

## Supporting Information

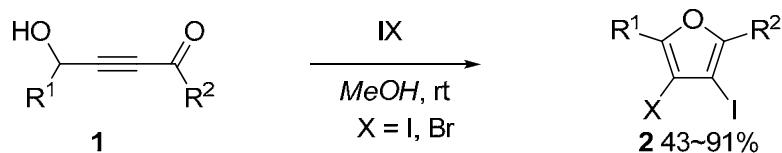
## Facile Synthesis of 3,4-Dihalofurans via Electrophilic Iodocyclization

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**General Information.**  $^1\text{H}$  NMR and  $^{13}\text{C}$  NMR spectra were recorded on JEOL JMTC-270/54/SS (JASTEC, 300 MHz, 400 MHz, 500 MHz) spectrometers.  $^1\text{H}$  NMR spectra are reported as follows: chemical shift in ppm ( $\delta$ ) relative to the chemical shift of  $\text{CDCl}_3$  at 7.26 ppm, integration, multiplicities (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet and br = broadened), and coupling constants (Hz).  $^{13}\text{C}$  NMR spectra reported in ppm ( $\delta$ ) relative to the central line of triplet for  $\text{CDCl}_3$  at 77 ppm. IR spectra were recorded on JASCO FT/IR-4100 spectrometer; absorptions are reported in  $\text{cm}^{-1}$ . Melting points were measured by MRK (No. 8026). High-resolution mass spectra were obtained on a BRUKER APEXIII spectrometer. Column chromatography was carried out employing Slica gel 60 N (spherical, neutral, 40~100  $\mu\text{m}$ , KANTO Chemical Co.). Analytical thin-layer chromatography (TLC) was performed on 0.2 mm precoated plate Kieselgel 60 F<sub>254</sub> (Merk).

**Materials.** Anhydrous  $\text{CH}_2\text{Cl}_2$ , THF,  $\text{CH}_3\text{Cl}$ , MeOH,  $\text{CH}_3\text{CN}$ , DMF, benzene (WAKO),  $\text{I}_2$  (TCI), IBr (Aldrich) were purchased and used as received. 4-hydroxy but-2-yn-1-ones **1** were prepared by according to the reported literatures.<sup>1,2</sup> The structures of products were determined by  $^1\text{H}$ ,  $^{13}\text{C}$  NMR, high-resolution mass (ESI), and **2b'** was confirmed by X-ray crystal analysis.

## References

1. Pridmore, J. S., Slatfors, P. A.; Jonathan.; Williams, M. J. *Tetrahedron Lett.* **2007**, *48*, 5111-5114.
2. Obrecht, D. *Helv. Chim. Acta* **1989**, *72*, 447-456.

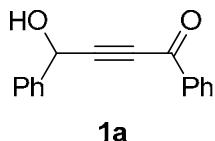
## General procedure

### Typical procedure for the formation of 3,4-diido-2,5-diphenylfuran (**2a**).

$\text{I}_2$  (0.6 mmol, 152 mg) was added to a solution of 4-hydroxy-1,4-diphenylbut-2-yn-1-one **1a** (0.2 mmol, 48 mg) in MeOH (2 mL), and the resulting mixture was stirred at room temperature for 5 h. The reaction mixture was diluted with 10 mL ether, washed with saturated sodium thiosulfate. The organic layer was dried with an anhydrous  $\text{MgSO}_4$ . After concentration of the filtrate, the residue was purified by chromatography on silica gel to afford 3,4-diido-2,5-diphenylfuran **2a** (82.2 mg, 87%) as a white solid.

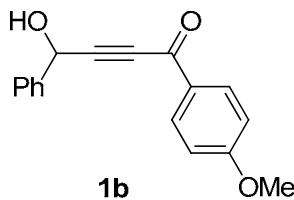
### Analytical data

#### 4-Hydroxy-1,4-diphenylbut-2-yn-1-one (1a)



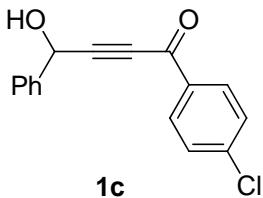
White solid;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.13-8.11 (m, 2H), 7.62-7.58 (m, 3H), 7.48-7.36 (m, 5H), 5.74 (d,  $J$  = 6.0 Hz, 1H), 3.42 (m, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  64.52, 83.61, 93.36, 126.68, 128.62, 128.85, 129.65 (x2), 134.39, 136.26, 138.79, 177.81; IR(neat): 1174 (C-O), 1260 (C-O), 1597 (C=C), 1641 (C=O), 2227 (C-C), 3380 (O-H)  $\text{cm}^{-1}$ ; HRMS (ESI positive):  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{16}\text{H}_{12}\text{O}_2\text{Na}$ , 259.0730; found, 259.0729.

#### 4-Hydroxy-1-(4-methoxyphenyl)-4-phenylbut-2-yn-1-one (1b)



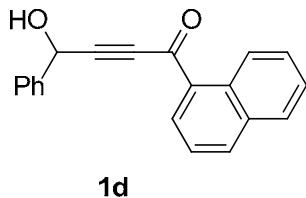
Yellow solid;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.99 (d, 2H,  $J$  = 9.2 Hz), 7.50 (m, 2H), 7.33-7.23 (m, 3H), 6.80 (d, 2H,  $J$  = 9.2 Hz), 5.64 (s, 1H), 4.15 (bs, 1H), 3.75 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  55.45, 64.25, 83.44, 93.05, 113.80, 126.63, 128.58, 128.67, 129.57, 132.07, 138.97, 164.57, 176.47; IR(neat): 847 (*p*-aromatic disubstituted), 1174 (C-O), 1259 (C-O), 1595 (C=C), 1623 (C=O), 2226 (C-C), 3321 (O-H)  $\text{cm}^{-1}$ ; HRMS (ESI positive):  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{17}\text{H}_{14}\text{O}_3\text{Na}$ , 289.0835; found, 289.0835.

#### 1-(4-Chlorophenyl)-4-hydroxy-4-phenylbut-2-yn-1-one (1c)



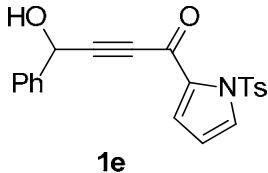
Yellow solid;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  8.08-8.03 (m, 2H), 7.60-7.56 (m, 2H), 7.47-7.36 (m, 5H), 5.74 (d, 2H,  $J$  = 6.3 Hz), 2.79 (d, 2H,  $J$  = 6.3 Hz);  $^{13}\text{C}$  NMR (75.45 MHz,  $\text{CDCl}_3$ )  $\delta$  64.68, 83.38, 93.43, 126.68, 128.98, 129.05, 129.08, 130.94, 134.74, 138.63, 141.08, 176.31; IR(neat): 1229 (C-O), 1583 (C=C), 1626 (C=O), 2228 (C-C), 3356 (O-H)  $\text{cm}^{-1}$ ; HRMS (ESI positive):  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{16}\text{H}_{11}\text{ClO}_2\text{Na}$ , 293.0340; found, 293.0339.

**4-Hydroxy-1-(naphthalen-1-yl)-4-phenylbut-2-yn-1-one (1d)**



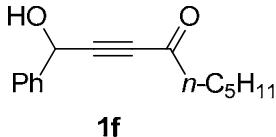
Yellow solid;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  9.22 (d, 1H,  $J = 8.1$  Hz), 8.56 (dd,  $J = 7.2, 1.5$  Hz, 1H), 7.91 (d,  $J = 8.1$  Hz, 1H), 7.79 (d,  $J = 7.2$  Hz, 1H), 7.72-7.69 (m, 2H), 7.64-7.58 (m, 1H), 7.34 (5H, m), 5.87 (s, 1H), 5.20 (bs, 1H);  $^{13}\text{C}$  NMR (75.45 MHz,  $\text{CDCl}_3$ )  $\delta$  64.07, 84.67, 92.59, 124.12, 125.40, 126.42, 126.55, 128.31, 128.41, 128.53, 128.55, 128.78, 130.17, 131.48, 133.29, 135.28, 138.81, 179.43; IR(neat): 1129 (C-O), 1231 (C-O), 1589 (C=C), 1637 (C=O), 2214 (C-C), 3352 (O-H)  $\text{cm}^{-1}$ ; HRMS (ESI positive):  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{20}\text{H}_{14}\text{O}_2\text{Na}$ , 309.0886; found, 309.0886.

