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Silver-catalyzed ring-opening [3 + 2] annulation of cyclopropenones with amides

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Supporting Information

General. All reactions were carried out with standard Schlenk techniques under a nitrogen atmosphere. Column chromatography was carried out on Wakogel® C-200 (75–150 μm). Preparative thin-layer chromatography (TLC) was performed on Wakogel® B-5F. Proton chemical shifts (δ) were referenced to residual $CHCl_3$ (at 7.26 ppm) or C_6D_6 (at 7.16 ppm). Carbon chemical shifts (δ) were referenced to $CDCl_3$ (at 77.0 ppm) or acetone-d_6 (at 29.9 and 206.3 ppm).

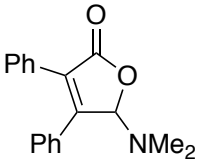
Materials. Cyclopropenones **1b–f**[1] and amide **2h**[2] were prepared according to the literature methods. All other reagents and solvents were obtained from commercial sources and used without further purification.

1 (*a*) C. M. Vanos and T. H. Lambert, *Angew. Chem. Int. Ed.*, 2011, **50**, 12222–12226; (*b*) E. D. Nacsa and T. H. Lambert, *Org. Lett.*, 2013, **15**, 38–41; (*c*) P. A. Wender, T. J. Paxton and T. J. Williams, *J. Am. Chem. Soc.*, 2006, **128**, 14814−14815; (*d*) P. A. Peart and J. D. Tovar, *J. Org. Chem.*, 2010, **75**, 5689–5696; (*e*) J.-R. Syu, C.-H. Lin, C.-W. Kuo and D.-Y. Yang, *Tetrahedron Lett.*, 2014, **55**, 1207–1211.

2 D. Habibi, P. Rahmani, Z. Akbaripanah, *J. Chem.*, 2013, 972960.

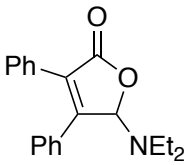
General Procedure for the Silver(I)-Catalyzed Ring-Opening Annulation of Cyclopropenones 1 with Amides 2: Neat Conditions (GP-1). A Schlenk tube was charged with AgOTf (0.010 mmol) and cyclopropenone **1** (0.10 mmol), and the tube was evacuated and backfilled with nitrogen. Amide **2** (0.50 mL) was added via a syringe through the septum, and the mixture was heated at 130 °C for 2 h. After cooling to room temperature, the reaction mixture was filtered through a plug of silica gel eluting with hexane–AcOEt, and the filtrate was concentrated. The residue was purified by preparative TLC or column chromatography on silica gel to afford furan-2(5*H*)-one **3**.

General Procedure for the Silver(I)-Catalyzed Ring-Opening Annulation of Cyclopropenones 1 with Amides 2: Dropwise Addition (GP-2). A Schlenk tube was charged with AgOTf (0.010 mmol), and the tube was evacuated and backfilled with nitrogen. Amide **2** (1.00 or 2.00 mmol) and chlorobenzene (0.20 mL) were added successively via a syringe through the septum, and the mixture was heated at 130 °C. To the mixture was added dropwise a solution of cyclopropenone **1** (0.10 mmol) in chlorobenzene (0.40 mL) over 80 min, and the resulting solution was further heated for 40 min. After cooling to room temperature, the reaction mixture was filtered through a plug of silica gel eluting with hexane–AcOEt, and the filtrate was concentrated. The residue was purified by preparative TLC or column chromatography on silica gel to afford furan-2(5*H*)-one **3**.

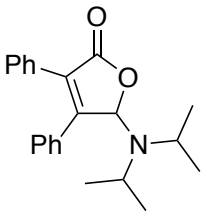


5-(Dimethylamino)-3,4-diphenylfuran-2(5*H*)-one (3a). The general procedure (GP-1) was followed using **1a** (20.6 mg, 0.100 mmol) and **2a** (0.5 mL, 6.5 mmol). Purification by

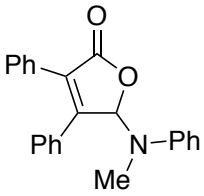
preparative TLC (hexane:AcOEt = 3:1) yielded **3a** (26.6 mg, 0.095 mmol, 95%) as a white solid. According to GP-2, **3a** (27.0/27.3 mg, 0.097/0.098 mmol, 97/98%) was obtained by reacting **1a** (20.6/20.6 mg, 0.100/0.100 mmol) with **2a** (72.6/146.7 mg, 0.993/2.007 mmol). Mp 140–143 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.46 (s, 6H), 6.05 (s, 1H), 7.28–7.43 (m, 10H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 38.8, 98.0, 128.52, 128.54, 128.6, 128.7, 128.8, 129.4, 129.7, 130.0, 130.9, 154.2, 171.3; HRMS (ESI) cacld for $C_{18}H_{18}NO_2$ $[M + H]^+$ 280.1332, found 280.1334; IR (ν/cm^{-1}): 1730, 967, 696.



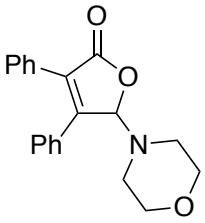
5-(Diethylamino)-3,4-diphenylfuran-2(5*H*)-one (3b). The general procedure (GP-1) was followed using **1a** (20.6 mg, 0.100 mmol) and **2b** (0.5 mL, 4.5 mmol). Purification by preparative TLC (hexane:AcOEt = 4:1) yielded **3b** (30.1 mg, 0.098 mmol, 98%) as a white solid. According to GP-2, **3b** (22.8/22.8 mg, 0.074/0.074 mmol, 76/79%) was obtained by reacting **1a** (20.1/19.3 mg, 0.097/0.094 mmol) with **2b** (101.5/202.0 mg, 1.003/1.997 mmol). Mp 121–128 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.02 (t, J = 7.0 Hz, 3H+3H), 2.74–2.87 (m, 4H), 6.26 (s, 1H), 7.25–7.32 (m, 2H), 7.32–7.45 (m, 8H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 13.2, 42.3, 96.9, 128.3, 128.5, 128.7, 128.8, 129.4, 129.7, 129.9, 130.2, 131.1, 154.4, 171.5; HRMS (ESI) cacld for $C_{20}H_{22}NO_2$ $[M + H]^+$ 308.1645, found 308.1645; IR (ν/cm^{-1}): 1739, 1173, 693.



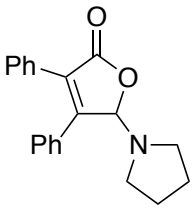
5-(Diisopropylamino)-3,4-diphenylfuran-2(5*H*)-one (3c). The general procedure (GP-1) was followed using **1a** (20.6 mg, 0.100 mmol) and **2c** (0.5 mL, 3.4 mmol). Purification by column chromatography on silica gel (hexane:AcOEt = 10:1) yielded **3c** (29.6 mg, 0.088 mmol, 88%) as a white solid. According to GP-2, **3c** (18.3/18.1 mg, 0.055/0.054 mmol, 59/56%) was obtained by reacting **1a** (19.1/20.0 mg, 0.093/0.097 mmol) with **2c** (131.1/249.3 mg, 1.015/1.930 mmol). Mp 141–143 °C; ^{1}H NMR (500 MHz, C_6D_6) δ 0.57 (br s, 6H), 1.09 (br s, 6H), 2.86 (septet, J = 6.5 Hz, 1H+1H), 5.70 (s, 1H), 6.90–6.96 (m, 3H), 6.98–7.07 (m, 3H), 7.07–7.12 (m, 2H), 7.67–7.71 (m, 2H); ^{13}C NMR (126 MHz, acetone-d_6) δ 22.4, 45.4, 94.4, 128.3, 128.4, 128.6, 129.0, 129.5, 129.9, 130.0, 131.0, 132.4, 156.4, 170.7; HRMS (ESI) cacld for $C_{22}H_{26}NO_2$ $[M + H]^+$ 336.1958, found 336.1957; IR (ν/cm^{-1}): 2966, 1728, 1147, 691.



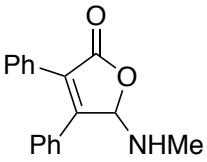
5-[Methyl(phenyl)amino]-3,4-diphenylfuran-2(5*H*)-one (3d). The general procedure (GP-1) was followed using **1a** (20.6 mg, 0.100 mmol) and **2d** (0.5 mL, 4.1 mmol). Purification by column chromatography on silica gel (hexane:AcOEt = 5:1) yielded **3d** (23.3 mg, 0.068 mmol, 68%) as a white solid. According to GP-2, **3d** (10.7/16.9 mg, 0.031/0.050 mmol, 32/55%) was obtained by reacting **1a** (20.3/18.4 mg, 0.098/0.089 mmol) with **2d** (135.4/270.4 mg, 1.002/2.001 mmol). Mp 148–155 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.73 (s, 3H), 6.84 (s, 1H), 7.02 (d, J = 7.5 Hz, 1H), 7.13 (d, J = 8.0 Hz, 2H), 7.27–7.41 (m, 10H), 7.44–7.49 (m, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 32.6, 93.0, 117.8, 121.6, 128.5, 128.66, 128.75, 129.1, 129.3, 129.4, 129.81, 129.83, 130.4, 130.5, 148.8, 156.7, 171.1; HRMS (ESI) cacld for $C_{23}H_{20}NO_2$ $[M + H]^+$ 342.1489, found 342.1487; IR (ν/cm^{-1}): 1742, 948, 753, 692.



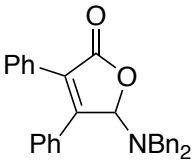
5-Morpholino-3,4-diphenylfuran-2(5*H*)-one (3e). The general procedure (GP-1) was followed using **1a** (20.6 mg, 0.100 mmol) and **2e** (0.5 mL, 5.0 mmol). Purification by column chromatography on silica gel (hexane:AcOEt = 2:1) yielded **3e** (21.6 mg, 0.067 mmol, 67%) as a white solid. According to GP-2, **3e** (18.5/23.4 mg, 0.058/0.073 mmol, 59/73%) was obtained by reacting **1a** (20.0/20.6 mg, 0.097/0.100 mmol) with **2e** (120.9/230.6 mg, 1.050/2.003 mmol). Mp 159–160 °C; ^{1}H NMR (301 MHz, $CDCl_3$) δ 2.76–2.91 (m, 4H), 3.54–3.72 (m, 4H), 6.00 (s, 1H), 7.26–7.50 (m, 10H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 47.2, 66.7, 96.9, 128.5, 128.6, 128.7, 129.0, 129.4, 129.9, 130.0, 130.2, 130.7, 153.1, 171.0; HRMS (ESI) cacld for $C_{20}H_{20}NO_3$ [M + H]$^+$ 322.1438, found 322.1442; IR (ν/cm^{-1}): 1742, 1113, 784, 694.



3,4-Diphenyl-5-(pyrrolidin-1-yl)furan-2(5*H*)-one (3f). The general procedure (GP-1) was followed using **1a** (20.6 mg, 0.100 mmol) and **2f** (0.5 mL, 5.2 mmol). Purification by preparative TLC (hexane:AcOEt = 3:1) yielded **3f** (3.6 mg, 0.012 mmol, 12%) as a yellow solid. According to GP-2, **3f** (24.1/26.0 mg, 0.079/0.085 mmol, 92/96%) was obtained by reacting **1a** (17.6/18.2 mg, 0.085/0.088 mmol) with **2f** (99.3/200.8 mg, 1.002/2.026 mmol). Mp 144–145 °C; ^{1}H NMR (301 MHz, $CDCl_3$) δ 1.67–1.82 (m, 4H), 2.76–2.96 (m, 4H), 6.26 (s, 1H), 7.22–7.42 (m, 10H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 24.5, 46.3, 94.5, 128.46, 128.52, 128.6, 128.7, 129.1, 129.4, 130.0, 130.2, 131.1, 154.8, 171.7; HRMS (ESI) cacld for $C_{20}H_{20}NO_2$ [M + H]$^+$ 306.1489, found 306.1489; IR (ν/cm^{-1}):1737, 1315, 1150, 787, 696.

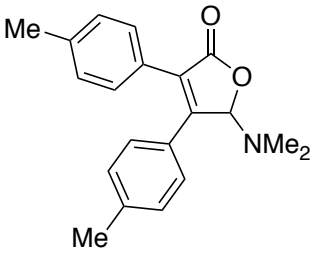


5-(Methylamino)-3,4-diphenylfuran-2(5*H*)-one (3g). The general procedure (GP-2) was followed using **1a** (20.6/17.1 mg, 0.100/0.083 mmol) and **2g** (58.6/118.4 mg, 0.992/2.004 mmol). Purification by preparative TLC (hexane:AcOEt = 3:1) yielded **3g** (8.3/7.5 mg, 0.031/0.028 mmol, 31/34%) as a yellow solid. Mp 68–70 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 3.16 (s, 3H), 6.96 (s, 1H), 7.10–7.14 (m, 2H), 7.18–7.29 (m, 5H), 7.32–7.36 (m, 3H), 9.30 (s, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 27.1, 128.4, 128.8, 128.9, 129.0, 129.3, 129.7, 133.9, 134.2, 135.4, 135.9, 164.1, 172.7; HRMS (ESI) cacld for $C_{17}H_{15}NNaO_2$ $[M + Na]^+$ 288.0995, found 288.0995; IR (ν/cm^{-1}): 1657, 1272, 691.

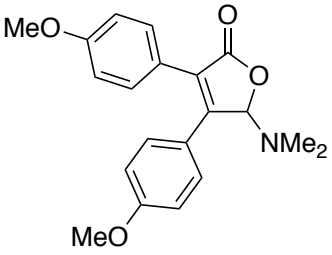


5-(Dibenzylamino)-3,4-diphenylfuran-2(5*H*)-one (3h). The general procedure (GP-2) was followed using **1a** (19.3/18.9 mg, 0.094/0.092 mmol) and **2h** (225.2/450.0 mg, 1.000/1.997 mmol). Purification by column chromatography on silica gel (hexane:AcOEt = 10:1) followed by preparative TLC (hexane:AcOEt = 5:1/3:1) yielded **3h** (20.3/27.0 mg, 0.047/0.063 mmol, 50/68%) as a yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.48 (s, 2H), 4.61 (s, 2H), 6.77 (s, 1H), 6.95–7.07 (m, 4H), 7.12–7.22 (m, 5H), 7.26–7.34 (m, 9H), 7.35–7.41 (m, 2H); ^{13}C NMR (126 MHz, acetone-d_6) δ 52.3, 93.5, 126.4, 127.6, 128.5, 128.6, 128.8, 129.3, 129.4, 129.8, 129.9, 130.1, 130.8, 131.5, 138.1, 155.9, 170.5; HRMS (ESI) cacld for $C_{30}H_{26}NO_2$ $[M + H]^+$ 432.1958, found 432.1959;

IR (ν/cm^{-1}): 1628, 748, 693.

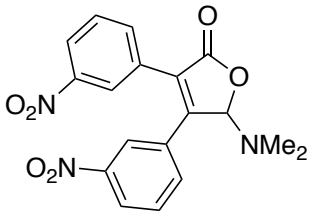


5-(Dimethylamino)-3,4-bis(4-methylphenyl)furan-2(5*H*)-one (3i). The general procedure (GP-1) was followed using **1b** (23.3 mg, 0.099 mmol) and **2a** (0.5 mL, 6.5 mmol). Purification by preparative TLC (hexane:AcOEt = 3:1 × 2) yielded **3i** (24.8 mg, 0.081 mmol, 81%) as a colorless oil. According to GP-2, **3i** (21.8/23.4 mg, 0.071/0.076 mmol, 74/81%) was obtained by reacting **1b** (22.6/22.1 mg, 0.096/0.094 mmol) with **2a** (75.6/147.5 mg, 1.034/2.018 mmol). ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.35 (s, 3H), 2.36 (s, 3H), 2.44 (s, 6H), 6.01 (s, 1H), 7.12 (d, J = 8.0 Hz, 2H), 7.16 (d, J = 8.0 Hz, 2H), 7.31 (d, J = 8.0 Hz, 2H+2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 21.4, 21.5, 38.8, 97.9, 127.3, 128.2, 128.6, 128.8, 129.25, 129.28, 138.7, 140.3, 153.6, 171.7; HRMS (ESI) cacld for $C_{20}H_{22}NO_2$ $[M + H]^+$ 308.1645, found 308.1645; IR (ν/cm^{-1}): 1739, 1093, 965, 754.

