Base Promoted Nitrile-Alkyne Domino-Type Cyclization: A General Method to Trisubstituted Imidazoles

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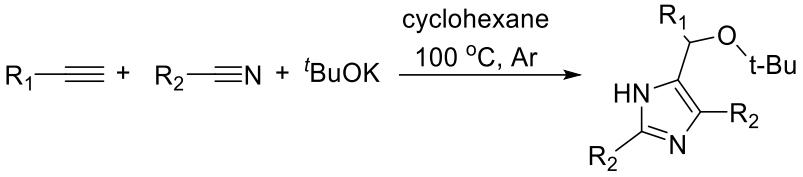
1. General Remarks

All commercially available organic compounds were purchased from adamas-beta, Alfa Asar, and accelachem in China. Unless otherwise noted, reactions were carried out under Ar atmosphere.

For Column chromatography, 200-300 mesh silica gel was employed. Analytical TLC was performed with silica gel GF254 plates. ^{1}H NMR (400 MHz) and ^{13}C NMR (100 MHz) were recorded in $CDCl_3$ using TMS as internal standard. All new products were further characterized by high resolution mass spectra (HRMS); copies of their ^{1}H NMR and ^{13}C NMR spectra are provided. All solvents (AR) were used directly. All reactions are heated with oil bath.

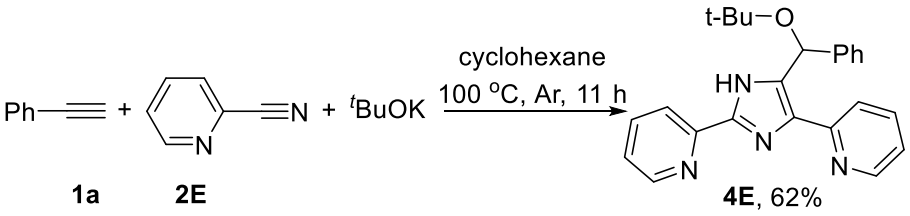
2. General Experimental Procedure

i) General experimental procedure for 0.2 mmol reaction.



An oven-dried tube was charged with alkyne (0.20 mmol), nitrile (0.40 mmol), ^{t}BuOK (2.5 mmol). The tube was evacuated and backfilled with Ar. Then, cyclohexane (1.5 mL) was added. The reaction mixture was stirring at 100 ^{o}C for 11 h. while the reaction finished, a small amount of silica gel was added into reaction system. The solvent was removed under reduced pressure. The aimed products of imidazoles were purified and isolated by flash column chromatography (acidic silica gel).

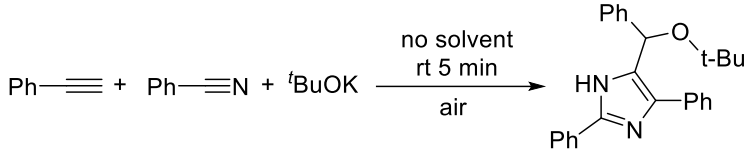
ii) General experimental procedure for synthesis of **4E** on 1.0 mmol scale reaction.



To a stirred solution of alkyne (**1a**, 1.0 equiv, 102 mg) and nitrile (**2E**, 4.0 equiv, 416 mg) in cyclohexane (20 mL) were added ^{t}BuOK (2.5 equiv, 280 mg) successively. The round bottom flask was evacuated and backfilled with Ar. The reaction mixture was stirring at 100 ^{o}C for 11 h. while the reaction finished, a small amount of silica gel was added into reaction system. The solvent was removed under reduced pressure. The aimed products of imidazoles were purified and isolated in 62% yield (238 mg)

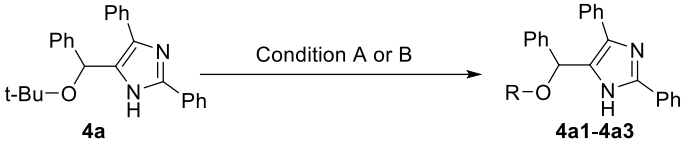
by flash column chromatography (acidic silica gel).

iii) General experimental procedure for solvent-free reactions.



A round bottom flask was charged with alkyne (1.0 equiv) and nitrile (4.0 equiv), And then ^{t}BuOK (2.5 equiv) was slowly added into the mixture with stirring. Then reaction system became thick and the stirring device could not continue to work well. Ethyl acetate and a little of silica gel was added into reaction system to help mix well. The solvent was removed under reduced pressure. The aimed products of imidazoles were purified and isolated by flash column chromatography (acidic silica gel).

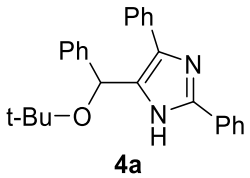
iv) General experimental procedure for synthesis of **4a1**-**4a3**.



Condition A: An oven-dried tube was charged with imidazole **4a** (0.20 mmol), concentrated hydrochloric acid and alcohol (V_{HCl}:V_{ROH} = 1:7), The reaction mixture was stirring at 70 ^{o}C for 2 h. The solvent was removed under reduced pressure. The aimed products of imidazoles were purified and isolated by flash column chromatography.

Condition B: An oven-dried tube was charged with imidazole **4a** (0.20 mmol), ROH (5.0 equiv) concentrated hydrochloric acid and dichloromethane (V_{HCl}:$V_{CH_2Cl_2}$ = 1:7), The reaction mixture was stirring at 60 ^{o}C for 2 h. The solvent was removed under reduced pressure. The aimed products of imidazoles were purified and isolated by flash column chromatography.

3. Characterization Data of 4a-4y, 4A-4E and 4a1-4a3.



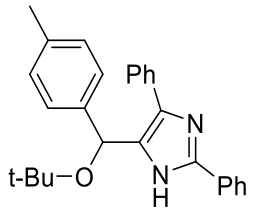
5-(tert-butoxy(phenyl)methyl)-2,4-diphenyl-1H-imidazole (4a)

The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a yellow solid in 93% yield (71 mg, mp 200-202 ^{o}C.) **^{1}H NMR (400 MHz, Chloroform-*d*)** δ 9.44 (s, 1H), 7.88 (d, *J* = 7.3 Hz, 2H), 7.68 (d, *J* = 7.5 Hz, 2H), 7.45 – 7.33 (m, 9H), 7.31 – 7.25 (m, 2H), 6.02 (s, 1H), 1.18 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 145.7, 141.8, 138.5, 134.5, 130.0, 129.8, 128.8, 128.7, 128.6, 128.5, 127.7, 127.1, 126.9, 126.9, 125.1, 75.7, 67.6, 28.4.

HRMS (ESI) *m/z* Calcd for $C_{26}H_{26}N_2OH^+$ 383.2118, found 383.2111.

IR (KBr, cm^{-1}) ν**:** 3057, 2973, 1601, 1583, 1520, 1456, 1413, 1042, 1009, 772, 694.



4b

5-(tert-butoxy(p-tolyl)methyl)-2,4-diphenyl-1H-imidazole (4b)

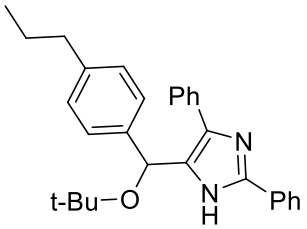
The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a yellow solid in 70% yield (55 mg, mp 144-146 °C).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.62 (s, 1H), 7.86 (d, *J* = 7.4 Hz, 2H), 7.66 – 7.65 (m, 2H), 7.40 – 7.31 (m, 5H), 7.30 – 7.20 (m, 3H), 7.13 (d, *J* = 7.8 Hz, 2H), 5.97 (s, 1H), 2.31 (s, 3H), 1.15 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 145.6, 138.9, 138.2, 137.3, 134.5, 130.1, 130.0, 129.1, 128.7, 128.5, 128.4, 127.1, 126.9, 126.8, 125.1, 75.5, 67.5, 28.3, 21.0.

HRMS (ESI) *m/z* Calcd for $C_{27}H_{28}N_2OH^+$ 397.2274, found 397.2273.

IR (KBr, cm^{-1}) ν**:** 3053, 2974, 1606, 1583, 1493, 1457, 1390, 1190, 1043, 1008, 698.



4c

5-(tert-butoxy(4-propylphenyl)methyl)-2,4-diphenyl-1H-imidazole (4c)

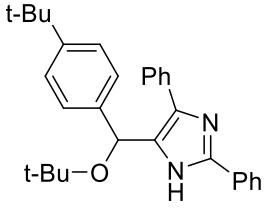
The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a yellow oil in 70% yield (58 mg).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.54 (s, 1H), 7.89 – 7.87 (m, 2H), 7.68 (d, *J* = 7.5 Hz, 2H), 7.44 – 7.34 (m, 5H), 7.32 – 7.24 (m, 3H), 7.16 (d, *J* = 8.0 Hz, 2H), 5.99 (s, 1H), 2.57 (t, *J* = 7.7 Hz, 2H), 1.62 (q, *J* = 7.5 Hz, 2H), 1.17 (s, 9H), 0.93 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 145.6, 142.2, 139.0, 138.3, 134.6, 130.1, 128.8, 128.6, 128.6, 128.5, 127.1, 126.9, 126.8, 125.1, 75.6, 67.5, 37.6, 28.4, 24.5, 13.8.

HRMS (ESI) *m/z* Calcd for $C_{29}H_{32}N_2OH^+$ 425.2587, found 425.2588.

IR (KBr, cm^{-1}) ν**:** 3188, 2970, 1606, 1580, 1493, 1460, 1311, 1108, 1017, 696.



4d

5-(tert-butoxy(4-(tert-butyl)phenyl)methyl)-2,4-diphenyl-1H-imidazole (4d)

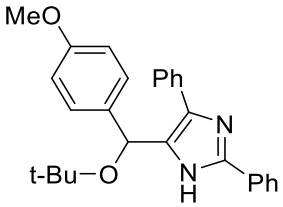
The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a brown oil in 60% yield (53 mg).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.60 (s, 1H), 7.86 (d, *J* = 7.6 Hz, 2H), 7.68 (d, *J* = 7.6 Hz, 2H), 7.38 – 7.24 (m, 10H), 6.00 (s, 1H), 1.30 (s, 9H), 1.15 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 150.5, 145.6, 138.8, 138.4, 134.6, 130.1, 128.7, 128.5, 128.5, 127.1, 126.8, 126.6, 125.4, 125.1, 75.5, 67.4, 34.4, 31.3, 28.3.

HRMS (ESI) *m/z* Calcd for $C_{30}H_{34}N_2OH^+$ 439.2744, found 439.2742.

IR (KBr, cm^{-1}) ν: 3208, 2966, 1697, 1606, 1493, 1447, 1365, 1266, 1191, 1017, 773, 737, 698.



4e

5-(tert-butoxy(4-methoxyphenyl)methyl)-2,4-diphenyl-1H-imidazole (4e)

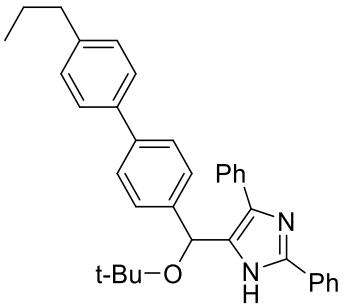
The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a brown oil in 76% yield (62 mg).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.74 (s, 1H), 7.88 (d, *J* = 7.4 Hz, 2H), 7.63 (d, *J* = 7.0 Hz, 2H), 7.42 – 7.33 (m, 5H), 7.30 – 7.24 (m, 3H), 6.85 (d, *J* = 8.6 Hz, 2H), 5.94 (s, 1H), 3.76 (s, 3H), 1.15 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 159.0, 145.5, 138.1, 134.5, 133.9, 130.0, 128.8, 128.6, 128.5, 128.2, 127.2, 127.1, 126.8, 125.1, 113.9, 75.5, 67.3, 55.2, 28.4.

HRMS (ESI) *m/z* Calcd for $C_{27}H_{28}N_2O_2H^+$ 413.2224, found 413.2221.

IR (KBr, cm^{-1}) ν: 3060, 2970, 1608, 1585, 1511, 1461, 1248, 1174, 1031, 774, 736, 697.



4f

5-(tert-butoxy(4'-propyl-[1,1'-biphenyl]-4-yl)methyl)-2,4-diphenyl-1H-imidazole (4f)

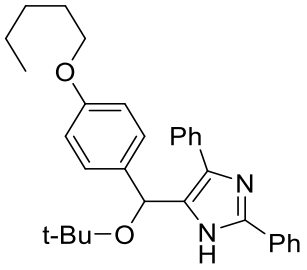
The crude product was purified by flash column chromatography (8:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a brown oil in 72% yield (72 mg).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.67 (s, 1H), 7.87 (d, *J* = 7.7 Hz, 2H), 7.70 (d, *J* = 7.3 Hz, 2H), 7.55 – 7.53 (m, 2H), 7.48 – 7.44 (m, 4H), 7.38 (s, 4H), 7.32 – 7.28 (m, 2H), 7.23 – 7.18 (m, 2H), 6.04 (s, 1H), 2.60 (t, *J* = 7.7 Hz, 2H), 1.65 (h, *J* = 7.4 Hz, 2H), 1.17 (s, 9H), 0.95 (t, *J* = 7.3 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 145.7, 141.9, 140.5, 138.5, 138.0, 134.5, 131.4, 130.0, 128.8, 128.7, 128.7, 128.6, 128.5, 127.3, 127.2, 127.0, 126.9, 126.8, 125.1, 75.7, 67.5, 37.6, 28.3, 24.4, 13.8.

HRMS (ESI) *m/z* Calcd for $C_{35}H_{36}N_2OH^+$ 501.2900, found 501.2900.

IR (KBr, cm^{-1}) ν: 3415, 3215, 2965, 1638, 1522, 1460, 1265, 739, 700.



4g

5-(tert-butoxy(4-(pentyloxy)phenyl)methyl)-2,4-diphenyl-1H-imidazole (4g)

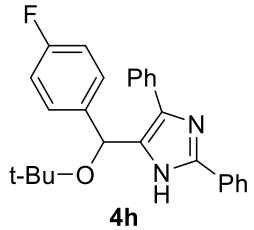
The crude product was purified by flash column chromatography (8:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a brown oil in 65% yield (61 mg).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.63 (s, 1H), 7.89 (d, *J* = 7.3 Hz, 2H), 7.65 (d, *J* = 7.6 Hz, 2H), 7.44 – 7.41 (m, 2H), 7.39 – 7.33 (m, 3H), 7.30 – 7.24 (m, 3H), 6.86 (d, *J* = 8.4 Hz, 2H), 5.95 (s, 1H), 3.93 (t, *J* = 6.6 Hz, 2H), 1.76 (p, *J* = 6.8 Hz, 2H), 1.46 – 1.32 (m, 4H), 1.16 (s, 9H), 0.92 (t, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 158.7, 145.5, 138.2, 136.6, 134.5, 133.6, 130.1, 128.8, 128.6, 128.5, 128.2, 127.1, 126.8, 125.1, 114.5, 75.6, 68.0, 67.4, 28.9, 28.4, 28.2, 22.4, 14.0.

HRMS (ESI) *m/z* Calcd for $C_{31}H_{36}N_2O_2H^+$ 469.2850, found 469.2851.

IR (KBr, cm^{-1}) ν**:** 3406, 3058, 2932, 1686, 1608, 1584, 1510, 1461, 1395, 1248, 1027, 774, 738, 697.



4h

5-(tert-butoxy(4-fluorophenyl)methyl)-2,4-diphenyl-1H-imidazole (4h)

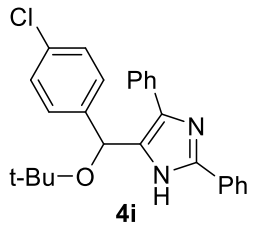
The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a yellow solid in 68% yield (54 mg, mp 132-134 °C).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.48 (s, 1H), 7.88 (d, *J* = 7.6 Hz, 2H), 7.63 (s, 2H), 7.43 – 7.25 (m, 8H), 7.01 (t, *J* = 8.6 Hz, 2H), 5.98 (s, 1H), 1.17 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 162.2 (d, *J* = 244.1 Hz), 145.7, 138.4, 137.6, 134.4, 130.0, 129.6 (d, *J* = 4.6 Hz), 128.8, 128.7, 128.7, 128.6, 128.6, 127.1 (d, *J* = 7.3 Hz), 125.2, 115.3 (d, *J* = 21.2 Hz), 75.8, 67.2, 28.4.

HRMS (ESI) *m/z* Calcd for $C_{26}H_{25}FN_2OH^+$ 401.2024, found 401.2025.

IR (KBr, cm^{-1}) ν**:** 3400, 3036, 1604, 1592, 1493, 1299, 1092, 772, 719, 695.



4i

5-(tert-butoxy(4-chlorophenyl)methyl)-2,4-diphenyl-1H-imidazole (4i)

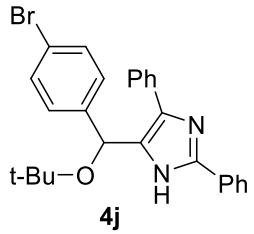
The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a yellow solid in 58% yield (48 mg, mp 172-174 °C).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.52 (s, 1H), 7.87 (d, *J* = 7.4 Hz, 2H), 7.61 (d, *J* = 6.8 Hz, 2H), 7.44 – 7.35 (m, 5H), 7.34 – 7.25 (m, 5H), 5.96 (s, 1H), 1.16 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 145.9, 140.5, 138.5, 134.3, 133.5, 129.9, 129.3, 128.8, 128.7, 128.6, 128.6, 128.3, 127.1, 127.1, 125.2, 75.9, 67.1, 28.3.

HRMS (ESI) *m/z* Calcd for $C_{26}H_{25}ClN_2OH^+$ 417.1728, found 417.1729.

IR (KBr, cm^{-1}) ν**:** 3056, 2976, 1665, 1490, 1459, 1395, 1367, 1183, 1045, 1009, 772, 701.



4j

5-((4-bromophenyl)(tert-butoxy)methyl)-2,4-diphenyl-1H-imidazole (4j)

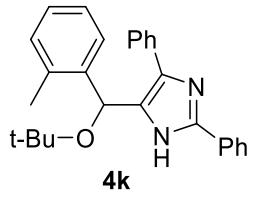
The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a yellow solid in 74% yield (68 mg, mp 160-162 °C).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.43 (s, 1H), 7.88 (d, *J* = 7.2 Hz, 2H), 7.65 (d, *J* = 7.2 Hz, 2H), 7.47 – 7.37 (m, 8H), 7.31 (d, *J* = 7.3 Hz, 1H), 7.27 (s, 1H), 5.96 (s, 1H), 1.17 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 145.8, 141.0, 138.5, 134.4, 131.6, 129.9, 129.2, 128.8, 128.8, 128.7, 128.6, 127.1, 127.1, 125.2, 121.7, 75.9, 67.2, 28.3.

HRMS (ESI) *m/z* Calcd for $C_{26}H_{25}BrN_2OH^+$ 461.1223, found 461.1227.

IR (KBr, cm^{-1}) ν**:** 3163, 2980, 1682, 1492, 1392, 1160, 1046, 739, 695.



4k

5-(tert-butoxy(o-tolyl)methyl)-2,4-diphenyl-1H-imidazole (4k)

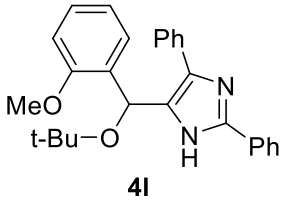
The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a yellow oil in 63% yield (50 mg).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.32 (s, 1H), 7.82 – 7.79 (m, 2H), 7.60 (t, *J* = 8.5 Hz, 3H), 7.38 – 7.36 (m, 4H), 7.33 – 7.28 (m, 2H), 7.20 – 7.14 (m, 2H), 7.10 (d, *J* = 7.1 Hz, 1H), 6.05 (s, 1H), 2.23 (s, 3H), 1.14 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 151.0, 143.7, 138.8, 137.0, 136.2, 135.8, 134.9, 130.2, 129.2, 128.7, 128.4, 128.4, 128.4, 127.5, 127.3, 126.9, 125.0, 75.2, 66.0, 28.5, 20.7.

HRMS (ESI) *m/z* Calcd for $C_{27}H_{28}N_2OH^+$ 397.2274, found 397.2272.

IR (KBr, cm^{-1}) ν**:** 3435,3059, 2971, 2922, 1669, 1609, 1490, 1460, 1265, 773, 737, 697.



4l

5-(tert-butoxy(2-methoxyphenyl)methyl)-2,4-diphenyl-1H-imidazole (4l)

The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc)

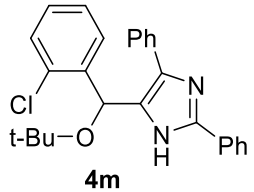
on silica gel to provide the title compound as a yellow solid in 76% yield (62 mg).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.63 (s, 1H), 7.83 (d, *J* = 7.2 Hz, 2H), 7.75 (d, *J* = 7.6 Hz, 2H), 7.56 – 7.53 (m, 1H), 7.37 (t, *J* = 7.7 Hz, 4H), 7.30 – 7.20 (m, 3H), 6.94 (t, *J* = 7.5 Hz, 1H), 6.83 (d, *J* = 8.2 Hz, 1H), 6.31 (s, 1H), 3.72 (s, 3H), 1.10 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 155.8, 144.8, 137.4, 135.0, 130.8, 130.2, 129.9, 128.7, 128.6, 128.3, 128.1, 127.9, 127.6, 126.4, 125.0, 120.8, 110.9, 75.3, 63.1, 55.1, 28.3.

HRMS (ESI) *m/z* Calcd for $C_{27}H_{28}N_2O_2H^+$ 413.2224, found 413.2221.

IR (KBr, cm^{-1}) ν**:** 3409,3059, 2971, 2837, 1705, 1600, 1492, 1367, 1204, 774, 737, 697.



4m

5-(tert-butoxy(2-chlorophenyl)methyl)-2,4-diphenyl-1H-imidazole (4m)

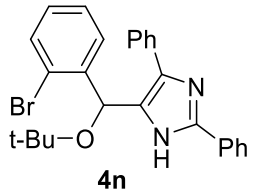
The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a yellow solid 75% yield (62 mg, mp 128-130 °C).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.47 (s, 1H), 7.82 (d, *J* = 7.4 Hz, 2H), 7.61 (s, 2H), 7.38 – 7.22 (m, 9H), 7.19 – 7.17 (m, 1H), 6.28 (s, 1H), 1.13 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 145.5, 140.1, 136.0, 133.4, 132.1, 129.9, 129.6, 128.7, 128.6, 128.4, 128.1, 128.1, 127.0, 126.9, 125.3, 75.8, 65.8, 28.5.

HRMS (ESI) *m/z* Calcd for $C_{26}H_{25}ClN_2OH^+$ 417.1728, found 417.1730.

IR (KBr, cm^{-1}) ν**:** 3447, 3065, 2973, 1581, 1568, 1493, 1456, 1390, 1366, 1191, 1005, 754, 733, 693.



4n

5-((2-bromophenyl)(tert-butoxy)methyl)-2,4-diphenyl-1H-imidazole (4n)

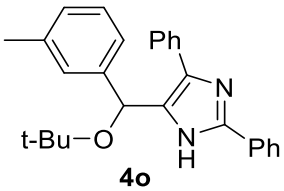
The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a yellow solid in 54% yield (50 mg, mp 170-172 °C).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.47 (s, 1H), 7.88 – 7.86 (d, *J* = 7.4 Hz, 2H), 7.68 – 7.67(d, *J* = 6.2 Hz, 2H), 7.43 – 7.38 (m, 5H), 7.36 – 7.32 (d, *J* = 7.5 Hz, 3H), 7.29 – 7.26(d, *J* = 7.1 Hz, 2H), 6.01 (s, 1H), 1.17 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 145.68, 141.93, 138.51, 134.63, 130.13, 129.91, 128.85, 128.69, 128.57, 127.69, 127.21, 126.98 (d, *J* = 2.7 Hz), 125.19, 77.37, 77.05, 76.73, 67.70, 28.44.

HRMS (ESI) *m/z* Calcd for $C_{26}H_{25}BrN_2OH^+$ 461.1223, found 461.1226.

IR (KBr, cm^{-1}) ν**:** 3447, 3065, 2973, 1581, 1568, 1493, 1456, 1390, 1366, 1191, 1005, 754, 733, 693.



4o

5-(tert-butoxy(m-tolyl)methyl)-2,4-diphenyl-1H-imidazole (4o)

The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc)

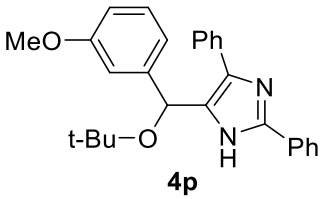
on silica gel to provide the title compound as a yellow oil in 75% yield (59 mg).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.57 (s, 1H), 7.88 (d, *J* = 7.5 Hz, 2H), 7.67 (d, *J* = 7.6 Hz, 2H), 7.44 – 7.34 (m, 5H), 7.28 (t, *J* = 7.6 Hz, 1H), 7.21 (d, *J* = 7.8 Hz, 3H), 7.09 (d, *J* = 6.7 Hz, 1H), 5.98 (s, 1H), 2.32 (s, 3H), 1.16 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 145.6, 141.8, 138.4, 138.3, 134.6, 130.1, 130.0, 128.8, 128.6, 128.5, 128.5, 128.5, 127.6, 127.2, 126.9, 125.1, 124.1, 75.7, 67.7, 28.4, 21.6.

HRMS (ESI) *m/z* Calcd for $C_{27}H_{28}N_2OH^+$ 397.2274, found 397.2273.

IR (KBr, cm^{-1}) ν**:** 3407, 3057, 2974, 1702, 1606, 1587, 1491, 1461, 1265, 1051, 773, 737, 697.



4p

5-(tert-butoxy(3-methoxyphenyl)methyl)-2,4-diphenyl-1H-imidazole (4p)

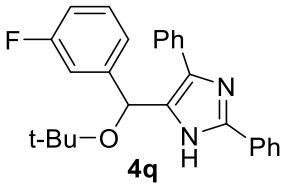
The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a yellow solid in 52% yield (43 mg, mp 151-153 °C).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.68 (s, 1H), 7.87 (d, *J* = 7.6 Hz, 2H), 7.66 (d, *J* = 7.5 Hz,2H), 7.40 – 7.23 (m, 7H), 7.00 – 6.97 (m, 2H), 6.80 (d, *J* = 8.2 Hz, 1H), 5.97 (s, 1H), 3.73 (s, 3H), 1.15 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 159.7, 145.7, 143.6, 138.4, 134.5, 130.0, 129.5, 128.7, 128.6, 128.5, 127.2, 126.9, 125.1, 119.1, 112.8, 112.6, 75.6, 67.5, 55.1, 28.3.

HRMS (ESI) *m/z* Calcd for $C_{27}H_{28}N_2O_2H^+$ 413.2224, found 413.2221.

IR (KBr, cm^{-1}) ν**:** 3063, 2973, 1610, 1587, 1491, 1457, 1263, 1192, 1043, 1020, 912, 771, 695.



4q

5-(tert-butoxy(3-fluorophenyl)methyl)-2,4-diphenyl-1H-imidazole (4q)

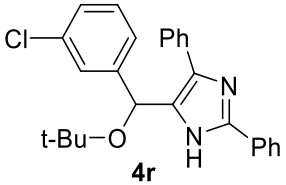
The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a yellow oil in 60% yield (48mg).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.41 (s, 1H), 7.88 (d, *J* = 7.6 Hz, 2H), 7.67 (d, *J* = 7.6 Hz, 2H), 7.43 – 7.36 (m, 5H), 7.32 – 7.27 (m, 2H), 7.17 (d, *J* = 10.2 Hz, 1H), 7.12 (d, *J* = 7.9 Hz, 1H), 6.99 – 6.95 (m, 1H), 5.99 (s, 1H), 1.17 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 163.1 (d, *J* = 246.4 Hz), 145.9, 144.7 (d, *J* = 6.9 Hz), 138.7, 134.5, 130.1, 130.0 (d, *J* = 2.8 Hz), 129.3, 128.9, 128.8, 128.6, 127.2, 127.1, 125.2, 122.4 (d, *J* = 2.3 Hz), 114.6 (d, *J* = 21.3 Hz), 113.9 (d, *J* = 22.4 Hz), 76.0, 67.1, 28.3.

HRMS (ESI) *m/z* Calcd for $C_{26}H_{25}FN_2OH^+$ 401.2024, found 401.2023.

IR (KBr, cm^{-1}) ν**:** 3413, 3062, 2968, 1688, 1574, 1476, 1414, 1390, 1254, 1188, 1047, 966, 786, 695.



4r

5-(tert-butoxy(3-chlorophenyl)methyl)-2,4-diphenyl-1H-imidazole (4r)

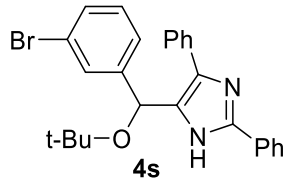
The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a yellow solid in 53% yield (44 mg, mp 196-198 °C).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.44 (s, 1H), 7.89 – 7.86 (m, 2H), 7.67 – 7.65 (m, 2H), 7.44 – 7.42 (m, 3H), 7.41 – 7.39 (m, 2H), 7.37 – 7.30 (m, 2H), 7.26 – 7.23 (m, 3H), 5.98 (s, 1H), 1.17 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 145.9, 144.1, 138.7, 134.6, 134.4, 129.9, 129.8, 129.1, 128.9, 128.8, 128.6, 127.9, 127.2, 127.1, 127.0, 125.2, 125.0, 76.0, 67.1, 28.3.

HRMS (ESI) *m/z* Calcd for $C_{26}H_{25}ClN_2OH^+$ 417.1728, found 417.1727.

IR (KBr, cm^{-1}) ν: 3065, 2974, 1699, 1597, 1574, 1493, 1475, 1457, 1367, 1182, 1047, 904, 771, 735, 695.



4s

5-((3-bromophenyl)(tert-butoxy)methyl)-2,4-diphenyl-1H-imidazole (4s)

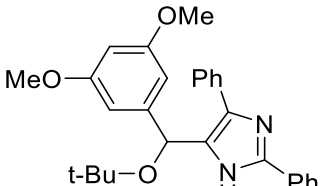
The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a brown oil in 57% yield (52 mg).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.52 (s, 1H), 7.88 (d, *J* = 7.2 Hz, 2H), 7.63 (s, 3H), 7.45 – 7.36 (m, 6H), 7.32 – 7.25 (m, 2H), 7.19 – 7.16(m, 1H), 5.96 (s, 1H), 1.16 (s, 9H). .

^{13}C NMR (101 MHz, Chloroform-*d*) δ 146.0, 144.2, 138.7, 134.3, 130.8, 130.1, 129.9, 129.8, 129.0, 128.8, 128.8, 128.8, 128.6, 127.1, 125.5, 125.2, 122.8, 76.0, 67.0, 28.3.

HRMS (ESI) *m/z* Calcd for $C_{26}H_{25}BrN_2OH^+$ 461.1223, found 461.1230.

IR (KBr, cm^{-1}) ν: 3438, 3064, 2973, 1719, 1592, 1571, 1461, 1368, 1190, 1070, 909, 773, 732, 696.



4t

5-(tert-butoxy(3,5-dimethoxyphenyl)methyl)-2,4-diphenyl-1H-imidazole (4t)

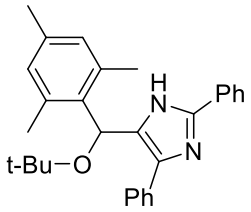
The crude product was purified by flash column chromatography (5:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a brown oil in 73% yield (65 mg).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.55 (s, 1H), 7.89 – 7.87 (m, 2H), 7.70 (d, *J* = 7.6 Hz, 2H), 7.43 – 7.38 (m, 4H), 7.35 (d, *J* = 7.3 Hz, 1H), 7.32 – 7.27 (m, 1H), 6.57 (s, 2H), 6.36 (s, 1H), 5.95 (s, 1H), 3.71 (s, 6H), 1.16 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 160.9, 145.6, 144.5, 138.4, 134.6, 130.0, 129.7, 128.8, 128.6, 128.5, 127.2, 126.9, 125.1, 104.9, 99.1, 75.7, 67.5, 55.2, 28.3.

HRMS (ESI) *m/z* Calcd for $C_{28}H_{30}N_2O_3H^+$ 443.2329, found 443.2272.

IR (KBr, cm^{-1}) ν: 3419, 3059, 2971, 1705, 1596, 1460, 1427, 1203, 1154, 1059, 927, 774, 736, 697.



