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Supporting Information
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Thieme

Mn(III)-Based Oxidative Cyclization of *N*-Aryl-2-oxocycloalkane-1-carboxamides: Synthesis of Spiroindolinones

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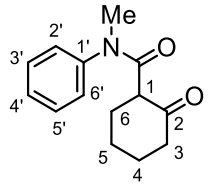
Supporting Information

Preparation of *N*-aryl-2-oxocycloalkane-1-carboxamides **1a-y**, spectroscopic data of **1a-y**, ^{1}H NMR and ^{13}C NMR spectra of the 2-oxocycloalkane-1-carboxamides **1a-y**, the product spiroindolinones **2a-y**, the alcohols **3** and **4**, the Baeyer-Villiger product **5**, and the hydrazones **6a-c**.

Preparation of Materials

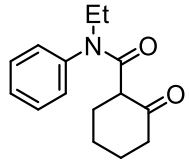
A mixture of *N*-methylaniline (0.650 mL) and ethyl 2-oxocyclohexane-1-carboxylate (0.170 g) was heated under reflux for 24 h, then the crude products were separated by silica gel column chromatography eluting with EtOAc/hexane/acetone (2:7:1 v/v), giving *N*-methyl-2-oxo-*N*-phenylcyclohexane-1-carboxamide (**1a**) (0.126 g; 54% yield). The other *N*-alkyl-2-oxocycloalkane-1-carboxamides **1b-y** were prepared according to a procedure similar to that already described.

***N*-Methyl-2-oxo-*N*-phenylcyclohexanecarboxamide (1a)** [1]



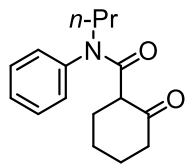
Yield (54%); R_f = 0.25 (2:7:1 AcOEt-hexane-acetone).
Colorless microcrystals (from EtOH-hexane): mp 59-61 °C.
IR (neat): ν = 1709 (-C=O), 1690 (-N-C=O).
^{1}H NMR (500 Hz, $CDCl_3$): δ 7.40-7.33 (3H, m, arom H), 7.17 (2H, dd, J = 8.5, 1.4 Hz, arom H), 3.31 (3H, s, =N-Me), 3.23 (1H, dd, J = 11.6, 5.8 Hz, *H*-CH), 2.41 (1H, d, J = 13.5 Hz, *H*-CH), 2.19 (1H, dq, J = 12.4, 3.8 Hz, *H*-CH), 2.04-1.98 (2H, m, CH_2), 1.95-1.88 (2H, m, CH_2), 1.77-1.68 (1H, m, *H*-CH), 1.51-1.41 (1H, m, *H*-CH).
^{13}C NMR (125 MHz, $CDCl_3$): δ 207.2 (C=O), 169.4 (-N-C=O), 143.4 (C-1'), 129.6 (2C) (C-2' and 6'), 127.9 (C-4'), 126.9 (2C) (C-3' and 5'), 54.9 (C-1), 41.3 (CH_2), 37.1 (=N-Me), 30.2, 26.6, 23.4 (CH_2).
FAB HRMS (acetone-NBA): calcd for $C_{15}H_{20}NO_2$ 232.1338 (M+H); found 232.1332.

***N*-Ethyl-2-oxo-*N*-phenylcyclohexanecarboxamide (1b)**



Yield (45%); R_f = 0.24 (2:7:1 AcOEt-hexane-acetone).
Colorless microcrystals (from EtOH-hexane): mp 63-65 °C.
IR (neat): ν = 1713 (-C=O), 1657 (-N-C=O).
^{1}H NMR (500 Hz, $CDCl_3$): δ 7.41-7.34 (3H, m, arom H), 7.16 (2H, d, J = 8.0 Hz, arom H), 3.88-3.81 (1H, m, *H*-CH-CH_3), 3.75-3.68 (1H, m, *H*-CH-CH_3), 3.16 (1H, dd, J = 11.5, 6.5 Hz, *H*-CH), 2.39 (1H, d, J = 14.0, Hz, *H*-CH), 2.17 (1H, q, J = 14.0, Hz, *H*-CH), 2.03-1.96 (2H, m, CH_2), 1.94-1.87 (2H, m, CH_2), 1.74-1.67 (1H, m, *H*-CH), 1.50-1.42 (1H, m, *H*-CH), 1.14 (3H, t, J = 7.3 Hz, -CH_2-$\underline{C}H_3$).
^{13}C NMR (125 MHz, $CDCl_3$): δ 207.1 (C=O), 168.8 (-N-C=O), 141.8 (C-1'), 129.4 (2C) (C-2' and 6'), 128.1 (2C) (C-3' and 5'), 128.0 (C-4'), 55.2 (C-1), 43.9 (-$\underline{C}H_2$-CH_3), 41.4, 30.1, 26.6, 23.4 (CH_2), 12.8 (-CH_2-$\underline{C}H_3$).
FAB HRMS (acetone-NBA): calcd for $C_{15}H_{20}NO_2$ 246.1494 (M+H); found 246.1488.

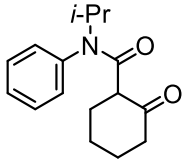
2-Oxo-*N*-phenyl-*N*-*n*-propylcyclohexanecarboxamide (1c)



Yield (55%); R_f = 0.30 (2:7:1 AcOEt-hexane-acetone);
Colorless microcrystals (from EtOH-hexane): mp 90-92 °C.
IR (neat): ν = 1713 (-C=O), 1655 (-N-C=O).

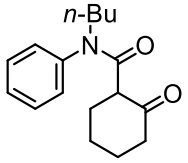
^{1}H NMR (500 Hz, $CDCl_3$): δ 7.40-7.33 (3H, m, arom H), 7.15 (2H, d, J = 8.0 Hz, arom H), 3.83-3.78 (1H, m, *H*-CH-CH_2-CH_3), 3.61-3.55 (1H, m, *H*-CH-CH_2-CH_3), 3.14 (1H, dd, J = 11.5, 6.0 Hz, *H*-CH), 2.39 (1H, d, J = 13.5, Hz, *H*-CH), 2.17 (1H, q, J = 12.6 Hz, *H*-CH), 2.01-1.95 (2H, m, CH_2), 1.93-1.86 (2H, m, CH_2), 1.75-1.67 (1H, m, *H*-CH), 1.59-1.51 (2H, m, -CH_2-C$\underline{H}_2$-CH_3), 1.48-1.41 (1H, m, *H*-CH), 0.92 (3H, t, J = 7.3 Hz, -CH_2-CH_2-C$\underline{H}_3$).
^{13}C NMR (125 MHz, $CDCl_3$): δ 207.4 (C=O), 169.2 (-N-C=O), 142.3 (C-1'), 129.6 (2C) (C-2' and 6'), 128.2 (C-4'), 128.1 (2C) (C-3' and 5'), 55.5 (C-1), 50.8 (-$\underline{C}H_2$-CH_2-CH_3), 41.6, 30.4, 26.8, 23.7 (CH_2), 21.0 (-CH_2-$\underline{C}H_2$-CH_3), 11.2 (-CH_2-CH_2-$\underline{C}H_3$).
Anal. Calcd for $C_{16}H_{21}NO_2$: C, 74.10; H, 8.16; N, 5.40. Found: C, 74.07; H, 8.41; N, 5.36.

2-Oxo-*N*-phenyl-*N*-*i*-propylcyclohexanecarboxamide (1d)



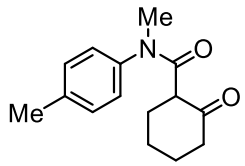
Yield (84%); R_f = 0.27 (2:7:1 AcOEt-hexane-acetone).
Colorless microcrystals (from EtOH-hexane): mp 114-116 °C.
IR (neat): ν = 1713 (-C=O), 1651 (-N-C=O);
^{1}H NMR (500 Hz, $CDCl_3$): δ 7.44-7.34 (3H, m, arom H), 7.12 (1H, d, J = 7.5 Hz, arom H), 7.07 (1H, d, J = 7.5 Hz, arom H), 5.07 (1H, m, J = 6.5 Hz, -C$\underline{H}$-(CH_3)$_2$), 3.06 (1H, dd, J = 12.0, 5.5 Hz, *H*-CH), 2.36 (1H, d, J = 12.5, Hz, *H*-CH), 2.16 (1H, q, J = 12.3 Hz, *H*-CH), 2.02-1.98 (1H, m, *H*-CH), 1.93-1.85 (3H, m, $\underline{H}$-CH-C$\underline{H}_2$), 1.73-1.66 (1H, m, *H*-CH), 1.47-1.39 (1H, m, *H*-CH), 1.10 (3H, d, J = 7.0 Hz, -CH-(C$\underline{H}_3$)$_2$) , 1.04 (3H, d, J = 7.0 Hz, -CH-(C$\underline{H}_3$)$_2$).
^{13}C NMR (125 MHz, $CDCl_3$): δ 207.3 (C=O), 168.7 (-N-C=O), 138.1 (C-1'), 130.3, 129.8, 129.0, 128.9, 128.3 (arom C), 55.9 (C-1), 45.9 (-$\underline{C}$H-(CH_3)$_2$)), 41.5, 30.2, 26.8, 23.6 (CH_2), 20.9 (-CH-($\underline{C}H_3$)$_2$), 20.8 (-CH-($\underline{C}H_3$)$_2$).
Anal. Calcd for $C_{16}H_{21}NO_2$: C, 74.10; H, 8.16; N, 5.40. Found: C, 74.01; H, 8.43; N, 5.42.

***N*-Buthyl-2-oxo-*N*-phenylcyclohexanecarboxamide (1e)**



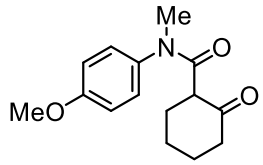
Yield (83%); R_f = 0.30 (2:7:1 AcOEt-hexane-acetone).
Orange oil.
IR ($CHCl_3$): ν = 1713 (-C=O), 1655 (-N-C=O).
^{1}H NMR (500 Hz, $CDCl_3$): δ 7.41-7.34 (3H, m, arom H), 7.15 (2H, d, J = 7.0 Hz, arom H), 3.88-3.82 (1H, m, J = 6.5 Hz, *H*-CH-CH_2-CH_2-CH_3), 3.64-3.58 (1H, m, J = 6.5 Hz, *H*-CH-CH_2-CH_2-CH_3), 3.15 (1H, dd, J = 11.5, 5.0 Hz, *H*-CH), 2.39 (1H, d, J = 13.5, Hz, *H*-CH), 2.18 (1H, q, J = 12.8 Hz, *H*-CH), 2.03-1.96 (2H, m, CH_2), 1.94-1.87 (2H, m, CH_2), 1.74-1.67 (1H, m, *H*-CH), 1.56-1.44 (3H, m, *H*-CH/-CH_2-C$\underline{H}_2$-CH_2-CH_3), 1.39-1.32 (2H, m, -CH_2-CH_2-C$\underline{H}_2$-CH_3), 0.90 (3H, t, J = 7.5 Hz, -CH_2-CH_2-CH_2-C$\underline{H}_3$).
^{13}C NMR (125 MHz, $CDCl_3$): δ 207.3 (C=O), 169.1 (-N-C=O), 142.1 (C-1'), 129.5 (2C) (C-2' and 6'), 128.1 (C-4'), 128.0 (2C) (C-3' and 5'), 55.3 (C-1), 45.9 (-$\underline{C}H_2$-CH_2-CH_2-CH_3), 41.5, 30.2 (CH_2), 29.7 (-CH_2-$\underline{C}H_2$-CH_2-CH_3), 26.7, 23.5 (CH_2), 19.8 (-CH_2-CH_2-$\underline{C}H_2$-CH_3), 13.7 (-CH_2-CH_2-CH_2-$\underline{C}H_3$).
FAB HRMS (acetone-NBA): calcd for $C_{17}H_{24}NO_2$ 274.1807 (M+H); found 274.1805.

***N*-(4-Methylphenyl)-*N*-methyl-2-oxocyclohexanecarboxamide (1f)**



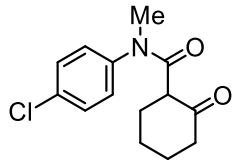
Yield (73%); R_f = 0.31 (1:8:1 AcOEt-hexane-acetone).
Orange oil.
IR ($CHCl_3$): ν = 1709 (-C=O), 1651 (-N-C=O).
^{1}H NMR (500 Hz, $CDCl_3$): δ 7.17 (2H, d, J = 8.0 Hz, arom H), 7.04 (2H, d, J = 8.0 Hz, arom H), 3.28 (3H, s, =N-Me), 3.25 (1H, dd, J = 11.5, 6.0 Hz, *H*-CH), 2.41 (1H, d, J = 14.0 Hz, *H*-CH), 2.37 (3H, s, Me), 2.17 (1H, q, J = 12.0 Hz, *H*-CH), 2.03-1.97 (2H, m, CH_2), 1.94-1.86 (2H, m, CH_2), 1.77-1.68 (1H, m, *H*-CH), 1.51-1.42 (1H, m, *H*-CH).
^{13}C NMR (125 MHz, $CDCl_3$): δ 207.4 (C=O), 169.7 (-N-C=O), 141.1 (C-1'), 138.0 (C-4'), 130.4 (2C) (C-2' and 6'), 126.9 (2C) (C-3' and 5'), 55.1 (C-1), 41.6 (CH_2), 37.4 (=N-Me), 30.4, 26.8, 23.7 (CH_2), 21.1 (Me).
FAB HRMS (acetone-NBA): calcd for $C_{15}H_{20}NO_2$ 246.1494 (M+H); found 246.1494.

_N_-(4-Methoxyphenyl)-_N_-methyl-2-oxocyclohexanecarboxamide (1g)



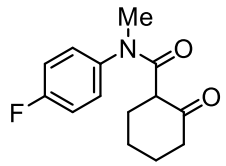
Yield (71%); R_f = 0.26 (1:8:1 AcOEt-hexane-acetone).
Yellow oil.
IR ($CHCl_3$): ν = 1709 (-C=O), 1651 (-N-C=O).
^{1}H NMR (500 Hz, $CDCl_3$): δ 7.09 (2H, d, J = 9.0 Hz, arom H), 6.88 (2H, d, J = 8.5 Hz, arom H), 3.82 (3H, s, MeO), 3.27 (3H, s, =N-Me), 3.24 (1H, dd, J = 12.0, 6.5 Hz, *H*-CH), 2.41 (1H, d, J = 13.5 Hz, *H*-CH), 2.17 (1H, q, J = 12.7 Hz, *H*-CH), 2.04-1.97 (2H, m, CH_2), 1.95-1.87 (2H, m, CH_2), 1.77-1.68 (1H, m, *H*-CH), 1.50-1.43 (1H, m, *H*-CH).
^{13}C NMR (125 MHz, $CDCl_3$): δ 207.2 (C=O), 169.6 (-N-C=O), 158.8 (C-4'), 136.1 (C-1'), 128.0 (2C) (C-2' and 6'), 114.5 (2C) (C-3' and 5'), 55.2 (MeO), 54.8 (C-1), 41.4 (CH_2), 37.2 (=N-Me), 30.1, 26.5, 23.4 (CH_2).
FAB HRMS (acetone-NBA): calcd for $C_{15}H_{20}NO_3$ 262.1443 (M+H); found 262.1494.

_N_-(4-Chlorophenyl)-_N_-methyl-2-oxocyclohexanecarboxamide (1h)



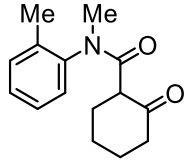
Yield (38%); R_f = 0.30 (1:8:1 AcOEt-hexane-acetone).
Yellow oil.
IR ($CHCl_3$): ν = 1709 (-C=O), 1651 (-N-C=O);
^{1}H NMR (500 Hz, $CDCl_3$); δ 7.36 (2H, d, J = 8.5 Hz, arom H), 7.13 (2H, d, J = 9.0 Hz, arom H), 3.28 (3H, s, =N-Me), 3.20 (1H, dd, J = 12.5, 6.0 Hz, *H*-CH), 2.42 (1H, d, J = 14.0 Hz, *H*-CH), 2.19 (1H, q, J = 12.7 Hz, *H*-CH), 2.05-1.99 (2H, m, CH_2), 1.97-1.89 (2H, m, CH_2), 1.77-1.69 (1H, m, *H*-CH), 1.51-1.44 (1H, m, *H*-CH).
^{13}C NMR (125 MHz, $CDCl_3$): δ 207.1 (C=O), 169.2 (-N-C=O), 141.8 (C-1'), 133.6 (C-4'), 129.7 (2C) (C-2' and 6'), 128.4 (2C) (C-3' and 5'), 54.9 (C-1), 41.3 (CH_2), 37.1 (=N-Me), 30.1, 26.4, 23.3 (CH_2).
FAB HRMS (acetone-NBA): calcd for $C_{14}H_{17}ClNO_2$ 266.0948 (M+H); found 266.0941.

_N_-(4-Fluorophenyl)-_N_-methyl-2-oxocyclohexanecarboxamide (1i)



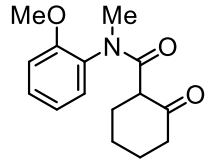
Yield (54%); R_f = 0.28 (1:8:1 AcOEt-hexane-acetone).
Yellow oil.
IR ($CHCl_3$): ν = 1707 (-C=O), 1651 (-N-C=O).
^{1}H NMR (500 Hz, $CDCl_3$) (1:1 rotamer mixture): δ 7.21 and 7.19 (2H, d, J = 9.0 Hz, arom H), 7.11 and 7.09 (2H, d, J = 9.0 Hz, arom H), 3.29 (3H, s, =N-Me), 3.24 (1H, dd, J = 11.9, 5.9 Hz, *H*-CH), 2.41 (1H, dt, J = 13.7, 3.6 Hz, *H*-CH), 2.18 (1H, dd, J = 13.5, 3.7 Hz, *H*-CH), 2.09-2.02 (2H, m, CH_2), 2.00-1.89 (2H, m, CH_2), 1.77-1.68 (1H, m, *H*-CH), 1.56-1.47 (1H, m, *H*-CH).
^{13}C NMR (125 MHz, $CDCl_3$): δ 207.0 (C=O), 169.2 (-N-C=O), 161.4 (d, J = 247 Hz, C-4'), 139.3 (d, J = 3 Hz, C-1'), 128.7 (2C) (d, J = 9 Hz, C-2' and 6'), 116.3 (2C) (d, J = 9 Hz, C-3'5'), 54.8 (C-1), 41.3 (CH_2), 37.1 (=N-Me), 30.0, 26.4, 23.3 (CH_2).
FAB HRMS (acetone-NBA): calcd for $C_{14}H_{17}FNO_2$ 250.1243 (M+H); found 250.1238.

_N_-(2-Methylphenyl)-_N_-methyl-2-oxocyclohexanecarboxamide (1j)



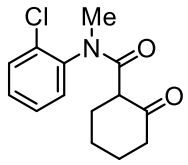
Yield (62%); R_f = 0.26 (1:8:1 AcOEt-hexane-acetone).
Colorless microcrystals (from EtOH-hexane): mp 73-75 °C.
IR (neat): ν = 1705 (-C=O), 1653 (-N-C=O).
^{1}H NMR (500 Hz, $CDCl_3$) (1:2 rotamer mixture): δ 7.29-7.03 (5H, m, arom H), 3.23 and 3.21 (3H, s, =N-Me), 3.16 and 2.99 (1H, dd, J = 12.0, 5.7 Hz, H-1), 2.54 and 2.37 (1H, dt, J = 14.5, 5.3 Hz, *H*-CH), 2.33-2.05 (2H, m, *H*-CH), 2.26 and 2.20 (3H, s, Me), 2.03-1.66 (3H, m), 1.51-1.41 (1H, m).
^{13}C NMR (125 MHz, $CDCl_3$) (1:2 rotamer mixture): δ 206.9 and 206.8 (C=O), 170.0, 169.5 (-N-C=O), 141.9 (C-1'), 136.2 and 134.8 (C-2'), 131.7 and 131.0, 128.4 and 128.3, 127.9 and 127.5, 127.3 and 127.1 (arom C), 55.1 and 54.0 (C-1), 41.4 and 41.2 (CH_2), 35.7 and 35.6 (=N-Me), 30.3 and 30.2, 26.6 and 26.1, 23.6 and 22.8 (CH_2), 17.2 and 17.1 (Me).
FAB HRMS (acetone-NBA): calcd for $C_{15}H_{20}NO_2$ 246.1494 (M+H); found 246.1503.

_N_-(2-Methoxyphenyl)-_N_-methyl-2-oxocyclohexanecarboxamide (1k)



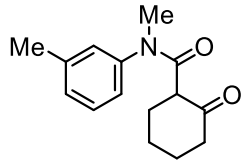
Yield (30%); R_f = 0.30 (2:7:1 AcOEt-hexane-acetone).
Colorless microcrystals (from EtOH-hexane): mp 98-100 °C.
IR (neat): ν = 1709 (-C=O), 1651 (-N-C=O).
^{1}H NMR (500 Hz, $CDCl_3$): δ 7.33-7.30 (1H, m, arom H), 7.17 (1H, d, J = 7.5 Hz, arom H), 6.97-6.90 (2H, m, arom H), 3.84 (3H, s, MeO), 3.21 (3H, s, =N-Me), 3.09 (1H, dd, J = 11.5, 5.5 Hz, *H*-CH), 2.40 (1H, d, J = 13.5 Hz, *H*-CH), 2.21-2.14 (1H, m, *H*-CH), 2.02-1.93 (2H, m, CH_2), 1.89-1.87 (2H, m, CH_2), 1.76-1.71 (1H, m, *H*-CH), 1.50-1.44 (1H, m, *H*-CH).
^{13}C NMR (125 MHz, $CDCl_3$): δ 207.5 (C=O), 170.1 (-N-C=O), 154.8 (C-2'), 131.9 (C-1'), 129.5, 129.3, 121.1, 111.6 (arom C), 55.5 (C-1), 55.1 (MeO), 41.6 (CH_2), 36.0 (=N-Me), 30.3, 26.8, 23.7 (CH_2).
FAB HRMS (acetone-NBA): calcd for $C_{15}H_{20}NO_3$ 262.1443 (M+H); found 262.1424.

_N_-(2-Chlorophenyl)-_N_-methyl-2-oxocyclohexanecarboxamide (1l)



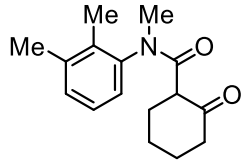
Yield (82%); R_f = 0.38(2:7:1 AcOEt-hexane-acetone).
Colorless microcrystals (from EtOH-hexane): mp 104-106 °C.
IR (neat): ν = 1707 (-C=O), 1662 (-N-C=O).
^{1}H NMR (500 Hz, $CDCl_3$): δ 7.50 (1H, d, J = 8.0 Hz, arom H), 7.35-7.28 (3H, m, arom H), 3.24 (3H, s, =N-Me), 2.98 (1H, dd, J = 11.5, 5.5 Hz, *H*-CH), 2.39-2.35 (1H, m, *H*-CH), 2.26-2.17 (1H, m, *H*-CH), 2.11-2.07 (1H, m, CH_2), 1.98-1.91 (3H, m, CH_2), 1.75-1.70 (1H, m, *H*-CH), 1.53-1.45 (1H, m, *H*-CH).
^{13}C NMR (125 MHz, $CDCl_3$): δ 207.1 (C=O), 169.5 (-N-C=O), 140.6 (C-1'), 132.7 (C-2'), 130.3, 130.1, 129.7, 128.3 (arom C), 55.4 (C-1), 41.6 (CH_2), 35.8 (=N-Me), 30.3, 26.7, 23.7 (CH_2).
FAB HRMS (acetone-NBA): calcd for $C_{14}H_{17}ClNO_2$ 266.0948 (M+H); found 266.0945.

_N_-(3-Methylphenyl)-_N_-methyl-2-oxocyclohexanecarboxamide (1m)



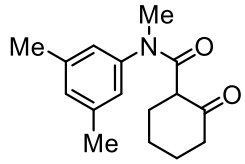
Yield (52%); R_f = 0.25 (1:8:1 AcOEt-hexane-acetone).
Yellow oil.
IR ($CHCl_3$): ν = 1709 (-C=O), 1653 (-N-C=O).
^{1}H NMR (500 Hz, $CDCl_3$): δ 7.27-7.24 (1H, m, arom H), 7.14 (1H, d, J = 8.0 Hz, arom H), 6.96 (2H, d, J = 12.0 Hz, arom H), 3.29 (3H, s, =N-Me), 3.24 (1H, dd, J = 11.0, 5.5 Hz, *H*-CH), 2.43 (1H, d, J = 14.0 Hz, *H*-CH), 2.35 (3H, s, Me), 2.18 (1H, q, J = 12.7 Hz, *H*-CH), 2.03-1.97 (2H, m, CH_2), 1.94-1.87 (2H, m, CH_2), 1.78-1.69 (1H, m, *H*-CH), 1.51-1.43 (1H, m, *H*-CH).
^{13}C NMR (125 MHz, $CDCl_3$): δ 207.4 (C=O), 169.5 (-N-C=O), 143.5 (C-1'), 139.8 (C-4'), 129.4, 128.7, 127.5, 123.9 (arom C), 55.0 (C-1), 41.5 (CH_2), 37.2 (=N-Me), 30.3, 26.7, 23.5 (CH_2), 21.1 (Me).
FAB HRMS (acetone-NBA): calcd for $C_{15}H_{20}NO_2$ 246.1494 (M+H); found 246.1484.

_N_-(2,3-Dimethylphenyl)-_N_-methyl-2-oxocyclohexanecarboxamide (1n)



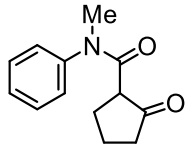
Yield (39%); R_f = 0.34 (1:8:1 AcOEt-hexane-acetone).
Yellow oil.
IR ($CHCl_3$): ν = 1709 (-C=O), 1651 (-N-C=O).
^{1}H NMR (500 Hz, $CDCl_3$) (2:3 rotamer mixture): δ 7.17 and 7.16 (1H, d, J = 7.7 Hz, arom H), 7.13 and 7.07 (1H, t, J = 7.7 Hz, arom H), 6.99 and 6.91 (1H, d, J = 7.7 Hz, arom H), 3.21 and 3.19 (3H, s, =N-Me), 3.19 and 3.04 (1H, dd, J = 12.0, 5.7 Hz, H-1), 2.52 and 2.37 (1H, dt, J = 14.2, 5.7 Hz, *H*-CH), 2.29-2.15 (2H, m, CH_2), 2.34 and 2.31 (3H, s, Me), 2.12 and 2.11 (3H, s, Me) 2.03-1.66 (4H, m, CH_2), 1.53-1.46 (1H, m, *H*-CH).
^{13}C NMR (125 MHz, $CDCl_3$) (2:3 rotamer mixture): δ 206.8 (C=O), 170.0 and 169.6 (-N-C=O), 141.9 (C-1'), 139.0 and 138.3 (C-3'), 134.7 and 133.3 (C-2'), 129.7 and 129.6, 126.5 and 126.3, 125.3 and 124.9 (arom C), 54.9 and 54.0 (C-1), 41.3 and 41.2 (CH_2), 36.0 and 35.9 (=N-Me), 30.2 and 30.1, 26.5 and 26.1, 23.5 and 22.8 (CH_2), 20.2 (Me-C-2'), 13.7 and 13.5 (Me-C-3').
FAB HRMS (acetone-NBA): calcd for $C_{16}H_{22}NO_2$ 260.1651 (M+H); found 260.1640.

_N_-(3,5-Dimethylphenyl)-_N_-methyl-2-oxocyclohexanecarboxamide (1o)



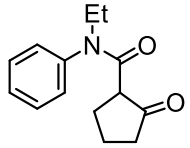
Yield (82%); R_f = 0.31 (1:8:1 AcOEt-hexane-acetone).
Orange oil.
IR ($CHCl_3$): ν = 1713 (-C=O), 1651 (-N-C=O).
^{1}H NMR (500 Hz, $CDCl_3$): δ 6.96 (1H, s, arom H), 6.77 (2H, s, arom H), 3.28-3.25 (4H, m, =N-Me and *H*-CH), 2.44 (1H, d, J = 14.5 Hz, *H*-CH), 2.30 (6H, s, Me), 2.18 (1H, q, J = 12.5 Hz, *H*-CH), 2.06-1.99 (2H, m, CH_2), 1.95-1.87 (2H, m, CH_2), 1.78-1.70 (1H, m, *H*-CH), 1.52-1.44 (1H, m, *H*-CH).
^{13}C NMR (125 MHz, $CDCl_3$): δ 207.5 (C=O), 169.6 (-N-C=O), 143.6 (C-1'), 139.5 (2C) (C-3' and 5'), 129.7 (2C) (C-2' and 6'), 124.5 (C-4'), 55.1 (C-1), 41.5 (CH_2), 37.3 (=N-Me), 30.4, 26.8, 23.6 (CH_2), 21.1 (2C, Me).
FAB HRMS (acetone-NBA): calcd for $C_{16}H_{22}NO_2$ 260.1651 (M+H); found 260.1646.

_N_-Methyl-2-oxo-_N_-phenylcyclopentanecarboxamide (1p)[2]



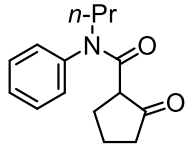
Yield (87%); R_f = 0.25 (2:7:1 AcOEt-hexane-acetone).
Colorless microcrystals (from EtOH-hexane): mp 59-61 °C.
IR (neat): ν = 1744 (-C=O), 1643 (-N-C=O).
^{1}H NMR (500 Hz, $CDCl_3$): δ 7.42 (2H, t, J = 7.5 Hz, arom H), 7.37-7.33 (3H, m, arom H), 3.31 (3H, s, =N-Me), 3.11 (1H, t, J = 9.5 Hz, *H*-CH), 2.43-2.35 (1H, m, *H*-CH), 2.32-2.18 (2H, m, CH_2), 2.14-2.07 (2H, m, CH_2), 1.71-1.61 (1H, m, *H*-CH).
^{13}C NMR (125 MHz, $CDCl_3$): δ 214.8 (C=O), 169.9 (-N-C=O), 143.7 (C-1'), 129.8 (2C) (C-2' and 6'), 128.0 (C-4'), 127.5 (2C) (C-3' and 5'), 52.6 (C-1), 38.4 (CH_2), 37.5 (=N-Me), 28.3, 21.0 (CH_2).

_N_-Ethyl-2-oxo-_N_-phenylcyclopentanecarboxamide (1q)



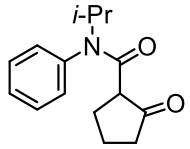
Yield (78%); R_f = 0.24 (1:8:1 AcOEt-hexane-acetone).
Colorless microcrystals (from EtOH-hexane): mp 60-62 °C.
IR (neat): ν = 1740 (-C=O), 1643 (-N-C=O).
^{1}H NMR (500 Hz, $CDCl_3$): δ 7.42 (2H, t, J = 7.5 Hz, arom H), 7.38-7.28 (3H, m, arom H), 3.89-3.82 (1H, m, *H*-CH-CH_3), 3.75-3.68 (1H, m, *H*-CH-CH_3), 3.01 (1H, t, J = 9.5 Hz, *H*-CH), 2.42-2.34 (1H, m, *H*-CH), 2.30-2.14 (2H, m, CH_2), 2.11-2.06 (2H, m, CH_2), 1.69-1.61 (1H, m, *H*-CH), 1.13 (3H, t, J = 7.3 Hz, -CH_2-$C\underline{H}_3$).
^{13}C NMR (125 MHz, $CDCl_3$): δ 214.5 (C=O), 169.1 (-N-C=O), 141.8 (C-1'), 129.5 (2C) (C-2' and 6'), 128.4 (C-4'), 127.9 (2C) (C-3' and 5'), 52.7 (C-1), 44.1 (-$\underline{C}H_2$-CH_3), 38.2, 28.1, 20.8 (CH_2), 12.8 (-CH_2-$\underline{C}H_3$).
FAB HRMS (acetone-NBA): calcd for $C_{14}H_{18}NO_2$ 232.1338 (M+H); found 232.1338.

2-Oxo-_N_-phenyl-_N_-_n_-propylcyclopentanecarboxamide (1r)



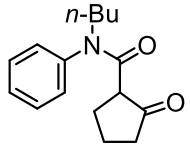
Yield (83%); R_f = 0.32 (1:8:1 AcOEt-hexane-acetone).
Colorless microcrystals (from EtOH-hexane): mp 81-83 °C.
IR (neat): ν = 1736 (-C=O), 1636 (-N-C=O).
^{1}H NMR (500 Hz, $CDCl_3$): δ 7.42 (2H, t, J = 7.5 Hz, arom H), 7.37-7.28 (3H, m, arom H), 3.85-3.79 (1H, m, *H*-CH-CH_2-CH_3), 3.59-3.54 (1H, m, *H*-CH-CH_2-CH_3), 3.02 (1H, t, J = 9.0 Hz, *H*-CH), 2.42-2.34 (1H, m, *H*-CH), 2.30-2.16 (2H, m, CH_2), 2.14-2.05 (2H, m, CH_2), 1.69-1.61 (1H, m, *H*-CH), 1.57-1.50 (2H, m, J = 7.2 Hz, -CH_2-C<u>H</u>$_2$-CH_3), 0.90 (3H, t, J = 7.5 Hz, -CH_2-CH_2-C<u>H</u>$_3$).
^{13}C NMR (125 MHz, $CDCl_3$): δ 214.8 (C=O), 169.6 (-N-C=O), 142.3 (C-1'), 129.6 (2C-) (C2' and 6'), 128.6 (C-4'), 128.0 (2C) (C-3' and 5'), 52.8 (C-1), 50.9 (<u>C</u>H_2-CH_2-CH_3), 38.4, 28.2, 21.0 (CH_2), 20.8 (-CH_2-<u>C</u>H_2-CH_3), 11.1 (-CH_2-CH_2-<u>C</u>H_3).
Anal. Calcd for $C_{15}H_{19}NO_2$: C, 73.44; H, 7.81; N, 5.71. Found: C, 73.25; H, 8.02; N, 5.68.

2-Oxo-*N*-phenyl-*N*-*i*-propylcyclopentanecarboxamide (1s)



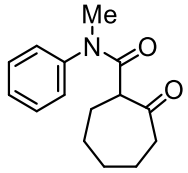
Yield (44%); R_f = 0.28 (1:8:1 AcOEt-hexane-acetone).
Colorless microcrystals (from EtOH-hexane): mp 109-111 °C.
IR (neat): ν = 1738 (-C=O), 1638 (-N-C=O).
^{1}H NMR (500 Hz, $CDCl_3$): δ 7.42-7.39 (4H, m, arom H), 7.05 (1H, d, J = 5.0 Hz, arom H), 5.01 (1H, m, J = 6.8 Hz, C<u>H</u>-(CH_3)$_2$), 2.83 (1H, t, J = 9.3 Hz, *H*-CH), 2.40-2.32 (1H, m, *H*-CH), 2.28-2.12 (2H, m, CH_2), 2.08-2.01 (2H, m, CH_2), 1.66-1.58 (1H, m, *H*-CH), 1.09 (3H, t, J = 7.0 Hz, -CH-(C<u>H</u>$_3$)$_2$), 1.05 (3H, t, J = 6.5 Hz, -CH-(C<u>H</u>$_3$)$_2$).
^{13}C NMR (125 MHz, $CDCl_3$): δ 214.8 (C=O), 169.3 (-N-C=O), 138.3 (C-1'), 131.3, 129.9, 128.9, 128.4 (arom C), 53.5 (C-1), 46.4 (-<u>C</u>H-(CH_3)$_2$), 38.4, 28.2 (CH_2), 21.1 (2C, -CH-(<u>C</u>H_3)$_2$), 20.9 (CH_2).
Anal. Calcd for $C_{15}H_{19}NO_2$: C, 73.44; H, 7.81; N, 5.71. Found: C, 73.27; H, 8.05; N, 5.70.

***N*-*n*-Buthyl-2-oxo-*N*-phenylcyclopentanecarboxamide (1t)**



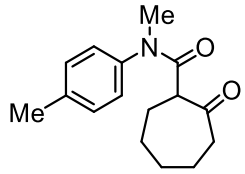
Yield (55%); R_f = 0.33 (1:8:1 AcOEt-hexane-acetone).
Colorless microcrystals (from EtOH-hexane): mp 89-91 °C.
IR (neat): ν = 1742 (-C=O), 1638 (-N-C=O).
^{1}H NMR (500 Hz, $CDCl_3$): δ 7.42 (2H, t, J = 8.0 Hz, arom H), 7.37-7.27 (3H, m, arom H), 3.87-3.82 (1H, m, *H*-CH-CH_2-CH_2-CH_3), 3.62-3.57 (1H, m, *H*-CH-CH_2-CH_2-CH_3), 3.01 (1H, t, J = 9.0 Hz, *H*-CH), 2.42-2.34 (1H, m, *H*-CH), 2.30-2.15 (2H, m, CH_2), 2.14-2.04 (2H, m, CH_2), 1.69-1.59 (1H, m, *H*-CH), 1.54-1.45 (2H, m, -CH_2-C<u>H</u>$_2$-CH_2-CH_3), 1.38-1.28 (2H, m, -CH_2-CH_2-C<u>H</u>$_2$-CH_3), 0.89 (3H, t, J = 7.0 Hz, -CH_2-CH_2-CH_2-C<u>H</u>$_3$),).
^{13}C NMR (125 MHz, $CDCl_3$): δ 214.8 (C=O), 169.5 (-N-C=O), 142.3 (C-1'), 129.6 (2C) (C-2' and 6'), 128.5 (C-4'), 128.0 (2C) (C-3' and 5'), 52.8 (C-1), 49.2 (-<u>C</u>H_2-CH_2-CH_2-CH_3), 38.5 (CH_2), 29.7 (-CH_2-<u>C</u>H_2-CH_2-CH_3), 28.3, 21.0 (CH_2), 19.9 (-CH_2-CH_2-<u>C</u>H_2-CH_3), 13.8 (-CH_2-CH_2-CH_2-<u>C</u>H_3).
Anal. Calcd for $C_{16}H_{21}NO_2$: C, 74.10; H, 8.16; N, 5.40. Found: C, 74.04; H, 8.45; N, 5.40.

***N*-Methyl-2-oxo-*N*-phenylcycloheptanecarboxamide (1u)**



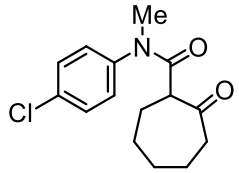
Yield (82%); R_f = 0.28 (1:8:1 AcOEt-hexane-acetone).
Yellow oil.
IR ($CHCl_3$): ν = 1703 (-C=O), 1645 (-N-C=O).
^{1}H NMR (500 Hz, $CDCl_3$): δ 7.42 (2H, t, J = 7.5 Hz, arom H), 7.36 (1H, t, J = 7.5 Hz, arom H), 7.21 (2H, d, J = 8.0 Hz, arom H), 3.47 (1H, dd, J = 10.5, 3.5 Hz, *H*-C-1), 3.27 (3H, s, =N-Me), 2.57 (1H, t, J = 13.3 Hz, *H*-CH), 2.13-2.08 (1H, m, *H*-CH), 2.01-1.95 (1H, m, *H*-CH), 1.92-1.84 (2H, m, CH_2), 1.82-1.76 (2H, m, CH_2), 1.35-1.12 (3H, m, CH_2-CH_2).
^{13}C NMR (125 MHz, $CDCl_3$): δ 210.7 (C=O), 170.4 (-N-C=O), 143.4 (C-1'), 129.8 (2C) (C-2' and 6'), 128.1 (C-4'), 127.9 (2C) (C-3' and 5'), 56.5 (C-1), 43.2 (CH_2), 37.4 (=N-Me), 29.5, 28.5, 28.3, 24.5 (CH_2).
FAB HRMS (acetone-NBA): calcd for $C_{16}H_{22}NO_2$ 246.1494 (M+H); found 246.1488.

_N_-(4-Methylphenyl)-_N_-methyl-2-oxocycloheptanecarboxamide (1v)



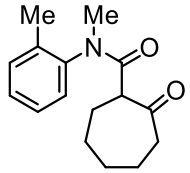
Yield (39%); R_f = 0.35 (1:8:1 AcOEt-hexane-acetone).
Yellow oil.
IR ($CHCl_3$): ν = 1703 (-C=O), 1645 (-N-C=O).
^{1}H NMR (500 Hz, $CDCl_3$): δ 7.22 (2H, d, J = 8.0 Hz, arom H), 7.09 (2H, d, J = 8.0 Hz, arom H), 3.49 (1H, q, J = 11.0, 4.0 Hz, *H*-C-1), 3.24 (3H, s, =N-Me), 2.57 (1H, t, J = 13.0 Hz, *H*-CH), 2.39 (3H, s, Me), 2.16-2.11 (1H, m, *H*-CH), 2.00-1.95 (1H, m, *H*-CH), 1.93-1.84 (2H, m, CH_2), 1.82-1.77 (2H, m, CH_2), 1.33-1.14 (3H, m, CH_2-CH_2).
^{13}C NMR (125 MHz, $CDCl_3$): δ 210.6 (C=O), 170.4 (-N-C=O), 140.6 (C-1'), 137.8 (C-4'), 130.1 (2C) (C-2' and 6'), 127.3 (2C) (C-3' and 5'), 56.2 (C-1), 43.0 (CH_2), 37.2 (=N-Me), 29.3, 28.3, 28.1, 24.3 (CH_2), 20.9 (Me).
FAB HRMS (acetone-NBA): calcd for $C_{16}H_{22}NO_2$ 260.1651 (M+H); found 260.1651.

_N_-(4-Chlorophenyl)-_N_-methyl-2-oxocycloheptanecarboxamide (1w)



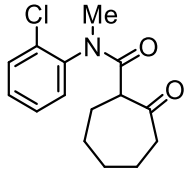
Yield (31%); R_f = 0.30 (1:8:1 AcOEt-hexane-acetone).
Yellow oil.
IR (neat): ν = 1703 (-C=O), 1651 (-N-C=O).
^{1}H NMR (500 Hz, $CDCl_3$): δ 7.41 (2H, d, J = 8.0 Hz, arom H), 7.19 (2H, d, J = 8.5 Hz, arom H), 3.44 (1H, dd, J = 10.5, 4.0 Hz, *H*-C-1), 3.25 (3H, s, =N-Me), 2.62 (1H, t, J = 13.0 Hz, *H*-CH), 2.22-2.18 (1H, m, *H*-CH), 1.96-1.88 (2H, m, CH_2), 1.82-1.78 (2H, m, CH_2), 1.37-1.15 (4H, m, CH_2-CH_2).
^{13}C NMR (125 MHz, $CDCl_3$): δ 210.3 (C=O), 170.0 (-N-C=O), 141.7 (C-1'), 133.6 (C-4'), 129.7 (2C) (C-2' and 6'), 129.1 (2C) (C-3' and 5'), 56.3 (C-1), 43.0 (CH_2), 37.2 (=N-Me), 29.4, 28.2, 28.0, 24.4 (CH_2).
FAB HRMS (acetone-NBA): calcd for $C_{15}H_{19}ClNO_2$ 280.1104 (M+H); found 280.1101.

_N_-(2-Methylphenyl)-_N_-methyl-2-oxocycloheptanecarboxamide (1x)



Yield (45%); R_f = 0.34 (1:8:1 AcOEt-hexane-acetone).
Yellow oil.
IR ($CHCl_3$): ν = 1705 (-C=O), 1645 (-N-C=O).
^{1}H NMR (500 Hz, $CDCl_3$) (1:1 rotamer mixture): δ 7.24-7.14 (6H, m, arom H), 7.07 (1H, d, J = 7.0 Hz, arom H), 7.01 (3H, d, J = 7.5 Hz, arom H), 3.19-3.16 (2H, m), 3.12 (3H, s, =N-Me), 3.09 (3H, s, =N-Me), 2.63 (1H, m, J = 12.5, 3.5 Hz), 2.42 (1H, m, J =15.0, 12.0, 3.0 Hz), 2.21 (3H, s, Me), 2.15 (3H, s, Me), 2.08-2.04 (1H, m), 2.02-1.97 (1H, m), 1.94-1.62 (10H, m), 1.31-1.08 (5H, m), 0.98 (1H, q, J = 12.5 Hz).
^{13}C NMR (125 MHz, $CDCl_3$) (1:1 rotamer mixture): δ 210.3, 210.1 (C=O), 170.8, 170.4 (-N-C=O), 141.7 (C-1'), 136.6, 135.1 (C-2'), 131.8, 131.1, 129.0, 128.6, 128.5, 127.8, 127.4, 127.0 (arom C), 56.7, 56.3 (C-1), 43.0 (CH_2), 35.9, 35.7 (=N-Me), 35.9, 35.7, 29.6, 29.3, 28.6, 28.4, 28.3, 27.9, 25.0, 24.2 (CH_2), 17.4, 17.3 (Me).
FAB HRMS (acetone-NBA): calcd for $C_{16}H_{22}NO_2$ 260.1651 (M+H); found 260.1649.

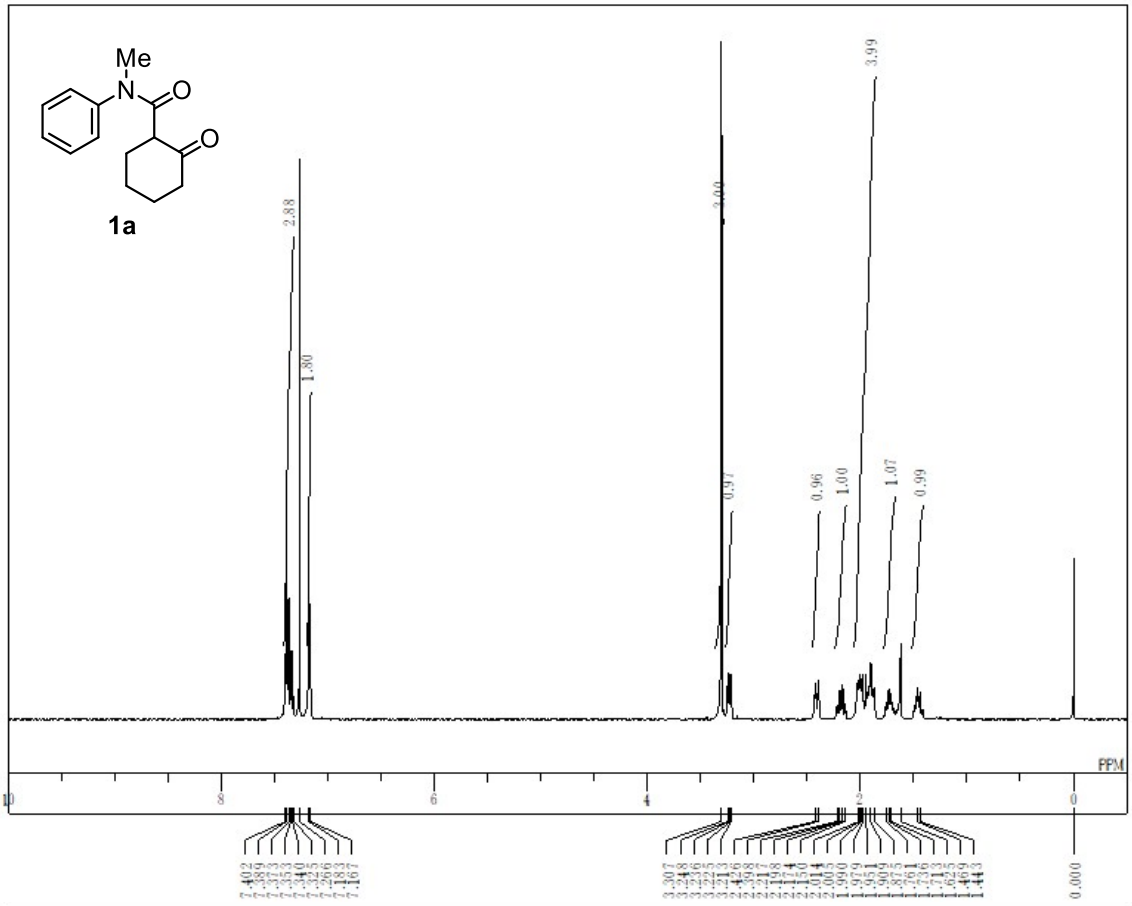
_N_-(2-Chlorophenyl)-_N_-methyl-2-oxocycloheptanecarboxamide (1y)



Yield (42%); R_f = 0.35 (1:8:1 AcOEt-hexane-acetone).
Yellow oil.
IR ($CHCl_3$): ν = 1705 (-C=O), 1651 (-N-C=O).
^{1}H NMR (500 Hz, $CDCl_3$): δ 7.53 (1H, d, J = 9.0 Hz, arom H), 7.38-7.34 (3H, m, arom H), 3.25 (1H, q, J = 10.0, 4.0 Hz, *H*-C-1), 3.21 (3H, s, =N-Me), 2.55 (1H, t, J = 12.8 Hz, *H*-CH), 2.13-2.03 (2H, m, CH_2), 1.95-1.86 (2H, m, CH_2), 1.80-1.74 (2H, m, CH_2), 1.37-1.18 (3H, m,CH_2-CH_2).
^{13}C NMR (125 MHz, $CDCl_3$): δ 210.2 (C=O), 170.0 (-N-C=O), 140.2 (C-1'), 132.6, 130.9, 130.2, 129.7, 128.1 (arom C), 56.6 (C-1), 42.9 (CH_2), 35.7 (=N-Me), 29.3, 28.1, 28.1, 24.3 (CH_2).
FAB HRMS (acetone-NBA): calcd for $C_{16}H_{22}NO_2$ 280.1104 (M+H); found 280.1105.

