Supporting Information

NaHS·nH$_2$O-Induced Umpolung: The Synthesis of 2-Acyl-3-aminoindoles from Aryl Methyl Ketones and 2-Aminobenzonitriles

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1. General

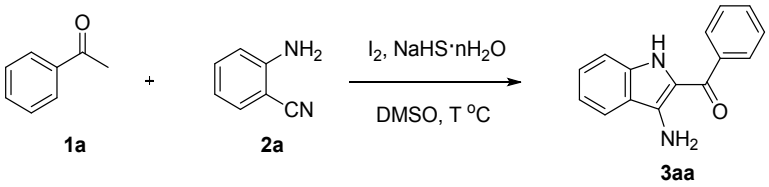
All substrates and reagents were commercially available and used without further purification. TLC analysis was performed using pre-coated glass plates. Column chromatography was performed using silica gel (200–300 mesh). IR spectra were recorded on a Perkin-Elmer PE-983 infrared spectrometer as KBr pellets with absorption in cm^{-1}. ^1H spectra were recorded in $CDCl_3$ or DMSO-d6 on 300/400/600 MHz NMR spectrometers and resonances (δ) are given in parts per million relative to tetramethylsilane. Data are reported as follows: chemical shift, multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet), coupling constants (Hz) and integration. ^{13}C spectra were recorded in $CDCl_3$ or DMSO-d6 on 75/100/150 MHz NMR spectrometers and resonances (δ) are given in ppm. HRMS were obtained on a Bruker 7-tesla FT-ICR MS equipped with an electrospray source. Melting points were determined using XT-4 apparatus and not corrected.

2. General procedure for the synthesis of 3 (3aa as an example)

A sealed tube was charged with acetophenone (**1a**) (60 mg, 0.5 mmol), iodine (203 mg, 0.8 mmol) at room temperature, and DMSO (2 mL) was added. The resulting mixture was stirred at 100 °C, after disappearance of the reactant (monitored by TLC), then added 2-aminobenzonitrile (**2a**) (59 mg, 0.5 mmol) and $NaHS{\cdot}nH_2O$ (197 mg, 2.5 mmol) at 100 °C for another 7 h. After the reaction completed, the mixture was quenched with saturation $Na_2S_2O_3$ solution (50 mL), extracted with EtOAc (3 × 50 mL). The combined organic layers were washed with brine, dried over anhydrous Na_2SO_4 and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether/EtOAc = 5:1) to yield the desired product **3aa** as a yellow oil.

3. Optimization of the Reaction Conditions

Table 1. Optimization of the Standard Conditions [a] (Table S1)



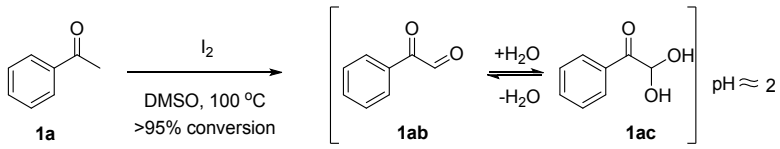
1a **2a** **3aa**

Entry	I_2 (equiv.)	Temp (℃)	'S'	Yield [b] (%)
1	1.6	90	$NaHS{\cdot}nH_2O$	58
2	1.6	80	$NaHS{\cdot}nH_2O$	54
3	1.6	100	$NaHS{\cdot}nH_2O$	71
4	1.6	110	$NaHS{\cdot}nH_2O$	47
5	1.6	120	$NaHS{\cdot}nH_2O$	Trace
6	0.8	100	$NaHS{\cdot}nH_2O$	51
7	1.2	100	$NaHS{\cdot}nH_2O$	57
8	2.0	100	$NaHS{\cdot}nH_2O$	Trace
9	1.6	100	$Na_2S{\cdot}9H_2O$	23

10	1.6	100	S_8	N.D
11	1.6	100	K_2S	11
12[c]	1.6	100	$NaHS{\cdot}nH_2O$	36
13[d]	1.6	100	$NaHS{\cdot}nH_2O$	66
14	1.6	100	K_2CO_3	53
15	1.6	100	$NaHCO_3$	48
16	1.6	100	DBU	26
17	1.6	100	Et_3N	Trace
18	1.6	100	HCl	15
19	1.6	100	HI	10
20	1.6	100	$Cu(OAc)_2$	21
21	1.6	100	$FeCl_3$	33

[a]Reaction conditions: **1a** (0.5 mmol), I_2, heated in 2 mL of DMSO within 45 mins, and then **2a** (0.5 mmol), $NaHS{\cdot}nH_2O$ (2.5 mmol) were added for 7h, unless otherwise noted. [b]Isolated yields. [c]$NaHS{\cdot}nH_2O$ (2.0 mmol.). [d]$NaHS{\cdot}nH_2O$ (3.0 mmol.).

Table 2 Optimization of the Reaction Conditions B [a]



Acetophenone (**1a**, 0.5 mmol) was reacted with I_2 (0.8 mmol) in DMSO (2 mL) at 100 °C to give phenylglyoxal (**1ab**) and the corresponding hydrated species (**1ac**) in quantitative yield. Due to the in-situ generating of HI, we found that the pH ≈ 2 under carefully measurements after the first step.

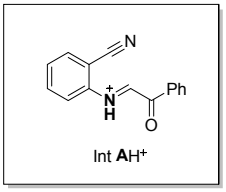
entry	I_2 (equiv)	HI	yield[b] (%)
1	1.6	0	16
2	1.6	0.03 mL	23
3	1.2	0.03 mL	43
4	1.0	0.03 mL	53
5	0.8	0.03 mL	45

6	0.4	0.03 mL	trace
7	0	0.03 mL	0

[a]Reaction conditions: **1ac** (0.5 mmol), **2a** (0.5 mmol), NaHS·nH_2O (1.0 mmol), HI, I_2, heated in 2 mL of DMSO within 7 h unless otherwise noted. [b]Isolated yields.

4. Mechanism research

(1) We used MS to monitor the model reaction, and the possible intermediate A was observed (see below).



detected by MS

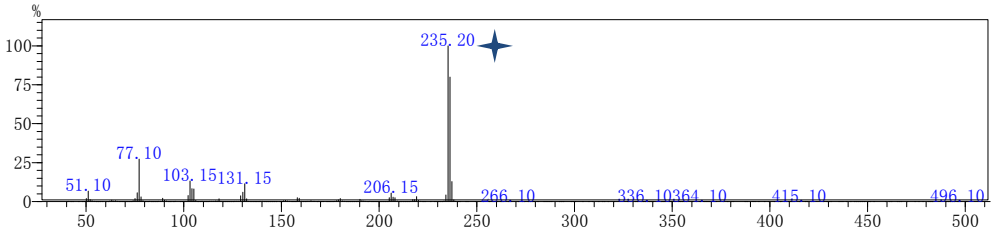
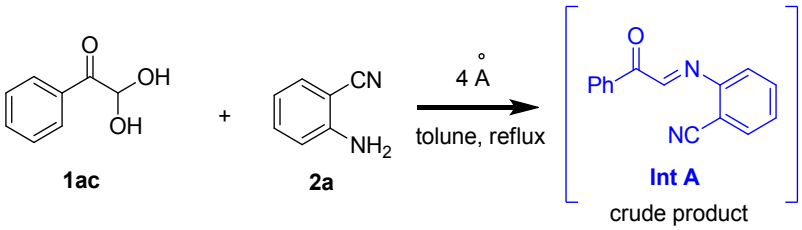
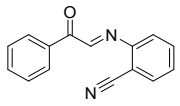


Figure 1. The product Int. **A** from standard conditions was detected by MS.

(2) General procedure for the synthesis of intermediate A



To a 50 mL round-bottom flask equipped with Dean-Stark distillation set-up were added 2-Aminobenzonitrile 2a (0.5 mmol) and toluene (10 mL). To the above solution were added hydrated species 1ac (0.6 mmol) and 4 Å molecular sieves (100 mg). The mixture was refluxed, after the complete conversion of starting material, the reaction mixture was concentrated in vacuo to furnish the crude product Int. A as yellow oil (detected by MS see below).



Int A

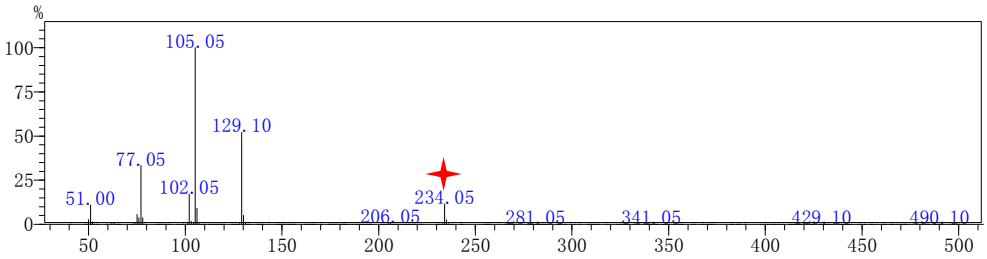
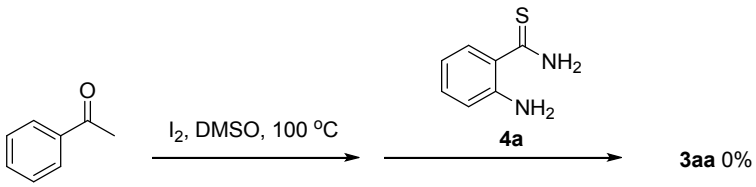


Figure 2. The product Int A from 1ac and 2a was detected by MS.

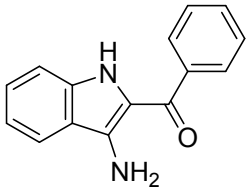
(3) 2-aminobenzothioamide is intermediate?



1a

Acetophenone (**1a**, 0.5 mmol) was reacted with I_2 (0.8 mmol) in DMSO (2 mL) at 100 °C for 45 mins and then added 2-aminobenzothioamide (**4a**, 0.5 mmol) at 100 °C for another 7 hours. However, **3aa** could not be got in this conditions. This result implied that 2-aminobenzothioamide (**4a**) is not intermediate.

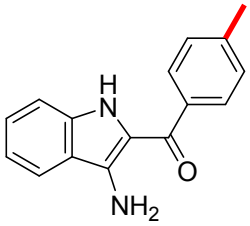
5. Characterization data for compounds 3



3aa

(3-amino-1H-indol-2-yl)(phenyl)methanone (3aa)

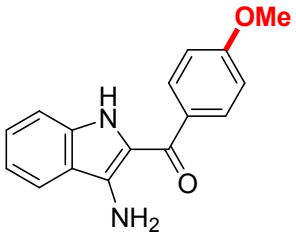
Yield 71%; 167.75 mg; yellow solid; mp 261–263℃; IR (KBr): 3451.83, 1748.66, 1654.10, 1611.92,1384.37, 1335.86, 796.81, 732.79 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.95 (br s, 1H), 7.75–7.68 (m, 2H), 7.53 (d, *J* = 8.4 Hz, 1H), 7.44–7.35 (m, 3H), 7.25 (t, *J* = 7.8 Hz, 1H), 7.12 (d, *J* = 8.4 Hz, 1H), 6.95 (t, *J* = 7.8 Hz, 1H), 5.65 (br s, 2H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 185.6, 139.4, 139.2, 137.8, 130.8, 128.5, 128.0, 127.7, 120.0, 118.7, 118.5, 117.3, 112.1.HRMS (ESI) m/z calcd for $C_{15}H_{13}N_2O^+$ $(M+H)^+$ 237.10224, found 237.10228.



3ba

(3-amino-1H-indol-2-yl)(p-tolyl)methanone (3ba)

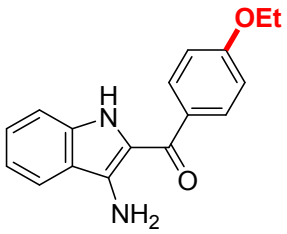
Yield 63%;157.69 mg; yellow solid; mp 147–149℃; IR (KBr): 3442.66, 3320.94, 1607.78, 1508.16, 1423.57, 1351.22, 1322.68, 1275.48, 746.55cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.73 (br s, 1H), 7.70 (d, *J* = 7.8 Hz, 2H), 7.58 (d, *J* = 7.8 Hz, 1H), 7.32 (t, *J* = 7.8 Hz, 1H), 7.28 (d, *J* = 7.8 Hz, 2H), 7.20 (d, *J* = 8.4 Hz, 1H), 7.02 (t, *J* = 7.8 Hz, 1H), 5.60 (br s, 2H), 2.40 (s, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 185.8, 141.5, 138.7, 137.6, 136.9, 129.4, 128.0, 127.9, 120.0, 118.9, 117.5, 112.2, 21.5. HRMS (ESI) m/z calcd for $C_{16}H_{15}N_2O^+$ $(M+H)^+$ 251.11789, found 251.11795.



3ca

(3-amino-1H-indol-2-yl)(4-methoxyphenyl)methanone (3ca)

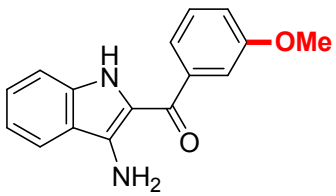
Yield 69%; 183.74 mg; yellow solid; mp 130–132℃ IR (KBr): 3444.31, 1619.35, 1565.13,1349.97, 1322.74, 1256.06, 1028.48, 842.70, 746.80 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.89 (br s, 1H), 7.79 (d, *J* = 8.4 Hz, 2H), 7.57 (d, *J* = 7.8 Hz, 1H), 7.31 (t, *J* = 7.8 Hz, 1H), 7.21 (d, *J* = 8.4 Hz, 1H), 7.01 (t, *J* = 7.2 Hz, 1H), 6.93 (d, *J* = 8.4 Hz, 2H), 5.60 (br s, 2H), 3.80 (s, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 185.0, 161.8, 138.7, 137.7, 132.1, 130.0, 127.8, 119.9, 119.0, 118.9, 117.6, 113.9, 112.2, 55.3.HRMS (ESI) m/z calcd for $C_{16}H_{15}N_2O_2^+$ $(M+H)^+$ 267.11280, found 267.11298.



3da

(3-amino-1H-indol-2-yl)(4-ethoxyphenyl)methanone (3da)

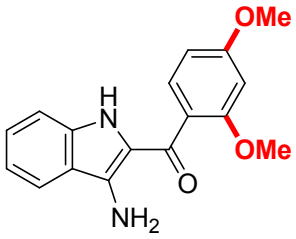
Yield 61%; 171.00 mg; yellow solid; mp 54–56℃; IR (KBr): 1730.53, 1679.50, 1606.13,1507.24, 1255.51, 1171.25, 747.65 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.83 (d, *J* = 9.0 Hz, 2H), 7.64 (br s, 1H), 7.61 (d, *J* = 8.4 Hz, 1H), 7.36 (t, *J* = 7.2 Hz, 1H), 7.24 (d, *J* = 8.4 Hz, 1H), 7.07 (t, *J* = 7.8 Hz, 1H), 7.00 (d, *J* = 8.4 Hz, 2H), 5.55 (br s, 2H), 4.14–4.09 (m, 2H), 1.46 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 185.2, 161.4, 138.4, 137.5, 132.0, 130.0, 127.9, 119.9, 119.2, 119.0, 117.6, 114.5, 112.2, 63.7, 14.7.HRMS (ESI) m/z calcd for $C_{17}H_{17}N_2O_2^+$ $(M+H)^+$ 281.12845, found 281.12845.



3ea

(3-amino-1H-indol-2-yl)(3-methoxyphenyl)methanone (3ea)

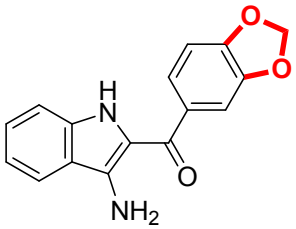
Yield 80%; 213.04 mg; yellow oil; IR (KBr): 1606.41, 1573.21, 1511.30,1482.04, 1434.66, 1323.45, 1282.25, 1244.34, 744.34 cm^{-1};^{1}H NMR (300 MHz, $CDCl_3$) δ 7.77 (br s, 1H), 7.58 (d, J = 8.1 Hz, 1H), 7.40 - 7.28 (m, 4H), 7.19 (d, J = 8.4 Hz, 1H), 7.08 – 6.98 (m, 2H), 5.28 (br s, 2H), 3.82 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 185.4, 159.9, 141.0, 139.2, 137.8, 129.8, 128.2, 120.0, 119.9, 118.9, 118.8, 117.4, 117.2, 112.6, 112.2, 55.4.HRMS (ESI) m/z calcd for $C_{16}H_{15}N_2O_2^+$ $(M+H)^+$ 267.11280, found 267.11298.



3fa

(3-amino-1H-indol-2-yl)(2,4-dimethoxyphenyl)methanone (3fa)

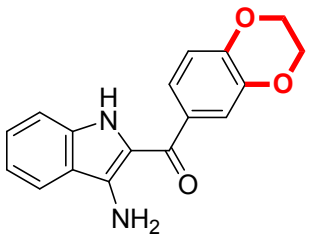
Yield 58%; 171.87mg; yellow solid; mp 129–131℃; IR (KBr): 3431.38, 1615.27, 1249.38, 829.17, 808.21, 760.43, 746.71 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.87 (br s, 1H), 7.57 (d, *J* = 7.8 Hz, 1H), 7.45 (d, *J* = 8.4 Hz, 1H), 7.30 (t, *J* = 7.8 Hz, 1H), 7.17 (d, *J* = 8.4 Hz, 1H), 7.00 (t, *J* = 7.2 Hz, 1H), 6.57 (d, *J* = 8.4 Hz, 1H), 6.55 (s, 1H), 5.56 (br s, 2H), 3.84 (s, 3H), 3.81 (s, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 184.5, 162.5, 157.6, 137.7, 132.2, 130.7, 127.9, 122.5, 120.0, 118.89, 118.85, 118.6, 112.1, 105.2, 99.3, 56.0, 55.5. HRMS (ESI) m/z calcd for $C_{17}H_{17}N_2O_3^+$ $(M+H)^+$ 297.12337, found 297.12360.



3ga

(3-amino-1H-indol-2-yl)(benzo[d][1,3]dioxol-5-yl)methanone (3ga)

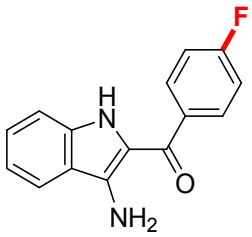
Yield 69%; 193.39 mg; yellow oil; IR (KBr): 3433.84, 1615.85, 1510.24, 1480.70, 1439.22, 1281.81, 1246.95, 1037.09, 746.37 cm^{-1};^{1}H NMR (300 MHz, $CDCl_3$) δ 7.68 (br s, 1H), 7.59 (d, J = 8.1 Hz, 1H), 7.44–7.29 (m, 3H), 7.27 – 7.20 (m, 1H), 7.09–7.01 (m, 1H), 6.90 (d, J = 8.1 Hz, 1H), 6.03 (s, 2H), 5.60 (br s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 184.4, 150.1, 148.1, 138.9, 137.7, 133.9, 128.0, 123.1, 120.0, 119.1, 117.3, 112.2, 108.4, 108.3, 101.6. HRMS (ESI) m/z calcd for $C_{16}H_{13}N_2O_3^+$ $(M+H)^+$ 281.09207, found 281.09244.



3ha

(3-amino-1H-indol-2-yl)(2,3-dihydrobenzo[b][1,4]dioxin-6-yl)methanone (3ha)

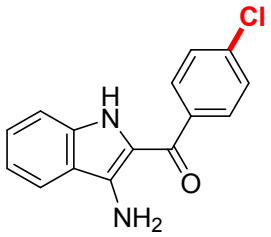
Yield 76%; 223.67 mg; yellow oil; IR (KBr): 3436.74, 1615.49, 1506.81, 1485.49, 1436.32, 1319.43, 1286.92, 1064.37, 744.59 cm^{-1};^{1}H NMR (300 MHz, $CDCl_3$) δ 7.78 (br s, 1H), 7.58 (d, J = 8.1 Hz, 1H), 7.41–7.28 (m, 3H), 7.23 (t, J = 8.4 Hz, 1H), 7.08–6.99 (m, 1H), 6.94 (d, J = 8.1 Hz, 1H), 5.61 (br s, 2H), 4.36–4.18 (m, 4H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 184.6, 146.2, 143.5, 138.8, 137.6, 133.1, 127.9, 121.7, 119.9, 119.0, 118.9, 117.5, 117.42, 117.35, 112.2, 64.5, 64.2. HRMS (ESI) m/z calcd for $C_{17}H_{15}N_2O_3^+$ $(M+H)^+$ 295.10772, found 295.10788.



3ia

(3-amino-1H-indol-2-yl)(4-fluorophenyl)methanone (3ia)

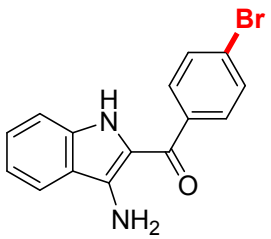
Yield 72%; 183.07 mg; yellow oil; IR (KBr): 3444.85, 3303.86, 1599.67, 1518.92, 1470.37, 1151.17, 844.56, 766.58 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.82–7.77 (m, 2H), 7.76 (br s, 1H), 7.59 (d, *J* = 7.8 Hz, 1H), 7.34 (t, *J* = 8.4 Hz, 1H), 7.21 (d, *J* = 8.4 Hz, 1H), 7.15–7.10 (m, 2H), 7.03 (t, *J* = 7.8 Hz, 1H), 5.69 (br s, 2H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 184.2, 164.2 (d, *J* = 250.5 Hz,$^1J_{CF}$), 139.6, 138.0, 135.7, 130.0 (d, *J* = 9.0 Hz,$^3J_{CF}$), 128.3, 120.0, 119.1, 118.7, 117.2, 115.7 (d, *J* = 22.5 Hz,$^2J_{CF}$), 112.3. HRMS (ESI) m/z calcd for $C_{15}H_{12}FN_2O^+$ $(M+H)^+$ 255.09282, found 255.09288.



3ja

(3-amino-1H-indol-2-yl)(4-chlorophenyl)methanone (3ja)

Yield 75%; 203.04 mg; yellow solid; mp 117–119℃; IR (KBr): 3416.68, 1617.07, 1562.11, 1508.19, 1314.56, 744.78 cm^{-1}; ^{1}H NMR (600 MHz, $CDCl_3$) δ 7.72 (d, *J* = 7.8 Hz, 3H), 7.59 (d, *J* = 7.8 Hz, 1H), 7.42 (d, *J* = 8.4 Hz, 2H), 7.34 (t, *J* = 7.8 Hz, 1H), 7.21 (d, *J* = 8.4 Hz, 1H), 7.04 (t, *J* = 7.8 Hz, 1H), 5.71 (br s, 2H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 184.1, 139.9, 138.2, 137.8, 137.1, 129.3, 128.9, 128.5, 120.1, 119.1, 118.7, 117.2, 112.3. HRMS (ESI) m/z calcd for $C_{15}H_{12}ClN_2O^+$ $(M+H)^+$ 271.06327, found 271.06360.

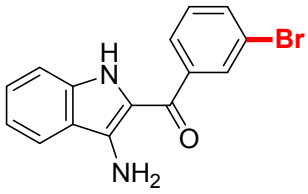


3ka

(3-amino-1H-indol-2-yl)(4-bromophenyl)methanone (3ka)

Yield 72%; 233.22 mg; yellow solid; mp 99–101℃; IR (KBr): 3405.27, 3313.95, 1610.57, 1585.95, 1508.35, 1476.45, 1435.64, 1349.89, 1315.84,1268.90, 744.90 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.69–7.56 (m, 6H), 7.39–7.31 (m, 1H), 7.26–7.19 (m, 1H), 7.09–7.01 (m, 1H), 5.71 (br s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 184.2, 139.8, 138.3, 138.2,

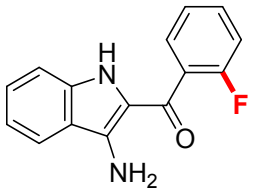
131.9, 129.5, 128.5, 125.6, 120.1, 119.2, 118.8, 117.2, 112.3. HRMS (ESI) m/z calcd for $C_{15}H_{12}BrN_2O^+$ $(M+H)^+$ 315.01275, found 315.01294.



3la

(3-amino-1H-indol-2-yl)(3-bromophenyl)methanone (3la)

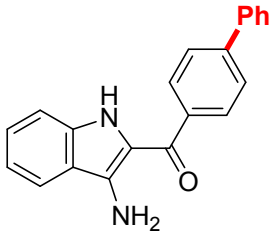
Yield 71%; 223.77 mg; yellow oil; IR (KBr): 3437.62, 1617.84, 1509.95, 741.89 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.01 (s, 1H), 7.82 (s, 1H), 7.61 (d, *J* = 7.8 Hz, 1H), 7.53 (d, *J* = 7.8 Hz, 1H), 7.50 (d, *J* = 7.8 Hz, 1H), 7.28 (t, *J* = 7.8 Hz, 1H), 7.21 (t, *J* = 7.8 Hz, 1H), 7.15 (d, *J* = 8.4 Hz, 1H), 6.97 (t, *J* = 7.2 Hz, 1H), 5.77 (br s, 2H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 183.3, 141.1, 140.3, 138.3, 133.6, 130.7, 130.1, 128.5, 126.3, 122.6, 120.1, 118.9, 118.3, 117.0, 112.3. HRMS (ESI) m/z calcd for $C_{15}H_{12}BrN_2O^+$ $(M+H)^+$ 315.01275, found 315.01291.



3ma

(3-amino-1H-indol-2-yl)(2-fluorophenyl)methanone (3ma)

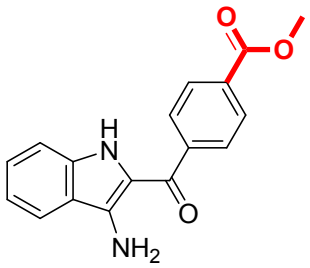
Yield 53%; 134.76 mg; yellow oil; IR (KBr): 3438.99, 1614.90, 1513.23, 1480.92, 1434.49, 747.52 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.64–7.55 (m, 3H), 7.52–7.42 (m, 1H), 7.38–7.15 (m, 4H), 7.02 (t, J = 7.8 Hz, 1H), 4.96 (br s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 181.3, 158.9 (d, *J* = 248.2 Hz, $^1J_{CF}$), 139.7, 138.3, 132.2 (d, *J* = 8.3 Hz, $^1J_{CF}$), 129.7 (d, *J* = 3.0 Hz, $^3J_{CF}$), 128.6, 127.8 (d, *J* = 15.8 Hz, $^2J_{CF}$), 124.71, 124.66, 120.2, 119.0, 118.5, 118.1, 116.4 (d, *J* = 21.7 Hz, $^2J_{CF}$), 112.2, HRMS (ESI) m/z calcd for $C_{15}H_{12}FN_2O^+$ $(M+H)^+$ 255.09282, found 255.09294.



3na

[1,1'-biphenyl]-4-yl(3-amino-1H-indol-2-yl)methanone (3na)

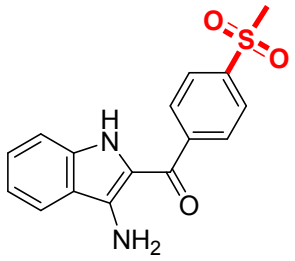
Yield 67%; 209.28 mg; yellow solid; mp 197–199℃;IR (KBr): 3468.07, 3339.18, 1603.81, 1511.01, 1478.36, 1436.97, 1357.28, 1327.71, 1282.17, 844.94, 744.53, 691.91 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.87 (d, J = 7.8 Hz, 2H), 7.78–7.68 (m, 3H), 7.61 (d, J = 6.6 Hz, 3H), 7.50–7.44 (m, 2H), 7.42–7.32 (m, 2H), 7.23 (d, J = 8.4 Hz, 1H), 7.06 (t, J = 7.2 Hz, 1H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 185.4, 143.8, 140.0, 139.2, 138.4, 137.8, 128.9, 128.4, 128.2, 128.0, 127.4, 127.2, 120.0, 119.1, 118.9, 117.6, 112.3. HRMS (ESI) m/z calcd for $C_{21}H_{17}N_2O^+$ $(M+H)^+$ 313.13354, found 313.13379.



