SUPPORTING INFORMATION

High Enantioselective and Organocatalytic α-Aminantion of 2-Oxindoles

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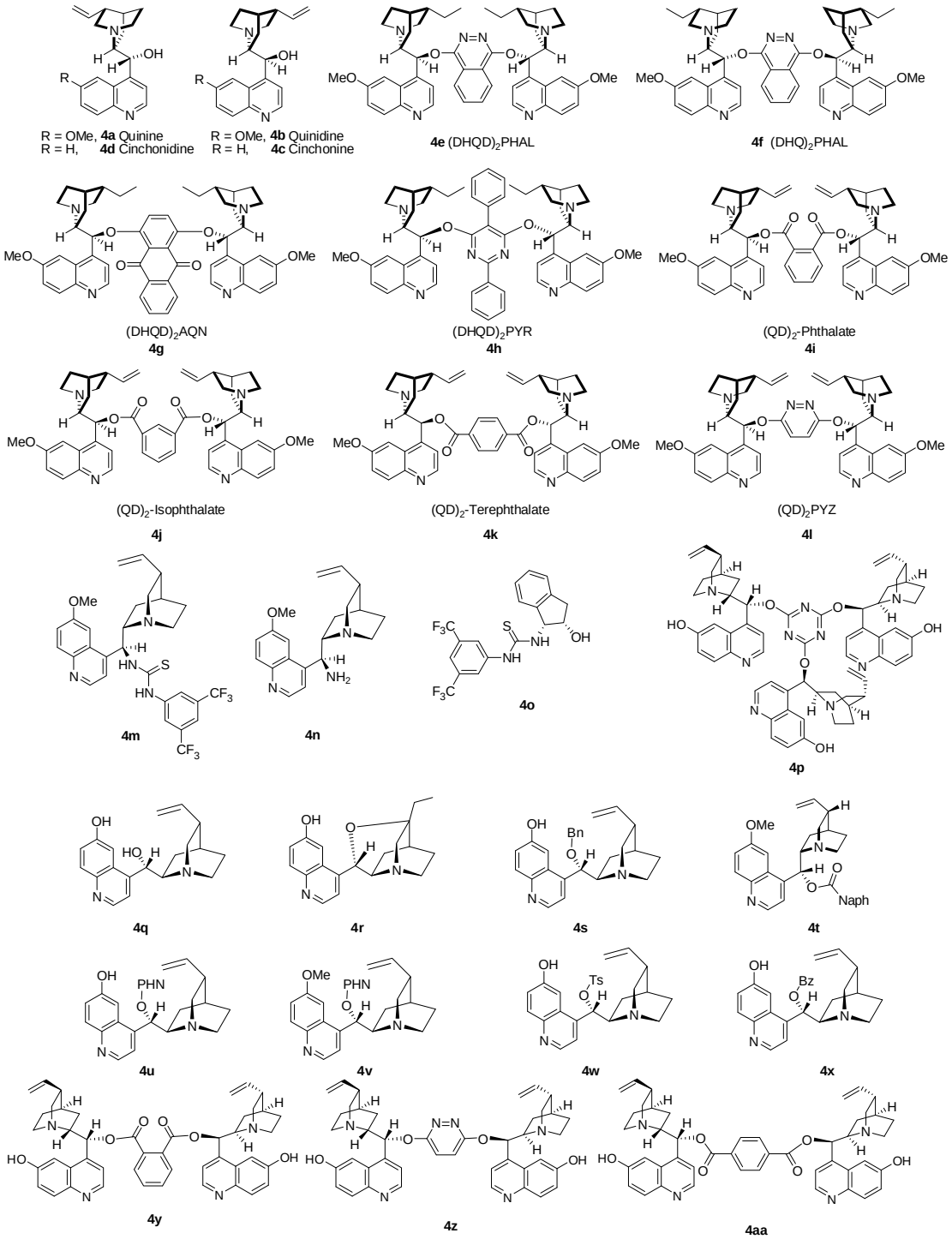
1. GENERAL EXPERIMENTAL

Unless otherwise noted, all reagents were obtained from commercial suppliers and were used without further purification. Tetrahydrofuran, toluene and diethyl ether were distilled from sodium/benzophenone. Dry dichloromethane was distilled over calcium hydride. 1,1,2-Trichloroethane was washed successively with concentrated sulfuric acid, aqueous hydrochloric acid (10%), aqueous sodium bicarbonate (10%) and aqueous sodium chloride (10%), distilled over anhydrous calcium chloride before use.

