, CDCl₃) δ: 7.89 (d, *J* = 7.4 Hz, 2H), 7.52 – 7.41 (m, 4H), 7.38 (t, *J* = 7.4 Hz, 1H), 7.10 (t, *J* = 7.8 Hz, 1H), 7.06 (s, 1H) ppm.

¹³C NMR (101 MHz, CDCl₃) δ: 155.60, 150.89, 129.42, 128.76, 127.97, 127.80, 126.18, 124.09, 123.17, 118.97, 102.89, 100.77, 76.31, 75.99, 75.67 ppm.



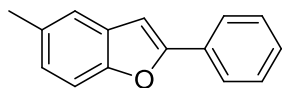
16¹²¹ ¹H NMR (400 MHz, CDCl₃) δ 7.85 (d, *J* = 7.4 Hz, 2H), 7.71 (s, 1H), 7.46 (t, *J* = 7.5 Hz, 2H), 7.39 (q, *J* = 8.4 Hz, 3H), 6.97 (s, 1H) ppm.

¹³C NMR (101 MHz, CDCl₃) δ: 157.28, 153.92, 131.19, 129.95, 129.02, 128.86, 127.07, 125.05, 123.46, 116.00, 112.61, 100.62 ppm.



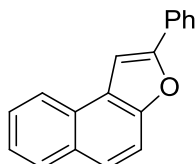
17¹³¹ ¹H NMR (400 MHz, CDCl₃) δ: 7.85 (d, *J* = 7.4 Hz, 2H), 7.45 (dd, *J* = 14.5, 6.8 Hz, 3H), 7.37 (t, *J* = 7.4 Hz, 1H), 7.23 (dd, *J* = 8.6, 2.6 Hz, 1H), 7.04 – 6.94 (m, 2H) ppm.

^{13}C NMR (101 MHz, CDCl_3) δ : 157.66, 151.08, 130.09, 128.89, 128.84, 124.98, 111.98, 111.77, 111.72, 111.68, 106.44, 106.19, 101.43, 101.39 ppm.



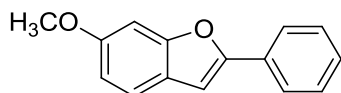
18² ^1H NMR (400 MHz, CDCl_3) δ : 7.85 (d, $J = 7.2$ Hz, 2H), 7.44 (t, $J = 6.9$ Hz, 2H), 7.40 (d, $J = 8.4$ Hz, 1H), 7.35 (dd, $J = 12.8, 5.3$ Hz, 2H), 7.09 (d, $J = 9.7$ Hz, 1H), 6.96 (s, 1H), 2.45 (s, 3H) ppm.

^{13}C NMR (101 MHz, CDCl_3) δ : 155.99, 153.35, 132.35, 130.64, 129.32, 128.76, 128.42, 125.54, 124.86, 120.73, 110.66, 101.09, 21.36 ppm.



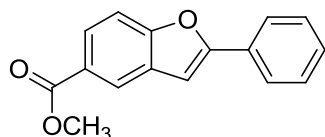
19⁸ ^1H NMR (400 MHz, CDCl_3) δ : 8.18 (d, $J = 8.1$ Hz, 1H), 7.95 (t, $J = 7.2$ Hz, 3H), 7.72 (q, $J = 9.0$ Hz, 2H), 7.60 (t, $J = 7.5$ Hz, 1H), 7.53 (s, 1H), 7.51 – 7.46 (m, 3H), 7.36 (m, 1H) ppm.

^{13}C NMR (101 MHz, CDCl_3) δ : 155.35, 152.34, 130.61, 130.39, 128.85, 128.80, 128.34, 128.27, 127.58, 126.26, 125.18, 124.64, 124.57, 124.52, 123.53, 123.45, 112.30, 100.44 ppm.



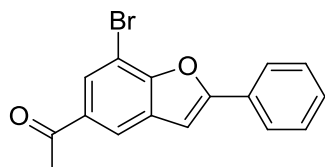
20¹⁵¹ ^1H NMR (400 MHz, CDCl_3) δ : 7.85 – 7.78 (m, 2H), 7.43 (t, $J = 8.3$ Hz, 3H), 7.32 (t, $J = 7.4$ Hz, 1H), 7.08 (d, $J = 2.0$ Hz, 1H), 6.95 (d, $J = 0.7$ Hz, 1H), 6.87 (dd, $J = 8.5, 2.3$ Hz, 1H), 3.88 (s, 3H) ppm.

^{13}C NMR (101 MHz, CDCl_3) δ : 158.04, 155.13, 130.69, 128.75, 128.04, 124.43, 122.53, 120.99, 111.96, 101.14, 95.85, 55.74 ppm.



21¹¹¹ ^1H NMR (400 MHz, CDCl_3) δ : 8.32 (d, $J = 1.5$ Hz, 1H), 8.02 (dd, $J = 8.6, 1.7$ Hz, 1H), 7.91 – 7.84 (m, 2H), 7.55 (d, $J = 8.6$ Hz, 1H), 7.47 (t, $J = 7.5$ Hz, 2H), 7.38 (t, $J = 7.4$ Hz, 1H), 7.08 (s, 1H), 3.95 (s, 3H) ppm.

