(*54 pages*)

Supporting Information For

One-pot cascade synthesis of *N*-methoxyisoquinolinediones *via* Rh(III)-catalyzed carbenoid insertion C–H activation/cyclization

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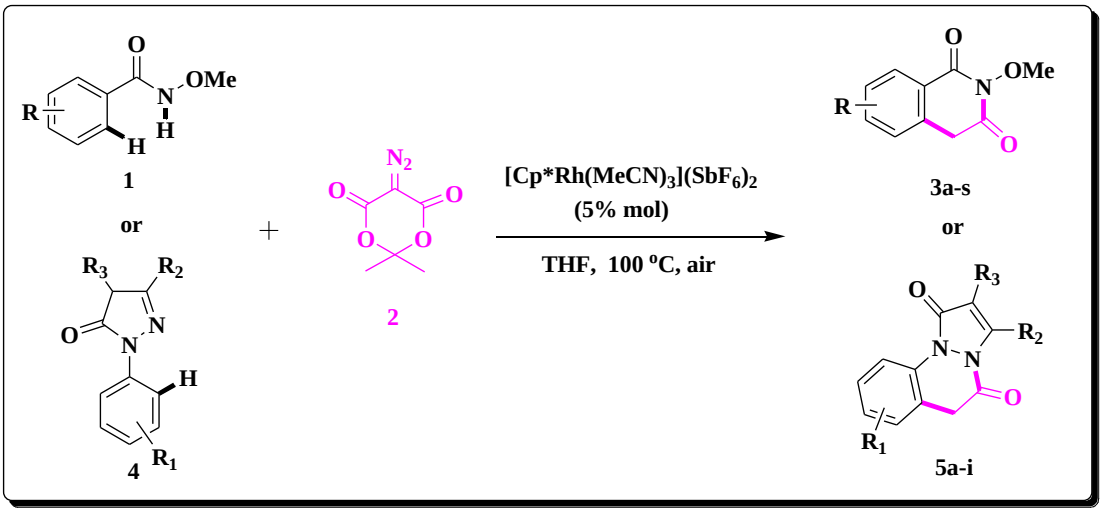
Table of contents

General methods and materials:

$[Cp^*RhCl_2]_2$ and $AgSbF_6$ were purchased from Aldrich and used without further purification. Catalysts $[Cp^*Rh(OAc)_2]_2$[S1] and $[Cp^*Rh(MeCN)_3][SbF_6]_2$,[S2] substrates *N*-methoxybenzamides,[S3-6] 3-methyl-1-phenyl-1*H*-pyrazol-5(4*H*)-ones[S7-10] and 5-diazo-2,2-dimethyl-1,3-dioxane-4,6-dione[S11] were synthesized according to published procedures. Other chemicals were purchased from commercial suppliers and were dried and purified when necessary. The water used was re-distillated and ion-free.

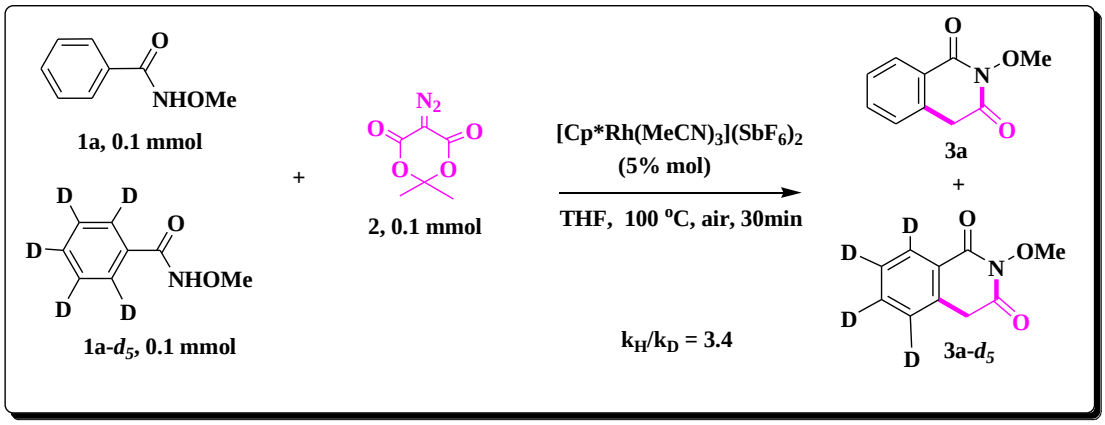
Melting points were determined on a WRS-1B digital instrument without correction. ^1H and ^{13}C NMR spectra were recorded on Varian Mercury-Plus 400 NMR and Varian Mercury-Plus 500 NMR instruments (^1H 400 MHz; ^{13}C 100 MHz) in either $CDCl_3$ or DMSO-d_6. Abbreviations for data quoted are s, singlet; brs, broad singlet; d, doublet; t, triplet; dd, doublet of doublets; m, multiplet. Mass spectra and high-resolution mass spectra were measured on an agilent TOF-G6230B mass spectrometer. Thin-layer chromatographies were done on pre-coated silica gel 60 F254 plates (Merck). Silica gel 60H (200-300 mesh) manufactured by Qingdao Haiyang Chemical Group Co. (China) was used for general chromatography.

General procedure for C–H activation/cyclization:

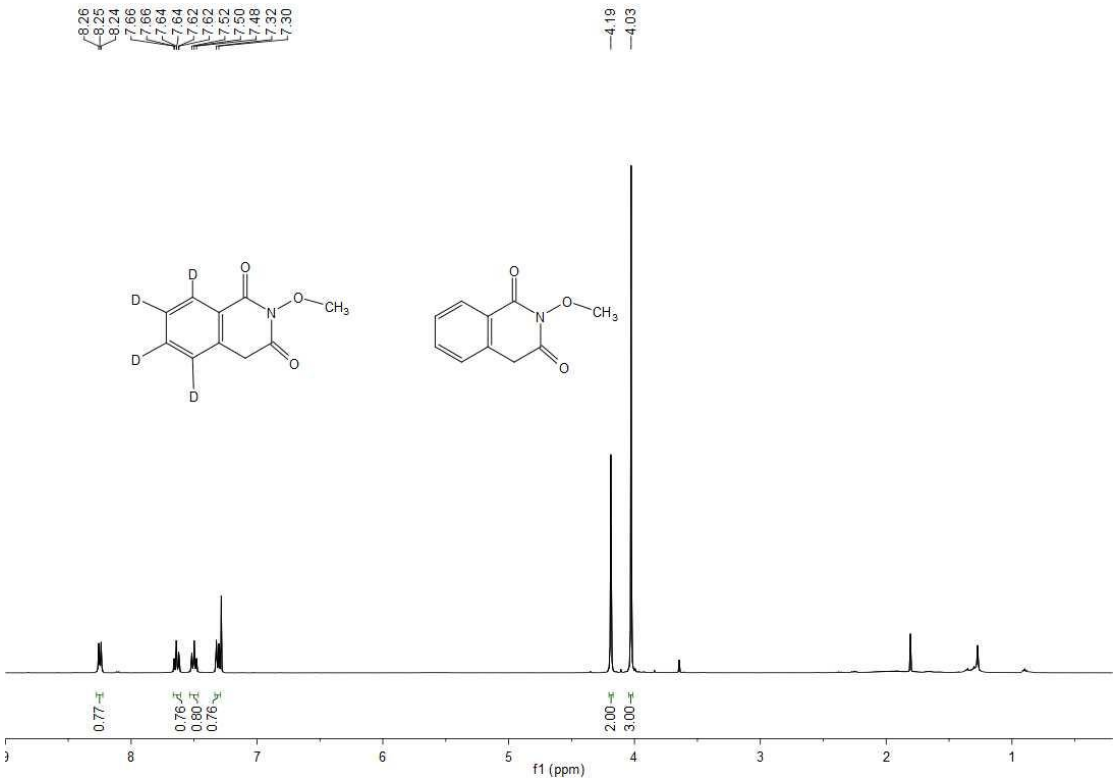


The mixture of $[Cp^*Rh(MeCN)_3][SbF_6]_2$ (8.3 mg, 0.01mmol, 0.05 equiv), substrate **1** or **4** (0.20 mmol, 1.0 equiv), **2** (0.22 mmol, 1.1 equiv) and THF (1.0 mL) were stirred at 100 ℃ for 5 h or 12 h under air. The resulting mixture was cooled to room temperature, silica gel column directly to give the desired product **3a-s** and **5a-i**.

General procedure for estimation of the KIE:

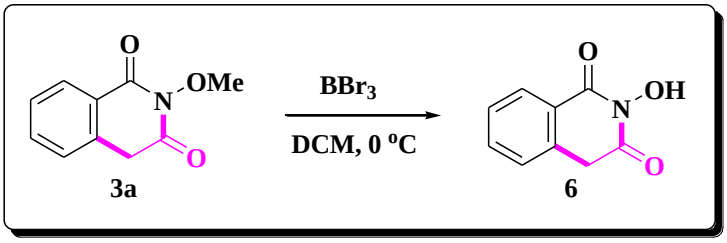


An equimolar mixture of **1a** and [D_5]-**1a** were allowed to react with **2** (1:1:1 ratio) in the presence of 5% mol [$Cp^*Rh^{III}(MeCN)_3$](SbF_6)$_2$. The reaction was stopped after 30 min, and the product generated was isolated by using column chromatography and was analyzed by ^{1}H NMR spectroscopy ($CDCl_3$, 400 MHz). The four multiplets at d 8.25 (77% H), 7.64 (76%, 1H), 7.50 (80%, 1H), 7.31 (76%, 1H) were used for calculation and an average value of k_H/k_D = 3.4 was obtained.



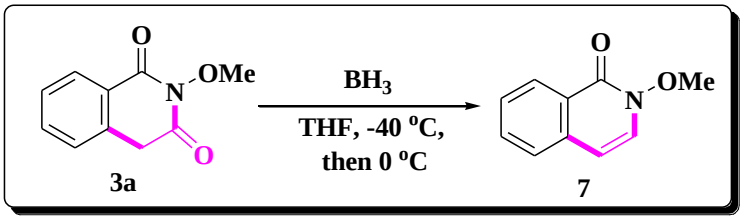
Procedure for demethylation and selective reduction:

(1) Procedure for etherdeprotection:



A suspension of **3a** (95 mg, 0.5 mmol) in dry DCM (3.0 mL) under N_2 was added borontribromide (1.0 mmol) and stirred at 0 ^oC for 4 h. It was slowly added water and allowed to reach room temperature, diluted with DCM (20 mL) and washed brine. The combined organic phase was dried (Na_2SO_4). After evaporation of the solvents under reduced pressure, the crude product was purified on a silica gel column to afford the product **6**.

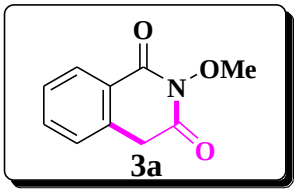
(2) Procedure for selective reduction:



A suspension of **3a** (95 mg, 0.5 mmol) in dry THF (3.0 mL) under N_2 was added borane (1.0 mmol) -40 ^oC and the reaction mixture were stirred at 0 ^oC for 4 h. Then the reaction solution was slowly added water and allowed to reach room temperature, diluted with EtOAc (20 mL) and washed brine. The combined organic phase was dried (Na_2SO_4). After evaporation of the solvents under reduced pressure, the crude product was purified on a silica gel column to give the product **7**.

Characterizations of products 3a-s, 5a-i and 6-7:

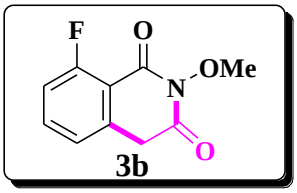
2-methoxyisoquinoline-1, 3(2*H*, 4*H*)-dione



3a

This compound was obtained in 80 % yield as a yellow solid. Mp 100-101 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.24 (d, *J* = 7.8 Hz, 1H), 7.63 (t, *J* = 7.4 Hz, 1H), 7.49 (t, *J* = 7.4 Hz, 1H), 7.35 – 7.23 (m, 1H), 4.18 (s, 2H), 4.02 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.6, 161.2, 134.0, 133.2, 129.2, 128.0, 127.4, 125.3, 64.2, 37.6; HRMS (ESI) calcd for 192.0661 ($[M+H]^+$), found 192.0658 ($[M+H]^+$).

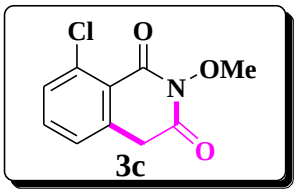
8-fluoro-2-methoxyisoquinoline-1, 3(2*H*, 4*H*)-dione



3b

This compound was obtained in 68 % yield as a yellow solid. Mp 180-182 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.62-7.57 (m, 1H), 7.17 (dd, *J* = 10.6, 8.8 Hz, 1H), 7.11 (d, *J* = 7.7 Hz, 1H), 4.18 (s, 2H), 4.00 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 164.6, 162.8 (d, *J* = 266 Hz), 157.8 (d, *J* = 4.9 Hz), 135.4 (d, *J* = 5.4 Hz), 135.2 (d, *J* = 10.3 Hz), 123.2 (d, *J* = 4.3 Hz), 116.4 (d, *J* = 21.4 Hz), 114.0 (d, *J* = 5.5 Hz), 64.2, 37.4 (d, *J* = 2.3 Hz); HRMS (ESI) calcd for 210.0566 ($[M+H]^+$), found 210.0562 ($[M+H]^+$).

8-chloro-2-methoxyisoquinoline-1, 3(2*H*, 4*H*)-dione

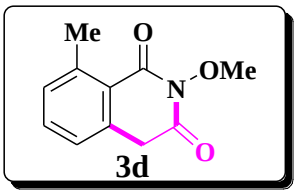


3c

This compound was obtained in 37 % yield as a yellow solid. Mp 143-145 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.54 – 7.43 (m, 2H), 7.21 (d, *J* = 6.8 Hz, 1H), 4.16 (s,

2H), 4.00 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 164.2, 158.8, 136.9, 136.1, 133.6, 131.9, 126.4, 122.0, 64.2, 37.8. HRMS (ESI) calcd for 226.0271 (^{35}Cl, $[M+H]^+$) and 228.0241 (^{37}Cl, $[M+H]^+$), found 226.0278 (^{35}Cl, $[M+H]^+$) and 228.0246 (^{37}Cl, $[M+H]^+$).

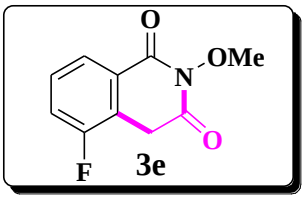
2-methoxy-8-methylisoquinoline-1, 3(2*H*, 4*H*)-dione



3d

This compound was obtained in 76 % yield as a yellow solid. Mp 145-146 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.44 (t, *J* = 7.6 Hz, 1H), 7.25 (d, *J* = 7.8 Hz, 1H), 7.12 (d, *J* = 7.6 Hz, 1H), 4.12 (s, 2H), 3.99 (s, 3H), 2.78 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.2, 161.6, 143.4, 134.4, 133.0, 131.7, 125.6, 123.2, 64.0, 37.9, 23.2. HRMS (ESI) calcd for 206.0817 ($[M+H]^+$), found 206.0826 ($[M+H]^+$).

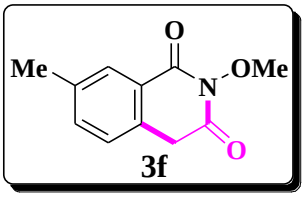
5-fluoro-2-methoxyisoquinoline-1,3(2*H*,4*H*)-dione



3e

This compound was obtained in 73 % yield as a yellow solid. Mp 152-154 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.05 (d, *J* = 8.0 Hz, 1H),7.52-7.47 (m, 1H), 7.37 (t, *J* = 8.4 Hz, 1H), 4.13 (s, 2H), 4.01 (s, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ: 164.4, 159.0 (d, *J* = 249 Hz), 160.2 (d, *J* = 3.3 Hz), 129.4 (d, *J* = 7.9 Hz), 127.2(d, *J* = 3.4 Hz), 124.8 (d, *J* = 3.6 Hz), 120.8 (d, *J* = 18.5 Hz), 120.4(d, *J* = 20.4 Hz), 64.0, 31.8 (d, *J* = 2.6 Hz); HRMS (ESI) calcd for 216.0566 ($[M+H]^+$), found 216.0569 ($[M+H]^+$).

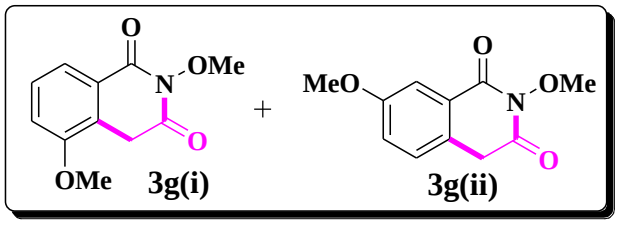
2-methoxy-7-methylisoquinoline-1, 3(2*H*, 4*H*)-dione



3f

This compound was obtained in 69 % yield as a yellow solid. Mp 142-143 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.93 (s, 1H), 7.34 (d, *J* = 7.8 Hz, 1H), 7.09 (d, *J* = 7.8 Hz, 1H), 4.03 (s, 2H), 3.91 (s, 3H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.8, 161.2, 138.0, 135.0, 130.2, 129.2, 127.3, 125.0, 64.0, 37.2, 21.0; HRMS (ESI) calcd for 206.0817 ($[M+H]^+$), found 206.0815 ($[M+H]^+$).

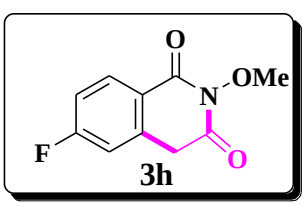
2,7-dimethoxyisoquinoline-1,3(2*H*,4*H*)-dione
and 2,5-dimethoxyisoquinoline-1,3(2*H*,4*H*)-dione



3g(i) **3g(ii)**

This compound was obtained in 79 % yield [mixture of **3g (i)** and **3g(ii)** (1.1 : 1)] as a yellow solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.82 [d, *J* = 8.0 Hz, 1H, **3g (i)**], 7.67 [d, *J* = 1.6 Hz, 1H, **3g(ii)**], 7.45 [t, *J* = 8.0 Hz, 1H, **3g (i)**], 7.21 – 7.16 [m, 2H, **3g(ii)**], 7.12 [d, *J* = 8.0 Hz, 1H, **3g (i)**], 4.11 [s, 2H, **3g(ii)**], 4.02 [s, 2H, **3g (i)**], 4.01 [s, 3H, **3g(ii)**], 4.00 [s, 3H, **3g (i)**], 3.92 [s, 3H, **3g (i)**], 3.90 [s, 3H, **3g(ii)**]; ^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.8, 165.7, 161.2, 159.3, 155.8, 128.8, 128.6, 126.2, 126.2, 125.3, 122.4, 122.2, 120.4, 114.6, 111.2, 64.1, 64.1, 55.8, 55.7, 36.9, 33.1; HRMS (ESI) calcd for 222.0766 ($[M+H]^+$), found 222.0767 ($[M+H]^+$).

6-fluoro-2-methoxyisoquinoline-1, 3(2*H*, 4*H*)-dione

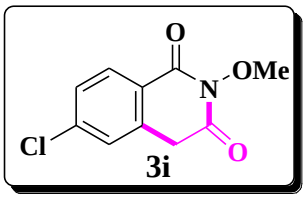


3h

This compound was obtained in 76 % yield as a yellow solid. Mp 157-158 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.25 (dd, *J* = 8.8, 5.6 Hz, 1H), 7.18-7.16 (m, 1H), 7.01 (d,

J = 8.0 Hz, 1H), 4.17 (s, 2H), 4.00 (s, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ: 166.1 (d, *J* =257 Hz), 164.9, 160.0 , 136.0 (d, *J* = 9.7 Hz), 132.4(d, *J* = 9.9 Hz), 121.6 (d, *J* = 2.6 Hz), 116.0 (d, *J* = 22.3 Hz), 114.2 (d, *J* = 23.0 Hz), 64.0, 37.6 (d, *J* =1.7 Hz); HRMS (ESI) calcd for 210.0566 ($[M+H]^+$), found 210.0564($[M+H]^+$).

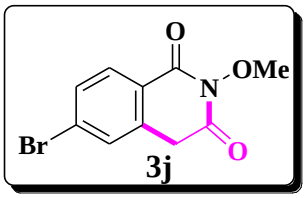
6-chloro-2-methoxyisoquinoline-1, 3(2*H*, 4*H*)-dione



3i

This compound was obtained in 47 % yield as a yellow solid. Mp 195-196 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.17 (d, *J* = 8.4 Hz, 1H), 7.47 (dd, *J* = 8.4, 2.0 Hz, 1H), 7.32 (d, *J* = 0.6 Hz, 1H), 4.15 (s, 2H), 4.01 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 164.8, 160.4, 140.8, 134.8, 130.6, 128.8, 127.4, 123.8, 64.4, 37.2; HRMS (ESI) calcd for 226.0271 (^{35}Cl, $[M+H]^+$) and 228.0241 (^{37}Cl, $[M+H]^+$), found 226.0264 (^{35}Cl, $[M+H]^+$) and 228.0231 (^{37}Cl, $[M+H]^+$).

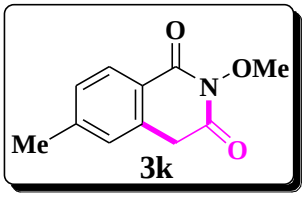
6-bromo-2-methoxyisoquinoline-1, 3(2*H*, 4*H*)-dione



3j

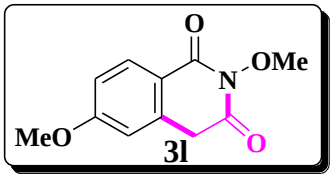
This compound was obtained in 66 % yield as a yellow solid. Mp 202-203 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.09 (d, *J* = 8.4 Hz, 1H), 7.63 (d, *J* = 8.4 Hz, 1H), 7.50 (s, 1H), 4.16 (s, 2H), 4.01 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 164.8, 160.4, 134.8, 131.6, 130.6, 130.4, 129.2, 124.0, 64.4, 37.2; HRMS (ESI) calcd for 269.9766 (^{79}Br, $[M+H]^+$) and 271.9745 (^{81}Br, $[M+H]^+$), found 269.9769 (^{79}Br, $[M+H]^+$) and 271.9751 (^{81}Br, $[M+H]^+$).

2-methoxy-6-methylisoquinoline-1, 3(2*H*, 4*H*)-dione



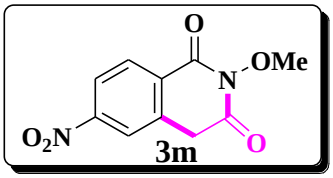
This compound was obtained in 81 % yield as a yellow solid. Mp 85-86 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.07 (d, *J* = 8.2 Hz, 1H), 7.25 (d, *J* = 8.2 Hz, 1H), 7.07 (s, 1H), 4.09 (s, 2H), 3.97 (s, 3H), 2.41 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.6, 161.2, 145.2, 133.2, 129.2, 129.0, 127.8, 122.8, 64.0, 37.6, 21.6; HRMS (ESI) calcd for 206.0817 ($[M+H]^+$), found 206.0815($[M+H]^+$).