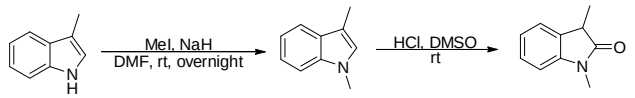
Catalysts for screening



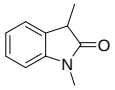
4a-**4h** were purchased commercially and used directly as received. All the other modified cinchona alkaloids catalysts **4i**-**4aa** were prepared according to literature procedures.

2. SYNTHESIS OF STARTING MATERIALS

2.1 Synthesis of oxindole **1c**[1]

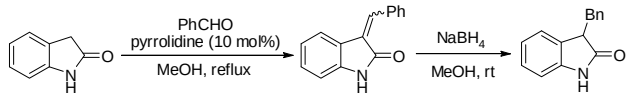


The sodium hydride (72 mg, 3 mmol) was dispersed in anhydrous *N*,*N*-dimethyl formate (5 mL, dried over molecular sieves 3Å) and treated with 3-methyl indole (131 mg, 1 mmol) in portions. The mixture was stirred at room temperature for 1 hour and cooled to 0 ^{o}C, which was added with methyl iodide (excess). The resulting solution was stirred from 0 ^{o}C to room temperature overnight. After quenched by saturated aqueous ammonium chloride, the mixture was extracted with ethyl acetate, dried over anhydrous magnesium sulfate and evaporated to give the product 1,3-dimethylindole, which was dissolved in dimethyl sulfate (5 mL) and treated with concentrated hydrochloric acid (15 mL) dropwise at room temperature. After consummation of the starting material, the reaction was diluted with ethyl acetate and washed with brine, dried over anhydrous sodium sulfate and evaporated to give a residue, which was purified by column chromatography on silica gel (petroleum ether/ethyl acetate 2/1) to afford the 1,3-dimethylindolin-2-one (**1c**) as a yellow oil.



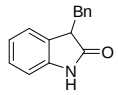
1,3-dimethylindolin-2-one (1c):
Yellow oil. ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.47-1.49 (d, 3H, *J* = 7.5 Hz), 3.21 (s, 3H), 3.40-3.48 (q, 1H, *J* = 7.6 Hz), 6.82-6.84 (d, 1H, *J* = 7.8 Hz), 7.03-7.09 (dt, 1H, *J* = 0.9, 7.5 Hz), 7.23-7.25 (m, 1H), 7.28-7.31 (m, 1H).

2.2 Synthesis of oxindole **1b**[2]

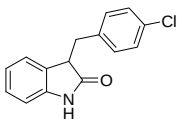


To a solution of 2-oxindole (133 mg, 1 mmol) in methanol was added benzaldehyde (101 mg, 1 mmol) and catalytic amount of pyrrolidine (10 μL). The mixture was heated to reflux for 3 hours and cooled to room temperature. To this yellow solution was added sodium hydroborate (190 mg, 5 mmol) portionably and the color faded and disappeared in 1 hour. The reaction was quenched by water and extracted with ethyl acetate. The organic layer was dried over anhydrous sodium sulfate, evaporated to give a residue which was purified by column chromatography on silica gel (petroleum ether/ethyl acetate 3/1) or recrystalization from ethanol to afford 3-benzylindolin-2-one (**1b**) as a white solid.

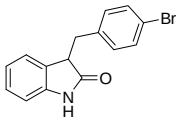
Oxindoles **1e-p** were synthesized analogously, the structures were further established by the X-Ray analysis of the single crystal of 3-(thiophen-2-ylmethyl)-2-oxindole **1o**.