4u

5-(tert-butoxy(mesityl)methyl)-2,4-diphenyl-1H-imidazole (4u)

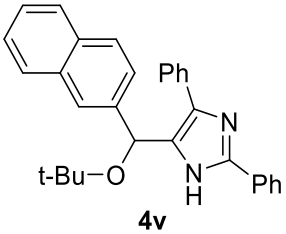
The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a brown oil in 50% yield (42 mg).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.30 (s, 1H), 7.77 (d, *J* = 7.1 Hz, 2H), 7.39 (t, *J* = 7.6 Hz, 2H), 7.32 (d, *J* = 7.4 Hz, 1H), 7.23 – 7.19 (m, 5H), 6.72 (s, 2H), 6.09 (s, 1H), 2.27 (s, 6H), 2.23 (s, 3H), 1.13 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 145.3, 141.2, 140.6, 135.0, 130.9, 130.0, 128.7, 128.5, 128.3, 128.3, 128.0, 127.5, 127.0, 126.1, 125.2, 75.5, 69.2, 31.2, 28.7, 19.7.

HRMS (ESI) *m/z* Calcd for $C_{29}H_{32}N_2OH^+$ 425.2587, found 425.2584.

IR (KBr, cm^{-1}) ν**:** 3417, 3059, 2970, 1663, 1604, 1491, 1460, 1395, 1029, 909, 772, 731, 696.



4v

5-(tert-butoxy(naphthalen-2-yl)methyl)-2,4-diphenyl-1H-imidazole (4v)

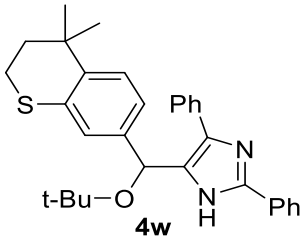
The crude product was purified by flash column chromatography (5:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a yellow solid in 58% yield (50 mg, mp 182-184 °C).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.64 (s, 1H), 7.87 (d, *J* = 7.4 Hz, 2H), 7.83 – 7.74 (m, 4H), 7.70 – 7.69 (m, 2H), 7.57 (d, *J* = 7.9 Hz, 1H), 7.44 – 7.42 (m, 2H), 7.38 – 7.31 (m, 5H), 7.28 – 7.21 (m, 1H), 6.15 (s, 1H), 1.20 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 145.8, 139.3, 138.6, 134.5, 133.2, 132.8, 130.0, 129.7, 129.0, 128.8, 128.6, 128.5, 128.1, 127.5, 127.2, 127.0, 127.0, 125.6, 125.1, 124.5, 120.3, 75.8, 67.9, 28.4.

HRMS (ESI) *m/z* Calcd for $C_{30}H_{28}N_2OH^+$ 433.2274, found 433.2272.

IR (KBr, cm^{-1}) ν**:** 3427, 3057, 2970, 1600, 1579, 1495, 1458, 1390, 1363, 1190, 1052, 884, 776, 702.



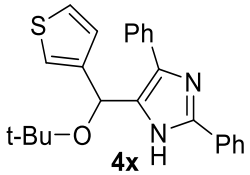
4w

5-(tert-butoxy(4,4-dimethylthiochroman-7-yl)methyl)-2,4-diphenyl-1H-imidazole (4w)

The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a yellow oil in 65% yield (63 mg).

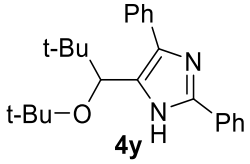
^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.58 (s, 1H), 7.87 (d, *J* = 8.8 Hz, 2H), 7.65 (d, *J* = 7.6 Hz, 2H), 7.43 – 7.32 (m, 6H), 7.28 (d, *J* = 7.3 Hz, 1H), 7.04 (s, 2H), 5.93 (s, 1H), 3.00 – 2.87(m, 2H), 1.93 – 1.90 (m, 2H), 1.24 (s, 3H), 1.23 (s, 3H), 1.18 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 145.5, 142.0, 138.2, 137.6, 134.6, 131.3, 130.1, 128.8, 128.6, 128.5, 128.2, 127.2, 126.9, 126.6, 125.1, 125.1, 124.5, 124.3, 75.6, 67.6, 37.6, 33.0, 30.2, 28.4, 23.0.
HRMS (ESI) *m/z* Calcd for $C_{31}H_{34}N_2OSH^+$ 483.2465, found 483.2465.
IR (KBr, cm^{-1}) ν**:** 3417, 3063, 2929, 1715, 1591, 1460, 1368, 1363, 1190, 1070, 1052, 884, 773, 700, 696.



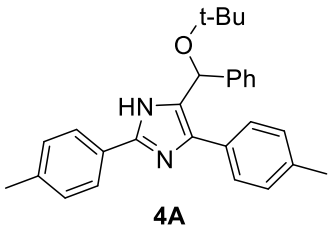
4x

5-(tert-butoxy(thiophen-3-yl)methyl)-2,4-diphenyl-1H-imidazole (4x)
The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a yellow solid in 49% yield (38 mg, mp 110-112 °C).
^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.47 (s, 1H), 7.91 – 7.88 (m, 2H), 7.69 – 7.67 (m, 2H), 7.47 – 7.37 (m, 5H), 7.35 – 7.27 (m, 2H), 7.14 – 7.07 (m, 2H), 6.00 (s, 1H), 1.17 (s, 9H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 145.6, 143.5, 137.8, 134.4, 130.0, 129.9, 128.8, 128.7, 128.6, 127.1, 126.9, 126.8, 126.4, 125.1, 122.5, 76.7, 64.8, 28.3.
HRMS (ESI) *m/z* Calcd for $C_{24}H_{24}N_2OSH^+$ 389.1682, found 389.1684.
IR (KBr, cm^{-1}) ν**:** 3057, 2922, 1687, 1603, 1491, 1460, 1446, 1401, 1235, 1072, 838, 772, 695.



4y

5-(1-(tert-butoxy)-2,2-dimethylpropyl)-2,4-diphenyl-1H-imidazole (4y)
The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a yellow solid in 54% yield (39 mg, mp 118-120 °C).
^{1}H NMR (600 MHz, Chloroform-*d*) δ 9.37 (s, 1H), 7.89 (d, *J* = 7.6 Hz, 2H), 7.75 (d, *J* = 7.6 Hz, 2H), 7.45 (t, *J* = 7.6 Hz, 2H), 7.43 – 7.38 (m, 2H), 7.36 (t, *J* = 7.5 Hz, 1H), 7.29 – 7.24 (m, 1H), 4.72 (s, 1H), 1.16 (s, 9H), 0.88 (s, 9H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 144.3, 137.9, 135.6, 130.1, 130.0, 128.8, 128.5, 128.4, 127.4, 126.6, 125.0, 74.4, 73.3, 37.3, 28.5, 26.4.
HRMS (ESI) *m/z* Calcd for $C_{24}H_{30}N_2OH^+$ 363.2431, found 363.2428.
IR (KBr, cm^{-1}) ν**:** 3337, 2974, 1607, 1494, 1462, 1390, 1366, 1237, 1191, 1069, 1029, 774, 697.



4A

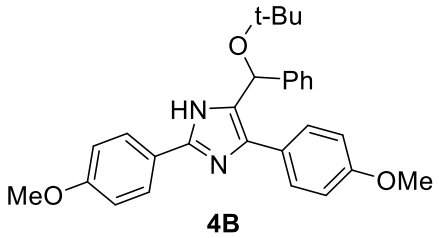
5-(tert-butoxy(phenyl)methyl)-2,4-di-p-tolyl-1H-imidazole (4A)
The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a yellow solid in 40% yield (33 mg, mp 160-162 °C).
^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.40 (s, 1H), 7.76 (d, *J* = 7.8 Hz, 2H), 7.55 (d, *J* = 7.6 Hz, 2H),

7.41 (d, *J* = 7.7 Hz, 2H), 7.36 – 7.32 (m, 2H), 7.28 (d, *J* = 6.6 Hz, 1H), 7.24 – 7.18 (m, 4H), 5.99 (s, 1H), 2.37 (d, *J* = 5.6 Hz, 6H), 1.17 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 145.7 , 142.0 , 138.6 , 138.4 , 136.5 , 131.7 , 129.5 , 129.2 , 129.0 , 128.8 , 128.5 , 127.6 , 127.3 , 127.0 , 125.0 , 75.6 , 67.6 , 28.4 , 21.3 , 21.2 .

HRMS (ESI) *m/z* Calcd for $C_{28}H_{30}N_2OH^+$ 411.2431, found 411.2431.

IR (KBr, cm^{-1}) ν**:** 3440, 2973, 2923, 1618, 1509, 1492, 1449, 1389, 1369, 1190, 1050, 1020, 909, 823, 731, 697.



4B

5-(tert-butoxy(phenyl)methyl)-2,4-bis(4-methoxyphenyl)-1H-imidazole (4B)

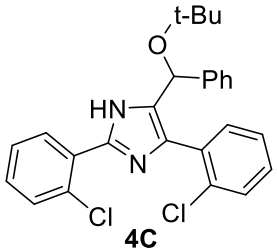
The crude product was purified by flash column chromatography (5:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a yellow solid in 53% yield (47 mg, mp 110-112 ºC).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.47 (s, 1H), 7.80 (d, *J* = 8.9 Hz, 2H), 7.57 (d, *J* = 8.3 Hz, 2H), 7.41 (d, *J* = 7.6 Hz, 2H), 7.33 (t, *J* = 7.4 Hz, 2H), 7.27 (d, *J* = 7.1 Hz, 1H), 6.94 – 6.92 (m, 4H), 5.95 (s, 1H), 3.81 (s, 3H), 3.80 (s, 3H), 1.16 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 159.9 , 158.6 , 145.6 , 142.0 , 138.1 , 128.5 , 128.4 , 127.5 , 127.3 , 126.9 , 126.6 , 123.0 , 114.1 , 113.9 , 99.9 , 75.6 , 67.5 , 55.3 , 55.2 , 28.3 .

HRMS (ESI) *m/z* Calcd for $C_{28}H_{30}N_2O_3H^+$ 443.2329, found 443.2326.

IR (KBr, cm^{-1}) ν**:** 3442, 2973, 2837, 2251, 1615, 1509, 1442, 1390, 1368, 1292, 1177, 1035, 909, 836, 732, 649.



4C

5-(tert-butoxy(phenyl)methyl)-2,4-bis(2-chlorophenyl)-1H-imidazole (4C)

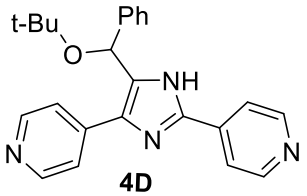
The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a yellow oil in 49% yield (44 mg).

^{1}H NMR (400 MHz, Chloroform-*d*) δ 10.30 (s, 1H), 8.27 (d, *J* = 6.9 Hz, 1H), 7.37 – 7.31 (m, 2H), 7.24 – 7.12 (m, 10H), 5.70 (s, 1H), 1.10 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 142.3 , 142.0 , 135.1 , 133.9 , 133.6 , 132.4 , 131.5 , 130.6 , 130.4 , 129.5 , 129.3 , 129.0 , 128.3 , 128.0 , 127.7 , 127.4 , 127.3 , 126.9 , 126.5 , 75.6 , 68.1 , 28.4 .

HRMS (ESI) *m/z* Calcd for $C_{26}H_{24}Cl_2N_2OH^+$ 451.1338, found 451.1339.

IR (KBr, cm^{-1}) ν: 3446, 3064, 2974, 1601, 1569, 1472, 1452, 1391, 1367, 1220, 1188, 1050, 908, 762, 732, 698, 647.



4D

4,4'-(5-(tert-butoxy(phenyl)methyl)-1H-imidazole-2,4-diyl)dipyridine (4D)

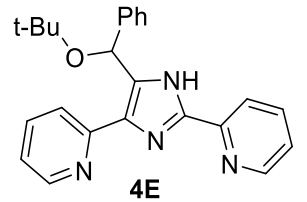
The crude product was purified by flash column chromatography (5:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a yellow solid in 58% yield (45 mg, mp 208-210 °C)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 12.68 (s, 1H), 8.56 – 8.52 (m, 4H), 7.91 (d, *J* = 6.2 Hz, 2H), 7.66 (d, *J* = 6.3 Hz, 2H), 7.33 – 7.25 (m, 5H), 6.07 (s, 1H), 1.16 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 149.9, 149.6, 144.4, 142.2, 141.0, 137.5, 136.3, 135.2, 128.5, 127.7, 126.6, 121.3, 119.6, 75.8, 67.5, 28.2.

HRMS (ESI) *m/z* Calcd for $C_{24}H_{24}N_4OH^+$ 385.2023, found 385.2020.

IR (KBr, cm^{-1}) ν: 3370, 2944, 2833, 1607, 1449, 1421, 1027, 836, 739, 697.



4E

2,2'-(5-(tert-butoxy(phenyl)methyl)-1H-imidazole-2,4-diyl)dipyridine (4E)

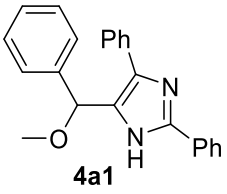
The crude product was purified by flash column chromatography (5:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a yellow solid in 69% yield (53 mg, mp 200-202 °C)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 10.44 (s, 1H), 8.59 (d, *J* = 3.7 Hz, 1H), 8.51 (d, *J* = 4.4 Hz, 1H), 8.23 (d, *J* = 7.9 Hz, 1H), 8.18 (d, *J* = 8.0 Hz, 1H), 7.80 – 7.69 (m, 2H), 7.52 (d, *J* = 7.3 Hz, 2H), 7.28 – 7.25 (m, 2H), 7.24 – 7.15 (m, 3H), 7.14 – 7.09 (m, 1H), 1.26 (s, 9H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 154.6, 148.9, 148.8, 148.4, 144.9, 143.1, 136.8, 136.5, 136.2, 135.9, 128.1, 126.8, 126.3, 123.0, 120.9, 120.1, 119.7, 75.5, 67.2, 28.4.

HRMS (ESI) *m/z* Calcd for $C_{24}H_{24}N_4OH^+$ 385.2023, found 385.2018.

IR (KBr, cm^{-1}) ν: 3368, 2953, 2834, 1655, 1581, 1508, 1388, 1115, 1027, 836, 733, 697.



4a1

5-(methoxy(phenyl)methyl)-2,4-diphenyl-1H-imidazole (4a1)

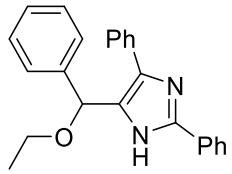
The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a white solid in 97% yield (66 mg, mp 168-170 °C)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.41 (s, 1H), 7.85 (d, *J* = 6.9 Hz, 2H), 7.65 (s, 2H), 7.43 – 7.28 (m, 11H), 5.64 (s, 1H), 3.38 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 146.1, 139.8, 134.4, 133.3, 130.9, 129.8, 128.8, 128.7, 128.6, 128.4, 128.0, 127.7, 127.2, 127.0, 125.4, 65.6, 56.9.

HRMS (ESI) *m/z* Calcd for $C_{23}H_{20}N_2OH^+$ 341.1648, found 341.1646.

IR (KBr, cm^{-1}) ν: 3415, 3060, 2930, 2821, 1720, 1703, 1603, 1586, 1493, 1460, 1398, 1265, 1073, 911, 772, 736, 697.



4a2

5-(ethoxy(phenyl)methyl)-2,4-diphenyl-1H-imidazole (4a2)

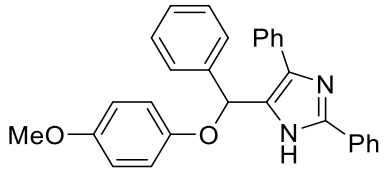
The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a white solid in 95% yield (67 mg, mp 154-156 ^{o}C)

^{1}H NMR (400 MHz, Chloroform-*d*) δ 9.44 (s, 1H), 7.83 – 7.81 (m, 2H), 7.65 (s, 2H), 7.43– 7.23 (m, 11H), 5.74 (s, 1H), 3.59 – 3.47(m, 2H), 1.22 (t, *J* = 7.0 Hz, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 146.0, 140.2, 138.8, 133.4, 129.8, 128.7, 128.6, 128.4, 127.9, 127.7, 127.1, 126.9, 125.3, 75.1, 64.7, 15.2.

HRMS (ESI) *m/z* Calcd for $C_{24}H_{22}N_2OH^+$ 355.1805, found 355.1802.

IR (KBr, cm^{-1}) ν: 3059, 2929, 1602, 1586, 1510, 1460, 1398, 1280, 1089, 1072, 1028, 771, 695.



4a3

5-((4-methoxyphenoxy)(phenyl)methyl)-2,4-diphenyl-1H-imidazole (4a3)

The crude product was purified by flash column chromatography (10:1 of Petroleum ether:EtOAc) on silica gel to provide the title compound as a brown oil in 88% yield (76 mg)

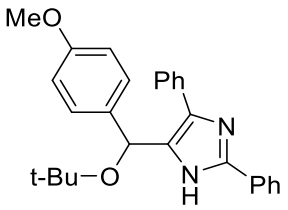
^{1}H NMR (400 MHz, Chloroform-*d*) δ 10.17 (s, 1H), 7.67 – 7.65 (m, 2H), 7.37 – 7.35 (m, 2H), 7.33 – 7.29 (m, 2H), 7.28 – 7.26 (m, 1H), 7.23 – 7.14 (m, 4H), 7.16 – 7.09 (m, 5H), 6.84 (d, *J* = 8.7 Hz, 1H), 6.80 (d, *J* = 3.0 Hz, 1H), 6.73 – 6.70 (m, 1H), 5.32 (s, 1H), 3.75 (s, 3H).

^{13}C NMR (101 MHz, Chloroform-*d*) δ 153.0, 149.7, 145.1, 141.9, 130.6, 129.7, 129.0, 129.0, 128.8, 128.5, 128.1, 128.0, 127.6, 127.5, 126.2, 125.2, 120.6, 117.7, 113.2, 55.8, 48.0.

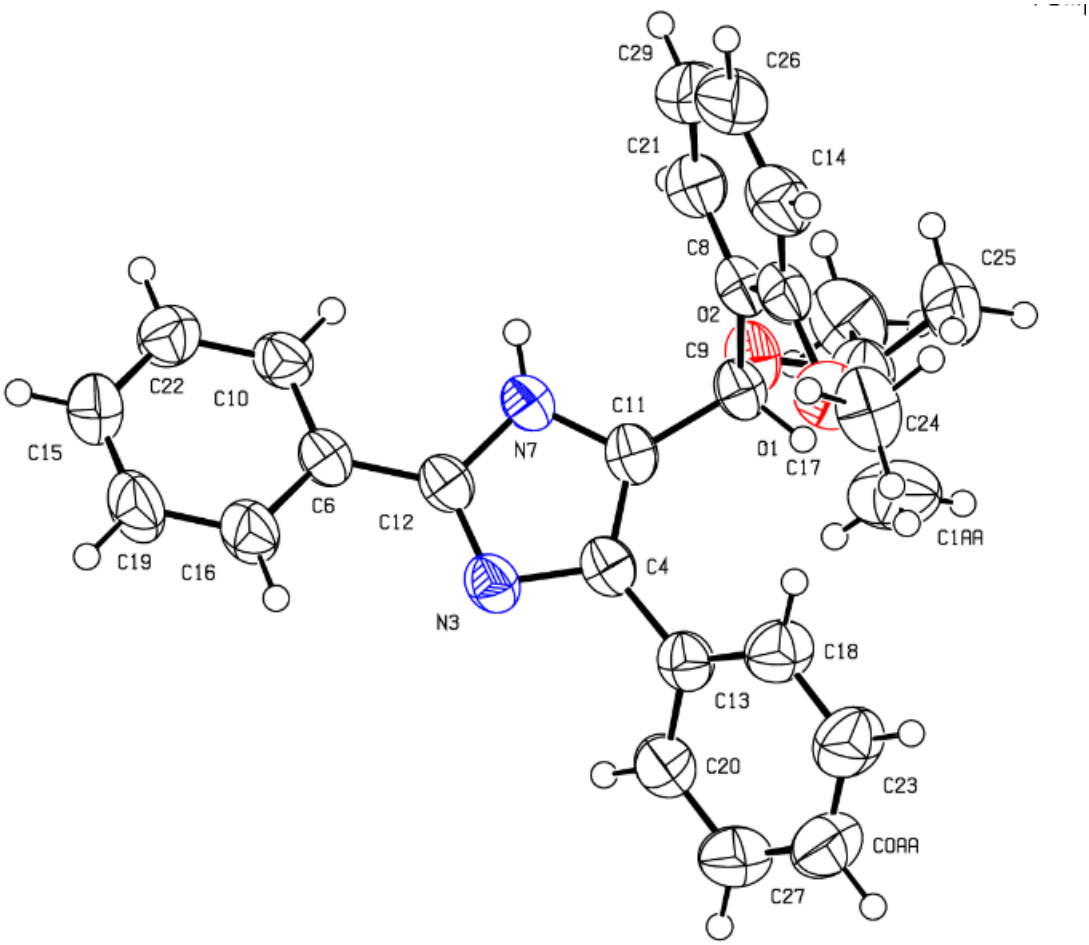
HRMS (ESI) *m/z* Calcd for $C_{29}H_{24}N_2O_2H^+$ 433.1911, found 433.1908.

IR (KBr, cm^{-1}) ν: 3426, 3055, 2962, 1722, 1602, 1586, 1511, 1459, 1398, 1264, 1124, 1028, 912, 773, 739, 700.

4. Crystallographic Data of 4e and 4v.



4e **CCDC:1961141**



Datablock: 4e

Bond precision:	C-C = 0.0028 A		Wavelength=1.54184
Cell:	a=10.7812(4)	b=19.7870(7)	c=10.8434(4)
	alpha=90	beta=99.281(3)	gamma=90
Temperature:	292 K		

	Calculated	Reported
Volume	2282.92(15)	2282.93(15)
Space group	P 21/c	P 1 21/c 1
Hall group	-P 2ybc	-P 2ybc

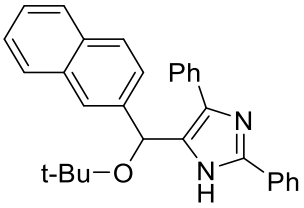
Moiety formula	C27 H28 N2 O2	C27 H28 N2 O2
Sum formula	C27 H28 N2 O2	C27 H28 N2 O2
Mr	412.51	412.51
Dx,g cm-3	1.200	1.200
Z	4	4
Mu (mm-1)	0.596	0.596
F000	880.0	880.0
F000'	882.46	
h,k,lmax	12,23,12	12,23,12
Nref	4032	3945
Tmin,Tmax	0.909,0.931	0.780,1.000
Tmin'	0.909	

Correction method= # Reported T Limits: Tmin=0.780 Tmax=1.000 AbsCorr = MULTI-SCAN

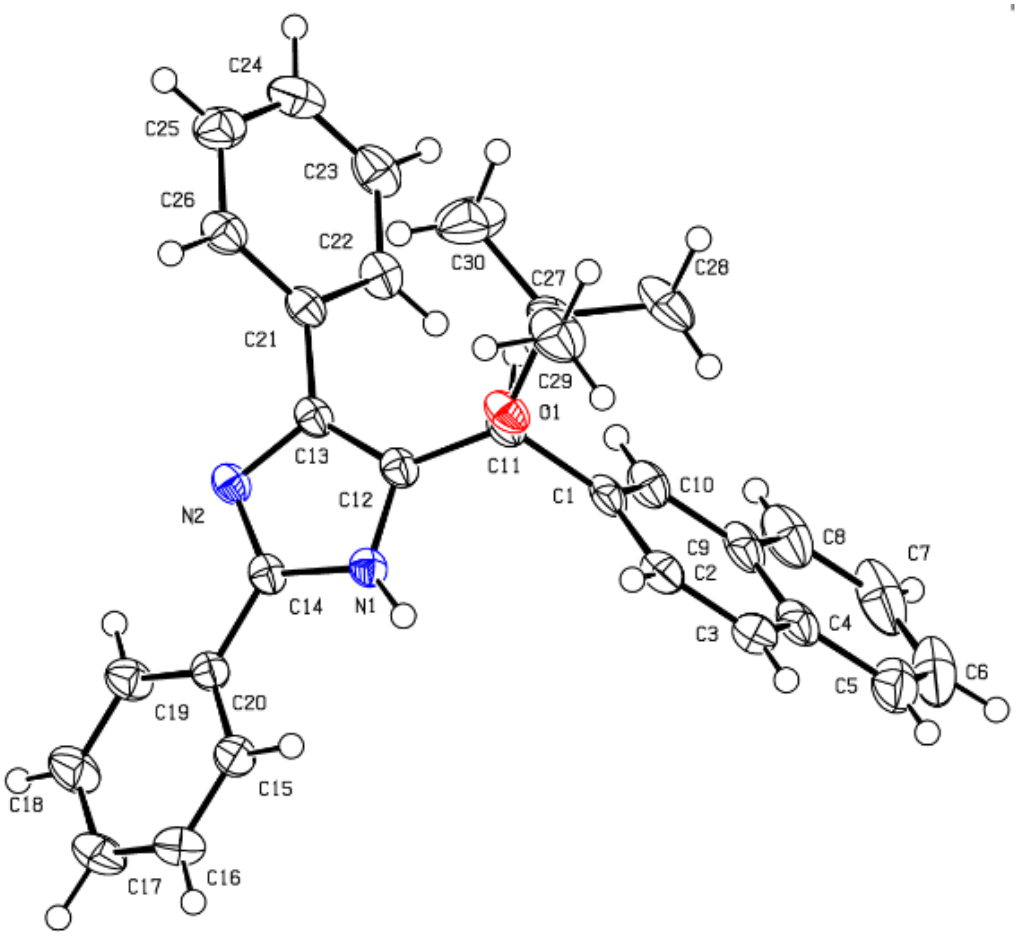
Data completeness= 0.978 Theta(max)= 66.579

R(reflections)= 0.0469(3062) wR2(reflections)= 0.1369(3945)

S = 1.024 Npar= 284



4v **CCDC:1961140**



Datablock: 4v

Bond precision: C-C = 0.0025 A Wavelength=1.54184

Cell: a=11.0585(2) b=20.8807(5) c=10.2359(2)
alpha=90 beta=101.3355(8) gamma=90

Temperature: 153 K

	Calculated	Reported
Volume	2317.46(8)	2317.46(8)
Space group	P 21/a	P 1 21/a 1
Hall group	-P 2yab	-P 2yab
Moiety formula	C30 H28 N2 O	C30 H28 N2 O
Sum formula	C30 H28 N2 O	C30 H28 N2 O

Mr	432.54	432.54
Dx,g cm-3	1.240	1.240
Z	4	4
Mu (mm-1)	0.581	0.581
F000	920.0	920.0
F000'	922.47	
h,k,lmax	13,24,12	12,24,12
Nref	4090	3925
Tmin,Tmax	0.901,0.922	0.689,0.753
Tmin'	0.875	

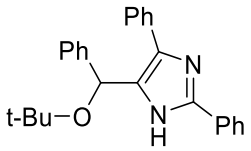
Correction method= # Reported T Limits: Tmin=0.689 Tmax=0.753 AbsCorr = MULTI-SCAN

Data completeness= 0.960 Theta(max)= 66.562

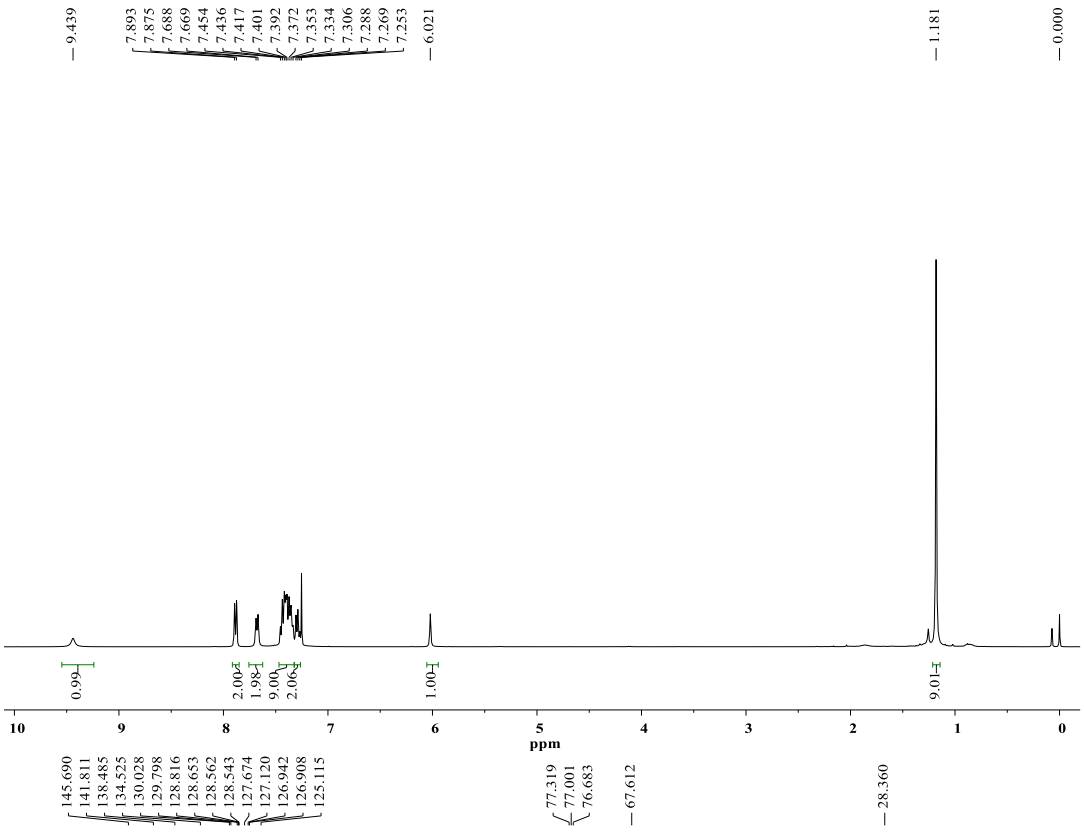
R(reflections)= 0.0478(3679) wR2(reflections)= 0.1261(3925)

S = 1.048 Npar= 305

5. ^{1}H NMR、^{13}C NMR Spectra for Substrates 4a-4y, 4A, 4B and 4a1-4a3.


