Supporting Information

Photocatalyzed redox-neutral decarboxylative alkylation of heteroaryl methanamines

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Experimental Section

General Information

The starting materials, reagents and solvents, purchased from commercial suppliers, were used without further purification. Literature procedures were used for the preparation of heteroaryl methanamines (*Med. Chem. Res.* **2018**, *27*, 864) and NHPI esters (*Eur. J. Org. Chem.* **2020**, 5801 and refs therein). Analytical TLC was performed with silica gel GF254 plates, and the products were visualized by UV detection. Flash chromatography was carried out using silica gel 200−300. ^{1}HNMR (400 MHz or 600 MHz) and ^{13}CNMR (151 MHz) spectra were measured with $CDCl_3$ and DMSO-d_6 as solvent. All chemical shifts (δ) are reported in ppm and coupling constants (*J*) in Hz. High resolution mass spectra (HR-MS) were recorded under electrospray ionization (ESI) conditions.

General procedure for the photocatalytic decarboxylative alkylation of heteroaryl methanamines

To a dried reaction tube (10 mL) with a magnetic stirring bar were added heteroaryl methanamines (**1,** 0.2 mmol), NHPI esters (**2**, 0.3 mmol), $Cu(acac)_2$ (10 mol %), xantphos (15 mol %), tmp (15 mol %), and DABCO (0.4 mmol) successively. Air was then withdrawn and backfilled with argon for 3 times. Subsequently, degassed acetonitrile (2 mL) was added and the resulting reaction mixture was performed at room temperature under blue LEDs irradiation (6 W) for 12-24 hours. After the reaction was completed, the reaction mixture was concentrated under reduced pressure, and the residue was purified by column chromatography to afford the desired compounds **3** (ethyl acetate/petroleum ether = 1:50 to 1:20).

Scale-up experiment

To a dried reaction tube (50 mL) with a magnetic stirring bar were added heteroaryl methanamine **1b (**0.98 g), NHPI ester **2a** (1.91 g), $Cu(acac)_2$ (92 mg), xantphos (304 mg), tmp (124 mg), and DABCO (785 mg) successively. Air was then withdrawn and backfilled with argon for 3 times. Subsequently, degassed acetonitrile (10 mL) was added and the resulting reaction mixture was performed at room temperature under blue LEDs irradiation (6 W) for 20 hours. After the reaction was completed, the reaction mixture was concentrated under reduced pressure, and the residue was purified by column chromatography (ethyl acetate/petroleum ether = 1:20) to afford the desired compounds **3ba** (1.09 g, 86%).

One-pot scale-up experiment

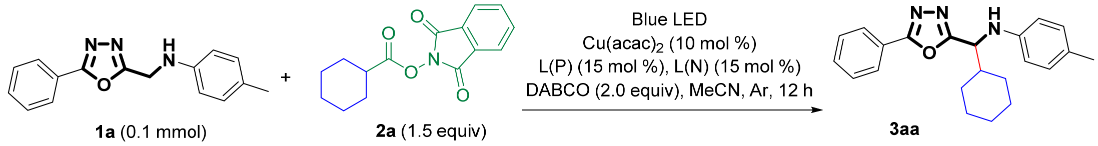
To a dried reaction tube (50 mL) with a magnetic stirring bar were added NHPI (951 mg), cyclohexanecarboxylic acid (897 mg), DMAP (35 mg), DCC (1.44 g), heteroaryl methanamine **1b** **(**0.98 g), $Cu(acac)_2$ (92 mg), xantphos (304 mg), tmp (124 mg), and DABCO (785 mg) successively. Air was then withdrawn and backfilled with argon for 3 times. Subsequently, degassed acetonitrile (10 mL) was added and the resulting reaction mixture was performed at room temperature under blue LEDs irradiation (6 W) for 20 hours. After the reaction was completed, the reaction mixture was concentrated under reduced pressure, and the residue was purified by column chromatography (ethyl acetate/petroleum ether = 1:30) to afford the desired compounds **3ba** (1.03 g, 81%). NHPI was recovered in 84% yield (0.8 g).

Deprotection of 3ba

To a stirred solution of compound **3ba** (**1.09 g**) in $MeCN/H_2O$ (1:1, 50 mL), HCl (3 mL) and TCCA (180 mg) were added. The reactions were performed at room temperature and completed as monitored by TLC. Then, MeCN was removed in vacuo. The concentrated aqueous solution was extracted with DCM for 2 times. Subsequently, water was removed in vacuo and MeOH (20 mL) was added to the residue. Non-soluble inorganic impurities were filtered and the product **4ba** (58%) was obtained by removing methanol in vacuo.

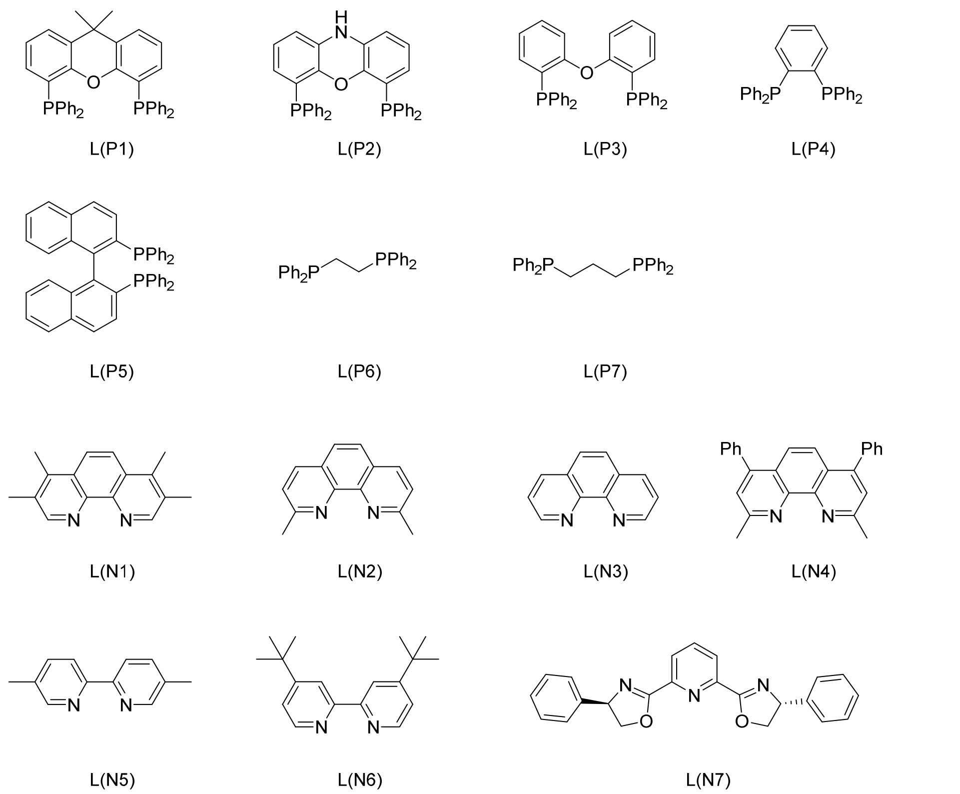
Optimization of reaction conditions

Table 1. Ligands screening for the coupling of **1a** and **2a**.[a]



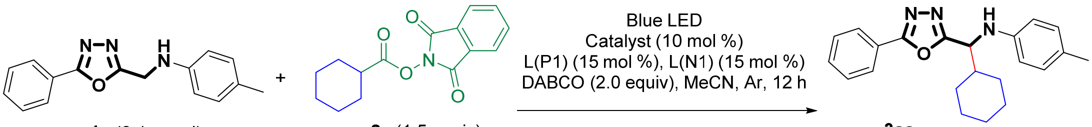
1a (0.1 mmol) **2a** (1.5 equiv) **3aa**

Entry	Ligand 1 (15 mol %)	Ligand 2 (15 mol %)	Yield (%)[b]
1	**L(P1)**	**L(N1)**	**88%**
2	L(P2)	L(N1)	17%
3	L(P3)	L(N1)	32%
4	L(P4)	L(N1)	trace
5	L(P5)	L(N1)	trace
6	L(P6)	L(N1)	trace
7	L(P7)	L(N1)	12%
8	L(P1)	L(N2)	63%
9	L(P1)	L(N3)	48%
10	L(P1)	L(N4)	43%
11	L(P1)	L(N5)	35%
12	L(P1)	L(N6)	54%
13	L(P1)	L(N7)	56%



[a] 0.1 mmol scale. [b] Isolated Yields.

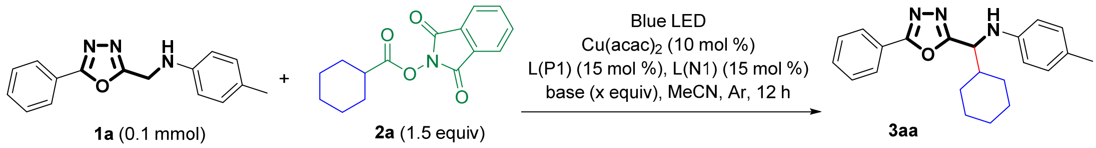
Table 2. Catalyst screening for the coupling of **1a** and **2a**.[a]



Entry	Catalyst	Yield (%)[b]
1	CuCl (10 mol %)	61%
2	CuBr (10 mol %)	60%
3	CuOAc (10 mol %)	72%
4	Cu_2O (10 mol %)	49%
5	$Cu(OAc)_2$ (10 mol %)	59%
6	$Cu(OTf)_2$ (10 mol %)	61%
7	**$Cu(acac)_2$ (10 mol %)**	**88%**
8	$CuCl_2$ (10 mol %)	71%
9	$CuBr_2$ (10 mol %)	67%
10	$Cu(MeCN)_4PF_6$ (10 mol %)	63%
11	$Fe(acac)_2$(10 mol %)	trace
12	$Co(acac)_2$(10 mol %)	15%
13	$Ni(acac)_2$(10 mol %)	trace
14	$FeCl_2$(10 mol %)	trace
15	$Co(OAc)_2$(10 mol %)	25%
16	$Ni(OTf)_2$(10 mol %)	20%

[a] 0.1 mmol scale. [b] Isolated Yields.

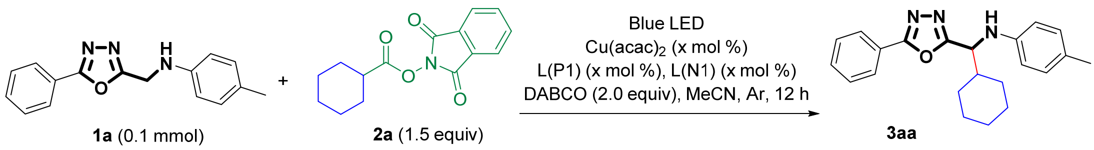
Table 3. Base screening for the coupling of **1a** and **2a**.[a]



Entry	Base	Yield (%)[b]
1	**DABCO (2.0 eq)**	**88%**
2	Et_3N (2.0 eq)	34%
3	DBU (2.0 eq)	trace
4	DMAP (2.0 eq)	16%
5	Pyridine (2.0 eq)	trace
6	Na2CO_3 (2.0 eq)	19%
7	$KHCO_3$ (2.0 eq)	53%
8	Cs_2CO_3 (2.0 eq)	40%
9	NaOAc (2.0 eq)	trace
10	DABCO (2.5 eq)	64%
11	DABCO (1.5 eq)	67%
12	DABCO (1.0 eq)	35%

[a] 0.1 mmol scale. [b] Isolated Yields.

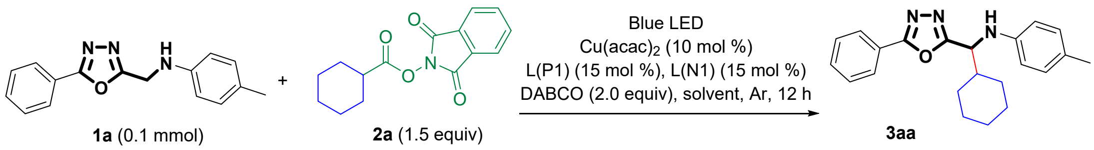
Table 4. Loading of copper salt and ligands screening for the coupling of **1a** and **2a**.[a]



Entry	Salt	Ligand 1	Ligand 2	Yield (%)[b]
1	$Cu(acac)_2$ (4 mol %)	L(P1) (15 mol %)	L(N1) (15 mol %)	62%
2	$Cu(acac)_2$ (6 mol %)	L(P1) (15 mol %)	L(N1) (15 mol %)	75%
3	$Cu(acac)_2$ (8 mol %)	L(P1) (15 mol %)	L(N1) (15 mol %)	82%
4	**$Cu(acac)_2$ (10 mol %)**	**L(P1) (15 mol %)**	**L(N1) (15 mol %)**	**88%**
5	$Cu(acac)_2$ (12 mol %)	L(P1) (15 mol %)	L(N1) (15 mol %)	52%
6	$Cu(acac)_2$ (14 mol %)	L(P1) (15 mol %)	L(N1) (15 mol %)	30%
7	$Cu(acac)_2$ (10 mol %)	L(P1) (15 mol %)	L(N1) (5 mol %)	47%
8	$Cu(acac)_2$ (10 mol %)	L(P1) (15 mol %)	L(N1) (10 mol %)	50%
9	$Cu(acac)_2$ (10 mol %)	L(P1) (15 mol %)	L(N1) (20 mol %)	30%
10	$Cu(acac)_2$ (10 mol %)	L(P1) (5 mol %)	L(N1) (15 mol %)	trace
11	$Cu(acac)_2$ (10 mol %)	L(P1) (10 mol %)	L(N1) (15 mol %)	18%
12	$Cu(acac)_2$ (10 mol %)	L(P1) (20 mol %)	L(N1) (15 mol %)	70%

[a] 0.1 mmol scale. [b] Isolated Yields.

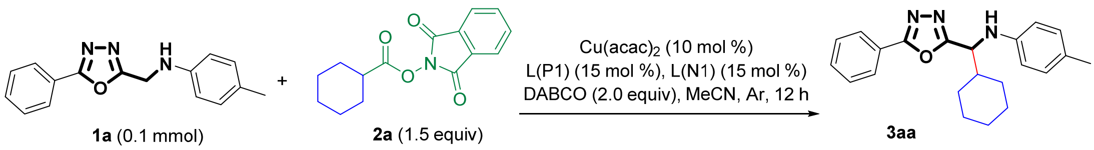
Table 5. Solvent screening for the coupling of **1a** and **2a**.[a]



Entry	Solvent	Yield (%)[b]
1	**MeCN**	**88%**
2	DMF	9%
3	Toluene	38%
4	THF	59%
5	DCM	28%
6	DCE	82%
7	Acetone	35%
8	EtOH	-
9	EA	21%

[a] 0.1 mmol scale. [b] Isolated Yields.

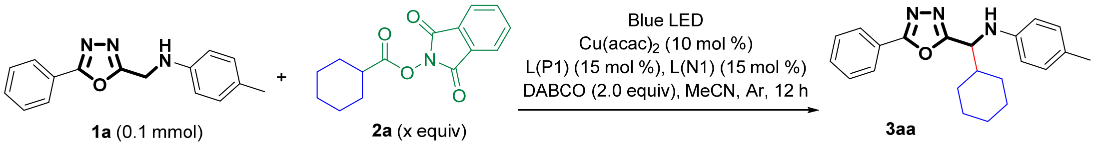
Table 6. Light sources screening for the coupling of **1a** and **2a**.[a]



Entry	Light source (6 W)	Yield (%)[b]
1	**Blue LEDs**	**88%**
2	Green LEDs	trace
3	White LEDs	41%

[a] 0.1 mmol scale. [b] Isolated Yields.

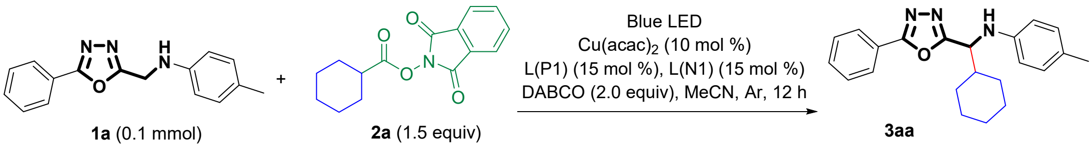
Table 7. Screening the reactant ratio.[a]



Entry	Reactant ratio(**2a**/**1a**)	Yield (%)[b]
1	1.2	76%
2	**1.5**	**88%**
3	1.8	86%
4	2.0	86%

[a] 0.1 mmol scale. [b] Isolated Yields.

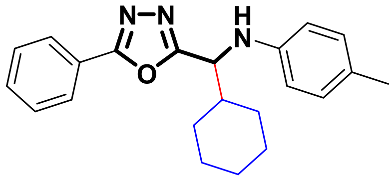
Table 8. Reaction time.[a]



Entry	Reaction time	Yield (%)[b]
1	10	71%
2	**12**	**88%**
3	18	87%
4	20	82%

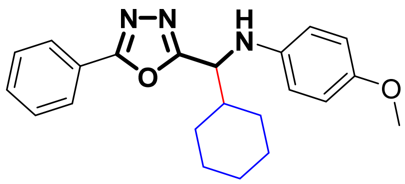
[a] 0.1 mmol scale. [b] Isolated Yields.

Characterization of the products



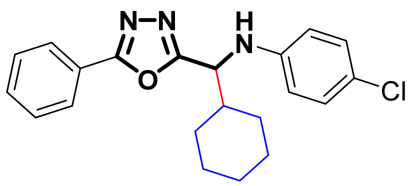
***N*-(cyclohexyl(5-phenyl-1,3,4-oxadiazol-2-yl)methyl)-4-methylaniline (3aa)**

The desired pure product was obtained in 88% yield (61.1 mg) as a white solid. M. p. 110 – 111 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.00 (d, *J* = 6.8 Hz, 2H), 7.53 – 7.45 (m, 3H), 6.96 (d, *J* = 8.1 Hz, 2H), 6.63 (d, *J* = 8.4 Hz, 2H), 4.61 (t, *J* = 6.4 Hz, 1H), 4.02 (d, *J* = 5.6 Hz, 1H), 2.19 (s, 3H), 2.06 (d, *J* = 12.8 Hz, 1H), 1.99 – 1.91 (m, 1H), 1.83 – 1.73 (dd, *J* = 28.8, 13.3 Hz, 2H), 1.68 (d, *J* = 12.9 Hz, 1H), 1.62 (d, *J* = 7.7 Hz, 1H), 1.58 (d, *J* = 12.4 Hz, 1H), 1.30 – 1.24 (m, 2H), 1.19 – 1.14 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 167.0, 164.7, 144.2, 131.6, 129.8, 128.9, 127.9, 126.9, 123.9, 113.7, 56.0, 42.2, 29.7, 29.5, 26.1, 25.9, 25.8, 20.3. HRMS (ESI) exact mass calcd for $C_{22}H_{26}N_3O$ [M+H] m/z 348.2070, found 348.2069.



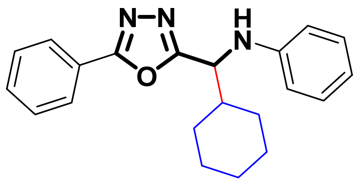
***N*-(cyclohexyl(5-phenyl-1,3,4-oxadiazol-2-yl)methyl)-4-methoxyaniline(3ba)**

The desired pure product was obtained in 98% yield (71.2 mg) as a white solid. M. p. 132 – 133 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.02 – 7.97 (m, 2H), 7.53 – 7.45 (m, 3H), 6.75 – 6.71 (m, 2H), 6.70 – 6.66 (m, 2H), 4.54 (d, *J* = 7.0 Hz, 1H), 3.90 (s, 1H), 3.70 (s, 3H), 2.06 (d, *J* = 12.9 Hz, 1H), 1.98 – 1.90 (m, 1H), 1.83 – 1.56 (m, 5H), 1.31 – 1.24 (m, 2H), 1.22 – 1.18 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 167.1, 164.7, 152.9, 140.5, 131.6, 129.0, 126.9, 123.9, 115.3, 114.9, 56.9, 55.7, 42.2, 29.7, 29.5, 26.1, 25.9, 25.8. HRMS (ESI) exact mass calcd for $C_{22}H_{26}N_3O_2$ [M+H] m/z 364.2020, found 364.2021.



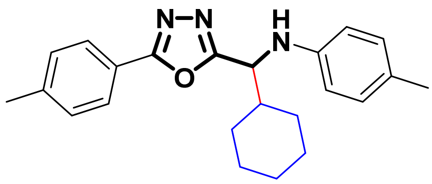
***4-Chloro-N*-(cyclohexyl(5-phenyl-1,3,4-oxadiazol-2-yl)methyl)aniline (3ca)**

The desired pure product was obtained in 46% yield (33.8 mg) as a white solid. M. p. 117 – 118 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.00 (d, *J* = 7.0 Hz, 2H), 7.52 – 7.46 (m, 3H), 7.09 (d, *J* = 8.8 Hz, 2H), 6.63 (d, *J* = 8.9 Hz, 2H), 4.60 – 4.56 (dd, *J* =9.0, 7.3 Hz, 1H), 4.16 (d, *J* = 9.0 Hz, 1H), 2.04 (d, *J* = 12.9 Hz, 1H), 1.99 – 1.94 (m, 1H), 1.78 (dd, *J* = 28.7, 13.6 Hz, 2H), 1.29 – 1.24 (m, 3H), 1.21 – 1.15 (m, 3H), 0.87 (t, *J* = 7.0 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.4, 164.9, 145.1, 131.7, 129.2, 129.0, 126.9, 123.8, 123.4, 114.7, 55.8, 42.1, 29.6, 29.5, 26.0, 25.8, 25.7. HRMS (ESI) exact mass calcd for $C_{21}H_{23}ClN_3O$ [M+H] m/z 368.1524, found 368.1519.



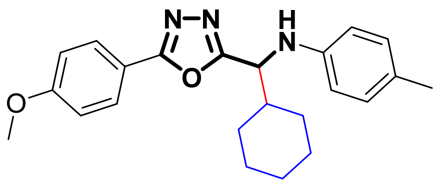
N-(cyclohexyl(5-phenyl-1,3,4-oxadiazol-2-yl)methyl)aniline (3da)

The desired pure product was obtained in 52% yield (34.7 mg) as a white solid. M. p. 165 – 166 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.00 (d, *J* = 6.9 Hz, 2H), 7.52 – 7.45 (m, 3H), 7.17 – 7.13 (t, *J* = 7.9 Hz, 2H), 6.72 (m, 3H), 4.67 – 4.62 (*J* = 8.7, 7.5 Hz, 1H), 4.14 (d, *J* = 9.0 Hz, 1H), 2.06 (d, *J* = 12.9 Hz, 1H), 1.99 – 1.94 (m, 1H), 1.83 – 1.74 (m, 2H), 1.68 (d, *J* = 12.7 Hz, 1H), 1.33 – 1.24 (m, 3H), 1.22 – 1.16 (m, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.8, 164.8, 146.5, 131.6, 129.4, 129.0, 126.9, 123.9, 118.6, 113.5, 55.6, 42.2, 29.7, 29.5, 26.1, 25.9, 25.8. HRMS (ESI) exact mass calcd for $C_{21}H_{24}N_3O$ [M+H] m/z 334.1914, found 334.1916.



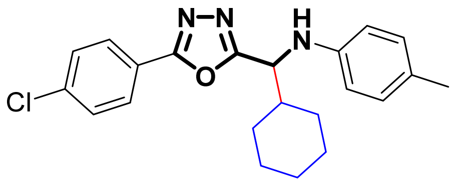
N-(cyclohexyl(5-(p-tolyl)-1,3,4-oxadiazol-2-yl)methyl)-4-methylaniline (3ea)

The desired pure product was obtained in 80% yield (57.8 mg) as a white solid. M. p. 128 – 129 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.88 (d, *J* = 8.2 Hz, 2H), 7.27 (d, *J* = 7.9 Hz, 2H), 6.95 (d, *J* = 8.0 Hz, 2H), 6.65 (d, *J* = 7.7 Hz, 2H), 4.59 (d, *J* = 7.3 Hz, 1H), 2.40 (s, 3H), 2.19 (s, 3H), 2.06 (d, *J* = 12.9 Hz, 1H), 1.99 – 1.91 (m, 1H), 1.81 – 1.72 (m, 2H), 1.70 – 1.65 (m, 1H), 1.58 (d, *J* = 12.7 Hz, 1H), 1.29 – 1.15 (m, 5H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.7, 164.8, 144.2, 142.1, 129.8, 129.6, 127.9, 126.8, 121.1, 113.8, 56.0, 42.2, 29.7, 29.5, 26.1, 25.9, 25.8, 21.6, 20.3. HRMS (ESI) exact mass calcd for $C_{23}H_{28}N_3O$ [M+H] m/z 362.2227, found 362.2231.



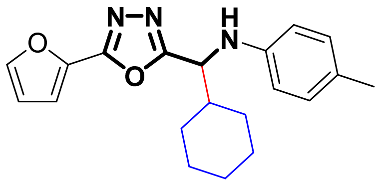
***N*-(cyclohexyl(5-(4-methoxyphenyl)-1,3,4-oxadiazol-2-yl)methyl)-4-methylaniline (3fa)**

The desired pure product was obtained in79% yield (59.6 mg) as a white solid. M. p. 113 – 114 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 9.0 Hz, 2H), 7.87 (m, 1H), 7.76 (m, 1H), 6.98 – 6.94 (m, 1H), 6.63 (d, *J* = 8.5 Hz, 1H), 4.59 (t, *J* = 7.3 Hz, 1H), 4.06 (d, *J* = 7.3 Hz, 1H), 3.86 (s, 3H), 2.19 (s, 3H), 2.06 (d, *J* = 11.1 Hz, 1H), 1.98 – 1.87 (m, 1H), 1.77 (t, *J* = 15.7 Hz, 3H), 1.67 (d, *J* = 11.7 Hz, 1H), 1.57 (d, *J* = 11.8 Hz, 1H), 1.29 – 1.23 (m, 2H), 1.20 – 1.14 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 167.9, 166.4, 164.6, 162.2, 144.2, 134.3, 132.6, 129.8, 128.6, 127.8, 123.6, 116.4, 114.4, 113.7, 55.9, 55.4, 42.2, 29.7, 29.5, 26.1, 25.9, 25.8, 20.3. HRMS (ESI) exact mass calcd for $C_{23}H_{28}N_3O_2$ [M+H] m/z 378.2176, found 378.2173.



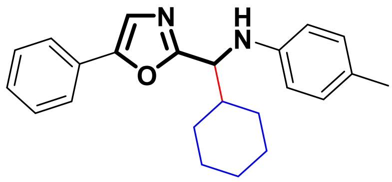
***N*-((5-(4-chlorophenyl)-1,3,4-oxadiazol-2-yl)(cyclohexyl)methyl)-4-methylaniline (3ga)**

The desired pure product was obtained in 30% yield (22.9 mg) as a white solid. M. p. 176 – 177 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.93 (d, *J* = 8.6 Hz, 2H), 7.45 (d, *J* = 8.7 Hz, 2H), 6.96 (d, *J* = 8.2 Hz, 2H), 6.63 (d, *J* = 8.2 Hz, 2H), 4.60 (d, *J* = 7.3 Hz, 1H), 2.19 (s, 3H), 2.05 (d, *J* = 13.0 Hz, 1H), 1.98 – 1.91 (m, 1H), 1.82 – 1.73 (m, 2H), 1.68 (d, *J* = 12.8 Hz, 1H), 1.57 (d, *J* = 12.7 Hz, 1H), 1.27 – 1.23 (m, 2H), 1.21 – 1.15 (m, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 167.2, 163.9, 144.1, 137.9, 129.9, 129.3, 128.1, 128.0, 122.4, 113.7, 56.0, 42.2, 29.7, 29.5, 26.1, 25.9, 25.8, 20.3. HRMS (ESI) exact mass calcd for $C_{22}H_{25}ClN_3O$ [M+H] m/z 382.1681, found 382.1679.



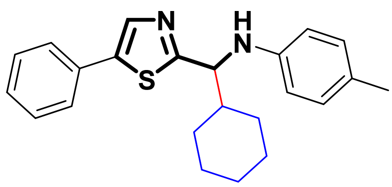
N-(cyclohexyl(5-phenyl-1,3,4-oxadiazol-2-yl)methyl)furan-2-amine (3ha)

The desired pure product was obtained in 62% yield (40.1 mg) as a white solid. M. p. 161 – 162 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.61 – 7.58 (m, 1H), 7.11 (d, *J* = 3.5 Hz, 1H), 6.95 (d, *J* = 8.5 Hz, 2H), 6.61 (d, *J* = 8.4 Hz, 2H), 6.56 (dd, *J* = 3.5, 1.7 Hz, 1H), 4.59 (d, *J* = 7.5 Hz, 1H), 4.02 (s, 1H), 2.19 (s, 3H), 2.05 (d, *J* = 13.0 Hz, 1H), 1.93 (m, 1H), 1.81 – 1.73 (m, 2H), 1.67 (d, *J* = 12.7 Hz, 1H), 1.54 (d, *J* = 12.7 Hz, 1H), 1.29 – 1.23 (m, 2H), 1.21 – 1.13 (m, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.3, 157.6, 145.5, 144.0, 139.4, 129.8, 128.0, 113.9, 113.7, 112.1, 55.8, 42.1, 29.7, 29.6, 26.1, 25.83, 25.77, 20.3. HRMS (ESI) exact mass calcd for $C_{20}H_{24}N_3O_2$ [M+H] m/z 338.1863, found 338.1860.



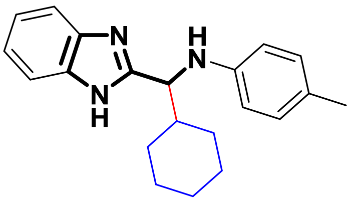
N-(cyclohexyl(5-phenyloxazol-2-yl)methyl)-4-methylaniline (3ia)

The desired pure product was obtained in 61% yield (42.3 mg) as a white solid. M. p. 138 – 139 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.58 (d, *J* = 7.2 Hz, 2H), 7.39 (t, *J* = 7.7 Hz, 2H), 7.30 (t, *J* = 7.4 Hz, 1H), 7.22 (s, 1H), 6.95 (d, *J* = 8.0 Hz, 2H), 6.62 (d, *J* = 8.5 Hz, 2H), 4.44 (d, *J* = 7.0 Hz, 1H), 2.19 (s, 3H), 2.02 (d, *J* = 13.0 Hz, 1H), 1.96 – 1.89 (m, 1H), 1.82 – 1.72 (m, 2H), 1.66 (d, *J* = 12.8 Hz, 1H), 1.56 (d, *J* = 12.8 Hz, 1H), 1.30 – 1.22 (m, 2H), 1.20 – 1.13 (m, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 164.6, 151.0, 144.8, 129.7, 128.8, 128.2, 128.1, 127.4, 124.1, 121.7, 113.7, 57.8, 42.7, 29.8, 29.5, 26.2, 26.0, 25.9, 20.4. HRMS (ESI) exact mass calcd for $C_{23}H_{27}N_2O$ [M+H] m/z 347.2118, found 347.2117.



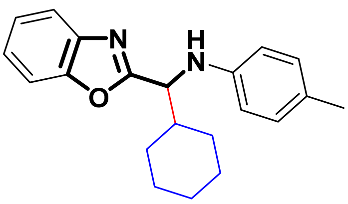
N-(cyclohexyl(5-phenylthiazol-2-yl)methyl)-4-met*hyla*niline (3ja)

The desired pure product was obtained in 56% yield (40.6 mg) as a colourless oily liquid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.89 (s, 1H), 7.49 (d, *J* = 7.2 Hz, 2H), 7.34 (t, *J* = 7.6 Hz, 2H), 7.29 – 7.24 (m, 1H), 6.95 (d, *J* = 8.1 Hz, 2H), 6.56 (d, *J* = 8.5 Hz, 2H), 4.49 (d, *J* = 5.6 Hz, 1H), 4.18 (s, 1H), 2.20 (s, 3H), 1.99 – 1.93 (m, 1H), 1.89 (d, *J* = 12.5 Hz, 1H), 1.81 – 1.75 (m, 2H), 1.71 – 1.66 (m, 2H), 1.62 (s, 1H), 1.30 – 1.25 (m, 2H), 1.22 – 1.16 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 175.3, 144.9, 138.8, 137.9, 131.6, 129.7, 128.9, 127.9, 127.4, 126.5, 113.5, 62.2, 44.4, 30.0, 28.9, 26.3, 26.2, 26.1, 20.3. HRMS (ESI) exact mass calcd for $C_{23}H_{27}N_2S$ [M+H] m/z 363.1890, found 363.1889.



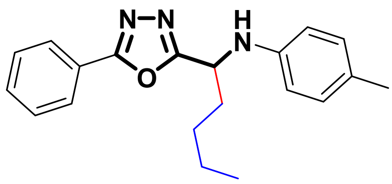
N-((1H-benzo[d]imidazol-2-yl)(cyclohexyl)methyl)-4-methylaniline (3ka)

The desired pure product was obtained in 96% yield (61.3 mg) as a white solid. M. p. 179 – 180 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.86 (dd, *J* = 5.4, 3.1 Hz, 1H), 7.75 (dd, *J* = 5.5, 3.0 Hz, 1H), 7.54 (s, 1H), 7.23 – 7.19 (m, 2H), 6.91 (d, *J* = 8.4 Hz, 2H), 6.51 (d, *J* = 8.4 Hz, 2H), 4.51 (d, *J* = 4.8 Hz, 1H), 4.11 (s, 1H), 2.18 (s, 3H), 2.12 – 2.06 (m, 1H), 1.74 (d, *J* = 11.7 Hz, 4H), 1.65 (d, *J* = 12.9 Hz, 1H), 1.30 – 1.23 (m, 3H), 1.18 – 1.08 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 156.2, 145.1, 134.2, 129.9, 128.0, 123.5, 122.3, 113.6, 59.7, 43.7, 29.9, 28.8, 26.1, 20.3. HRMS (ESI) exact mass calcd for $C_{21}H_{26}N_3$ [M+H] m/z 320.2121, found 320.2127.



