Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Supporting Information

Copper-Catalyzed Cascade Addition/Cyclization: An Efficient Access to Phosphonylated Quinoline-2,4(1*H*,3*H*)-diones

Ya-Min Li,* Shi-Sheng Wang, Fuchao Yu, Yuehai Shen, Kwen-Jen Chang

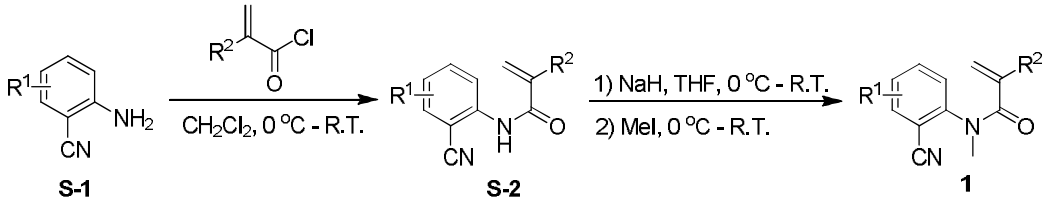
Faculty of Life Science and Technology, Kunming University of Science and Technology, Kunming 650500, P. R. China. E-mail: liym@kmust.edu.cn.

1. General information

NMR spectra were recorded on Bruker AVANCE III 400 (400 MHz for ^{1}H; 100 MHz for ^{13}C; 162 MHz for ^{13}C; 376 MHz for ^{19}F) and Bruker AVANCE DRX 500 (500 MHz for ^{1}H; 126 MHz for ^{13}C) instruments internally referenced to TMS signal. Data are reported as follows: Chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), Coupling constants, *J*, are reported in hertz. Mass spectra were mearsured using Agilent 6530 Accurate-Mass Q-TOF LC/MS. IR spectra were recorded on a Bruker Tensor 27 FT-IR spectrometer and only major peaks are reported in cm^{-1}. The starting materials were purchased from Aldrich, Acros Organics, TCI or J&K Chemicals and used without further purification. Solvents were dried and purified according to the procedure from "Purification of Laboratory Chemicals book". Column chromatography was carried out on silica gel (particle size 200-400 mesh ASTM).

2. Typical procedures for the synthesis of substrates

The preparation of amide **1** were described in previous reports. [S1]



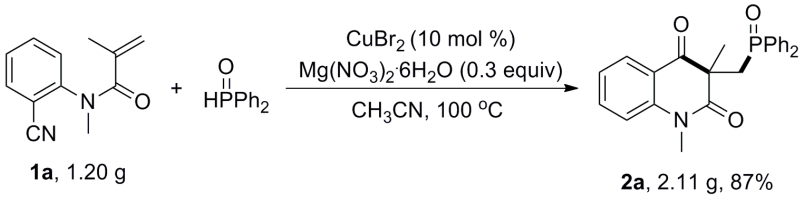
To the solution of anthranilonitrile **S-1** (2.0 mmol) and Et_3N (2.4 mmol) in 20 mL dry CH_2Cl_2 was added acryloyl chloride (2.2 mmol) at 0 ℃. The mixture was allowed to stir at room temperature. After completion of the reaction, the reaction was quenched with saturated $NaHCO_3$ solution, then extracted with CH_2Cl_2, washed with brine, dried over $MgSO_4$ and concentrated by evaporator affording amide **S-2** without any further purification.

To the solution of amide **S-2** (2.0 mmol) in 20 mL dry THF was added NaH (3.0 mmol) at 0 ℃ under argon. The mixture was allowed to stir at room temperature for 1h, then MeI (3.0 mmol) was added to the reaction mixture dropwise at 0℃. The reaction mixture was warmed to room temperature. After completion of the reaction, the reaction was cooled to 0 ℃ and quenched with H_2O and extracted with ether. The extract was washed with brine and dried over $MgSO_4$. Concentration under reduced pressure and purification by silica gel flash chromatography to afford amide **1**.

3. General procedure for phosphonylation/cyclization cascade

In a Schlenk tube, **1** (0.30 mmol), $HP(O)Ph_2$ (0.60 mmol), $CuBr_2$ (0.03 mmol), and $Mg(NO_3)_2{\cdot}6H_2O$ (0.09 mmol) were added and charged with N_2 three times. Then, CH_3CN (3.0 mL) were added. The mixture was allowed to stir at 100 ℃ for 10 hours (monitored by TLC). After completion of the reaction, the reaction was cooled to room temperature and diluted with CH_3CN, then filtering through a bed of Celite. The filtered reaction mixture was concentrated by rotary evaporation and purified by flash chromatography on silica gel with dichloromethane / isopropanol as the eluent to give the product **2**.

4. Gram-scale synthesis of 2a

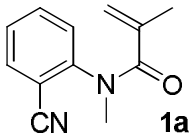


Reference:

(S1) (a) A. Pinto, Y. Jia, L. Neuville and J. Zhu, *Chem. –Eur. J.*, 2007, **13**, 961; (b) H. Wei, T. Piou, J. Dufour, L.

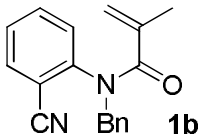
Neuville and J. Zhu, *Org. Lett.*, 2011, **13**, 2244; (c) X. Mu, T. Wu, H. Wang, Y. Guo and G. Liu, *J. Am. Chem. Soc.*, 2012, **134**, 878; (d) N. Campbell, A. Finch and S. Rokita, *ChemPhysChem*, 2010, **11**, 1768; (e) D. Walba, W. Thurmes and R. Haltiwanger, *J. Org. Chem.*, 1988, **53**, 1046; (f) C. Shaffer, S. Harriman, Y. Koen and R. Hanzlik, *J. Am. Chem. Soc.*, 2001, **123**, 8502.

5. Characterization of new compounds



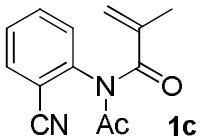
1a

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.72 (dd, *J* = 7.7, 1.2 Hz, 1H), 7.66 (t, *J* = 7.8 Hz, 1H), 7.44 (t, *J* = 7.7 Hz, 1H), 7.30 (d, *J* = 8.0 Hz, 1H), 5.13 (s, 1H), 5.02 (s, 1H), 3.40 (s, 3H), 1.89 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.50, 147.24, 139.70, 133.91, 133.60, 128.52, 127.82, 119.79, 116.10, 111.66, 37.53, 19.91. IR (film) υ_{max}: 2947, 2229, 1654, 1627, 1485, 1365, 1233, 1090, 920, 775, 556 cm^{-1}. HRMS calc. for $C_{12}H_{12}N_2O$ $(M+H)^+$, 201.1022; found, 201.1033.



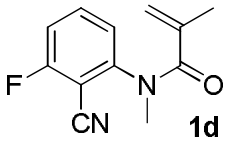
1b

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63 (d, *J* = 7.7 Hz, 1H), 7.52–7.44 (m, 1H), 7.34 (t, *J* = 7.6 Hz, 1H), 7.30–7.24 (m, 3H), 7.21 (dd, *J* = 7.8, 5.0 Hz, 2H), 6.99 (d, *J* = 8.0 Hz, 1H), 5.37 (s, 1H), 5.11 (s, 1H), 5.00 (s, 1H), 4.74 (s, 1H), 1.91 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.15, 145.22, 139.89, 136.01, 133.61, 133.41, 129.70, 128.90, 128.45, 127.84, 127.74, 120.00, 116.28, 112.61, 52.85, 20.13. IR (film) υ_{max}: 3031, 2957, 2228, 1647, 1489, 1451, 1386, 1326, 1203, 1110, 989, 843, 785, 697 cm^{-1}. HRMS calc. for $C_{18}H_{16}N_2O$ $(M+H)^+$, 277.1335; found, 277.1341.



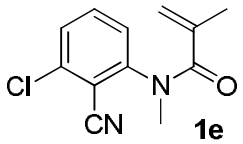
1c

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.76 (dd, *J* = 7.7, 1.2 Hz, 1H), 7.67 (td, *J* = 7.9, 1.4 Hz, 1H), 7.50 (t, *J* = 7.7 Hz, 1H), 7.24 (d, *J* = 8.0 Hz, 1H), 5.59 (s, 1H), 5.41 (d, *J* = 1.3 Hz, 1H), 2.43 (s, 3H), 1.94 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.74, 172.64, 141.59, 140.38, 133.84, 133.64, 129.67, 128.95, 122.66, 115.87, 113.09, 25.61, 18.99. IR (film) υ_{max}: 2924, 2230, 1699, 1635, 1449, 1296, 1217, 1022, 770, 669, 591 cm^{-1}. HRMS calc. for $C_{13}H_{12}N_2O_2$ $(M+Na)^+$, 251.0791; found, 251.0812.



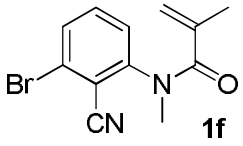
1d

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.64 (td, *J* = 8.3, 6.3 Hz, 1H), 7.20 (t, *J* = 8.4 Hz, 1H), 7.12 (d, *J* = 8.1 Hz, 1H), 5.19 (s, 1H), 5.04 (s, 1H), 3.40 (s, 3H), 1.93 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.40, 163.52 (d, $J_{C\text{-}F}$ = 261.5 Hz), 148.59, 139.55, 135.03 (d, $J_{C\text{-}F}$ = 9.7 Hz), 124.09, 120.02, 115.03 (d, $J_{C\text{-}F}$ = 19.5 Hz), 111.36, 101.33 (d, $J_{C\text{-}F}$ = 15.4 Hz), 37.65, 19.86. IR (film) υ_{max}: 2236, 1656, 1579, 1480, 1363, 1262, 1055, 929, 811, 621 cm^{-1}. HRMS calc. for $C_{12}H_{11}FN_2O$ $(M+H)^+$, 219.0928; found, 219.0939.



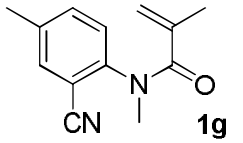
1e

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58 (t, J = 8.1 Hz, 1H), 7.49 (d, J = 8.2 Hz, 1H), 7.22 (d, J = 8.0 Hz, 1H), 5.19 (s, 1H), 5.05 (s, 1H), 3.40 (s, 3H), 1.93 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.40, 149.03, 139.51, 137.95, 134.08, 128.61, 126.68, 120.07, 113.43, 112.93, 37.67, 19.91. IR (film) υ_{max}: 2234, 1658, 1631, 1564, 1458, 1353, 1187, 926, 807, 741, 607 cm^{-1}. HRMS calc. for $C_{12}H_{11}ClN_2O$ $(M+H)^+$, 235.0633; found, 235.0642.



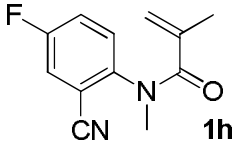
1f

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.65 (d, J = 8.2 Hz, 1H), 7.50 (t, J = 8.1 Hz, 1H), 7.25 (d, J = 7.9 Hz, 1H), 5.18 (s, 1H), 5.04 (s, 1H), 3.39 (s, 3H), 1.92 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.40, 149.25, 139.52, 134.21, 131.80, 127.22, 126.28, 119.99, 115.36, 114.66, 77.25, 77.00, 76.75, 37.70, 19.93. IR (film) υ_{max}: 2233, 1657, 1630, 1557, 1454, 1358, 1185, 925, 806, 740, 603 cm^{-1}. HRMS calc. for $C_{12}H_{11}BrN_2O$ $(M+H)^+$, 279.0128; found, 279.0137.



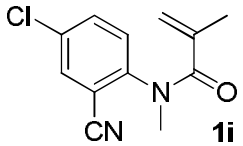
1g

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.50 (s, 1H), 7.43 (d, J = 8.0 Hz, 1H), 7.17 (d, J = 7.9 Hz, 1H), 5.09 (s, 1H), 5.00 (s, 1H), 3.37 (s, 3H), 2.42 (s, 3H), 1.87 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.62, 144.71, 139.82, 138.26, 134.68, 133.79, 128.33, 119.62, 116.23, 111.37, 37.41, 20.62, 19.97. IR (film) υ_{max}: 2946, 2232, 1656, 1627, 1507, 1366, 1097, 992, 851, 577 cm^{-1}. HRMS calc. for $C_{13}H_{14}N_2O$ $(M+H)^+$, 215.1179; found, 215.1190.



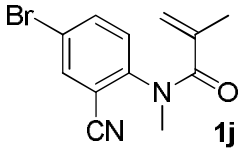
1h

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49–7.21 (m, 3H), 5.16 (s, 1H), 5.01 (s, 1H), 3.39 (s, 3H), 1.90 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.55, 160.41 (d, $J_{C\text{-}F}$ = 251.8 Hz), 143.65, 139.59, 130.52 (d, $J_{C\text{-}F}$ = 8.4 Hz), 121.47 (d, $J_{C\text{-}F}$ = 22.5 Hz), 120.24 (d, $J_{C\text{-}F}$ = 25.7 Hz), 114.92, 113.13, 37.47, 19.91. IR (film) υ_{max}: 3071, 2929, 2237, 1658, 1626, 1496, 1361, 1226, 931, 853, 785, 566 cm^{-1}. HRMS calc. for $C_{12}H_{11}FN_2O$ $(M+H)^+$, 219.0928; found, 219.0942.



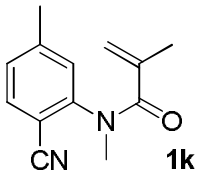
1i

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.68 (d, J = 2.4 Hz, 1H), 7.60 (dd, J = 8.6, 2.4 Hz, 1H), 7.25 (d, J = 8.6 Hz, 1H), 5.18 (s, 1H), 5.02 (s, 1H), 3.38 (s, 3H), 1.91 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.41, 145.86, 143.14, 139.55, 134.19, 133.55, 133.15, 129.78, 120.06, 114.89, 113.16, 37.66, 19.91. IR (film) υ_{max}: 3066, 2237, 1654, 1627, 1484, 1363, 1109, 916, 774, 569 cm^{-1}. HRMS calc. for $C_{12}H_{11}ClN_2O$ $(M+H)^+$, 235.0633; found, 235.0647.



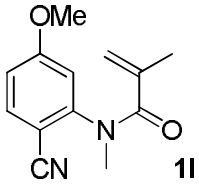
1j

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.82 (d, *J* = 2.1 Hz, 1H), 7.75 (dd, *J* = 8.5, 2.1 Hz, 1H), 7.18 (d, *J* = 8.5 Hz, 1H), 5.19 (s, 1H), 5.02 (s, 1H), 3.38 (s, 3H), 1.91 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.35, 146.33, 139.51, 137.13, 136.03, 129.94, 120.93, 120.17, 114.76, 113.43, 37.62, 19.91. IR (film) υ_{max}: 2234, 1656, 1631, 1483, 1362, 1102, 913, 854, 590 cm^{-1}. HRMS calc. for $C_{12}H_{11}BrN_2O$ $(M+H)^+$, 279.0128; found, 279.0138.



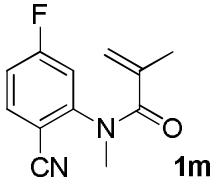
1k

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.58 (d, *J* = 7.9 Hz, 1H), 7.23 (d, *J* = 7.8 Hz, 1H), 7.09 (s, 1H), 5.12 (s, 1H), 5.03 (s, 1H), 3.38 (s, 3H), 2.44 (s, 3H), 1.90 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.54, 147.18, 145.30, 139.80, 133.34, 129.13, 128.66, 119.57, 116.37, 108.61, 37.53, 21.64, 19.98. IR (film) υ_{max}: 2974, 2223, 1650, 1622, 1606, 1425, 1359, 1237, 1099, 930, 839, 786, 553 cm^{-1}. HRMS calc. for $C_{13}H_{14}N_2O$ $(M+H)^+$, 215.1179; found, 215.1190.



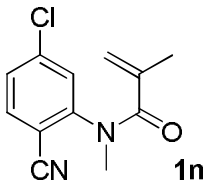
1l

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.62 (d, *J* = 8.7 Hz, 1H), 6.92 (dd, *J* = 8.7, 2.4 Hz, 1H), 6.76 (d, *J* = 1.5 Hz, 1H), 5.13 (s, 1H), 5.07 (s, 1H), 3.88 (s, 3H), 3.38 (s, 3H), 1.91 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.49, 163.52, 149.01, 139.78, 134.90, 119.52, 116.49, 114.57, 113.33, 103.19, 55.78, 37.43, 19.98. IR (film) υ_{max}: 3093, 2223, 1660, 1604, 1499, 1367, 1317, 1275, 1148, 1046, 955, 880, 833, 697, 555 cm^{-1}. HRMS calc. for $C_{13}H_{14}N_2O_2$ $(M+H)^+$, 231.1128; found, 231.1144.



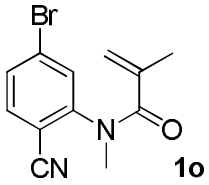
1m

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.73 (dd, *J* = 8.6, 5.9 Hz, 1H), 7.16 (td, *J* = 8.6, 2.3 Hz, 1H), 7.04 (dd, *J* = 8.8, 2.2 Hz, 1H), 5.20 (s, 1H), 5.05 (s, 1H), 3.41 (s, 3H), 1.94 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.29, 165.00 (d, *J* = 259.2 Hz), 149.62 (d, *J* = 10.6 Hz), 139.48, 135.47 (d, *J* = 10.2 Hz), 120.10, 116.35 (d, *J* = 23.1 Hz), 115.67, 115.47 (d, *J* = 5.2 Hz), 107.96, 37.65, 19.83. IR (film) υ_{max}: 3045, 2232, 1656, 1605, 1497, 1363, 1207, 1085, 925, 839, 782, 555 cm^{-1}. HRMS calc. for $C_{12}H_{11}FN_2O$ $(M+H)^+$, 219.0928; found, 219.0942.



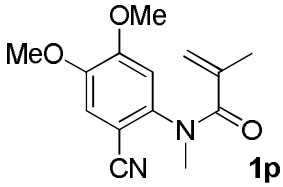
1n

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.65 (dd, *J* = 8.4, 1.4 Hz, 1H), 7.42 (d, *J* = 8.4 Hz, 1H), 7.32 (s, 1H), 5.21 (s, 1H), 5.05 (s, 1H), 3.40 (s, 3H), 1.94 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.31, 148.34, 139.96, 139.45, 134.35, 128.90, 128.24, 120.13, 115.42, 110.18, 37.71, 19.87. IR (film) υ_{max}: 3033, 2231, 1656, 1628, 1590, 1559, 1482, 1267, 1229, 1112, 1081, 924, 843, 778, 552 cm^{-1}. HRMS calc. for $C_{12}H_{11}ClN_2O$ $(M+H)^+$, 235.0633; found, 235.0643.



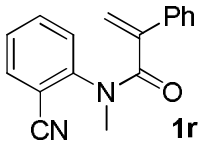
1o

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.57 (s, 2H), 7.47 (s, 1H), 5.21 (s, 1H), 5.04 (s, 1H), 3.39 (d, *J* = 1.2 Hz, 3H), 1.93 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.34, 148.24, 139.48, 134.36, 131.83, 131.16, 128.14, 120.18, 115.53, 110.67, 37.77, 19.90. IR (film) υ_{max}: 3029, 2230, 1658, 1627, 1479, 1358, 1229, 1102, 944, 842, 777, 552 cm^{-1}. HRMS calc. for $C_{12}H_{11}BrN_2O$ $(M+H)^+$, 279.0128; found, 279.0143.



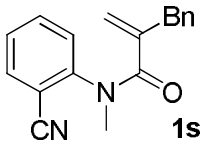
1p

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.07 (s, 1H), 6.72 (s, 1H), 5.11 (s, 1H), 5.06 (s, 1H), 3.93 (s, 3H), 3.93 (s, 3H), 3.37 (s, 3H), 1.89 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.63, 153.12, 148.19, 141.95, 139.94, 119.21, 116.41, 113.85, 111.25, 102.86, 56.26, 37.39, 20.04. IR (film) υ_{max}: 2217, 1653, 1624, 1522, 1443, 1358, 1267, 1238, 1158, 1041, 944, 798, 570 cm^{-1}. HRMS calc. for $C_{14}H_{16}N_2O_3$ $(M+H)^+$, 261.1234; found, 261.1253.



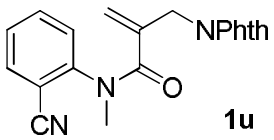
1r

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40 (d, *J* = 7.3 Hz, 1H), 7.31 (t, *J* = 7.5 Hz, 1H), 7.24–7.12 (m, 4H), 7.02 (d, *J* = 3.7 Hz, 2H), 6.95 (d, *J* = 7.9 Hz, 1H), 5.66 (s, 1H), 5.46 (s, 1H), 3.40 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 170.16, 145.89, 145.54, 136.21, 133.23, 133.16, 129.52, 128.33, 127.98, 127.76, 125.88, 119.59, 116.07, 111.97, 36.81. IR (film) υ_{max}: 2922, 2229, 1656, 1490, 1366, 1088, 770, 700, 534 cm^{-1}. HRMS calc. for $C_{17}H_{14}N_2O$ $(M+H)^+$, 263.1179; found, 263.1202.



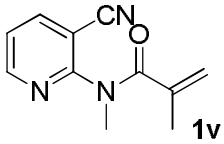
1s

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.66 (d, *J* = 8.5 Hz, 1H), 7.47–7.21 (m, 5H), 7.13 (s, 2H), 6.43 (s, 1H), 5.01 (s, 2H), 3.70 (s, 1H), 3.51 (s, 1H), 3.31 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 170.90, 147.11, 143.52, 137.41, 133.80, 133.50, 129.39, 128.83, 128.44, 127.73, 126.56, 119.43, 116.22, 111.60, 40.02, 37.55. IR (film) υ_{max}: 3074, 2227, 1655, 1625, 1490, 1366, 1234, 1091, 925, 787, 700, 511 cm^{-1}. HRMS calc. for $C_{18}H_{16}N_2O$ $(M+H)^+$, 277.1335; found, 277.1357.



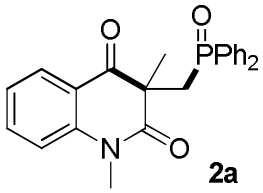
1u

^{1}H NMR (500 MHz, $CDCl_3$) δ 7.85 (dd, J = 5.1, 3.0 Hz, 2H), 7.78–7.69 (m, 3H), 7.64 (t, J = 7.8 Hz, 1H), 7.43 (t, J = 7.4 Hz, 2H), 5.32–5.04 (m, 2H), 4.51 (s, 2H), 3.41 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 168.85, 167.54, 146.67, 137.82, 134.06, 133.70, 131.81, 128.93, 128.11, 123.35, 120.19, 116.12, 111.74, 77.25, 77.00, 76.75, 39.57, 37.56. IR (film) υ_{max}: 2229, 1718, 1661, 1630, 1595, 1427, 1396, 1116, 958, 791, 715 cm^{-1}. HRMS calc. for $C_{20}H_{15}N_3O_3$ $(M+H)^+$, 346.1186; found, 346.1192.



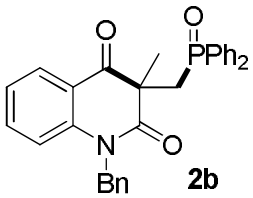
1v

^{1}H NMR (500 MHz, $CDCl_3$) δ 8.69 (dd, J = 4.9, 1.9 Hz, 1H), 8.03 (dd, J = 7.8, 1.9 Hz, 1H), 7.36 (dd, J = 7.8, 4.9 Hz, 1H), 5.17 (d, J = 0.6 Hz, 1H), 4.92 (s, 1H), 3.48 (s, 3H), 2.03 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.82, 158.50, 152.41, 142.02, 140.42, 121.60, 120.04, 115.03, 107.17, 35.87, 19.42. IR (film) υ_{max}: 2923, 2232, 1663, 1627, 1443, 1415, 1353, 1097, 774, 606 cm^{-1}. HRMS calc. for $C_{11}H_{11}N_3O$ $(M+H)^+$, 224.0794; found, 224.0814



2a

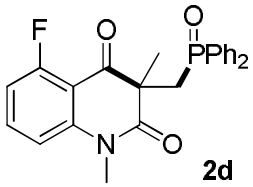
Purified by column chromatography (dichloromethane : isopropanol = 40 : 1). White solid, mp 192–193 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.97 (dd, J = 7.7, 1.6 Hz, 1H), 7.77–7.63 (m, 4H), 7.62–7.54 (m, 1H), 7.50–7.37 (m, 6H), 7.17–7.08 (m, 2H), 3.43 (s, 3H), 3.40–3.31 (m, 2H), 1.49 (d, J = 2.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.80 (d, $J_{C\text{-}P}$ = 1.6 Hz), 173.11 (d, $J_{C\text{-}P}$ = 1.9 Hz), 143.15, 135.60, 134.35 (d, $J_{C\text{-}P}$ = 100.1 Hz), 131.28 (d, $J_{C\text{-}P}$ = 2.7 Hz), 130.81 (d, $J_{C\text{-}P}$ = 9.8 Hz), 130.69 (d, $J_{C\text{-}P}$ = 9.7 Hz), 128.30, 128.26 (d, $J_{C\text{-}P}$ = 1.9 Hz), 128.14 (d, $J_{C\text{-}P}$ = 1.9 Hz), 122.64, 119.44, 114.76, 54.82 (d, $J_{C\text{-}P}$ = 4.1 Hz), 36.10 (d, $J_{C\text{-}P}$ = 69.4 Hz), 29.87, 27.47 (d, $J_{C\text{-}P}$ = 13.9 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 28.95. IR (film) υ_{max}: 1688, 1654, 1601, 1472, 1382, 1353, 1179, 1123, 1090, 754, 740, 699, 546 cm^{-1}. HRMS calc. for $C_{24}H_{22}NO_3P$ $(M+H)^+$, 404.1410; found, 404.1460.



2b

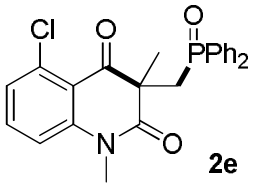
Purified by column chromatography (dichloromethane : isopropanol = 40 : 1). White solid, mp 174–175 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.03 (dd, J = 7.7, 1.4 Hz, 1H), 7.80–7.67 (m, 4H), 7.53–7.35 (m, 9H), 7.31 (t, J = 7.6 Hz, 2H), 7.22 (t, J = 7.2 Hz, 1H), 7.07 (t, J = 7.5 Hz, 1H), 7.00 (d, J = 8.4 Hz, 1H), 5.27 (dd, J = 34.7, 16.5 Hz, 2H), 3.54–3.34 (m, 2H), 1.58 (d, J = 1.7 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.80 (d, $J_{C\text{-}P}$ = 1.2 Hz), 173.76 (d, $J_{C\text{-}P}$ = 1.8 Hz), 142.33, 136.24, 135.49, 134.72 (d, $J_{C\text{-}P}$ = 99.9 Hz), 134.39 (d, $J_{C\text{-}P}$ = 100.7 Hz), 131.35 (d, $J_{C\text{-}P}$ = 2.6 Hz), 131.29 (d, $J_{C\text{-}P}$ = 2.7 Hz), 130.86 (d, $J_{C\text{-}P}$ = 2.3 Hz), 130.77 (d, $J_{C\text{-}P}$ = 2.1 Hz), 128.73, 128.51, 128.33 (d, $J_{C\text{-}P}$ = 10.6 Hz), 128.21 (d, $J_{C\text{-}P}$ = 10.6 Hz), 127.03, 126.34, 122.79, 119.65, 115.75, 55.37 (d, $J_{C\text{-}P}$ = 4.3 Hz), 46.32, 35.83 (d, $J_{C\text{-}P}$ = 69.4 Hz), 27.46 (d, $J_{C\text{-}P}$

= 13.8 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.59. IR (film) υ_{max}: 1698, 1662, 1601, 1469, 1379, 1321, 1185, 1119, 740, 696, 546 cm^{-1}. HRMS calc. for $C_{30}H_{26}NO_3P$ $(M+H)^+$, 480.1723; found, 480.1756.



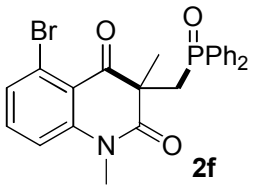
2d

Purified by column chromatography (dichloromethane : isopropanol = 40 : 1). White solid, mp 206–207 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.77–7.66 (m, 4H), 7.54–7.38 (m, 7H), 6.96 (d, J = 8.5 Hz, 1H), 6.81 (dd, J = 10.0, 8.6 Hz, 1H), 3.44 (s, 3H), 3.40–3.24 (m, 2H), 1.50 (d, J = 2.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.06, 172.75 (d, $J_{C\text{-}P}$ = 1.9 Hz), 162.47 (d, $J_{C\text{-}F}$ = 265.6 Hz), 144.40 (d, $J_{C\text{-}F}$ = 3.3 Hz), 135.73 (d, $J_{C\text{-}P}$ = 11.8 Hz), 134.62 (d, $J_{C\text{-}P}$ = 100.5 Hz), 134.33 (d, $J_{C\text{-}P}$ = 100.6 Hz), 131.30 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.27 (d, $J_{C\text{-}P}$ = 2.9 Hz), 130.80 (d, $J_{C\text{-}P}$ = 9.8 Hz), 130.70 (d, $J_{C\text{-}P}$ = 9.7 Hz), 128.30 (d, $J_{C\text{-}P}$ = 6.1 Hz), 128.18 (d, $J_{C\text{-}F}$ = 6.1 Hz), 110.83 (d, $J_{C\text{-}F}$ = 21.6 Hz), 110.64 (d, $J_{C\text{-}F}$ = 3.6 Hz), 109.22 (d, $J_{C\text{-}F}$ = 9.2 Hz), 55.91 (d, $J_{C\text{-}P}$ = 4.4 Hz), 35.40 (d, $J_{C\text{-}P}$ = 69.4 Hz), 30.73, 26.89 (d, $J_{C\text{-}P}$ = 13.6 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.22. ^{19}F NMR (376 MHz, $CDCl_3$) δ –110.36. IR (film) υ_{max}: 1698, 1664, 1611, 1477, 1380, 1351, 1176, 1119, 1056, 889, 807, 739, 697, 541, 474 cm^{-1}. HRMS calc. for $C_{24}H_{21}FNO_3P$ $(M+H)^+$, 422.1316; found, 422.1317.



2e

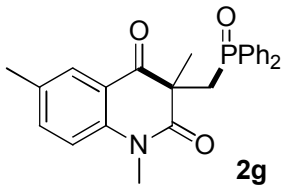
Purified by column chromatography (dichloromethane : isopropanol = 40 : 1). White solid, mp 191–192 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78–7.68 (m, 4H), 7.53–7.39 (m, 7H), 7.16 (d, J = 7.9 Hz, 1H), 7.10 (d, J = 8.4 Hz, 1H), 3.46 (s, 3H), 3.31 (d, J = 9.0 Hz, 2H), 1.48 (d, J = 2.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.65 (d, $J_{C\text{-}P}$ = 1.6 Hz), 172.45 (d, $J_{C\text{-}P}$ = 2.0 Hz), 144.94, 135.84, 134.88 (d, $J_{C\text{-}P}$ = 100.6 Hz), 134.46 (d, $J_{C\text{-}P}$ = 100.9 Hz), 134.11, 131.30 (d, $J_{C\text{-}P}$ = 3.0 Hz), 131.27 (d, $J_{C\text{-}P}$ = 3.0 Hz), 130.86 (d, $J_{C\text{-}P}$ = 9.8 Hz), 130.70 (d, $J_{C\text{-}P}$ = 9.7 Hz), 128.34 (d, $J_{C\text{-}P}$ = 7.4 Hz), 128.22 (d, $J_{C\text{-}P}$ = 7.3 Hz), 126.13, 117.37, 113.81, 56.25 (d, $J_{C\text{-}P}$ = 4.6 Hz), 35.11 (d, $J_{C\text{-}P}$ = 69.7 Hz), 30.91, 26.24 (d, $J_{C\text{-}P}$ = 13.5 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.12. IR (film) υ_{max}: 1666, 1590, 1461, 1343, 1180, 1120, 881, 734, 696, 548, 495 cm^{-1}. HRMS calc. for $C_{24}H_{21}ClNO_3P$ $(M+H)^+$, 438.1020; found, 438.1020.



2f

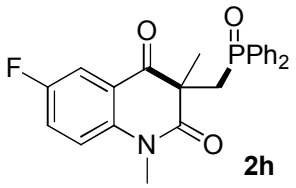
Purified by column chromatography (dichloromethane : isopropanol = 40 : 1). White solid, mp 294–295 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78–7.68 (m, 4H), 7.53–7.41 (m, 6H), 7.40 (dd, J = 7.9, 1.0 Hz, 1H), 7.34 (t, J = 8.1 Hz, 1H), 7.15 (dd, J = 8.2, 0.8 Hz, 1H), 3.46 (s, 3H), 3.31 (d, J = 9.0 Hz, 2H), 1.48 (d, J = 2.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.81 (d, $J_{C\text{-}P}$ = 1.6 Hz), 172.40 (d, $J_{C\text{-}P}$ = 2.0 Hz), 145.09, 134.87 (d, $J_{C\text{-}P}$ = 100.6 Hz), 134.46 (d, $J_{C\text{-}P}$ = 100.9 Hz), 134.31, 131.31 (d, $J_{C\text{-}P}$ = 2.7 Hz), 131.29 (d, $J_{C\text{-}P}$ = 2.6 Hz), 130.87 (d, $J_{C\text{-}P}$ = 9.8 Hz), 130.74 (d, $J_{C\text{-}P}$ = 9.7 Hz), 129.78, 128.36 (d, $J_{C\text{-}P}$ = 6.7 Hz), 128.24 (d, $J_{C\text{-}P}$ = 6.7 Hz), 123.54, 118.44, 114.52, 55.96 (d, $J_{C\text{-}P}$ = 4.6 Hz), 35.19 (d, $J_{C\text{-}P}$ = 69.7 Hz), 30.86, 26.13 (d, $J_{C\text{-}P}$ = 13.4 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.11. IR (film) υ_{max}: 1702, 1664, 1585, 1462, 1387,

1342, 1181, 1114, 1036, 875, 746, 695, 549 cm^{-1}. HRMS calc. for $C_{24}H_{21}BrNO_3P$ $(M+H)^+$, 482.0515; found, 482.0516.



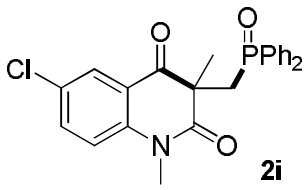
2g

Purified by column chromatography (dichloromethane : isopropanol = 40 : 1). White solid, mp 172–174 ℃. ^1H NMR (400 MHz, $CDCl_3$) δ 7.77 (s, 1H), 7.75–7.61 (m, 4H), 7.54–7.35 (m, 7H), 7.05 (d, J = 8.4 Hz, 1H), 3.47–3.25 (m, 5H), 2.32 (s, 3H), 1.48 (d, J = 1.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 196.11 (d, $J_{C\text{-}P}$ = 1.5 Hz), 172.94 (d, $J_{C\text{-}P}$ = 1.8 Hz), 140.96, 136.43, 134.36 (d, $J_{C\text{-}P}$ = 100.1 Hz), 134.33 (d, $J_{C\text{-}P}$ = 100.0 Hz), 132.29, 131.29 (d, $J_{C\text{-}P}$ = 2.7 Hz), 131.26 (d, $J_{C\text{-}P}$ = 2.7 Hz), 130.84 (d, $J_{C\text{-}P}$ = 9.8 Hz), 130.69 (d, $J_{C\text{-}P}$ = 9.7 Hz), 128.26 (d, $J_{C\text{-}P}$ = 2.2 Hz), 128.25, 128.15 (d, $J_{C\text{-}P}$ = 2.3 Hz), 119.17, 114.77, 54.71 (d, $J_{C\text{-}P}$ = 4.1 Hz), 36.05 (d, $J_{C\text{-}P}$ = 69.5 Hz), 29.85, 27.53 (d, $J_{C\text{-}P}$ = 14.0 Hz), 20.23. ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.02. IR (film) υ_{max}: 1687, 1655, 1618, 1434, 1342, 1177, 1118, 826, 743, 697, 548, 492 cm^{-1}. HRMS calc. for $C_{25}H_{24}NO_3P$ $(M+H)^+$, 418.1567; found, 418.1567.



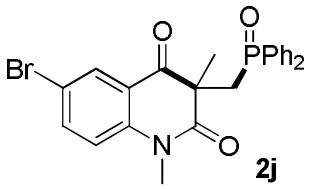
2h

Purified by column chromatography (dichloromethane : isopropanol = 40 : 1). White solid, mp 160–161 ℃. ^1H NMR (400 MHz, $CDCl_3$) δ 7.76–7.61 (m, 5H), 7.53–7.39 (m, 6H), 7.34–7.27 (m, 1H), 7.17–7.09 (m, 1H), 3.42 (s, 3H), 3.36 (dd, J = 16.9, 8.9 Hz, 2H), 1.49 (d, J = 2.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.12, 172.76 (d, $J_{C\text{-}P}$ = 1.8 Hz), 158.31 (d, $J_{C\text{-}F}$ = 244.3 Hz), 139.63 (d, $J_{C\text{-}F}$ = 2.2 Hz), 134.19 (d, $J_{C\text{-}P}$ = 100.2 Hz), 134.15 (d, $J_{C\text{-}P}$ = 100.4 Hz), 131.42 (d, $J_{C\text{-}P}$ = 2.4 Hz), 130.82 (d, $J_{C\text{-}P}$ = 9.8 Hz), 130.68 (d, $J_{C\text{-}P}$ = 9.7 Hz), 128.35 (d, $J_{C\text{-}P}$ = 3.7 Hz), 128.23 (d, $J_{C\text{-}P}$ = 3.7 Hz), 122.57 (d, $J_{C\text{-}F}$ = 23.4 Hz), 120.65 (d, $J_{C\text{-}F}$ = 6.4 Hz), 116.60 (d, $J_{C\text{-}F}$ = 7.2 Hz), 113.97 (d, $J_{C\text{-}F}$ = 23.3 Hz), 54.64 (d, $J_{C\text{-}P}$ = 3.5 Hz), 36.40 (d, $J_{C\text{-}P}$ = 69.2 Hz), 30.17, 27.30 (d, $J_{C\text{-}P}$ = 13.8 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.09. ^{19}F NMR (376 MHz, $CDCl_3$) δ –120.40. IR (film) υ_{max}: 1652, 1508, 1463, 1173, 1114, 759, 552 cm^{-1}. HRMS calc. for $C_{24}H_{21}FNO_3P$ $(M+H)^+$, 422.1316; found, 422.1319.



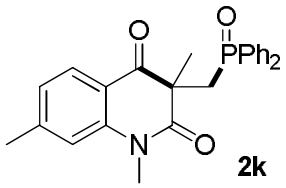
2i

Purified by column chromatography (dichloromethane : isopropanol = 50 : 1). White solid, mp 153–155 ℃. ^1H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, J = 2.6 Hz, 1H), 7.76–7.60 (m, 4H), 7.57–7.40 (m, 7H), 7.12 (d, J = 8.9 Hz, 1H), 3.43 (s, 3H), 3.41–3.27 (m, 2H), 1.49 (d, J = 2.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.94 (d, $J_{C\text{-}P}$ = 1.6 Hz), 172.93 (d, $J_{C\text{-}P}$ = 1.8 Hz), 141.72, 135.26, 134.12 (d, $J_{C\text{-}P}$ = 100.5 Hz), 134.08 (d, $J_{C\text{-}P}$ = 100.4 Hz), 131.47 (d, $J_{C\text{-}P}$ = 2.7 Hz), 130.86, 130.74 (d, $J_{C\text{-}P}$ = 3.9 Hz), 130.63, 128.50, 128.39 (d, $J_{C\text{-}P}$ = 3.0 Hz), 128.27 (d, $J_{C\text{-}P}$ = 3.0 Hz), 127.81, 120.45, 116.48, 54.85 (d, $J_{C\text{-}P}$ = 4.1 Hz), 36.32 (d, $J_{C\text{-}P}$ = 69.1 Hz), 30.12, 27.23 (d, $J_{C\text{-}P}$ = 13.8 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.16. IR (film) υ_{max}: 1698, 1656, 1596, 1465, 1335, 1180, 1115, 749, 694, 548, 499 cm^{-1}. HRMS calc. for $C_{24}H_{21}ClNO_3P$ $(M+H)^+$, 438.1020; found, 438.1019.



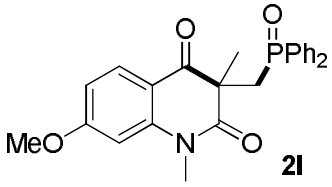
2j

Purified by column chromatography (dichloromethane : isopropanol = 40 : 1). White solid, mp 89–91 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.05 (d, J = 2.4 Hz, 1H), 7.76–7.60 (m, 5H), 7.55–7.37 (m, 6H), 7.05 (d, J = 8.9 Hz, 1H), 3.41 (s, 3H), 3.35 (dd, J = 12.6, 8.9 Hz, 2H), 1.48 (d, J = 2.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.76, 172.89, 142.19, 138.08, 134.11 (d, $J_{C\text{-}P}$ = 100.3 Hz), 134.05 (d, $J_{C\text{-}P}$ = 100.4 Hz), 131.44 (d, $J_{C\text{-}P}$ = 2.3 Hz), 130.79 (d, $J_{C\text{-}P}$ = 8.9 Hz), 130.68 (d, $J_{C\text{-}P}$ = 11.0 Hz), 128.36 (d, $J_{C\text{-}P}$ = 2.9 Hz), 128.24 (d, $J_{C\text{-}P}$ = 3.0 Hz), 120.78, 116.76, 115.74, 54.81 (d, $J_{C\text{-}P}$ = 3.9 Hz), 36.42 (d, $J_{C\text{-}P}$ = 69.1 Hz), 30.04, 27.19 (d, $J_{C\text{-}P}$ = 13.8 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.13. IR (film) υ_{max}: 1699, 1663, 1595, 1466, 1428, 1333, 1185, 1120, 787, 696, 548 cm^{-1}. HRMS calc. for $C_{24}H_{21}BrNO_3P$ $(M+H)^+$, 482.0515; found, 482.0514.



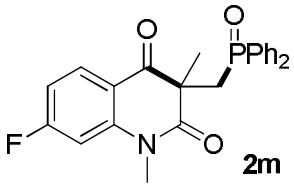
2k

Purified by column chromatography (dichloromethane : isopropanol = 40 : 1). White solid, mp 83–84 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.85 (d, J = 8.3 Hz, 1H), 7.77–7.61 (m, 4H), 7.51–7.36 (m, 6H), 6.95 (s, 1H), 6.94 (d, J = 7.4 Hz, 1H), 3.42 (s, 3H), 3.34 (dd, J = 9.1, 2.7 Hz, 2H), 2.43 (s, 3H), 1.48 (d, J = 2.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.41 (d, $J_{C\text{-}P}$ = 1.5 Hz), 173.39 (d, $J_{C\text{-}P}$ = 1.9 Hz), 146.96, 143.25, 134.37 (d, $J_{C\text{-}P}$ = 100.1 Hz), 134.30 (d, $J_{C\text{-}P}$ = 100.1 Hz), 131.31 (d, $J_{C\text{-}P}$ = 2.7 Hz), 130.86 (d, $J_{C\text{-}P}$ = 9.9 Hz), 130.76 (d, $J_{C\text{-}P}$ = 9.9 Hz), 128.40, 128.22 (d, $J_{C\text{-}P}$ = 12.0 Hz), 123.76, 117.35, 115.25, 54.59 (d, $J_{C\text{-}P}$ = 4.1 Hz), 36.21 (d, $J_{C\text{-}P}$ = 69.5 Hz), 29.86, 27.71 (d, $J_{C\text{-}P}$ = 14.0 Hz), 22.30. ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.02. IR (film) υ_{max}: 1693, 1655, 1610, 1468, 1437, 1341, 1299, 1181, 1119, 734, 696, 542 cm^{-1}. HRMS calc. for $C_{25}H_{24}NO_3P$ $(M+H)^+$, 418.1567; found, 418.1572.



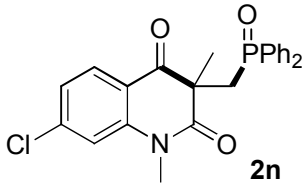
2l

Purified by column chromatography (dichloromethane : isopropanol = 40 : 1). White solid, mp 128–129 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.92 (d, J = 8.7 Hz, 1H), 7.77–7.60 (m, 4H), 7.51–7.36 (m, 6H), 6.65 (dd, J = 8.7, 2.2 Hz, 1H), 6.60 (d, J = 2.1 Hz, 1H), 3.89 (s, 3H), 3.40 (s, 3H), 3.33 (d, J = 9.1 Hz, 2H), 1.49 (d, J = 2.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.27 (d, $J_{C\text{-}P}$ = 1.5 Hz), 173.61 (d, $J_{C\text{-}P}$ = 1.8 Hz), 165.65, 145.11, 134.43 (d, $J_{C\text{-}P}$ = 99.9 Hz), 134.27 (d, $J_{C\text{-}P}$ = 99.9 Hz), 131.30 (d, $J_{C\text{-}P}$ = 1.8 Hz), 130.87, 130.79 (d, $J_{C\text{-}P}$ = 2.7 Hz), 130.74, 130.71, 128.27 (d, $J_{C\text{-}P}$ = 1.5 Hz), 128.15 (d, $J_{C\text{-}P}$ = 1.6 Hz), 113.44, 108.02, 100.73, 55.65, 54.23 (d, $J_{C\text{-}P}$ = 4.0 Hz), 36.19 (d, $J_{C\text{-}P}$ = 69.5 Hz), 29.87, 27.91 (d, $J_{C\text{-}P}$ = 14.1 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 28.82. IR (film) υ_{max}: 1682, 1647, 1606, 1451, 1437, 1370, 1341, 1225, 1178, 1120, 1105, 733, 695, 543, 502 cm^{-1}. HRMS calc. for $C_{25}H_{24}NO_4P$ $(M+H)^+$, 434.1516; found, 434.1521.



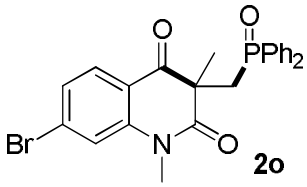
2m

Purified by column chromatography (dichloromethane : isopropanol = 40 : 1). White solid, mp 186–187 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (dd, J = 8.5, 6.6 Hz, 1H), 7.77–7.60 (m, 4H), 7.52–7.37 (m, 6H), 6.88–6.77 (m, 2H), 3.40 (s, 3H), 3.38–3.32 (m, 2H), 1.48 (d, J = 2.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.34 (d, $J_{C\text{-}P}$ = 1.5 Hz), 173.32 (d, $J_{C\text{-}P}$ = 1.8 Hz), 167.34 (d, $J_{C\text{-}F}$ = 254.9 Hz), 145.45 (d, $J_{C\text{-}F}$ = 11.7 Hz), 134.23 (d, $J_{C\text{-}P}$ = 101.0 Hz), 134.20 (d, $J_{C\text{-}P}$ = 101.0 Hz), 131.38 (d, $J_{C\text{-}P}$ = 2.8 Hz), 131.22 (d, $J_{C\text{-}P}$ = 11.2 Hz), 130.73 (d, $J_{C\text{-}P}$ = 19.2 Hz), 130.72, 128.26 (d, $J_{C\text{-}F}$ = 11.9 Hz), 116.10 (d, $J_{C\text{-}F}$ = 2.3 Hz), 110.01 (d, $J_{C\text{-}F}$ = 22.2 Hz), 102.32 (d, $J_{C\text{-}F}$ = 27.5 Hz), 54.66 (d, J = 4.1 Hz), 36.24 (d, J = 69.2 Hz), 30.04, 27.45 (d, J = 13.9 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 28.98. ^{19}F NMR (376 MHz, $CDCl_3$) δ –100.17. IR (film) υ_{max}: 1695, 1660, 1616, 1466, 1438, 1341, 1307, 1189, 1176, 1119, 1102, 842, 734, 698, 547, 500 cm^{-1}. HRMS calc. for $C_{24}H_{21}FNO_3P$ $(M+H)^+$, 422.1316; found, 422.1319.



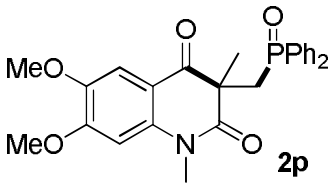
2n

Purified by column chromatography (dichloromethane : isopropanol = 40 : 1). White solid, mp 177–178 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, J = 8.4 Hz, 1H), 7.76–7.60 (m, 4H), 7.53–7.37 (m, 6H), 7.16 (d, J = 1.2 Hz, 1H), 7.10 (dd, J = 1.2, 8.0, 1H), 3.41 (s, 3H), 3.35 (dd, J = 8.9, 3.9 Hz, 2H), 1.48 (d, J = 2.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.76 (d, $J_{C\text{-}P}$ = 1.5 Hz), 173.22 (d, $J_{C\text{-}P}$ = 1.9 Hz), 144.15, 141.89, 134.19 (d, $J_{C\text{-}P}$ = 101.0 Hz), 134.17 (d, $J_{C\text{-}P}$ = 101.0 Hz), 131.41 (d, $J_{C\text{-}P}$ = 2.8 Hz), 130.84, 130.73 (d, $J_{C\text{-}P}$ = 1.7 Hz), 130.63, 129.73, 128.34 (d, $J_{C\text{-}P}$ = 1.7 Hz), 128.22 (d, $J_{C\text{-}P}$ = 1.7 Hz), 122.93, 117.88, 115.10, 54.78 (d, $J_{C\text{-}P}$ = 4.1 Hz), 36.35 (d, $J_{C\text{-}P}$ = 69.2 Hz), 30.01, 27.35 (d, $J_{C\text{-}P}$ = 13.8 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 28.99. IR (film) υ_{max}: 1695, 1660, 1597, 1434, 1335, 1184, 1121, 1100, 748, 700, 537 cm^{-1}. HRMS calc. for $C_{24}H_{21}ClNO_3P$ $(M+H)^+$, 438.1020; found, 438.1048.



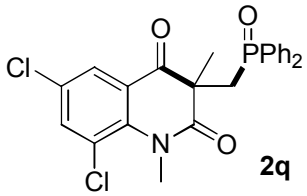
2o

Purified by column chromatography (dichloromethane : isopropanol = 40 : 1). White solid, mp 211–212 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 (d, J = 8.3 Hz, 1H), 7.76–7.58 (m, 4H), 7.54–7.37 (m, 6H), 7.33 (d, J = 1.5 Hz, 1H), 7.27 (dd, J = 8.3, 1.5 Hz, 1H), 3.41 (s, 3H), 3.35 (dd, J = 8.9, 4.6 Hz, 1H), 1.48 (d, J = 1.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.98, 173.19 (d, $J_{C\text{-}P}$ = 1.6 Hz), 144.06, 134.19 (d, $J_{C\text{-}P}$ = 100.0 Hz), 134.16 (d, $J_{C\text{-}P}$ = 100.0 Hz), 131.43 (d, $J_{C\text{-}P}$ = 2.6 Hz), 130.86, 130.75 (d, $J_{C\text{-}P}$ = 1.9 Hz), 130.65, 129.70, 128.36 (d, $J_{C\text{-}P}$ = 1.7 Hz), 128.24 (d, $J_{C\text{-}P}$ = 1.8 Hz), 125.91, 118.25, 118.05, 54.84 (d, $J_{C\text{-}P}$ = 4.0 Hz), 36.38 (d, $J_{C\text{-}P}$ = 69.1 Hz), 30.04, 27.33 (d, $J_{C\text{-}P}$ = 13.8 Hz). ^{31}P NMR (162 MHz, $CDCl_3$): δ 29.04. IR (film) υ_{max}: 1693, 1659, 1587, 1423, 1331, 1286, 1182, 1092, 1025, 897, 748, 702, 530 cm^{-1}. HRMS calc. for $C_{24}H_{21}BrNO_3P$ $(M+H)^+$, 482.0515; found, 482.0520.



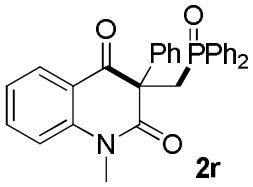
2p

Purified by column chromatography (ethyl acetate : methanol = 40 : 1). White solid, mp 225–226 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76–7.62 (m, 4H), 7.51–7.39 (m, 6H), 7.38 (s, 1H), 6.61 (s, 1H), 3.98 (s, 3H), 3.88 (s, 3H), 3.44 (s, 3H), 3.37–3.28 (m, 2H), 1.50 (d, J = 1.9 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.46 (d, $J_{C\text{-}P}$ = 1.6 Hz), 173.45 (d, $J_{C\text{-}P}$ = 1.9 Hz), 155.29, 144.89, 139.35, 134.36 (d, $J_{C\text{-}P}$ = 100.0 Hz), 134.19 (d, $J_{C\text{-}P}$ = 99.7 Hz), 131.29 (d, $J_{C\text{-}P}$ = 1.3 Hz), 130.79 (d, $J_{C\text{-}P}$ = 2.9 Hz), 130.69 (d, $J_{C\text{-}P}$ = 2.9 Hz), 128.20 (d, $J_{C\text{-}P}$ = 11.9 Hz), 112.11, 109.03, 98.14, 56.20, 56.05, 53.98 (d, $J_{C\text{-}P}$ = 4.0 Hz), 36.36 (d, $J_{C\text{-}P}$ = 69.6 Hz), 29.95, 28.07 (d, $J_{C\text{-}P}$ = 14.1 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 28.84. IR (film) υ_{max}: 1647, 1524, 1472, 1381, 1361, 1249, 1177, 1111, 1028, 741, 700, 537 cm^{-1}. HRMS calc. for $C_{26}H_{26}NO_5P$ $(M+H)^+$, 464.1621; found, 464.1622.



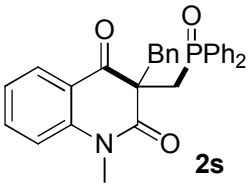
2q

Purified by column chromatography (petroleum ether : ethyl acetate = 2.5 : 1). White solid, mp 71–72 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.81 (d, J = 2.3 Hz, 1H), 7.78–7.66 (m, 4H), 7.60 (d, J = 2.4 Hz, 1H), 7.50 (dt, J = 14.1, 7.6 Hz, 6H), 3.59 (s, 3H), 3.34 (m, 2H), 1.43 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 194.97 (d, $J_{C\text{-}P}$ = 1.7 Hz), 174.12 (d, J = 1.6 Hz), 140.90, 136.93, 134.37 (d, $J_{C\text{-}P}$ = 100.8 Hz), 134.34 (d, $J_{C\text{-}P}$ = 101.4 Hz), 131.42 (d, $J_{C\text{-}P}$ = 2.6 Hz), 130.77 (d, $J_{C\text{-}P}$ = 9.9 Hz), 130.54 (d, $J_{C\text{-}P}$ = 9.7 Hz), 129.51, 128.41 (d, $J_{C\text{-}P}$ = 2.9 Hz), 128.29 (d, $J_{C\text{-}P}$ = 3.0 Hz), 126.57, 124.17, 123.42, 55.45 (d, J = 4.6 Hz), 38.30, 36.26 (d, $J_{C\text{-}P}$ = 69.3 Hz), 25.47 (d, $J_{C\text{-}P}$ = 13.3 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.67. IR (film) υ_{max}: 1707, 1673, 1588, 1475, 1430, 1331, 1175, 1122, 805, 741, 695, 549 cm^{-1}. HRMS calc. for $C_{24}H_{20}Cl_2NO_3P$ $(M+H)^+$, 472.0631; found, 472.0628.



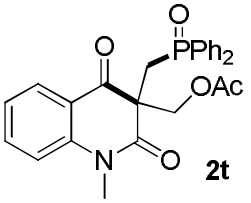
2r

Purified by column chromatography (dichloromethane : isopropanol = 40 : 1). White solid, mp 183–184 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.96 (dd, J = 7.8, 1.4 Hz, 1H), 7.85–7.69 (m, 4H), 7.56–7.39 (m, 7H), 7.29–7.16 (m, 5H), 7.08–7.04 (m, 2H), 3.61 (dd, J = 8.4, 0.9 Hz, 2H), 3.54 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.78, 171.19, 142.88, 136.78 (d, $J_{C\text{-}P}$ = 12.8 Hz), 135.42, 135.05 (d, $J_{C\text{-}P}$ = 100.5 Hz), 134.93 (d, $J_{C\text{-}P}$ = 98.9 Hz), 131.28, 130.90 (d, $J_{C\text{-}P}$ = 9.8 Hz), 130.77 (d, $J_{C\text{-}P}$ = 9.7 Hz), 129.04, 128.36, 128.27 (d, $J_{C\text{-}P}$ = 5.4 Hz), 128.11 (d, $J_{C\text{-}P}$ = 13.5 Hz), 126.70, 122.75, 120.94, 114.85, 64.30 (d, $J_{C\text{-}P}$ = 4.0 Hz), 37.98 (d, $J_{C\text{-}P}$ = 66.6 Hz), 30.28. ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.26. IR (film) υ_{max}: 1691, 1650, 1602, 1472, 1362, 1304, 1183, 1118, 900, 767, 695, 514 cm^{-1}. HRMS calc. for $C_{29}H_{24}NO_3P$ $(M+H)^+$, 466.1567; found, 466.1605.



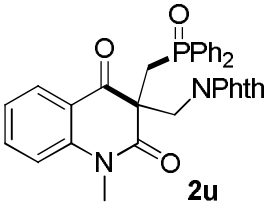
2s

Purified by column chromatography (dichloromethane : isopropanol = 50 : 1). White solid, mp 177–178 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (dd, *J* = 7.7, 1.4 Hz, 1H), 7.75–7.61 (m, 4H), 7.51–7.33 (m, 7H), 7.02–6.92 (m, 4H), 6.88–6.79 (m, 2H), 6.75 (d, *J* = 8.3 Hz, 1H), 3.44 (d, *J* = 9.2 Hz, 2H), 3.25–3.12 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.63 (d, $J_{C\text{-}P}$ = 1.5 Hz), 171.77 (d, $J_{C\text{-}P}$ = 1.9 Hz), 142.83, 135.31, 134.21 (d, $J_{C\text{-}P}$ = 100.1 Hz), 134.17 (d, $J_{C\text{-}P}$ = 100.1 Hz), 133.64 (d, $J_{C\text{-}P}$ = 2.2 Hz), 131.39 (d, $J_{C\text{-}P}$ = 2.7 Hz), 130.88 (d, $J_{C\text{-}P}$ = 9.8 Hz), 130.76 (d, $J_{C\text{-}P}$ = 9.8 Hz), 129.54, 128.31 (d, $J_{C\text{-}P}$ = 2.3 Hz), 128.19 (d, $J_{C\text{-}P}$ = 2.4 Hz), 127.44, 127.21 (d, $J_{C\text{-}P}$ = 13.4 Hz), 122.40, 121.36, 114.33, 59.53 (d, $J_{C\text{-}P}$ = 3.6 Hz), 49.01 (d, $J_{C\text{-}P}$ = 13.8 Hz), 36.96 (d, $J_{C\text{-}P}$ = 69.2 Hz), 29.40. ^{31}P NMR (162 MHz, $CDCl_3$) δ 28.79. IR (film) υ_{max}: 1678, 1643, 1602, 1474, 1369, 1293, 1177, 1120, 771, 739, 697, 550, 501 cm^{-1}. HRMS calc. for $C_{30}H_{26}NO_3P$ $(M+H)^+$, 480.1723; found, 480.1755.



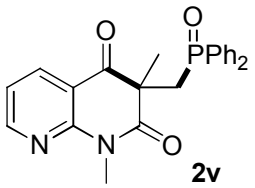
2t

Purified by column chromatography (dichloromethane : isopropanol = 40 : 1). White solid, mp 162–163 ℃. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.86 (d, *J* = 7.7 Hz, 1H), 7.74–7.57 (m, 5H), 7.53–7.35 (m, 6H), 7.20–7.07 (m, 2H), 4.48 (d, *J* = 10.2 Hz, 1H), 4.39 (d, *J* = 10.2 Hz, 1H), 3.39 (s, 3H), 3.32–3.18 (m, 2H), 1.77 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 193.55, 170.19, 169.59, 143.34, 135.97, 133.56 (d, $J_{C\text{-}P}$ = 100.9 Hz), 133.48 (d, $J_{C\text{-}P}$ = 100.6 Hz), 131.60, 130.76 (d, $J_{C\text{-}P}$ = 10.4 Hz), 130.67 (d, $J_{C\text{-}P}$ = 10.1 Hz), 128.35 (d, $J_{C\text{-}P}$ = 11.9 Hz), 127.32, 122.85, 120.85, 114.82, 70.23 (d, $J_{C\text{-}P}$ = 17.2 Hz), 57.33, 33.83 (d, $J_{C\text{-}P}$ = 69.7 Hz), 29.96, 20.24. ^{31}P NMR (202 MHz, $CDCl_3$) δ 28.53. IR (film) υ_{max}: 1745, 1695, 1657, 1600, 1472, 1374, 1226, 1181, 1116, 1044, 750, 701, 547, 502 cm^{-1}. HRMS calc. for $C_{26}H_{24}NO_5P$ $(M+H)^+$, 462.1465; found, 462.1466.



