Supporting Information

Tungstate catalysis: pressure-switched 2- and 6- electron reductive functionalization of CO_2 with amines and phenylsilane

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1. General experimental methods

General analytic methods. All of the products are characterized by ^{1}H NMR, ^{13}C NMR and mass spectroscopy, which are consistent with those reported in the literature. ^{1}H and ^{13}C NMR spectra are recorded on a Bruker 400 spectrometer at 20 ^{o}C. All ^{1}H NMR spectra are reported in parts per million (ppm) downfield of TMS and measured at 400 MHz relative to the signals for $CDCl_3$ (7.26 ppm), CD_3CN (1.94 ppm) with ^{1}H decoupling. Coupling constants, *J*, are reported in hertz (Hz). Multiplets are assigned as singlet (s), doublet (d), triplet (t), doublet of doublet (dd) and multiplet (m). ^{13}C NMR was recorded at 101 MHz relative to $CDCl_3$ (77.16 ppm), CD_3CN (118.26 ppm). Mass spectra are recorded on a Shimadzu GCMS-QP2010 equipped with a RTX-5MS capillary column at an ionization voltage of 70 eV. The data are given as mass units per charge (m/z). GC analyses are performed on a Shimadzu GC-2014 equipped with a capillary column (RTX-17 30 m × 0.25 μm) using a flame ionization detector.

Materials. Anhydrous CH_3CN was purified according to *Purification of Common Laboratory Chemicals*. Unless otherwise noted, carbon dioxide (99.999%) was used. The amines, hydrosilanes and other chemicals were commercially available from TCI, Aladdin or Alfa Aesar and used without further purification.

2. General experimental procedure

General procedure for reductive functionalization of CO_2 with amines to methylaimnes

Potassium tungstate (12.2 mg, 7.5 mol% relative to amine), amine (0.5 mmol), CH_3CN (2 mL) and phenylsilane (186 μL, 1.5 mmol) was added successively into a 10 mL over-dried Schlenk tube equipped with a stir-bar. The reaction mixture was stirred at 70 ^{o}C for 12 h under an atmosphere of CO_2 (99.999%, balloon). Upon completion, the conversion and yield were determined by GC or ^{1}H NMR technique using 1,3,5-trimethyoxybenzene (40.0 mg) as an internal standard. The reaction mixture was purified by column chromatography on silica gel using n-hexane/ethyl acetate as eluent to afford the corresponding methylamine.

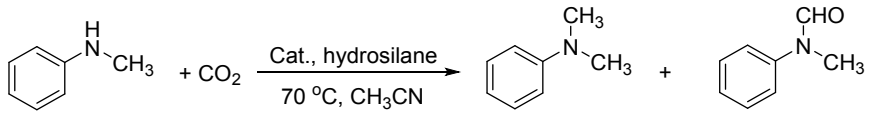
General procedure for reductive functionalization of CO_2 with amines to formylamides

Potassium tungstate (12.2 mg, 7.5 mol% relative to amine), amine (0.5 mmol), CH_3CN (2 mL)

and phenylsilane (186 μL, 1.5 mmol) was added successively into a 10 mL (inner volumes) stainless steel autoclave at room temperature. Then CO_2 was charged into the reactor up to 2 MPa. The autoclave was heated at 70 °C for 12 h. Upon completion, the reactor was cooled to 0 °C in ice-water bath and carefully depressurized to atmospheric pressure. The conversion and yield were determined by GC or ^{1}H NMR technique using 1,3,5-trimethyoxybenzene (40.0 mg) as an internal standard. The reaction mixture was purified by column chromatography on silica gel using n-hexane/ethyl acetate as eluent to afford the desired formylamide.

3. Investigation to various hydrosilanes

Table S1. Hydrosilane effect on the reductive functionalization of CO_2 with **1a** [a]



1a **2a** **3a**

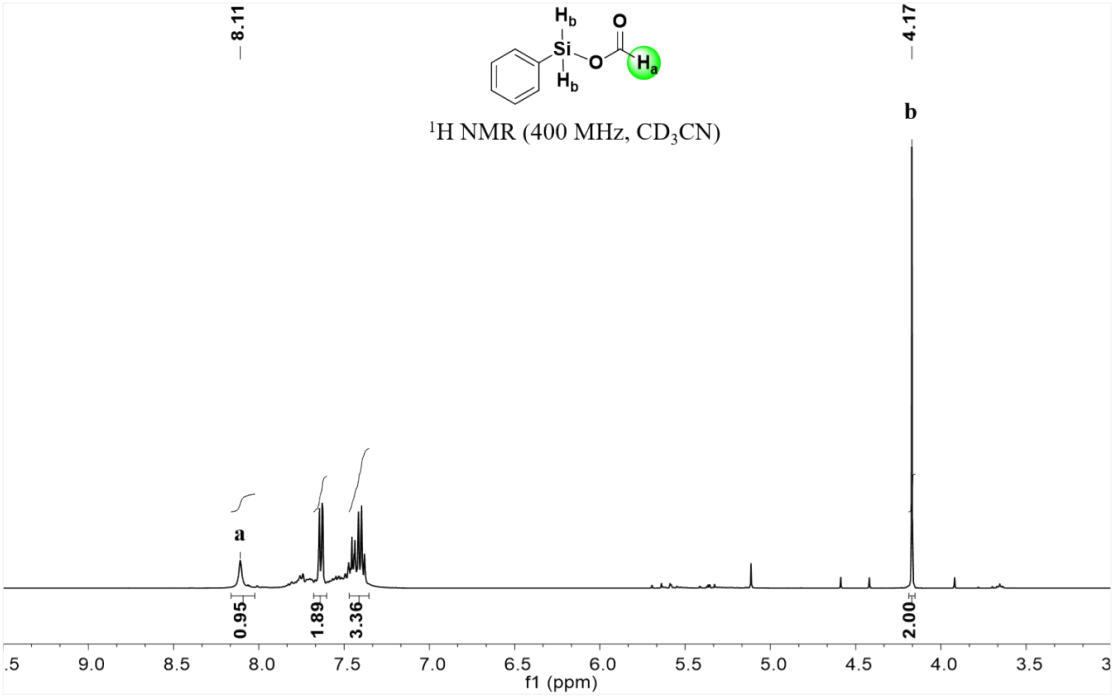
Entry	Hydrosilane/(equiv.)	Conv./%	Yield of **2a**/%[b]	Yield of **3a**/%[b]
1	PMHS(12)	0	–	–
2	TMDS(6)	0	–	–
3	Et_3SiH(12)	0	–	–
4	Ph_3SiH(12)	0	–	–
5	Me_2PhSiH(12)	0	–	–
6	$(EtO)_2MeSiH$(12)	0	–	–
7	$(EtO)_3SiH$(12)	0	–	–
8	Ph_2SiH_2(6)	20	10	–
9	$PhSiH_3$(4)	>99	97	trace
10	$PhSiH_3$(3)	>99	97	trace
11[c]	$PhSiH_3$(3)	>99	trace	94
12	$PhSiH_3$(2)	71	49	8
13[c]	$PhSiH_3$(2)	89	6	80

[a]Reaction conditions: **1a** (0.0535 g, 0.5 mmol), K_2WO_4 (0.0122 g, 7.5 mol%), hydrosilane (the amount as shown in the table), 1 bar CO_2, 70℃, CH_3CN (2 mL), 12 h. [b]Determined by GC using 1,3,5-trimethyoxybenzene as an internal standard. [c]2 MPa CO_2. PMHS: poly(methylhydrosiloxane), TMDS: 1,1,3,3-tetramethyldisilazane

4. NMR monitoring of silyl formate intermediate in *N*-formylation

Potassium tungstate (12.2 mg, 0.0375 mmol), CD_3CN (2 mL) and phenylsilane (186 μL, 1.5 mmol)

was added successively into a 10 mL (inner volumes) stainless steel autoclave at room temperature. Then CO_2 was charged into the reactor up to 2 MPa. The autoclave was heated at 70 ^oC for 4 h. Upon completion, the reactor was cooled to 0 ^oC in ice-water bath and carefully depressurized to atmospheric pressure. Then 0.5 mL reaction liquid was transferred into an NMR tube for ^1H NMR and ^{13}C NMR analysis, as shown in Fig.S1. The result indicates that a silyl formate is generated. ^1H NMR (400 MHz, CD_3CN) δ 8.11 (s, 1H), 7.64 (dd, J = 7.9, 1.5 Hz, 2H), 7.47 – 7.38 (m, 3H), 4.17 (s, 2H) ppm; ^{13}C NMR (101 MHz, CD_3CN) δ 160.33, 136.79, 135.52, 131.03, 129.25 ppm.



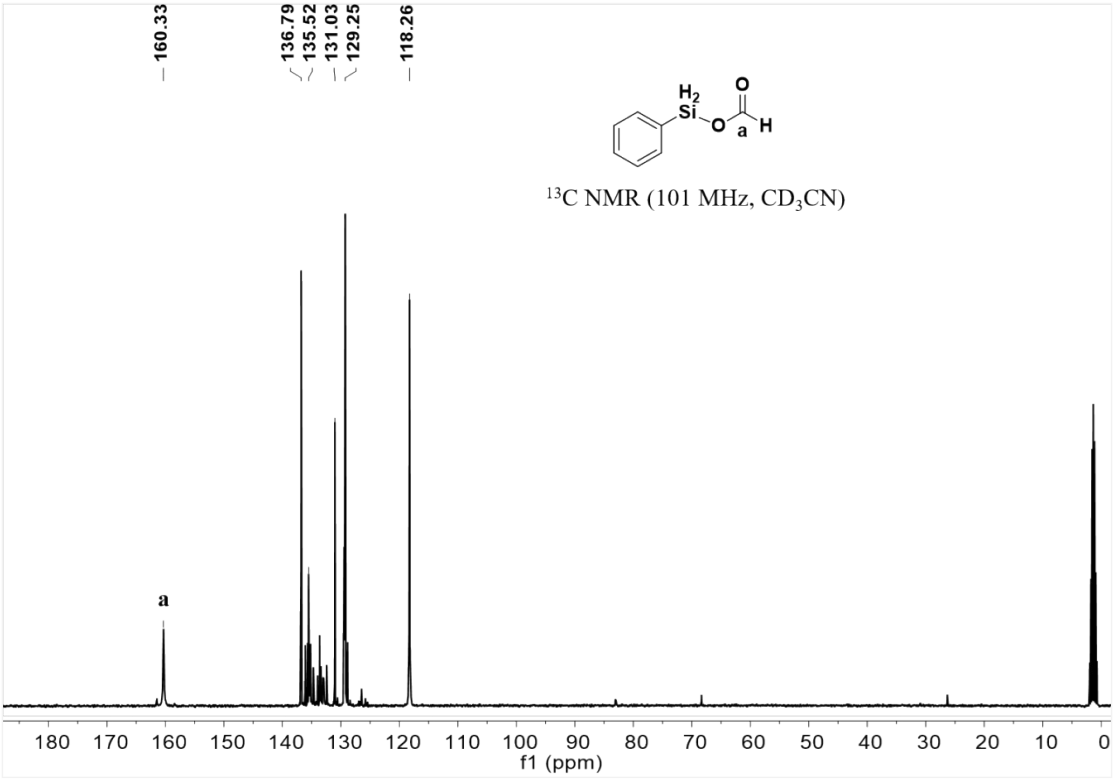
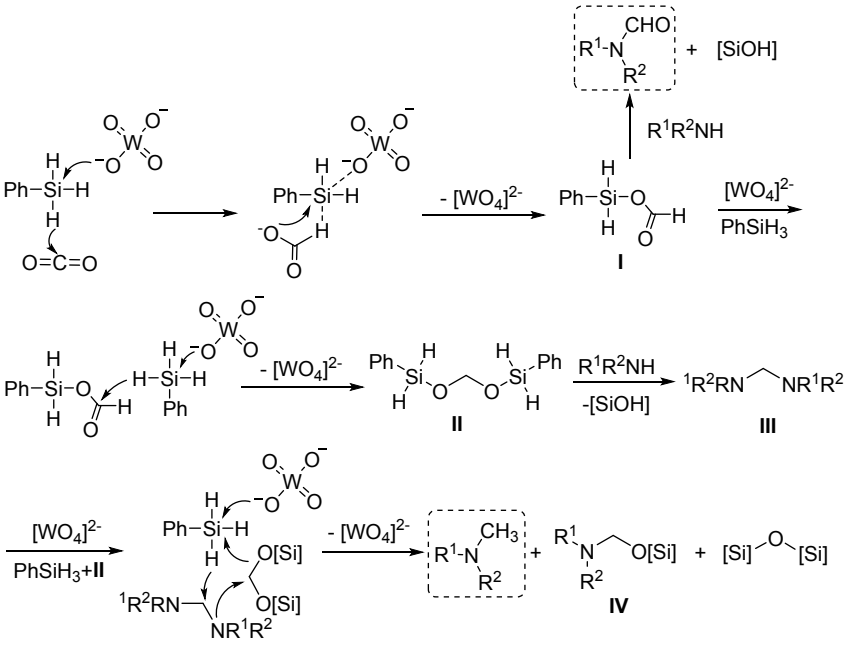


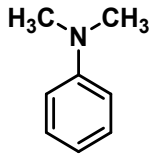
Fig. S1 ^{1}H NMR spectral (a) and ^{13}C NMR spectral (b) of the reaction mixture of CO_2, $PhSiH_3$ and K_2WO_4 (CD_3CN, 293 K)

5. Possible catalytic mechanism

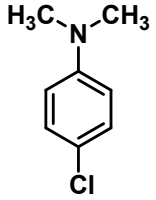


Scheme S1 Possible catalytic mechanism of tungstate to the reductive functionalization of CO_2.

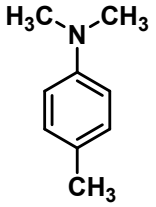
6. Characterization data of methylamines, formamides and aminal



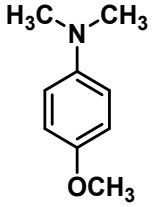
***N*,*N*-Dimethylaniline (2a)**: Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 (dd, J = 8.8, 7.3 Hz, 2H), 6.76 – 6.72 (m, 3H), 2.94 (s, 6H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.74, 129.19, 116.76, 112.78, 40.77 ppm. GC-MS (EI, 70 eV) m/z (%) 121.10 (81.27), 120.10 (100.00), 77.00 (29.54).



4-Chloro-*N*,*N*-dimethylaniline (2b): Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17 (d, J = 8.9 Hz, 2H), 6.64 (d, J = 8.7 Hz, 2H), 2.92 (s, 6H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.26, 128.91, 121.52, 113.75, 40.80 ppm. GC-MS (EI, 70 eV) m/z (%) 157.15 (25.26), 156.10 (39.01), 155.15 (82.98), 154.10 (100.00).

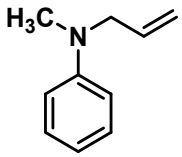


***N*,*N*-Dimethyl-p-toluidine (2c)**: Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.05 (d, J = 8.1 Hz, 2H), 6.69 (d, J = 8.2 Hz, 2H), 2.89 (s, 6H), 2.25 (s, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 148.93, 129.70, 126.25, 113.35, 41.21, 20.38 ppm. GC-MS (EI, 70 eV) m/z (%) 135.25 (77.13), 134.14 (100.00), 91.10 (22.95).

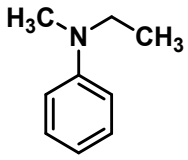


4-Methoxy-*N*,*N*-dimethylaniline (2d): White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.84 (d, J = 9.1 Hz, 2H), 6.76 (d, J = 9.1 Hz, 2H), 3.76 (s, 3H), 2.86 (s, 6H) ppm.^{13}C NMR (101 MHz, $CDCl_3$)

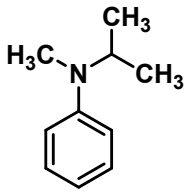
δ 152.28, 145.68, 115.20, 114.76, 55.88, 42.08 ppm. GC-MS (EI, 70 eV) m/z (%) 151.25 (59.48), 136.20 (100.00).



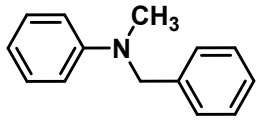
***N*-Allyl-*N*-methylaniline (2e)**: Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25 – 7.20 (m, 2H), 6.74 – 6.68 (m, 3H), 5.84 (ddt, J = 15.5, 10.2, 5.1 Hz, 1H), 5.18 – 5.13 (m, 2H), 3.92 (d, J = 4.9 Hz, 2H), 2.93 (s, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.59, 133.91, 129.23, 116.52, 116.28, 112.56, 55.39, 38.13 ppm. HRMS (ESI, m/z) calcd. For $C_{10}H_{13}N$ $[M+H]^+$: 148.1126, found: 148.1124.



***N*-Ethyl-*N*-methylaniline (2f)**: Yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (t, J = 7.7 Hz, 2H), 6.72 – 6.68 (m, 3H), 3.39 (q, J = 7.0 Hz, 2H), 2.89 (s, 3H), 1.10 (t, J = 7.1 Hz, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.21, 129.27, 116.13, 112.49, 46.91, 37.54, 11.29 ppm. GC-MS (EI, 70 eV) m/z (%) 135.14 (31.98), 120.14 (100.00), 77.04 (33.12).

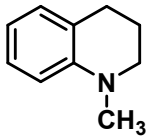


***N*-Isopropyl-*N*-methylaniline (2g)**: Yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (t, J = 7.9 Hz, 2H), 6.79 (d, J = 8.2 Hz, 2H), 6.69 (t, J = 7.2 Hz, 1H), 4.14 – 4.04 (m, 1H), 2.72 (s, 3H), 1.15 (d, J = 6.6 Hz, 6H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.32, 129.23, 116.54, 113.44, 49.03, 29.91, 19.42 ppm. GC-MS (EI, 70 eV) m/z (%) 149.19 (19.30), 134.14 (100.00), 77.04 (20.49).

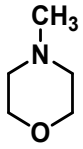


***N*-Benzyl-*N*-methylaniline (2h)**: Yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 – 7.29 (m, 2H), 7.25 – 7.19 (m, 4H), 6.76 – 6.69 (m, 4H), 4.53 (s, 2H), 3.01 (s, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.83, 139.13, 129.30, 128.68, 126.98, 126.84, 116.63, 112.45, 56.74, 38.64 ppm. GC-MS (EI, 70 eV) m/z (%) 197.15 (79.61), 196.10 (25.26), 120.10 (71.12), 91.10 (100.00),

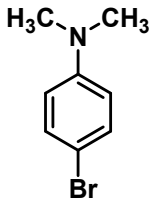
77.05 (27.50).



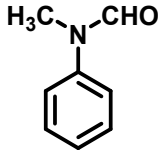
1-Methyl-1,2,3,4-tetrahydroquinoline (2i): Yellow liquid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.09 (t, J = 7.7 Hz, 1H), 6.97 (d, J = 7.7 Hz, 1H), 6.62 (t, J = 7.4 Hz, 2H), 3.25 – 3.22 (m, 2H), 2.90 (s, 3H), 2.79 (t, J = 6.5 Hz, 2H), 2.03 – 1.97 (m, 2H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 146.86, 128.94, 127.17, 123.01, 116.35, 111.11, 51.42, 39.27, 27.93, 22.59 ppm. GC-MS (EI, 70 eV) m/z (%) 148.12 (14.62), 147.10 (90.27), 146.16 (100.00), 132.20 (13.61), 131.20 (27.19), 130.22 (17.58).



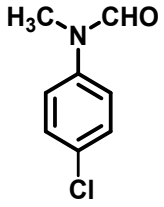
4-Methylmorpholine (2j): Colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 3.72 – 3.70 (m, 4H), 2.41 (s, 4H), 2.29 (s, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 66.83, 55.36, 46.39 ppm. GC-MS (EI, 70 eV) m/z (%) 101.10 (100.00), 100.10 (36.36), 71.08 (60.10).



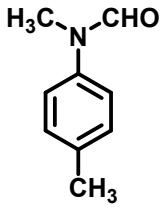
4-Bromo-*N*,*N*-dimethylaniline (2m): Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 (dd, J = 8.8, 7.3 Hz, 2H), 6.59 (d, J = 8.8 Hz, 3H), 2.94 (s, 6H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.62, 131.80, 114.22, 108.61, 40.69 ppm. GC-MS (EI, 70 eV) m/z (%) 201.05 (93.40), 200.05 (100.00), 199.05 (99.31), 198.05 (97.75), 118.20 (45.21), 77.15 (20.97)



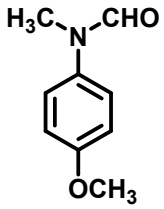
***N*-Methylformanilide (3a)**: Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.48 (s, 1H), δ 7.42 (t, J = 7.8 Hz, 2H), 7.28 (t, J = 7.4 Hz, 1H), 7.18 (d, J = 7.6 Hz, 2H), 3.33 (s, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.37, 142.19, 129.65, 126.42, 122.37, 32.06 ppm. GC-MS (EI, 70 eV) m/z (%) 135.20 (70.67), 106.15 (100.00), 94.15 (30.44), 79.15 (20.53), 77.15 (42.47), 66.10 (21.04).