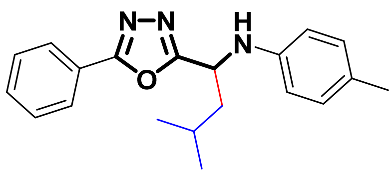
N-(benzo[d]oxazol-2-yl(cyclohexyl)methyl)-4-methylaniline (3la)

The desired pure product was obtained in 76% yield (48.7 mg) as a white solid. M. p. 178 – 179 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.69 – 7.64 (m, 1H), 7.49 – 7.44 (m, 1H), 7.31 – 7.27 (m, 2H), 6.93 (d, *J* = 8.1 Hz, 2H), 6.63 (d, *J* = 8.5 Hz, 2H), 4.53 (s, 1H), 4.18 (s, 1H), 2.18 (s, 3H), 2.05 (d, *J* = 12.7 Hz, 1H), 2.01 – 1.94 (m, 1H), 1.81 – 1.61 (m, 4H), 1.28 – 1.22 (m, 2H), 1.21 – 1.14 (m, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 167.1, 150.6, 144.6, 140.9, 129.8, 127.5, 124.7, 124.2, 119.9, 113.6, 110.6, 58.1, 42.7, 29.7, 29.6, 26.2, 26.0, 25.9, 20.3. HRMS (ESI) exact mass calcd for $C_{21}H_{25}N_2O$ [M+H] m/z 321.1961, found 321.1965.



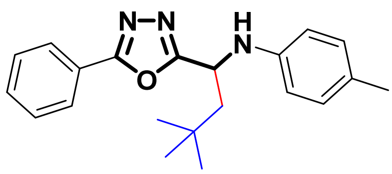
4-Methyl-N-(1-(5-phenyl-1,3,4-oxadiazol-2-yl)pentyl)aniline (3ab).

The desired pure product was obtained in 34% yield (21.8 mg) as a white solid. M. p. 104 – 105 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.00 (d, *J* = 6.9 Hz, 2H), 7.53 – 7.45 (m, 3H), 6.97 (d, *J* = 8.0 Hz, 2H), 6.65 (d, *J* = 8.4 Hz, 2H), 4.80 (t, *J* = 7.1 Hz, 1H), 2.20 (s, 3H), 2.03 (dd, *J* = 13.9, 7.0 Hz, 2H), 1.50 – 1.36 (m, 4H), 0.90 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 167.5, 164.8, 143.8, 131.6, 129.9, 128.9, 128.1, 126.9, 123.9, 113.8, 50.7, 34.2, 27.9, 22.3, 20.3, 13.8. HRMS (ESI) exact mass calcd for $C_{20}H_{24}N_3O$ [M+H] m/z 322.1914, found 322.1911.



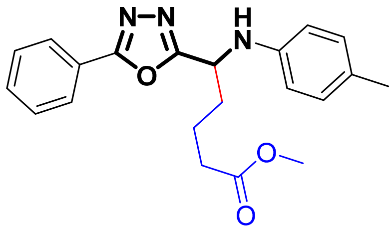
4-Methyl-N-(3-methyl-1-(5-phenyl-1,3,4-oxadiazol-2-yl)butyl)aniline (3ac).

The desired pure product was obtained in 41% yield (26.4 mg) as a white solid. M. p. 138 – 139 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 6.6 Hz, 2H), 7.53 – 7.44 (m, 3H), 6.97 (d, *J* = 8.0 Hz, 2H), 6.66 (d, *J* = 8.5 Hz, 2H), 4.95 – 4.83 (m, 1H), 3.89 (d, *J* = 8.6 Hz, 1H), 2.20 (s, 3H), 1.98 – 1.85 (m, 2H), 1.81 – 1.73 (m, 1H), 1.03 (d, *J* = 6.6 Hz, 3H), 0.96 (d, *J* = 6.5 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 167.7, 164.7, 143.8, 131.6, 129.9, 128.9, 128.1, 126.9, 123.9, 113.7, 48.9, 43.5, 24.9, 22.5, 22.2, 20.3. HRMS (ESI) exact mass calcd for $C_{20}H_{24}N_3O$ [M+H] m/z 322.1914, found 322.1909.



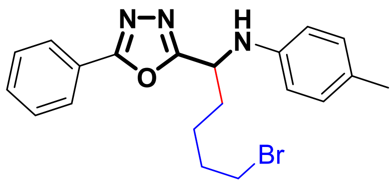
N-(3,3-dimethyl-1-(5-phenyl-1,3,4-oxadiazol-2-yl)butyl)-4-methylaniline (3ad)

The desired pure product was obtained in 68% yield (45.6 mg) as a white solid. M. p. 141 – 142 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 8.1 Hz, 2H), 7.55 – 7.42 (m, 3H), 6.97 (d, *J* = 8.1 Hz, 2H), 6.66 (d, *J* = 7.8 Hz, 2H), 4.95 – 4.86 (m, 1H), 3.82 (d, *J* = 8.2 Hz, 1H), 2.20 (s, 3H), 2.06 – 1.88 (m, 2H), 0.99 (s, 9H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 168.0, 164.6, 143.4, 131.6, 129.9, 128.9, 128.0, 126.9, 123.9, 113.7, 48.1, 47.9, 30.6, 29.8, 20.3. HRMS (ESI) exact mass calcd for $C_{21}H_{26}N_3O$ [M+H] m/z 336.2070, found 336.2068.



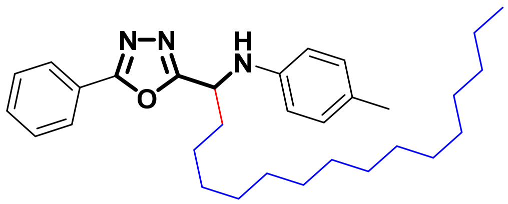
Methyl 5-(5-phenyl-1,3,4-oxadiazol-2-yl)-5-(p-tolylamino)pentanoate (3ae)

The desired pure product was obtained in 40% yield (29.2 mg) as a white solid. M. p. 117 – 118 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.99 (d, *J* = 7.0 Hz, 2H), 7.53 – 7.44 (m, 3H), 6.97 (d, *J* = 8.4 Hz, 2H), 6.65 (d, *J* = 8.4 Hz, 2H), 4.81 (q, 7.3 Hz, 1H), 4.02 (d, *J* = 8.6 Hz, 1H), 3.65 (s, 3H), 2.40 (t, *J* = 7.3 Hz, 2H), 2.20 (s, 3H), 2.13 – 2.03 (m, 2H), 1.93 – 1.85 (m, 1H), 1.82 – 1.74 (m, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 173.3, 167.2, 164.9, 143.7, 131.7, 129.9, 129.0, 128.2, 126.9, 123.8, 113.8, 51.6, 50.5, 33.6, 33.3, 21.2, 20.4. HRMS (ESI) exact mass calcd for $C_{21}H_{24}N_3O_3$ [M+H] m/z 366.1812, found 366.1814.



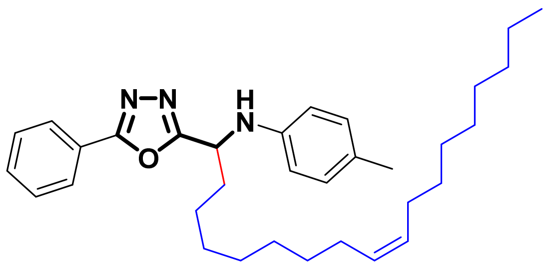
N-(5-bromo-1-(5-phenyl-1,3,4-oxadiazol-2-yl)pentyl)-4-methylaniline (3af)

The desired pure product was obtained in 32% yield (25.6 mg) as a white solid. M. p. 122 – 123 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.89 (d, *J* = 7.0 Hz, 2H), 7.51 – 7.40 (m, 3H), 7.03 (d, *J* = 8.2 Hz, 2H), 6.92 (d, *J* = 8.6 Hz, 2H), 5.15 (t, *J* = 4.4 Hz, 1H), 3.41 – 3.36 (m, 1H), 3.33 – 3.28 (m, 1H), 2.23 (s, 3H), 2.20 – 2.13 (m, 1H), 1.89 – 1.84 (m, 1H), 1.79 – 1.72 (m, 3H), 1.30 – 1.23 (m, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.7, 164.4, 148.3, 131.5, 130.0, 129.6, 128.9, 126.8, 123.9, 117.9, 54.1, 46.8, 29.3, 25.4, 20.4, 20.3. HRMS (ESI) exact mass calcd for $C_{20}H_{23}BrN_3O$ [M+H] m/z 400.1019, found 400.1022.



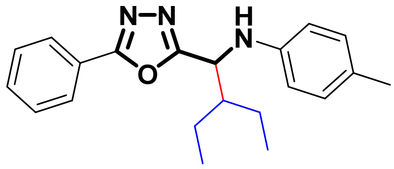
4-Methyl-N-(1-(5-phenyl-1,3,4-oxadiazol-2-yl)hexadecyl)aniline (3ag)

The desired pure product was obtained in 58% yield (55.1 mg) as a white solid. M. p. 94 – 95 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.99 (d, *J* = 7.0 Hz, 2H), 7.53 – 7.44 (m, 3H), 6.97 (d, *J* = 8.2 Hz, 2H), 6.65 (d, *J* = 8.4 Hz, 2H), 4.85 – 4.75 (m, 1H), 3.96 (d, *J* = 8.4 Hz, 1H), 2.20 (s, 3H), 2.06 – 1.97 (m, 2H), 1.67 – 1.62 (m, 1H), 1.53 – 1.47 (m, 1H), 1.38 – 1.31 (m, 2H), 1.30 – 1.20 (m, 22H), 0.87 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 167.6, 164.8, 143.8, 131.6, 129.9, 128.9, 128.0, 126.9, 123.9, 113.8, 50.7, 34.5, 31.9, 29.67, 29.66, 29.64, 29.63, 29.61, 29.57, 29.5, 29.3, 29.2, 25.8, 22.7, 20.3, 14.1. HRMS (ESI) exact mass calcd for $C_{31}H_{46}N_3O$ [M+H] m/z 476.3635, found 476.3633.



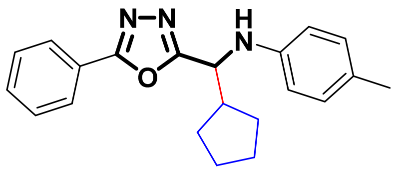
(Z)-4-methyl-N-(1-(5-phenyl-1,3,4-oxadiazol-2-yl)octadec-9-en-1-yl)aniline (3ah)

The desired pure product was obtained in 48% yield (48.2 mg) as a colourless oily liquid. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.22 (d, *J* = 7.1 Hz, 1H), 7.99 (d, *J* = 7.0 Hz, 3H), 7.48 (m, 4H), 7.30 (d, *J* = 8.3 Hz, 1H), 6.97 (d, *J* = 8.3 Hz, 3H), 6.65 (d, *J* = 8.4 Hz, 3H), 4.80 (t, *J* = 6.9 Hz, 2H), 3.96 (s, 1H), 2.20 (s, 4H), 2.05 – 1.96 (m, 9H), 1.38 – 1.21 (m, 31H), 0.87 (t, *J* = 7.0 Hz, 4H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 167.6, 164.8, 143.8, 142.3, 131.6, 130.1, 130.0, 129.9, 129.7, 129.1, 128.9, 128.1, 127.6, 126.9, 121.4, 113.8, 50.7, 34.5, 31.9, 29.7, 29.6, 29.5, 29.3, 29.2, 29.1, 29.0, 27.2, 27.1, 25.8, 22.7, 21.2, 20.4, 14.1. HRMS (ESI) exact mass calcd for $C_{33}H_{48}N_3O$ [M+H] m/z 502.3792, found 502.3788.



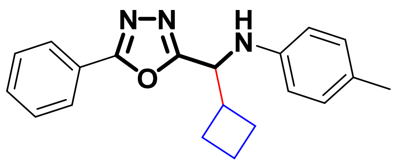
N-(2-ethyl-1-(5-phenyl-1,3,4-oxadiazol-2-yl)butyl)-4-methylaniline (3ai)

The desired pure product was obtained in 79% yield (26.5 mg) as a white solid. M. p. 105 – 106 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.99 (d, *J* = 6.9 Hz, 2H), 7.52 – 7.44 (m, 3H), 6.96 (d, *J* = 8.3 Hz, 2H), 6.64 (d, *J* = 8.4 Hz, 2H), 4.80 (t, *J* = 6.7 Hz, 1H), 4.00 (d, *J* = 7.0 Hz, 1H), 2.19 (s, 3H), 1.91 – 1.86 (m, 1H), 1.64 – 1.61 (m, 1H), 1.50 – 1.44 (m, 1H), 1.36 – 1.31 (m, 1H), 0.97 (t, *J* = 7.4 Hz, 3H), 0.93 (t, *J* = 7.4 Hz, 3H), 0.88 (t, *J* = 7.0 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 167.3, 164.7, 144.1, 131.6, 129.9, 129.0, 128.0, 126.8, 123.9, 113.7, 52.9, 45.3, 22.3, 21.8, 20.3, 11.3, 10.9. HRMS (ESI) exact mass calcd for $C_{21}H_{26}N_3O$ [M+H] m/z 336.2070, found 336.2072.



N-(cyclopentyl(5-phenyl-1,3,4-oxadiazol-2-yl)methyl)-4-methylaniline (3aj)

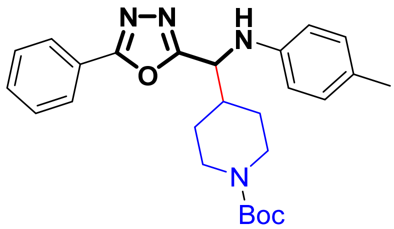
The desired pure product was obtained in 75% yield (25.0 mg) as a white solid. M. p. 145 – 146 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.99 (m, 2H), 7.53 – 7.44 (m, 3H), 6.96 (d, *J* = 8.1 Hz, 2H), 6.65 (d, *J* = 8.5 Hz, 2H), 4.59 (t, *J* = 8.7 Hz, 1H), 4.00 (d, *J* = 8.5 Hz, 1H), 2.54 – 2.46 (m, 1H), 2.19 (s, 3H), 2.02 – 1.94 (m, 1H), 1.72 – 1.63 (m, 3H), 1.61 – 1.53 (m, 2H), 1.47 – 1.40 (m, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 167.4, 164.7, 144.0, 131.6, 129.8, 128.9, 128.0, 126.9, 123.9, 113.7, 55.1, 43.9, 30.0, 29.3, 25.4, 25.3, 20.3. HRMS (ESI) exact mass calcd for $C_{21}H_{24}N_3O$ [M+H] m/z 334.1914, found 334.1911.



N-(cyclobutyl(5-phenyl-1,3,4-oxadiazol-2-yl)methyl)-4-methylaniline (3ak)

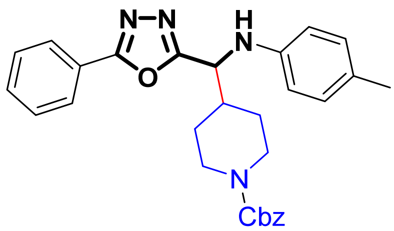
The desired pure product was obtained in 60% yield (38.3 mg) as a white solid. M. p. 132 – 133 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.98 (d, *J* = 6.9 Hz, 2H), 7.52 – 7.44 (m, 3H), 6.97 (d, *J* = 8.2 Hz, 2H), 6.65

(d, J = 8.5 Hz, 2H), 4.74 (t, J = 8.6 Hz, 1H), 3.88 (d, J = 8.0 Hz, 1H), 2.94 – 2.84 (m, 1H), 2.20 (s, 3H), 2.04 – 1.89 (m, 5H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.6, 164.8, 144.1, 131.6, 129.8, 128.9, 128.1, 126.9, 123.9, 113.8, 55.5, 38.8, 25.7, 24.7, 20.3, 17.8. HRMS (ESI) exact mass calcd for $C_{20}H_{22}N_3O$ [M+H] m/z 320.1757, found 320.1751.



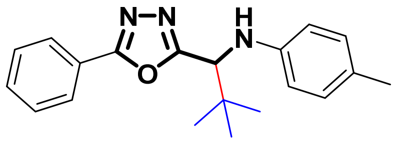
Tert-butyl 4-((5-phenyl-1,3,4-oxadiazol-2-yl)(p-tolylamino)methyl)piperidine-1-carboxylate (3al)

The desired pure product was obtained in 65% yield (58.3 mg) as a white solid. M. p. 141 – 142 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.99 (d, J = 7.0 Hz, 2H), 7.54 – 7.45 (m, 3H), 6.97 (d, J = 8.1 Hz, 2H), 6.64 (d, J = 8.4 Hz, 2H), 4.65 (d, J = 6.9 Hz, 1H), 4.17 (s, 2H), 4.01 (s, 1H), 2.70 (s, 2H), 2.20 (s, 3H), 2.13 – 2.07 (m, 1H), 2.03 (d, J = 12.6 Hz, 1H), 1.43 (s, 9H), 1.28 – 1.24 (m, 1H), 0.88 (t, J = 7.0 Hz, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.4, 164.9, 154.6, 143.8, 131.8, 129.9, 129.0, 128.4, 126.9, 123.7, 114.0, 79.6, 55.4, 40.7, 31.6, 28.5, 28.4, 22.6, 20.3, 14.1. HRMS (ESI) exact mass calcd for $C_{26}H_{33}N_4O_3$ [M+H] m/z 449.2547, found 449.2550.



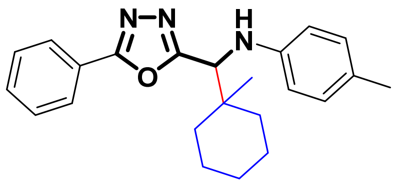
Benzyl 4-((5-phenyl-1,3,4-oxadiazol-2-yl)(p-tolylamino)methyl)piperidine-1-carboxylate (3am)

The desired pure product was obtained in 58% yield (56.0 mg) as a white solid. M. p. 209 – 210 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, J = 6.8 Hz, 2H), 7.54 – 7.46 (m, 3H), 7.41 – 7.27 (m, 5H), 6.98 (d, J = 8.1 Hz, 2H), 6.64 (d, J = 8.3 Hz, 2H), 5.11 (s, 2H), 4.65 (dd, J = 9.5, 7.6 Hz, 1H), 4.26 (s, 2H), 4.02 (d, J = 9.8 Hz, 1H), 2.80 (s, 2H), 2.21 (s, 3H), 2.17 – 2.01 (m, 2H), 1.59 – 1.25 (m, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.3, 164.9, 155.1, 143.7, 136.7, 131.8, 129.9, 129.0, 128.5, 128.4, 128.0, 127.9, 126.9, 123.7, 114.0, 67.1, 55.3, 43.8, 43.6, 40.6, 20.3. HRMS (ESI) exact mass calcd for $C_{29}H_{31}N_4O_3$ [M+H] m/z 483.2391, found 483.2385.



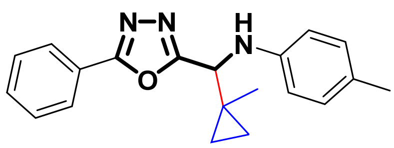
***N*-(2,2-dimethyl-1-(5-phenyl-1,3,4-oxadiazol-2-yl)propyl)-4-methylaniline (3an)**

The desired pure product was obtained in 75% yield (48.2 mg) as a white solid. M. p. 179 – 180 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.99 (d, *J* = 6.9 Hz, 2H), 7.54 – 7.44 (m, 3H), 6.95 (d, *J* = 8.2 Hz, 2H), 6.66 (d, *J* = 8.2 Hz, 2H), 4.55 (s, 1H), 2.19 (s, 3H), 1.14 (s, 9H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.7, 164.6, 144.4, 131.6, 129.8, 129.0, 128.1, 126.8, 123.9, 114.1, 60.0, 35.3, 26.6, 20.3. HRMS (ESI) exact mass calcd for $C_{20}H_{24}N_3O$ [M+H] m/z 322.1914, found 322.1916.



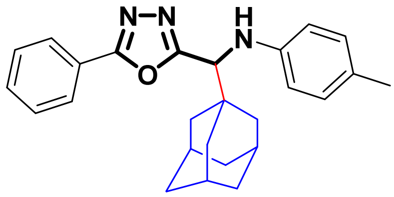
4-Methyl-*N*-((1-methylcyclohexyl)(5-phenyl-1,3,4-oxadiazol-2-yl)methyl)aniline (3ao)

The desired pure product was obtained in 95% yield (68.7 mg) as a white solid. M. p. 185 – 186 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.99 (d, *J* = 6.8 Hz, 2H), 7.52 – 7.44 (m, 3H), 6.95 (d, *J* = 8.3 Hz, 2H), 6.65 (d, *J* = 8.4 Hz, 2H), 4.70 (d, *J* = 10.2 Hz, 1H), 4.09 (d, *J* = 10.2 Hz, 1H), 2.18 (s, 3H), 1.61 – 1.46 (m, 7H), 1.36 – 1.23 (m, 3H), 1.14 (s, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.6, 164.6, 144.5, 131.6, 129.8, 129.0, 128.0, 126.8, 123.9, 114.0, 37.9, 34.9, 34.8, 26.0, 21.8, 21.5, 20.3. HRMS (ESI) exact mass calcd for $C_{23}H_{28}N_3O$ [M+H] m/z 362.2227, found 362.2229.



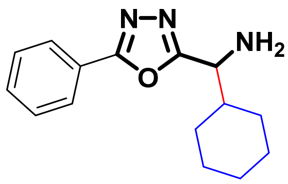
4-Methyl-*N*-((1-methylcyclopropyl)(5-phenyl-1,3,4-oxadiazol-2-yl)methyl)aniline (3ap)

The desired pure product was obtained in 52% yield (33.22 mg) as a white solid. M. p. 115 – 116 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.01 (d, *J* = 6.5 Hz, 2H), 7.54 – 7.46 (m, 3H), 6.96 (d, *J* = 8.0 Hz, 2H), 6.60 (d, *J* = 8.4 Hz, 2H), 4.34 (d, *J* = 7.3 Hz, 1H), 4.24 (d, *J* = 7.3 Hz, 1H), 2.20 (s, 3H), 1.27 (s, 3H), 0.91 – 0.82 (m, 1H), 0.67 – 0.59 (m, 1H), 0.56 – 0.43 (m, 2H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.7, 164.8, 144.1, 131.7, 129.8, 129.0, 128.2, 126.9, 123.9, 113.9, 57.9, 20.3, 20.0, 19.4, 12.6, 11.5. HRMS (ESI) exact mass calcd for $C_{20}H_{22}N_3O$ [M+H] m/z 320.1757, found 320.1754.



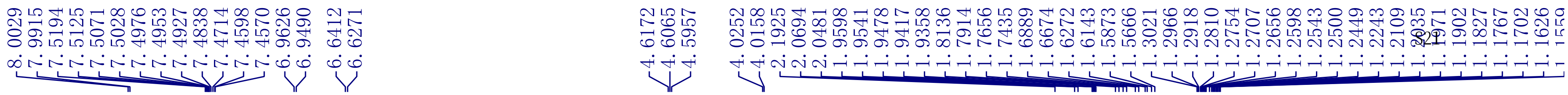
N-(((3r,5r,7r)-adamantan-1-yl)(5-phenyl-1,3,4-oxadiazol-2-yl)methyl)-4-methylaniline (3aq)

The desired pure product was obtained in 92% yield (73.51 mg) as a white solid. M. p. 105 – 106 °C. ^{1}H NMR (600 MHz, $CDCl_3$) δ 8.00 (d, *J* = 6.7 Hz, 2H), 7.54 – 7.45 (m, 3H), 6.94 (d, *J* = 8.3 Hz, 2H), 6.63 (d, *J* = 8.5 Hz, 2H), 4.41 (d, *J* = 9.9 Hz, 1H), 4.12 (d, *J* = 10.0 Hz, 1H), 2.18 (s, 3H), 2.04 (s, 3H), 1.88 (dd, *J* = 12.2, 1.8 Hz, 3H), 1.73 (d, *J* = 12.3 Hz, 3H), 1.65 (d, *J* = 11.7 Hz, 3H), 1.60 (d, *J* = 1.5 Hz, 2H), 1.29 – 1.24 (m, 1H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 166.2, 164.6, 144.5, 131.6, 129.8, 129.0, 128.0, 126.9, 124.0, 114.0, 60.7, 38.9, 37.0, 36.7, 28.3, 20.3. HRMS (ESI) exact mass calcd for $C_{26}H_{30}N_3O$ [M+H] m/z 400.2383, found 400.2384.

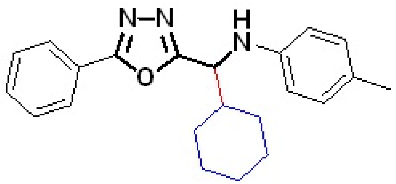


Cyclohexyl(5-phenyl-1,3,4-oxadiazol-2-yl)methanamine (4ba)

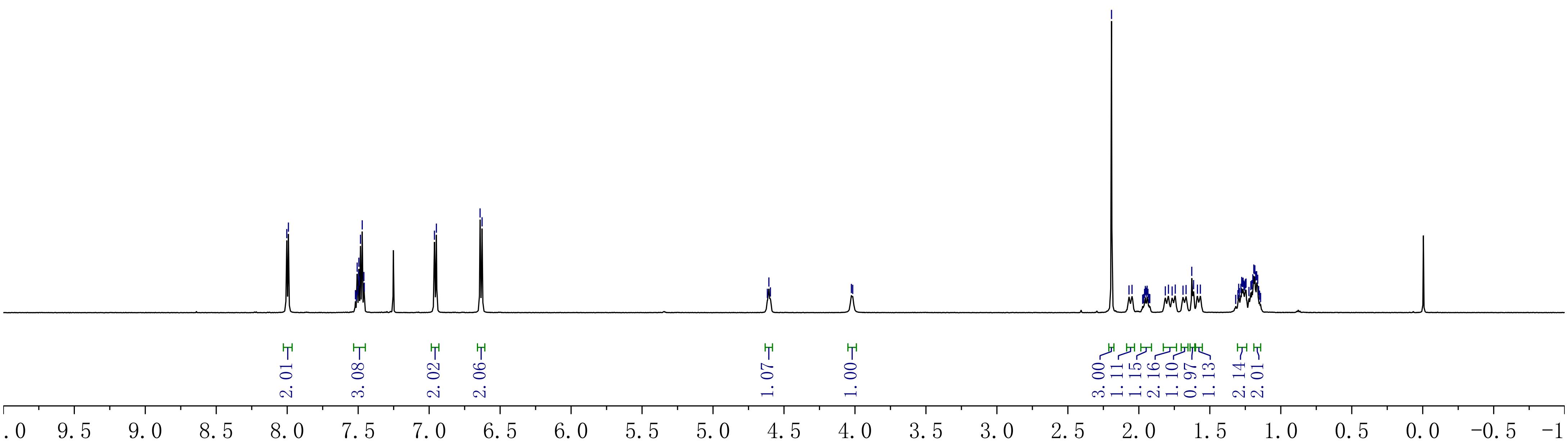
^{1}H NMR (400 MHz, DMSO-d_6) δ 9.17 (s, 2H), 8.03 (d, *J* = 6.4 Hz, 2H), 7.68 – 7.59 (m, 3H), 4.66 (d, *J* = 6.7 Hz, 1H), 2.06 (d, *J* = 7.0 Hz, 1H), 1.87 (d, *J* = 11.9 Hz, 1H), 1.73 (d, *J* = 11.8 Hz, 1H), 1.62 (d, *J* = 16.6 Hz, 2H), 1.26 – 0.94 (m, 6H). ^{13}C NMR (151 MHz, DMSO) δ 165.2, 163.0, 150.3, 132.9, 130.0, 127.2, 123.2, 51.1, 28.7, 27.9, 25.6, 25.5, 25.4. HRMS (ESI) exact mass calcd for $C_{15}H_{20}N_3O$ [M+H] m/z 258.1601, found 258.1606.



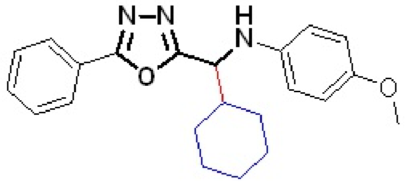
[1]H NMR (600 MHz, $CDCl_3$) δ 8.00 (d, J = 6.8 Hz, 2H), 7.53 – 7.45 (m, 3H), 6.96 (d, J = 8.1 Hz, 2H), 6.63 (d, J = 8.4 Hz, 2H), 4.61 (t, J = 6.4 Hz, 1H), 4.02 (d, J = 5.6 Hz, 1H), 2.19 (s, 3H), 2.06 (d, J = 12.8 Hz, 1H), 1.99 – 1.91 (m, 1H), 1.83 – 1.73 (dd, J = 28.8, 13.3 Hz, 2H), 1.68 (d, J = 12.9 Hz, 1H), 1.62 (d, J = 7.7 Hz, 1H), 1.58 (d, J = 12.4 Hz, 1H), 1.30 – 1.24 (m, 2H), 1.19 – 1.14 (m, 2H).



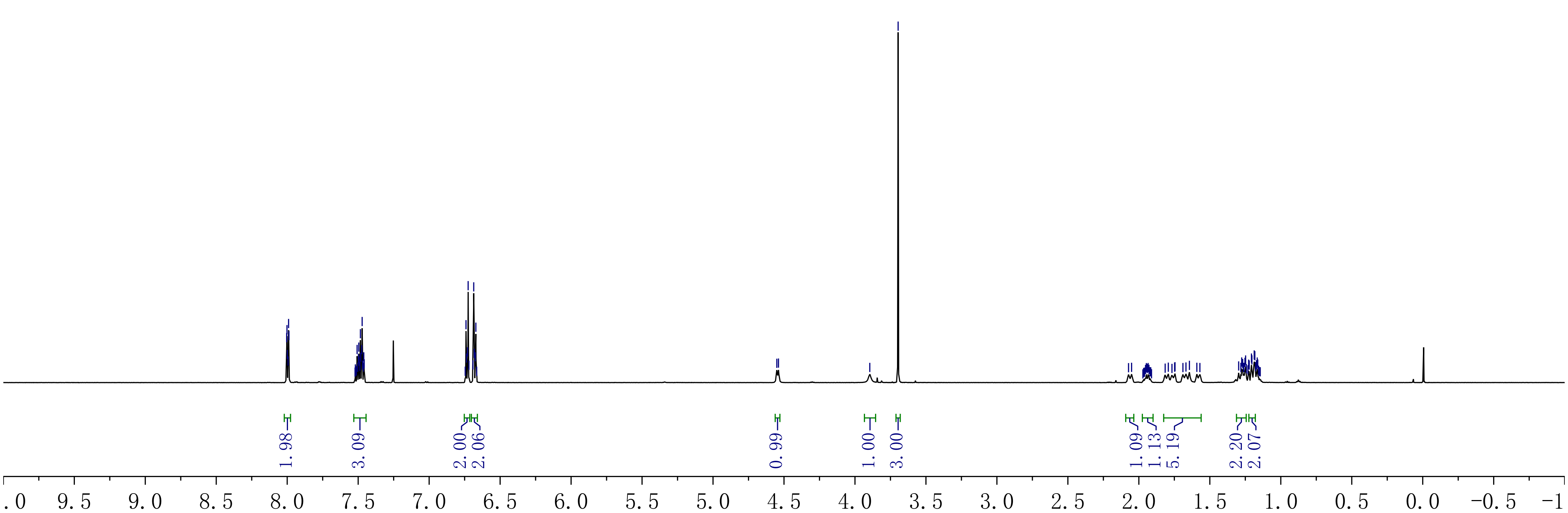
3aa



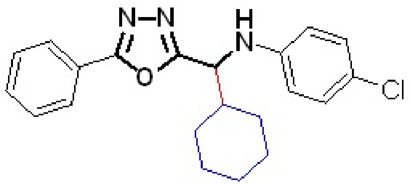
[1]H NMR (600 MHz, $CDCl_3$) δ 8.02 – 7.97 (m, 2H), 7.53 – 7.45 (m, 3H), 6.75 – 6.71 (m, 2H), 6.70 – 6.66 (m, 2H), 4.54 (d, *J* = 7.0 Hz, 1H), 3.90 (s, 1H), 3.70 (s, 3H), 2.06 (d, *J* = 12.9 Hz, 1H), 1.98 – 1.90 (m, 1H), 1.83 – 1.56 (m, 5H), 1.31 – 1.24 (m, 2H), 1.22 – 1.18 (m, 2H).



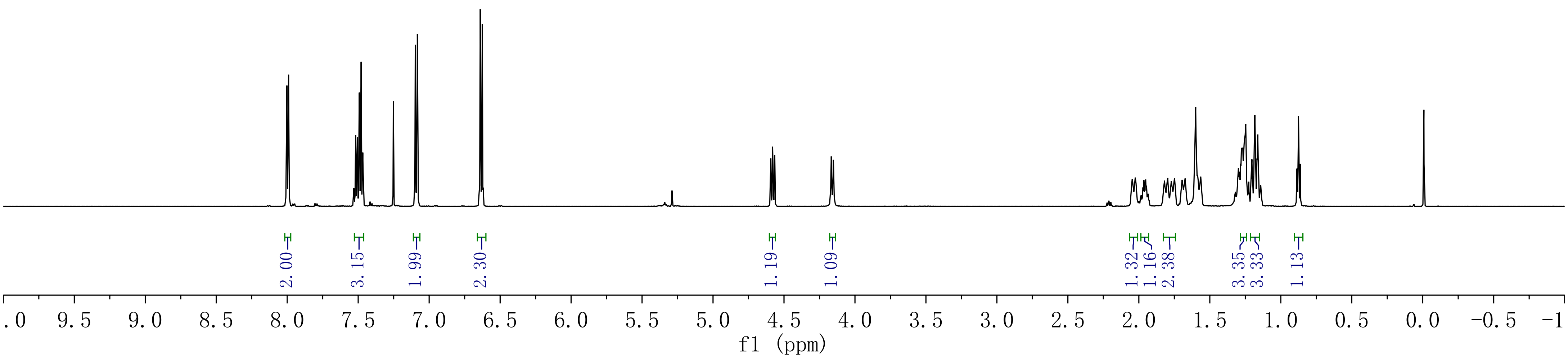
3ba



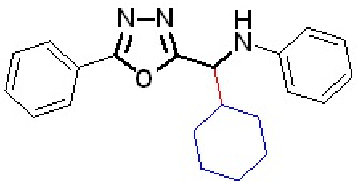
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.00 (d, J = 7.0 Hz, 2H), 7.52 – 7.46 (m, 3H), 7.09 (d, J = 8.8 Hz, 2H), 6.63 (d, J = 8.9 Hz, 2H), 4.60 – 4.56 (dd, J =9.0, 7.3 Hz, 1H), 4.16 (d, J = 9.0 Hz, 1H), 2.04 (d, J = 12.9 Hz, 1H), 1.99 – 1.94 (m, 1H), 1.78 (dd, J = 28.7, 13.6 Hz, 2H), 1.29 – 1.24 (m, 3H), 1.21 – 1.15 (m, 3H), 0.87 (t, J = 7.0 Hz, 1H).