Ph
Ph
N
t-Bu–O
N
H
Ph

4a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7.306
7.288
7.269
7.253
6.021
1.181
0.000
0.99
2.00
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1.00
9.01
10
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8
7
6
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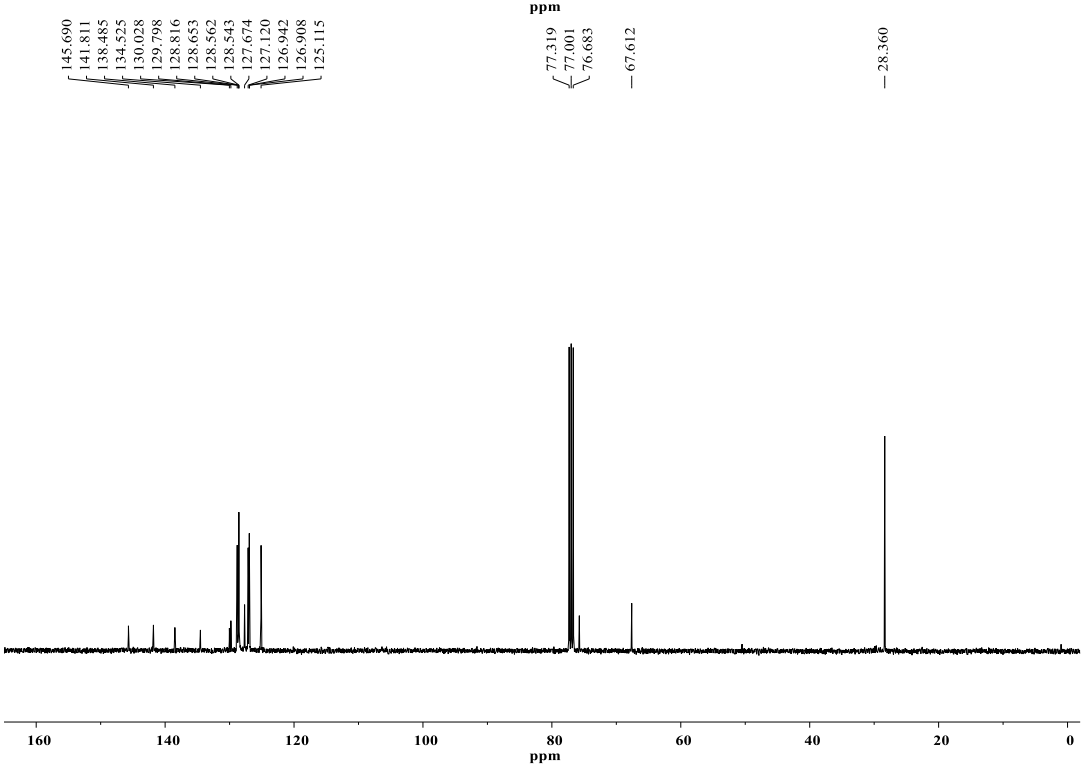
ppm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128.653
128.562
128.543
127.674
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126.908
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77.001
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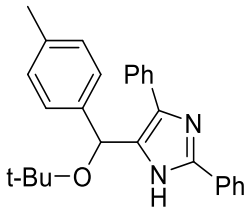
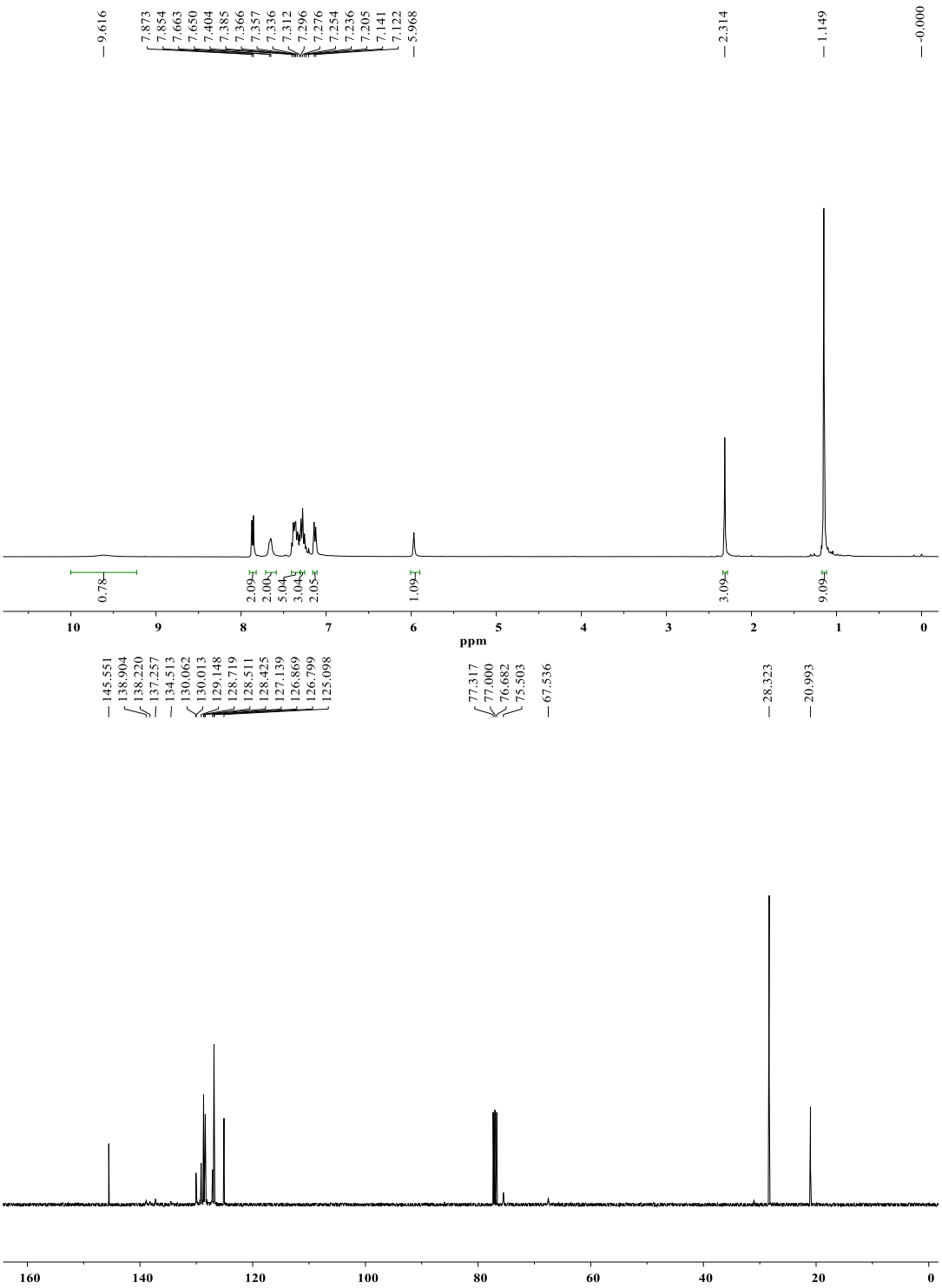
140
120
100
80
60
40
20
0
ppm


Ph
N
t-Bu–O
N
H
Ph

4b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7.236
7.205
7.141
7.122
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2.00
5.04
3.04
2.05
1.09
3.09
9.09
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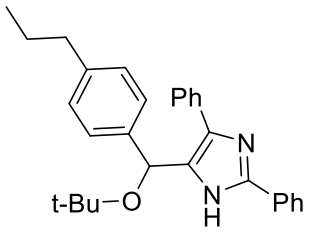
Ph
N
t-Bu–O
N
H
Ph

4c


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7.401
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7.315
7.294
7.274
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7.238
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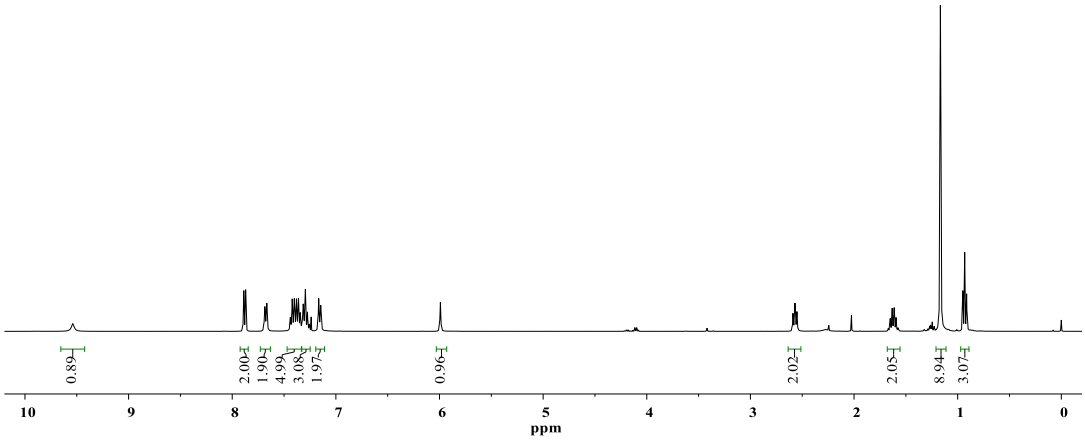
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6
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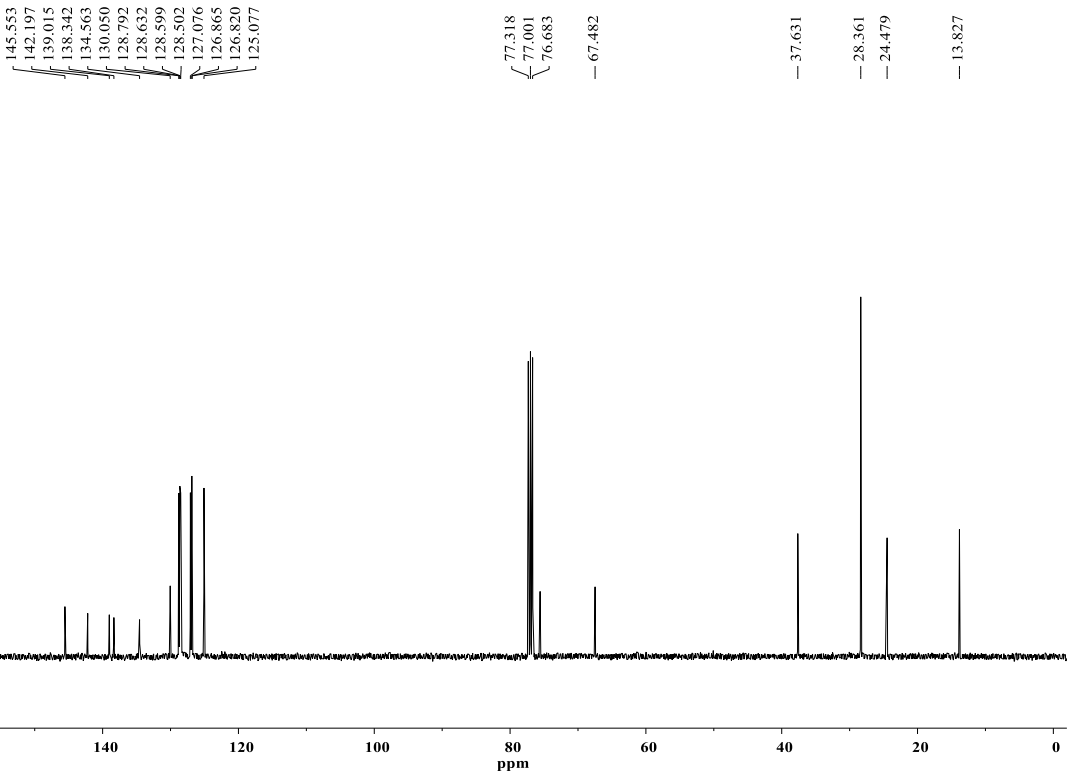
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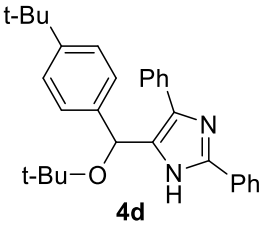
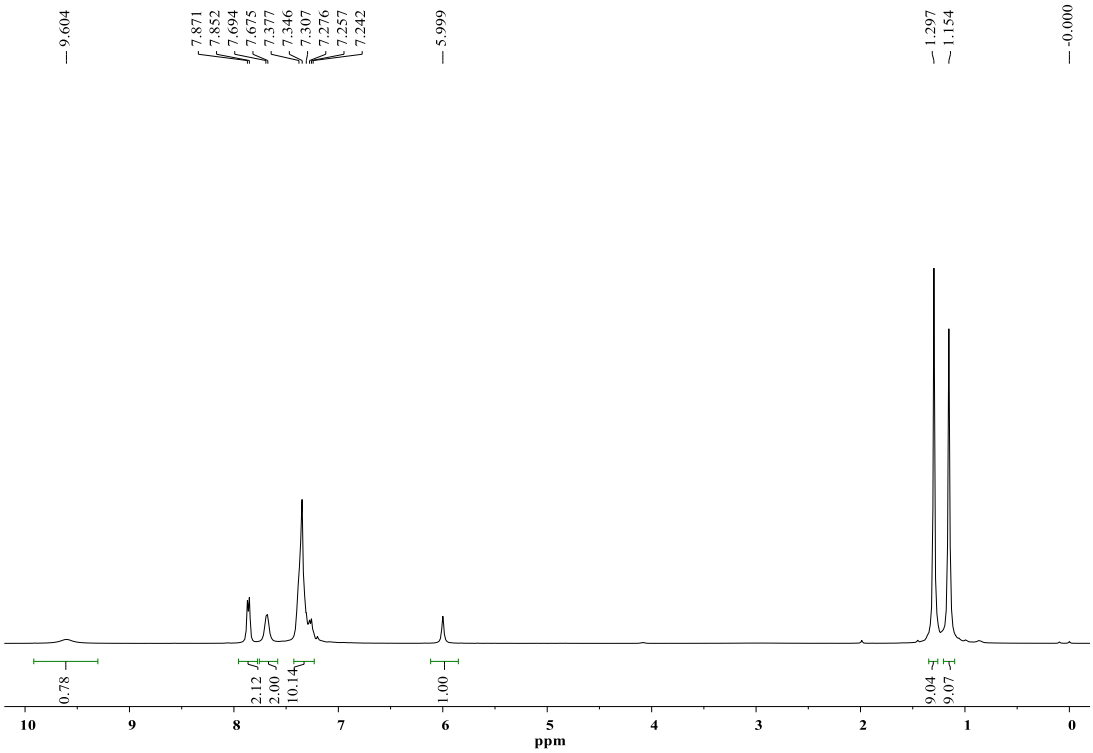

t-Bu
Ph
N
t-Bu–O
N
H
Ph

4d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1.154
0.000
0.78
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10
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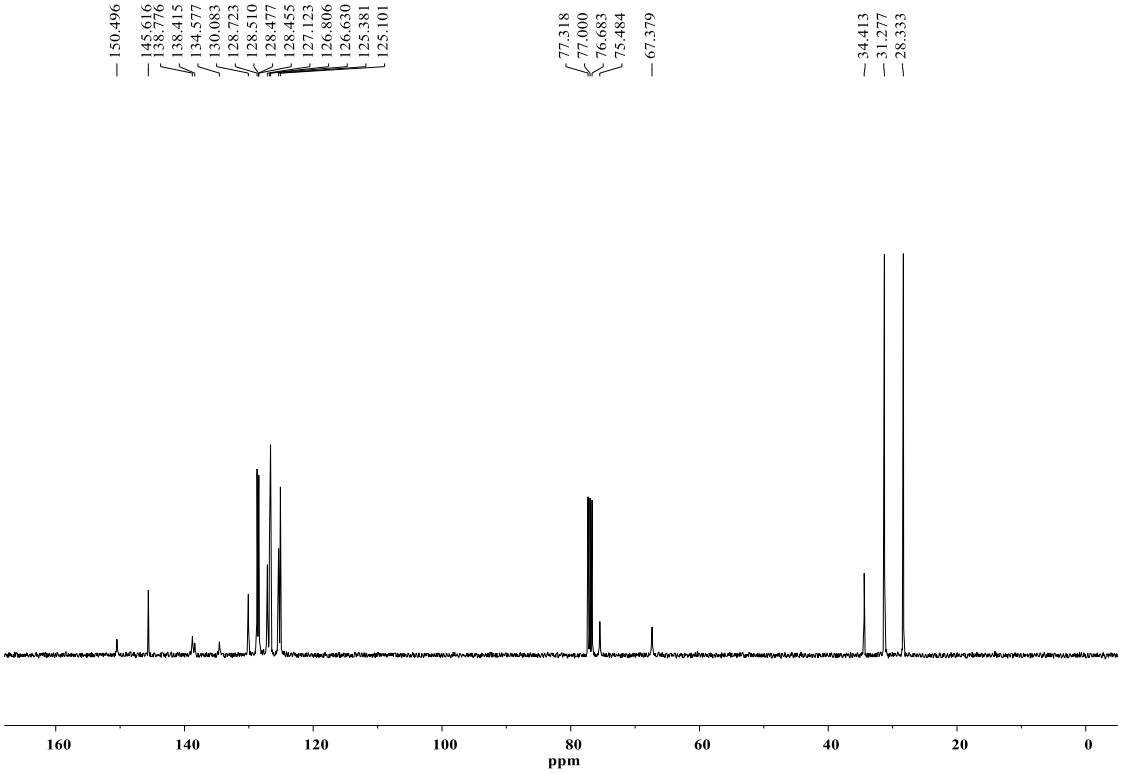
ppm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126.630
125.381
125.101
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77.000
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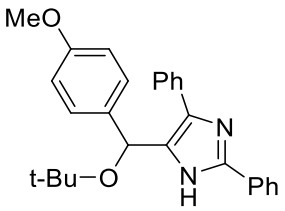
0
ppm


MeO
Ph
N
t-Bu—O
N
H
Ph

4e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7.868
7.636
7.618
7.416
7.398
7.377
7.357
7.339
7.328
7.303
7.281
7.260
7.235
6.862
6.841
5.944
3.762
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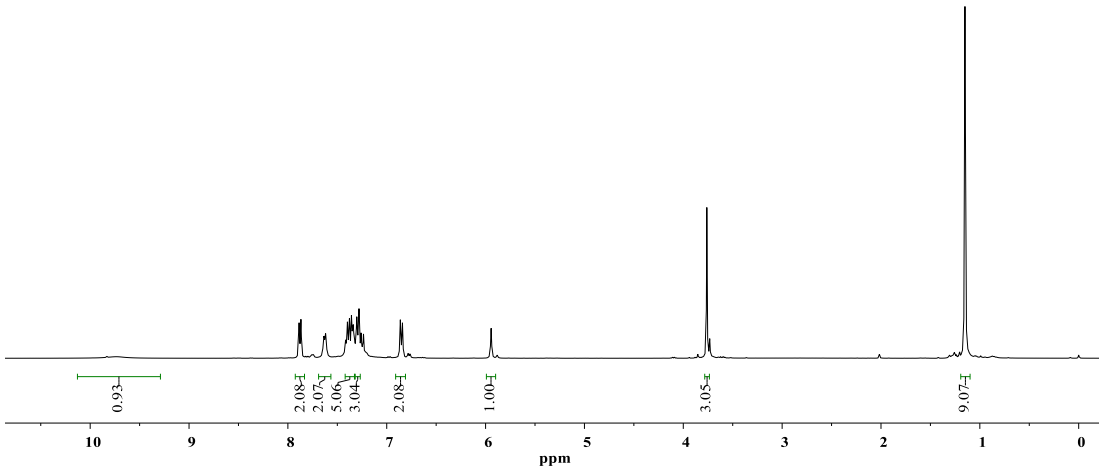
-0.000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2.07
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2.08
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3.05
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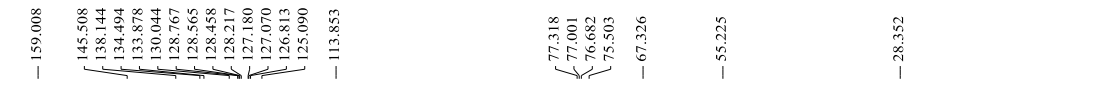
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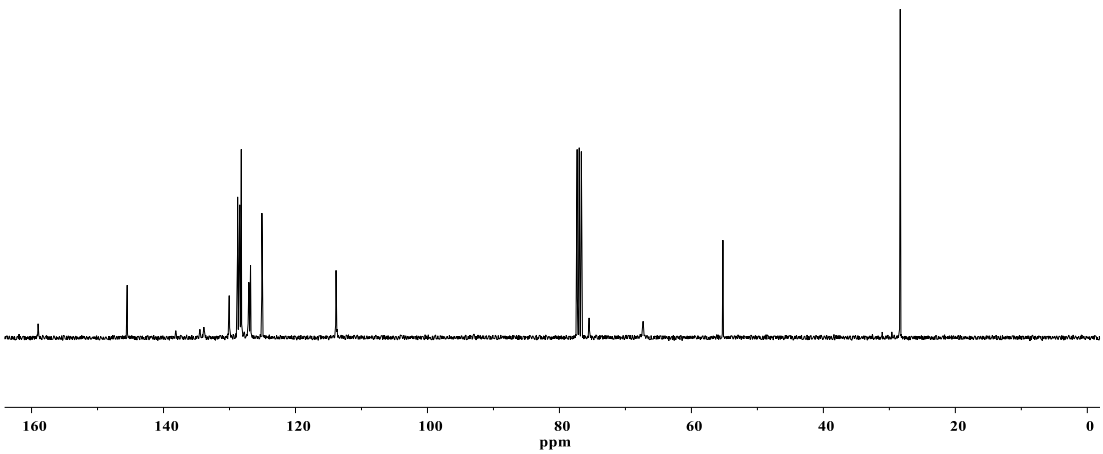
138.144
134.494
133.878
130.044
128.767
128.565
128.458
128.217
127.180
127.070
126.813
125.090
113.853
77.318
77.001
76.682
75.503
67.326
55.225
28.352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100
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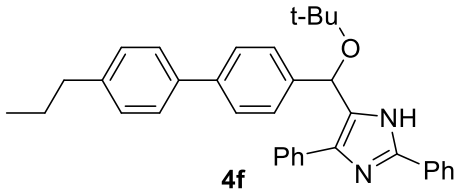
0
ppm


t-Bu
O
NH
Ph
N
Ph

4f


9.666
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7.484
7.463
7.441
7.377
7.324
7.306
7.281
7.262
7.226
7.206
7.183
6.044
2.614
2.595
2.576
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1.661
1.642
1.624
1.605
1.172
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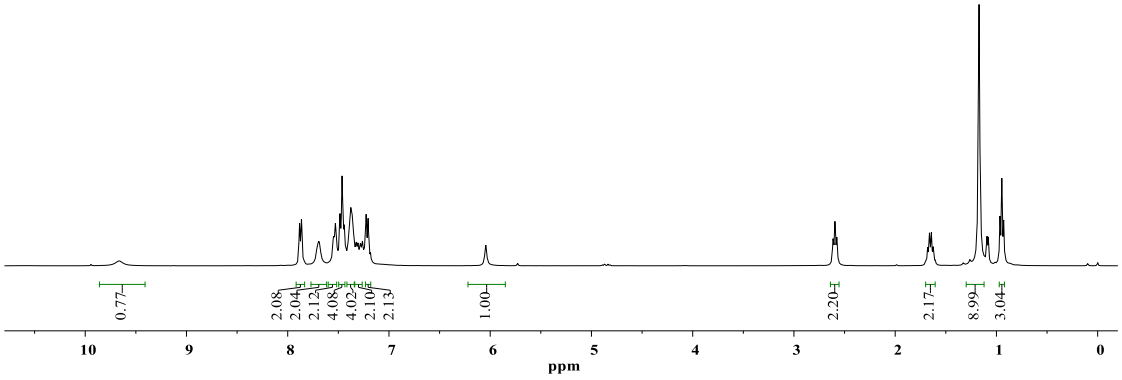
0.928
-0.000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4.02
2.10
2.13
1.00
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3.04
10
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8
7
6
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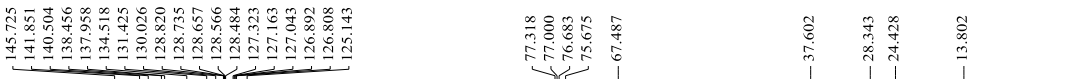
3
2
1
0
ppm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130.026
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128.657
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128.484
127.323
127.163
127.043
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125.143
77.318
77.000
76.683
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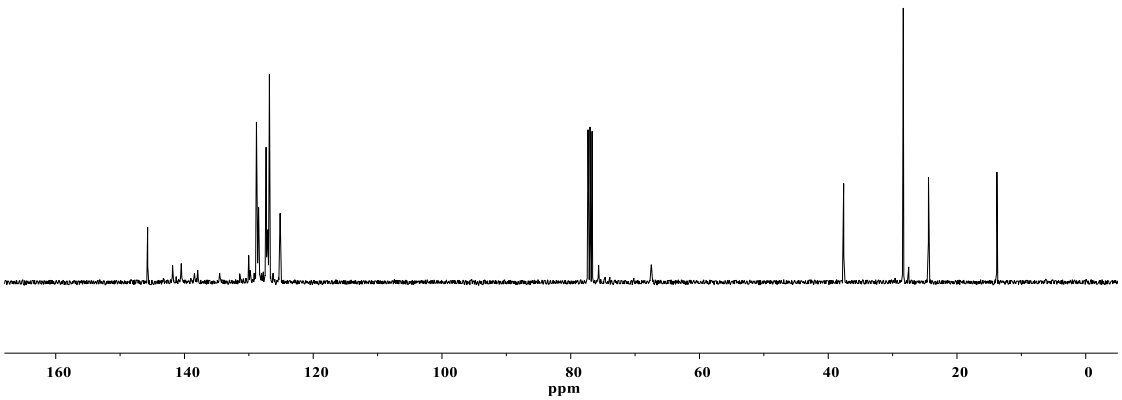
67.487
37.602
28.343
24.428
13.802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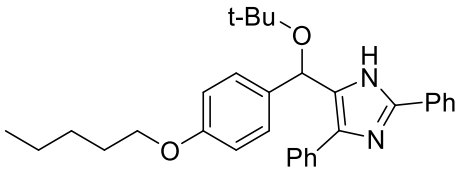
t-Bu
O
H
N
Ph
N
Ph
O

4g


9.629
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7.879
7.658
7.639
7.442
7.424
7.405
7.390
7.370
7.351
7.328
7.295
7.273
7.250
7.243
6.869
6.848
5.951
3.943
3.927
3.910
1.799
1.782
1.765
1.747
1.729
1.458
1.442
1.422
1.404
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1.373
1.355
1.336
1.317
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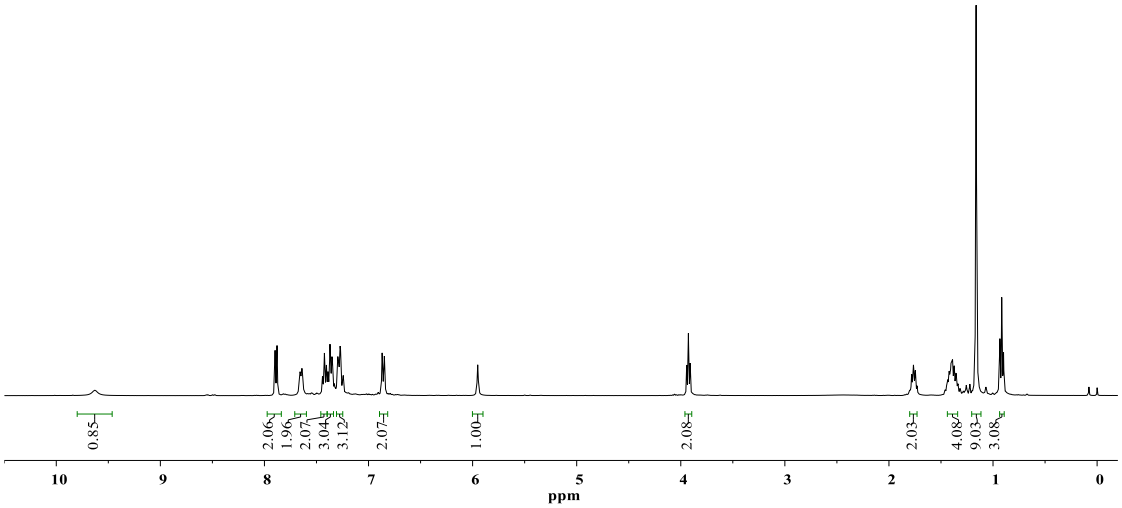
0.934
0.916
0.899
0.000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3.12
2.07
1.00
2.08
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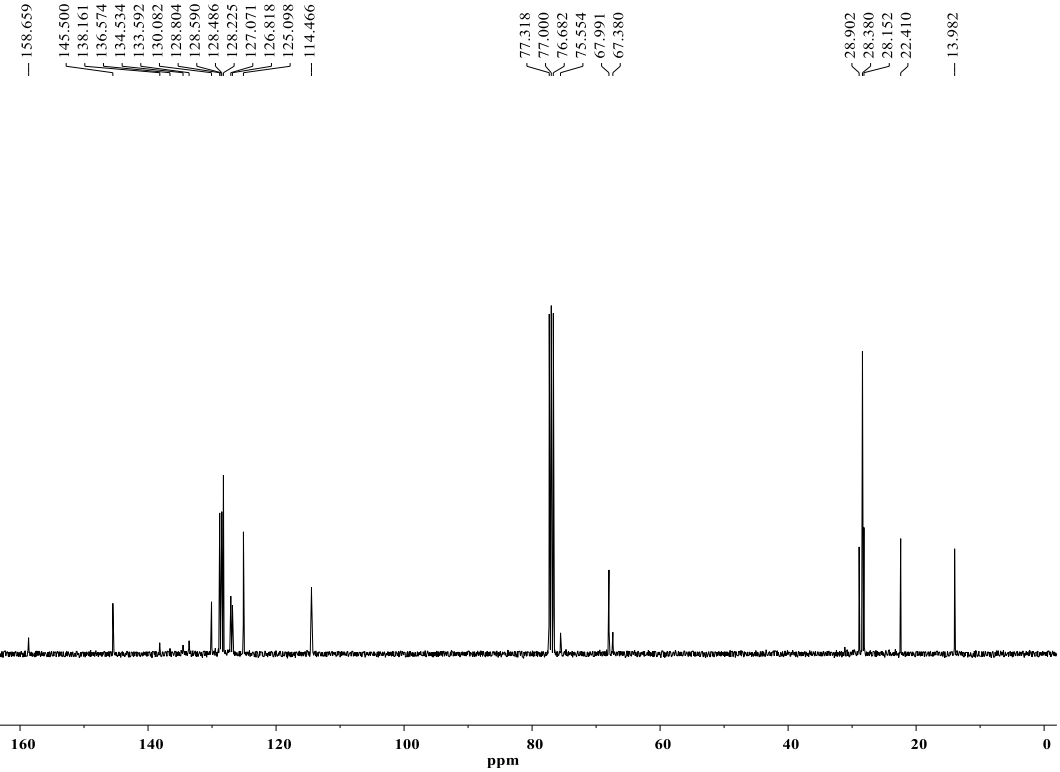
9.03
3.08
10
9
8
7
6
5
4
3
2
1
0
ppm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133.592
130.082
128.804
128.590
128.486
128.225
127.071
126.818
125.098
114.466
77.318
77.000
76.682
75.554
67.991
67.380
28.902
28.380
28.152
22.410
13.982
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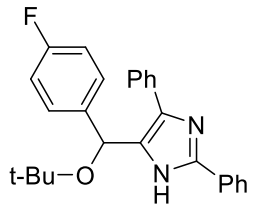
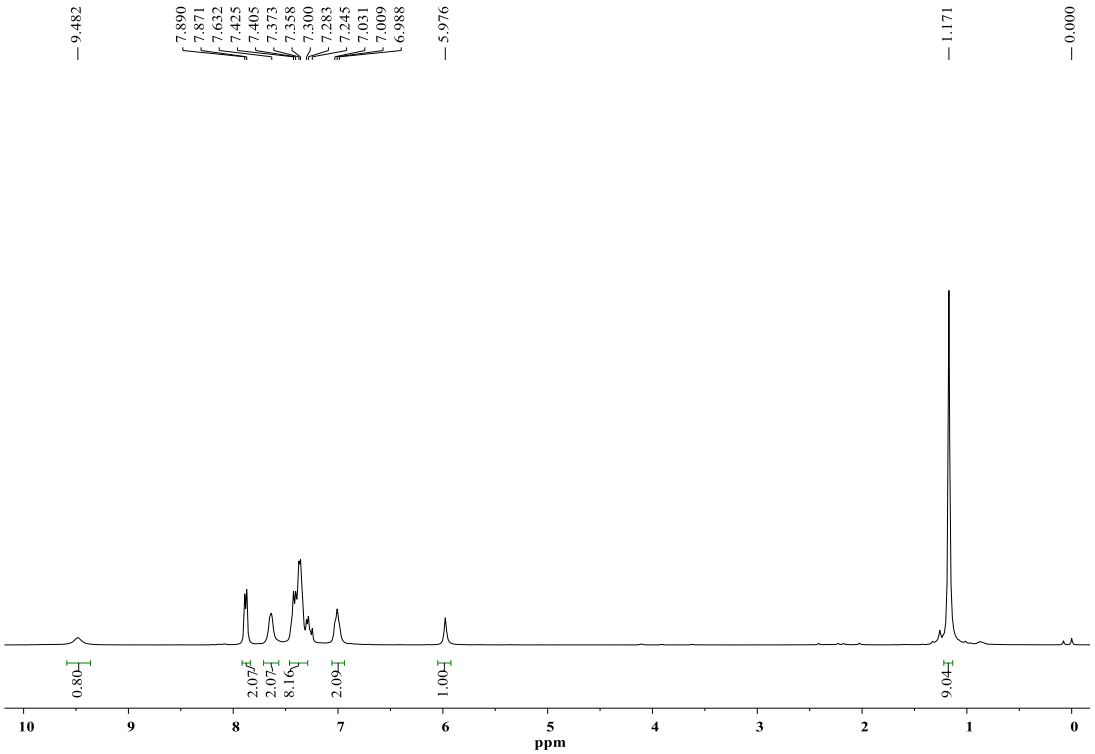
140
120
100
80
60
40
20
0
ppm