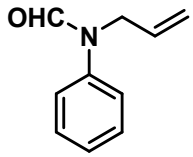
***N*-(4-Chlorophenyl)-*N*-methylformamide (3b)**: Yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.45 (s, 1H), 7.39 (d, J = 8.7 Hz, 2H), 7.12 (d, J = 8.7 Hz, 2H), 3.30 (s, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.10, 140.84, 132.13, 129.87, 123.62, 32.17 ppm. GC-MS (EI, 70 eV) m/z (%) 169.15 (54.96), 142.15 (31.48), 140.15 (100.00), 128.15 (44.00), 77.15 (26.62).



***N*,4'-Dimethylformanilide (3c)**: Yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.42 (s, 1H), 7.21 (d, J = 8.0 Hz, 2H), 7.06 (d, J = 8.3 Hz, 2H), 3.30 (s, 3H), 2.37 (s, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.50, 139.78, 136.49, 130.26, 122.66, 32.34, 20.98 ppm. GC-MS (EI, 70 eV) m/z (%) 149.25 (76.70), 120.20 (100.00), 108.15 (42.14), 91.15 (33.91), 65.15 (21.57).

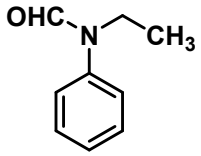


***N*-Methyl-4-methoxylformanilde (3d)**: Brown oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.34 (s, 1H), 7.10 (d, J = 8.9 Hz, 2H), 6.93 (d, J = 8.9 Hz, 2H), 3.82 (s, 3H), 3.27 (s, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.44, 158.27, 135.22, 124.63, 114.74, 55.54, 32.66 ppm. GC-MS (EI, 70 eV) m/z (%) 165.20 (100.00), 124.20 (62.19), 122.20 (95.97), 94.15 (33.79), 65.15 (21.68).

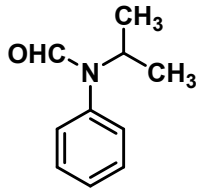


***N*-Allylformanilide (3e):** Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.49 (s, 1H), 7.42 – 7.38 (m, 2H), 7.30 – 7.18 (m, 3H), 5.85 (ddd, J = 22.8, 10.5, 5.6 Hz, 1H), 5.22 – 5.16 (m, 2H), 4.42 (dt, J = 5.6, 1.4 Hz, 2H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.12, 141.32, 132.63, 129.71, 126.84,

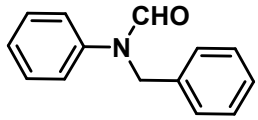
123.68, 117.77, 48.02 ppm. GC-MS (EI, 70 eV) 161.20 (24.46), 132.20 (100.00), 117.15 (20.40), 106.15 (31.65), 104.15 (33.25), 77.10 (53.95).



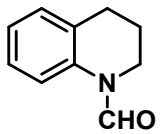
***N*-Ethyl-*N*-phenylformamide (3f):** Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.35 (s, 1H), 7.42 (t, J = 7.7 Hz, 2H), 7.30 (t, J = 7.6 Hz, 1H), 7.17 (d, J = 7.8 Hz, 2H), 3.86 (q, J = 7.2 Hz, 2H), 1.16 (t, J = 7.2 Hz, 3H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.13, 140.89, 129.71, 126.94, 124.34, 40.17, 13.14 ppm. GC-MS (EI, 70 eV) m/z (%) 150.06 (22.86), 149.04 (76.16), 121.06 (97.60), 120.18 (19.42), 107.13 (10.49), 106.18 (100.00).



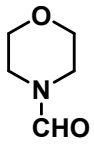
***N*-Isopropyl-*N*-phenylformamide (3g**, dr=31:5**)**: Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.40 (s, 0.14H, minor isomer), 8.15 (s, 0.86H, major isomer), 7.45 – 7.31 (m, 3H), 7.18 – 7.10 (m, 2H), 4.78 (hept, J = 6.8 Hz, 0.86H, major isomer), 4.09 (hept, J = 6.7 Hz, 0.14H, minor isomer), 1.25 (d, J = 6.8 Hz, 0.84H, minor isomer), 1.18 (d, J = 6.8 Hz, 5.16H, major isomer) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) major isomer δ 162.58, 138.43, 129.29, 129.00, 128.20, 45.83, 20.99; minor isomer δ 162.62, 136.65, 129.26, 129.17, 128.07, 51.58, 22.73 ppm. GC-MS (EI, 70 eV) m/z (%) 163.00 (33.19), 148.09 (25.21), 121.06 (100.00), 120.16 (96.50).



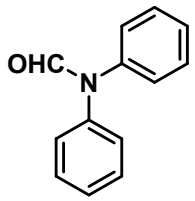
***N*-Benzyl-*N*-phenylformamide (3h)**: Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.58 (s, 1H), 7.37 – 7.27 (m, 8H), 7.12 (d, J = 8.0 Hz, 2H), 5.03 (s, 2H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.40, 140.92, 136.61, 129.55, 128.58, 127.80, 127.42, 126.86, 123.98, 48.80 ppm. GC-MS (EI, 70 eV) m/z (%) 212.11 (20.68), 211.07 (100.00), 210.21 (21.13), 184.12 (7.99), 183.15 (47.09), 182.12 (47.33), 91.28 (84.64).



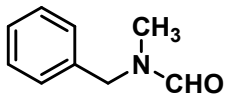
3,4-Dihydroquinoline-1(*2H*)-carbaldehyde (3i, dr=10.8:1**)**: Green oil. ^{1}H NMR (400 MHz, $CDCl_3$) major isomer δ 8.76 (s, 1H), 7.24 – 7.04 (m, 4H), 3.81 – 3.78 (m, 2H), 2.80 (t, J = 6.4 Hz, 2H), 1.97 – 1.91 (m, 2H); minor isomer δ 8.30 (s, 0.09H), 7.42 – 7.30 (m, 0.36H), 3.64 – 3.62 (m, 0.18H), 2.89 (t, J = 6.7 Hz, 0.18H), 2.05 – 1.99 (m, 0.18H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) major isomer δ 161.25, 137.31, 129.72, 129.04, 127.20, 124.66, 117.13, 40.42, 27.22, 22.36; minor isomer δ 161.60, 134.36, 130.47, 129.37, 127.81, 126.37, 122.44, 46.30, 29.78, 23.20 ppm. GC-MS (EI, 70 eV) m/z (%) 162.02 (9.81), 161.07 (71.97), 133.15 (17.56), 132.16 (100.00), 118.22 (16.65), 117.24 (22.51).



Morpholine-4-carbaldehyde (3j): Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (s, 1H), 3.64 (t, J = 4.8 Hz, 2H), 3.60 (t, J = 4.8 Hz, 2H), 3.51 (t, J = 4.8 Hz, 2H), 3.35 (t, J = 4.8 Hz, 2H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 160.84, 67.22, 66.42, 45.78, 40.58 ppm. GC-MS (EI, 70 eV) m/z (%) 115.15 (100.00), 100.10 (77.82), 86.10 (51.09), 85.10 (22.02).

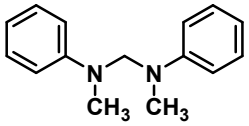


***N*,*N*-Diphenylformamide (3p)**: White solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.67 (s, 1H), 7.40 (dd, J = 12.9, 7.2 Hz, 4H), 7.34 – 7.26 (m, 4H), 7.17 (d, J = 7.5 Hz, 2H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 161.89, 141.93, 139.76, 129.84, 129.33, 127.19, 127.02, 126.27, 125.23 ppm. GC-MS (EI, 70 eV) m/z (%) 198.10 (14.26), 197.04 (100.00), 169.15 (54.78), 168.19 (86.89), 167.22 (46.12).



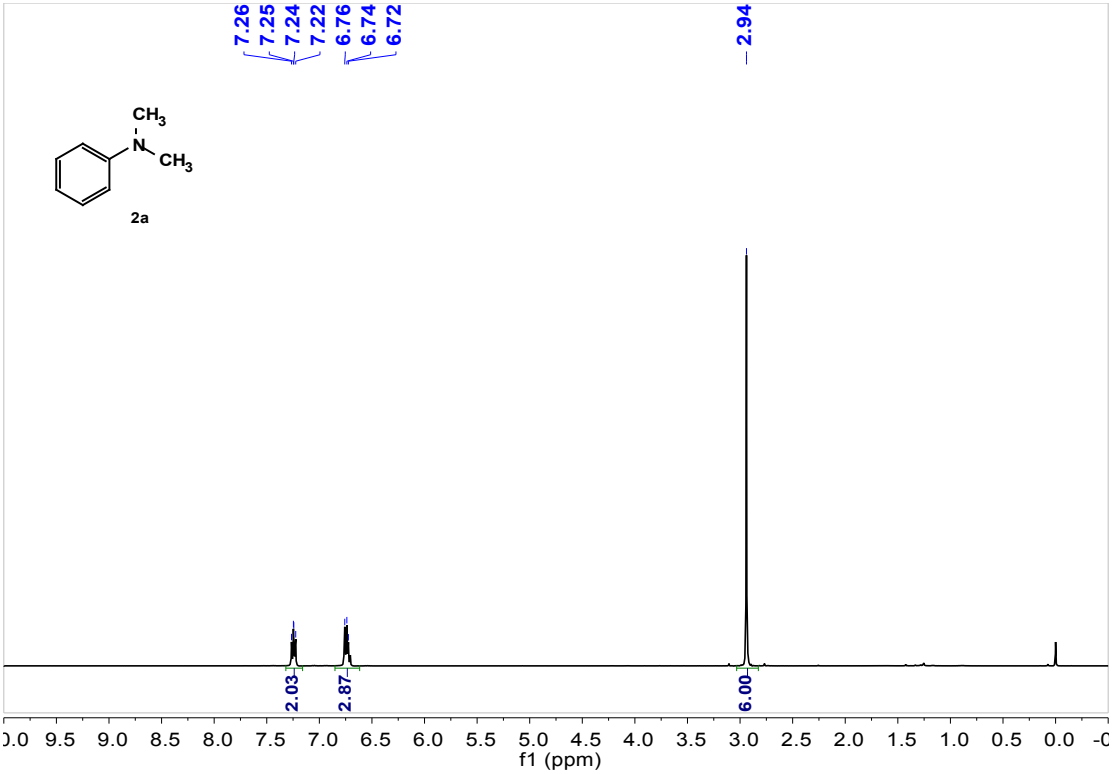
***N*-Benzyl-*N*-methylformamide (3q**, dr=4:3**)**: Purple oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.20 (s, 0.57H, major isomer), 8.07 (s, 0.43H, minor isomer), 7.30 – 7.11 (m, 5H), 4.44 (s, 0.86H, minor isomer), 4.30 (s, 1.14H, major isomer), 2.76 (s, 1.26H, minor isomer), 2.69 (s, 1.74H, major isomer) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 162.78, 162.61, 136.06, 135.80, 128.93, 128.72, 128.27, 128.12, 127.67, 127.43, 53.50, 47.77, 29.74, 29.47 ppm. GC-MS (EI, 70 eV) m/z (%)

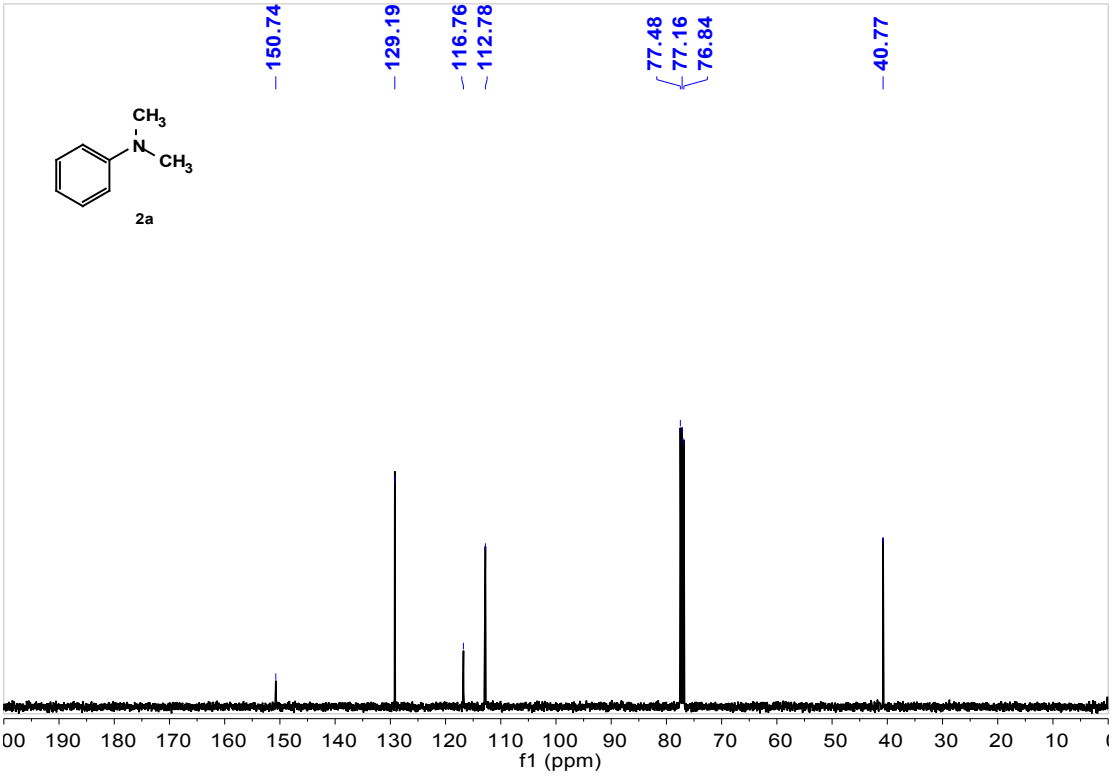
149.10 (100.00), 134.15 (17.80), 106.10 (29.35), 91.10 (57.23), 79.10 (28.00).

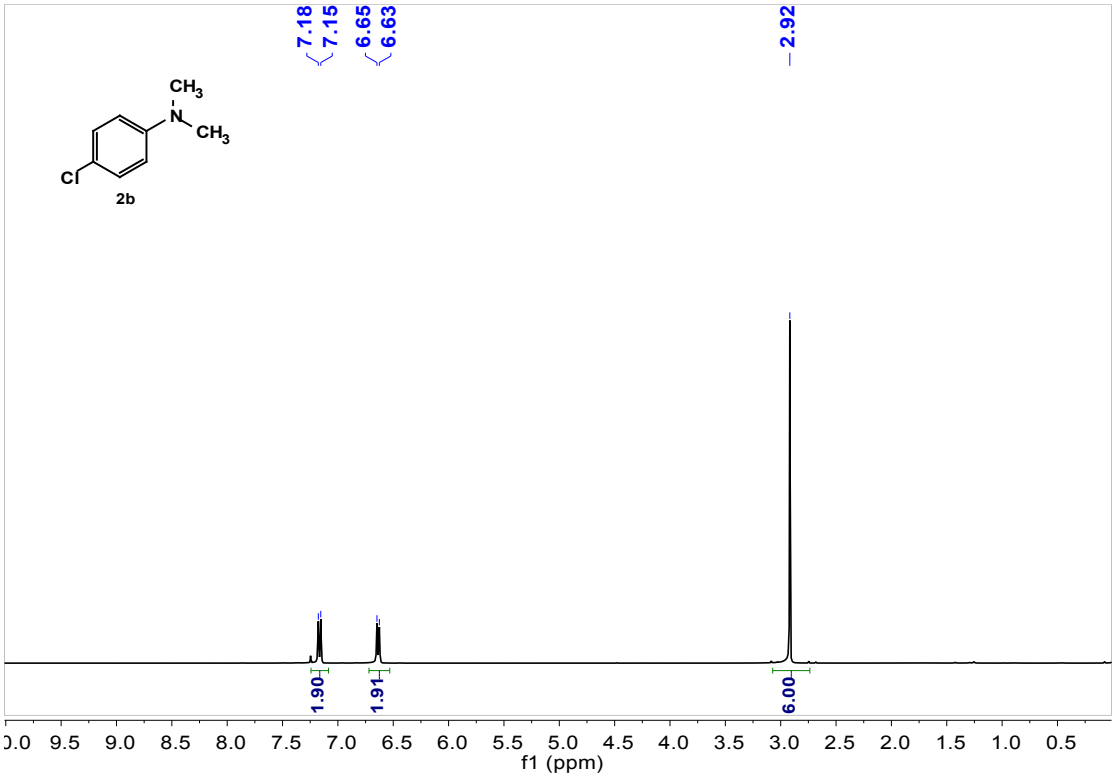


***N*,*N*'-Dimethyl-*N*,*N*'-diphenylmethanediamine** (**III**): Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 – 7.23 (m, 4H), 6.84 (d, J = 8.1 Hz, 4H), 6.78 (t, J = 7.3 Hz, 2H), 4.76 (s, 2H), 2.88 (s, 6H) ppm. ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.36, 129.33, 117.91, 113.81, 70.49, 36.36 ppm. GC-MS (EI, 70 eV) m/z (%) 226.20 (2.83), 120.15 (100), 107.15 (61.88), 106.15(71.81), 77.10 (39.67).