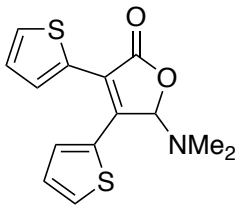


5-(Dimethylamino)-3,4-bis(4-methoxyphenyl)furan-2(5*H*)-one (3j). The general procedure (GP-1) was followed using **1c** (26.6 mg, 0.100 mmol) and **2a** (0.5 mL, 6.5 mmol). Purification by preparative TLC (hexane:AcOEt = 1:1 × 2) yielded **3j** (26.0 mg, 0.077 mmol, 77%) as a yellow oil. According to GP-2, **3j** (12.0/21.1 mg, 0.035/0.062 mmol, 36/67%) was obtained by reacting **1c** (26.3/24.8 mg, 0.099/0.093 mmol) with **2a** (73.8/146.7 mg, 1.010/2.007 mmol). ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.44 (s, 6H), 3.81 (s, 3H), 3.82 (s, 3H), 5.97 (s, 1H),

6.82 (d, J = 9.0 Hz, 2H), 6.89 (d, J = 8.5 Hz, 2H), 7.37 (d, J = 9.5 Hz, 2H), 7.42 (d, J = 8.5 Hz, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 24.9, 38.7, 55.2, 97.8, 113.9, 114.0, 122.6, 123.4, 127.2, 130.3, 130.7, 152.5, 159.8, 160.8, 172.0; HRMS (ESI) cacld for $C_{20}H_{22}NO_4$ $[M + H]^+$ 340.1543, found 340.1544; IR (ν/cm^{-1}): 1737, 1249, 833, 754.

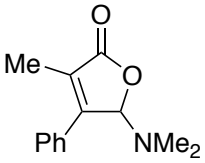


5-(Dimethylamino)-3,4-bis(3-nitrophenyl)furan-2(5*H*)-one (3k). The general procedure (GP-1) was followed using **1d** (29.6 mg, 0.100 mmol) and **2a** (0.5 mL, 6.5 mmol). Purification by preparative TLC (hexane:AcOEt = 1:1) yielded **3k** (25.6 mg, 0.069 mmol, 69%) as a yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.50 (s, 6H), 6.15 (s, 1H), 7.54 (t, J = 8.0 Hz, 1H), 7.59 (t, J = 8.0 Hz, 1H), 7.64–7.68 (m, 1H), 7.73–7.77 (m, 1H), 8.25–8.29 (m, 3H), 8.31 (t, J = 1.7 Hz, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 39.0, 98.2, 123.6, 124.38, 124.41, 125.3, 129.8, 130.11, 130.13, 130.7, 131.6, 134.3, 135.3, 148.4, 148.5, 153.3, 169.6; HRMS (ESI) cacld for $C_{18}H_{16}N_3O_6$ $[M + H]^+$ 370.1034, found 370.1035; IR (ν/cm^{-1}): 1744, 1525, 1342, 684.

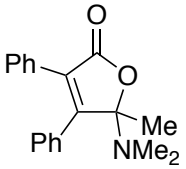


5-(Dimethylamino)-3,4-di(2-thienyl)furan-2(5*H*)-one (3l). The general procedure (GP-1) was followed using **1e** (21.8 mg, 0.100 mmol) and **2a** (0.5 mL, 6.5 mmol). Purification by preparative TLC (hexane:AcOEt = 3:1 × 3) yielded **3l** (25.3 mg, 0.087 mmol, 87%) as a yellow solid. According to GP-2, **3l** (24.8/26.8 mg, 0.085/0.092 mmol, 89/93%) was obtained by reacting **1e** (20.9/21.5 mg, 0.096/0.098 mmol) with **2a** (73.3/148.3 mg, 1.003/2.029 mmol). Mp

136–138 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.51 (s, 6H), 5.86 (s, 1H), 7.07 (dd, J = 5.5, 4.0 Hz, 1H), 7.12 (t, J = 4.3 Hz, 1H), 7.47–7.52 (m, 3H), 7.68–7.72 (m, 1H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 38.7, 97.8, 120.5, 127.2, 127.5, 128.3, 129.3, 130.2, 130.9, 131.0, 132.1, 147.3, 170.8; HRMS (ESI) cacld for $C_{14}H_{14}NO_2S_2$ $[M + H]^+$ 292.0460, found 292.0462; IR (ν/cm^{-1}): 1725, 1097, 933, 713.

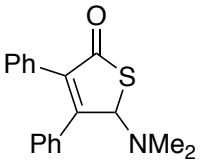


5-(Dimethylamino)-3-methyl-4-phenylfuran-2(5*H*)-one (3m). The general procedure (GP-1) was followed using **1f** (14.6 mg, 0.101 mmol) and **2a** (0.5 mL, 6.5 mmol). Purification by preparative TLC (hexane:AcOEt = 3:1) yielded **3m** (17.0 mg, 0.078 mmol, 77%) as a white solid. According to GP-2, **3m** (18.7/16.3 mg, 0.086/0.075 mmol, 87/79%) was obtained by reacting **1f** (14.2/13.7 mg, 0.098/0.095 mmol) with **2a** (77.0/149.7 mg, 1.053/2.048 mmol). Mp 95–98 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.12 (d, J = 1.0 Hz, 3H), 2.38 (s, 6H), 5.96 (q, J = 2.0 Hz, 1H), 7.39–7.49 (m, 3H), 7.50–7.54 (m, 2H); ^{13}C NMR (75.6 MHz, $CDCl_3$) δ 10.5, 38.7, 98.3, 126.9, 128.2, 128.7, 129.8, 131.4, 153.4, 173.3; HRMS (ESI) cacld for $C_{13}H_{16}NO_2$ $[M + H]^+$ 218.1176, found 218.1179; IR (ν/cm^{-1}): 1726, 1042, 907, 766, 701.



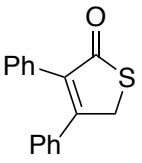
5-(Dimethylamino)-5-methyl-3,4-diphenylfuran-2(5*H*)-one (3n). The general procedure (GP-1) was followed using **1a** (20.7 mg, 0.100 mmol) and **2i** (0.5 mL, 5.4 mmol). Purification by preparative TLC (hexane:AcOEt = 3:1) yielded **3n** (9.7 mg, 0.033 mmol, 33%) as a yellow solid. According to GP-2, **3n** (5.7/8.0 mg, 0.019/0.027 mmol, 21/30%) was obtained

by reacting **1a** (19.0/18.7 mg, 0.092/0.091 mmol) with **2i** (88.2/174.9 mg, 1.012/2.008 mmol). Mp 133–136 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.66 (s, 3H), 2.56 (s, 6H), 7.27–7.40 (m, 8H), 7.55–7.58 (m, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 23.6, 38.2, 102.6, 127.7, 128.51, 128.53, 128.7, 129.2, 129.5, 129.9, 130.4, 130.9, 159.1, 171.0; HRMS (ESI) cacld for $C_{19}H_{20}NO_2$ [M + H]$^+$ 294.1489, found 294.1491; IR (ν/cm^{-1}): 1738, 743, 692.

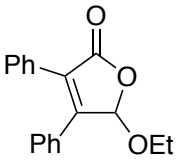


5-(Dimethylamino)-3,4-diphenylthiophen-2(5*H*)-one (3o). A Schlenk tube was charged with silver(I) triflate (2.7 mg, 0.011 mmol) and **1a** (20.6 mg, 0.100 mmol), and the tube was evacuated and backfilled with nitrogen. Chlorobenzene (0.50 mL) and *N*,*N*-dimethylthioformamide (**2j**, 17.8 mg, 0.200 mmol) were added successively via a syringe through the septum, and the mixture was heated at 130 °C for 4 h. After cooling to room temperature, the reaction mixture was filtered through a plug of silica gel eluting with hexane–AcOEt (1:1), and the filtrate was concentrated. The residue was purified by preparative TLC (hexane:AcOEt = 3:1) to afford **3o** (7.7 mg, 0.026 mmol, 26%) as an orange oil and **4** (6.8 mg, 0.027 mmol, 27%) as a red solid.

3o: ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.28 (s, 6H), 6.22 (s, 1H), 7.10–7.15 (m, 2H), 7.16–7.20 (m, 2H), 7.23–7.31 (m, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 40.9, 81.1, 128.17, 128.20, 128.3, 129.0, 129.2, 130.2, 131.4, 134.4, 141.1, 160.8, 196.6; HRMS (ESI) cacld for $C_{18}H_{18}NOS$ [M + H]$^+$ 296.1104, found 296.1104; IR (ν/cm^{-1}): 1671, 1650, 690, 617.



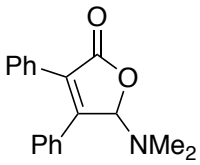
3,4-Diphenylthiophen-2(5*H*)-one (4). Mp 111–115 °C; ^{1}H NMR (500 MHz, $CDCl_3$) δ 4.36 (s, 2H), 7.15–7.23 (m, 4H), 7.26–7.35 (m, 6H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 37.1, 128.0, 128.3, 128.5, 128.7, 129.6, 129.9, 131.8, 135.3, 139.3, 160.2, 198.5; HRMS (ESI) cacld for $C_{16}H_{12}NaOS$ $[M + Na]^+$ 275.0501, found 275.0501; IR (ν/cm^{-1}): 1671, 748, 691.



5-Ethoxy-3,4-diphenylfuran-2(5*H*)-one (5). A Schlenk tube was charged with **3a** (27.8 mg, 0.100 mmol), and the tube was evacuated and backfilled with nitrogen. Ethanol (1.0 mL) and aqueous 35% HCl solution (0.2 mL) were added successively via a syringe through the septum, and the mixture was heated for 4 h at 100 °C. After cooling to room temperature, the reaction mixture was washed with saturated $NaHCO_3$ aqueous solution (× 3), and the aqueous layer was extracted with AcOEt (× 2). The combined organic layer was dried over Na_2SO_4, and concentrated to dryness. The residue was purified by preparative TLC (hexane:AcOEt = 3:1) to afford the title compound (22.6 mg, 0.081 mmol, 81%). ^{1}H NMR (399 MHz, $CDCl_3$) δ 1.31 (t, J = 7.2 Hz, 3H), 3.87 (dq, J = 9.4, 7.1 Hz, 1H), 3.99 (dq, J = 9.6, 7.1 Hz, 1H), 6.23 (s, 1H), 7.29–7.45 (m, 10H); ^{13}C NMR (75.6 MHz, $CDCl_3$) δ 15.1, 65.6, 101.7, 128.5, 128.6, 128.7, 128.8, 129.1, 129.4, 129.5, 130.3, 130.4, 153.3, 170.6; HRMS (ESI) cacld for $C_{18}H_{16}NaO_3$ $[M + Na]^+$ 303.0992, found 303.0991. The spectral data matched those reported in the literature.[3]

3 T. Mise, P. Hong and H. Yamazaki, *Chem. Lett.*, 1981, 993–996.

3a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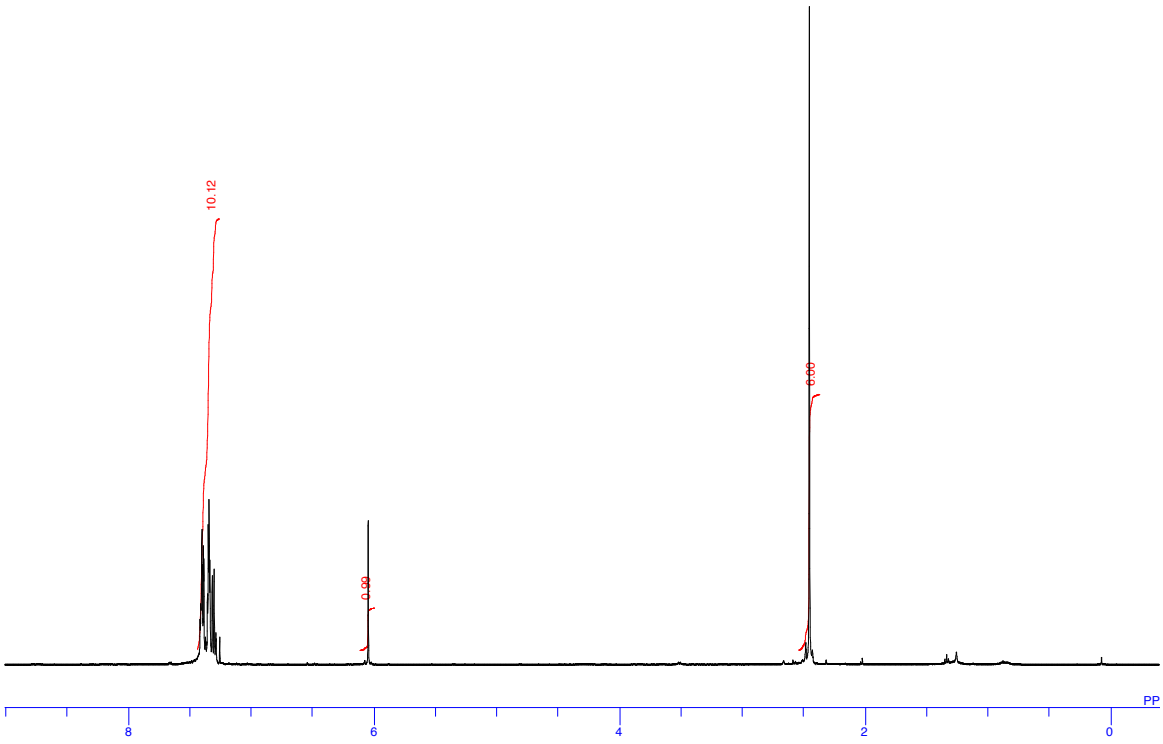
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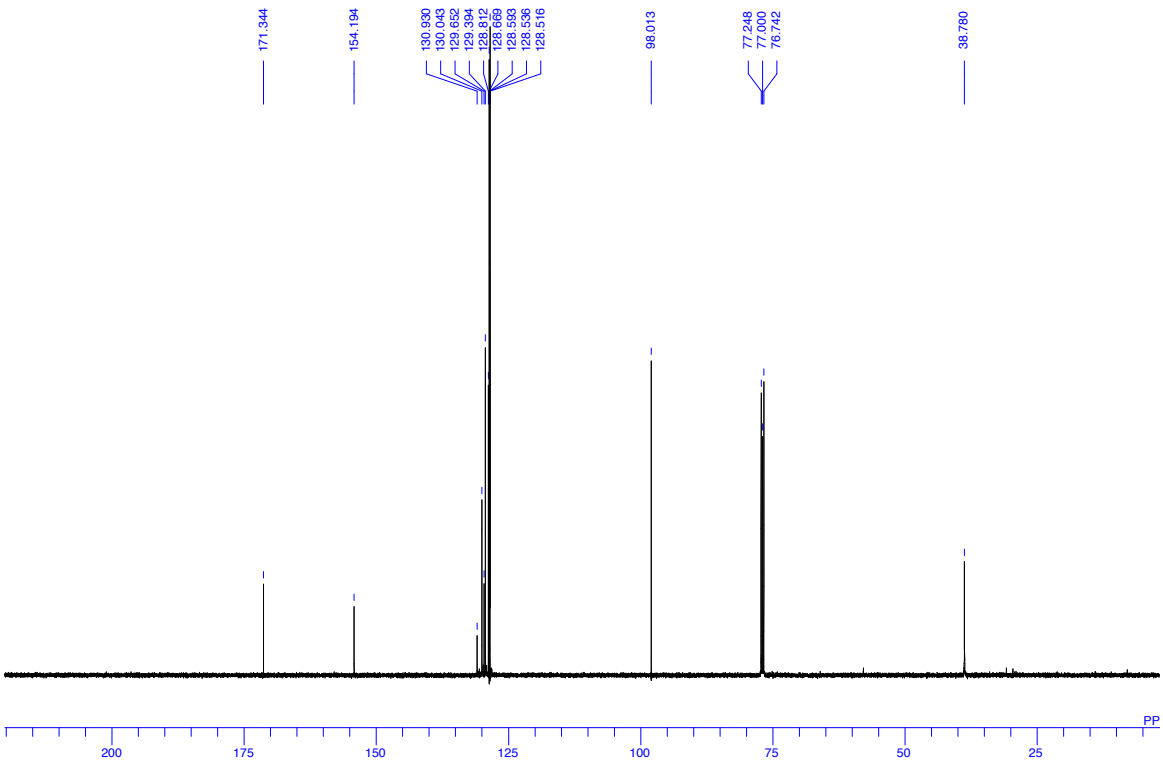
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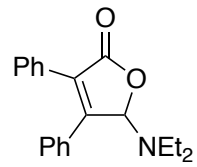
75
50
25

