One-Pot Facile Synthesis of Substituted Isoindolinones via an Ugi Four-Component Condensation/Diels-Alder Cycloaddition/ Deselenization-Aromatization Sequence

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Supporting Information

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1. Experiment procedures

General Methods.

Melting points were uncorrected. CH_2Cl_2 was distilled from CaH_2 immediately prior to use. Petroleum ether refers to the fraction with boiling point in the range 60–90 °C. All ^1H NMR (400 MHz) and ^{13}C NMR (100 MHz) spectra were measured in $CDCl_3$ with TMS as internal standard. Chemical shifts are expressed in ppm and J values are given in Hz.

General Procedure.

Synthesis of 2-(Phenylselanyl)acrylic acids 7a.

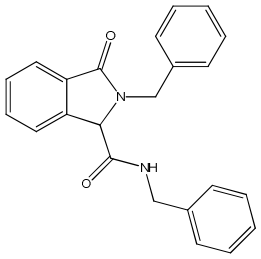
Phenylselenyl bromide (5.0 mmol) was dissolved in CH_2Cl_2 (20 mL), methyl acrylate (10 mmol) was added and the reaction mixture was stirred at 25 °C for 0.5 h. Then triethylamine (10 mmol) was added and the reaction mixture was stirred at 25 °C for another 1 h. The precipitate was filtered and the filtrate was concentrated. The residue was purified by flash chromatography (petroleum ether/ethyl acetate 9:1 v/v) to give methyl 2-(phenylselanyl)acrylate in 87% yield (4.35 mmol).

KOH-water solution (0.5M, 13 mL) was added to Methyl 2-(phenylselanyl)acrylate (4.35 mmol), this mixture was stirred overnight at 25 °C. Ether (20 mL) was added to the mixture and the layers were separated, the aqueous layer was extracted with ether (2 × 15 mL), then 3N HCl was added to the aqueous layer to acidfy it to PH=2~3. Ether (20 mL) was added to the aqueous layer and the layers were separated, the aqueous layer was extracted with ether (2 × 15 mL), The combined organic layers were washed with brine, dried over Na_2SO_4, and evaporated to afford 2-(phenylselanyl)acrylic acid in 92% yield (4.0 mmol).

General Procedure for Synthesis of Isoindolinones 10.

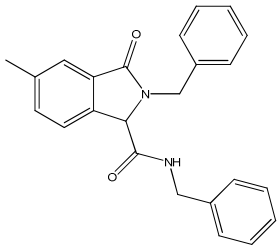
A 2-furaldehyde **5** (1.0 mmol), a primary amine **6** (1.2 mmol), a 2-(phenylselanyl)acrylic acid **7** (1.0 mmol) and an isocyanide **8** (1.0 mmol) were combined in methanol (4 mL), the reaction mixture was stirred at 25 °C for 16 h(For **10e**, **10f**, **10g**, **10h**, **10n** and **10o**, the reaction mixture was stirred at 25 °C for 12 h then reflux for 8 h. For **10b**, **10i**, **10j** and **10m**, the reaction mixture was stirred at 25 °C for 12 h then reflux for 24 h.). Then methanol was evaporated and anhydrous CH_2Cl_2 (5mL) and BF_3-OEt_2 (2.0 mmol) was added, stirring was continued at 25 °C for an additional 2 h before the reaction was quenched by adding sat. aq $NaHCO_3$ (20 mL). The layers were separated and the aqueous layer was extracted with CH_2Cl_2 (2 × 15 mL). The combined organic layers were washed with brine, dried over Na_2SO_4, and evaporated. The residue was purified by flash chromatography (petroleum ether/ethyl acetate 2:1 v/v) to give the product **10**.

2. Characterization data:



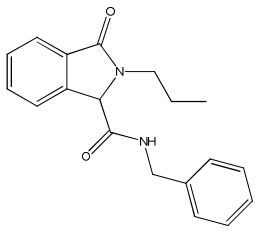
***N*,2-dibenzyl-3-oxoisoindoline-1-carboxamide (10a)**

pale solid, mp 169-171 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 (1H, d, J = 7.2 Hz), 7.48-7.51 (1H, m), 7.40 (1H, br, 1H of NH), 7.15-7.34 (12H, m), 5.24 (1H, d, J = 14.8 Hz, 1H of CH_2), 4.88 (1H, s, 1H of CH), 4.44 (1H, dd, J_1 = 14.8 Hz, J_2 = 6.0 Hz, 1H of CH_2), 4.38 (1H, dd, J_1 = 14.8 Hz, J_2 = 6.0 Hz, 1H of CH_2), 4.17 (1H, d, J = 14.8 Hz, 1H of CH_2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.5, 167.5, 141.2, 138.0, 135.9, 132.2, 130.4, 128.9, 128.6, 128.4, 128.0, 127.7, 127.4, 123.7, 112.7, 64.1, 45.6, 43.4; MS (EI) *m/z* 356 (M^+); IR $_{Vmax}$ (cm^{-1}) 1695, 1664, 1541, 1397, 1241, 740，699; HRMS (EI): *m/z* calcd for $C_{23}H_{20}N_2O_2$ (M^+): 356.1525; Found: 356.1522.



***N*,2-dibenzyl-5-methyl-3-oxoisoindoline-1-carboxamide (10b)**

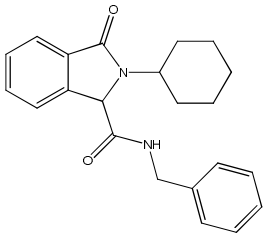
pale solid, mp 101-103 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.68 (1H, br, 1H of NH), 7.45 (1H, d, J = 7.6 Hz), 7.19-7.28 (11H, m), 6.89 (1H, s), 5.27 (1H, d, J = 15.2 Hz, 1H of CH_2), 4.84 (1H, s, 1H of CH), 4.46 (1H, dd, J_1 = 14.4 Hz, J_2 = 6.0 Hz, 1H of CH_2), 4.40 (1H, dd, J_1 = 15.2 Hz, J_2 = 6.4 Hz, 1H of CH_2), 4.16 (1H, d, J = 14.8 Hz, 1H of CH_2), 2.18 (3H, s, 3H of CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.6, 167.8, 138.9, 138.5, 138.2, 136.0, 133.2, 130.5, 128.9, 128.5, 128.4, 127.9, 127.8, 127.4, 123.8, 122.3, 63.9, 45.5, 43.4, 21.1; MS (EI) *m/z* 370 (M^+); IR $_{Vmax}$ (cm^{-1}) 1693, 1661, 1543, 1396, 1237, 738， 699; HRMS (EI): *m/z* calcd for $C_{24}H_{22}N_2O_2$ (M^+): 370.1681; Found: 370.1685.



***N*-benzyl-3-oxo-2-propylisoindoline-1-carboxamide (10c)**

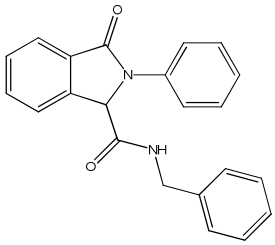
pale solid, mp 113-115 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70 (1H, br, 1H of NH), 7.63 (1H, d, J = 8.0 Hz), 7.47-7.50 (1H, m), 7.14-7.22 (6H, m), 7.07 (1H, d, J = 7.6

Hz), 5.09 (1H, s, 1H of CH), 4.48 (1H, dd, J_1 = 14.4 Hz, J_2 = 6.0 Hz, 1H of CH_2), 4.41 (1H, dd, J_1 = 14.4 Hz, J_2 = 6.0 Hz, 1H of CH_2), 3.80-3.88 (1H, m, 1H of C_3H_7), 3.05-3.12 (1H, m, 1H of C_3H_7), 1.52-1.65 (2H, m, 2H of C_3H_7), 0.83 (3H, t, J = 7.2 Hz, 3H of C_3H_7); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.3, 168.0, 141.0, 138.1, 131.8, 130.8, 128.7, 128.4, 127.6, 127.2, 123.3, 122.5, 64.6, 43.2, 21.1, 11.2; MS (EI) *m/z* 308 (M^+); IR $_{Vmax}$ (cm^{-1}) 1694, 1663, 1548, 1397, 1231, 751，699; HRMS (EI): *m/z* calcd for $C_{19}H_{20}N_2O_2$ (M^+): 308.1525; Found: 308.1531.



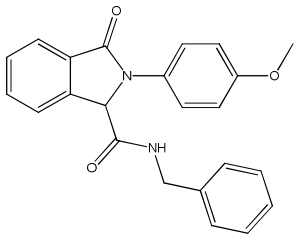
***N*-benzyl-2-cyclohexyl-3-oxoisoindoline-1-carboxamide (10d)**

pale solid, mp 174-176 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.61 (1H, d, J = 7.6 Hz), 7.50-7.54 (1H, m), 7.10-7.32 (8H, m), 5.11 (1H, s, 1H of CH), 4.44 (1H, dd, J_1 = 14.8 Hz, J_2 = 6.8 Hz, 1H of CH_2), 4.32 (1H, dd, J_1 = 14.4 Hz, J_2 = 6.0 Hz, 1H of CH_2), 3.94-4.00 (1H, m, 1H of C_6H_{11}), 1.08-1.93 (10H, m, 10H of C_6H_{11}); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.0, 169.2, 141.8, 137.7, 132.0, 131.2, 128.8, 128.4, 127.8, 127.4, 123.5, 122.4, 63.6, 53.6, 43.3, 30.8, 30.7, 25.7, 25.6, 25.1; MS (EI) *m/z* 348 (M^+); IR $_{Vmax}$ (cm^{-1}) 1695, 1664, 1544, 1398, 1230, 732，699; HRMS (EI): *m/z* calcd for $C_{22}H_{24}N_2O_2$ (M^+): 348.1838; Found: 348.1846.



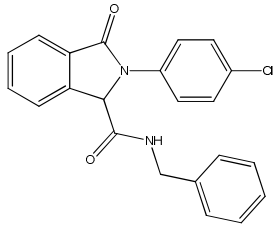
***N*-benzyl-3-oxo-2-phenylisoindoline-1-carboxamide (10e)**

pale solid, mp 204-206 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.70-7.76 (3H, m), 7.58-7.62 (1H, m), 7.07-7.44 (8H, m), 6.86 (1H, br, 1H of NH), 6.79 (2H, d, J = 6.8 Hz), 5.68 (1H, s, 1H of CH), 4.46 (1H, dd, J_1 = 15.2 Hz, J_2 = 6.8 Hz, 1H of CH_2), 4.16 (1H, dd, J_1 = 15.2 Hz, J_2 = 4.8 Hz, 1H of CH_2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.1, 167.6, 140.0, 137.8, 137.4, 132.8, 130.9, 129.3, 129.2, 128.3, 127.2, 127.1, 125.1, 124.1, 122.6, 120.1, 65.5, 43.1; MS (EI) *m/z* 342 (M^+); IR $_{Vmax}$ (cm^{-1}) 1704, 1666, 1498, 1368, 733，695; HRMS (EI): *m/z* calcd for $C_{22}H_{18}N_2O_2$ (M^+): 342.1368; Found: 342.1372.



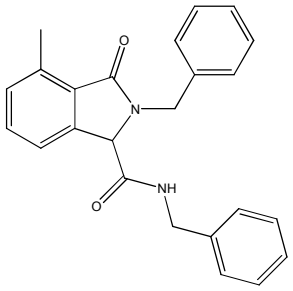
***N*-benzyl-2-(4-methoxyphenyl)-3-oxoisoindoline-1-carboxamide (10f)**

pale solid, mp 195-197 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.69 (1H, d, J = 7.6 Hz), 7.54-7.56 (3H, m), 7.21-7.29 (3H, m), 7.08-7.14 (3H, m), 6.79-6.85 (4H, m), 5.60 (1H, s, 1H of CH), 4.45 (1H, dd, J_1 = 15.2 Hz, J_2 = 6.8 Hz, 1H of CH_2), 4.18 (1H, dd, J_1 = 15.2 Hz, J_2 = 5.6 Hz, 1H of CH_2), 3.81 (3H, s, 3H of CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 167.9, 167.7, 157.0, 140.0, 137.6, 132.5, 130.9, 130.8, 129.1, 128.3, 127.2, 127.1, 123.9, 122.5, 122.1, 114.4, 65.9, 55.4, 43.1; MS (EI) *m/z* 372 (M^+); IR ν_{max} (cm^{-1}) 1700, 1665, 1513, 1378, 1250，734; HRMS (EI): *m/z* calcd for $C_{23}H_{20}N_2O_3$ (M^+):372.1474; Found: 372.1469.



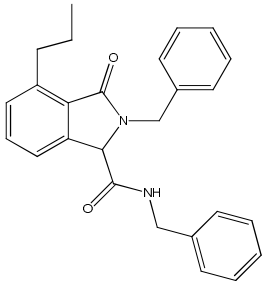
_N_-benzyl-2-(4-chlorophenyl)-3-oxoisoindoline-1-carboxamide (10g)

pale solid, mp 198-200 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.72 (1H, d, J = 7.6 Hz), 7.58-7.62 (3H, m), 7.32-7.35 (1H, m), 7.11-7.26 (7H, m), 6.85 (2H, d, J = 7.2 Hz), 5.60 (1H, s, 1H of CH), 4.48 (1H, dd, J_1 = 15.2 Hz, J_2 = 7.2 Hz, 1H of CH_2), 4.18 (1H, dd, J_1 = 15.2 Hz, J_2 = 5.2 Hz, 1H of CH_2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.1, 167.3, 139.9, 137.4, 136.4, 133.1, 130.5, 130.4, 129.3, 129.2, 128.4, 127.4, 127.3, 124.2, 122.6, 121.0, 65.6, 43.2; MS (EI) *m/z* 376 (M^+); IR ν_{max} (cm^{-1}) 1704, 1666, 1495, 1362, 1093，735; HRMS (EI): *m/z* calcd for $C_{22}H_{17}ClN_2O_2$ (M^+):376.0979; Found: 376.0977.