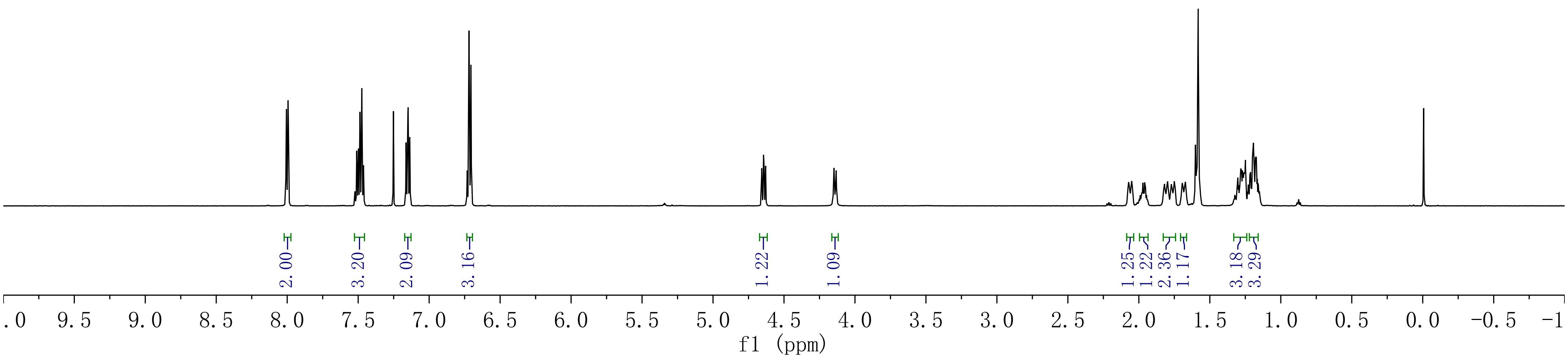
3ca



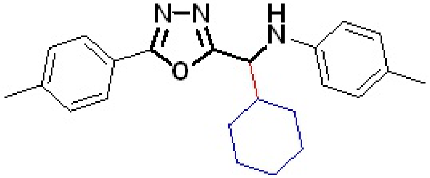
[1]H NMR (600 MHz, $CDCl_3$) δ 8.00 (d, *J* = 6.9 Hz, 2H), 7.52 – 7.45 (m, 3H), 7.17 – 7.13 (t, *J* = 7.9 Hz, 2H), 6.72 (m, 3H), 4.67 – 4.62 (dd, *J* = 8.7, 7.5 Hz, 1H), 4.14 (d, *J* = 9.0 Hz, 1H), 2.06 (d, *J* = 12.9 Hz, 1H), 1.99 – 1.94 (m, 1H), 1.83 – 1.74 (m, 2H), 1.68 (d, *J* = 12.7 Hz, 1H), 1.33 – 1.24 (m, 3H), 1.22 – 1.16 (m, 3H).



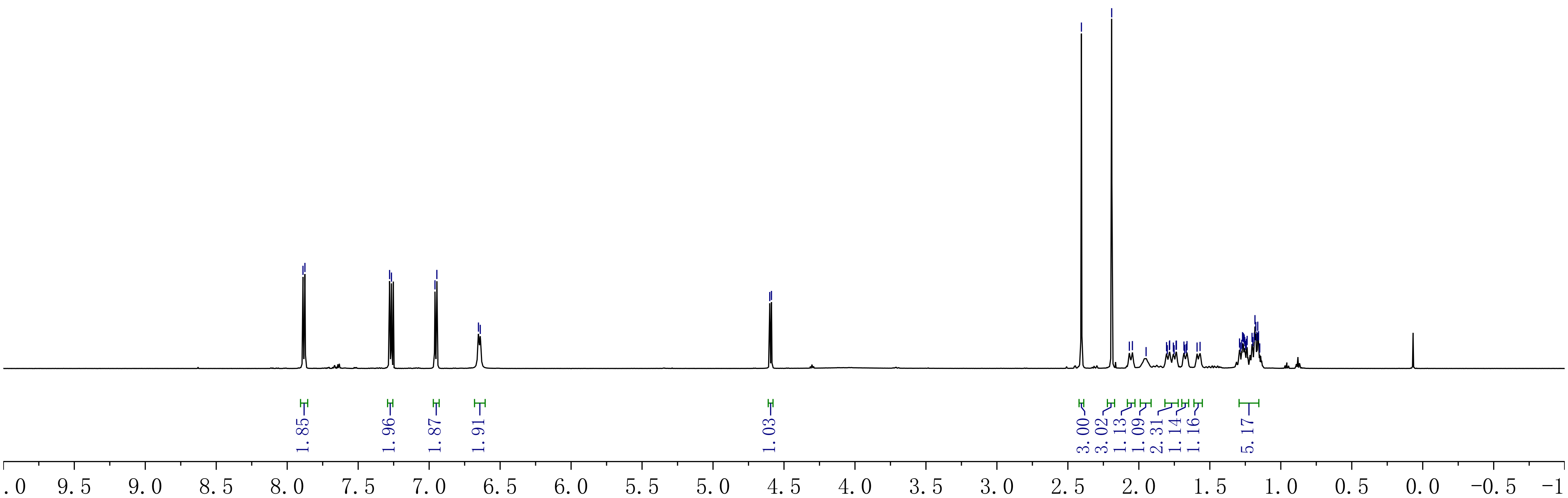
3da



^{1}H NMR (600 MHz, $CDCl_3$) δ 7.88 (d, *J* = 8.2 Hz, 2H), 7.27 (d, *J* = 7.9 Hz, 2H), 6.95 (d, *J* = 8.0 Hz, 2H), 6.65 (d, *J* = 7.7 Hz, 2H), 4.59 (d, *J* = 7.3 Hz, 1H), 2.40 (s, 3H), 2.19 (s, 3H), 2.06 (d, *J* = 12.9 Hz, 1H), 1.99 – 1.91 (m, 1H), 1.81 – 1.72 (m, 2H), 1.70 – 1.65 (m, 1H), 1.58 (d, *J* = 12.7 Hz, 1H), 1.29 – 1.15 (m, 5H).



3ea

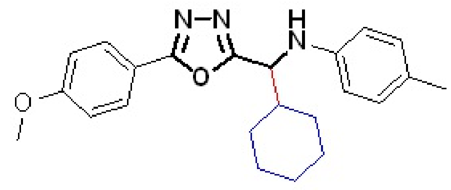


7.9413 7.9188 7.8791 7.8714 7.8656 7.8577 7.7639 7.7563 7.7501 7.7425 6.9799 6.9626 6.9575 6.9418 6.6448 6.6236

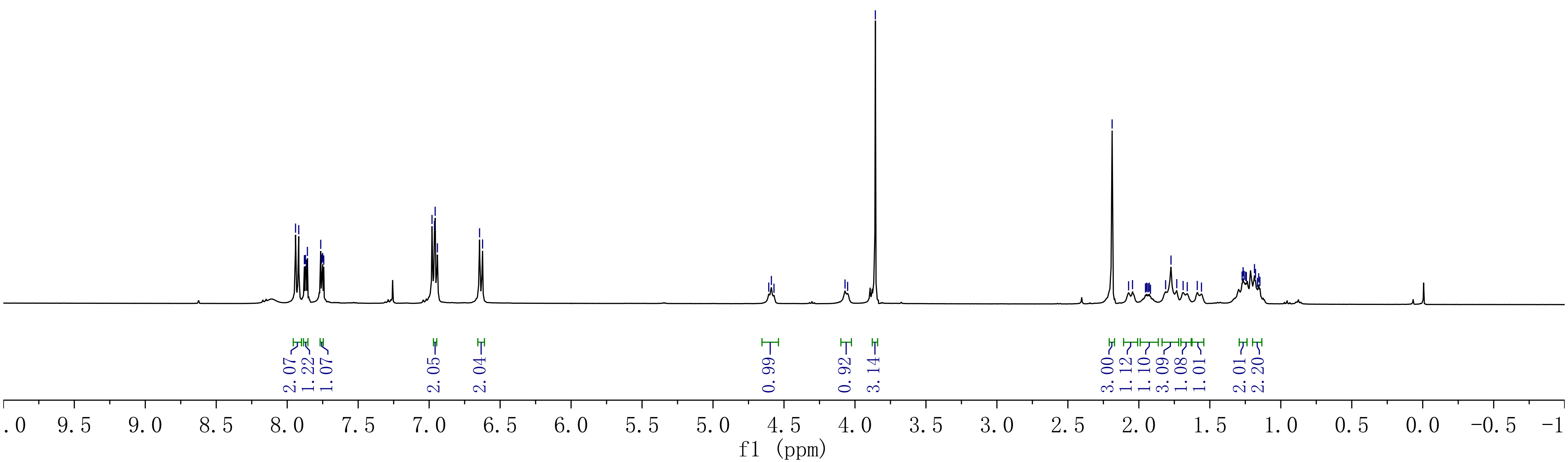
4.6073 4.5889 4.5706 4.0701 4.0518 3.8564

2.1884 2.0723 2.0446 1.9533 1.9452 1.9355 1.9263 1.9180 1.8114 1.7735 1.7329 1.6882 1.6590 1.5885 1.5589 1.2726 1.2657 1.2584 1.2477 1.2426 1.1854 1.1781 1.1645 1.1557 1.1471

[1]H NMR (400 MHz, $CDCl_3$) δ 7.93 (d, *J* = 9.0 Hz, 2H), 7.87 (m, 1H), 7.76 (m, 1H), 6.98 – 6.94 (m, 1H), 6.63 (d, *J* = 8.5 Hz, 1H), 4.59 (t, *J* = 7.3 Hz, 1H), 4.06 (d, *J* = 7.3 Hz, 1H), 3.86 (s, 3H), 2.19 (s, 3H), 2.06 (d, *J* = 11.1 Hz, 1H), 1.98 – 1.87 (m, 1H), 1.77 (m, 3H), 1.67 (d, *J* = 11.7 Hz, 1H), 1.57 (d, *J* = 11.8 Hz, 1H), 1.29 – 1.23 (m, 2H), 1.20 – 1.14 (m, 2H).

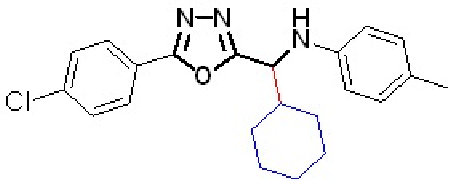


3fa

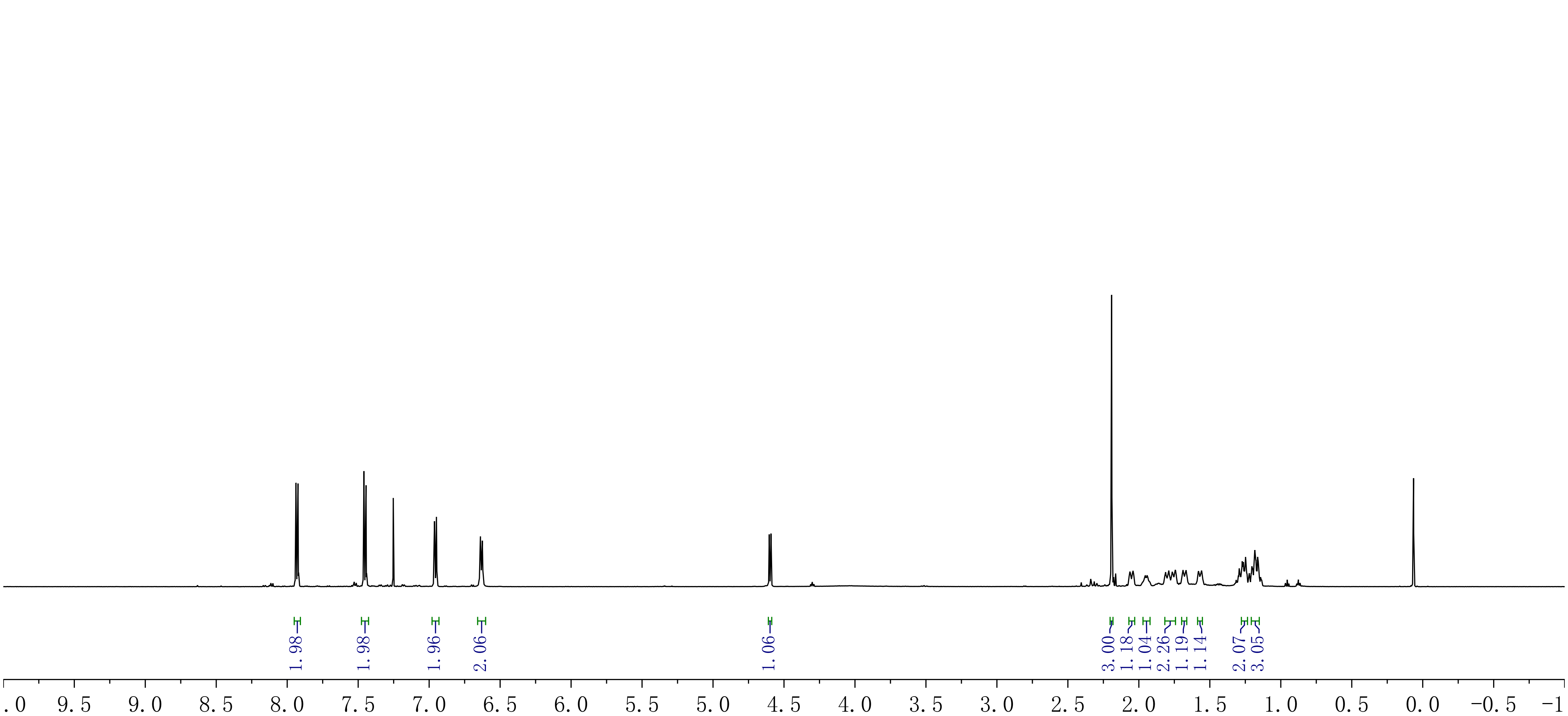


S27

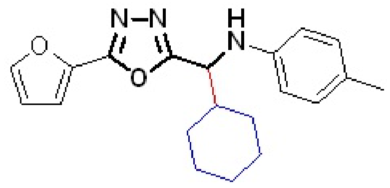
¹H NMR (600 MHz, $CDCl_3$) δ 7.93 (d, J = 8.6 Hz, 2H), 7.45 (d, J = 8.7 Hz, 2H), 6.96 (d, J = 8.2 Hz, 2H), 6.63 (d, J = 8.2 Hz, 2H), 4.60 (d, J = 7.3 Hz, 1H), 2.19 (s, 3H), 2.05 (d, J = 13.0 Hz, 1H), 1.98 – 1.91 (m, 1H), 1.82 – 1.73 (m, 2H), 1.68 (d, J = 12.8 Hz, 1H), 1.57 (d, J = 12.7 Hz, 1H), 1.27 – 1.23 (m, 2H), 1.21 – 1.15 (m, 3H).



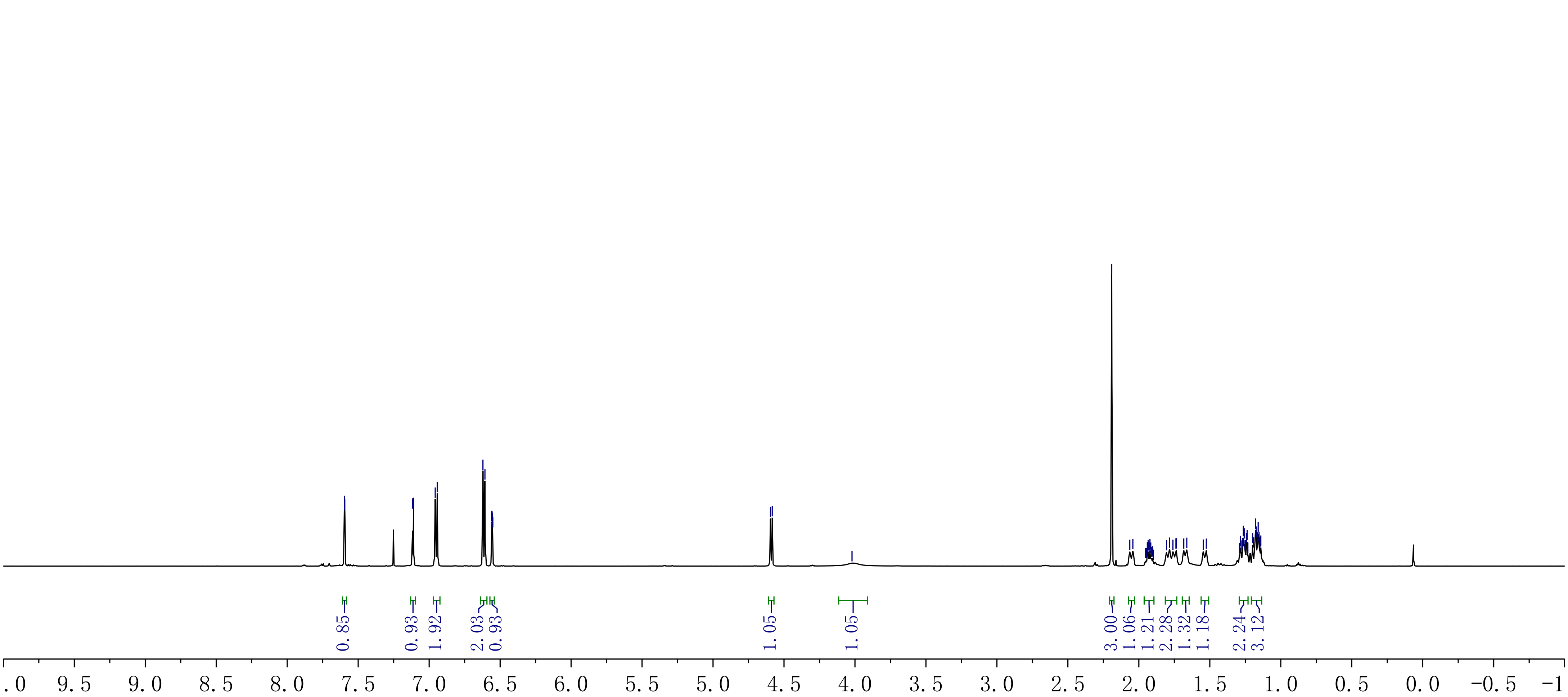
3ga

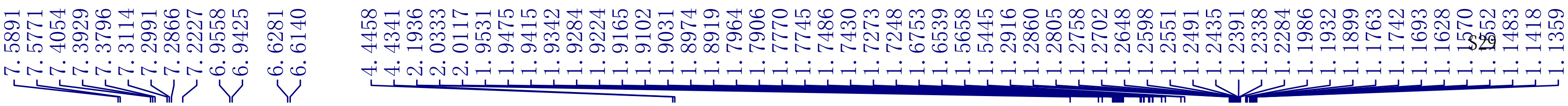


[1]H NMR (600 MHz, $CDCl_3$) δ 7.61 – 7.58 (m, 1H), 7.11 (d, J = 3.5 Hz, 1H), 6.95 (d, J = 8.5 Hz, 2H), 6.61 (d, J = 8.4 Hz, 2H), 6.56 (dd, J = 3.5, 1.7 Hz, 1H), 4.59 (d, J = 7.5 Hz, 1H), 4.02 (s, 1H), 2.19 (s, 3H), 2.05 (d, J = 13.0 Hz, 1H), 1.93 (m, 1H), 1.81 – 1.73 (m, 2H), 1.67 (d, J = 12.7 Hz, 1H), 1.54 (d, J = 12.7 Hz, 1H), 1.29 – 1.23 (m, 2H), 1.21 – 1.13 (m, 3H).

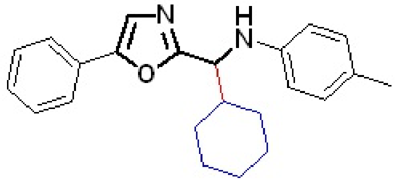


3ha

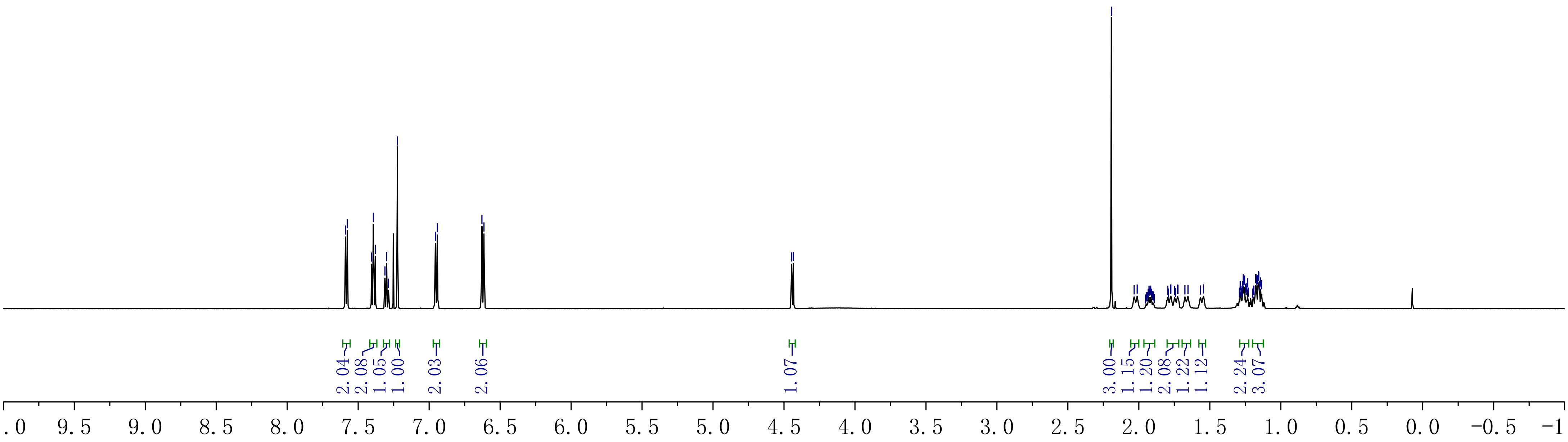


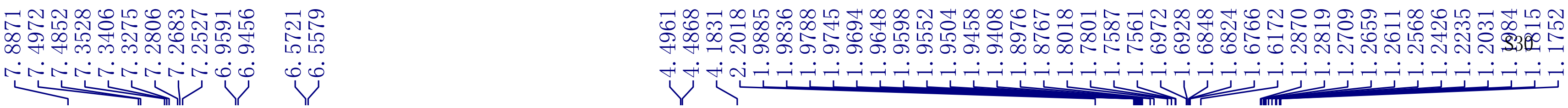


[1]H NMR (600 MHz, $CDCl_3$) δ 7.58 (d, J = 7.2 Hz, 2H), 7.39 (t, J = 7.7 Hz, 2H), 7.30 (t, J = 7.4 Hz, 1H), 7.22 (s, 1H), 6.95 (d, J = 8.0 Hz, 2H), 6.62 (d, J = 8.5 Hz, 2H), 4.44 (d, J = 7.0 Hz, 1H), 2.19 (s, 3H), 2.02 (d, J = 13.0 Hz, 1H), 1.96 – 1.89 (m, 1H), 1.82 – 1.72 (m, 2H), 1.66 (d, J = 12.8 Hz, 1H), 1.56 (d, J = 12.8 Hz, 1H), 1.30 – 1.22 (m, 2H), 1.20 – 1.13 (m, 3H).

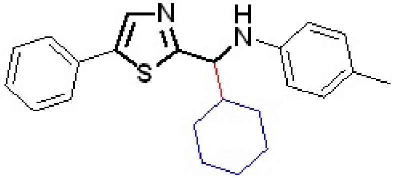


3ia

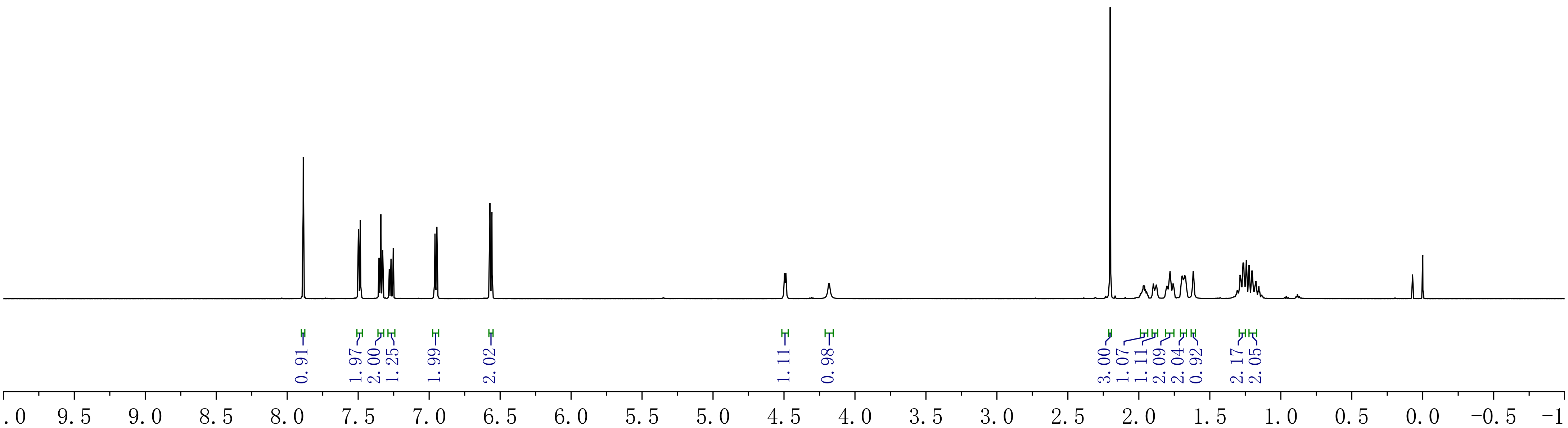


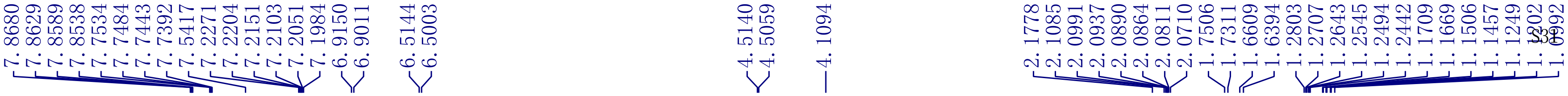


[1]H NMR (600 MHz, $CDCl_3$) δ 7.89 (s, 1H), 7.49 (d, *J* = 7.2 Hz, 2H), 7.34 (t, *J* = 7.6 Hz, 2H), 7.29 – 7.24 (m, 1H), 6.95 (d, *J* = 8.1 Hz, 2H), 6.56 (d, *J* = 8.5 Hz, 2H), 4.49 (d, *J* = 5.6 Hz, 1H), 4.18 (s, 1H), 2.20 (s, 3H), 1.99 – 1.93 (m, 1H), 1.89 (d, *J* = 12.5 Hz, 1H), 1.81 – 1.75 (m, 2H), 1.71 – 1.66 (m, 2H), 1.62 (s, 1H), 1.30 – 1.25 (m, 2H), 1.22 – 1.16 (m, 2H).

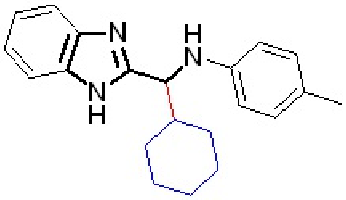


3ja

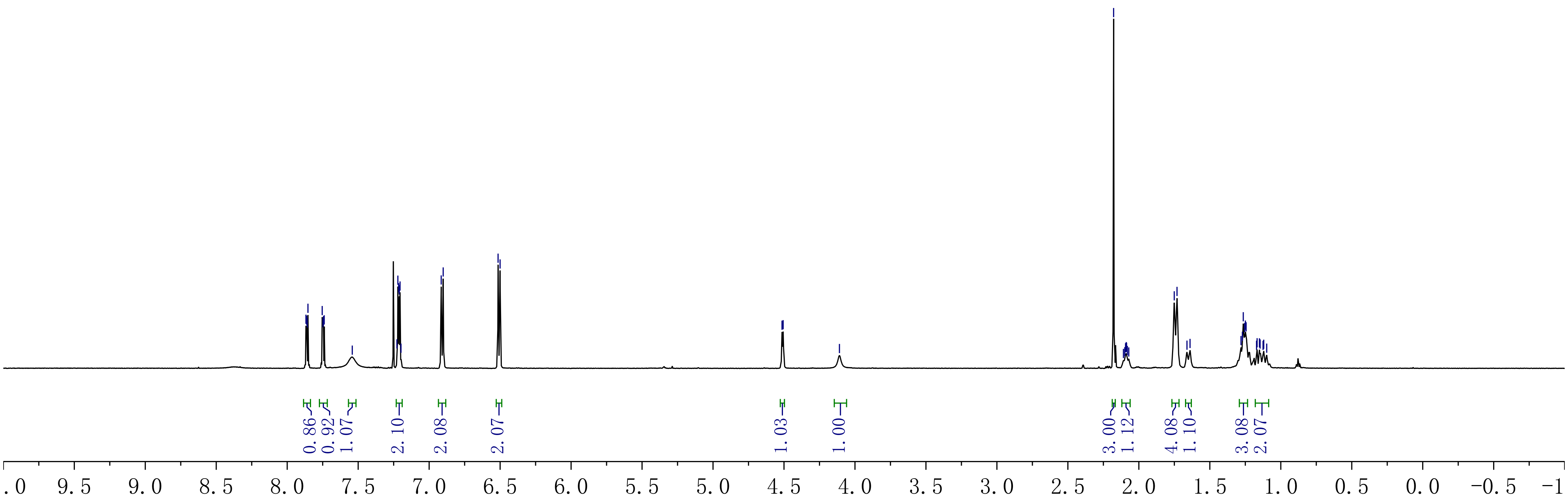




[1]H NMR (600 MHz, $CDCl_3$) δ 7.86 (dd, J = 5.4, 3.1 Hz, 1H), 7.75 (dd, J = 5.5, 3.0 Hz, 1H), 7.54 (s, 1H), 7.23 – 7.19 (m, 2H), 6.91 (d, J = 8.4 Hz, 2H), 6.51 (d, J = 8.4 Hz, 2H), 4.51 (d, J = 4.8 Hz, 1H), 4.11 (s, 1H), 2.18 (s, 3H), 2.12 – 2.06 (m, 1H), 1.74 (d, J = 11.7 Hz, 4H), 1.65 (d, J = 12.9 Hz, 1H), 1.30 – 1.23 (m, 3H), 1.18 – 1.08 (m, 2H).



3ka



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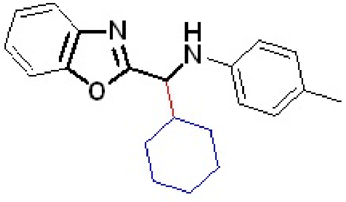
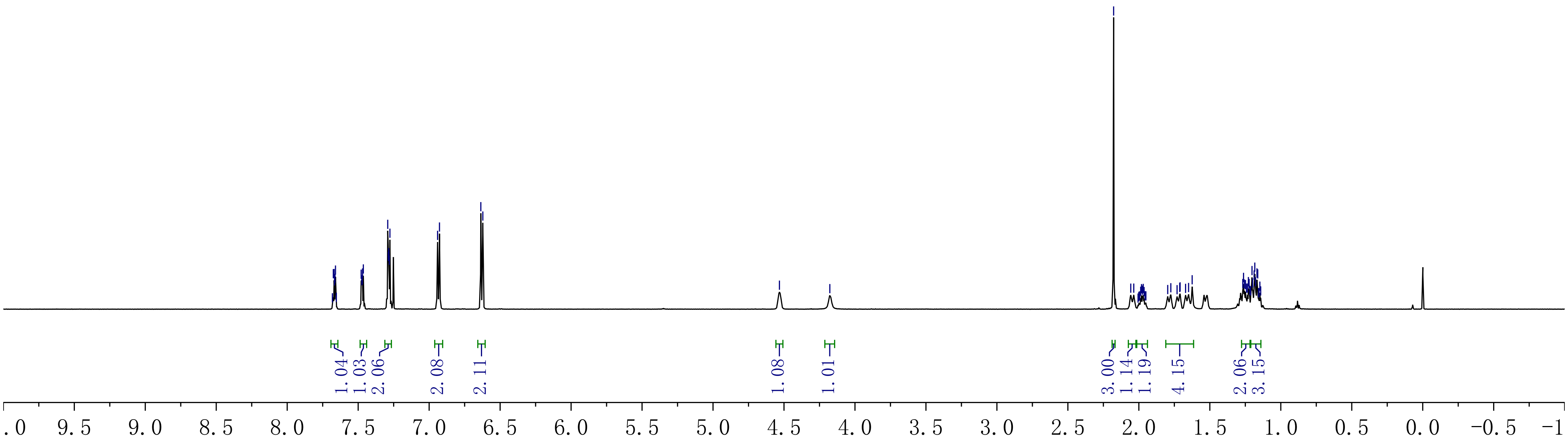
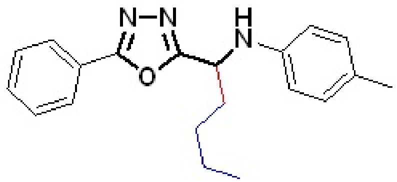
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.69 – 7.64 (m, 1H), 7.49 – 7.44 (m, 1H), 7.31 – 7.27 (m, 2H), 6.93 (d, J = 8.1 Hz, 2H), 6.63 (d, J = 8.5 Hz, 2H), 4.53 (s, 1H), 4.18 (s, 1H), 2.18 (s, 3H), 2.05 (d, J = 12.7 Hz, 1H), 2.01 – 1.94 (m, 1H), 1.81 – 1.61 (m, 4H), 1.28 – 1.22 (m, 2H), 1.21 – 1.14 (m, 3H).