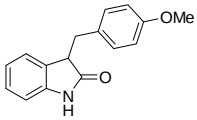
3-benzyl-2-oxindole (1b):
White solid, mp 130-131 °C. (Lit.[3] 131 ^{o}C). ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.92-2.99 (dd, 1H, J = 9.3, 13.8 Hz), 3.45-3.52 (dd, 1H, J = 4.8, 13.8 Hz), 3.72-3.77 (dd, 1H, J = 4.5, 9.0 Hz), 6.75-6.81 (t, 2H, *J* = 8.3 Hz), 6.87-6.92 (t, 1H, *J* = 7.6 Hz), 7.13-7.28 (m, 5H), 7.72 (s, 1H).



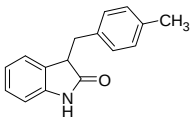
3-(4-chlorobenzyl)-2-oxindole (1e)[4]:
Pale yellow solid, mp 133-134 °C. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.85-2.92 (dd, 1H, *J* = 8.8, 13.7 Hz), 3.27-3.34 (dd, 1H, *J* = 4.4, 13.7 Hz), 3.60-3.64 (dd, 1H, *J* = 4.4, 7.9 Hz), 6.73-6.75 (d, 2H, *J* = 8.0 Hz), 6.81-6.86 (t, 1H, *J* = 7.7 Hz), 6.96-6.99 (d, 2H, *J* = 8.4 Hz), 7.07-7.10 (m, 3H), 9.06 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 35.8, 47.4, 109.9, 122.2, 124.6, 128.2, 128.4, 128.6, 130.8, 132.5, 136.1, 141.5, 179.5. HRMS (ESI): calcd for $C_{15}H_{12}NOCl$ $[M+H]^+$ 258.0680; found 258.0681.



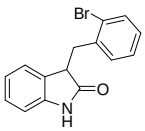
3-(4-bromobenzyl)-2-oxindole (1f)[4]:
White solid, mp 113 °C. FTIR ν_{max} (Film, cm^{-1}): 3220, 1707, 1471, 751. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.94-3.01 (dd, 1H, *J* = 8.5, 14.0 Hz), 3.35-3.41 (dd, 1H, *J* = 4.7, 13.7 Hz), 3.69-3.74 (dd, 1H, *J* = 4.7, 8.5 Hz), 6.81-6.85 (m, 2H), 6.90-6.95 (dt, 1H, *J* = 0.7, 7.5 Hz), 7.00-7.03 (d, 2H, *J* = 8.4 Hz), 7.14-7.19 (t, 1H, *J* = 7.5 Hz), 7.31-7.34 (m, 2H), 8.81 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 35.9, 47.3, 109.9, 120.7, 122.2, 124.7, 128.2, 128.6, 131.2, 132.4, 136.6, 141.5, 179.4. HRMS (ESI): calcd for $C_{15}H_{12}NOBr$ $[M+H]^+$ 302.0175; found 302.0177.



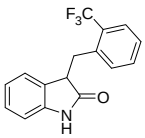
3-(4-methoxybenzyl)-2-oxindole (1g):
Orange solid, mp 110-111 °C. (Lit.[5] 114 °C). FTIR ν_{max} (Film, cm^{-1}): 3379, 1705, 1615, 1512, 1469, 1248, 1178, 1034, 751. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.88-2.96 (dd, 1H, *J* = 8.8, 13.7 Hz), 3.38-3.44 (dd, 1H, *J* = 4.6, 14.1 Hz), 3.68-3.72 (dd, 1H, *J* = 4.6, 8.8 Hz), 3.77 (s, 3H), 6.76-6.83 (m, 4H), 6.88-6.94 (m, 1H), 7.05-7.08 (m, 1H), 7.13-7.21 (m, 2H), 8.08 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 35.8, 47.6, 55.2, 109.5, 113.7, 122.0, 124.9, 127.9, 129.6, 129.7, 130.4, 141.3, 158.4, 179.1. HRMS (EI): calcd for $C_{16}H_{15}NO_2$ $[M]^+$ 253.1103; found 253.1104.