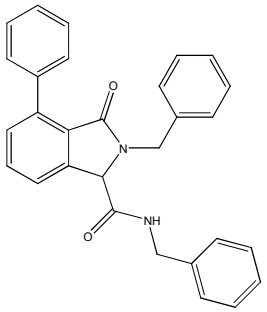
***N*,2-dibenzyl-4-methyl-3-oxoisoindoline-1-carboxamide (10h)**

pale solid, mp 181-183 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36-7.42 (2H, m), 7.16-7.29 (12H, m), 5.25 (1H, d, J = 14.8 Hz, 1H of CH_2), 4.83 (1H, s, 1H of CH), 4.45 (1H, dd, J_1 = 14.4 Hz, J_2 = 6.0 Hz, 1H of CH_2), 4.30 (1H, dd, J_1 = 14.4 Hz, J_2 = 6.0 Hz, 1H of CH_2), 4.14 (1H, d, J = 14.4 Hz, 1H of CH_2), 2.32 (3H, s, 3H of CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.0, 167.8, 141.7, 137.9, 137.8, 136.1, 131.9, 130.9, 128.9, 128.5, 128.5, 127.9, 127.7, 127.5, 127.4, 120.1, 63.5, 45.4, 43.4, 16.7; MS (EI) *m/z* 370 (M^+); IR ν_{max} (cm^{-1}) 1694, 1662, 1547, 1399, 752, 699; HRMS (EI): *m/z* calcd for $C_{24}H_{22}N_2O_2$ (M^+):370.1681; Found: 370.1679.



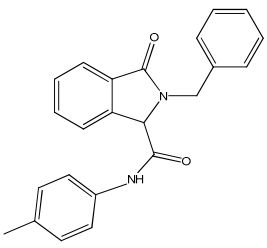
_N_,2-dibenzyl-3-oxo-4-propylisoindoline-1-carboxamide (10i)

pale solid, mp 142-144 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 (2H, d, J = 4.0 Hz), 7.20-7.30 (9H, m), 7.11 (2H, d, J = 7.6 Hz), 6.54 (1H, br, 1H of NH), 5.23 (1H, d, J = 14.8 Hz, 1H of CH_2), 4.83 (1H, s, 1H of CH), 4.42 (1H, dd, J_1 = 14.4 Hz, J_2 = 6.4 Hz, 1H of CH_2), 4.26 (1H, dd, J_1 = 14.4 Hz, J_2 = 5.2 Hz, 1H of CH_2), 4.17 (1H, d, J = 14.4 Hz, 1H of CH_2), 3.01-3.08 (1H, m, 1H of C_3H_7), 2.90-2.97 (1H, m, 1H of C_3H_7), 1.56-1.70 (2H, m, 2H of C_3H_7), 0.93 (3H, t, J = 7.2 Hz, 3H of C_3H_7); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.0, 167.8, 143.0, 141.7, 137.6, 136.1, 131.9, 130.1, 128.9, 128.7, 128.5, 128.0, 127.6, 127.5, 127.1, 120.3, 63.4, 45.6, 43.4, 32.6, 24.2, 13.9; MS (EI) *m/z* 398 (M^+); IR $_{Vmax}$ (cm^{-1}) 1698, 1663, 1549, 1402, 752, 698; HRMS (EI): *m/z* calcd for $C_{26}H_{26}N_2O_2$ (M^+):398.1994; Found: 398.1998.



_N_,2-dibenzyl-3-oxo-4-phenylisoindoline-1-carboxamide (10j)

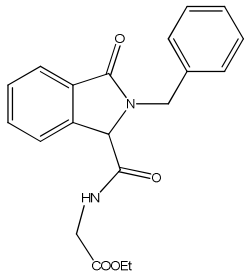
slurry. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.64 (1H, d, J = 7.2 Hz), 7.57 (1H, t, J = 6.8 Hz), 7.07-7.36 (17H, m), 5.17 (1H, d, J = 14.8 Hz, 1H of CH_2), 4.82 (1H, s, 1H of CH), 4.41 (1H, dd, J_1 = 14.8 Hz, J_2 = 6.8 Hz, 1H of CH_2), 4.00 (1H, d, J = 14.4 Hz, 1H of CH_2), 3.91 (1H, dd, J_1 = 14.4 Hz, J_2 = 4.8 Hz, 1H of CH_2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 168.5, 167.4, 142.3, 141.1, 138.0, 137.2, 135.8, 131.9, 131.3, 129.4, 128.8, 128.6, 128.5, 127.9, 127.8, 127.7, 127.5, 127.3, 126.3, 121.8, 63.0, 45.5, 43.4; MS (EI) *m/z* 432 (M^+); IR $_{Vmax}$ (cm^{-1}) 1696, 1666, 1541, 1397, 754, 698; HRMS (EI): *m/z* calcd for $C_{29}H_{24}N_2O_2$ (M^+):432.1838; Found: 432.1837.



2-benzyl-3-oxo-*N*-*p*-tolylisoindoline-1-carboxamide (10k)

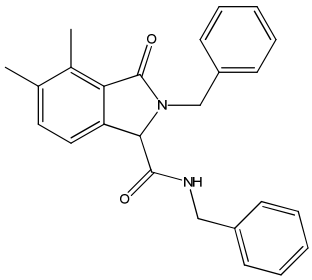
pale solid, mp 203-205 ^{o}C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.45 (1H, br, 1H of NH), 7.62-7.66 (3H, m), 7.45-7.49 (1H, m), 7.23-7.33 (7H, m), 7.09 (2H, d, J = 8.4 Hz),

5.42 (1H, d, J = 14.4 Hz, 1H of CH_2), 5.01 (1H, s, 1H of CH), 4.40 (1H, d, J = 14.8 Hz, 1H of CH_2), 2.30 (3H, s, 3H of CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.7, 165.8, 141.0, 135.8, 135.3, 134.1, 132.3, 130.5, 129.3, 129.0, 128.9, 128.5, 128.0, 123.4, 122.8, 120.1, 65.0, 45.9, 20.8; MS (EI) *m/z* 356 (M^+); IR $_{Vmax}$ (cm^{-1}) 1700, 1663, 1608, 1538, 1406，731; HRMS (EI): *m/z* calcd for $C_{23}H_{20}N_2O_2$ (M^+):356.1525; Found: 356.1518.



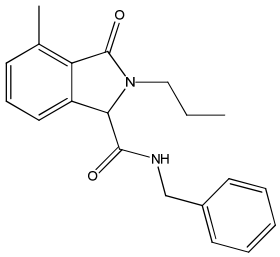
ethyl 2-(2-benzyl-3-oxoisoindoline-1-carboxamido)acetate (10l)

pale solid, mp 131-133 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.83 (1H, br, 1H of NH), 7.58-7.61 (2H, m), 7.50-7.54 (1H, m), 7.39-7.41 (1H, m), 7.24-7.28 (5H, m), 5.37 (1H, d, J = 14.8 Hz, 1H of CH_2), 4.89 (1H, s, 1H of CH), 4.39 (1H, d, J = 14.8 Hz, 1H of CH_2), 4.20 (1H, dd, J_1 = 13.6 Hz, J_2 = 6.4 Hz, 1H of CH_2), 4.15-4.23 (2H, m, 2H of C_2H_5), 3.87 (1H, dd, J_1 = 17.6 Hz, J_2 = 5.2 Hz, 1H of CH_2), 1.25 (3H, t, J = 7.2 Hz, 3H of C_2H_5); ^{13}C NMR (100 MHz, $CDCl_3$) δ 169.4, 169.3, 168.2, 140.9, 136.0, 132.2, 130.6, 129.0, 128.8, 128.5, 127.9, 123.7, 122.8, 63.6, 61.3, 45.2, 41.1, 14.1; MS (EI) *m/z* 352 (M^+); IR $_{Vmax}$ (cm^{-1}) 1700, 1663, 1608, 1538, 1406, 731; HRMS (EI): *m/z* calcd for $C_{20}H_{20}N_2O_4$ (M^+):352.1423; Found: 352.1426.



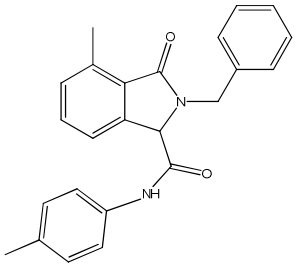
***N*,2-dibenzyl-4,5-dimethyl-3-oxoisoindoline-1-carboxamide (10m)**

pale solid, mp 153-155 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.20-7.31 (10H, m), 7.13 (2H, d, J = 7.2 Hz), 6.73 (1H, br, 1H of NH), 5.24 (1H, d, J = 14.4 Hz, 1H of CH_2), 4.79 (1H, s, 1H of CH), 4.42 (1H, dd, J_1 = 14.4 Hz, J_2 = 6.4 Hz, 1H of CH_2), 4.26 (1H, dd, J_1 = 14.8 Hz, J_2 = 5.6 Hz, 1H of CH_2), 4.15 (1H, d, J = 14.4 Hz, 1H of CH_2), 2.41 (3H, s, 3H of CH_3), 2.28 (3H, s, 3H of CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.5, 168.1, 139.3, 138.4, 137.7, 136.8, 136.2, 133.4, 128.9, 128.6, 128.5, 127.9, 127.7, 127.5, 127.3, 119.7, 62.9, 45.6, 43.4, 19.2, 12.6; MS (EI) *m/z* 384 (M^+); IR $_{Vmax}$ (cm^{-1}) 1691, 1661, 1543, 1391, 1238, 698; HRMS (EI): *m/z* calcd for $C_{25}H_{24}N_2O_2$ (M^+):384.1838; Found: 384.1833.



***N*-benzyl-4-methyl-3-oxo-2-propylisoindoline-1-carboxamide (10n)**

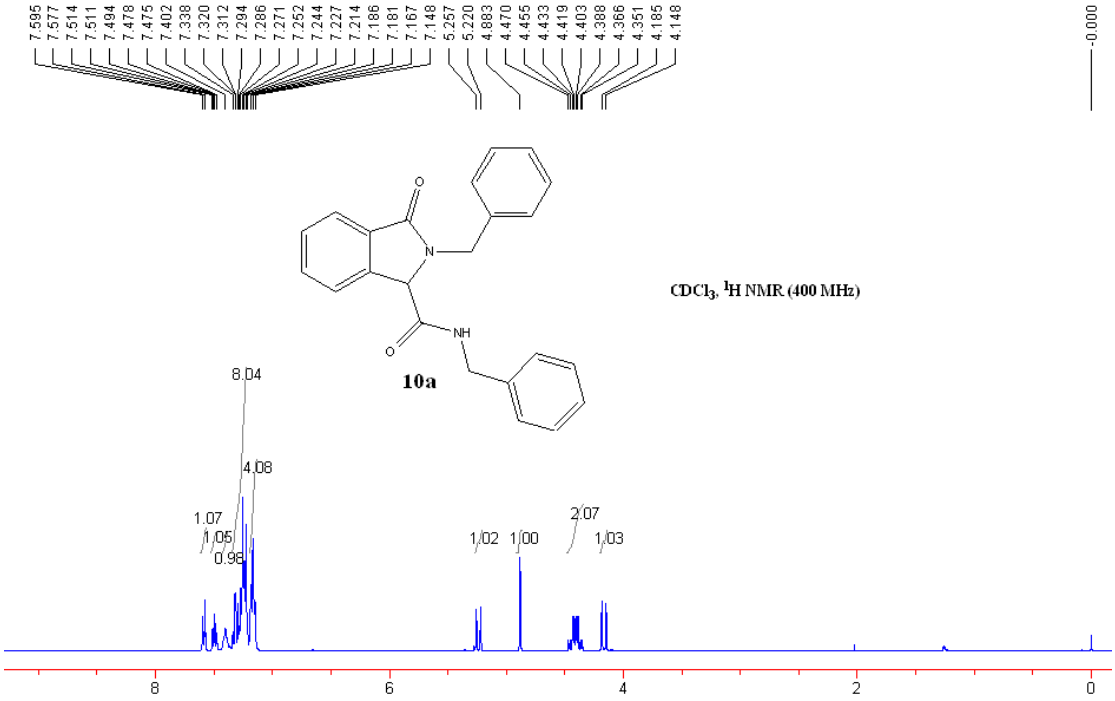
pale solid, mp 111-113 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 (1H, d, J = 7.6 Hz), 7.39-7.43 (1H, m), 7.21-7.26 (3H, m), 7.13-7.16 (3H, m), 7.02 (1H, br, 1H of NH), 5.04 (1H, s, 1H of CH), 4.50 (1H, dd, J_1 = 14.8 Hz, J_2 = 6.8 Hz, 1H of CH_2), 4.29 (1H, dd, J_1 = 14.4 Hz, J_2 = 5.2 Hz, 1H of CH_2), 3.84-3.91 (1H, m, 1H of C_3H_7), 3.03-3.10 (1H, m, 1H of C_3H_7), 2.31 (3H, s, 3H of CH_3), 1.56-1.65 (2H, m, 2H of C_3H_7), 0.87 (3H, t, J = 7.2 Hz, 3H of C_3H_7); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.2, 168.2, 141.5, 137.8, 131.7, 131.0, 128.5, 127.9, 127.6, 127.4, 120.1, 64.1, 43.4, 43.2, 21.1, 16.7, 11.3; MS (EI) *m/z* 322 (M^+); IR ν_{max} (cm^{-1}) 1698, 1664, 1521, 1377, 751，700; HRMS (EI): *m/z* calcd for $C_{20}H_{22}N_2O_2$ (M^+):322.1681; Found: 322.1687.

