Supporting Information

$K_2S_2O_8$-HFIP Synergistic Promoted *para*-selective sp^3 C-H Bond Diarylation of Glycine Esters

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1. General Information

^{1}H NMR spectra was recorded on 400 or 600 MHz (100 or 150 MHz for ^{13}C NMR) agilent NMR spectrometer with $CDCl_3$ as the solvent and tetramethylsilane (TMS) as the internal standard. Chemical shifts were reported in parts per million (ppm, δ scale) downfield from TMS at 0.00 ppm and referenced to the $CDCl_3$ at 7.26 ppm (for ^{1}H NMR) or 77.16 ppm (for ^{13}C NMR). High-resolution mass spectra were determined using GCT PremierTM (CI or ESI) or MALDI TOF Mass Spectrometer. Column chromatography was carried out on silica gel (200−300 mesh). All reactions were monitored using thin layer chromatography (TLC) on silica gel plates. All commercially available reagents, unless otherwise indicated, were used without further purification. The uncommercial ethyl *N*-arylglycinate **1a-h** and **1aa-ad** were readily prepared from 2-bromoacetate derivatives and the corresponding anilines (EP1220024).

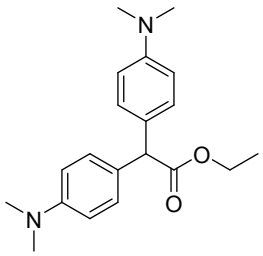
2. General Procedures (GP)

Cross-dehydrogenative coupling reaction (CDC) reaction of glycine derivatives with aromatic amines

Glycine derivatives **1** (0.25 mmol), aromatic amine **2** (0.75 mmol) and $K_2S_2O_8$ (0.50 mmol) were dissolved in HFIP (2 mL). The reaction mixture was stirred at 65 °C for 12 h under open air. The reaction mixture was cooled to room temperature. After removal of the solvent, the residue was purified by column chromatography on silica gel (**PE** : **EA** = 12 : 1) to afford corresponding products **3**.

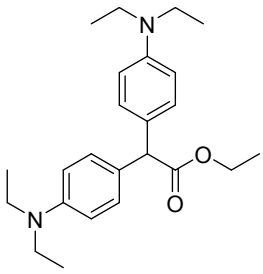
3. Characterization of Synthesized Compounds 3a-x and 3aa-ac

Ethyl 2, 2-bis(4-(dimethylamino)phenyl)acetate (3a)



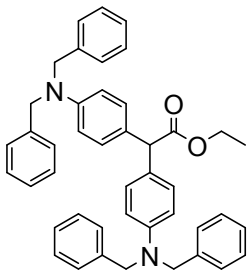
According to **GP**, ethyl (4-methoxyphenyl)glycinate **1e** (52 mg, 0.25 mmol) and *N*,*N*-dimethylaniline **2a** (91 mg, 0.75 mmol) were converted to the desired product **3a** (75 mg, 93%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17 (d, *J* = 8.4 Hz, 4H), 6.68 (d, *J* = 8.5 Hz, 4H), 4.83 (s, 1H), 4.17 (q, *J* = 7.0 Hz, 2H), 2.91 (s, 12H), 1.24 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 173.6, 149.6, 129.1, 127.5, 112.6, 60.8, 55.3, 40.6, 14.2. HRMS (CI) calcd for $C_{20}H_{27}N_2O_2$ $[M+H]^+$: 327.2073, found 327.2061.

Ethyl 2, 2-bis(4-(diethylamino)phenyl)acetate (3b)



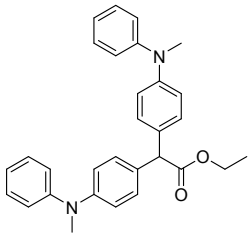
According to **GP**, ethyl (4-methoxyphenyl)glycinate **1e** (52 mg, 0.25 mmol) and *N*,*N*-diethylaniline **2b** (112 mg, 0.75 mmol) were converted to the desired product **3b** (87 mg, 92%) as a tawny solid (mp 58.7–61.4 ℃). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.15 (d, *J* = 8.4 Hz, 4H), 6.61 (d, *J* = 8.3 Hz, 4H), 4.79 (s, 1H), 4.18 (q, *J* = 7.0 Hz, 2H), 3.32 (q, *J* = 6.8 Hz, 8H), 1.26 (t, *J* = 7.0 Hz, 3H), 1.13 (t, *J* = 6.9 Hz, 12H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 173.8, 146.7, 129.3, 126.2, 111.6, 60.7, 55.2, 44.3, 14.2,12.6. HRMS (CI) calcd for $C_{24}H_{34}N_2O_2$ M^+: 382.2620, found 382.2604.

Ethyl 2, 2-bis(4-(dibenzylamino)phenyl)acetate (3c)



According to **GP**, ethyl (4-methoxyphenyl)glycinate **1e** (52 mg, 0.25 mmol) and *N*,*N*-dibenzylaniline **2c** (205 mg, 0.75 mmol) were converted to the desired product **3c** (147 mg, 93%) as a cyan oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.26 (m, 8H), 7.22 (d, *J* = 7.6 Hz, 12H), 7.09 (d, *J* = 8.4 Hz, 4H), 6.65 (d, *J* = 8.5 Hz, 4H), 4.74 (s, 1H), 4.60 (s, 8H), 4.13 (q, *J* = 7.1 Hz, 2H), 1.22 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 173.5, 148.1, 138.6, 129.2, 128.6, 127.5, 126.8, 126.7, 112.3, 60.8, 55.3, 54.2, 14.2. HRMS (ESI) calcd for $C_{44}H_{42}N_2O_2Na$ $[M+Na]^+$: 653.3144, found 653.3154.

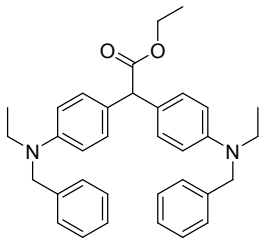
Ethyl 2,2-bis(4-(methyl(phenyl)amino)phenyl)acetate (3e)



According to **GP**, ethyl (4-methoxyphenyl)glycinate **1e** (52 mg, 0.25 mmol) and *N*-methyl-*N*-phenylaniline **2e** (137 mg, 0.75 mmol) were converted to the desired product **3e** (60 mg, 53%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.28 (d, *J* = 7.5 Hz, 4H), 7.22 (d, *J* = 8.1 Hz, 4H), 7.03 (d, *J* = 7.9 Hz, 4H), 6.95 (d, *J* = 7.9 Hz, 6H), 4.89 (s, 1H), 4.20 (q, *J* = 7.0 Hz, 2H), 3.29 (s, 6H), 1.27 (t, *J* = 6.9 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ

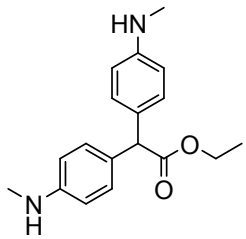
173.1 , 148.8, 148.0, 131.5, 129.3, 129.2, 121.7, 121.0, 119.8, 61.1, 55.8, 40.2, 14.2. HRMS (CI) calcd for $C_{30}H_{31}N_2O_2$ $[M+H]^+$: 451.2386, found 451.2389.

Ethyl 2-(4-(benzyl(ethyl)amino)phenyl)-2-(4-(ethyl(phenyl)amino)phenyl)acetate (3f)



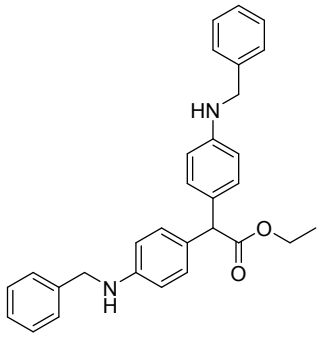
According to **GP**, ethyl (4-methoxyphenyl)glycinate **1e** (52 mg, 0.25 mmol) and *N*-benzyl-*N*-ethylaniline **2f** (158 mg, 0.75 mmol) were converted to the desired product **3f** (111 mg, 88%) as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 – 7.27 (m, 4H), 7.22 (d, J = 7.0 Hz, 6H), 7.11 (d, J = 8.4 Hz, 4H), 6.61 (d, J = 8.5 Hz, 4H), 4.75 (s, 1H), 4.47 (s, 4H), 4.15 (q, J = 7.1 Hz, 2H), 3.43 (q, J = 6.9 Hz, 4H), 1.23 (t, J = 7.1 Hz, 3H), 1.17 (t, J = 7.0 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.7, 147.5, 139.4, 129.3, 128.5, 126.9, 126.7, 126.6 112.1, 60.8, 55.3, 54.0, 45.1, 14.2, 12.1. HRMS (ESI) calcd for $C_{34}H_{38}N_2O_2Na$ $[M+Na]^+$: 529.2831, found 529.2828.

Ethyl 2,2-bis(4-(methylamino)phenyl)acetate (3g)



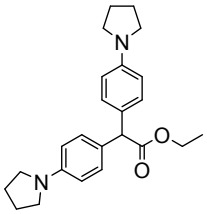
According to **GP**, ethyl (4-methoxyphenyl)glycinate **1e** (52 mg, 0.25 mmol) and *N*-methylaniline **2g** (80 mg, 0.75 mmol) were converted to the desired product **3g** (45 mg, 63%) as a red solid (mp 141.2–145.5 ℃). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.12 (d, J = 8.0 Hz, 4H), 6.55 (d, J = 8.0 Hz, 4H), 4.80 (s, 1H), 4.17 (q, J = 7.0 Hz, 2H), 3.66 (s, 2H), 2.81 (s, 6H), 1.24 (t, J = 7.0 Hz, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 173.6, 148.2, 129.3, 128.2, 112.4, 60.8, 55.5, 30.8, 14.2. HRMS (CI) calcd for $C_{18}H_{20}N_2O_2$ $[M-2H]^+$: 296.1525, found 296.1523.

Ethyl 2,2-bis(4-(benzylamino)phenyl)acetate (3h)



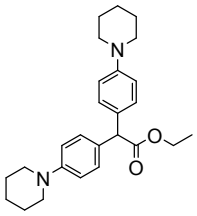
According to **GP**, ethyl (4-methoxyphenyl)glycinate **1e** (52 mg, 0.25 mmol) and *N*-benzylaniline **2h** (137 mg, 0.75 mmol) were converted to the desired product **3h** (57 mg, 51%) as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.30 (m, 8H), 7.29 – 7.25 (m, 2H), 7.10 (d, J = 8.3 Hz, 4H), 6.57 (d, J = 8.4 Hz, 4H), 4.78 (s, 1H), 4.29 (s, 4H), 4.16 (q, J = 7.1 Hz, 2H), 4.00 (s, 2H), 1.23 (t, J = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.5, 147.1, 139.5, 129.4, 128.6, 128.5, 127.5, 127.2, 112.8, 60.0, 55.5, 48.4, 14.2. HRMS (CI) calcd for $C_{30}H_{31}N_2O_2$ $[M+H]^+$:451.2386, found 451.2373.

Ethyl 2, 2-bis(4-(pyrrolidin-1-yl)phenyl)acetate (3j)



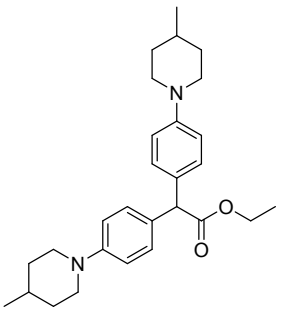
According to **GP**, ethyl (4-methoxyphenyl)glycinate **1e** (52 mg, 0.25 mmol) and 1-phenylpyrrolidine **2j** (110 mg, 0.75 mmol) were converted to the desired product **3j** (71 mg, 76%) as a brown oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.15 (d, *J* = 8.4 Hz, 4H), 6.50 (d, *J* = 8.4 Hz, 4H), 4.82 (s, 1H), 4.17 (q, *J* = 7.1 Hz, 2H), 3.24 (d, *J* = 6.0 Hz, 8H), 1.97 (s, 8H), 1.24 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 173.8 , 146.8 , 129.2, 126.3, 111.5, 60.7, 55.4, 47.6, 25.4, 14.2. HRMS (CI) calcd for $C_{24}H_{31}N_2O_2$ $[M+H]^+$: 379.2386, found 379.2375.

Ethyl 2,2-bis(4-(piperidin-1-yl)phenyl)acetate (3k)



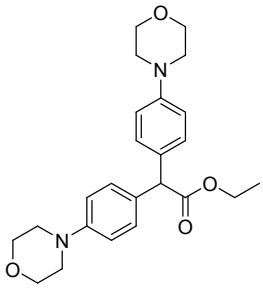
According to **GP**, ethyl (4-methoxyphenyl)glycinate **1e** (52 mg, 0.25 mmol) and 1-phenylpiperidine **2k** (121 mg, 0.75 mmol) were converted to the desired product **3k** (74 mg, 74%) as a white solid (mp 100.3–103.5 ℃). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17 (d, *J* = 8.4 Hz, 4H), 6.86 (d, *J* = 8.4 Hz, 4H), 4.84 (s, 1H), 4.18 (q, *J* = 7.1 Hz, 2H), 3.14 – 3.09 (m, 8H), 1.68 (d, *J* = 4.2 Hz, 8H), 1.56 (d, *J* = 5.0 Hz, 4H), 1.24 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.3, 151.1, 129.8 129.1, 116.3, 60.8, 55.5, 50.6, 25.9, 24.3, 14.2. HRMS (CI) calcd for $C_{26}H_{35}N_2O_2$ $[M+H]^+$: 407.2699, found 407.2701.

Ethyl 2,2-bis(4-(4-methylpiperidin-1-yl)phenyl)acetate (3l)



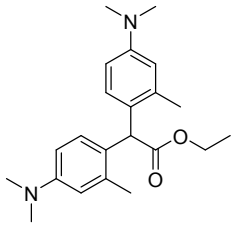
According to **GP**, ethyl (4-methoxyphenyl)glycinate **1e** (52 mg, 0.25 mmol) and 4-methyl-1-phenylpiperidine **2l** (131 mg, 0.75 mmol) were converted to the desired product **3l** (80 mg, 74%) as a yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16 (d, *J* = 8.5 Hz, 4H), 6.86 (d, *J* = 8.6 Hz, 4H), 4.83 (s, 1H), 4.17 (q, *J* = 7.1 Hz, 2H), 3.61 (d, *J* = 12.1 Hz, 4H), 2.65 (t, *J* = 11.3 Hz, 4H), 1.71 (d, *J* = 12.4 Hz, 4H), 1.54 – 1.44 (m, 2H), 1.38 – 1.29 (m, 4H), 1.24 (t, *J* = 7.1 Hz, 3H), 0.96 (d, *J* = 6.4 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.3, 150.8, 129.7, 129.1, 116.3, 60.9, 55.5, 49.9, 34.2, 30.7, 21.9, 14.2. HRMS (CI) calcd for $C_{28}H_{39}N_2O_2$ $[M+H]^+$: 435.3012, found 435.3019.

Ethyl 2,2-bis(4-morpholinophenyl)acetate (3m)



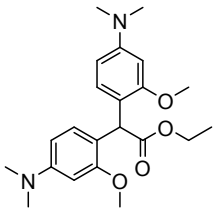
According to **GP**, ethyl (4-methoxyphenyl)glycinate **1e** (52 mg, 0.25 mmol) and 4-phenylmorpholine **2m** (122 mg, 0.75 mmol) were converted to the desired product **3m** (84 mg, 82%) as a brown oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20 (d, *J* = 8.5 Hz, 4H), 6.85 (d, *J* = 8.6 Hz, 4H), 4.87 (s, 1H), 4.18 (q, *J* = 7.1 Hz, 2H), 3.86 – 3.81 (m, 8H), 3.16 – 3.10 (m, 8H), 1.24 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.1, 150.2, 130.5, 129.3, 115.6, 66.9, 61.0, 55.5, 49.2, 14.2. HRMS (CI) calcd for $C_{24}H_{31}N_2O_4$ $[M+H]^+$: 411.2284, found 411.2288.

Ethyl 2,2-bis(4-(dimethylamino)-2-methylphenyl)acetate (3n)



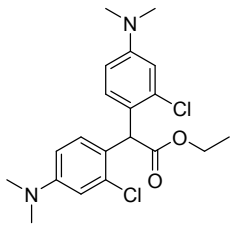
According to **GP**, ethyl (4-methoxyphenyl)glycinate **1e** (52 mg, 0.25 mmol) and *N*,*N*,3-trimethylaniline **2n** (101 mg, 0.75 mmol) were converted to the desired product **3n** (68 mg, 78%) as a white solid (mp 128.1–132.3 ℃). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.94 (d, *J* = 8.5 Hz, 2H), 6.57 – 6.50 (m, 4H), 5.09 (s, 1H), 4.19 (q, *J* = 7.1 Hz, 2H), 2.91 (s, 12H), 2.21 (s, 6H), 1.25 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.9, 149.5, 136.9, 129.0, 125.6, 114.8, 110.4, 60.8, 49.5, 40.7, 20.1, 14.3. HRMS (CI) calcd for $C_{22}H_{31}N_2O_2$ $[M+H]^+$: 355.2386, found 355.2375.

Ethyl 2,2-bis(4-(dimethylamino)-2-methoxyphenyl)acetate (3p)



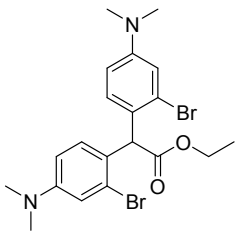
According to **GP**, ethyl (4-methoxyphenyl)glycinate **1e** (52 mg, 0.25 mmol) and 3-methoxy-*N*,*N*-dimethylaniline **2p** (113 mg, 0.75 mmol) were converted to the desired product **3p** (86 mg, 89%) as a white solid (mp 158.4–161.5 ℃). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.90 (d, *J* = 8.3 Hz, 2H), 6.29 – 6.23 (m, 4H), 5.39 (s, 1H), 4.16 (q, *J* = 7.1 Hz, 2H), 3.79 (s, 6H), 2.93 (s, 12H), 1.22 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 174.5, 158.0, 151.1, 129.6, 115.9, 104.7, 96.4, 60.4, 55.4, 43.4, 40.8, 14.4. HRMS (CI) calcd for $C_{22}H_{31}N_2O_4$ $[M+H]^+$: 387.2284, found 387.2285.

Ethyl 2,2-bis(2-chloro-4-(dimethylamino)phenyl)acetate (3r)



According to **GP**, ethyl (4-methoxyphenyl)glycinate **1e** (52 mg, 0.25 mmol) and 3-chloro-*N*,*N*-dimethylaniline **2r** (116 mg, 0.75 mmol) were converted to the desired product **3r** (91 mg, 92%) as a white solid (mp 132.2–135.1 ℃). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.94 (d, *J* = 8.7 Hz, 2H), 6.73 (d, *J* = 2.3 Hz, 2H), 6.54 (dd, *J* = 8.7, 2.3 Hz, 2H), 5.58 (s, 1H), 4.22 (q, *J* = 7.1 Hz, 2H), 2.92 (s, 12H), 1.25 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.7, 150.4, 135.1, 130.0, 123.2, 113.1, 110.8, 61.1, 50.0, 40.3, 14.20. HRMS (CI) calcd for $C_{20}H_{25}Cl_2N_2O_2$ $[M+H]^+$: 395.1293, found 395.1275.

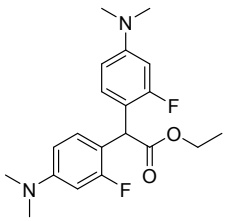
Ethyl 2,2-bis(2-bromo-4-(dimethylamino)phenyl)acetate (3s)



According to **GP**, ethyl (4-methoxyphenyl)glycinate **1e** (52 mg, 0.25 mmol) and 3-bromo-*N*,*N*-dimethylaniline **2s** (150 mg, 0.75 mmol) were converted to the desired product **3s** (88 mg, 73%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.93 (d, *J* = 2.9 Hz, 3H), 6.90 (s, 1H), 6.60 (d, *J* = 2.7 Hz, 1H), 6.58 (d, *J* = 2.7 Hz, 1H), 5.54 (s, 1H), 4.23 (q, *J* = 7.1 Hz, 2H), 2.93 (s, 12H), 1.27 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 172.7, 150.5, 129.9, 126.1, 124.9,

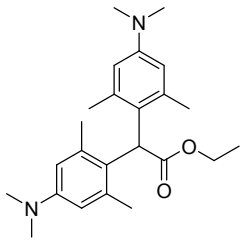
116.3, 111.4, 61.2, 55.1, 40.3, 14.3. HRMS (CI) calcd for $C_{20}H_{25}{}^{79}Br_2N_2O_2$ $[M+H]^+$: 483.0283, found 483.0287.

Ethyl 2,2-bis(4-(dimethylamino)-2-fluorophenyl)acetate (3t)



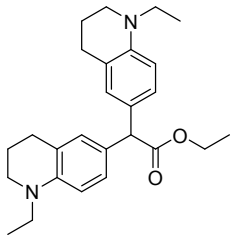
According to **GP**, ethyl (4-methoxyphenyl)glycinate **1e** (52 mg, 0.25 mmol) and 3-fluoro-*N,N*-dimethylaniline **2t** (104 mg, 0.75 mmol) were converted to the desired product **3t** (79 mg, 87%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.03 (t, *J* = 9.0 Hz, 2H), 6.43 (d, *J* = 2.6 Hz, 1H), 6.40 (s, 2H), 6.37 (d, *J* = 2.5 Hz, 1H), 5.30 (s, 1H), 4.20 (q, *J* = 7.1 Hz, 2H), 2.91 (s, 12H), 1.24 (t, *J* = 7.1 Hz, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 172.5, 161.4 (d, *J* = 243.1 Hz), 151.3 (d, *J* = 10.9 Hz), 130.1 (d, *J* = 6.1 Hz), 112.6 (d, *J* = 15.8 Hz), 107.9 (d, *J* = 2.0 Hz), 99.3 (d, *J* = 26.7 Hz), 61.1, 42.1, 40.4, 14.2. HRMS (CI) calcd for $C_{20}H_{25}F_2N_2O_2$ $[M+H]^+$: 363.1884, found 363.1877.