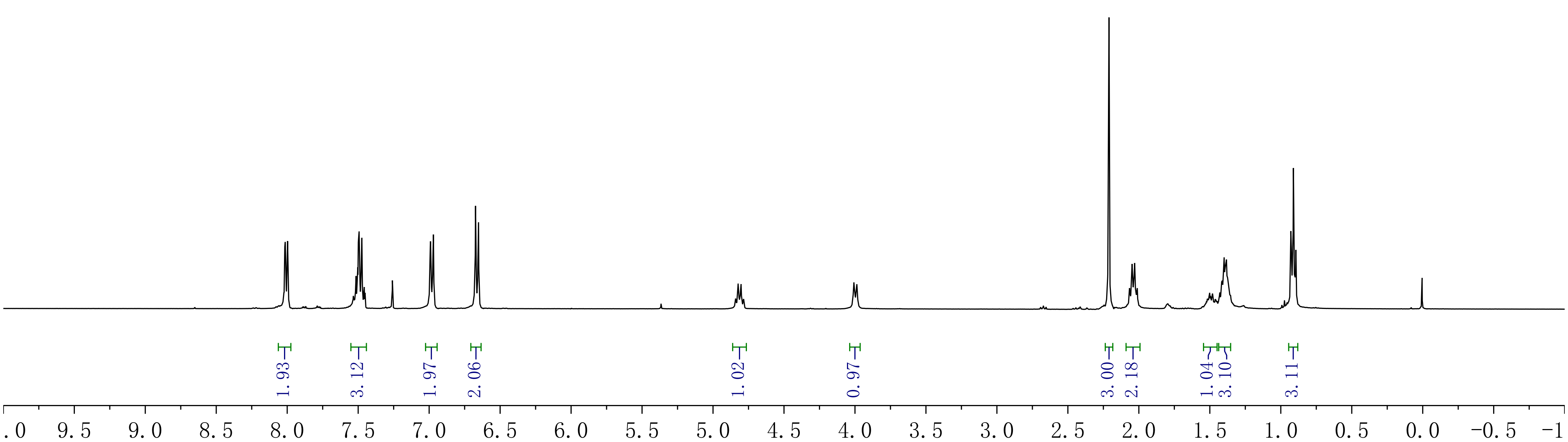


3la




N–N
H
N
O

3ab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7.9930
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7.5084
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7.4799
7.4771
7.4737
7.4616
7.4575
7.4516
6.9903
6.9701
6.6732
6.6520
4.8411
4.8231
4.8028
4.7847
4.0075
3.9868
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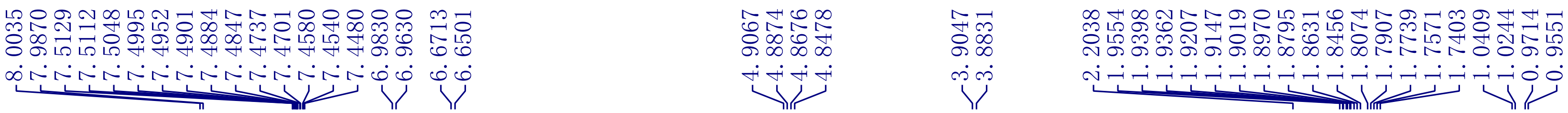
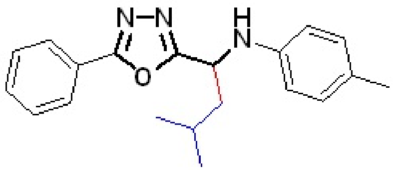
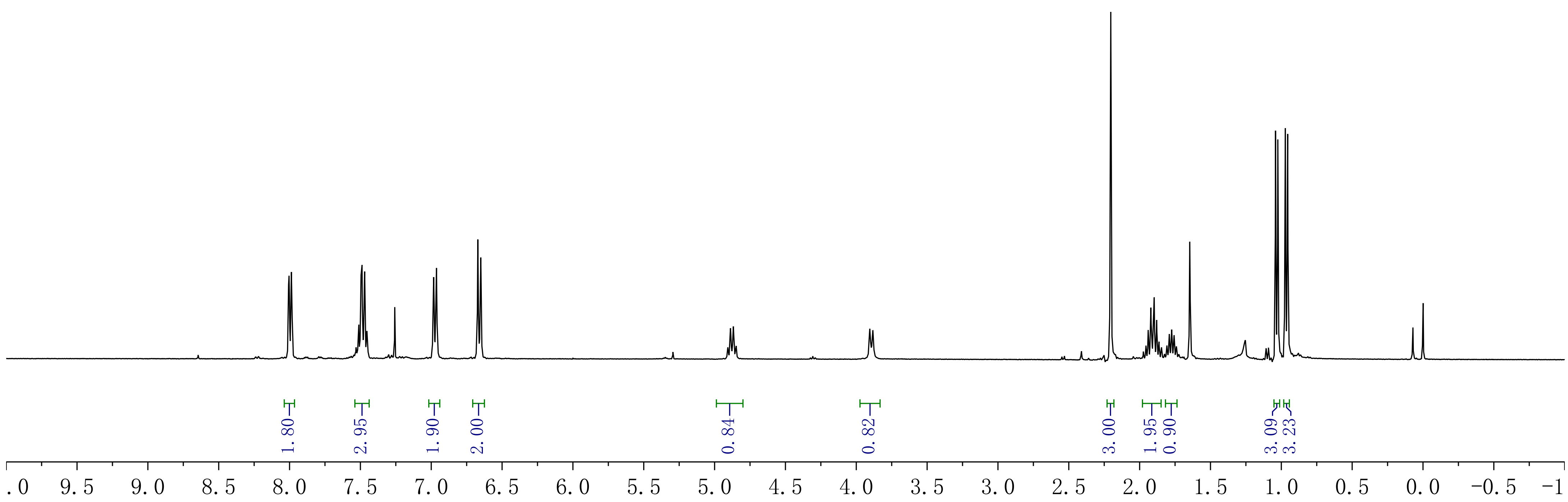
2.0660
2.0474
2.0293
2.0112
1.5173
1.5113
1.5011
1.4954
1.4814
1.4620
1.4568
1.4298
1.4165
1.4122
1.3991
1.3937
1.3891
1.3854
1.3768
1.3716
1.3535
0.9287
0.9112
0.8933
1.93
3.12
1.97
2.06
1.02
0.97
3.00
2.18
1.04
3.10
3.11
.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5 -1



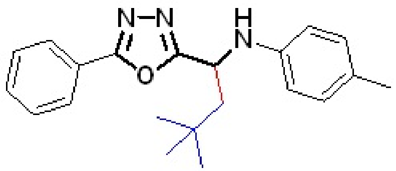
[1]H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 6.6 Hz, 2H), 7.53 – 7.44 (m, 3H), 6.97 (d, *J* = 8.0 Hz, 2H), 6.66 (d, *J* = 8.5 Hz, 2H), 4.95 – 4.83 (m, 1H), 3.89 (d, *J* = 8.6 Hz, 1H), 2.20 (s, 3H), 1.98 – 1.85 (m, 2H), 1.81 – 1.73 (m, 1H), 1.03 (d, *J* = 6.6 Hz, 3H), 0.96 (d, *J* = 6.5 Hz, 3H).



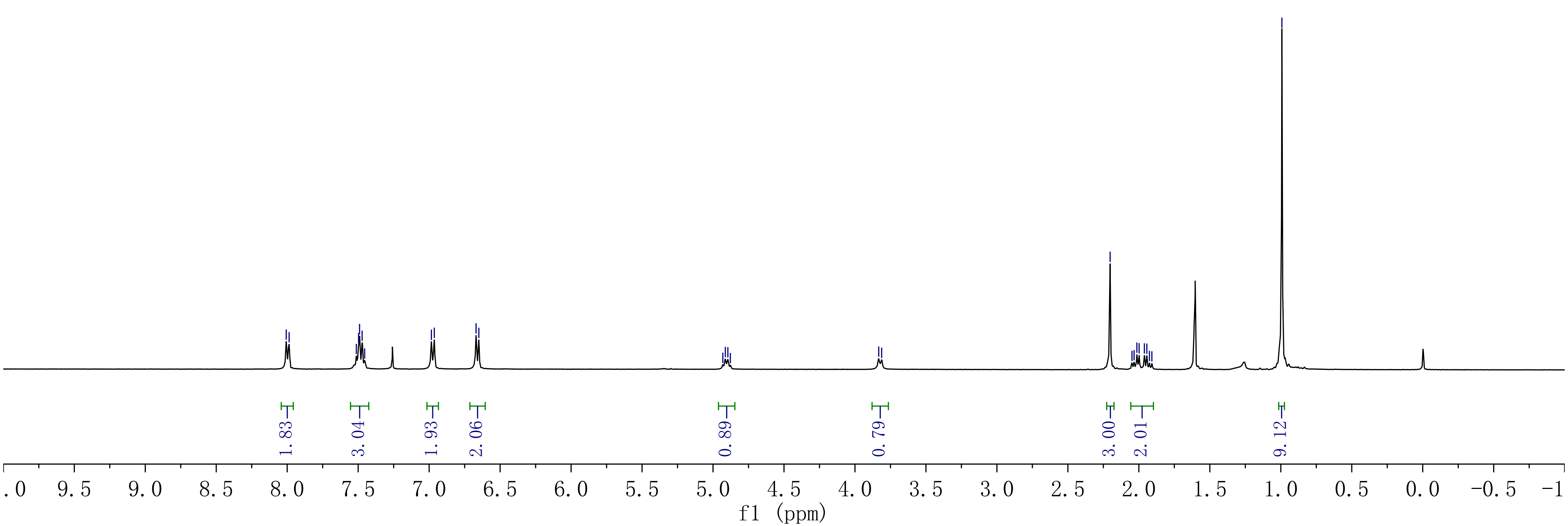
3ac



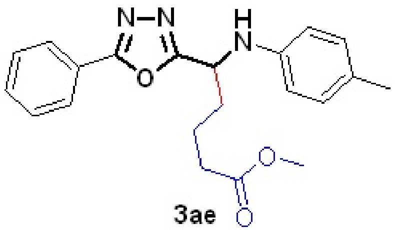
[1]H NMR (400 MHz, $CDCl_3$) δ 8.00 (d, *J* = 8.1 Hz, 2H), 7.55 – 7.42 (m, 3H), 6.97 (d, *J* = 8.1 Hz, 2H), 6.66 (d, *J* = 7.8 Hz, 2H), 4.95 – 4.86 (m, 1H), 3.82 (d, *J* = 8.2 Hz, 1H), 2.20 (s, 3H), 2.06 – 1.88 (m, 2H), 0.99 (s, 9H).



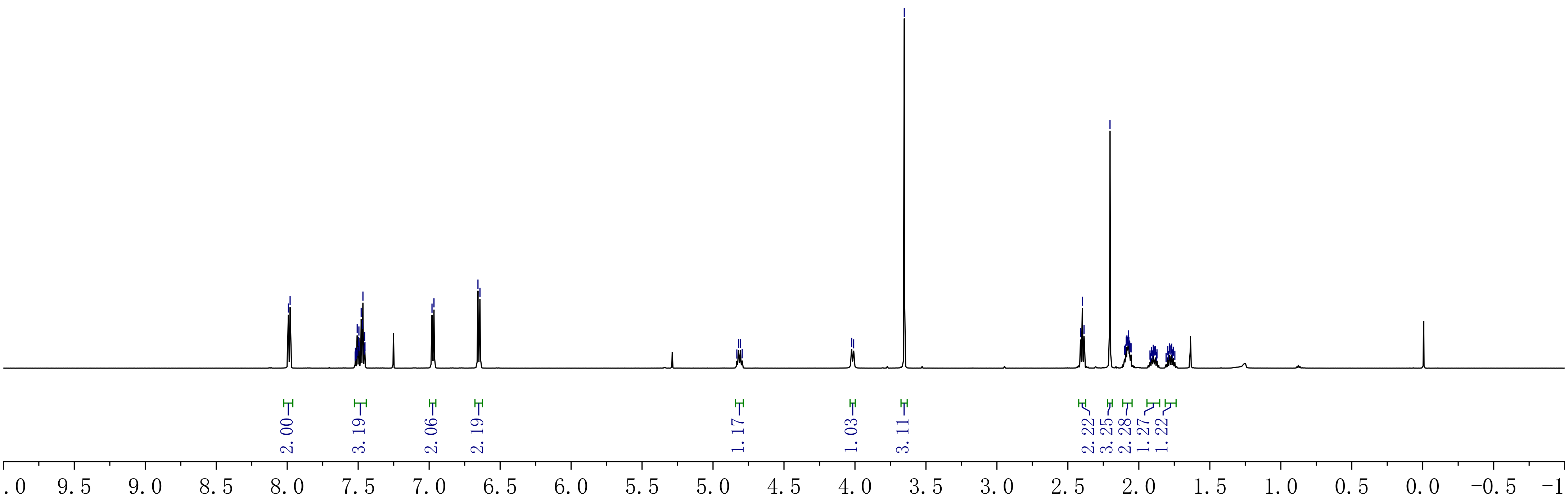
3ad



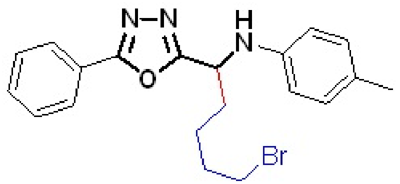
[1]H NMR (600 MHz, $CDCl_3$) δ 7.99 (d, J = 7.0 Hz, 2H), 7.53 – 7.44 (m, 3H), 6.97 (d, J = 8.4 Hz, 2H), 6.65 (d, J = 8.4 Hz, 2H), 4.81 (q, 7.3 Hz, 1H), 4.02 (d, J = 8.6 Hz, 1H), 3.65 (s, 3H), 2.40 (t, J = 7.3 Hz, 2H), 2.20 (s, 3H), 2.13 – 2.03 (m, 2H), 1.93 – 1.85 (m, 1H), 1.82 – 1.74 (m, 1H).



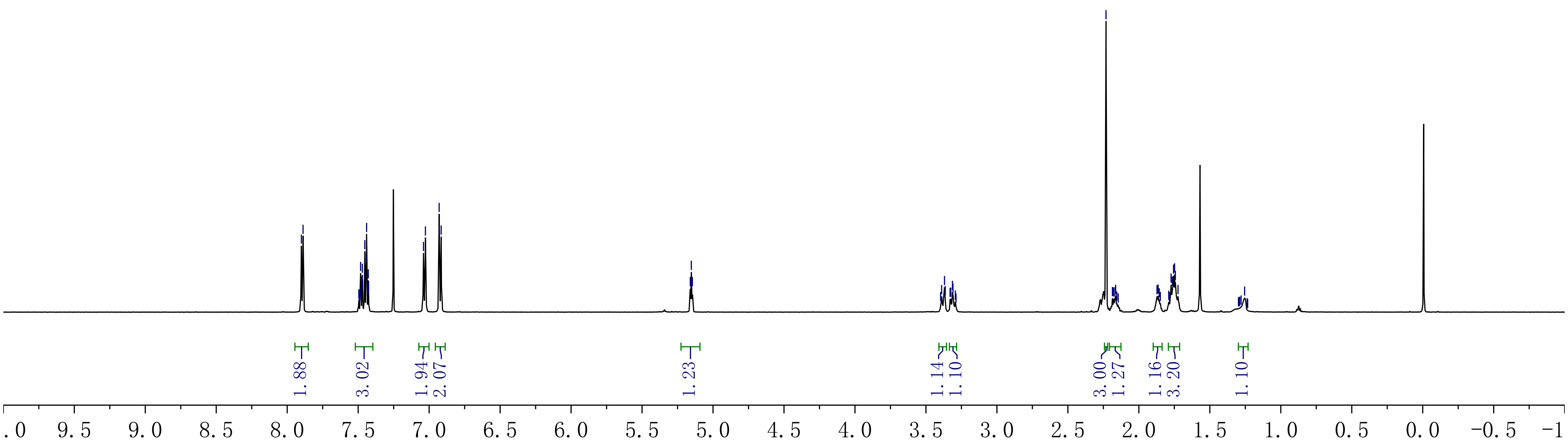
3ae

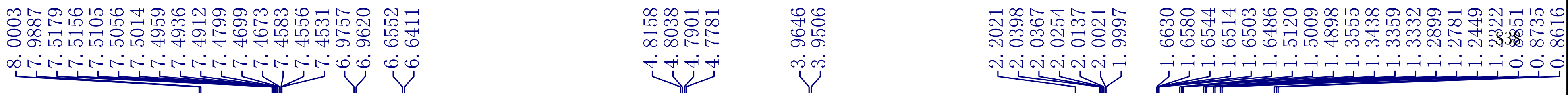


[1]H NMR (600 MHz, $CDCl_3$) δ 7.89 (d, J = 7.0 Hz, 2H), 7.51 – 7.40 (m, 3H), 7.03 (d, J = 8.2 Hz, 2H), 6.92 (d, J = 8.6 Hz, 2H), 5.15 (t, J = 4.4 Hz, 1H), 3.41 – 3.36 (m, 1H), 3.33 – 3.28 (m, 1H), 2.23 (s, 3H), 2.20 – 2.13 (m, 1H), 1.89 – 1.84 (m, 1H), 1.79 – 1.72 (m, 3H), 1.30 – 1.23 (m, 1H).

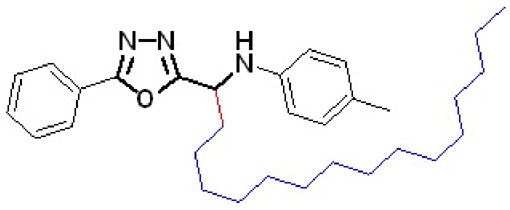


3af

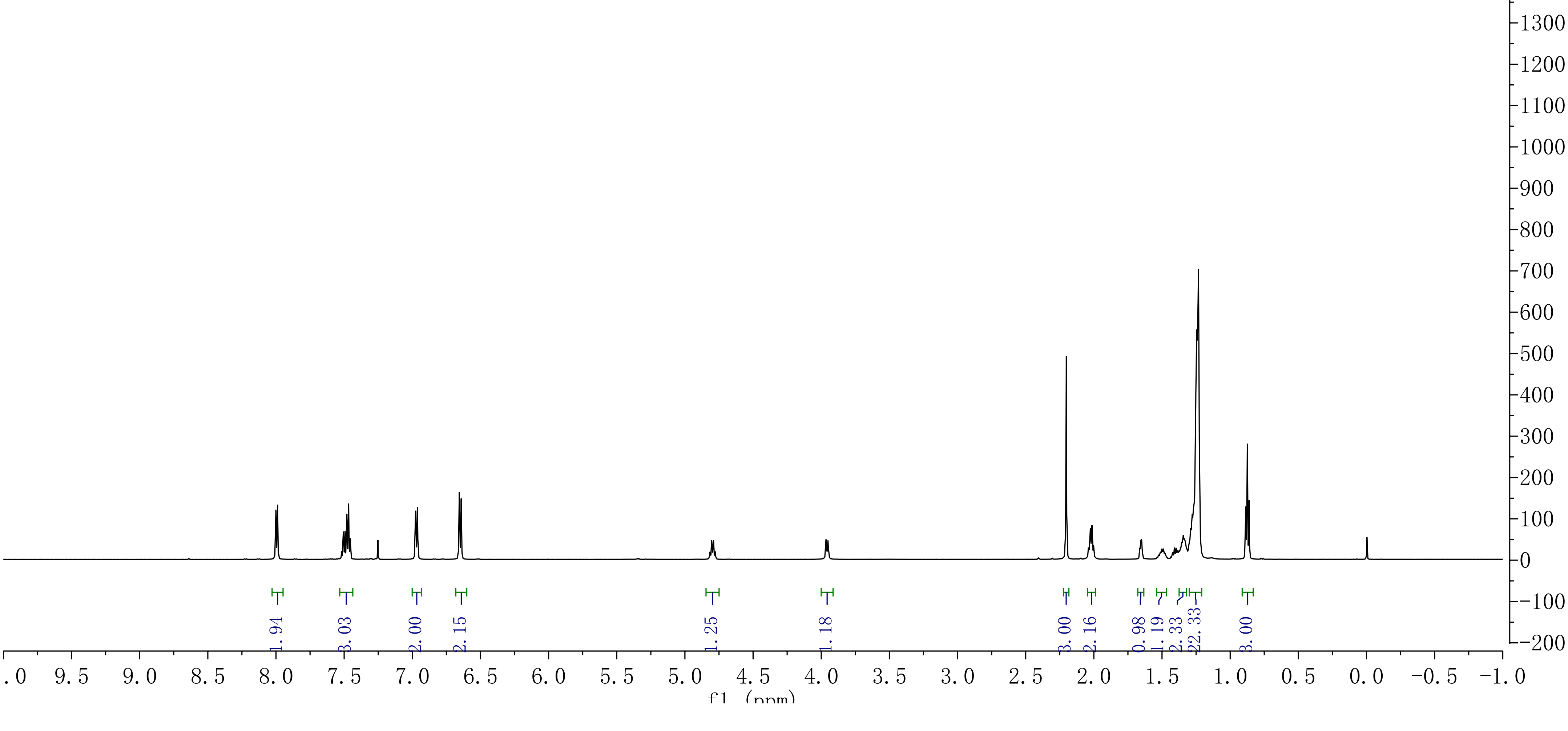




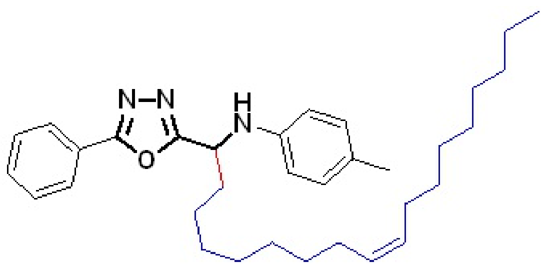
[1]H NMR (600 MHz, $CDCl_3$) δ 7.99 (d, J = 7.0 Hz, 2H), 7.53 – 7.44 (m, 3H), 6.97 (d, J = 8.2 Hz, 2H), 6.65 (d, J = 8.4 Hz, 2H), 4.85 – 4.75 (m, 1H), 3.96 (d, J = 8.4 Hz, 1H), 2.20 (s, 3H), 2.06 – 1.97 (m, 2H), 1.67 – 1.62 (m, 1H), 1.53 – 1.47 (m, 1H), 1.38 – 1.31 (m, 2H), 1.30 – 1.20 (m, 22H), 0.87 (t, J = 7.0 Hz, 3H).



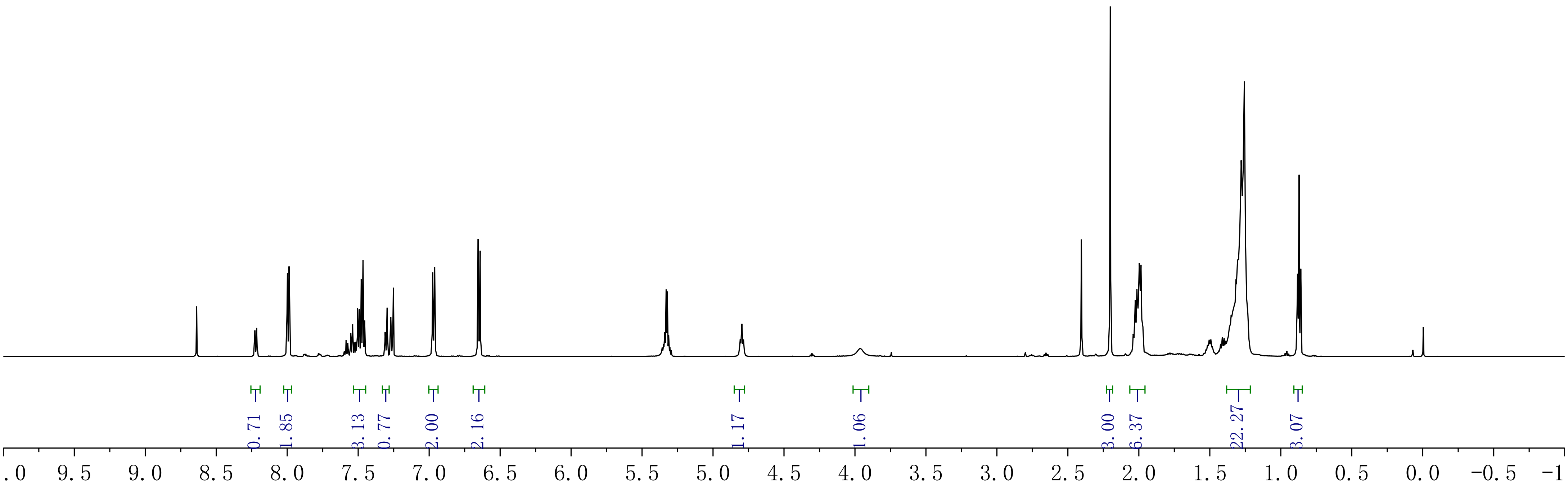
3ag



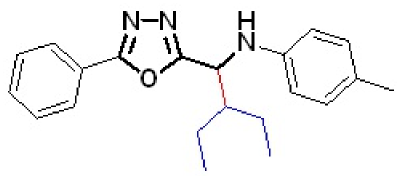
[1]H NMR (600 MHz, $CDCl_3$) δ 8.22 (d, *J* = 7.1 Hz, 1H), 7.99 (d, *J* = 7.0 Hz, 3H), 7.48 (m, 4H), 7.30 (d, *J* = 8.3 Hz, 1H), 6.97 (d, *J* = 8.3 Hz, 3H), 6.65 (d, *J* = 8.4 Hz, 3H), 4.80 (t, *J* = 6.9 Hz, 2H), 3.96 (s, 1H), 2.20 (s, 4H), 2.05 – 1.96 (m, 9H), 1.38 – 1.21 (m, 31H), 0.87 (t, *J* = 7.0 Hz, 4H).



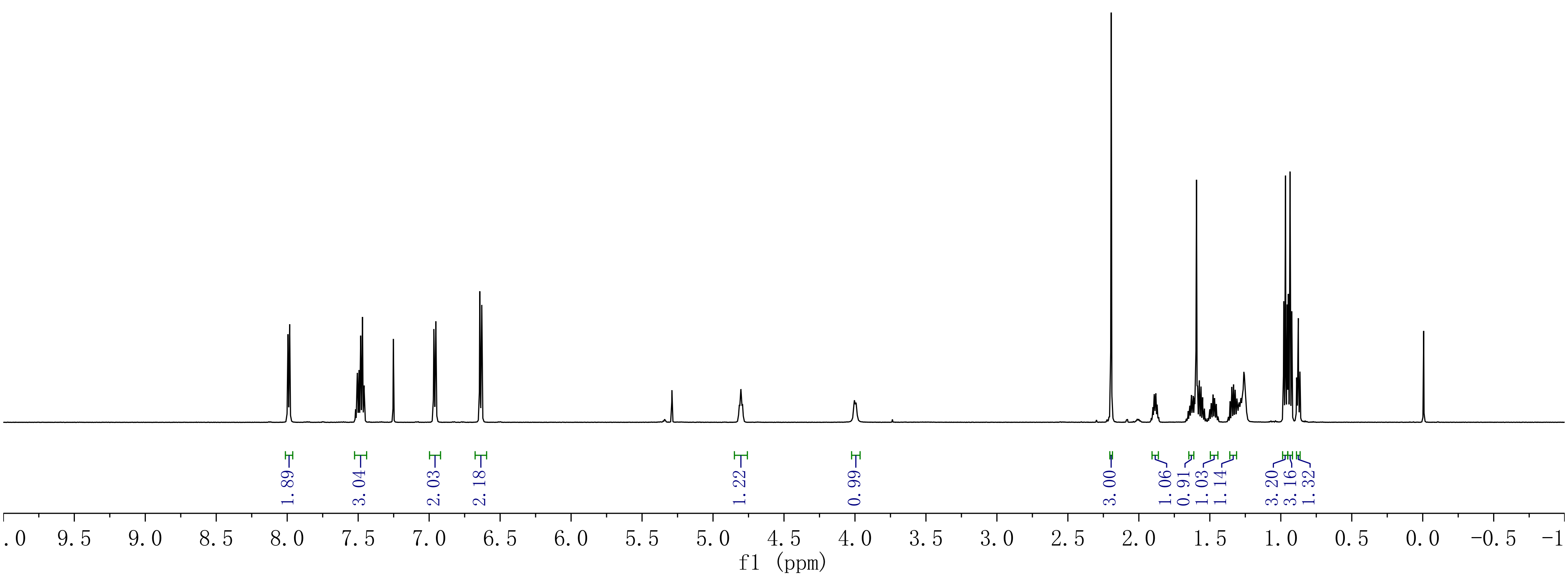
3ah



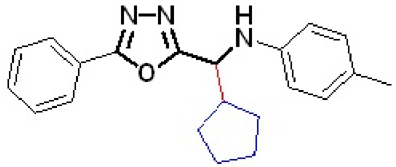
[1]H NMR (600 MHz, $CDCl_3$) δ 7.99 (d, J = 6.9 Hz, 2H), 7.52 – 7.44 (m, 3H), 6.96 (d, J = 8.3 Hz, 2H), 6.64 (d, J = 8.4 Hz, 2H), 4.80 (t, J = 6.7 Hz, 1H), 4.00 (d, J = 7.0 Hz, 1H), 2.19 (s, 3H), 1.91 – 1.86 (m, 1H), 1.64 – 1.61 (m, 1H), 1.50 – 1.44 (m, 1H), 1.36 – 1.31 (m, 1H), 0.97 (t, J = 7.4 Hz, 3H), 0.93 (t, J = 7.4 Hz, 3H), 0.88 (t, J = 7.0 Hz, 1H).



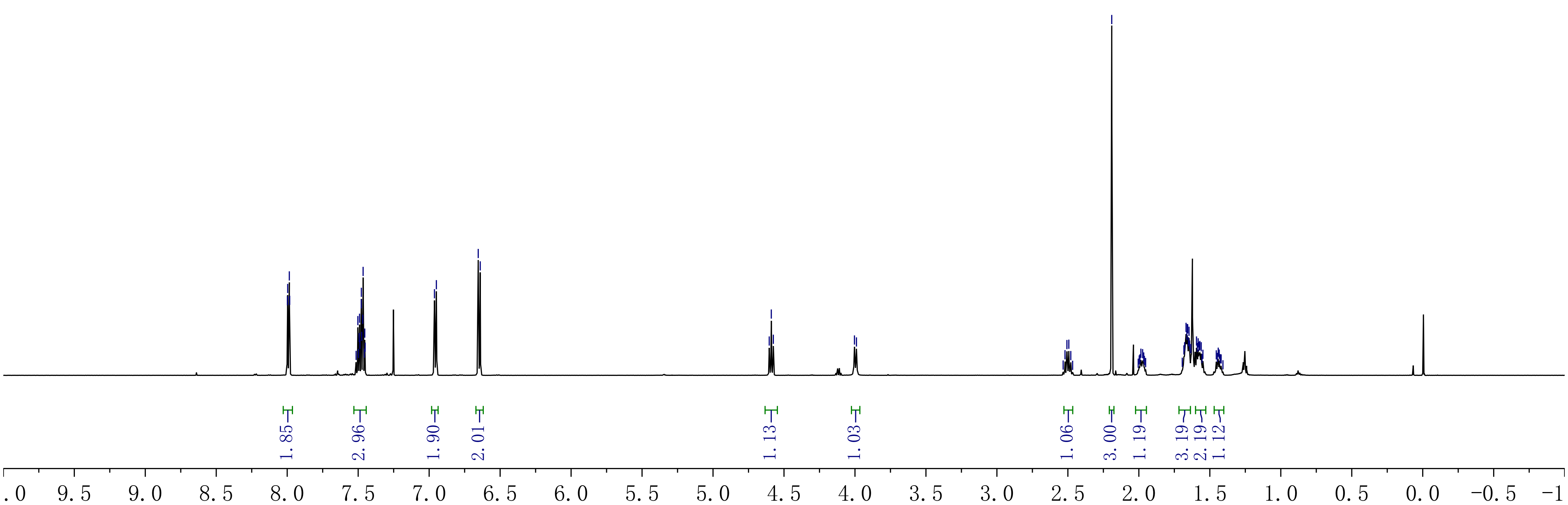
3ai



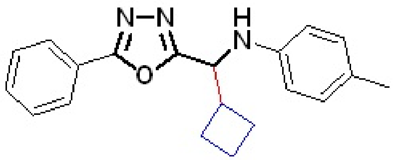
[1]H NMR (600 MHz, $CDCl_3$) δ 7.99 (m, 2H), 7.53 – 7.44 (m, 3H), 6.96 (d, *J* = 8.1 Hz, 2H), 6.65 (d, *J* = 8.5 Hz, 2H), 4.59 (t, *J* = 8.7 Hz, 1H), 4.00 (d, *J* = 8.5 Hz, 1H), 2.54 – 2.46 (m, 1H), 2.19 (s, 3H), 2.02 – 1.94 (m, 1H), 1.72 – 1.63 (m, 3H), 1.61 – 1.53 (m, 2H), 1.47 – 1.40 (m, 1H).