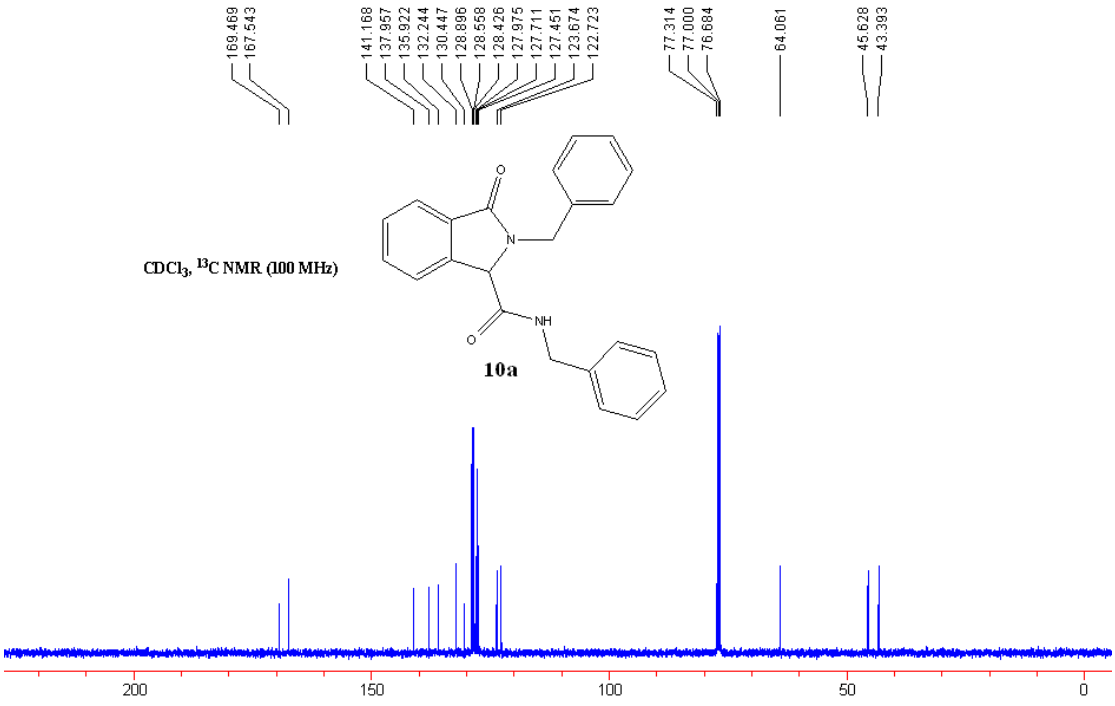


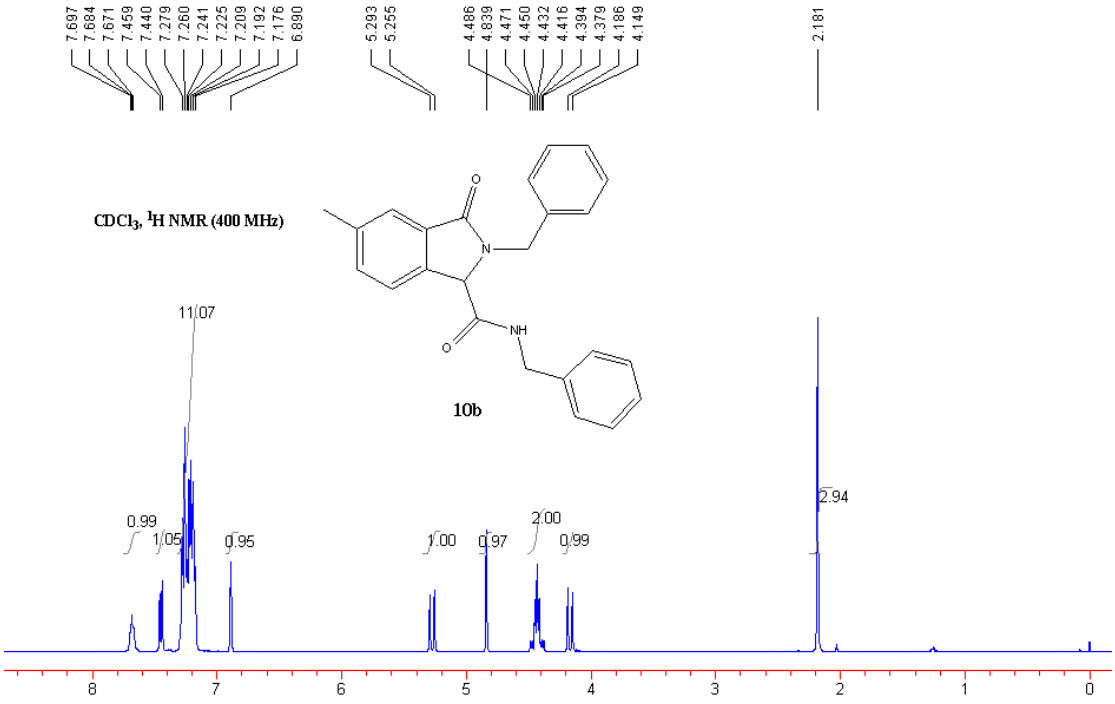
2-benzyl-4-methyl-3-oxo-*N*-*p*-tolylisoindoline-1-carboxamide (10o)

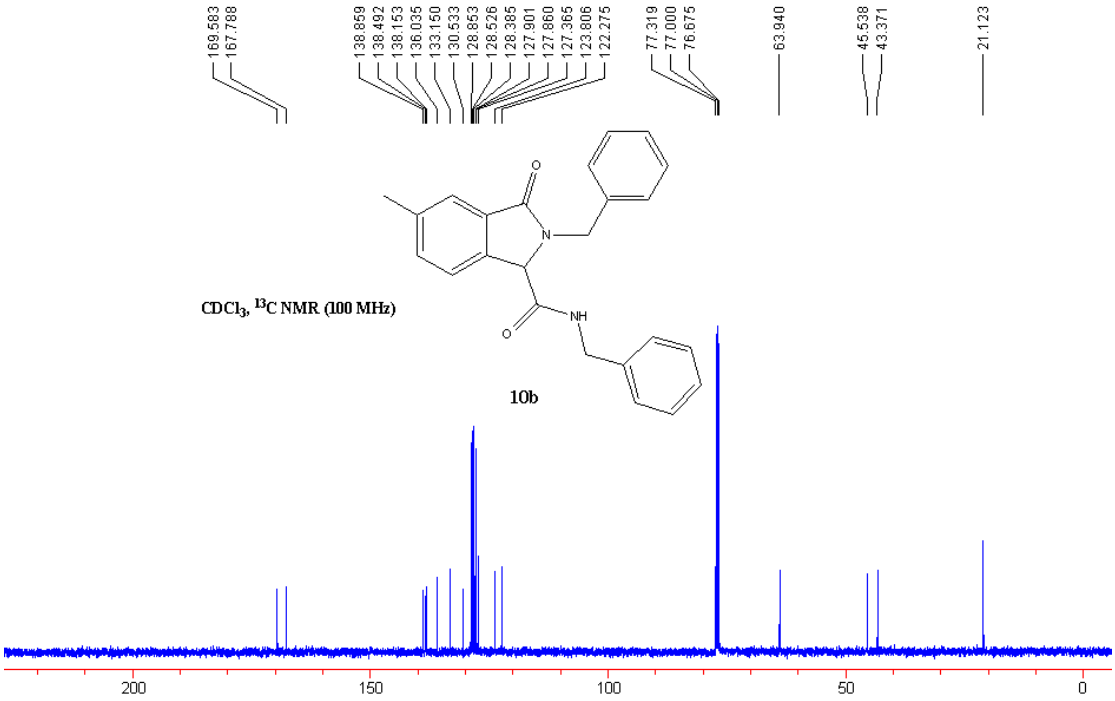
pale solid, mp 226-228 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 9.04 (1H, br, 1H of NH), 7.56 (2H, d, J = 8.8 Hz), 7.48 (1H, d, J = 7.6 Hz), 7.36-7.40 (1H, m), 7.28-7.32 (5H, m), 7.08-7.10 (3H, m), 5.47 (1H, d, J = 14.4 Hz, 1H of CH_2), 4.96 (1H, s, 1H of CH), 4.40 (1H, d, J = 14.8 Hz, 1H of CH_2), 2.30 (3H, s, 3H of CH_3), 2.11 (3H, s, 3H of CH_3); ^{13}C NMR (100 MHz, $CDCl_3$) δ 170.2, 166.0, 141.5, 138.0, 136.0, 135.2, 134.1, 132.0, 131.0, 129.3, 129.0, 128.6, 128.0, 127.5, 120.3, 120.1, 64.5, 45.8, 20.8, 16.5; MS (EI) *m/z* 370 (M^+); IR ν_{max} (cm^{-1}) 1703, 1604, 1515, 1481, 1405，736; HRMS (EI): *m/z* calcd for $C_{24}H_{22}N_2O_2$ (M^+):370.1681; Found: 370.1677.