Ethyl 2,2-bis(4-(dimethylamino)-2,6-dimethylphenyl)acetate (3u)



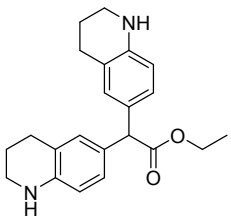
According to **GP**, ethyl (4-methoxyphenyl)glycinate **1e** (52 mg, 0.25 mmol) and *N*,*N*,3,5-tetramethylaniline **2s** (112 mg, 0.75 mmol) were converted to the desired product **3s** (69 mg, 72%) as a yellow solid (mp 91.5–95.7 ℃). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.38 (s, 4H), 5.26 (s, 1H), 4.23 (q, *J* = 7.0 Hz, 2H), 2.89 (s, 12H), 2.09 (s, 12H), 1.27 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 175.2, 148.9, 138.1, 125.3, 113.9, 60.8, 50.1, 40.6, 21.5, 14.2. HRMS (CI) calcd for $C_{24}H_{35}N_2O_2$ $[M+H]^+$: 383.2699, found 383.2696.

Ethyl 2,2-bis(1-ethyl-1,2,3,4-tetrahydroquinolin-6-yl)acetate (3w)



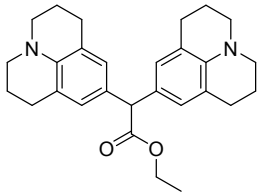
According to **GP**, ethyl (4-methoxyphenyl)glycinate **1e** (52 mg, 0.25 mmol) and 1-ethyl-1,2,3,4-tetrahydroquinoline **2u** (121 mg, 0.75 mmol) were converted to the desired product **3u** (67 mg, 66%) as a red solid (mp 80.8–85.6 ℃). ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.98 (d, *J* = 8.4 Hz, 2H), 6.88 (s, 2H), 6.51 (d, *J* = 8.5 Hz, 2H), 4.68 (s, 1H), 4.16 (q, *J* = 7.1 Hz, 2H), 3.30 (q, *J* = 7.0 Hz, 4H), 3.24 – 3.17 (m, 4H), 2.70 (t, *J* = 6.2 Hz, 4H), 1.95 – 1.87 (m, 4H), 1.24 (t, *J* = 7.1 Hz, 3H), 1.10 (t, *J* = 7.0 Hz, 6H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.9, 143.9, 129.2, 127.0, 126.3, 122.3, 110.4, 60.7, 55.4, 48.4, 45.4, 28.2, 22.3, 14.3, 10.9.HRMS (CI) calcd for $C_{26}H_{34}N_2O_2$ $[M]^+$: 406.2620, found 406.2629.

Ethyl 2,2-bis(1,2,3,4-tetrahydroquinolin-6-yl)acetate (3x)



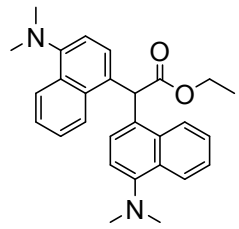
According to **GP**, ethyl (4-methoxyphenyl)glycinate **1e** (52 mg, 0.25 mmol) and 1,2,3,4-tetrahydroquinoline **2v** (100 mg, 0.75 mmol) were converted to the desired product **3v** (41 mg, 48%) as a brown oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.89 (d, *J* = 10.5 Hz, 4H), 6.40 (d, *J* = 8.0 Hz, 2H), 4.69 (s, 1H), 4.16 (q, *J* = 7.1 Hz, 2H), 3.80 (d, *J* = 46.4 Hz, 2H), 3.30 – 3.22 (m, 4H), 2.71 (t, *J* = 6.2 Hz, 4H), 1.94 – 1.86 (m, 4H), 1.24 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.7, 143.5, 129.6, 128.1, 126.8, 121.5, 114.3, 60.7, 55.7, 42.0, 27.0, 22.2, 14.2. HRMS (CI) calcd for $C_{22}H_{27}N_2O_2$ $[M+H]^+$: 351.2073, found 351.2086.

Ethyl 2,2-bis(2,3,6,7-tetrahydro-1*H*,5*H*-pyrido[3,2,1-*ij*]quinolin-9-yl)acetate (3y)



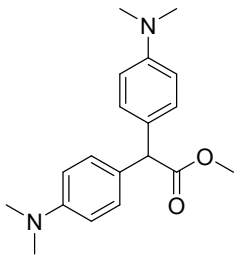
According to **GP**, ethyl (4-methoxyphenyl)glycinate **1e** (52 mg, 0.25 mmol) and 2,3,6,7-tetrahydro-1*H*,5*H*-pyrido[3,2,1-ij]quinoline **2w** (130 mg, 0.75 mmol) were converted to the desired product **3w** (17 mg, 15%) as a red oil ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.72 (s, 4H), 4.59 (s, 1H), 4.15 (q, *J* = 7.0 Hz, 2H), 3.10 – 3.05 (m, 8H), 2.71 (t, *J* = 6.3 Hz, 8H), 1.96 – 1.90 (m, 8H), 1.24 (t, *J* = 7.0 Hz, 3H).^{13}C NMR (150 MHz, $CDCl_3$) δ 173.8, 141.9, 126.8, 126.6, 121.4, 60.6, 55.5, 50.0, 27.6, 22.1, 14.2. HRMS (CI) calcd for $C_{28}H_{35}N_2O_2$ $[M+H]^+$: 431.2699, found 431.2684.

Ethyl 2, 2-bis(4-(dimethylamino)naphthalen-1-yl)acetate (3z)



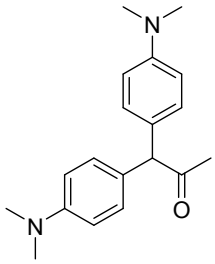
According to **GP**, ethyl (4-methoxyphenyl)glycinate **1e** (52 mg, 0.25 mmol) and *N*,*N*-dimethylnaphthalen-1-amine **2x** (128 mg, 0.75 mmol) were converted to the desired product **3x** (68 mg, 64%) as a brown solid (mp 145.3–149.5 ℃). ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.32 (d, *J* = 8.2 Hz, 2H), 7.93 (d, *J* = 8.2 Hz, 2H), 7.47 (dt, *J* = 15.1, 6.9 Hz, 4H), 7.19 (d, *J* = 7.8 Hz, 2H), 6.98 (d, *J* = 7.8 Hz, 2H), 6.36 (s, 1H), 4.28 (q, *J* = 7.1 Hz, 2H), 2.88 (s, 12H), 1.28 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 173.4, 132.9, 129.2 , 126.6, 126.4, 125.0, 123.6, 113.8, 61.3, 49.8, 45.3, 14.3. HRMS (CI) calcd for $C_{28}H_{31}N_2O_2$ $[M+H]^+$: 427.2386, found 427.2379.

Methyl 2,2-bis(4-(dimethylamino)phenyl)acetate (3aa)



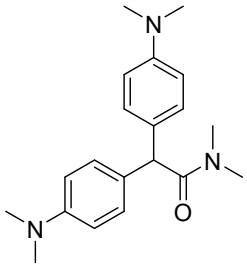
According to **GP**, methyl (4-methoxyphenyl)glycinate **1aa** (49 mg, 0.25 mmol) and *N*,*N*-dimethylaniline **2a** (91 mg, 0.75 mmol) were converted to the desired product **3aa** (73 mg, 93%) as a colorless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17 (d, *J* = 8.2 Hz, 4H), 6.69 (d, *J* = 8.3 Hz, 4H), 4.86 (s, 1H), 3.72 (s, 3H), 2.92 (s, 12H).^{13}C NMR (100 MHz, CDCl3) δ 174.1, 149.6, 129.2, 127.3, 112.7, 55.3, 52.1, 40.7. MALDI-TOF-MS calcd for $C_{19}H_{24}N_2O_2$ Mol.Wt.: 312.4130, found 312.4140.

1, 1-bis(4-(dimethylamino)phenyl)propan-2-one (3ab)



According to **GP**, 1-((4-methoxyphenyl)amino)propan-2-one **1ab** (41 mg, 0.25 mmol) and *N*,*N*-dimethylaniline **2a** (91 mg, 0.75 mmol) were converted to the desired product **3ab** (20 mg, 28%) as a brown oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.07 (d, *J* = 8.5 Hz, 4H), 6.69 (d, *J* = 8.5 Hz, 4H), 4.93 (s, 1H), 2.92 (s, 12H), 2.21 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$) δ 208.0, 149.6, 129.6 , 126.8, 112.8, 63.4, 40.6, 29.7.HRMS (CI) calcd for $C_{19}H_{25}N_2O$ $[M+H]^+$: 297.1967, found 297.1962.

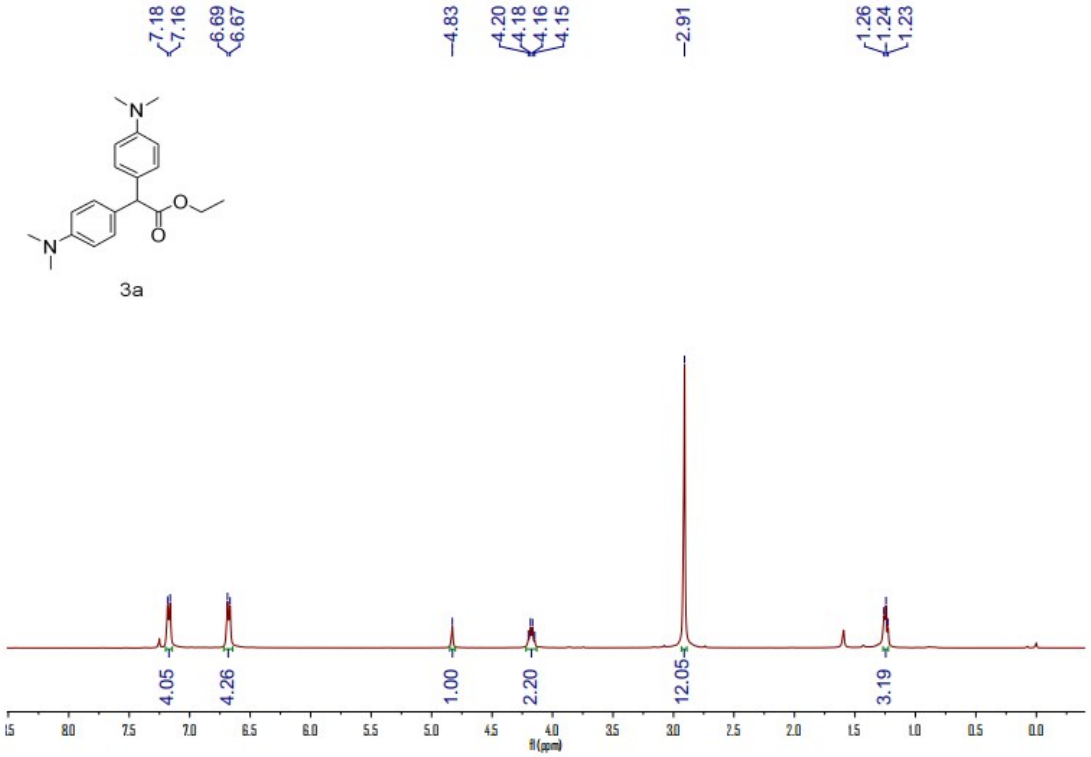
2, 2-bis(4-(dimethylamino)phenyl)-N,N-dimethylacetamide (3ac)

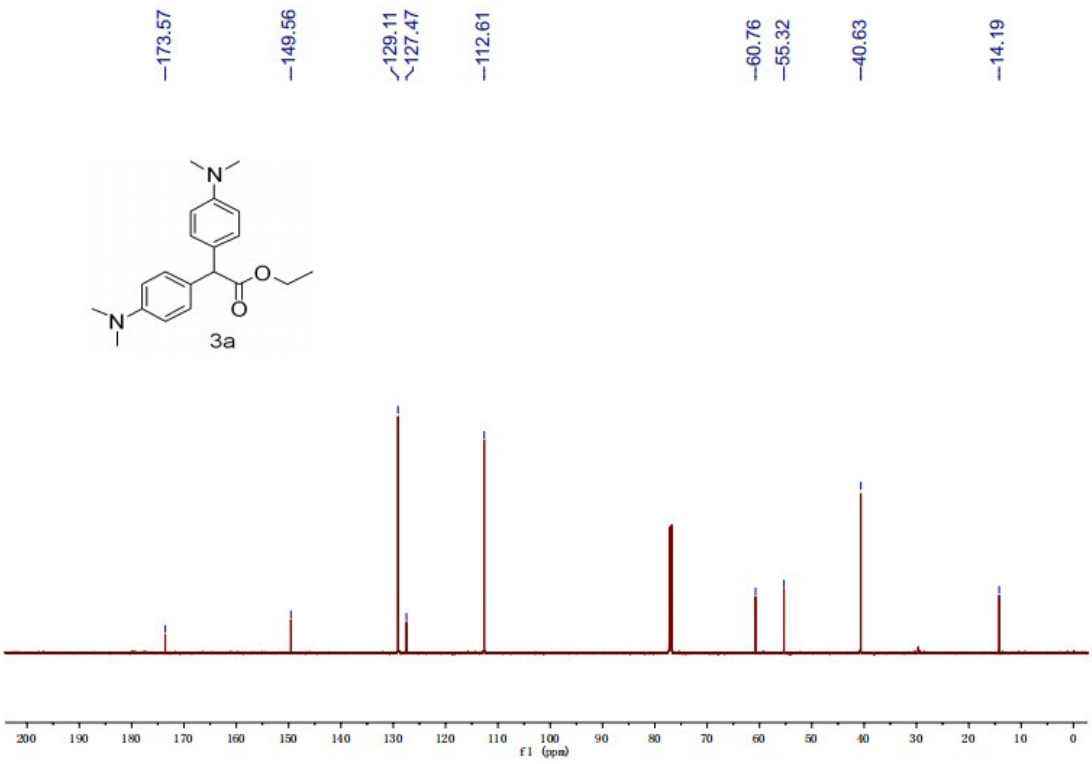


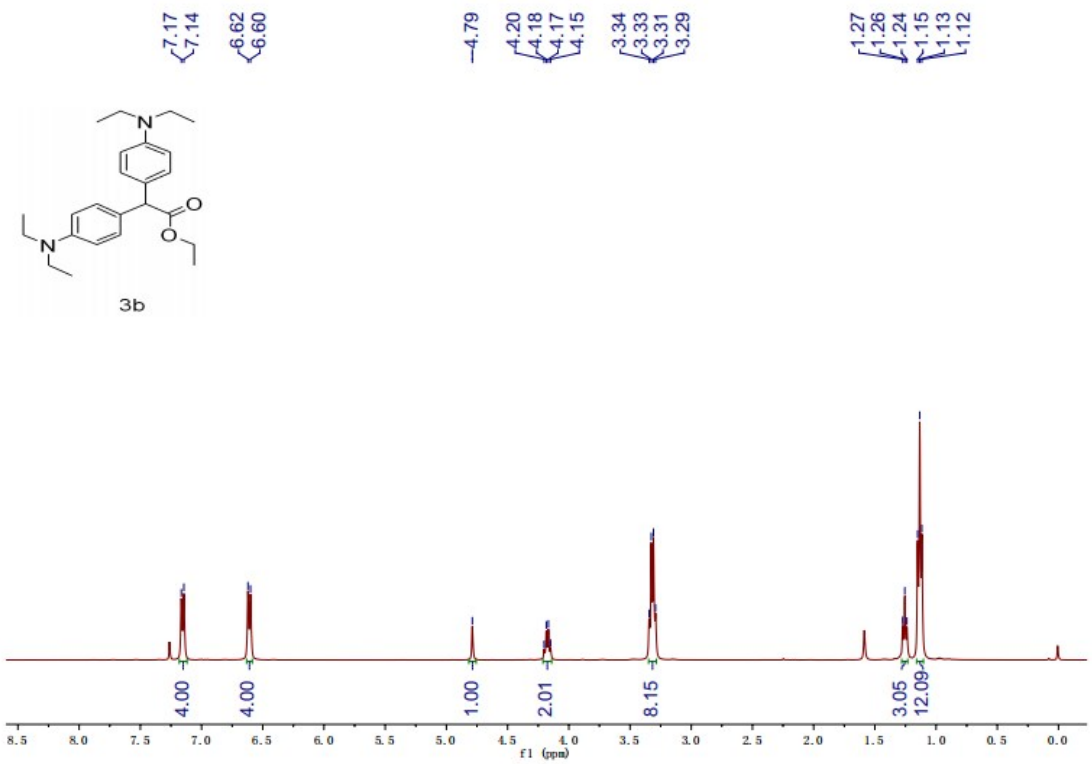
According to **GP**, 2-((4-methoxyphenyl)amino)-*N*,*N*-dimethylacetamide **1ac** (48 mg, 0.25 mmol) and *N*,*N*-dimethylaniline **2a** (91 mg, 0.75 mmol) were converted to the desired product **3ac** (45 mg, 56%) as a white solid (mp 152.1–155.5 ℃). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.10 (d, *J* = 8.5 Hz, 4H), 6.67 (d, *J* = 8.6 Hz, 4H), 5.03 (s, 1H), 2.99 (d, *J* = 6.1 Hz, 6H), 2.90 (s, 12H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 173.0 , 149.4, 129.5, 128.4, 112.7, 53.0, 40.7, 37.5, 36.0.HRMS (CI) calcd for $C_{20}H_{28}N_3O$ $[M+H]^+$: 326.2232, found 326.2221.

.

