-SUPPORTING INFORMATION-

Iron-catalyzed synthesis of oxindoles: application to the prepara-tion of pyrroloindolines

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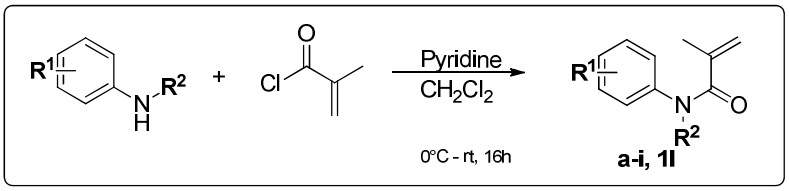
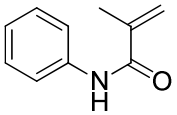
A. General Information

The reagents were purchased from Sigma-Aldrich. Solvents were purified by standard procedures. Reactions were monitored using GCM-QP2010SE (Shimadzu) with low-resolution electron impact (EI, 70 eV) equipped with a RTX®-5MS capillary column. GC/MS conditions: injector 260°C; detector: 110°C; pressure: 100 kPa. Column temperature: 80°C, 1°C/min up to 280°C. Thin-layer chromatography (TLC) was conducted with Merck silica gel 60 F254 precoated plates and visualized with UV and vanillin stain. Flash column chromatography was performed on silica gel (200−300 mesh). ^{1}H NMR spectra were recorded on a Varian Inova-300 (300 MHz) spectrometer and are reported in ppm using TMS as an internal standard ($CDCl_3$ at δ 7.26 ppm). The following abbreviations were used to describe peak splitting patterns when appropriate: br s = broad singlet, s = singlet, d = doublet, t = triplet, q = quartet, and m = multiplet. Coupling constant, *J*, was reported in hertz (Hz). Proton-decoupled ^{13}C NMR spectra were recorded on a Varian-300 (75 MHz) spectrometer and are reported in ppm using TMS as an internal standard ($CDCl_3$ at δ 77.0). High-resolution mass spectra were recorded using MicroToF Bruker Daltonics, ESI-TOF techniques.

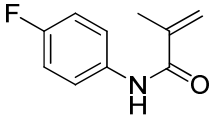
B. General procedure for the preparation of *N*-Arylacrylamides:[1]



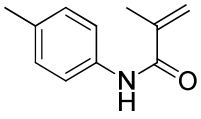
To a 250 mL round bottom flask charged with magnetic stirrer, aniline (20 mmol), pyridine (2.4 mL, 30 mmol) was added 80 mL anhydrous dichloromethane, under N_2 atmosphere. The resulting mixture was cooled to 0 °C, followed by the drop wise addition of methacryloyl chloride (2.34 mL, 24 mmol) and stirring 15 minutes. The mixture was brought to room temperature and stirred for 16 h. The reaction was quenched with 40 mL H_2O. The organic phase was washed with H_2O (40 mL), saturated $NaHCO_3$ (2 × 40 mL) and saturated NaCl (40 mL). The combined aqueous phase was extracted with dichloromethane (3 x 40 mL). The combined organic phase was dried over $MgSO_4$ and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel using ethyl acetate/hexane as eluent (15 to 30%, AcOEt). The product was characterized by ^{1}H NMR spectroscopy and mass spectrometry.



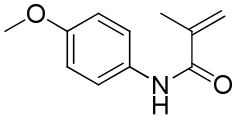
***N*-phenyl-methacrylamide (a or 1k)**: ^{1}H NMR ($CDCl_3$, 300Hz) δ 7.55 (d, 2H, *J*=6.0 Hz), 7.33 (t, 2H, *J*=9.0 Hz), 7.12 (t, 1H, *J*=9.0 Hz), 5.78 (s, 1H), 5.45 (s, 1H, *J*=3.0 Hz), 2.06 (s, 3H) ppm.



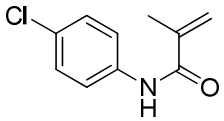
***N*-(4-fluorphenyl)-methacrylamide (b)**: ^{1}H NMR ($CDCl_3$, 300Hz) δ 7.04 (d, 2H, *J*=9.0Hz), 6.84 (d, 2H, *J*=9.0Hz), 4.99 (s, 1H), 4.96 (s, 1H), 3.78 (s, 3H), 3.28 (s, 3H), 1.71 (s, 3H) ppm.



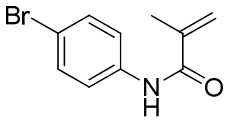
***N*-(4-Methylphenyl)-methacrylamide (c)**: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.44 (d, 2H, *J*=9 Hz), 7.12 (d, 2H, *J*= 9 Hz), 5.78 (s, 1H), 5.44 (s, 1H), 2.32 (s, 3H), 2.06 (s, 3H) ppm.



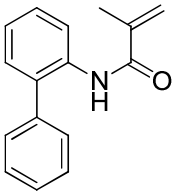
***N*-(4-methoxyphenyl)-methacrylamide (d)**: ^{1}H NMR ($CDCl_3$, 300Hz) δ 7.46 (d, 3H, *J*=9.0 Hz), 6.87 (d, 2H, *J*=9.0 Hz), 5.77 (s, 1H), 5.42 (s, 1H), 3.79 (s, 3H), 2.05 (s, 3H) ppm.



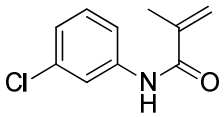
***N*-(4-chlorophenyl)-methacrylamide (e)**: ^{1}H NMR ($CDCl_3$, 300Hz) δ 7.76 (s, 1H), 7.52 (d, 2H, *J*=9.0Hz), 7.28 (d, 2H, *J*=9.0Hz), 5.78 (s, 1H), 5.45 (s, 1H), 2.03 (s, 3H) ppm.



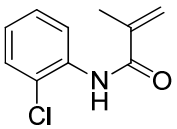
***N*-(4-bromophenyl)-methacrylamide (f)**: ^{1}H NMR ($CDCl_3$, 300 Hz) δ 7.46 (d, 2H, *J*=9.0Hz), 7.41 (d, 2H, *J*=9.0 Hz), 5.77 (s, 1H), 5.44 (s, 1H), 2.02 (s, 3H) ppm.



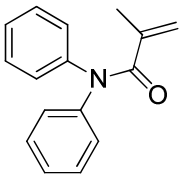
***N*-([1,1'-biphenyl]-2-yl)-methacrylamide (g)**: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 8.43 (d, 1H, *J*=9Hz), 7.64 (s, 1H) 7.54-7.37 (m, 6H), 7.29-7.16 (m, 2H), 5.54 (s,1H), 5.31 (s, 1H), 1.85 (s, 3H) ppm.



***N*-(3-chlorophenyl)-methacrylamide (h)**: ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.70-7.68 (m, 1H), 7.50 (s, 1H), 7.42-7.39 (m, 1H), 7.28-7.23 (m, 1H), 7.12-7.08 (m, 1H), 5.79 (s, 1H), 5.49 (s, 1H), 2.06 (s, 3H) ppm.

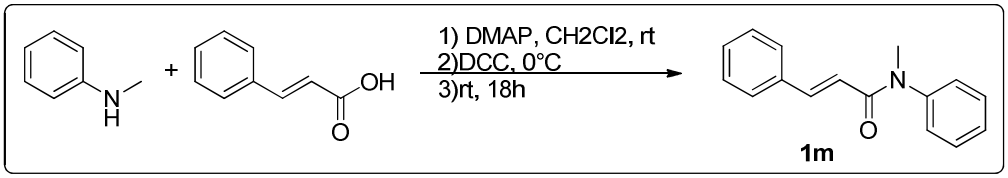


***N*-(2-chlorophenyl)-methacrylamide (i)**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 8.47 (dd, 1H, *J*= 7.5 Hz, J= 3 Hz), 8.13 (s, 1H), 7.40-7,27 (m, 2H), 7.08-7.06 (m,1H), 5.91 (s, 1H), 5.53 (s, 1H), 2.11 (s, 3H) ppm.

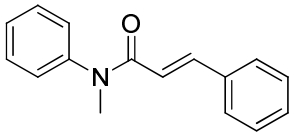


***N*,*N*-diphenylmethacrylamide (1l)**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.36-7.31 (m, 4H), 7.26-7.15 (m, 6H), 5.23 (s, 1H), 5.17 (s, 1H), 1.84 (s, 3H) ppm.[2]

B.1 Procedure for the preparation of *N*-methyl-*N*-phenylcinnamamide:[3]

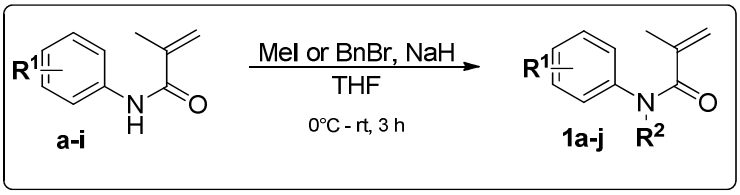


To a stirring solution of cinnamic acid (5 mmol) in dry CH_2Cl_2 (10 mL), the N-methylaniline (5 mmol) and 4-Dimethylaminopyridine (DMAP) (0.125 mmol) were added. The mixture was cooled to 0ºC and *N,N'*-dicyclohexylcarbodiimide (DCC) (5 mmol) was added, then the mixture was maintained at 0ºC. After 10 min, the reaction was allowed to reach room temperature and stirred for 18h. Subsequently, CH_2Cl_2 (10 mL) was added and the mixture was filtrated. Afterwards, the filtrate was washed with a 10% aqueous solution of HCl (2 × 10 mL) and with a saturated solution of Na_2CO_3 (2 × 10 mL). The combined organic extracts were dried over $MgSO_4$ and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel using ethyl acetate/hexane as eluent (10%, AcOEt).

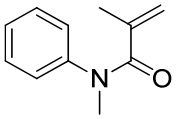


***N*-methyl-*N*-phenylcinnamamide (1m):** ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.68 (d, 1H, *J*= 18 Hz), 7.47-7.23 (m, 10H), 6.37 (d, 1H, *J*= 18 Hz), 3.42 (s, 3 H) ppm.

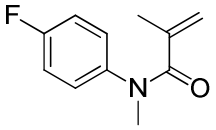
C. General procedure for the Methylation of *N*-Arylacrylamides:[4]



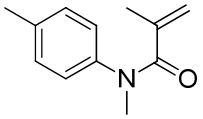
To a 250 mL round bottom flask charged with magnetic stirrer, a solution of *N*-acrylamide (20 mmol) in 32 mL THF was added to a suspension of NaH (1.2 g, 30 mmol) in 32 mL THF, under N_2. The mixture was stirred at 0 °C for 40 min. Subsequently, CH_3I (2.75 mL, 50 mmol) or BnBr (5.94 mL, 50 mmol) was added dropwise, and then mixture was brought to room temperature and stirred for 3 h. The reaction was quenched with H_2O (40 mL) and the aqueous phase was extracted with ethyl acetate (3 x 40 mL). The organic phase was washed with saturated NaCl (40 mL), dried over $MgSO_4$ and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel using ethyl acetate/hexane as eluent (15 to 30%, AcOEt). The product was characterized by ^1H, ^{13}C NMR spectroscopy and mass spectrometry.



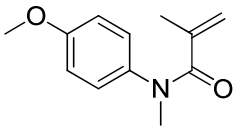
***N*-methyl-*N*-phenylmethacrylamide (1a)**: ^1H NMR ($CDCl_3$, 300Hz) δ 7.32-7.36 (m, 2H), 7.23-7.28 (m, 1H), 7.12-7.15 (m, 2H), 5.03 (s, 1H), 4.99 (s, 1H), 3.35 (s, 3H), 1.71-1.77 (m, 3H);^{13}C NMR ($CDCl_3$, 75Hz) δ 172.0, 144.6, 140.7, 129.2, 126.9, 126.5 (2C), 119.3, 37.6, 20.3 ppm.[5]



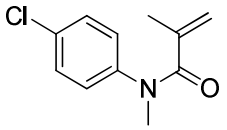
***N*-(4-fluorophenyl)-*N*-methylmethacrylamide (1b)**: ^1H NMR ($CDCl_3$, 300Hz) δ 7.06-7.11 (m, 2H), 6.96-7.04 (m, 2H), 5.02 (s, 1H), 4.95 (s, 1H), 3.29 (s, 3H), 1.73 (s, 3H); ^{13}C NMR ($CDCl_3$, 75Hz) δ 171.9, 161,2 (d, J=247,5Hz), 140.5, 128.2 (d, J=7,5Hz), 119.3, 116.1 (d, *J*=22,5Hz), 37.8, 20.3 ppm.[6]



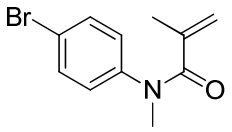
***N*-methyl-*N*-(4-tolyl)methacrylamide (1c)**: ^1H NMR (300 MHz, $CDCl_3$) δ 7.14 (d, 2H, *J* = 9), 7.01 (d, 2H, *J*= 9), 5.02 (s, 1H), 4.99 (s, 1H), 3.32 (s, 3H), 2.35 (s, 3H), 1.76 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.04, 141.98, 140.78, 136.78, 129.80, 126.30, 119.09, 37.68, 20.98, 20.33 ppm.[7]



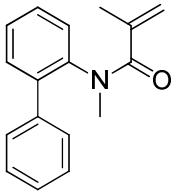
***N*-(4-methoxyphenyl)-*N*-methylmethacrylamide (1d)**: ^1H NMR ($CDCl_3$, 300Hz) δ7.03 (d, 2H, *J*=9.0Hz), 6.83 (d, 2H, *J*=9.0Hz), 4.99 (s, 1H), 4.96 (s, 1H), 3.78 (s, 3H), 3.28 (s, 3H), 1.71 (s, 3H); ^{13}C NMR ($CDCl_3$, 75Hz) δ 20.3, 37.7, 55.3, 114.2 (2C), 118.8, 127.6 (2C), 137.3, 140.8, 158.2, 172.0 ppm.[8]



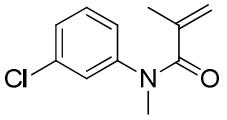
***N*-(4-chlorophenyl)-*N*-methylmethacrylamide (1e)**: ^{1}H NMR ($CDCl_3$, 300Hz) δ 7.30 (d, 2H, *J*=6.0Hz), 7.07 (d, 2H, *J*=9.0Hz), 5.06 (s, 1H), 4.97(s, 1H), 3.31 (s, 3H), 1.77 (s, 3H); ^{13}C NMR ($CDCl_3$, 75Hz) δ 20.4, 37.8, 119.8, 127.9, 129.6, 132.7, 140.6, 143.3, 172.0 ppm.[3]



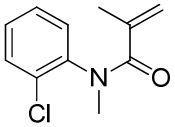
***N*-(4-bromophenyl)-*N*-methylmethacrylamide (1f)**: ^{1}H NMR ($CDCl_3$, 300Hz) δ 7.46 (d, 2H, *J*=9.0Hz), 7.01 (d, 2H, *J*=9.0Hz), 5.07 (s, 1H), 4.97(s, 1H), 3.31 (s, 3H), 1.77 (s, 3H); ^{13}C NMR ($CDCl_3$, 75Hz) δ 20.3, 37.6, 119.7, 120.4, 128.0 (2C), 132.4 (2C), 140.3, 143.7, 171.7 ppm.[3]



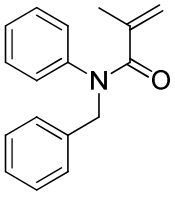
***N*-([1,1'-biphenyl]-2-yl)-*N*-methylmethacrylamide (1g)**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.41-7.26 (m, 9H), 4.93 (s, 1H), 4.67 (s, 1H), 3.28 (s, 3H), 1.31 (s, 3H) ppm. ^{13}C NMR (75 MHz, $CDCl_3$) δ 142.28, 139.87, 138.76, 138.72, 131.27, 128.85, 128.73, 128.36, 128.02, 127.60, 127.50, 119.61, 38.43, 19.66 ppm.[9]



***N*-(3-chlorophenyl)-*N*-methylmethacrylamide (1h)**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.32-7.22 (m, 2H), 7.18-7.16 (m, 1H), 7.07-7.03 (m, 1H), 5.09 (s, 1H), 5.00 (s, 1H), 3.34 (s, 3H), 1.80 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 171.70, 145.75, 140.24, 134.56, 130.14, 126.99, 126.57, 124.64, 119.71, 37.56, 20.19 ppm.[3]



***N*-(2-chlorophenyl)-*N*-methylmethacrylamide (1i)**: ^{1}H NMR (300 MHz, $CDCl_3$) δ 7.46-7.43 (m, 1H), 7,28-7,19 (m, 3H), 4.98 (s, 2H), 3.26 (s, 3H), 1.82 (s, 3H). ^{13}C NMR (75 MHz, $CDCl_3$) δ 172.05, 140.13, 132.44, 130.52, 129.72, 128.94 (2C), 127.79, 118.48, 36.26, 20.10 ppm. IR (ATR), *v* max: 2928, 1657, 1628, 1482, 1363, 1130, 1065, 765, 734 cm-1. MS-EI: m/z (%) 174 (100), 77 (15), 69 (76), 41 (74). HRMS (ESI-TOF) m/z, calculated for $C11H12ClNO+H^+$ = 210.0686. Found: 210.0670.

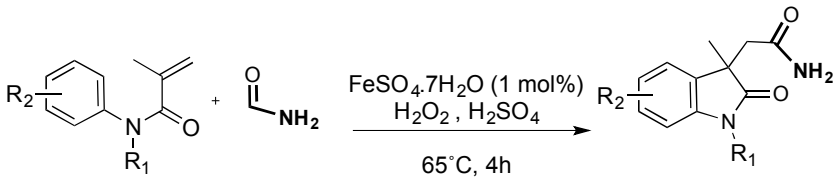


***N*-benzyl-*N*-phenylmethacrylamide (1j)**: ^{1}H NMR ($CDCl_3$, 300Hz) δ 7.26-7.19 (m, 8H), 6.98 (d, 2H, *J*=6.0Hz), 5.04 (d, 2H, *J*=6.0Hz), 4.98 (s, 2H), 1.79 (s, 3H); ^{13}C NMR ($CDCl_3$, 75Hz) δ 20.4, 53.2, 119.4, 127.1, 127.3, 127.4, 128.4, 129.1, 137.5, 140.7, 143.2, 171.8. [3]

D. General procedure for the preparation of *N*-butyl and *N*-phenyl formamides

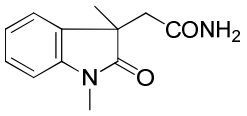
The *N*-phenylformamide was synthesized according to a procedure described in the literature[10] and *N*-butylformamide according to a procedure recently described by our research group.[11]

E. Typical procedure for carbamoylation with formamide:[12]

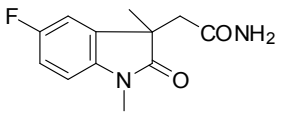


1 a-m **2, 6-17**

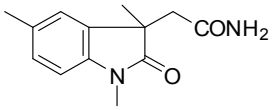
To a stirring solution of *N*-arylacrylamide (1.00 mmol) and $FeSO_4.7H_2O$ (2.8 mg, 0.01 mmol) in 5.0 mL of formamide was added sulfuric acid (56.0 µL, 1.00 mmol) followed by hydrogen peroxide (213.5 µL, 2.00 mmol). The mixture was heated to 65 ºC and stirred for 4 h. Then sodium bicarbonate (330 mg, 3.9 mmol) was added and excess of formamide was removed under reduced pressure distillation. The crude mixture was purified by flash column chromatography on silica gel using methanol/dichloromethane as eluent (10 to 20%, methanol). The product was characterized by NMR spectroscopy, mass spectrometry and high resolution mass spectrometry.



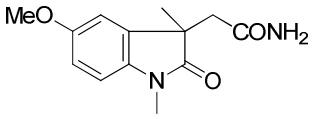
2-(1,3-dimethyl-2-oxoindolin-3-yl)acetamide (2): (starting material: 2.0 mmol; 0.401 g. Yield, 92%. yellow solid) ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.29-7.26 (m, 2H), 7.11-7.06 (m, 1H), 6.86 (d, 1H, *J*=9.0 Hz), 6.42 (br s, 1H), 5.38 (br s, 1H), 3.24 (s, 3H), 2.82 (d, 1H, *J*=15.0 Hz), 2.68 (d, 1H, *J*=15.0 Hz), 1.44 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 180.5, 171.3, 142.8, 133.2, 128.2, 122.9, 122.7, 108.4, 46.0, 43.4, 26.4, 23.5 ppm; MS-EI: m/z (%) 218 (M^+, 40), 160 (100).[13]



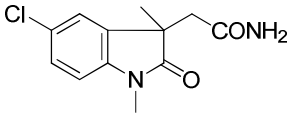
2-(5-fluor-1,3-dimethyl-2-oxoindolin-3-yl)acetamide (6): (starting material: 2.0 mmol; 0.418 g. Yield, 90%. light brown solid) ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.05-6.98 (m, 2H), 6.80-6.78 (m, 1H), 6.17 (br s, 1H), 5.25 (br s, 1H), 3.23 (s, 3H), 2.83 (d, 1H, *J*=15.0 Hz), 2.69 (d, 1H, *J*=15.0 Hz), 1.43 (s, 3H); ^{13}C NMR (CD_3OD, 75 MHz) δ 181.1, 172.6, 159.4 (d, C-F, *J*=238 Hz), 139.5, 135.2 (d, C-F, J=8 Hz), 113.6 (d, C-F, *J*=24 Hz), 110.1 (d, C-F, *J*=25 Hz), 108.9 (d, C-F, *J*=8.25 Hz), 46.2, 41.7, 25.4, 23.2 ppm; MS-EI: m/z (%) 236 (M+, 54), 178 (100). HRMS (ESI-TOF) m/z, calculated for: C12H14FN2O2 $[M+H]^+$ 237.1039, found: 237.1027.