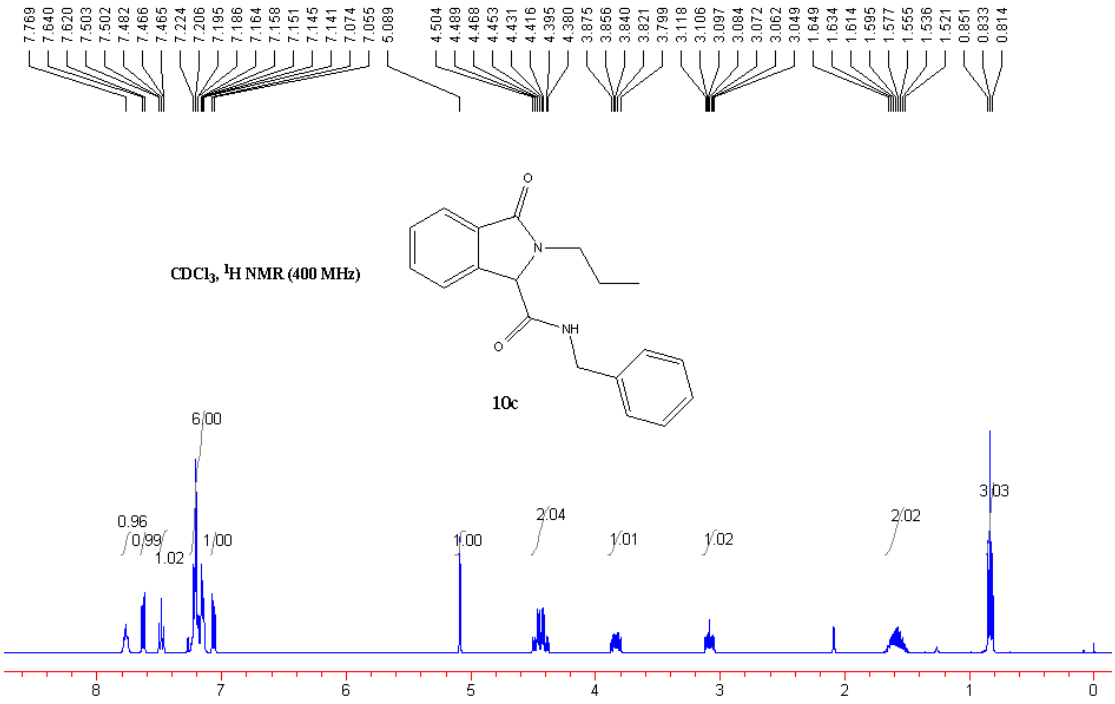
3. ^{1}H NMR and ^{13}C NMR spectra

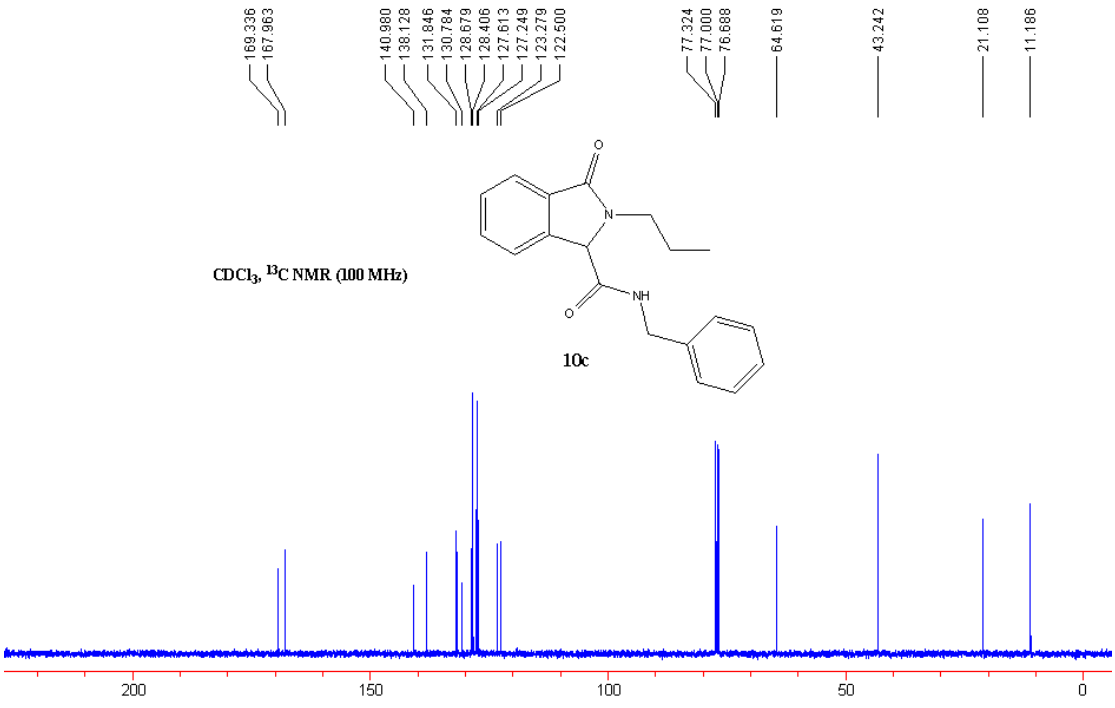


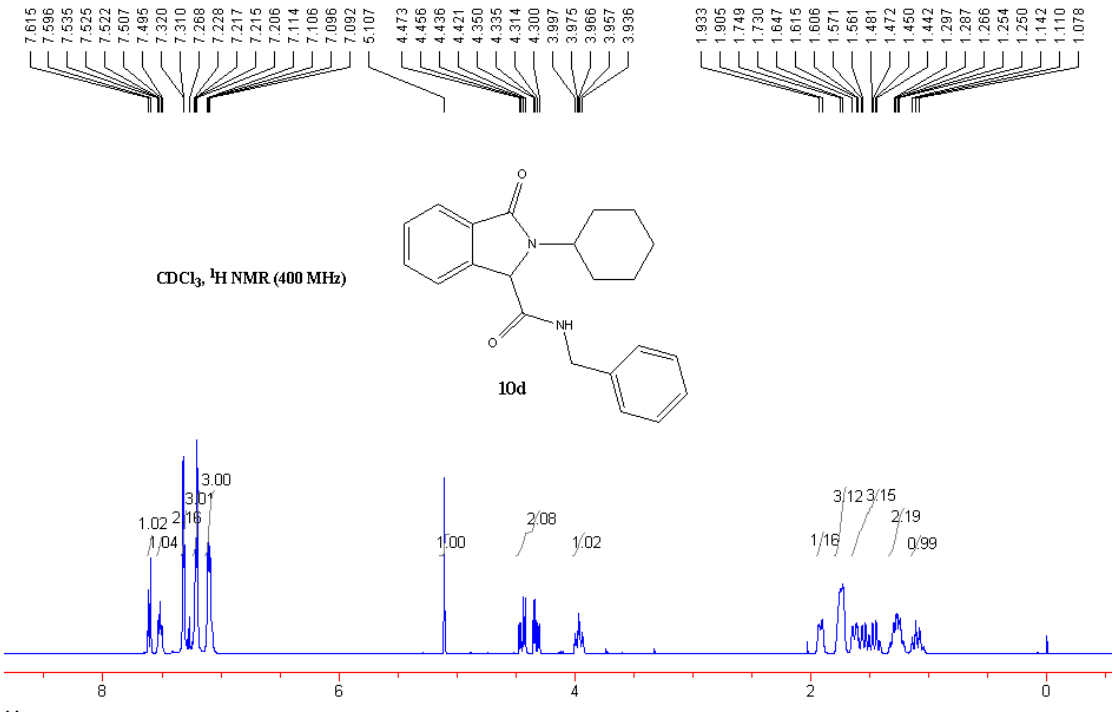


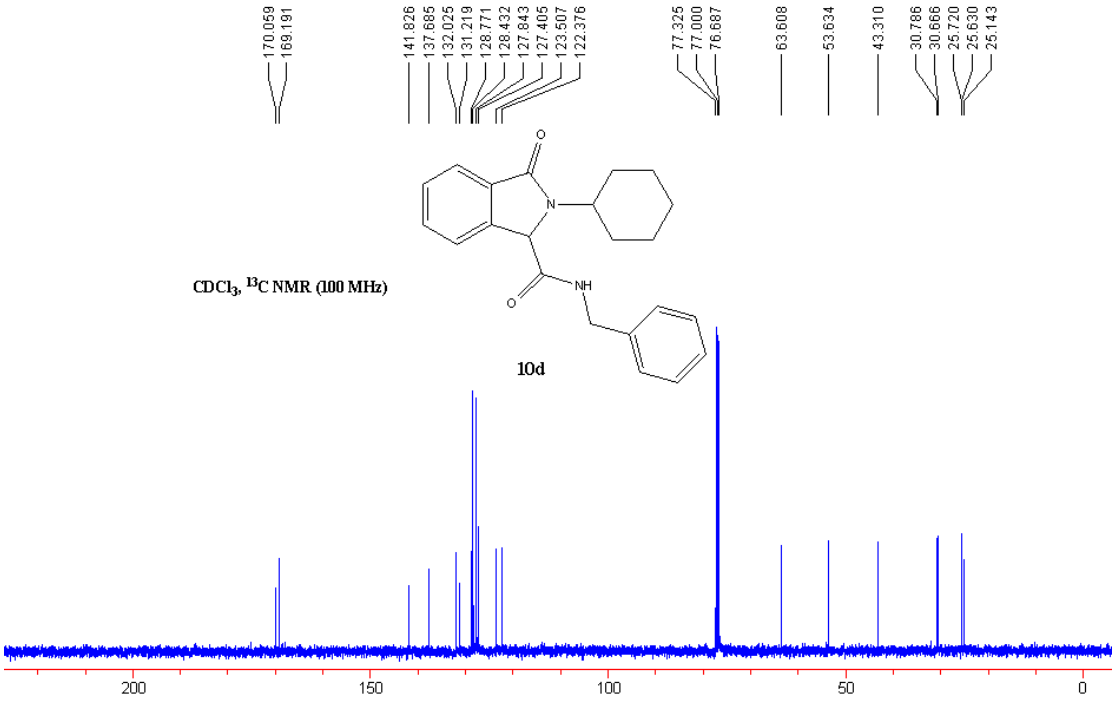

7.697
7.684
7.671
7.459
7.440
7.279
7.260
7.241
7.225
7.209
7.192
7.176
6.890
5.293
5.255
4.486
4.839
4.471
4.450
4.432
4.416
4.394
4.379
4.186
4.149
2.181
CDCl3, 1H NMR (400 MHz)
O
N
NH
O
10b
11.07
0.99
1.05
0.95
1.00
0.97
2.00
0.99
2.94
8
7
6
5
4
3
2
1
0

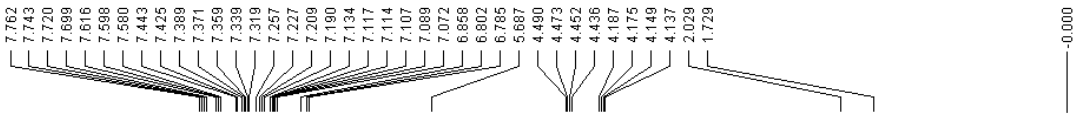

169.583
167.788
138.859
138.492
138.153
136.035
133.150
130.533
128.853
128.526
128.385
127.901
127.860
127.365
123.806
122.275
77.319
77.000
76.675
63.940
45.538
43.371
21.123
CDCl3, 13C NMR (100 MHz)
O
N
NH
O
10b
200
150
100
50
0

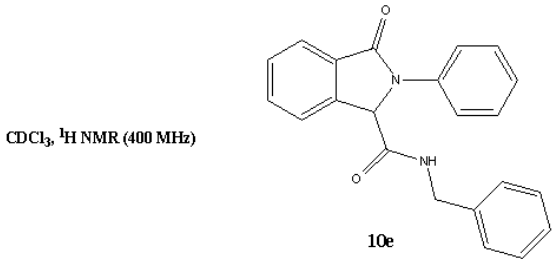

7.769
7.640
7.620
7.503
7.502
7.482
7.466
7.465
7.224
7.206
7.195
7.186
7.164
7.158
7.151
7.145
7.141
7.074
7.055
5.089
4.504
4.489
4.468
4.453
4.431
4.416
4.395
4.380
3.875
3.856
3.840
3.821
3.799
3.118
3.106
3.097
3.084
3.072
3.062
3.049
1.649
1.634
1.614
1.595
1.577
1.555
1.536
1.521
0.851
0.833
0.814
CDCl3, 1H NMR (400 MHz)
O
N
NH
O
10c
6.00
0.96
0.99
1.02
1.00
1.00
2.04
1.01
1.02
2.02
3.03
8
7
6
5
4
3
2
1
0


169.336
167.963
140.980
138.128
131.846
130.784
128.679
128.406
127.613
127.249
123.279
122.500
77.324
77.000
76.688
64.619
43.242
21.108
11.186
CDCl3, 13C NMR (100 MHz)
O
N
NH
O
10c
200
150
100
50
0


7.615
7.596
7.535
7.525
7.522
7.507
7.495
7.320
7.310
7.268
7.228
7.217
7.215
7.206
7.114
7.106
7.096
7.092
5.107
4.473
4.456
4.436
4.421
4.350
4.335
4.314
4.300
3.997
3.975
3.966
3.957
3.936
1.933
1.905
1.749
1.730
1.647
1.615
1.606
1.571
1.561
1.481
1.472
1.450
1.442
1.297
1.287
1.266
1.254
1.250
1.142
1.110
1.078
CDCl3, 1H NMR (400 MHz)
O
N
NH
O
10d
1.02
1.04
2.16
3.01
3.00
1.00
2.08
1.02
1.16
3.12
3.15
2.19
0.99
8
6
4
2
0

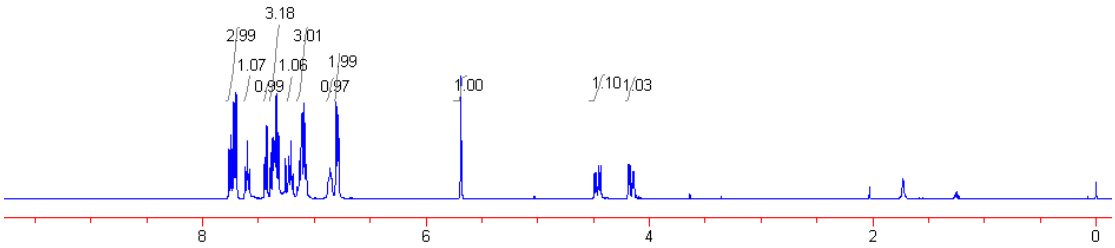

170.059
169.191
141.826
137.685
132.025
131.219
128.771
128.432
127.843
127.405
123.507
122.376
77.325
77.000
76.687
63.608
53.634
43.310
30.786
30.666
25.720
25.630
25.143
CDCl3, 13C NMR (100 MHz)
O
N
NH
O
10d
200
150
100
50
0

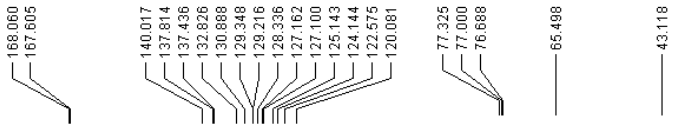


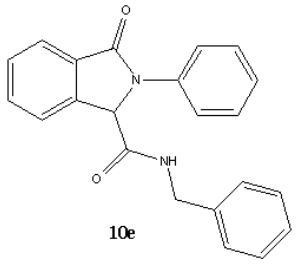


$CDCl_3$, ^1H NMR (400 MHz)