References

(1) Hurst, E. T.; Gorman, R.; Drouhin, P.; Taylor, J. K. R. *Tetrahedron* **2018**, *74*, 6485-6496.
(2) Hayamizu, K.; Terada, N.; Hashizume, D.; Dodo, K.; Sodeoka, M. *Tetrahedron* **2015**, *71*, 6594-6601.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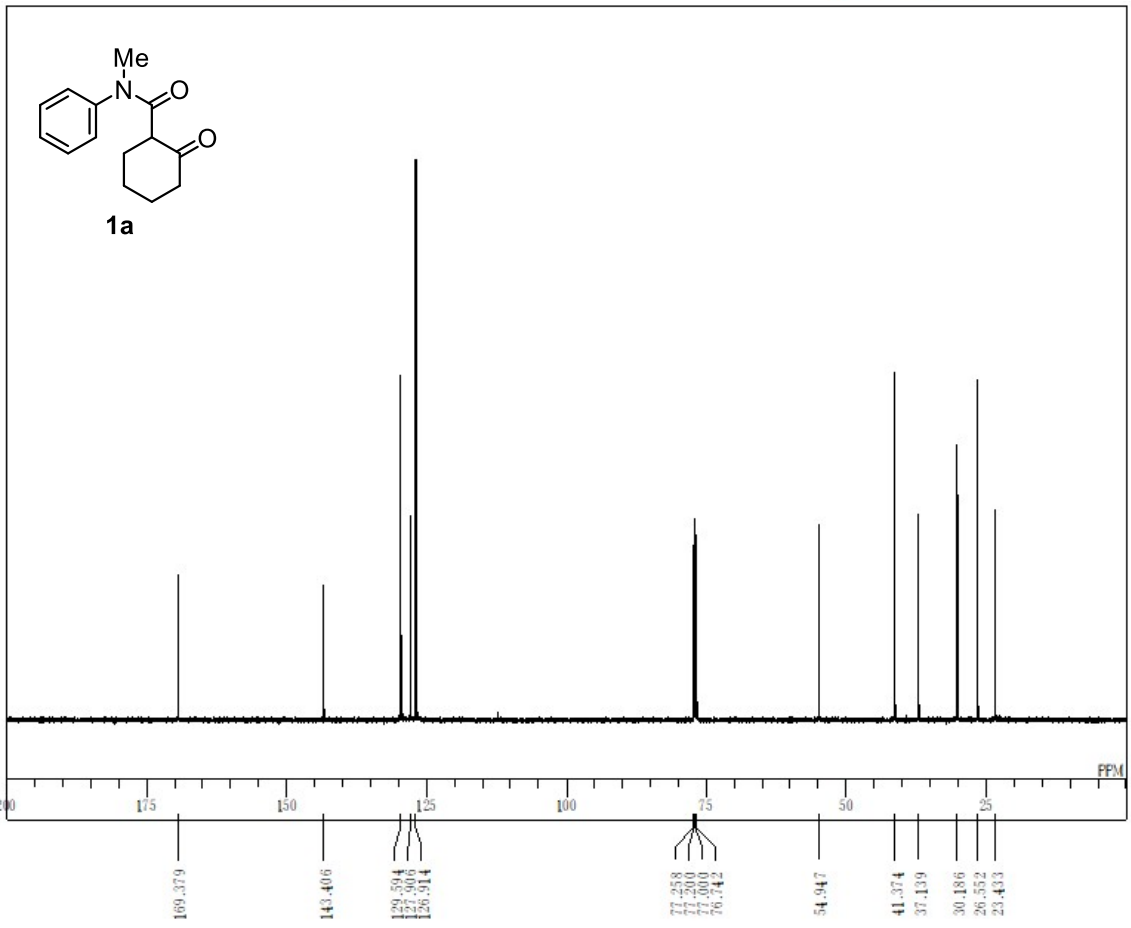
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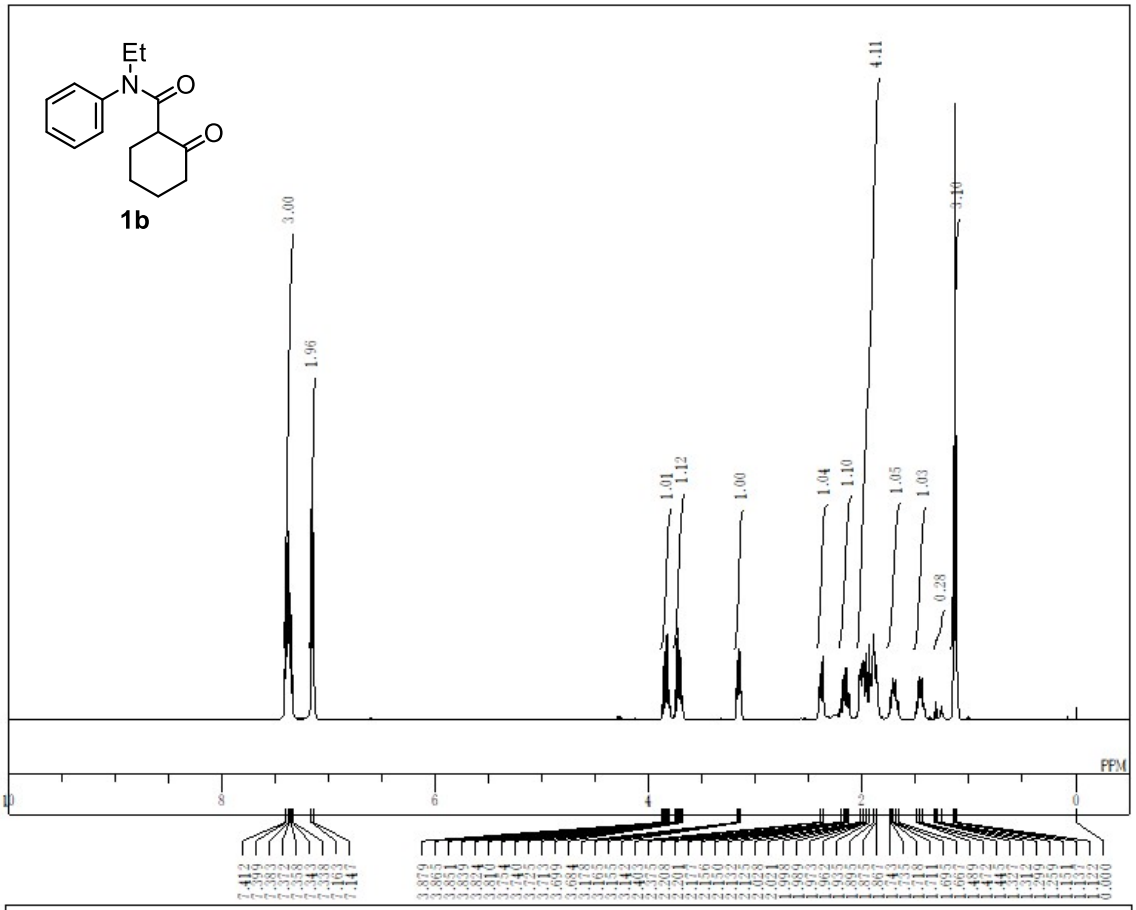
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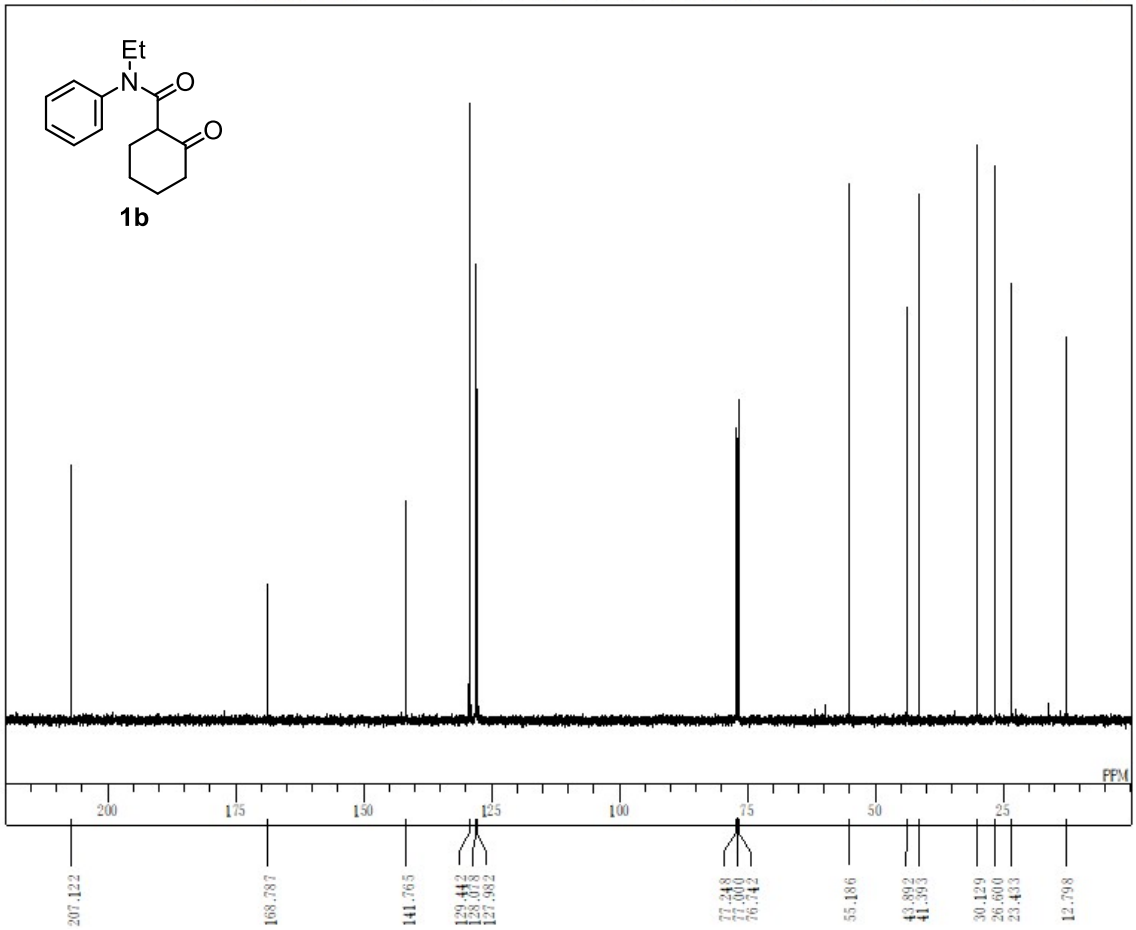
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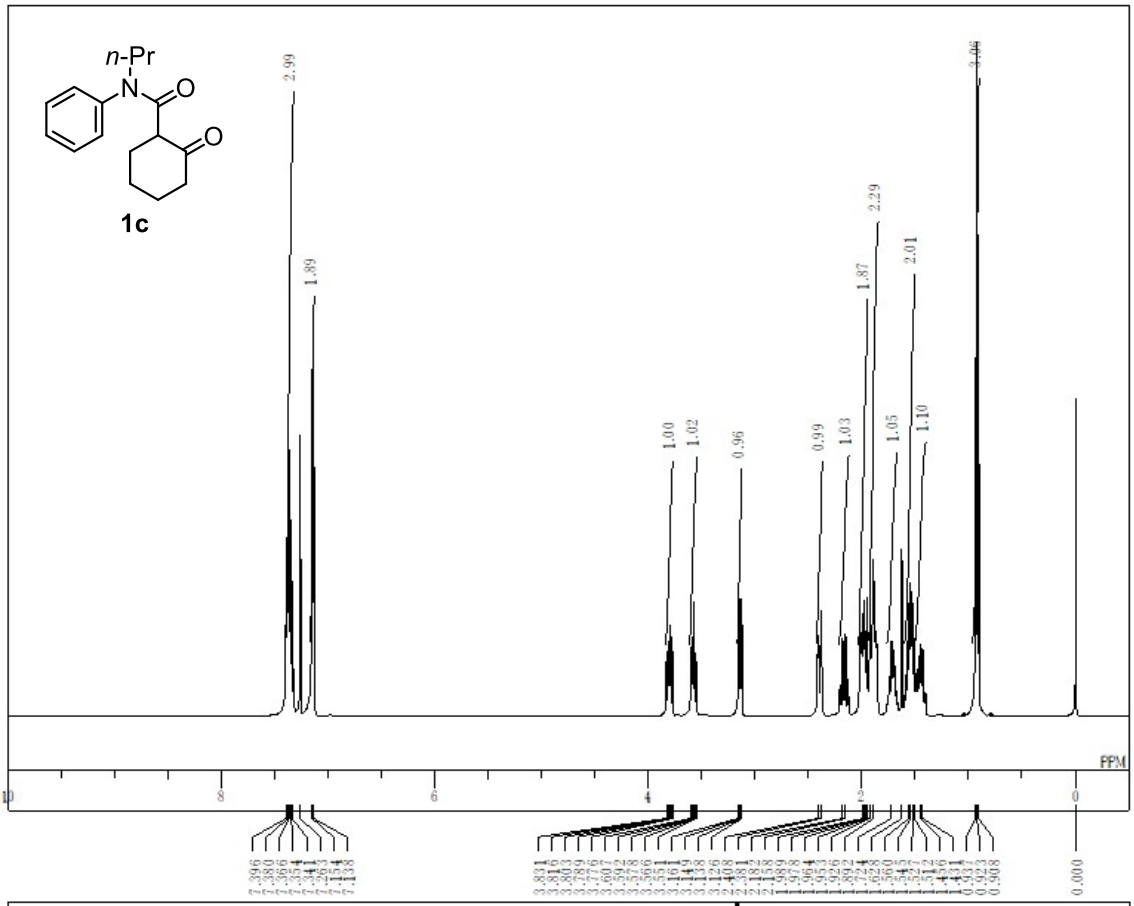
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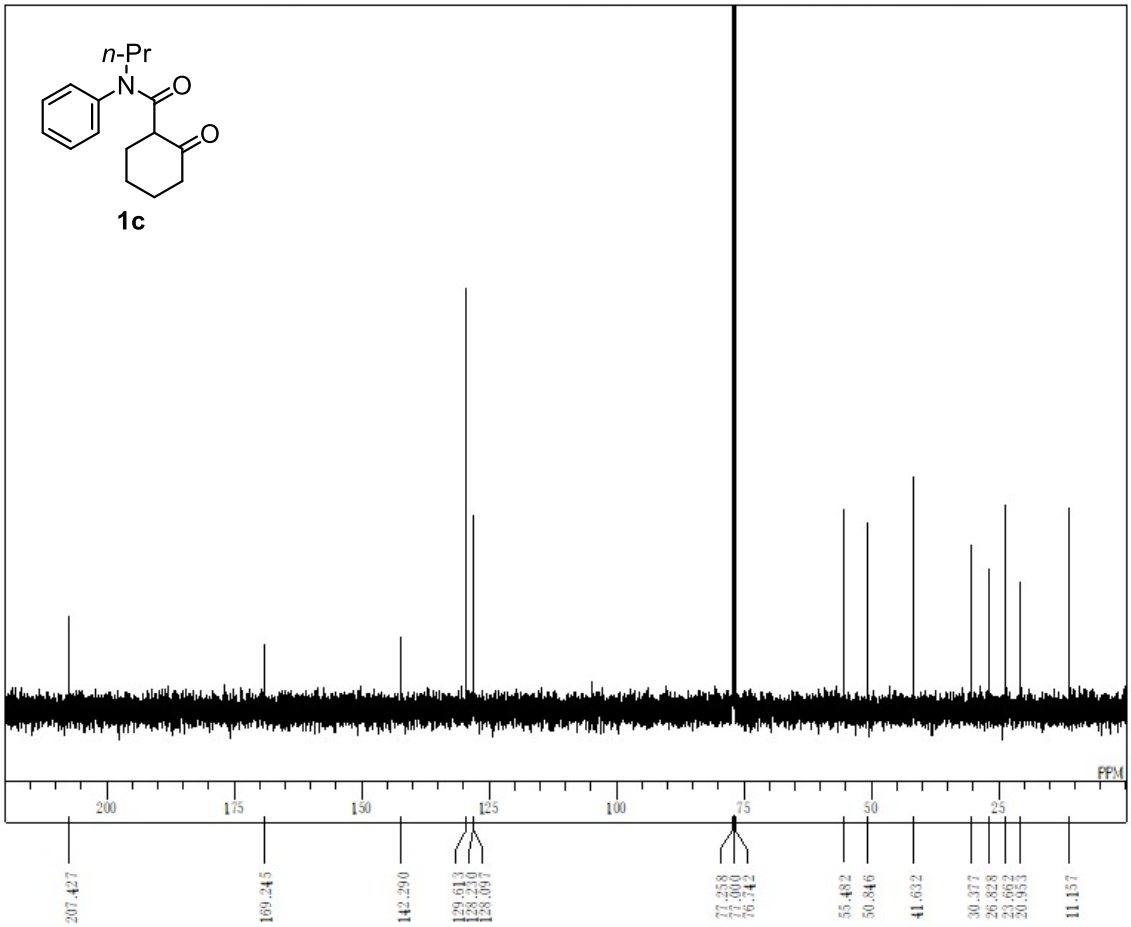
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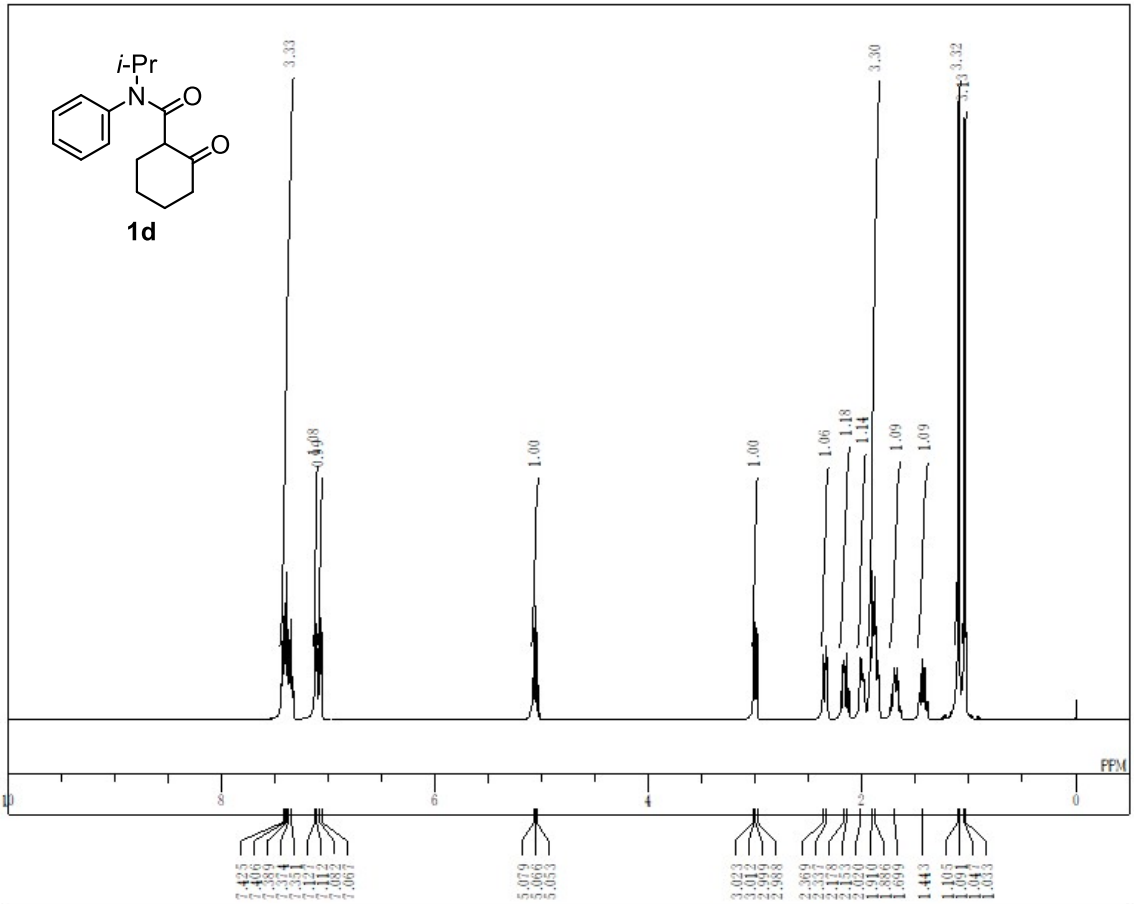
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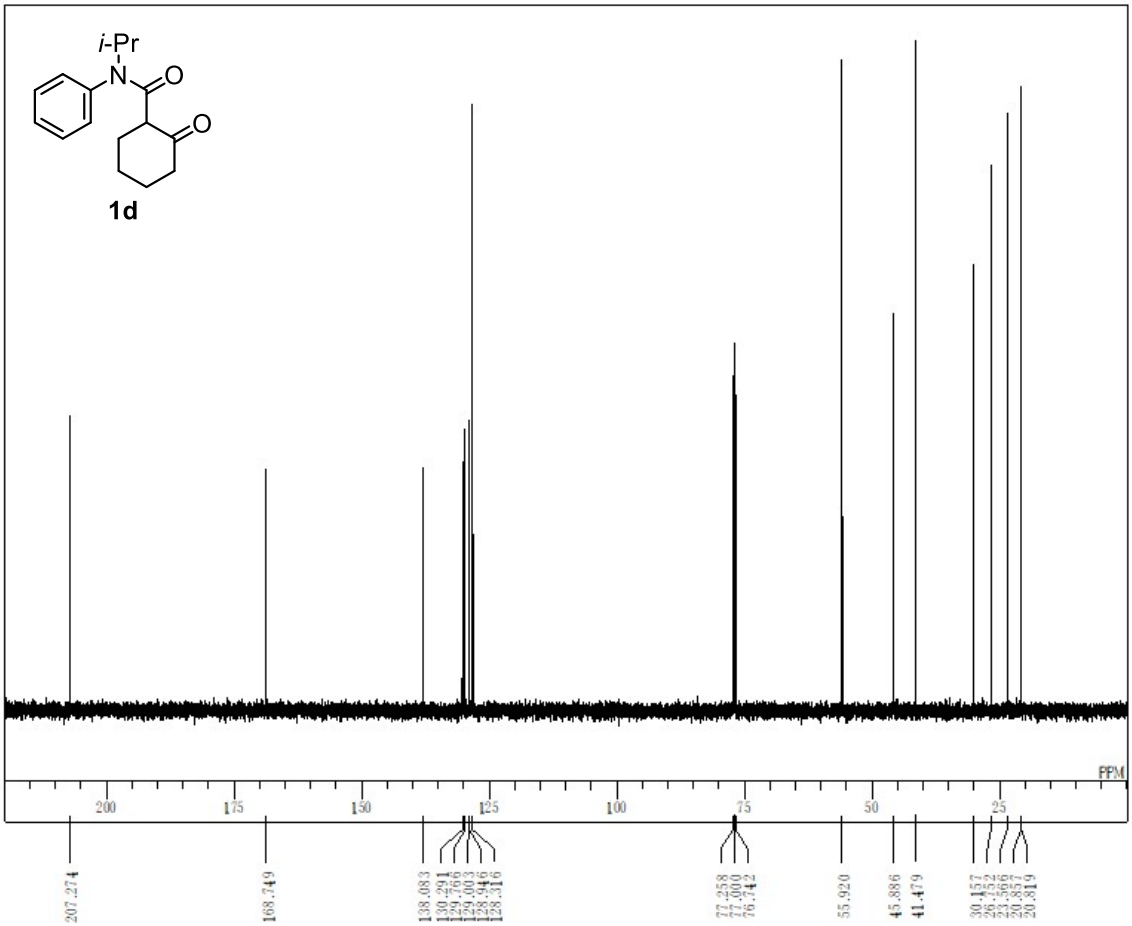
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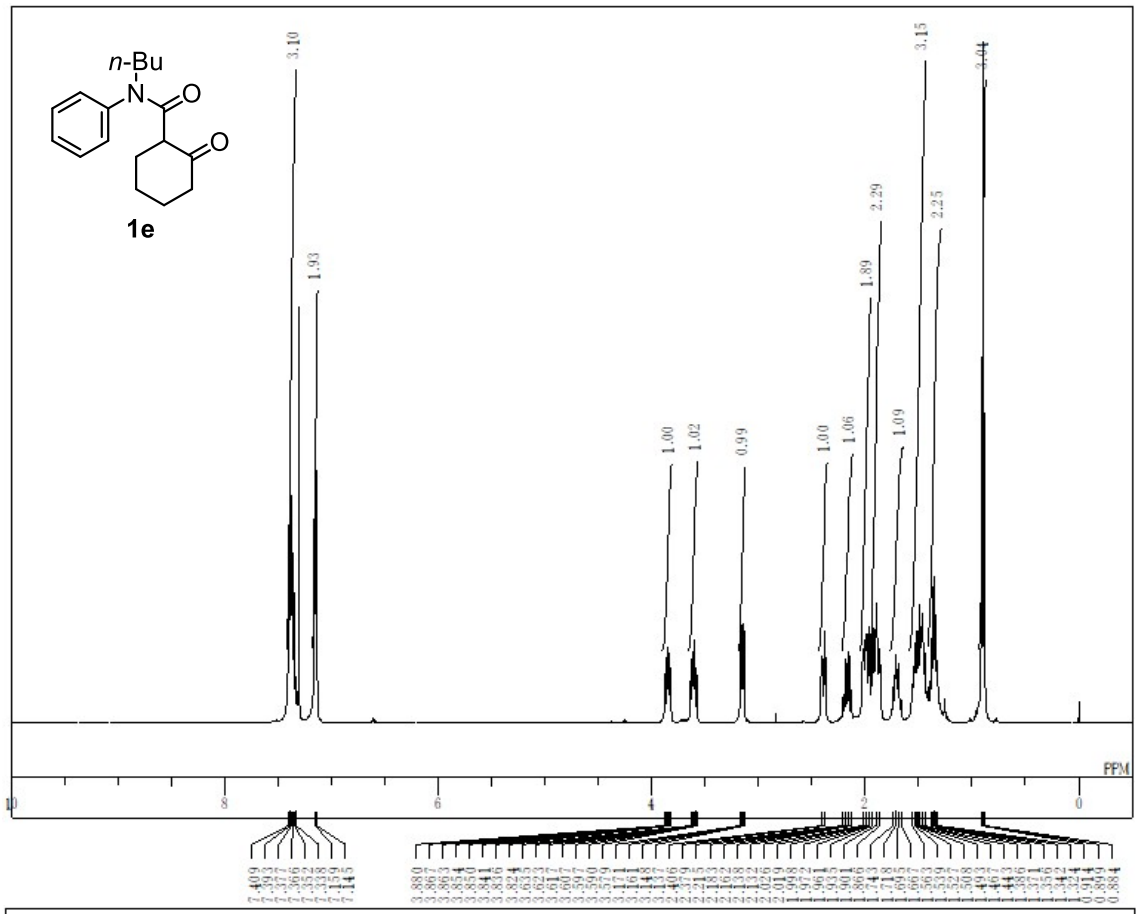
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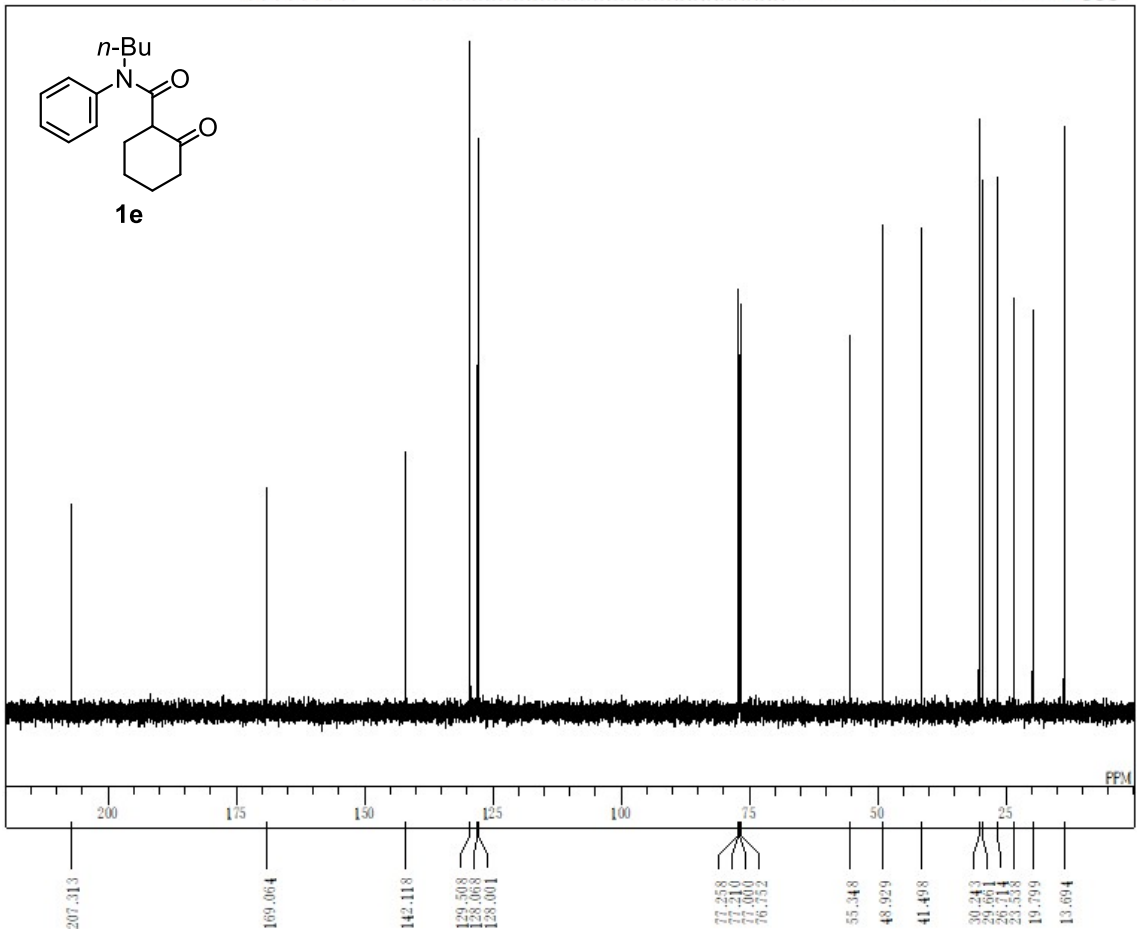
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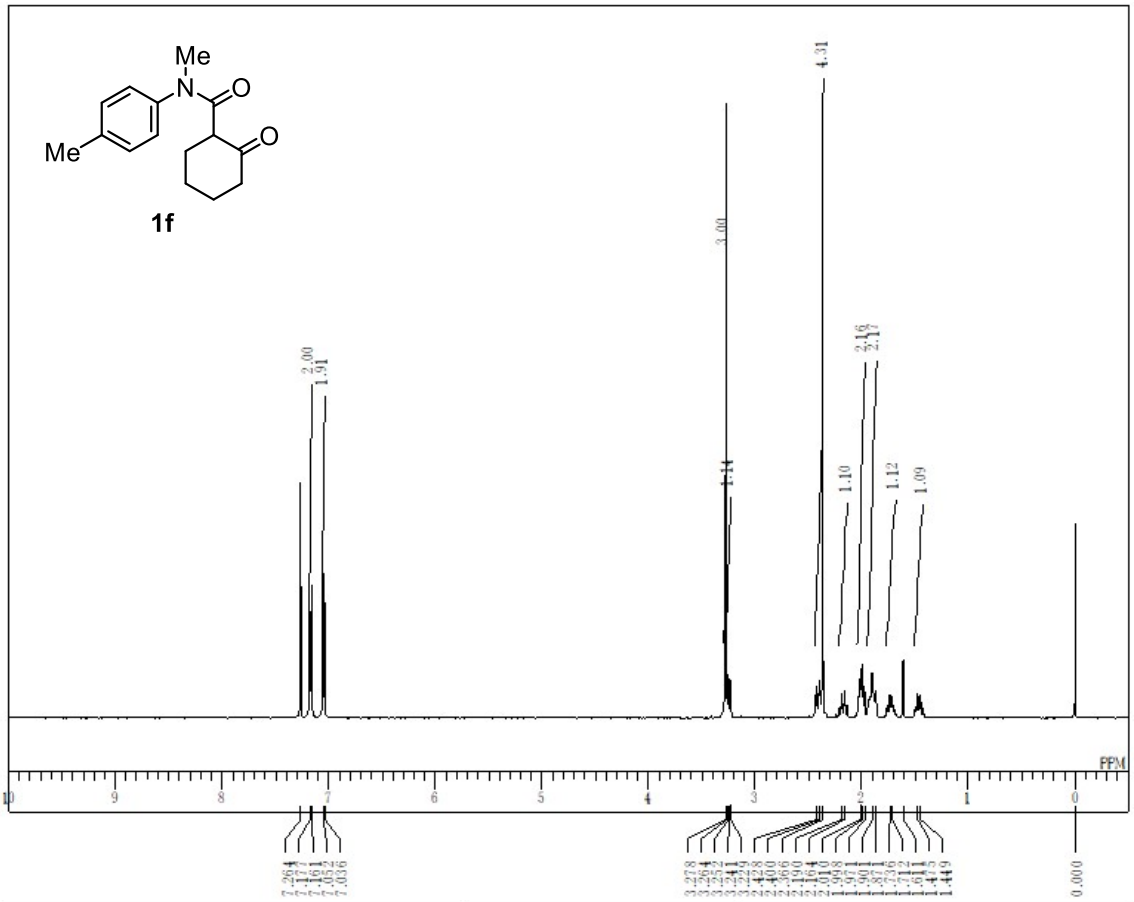
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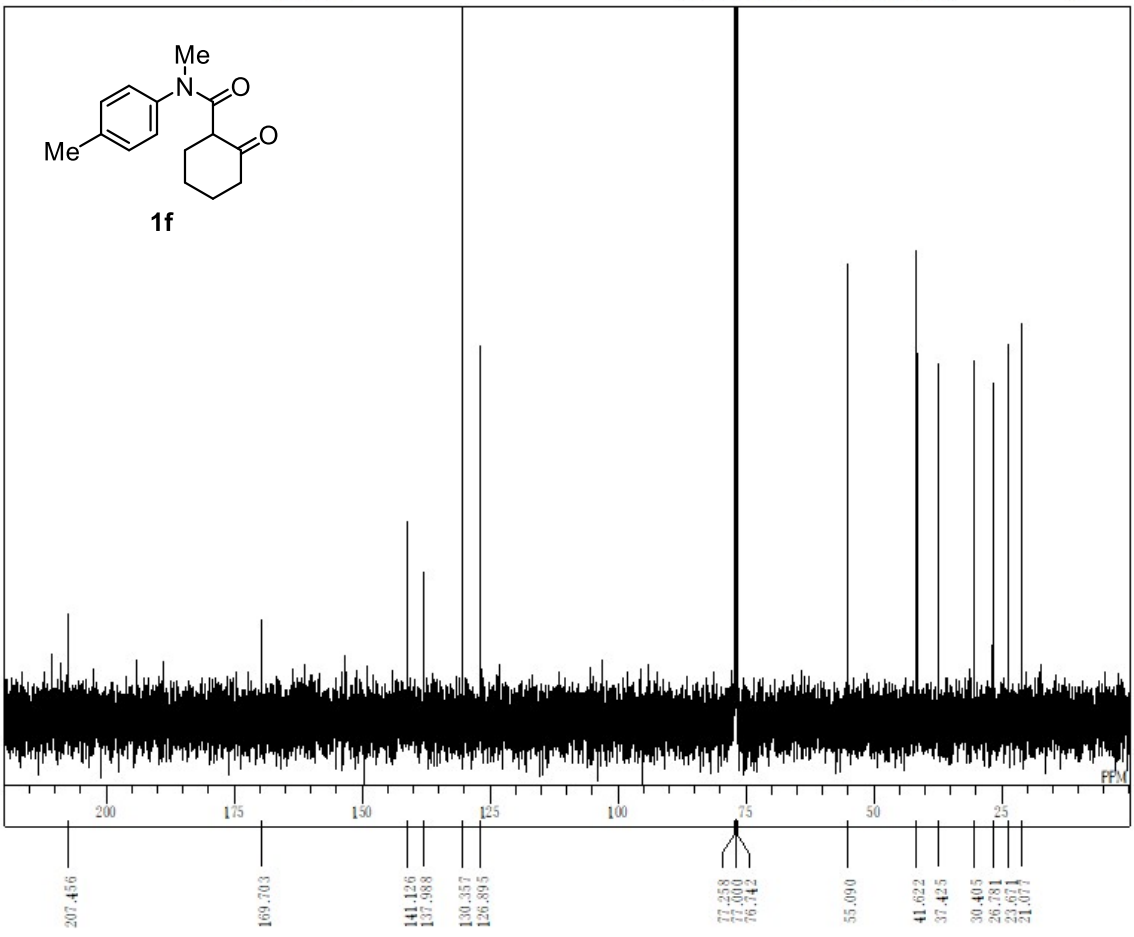
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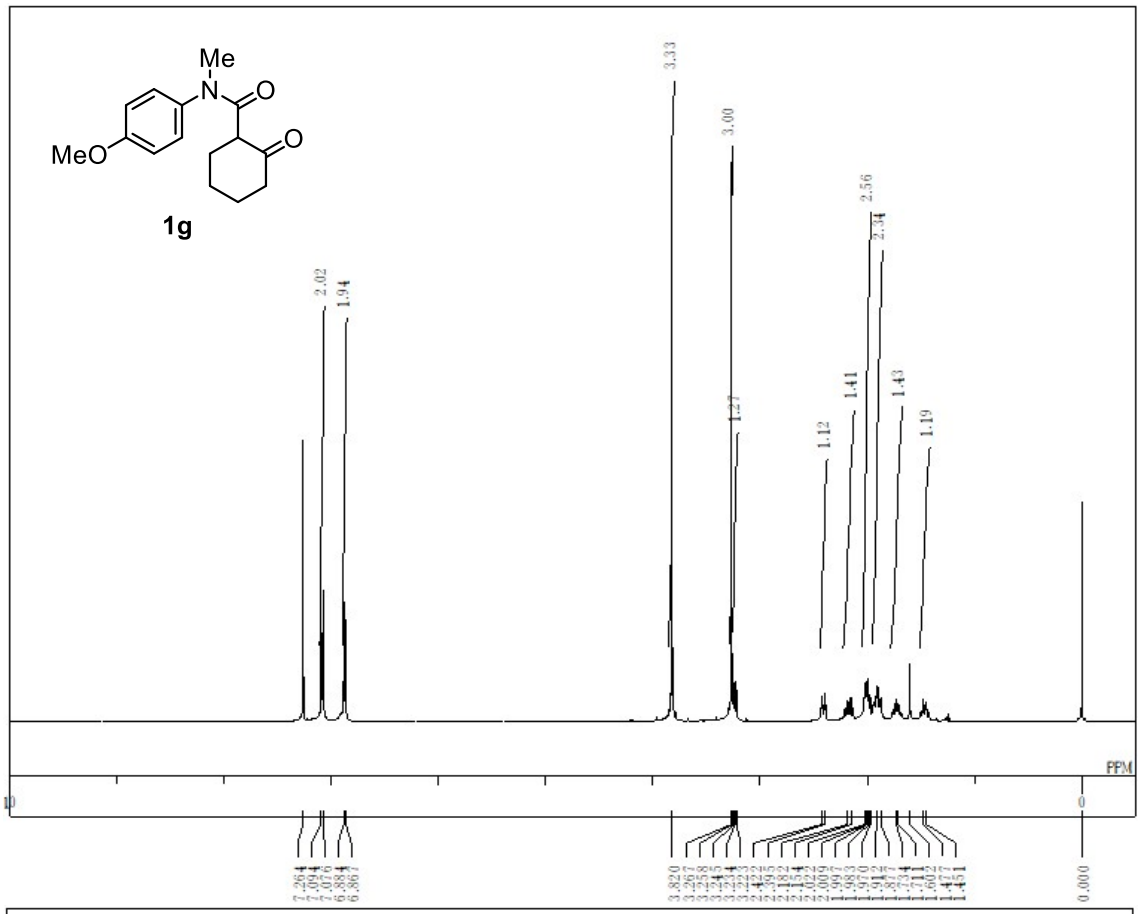
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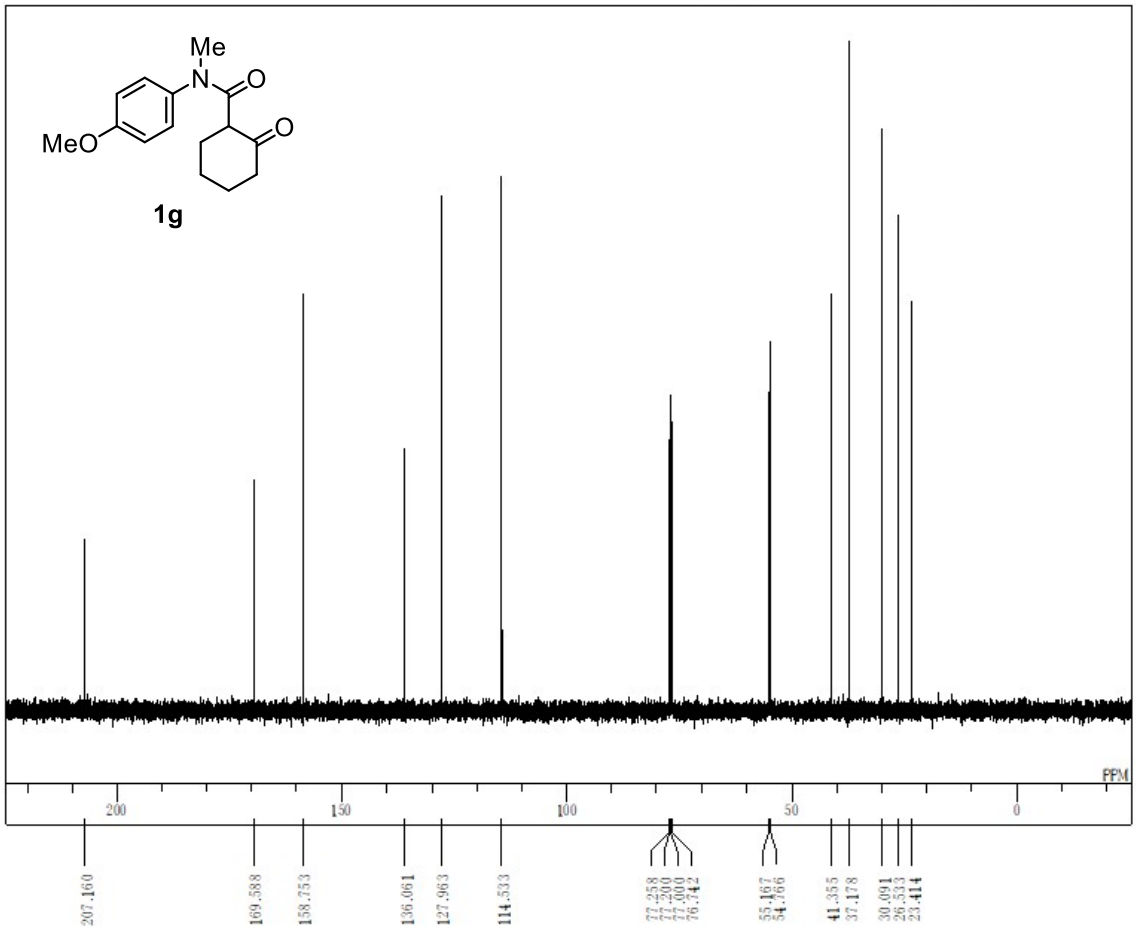
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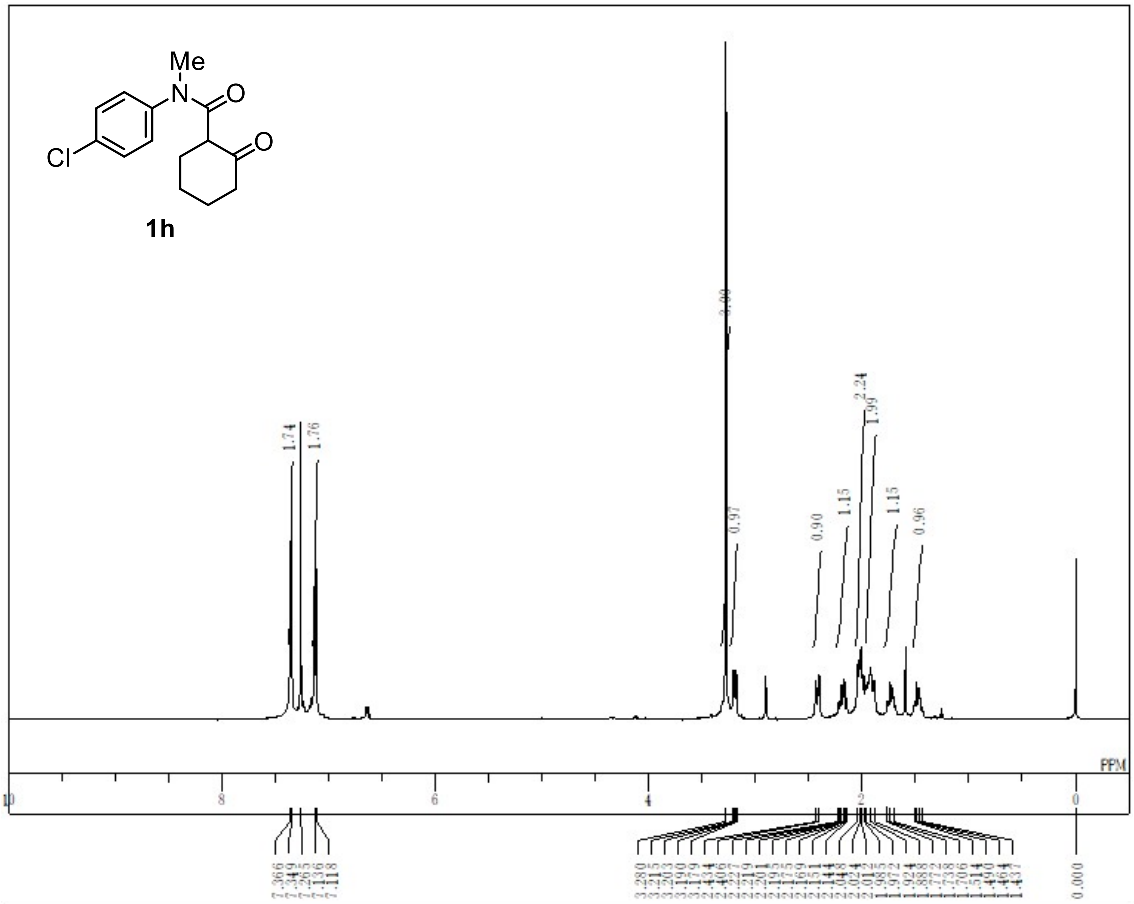
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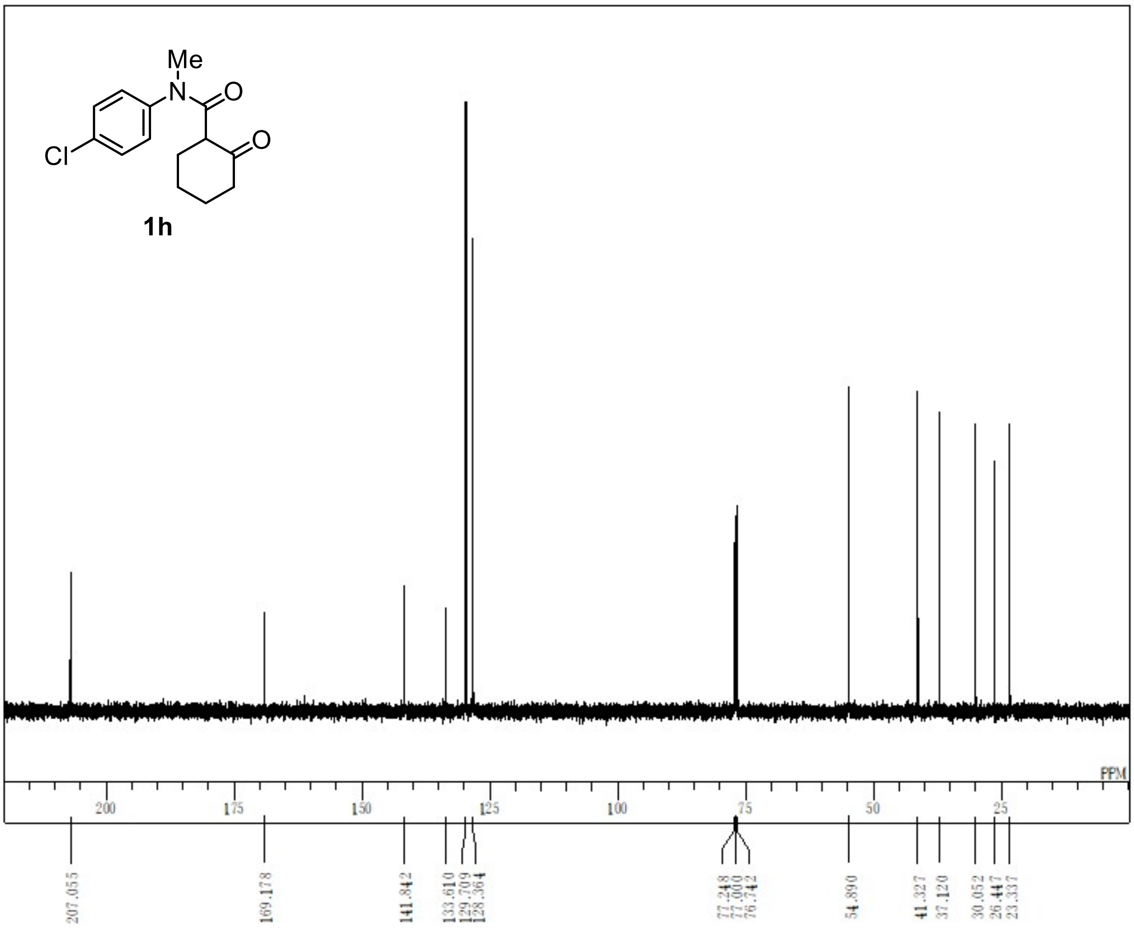
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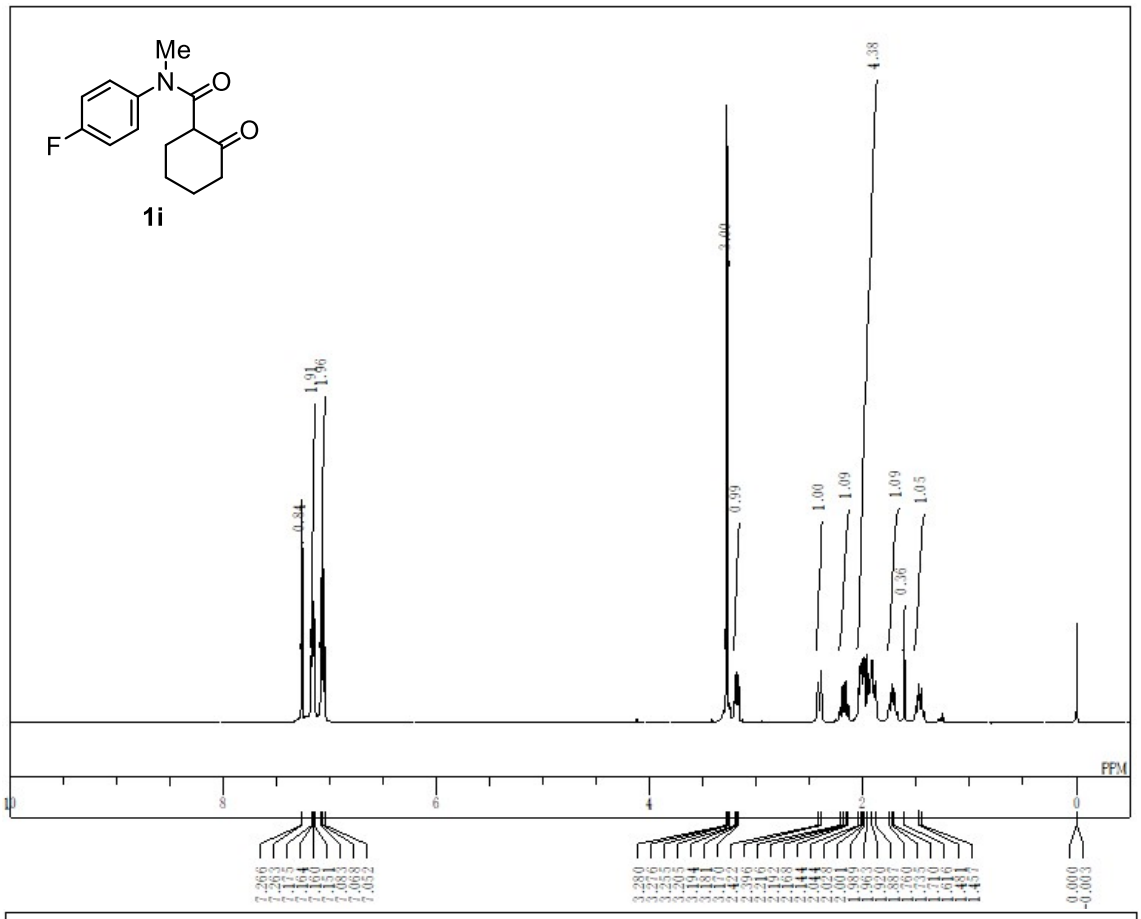
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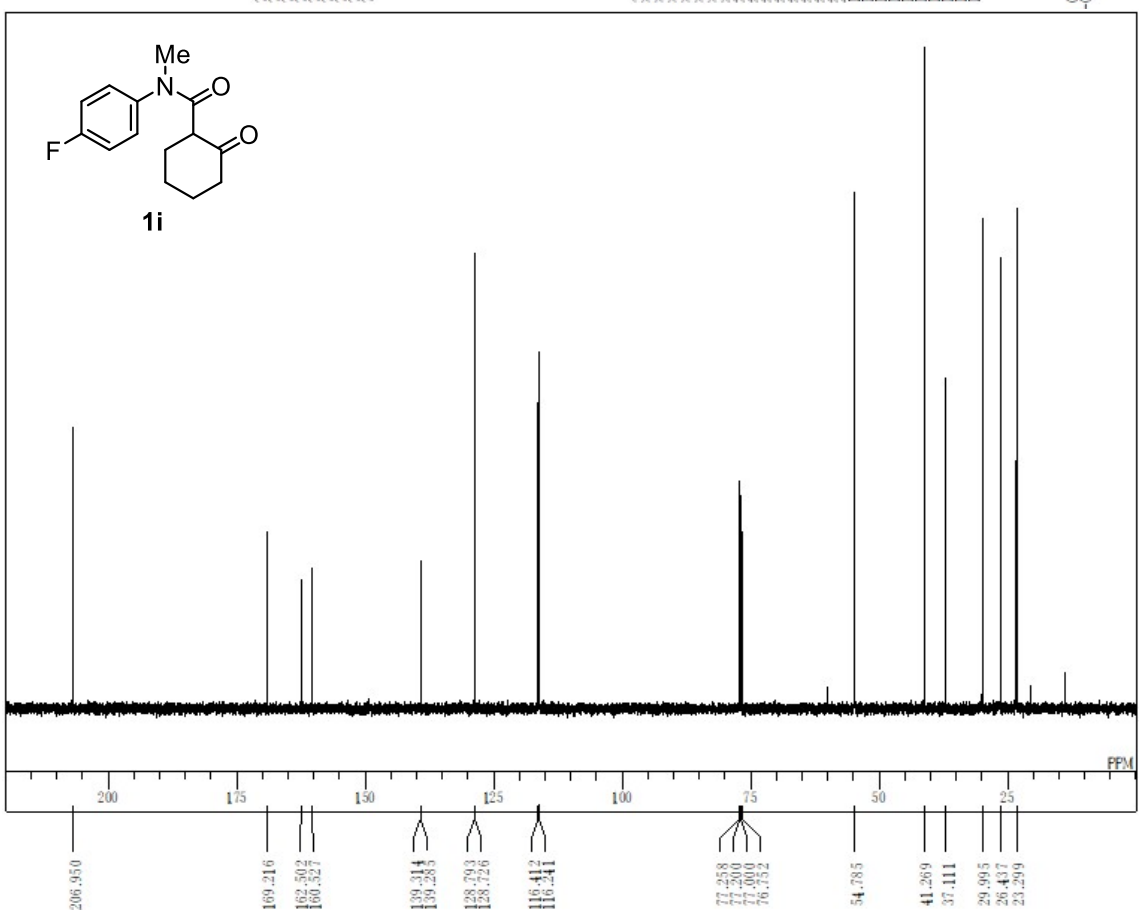
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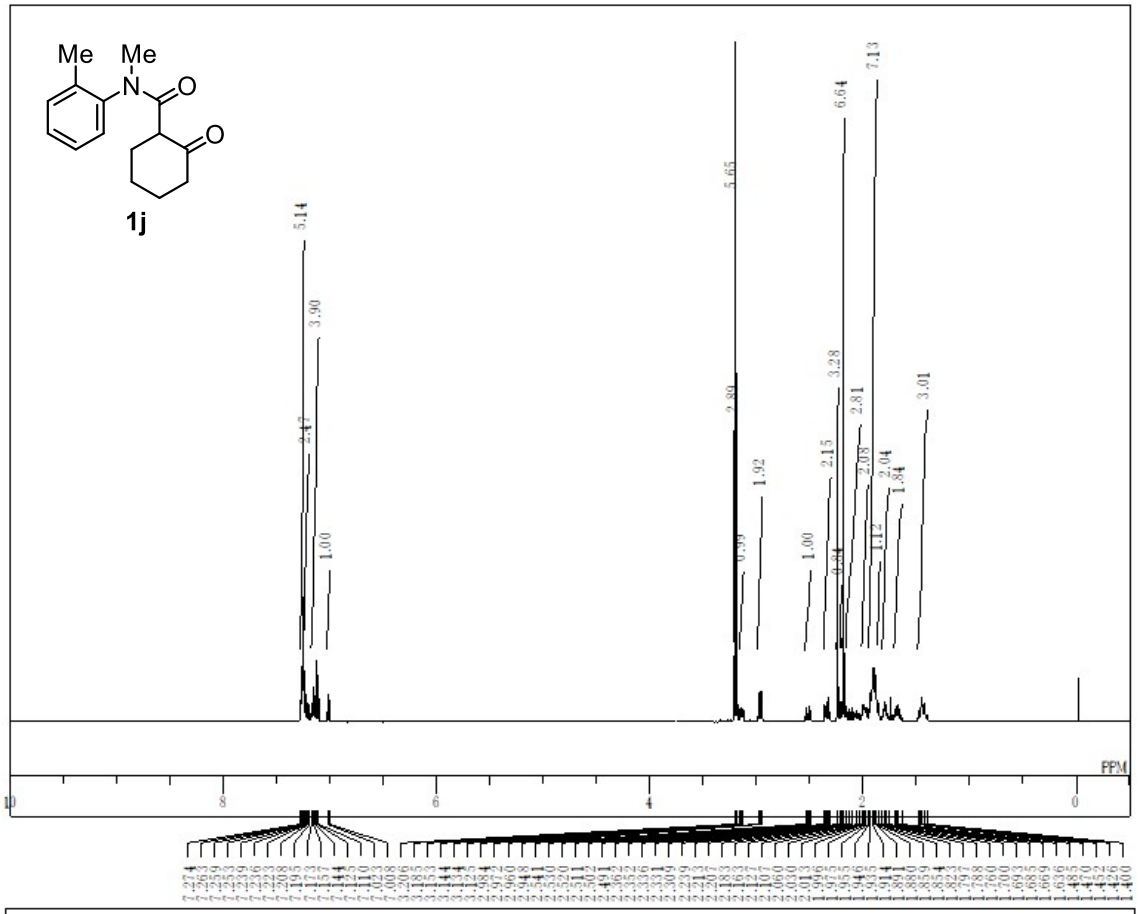
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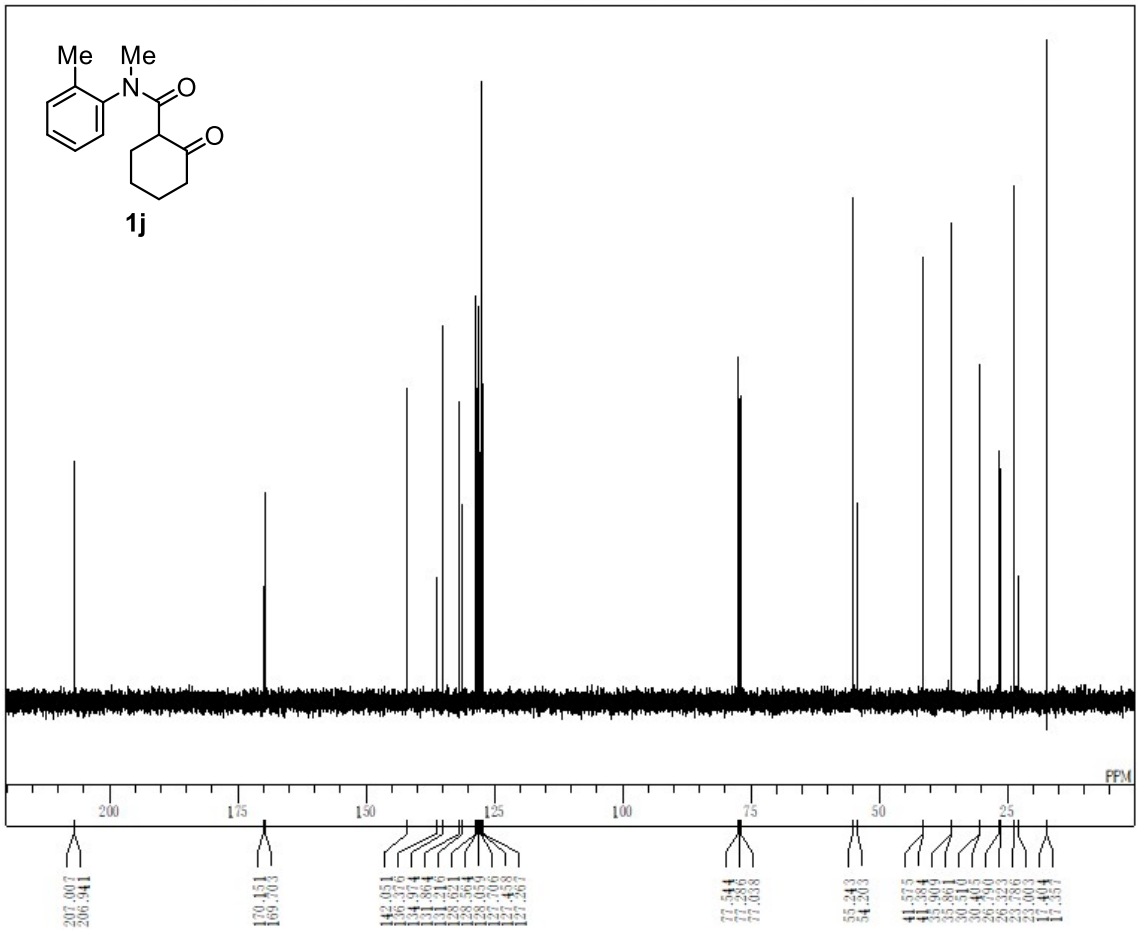
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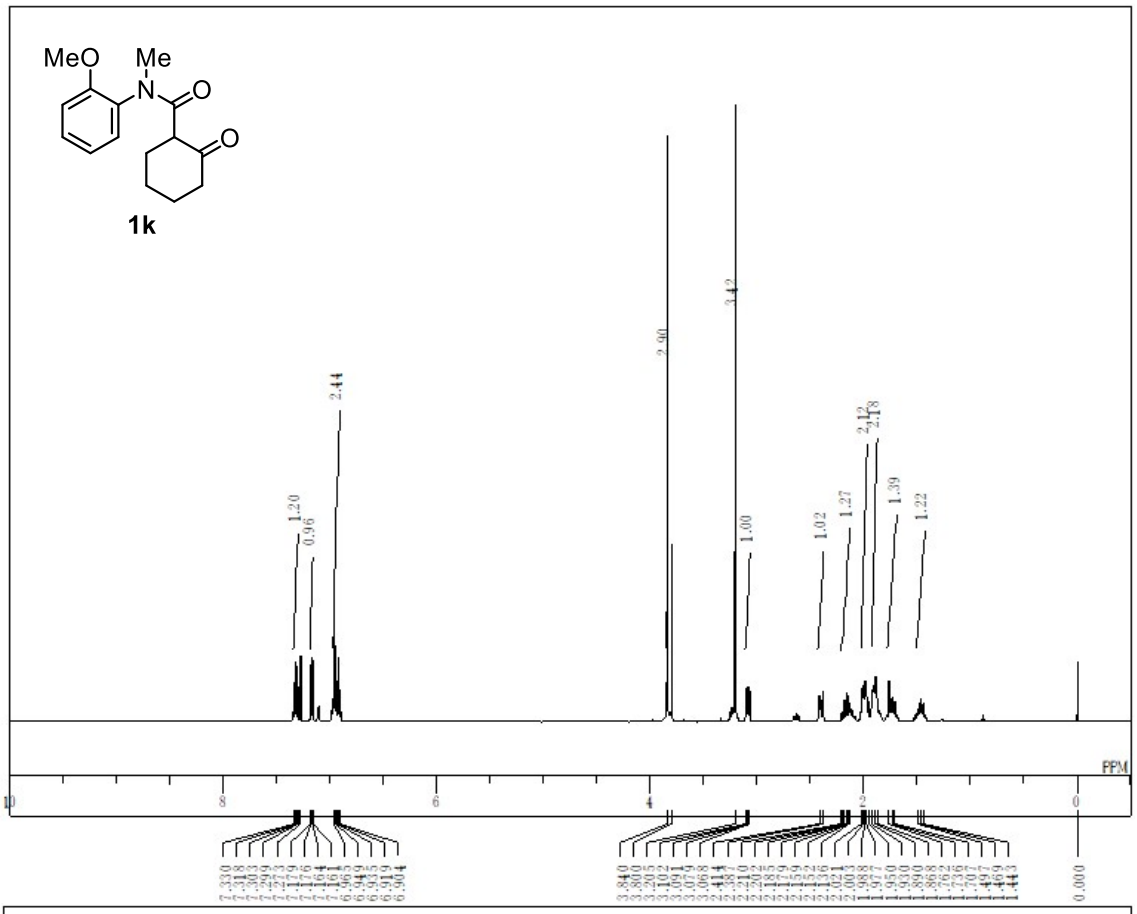
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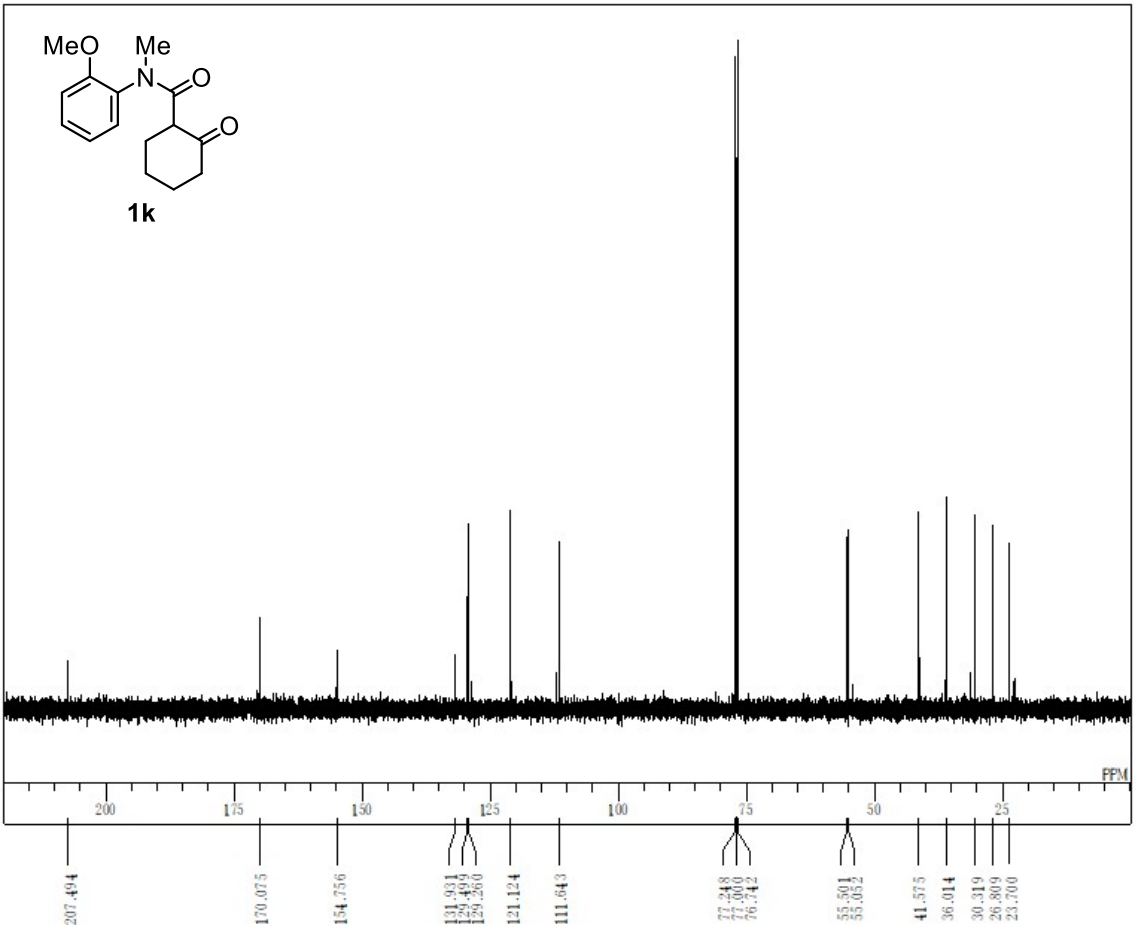
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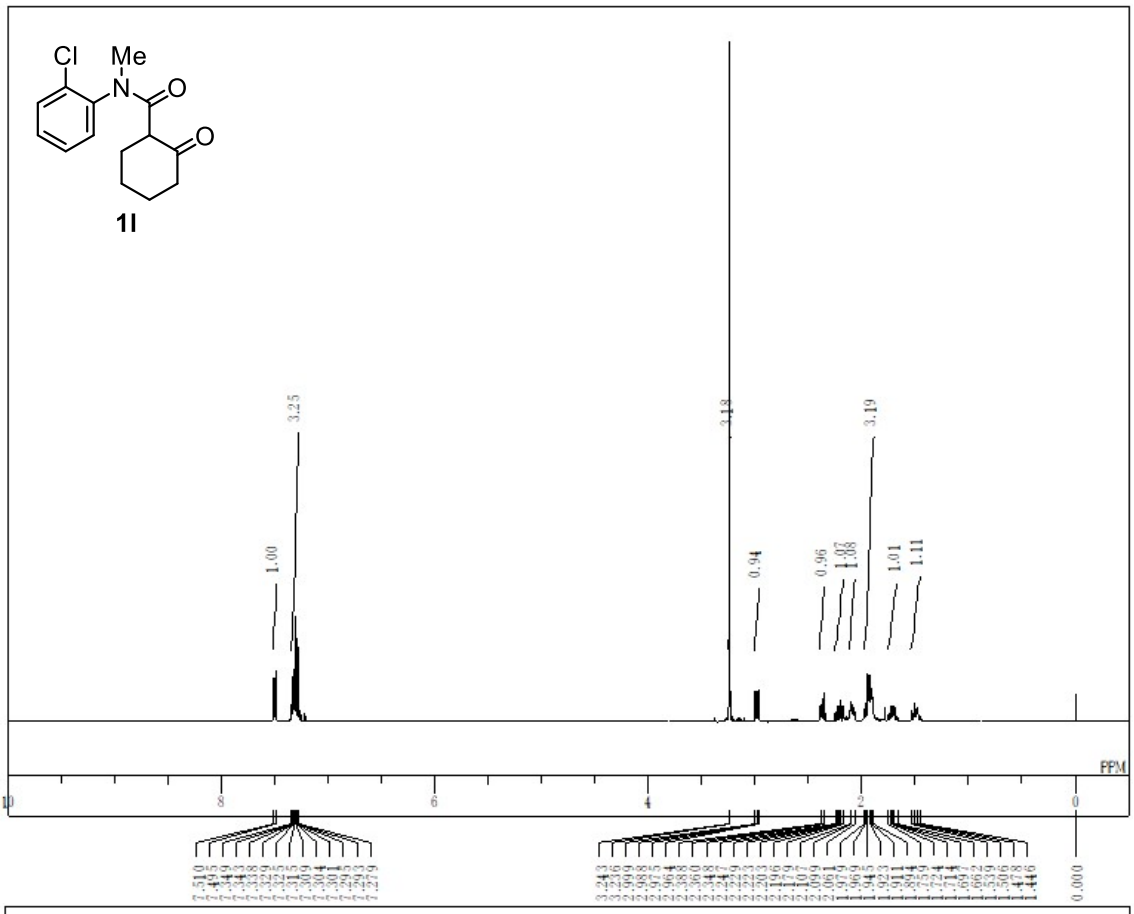
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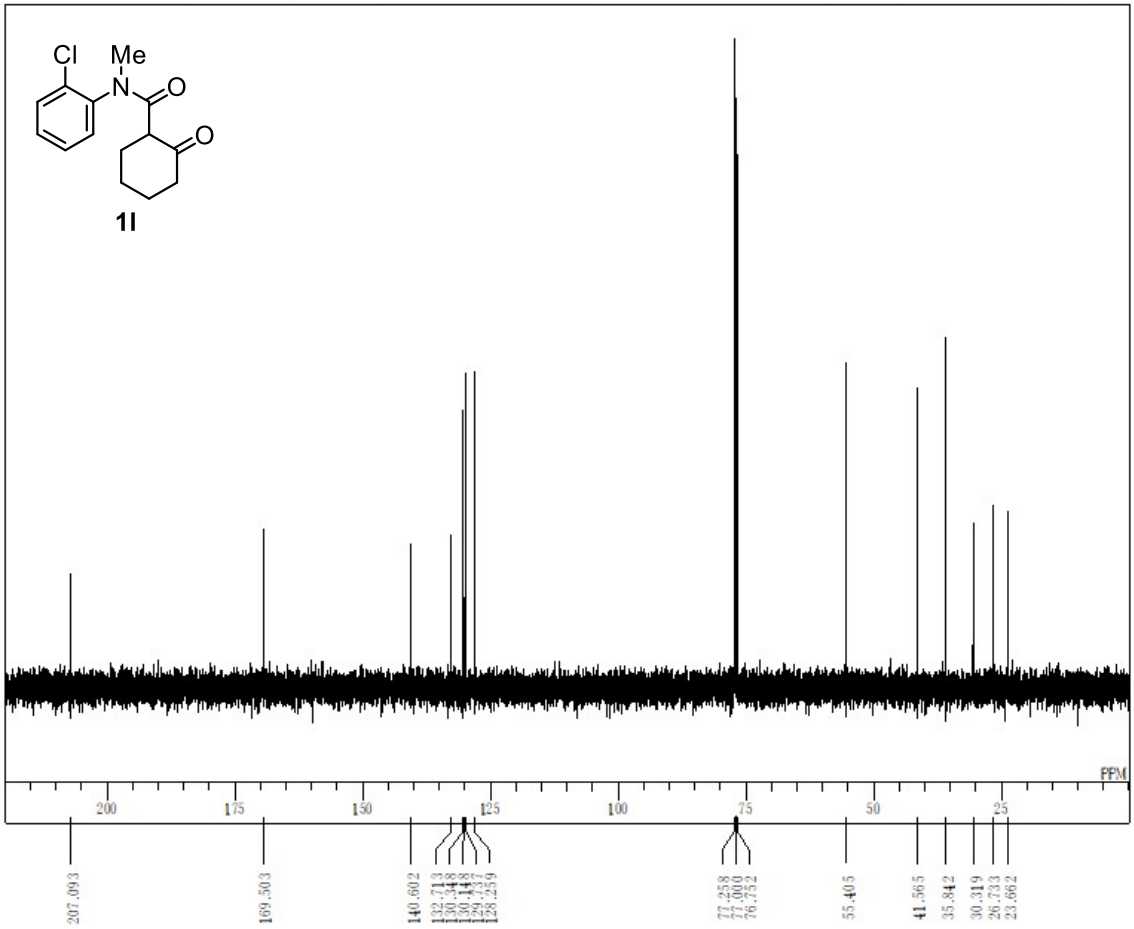
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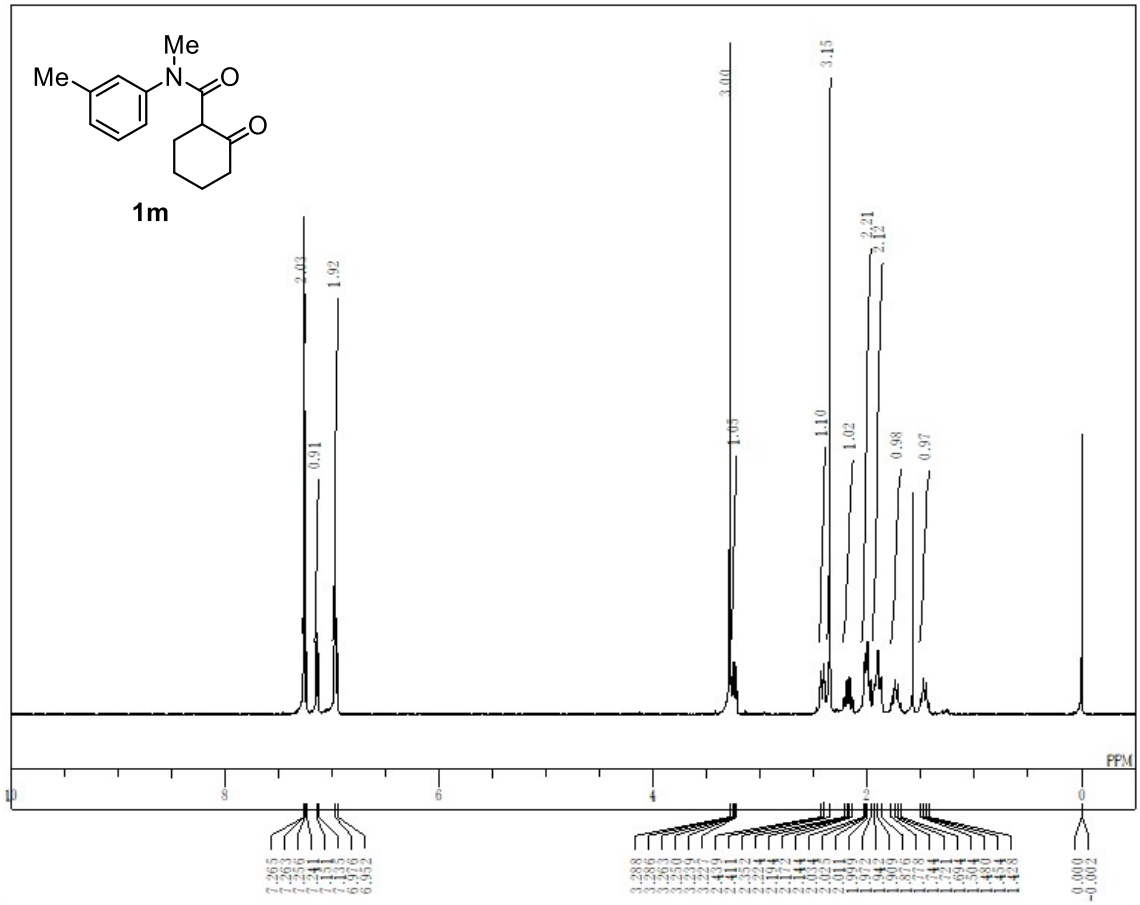
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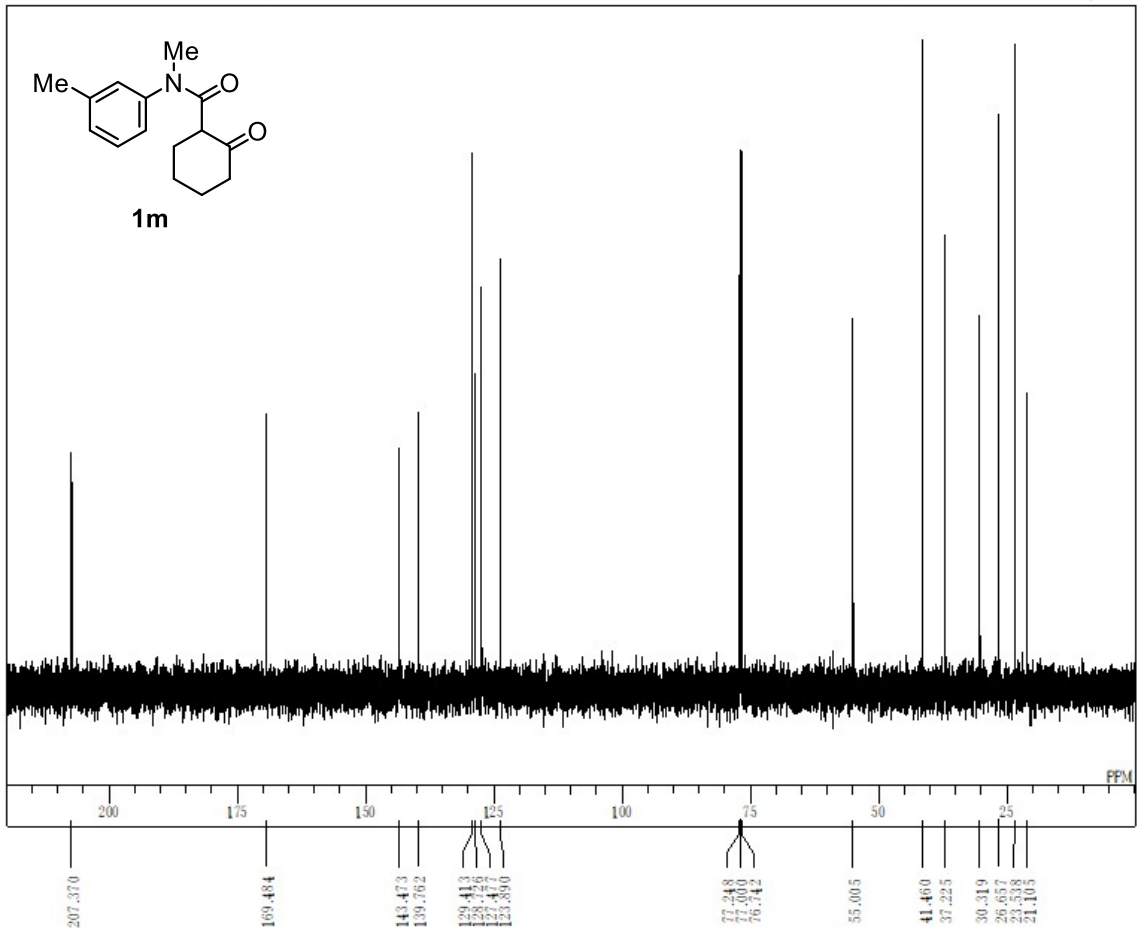
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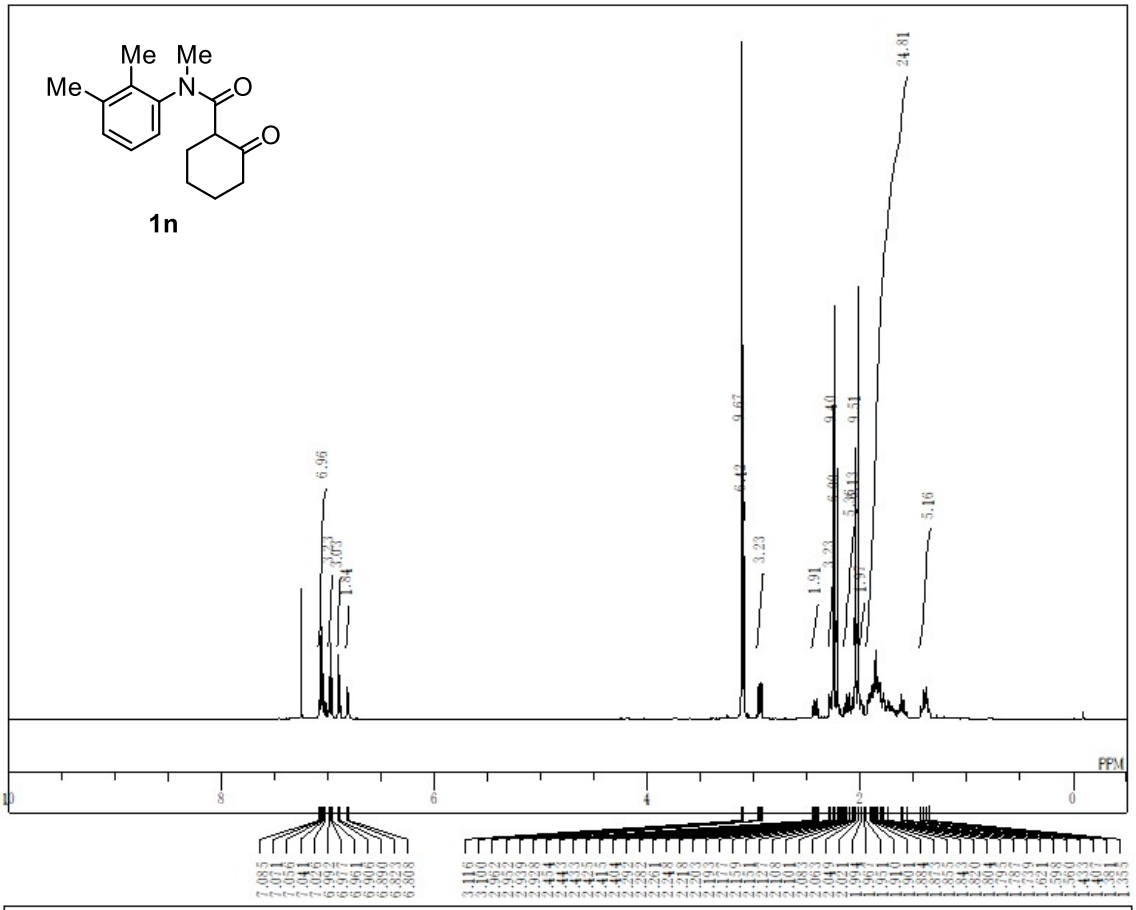
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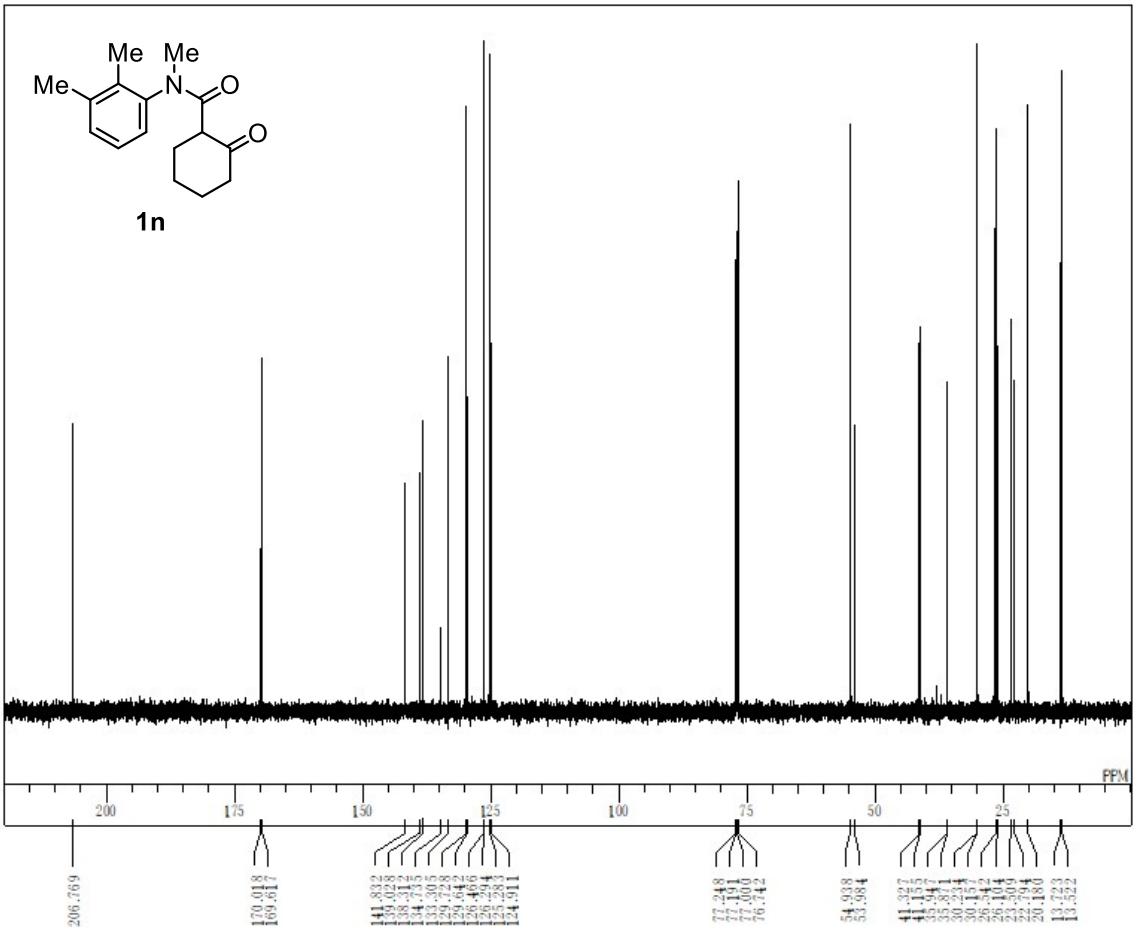
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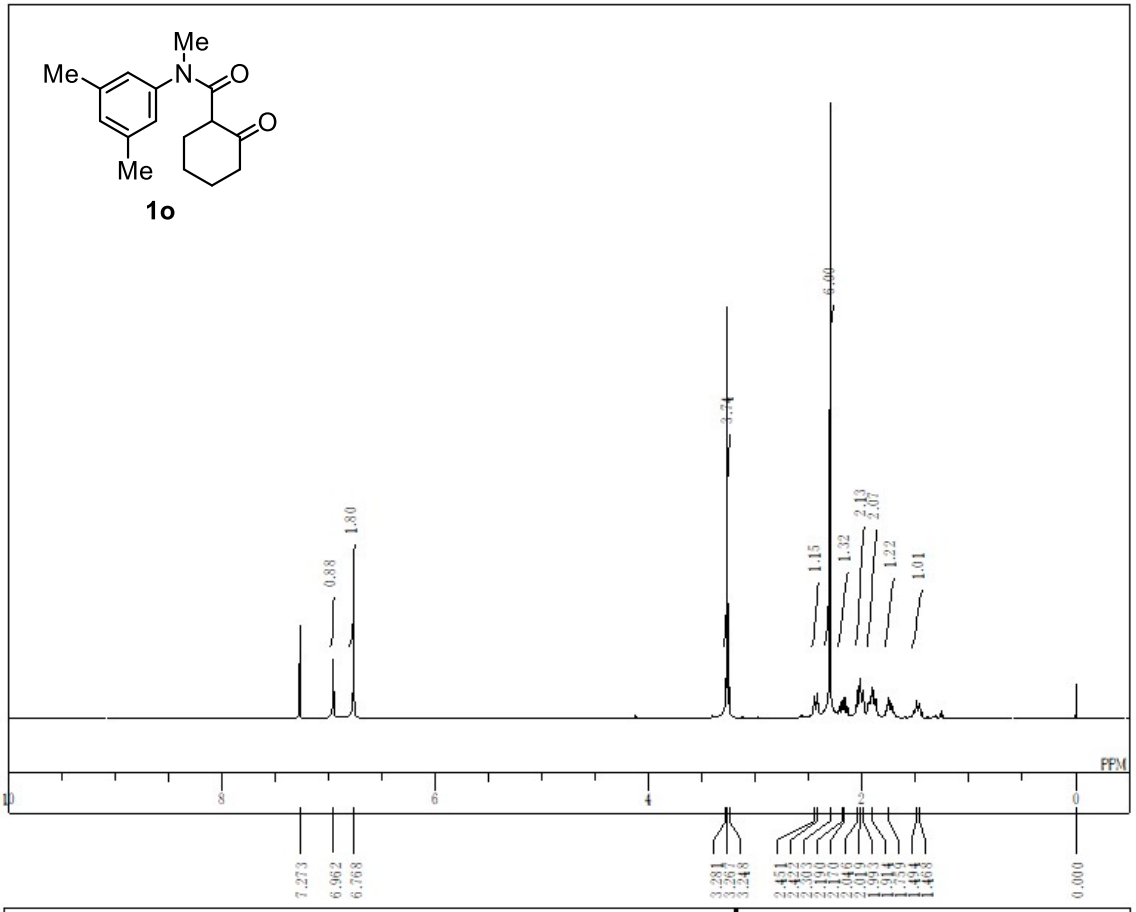
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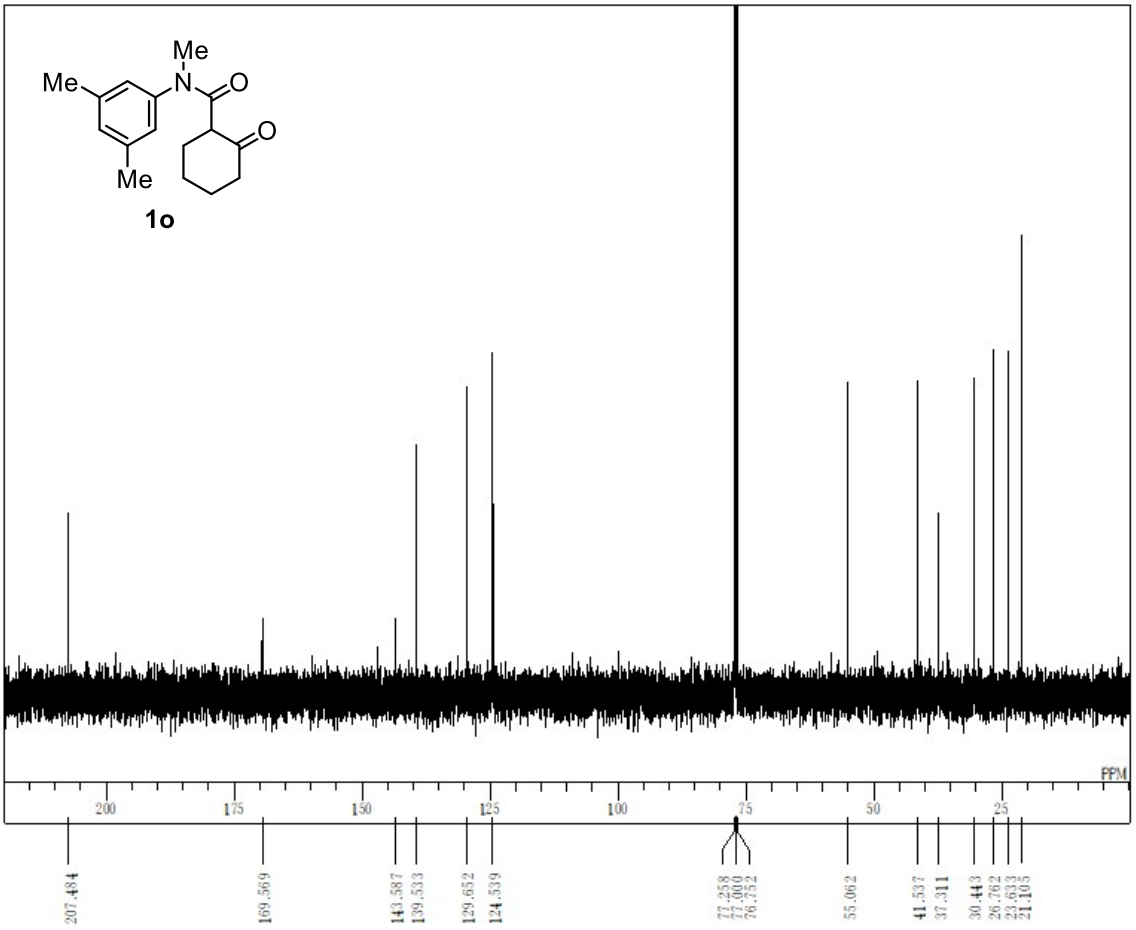
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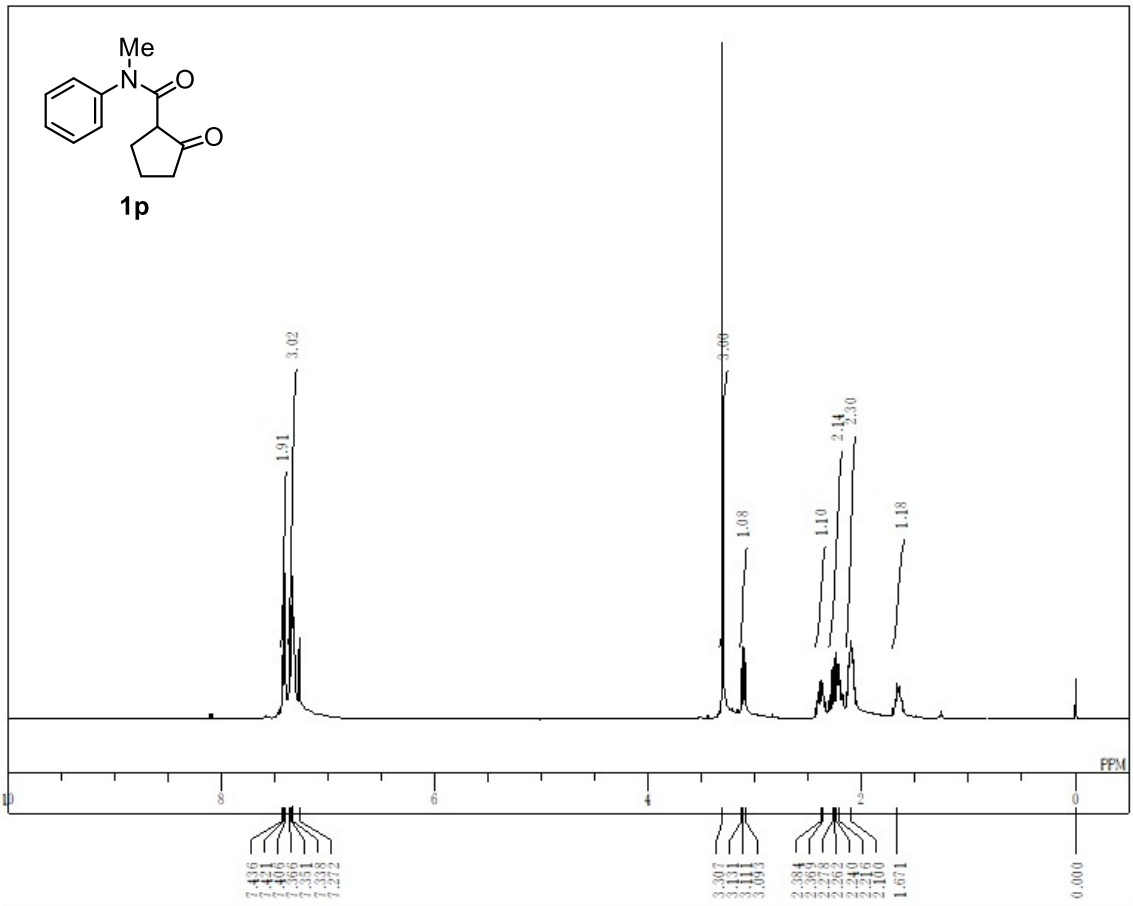
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8
6
4
2
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7.431
7.426
7.366
7.351
7.338
7.322
3.307
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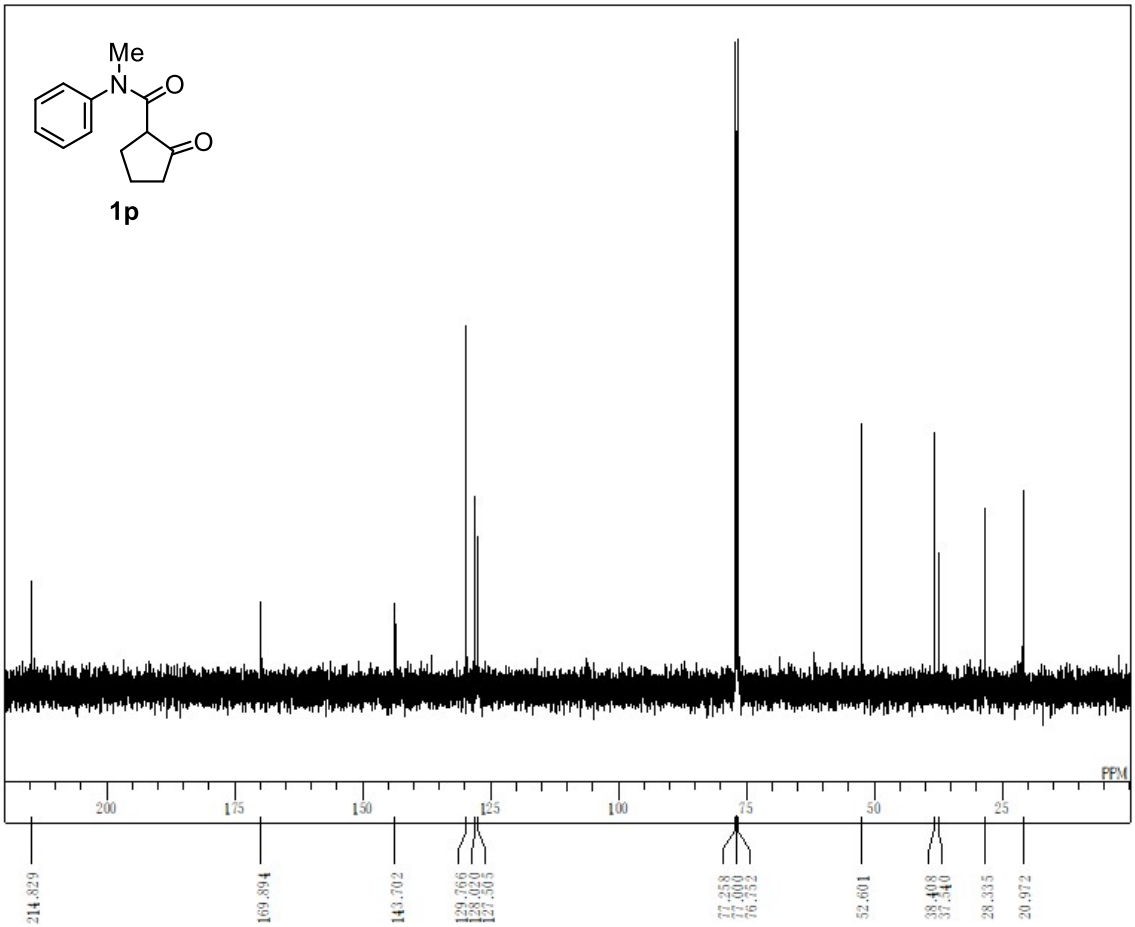
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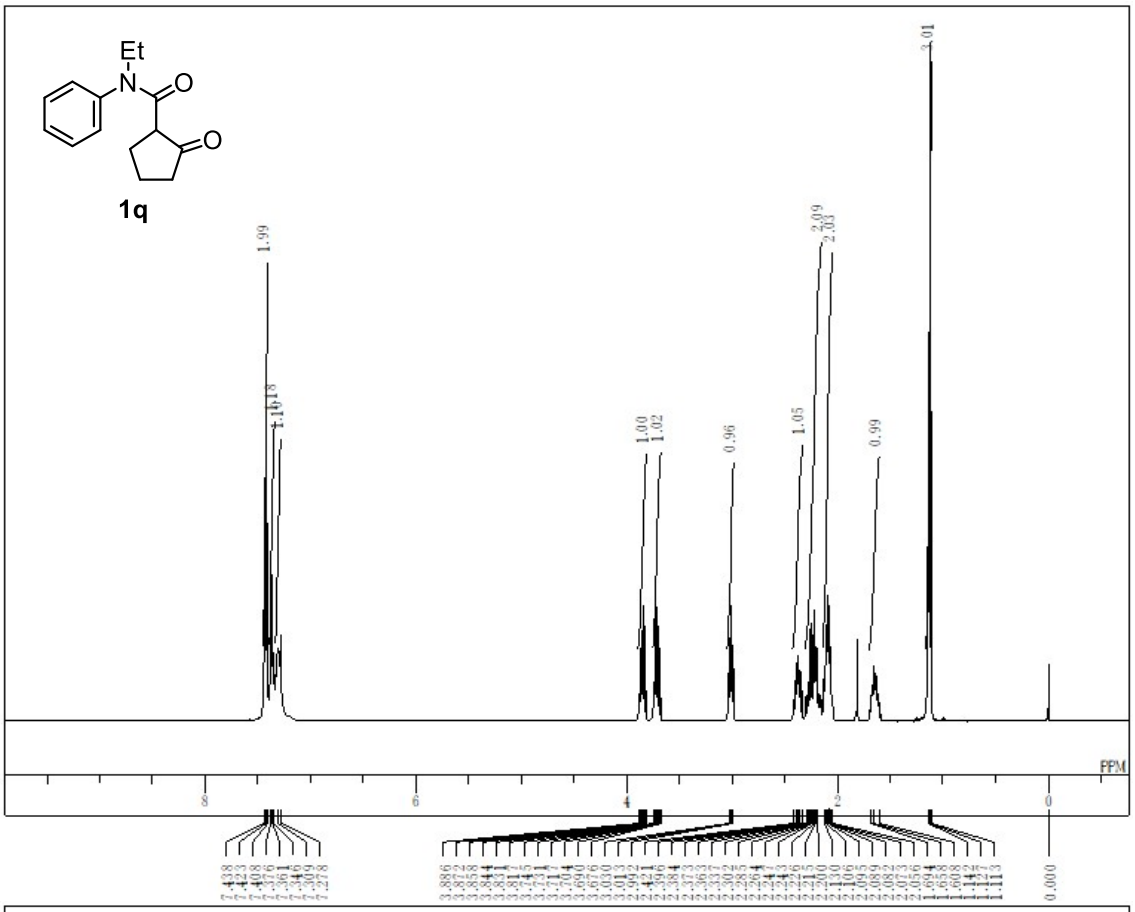
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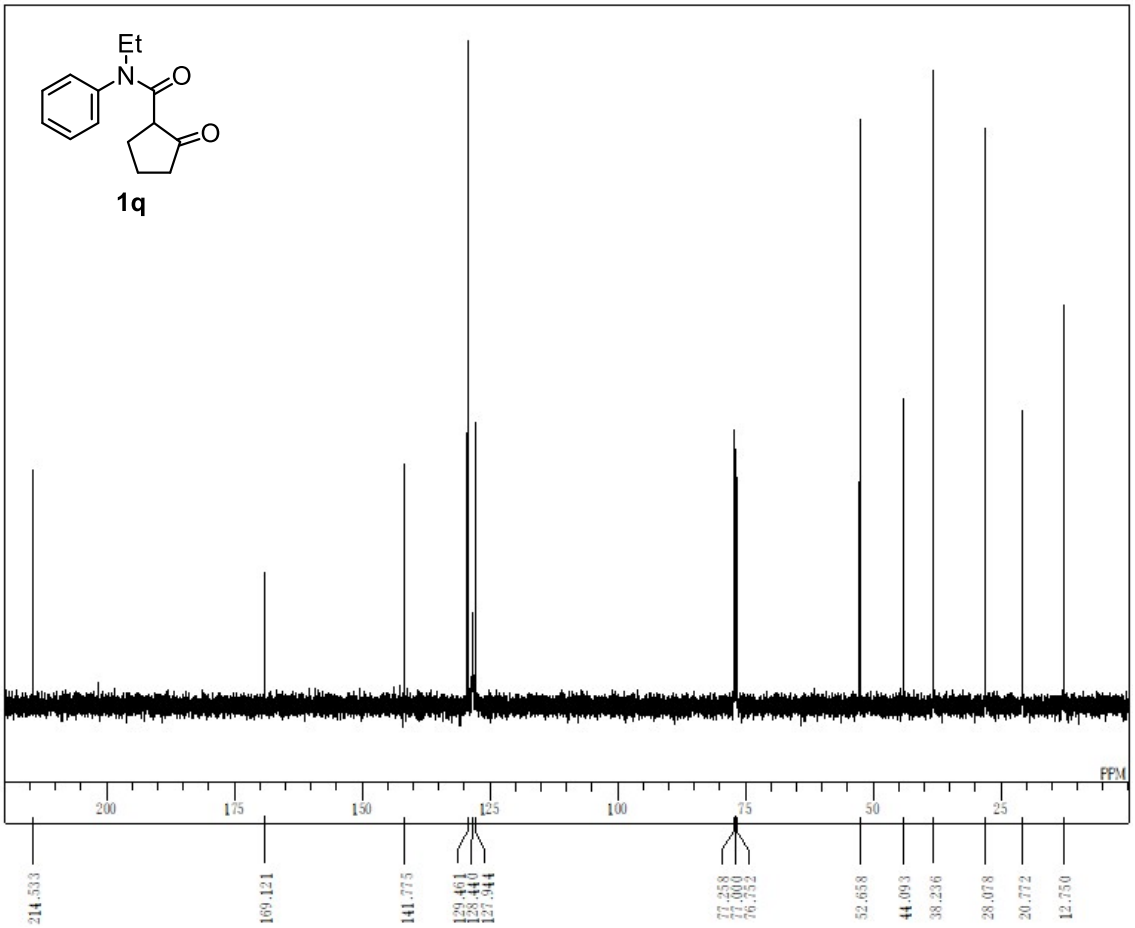