**4-Hydroxy-4-phenyl-1-(1-tosyl-1*H*-pyrrol-2-yl)but-2-yn-1-one (1e)**



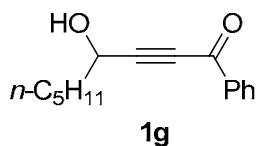
Brown solid;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.82-7.76 (m, 3H), 7.42-7.39 (m, 2H), 7.27-7.17 (m, 6H), 6.24-6.22 (m, 1H), 5.51 (s, 1H), 4.18 (bs, 1H), 2.28 (s, 3H);  $^{13}\text{C}$  NMR (75.45 MHz,  $\text{CDCl}_3$ )  $\delta$  21.37, 63.76, 82.91, 90.69, 111.01, 126.37, 128.29, 128.38, 128.47, 129.21, 129.61, 131.81, 132.70, 134.54, 138.70, 145.16, 163.68; IR(neat): 1142 (R-SO<sub>2</sub>-N), 1635 (C=O), 2228 (C-C), 3399 (O-H)  $\text{cm}^{-1}$ ; HRMS (ESI positive):  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{21}\text{H}_{17}\text{NO}_4\text{SNa}$ , 402.0770; found, 402.0771.

**1-Hydroxy-1-phenylnon-2-yn-4-one (1f)**



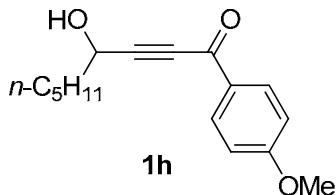
Yellow oil;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.52-7.44 (m, 2H), 7.41-7.33 (m, 3H), 5.56 (s, 1H), 3.67 (bs, 1H), 2.56 (t,  $J = 7.5$  Hz, 2H), 1.69-1.59 (m, 2H), 1.31-1.25 (m, 4H), 0.88 (t,  $J = 6.9$  Hz, 3H);  $^{13}\text{C}$  NMR (75.45 MHz,  $\text{CDCl}_3$ )  $\delta$  13.74, 22.23, 23.43, 30.92, 45.25, 64.09, 84.50, 90.93, 126.55, 126.58, 128.68, 138.78, 188.22; IR(neat): 1152 (C-O), 1240 (C-O), 1672 (C=O), 2212 (C-C), 3406 (O-H)  $\text{cm}^{-1}$ ; HRMS (ESI positive):  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{15}\text{H}_{18}\text{O}_2\text{Na}$ , 253.1199; found, 253.1198.

**4-Hydroxy-1-phenylnon-2-yn-1-one (1g)**



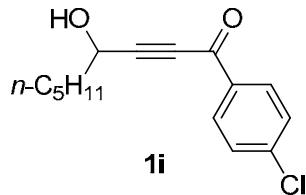
Colorless oil;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  8.13-8.10 (m, 2H), 7.63-7.58 (m, 1H), 7.49-7.44 (m, 2H), 4.70-4.64 (m, 1H), 2.93 (bs, 1H), 1.90-1.83 (m, 2H), 1.56-1.53 (2H, m), 1.35-1.32 (4H, m), 0.92-0.87 (t,  $J = 6.9$  Hz, 3H);  $^{13}\text{C}$  NMR (75.45 MHz,  $\text{CDCl}_3$ )  $\delta$  13.92, 22.46, 24.77, 31.31, 36.99, 62.38, 82.33, 95.29, 128.58, 129.60, 134.28, 136.35, 177.91; IR(neat): 1175 (C=O), 1260 (C=O), 1597 (C=C), 1644 (C=O), 2214 (C=C), 3406 (O-H)  $\text{cm}^{-1}$ ; HRMS (ESI positive):  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{15}\text{H}_{18}\text{O}_2\text{Na}$ , 253.1199; found, 253.1198.

#### **4-Hydroxy-1-(4-methoxyphenyl)non-2-yn-1-one (1h)**



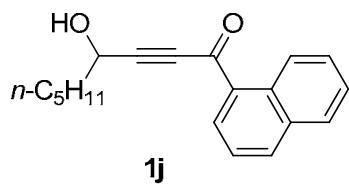
Colorless oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.06 (d,  $J = 9.2$  Hz, 2H), 6.89 (d,  $J = 9.2$  Hz, 2H), 4.67-4.62 (m, 1H), 3.84 (s, 3H), 3.54 (bs, 1H), 1.87-1.80 (2H, m), 1.54-1.48 (2H, m), 1.34-1.29 (4H, m), 0.88 (t,  $J = 7.2$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  13.87, 22.41, 24.75, 31.28, 36.95, 55.48, 62.21, 82.18, 94.85, 113.76, 129.68, 132.02, 164.52, 176.61; IR(neat): 688, 757, 844 (p-aromatic substituted), 1164 (C-O), 1252 (C-O), 1593 (C=C), 1639 (C=O), 2214 (C=C), 3408 (O-H)  $\text{cm}^{-1}$ ; HRMS (ESI positive):  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{16}\text{H}_{20}\text{O}_3\text{Na}$ , 283.1305; found, 283.1303.

#### **1-(4-Chlorophenyl)-4-hydroxynon-2-yn-1-one (1i)**



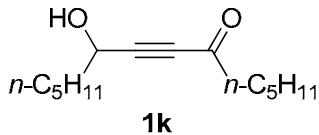
Colorless oil;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  8.04-7.99 (m, 2H), 7.42-7.38 (m, 2H), 4.66 (bs, 1H), 3.44 (bs, 1H), 1.88-1.80 (m, 2H), 1.53-1.49 (2H, m), 1.33-1.25 (4H, m), 0.90-0.85 (3H, m);  $^{13}\text{C}$  NMR (75.45 MHz,  $\text{CDCl}_3$ )  $\delta$  13.88, 22.42, 24.76, 31.26, 36.88, 62.26, 81.89, 96.06, 128.91, 130.86, 134.64, 140.94, 176.64; IR(neat): 1092 (C-O), 1256 (C-O), 1586 (C=C), 1649 (C=O), 2214 (C=C), 3391 (O-H)  $\text{cm}^{-1}$ ; HRMS (ESI positive):  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{15}\text{H}_{17}\text{ClO}_2\text{Na}$ , 287.0809; found, 287.0808.

#### **4-Hydroxy-1-(naphthalen-1-yl)non-2-yn-1-one (1j)**



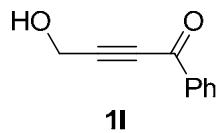
Colorless oil;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  9.19 (d,  $J = 8.4$  Hz, 1H), 8.54 (dd,  $J = 7.5, 1.2$  Hz, 1H), 7.99 (d,  $J = 8.4$  Hz, 1H), 7.84 (d,  $J = 7.5$  Hz, 1H), 7.63-7.55 (m, 1H), 7.53-7.44 (m, 2H), 4.74 (dt,  $J = 5.7, 5.7$  Hz, 1H), 3.84 (d,  $J = 5.7$  Hz, 1H), 1.96-1.88 (m, 2H), 1.63-1.53 (m, 2H), 1.37-1.28 (m, 4H), 0.91 (m, 3H);  $^{13}\text{C}$  NMR (75.45 MHz,  $\text{CDCl}_3$ )  $\delta$  13.86, 22.40, 24.77, 31.25, 36.92, 62.24, 83.67, 94.17, 124.26, 125.68, 126.60, 128.43, 128.89, 130.46, 131.98, 133.58, 135.10, 135.24, 179.59; IR(neat): 1139 (C-O), 1233 (C-O), 1637 (C=O), 2210 (C=C), 3385 (O-H)  $\text{cm}^{-1}$ ; HRMS (ESI positive):  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{19}\text{H}_{20}\text{O}_2\text{Na}$ , 303.1356; found, 303.1355.