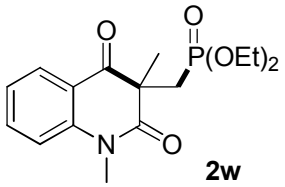
2u

Purified by column chromatography (dichloromethane : isopropanol = 40 : 1). White solid, mp 114–115 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (dd, *J* = 7.7, 1.6 Hz, 1H), 7.82–7.74 (m, 2H), 7.73–7.63 (m, 6H), 7.62–7.54 (m, 1H), 7.52–7.36 (m, 6H), 7.16 (dd, *J* = 14.4, 7.6 Hz, 2H), 4.00 (ddd, *J* = 14.8, 13.9, 1.6 Hz, 2H), 3.52 – 3.42 (m, 5H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 193.23 (d, $J_{C\text{-}P}$ = 1.7 Hz), 170.01 (d, $J_{C\text{-}P}$ = 1.8 Hz), 167.72, 143.28, 135.90, 134.45 (d, $J_{C\text{-}P}$ = 101.1 Hz), 134.37 (d, $J_{C\text{-}P}$ = 101.3 Hz), 134.18, 131.56, 131.40 (d, $J_{C\text{-}P}$ = 1.7 Hz), 130.90 (d, $J_{C\text{-}P}$ = 10.1 Hz), 130.80 (d, $J_{C\text{-}P}$ = 10.0 Hz), 128.69, 128.35 (d, $J_{C\text{-}P}$ = 3.9 Hz), 128.23 (d, $J_{C\text{-}P}$ = 4.0 Hz), 123.54, 122.84, 119.89, 114.94, 60.17 (d, $J_{C\text{-}P}$ = 3.9 Hz), 44.09 (d, $J_{C\text{-}P}$ = 14.3 Hz), 33.07 (d, $J_{C\text{-}P}$ = 69.5 Hz), 30.23. ^{31}P NMR (162 MHz, $CDCl_3$) δ 30.57. IR (film) υ_{max}: 1651, 1550, 1464, 1390, 1113, 754, 551 cm^{-1}. HRMS calc. for $C_{32}H_{25}N_2O_5P$ $(M+ Na)^+$, 571.1393; found, 571.1399.



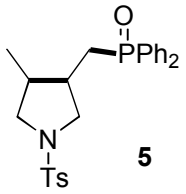
2v

Purified by column chromatography (dichloromethane : isopropanol = 40 : 1). White solid, mp 281–282 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.58 (dd, *J* = 4.7, 1.8 Hz, 1H), 8.24 (dd, *J* = 7.6, 1.8 Hz, 1H), 7.77–7.61 (m, 4H), 7.54–7.39 (m, 6H), 7.09 (dd, *J* = 7.6, 4.8 Hz, 1H), 3.55 (s, 3H), 3.46–3.29 (m, 2H), 1.51 (d, *J* = 1.8 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.46 (d, *J* = 1.6 Hz), 173.72 (d, *J* = 1.9 Hz), 154.12, 153.76, 136.77, 134.17 (d, *J* = 100.3 Hz), 133.83 (d, *J* = 100.5 Hz), 131.48 (d, *J* = 2.5 Hz), 130.82 (d, *J* = 9.9 Hz), 130.64 (d, *J* = 9.8 Hz), 128.38 (d, *J* = 5.3 Hz), 128.26 (d, *J* = 5.3 Hz), 118.54, 114.75, 54.98 (d, *J* = 4.1 Hz), 36.31 (d, *J* = 69.0 Hz), 28.77, 27.32 (d, *J* = 13.8 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 29.21. IR (film) υ_{max}: 1701, 1666, 1588, 1448, 1389, 1325, 1181, 1116, 780, 742, 701, 548, 498 cm^{-1}. HRMS calc. for $C_{23}H_{21}N_2O_3P$ $(M+H)^+$, 405.1363; found, 405.1363.



2w

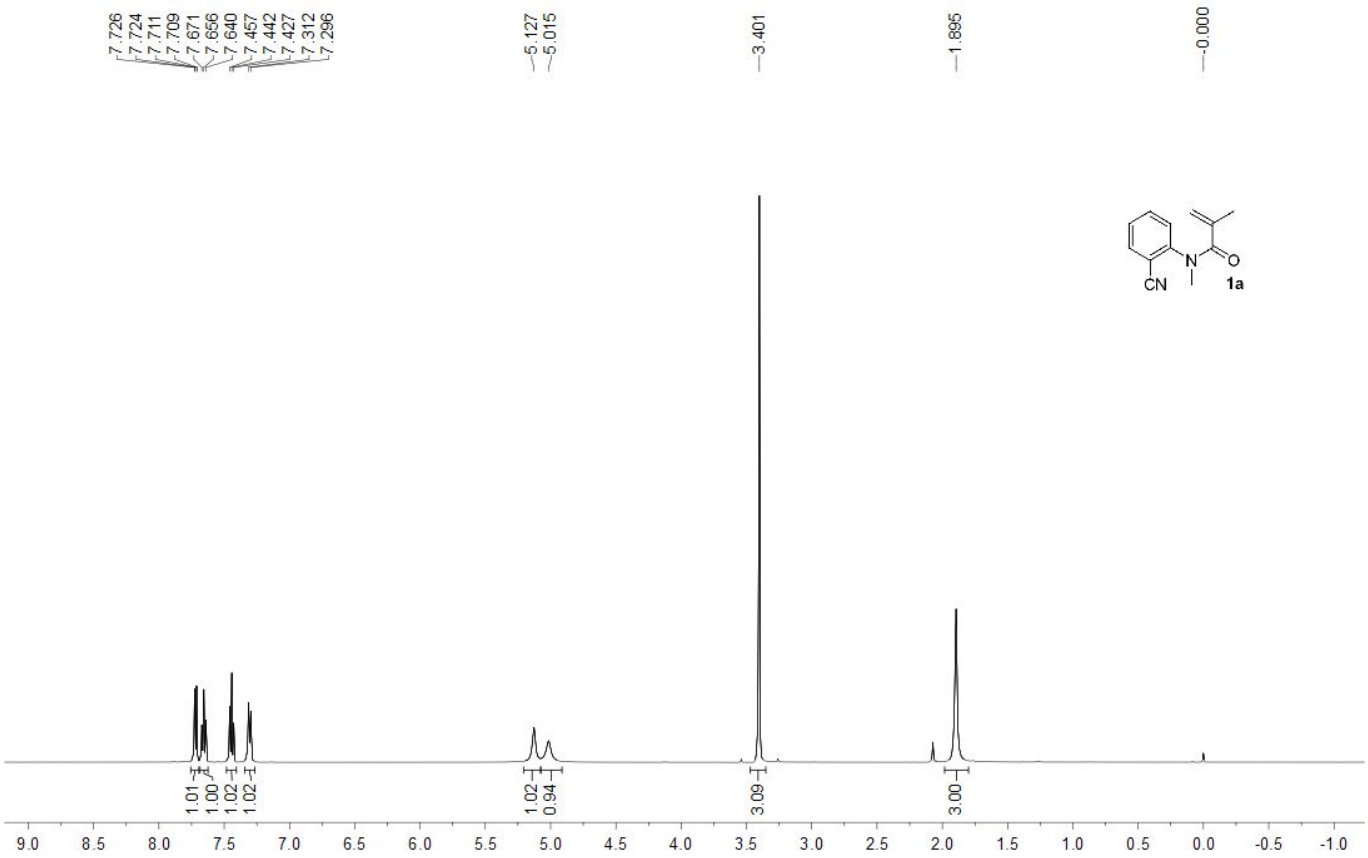
Purified by column chromatography (petroleum ether : ethyl acetate = 5 : 1). White solid, mp 133–134 ℃. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.06 (d, *J* = 7.3 Hz, 1H), 7.63 (t, *J* = 7.3 Hz, 1H), 7.19 (d, *J* = 7.6 Hz, 2H), 4.16–3.84 (m, 4H), 3.51 (s, 3H), 2.76 (d, *J* = 17.1 Hz, 2H), 1.45 (s, 3H), 1.22 (d, *J* = 6.1 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 195.89, 173.00 (d, $J_{C\text{-}P}$ = 1.9 Hz), 143.13, 135.95, 128.33, 122.86, 119.41, 114.79, 61.65 (d, $J_{C\text{-}P}$ = 6.1 Hz), 61.53 (d, $J_{C\text{-}P}$ = 6.0 Hz), 54.40 (d, $J_{C\text{-}P}$ = 3.2 Hz), 32.55 (d, $J_{C\text{-}P}$ = 138.1 Hz), 29.88, 27.62 (d, $J_{C\text{-}P}$ = 18.8 Hz), 16.17 (d, $J_{C\text{-}P}$ = 3.7 Hz), 16.11 (d, $J_{C\text{-}P}$ = 3.7 Hz). ^{31}P NMR (162 MHz, $CDCl_3$) δ 27.89. IR (film) υ_{max}: 1696, 1659, 1605, 1474, 1353, 1235, 1057, 1028, 974, 809, 765 cm^{-1}. HRMS calc. for $C_{16}H_{22}NO_5P$ $(M+H)^+$, 340.1308; found, 340.1349.

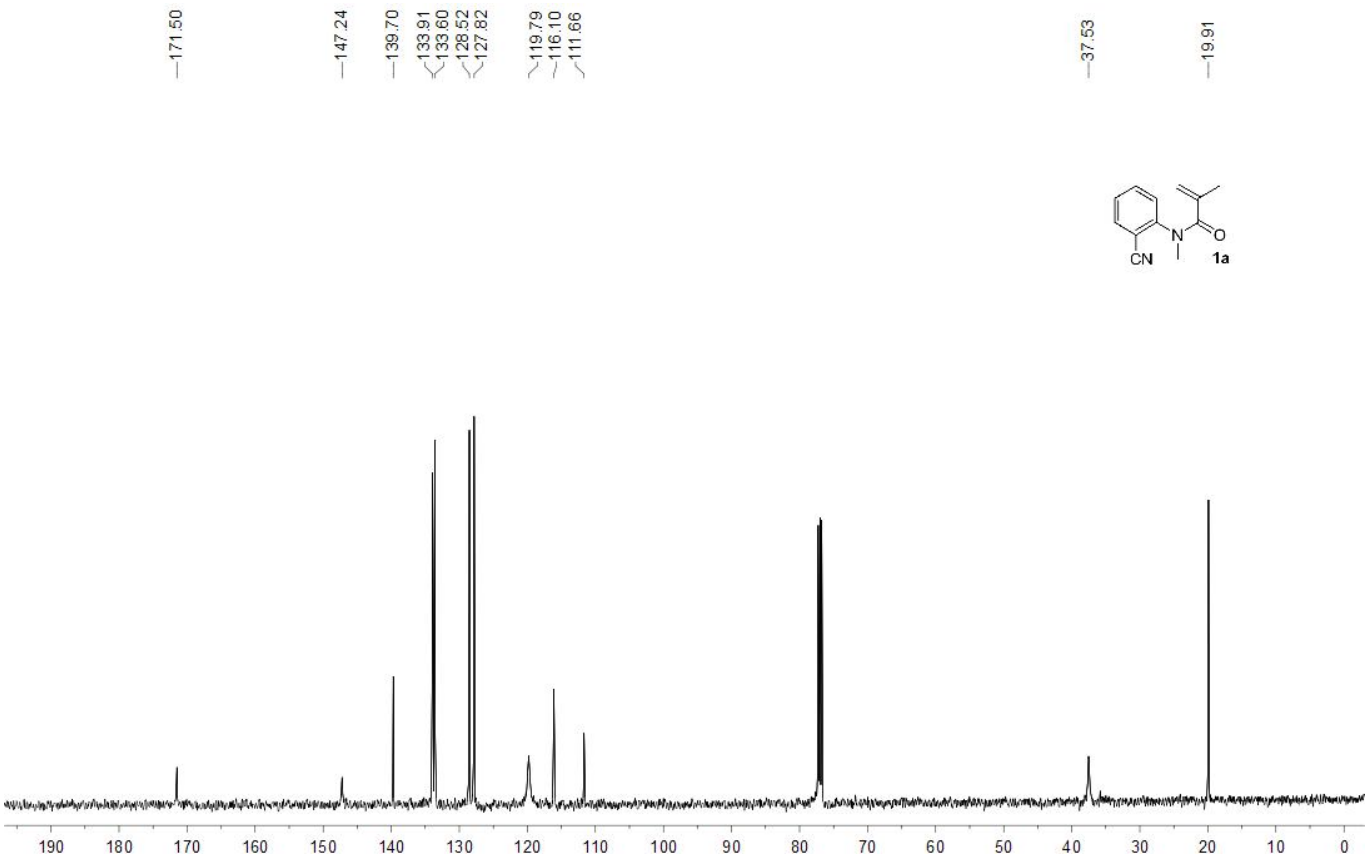


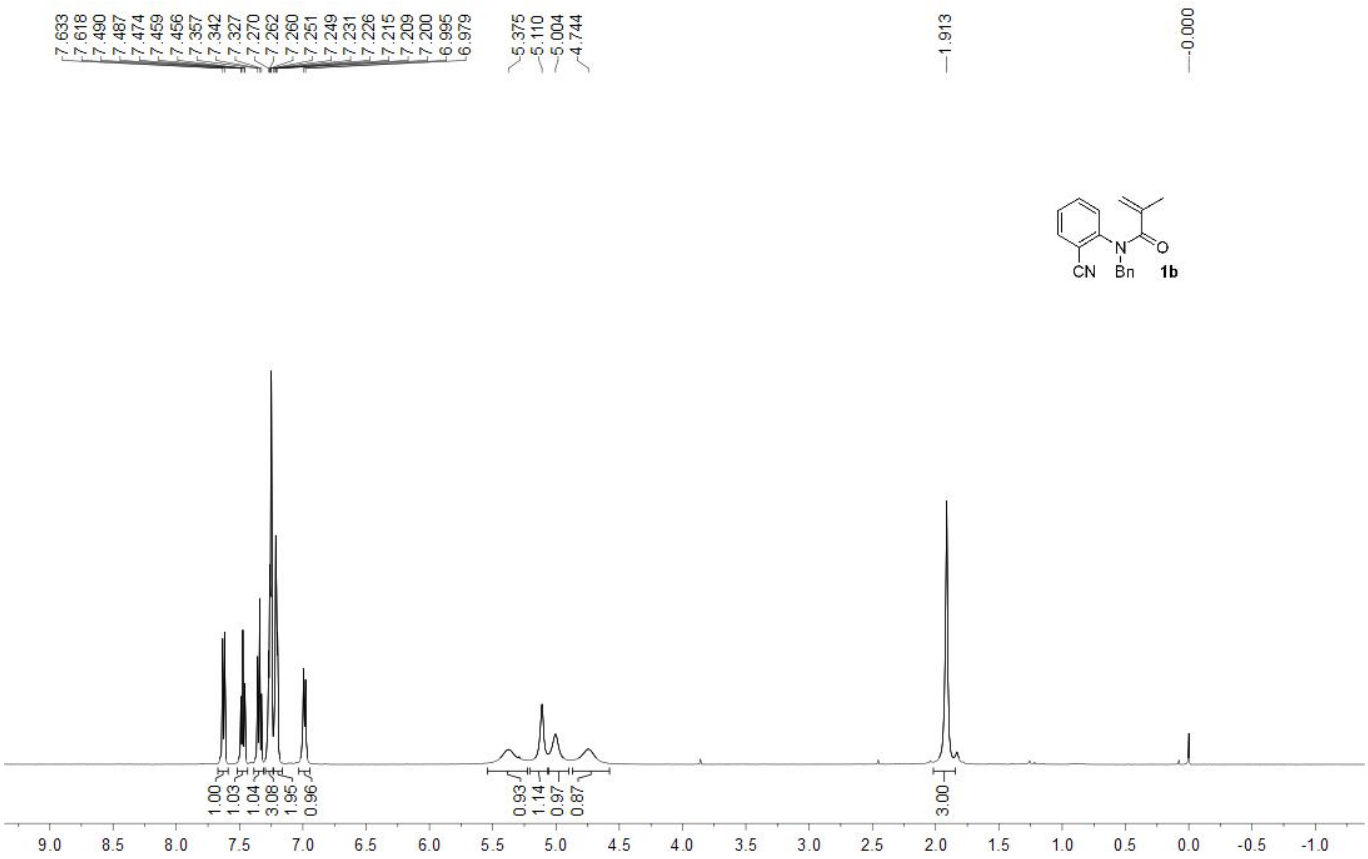
5

Purified by column chromatography (petroleum ether : ethyl acetate = 1 : 3). Product **4** is inseparable mixture. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.76–7.42 (m, 12H), 7.28–7.26 (m, 2H), 3.55–3.20 (m, 2H), 3.09–2.62 (m, 2H), 2.47–2.29 (m, 4H), 2.30–1.75 (m, 3H), 0.94–0.68 (m, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 143.27, 143.24, 133.63, 133.38, 132.78 (d, $J_{C\text{-}P}$ = 98.5 Hz), 132.64 (d, $J_{C\text{-}P}$ = 98.6 Hz), 132.55 (d, $J_{C\text{-}P}$ = 99.0 Hz), 131.98 (d, $J_{C\text{-}P}$ = 2.7 Hz), 131.87 (d, $J_{C\text{-}P}$ = 2.6 Hz), 130.54 (d, $J_{C\text{-}P}$ = 9.3 Hz), 129.54, 128.76 (d, $J_{C\text{-}P}$ = 2.3 Hz), 128.65 (d, $J_{C\text{-}P}$ = 2.3 Hz), 127.38 (d, $J_{C\text{-}P}$ = 7.6 Hz), 54.07, 53.65, 53.45 (d, $J_{C\text{-}P}$ = 3.1 Hz), 51.39 (d, $J_{C\text{-}P}$ = 6.0 Hz), 40.24 (d, $J_{C\text{-}P}$ = 12.7 Hz), 39.62 (d, $J_{C\text{-}P}$ = 3.9 Hz), 36.11 (d, $J_{C\text{-}P}$ = 9.6 Hz), 35.71 (d, $J_{C\text{-}P}$ = 3.4 Hz), 32.17 (d, $J_{C\text{-}P}$ = 71.2 Hz), 28.05 (d, $J_{C\text{-}P}$ = 71.7 Hz), 21.45, 15.69, 13.22. ^{31}P NMR (162 MHz, $CDCl_3$) δ 30.47, 29.92. IR (film) υ_{max}: 1601, 1442, 1338, 1161, 1105, 814, 751, 706, 663, 588, 540 cm^{-1}. HRMS calc. for $C_{25}H_{28}NO_3PS$ $(M+H)^+$, 454.1600; found, 454.1603.