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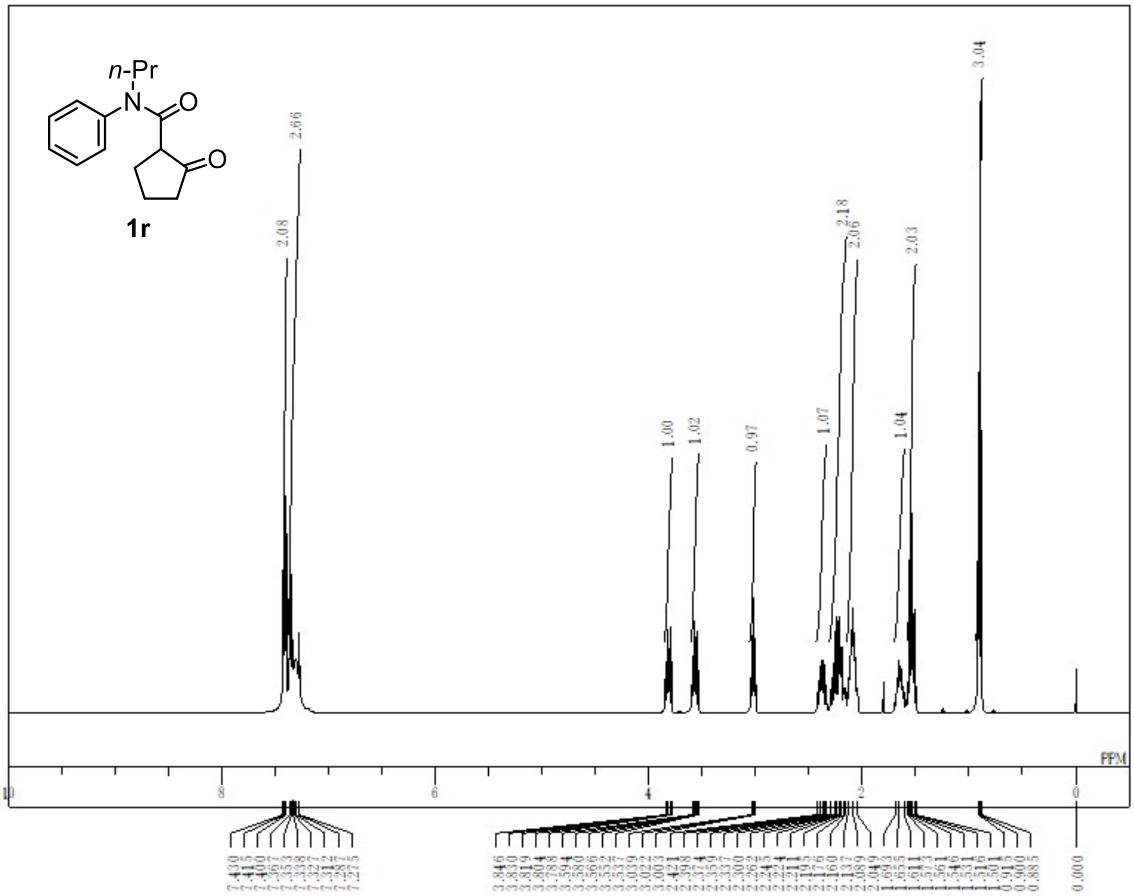
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1.02
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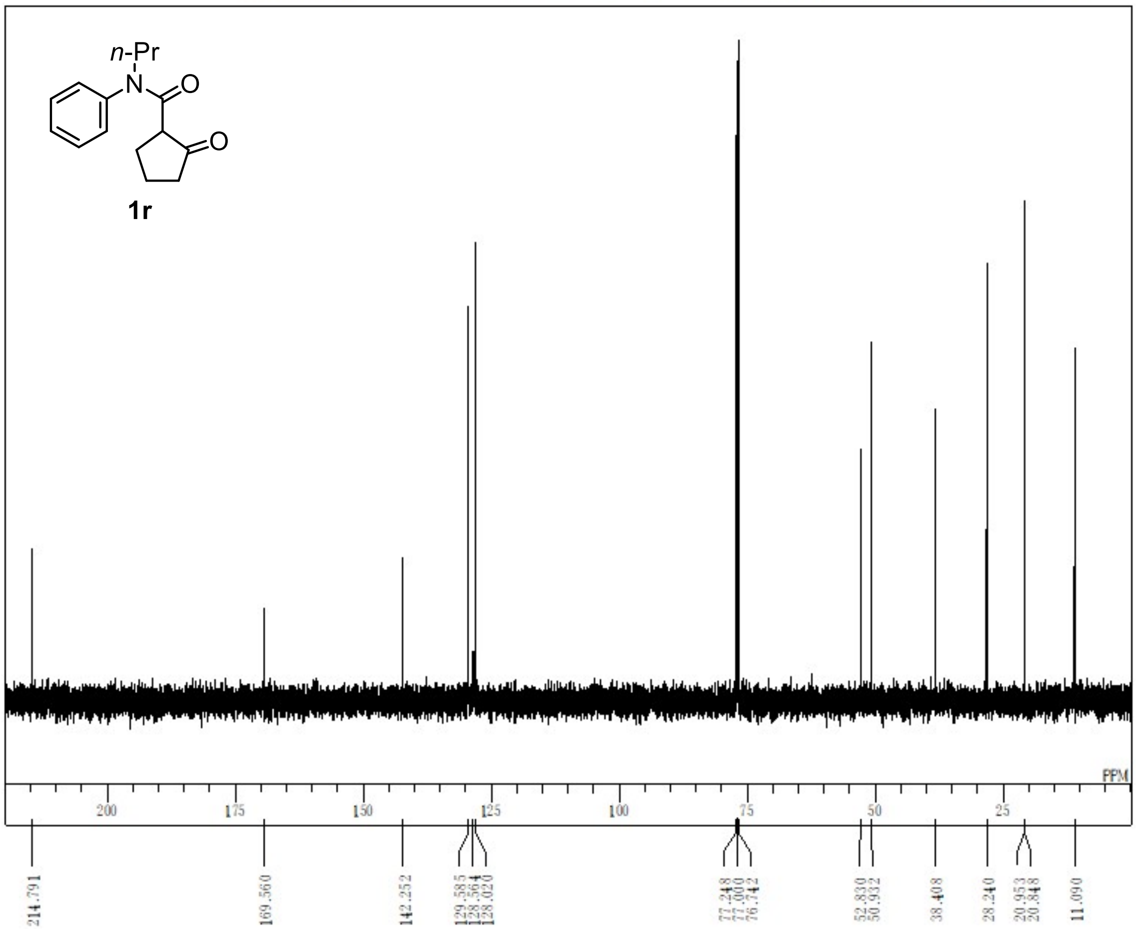
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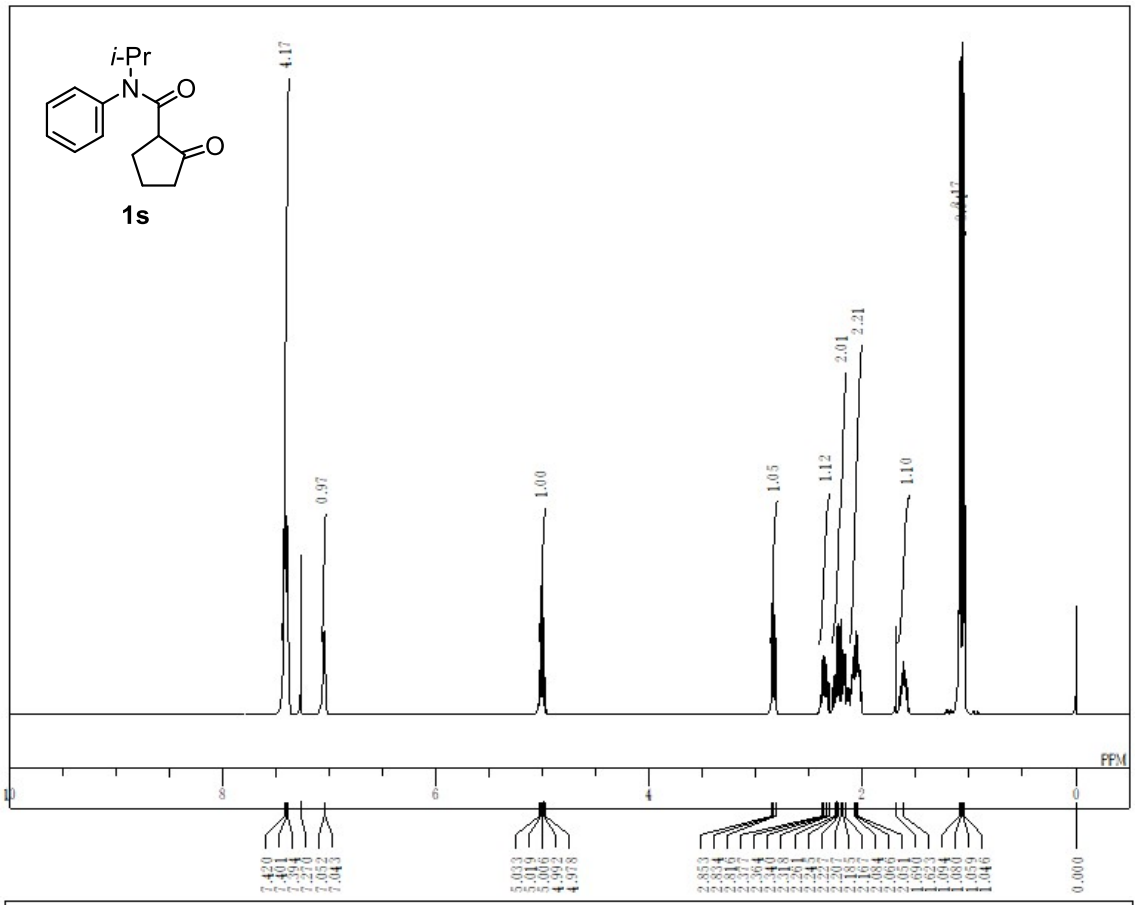
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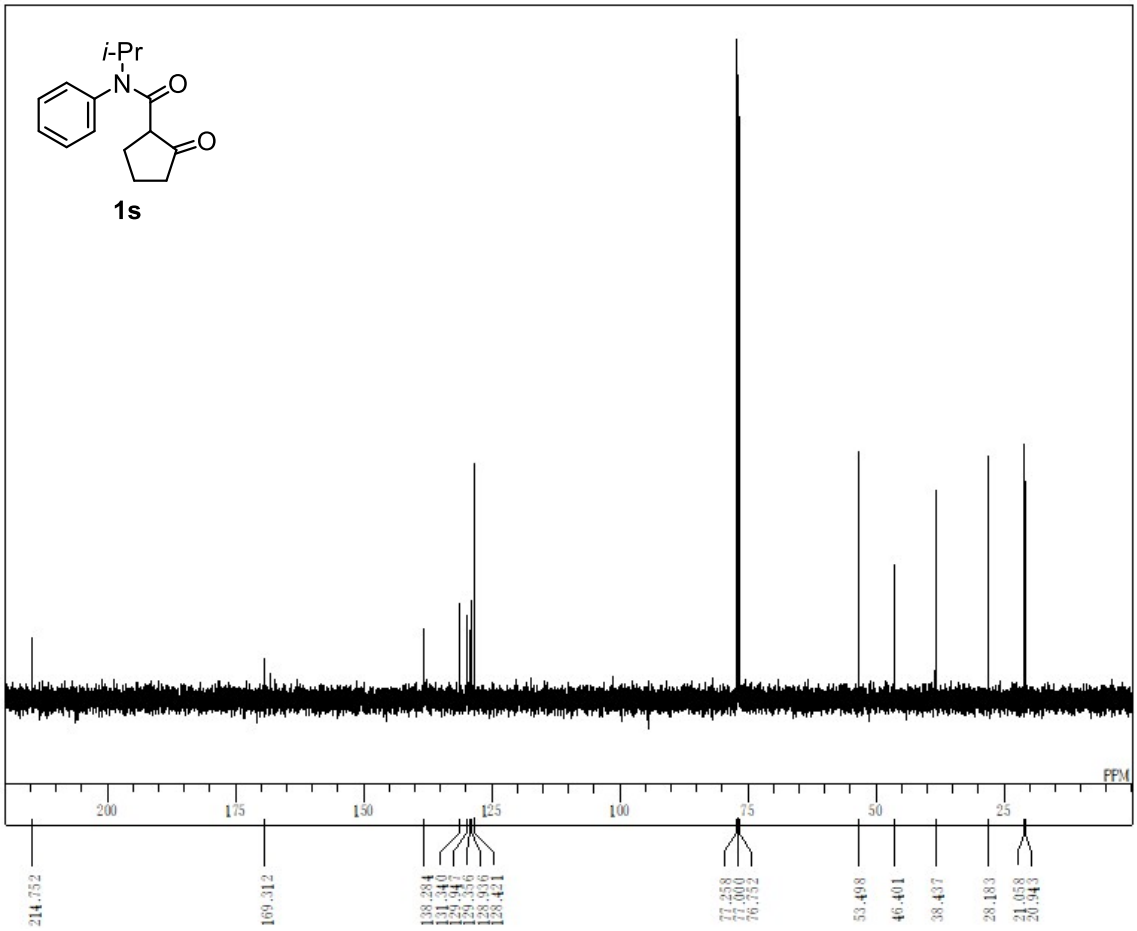
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1s
PPM
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50
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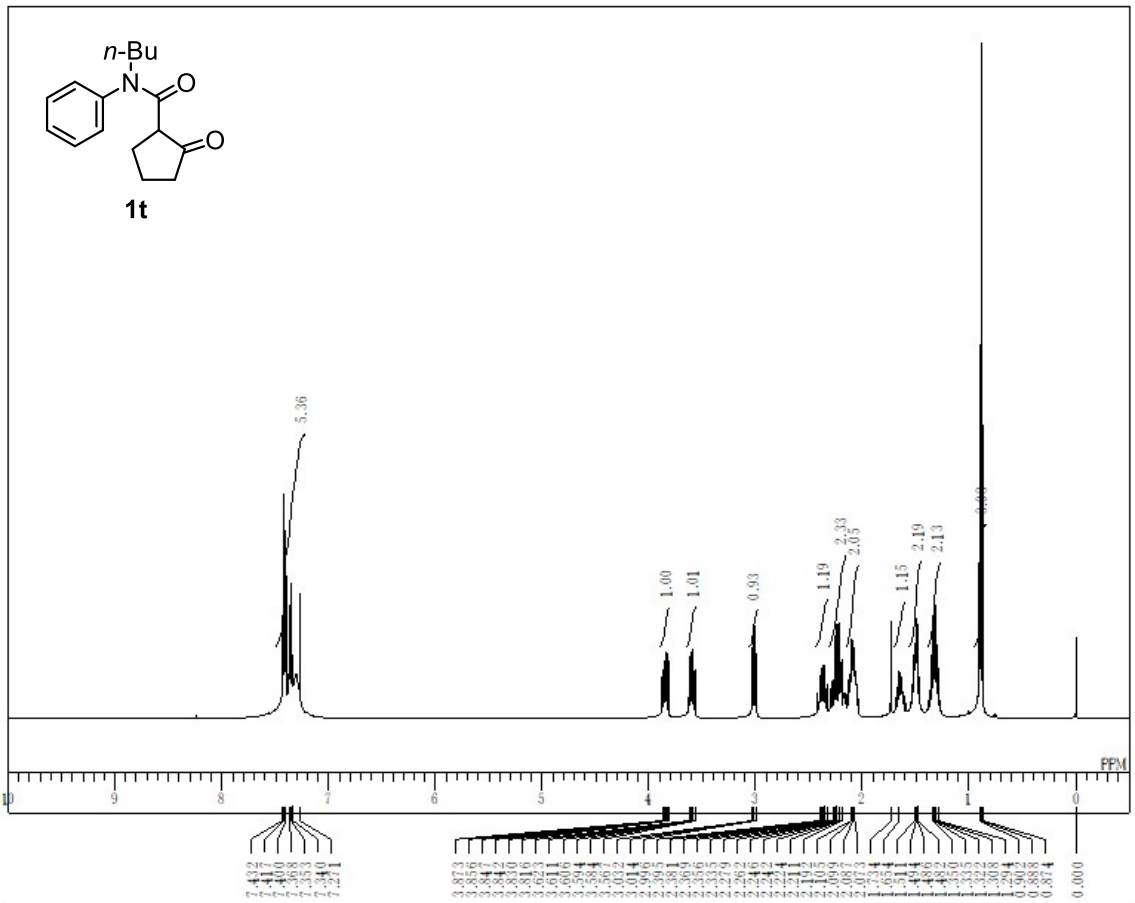
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1.19
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2.13
3.00
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7
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3
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1
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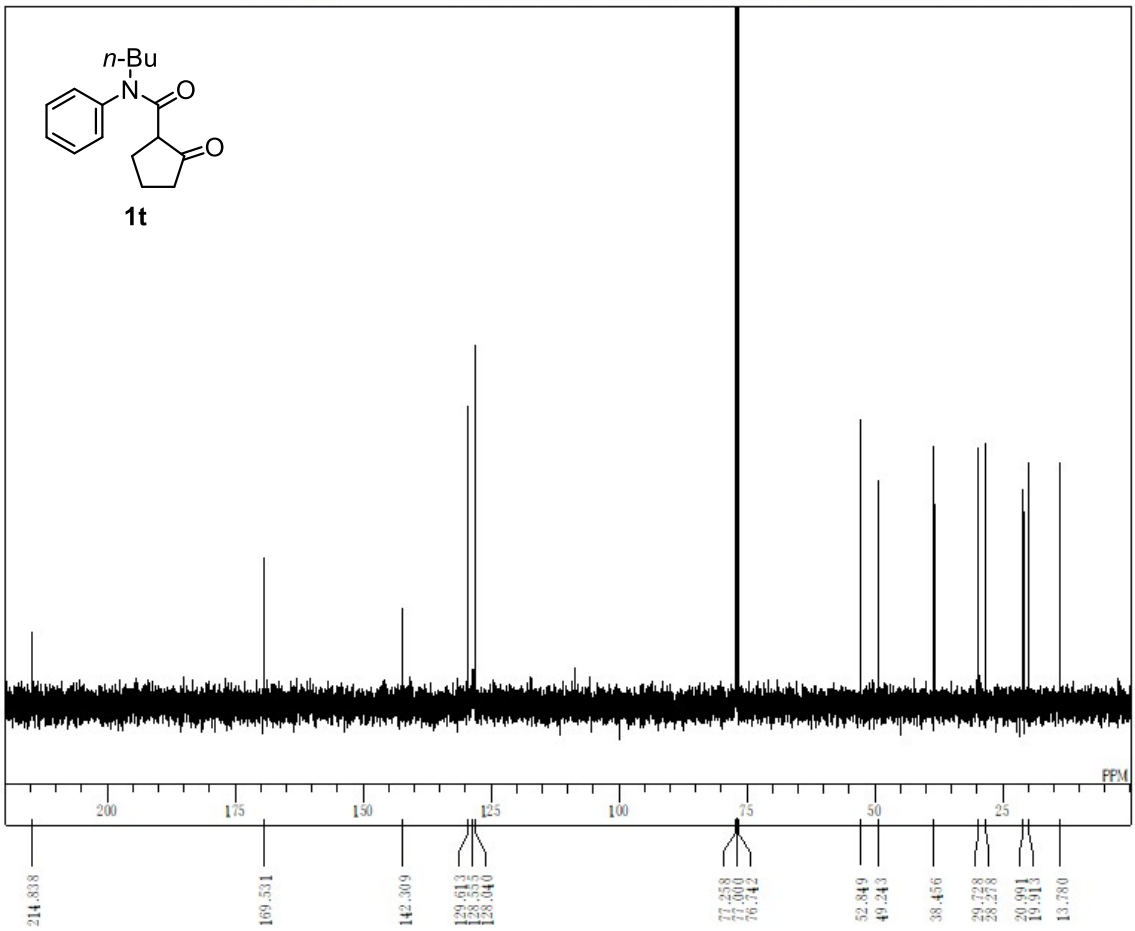
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O
1t
PPM
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125
100
75
50
25
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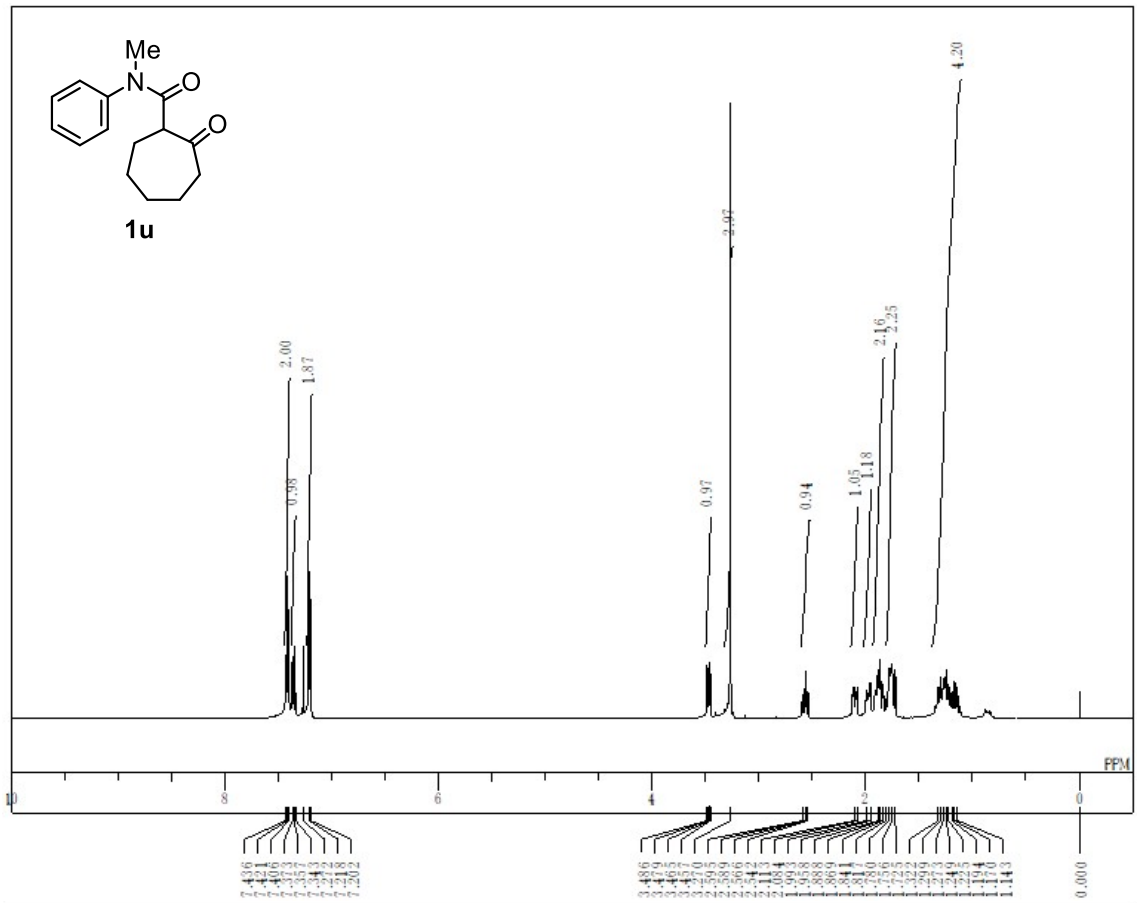
13.780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4
2
0
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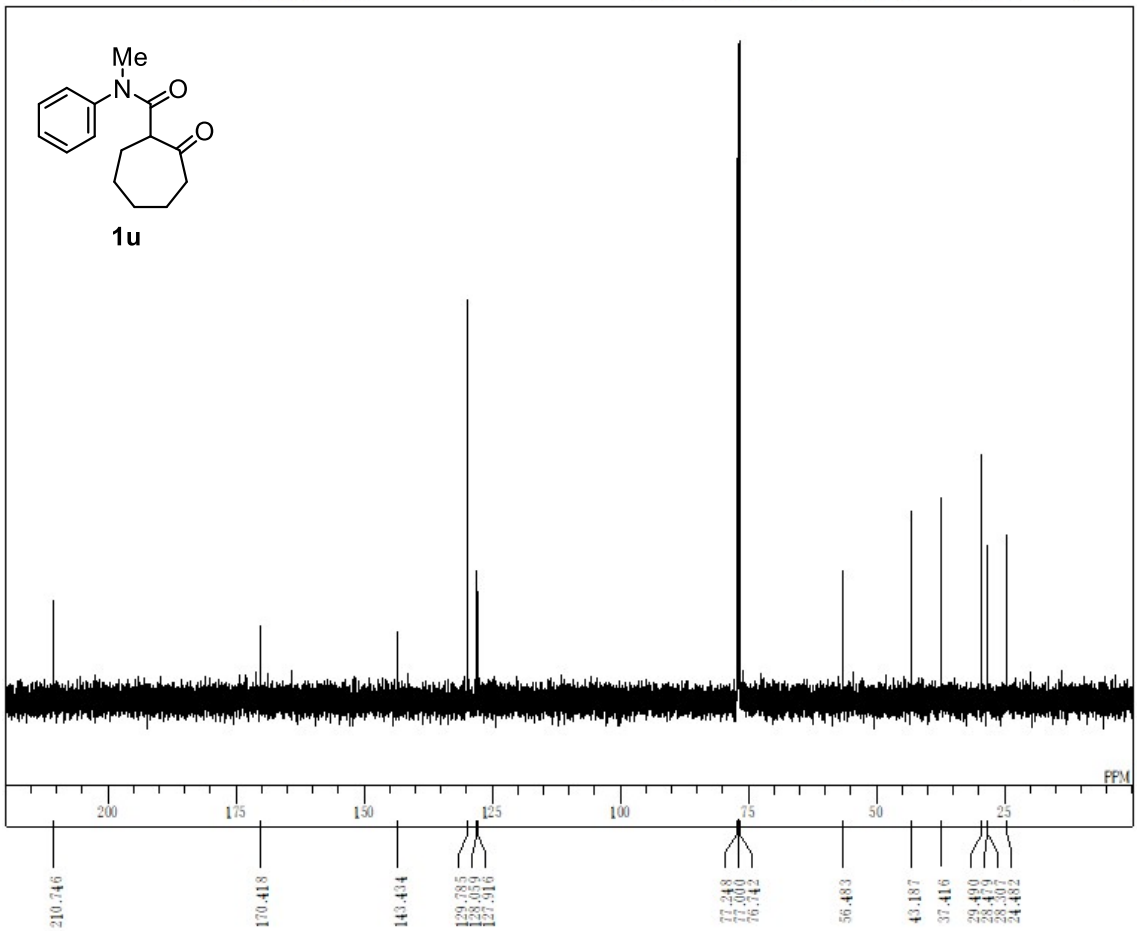
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O
O
1u
PPM
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175
150
125
100
75
50
25
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129.785
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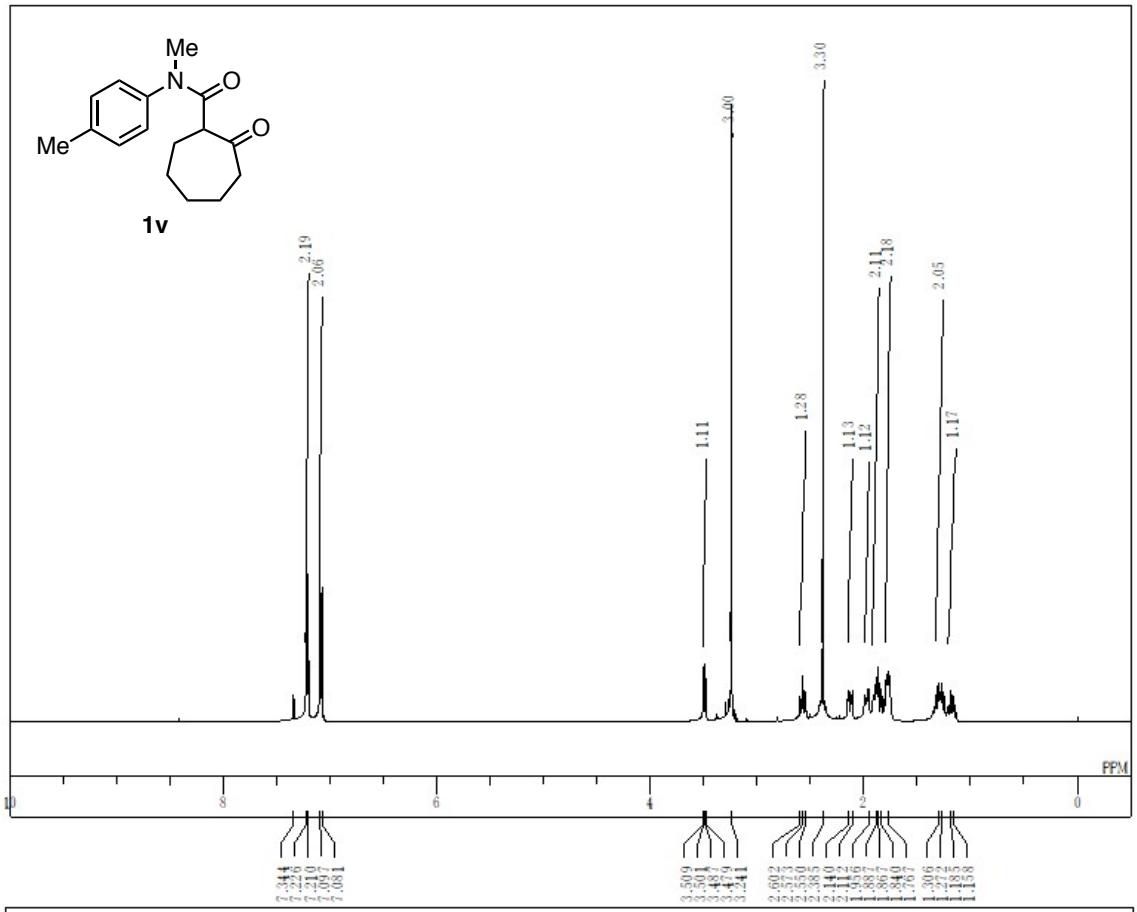
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2.18
2.05
1.17
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8
6
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2
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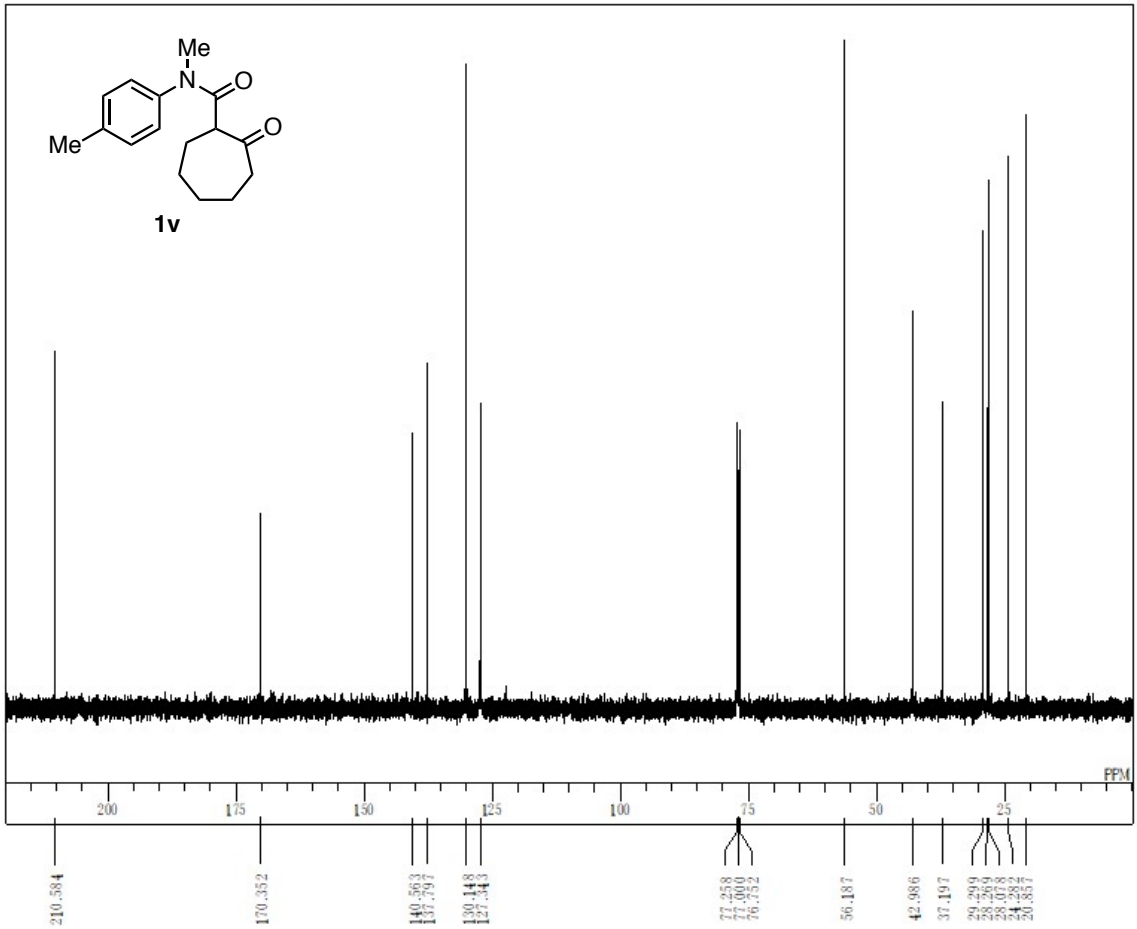
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O
Me
1v
PPM
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75
50
25
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130.148
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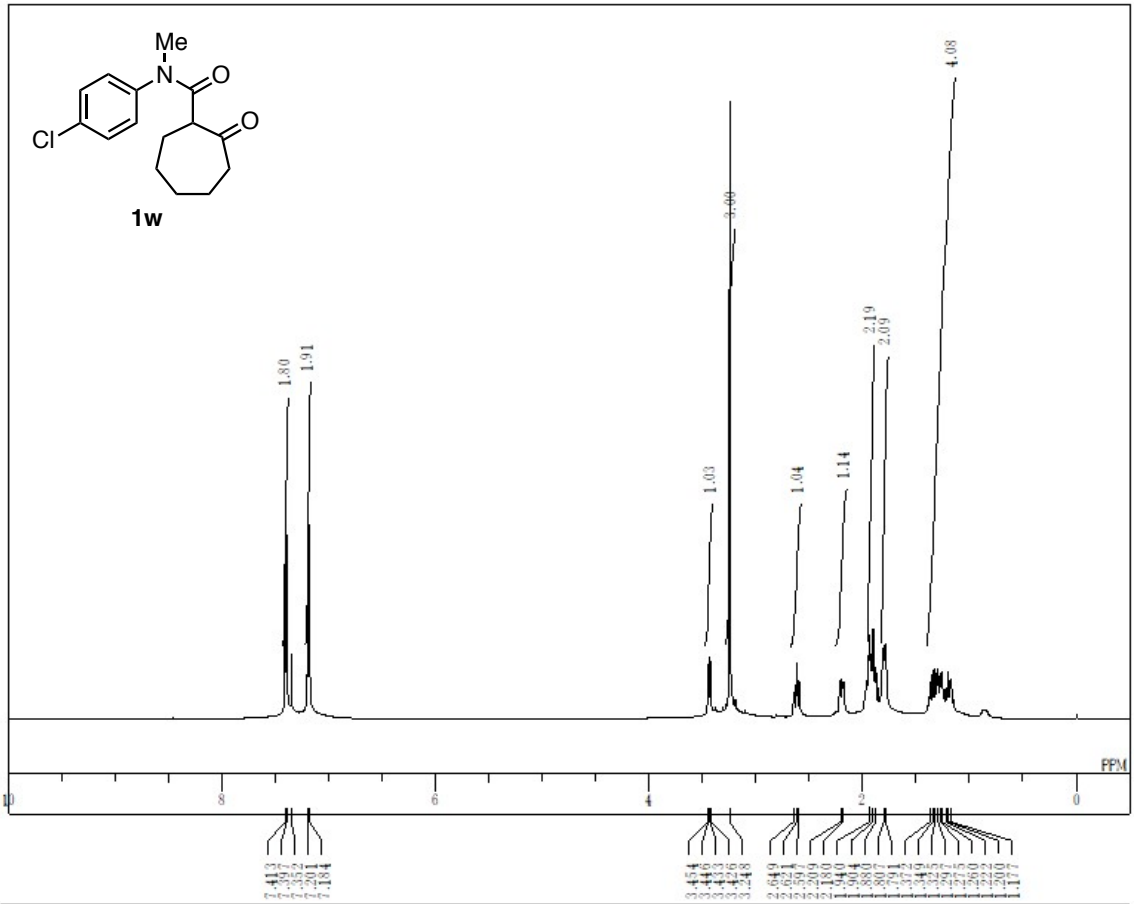
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6
4
2
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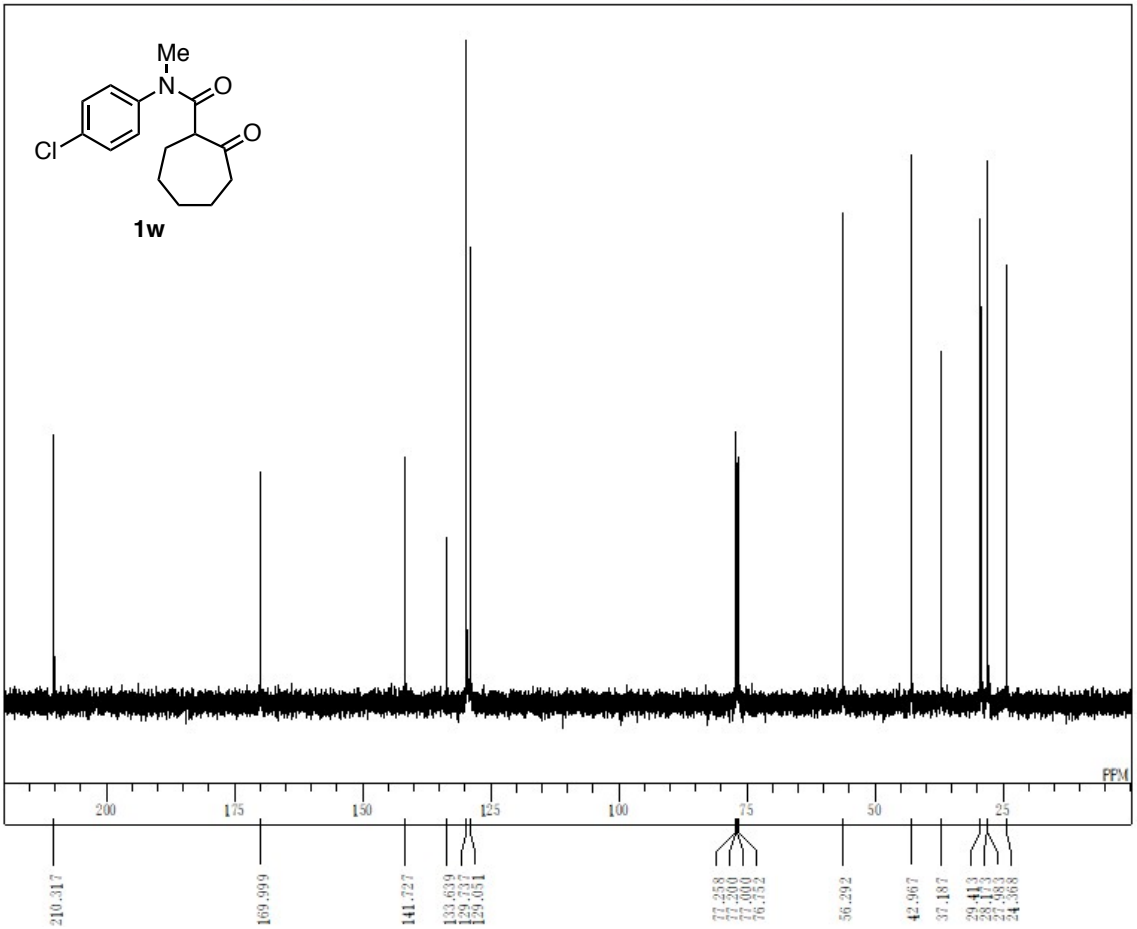
Me
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O
O
Cl
O
1w
PPM
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150
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100
75
50
25
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133.639
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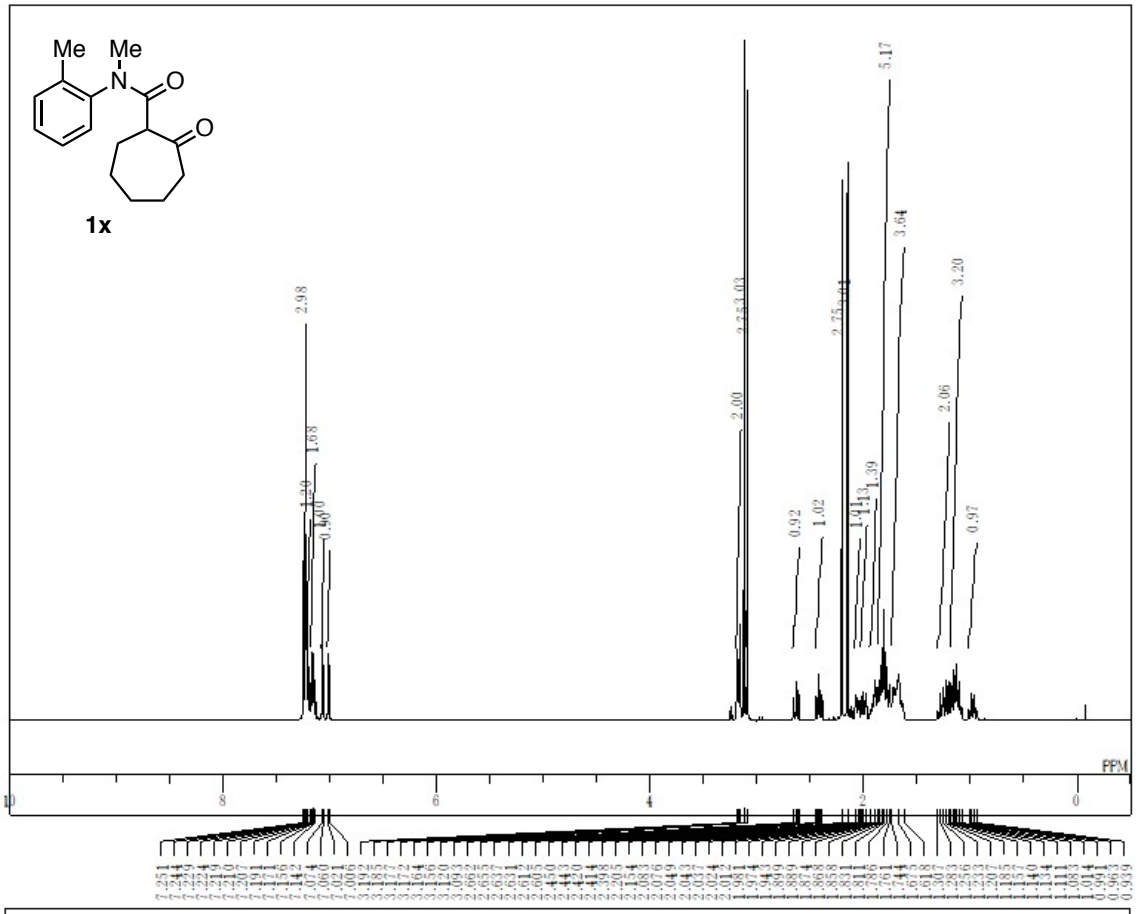
24.368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0.97
1.39
PPM
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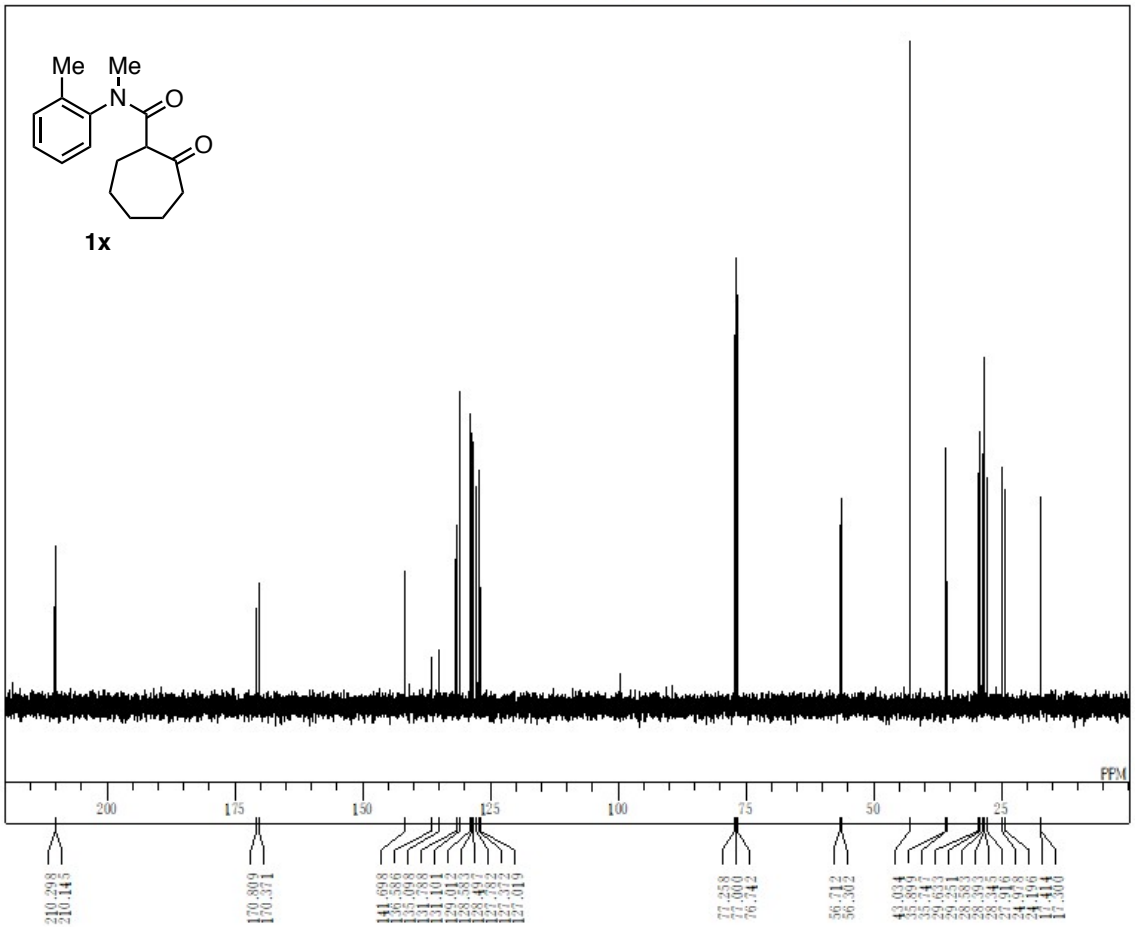
8
6
4
2
0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Me
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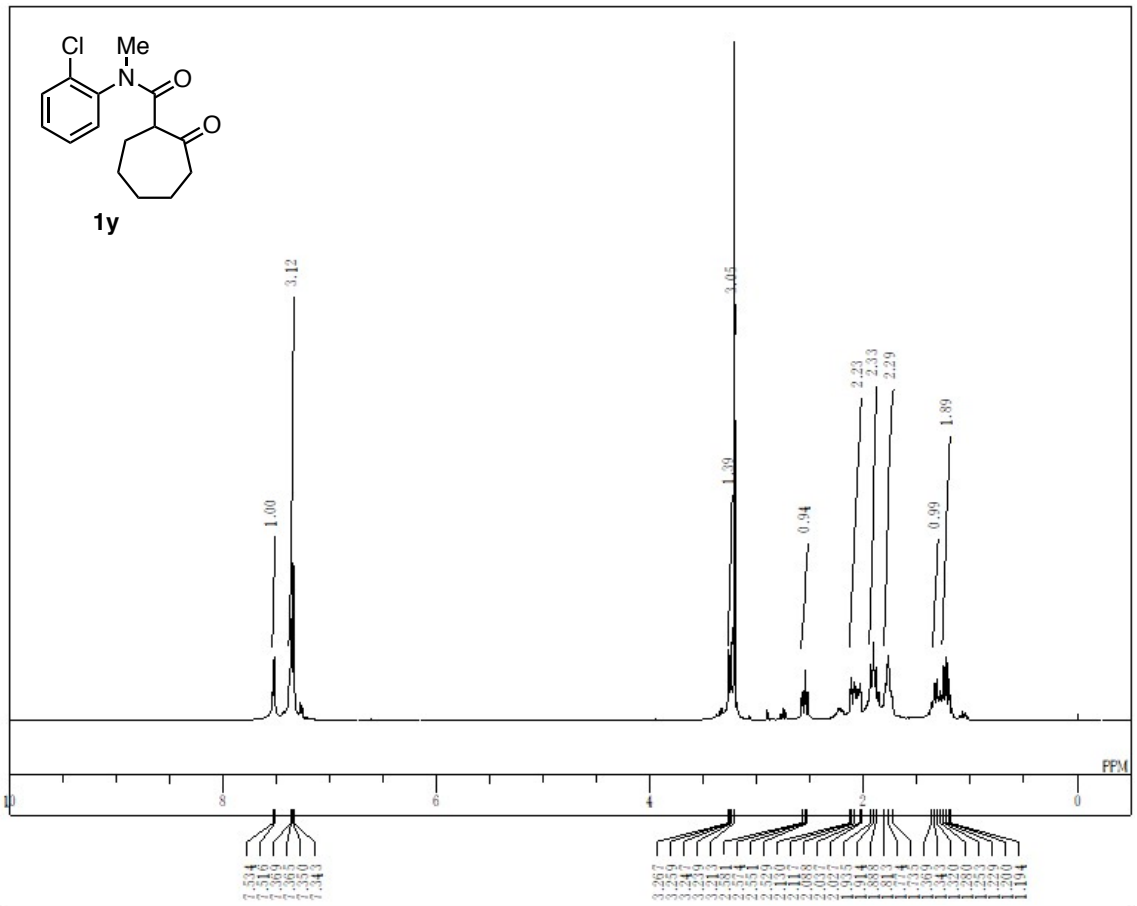
O
O
1x
PPM
200
175
150
125
100
75
50
25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Me
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O
1y
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1.39
1.00
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PPM
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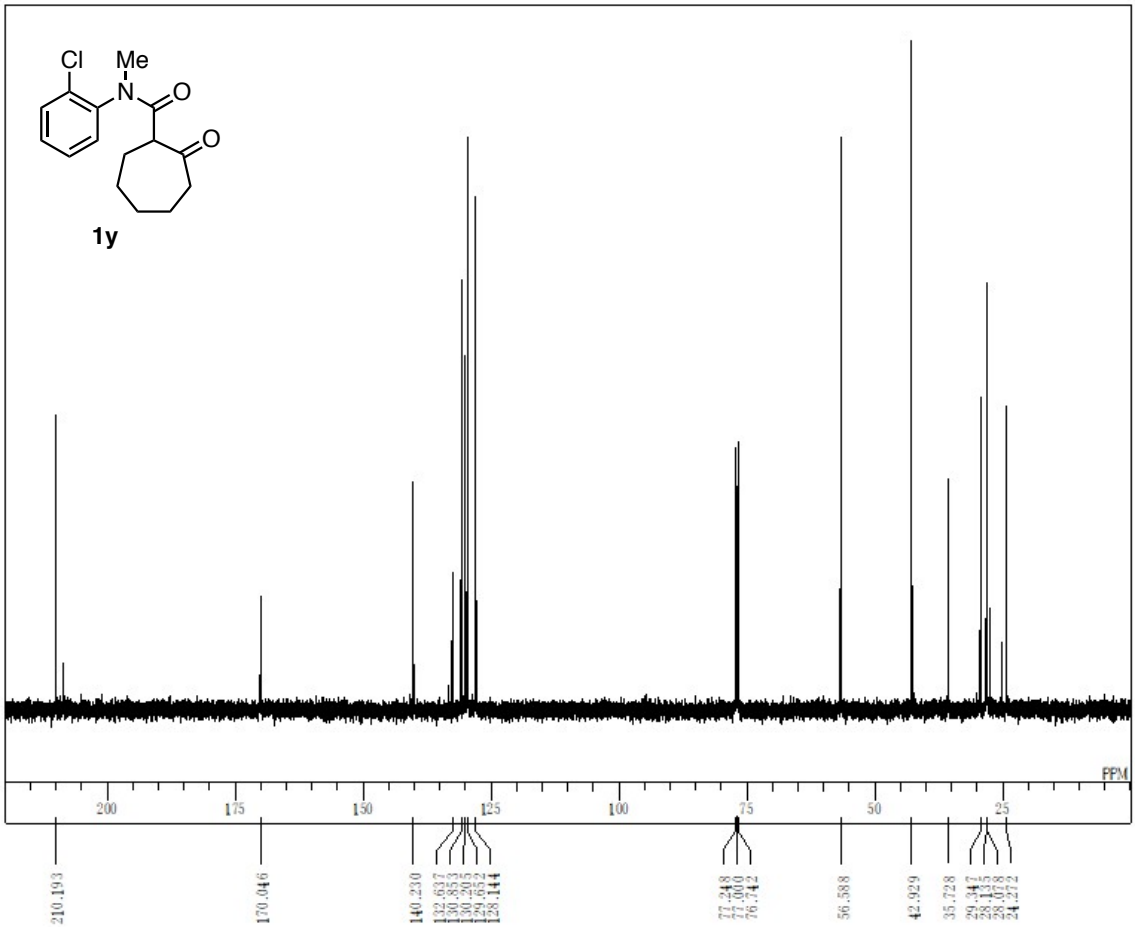
8
6
4
2
0