2, 6-dimethoxyisoquinoline-1, 3(2*H*, 4*H*)-dione



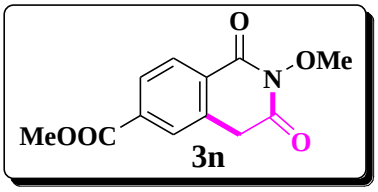
This compound was obtained in 80 % yield as a yellow solid. Mp 135-136 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.15 (d, *J* = 8.8 Hz, 1H), 6.99 (dd, *J* = 8.8, 2.4 Hz, 1H), 6.73 (d, *J* = 2.4 Hz, 1H), 4.12 (s, 2H), 4.00 (d, *J* = 2.4 Hz, 3H), 3.90 (s, 3H);^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.6, 164.0, 160.8, 135.4, 131.4, 118.0, 114.4, 111.6, 64.0, 55.6, 37.6; HRMS (ESI) calcd for 222.0766 ($[M+H]^+$), found 222.0770 ($[M+H]^+$).

2-methoxy-6-nitroisoquinoline-1, 3(2*H*, 4*H*)-dione



This compound was obtained in 72 % yield as a yellow solid. Mp 214-215 ^{o}C; ^{1}H NMR (400 MHz, DMSO-d_6) δ: 8.32 (s, 1H), 8.27 (m, 2H),4.39 (s, 2H), 3.86 (s, 3H); ^{13}C NMR (100 MHz, DMSO-d_6) δ: 165.6, 160.4, 150.4, 137.4, 130.8, 130.0, 123.2, 122.4, 63.8, 38.0; HRMS (ESI) calcd for 237.0511 ($[M+H]^{+)}$, found 237.0507 ($[M+H]^+$).

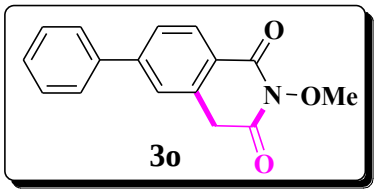
methyl 2-methoxy-1, 3-dioxo-1, 2, 3, 4-tetrahydroisoquinoline-6-carboxylate



3n

This compound was obtained in 79 % yield as a yellow solid. Mp 186-187 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.31 (d, *J* = 8.0 Hz, 1H), 8.12 (d, *J* = 8.0 Hz, 1H), 7.99 (s, 1H), 4.23 (s, 2H), 4.03 (s, 3H), 3.99 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.6, 165.0, 160.5, 135.0, 133.2, 129.4, 128.7, 128.6, 128.6, 64.3, 52.8, 37.4. HRMS (ESI) calcd for 250.0715 ($[M+H]^+$), found 250.0708 ($[M+H]^+$).

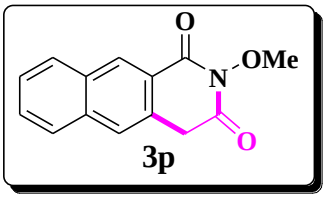
2-methoxy-6-phenylisoquinoline-1, 3(2*H*, 4*H*)-dione



3o

This compound was obtained in 56 % yield as a yellow solid. Mp 159-161 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.29 (d, J = 8.2 Hz, 1H), 7.71 (d, *J* = 8.0 Hz, 1H), 7.63 (d, *J* = 7.2 Hz, 2H), 7.49 (dd, *J* = 21.2, 7.6 Hz, 4H), 4.24 (s, 2H), 4.04 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.6, 161.2, 147.0, 139.0, 133.6, 130.0, 129.6, 128.8, 127.2, 126.8, 125.8, 124.0, 64.0, 37.6; HRMS (ESI) calcd for 268.0974 ($[M+H]^+$), found 268.0968 ($[M+H]^+$).

2-methoxybenzo[*g*]isoquinoline-1, 3(2*H*, 4*H*)-dione

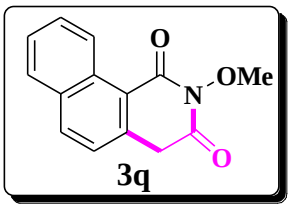


3p

This compound was obtained in 80 % yield as a yellow solid. Mp 197-199 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.71 (s, 1H), 7.91 (d, *J* = 8.2 Hz, 1H), 7.75 (d, *J* = 8.2 Hz, 1H), 7.63 (s, 1H), 7.56 (t, *J* = 7.4 Hz, 1H), 7.48 (t, *J* = 7.4 Hz, 1H), 4.22 (s, 2H), 3.97

(s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.8, 161.4, 135.7, 131.8, 131.3, 129.6, 129.5, 127.4, 127.3, 127.1, 126.3, 122.8, 64.2, 37.4; HRMS (ESI) calcd for 242.0817 ($[M+H]^+$), found 242.0815 ($[M+H]^+$).

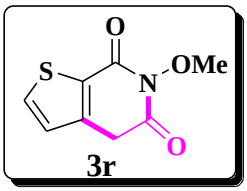
2-methoxybenzo[*h*]isoquinoline-1, 3(2*H*, 4*H*)-dione



3q

This compound was obtained in 54 % yield as a yellow solid. Mp 186-188 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.68 (d, *J* = 8.8 Hz, 1H), 8.08 (d, *J* = 8.4 Hz, 1H), 7.91 (d, *J* = 8.0 Hz, 1H), 7.79 – 7.70 (m, 1H), 7.63 (t, *J* = 7.2 Hz, 1H), 7.32 (d, *J* = 8.4 Hz, 1H), 4.29 (s, 2H), 4.08 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 164.8, 162.0, 135.8, 135.5, 133.0, 131.6, 129.6, 128.8, 127.0, 126.1, 124.5, 119.2, 64.0, 38.4; HRMS (ESI) calcd for 242.0817 ($[M+H]^+$), found 242.0813 ($[M+H]^+$).

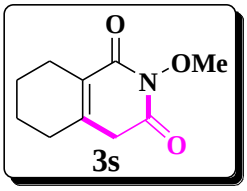
6-methoxythieno [2, 3-*c*] pyridine-5, 7(4*H*, 6*H*)-dione



3r

This compound was obtained in 83 % yield as a yellow solid. Mp 169-171 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 7.67 (d, *J* = 5.0 Hz, 1H), 6.93 (d, *J* = 5.0 Hz, 1H), 4.01 (s, 2H), 3.90 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.8, 156.8, 140.0, 135.2, 128.0, 126.6, 64.3, 35.6; HRMS (ESI) calcd for 198.0225 ($[M+H]^+$), found 198.0224 ($[M+H]^+$).

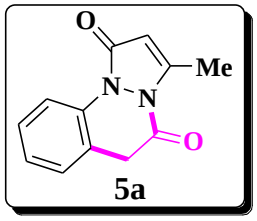
2-methoxy-5, 6, 7, 8-tetrahydroisoquinoline-1, 3(2*H*,4*H*)-dione



3s

This compound was obtained in 52 % yield as a yellow solid. Mp 105-107 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 3.93 (s, 3H), 3.42 (s, 2H), 2.38 (m, 2H), 2.18 (m, 2H), 1.72 (m, 4H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 165.6, 162.4, 145.6, 125.6, 64.0, 40.4, 29.6, 22.8, 21.6, 21.6; HRMS (ESI) calcd for 196.0974 ($[M+H]^+$), found 196.0979 ($[M+H]^+$).

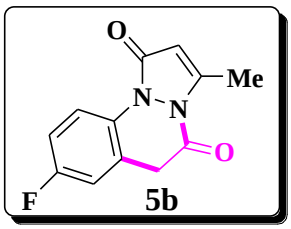
3-methyl-1*H*-pyrazolo [1, 2-*a*] cinnoline-1, 5(6*H*)-dione



5a

This compound was obtained in 83 % yield as a yellow solid. Mp 157-158 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.66 (d, *J* = 8.2 Hz, 1H), 7.37 (m, 1H), 7.23 – 7.17 (m, 2H), 5.68 (d, *J* = 0.6 Hz, 1H), 3.91 (s, 2H), 2.64 (d, *J* = 0.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 161.9, 161.7, 148.3, 132.8, 128.5, 127.7, 125.6, 118.7, 116.0, 105.1, 36.6, 15.9; HRMS (ESI) calcd for 215.0821 ($[M+H]^+$), found 215.0818 ($[M+H]^+$).

8-fluoro-3-methyl-1*H*-pyrazolo [1, 2-*a*] cinnoline-1, 5(6*H*)-dione

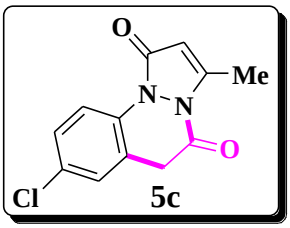


5b

This compound was obtained in 87 % yield as a yellow solid. Mp 164-166 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.67 (dd, *J* = 9.2, 5.0 Hz, 1H), 7.10-7.05 (m, 1H), 6.93 (dd, *J* = 8.2, 2.8 Hz, 1H), 5.67 (s, 1H), 3.90 (s, 2H), 2.64 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 161.6, 160.8, 159.8 (d, J = 246 Hz), 148.4, 129.2 (d, J = 2.8 Hz), 120.8 (d, J

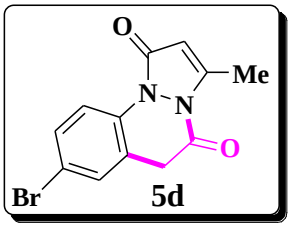
= 8.0 Hz), 117.6 (d, J = 8.2 Hz), 115.4 (d, J = 22.4 Hz), 114.4 (d, J = 23.8 Hz), 105.0, 36.4, 15.8; HRMS (ESI) calcd for 233.0726 ($[M+H]^+$), found 233.0720($[M+H]^+$).

8-chloro-3-methyl-1*H*-pyrazolo [1, 2-*a*] cinnoline-1, 5(6*H*)-dione



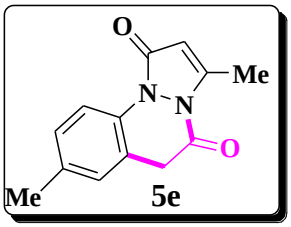
This compound was obtained in 90 % yield as a yellow solid. Mp 146-148 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ : 8.62 (d, *J* = 9.0 Hz, 1H), 7.31 (dd, *J* = 9.0, 2.0 Hz, 1H), 7.18 (d, *J* = 2.0 Hz, 1H), 5.65 (s, 1H), 3.87 (s, 2H), 2.63 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 161.6, 160.8, 148.8, 131.2, 130.4, 128.4, 127.4, 120.4, 117.2, 105.0, 36.0, 16.0; HRMS (ESI) calcd for 249.0431 (^{35}Cl, $[M+H]^+$) and 251.0401 (^{37}Cl, $[M+H]^+$), found 249.0427 (^{35}Cl, $[M+H]^+$) and 251.0400 (^{37}Cl, $[M+H]^+$).

8-bromo-3-methyl-1*H*-pyrazolo [1, 2-*a*] cinnoline-1, 5(6*H*)-dione



This compound was obtained in 43 % yield as a yellow solid. Mp 159-161 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.61 (d, J = 8.8 Hz, 1H), 7.49 (dd, J = 8.8, 2.0 Hz, 1H), 7.40 – 7.32 (m, 1H), 5.68 (s, 1H), 3.90 (s, 2H), 2.65 (d, J = 0.8 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 161.6, 160.8, 148.8, 131.8, 131.4, 130.4, 120.8, 118.2, 117.6, 105.2, 36.0, 16.0; HRMS (ESI) calcd for 292.9926 (^{79}Br, $[M+H]^+$) and 294.9905 (^{79}Br, $[M+H]^+$), found 292.9917 (^{79}Br, $[M+H]^+$) and 294.9897 (^{81}Br, $[M+H]^+$).

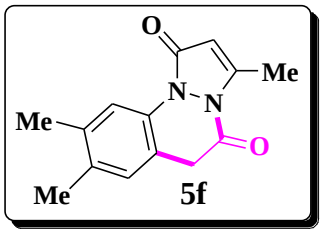
3, 8-dimethyl-1*H*-pyrazolo [1, 2-*a*] cinnoline-1, 5(6*H*)-dione



5e

This compound was obtained in 69 % yield as a yellow solid. Mp 171-172 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.52 (d, *J* = 8.4 Hz, 1H), 7.15 (d, *J* = 8.4 Hz, 1H), 6.99 (s, 1H), 5.65 (d, *J* = 0.8 Hz, 1H), 3.85 (s, 2H), 2.62 (d, *J* = 0.8 Hz, 3H), 2.34 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 161.8, 161.4, 148.0, 135.4, 130.4, 129.0, 128.0, 118.6, 115.8, 105.2, 36.4, 20.8, 16.0; HRMS (ESI) calcd for 229.0977 ($[M+H]^+$), found 229.0973 ($[M+H]^+$).

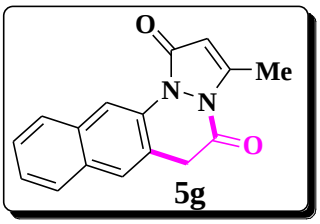
3, 8, 9-trimethyl-1*H*-pyrazolo [1, 2-*a*] cinnoline-1, 5(6*H*)-dione



5f

This compound was obtained in 85 % yield as a yellow solid. Mp 170-172 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.43 (s, 1H), 6.92 (s, 1H), 5.64 (s, 1H), 3.81 (s, 2H), 2.62 (s, 3H), 2.29 (s, 3H), 2.24 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ:162.0, 161.6, 147.8, 137.0, 134.2, 130.6, 128.4, 116.6, 115.8, 105.2, 36.0, 20.0, 19.4, 16.0; HRMS (ESI) calcd for 243.1134 ($[M+H]^+$), found 243.1143 ($[M+H]^+$).

3-methyl-1*H*-benzo[*g*] pyrazolo [1, 2-*a*] cinnoline-1, 5(6*H*)-dione

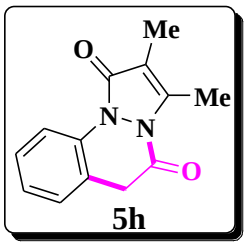


5g

This compound was obtained in 60 % yield as a yellow solid. Mp 201-203 ^{o}C; ^{1}H NMR (400 MHz, $CDCl_3$) δ: 9.10 (s, 1H), 7.98 – 7.39 (m, 5H), 5.73 (s, 1H), 4.07 (s, 2H), 2.67 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 162.4, 162.0, 148.8, 132.8, 130.8,

130.4, 128.4, 127.0, 126.8, 126.4, 126.0, 119.2, 113.6, 105.2, 37.2, 16.0; HRMS (ESI) calcd for 265.0977 ($[M+H]^+$), found 265.0970 ($[M+H]^+$).

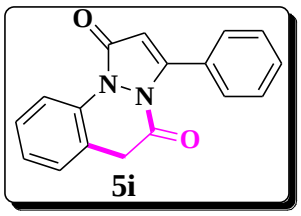
2,3-dimethyl-1*H*-pyrazolo [1, 2-*a*] cinnoline-1, 5(6*H*)-dione



5h

This compound was obtained in 53 % yield as a yellow solid. Mp 161-163 ^oC; ^1H NMR (400 MHz, $CDCl_3$) δ: 8.69 (d, J = 8.4 Hz, 1H), 7.41 – 7.34 (m, 1H), 7.24 – 7.16 (m, 2H), 3.89 (s, 2H), 2.61 (d, J = 0.6 Hz, 3H), 1.94 (d, J = 0.6 Hz, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 162.8, 161.4, 142.8, 132.8, 128.4, 127.6, 125.4, 119.0, 116.0, 112.2, 36.4, 13.6, 6.4; HRMS (ESI) calcd for 229.0977 ($[M+H]^+$), found 229.0973 ($[M+H]^+$).

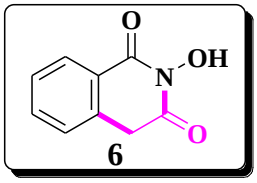
3-phenyl-1*H*-pyrazolo [1, 2-*a*] cinnoline-1, 5(6*H*)-dione



5i

This compound was obtained in 59 % yield as a yellow solid. Mp 202-204 ^oC; ^1H NMR (400 MHz, $CDCl_3$) δ: 8.63 (d, J = 8.2 Hz, 1H), 7.55 – 7.40 (m, 6H), 7.25 (t, J = 6.3 Hz, 2H), 5.96 (s, 1H), 3.92 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 162.0, 161.2, 150.6, 132.8, 130.4, 129.0, 128.8, 128.4, 128.0, 127.8, 126.0, 119.6, 116.8, 106.8, 36.8; HRMS (ESI) calcd for 277.0977 ($[M+H]^+$), found 277.0974($[M+H]^+$).

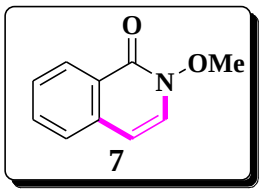
2-hydroxyisoquinoline-1, 3(2*H*, 4*H*)-dione



6

This compound was obtained in 76 % yield as a white solid. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.25 (d, *J* = 7.8 Hz, 1H), 7.67 (t, *J* = 7.2 Hz, 1H), 7.53 (t, *J* = 7.6 Hz, 1H), 7.35 (d, *J* = 7.8 Hz, 1H), 4.23 (s, 2H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 163.6, 160.2, 134.2, 133.0, 129.0, 128.2, 127.6, 124.0, 36.4; HRMS (ESI) calcd for 178.0504 ($[M+H]^+$), found 178.0496 ($[M+H]^+$).

2-methoxyisoquinolin-1(2*H*)-one



7

This compound was obtained in 68 % yield as a colourless oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ: 8.48 (d, *J* = 8.0 Hz, 1H), 7.71 – 7.65 (m, 1H), 7.61 – 7.50 (m, 2H), 7.36 (d, *J* = 7.6 Hz, 1H), 6.50 (d, *J* = 7.6 Hz, 1H), 4.13 (s, 3H); ^{13}C NMR (100 MHz, $CDCl_3$) δ: 158.2, 136.2, 132.3, 129.4, 127.7, 127.5, 126.9, 126.2, 105.6, 64.4; HRMS (ESI) calcd for 176.0712 ($[M+H]^+$), found 176.0716 ($[M+H]^+$).

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S7 Z. Fan, K. Wu, L. Xing, Q. Yao and A. Zhang, *Chem. Commun.*, 2014, **50**, 1682.

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S9 K. Wu, Z. Fan, Y. Xue, Q. Yao and A. Zhang, *Org. Lett.*, 2014, **16**, 42.

S10 M. I. Marzouk, G. H. Sayed, M. S. A. ElHalim and S. Y. Mansour, *Eur. J. Chem.*, 2014, **5**, 24.

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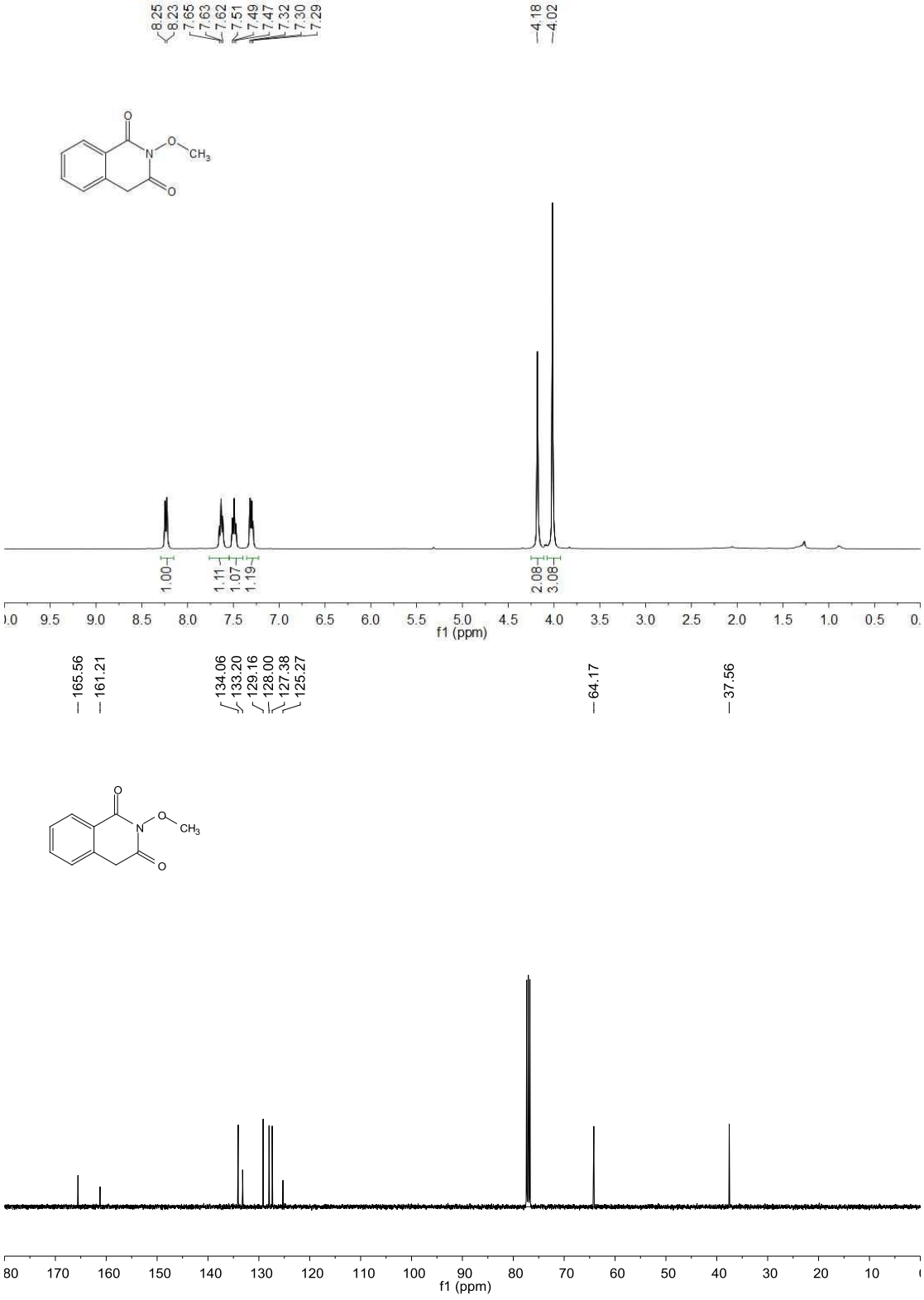
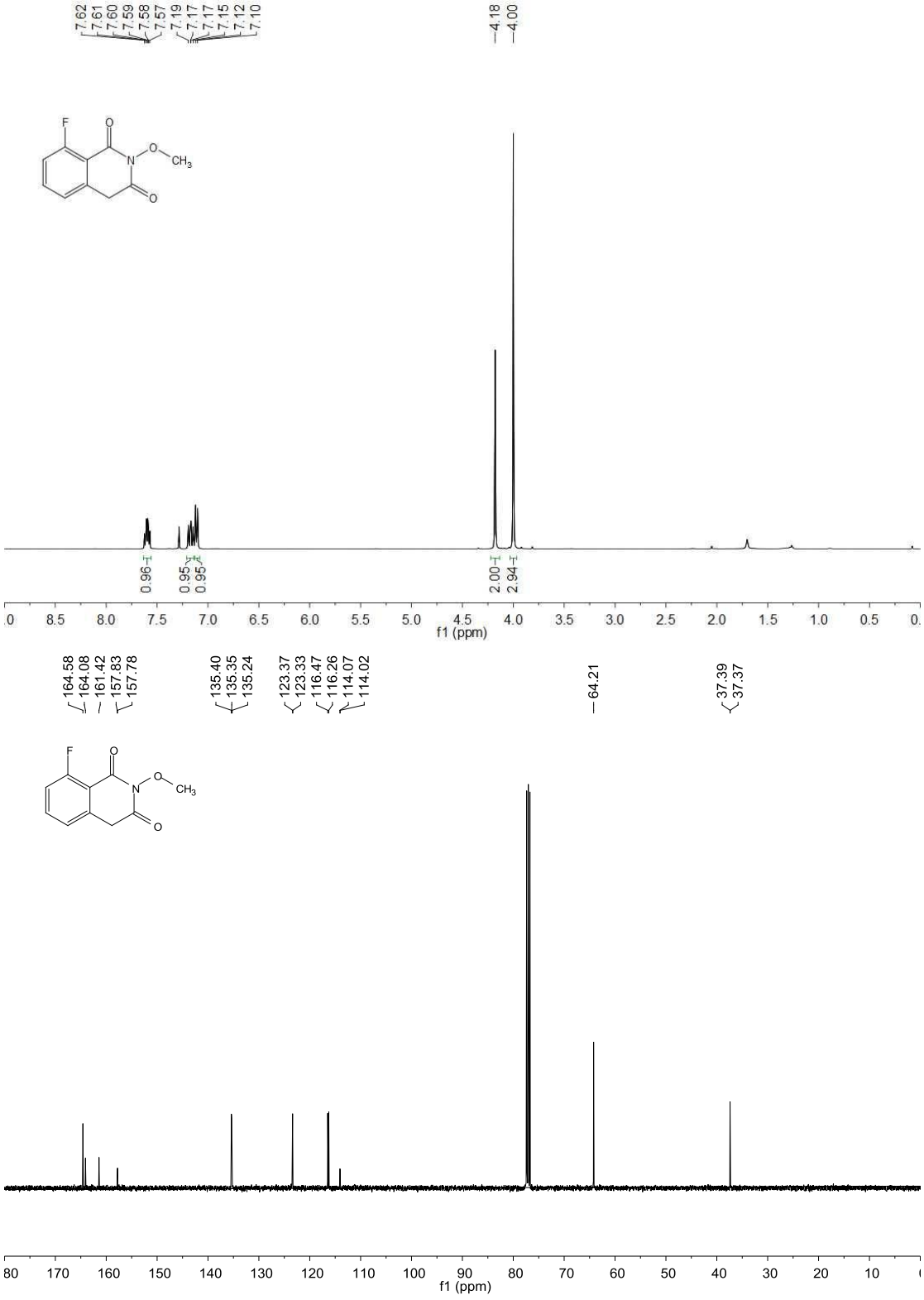
^{1}H and ^{13}C NMR spectra of products 3a-s, 5a-i and 6-7:

3a



3b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7.58
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7.17
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7.15
7.12
7.10
4.18
4.00
F
O
N
O
CH3
O
0.96
0.95
0.95
2.00
2.94
f1 (ppm)
164.58
164.08
161.42
157.83
157.78
135.40
135.35
135.24
123.37
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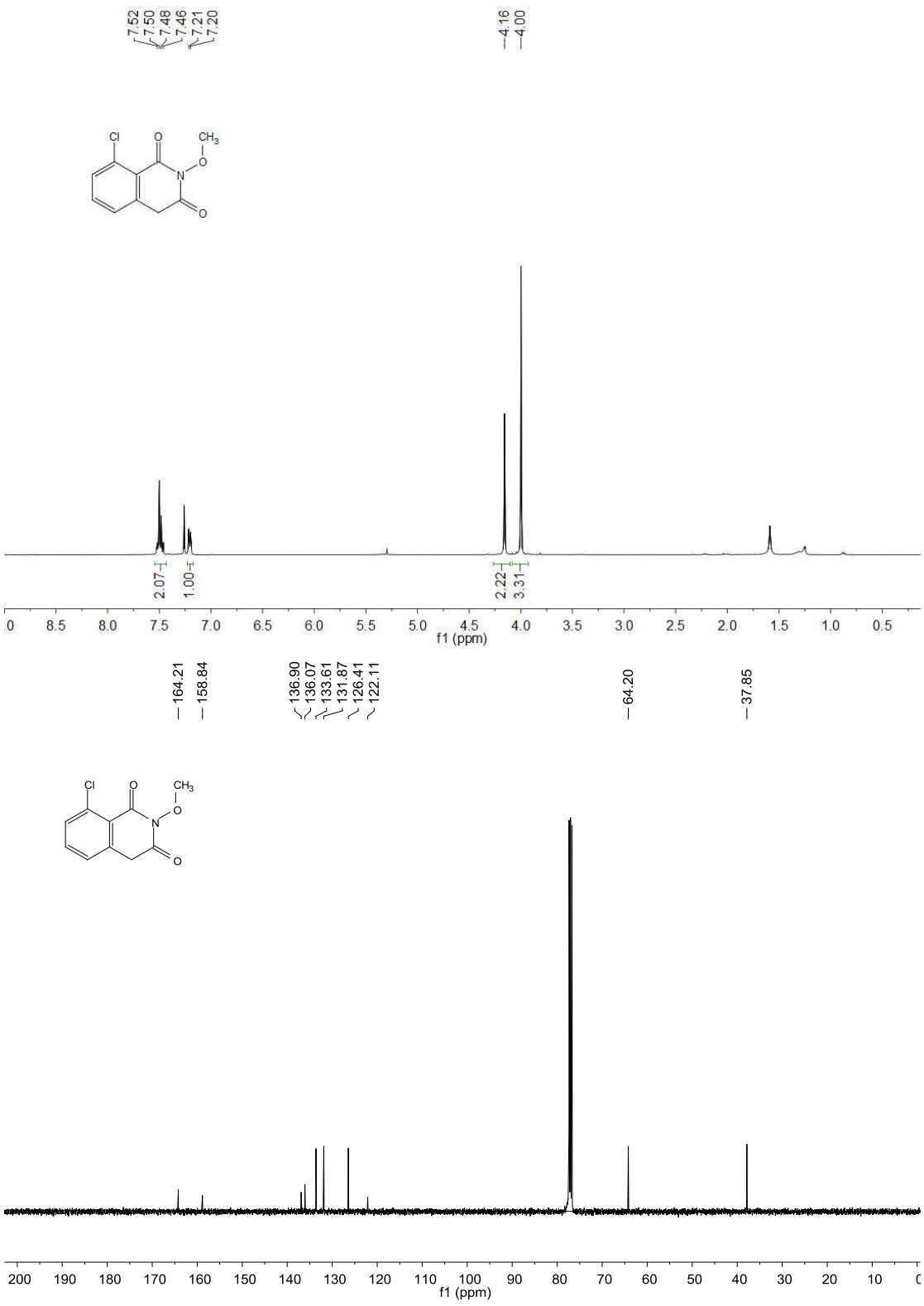
64.21
37.39
37.37
f1 (ppm)

3c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CH3
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O
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2.07
1.00
2.22
3.31
f1 (ppm)
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64.20
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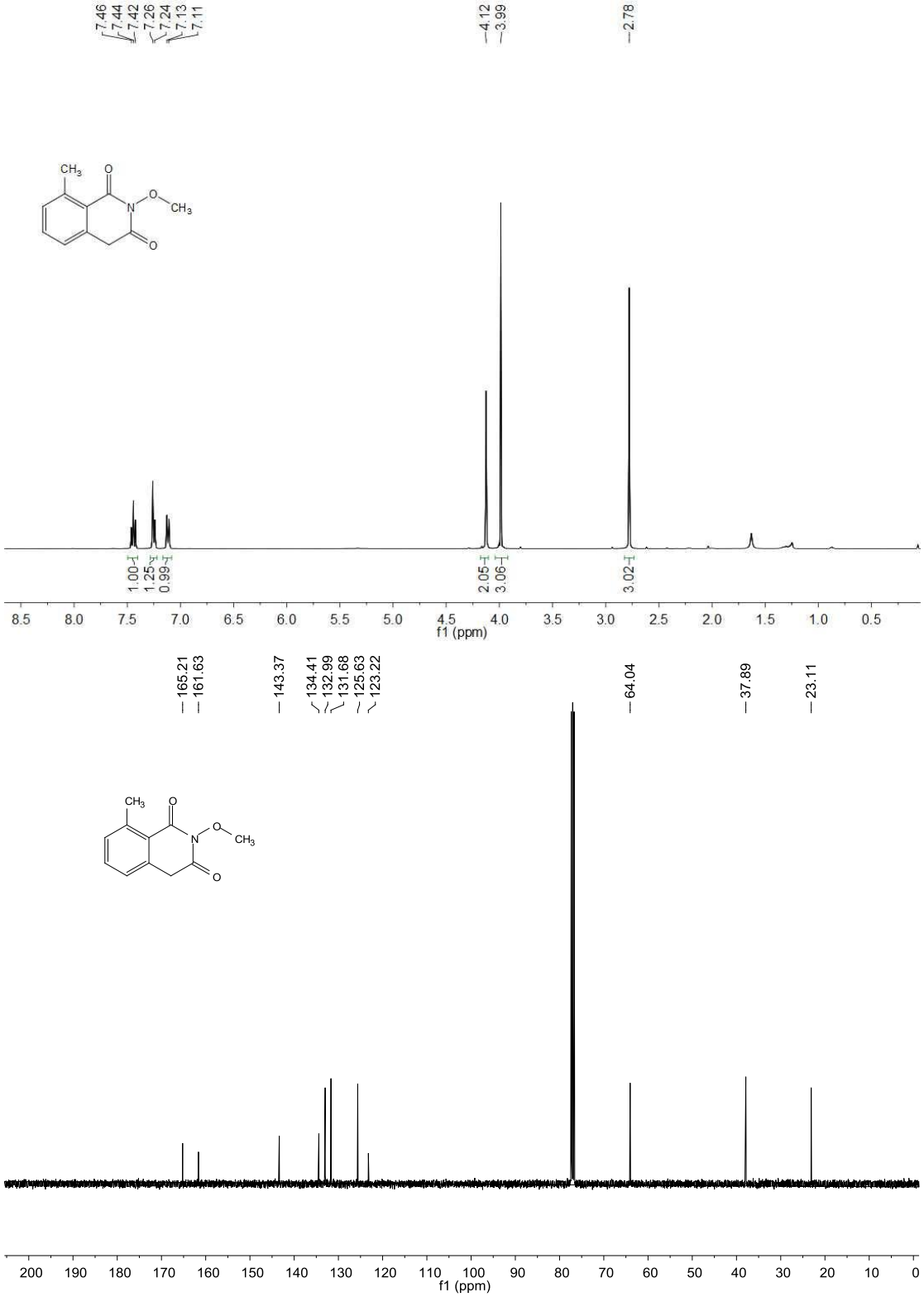
f1 (ppm)

3d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3.02
f1 (ppm)
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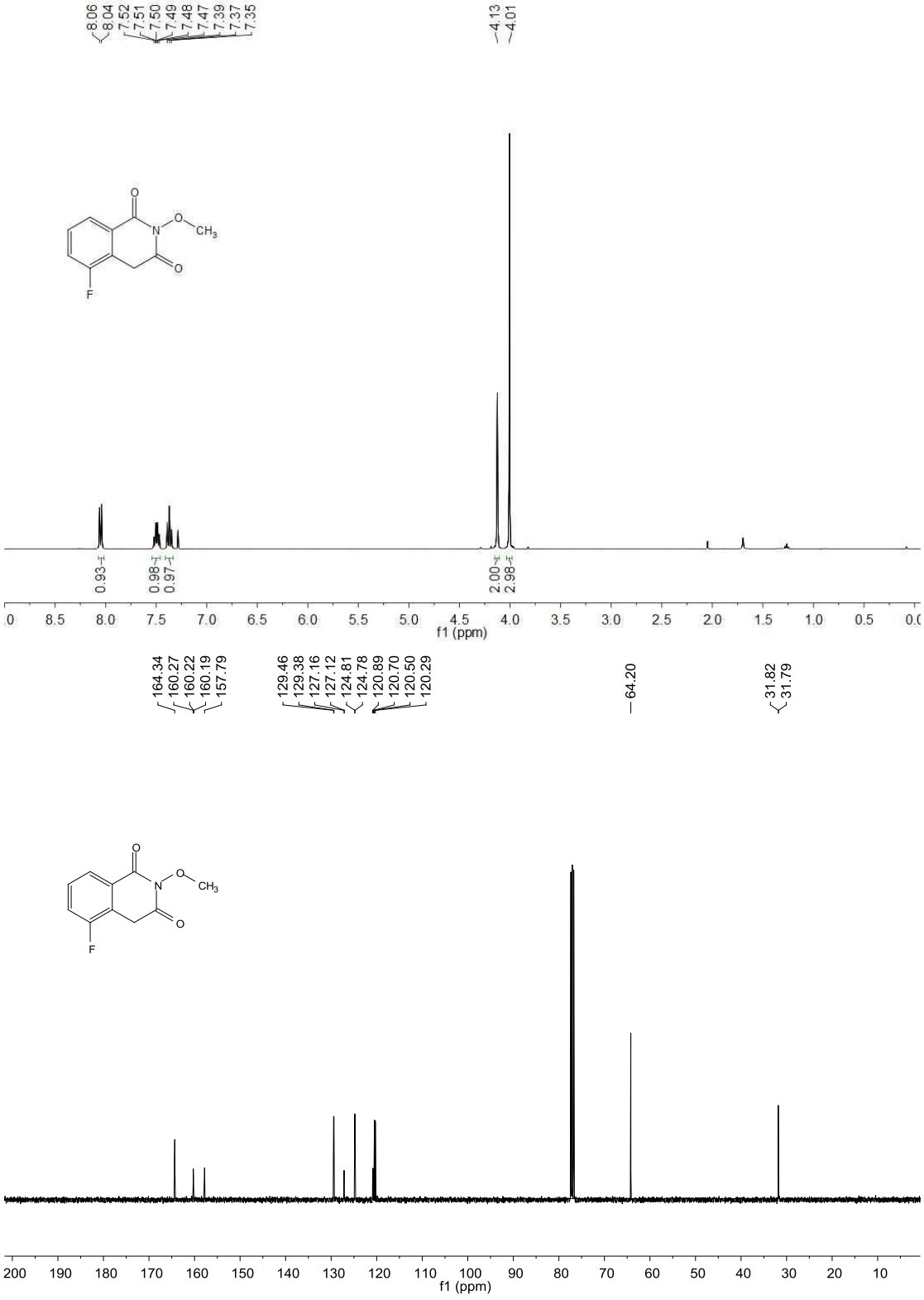
f1 (ppm)

3e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O
N
O
CH3
O
F
0.93
0.98
0.97
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2.98
f1 (ppm)
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160.27
160.22
160.19
157.79
129.46
129.38
127.16
127.12
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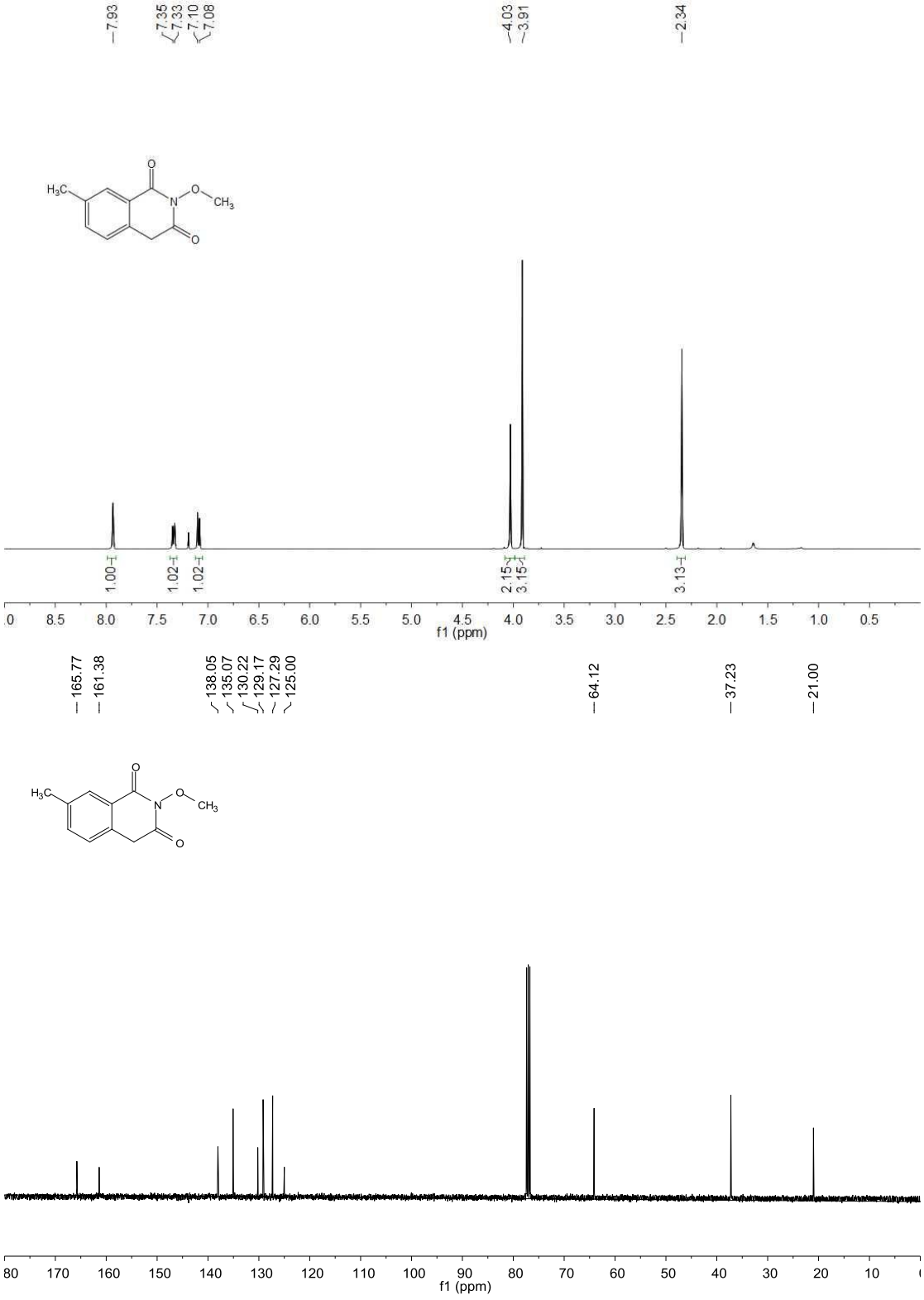
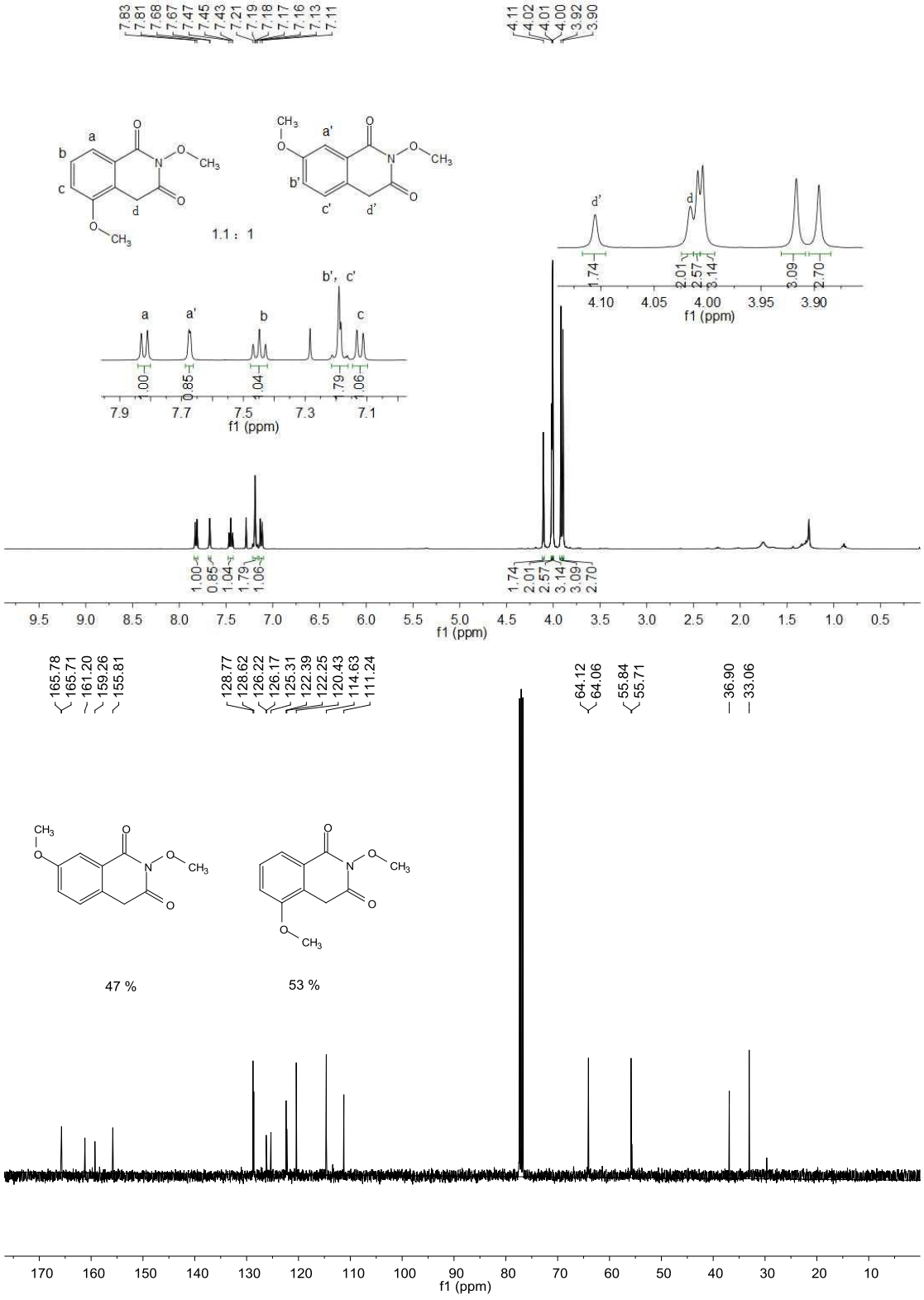
120.50
120.29
64.20
31.82
31.79
f1 (ppm)