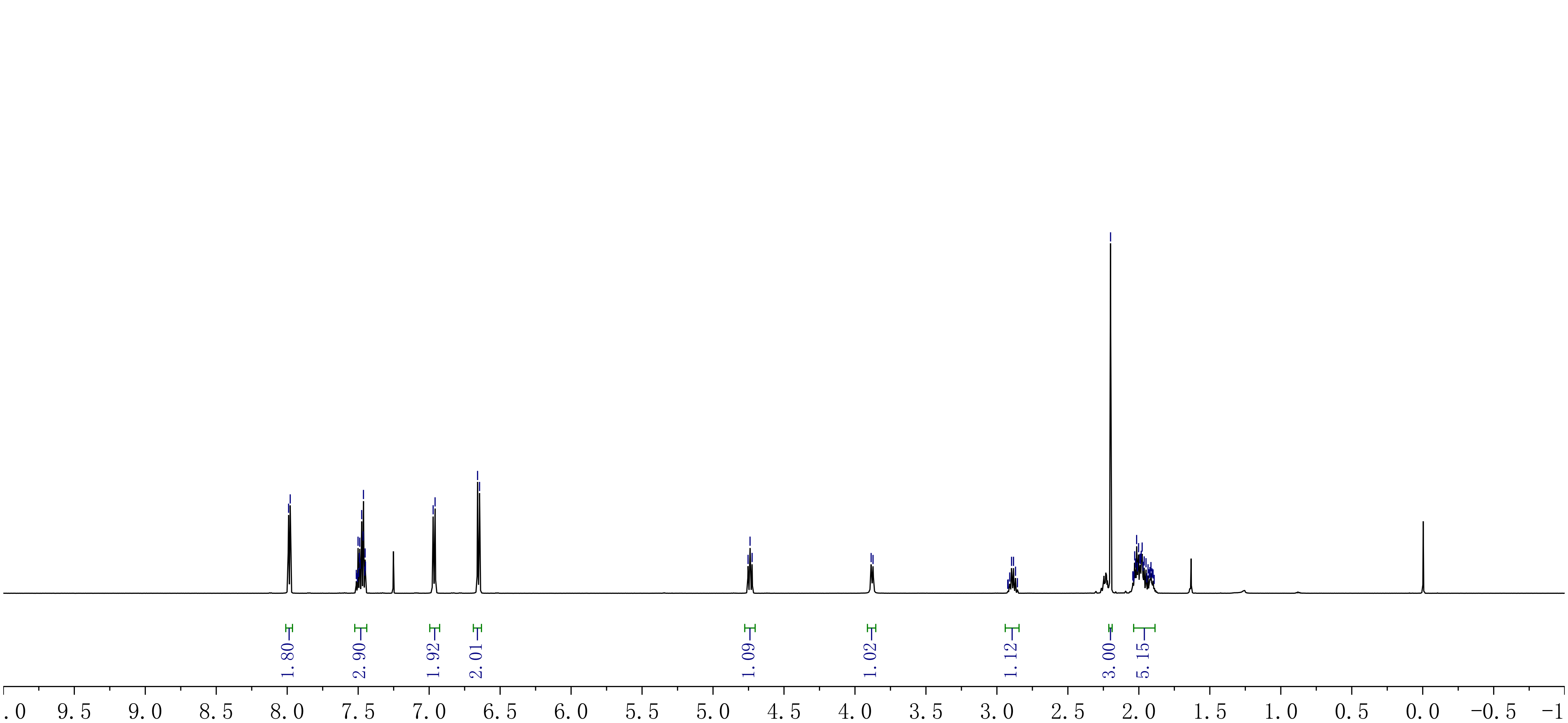
3aj



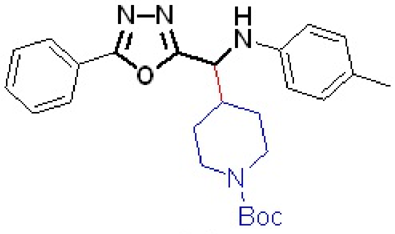
[1]H NMR (600 MHz, $CDCl_3$) δ 7.98 (d, J = 6.9 Hz, 2H), 7.52 – 7.44 (m, 3H), 6.97 (d, J = 8.2 Hz, 2H), 6.65 (d, J = 8.5 Hz, 2H), 4.74 (t, J = 8.6 Hz, 1H), 3.88 (d, J = 8.0 Hz, 1H), 2.94 – 2.84 (m, 1H), 2.20 (s, 3H), 2.04 – 1.89 (m, 5H).



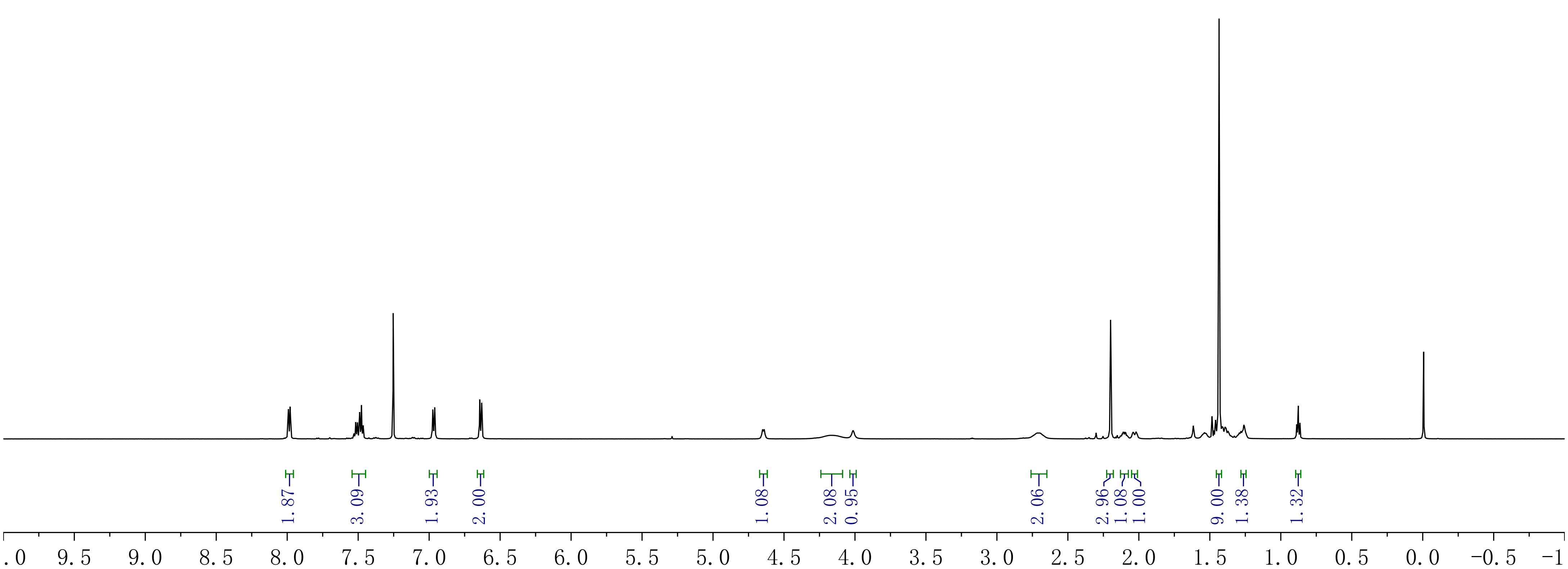
3ak



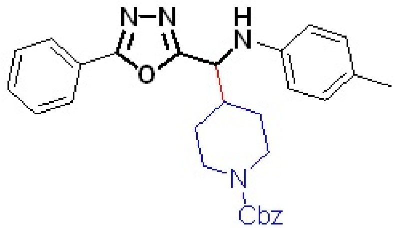
[1]H NMR (600 MHz, $CDCl_3$) δ 7.99 (d, J = 7.0 Hz, 2H), 7.54 – 7.45 (m, 3H), 6.97 (d, J = 8.1 Hz, 2H), 6.64 (d, J = 8.4 Hz, 2H), 4.65 (d, J = 6.9 Hz, 1H), 4.17 (s, 2H), 4.01 (s, 1H), 2.70 (s, 2H), 2.20 (s, 3H), 2.13 – 2.07 (m, 1H), 2.03 (d, J = 12.6 Hz, 1H), 1.43 (s, 9H), 1.28 – 1.24 (m, 1H), 0.88 (t, J = 7.0 Hz, 1H).



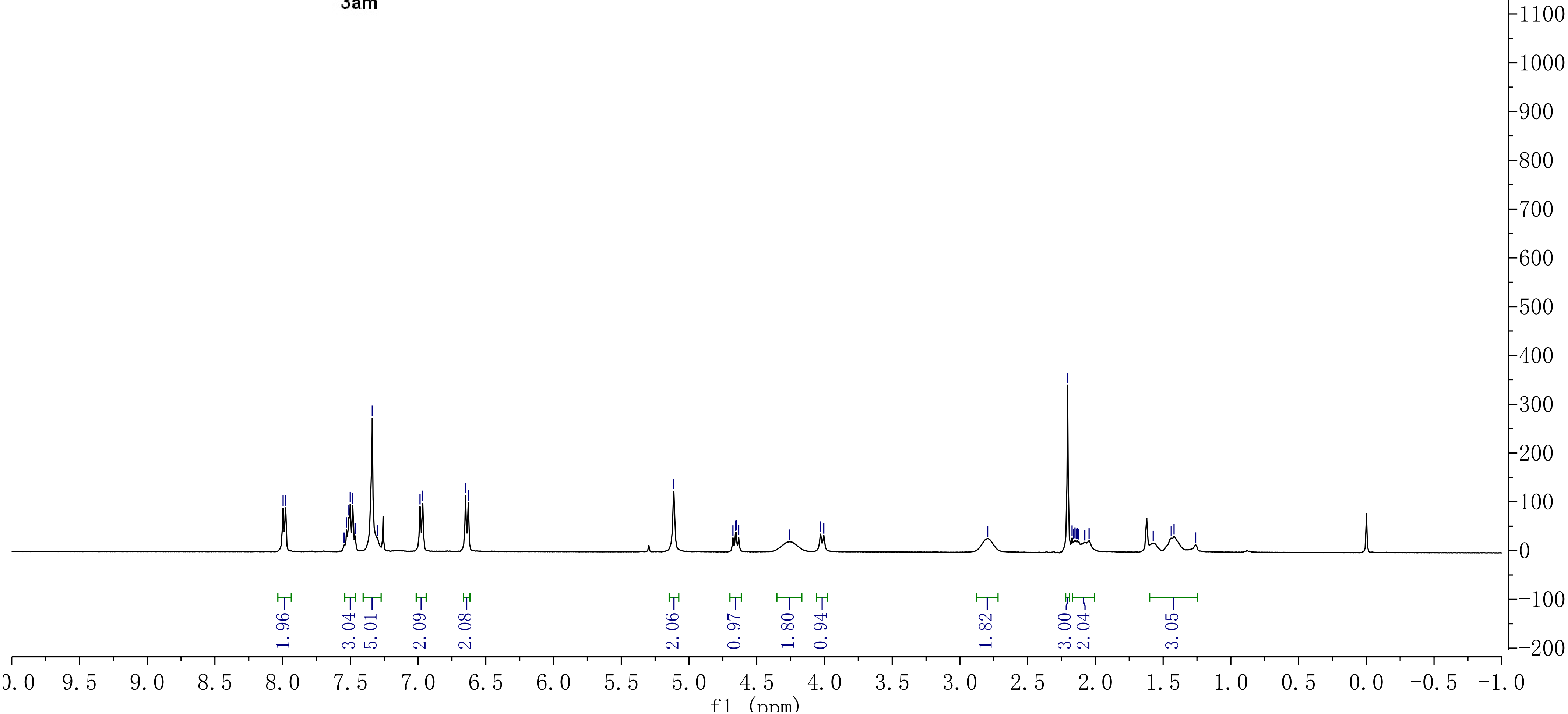
3al



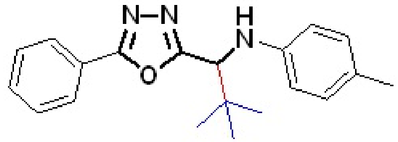
[1]H NMR (400 MHz, $CDCl_3$) δ 7.99 (d, J = 6.8 Hz, 2H), 7.54 – 7.46 (m, 3H), 7.41 – 7.27 (m, 5H), 6.98 (d, J = 8.1 Hz, 2H), 6.64 (d, J = 8.3 Hz, 2H), 5.11 (s, 2H), 4.65 (dd, J = 9.5, 7.6 Hz, 1H), 4.26 (s, 2H), 4.02 (d, J = 9.8 Hz, 1H), 2.80 (s, 2H), 2.21 (s, 3H), 2.17 – 2.01 (m, 2H), 1.59 – 1.25 (m, 3H).



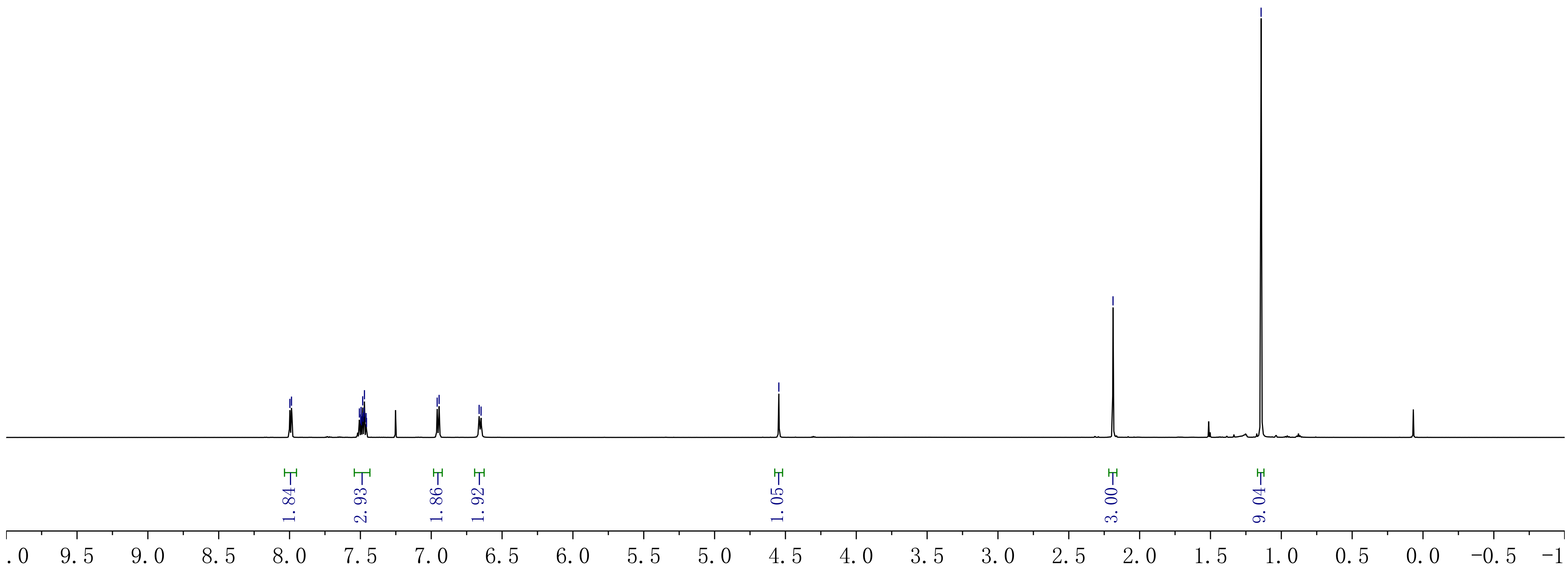
3am

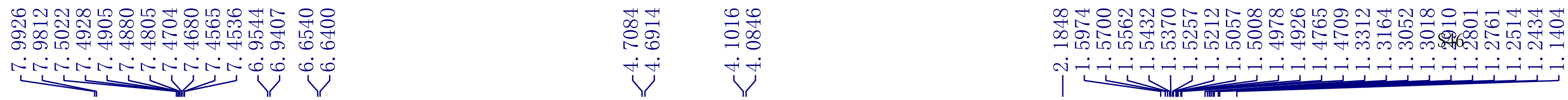


[1]H NMR (600 MHz, $CDCl_3$) δ 7.99 (d, *J* = 6.9 Hz, 2H), 7.54 – 7.44 (m, 3H), 6.95 (d, *J* = 8.2 Hz, 2H), 6.66 (d, *J* = 8.2 Hz, 2H), 4.55 (s, 1H), 2.19 (s, 3H), 1.14 (s, 9H).

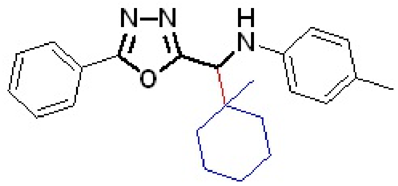


3an

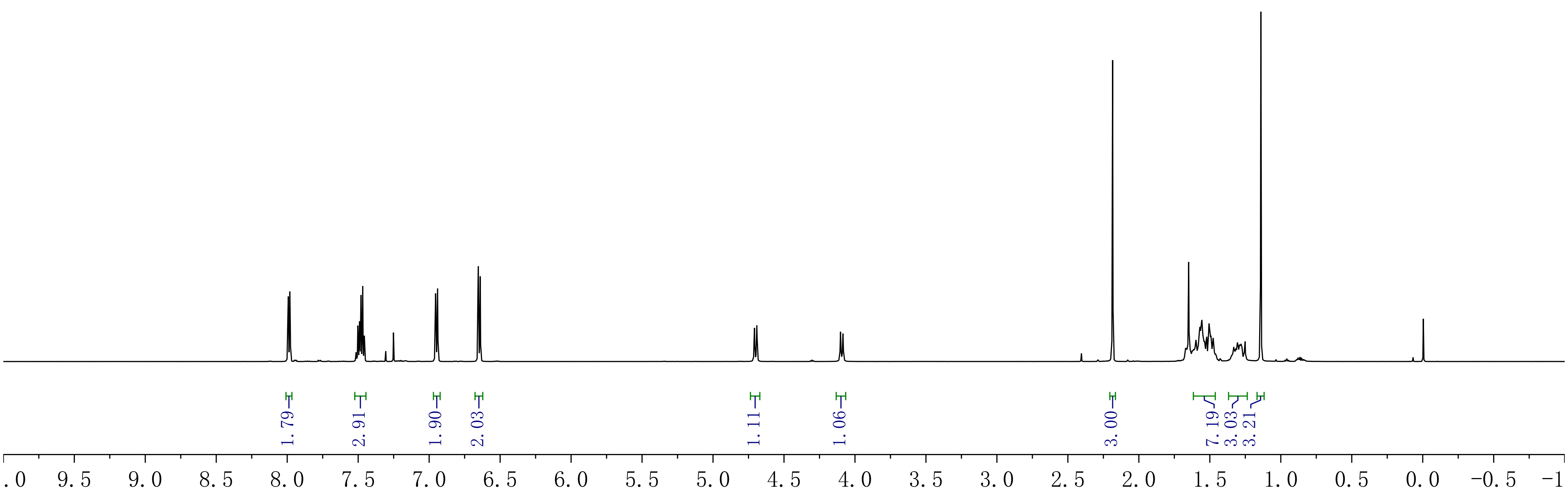


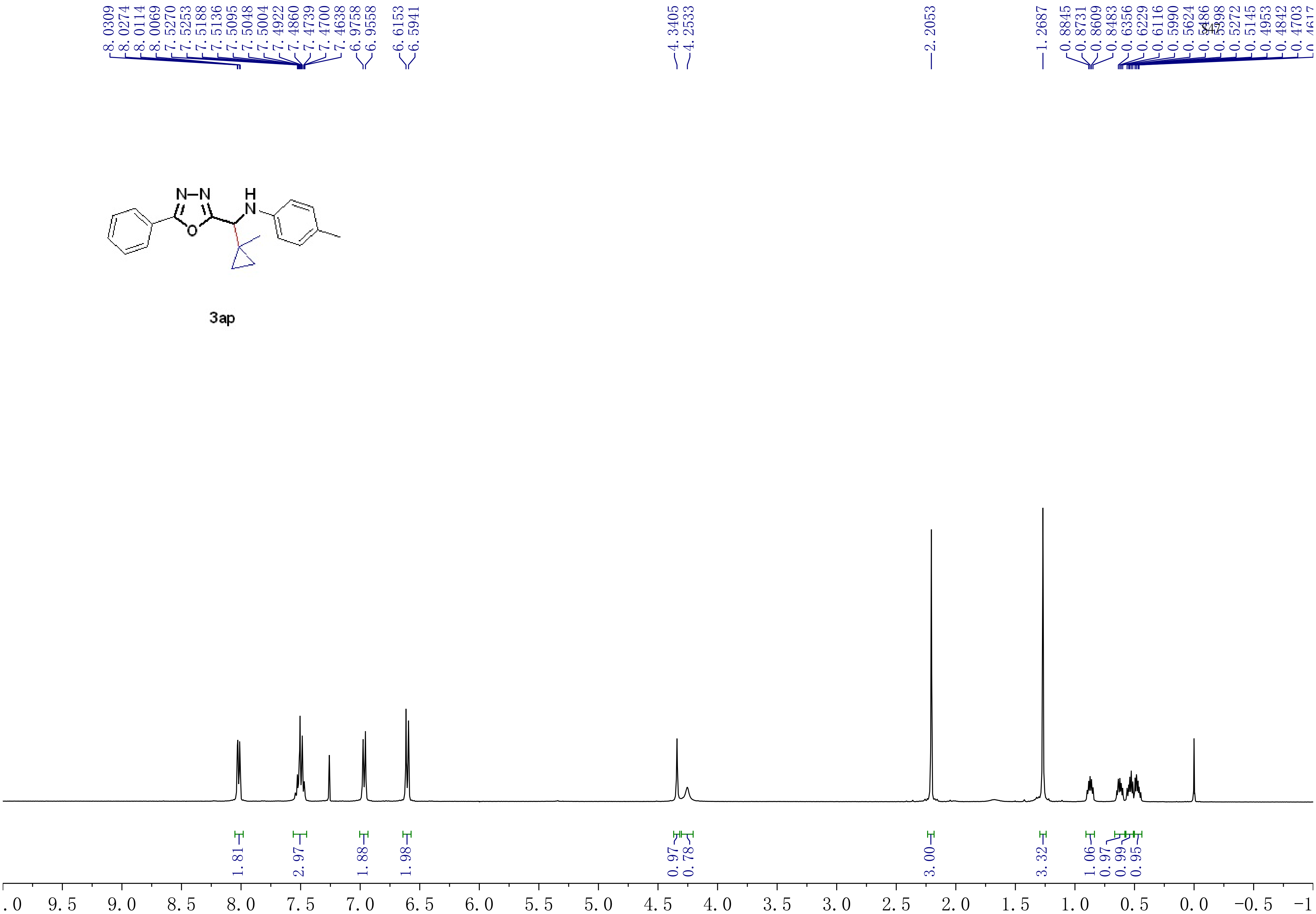


[1]H NMR (600 MHz, $CDCl_3$) δ 7.99 (d, J = 6.8 Hz, 2H), 7.52 – 7.44 (m, 3H), 6.95 (d, J = 8.3 Hz, 2H), 6.65 (d, J = 8.4 Hz, 2H), 4.70 (d, J = 10.2 Hz, 1H), 4.09 (d, J = 10.2 Hz, 1H), 2.18 (s, 3H), 1.61 – 1.46 (m, 7H), 1.36 – 1.23 (m, 3H), 1.14 (s, 3H).



3ao




N–N
H
N
O
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8.0069
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7.5253
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6.5941
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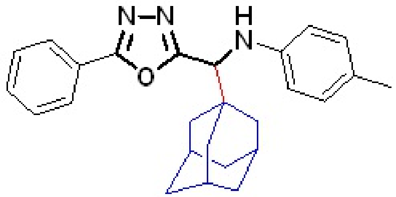
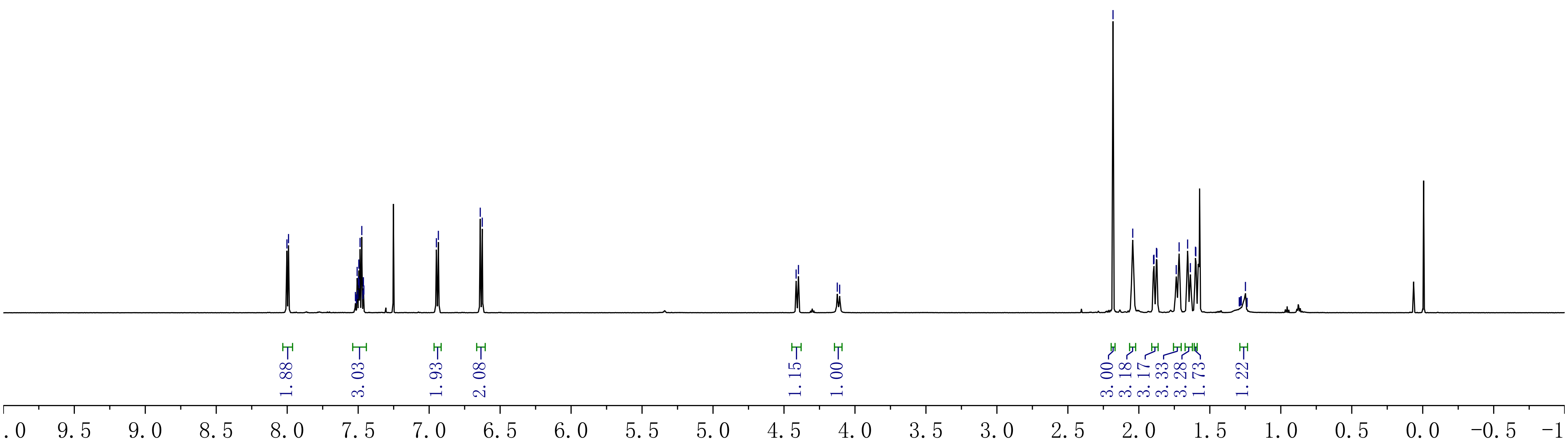
4.2533
2.2053
1.2687
0.8845
0.8731
0.8609
0.8483
0.6356
0.6229
0.6116
0.5990
0.5624
0.5486
0.5398
0.5272
0.5145
0.4953
0.4842
0.4703
1.81
2.97
1.88
1.98
0.97
0.78
3.00
3.32
1.06
0.97
0.99
0.95
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5

[1]H NMR (600 MHz, $CDCl_3$) δ 8.00 (d, *J* = 6.7 Hz, 2H), 7.54 – 7.45 (m, 3H), 6.94 (d, *J* = 8.3 Hz, 2H), 6.63 (d, *J* = 8.5 Hz, 2H), 4.41 (d, *J* = 9.9 Hz, 1H), 4.12 (d, *J* = 10.0 Hz, 1H), 2.18 (s, 3H), 2.04 (s, 3H), 1.88 (dd, *J* = 12.2, 1.8 Hz, 3H), 1.73 (d, *J* = 12.3 Hz, 3H), 1.65 (d, *J* = 11.7 Hz, 3H), 1.60 (d, *J* = 1.5 Hz, 2H), 1.29 – 1.24 (m, 1H).



3aq



9.1693
8.0374
8.0214
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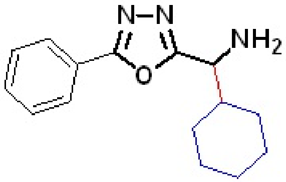
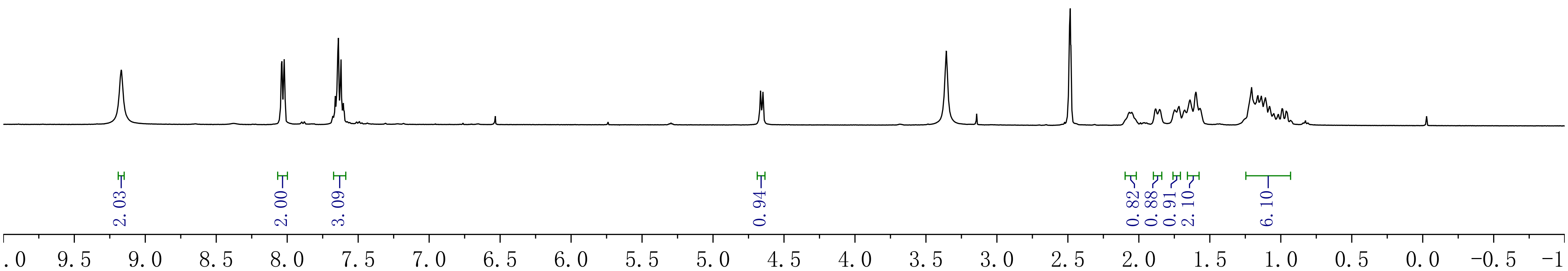
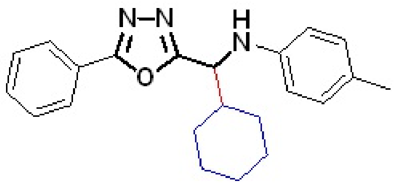
2.0497
1.8825
1.8527
1.7474
1.7179
1.6398
1.5982
1.2057
1.1609
1.1371
1.1081
1.0792
1.0491
1.0165
0.9913
0.9619

[1]H NMR (400 MHz, DMSO) δ 9.17 (s, 2H), 8.03 (d, *J* = 6.4 Hz, 2H), 7.68 – 7.59 (m, 3H), 4.66 (d, *J* = 6.7 Hz, 1H), 2.06 (d, *J* = 7.0 Hz, 1H), 1.87 (d, *J* = 11.9 Hz, 1H), 1.73 (d, *J* = 11.8 Hz, 1H), 1.62 (d, *J* = 16.6 Hz, 2H), 1.26 – 0.94 (m, 6H).

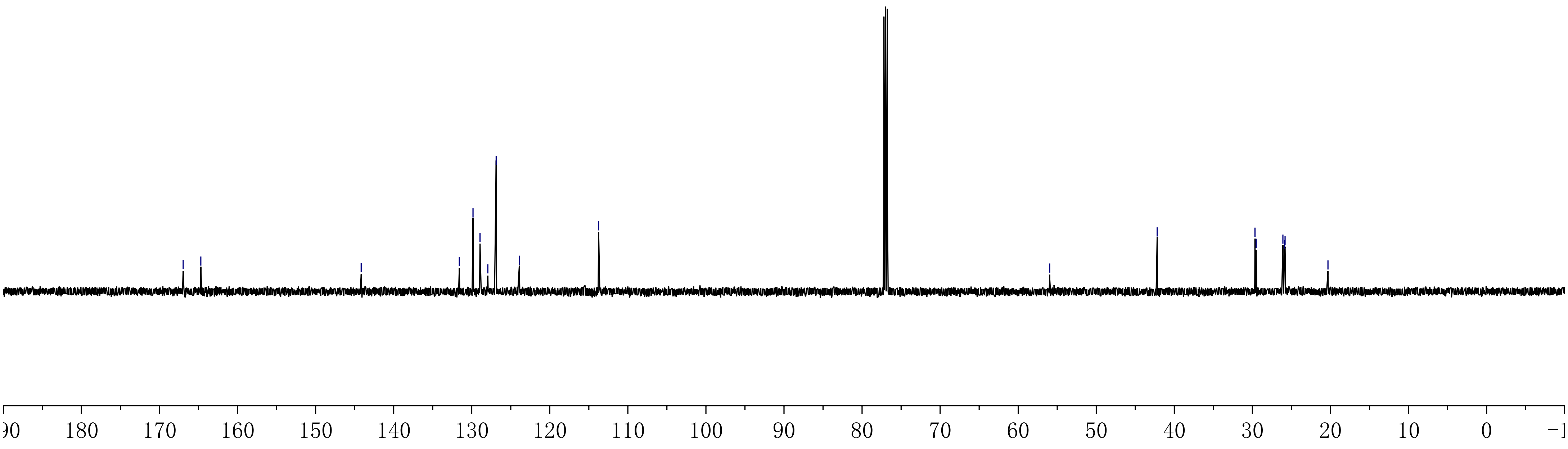




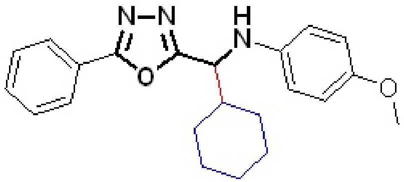
^{13}C NMR (151 MHz, $CDCl_3$) δ 166.96, 164.70, 144.16, 131.59, 129.84, 128.94, 127.94, 126.87, 123.91, 113.74, 55.96, 42.20, 29.67, 29.52, 26.09, 25.88, 25.82, 20.32.