#### **9-Hydroxytetradec-7-yn-6-one (1k)**



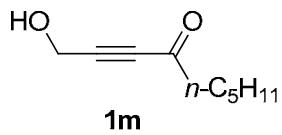
Yellow oil;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  4.58-4.52 (m, 1H), 3.08 (bs, 1H), 2.58 (t,  $J = 7.5$  Hz, 2H), 1.83-1.66 (m, 4H), 1.53-1.43 (m, 2H), 1.36-1.31 (m, 8H), 0.94-0.90 (m, 6H);  $^{13}\text{C}$  NMR (75.45 MHz,  $\text{CDCl}_3$ )  $\delta$  13.76, 13.87, 22.27, 22.41, 23.57, 24.64, 30.99, 31.26, 36.85, 45.29, 62.05, 83.36, 92.75, 188.28; IR(neat): 1158 (C-O), 1239 (C-O), 1647 (C=O), 2212 (C=C), 3404 (O-H)  $\text{cm}^{-1}$ ; HRMS (ESI positive):  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{14}\text{H}_{24}\text{O}_2\text{Na}$ , 247.1669; found, 247.1667.

#### **4-Hydroxy-1-phenylbut-2-yn-1-one (1l)**



Brown oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.09-8.07 (m, 2H), 7.57-7.54 (m, 1H), 7.43-7.39 (m, 2H), 7.43-7.39 (m, 2H), 4.57 (s, 2H), 3.97 (bs, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  50.61, 82.89, 93.18, 128.50, 129.58, 134.39, 135.94, 178.10; IR(neat): 1101 (C-O), 1261 (C-O), 1590 (C=C), 1640 (C=O), 2230 (C=C), 3412 (O-H)  $\text{cm}^{-1}$ ; HRMS (ESI positive):  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{10}\text{H}_8\text{O}_2\text{Na}$ , 183.0417; found, 183.0416.

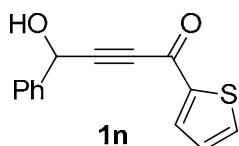
#### **1-Hydroxynon-2-yn-4-one (1m)**



Yellow oil;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  4.38 (s, 2H), 3.73 (bs, 1H), 2.51 (t,  $J = 7.2$  Hz, 2H), 1.65-1.55 (m, 2H), 1.29-1.28 (m, 4H), 0.83 (t,  $J = 7.2$  Hz, 3H);  $^{13}\text{C}$  NMR (75.45 MHz,  $\text{CDCl}_3$ )

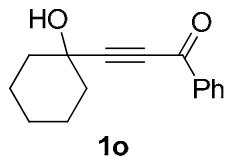
$\delta$  13.68, 22.16, 23.38, 30.88, 45.09, 50.28, 83.97, 90.43, 188.48; IR(neat): 1158 (C-O), 1239 (C-O), 1671 (C=O), 2217 (C=C), 3421 (O-H)  $\text{cm}^{-1}$ ; HRMS (ESI positive): [M+Na]<sup>+</sup> calcd for C<sub>9</sub>H<sub>14</sub>O<sub>2</sub>Na, 177.0886; found, 177.0886.

**4-Hydroxy-4-phenyl-1-(thiophen-2-yl)but-2-yn-1-one (1n)**



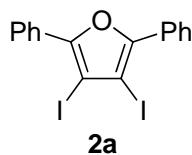
Yellow solid; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  7.92-7.90 (m, 1H), 7.70-7.68 (m, 1H), 7.68-7.54 (m, 2H), 7.42-7.35 (m, 3H), 7.11-7.09 (m, 1H), 5.69 (d,  $J$  = 6.0 Hz, 1H), 3.86 (d,  $J$  = 6.0 Hz, 1H); <sup>13</sup>C NMR (75.45 MHz, CDCl<sub>3</sub>)  $\delta$  64.28, 82.98, 92.12, 126.63, 128.42, 128.77, 135.85, 136.02 ( $\times 2$ ), 138.66, 144.11, 169.58; IR(neat): 1587 (C=C), 1613 (C=O), 2223 (C=C), 3087 (thienyl C-H), 3334 (O-H)  $\text{cm}^{-1}$ ; HRMS (ESI positive): [M+Na]<sup>+</sup> calcd for C<sub>14</sub>H<sub>10</sub>O<sub>2</sub>Na, 265.0294; found, 265.0293.

**3-(1-Hydroxycyclohexyl)-1-phenylprop-2-yn-1-one (1o)**



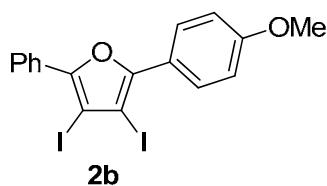
Yellow oil; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  1.76-1.20 (8H, m), 2.08-2.00 (2H, m), 3.88 (1H, s), 7.43-7.38 (2H, m), 7.57-7.51 (1H, m), 8.11-8.07 (2H, m); <sup>13</sup>C NMR (75.45 MHz, CDCl<sub>3</sub>)  $\delta$  22.90, 24.80, 39.03, 68.56, 81.66, 98.20, 128.42, 129.44, 134.07, 136.33, 178.06; IR(neat): 1061 (C-O), 1258 (C-O), 1641 (C=O), 2206 (C=C), 3387 (O-H)  $\text{cm}^{-1}$ ; HRMS (ESI positive): [M+Na]<sup>+</sup> calcd for C<sub>15</sub>H<sub>16</sub>O<sub>2</sub>Na, 251.1043; found, 251.1043.

**3,4-Diiodo-2,5-diphenylfuran (2a)**



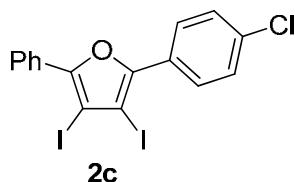
Yellow solid; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>)  $\delta$  8.08-8.04 (m, 4H), 7.50-7.35 (m, 4H), 7.42-7.39 (m, 2H); <sup>13</sup>C NMR (75.45 MHz, CDCl<sub>3</sub>)  $\delta$  78.97, 126.60, 128.45, 128.76, 129.68, 152.58; IR(neat): 761, 934, 957, 1474, 1598  $\text{cm}^{-1}$ ; EI: [m/z] calcd for C<sub>16</sub>H<sub>10</sub>I<sub>2</sub>O, 471.8821; found, 471.8818.

**3,4-Diiodo-2-(4-methoxyphenyl)-5-phenylfuran (2b)**



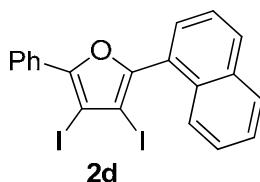
Yellow solid;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  8.06-7.97 (m, 4H), 7.49-7.35 (m, 3H), 7.01-6.98 (m, 2H), 3.87 (s, 3H);  $^{13}\text{C}$  NMR (75.45 MHz,  $\text{CDCl}_3$ )  $\delta$  55.13, 78.41, 113.62, 113.65, 122.22, 126.27, 127.98, 128.20, 128.36, 129.59, 151.81, 152.58, 159.72; IR(neat): 761, 832, 934, 958, 1493, 1610  $\text{cm}^{-1}$ ; EI: [m/z] calcd for  $\text{C}_{17}\text{H}_{12}\text{I}_2\text{O}_2$ , 501.8927; found, 501.8927.

**2-(4-Chlorophenyl)-3,4-diodo-5-phenylfuran (2c)**



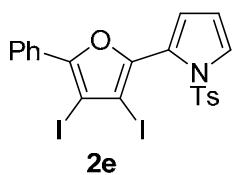
Yellow solid;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  8.05-7.98 (m, 4H), 7.50-7.37 (m, 5H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  79.06, 79.52, 126.65, 127.71, 128.11, 128.49, 128.70, 128.93, 129.50, 134.55, 151.54, 152.83; IR(neat): 759, 830, 933, 959, 1474  $\text{cm}^{-1}$ ; EI: [m/z] calcd for  $\text{C}_{16}\text{H}_9\text{ClI}_2\text{O}$ , 505.8431; found, 505.8434.

**3,4-Diodo-2-(naphthalen-1-yl)-5-phenylfuran (2d)**



Yellow solid;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  8.09-8.05 (m, 2H), 7.99-7.93 (m, 3H), 7.78-7.75 (m, 1H), 7.61-7.52 (m, 3H), 7.50-7.36 (m, 3H);  $^{13}\text{C}$  NMR (75 MHz,  $\text{CDCl}_3$ )  $\delta$  76.63, 83.41, 124.91, 125.91, 126.27, 126.52, 126.86, 127.48, 128.45, 128.48, 128.76, 129.51, 129.74, 130.35, 131.51, 133.69, 153.35, 154.09; IR(neat): 761, 799, 928, 948, 1473, 1541  $\text{cm}^{-1}$ ; EI: [m/z] calcd for  $\text{C}_{20}\text{H}_{12}\text{I}_2\text{O}$ , 521.8978; found, 521.8977.