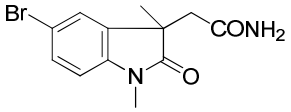
2-(1,3,5-trimethyl-2-oxoindolin-3-yl)acetamide (7): (starting material: 2.0 mmol; 0.413 g. Yield, 89%. white solid) ^{1}H NMR ($CDCl_3$, 300 MHz) δ7.09-7.07 (m, 2H), 6.75 (d, 1H, *J*=6.0Hz), 6.54(br s, 1H), 5.35 (br s, 1H), 3.22 (s, 3H), 2.80 (d, 1H, *J*=15.0 Hz), 2.66 (d, 1H, *J*=15.0 Hz), 2.34 (s, 3H), 1.44 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 180.5, 171.5, 140.3, 133.3, 132.5, 128.5, 123.6, 108.1, 46.0, 43.4, 26.5, 23.5, 21.2 ppm; MS-EI: m/z (%) 232 (M+, 38), 174 (100). HRMS (ESI-TOF) m/z, calculated for: C13H17N2O2 $[M+H]^+$ 233.1290, found: 233.1291.



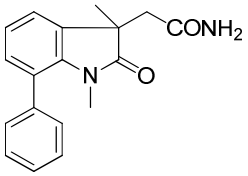
2-(5-methoxi-1,3-dimethyl-2-oxoindolin-3-yl)acetamide (8): (starting material: 1.0 mmol; 0.226 g. Yield, 91%. white solid) ^{1}H NMR ($CDCl_3$, 300 MHz) δ 6.90 (d, 2H, *J*=3.0 Hz), 6.82-6.75 (m, 2H), 6.58 (br s, 1H), 5.31 (br s, 1H), 3.79 (s, 3H), 3.22 (s, 3H), 2.80 (d, 1H, *J*=15.0 Hz), 2.67 (d, 1H, *J*=15.0 Hz), 1.45 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 180.2, 171.3, 156.3, 136.1, 134.7, 112.4, 110.3, 108.7, 55.8, 46.3, 43.4, 26.5, 23.4 ppm; MS-EI: m/z (%) 248 (M+, 82), 190 (100). HRMS (ESI-TOF) m/z, calculated for: C13H17N2O3 $[M+H]^+$ 249.1239, found: 249.1232.[14]



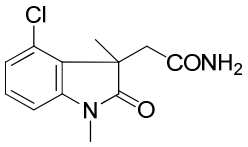
2-(5-chloro-1,3-dimethyl-2-oxoindolin-3-yl)acetamide (9): (starting material: 5.0 mmol; 1.102 g. Yield, 87%. light brown solid) ^{1}H NMR (CD_3OD, 300 MHz) δ 7.31-7.25 (m, 2H), 6.94 (d, 1H, *J*=6.0 Hz) , 3.30 (s, 2H), 3.21 (s, 3H), 2.86 (s, 2H), 1.31 (s, 3H); ^{13}C NMR (CD_3OD, 75 MHz) δ 180.96, 172.56, 142.26, 135.31, 127.51, 127.43, 122.46, 109.34, 45.99, 41.69, 25.38, 23.17 ppm; MS-EI: m/z (%) 252 (M+, 60), 194 (100). HRMS (ESI-TOF) m/z, calculated for: C12H14ClN2O2 $[M+H]^+$ 253.0744, found: 253.0742.



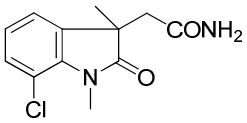
2-(5-bromo-1,3-dimethyl-2-oxoindolin-3-yl)acetamide (10): (starting material: 1.0 mmol; 0.220 g. Yield, 72%. white solid) ^{1}H NMR ($CDCl_3$, 300 MHz) δ7.42-7.37 (m, 2H), 6.74 (d, 1H, *J*=6.0 Hz), 6.04 (br s, 1H), 5.25 (br s, 1H), 3.23 (s, 3H), 2.86 (d, 1H, *J*=15.0 Hz), 2.68 (d, 1H, *J*=15.0 Hz), 1.42 (s, 3H); ^{13}C NMR (CD_3OD, 75 MHz) δ 180.8, 172.6, 142.8, 135.7, 130.5, 125.2, 114.6, 109.9, 45.9, 41.7, 25.4, 23.2 ppm; MS-EI: m/z (%) 296 e 298 (M+, 64), 238 (100). HRMS (ESI-TOF) m/z, calculated for: C12H14BrN2O2 $[M+H]^+$ 297.0239, found: 297.0240.



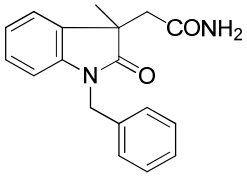
2-(1,3-dimethyl-2-oxo-7-phenylindolin-3-yl)acetamide 11): (starting material: 4.0 mmol; 1.006 g. Yield, 85%. white solid) ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.43-7.35 (m, 5H), 7.28-7.25 (m, 1H), 7.13-7.06 (m, 2H), 6.52 (br s, 1H), 5.40 (br s, 1H), 2.89-2.71 (m, 5H), 1.50 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ181.6, 171.4, 139.7, 138.8, 134.2, 131.2, 129.9, 127.8, 127.7, 125.7, 122.2(2C), 121.6 (2C), 45.3, 43.7, 30.4, 23.9 ppm; MS-EI: m/z (%) 294 (M+, 46), 236 (100). HRMS (ESI-TOF) m/z, calculated for: C18H19N2O2 $[M+H]^+$ 295.1446, found: 295.1450.



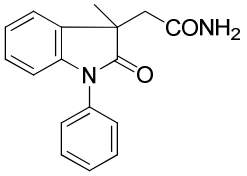
2-(4-chloro-3-methyl-2-oxoindolin-3-yl)acetamide (12+12' = 10:1) (major isomer): (starting material: 2.0 mmol; 0.321 g. Yield, 64%. white solid) ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.24-7.18 (m, 1H), 6.98 (d, 1H, *J*=9.0 Hz), 6.76 (d, 1H, *J*=6.0 Hz), 5.62 (br s, 1H), 5.12 (br s, 1H), 3.24 (s, 3H), 3.20 (d, 1H, *J*=15.0 Hz), 3.00 (d, 1H, *J*=15.0 Hz), 1.50 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 179.6, 171.0, 145.4, 129.9, 129.4, 129.0, 123.3, 106.9, 47.4, 40.9, 26.7, 21.3 ppm; MS-EI: m/z (%) 252 (M+, 50), 194 (100). HRMS (ESI-TOF) m/z, calculated for: C12H14ClN2O2 $[M+H]^+$ 253.0744, found: 253.0732.



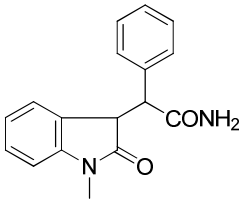
2-(7-Chloro-1,3-dimethyl-2-oxoindolin-3-yl)acetamide (13): (starting material: 4.0 mmol; 0.369 g. Yield, 73%. white solid) ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.20 (d, 1H, *J*=9.0 Hz), 7.14 (d, 1H, *J*=9.0 Hz), 7.00-6.95 (m, 1H), 6.16 (br s, 1H), 5.33 (br s, 1H), 3.61 (s, 3H), 2.85 (d, 1H, *J*=15.0 Hz), 2.68 (d, 1H, *J*=15.0 Hz), 1.41 (s, 3H); ^{13}C NMR (CD_3OD, 75 MHz) δ 180.8, 171.0, 138.9, 136.0, 130.5, 123.5, 121.1, 115.8, 45.8, 43.4, 29.8, 24.1 ppm; MS-EI: m/z (%) 252 (M+, 52), 194 (100). HRMS (ESI-TOF) m/z, calculated for: C12H14ClN2O2 $[M+H]^+$ 253.0744, found: 253.0740.



2-(1-benzyl-3-methyl-2-oxoindolin-3-yl)acetamide (14): (starting material: 1.0 mmol; 0.279 g. Yield, 95%. dark yellow solid) ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.32 -7.26 (m, 6H), 7.18-7.13 (m, 1H), 7.07-7.02 (m, 1H), 6.74 (d, 1H, *J*=9.0 Hz), 6.27 (br. s, 1H), 5.34 (br s, 1H), 4.94 (s, 2H), 2.89 (d, 1H, *J*=12.0 Hz), 2.74 (d, 1H, *J*=15.0 Hz), 1.49 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 180.6, 171.2, 141.9, 135.7, 133.2, 128.8 (2C), 128.1, 127.6, 127.1 (2C), 122.9, 122.7, 109.4, 46.1, 43.9, 43.2, 24.1 ppm; MS-EI: m/z (%) 294 (M+, 18), 91 (100). HRMS (ESI-TOF) m/z, calculated for: C18H19N2O2 $[M+H]^+$ 295.1446, found: 295.1446.

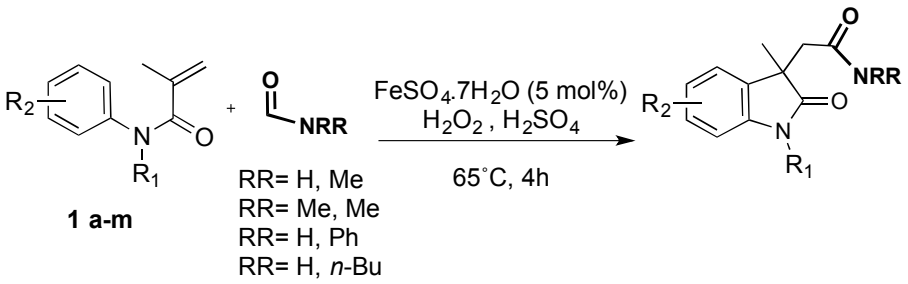


2-(3-methyl-2-oxo-1-phenylindolin-3-yl)acetamide (15): (starting material: 0.5 mmol; 0.119 g. Yield, 85%. light yellow solid) ^{1}H NMR ($DMSO_3$, 300 MHz) δ 7.45-7.21(m, 6H), 7.02 (*t*, 1H, *J* = 6 Hz), 6.90 (*t*, 1H, *J* = 6 Hz), 6.52 (*d*, 1H, *J* = 9 Hz), 2.75 (*d*, 1H, *J* = 15 Hz), 2.62 (*d*, 1H, *J* = 15 Hz), 1.19 (s, 3H); ^{13}C NMR (DMSO, 75 MHz) δ 179.8, 170.9, 143.9, 135.5, 133.8, 129.7 (2C), 128.0, 127.8, 127.2 (2C), 123.0, 122.4, 108.7, 45.4, 43.1, 24.9 ppm; MS-EI: m/z (%) 280 (M+, 72), 236 (47), 235 (67), 222 (100). HRMS (ESI-TOF) m/z, calculated for: C17H17N2O2 $[M+H]^+$ 281.1290, found: 281.1272.

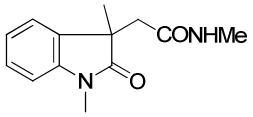


2-(1-methyl-2-oxoindolin-3-yl)-2-phenylacetamide (16:16' = 10:1) (major isomer): (starting material: 0.5 mmol; 0.076 g. Yield, 54%. white solid) ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.31-7.25 (m, 3H), 7.23-7.20 (m, 2H), 7.13-7.05 (m, 4H), 6.23 (br s, 1H), 5.34 (br s, 1H), 4.88 (br s, 1H), 3.80 (*d*, 1H, *J* = 5 Hz), 3.43 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 168.5, 167.3, 140.9, 138.6, 129.5, 128.9 (2C), 128.1, 127.6 (2C), 127.2, 124.1, 115.0, 55.4, 42.8, 30.1 ppm; MS-EI: m/z (%) 280 (M^+, 2.6), 236 (100). HRMS (ESI-TOF) m/z, calculated for: C17H17N2O2 $[M+H]^+$ 281.1290, found: 281.1271.

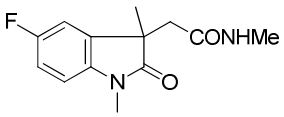
F. Typical procedure for carbamoylation with *N*-alkyl-formamide or *N*-aryl-formamide :[8]



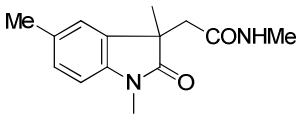
To a stirring solution of *N*-arylacrylamide (1.00 mmol) and $FeSO_4.7H_2O$ (13.9 mg, 0.05 mmol) was added the corresponding formamide (5.0 mL, in case of *N*-methylformamide and *N,N*-dimethylformamide; 2.5 mL or 2.5 g, in case of *N*-butylformamide or *N*-phenylformamide). When required, 3 mL of *t*-ButOH were added as co-solvent (see Table 1). Then, concentrated sulfuric acid 96% (56 µL, 1.00 mmol) followed by hydrogen peroxide 29% (214 µL, 2.00 mmol) were added. The mixture was heated to 65 ºC and stirred for 4 h. Then, sodium bicarbonate (330.0 mg, 3.9 mmol) was added and excess of *N*-methyl-formamide was removed under reduced pressure distillation. The crude mixture was purified by flash column chromatography on silica gel using methanol/dichloromethane as eluent (10 to 20%, methanol). The product was characterized by NMR spectroscopy, mass spectrometry and high resolution mass spectrometry.



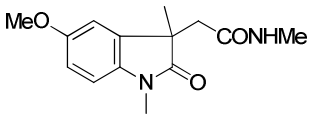
2-(1,3-dimethyl-2-oxoindolin-3-yl)-*N*-methylacetamide (3): (starting material: 5.0 mmol; 1.086 g. Yield, 94%. yellow solid) ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.30-7.24 (m, 2H), 7.07 (t, 1H, *J*=9.0 Hz), 6.86 (d, 1H, *J*=6.0 Hz), 6.39 (br s, 1H), 3.25 (s, 3H), 2.78 (d, 1H, *J*=15.0 Hz), 2.70-2.63 (m, 4H, *J*=15.0 Hz), 1.42 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 180.7, 169.6, 142.8, 133.5, 128.1, 122.8, 122.6, 108.3, 46.0, 43.7, 26.4, 26.2, 23.4 ppm; MS-EI: m/z (%) 232 (M+, 54), 160 (100). HRMS (ESI-TOF) m/z, calculated for: C13H17N2O2 $[M+H]^+$ 233.1290, found: 233.1281.



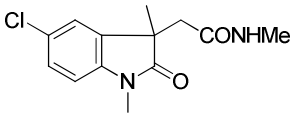
2-(5-fluor-1,3-dimethyl-2-oxoindolin-3-yl)-*N*-methylacetamide (18): (starting material: 1.0 mmol; 0.237 g. Yield, 95%. light brown solid) ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.04-6.93 (m, 2H), 6.77 (dd, 1H, *J*=3.0 Hz, 9.0 Hz), 6.13 (br s, 1H), 3.23 (s, 3H), 2.80 (d, 1H, *J*=15.0 Hz), 2.68- 2.63 (m, 4H), 1.39 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 180.2, 169.4, 159.2 (d, C-F, *J*=240 Hz), 139.0, 135.0 (d, C-F, *J*=7.5 Hz), 114.0 (d, C-F, *J*=22.5 Hz), 110.8 (d, C-F, *J*=30 Hz), 108.6 (d, C-F, *J*=7.5 Hz), 46.4, 43.1, 26.4, 26.0, 23.7 ppm; MS-EI: m/z (%) 250 (M+, 68), 178 (100). HRMS (ESI-TOF) m/z, calculated for: C13H16FN2O2 $[M+H]^+$ 251.1195. Found: 251.1180.



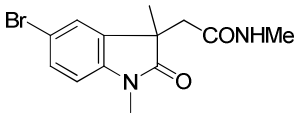
2-(1,3,5-trimethyl-2-oxoindolin-3-yl)*N*-methyl-acetamide (19): (starting material: 4.0 mmol; 0.927 g. Yield, 94%. light yellow solid) ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.08-7.06 (m,2H), 6.74 (d, 1H, *J*=9.0 Hz), 6.49 (br s, 1H), 3.21 (s, 3H), 2.72-2.66 (m, 5H), 2.33 (s, 3H), 1.40 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 180.7, 169.8, 140.3, 133.6, 132.3, 128.4, 123.6, 108.0, 46.1, 43.7, 26.4, 26.2, 23.4, 21.2 ppm; MS-EI: m/z (%) 246 (M+, 50), 174 (100). HRMS (ESI-TOF) m/z, calculated for: C14H19N2O2 $[M+H]^+$ 247.1446, found: 247.1439.



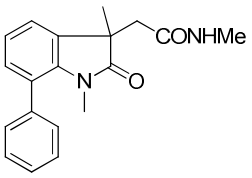
2-(5-methoxi-1,3-dimethyl-2-oxoindolin-3-yl)-*N*-methylacetamide (20): (starting material: 4.0 mmol; 0.997 g. Yield, 95%. white solid) ^{1}H NMR ($CDCl_3$, 300 MHz) δ 6.88 (d, 1H, *J*=3.0 Hz), 6.74-6.81 (m,2H), 6.47 (br s, 1H), 3.79 (s, 3H), 3.22 (s, 3H), 2.70-2.78 (m,4H), 2.64 (d, 1H, *J*=15.0 Hz), 1.41 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 180.4, 169.7, 156.1, 136.2, 134.9, 112.2, 110.3, 108.5, 55.8, 46.4, 43.6, 26.4, 26.1, 23.5 ppm; MS-EI: m/z (%) 262 (M+, 90), 190 (100). HRMS (ESI-TOF) m/z, calculated for: C14H19N2O3 $[M+H]^+$ 263.1396, found 263.1385.[11]



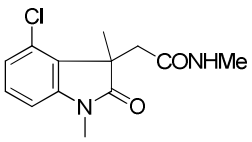
2-(5-chloro-1,3-dimethyl-2-oxoindolin-3-yl)-*N*-methylacetamide (21): (starting material: 2.0 mmol; 0469 g. Yield, 88%. white solid) ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.22-7.26 (m, 2H), 6.77 (d, 1H, *J*=6.0 Hz), 5.96 (br s, 1H), 3.23 (s, 3H), 2.82 (d, 1H, *J*=15.0 Hz), 2.62-2.68 (m, 4H), 1.38 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 180.1, 169.2, 141.7, 135.2, 128.0, 127.9, 123.1, 109.2, 46.2, 43.3, 26.5, 26.2, 23.7 ppm; MS-EI: m/z (%) 266 (M+, 70), 194(100). HRMS (ESI-TOF) m/z, calculated for: C13H16ClN2O2 $[M+H]^+$ 267.0900, found: 267.0896.



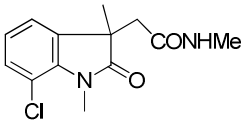
2-(5-bromo-1,3-dimethyl-2-oxoindolin-3-yl)-*N*-methylacetamide (22): (starting material: 2.0 mmol; 0.560 g. Yield, 89%. white solid) ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.34-7.40 (m, 2H), 6.72 (d, 1H, *J*=9.0 Hz), 5.97 (br s, 1H), 3.22 (s, 3H), 2.82 (d, 1H, *J*=15.0 Hz), 2.62-2.68 (m, 4H), 1.38 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ180.0, 169.2, 142.2, 135.6, 130.9, 125.8, 115.2, 109.7, 46.2, 43.3, 26.5, 26.2, 23.8 ppm; MS-EI: m/z (%) 310 (M+, 56), 238 (100). HRMS (ESI-TOF) m/z, calculated for: C13H16BrN2O2 $[M+H]^+$ 311.0395, found: 311.0394.



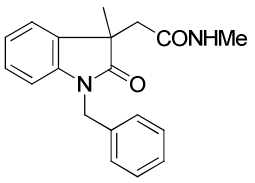
2-(1,3-dimethyl-2-oxo-7-phenylindolin-3-yl)-*N*-methylacetamide (23): (starting material: 4.0 mmol; 1.007 g. Yield, 81%. light yellow solid) ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.35 (s, 5H), 7.21-7.24 (m, 1H), 7.00-7.07 (m, 2H), 6.67 (br s, 1H), 2.63-2.87 (m, 8H), 1.42 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 181.9, 180.3, 174.8, 169.9, 139.9, 138.9, 134.5, 131.0, 129.9, 127.8, 127.6, 125.5, 121.9, 121.6, 45.4, 43.7, 30.4, 26.1, 24.2 ppm.; MS-EI: m/z (%) 308 (M+, 84), 236 (100). HRMS (ESI-TOF) m/z, calculated for: C19H21N2O2 $[M+H]^+$ 309.1603, found: 309.1604.



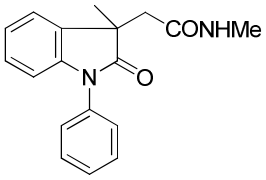
2-(4-chloro-1,3-dimethyl-2-oxoindolin-3-yl)-*N*-methylacetamide (24:24' = 10:1) (major isomer): (starting material: 4.0 mmol; 0.779 g. Yield, 73%. light yellow solid) ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.14-7.21 (m, 1H), 6.93 (d, 1H, *J*=6.0Hz), 6.74 (d, 1H, *J*=9.0Hz), 5.79 (br s, 1H), 3.24 (s, 3H), 3.15 (d, 1H, *J*=15.0Hz), 2.95 (d, 1H, *J*=15.0Hz), 2.56 (d, 3H, *J*=6.0Hz), 1.45 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 179.8, 169.5, 145.5, 129.7, 129.3, 129.2, 123.1, 106.8, 47.4, 41.5, 26.6, 26.1, 21.4 ppm; MS-EI: m/z (%) 266 (M+, 50), 194 (100). HRMS (ESI-TOF) m/z, calculated for: C13H16ClN2O2 $[M+H]^+$ 267.0900, found: 267.0887.



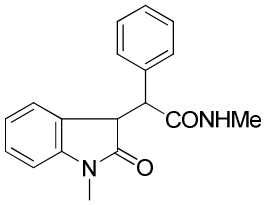
2-(7-Chloro-1,3-dimethyl-2-oxoindolin-3-yl)-*N*-methylacetamide (25): (starting material: 2.0 mmol; 0.347 g. Yield, 65%. white solid) ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.10-7.19 (m, 2H), 6.93-6.98 (m, 1H), 6.09 (br, 1H), 3.61 (s, 3H), 2.84 (d, 1H, *J*=15.0 Hz), 2.62-2.68 (m, 4H), 1.38 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 180.9, 169.4, 139.0, 136.3, 130.4, 123.4, 121.0, 115.7, 45.8, 43.6, 29.8, 26.2, 24.2 ppm; MS-EI: m/z (%) 266 (M+, 60), 194 (100). HRMS (ESI-TOF) m/z, calculated for: C13H16ClN2O2 $[M+H]^+$ 267.0900, found: 267.0897.