7. ^{1}H NMR and ^{13}C NMR spectrum



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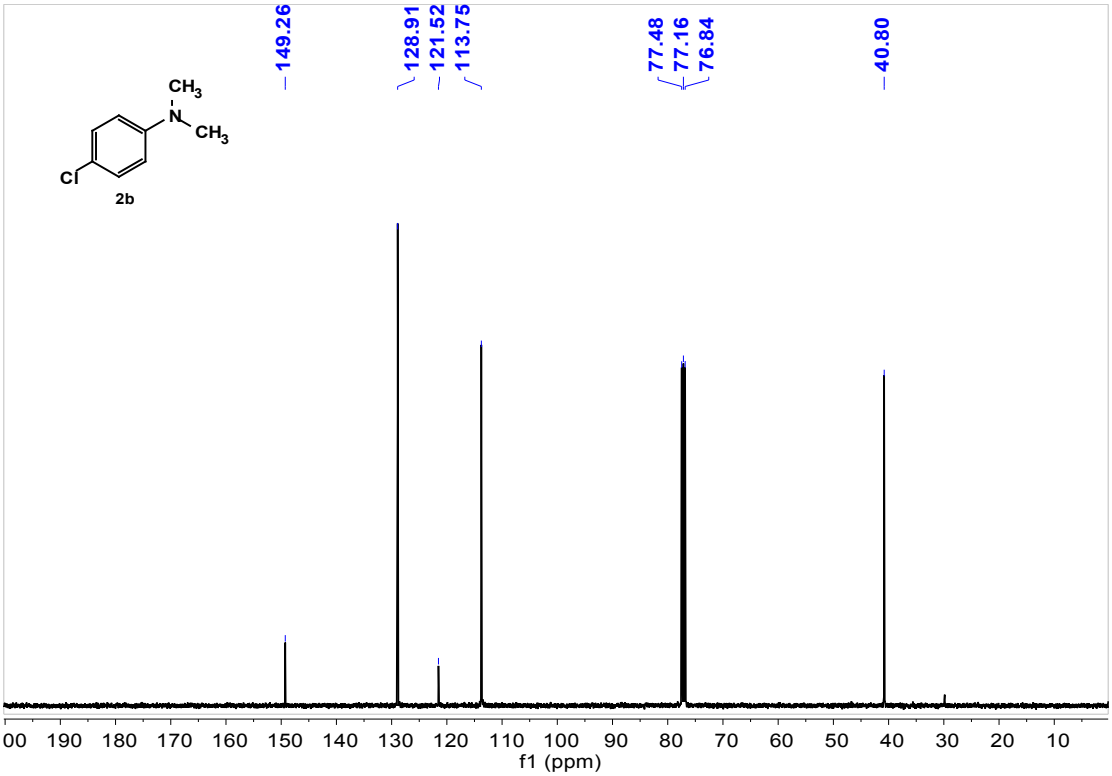
CH3
Cl
2b
1.90
1.91
6.00
f1 (ppm)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77.16
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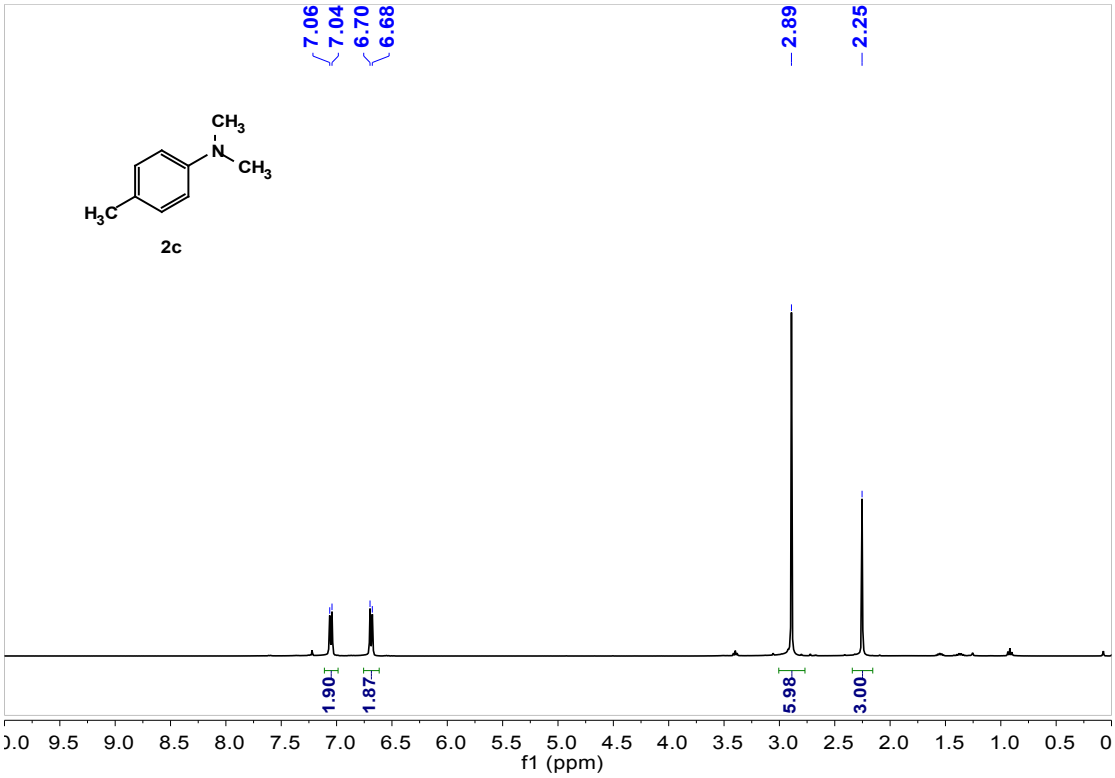
40.80
CH3
N
CH3
Cl
2b
f1 (ppm)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CH3
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2c
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1.87
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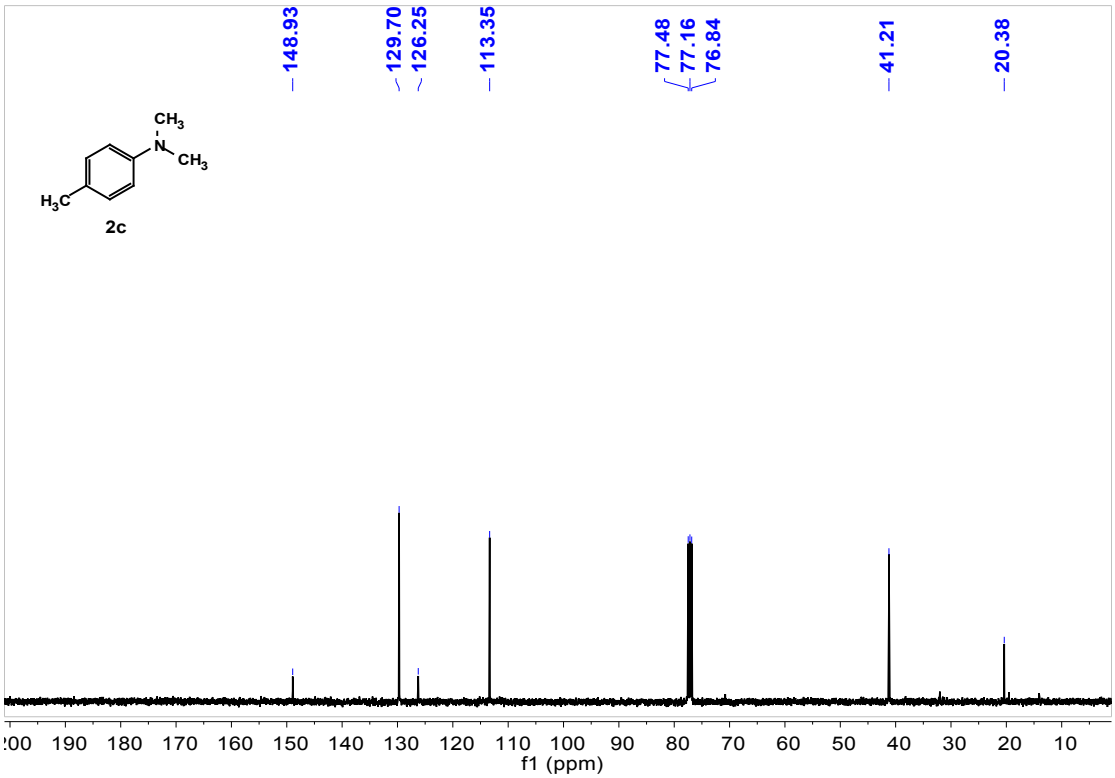
3.00
f1 (ppm)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129.70
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77.16
76.84
41.21
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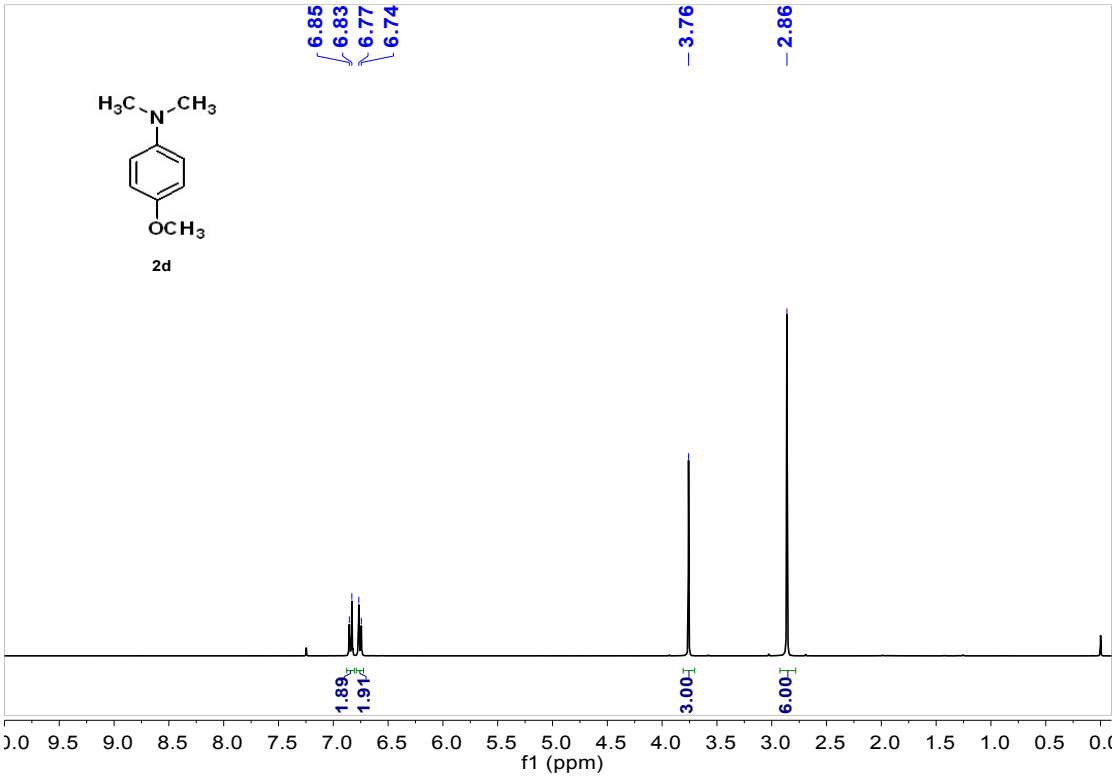
CH3
N
CH3
H3C
2c
f1 (ppm)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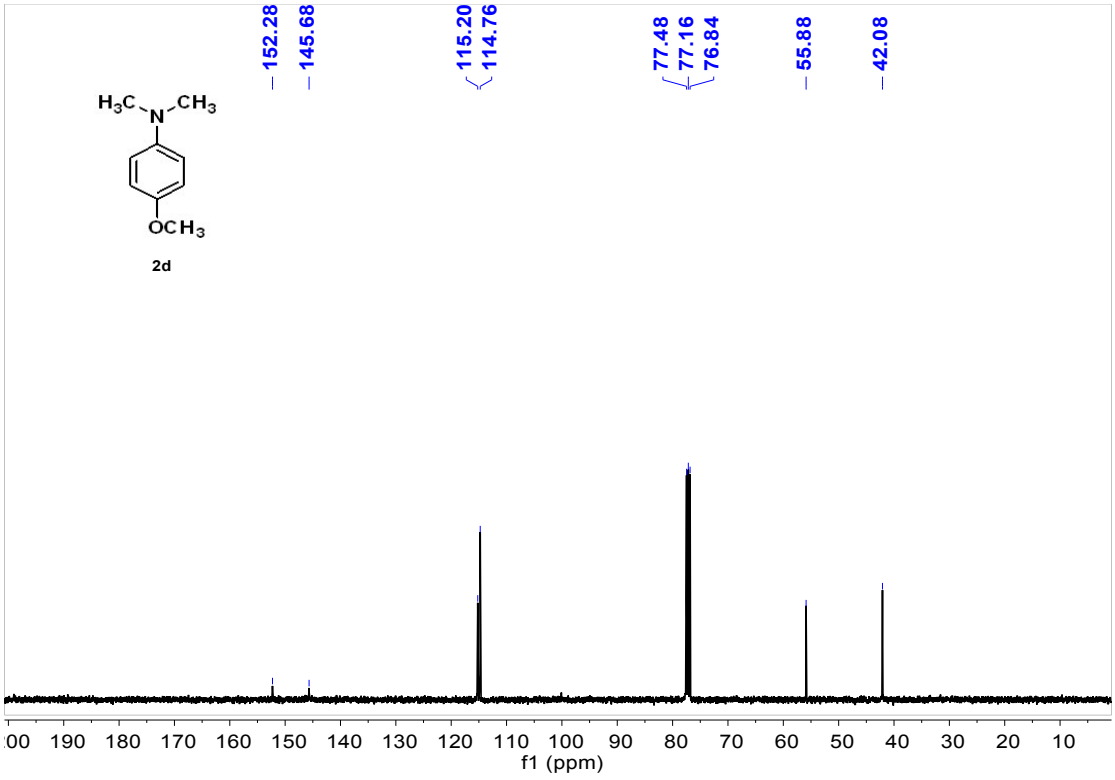
1.91
3.00
6.00
f1 (ppm)

2d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115.20
114.76
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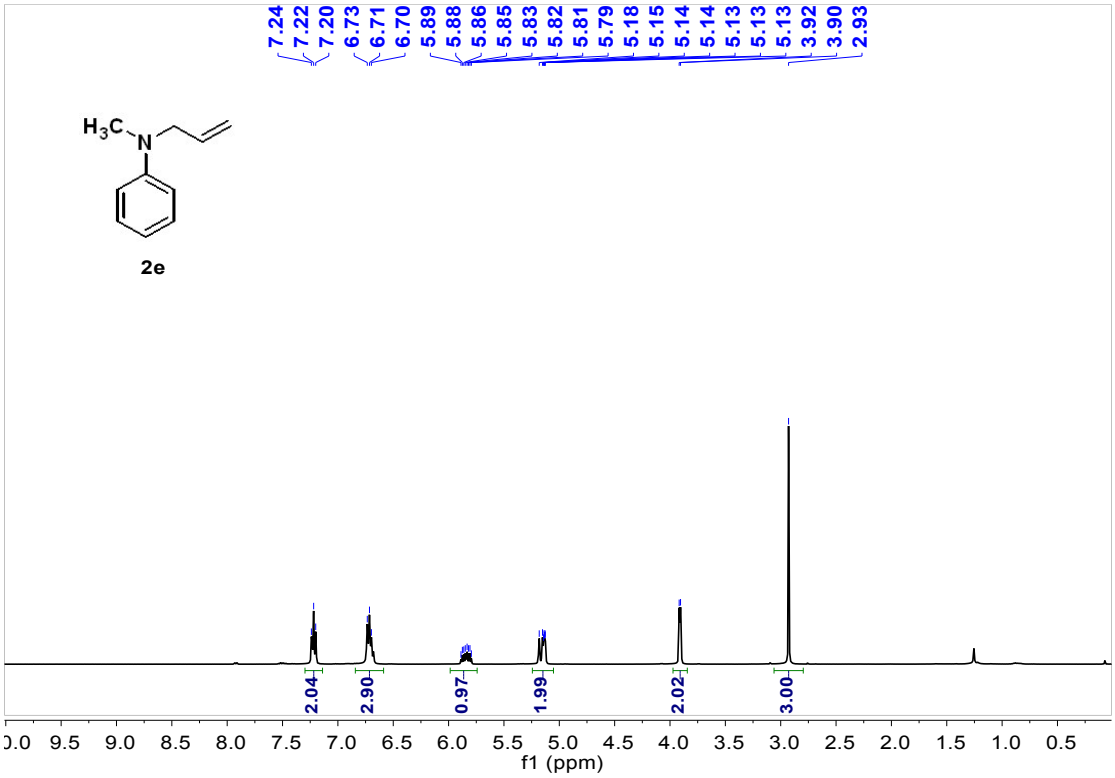
77.16
76.84
55.88
42.08
f1 (ppm)