3b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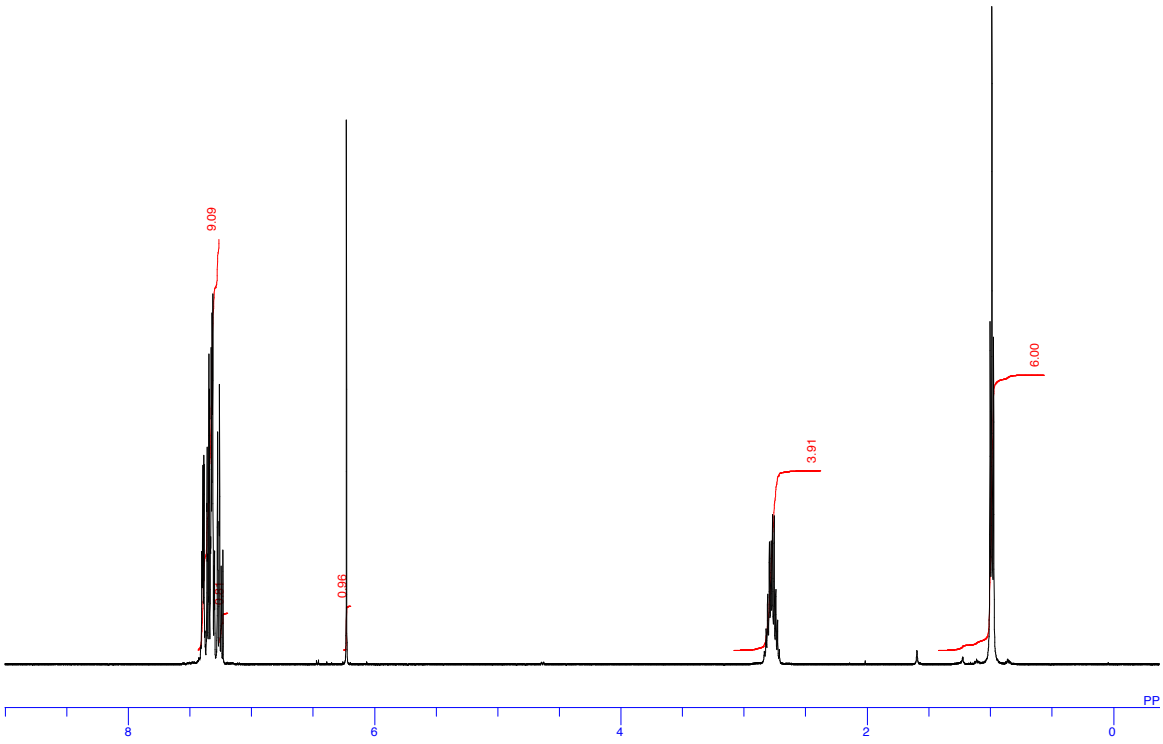
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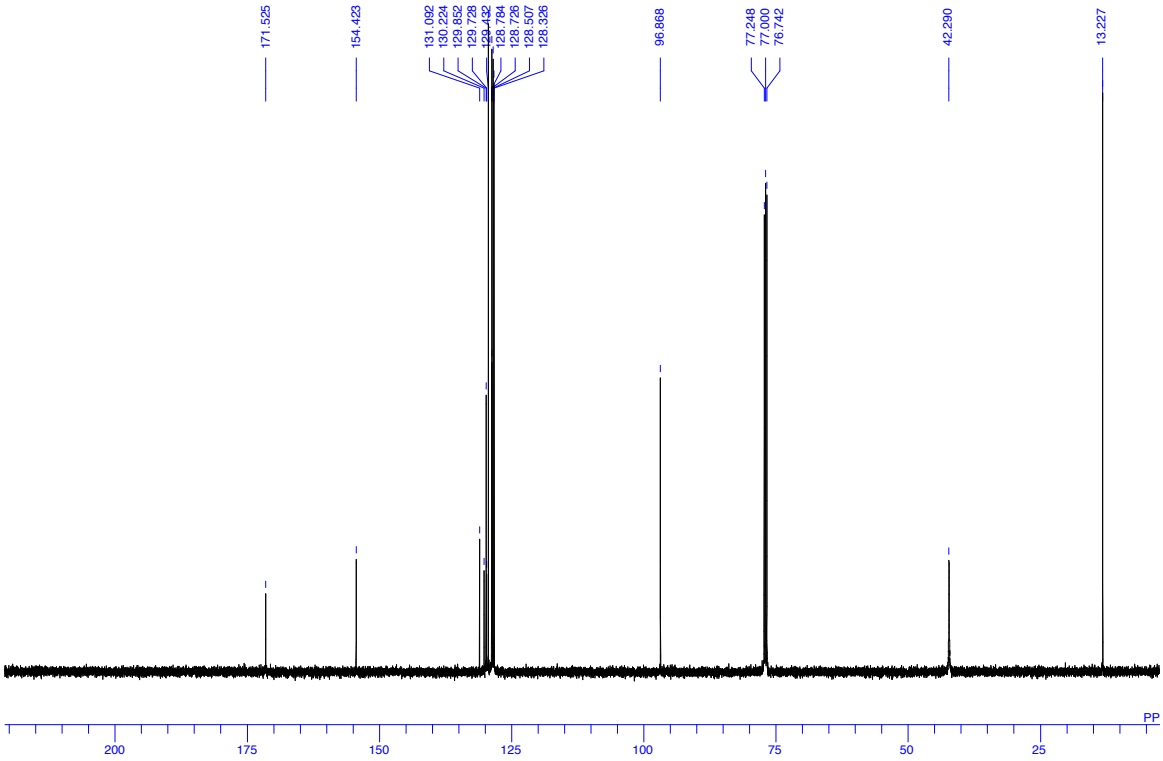
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75
50
25

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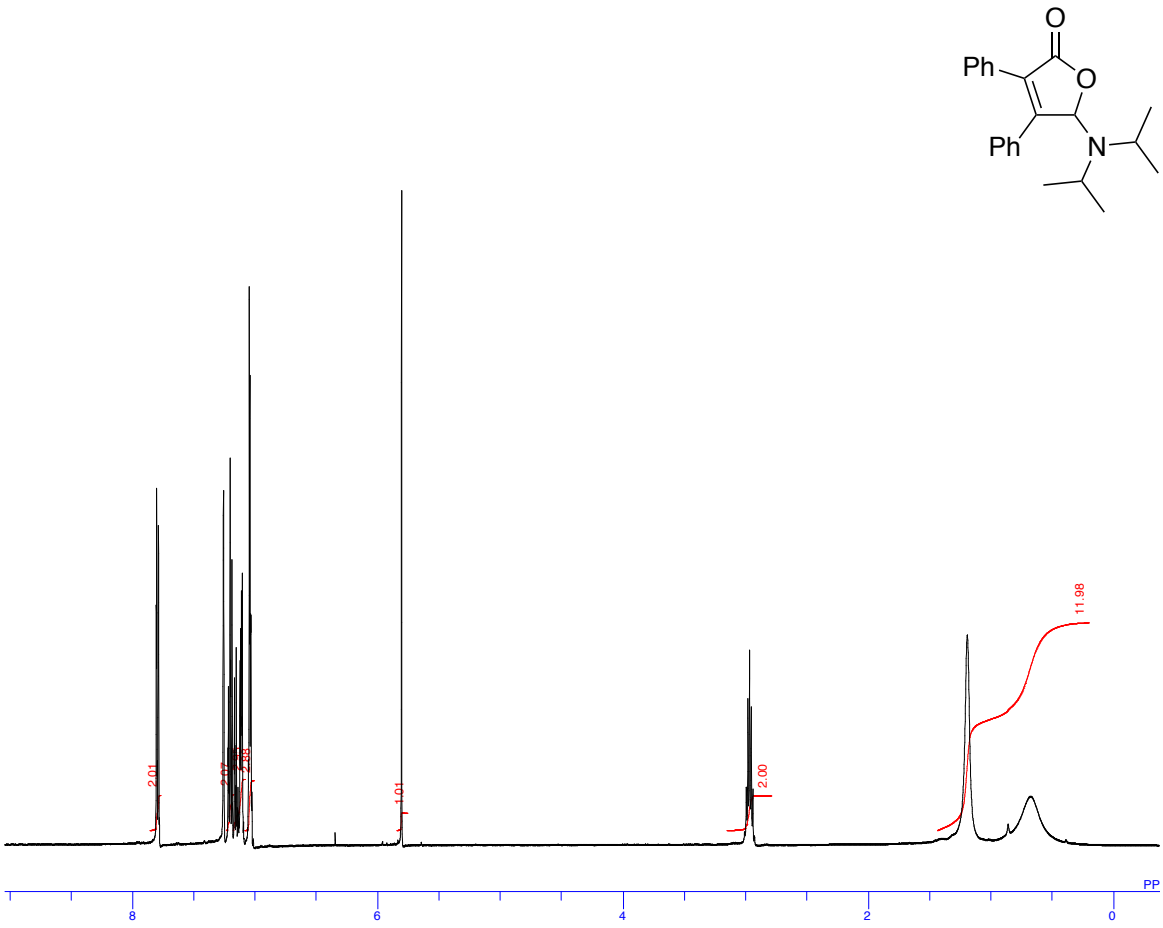
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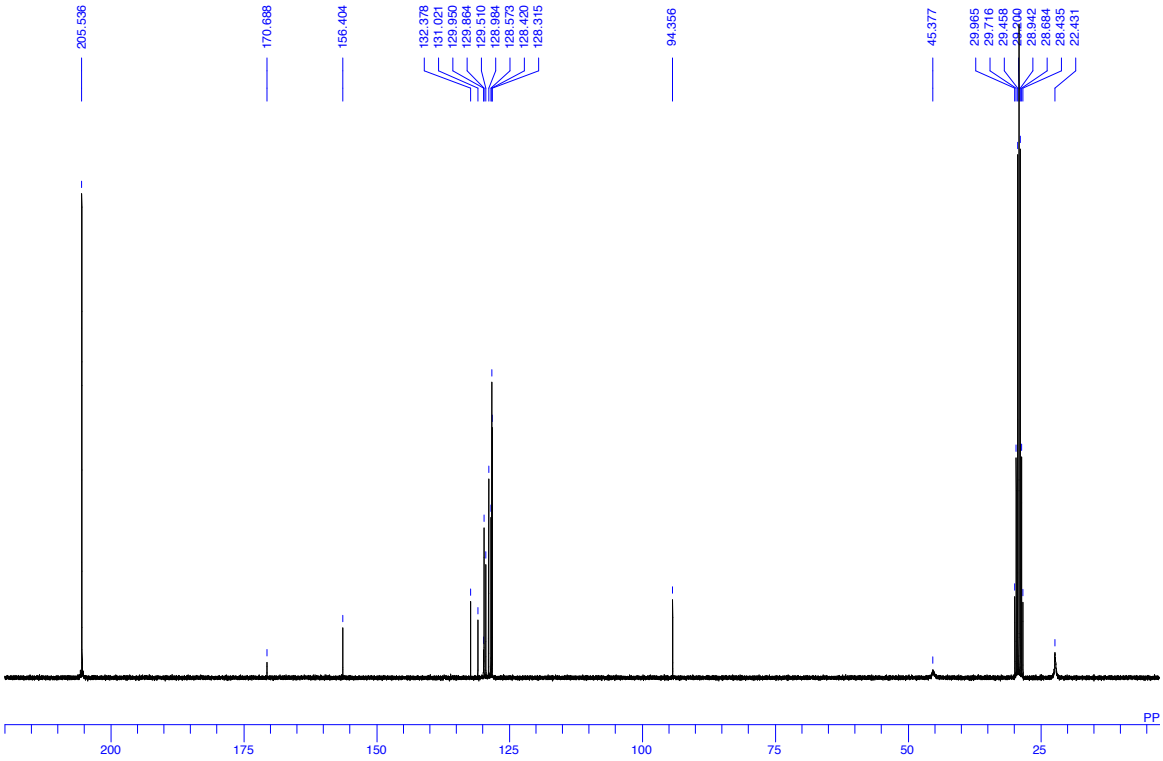
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29.716
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75
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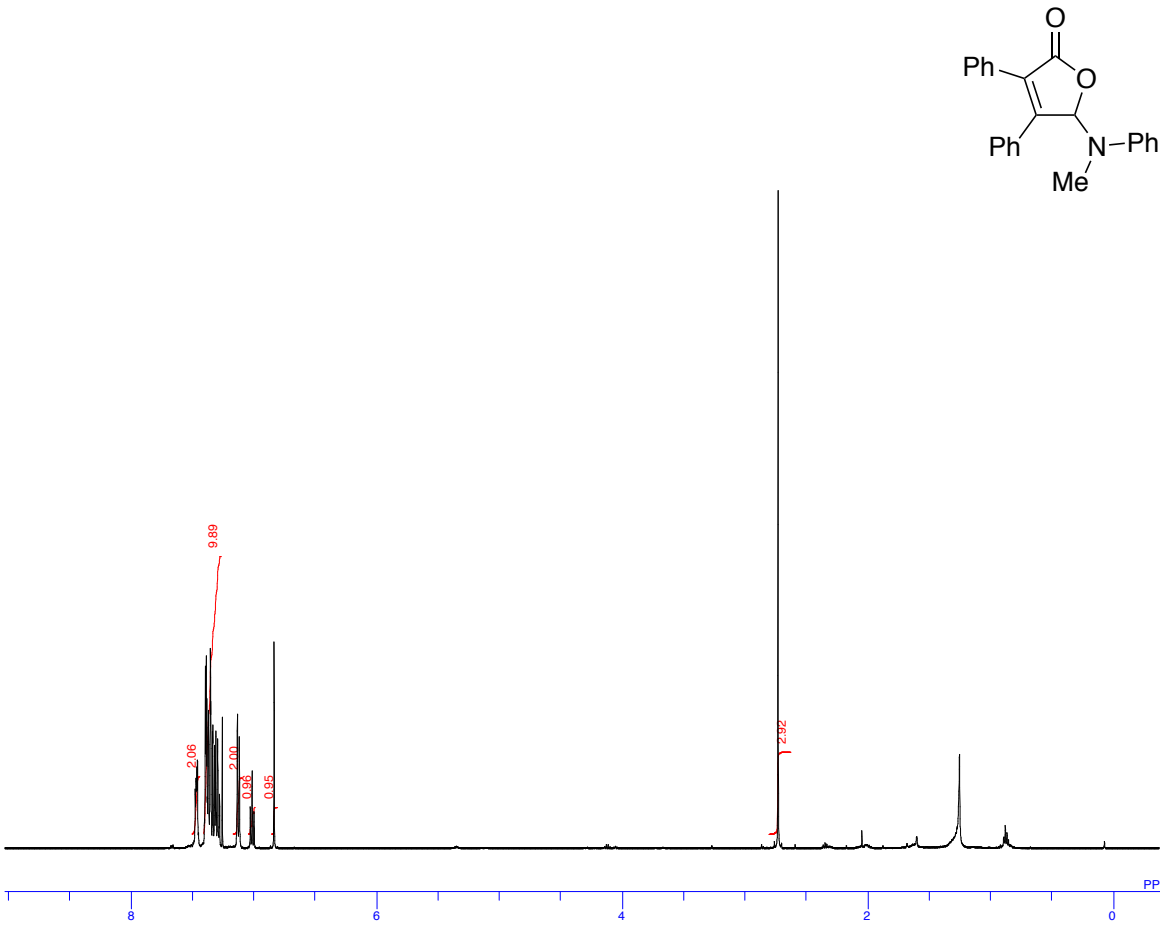
25