6. Charts of new compounds



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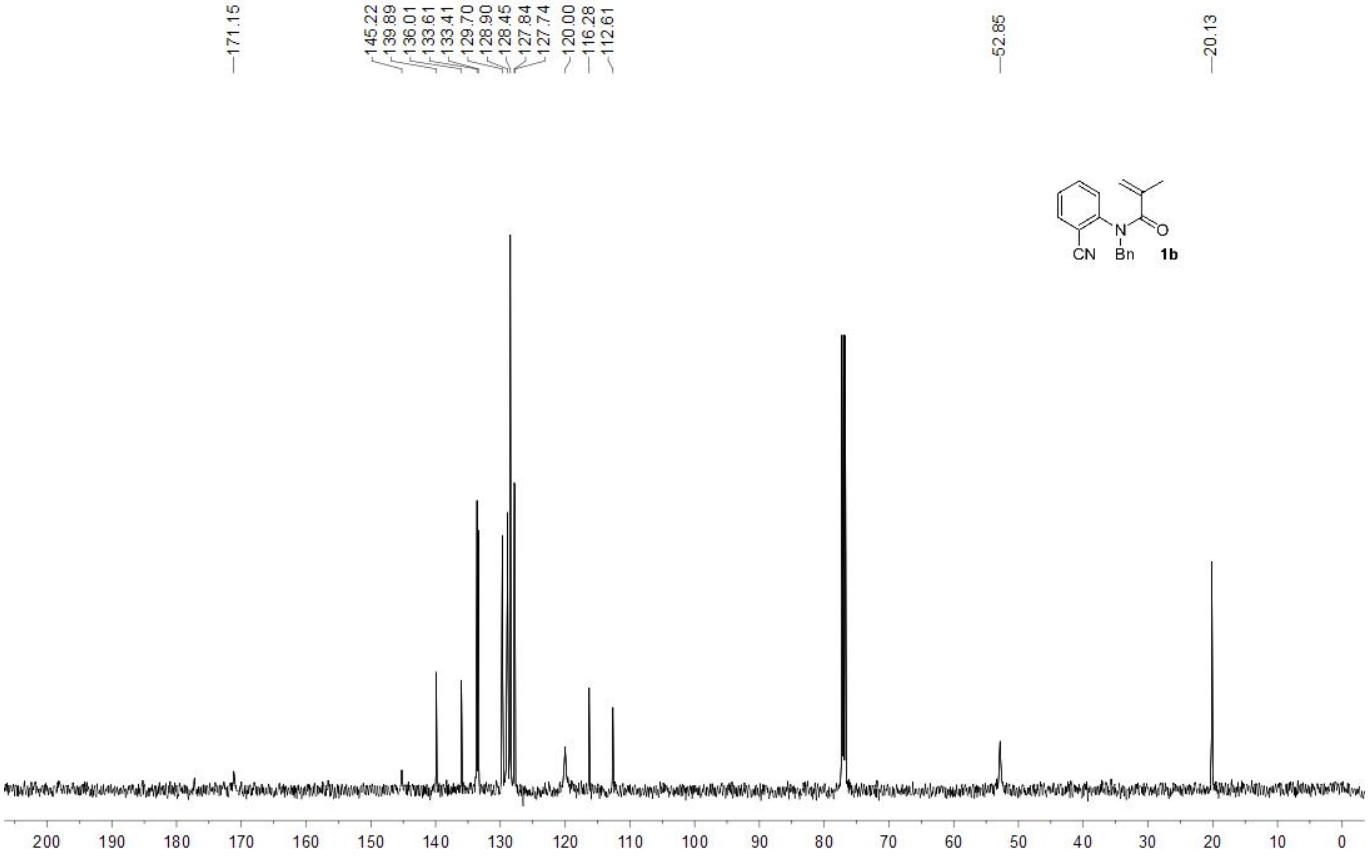
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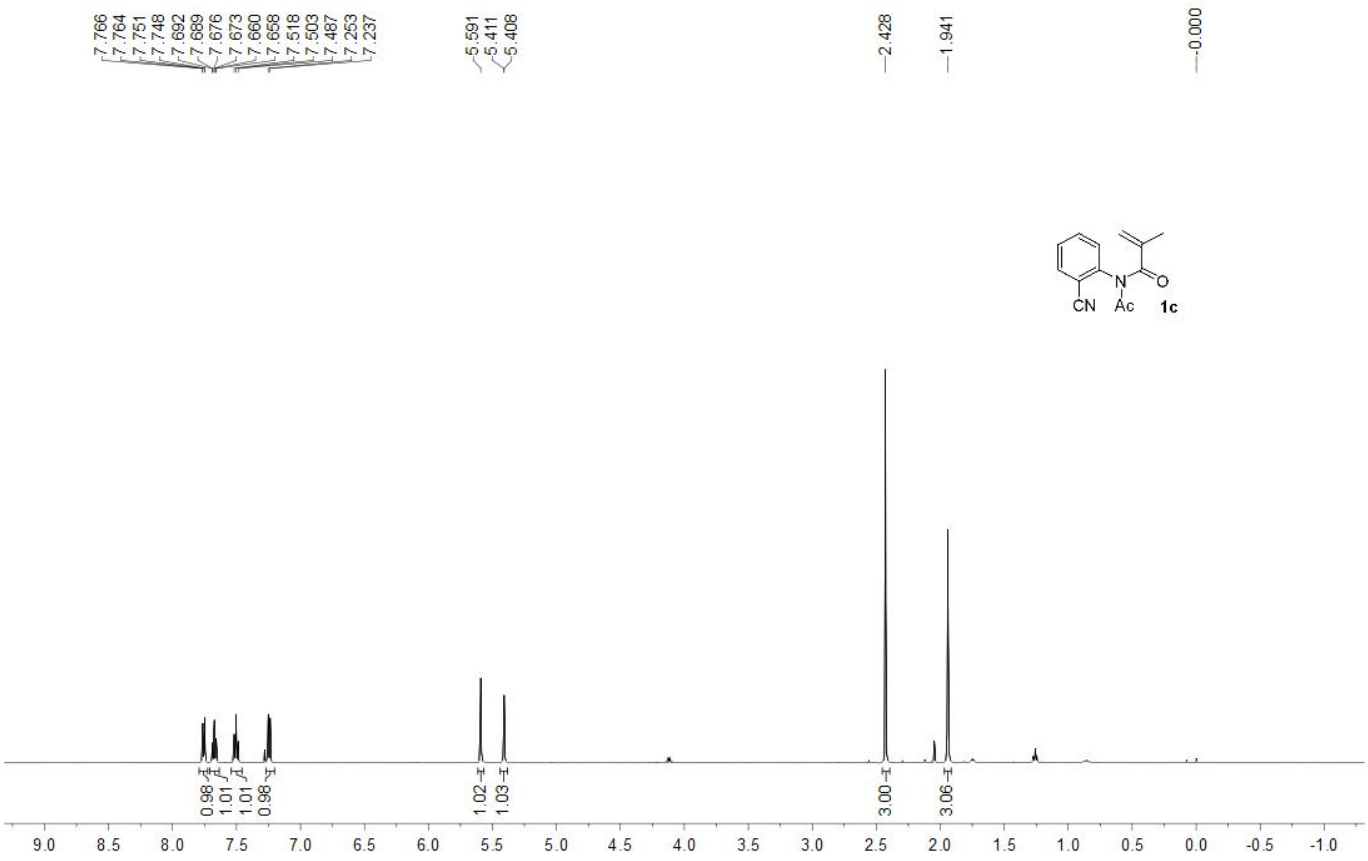
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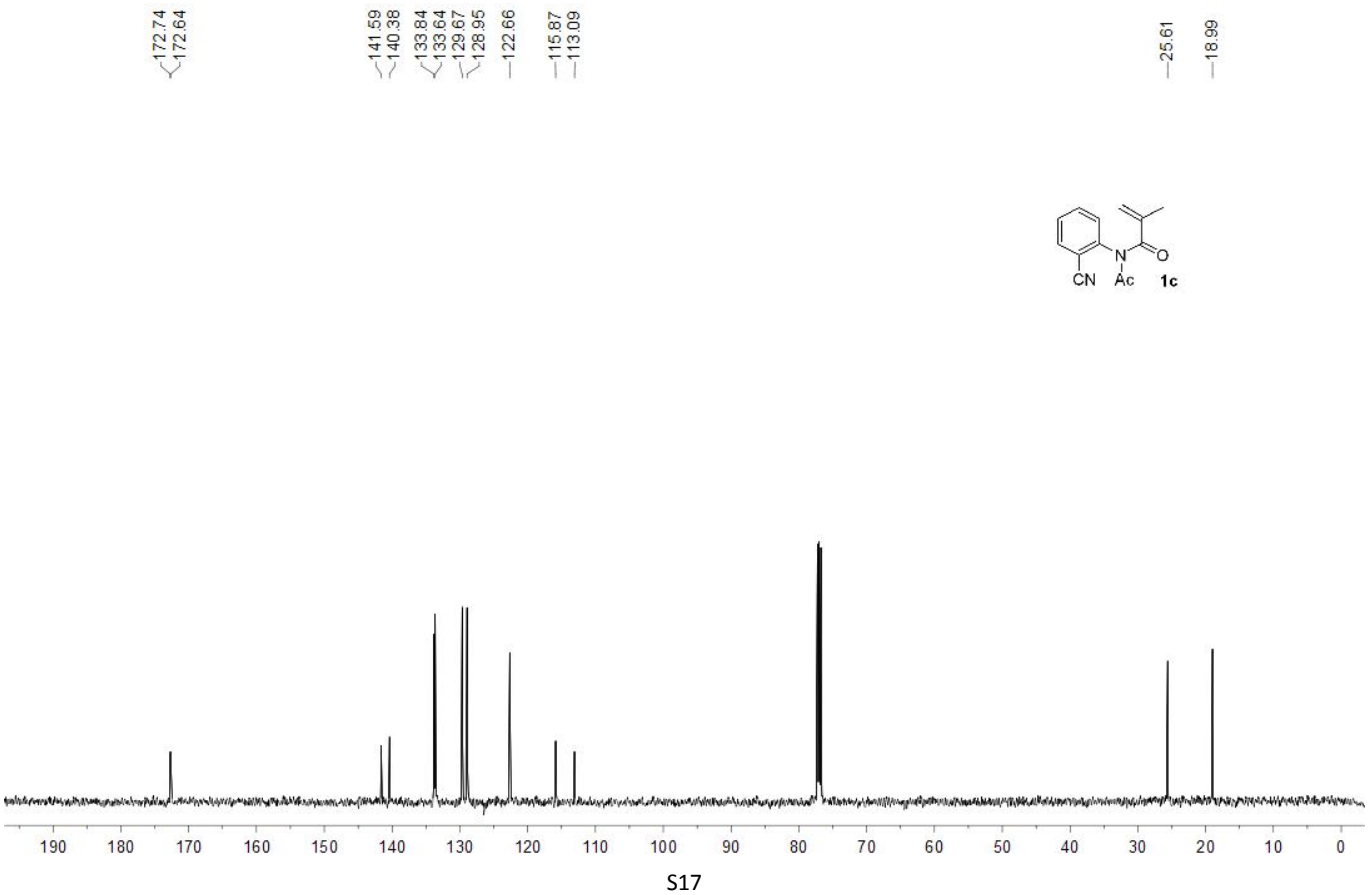
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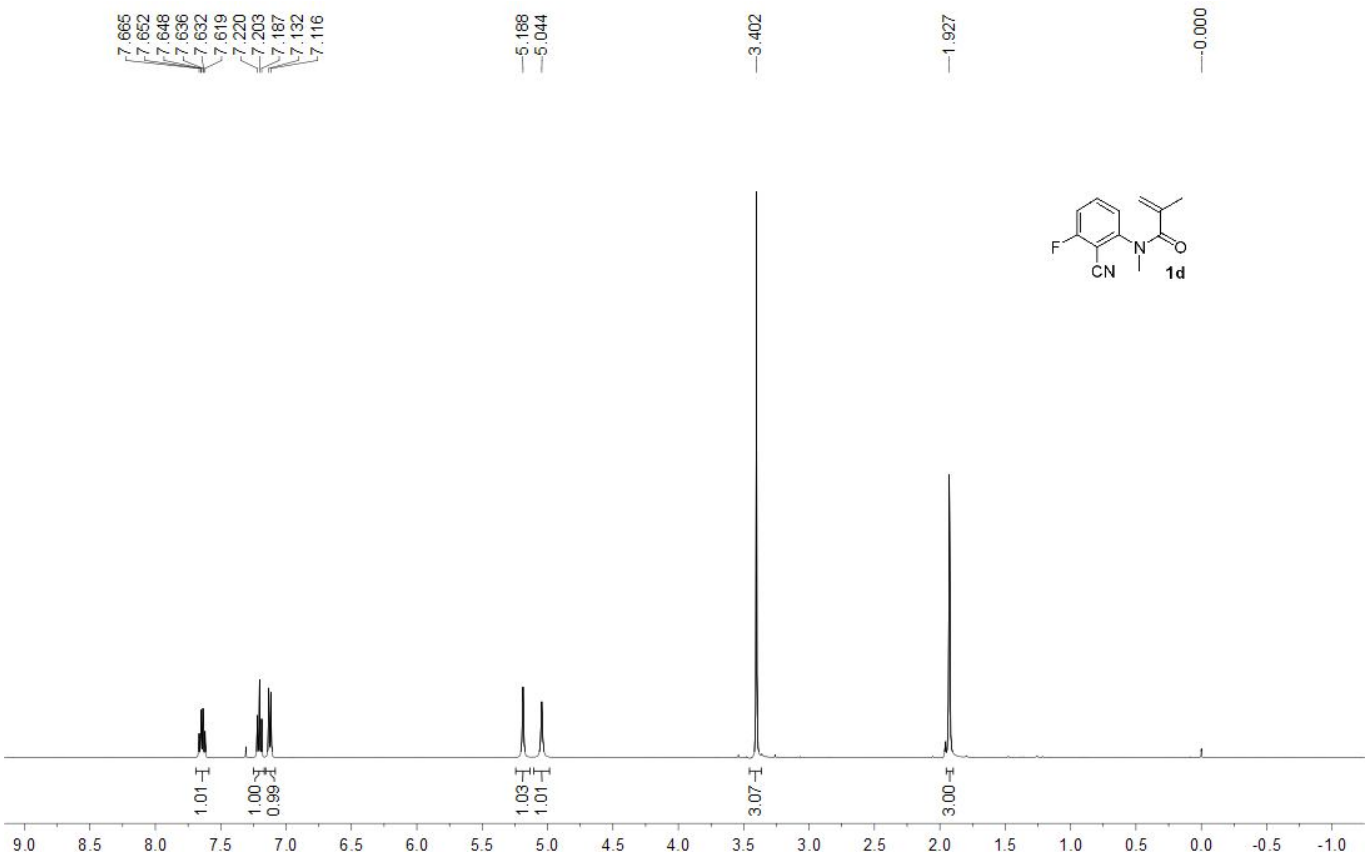
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3.00
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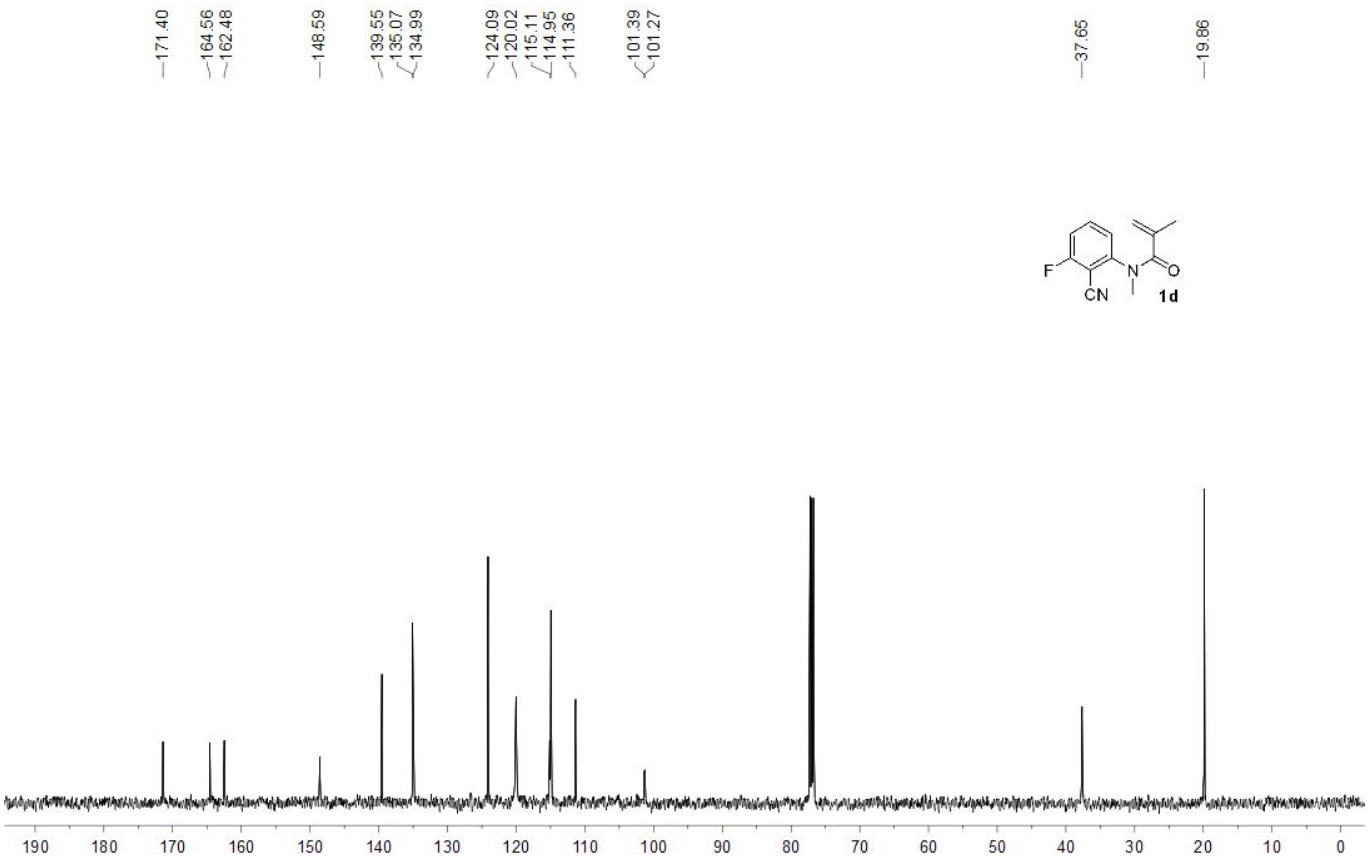
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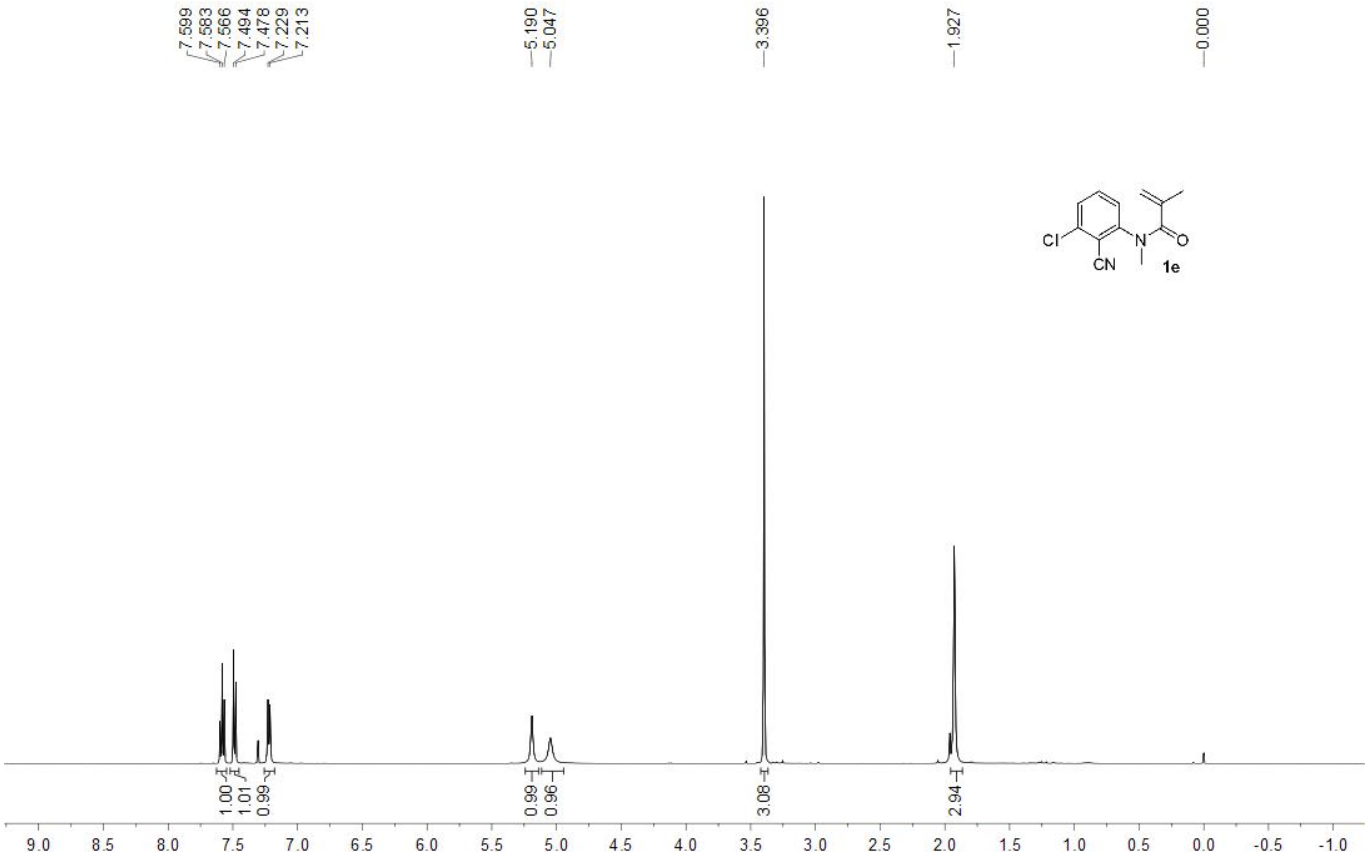
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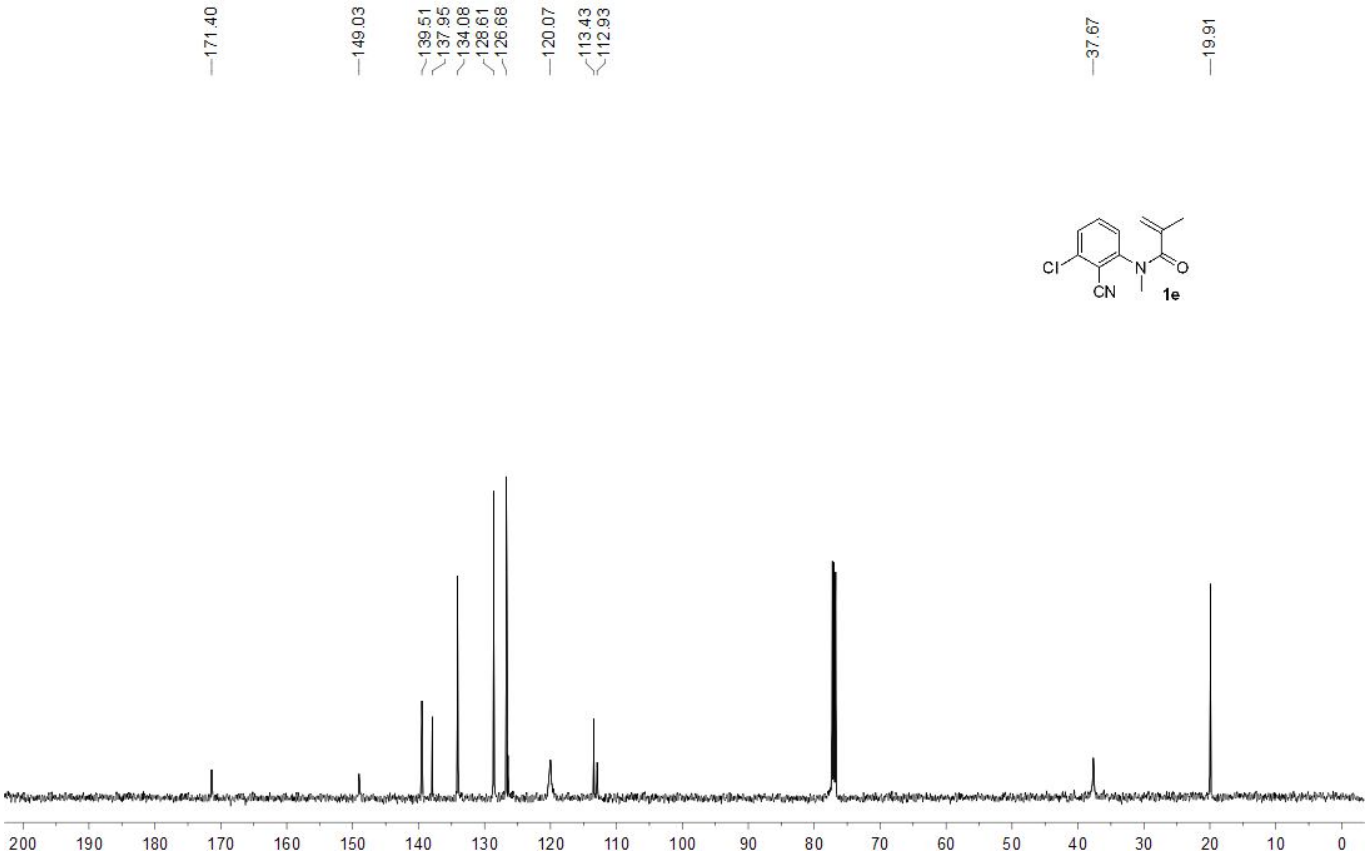
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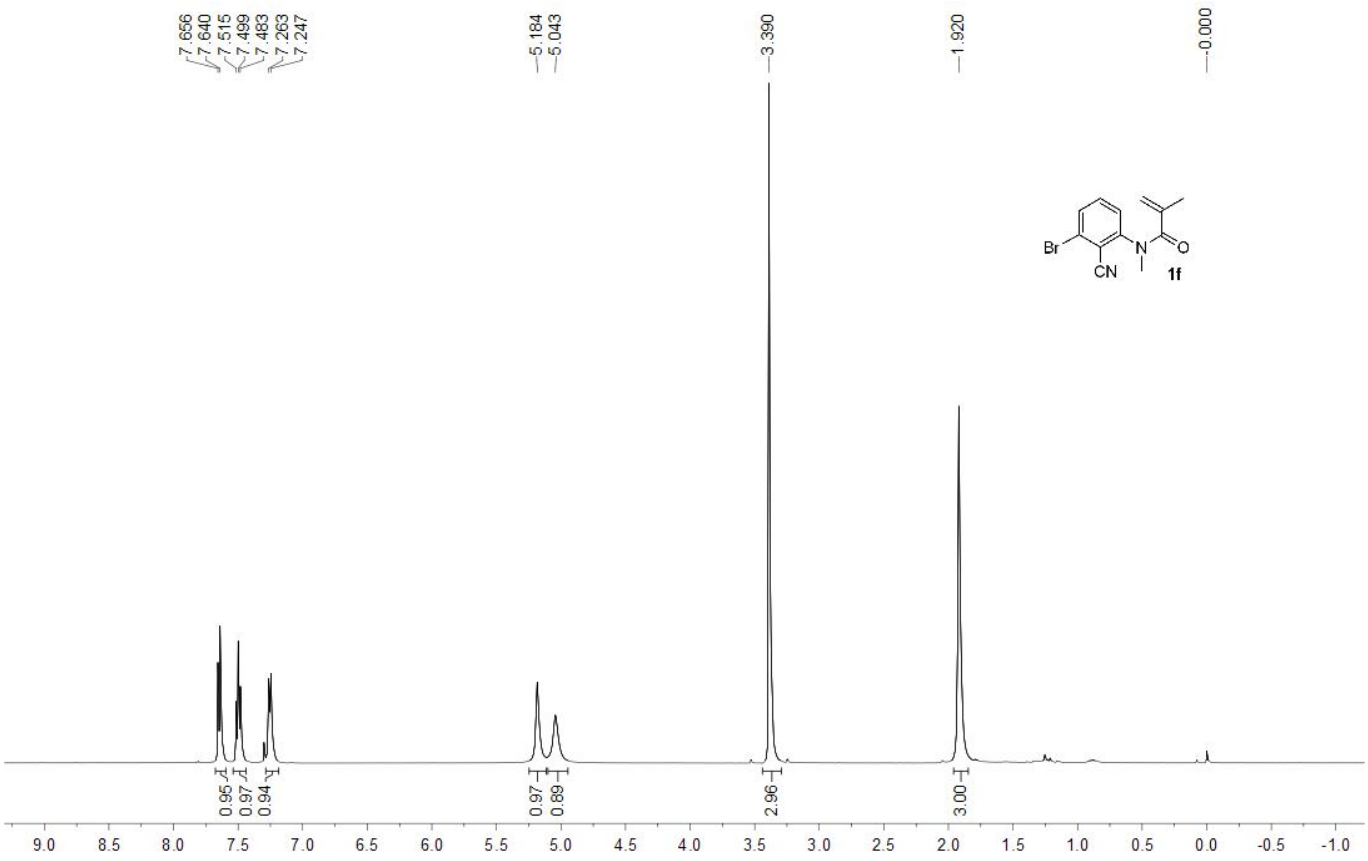
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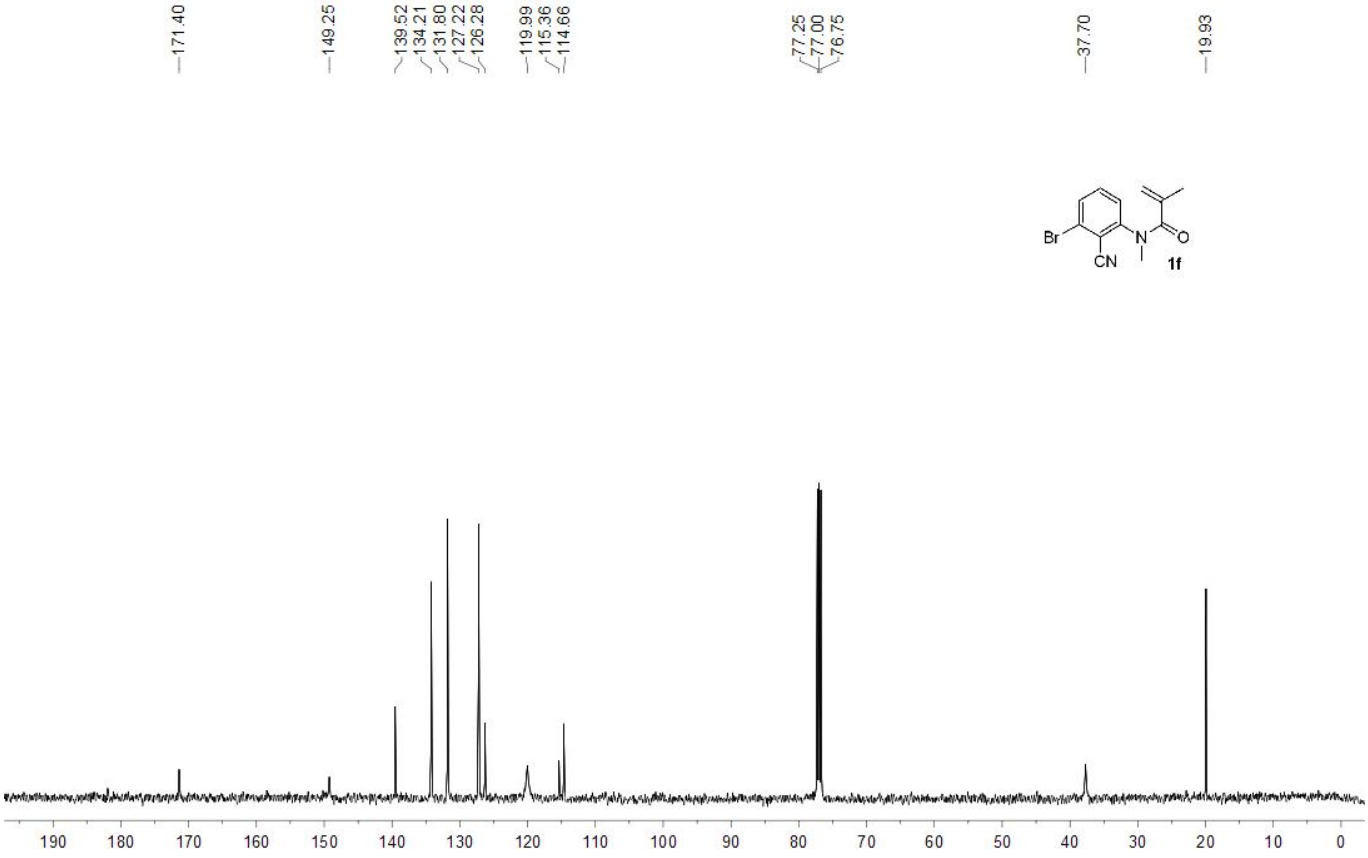
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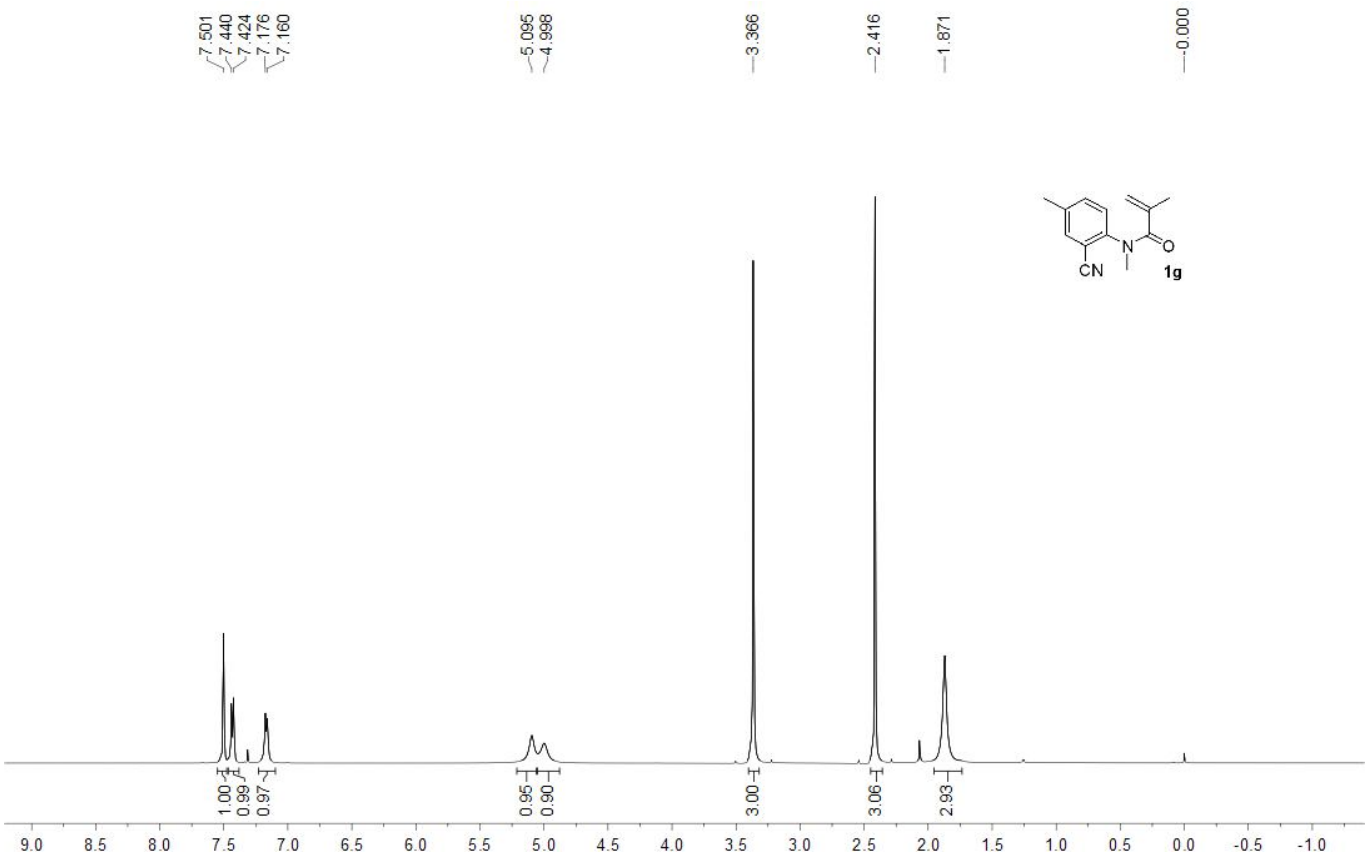
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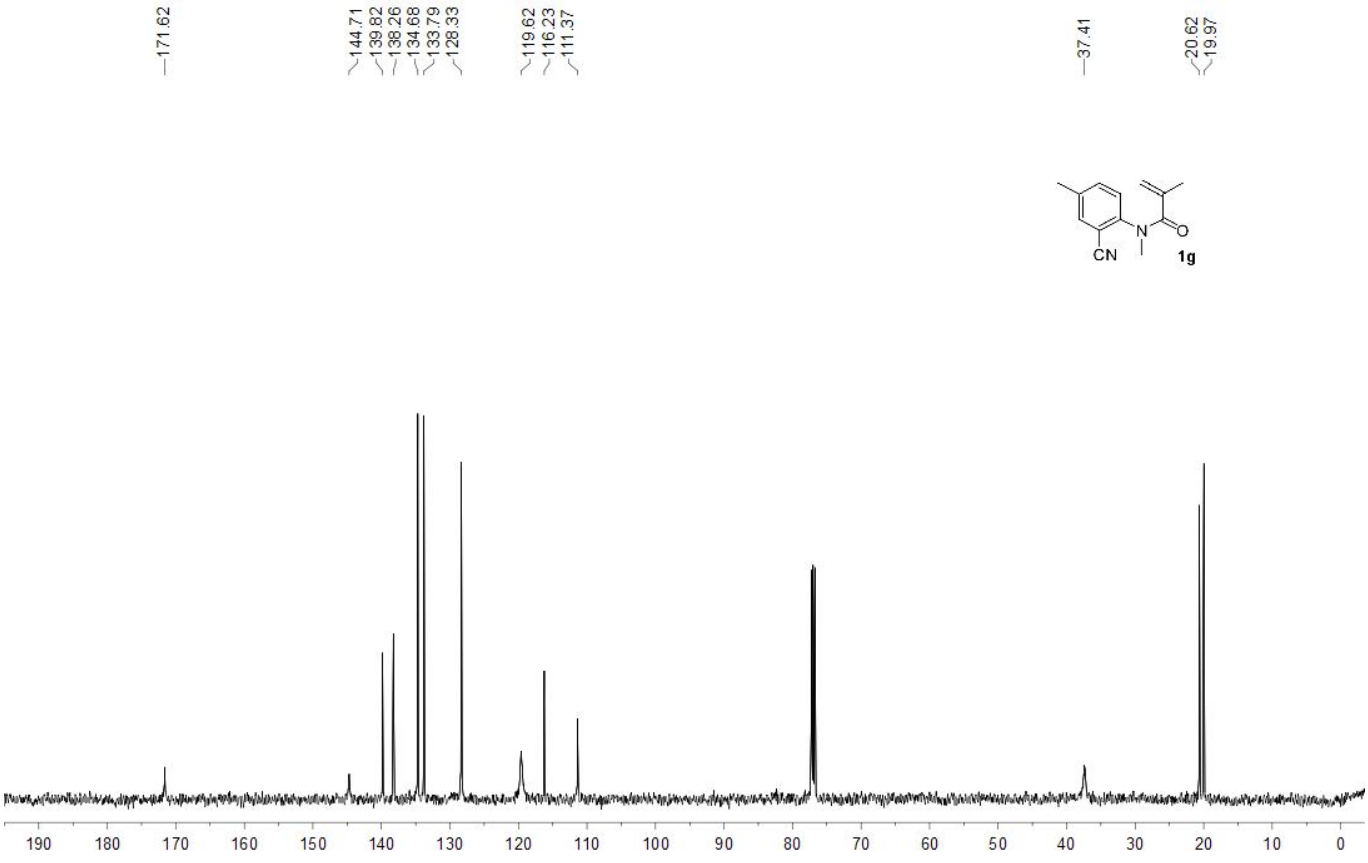
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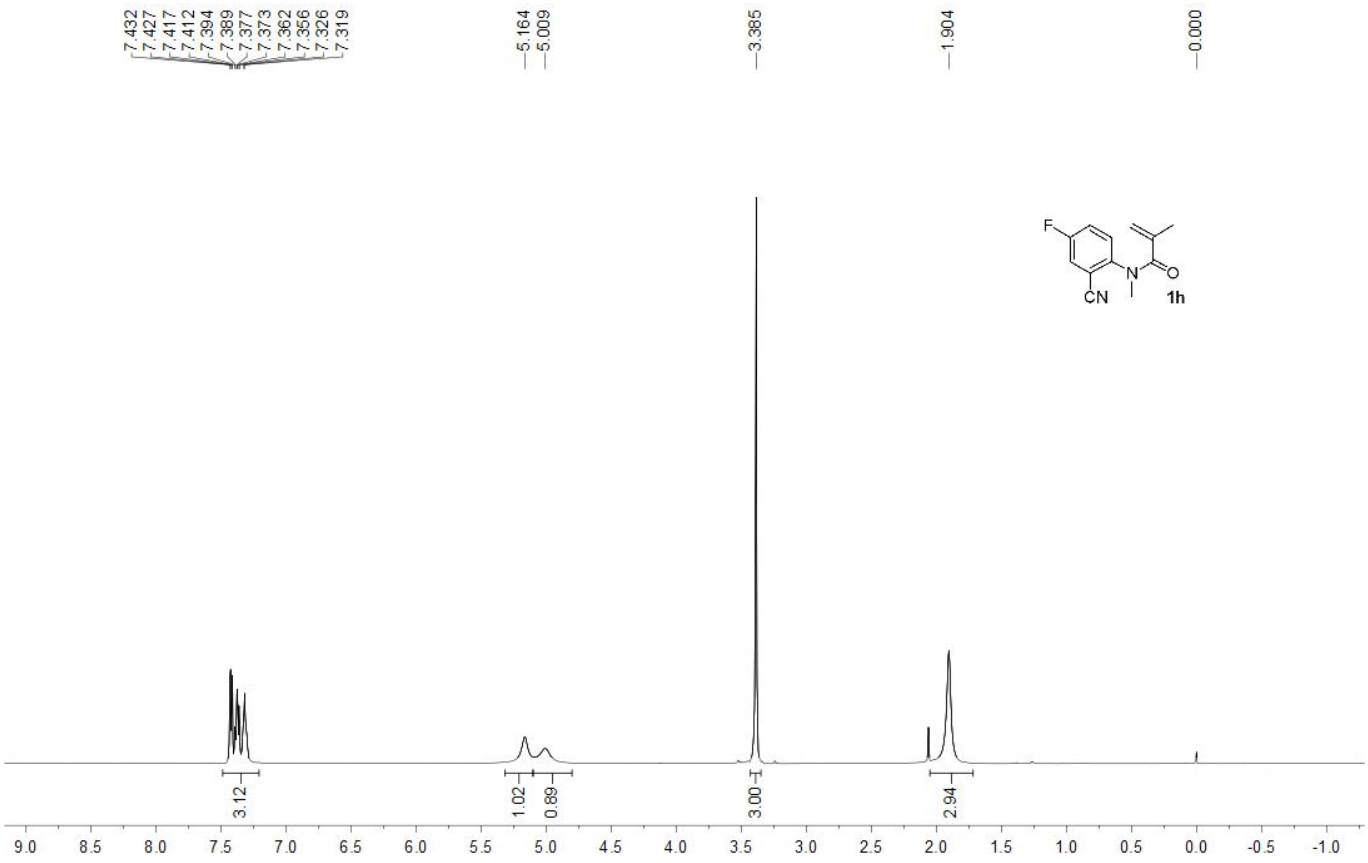
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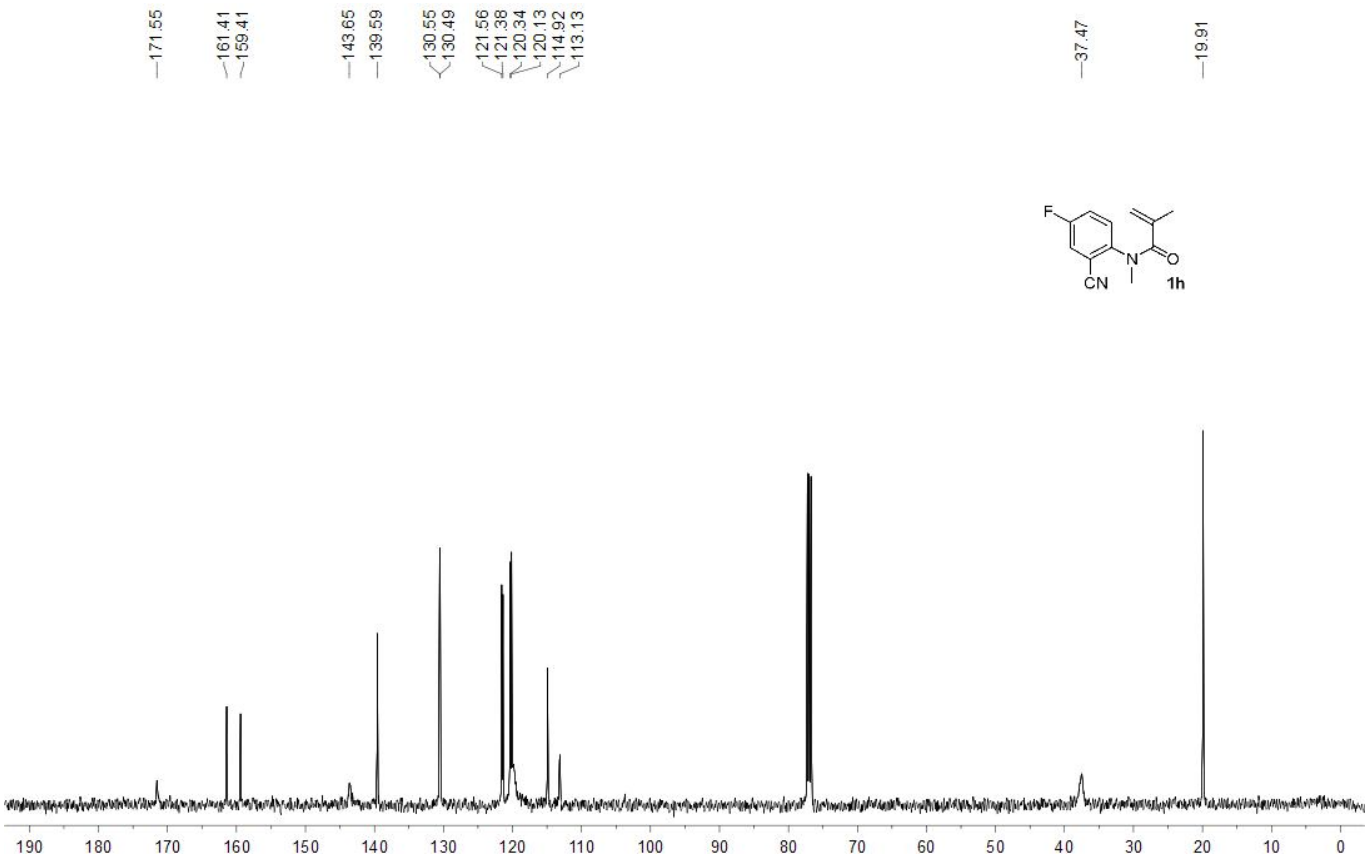
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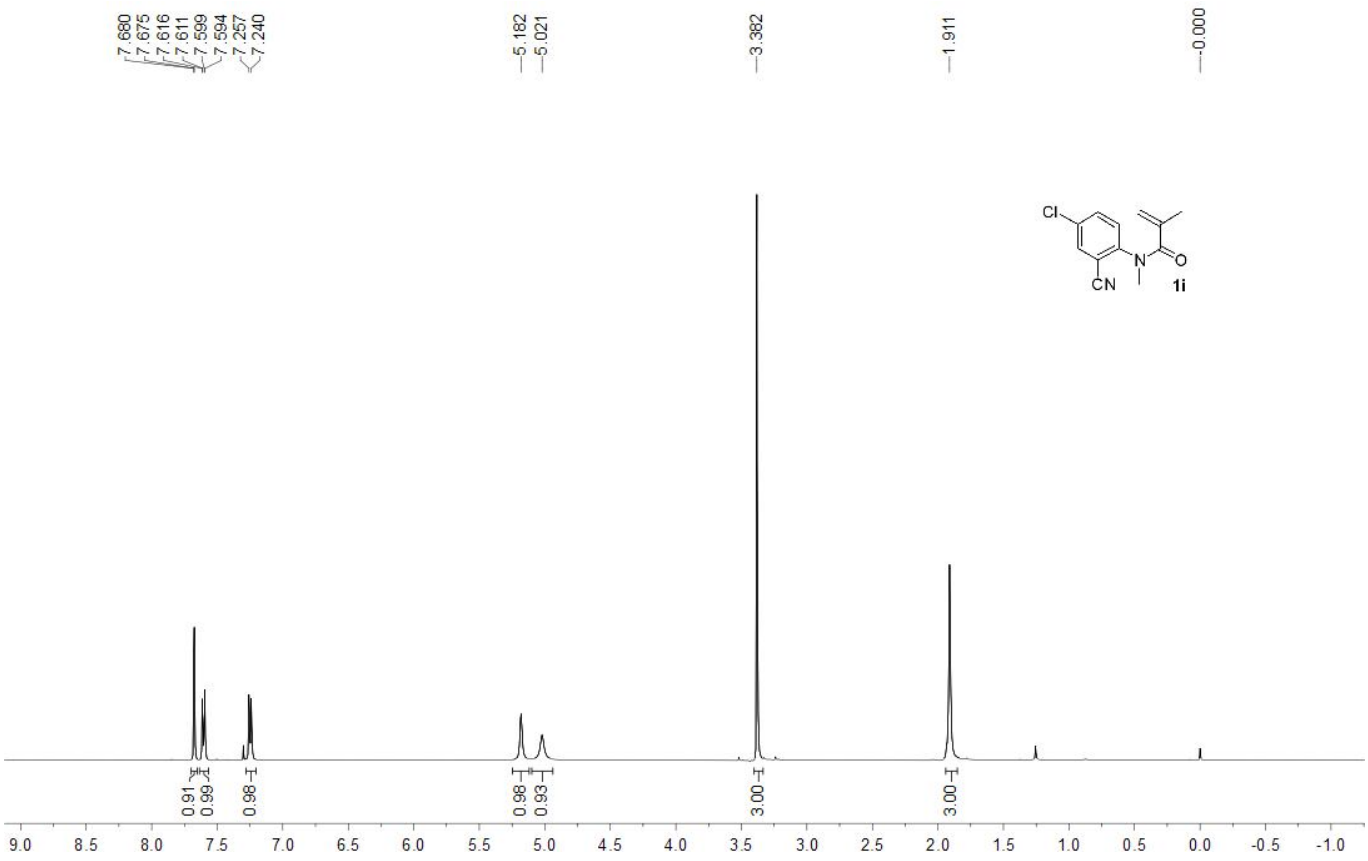
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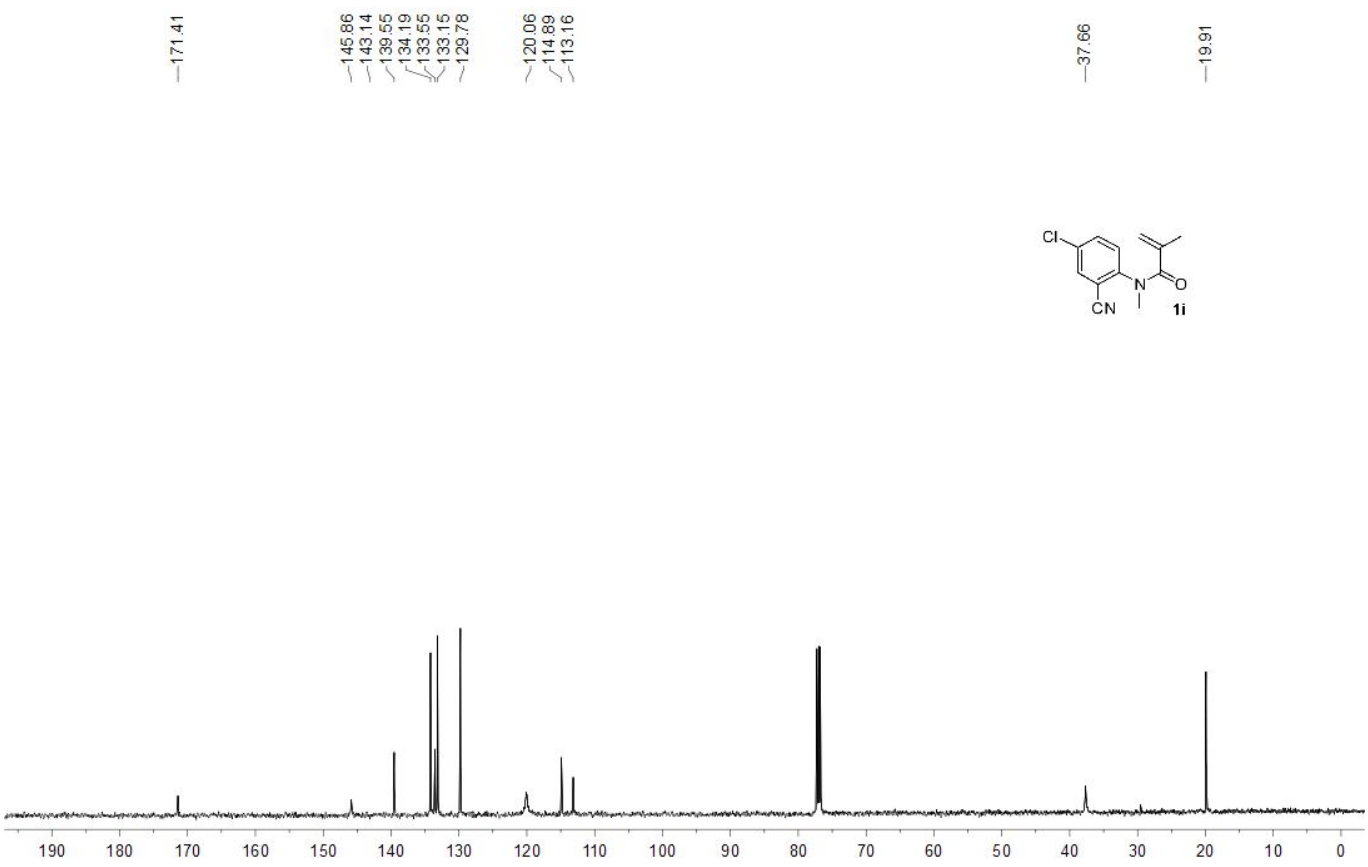
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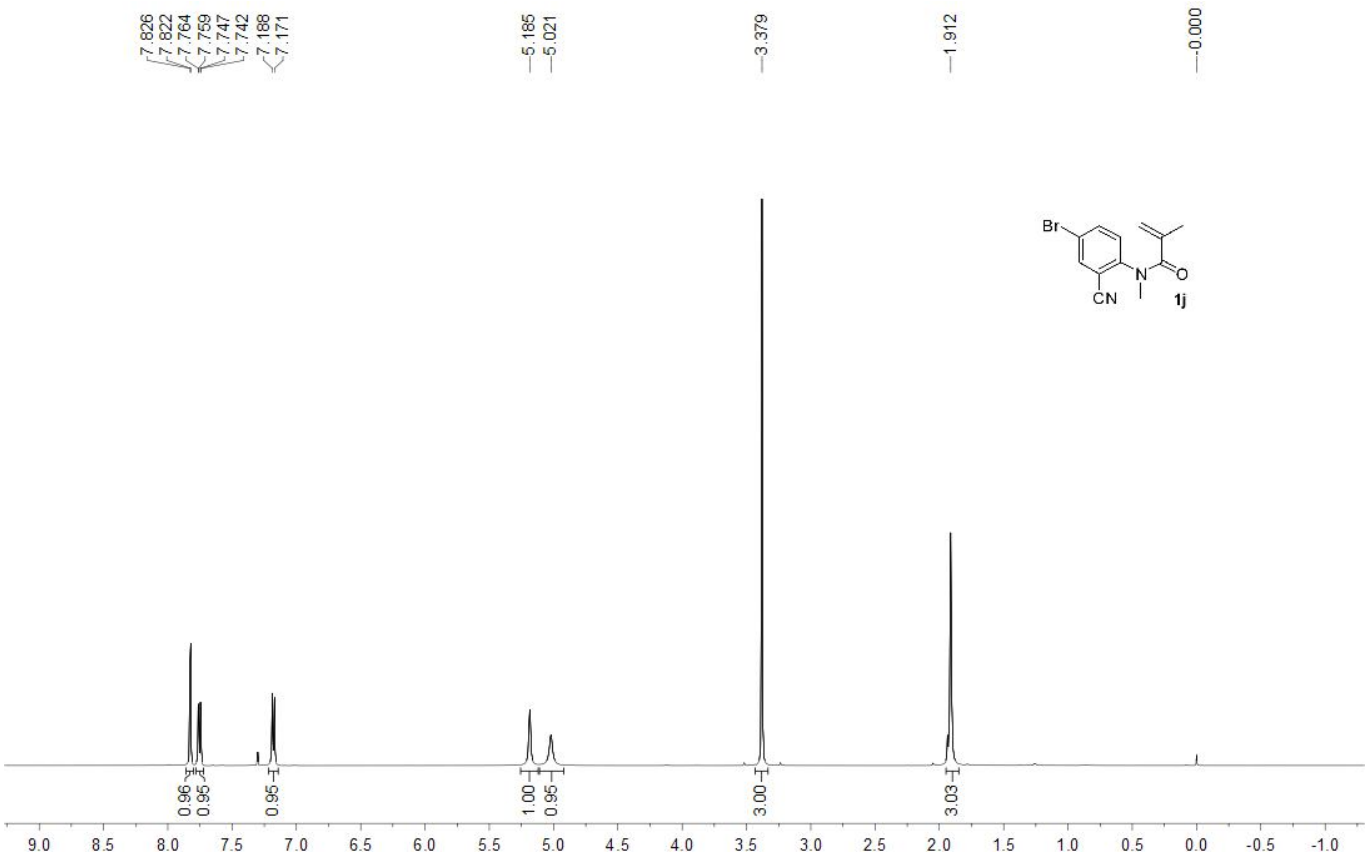
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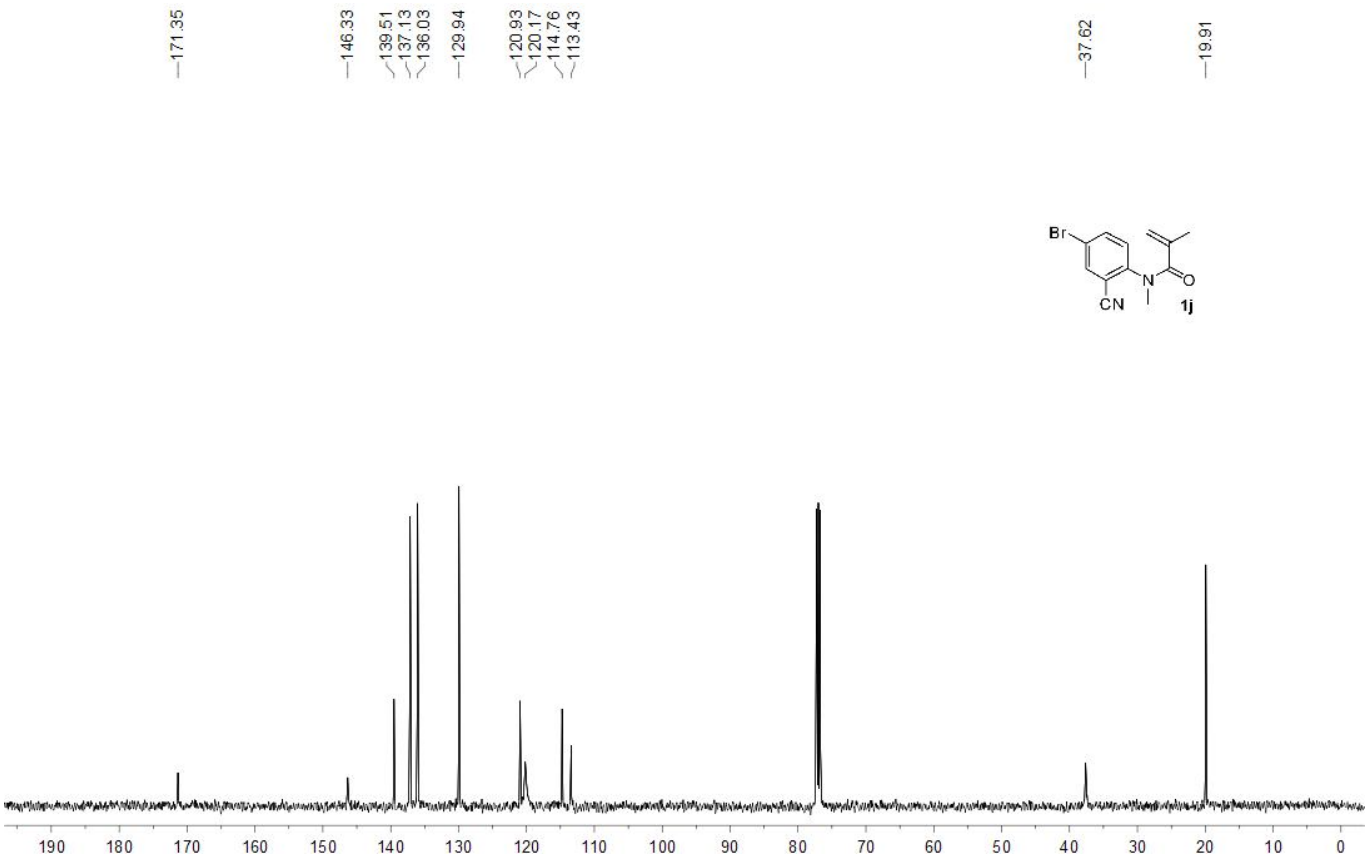
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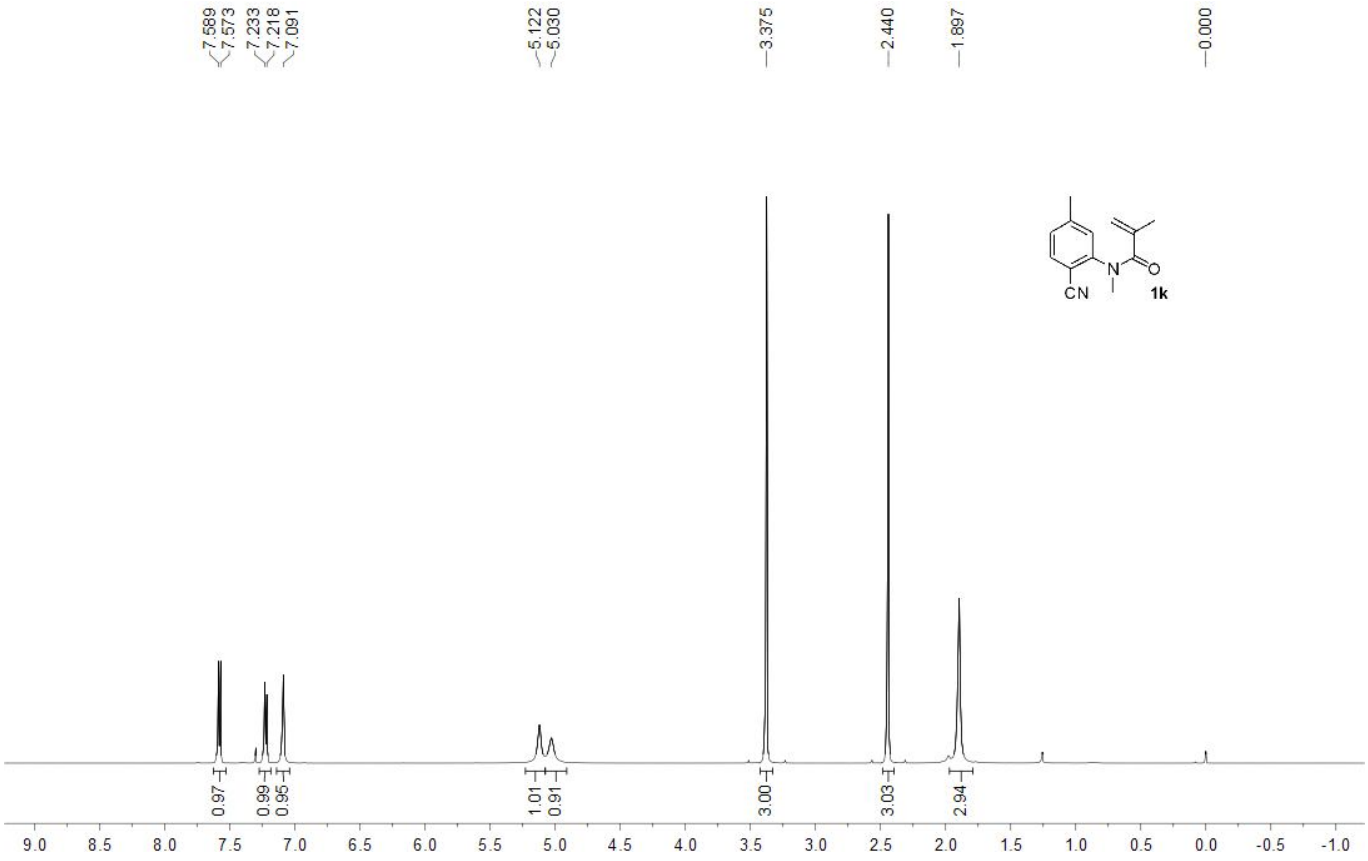
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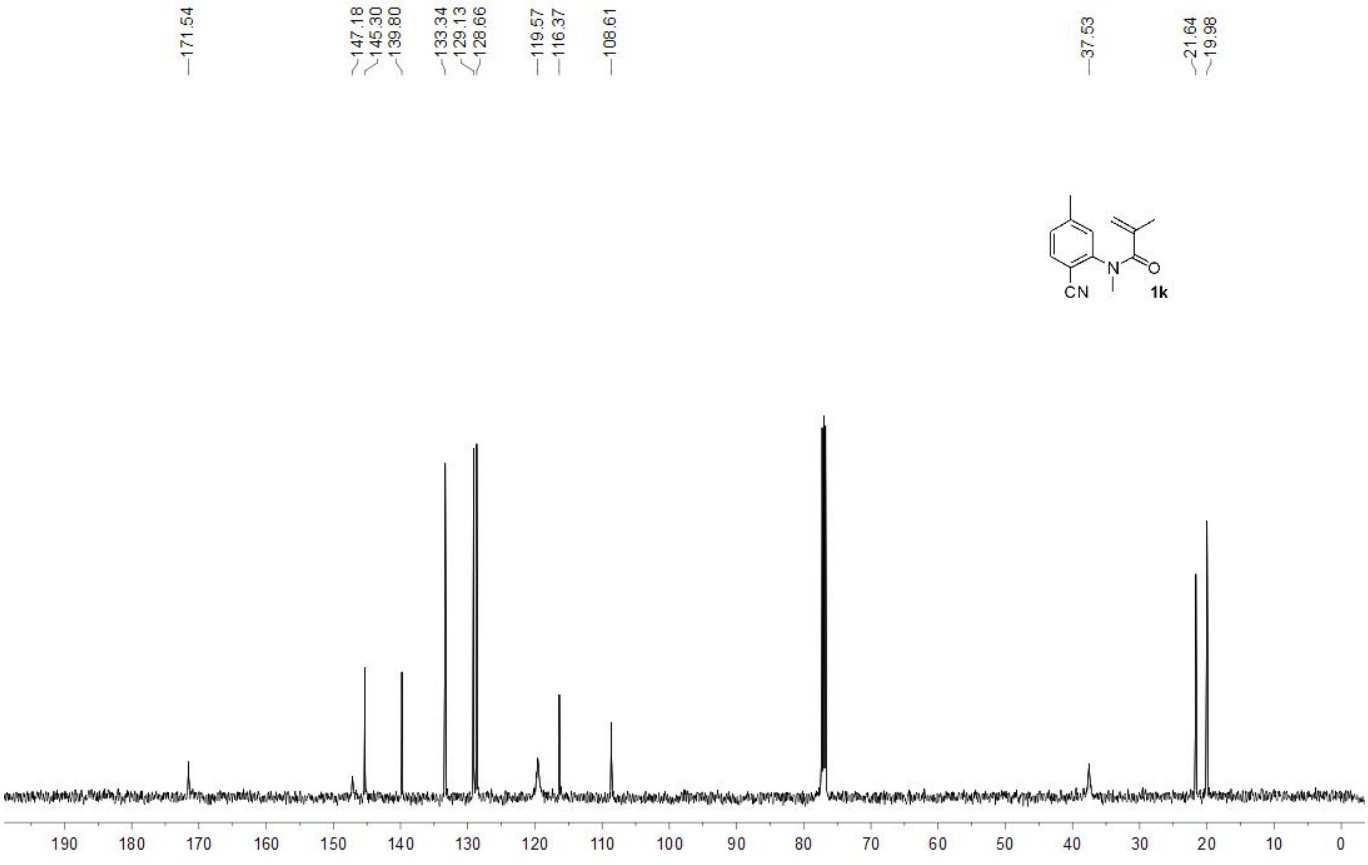
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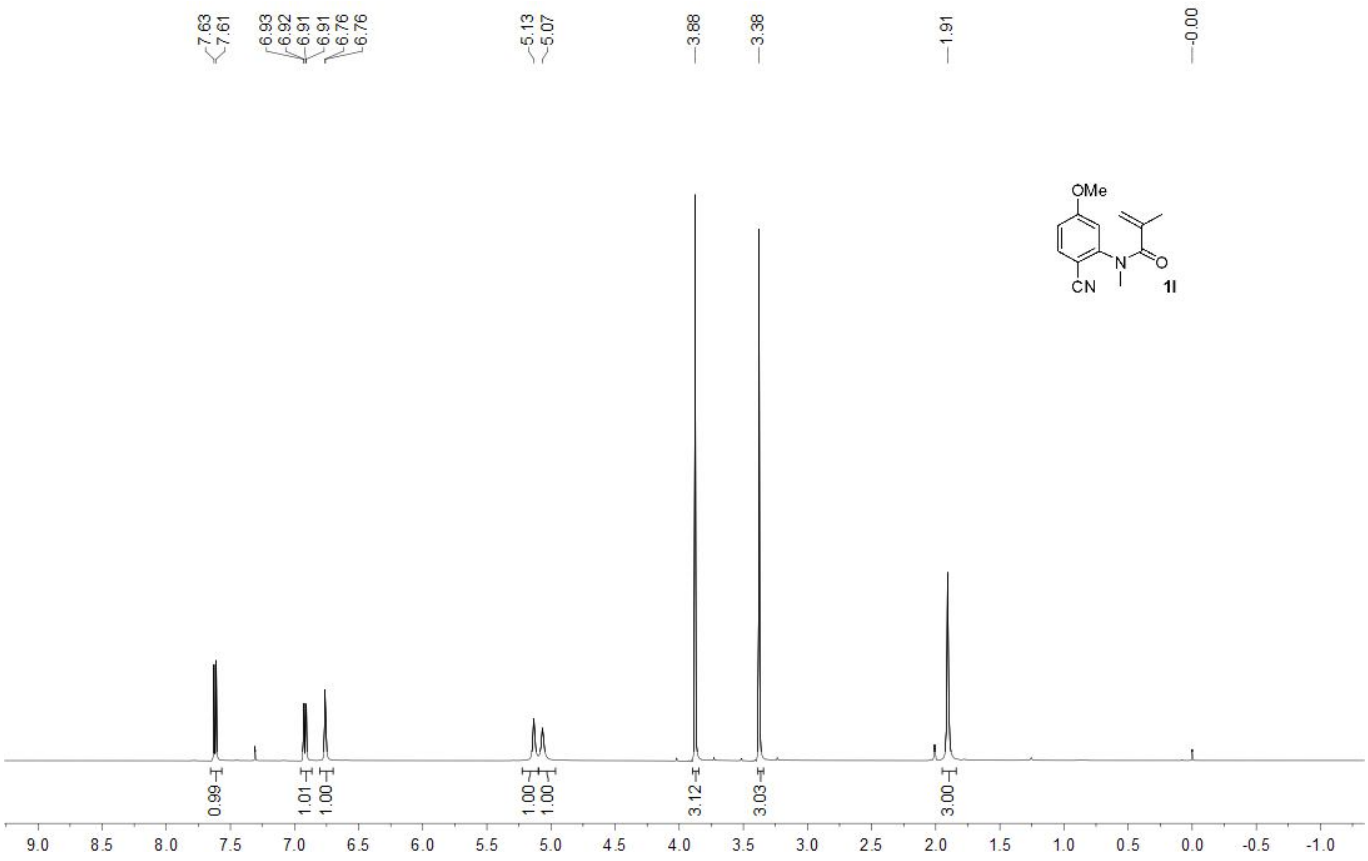
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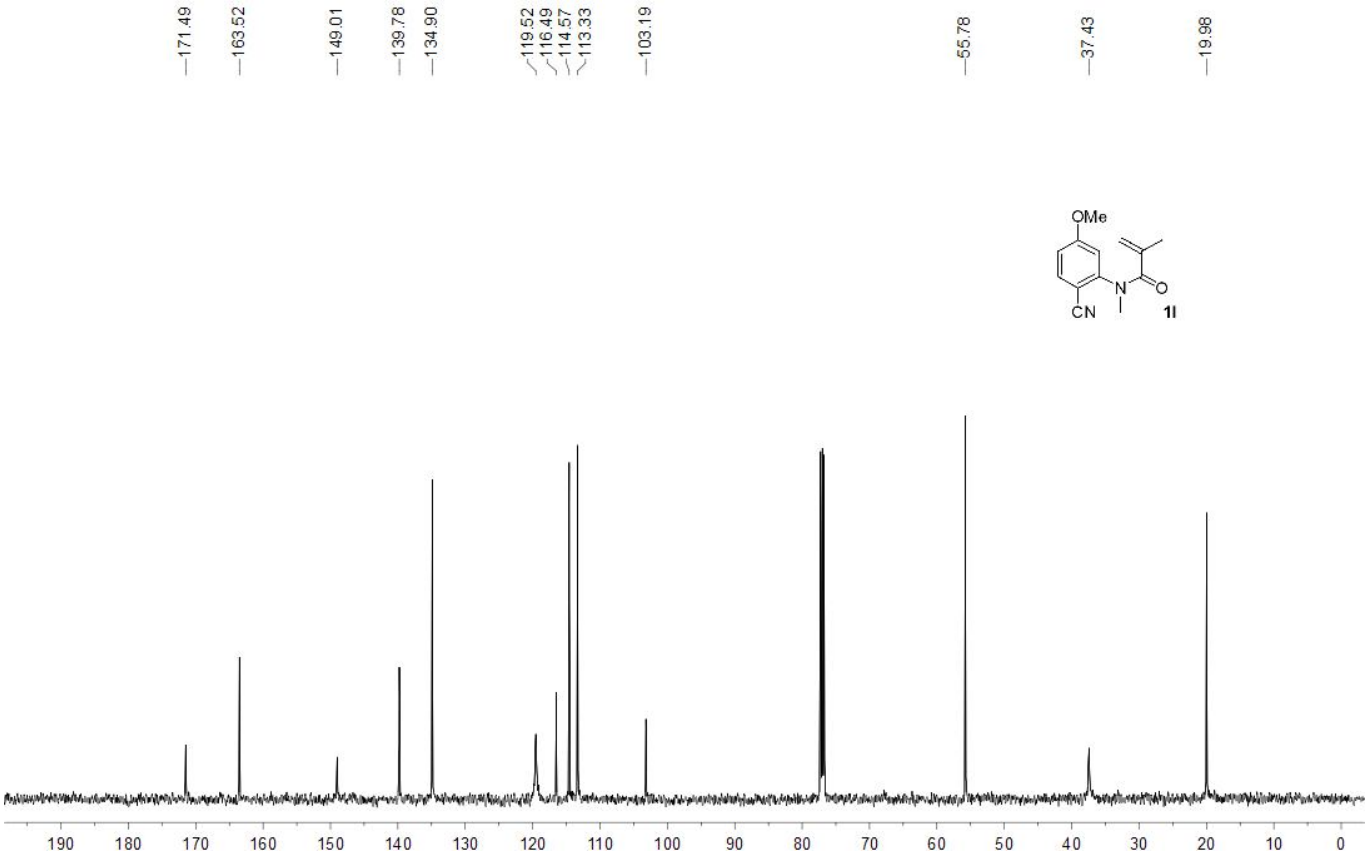
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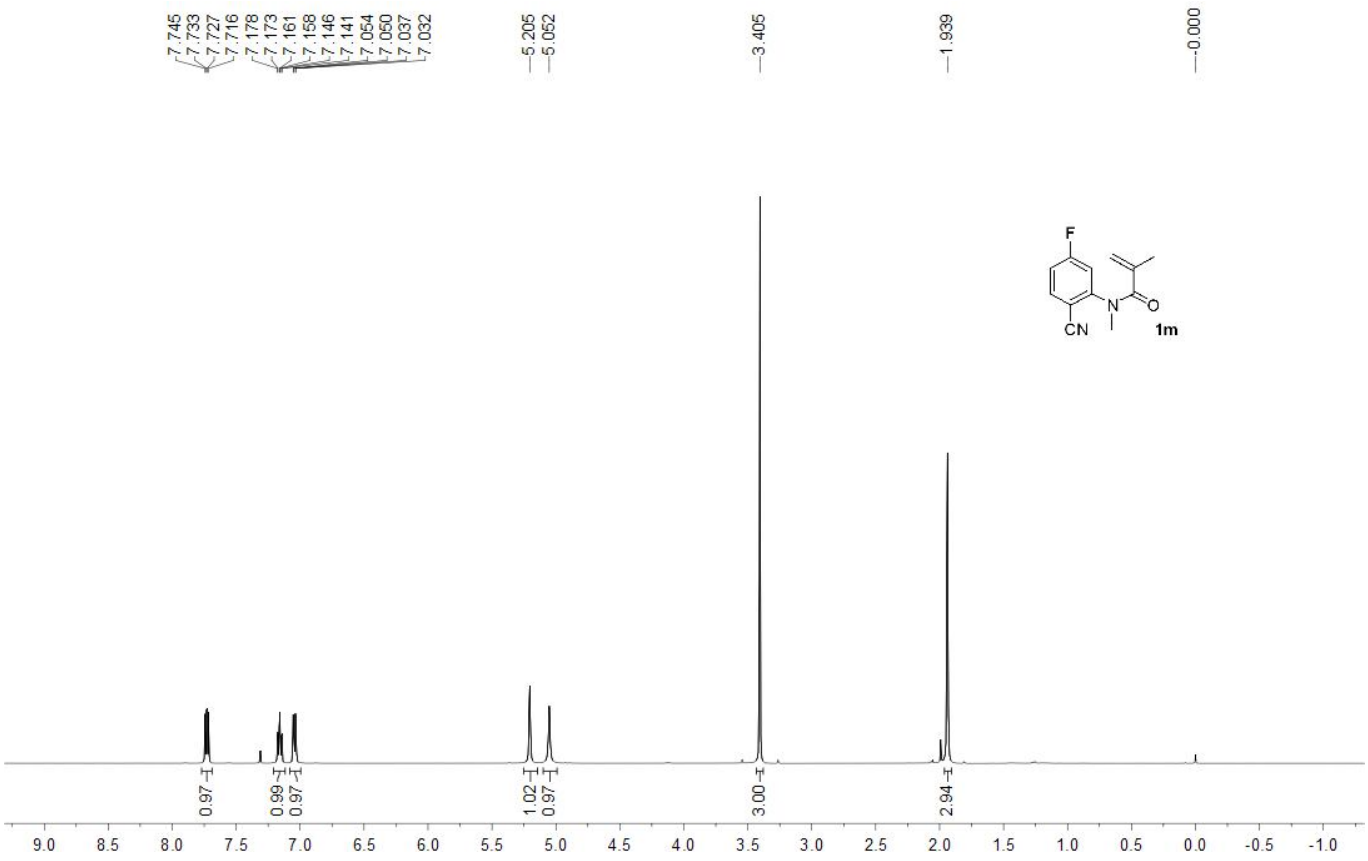
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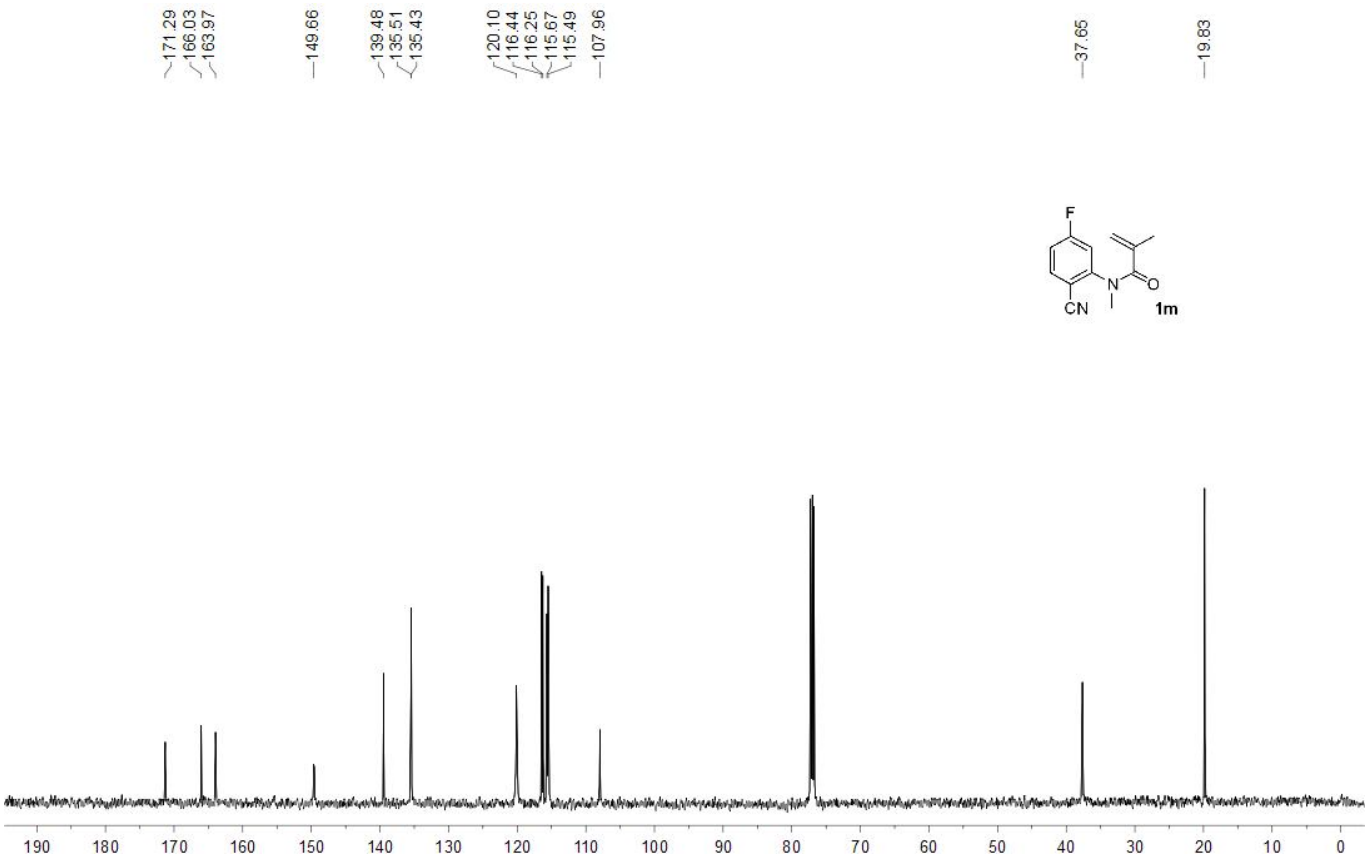
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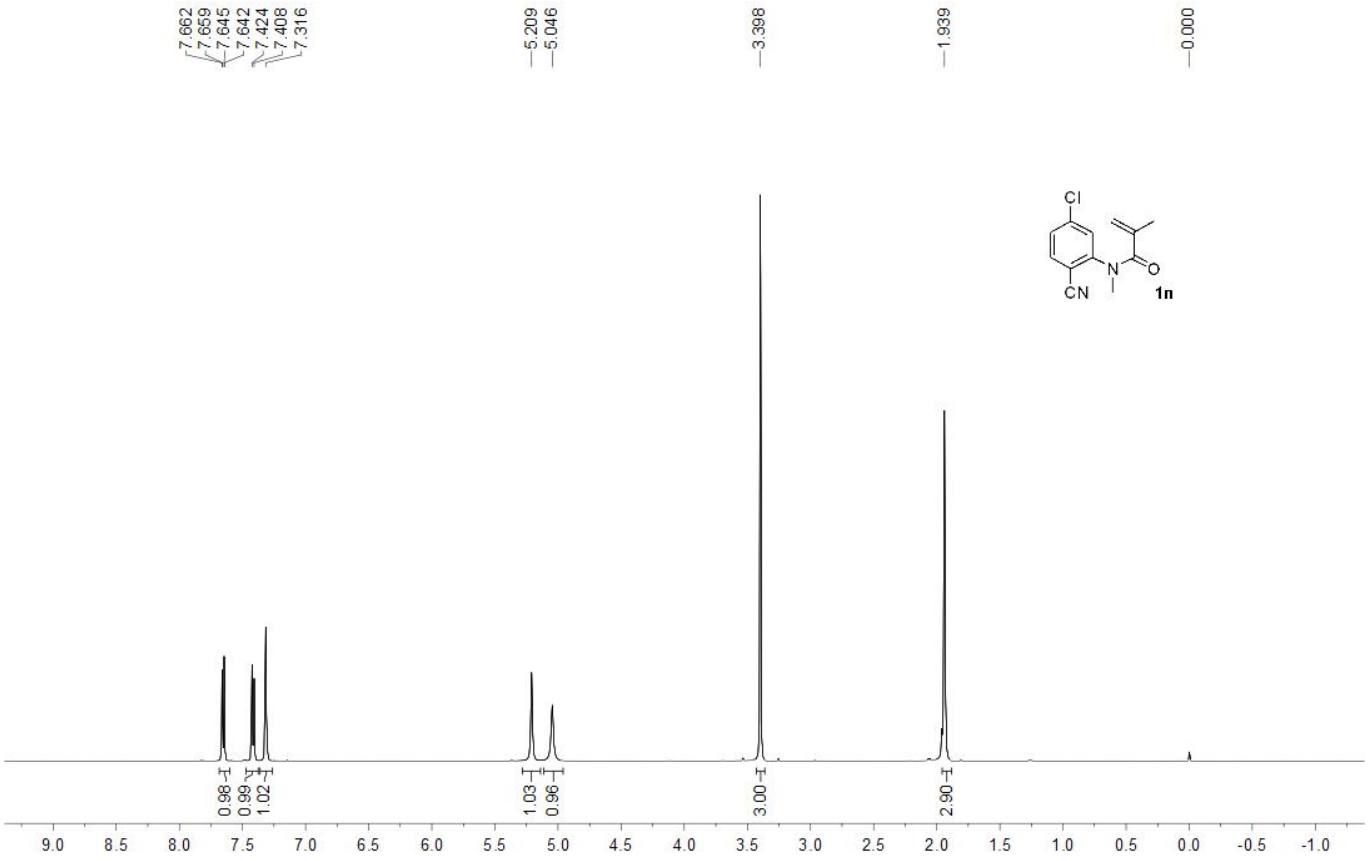
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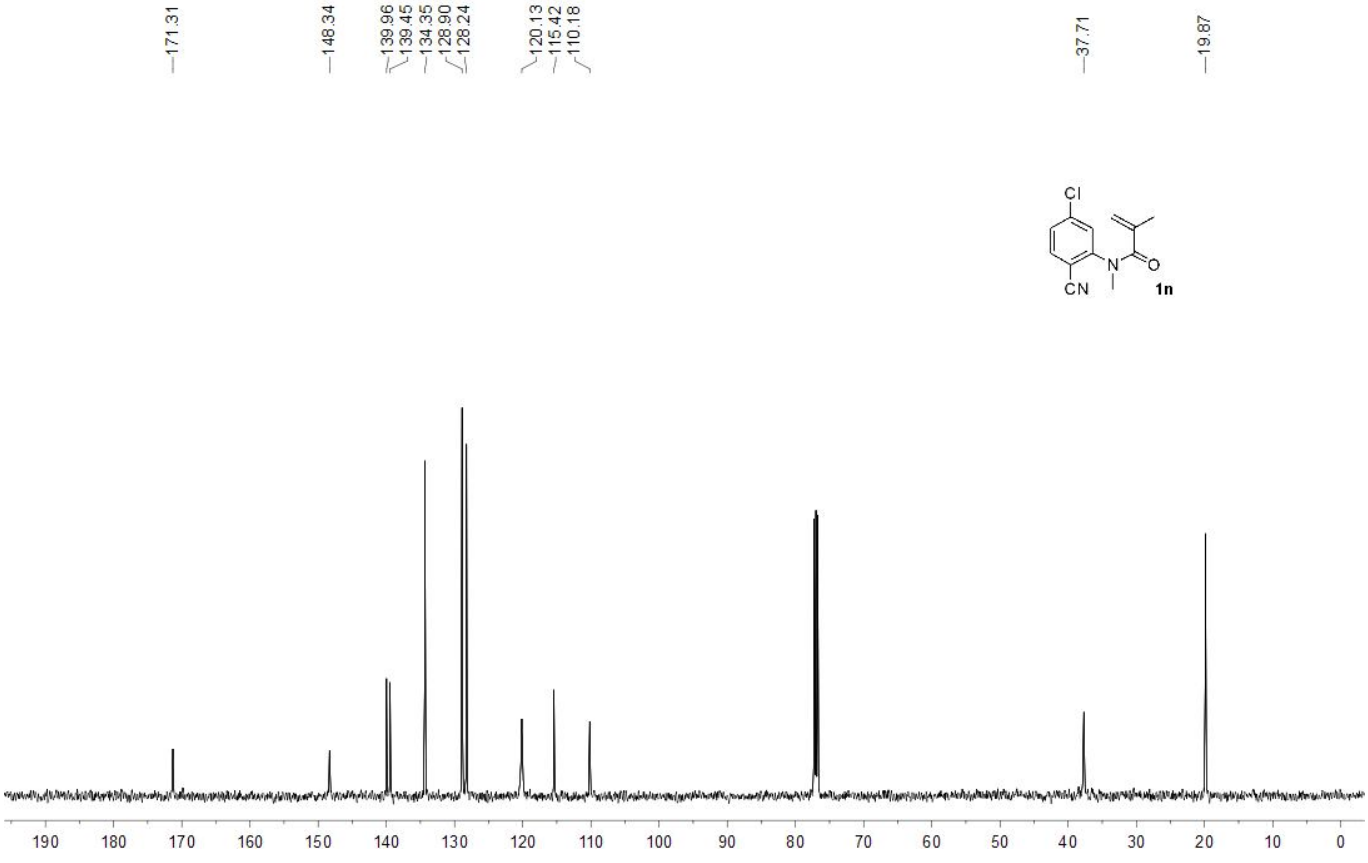
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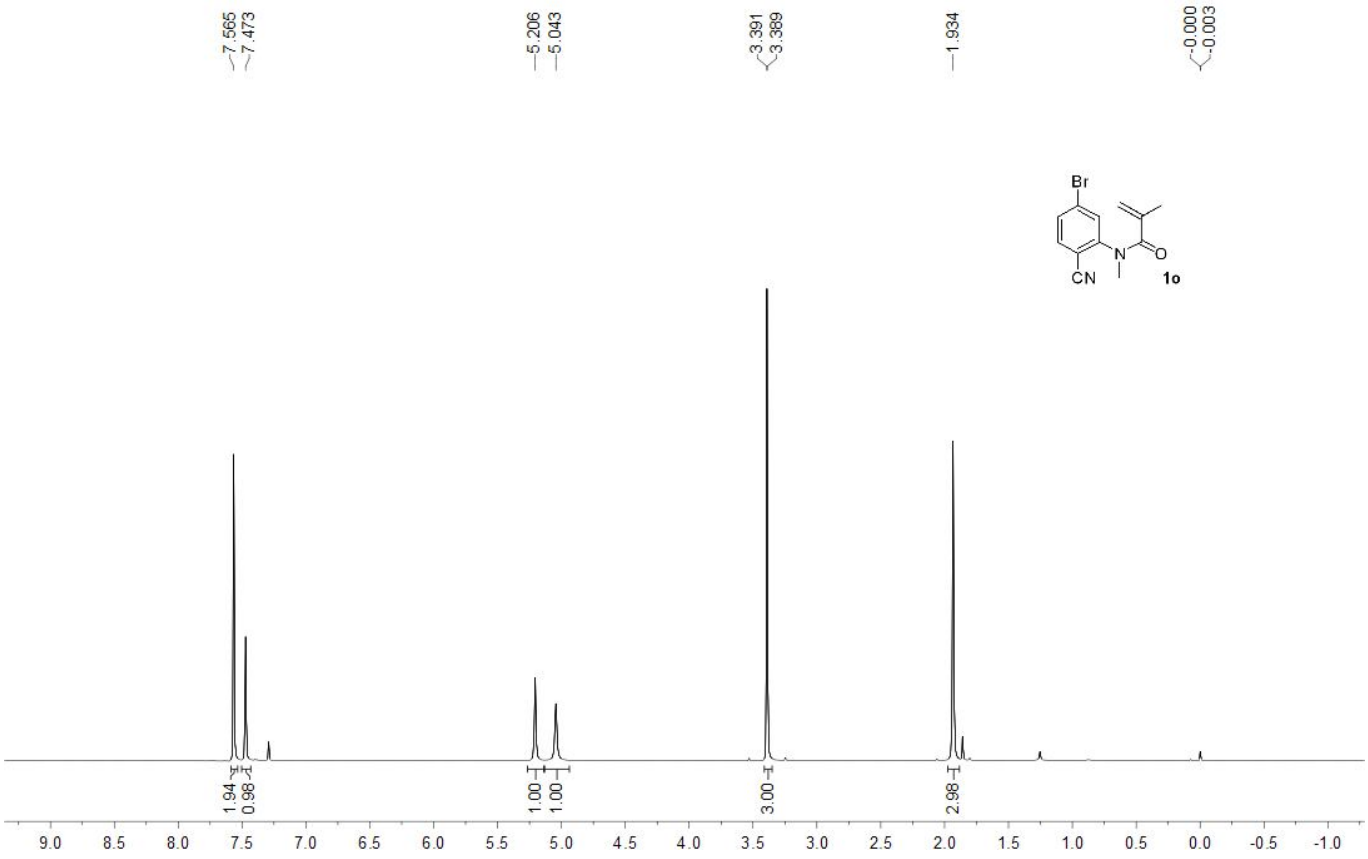
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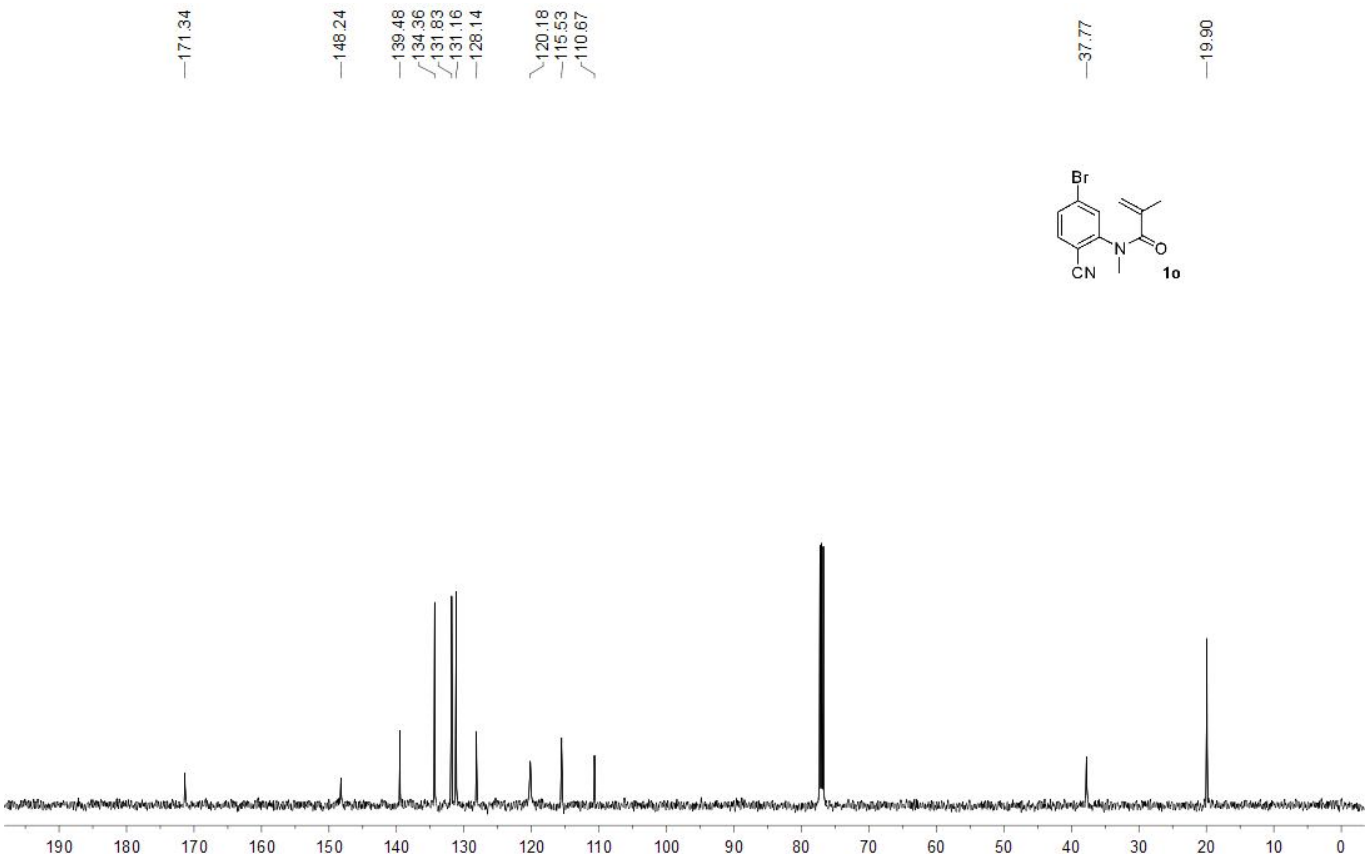
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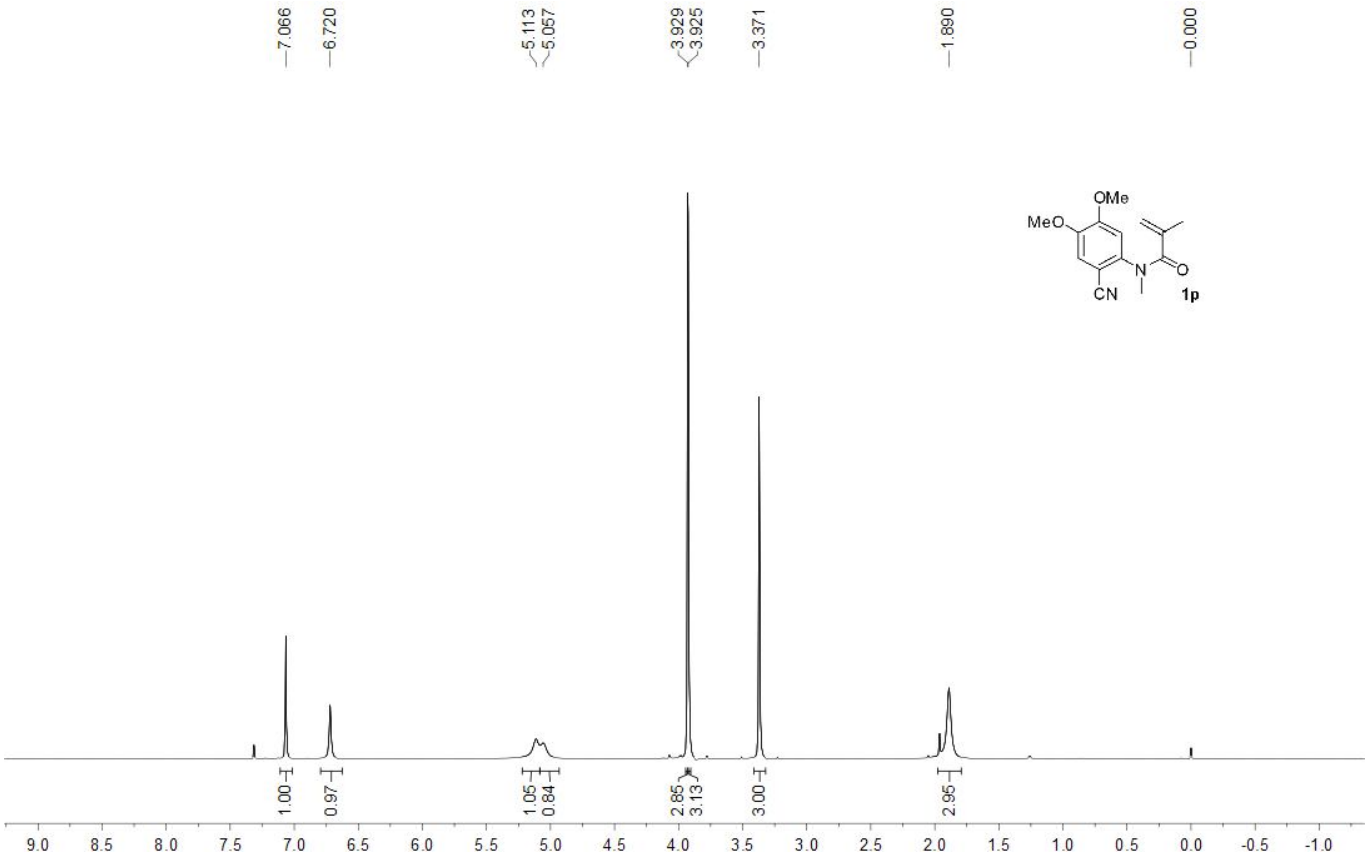
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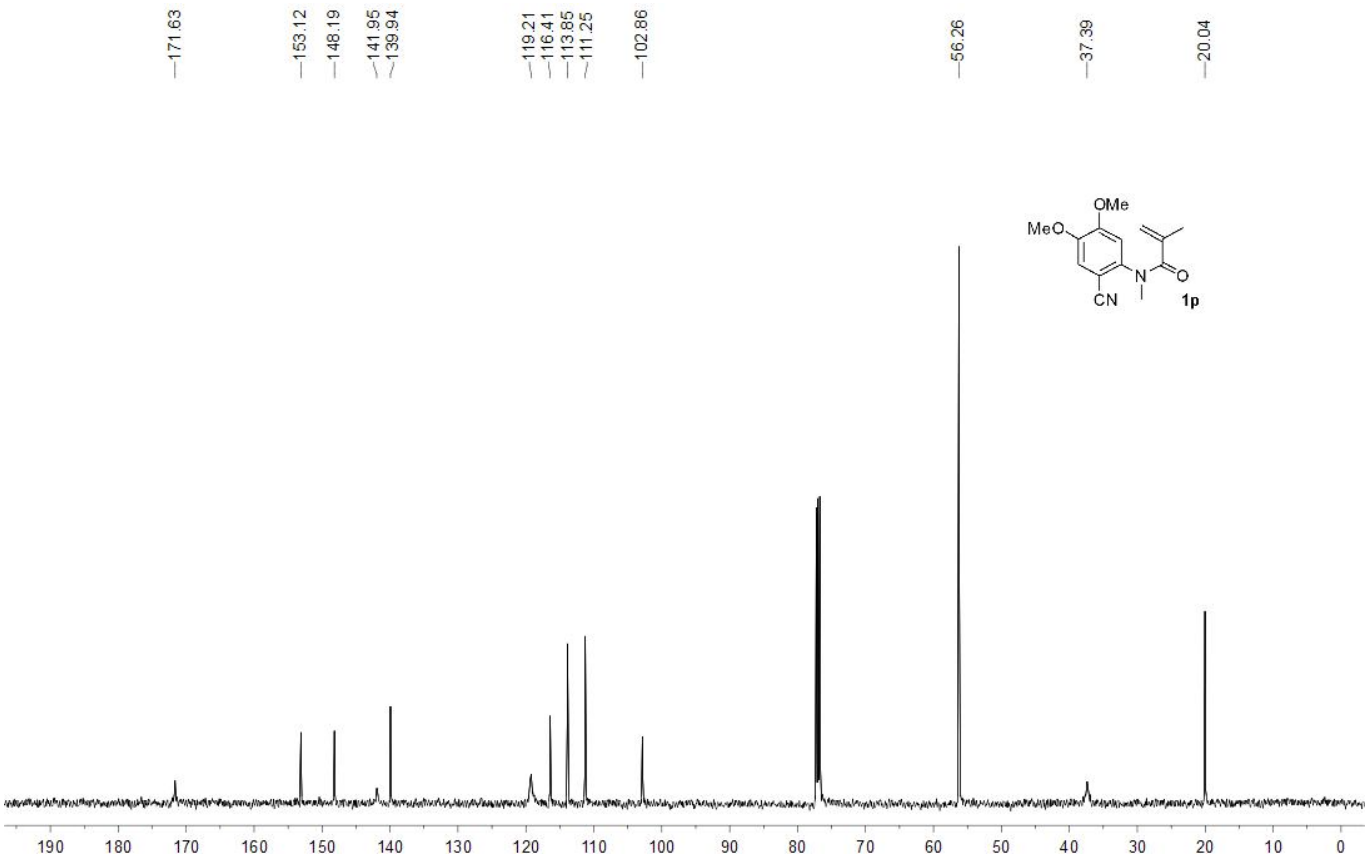
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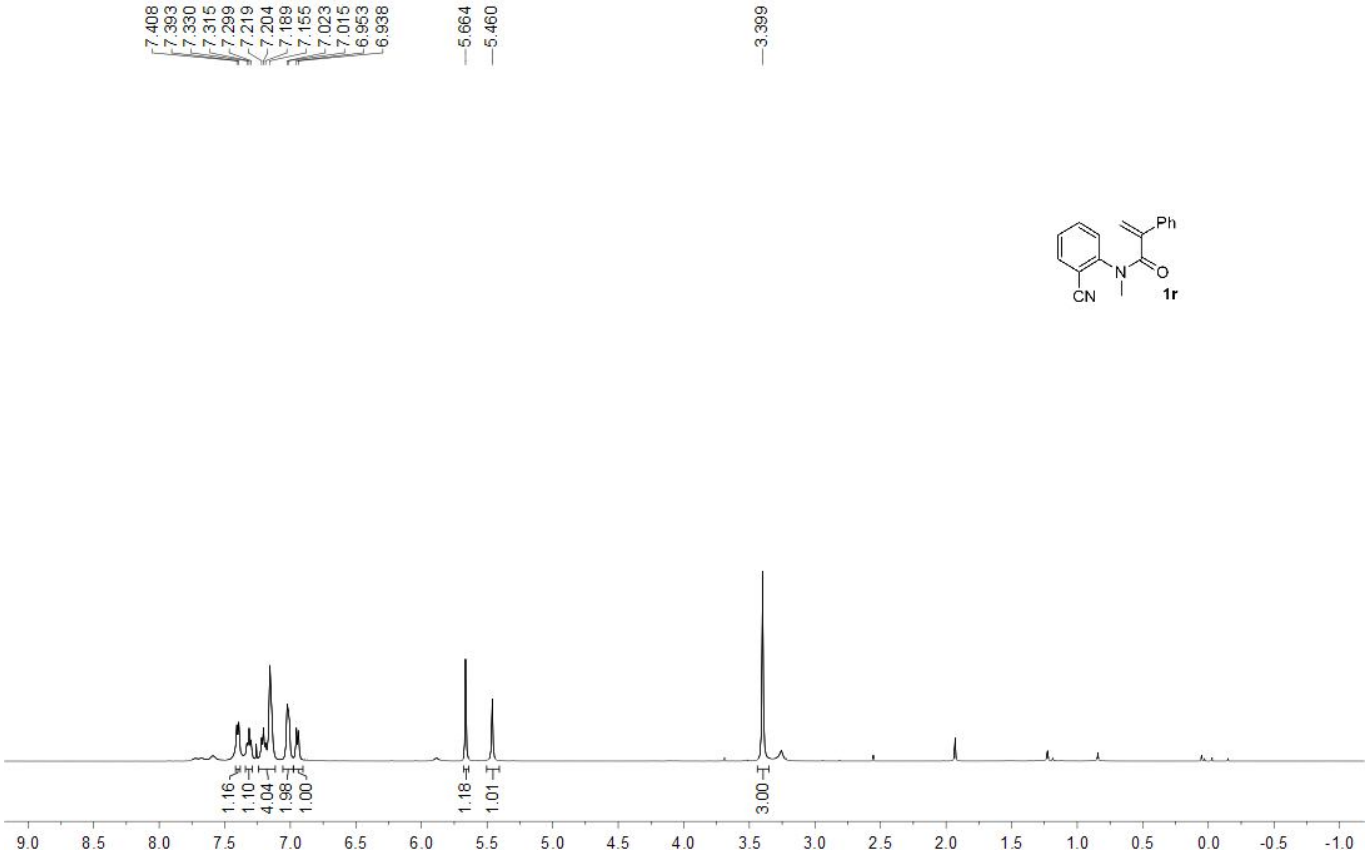
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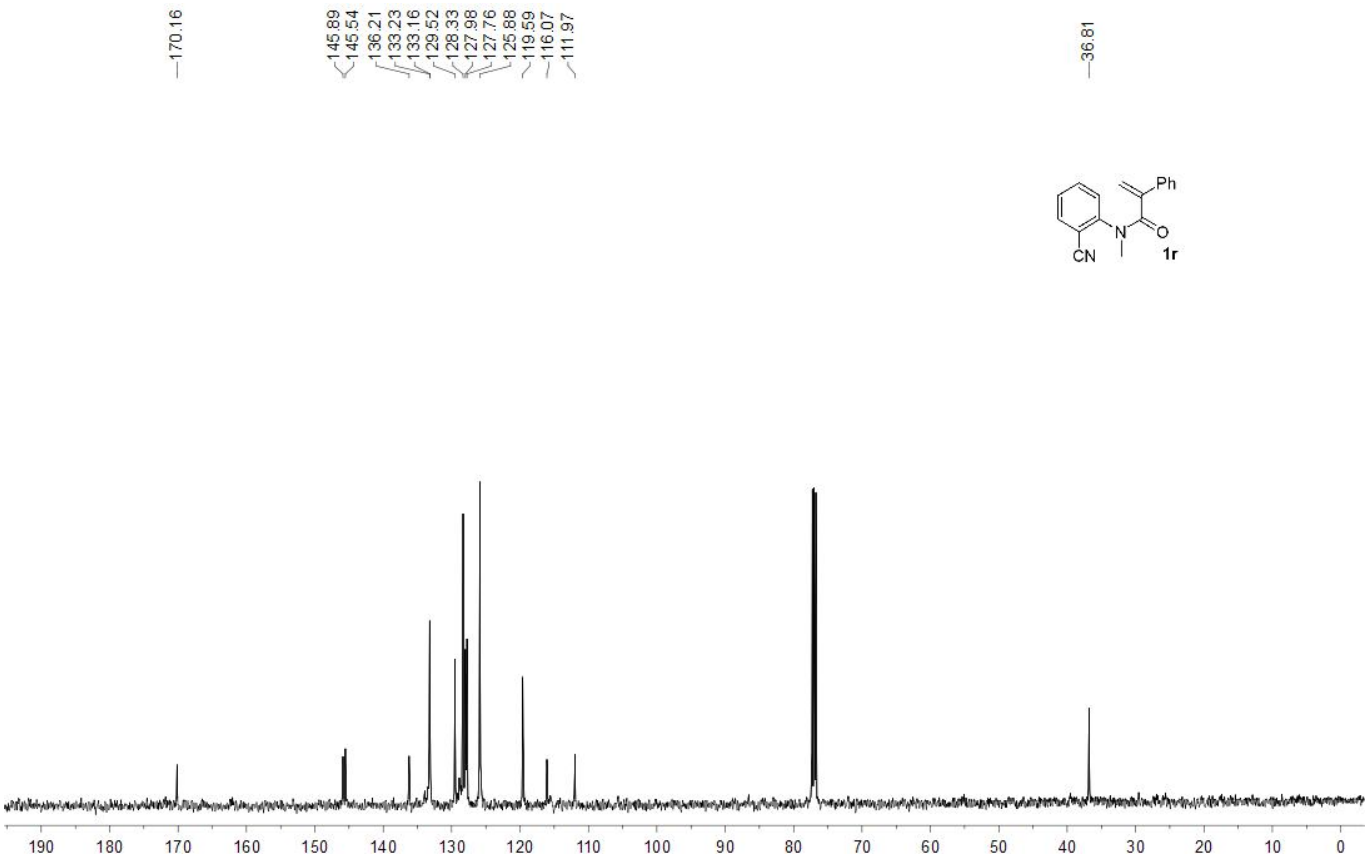
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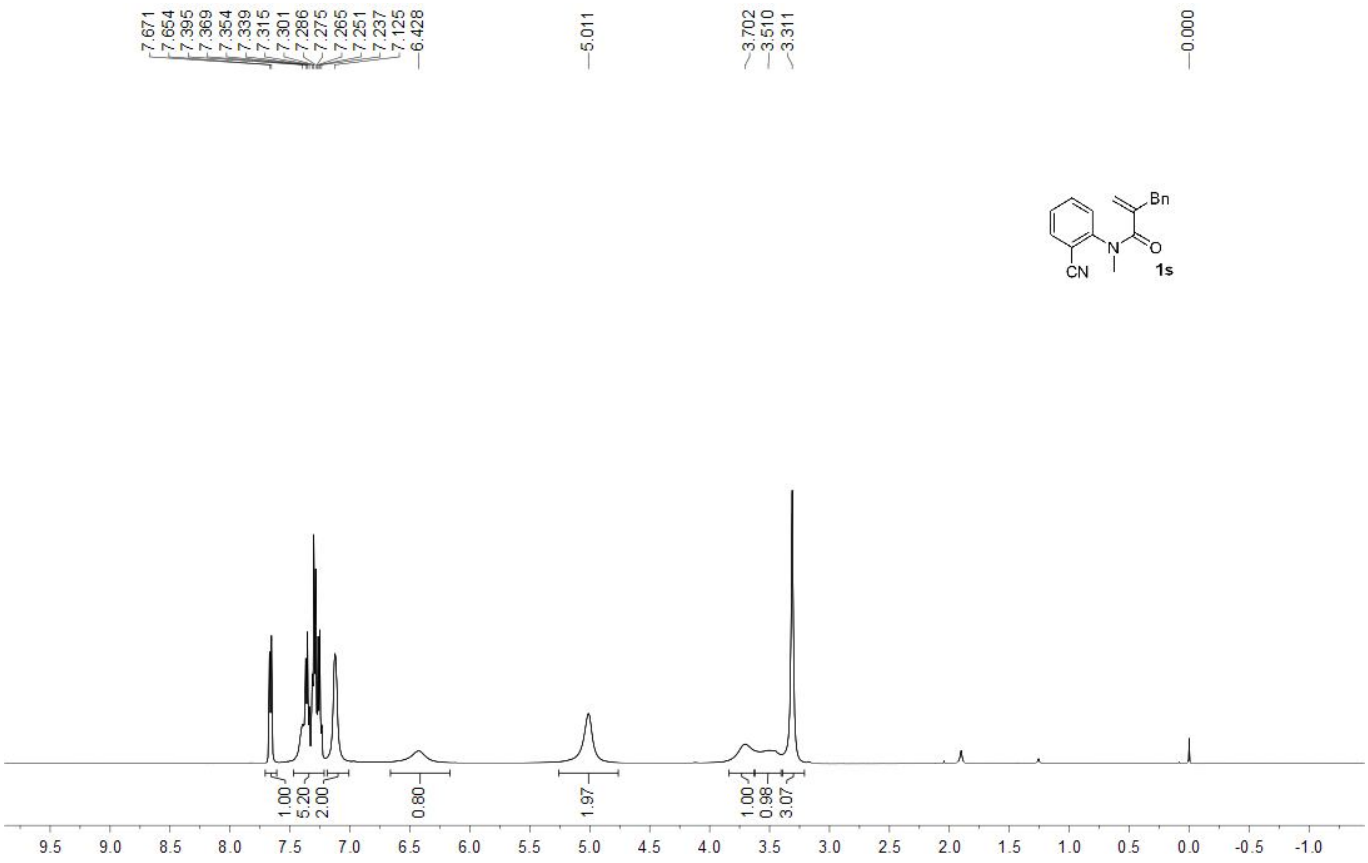
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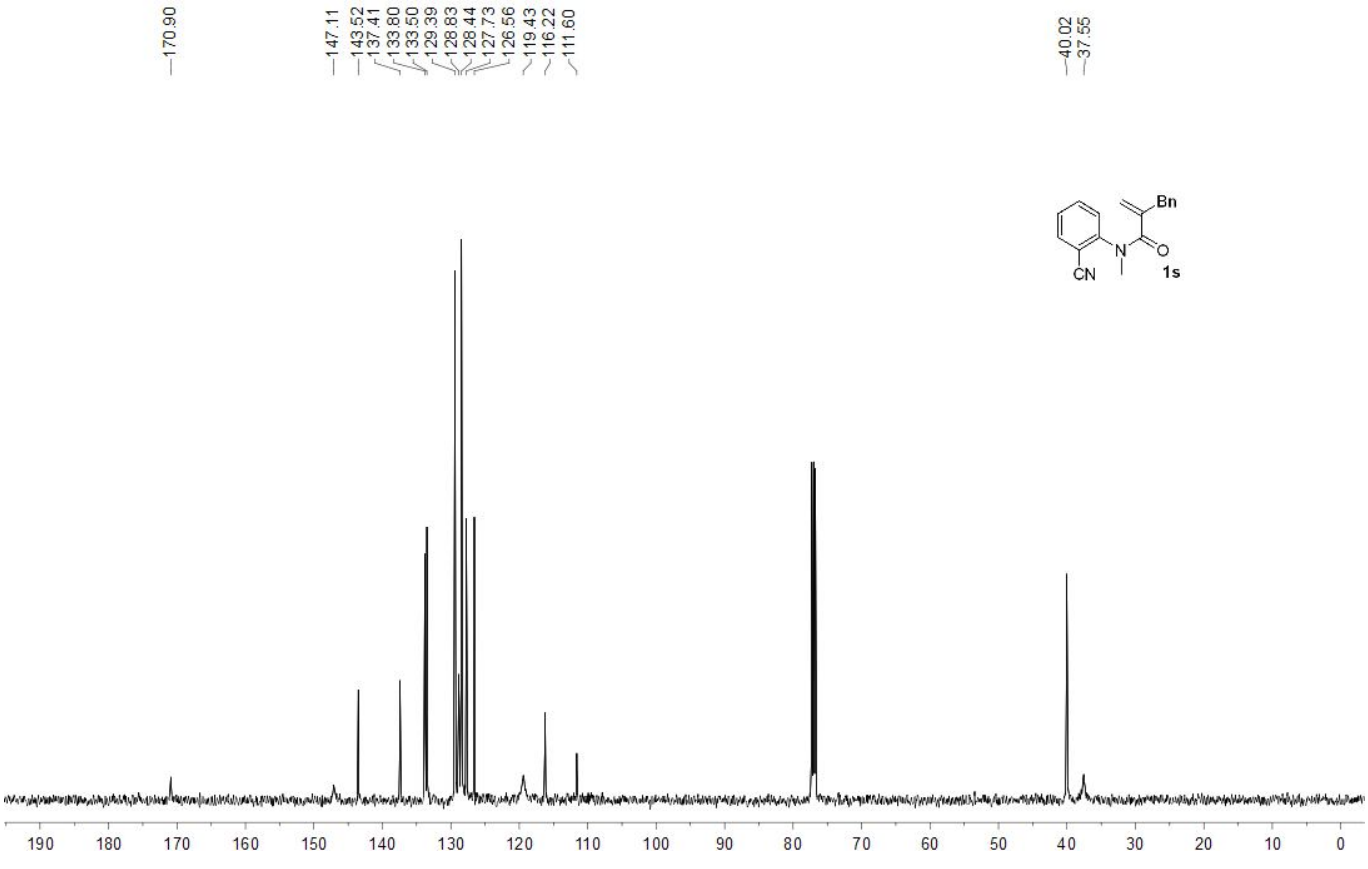
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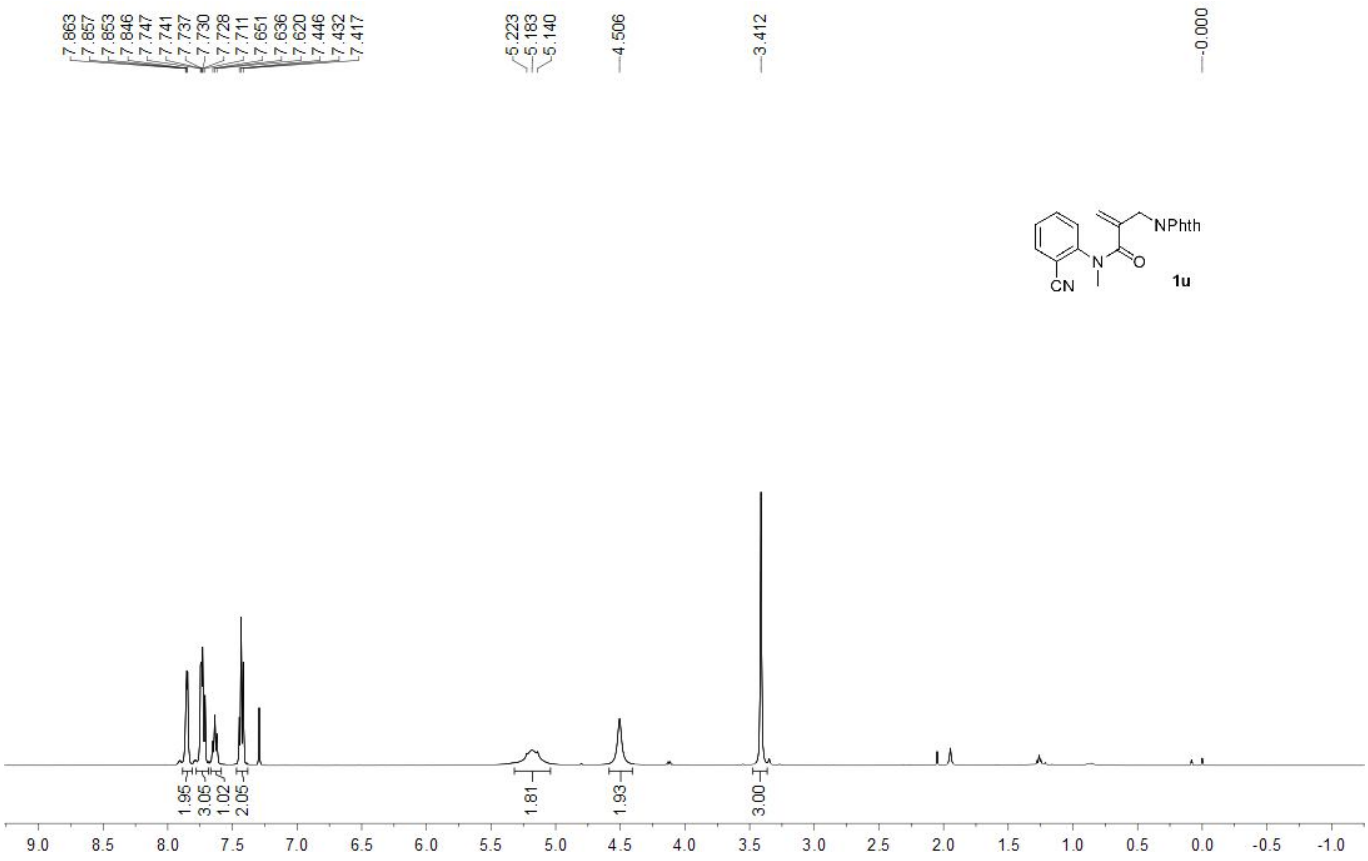
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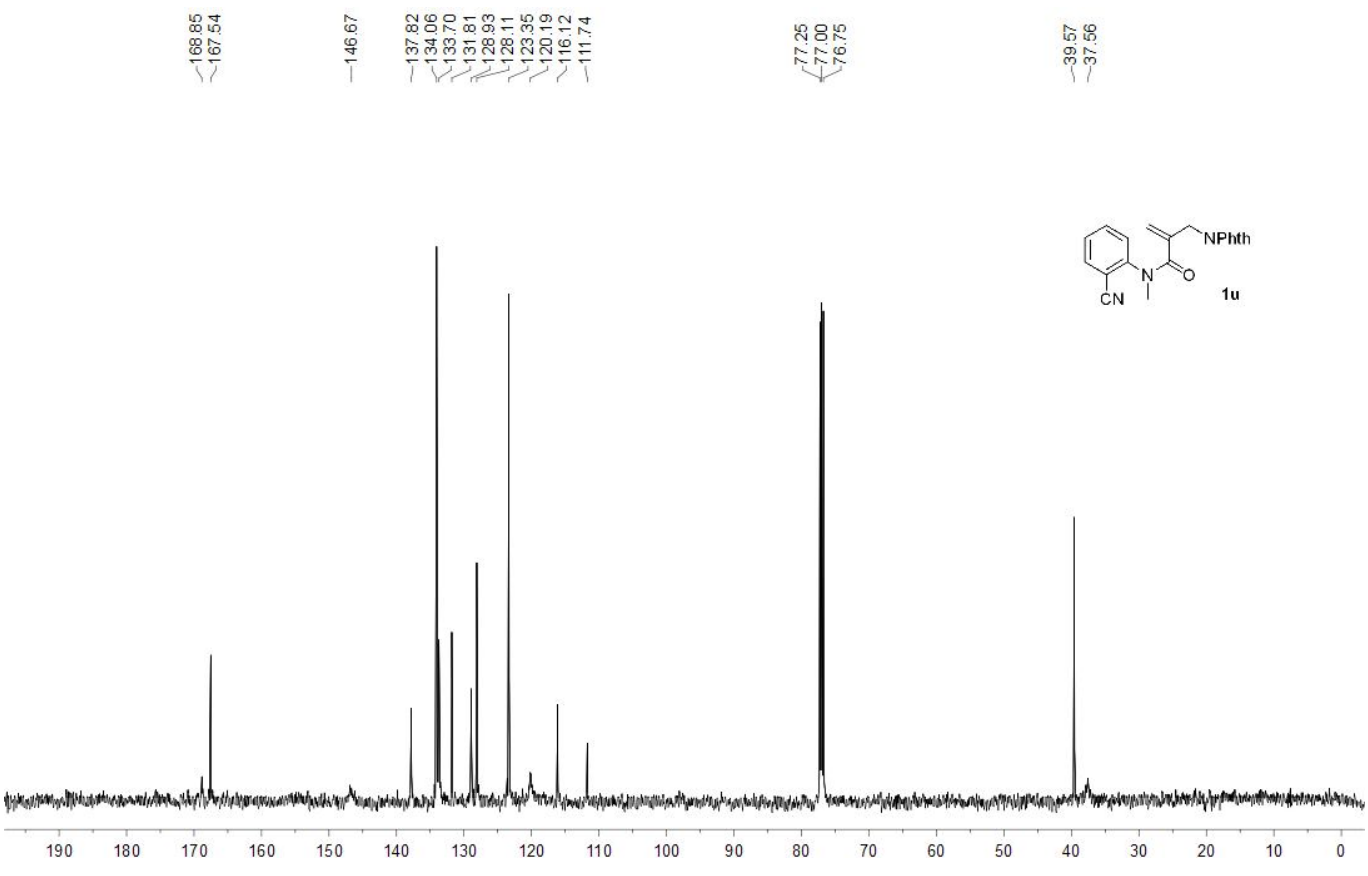
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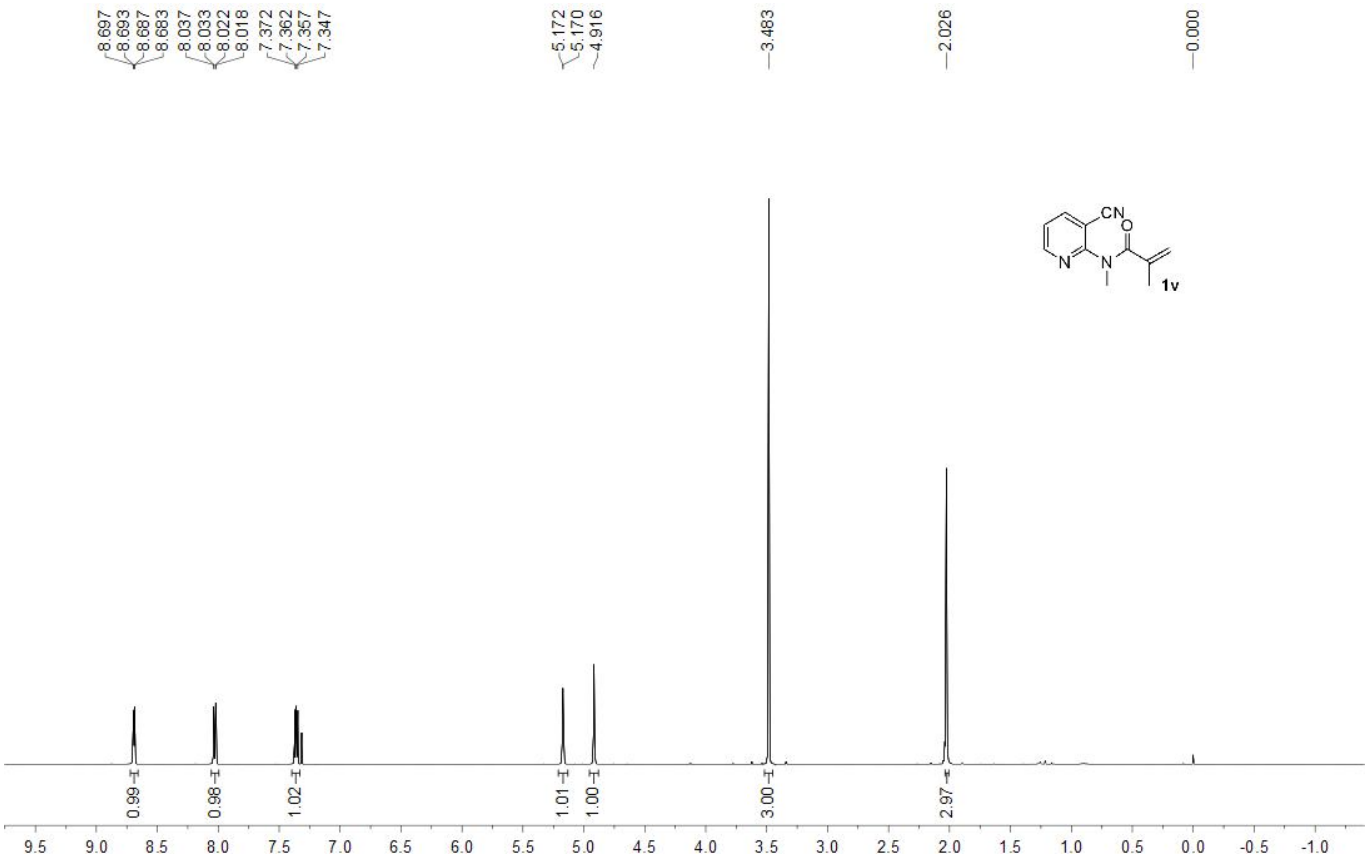
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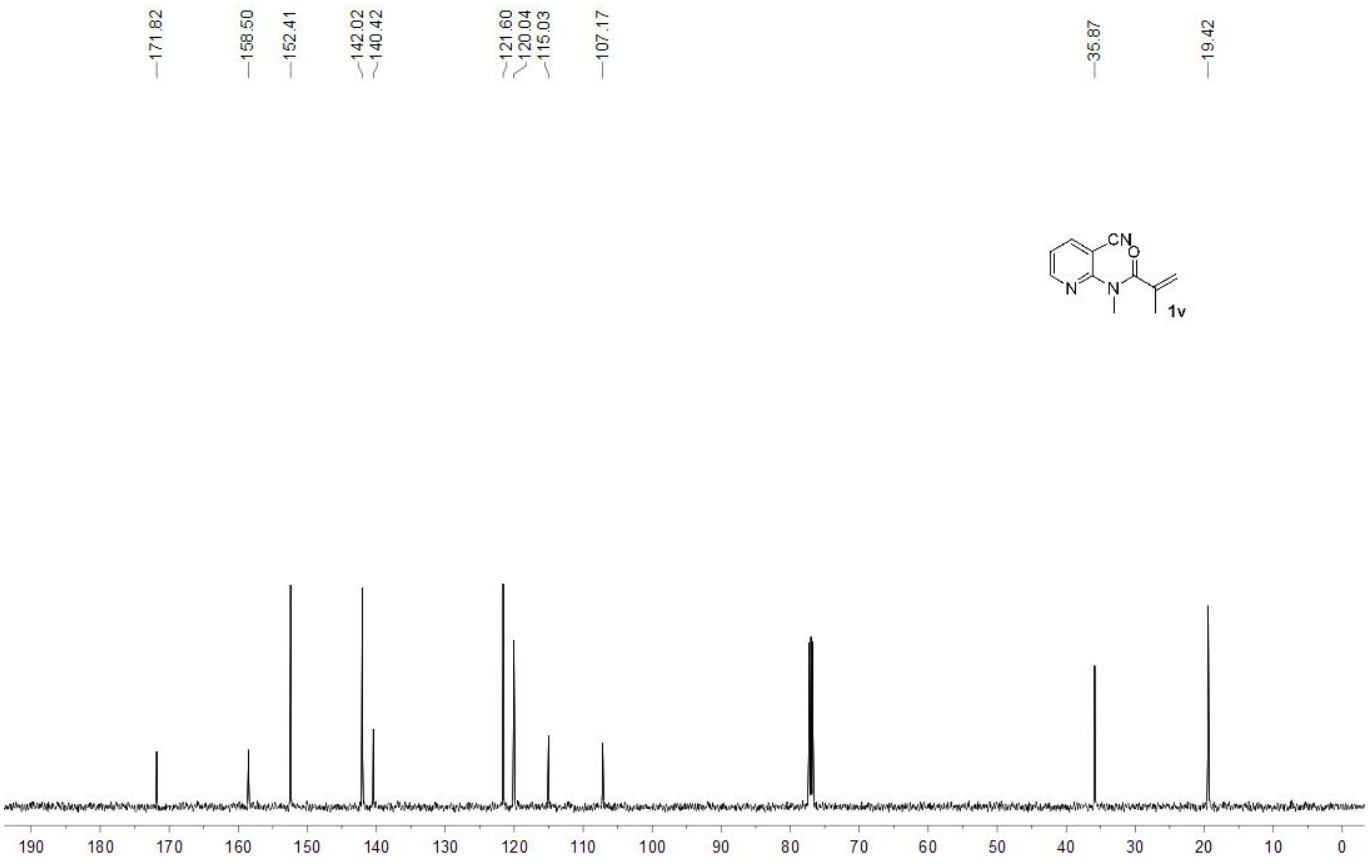
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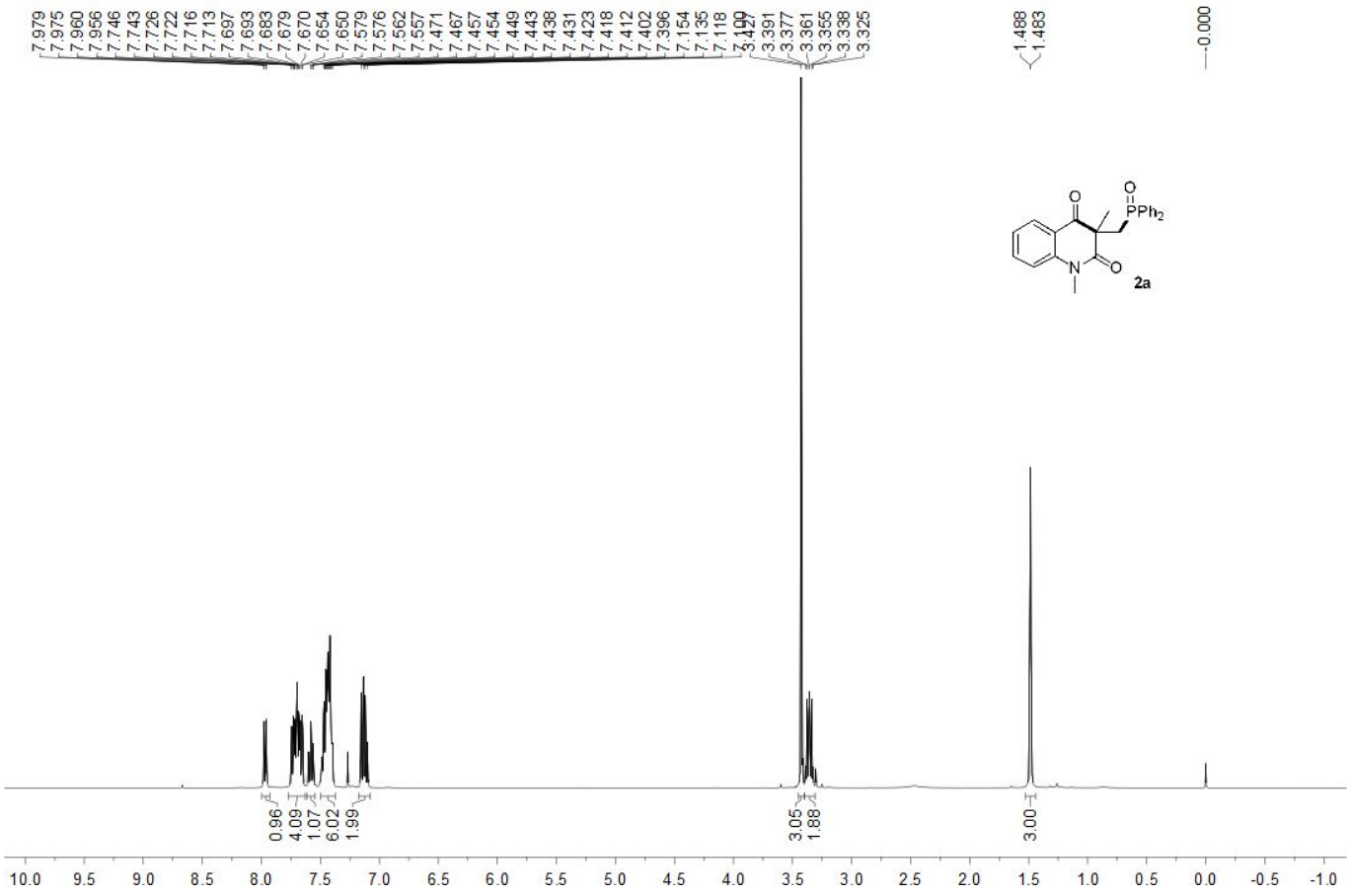
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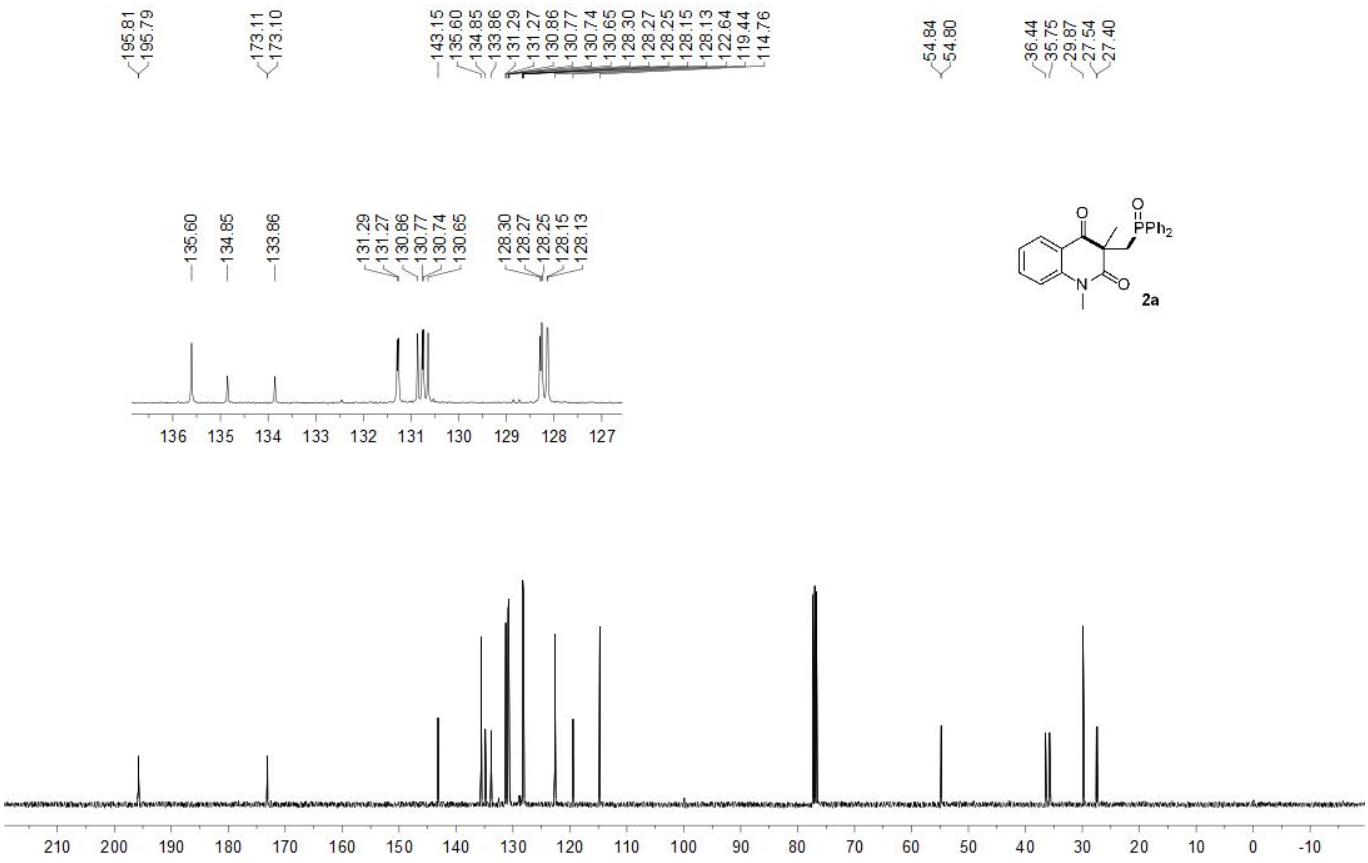
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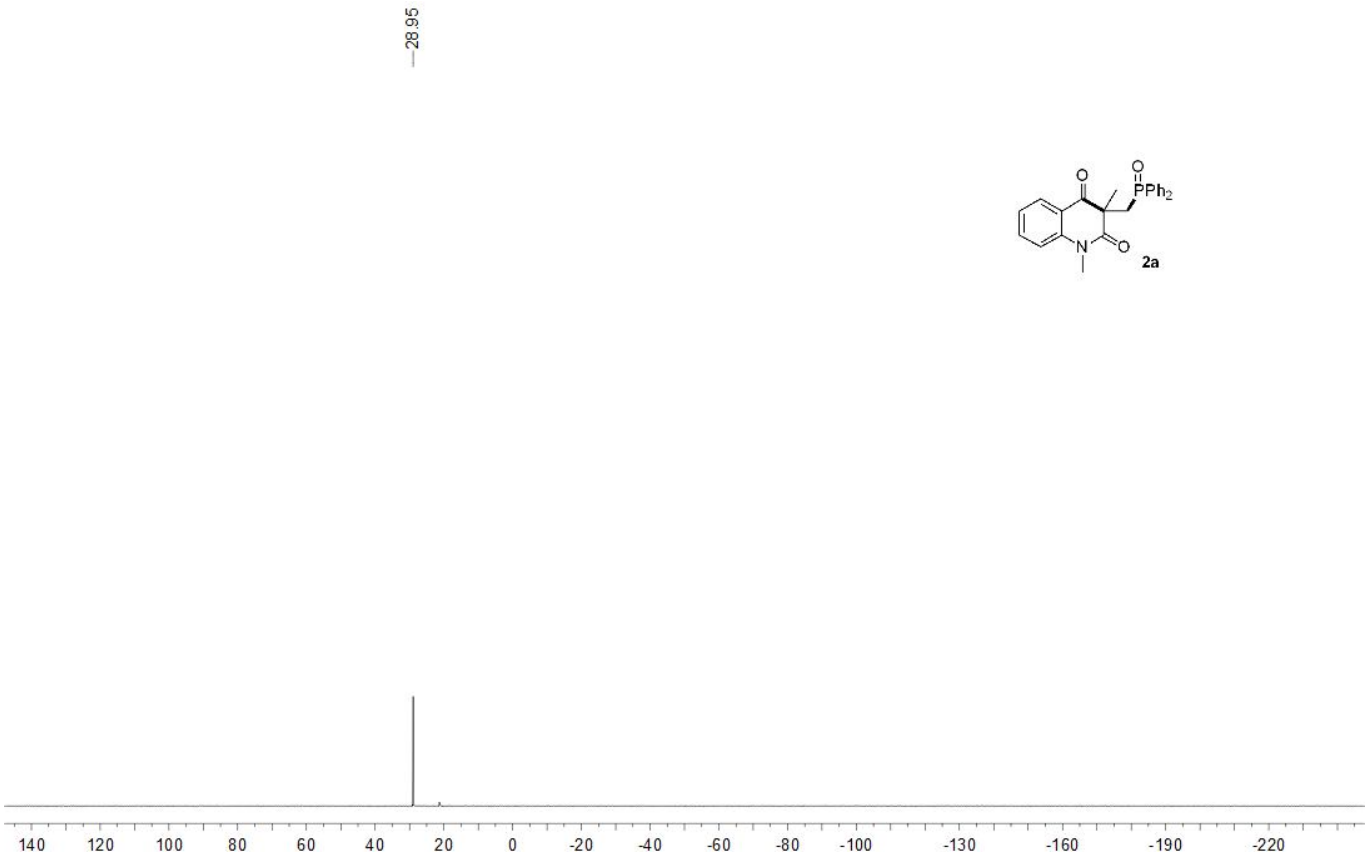
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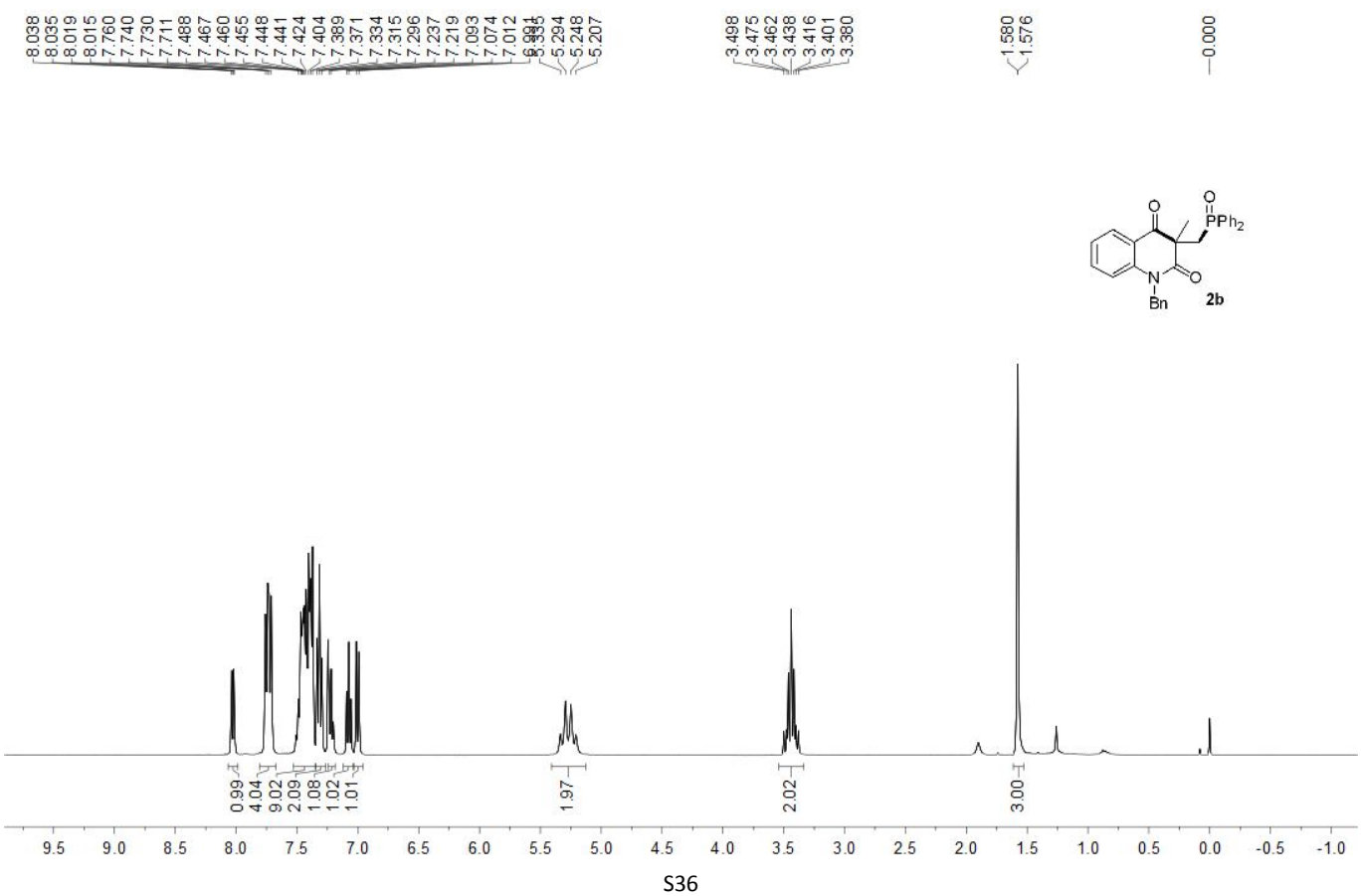
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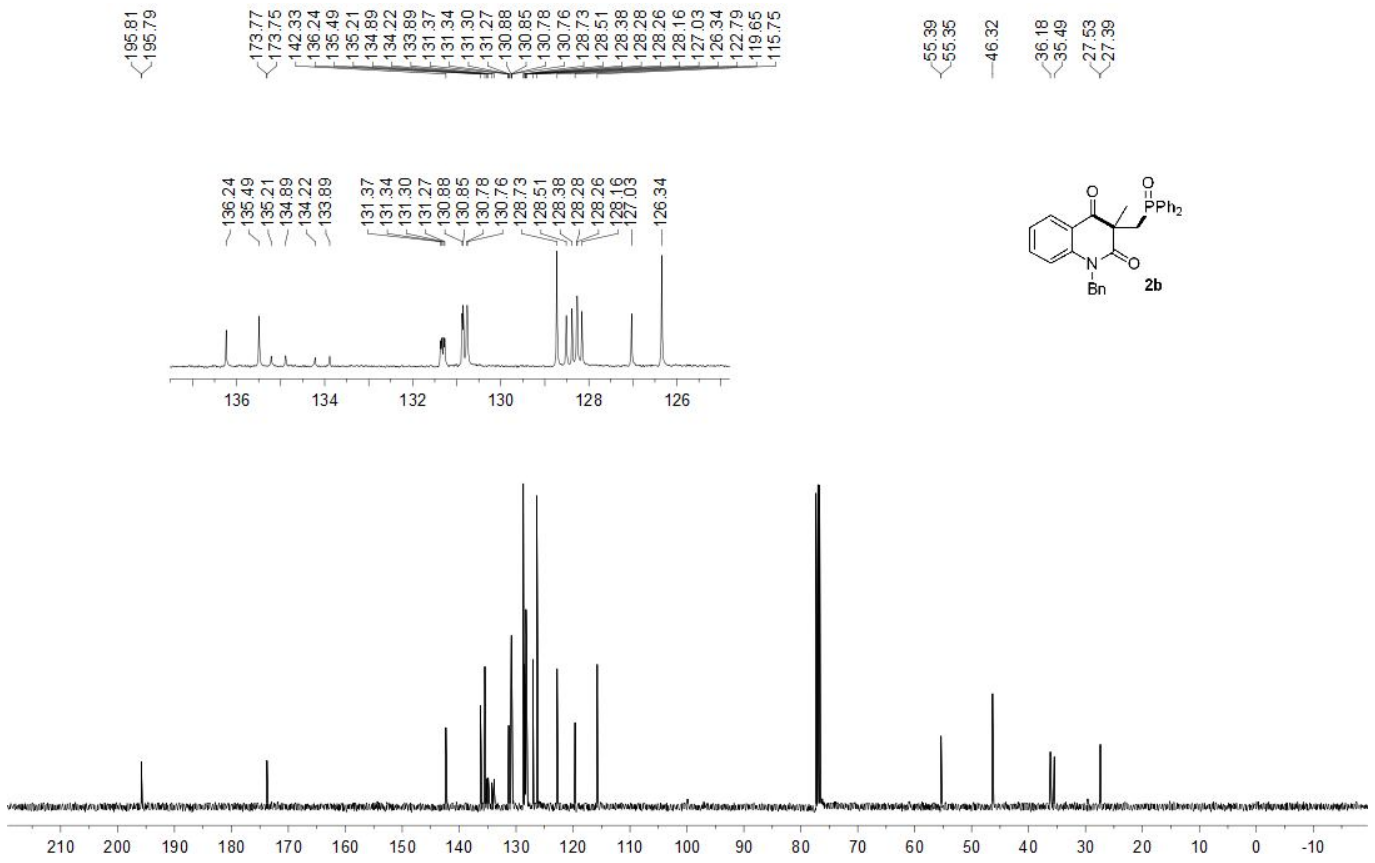
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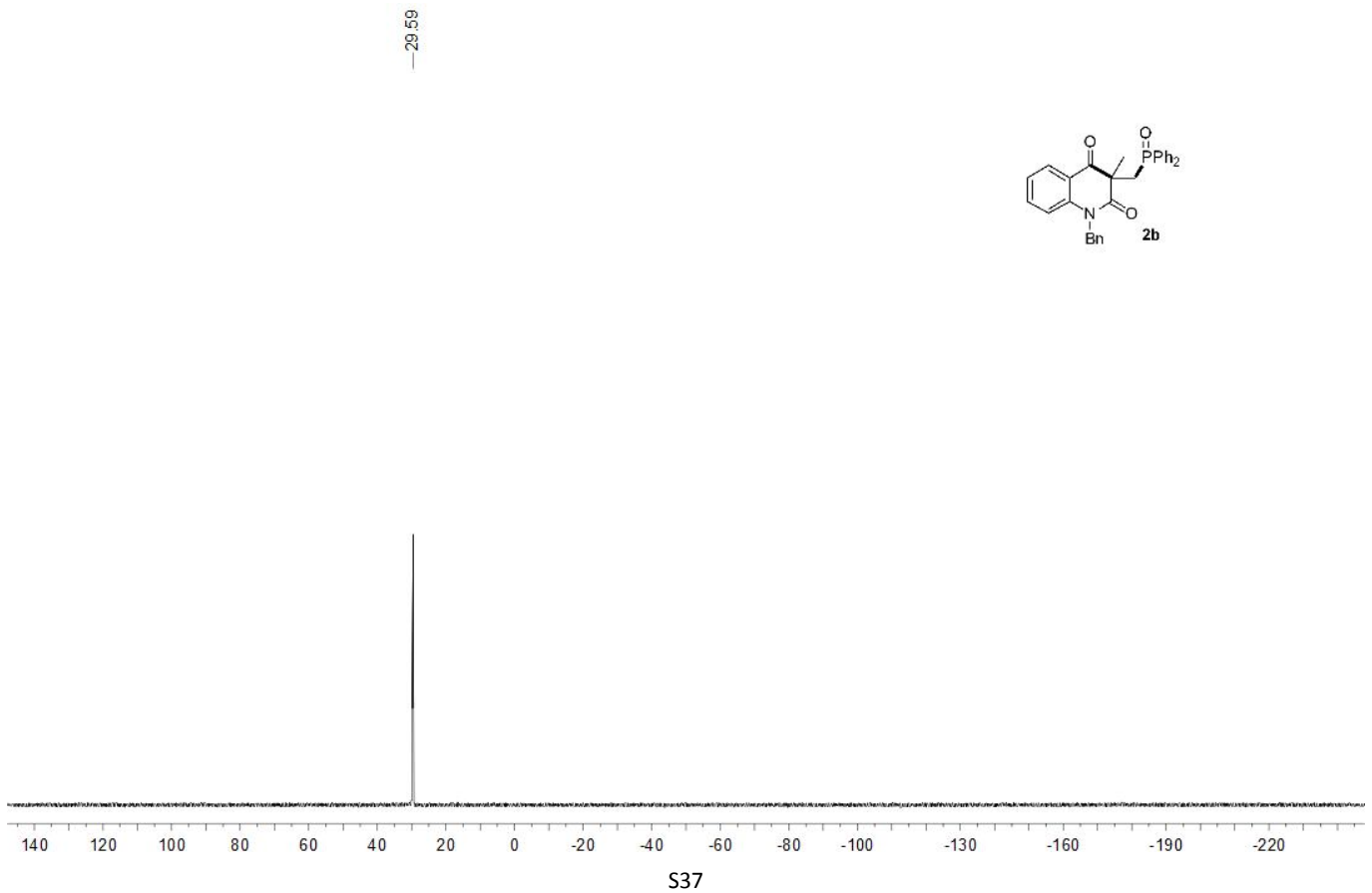
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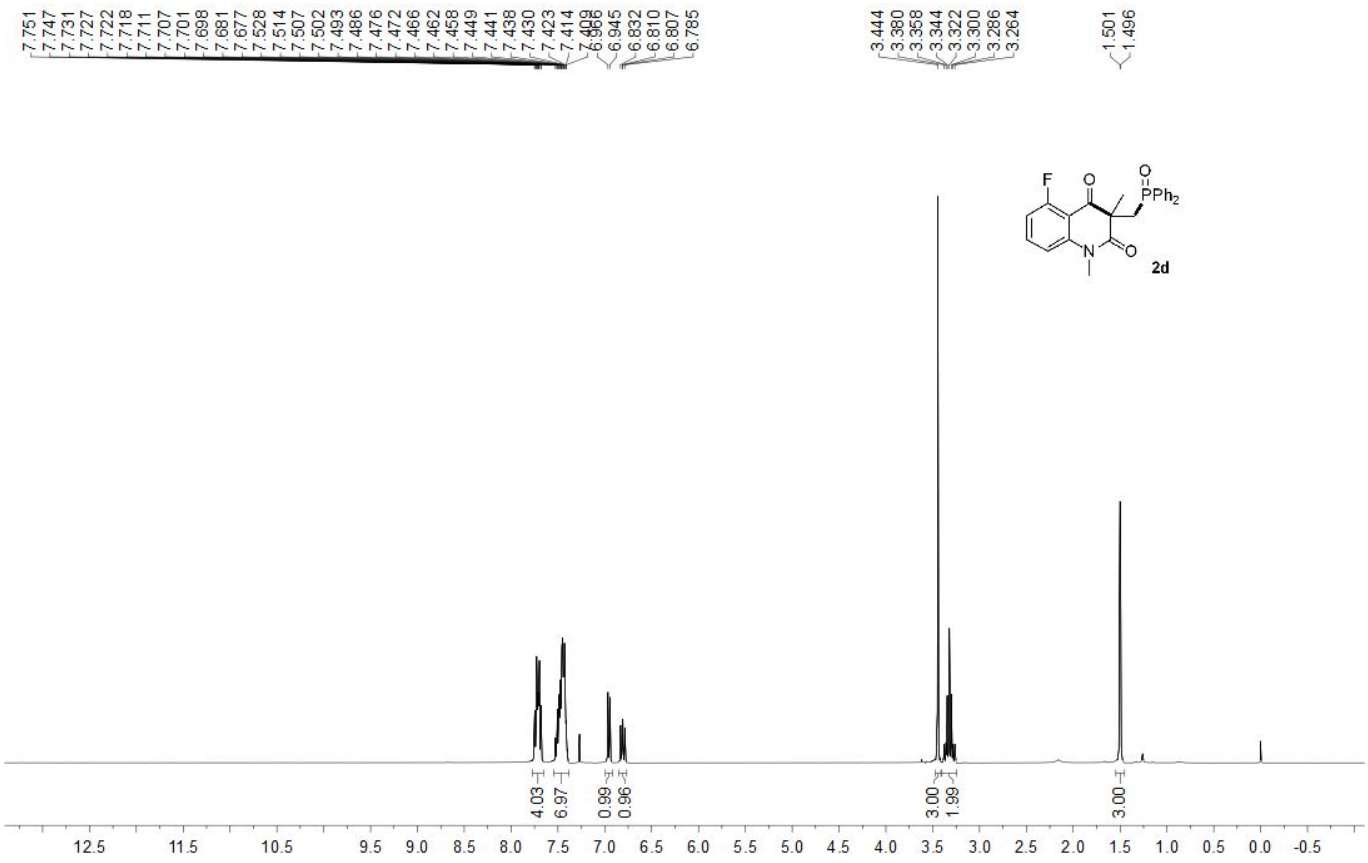
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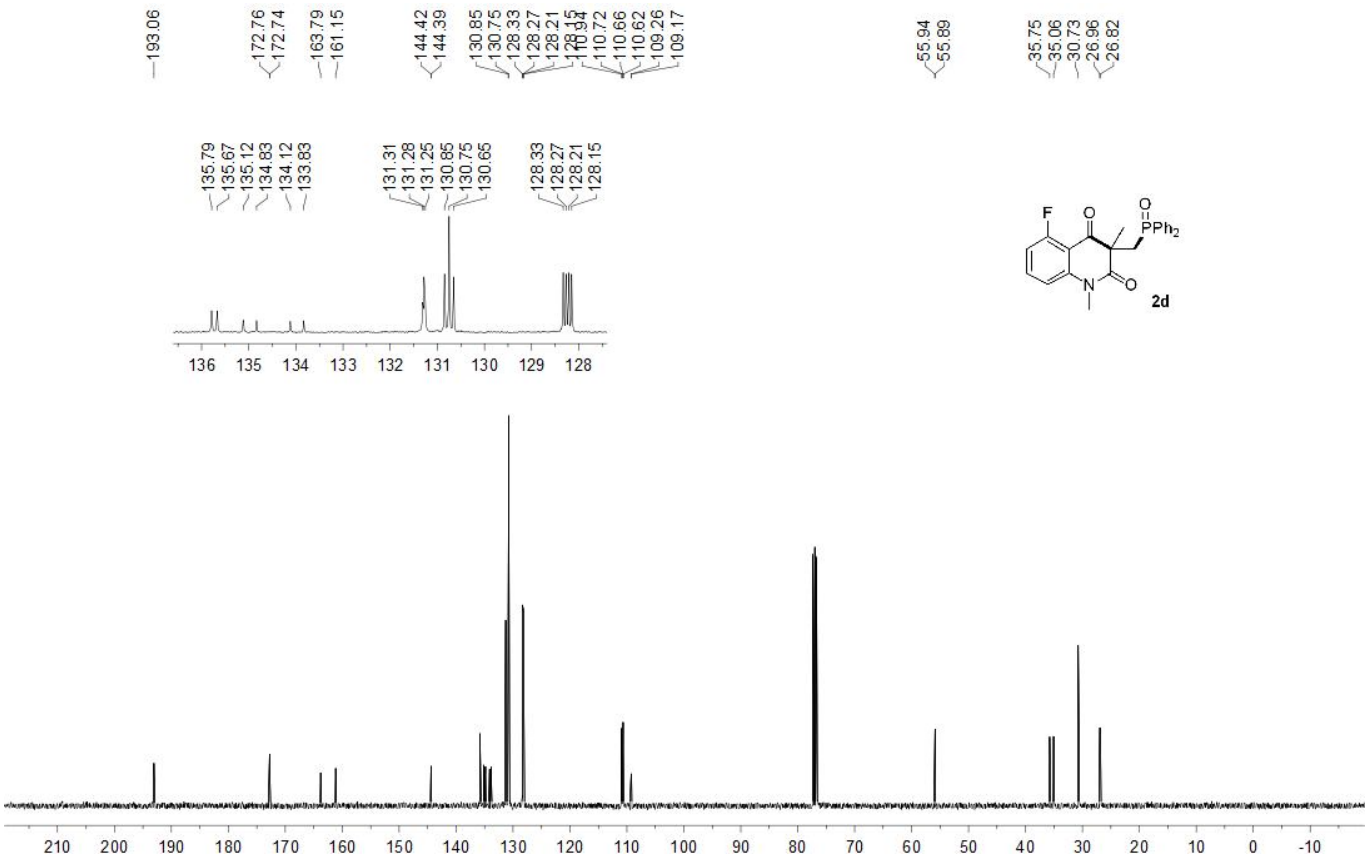
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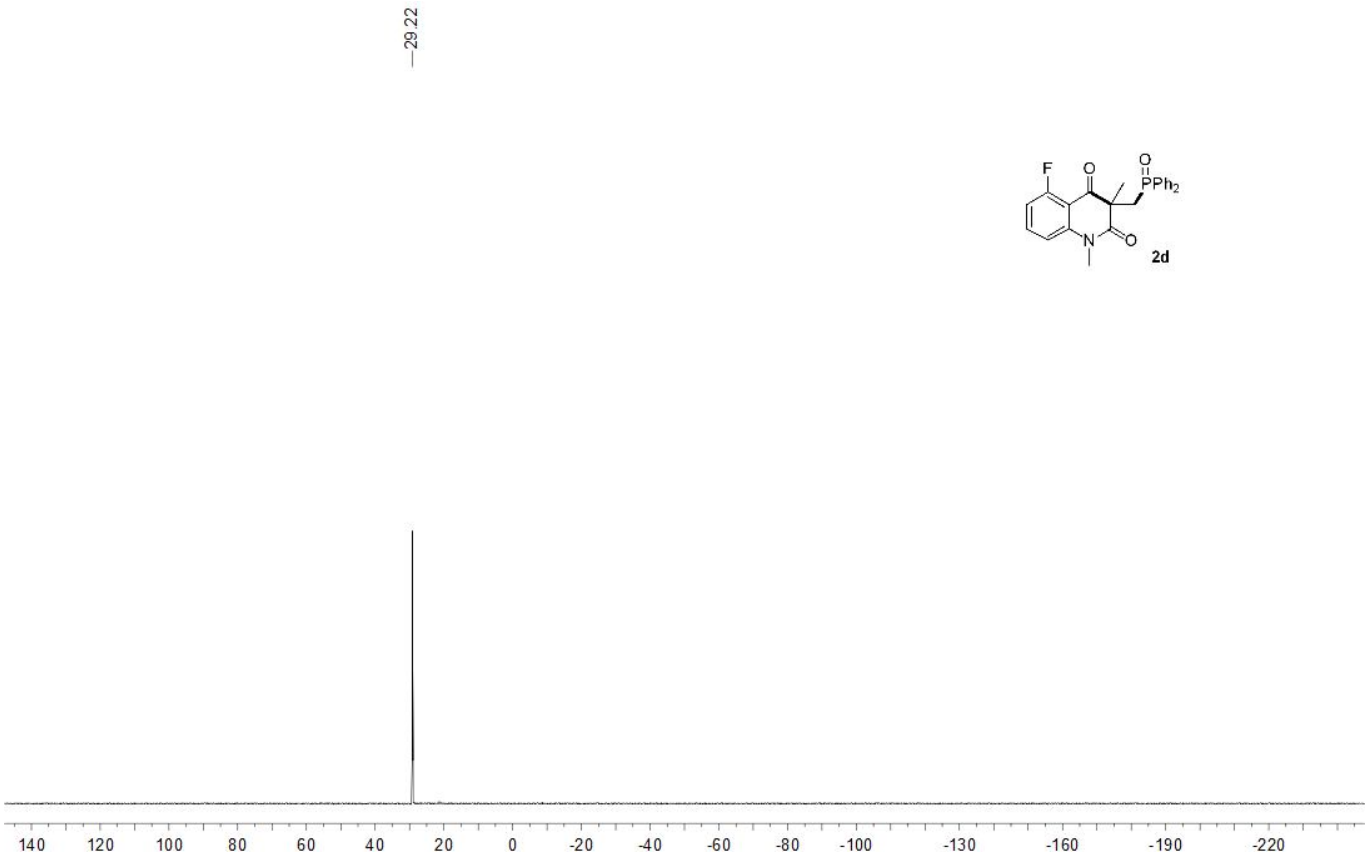
O
2d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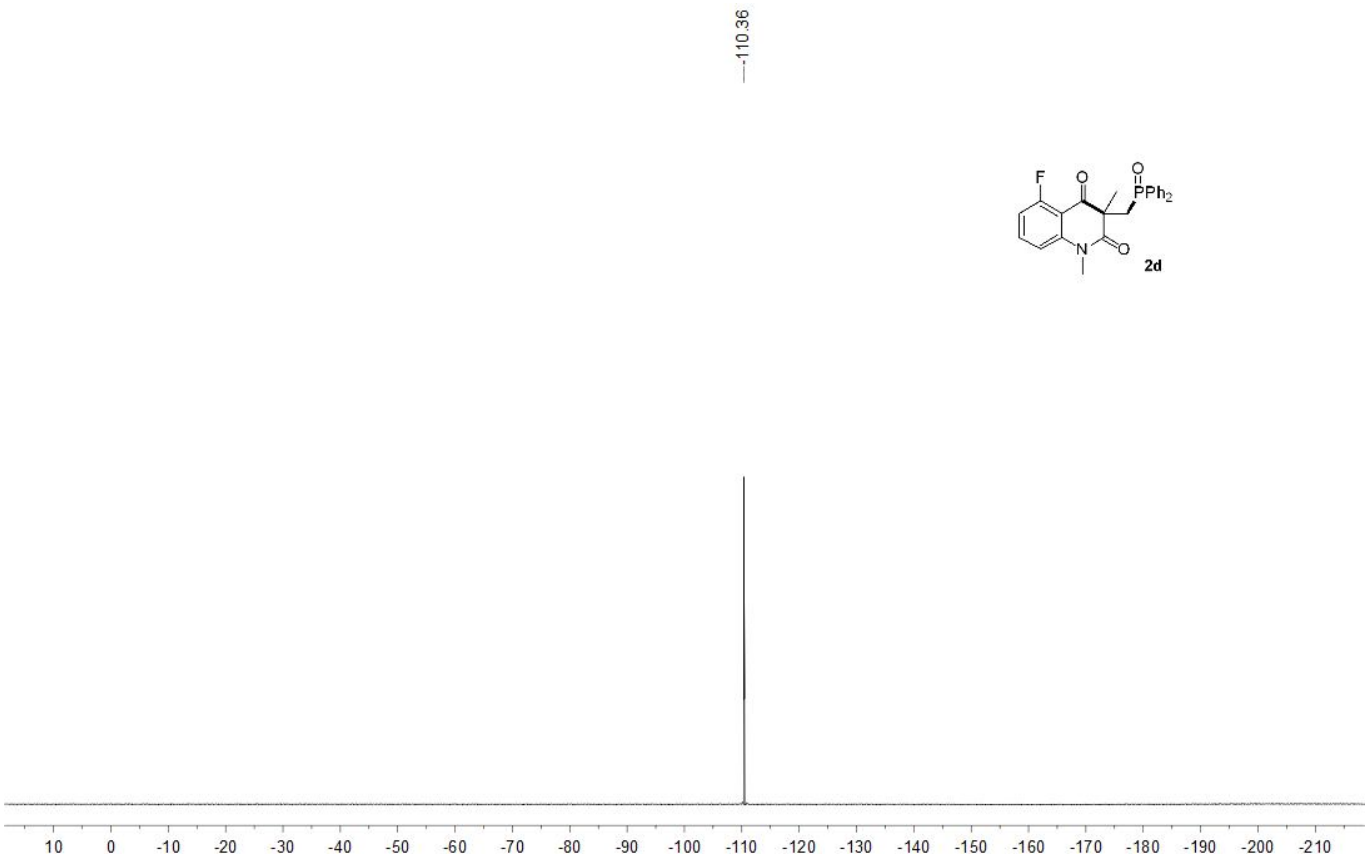
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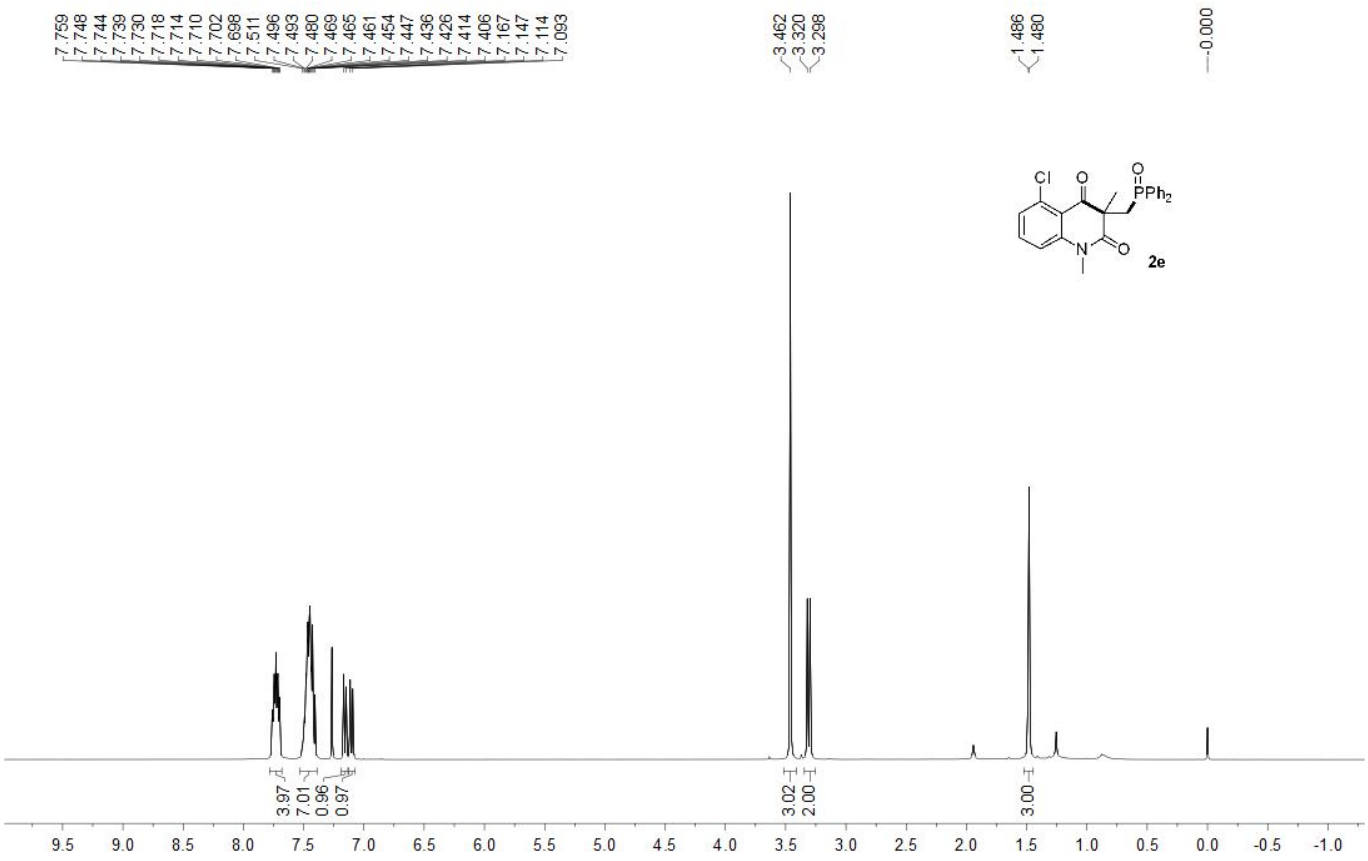
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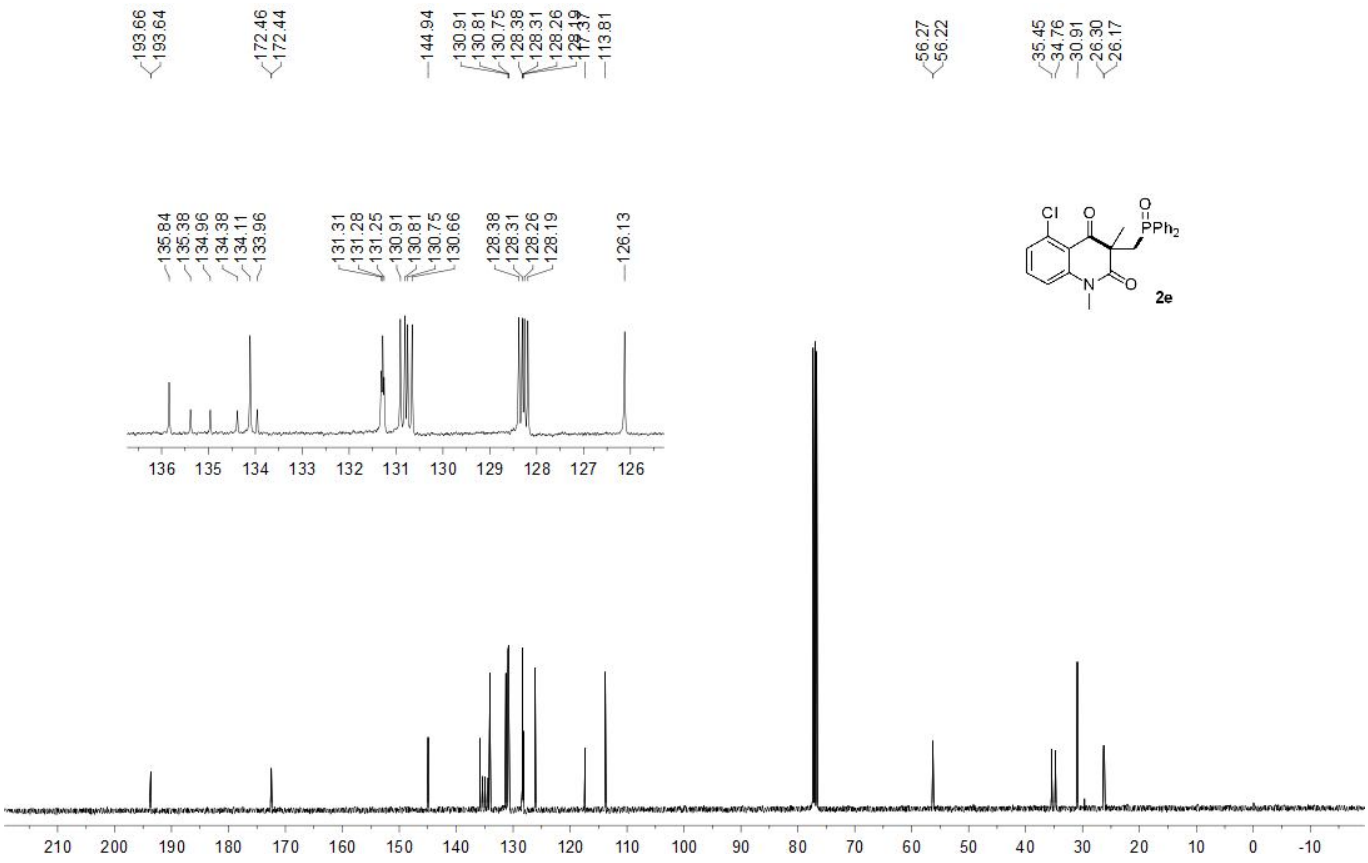
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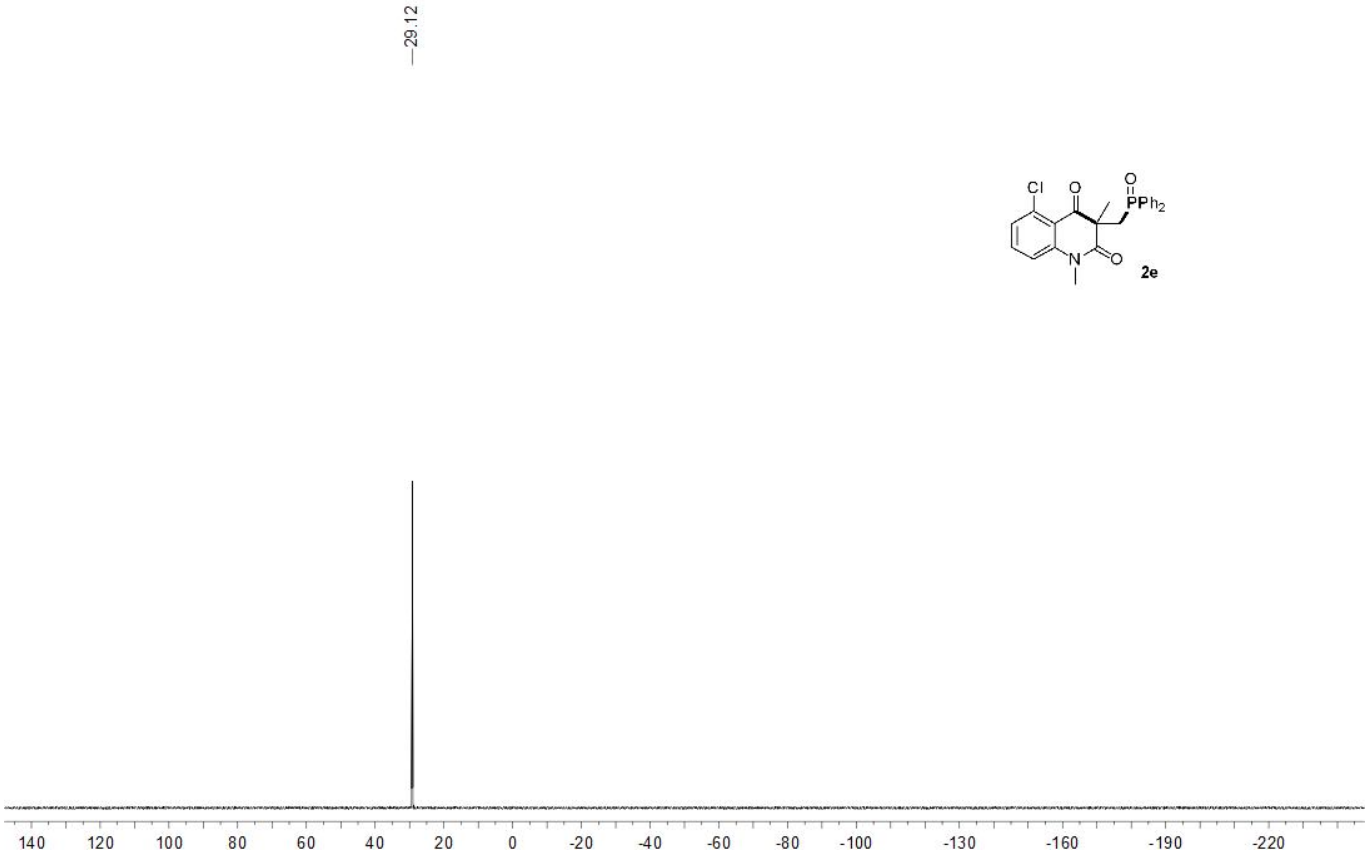
O
2e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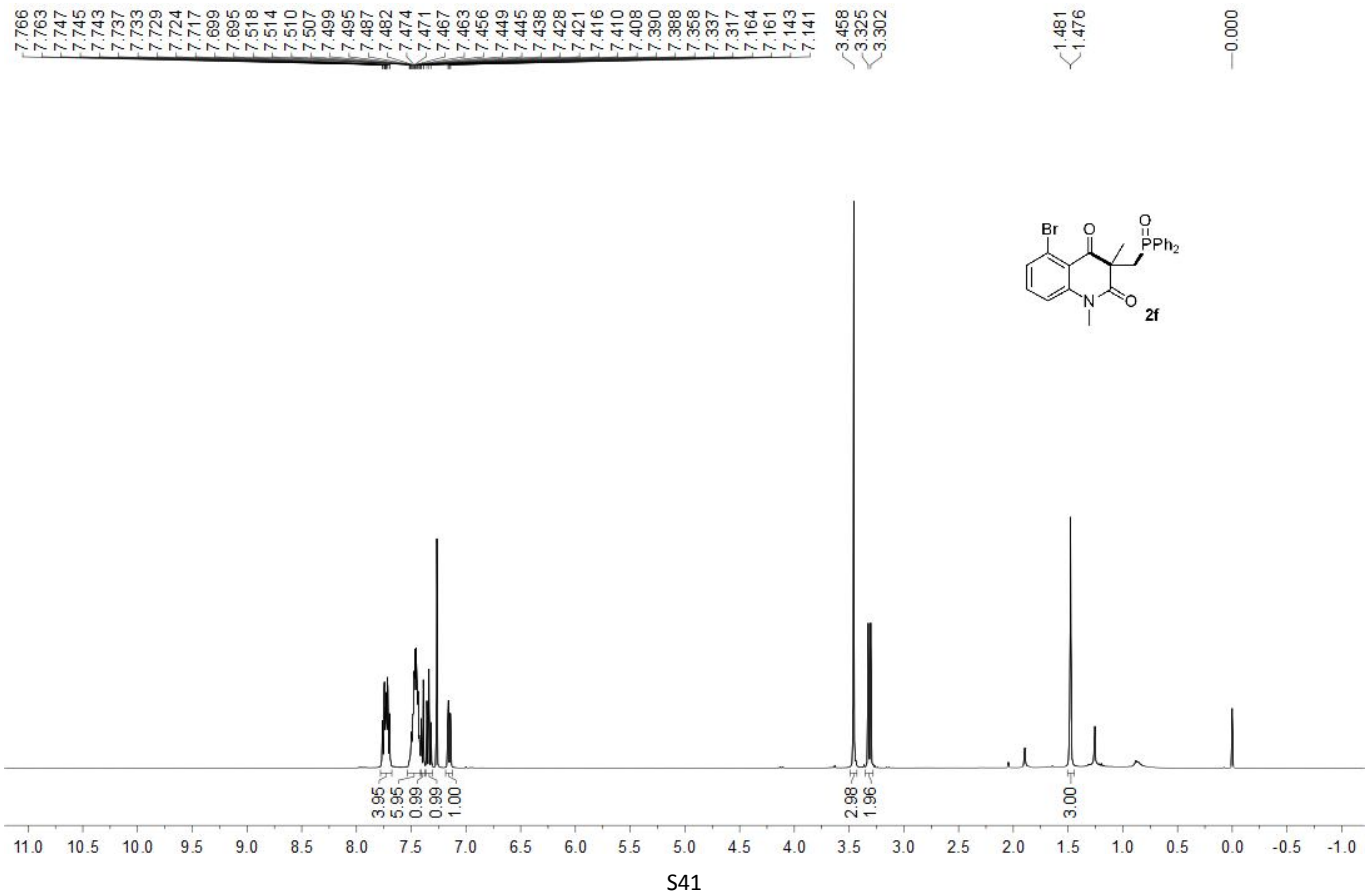
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1.476