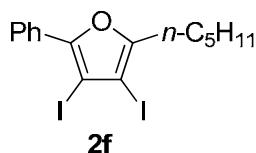
**2-(3,4-Diodo-5-phenylfuran-2-yl)-1-tosyl-1H-pyrrole (2e)**



Yellow solid;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.97-7.95 (m, 2H), 7.55-7.43 (m, 6H), 7.16-7.14 (m, 2H), 6.58-6.57 (m, 1H), 6.39-6.37 (m, 1H), 2.35 (s, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  21.61, 75.45, 87.12, 111.41, 120.20, 122.48, 125.09, 126.80, 127.37, 128.40, 128.98, 129.46, 129.90,

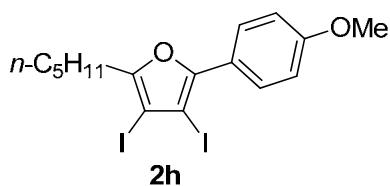
135.28, 145.12, 145.29, 153.81 cm<sup>-1</sup> IR(neat): 741, 763, 810, 1147 (R-SO<sub>2</sub>-N) cm<sup>-1</sup>; HRMS (ESI positive): [M+Na]<sup>+</sup> calcd for C<sub>21</sub>H<sub>15</sub>I<sub>2</sub>NO<sub>3</sub>Na, 637.8754; found, 637.8752.

**3,4-Diiodo-2-pentyl-5-phenylfuran (2f)**



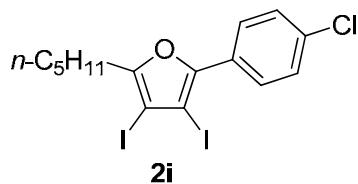
Colorless oil; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.95-7.93 (m, 2H), 7.45-7.41 (m, 2H), 7.36-7.32 (m, 1H), 1.69 (t, *J* = 7.2 Hz, 2H), 1.38-1.34 (m, 4H), 0.92 (t, *J* = 6.4 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 13.96, 22.34, 27.67, 28.72, 31.12, 74.76, 79.45, 126.21, 128.30, 128.37, 130.05, 151.78, 157.46; IR(neat): 761, 946, 1484, 1572 cm<sup>-1</sup>; EI: [m/z] calcd for C<sub>15</sub>H<sub>16</sub>I<sub>2</sub>O, 465.9291; found, 465.9290.

**3,4-Diiodo-2-(4-methoxyphenyl)-5-pentylfuran (2h)**



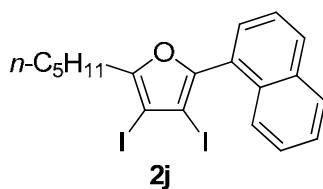
Colorless oil; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.88 (d, *J* = 9.2 Hz, 2H), 6.96 (d, *J* = 9.2 Hz, 2H), 3.85 (s, 3H), 2.81 (t, *J* = 7.2 Hz, 2H), 1.73-1.66 (m, 2H), 1.40-1.35 (m, 4H), 0.93 (t, *J* = 6.8 Hz, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 13.97, 22.32, 27.69, 28.66, 31.10, 55.29, 73.20, 79.02, 113.71, 122.82, 127.74, 151.90, 156.75, 159.52; IR(neat): 783, 798, 946, 1493, 1611 cm<sup>-1</sup>; EI: [m/z] calcd for C<sub>16</sub>H<sub>18</sub>I<sub>2</sub>O<sub>2</sub>, 495.9396; found, 495.9391.

**2-(4-Chlorophenyl)-3,4-diiodo-5-pentylfuran (2i)**



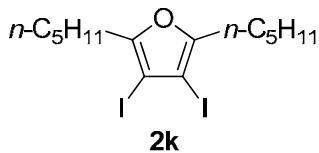
Colorless oil; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>) δ 7.90 (d, *J* = 8.4 Hz, 2H), 7.40 (d, *J* = 8.4 Hz, 2H), 2.82 (t, *J* = 7.2 Hz, 2H), 1.73-1.66 (m, 2H), 1.40-1.35 (m, 4H), 0.93 (t, *J* = 6.8 Hz, 3H); <sup>13</sup>C NMR (75 MHz, CDCl<sub>3</sub>) δ 13.96, 22.32, 27.64, 28.70, 31.11, 75.42, 79.65, 127.29, 128.44, 128.58, 133.99, 150.73, 157.69; IR(neat): 712, 825, 946, 1478, 1540 cm<sup>-1</sup>; EI: [m/z] calcd for C<sub>15</sub>H<sub>15</sub>ClI<sub>2</sub>O, 499.8901; found, 499.8905.

**3,4-Diiodo-2-(naphthalen-1-yl)-5-pentylfuran (2j)**



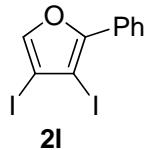
Colrless oil;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.95-7.91 (m, 3H), 7.70-7.68 (m, 1H), 7.57-7.51 (m, 3H), 2.89 (t,  $J = 7.6$  Hz, 2H), 1.78-1.70 (m, 2H), 1.46-1.38 (m, 4H), 0.94 (t,  $J = 7.2$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  13.99, 22.34, 27.69, 28.76, 31.14, 77.38, 79.18, 124.90, 125.90, 126.14, 126.62, 127.75, 128.38, 129.27, 130.01, 131.51, 133.68, 153.13, 158.08; IR(neat): 772, 799, 906, 1506, 1569  $\text{cm}^{-1}$ ; EI: [m/z] calcd for  $\text{C}_{19}\text{H}_{18}\text{I}_2\text{O}$ , 515.9447; found, 515.9447.

### 3,4-Diiodo-2,5-dipentylfuran (2k)



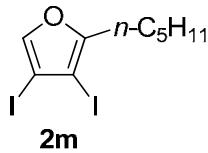
Colrless oil;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  2.70 (t,  $J = 7.5$  Hz, 4H), 1.65-1.55 (m, 4H), 1.37-1.26 (m, 8H), 0.90 (t,  $J = 6.9$  Hz, 6H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  12.65, 21.74, 47.95, 55.73, 55.73, 103.25, 106.05, 118.64, 121.24, 125.08, 130.55, 133.38, 146.65, 148.31, 149.37; IR(neat): 732, 948, 1457, 1559  $\text{cm}^{-1}$ ; EI: [m/z] calcd for  $\text{C}_{14}\text{H}_{22}\text{I}_2\text{O}$ , 459.9760; found, 459.9760.

### 3,4-Diiodo-2-phenylfuran (2l)



Brown solid;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.97-7.95 (m, 2H), 7.56 (s, 1H), 7.47-7.43 (m, 2H), 7.40-7.36 (m, 1H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  74.44, 81.57, 126.45, 128.43, 128.80, 129.56, 145.19, 153.56; IR(neat): 762, 899, 947, 1477, 1507,  $\text{cm}^{-1}$ ; EI: [m/z] calcd for  $\text{C}_{10}\text{H}_{16}\text{I}_2\text{O}$ , 395.8508; found, 395.8502.

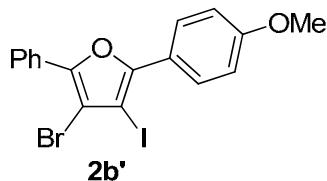
### 3,4-Diiodo-2-pentylfuran (2m)



Colrless oil;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.41 (s, 1H), 2.74 (t,  $J = 7.5$  Hz, 2H), 1.67-1.57 (m, 2H), 1.37-1.25 (m, 4H), 0.89 (t,  $J = 6.9$  Hz, 3H);  $^{13}\text{C}$  NMR (100 MHz,  $\text{CDCl}_3$ )  $\delta$  13.94, 22.30, 27.50, 28.56, 31.04, 75.08, 77.54, 144.32, 158.31; IR(neat): 747, 913, 949, 1457, 1508, 1567

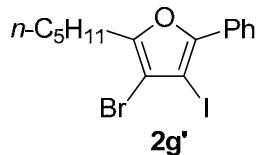
cm<sup>-1</sup>; EI: [m/z] calcd for C<sub>9</sub>H<sub>12</sub>I<sub>2</sub>O, 389.8978; found, 389.8975.