3oa

methyl 4-(3-amino-1H-indole-2-carbonyl)benzoate (3oa)

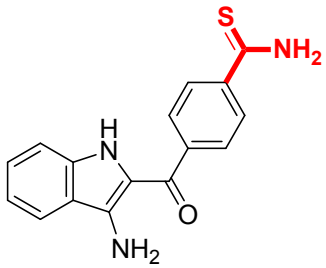
Yield 75%;220.73mg; yellow solid; mp 121–123℃; IR (KBr): 3444.15, 1721.13, 1616.24, 1511.62, 1434.30, 1278.30, 1105.33, 735.36 cm^{-1}; ^1H NMR (600 MHz, DMSO-d_6) δ 10.13 (br s, 1H), 8.14 (d, *J* = 8.4 Hz, 2H), 7.91 (d, *J* = 7.8 Hz, 3H), 7.32 (t, *J* = 7.2 Hz, 1H), 7.27 (d, *J* = 8.4 Hz, 1H), 7.01–6.93 (m, 3H), 3.94 (s, 3H). ^{13}C NMR (150 MHz, DMSO-d_6) δ 182.8, 165.9, 143.9, 141.4, 138.9, 131.0, 129.3, 128.3, 127.9, 121.5, 117.7, 116.4, 115.9, 112.7, 52.4. HRMS (ESI) m/z calcd for $C_{17}H_{15}N_2O_3^+$ $(M+H)^+$ 295.10772, found 295.10779.



3pa

(3-amino-1H-indol-2-yl)(4-(methylsulfonyl)phenyl)methanone (3pa)

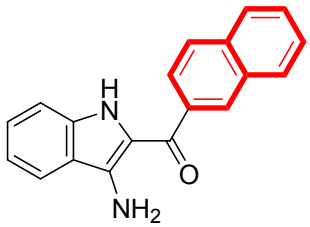
Yield 76%; 238.91 mg; yellow solid; mp 204–206℃; IR (KBr): 3448.72, 3334.13, 1607.28, 1517.88, 1303.56, 1282.32, 1144.78, 771.11, 752.19, 737.28 cm^{-1}; ^1H NMR (600 MHz, DMSO-d_6) δ 10.13 (br s, 1H), 8.09 (d, *J* = 8.4 Hz, 2H), 7.98 (d, *J* = 7.8 Hz, 2H), 7.89 (d, *J* = 8.4 Hz, 1H), 7.28 (t, *J* = 7.2 Hz, 1H), 7.23 (d, *J* = 8.4 Hz, 1H), 7.02 (br s, 2H), 6.92 (t, *J* = 7.2 Hz, 1H), 3.28 (s, 3H). ^{13}C NMR (150 MHz, DMSO-d_6) δ 182.3, 144.3, 142.1, 141.8, 139.1, 128.9, 128.2, 127.3, 121.6, 117.9, 117.7, 116.4, 112.7, 43.5. HRMS (ESI) m/z calcd for $C_{16}H_{15}N_2O_3S^+$ $(M+H)^+$ 315.07979, found 315.07986.



3qa

4-(3-amino-1H-indole-2-carbonyl)benzothioamide (3qa)

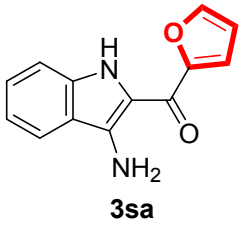
Yield 68%; 200.84 mg; yellow solid; mp 208–210℃;IR (KBr): 3453.08, 3422.66, 3311.09, 3179.96, 1685.44, 1610.32, 1511.56, 1474.96, 1435.17, 1273.40, 890.72, 743.73, 712.44 cm^{-1}; ^1H NMR (600 MHz, DMSO-d_6) δ 10.15 (s, 1H), 10.05 (s, 1H), 9.67 (s, 1H), 8.06 (d, *J* = 7.8 Hz, 2H), 7.88 (d, *J* = 7.8 Hz, 1H), 7.80 (d, *J* = 7.2 Hz, 2H), 7.32–7.23 (m, 2H), 6.97–6.89 (m, 3H). ^{13}C NMR (150 MHz, DMSO-d_6) δ 199.4, 183.1, 141.9, 141.1, 140.9, 138.8, 127.8, 127.6, 127.4, 121.4, 117.8, 117.7, 116.4, 112.7. HRMS (ESI) m/z calcd for $C_{16}H_{14}N_3OS^+$ $(M+H)^+$ 296.08521, found 296.08542.



3ra

(3-amino-1H-indol-2-yl)(naphthalen-2-yl)methanone (3ra)

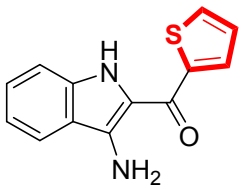
Yield 69%; 197.57 mg; yellow oil; IR (KBr): 3435.65, 1600.51, 1511.94,1482.93, 1356.18, 1318.88, 823.78, 744.14 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.19 (s, 1H), 7.90 (s, 1H), 7.86–7.75 (m, 4H), 7.56 (d, *J* = 7.8 Hz, 1H), 7.53–7.49 (m, 1H), 7.49–7.45 (m, 1H), 7.30 (t, *J* = 7.8 Hz, 1H), 7.18 (d, *J* = 8.4 Hz, 1H), 7.01 (t, *J* = 7.8 Hz, 1H), 5.65 (br s, 2H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 185.6, 139.3, 137.9, 136.7, 134.3, 132.4, 128.8, 128.6, 128.13, 128.09, 127.7, 127.5, 126.6, 124.5, 120.0, 118.9, 118.7, 117.6, 112.3. HRMS (ESI) m/z calcd for $C_{19}H_{15}N_2O^+$ $(M+H)^+$ 287.11789, found 287.11795.



3sa

(3-amino-1H-indol-2-yl)(furan-2-yl)methanone (3sa)

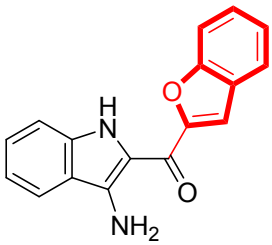
Yield 76%; 171.94 mg; yellow solid; mp 118–120℃; IR (KBr): 3454.13, 1614.23, 1572.51, 1513.47, 1467.12, 1430.72, 1404.79, 746.17 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.77 (s, 1H), 7.68 (s, 1H), 7.61 (d, *J* = 7.8 Hz, 1H), 7.40–7.34 (m, 2H), 7.31 (d, *J* = 8.4 Hz, 1H), 7.03 (t, *J* = 7.2 Hz, 1H), 6.61 (s, 1H), 6.01 (br s, 2H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 170.6, 154.4, 144.7, 141.2, 138.0, 128.4, 120.0, 118.7, 117.7, 116.4, 116.2, 112.4, 112.2. HRMS (ESI) m/z calcd for $C_{13}H_{11}N_2O_2^+$ $(M+H)^+$ 227.08150, found 227.08142.



3ta

(3-amino-1H-indol-2-yl)(thiophen-2-yl)methanone (3ta)

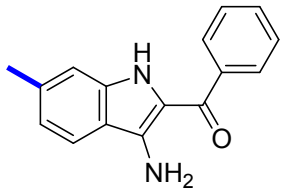
Yield 60%; 145.38 mg; yellow solid; mp 66–68℃; IR (KBr): 3429.35, 1615.97, 1510.72, 1488.65, 1425.96, 1384.89, 1353.90, 741.02, 717.40cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.84 (d, *J* = 3.0 Hz, 1H), 7.78 (s, 1H), 7.62–7.57 (m, 2H), 7.37 (t, *J* = 7.8 Hz, 1H), 7.29 (d, *J* = 8.4 Hz, 1H), 7.18–7.15 (m, 1H), 7.07 (t, *J* = 7.2 Hz, 1H), 5.84 (br s, 2H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 176.3, 144.2, 140.7, 138.6, 131.2, 130.0, 128.4, 127.9, 120.0, 119.4, 119.2, 117.0, 112.6. HRMS (ESI) m/z calcd for $C_{13}H_{11}N_2OS^+$ $(M+H)^+$ 243.05866, found 243.05858.



3ua

(3-amino-1H-indol-2-yl)(benzofuran-2-yl)methanone (3ua)

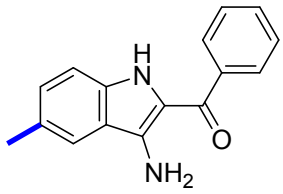
Yield 81%; 223.79 mg; yellow oil; mp 218–220℃; IR (KBr): 3474.54, 108.20, 1587.54, 1561.13, 1345.53, 1318.16, 870.58, 747.76, 737.18 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.89 (br s, 1H), 7.71 (d, *J* = 7.2 Hz, 1H), 7.67–7.63 (m, 1H), 7.60 (d, *J* = 8.4 Hz, 1H), 7.46 (t, *J* = 7.2 Hz, 1H), 7.42–7.37 (m, 1H), 7.35 (d, *J* = 7.8 Hz, 1H), 7.32 (t, *J* = 7.2 Hz, 1H), 7.03 (t, *J* = 7.2 Hz, 1H), 6.05 (br s, 2H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 171.1, 155.2, 154.7, 142.0, 138.3, 128.9, 127.2, 127.1, 123.9, 122.9, 120.1, 118.8, 117.5, 117.0, 112.3, 112.0. HRMS (ESI) m/z calcd for $C_{17}H_{13}N_2O_2^+$ $(M+H)^+$ 277.09715, found 277.09732.



3ab

(3-amino-6-methyl-1H-indol-2-yl)(phenyl)methanone (3ab)

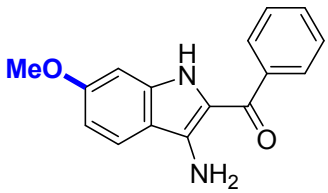
Yield 63%; 157.69 mg; yellow solid; mp 207–209℃; IR (KBr): 3446.62, 1610.54, 1587.18, 1568.97, 1356.32,1320.36, 1276.03, 806.54, 792.91, 758.30, 733.30 cm^{-1}; ^1H NMR (300 MHz, $CDCl_3$) δ 7.81–7.74 (m, 2H), 7.59–7.43 (m, 5H), 6.97 (s, 1H), 6.89–6.84 (m, 1H), 5.63 (br s, 2H), 2.41 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 185.3, 139.8, 139.5, 138.8, 138.5, 130.9, 128.7, 127.7, 121.1, 119.7, 117.3, 116.8, 111.8, 22.1. HRMS (ESI) m/z calcd for $C_{16}H_{15}N_2O^+$ $(M+H)^+$ 251.11789, found 251.11795.



3ac

(3-amino-5-methyl-1H-indol-2-yl)(phenyl)methanone (3ac)

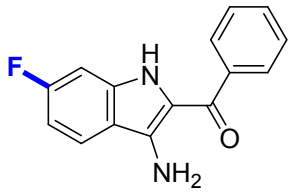
Yield 68%; 170.20 mg; yellow oil; IR (KBr): 3444.86, 1727.52, 1664.67, 1601.04, 1518.10, 1483.01, 1444.94, 1272.43, 717.38 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ7.80–7.72 (m, 2H), 7.61 (s, 1H), 7.46 (d, *J* = 7.8 Hz, 3H), 7.34 (s, 1H), 7.14 (d, *J* = 8.4 Hz, 1H), 7.08 (d, *J* = 8.4 Hz, 1H), 5.60 (br s, 2H), 2.39 (s, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 185.7, 139.7, 138.9, 136.5, 130.9, 130.2, 128.7, 128.3, 127.8, 119.2, 118.9, 117.8, 112.0, 21.3. HRMS (ESI) m/z calcd for $C_{16}H_{15}N_2O^+$ $(M+H)^+$ 251.11789, found 251.11784.



3ad

(3-amino-6-methoxy-1H-indol-2-yl)(phenyl)methanone (3ad)

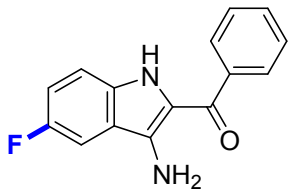
Yield 73%; 194.40 mg; yellow oil; IR (KBr): 3443.61, 1625.65, 1498.94, 1462.11, 1352.23, 1279.08, 821.42, 792.94, 752.99 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 7.79 (br s, 1H), 7.74 (d, *J* = 6.6 Hz, 2H), 7.49–7.40 (m, 4H), 6.66–6.62 (m, 1H), 6.56 (s, 1H), 5.69 (br s, 2H), 3.74 (s, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 184.3, 161.0, 139.8, 139.5, 130.7, 128.6, 127.6, 121.1, 117.1, 112.9, 110.5, 93.5, 55.3. HRMS (ESI) m/z calcd for $C_{16}H_{15}N_2O_2^+$ $(M+H)^+$ 267.11280, found 267.11280.



3ae

(3-amino-6-fluoro-1H-indol-2-yl)(phenyl)methanone (3ae)

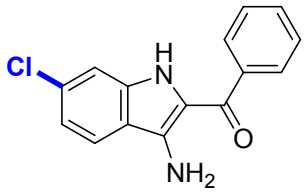
Yield 54%; 137.30 mg; yellow oil; IR (KBr): 3443.04, 1513.50, 1324.44, 1280.47, 836.04, 794.09, 750.54, 727.91 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 8.10 (br s, 1H), 7.70 (d, *J* = 7.2 Hz, 2H), 7.52–7.47 (m, 1H), 7.46–7.38 (m, 3H), 6.79 (d, *J* = 9.6 Hz, 1H), 6.72 (t, *J* = 9.0 Hz, 1H), 5.70 (br s, 2H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 185.1, 163.4 (d, *J* = 243.0 Hz,$^1J_{CF}$), 139.3, 138.0 (d, *J* = 13.5 Hz,$^3J_{CF}$), 130.9, 128.6, 127.6, 121.6 (d, *J* = 12.0 Hz,$^3J_{CF}$), 117.7, 115.4, 108.4 (d, *J* = 25.5 Hz,$^2J_{CF}$), 97.9 (d, *J* = 27.0 Hz,$^2J_{CF}$). HRMS (ESI) m/z calcd for $C_{15}H_{12}FN2O+$ $(M+H)+$ 255.09282, found 255.09294.



3af

(3-amino-5-fluoro-1H-indol-2-yl)(phenyl)methanone (3af)

Yield 59%; 150.01 mg; yellow solid; mp 149–151℃; IR (KBr): 3429.74, 3354.49, 3300.25, 1630.62, 1522.46, 1466.28, 1440.30, 1345.06, 1265.59, 731.11 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ7.80–7.76 (m, 1H), 7.75 (s, 1H), 7.55–7.47 (m, 3H), 7.25–7.21 (m, 1H), 7.17–7.13 (m, 1H), 7.12–7.07 (m, 1H), 5.52 (br s, 2H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 186.1, 156.7 (d, *J* = 236.9 Hz,$^1J_{CF}$), 139.3, 138.6, 134.3, 131.2, 128.8, 127.8, 118.8, 118.6, 117.2 (d, *J* = 25.5 Hz,$^2J_{CF}$), 113.4, 104.5 (d, *J* = 22.5 Hz,$^2J_{CF}$). HRMS (ESI) m/z calcd for $C_{15}H_{12}FN_2O^+$ $(M+H)^+$ 255.09282, found 255.09294.

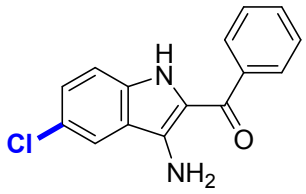


3ag

(3-amino-6-chloro-1H-indol-2-yl)(phenyl)methanone (3ag)

Yield 67%; 181.38 mg; yellow oil; IR (KBr): 3438.75, 1622.80, 1562.11, 1500.21, 1104.33, 1057.47, 698.85 cm^{-1};

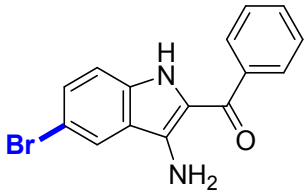
^{1}H NMR (600 MHz, $CDCl_3$) δ7.79–7.78 (m, 1H), 7.78–7.77 (m, 1H), 7.70 (s, 1H), 7.55–7.53 (m, 2H), 7.52–7.50 (m, 2H), 7.22–7.20 (m, 1H), 7.03–7.00 (m, 1H), 5.61 (br s, 2H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 185.8, 139.4, 138.6, 137.7, 134.1, 131.3, 128.9, 127.7, 121.1, 120.0, 117.7, 117.4, 112.0. HRMS (ESI) m/z calcd for $C_{15}H_{12}ClN_2O^+$ $(M+H)^+$ 271.06327, found 271.06351.



3ah

(3-amino-5-chloro-1H-indol-2-yl)(phenyl)methanone (3ah)

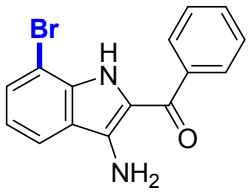
Yield 56%; 151.60 mg; yellow solid; mp 167–169℃; IR (KBr): 3434.17, 1515.83, 1340.42, 729.81cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.83 (s, 1H), 7.79–7.72 (m, 2H), 7.57–7.54 (m, 1H), 7.53–7.44 (m, 3H), 7.29–7.23 (m, 1H), 7.12 (d, *J* = 8.7 Hz, 1H), 5.52 (br s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 186.1, 139.3, 138.0, 135.8, 131.3, 128.8, 128.5, 127.7, 124.5, 119.6, 119.4, 118.2, 113.4. HRMS (ESI) m/z calcd for $C_{15}H_{12}ClN_2O^+$ $(M+H)^+$ 271.06327, found 271.06351.



3ai

(3-amino-5-bromo-1H-indol-2-yl)(phenyl)methanone (3ai)

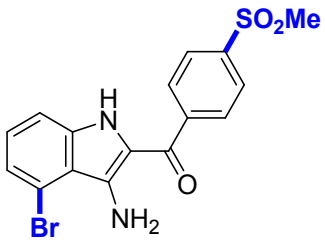
Yield 72%; 226.92 mg; yellow solid; mp 122–124℃; IR (KBr): 3368.75, 1514.87, 1338.60, 728.90 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.06 (s, 1H), 7.73–7.65 (m, 3H), 7.46–7.36 (m, 3H), 7.32–7.27 (m, 1H), 7.03–6.98 (m, 1H), 5.56 (br s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 185.9, 139.1, 137.9, 136.1, 131.1, 130.7, 128.7, 127.7, 122.5, 120.1, 117.9, 113.8, 111.4. HRMS (ESI) m/z calcd for $C_{15}H_{12}BrN_2O^+$ $(M+H)^+$ 315.01275, found 315.01285.



3aj

(3-amino-7-bromo-1H-indol-2-yl)(phenyl)methanone (3aj)

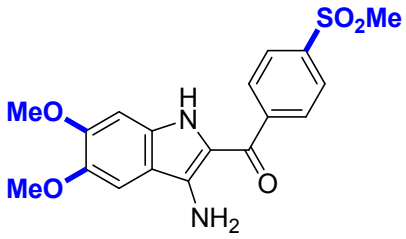
Yield 62%; 195.40 mg; yellow solid; mp 147–149℃; IR (KBr): 3462.33, 3295.03, 1635.98, 1615.05, 1522.15, 1477.01, 1321.39, 730.15, 709.11, 637.43 cm^{-1}; ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.88 – 7.80 (m, 2H), 7.70 (s, 1H), 7.61 – 7.48 (m, 5H), 6.93 (t, J = 7.8 Hz, 1H), 5.35 (br s, 2H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 185.9, 139.4, 139.3, 136.0, 131.4, 130.1, 128.9, 127.7, 120.0, 119.9, 119.3, 117.4, 105.7. HRMS (ESI) m/z calcd for $C_{15}H_{12}BrN_2O^+$ $(M+H)^+$ 315.01275, found 315.01291.



3qk

(3-amino-4-bromo-1H-indol-2-yl)(4-(methylsulfonyl)phenyl)methanone (3qk)

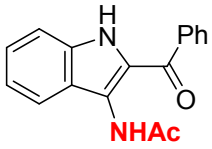
Yield 57%; 224.16 mg; yellow solid; mp 224–226℃; IR (KBr): 3426.58, 1645.85, 1025.65, 998.51, 765.96, 629.25 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ 10.66 (br s, 1H), 8.11 (d, J = 8.4 Hz, 2H), 7.96 (d, J = 8.4 Hz, 2H), 7.29 – 7.25 (m, 1H), 7.21–7.09 (m, 2H), 6.70 (br s, 2H), 3.31 (s, 3H). ^{13}C NMR (75 MHz, DMSO-d_6) δ 183.2, 143.8, 142.4, 140.3, 139.9, 128.9, 128.8, 127.3, 122.0, 116.9, 115.3, 114.8, 112.6, 43.4. HRMS (ESI) m/z calcd for $C_{16}H_{14}BrN_2O_3S^+$ $(M+H)^+$ 392.99030, found 392.99020.



3ql

(3-amino-5,6-dimethoxy-1H-indol-2-yl)(4-(methylsulfonyl)phenyl)methanone (3ql)

Yield 52%; 194.69 mg; yellow oil; IR (KBr): 3436.57, 2925.40, 1637.28, 1384.27, 1025.08, 994.51, 766.68, 603.75 cm^{-1}; ^1H NMR (300 MHz, DMSO-d_6) δ 9.83 (br s, 1H), 8.07 (d, J = 8.1 Hz, 2H), 7.96 (d, J = 6.9 Hz, 2H), 7.39 (s, 1H), 6.87 (br s, 2H), 6.69 (s, 1H), 3.78 (s, 3H), 3.77 (s, 3H), 3.30 (s, 3H). ^{13}C NMR (75 MHz, DMSO-d_6) δ 180.3, 152.2, 144.6, 143.9, 142.2, 141.8, 135.6, 128.8, 127.1, 116.5, 109.8, 102.0, 94.3, 55.7, 55.3, 43.4, 40.3. HRMS (ESI) m/z calcd for $C_{18}H_{19}N_2O_5S^+$ $(M+H)^+$ 375.10092, found 375.10117.

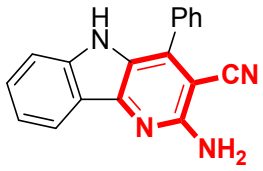


4a

N-(2-benzoyl-1H-indol-3-yl)acetamide (4a)

A 10 mL oven-dried reaction vessel was charged with 2-acyl-3-aminoindole (3aa, 0.5 mmol), Ac_2O (0.6 mmol, 1.2 equiv), Et_3N (1.5 mmol, 3.0 equiv), CH_2Cl_2 (1.0 mL) under air. The reaction vessel was stirred at room temperature for 24 h. The volatiles were removed under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate = 5:1) to yield the desired product 4a.

Yield 78%; 217.08 mg; yellow oil; IR (KBr): 3368.09, 1671.83, 1623.05, 1580.07, 1544.36, 1334.15, 1262.13, 732.05 cm^{-1}; ^1H NMR (600 MHz, $CDCl_3$) δ 9.57 (d, J = 27.0 Hz, 2H), 7.93 (d, J = 7.2 Hz, 1H), 7.68 (d, J = 6.6 Hz, 2H), 7.45 (s, 1H), 7.35 (s, 2H), 7.20 (s, 2H), 6.96 (s, 1H), 1.95 (s, 3H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 188.0, 168.7, 137.8, 136.4, 131.9, 128.4, 128.3, 126.8, 124.4, 124.2, 122.6, 120.9, 120.1, 112.2, 23.4. HRMS (ESI) m/z calcd for $C_{17}H_{15}N_2O_2^+$ $(M+H)^+$ 279.11280, found 279.11295.



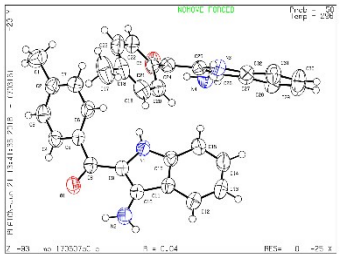
4b

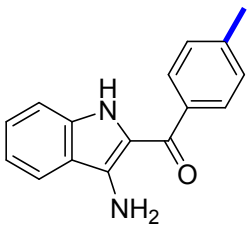
2-amino-4-phenyl-5H-pyrido[3,2-b]indole-3-carbonitrile (4b)

A 10 mL oven-dried reaction vessel was charged with 2-acyl-3-aminoindole (3aa, 0.5 mmol), malononitrile (1.5 mmol, 3.0 equiv), and TFA (0.5 mmol, 1.0 equiv) was heated at 80 °C in 5 mL of $CHCl_3$ in a sealed vessel for 3 h. The volatiles were removed under reduced pressure. The crude product was purified by column chromatography on silica gel (eluent: petroleum ether/EtOAc = 8/1) to afford the product 4b.

Yield 83%; 235.98 mg; yellow solid; mp 205–207℃; IR (KBr): 3376.50, 2258, 1625.31, 1552.05, 1459.78, 1380.74, 1211,17, 742,45, 687.44, 627.95 cm^{-1}; ^1H NMR (400 MHz, DMSO-d_6) δ 11.87 (br s, 1H), 8.21 (d, J = 8.0 Hz, 1H), 8.15 (d, J = 7.2 Hz, 2H), 7.68–7.54 (m, 6H), 7.28 (t, J = 7.2 Hz, 1H), 4.51 (br s, 2H). ^{13}C NMR (100 MHz, DMSO-d_6) δ 151.4, 148.1, 147.3, 142.7, 135.7, 130.4, 129.0, 128.7, 126.2, 121.3, 120.5, 119.4, 118.2, 113.0, 27.6. HRMS (ESI) m/z calcd for $C_{18}H_{13}N_4^+$ $(M+H)^+$ 285.11347, found 285.11368.

6. Crystallographic data and molecular structure of compounds 3ba and 3pa





3ba

***Figure S1*.** X-ray crystal structure of **3ba.**

Crystal Data for Compound **3ba**: CCDC 1850883 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic.

Datablock: mo_170607a_0m

Bond precision: C-C = 0.0042 A Wavelength=0.71073

Cell: a=9.7923(13) b=17.627(2) c=15.121(2)
alpha=90 beta=91.028(2) gamma=90
Temperature: 296 K

	Calculated	Reported
Volume	2609.6(6)	2609.6(6)
Space group	C c	C c
Hall group	C -2yc	C -2yc
Moiety formula	C16 H14 N2 O	?
Sum formula	C16 H14 N2 O	C32 H28 N4 O2
Mr	250.29	500.58
Dx,g cm-3	1.274	1.274
Z	8	4
Mu (mm-1)	0.081	0.081
F000	1056.0	1056.0
F000'	1056.41	
h,k,lmax	12,22,19	12,22,19
Nref	5697[2854]	5481
Tmin,Tmax	0.990,0.992	
Tmin'	0.990	

Correction method= Not given

Data completeness= 1.92/0.96 Theta(max)= 26.994

R(reflections)= 0.0374(4538) wR2(reflections)= 0.0949(5481)

S = 1.059 Npar= 345

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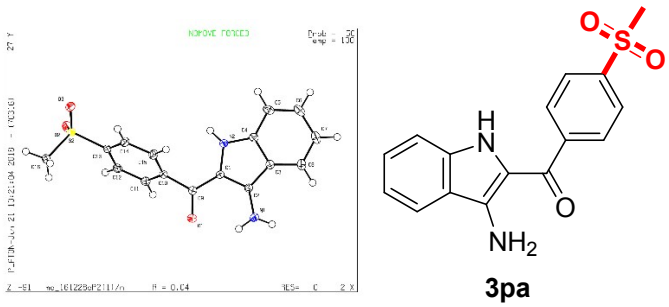
Click on the hyperlinks for more details of the test.



3pa

***Figure S2*.** X-ray crystal structure of **3pa.**

Crystal Data for Compound **3pa**: CCDC 1850886 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic.