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PPh2
N
O
2f
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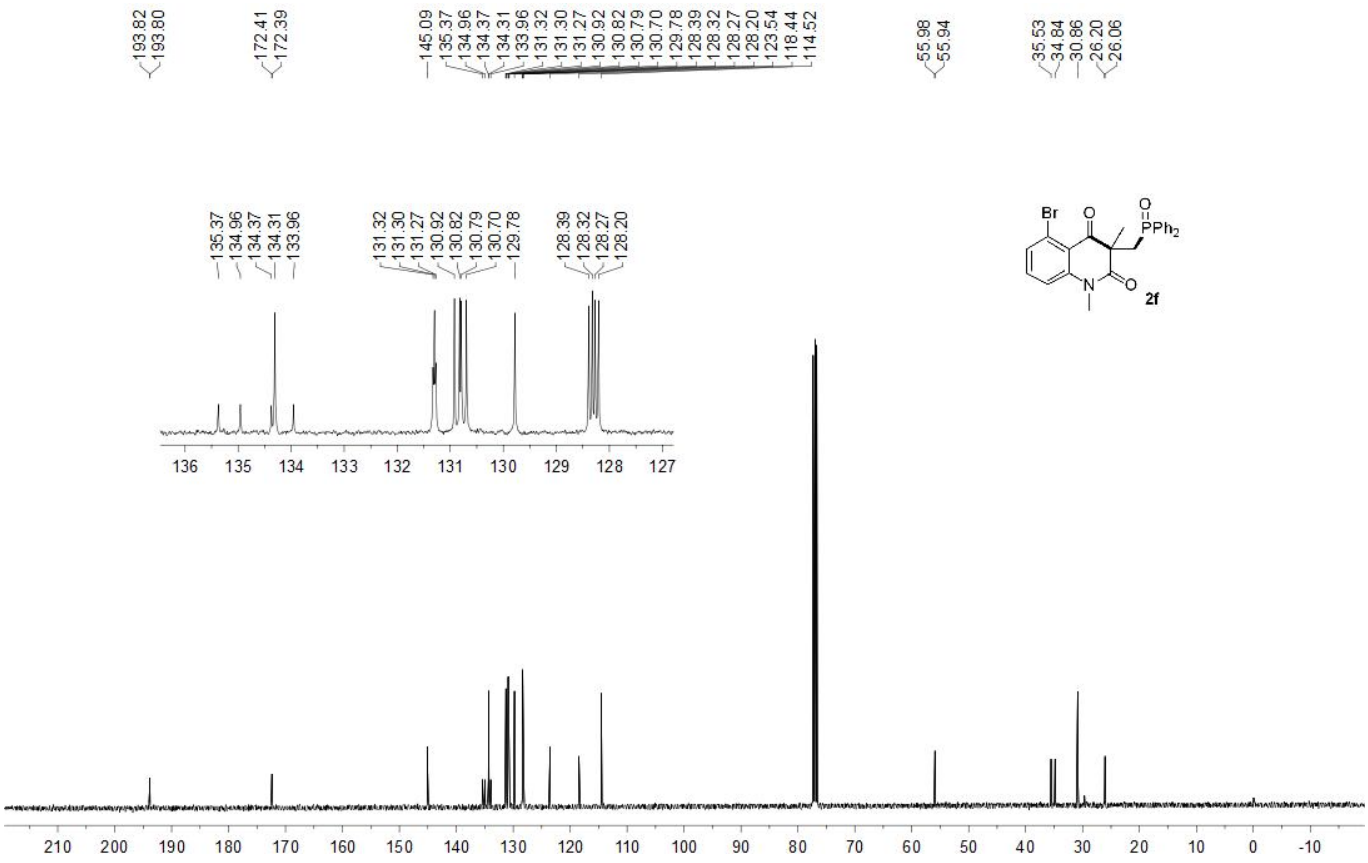
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130.70
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128.27
128.20
123.54
118.44
114.52
55.98
55.94
35.53
34.84
30.86
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O
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O
2f
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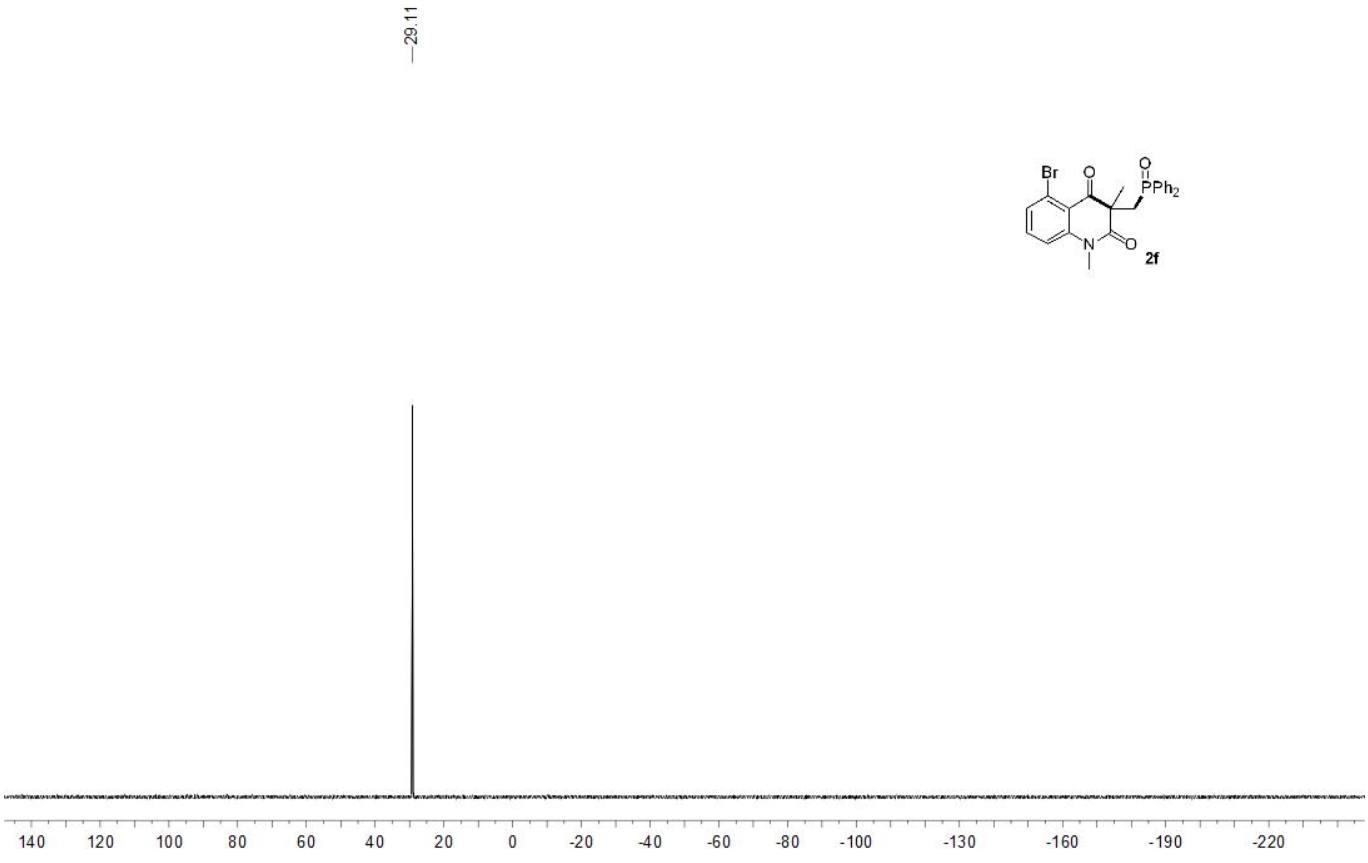
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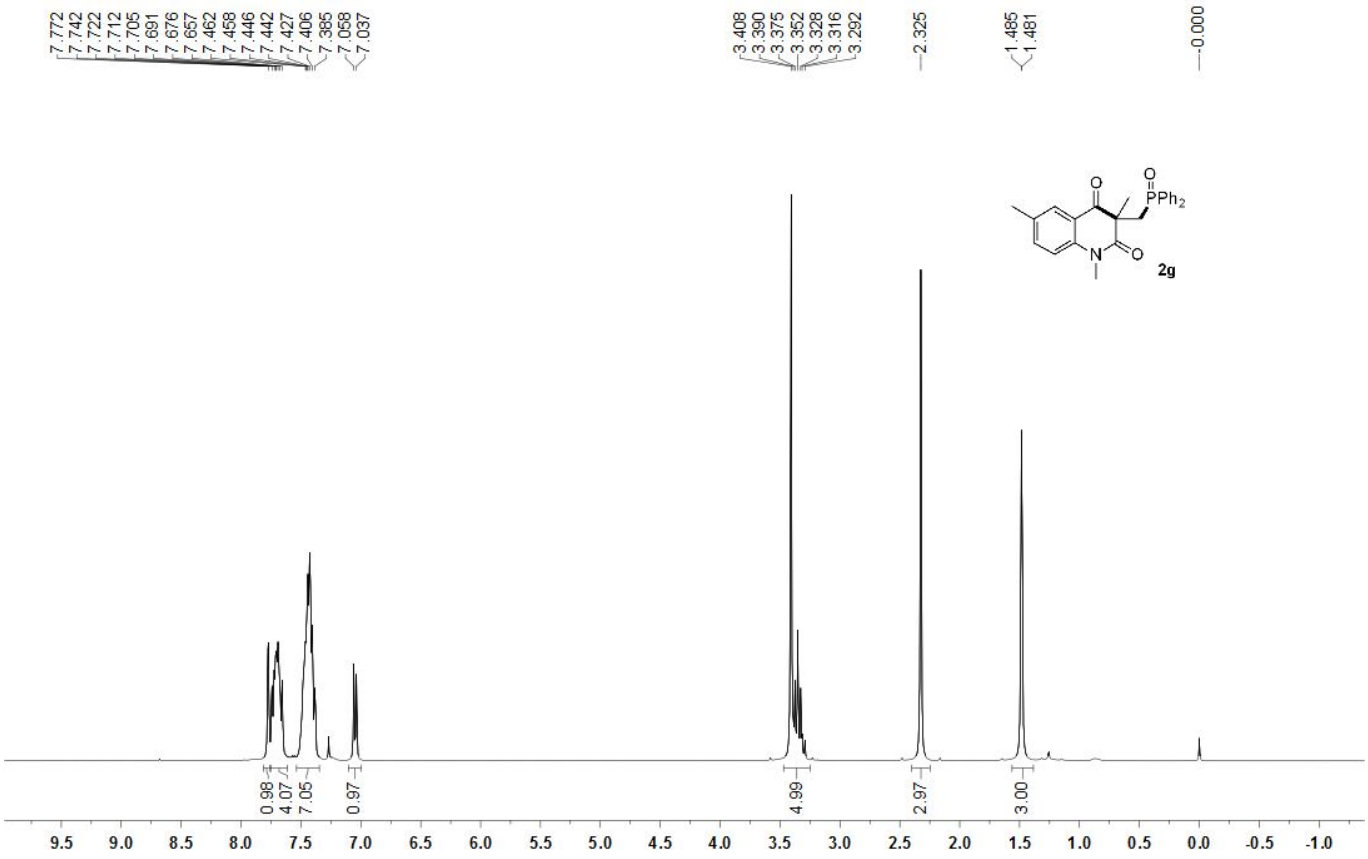
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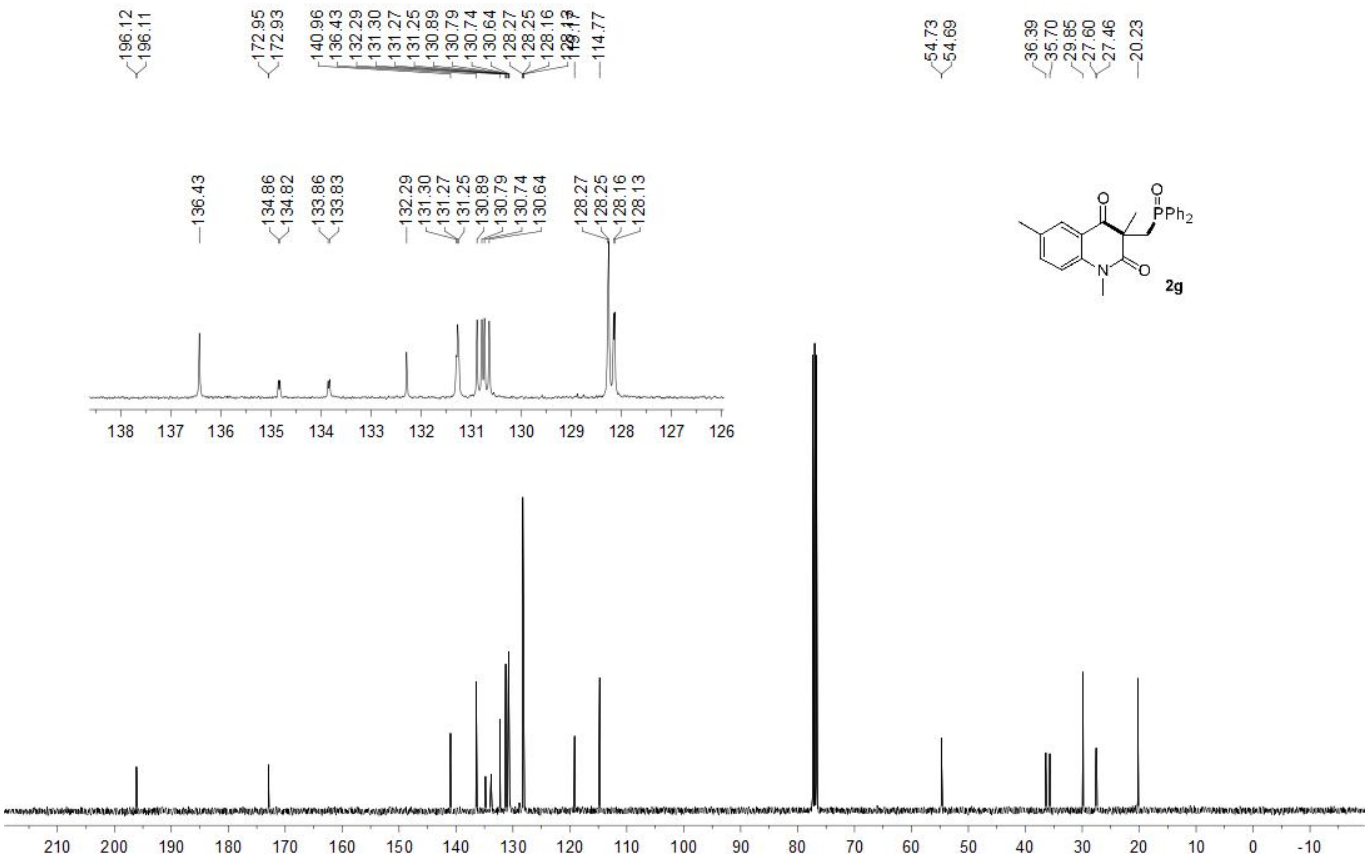
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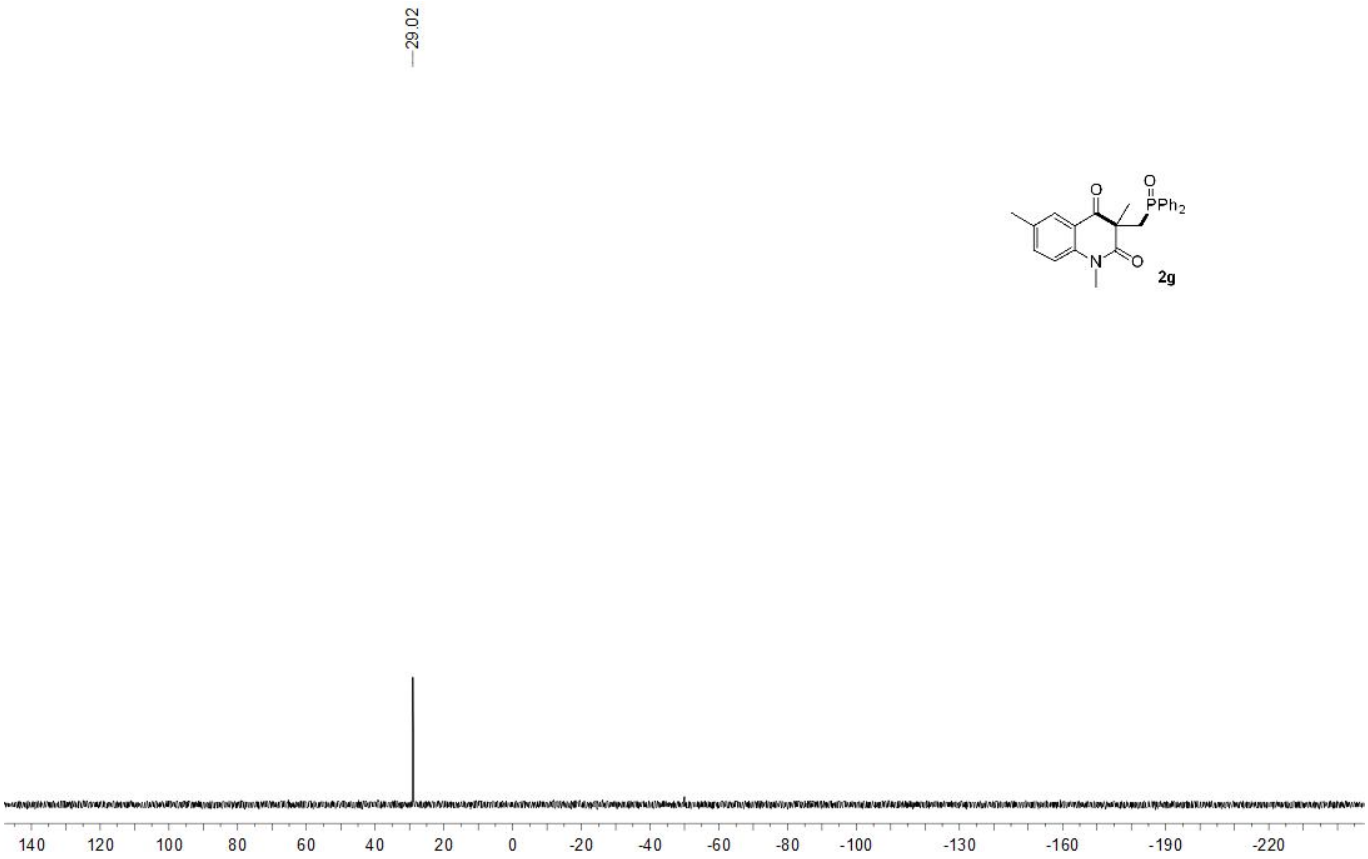
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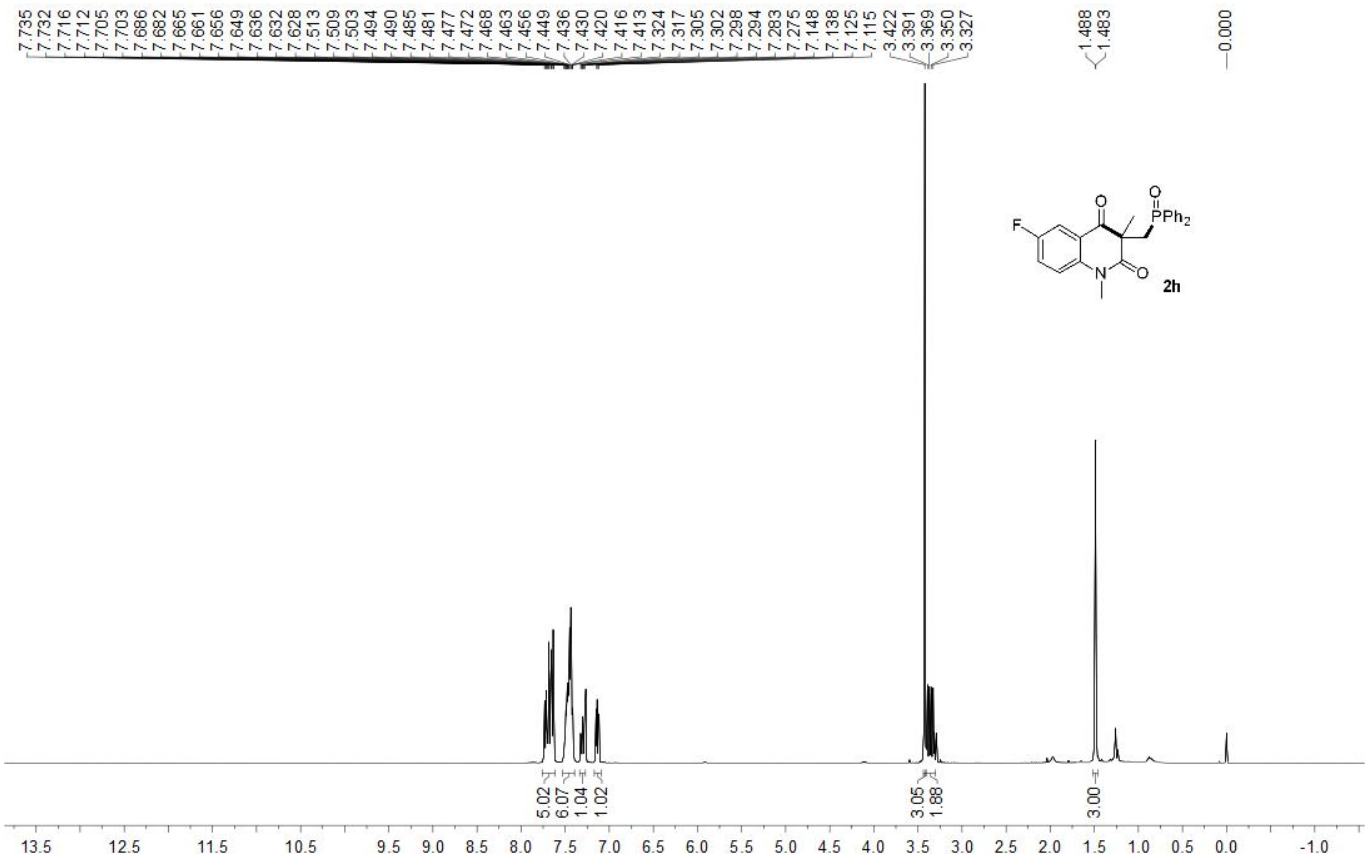
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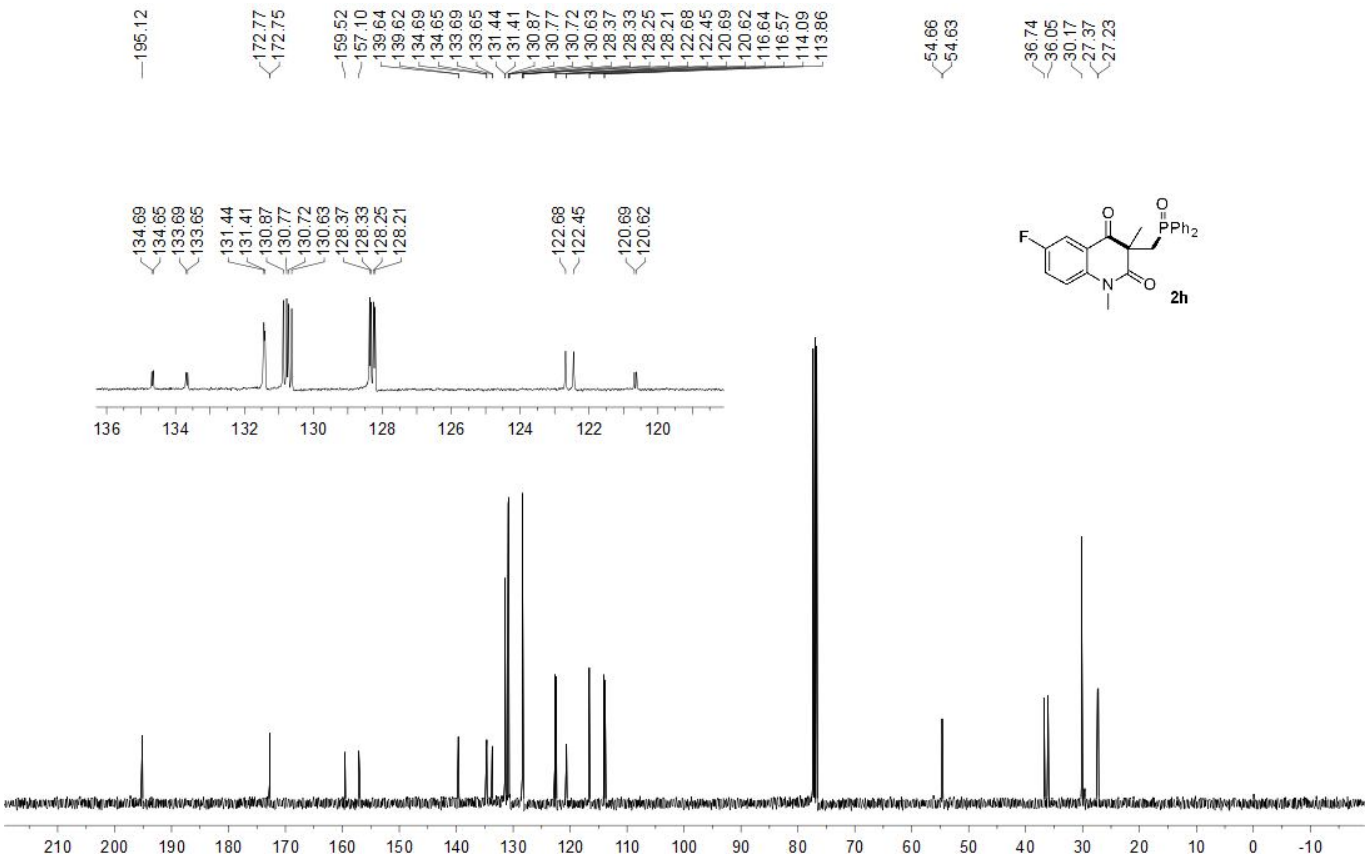
3.422 3.391 3.369 3.350 3.327
1.488 1.483
0.000
2h
5.02 6.07 1.04 1.02
3.05 1.88
3.00
13.5 12.5 11.5 10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -1.0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27.23
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PPh2
N
O
2h
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134
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130
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124
122
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200
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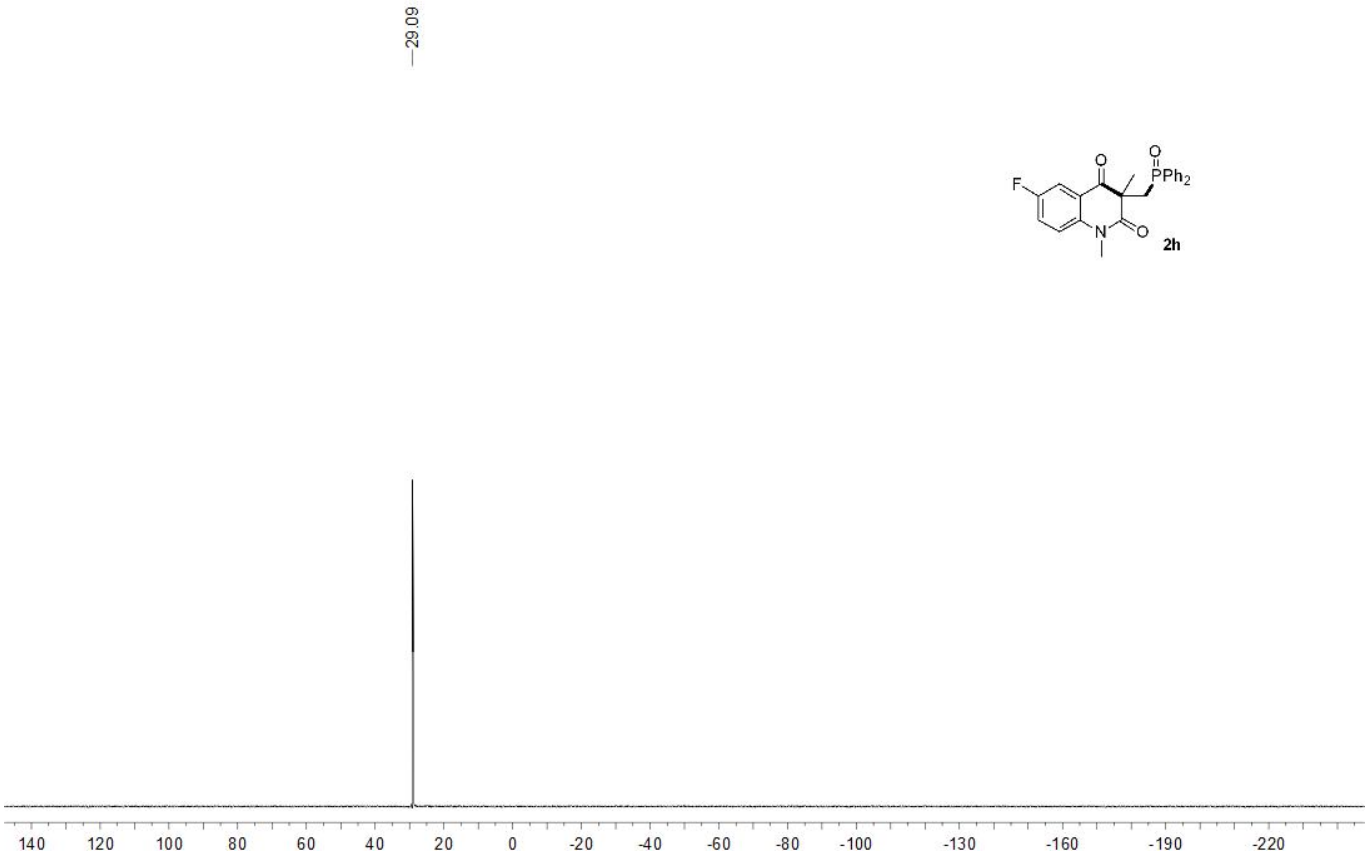
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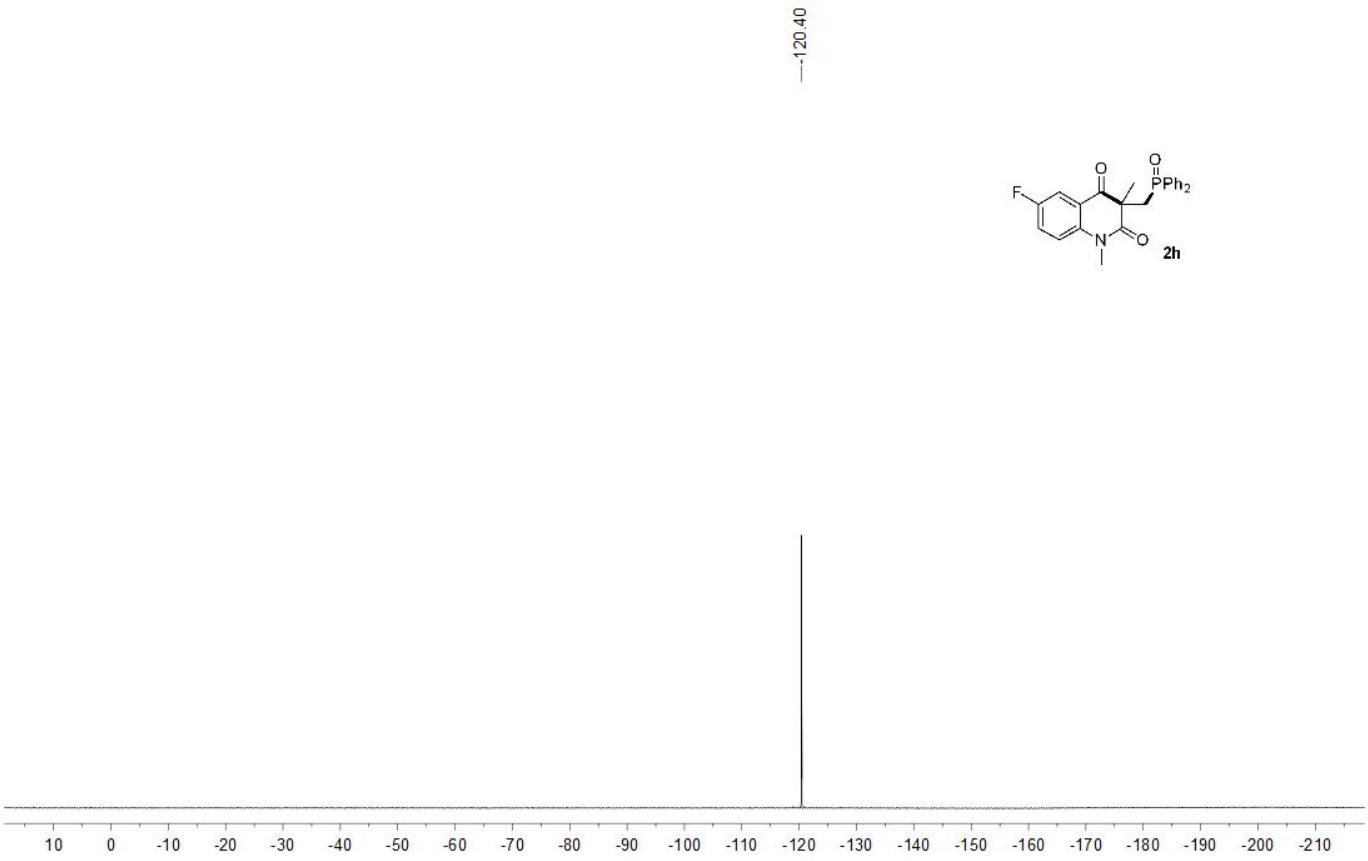
-220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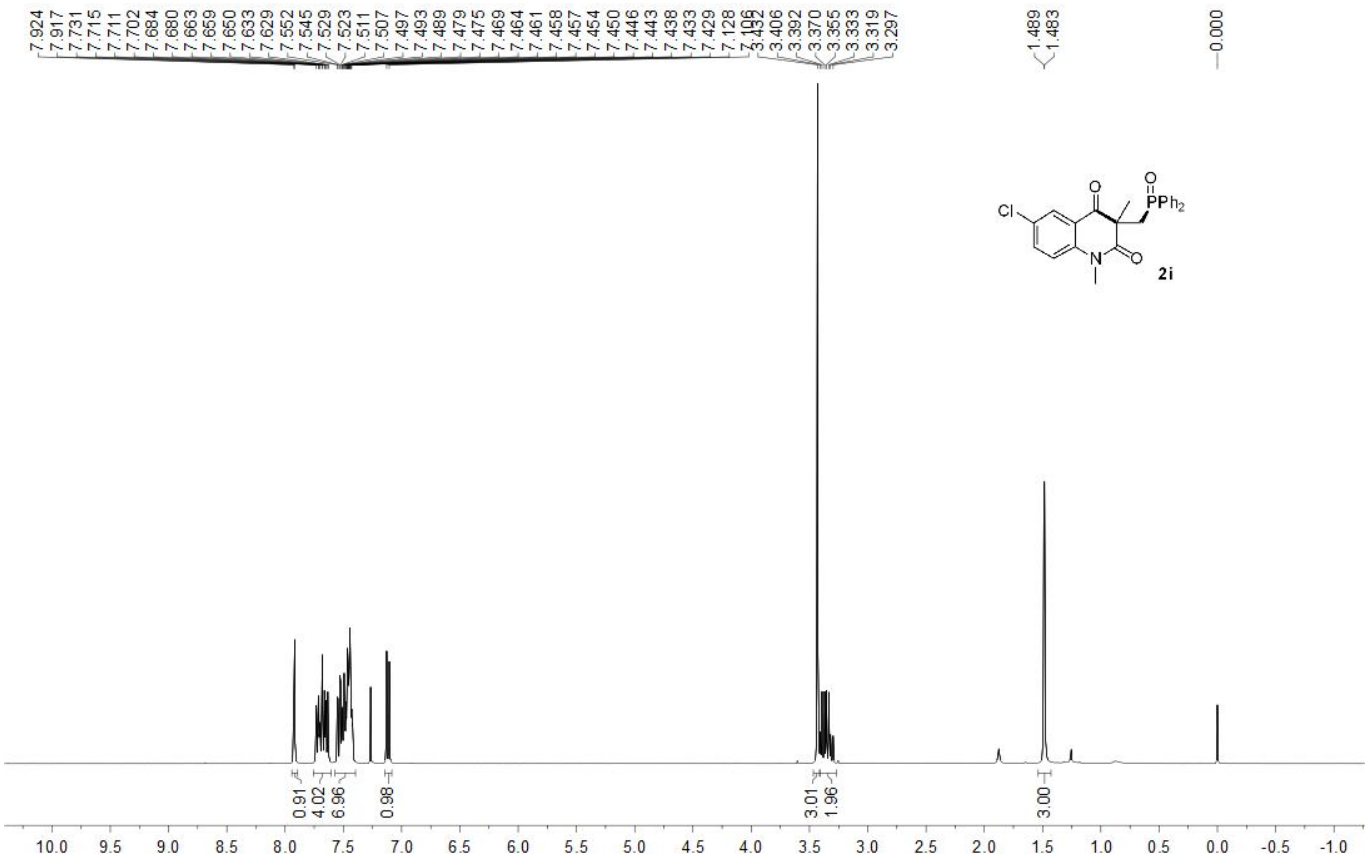
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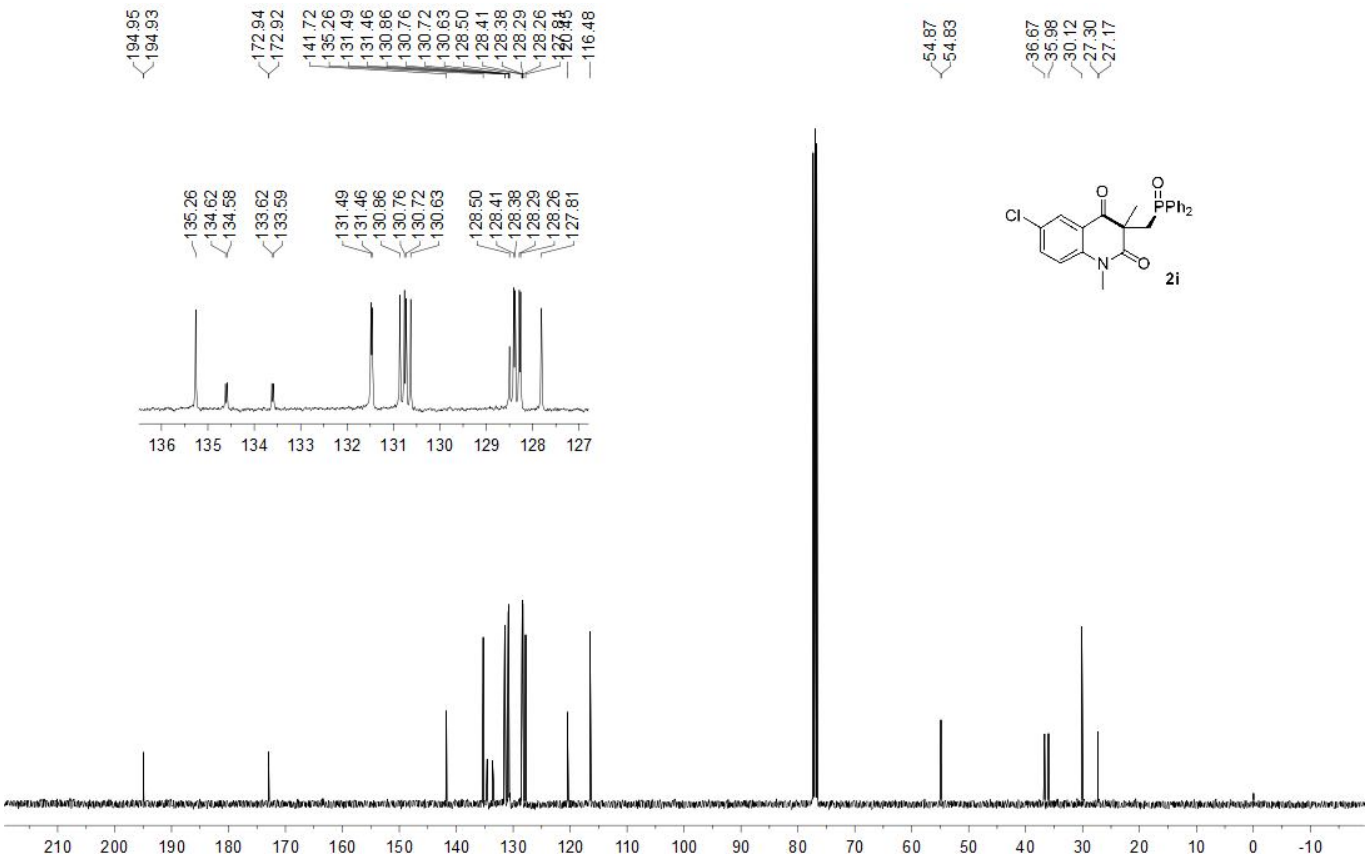
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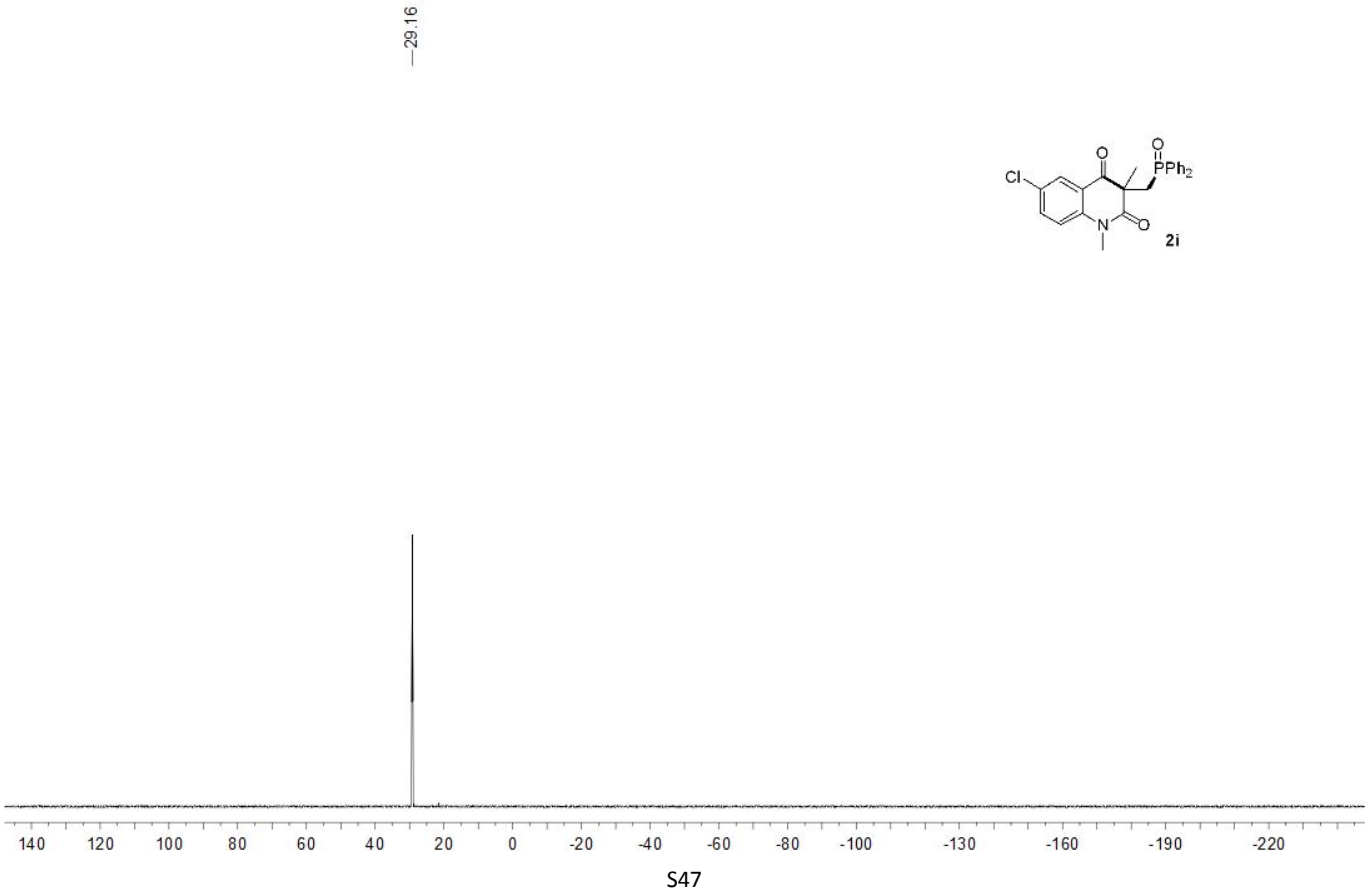
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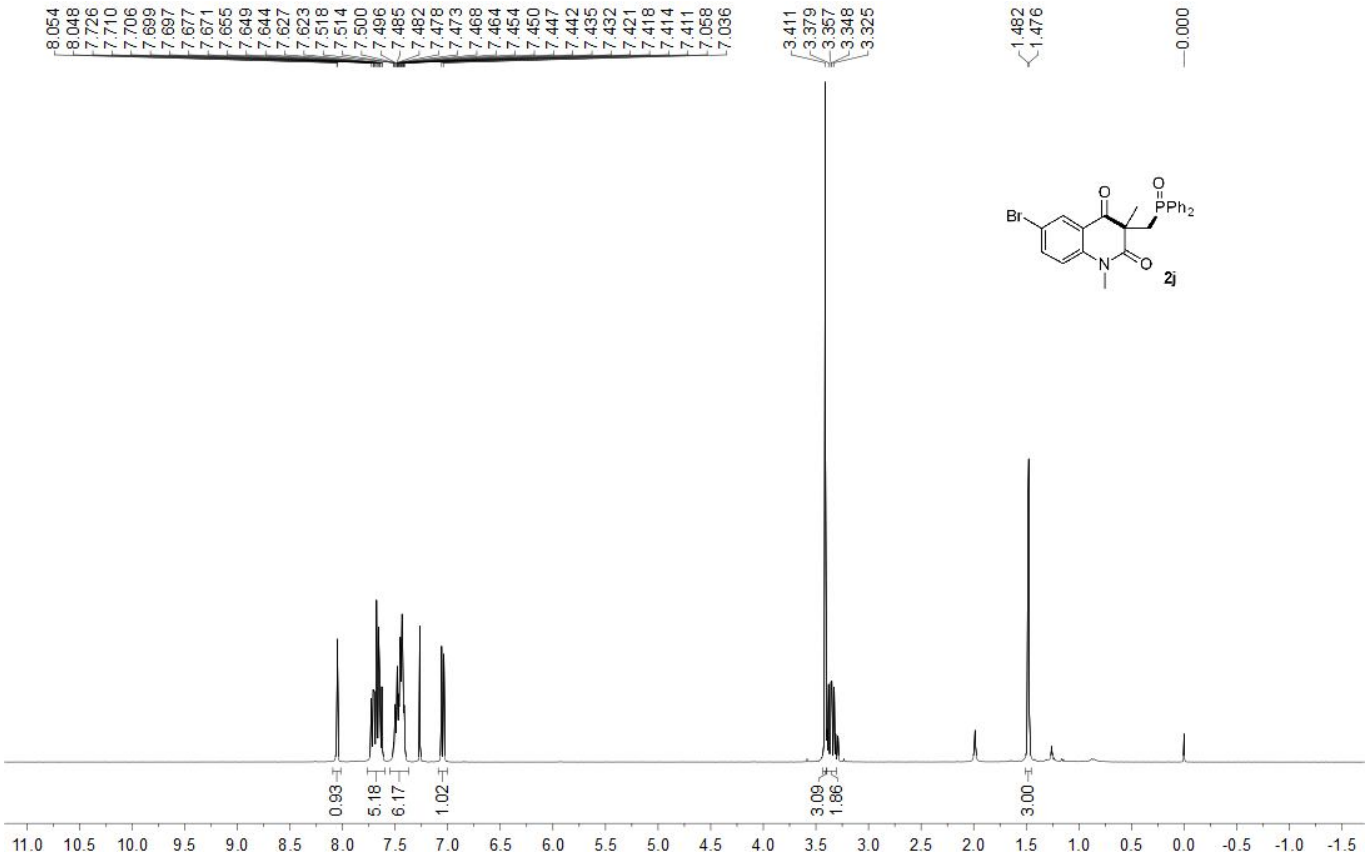
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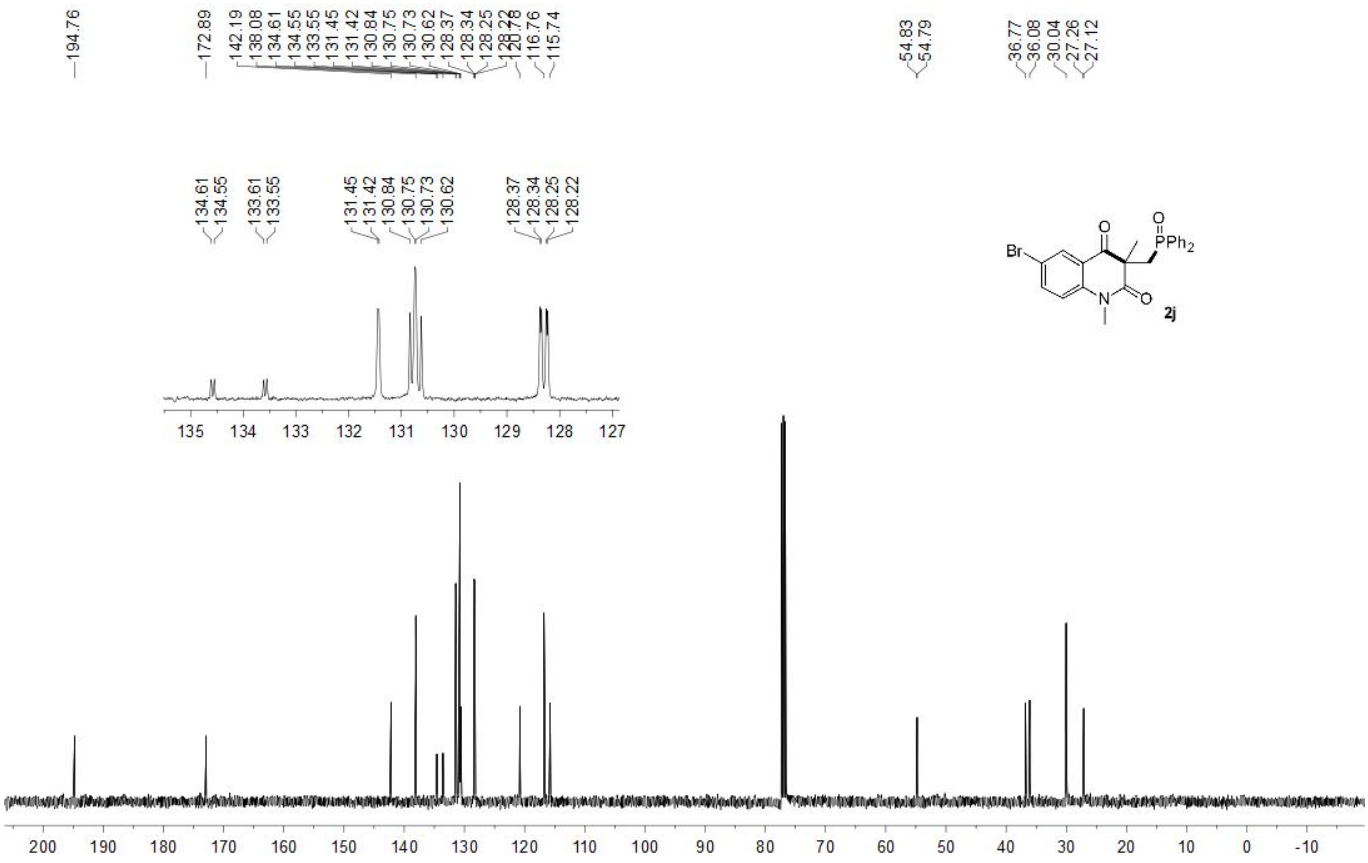
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30.04
27.26
27.12
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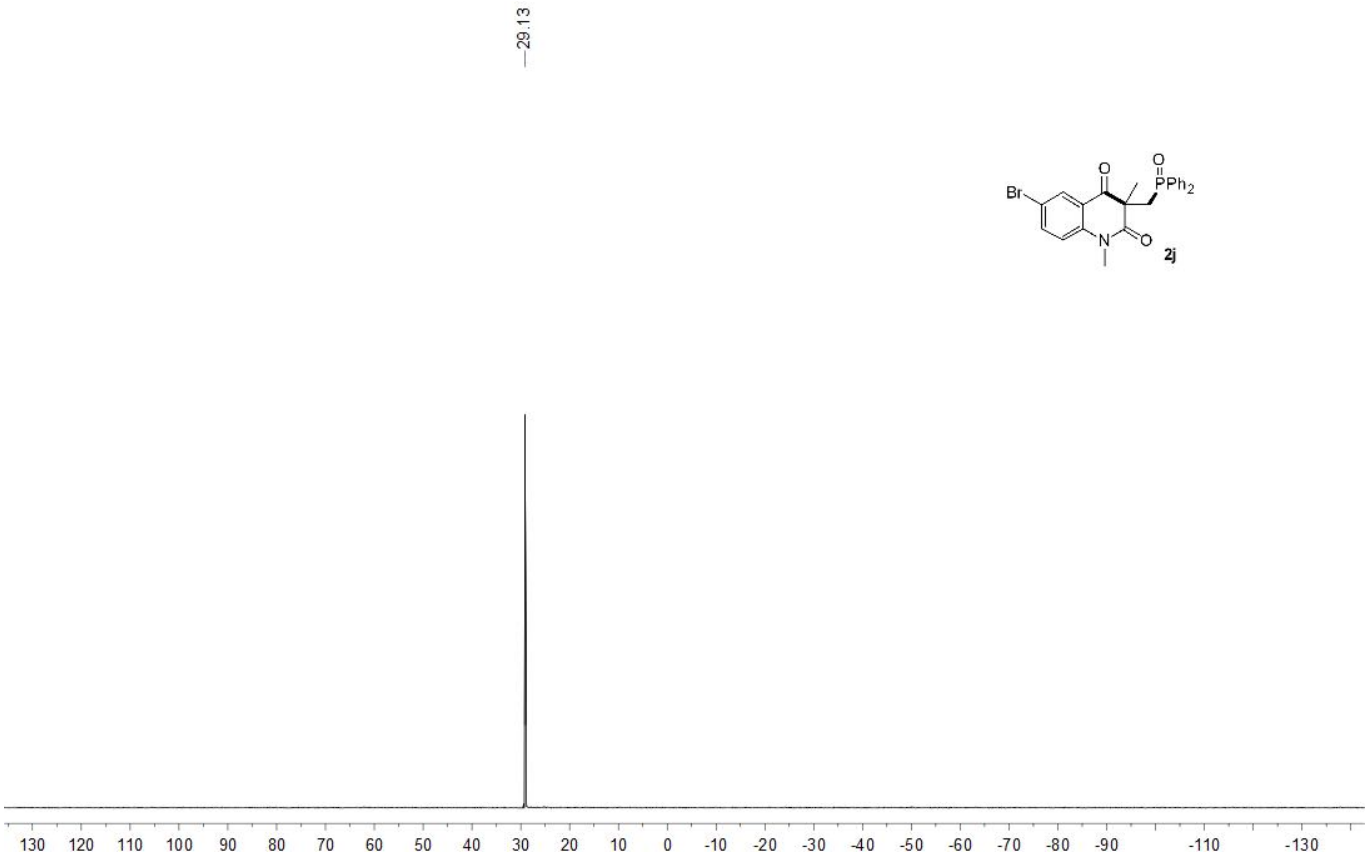
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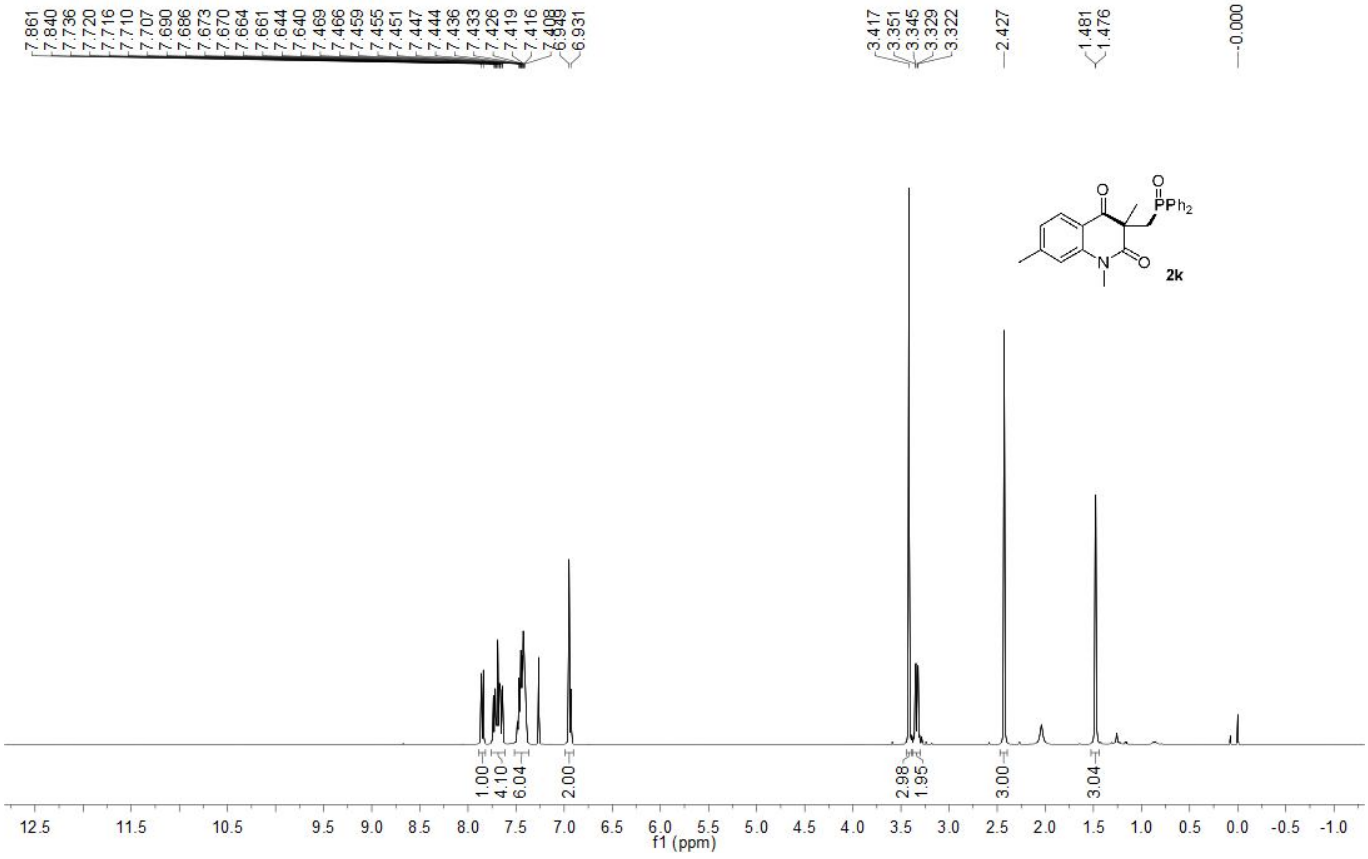
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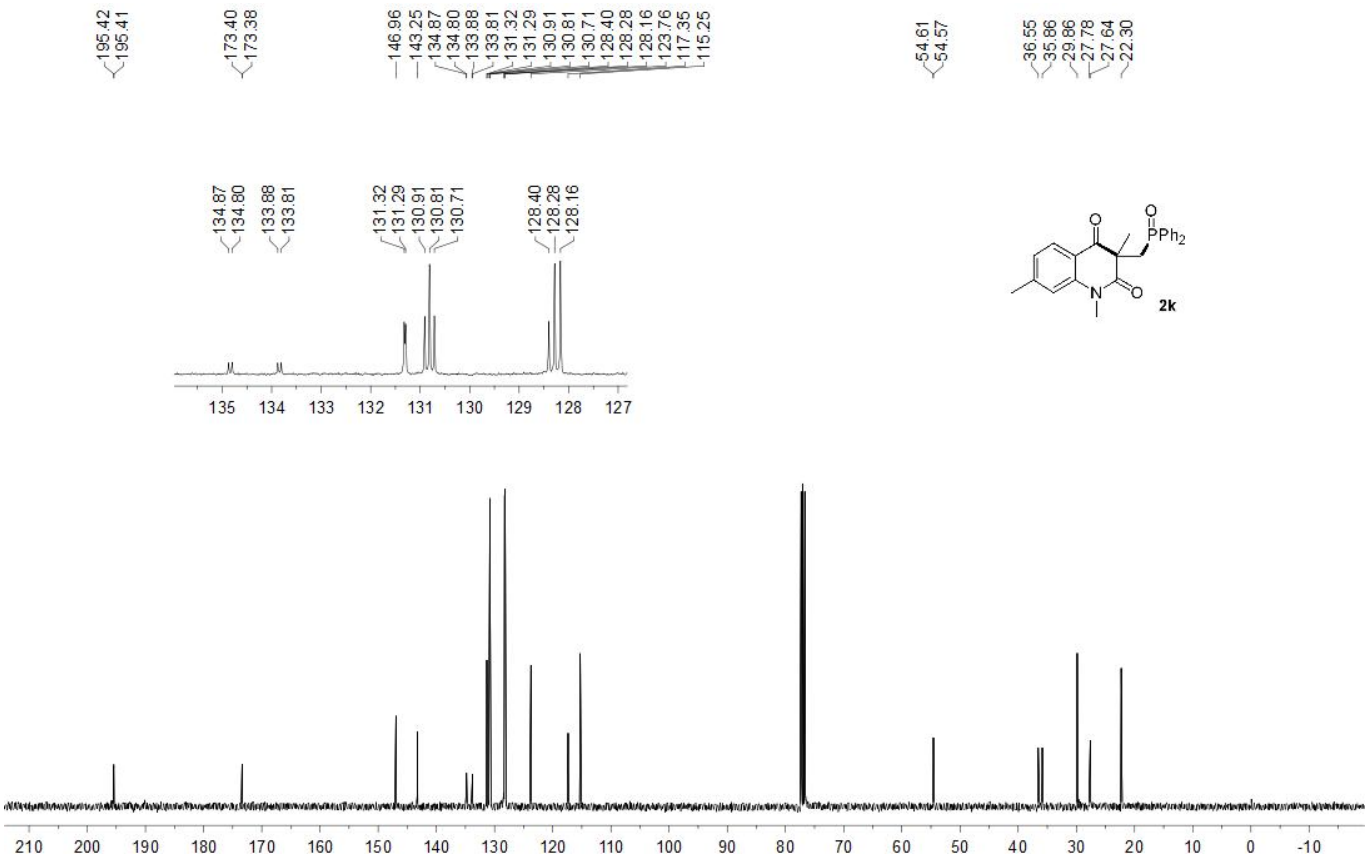
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134.80
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133.81
131.32
131.29
130.91
130.81
130.71
128.40
128.28
128.16
123.76
117.35
115.25
54.61
54.57
36.55
35.86
29.86
27.78
27.64
22.30
134.87
134.80
133.88
133.81
131.32
131.29
130.91
130.81
130.71
128.40
128.28
128.16
135 134 133 132 131 130 129 128 127
O
O
PPh2
N
O
2k
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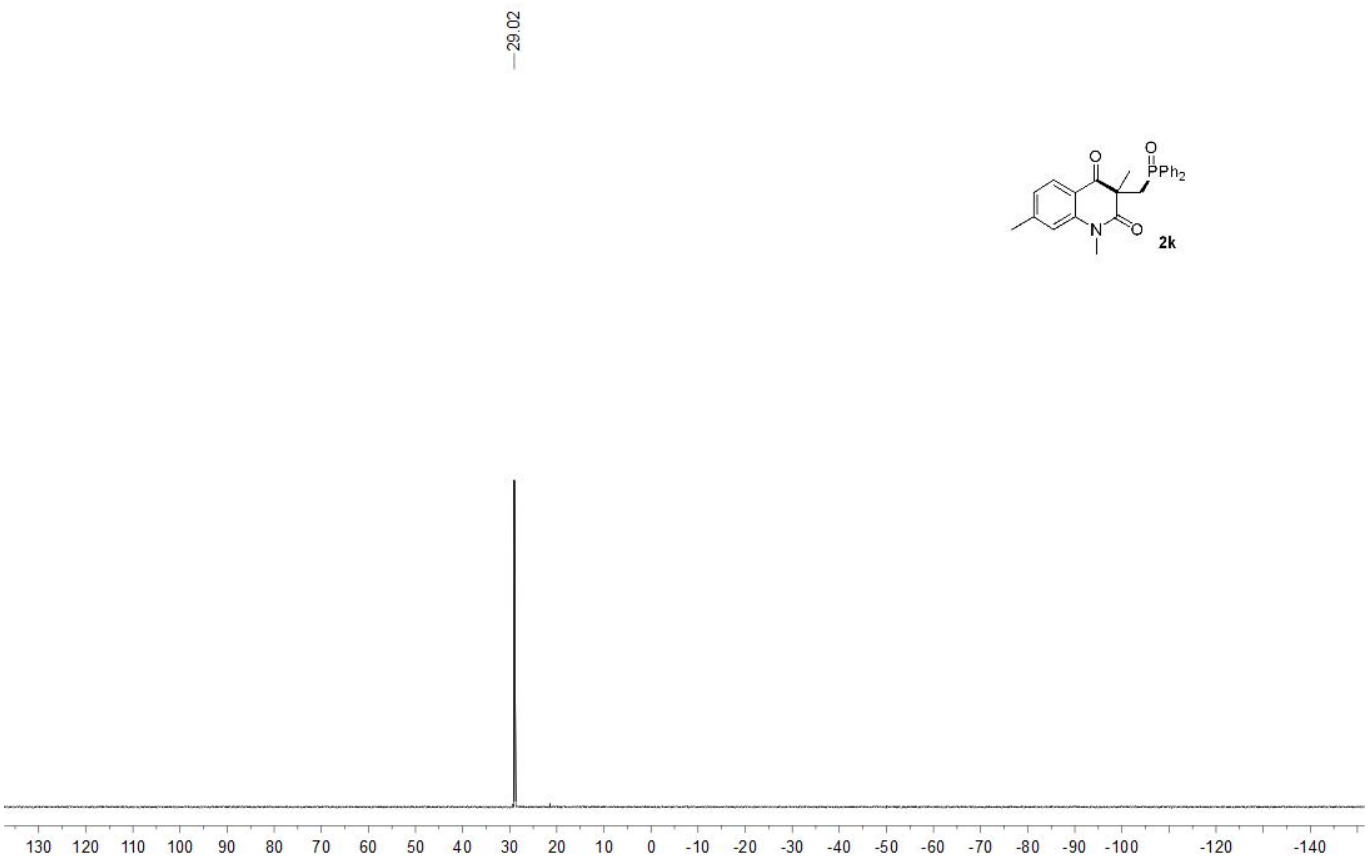
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O
PPh2
N
O
2k
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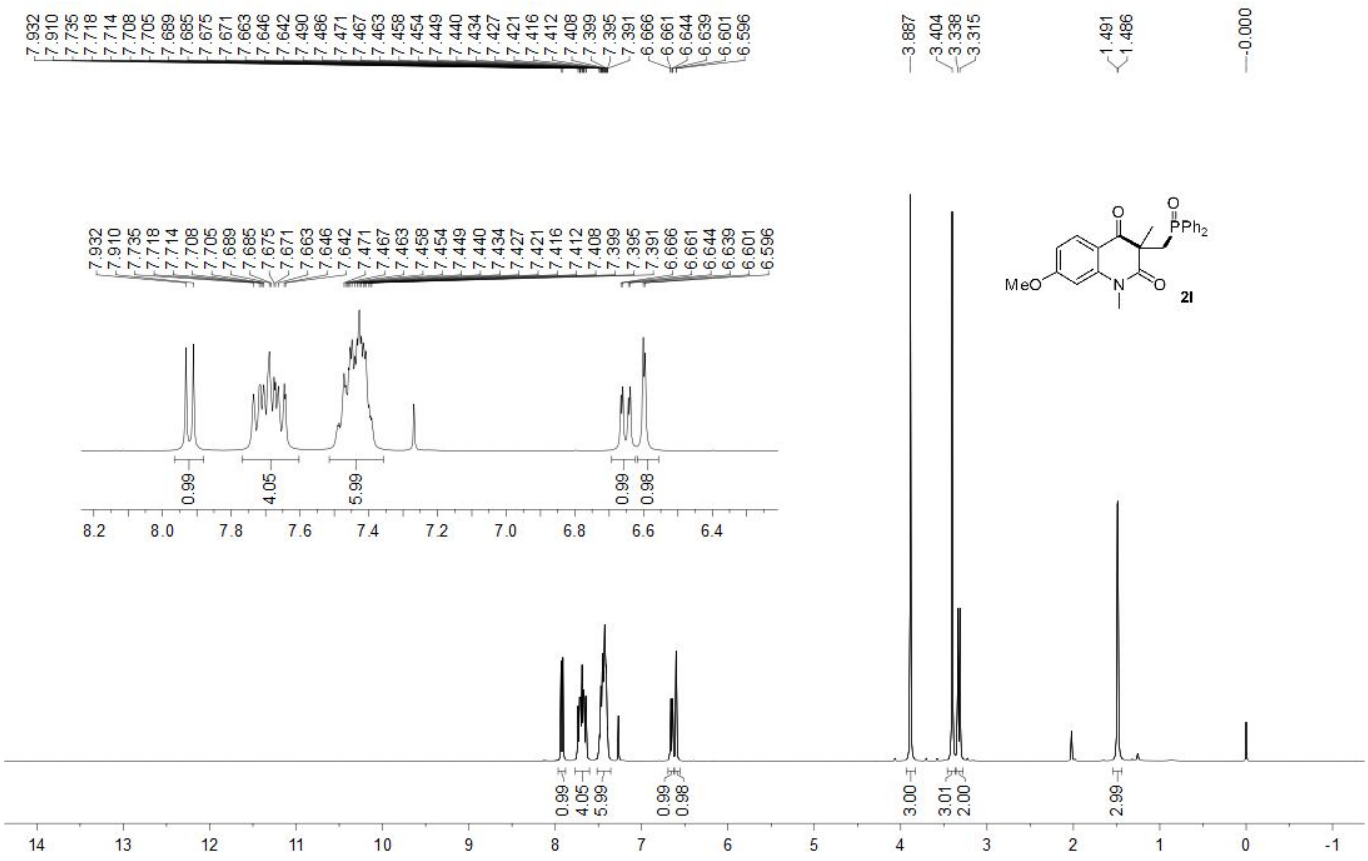
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7.646
7.642
7.490
7.486
7.471
7.467
7.463
7.458
7.454
7.449
7.440
7.434
7.427
7.421
7.416
7.412
7.408
7.399
7.395
7.391
6.666
6.661
6.644
6.639
6.601
6.596
3.887
3.404
3.338
3.315
1.491
1.486
-0.000
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PPh2
2l
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0.98
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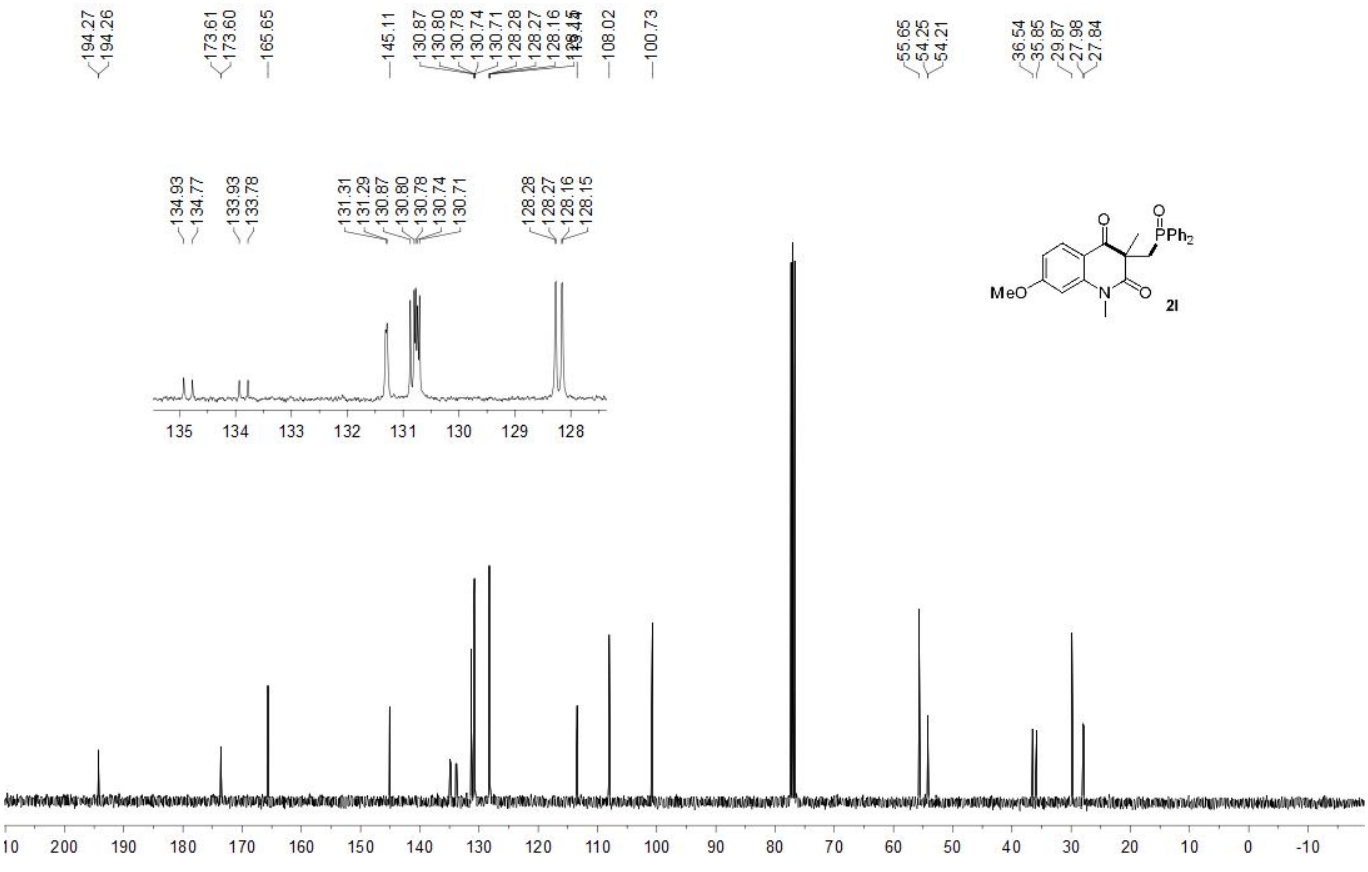
3.01
2.00
2.99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128.28
128.27
128.16
108.02
100.73
55.65
54.25
54.21
36.54
35.85
29.87
27.98
27.84
134.93
134.77
133.93
133.78
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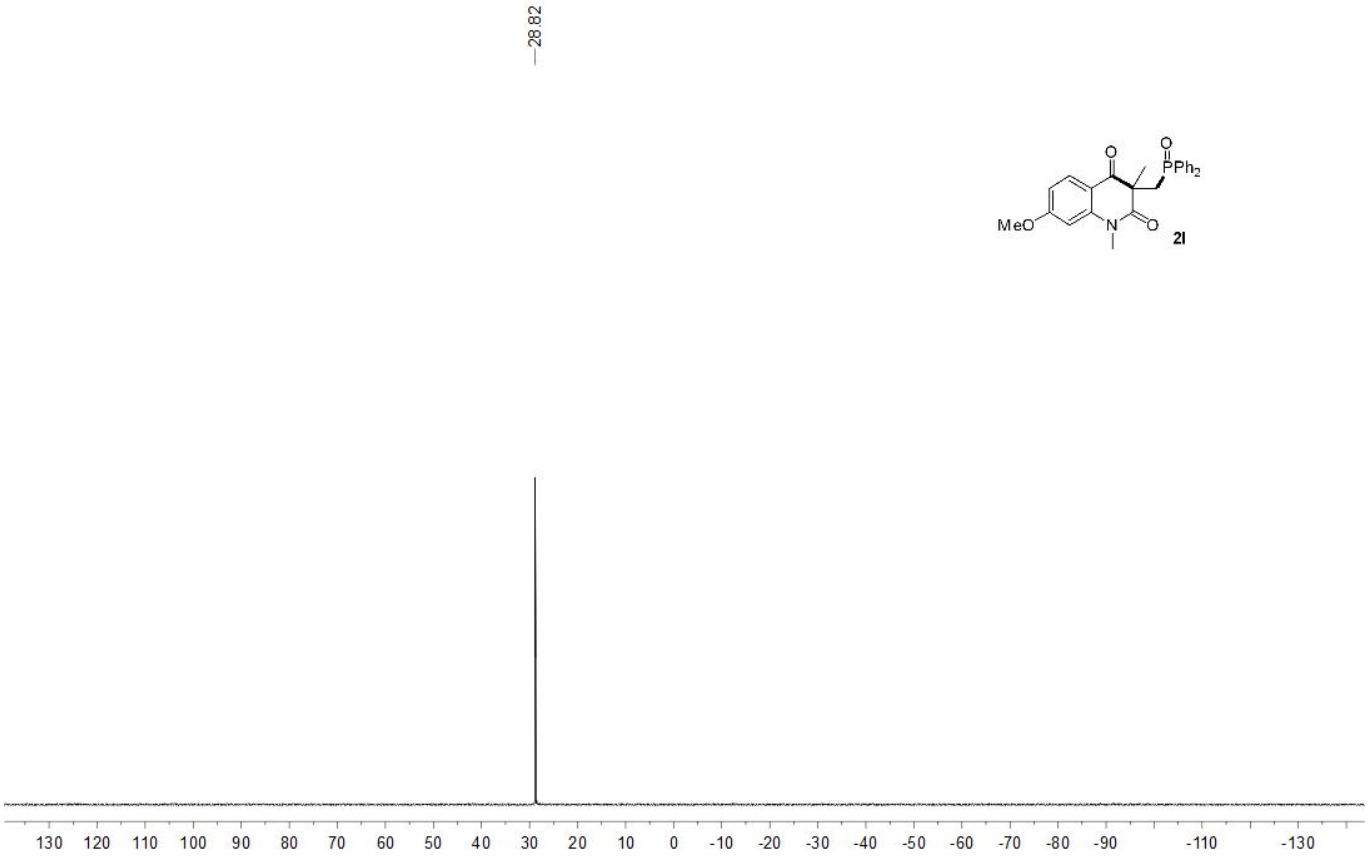
MeO
PPh2
2l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MeO
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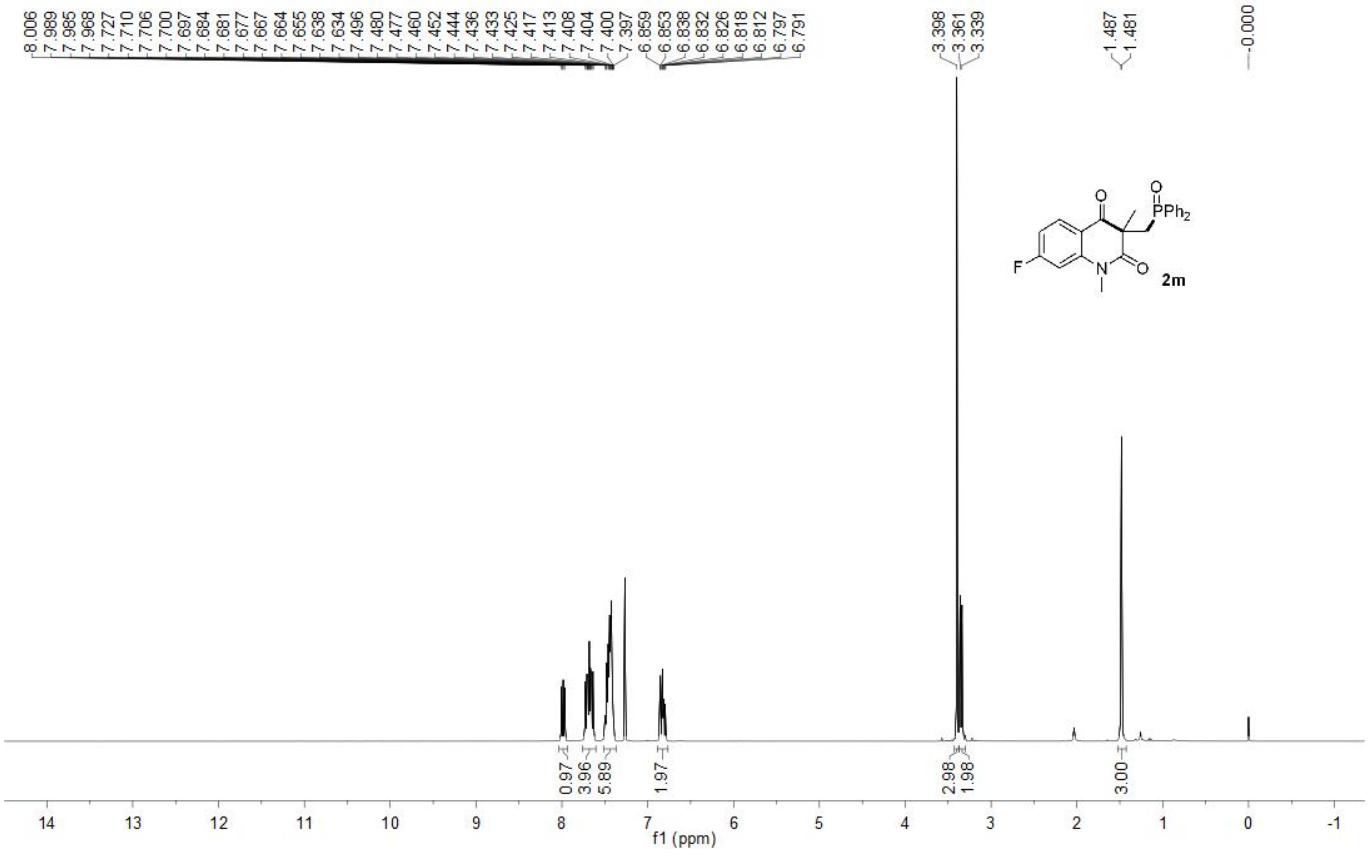
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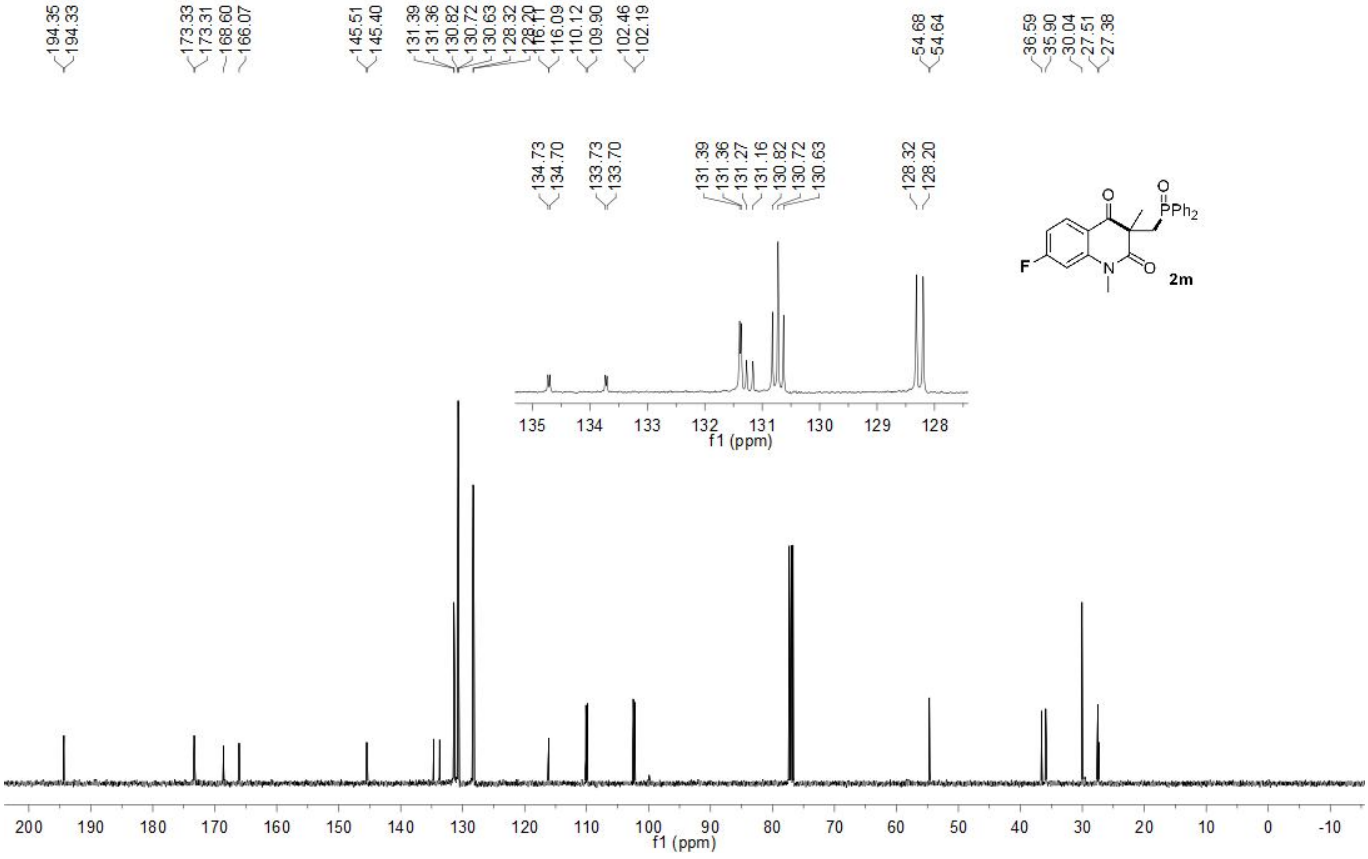
3.398 3.361 3.339
1.487 1.481
0.000
F
2m
0.97 3.96 5.89 1.97 2.98 1.98 3.00
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)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194.33
173.33
173.31
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166.07
145.51
145.40
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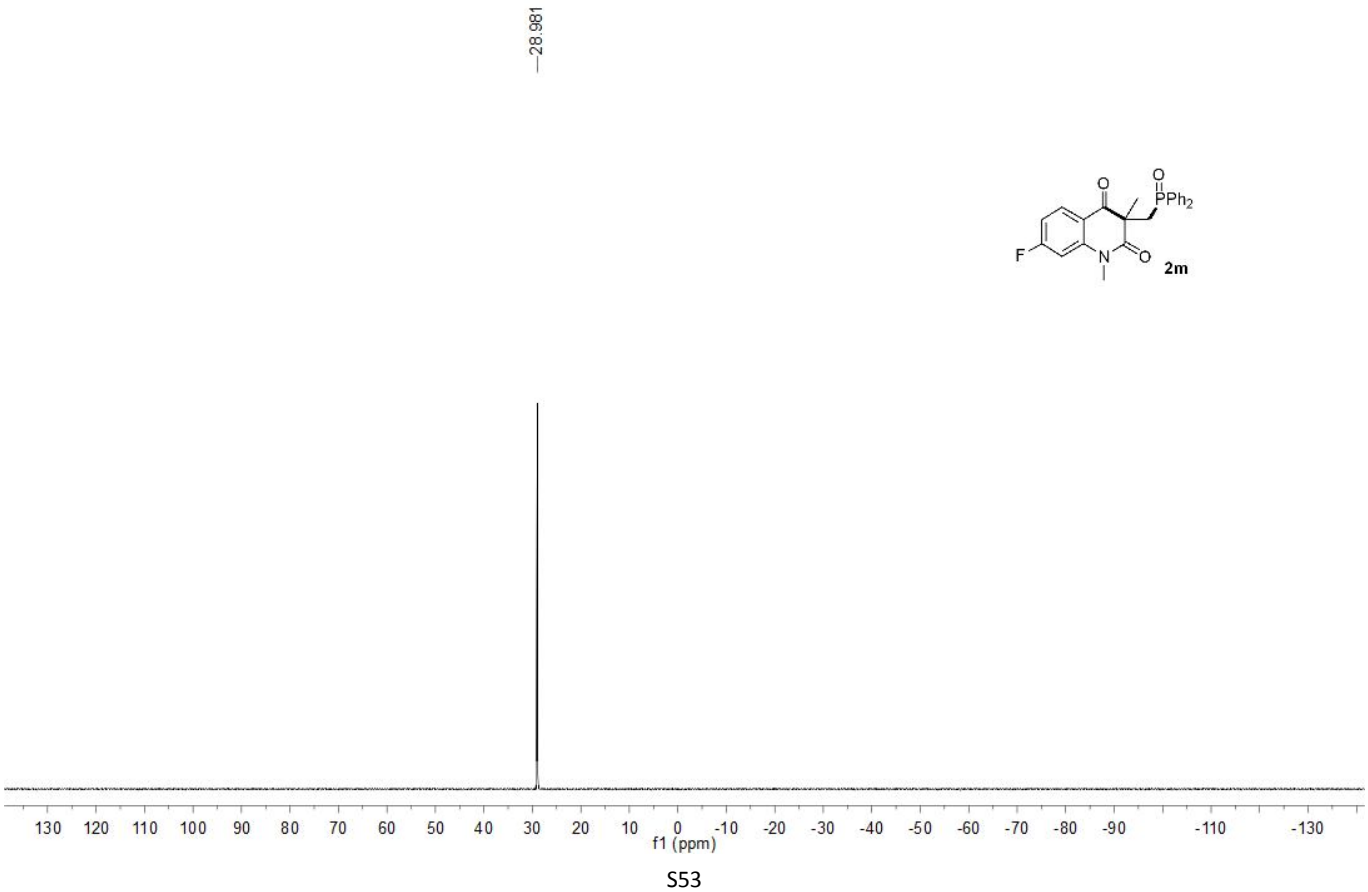
130.82
130.72
130.63
128.32
116.09
110.12
109.90
102.46
102.19
54.68
54.64
36.59
35.90
30.04
27.51
27.38
134.73
134.70
133.73
133.70
131.39
131.36
131.27
131.16
130.82
130.72
130.63
128.32
128.20
F
O
PPh2
N
2m
f1 (ppm)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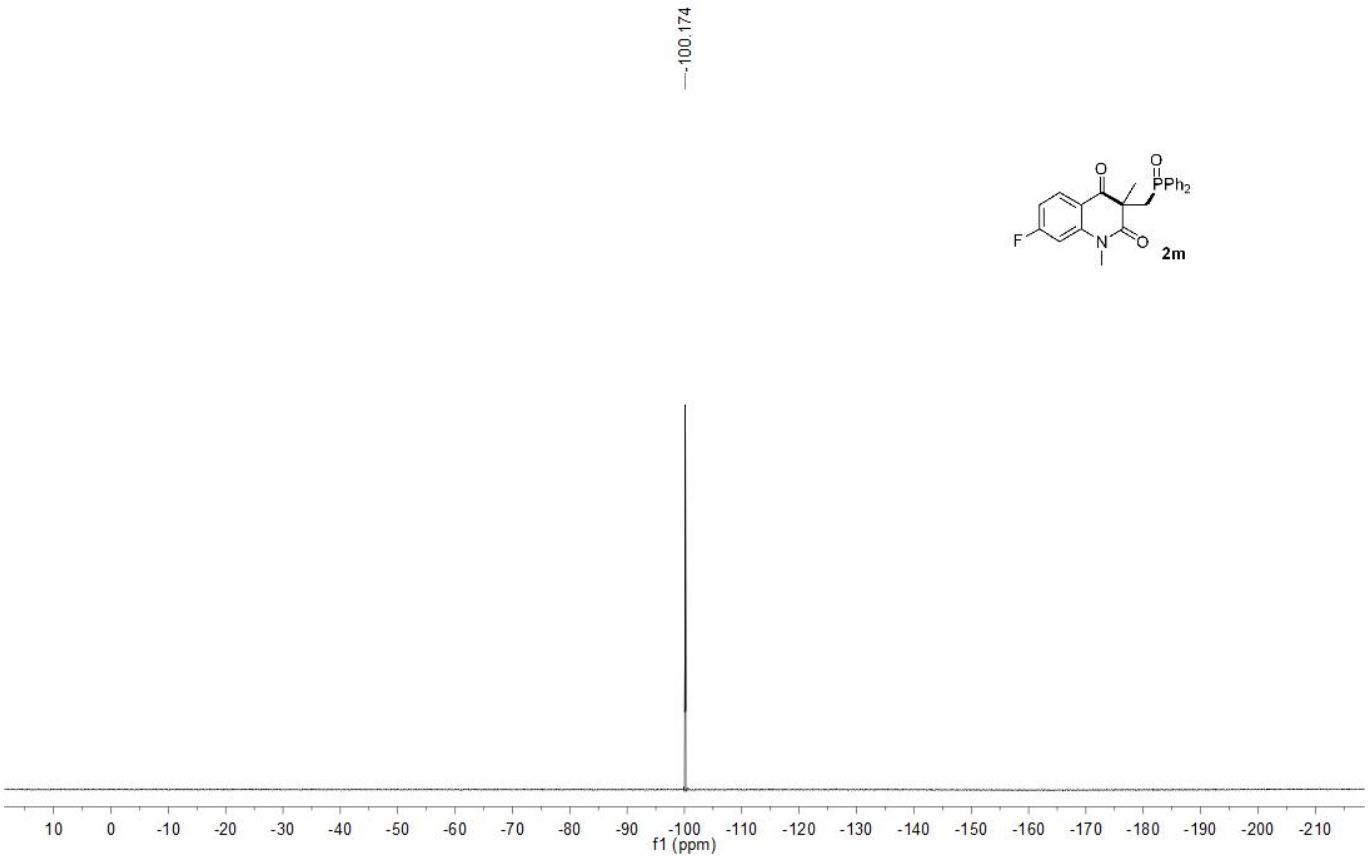
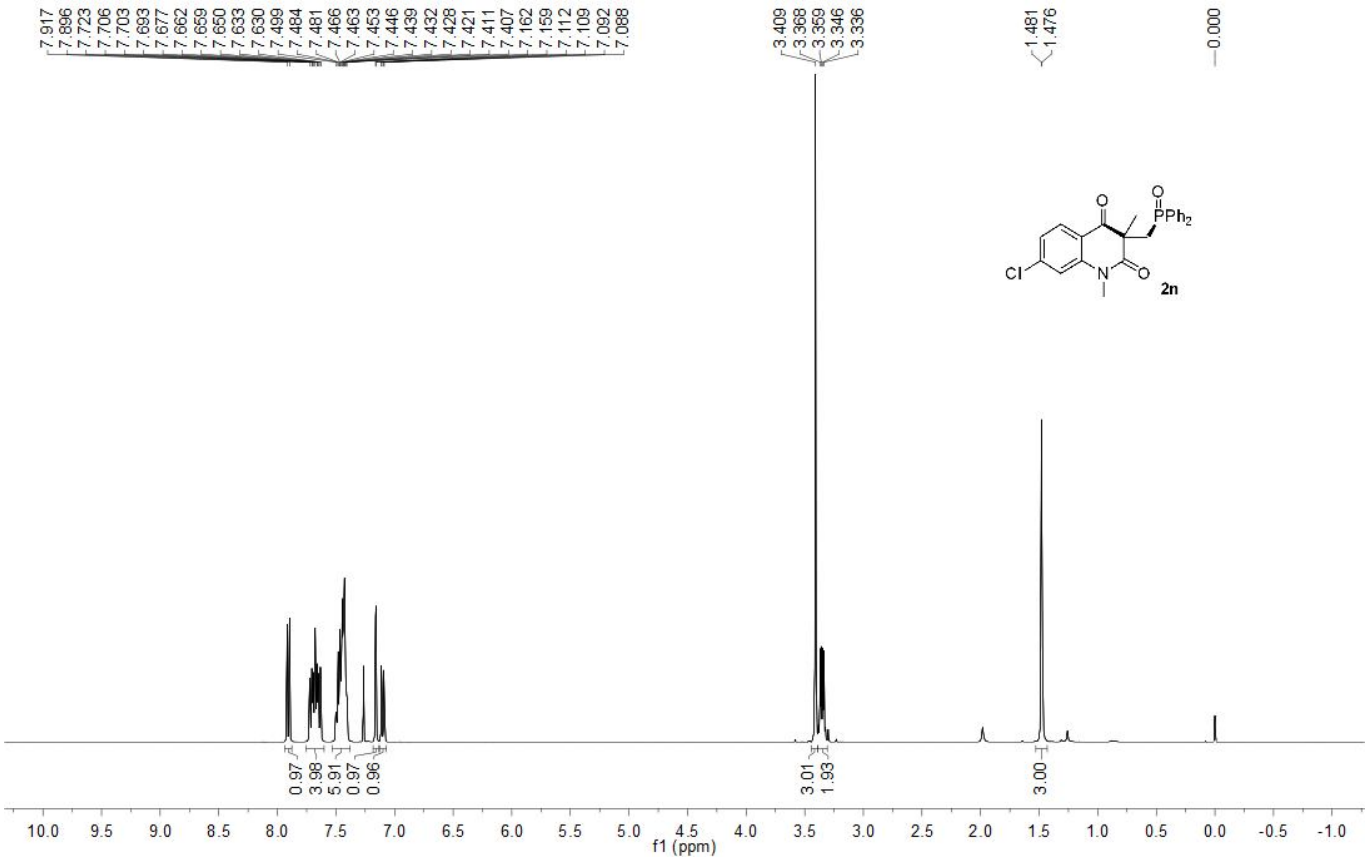
O
PPh2
N
2m
f1 (ppm)