10e

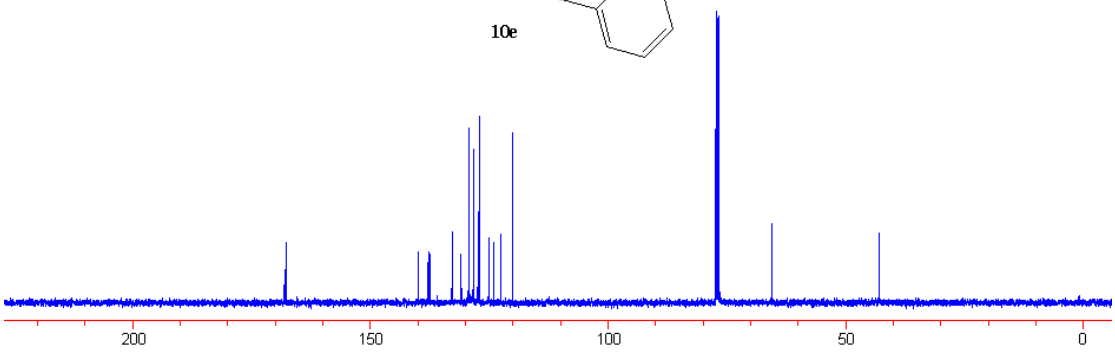


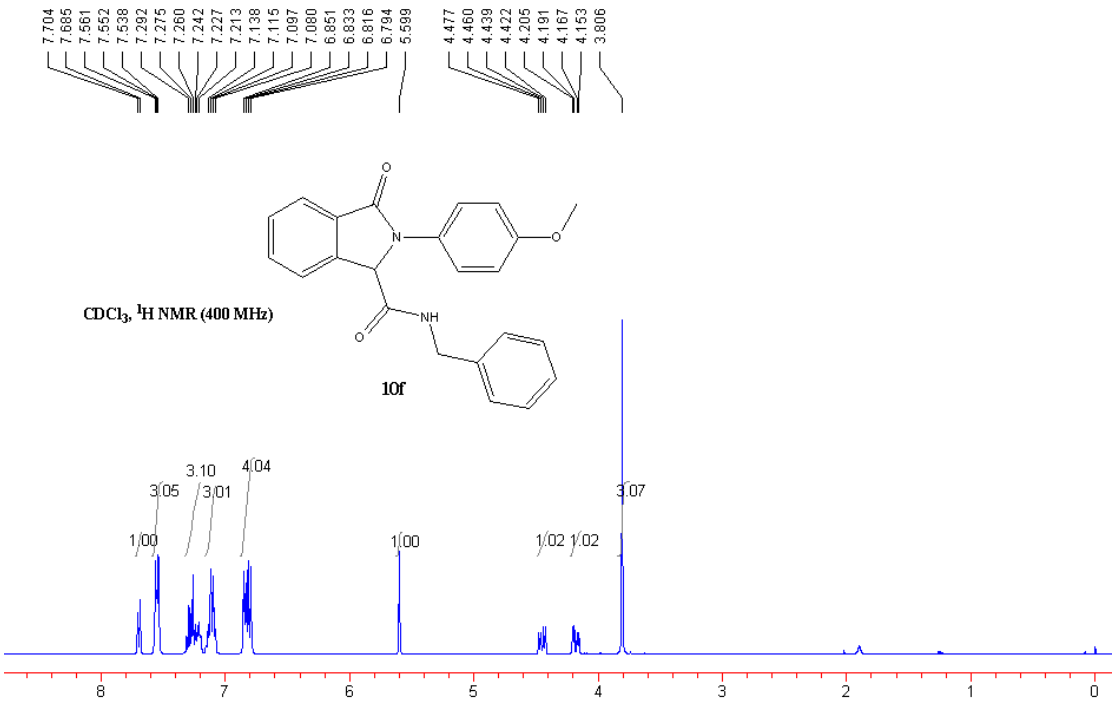


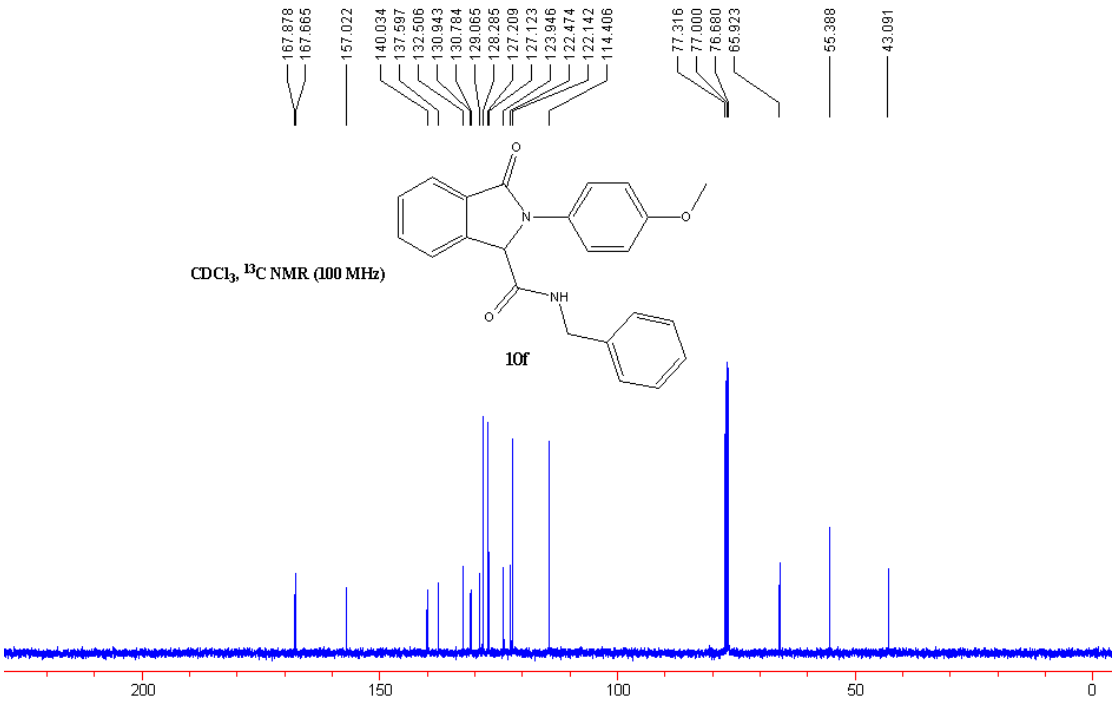


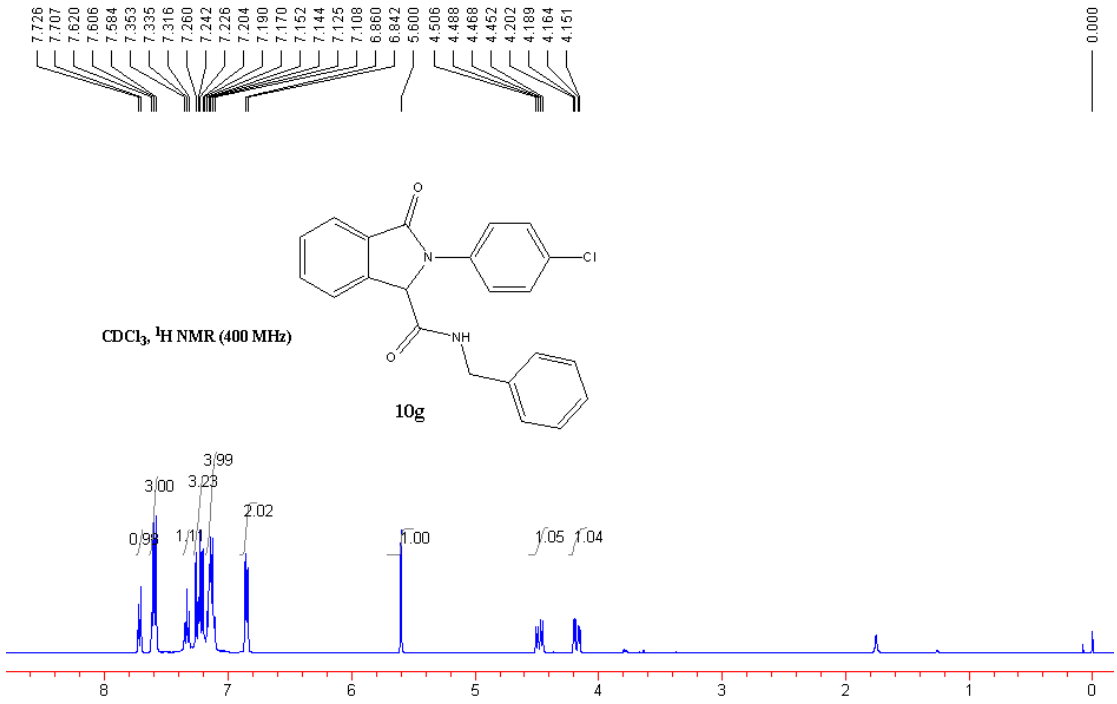
$CDCl_3$, ^{13}C NMR (100 MHz)

10e



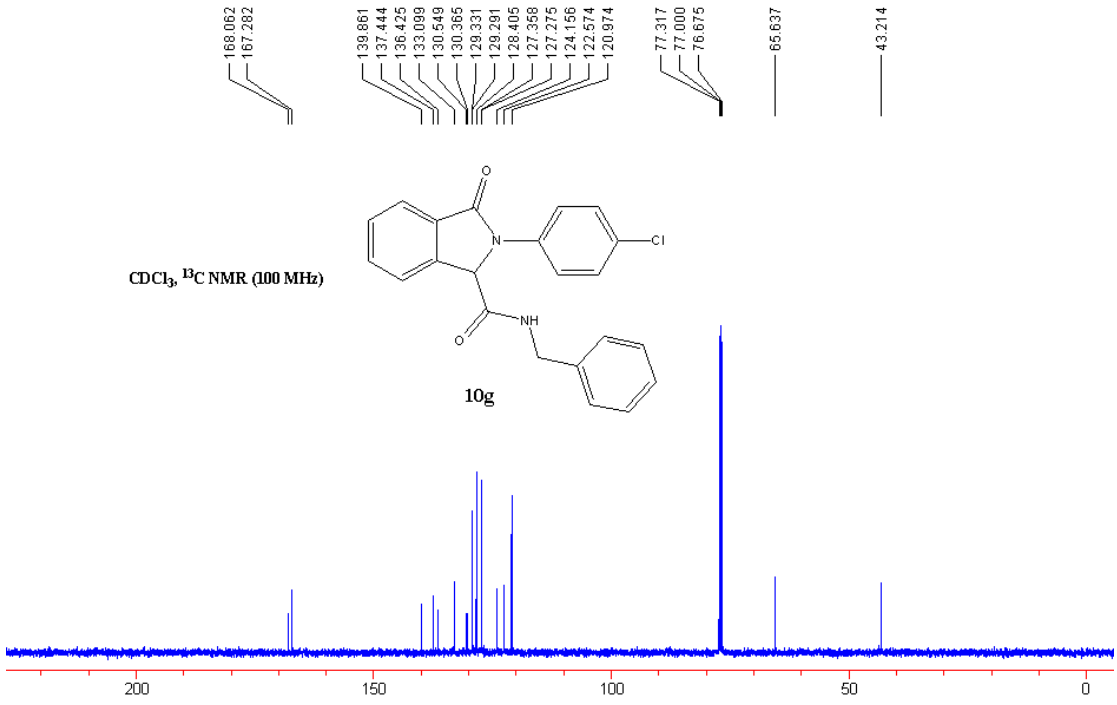

7.704
7.685
7.561
7.552
7.538
7.292
7.275
7.260
7.242
7.227
7.213
7.138
7.115
7.097
7.080
6.851
6.833
6.816
6.794
5.599
4.477
4.460
4.439
4.422
4.205
4.191
4.167
4.153
3.806
O
N
O
NH
O
CDCl3, 1H NMR (400 MHz)
10f
3.10
4.04
3.05
3.01
3.07
1.00
1.00
1.02
1.02
8
7
6
5
4
3
2
1
0


167.878
167.665
157.022
140.034
137.597
132.506
130.943
130.784
129.065
128.285
127.209
127.123
123.946
122.474
122.142
114.406
77.316
77.000
76.680
65.923
55.388
43.091
O
N
O
NH
O
CDCl3, 13C NMR (100 MHz)
10f
200
150
100
50
0



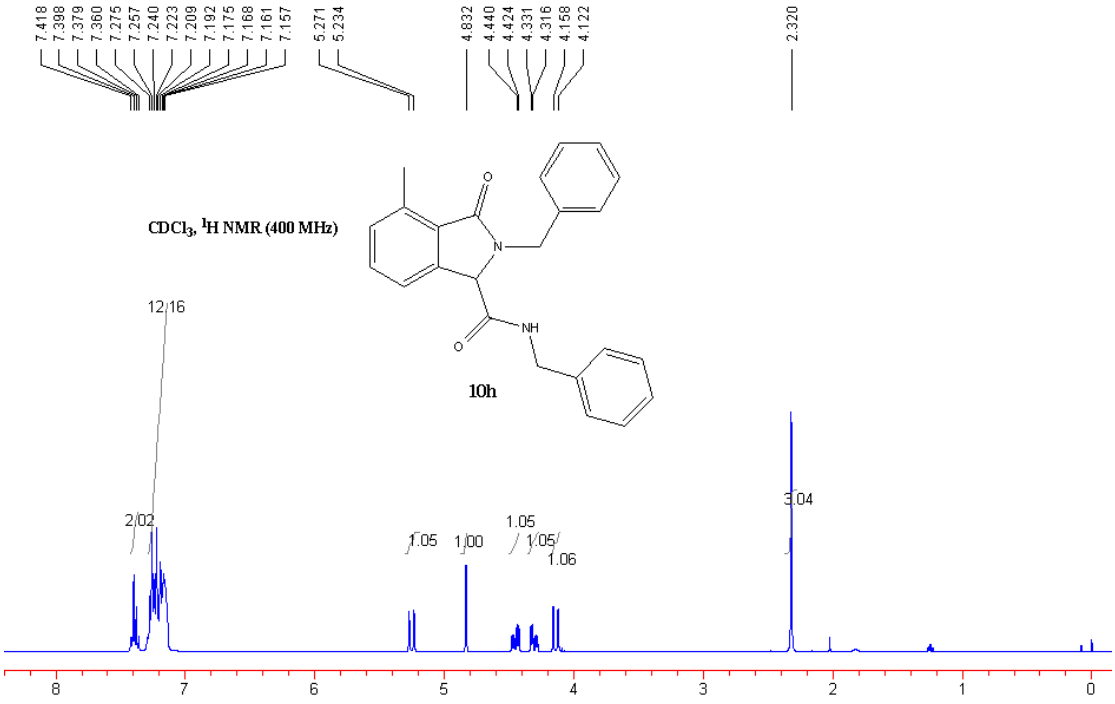
$CDCl_3$, ^1H NMR (400 MHz)