Datablock: mo_161228c_0m

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Bond precision:  C-C = 0.0018 A          Wavelength=0.71073

Cell:            a=5.7269(7)     b=28.551(4)          c=8.8421(11)
                 alpha=90        beta=103.507(2)      gamma=90
Temperature:     100 K

                 Calculated                Reported
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Space group      P 21/n                    P2(1)/n
Hall group       -P 2yn                    ?
Moiety formula   C16 H14 N2 O3 S           ?
Sum formula      C16 H14 N2 O3 S           C16 H14 N2 O3 S
Mr               314.35                    314.35
Dx,g cm-3        1.485                     1.485
Z                4                         4
Mu (mm-1)        0.245                     0.245
F000             656.0                     656.0
F000'            656.78
h,k,lmax         8,40,12                   8,40,12
Nref             4082                      4071
Tmin,Tmax        0.952,0.976               0.953,0.976
Tmin'            0.952

Correction method= # Reported T Limits: Tmin=0.953 Tmax=0.976
AbsCorr = NONE

Data completeness= 0.997          Theta(max)= 30.000

R(reflections)= 0.0390( 3709)     wR2(reflections)= 0.0963( 4071)

S = 1.129                 Npar= 209
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The following ALERTS were generated. Each ALERT has the format
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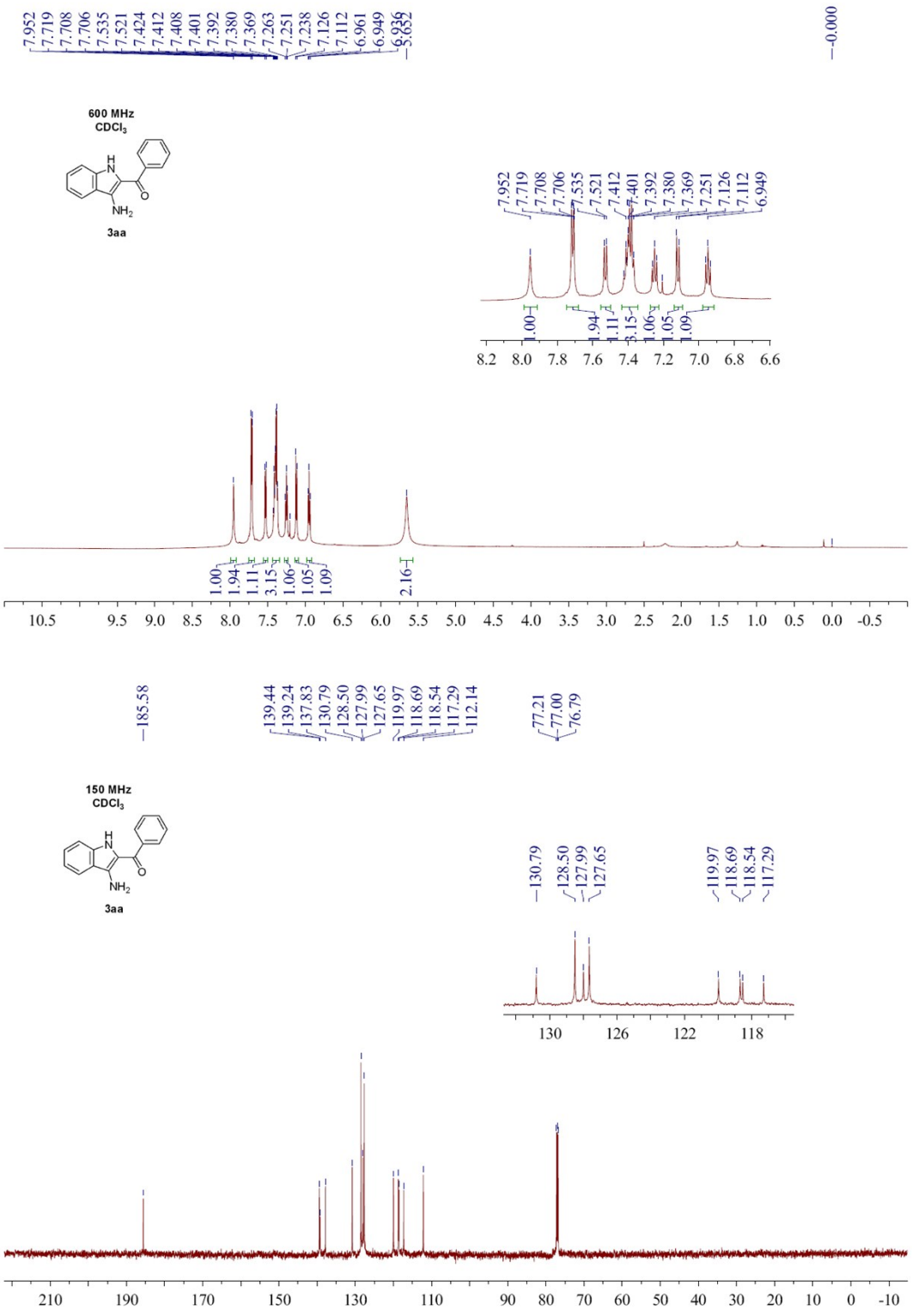
Click on the hyperlinks for more details of the test.

7. ^{1}H and ^{13}C NMR spectra of compounds 3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7.380
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7.263
7.251
7.238
7.126
7.112
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6.949
6.936
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1.00
1.94
1.11
3.15
1.06
1.05
1.09
2.16
150 MHz
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139.24
137.83
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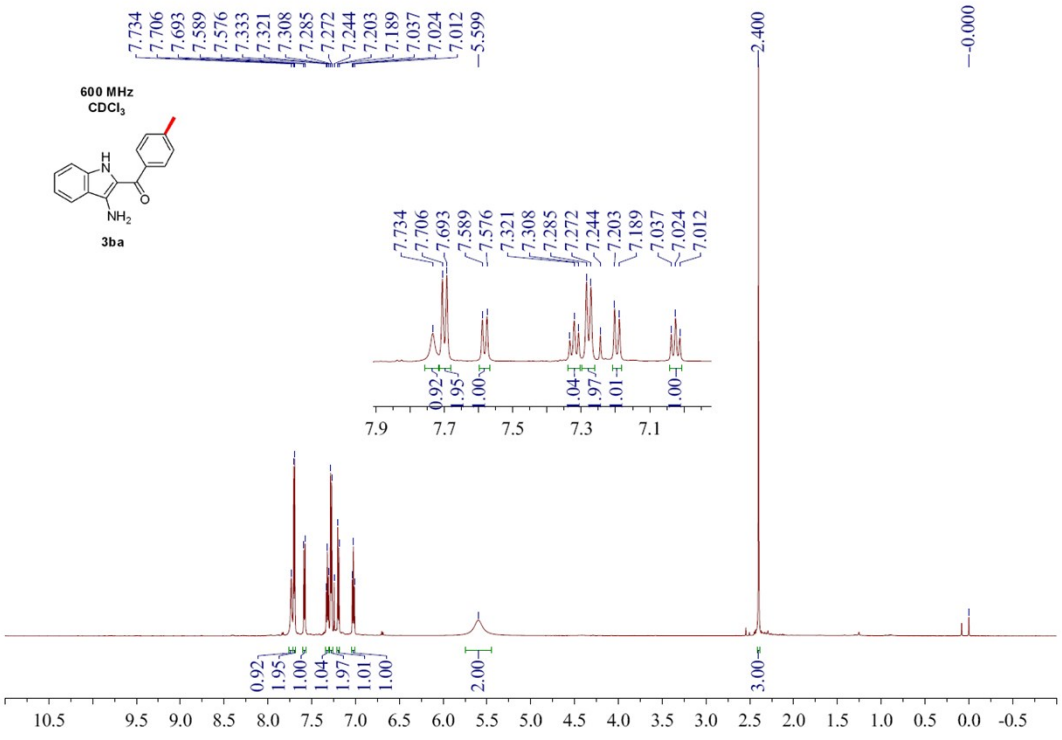
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7.203
7.189
7.037
7.024
7.012
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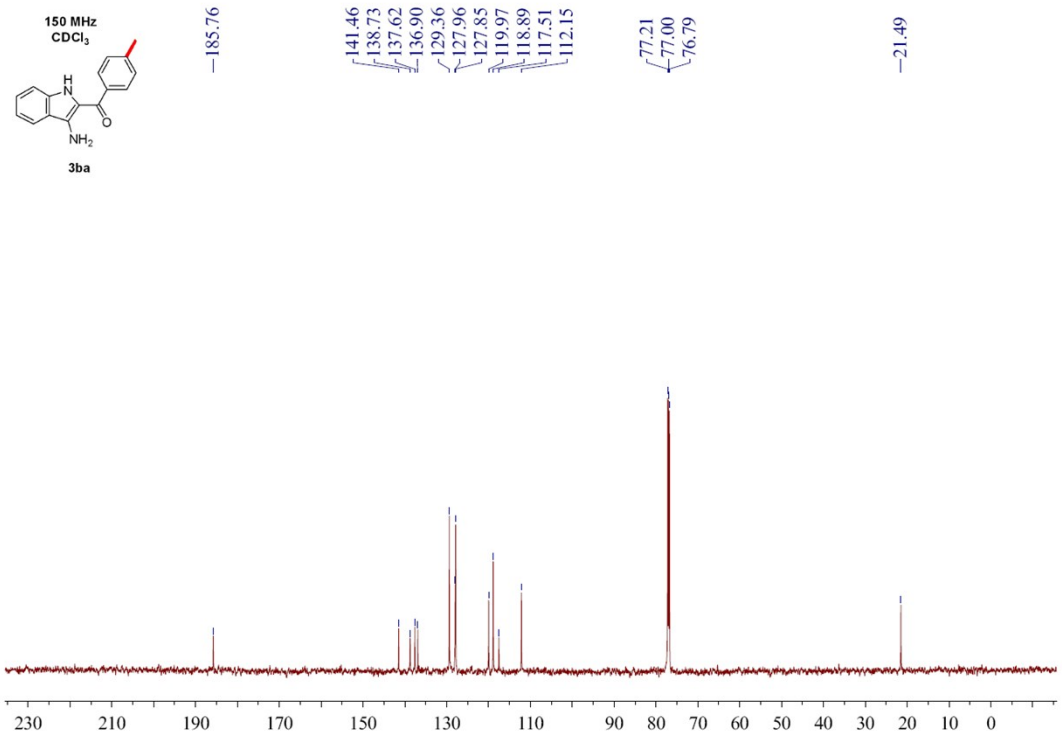
3.00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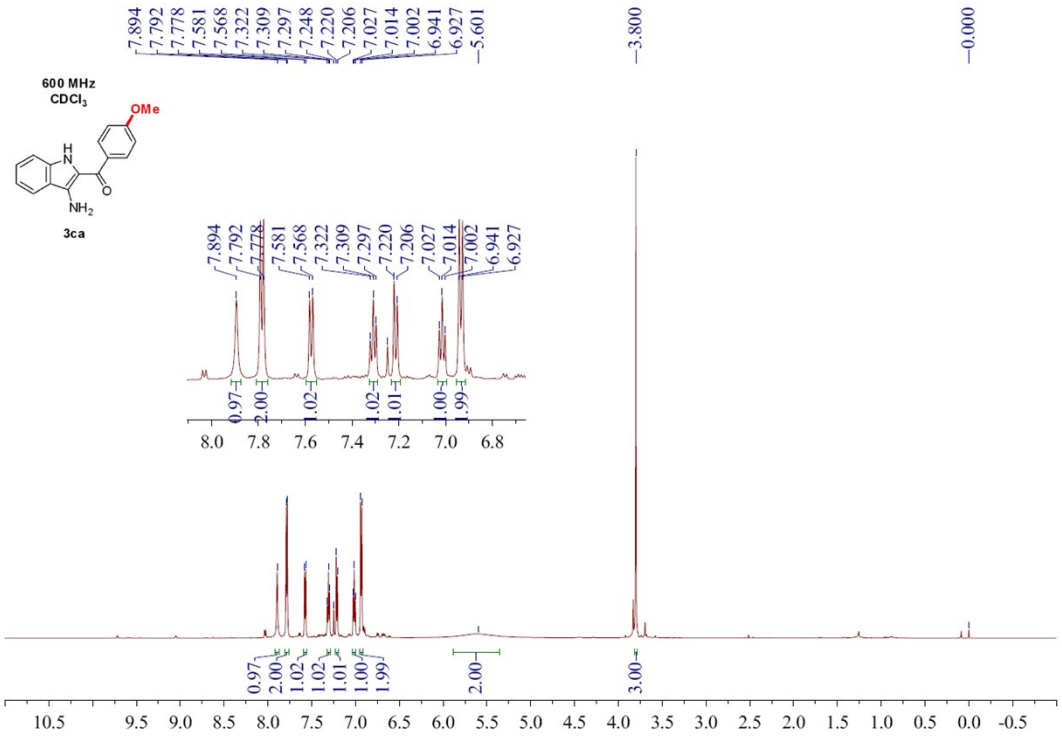
76.79
21.49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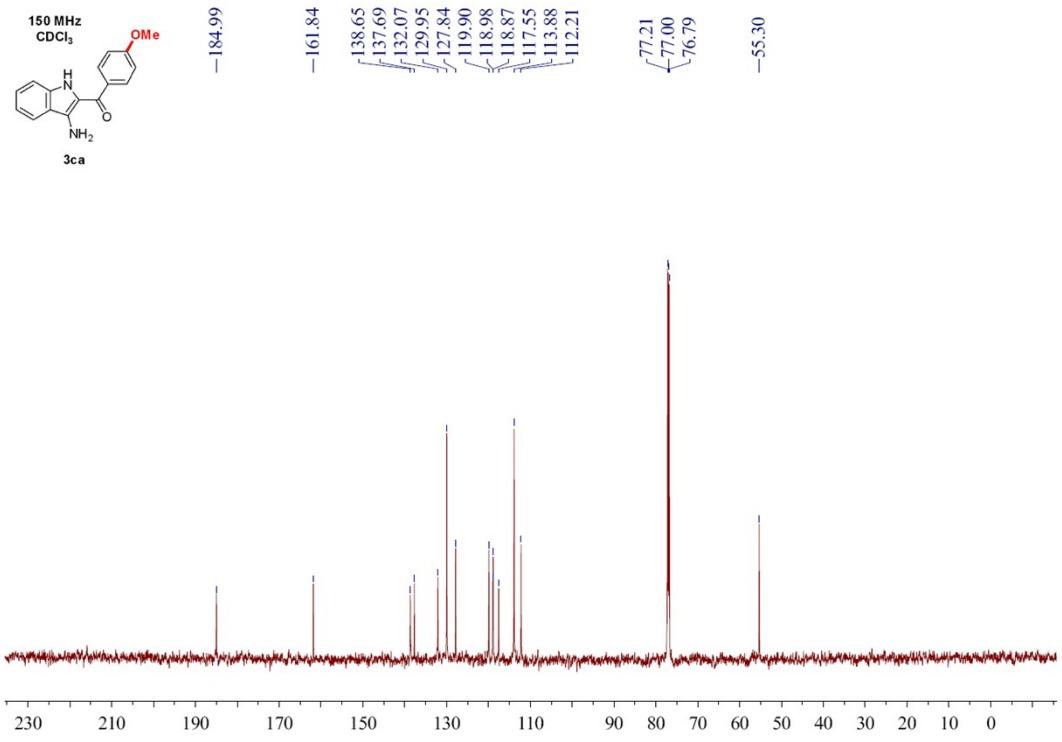
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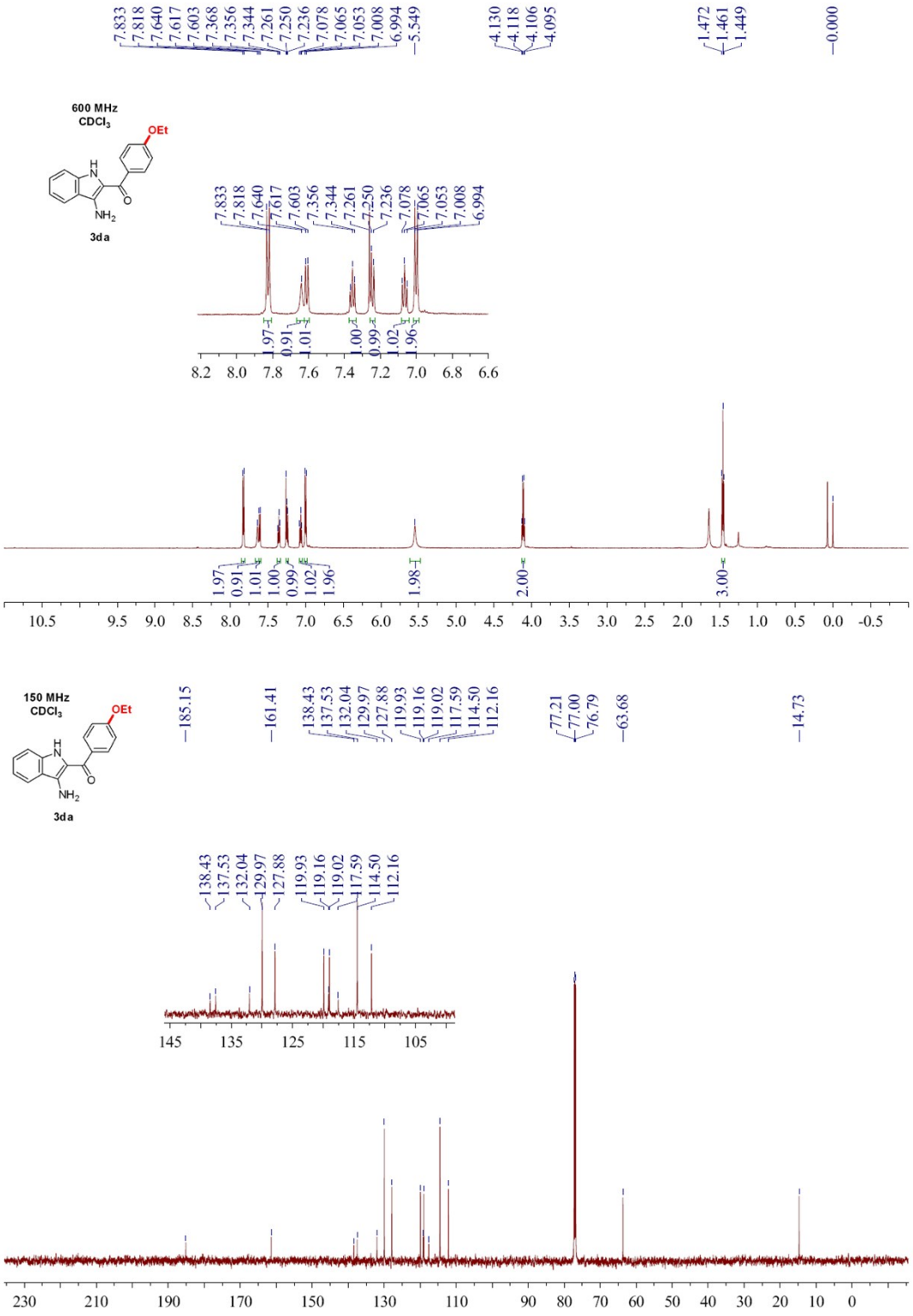
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1.00
0.99
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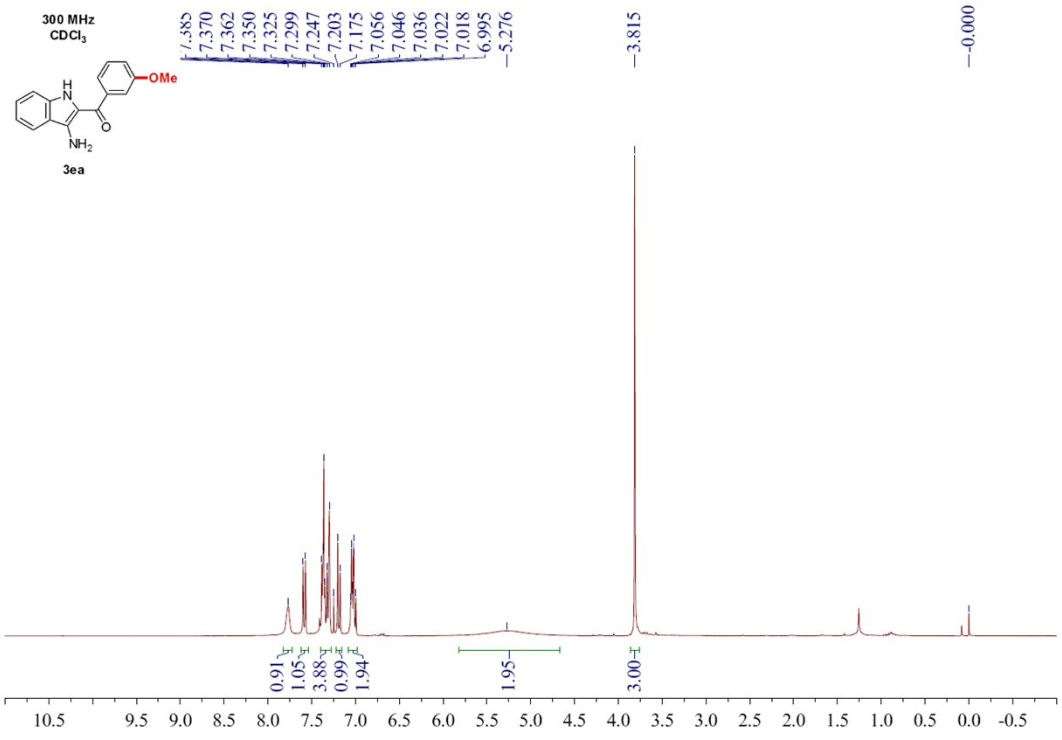
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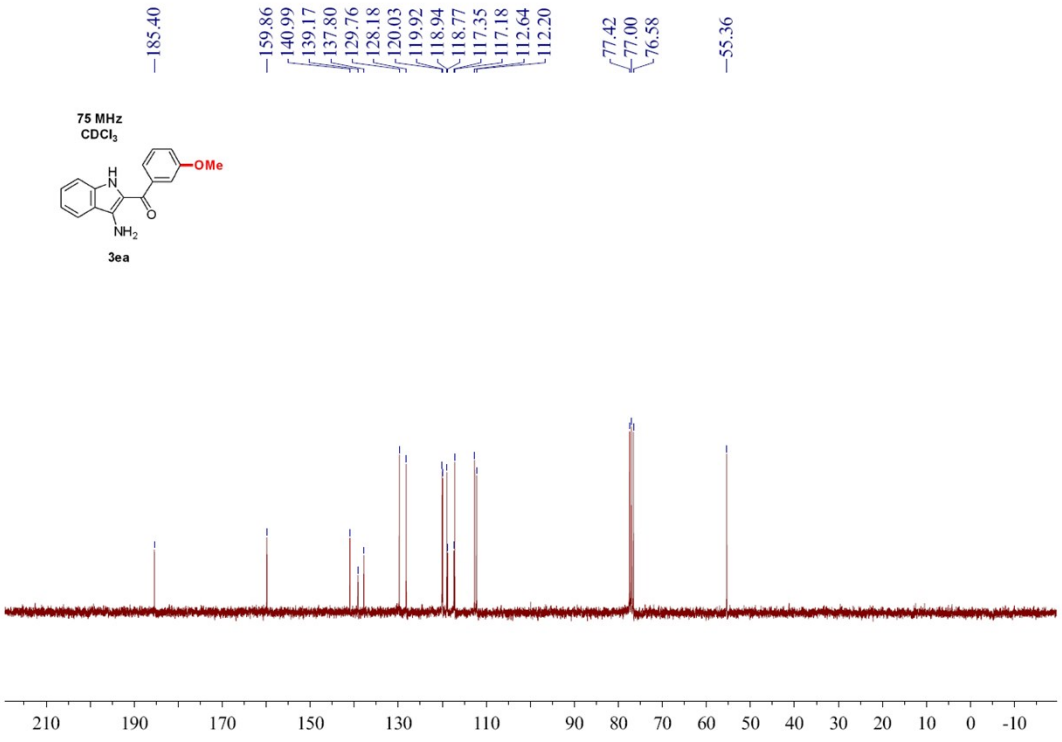
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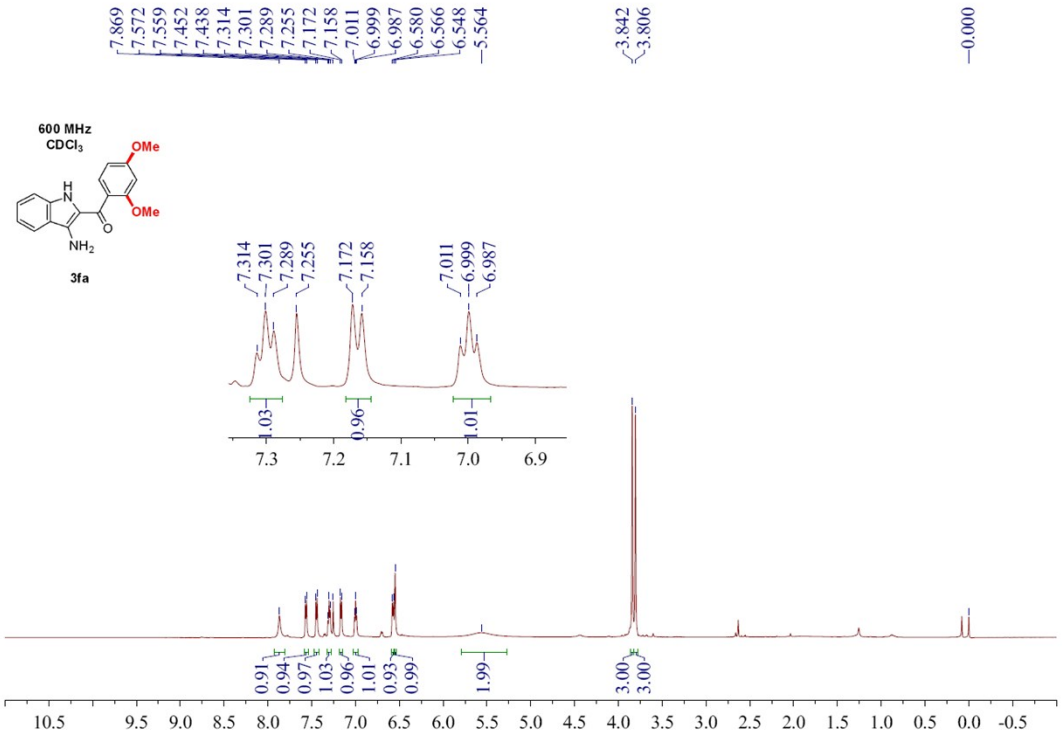
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600 MHz
CDCl3
OMe
OMe
NH2
3fa
7.314
7.301
7.289
7.255
7.172
7.158
7.011
6.999
6.987
1.03
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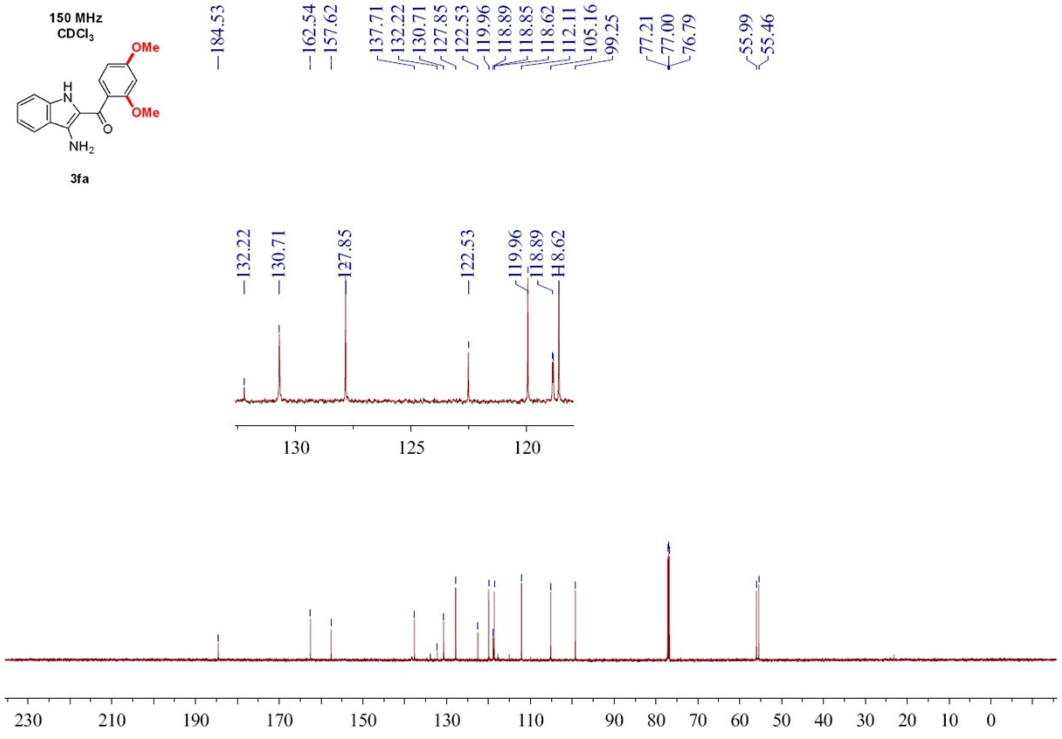
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CDCl3
OMe
OMe
NH2
3fa
184.53
162.54
157.62
137.71
132.22
130.71
127.85
122.53
119.96
118.89
118.85
118.62
112.11
105.16
99.25
77.21
77.00
76.79
55.99
55.46
132.22
130.71
127.85
122.53
119.96
118.89
118.62
130
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210
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130
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90
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70
60
50
40
30
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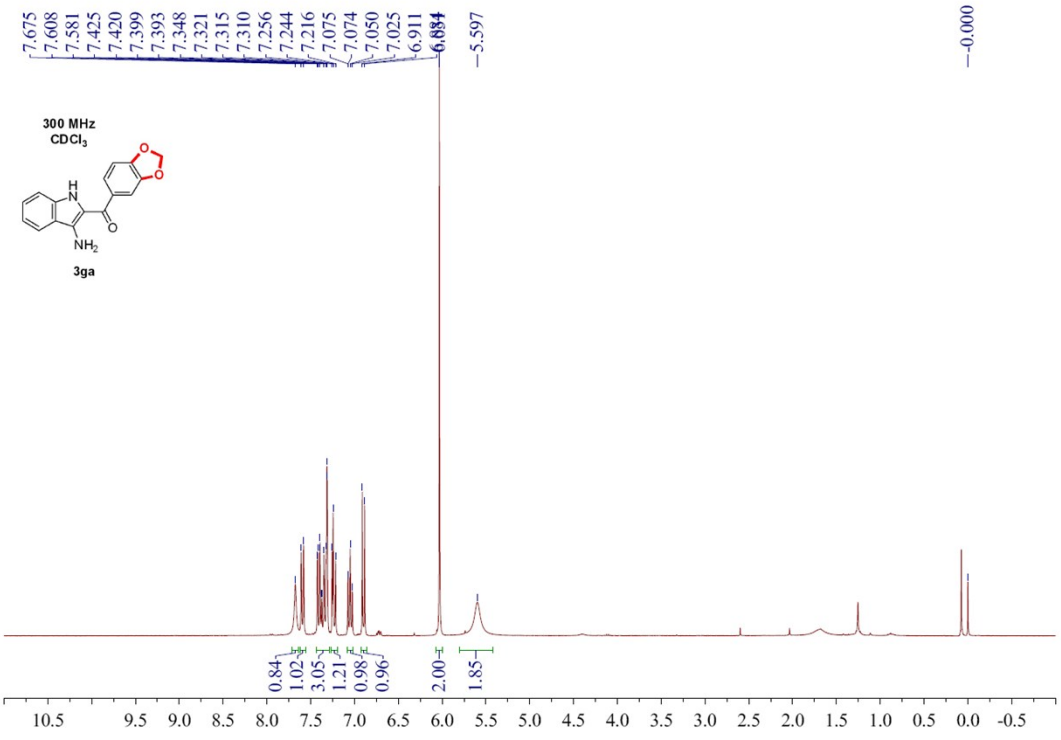
7.675
7.608
7.581
7.425
7.420
7.399
7.393
7.348
7.321
7.315
7.310
7.256
7.244
7.216
7.075
7.074
7.050
7.025
6.911
5.597
0.000
300 MHz
CDCl3
3ga
0.84
1.02
3.05
1.21
0.98
0.96
2.00
1.85
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
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3.5
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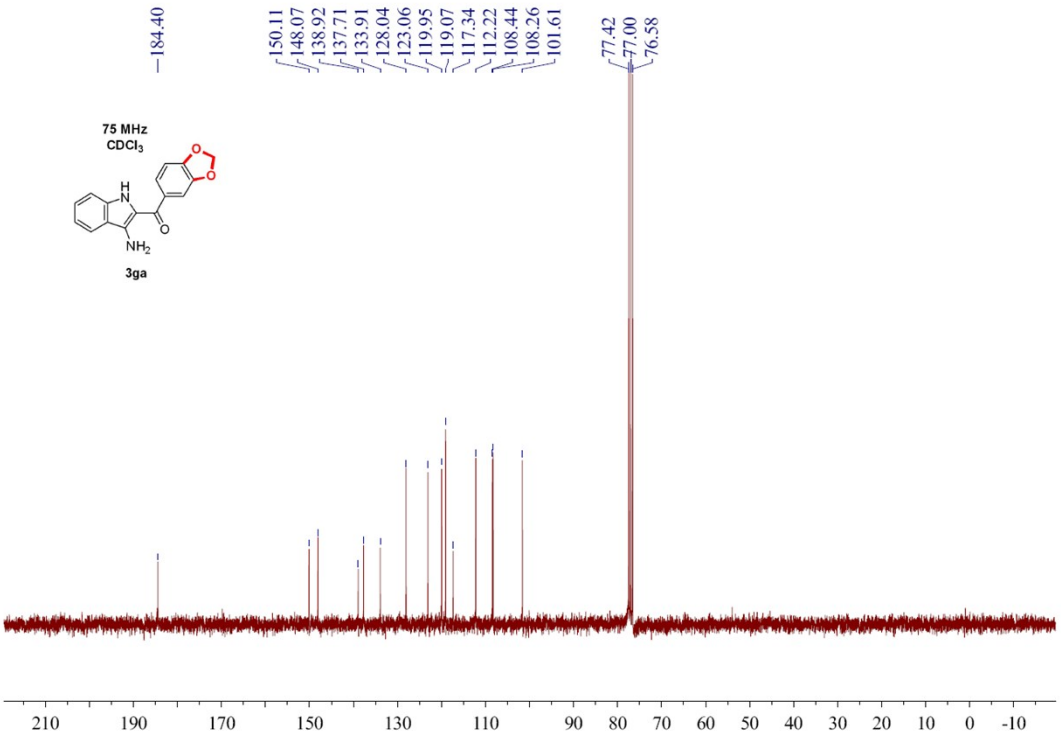
-0.5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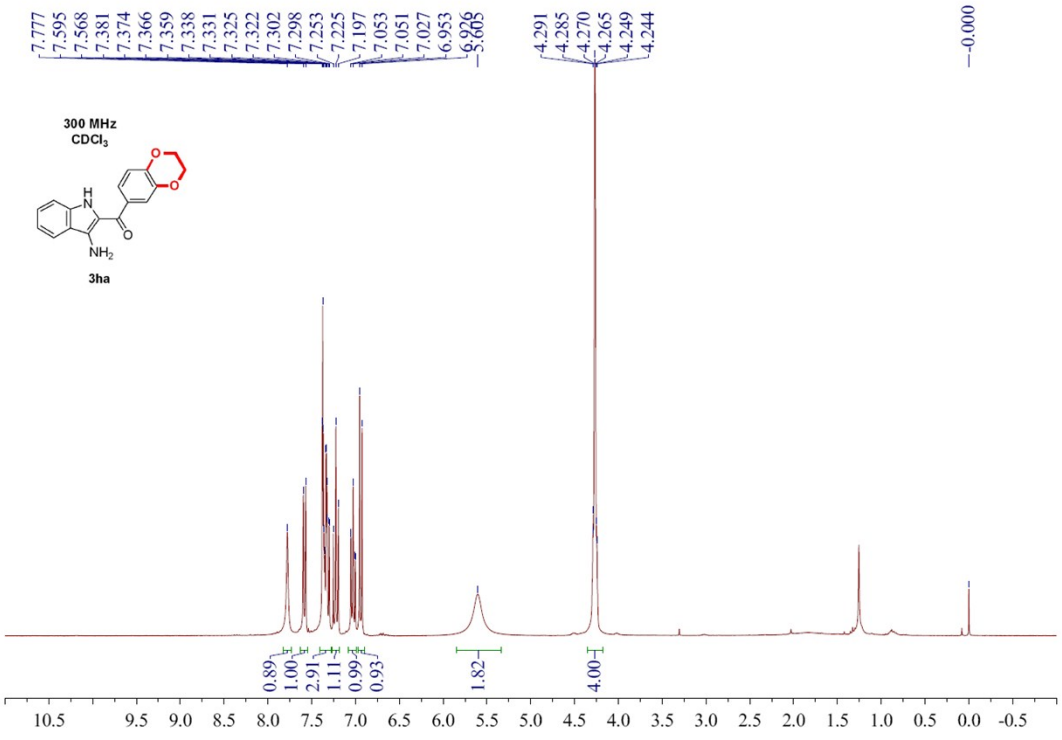
150.11
148.07
138.92
137.71
133.91
128.04
123.06
119.95
119.07
117.34
112.22
108.44
108.26
101.61
77.42
77.00
76.58
75 MHz
CDCl3
3ga
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10