Cl
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O
1y
PPM
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150
125
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75
50
25
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132.637
130.853
130.205
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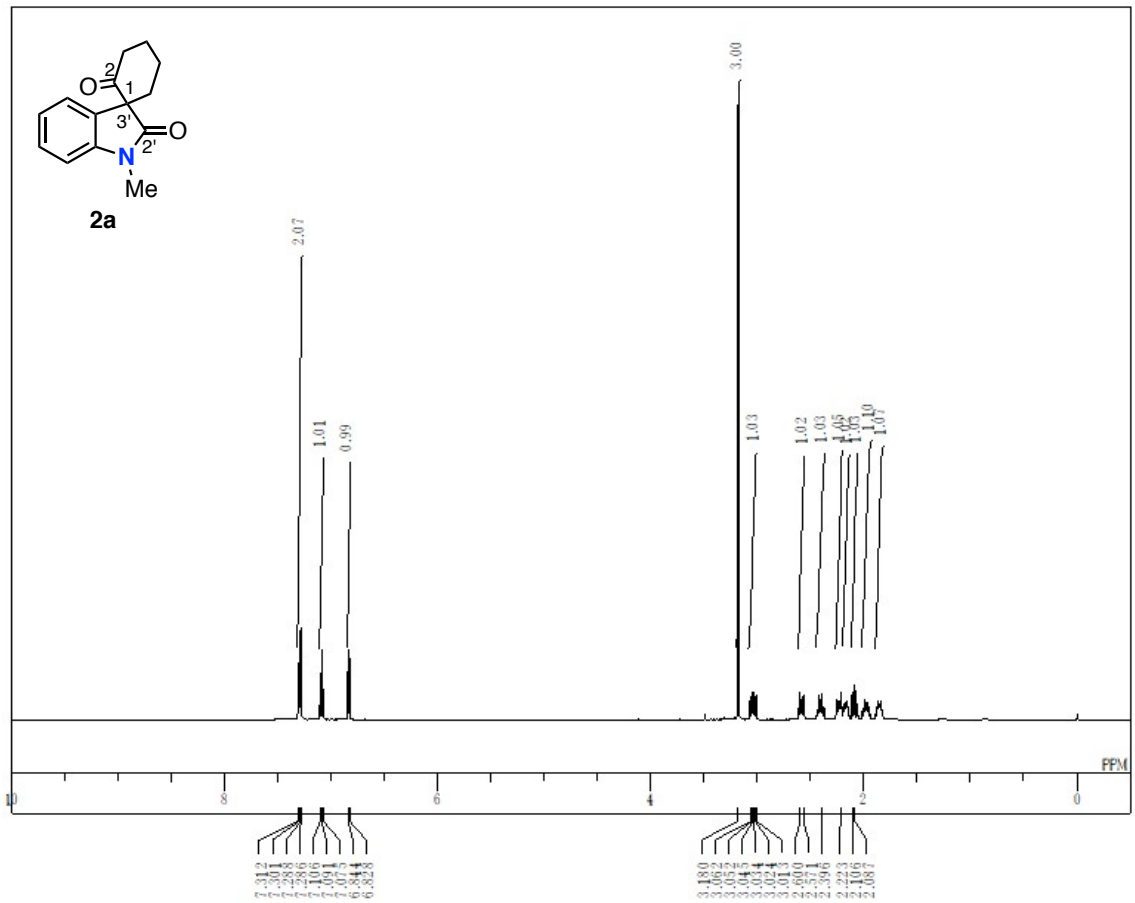
24.272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O
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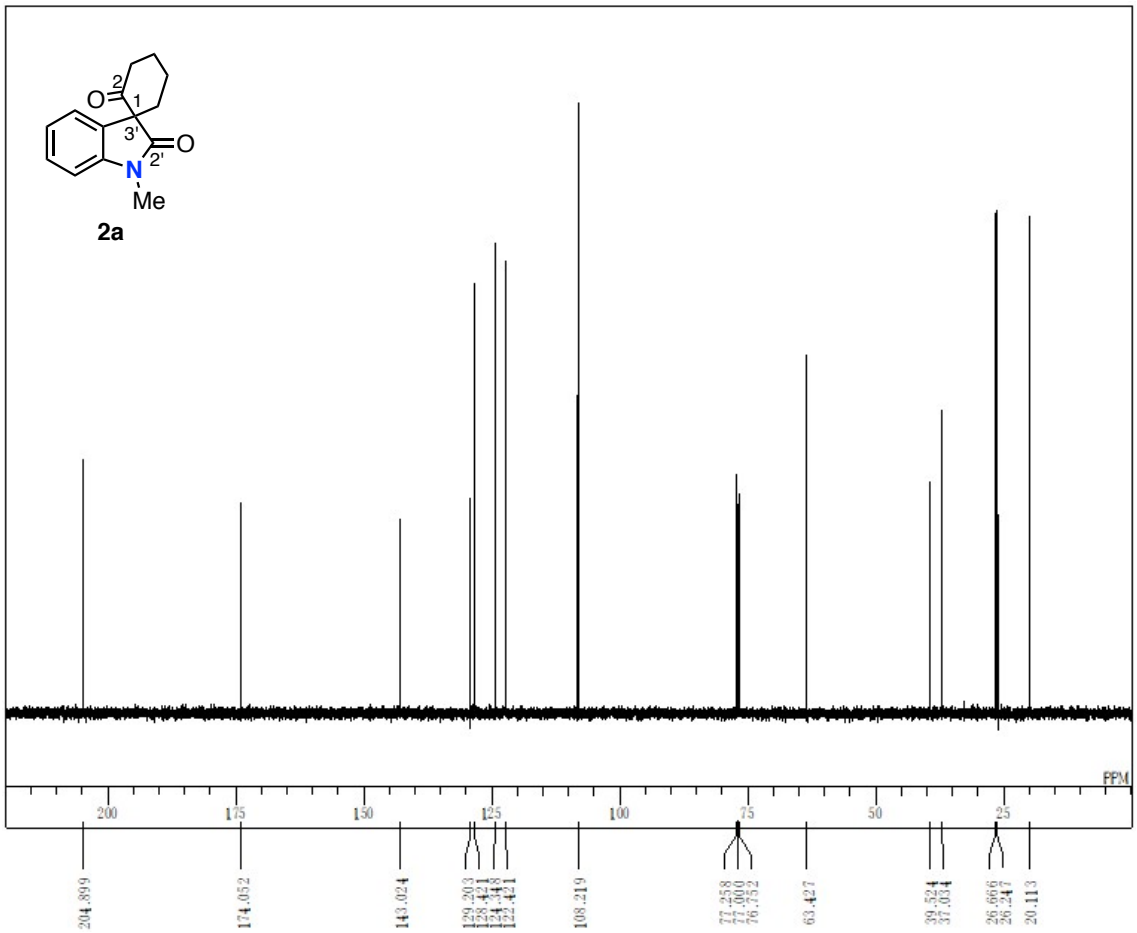
1.03
1.05
1.05
1.10
1.07
PPM
10
8
6
4
2
0