4. ^{1}H and ^{13}C-NMR Spectra



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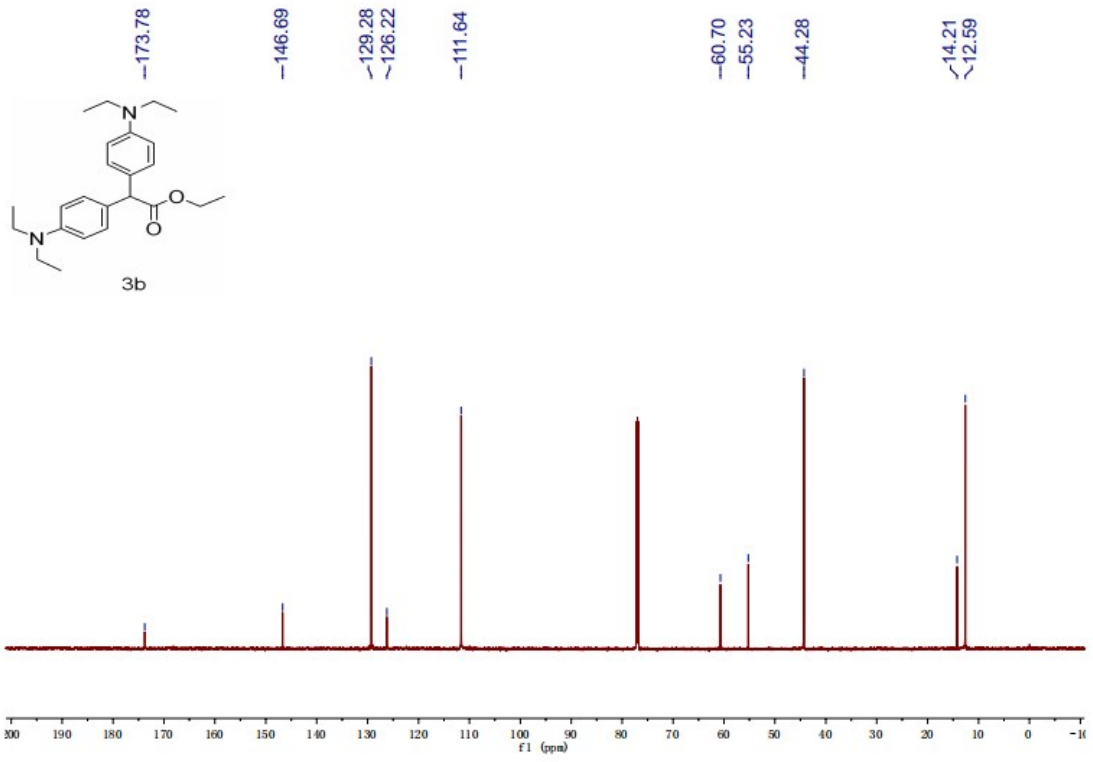
7.14
6.62
6.60
4.79
4.20
4.18
4.17
4.15
3.34
3.33
3.31
3.29
1.27
1.26
1.24
1.15
1.13
1.12
3b
4.00
4.00
1.00
2.01
8.15
3.05
12.09
f1 (ppm)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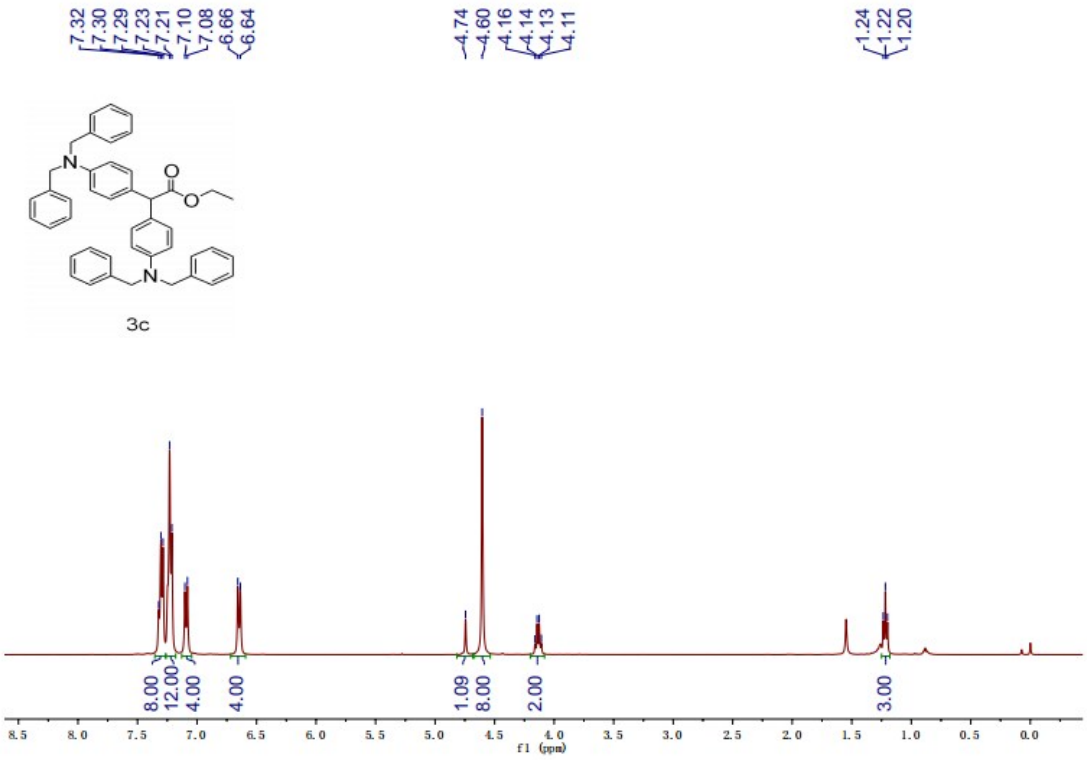
111.64
60.70
55.23
44.28
14.21
12.59
3b
f1 (ppm)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4.60
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4.14
4.13
4.11
1.24
1.22
1.20
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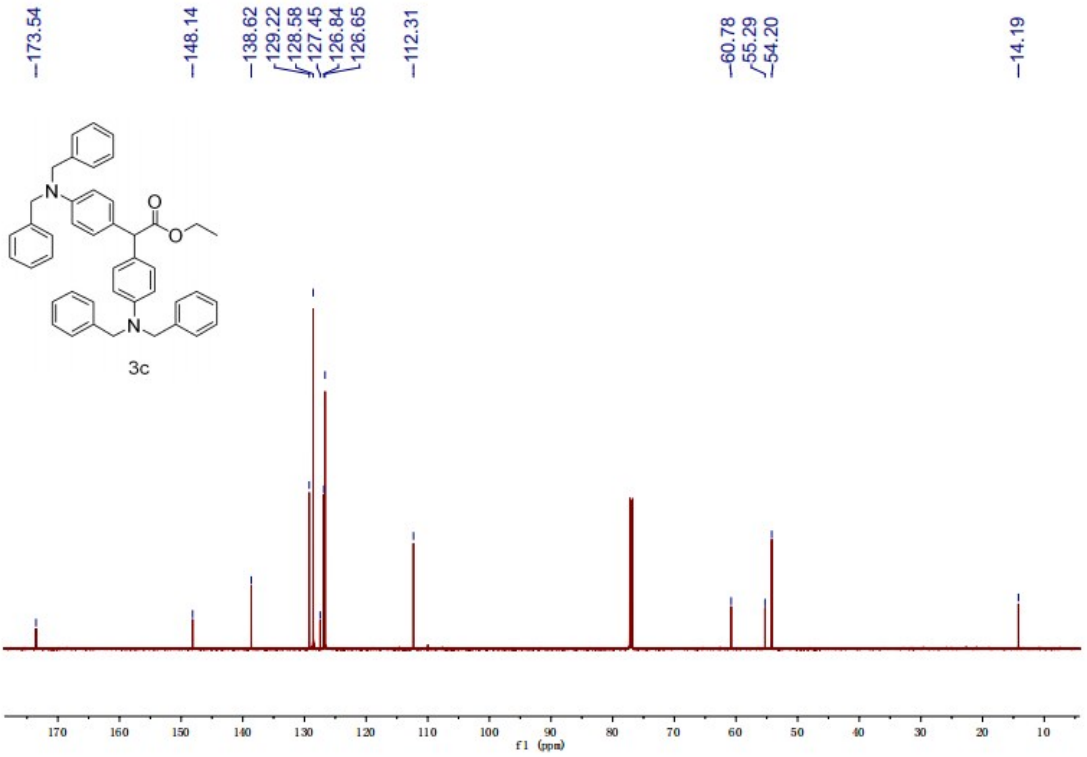
12.00
4.00
4.00
1.09
8.00
2.00
3.00
f1 (ppm)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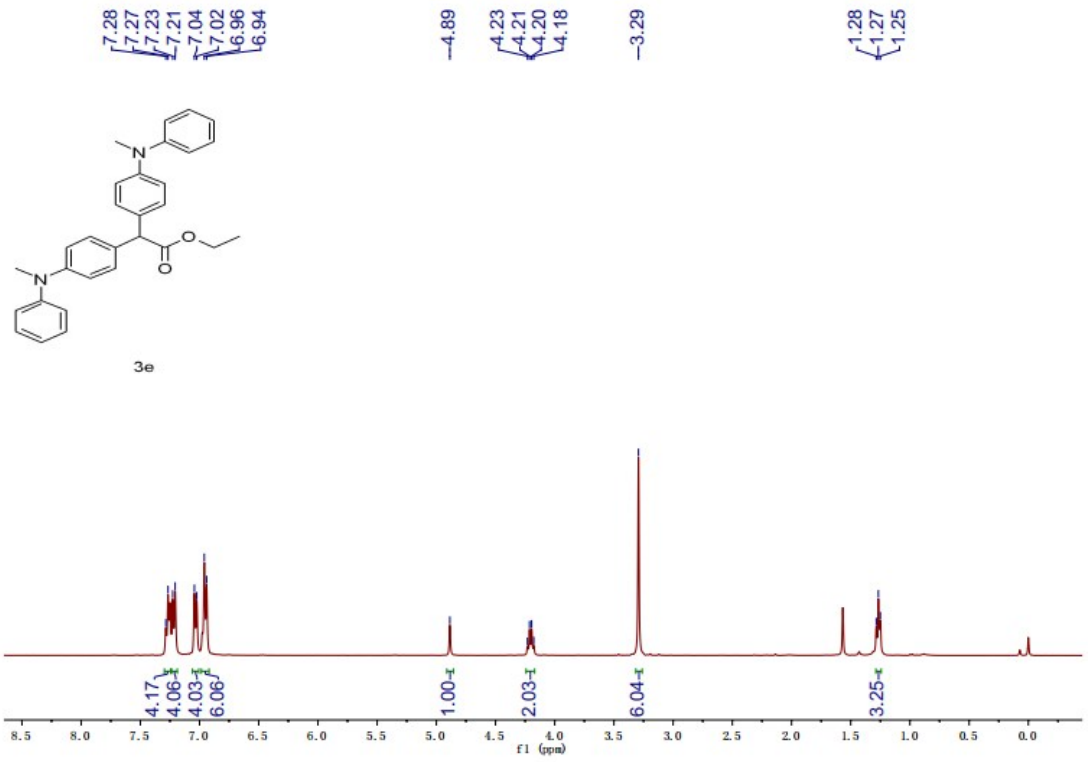
14.19
3c
f1 (ppm)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7.02
6.96
6.94
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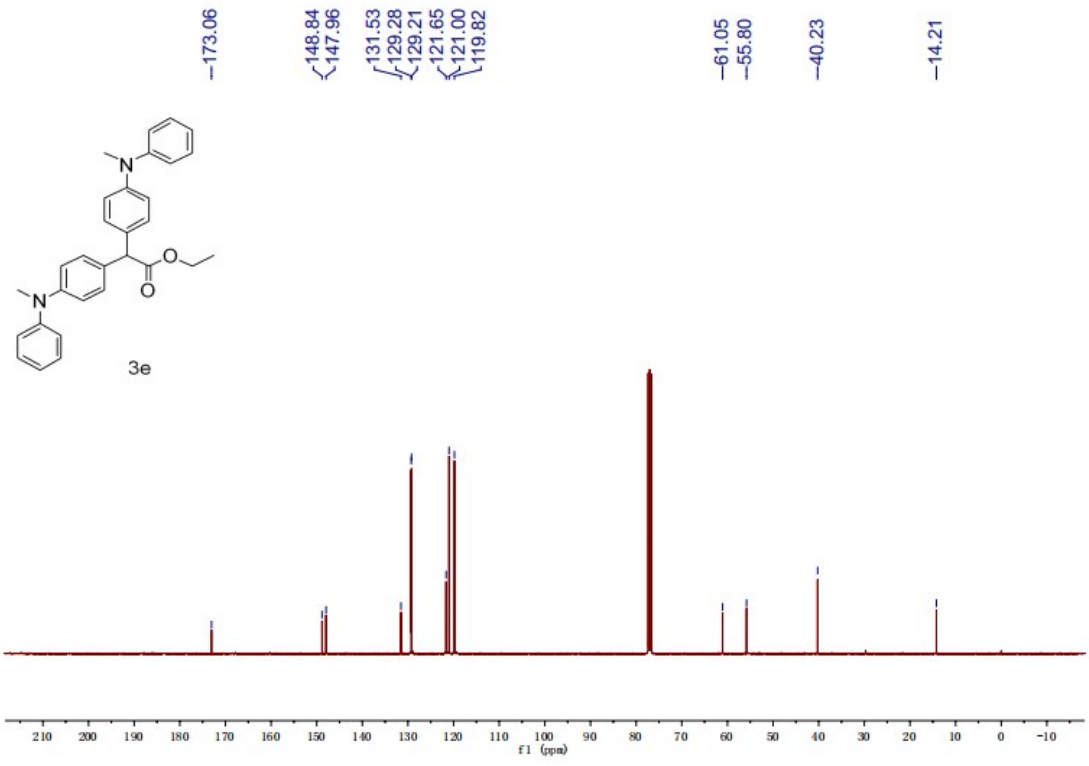
4.23
4.21
4.20
4.18
3.29
1.28
1.27
1.25
3e
4.17
4.06
4.03
6.06
1.00
2.03
6.04
3.25
f1 (ppm)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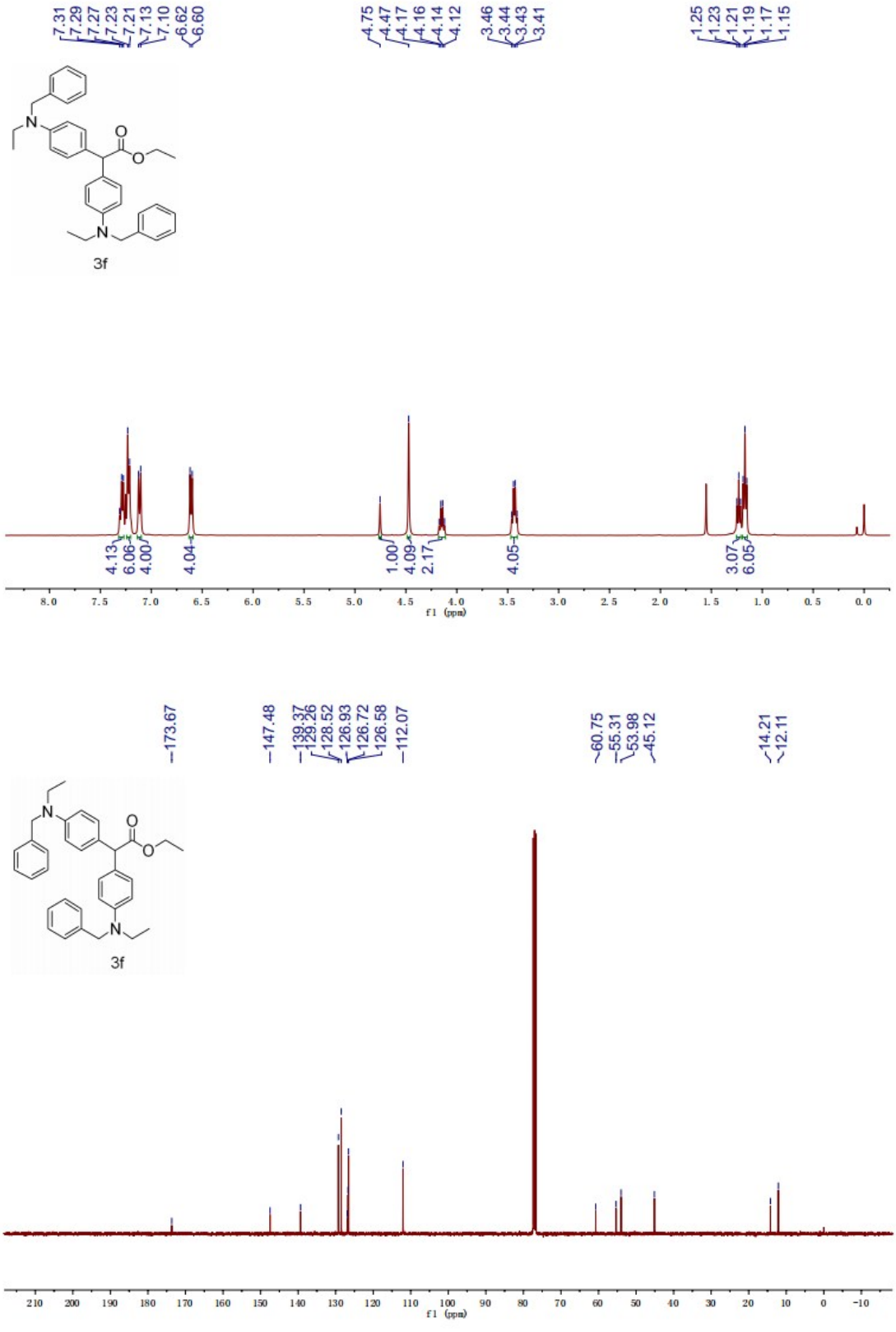
f1 (ppm)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4.16
4.14
4.12
3.46
3.44
3.43
3.41
1.25
1.23
1.21
1.19
1.17
1.15
3f
4.13
6.06
4.00
4.04
1.00
4.09
2.17
4.05
3.07
6.05
f1 (ppm)
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147.48
139.37
129.26
128.52
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112.07
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45.12
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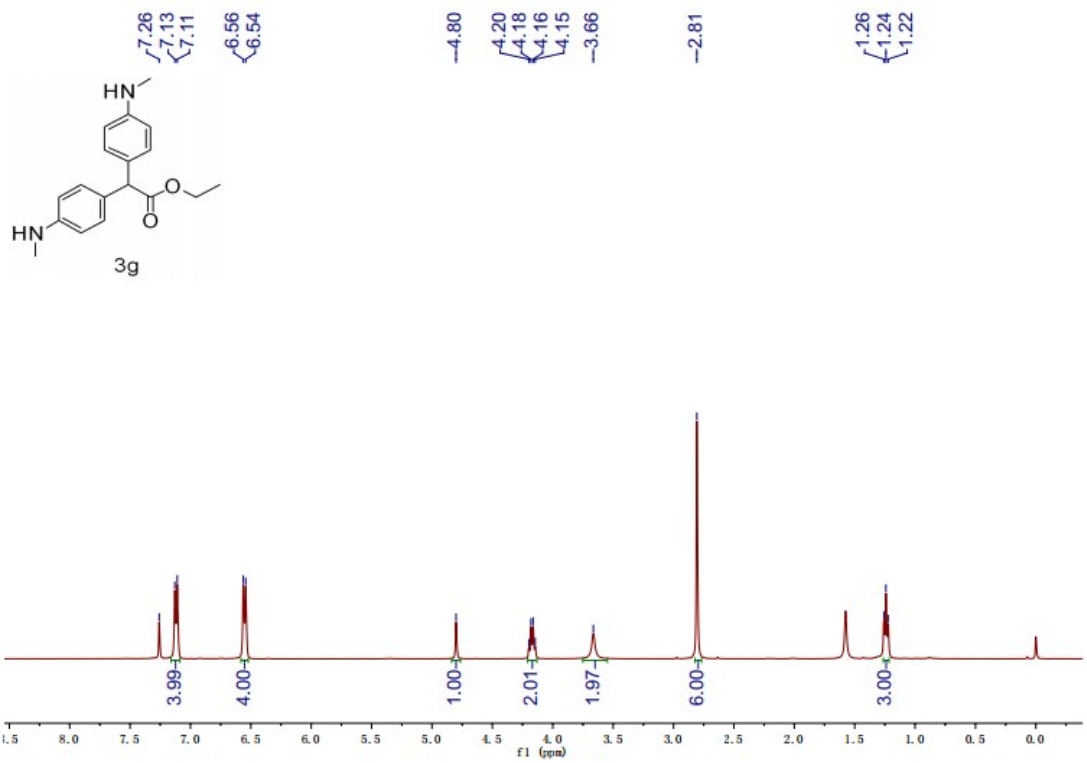
3f
f1 (ppm)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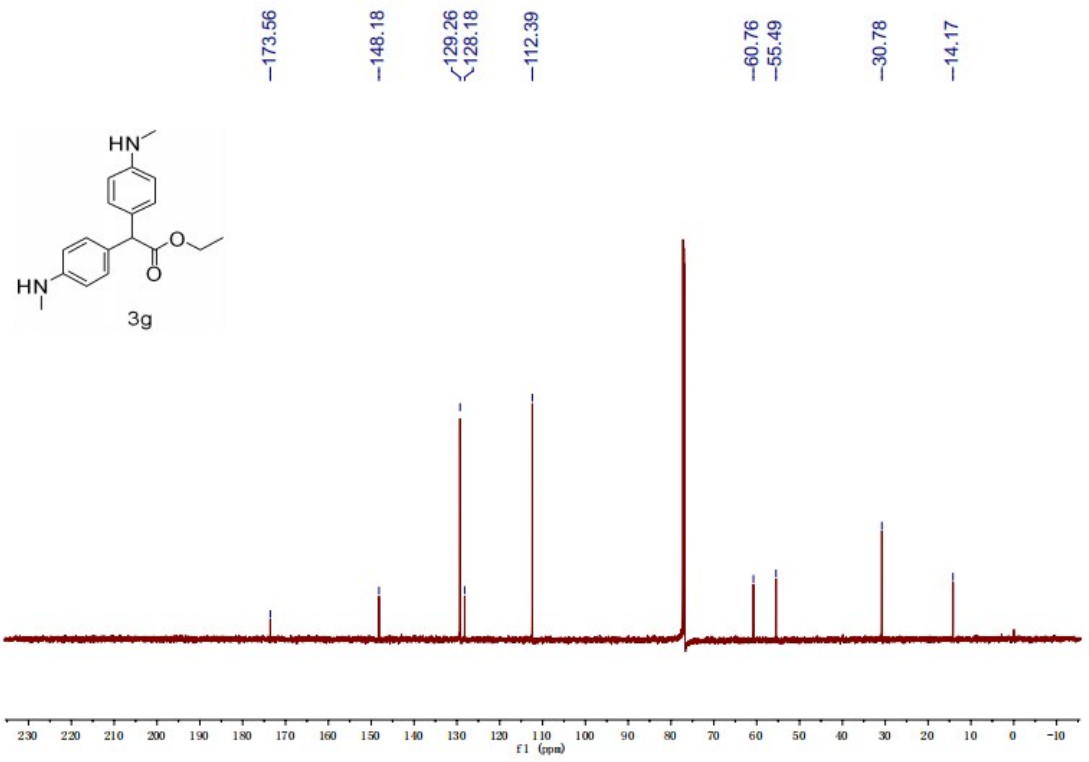
4.20
4.18
4.16
4.15
3.66
2.81
1.26
1.24
1.22
HN
O
O
HN
3g
3.99
4.00
1.00
2.01
1.97
6.00
3.00
f1 (ppm)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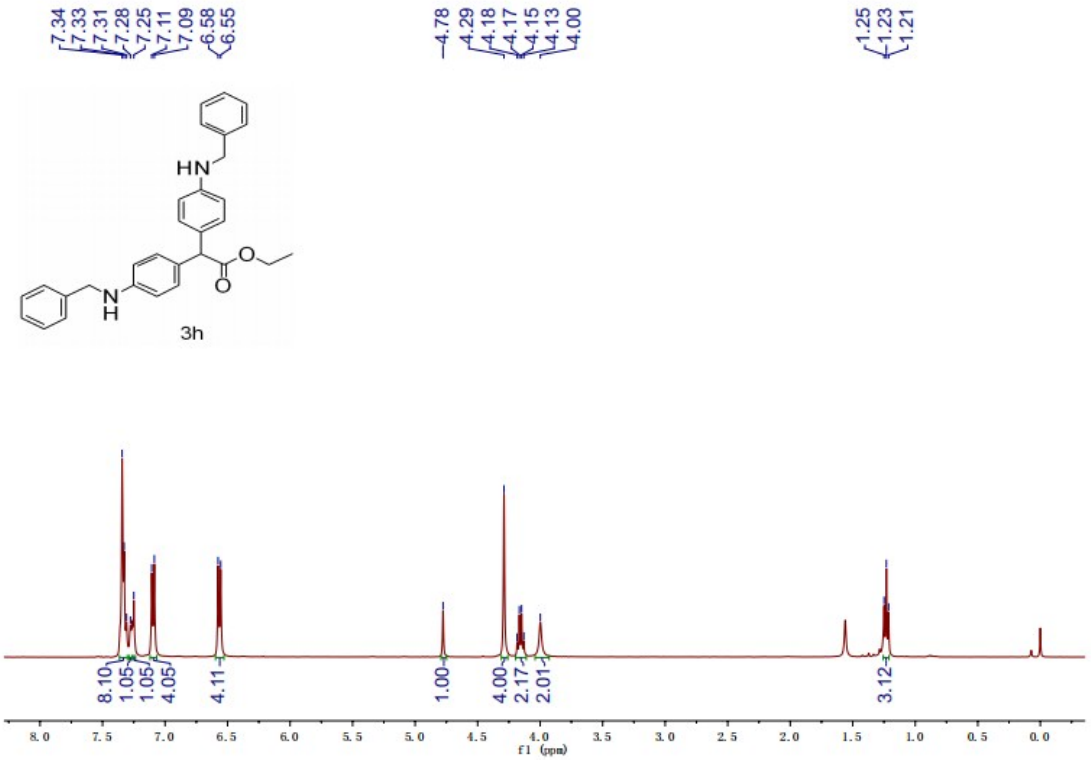
14.17
HN
O
O
HN
3g
f1 (ppm)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4.15
4.13
4.00
1.25
1.23
1.21
HN
O
O
N
H
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1.05
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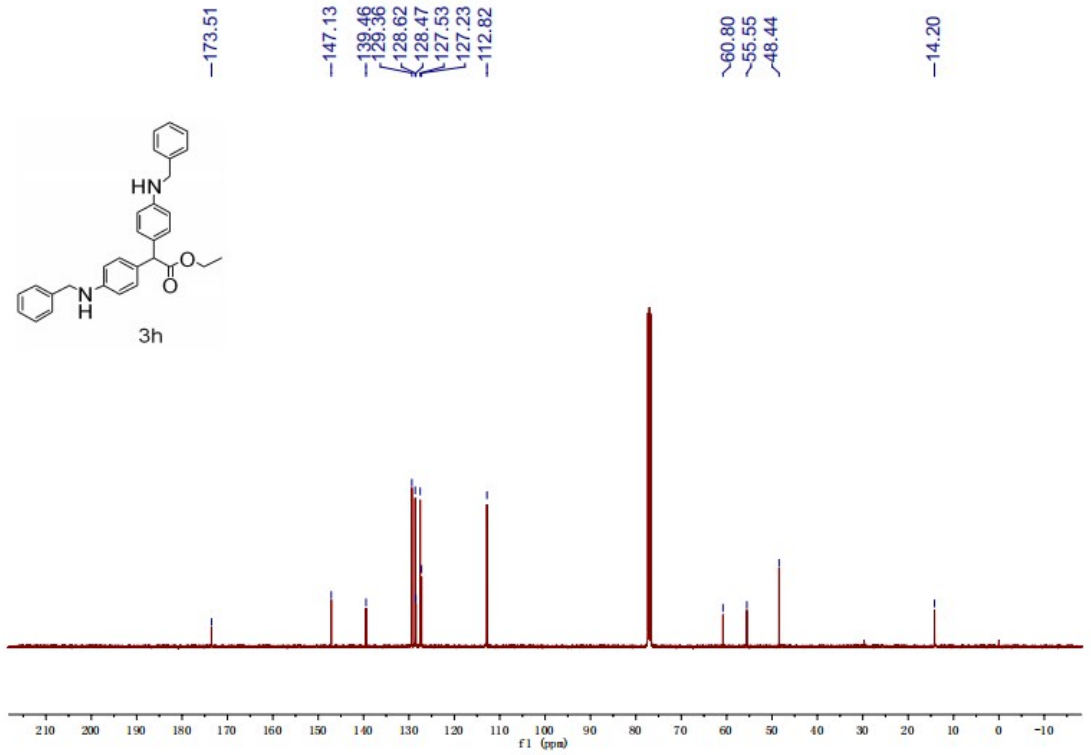
4.11
1.00
4.00
2.17
2.01
3.12
f1 (ppm)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O
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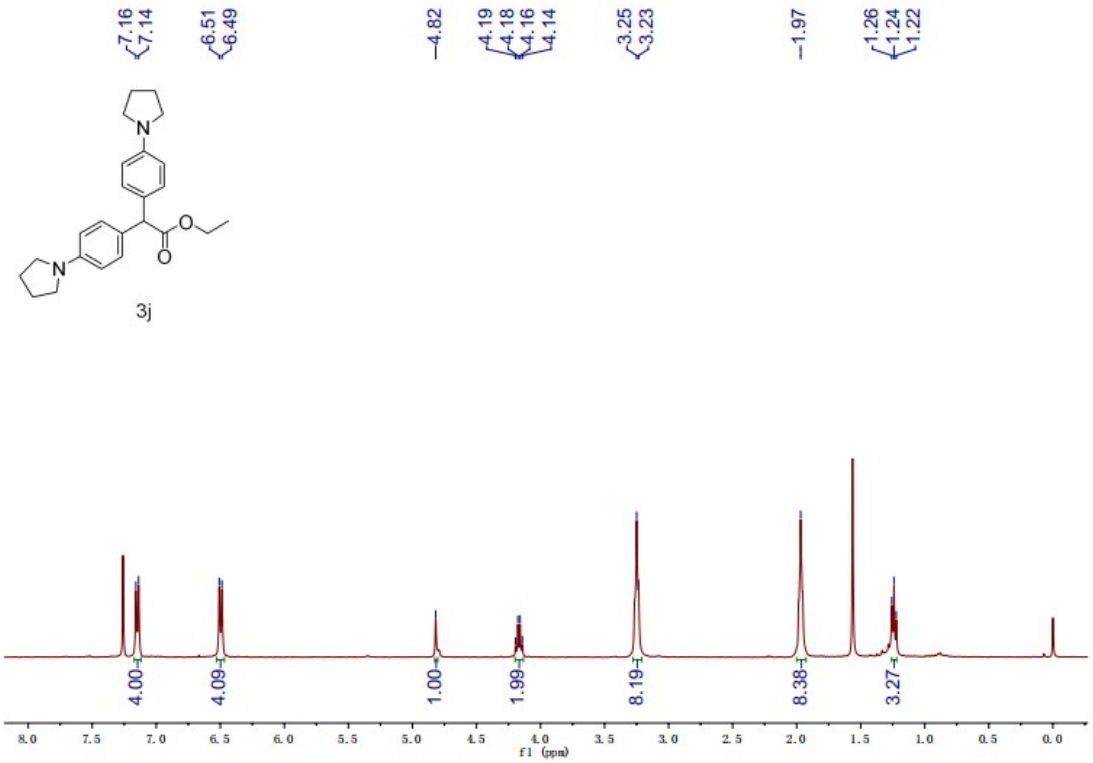
3h
f1 (ppm)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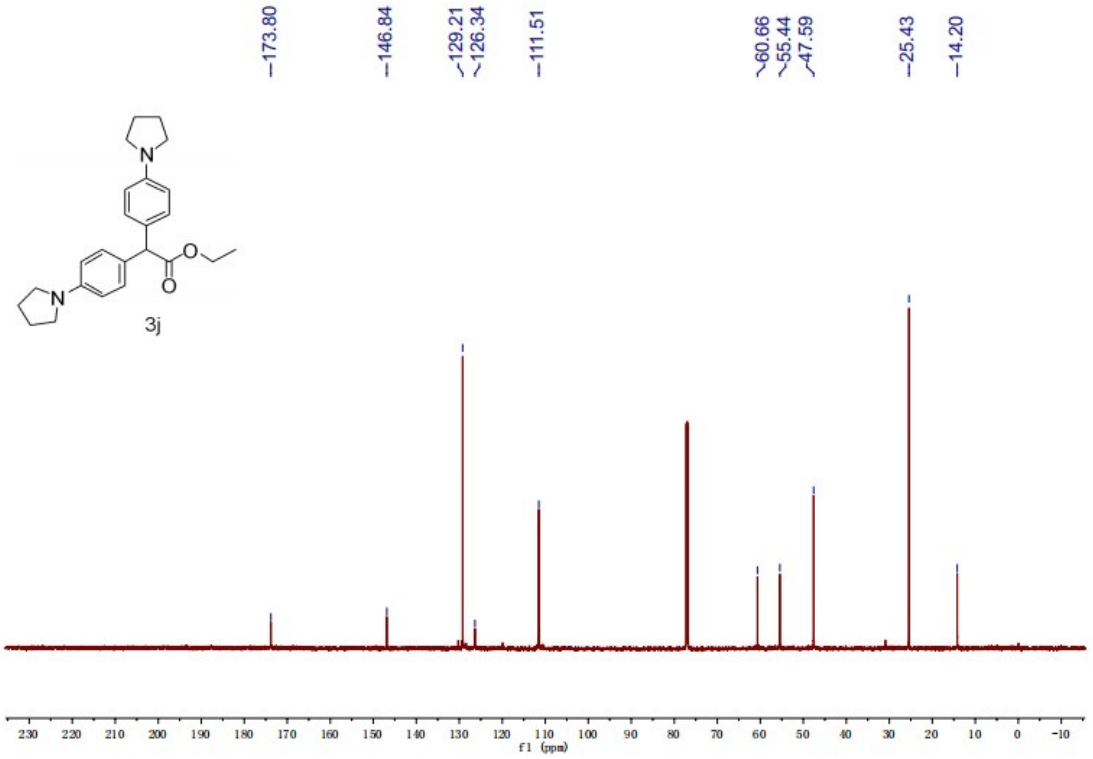
1.99
8.19
8.38
3.27
f1 (ppm)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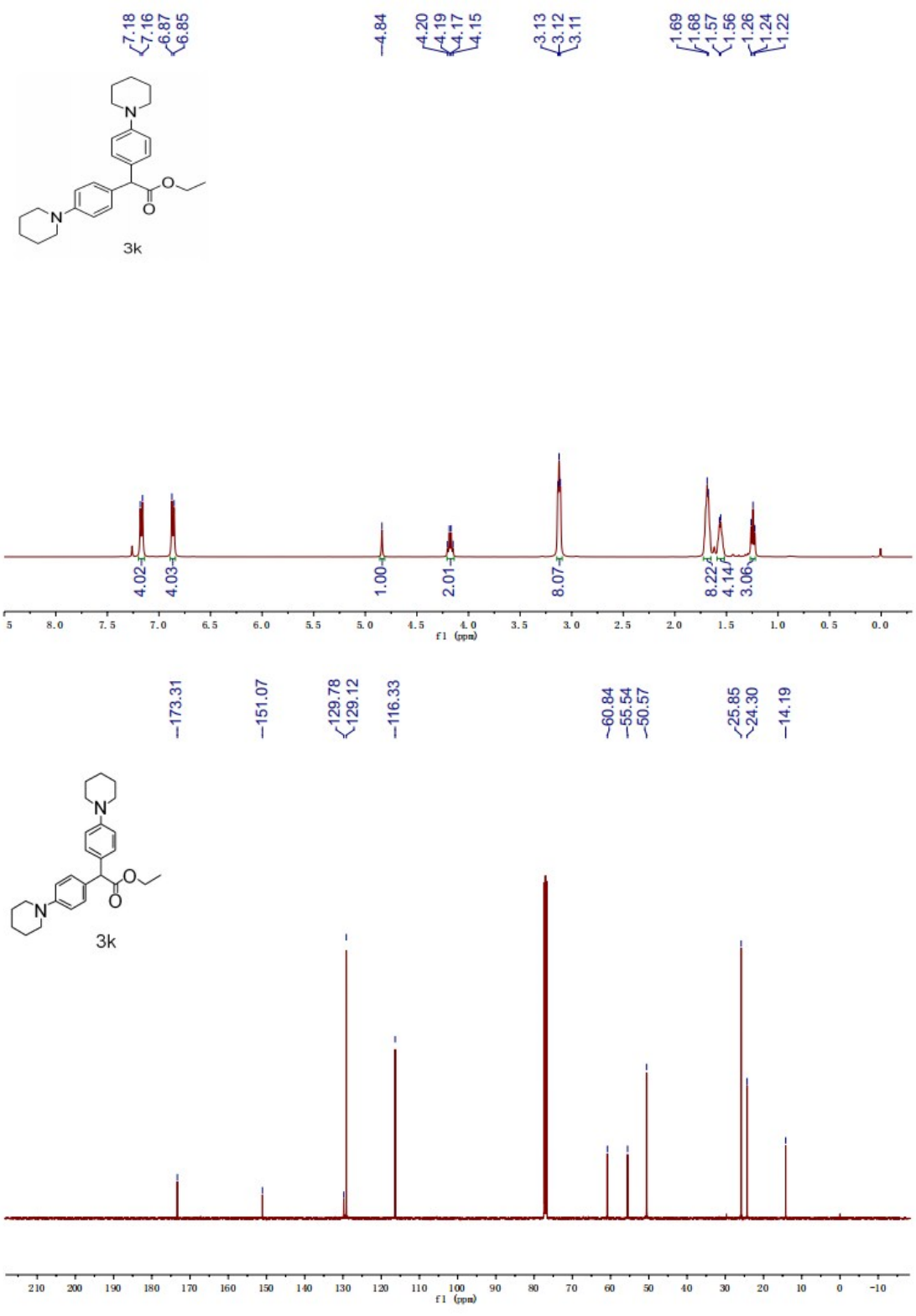
47.59
25.43
14.20
3j
f1 (ppm)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1.68
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1.56