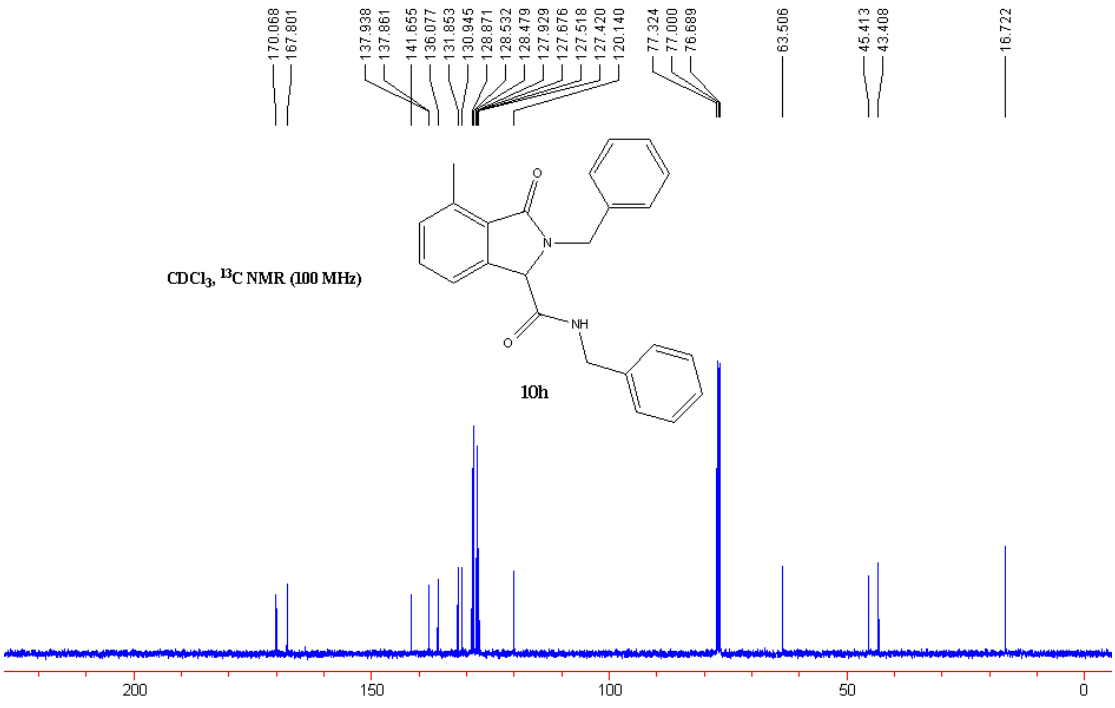
10g

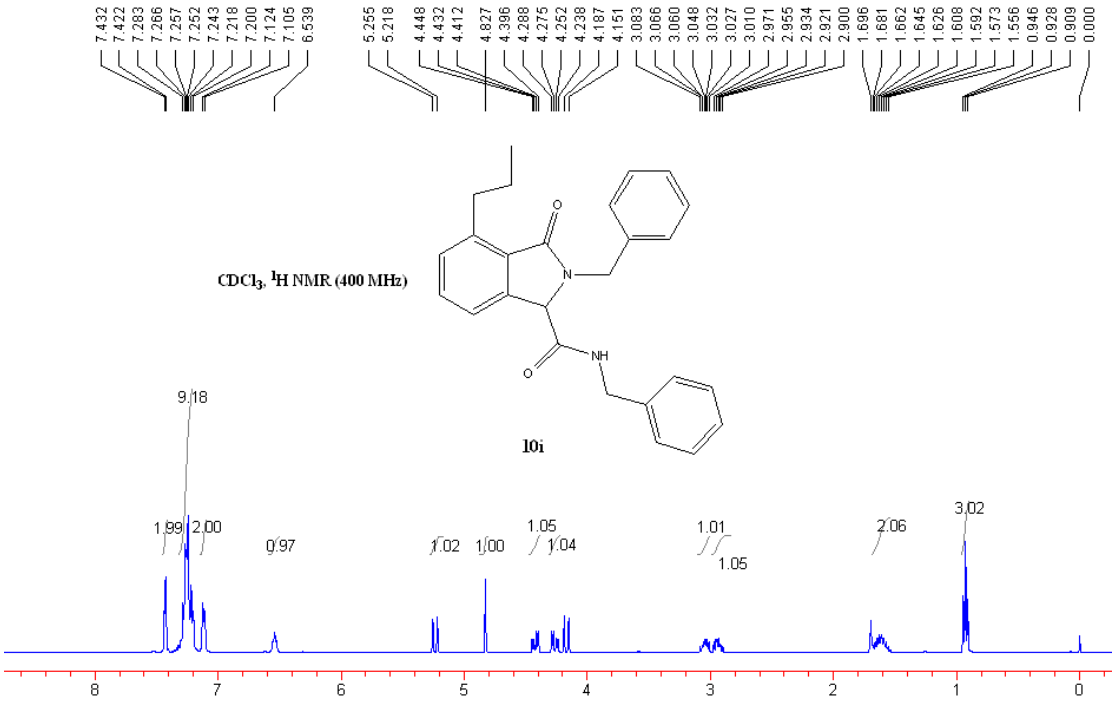


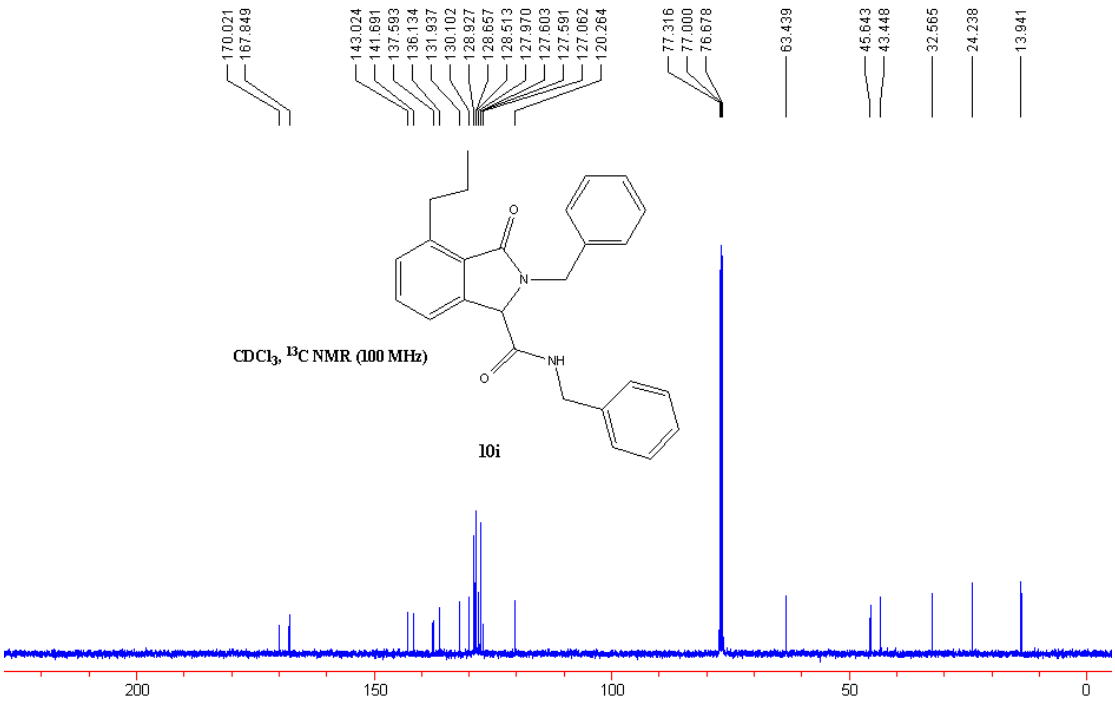
$CDCl_3$, ^{13}C NMR (100 MHz)

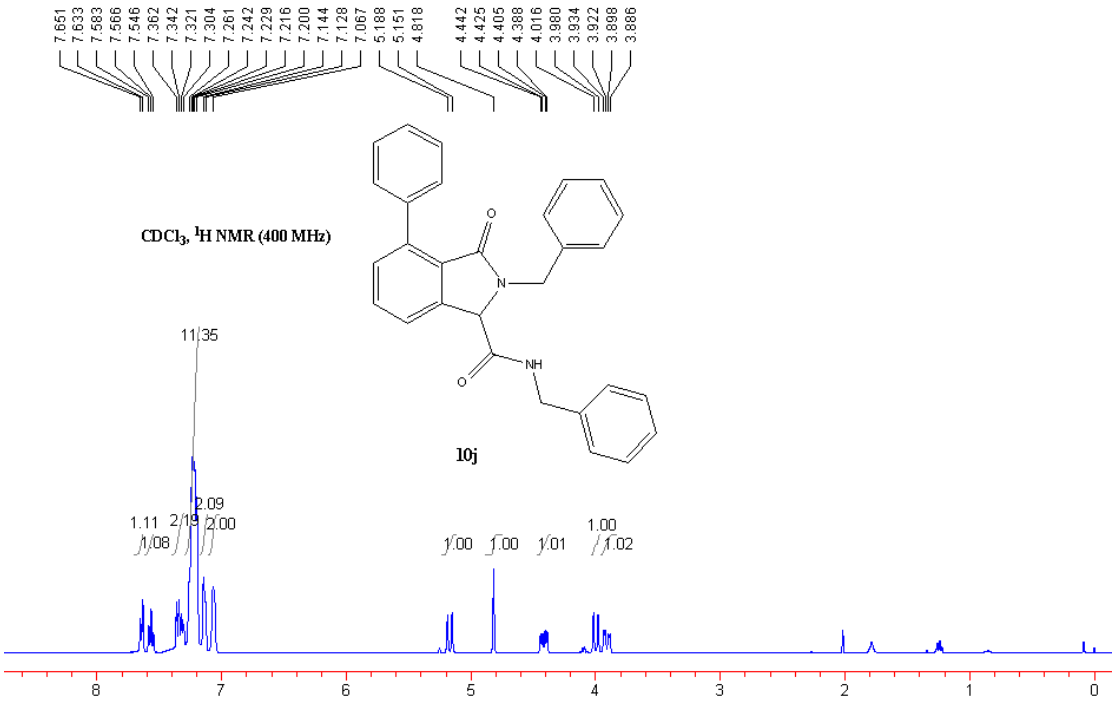
10g

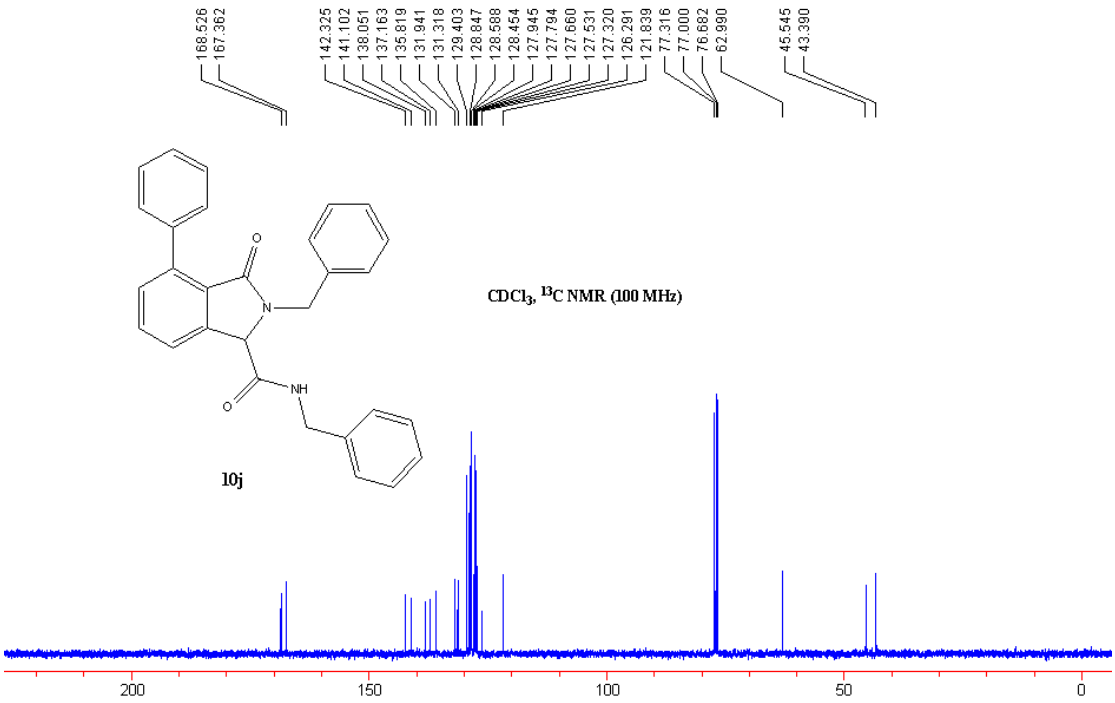

7.418
7.398
7.379
7.360
7.275
7.257
7.240
7.223
7.209
7.192
7.175
7.168
7.161
7.157
5.271
5.234
4.832
4.440
4.424
4.331
4.316
4.158
4.122
2.320
CDCl3, 1H NMR (400 MHz)
O
N
NH
O
10h
12.16
2.02
1.05
1.00
1.05
1.05
1.06
3.04
8
7
6
5
4
3
2
1
0

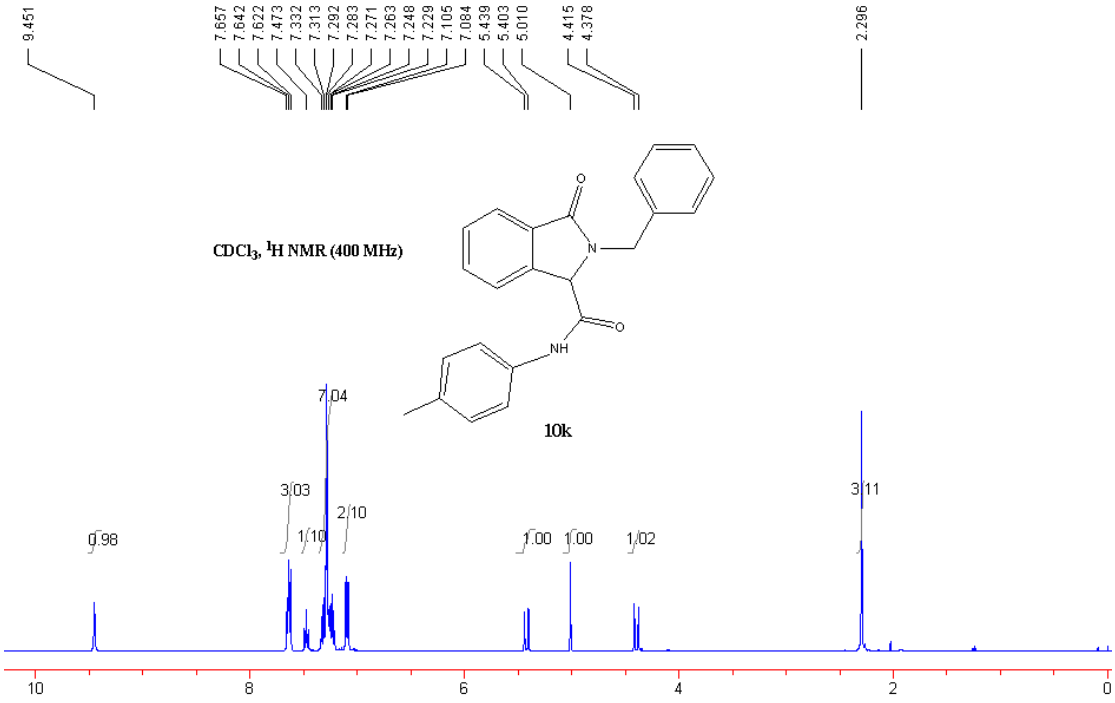

170.068
167.801
137.938
137.861
141.655
136.077
131.853
130.945
128.871
128.532
128.479
127.929
127.676
127.518
127.420
120.140
77.324
77.000
76.689
63.506
45.413
43.408
16.722
CDCl3, 13C NMR (100 MHz)
O
N
NH
O
10h
200
150
100
50
0