2a
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N
O
PPM
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150
125
100
75
50
25
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174.052
143.024
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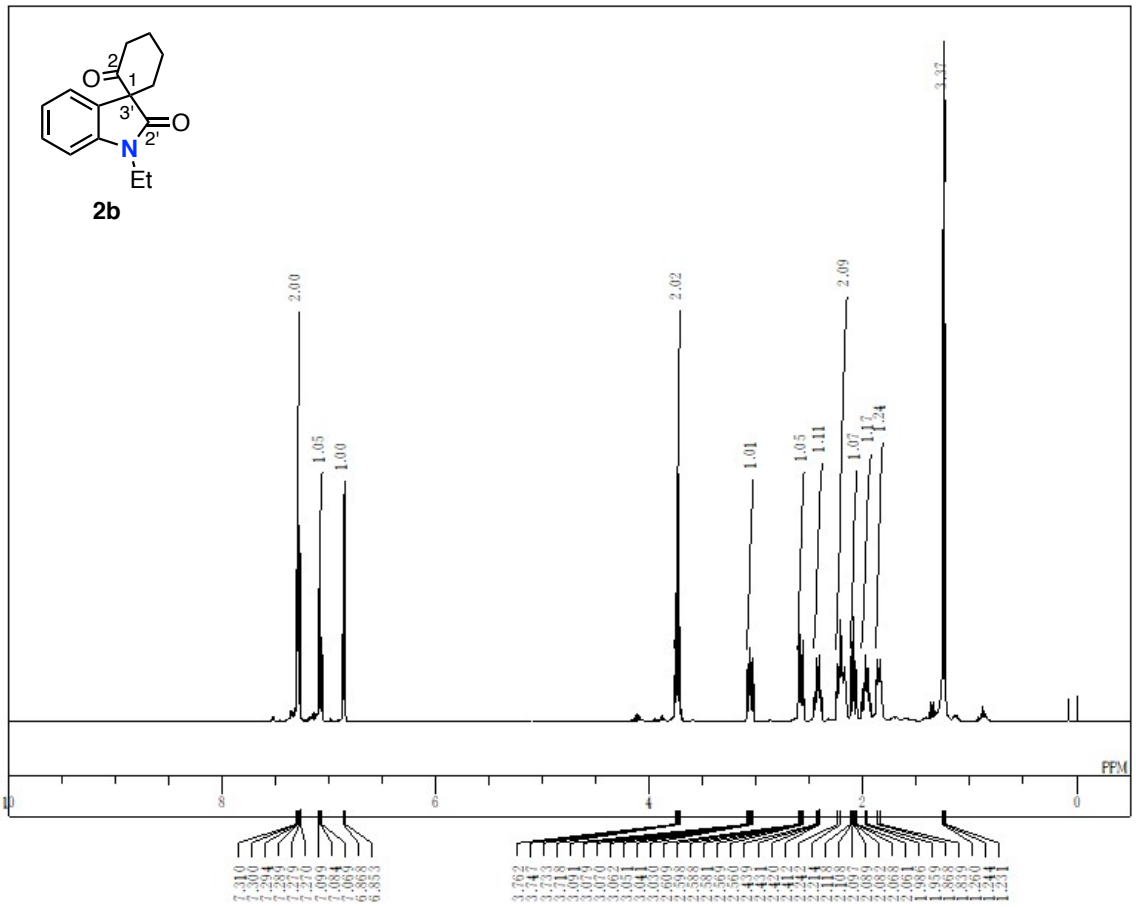
26.247
20.113


2b
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1.11
2.09
1.07
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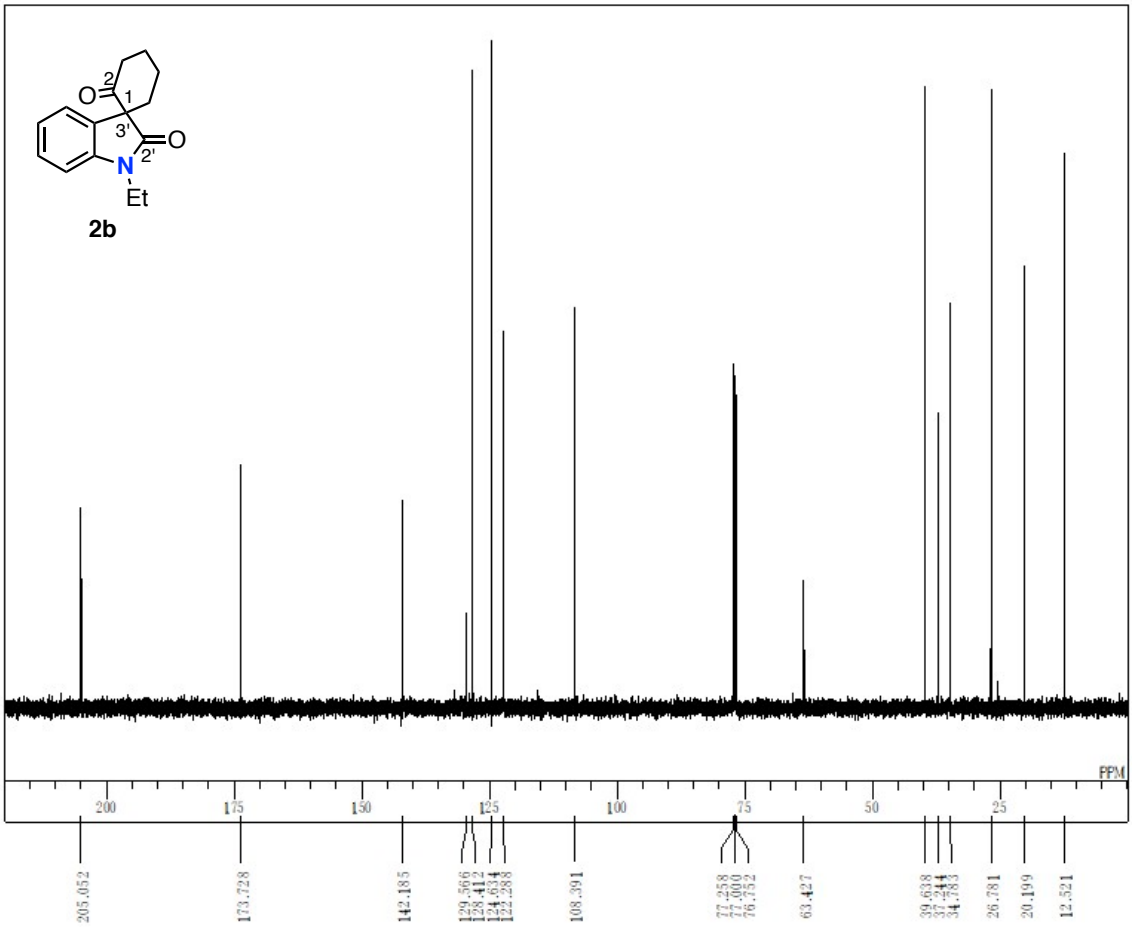
1.24
3.37
PPM


2b
PPM
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128.412
124.634
122.288
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77.258
77.000
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37.244
34.183
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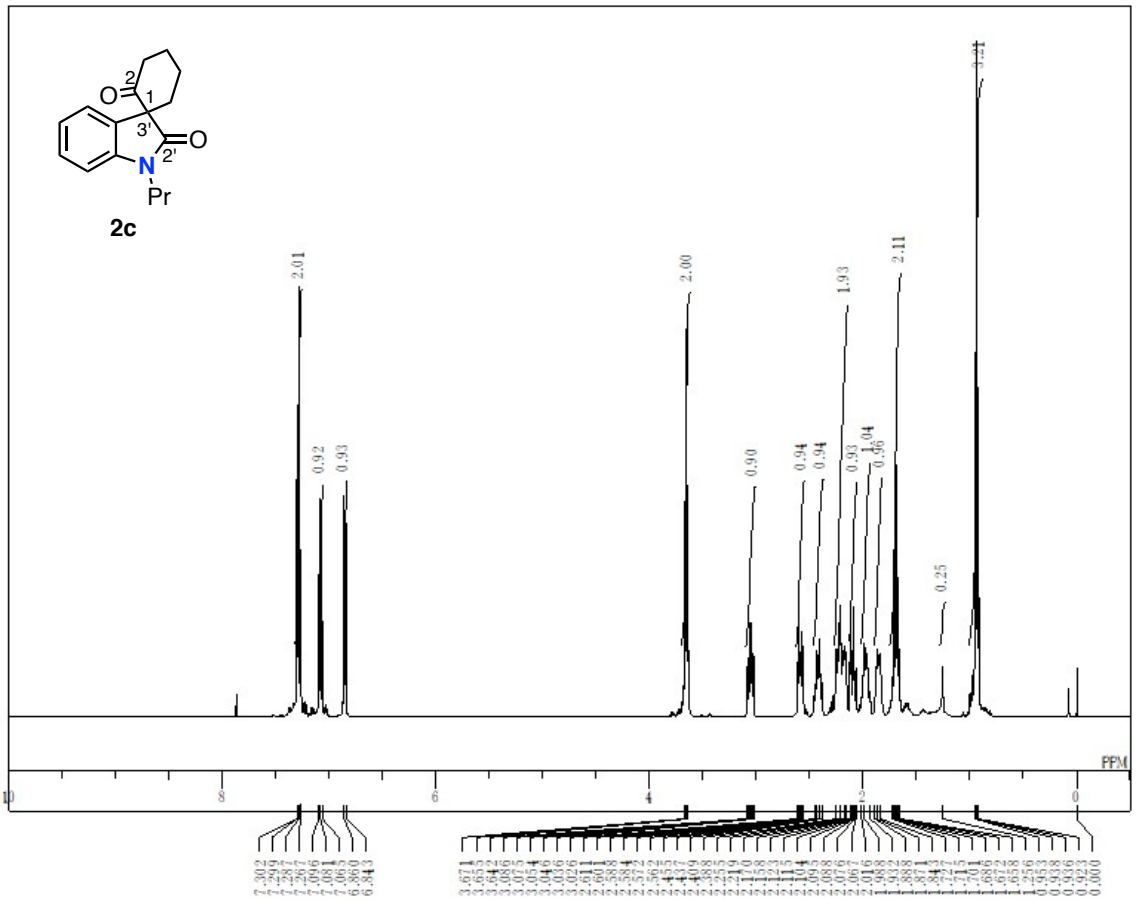
20.199
12.521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0.94
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0.46
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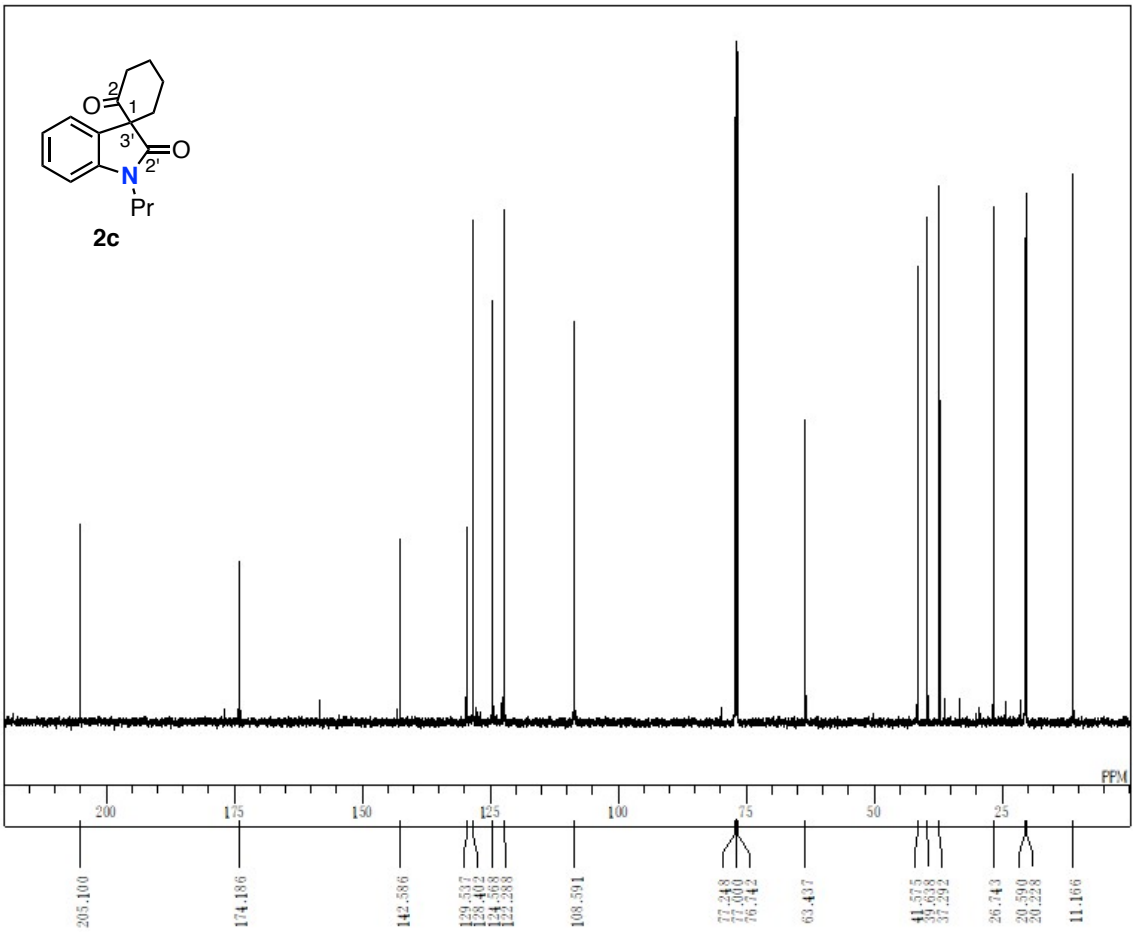
0.25
3.21
PPM


2c
PPM
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122.288
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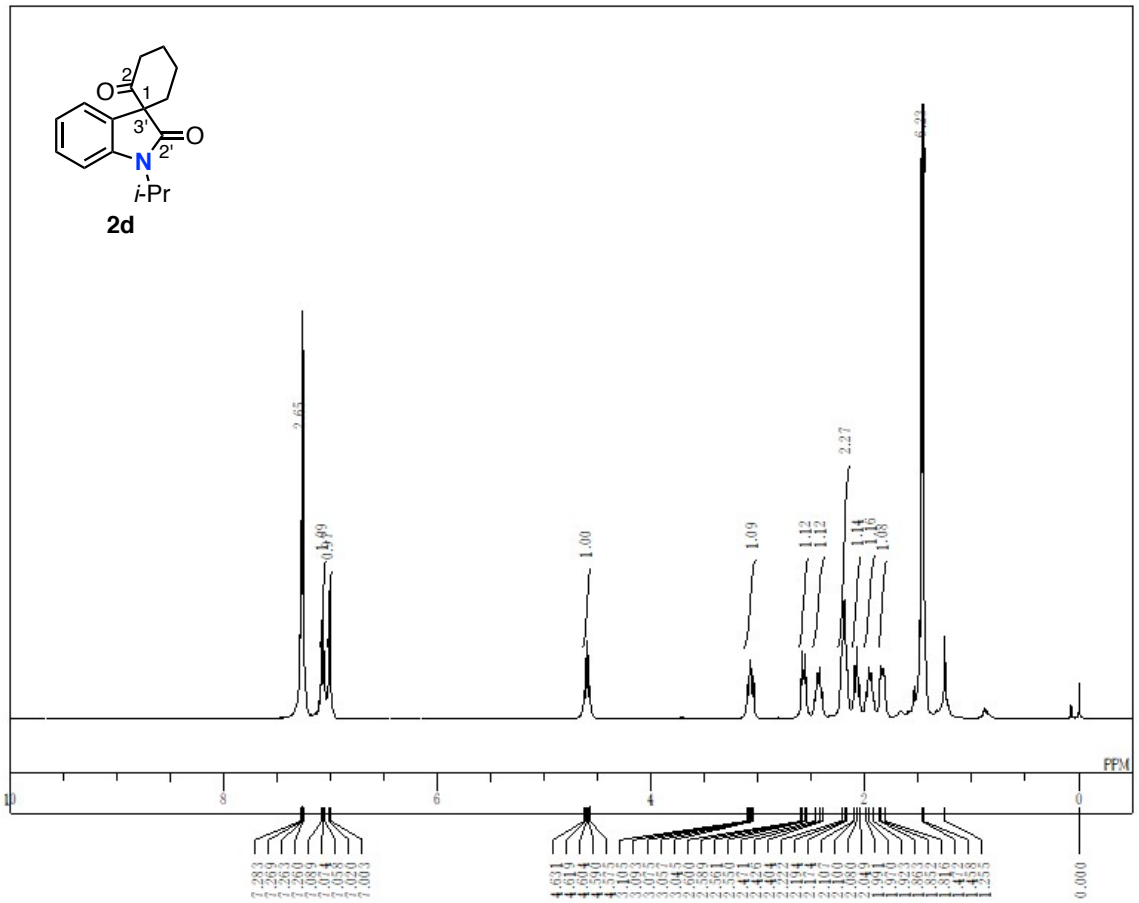
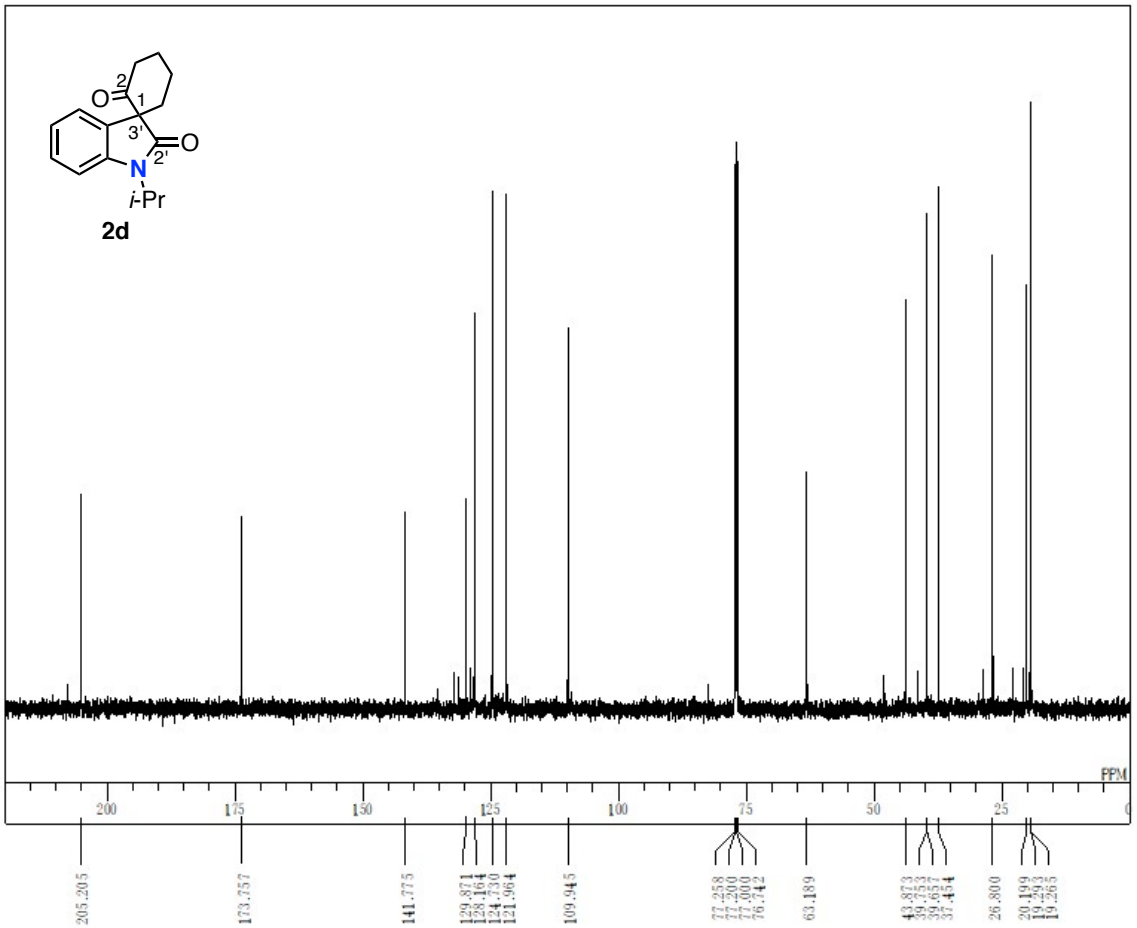
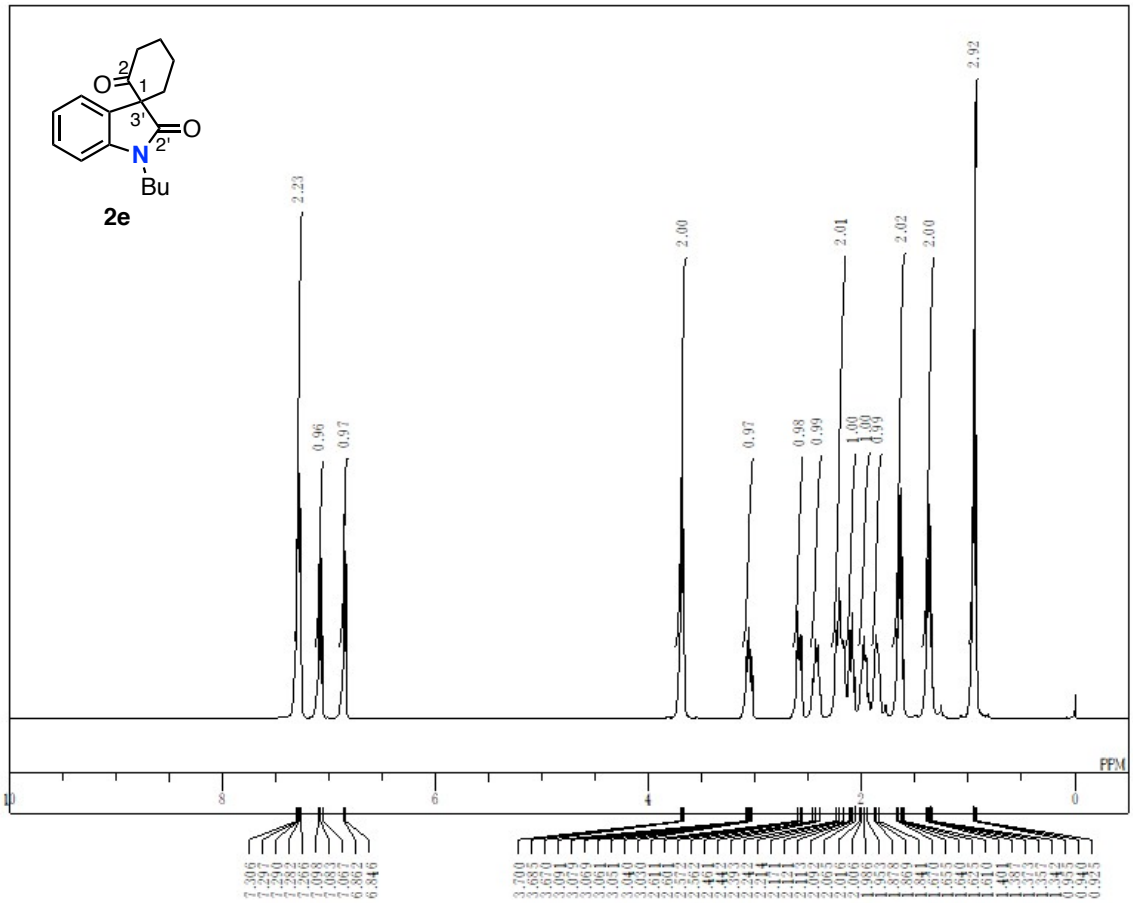
20.590
20.228
11.166


O
2
1
3'
O
2'
N
i-Pr
2d
PPM


O
2
1
3'
O
2'
N
i-Pr
2d
PPM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1.00
0.99
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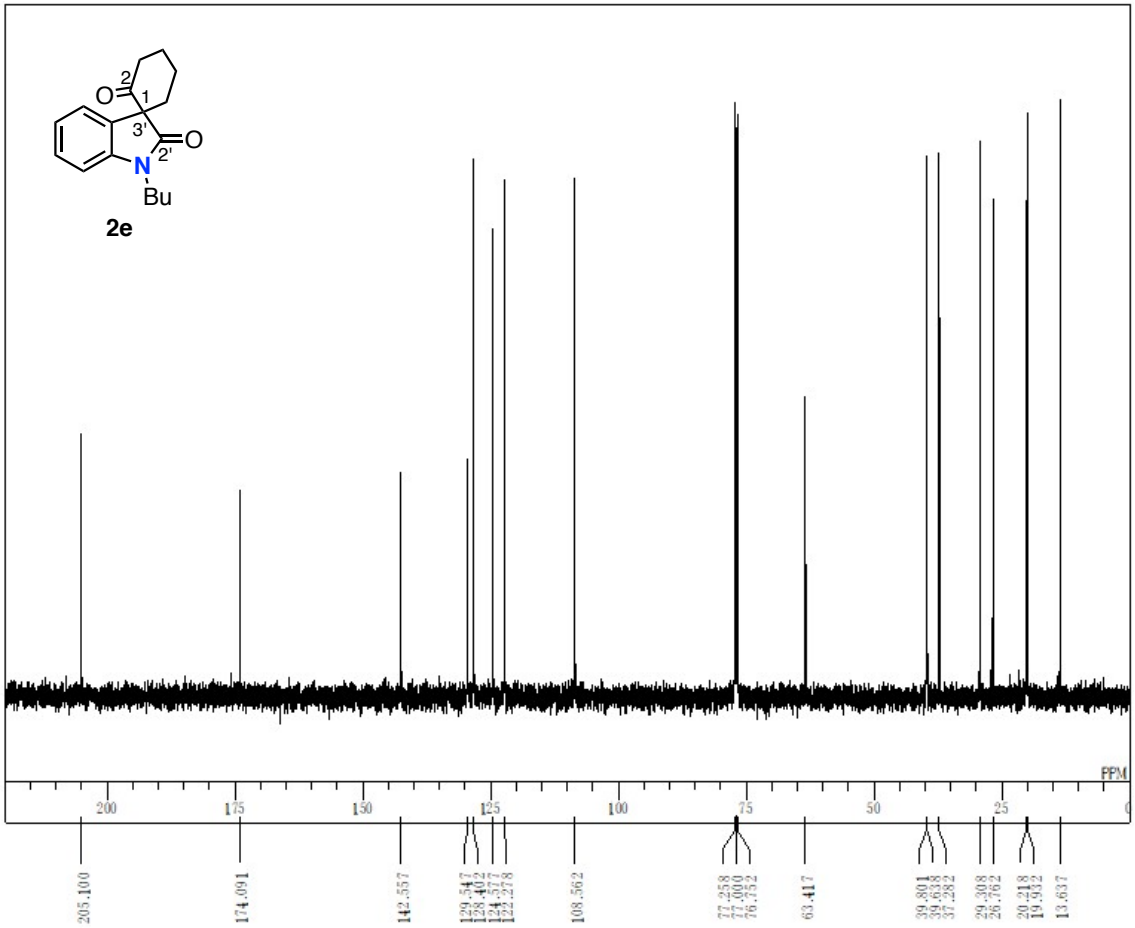
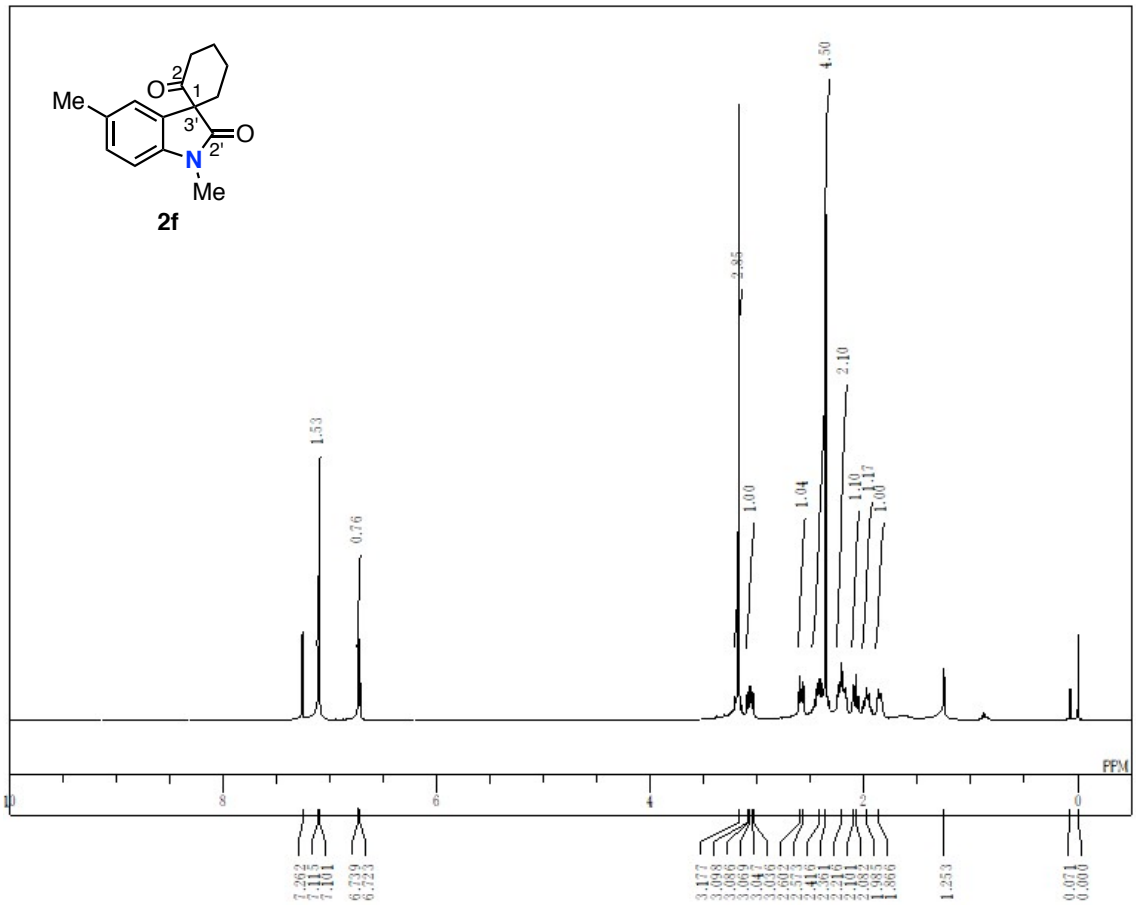
PPM


2e
PPM


Me
O
2
1
3'
O
2'
N
Me
2f
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2.85
2.10
1.53
1.00
1.04
1.10
1.17
1.00
0.76
PPM
10
8
6
4
2
0
7.262
7.115
7.101
6.739
6.723
3.177
3.098
3.086
3.069
3.047
3.036
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2.573
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2.101
2.082
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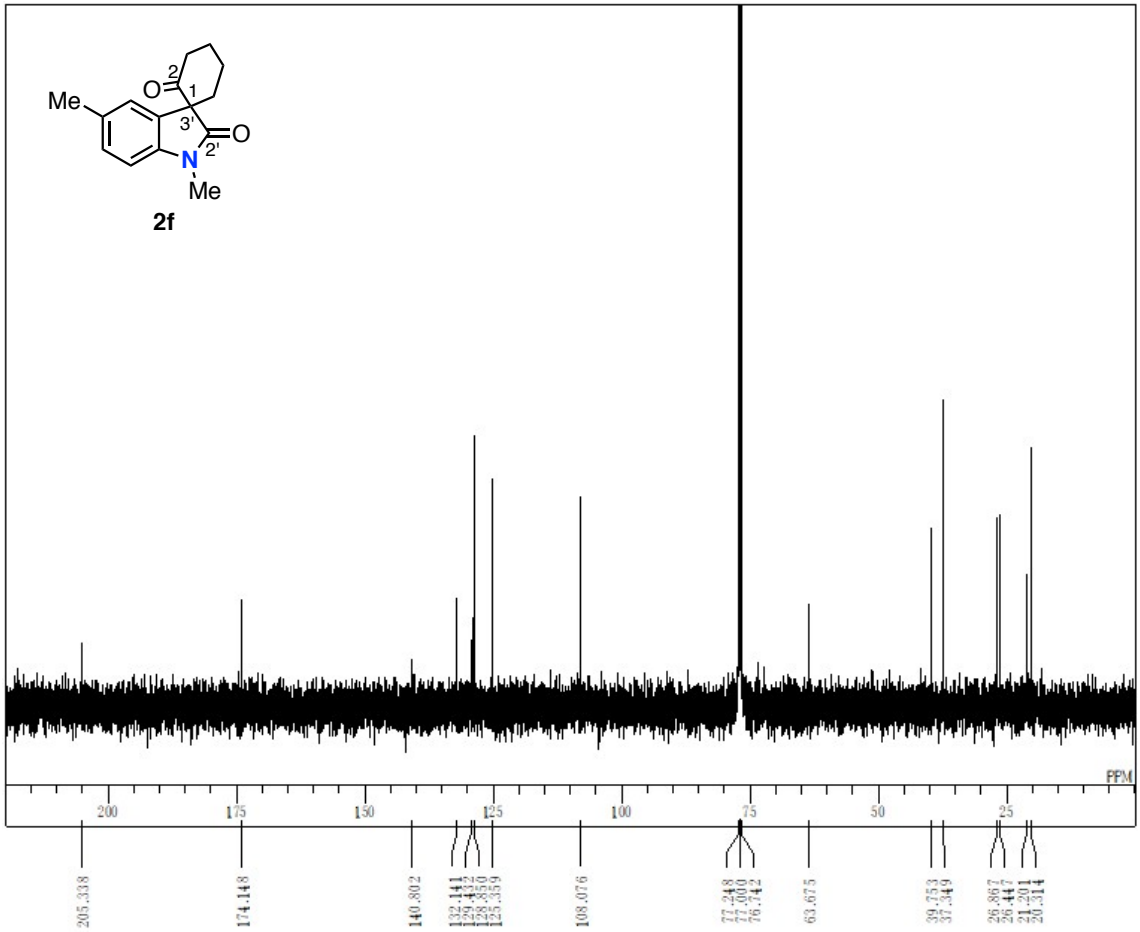
Me
O
2
1
3'
O
2'
N
Me
2f
PPM
200
175
150
125
100
75
50
25
205.338
174.148
140.802
132.141
129.432
128.959
125.359
108.076
77.248
77.000
76.742
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39.753
37.349
26.867
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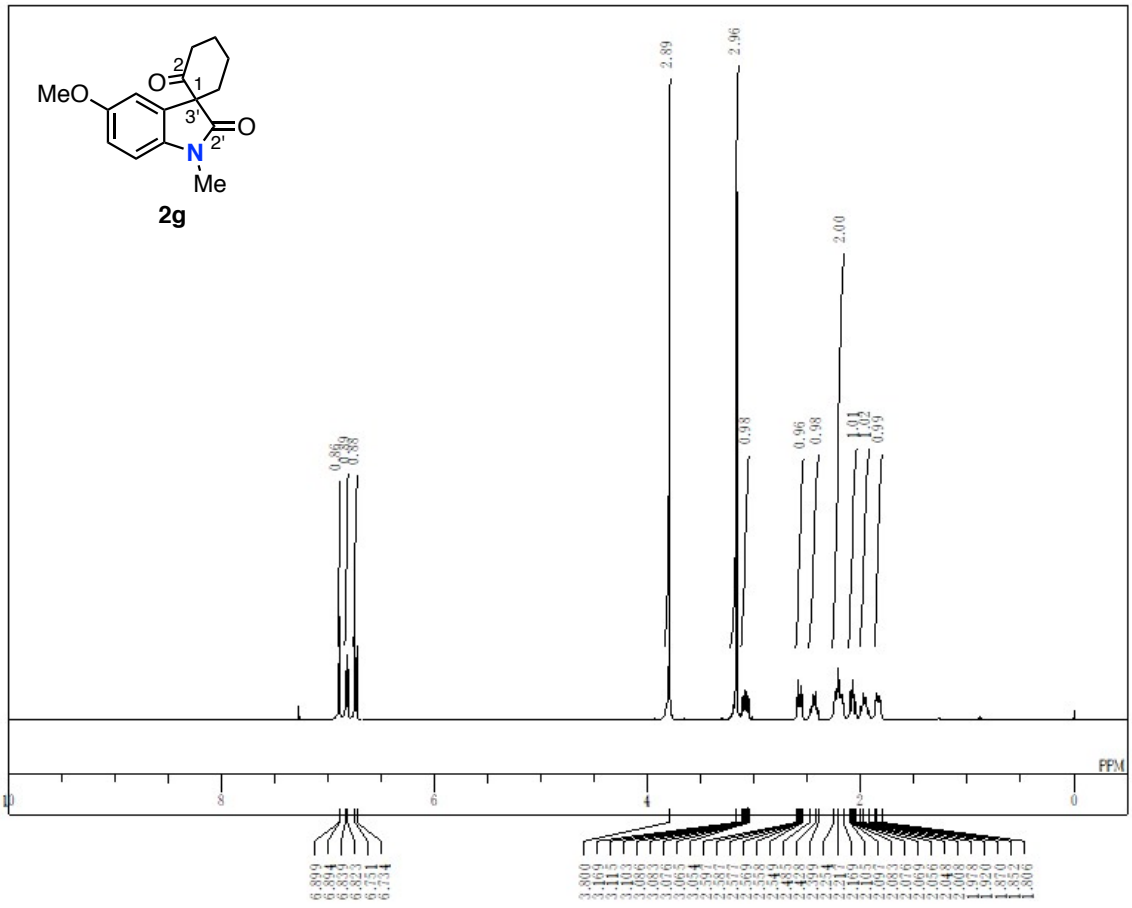
21.291
20.314


MeO
O
O
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Me
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3'
2'
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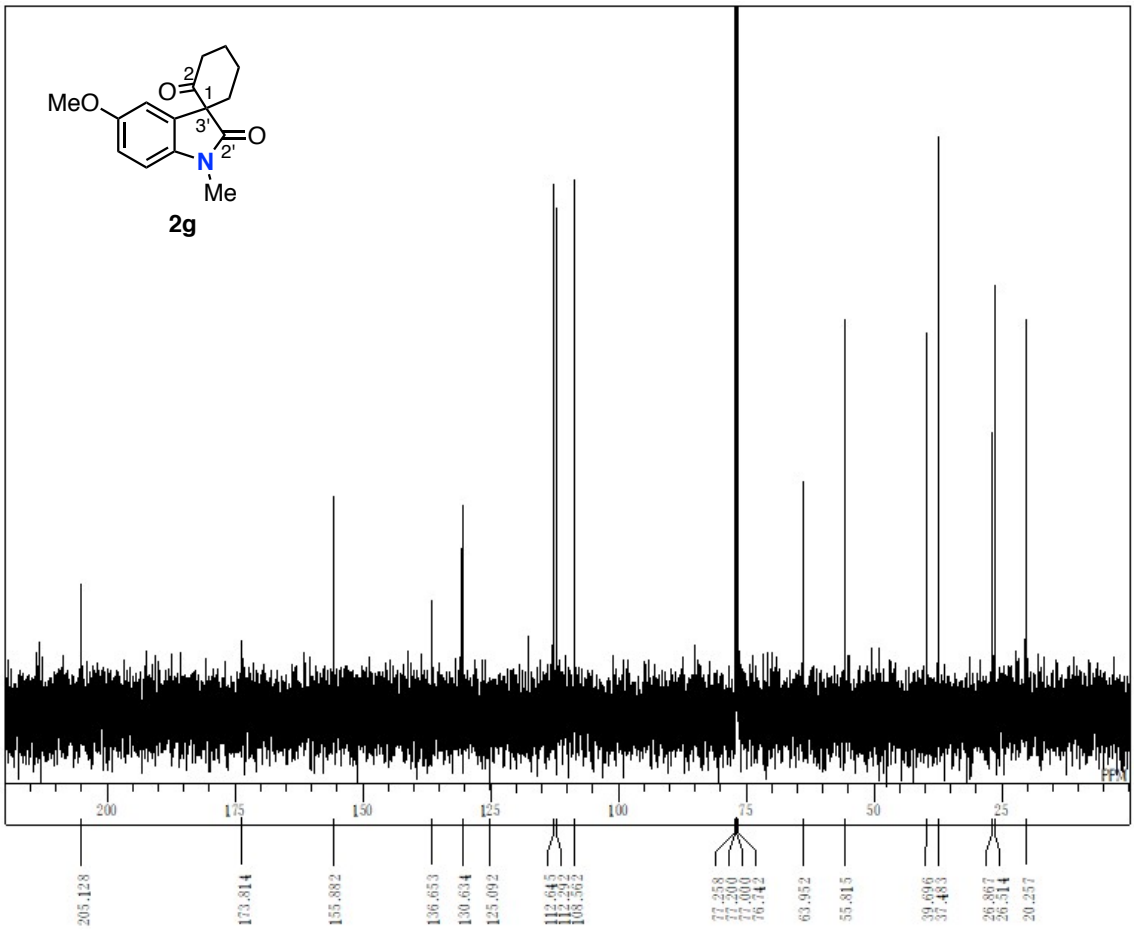
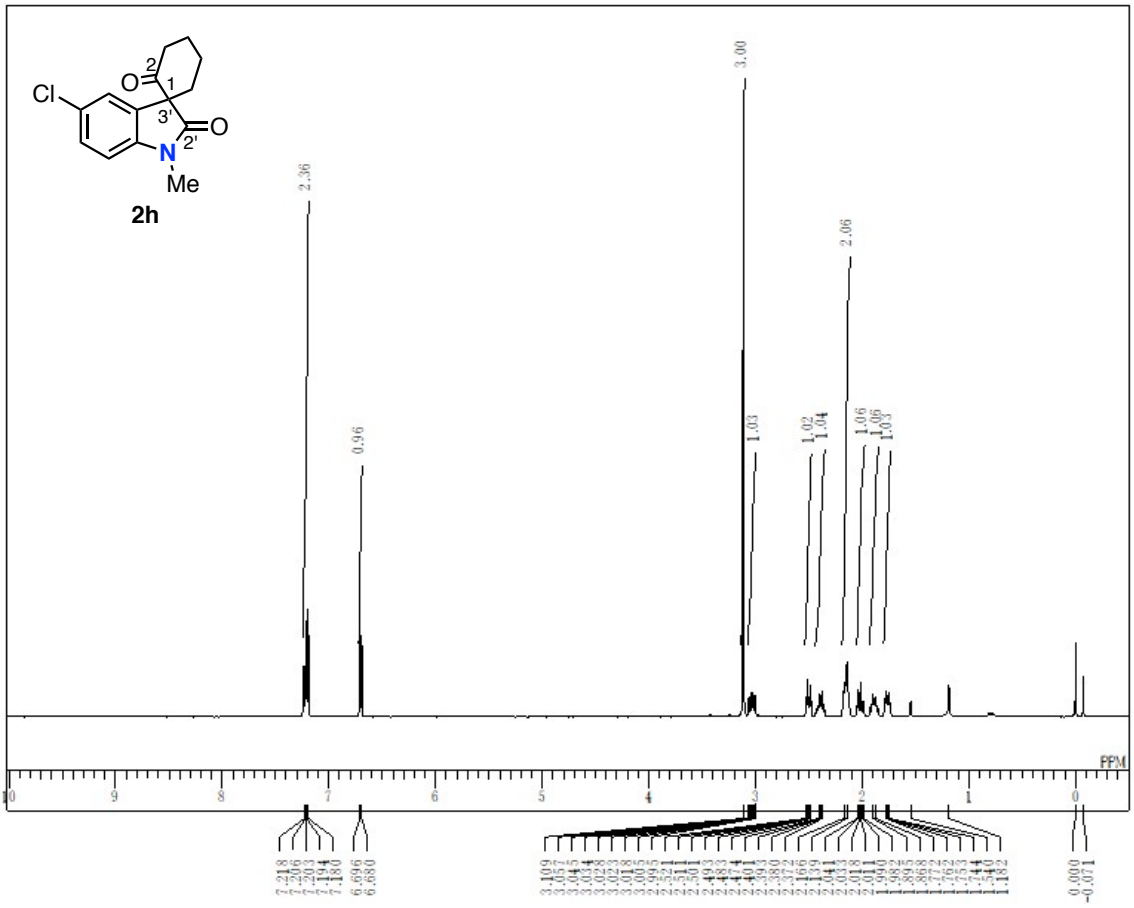
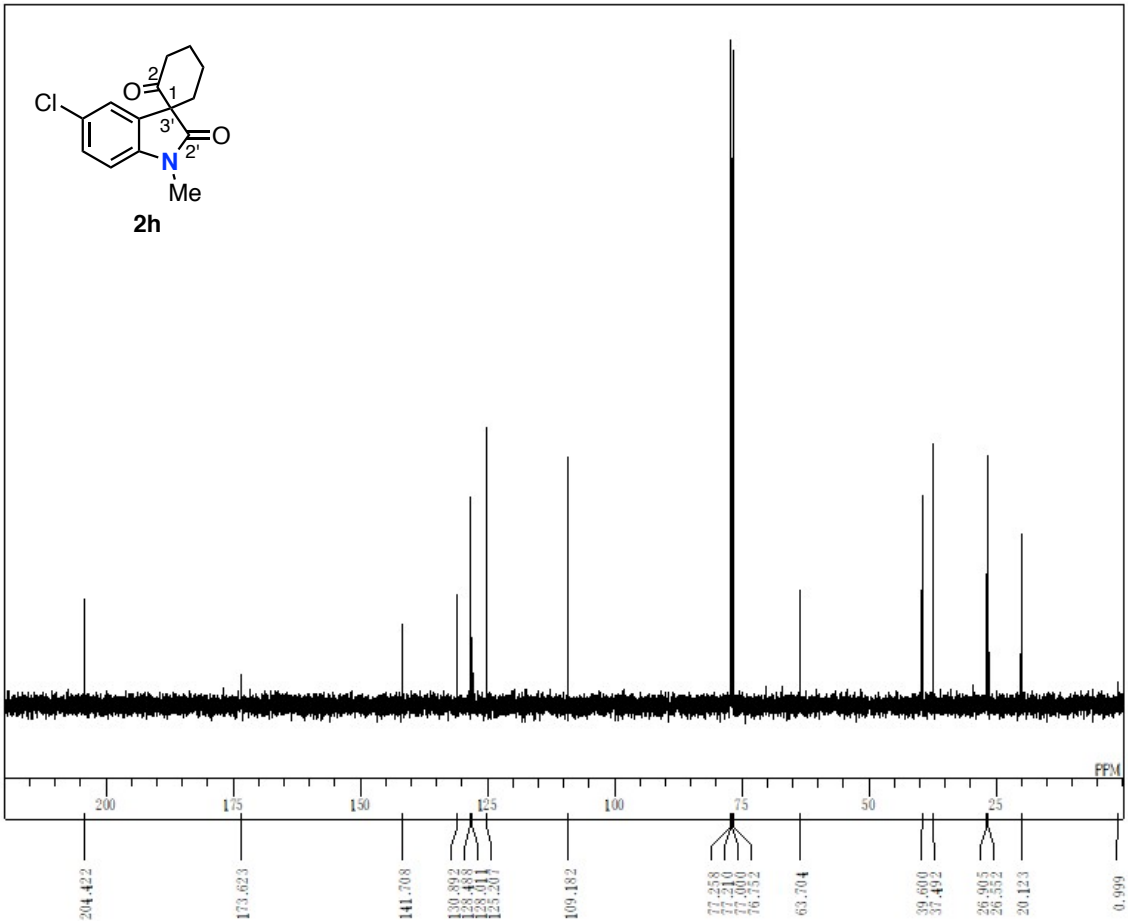
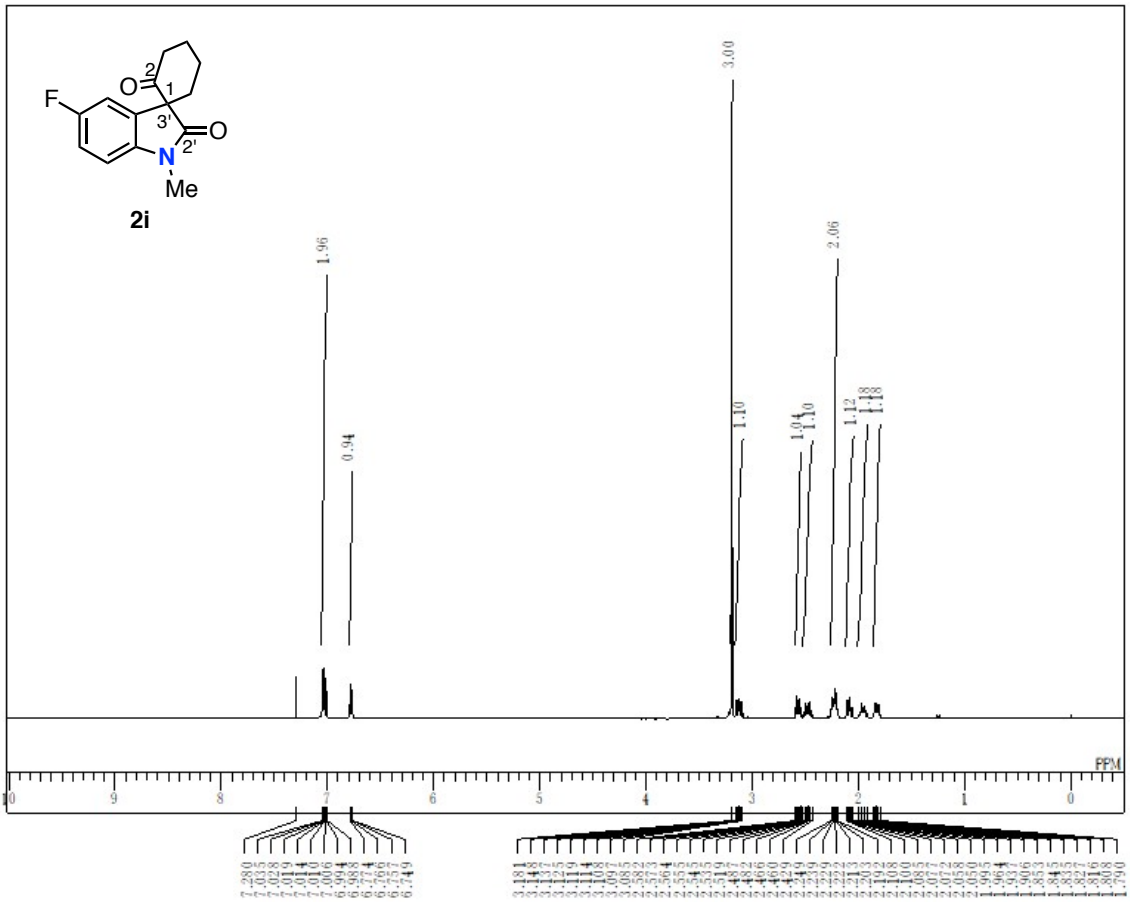
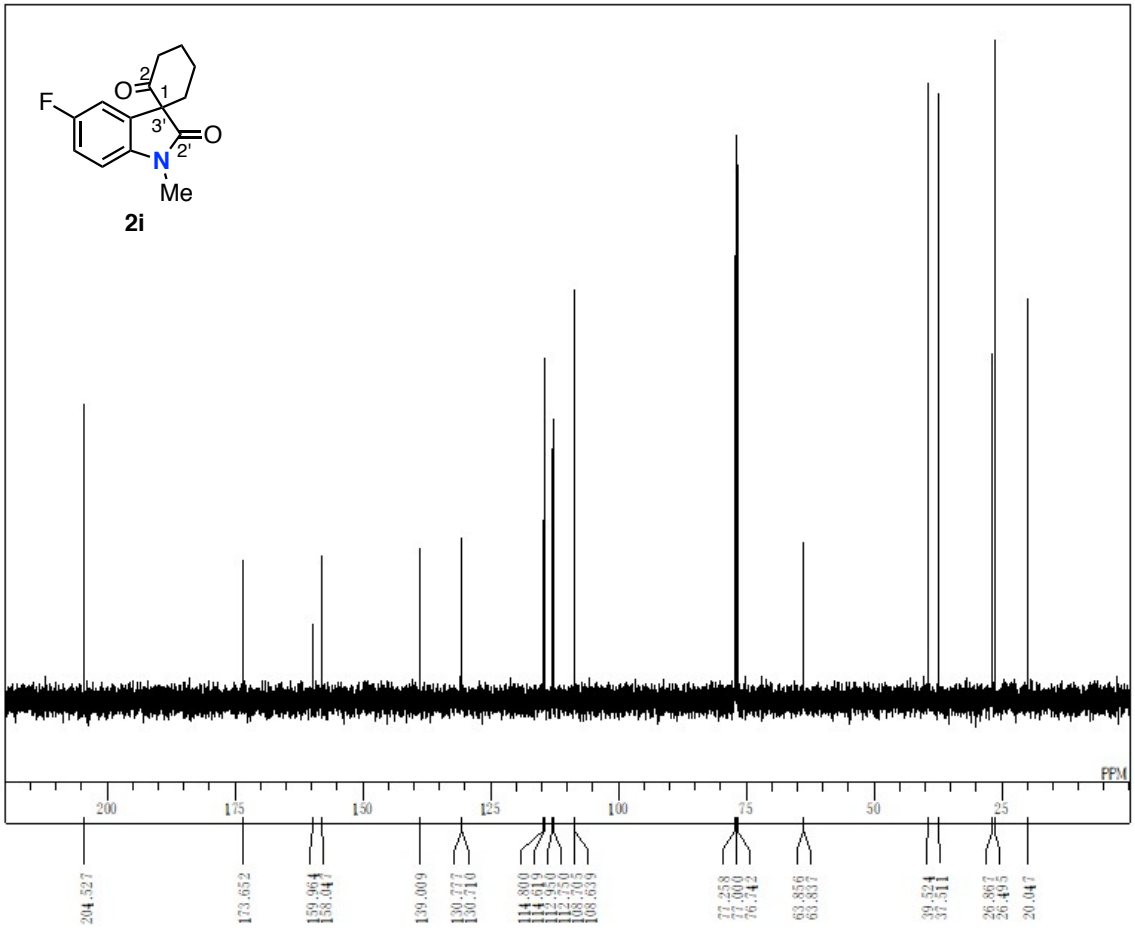
2.96
2.00
0.98
0.96
0.98
1.01
1.02
0.99
0.86
0.89
0.88
PPM
10
8
6
4
2
0