1.26
1.24
1.22
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4.03
1.00
2.01
8.07
8.22
4.14
3.06
f1 (ppm)
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151.07
129.78
129.12
116.33
60.84
55.54
50.57
25.85
24.30
14.19
3k
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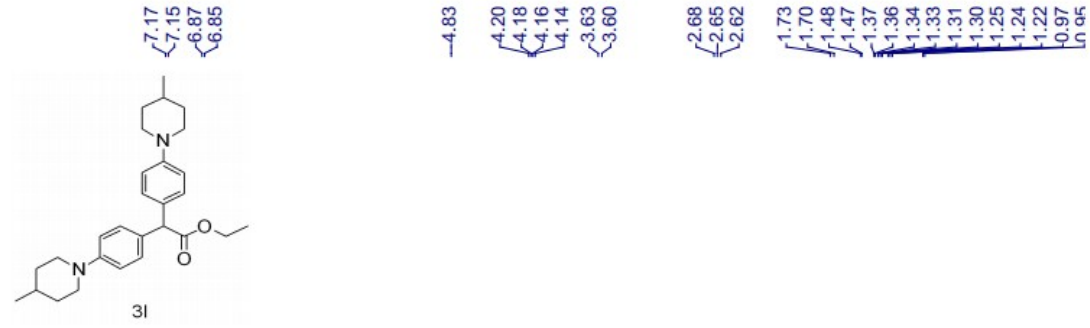
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4.16
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2.65
2.62
1.73
1.70
1.48
1.47
1.37
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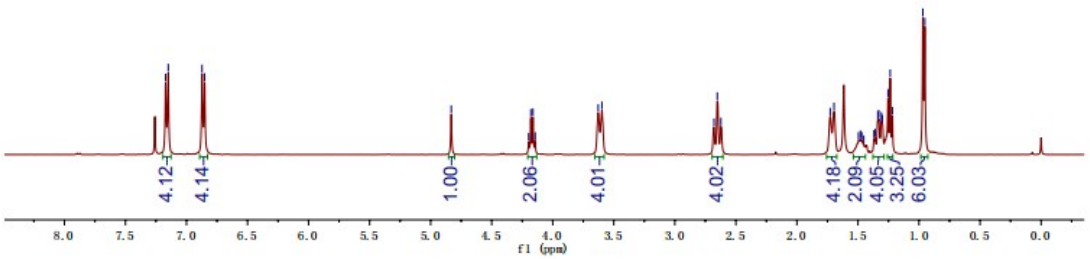
N
O
O
N
3l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4.02
4.18
2.09
4.05
3.25
6.03
8.0
7.5
7.0
6.5
6.0
5.5
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2.0
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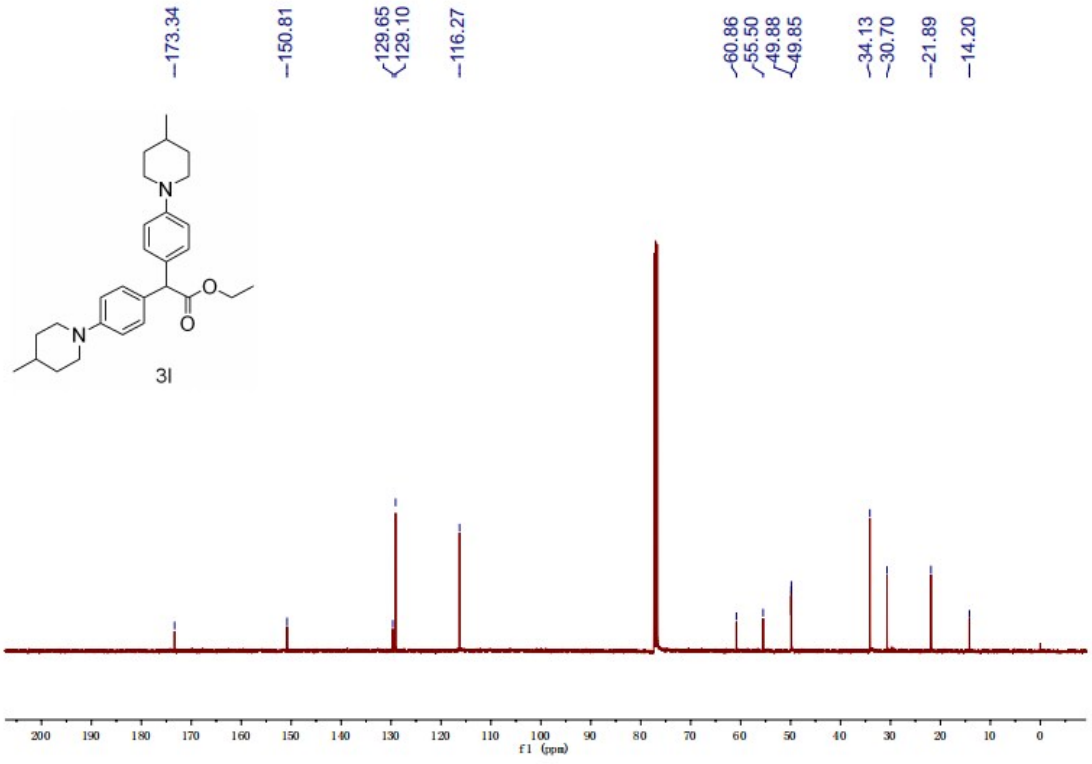
f1 (ppm)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14.20
N
O
O
N
3l
200
190
180
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50
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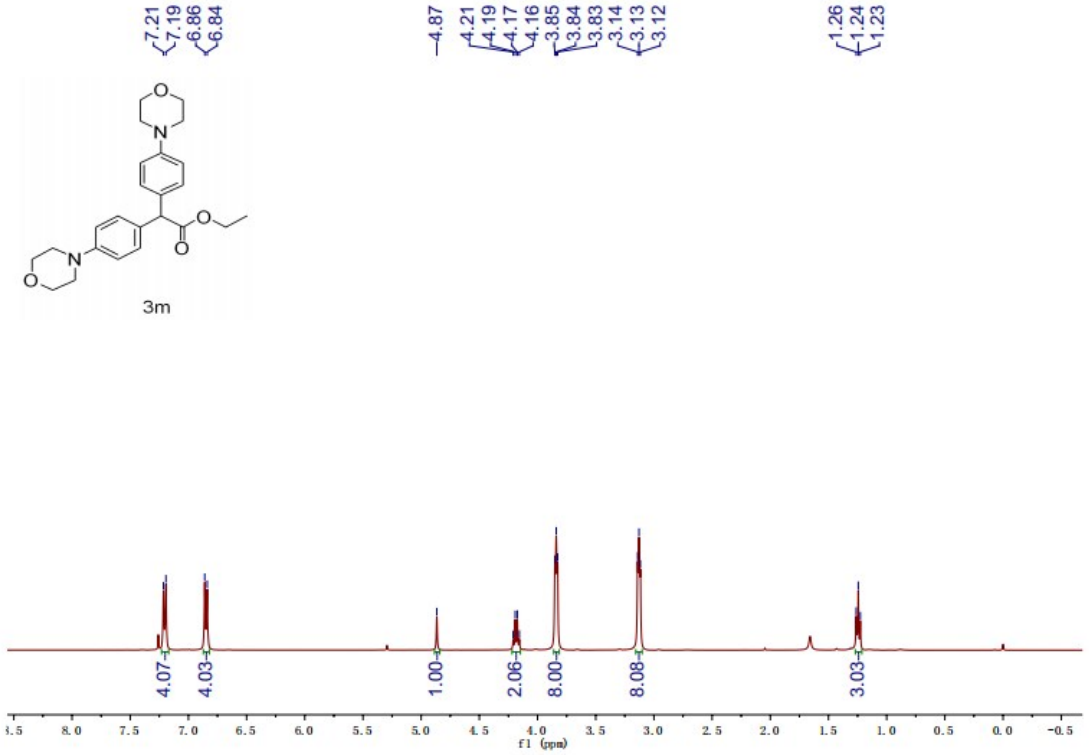
f1 (ppm)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3.13
3.12
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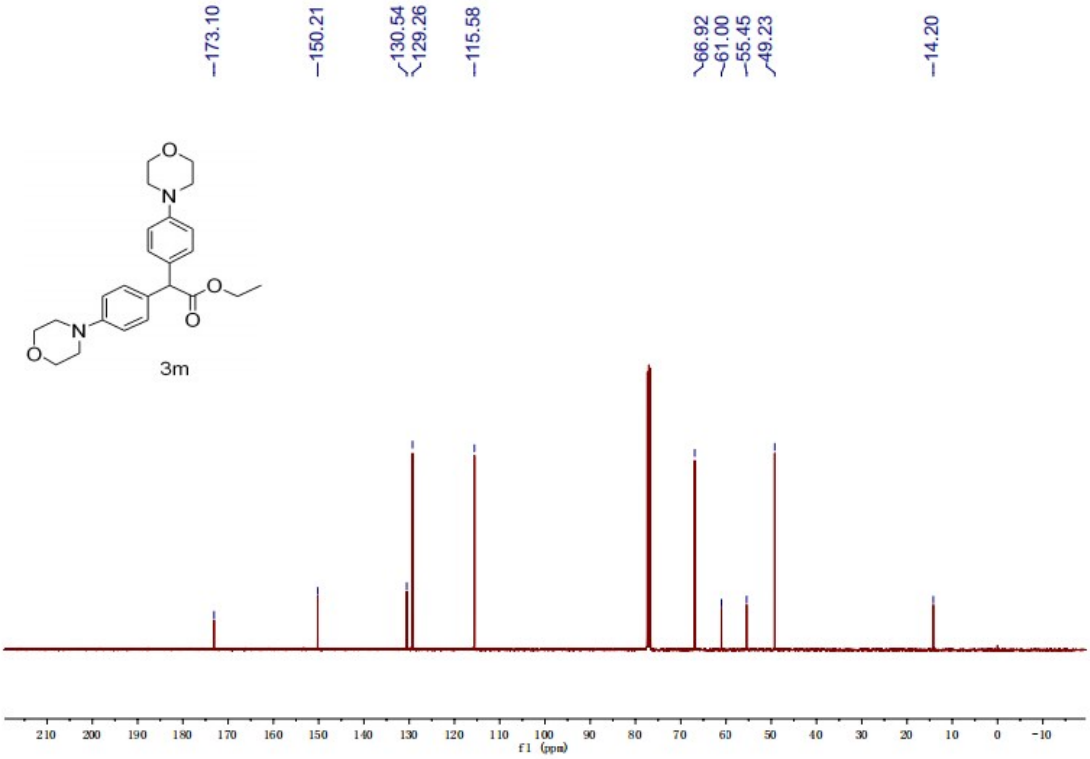
3.03
f1 (ppm)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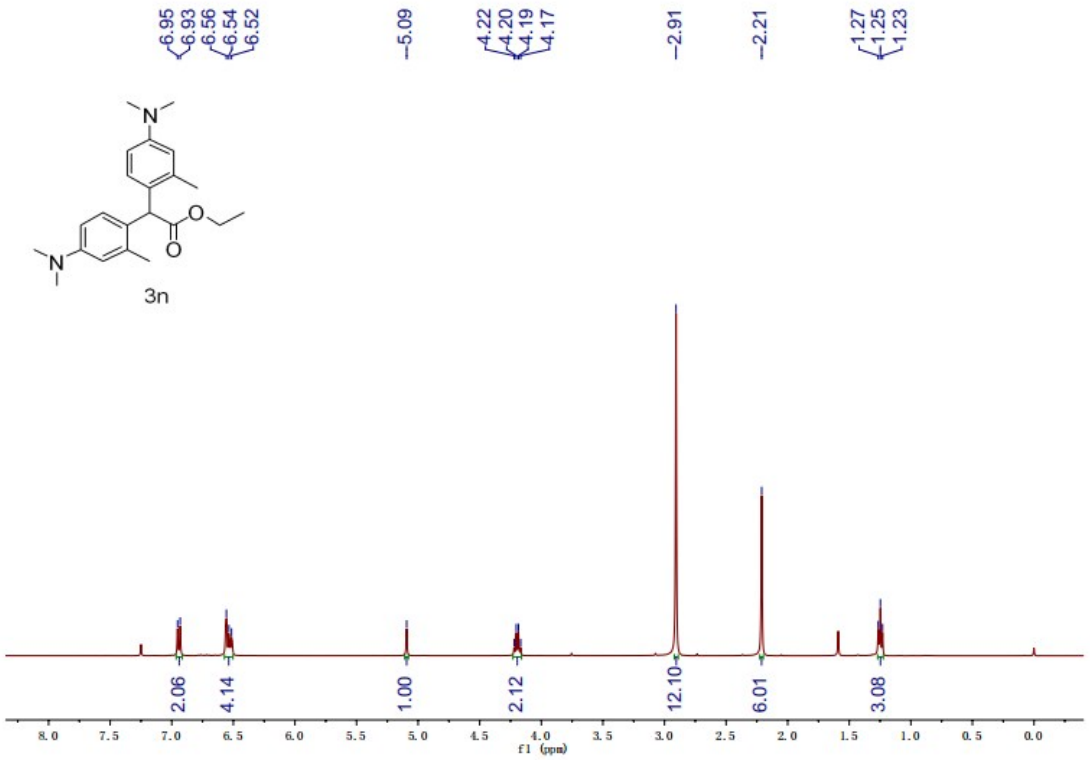
f1 (ppm)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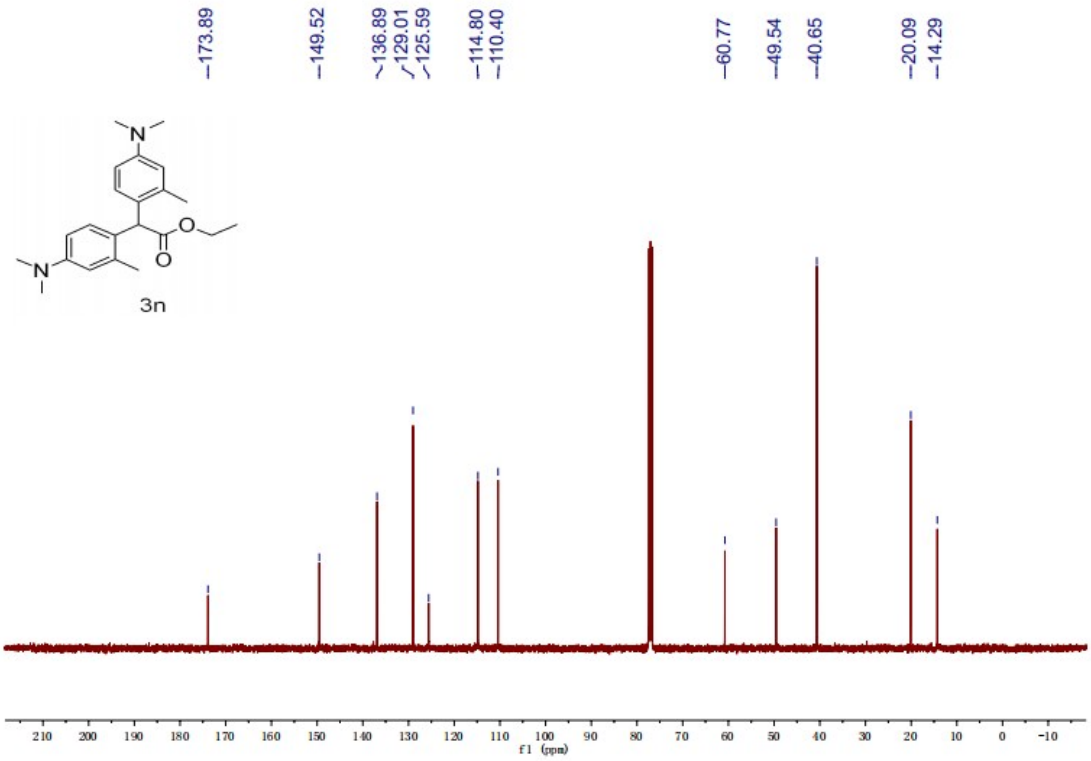
1.25
1.23
3n
2.06
4.14
1.00
2.12
12.10
6.01
3.08
f1 (ppm)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40.65
20.09
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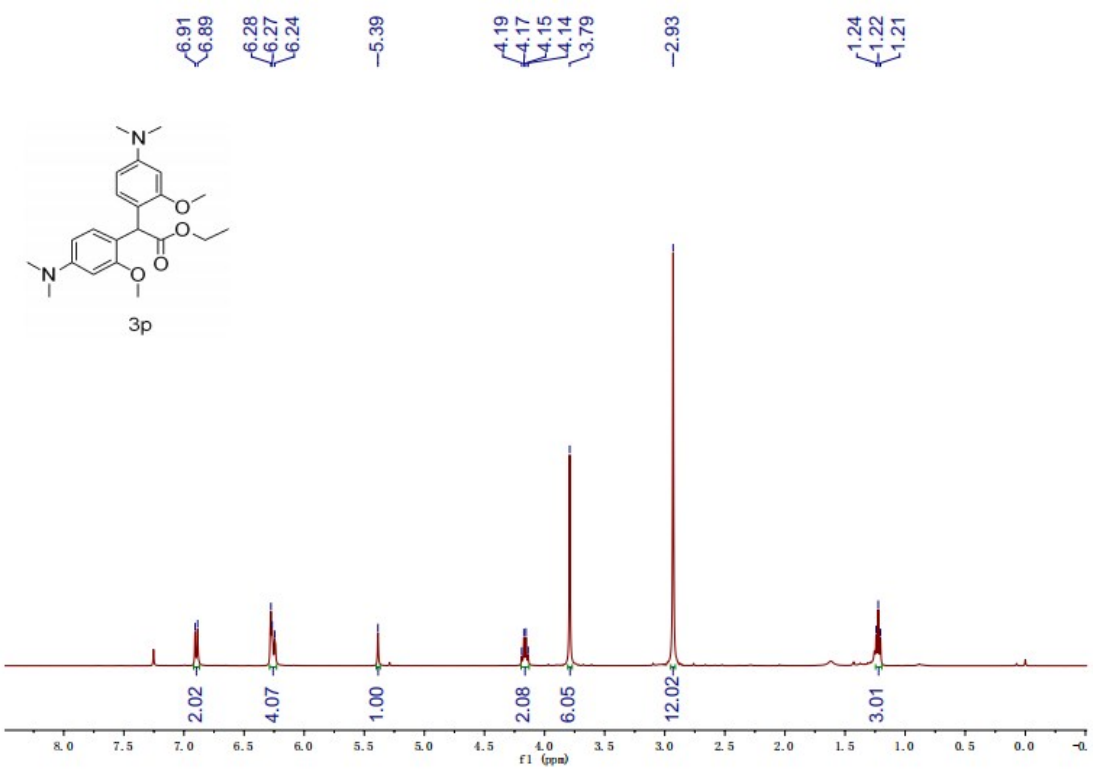
3n
f1 (ppm)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4.17
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4.14
3.79
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1.22
1.21
3p
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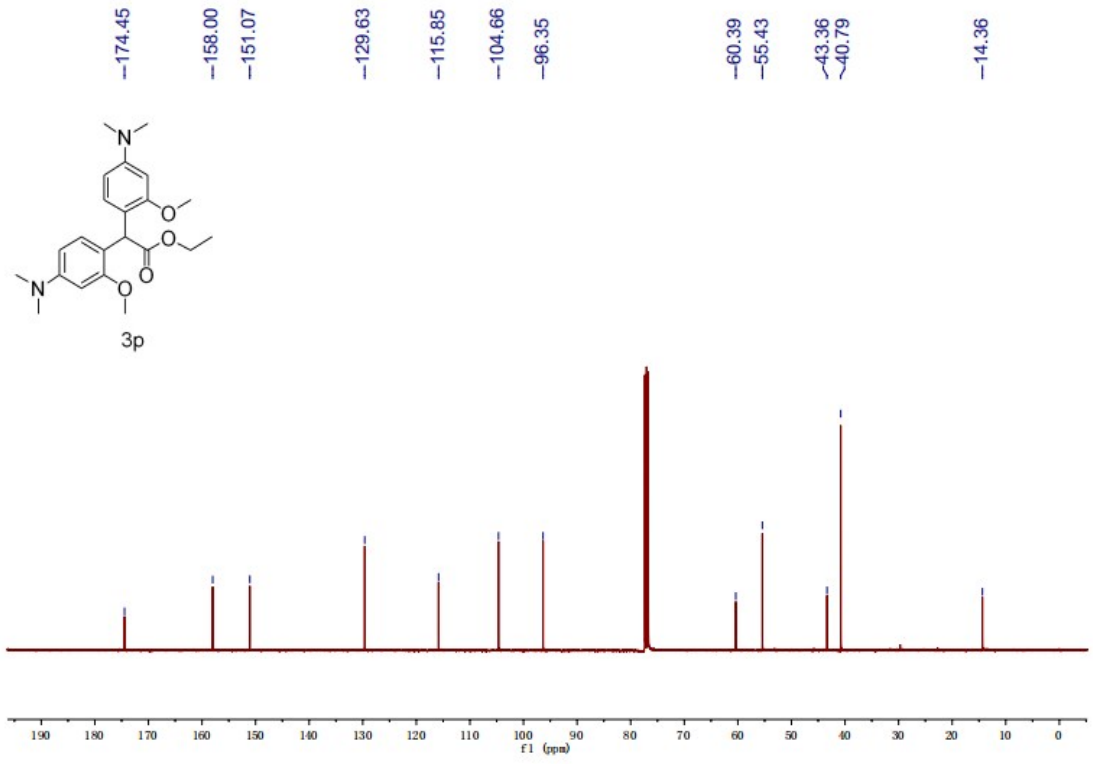
4.07
1.00
2.08
6.05
12.02
3.01
f1 (ppm)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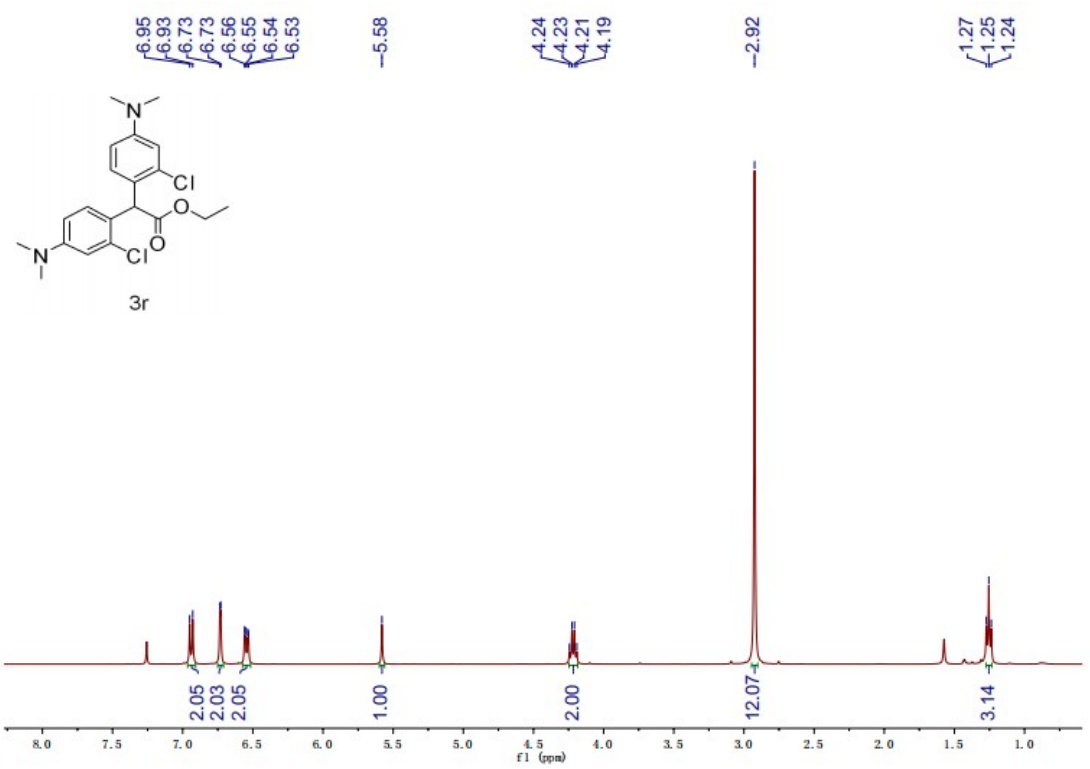
f1 (ppm)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6.53
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4.24
4.23
4.21
4.19
2.92
1.27
1.25
1.24
3r
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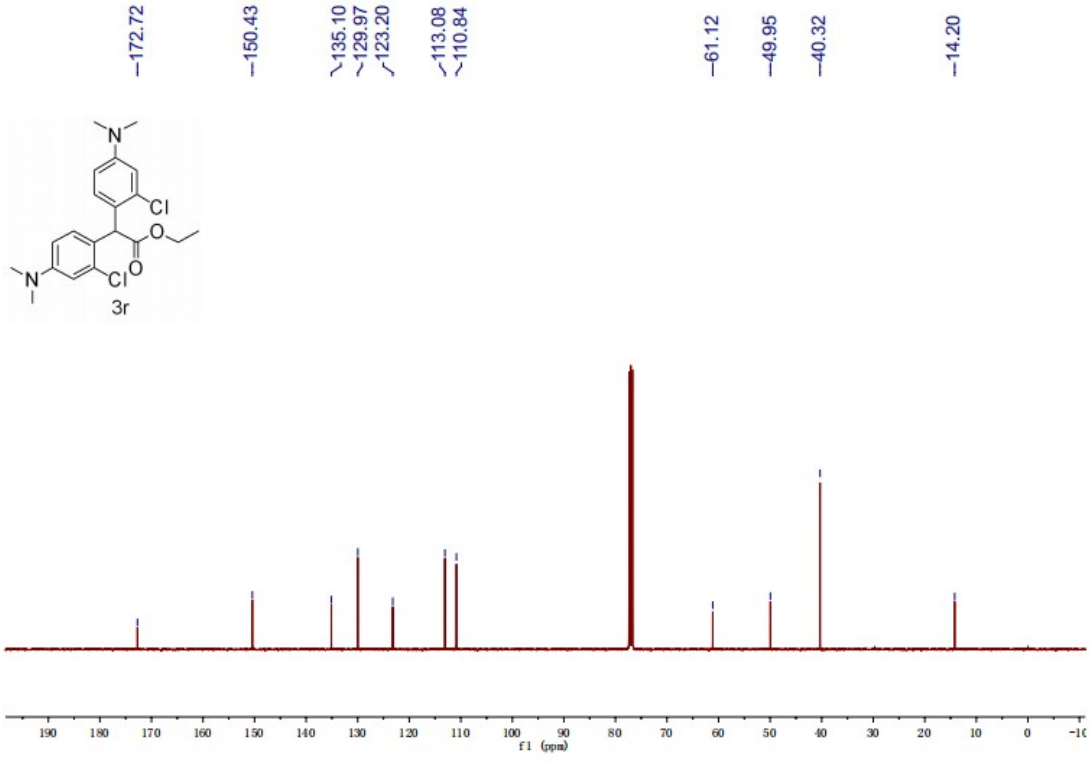
2.03
2.05
1.00
2.00
12.07
3.14
f1 (ppm)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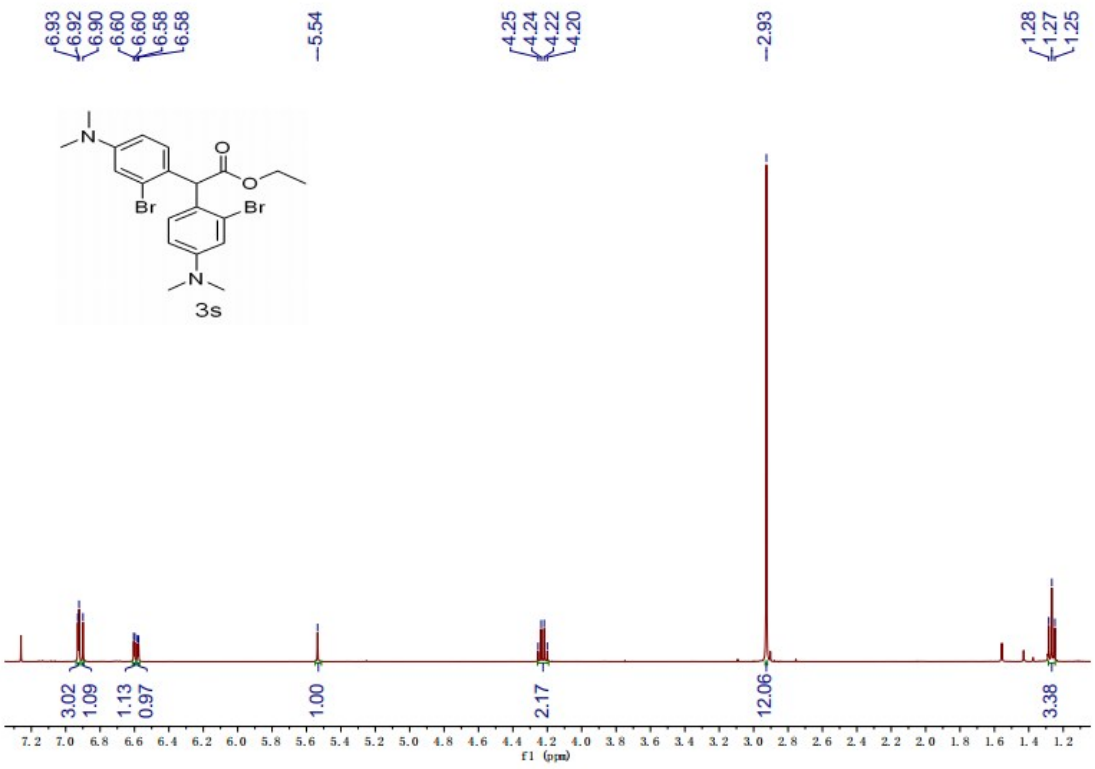
61.12
49.95
40.32
14.20
3r
f1 (ppm)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6.92
6.90
6.60
6.60
6.58
6.58
5.54
4.25
4.24
4.22
4.20
2.93
1.28
1.27
1.25
Br
Br
3s
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1.09
1.13
0.97
1.00
2.17