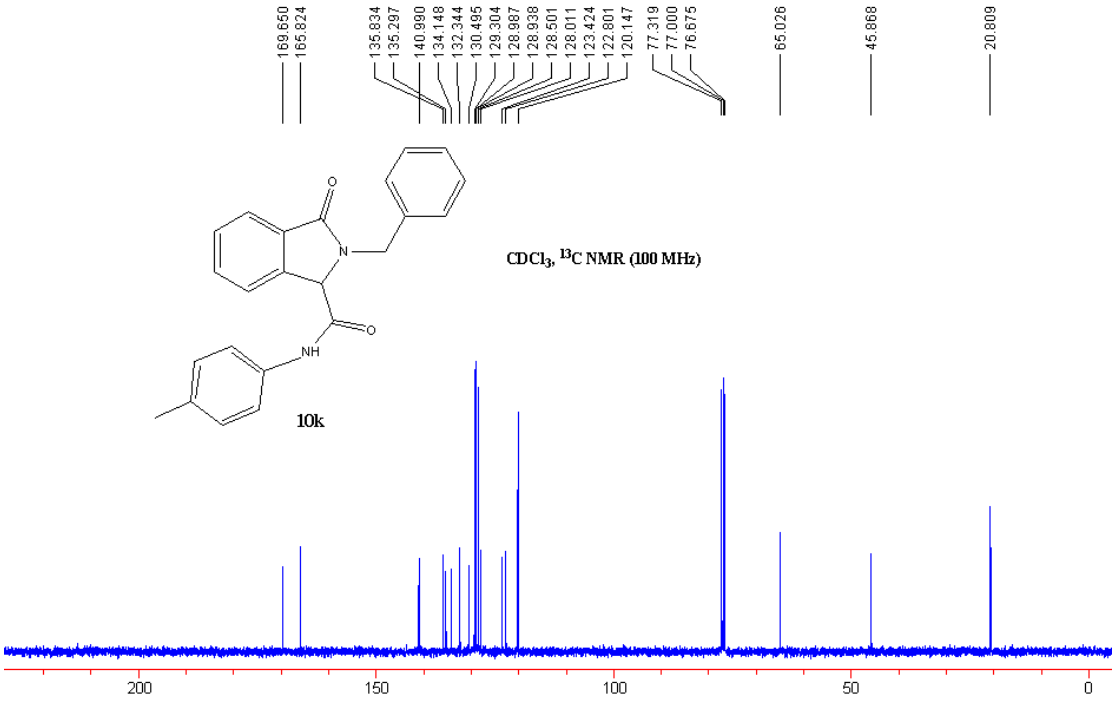

7.432
7.422
7.283
7.266
7.257
7.252
7.243
7.218
7.200
7.124
7.105
6.539
5.255
5.218
4.448
4.432
4.412
4.827
4.396
4.288
4.275
4.252
4.238
4.187
4.151
3.083
3.066
3.060
3.048
3.032
3.027
3.010
2.971
2.955
2.934
2.921
2.900
1.696
1.681
1.662
1.645
1.626
1.608
1.592
1.573
1.556
0.946
0.928
0.909
0.000
CDCl3, 1H NMR (400 MHz)
O
N
NH
O
10i
9.18
1.99
2.00
0.97
1.02
1.00
1.05
1.04
1.01
1.05
2.06
3.02
8
7
6
5
4
3
2
1
0

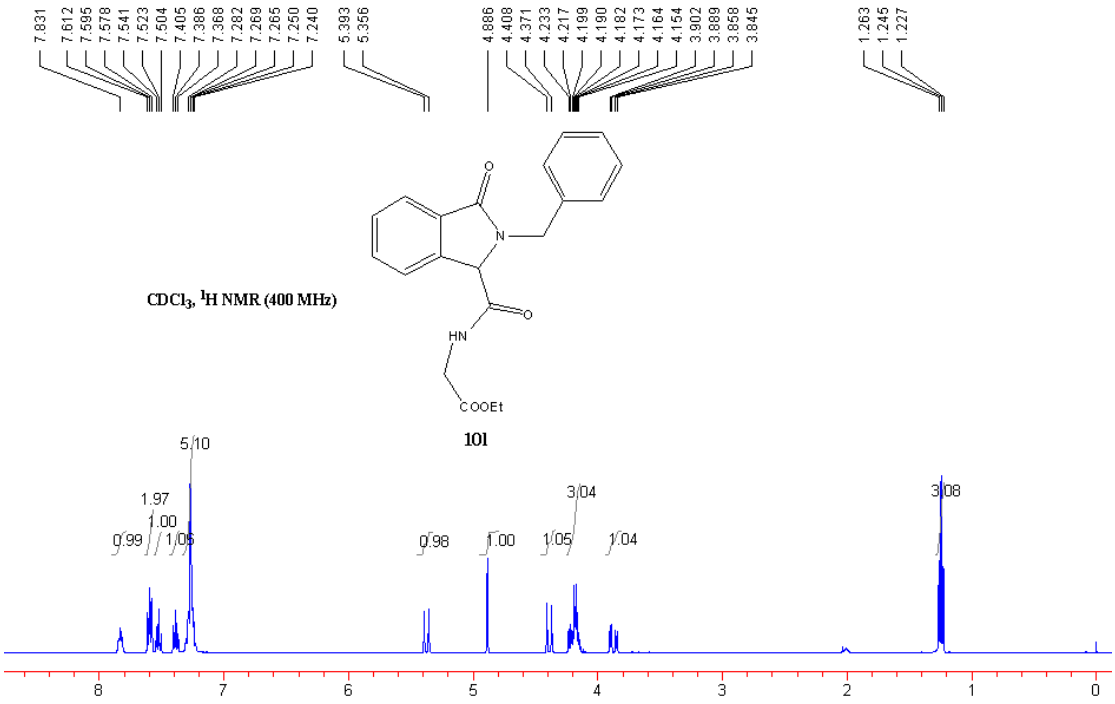

170.021
167.849
143.024
141.691
137.593
136.134
131.937
130.102
128.927
128.657
128.513
127.970
127.603
127.591
127.062
120.264
77.316
77.000
76.678
63.439
45.643
43.448
32.565
24.238
13.941
CDCl3, 13C NMR (100 MHz)
O
N
NH
O
10i
200
150
100
50
0


7.651
7.633
7.583
7.566
7.546
7.362
7.342
7.321
7.304
7.261
7.242
7.229
7.216
7.200
7.144
7.128
7.067
5.188
5.151
4.818
4.442
4.425
4.405
4.388
4.016
3.980
3.934
3.922
3.898
3.886
CDCl3, 1H NMR (400 MHz)
O
N
NH
O
10j
11.35
2.09
1.11
2.19
2.00
1.08
1.00
1.00
1.00
1.01
1.02
8
7
6
5
4
3
2
1
0


168.526
167.362
142.325
141.102
138.051
137.163
135.819
131.941
131.318
129.403
128.847
128.588
128.454
127.945
127.794
127.660
127.531
127.320
126.291
121.839
77.316
77.000
76.682
62.990
45.545
43.390
CDCl3, 13C NMR (100 MHz)
O
N
NH
O
10j
200
150
100
50
0

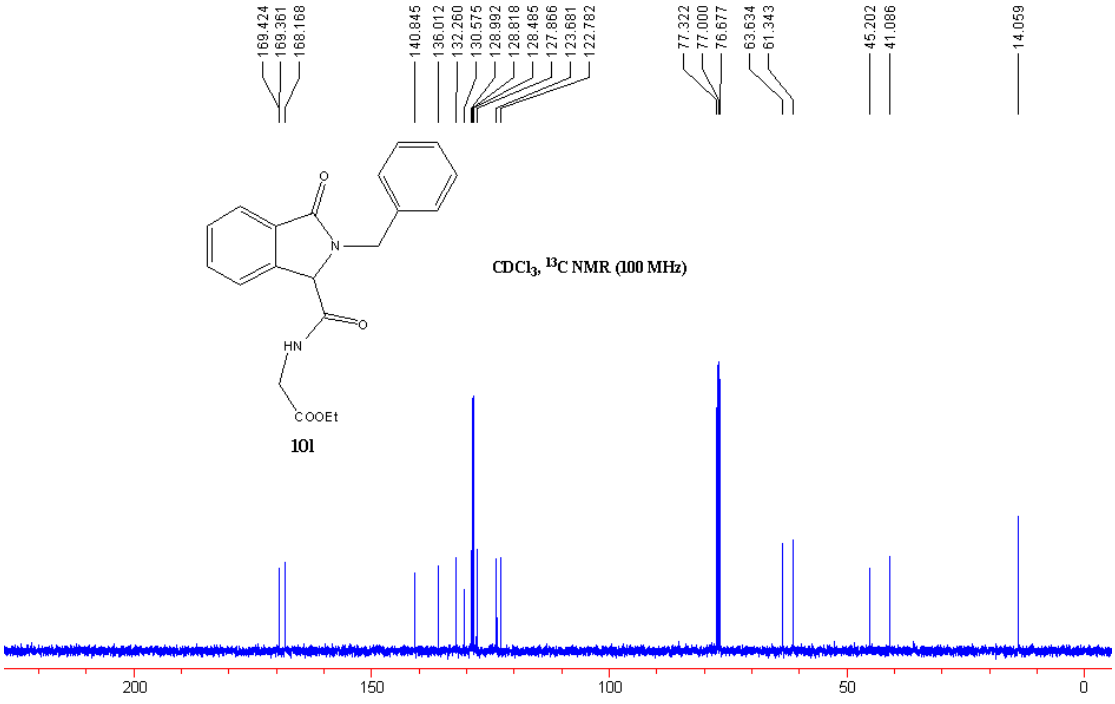

9.451
7.657
7.642
7.622
7.473
7.332
7.313
7.292
7.283
7.271
7.263
7.248
7.229
7.105
7.084
5.439
5.403
5.010
4.415
4.378
2.296
CDCl3, 1H NMR (400 MHz)
O
N
O
NH
10k
7.04
3.03
2.10
1.10
0.98
1.00
1.00
1.02
3.11
10
8
6
4
2
0


169.650
165.824
135.834
135.297
140.990
134.148
132.344
130.495
129.304
128.987
128.938
128.501
128.011
123.424
122.801
120.147
77.319
77.000
76.675
65.026
45.868
20.809
CDCl3, 13C NMR (100 MHz)
O
N
O
NH
10k
200
150
100
50
0



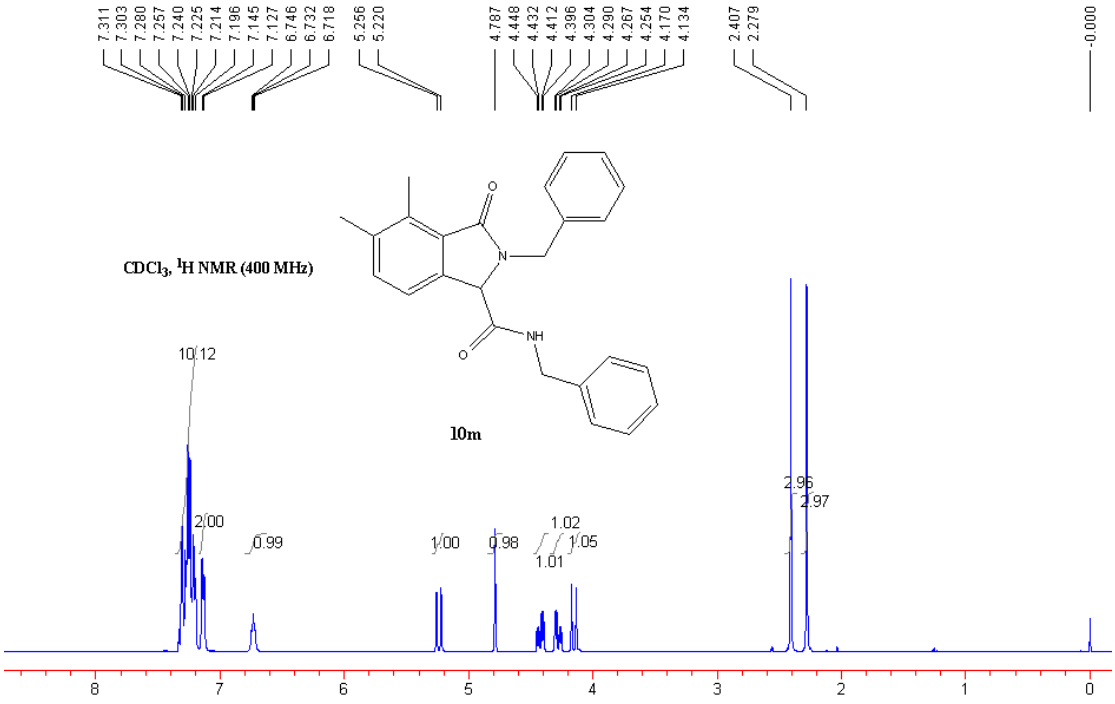
$CDCl_3$, ^1H NMR (400 MHz)