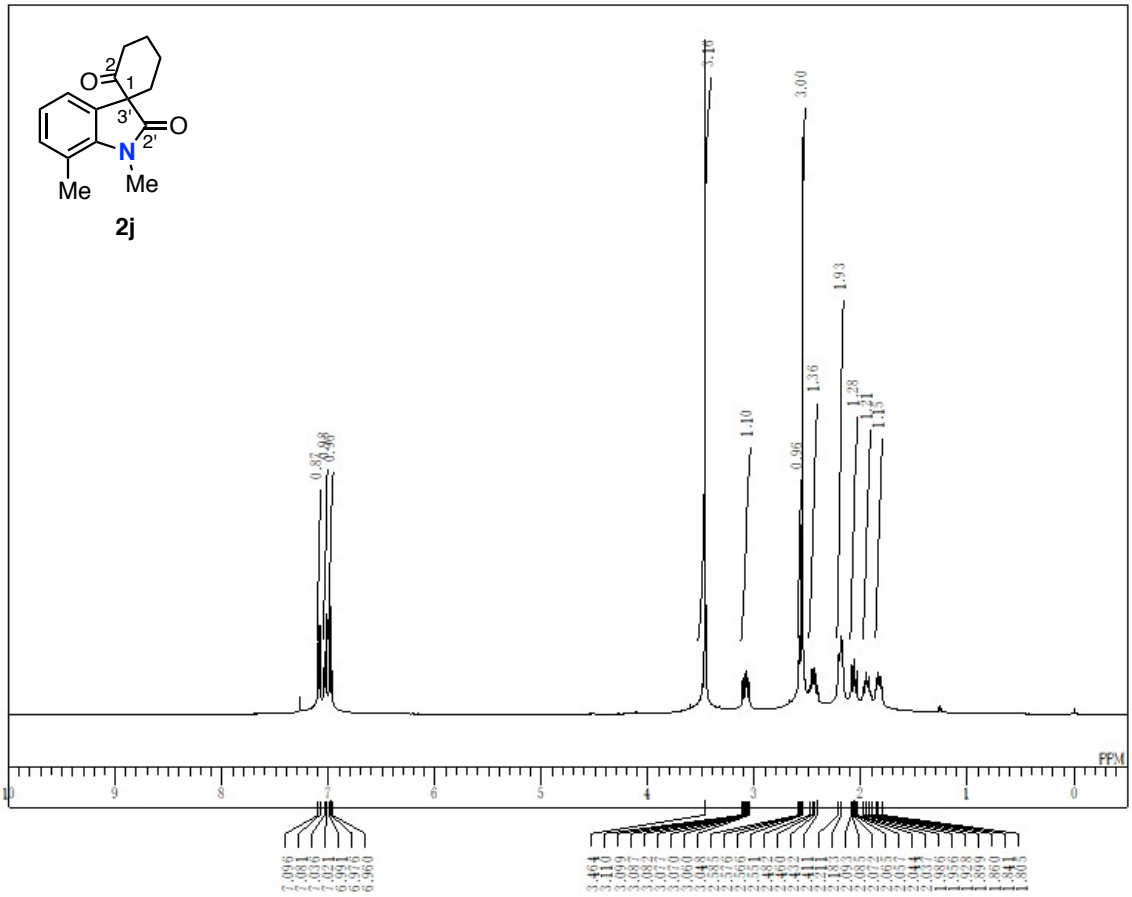

MeO
O
O
N
Me
1
2
3'
2'
2g
PPM
200
175
150
125
100
75
50
25
205.128
173.814
155.882
136.653
130.634
125.092
112.645
112.492
112.242
108.562
77.258
77.200
77.000
76.742
63.952
55.815
39.696
37.483
26.867
26.514
20.257

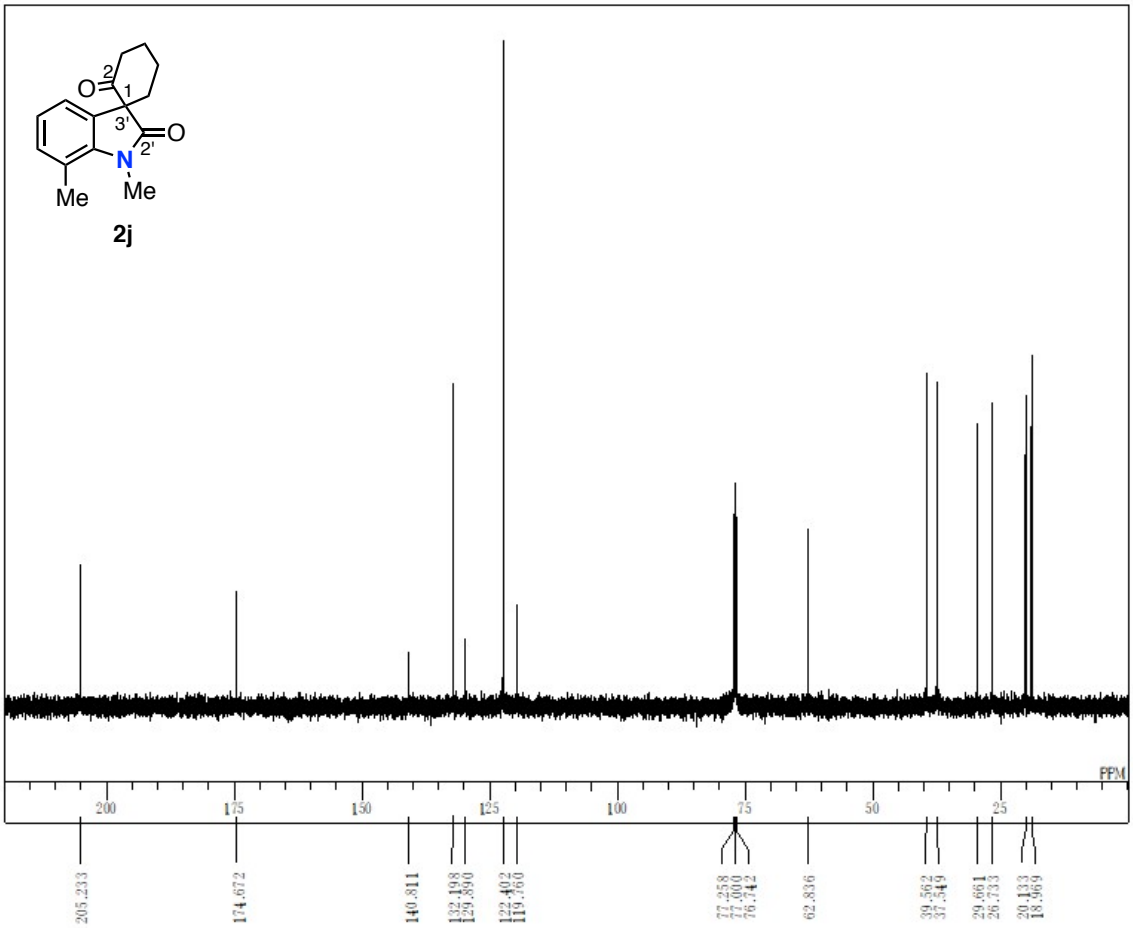

Cl
O
2
1
3'
O
2'
N
Me
2h
2.36
0.96
3.00
1.03
1.02
1.04
2.06
1.06
1.06
1.03
PPM
10
9
8
7
6
5
4
3
2
1
0

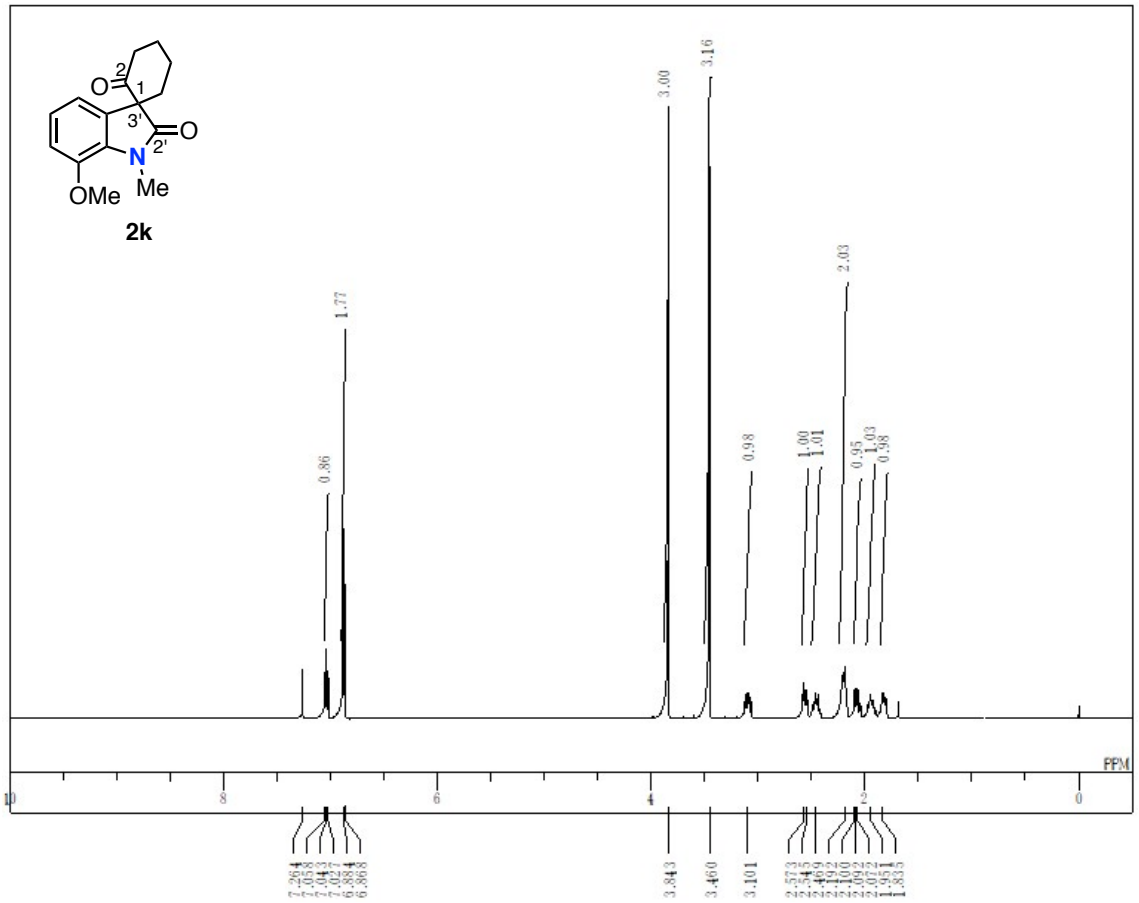

Cl
O
2
1
3'
O
2'
N
Me
2h
PPM
200
175
150
125
100
75
50
25
204.422
173.623
141.708
130.892
128.488
128.011
125.201
109.182
77.258
77.210
77.000
76.752
63.704
39.600
37.492
26.905
26.552
20.123
0.999

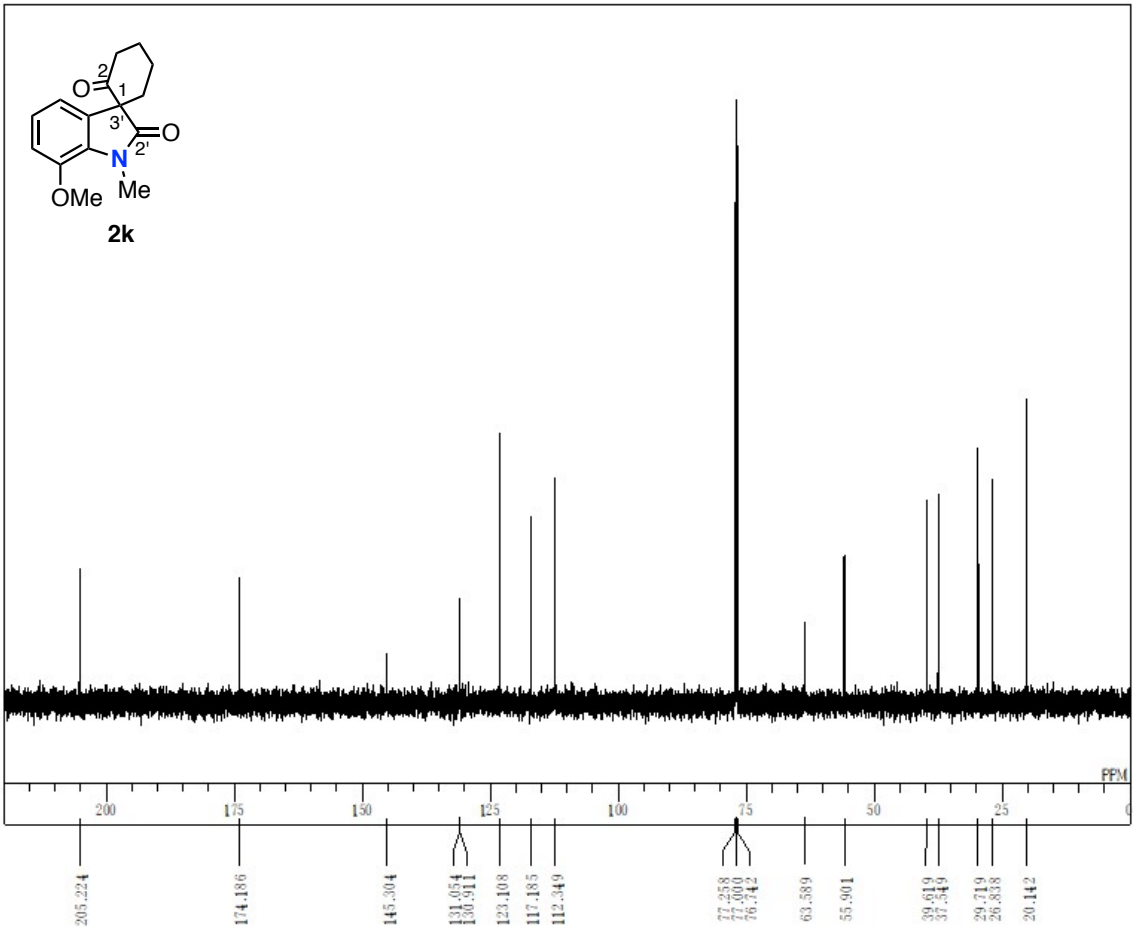

F
O
2
1
3'
O
2'
N
Me
2i
1.96
0.94
3.00
1.10
1.04
1.10
2.06
1.12
1.18
PPM

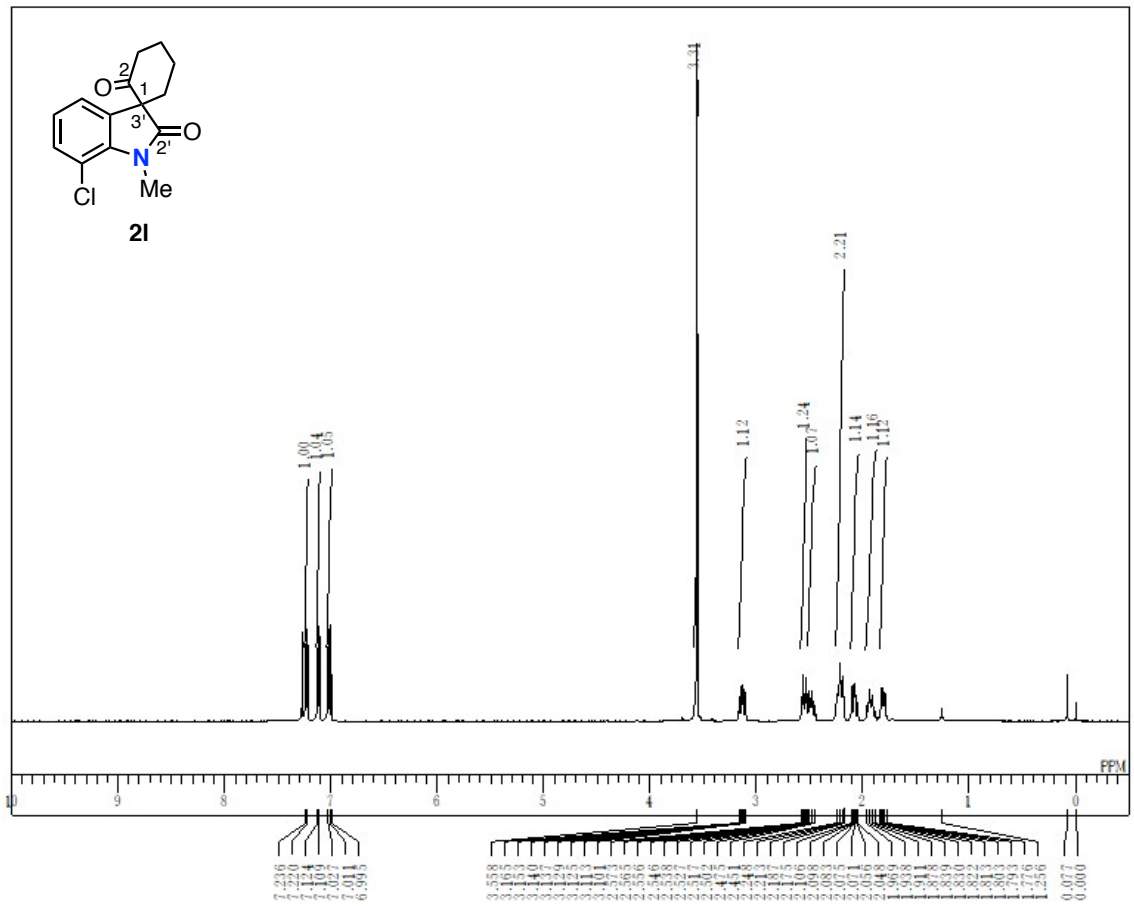

F
O
2
1
3'
O
2'
N
Me
2i
PPM

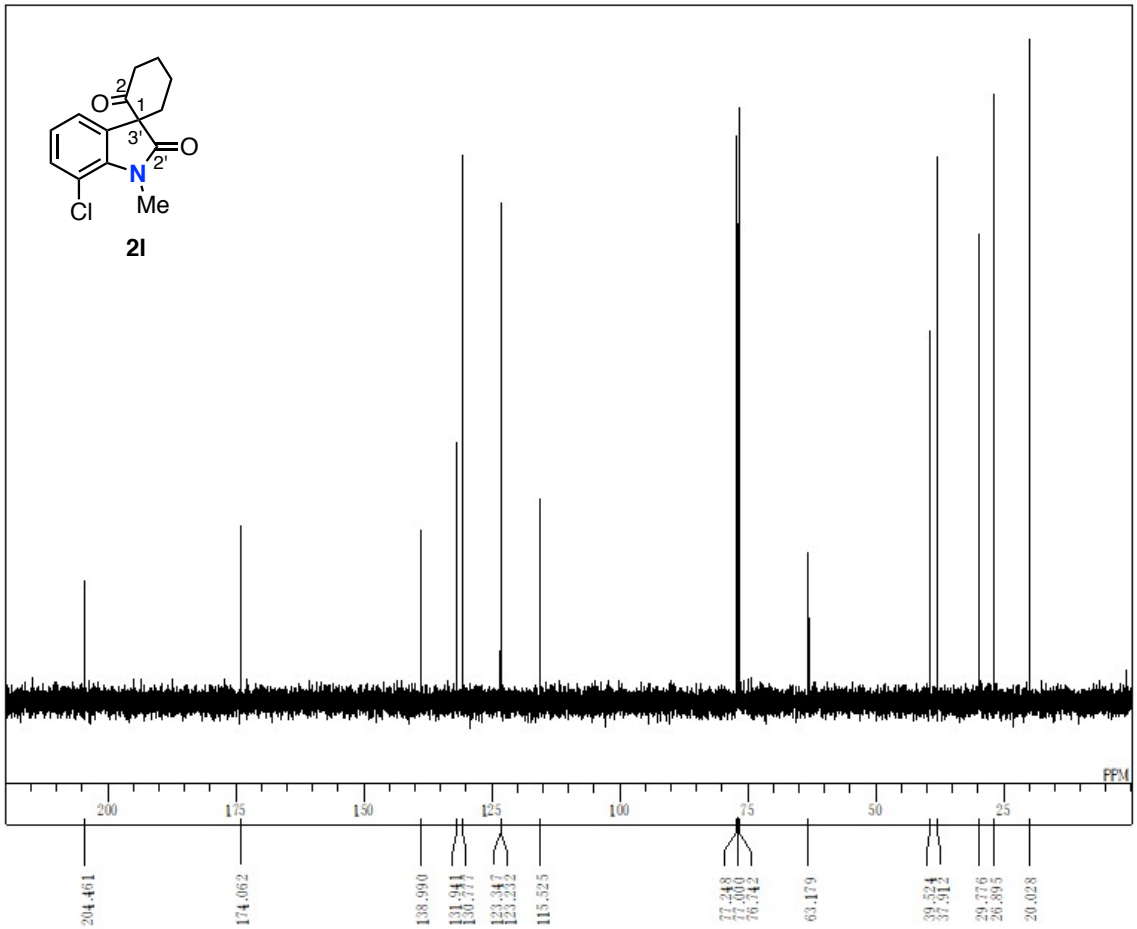

O
2
1
3'
O
2'
N
Me
Me
2j
3.16
3.00
1.93
1.36
1.28
1.10
0.96
PPM

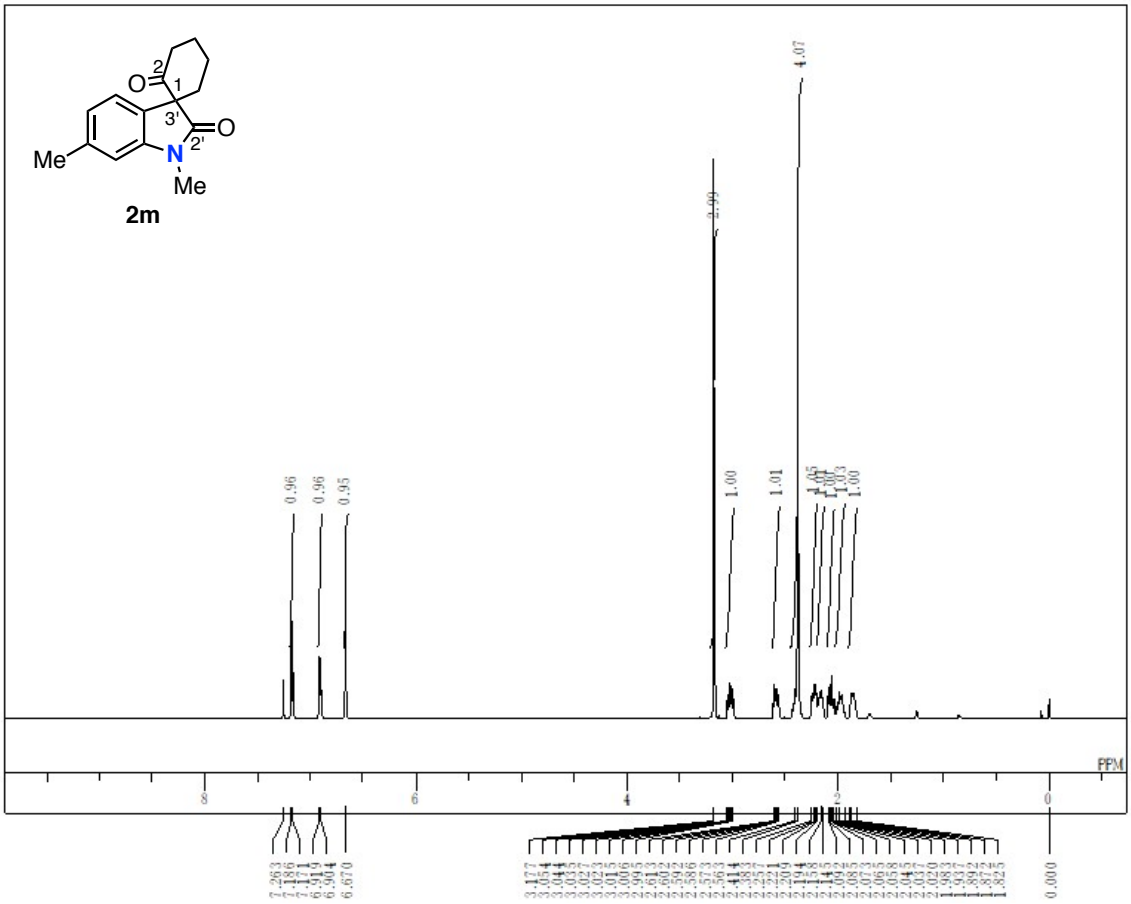

O
2
1
3'
O
2'
N
Me
Me
2j
PPM
205.233
174.672
140.811
132.198
129.890
122.402
119.760
77.258
77.000
76.742
62.836
39.562
37.549
29.661
26.733
20.133
18.969

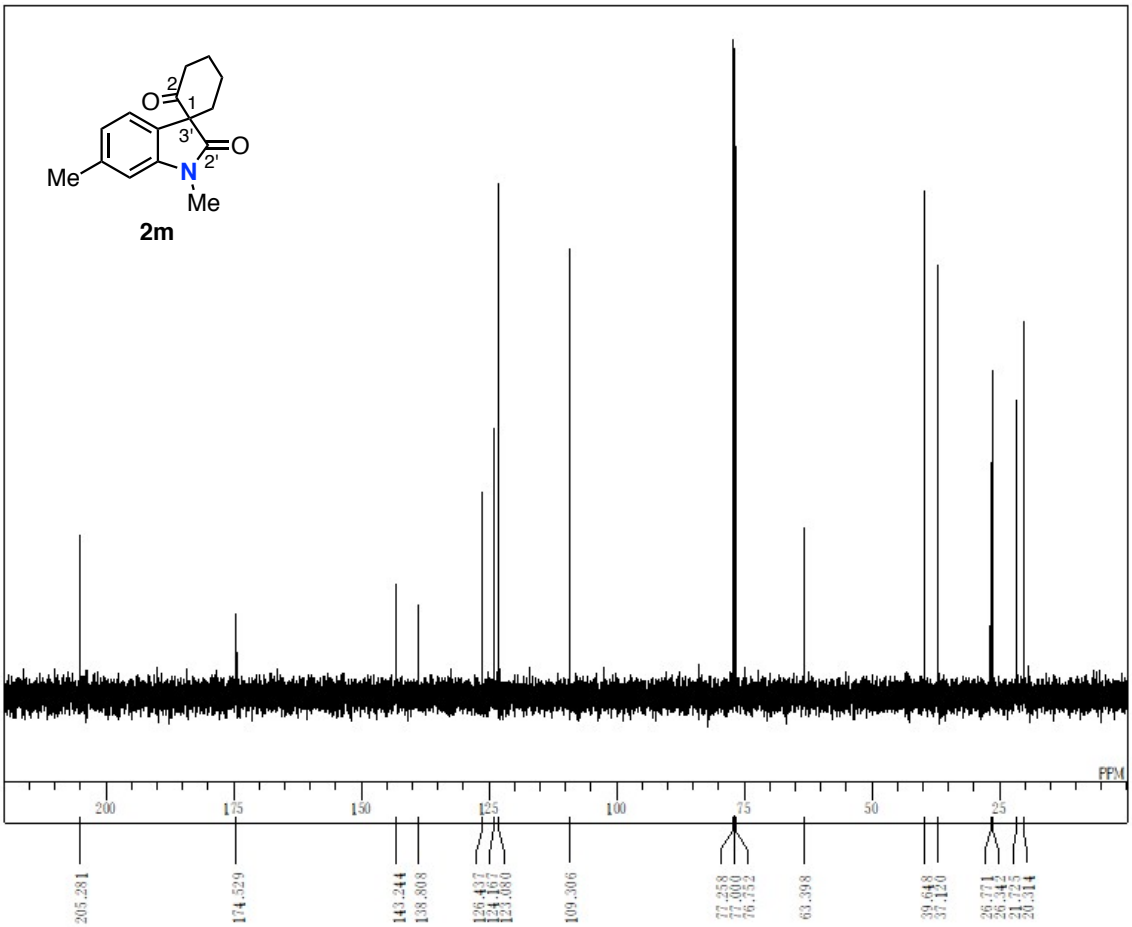

2k
3.00
3.16
1.77
2.03
0.86
0.98
1.00
1.01
0.95
1.03
0.98
PPM
10
8
6
4
2
0
7.264
7.158
7.143
7.027
6.884
6.868
3.843
3.460
3.101
2.573
2.545
2.469
2.192
2.160
2.092
2.062
2.051
1.833

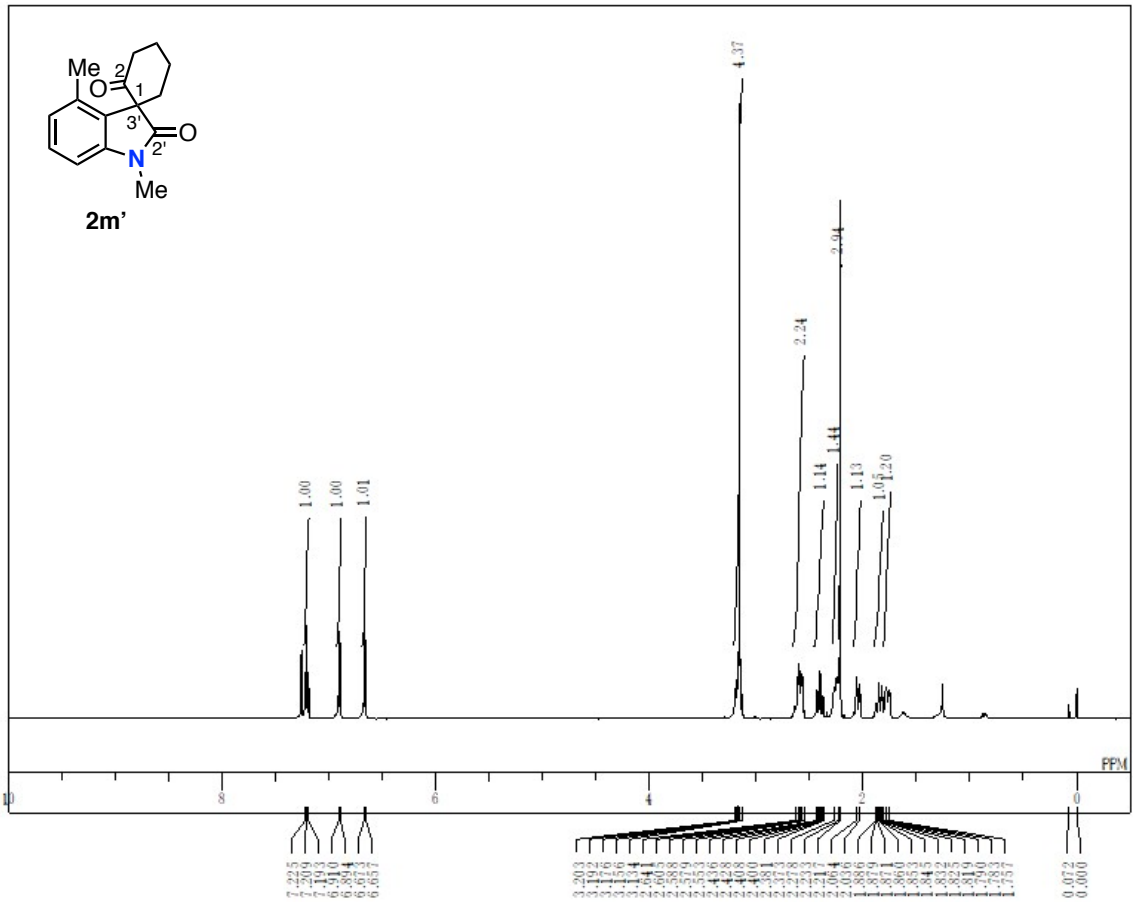

2k
PPM
200
175
150
125
100
75
50
25
205.224
174.186
145.304
131.054
130.911
123.108
117.185
112.349
77.258
77.000
76.742
63.589
55.901
39.619
37.349
29.719
26.838
20.142

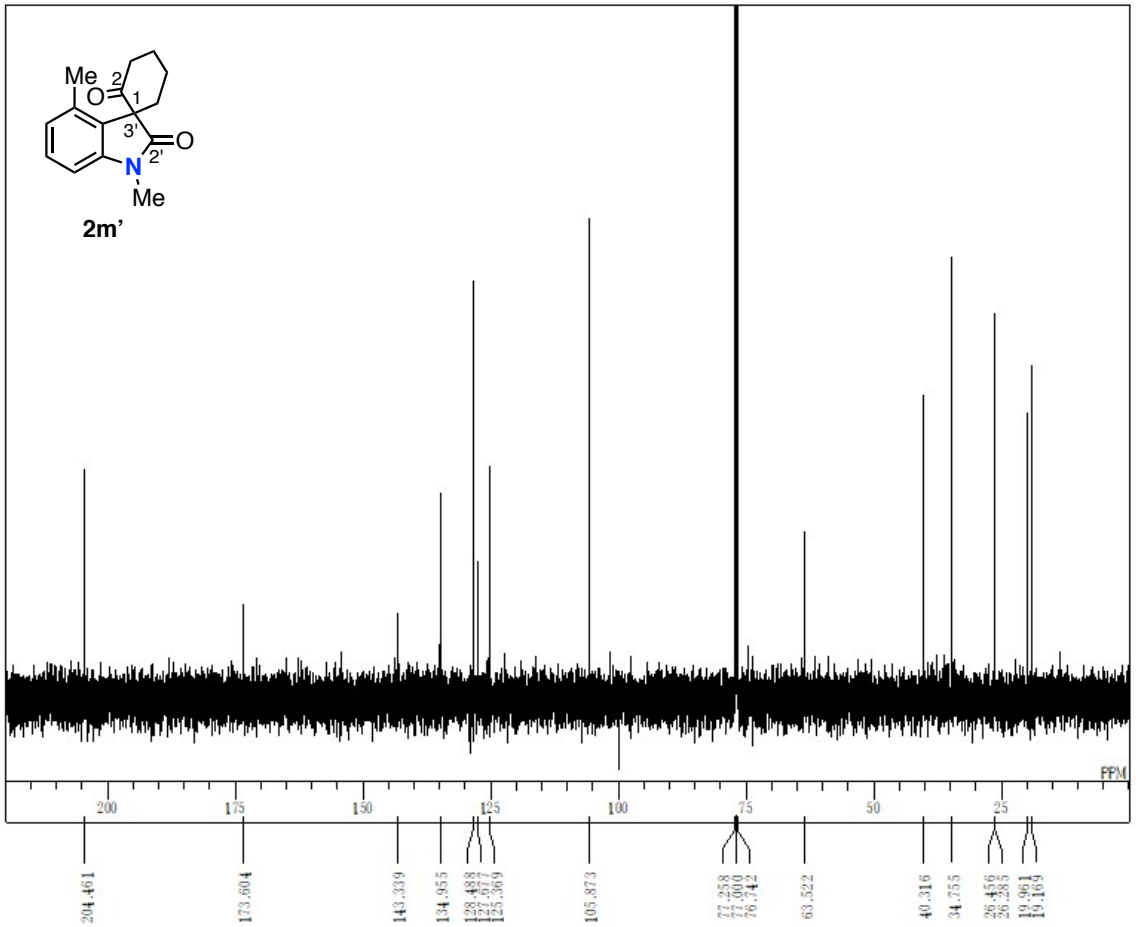

2
1
O
3'
O
2'
N
Me
Cl
2l
3.31
2.21
1.12
1.00
1.04
1.05
1.24
1.14
1.16
1.12
PPM

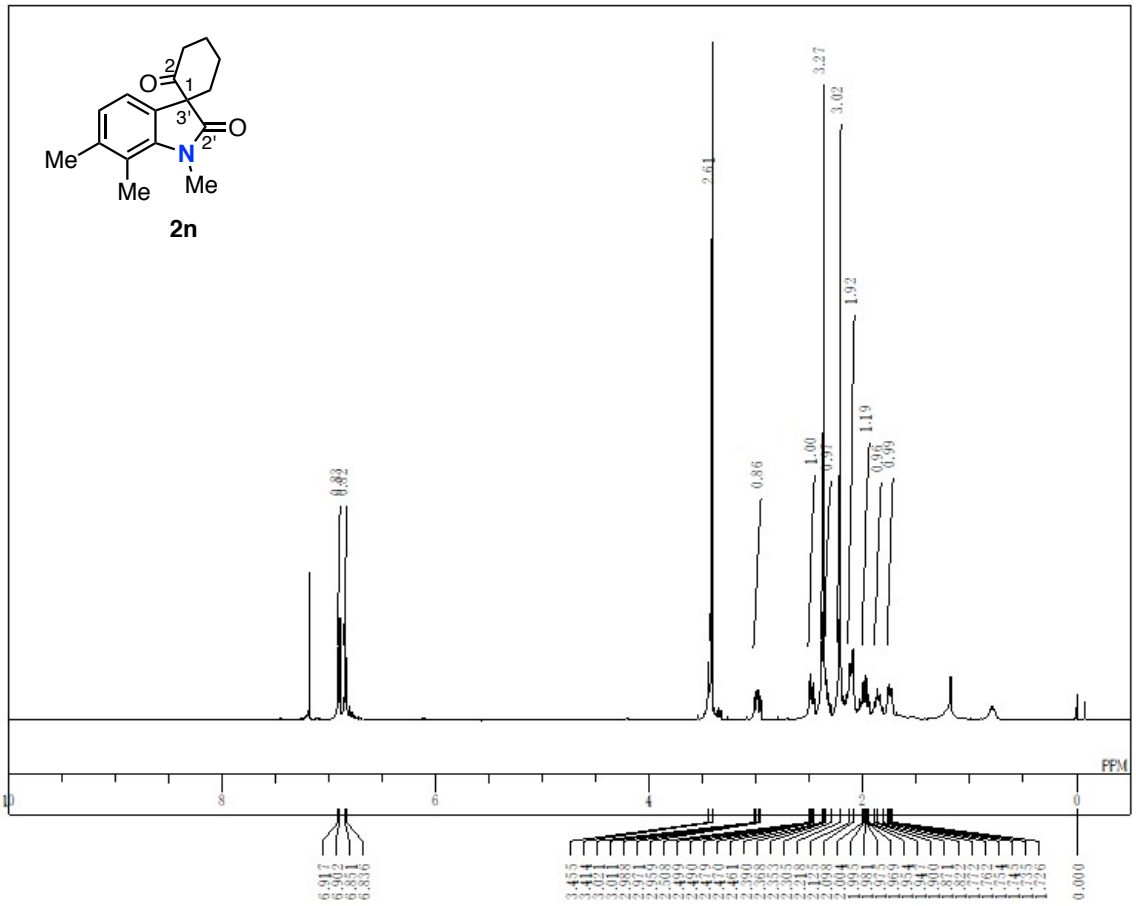

2
1
O
3'
O
2'
N
Me
Cl
2l
PPM
204.461
174.062
138.990
131.941
130.777
123.347
123.232
115.525
77.248
77.000
76.742
63.179
39.524
37.912
29.776
26.895
20.028

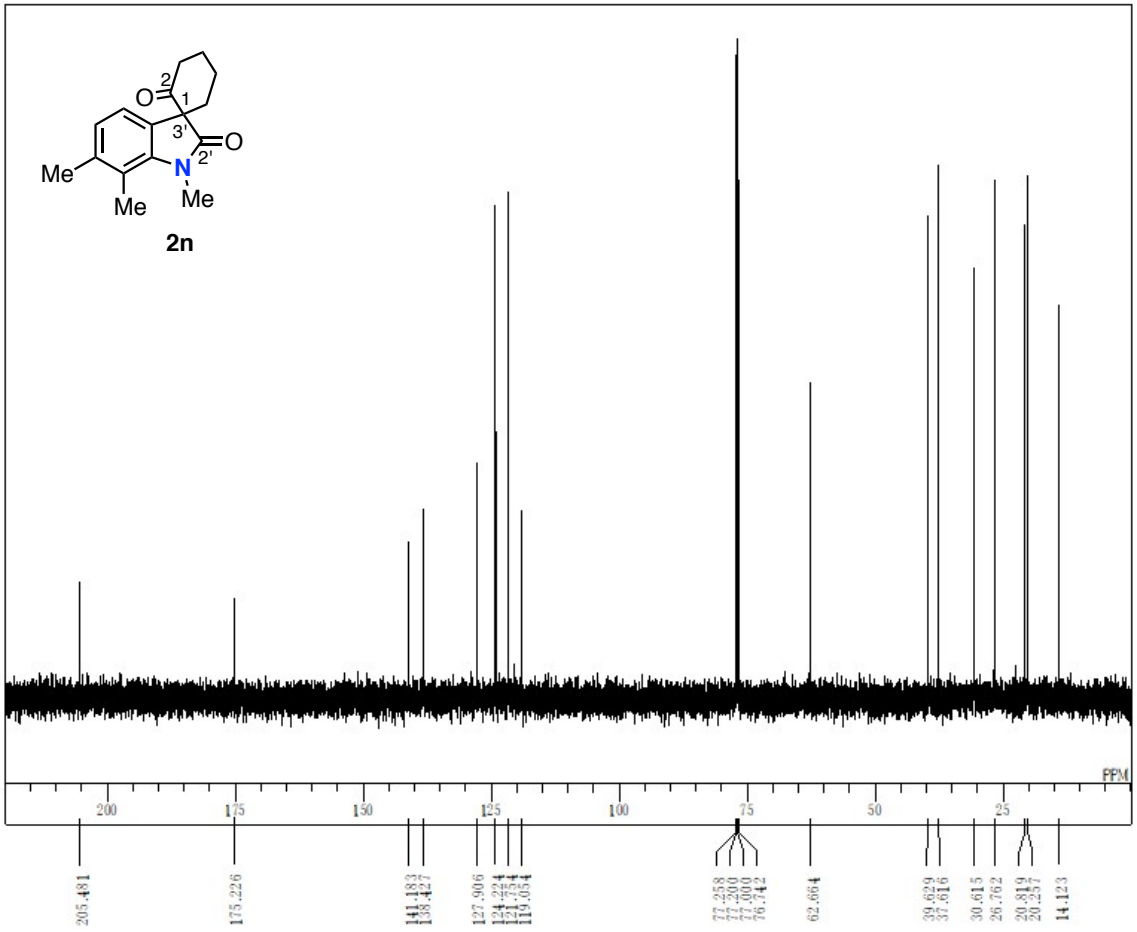

Me
N
Me
2m
PPM

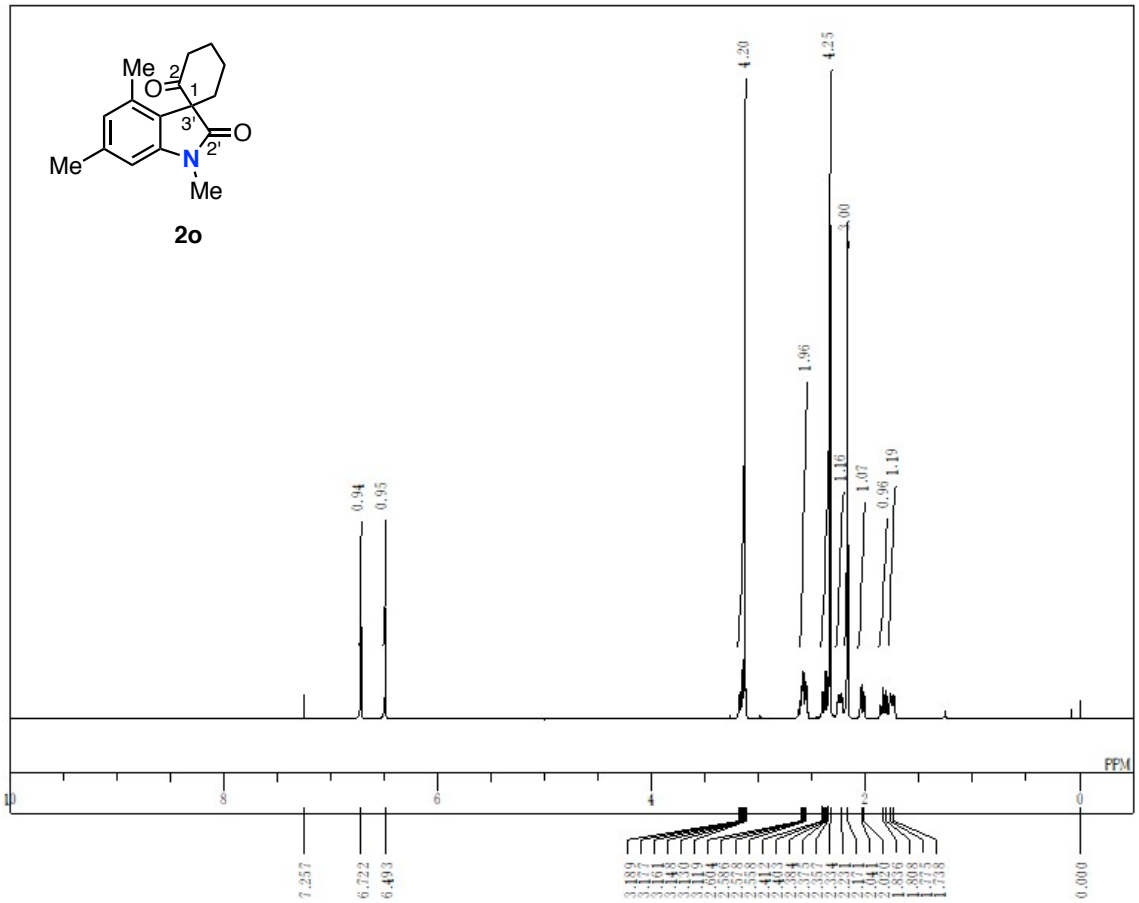

Me
N
Me
2m
PPM


Me
O
2
1
3'
O
2'
N
Me
2m'
4.37
2.24
2.04
1.44
1.14
1.13
1.05
1.20
1.00
1.00
1.01
PPM
10
8
6
4
2
0

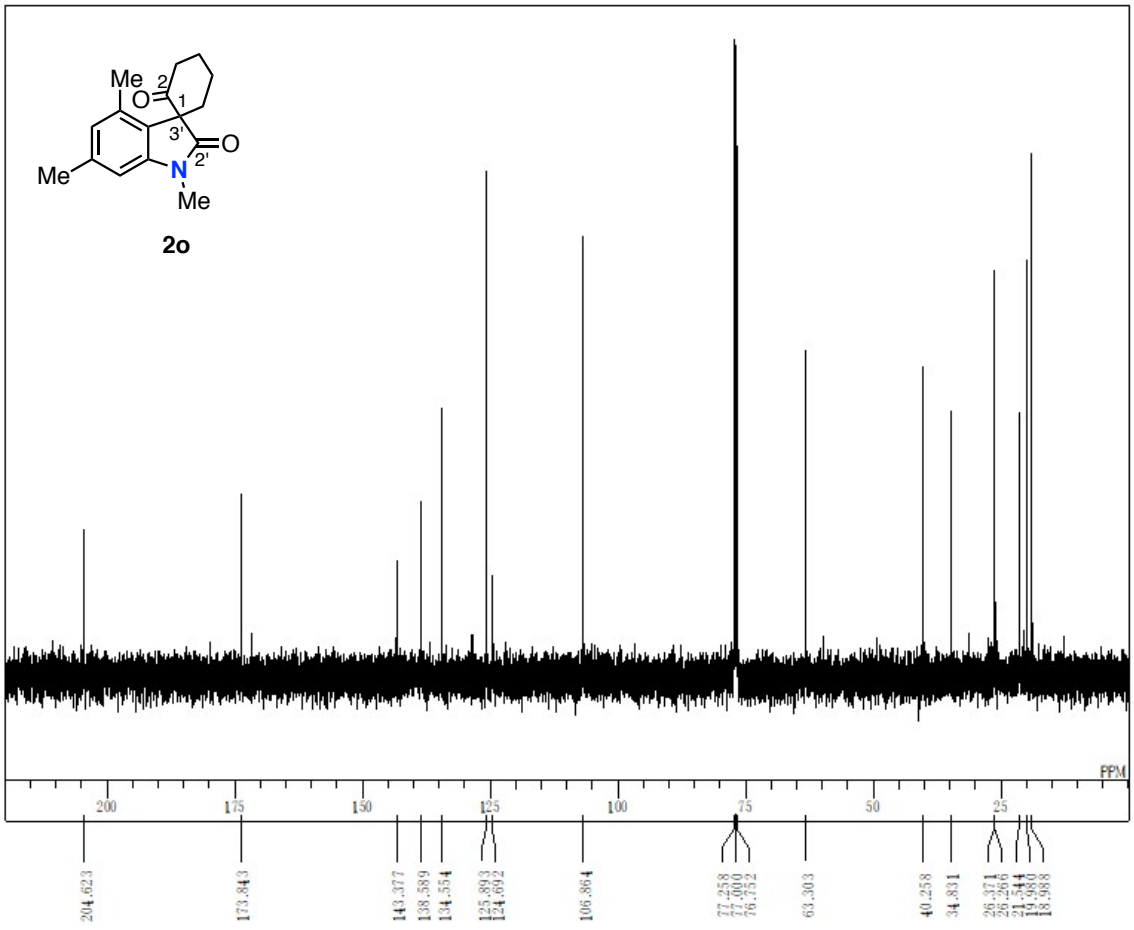

Me
O
2
1
3'
O
2'
N
Me
2m'
PPM
200
175
150
125
100
75
50
25
204.461
173.604
143.339
134.955
128.488
127.677
125.369
105.873
77.258
77.000
76.742
63.522
40.316
34.755
26.456
26.285
19.961
19.169


2n
PPM

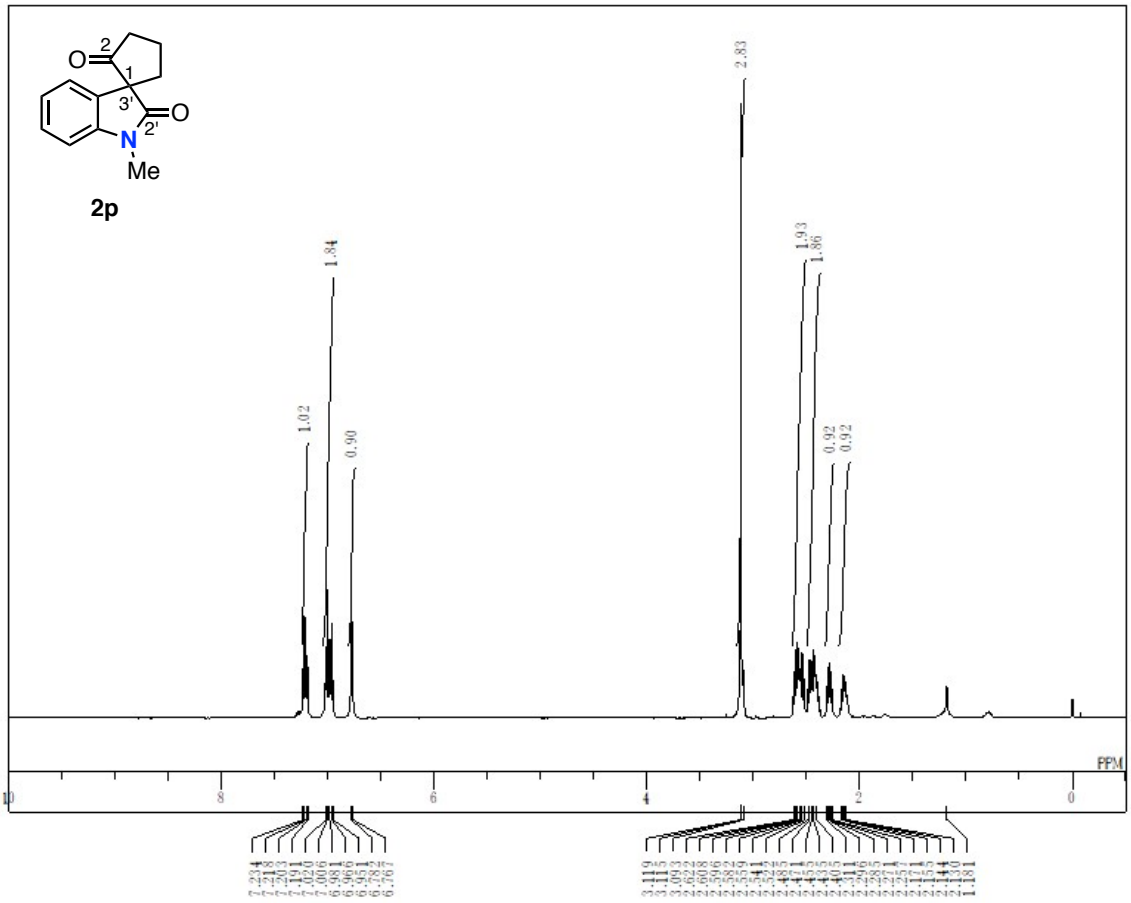

2n
PPM

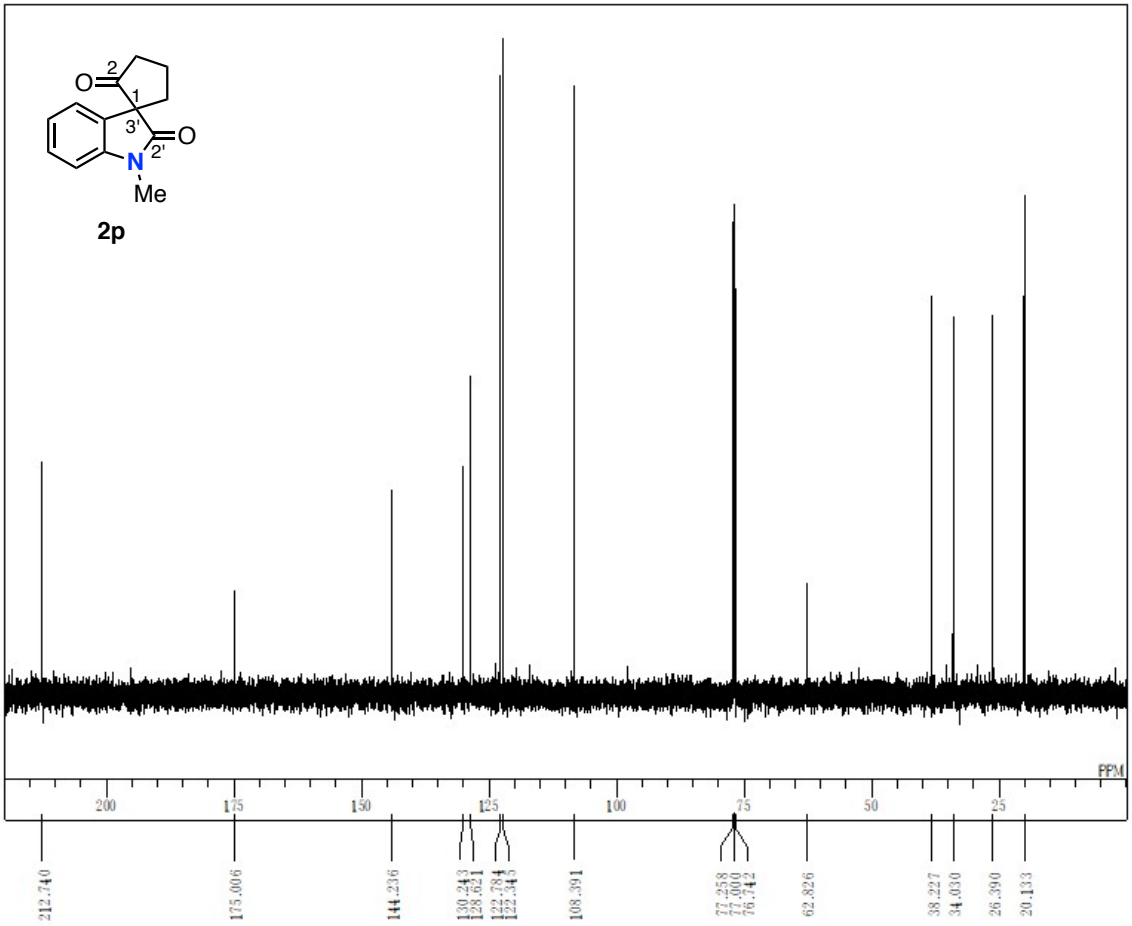

Me
O
2
1
3'
O
2'
N
Me
Me
4.20
4.25
3.00
1.96
1.16
1.07
0.96
1.19
0.94
0.95
PPM
10
8
6
4
2
0
7.257
6.722
6.493
0.000