2d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5.88
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5.85
5.83
5.82
5.81
5.79
5.18
5.15
5.14
5.14
5.13
5.13
5.13
3.92
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H3C
N
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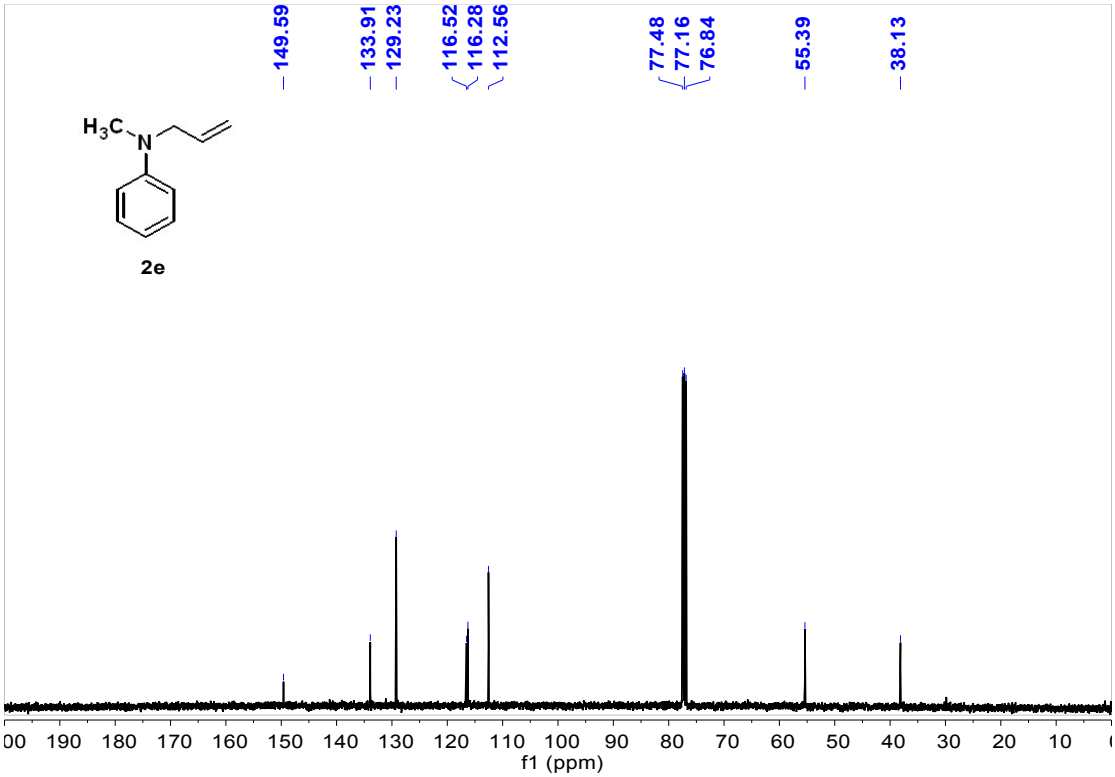
2.90
0.97
1.99
2.02
3.00
f1 (ppm)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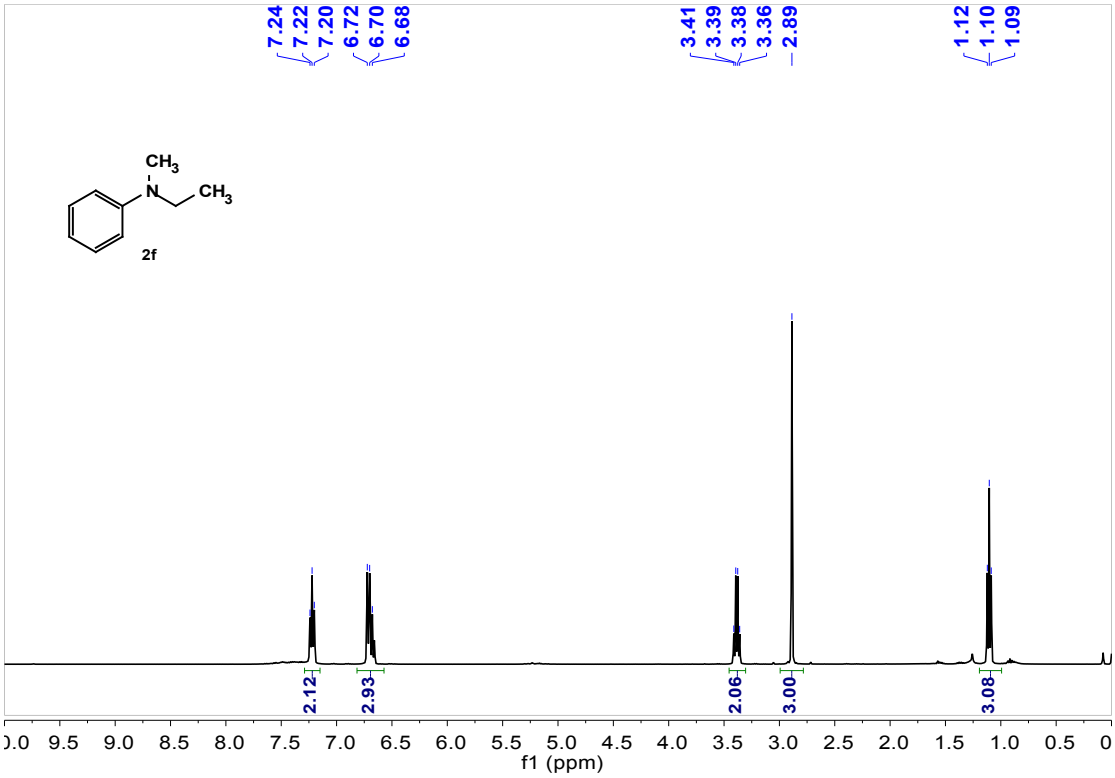
f1 (ppm)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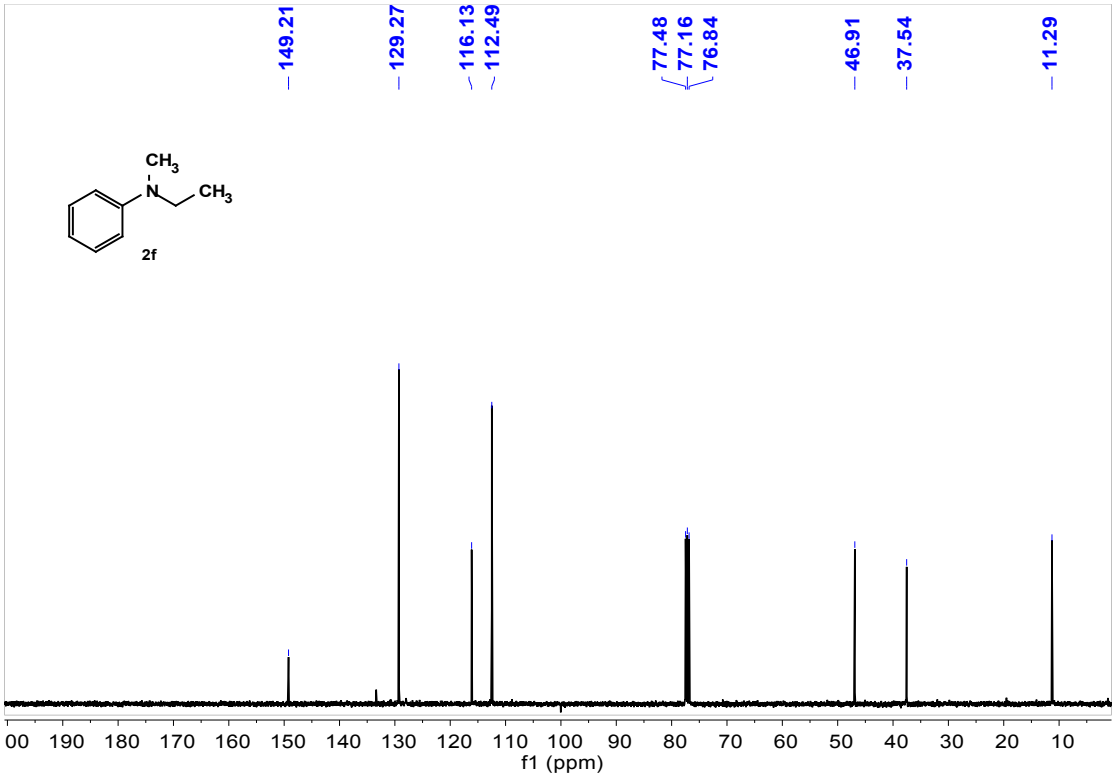
f1 (ppm)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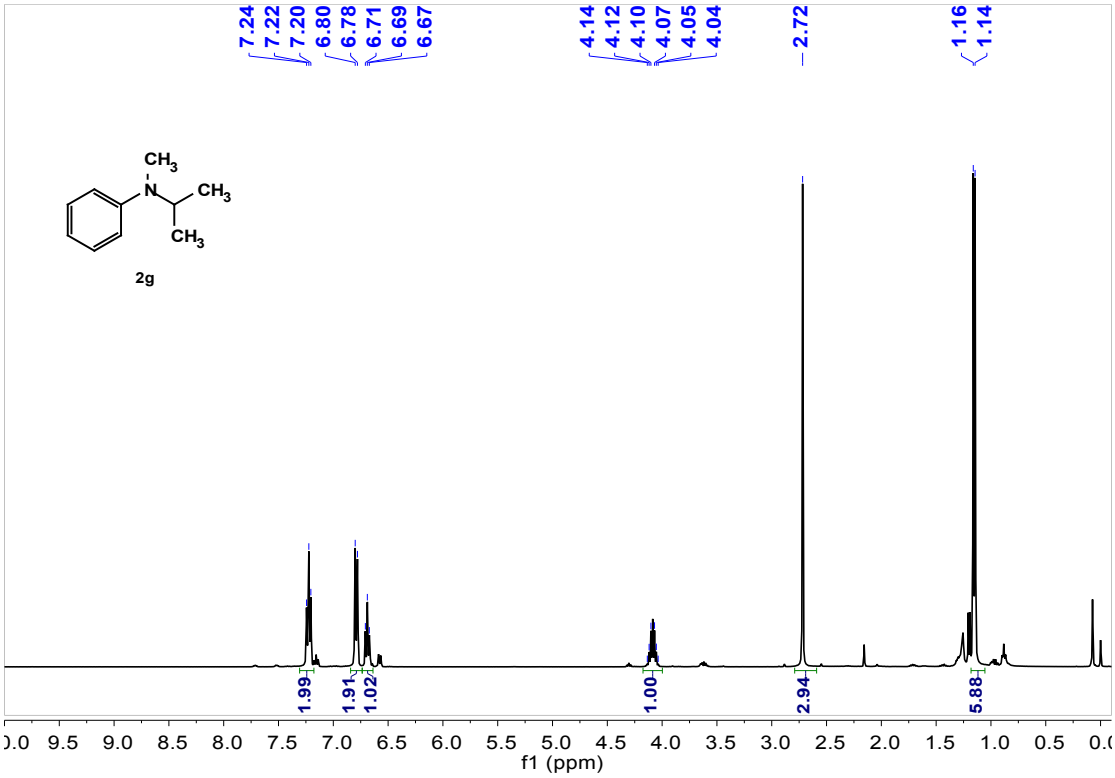
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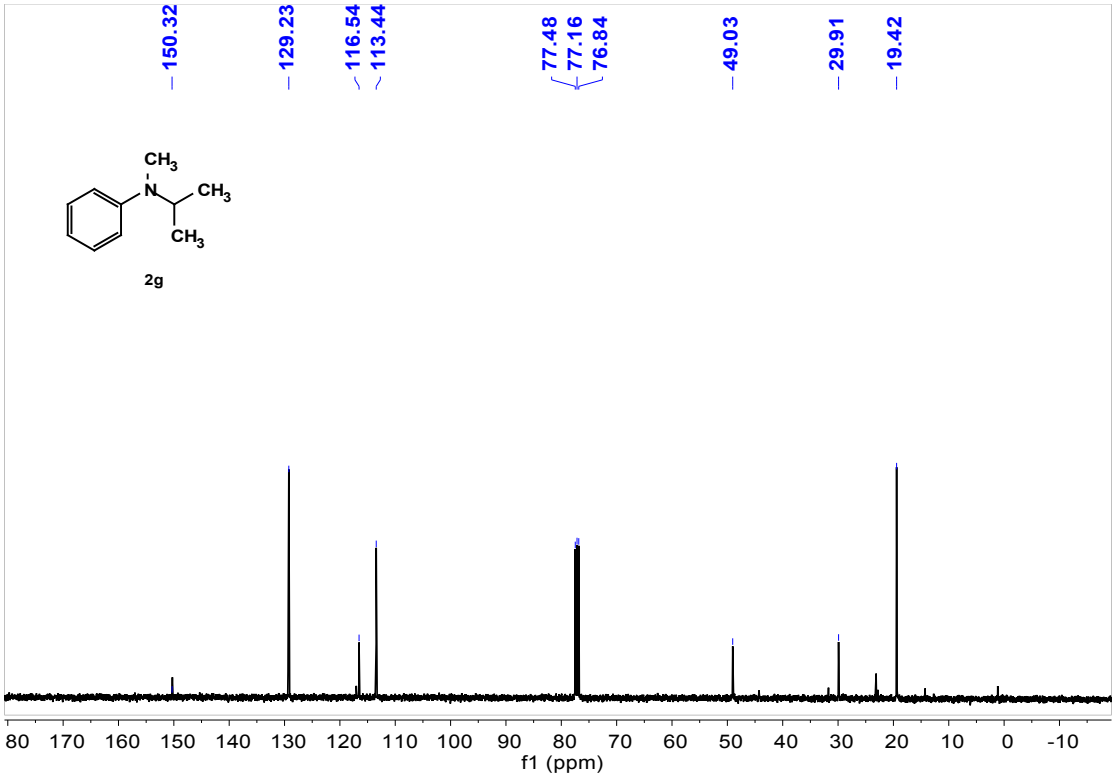
f1 (ppm)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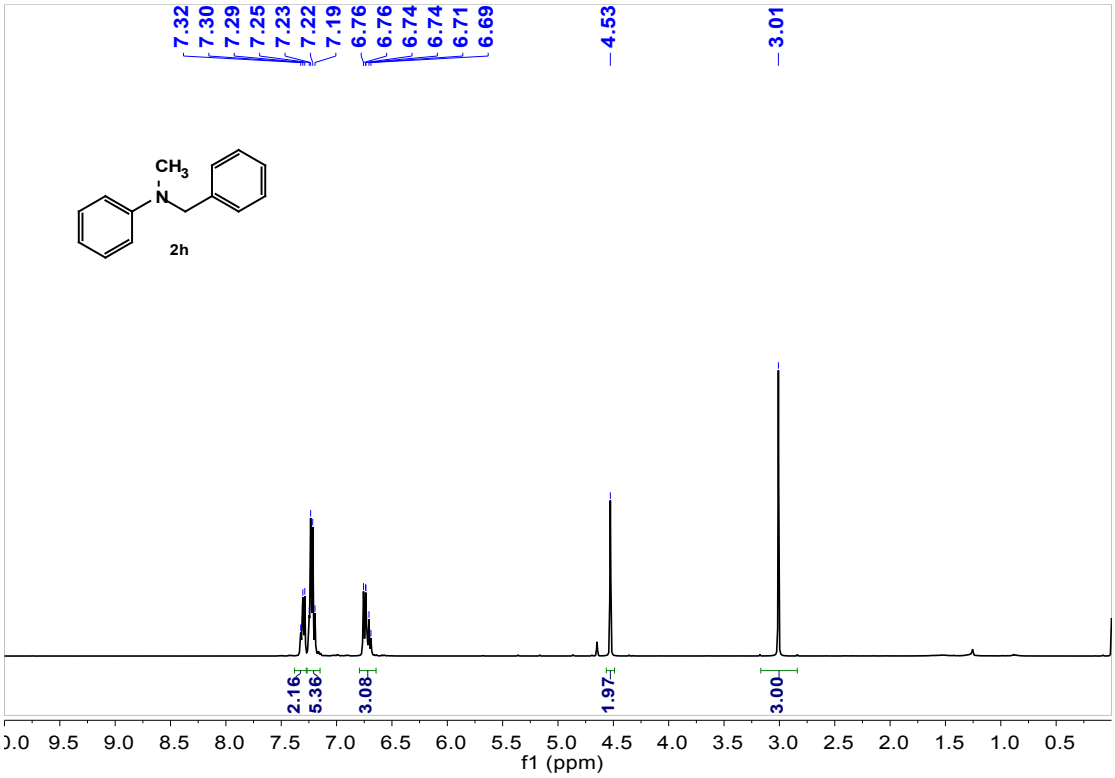
f1 (ppm)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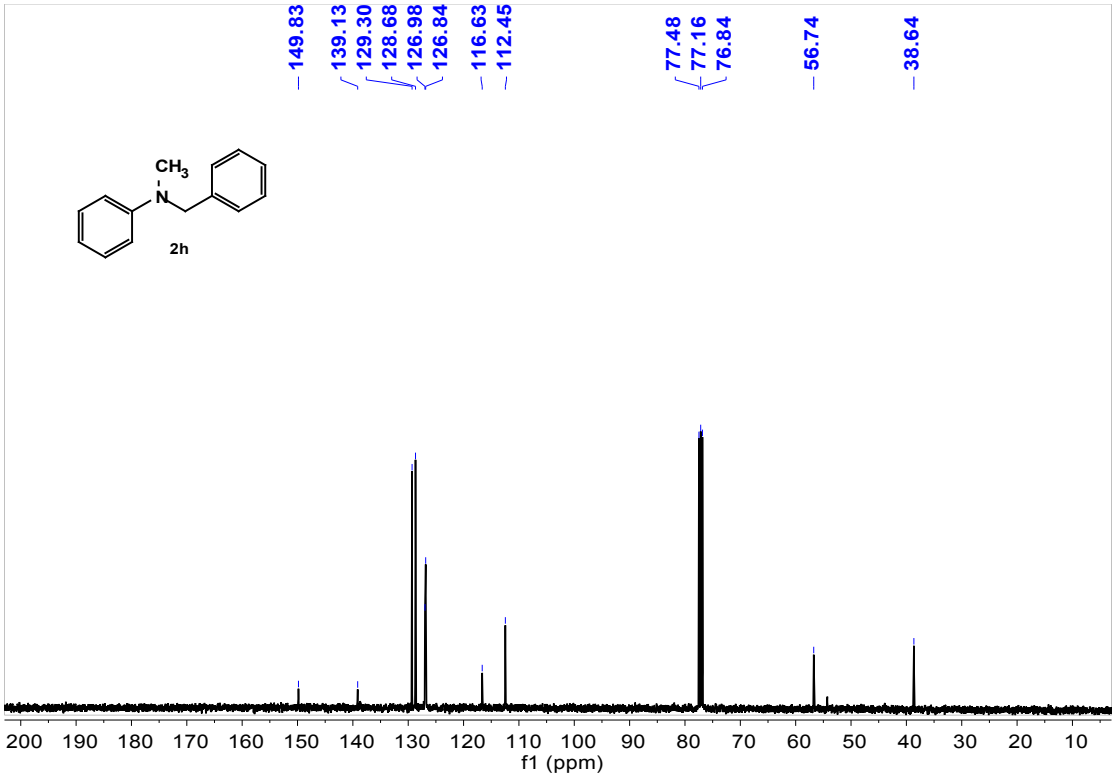
f1 (ppm)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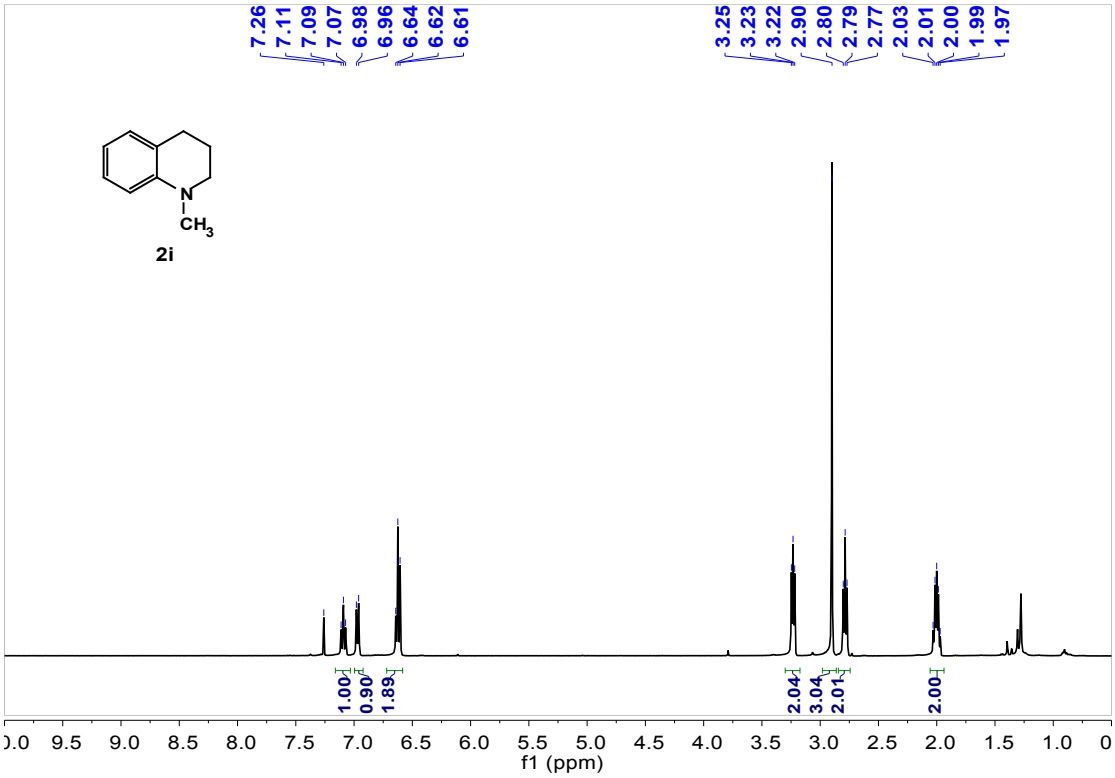
2h
f1 (ppm)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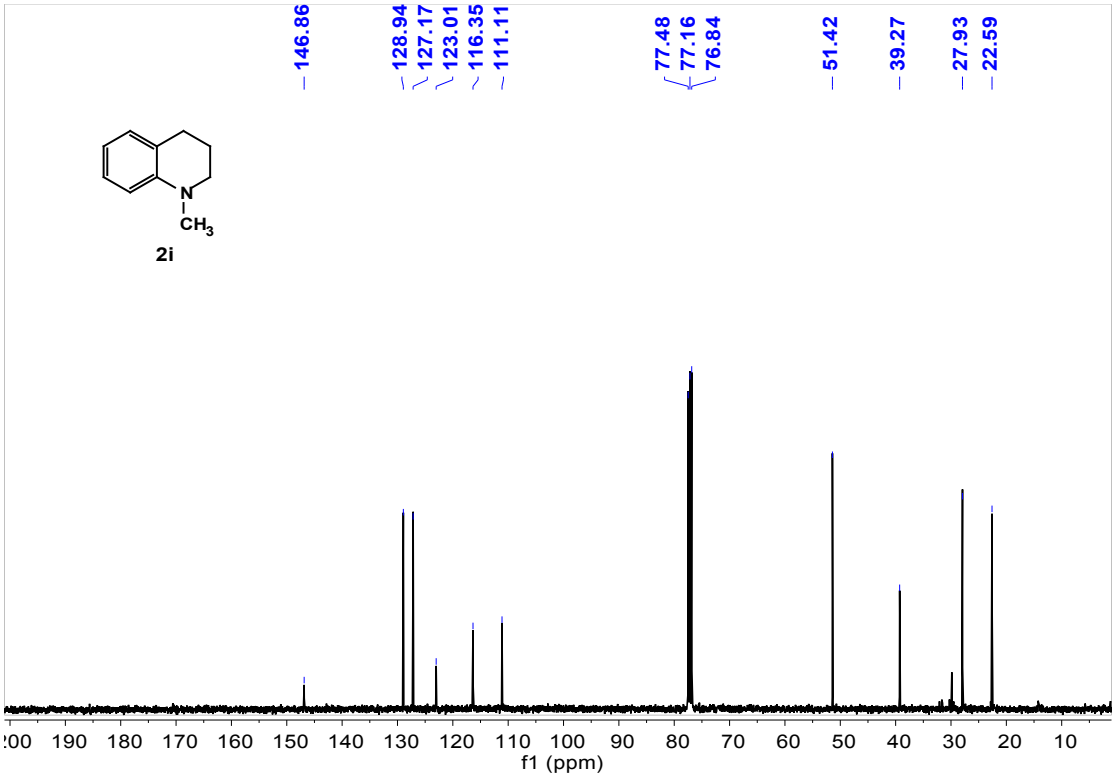
2i