3d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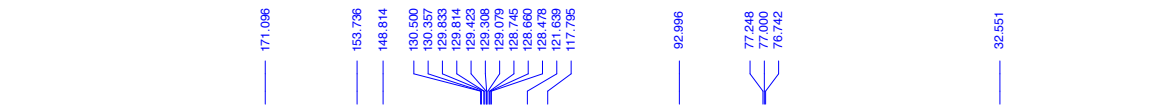
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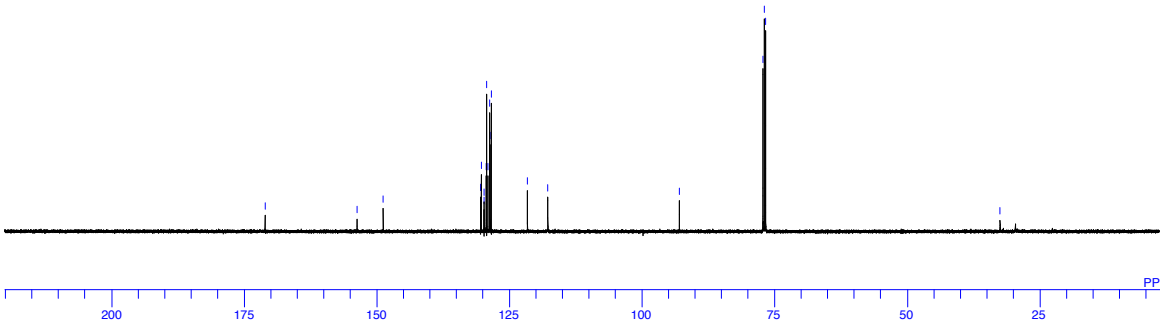
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50
25