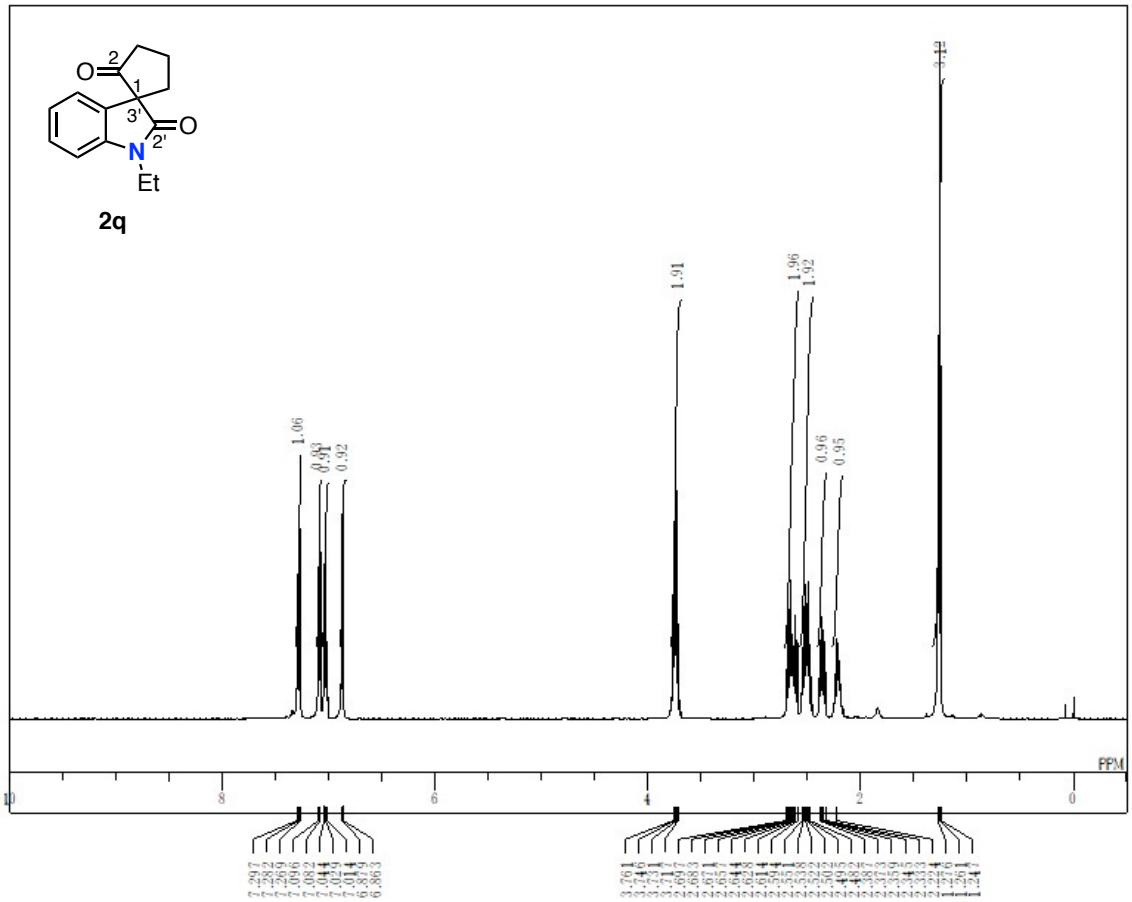
2o

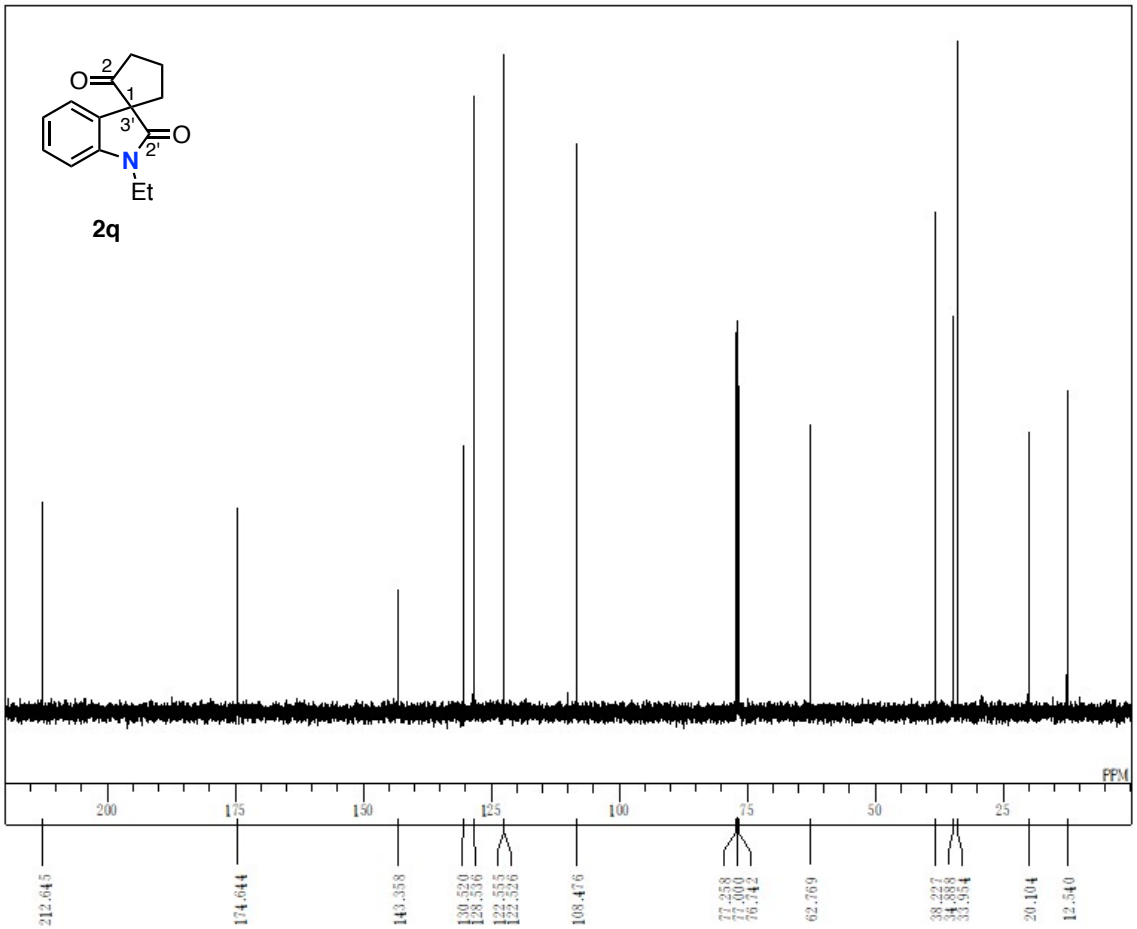

Me
O
2
1
3'
O
2'
N
Me
Me
PPM
200
175
150
125
100
75
50
25
204.623
173.843
143.377
138.589
134.554
125.893
124.692
106.864
77.258
77.000
76.732
63.303
40.258
34.831
26.371
26.266
21.544
19.980
18.988

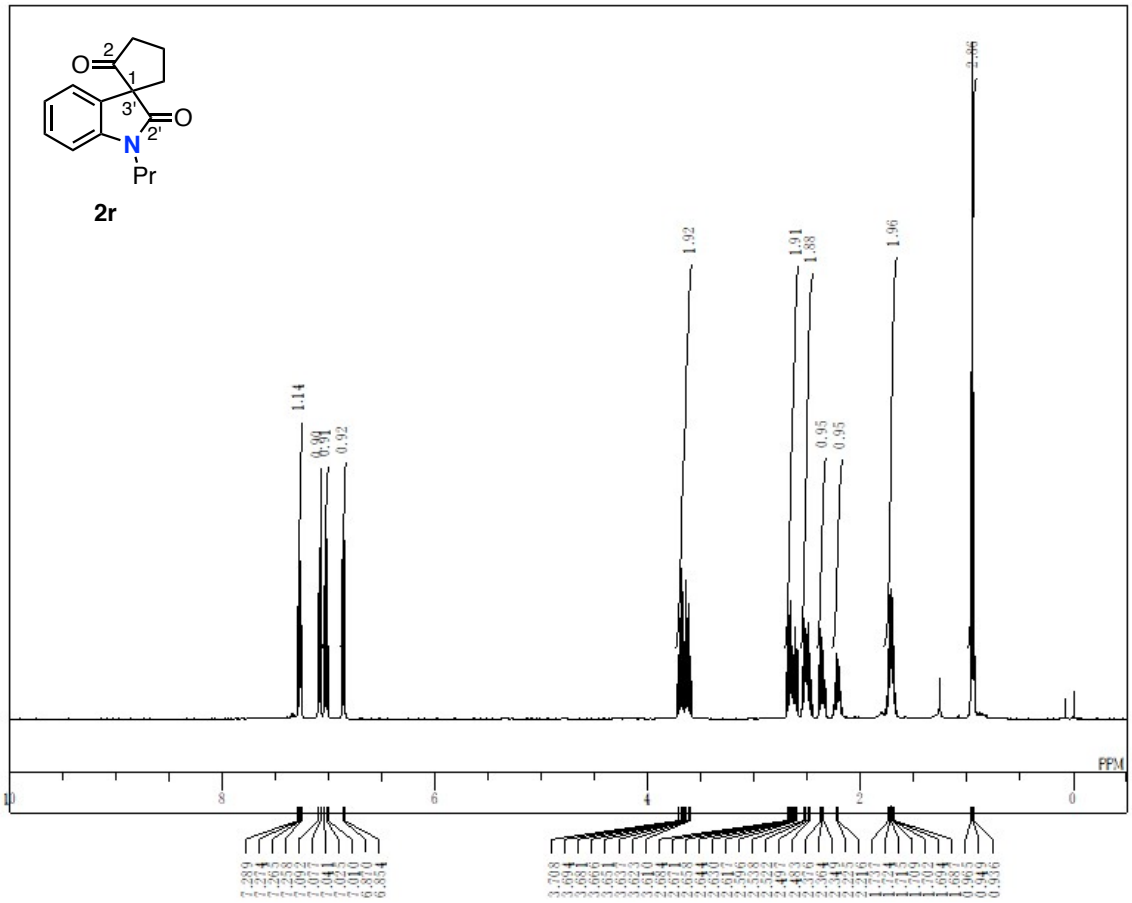
2o

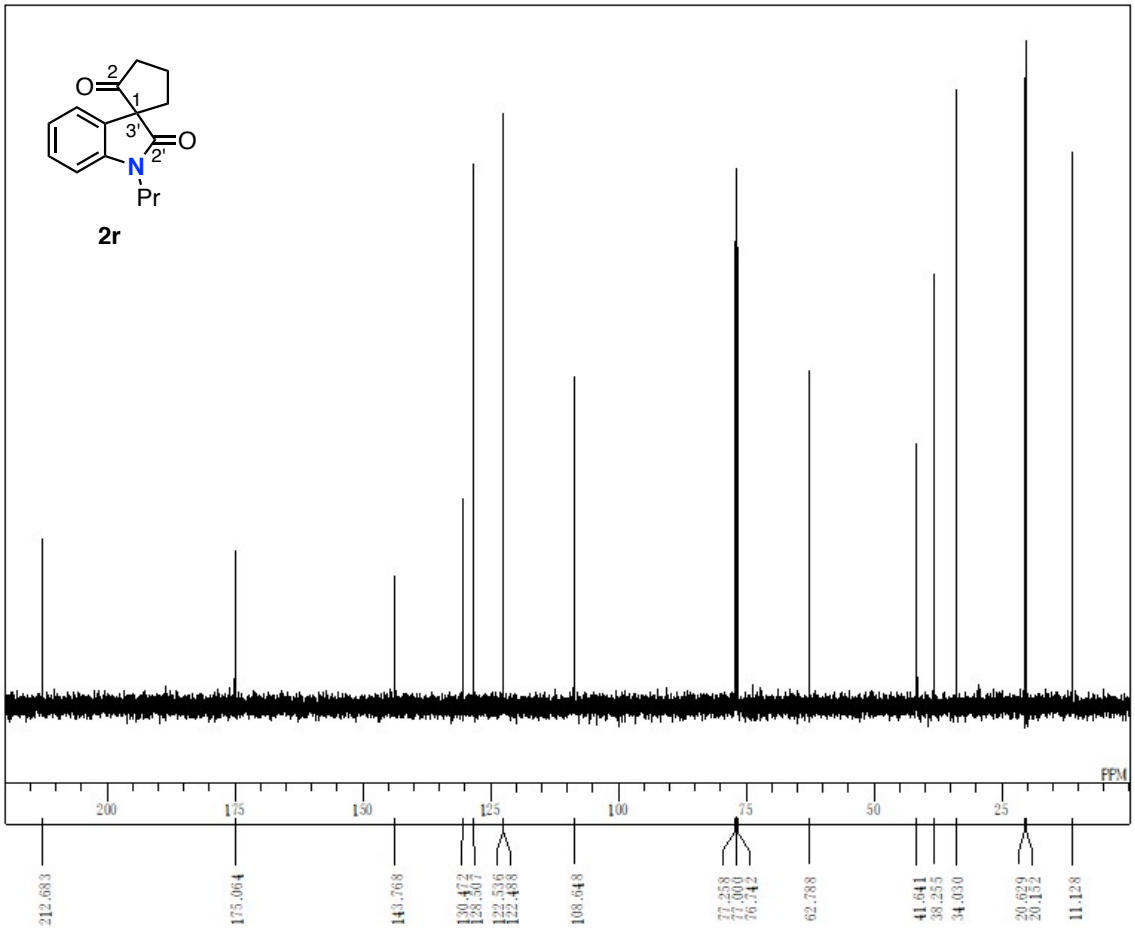

2p
1.02
1.84
0.90
2.83
1.93
1.86
0.92
0.92
PPM

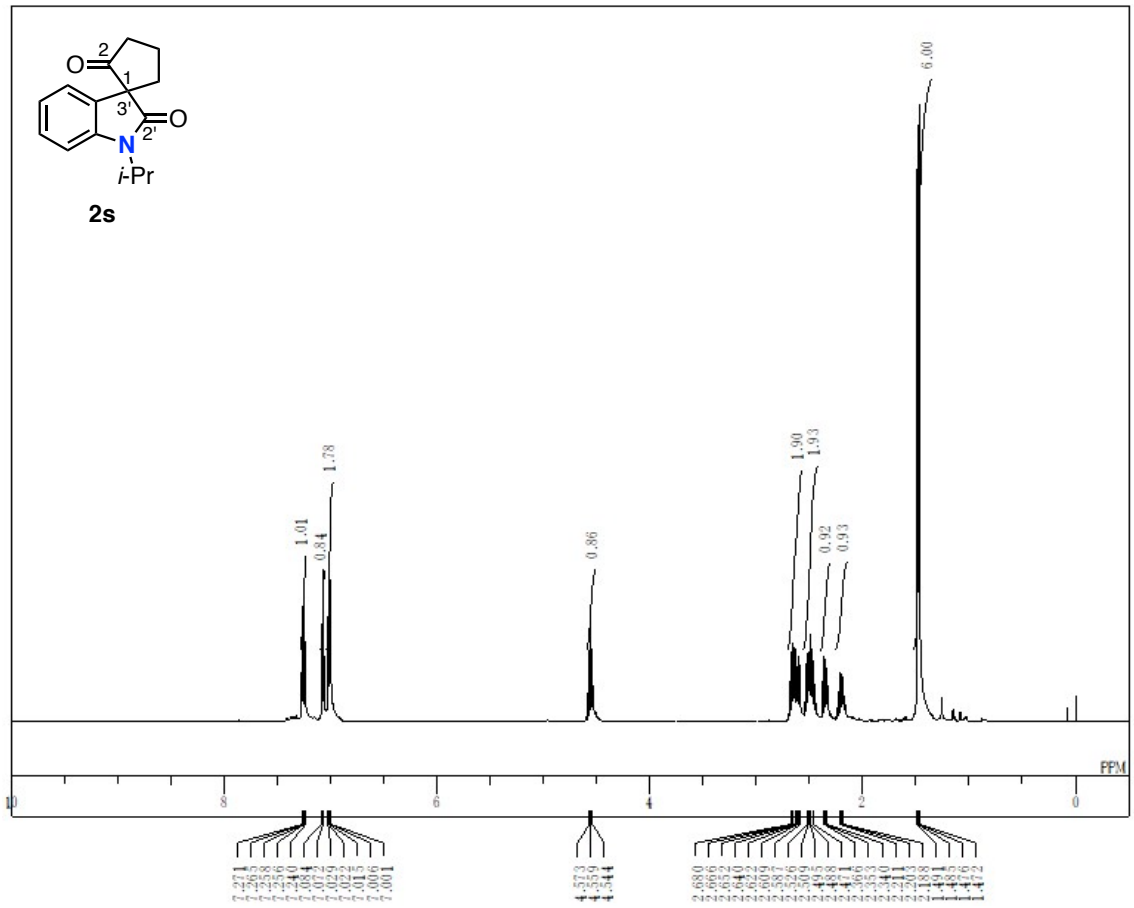

2p
PPM

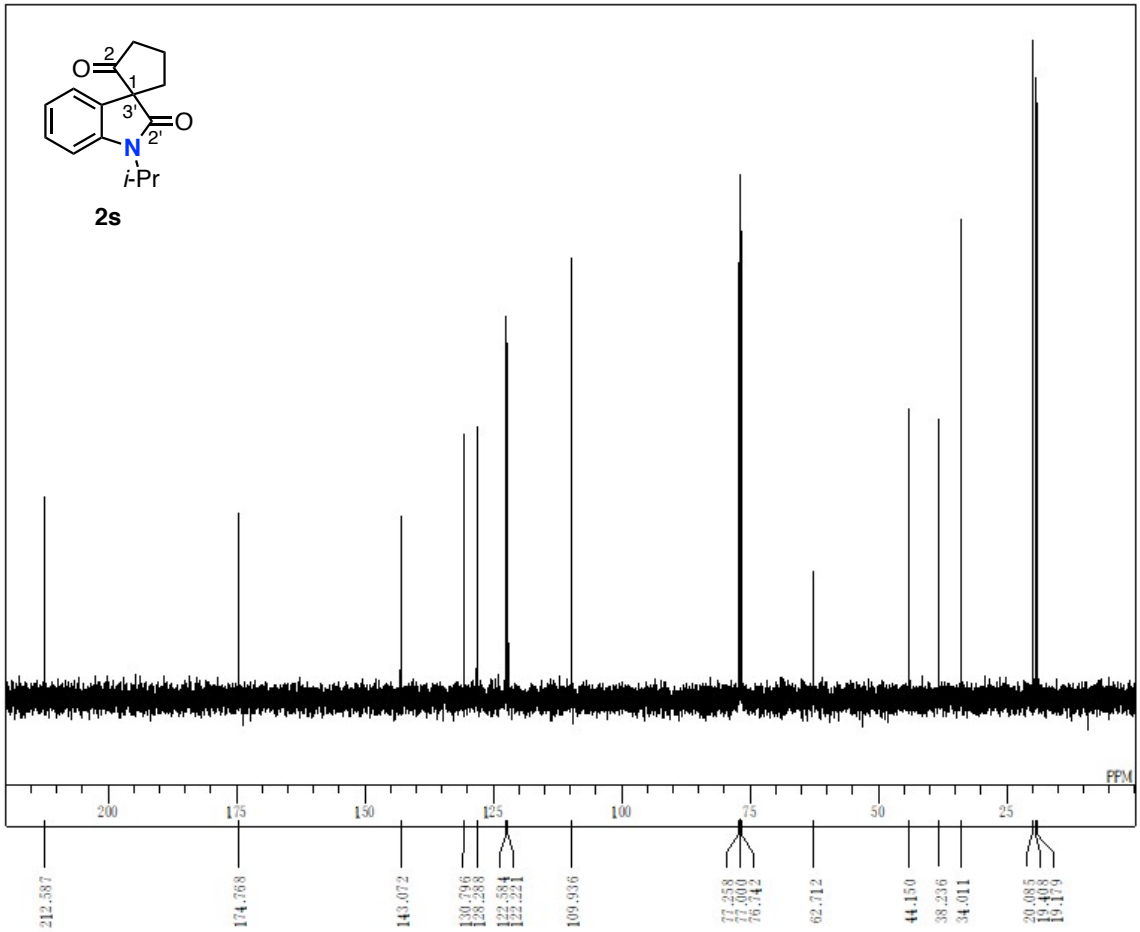

2q
1.06
1.03
1.01
0.92
1.91
1.96
1.92
0.96
0.95
3.12
PPM

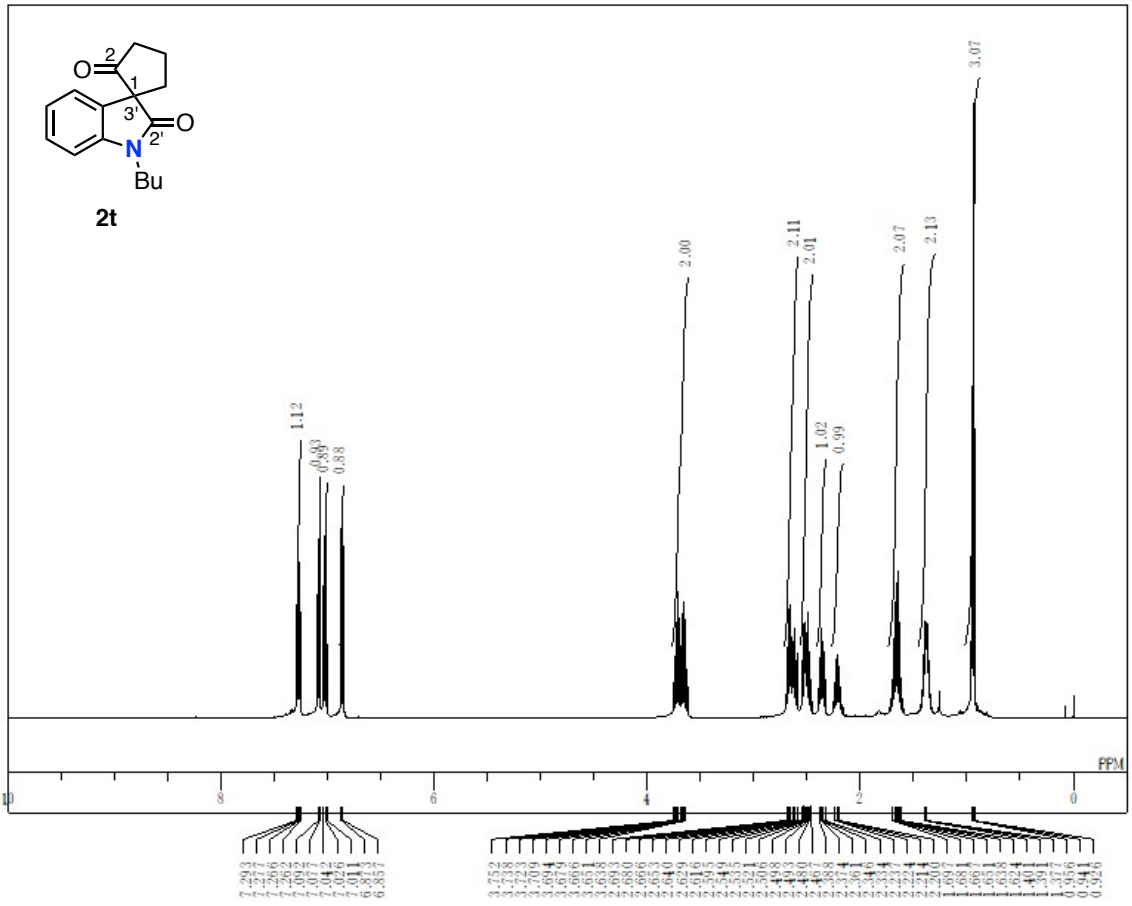

2q
PPM
212.645
174.644
143.358
130.520
128.356
122.555
122.526
108.476
77.258
77.000
76.742
62.769
38.227
34.888
33.954
20.104
12.540

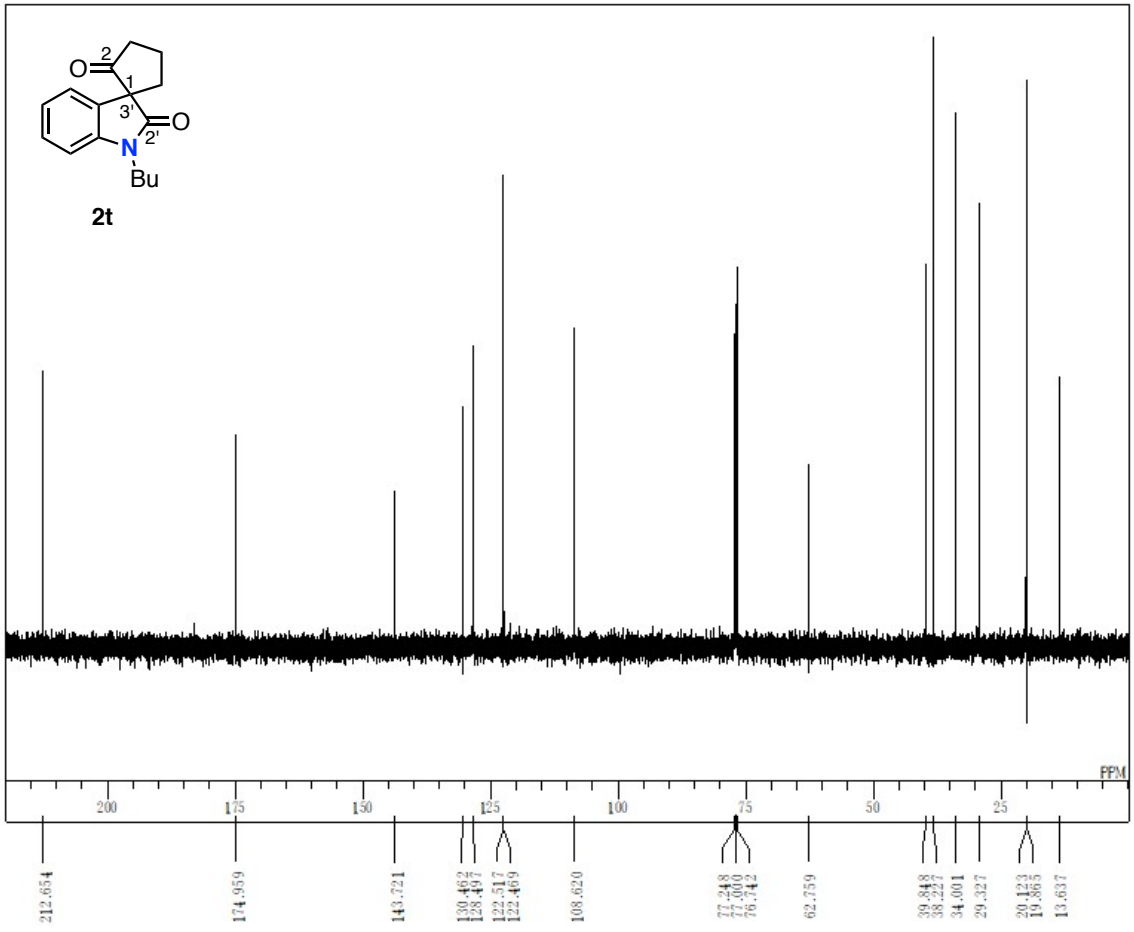

2r
1.14
0.91
0.92
1.92
1.91
1.88
0.95
0.95
1.96
2.86
PPM

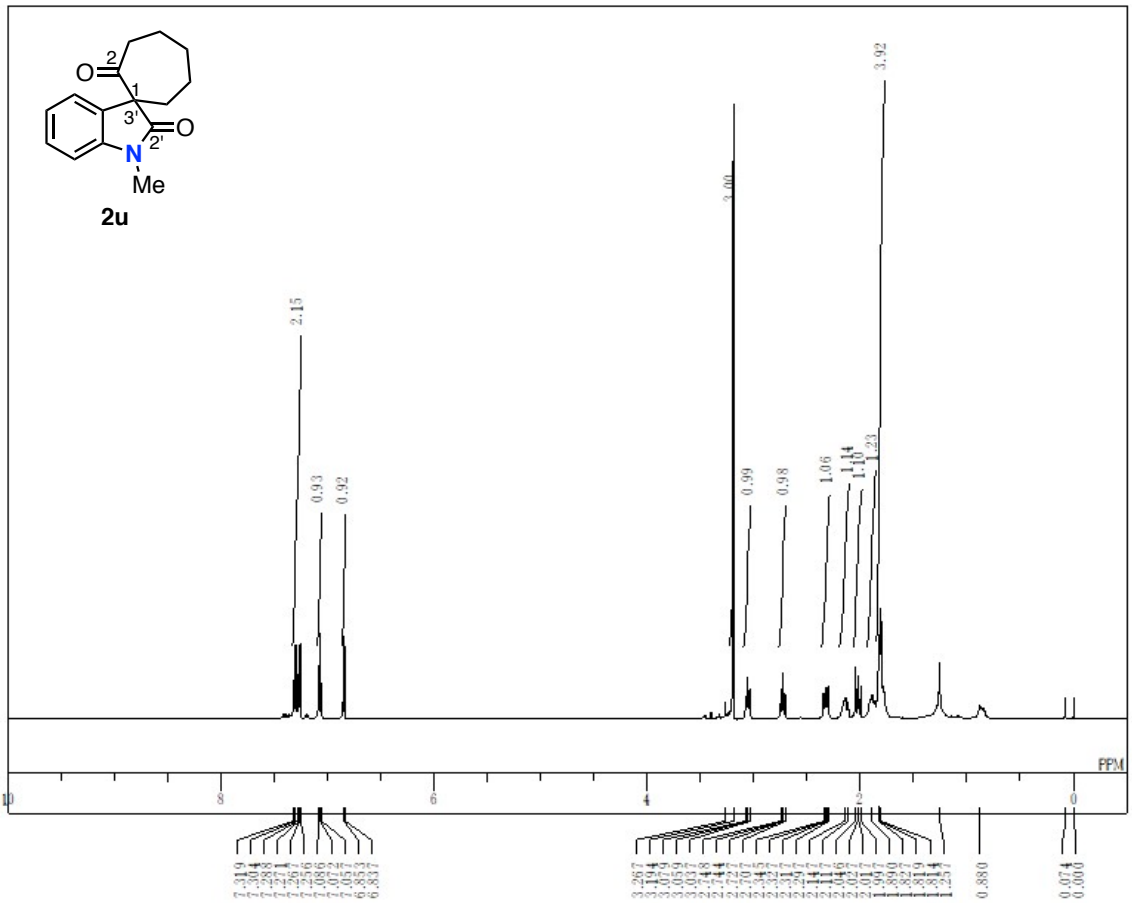

2r
PPM

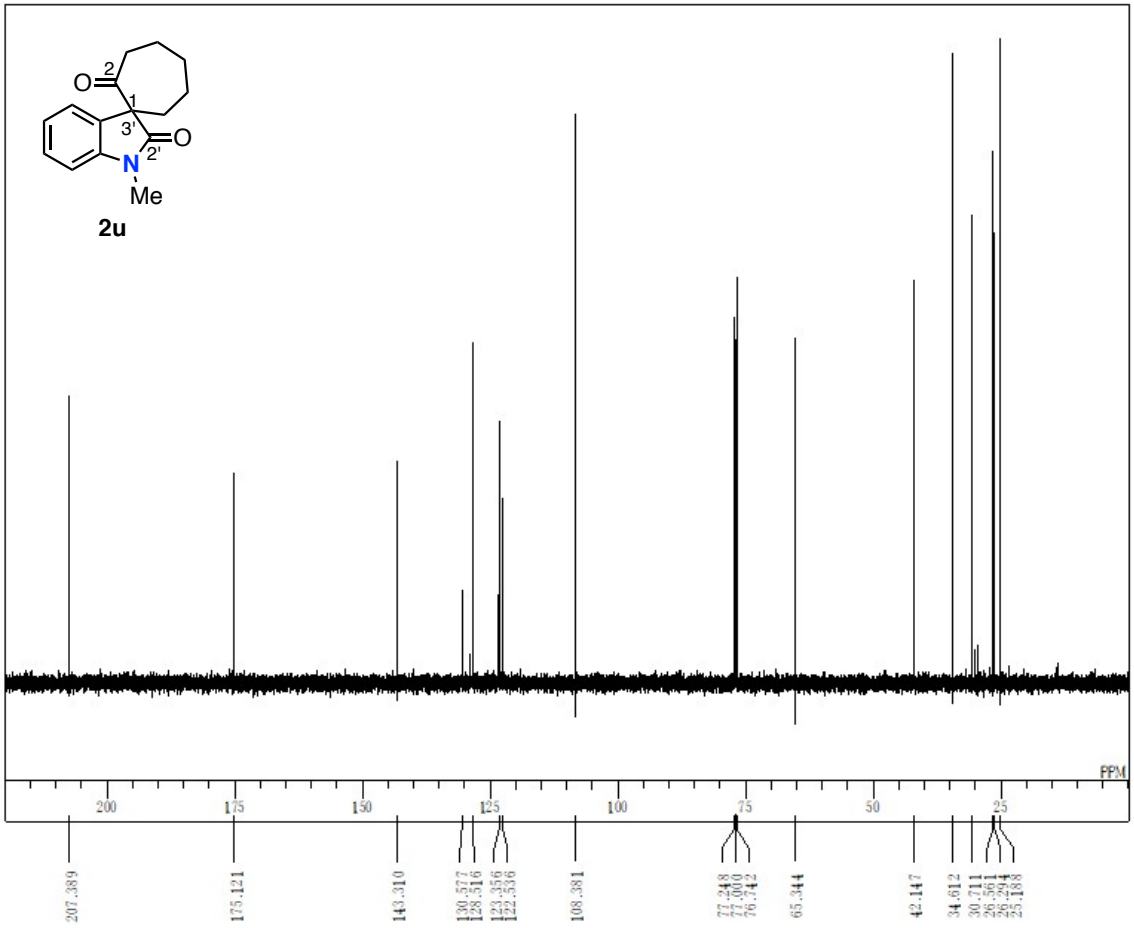

O
2
1
3'
O
2'
N
i-Pr
2s
6.00
1.90
1.93
1.78
1.01
0.84
0.86
0.92
0.93
PPM
10
8
6
4
2
0

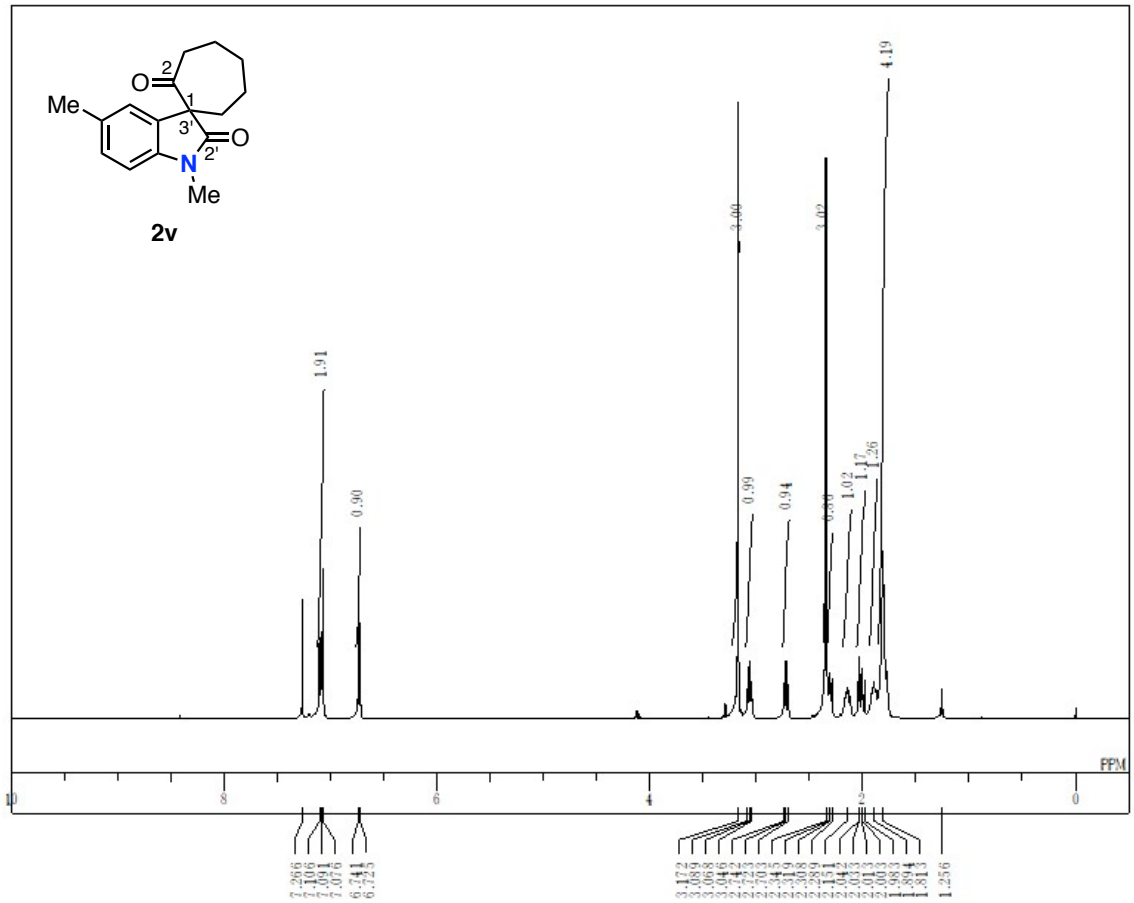

O
2
1
3'
O
2'
N
i-Pr
2s
PPM
200
175
150
125
100
75
50
25
212.587
174.768
143.072
130.796
128.288
122.584
122.221
109.936
77.258
77.000
76.742
62.712
44.150
38.236
34.011
20.085
19.408
19.179

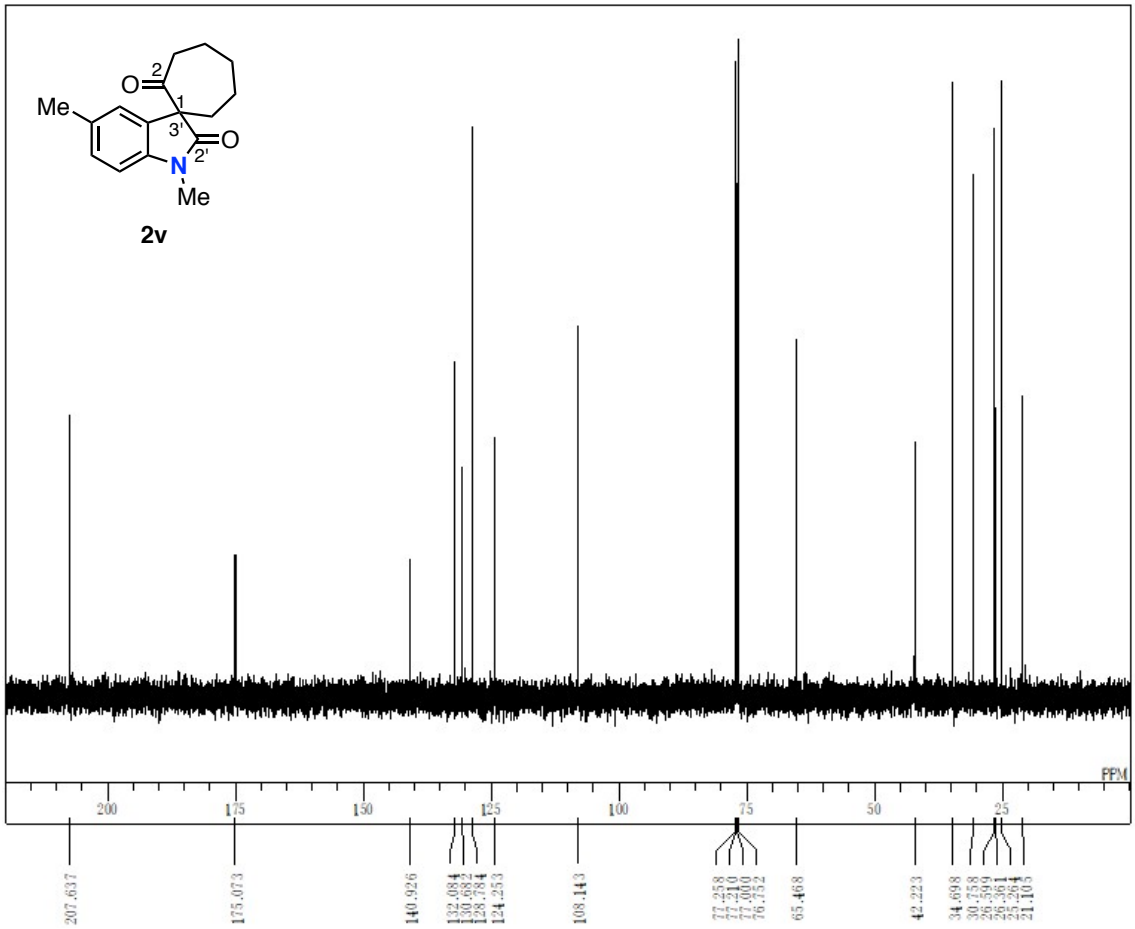

2t
1.12
0.93
0.89
0.88
2.00
2.11
2.01
1.02
0.99
2.07
2.13
3.07
PPM

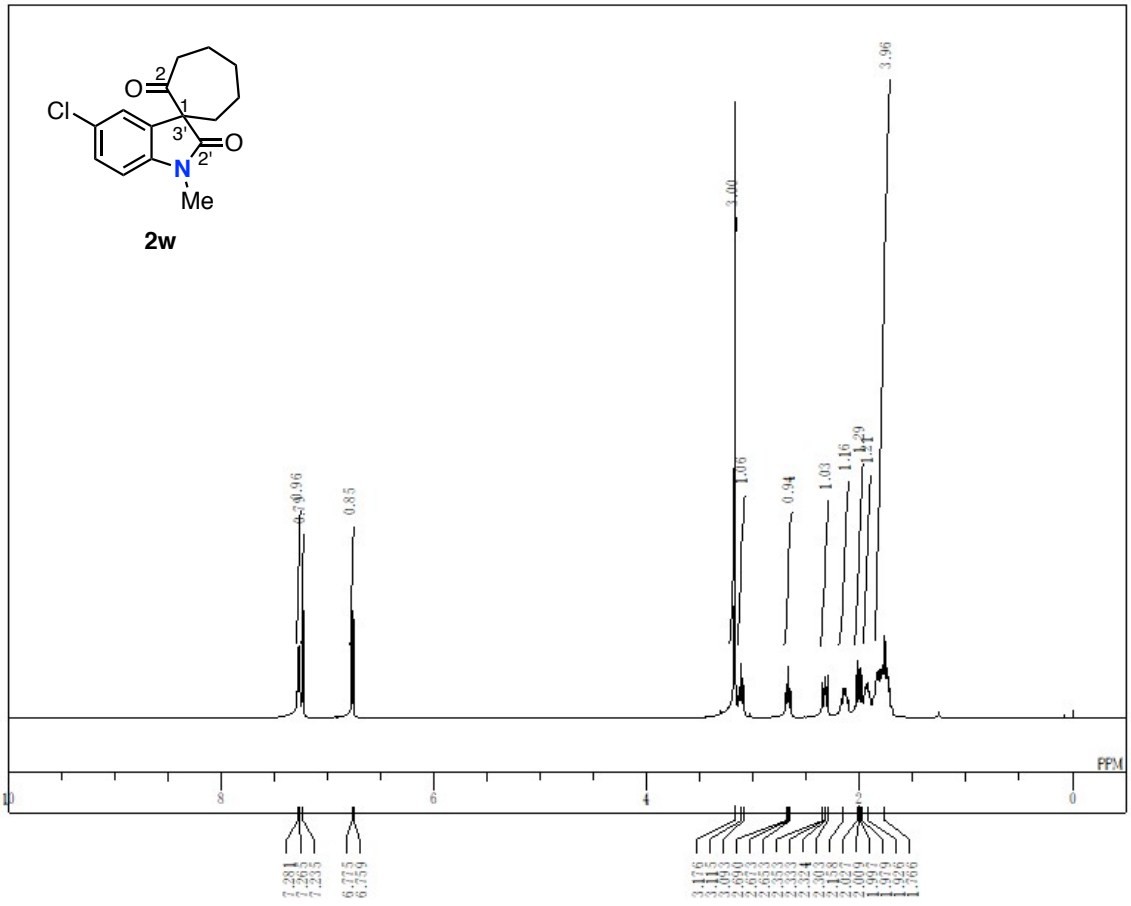

2t
PPM

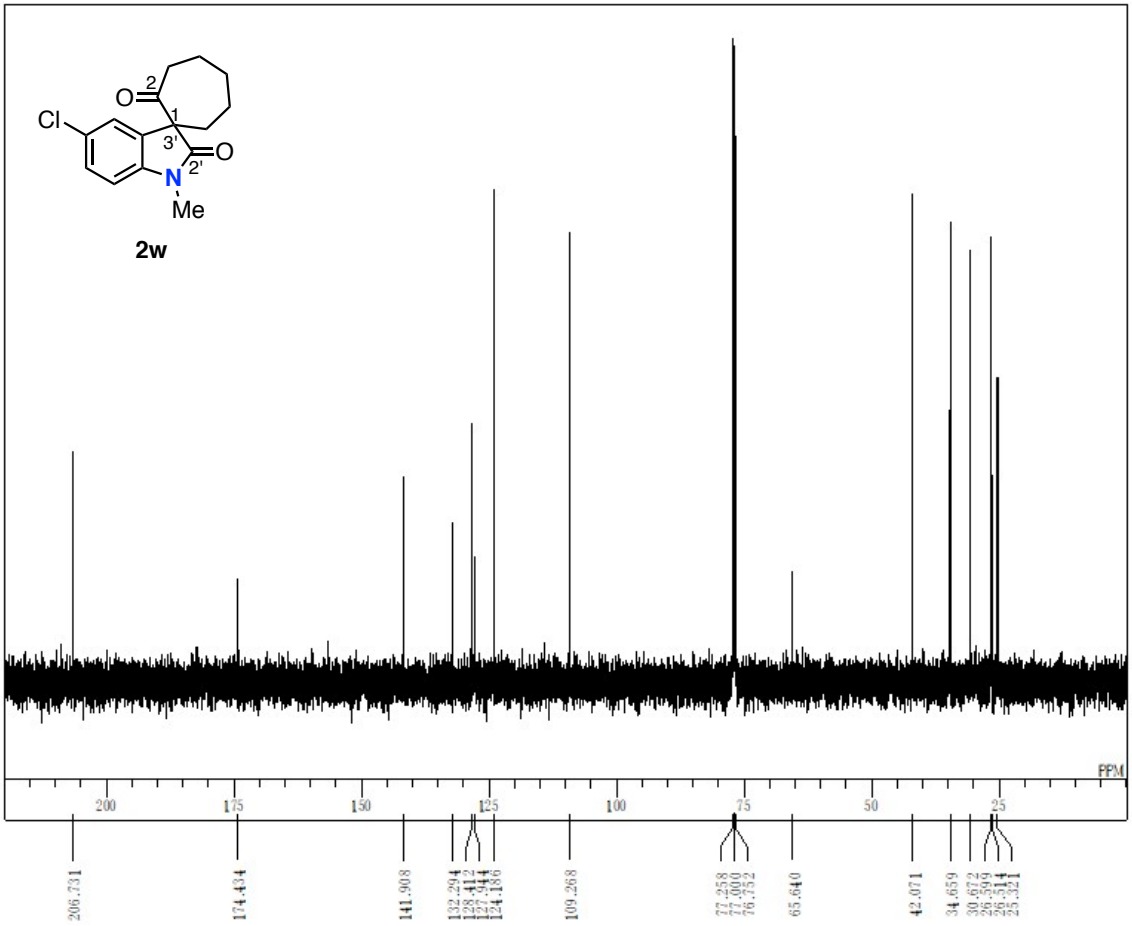

2u
2.15
0.93
0.92
3.00
0.99
0.98
1.06
1.14
1.10
1.23
3.92
PPM

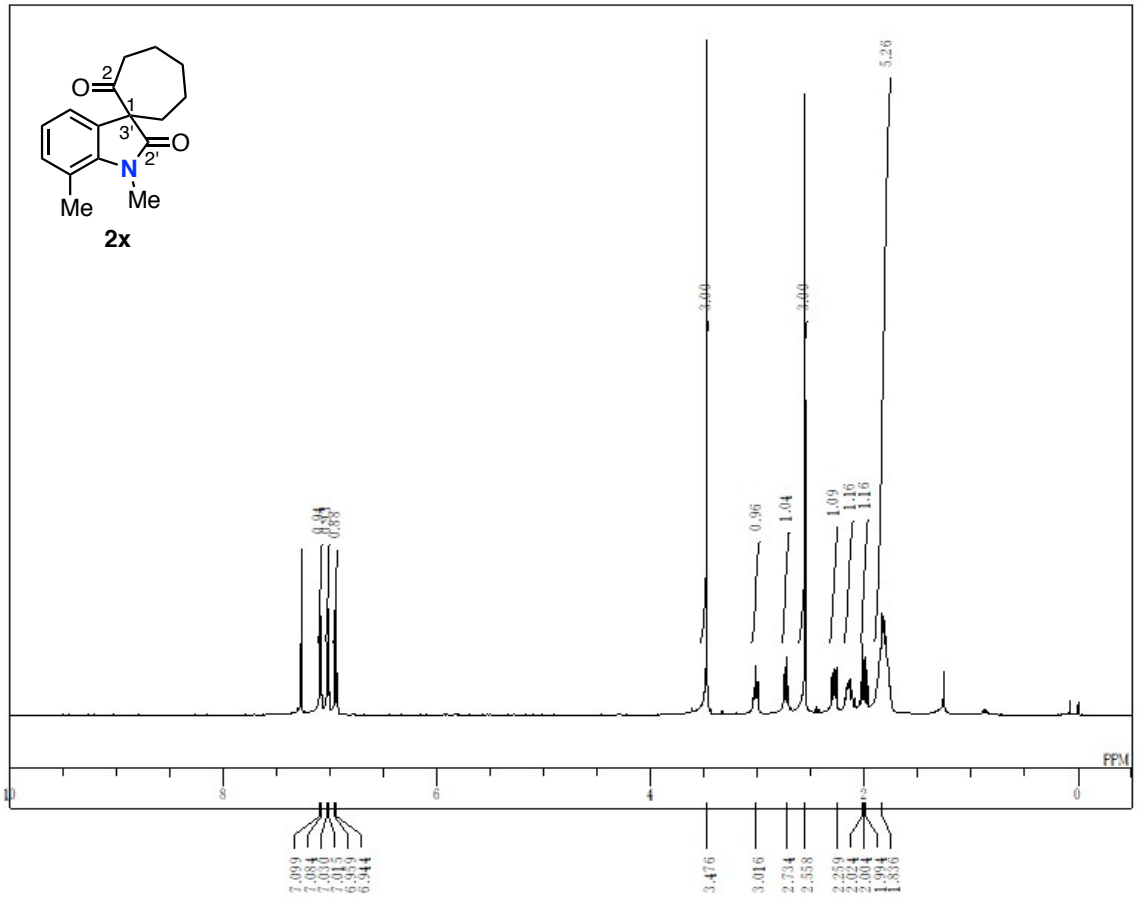

2u
PPM

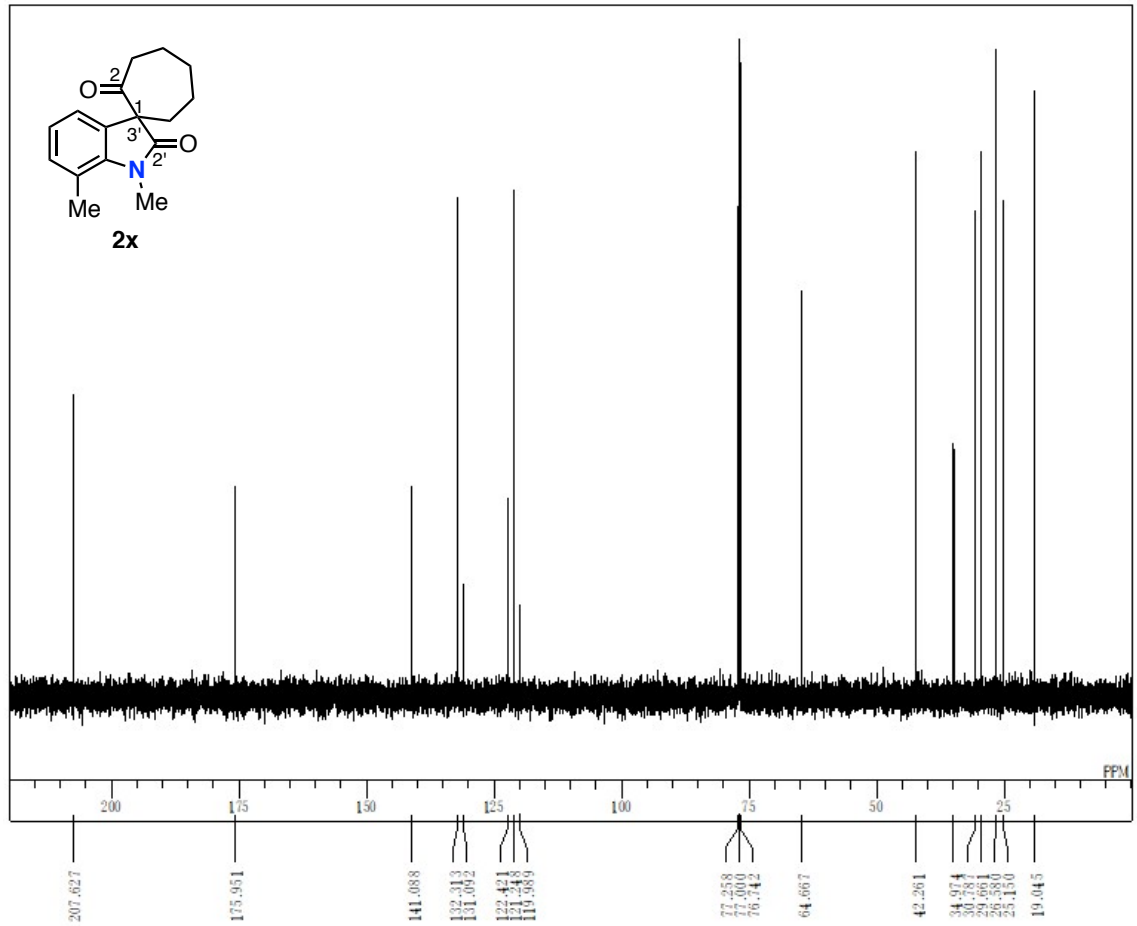

Me
O
2
1
3'
O
2'
N
Me
2v
4.19
3.00
3.02
1.91
0.90
0.99
0.94
1.02
1.17
1.26
PPM
10
8
6
4
2
0