7.777
7.595
7.568
7.381
7.374
7.366
7.359
7.338
7.331
7.325
7.322
7.302
7.298
7.253
7.225
7.197
7.053
7.051
7.027
6.953
6.926
3.605
4.291
4.285
4.270
4.265
4.249
4.244
0.000
300 MHz
CDCl3
NH2
3ha
0.89
1.00
2.91
1.11
0.99
0.93
1.82
4.00
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
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3.0
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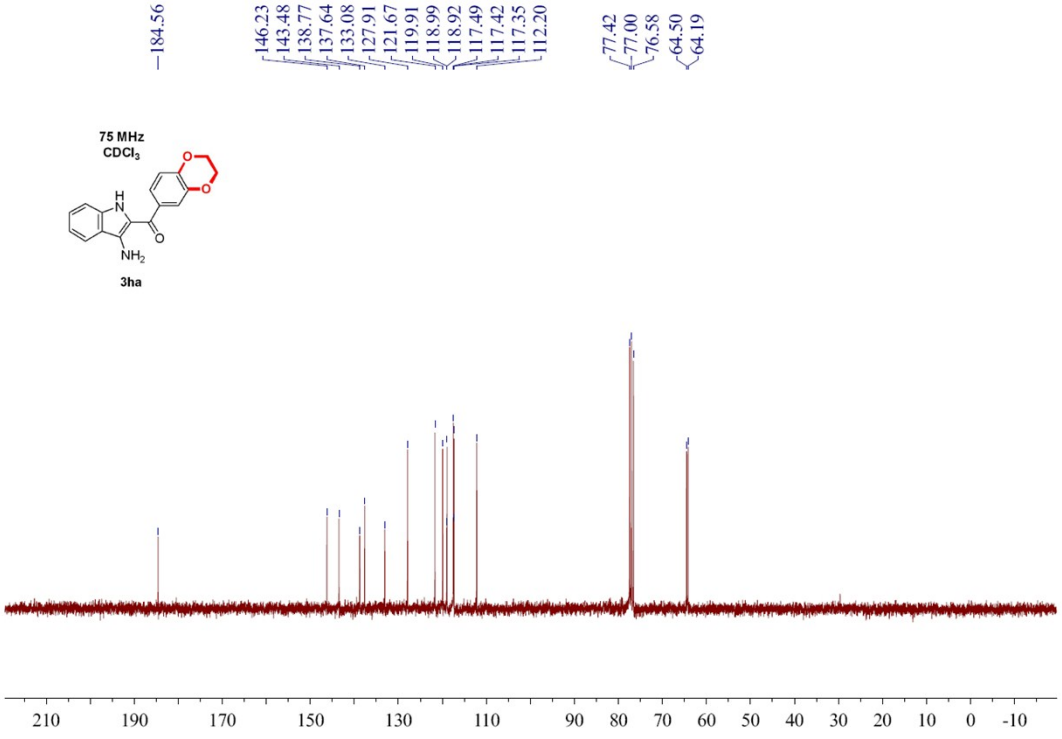
2.0
1.5
1.0
0.5
0.0
-0.5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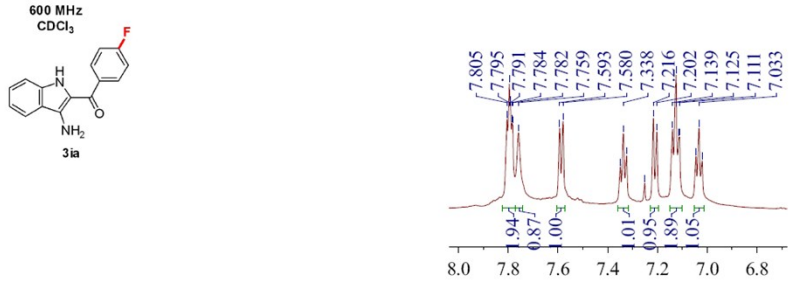
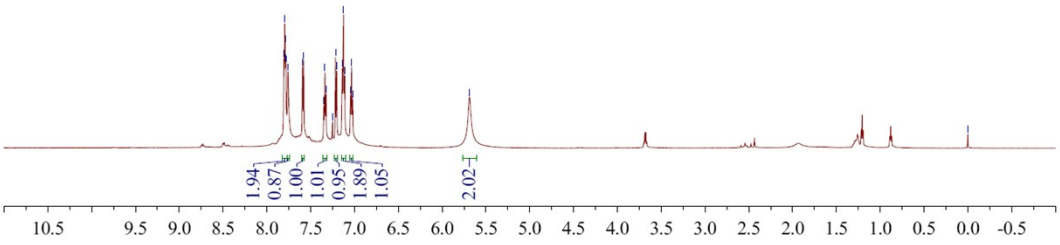
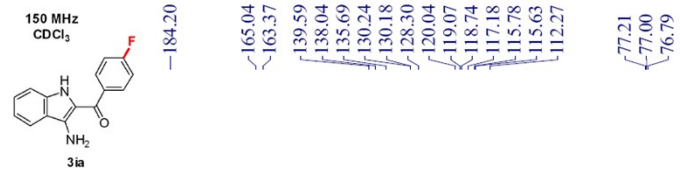
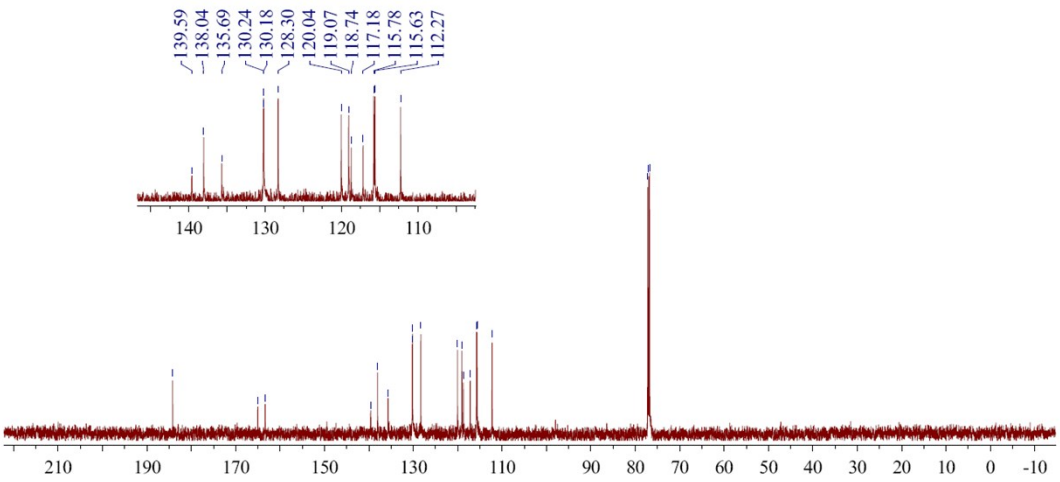
146.23
143.48
138.77
137.64
133.08
127.91
121.67
119.91
118.99
118.92
117.49
117.42
117.35
112.20
77.42
77.00
76.58
64.50
64.19
75 MHz
CDCl3
NH2
3ha
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10

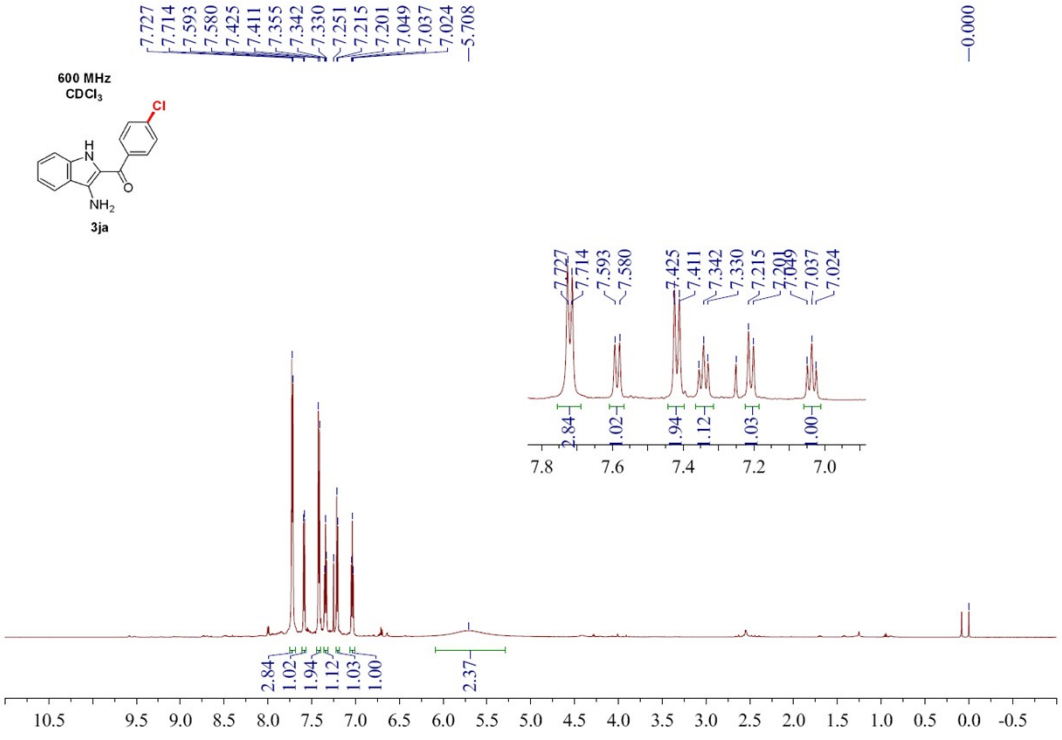

7.805
7.795
7.791
7.784
7.782
7.759
7.593
7.580
7.349
7.338
7.324
7.251
7.216
7.202
7.139
7.125
7.113
7.111
7.045
7.033
7.020
5.687
0.000

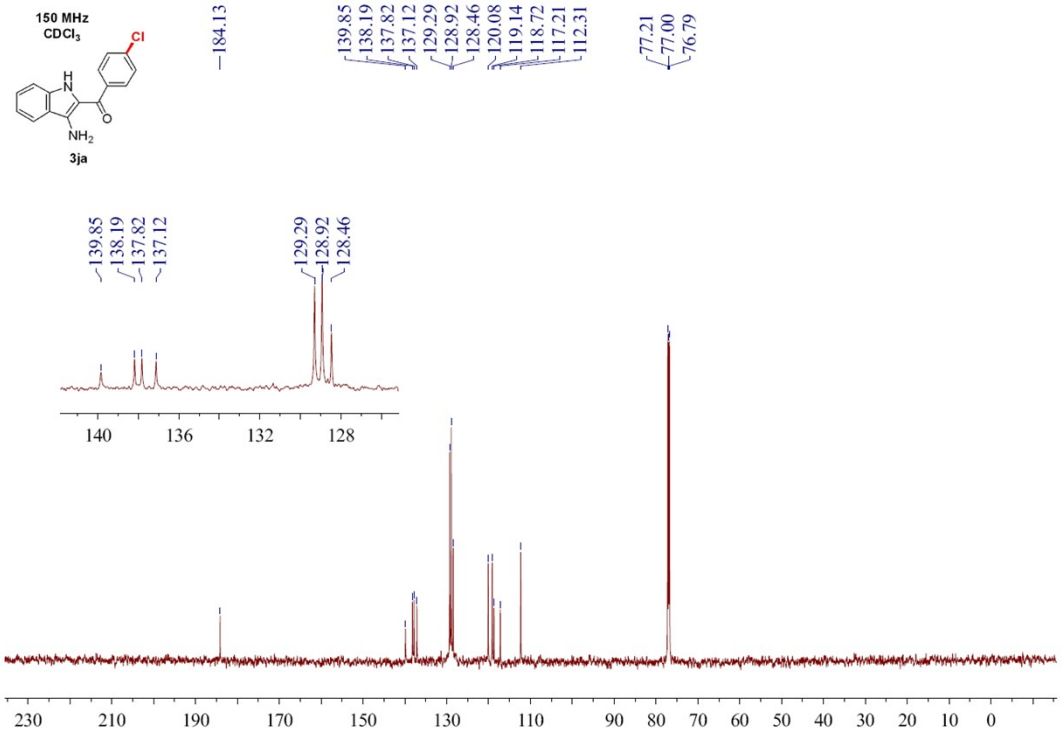

600 MHz
CDCl3
F
H
N
O
NH2
3ia
7.805
7.795
7.791
7.784
7.782
7.759
7.593
7.580
7.338
7.216
7.202
7.139
7.125
7.111
7.033
1.94
0.87
1.00
1.01
0.95
1.89
1.05
8.0
7.8
7.6
7.4
7.2
7.0
6.8

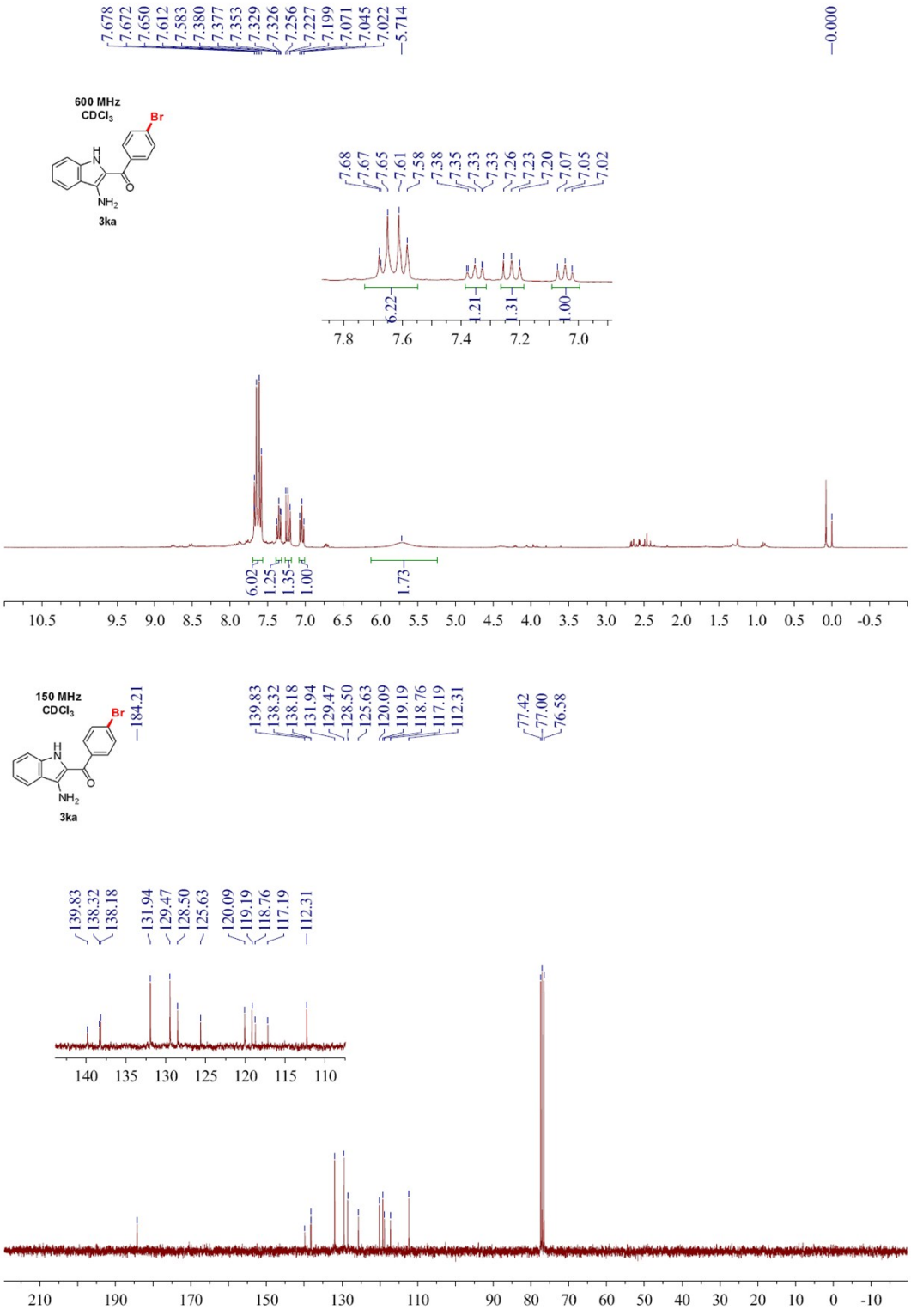

1.94
0.87
1.00
1.01
0.95
1.89
1.05
2.02
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5


150 MHz
CDCl3
F
H
N
O
NH2
3ia
184.20
165.04
163.37
139.59
138.04
135.69
130.24
130.18
128.30
120.04
119.07
118.74
117.18
115.78
115.63
112.27
77.21
77.00
76.79


139.59
138.04
135.69
130.24
130.18
128.30
120.04
119.07
118.74
117.18
115.78
115.63
112.27
140
130
120
110
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10


7.727
7.714
7.593
7.580
7.425
7.411
7.355
7.342
7.330
7.251
7.215
7.201
7.049
7.037
7.024
5.708
0.000
600 MHz
CDCl3
Cl
H
N
O
NH2
3ja
2.84
1.02
1.94
1.12
1.03
1.00
2.37
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
7.8
7.6
7.4
7.2
7.0


184.13
139.85
138.19
137.82
137.12
129.29
128.92
128.46
120.08
119.14
118.72
117.21
112.31
77.21
77.00
76.79
150 MHz
CDCl3
Cl
H
N
O
NH2
3ja
140
136
132
128
230
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0