**3-Bromo-4-iodo-5-(4-methoxyphenyl)-2-phenylfuran (2b')**



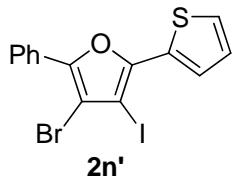
White solid; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 8.05-7.99 (m, 4H), 7.49-7.34 (m, 3H), 7.01-6.98 (m, 2H), 3.86 (s, 3H); <sup>13</sup>C NMR (75.45 MHz, CDCl<sub>3</sub>) δ 55.29, 71.63, 106.26, 113.84, 122.30, 125.52, 127.94, 128.26, 128.48, 129.17, 148.14, 151.55, 159.91; IR(neat): 759, 818, 835, 936, 1494, 1605 cm<sup>-1</sup>; EI: [m/z] calcd for C<sub>17</sub>H<sub>12</sub>BrIO<sub>2</sub>, 453.9065; found, 453.9055.

**3-Bromo-4-iodo-2-pentyl-5-phenylfuran (2g')**



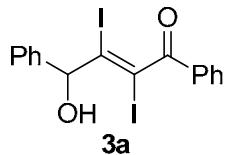
Colorless oil; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 7.97-7.93 (m, 2H), 7.46-7.41 (m, 2H), 7.37-7.34 (m, 1H), 2.78 (t, J = 7.5 Hz, 2H), 1.75-1.65 (m, 2H), 1.40-1.33 (m, 4H), 0.91 (t, 3H); <sup>13</sup>C NMR (100 MHz, CDCl<sub>3</sub>) δ 13.95, 22.30, 27.11, 27.36, 31.13, 69.65, 106.47, 126.06, 128.31, 128.39, 130.01, 150.72, 153.26; IR(neat): 761, 947, 1587 cm<sup>-1</sup>; EI: [m/z] calcd for C<sub>15</sub>H<sub>16</sub>BrIO, 417.9429; found, 417.9427.

**3-Bromo-4-iodo-2-phenyl-5-(thiophen-2-yl)furan (2n')**



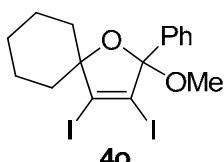
Yellow solid; <sup>1</sup>H NMR (300 MHz, CDCl<sub>3</sub>) δ 8.01-7.98 (m, 2H), 7.78-7.77 (m, 1H), 7.47-7.32 (m, 4H), 7.14-7.12 (m, 1H); <sup>13</sup>C NMR (125 MHz, CDCl<sub>3</sub>) δ 72.84, 106.13, 125.71, 125.73, 126.14, 126.58, 127.54, 128.58, 128.60, 131.62, 148.22, 148.79; IR(neat): 760, 847, 900, 961, 1482 cm<sup>-1</sup>; EI: [m/z] calcd for C<sub>14</sub>H<sub>18</sub>BrIOS, 429.8524; found, 429.8528.

**(E)-4-Hydroxy-2,3-diodo-1,4-diphenylbut-2-en-1-one (3a)**



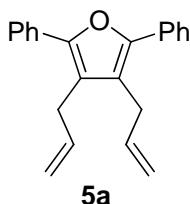
Yellow solid;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.98-7.95 (m, 2H), 7.63-7.60 (m, 3H), 7.51-7.42 (m, 5H), 5.62 (s, 1H), 2.88 (bs, 1H);  $^{13}\text{C}$  NMR (75.45 MHz,  $\text{CDCl}_3$ )  $\delta$  78.99, 91.51, 111.35, 125.78, 128.46, 128.63, 129.11, 130.42, 131.24, 134.34, 139.65, 191.10; IR(neat): 1230 (C-O), 1645 (C=C), 1738 (C=O), 3497 (O-H)  $\text{cm}^{-1}$ ; HRMS (ESI positive):  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{16}\text{H}_{12}\text{I}_2\text{O}_2\text{Na}$ , 512.8819; found, 512.8816.

**3,4-diiodo-2-methoxy-2-phenyl-1-oxaspiro[4.5]dec-3-ene (4o)**



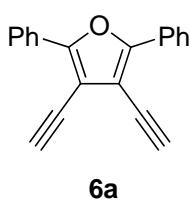
White solid;  $^1\text{H}$  NMR (300 MHz,  $\text{CDCl}_3$ )  $\delta$  7.61-7.58 (m, 2H), 7.41-7.34 (m, 3H), 3.30 (s, 3H), 1.92-1.74 (m, 9H), 1.34-1.20 (m, 1H);  $^{13}\text{C}$  NMR (75.45 MHz,  $\text{CDCl}_3$ )  $\delta$  21.80, 22.05, 24.79, 35.17, 35.54, 50.18, 93.13, 108.88, 113.61, 120.04, 127.52, 127.88, 128.53, 139.15; IR(neat): 1243 (C-O-C), 1586 (C=C)  $\text{cm}^{-1}$ ; HRMS (ESI positive):  $[\text{M}+\text{Na}]^+$  calcd for  $\text{C}_{16}\text{H}_{18}\text{I}_2\text{O}_2\text{Na}$ , 518.9288; found, 518.9284.

**3,4-Diallyl-2,5-diphenylfuran (5a)**



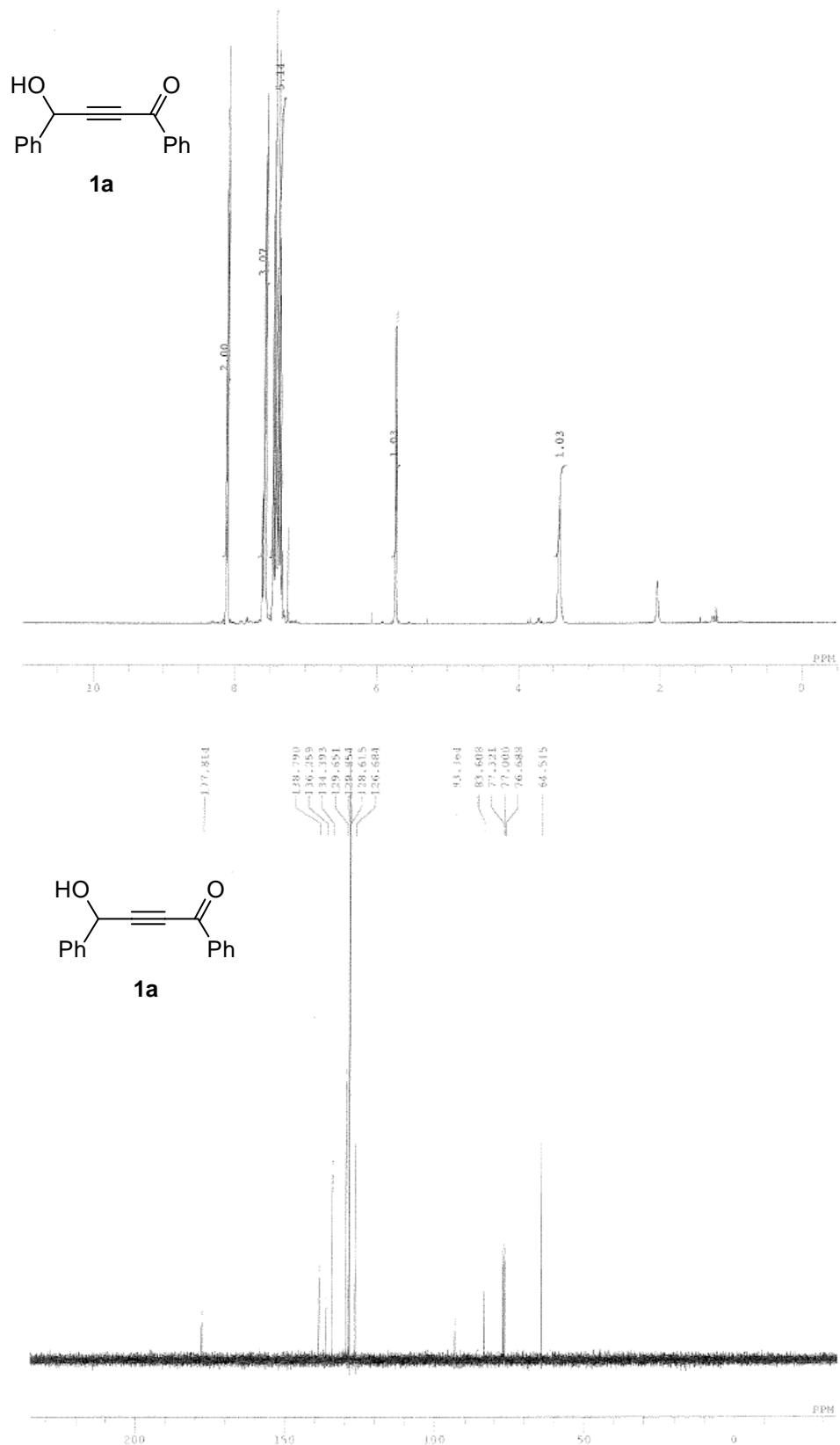
White solid;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  7.67 (d, 4H,  $J = 8.0$  Hz), 7.39 (d, 4H,  $J = 8.0$  Hz), 7.27-7.24 (m, 2H), 6.07-5.98 (m, 2H), 5.10 (d, 2H,  $J = 11.6$  Hz), 5.06 (d, 2H,  $J = 18.4$  Hz), 3.38-3.36 (m, 4H);  $^{13}\text{C}$  NMR (100.40 MHz,  $\text{CDCl}_3$ )  $\delta$  28.18, 115.78, 120.31, 125.50, 126.97, 128.46, 131.23, 135.65, 148.53; IR(neat): 759, 1491, 1593  $\text{cm}^{-1}$ ; EI: [m/z] calcd for  $\text{C}_{22}\text{H}_{20}\text{O}$ , 300.1514; found, 300.1513.