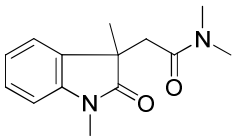
2-(1-Benzyl-3-Methyl-2-oxoindolin-3-yl)-*N*-methylacetamide (26): (starting material: 1.0 mmol; 0.251 g. Yield, 89%. dark yellow solid) ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.29-7.31 (m, 6H), 7.13-7.18 (m, 1H), 7.02-7.07 (m, 1H), 6.74 (d, 1H, J=6.0 Hz), 6.27 (br s, 1H), 4.98 (d, 1H, *J*=18 Hz), 4.92 (d, 1H, *J*=15.0 Hz), 2.84 (d, 1H, *J*=15.0 Hz), 2.72 (d, 1H, *J*=12.0 Hz), 2.66 (d, 3H, *J*=3.0 Hz), 1.48 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 180.8, 169.6, 141.8, 135.8, 133.3, 128.8 (2C), 128.1, 127.6, 127.2 (2C), 122.9, 122.8, 109.3, 46.3, 43.8, 43.7, 26.2, 24.1 ppm; MS-EI: m/z (%) 308 (M+, 28), 207 (52), 91 (100). HRMS (ESI-TOF) m/z, calculated for: C19H21N2O2 $[M+H]^+$ 309.1603, found: 309.1600.



N-methyl-2-(3-methyl-2-oxo-1-phenylindolin-3-yl)acetamide (27): (starting material: 0.5 mmol; 0.100 g. Yield, 68%. light yellow solid) ^{1}H NMR ($CHCl_3$, 300 MHz) δ 7.55-7.38 (m, 5H), 7.31-7.27 (m, 1H), 7.19 (*t*, 1H, *J* = 9 Hz), 7.09 (*t*, 1H, *J* = 9 Hz), 6.80 (*d*, 1H, *J* = 9 Hz), 6.24 (br s, 1H), 2.92 (*d*, 1H, *J* = 15 Hz), 2.76 (*d*, 1H, *J* = 15 Hz), 2.64 (*d*, 3H, *J* = 3 Hz), 1.51 (*s*, 3H); ^{13}C NMR ($CHCl_3$, 75 MHz) δ 180.3, 169.6, 143.1, 134.6, 133.1, 129.6 (2C), 128.1, 128.0, 126.7 (2C), 123.1, 122.8, 109.5, 46.2, 44.0, 26.2, 24.1 ppm; MS-EI: m/z (%) 294 (M+, 80), 236 (39), 222 (100). HRMS (ESI-TOF) m/z, calculated for: C18H19N2O2 $[M+H]^+$ 295.1446, found: 295.1433.

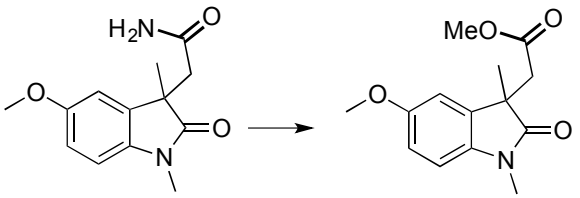


N-methyl-2-(1-methyl-2-oxoindolin-3-yl)-2-phenylacetamide (28): (starting material: 0.5 mmol; 0.077 g. Yield, 53%. yellow solid) ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.31-7.17 (m, 5H), 7.08-7.03 (m, 4H), 6.38 (br s, 1H), 4.89 (*d*, 1H, *J* = 3 Hz), 3.74 (*d*, 1H, *J* = 3 Hz), 3.41 (s, 3H), 2.69 (*d*, 3H, *J* = 3 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 167.6, 166.6, 141.3, 138.6, 129.5, 128.9 (2C), 128.0, 127.5 (2C), 127.1, 124.2, 114.9, 55.8, 42.7, 30.0, 26.6 ppm; MS-EI: m/z (%) 294 (M^+, 5), 236 (100), 208 (40). HRMS (ESI-TOF) m/z, calculated for: C18H19N2O2 $[M+H]^+$ 295.1446, found: 295.1447.



2-(1,3-dimethyl-2-oxoindolin-3-yl)-N,N-dimethylacetamide (30): (starting material: 0.5 mmol; 0.0525 g. Yield, 43%. yellow liquid) ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.34-7.09 (m, 2H), 7.00 (*t*, 1H, *J*=10.0 Hz), 6.90-6.85 (m, 1H), 3.27 (s, 3H), 3.03-2.94 (m, 5H), 2.76 (s, 3H), 1.36 (s, 3H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 181.0, 168.9, 143.8, 134.2, 127.6, 121.9, 121.4, 108.1, 45.7, 40.7, 37.2, 35.3, 26.4, 24.9 ppm; MS-EI: m/z (%) 246 (M^+, 67), 160 (100). HRMS (ESI-TOF) m/z, calculated for: C14H19N2O2 $[M+H]^+$ 247.1446, found: 247.1433.

G. Procedure for direct conversion of 8 to the corresponding methyl ester, 31:[15]



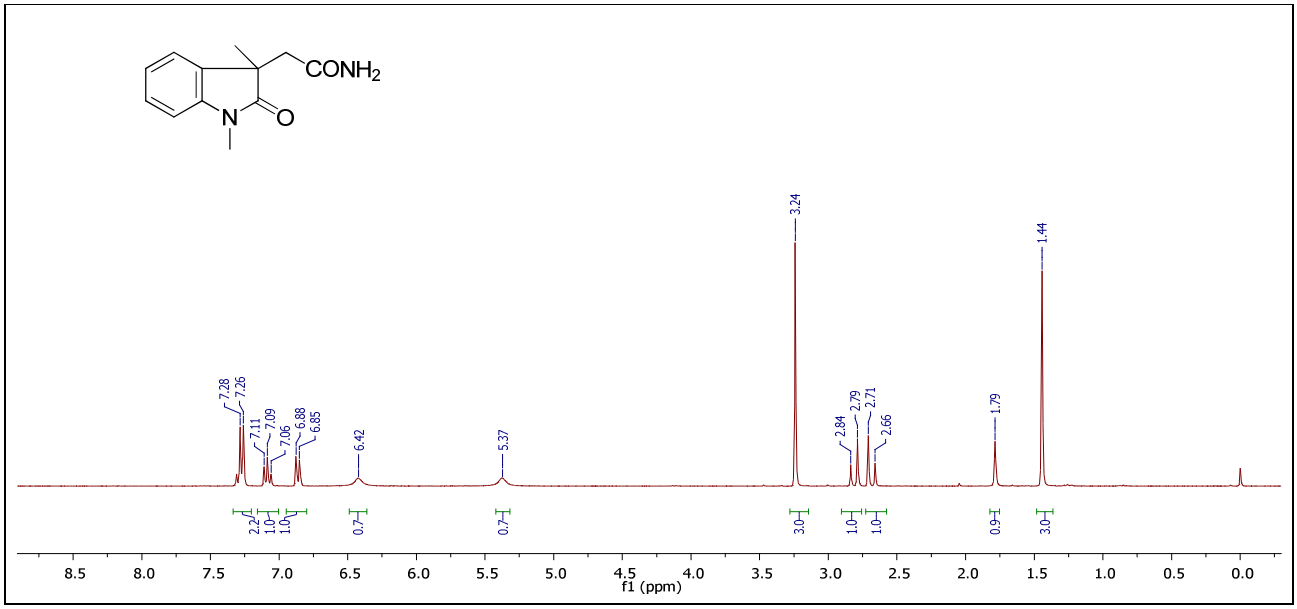
8 **31**

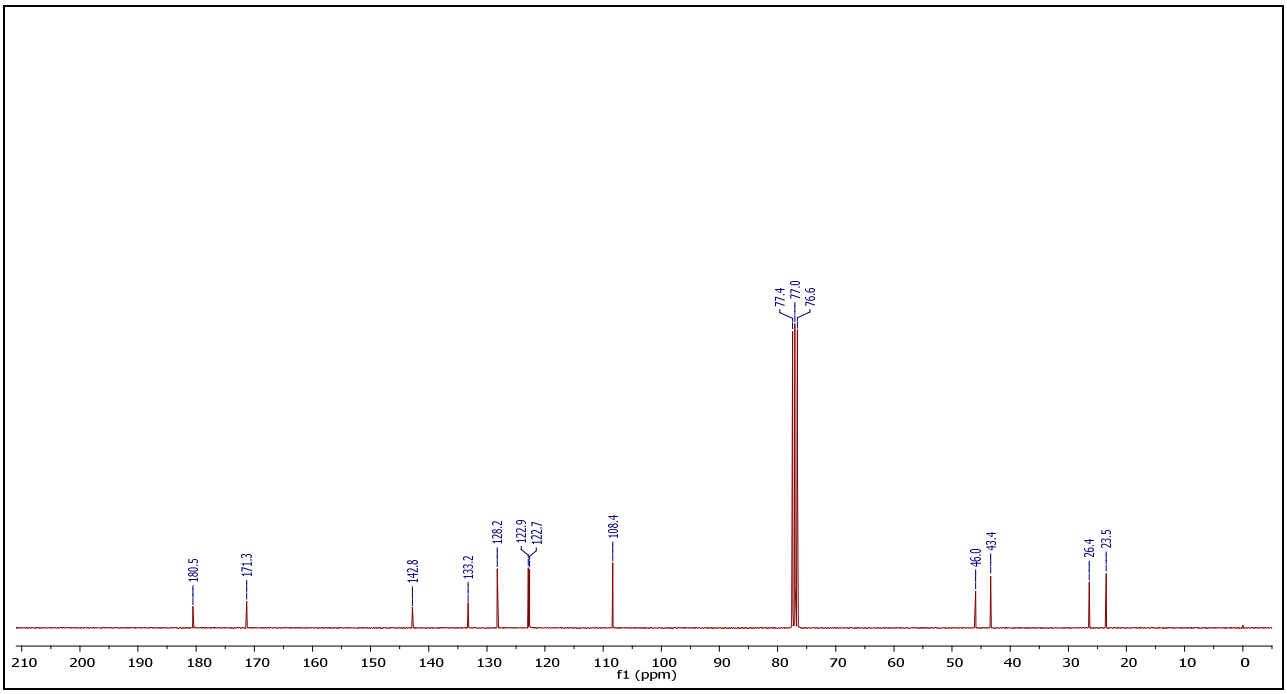
To the cooled methanol (20 mL, -15 ºC) was added the $SOCl_2$ (13.0 mg, 0.12 mmol), the mixture was stirred at -0ºC for 30 min., then the 2-(5-methoxi-1,3-dimethyl-2-oxoindolin-3-yl)acetamide (**8**) (24.8 mg, 0.1 mmol) was added into the solution. This reaction temperature was then warmed up to 50ºC for 2 days. Then a mixture of water/methanol (1/5, v/v, 5 mL) was added into the solution and then the mixture was extract by ethyl acetate three times (3 x 25 mL). The combined organic phase was dried over $MgSO_4$ and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel using methanol/dichloromethane as eluent (10%, methanol). The product was characterized by ^{1}H and ^{13}C NMR spectroscopy and mass spectrometry.

Methyl 2-(5-Methoxy-1,3-dimethyl-2-oxoindolin-3-yl)acetate (31): ^{1}H NMR (300 MHz, $CDCl_3$) δ 6.83-6.81 (m, 1H), 6.79-6.75 (m, 2H), 3.78 (s, 3H), 3.48 (s, 3H), 3.23 (s, 3H), 3.00 (d, 1H, J = 15.0 Hz), 2.83 (d, J =18.0 Hz, 1H), 1.37 (s, 3H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 179.5, 170.2, 155.8, 137.1, 134.3, 111.8, 110.1, 108.3, 55.7, 51.6, 45.8, 41.2, 26.4, 24.2; MS-EI: m/z (%) 263 (M+, 86), 248 (39), 190 (100), 175 (16).[16]

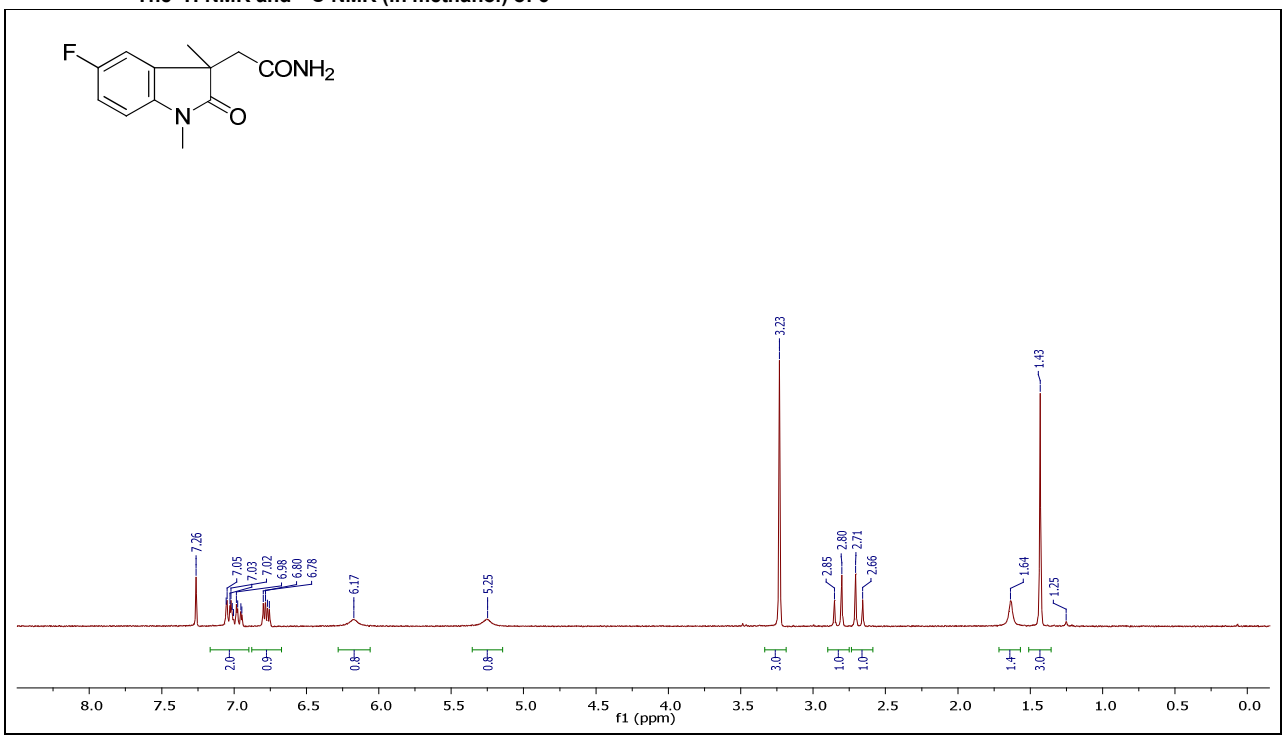
H. NMR Spectra for the compounds prepared

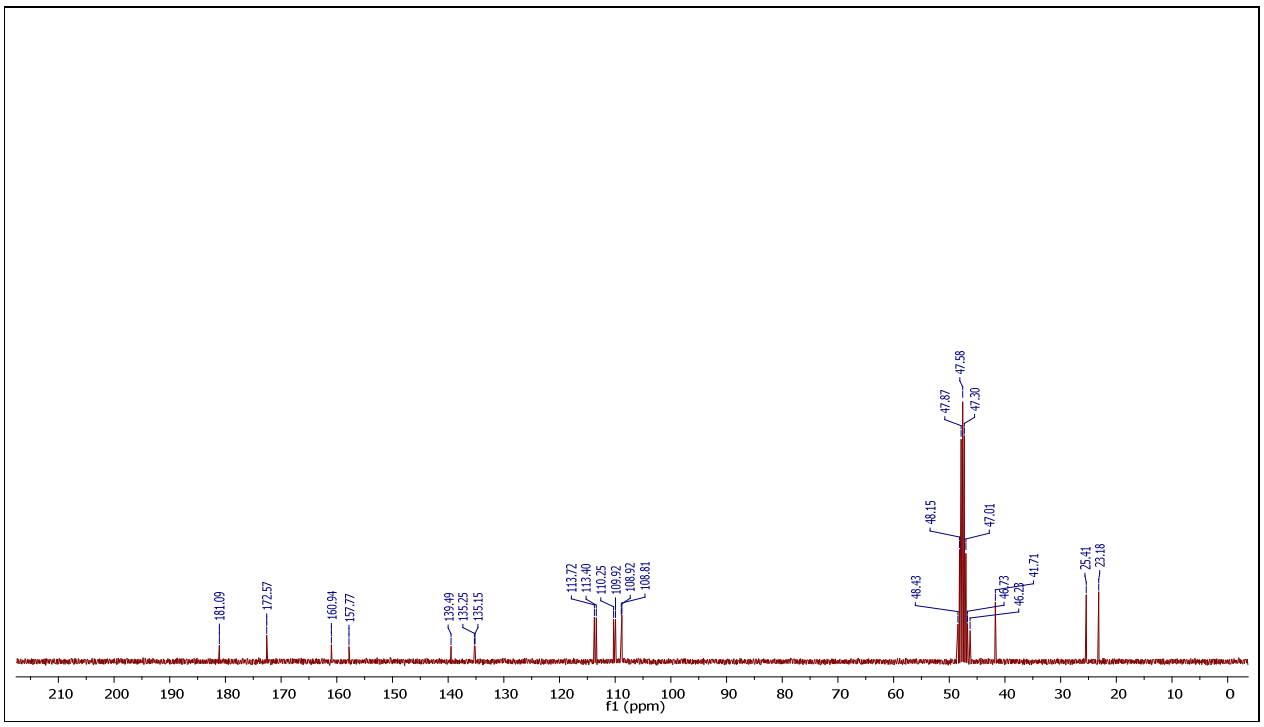
The ^{1}H NMR and ^{13}C NMR of 2



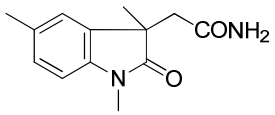


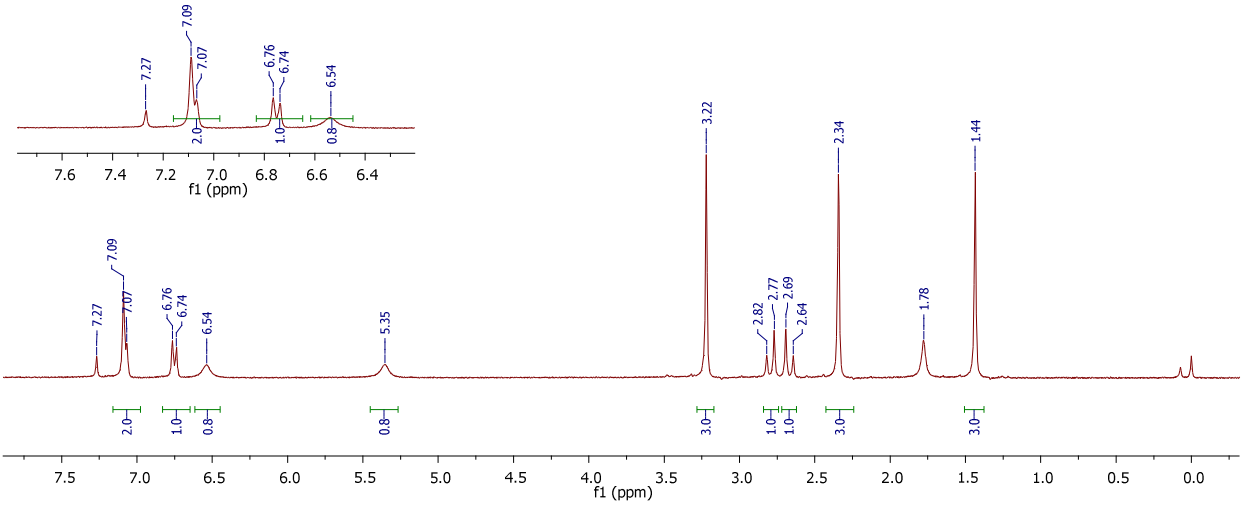
The ^{1}H NMR and ^{13}C NMR (in methanol) of 6