F
Ph
N
t-Bu–O
N
H
Ph

4h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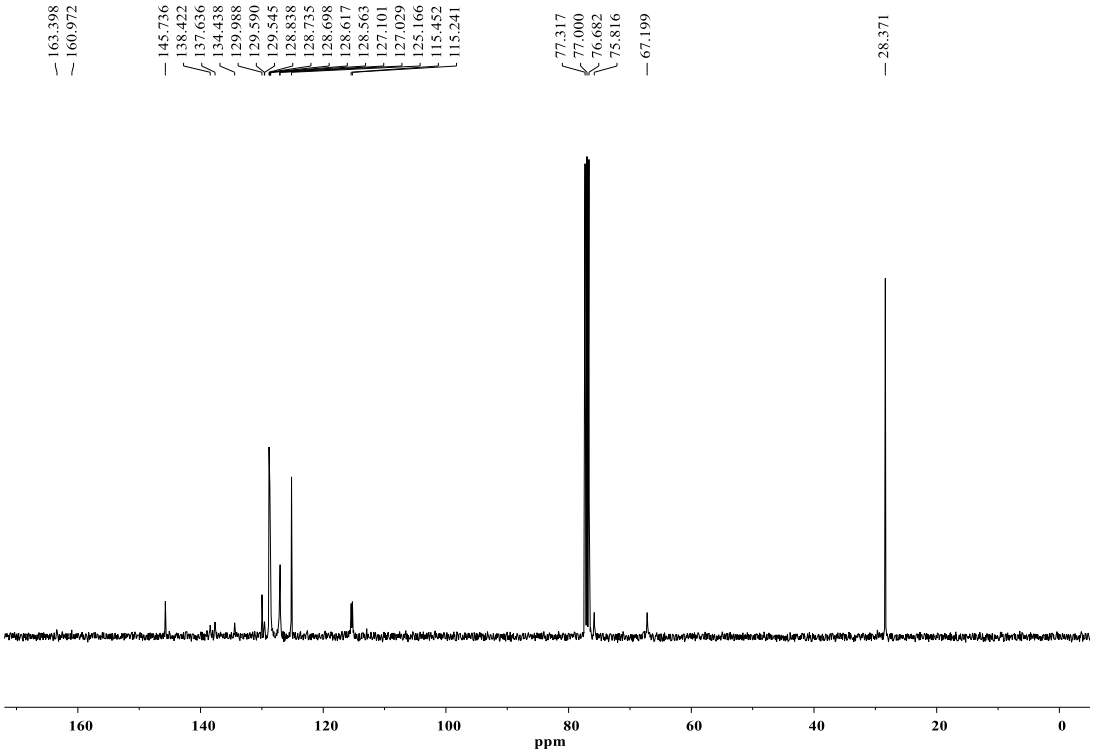
7.871
7.632
7.425
7.405
7.373
7.358
7.300
7.283
7.245
7.031
7.009
6.988
5.976
1.171
0.000
0.80
2.07
2.07
8.16
2.09
1.00
9.04
ppm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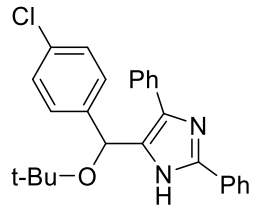
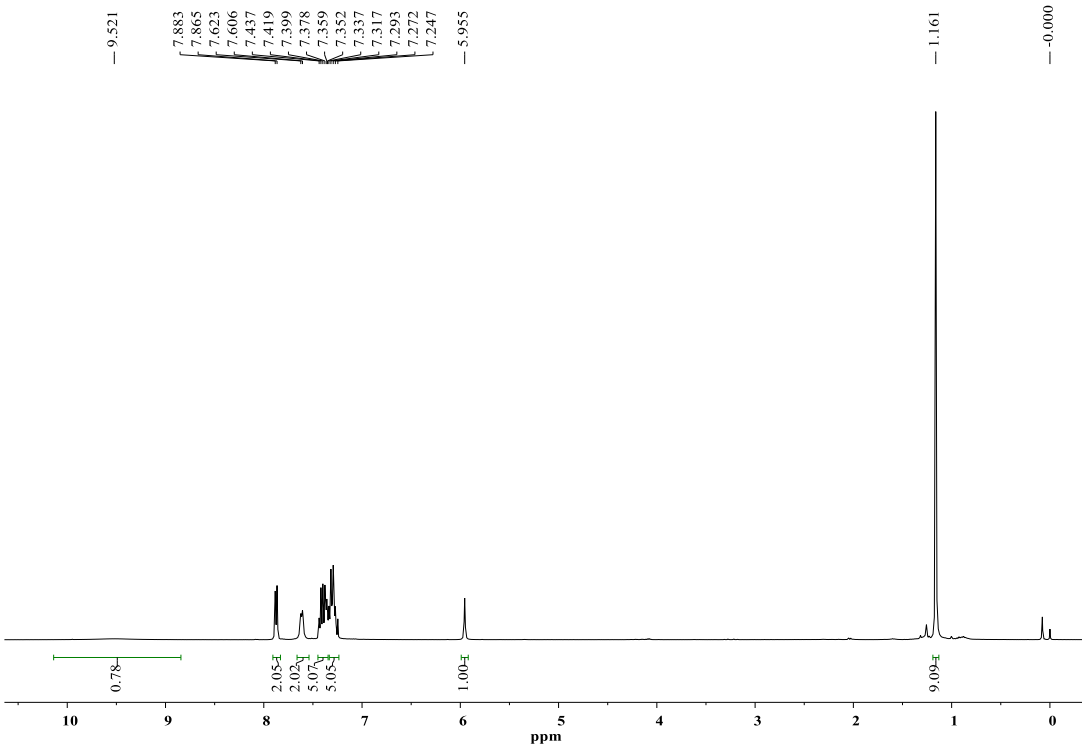
145.736
138.422
137.636
134.438
129.988
129.590
129.545
128.838
128.735
128.698
128.617
128.563
127.101
127.029
125.166
115.452
115.241
77.317
77.000
76.682
75.816
67.199
28.371
ppm


Cl
Ph
N
t-Bu−O
N
H
Ph

4i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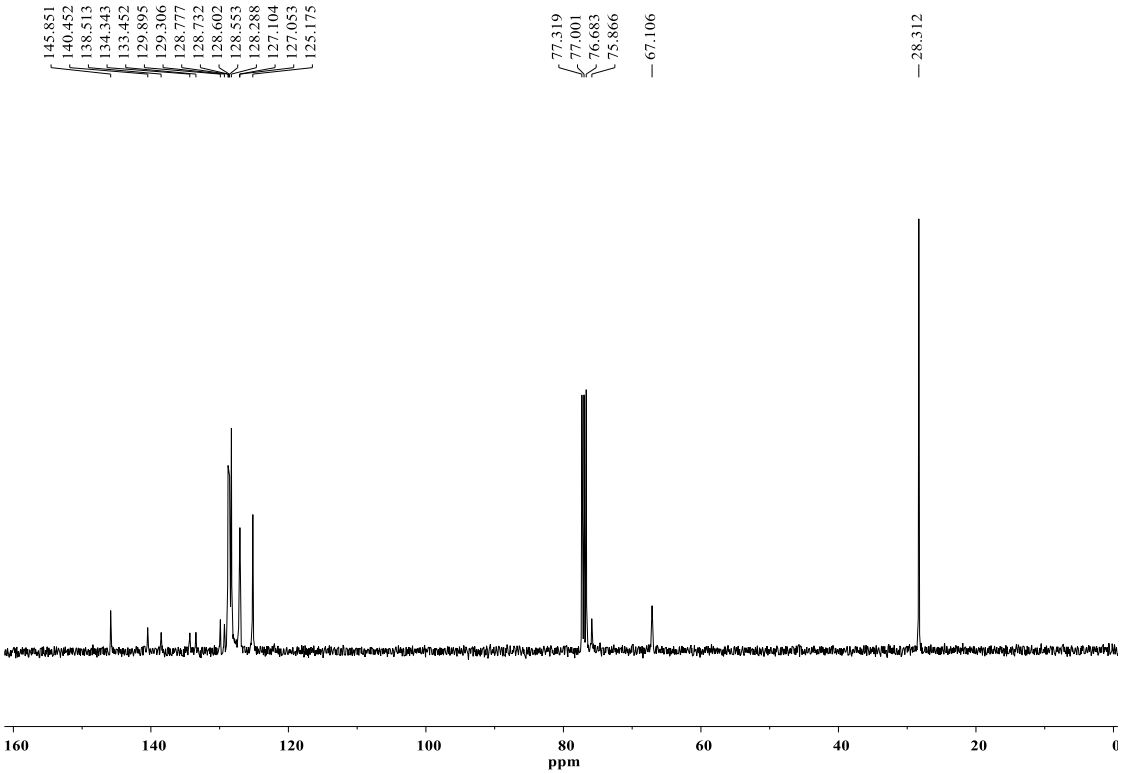
7.606
7.437
7.419
7.399
7.378
7.359
7.352
7.337
7.317
7.293
7.272
7.247
5.955
1.161
-0.000
0.78
2.05
2.02
5.07
5.05
1.00
9.09
10
9
8
7
6
5
4
3
2
1
0
ppm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134.343
133.452
129.895
129.306
128.777
128.732
128.602
128.553
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127.104
127.053
125.175
77.319
77.001
76.683
75.866
67.106
28.312
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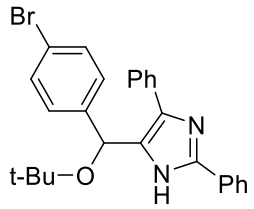
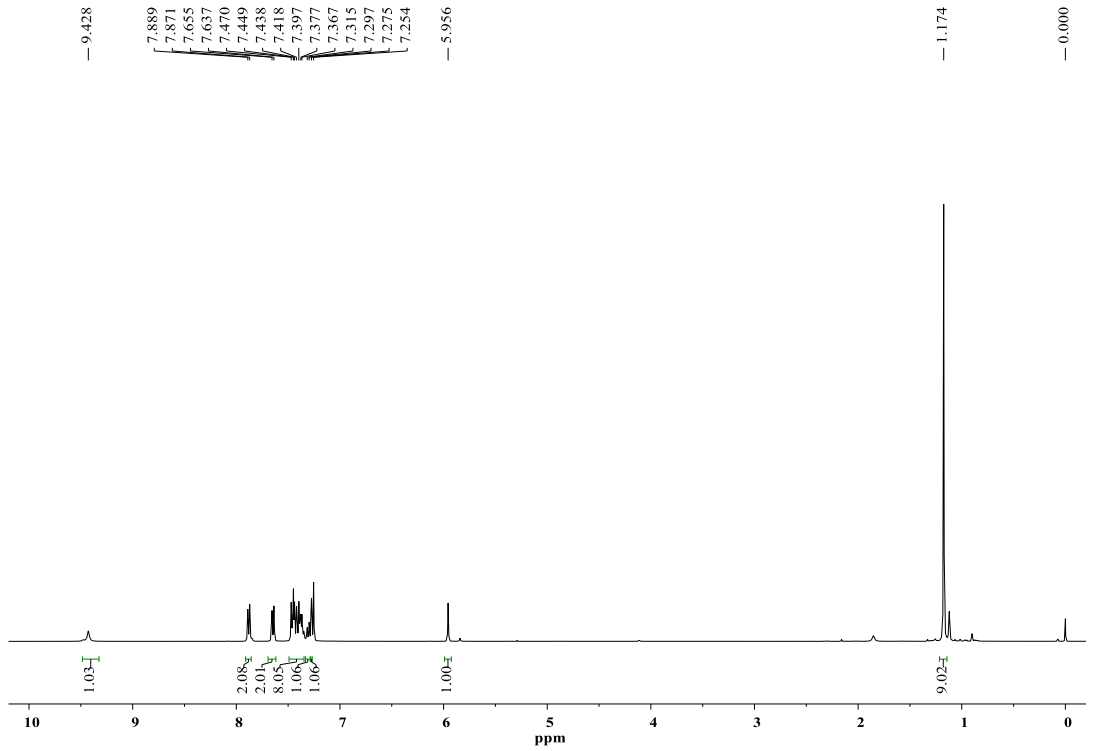
140
120
100
80
60
40
20
ppm


Br
Ph
N
t-Bu−O
N
H
Ph

4j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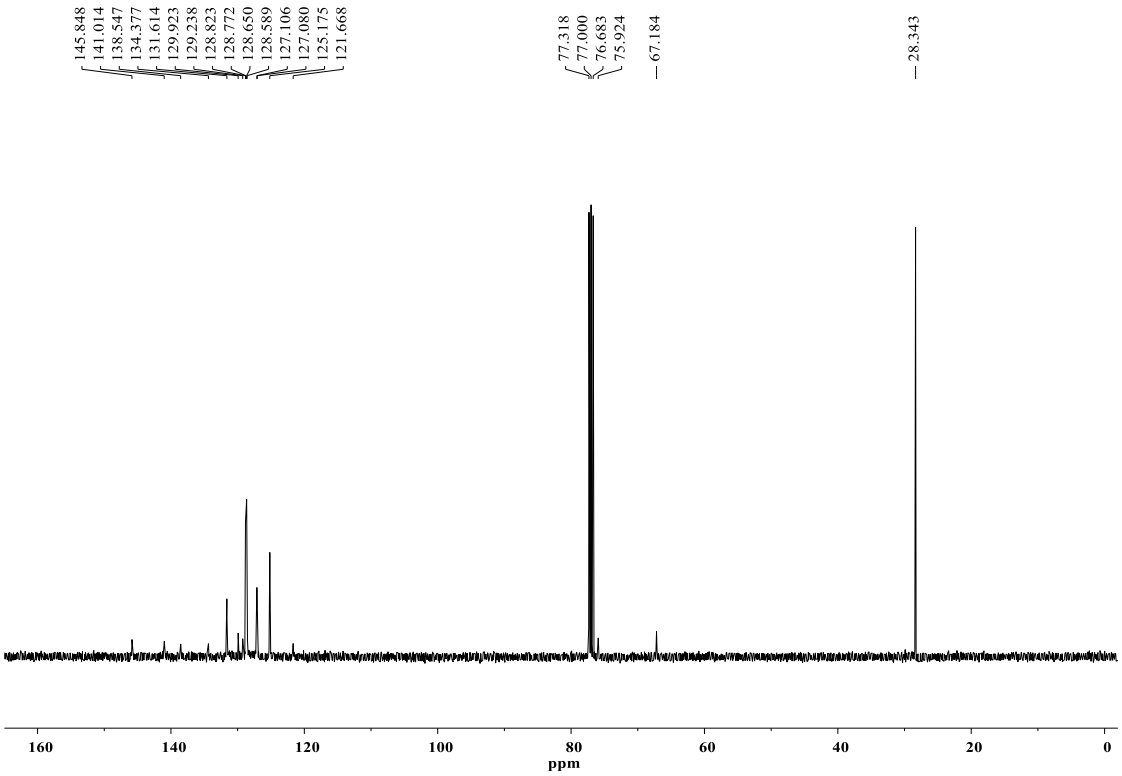
7.438
7.418
7.397
7.377
7.367
7.315
7.297
7.275
7.254
5.956
1.174
0.000
1.03
2.08
2.01
8.05
1.06
1.06
1.00
9.02
10
9
8
7
6
5
4
3
2
1
0
ppm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134.377
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129.923
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128.823
128.772
128.650
128.589
127.106
127.080
125.175
121.668
77.318
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76.683
75.924
67.184
28.343
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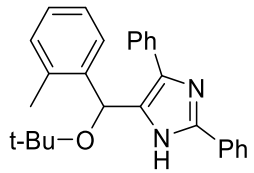
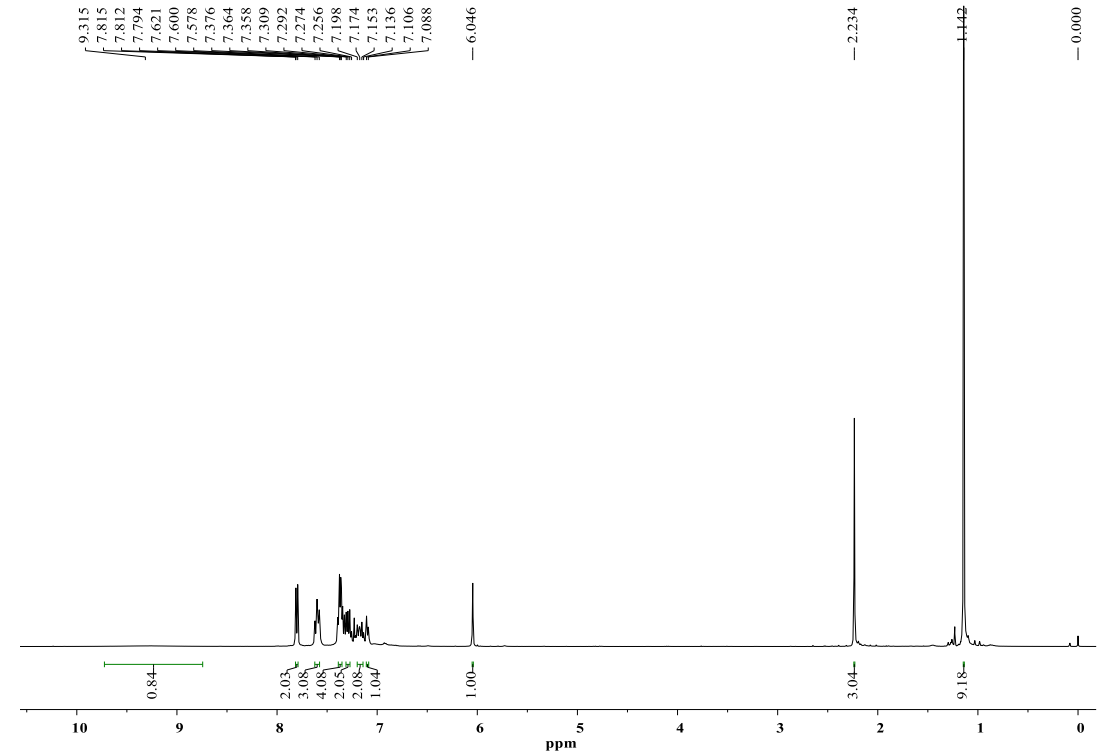
140
120
100
80
60
40
20
0
ppm


Ph
N
t-Bu−O
N
H
Ph

4k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7.812
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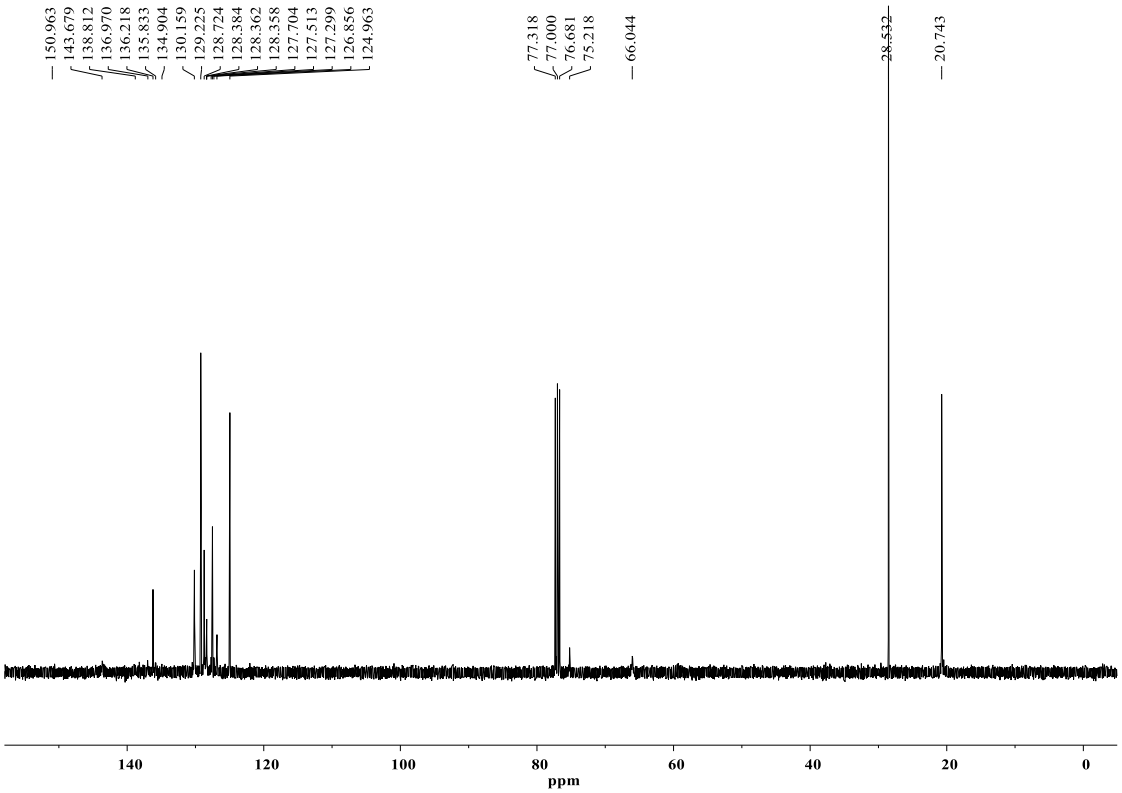
7.600
7.578
7.376
7.364
7.358
7.309
7.292
7.274
7.256
7.198
7.174
7.153
7.136
7.106
7.088
6.046
2.234
1.142
0.000
0.84
2.03
3.08
4.08
2.05
2.08
1.04
1.00
3.04
9.18
10
9
8
7
6
5
4
3
2
1
0
ppm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136.970
136.218
135.833
134.904
130.159
129.225
128.724
128.384
128.362
128.358
127.704
127.513
127.299
126.856
124.963
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77.000
76.681
75.218
66.044
28.532
20.743
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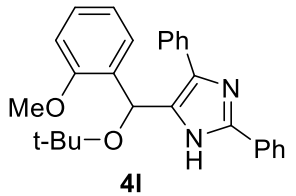
120
100
80
60
40
20
0
ppm


Ph
N
MeO
t-Bu–O
N
H
Ph

4l


7.844
7.826
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7.740
7.538
7.389
7.370
7.351
7.304
7.286
7.266
7.248
7.235
7.230
7.216
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6.943
6.924
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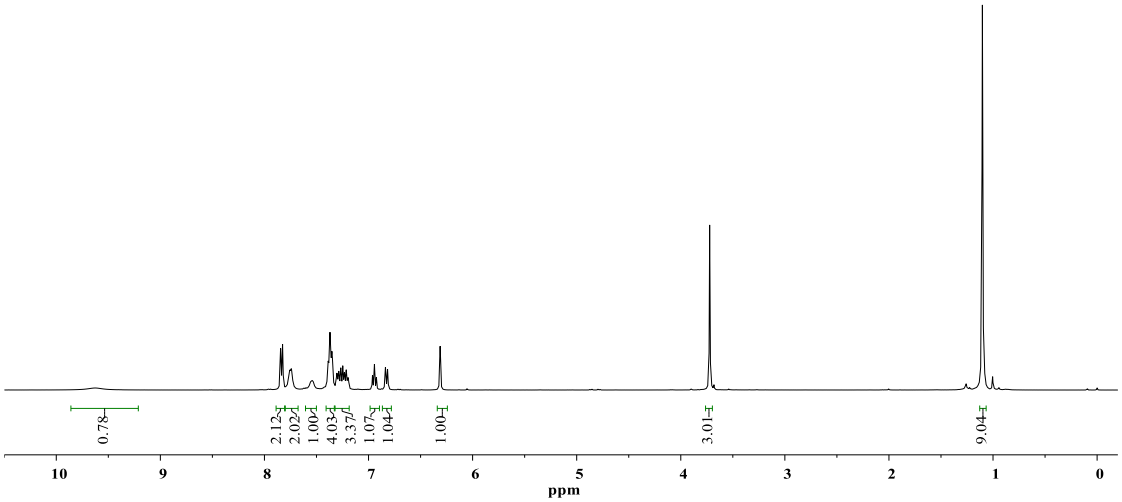
6.817
6.312
3.722
1.102
-0.000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4.03
3.37
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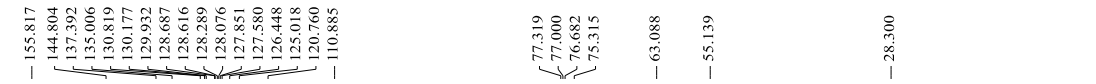
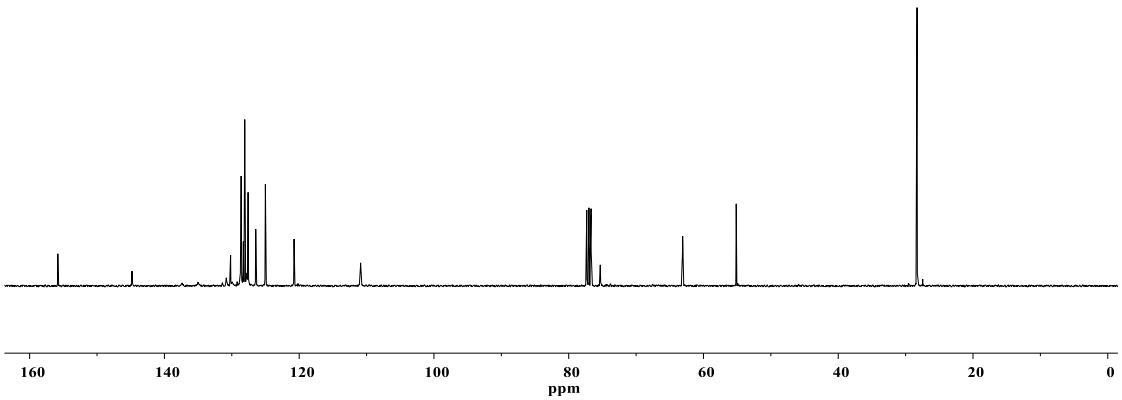
1.04
1.00
3.01
9.04
10
9
8
7
6
5
4
3
2
1
0
ppm


155.817
144.804
137.392
135.006
130.819
130.177
129.932
128.687
128.616
128.289
128.076
127.851
127.580
126.448
125.018
120.760
110.885
77.319
77.000
76.682
75.315
63.088
55.139
28.300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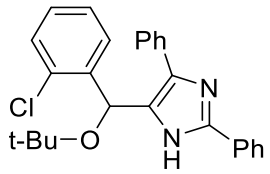
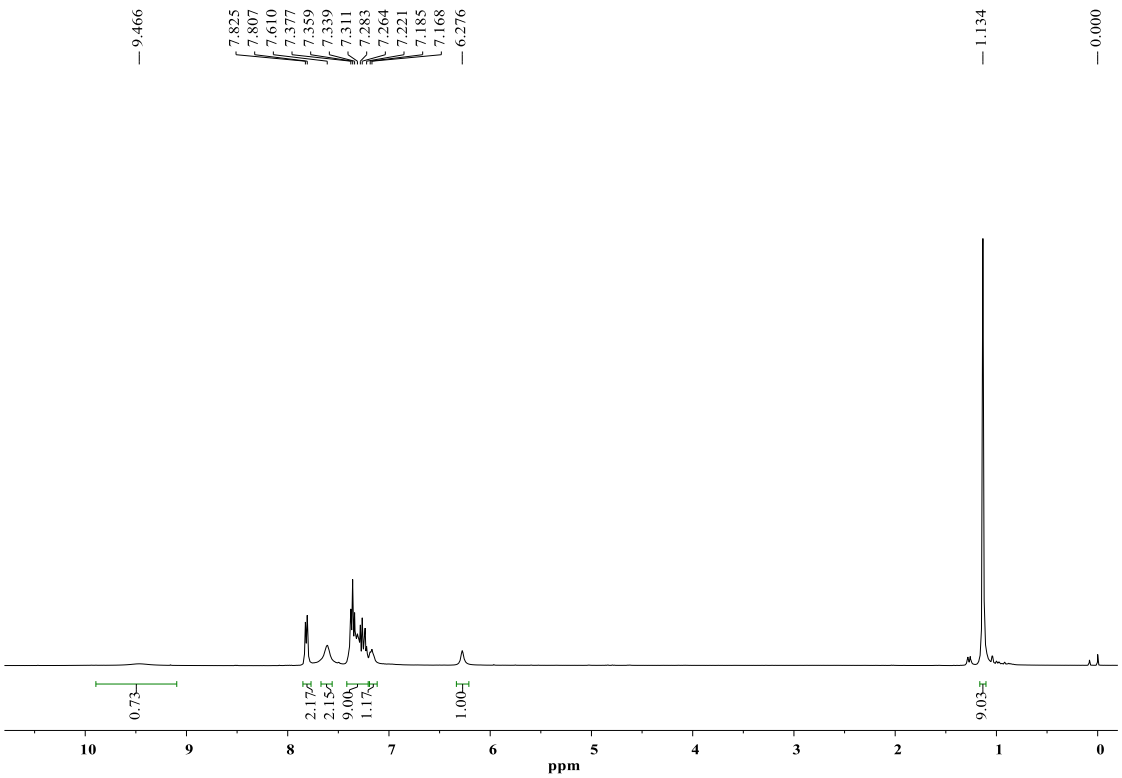
140
120
100
80
60
40
20
0
ppm


Ph
N
Cl
t-Bu—O
N
H
Ph

4m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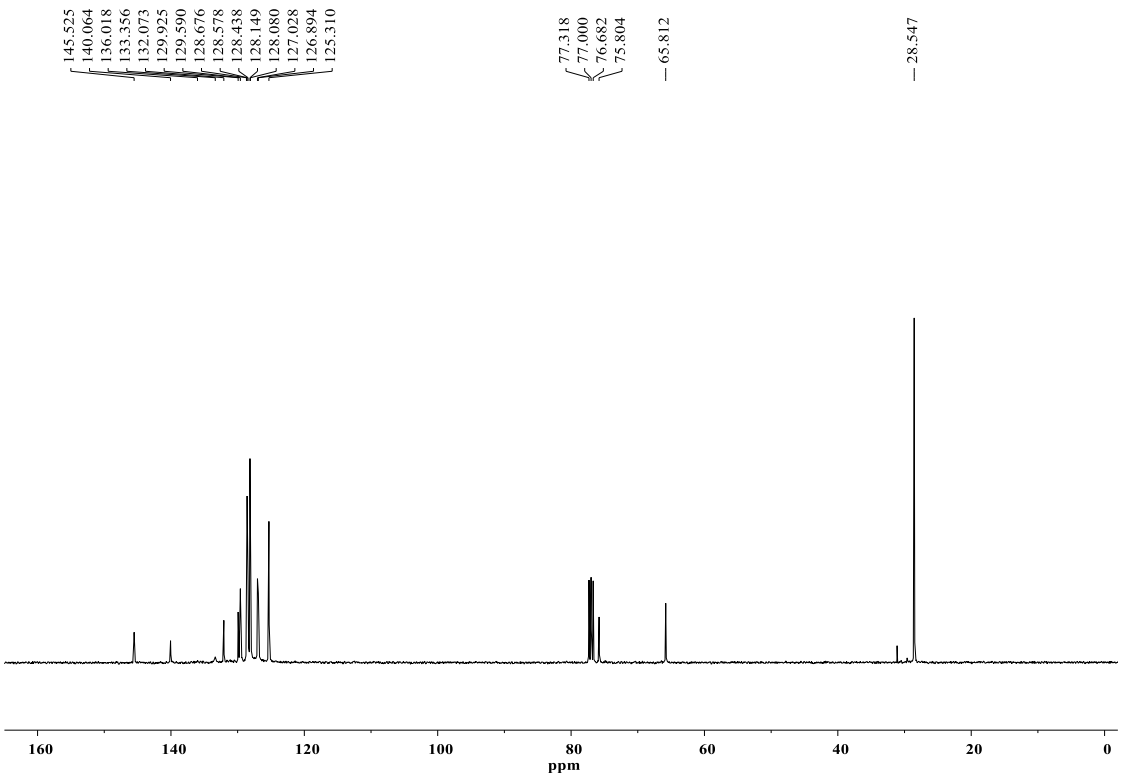
7.377
7.359
7.339
7.311
7.283
7.264
7.221
7.185
7.168
6.276
1.134
0.000
0.73
2.17
2.15
9.00
1.17
1.00
9.03
10
9
8
7
6
5
4
3
2
1
0
ppm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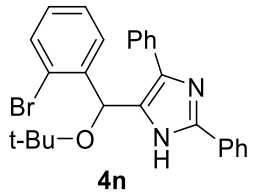
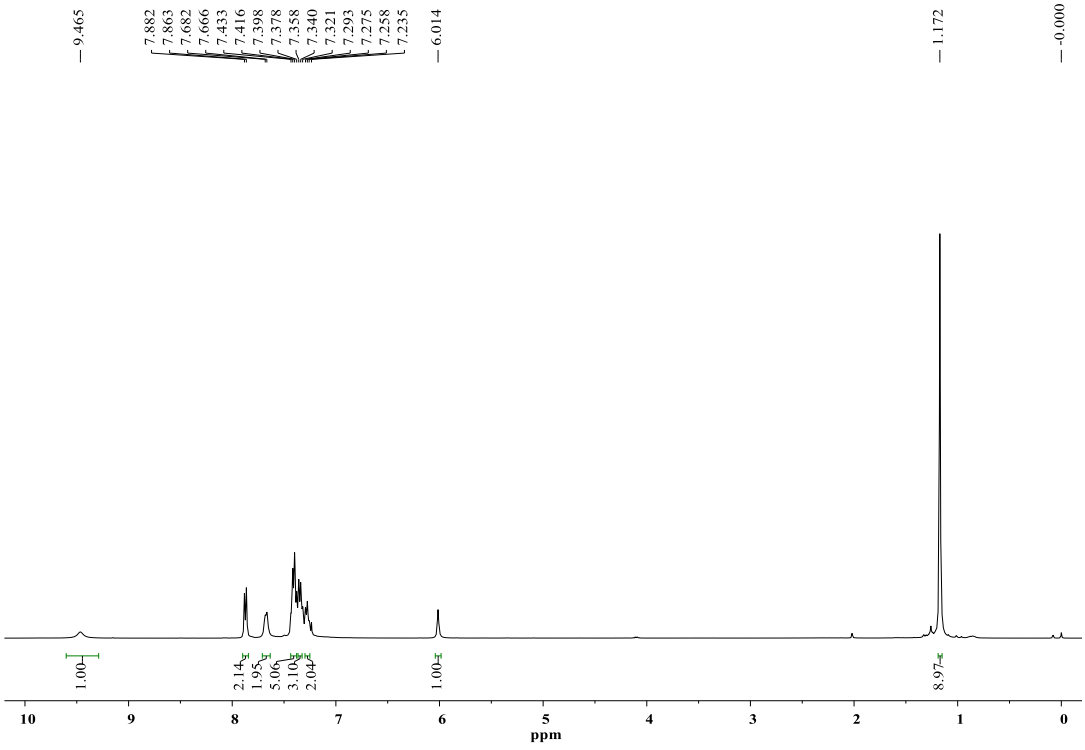
132.073
129.925
129.590
128.676
128.578
128.438
128.149
128.080
127.028
126.894
125.310
77.318
77.000
76.682
75.804
65.812
28.547
160
140
120
100
80
60
40
20
0
ppm