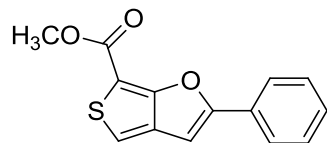
^{13}C NMR (101 MHz, CDCl_3) δ : 167.31, 157.37, 129.87, 129.24, 129.04, 128.88, 126.05, 125.30, 125.06, 123.31, 111.01, 101.52, 52.14 ppm.



22¹ ^1H NMR (400 MHz, CDCl_3) δ : 8.24 (d, $J = 1.7$ Hz, 1H), 7.96 (dd, $J = 8.6, 1.8$ Hz, 1H), 7.88 (d, $J = 7.3$ Hz, 2H), 7.56 (d, $J = 8.6$ Hz, 1H), 7.47 (t, $J = 7.5$ Hz, 2H), 7.39 (t, $J = 6.9$ Hz, 1H), 7.09 (s, 1H), 2.68 (s, 3H) ppm.

^{13}C NMR (101 MHz, CDCl_3) δ : 197.70, 157.56, 157.45, 132.89, 129.80, 129.35, 129.11, 128.91, 125.08, 122.17, 111.17, 101.63, 26.82 ppm.

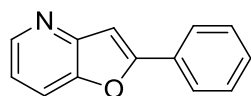
MS (ACPI): Calc'd for $\text{C}_{16}\text{H}_{11}\text{BrO}_2$, 315.1613; found, 315.00.



^{23}H NMR (400 MHz, CDCl_3) δ : 7.84 (d, $J = 7.3$ Hz, 2H), 7.45 (t, $J = 7.3$ Hz, 2H), 7.39 (t, $J = 7.3$ Hz, 1H), 7.18 (s, 1H), 6.72 (s, 1H), 3.96 (s, 3H) ppm.

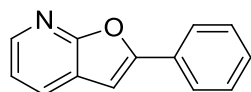
^{13}C NMR (101 MHz, CDCl_3) δ : 164.85, 161.69, 157.58, 136.99, 129.82, 129.37, 128.80, 125.07, 114.47, 97.58, 52.11 ppm.

MS (ACPI): Calc'd for $\text{C}_{14}\text{H}_{10}\text{O}_3\text{S}$, 258.2924; found, 258.17.



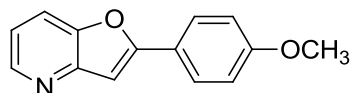
^{24}H NMR (400 MHz, CDCl_3) δ : 8.53 (d, $J = 4.6$ Hz, 1H), 7.92 (d, $J = 7.6$ Hz, 2H), 7.80 (d, $J = 8.2$ Hz, 1H), 7.49 (t, $J = 7.5$ Hz, 2H), 7.43 (t, $J = 7.2$ Hz, 1H), 7.23 (m, 2H) ppm.

^{13}C NMR (101 MHz, CDCl_3) δ : 159.69, 148.99, 148.04, 145.97, 129.69, 129.60, 128.94, 125.34, 118.80, 117.91, 117.88, 102.36 ppm.



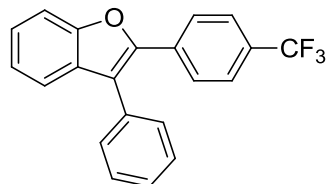
^{25}H NMR (400 MHz, CDCl_3) δ : 8.31 (dd, $J = 4.9, 1.6$ Hz, 1H), 7.98 – 7.87 (m, 3H), 7.48 (t, $J = 7.4$ Hz, 2H), 7.44 – 7.37 (m, 1H), 7.26 – 7.20 (m, 1H), 7.03 (s, 1H) ppm.

^{13}C NMR (101 MHz, CDCl_3) δ : 161.53, 155.80, 143.39, 129.87, 129.46, 129.40, 128.92, 125.21, 121.70, 119.57, 100.01 ppm.



^{26}H NMR (400 MHz, CDCl_3) δ : 8.49 (d, $J = 4.9$ Hz, 1H), 7.85 (t, $J = 9.4$ Hz, 3H), 7.24 (dd, $J = 8.2, 5.1$ Hz, 1H), 7.19 (s, 1H), 7.01 (d, $J = 8.8$ Hz, 2H), 3.88 (s, 3H) ppm.

^{13}C NMR (101 MHz, CDCl_3) δ : 161.18, 148.25, 148.06, 143.68, 127.23, 121.84, 118.80, 118.28, 114.50, 99.74, 55.46 ppm.



^{27}H NMR (600 MHz, CDCl_3) δ : 7.72 (d, $J = 7.9$ Hz, 2H), 7.65 – 7.60 (m, 4H), 7.58 (d, $J = 8.2$ Hz, 1H), 7.49 (d, $J = 7.5$ Hz, 1H), 7.36 (m, 4H), 7.27 (d, $J = 7.5$ Hz, 1H) ppm.

^{13}C NMR (151 MHz, CDCl_3) δ : 154.10, 151.29, 136.89, 130.16, 130.07, 129.59, 128.84, 128.63, 127.24, 125.94, 125.02, 123.24, 119.67, 116.11, 111.33 ppm.

MS (ACPI): Calc'd for $\text{C}_{14}\text{H}_{10}\text{O}_3\text{S}$, 338.3225; found, 338.26.

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