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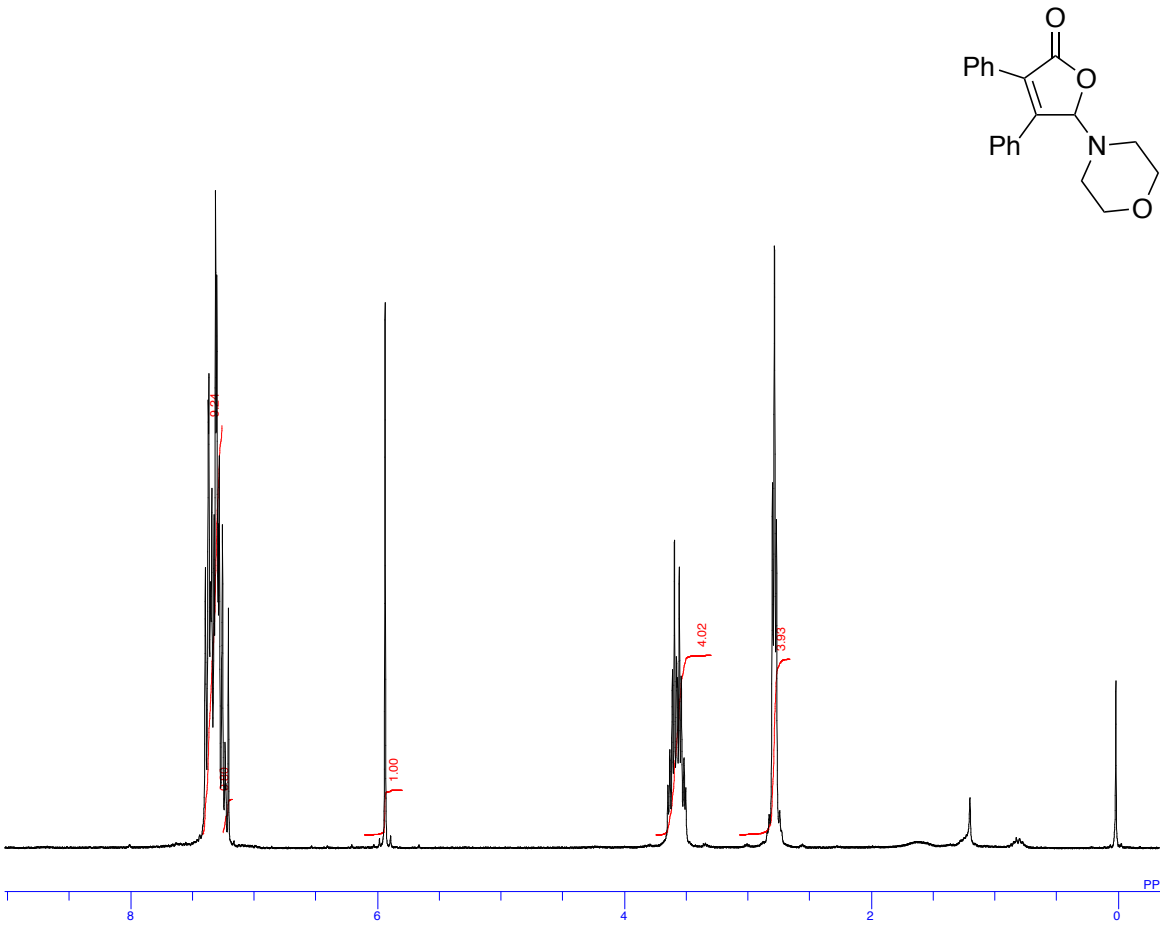
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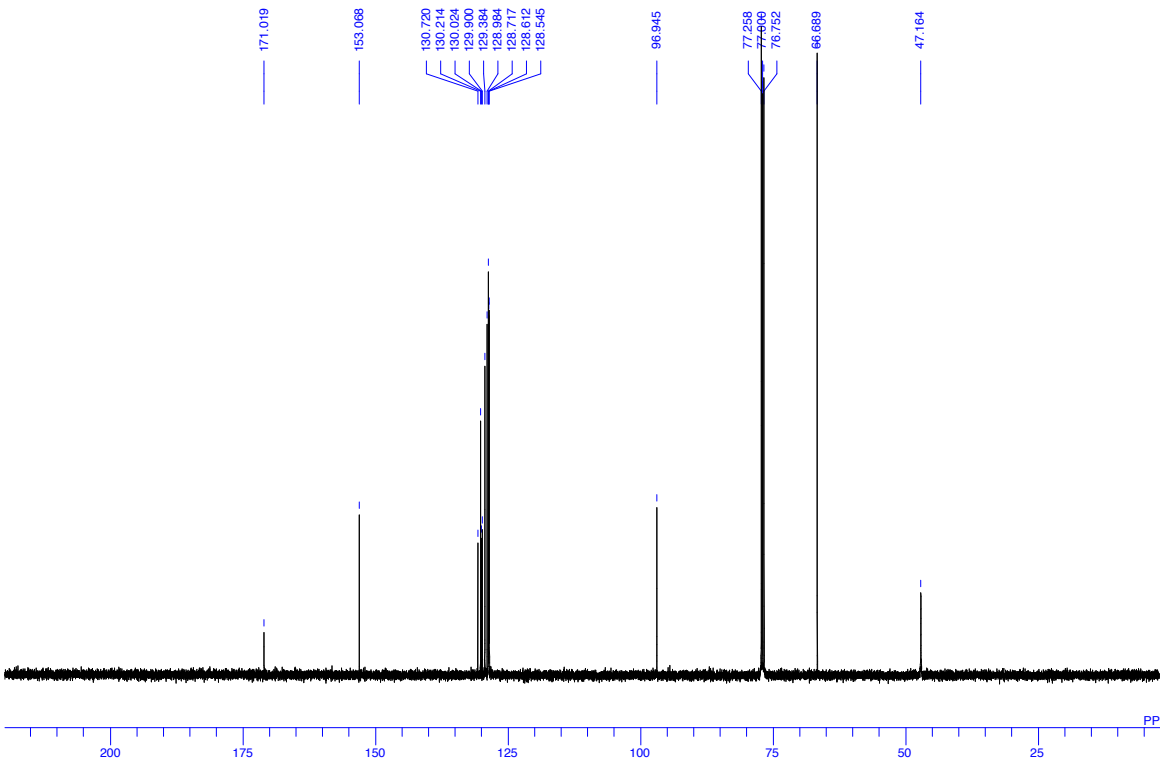
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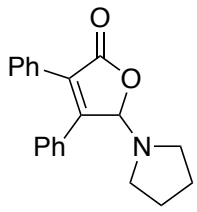
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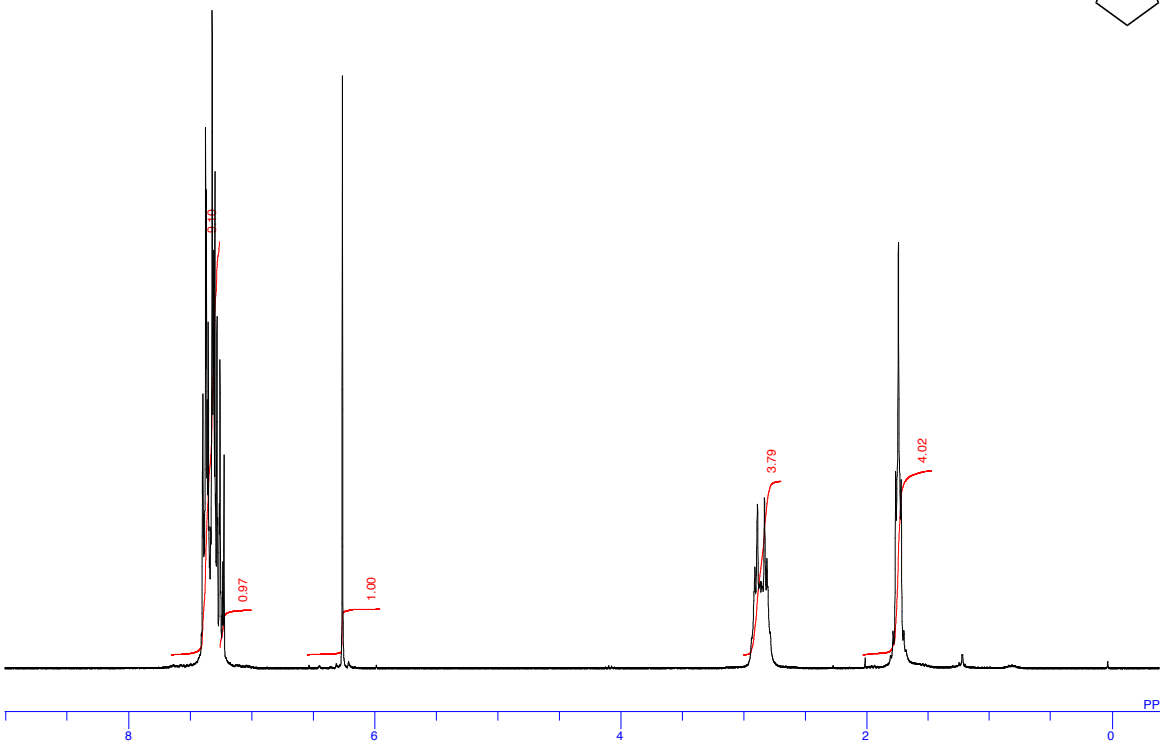
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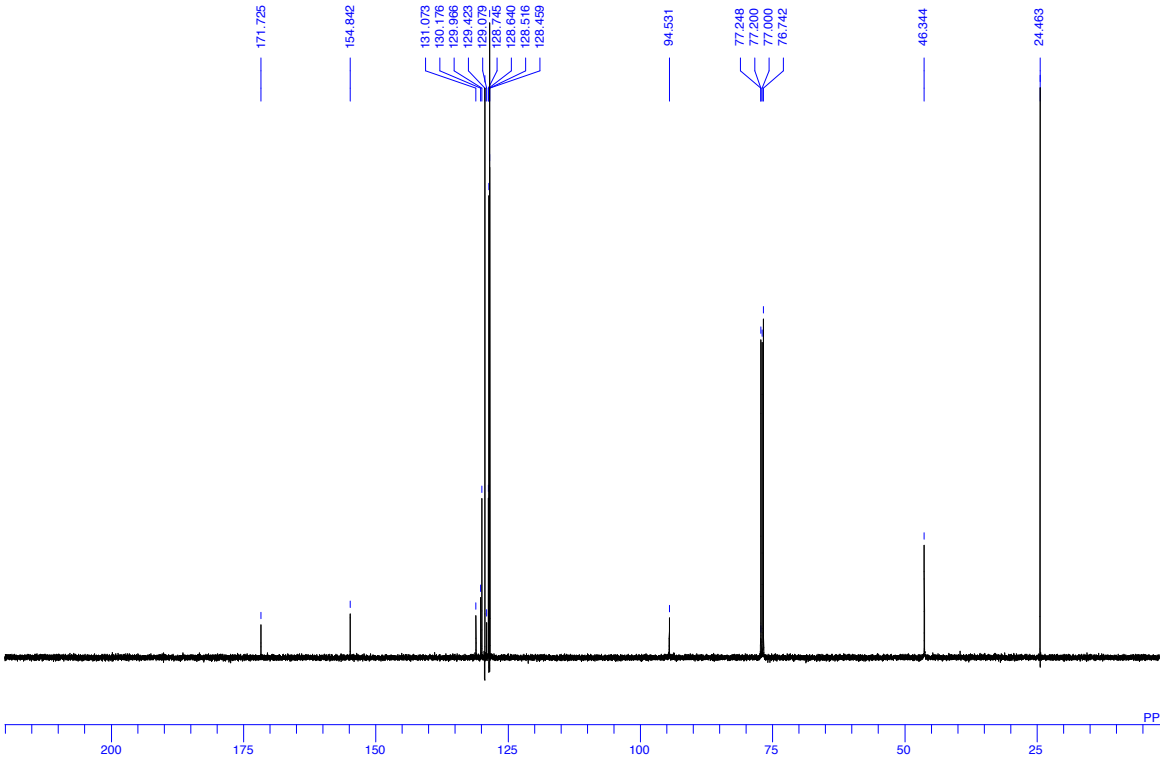
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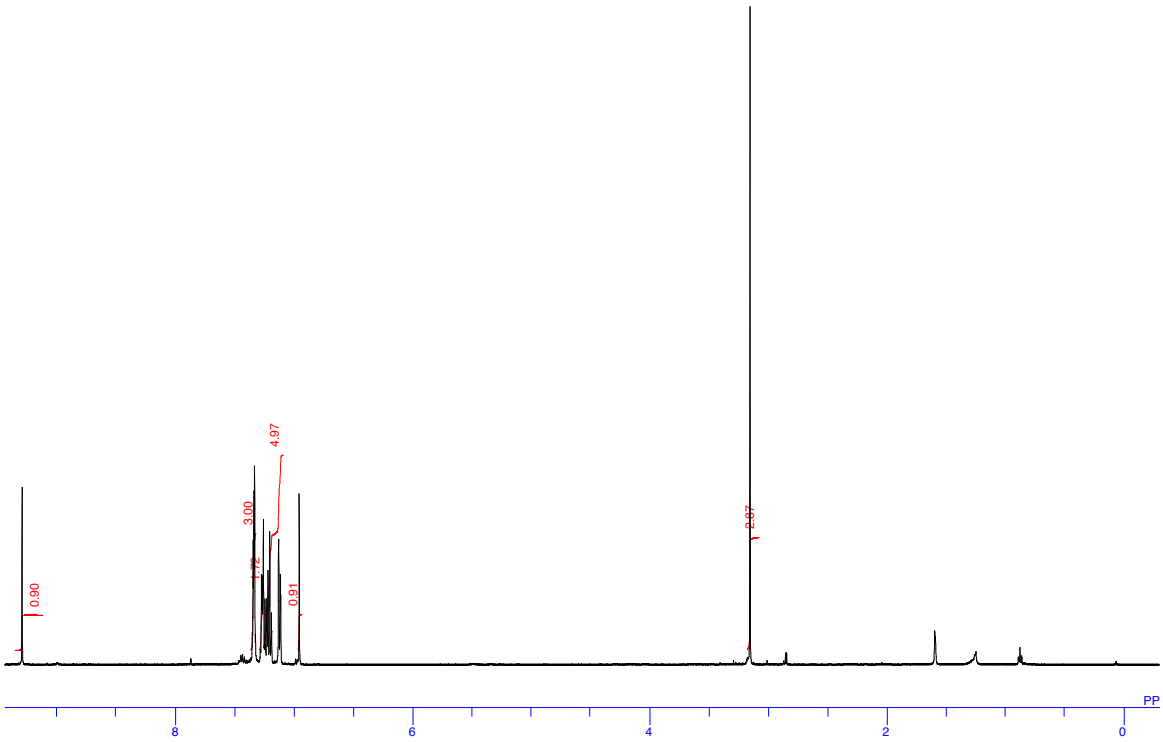
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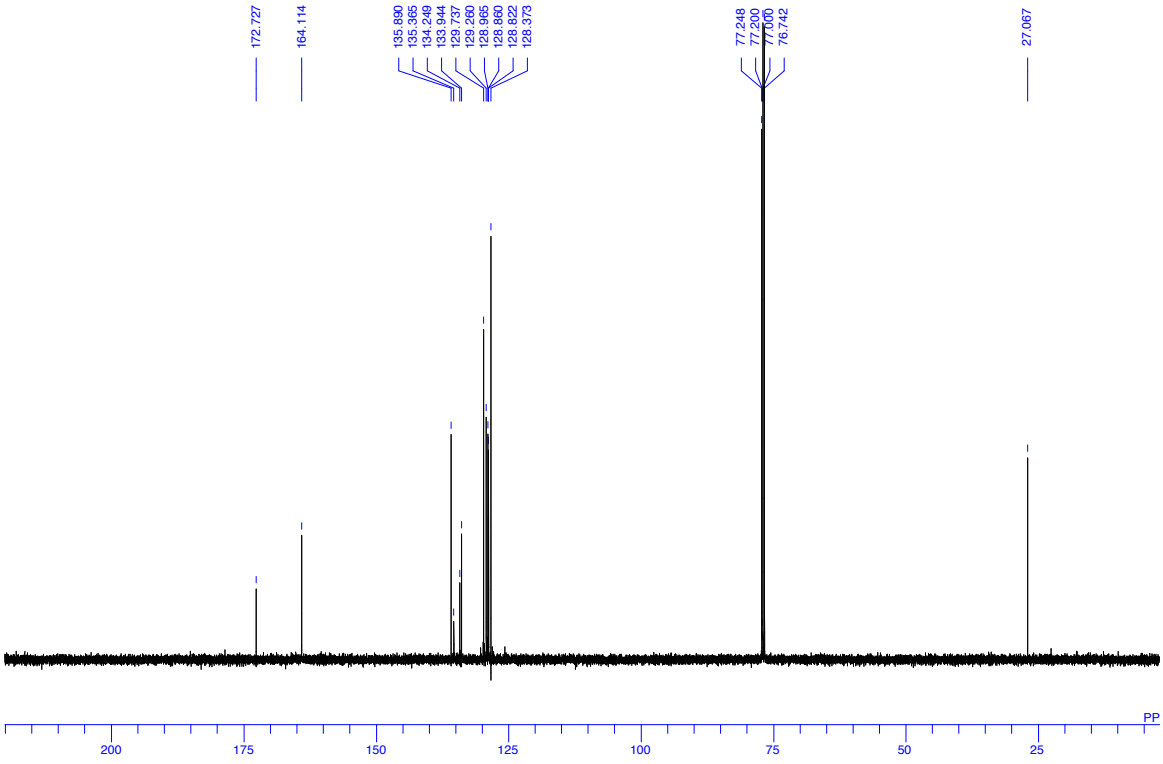
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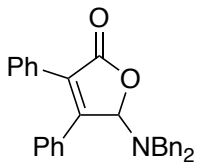
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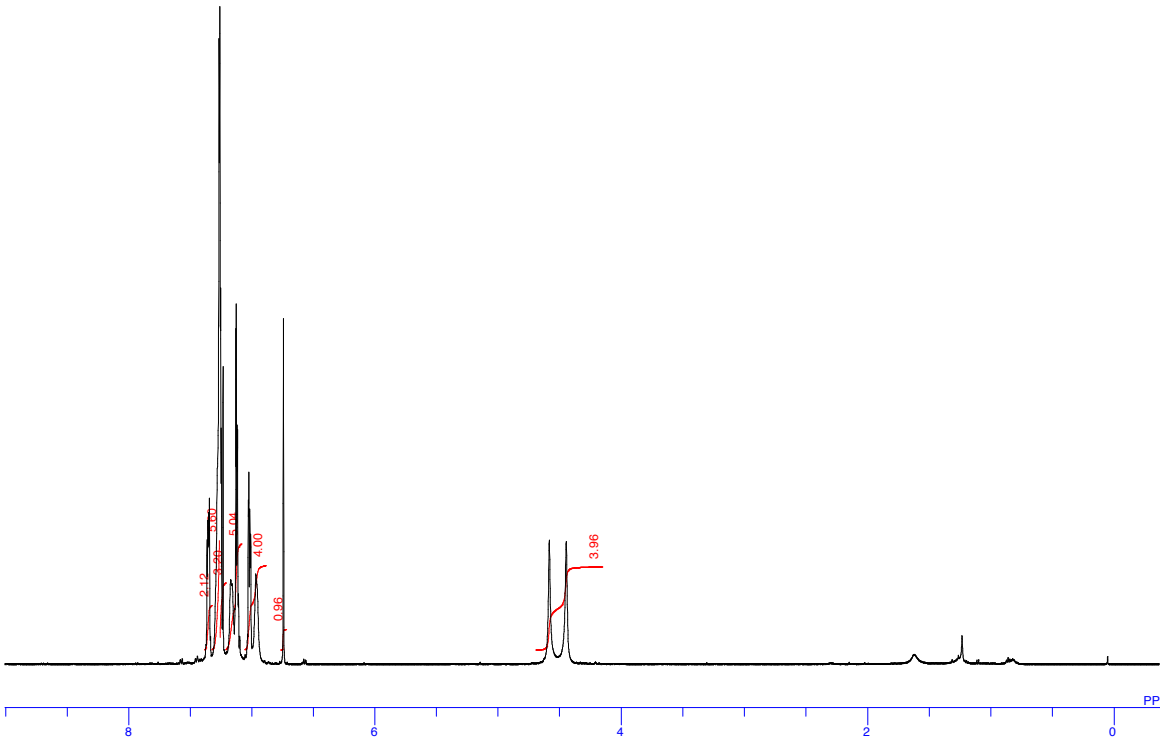
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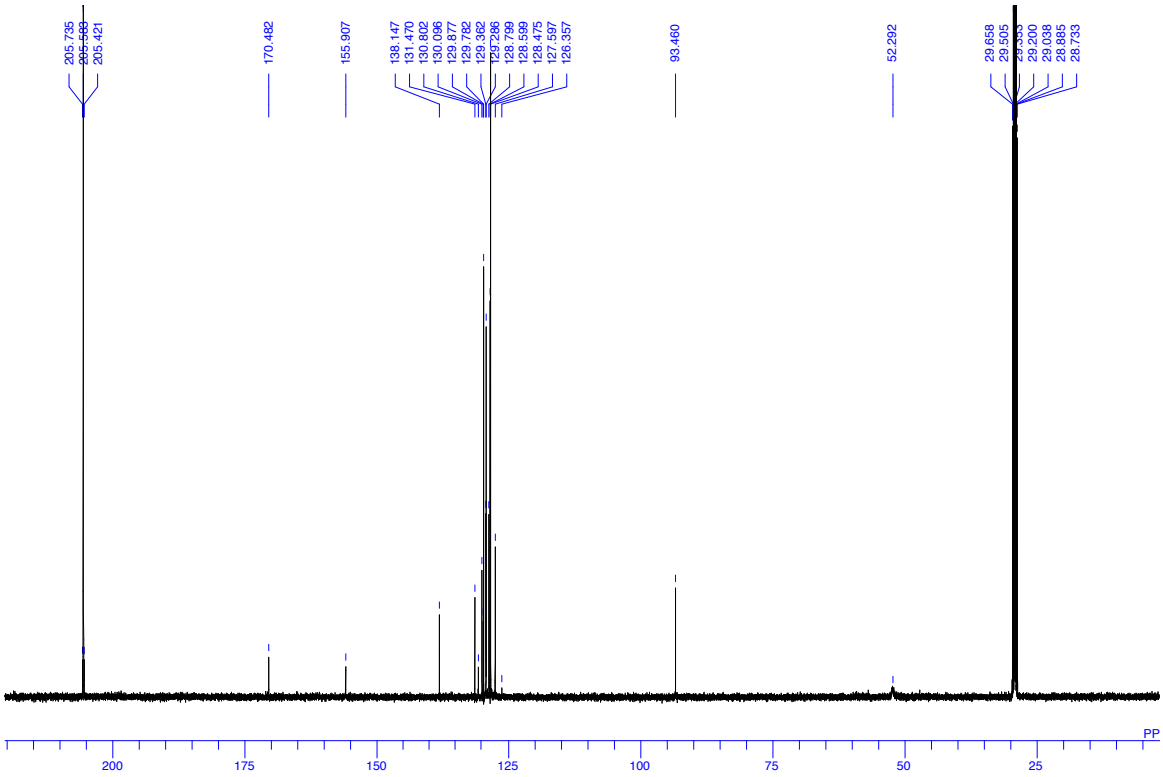
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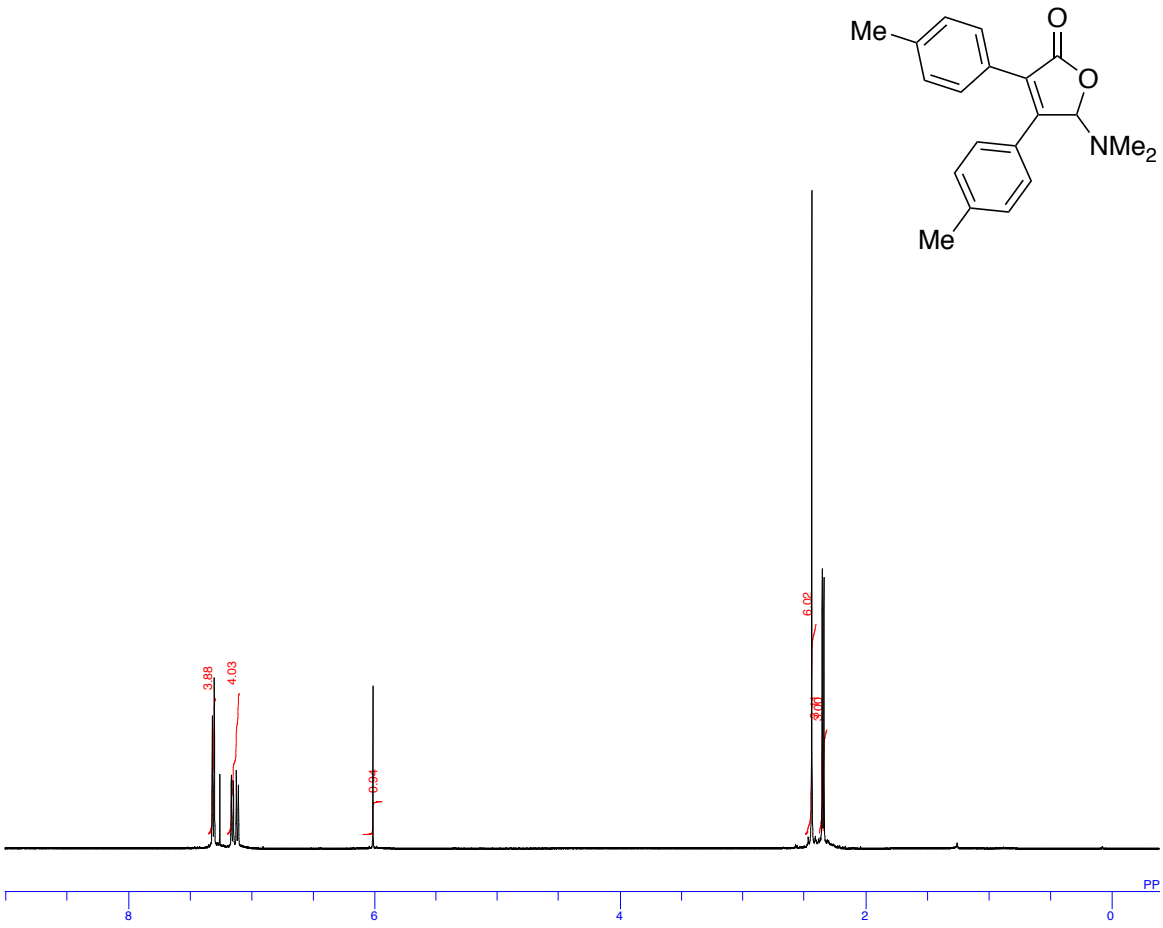
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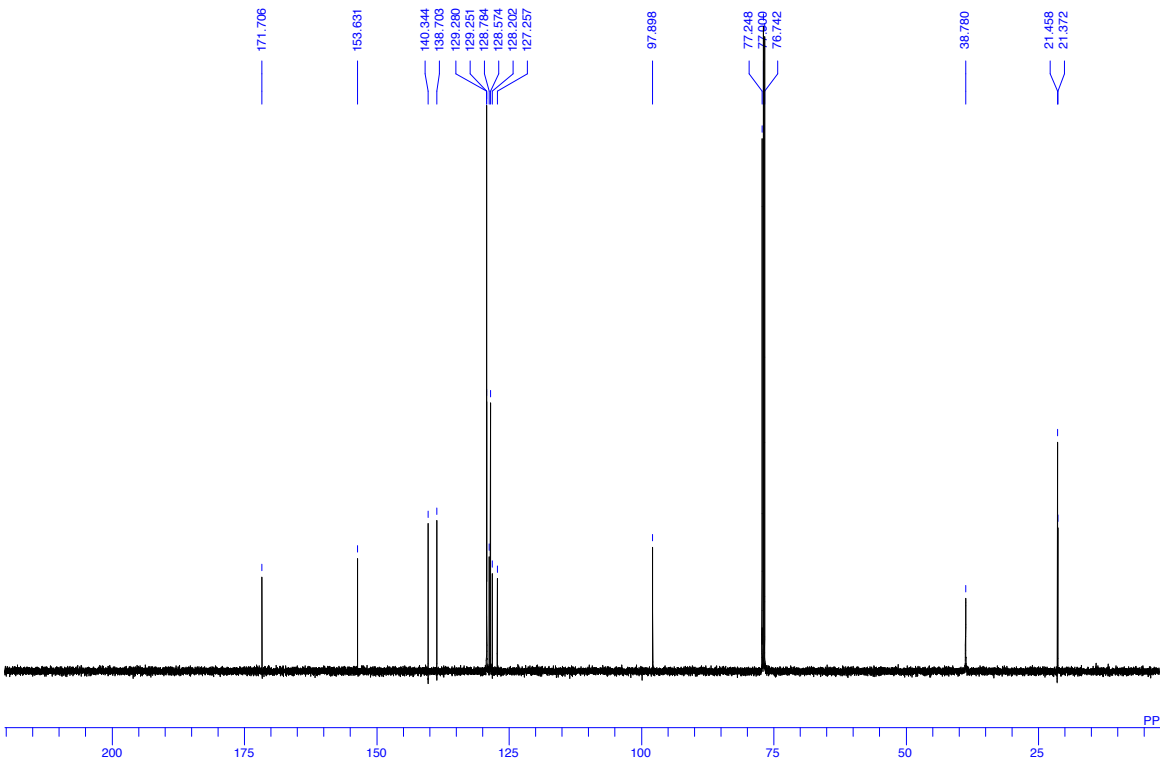
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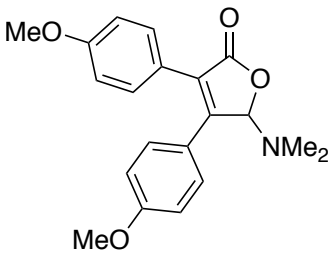
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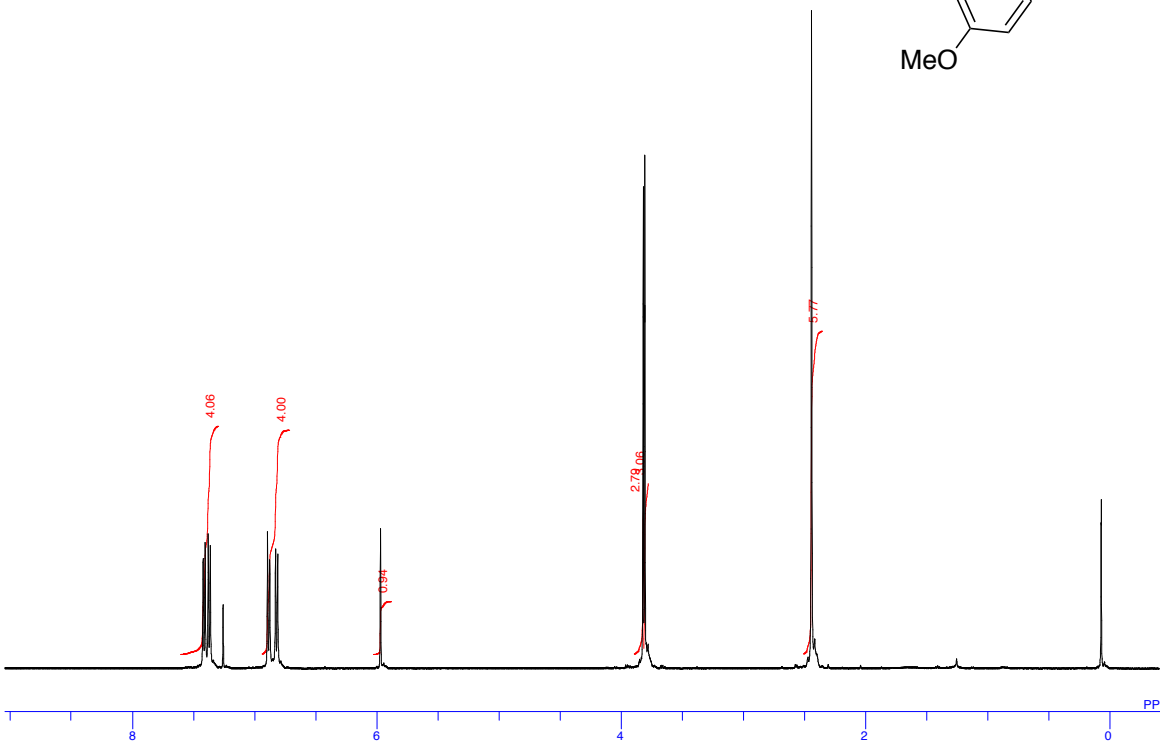
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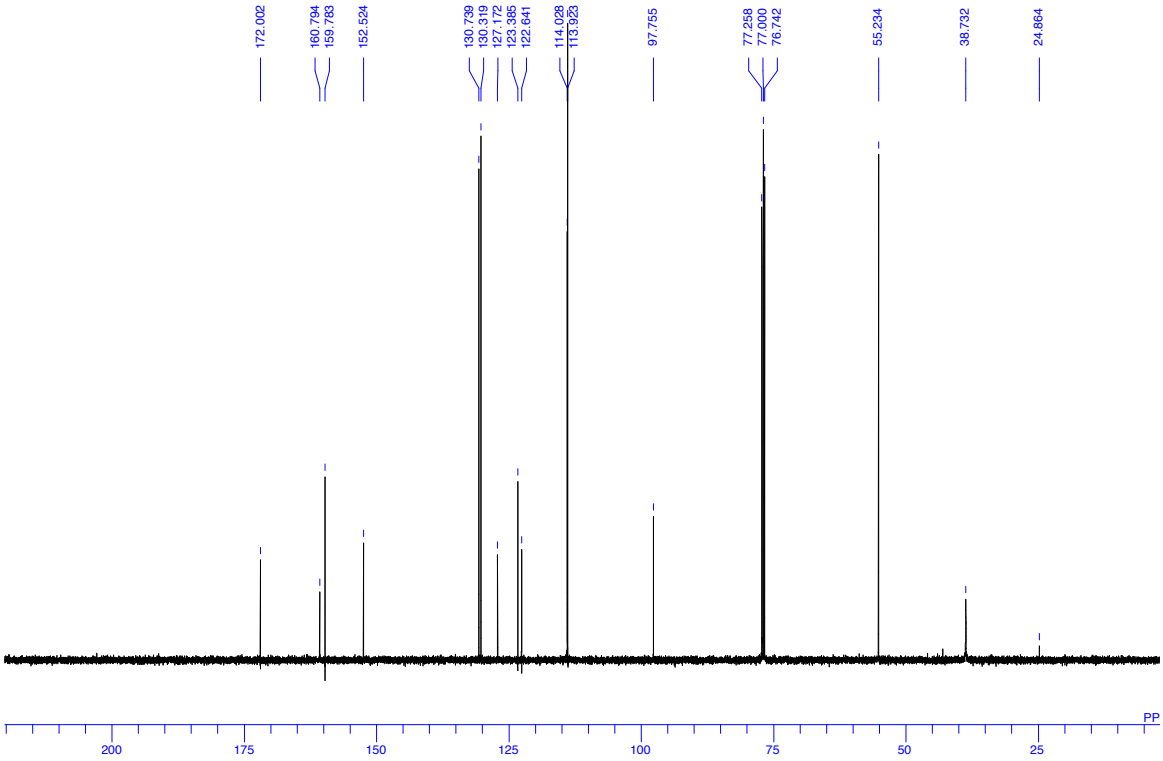
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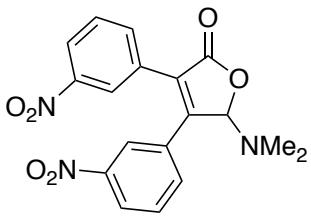
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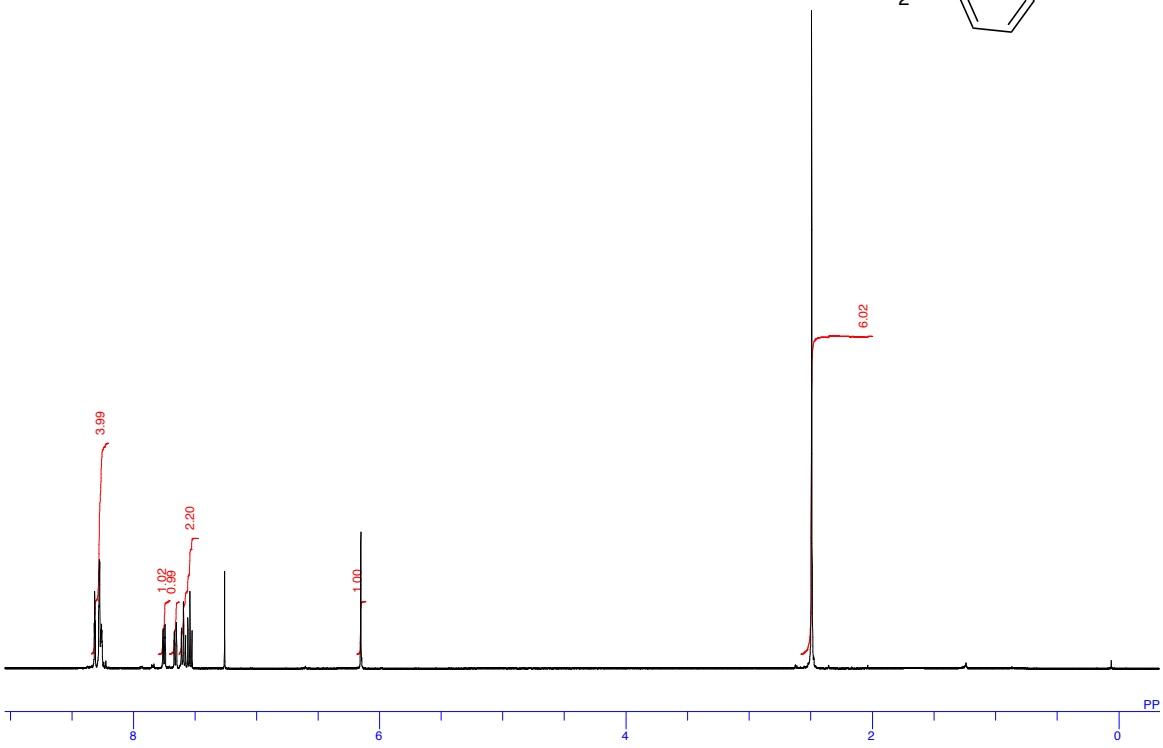
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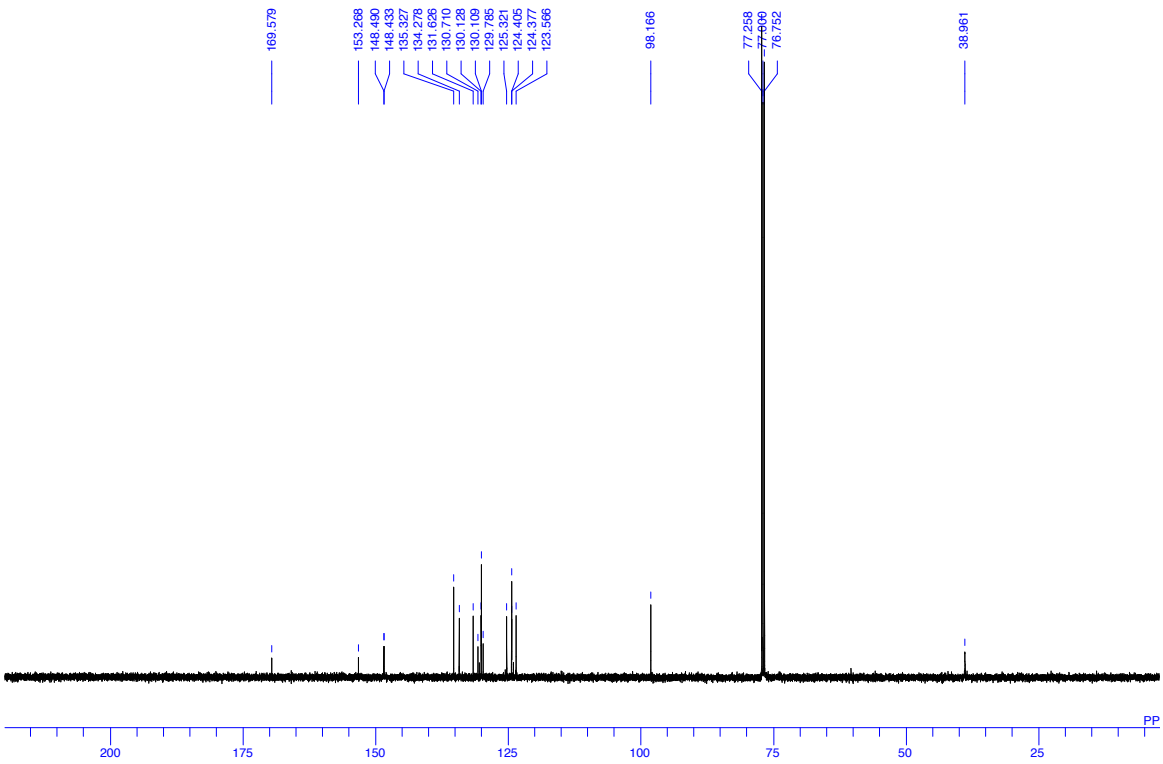
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148.490
148.433
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131.626
130.710
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124.405
124.377
123.566
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77.006
76.752
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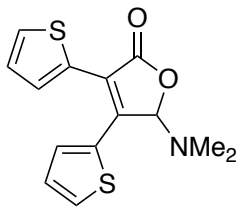
125
100
75
50
25