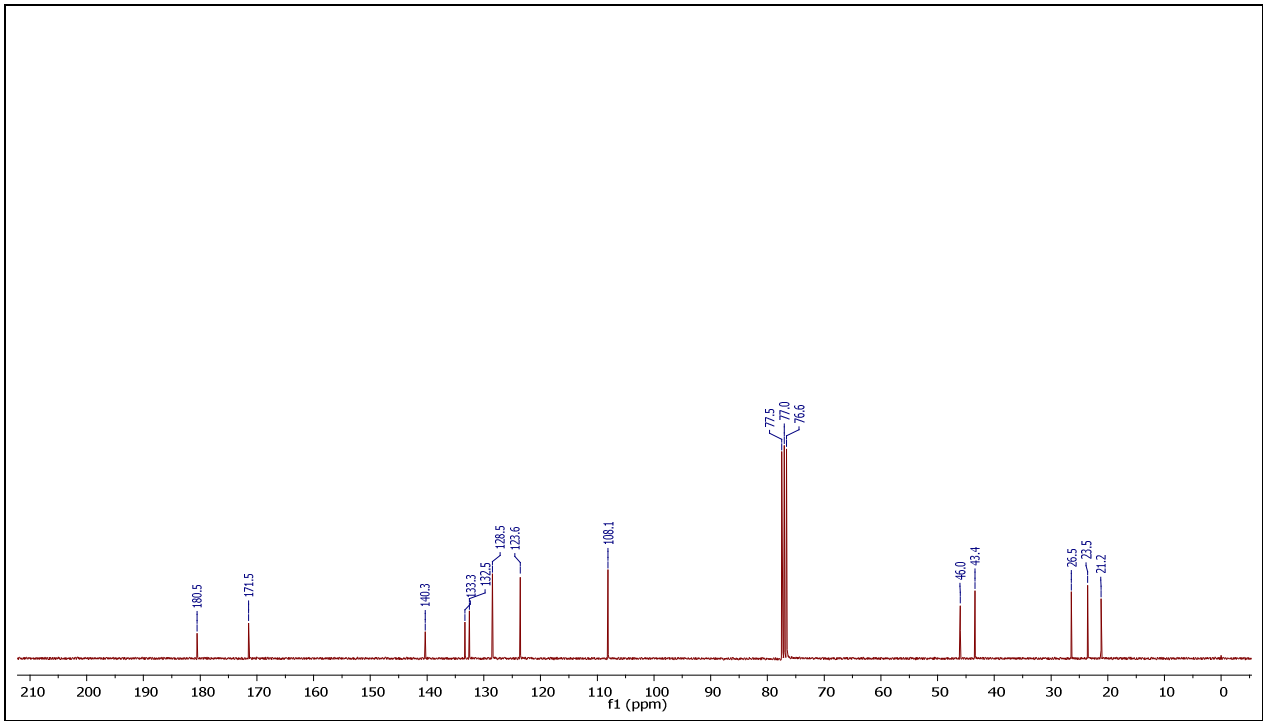




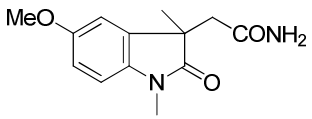
The ^{1}H NMR and ^{13}C NMR of 7

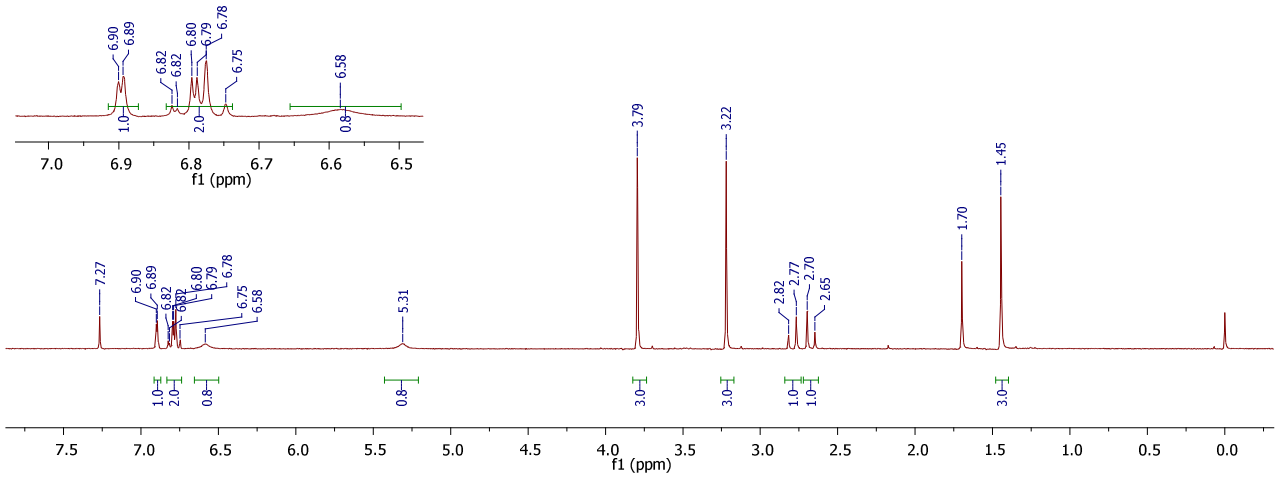


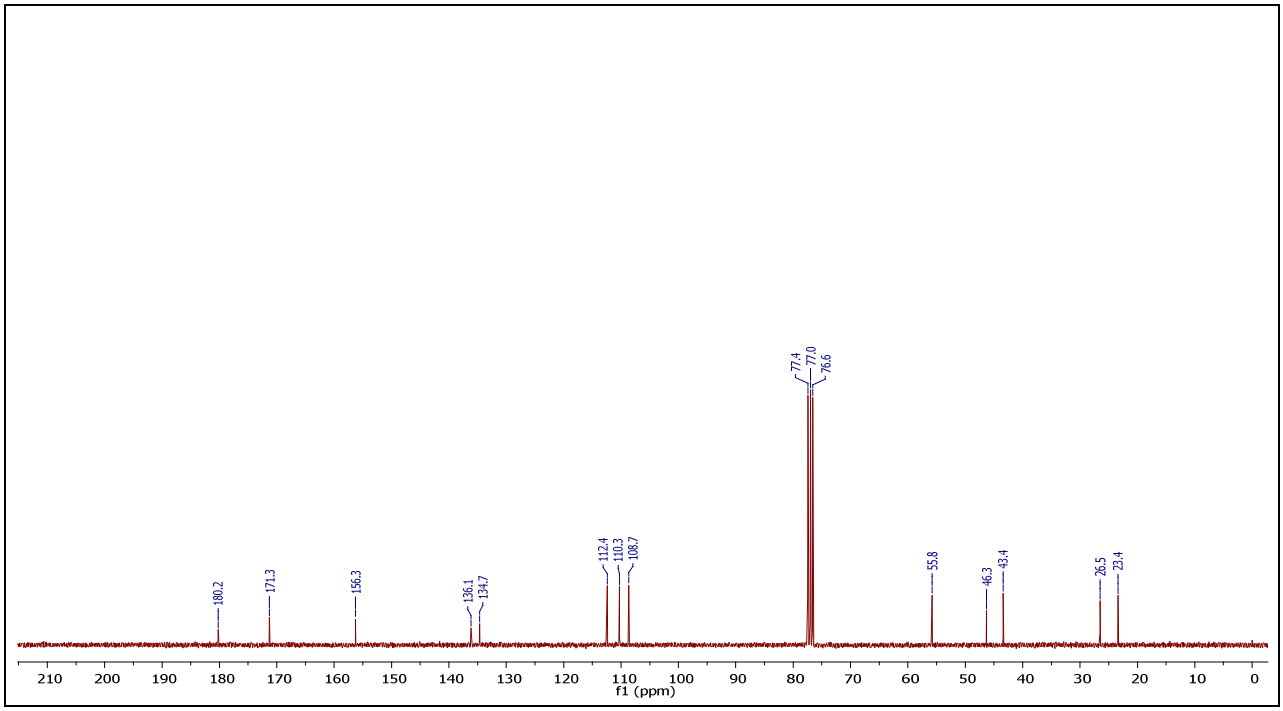




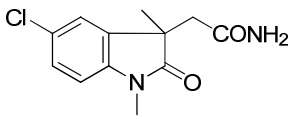
The ^{1}H NMR and ^{13}C NMR of 8

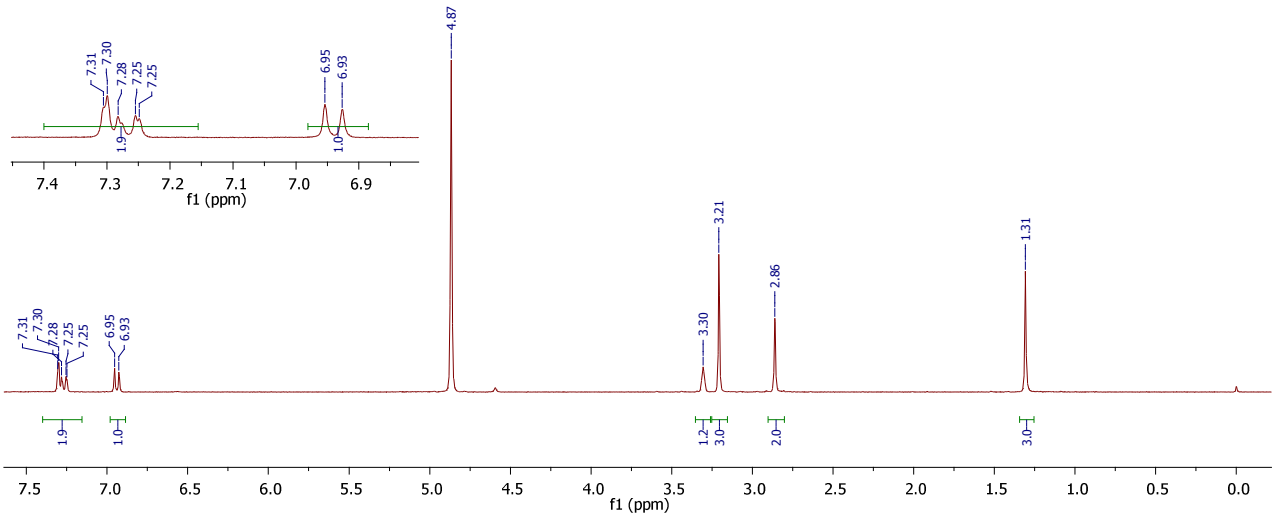


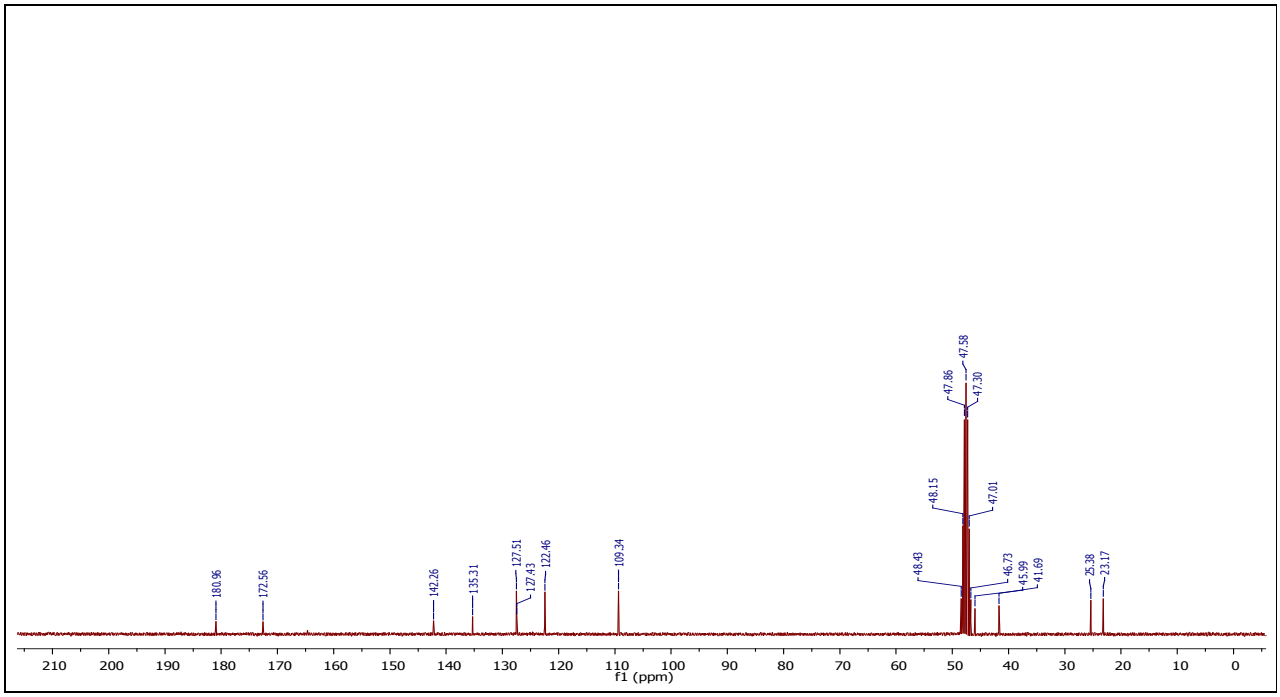




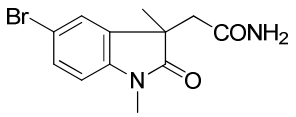
The ^{1}H NMR and ^{13}C NMR (in Methanol) of 9