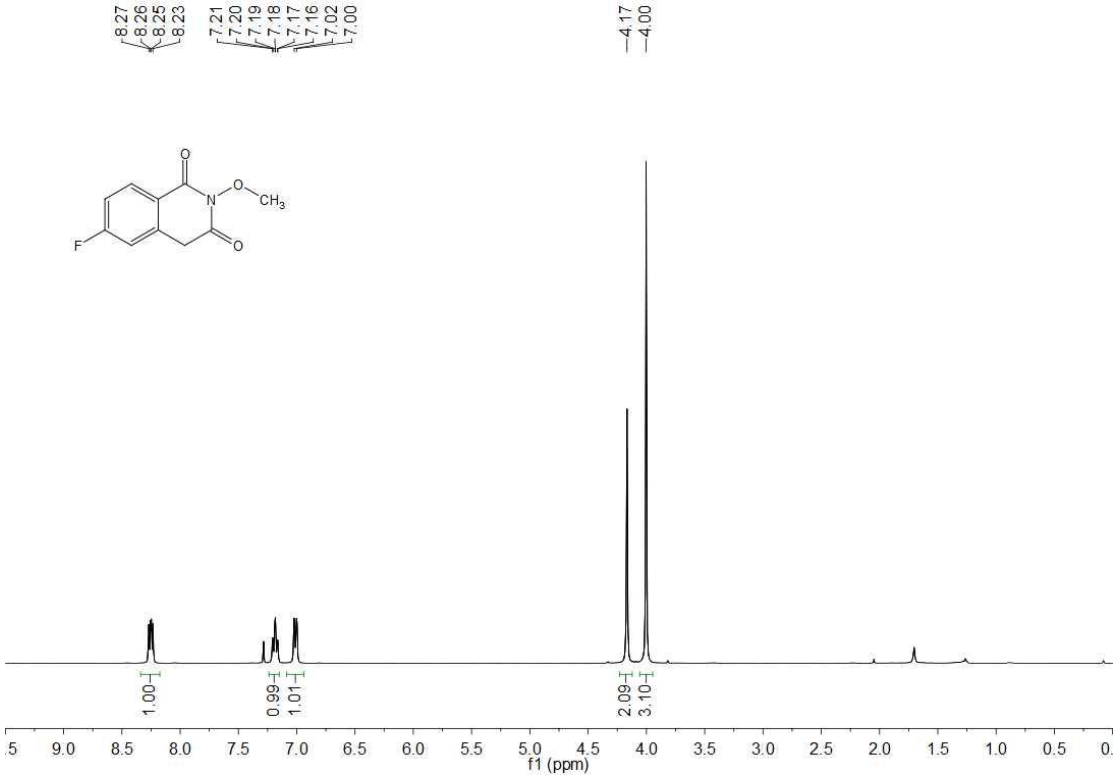
3f

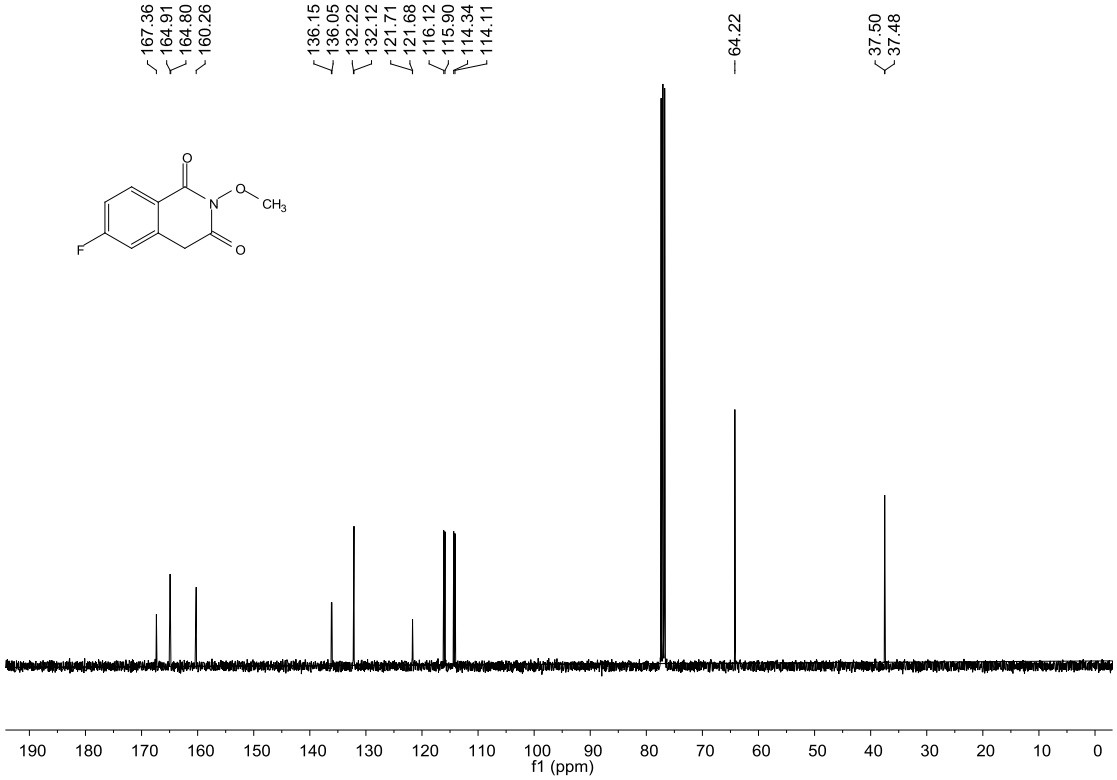

7.93
7.35
7.33
7.10
7.08
4.03
3.91
2.34
1.00
1.02
1.02
2.15
3.15
3.13
f1 (ppm)
165.77
161.38
138.05
135.07
130.22
129.17
127.29
125.00
64.12
37.23
21.00
f1 (ppm)

3g (i) and 3g (ii)

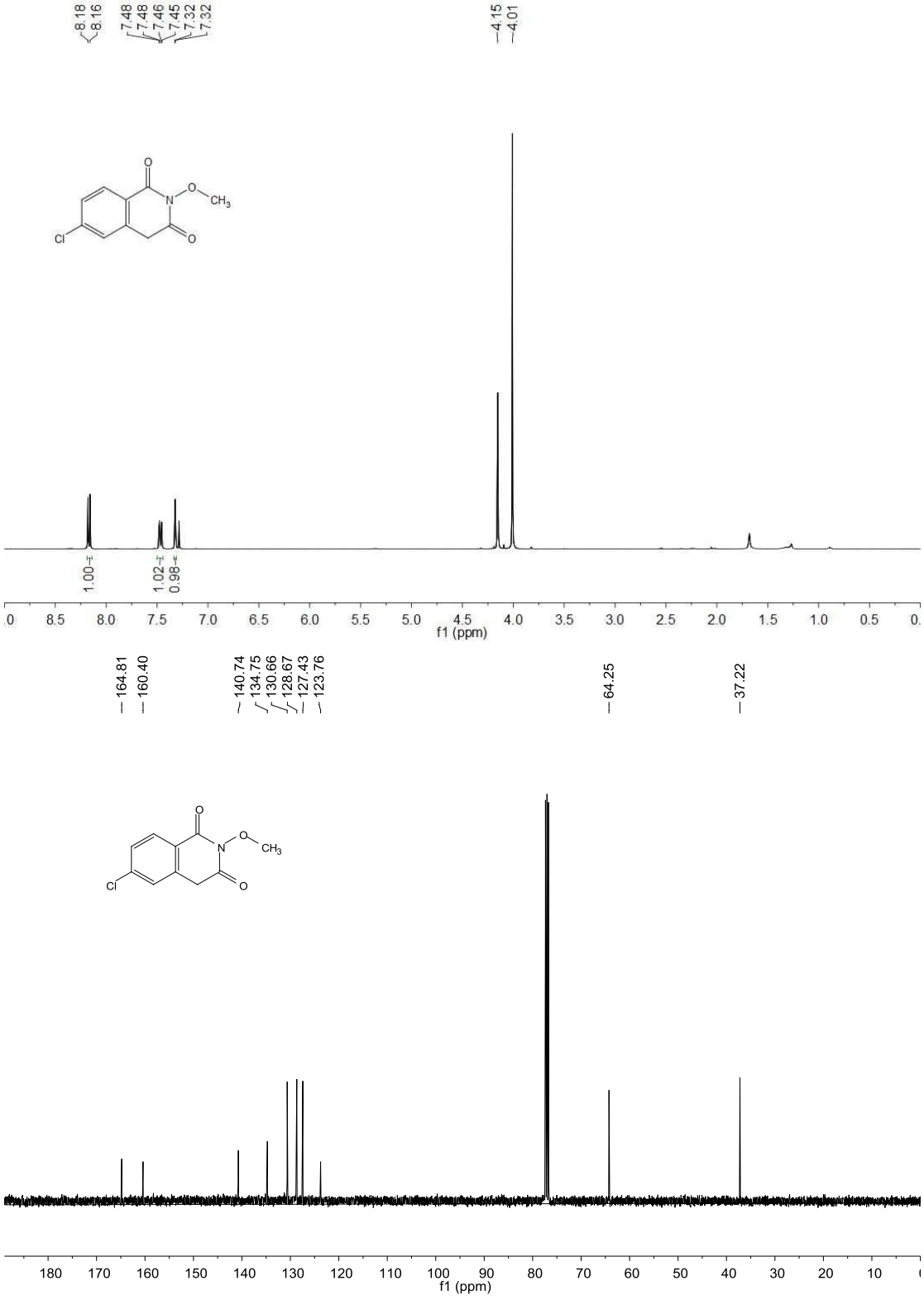

7.83
7.81
7.68
7.67
7.47
7.45
7.43
7.21
7.19
7.18
7.17
7.16
7.13
7.11
4.11
4.02
4.01
4.00
3.92
3.90
a
b
c
d
a'
b'
c'
d'
1.1 : 1
b', c'
1.00
0.85
1.04
1.79
1.06
1.74
2.01
2.57
3.14
3.09
2.70
f1 (ppm)
165.78
165.71
161.20
159.26
155.81
128.77
128.62
126.22
126.17
125.31
122.39
122.25
120.43
114.63
111.24
64.12
64.06
55.84
55.71
36.90
33.06
47 %
53 %

3h

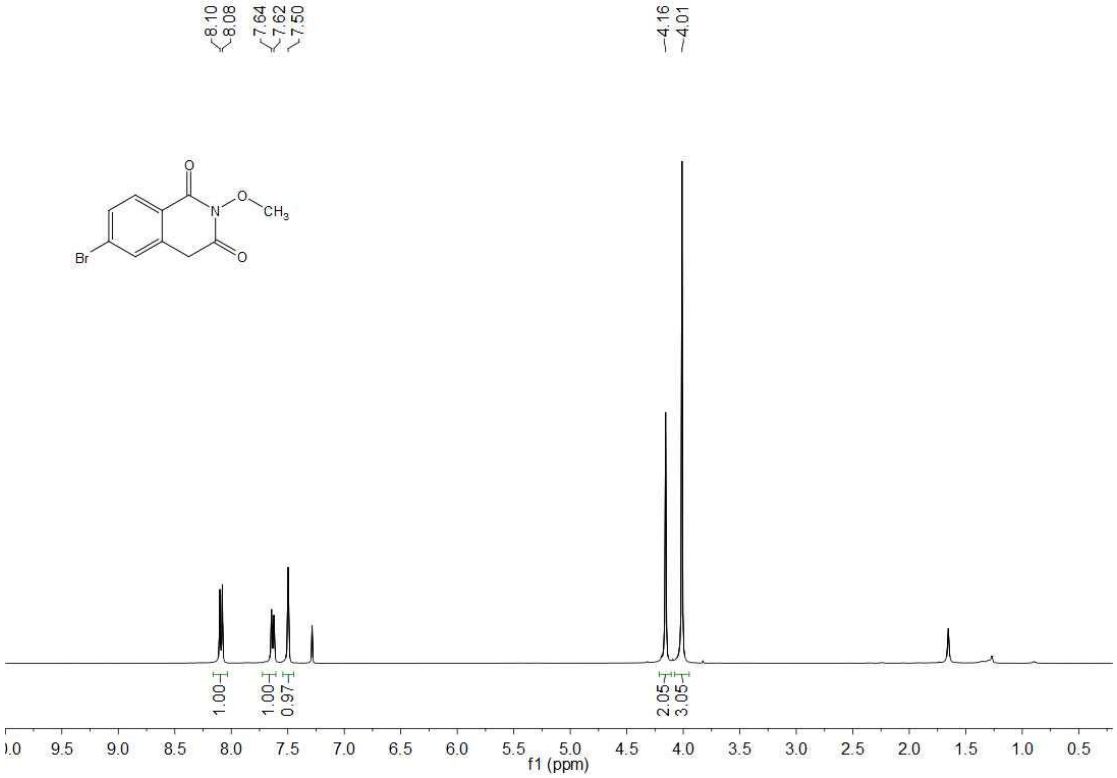

8.27
8.26
8.25
8.23
7.21
7.20
7.19
7.18
7.17
7.16
7.02
7.00
4.17
4.00
O
N
O
CH3
F
O
1.00
0.99
1.01
2.09
3.10
f1 (ppm)

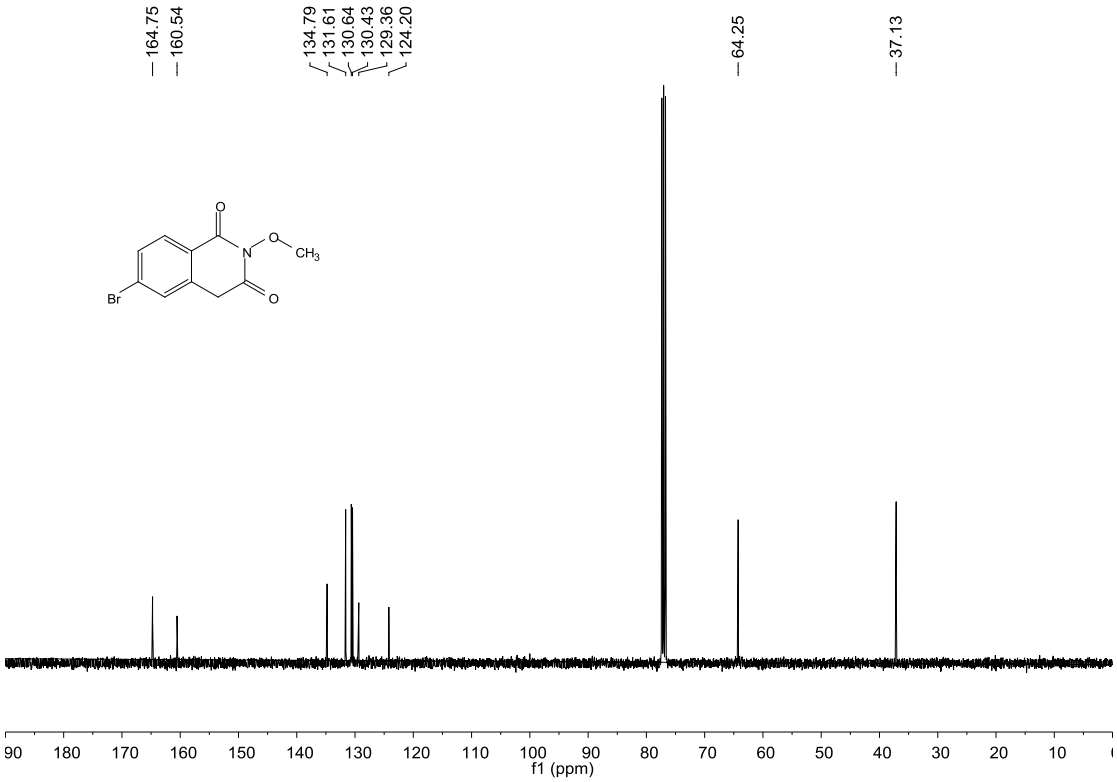

167.36
164.91
164.80
160.26
136.15
136.05
132.22
132.12
121.71
121.68
116.12
115.90
114.34
114.11
64.22
37.50
37.48
O
N
O
CH3
F
O
f1 (ppm)

3i

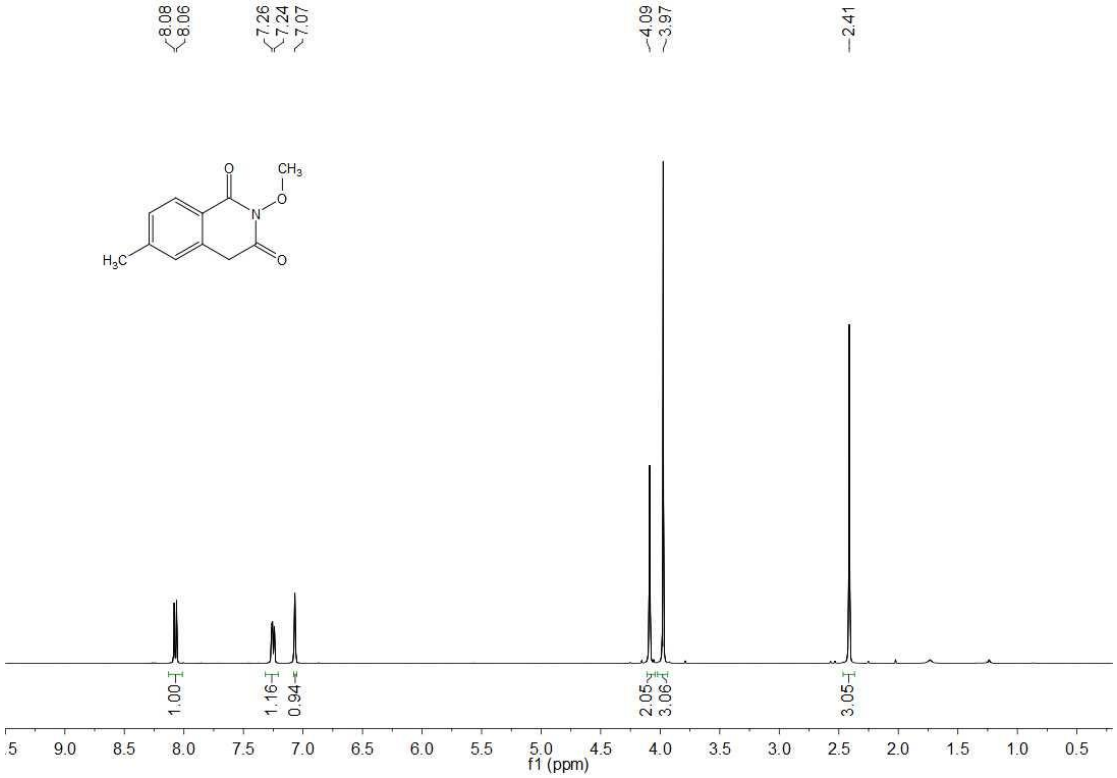

8.18
8.16
7.48
7.48
7.46
7.45
7.32
7.32
4.15
4.01
O
N
O
CH3
Cl
O
1.00
1.02
0.98
f1 (ppm)
164.81
160.40
140.74
134.75
130.66
128.67
127.43
123.76
64.25
37.22
f1 (ppm)

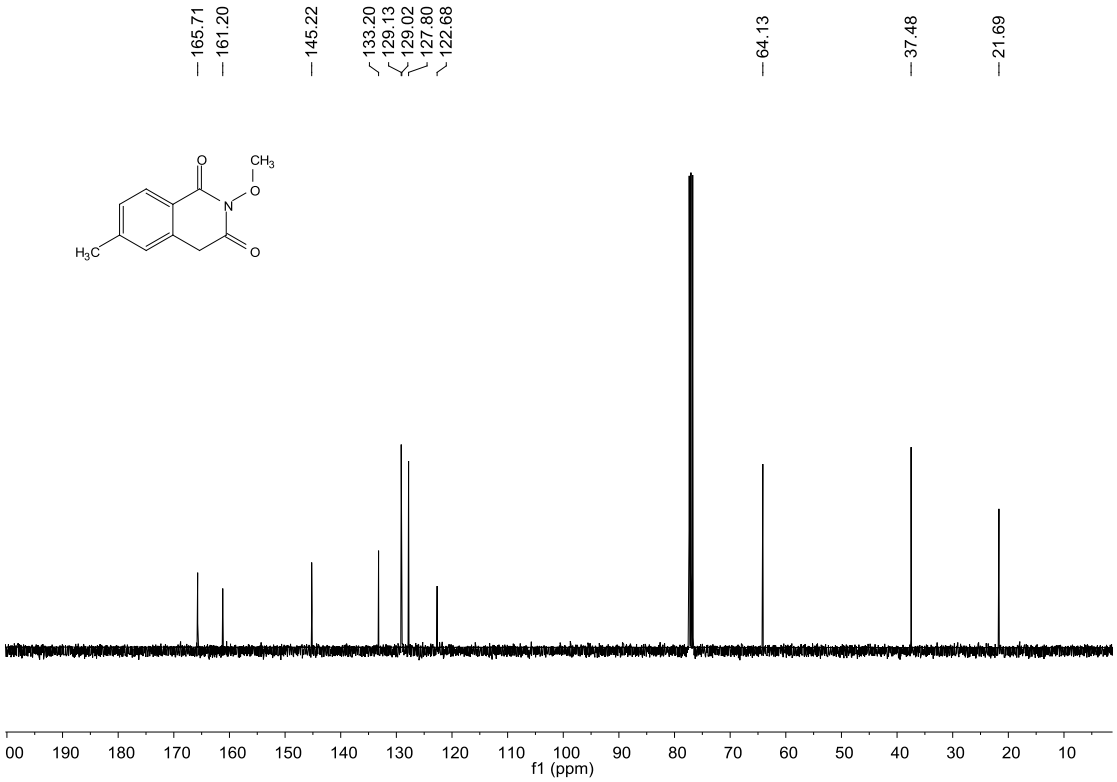
3j


8.10
8.08
7.64
7.62
7.50
4.16
4.01
O
N
O
CH3
Br
O
1.00
1.00
0.97
2.05
3.05
f1 (ppm)