3l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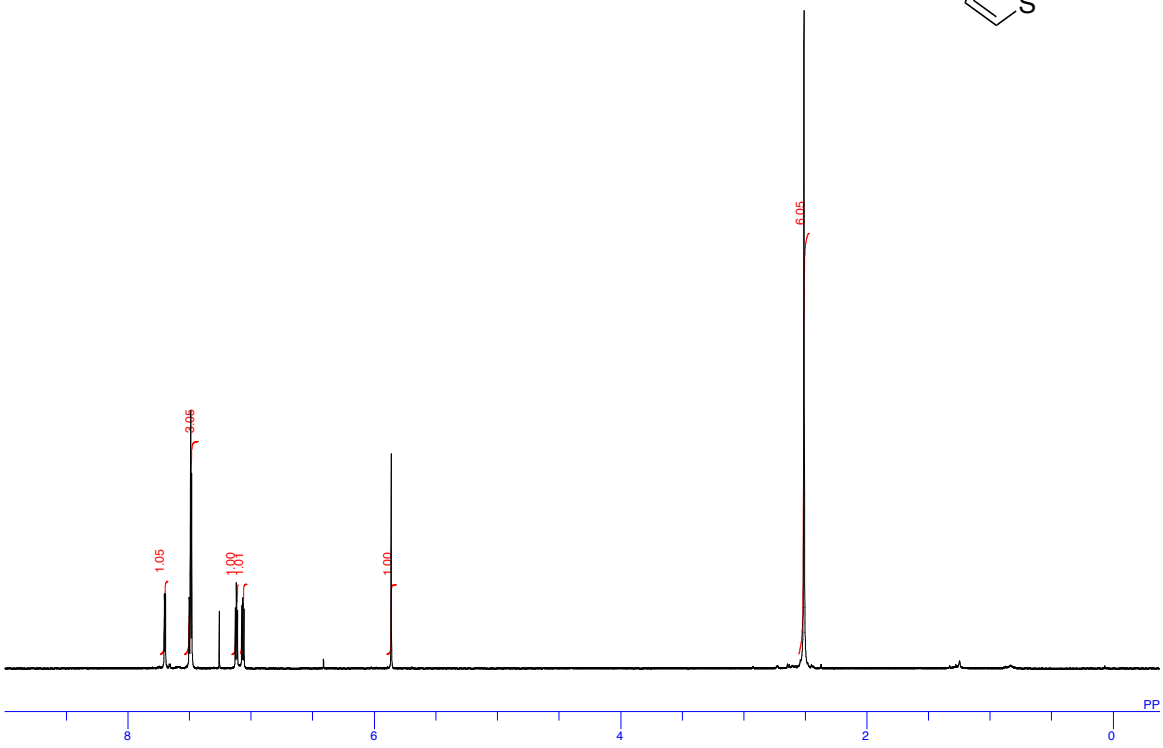
NMe2
S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1.00
1.01
1.00
6.05
PP
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6
4
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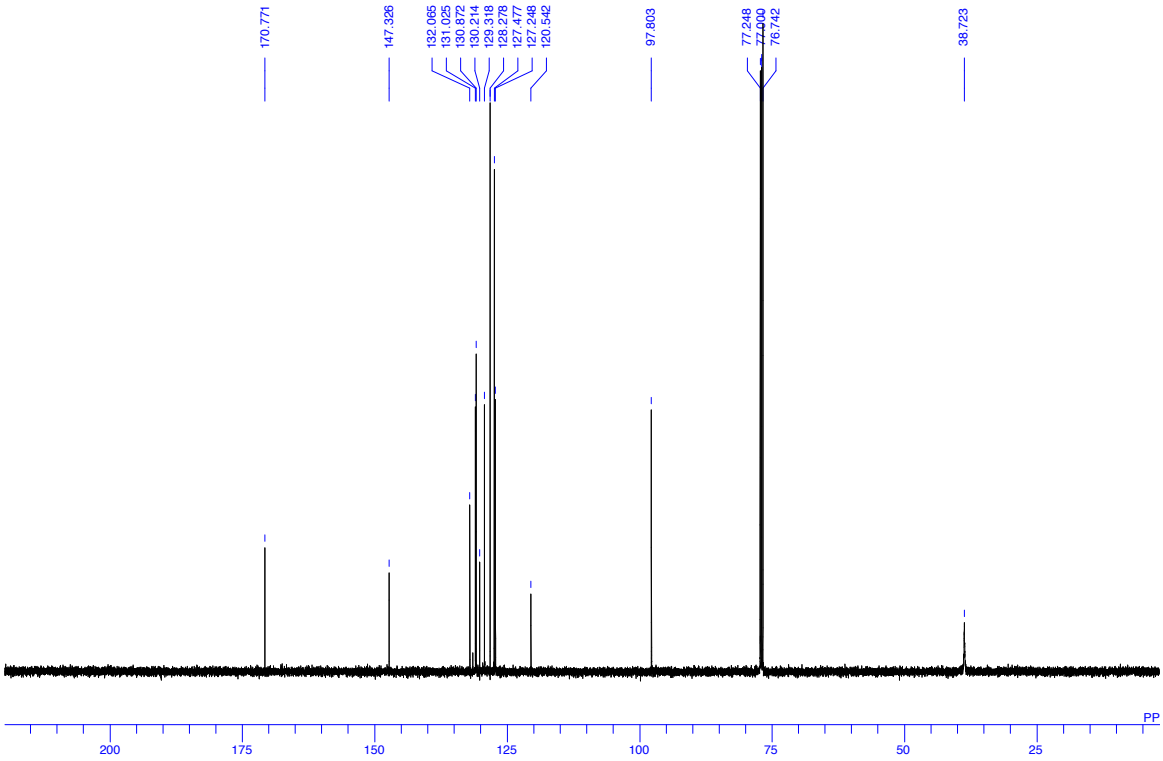
0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147.326
132.065
131.025
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130.214
129.318
128.278
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127.248
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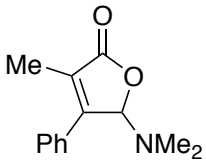
77.000
76.742
38.723
PP
200
175
150
125
100
75
50
25