7.678
7.672
7.650
7.612
7.583
7.380
7.377
7.353
7.329
7.326
7.256
7.227
7.199
7.071
7.045
7.022
5.714
0.000
600 MHz
CDCl3
Br
NH2
3ka
7.68
7.67
7.65
7.61
7.58
7.38
7.35
7.33
7.33
7.26
7.23
7.20
7.07
7.05
7.02
6.22
1.21
1.31
1.00
7.8
7.6
7.4
7.2
7.0
6.02
1.25
1.35
1.00
1.73
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
150 MHz
CDCl3
Br
NH2
3ka
184.21
139.83
138.32
138.18
131.94
129.47
128.50
125.63
120.09
119.19
118.76
117.19
112.31
77.42
77.00
76.58
140
135
130
125
120
115
110
210
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150
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90
80
70
60
50
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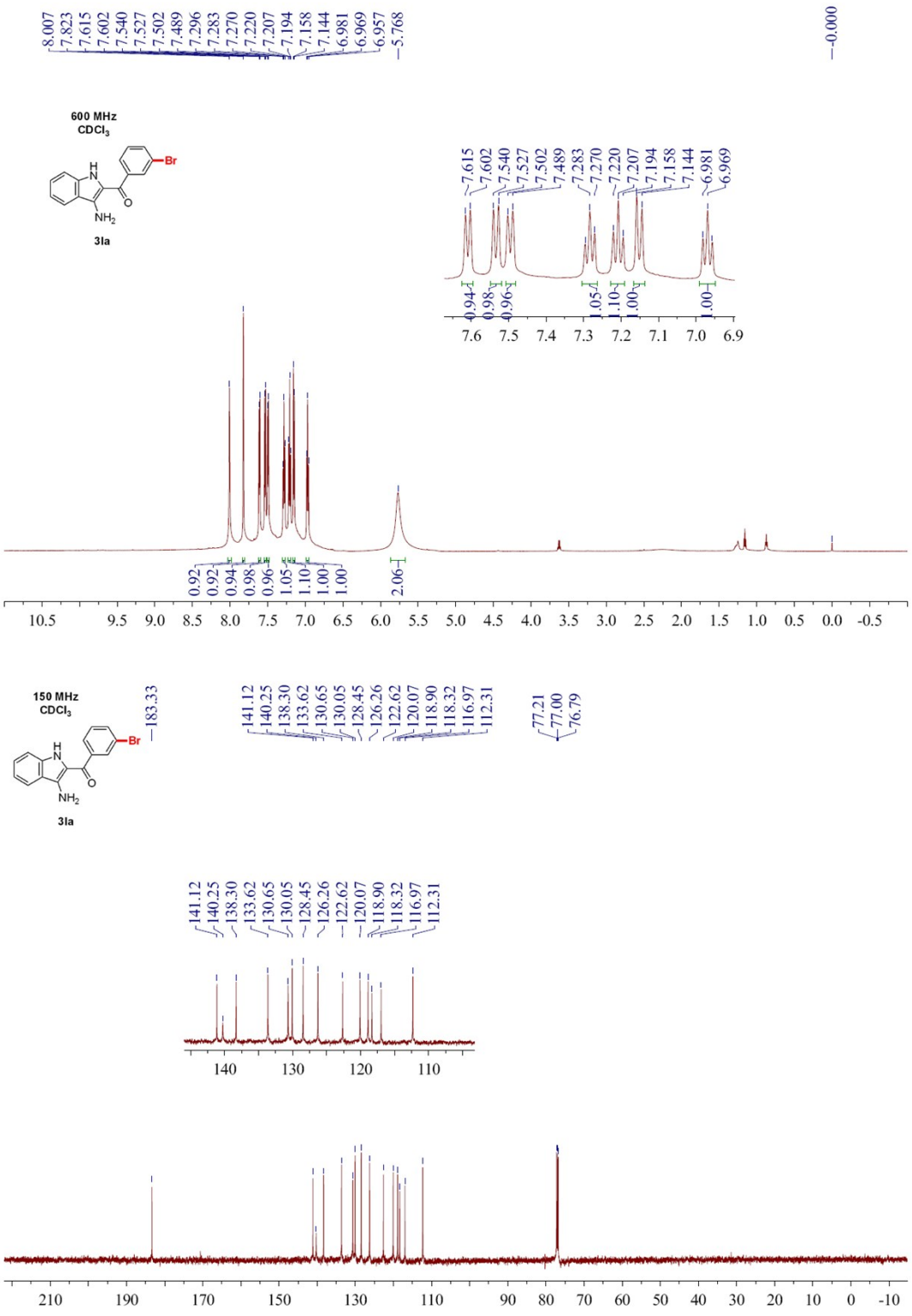
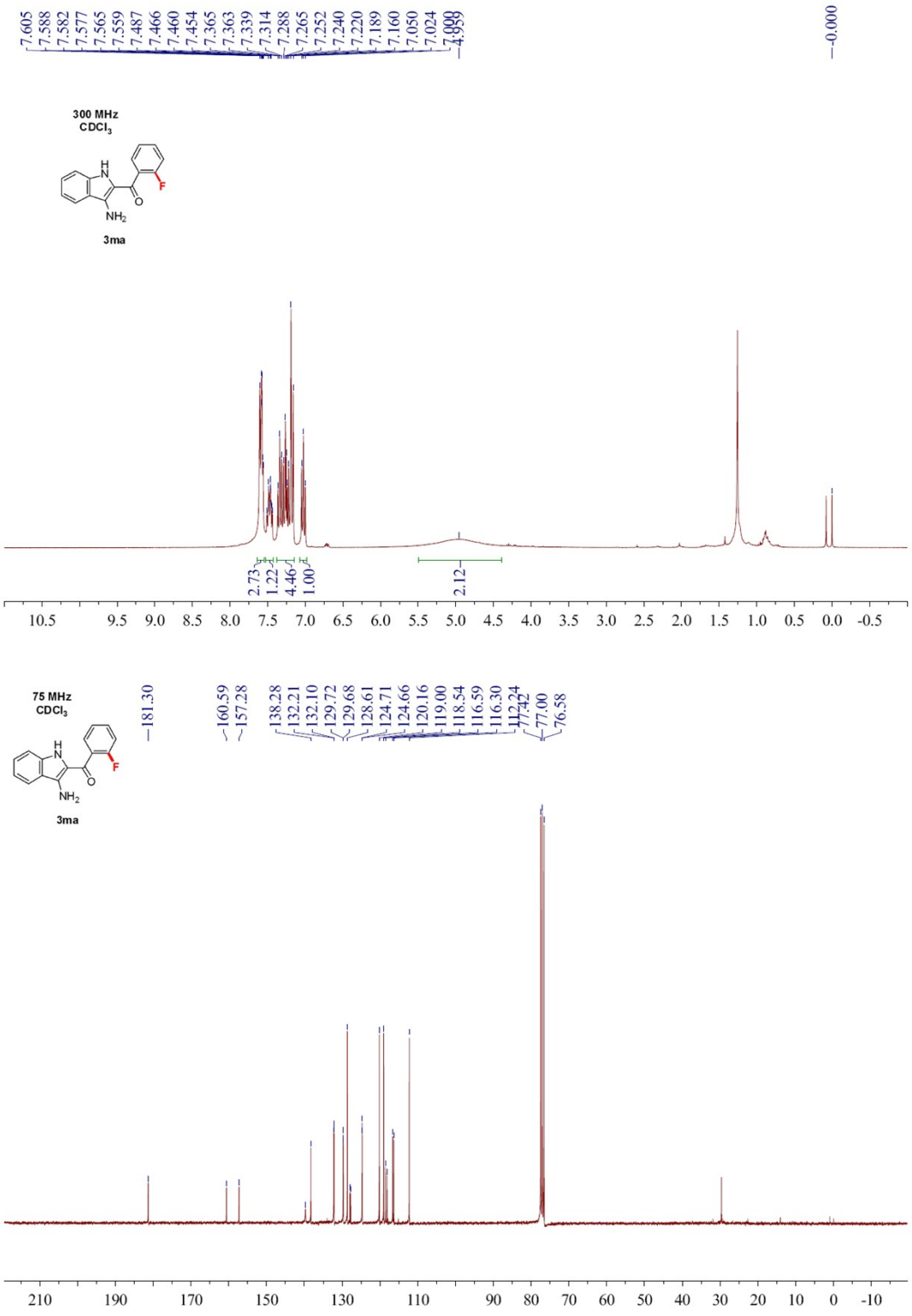
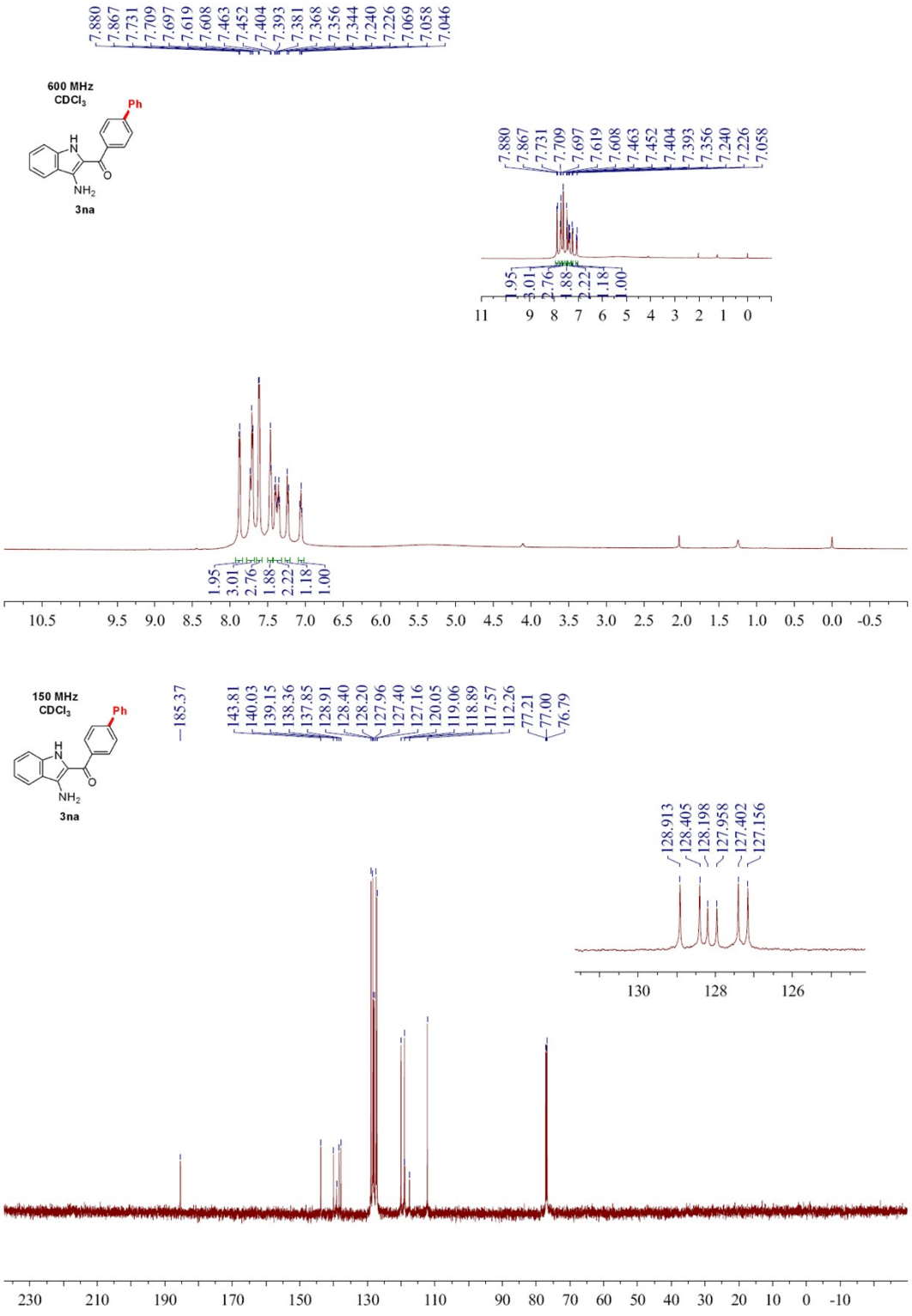
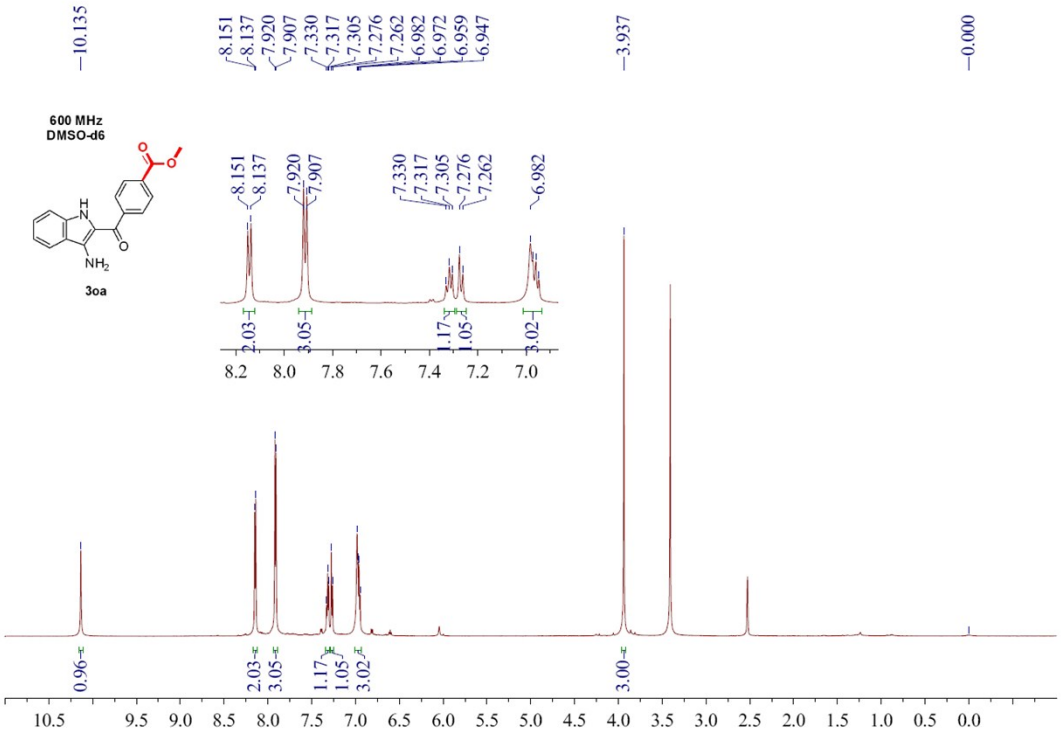
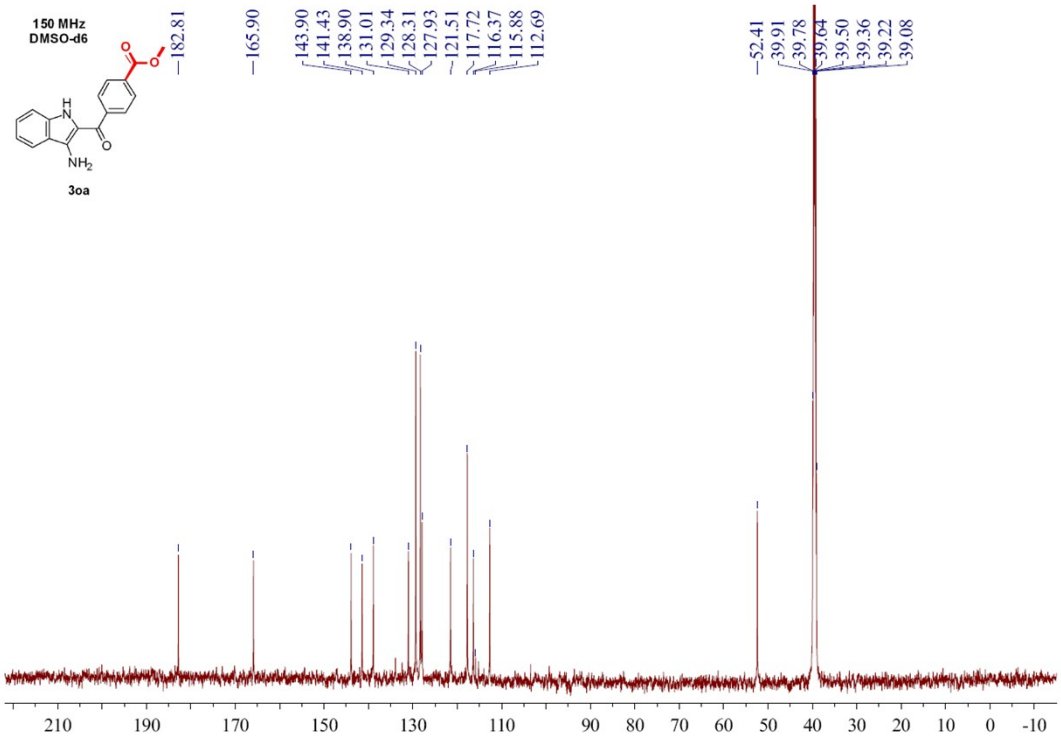
30
20
10
0
-10

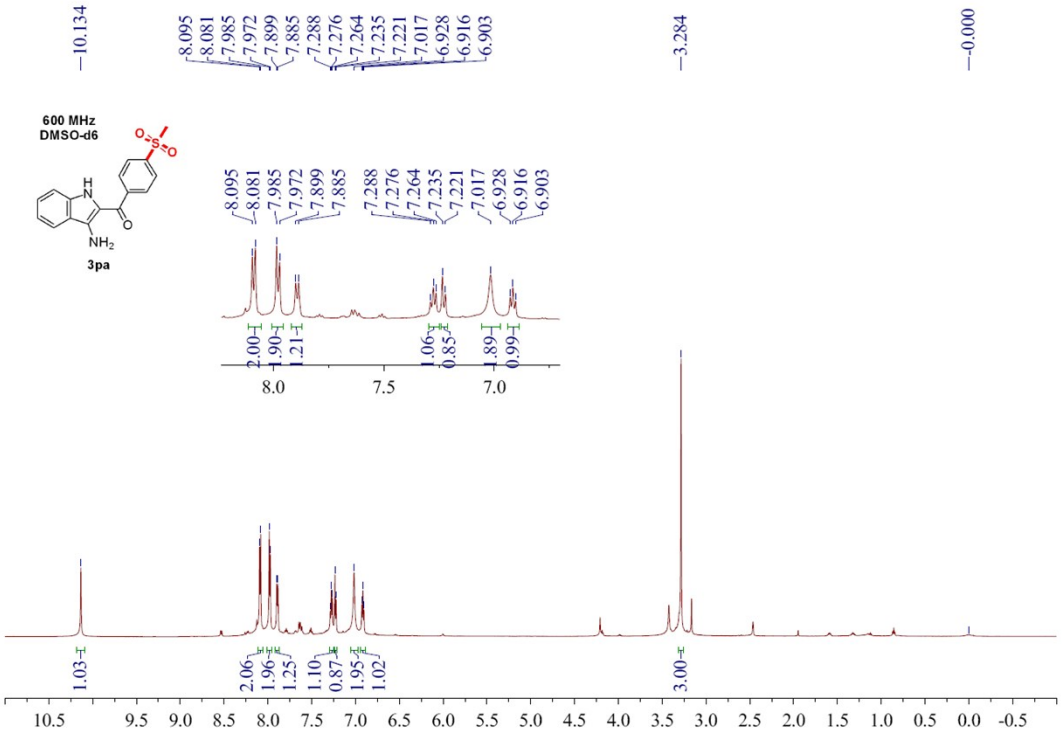

600 MHz
CDCl3
3la
150 MHz
CDCl3
3la

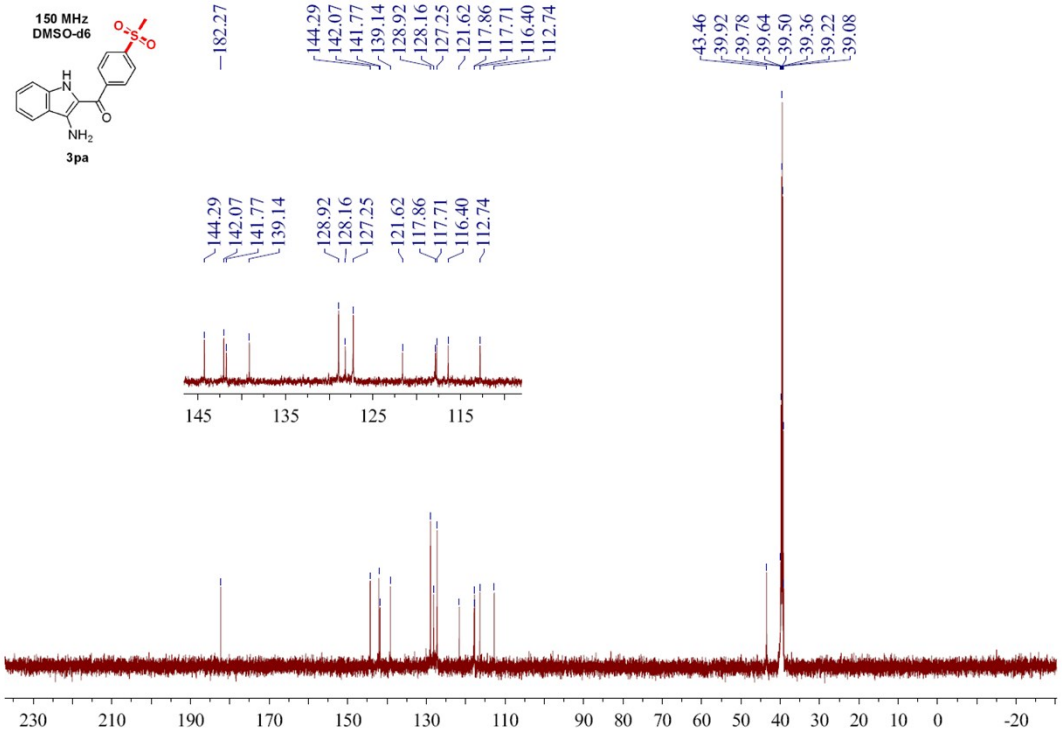

7.605
7.588
7.582
7.577
7.565
7.559
7.487
7.466
7.460
7.454
7.365
7.363
7.339
7.314
7.288
7.265
7.252
7.240
7.220
7.189
7.160
7.050
7.024
0.000
300 MHz
CDCl3
3ma
2.73
1.22
4.46
1.00
2.12
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
75 MHz
CDCl3
3ma
181.30
160.59
157.28
138.28
132.21
132.10
129.72
129.68
128.61
124.71
124.66
120.16
119.00
118.54
116.59
116.30
112.24
77.42
77.00
76.58
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10

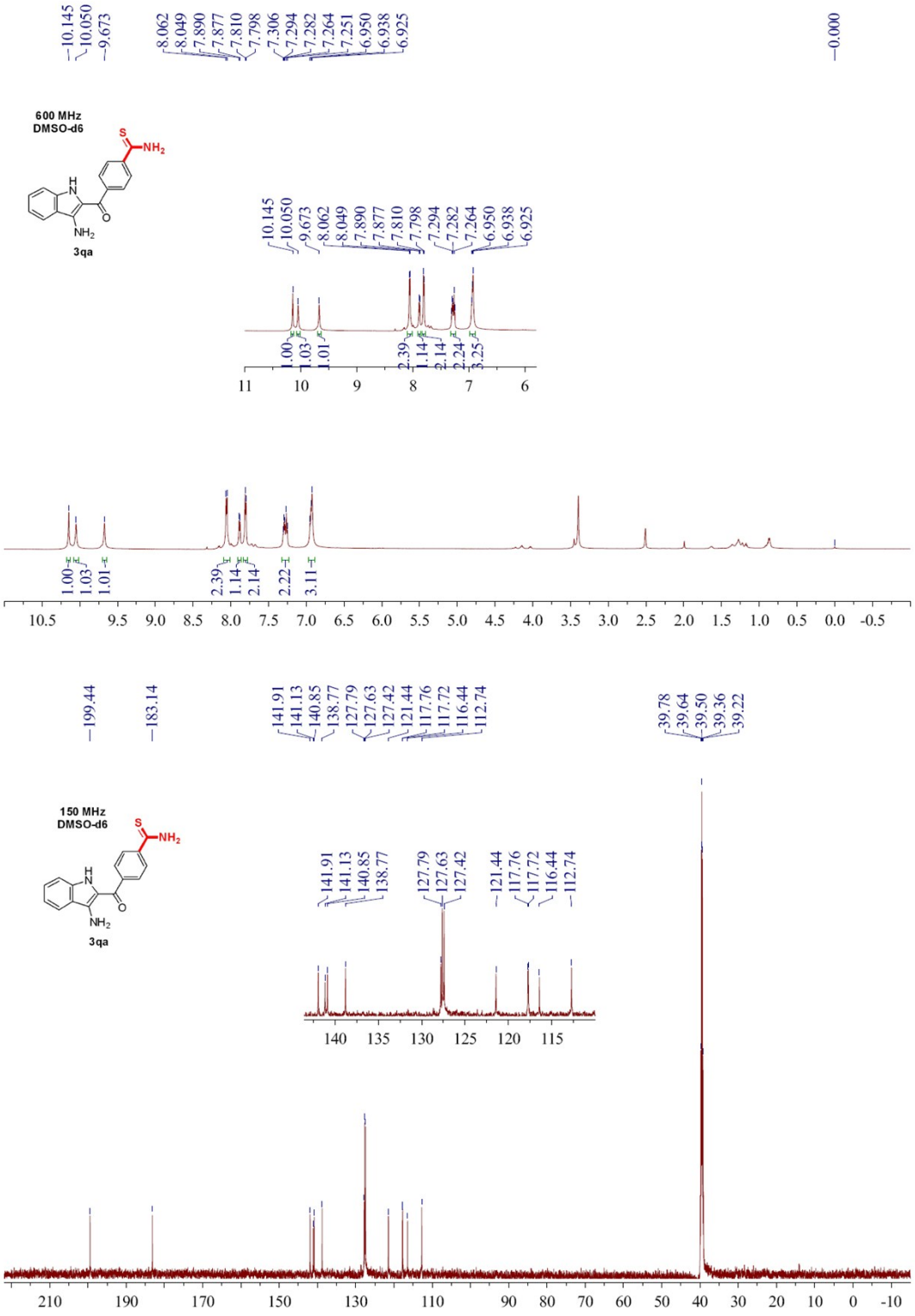

600 MHz
CDCl3
Ph
NH2
3na
7.880
7.867
7.731
7.709
7.697
7.619
7.608
7.463
7.452
7.404
7.393
7.381
7.368
7.356
7.344
7.240
7.226
7.069
7.058
7.046
1.95
3.01
2.76
1.88
2.22
1.18
1.00
150 MHz
CDCl3
185.37
143.81
140.03
139.15
138.36
137.85
128.91
128.40
128.20
127.96
127.40
127.16
120.05
119.06
118.89
117.57
112.26
77.21
77.00
76.79
128.913
128.405
128.198
127.958
127.402
127.156

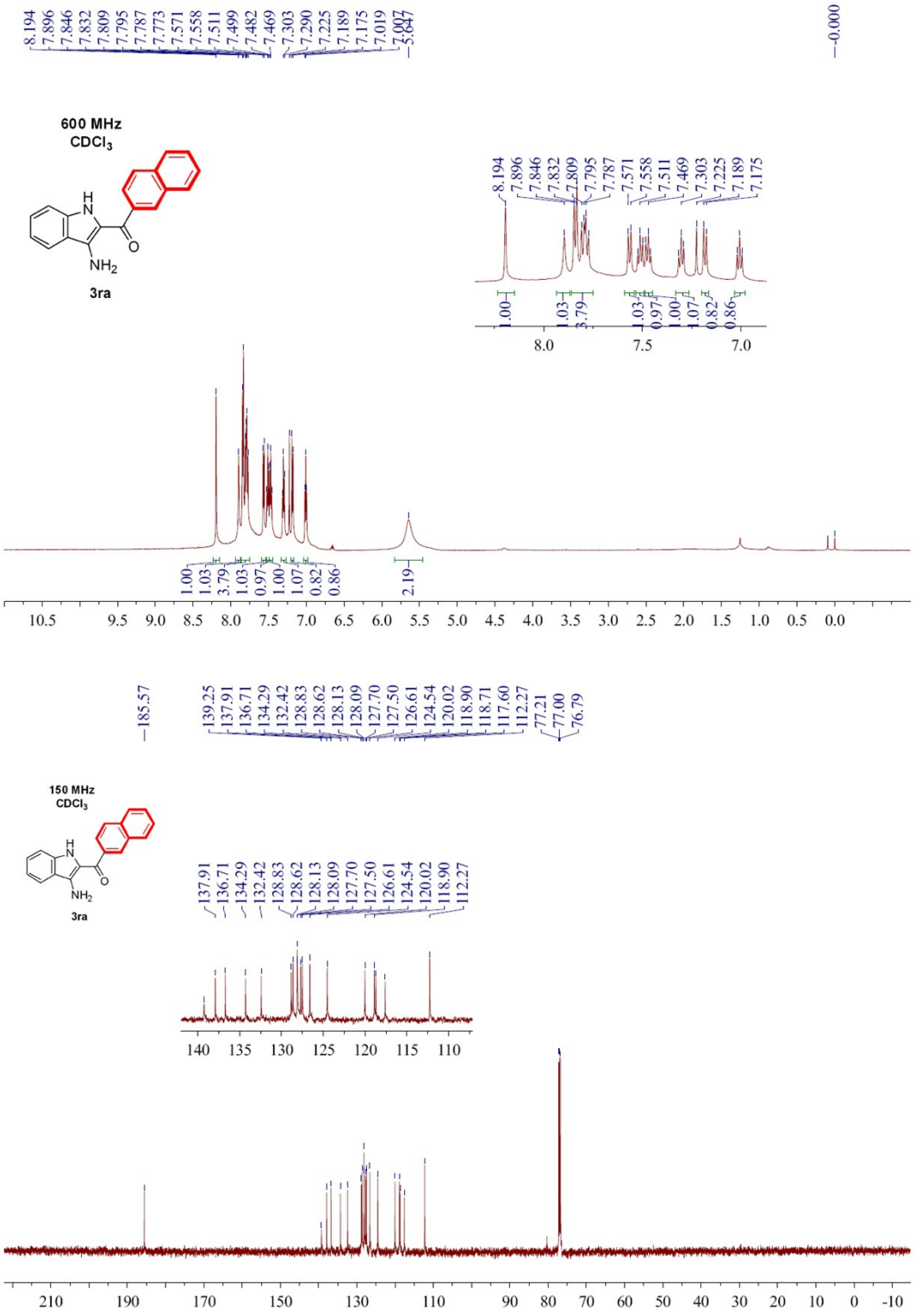

600 MHz
DMSO-d6
3oa
10.135
8.151
8.137
7.920
7.907
7.330
7.317
7.305
7.276
7.262
6.982
6.972
6.959
6.947
3.937
0.000
0.96
2.03
3.05
1.17
1.05
3.02
3.00