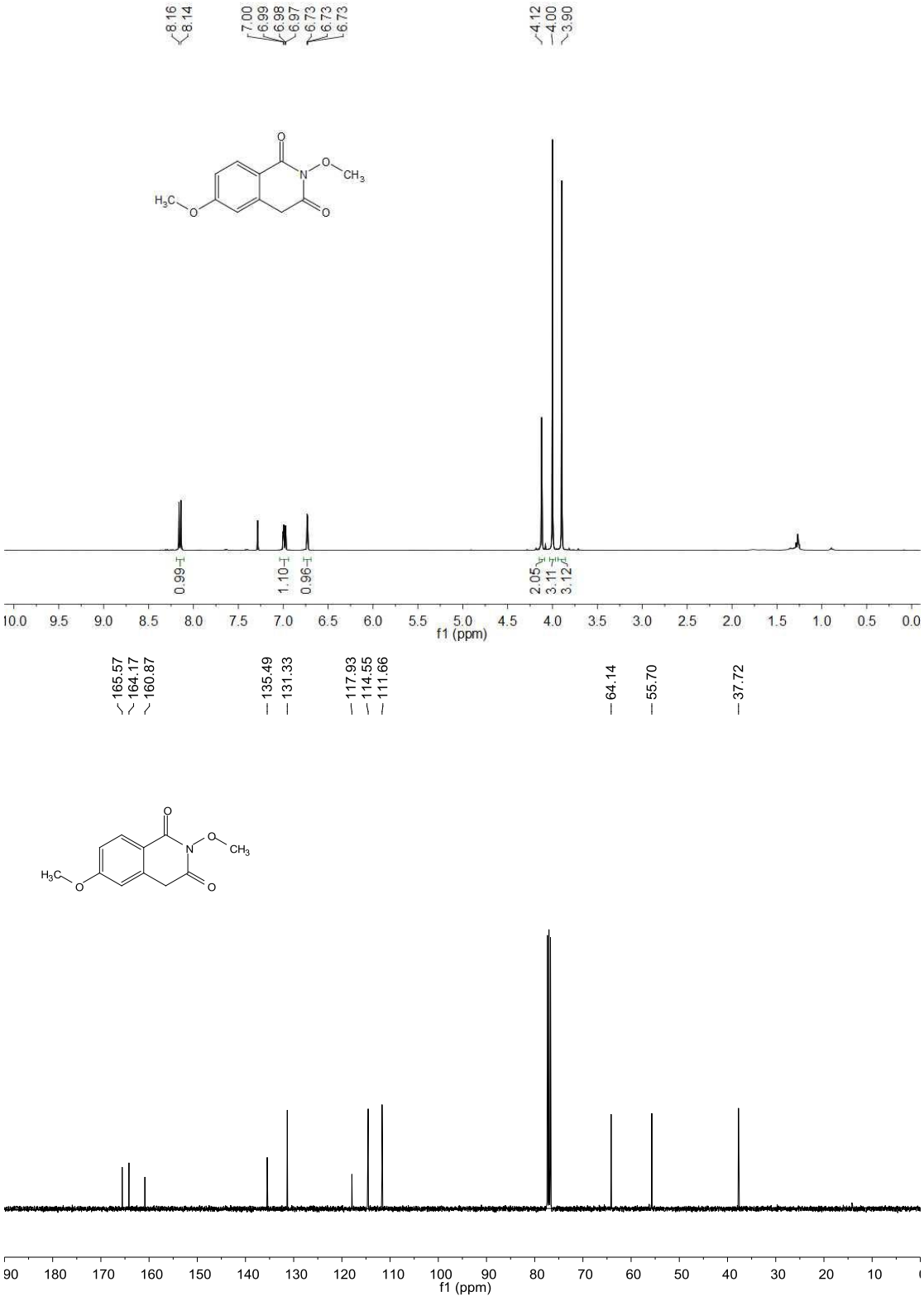

164.75
160.54
134.79
131.61
130.64
130.43
129.36
124.20
64.25
37.13
O
N
O
CH3
Br
O
f1 (ppm)

3k

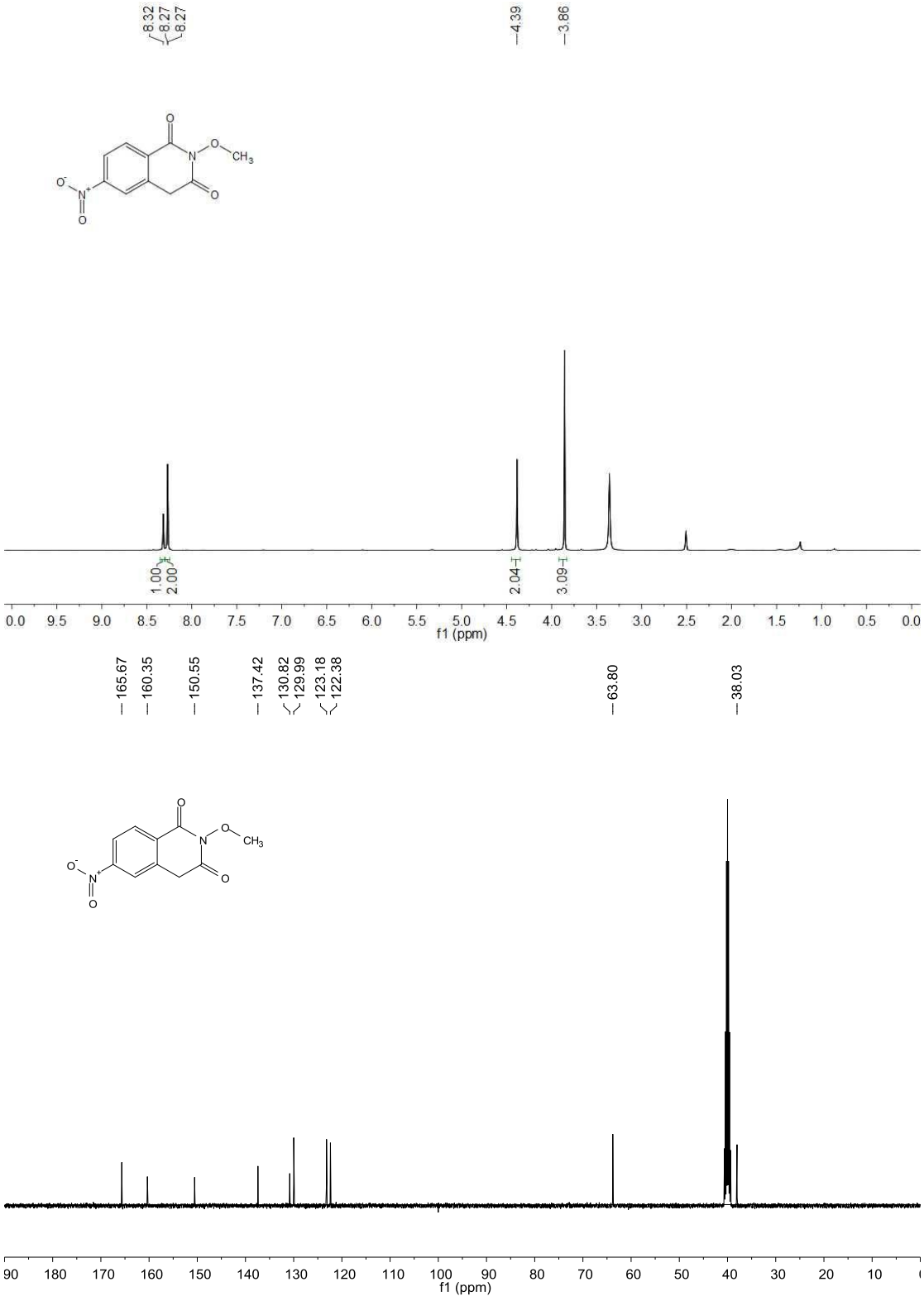

8.08
8.06
7.26
7.24
7.07
4.09
3.97
2.41
1.00
1.16
0.94
2.05
3.06
3.05
f1 (ppm)


165.71
161.20
145.22
133.20
129.13
129.02
127.80
122.68
64.13
37.48
21.69
f1 (ppm)

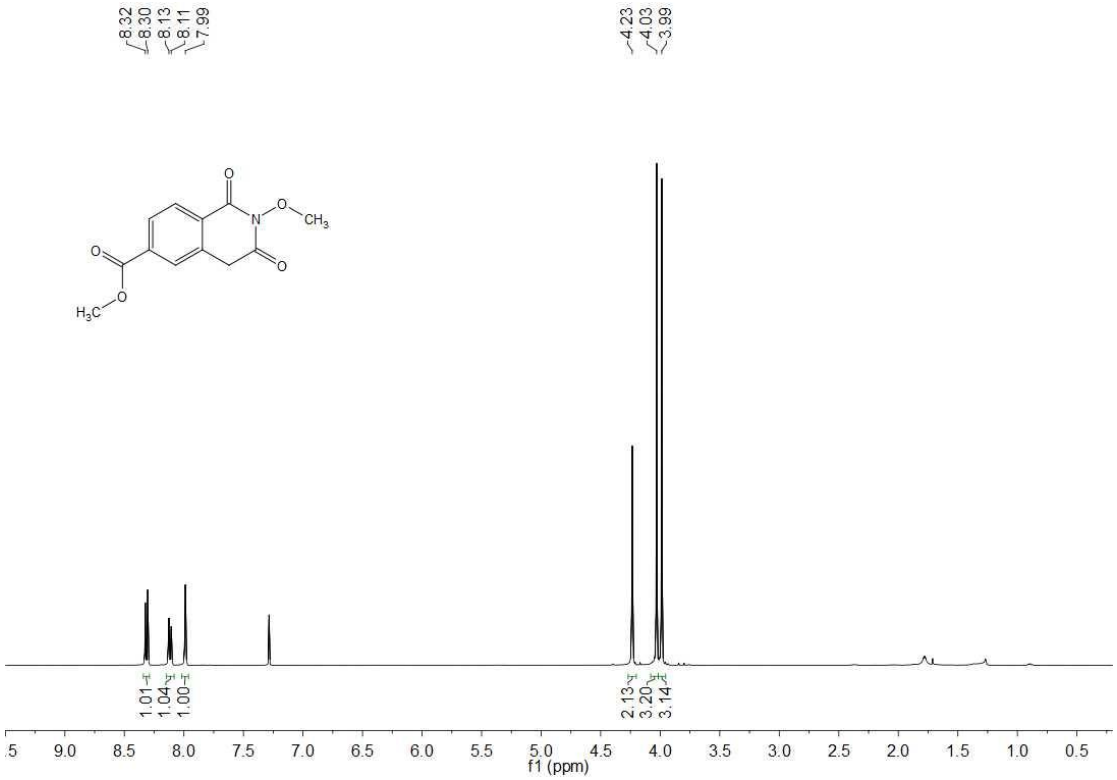
3l

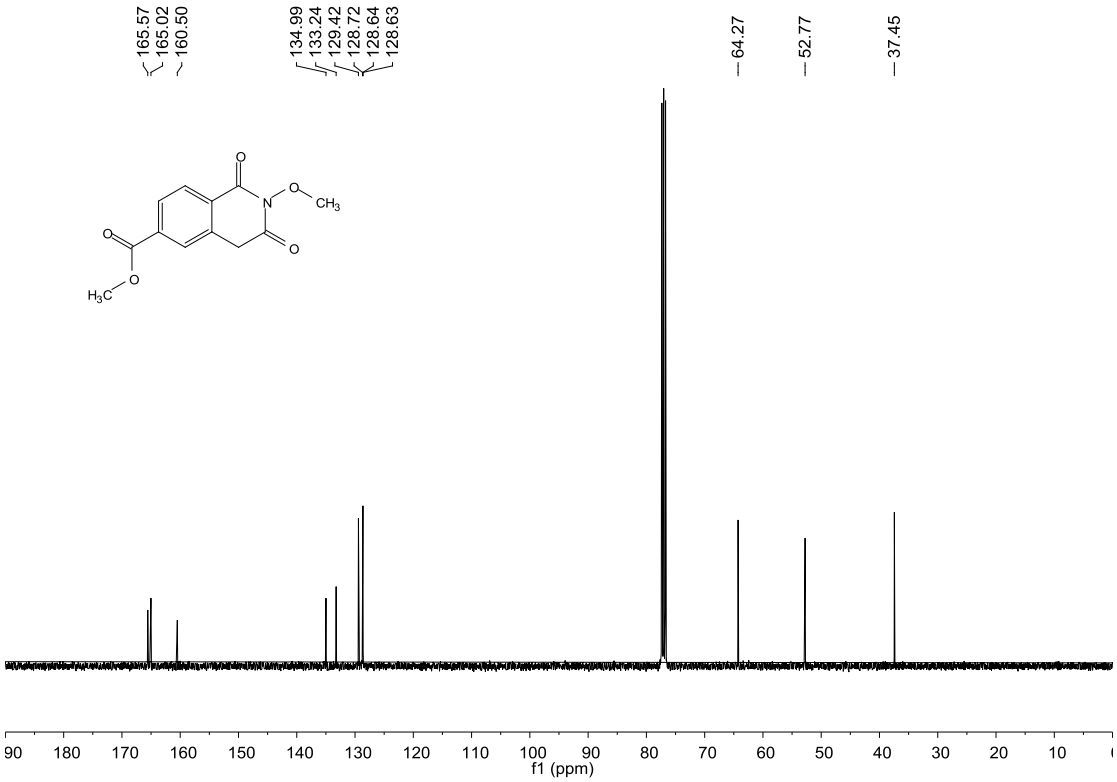

8.16
8.14
7.00
6.99
6.98
6.97
6.73
6.73
6.73
4.12
4.00
3.90
0.99
1.10
0.96
2.05
3.11
3.12
f1 (ppm)
165.57
164.17
160.87
135.49
131.33
117.93
114.55
111.66
64.14
55.70
37.72
f1 (ppm)

3m

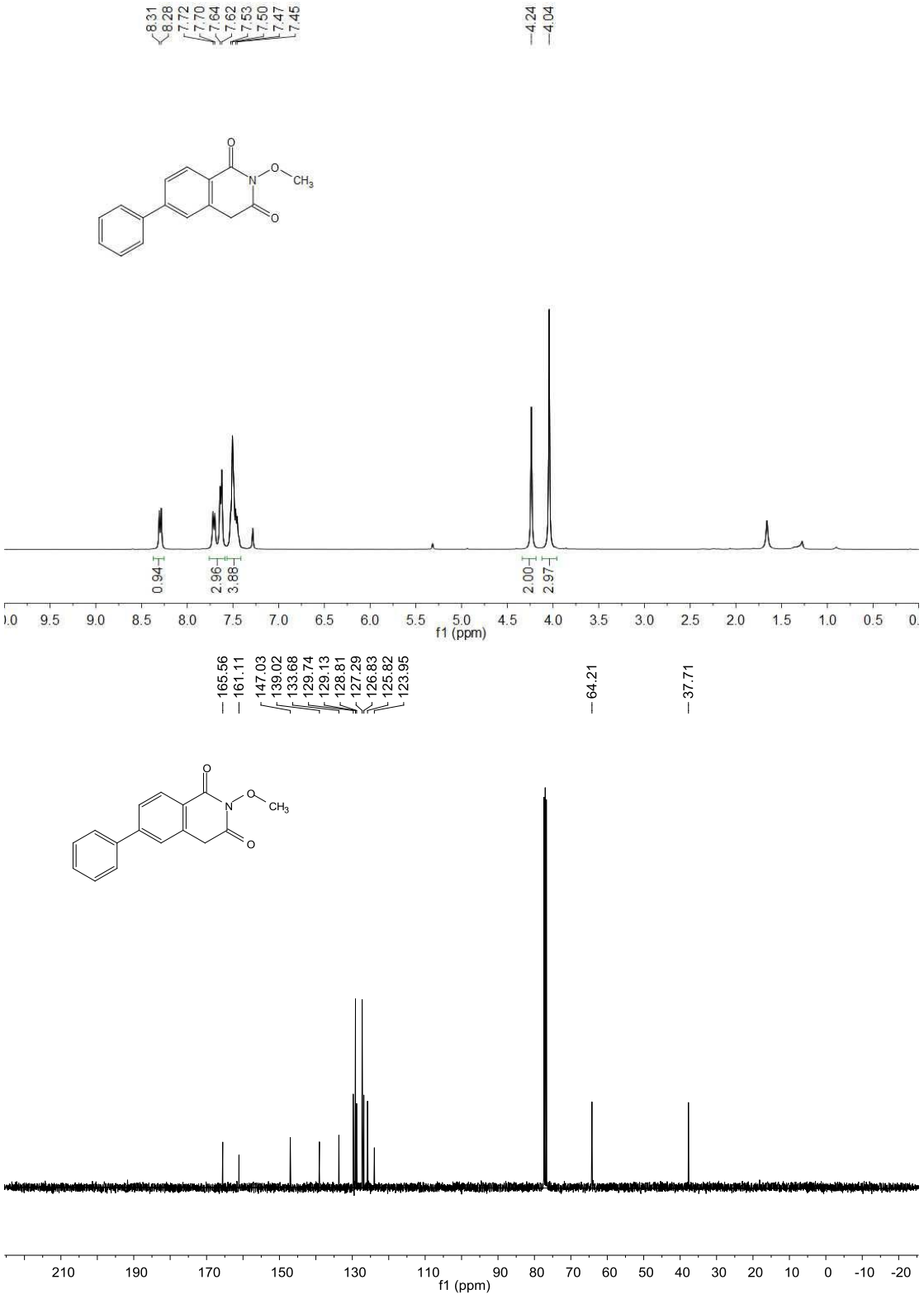

8.32
8.27
8.27
4.39
3.86
1.00
2.00
2.04
3.09
f1 (ppm)
165.67
160.35
150.55
137.42
130.82
129.99
123.18
122.38
63.80
38.03
f1 (ppm)

3n

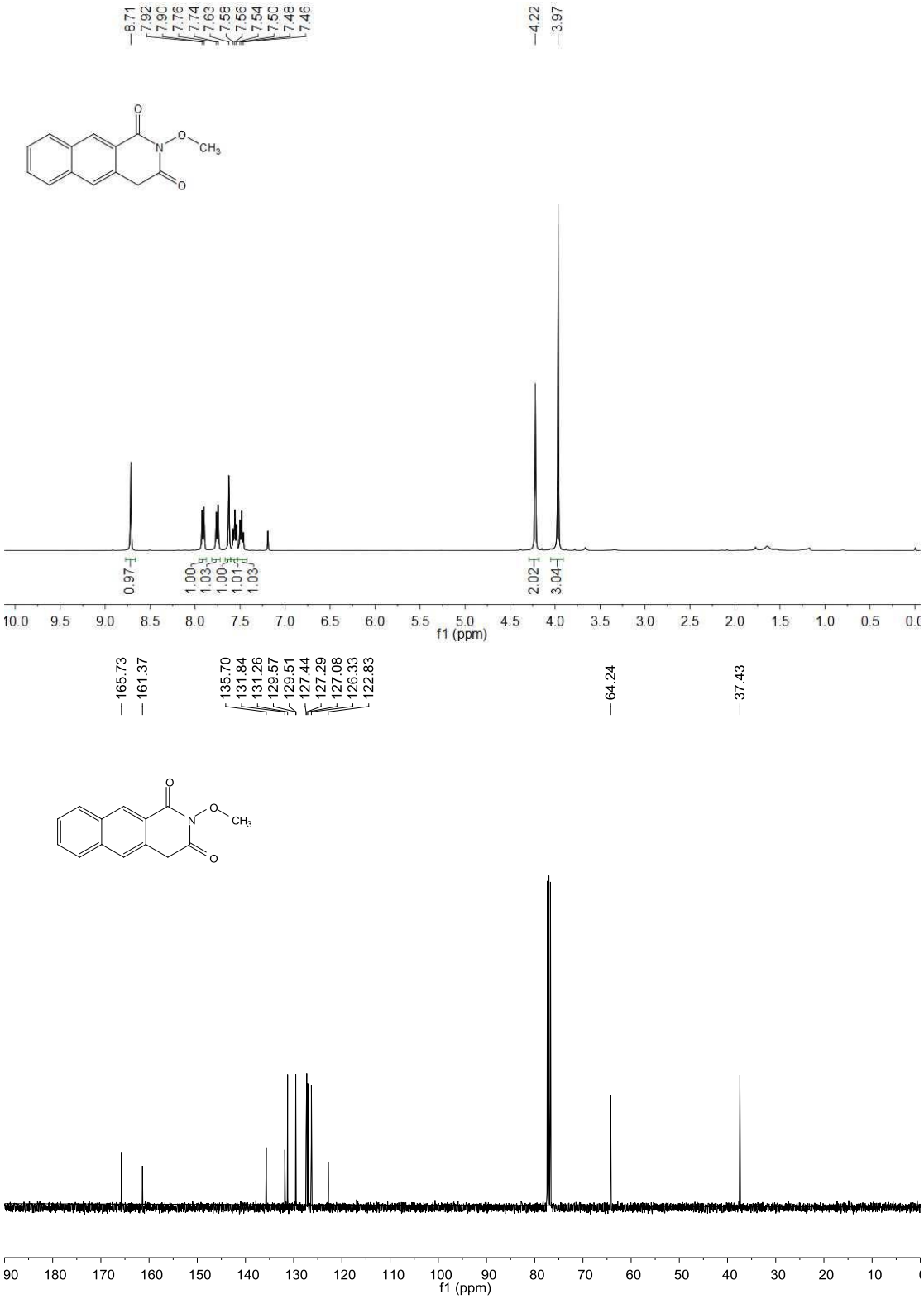

8.32
8.30
8.13
8.11
7.99
4.23
4.03
3.99
1.01
1.04
1.00
2.13
3.20
3.14
f1 (ppm)


165.57
165.02
160.50
134.99
133.24
129.42
128.72
128.64
128.63
64.27
52.77
37.45
f1 (ppm)

3o


8.31
8.28
7.72
7.70
7.64
7.62
7.53
7.50
7.47
7.45
4.24
4.04
O
N
O
CH3
O
0.94
2.96
3.88
2.00
2.97
f1 (ppm)
165.56
161.11
147.03
139.02
133.68
129.74
129.13
128.81
127.29
126.83
125.82
123.95
64.21
37.71
O
N
O
CH3
O
f1 (ppm)

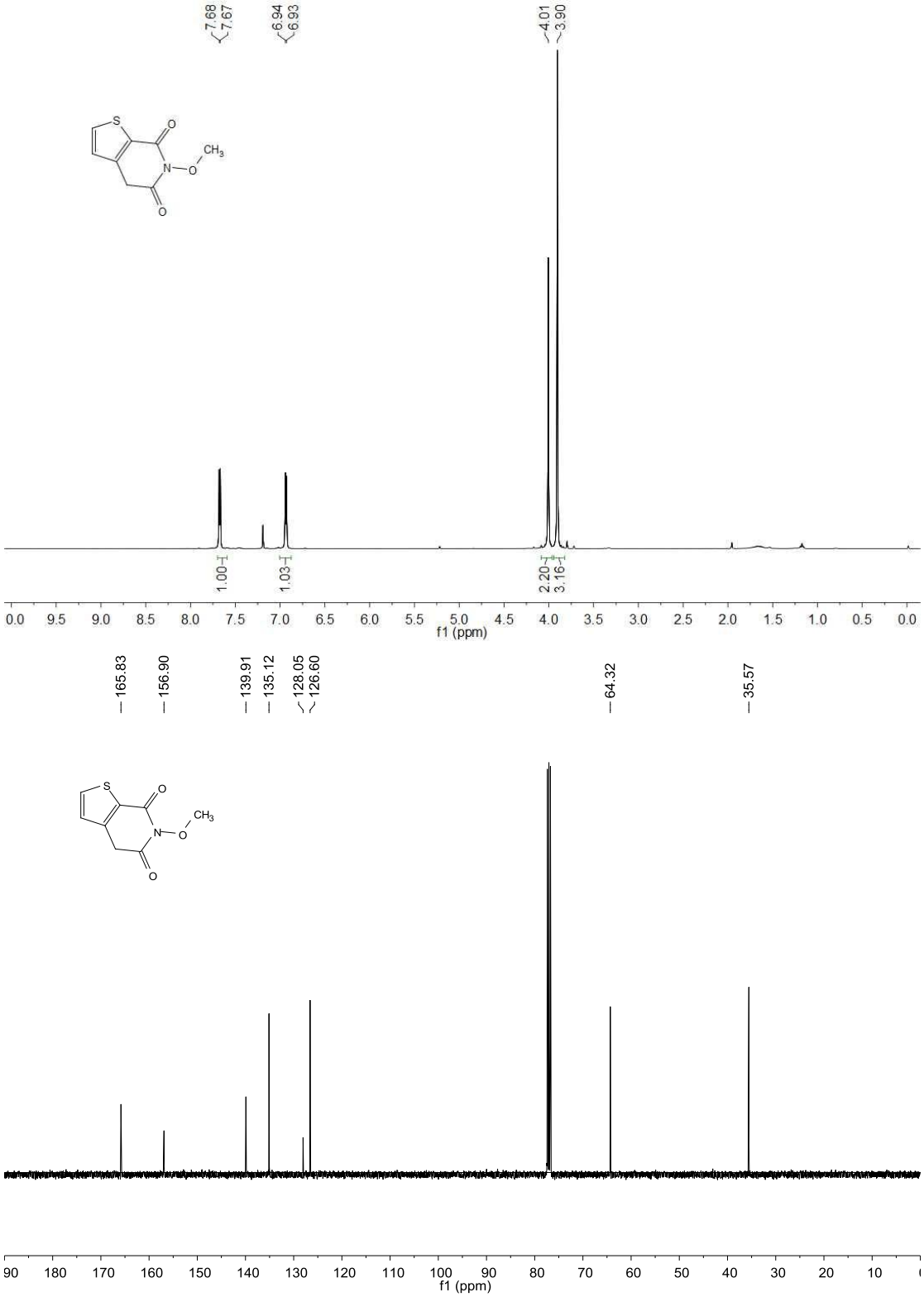
3p


8.71
7.92
7.90
7.76
7.74
7.63
7.58
7.56
7.54
7.50
7.48
7.46
4.22
3.97
O
N
O
CH3
O
0.97
1.00
1.03
1.00
1.01
1.03
2.02
3.04
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)
165.73
161.37
135.70
131.84
131.26
129.57
129.51
127.44
127.29
127.08
126.33
122.83
64.24
37.43
O
N
O
CH3
O
90 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10
f1 (ppm)