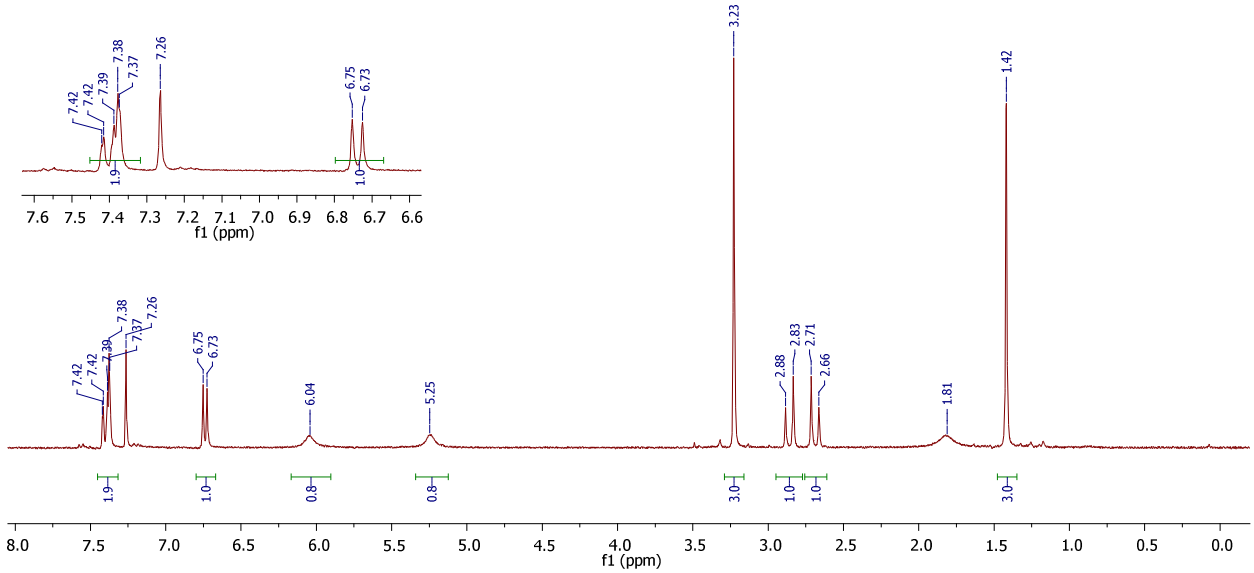


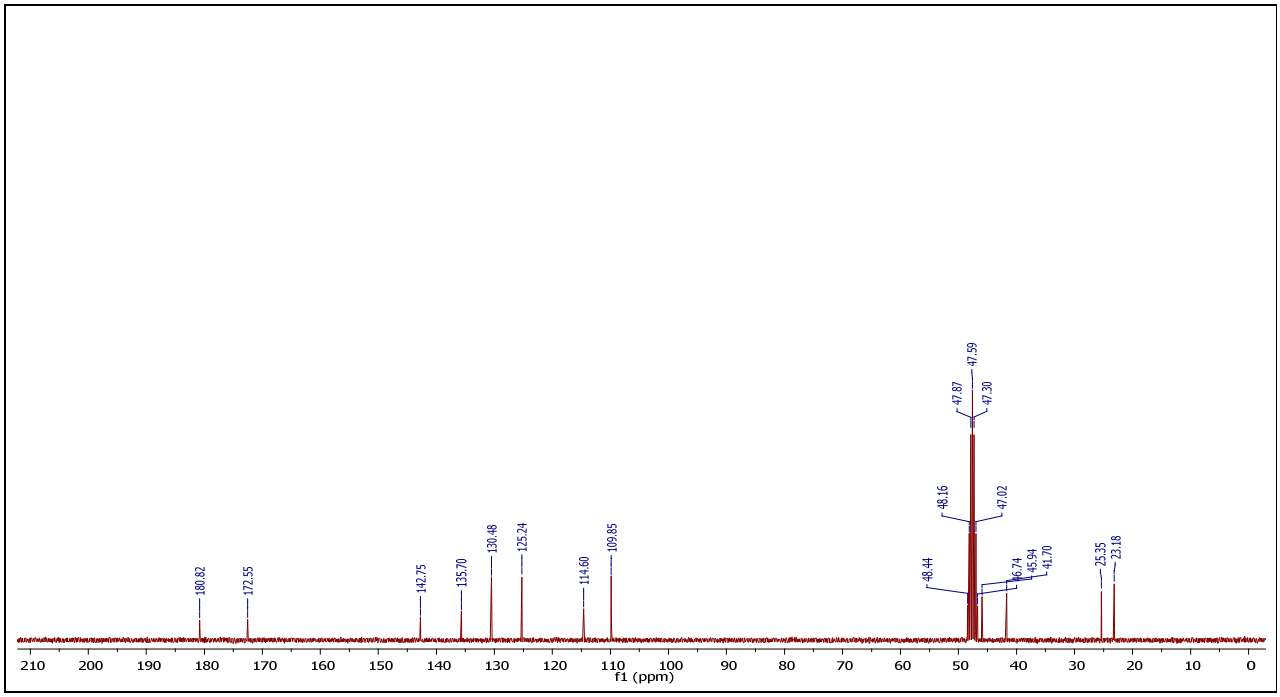




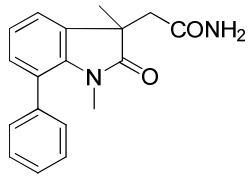
The ^{1}H NMR and ^{13}C NMR of 10

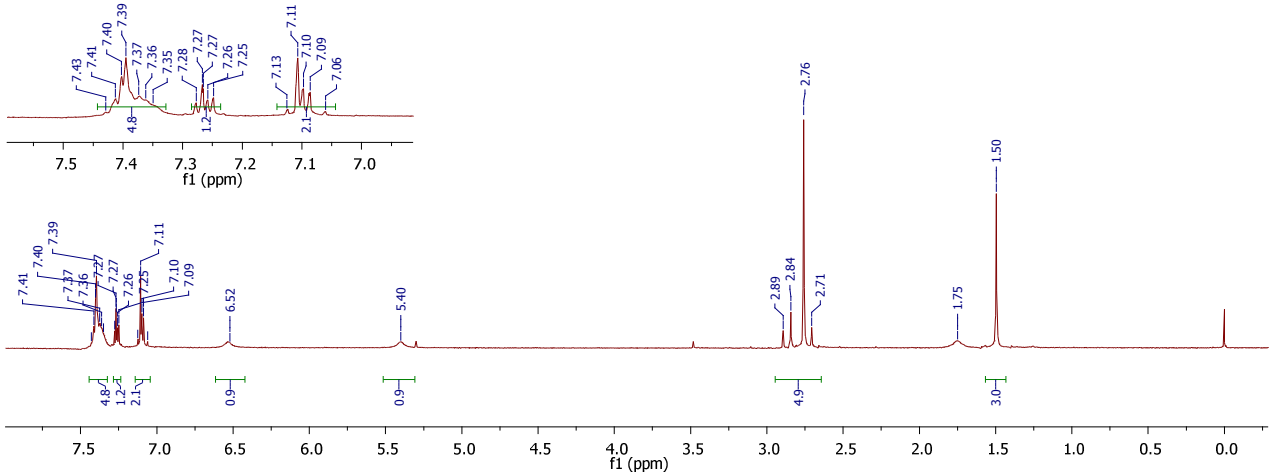


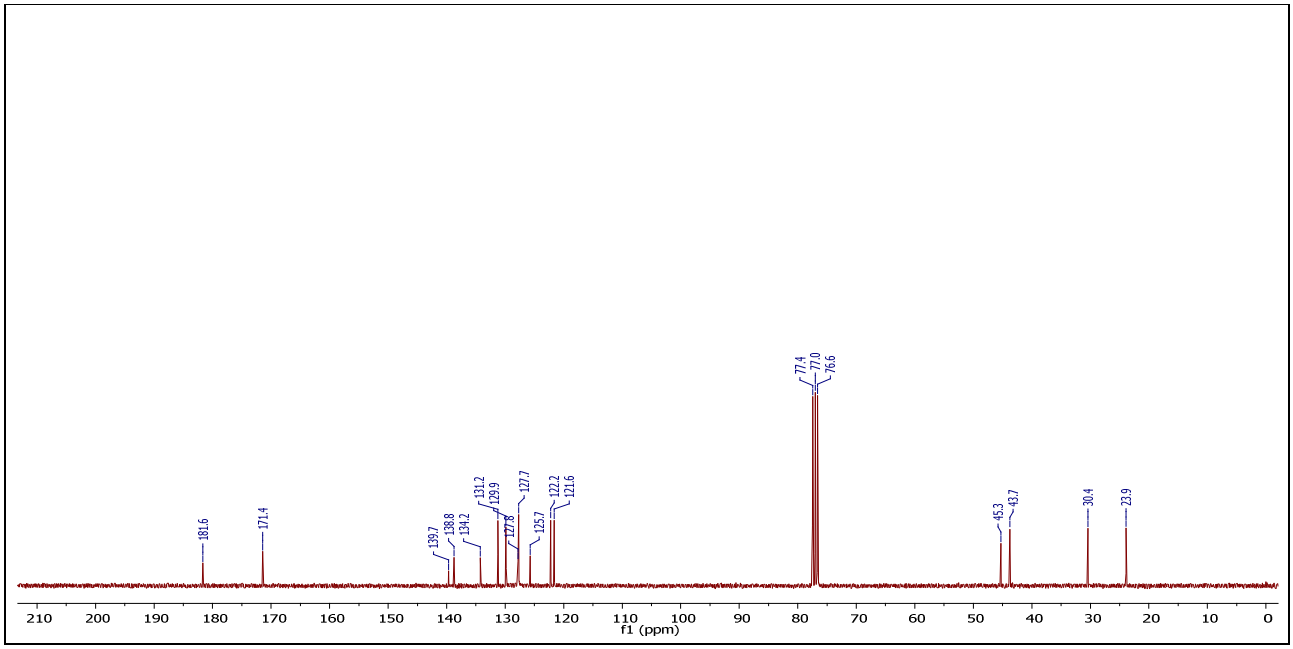




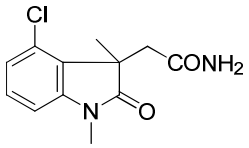
The ^{1}H NMR and ^{13}C NMR of 11

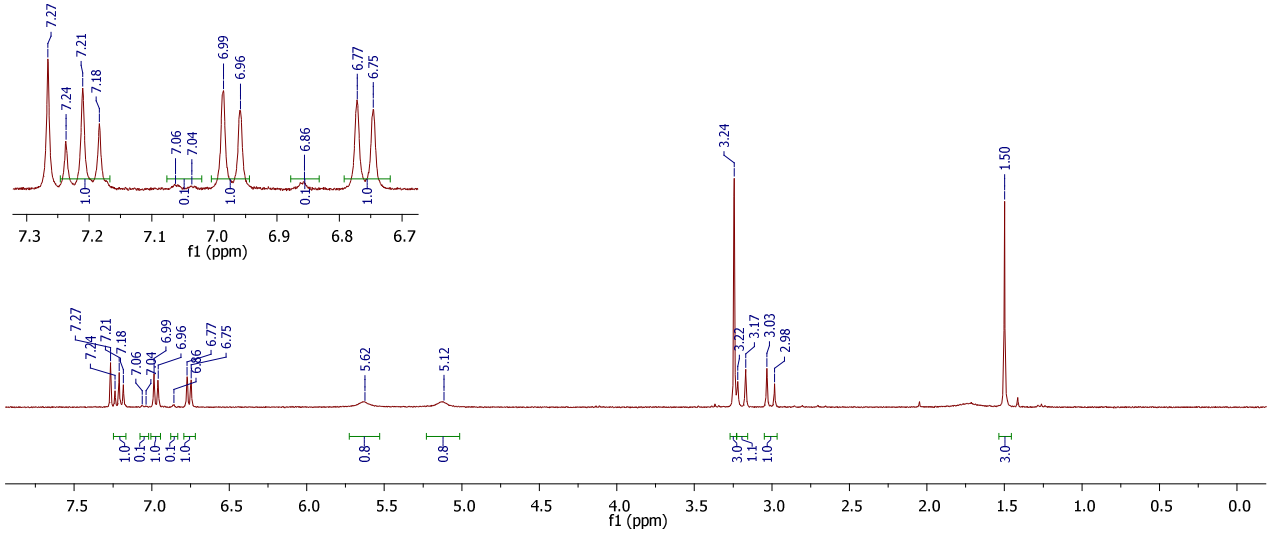


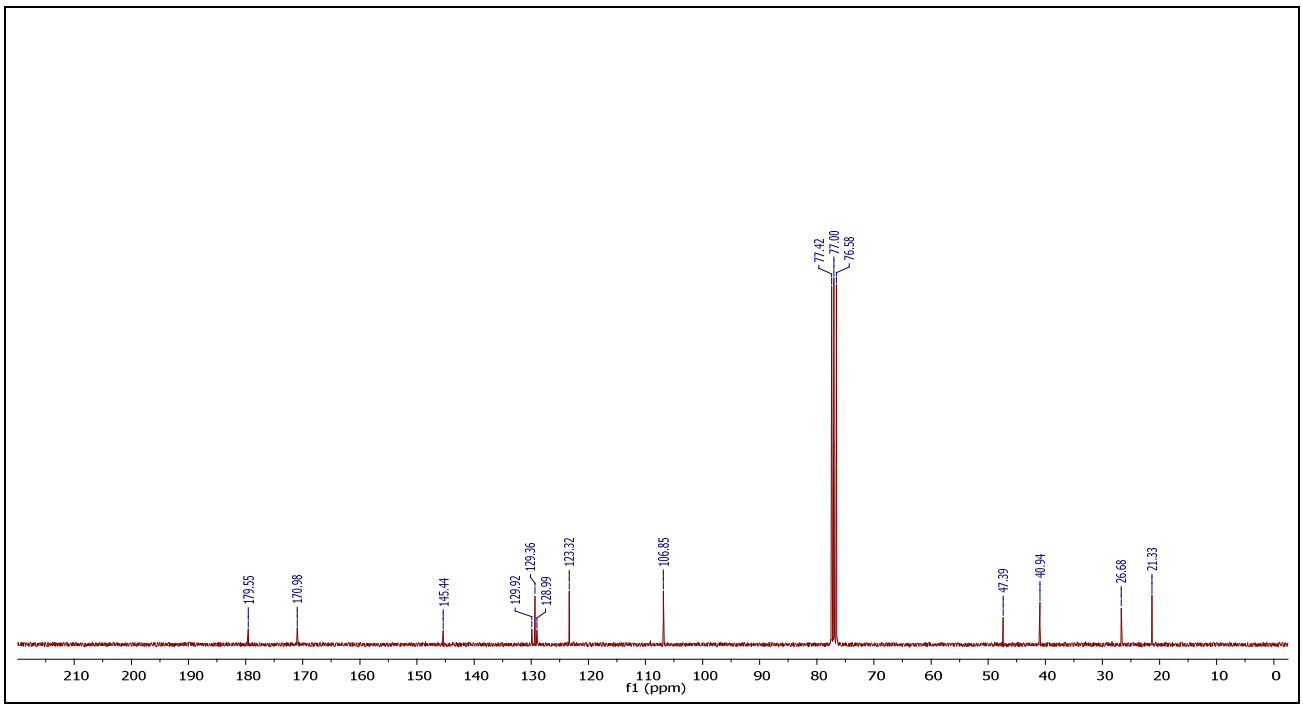




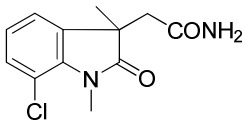
The ^{1}H NMR and ^{13}C NMR of 12+12'

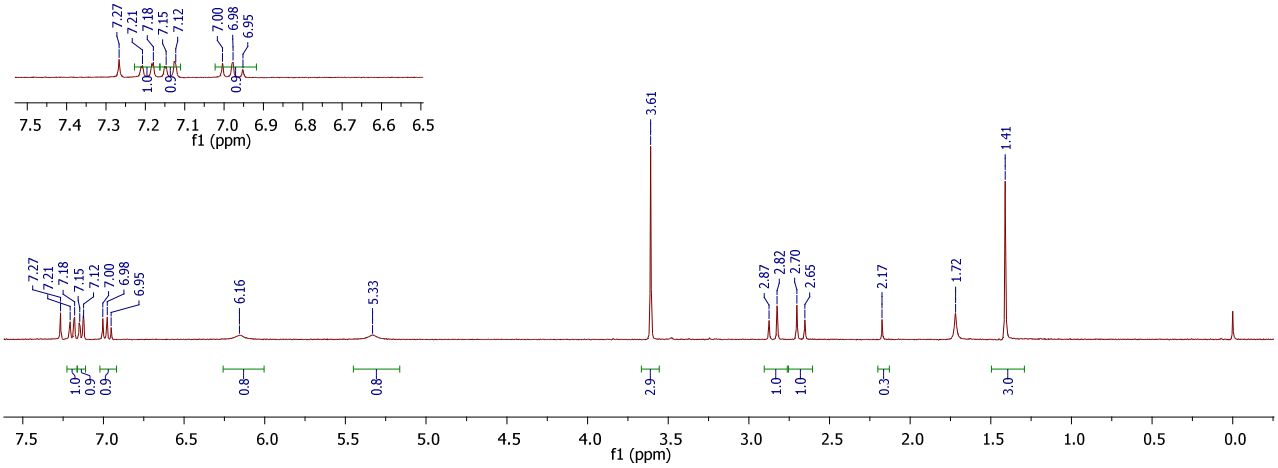


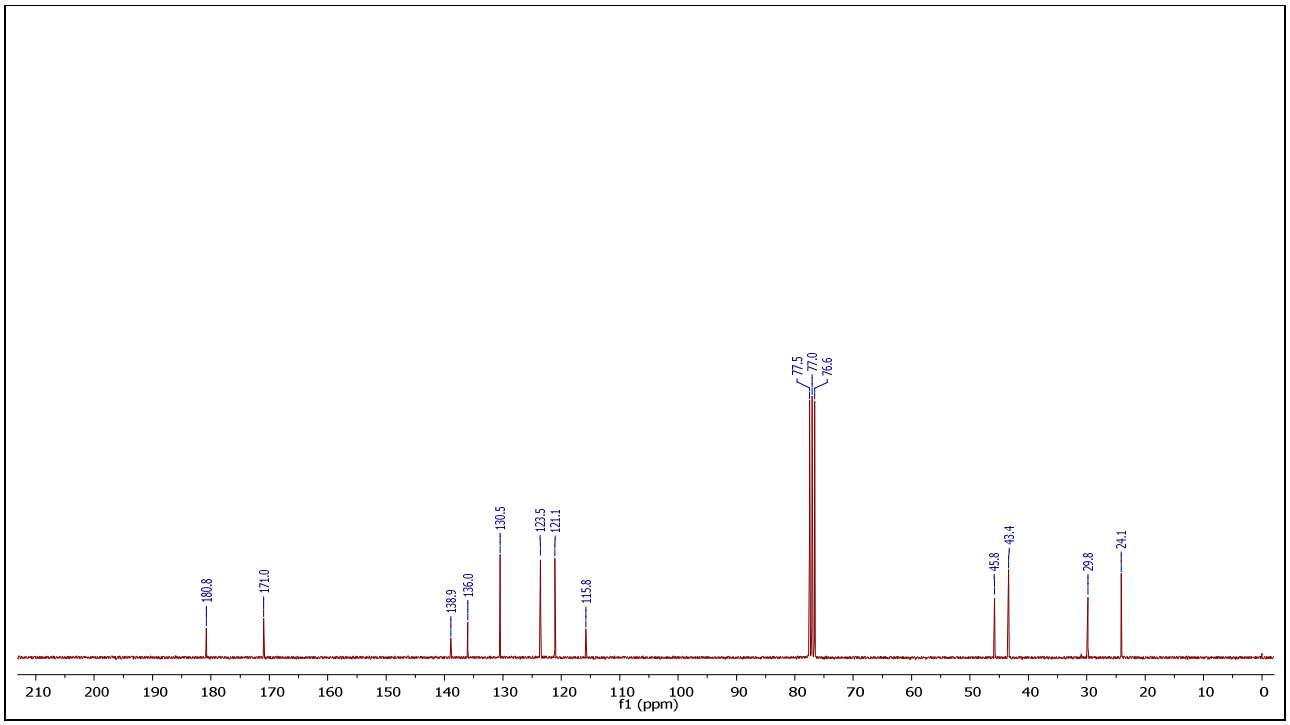




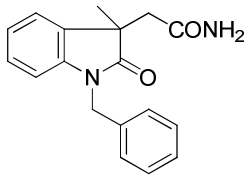
The ^{1}H NMR and ^{13}C NMR of 13

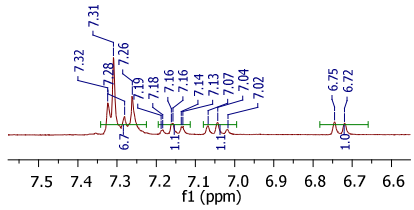


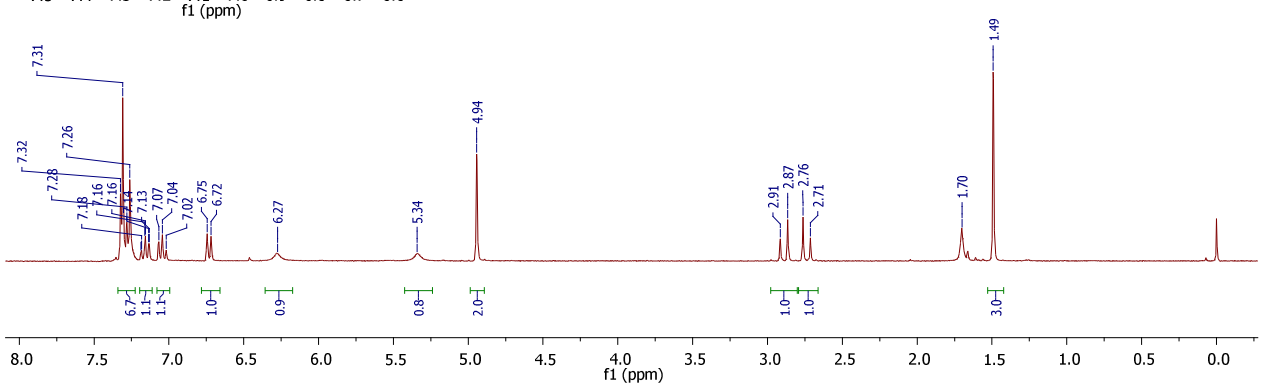


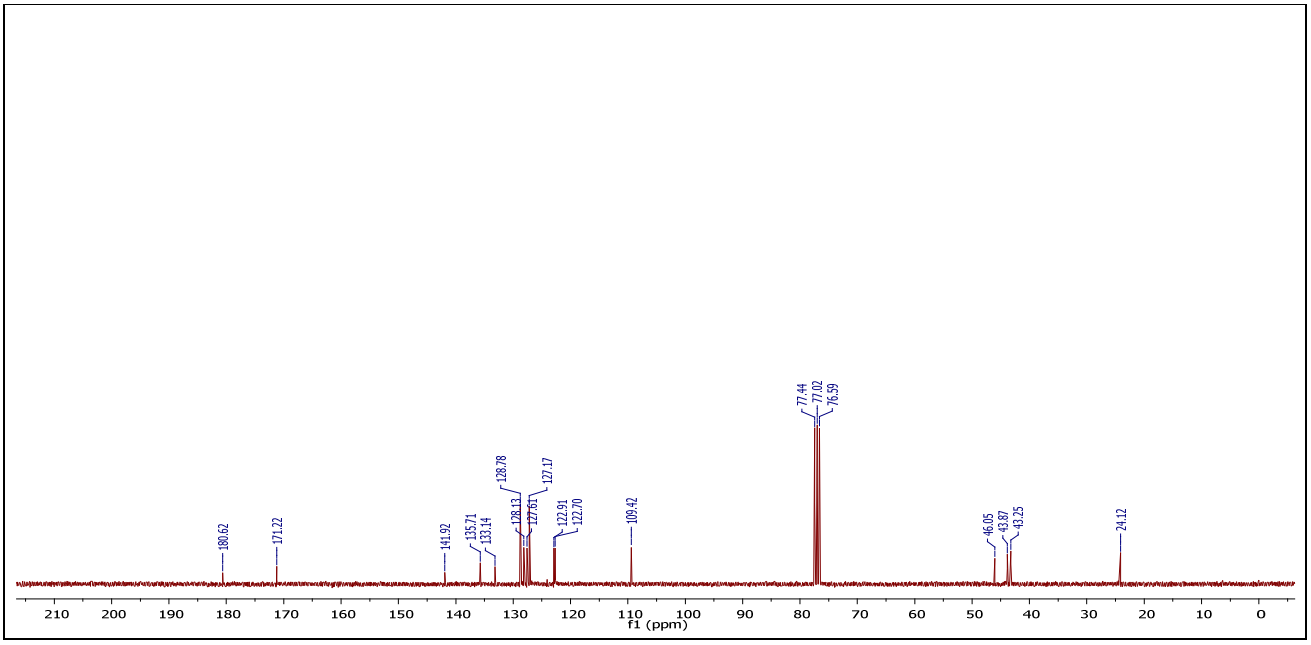


The ^{1}H NMR and ^{13}C NMR of 14

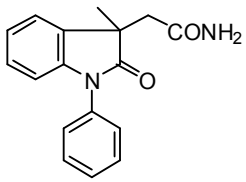


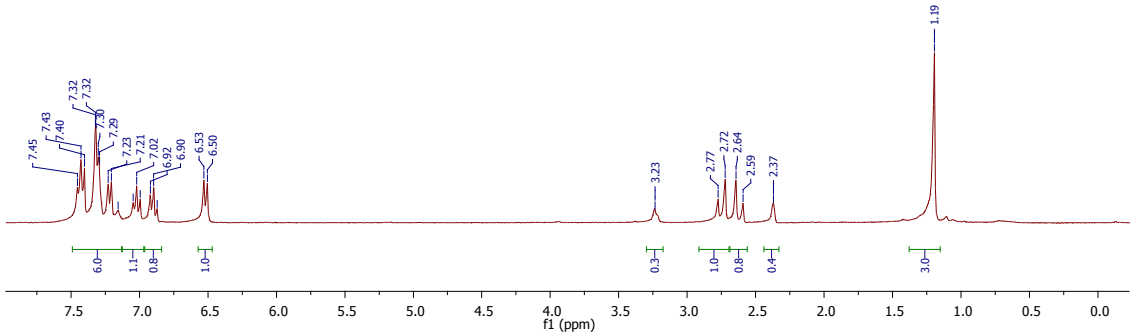


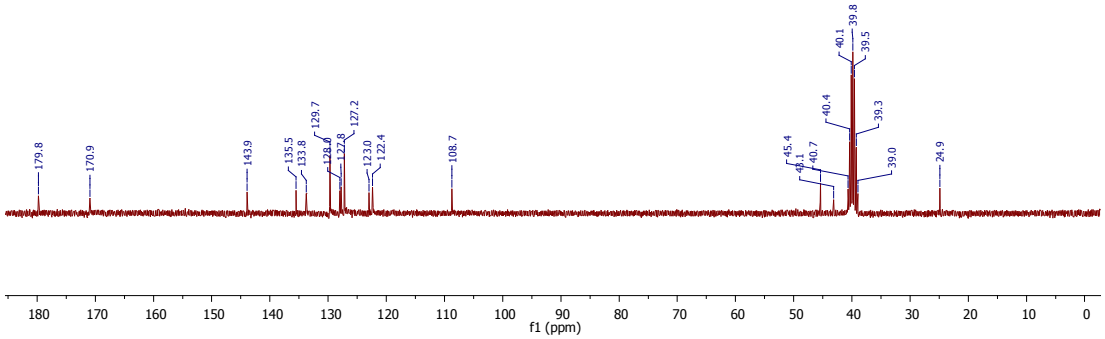




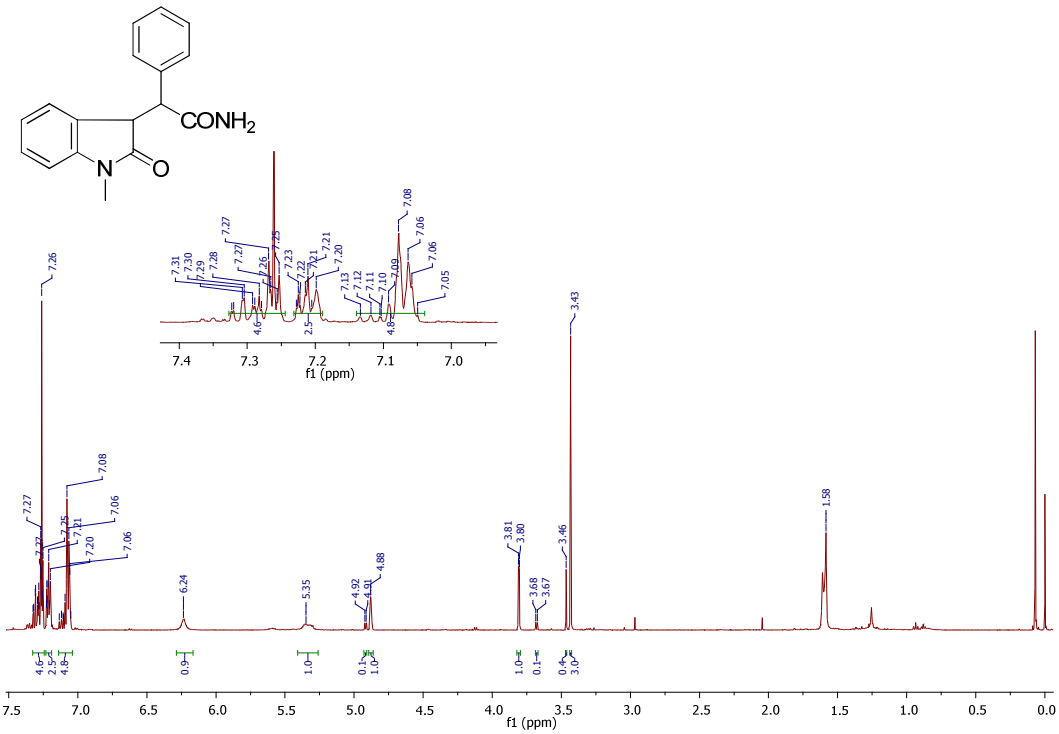
The ^{1}H NMR and ^{13}C NMR (in DMSO) of 15