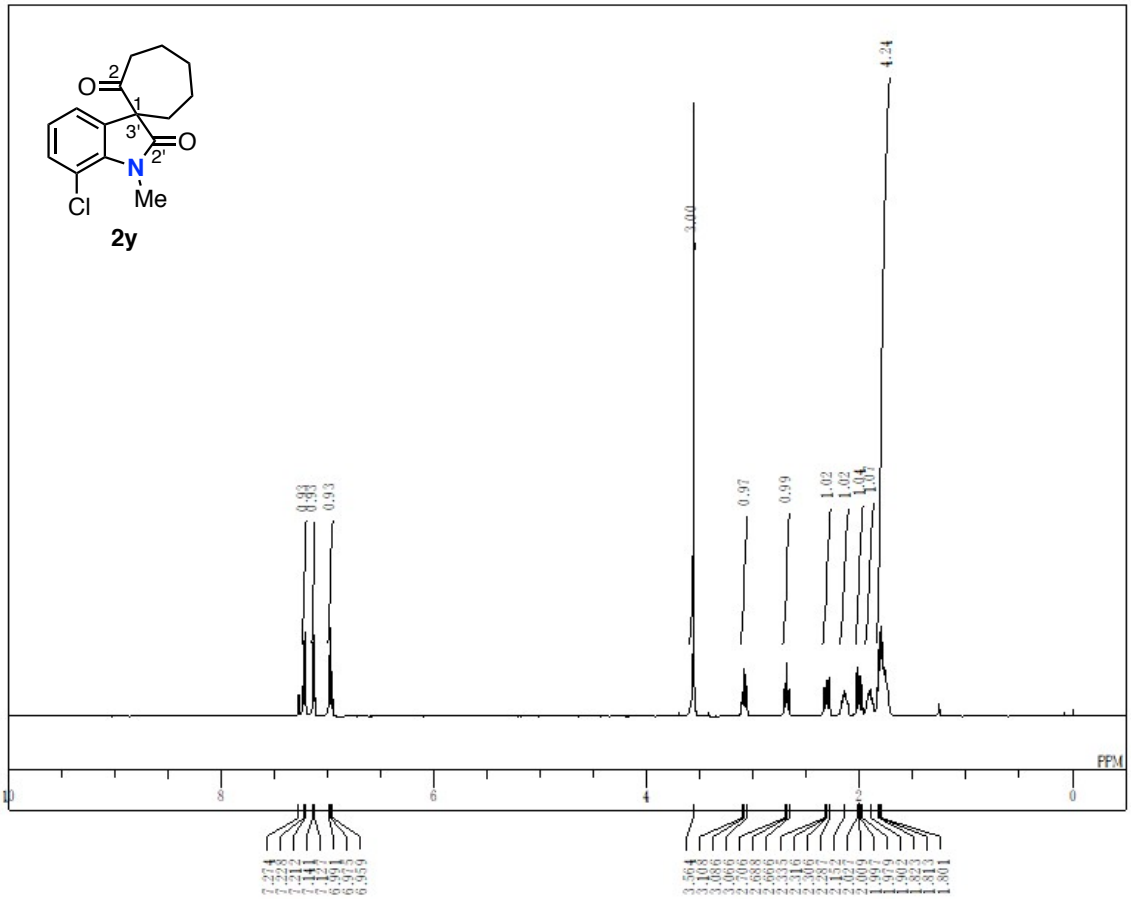

Me
O
2
1
3'
O
2'
N
Me
2v
PPM
200
175
150
125
100
75
50
25

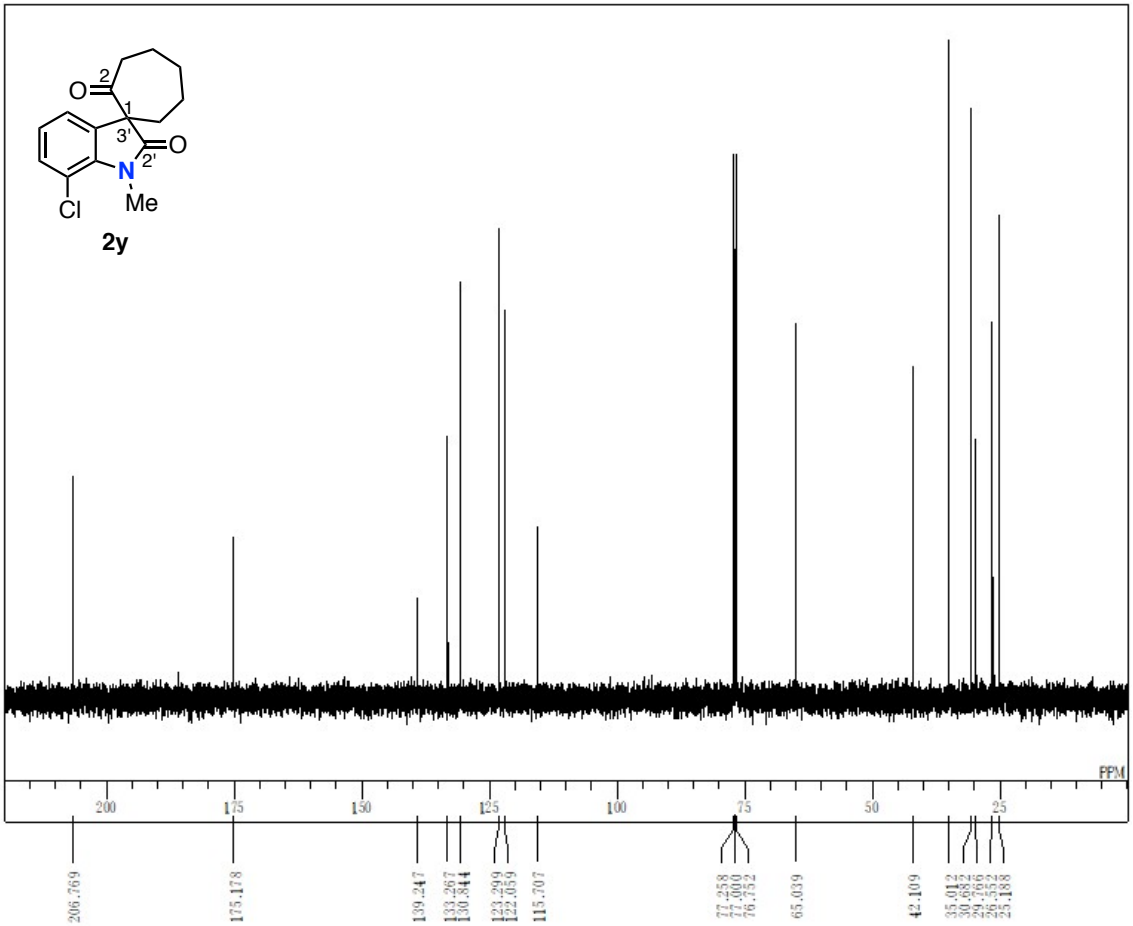

Cl
O
O
N
Me
1
2
3'
2'
2w
3.96
3.00
1.06
0.94
1.03
1.16
1.29
1.41
0.96
0.85
PPM
10
8
6
4
2
0

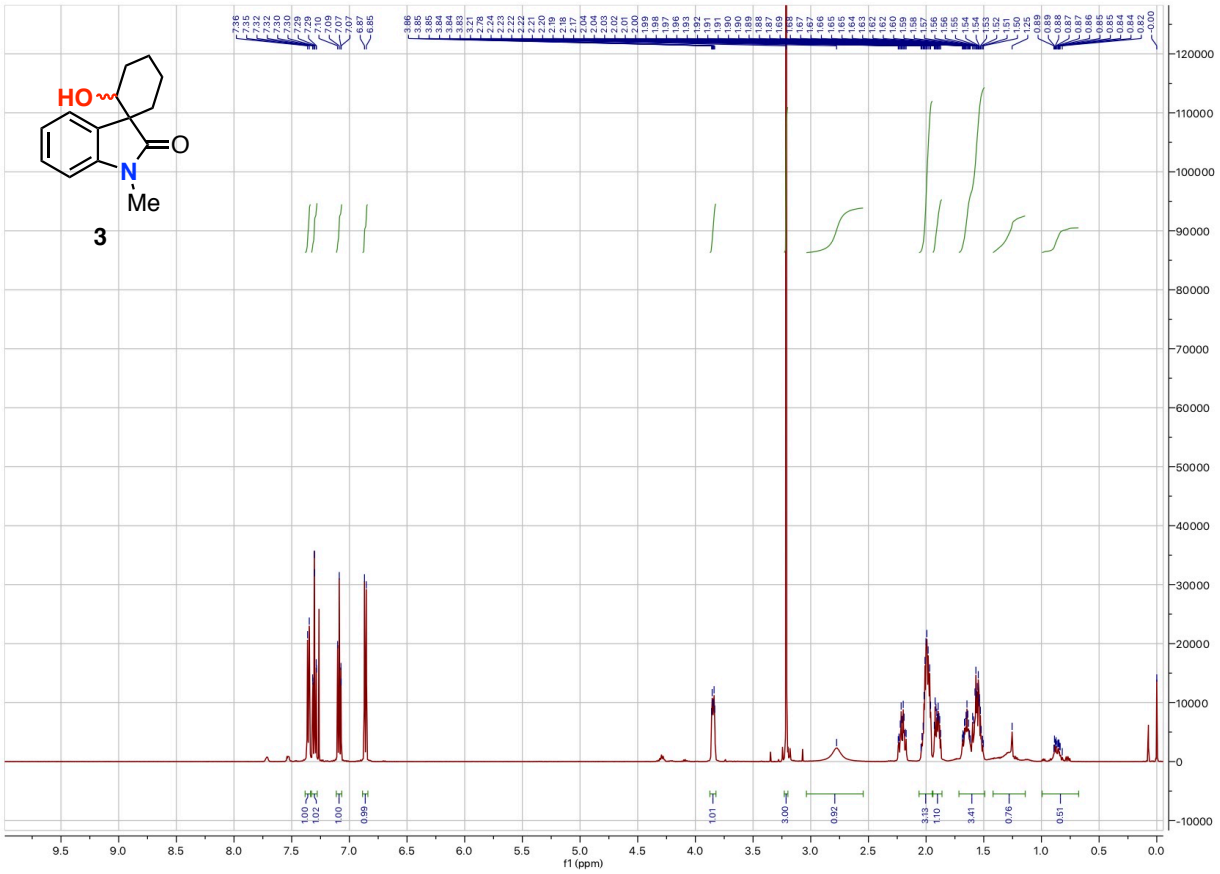

Cl
O
O
N
Me
1
2
3'
2'
2w
PPM
200
175
150
125
100
75
50
25

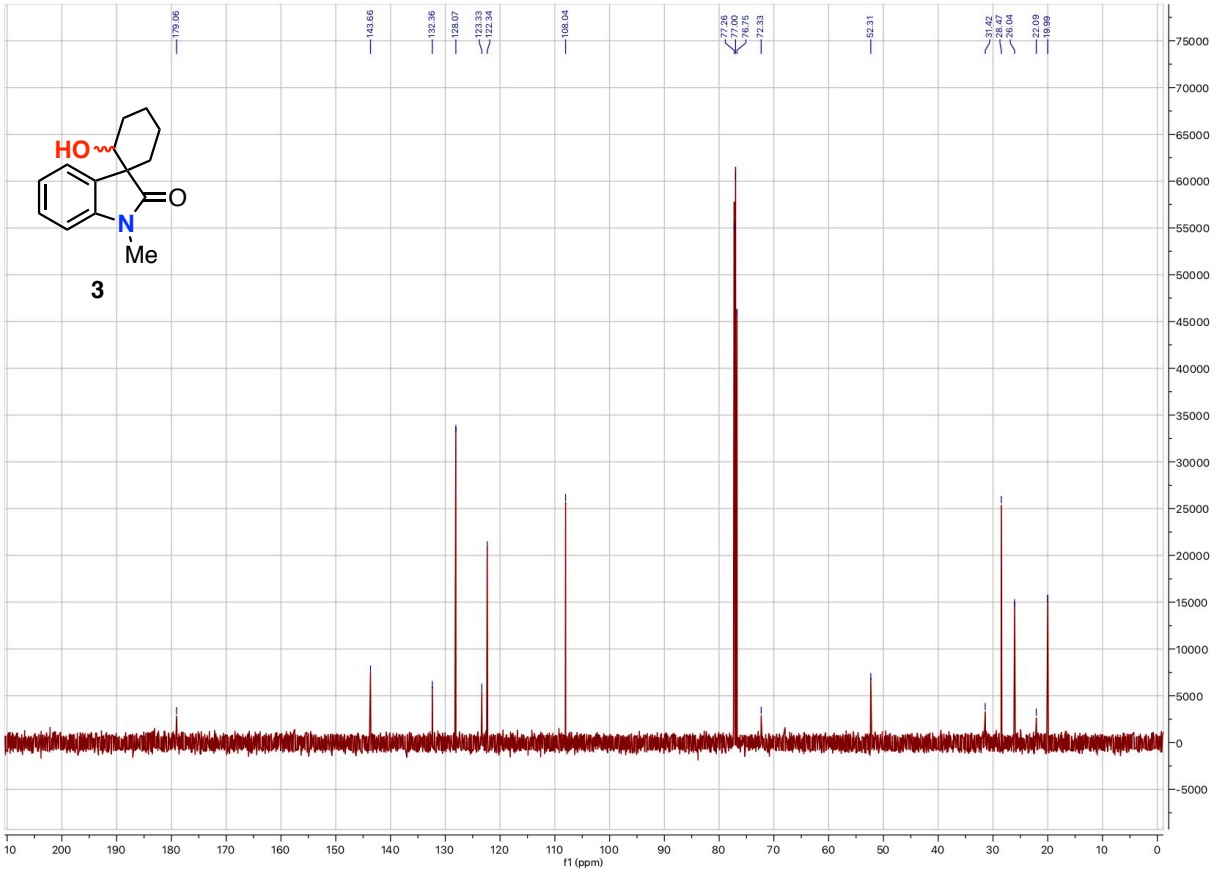

2x
PPM

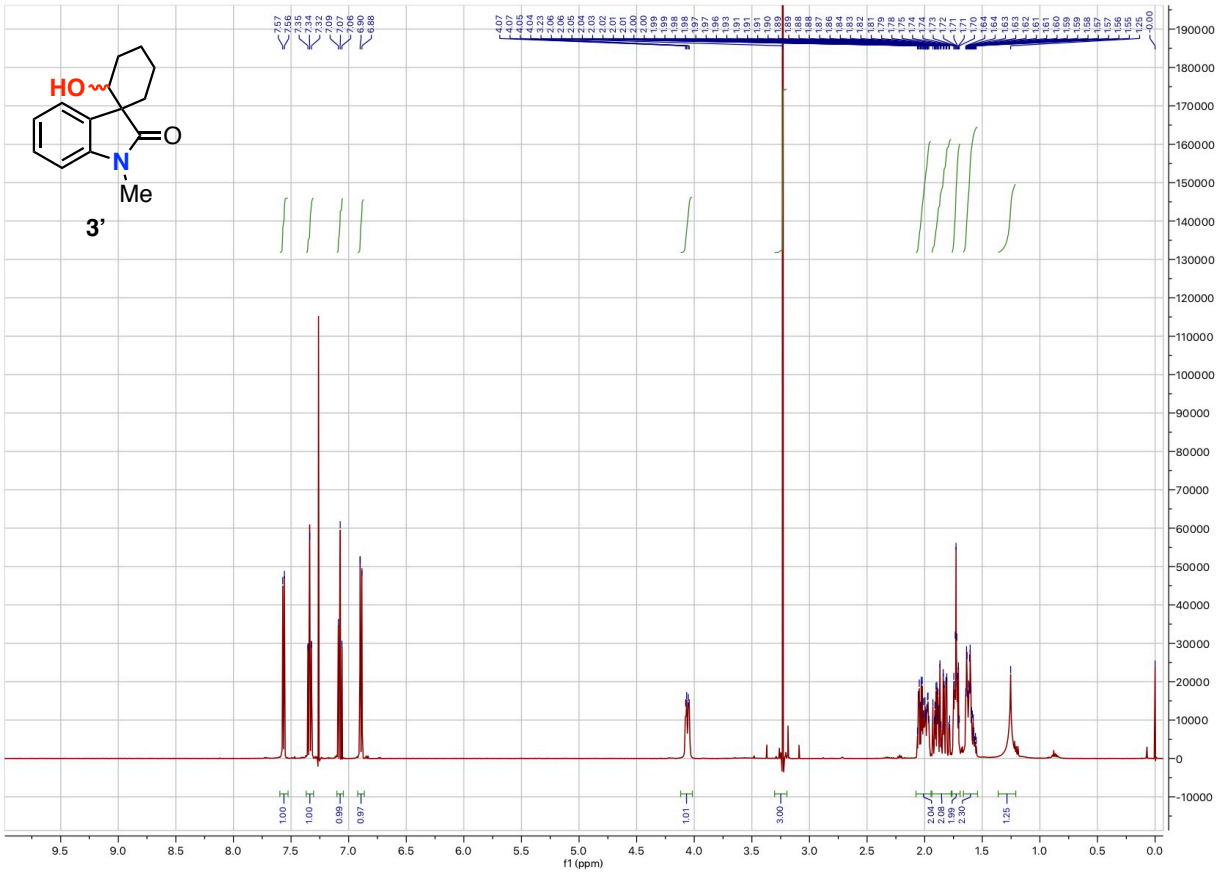

2x
PPM

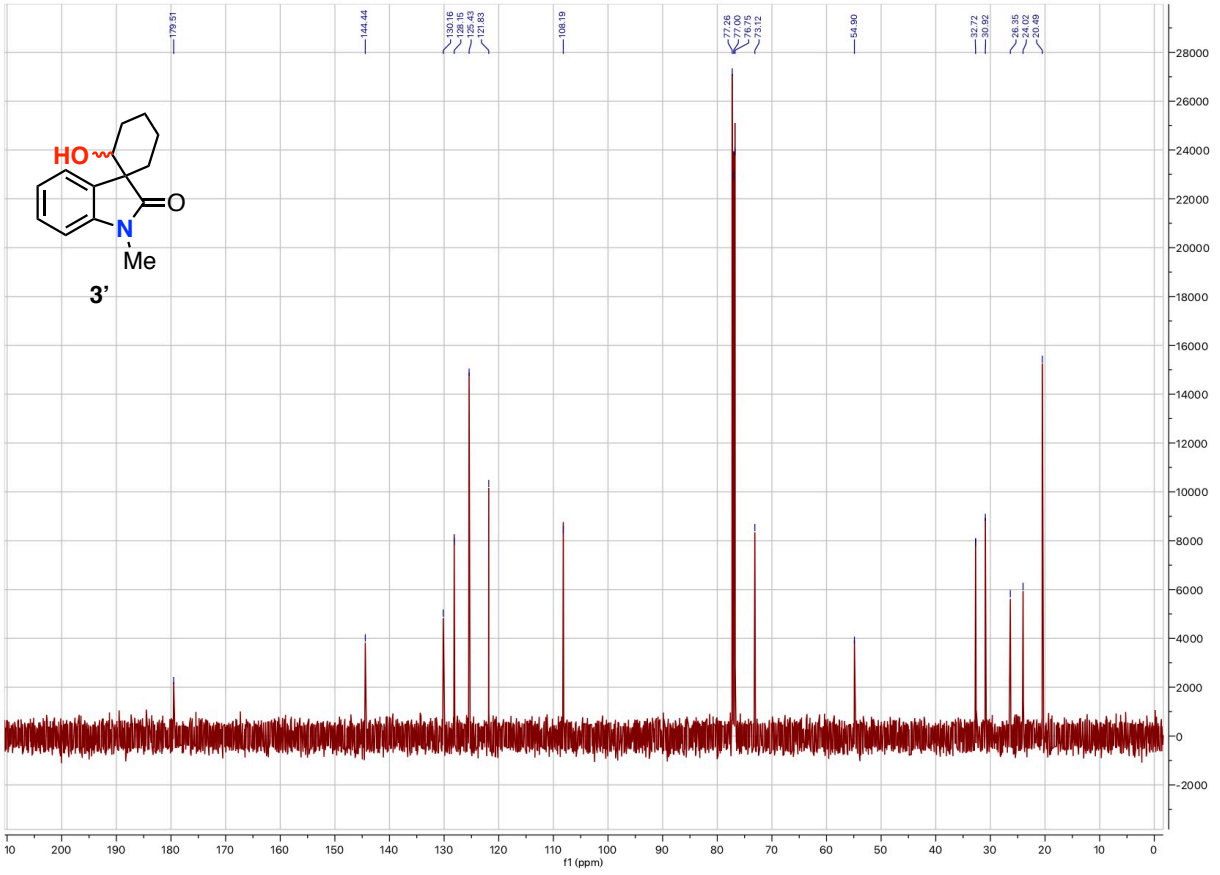

2y
3.00
4.24
0.97
0.99
1.02
1.02
1.04
1.07
PPM

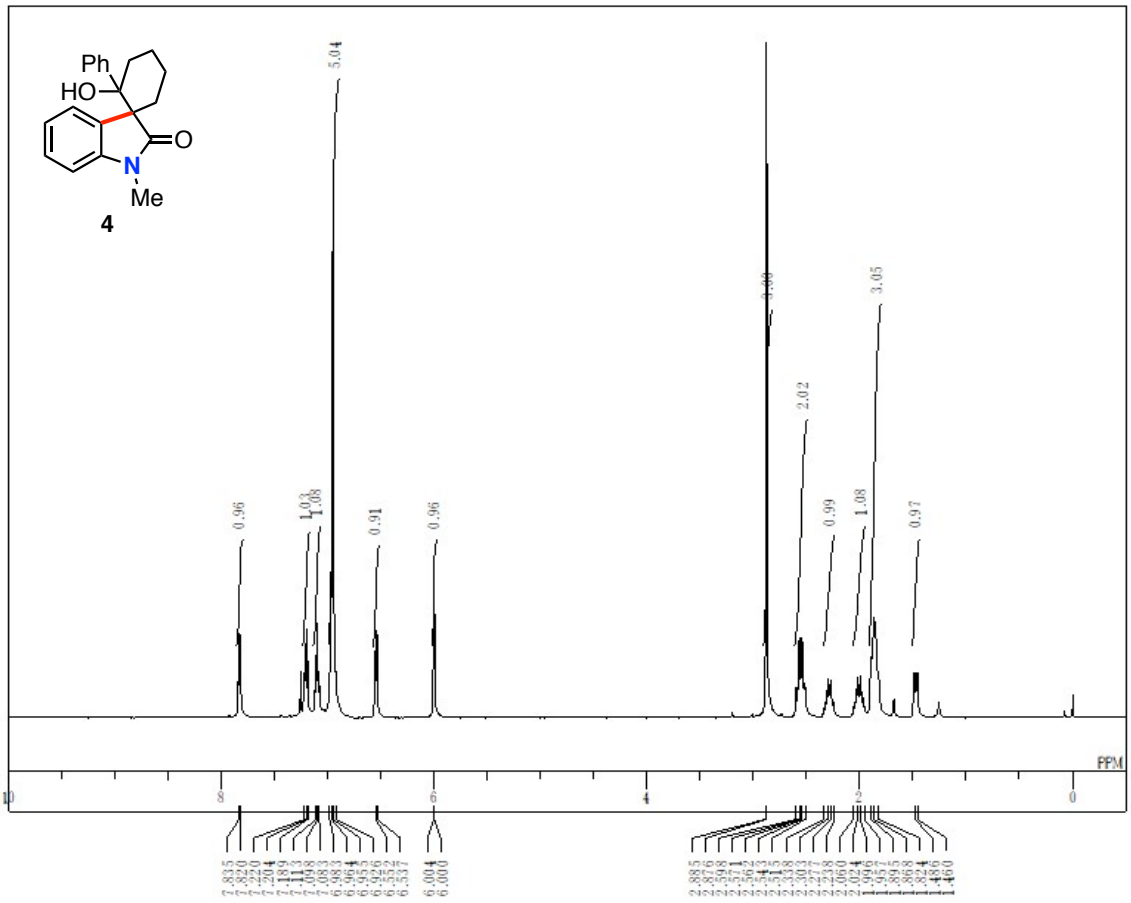

2y
PPM
206.769
175.178
139.247
133.267
130.844
123.299
122.059
115.707
77.258
77.000
76.752
65.039
42.109
35.012
30.682
29.766
26.552
25.188

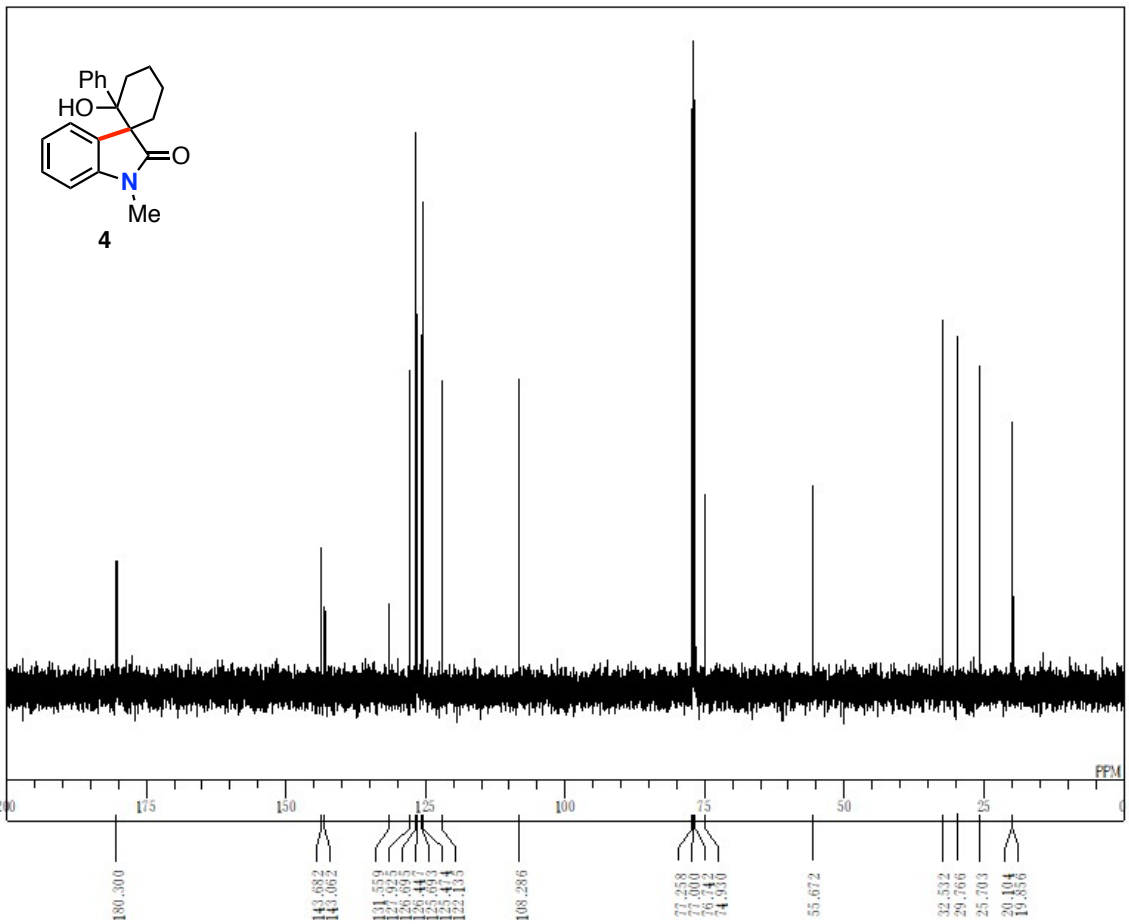

HO
O
N
Me
3
f1 (ppm)

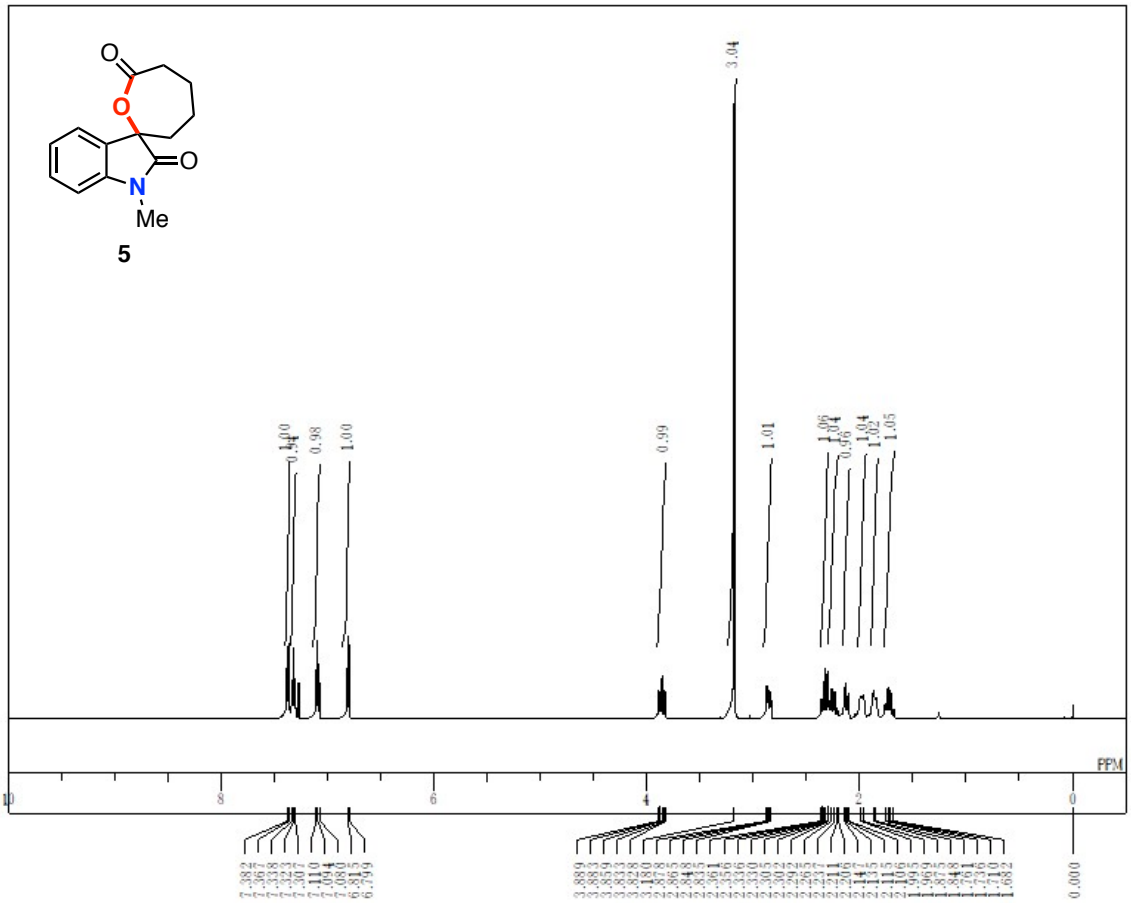

HO
O
N
Me
3
f1 (ppm)

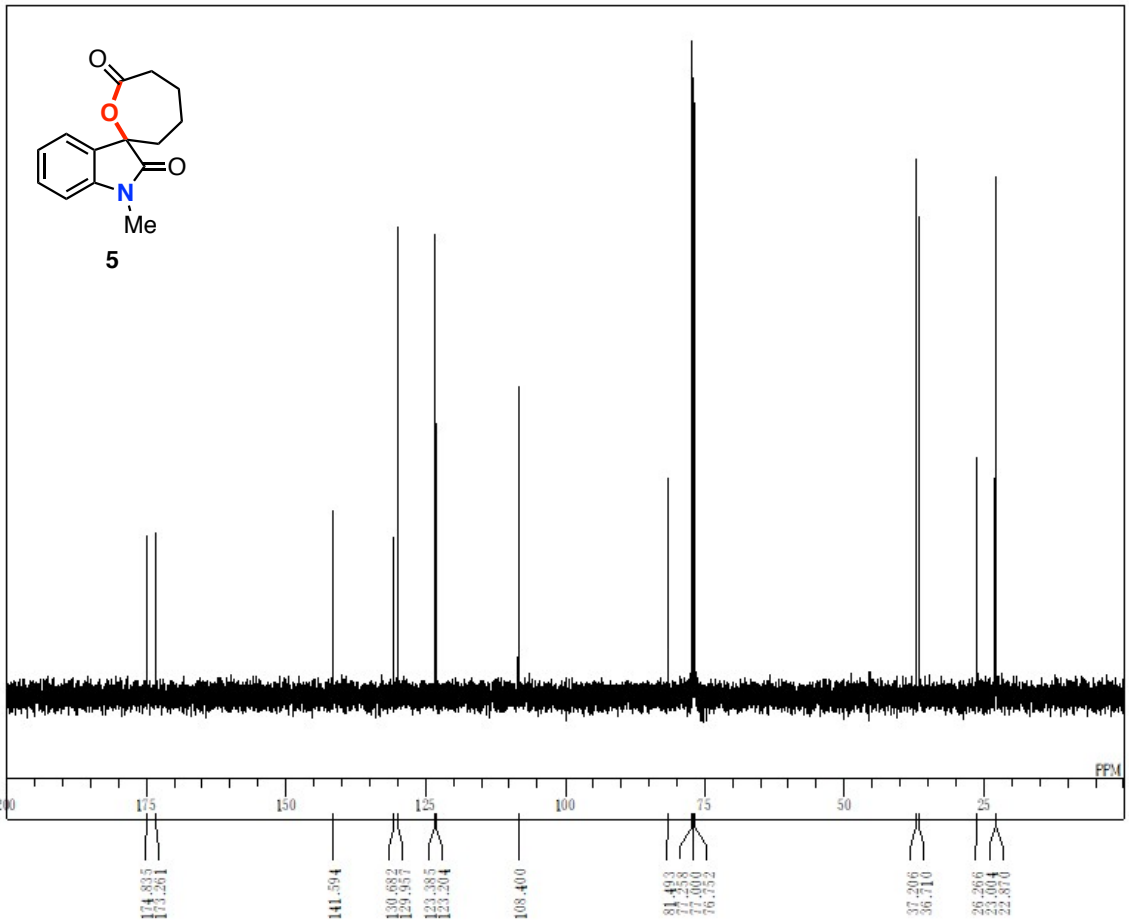

HO
O
N
Me
3’
f1 (ppm)

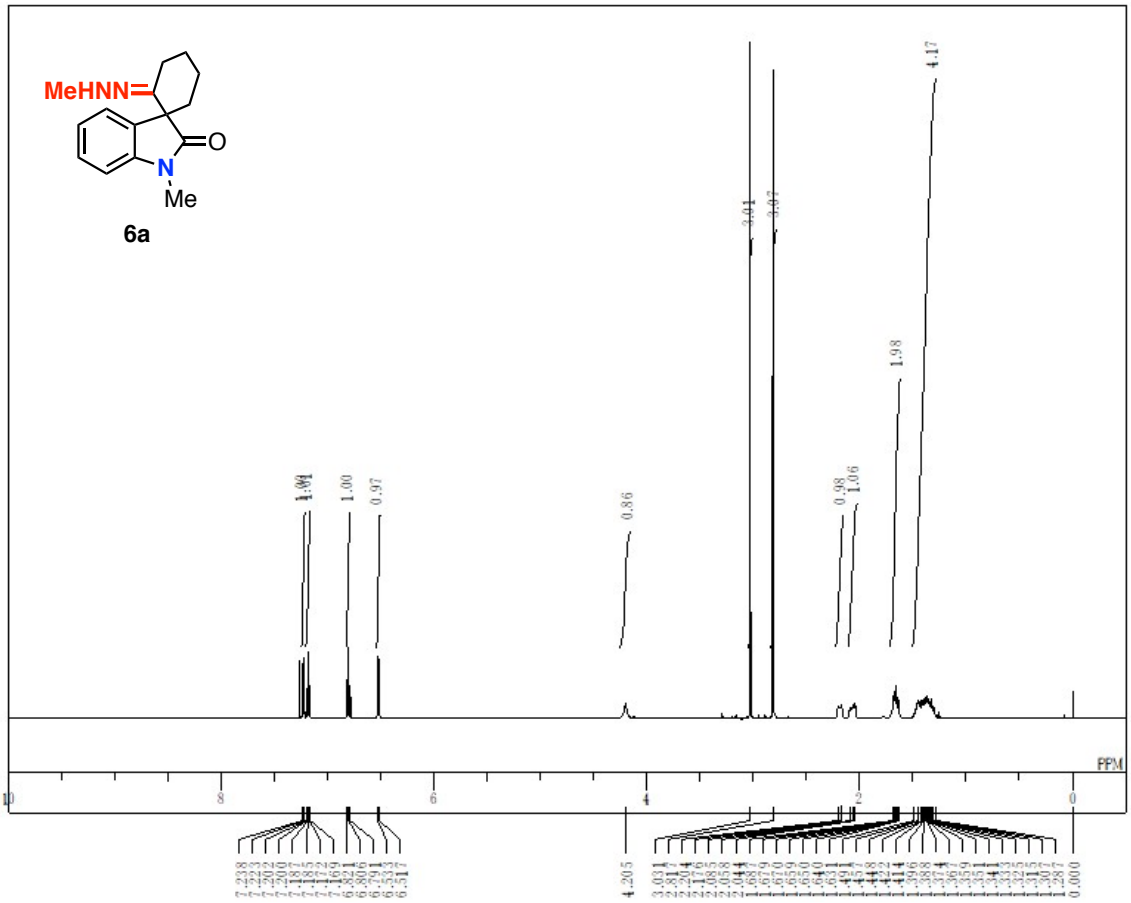

HO
O
N
Me
3’
f1 (ppm)

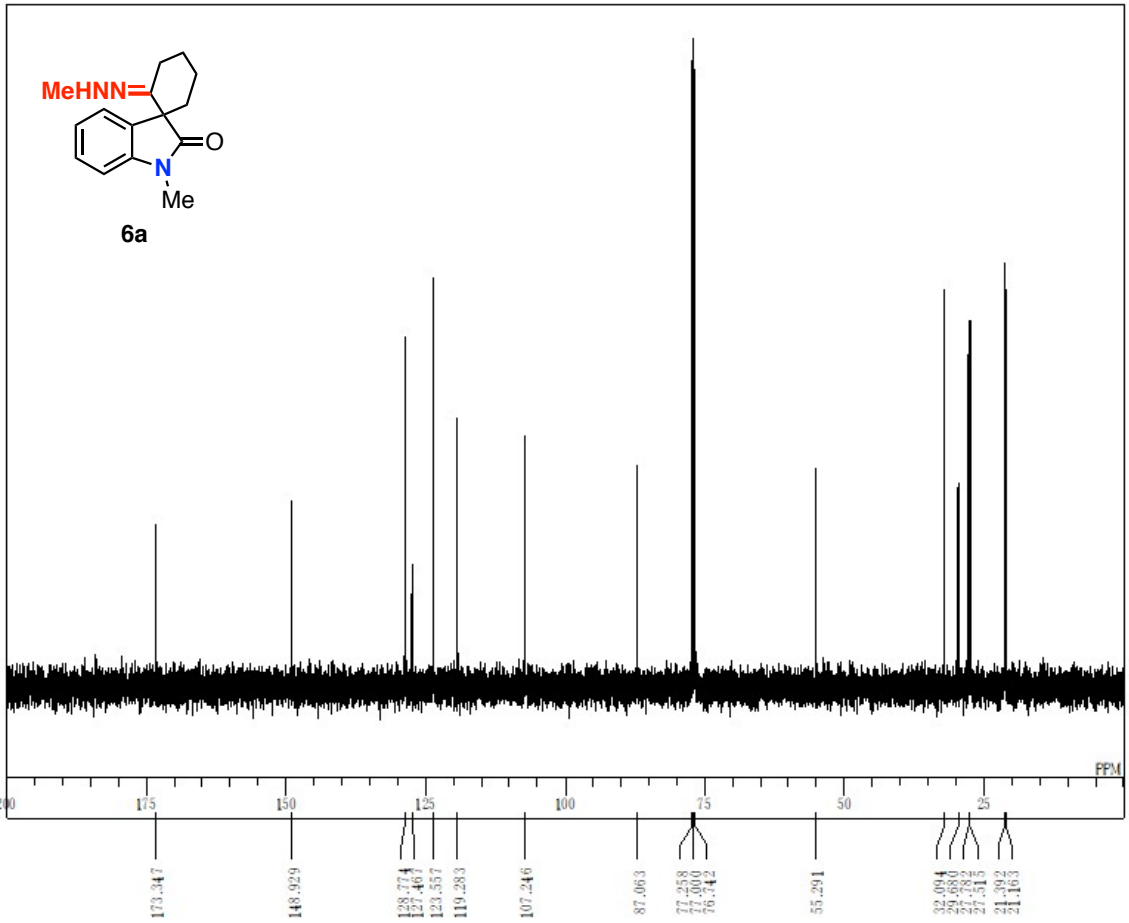

Ph
HO
O
N
Me
4
5.04
3.00
3.05
2.02
1.08
1.03
1.08
0.96
0.91
0.96
0.99
1.08
0.97
PPM

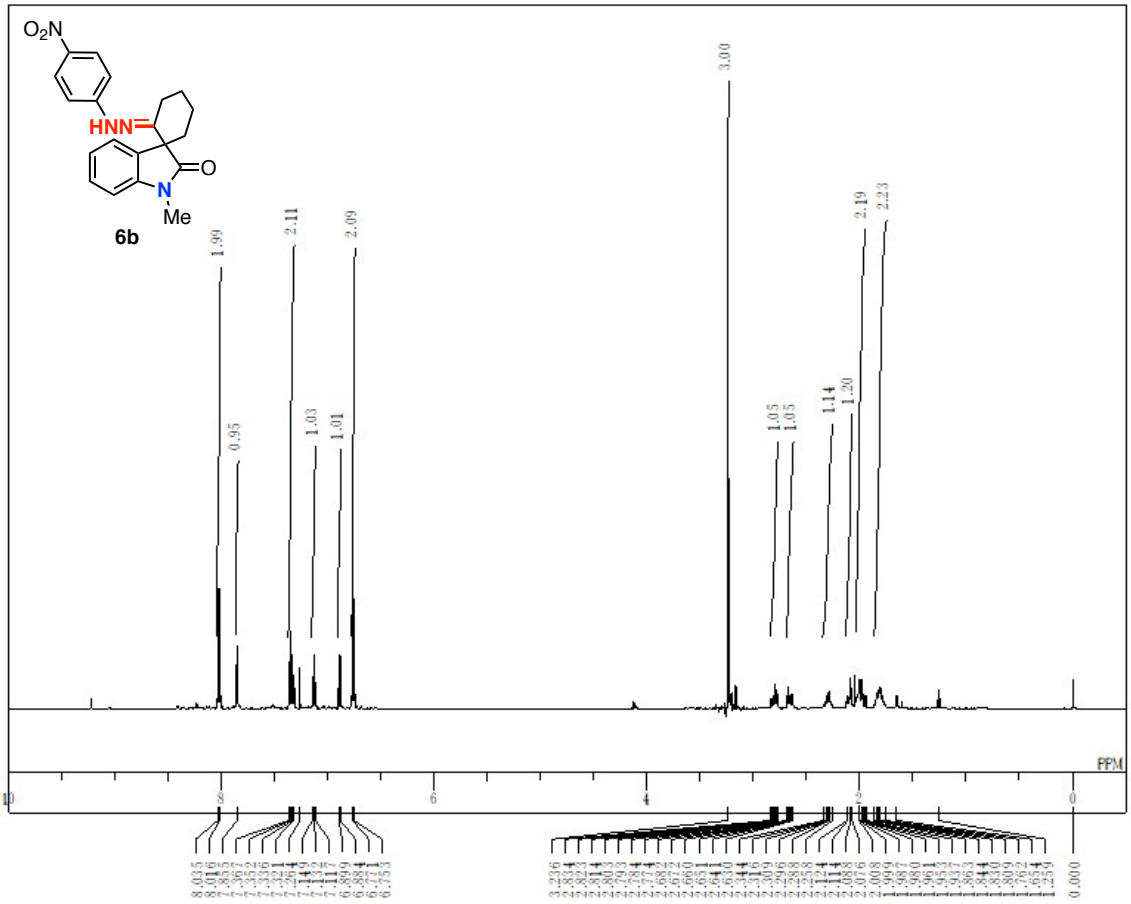

Ph
HO
O
N
Me
4
PPM

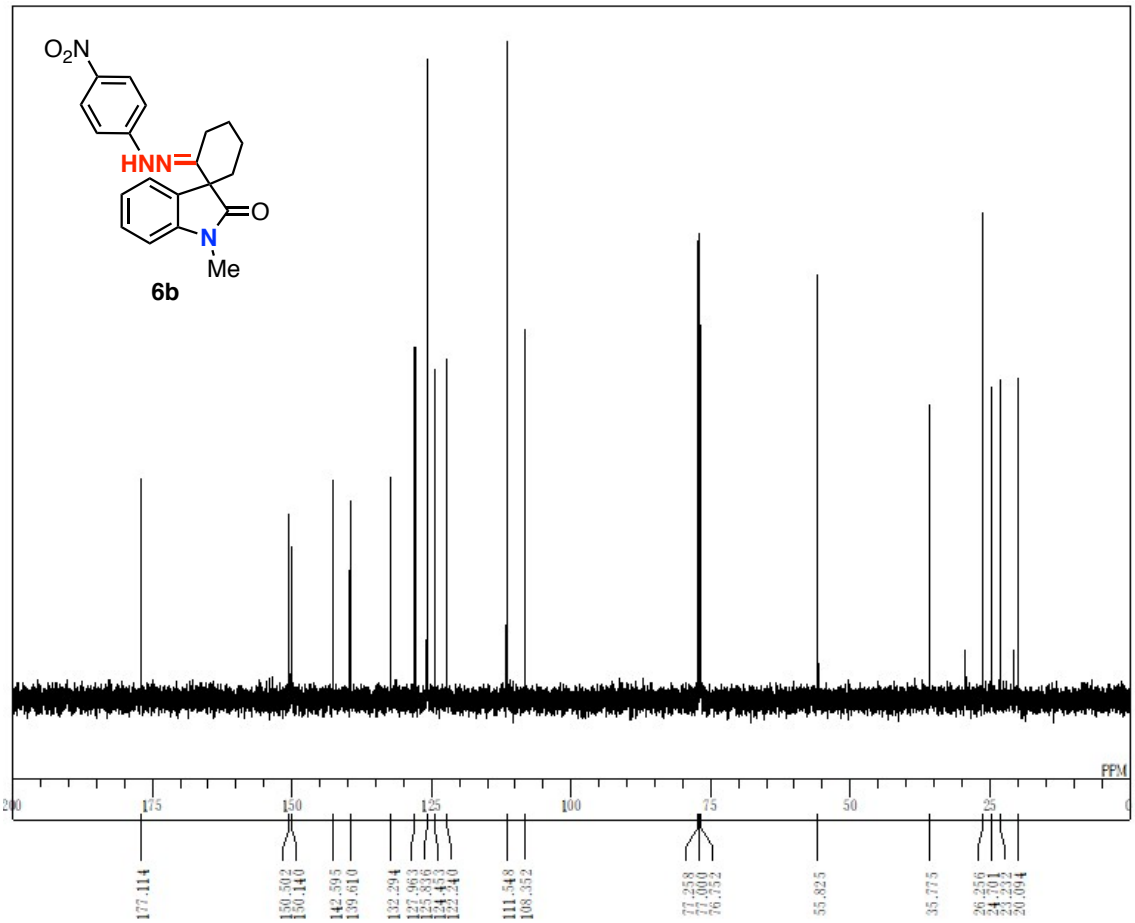

O
O
O
N
Me
5
3.04
0.99
1.01
PPM

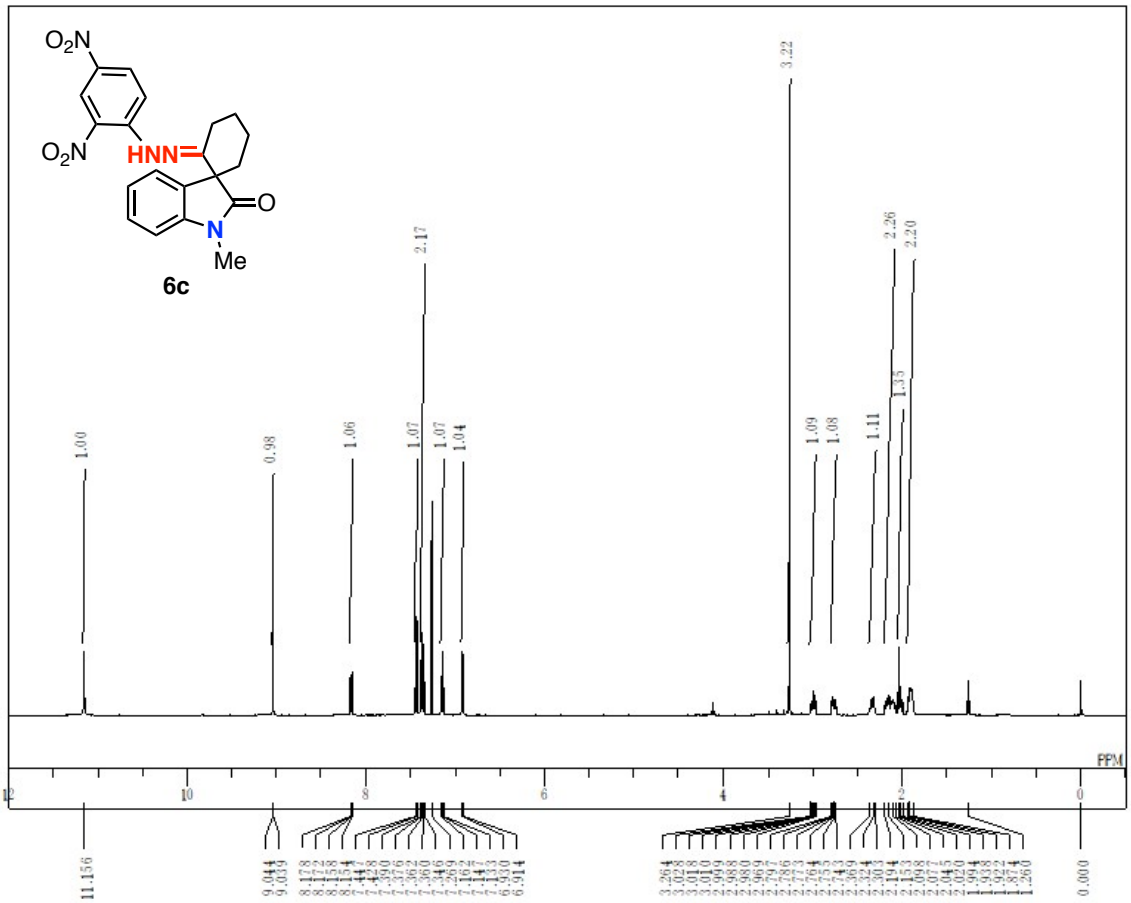

O
O
O
N
Me
5
PPM

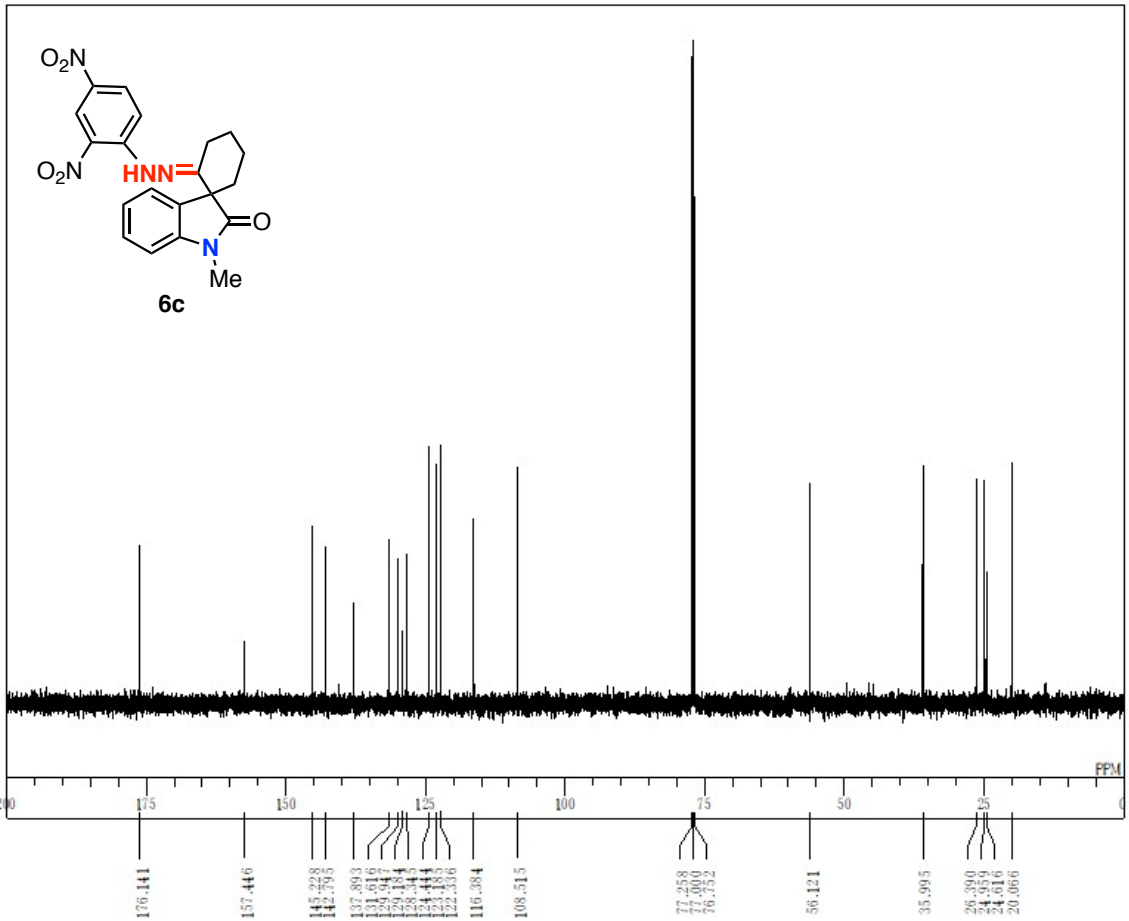

MeHNN=
O
N
Me
6a
4.17
3.04
3.02
1.98
1.06
1.00
0.98
0.97
0.86
PPM


MeHNN=
O
N
Me
6a
PPM


O2N
HNN
O
N
Me
6b
1.99
0.95
2.11
1.03
1.01
2.09
3.00
1.05
1.05
1.14
1.20
2.19
2.23
PPM


O2N
HNN
O
N
Me
6b
PPM


O2N
O2N
HNN
O
N
Me
6c
3.22
2.26
2.20
2.17
1.35
1.11
1.09
1.08
1.07
1.07
1.06
1.04
1.00
0.98
PPM


O2N
O2N
HNN
O
N
Me
6c
PPM