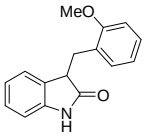
3-(4-methylbenzyl)-2-oxindole (1h)[6]:
White power, mp 146-147 °C. FTIR ν_{max} (Film, cm^{-1}): 3749, 1699, 1541. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.19 (s, 3H), 2.74-2.81 (dd, 1H, *J* = 9.1, 13.6 Hz), 3.31-3.37 (dd, 1H, *J* = 4.0, 13.6 Hz), 3.57-3.62 (dd, 1H, *J* = 4.0, 8.6 Hz), 6.64-6.72 (m, 2H), 6.76-6.81 (1H, *J* = 7.6 Hz), 6.91-7.05 (m, 5H), 9.05 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.1, 36.2, 47.7, 109.9, 122.0, 124.8, 127.9, 129.1, 129.2, 129.3, 134.8, 136.2, 141.7, 180.2. HRMS (ESI): calcd for $C_{16}H_{15}NO$ $[M+H]^+$ 238.1226; found 238.1227.



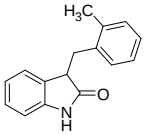
3-(2-bromobenzyl)-2-oxindole (1i):
White power, mp 120-121 °C. FTIR ν_{max} (Film, cm^{-1}): 3228, 1707, 1620, 1469, 749. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.95-3.03 (dd, 1H, *J* = 9.6, 13.4 Hz), 3.58-3.64 (dd, 1H, *J* = 5.8, 13.4 Hz), 3.91-3.96 (dd, 1H, *J* = 5.1, 9.6 Hz), 6.60-6.63 (d, 1H, *J* = 7.0 Hz), 6.87-6.93 (m, 2H), 7.15-7.27 (m, 4H), 7.59-7.62 (d, 1H, *J* = 7.7 Hz), 8.96 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 37.4, 45.3, 109.8, 122.0, 124.9, 125.0, 127.3, 128.0, 128.6, 128.8, 132.0, 133.1, 137.6, 141.4, 179.7. HRMS (ESI): calcd for $C_{15}H_{12}BrNO$ $[M+H]^+$ 302.0175; found 302.0176.



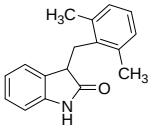
3-[(2-trifluoromethyl)benzyl]-2-oxindole (1j):
White power, mp 132-133 °C. FTIR ν_{max} (Film, cm^{-1}): 3419, 1710, 1313, 1119, 751. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.06-3.14 (dd, 1H, *J* = 9.7, 13.7 Hz), 3.58-3.65 (dd, 1H, *J* = 6.1, 14.6 Hz), 3.79-3.84 (dd, 1H, *J* = 7.0, 8.8 Hz), 6.54-6.58 (t, 1H, *J* = 5.8 Hz), 6.84-6.93 (m, 2H), 7.15-7.21 (m, 1H), 7.36-7.53 (m, 3H), 7.69-7.71 (d, 1H, *J* = 7.6 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 33.6, 46.4, 109.9, 122.1, 122.7, 124.9, 126.3, 126.37, 126.45, 127.0, 128.1, 128.6, 131.7, 132.0, 137.0, 141.4. HRMS (EI): calcd for $C_{16}H_{12}NOF_3$ $[M]^+$ 291.0871; found 291.0874.



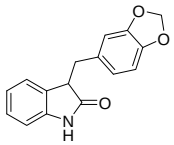
3-(2-methoxybenzyl)-2-oxindole (1k):
Orange solid, mp 110-111 °C. FTIR ν_{max} (Film, cm^{-1}): 3215, 1706, 1620, 1494, 1469, 1338, 1245, 1111, 1031, 751. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.71-2.79 (dd, 1H, *J* = 10.2, 13.4 Hz), 3.57-3.63 (dd, 1H, *J* = 5.1, 13.4 Hz), 3.82 (s, 3H), 3.89-3.94 (dd, 1H, *J* = 5.1, 9.6 Hz), 6.52-6.55 (m, 1H), 6.85-6.89 (m, 3H), 7.09-7.16 (m, 2H), 7.26-7.28 (m, 2H), 8.53 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 32.2, 45.5, 55.2, 109.6, 110.3, 120.2, 121.7, 125.1, 126.7, 127.6, 128.1, 129.7, 131.4, 141.6, 157.9, 180.7. HRMS (EI): calcd for $C_{16}H_{15}NO_2$ $[M]^+$ 253.1103; found 253.1105.