-100.174
2m
10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210
f1 (ppm)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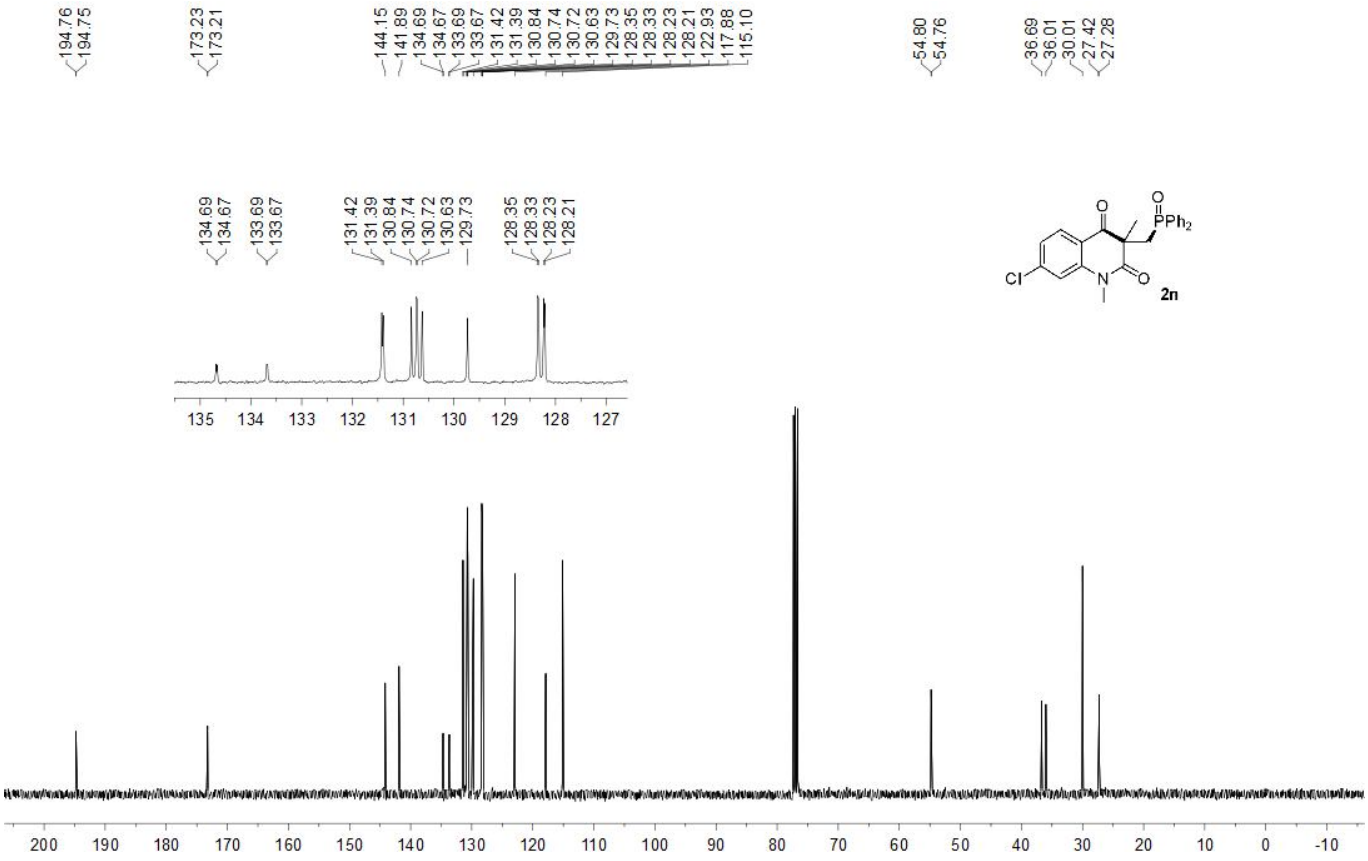
7.630
7.499
7.484
7.481
7.466
7.463
7.453
7.446
7.439
7.432
7.428
7.421
7.411
7.407
7.162
7.159
7.112
7.109
7.092
7.088
3.409
3.368
3.359
3.346
3.336
1.481
1.476
0.000
2n
0.97
3.98
5.91
0.97
0.96
3.01
1.93
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)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173.21
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141.89
134.69
134.67
133.69
133.67
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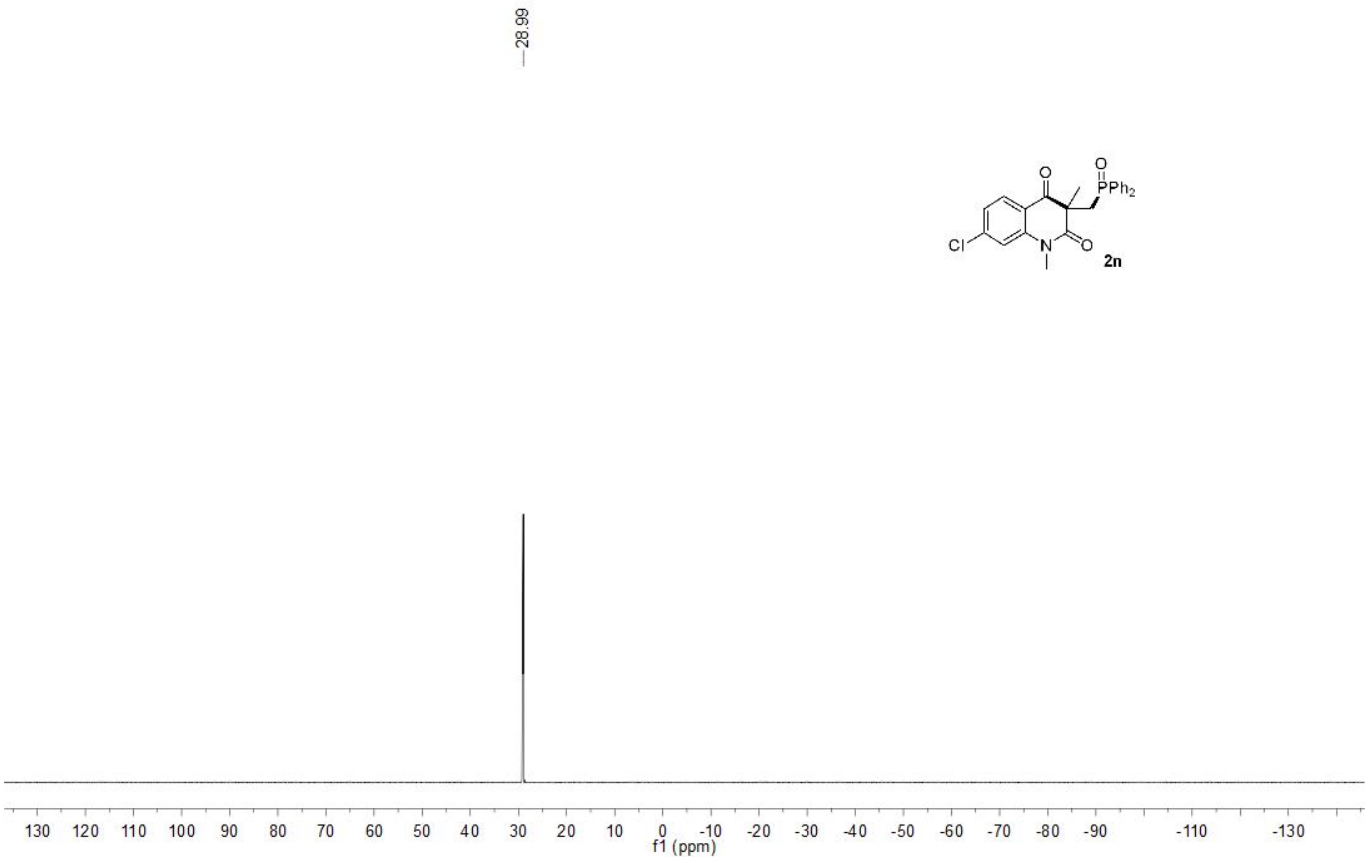
130.84
130.74
130.72
130.63
129.73
128.35
128.33
128.23
128.21
122.93
117.88
115.10
54.80
54.76
36.69
36.01
30.01
27.42
27.28
O
PPh2
Cl
N
O
2n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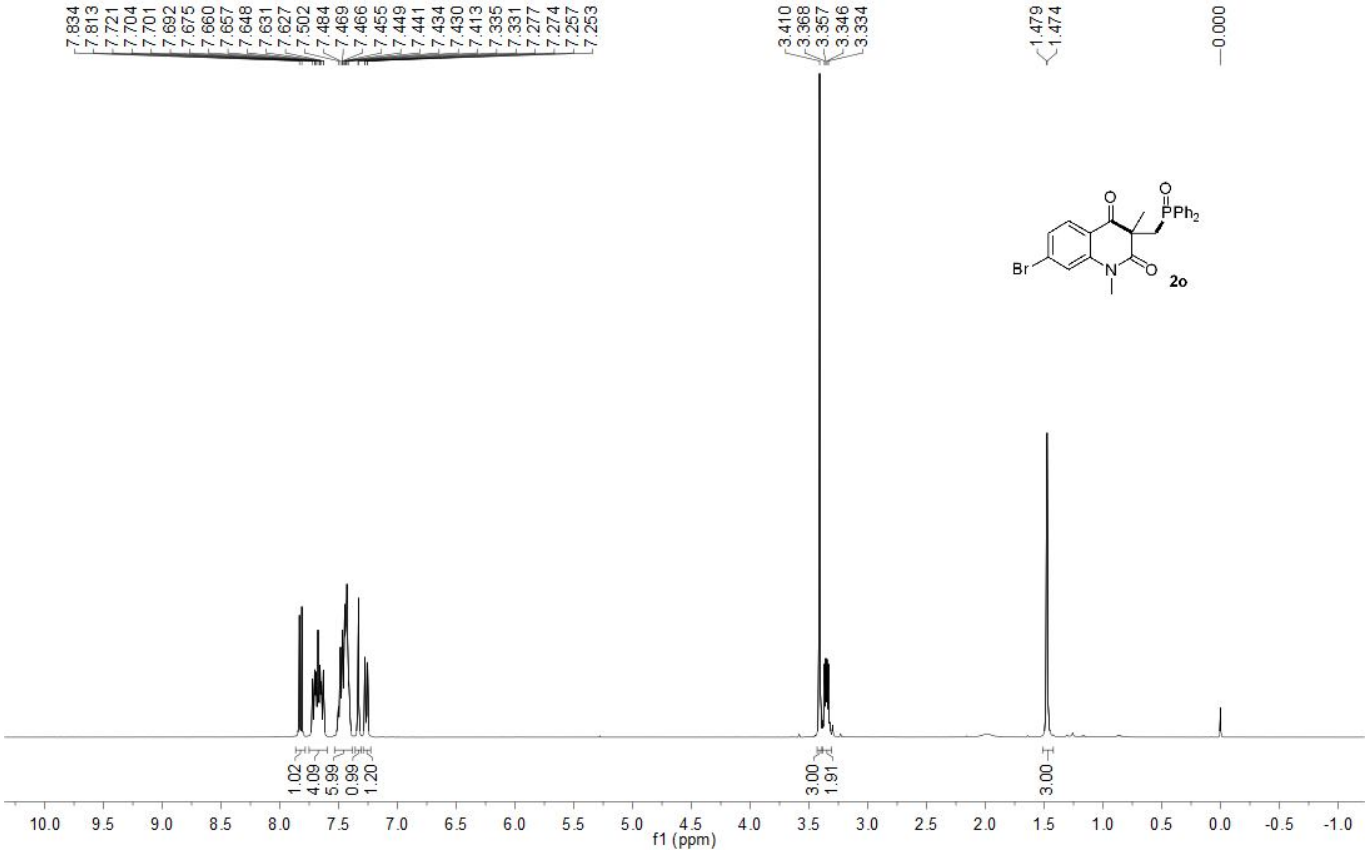
O
PPh2
Cl
N
O
2n
f1 (ppm)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7.627
7.502
7.484
7.469
7.466
7.455
7.449
7.441
7.434
7.430
7.413
7.335
7.331
7.277
7.274
7.257
7.253
3.410
3.368
3.357
3.346
3.334
1.479
1.474
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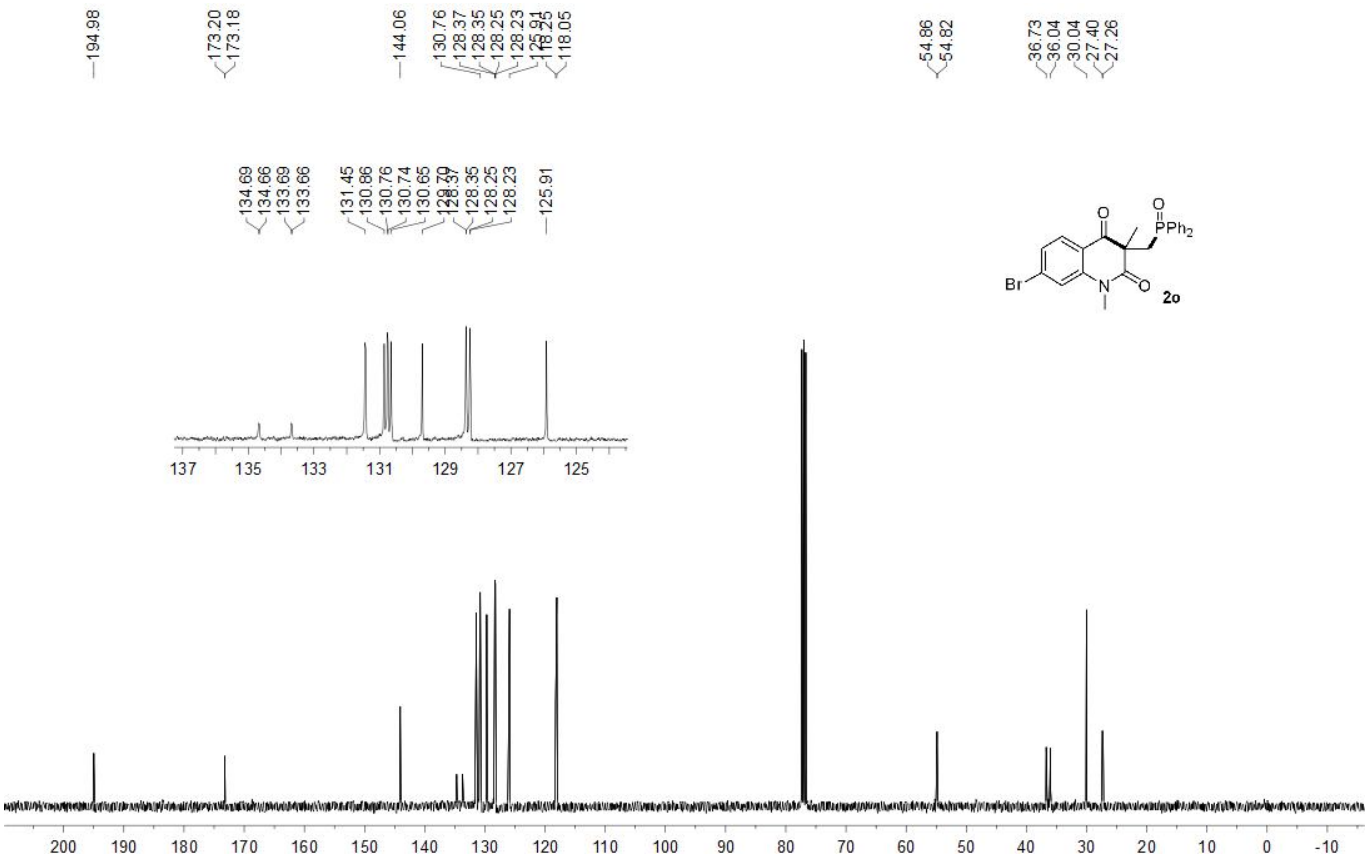
Br
2o
1.02
4.09
5.99
0.99
1.20
3.00
1.91
3.00
f1 (ppm)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118.05
54.86
54.82
36.73
36.04
30.04
27.40
27.26
134.69
134.66
133.69
133.66
131.45
130.86
130.76
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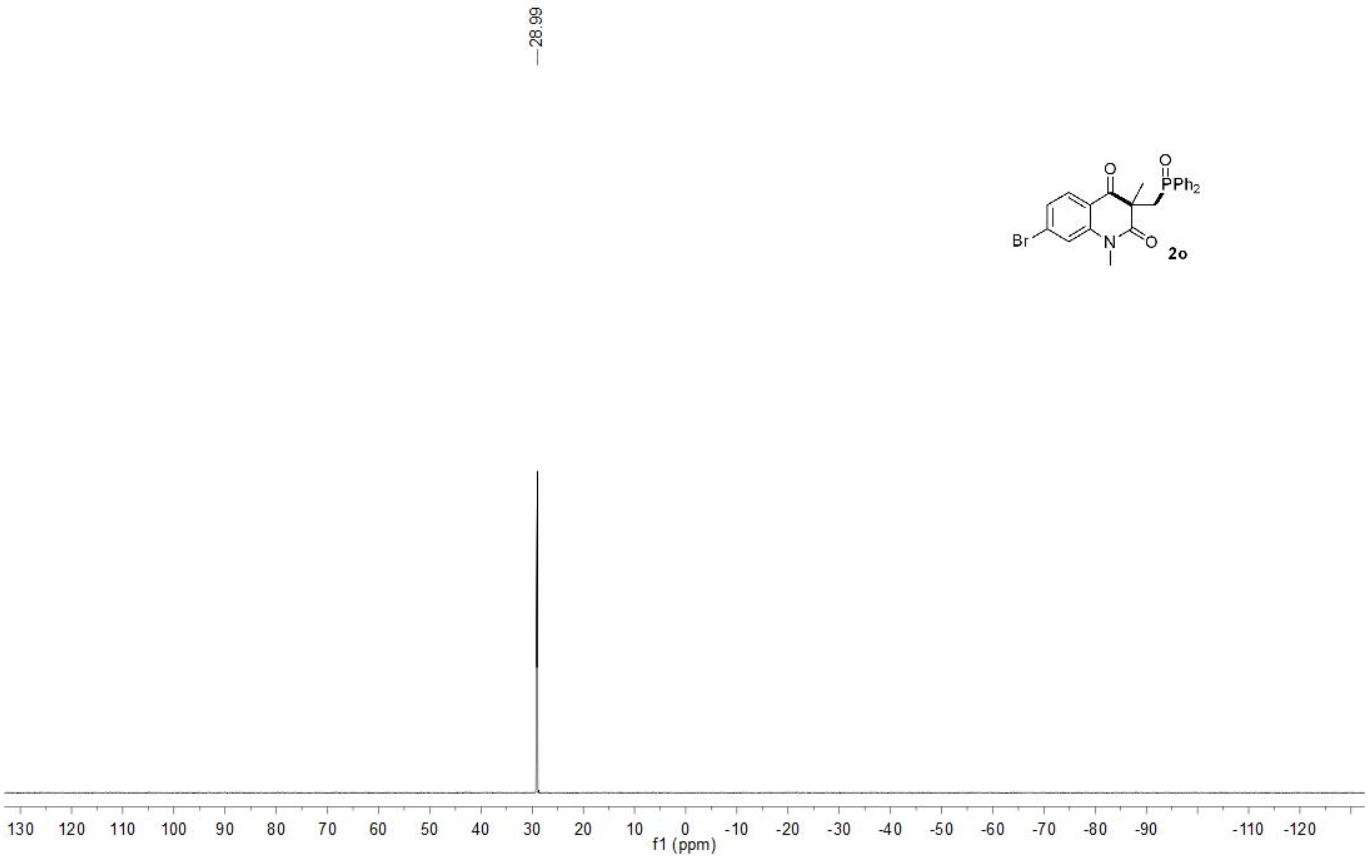
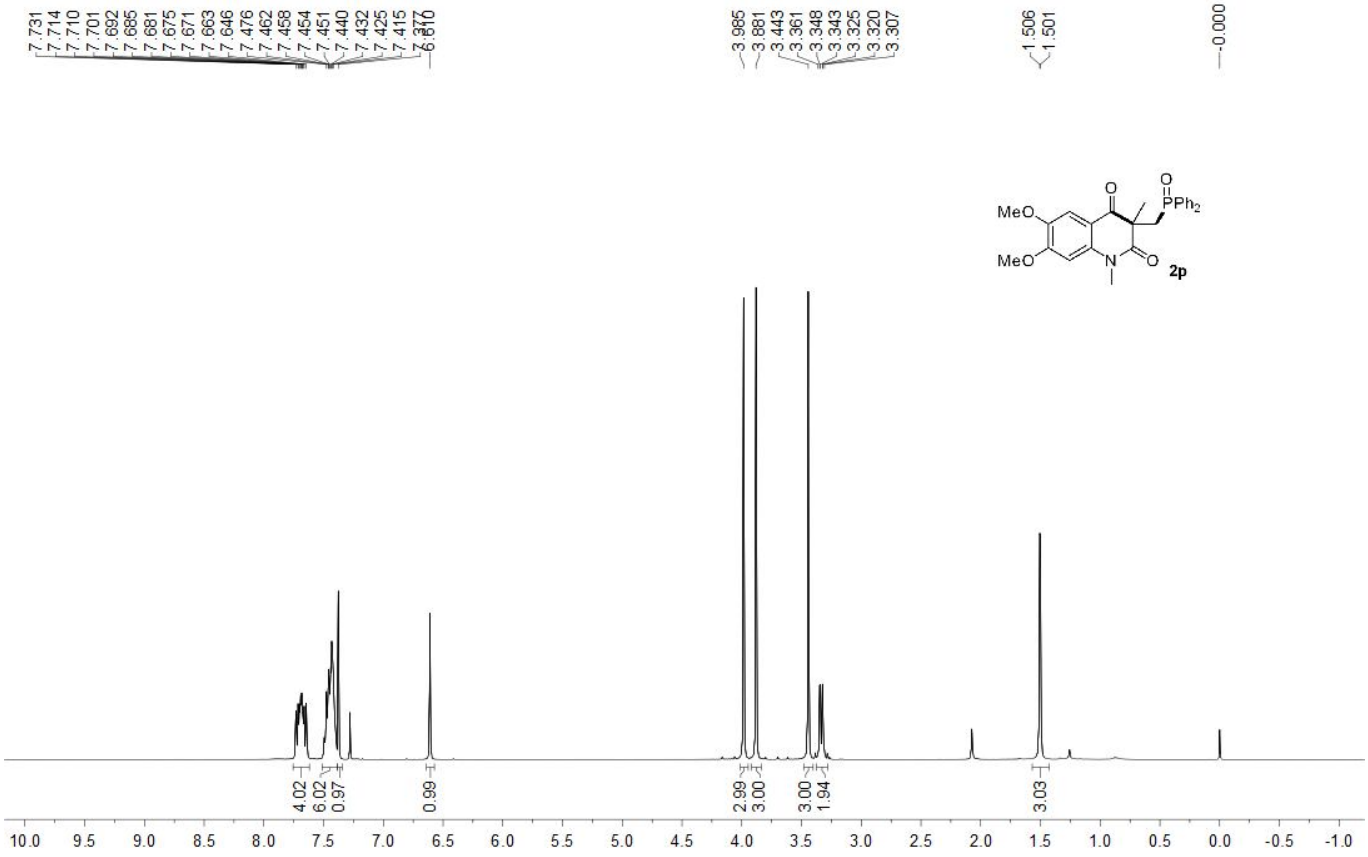
128.25
128.23
125.91
Br
2o
137
135
133
131
129
127
125