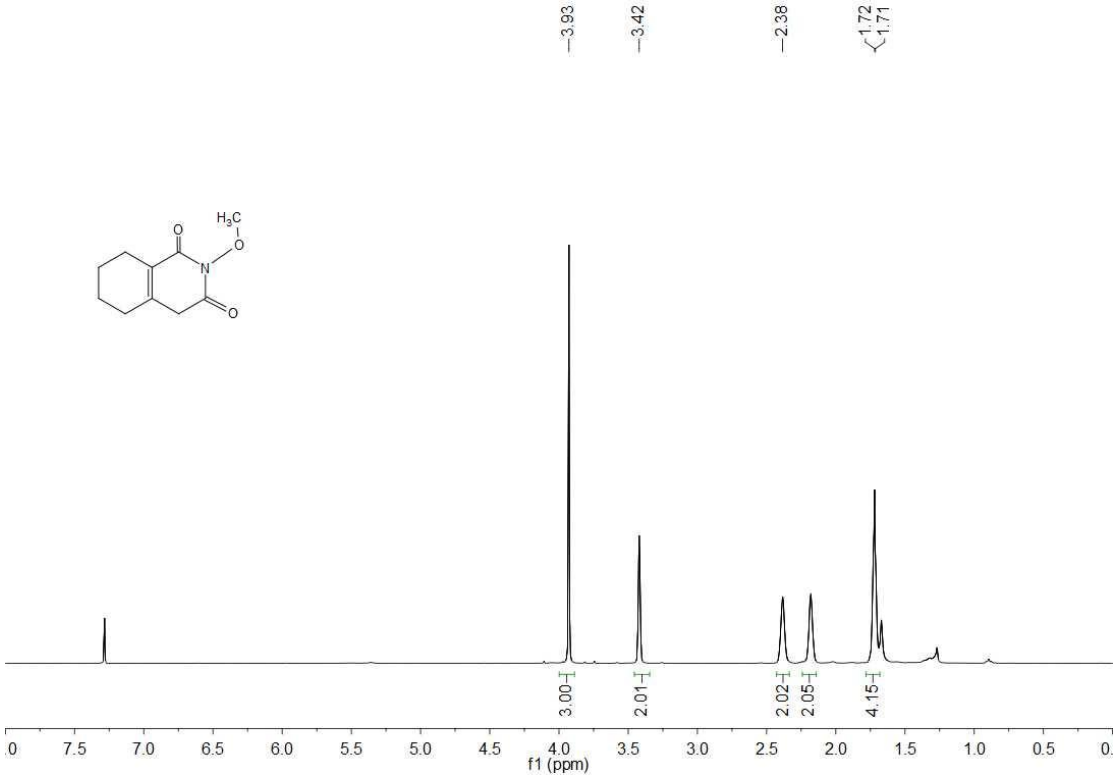
3q

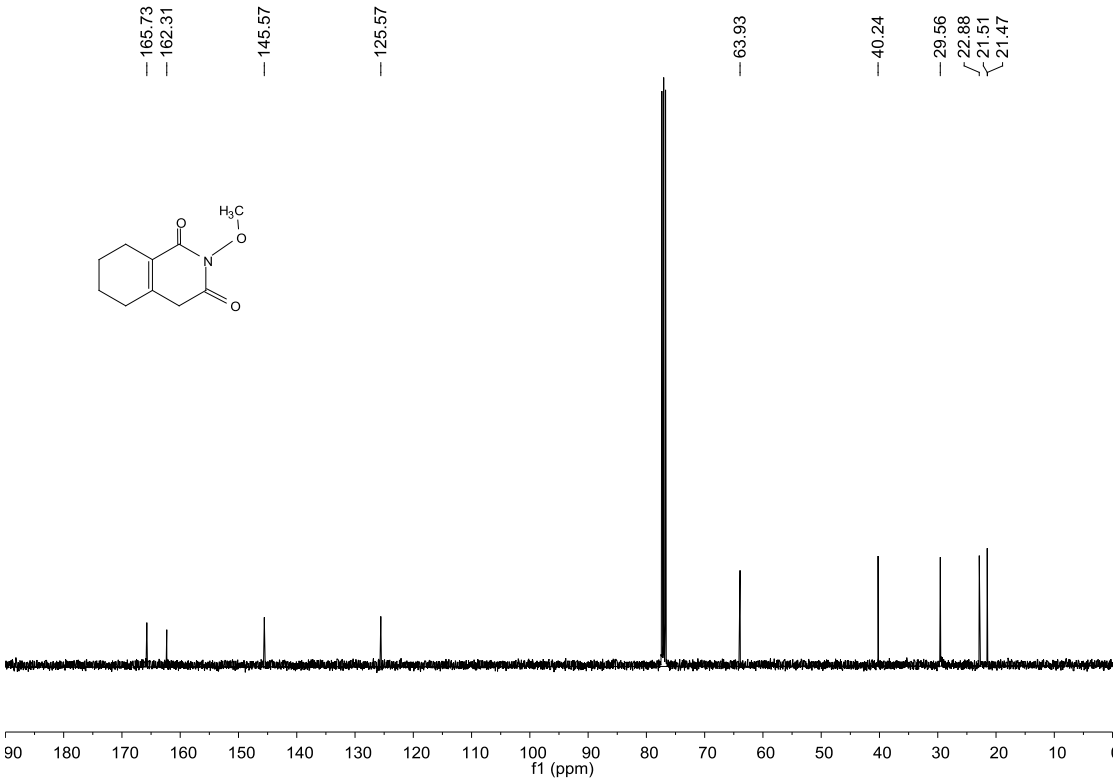

9.69
9.67
8.09
8.07
7.92
7.90
7.78
7.76
7.76
7.74
7.74
7.65
7.63
7.61
7.33
7.30
7.28
4.29
4.08
CH3
O
N
O
O
1.00
1.04
1.07
1.07
1.10
1.05
2.09
3.16
f1 (ppm)
164.91
161.96
135.77
135.47
133.00
131.66
129.66
128.81
126.97
126.07
124.47
119.20
64.15
38.55
f1 (ppm)

3r

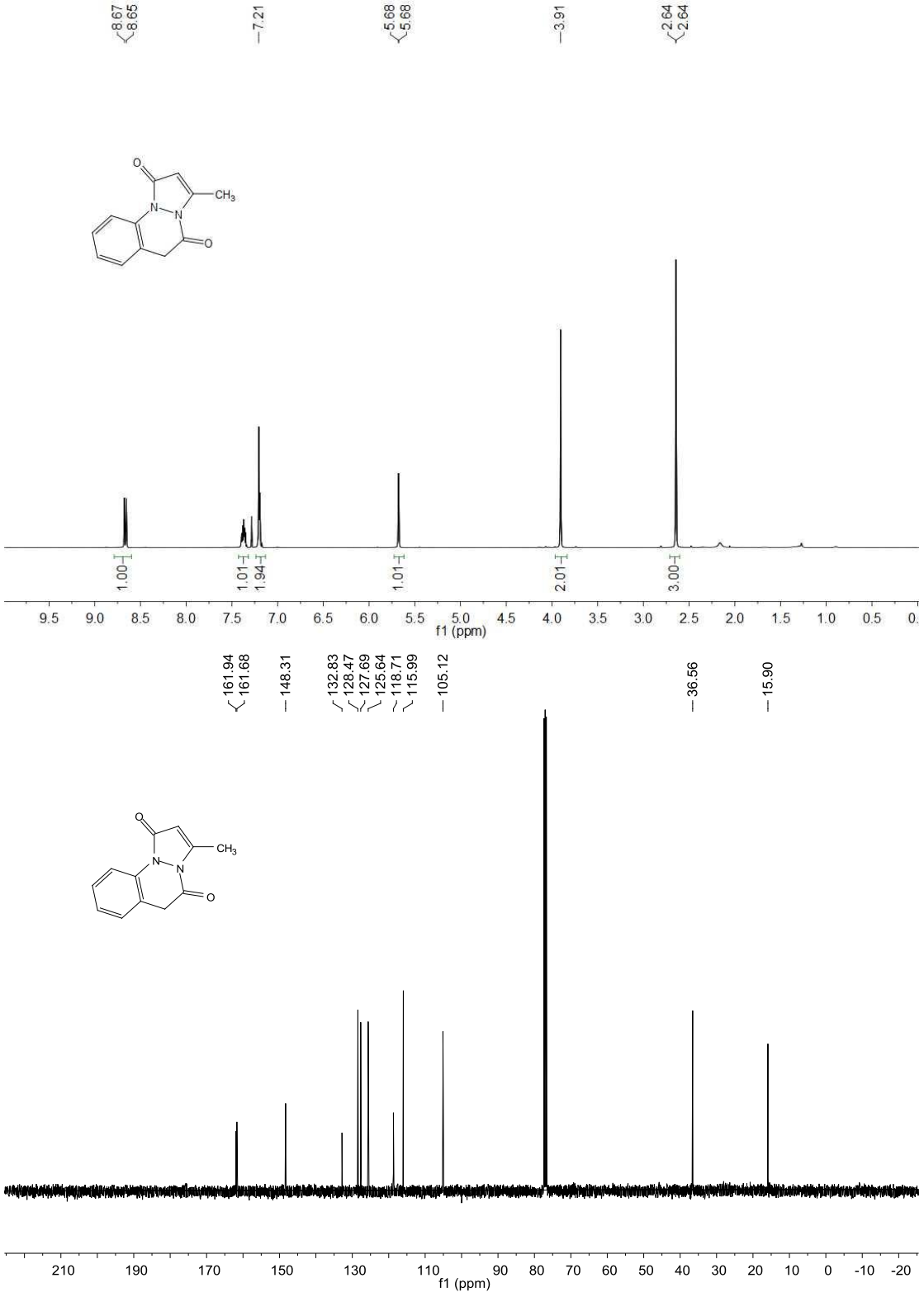

7.68
7.67
6.94
6.93
4.01
3.90
1.00
1.03
2.20
3.16
f1 (ppm)
165.83
156.90
139.91
135.12
128.05
126.60
64.32
35.57
f1 (ppm)

3s

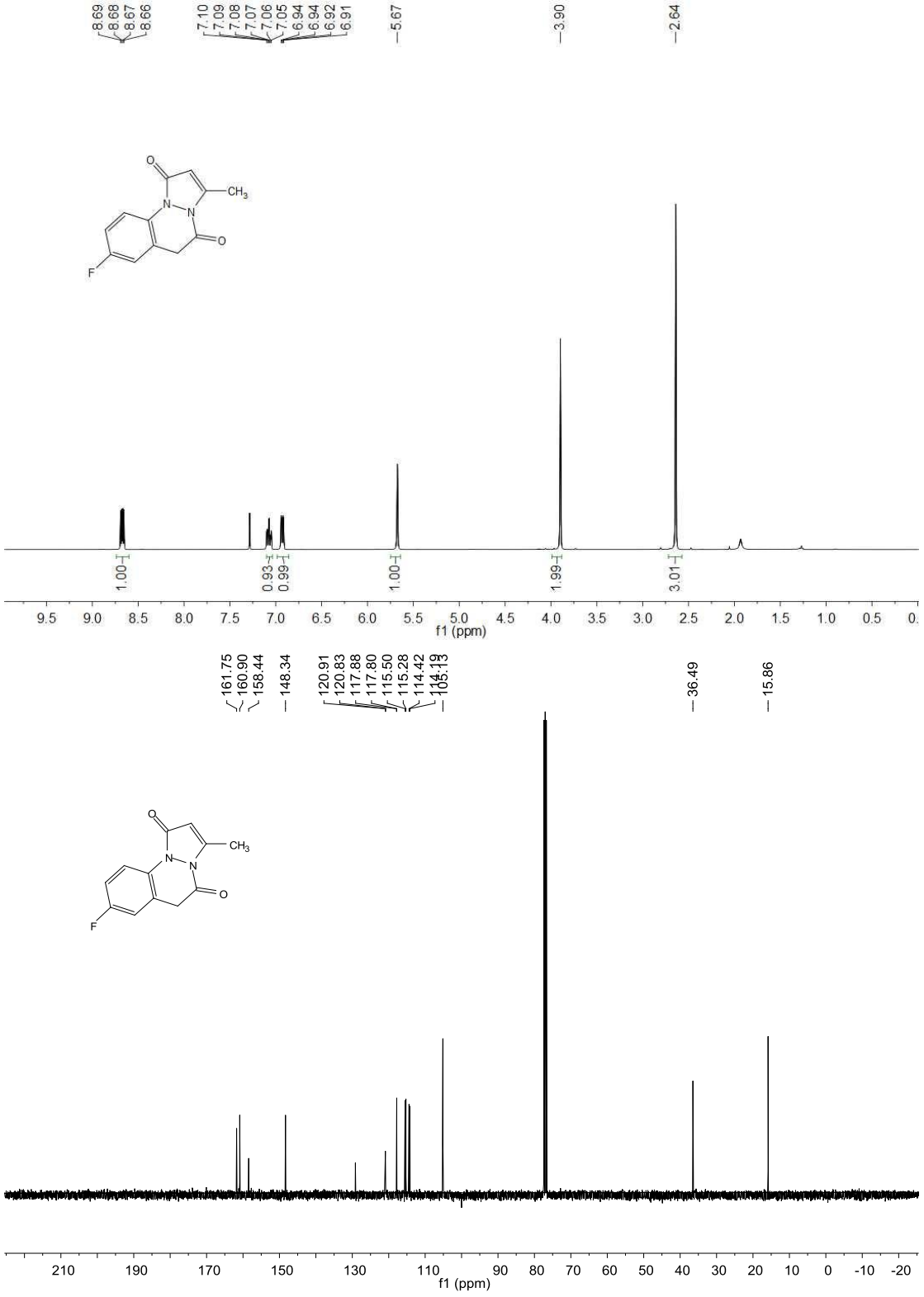

3.93
3.42
2.38
1.72
1.71
3.00
2.01
2.02
2.05
4.15
f1 (ppm)


165.73
162.31
145.57
125.57
63.93
40.24
29.56
22.88
21.51
21.47
f1 (ppm)

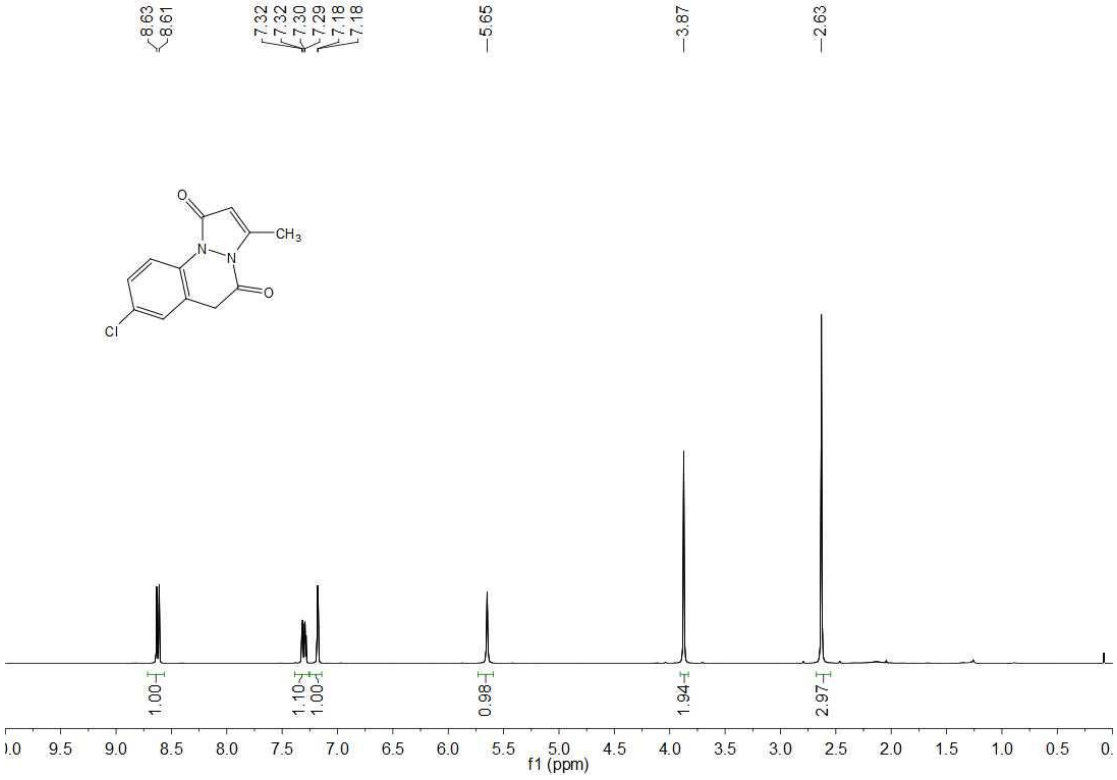
5a

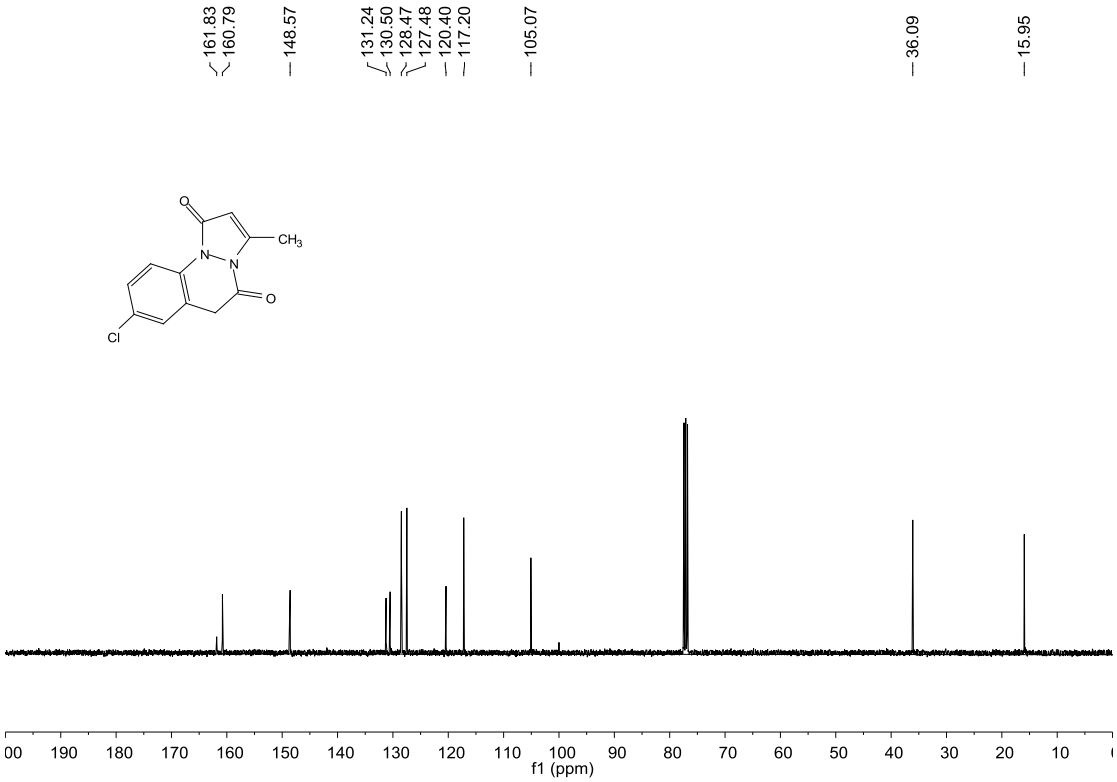

8.67
8.65
7.21
5.68
5.68
3.91
2.64
2.64
CH3
1.00
1.01
1.94
1.01
2.01
3.00
f1 (ppm)
161.94
161.68
148.31
132.83
128.47
127.69
125.64
118.71
115.99
105.12
36.56
15.90
CH3
f1 (ppm)