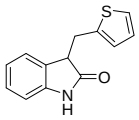
3-(2-methylbenzyl)-2-oxindole (1l):
White power, mp 108-109 °C. (Lit.[7] 108-109 °C). FTIR ν_{max} (Film, cm^{-1}): 3210, 1708, 1620, 1470, 1337, 1230, 1101, 750, 666, 575. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.29 (s, 3H), 2.78-2.86 (dd, 1H, *J* = 11.0, 13.9 Hz), 3.49-3.55 (dd, 1H, *J* = 4.7, 13.9 Hz), 3.69-3.74 (dd, 1H, *J* = 4.7, 11.0 Hz), 6.51-6.54 (d, 1H, *J* = 7.4 Hz), 6.80-6.85 (t, 1H, *J* = 7.4 Hz), 6.91-6.94 (d, 1H, *J* = 7.6 Hz), 7.14-7.22 (m, 5H), 9.46 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 19.7, 34.3, 46.4, 110.0, 122.0, 125.0, 126.0, 126.9, 128.0, 129.3, 130.1, 130.6, 136.6, 136.7, 141.6, 180.5. HRMS (EI): calcd for $C_{16}H_{15}NO$ $[M]^+$ 237.1154; found 237.1156.



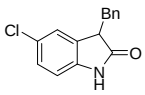
3-(2,6-dimethylbenzyl)-2-oxindole (1m):
White power, mp 134 °C. FTIR ν_{max} (Film, cm^{-1}): 3214, 1707, 1620, 1470, 751. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.22 (s, 3H), 2.29 (s, 3H), 2.72-2.80 (dd, 1H, *J* = 10.9, 13.7 Hz), 3.44-3.50 (dd, 1H, *J* = 4.2, 13.7 Hz), 3.67-3.72 (dd, 1H, *J* = 4.2, 10.9 Hz), 6.51-6.53 (d, 1H, *J* = 7.4 Hz), 6.80-6.85 (t, 1H, *J* = 7.4 Hz), 6.90-6.93 (d, 1H, *J* = 7.7 Hz), 6.98-7.08 (m, 3H), 7.13-7.18 (t, 1H, *J* = 7.7 Hz), 9.43 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 19.2, 21.0, 34.3, 46.5, 109.9, 121.9, 125.0, 127.6, 127.9, 129.3, 130.4, 130.8, 133.5, 135.3, 136.4, 141.6, 180.5. HRMS (ESI): calcd for $C_{17}H_{17}NO$ $[M+H]^+$ 252.1383; found 252.1383.



3-(benzo[*d*][1,3]dioxol-5-ylmethyl)-2-oxindole (1n):
White solid, mp 134-135 °C. (Lit.[5] 138-139 °C). FTIR ν_{max} (Film, cm^{-1}): 3405, 1706, 1488, 1248, 1039, 751. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.86-2.94 (dd, 1H, *J* = 9.2, 13.6 Hz), 3.36-3.42 (dd, 1H, *J* = 4.3, 13.6 Hz), 3.66-3.71 (dd, 1H, *J* = 4.3, 8.6 Hz), 5.92 (s, 2H), 6.59-6.70 (m, 3H), 6.80-6.85 (t, 2H, *J* = 7.4 Hz), 6.91-6.96 (t, 1H, *J* = 7.4 Hz), 7.15-7.20 (t, 1H, *J* = 7.4 Hz), 7.86 (br s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 36.3, 47.8, 100.9, 108.1, 109.6, 110.0, 122.0, 122.5, 124.8, 128.0, 129.0, 131.5, 141.7, 146.3, 147.6, 180.1. HRMS (EI): calcd for $C_{16}H_{13}NO_3$ $[M]^+$ 267.0895; found 267.0898.