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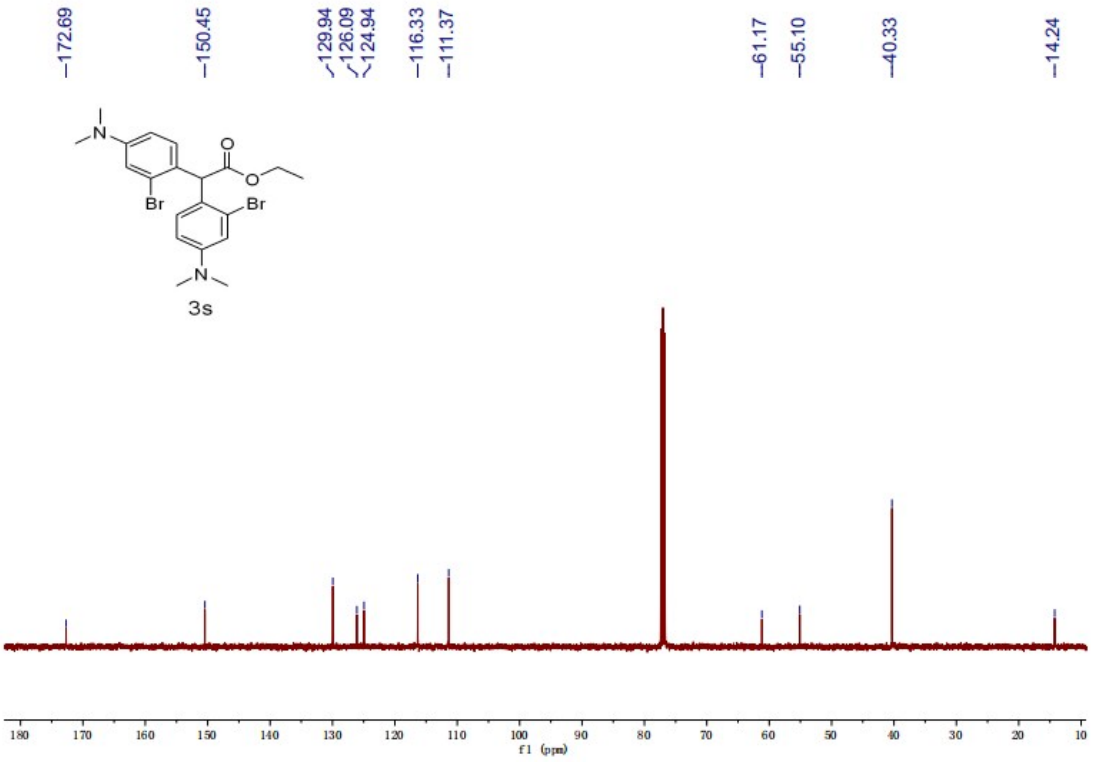
3.38
f1 (ppm)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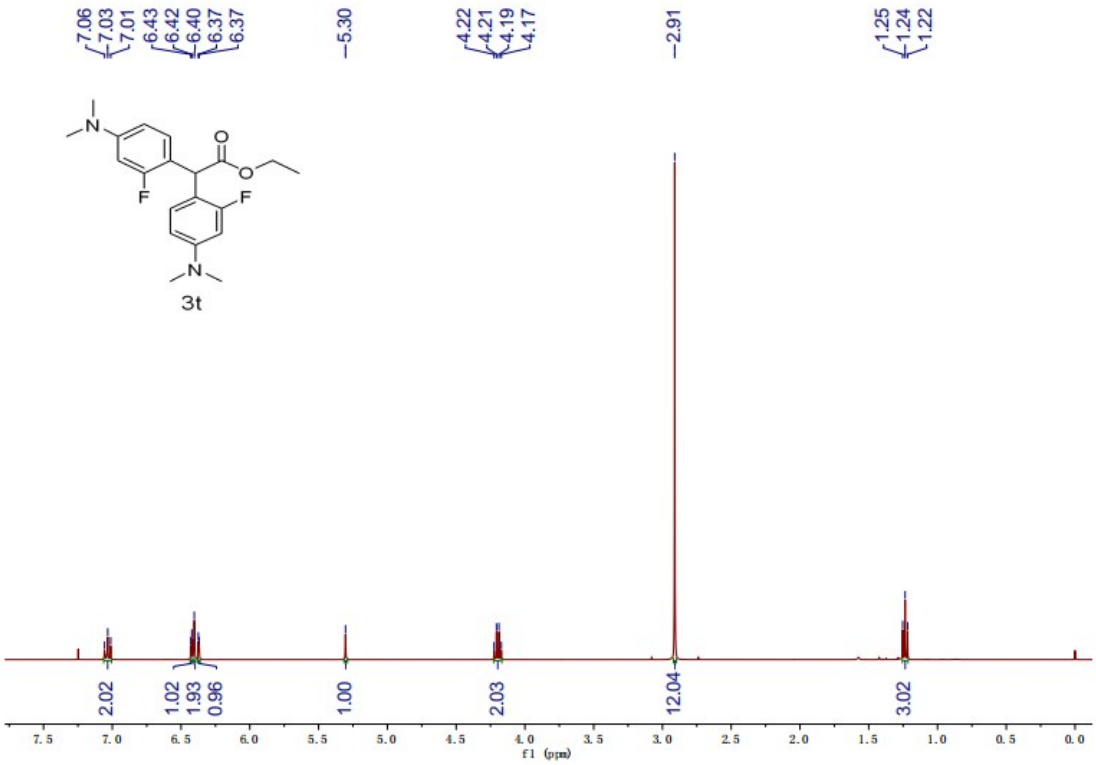
126.09
124.94
116.33
111.37
61.17
55.10
40.33
14.24
Br
Br
3s
f1 (ppm)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7.01
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6.42
6.40
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6.37
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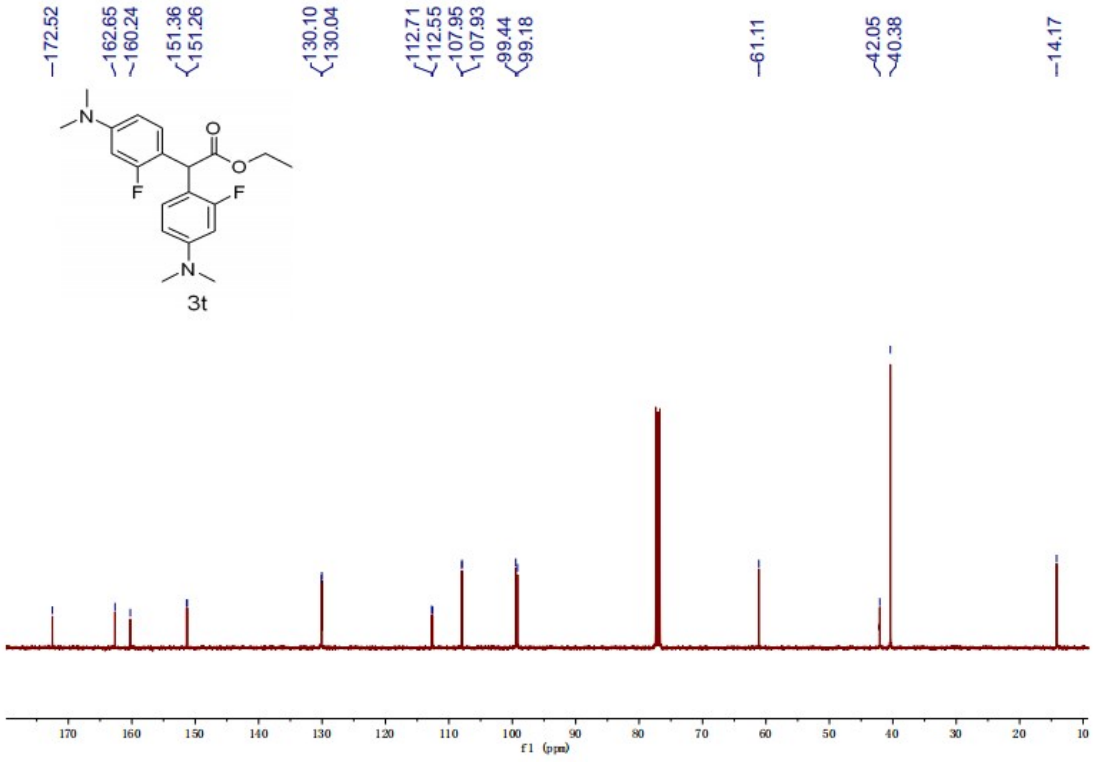
4.22
4.21
4.19
4.17
2.91
1.25
1.24
1.22
3t
2.02
1.02
1.93
0.96
1.00
2.03
12.04
3.02
f1 (ppm)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151.26
130.10
130.04
112.71
112.55
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107.93
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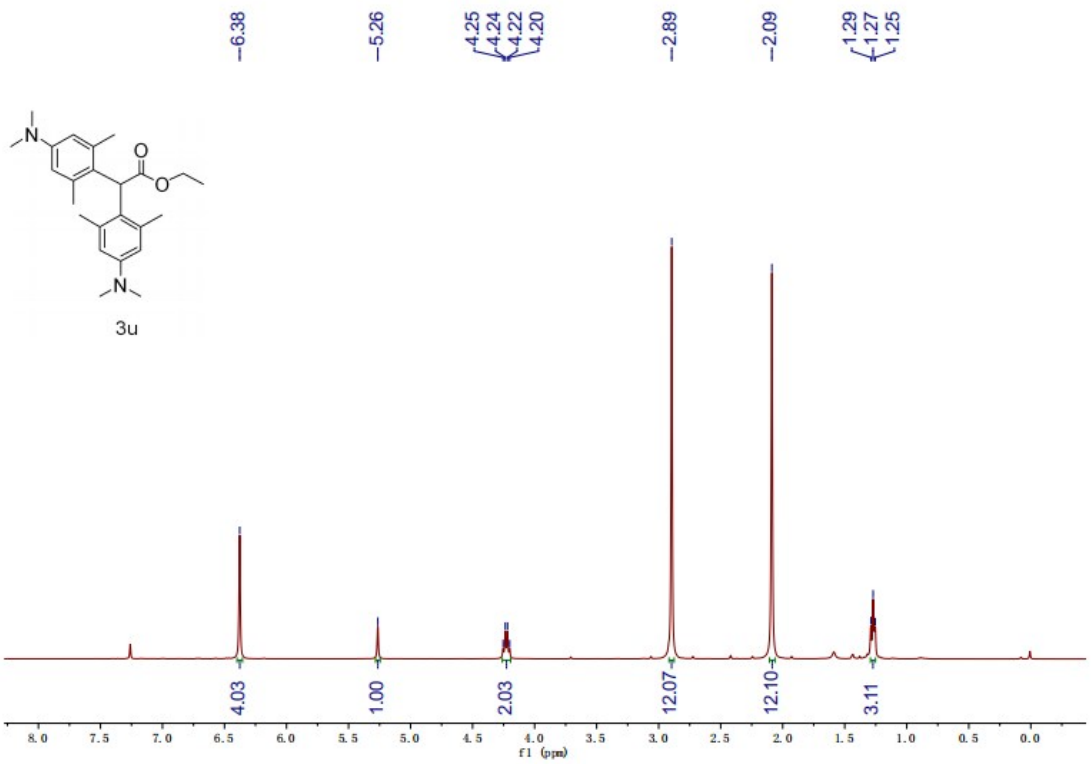
42.05
40.38
14.17
3t
f1 (ppm)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4.24
4.22
4.20
2.89
2.09
1.29
1.27
1.25
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4.03
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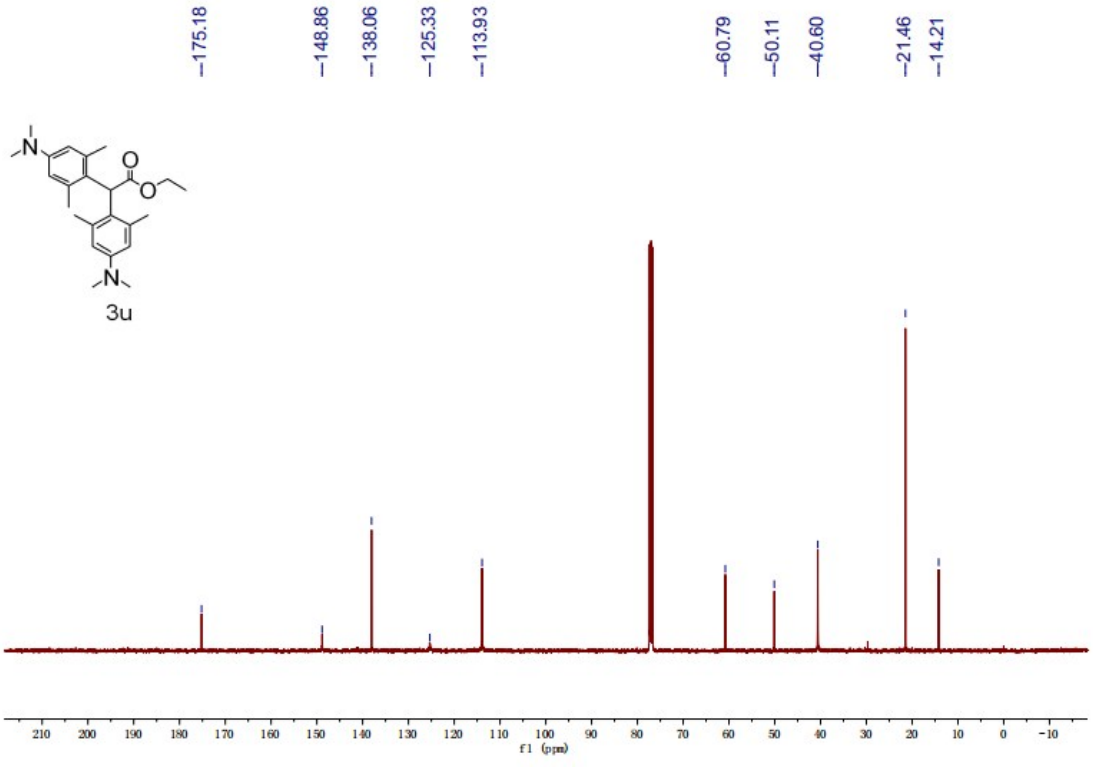
2.03
12.07
12.10
3.11
f1 (ppm)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21.46
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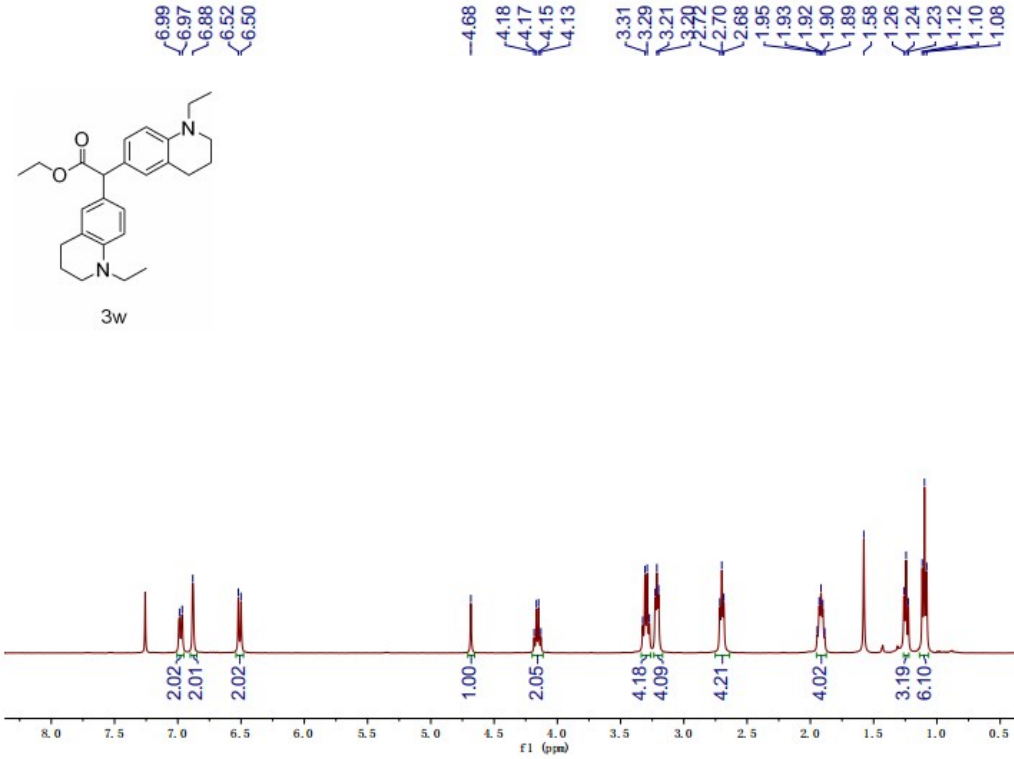
f1 (ppm)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4.17
4.15
4.13
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3.29
3.21
2.72
2.70
2.68
1.95
1.93
1.92
1.90
1.89
1.58
1.26
1.24
1.23
1.12
1.10
1.08
3w
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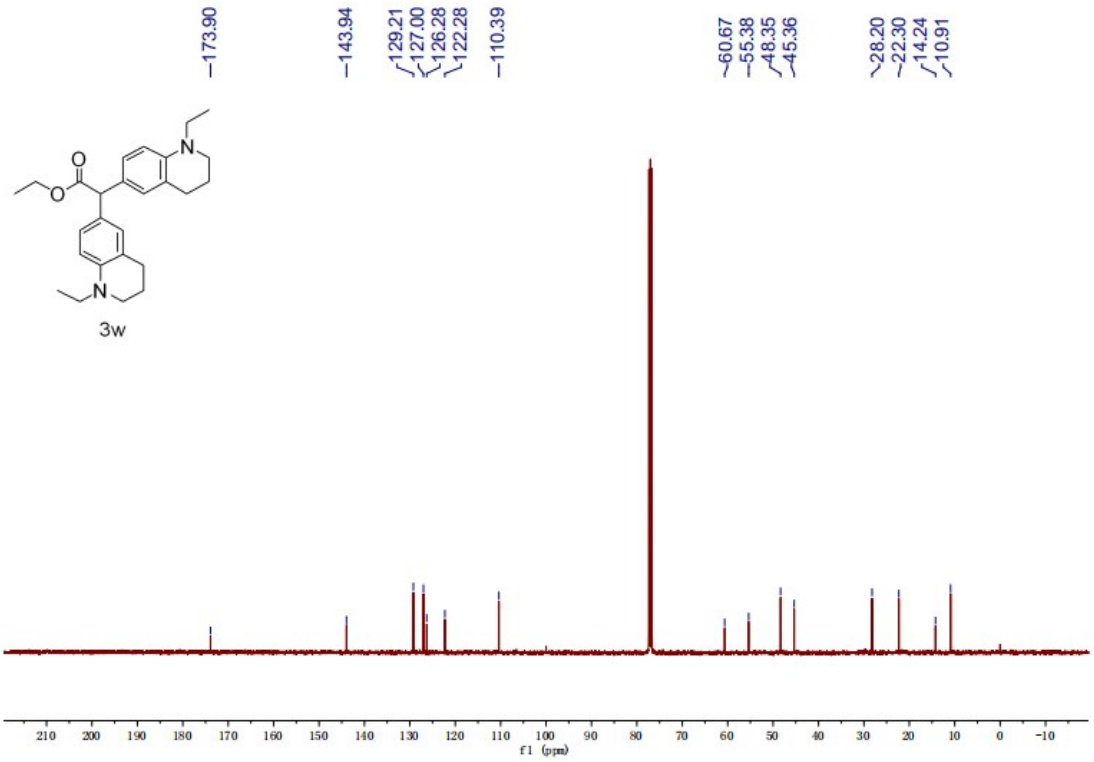
2.01
2.02
1.00
2.05
4.18
4.09
4.21
4.02
3.19
6.10
f1 (ppm)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22.30
14.24
10.91
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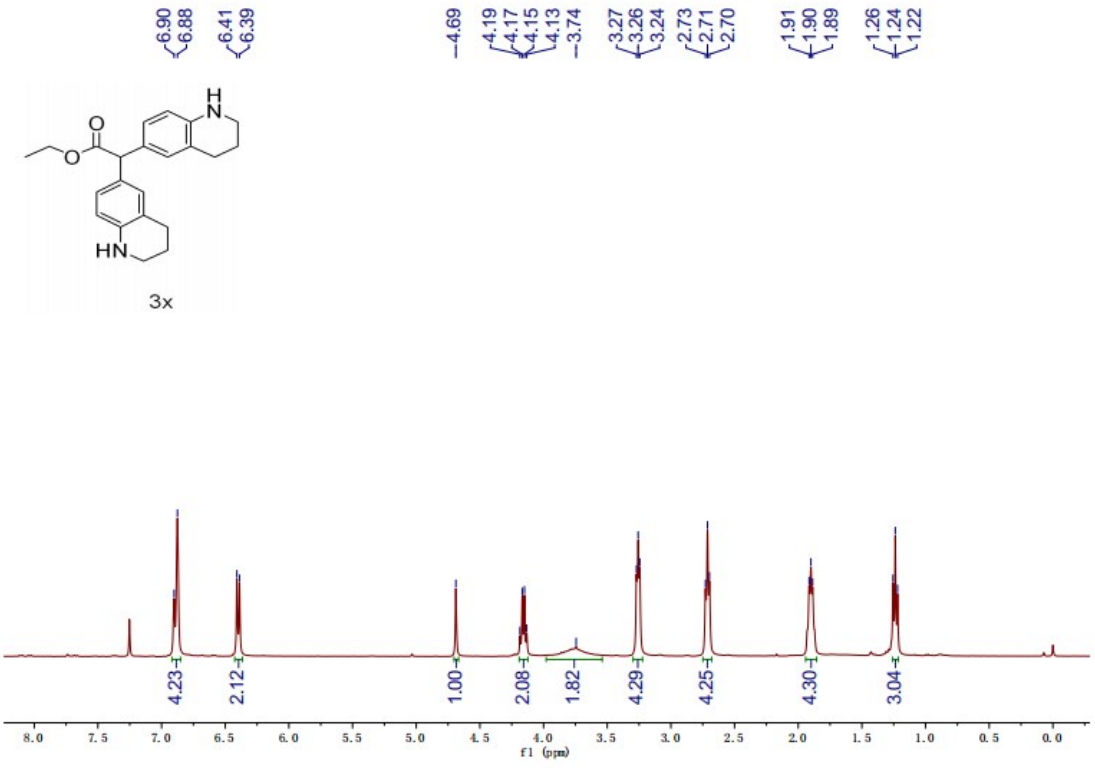
f1 (ppm)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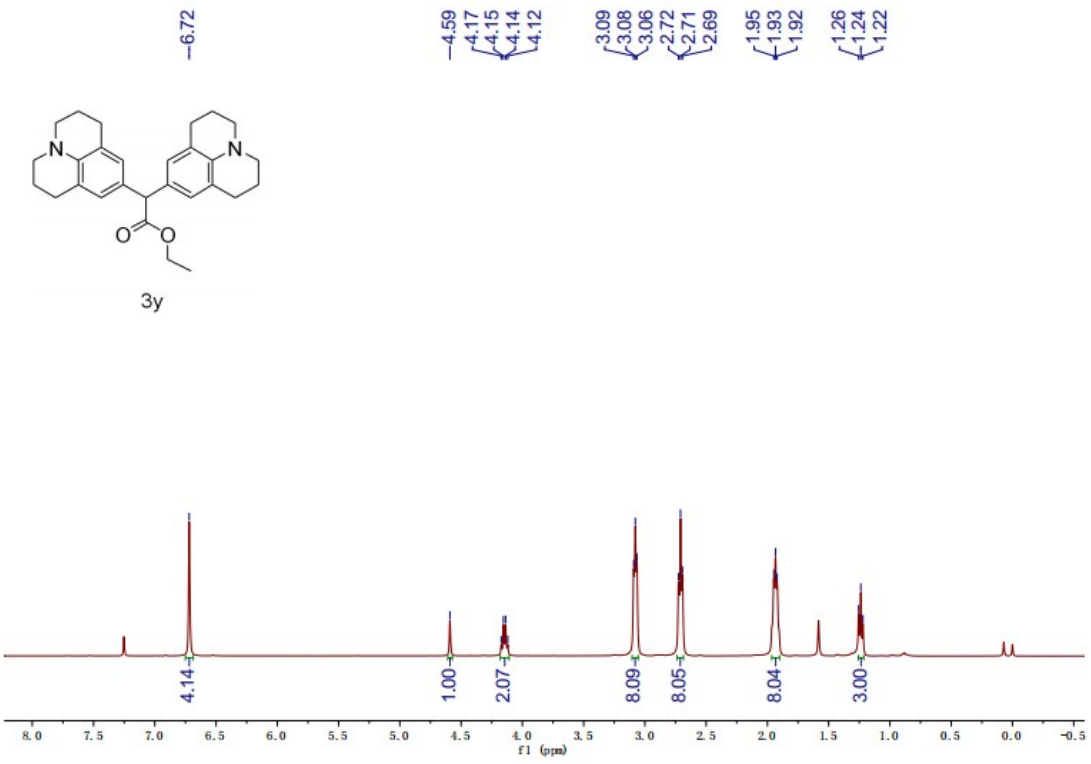
3.26
3.24
2.73
2.71
2.70
1.91
1.90
1.89
1.26
1.24
1.22
3x
4.23
2.12
1.00
2.08
1.82
4.29
4.25
4.30
3.04
f1 (ppm)
173.71
143.50
129.55
128.09
126.81
121.50
114.31
60.73
55.68
42.02
26.98
22.15
14.20
3x
f1 (ppm)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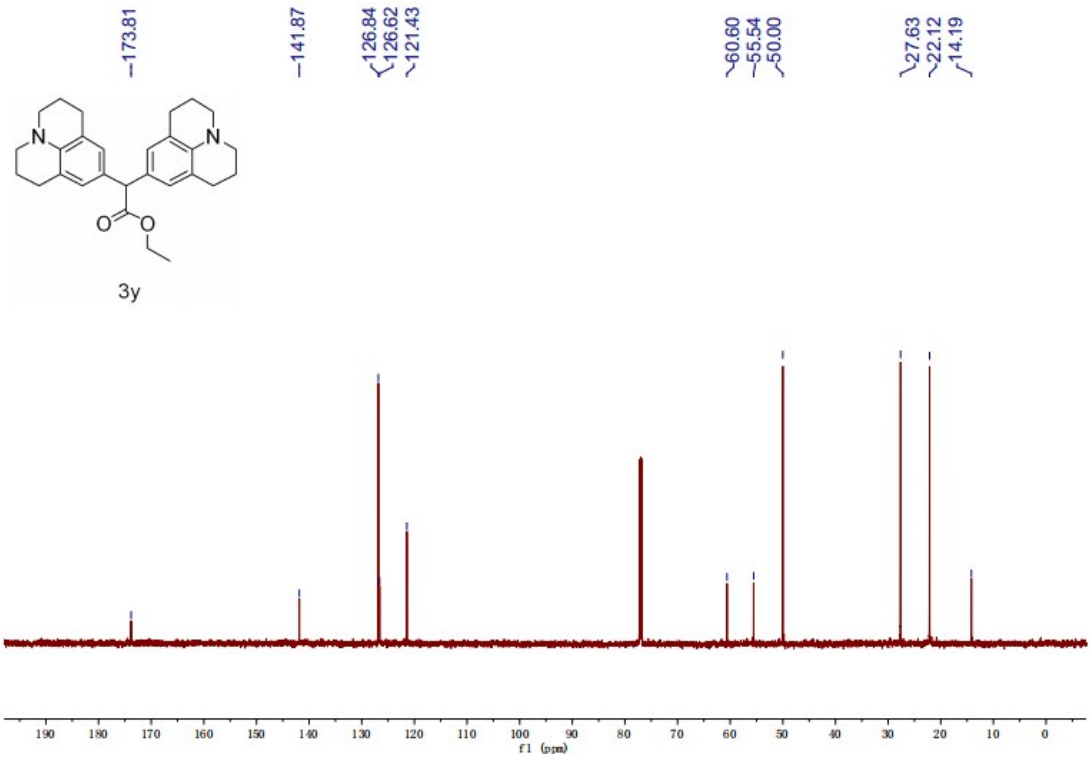
4.12
3.09
3.08
3.06
2.72
2.71
2.69
1.95
1.93
1.92
1.26
1.24
1.22
3y
4.14
1.00
2.07
8.09
8.05
8.04
3.00
f1 (ppm)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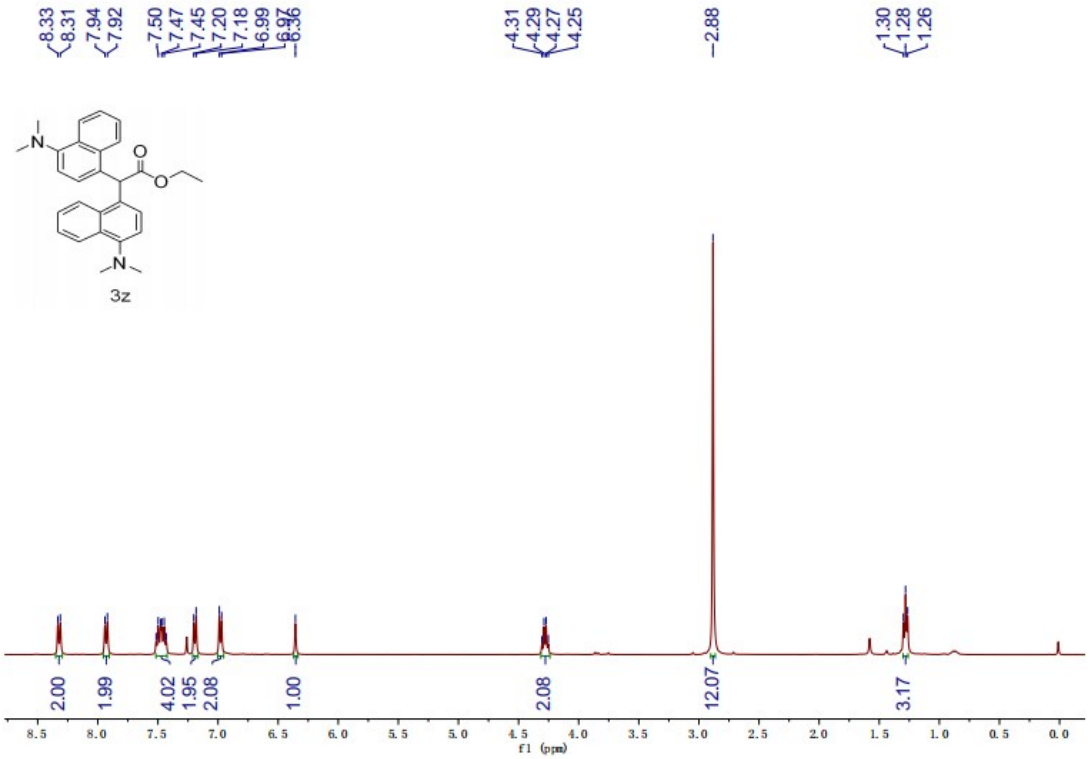
27.63
22.12
14.19
3y
f1 (ppm)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7.20
7.18
6.99
6.97
6.96
4.31
4.29
4.27
4.25
2.88
1.30
1.28
1.26
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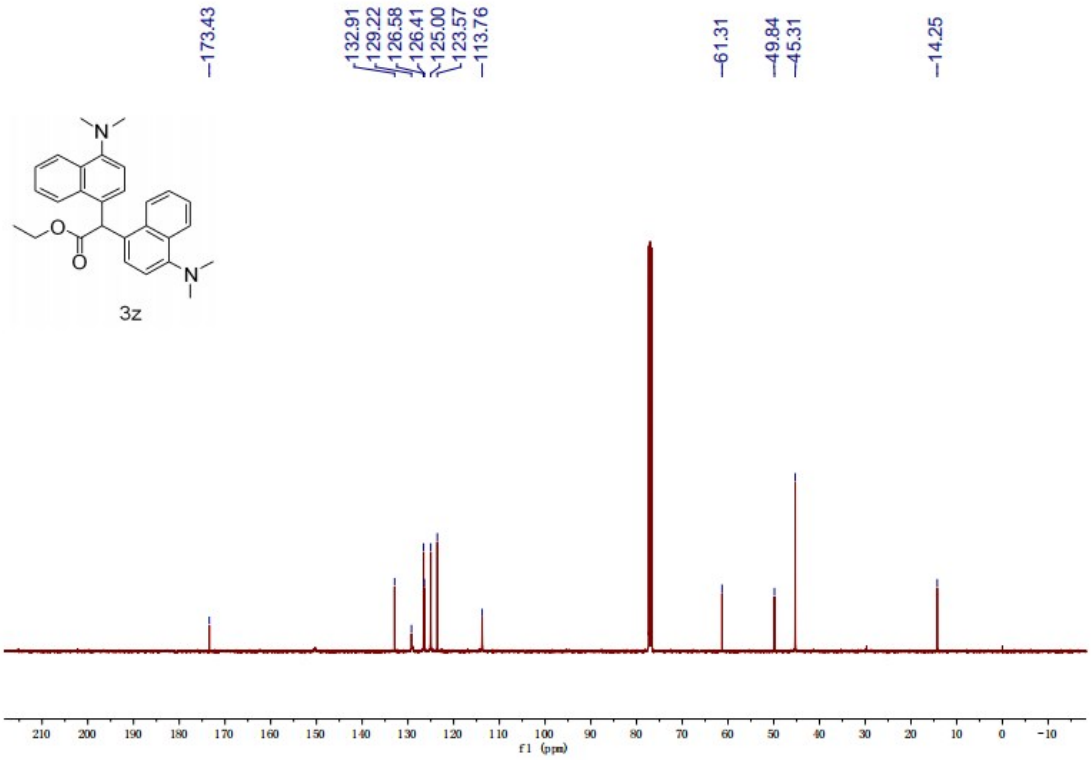
1.99
4.02
1.95
2.08
1.00
2.08
12.07
3.17
f1 (ppm)