5b

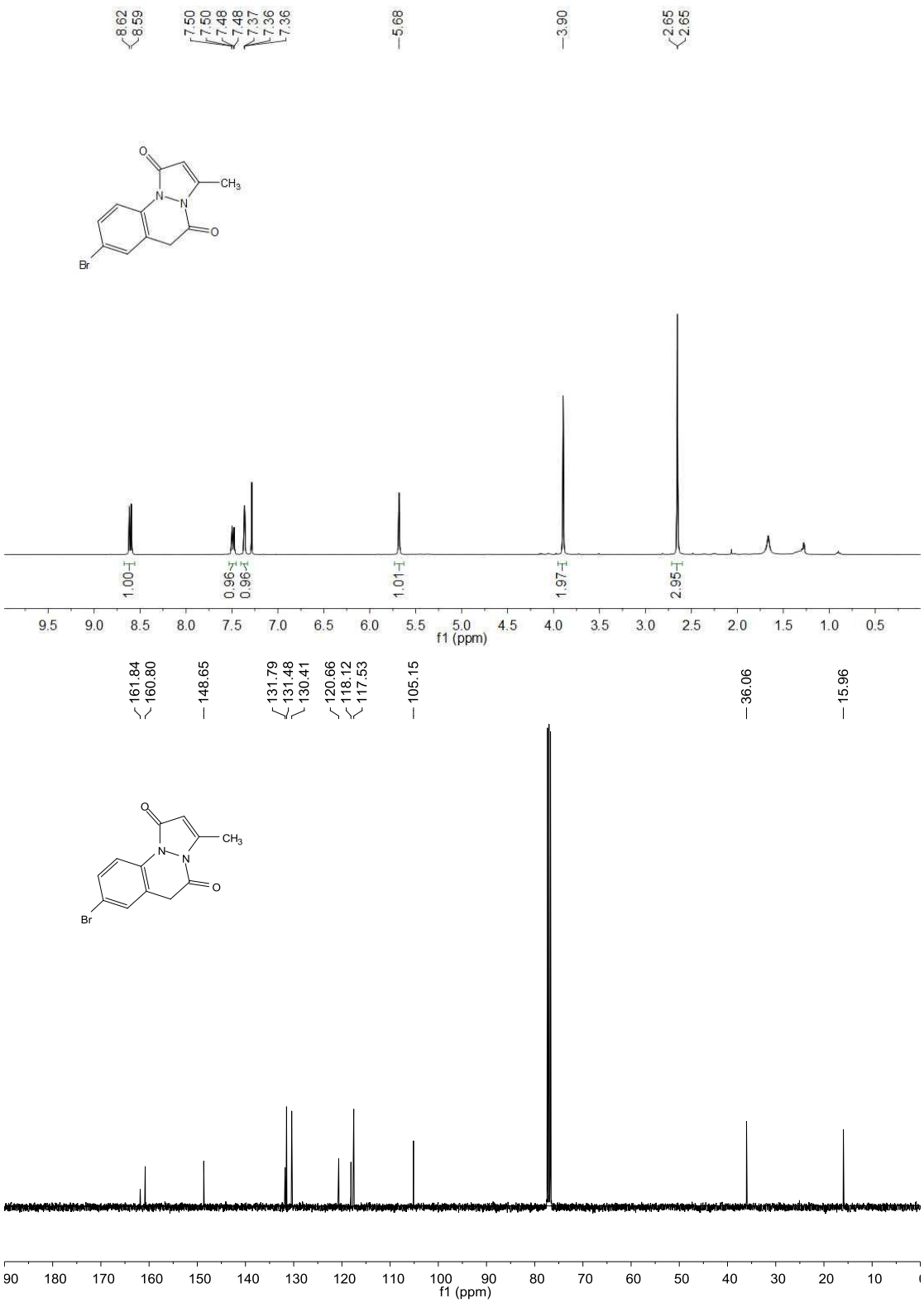

8.69
8.68
8.67
8.66
7.10
7.09
7.08
7.07
7.06
7.05
6.94
6.92
6.91
5.67
3.90
2.64
CH3
F
1.00
0.93
0.99
1.00
1.99
3.01
f1 (ppm)
161.75
160.90
158.44
148.34
120.91
120.83
117.88
117.80
115.50
115.28
114.42
36.49
15.86
f1 (ppm)

5c

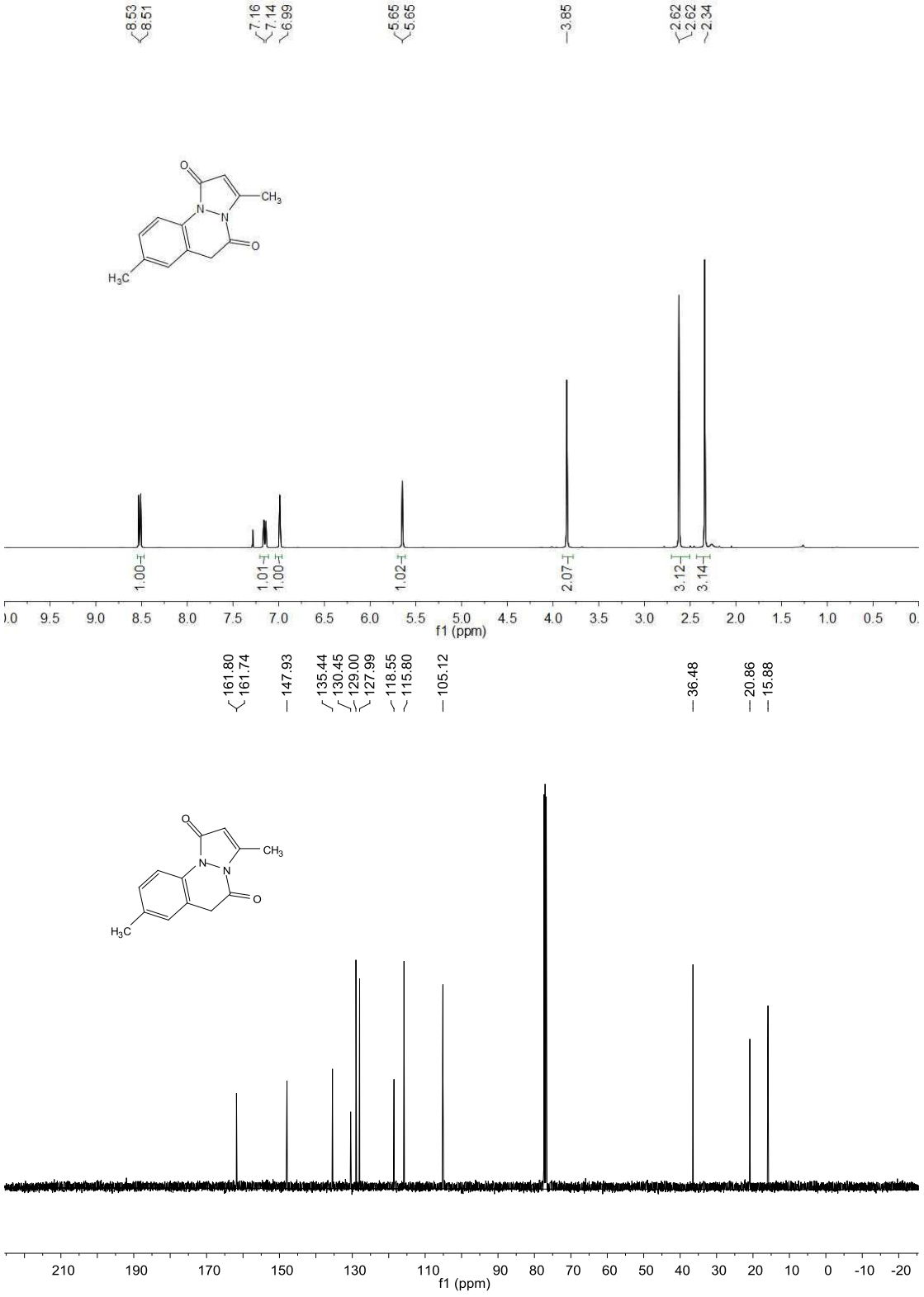

8.63
8.61
7.32
7.32
7.30
7.29
7.18
7.18
5.65
3.87
2.63
O
N
N
CH3
O
Cl
1.00
1.10
1.00
0.98
1.94
2.97
f1 (ppm)


161.83
160.79
148.57
131.24
130.50
128.47
127.48
120.40
117.20
105.07
36.09
15.95
O
N
N
CH3
O
Cl
f1 (ppm)

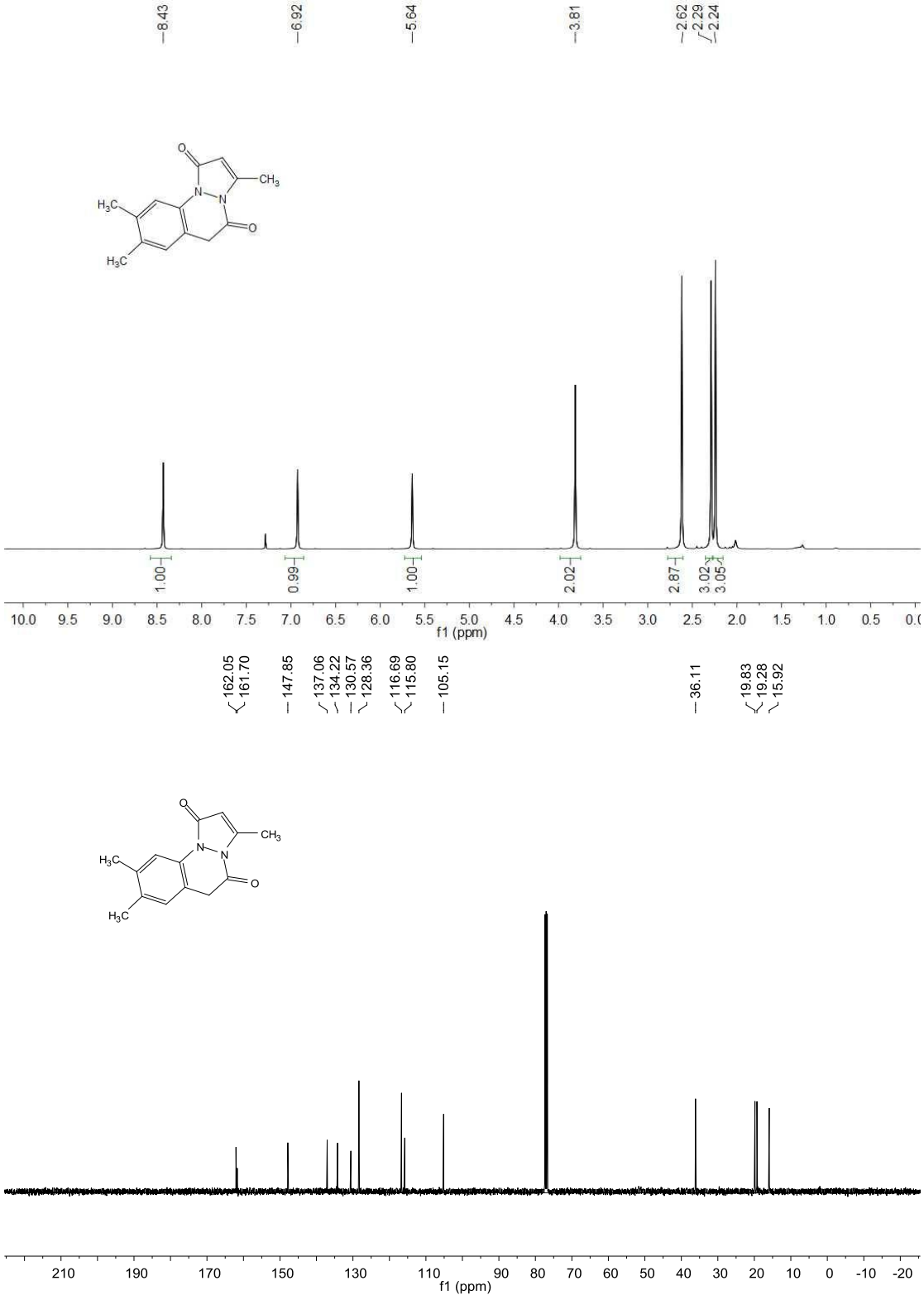
5d


8.62
8.59
7.50
7.50
7.48
7.48
7.37
7.36
7.36
5.68
3.90
2.65
2.65
O
N
N
CH3
O
Br
1.00
0.96
0.96
1.01
1.97
2.95
f1 (ppm)
161.84
160.80
148.65
131.79
131.48
130.41
120.66
118.12
117.53
105.15
36.06
15.96
f1 (ppm)

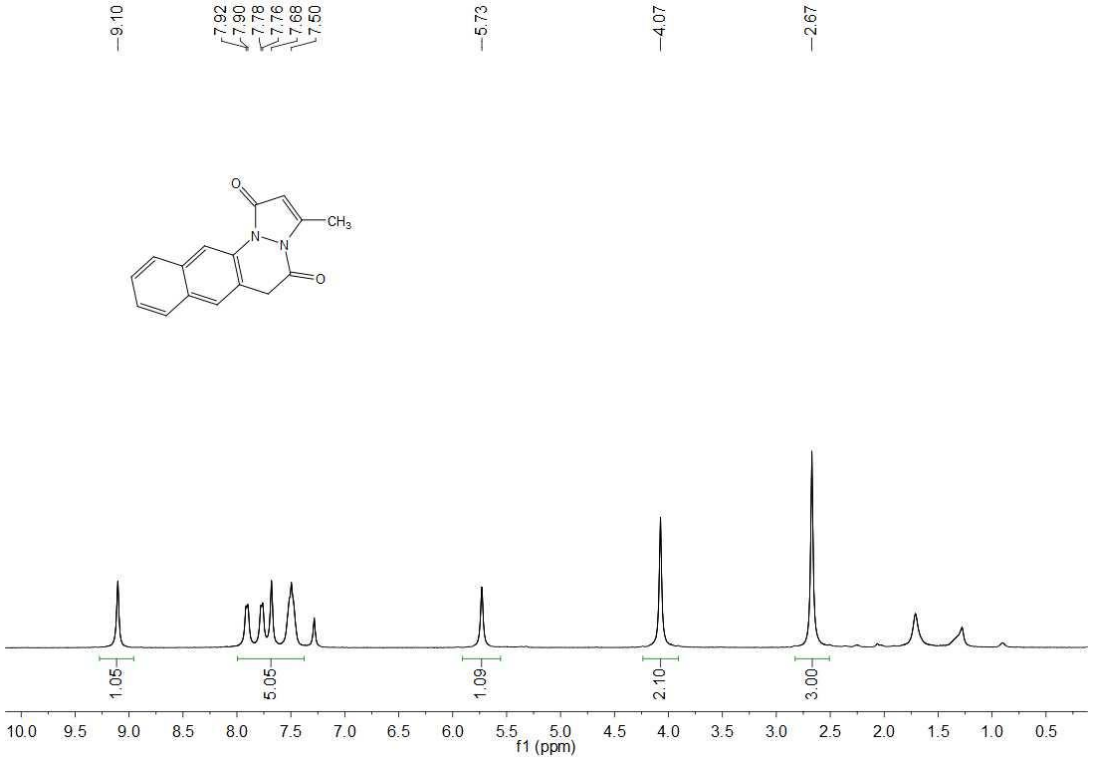
5e

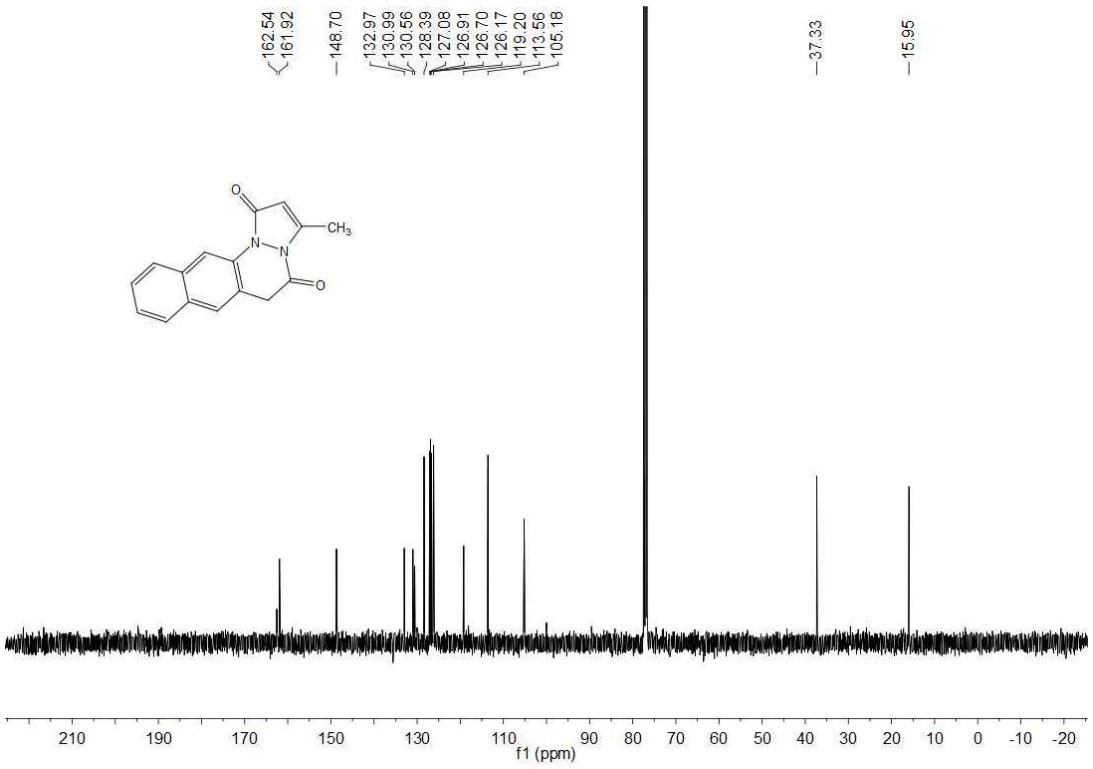

8.53
8.51
7.16
7.14
6.99
5.65
5.65
3.85
2.62
2.62
2.34
1.00
1.01
1.00
1.02
2.07
3.12
3.14
f1 (ppm)
161.80
161.74
147.93
135.44
130.45
129.00
127.99
118.55
115.80
105.12
36.48
20.86
15.88
f1 (ppm)

5f

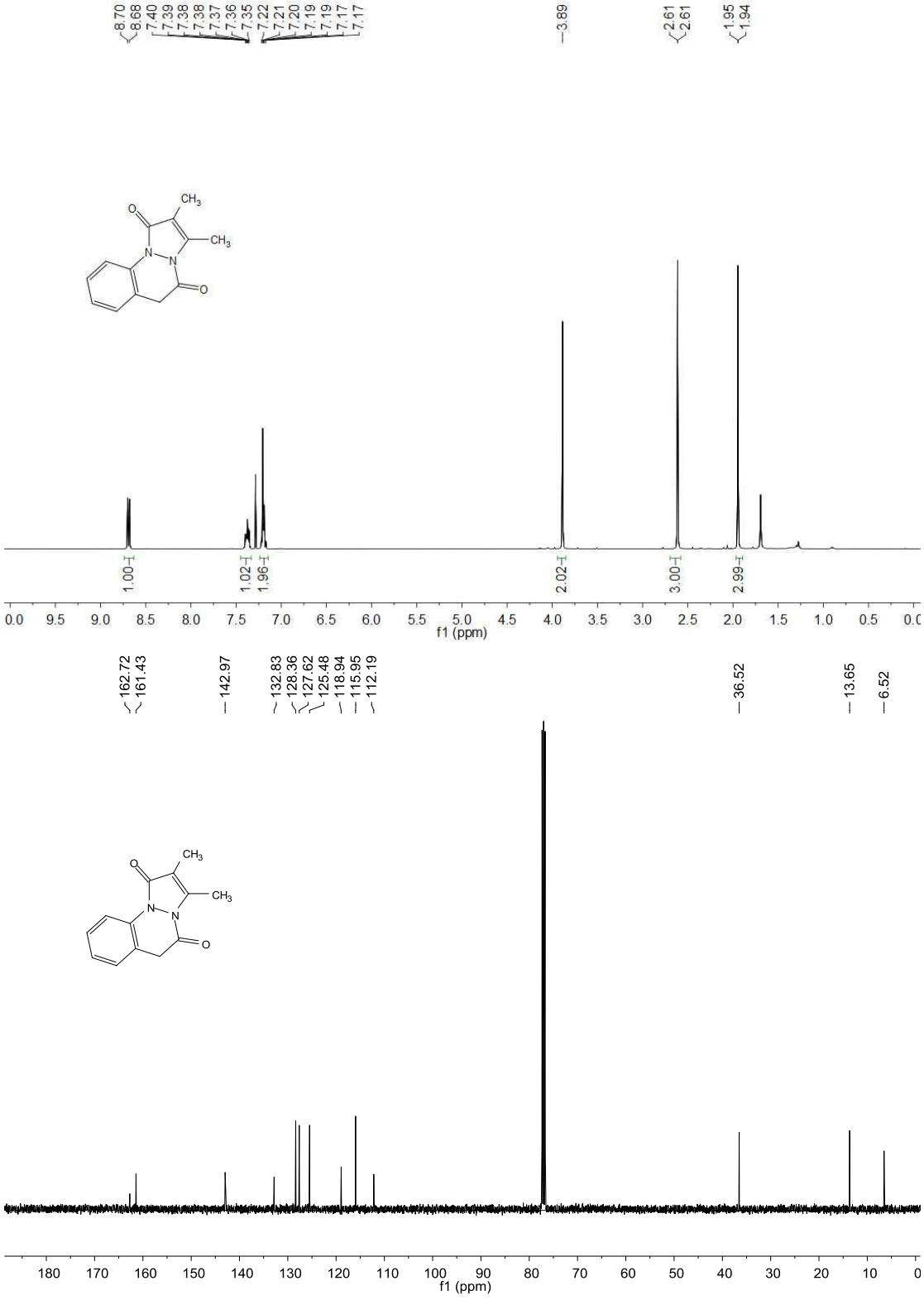

8.43
6.92
5.64
3.81
2.62
2.29
2.24
1.00
0.99
1.00
2.02
2.87
3.02
3.05
f1 (ppm)
162.05
161.70
147.85
137.06
134.22
130.57
128.36
116.69
115.80
105.15
36.11
19.83
19.28
15.92
f1 (ppm)

5g

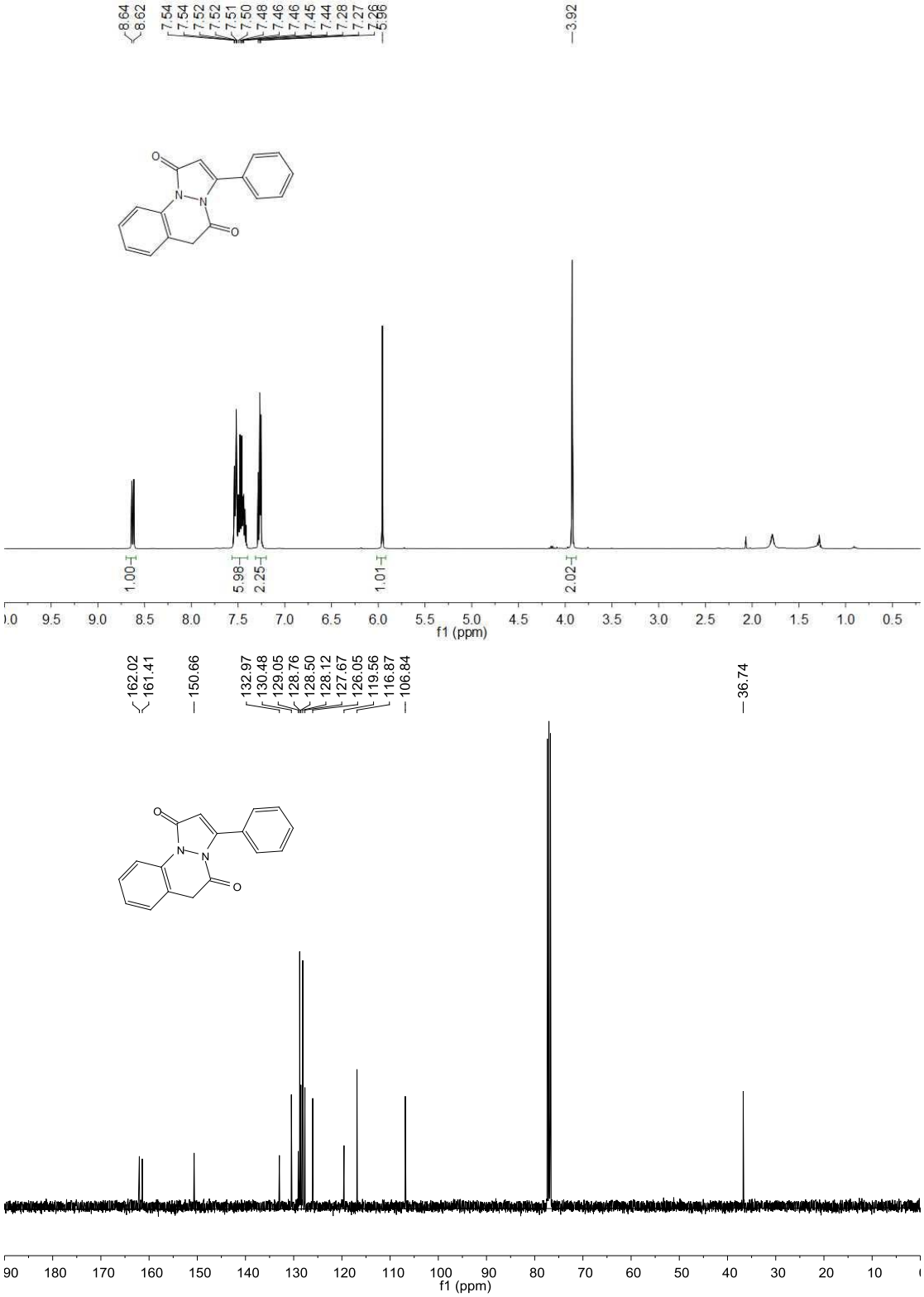

9.10
7.92
7.90
7.78
7.76
7.68
7.50
5.73
4.07
2.67
O
N
N
CH3
O
1.05
5.05
1.09
2.10
3.00
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5
f1 (ppm)