28.99
Br
2o
130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -110 -120
f1 (ppm)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7.681
7.675
7.671
7.663
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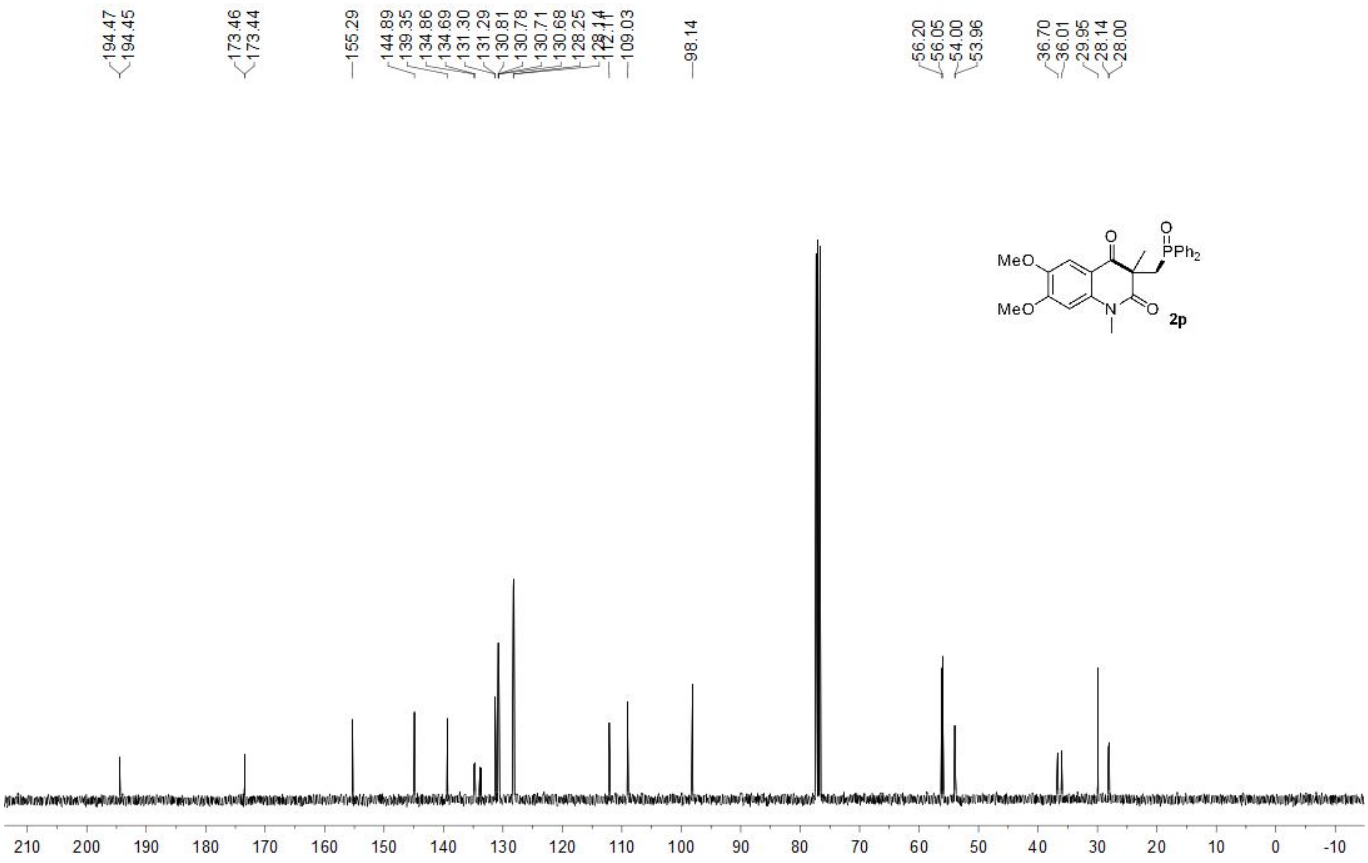
7.462
7.458
7.454
7.451
7.440
7.432
7.425
7.415
7.377
6.810
3.985
3.881
3.443
3.361
3.348
3.343
3.325
3.320
3.307
1.506
1.501
0.000
MeO
MeO
2p
4.02
6.02
0.97
0.99
2.99
3.00
3.00
1.94
3.03
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0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130.78
130.71
130.68
128.25
109.03
98.14
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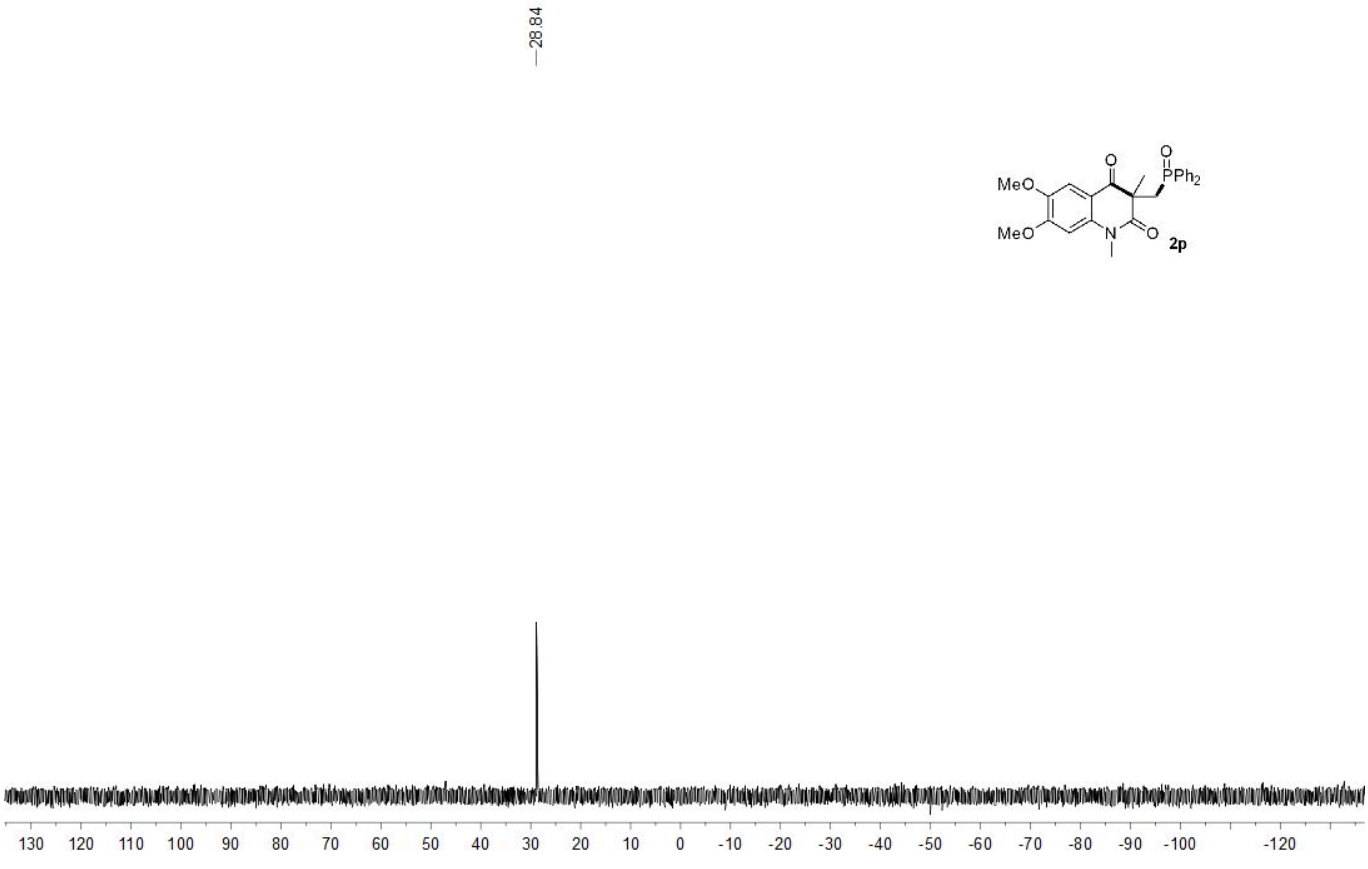
53.96
36.70
36.01
29.95
28.14
28.00
MeO
MeO
PPh2
2p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MeO
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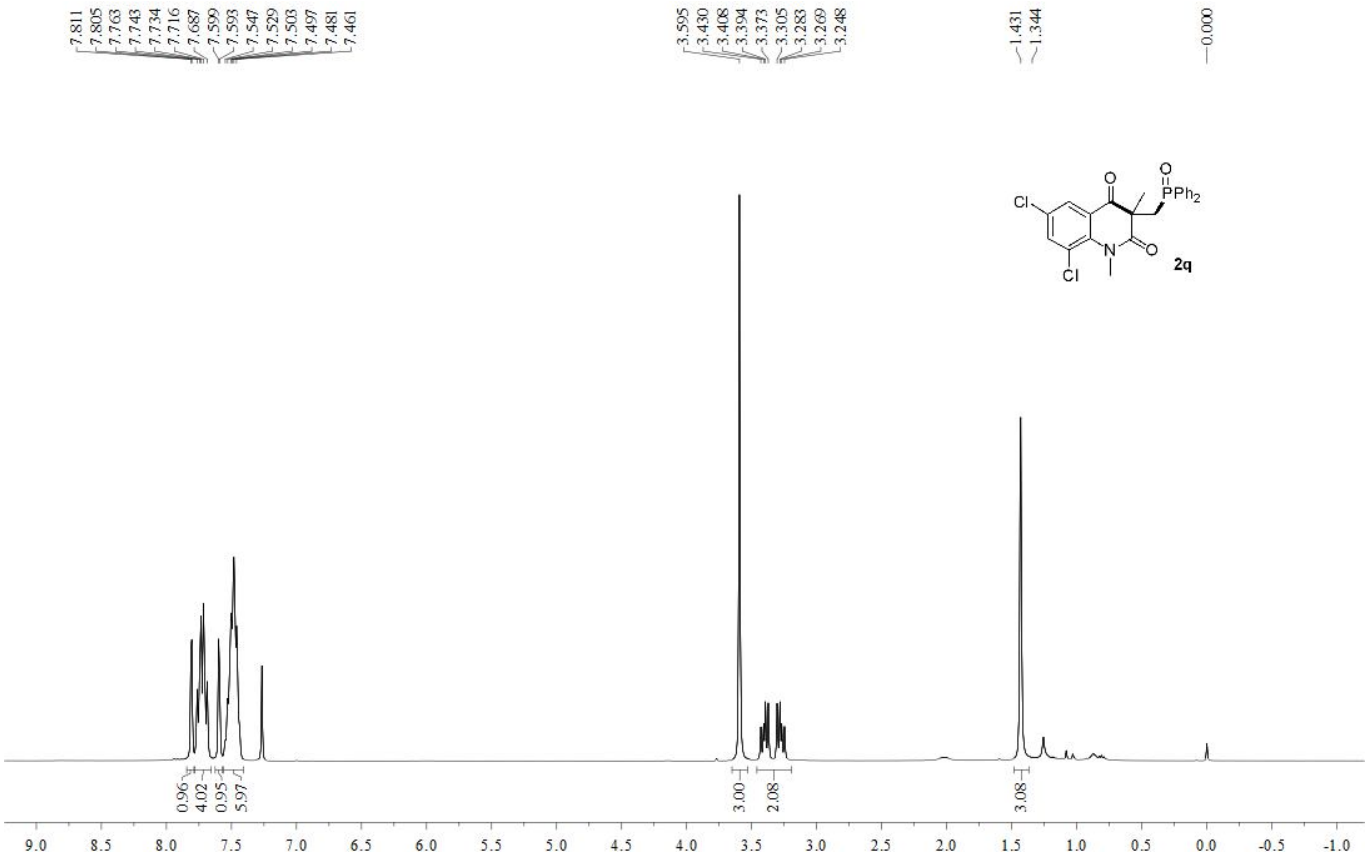
2p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7.497
7.481
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3.595
3.430
3.408
3.394
3.373
3.305
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3.248
1.431
1.344
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O
O
PPh2
N
O
Cl
2q
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4.02
0.95
5.97
3.00
2.08
3.08
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
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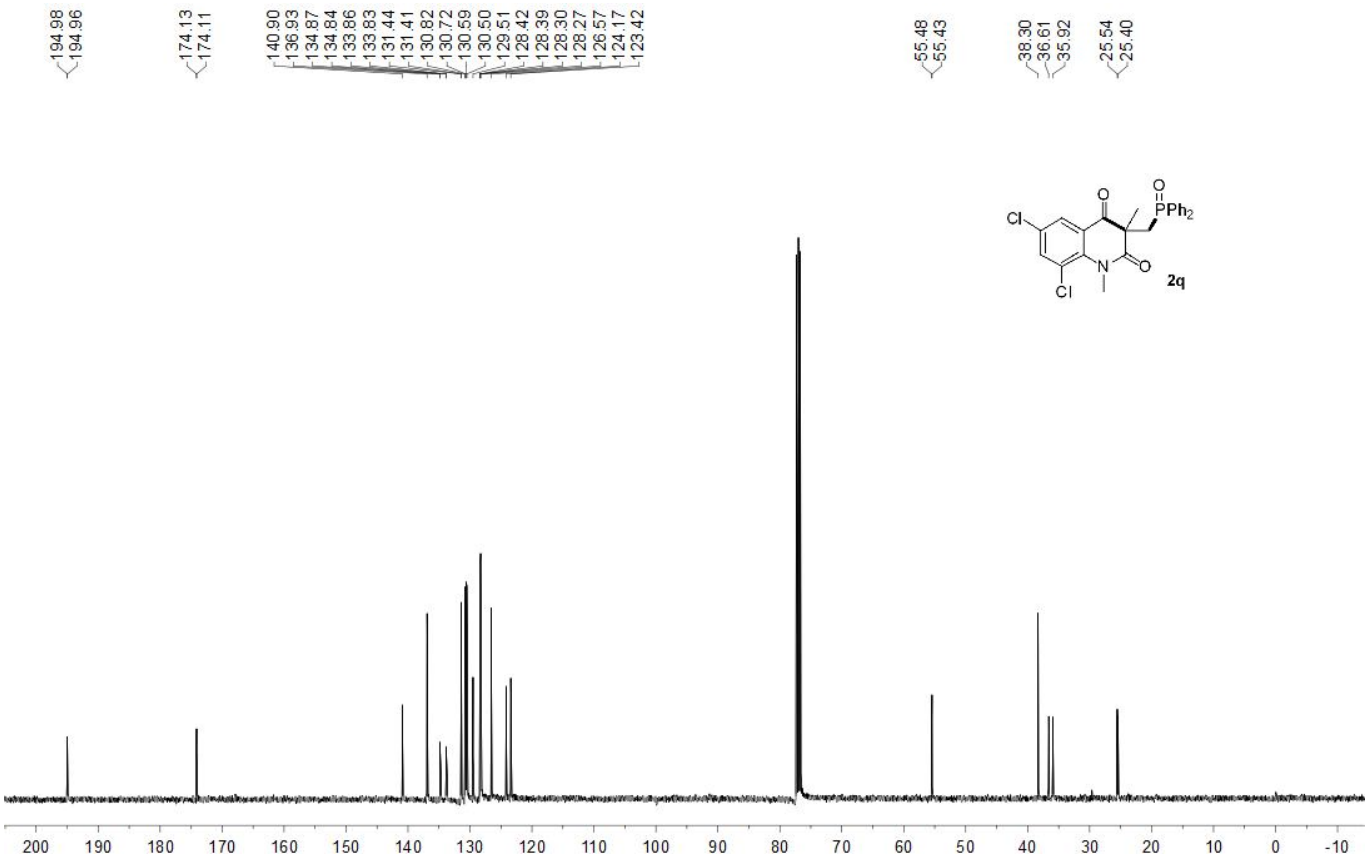
-1.0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128.39
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123.42
55.48
55.43
38.30
36.61
35.92
25.54
25.40
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O
O
PPh2
N
O
Cl
2q
200
190
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100
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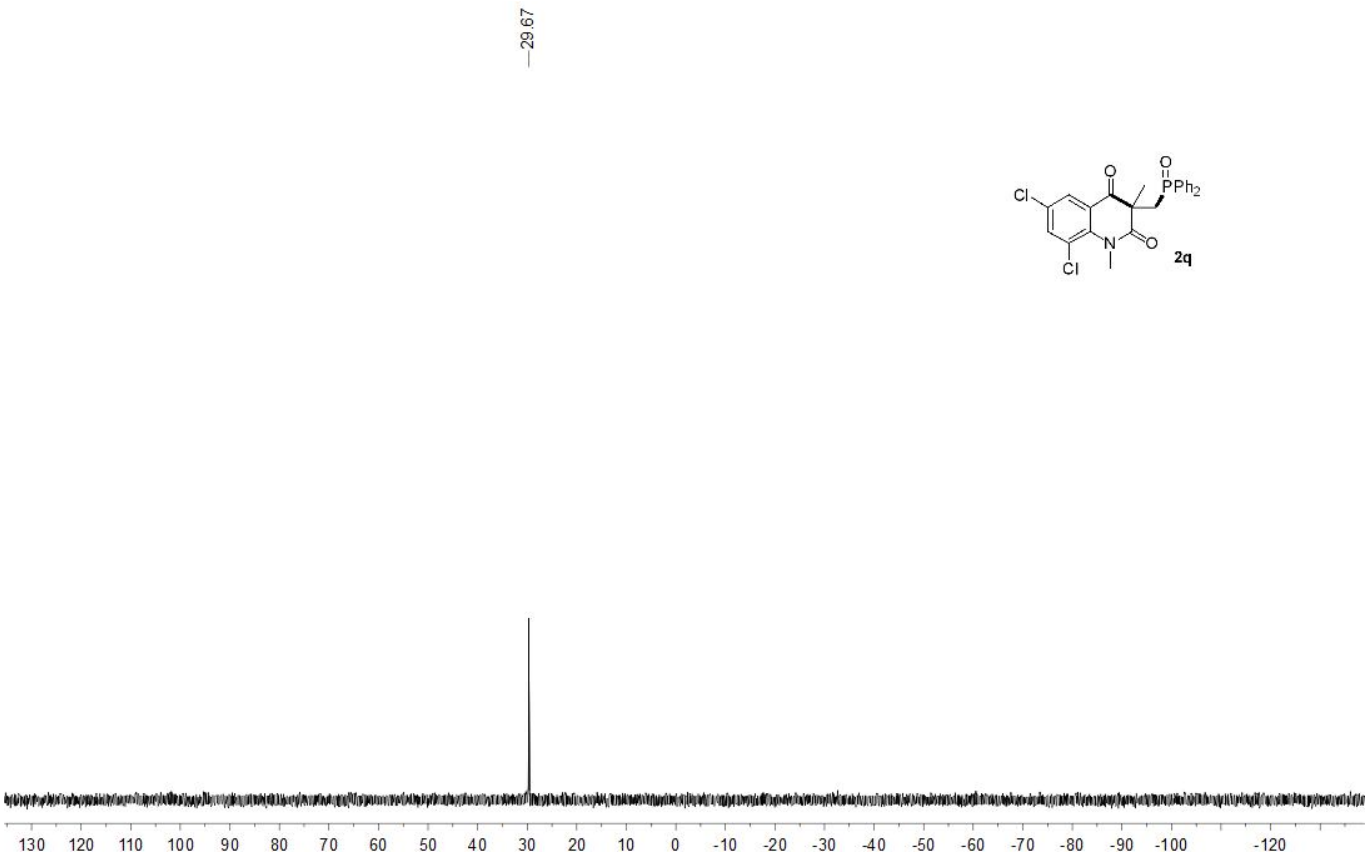
-10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PPh2
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O
Cl
2q
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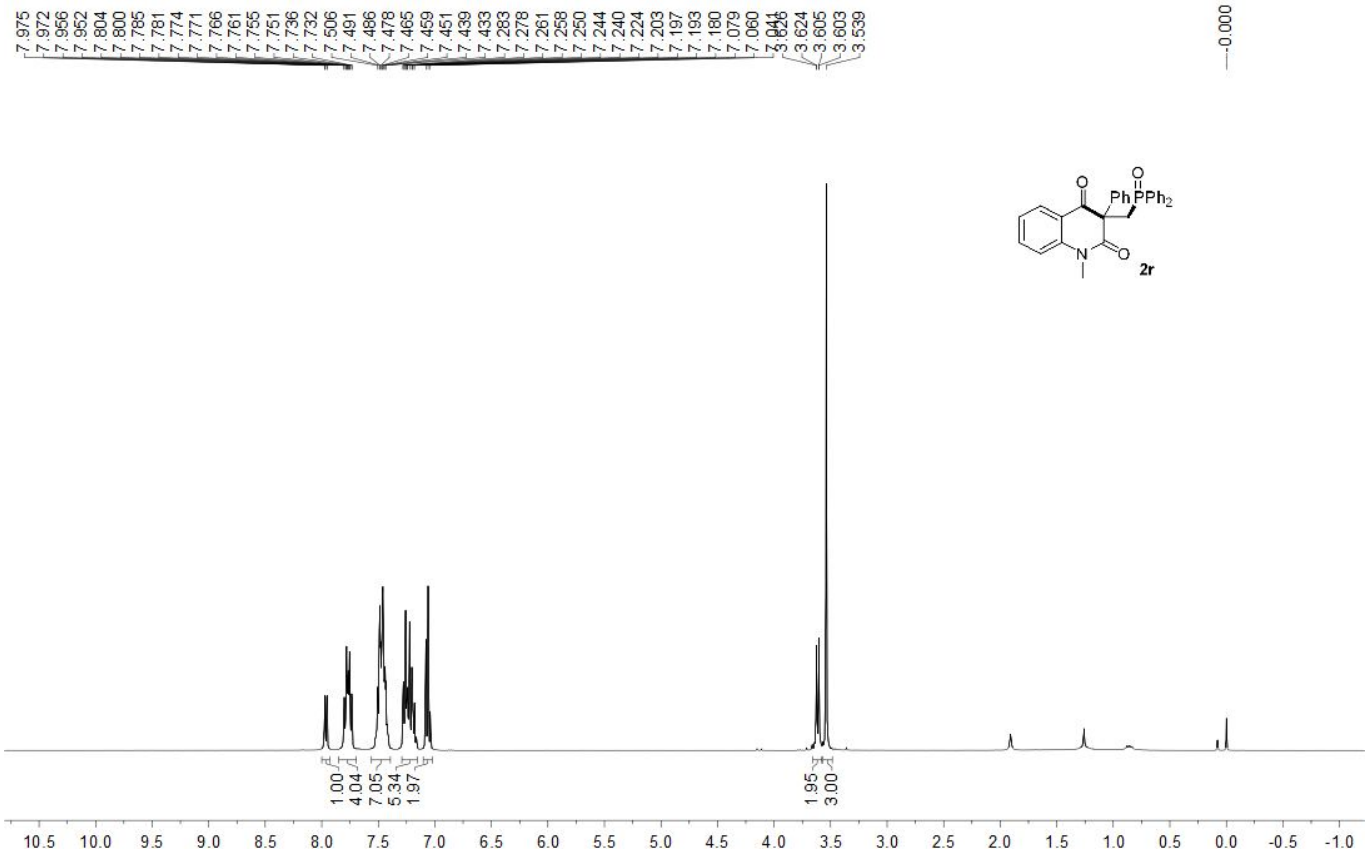
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7.755
7.751
7.736
7.732
7.506
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7.283
7.278
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7.258
7.250
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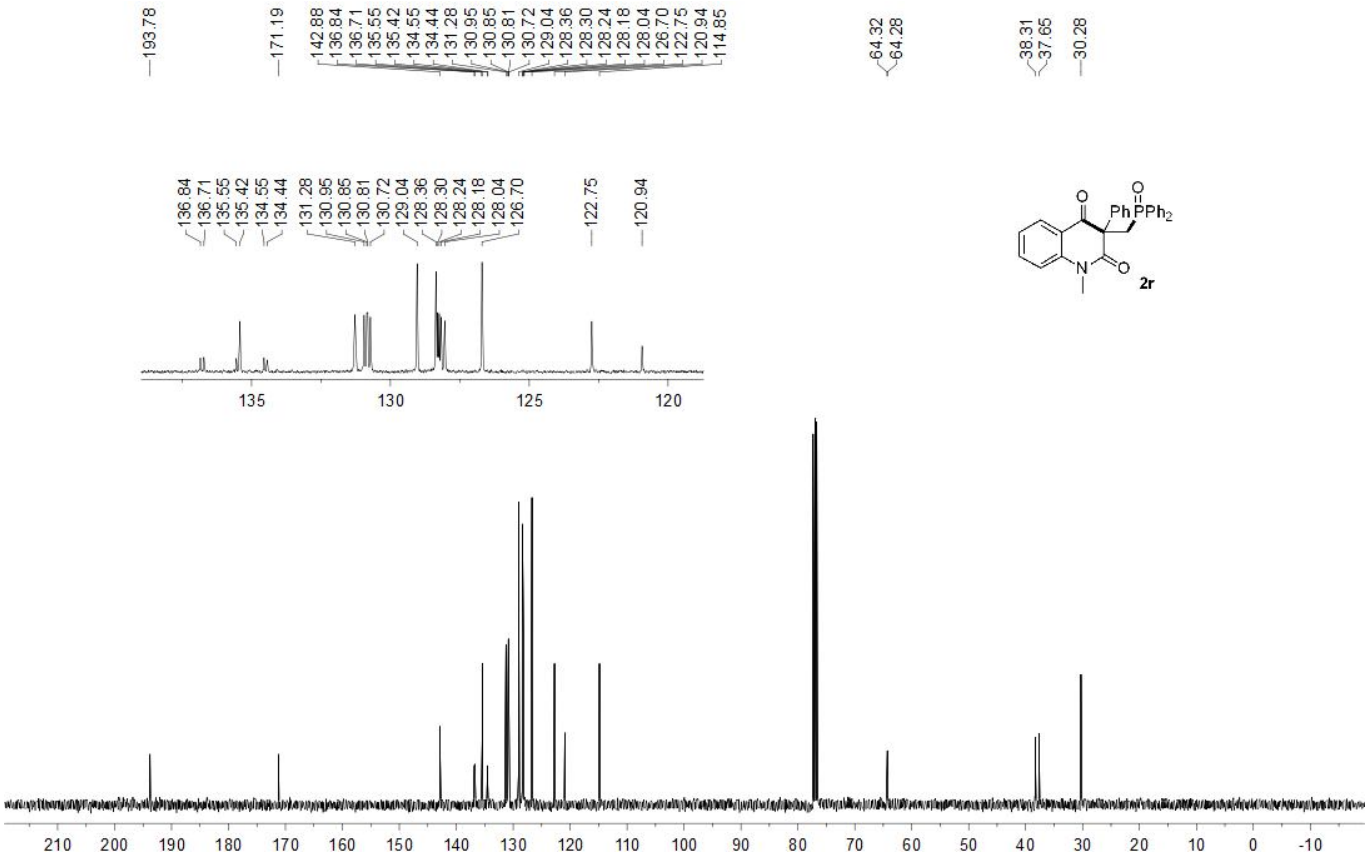
7.240
7.224
7.203
7.197
7.193
7.180
7.079
7.060
3.624
3.605
3.603
3.539
0.000
O
Ph
PPh2
N
O
2r
1.00
4.04
7.05
5.34
1.97
1.95
3.00
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0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130.81
130.72
129.04
128.36
128.30
128.24
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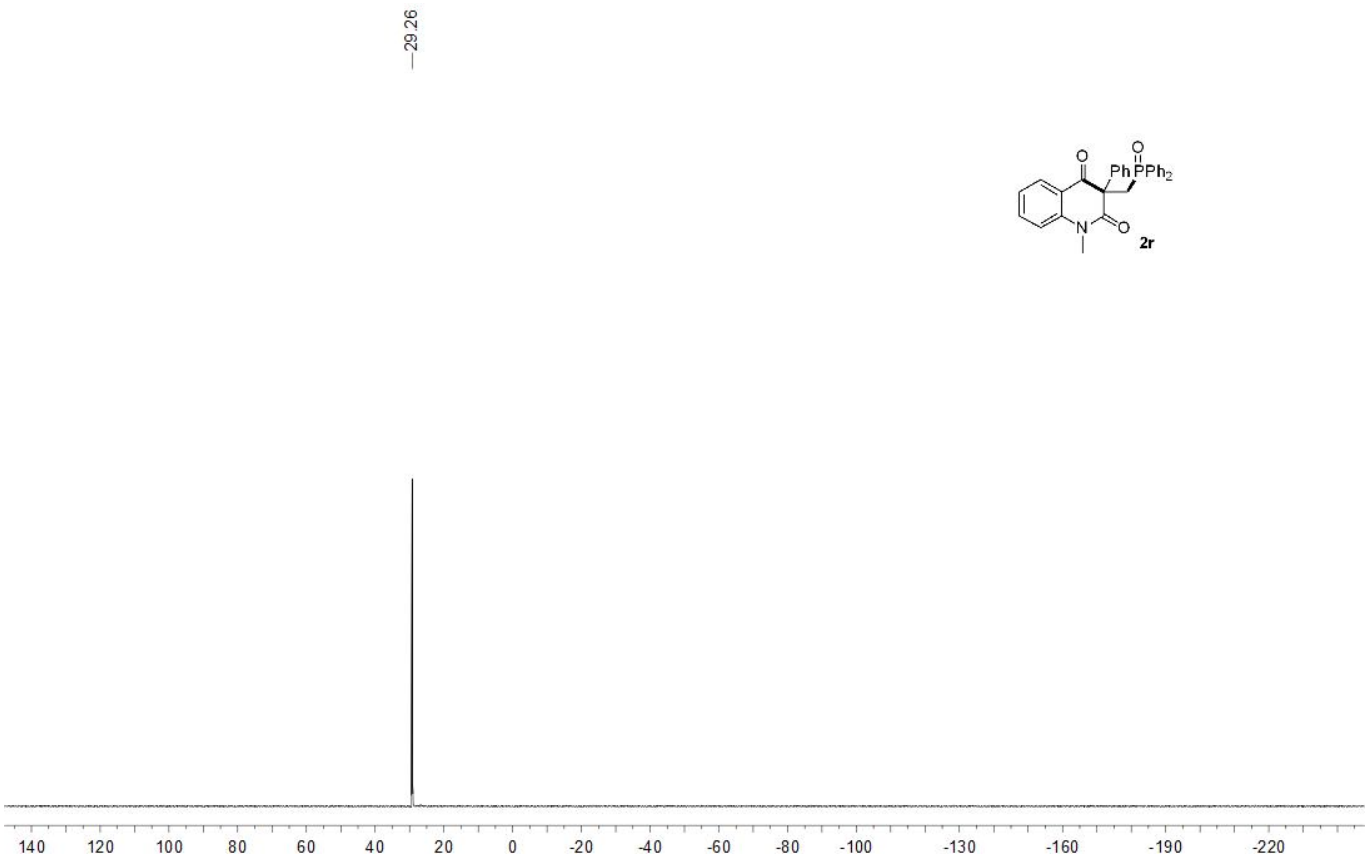
122.75
120.94
64.32
64.28
38.31
37.65
30.28
Ph
PPh2
2r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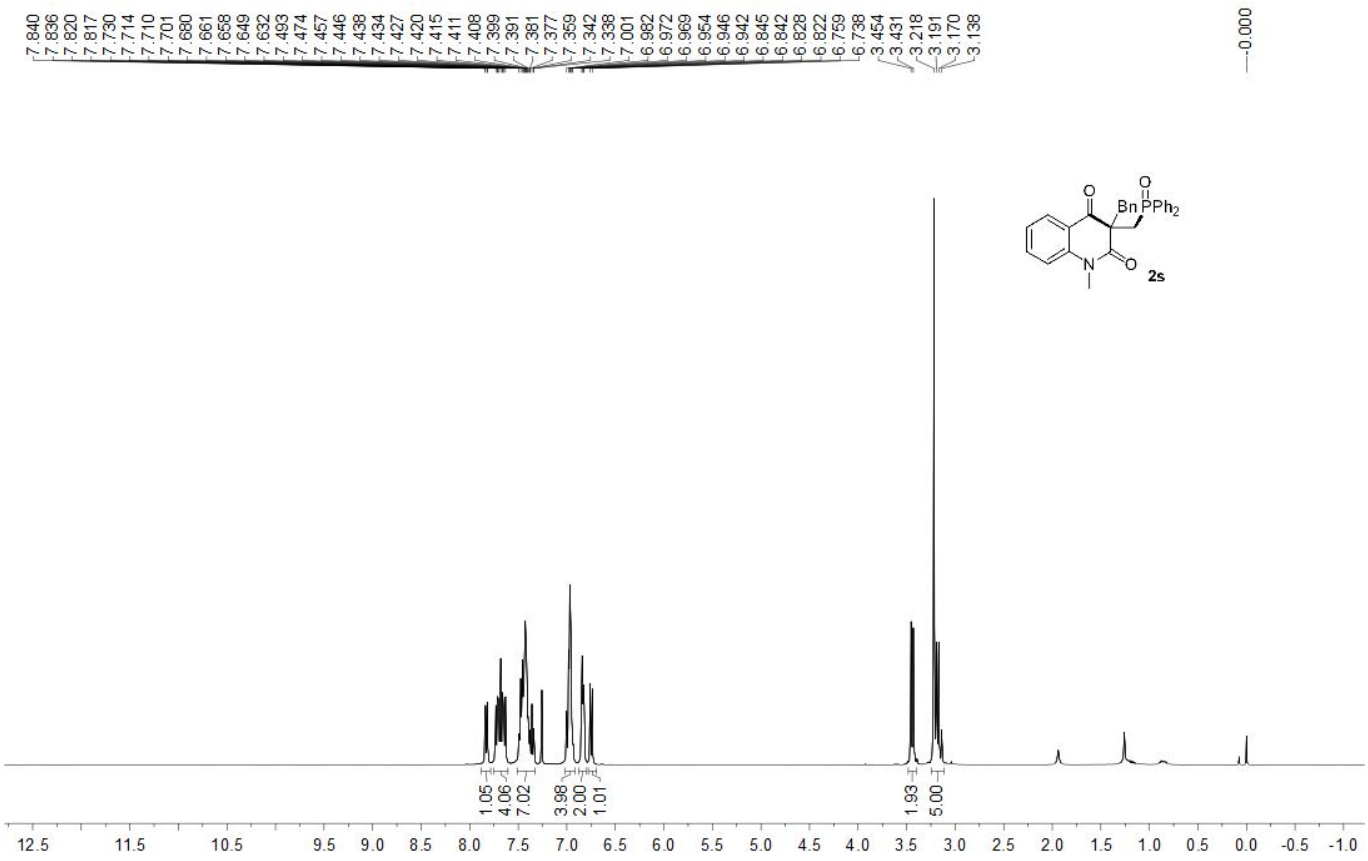
2r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6.845
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6.738
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3.431
3.218
3.191
3.170
3.138
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Bn
PPh2
2s
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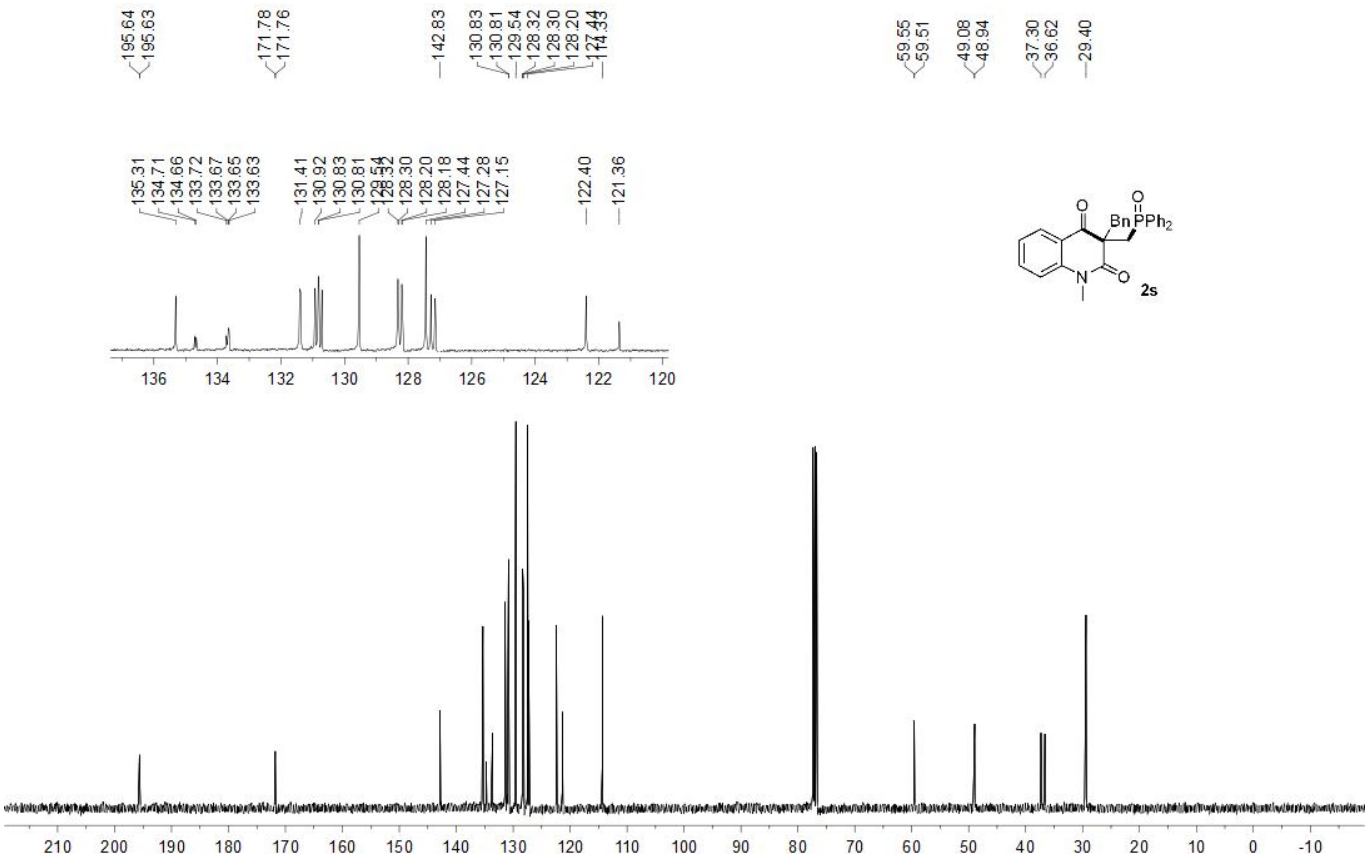
4.06
7.02
3.98
2.00
1.01
1.93
5.00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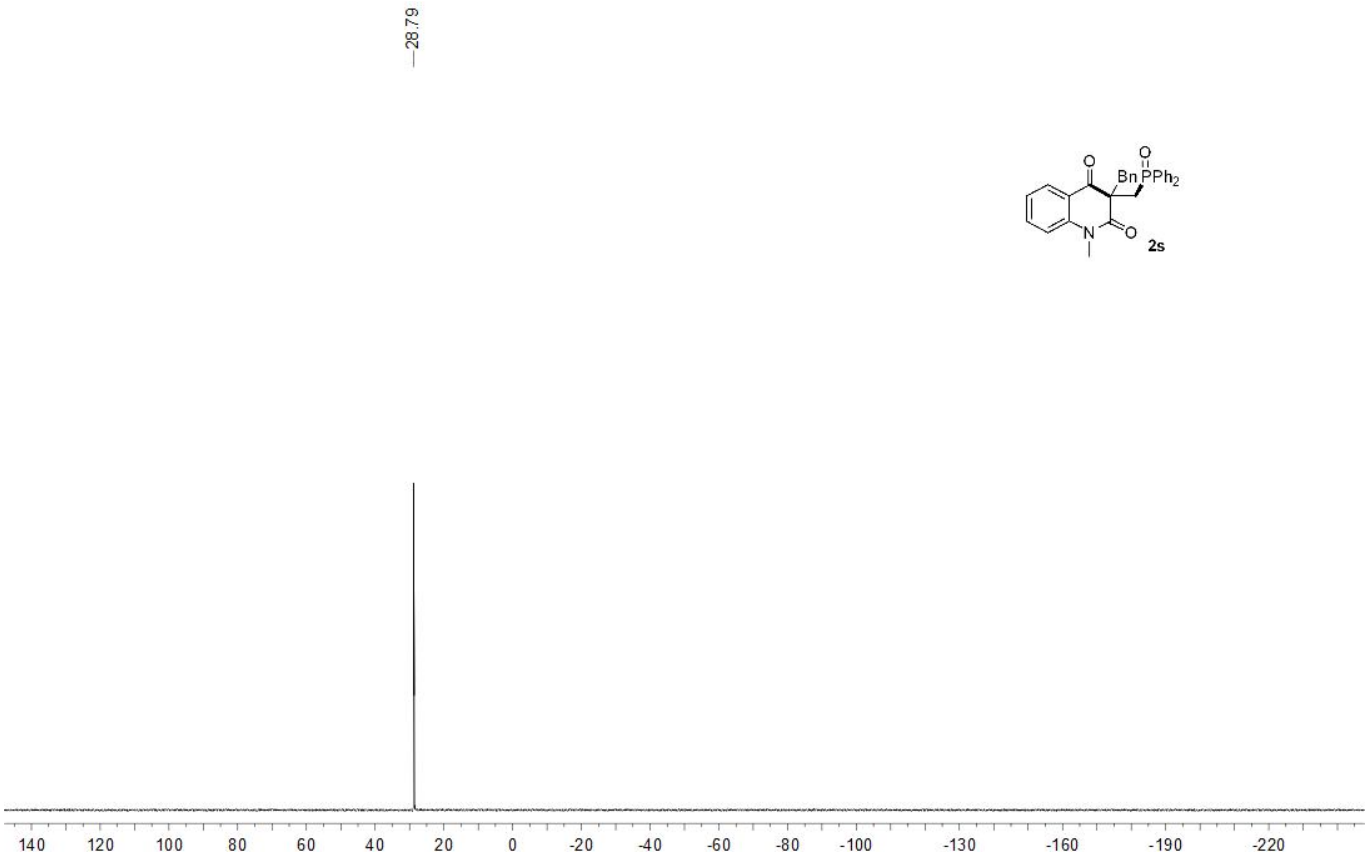
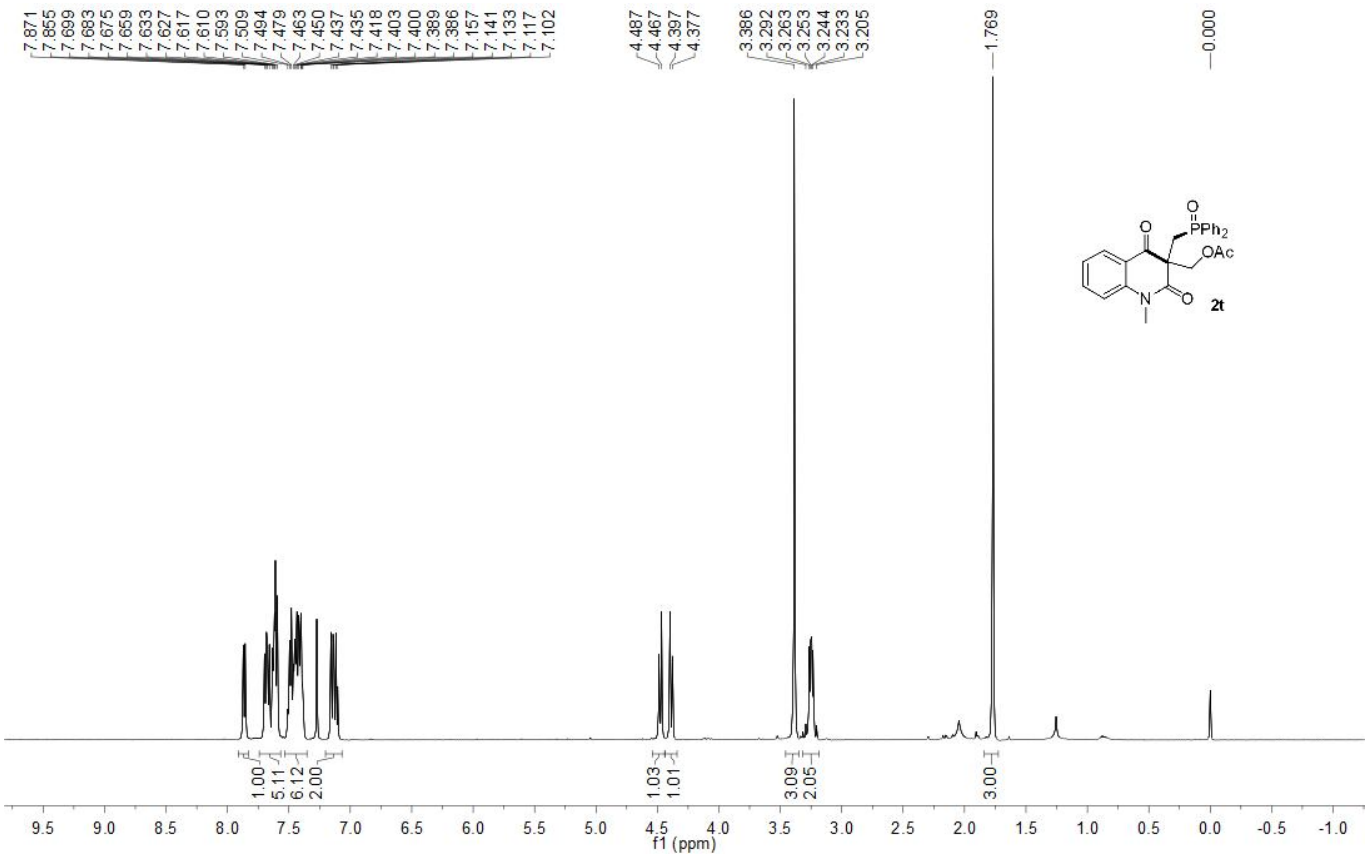
127.28
127.15
122.40
121.36
Bn
PPh2
2s