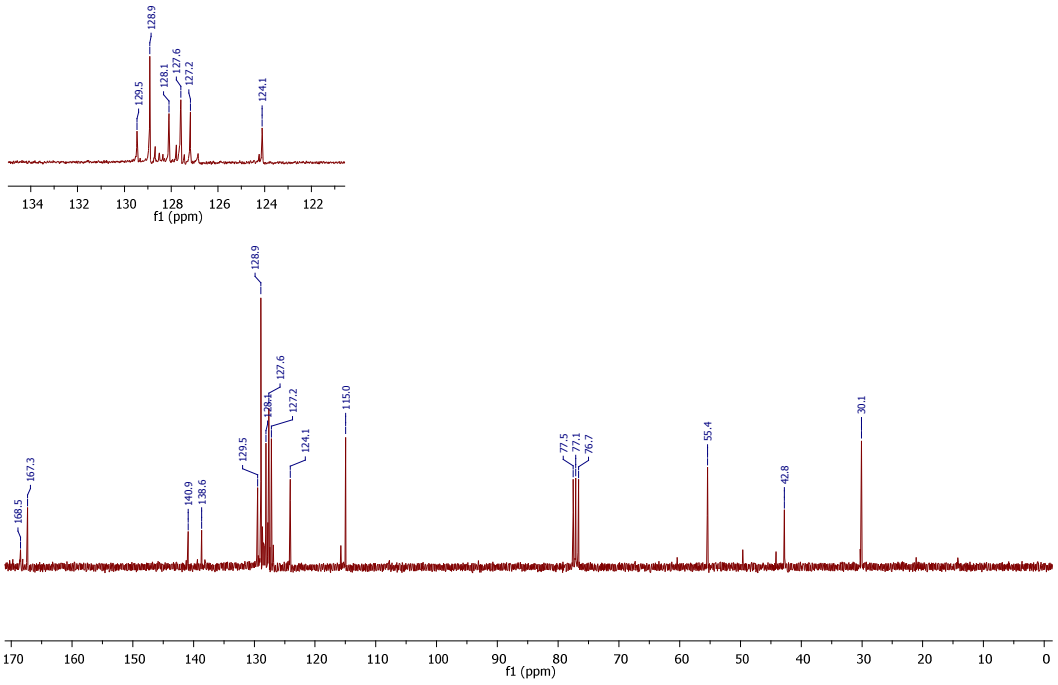




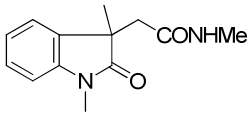


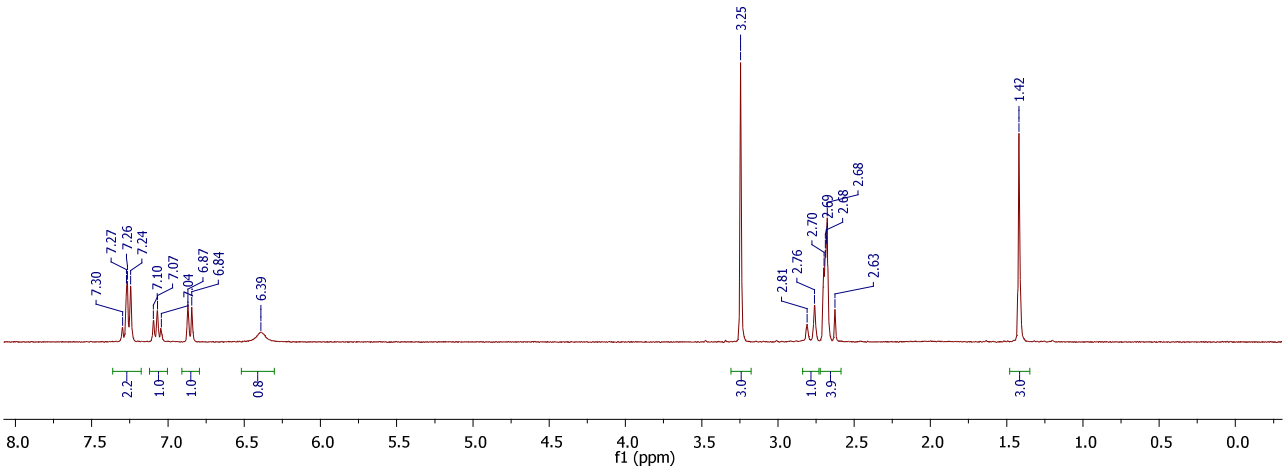
The ^{1}H NMR and ^{13}C NMR of 16

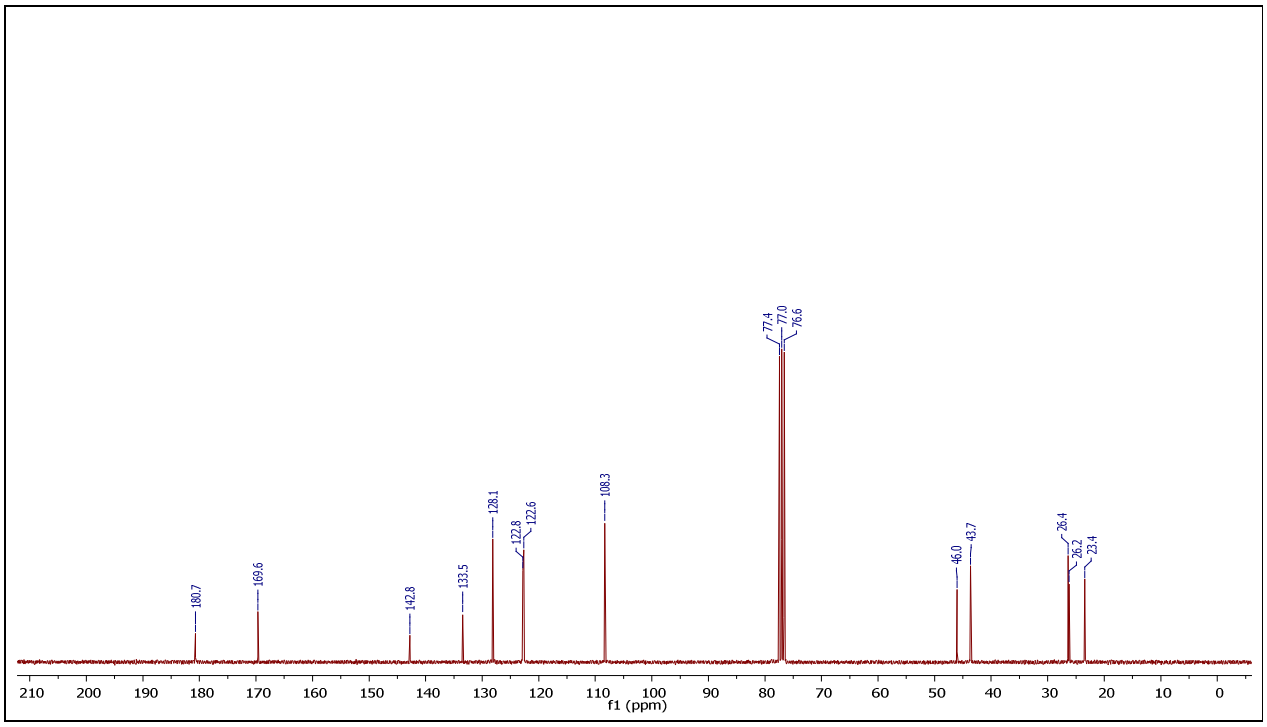




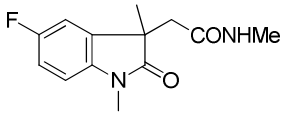
The ^{1}H NMR and ^{13}C NMR of 3

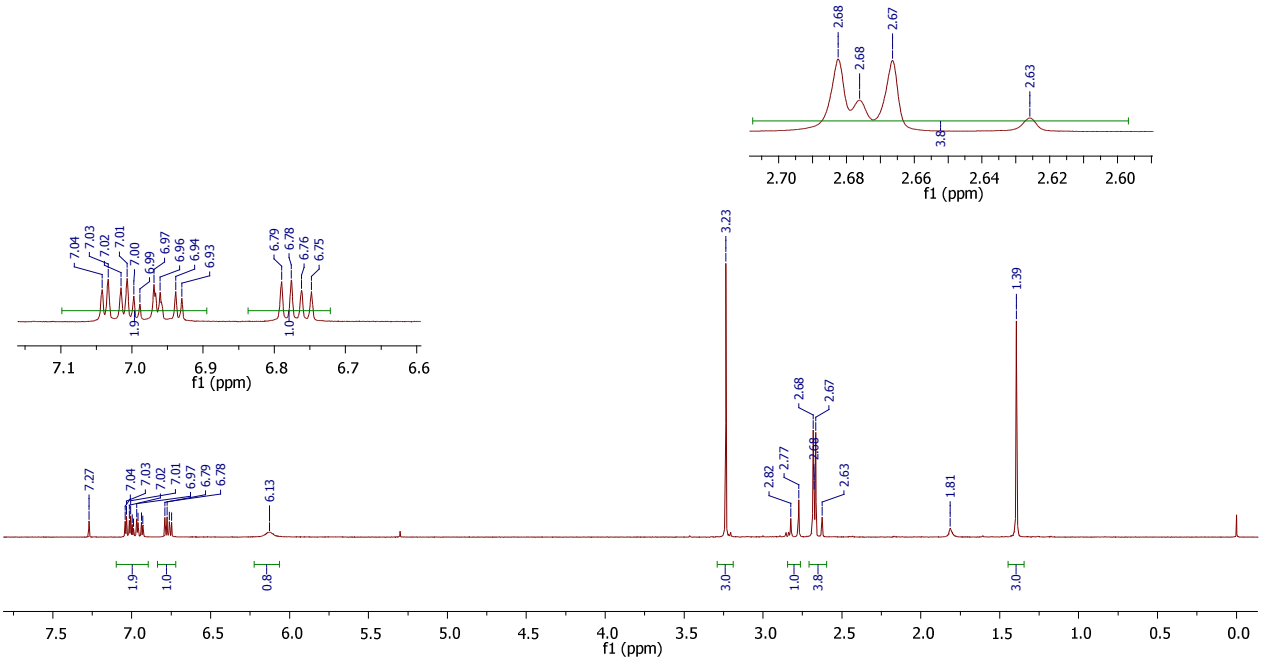


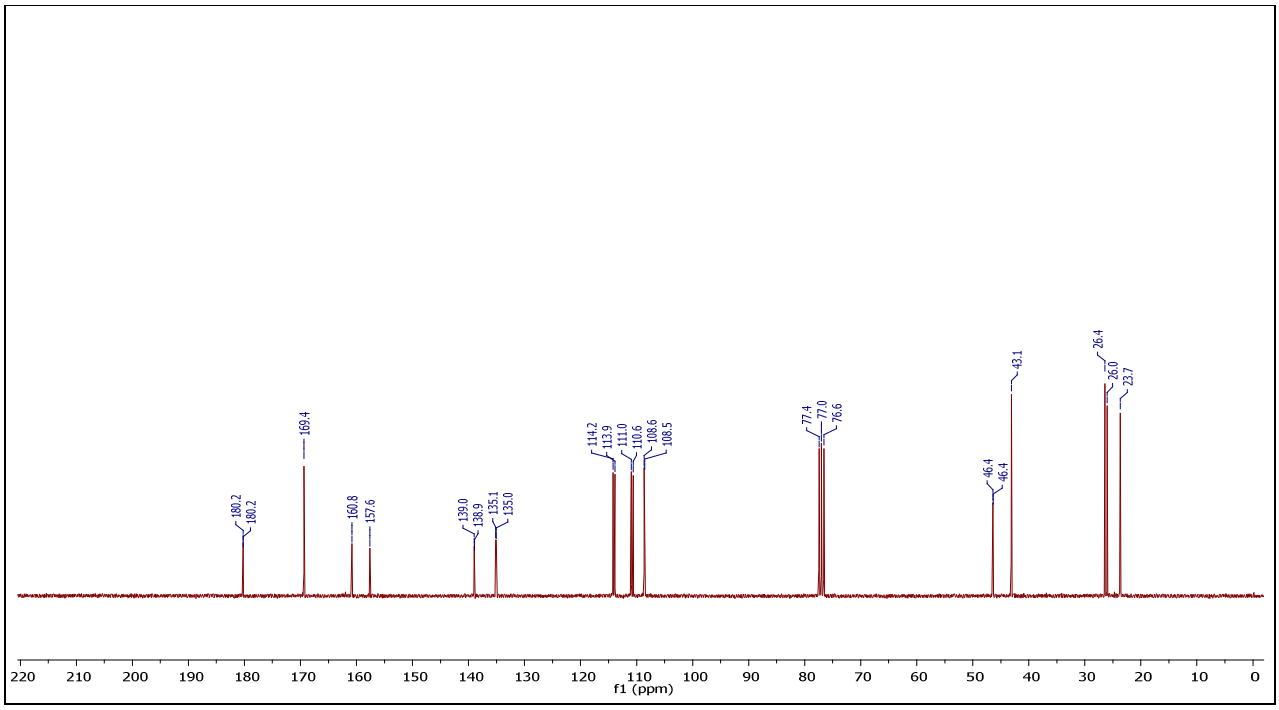




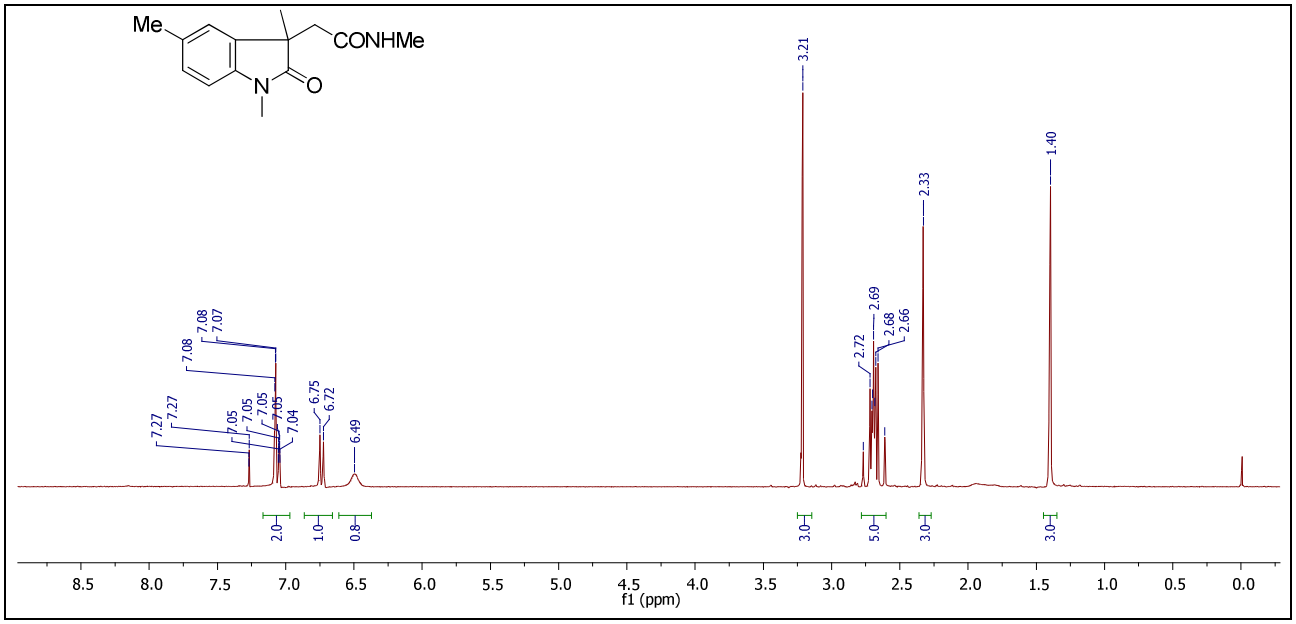
The ^{1}H NMR and ^{13}C NMR of 18

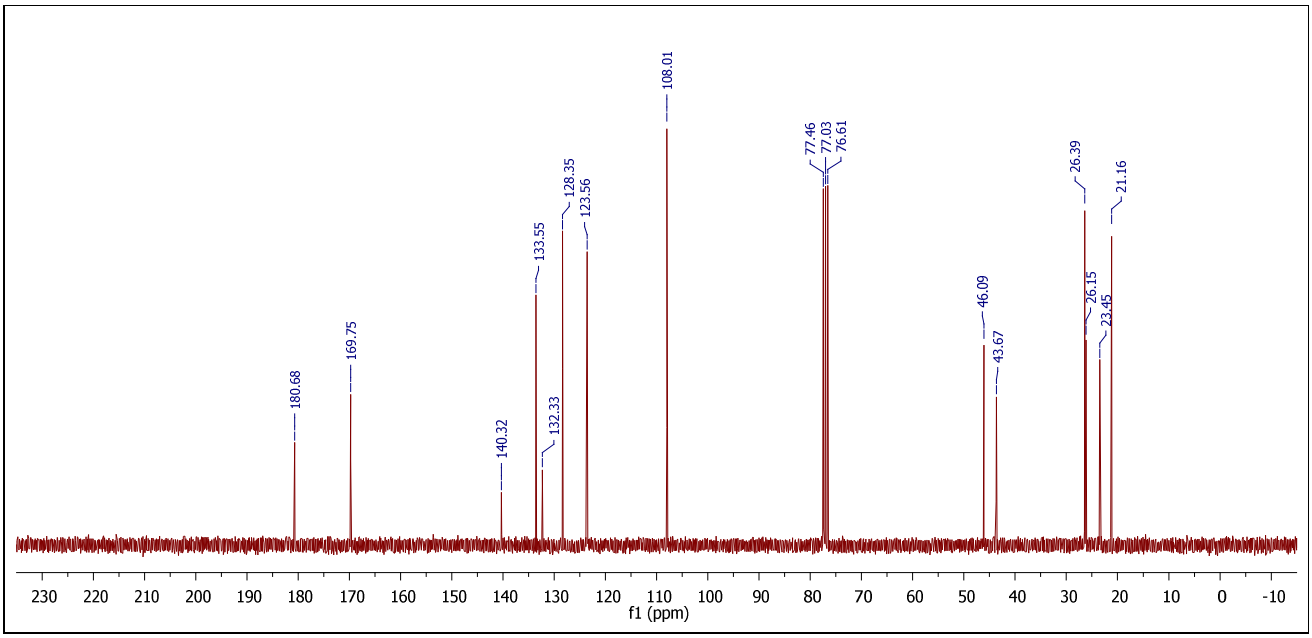

F
CONHMe
N
O


2.68
2.68
2.67
2.63
3.8
2.70
2.68
2.66
2.64
2.62
2.60
f1 (ppm)
7.04
7.03
7.02
7.01
7.00
6.99
6.97
6.96
6.94
6.93
6.79
6.78
6.76
6.75
1.9
1.0
7.1
7.0
6.9
6.8
6.7
6.6
f1 (ppm)
3.23
1.39
2.68
2.67
7.27
7.04
7.03
7.02
7.01
6.97
6.79
6.78
6.13
2.82
2.77
2.63
1.81
1.9
1.0
0.8
3.0
1.0
3.8
3.0
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)

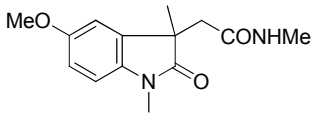

180.2
180.2
169.4
160.8
157.6
139.0
138.9
135.1
135.0
114.2
113.9
111.0
110.6
108.6
108.5
77.4
77.0
76.6
46.4
46.4
43.1
26.4
26.0
23.7
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)