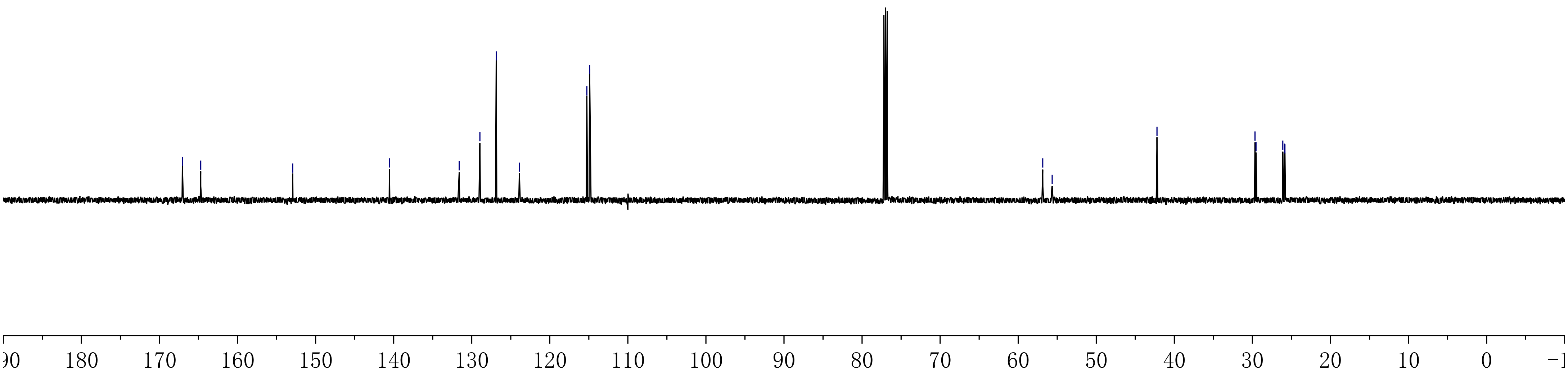
3aa



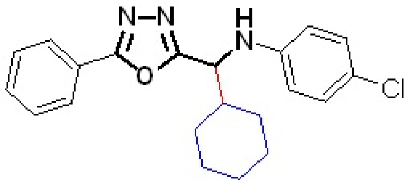
^{13}C NMR (151 MHz, $CDCl_3$) δ 167.06, 164.71, 152.93, 140.54, 131.61, 128.95, 126.86, 123.90, 115.25, 114.90, 56.86, 55.65, 42.22, 29.67, 29.54, 26.11, 25.89, 25.84.



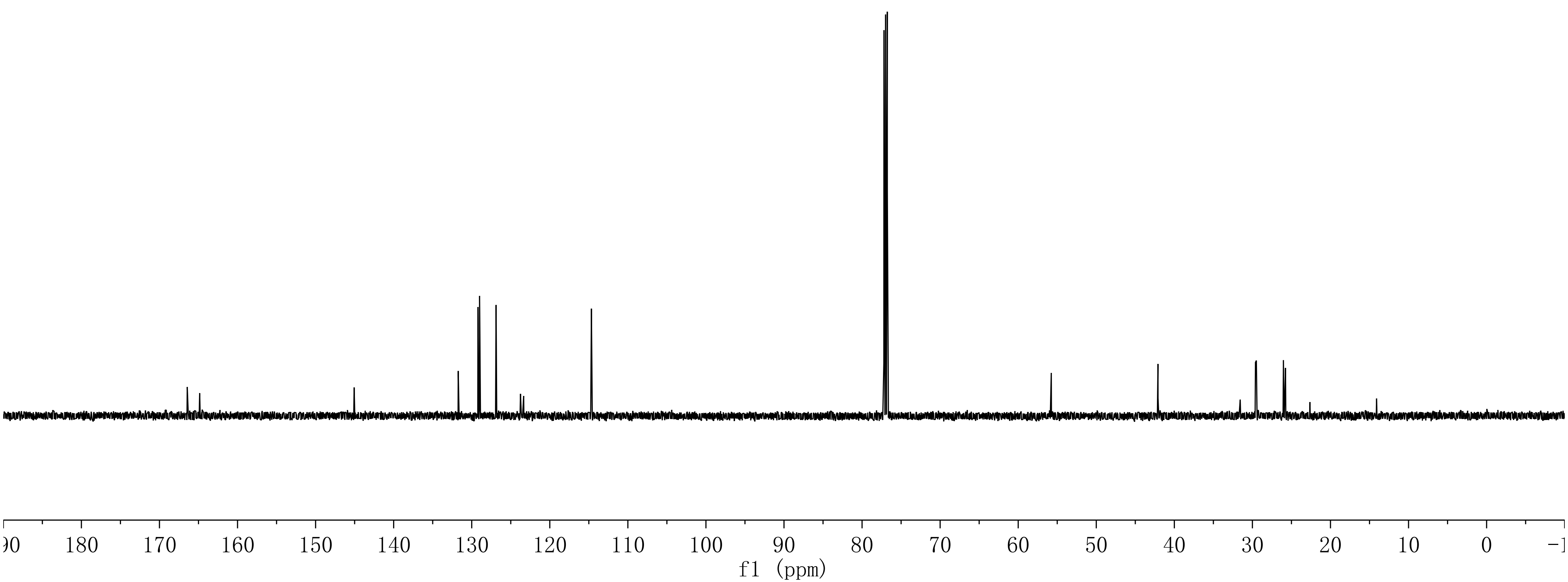
3ba



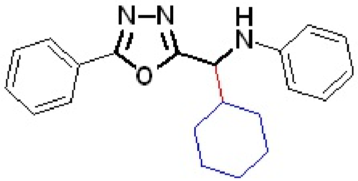
^{13}C NMR (151 MHz, $CDCl_3$) δ 166.44, 164.85, 145.06, 131.73, 129.21, 128.99, 126.88, 123.75, 123.35, 114.67, 55.76, 42.09, 29.61, 29.49, 26.03, 25.82, 25.76.



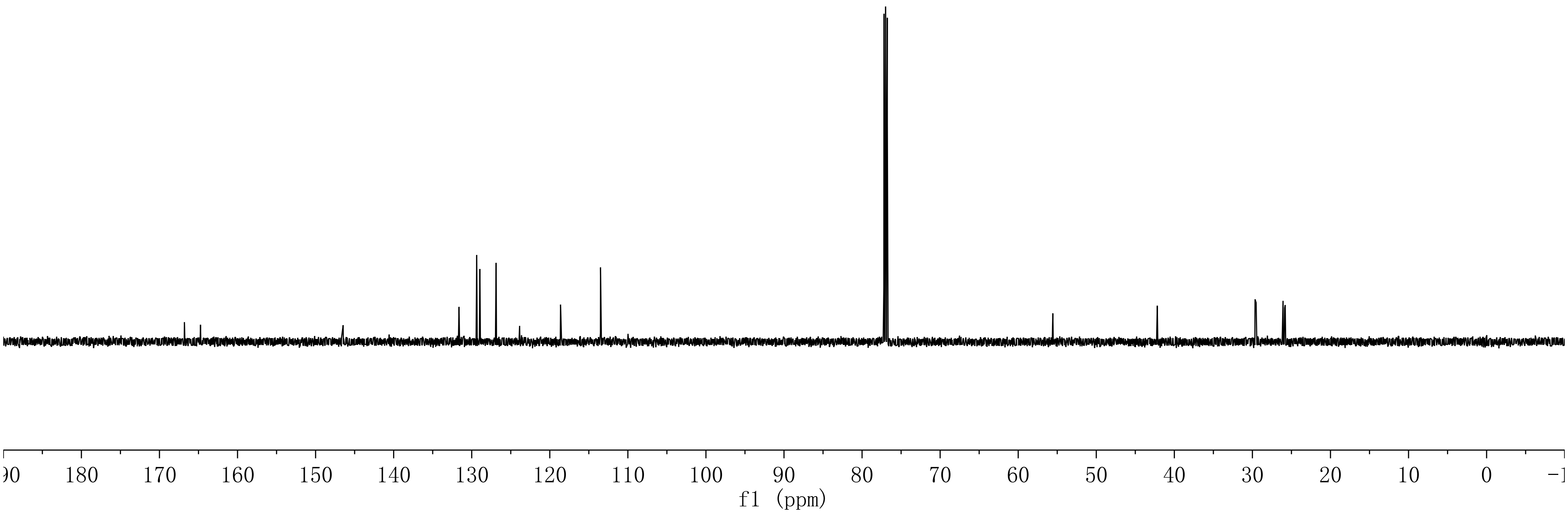
3ca



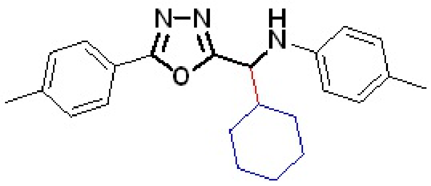
[13]C NMR (151 MHz, $CDCl_3$) δ 166.80, 164.75, 146.47, 131.63, 129.36, 128.96, 126.88, 123.87, 118.62, 113.50, 55.56, 42.19, 29.66, 29.52, 26.07, 25.87, 25.80.



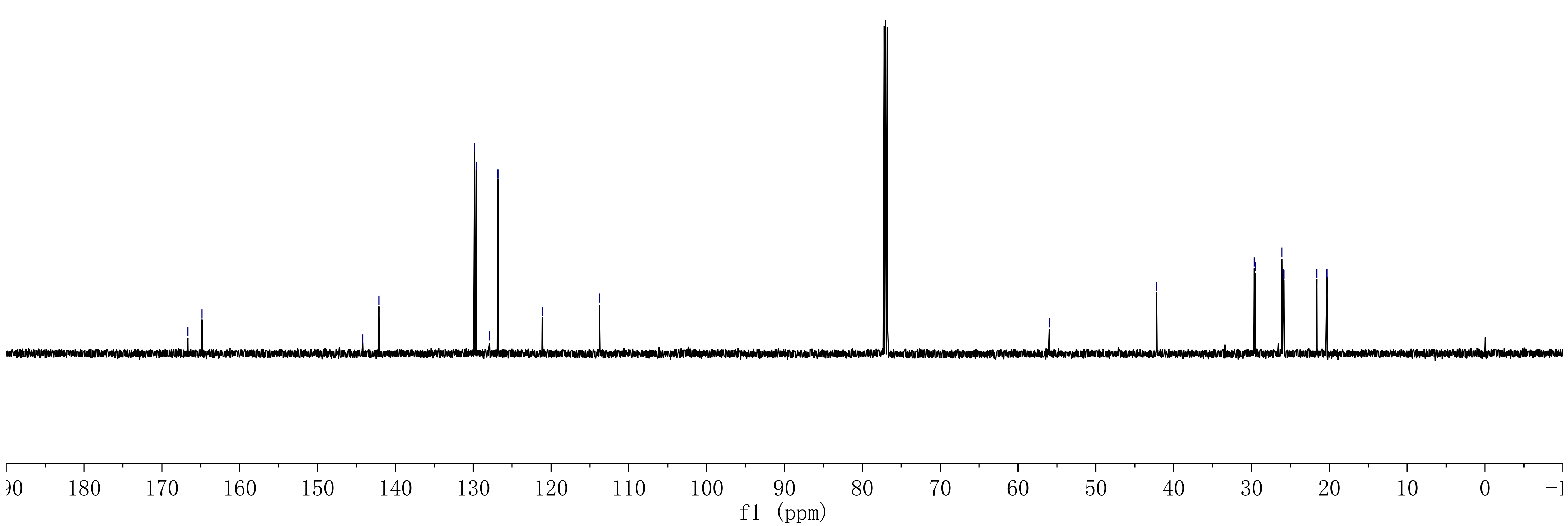
3da



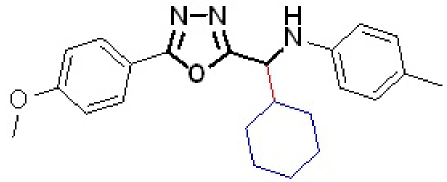
^{13}C NMR (151 MHz, $CDCl_3$) δ 166.65, 164.84, 144.20, 142.11, 129.82, 129.63, 127.89, 126.83, 121.14, 113.77, 55.98, 42.19, 29.67, 29.52, 26.10, 25.89, 25.82, 21.59, 20.33.



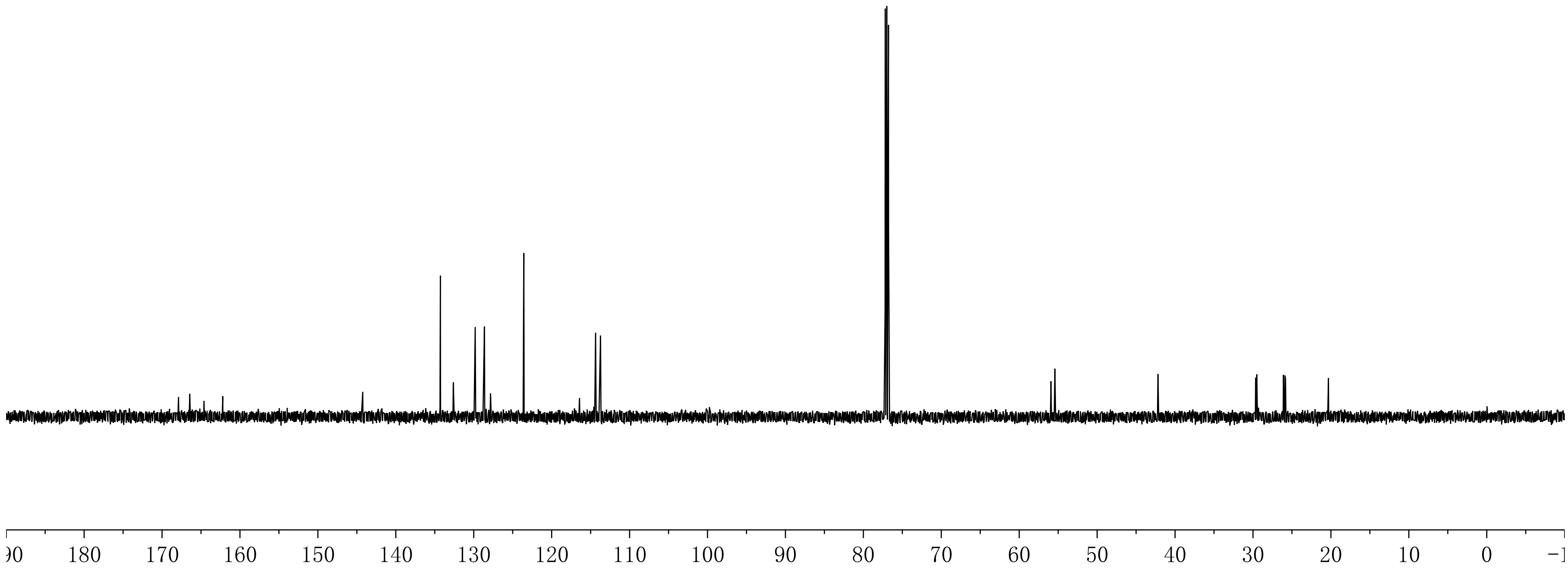
3ea



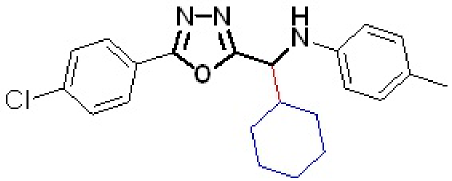
^{13}C NMR (151 MHz, $CDCl_3$) δ 167.89, 166.44, 164.61, 162.21, 144.24, 134.28, 132.62, 129.81, 128.62, 127.84, 123.57, 116.43, 114.36, 113.73, 55.92, 55.42, 42.19, 29.66, 29.51, 26.10, 25.89, 25.83, 20.31.



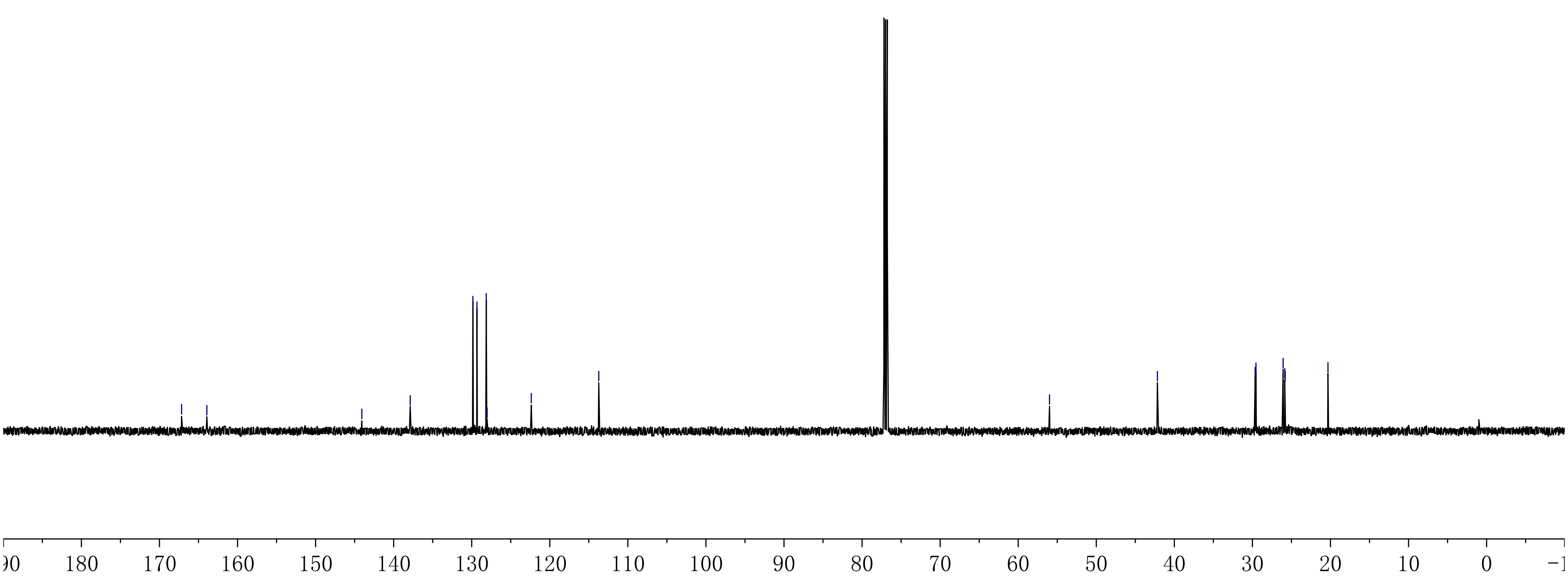
3fa



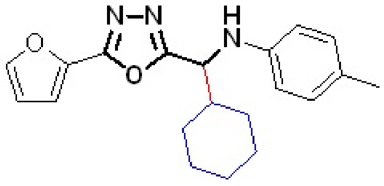
[13]C NMR (151 MHz, $CDCl_3$) δ 167.17, 163.93, 144.08, 137.88, 129.85, 129.33, 128.14, 128.03, 122.37, 113.73, 55.99, 42.17, 29.65, 29.54, 26.07, 25.85, 25.79, 20.32.



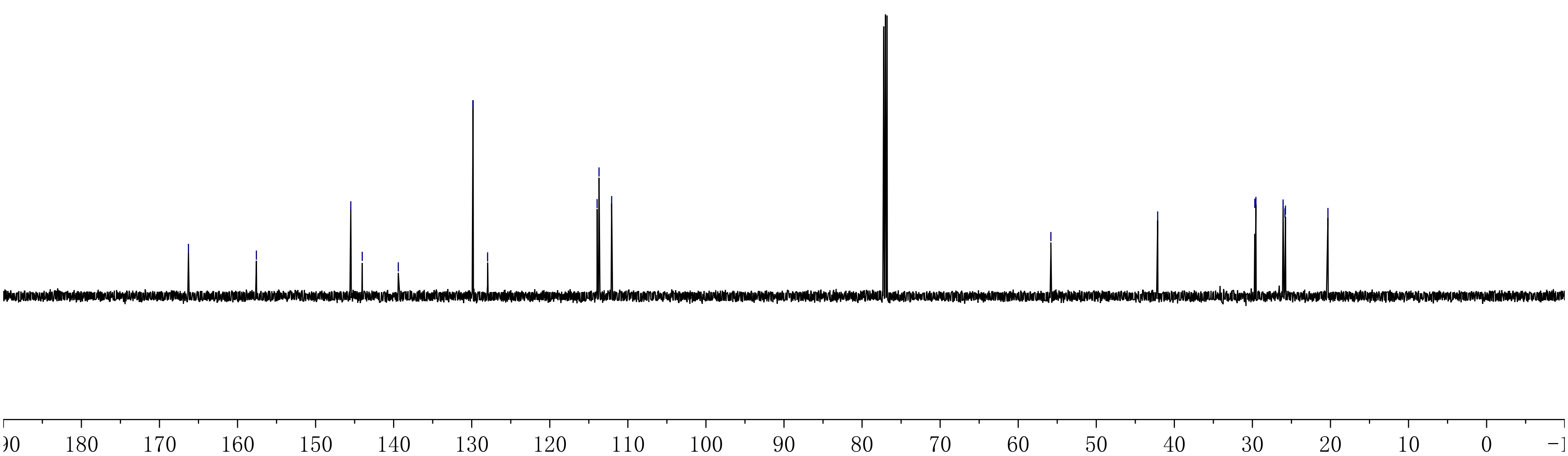
3ga



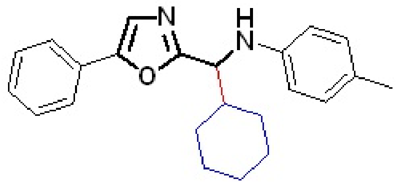
[13]C NMR (151 MHz, $CDCl_3$) δ 166.29, 157.58, 145.49, 144.02, 139.41, 129.83, 127.97, 113.94, 113.70, 112.08, 55.81, 42.13, 29.69, 29.55, 26.07, 25.83, 25.77, 20.32.



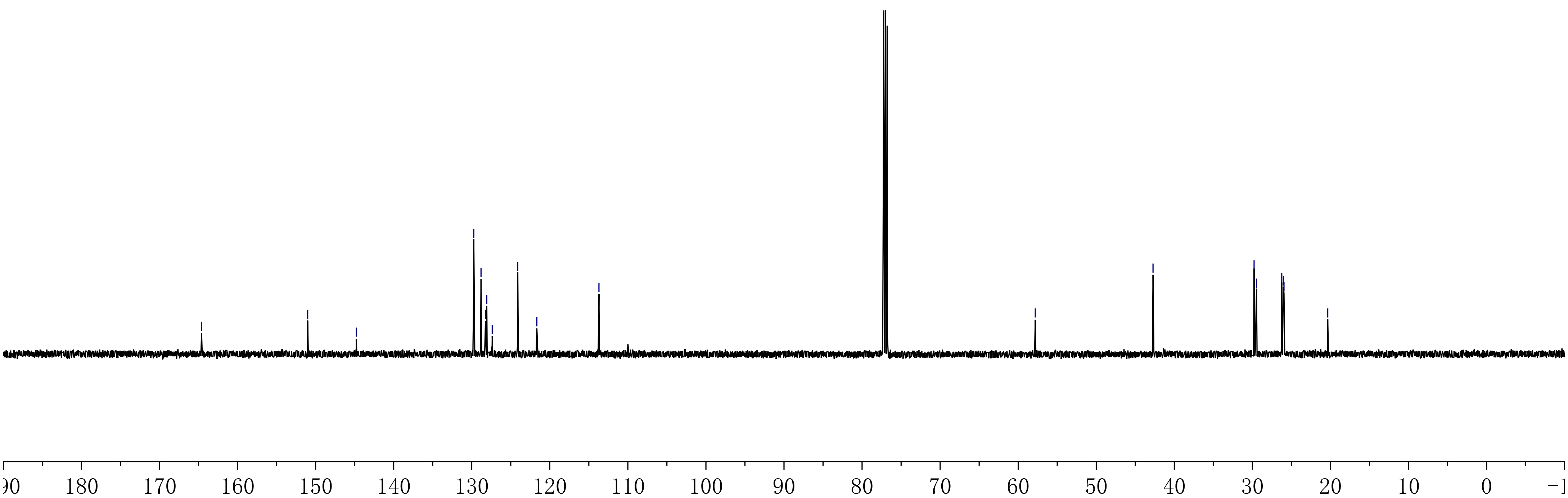
3ha



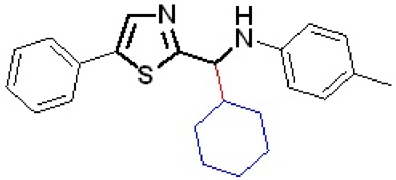
^{13}C NMR (151 MHz, $CDCl_3$) δ 164.60, 151.01, 144.78, 129.74, 128.81, 128.24, 128.07, 127.38, 124.10, 121.66, 113.70, 57.81, 42.73, 29.79, 29.47, 26.24, 26.03, 25.95, 20.35.



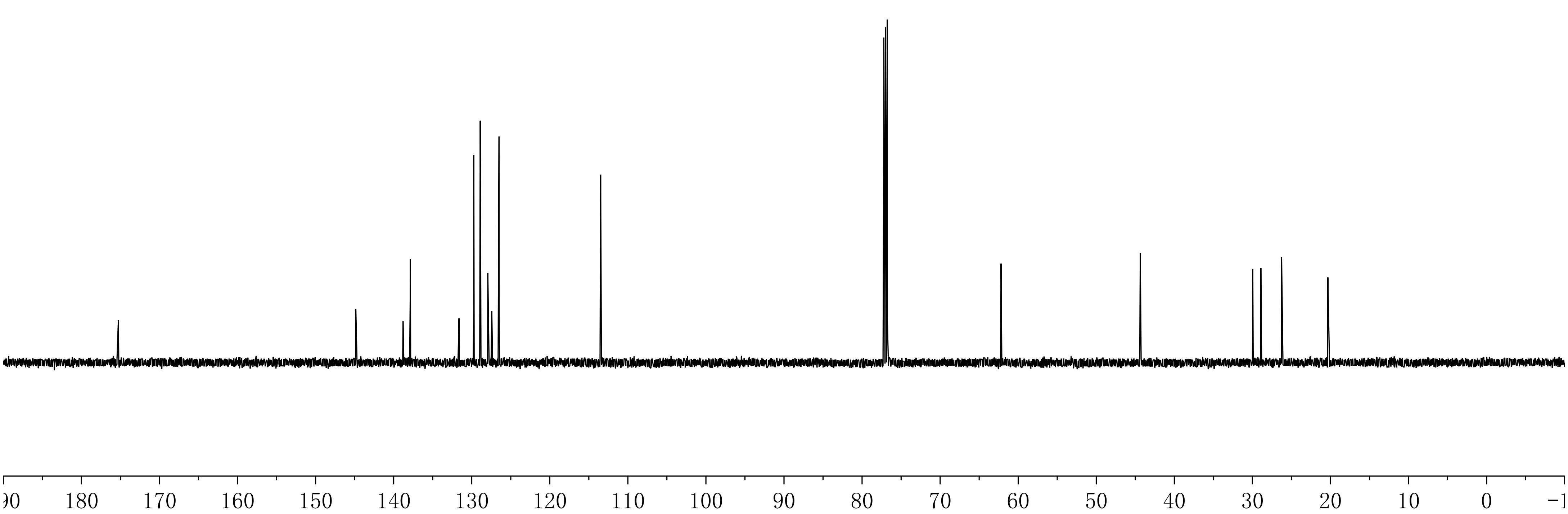
3ia



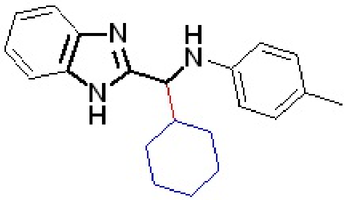
^{13}C NMR (151 MHz, $CDCl_3$) δ 175.26, 144.85, 138.80, 137.86, 131.63, 129.74, 128.91, 127.94, 127.43, 126.50, 113.49, 62.19, 44.36, 29.95, 28.92, 26.26, 26.18, 26.16, 20.34.



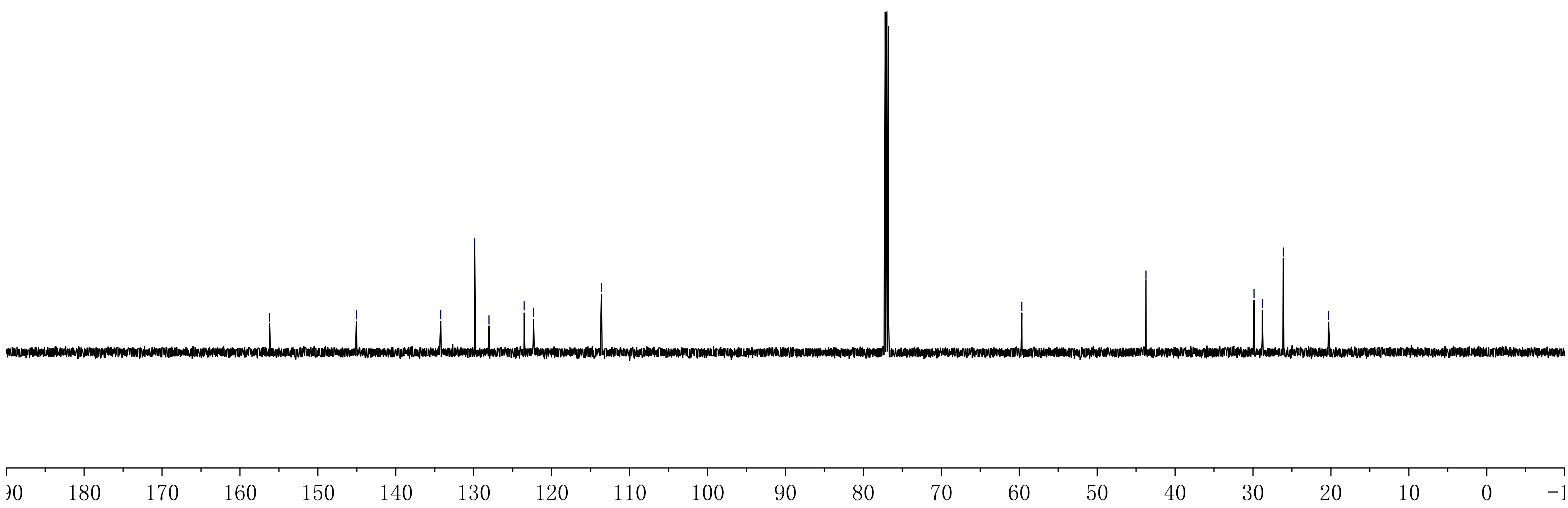
3ja



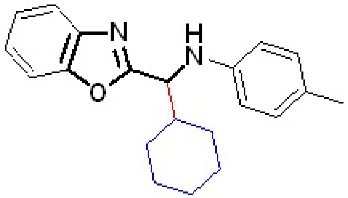
^{13}C NMR (151 MHz, $CDCl_3$) δ 156.20, 145.07, 134.23, 129.87, 128.04, 123.53, 122.33, 113.62, 59.67, 43.74, 29.87, 28.80, 26.12, 20.30.



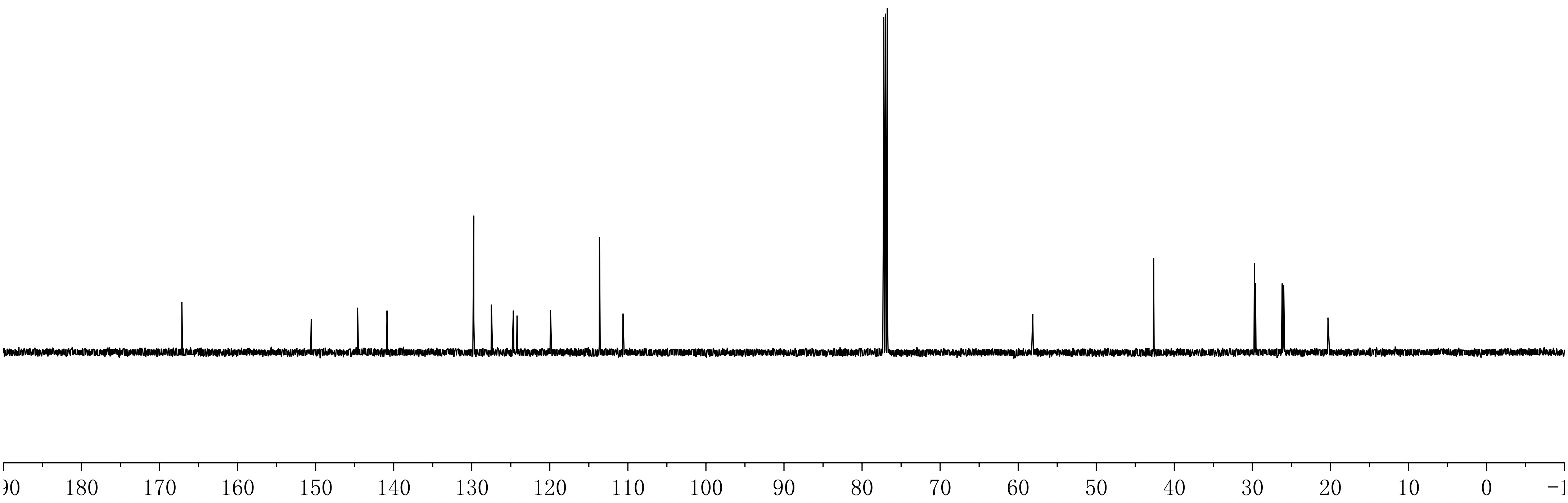
3ka

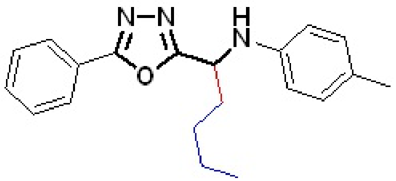


^{13}C NMR (151 MHz, $CDCl_3$) δ 167.13, 150.56, 144.63, 140.85, 129.75, 127.49, 124.67, 124.20, 119.92, 113.64, 110.62, 58.14, 42.66, 29.74, 29.61, 26.17, 25.99, 25.91, 20.32.