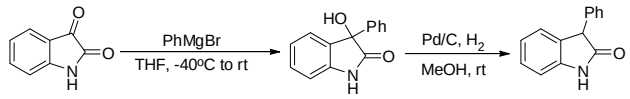


3-(thiophen-2-ylmethyl)-2-oxindole (1o)[8]:
White solid, mp 156 °C. FTIR ν_{max} (Film, cm^{-1}): 3212, 1706, 1621, 1470, 1337, 1225, 751, 698. ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.27-3.35 (dd, 1H, *J* = 8.0, 14.8 Hz), 3.55-3.61 (dd, 1H, *J* = 3.7, 14.2 Hz), 3.70-3.75 (dd, 1H, *J* = 4.3, 8.0 Hz), 6.77 (m, 1H), 6.82-6.85 (m, 2H), 6.93-6.95 (m, 2H), 7.05-7.07 (m, 1H), 7.15-7.18 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 28.5, 45.5, 107.8, 120.1, 122.1, 122.5, 124.2, 124.5, 126.1, 126.4, 137.5, 139.6, 177.2. HRMS (ESI): calcd for $C_{13}H_{11}NOS$ $[M+H]^+$ 230.0634; found 230.0636.



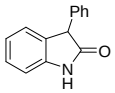
3-benzyl-5-chloro-2-oxindole (1p):
Pale yellow power, mp 112-113 °C. FTIR ν_{max} (Film, cm^{-1}): 3193, 1710, 1619, 1476, 1229, 815, 699. ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.91-2.98 (dd, 1H, *J* = 8.5, 13.2 Hz), 3.43-3.49 (dd, 1H, *J* = 4.6, 13.9 Hz), 3.71-3.76 (dd, 1H, *J* = 4.6, 9.3 Hz), 6.72-6.78 (m, 2H), 7.13-7.18 (m, 3H), 7.23-7.27 (m, 3H), 9.08 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 36.4, 47.7, 110.7, 125.2, 127.0, 127.4, 128.0, 128.5, 129.3, 130.7, 137.2, 140.1, 179.5. HRMS (ESI): calcd for $C_{15}H_{12}NOCl$ $[M+H]^+$ 258.0680; found 258.0681.

2.3 Synthesis of oxindole **1r**[9]



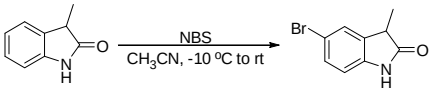
A solution of phenylmagnesium bromide (2-3 eq.) prepared *in situ* from phenyl bromide and magnesium in anhydrous diethyl ether at room temperature was added to a stirred cold (-30 ^{o}C) solution of isatin (1.0 eq.) in anhydrous tetrahydrofuran under nitrogen. The mixture was allowed to warm to room temperature and stirred for overnight. The reaction solution was quenched by saturated aqueous ammonium chloride, followed by extraction with diethyl ether. The organic phase was collected and washed with brine, dried over anhydrous sodium sulfate and evaporated to give a red residue, which was purified by column chromatography on silica gel (petroleum ether/ethyl acetate 3/1) to afford the 3-hydroxy-3-phenylindolin-2-one as an orange solid.

To the solution of 3-hydroxy-3-phenylindolin-2-one (1.0 eq.) in methanol was added palladium/carbon (10 %), the reaction was stirred under hydrogen atmosphere (balloon) for 24 hours at room temperature. After filtration, the solution was evaporated to give a yellow residue, which was purified by column chromatography on silica gel (petroleum ether: ethyl acetate = 3:1) to afford the 3-phenylindolin-2-one (**1r**) as a pale yellow solid.

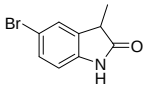


3-phenylindolin-2-one (1r):
Pale yellow solid, mp 182-183 °C. (Lit.[10] 185-187 ^{o}C). ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.63 (s, 1H), 6.92-6.95 (d, 1H, *J* = 7.7 Hz), 7.01-7.07 (t, 1H, *J* = 7.5 Hz), 7.12-7.15 (d, 1H, *J* = 7.4 Hz), 7.21-7.23 (m, 2H), 7.29-7.37 (m, 4H), 8.03 (br s, 1H). HRMS (ESI): calcd for $C_{14}H_{11}NO$ $[M+H]^+$ 210.0913; found 210.0914.