f1 (ppm)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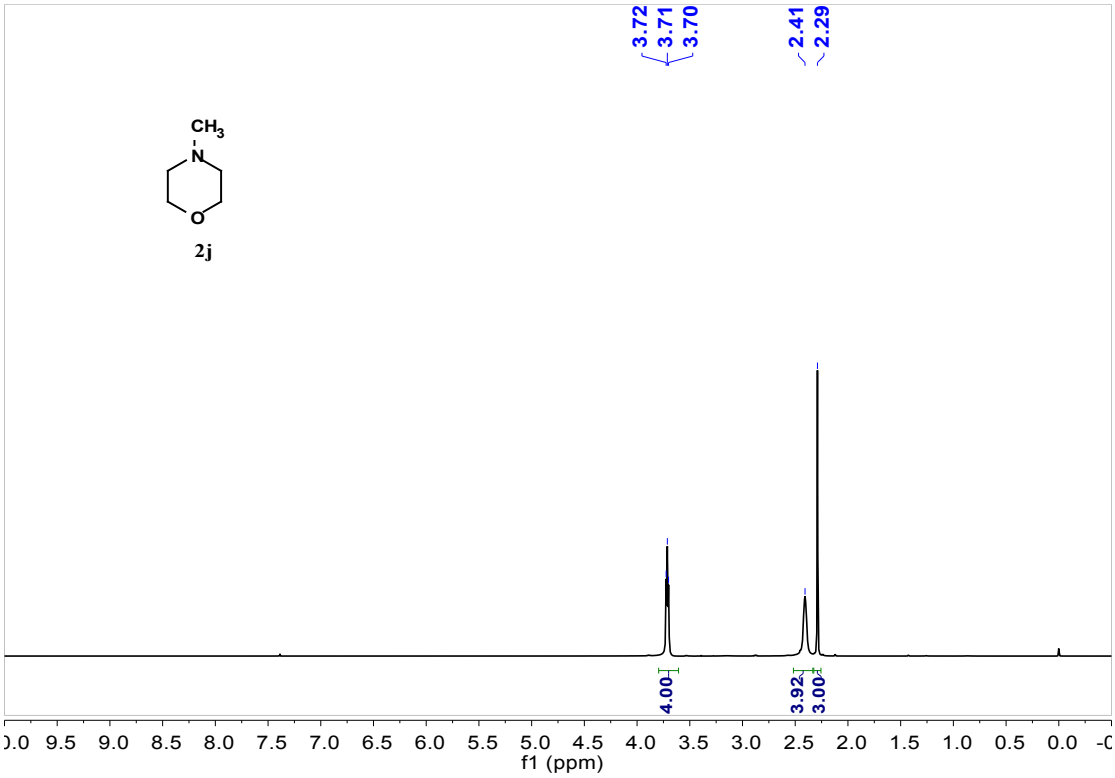
f1 (ppm)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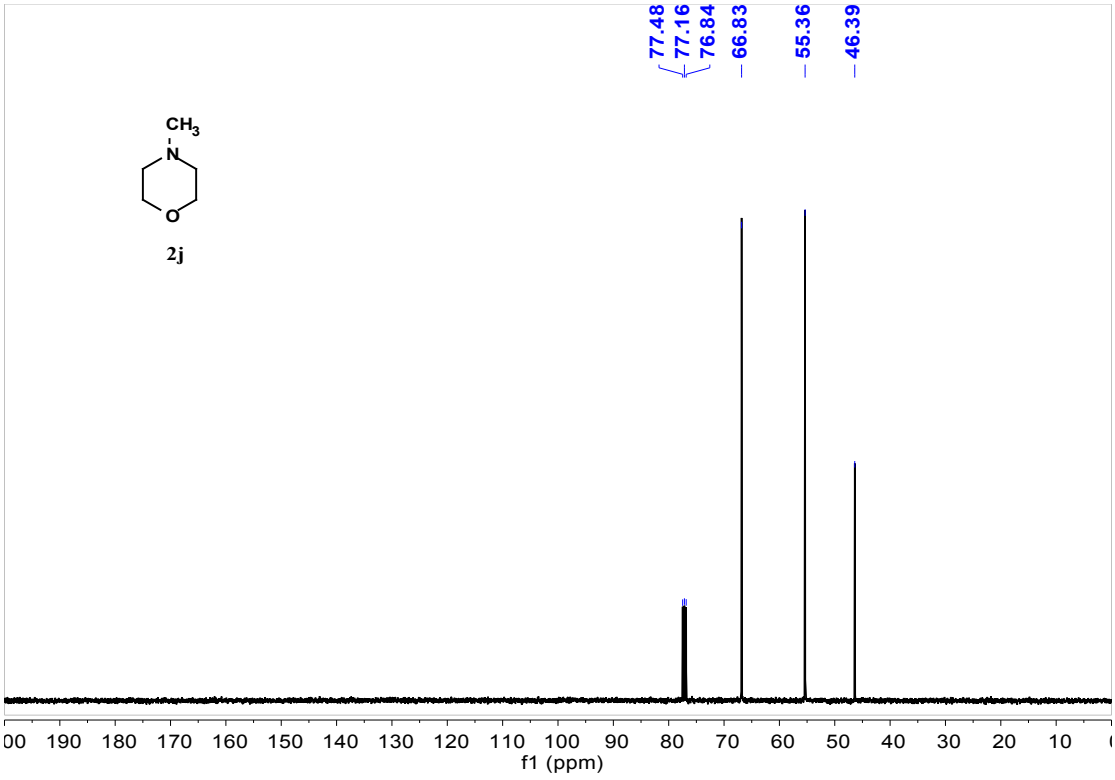
3.00
f1 (ppm)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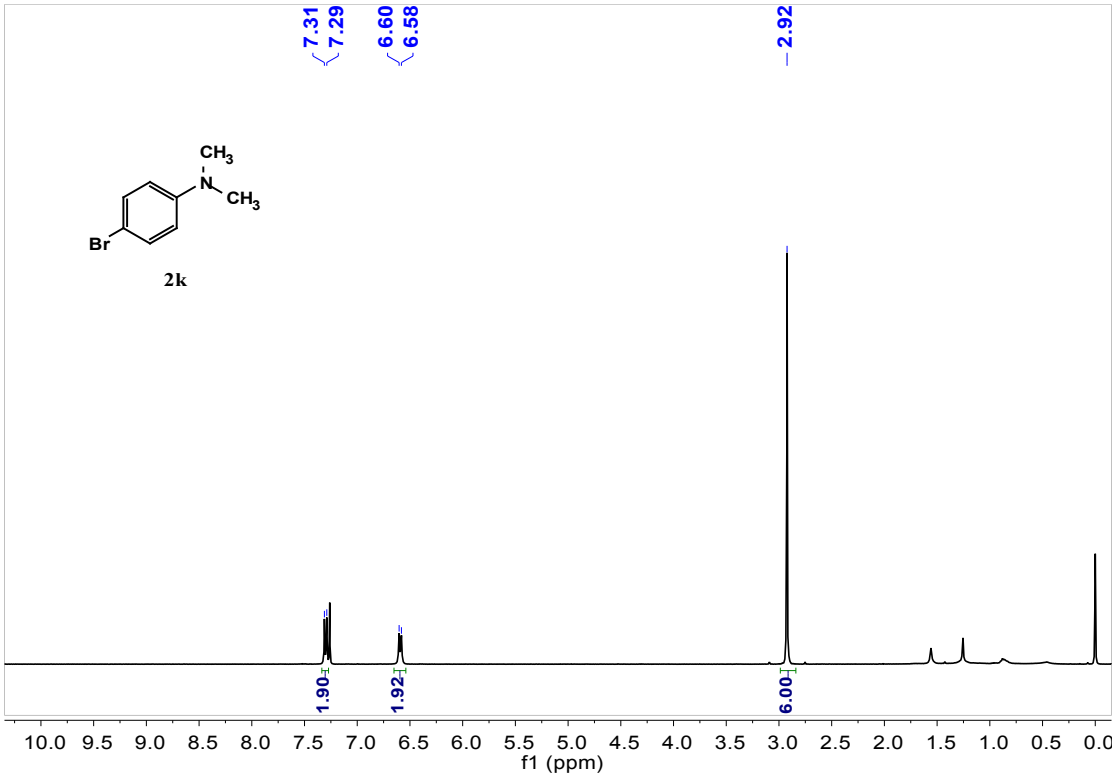
N
O
2j
f1 (ppm)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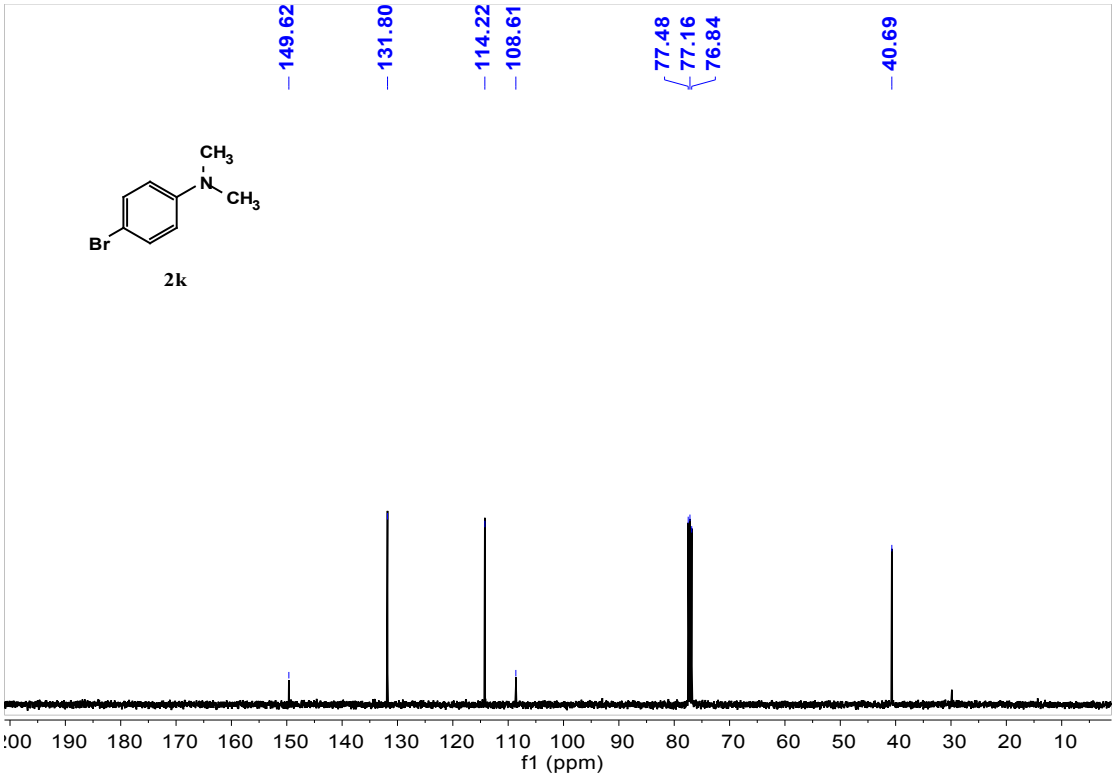
f1 (ppm)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40.69
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N
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Br
2k
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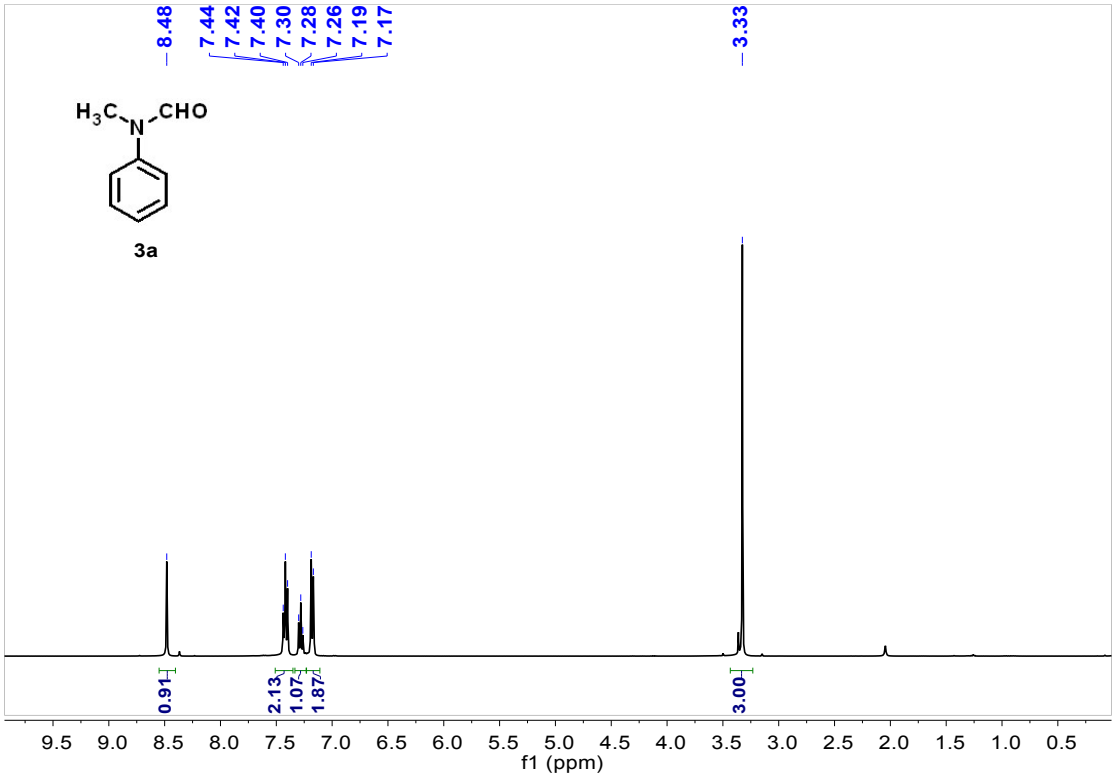
f1 (ppm)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7.30
7.28
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7.19
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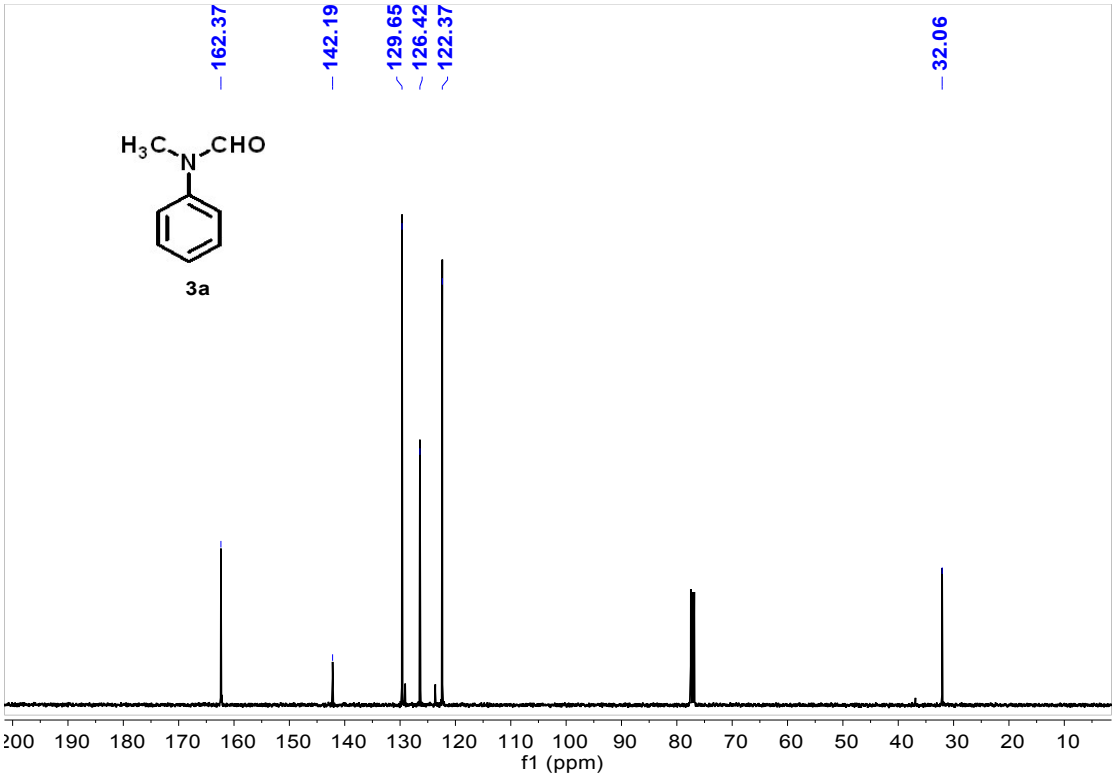
3.00
f1 (ppm)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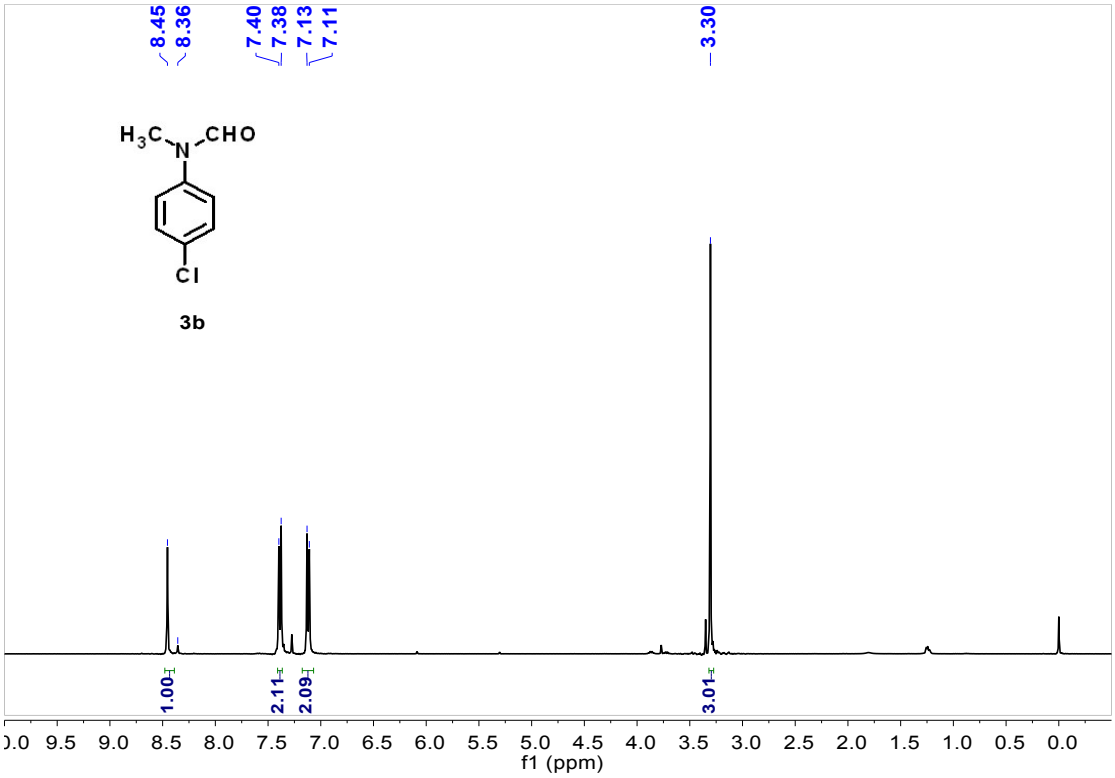
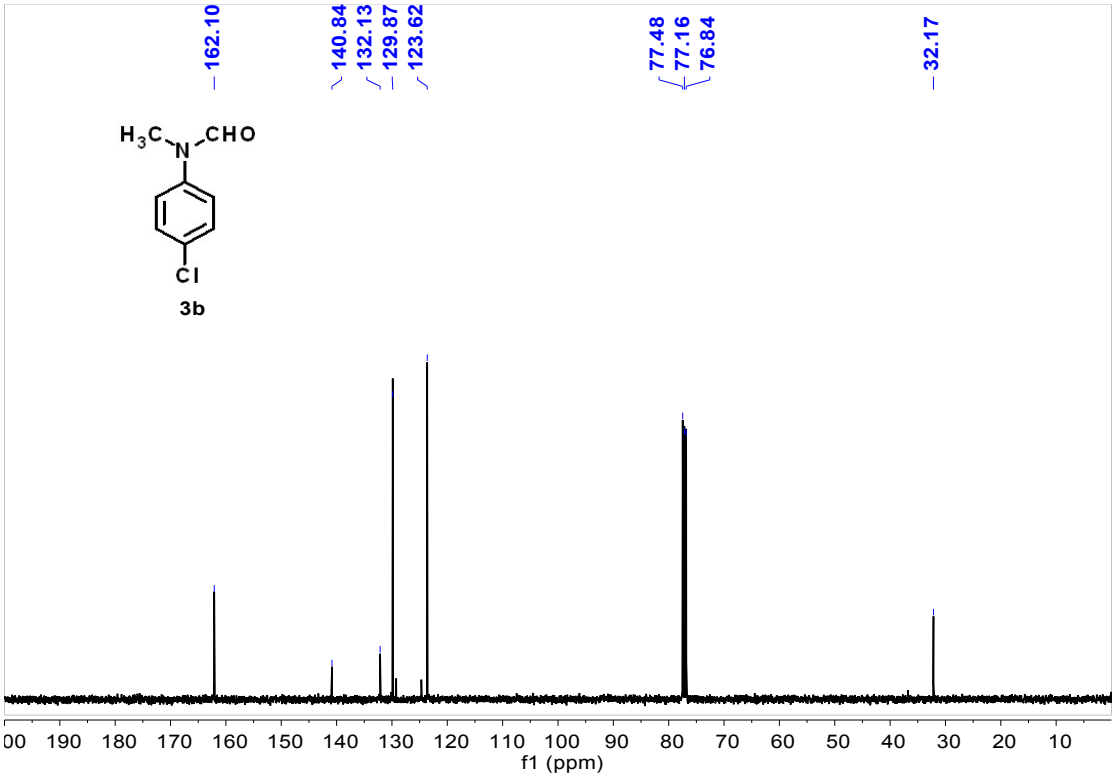
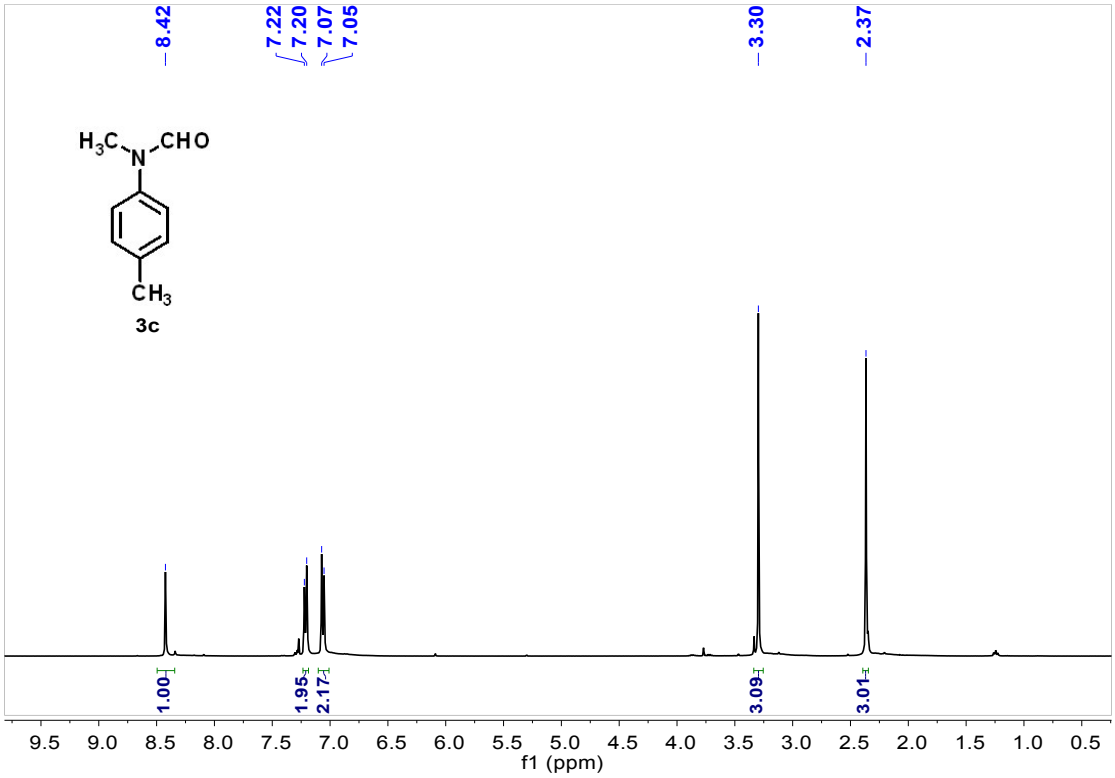
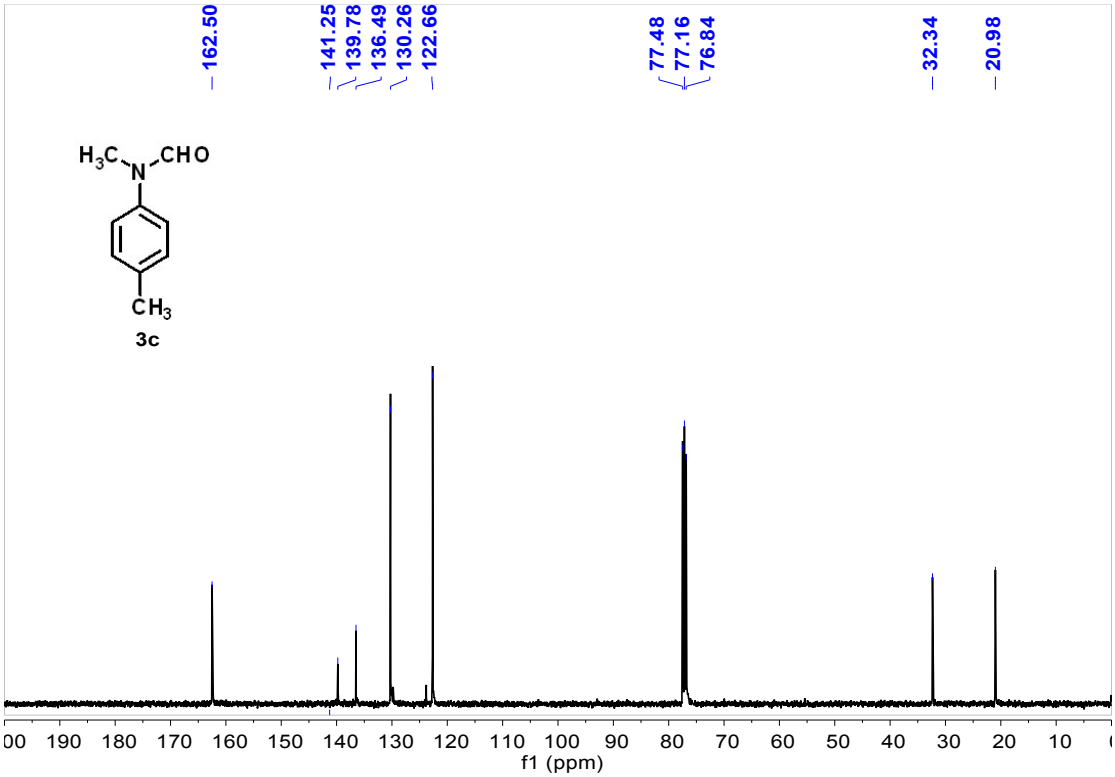
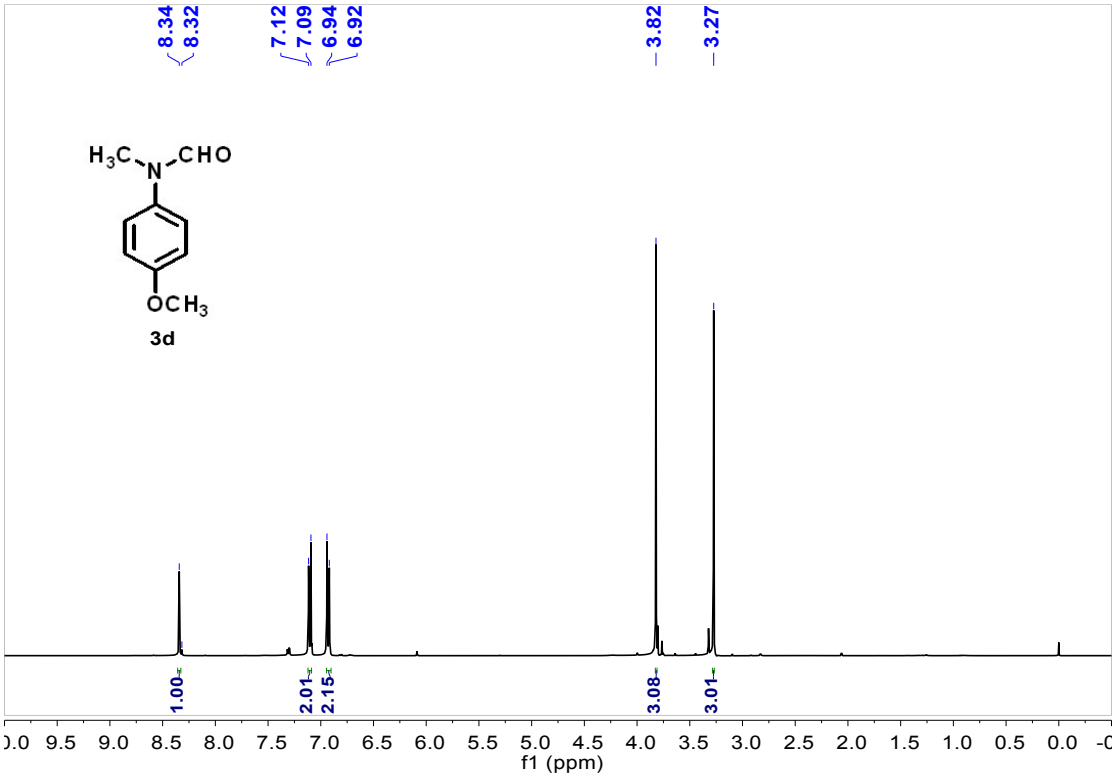
3a
f1 (ppm)