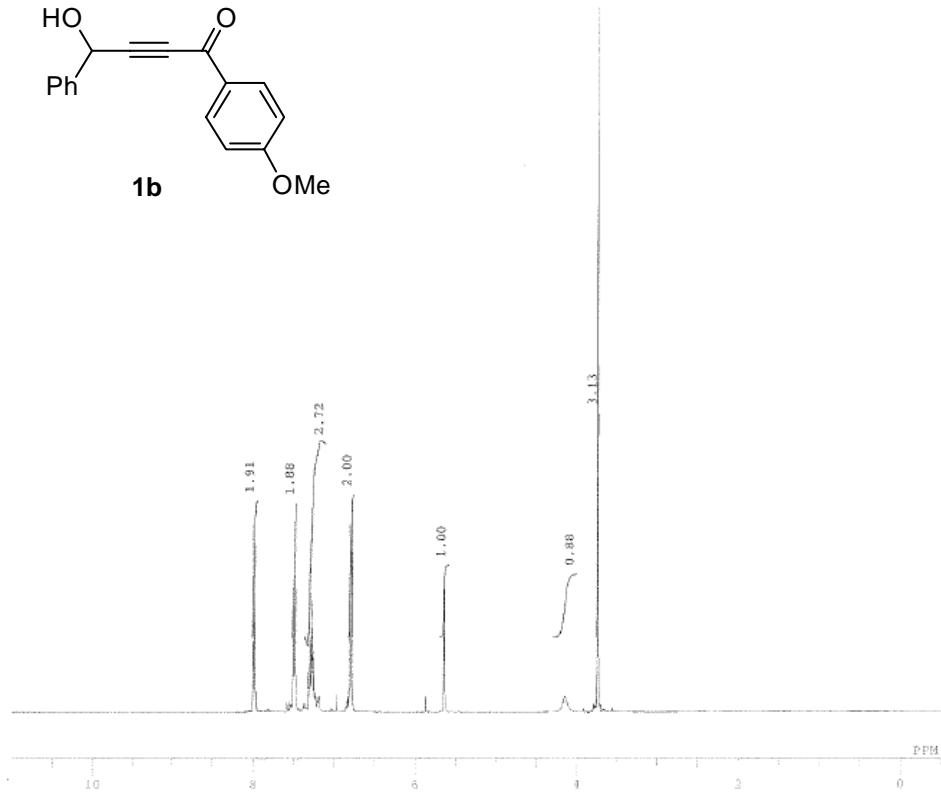
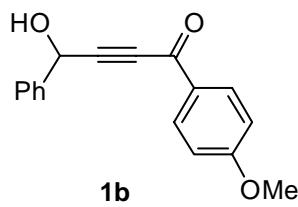
**3,4-Diethynyl-2,5-diphenylfuran (6a)**



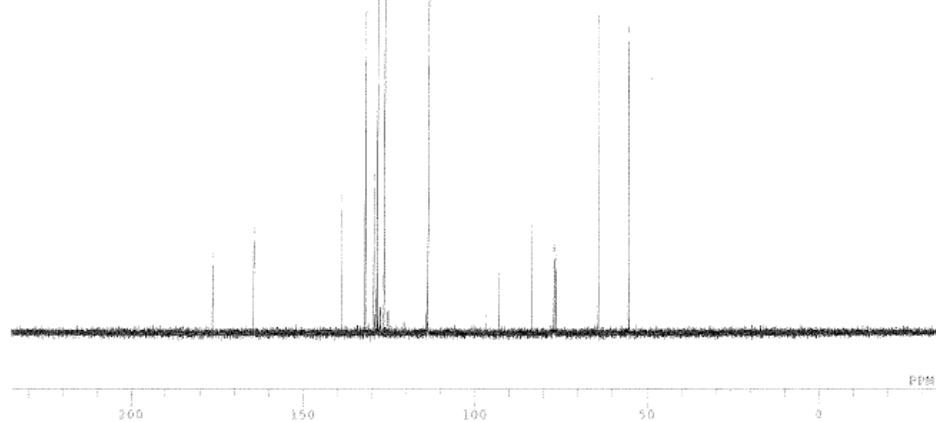
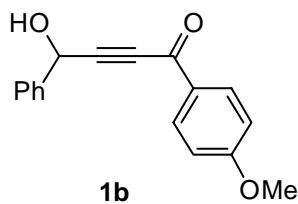
Gray solid;  $^1\text{H}$  NMR (400 MHz,  $\text{CDCl}_3$ )  $\delta$  8.16-8.13 (m, 4H), 7.49-7.44 (m, 4H), 7.40-7.35 (m, 2H), 3.57 (s, 2H);  $^{13}\text{C}$  NMR (100.40 MHz,  $\text{CDCl}_3$ )  $\delta$  75.21, 84.85, 106.28, 124.98, 128.59, 128.79, 129.20, 153.80; IR(neat): 2340 (C-C), 3292 (-C-H)  $\text{cm}^{-1}$ ; EI: [m/z] calcd for  $\text{C}_{20}\text{H}_{12}\text{O}$ ,

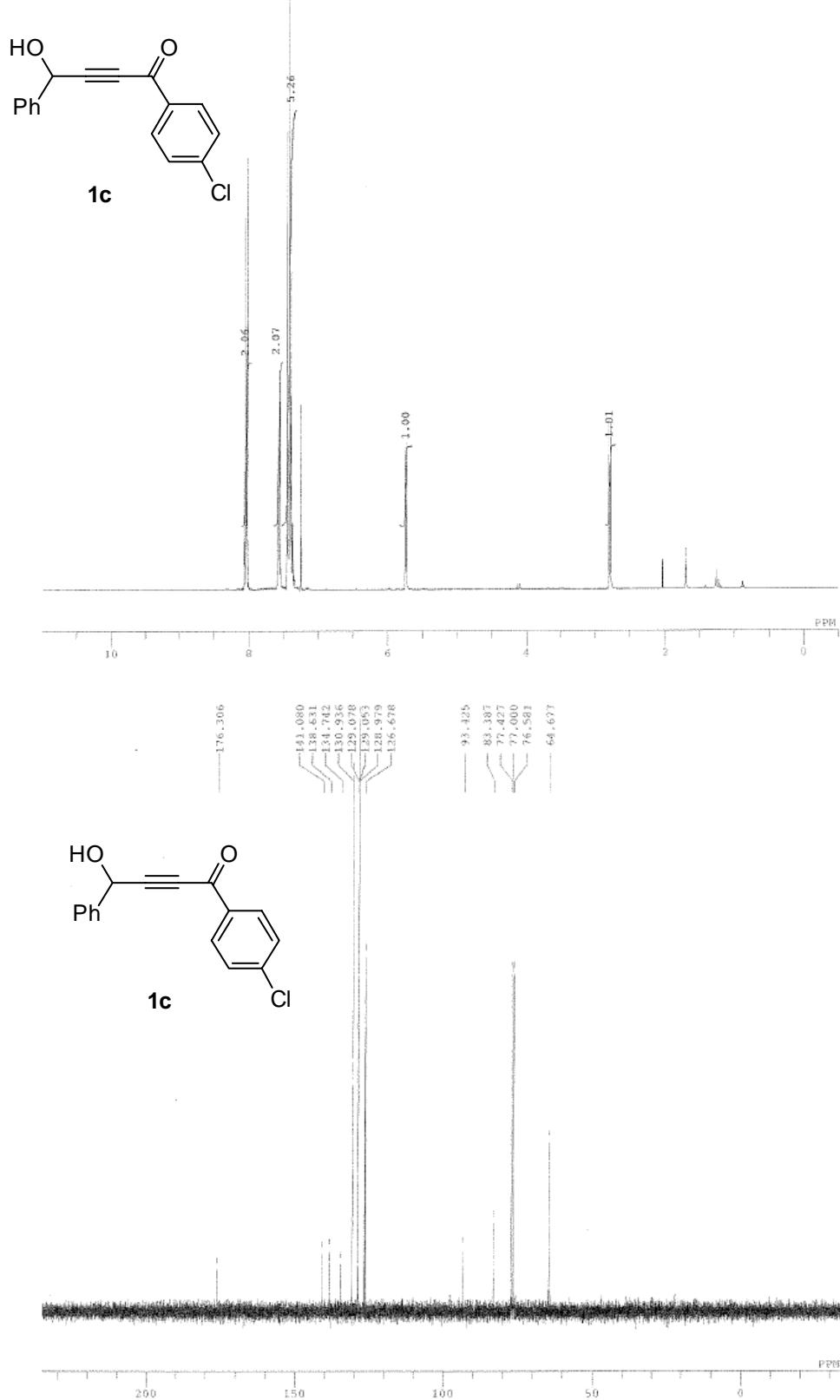
268.0888; found, 268.0888.