173.43
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125.00
123.57
113.76
61.31
49.84
45.31
14.25
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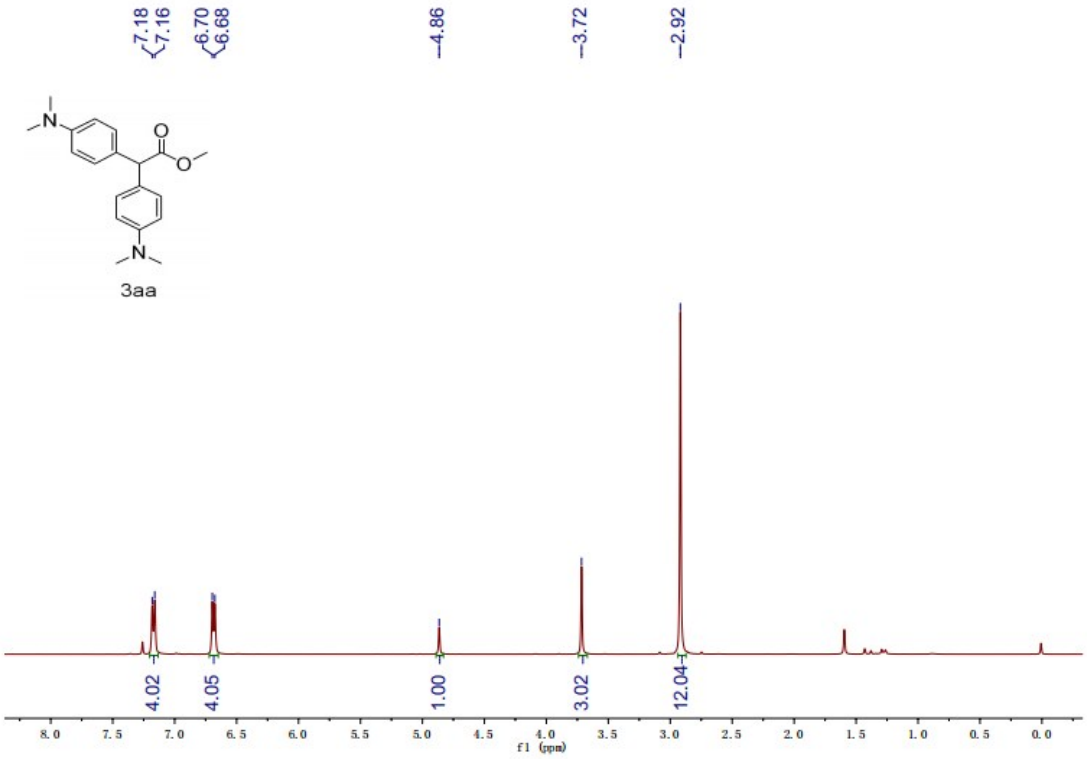
f1 (ppm)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4.02
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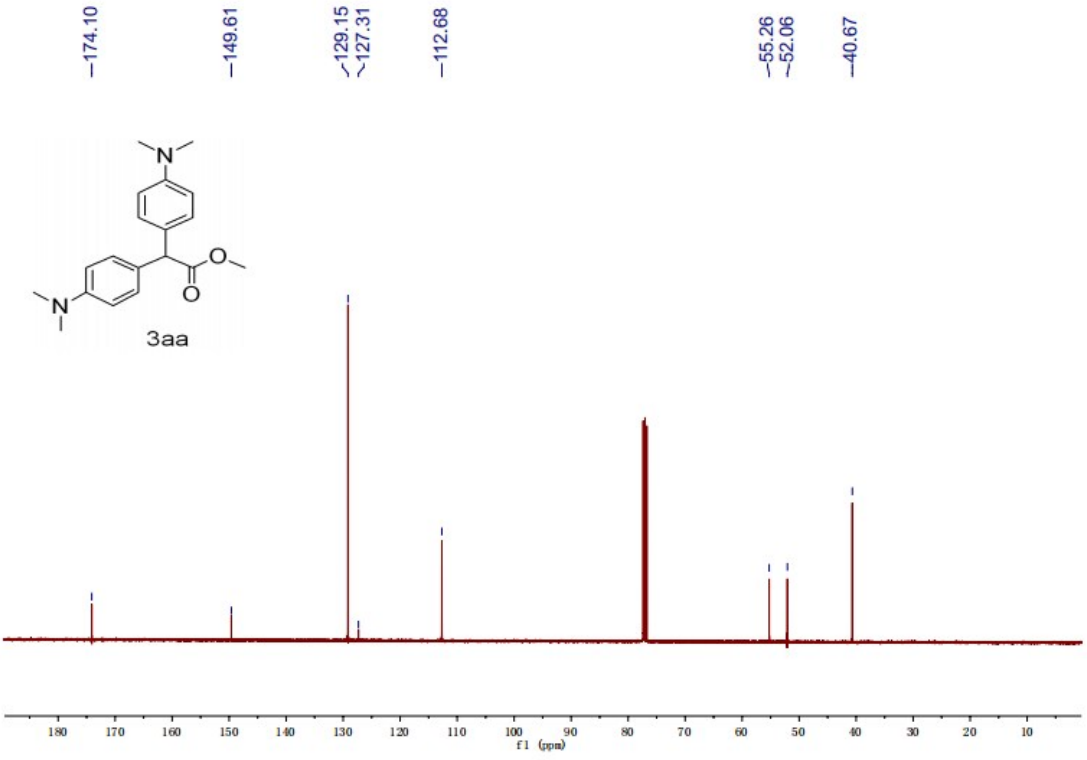
1.00
3.02
12.04
f1 (ppm)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127.31
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55.26
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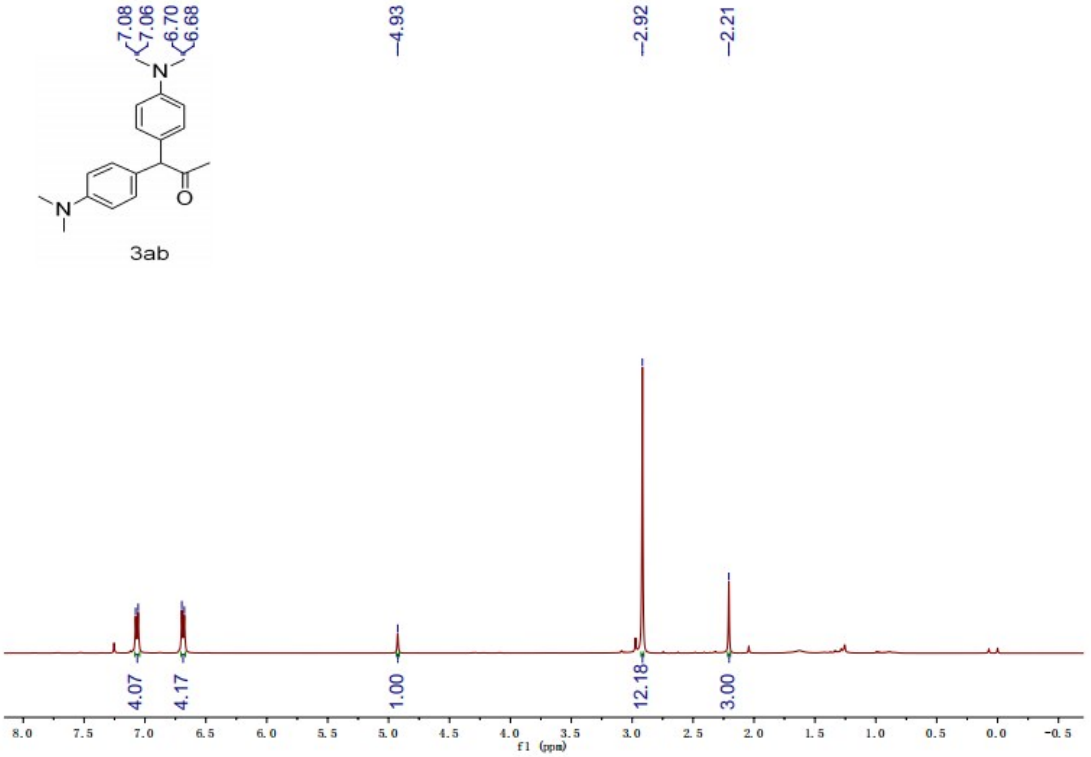
f1 (ppm)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4.93
2.92
2.21
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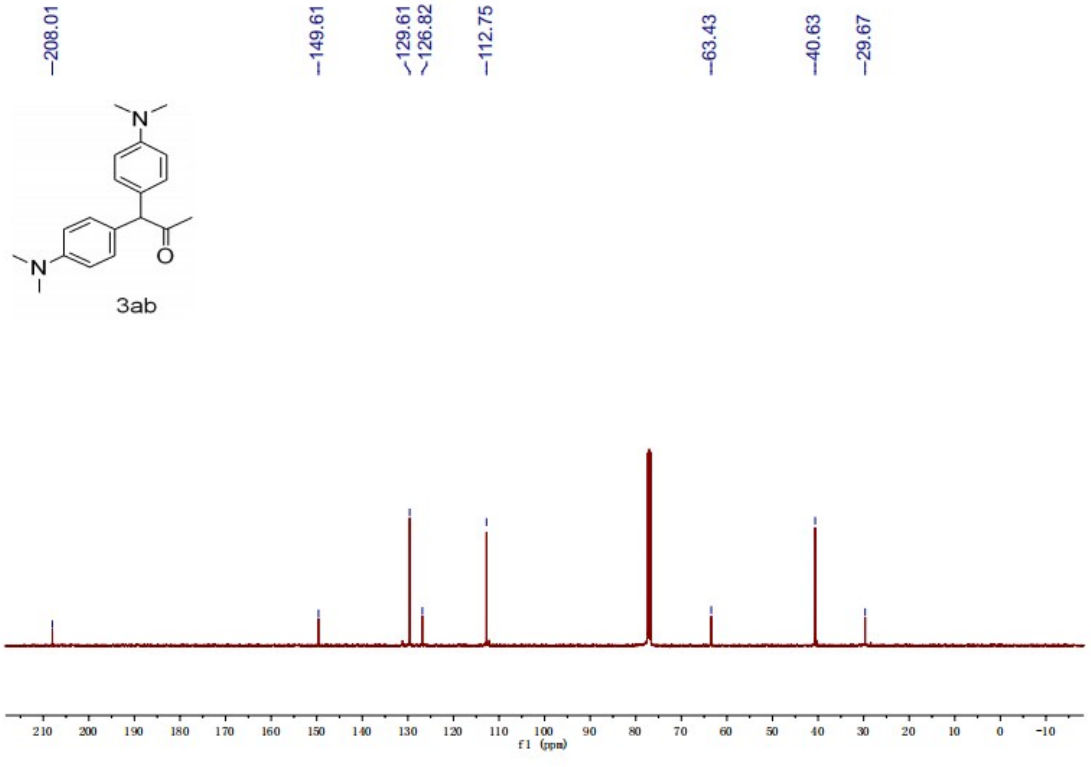
4.07
4.17
1.00
12.18
3.00
f1 (ppm)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40.63
29.67
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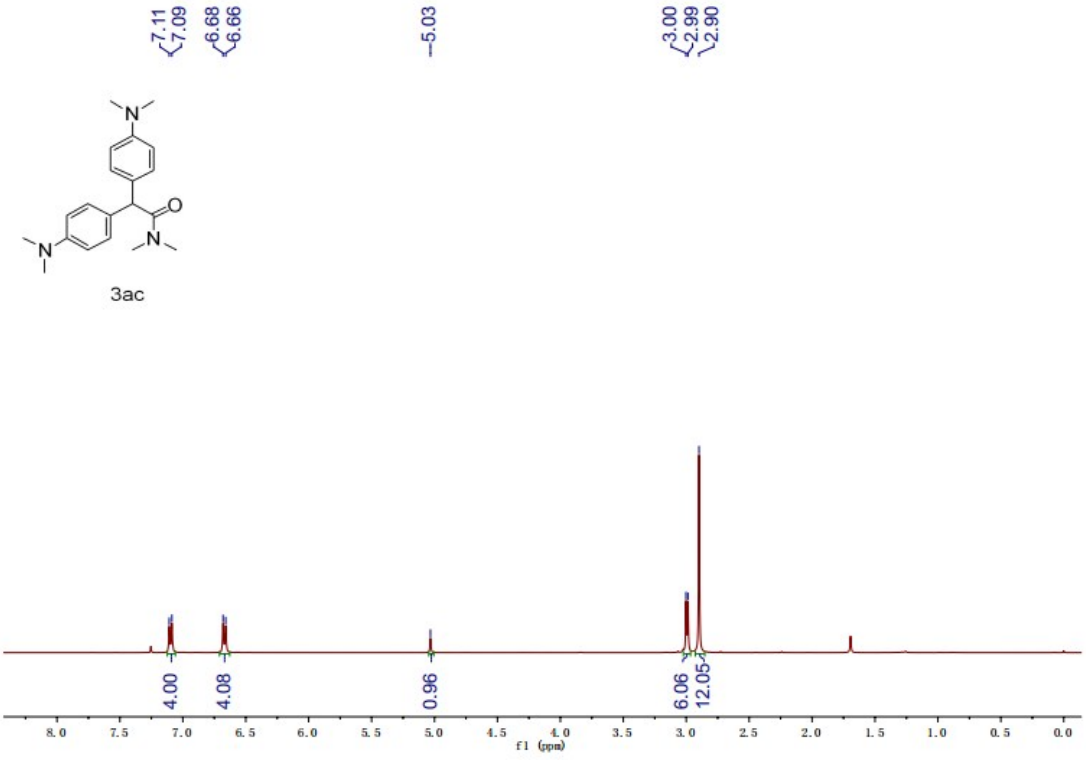
f1 (ppm)