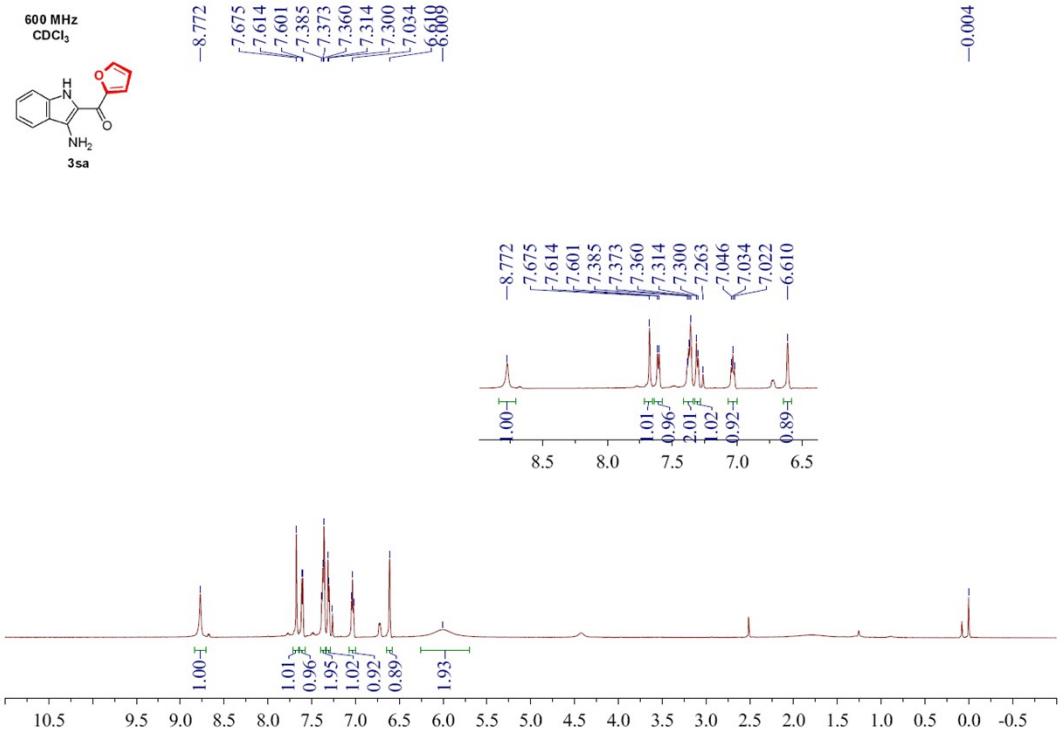

150 MHz
DMSO-d6
3oa
182.81
165.90
143.90
141.43
138.90
131.01
129.34
128.31
127.93
121.51
117.72
116.37
115.88
112.69
52.41
39.91
39.78
39.64
39.50
39.36
39.22
39.08

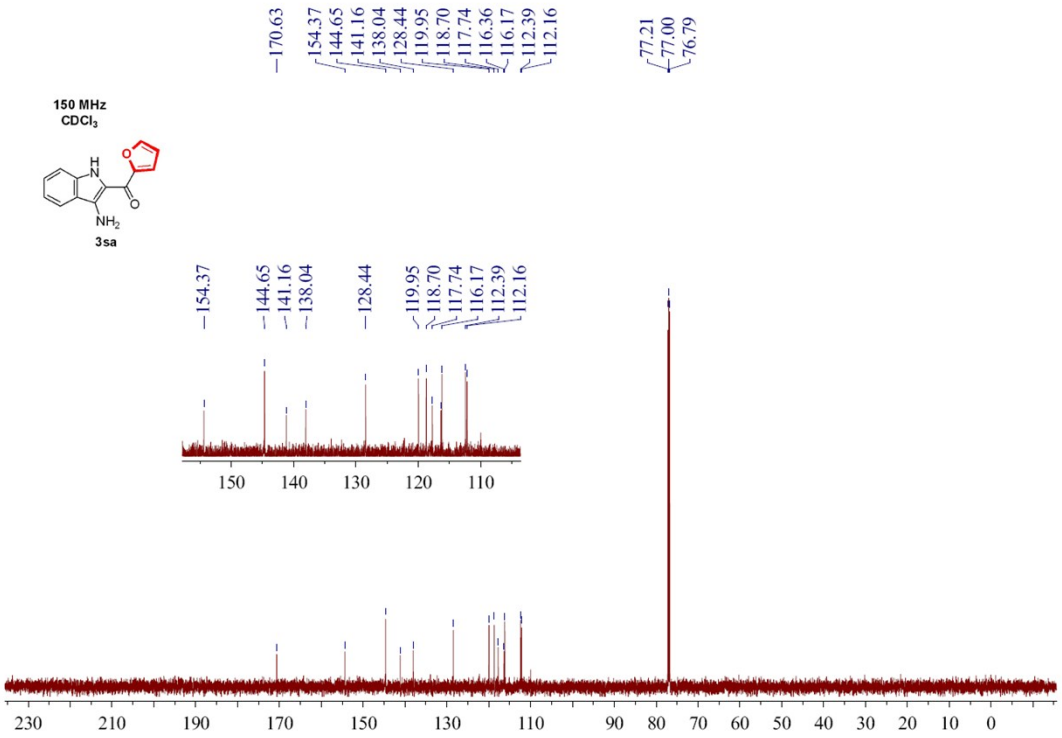

10.134
8.095
8.081
7.985
7.972
7.899
7.885
7.288
7.276
7.264
7.235
7.221
7.017
6.928
6.916
6.903
3.284
0.000
600 MHz
DMSO-d6
3pa
1.03
2.06
1.96
1.25
1.10
0.87
1.95
1.02
3.00


182.27
144.29
142.07
141.77
139.14
128.92
128.16
127.25
121.62
117.86
117.71
116.40
112.74
43.46
39.92
39.78
39.64
39.50
39.36
39.22
39.08
150 MHz
DMSO-d6
3pa


600 MHz
DMSO-d6
3qa
150 MHz
DMSO-d6
3qa


600 MHz
CDCl3
3ra
150 MHz
CDCl3
3ra


600 MHz
CDCl3
3sa
8.772
7.675
7.614
7.601
7.385
7.373
7.360
7.314
7.300
7.034
6.610
6.609
0.004
7.263
7.046
7.022
1.00
1.01
0.96
1.95
1.02
0.92
0.89
1.93


150 MHz
CDCl3
3sa
170.63
154.37
144.65
141.16
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128.44
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118.70
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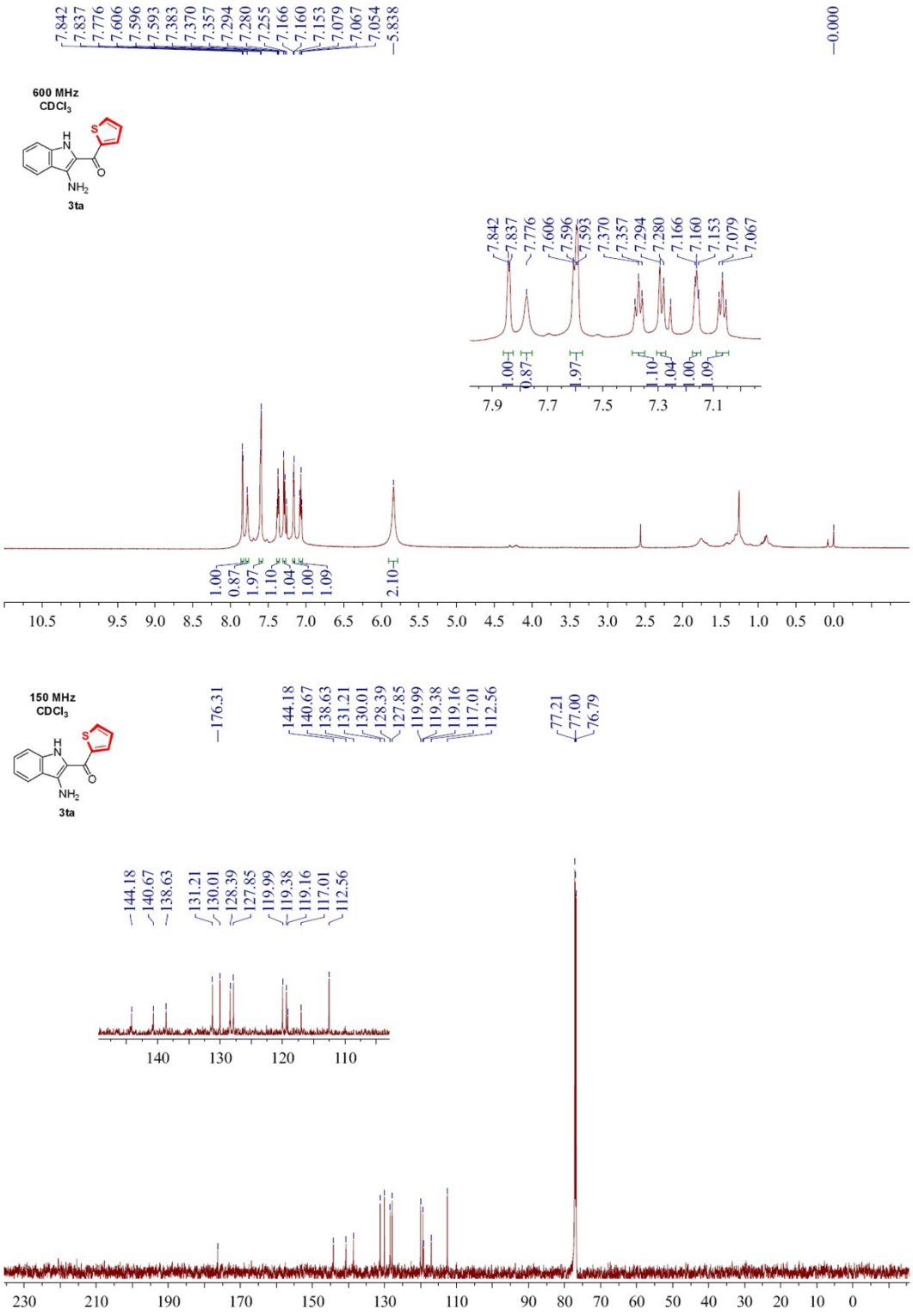
116.36
116.17
112.39
112.16
77.21
77.00
76.79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7.606
7.596
7.593
7.383
7.370
7.357
7.294
7.280
7.255
7.166
7.160
7.153
7.079
7.067
7.054
5.838
0.000
600 MHz
CDCl3
3ta
1.00
0.87
1.97
1.10
1.04
1.00
1.09
2.10
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
7.9
7.7
7.5
7.3
7.1
150 MHz
CDCl3
3ta
176.31
144.18
140.67
138.63
131.21
130.01
128.39
127.85
119.99
119.38
119.16
117.01
112.56
77.21
77.00
76.79
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30
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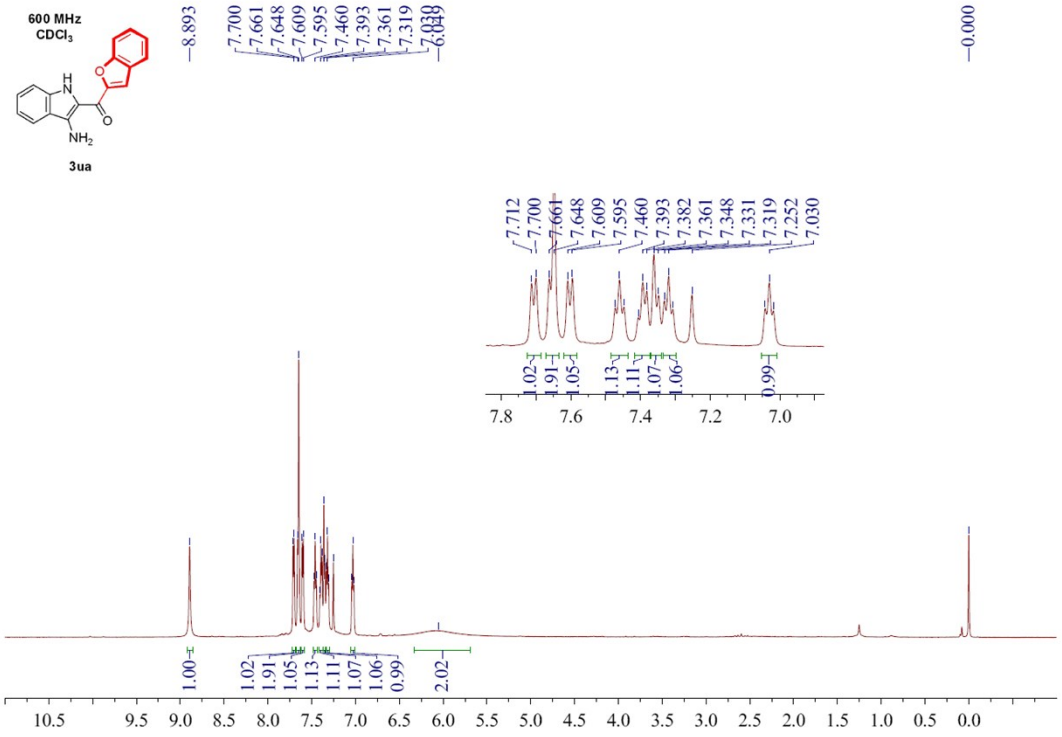
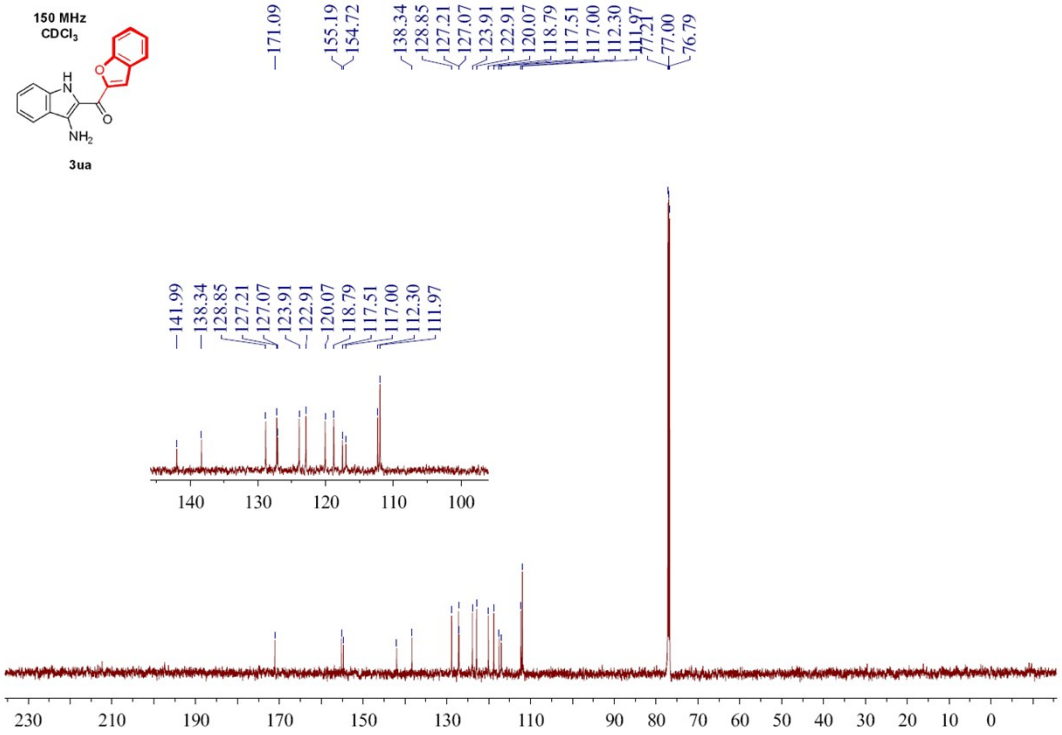
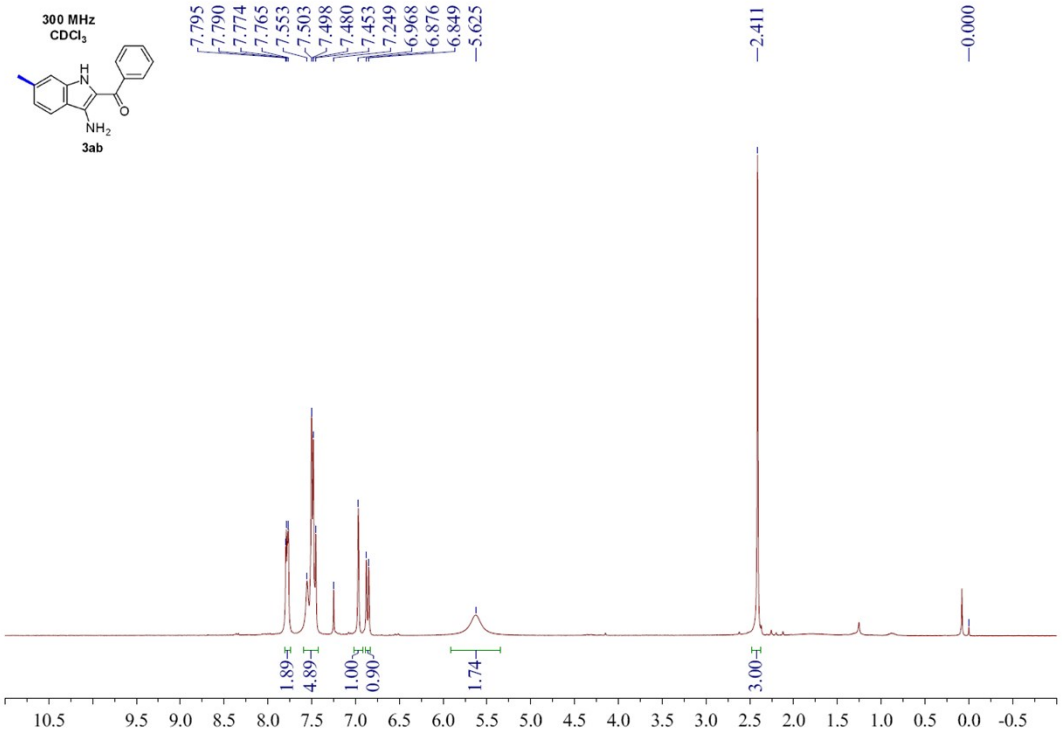
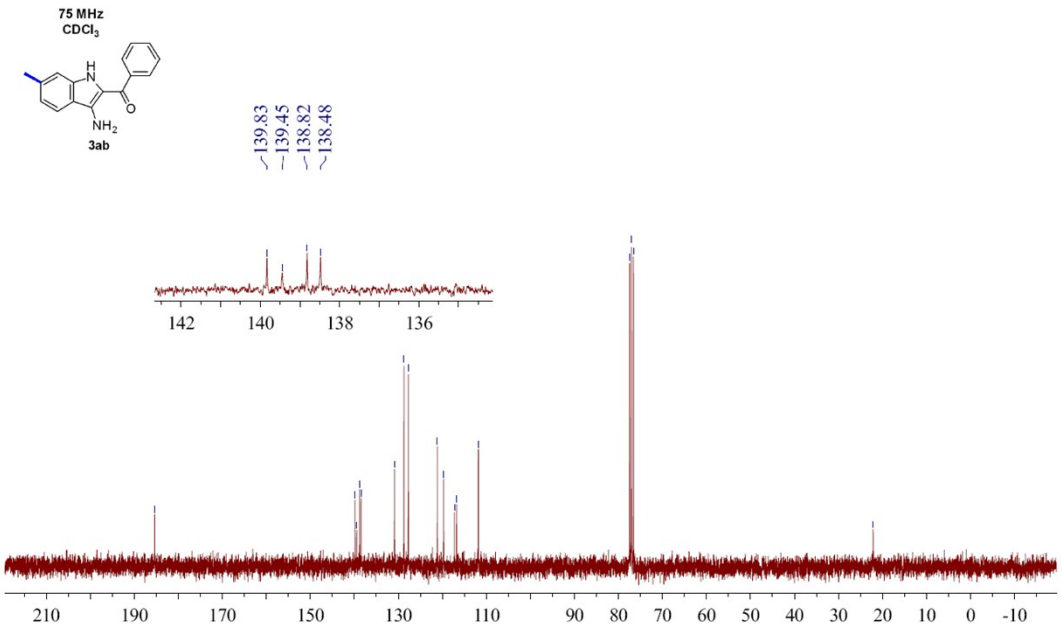
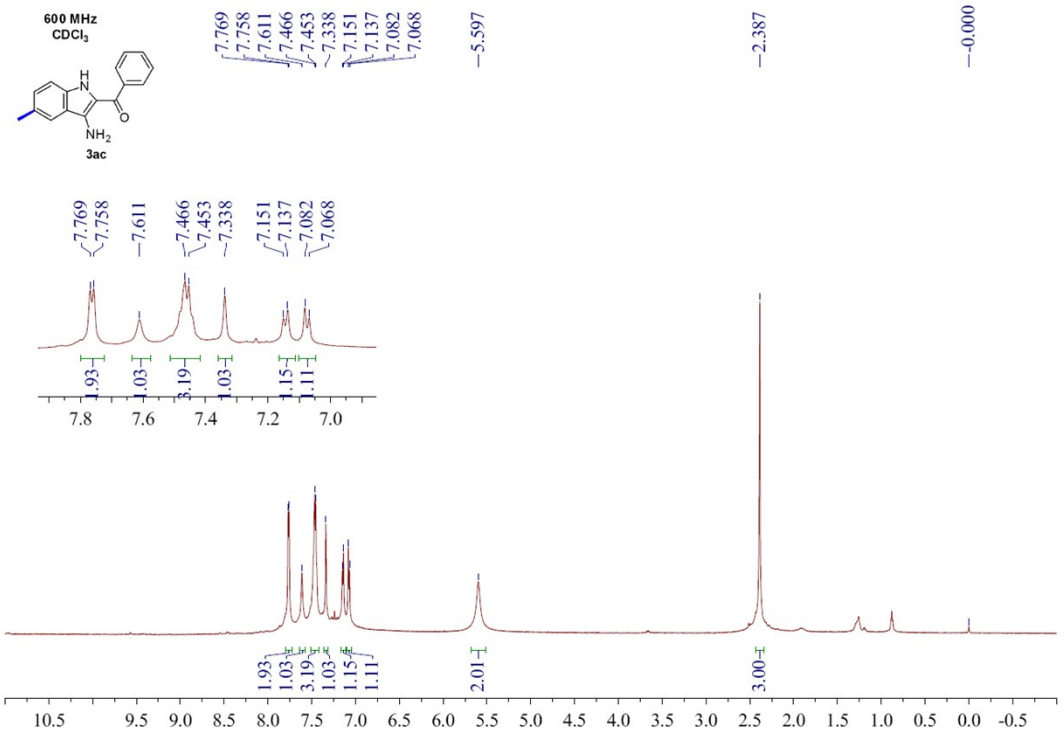
10
0

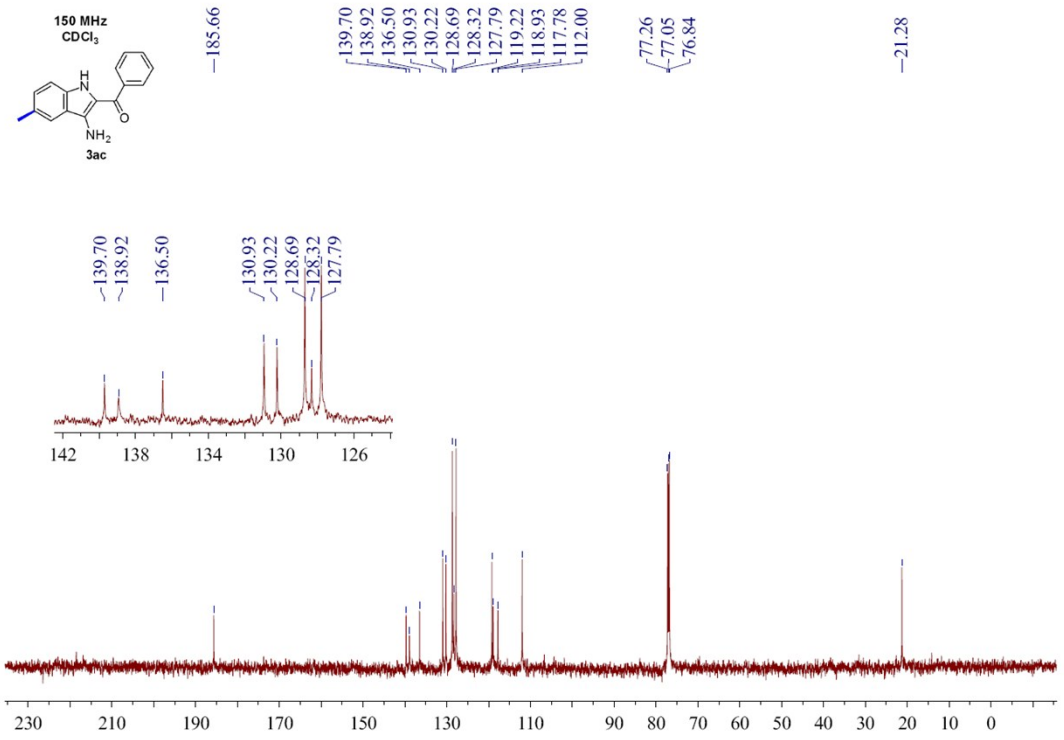

600 MHz
CDCl3
3ua
8.893
7.700
7.661
7.648
7.609
7.595
7.460
7.393
7.361
7.319
0.000
7.712
7.700
7.661
7.648
7.609
7.595
7.460
7.393
7.382
7.361
7.348
7.331
7.319
7.252
7.030
1.02
1.91
1.05
1.13
1.11
1.07
1.06
0.99
1.00
2.02

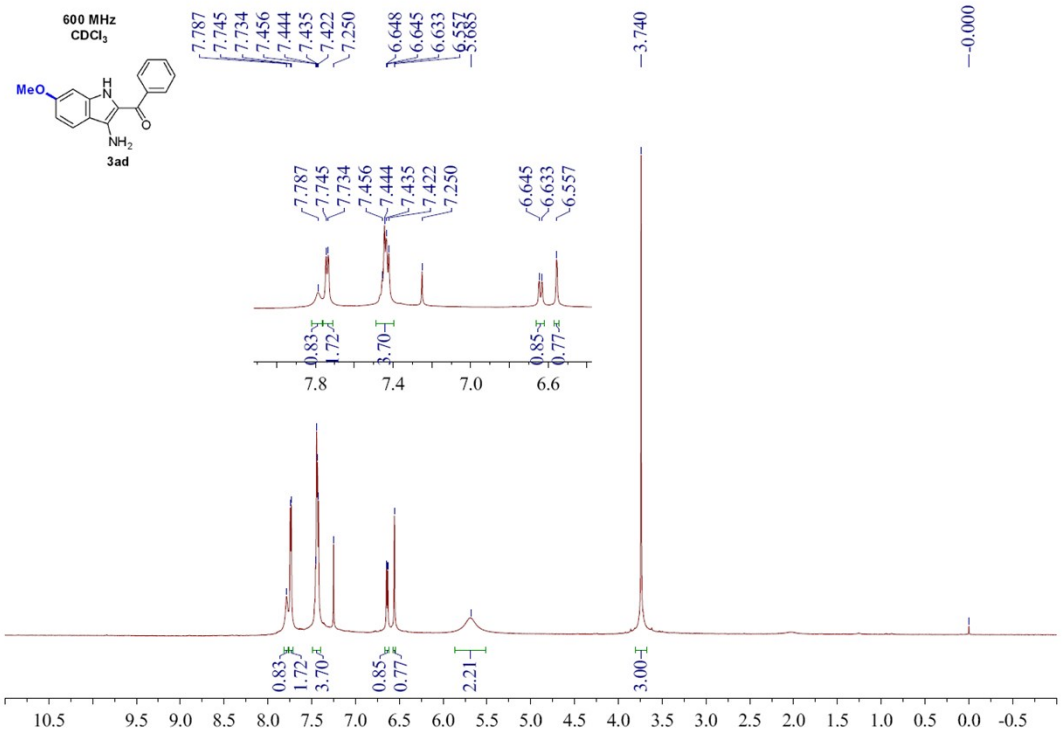

150 MHz
CDCl3
3ua
171.09
155.19
154.72
138.34
128.85
127.21
127.07
123.91
122.91
120.07
118.79
117.51
117.00
112.30
111.97
77.21
77.00
76.79
141.99
138.34
128.85
127.21
127.07
123.91
122.91
120.07
118.79
117.51
117.00
112.30
111.97