3m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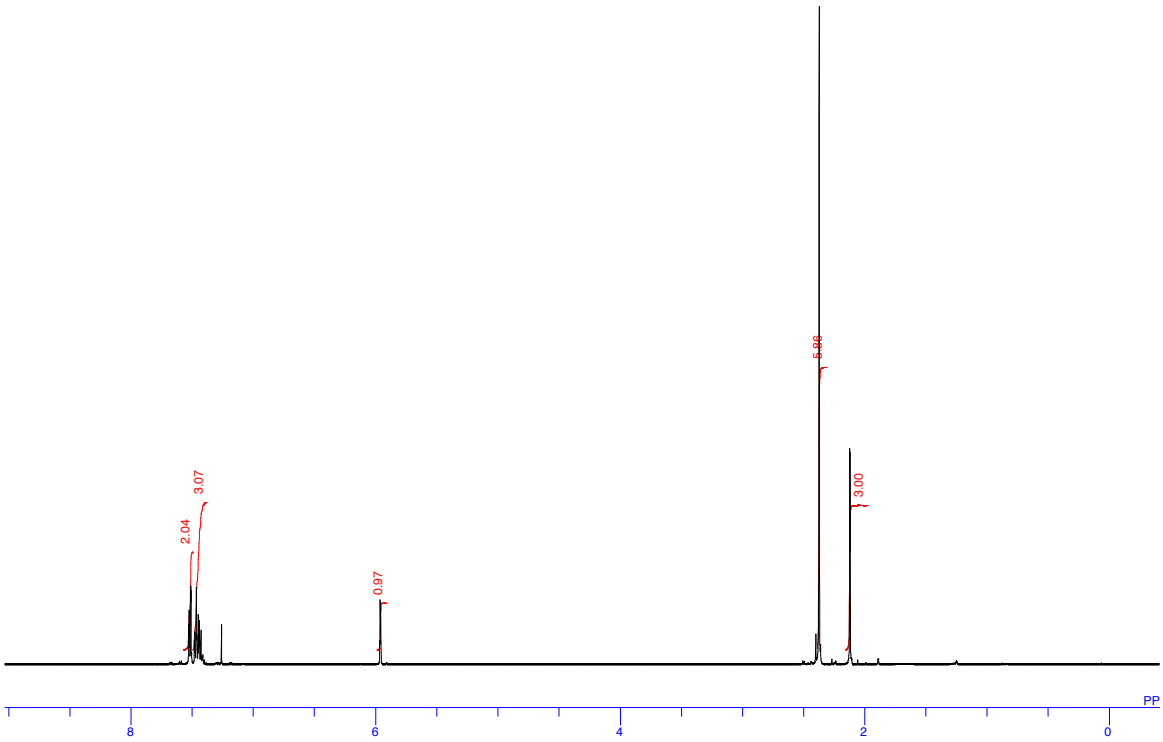
Me
O
Ph
NMe2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6
4
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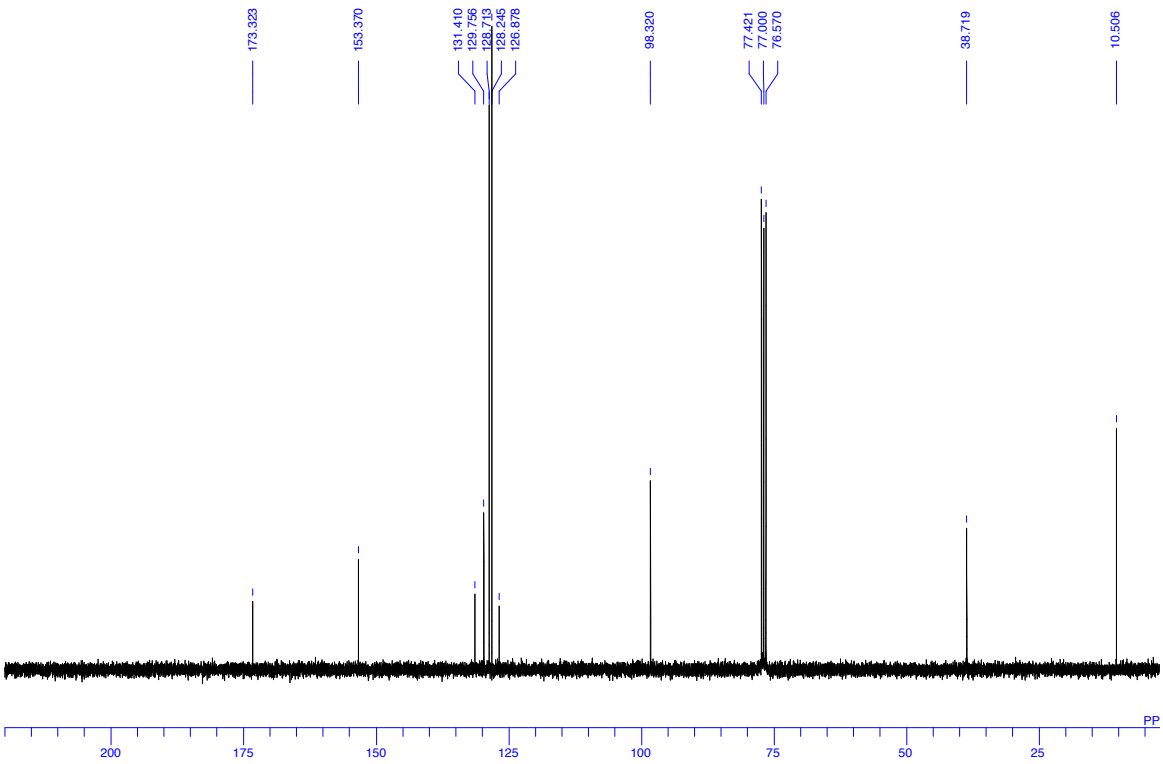
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175
150
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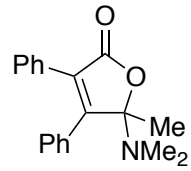
50
25

3n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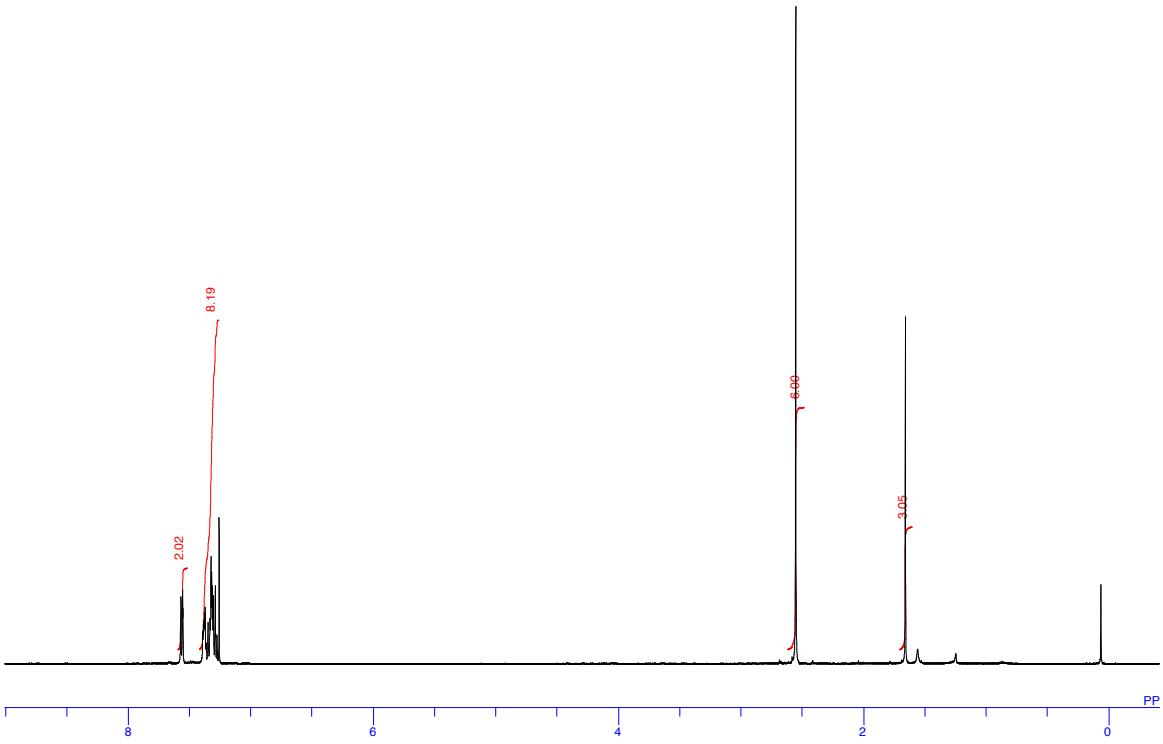
Ph
O
Me
Ph
NMe2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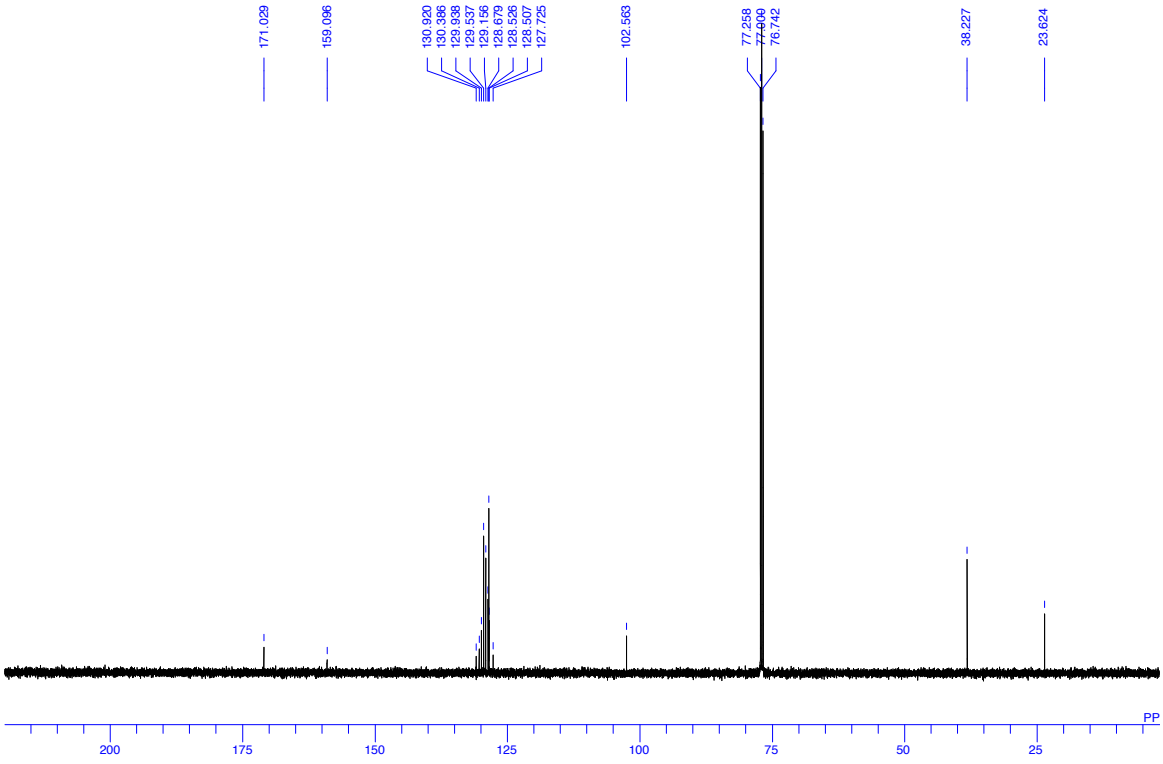
8.19
6.00
3.05
PP
8
6
4
2
0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128.507
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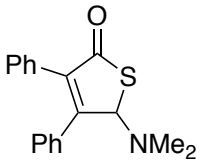
76.742
38.227
23.624
PP
200
175
150
125
100
75
50
25