2.4 Synthesis of oxindole **1q**[11]

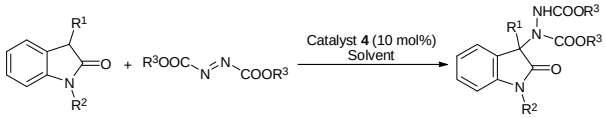


To a solution of 3-methyl-2-oxindole (133 mg, 1 mmol) in acetonitrile at -10 ^{o}C was added NBS (1 mmol). The mixture was stirred at -10 ^{o}C for 1 hour and then warmed to room temperature. The precipitate was collected and washed with water, dried to afford the product 5-bromo-3-methylindolin-2-one (**1q**) as a white solid.



5-bromo-3-methylindolin-2-one (1q):
White solid, mp 186-187 ^{o}C. (Lit.[11b] 183-186 ^{o}C). ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.48-1.51 (d, 3H, *J* = 7.7 Hz), 3.43-3.51 (q, 1H, *J* = 7.7 Hz), 6.76-6.79 (d, 1H, *J* = 8.7 Hz), 7.34-7.35 (m, 2H), 8.37 (s, 1H).

3. OPTIMISED GENERAL PROCEDURE



1a R^1 = Me, R^2 = H
1b R^1 = Bn, R^2 = H
1c R^1 = Me, R^2 = Me
1d R^1 = Me, R^2 = Boc

2a R^3 = Et
2b R^3 = Pr-*i*
2c R^3 = Bu-*t*
2d R^3 = Bn

3a R^1 = Me, R^2 = H, R^3 = Et
3b R^1 = Me, R^2 = H, R^3 = Pr-*i*
3c R^1 = Me, R^2 = H, R^3 = Bu-*t*
3d R^1 = Me, R^2 = H, R^3 = Bn
3e R^1 = Bn, R^2 = H, R^3 = Pr-*i*
3t R^1 = Me, R^2 = Me, R^3 = Pr-*i*
3u R^1 = Me, R^2 = Boc, R^3 = Pr-*i*