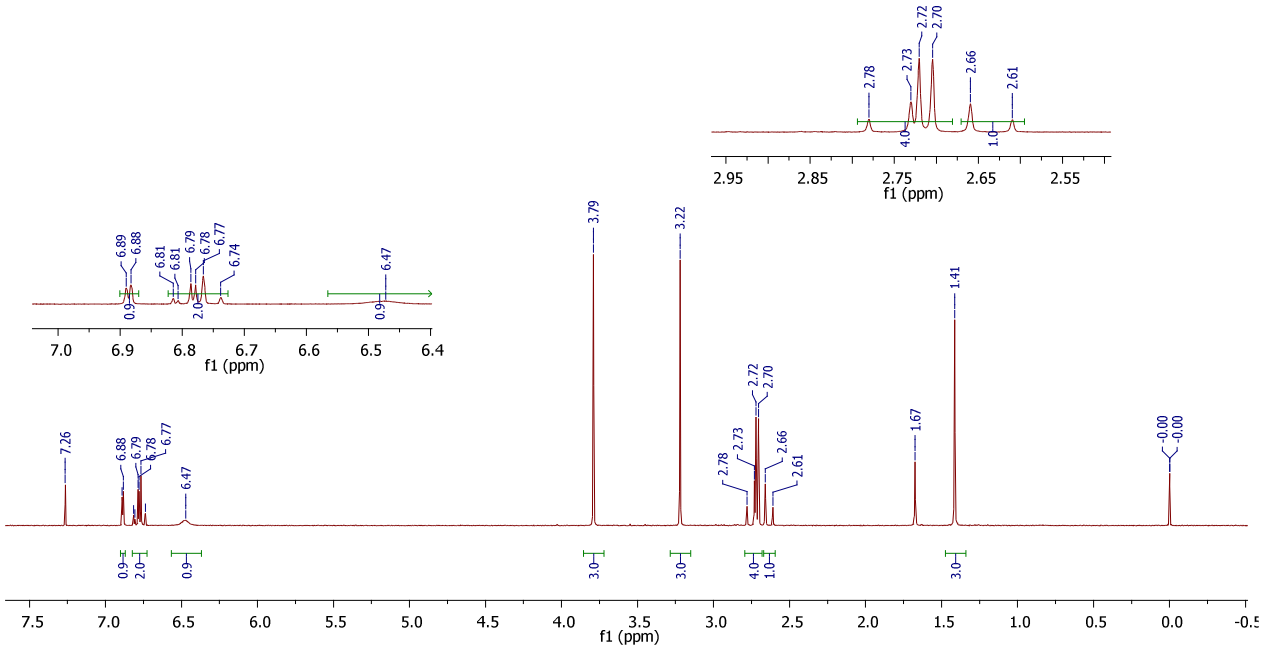
The ^{1}H NMR and ^{13}C NMR of 19

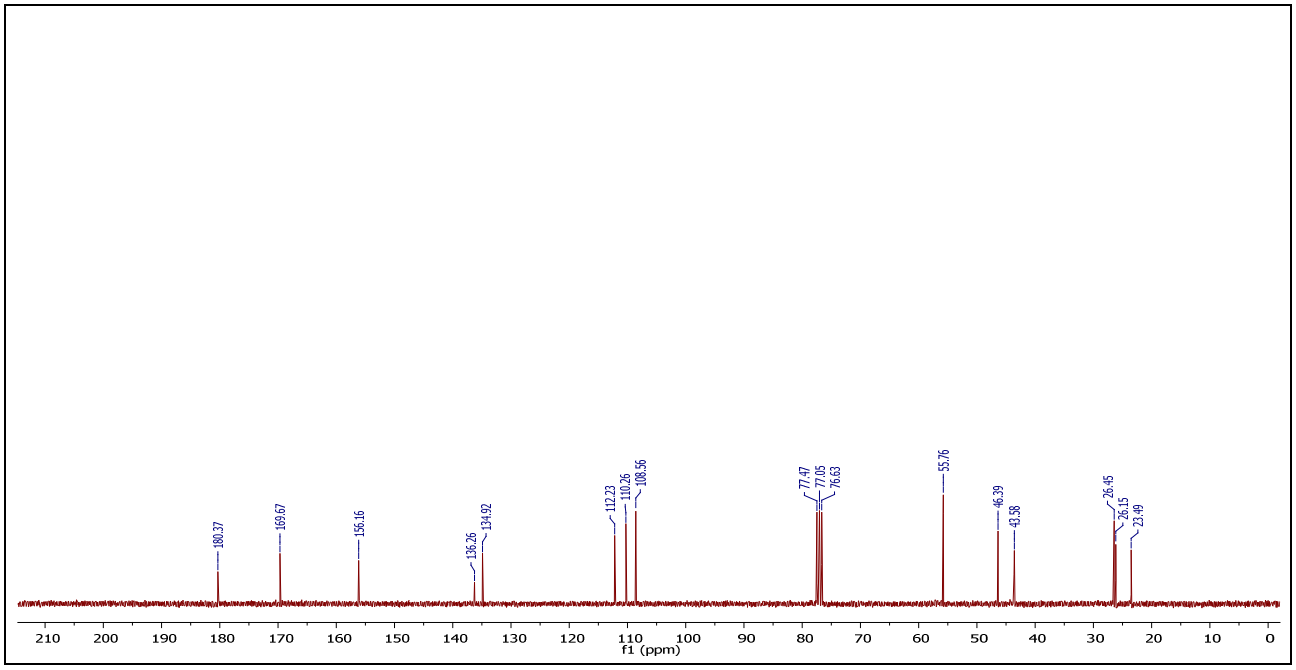




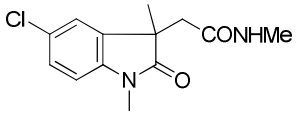
The ^{1}H NMR and ^{13}C NMR of 20

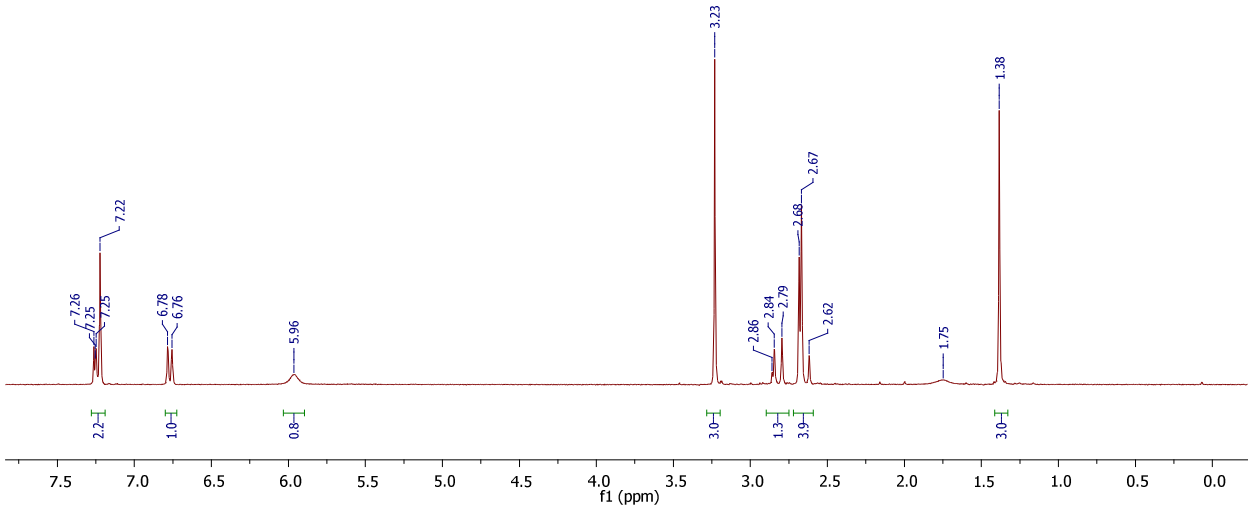


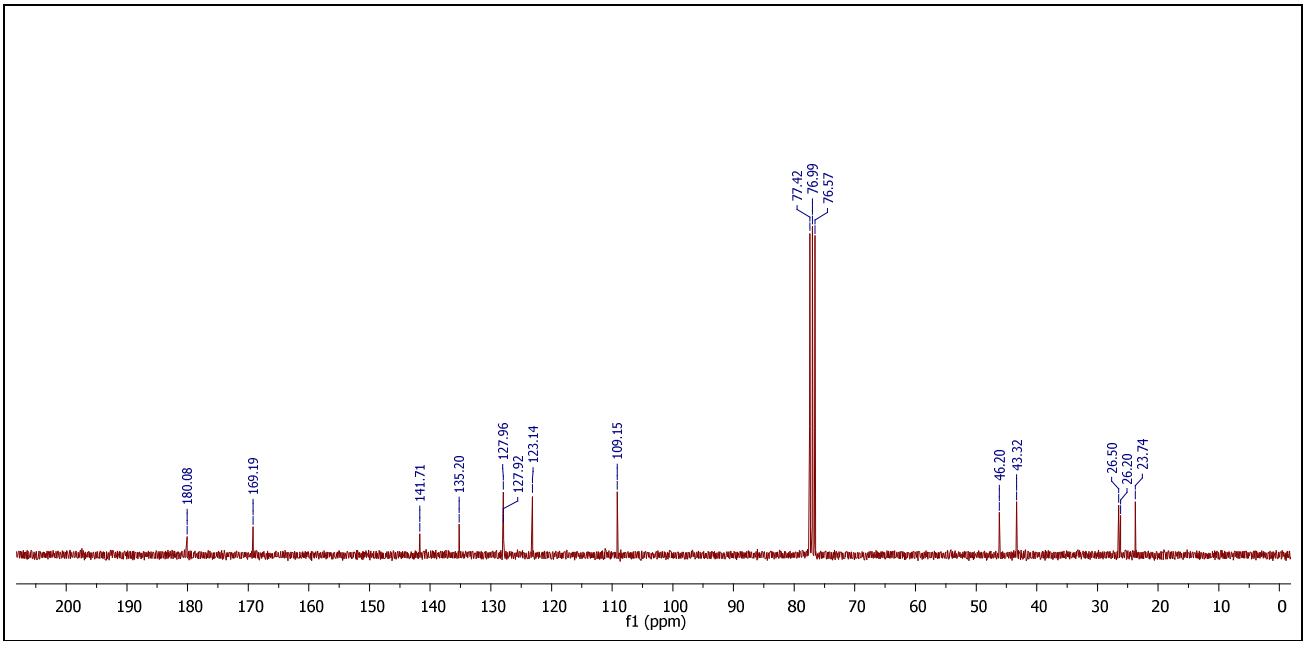




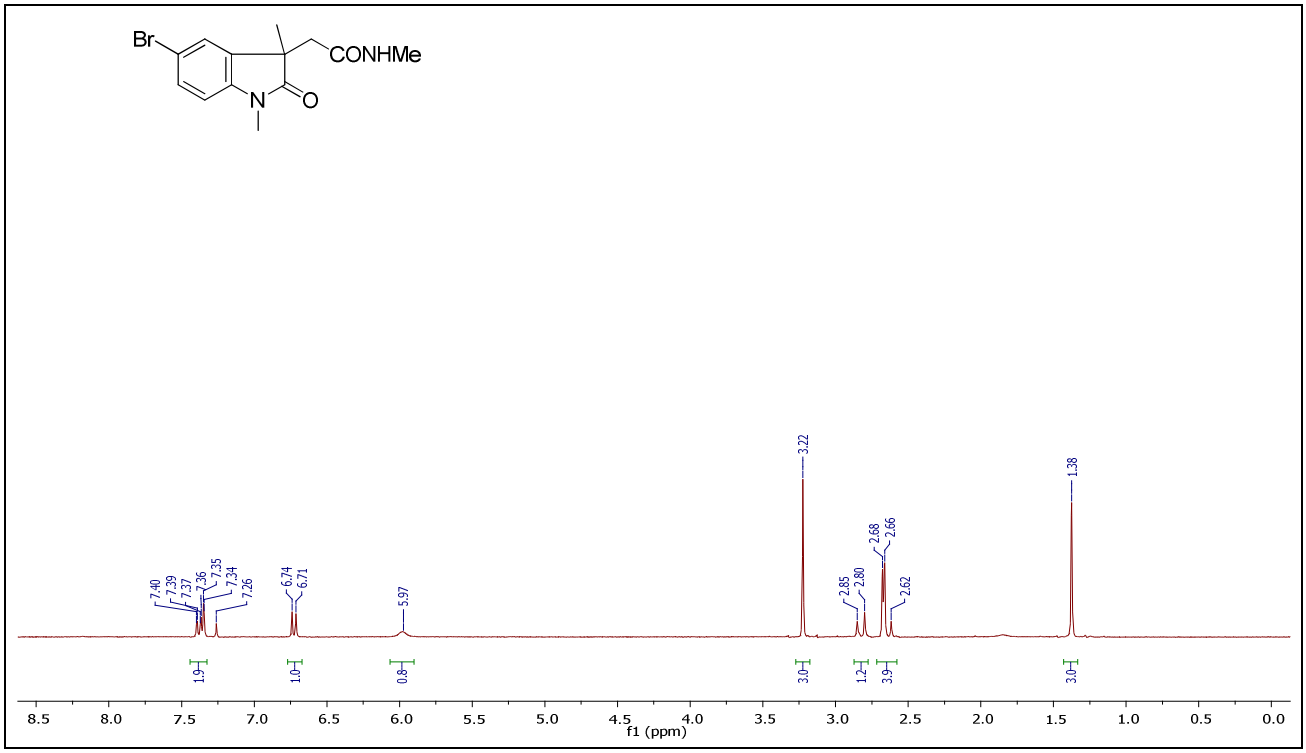
The ^{1}H NMR and ^{13}C NMR of 21

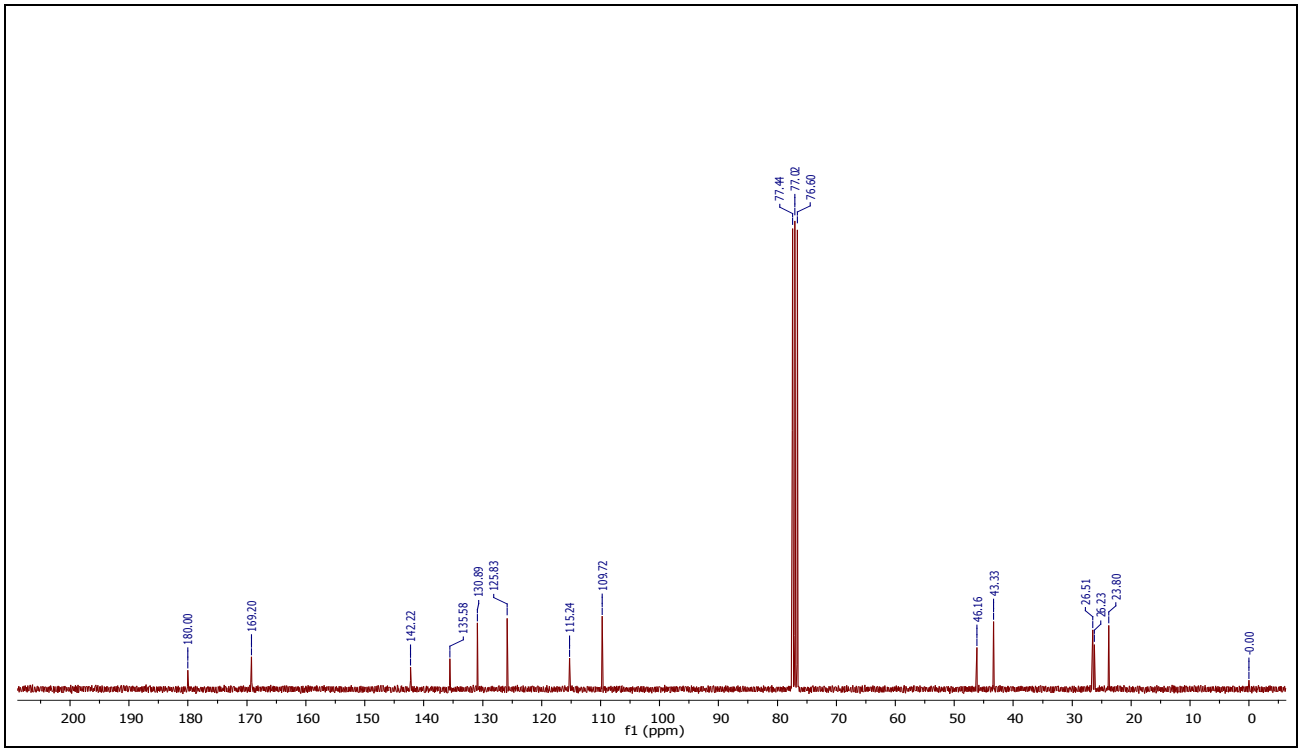




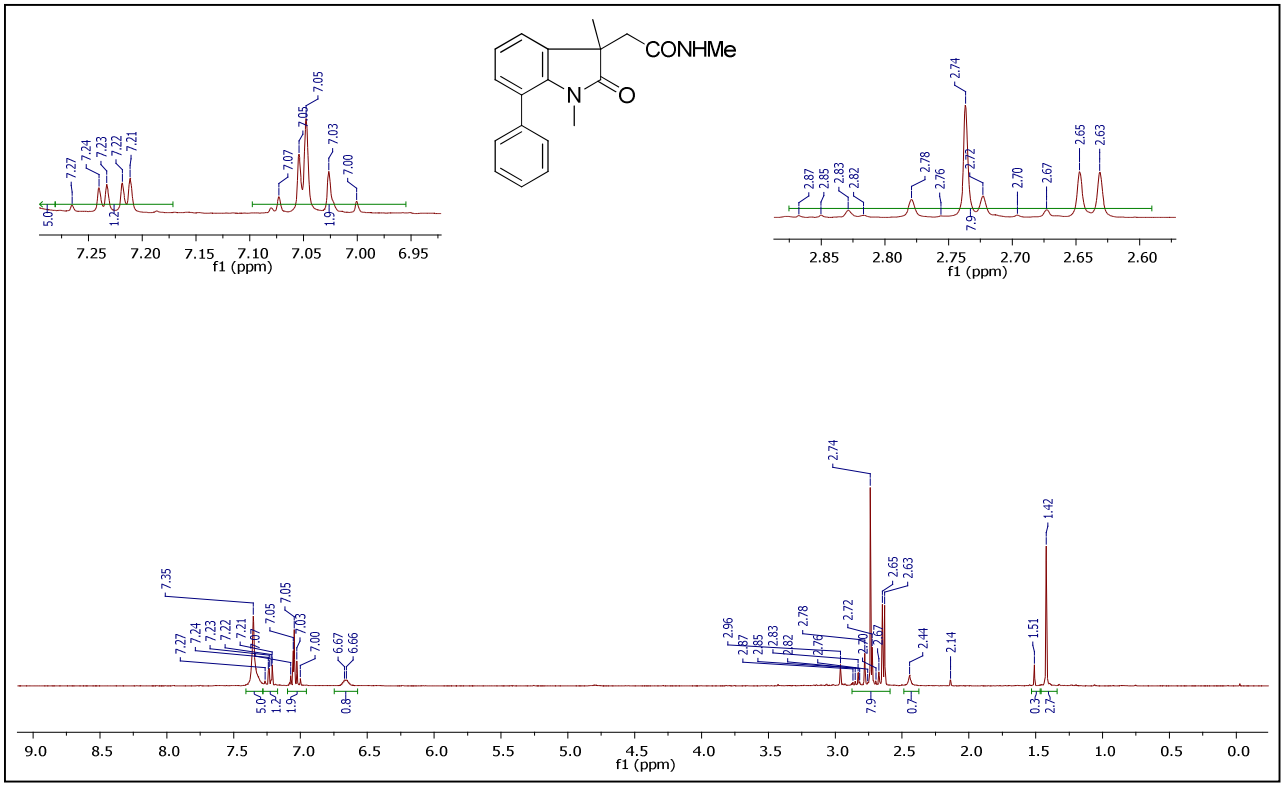


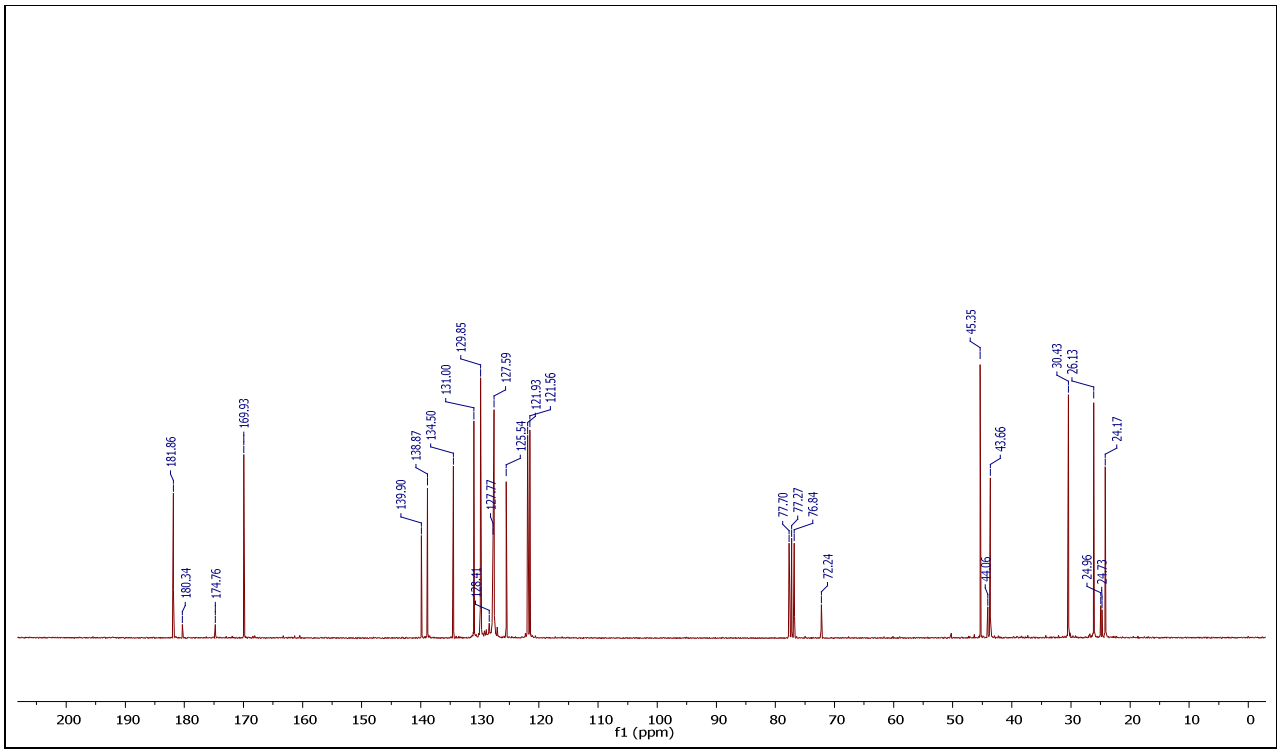
The ^{1}H NMR and ^{13}C NMR of 22



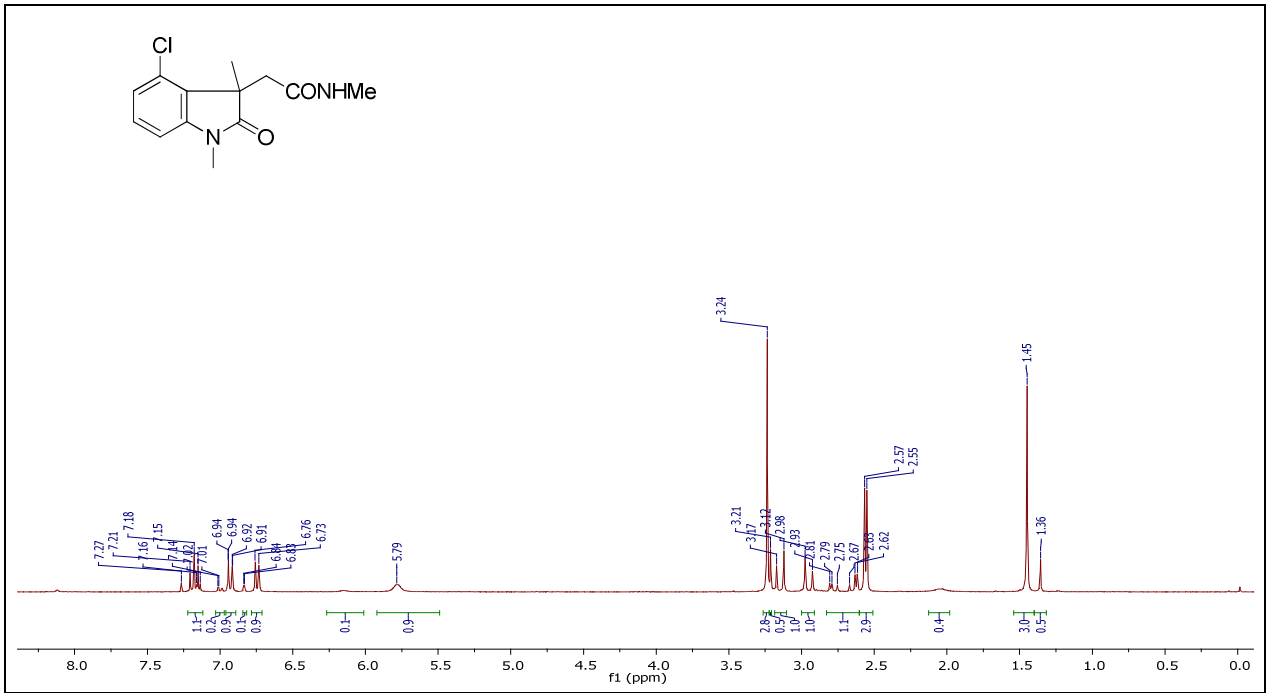


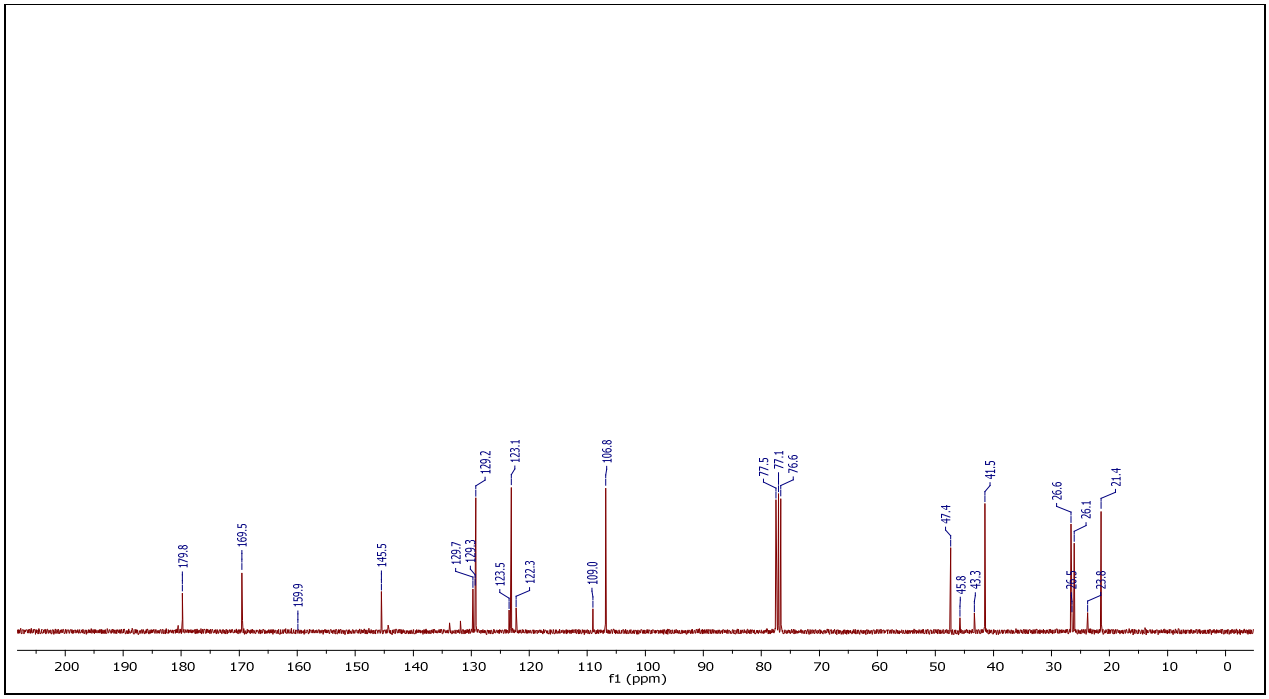
The ^{1}H NMR and ^{13}C NMR of 23



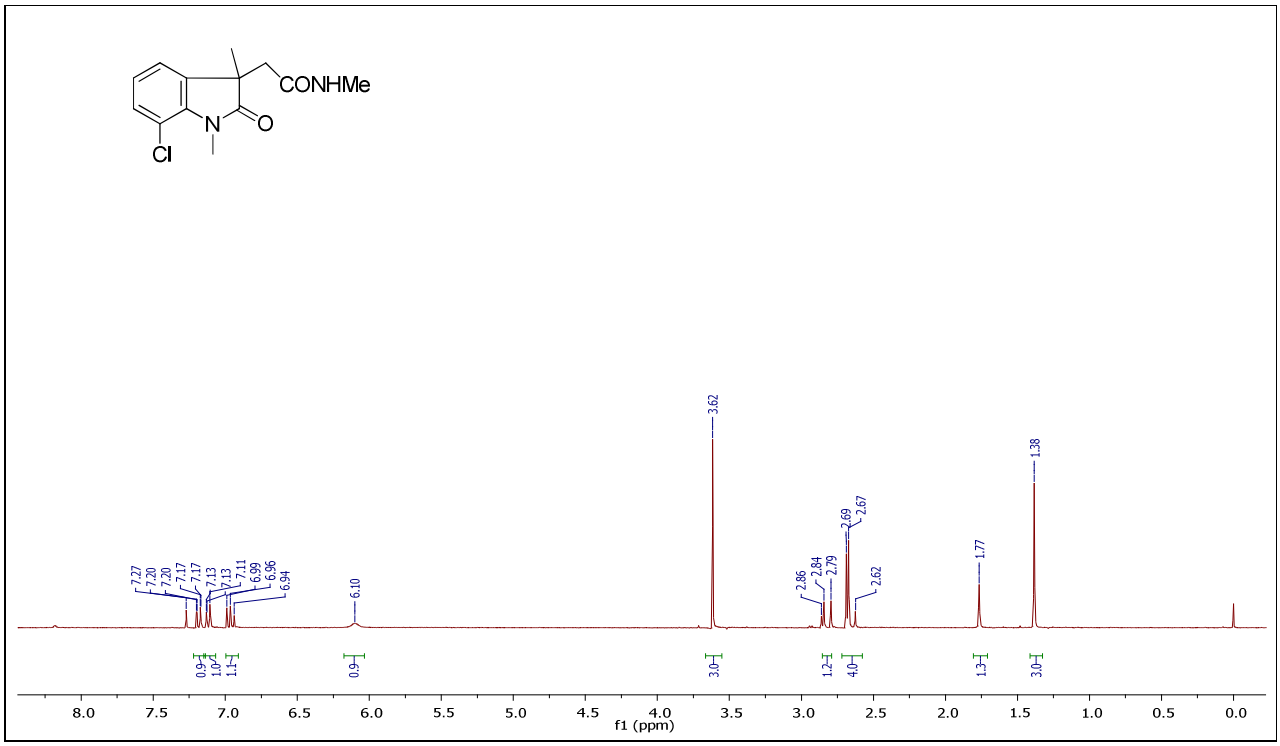


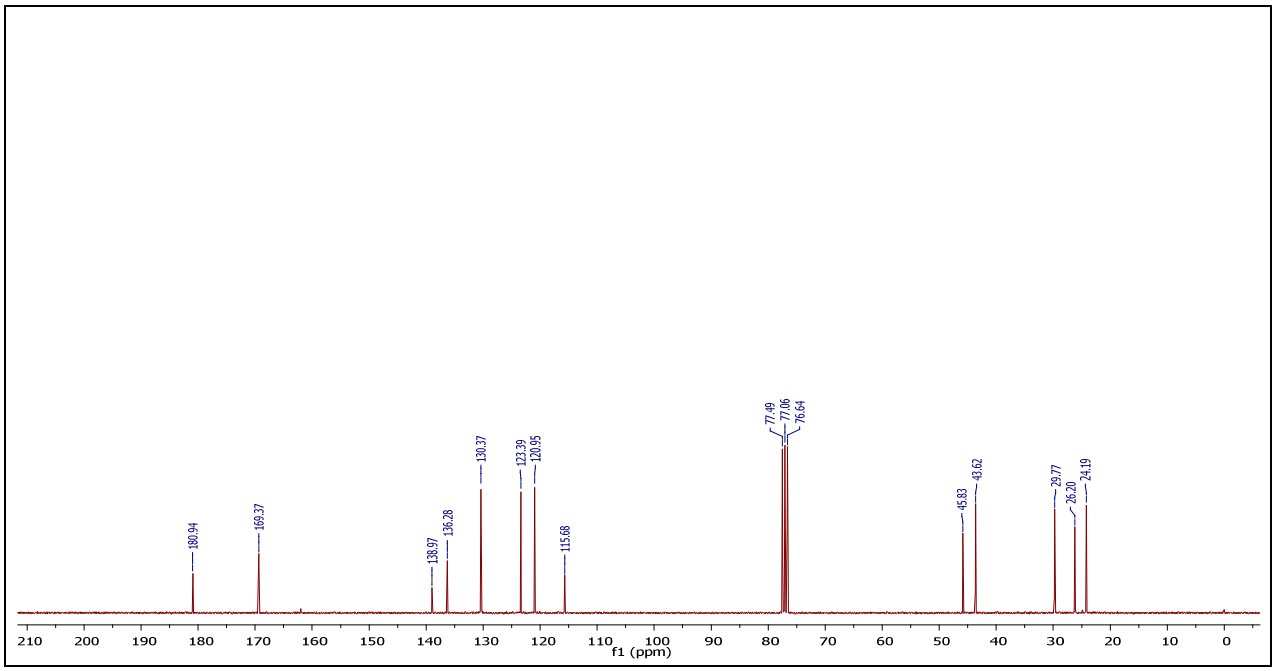
The ^{1}H NMR and ^{13}C NMR of 24



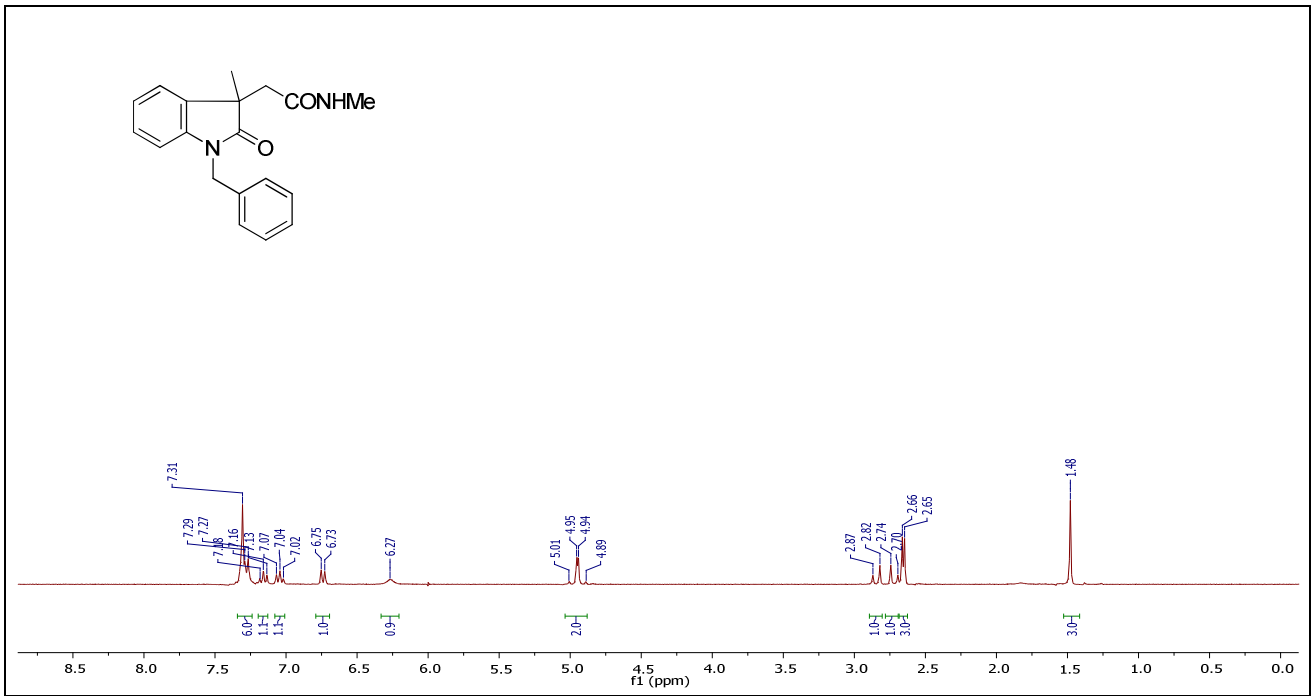


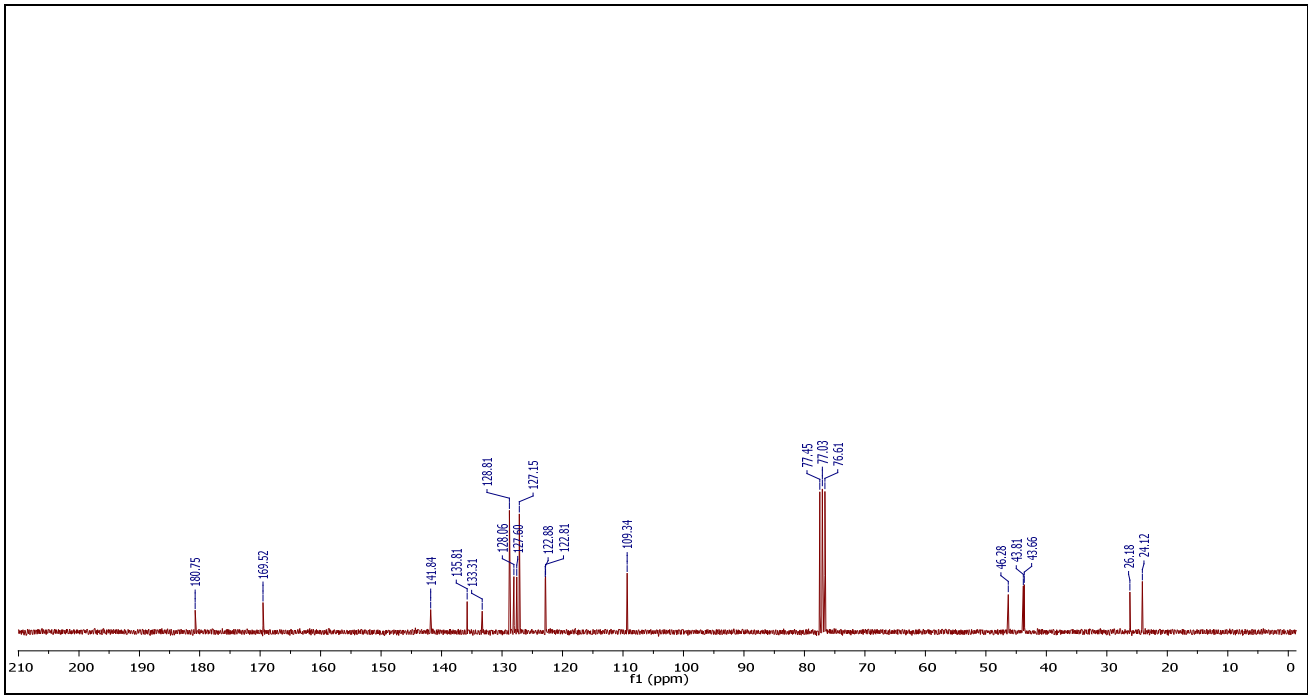