28.79
Bn
PPh2
2s
140
120
100
80
60
40
20
0
-20
-40
-60
-80
-100
-130
-160
-190
-220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7.855
7.699
7.683
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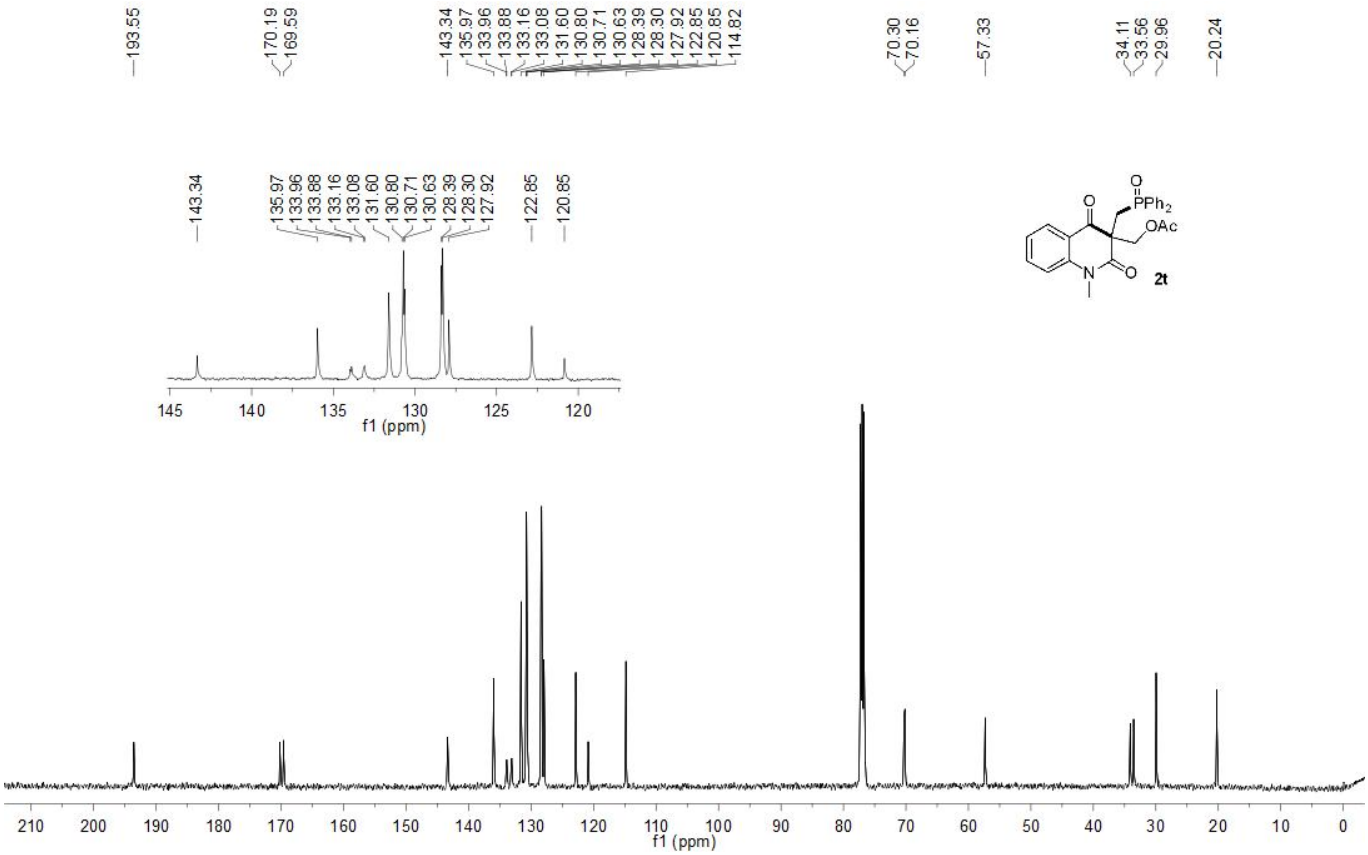
7.659
7.633
7.627
7.617
7.610
7.593
7.509
7.494
7.479
7.463
7.450
7.437
7.435
7.418
7.403
7.400
7.389
7.386
7.157
7.141
7.133
7.117
7.102
4.487
4.467
4.397
4.377
3.386
3.292
3.263
3.253
3.244
3.233
3.205
1.769
0.000
PPh2
OAc
2t
1.00
5.11
6.12
2.00
1.03
1.01
3.09
2.05
3.00
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
-1.0
f1 (ppm)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170.19
169.59
143.34
135.97
133.96
133.88
133.16
133.08
131.60
130.80
130.71
130.63
128.39
128.30
127.92
122.85
120.85
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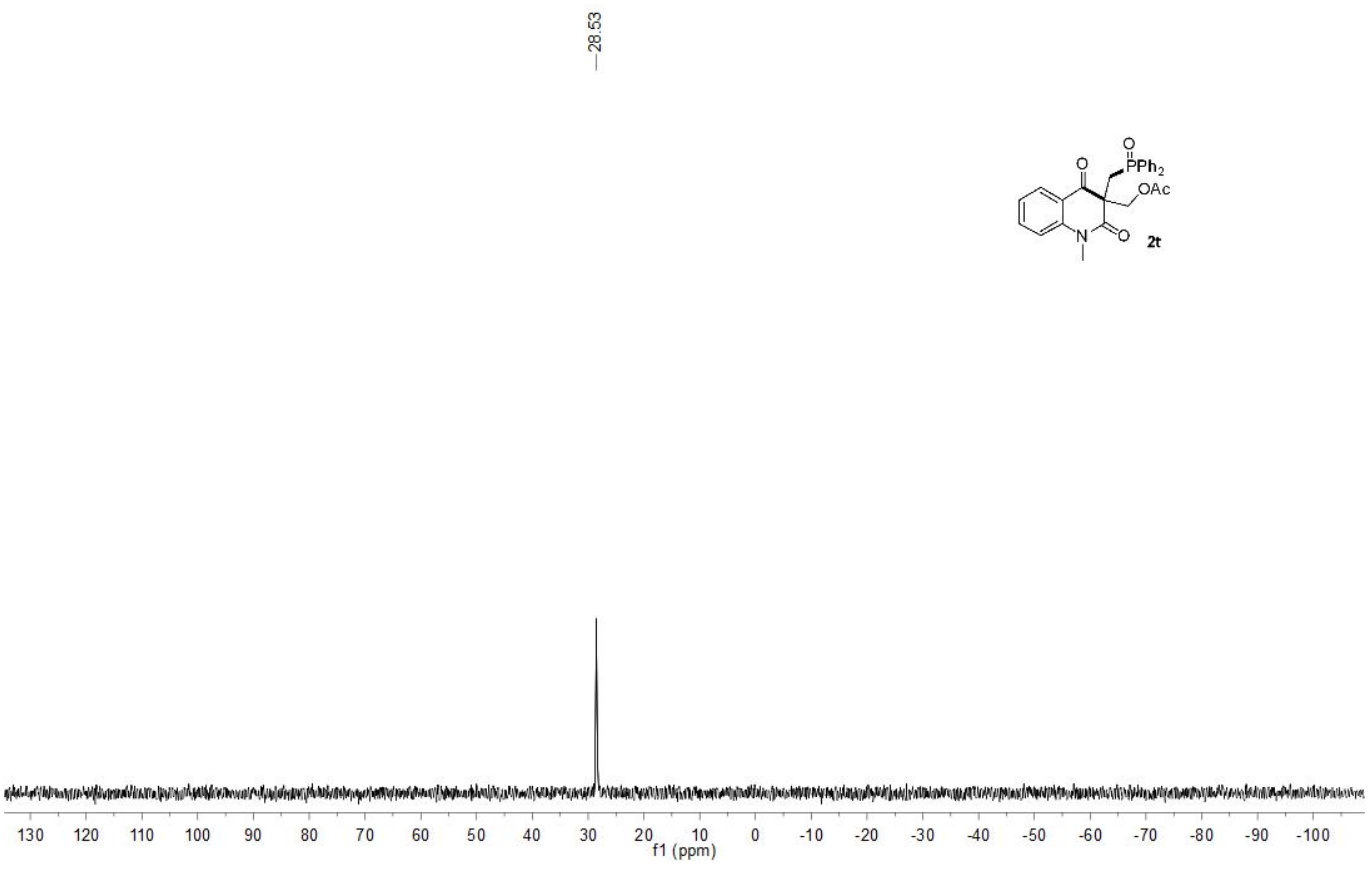
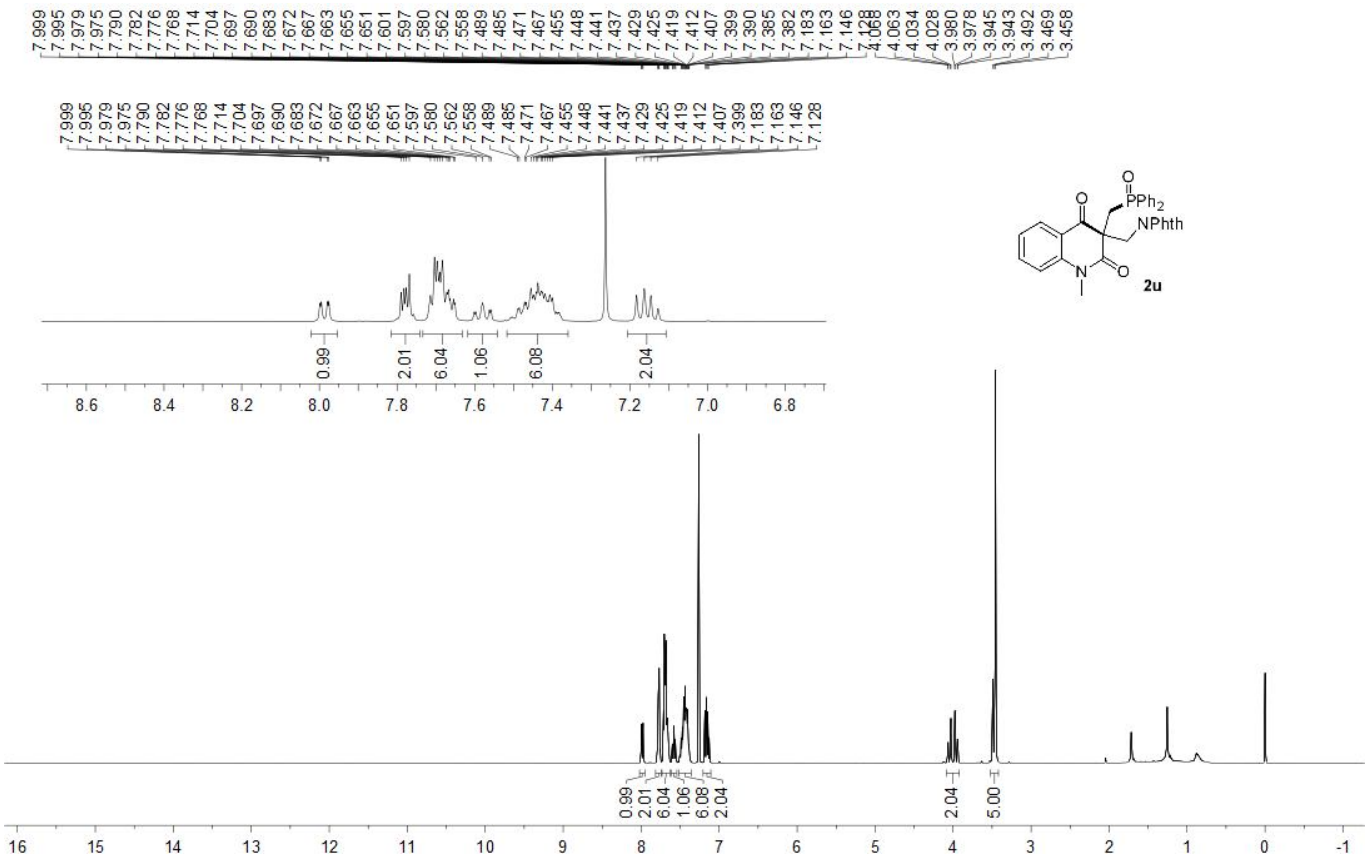
70.30
70.16
57.33
34.11
33.56
29.96
20.24
f1 (ppm)
2t