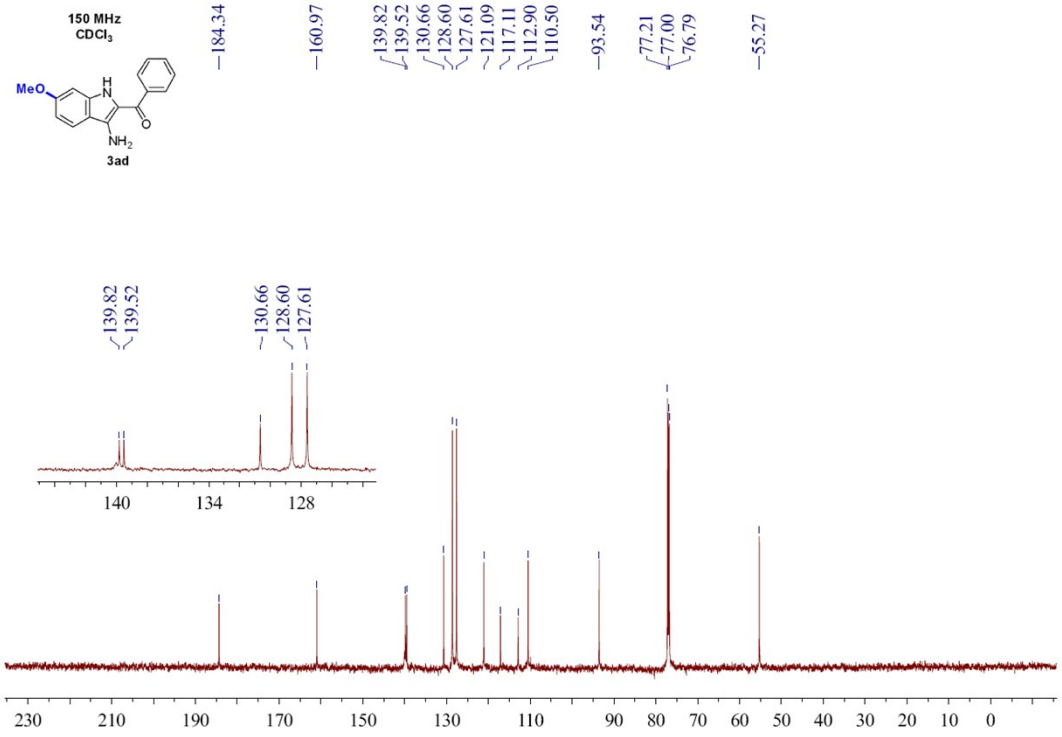

300 MHz
CDCl3
3ab
7.795
7.790
7.774
7.765
7.553
7.503
7.498
7.480
7.453
7.249
6.968
6.876
6.849
5.625
2.411
0.000
1.89
4.89
1.00
0.90
1.74
3.00

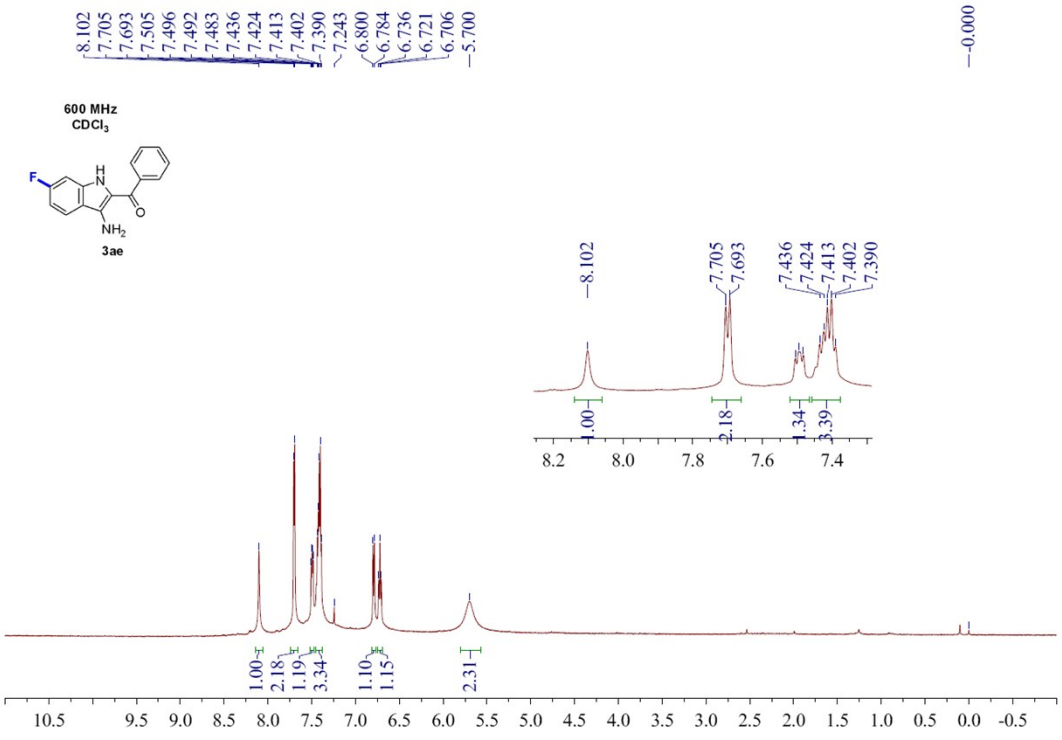

75 MHz
CDCl3
3ab
185.34
139.83
139.45
138.82
138.48
130.88
128.72
127.74
121.14
119.70
117.25
116.76
111.82
77.42
77.00
76.58
22.12

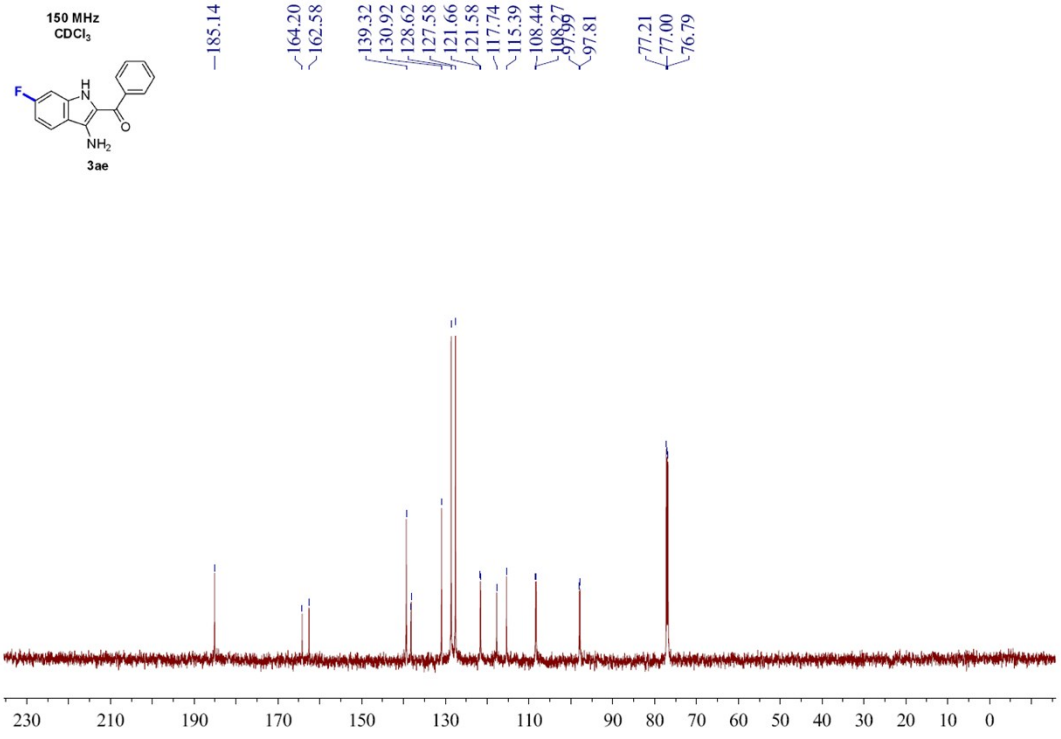

600 MHz
CDCl3
3ac
7.769
7.758
7.611
7.466
7.453
7.338
7.151
7.137
7.082
7.068
5.597
2.387
0.000
1.93
1.03
3.19
1.03
1.15
1.11
2.01
3.00

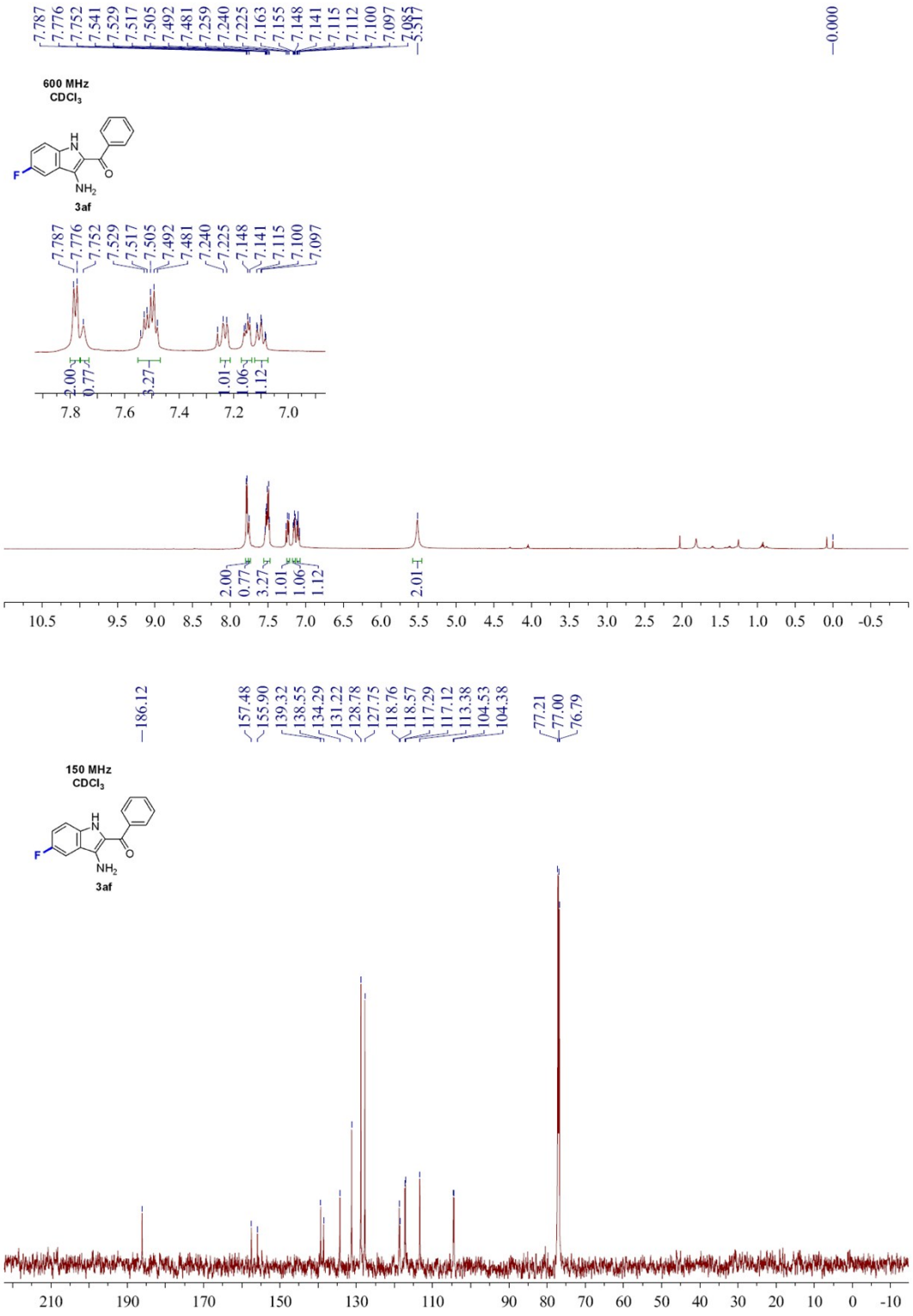

150 MHz
CDCl3
3ac
185.66
139.70
138.92
136.50
130.93
130.22
128.69
128.32
127.79
119.22
118.93
117.78
112.00
77.26
77.05
76.84
21.28

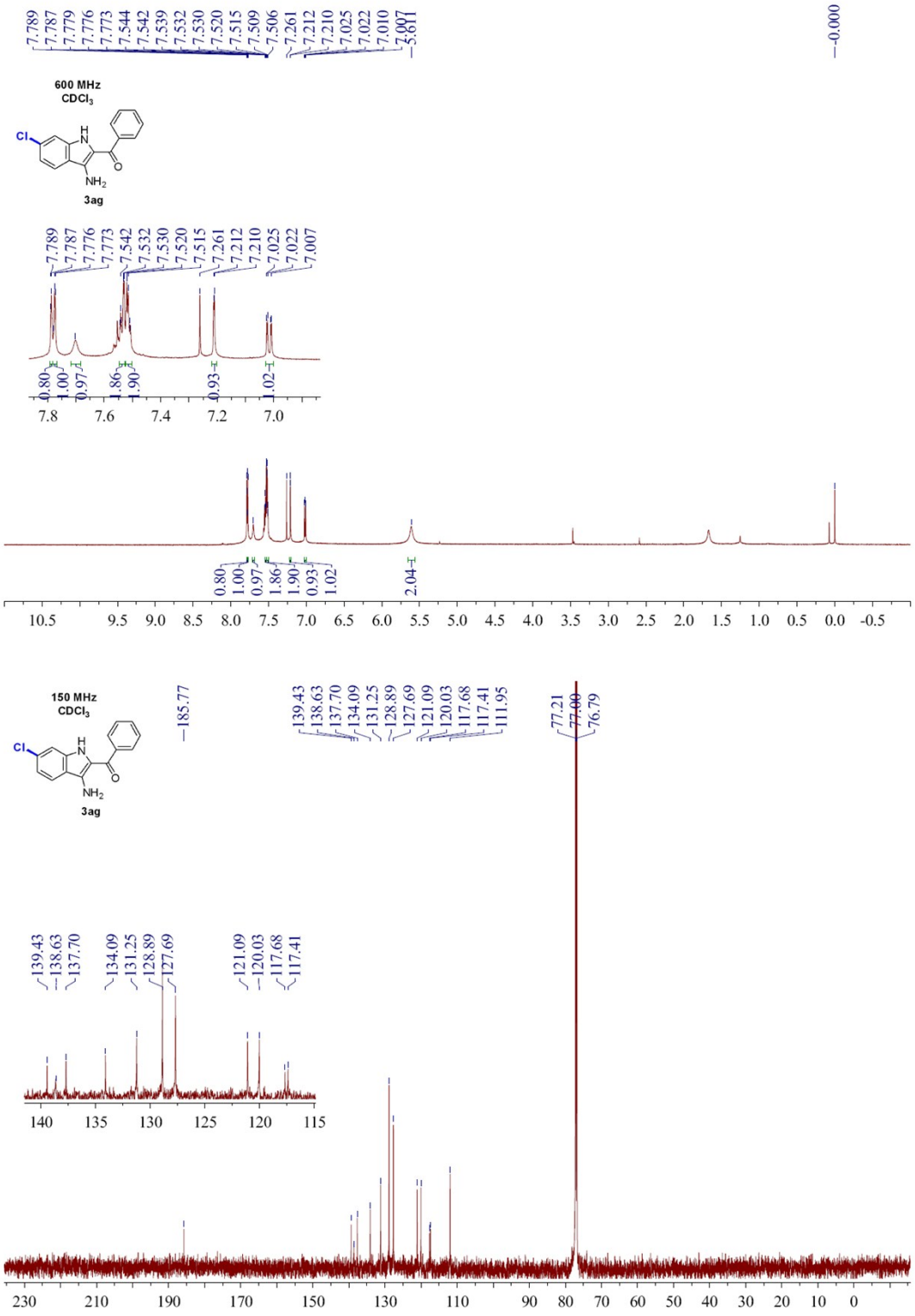

600 MHz
CDCl3
MeO
NH2
3ad
7.787
7.745
7.734
7.456
7.444
7.435
7.422
7.250
6.648
6.645
6.633
6.557
5.685
3.740
0.000
0.83
1.72
3.70
0.85
0.77
2.21
3.00

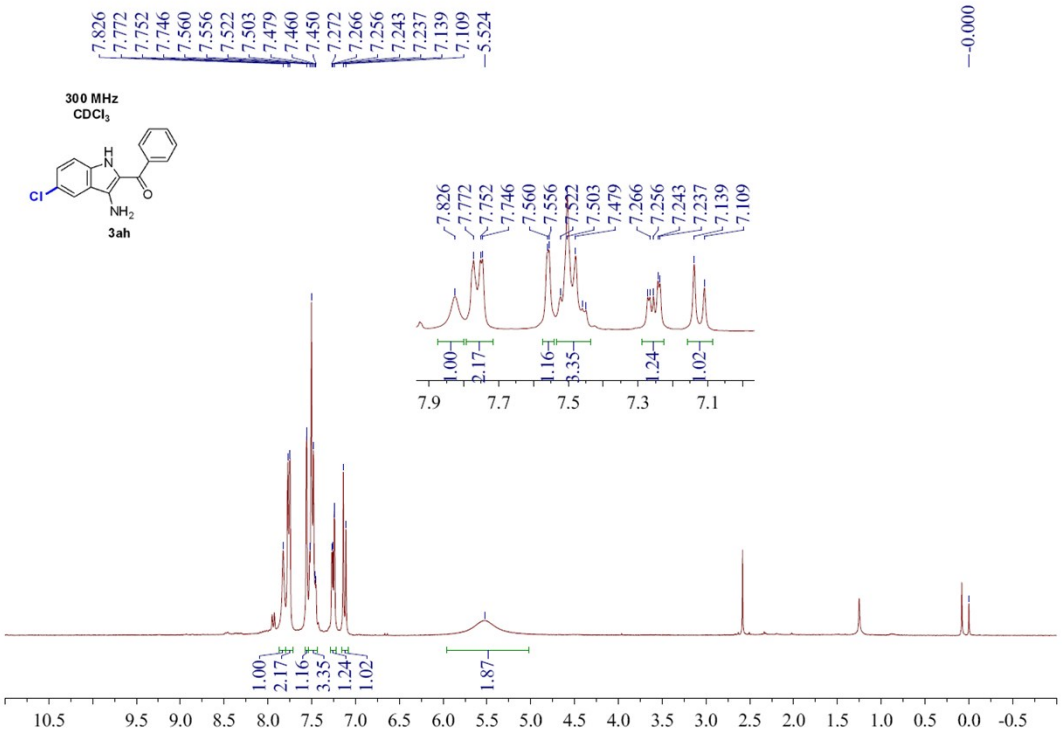

150 MHz
CDCl3
MeO
NH2
3ad
184.34
160.97
139.82
139.52
130.66
128.60
127.61
121.09
117.11
112.90
110.50
93.54
77.21
77.00
76.79
55.27

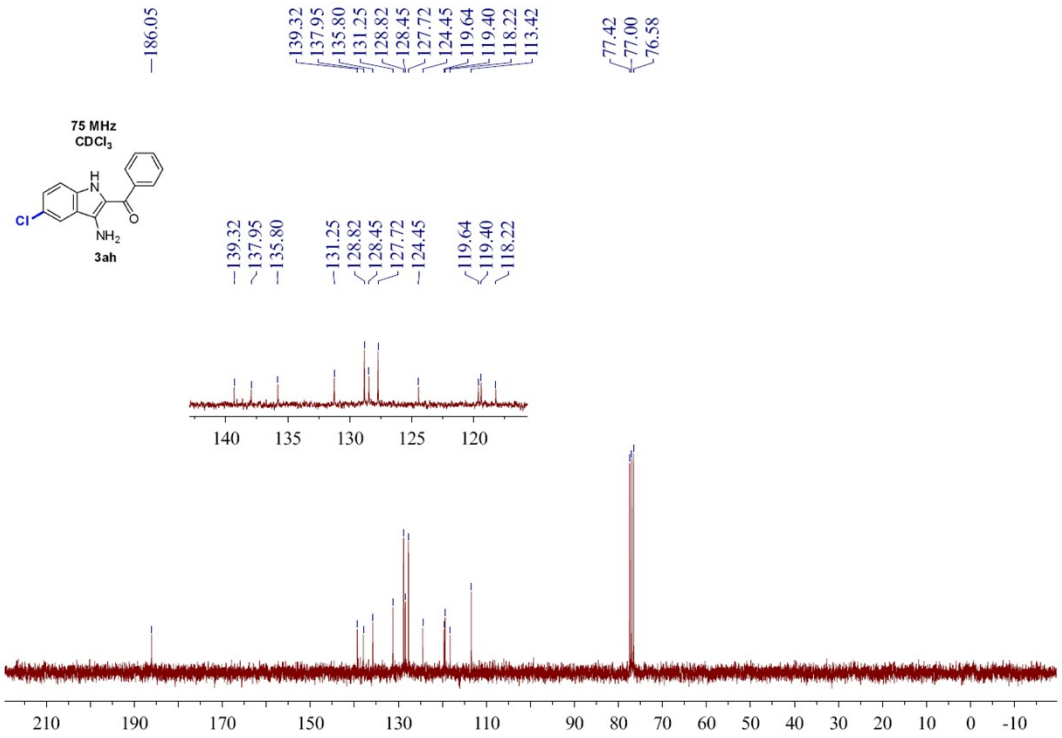

8.102
7.705
7.693
7.505
7.496
7.492
7.483
7.436
7.424
7.413
7.402
7.390
7.243
6.800
6.784
6.736
6.721
6.706
5.700
0.000
600 MHz
CDCl3
F
H
N
O
NH2
3ae
1.00
2.18
1.19
3.34
1.10
1.15
2.31
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5

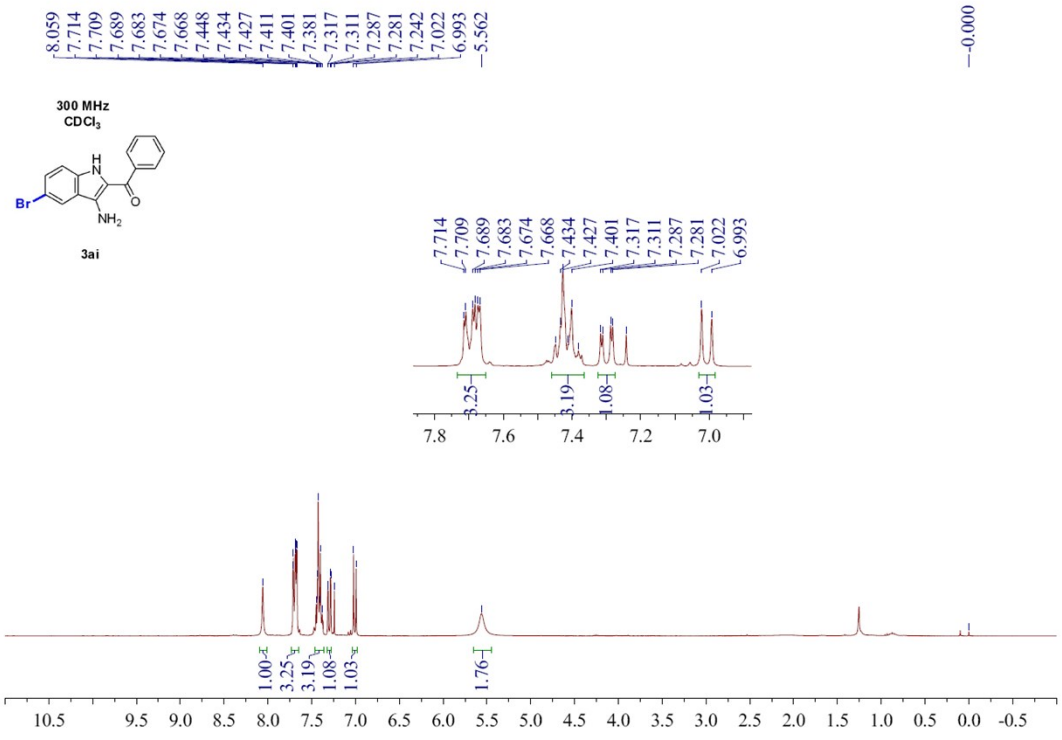

150 MHz
CDCl3
F
H
N
O
NH2
3ae
185.14
164.20
162.58
139.32
130.92
128.62
127.58
121.66
121.58
117.74
115.39
108.44
108.27
97.99
97.81
77.21
77.00
76.79
230
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0

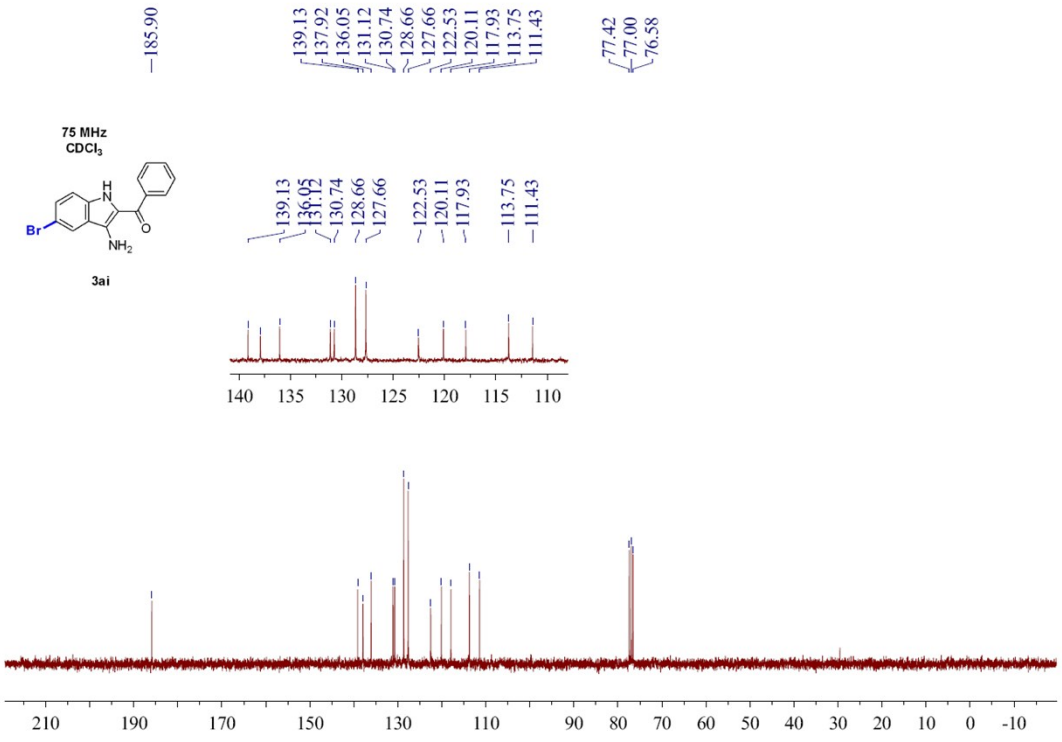

7.787
7.776
7.752
7.541
7.529
7.517
7.505
7.492
7.481
7.259
7.240
7.225
7.163
7.155
7.148
7.141
7.115
7.112
7.100
7.097
7.085
5.517
0.000
600 MHz
CDCl3
3af
2.00
0.77
3.27
1.01
1.06
1.12
2.01
186.12
157.48
155.90
139.32
138.55
134.29
131.22
128.78
127.75
118.76
118.57
117.29
117.12
113.38
104.53
104.38
77.21
77.00
76.79
150 MHz
CDCl3
3af