The ^{1}H NMR and ^{13}C NMR of 25


CONHMe
N
O
Cl
7.27
7.20
7.20
7.17
7.17
7.13
7.13
7.11
6.99
6.96
6.94
6.10
3.62
2.86
2.84
2.79
2.69
2.67
2.62
1.77
1.38
0.9
1.0
1.1
0.9
3.0
1.2
4.0
1.3
3.0
f1 (ppm)

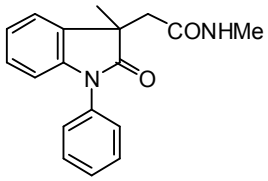

180.94
169.37
138.97
136.28
130.37
123.39
120.95
115.68
77.49
77.06
76.64
45.83
43.62
29.77
26.20
24.19
f1 (ppm)

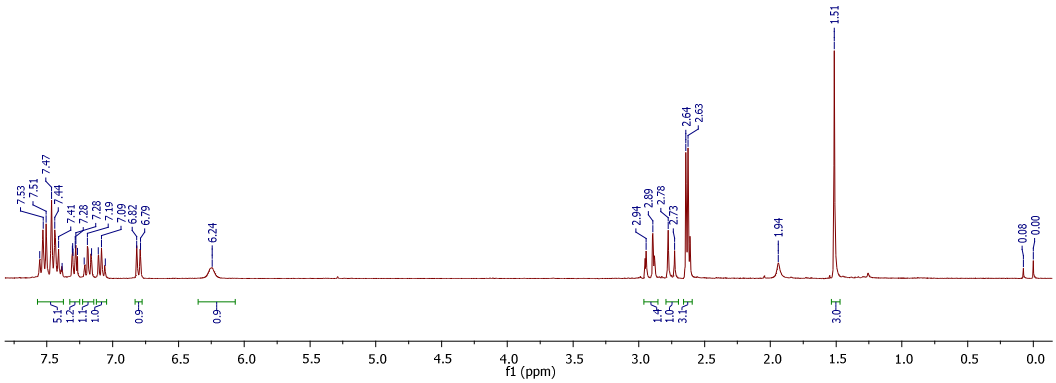
The ^{1}H NMR and ^{13}C NMR of 26

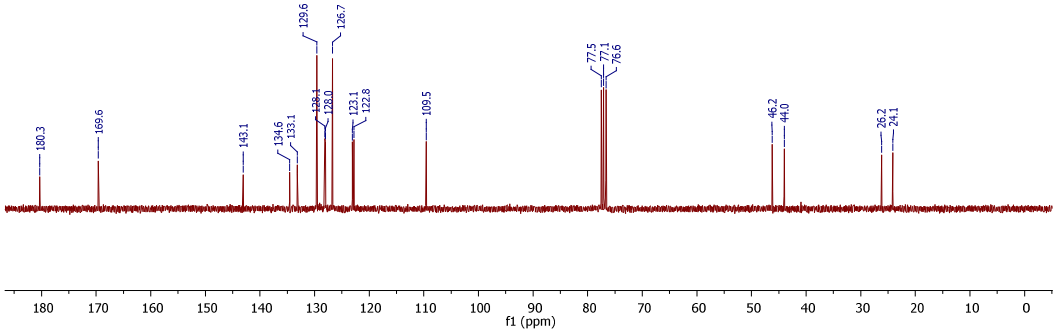




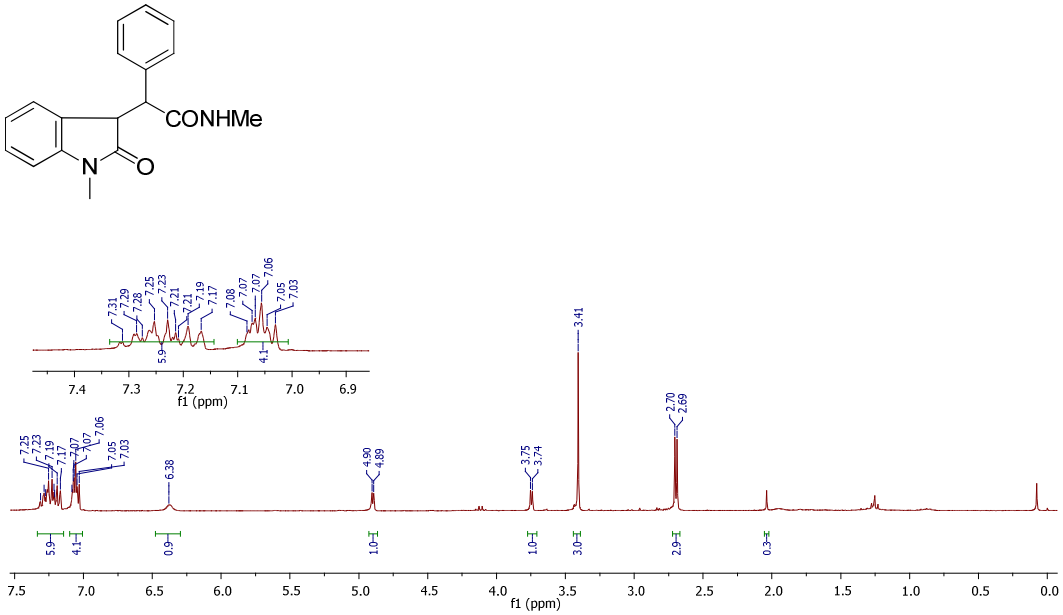
The ^{1}H NMR and ^{13}C NMR of 27

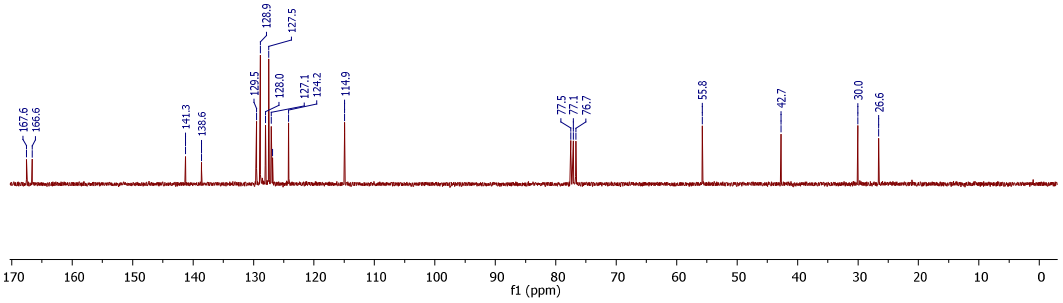




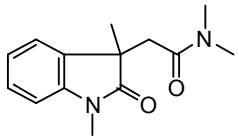


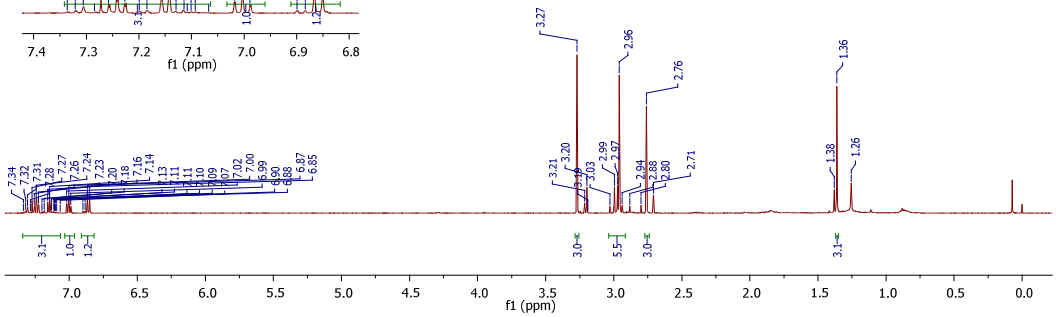
The ^{1}H NMR and ^{13}C NMR of 28

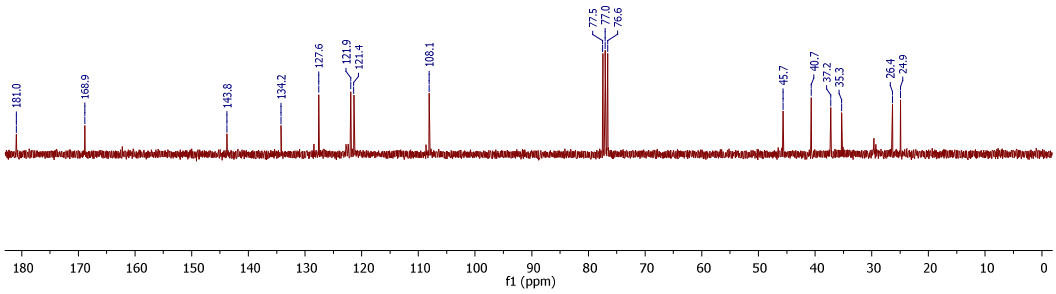




The ^{1}H NMR and ^{13}C NMR of 30







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