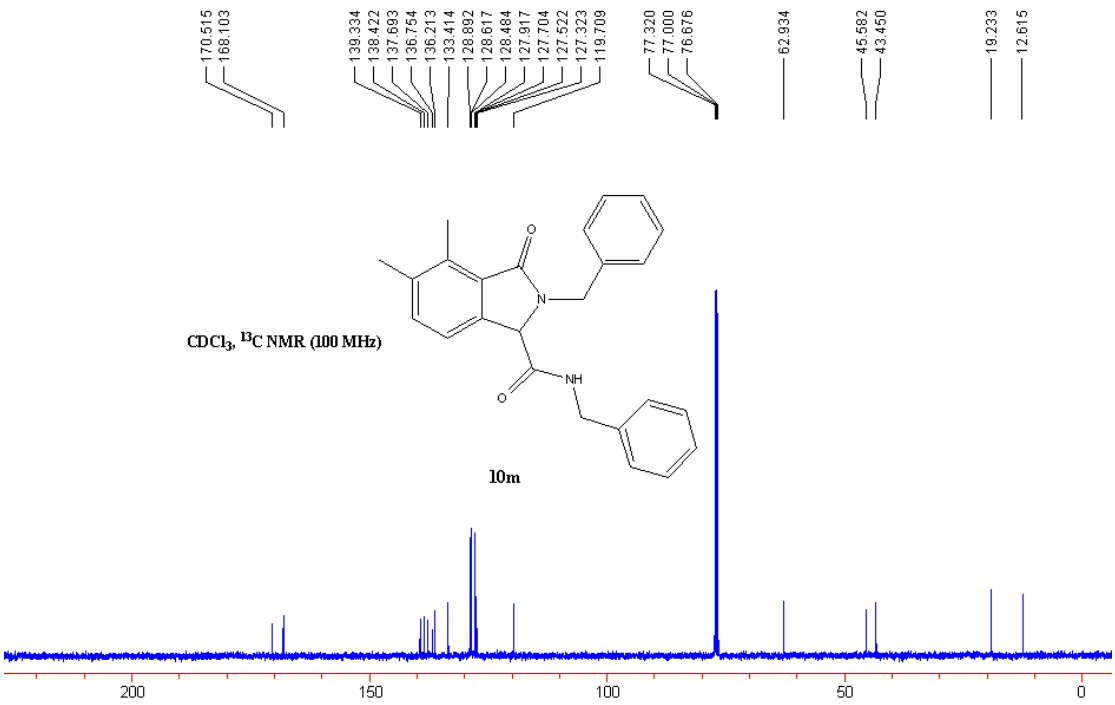
101

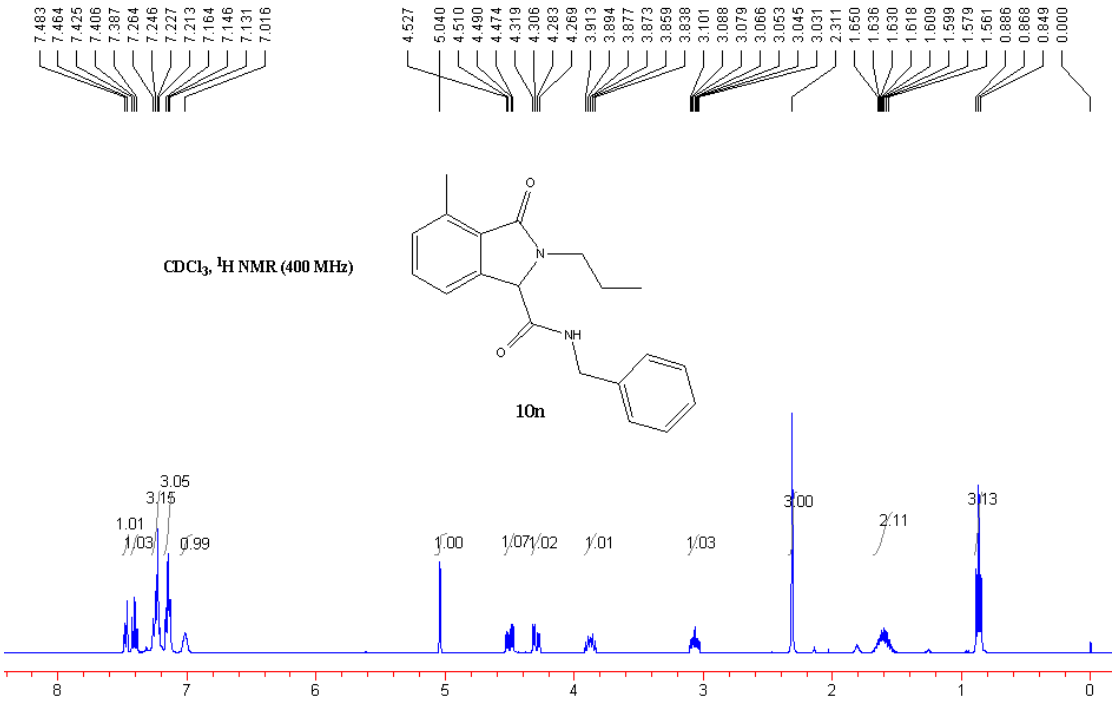


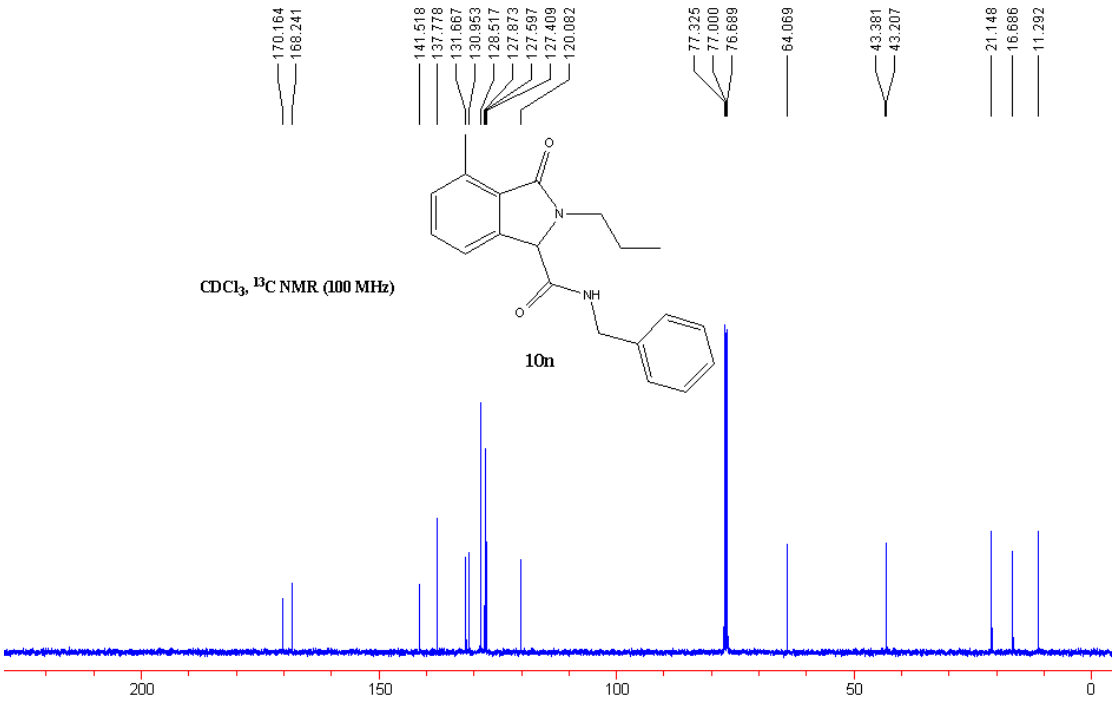
$CDCl_3$, ^{13}C NMR (100 MHz)

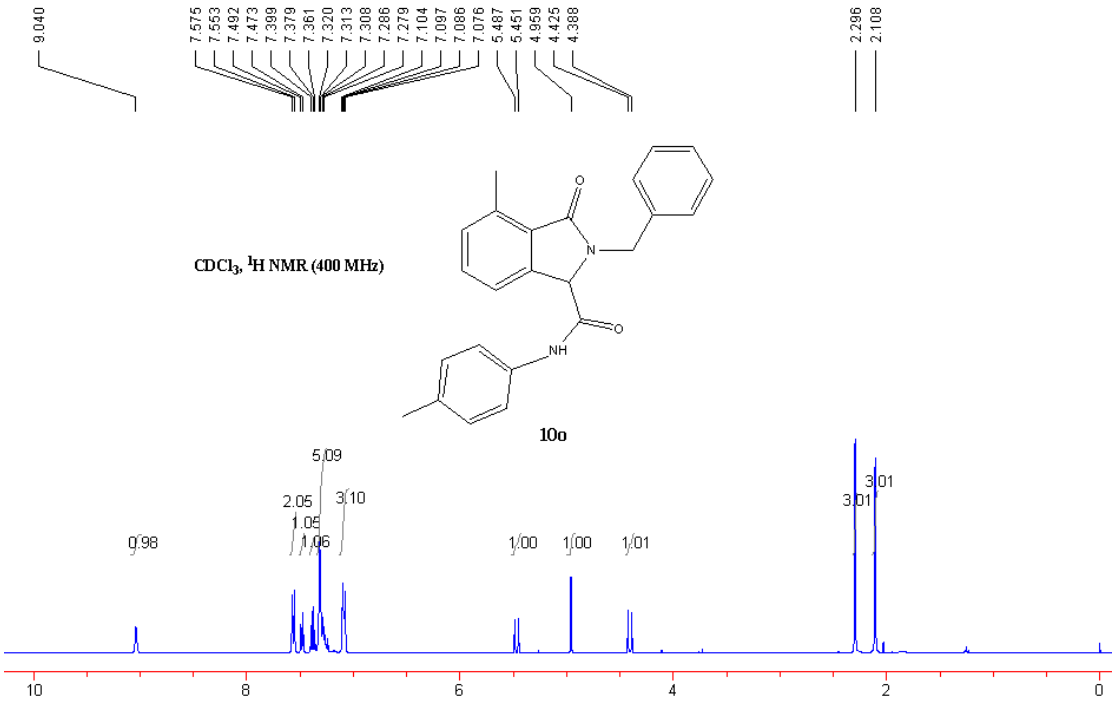
101

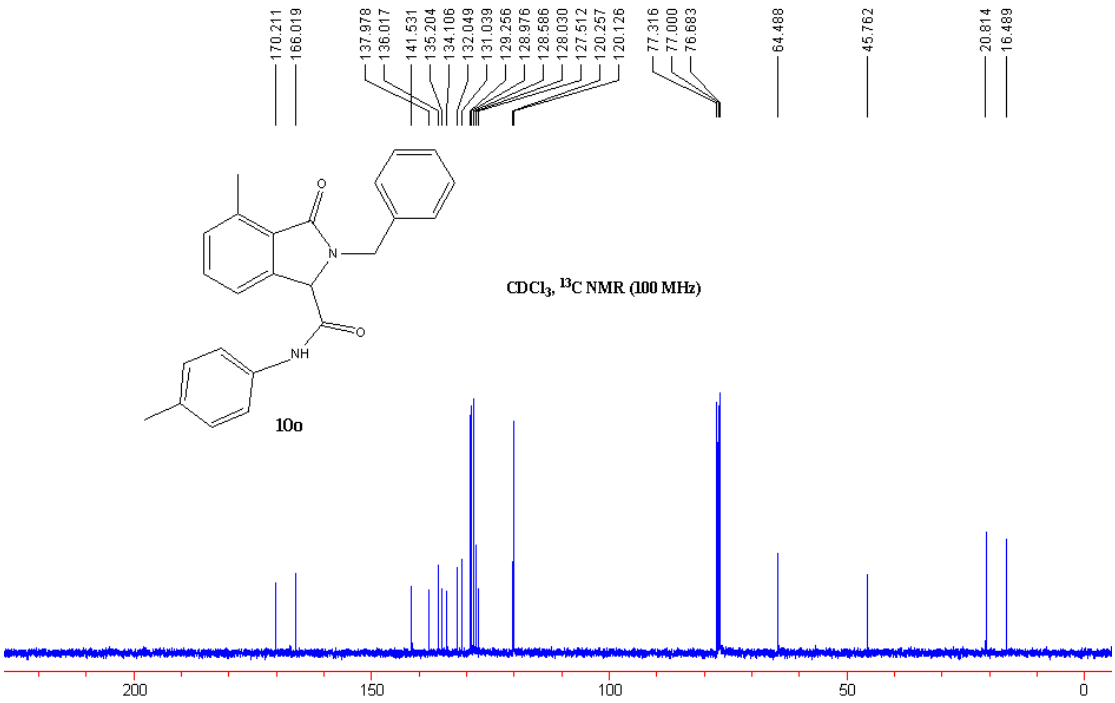

7.311
7.303
7.280
7.257
7.240
7.225
7.214
7.196
7.145
7.127
6.746
6.732
6.718
5.256
5.220
4.787
4.448
4.432
4.412
4.396
4.304
4.290
4.267
4.254
4.170
4.134
2.407
2.279
-0.000
CDCl3, 1H NMR (400 MHz)
O
N
NH
O
10m
10.12
2.00
0.99
1.00
0.98
1.02
1.01
1.05
2.96
2.97
8
7
6
5
4
3
2
1
0


170.515
168.103
139.334
138.422
137.693
136.754
136.213
133.414
128.892
128.617
128.484
127.917
127.704
127.522
127.323
119.709
77.320
77.000
76.676
62.934
45.582
43.450
19.233
12.615
CDCl3, 13C NMR (100 MHz)
O
N
NH
O
10m
200
150
100
50
0


7.483
7.464
7.425
7.406
7.387
7.264
7.246
7.227
7.213
7.164
7.146
7.131
7.016
4.527
5.040
4.510
4.490
4.474
4.319
4.306
4.283
4.269
3.913
3.894
3.877
3.873
3.859
3.838
3.101
3.088
3.079
3.066
3.053
3.045
3.031
2.311
1.650
1.636
1.630
1.618
1.609
1.599
1.579
1.561
0.886
0.868
0.849
0.000
CDCl3, 1H NMR (400 MHz)
O
N
NH
O
10n
3.05
3.15
1.01
1.03
0.99
1.00
1.07
1.02
1.01
1.03
3.00
2.11
3.13
8
7
6
5
4
3
2
1
0


170.164
168.241
141.518
137.778
131.667
130.953
128.517
127.873
127.597
127.409
120.082
77.325
77.000
76.689
64.069
43.381
43.207
21.148
16.686
11.292
CDCl3, 13C NMR (100 MHz)
O
N
NH
O
10n
200
150
100
50
0


9.040
7.575
7.563
7.492
7.473
7.399
7.379
7.361
7.320
7.313
7.308
7.286
7.279
7.104
7.097
7.086
7.076
5.487
5.451
4.959
4.425
4.388
2.296
2.108
CDCl3, 1H NMR (400 MHz)
O
N
O
NH
10o
5.09
2.05
3.10
1.05
1.06
0.98
1.00
1.00
1.01
3.01
3.01
10
8
6
4
2
0


170.211
166.019
137.978
136.017
141.531
135.204
134.106
132.049
131.039
129.256
128.976
128.586
128.030
127.512
120.257
120.126
77.316
77.000
76.683
64.488
45.762
20.814
16.489
CDCl3, 13C NMR (100 MHz)
O
N
O
NH
10o
200
150
100
50
0