162.54
161.92
148.70
132.97
130.99
130.56
128.39
127.08
126.91
126.70
126.17
119.20
113.56
105.18
37.33
15.95
O
N
N
CH3
O
210 190 170 150 130 110 90 80 70 60 50 40 30 20 10 0 -10 -20
f1 (ppm)

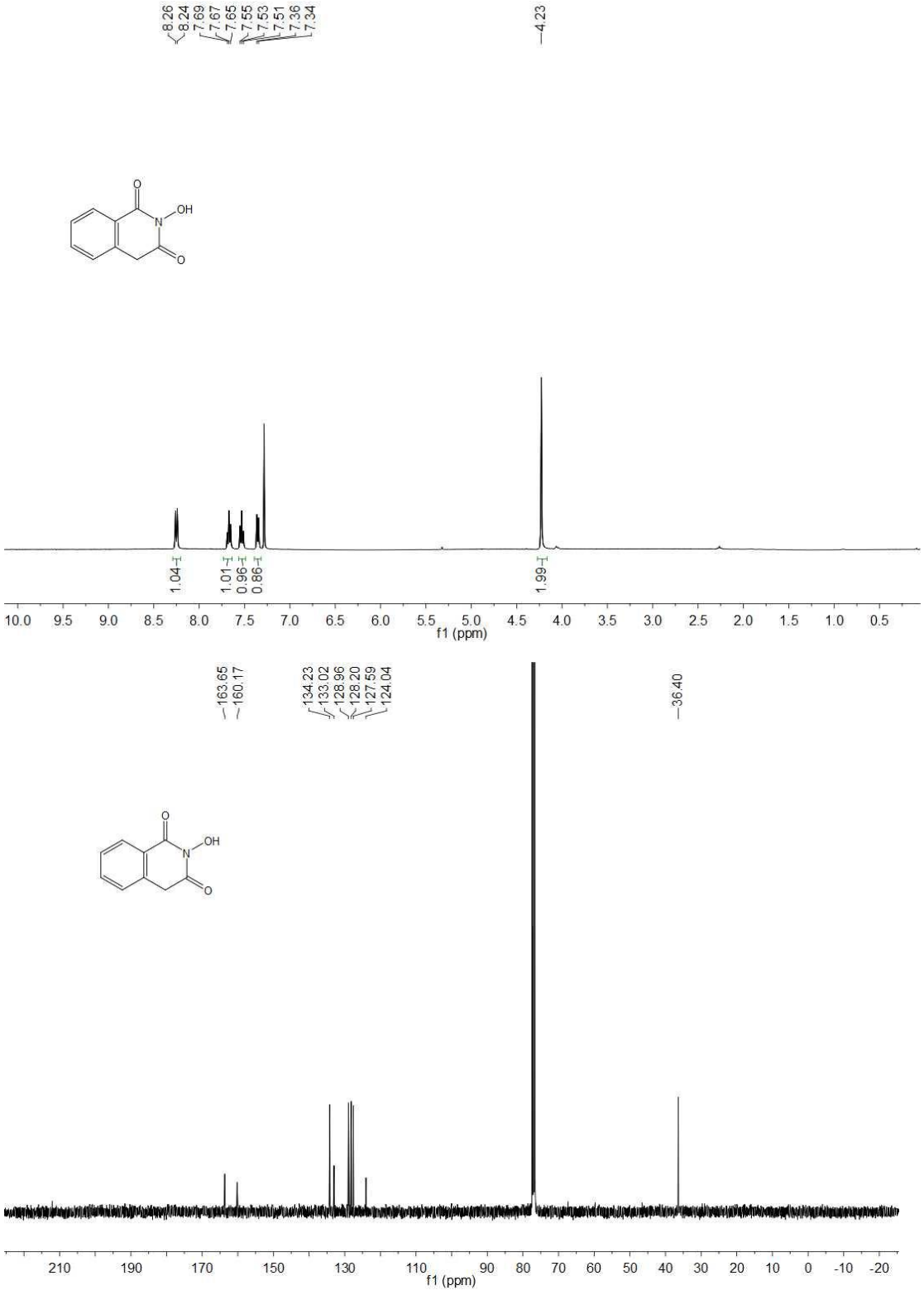
5h


8.70
8.68
7.40
7.39
7.38
7.38
7.37
7.36
7.35
7.22
7.21
7.20
7.19
7.19
7.17
7.17
3.89
2.61
2.61
1.95
1.94
CH3
CH3
O
N
N
O
1.00
1.02
1.96
2.02
3.00
2.99
f1 (ppm)
162.72
161.43
142.97
132.83
128.36
127.62
125.48
118.94
115.95
112.19
36.52
13.65
6.52
f1 (ppm)

5i


8.64
8.62
7.54
7.54
7.52
7.52
7.51
7.50
7.48
7.46
7.46
7.45
7.44
7.28
7.27
5.96
3.92
1.00
5.98
2.25
1.01
2.02
f1 (ppm)
162.02
161.41
150.66
132.97
130.48
129.05
128.76
128.50
128.12
127.67
126.05
119.56
116.87
106.84
36.74
f1 (ppm)

6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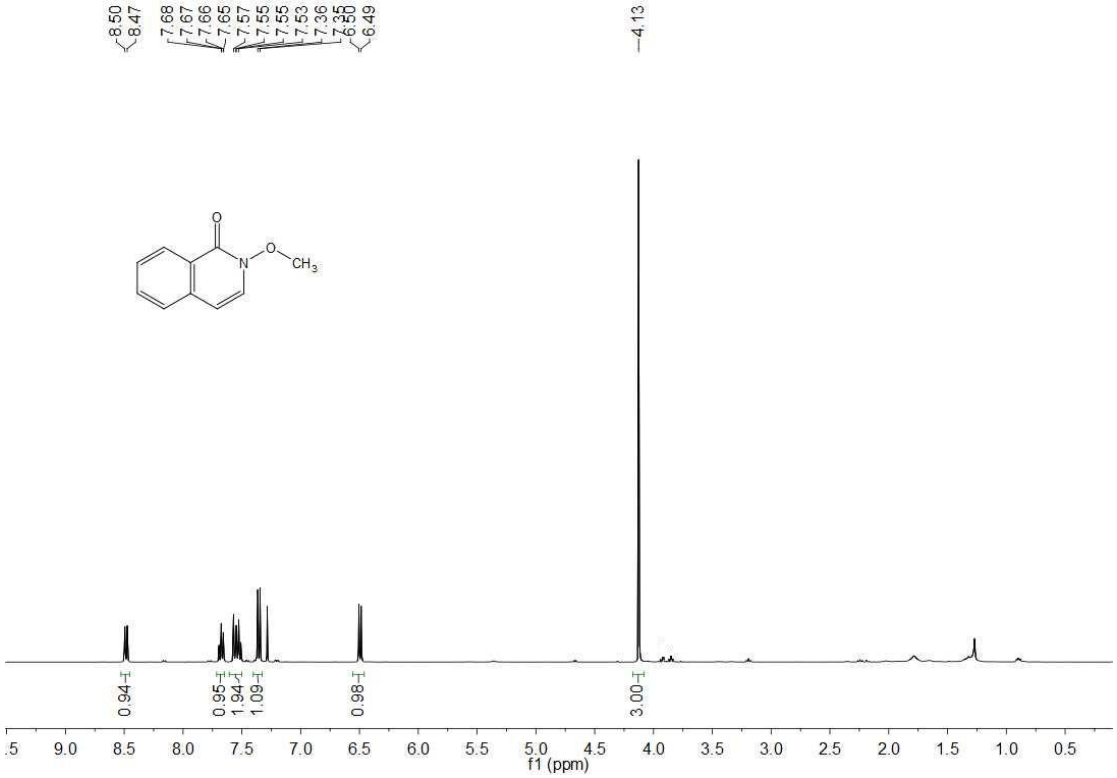
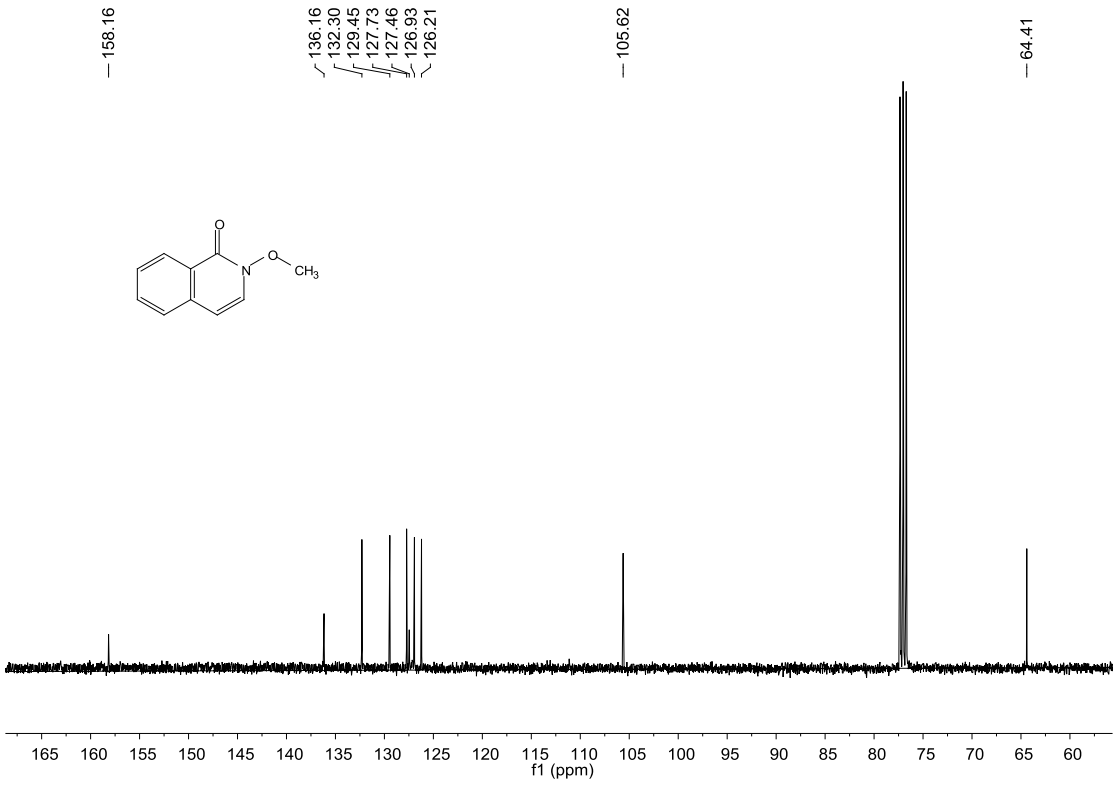
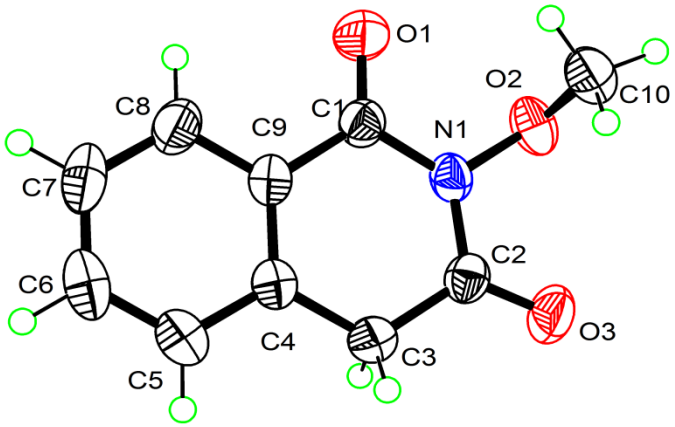
8.24
7.69
7.67
7.65
7.55
7.53
7.51
7.36
7.34
4.23
OH
1.04
1.01
0.96
0.86
1.99
f1 (ppm)
163.65
160.17
134.23
133.02
128.96
128.20
127.59
124.04
36.40
OH
f1 (ppm)

7


8.50
8.47
7.68
7.67
7.66
7.65
7.57
7.55
7.55
7.53
7.36
7.35
6.50
6.49
4.13
O
N
O
CH3
0.94
0.95
1.94
1.09
0.98
3.00
f1 (ppm)


158.16
136.16
132.30
129.45
127.73
127.46
126.93
126.21
105.62
64.41
O
N
O
CH3
f1 (ppm)

Crystal data and structure refinement for 3a:



Data collection: Bruker *APEX2*; cell refinement: Bruker *SAINT*; data reduction: Bruker *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: Bruker *SHELXTL*; software used to prepare material for publication: Bruker *SHELXTL*.

Crystal data

$C_{10}H_9NO_3$	$F(000)$ = 400
M_r = 191.18	D_x = 1.439 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, λ = 0.71073 Å
a = 9.3511 (2) Å	Cell parameters from 6169 reflections
b = 11.4869 (3) Å	θ = 4.5–54.9 °
c = 8.4156 (2) Å	μ = 0.11 mm^{-1}
β = 102.475 (2) °	T = 296 K
V = 882.62 (4) Å^3	Plate, whiteless
Z = 4	0.3 × 0.2 × 0.20 mm

Data collection

Bruker APEX-II CCD diffractometer	2038 independent reflections
Radiation source: fine-focus sealed tube	1718 reflections with $I > 2\sigma(I)$

graphite	R_{int} = 0.022
ϕ and ω scans	θ_{max} = 27.6 °, θ_{min} = 2.2 °
Absorption correction: multi-scan *SADABS*	h = -12→12
T_{min} = 0.653, T_{max} = 0.746	k = -14→14
7674 measured reflections	l = -10→10

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.042	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.149	H atoms treated by a mixture of independent and constrained refinement
S = 1.11	$w = 1/[\sigma^2(F_o^2) + (0.0835P)^2 + 0.1638P]$ where $P = (F_o^2 + 2F_c^2)/3$
2038 reflections	$(\Delta/\sigma)_{max} < 0.001$
128 parameters	$\Delta\rangle_{max}$ = 0.25 e Å^{-3}
0 restraints	$\Delta\rangle_{min}$ = -0.24 e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for

calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}
O2	0.01366 (10)	0.79659 (10)	-0.01900 (13)	0.0451 (3)
O1	0.26774 (13)	0.72907 (12)	-0.07358 (16)	0.0615 (4)
O3	0.01246 (11)	0.95556 (10)	0.20481 (16)	0.0524 (3)
N1	0.14310 (11)	0.85002 (10)	0.05931 (15)	0.0355 (3)
C6	0.66959 (16)	0.96408 (16)	0.2628 (2)	0.0543 (5)
H6	0.7576	0.9974	0.3159	0.065*
C5	0.53942 (17)	1.00639 (14)	0.2912 (2)	0.0472 (4)
H5	0.5402	1.0682	0.3628	0.057*
C4	0.40632 (14)	0.95678 (12)	0.21295 (17)	0.0351 (3)
C9	0.40864 (14)	0.86489 (12)	0.10706 (17)	0.0355 (3)
C1	0.27138 (15)	0.80827 (13)	0.02130 (17)	0.0380 (3)
C10	-0.00949 (18)	0.69041 (14)	0.0623 (2)	0.0494 (4)
H10A	0.0594	0.6327	0.0439	0.074*
H10B	-0.1073	0.6629	0.0204	0.074*
H10C	0.0040	0.7047	0.1770	0.074*
C7	0.67083 (16)	0.87297 (17)	0.1567 (2)	0.0561 (5)
H7	0.7592	0.8453	0.1381	0.067*
C8	0.54055 (16)	0.82281 (15)	0.0780 (2)	0.0474 (4)
H8	0.5407	0.7614	0.0061	0.057*
C3	0.26443 (15)	1.00432 (13)	0.2396 (2)	0.0414 (4)
H3A	0.2520	1.0820	0.1935	0.050*
H3B	0.2722	1.0120	0.3560	0.050*
C2	0.12869 (14)	0.93600 (11)	0.17095 (18)	0.0357 (3)

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O2	0.0317 (5)	0.0547 (6)	0.0432 (6)	-0.0089 (4)	-0.0047 (4)	0.0048 (5)
O1	0.0533 (7)	0.0720 (8)	0.0621 (8)	-0.0098 (6)	0.0187 (6)	-0.0330 (7)
O3	0.0356 (5)	0.0535 (7)	0.0727 (8)	0.0064 (4)	0.0213 (5)	0.0006 (6)
N1	0.0258 (5)	0.0423 (6)	0.0363 (6)	-0.0027 (4)	0.0019 (4)	-0.0008 (5)
C6	0.0311 (7)	0.0660 (10)	0.0622 (11)	-0.0096 (7)	0.0019 (7)	0.0096 (9)
C5	0.0414 (7)	0.0482 (8)	0.0494 (9)	-0.0089 (6)	0.0044 (6)	-0.0024 (7)
C4	0.0314 (6)	0.0370 (6)	0.0362 (7)	-0.0012 (5)	0.0059 (5)	0.0038 (5)
C9	0.0296 (6)	0.0403 (7)	0.0371 (7)	-0.0005 (5)	0.0086 (5)	0.0037 (5)
C1	0.0346 (7)	0.0446 (7)	0.0354 (7)	-0.0014 (5)	0.0090 (6)	-0.0030 (6)
C10	0.0444 (8)	0.0496 (8)	0.0536 (9)	-0.0113 (6)	0.0094 (7)	-0.0015 (7)
C7	0.0302 (7)	0.0668 (11)	0.0736 (12)	0.0039 (6)	0.0164 (7)	0.0114 (9)
C8	0.0389 (7)	0.0505 (8)	0.0562 (9)	0.0043 (6)	0.0182 (7)	0.0001 (7)
C3	0.0380 (7)	0.0386 (7)	0.0480 (8)	0.0005 (5)	0.0103 (6)	-0.0065 (6)
C2	0.0312 (6)	0.0365 (6)	0.0398 (7)	0.0050 (5)	0.0084 (5)	0.0052 (5)

Geometric parameters (Å, º)

O2—N1	1.3898 (13)	C4—C3	1.4960 (19)
O2—C10	1.4378 (19)	C9—C8	1.395 (2)
O1—C1	1.2061 (18)	C9—C1	1.4802 (18)
O3—C2	1.2028 (17)	C10—H10A	0.9600
N1—C2	1.3896 (19)	C10—H10B	0.9600
N1—C1	1.3918 (18)	C10—H10C	0.9600
C6—C7	1.377 (3)	C7—C8	1.381 (2)
C6—C5	1.379 (2)	C7—H7	0.9300
C6—H6	0.9300	C8—H8	0.9300
C5—C4	1.3977 (19)	C3—C2	1.4981 (19)
C5—H5	0.9300	C3—H3A	0.9700
C4—C9	1.385 (2)	C3—H3B	0.9700
N1—O2—C10	110.74 (10)	O2—C10—H10B	109.5
C2—N1—O2	115.64 (10)	H10A—C10—H10B	109.5

C2—N1—C1	127.92 (11)	O2—C10—H10C	109.5
O2—N1—C1	116.39 (11)	H10A—C10—H10C	109.5
C7—C6—C5	120.78 (14)	H10B—C10—H10C	109.5
C7—C6—H6	119.6	C6—C7—C8	119.86 (15)
C5—C6—H6	119.6	C6—C7—H7	120.1
C6—C5—C4	120.28 (15)	C8—C7—H7	120.1
C6—C5—H5	119.9	C7—C8—C9	119.58 (16)
C4—C5—H5	119.9	C7—C8—H8	120.2
C9—C4—C5	118.55 (13)	C9—C8—H8	120.2
C9—C4—C3	120.84 (12)	C4—C3—C2	117.13 (12)
C5—C4—C3	120.59 (13)	C4—C3—H3A	108.0
C4—C9—C8	120.95 (13)	C2—C3—H3A	108.0
C4—C9—C1	121.08 (12)	C4—C3—H3B	108.0
C8—C9—C1	117.96 (13)	C2—C3—H3B	108.0
O1—C1—N1	120.64 (13)	H3A—C3—H3B	107.3
O1—C1—C9	123.48 (13)	O3—C2—N1	121.17 (13)
N1—C1—C9	115.86 (12)	O3—C2—C3	122.98 (13)
O2—C10—H10A	109.5	N1—C2—C3	115.81 (11)
C10—O2—N1—C2	-95.52 (14)	C4—C9—C1—N1	-2.6 (2)
C10—O2—N1—C1	82.16 (15)	C8—C9—C1—N1	177.41 (13)
C7—C6—C5—C4	0.3 (3)	C5—C6—C7—C8	-0.2 (3)
C6—C5—C4—C9	0.0 (2)	C6—C7—C8—C9	-0.1 (3)
C6—C5—C4—C3	-178.31 (14)	C4—C9—C8—C7	0.4 (2)
C5—C4—C9—C8	-0.3 (2)	C1—C9—C8—C7	-179.57 (14)
C3—C4—C9—C8	177.95 (14)	C9—C4—C3—C2	10.4 (2)
C5—C4—C9—C1	179.63 (13)	C5—C4—C3—C2	-171.36 (13)
C3—C4—C9—C1	-2.1 (2)	O2—N1—C2—O3	5.3 (2)
C2—N1—C1—O1	176.59 (14)	C1—N1—C2—O3	-172.07 (14)
O2—N1—C1—O1	-0.8 (2)	O2—N1—C2—C3	-172.41 (11)
C2—N1—C1—C9	-1.9 (2)	C1—N1—C2—C3	10.2 (2)
O2—N1—C1—C9	-179.26 (10)	C4—C3—C2—O3	168.54 (14)

C4—C9—C1—O1	178.98 (15)	C4—C3—C2—N1	-13.79 (19)
C8—C9—C1—O1	-1.0 (2)		