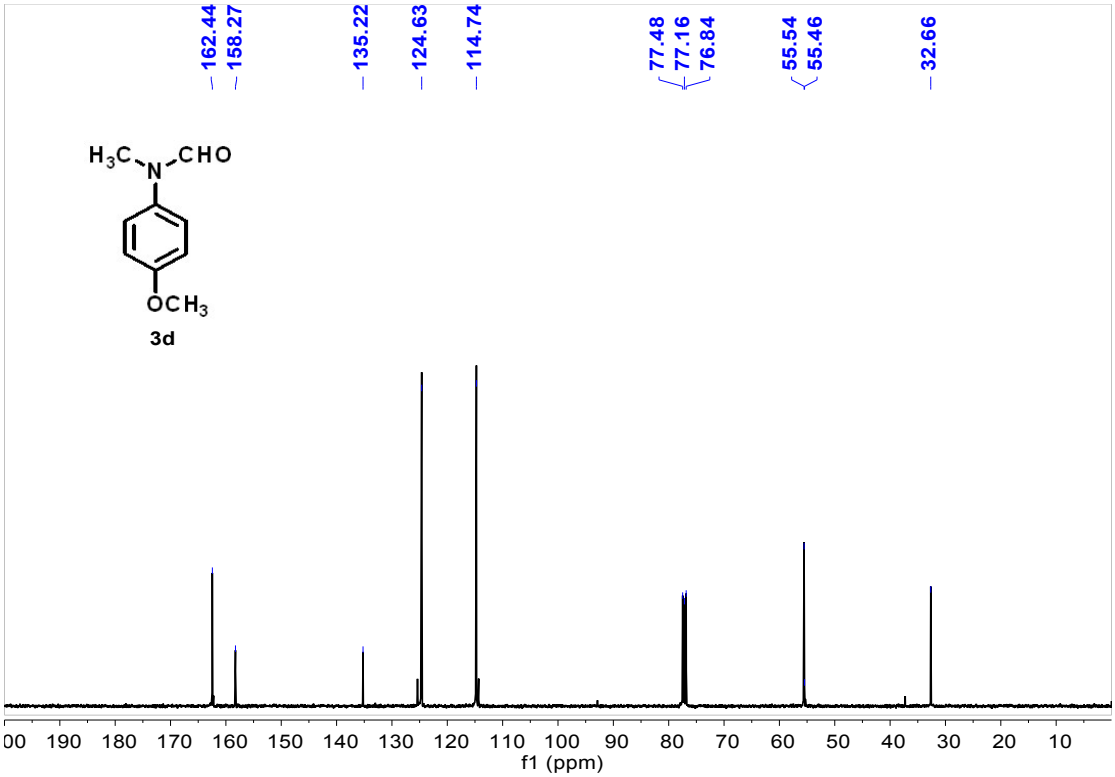

8.45
8.36
7.40
7.38
7.13
7.11
3.30
H3C
N
CHO
Cl
3b
1.00
2.11
2.09
3.01
f1 (ppm)

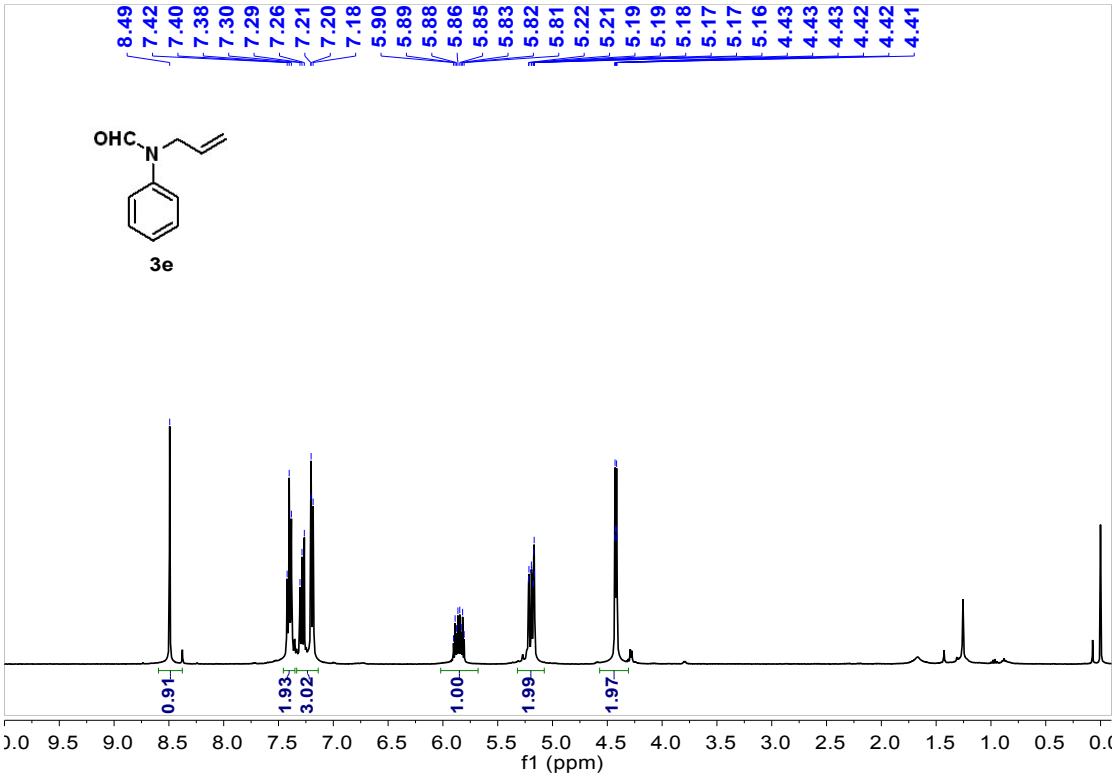

162.10
140.84
132.13
129.87
123.62
77.48
77.16
76.84
32.17
H3C
N
CHO
Cl
3b
f1 (ppm)

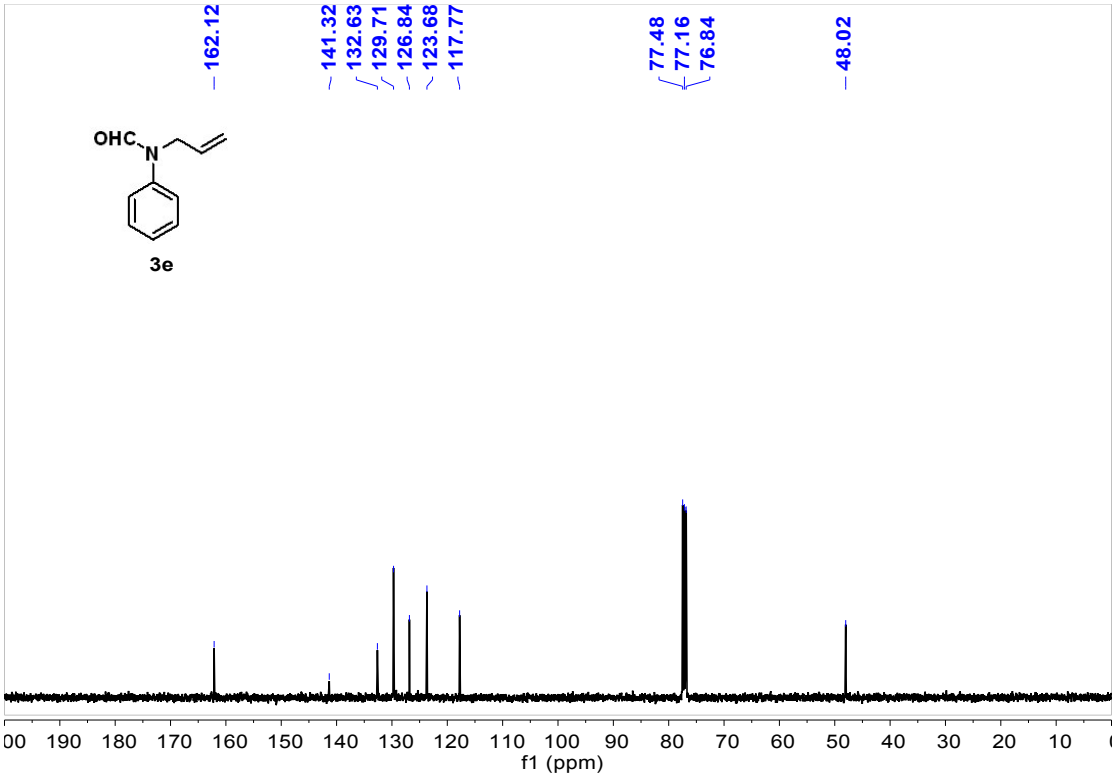

8.42
7.22
7.20
7.07
7.05
3.30
2.37
H3C
N
CHO
CH3
3c
1.00
1.95
2.17
3.09
3.01
f1 (ppm)

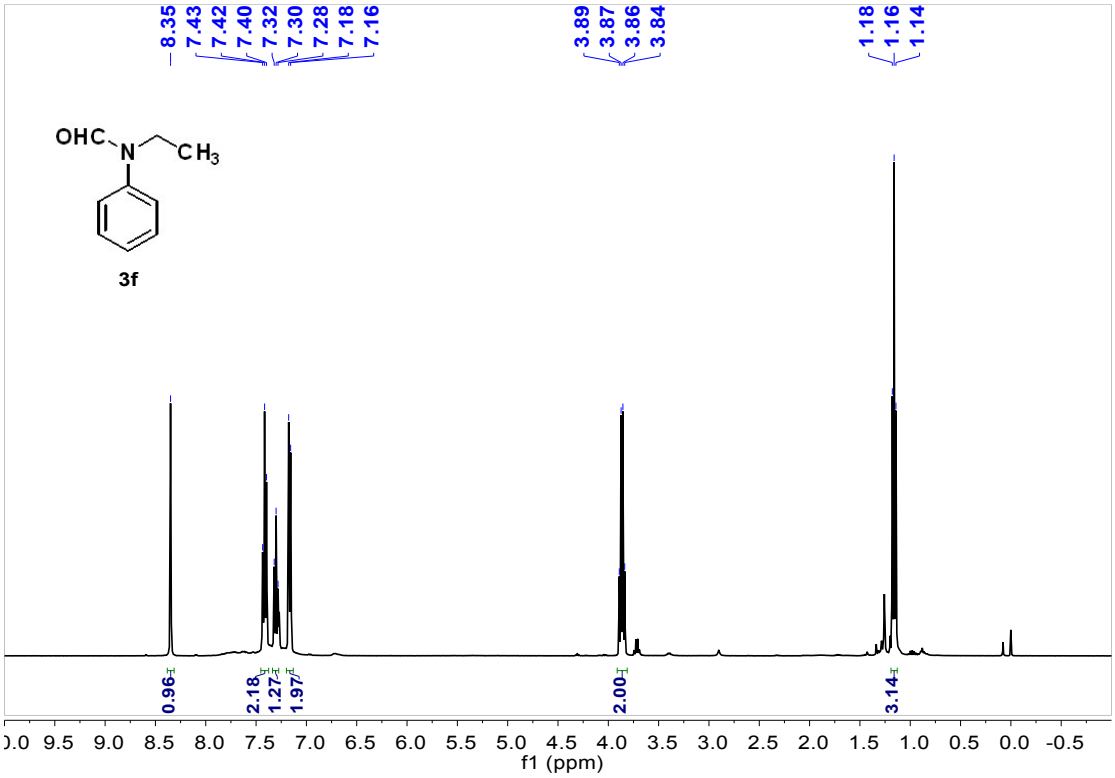

162.50
141.25
139.78
136.49
130.26
122.66
77.48
77.16
76.84
32.34
20.98
H3C
N
CHO
CH3
3c
f1 (ppm)

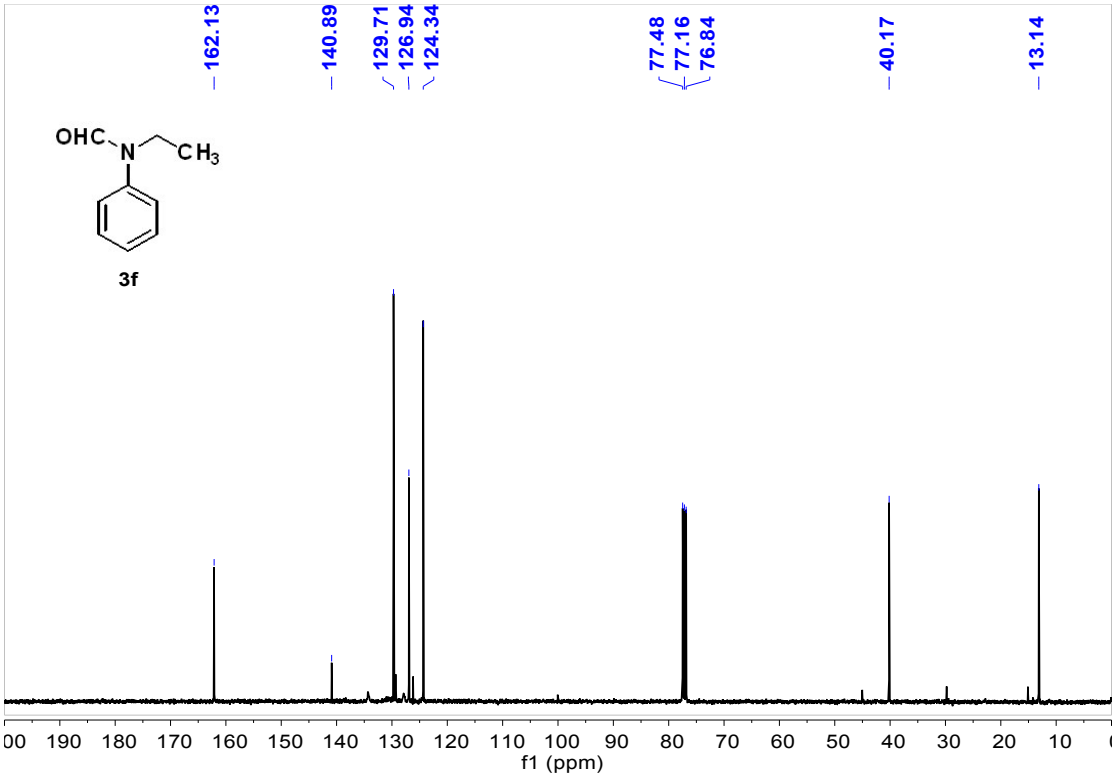

8.34
8.32
7.12
7.09
6.94
6.92
3.82
3.27
H3C
N
CHO
OCH3
3d
1.00
2.01
2.15
3.08
3.01
f1 (ppm)