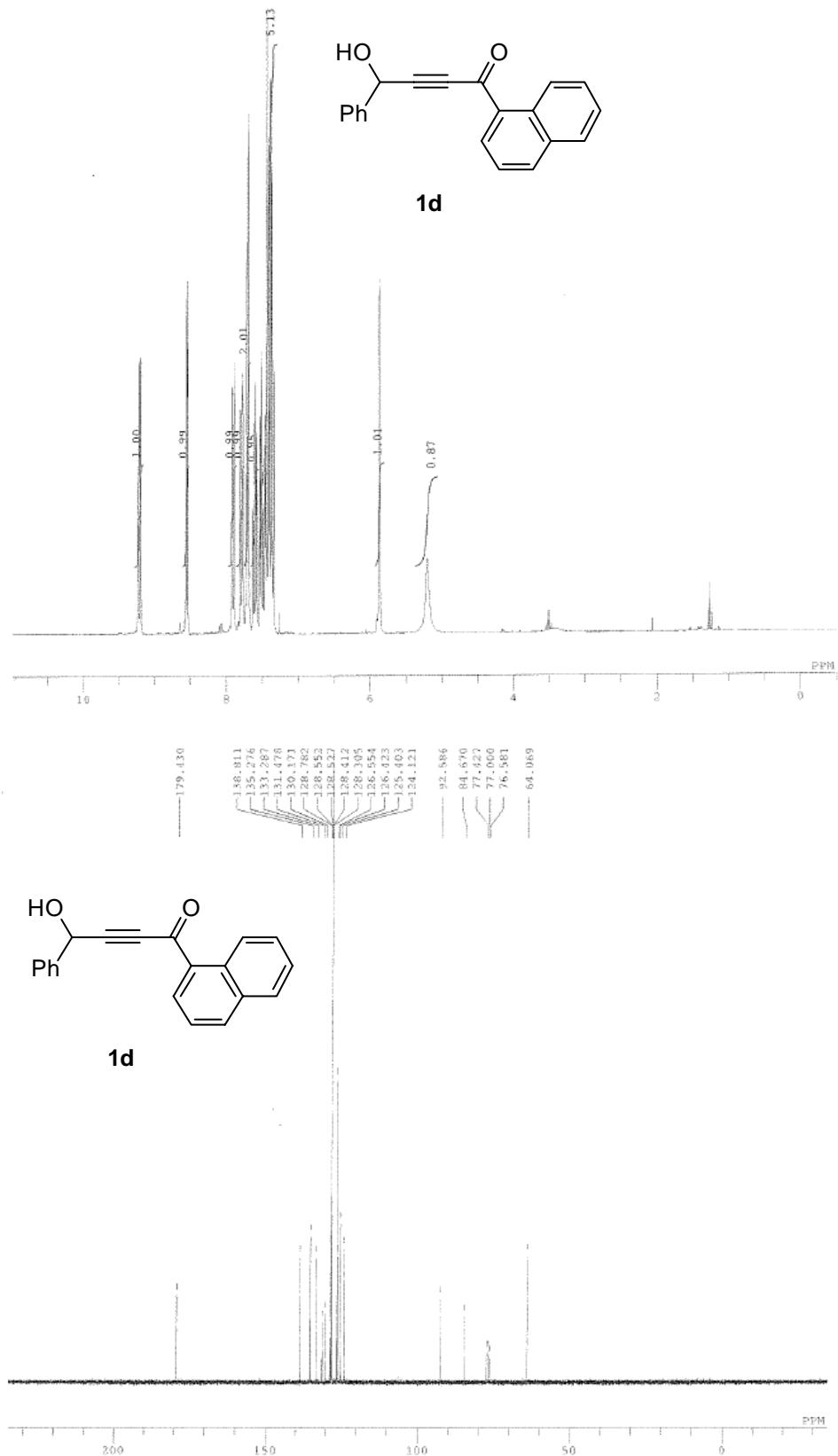


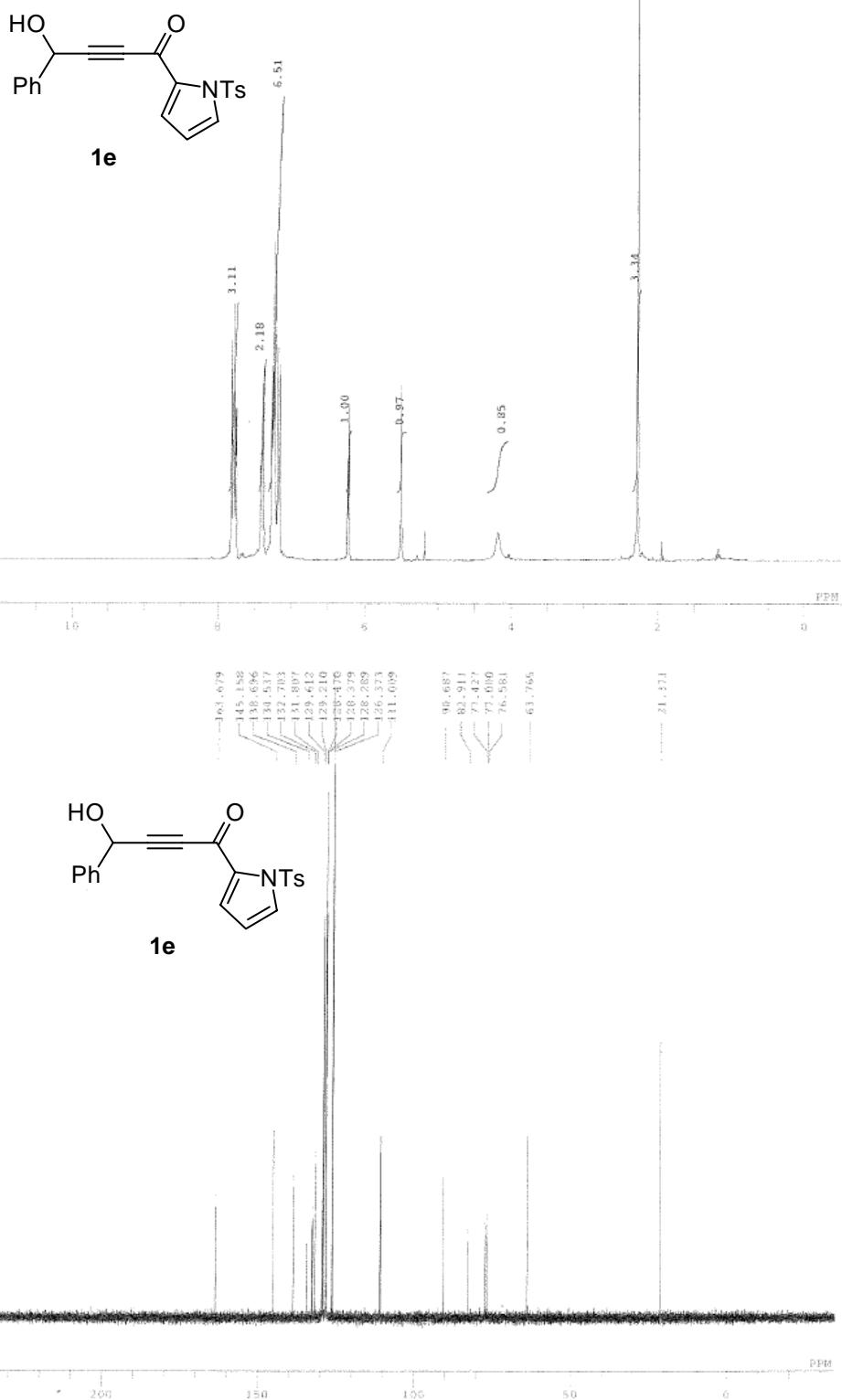


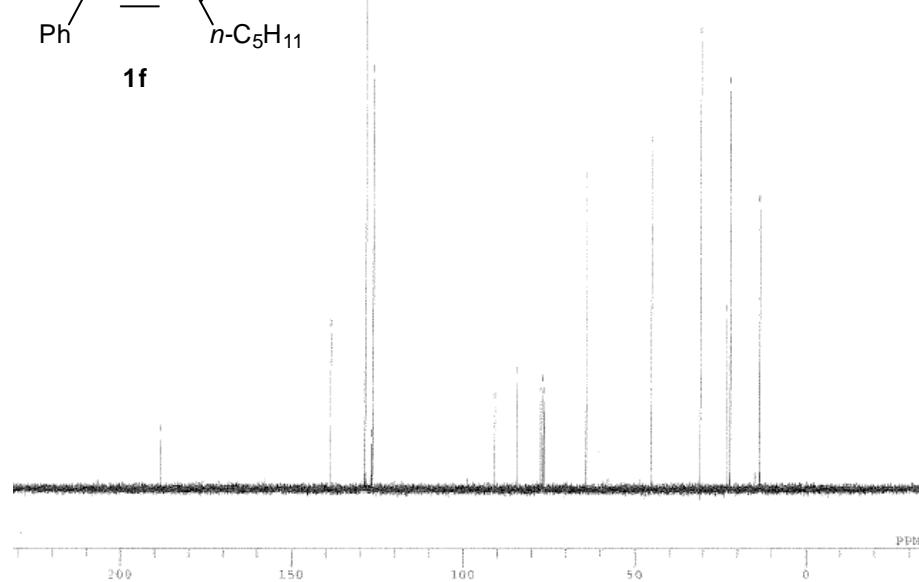
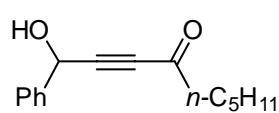
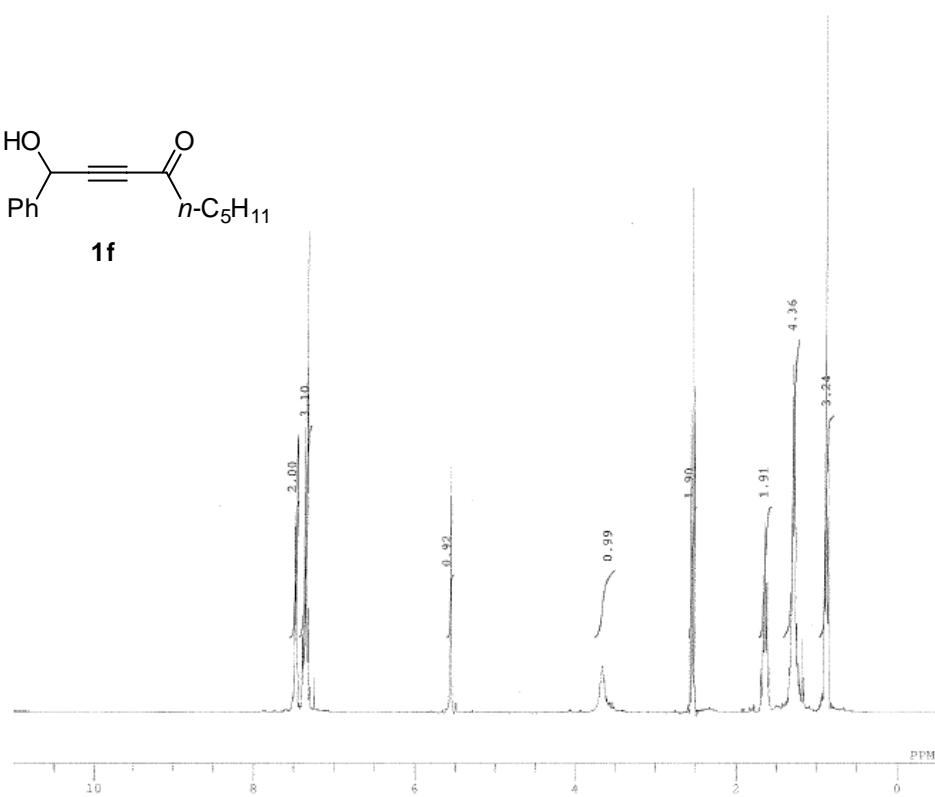
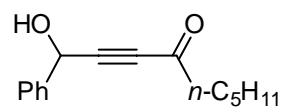
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164.565  