Ph
Br
t-Bu–O
N
N
H
Ph

4n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7.398
7.378
7.358
7.340
7.321
7.293
7.275
7.258
7.235
6.014
1.172
-0.000
1.00
2.14
1.95
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2.04
1.00
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10
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7
6
5
4
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2
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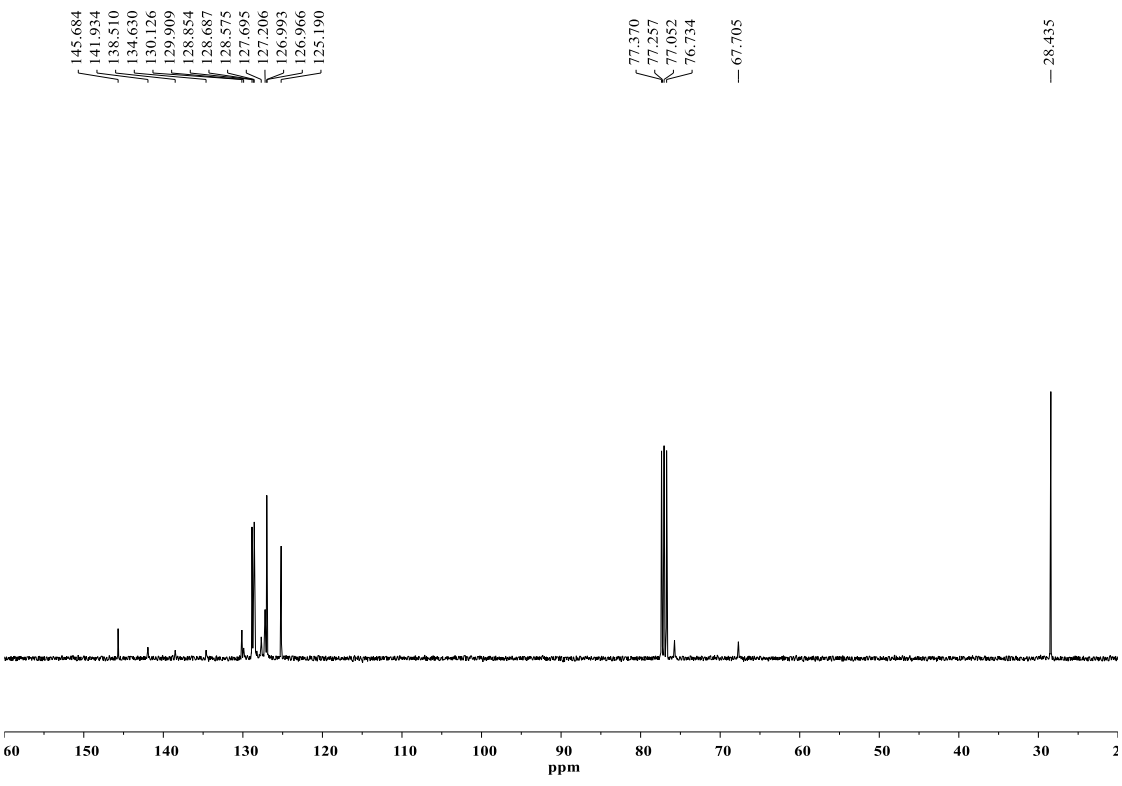
0
ppm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129.909
128.854
128.687
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127.695
127.206
126.993
126.966
125.190
77.370
77.257
77.052
76.734
67.705
28.435
60
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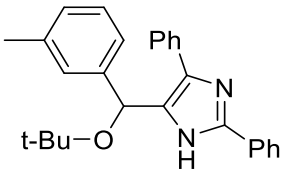
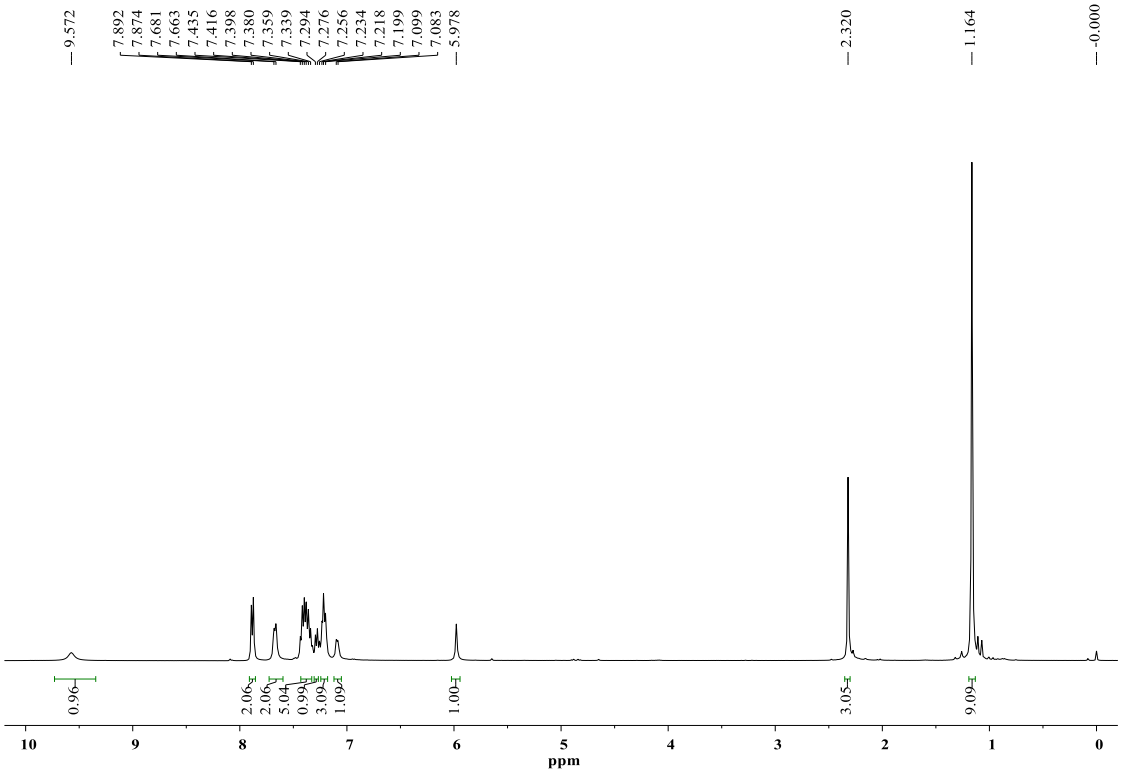
140
130
120
110
100
90
80
70
60
50
40
30
ppm


Ph
N
t-Bu—O
N
H
Ph

4o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7.359
7.339
7.294
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7.218
7.199
7.099
7.083
5.978
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1.164
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0.96
2.06
2.06
5.04
0.99
3.09
1.09
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3.05
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10
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6
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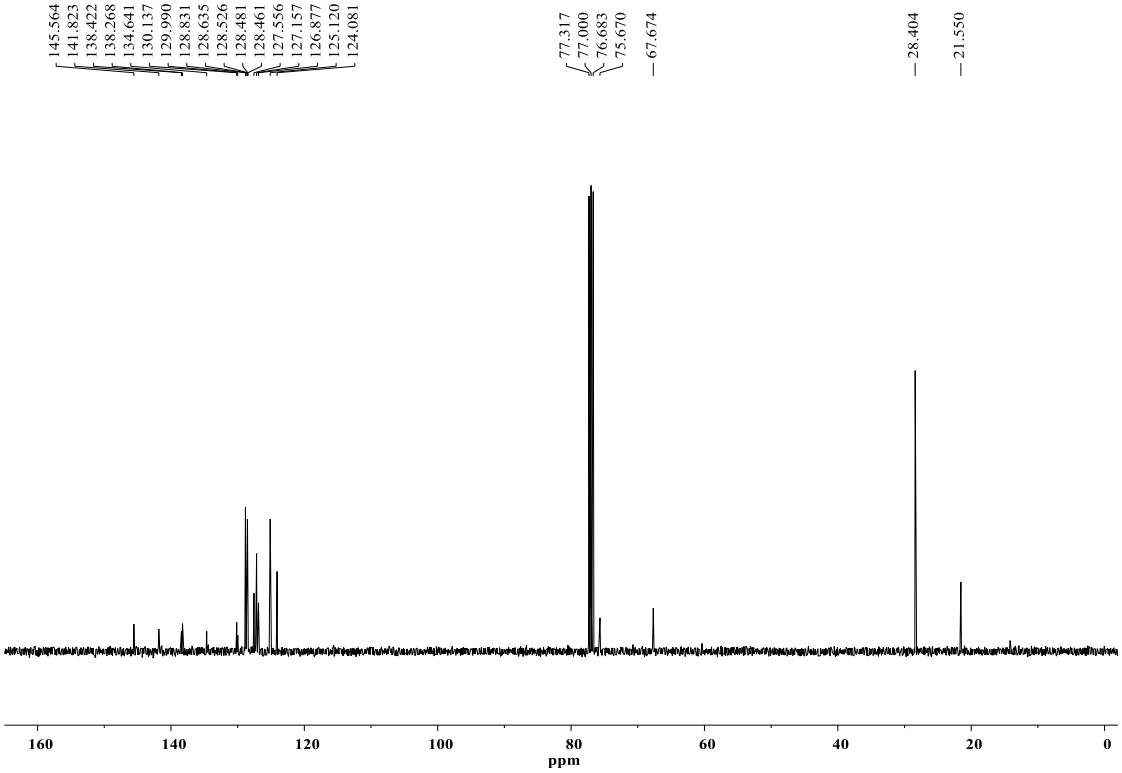
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128.831
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127.556
127.157
126.877
125.120
124.081
77.317
77.000
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75.670
67.674
28.404
21.550
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120
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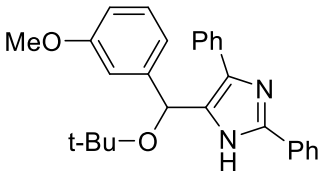
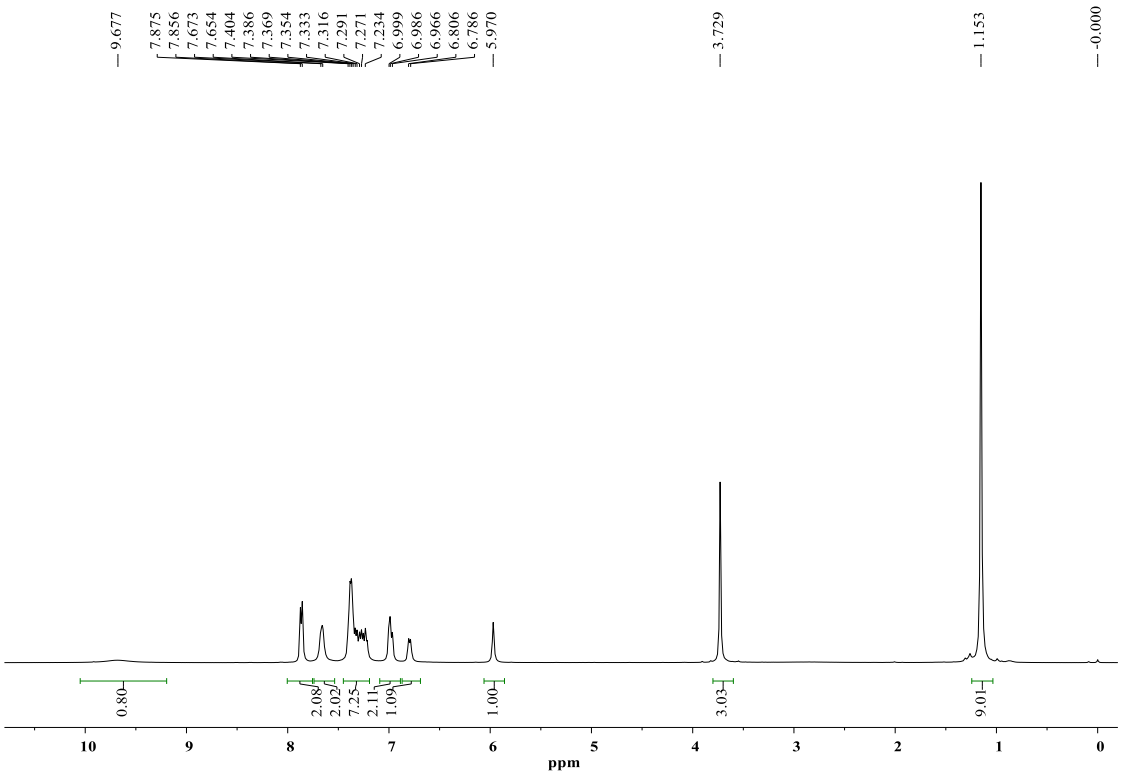

MeO
Ph
N
t-Bu−O
N
H
Ph

4p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7.369
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7.333
7.316
7.291
7.271
7.234
6.999
6.986
6.966
6.806
6.786
5.970
3.729
1.153
-0.000
0.80
2.08
2.02
7.25
2.11
1.09
1.00
3.03
9.01
10
9
8
7
6
5
4
3
2
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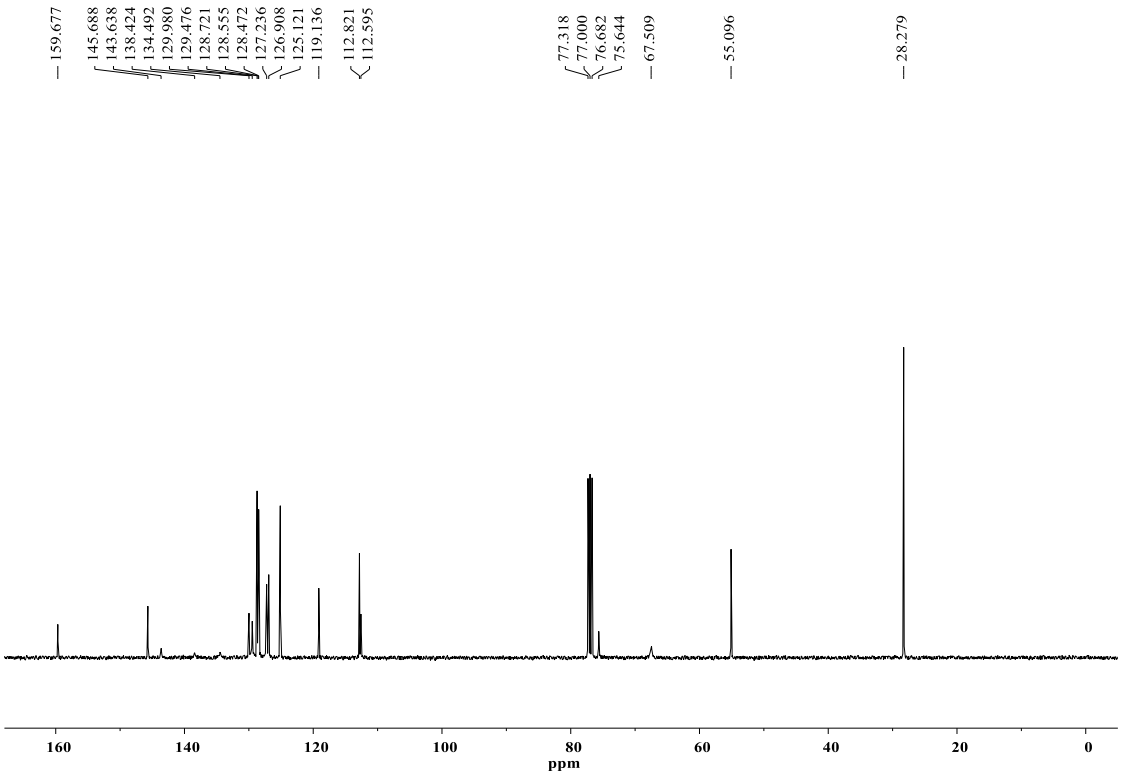
0
ppm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128.721
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128.472
127.236
126.908
125.121
119.136
112.821
112.595
77.318
77.000
76.682
75.644
67.509
55.096
28.279
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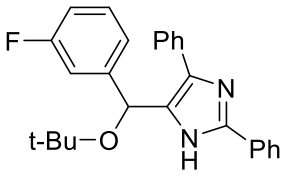
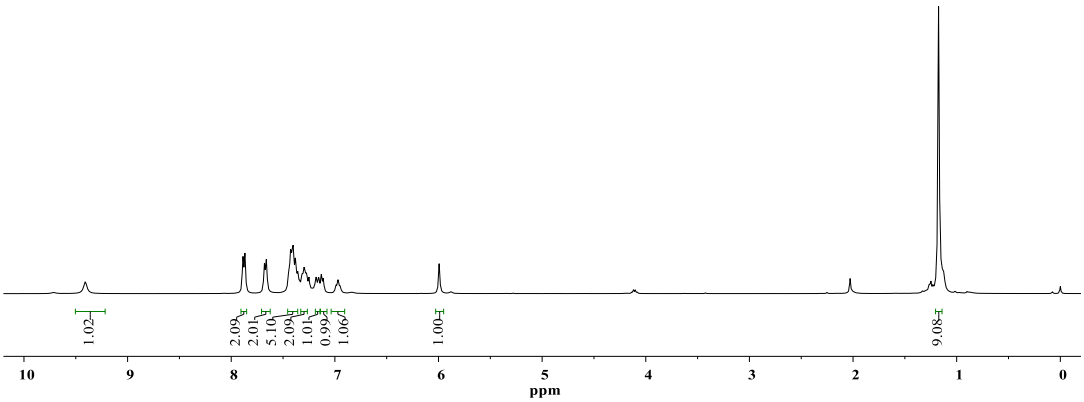
120
100
80
60
40
20
0
ppm


F
Ph
N
t-Bu–O
N
H
Ph

4q


9.408
7.887
7.868
7.680
7.661
7.428
7.410
7.401
7.380
7.358
7.317
7.298
7.290
7.274
7.182
7.157
7.132
7.112
6.994
6.988
6.972
6.966
6.952
5.994
1.175
0.000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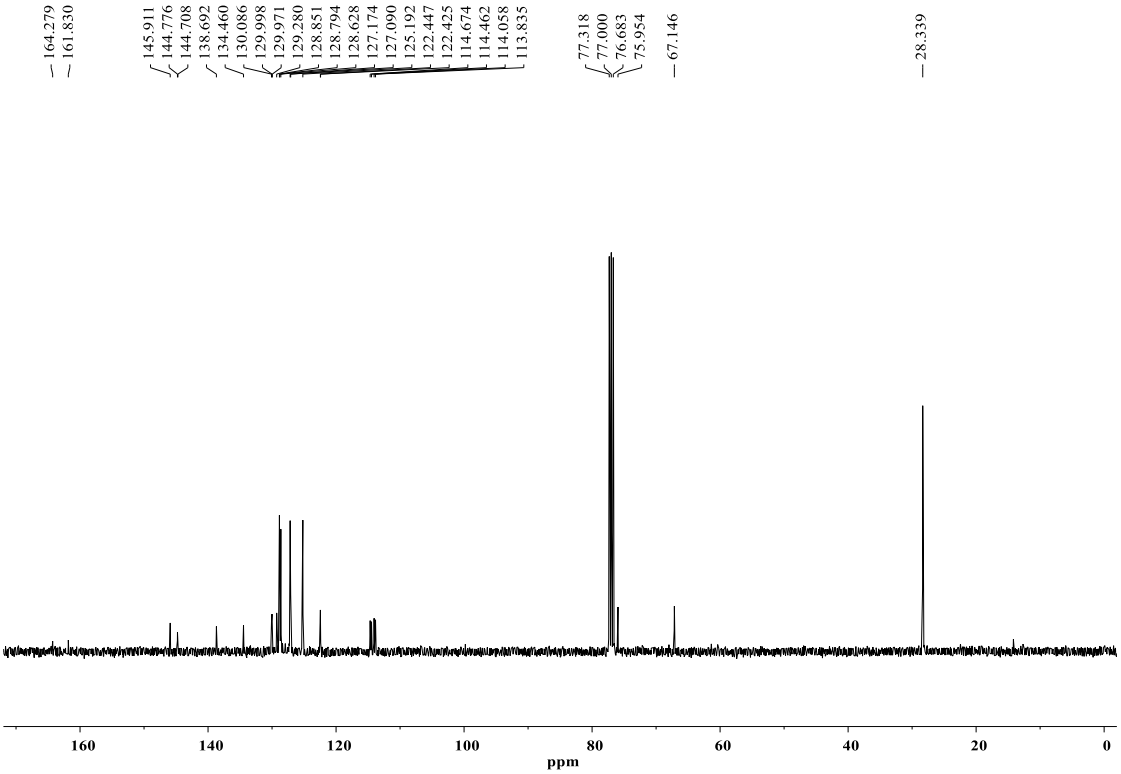
2.09
2.01
5.10
2.09
1.01
0.99
1.06
1.00
9.08
10
9
8
7
6
5
4
3
2
1
0
ppm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145.911
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138.692
134.460
130.086
129.998
129.971
129.280
128.851
128.794
128.628
127.174
127.090
125.192
122.447
122.425
114.674
114.462
114.058
113.835
77.318
77.000
76.683
75.954
67.146
28.339
160
140
120
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60
40
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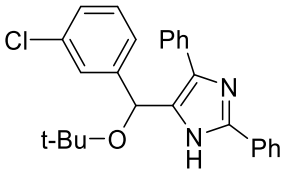
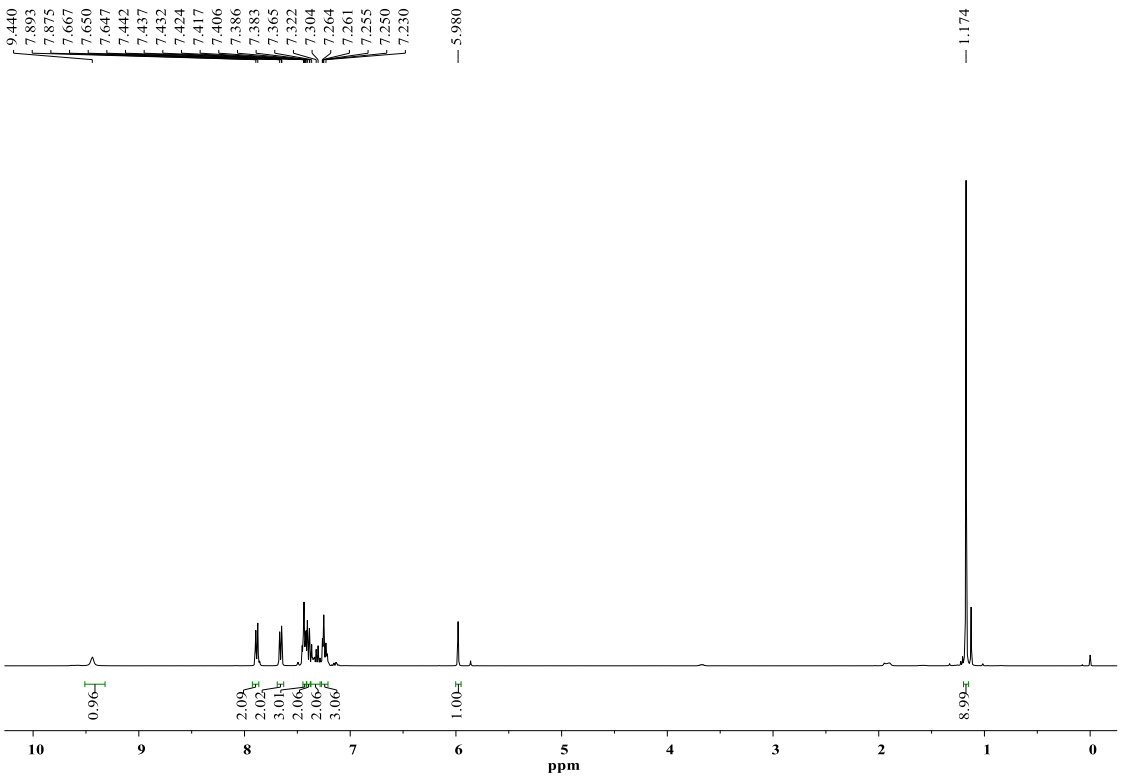
0
ppm


Cl
Ph
N
t-Bu–O
N
H
Ph

4r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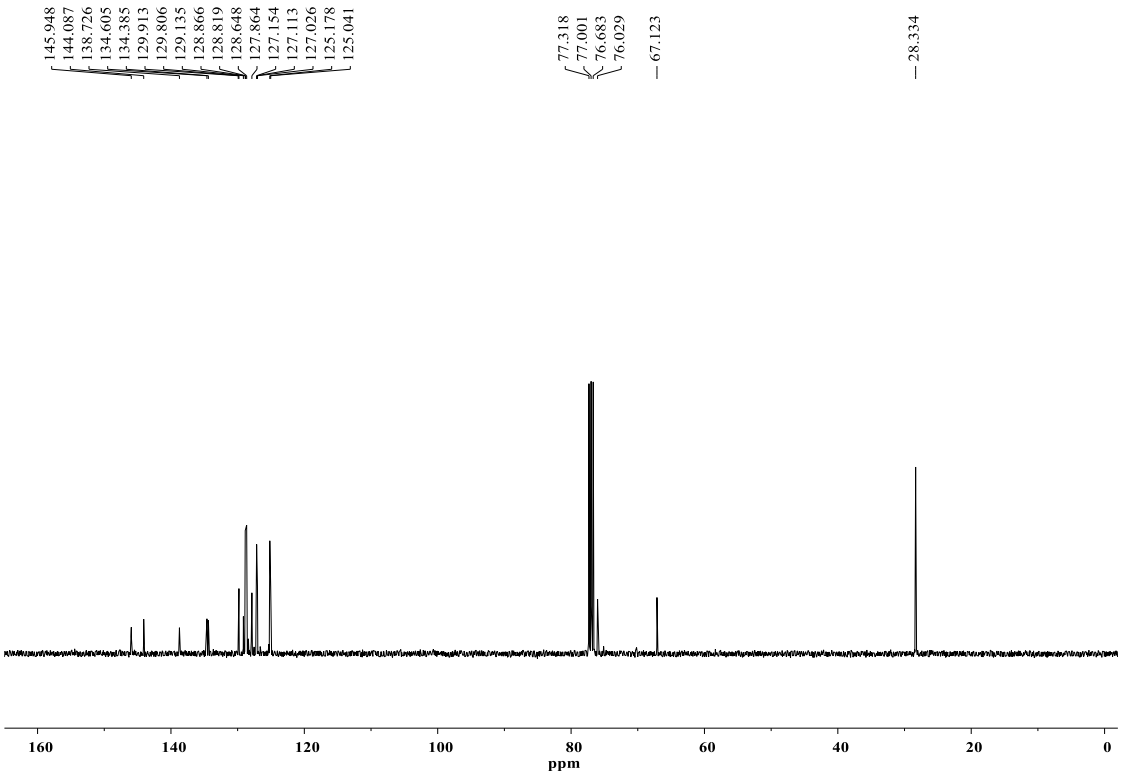
7.893
7.875
7.667
7.650
7.647
7.442
7.437
7.432
7.424
7.417
7.406
7.386
7.383
7.365
7.322
7.304
7.264
7.261
7.255
7.250
7.230
5.980
1.174
0.96
2.09
2.02
3.01
2.06
2.06
3.06
1.00
8.99
10
9
8
7
6
5
4
3
2
1
0
ppm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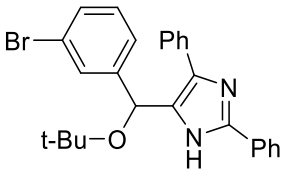
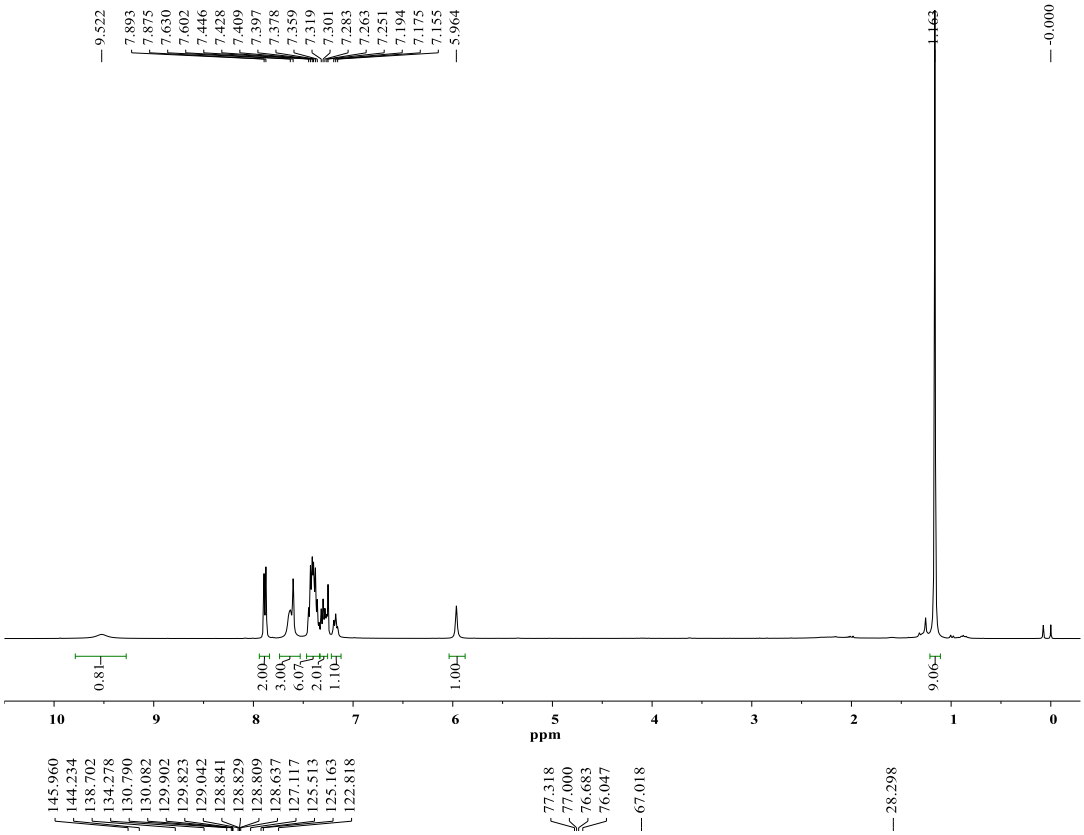
144.087
138.726
134.605
134.385
129.913
129.806
129.135
128.866
128.819
128.648
127.864
127.154
127.113
127.026
125.178
125.041
77.318
77.001
76.683
76.029
67.123
28.334
160
140
120
100
80
60
40
20
0
ppm