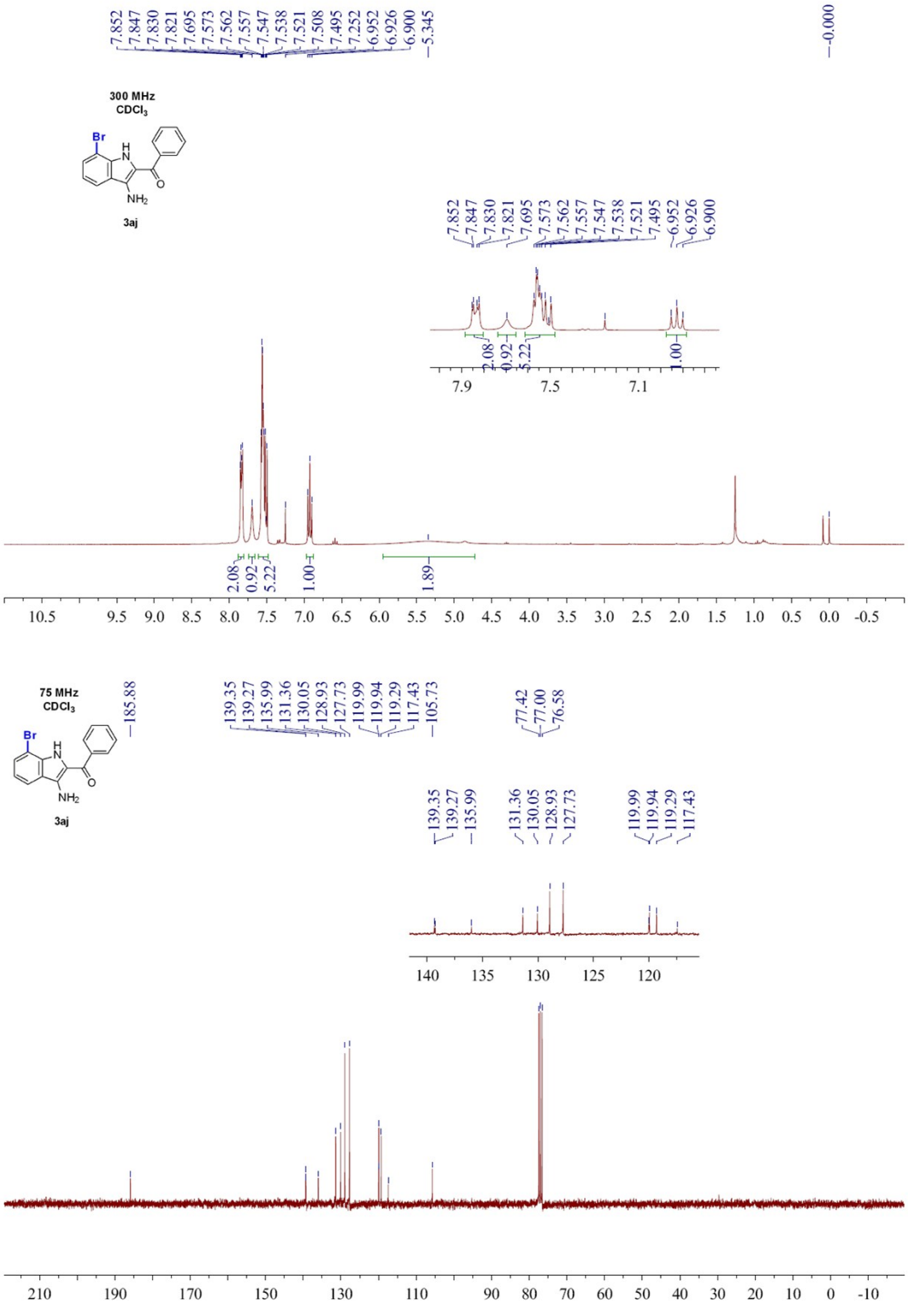

600 MHz
CDCl3
3ag
7.789
7.787
7.779
7.776
7.773
7.544
7.542
7.539
7.532
7.530
7.520
7.515
7.509
7.506
7.261
7.212
7.210
7.025
7.022
7.010
7.007
5.811
-0.000
0.80
1.00
0.97
1.86
1.90
0.93
1.02
2.04
150 MHz
CDCl3
3ag
185.77
139.43
138.63
137.70
134.09
131.25
128.89
127.69
121.09
120.03
117.68
117.41
111.95
77.21
77.00
76.79

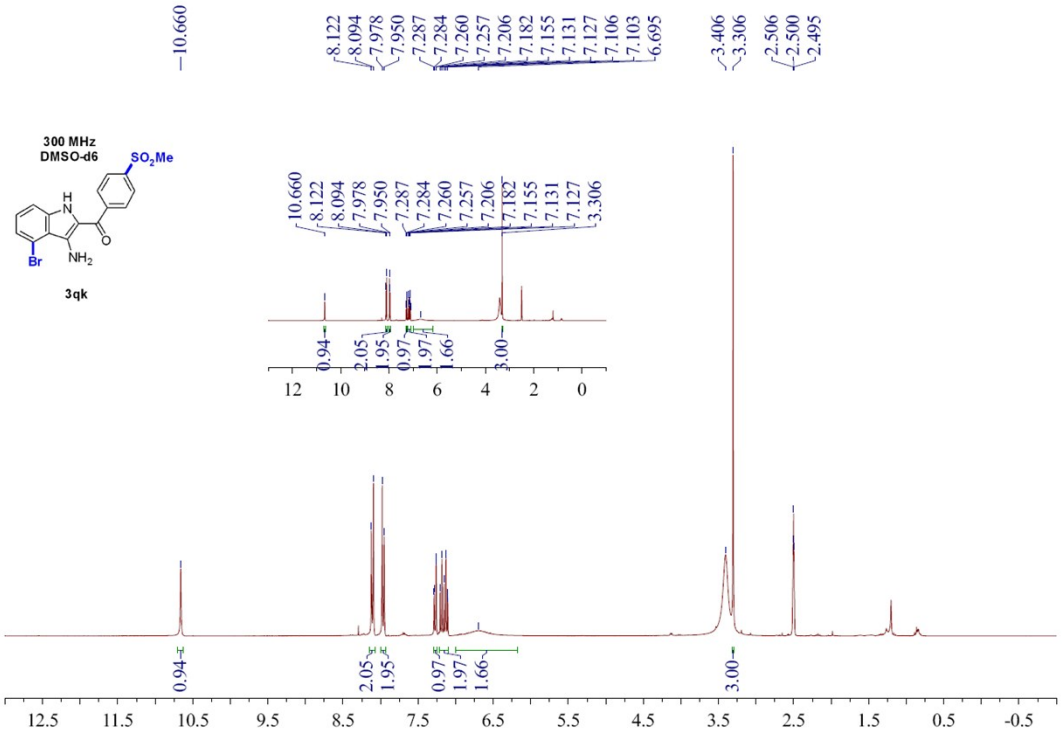

7.826
7.772
7.752
7.746
7.560
7.556
7.522
7.503
7.479
7.460
7.450
7.272
7.266
7.256
7.243
7.237
7.139
7.109
5.524
0.000
300 MHz
CDCl3
Cl
NH2
3ah
1.00
2.17
1.16
3.35
1.24
1.02
1.87

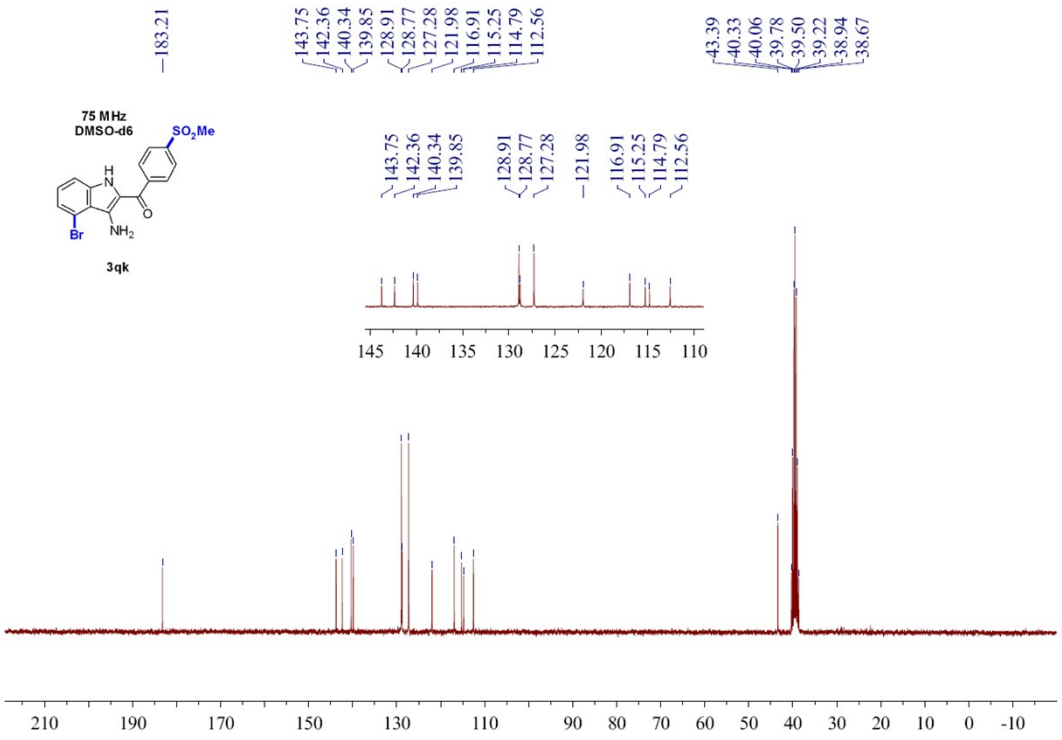

186.05
139.32
137.95
135.80
131.25
128.82
128.45
127.72
124.45
119.64
119.40
118.22
113.42
77.42
77.00
76.58
75 MHz
CDCl3
Cl
NH2
3ah

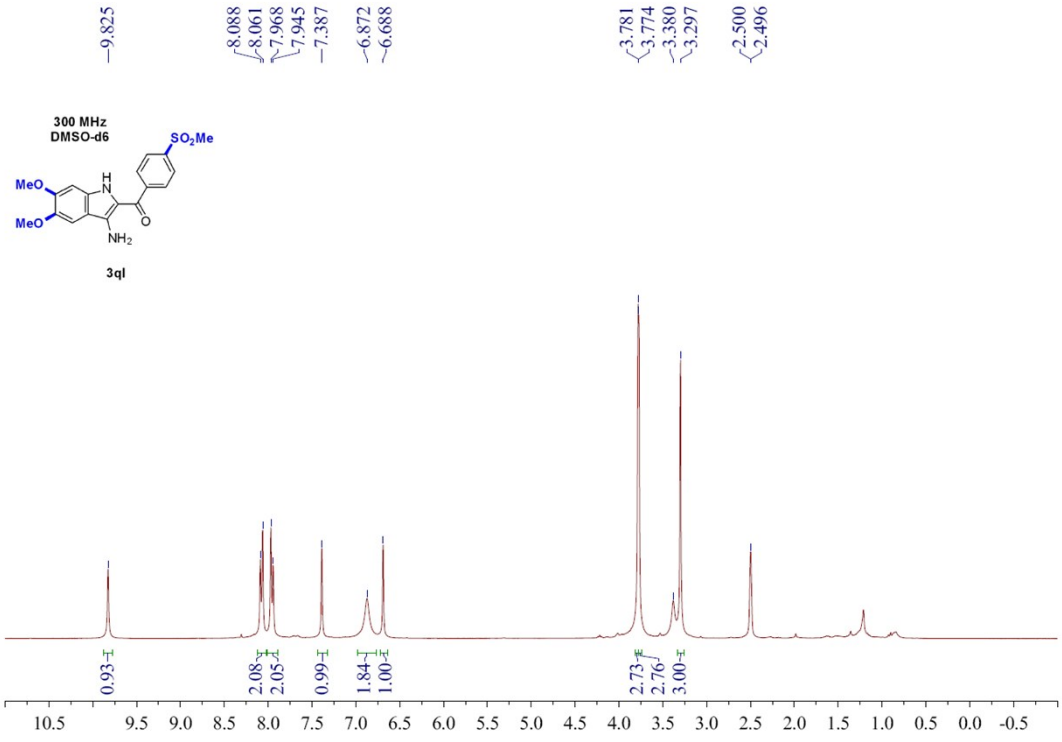

300 MHz
CDCl3
Br
NH2
O
H
N
3ai
8.059
7.714
7.709
7.689
7.683
7.674
7.668
7.448
7.434
7.427
7.411
7.401
7.381
7.317
7.311
7.287
7.281
7.242
7.022
6.993
5.562
0.000
3.25
3.19
1.08
1.03
1.00
3.25
3.19
1.08
1.03
1.76

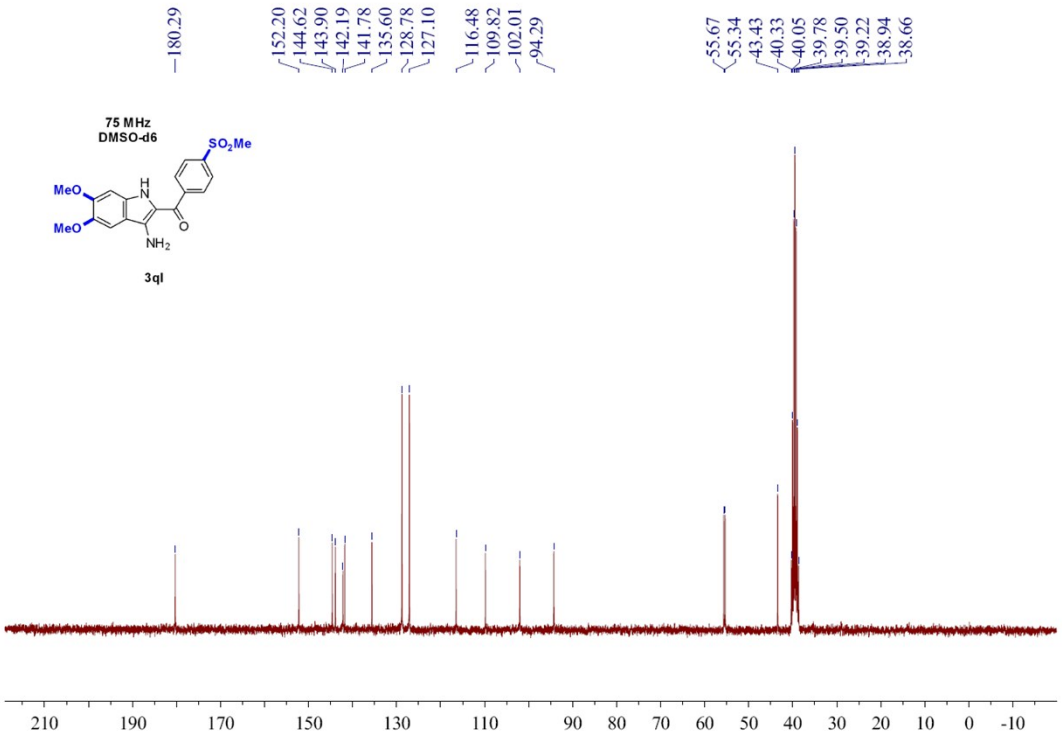

75 MHz
CDCl3
Br
NH2
O
H
N
3ai
185.90
139.13
137.92
136.05
131.12
130.74
128.66
127.66
122.53
120.11
117.93
113.75
111.43
77.42
77.00
76.58

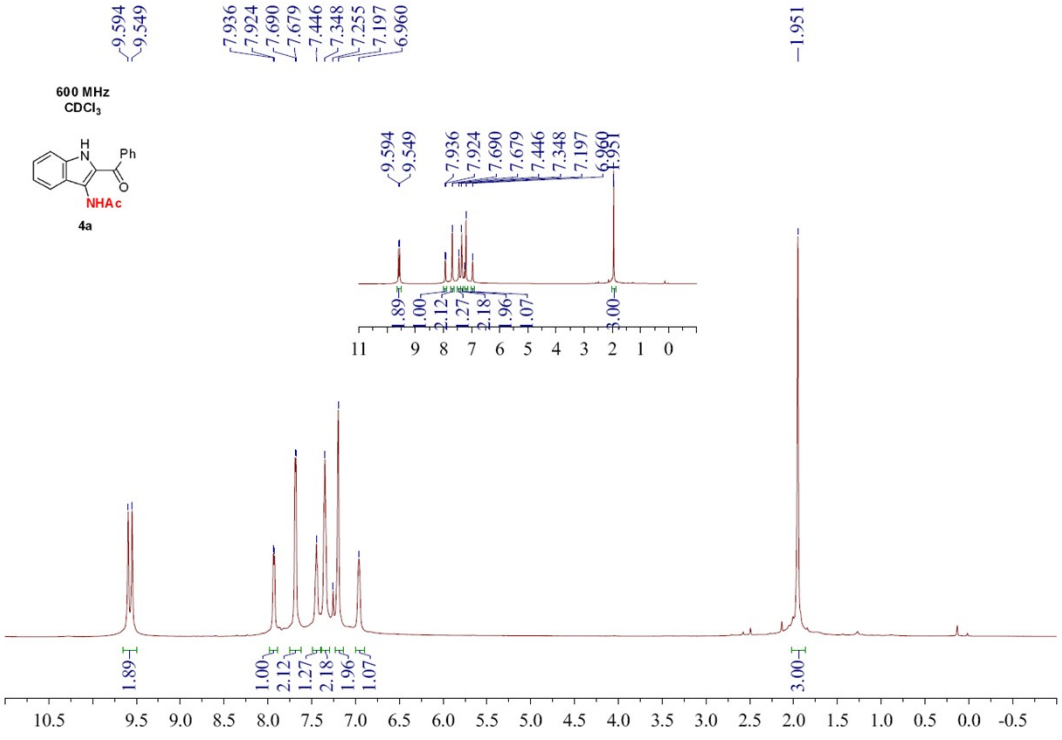

7.852
7.847
7.830
7.821
7.695
7.573
7.562
7.557
7.547
7.538
7.521
7.508
7.495
7.252
6.952
6.926
6.900
5.345
0.000
300 MHz
CDCl3
Br
NH
O
NH2
3aj
2.08
0.92
5.22
1.00
1.89
10.5
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
-0.5
7.9
7.5
7.1
75 MHz
CDCl3
185.88
139.35
139.27
135.99
131.36
130.05
128.93
127.73
119.99
119.94
119.29
117.43
105.73
77.42
77.00
76.58
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10
140
135
130
125
120

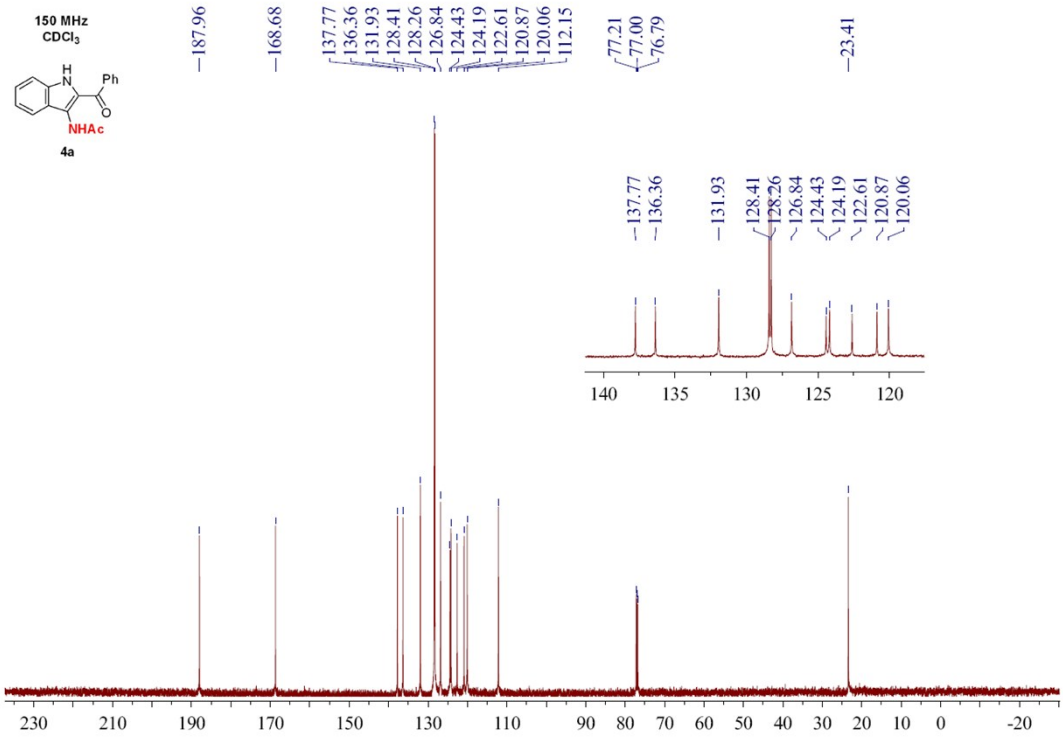

10.660
8.122
8.094
7.978
7.950
7.287
7.284
7.260
7.257
7.206
7.182
7.155
7.131
7.127
7.106
7.103
6.695
3.406
3.306
2.506
2.500
2.495
300 MHz
DMSO-d6
SO2Me
Br
NH2
3qk
0.94
2.05
1.95
0.97
1.97
1.66
3.00

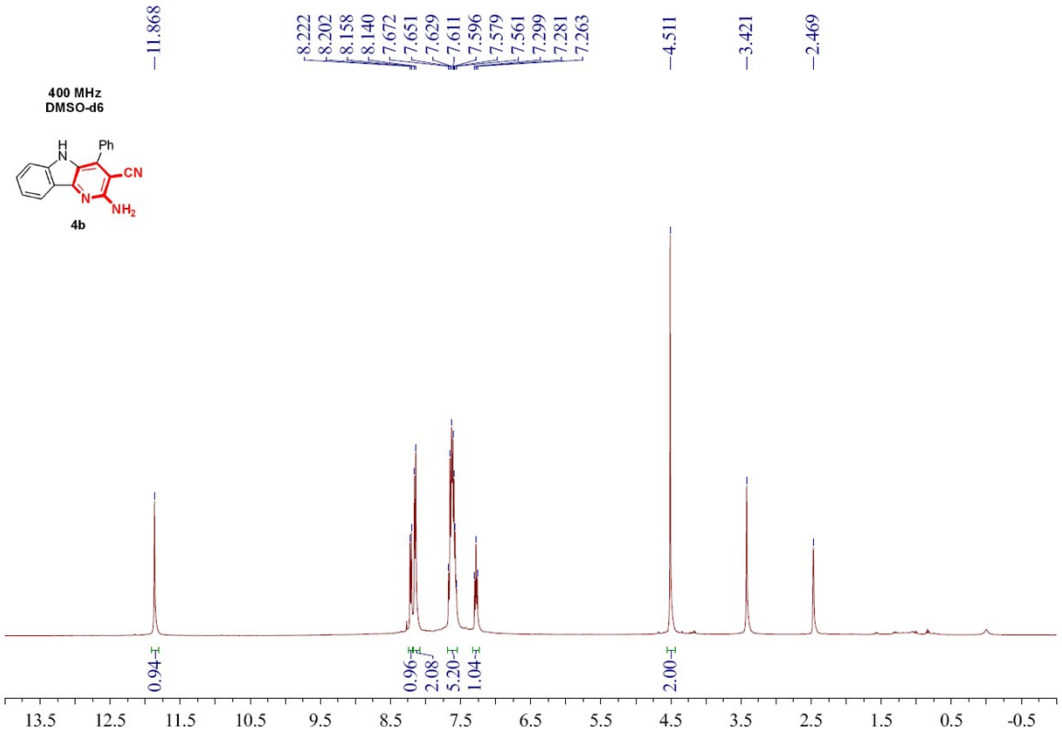

183.21
143.75
142.36
140.34
139.85
128.91
128.77
127.28
121.98
116.91
115.25
114.79
112.56
43.39
40.33
40.06
39.78
39.50
39.22
38.94
38.67
75 MHz
DMSO-d6
SO2Me
Br
NH2
3qk

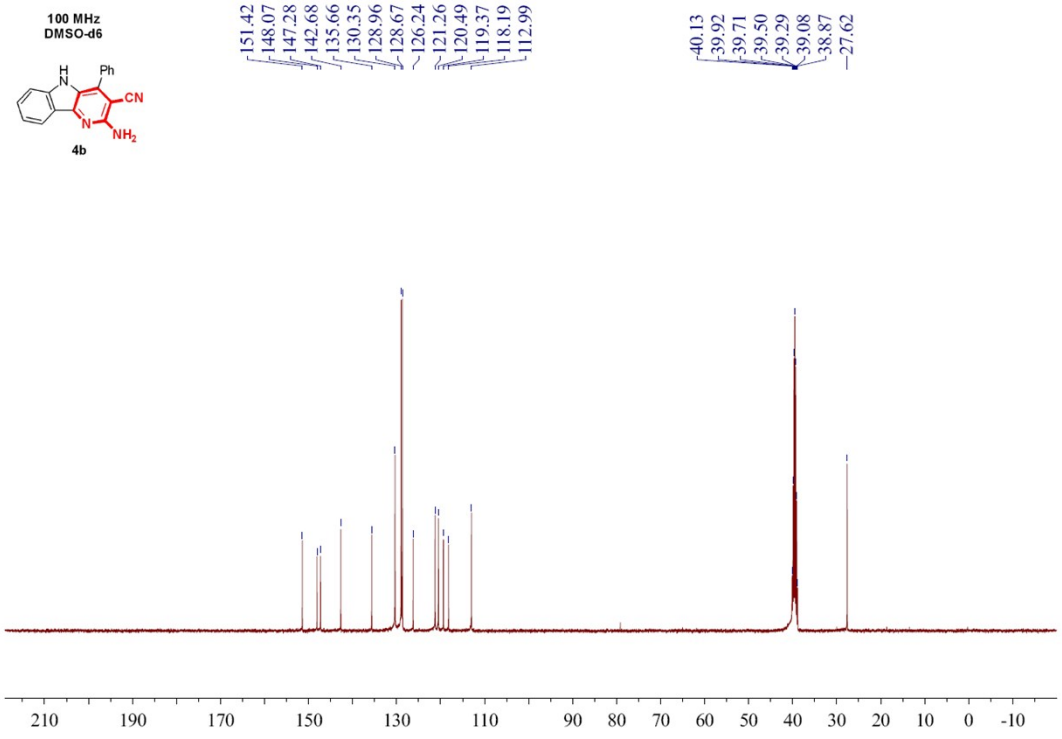

9.825
8.088
8.061
7.968
7.945
7.387
6.872
6.688
3.781
3.774
3.380
3.297
2.500
2.496
300 MHz
DMSO-d6
SO2Me
MeO
MeO
NH2
3ql
0.93
2.08
2.05
0.99
1.84
1.00
2.73
2.76
3.00
10.5 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5


180.29
152.20
144.62
143.90
142.19
141.78
135.60
128.78
127.10
116.48
109.82
102.01
94.29
55.67
55.34
43.43
40.33
40.05
39.78
39.50
39.22
38.94
38.66
75 MHz
DMSO-d6
SO2Me
MeO
MeO
NH2
3ql
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10


600 MHz
CDCl3
NHAc
4a
9.594
9.549
7.936
7.924
7.690
7.679
7.446
7.348
7.255
7.197
6.960
1.951
1.89
1.00
2.12
1.27
2.18
1.96
1.07
3.00


150 MHz
CDCl3
NHAc
4a
187.96
168.68
137.77
136.36
131.93
128.41
128.26
126.84
124.43
124.19
122.61
120.87
120.06
112.15
77.21
77.00
76.79
23.41


11.868
8.222
8.202
8.158
8.140
7.672
7.651
7.629
7.611
7.596
7.579
7.561
7.299
7.281
7.263
4.511
3.421
2.469
400 MHz
DMSO-d6
Ph
CN
NH2
4b
0.94
0.96
2.08
5.20
1.04
2.00
13.5
12.5
11.5
10.5
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5


151.42
148.07
147.28
142.68
135.66
130.35
128.96
128.67
126.24
121.26
120.49
119.37
118.19
112.99
40.13
39.92
39.71
39.50
39.29
39.08
38.87
27.62
100 MHz
DMSO-d6
Ph
CN
NH2
4b
210
190
170
150
130
110
90
80
70
60
50
40
30
20
10
0
-10