7.11
7.09
6.68
6.66
5.03
3.00
2.99
2.90
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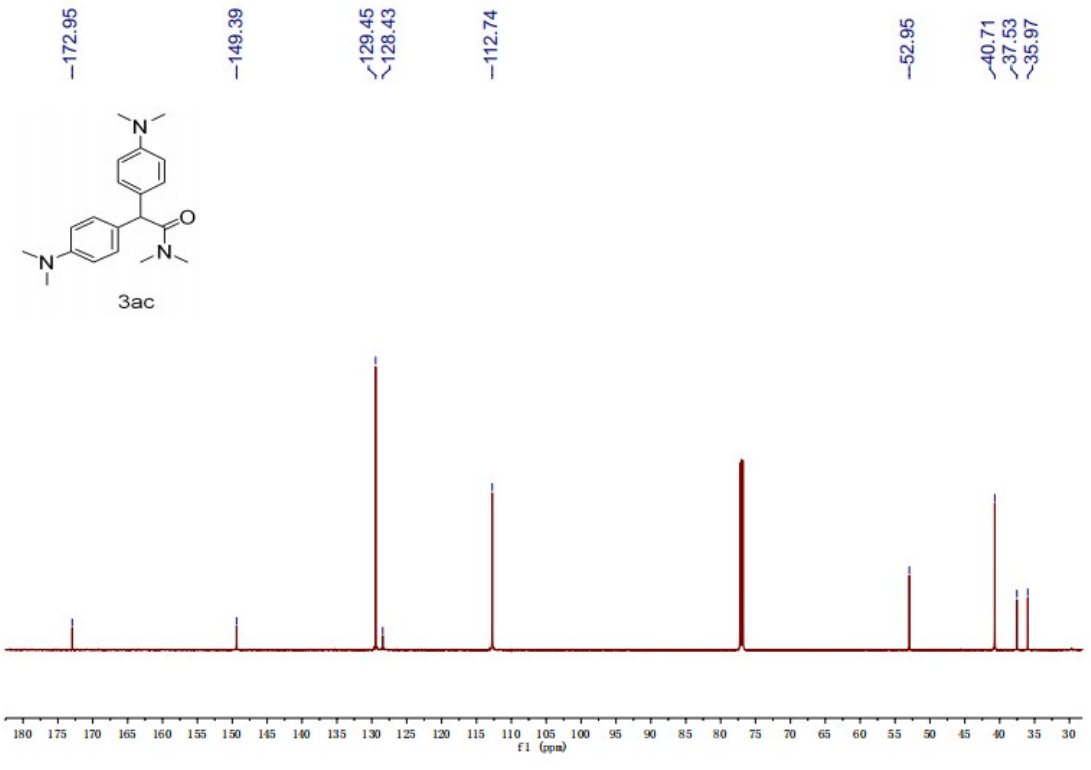
4.00
4.08
0.96
6.06
12.05
f1 (ppm)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128.43
112.74
52.95
40.71
37.53
35.97
3ac
f1 (ppm)