Br
Ph
N
t-Bu−O
N
H
Ph

4s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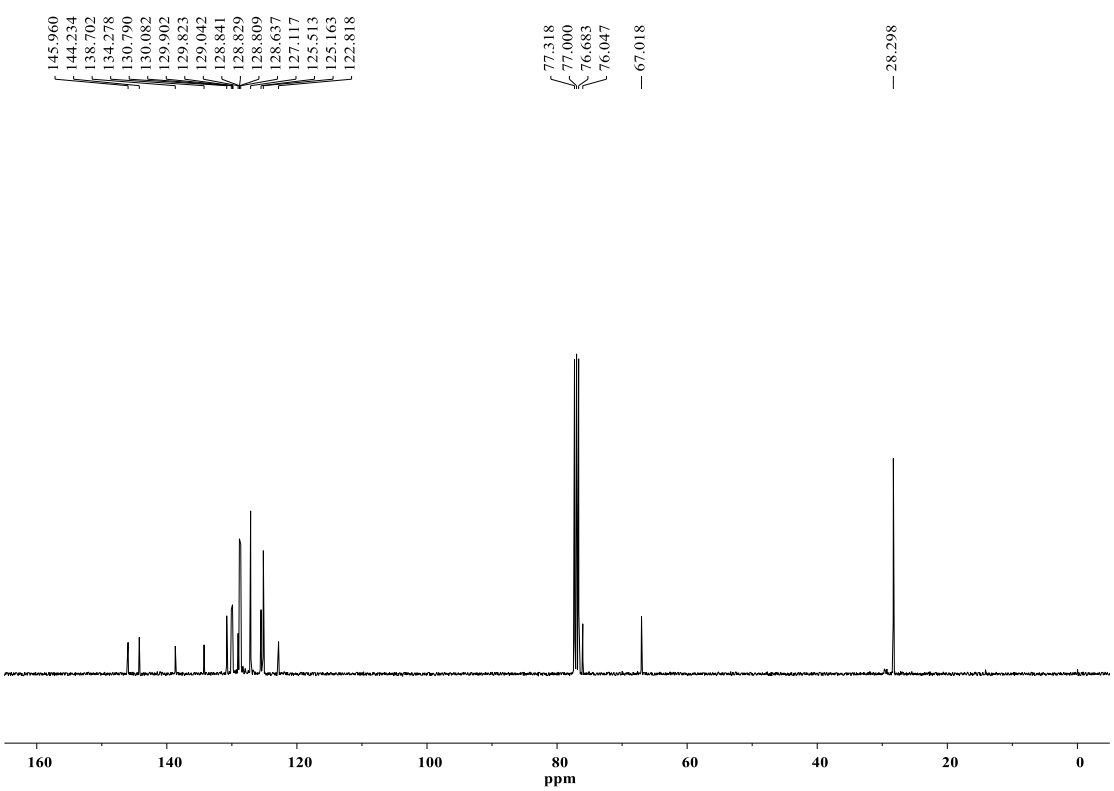
7.893
7.875
7.630
7.602
7.446
7.428
7.409
7.397
7.378
7.359
7.319
7.301
7.283
7.263
7.251
7.194
7.175
7.155
5.964
1.163
-0.000
0.81
2.00
3.00
6.07
2.01
1.10
1.00
9.06
10
9
8
7
6
5
4
3
2
1
0
ppm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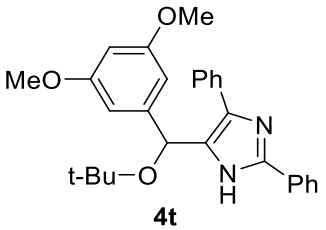
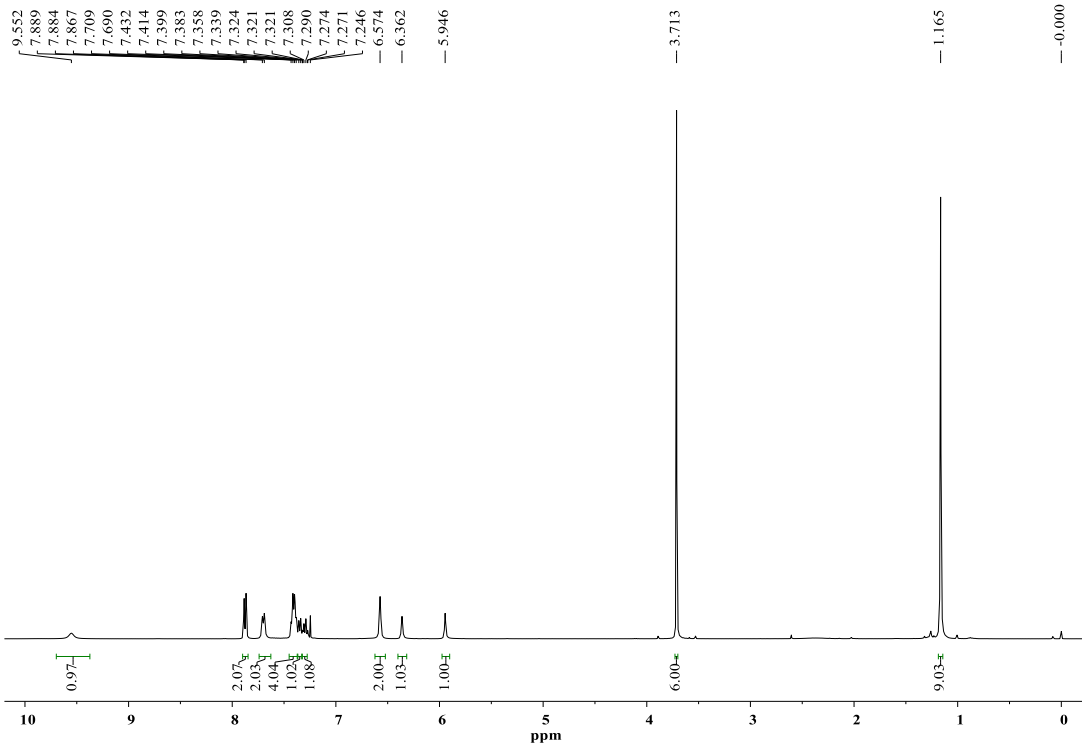
134.278
130.790
130.082
129.902
129.823
129.042
128.841
128.829
128.809
128.637
127.117
125.513
125.163
122.818
77.318
77.000
76.683
76.047
67.018
28.298
160
140
120
100
80
60
40
20
0
ppm


OMe
MeO
Ph
N
t-Bu–O
N
H
Ph

4t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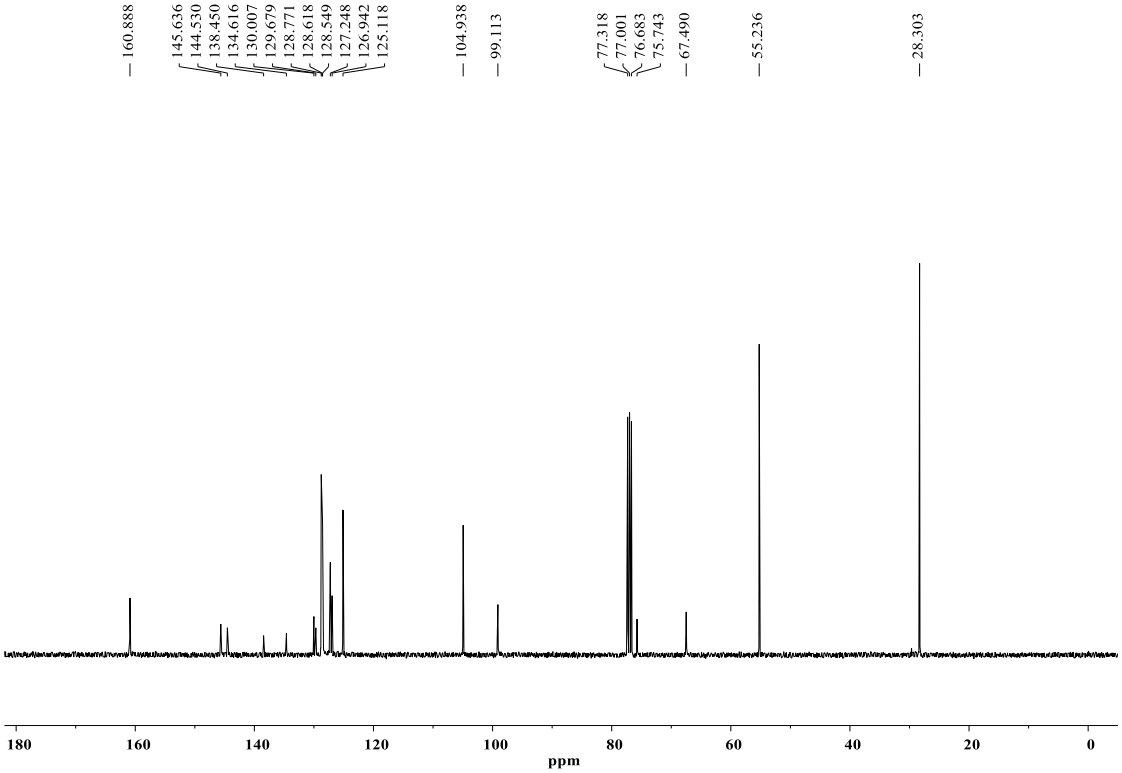
7.889
7.884
7.867
7.709
7.690
7.432
7.414
7.399
7.383
7.358
7.339
7.324
7.321
7.308
7.290
7.274
7.271
7.246
6.574
6.362
5.946
3.713
1.165
-0.000
0.97
2.07
2.03
4.04
1.02
1.08
2.00
1.03
1.00
6.00
9.03
10
9
8
7
6
5
4
3
2
1
0
ppm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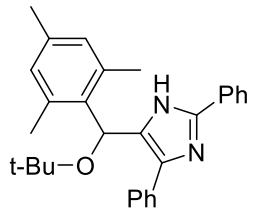
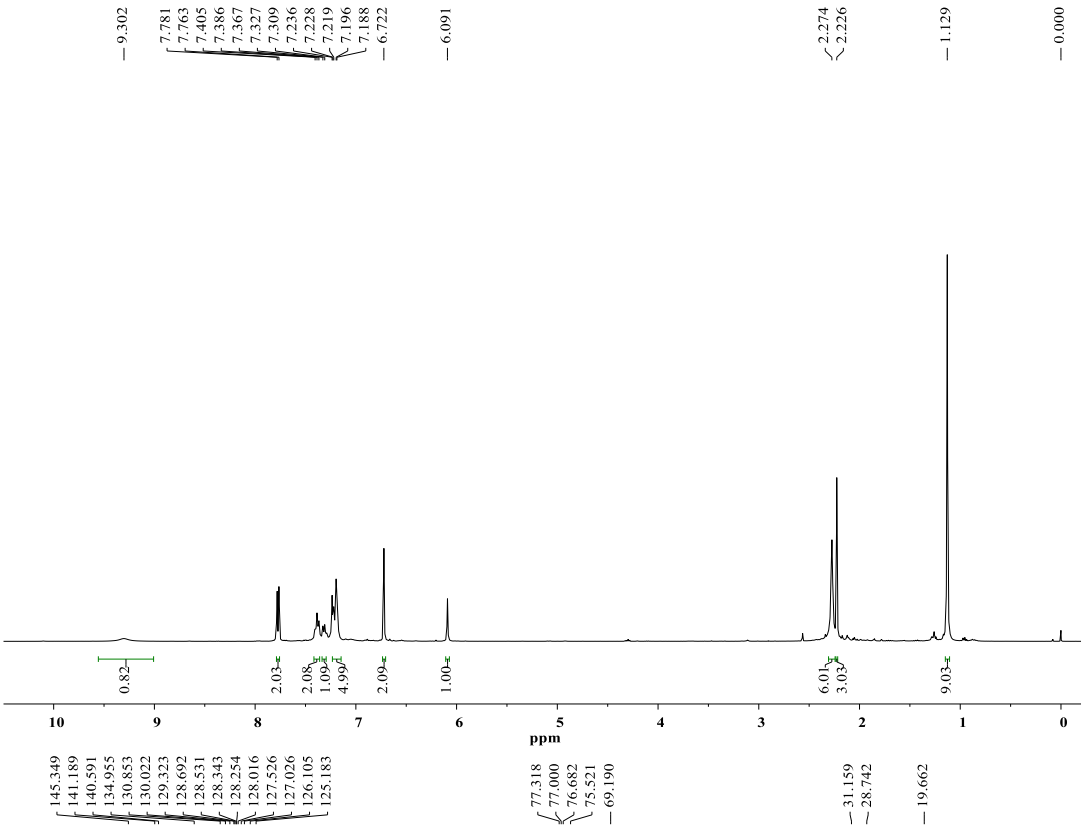
130.007
129.679
128.771
128.618
128.549
127.248
126.942
125.118
104.938
99.113
77.318
77.001
76.683
75.743
67.490
55.236
28.303
180
160
140
120
100
80
60
40
20
0
ppm


H
N
Ph
N
t-Bu−O
Ph

4u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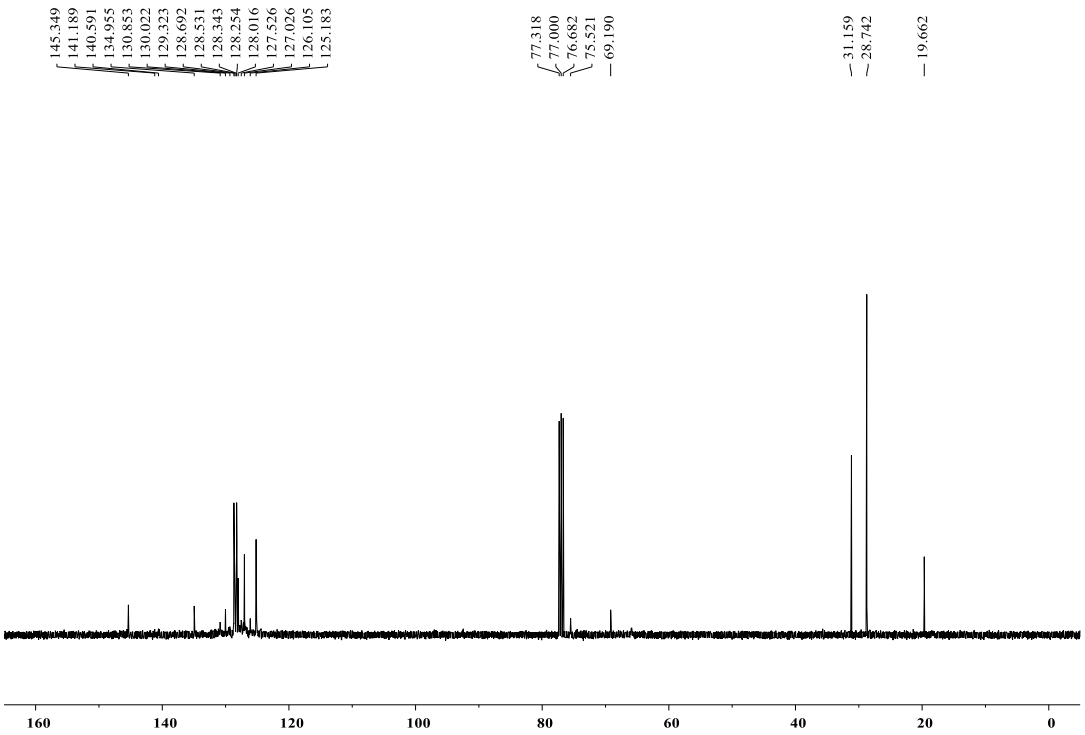
7.763
7.405
7.386
7.367
7.327
7.309
7.236
7.228
7.219
7.196
7.188
6.722
6.091
2.274
2.226
1.129
0.000
0.82
2.03
2.08
1.09
4.99
2.09
1.00
6.01
3.03
9.03
10
9
8
7
6
5
4
3
2
1
0
ppm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141.189
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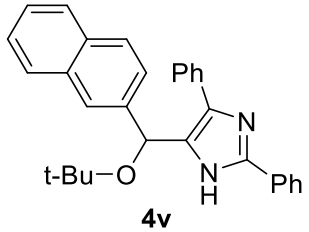
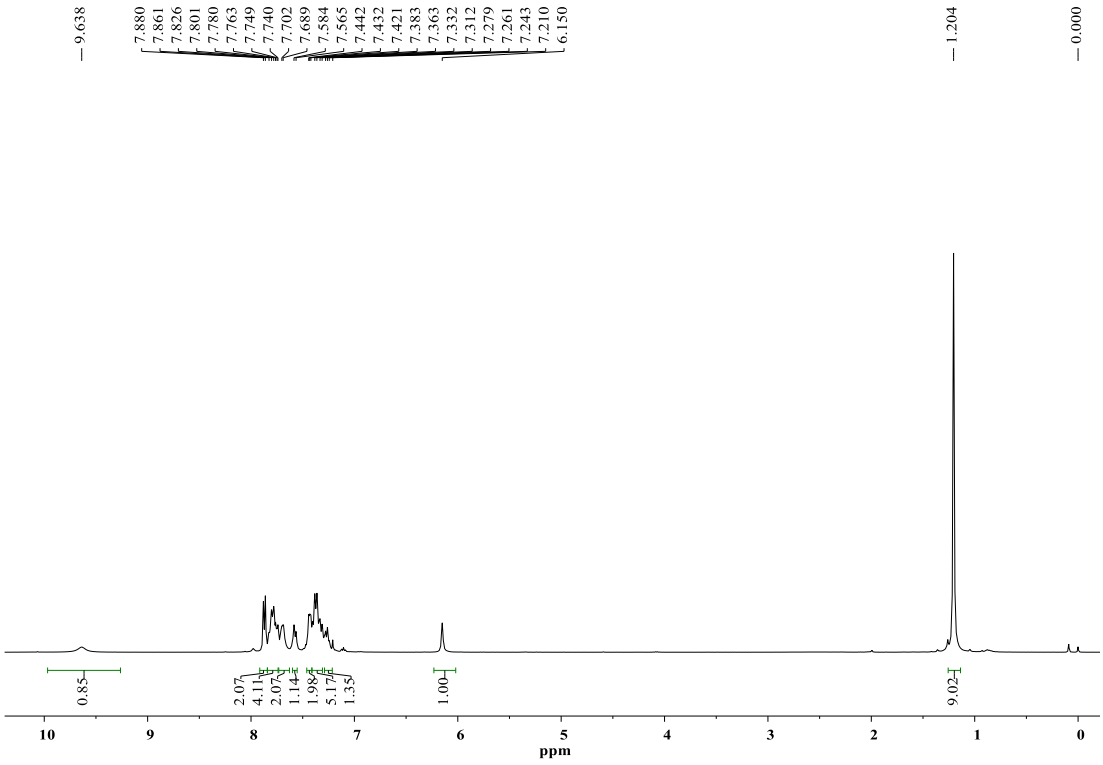
134.955
130.853
130.022
129.323
128.692
128.531
128.343
128.254
128.016
127.526
127.026
126.105
125.183
77.318
77.000
76.682
75.521
69.190
31.159
28.742
19.662
160
140
120
100
80
60
40
20
0
ppm


Ph
N
t-Bu—O
N
H
Ph

4v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7.780
7.763
7.749
7.740
7.702
7.689
7.584
7.565
7.442
7.432
7.421
7.383
7.363
7.332
7.312
7.279
7.261
7.243
7.210
6.150
1.204
0.000
0.85
2.07
4.11
2.07
1.14
1.98
5.17
1.35
1.00
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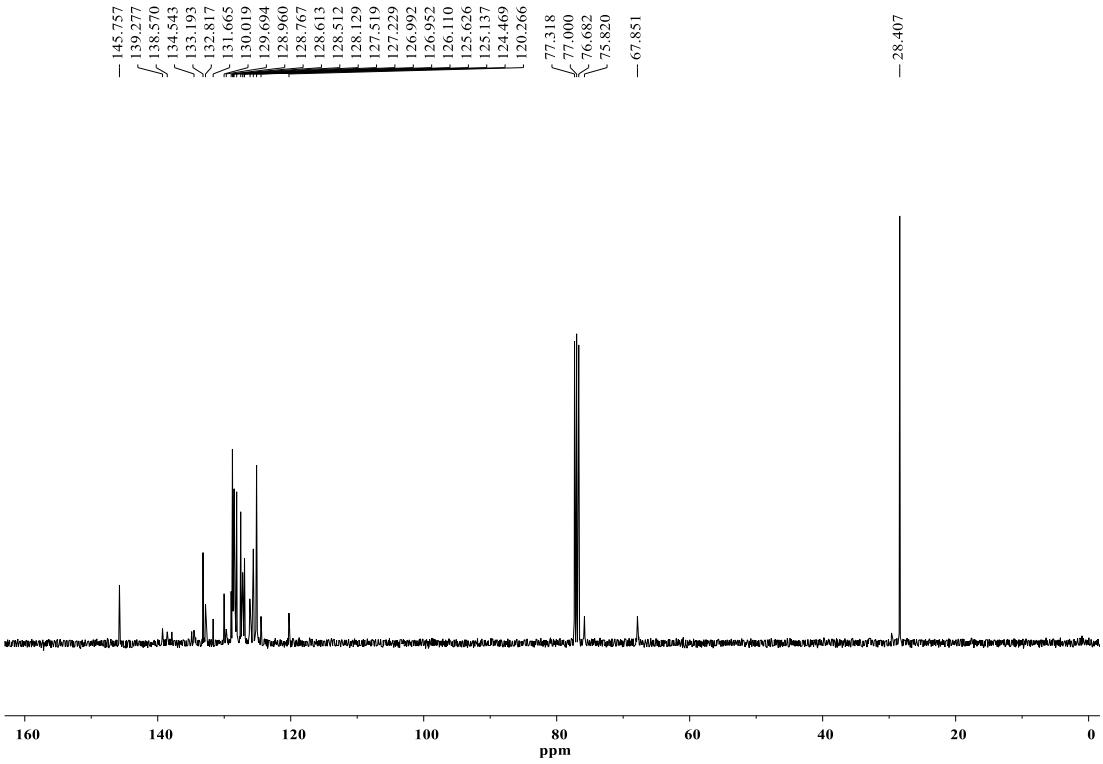
10
9
8
7
6
5
4
3
2
1
0
ppm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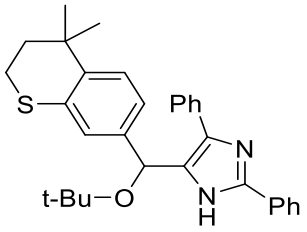
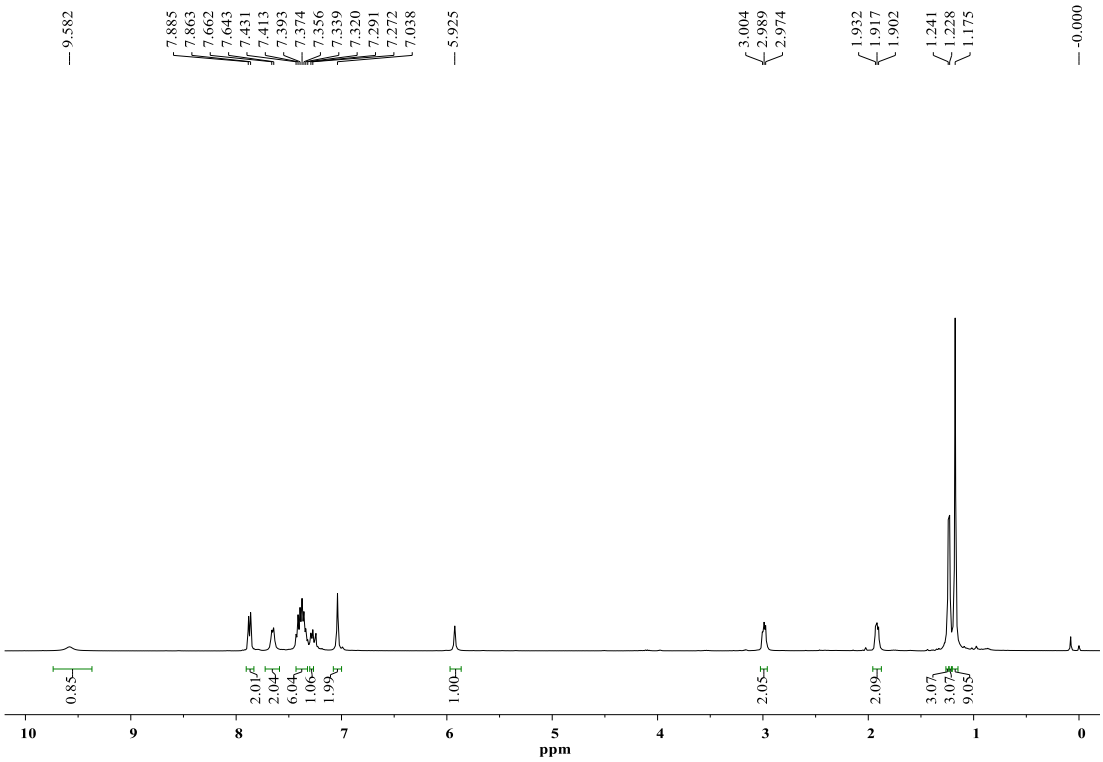
134.543
133.193
132.817
131.665
130.019
129.694
128.960
128.767
128.613
128.512
128.129
127.519
127.229
126.992
126.952
126.110
125.626
125.137
124.469
120.266
77.318
77.000
76.682
75.820
67.851
28.407
160
140
120
100
80
60
40
20
0
ppm


S
Ph
N
t-Bu—O
N
H
Ph

4w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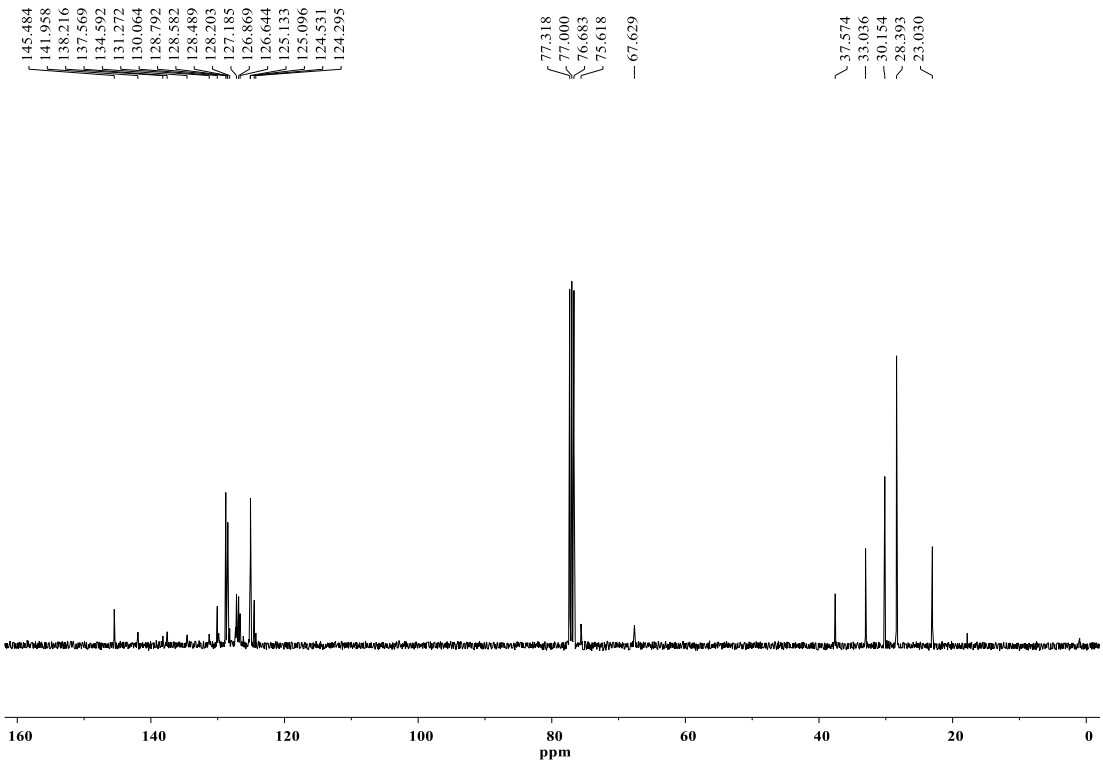
7.863
7.662
7.643
7.431
7.413
7.393
7.374
7.356
7.339
7.320
7.291
7.272
7.038
5.925
3.004
2.989
2.974
1.932
1.917
1.902
1.241
1.228
1.175
0.000
0.85
2.01
2.04
6.04
1.06
1.99
1.00
2.05
2.09
3.07
3.07
9.05
10
9
8
7
6
5
4
3
2
1
0
ppm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130.064
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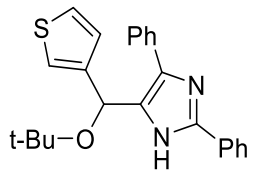
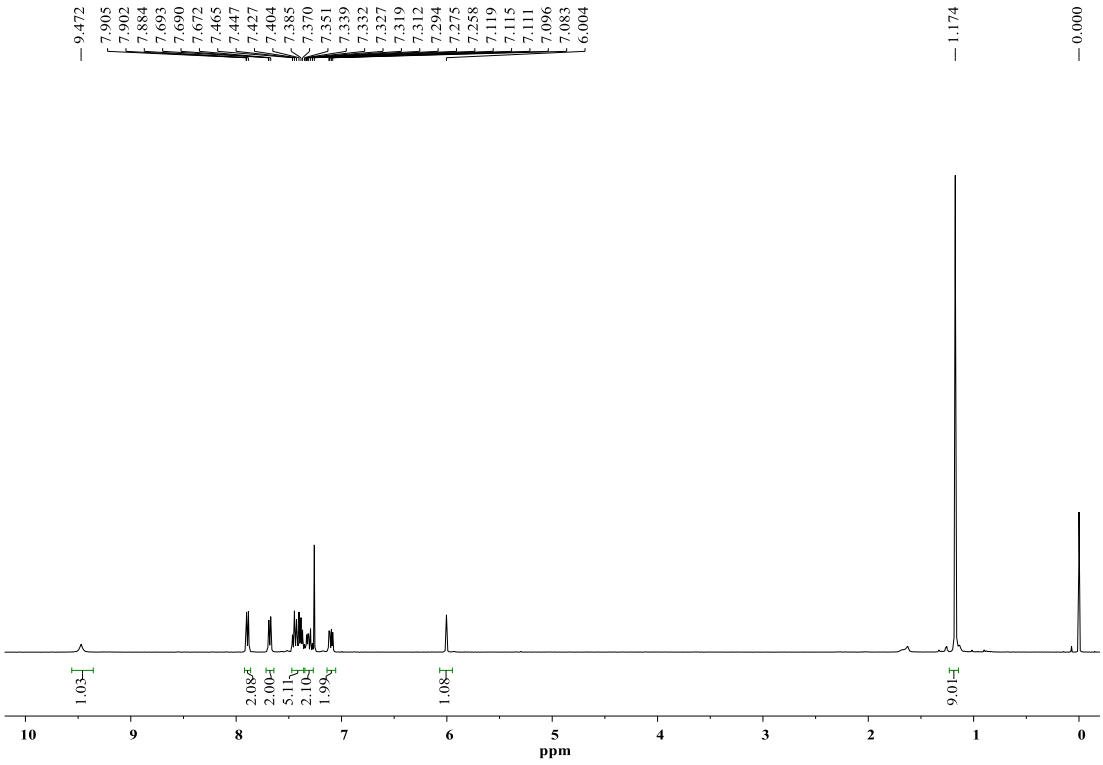
128.489
128.203
127.185
126.869
126.644
125.133
125.096
124.531
124.295
77.318
77.000
76.683
75.618
67.629
37.574
33.036
30.154
28.393
23.030
160
140
120
100
80
60
40
20
0
ppm