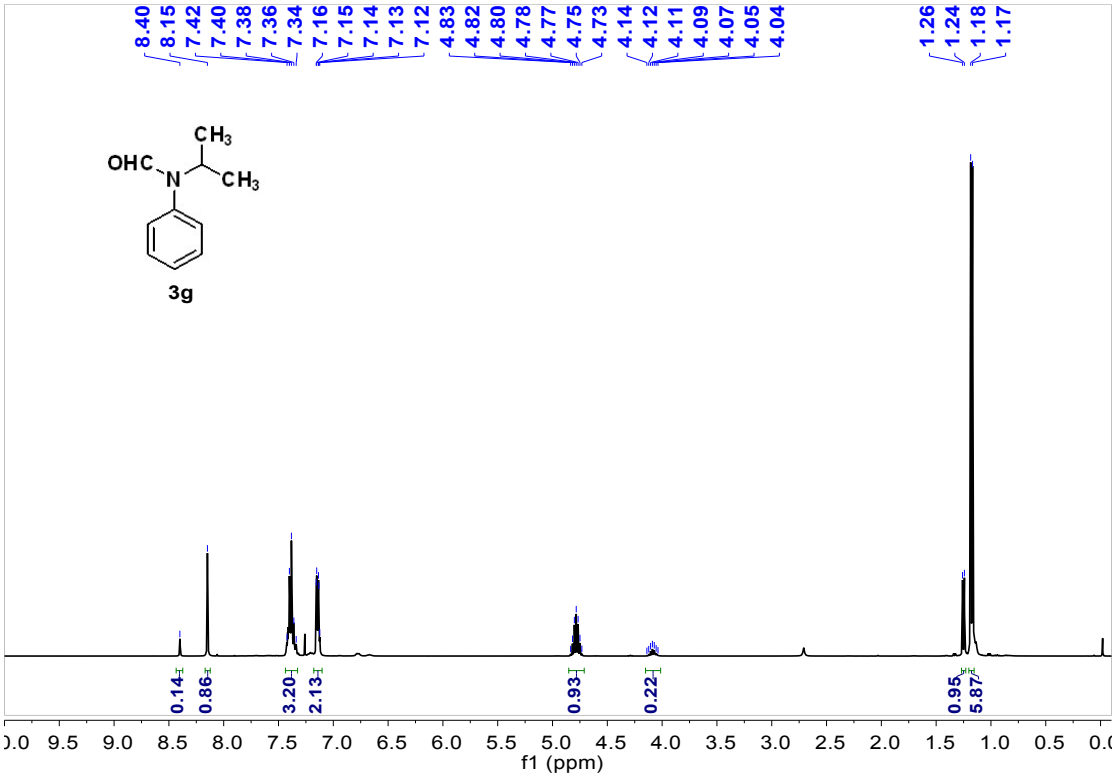

162.44
158.27
135.22
124.63
114.74
77.48
77.16
76.84
55.54
55.46
32.66
H3C
N
CHO
OCH3
3d
f1 (ppm)

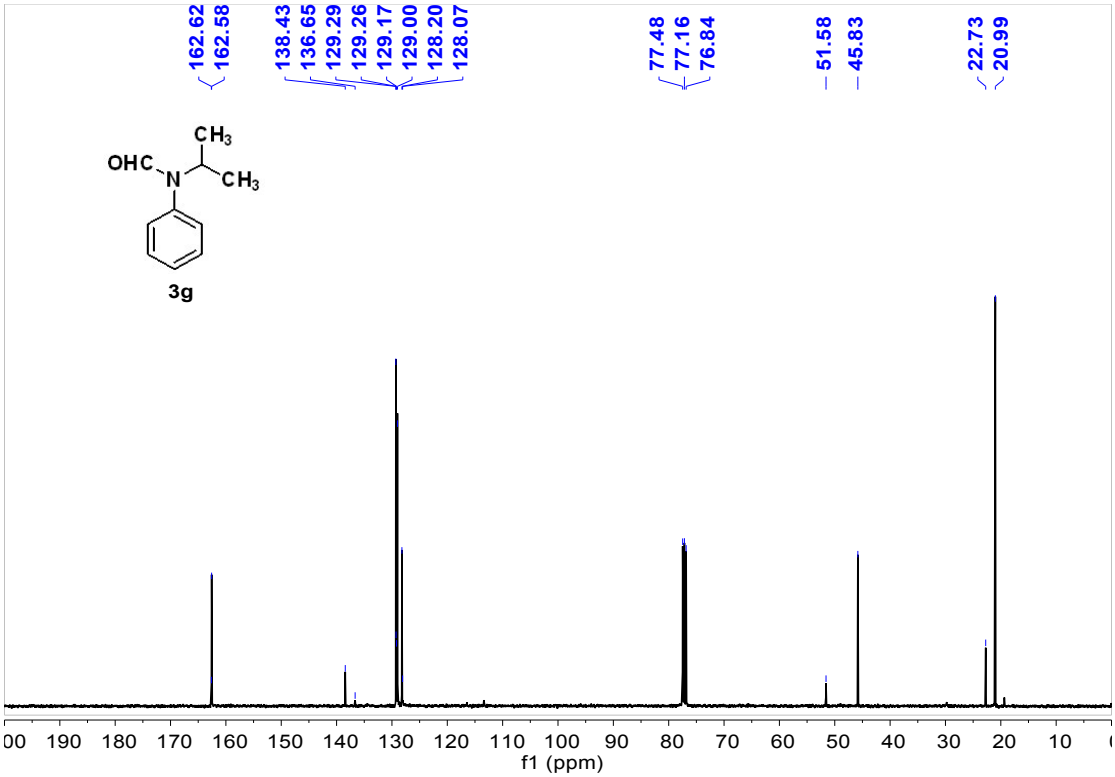

8.49
7.42
7.40
7.38
7.30
7.29
7.26
7.21
7.20
7.18
5.90
5.89
5.88
5.86
5.85
5.83
5.82
5.81
5.22
5.21
5.19
5.19
5.18
5.17
5.17
5.16
4.43
4.43
4.42
4.42
4.41
OHC
N
3e
0.91
1.93
3.02
1.00
1.99
1.97
f1 (ppm)

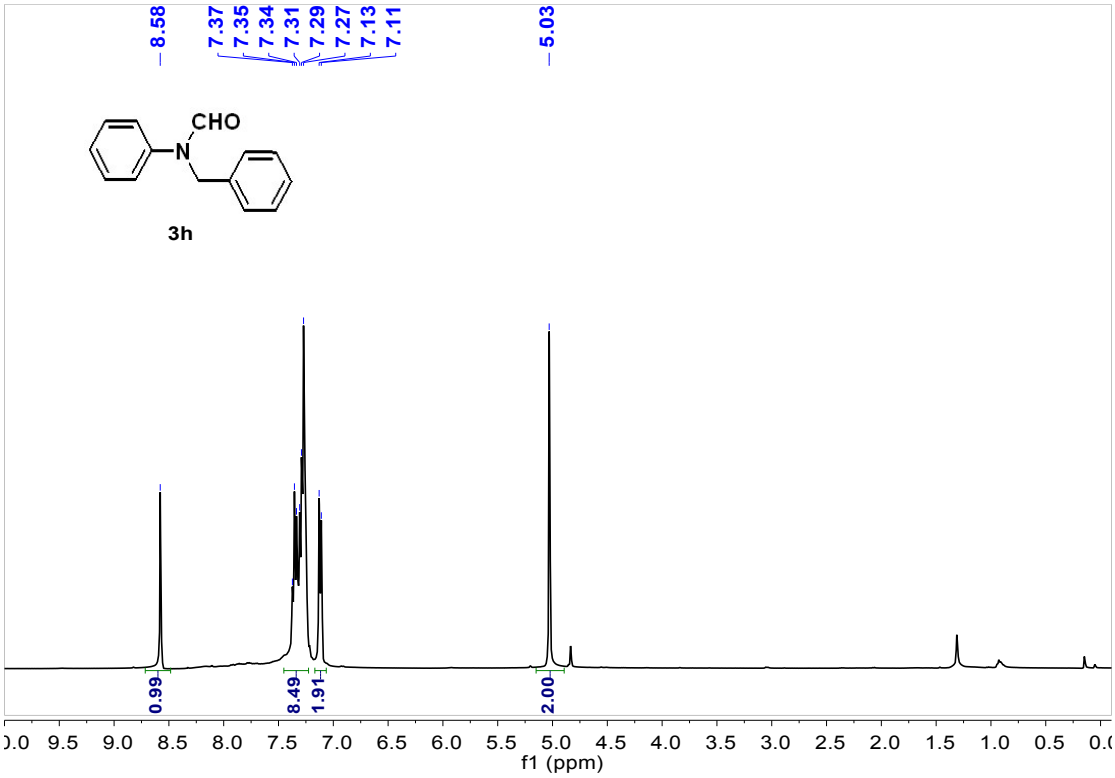

162.12
141.32
132.63
129.71
126.84
123.68
117.77
77.48
77.16
76.84
48.02
OHC
N
3e
f1 (ppm)

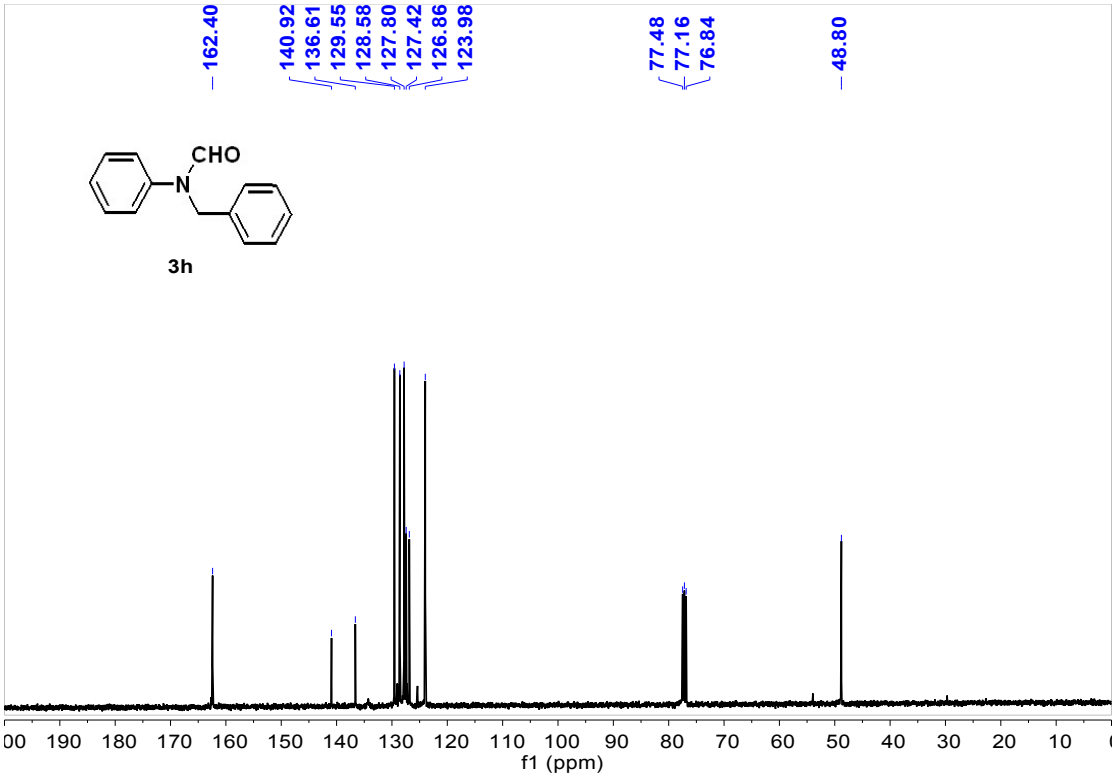

8.35
7.43
7.42
7.40
7.32
7.30
7.28
7.18
7.16
3.89
3.87
3.86
3.84
1.18
1.16
1.14
OHC
N
CH3
3f
0.96
2.18
1.27
1.97
2.00
3.14
f1 (ppm)

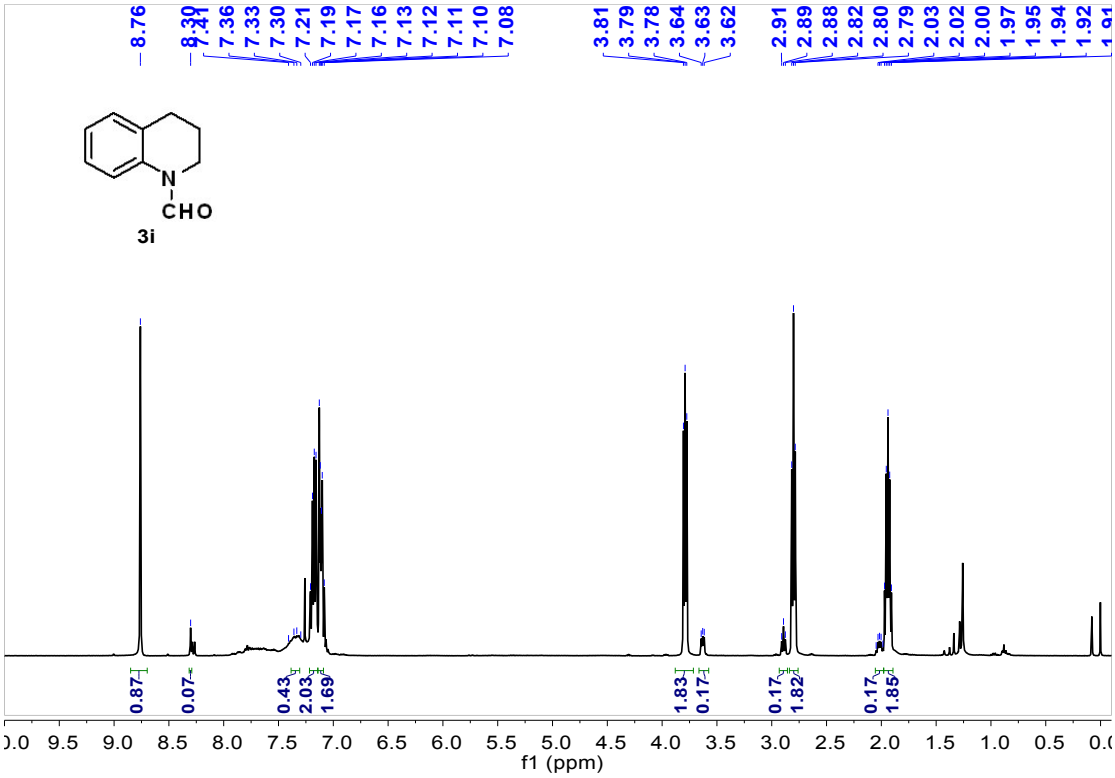

162.13
140.89
129.71
126.94
124.34
77.48
77.16
76.84
40.17
13.14
OHC
N
CH3
3f
f1 (ppm)

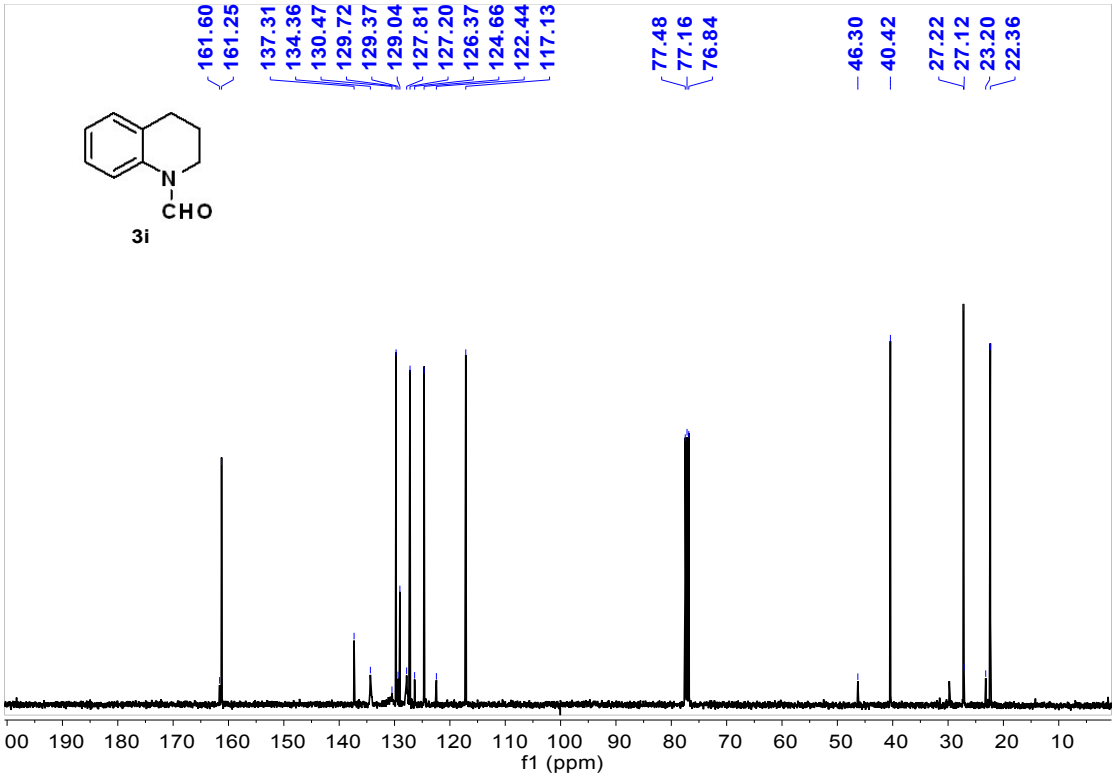

8.40
8.15
7.42
7.40
7.38
7.36
7.34
7.16
7.15
7.14
7.13
7.12
4.83
4.82
4.80
4.78
4.77
4.75
4.73
4.14
4.12
4.11
4.09
4.07
4.05
4.04
1.26
1.24
1.18
1.17
CH3
OHC
N
CH3
3g
0.14
0.86
3.20
2.13
0.93
0.22
0.95
5.87
f1 (ppm)

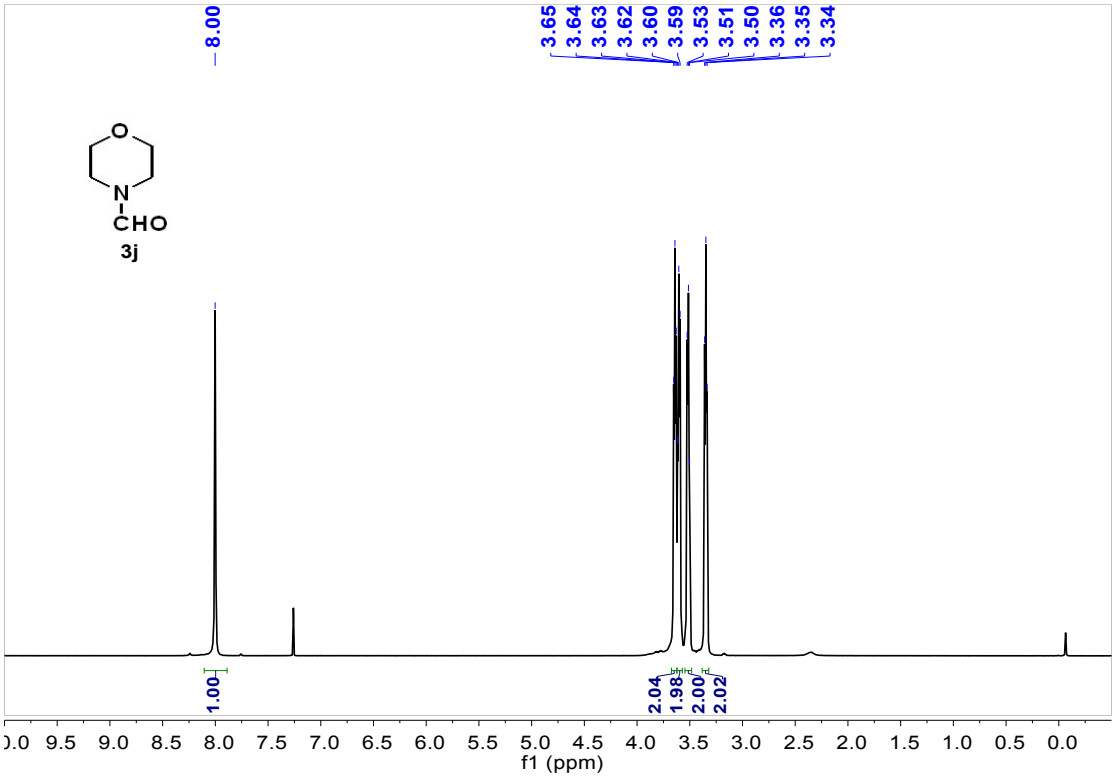

162.62
162.58
138.43
136.65
129.29
129.26
129.17
129.00
128.20
128.07
77.48
77.16
76.84
51.58
45.83
22.73
20.99
CH3
OHC
N
CH3
3g
f1 (ppm)