3o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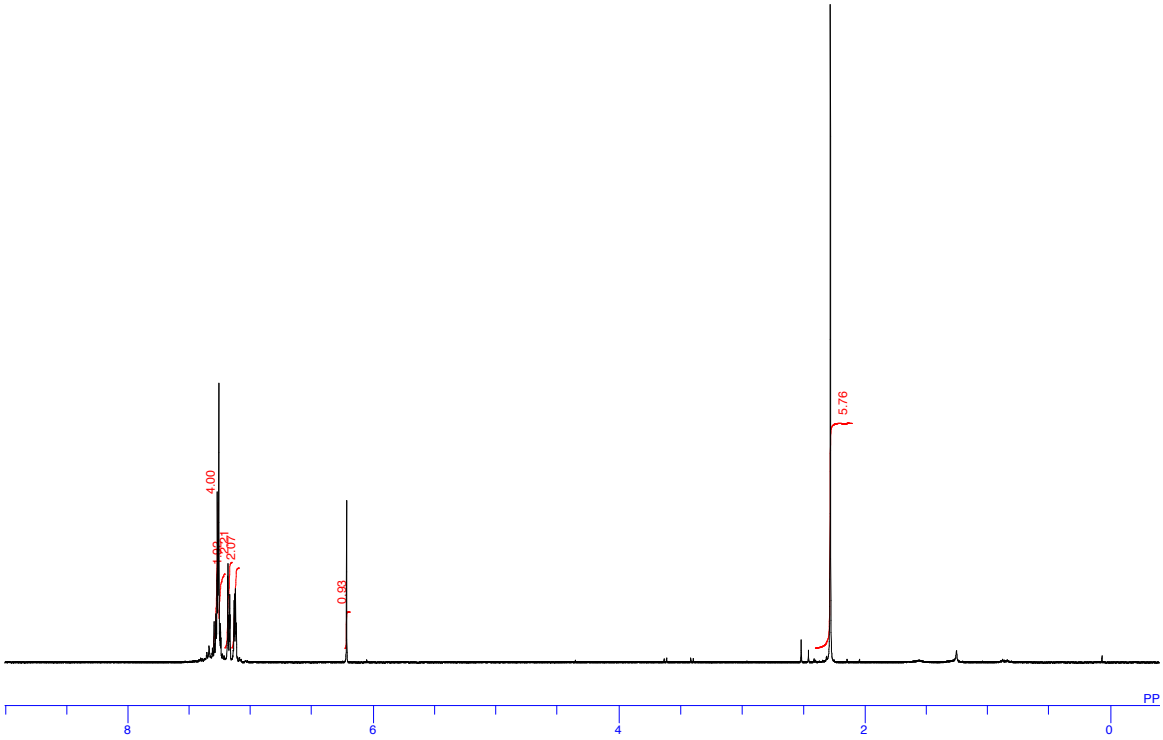
Ph
S
Ph
NMe2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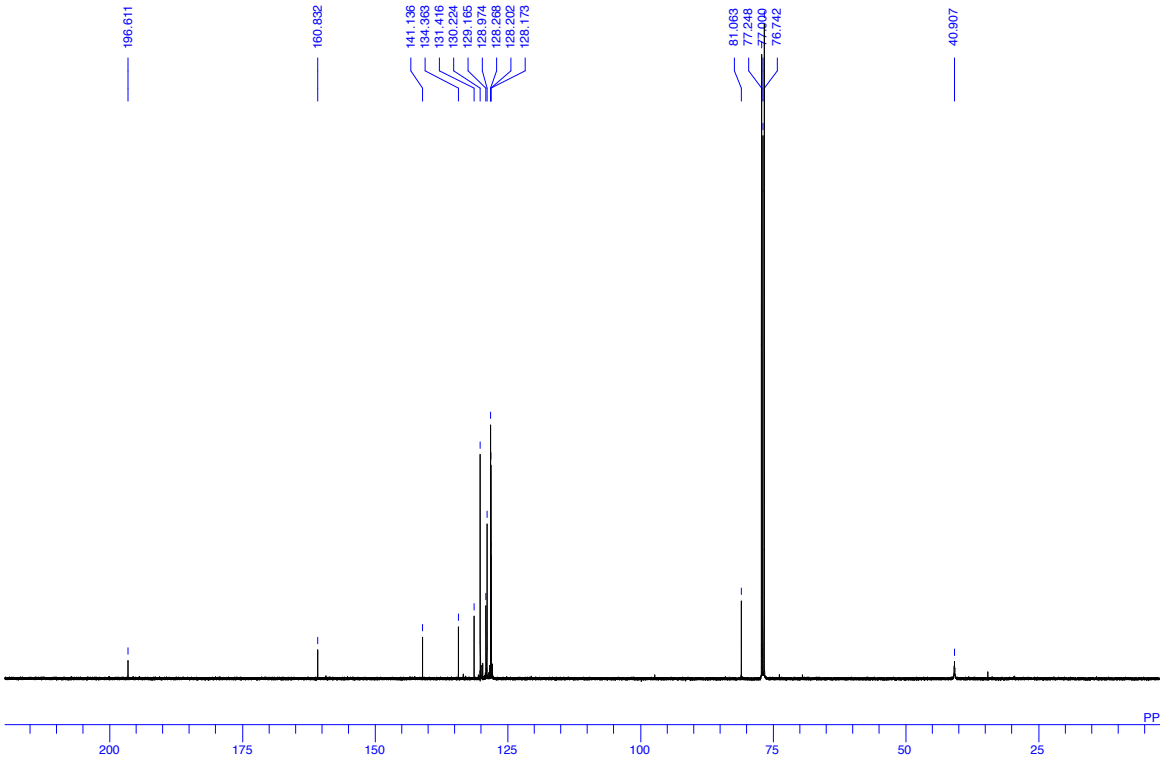
2.107
0.93
5.76
PP
8
6
4
2
0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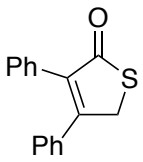
160.832
141.136
134.363
131.416
130.224
129.165
128.974
128.268
128.202
128.173
81.063
77.248
77.000
76.742
40.907
PP
200
175
150
125
100
75
50
25

4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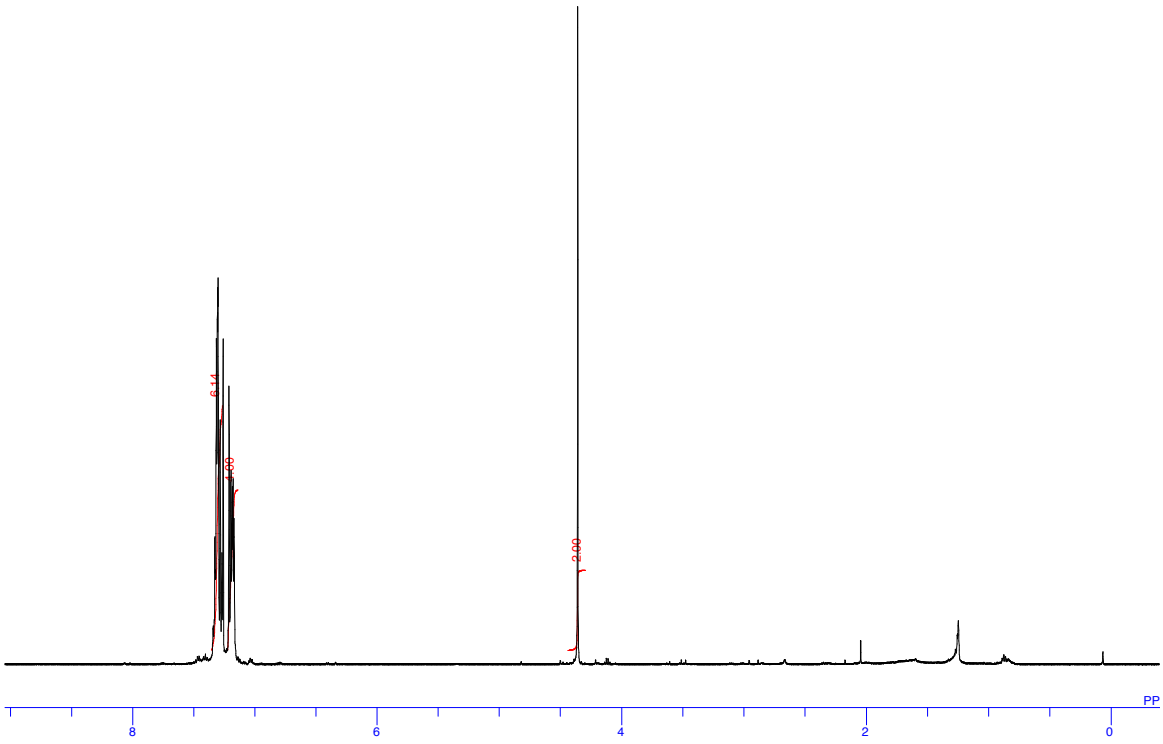
Ph
S
Ph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2.00
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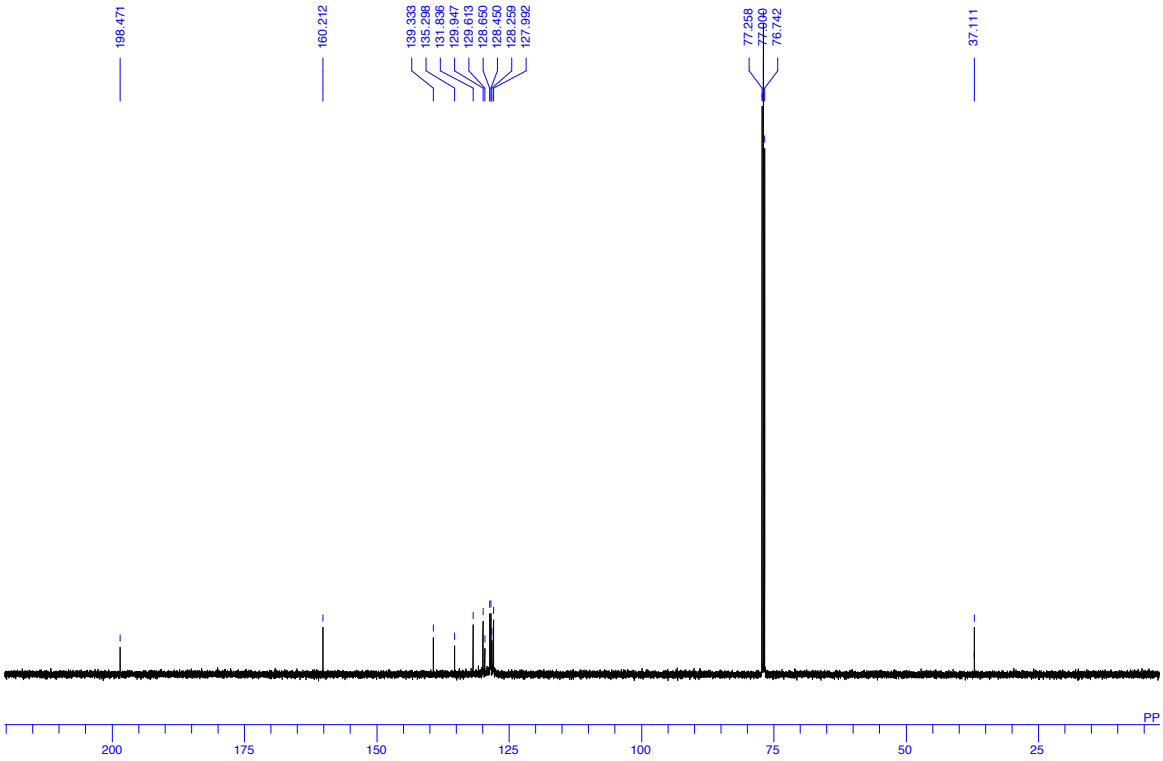
6
4
2
0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139.333
135.298
131.836
129.947
129.613
128.650
128.450
128.259
127.992
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77.000
76.742
37.111
PP
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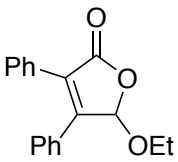
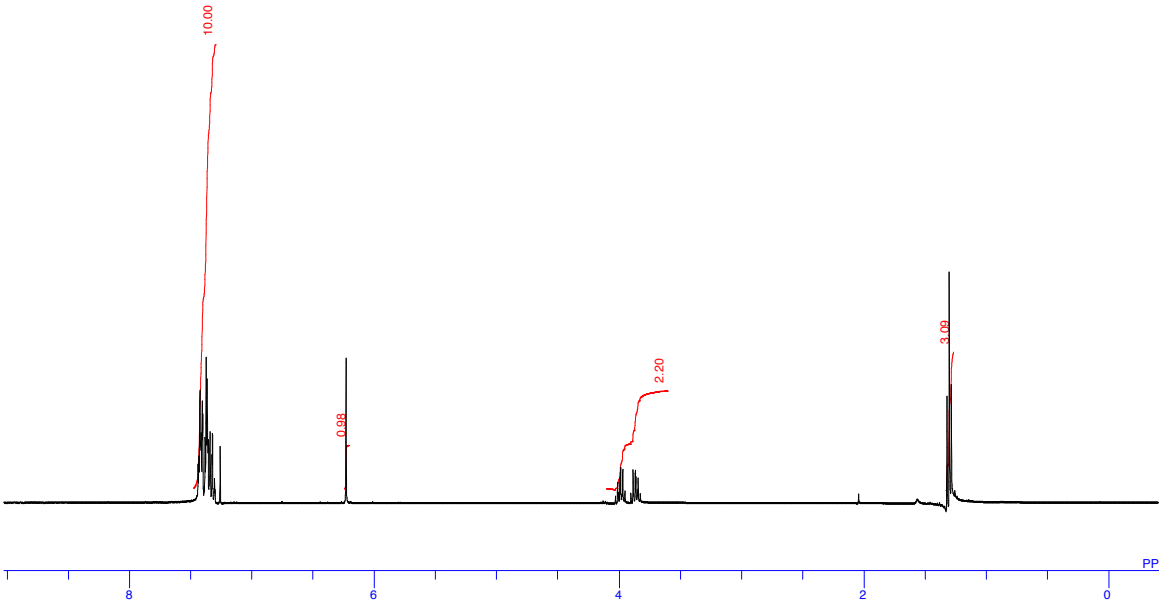
175
150
125
100
75
50
25

5


O
Ph
O
Ph
OEt


10.00
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2.20
3.09
PP
8
6
4
2
0


170.589
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130.425
130.339
129.545
129.364
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128.819
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128.637
128.513
101.695
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77.201
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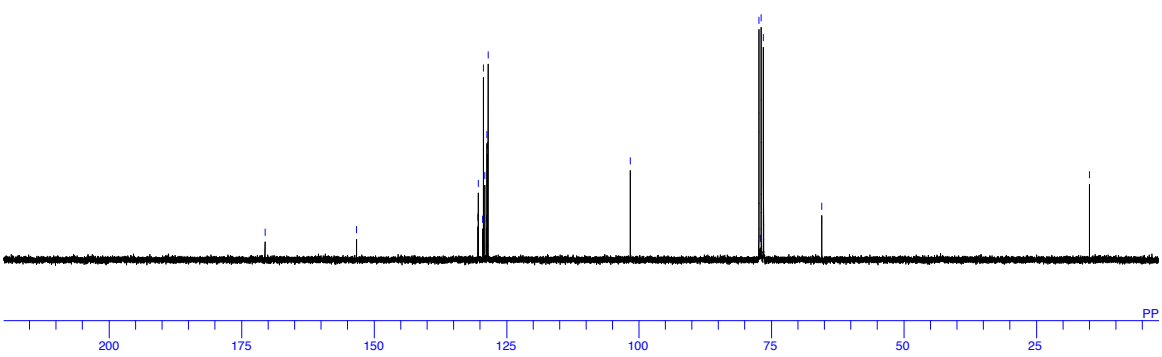
PP
200
175
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100
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