S
Ph
N
t-Bu–O
N
H
Ph

4x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7.693
7.690
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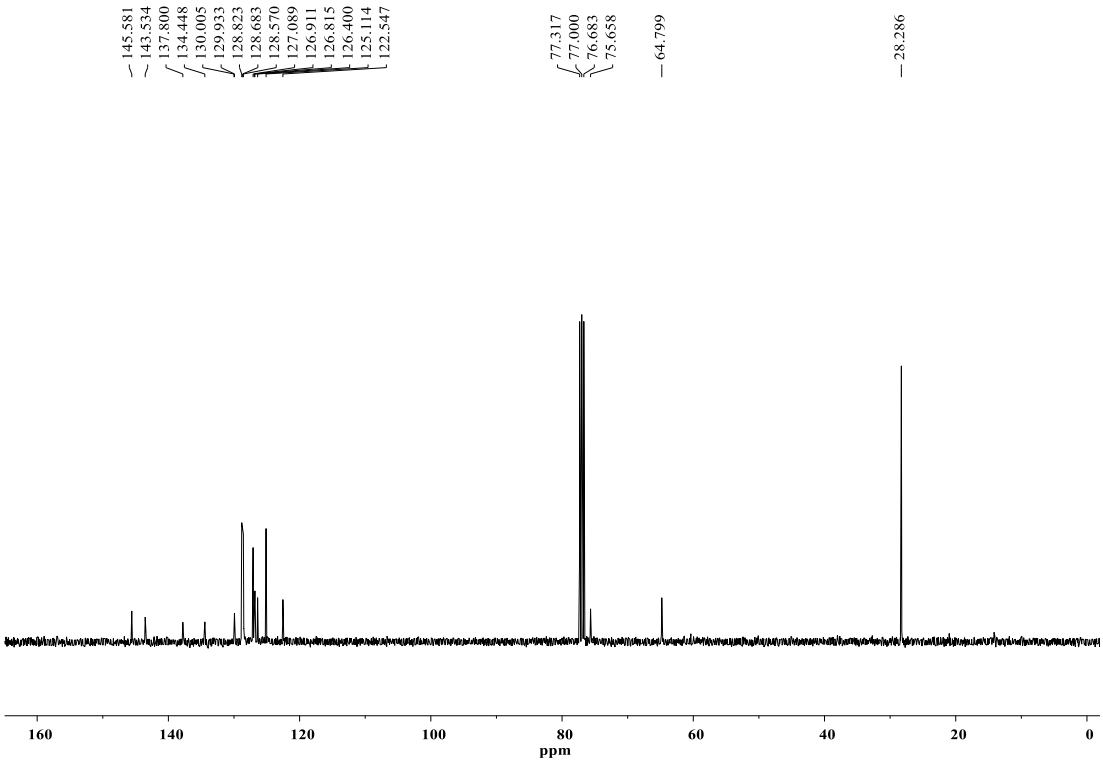
7.465
7.447
7.427
7.404
7.385
7.370
7.351
7.339
7.332
7.327
7.319
7.312
7.294
7.275
7.258
7.119
7.115
7.111
7.096
7.083
6.004
1.174
0.000
1.03
2.08
2.00
5.11
2.10
1.99
1.08
9.01
10
9
8
7
6
5
4
3
2
1
0
ppm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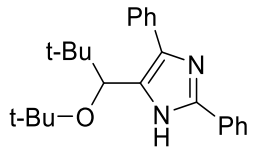
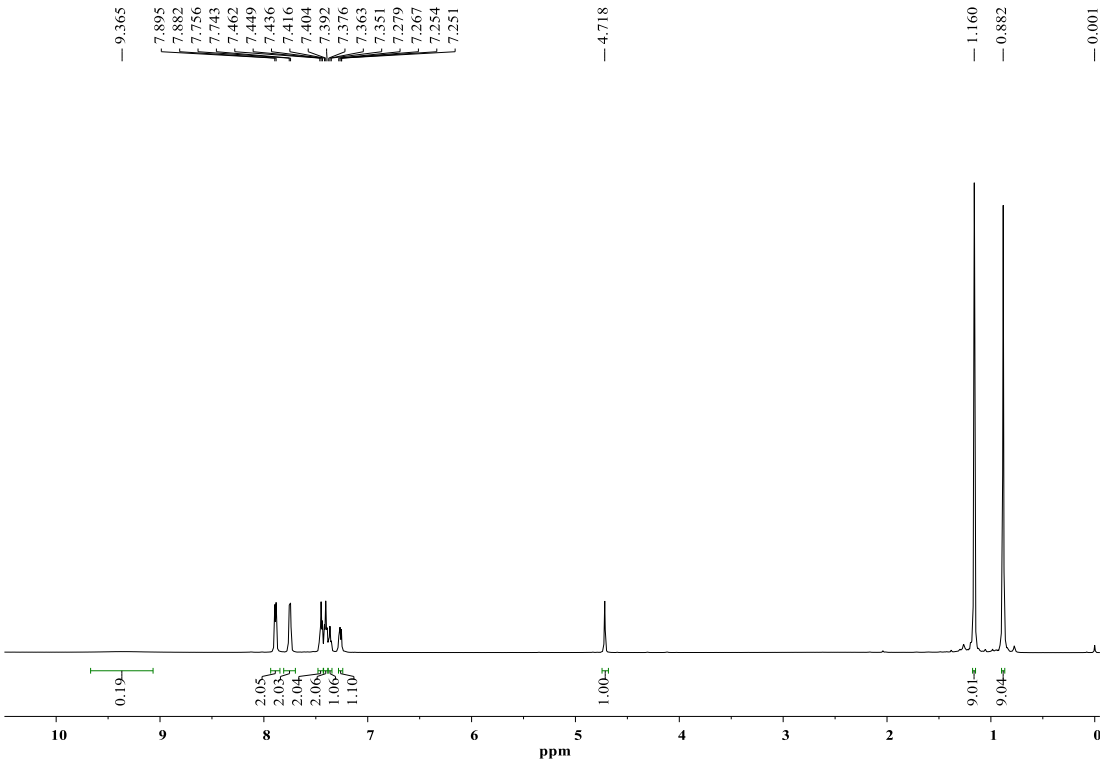
130.005
129.933
128.823
128.683
128.570
127.089
126.911
126.815
126.400
125.114
122.547
77.317
77.000
76.683
75.658
64.799
28.286
160
140
120
100
80
60
40
20
0
ppm


Ph
t-Bu
N
t-Bu–O
N
H
Ph

4y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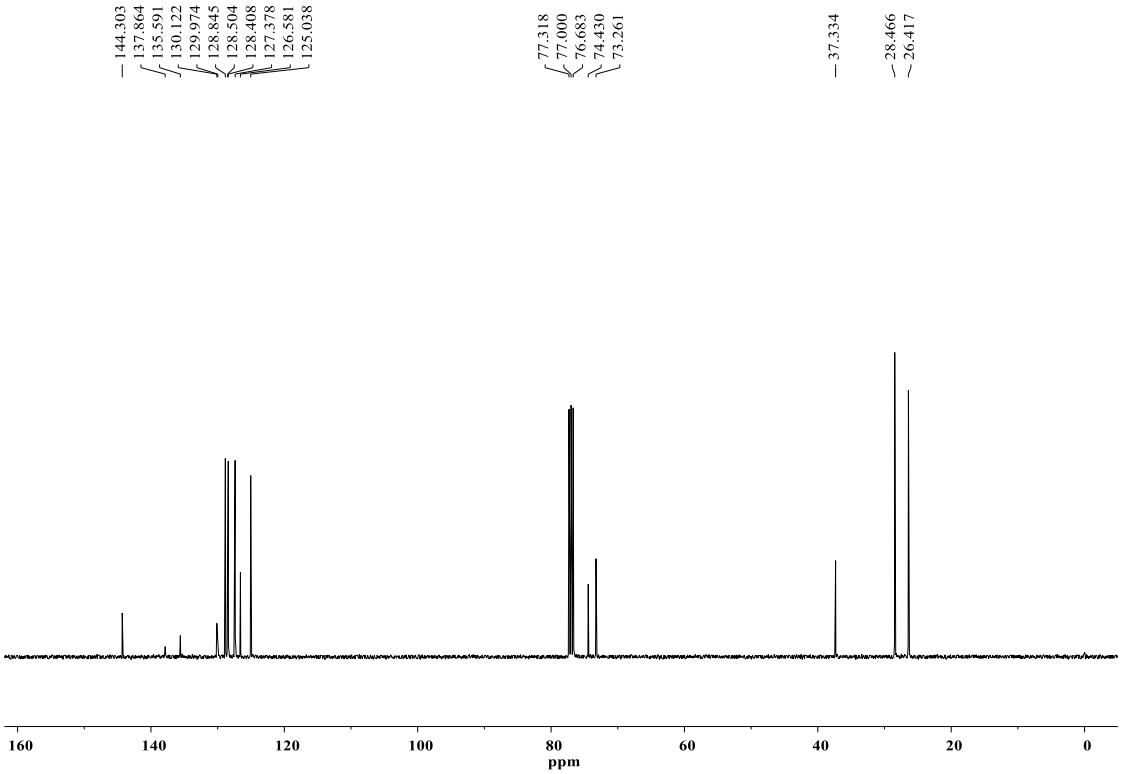
7.756
7.743
7.462
7.449
7.436
7.416
7.404
7.392
7.376
7.363
7.351
7.279
7.267
7.254
7.251
4.718
1.160
0.882
0.001
0.19
2.05
2.03
2.04
2.06
1.06
1.10
1.00
9.01
9.04
10
9
8
7
6
5
4
3
2
1
0
ppm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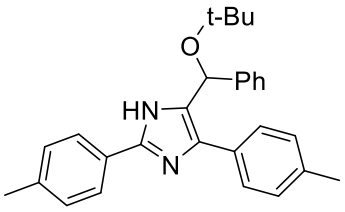
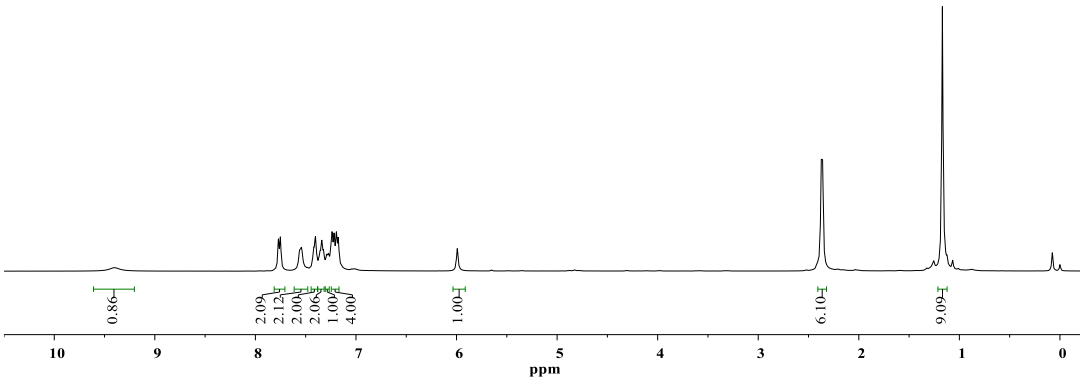
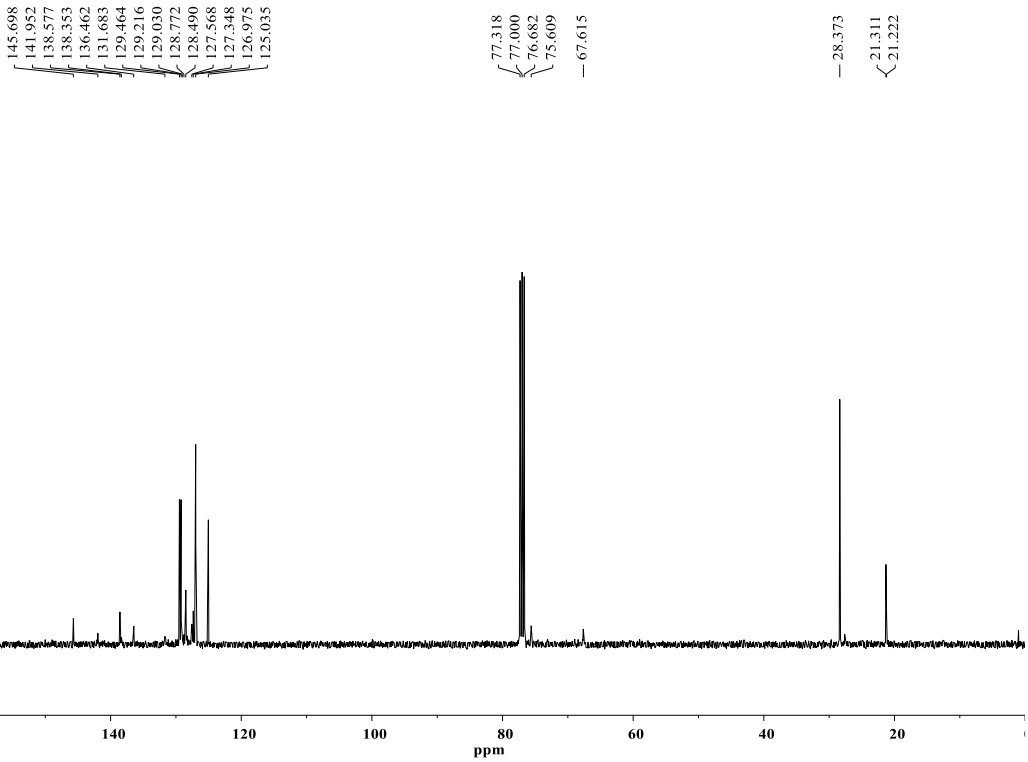
135.591
130.122
129.974
128.845
128.504
128.408
127.378
126.581
125.038
77.318
77.000
76.683
74.430
73.261
37.334
28.466
26.417
160
140
120
100
80
60
40
20
0
ppm

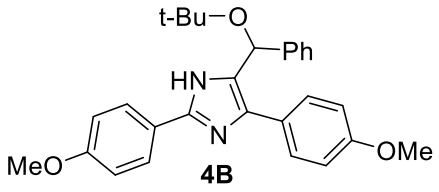

t-Bu
O
Ph
HN
N

4A

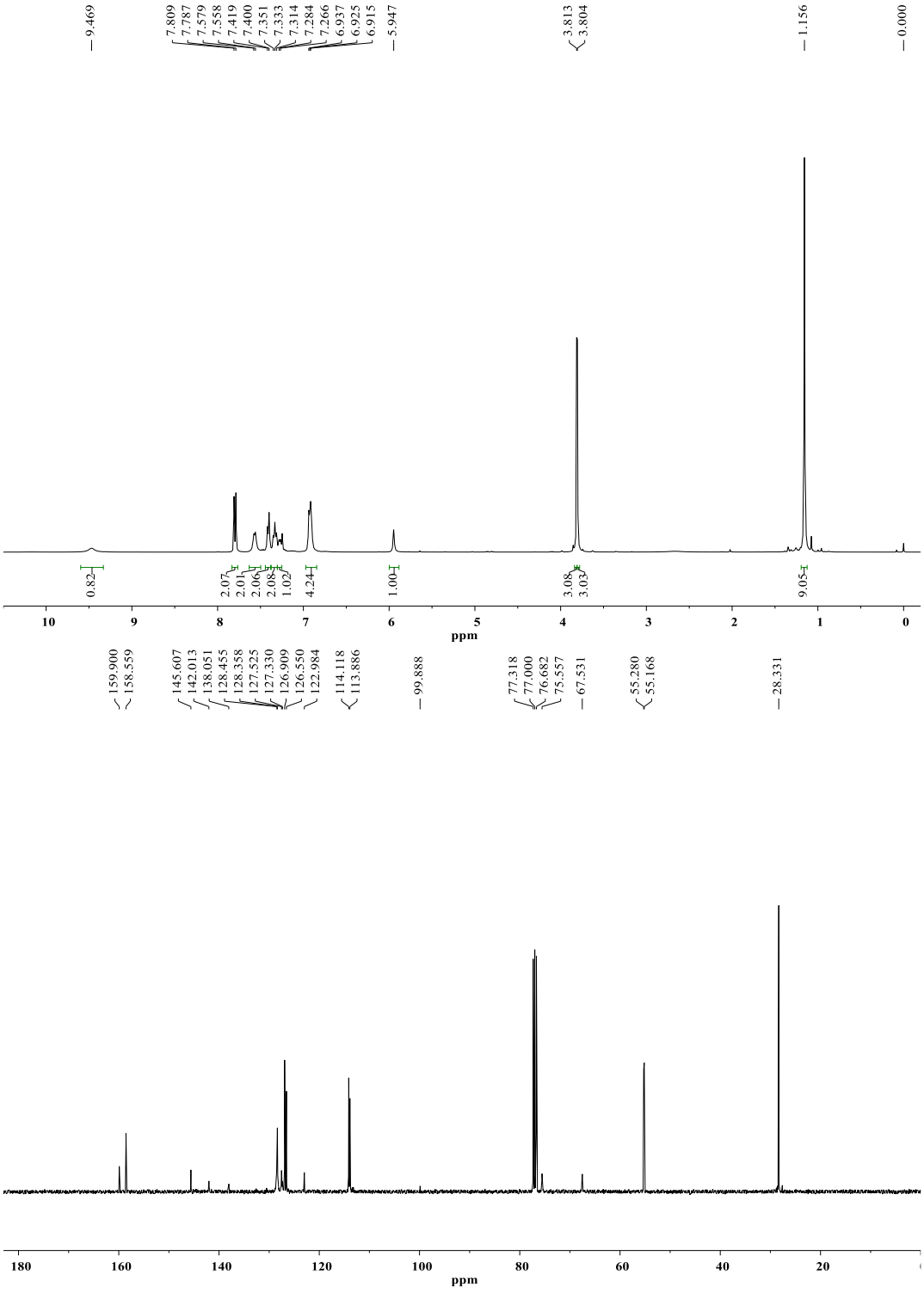

9.400
7.771
7.751
7.559
7.540
7.421
7.402
7.358
7.341
7.322
7.291
7.275
7.237
7.218
7.195
7.176
5.993
2.373
2.359
1.169
0.000

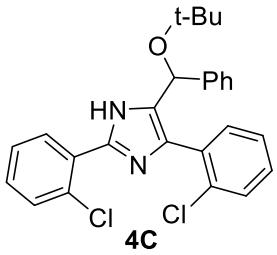

0.86
2.09
2.12
2.00
2.06
1.00
4.00
1.00
6.10
9.09
10
9
8
7
6
5
4
3
2
1
0
ppm


145.698
141.952
138.577
138.353
136.462
131.683
129.464
129.216
129.030
128.772
128.490
127.568
127.348
126.975
125.035
77.318
77.000
76.682
75.609
67.615
28.373
21.311
21.222
160
140
120
100
80
60
40
20
ppm

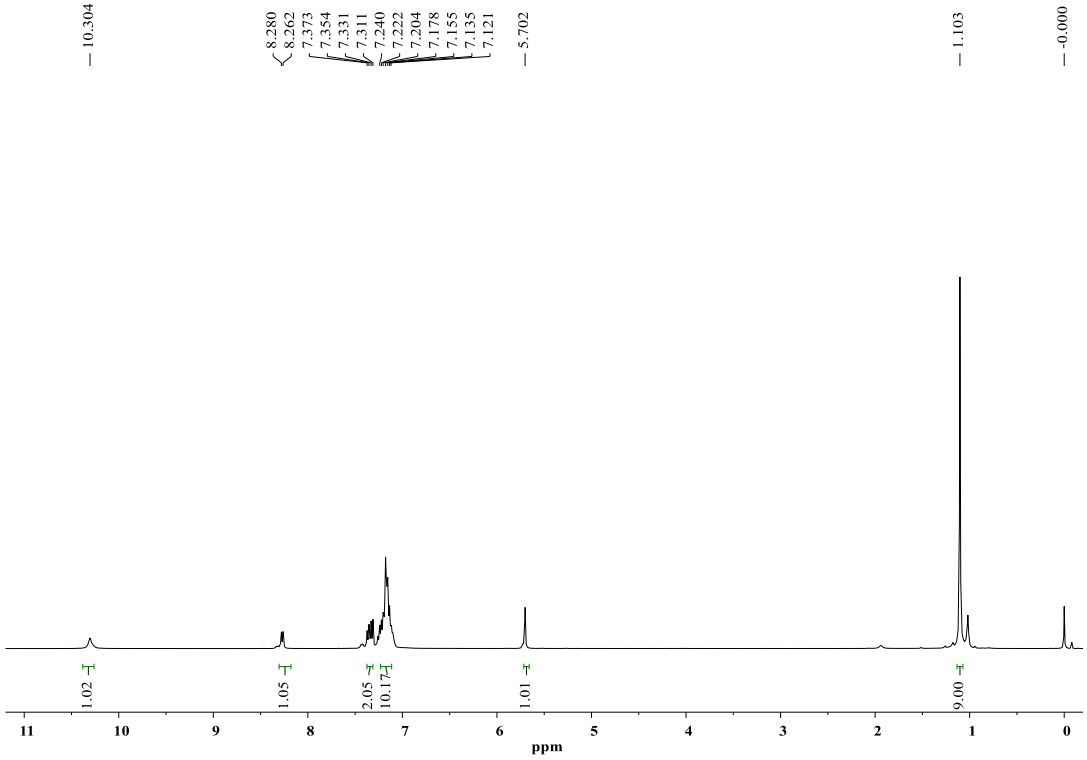

t-Bu−O
Ph
HN
N
MeO
OMe

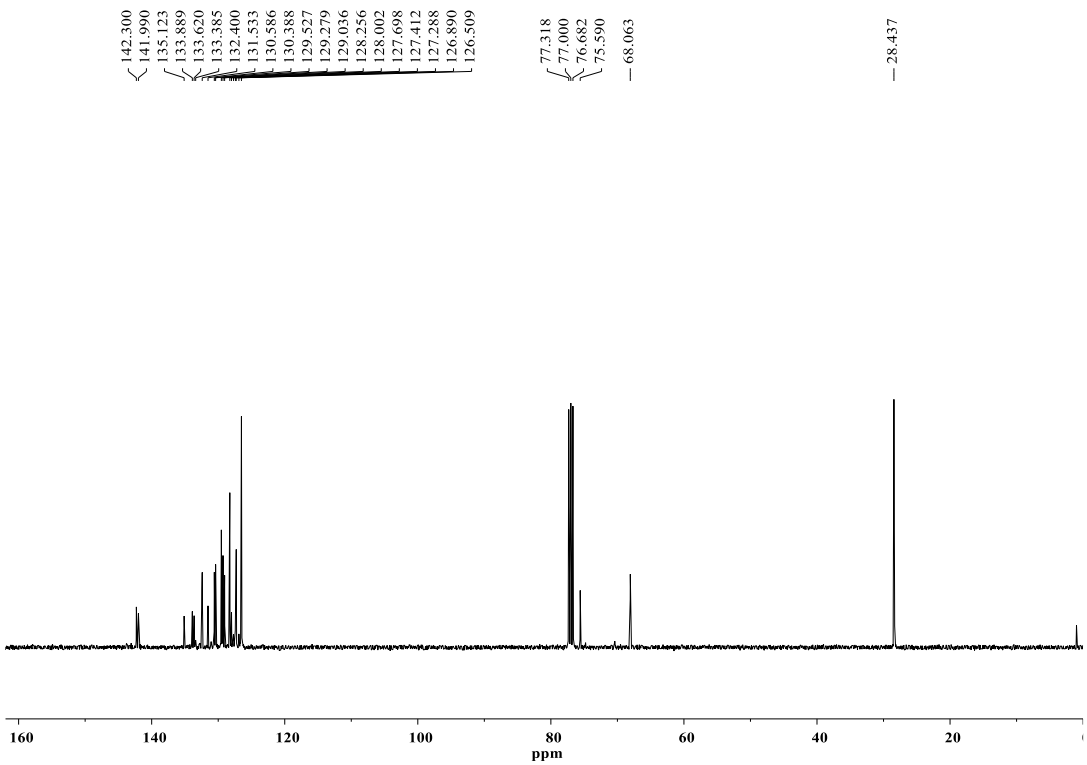
4B

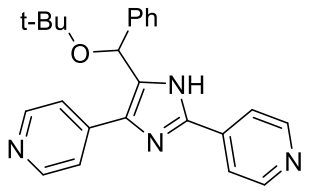

9.469
7.809
7.787
7.579
7.558
7.419
7.400
7.351
7.333
7.314
7.284
7.266
6.937
6.925
6.915
5.947
3.813
3.804
1.156
0.000
0.82
2.07
2.01
2.06
2.08
1.02
4.24
1.00
3.08
3.03
9.05
ppm
159.900
158.559
145.607
142.013
138.051
128.455
128.358
127.525
127.330
126.909
126.550
122.984
114.118
113.886
99.888
77.318
77.000
76.682
75.557
67.531
55.280
55.168
28.331
ppm


t-Bu
O
Ph
HN
N
Cl
Cl

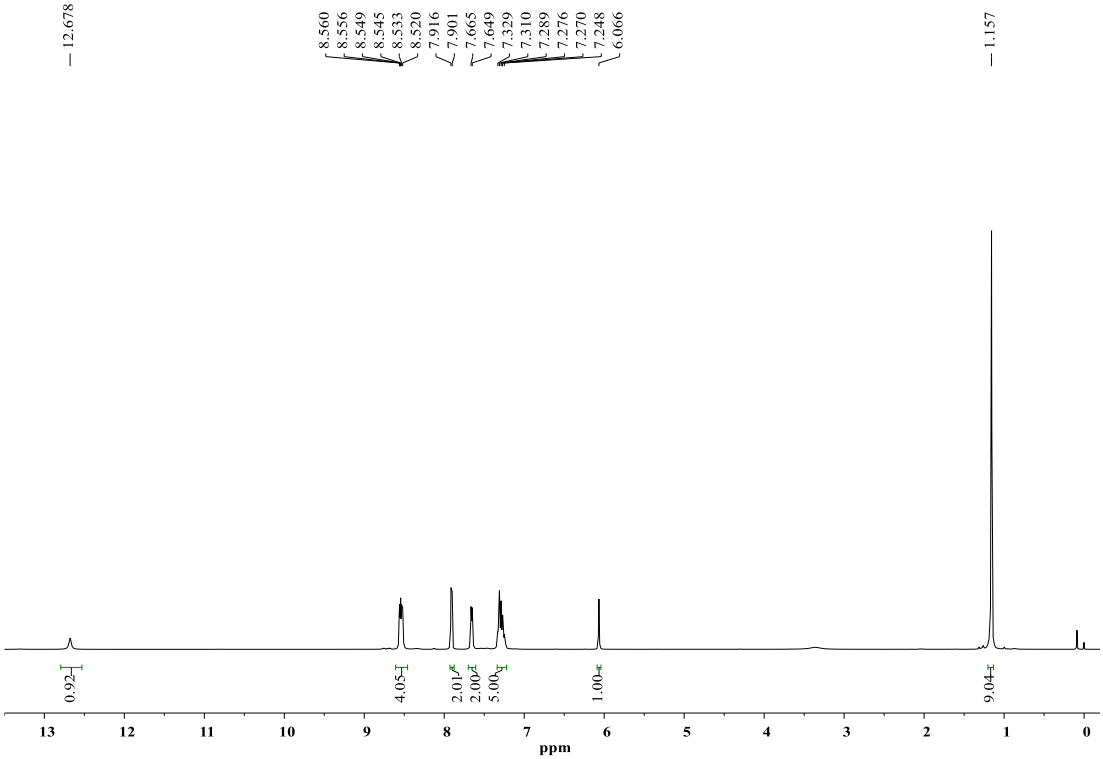
4C

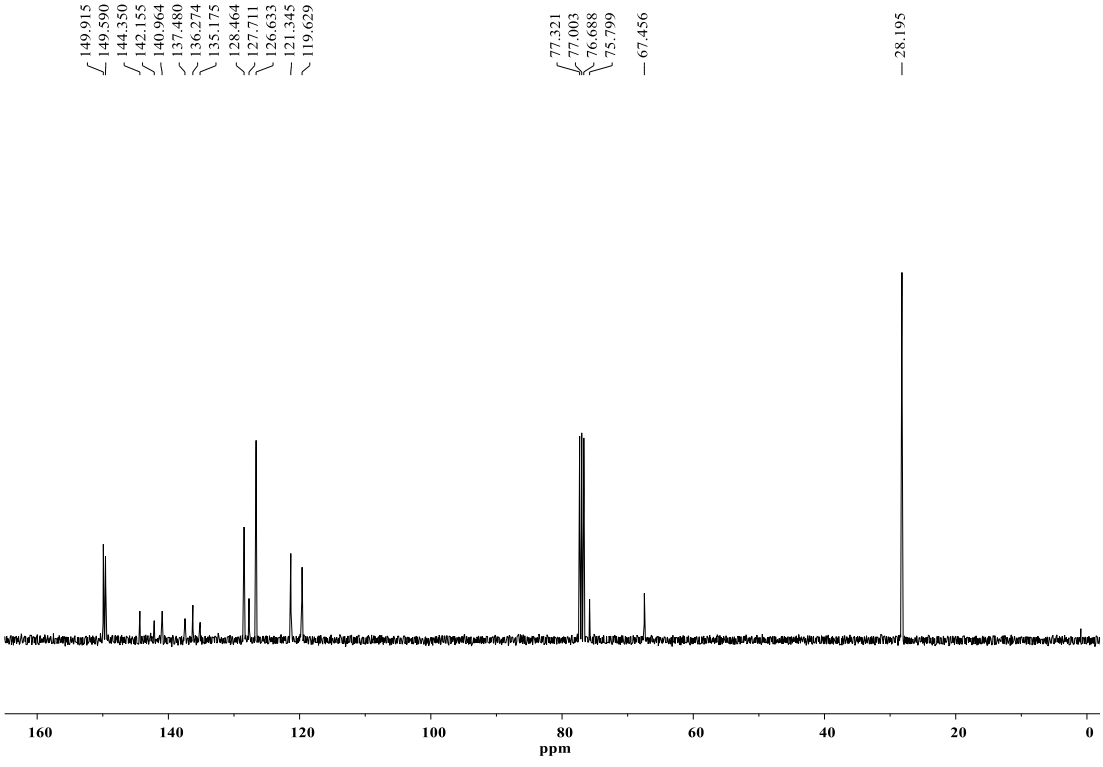

10.304
8.280
8.262
7.373
7.354
7.331
7.311
7.240
7.222
7.204
7.178
7.155
7.135
7.121
5.702
1.103
-0.000
1.02
1.05
2.05
10.17
1.01
9.00
ppm

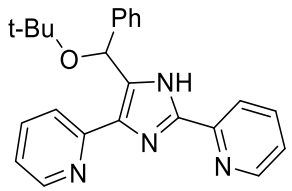

142.300
141.990
135.123
133.889
133.620
133.385
132.400
131.533
130.586
130.388
129.527
129.279
129.036
128.256
128.002
127.698
127.412
127.288
126.890
126.509
77.318
77.000
76.682
75.590
68.063
28.437
ppm


t-Bu
O
Ph
NH
N
N
N

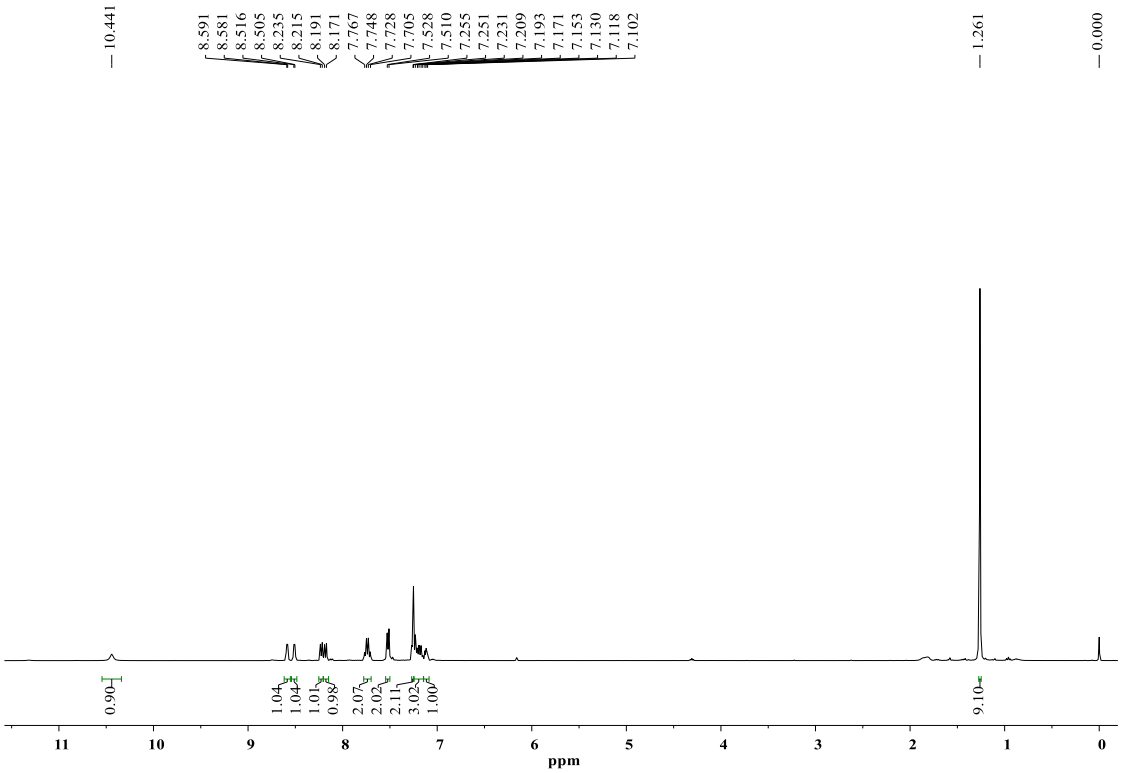
4D

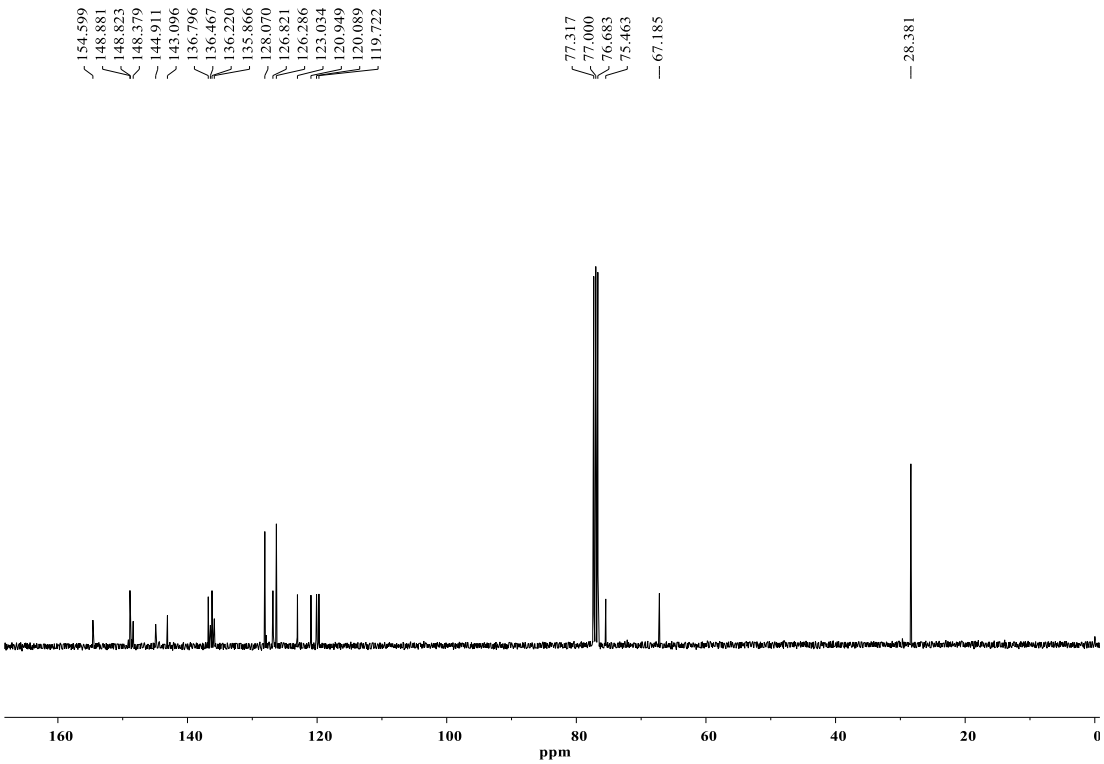

12.678
8.560
8.556
8.549
8.545
8.533
8.520
7.916
7.901
7.665
7.649
7.329
7.310
7.289
7.276
7.270
7.248
6.066
1.157
0.92
4.05
2.01
2.00
5.00
1.00
9.04
ppm