28.53
f1 (ppm)
2t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7.975
7.790
7.782
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7.768
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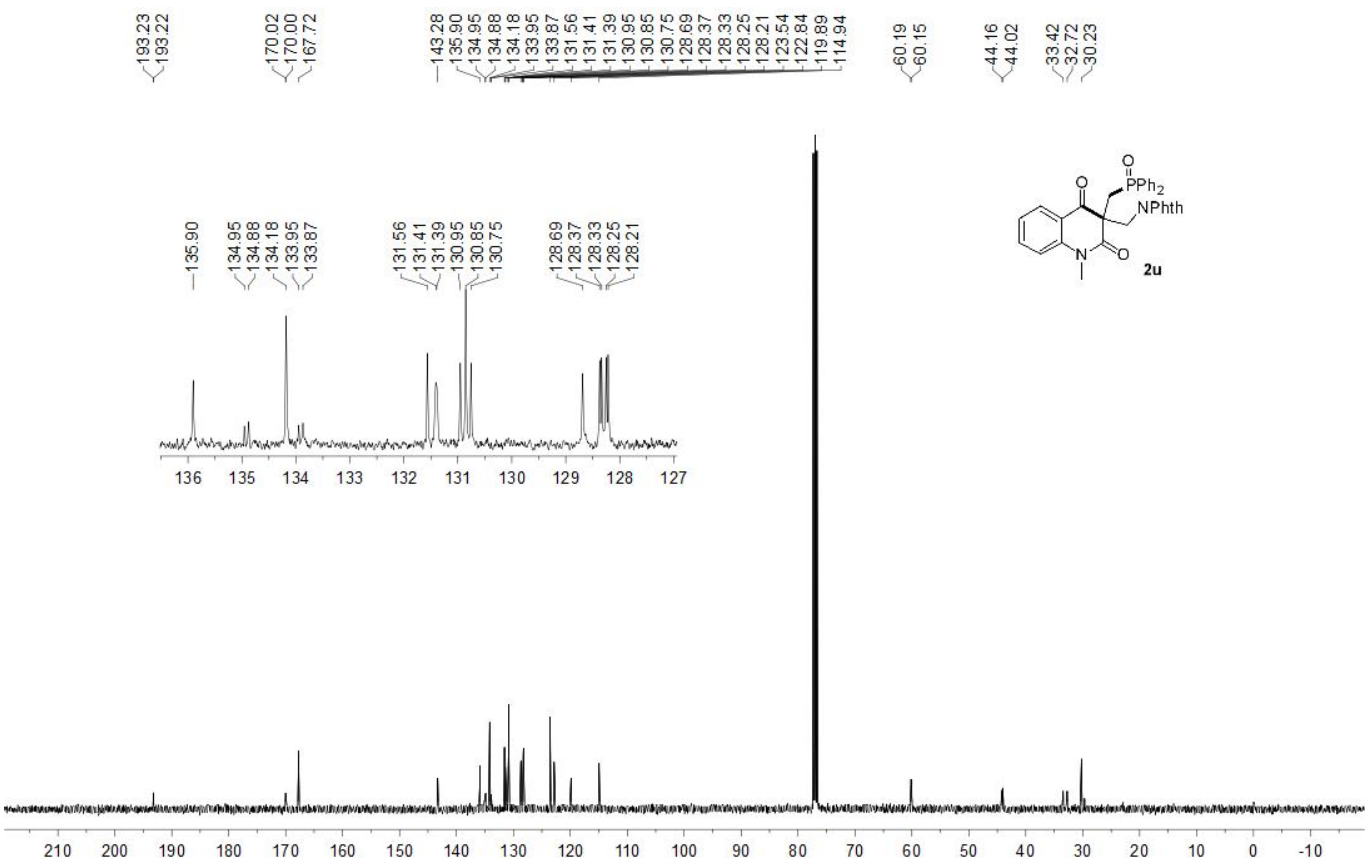
7.704
7.697
7.690
7.683
7.672
7.667
7.663
7.655
7.651
7.601
7.597
7.580
7.562
7.558
7.489
7.485
7.471
7.467
7.455
7.448
7.441
7.437
7.429
7.425
7.419
7.412
7.407
7.399
7.390
7.385
7.382
7.183
7.163
7.146
7.128
4.068
4.063
4.034
4.028
3.980
3.978
3.945
3.943
3.492
3.469
3.458
0.99
2.01
6.04
1.06
6.08
2.04
2.04
5.00
PPh2
NPhth
2u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193.22
170.02
170.00
167.72
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135.90
134.95
134.88
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131.56
131.41
131.39
130.95
130.85
130.75
128.69
128.37
128.33
128.25
128.21
123.54
122.84
119.89
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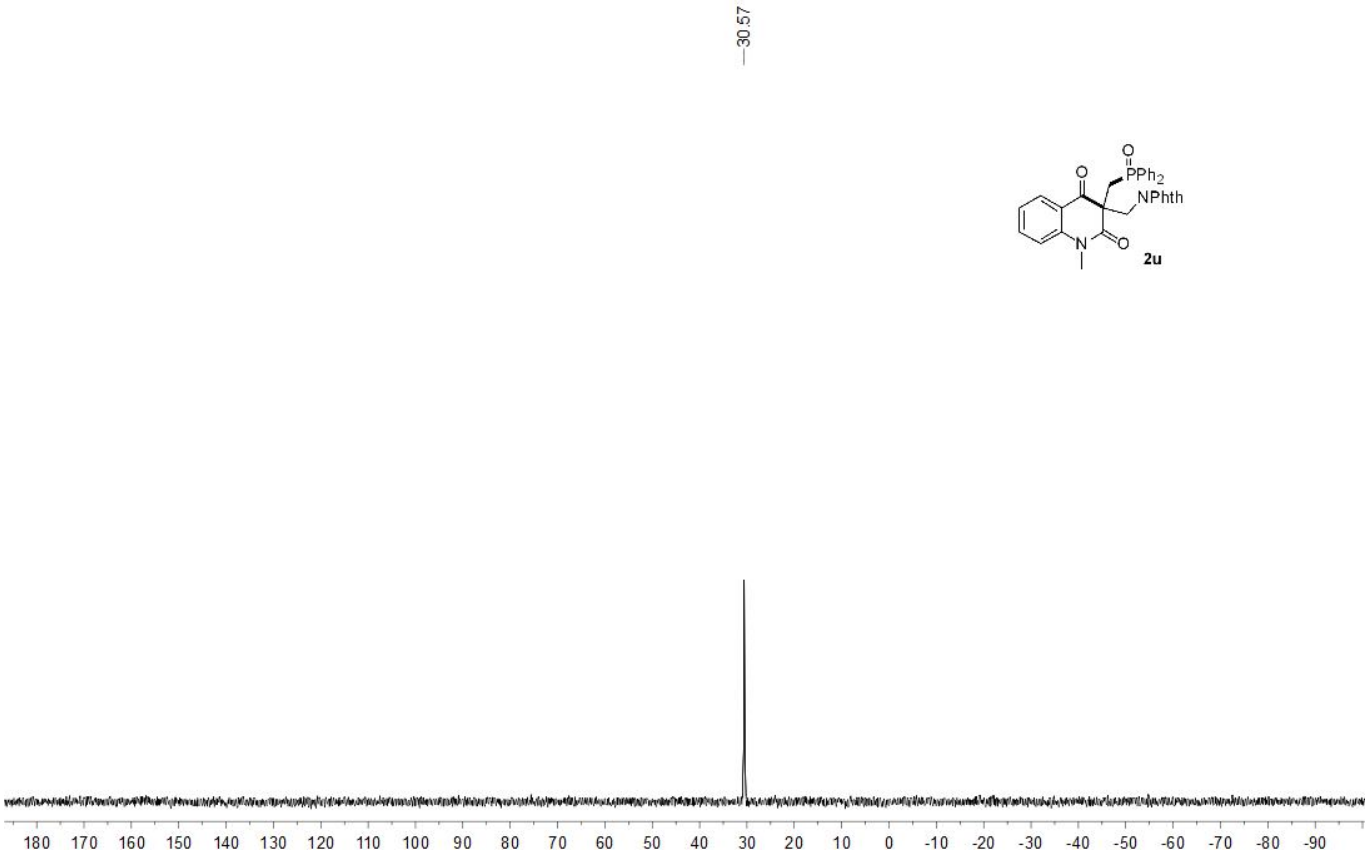
60.19
60.15
44.16
44.02
33.42
32.72
30.23
PPh2
NPhth
2u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NPhth
2u
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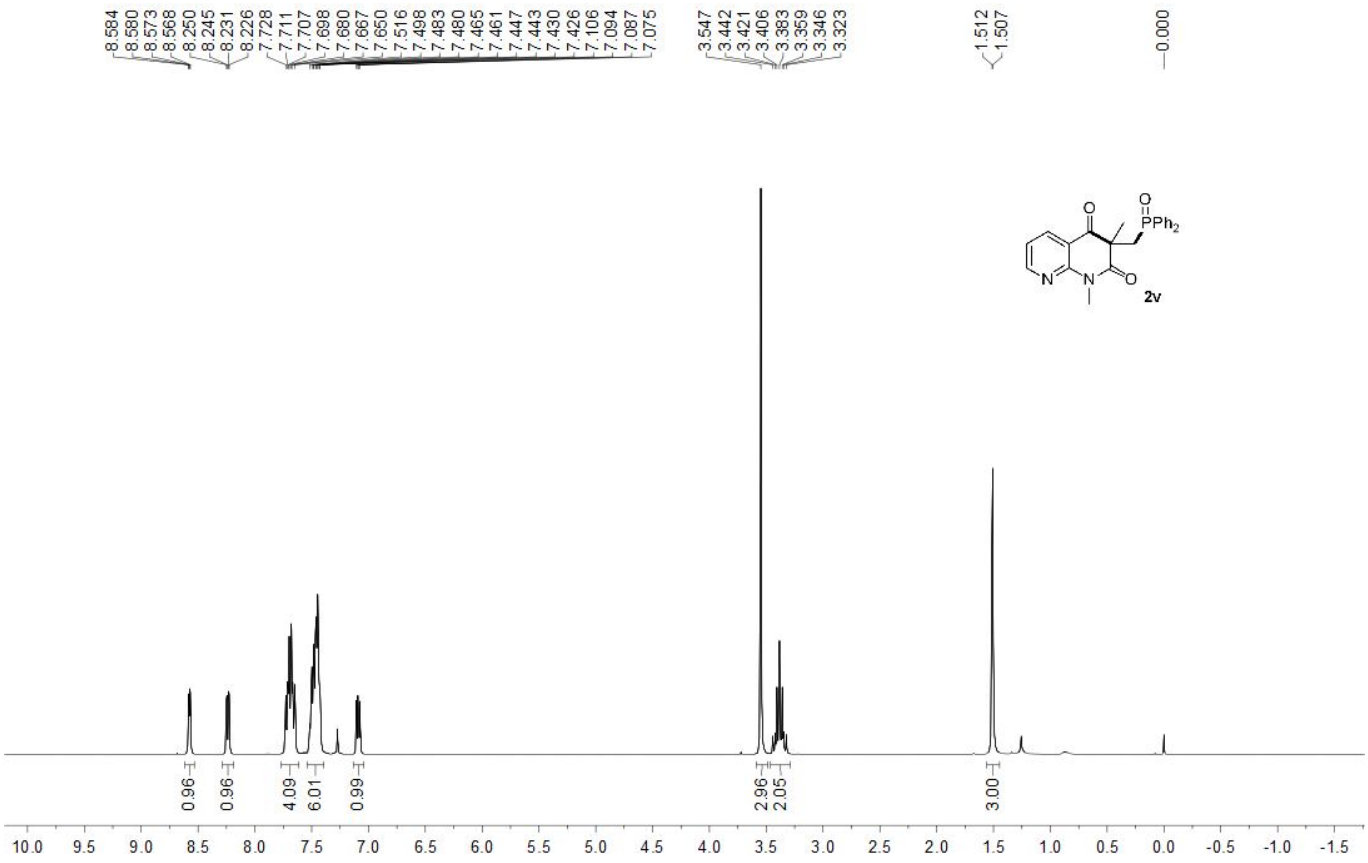
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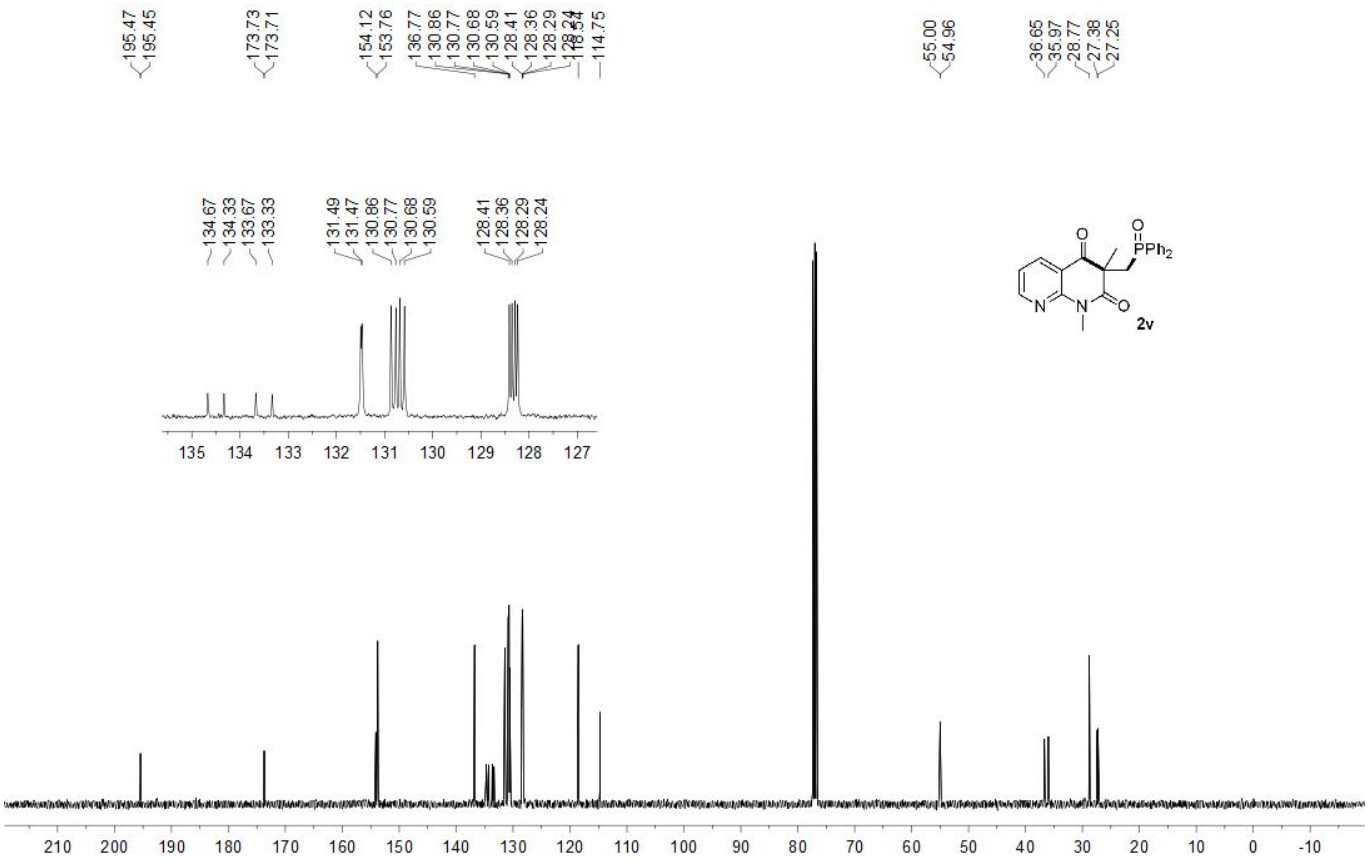
8.573
8.568
8.250
8.245
8.231
8.226
7.728
7.711
7.707
7.698
7.680
7.667
7.650
7.516
7.498
7.483
7.480
7.465
7.461
7.447
7.443
7.430
7.426
7.106
7.094
7.087
7.075
3.547
3.442
3.421
3.406
3.383
3.359
3.346
3.323
1.512
1.507
0.000
PPh2
2v
0.96
0.96
4.09
6.01
0.99
2.96
2.05
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0 -1.5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195.45
173.73
173.71
154.12
153.76
136.77
130.86
130.77
130.68
130.59
128.41
128.36
128.29
114.75
55.00
54.96
36.65
35.97
28.77
27.38
27.25
134.67
134.33
133.67
133.33
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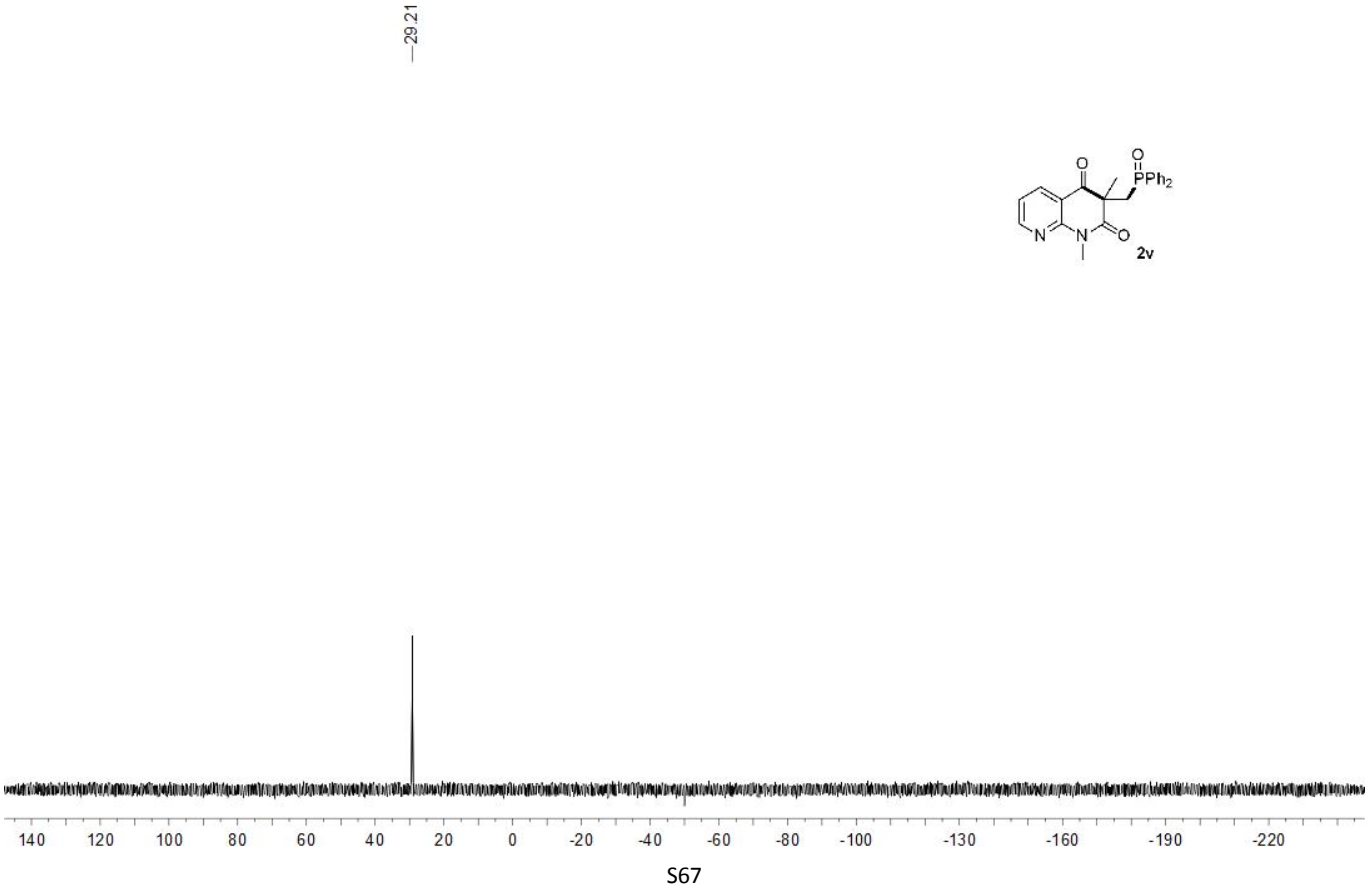
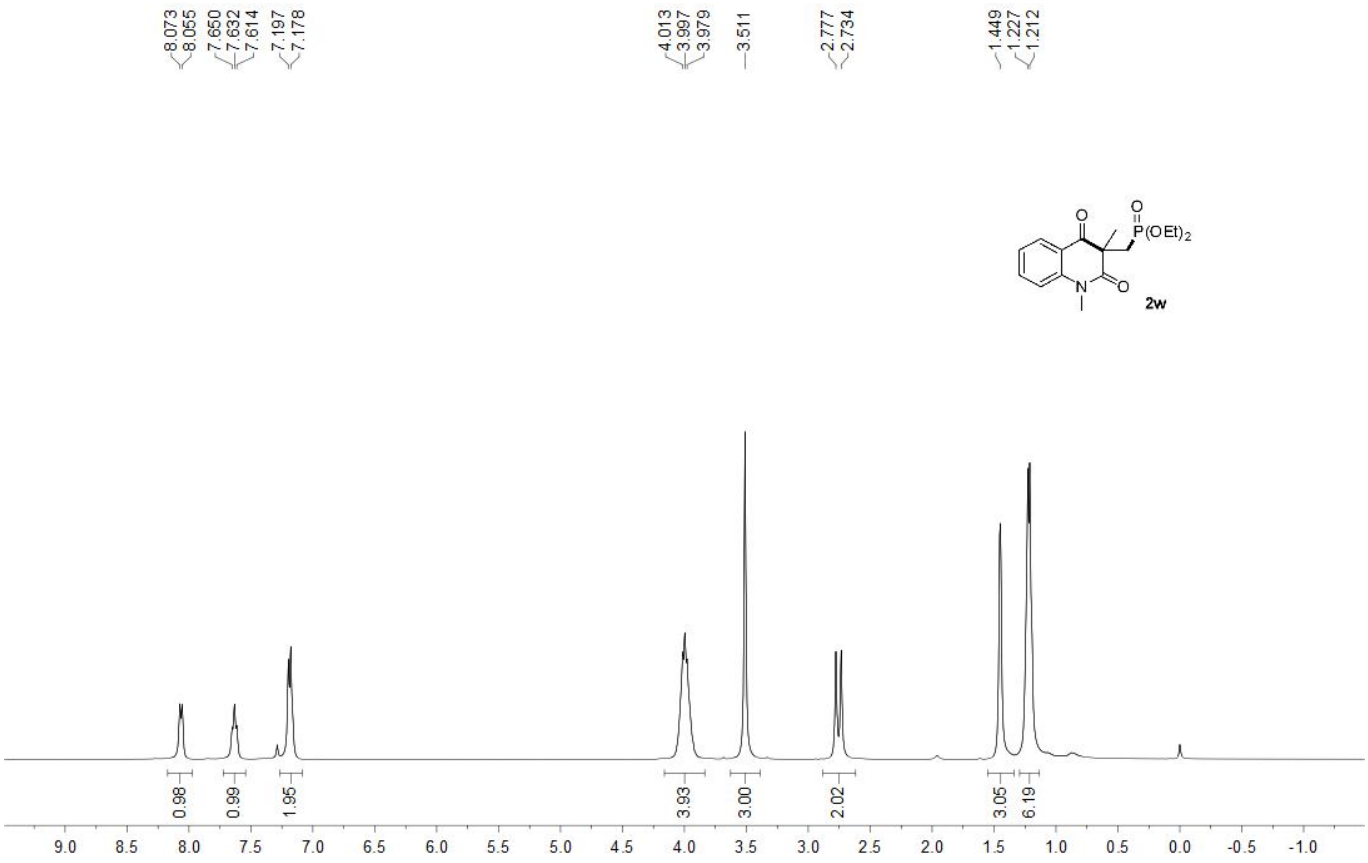
130.77
130.68
130.59
128.41
128.36
128.29
128.24
PPh2
2v


29.21
PPh2
2v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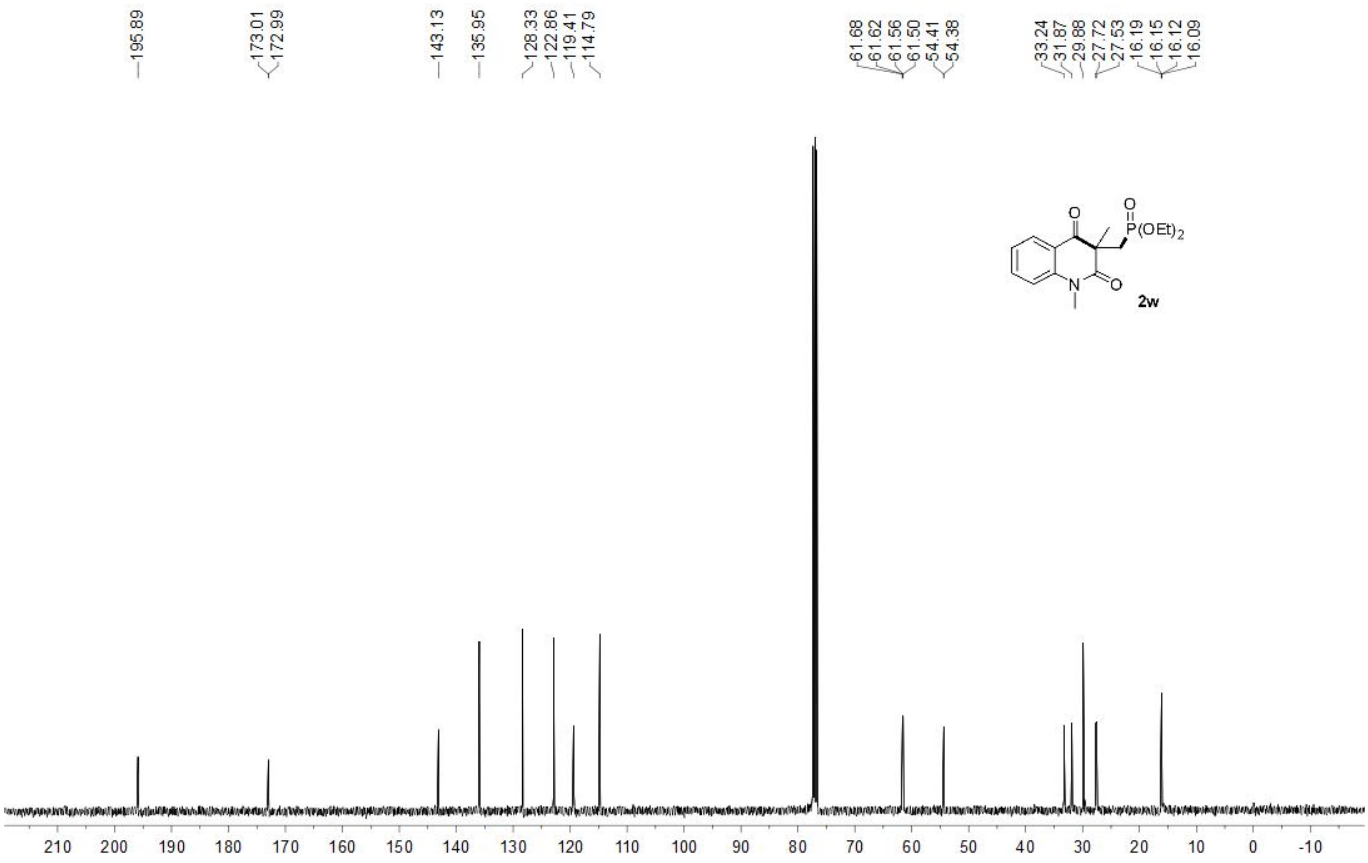
8.055
7.650
7.632
7.614
7.197
7.178
4.013
3.997
3.979
3.511
2.777
2.734
1.449
1.227
1.212
2w
0.98
0.99
1.95
3.93
3.00
2.02
3.05
6.19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173.01
172.99
143.13
135.95
128.33
122.86
119.41
114.79
61.68
61.62
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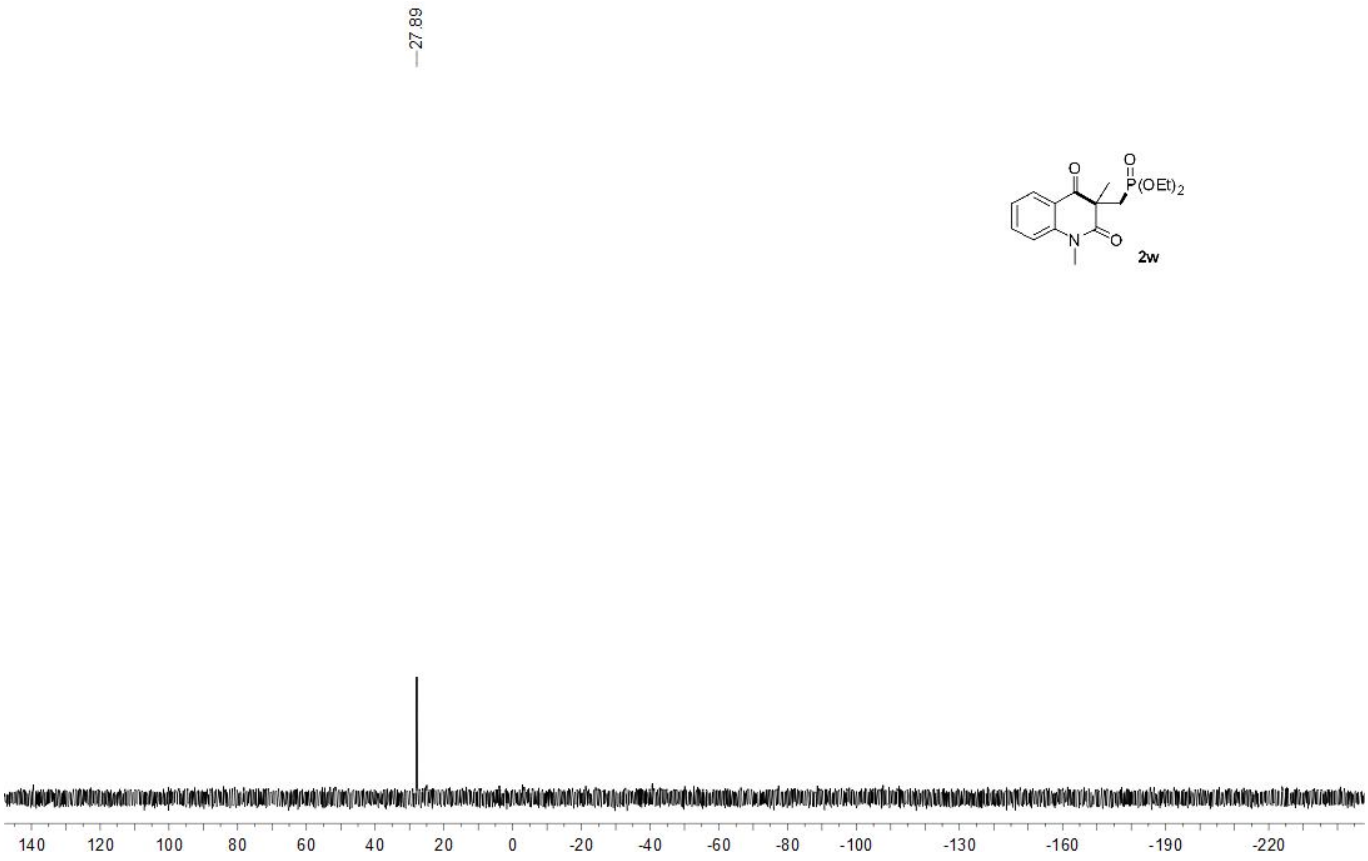
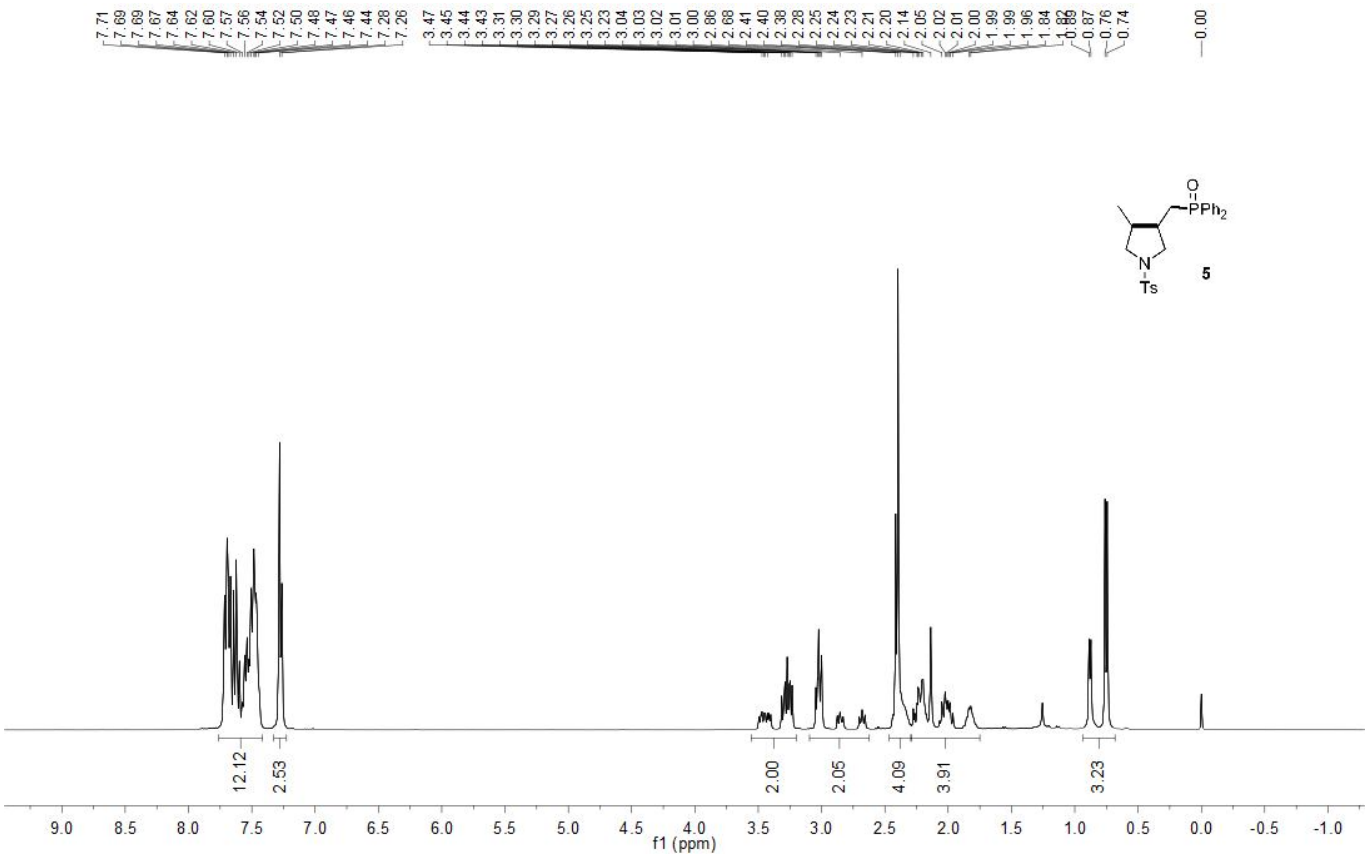
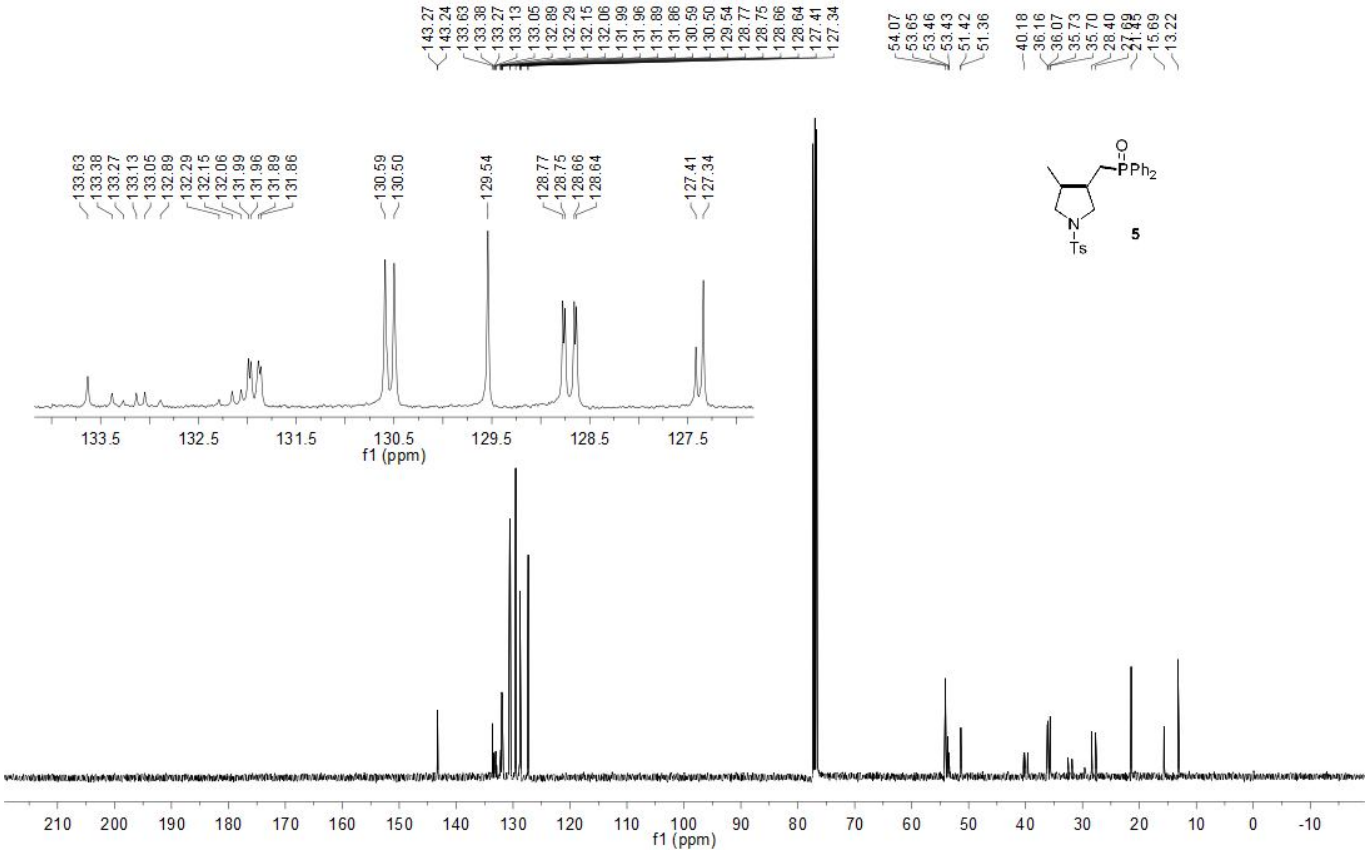
27.53
16.19
16.15
16.12
16.09
2w


27.89
O
O
P(OEt)2
N
O
2w
140 120 100 80 60 40 20 0 -20 -40 -60 -80 -100 -130 -160 -190 -220


7.71 7.69 7.69 7.67 7.64 7.62 7.60 7.57 7.56 7.54 7.52 7.50 7.48 7.47 7.46 7.44 7.28 7.26
3.47 3.45 3.44 3.43 3.31 3.30 3.29 3.27 3.26 3.25 3.23 3.04 3.03 3.02 3.01 3.00 2.86 2.68 2.41 2.40 2.38 2.28 2.25 2.24 2.23 2.21 2.20 2.14 2.05 2.02 2.01 2.00 1.99 1.99 1.96 1.84 0.89 0.87 0.76 0.74
0.00
O
PPh2
N
Ts
5
12.12 2.53 2.00 2.05 4.09 3.91 3.23
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1.0
f1 (ppm)


143.27
143.24
133.63
133.38
133.27
133.13
133.05
132.89
132.29
132.15
132.06
131.99
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131.89
131.86
130.59
130.50
129.54
128.77
128.75
128.66
128.64
127.41
127.34
54.07
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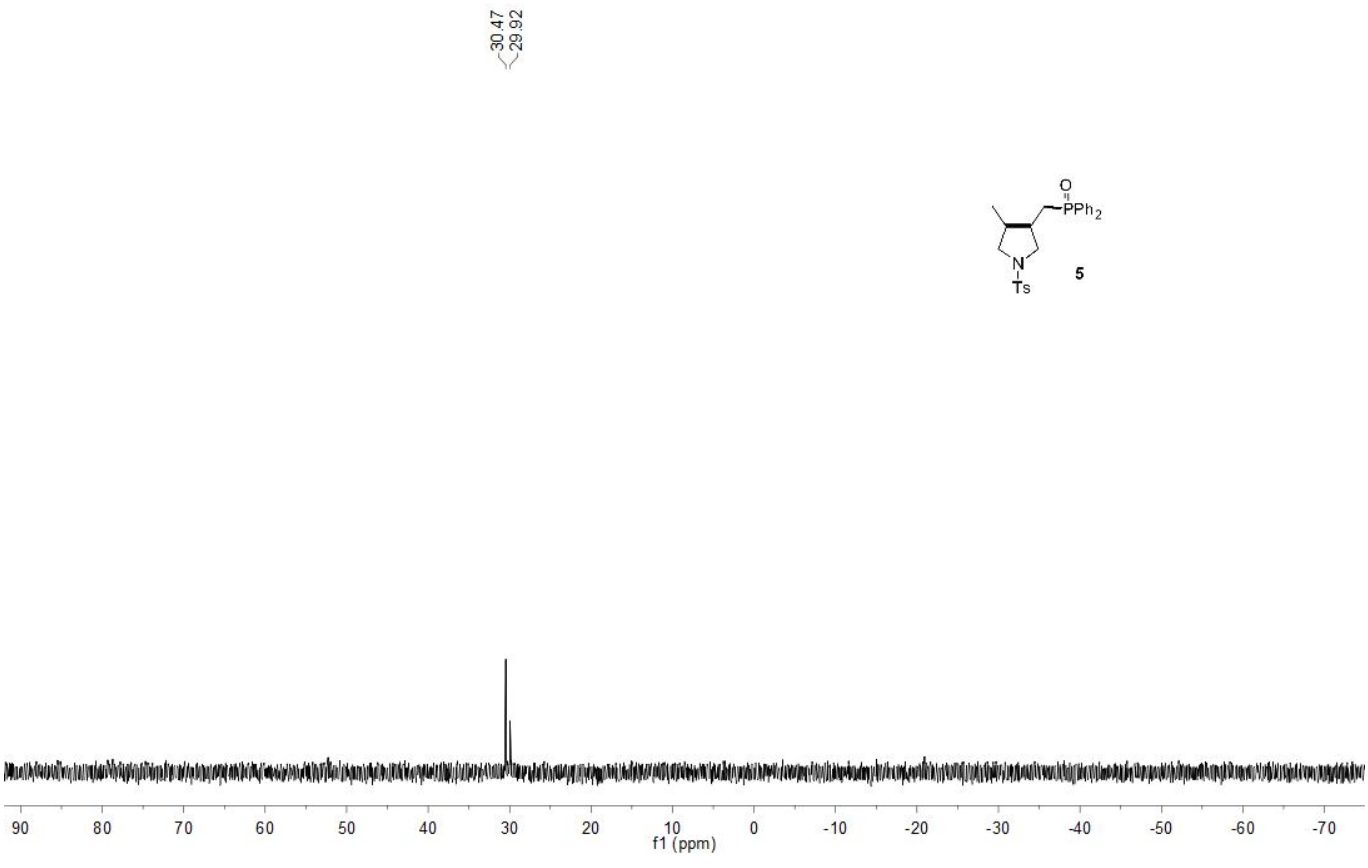
53.46
53.43
51.42
51.36
40.18
36.16
36.07
35.73
35.70
28.40
21.45
15.69
13.22
O
PPh2
N
Ts
5
f1 (ppm)


30.47
29.92
O
PPh2
N
Ts
5
f1 (ppm)