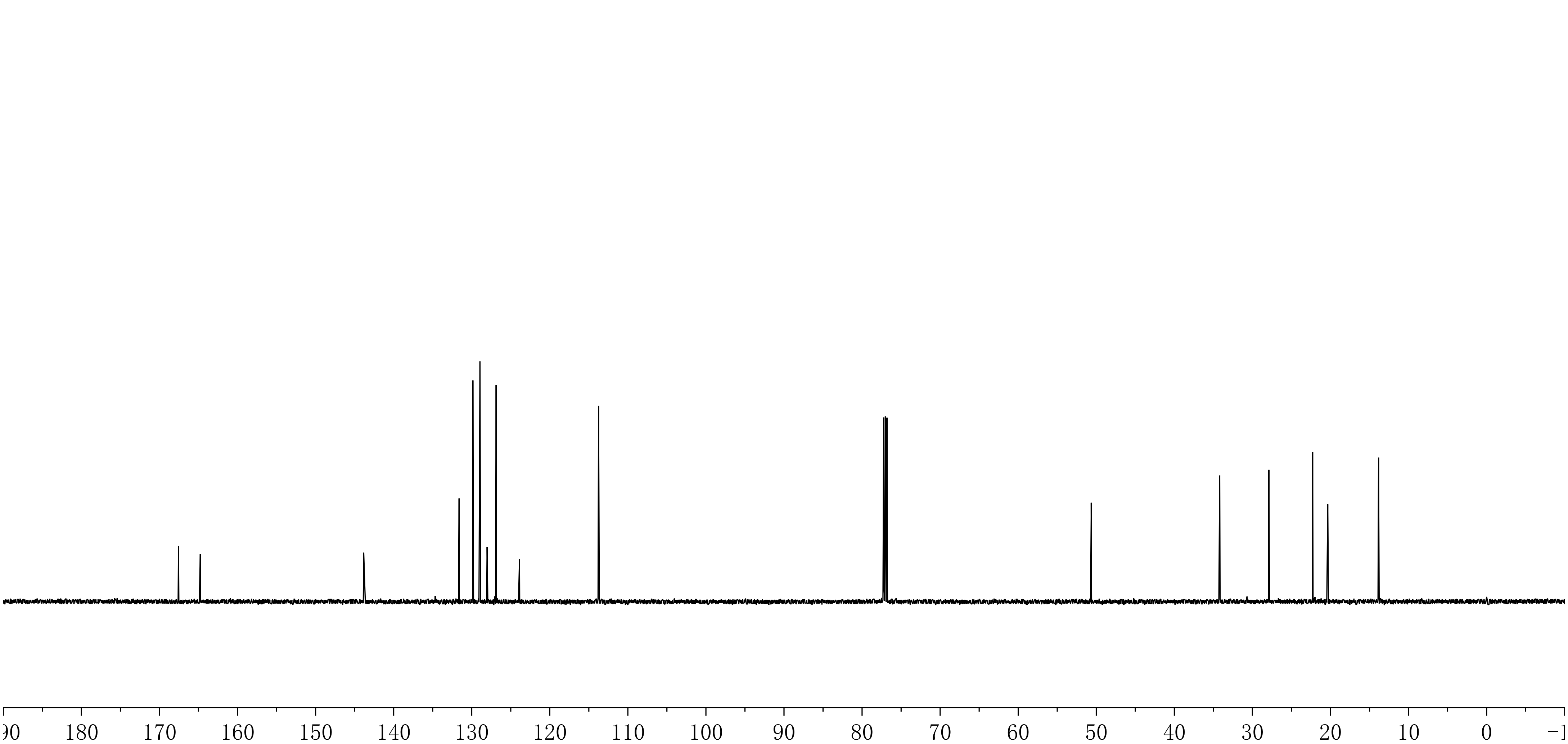


3la

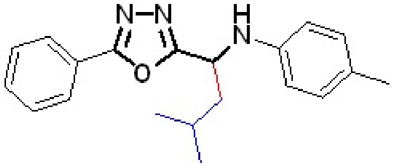




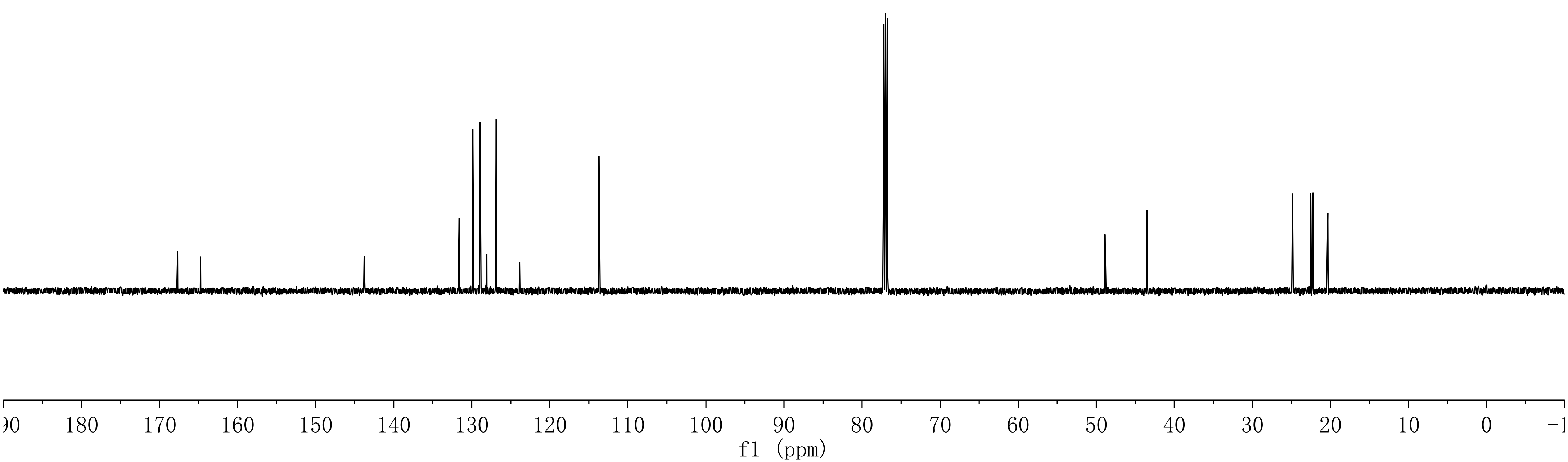
3ab


167.56
164.77
143.83
131.62
129.85
128.94
128.03
126.88
123.88
113.75
50.65
34.19
27.89
22.28
20.35
13.84
90
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
-1

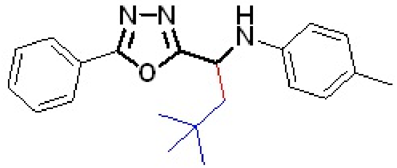
[13]C NMR (151 MHz, $CDCl_3$) δ 167.68, 164.73, 143.78, 131.62, 129.86, 128.93, 128.06, 126.87, 123.87, 113.71, 48.88, 43.47, 24.86, 22.52, 22.23, 20.34.



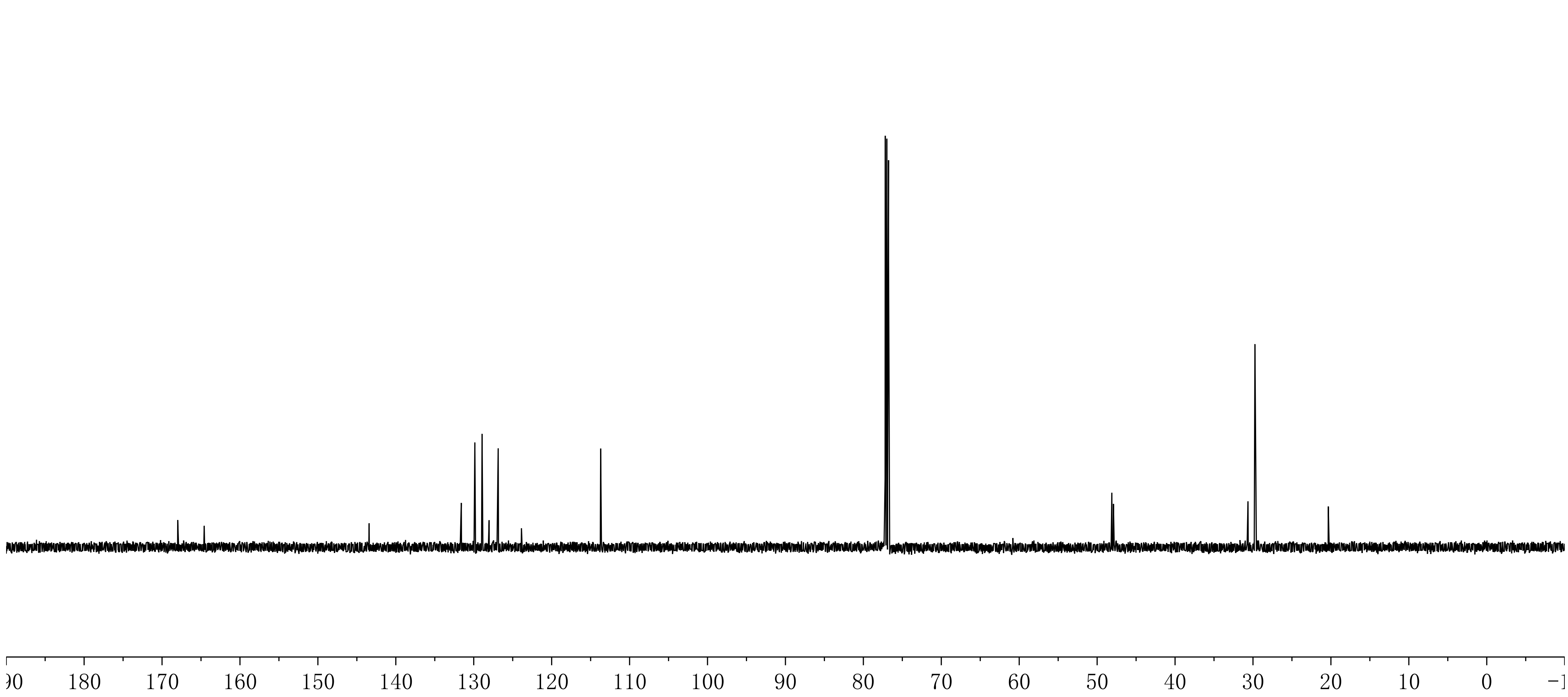
3ac



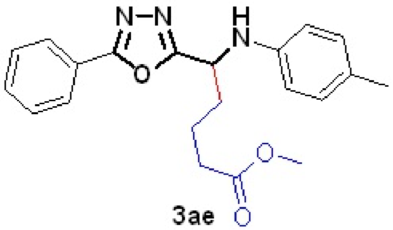
^{13}C NMR (151 MHz, $CDCl_3$) δ 167.99, 164.59, 143.43, 131.60, 129.86, 128.93, 128.03, 126.86, 123.88, 113.71, 48.11, 47.91, 30.64, 29.75, 20.33.



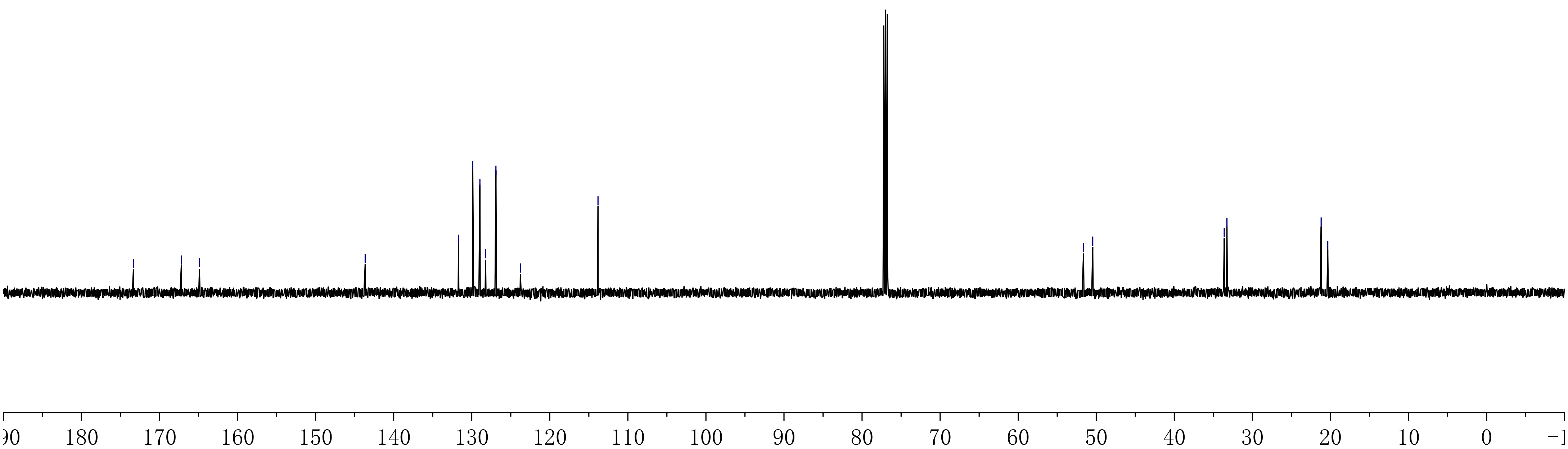
3ad



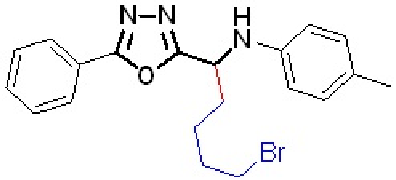
[13]C NMR (151 MHz, $CDCl_3$) δ 173.33, 167.20, 164.88, 143.65, 131.68, 129.87, 128.95, 128.22, 126.90, 123.77, 113.83, 51.63, 50.45, 33.61, 33.26, 21.19, 20.35.



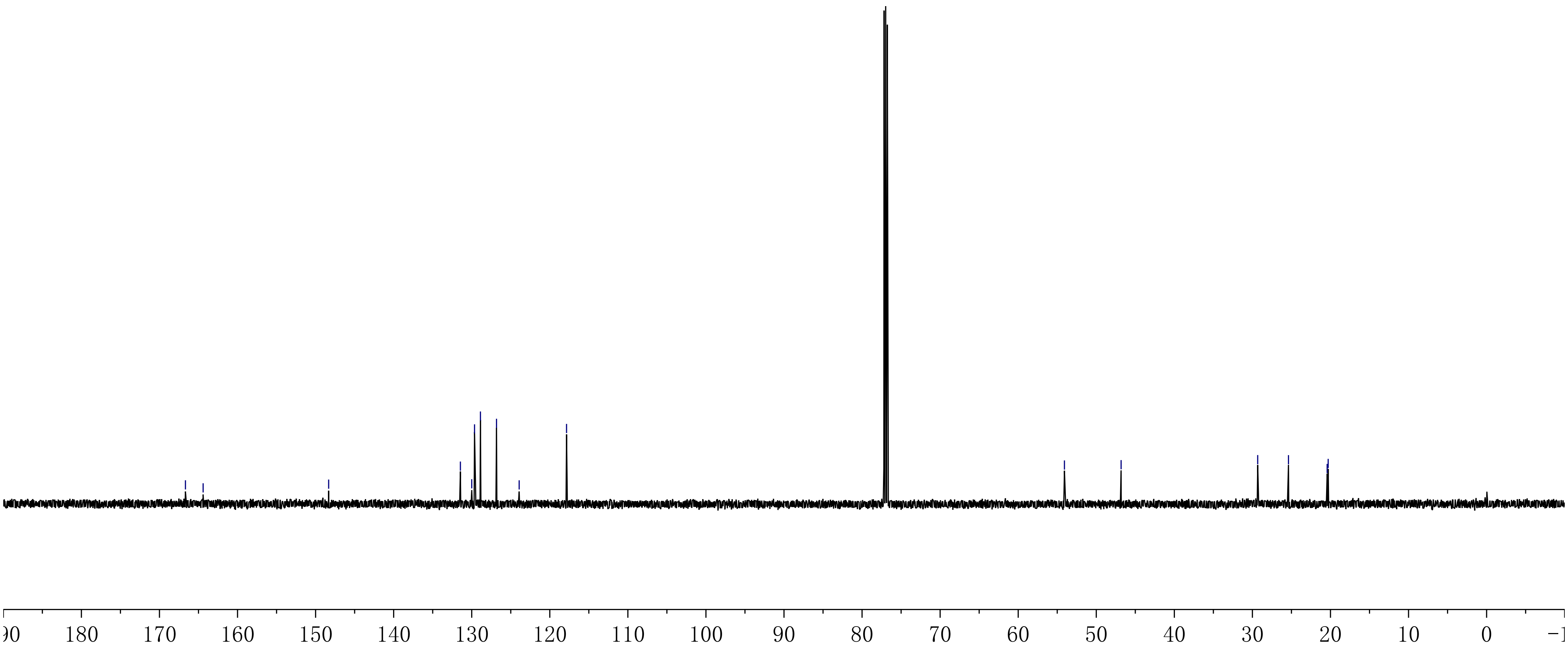
3ae



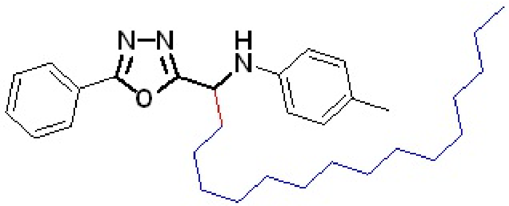
[13]C NMR (151 MHz, $CDCl_3$) δ 166.67, 164.41, 148.33, 131.45, 130.00, 129.64, 128.89, 126.82, 123.92, 117.85, 54.08, 46.82, 29.32, 25.38, 20.42, 20.29.



3af



^{13}C NMR (151 MHz, $CDCl_3$) δ 167.56, 164.76, 143.82, 131.62, 129.85, 128.94, 128.04, 126.88, 123.88, 113.75, 50.67, 34.47, 31.90, 29.67, 29.66, 29.64, 29.63, 29.61, 29.57, 29.48, 29.34, 29.15, 25.75, 22.67, 20.34, 14.10.



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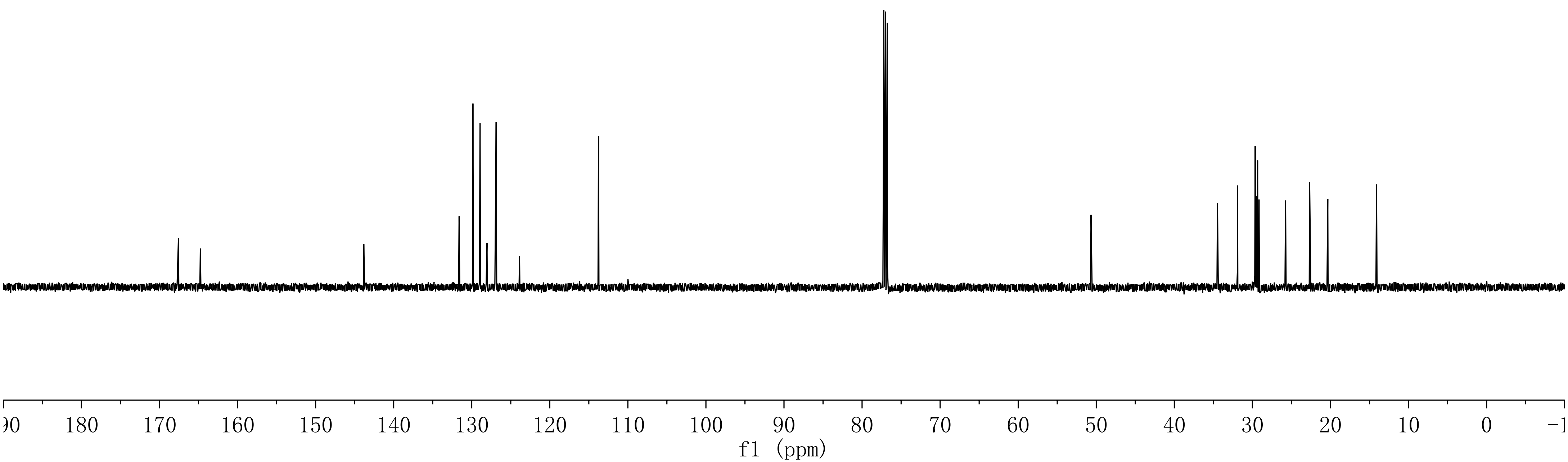
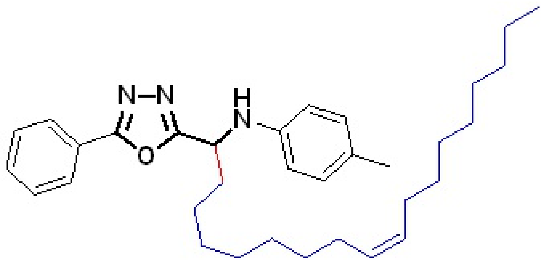
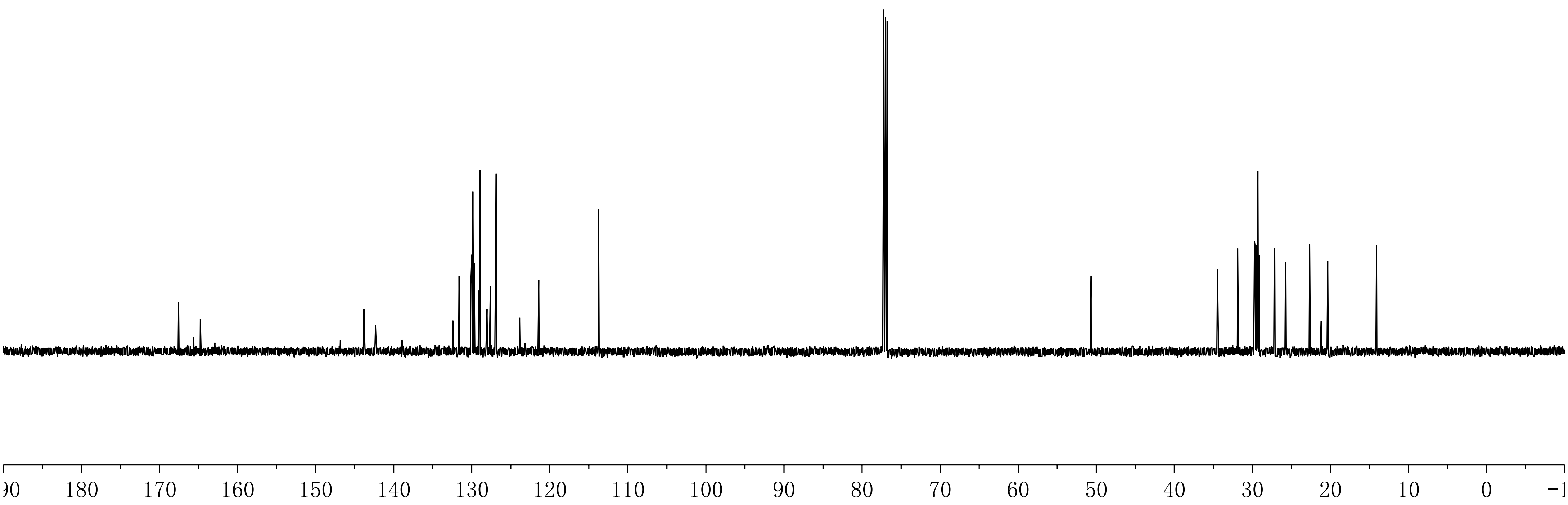


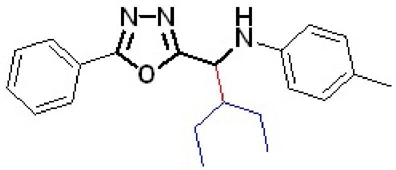
^{13}C NMR (151 MHz, $CDCl_3$) δ 167.55, 164.76, 143.81, 142.33, 131.62, 130.12, 129.98, 129.85, 129.69, 129.11, 128.94, 128.05, 127.62, 126.87, 121.41, 113.75, 50.66, 34.47, 31.88, 29.74, 29.67, 29.50, 29.30, 29.26, 29.14, 29.13, 27.19, 27.14, 25.76, 22.66, 21.19, 20.35, 14.10.



3ah



^{13}C NMR (151 MHz, $CDCl_3$) δ 167.27, 164.68, 144.12, 131.59, 129.86, 128.95, 127.96, 126.84, 123.91, 113.73, 52.89, 45.28, 22.28, 21.84, 20.33, 11.28, 10.93.



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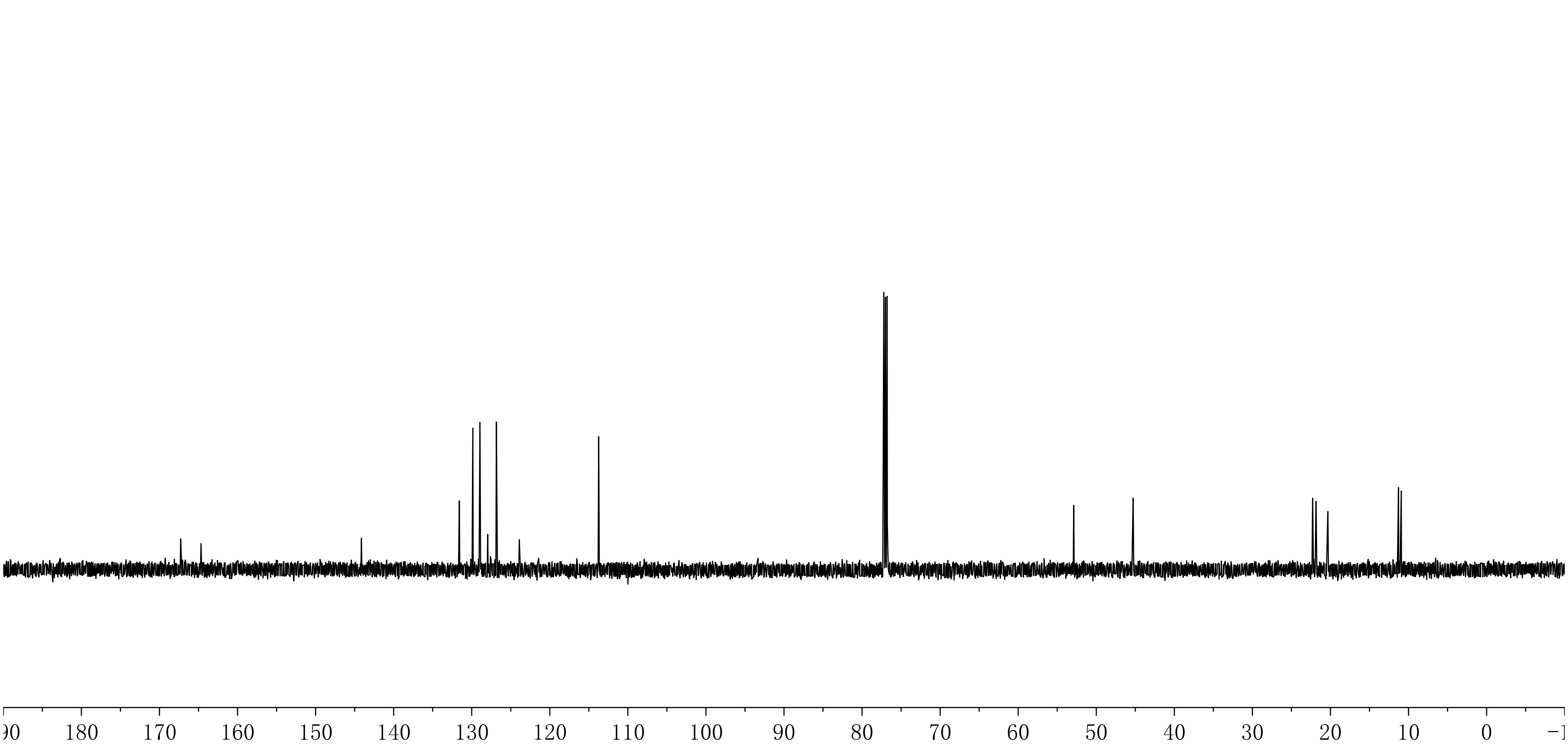
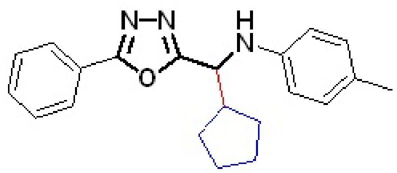


[13]C NMR (151 MHz, $CDCl_3$) δ 167.40, 164.66, 144.01, 131.59, 129.82, 128.93, 127.96, 126.86, 123.89, 113.66, 55.07, 43.93, 29.98, 29.29, 25.34, 25.33, 20.32.



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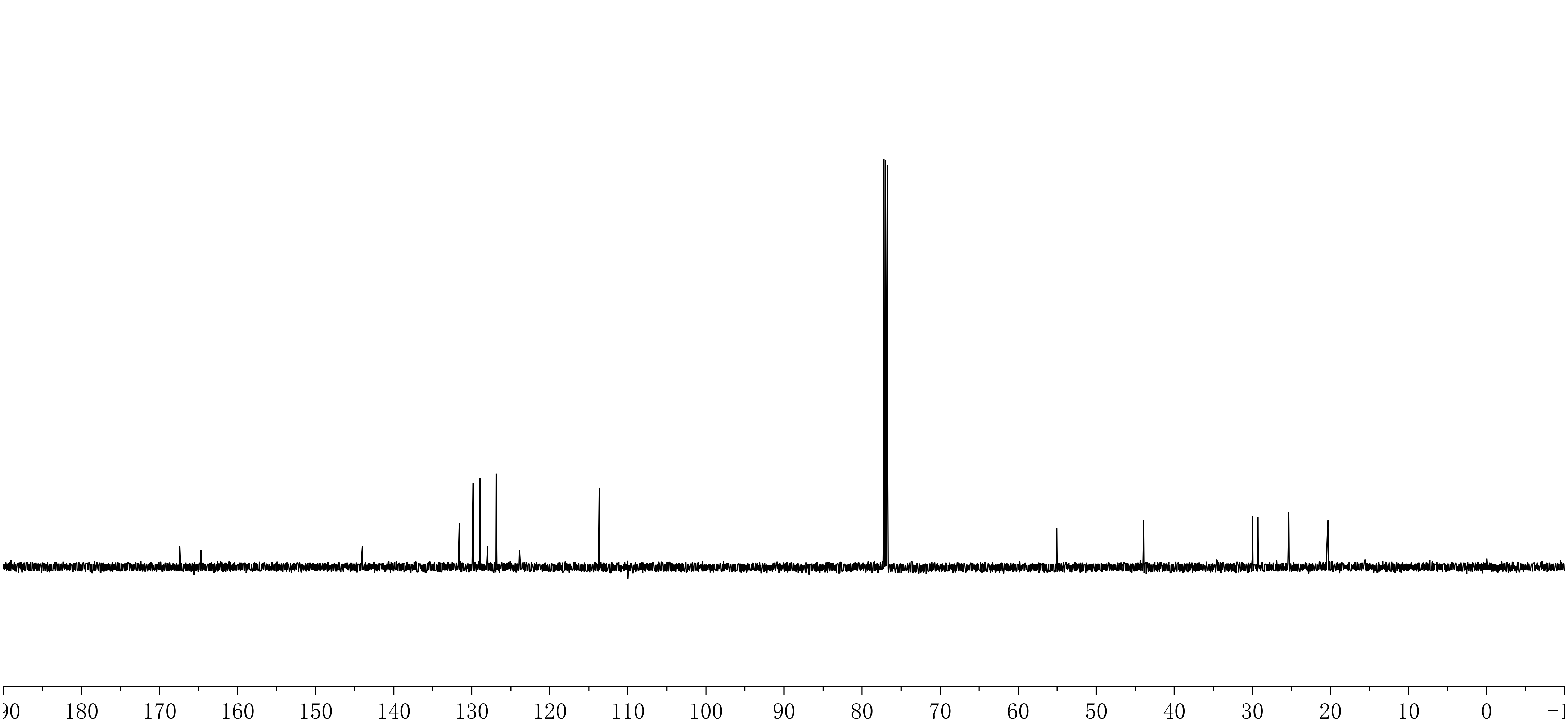
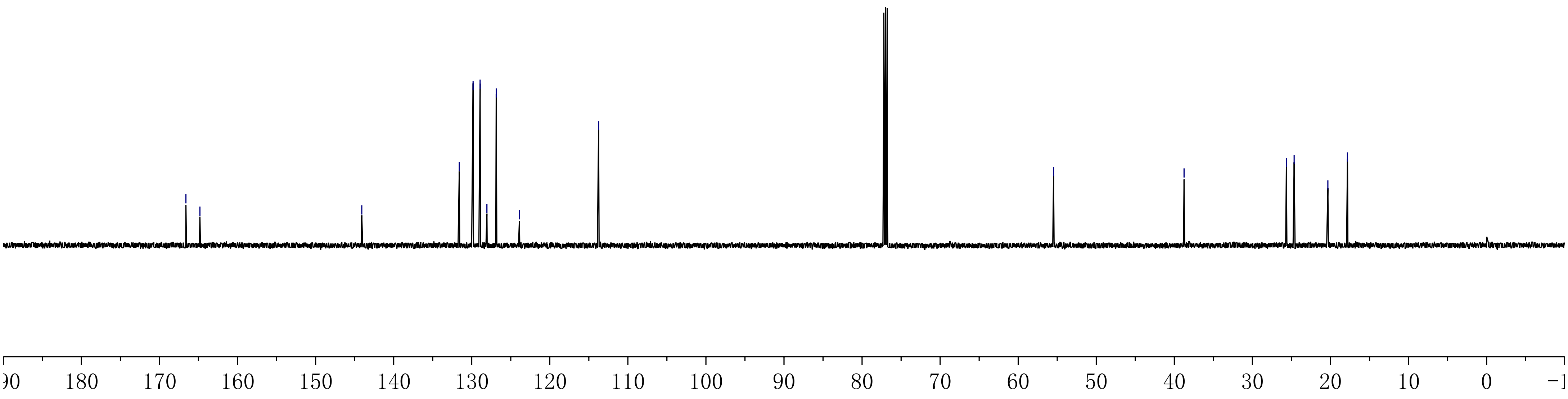
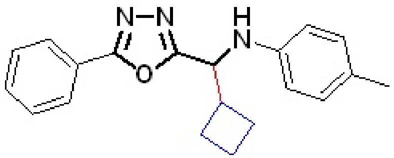


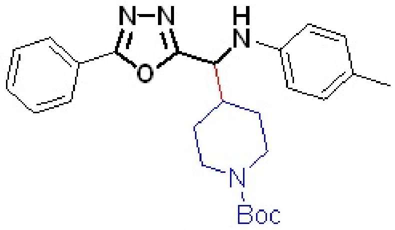


^{13}C NMR (151 MHz, $CDCl_3$) δ 166.61, 164.82, 144.08, 131.59, 129.83, 128.93, 128.06, 126.86, 123.90, 113.75, 55.47, 38.75, 25.65, 24.65, 20.33, 17.82.