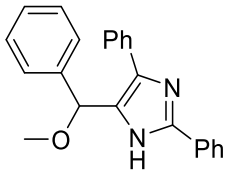

149.915
149.590
144.350
142.155
140.964
137.480
136.274
135.175
128.464
127.711
126.633
121.345
119.629
77.321
77.003
76.688
75.799
67.456
28.195
ppm


t-Bu
O
Ph
NH
N
N
N

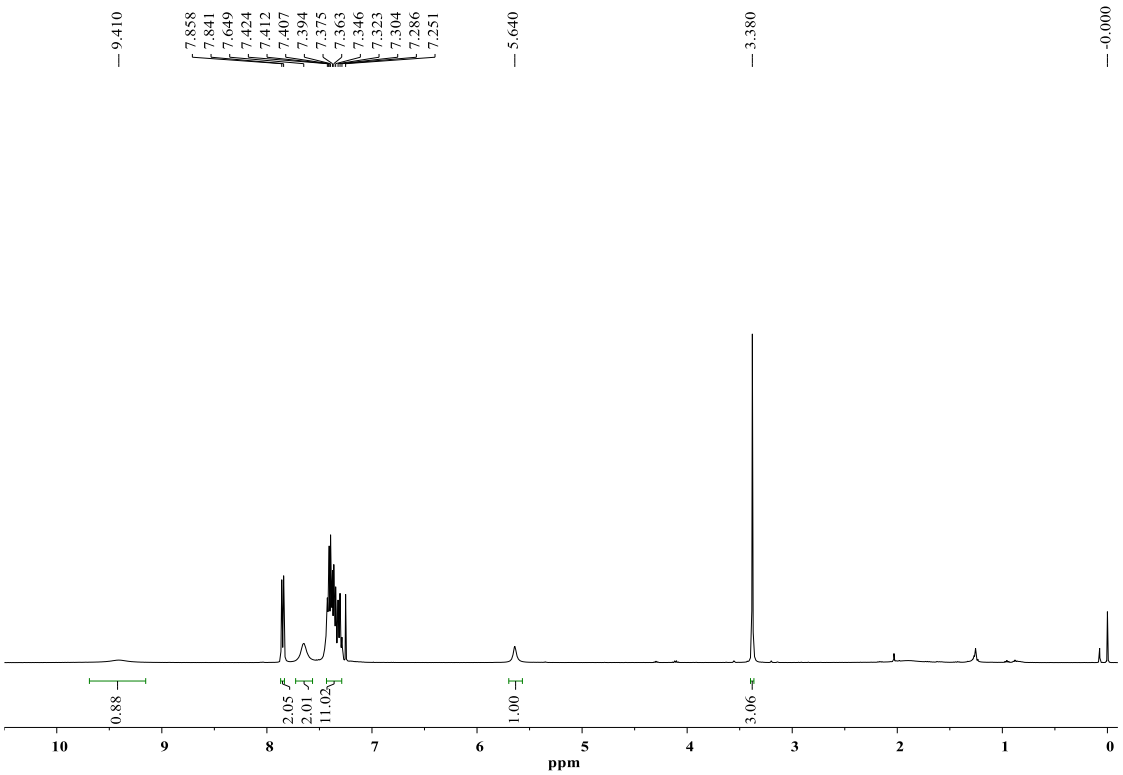
4E

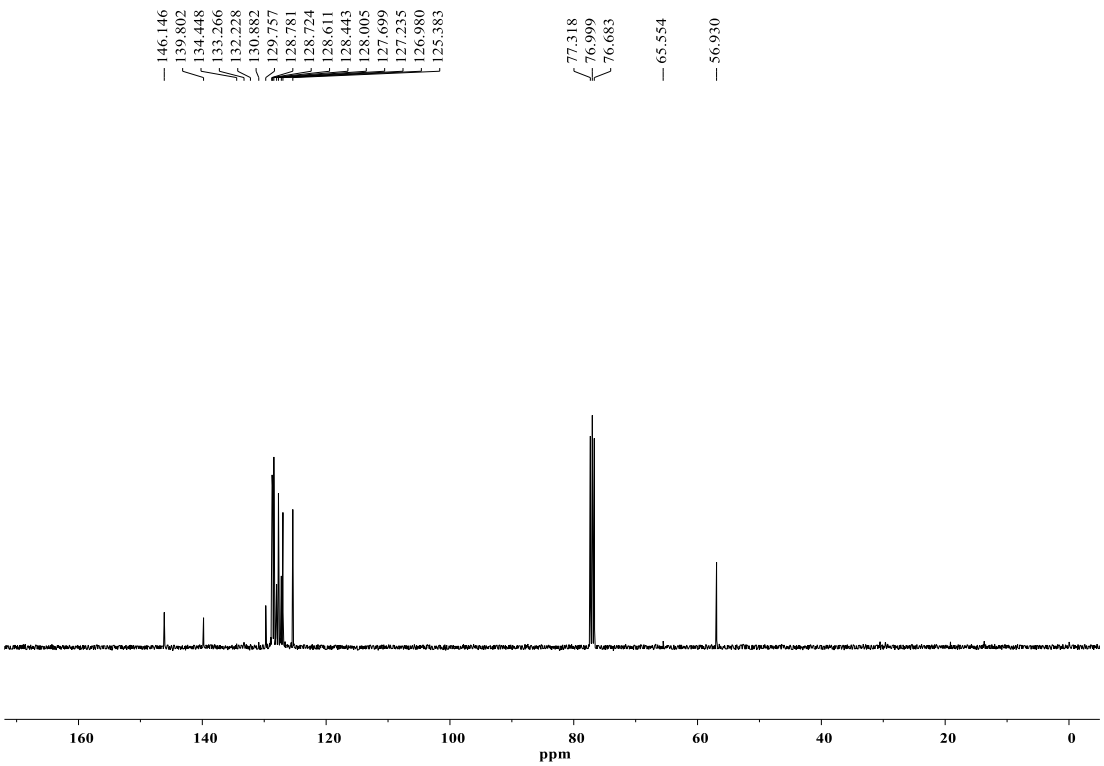

10.441
8.591
8.581
8.516
8.505
8.235
8.215
8.191
8.171
7.767
7.748
7.728
7.705
7.528
7.510
7.255
7.251
7.231
7.209
7.193
7.171
7.153
7.130
7.118
7.102
1.261
0.000
0.90
1.04
1.04
1.01
0.98
2.07
2.02
2.11
3.02
1.00
9.10
11
10
9
8
7
6
5
4
3
2
1
0
ppm

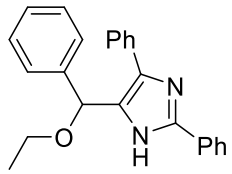

154.599
148.881
148.823
148.379
144.911
143.096
136.796
136.467
136.220
135.866
128.070
126.821
126.286
123.034
120.949
120.089
119.722
77.317
77.000
76.683
75.463
67.185
28.381
160
140
120
100
80
60
40
20
0
ppm


Ph
N
O
N
H
Ph

4a1

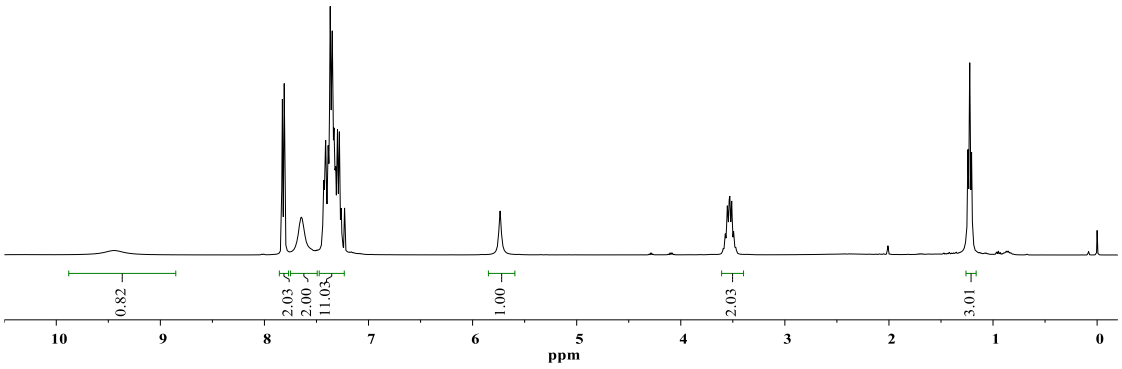

9.410
7.858
7.841
7.649
7.424
7.412
7.407
7.394
7.375
7.363
7.346
7.323
7.304
7.286
7.251
5.640
3.380
-0.000
0.88
2.05
2.01
11.02
1.00
3.06
10
9
8
7
6
5
4
3
2
1
0
ppm

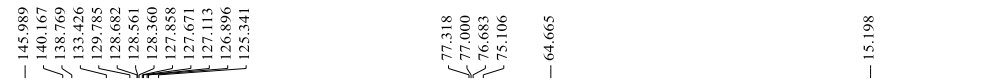

146.146
139.802
134.448
133.266
132.228
130.882
129.757
128.781
128.724
128.611
128.443
128.005
127.699
127.235
126.980
125.383
77.318
76.999
76.683
65.554
56.930
160
140
120
100
80
60
40
20
0
ppm

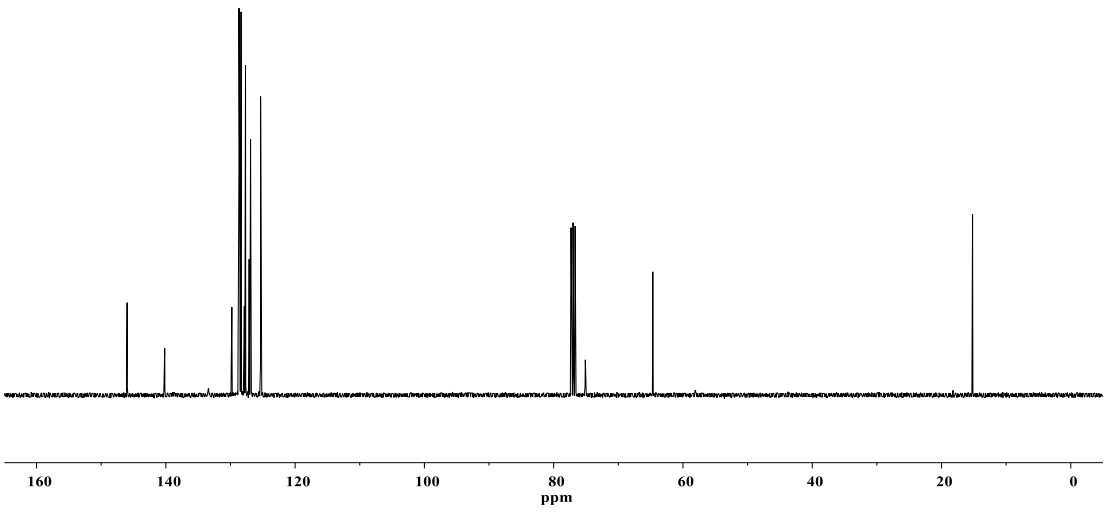

Ph
N
O
N
H
Ph

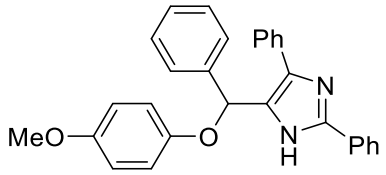
4a2


9.444
7.832
7.827
7.810
7.645
7.430
7.412
7.387
7.368
7.349
7.332
7.315
7.298
7.280
7.262
7.230
5.736
3.593
3.574
3.553
3.535
3.529
3.511
3.489
3.470
1.241
1.224
1.206
0.000

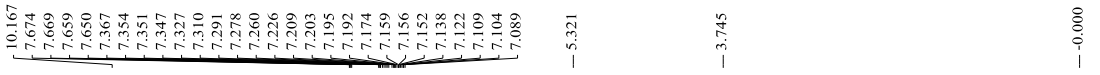

0.82
2.03
2.00
11.03
1.00
2.03
3.01
10
9
8
7
6
5
4
3
2
1
0
ppm

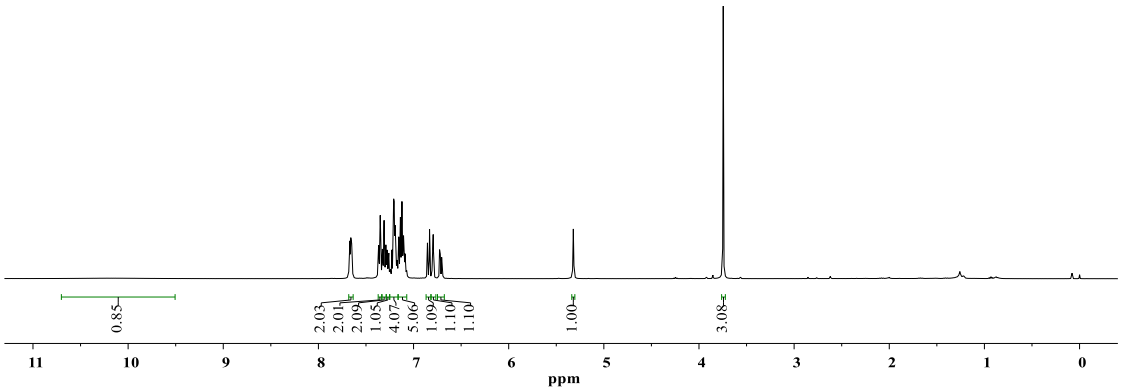

145.989
140.167
138.769
133.426
129.785
128.682
128.561
128.360
127.858
127.671
127.113
126.896
125.341
77.318
77.000
76.683
75.106
64.665
15.198

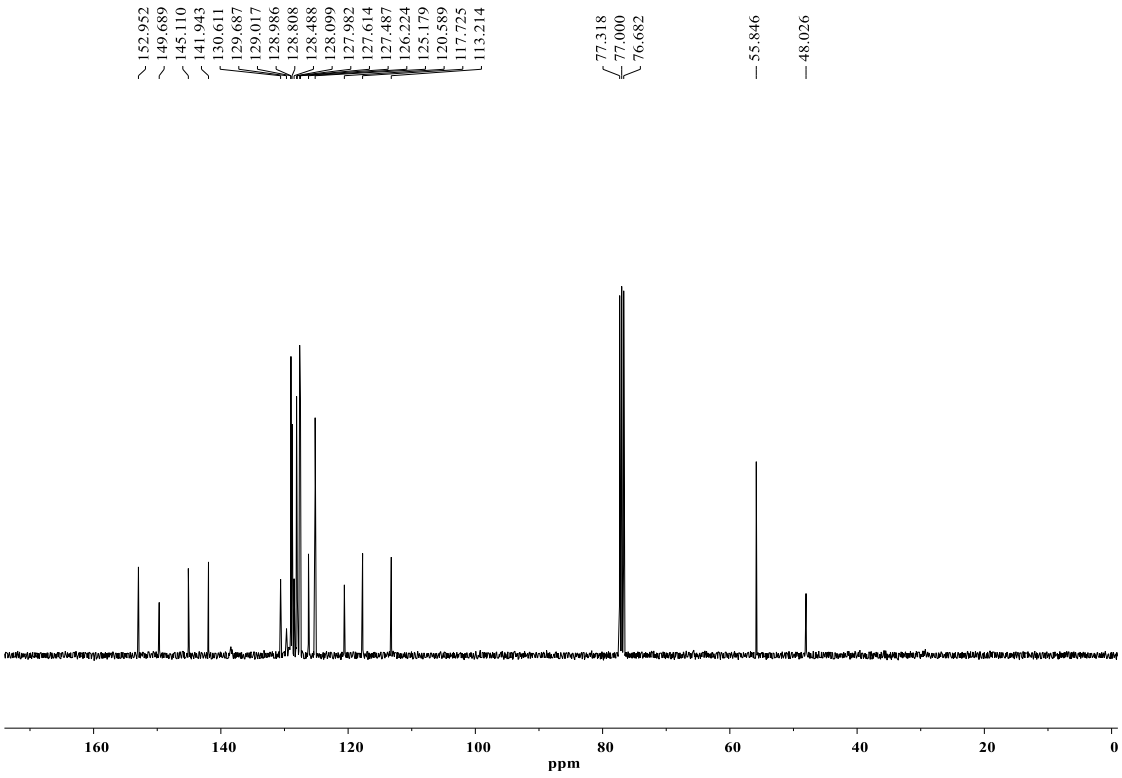

160
140
120
100
80
60
40
20
0
ppm


Ph
N
MeO
O
N
H
Ph

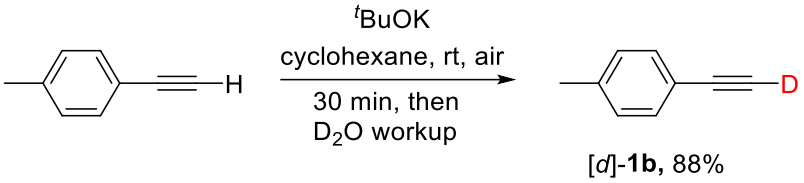
4a3


10.167
7.674
7.669
7.659
7.650
7.367
7.354
7.351
7.347
7.327
7.310
7.291
7.278
7.260
7.226
7.209
7.203
7.195
7.192
7.174
7.159
7.156
7.152
7.138
7.122
7.109
7.104
7.089
5.321
3.745
-0.000

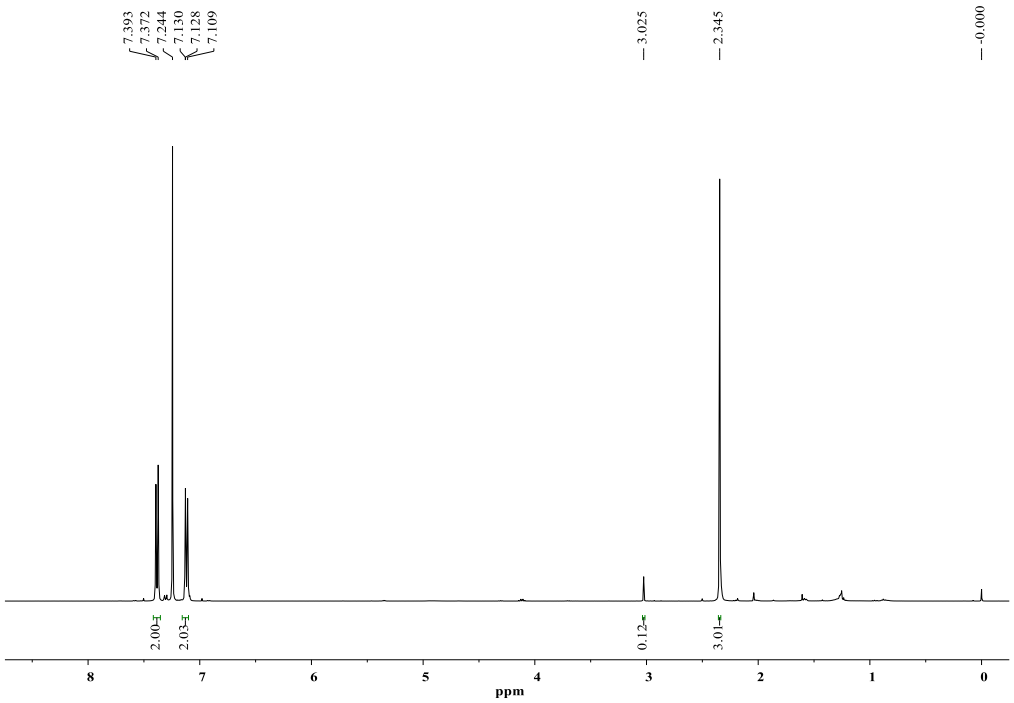

0.85
2.03
2.01
2.09
1.05
4.07
5.06
1.09
1.10
1.10
1.00
3.08
11
10
9
8
7
6
5
4
3
2
1
0
ppm

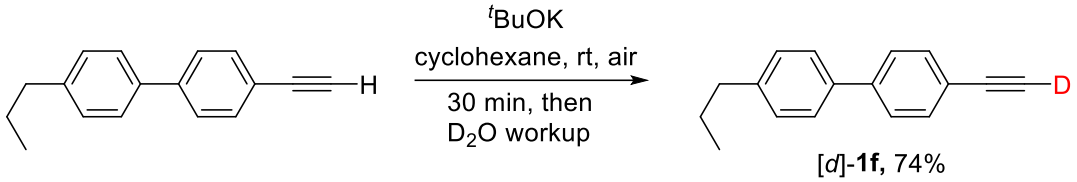

152.952
149.689
145.110
141.943
130.611
129.687
129.017
128.986
128.808
128.488
128.099
127.982
127.614
127.487
126.224
125.179
120.589
117.725
113.214
77.318
77.000
76.682
55.846
48.026
160
140
120
100
80
60
40
20
0
ppm

6. ^{1}H NMR Spectras of Mechanistic Investigations

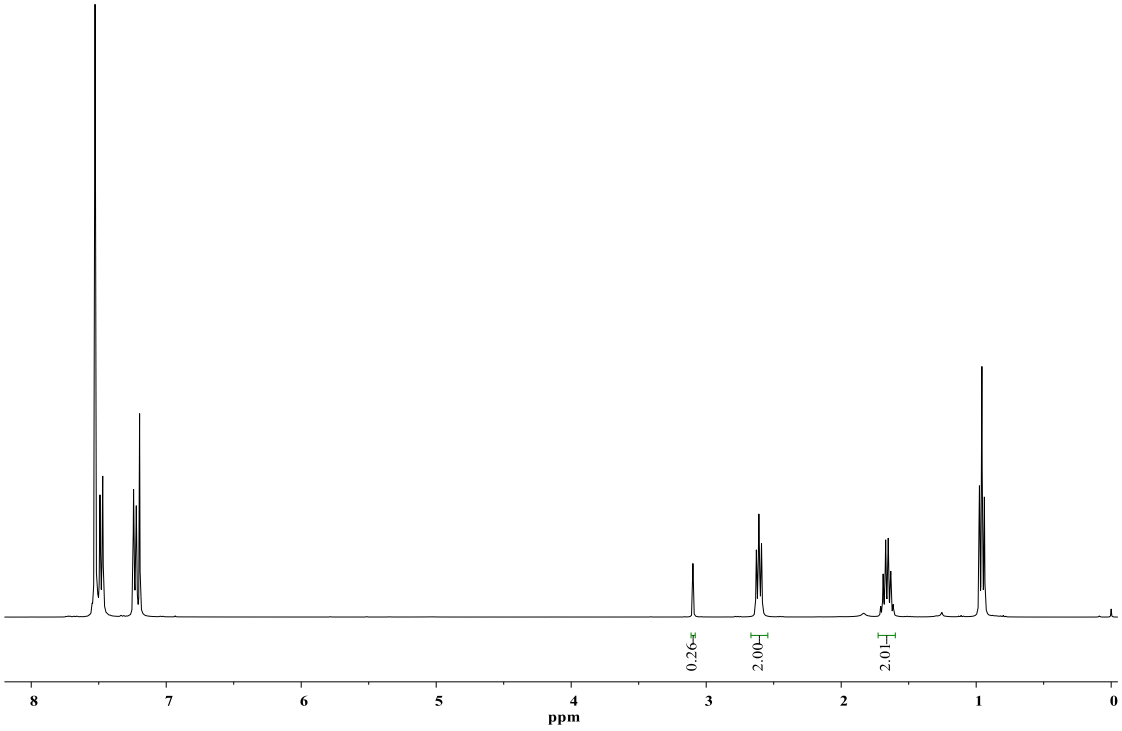


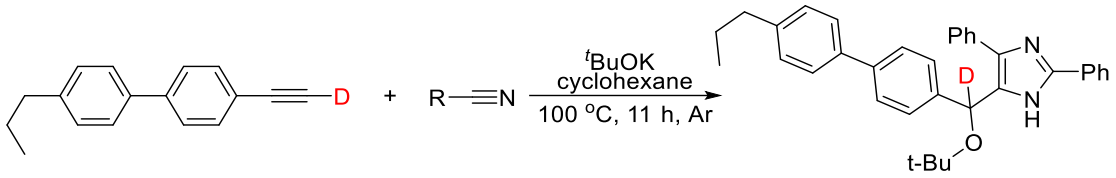
[*d*]-**1b,** 88%




tBuOK
cyclohexane, rt, air
30 min, then
D2O workup
H
D

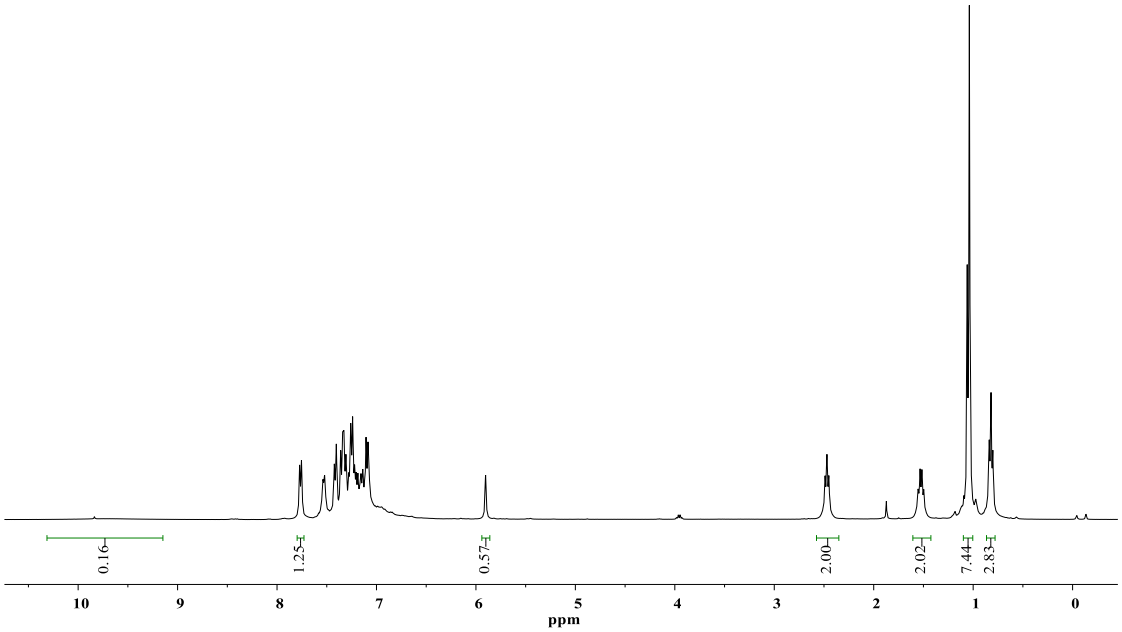
[*d*]-**1f,** 74%

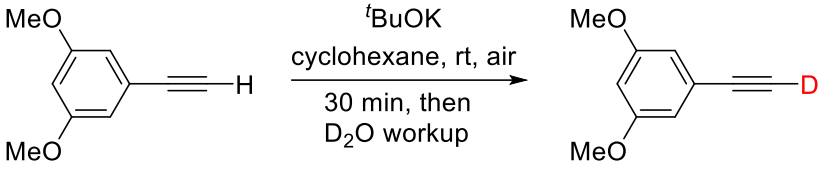

0.26
2.00
2.01
8
7
6
5
4
3
2
1
0
ppm


D
+
R—≡N
tBuOK
cyclohexane
100 °C, 11 h, Ar
Ph
N
Ph
D
N
H
O
t-Bu

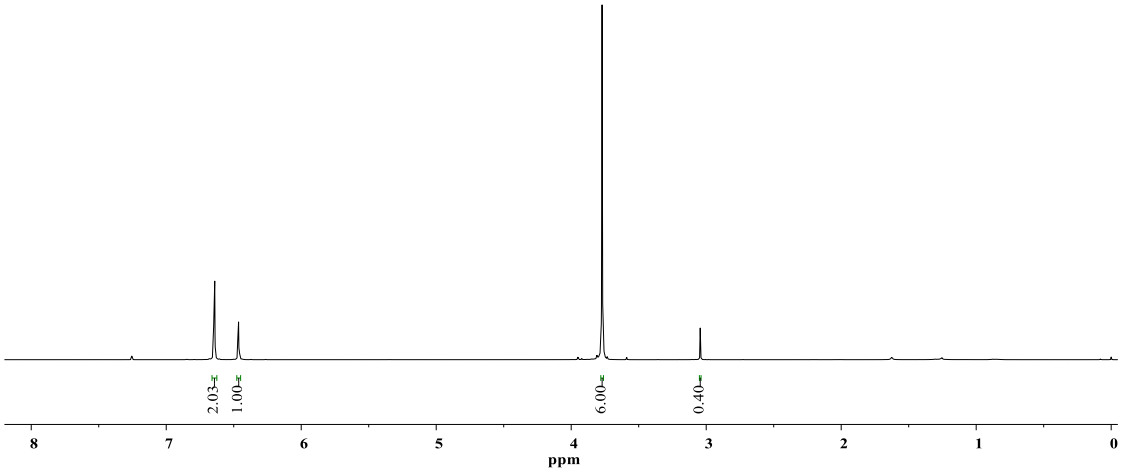
[*d*]-**1t,** 60%

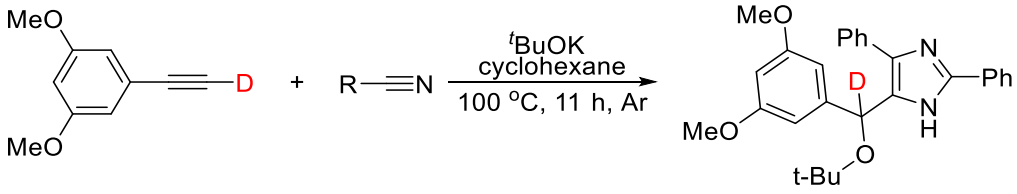
[*d*]-**4f,** 43%


0.16
1.25
0.57
2.00
2.02
7.44
2.83
10
9
8
7
6
5
4
3
2
1
0
ppm



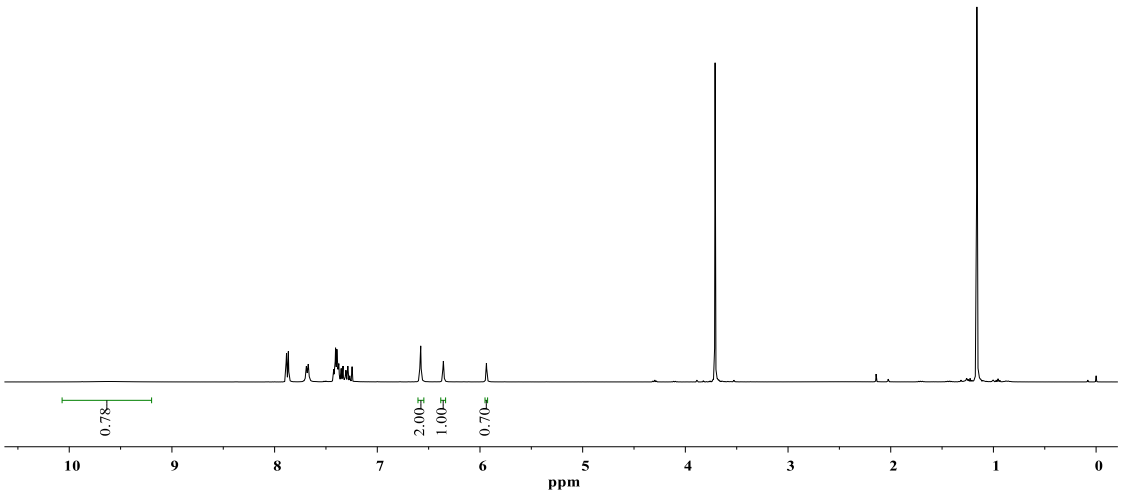
[*d*]-**1t,** 60%





[*d*]-**1t,** 60%

[*d*]-**4t,** 30%


0.78
2.00
1.00
0.70
10
9
8
7
6
5
4
3
2
1
0
ppm