138.971  
132.067  
129.569  
128.634  
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126.626  
113.796

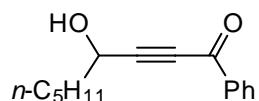




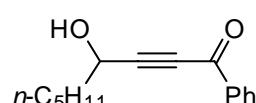
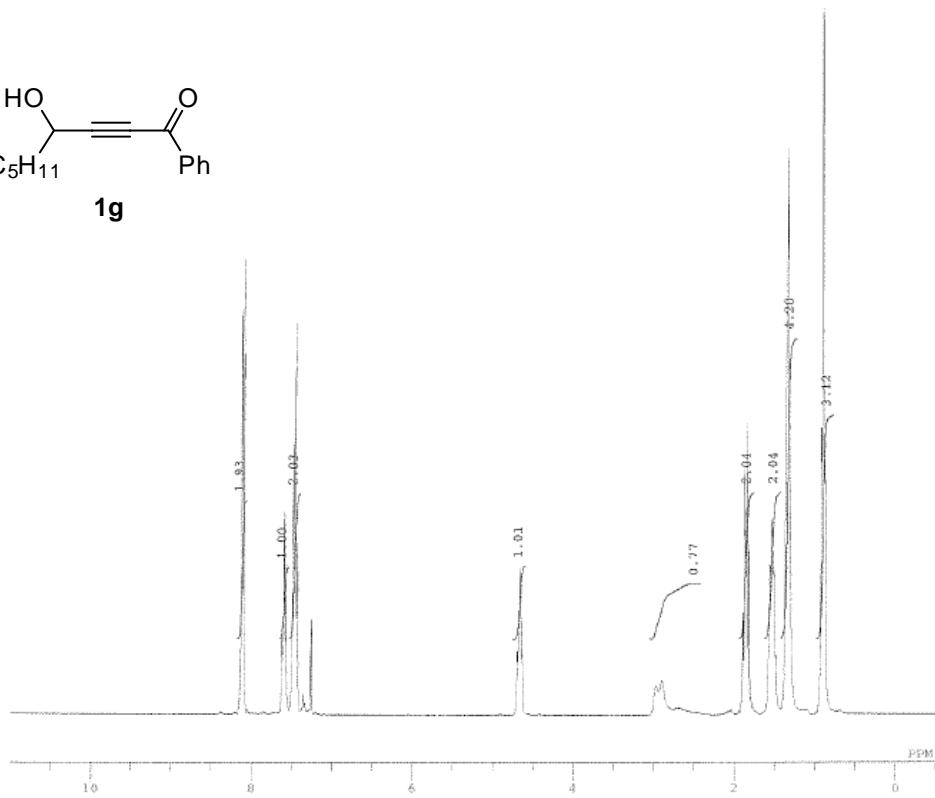








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