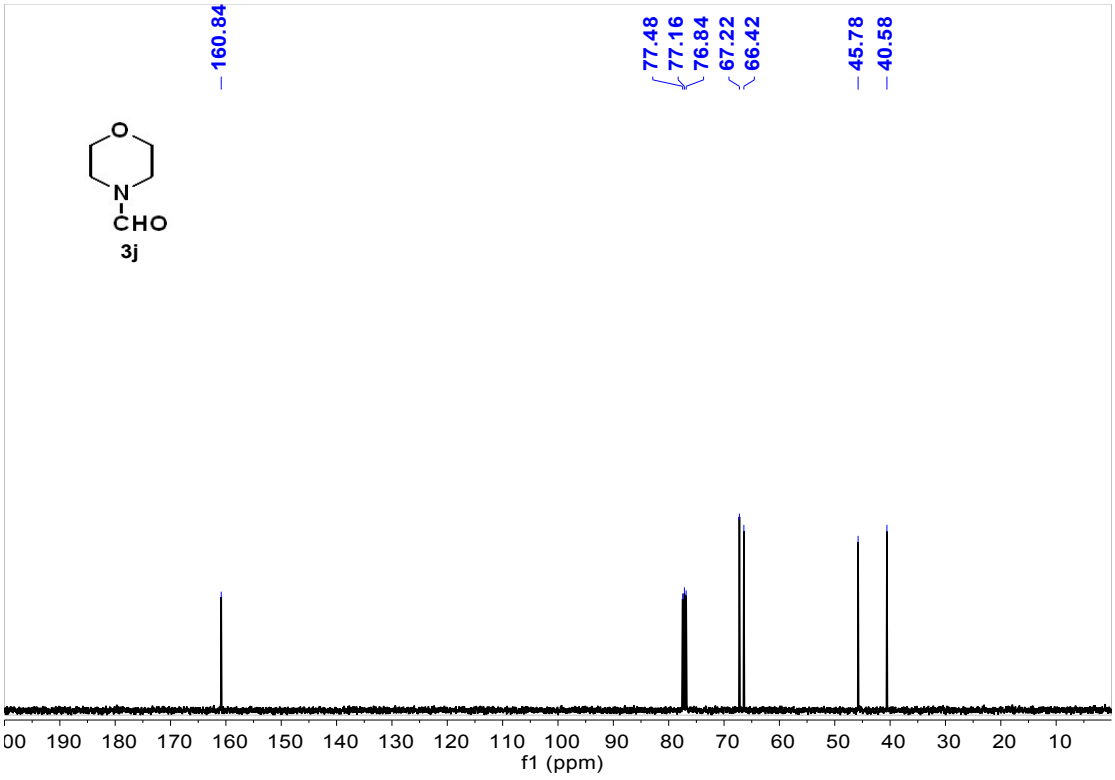

8.58
7.37
7.35
7.34
7.31
7.29
7.27
7.13
7.11
5.03
CHO
N
3h
0.99
8.49
1.91
2.00
f1 (ppm)

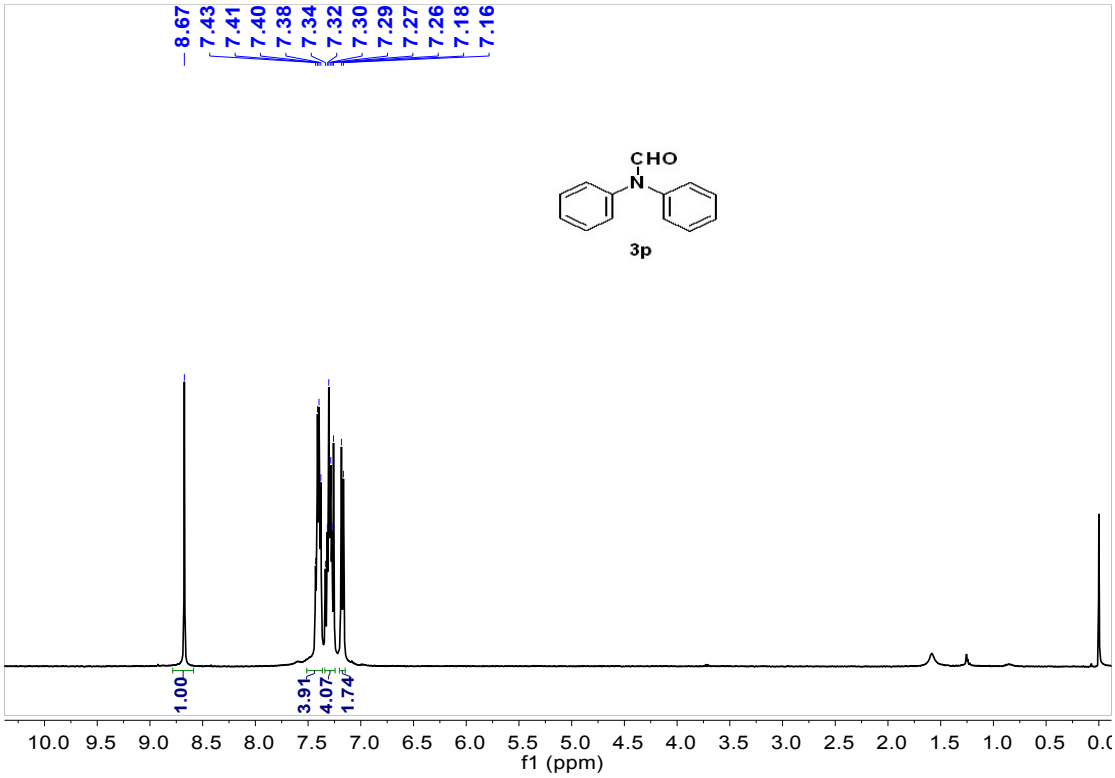

162.40
140.92
136.61
129.55
128.58
127.80
127.42
126.86
123.98
77.48
77.16
76.84
48.80
CHO
N
3h
f1 (ppm)

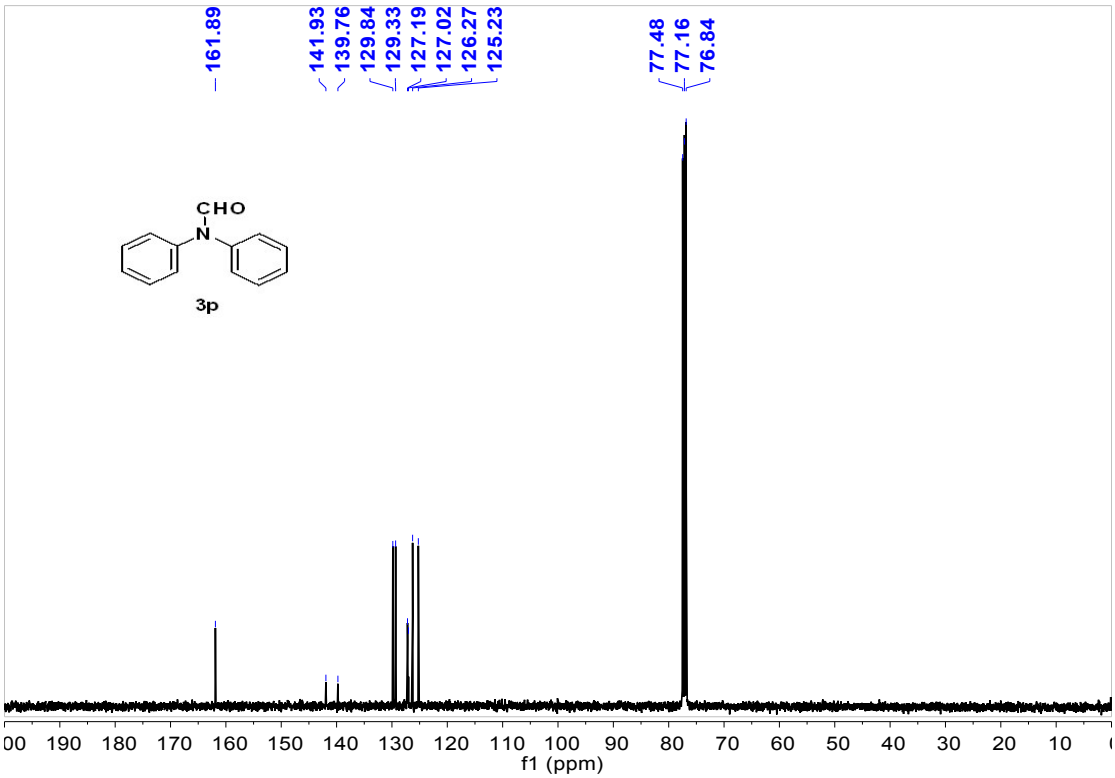

8.76
8.30
7.41
7.36
7.33
7.30
7.21
7.19
7.17
7.16
7.13
7.12
7.11
7.10
7.08
3.81
3.79
3.78
3.64
3.63
3.62
2.91
2.89
2.88
2.82
2.80
2.79
2.03
2.02
2.00
1.97
1.95
1.94
1.92
N
CHO
3i
0.87
0.07
0.43
2.03
1.69
1.83
0.17
0.17
1.82
0.17
1.85
f1 (ppm)

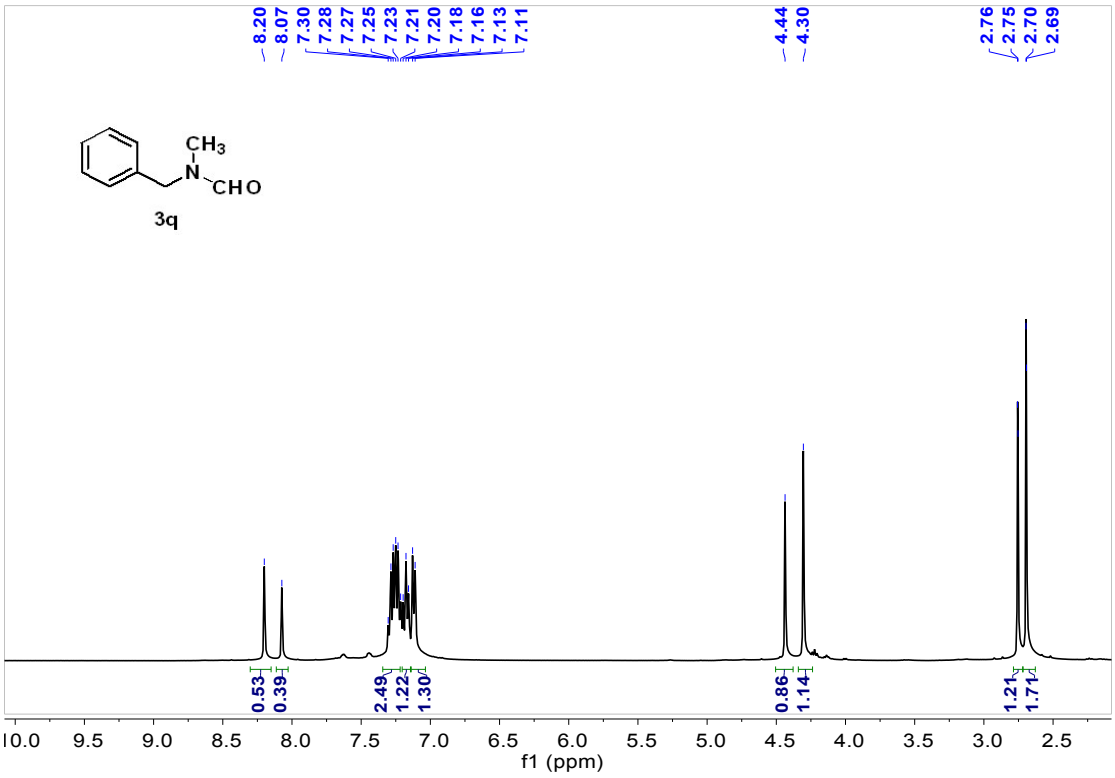

161.60
161.25
137.31
134.36
130.47
129.72
129.37
129.04
127.81
127.20
126.37
124.66
122.44
117.13
77.48
77.16
76.84
46.30
40.42
27.22
27.12
23.20
22.36
N
CHO
3i
f1 (ppm)

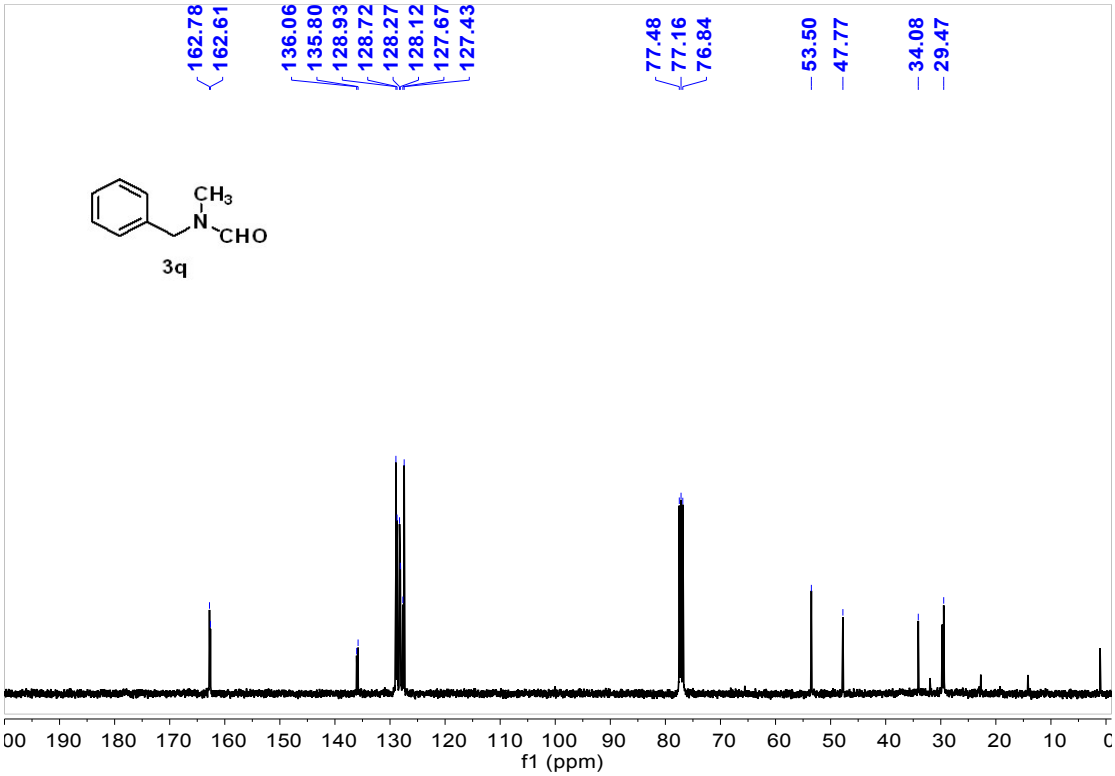

8.00
3.65
3.64
3.63
3.62
3.60
3.59
3.53
3.51
3.50
3.36
3.35
3.34
O
N
CHO
3j
1.00
2.04
1.98
2.00
2.02
f1 (ppm)

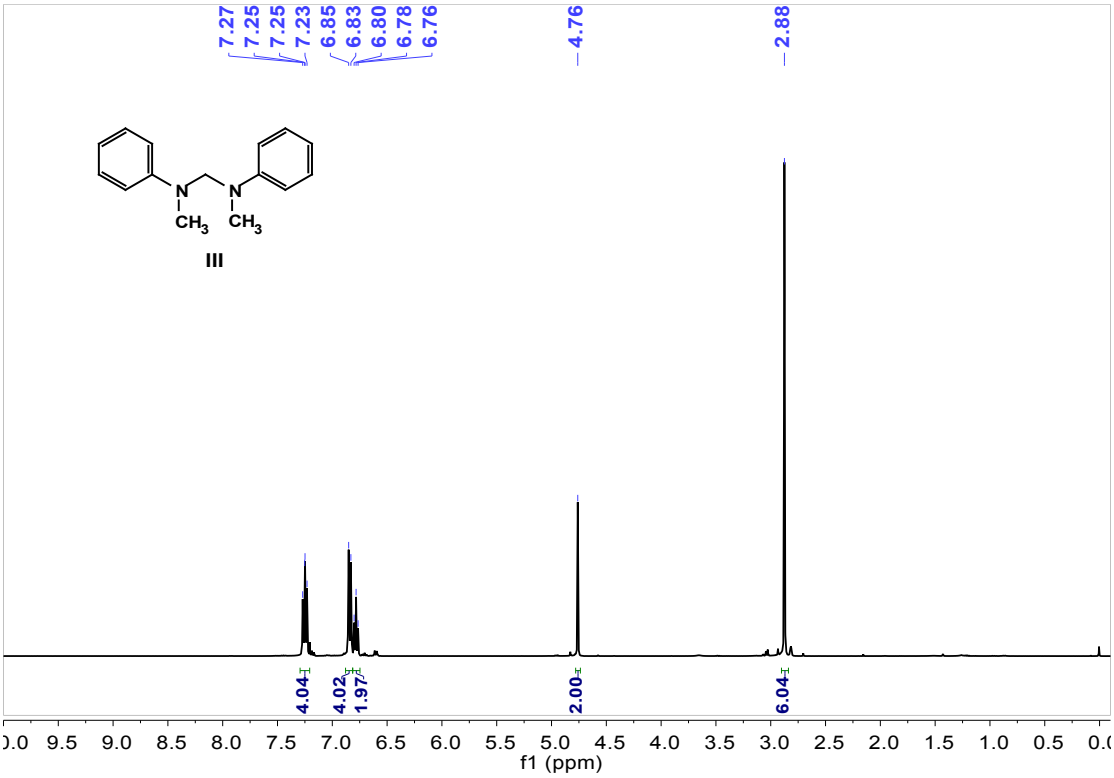

160.84
77.48
77.16
76.84
67.22
66.42
45.78
40.58
O
N
CHO
3j
f1 (ppm)

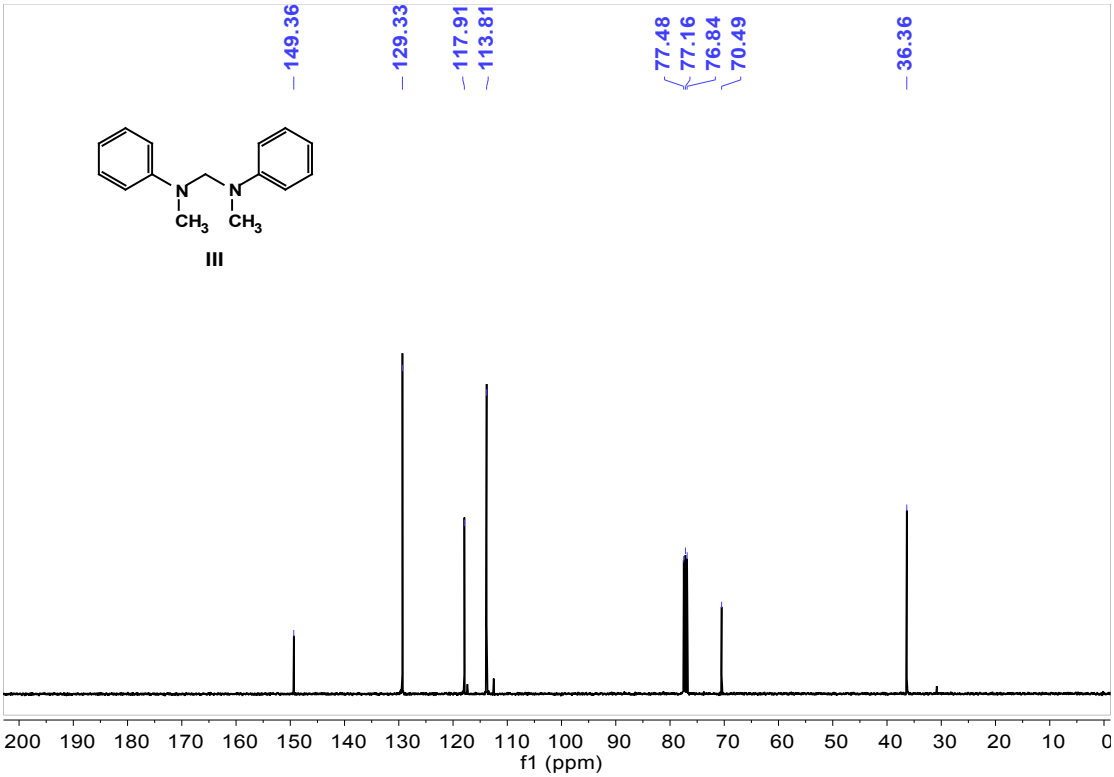

8.67
7.43
7.41
7.40
7.38
7.34
7.32
7.30
7.29
7.27
7.26
7.18
7.16
CHO
N
3p
1.00
3.91
4.07
1.74
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)


161.89
141.93
139.76
129.84
129.33
127.19
127.02
126.27
125.23
77.48
77.16
76.84
CHO
N
3p
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)


8.20
8.07
7.30
7.28
7.27
7.25
7.23
7.21
7.20
7.18
7.16
7.13
7.11
4.44
4.30
2.76
2.75
2.70
2.69
CH3
N
CHO
3q
0.53
0.39
2.49
1.22
1.30
0.86
1.14
1.21
1.71
f1 (ppm)


162.78
162.61
136.06
135.80
128.93
128.72
128.27
128.12
127.67
127.43
77.48
77.16
76.84
53.50
47.77
34.08
29.47
CH3
N
CHO
3q
f1 (ppm)


7.27
7.25
7.25
7.23
6.85
6.83
6.80
6.78
6.76
4.76
2.88
N
CH3
N
CH3
III
4.04
4.02
1.97
2.00
6.04
f1 (ppm)


149.36
129.33
117.91
113.81
77.48
77.16
76.84
70.49
36.36
N
CH3
N
CH3
III
f1 (ppm)