5. X-ray Crystallography of 3p

Table 1. Crystal data and structure refinement for a.

Identification code	a	
Empirical formula	C22 H30 N2 O4	
Formula weight	386.48	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	C2/c	
Unit cell dimensions	a = 30.064(7) Å	α= 90°.
	b = 9.445(2) Å	β= 105.189(7)°.
	c = 15.361(4) Å	γ = 90°.
Volume	4209.5(17) $Å^3$	
Z	8	
Density (calculated)	1.220 Mg/m^3	
Absorption coefficient	0.084 mm^{-1}	
F(000)	1664	
Crystal size	0.300 x 0.200 x 0.100 mm^3	
Theta range for data collection	2.739 to 27.608°.	
Index ranges	-38<=h<=38, -12<=k<=12, -19<=l<=19	
Reflections collected	55919	
Independent reflections	4834 [R(int) = 0.0604]	
Completeness to theta = 25.242°	99.0 %	
Absorption correction	None	
Max. and min. transmission	0.992 and 0.981	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	4834 / 0 / 260	
Goodness-of-fit on F^2	1.474	
Final R indices [I>2sigma(I)]	R1 = 0.0568, wR2 = 0.1861	
R indices (all data)	R1 = 0.0726, wR2 = 0.1978	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.261 and -0.264 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for a. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	3945(1)	2353(1)	7370(1)	65(1)
O(2)	4056(1)	1094(1)	6214(1)	63(1)
O(3)	3608(1)	4257(1)	4157(1)	56(1)
O(4)	2978(1)	1616(1)	6142(1)	51(1)
C(1)	4850(1)	7930(2)	4240(2)	77(1)
N(1)	4910(1)	7404(2)	5135(1)	73(1)
C(3)	4624(1)	6367(2)	5314(1)	49(1)
C(4)	4261(1)	5837(2)	4622(1)	45(1)
C(5)	3966(1)	4824(1)	4807(1)	40(1)
C(6)	4011(1)	4305(1)	5677(1)	40(1)
C(7)	3666(1)	3240(1)	5843(1)	40(1)
C(8)	3239(1)	3906(1)	6018(1)	37(1)
C(9)	3159(1)	5340(1)	5996(1)	43(1)
C(10)	2749(1)	5916(2)	6078(1)	48(1)
C(11)	2393(1)	5037(2)	6189(1)	48(1)
N(2)	1981(1)	5591(2)	6271(1)	70(1)
C(13)	1901(1)	7083(2)	6197(2)	90(1)
C(14)	1599(1)	4712(3)	6289(2)	91(1)
C(15)	3898(1)	2208(2)	6579(1)	45(1)
C(16)	4255(1)	-61(2)	6806(2)	84(1)
C(17)	3937(1)	-1256(3)	6644(2)	122(1)
C(18)	3571(1)	4556(2)	3239(1)	54(1)
C(19)	4676(1)	5828(2)	6186(1)	56(1)
C(20)	4374(1)	4839(2)	6349(1)	50(1)
C(2)	5309(1)	7840(2)	5816(2)	76(1)
C(22)	2884(1)	3030(1)	6136(1)	40(1)
C(12)	2638(1)	635(2)	6237(1)	57(1)
C(21)	2470(1)	3570(2)	6222(1)	46(1)

Table 3. Bond lengths [Å] and angles [°] for a.

O(1)-C(15)	1.1945(17)
O(2)-C(15)	1.3363(19)
O(2)-C(16)	1.446(2)
O(3)-C(5)	1.3712(16)
O(3)-C(18)	1.4135(18)
O(4)-C(22)	1.3645(17)
O(4)-C(12)	1.4160(18)
C(1)-N(1)	1.427(2)
C(1)-H(20)	0.9600
C(1)-H(21)	0.9600
C(1)-H(1)	0.9600
N(1)-C(3)	1.376(2)
N(1)-C(2)	1.431(2)
C(3)-C(4)	1.402(2)
C(3)-C(19)	1.403(2)
C(4)-C(5)	1.3828(19)
C(4)-H(25)	0.9300
C(5)-C(6)	1.3956(19)
C(6)-C(20)	1.386(2)
C(6)-C(7)	1.5146(18)
C(7)-C(15)	1.5172(19)
C(7)-C(8)	1.5156(18)
C(7)-H(26)	0.9800
C(8)-C(9)	1.375(2)
C(8)-C(22)	1.3980(19)
C(9)-C(10)	1.382(2)
C(9)-H(9)	0.9300
C(10)-C(11)	1.399(2)
C(10)-H(8)	0.9300
C(11)-N(2)	1.3828(19)
C(11)-C(21)	1.404(2)
N(2)-C(14)	1.421(2)
N(2)-C(13)	1.430(3)
C(13)-H(6)	0.9600
C(13)-H(7)	0.9600
C(13)-H(2)	0.9600

C(14)-H(4)	0.9600
C(14)-H(5)	0.9600
C(14)-H(3)	0.9600
C(16)-C(17)	1.456(4)
C(16)-H(10)	0.9700
C(16)-H(14)	0.9700
C(17)-H(13)	0.9600
C(17)-H(12)	0.9600
C(17)-H(11)	0.9600
C(18)-H(16)	0.9600
C(18)-H(17)	0.9600
C(18)-H(15)	0.9600
C(19)-C(20)	1.371(2)
C(19)-H(18)	0.9300
C(20)-H(19)	0.9300
C(2)-H(22)	0.9600
C(2)-H(23)	0.9600
C(2)-H(24)	0.9600
C(22)-C(21)	1.382(2)
C(12)-H(27)	0.9600
C(12)-H(29)	0.9600
C(12)-H(28)	0.9600
C(21)-H(30)	0.9300

C(15)-O(2)-C(16)	117.58(16)
C(5)-O(3)-C(18)	119.39(11)
C(22)-O(4)-C(12)	119.15(12)
N(1)-C(1)-H(20)	109.5
N(1)-C(1)-H(21)	109.5
H(20)-C(1)-H(21)	109.5
N(1)-C(1)-H(1)	109.5
H(20)-C(1)-H(1)	109.5
H(21)-C(1)-H(1)	109.5
C(3)-N(1)-C(1)	120.82(15)
C(3)-N(1)-C(2)	120.41(16)
C(1)-N(1)-C(2)	118.25(15)
N(1)-C(3)-C(4)	120.40(14)
N(1)-C(3)-C(19)	122.01(14)

C(4)-C(3)-C(19)	117.58(14)
C(5)-C(4)-C(3)	120.35(13)
C(5)-C(4)-H(25)	119.8
C(3)-C(4)-H(25)	119.8
O(3)-C(5)-C(4)	122.89(12)
O(3)-C(5)-C(6)	114.88(12)
C(4)-C(5)-C(6)	122.22(12)
C(20)-C(6)-C(5)	116.46(13)
C(20)-C(6)-C(7)	123.82(12)
C(5)-C(6)-C(7)	119.71(12)
C(6)-C(7)-C(15)	110.49(11)
C(6)-C(7)-C(8)	113.88(11)
C(15)-C(7)-C(8)	112.35(11)
C(6)-C(7)-H(26)	106.5
C(15)-C(7)-H(26)	106.5
C(8)-C(7)-H(26)	106.5
C(9)-C(8)-C(22)	116.84(12)
C(9)-C(8)-C(7)	123.73(12)
C(22)-C(8)-C(7)	119.21(12)
C(8)-C(9)-C(10)	122.68(13)
C(8)-C(9)-H(9)	118.7
C(10)-C(9)-H(9)	118.7
C(9)-C(10)-C(11)	120.35(13)
C(9)-C(10)-H(8)	119.8
C(11)-C(10)-H(8)	119.8
N(2)-C(11)-C(10)	121.30(14)
N(2)-C(11)-C(21)	120.90(14)
C(10)-C(11)-C(21)	117.80(13)
C(11)-N(2)-C(14)	121.90(15)
C(11)-N(2)-C(13)	120.12(16)
C(14)-N(2)-C(13)	117.27(16)
N(2)-C(13)-H(6)	109.5
N(2)-C(13)-H(7)	109.5
H(6)-C(13)-H(7)	109.5
N(2)-C(13)-H(2)	109.5
H(6)-C(13)-H(2)	109.5
H(7)-C(13)-H(2)	109.5
N(2)-C(14)-H(4)	109.5

N(2)-C(14)-H(5)	109.5
H(4)-C(14)-H(5)	109.5
N(2)-C(14)-H(3)	109.5
H(4)-C(14)-H(3)	109.5
H(5)-C(14)-H(3)	109.5
O(1)-C(15)-O(2)	123.89(14)
O(1)-C(15)-C(7)	126.37(14)
O(2)-C(15)-C(7)	109.73(12)
O(2)-C(16)-C(17)	109.45(19)
O(2)-C(16)-H(10)	109.8
C(17)-C(16)-H(10)	109.8
O(2)-C(16)-H(14)	109.8
C(17)-C(16)-H(14)	109.8
H(10)-C(16)-H(14)	108.2
C(16)-C(17)-H(13)	109.5
C(16)-C(17)-H(12)	109.5
H(13)-C(17)-H(12)	109.5
C(16)-C(17)-H(11)	109.5
H(13)-C(17)-H(11)	109.5
H(12)-C(17)-H(11)	109.5
O(3)-C(18)-H(16)	109.5
O(3)-C(18)-H(17)	109.5
H(16)-C(18)-H(17)	109.5
O(3)-C(18)-H(15)	109.5
H(16)-C(18)-H(15)	109.5
H(17)-C(18)-H(15)	109.5
C(20)-C(19)-C(3)	120.65(14)
C(20)-C(19)-H(18)	119.7
C(3)-C(19)-H(18)	119.7
C(19)-C(20)-C(6)	122.73(14)
C(19)-C(20)-H(19)	118.6
C(6)-C(20)-H(19)	118.6
N(1)-C(2)-H(22)	109.5
N(1)-C(2)-H(23)	109.5
H(22)-C(2)-H(23)	109.5
N(1)-C(2)-H(24)	109.5
H(22)-C(2)-H(24)	109.5
H(23)-C(2)-H(24)	109.5

O(4)-C(22)-C(21)	123.38(13)
O(4)-C(22)-C(8)	114.59(12)
C(21)-C(22)-C(8)	122.01(13)
O(4)-C(12)-H(27)	109.5
O(4)-C(12)-H(29)	109.5
H(27)-C(12)-H(29)	109.5
O(4)-C(12)-H(28)	109.5
H(27)-C(12)-H(28)	109.5
H(29)-C(12)-H(28)	109.5
C(22)-C(21)-C(11)	120.31(13)
C(22)-C(21)-H(30)	119.8
C(11)-C(21)-H(30)	119.8

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for a. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	71(1)	76(1)	43(1)	14(1)	7(1)	12(1)
O(2)	72(1)	45(1)	77(1)	12(1)	28(1)	16(1)
O(3)	53(1)	76(1)	35(1)	6(1)	2(1)	-23(1)
O(4)	48(1)	38(1)	68(1)	6(1)	16(1)	-1(1)
C(1)	68(1)	74(1)	91(2)	16(1)	24(1)	-21(1)
N(1)	55(1)	80(1)	77(1)	14(1)	6(1)	-28(1)
C(3)	40(1)	49(1)	56(1)	1(1)	10(1)	-3(1)
C(4)	43(1)	50(1)	42(1)	6(1)	12(1)	-1(1)
C(5)	35(1)	46(1)	37(1)	-1(1)	8(1)	-1(1)
C(6)	40(1)	43(1)	36(1)	0(1)	11(1)	1(1)
C(7)	42(1)	42(1)	34(1)	2(1)	9(1)	1(1)
C(8)	41(1)	39(1)	30(1)	3(1)	6(1)	1(1)
C(9)	47(1)	41(1)	40(1)	2(1)	9(1)	-2(1)
C(10)	55(1)	40(1)	47(1)	1(1)	10(1)	7(1)
C(11)	46(1)	53(1)	43(1)	-2(1)	10(1)	8(1)
N(2)	55(1)	64(1)	95(1)	-8(1)	26(1)	13(1)
C(13)	72(1)	73(1)	127(2)	1(1)	30(1)	29(1)
C(14)	56(1)	92(2)	135(2)	-23(1)	43(1)	1(1)
C(15)	42(1)	45(1)	48(1)	7(1)	14(1)	0(1)
C(16)	78(1)	59(1)	114(2)	30(1)	22(1)	22(1)
C(17)	148(3)	64(1)	146(3)	31(2)	25(2)	-2(2)
C(18)	60(1)	63(1)	35(1)	2(1)	6(1)	-5(1)
C(19)	49(1)	62(1)	48(1)	-2(1)	0(1)	-11(1)
C(20)	53(1)	57(1)	37(1)	4(1)	5(1)	-3(1)
C(2)	48(1)	65(1)	106(2)	-7(1)	6(1)	-13(1)
C(22)	45(1)	38(1)	35(1)	3(1)	7(1)	2(1)
C(12)	55(1)	46(1)	67(1)	6(1)	9(1)	-9(1)
C(21)	42(1)	49(1)	49(1)	4(1)	12(1)	-1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{-3}) for a.

	x	y	z	U(eq)
H(20)	4881	7166	3848	116
H(21)	5080	8635	4240	116
H(1)	4549	8344	4031	116
H(25)	4217	6168	4036	54
H(26)	3562	2685	5288	47
H(9)	3390	5948	5923	52
H(8)	2709	6893	6059	58
H(6)	2105	7554	6699	136
H(7)	1587	7278	6197	136
H(2)	1956	7421	5645	136
H(4)	1490	4247	5717	137
H(5)	1357	5280	6407	137
H(3)	1694	4015	6756	137
H(10)	4314	241	7430	101
H(14)	4545	-342	6696	101
H(13)	3651	-974	6757	183
H(12)	4068	-2021	7040	183
H(11)	3884	-1560	6029	183
H(16)	3516	5550	3131	81
H(17)	3319	4027	2868	81
H(15)	3852	4295	3095	81
H(18)	4917	6146	6657	67
H(19)	4415	4512	6936	60
H(22)	5215	8330	6286	114
H(23)	5492	8460	5555	114
H(24)	5488	7023	6064	114
H(27)	2569	784	6806	86
H(29)	2751	-311	6214	86
H(28)	2364	768	5757	86
H(30)	2242	2958	6303	56

Table 6. Torsion angles [°] for a.

C(1)-N(1)-C(3)-C(4)	-1.8(3)
C(2)-N(1)-C(3)-C(4)	-173.41(16)
C(1)-N(1)-C(3)-C(19)	179.84(17)
C(2)-N(1)-C(3)-C(19)	8.2(3)
N(1)-C(3)-C(4)-C(5)	-178.19(14)
C(19)-C(3)-C(4)-C(5)	0.3(2)
C(18)-O(3)-C(5)-C(4)	9.1(2)
C(18)-O(3)-C(5)-C(6)	-171.10(13)
C(3)-C(4)-C(5)-O(3)	-179.32(13)
C(3)-C(4)-C(5)-C(6)	0.9(2)
O(3)-C(5)-C(6)-C(20)	179.05(12)
C(4)-C(5)-C(6)-C(20)	-1.2(2)
O(3)-C(5)-C(6)-C(7)	-2.29(19)
C(4)-C(5)-C(6)-C(7)	177.46(12)
C(20)-C(6)-C(7)-C(15)	-35.31(18)
C(5)-C(6)-C(7)-C(15)	146.14(13)
C(20)-C(6)-C(7)-C(8)	92.23(16)
C(5)-C(6)-C(7)-C(8)	-86.32(15)
C(6)-C(7)-C(8)-C(9)	2.38(17)
C(15)-C(7)-C(8)-C(9)	128.96(13)
C(6)-C(7)-C(8)-C(22)	176.84(11)
C(15)-C(7)-C(8)-C(22)	-56.58(16)
C(22)-C(8)-C(9)-C(10)	-0.49(19)
C(7)-C(8)-C(9)-C(10)	174.08(12)
C(8)-C(9)-C(10)-C(11)	0.0(2)
C(9)-C(10)-C(11)-N(2)	-179.89(14)
C(9)-C(10)-C(11)-C(21)	0.6(2)
C(10)-C(11)-N(2)-C(14)	173.26(18)
C(21)-C(11)-N(2)-C(14)	-7.2(3)
C(10)-C(11)-N(2)-C(13)	3.2(3)
C(21)-C(11)-N(2)-C(13)	-177.29(17)
C(16)-O(2)-C(15)-O(1)	6.2(2)
C(16)-O(2)-C(15)-C(7)	-174.96(14)
C(6)-C(7)-C(15)-O(1)	88.84(17)
C(8)-C(7)-C(15)-O(1)	-39.5(2)
C(6)-C(7)-C(15)-O(2)	-89.95(14)

C(8)-C(7)-C(15)-O(2)	141.67(12)
C(15)-O(2)-C(16)-C(17)	106.6(2)
N(1)-C(3)-C(19)-C(20)	177.24(17)
C(4)-C(3)-C(19)-C(20)	-1.2(2)
C(3)-C(19)-C(20)-C(6)	0.9(3)
C(5)-C(6)-C(20)-C(19)	0.3(2)
C(7)-C(6)-C(20)-C(19)	-178.34(15)
C(12)-O(4)-C(22)-C(21)	0.2(2)
C(12)-O(4)-C(22)-C(8)	-178.61(12)
C(9)-C(8)-C(22)-O(4)	179.21(12)
C(7)-C(8)-C(22)-O(4)	4.37(17)
C(9)-C(8)-C(22)-C(21)	0.4(2)
C(7)-C(8)-C(22)-C(21)	-174.48(12)
O(4)-C(22)-C(21)-C(11)	-178.50(13)
C(8)-C(22)-C(21)-C(11)	0.3(2)
N(2)-C(11)-C(21)-C(22)	179.76(14)
C(10)-C(11)-C(21)-C(22)	-0.7(2)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for a [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)