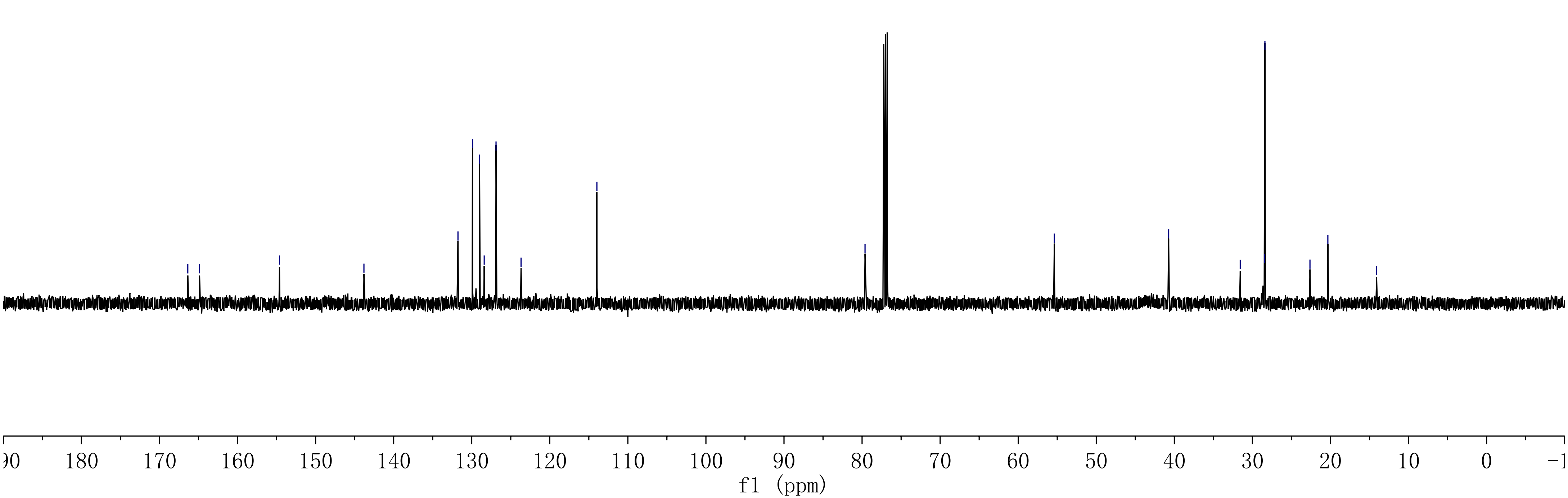


3ak

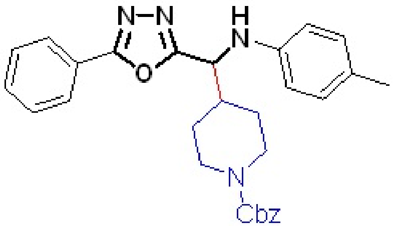
^{13}C NMR (151 MHz, $CDCl_3$) δ 166.37, 164.86, 154.62, 143.81, 131.76, 129.90, 129.00, 128.40, 126.89, 123.68, 113.97, 79.62, 55.38, 40.73, 31.56, 28.45, 28.40, 22.63, 20.33, 14.09.



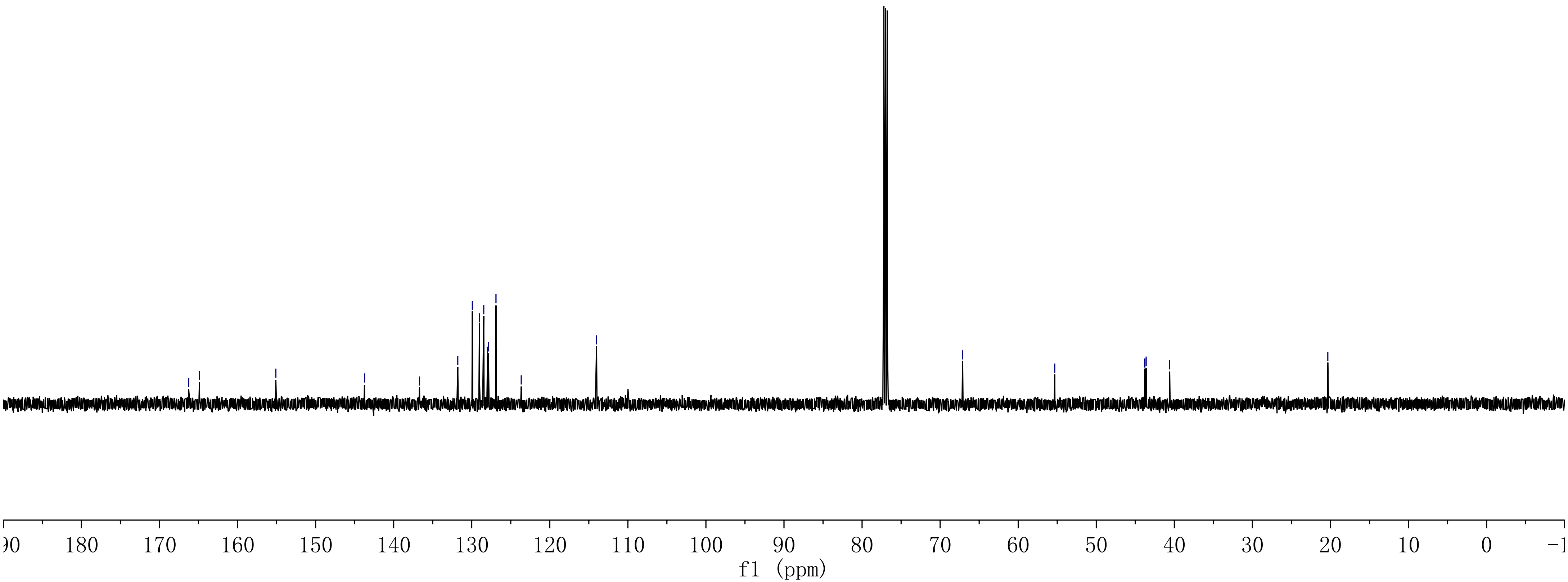
3al



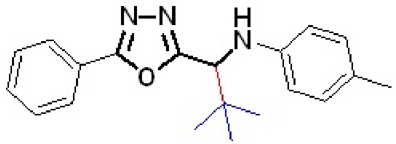
^{13}C NMR (151 MHz, $CDCl_3$) δ 166.26, 164.88, 155.09, 143.74, 136.69, 131.79, 129.92, 129.01, 128.50, 128.46, 127.98, 127.86, 126.89, 123.66, 114.01, 67.12, 55.32, 43.78, 43.61, 40.60, 20.34.



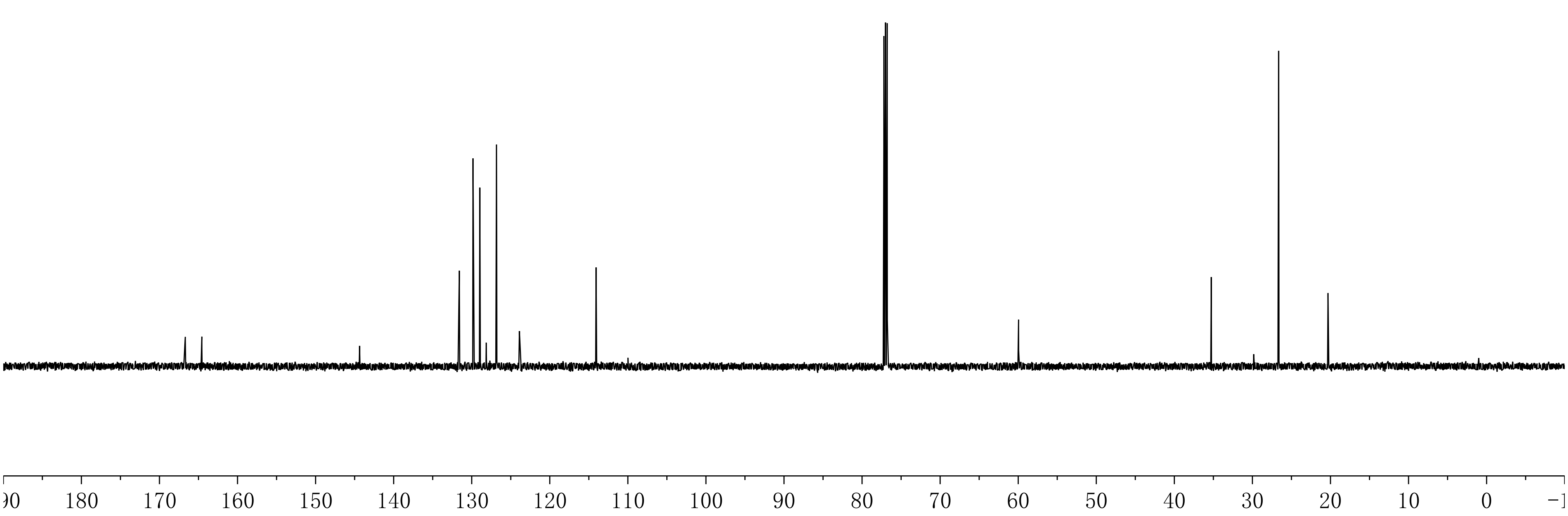
3am



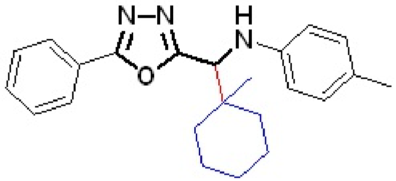
^{13}C NMR (151 MHz, $CDCl_3$) δ 166.70, 164.57, 144.36, 131.59, 129.84, 128.96, 128.14, 126.82, 123.89, 114.07, 59.96, 35.27, 26.63, 20.32.



3an



[13]C NMR (151 MHz, $CDCl_3$) δ 166.58, 164.56, 144.49, 131.55, 129.84, 128.95, 128.04, 126.82, 123.94, 114.04, 37.87, 34.90, 34.79, 25.95, 21.75, 21.51, 20.32.



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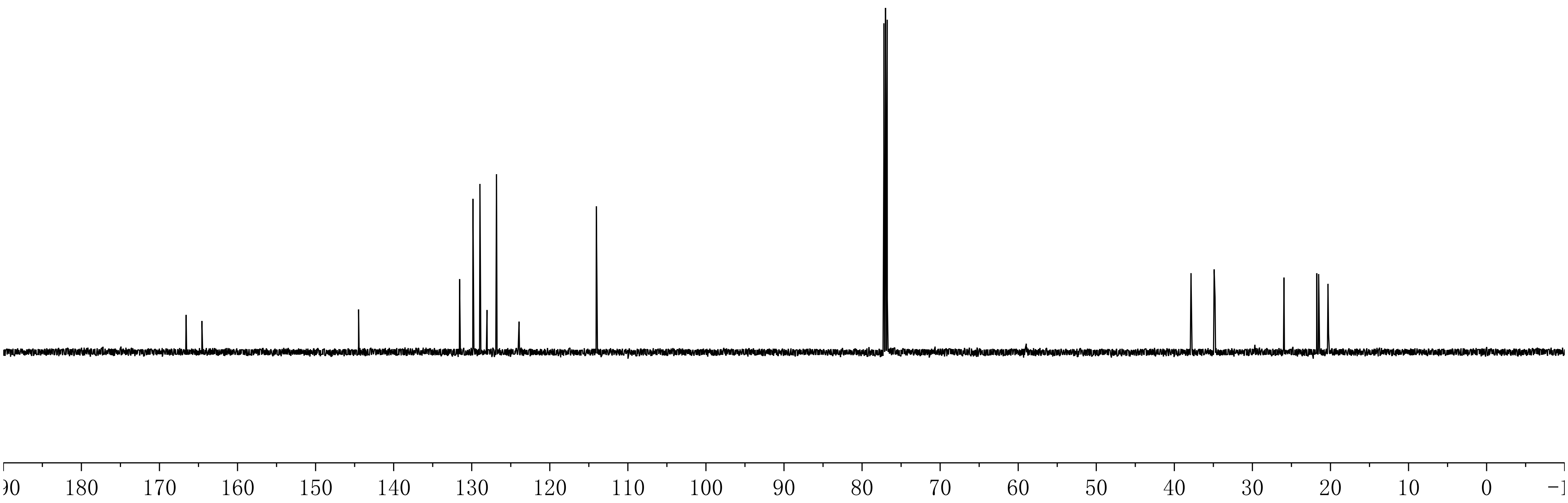
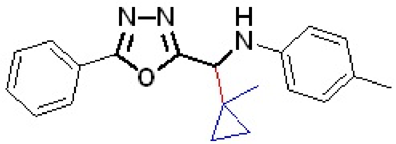


^{13}C NMR (151 MHz, $CDCl_3$) δ 166.65, 164.81, 144.14, 131.65, 129.81, 128.97, 128.15, 126.88, 123.86, 113.89, 57.86, 20.34, 19.97, 19.38, 12.60, 11.49.



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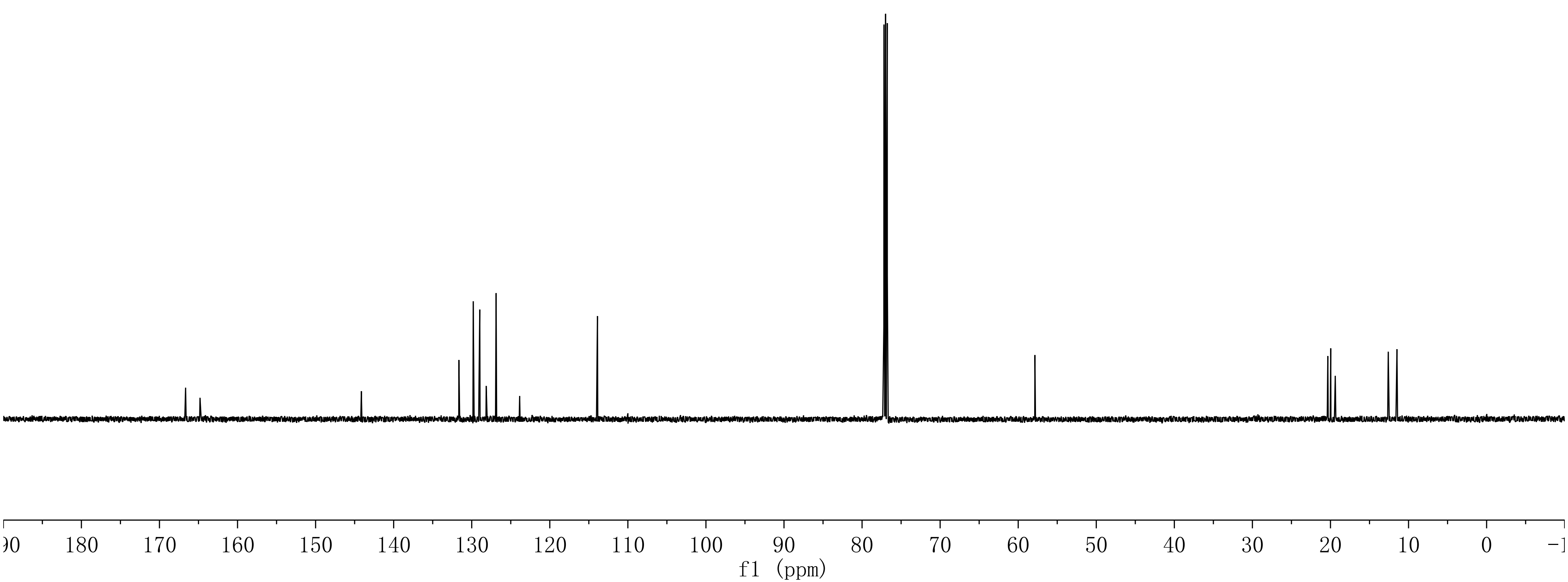
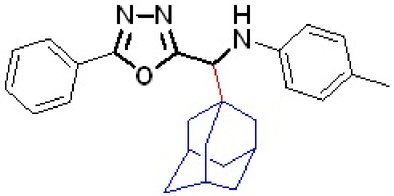


^{13}C NMR (151 MHz, $CDCl_3$) δ 166.23, 164.59, 144.54, 131.55, 129.82, 128.95, 127.97, 126.85, 123.97, 113.99, 60.67, 38.93, 37.03, 36.72, 28.26, 20.31.



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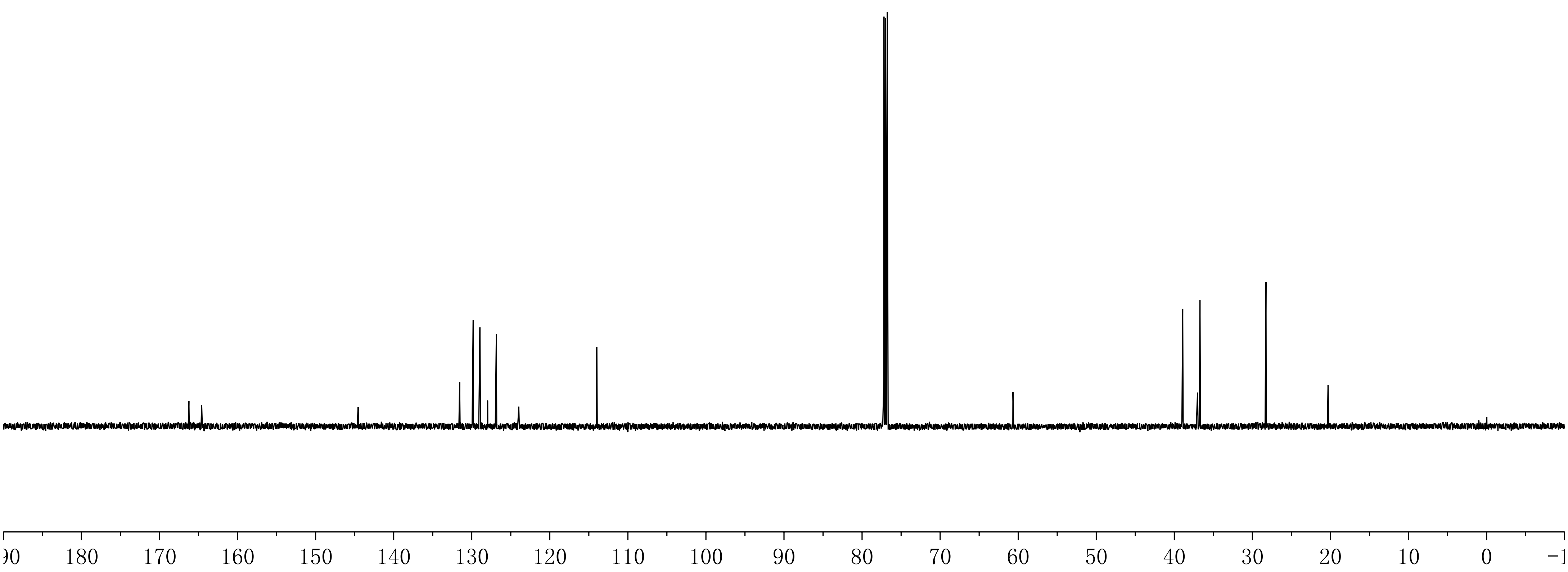
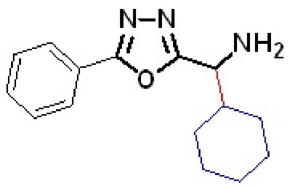
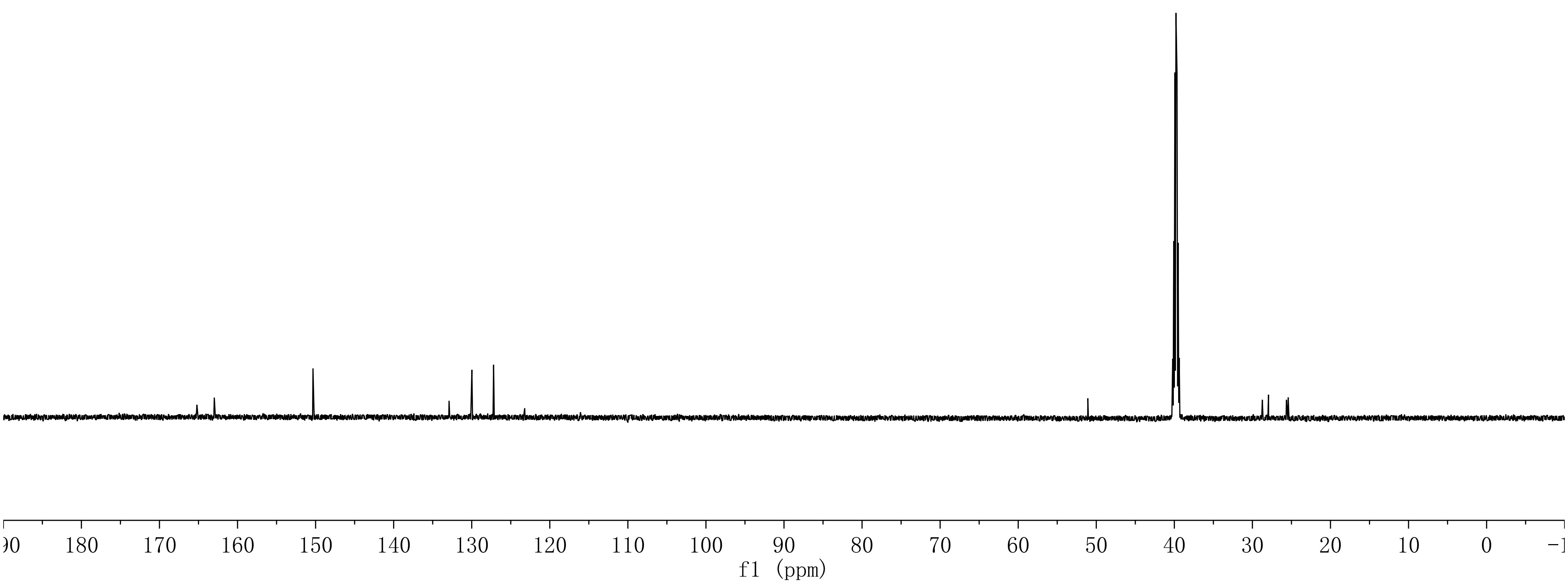
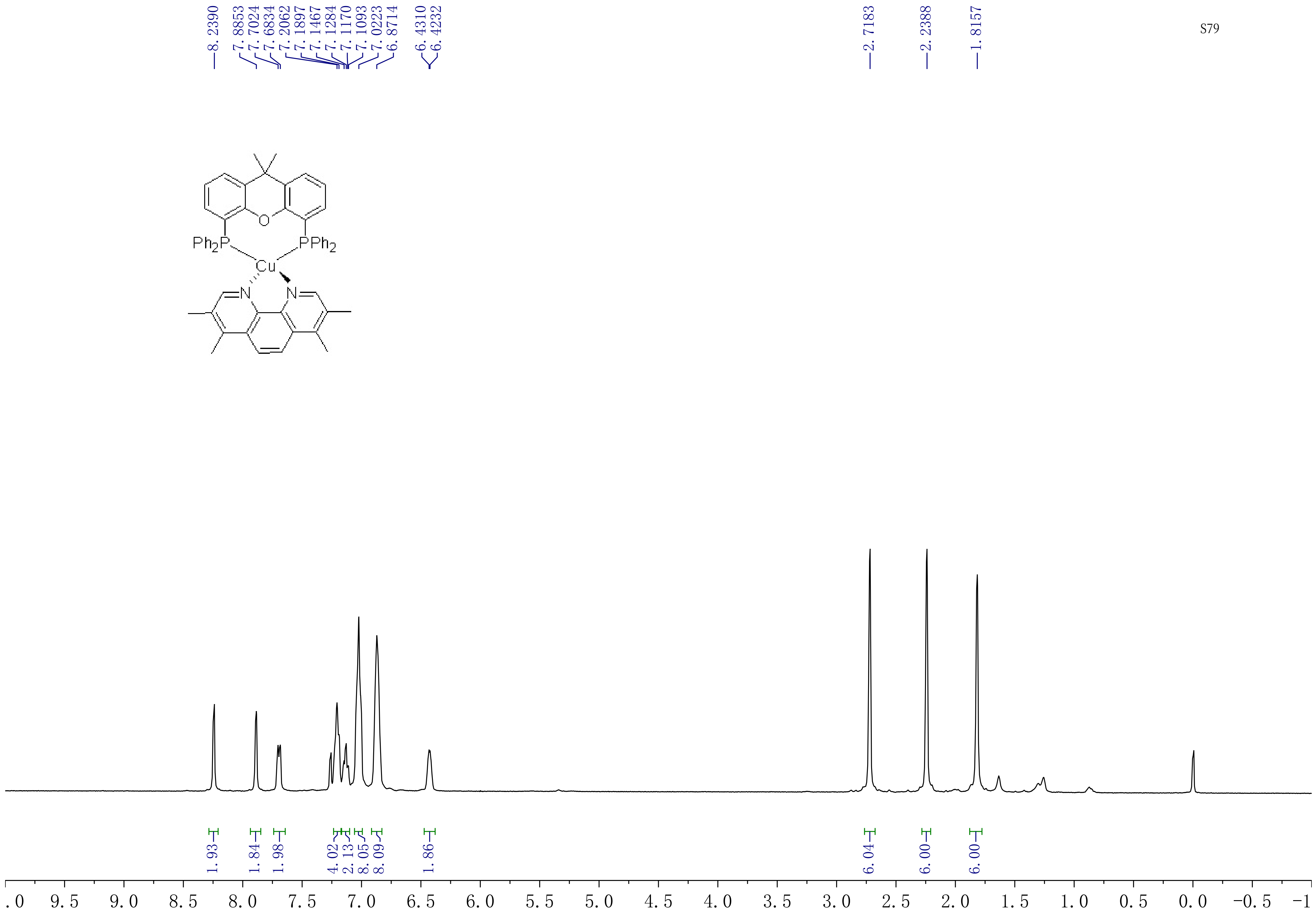


^{13}C NMR (151 MHz, DMSO) δ 165.20, 162.97, 150.33, 132.90, 129.98, 127.20, 123.22, 51.08, 28.73, 27.94, 25.64, 25.55, 25.42.



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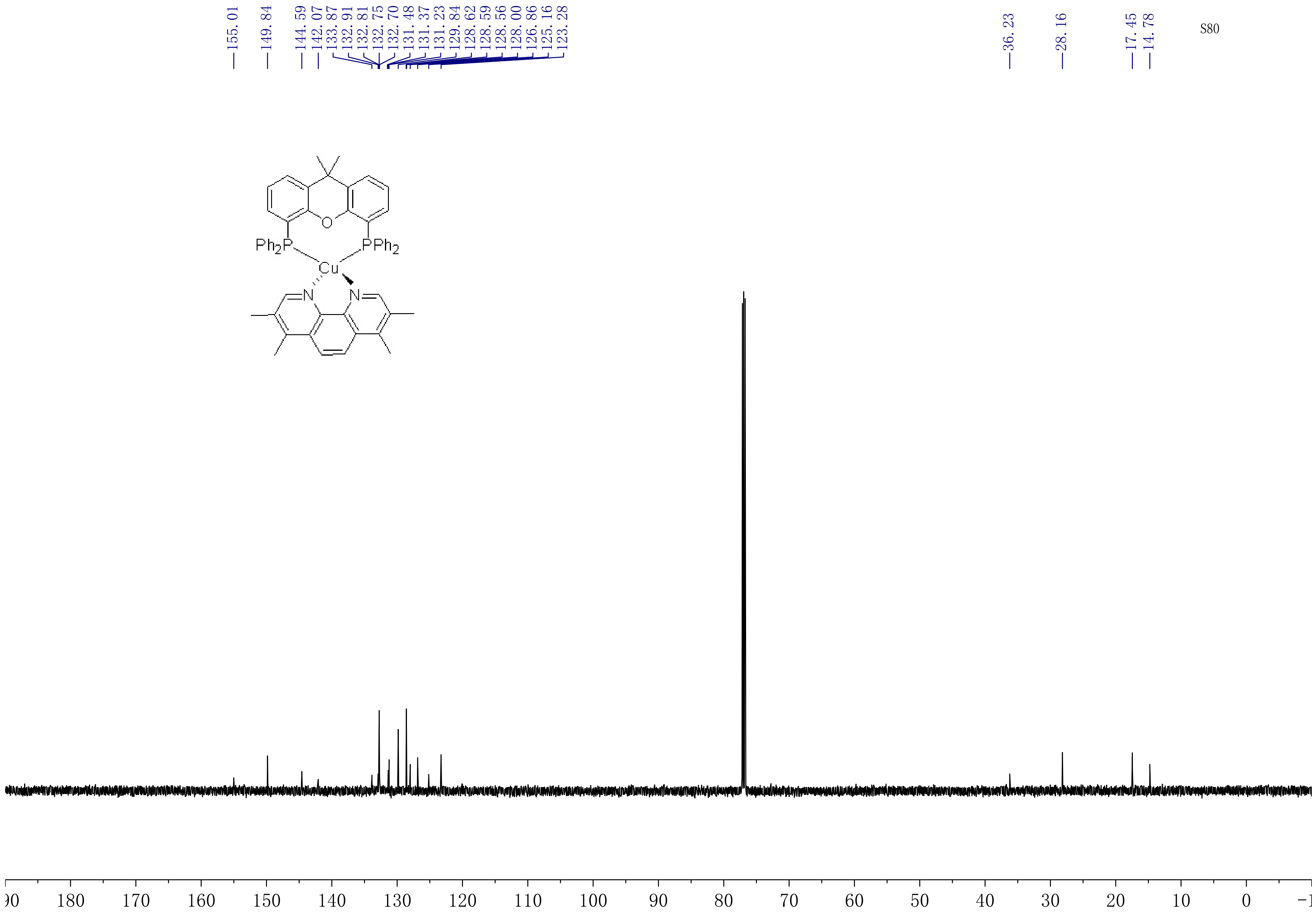
6.8714
6.4310
6.4232
2.7183
2.2388
1.8157
Ph2P
PPh2
O
Cu
N
N
1.93
1.84
1.98
4.02
2.13
8.05
8.09
1.86
6.04
6.00
6.00
.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 −0.5 −1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144.59
142.07
133.87
132.91
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132.75
132.70
131.48
131.37
131.23
129.84
128.62
128.59
128.56
128.00
126.86
125.16
123.28
36.23
28.16
17.45
14.78
Ph2P
PPh2
O
Cu
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10
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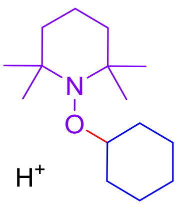
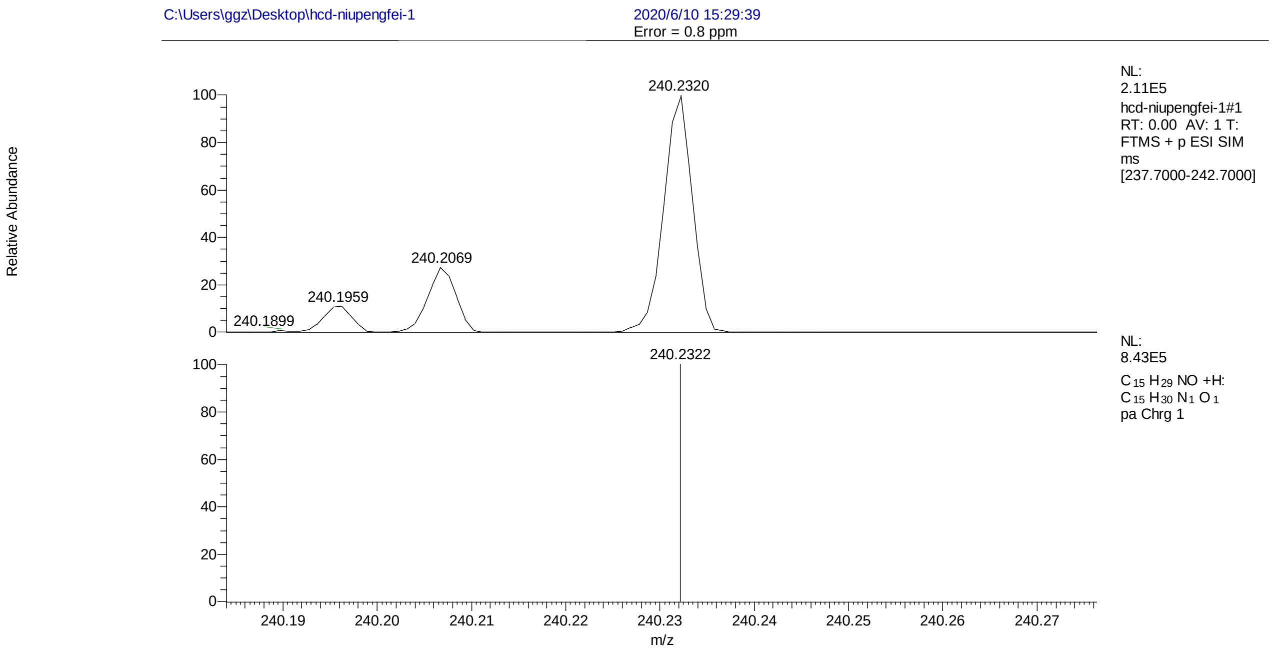
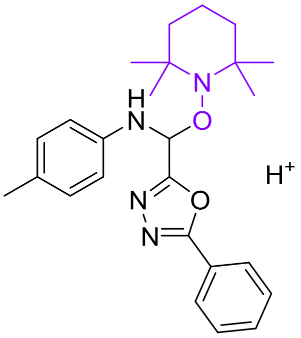


$C_{15}H_{30}NO^{+}$

[M+H] m/z 240.2322





$C_{25}H_{33}N_4O_2^+$

[M+H] m/z 421.2598

G:\自由基捕获质谱\hcd-niupengfei-2

2020/6/11 15:45:12

Error = 1.4 ppm