3.1 Optimization of the reaction conditions:[a]

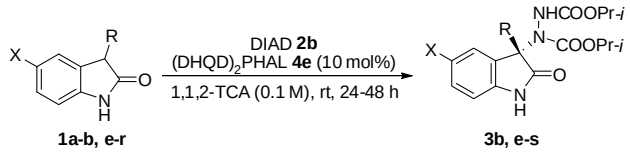
Entry	**1**	**2**	**4**	Solvent	**3**	Yield (%)[b]	ee (%)[c]
1	**1a**	**2a**	**4a**	CH_2Cl_2	**3a**	45	7
2	**1a**	**2a**	**4c**	CH_2Cl_2	**3a**	50	1
3	**1a**	**2a**	**4d**	CH_2Cl_2	**3a**	69	7
4	**1a**	**2a**	**4b**	CH_2Cl_2	**3a**	63	5
5	**1a**	**2a**	**4q**	CH_2Cl_2	**3a**	52	3
6	**1a**	**2a**	**4r**	CH_2Cl_2	**3a**	59	5
7	**1a**	**2a**	**4s**	CH_2Cl_2	**3a**	31	35
8	**1a**	**2a**	**4t**	CH_2Cl_2	**3a**	trace	-
9	**1a**	**2a**	**4u**	CH_2Cl_2	**3a**	39	6
10	**1a**	**2a**	**4v**	CH_2Cl_2	**3a**	50	21
11	**1a**	**2a**	**4w**	CH_2Cl_2	**3a**	48	11
12	**1a**	**2a**	**4x**	CH_2Cl_2	**3a**	31	7
13	**1a**	**2a**	**4m**	CH_2Cl_2	**3a**	31	23
14	**1a**	**2a**	**4n**	CH_2Cl_2	**3a**	44	35
15	**1a**	**2a**	**4o**	CH_2Cl_2	**3a**	31	3
16	**1a**	**2a**	**4e**	CH_2Cl_2	**3a**	47	33
17	**1a**	**2a**	**4f**	CH_2Cl_2	**3a**	48	31
18	**1a**	**2a**	**4z**	CH_2Cl_2	**3a**	69	6
19	**1a**	**2a**	**4aa**	CH_2Cl_2	**3a**	nd[e]	8
20	**1a**	**2a**	**4y**	CH_2Cl_2	**3a**	44	10
21	**1a**	**2a**	**4p**	CH_2Cl_2	**3a**	nd[e]	6
22	**1a**	**2a**	**4g**	CH_2Cl_2	**3a**	17	1
23	**1a**	**2a**	**4h**	CH_2Cl_2	**3a**	13	40
24	**1a**	**2a**	**4i**	CH_2Cl_2	**3a**	55	20
25	**1a**	**2a**	**4j**	CH_2Cl_2	**3a**	32	23
26	**1a**	**2a**	**4k**	CH_2Cl_2	**3a**	11	25
27	**1a**	**2a**	**4l**	CH_2Cl_2	**3a**	11	13
28	**1a**	**2b**	**4e**	CH_2Cl_2	**3b**	100	57
29	**1a**	**2c**	**4e**	CH_2Cl_2	**3c**	85	39
30	**1a**	**2d**	**4e**	CH_2Cl_2	**3d**	58	<10
31	**1a**	**2b**	**4e**	CH_3CN	**3b**	100	33
32	**1a**	**2b**	**4e**	THF	**3b**	100	43
33	**1a**	**2b**	**4e**	Et_2O	**3b**	99	39
34	**1a**	**2b**	**4e**	Dioxane	**3b**	100	60
35	**1a**	**2b**	**4e**	Toluene	**3b**	100	55
36	**1a**	**2b**	**4e**	CH_3OH	**3b**	77	11
37	**1a**	**2b**	**4e**	DMF	**3b**	100	11
38	**1a**	**2b**	**4e**	DMSO	**3b**	98	9
39	**1a**	**2b**	**4e**	CH_3COOEt	**3b**	99	39
40	**1a**	**2b**	**4e**	CH_3COCH_3	**3b**	100	26
41	**1a**	**2b**	**4e**	$CHCl_3$	**3b**	100	61
42	**1a**	**2b**	**4e**	DCE	**3b**	100	62

Entry	**1**	**2**	**4**	Solvent	**3**	Yield (%)[b]	ee (%)[c]
43	**1a**	**2b**	**4e**	CCl_4	**3b**	97	50
44	**1a**	**2b**	**4e**	1,1,2-TCA	**3b**	100	70
45[f]	**1a**	**2b**	**4e**	1,1,2-TCA	**3b**	100	68
46[g]	**1a**	**2b**	**4e**	1,1,2-TCA	**3b**	84	67
47	**1a**	**2b**	**4g**	1,1,2-TCA	**3b**	99	5
48	**1a**	**2b**	**4h**	1,1,2-TCA	**3b**	100	44
49	**1a**	**2b**	**4e**	1,1,2,2-TCE	**3b**	96	69
50	**1a**	**2b**	**4e**	$Cl_2C{=}CCl_2$	**3b**	100	43
51	**1a**	**2b**	**4e**	$ClCH{=}CCl_2$	**3b**	100	47
52	**1a**	**2b**	**4e**	PhCl	**3b**	100	56
53[d]	**1a**	**2b**	**4e**	1,1,2-TCA	**3b**	100	78
54[d]	**1a**	**2b**	**4e**	*i*-PrOH	**3b**	nd[e]	63
55[d]	**1a**	**2b**	**4e**	CH_3COOPr-*i*	**3b**	80	55
56[d]	**1a**	**2b**	**4e**	CH_3NO_2	**3b**	<10	52
57[d]	**1a**	**2b**	**4e**	H_2O	**3b**	nd[e]	27
58[d]	**1a**	**2b**	**4e**	PEG-400	**3b**	99	1
59[d]	**1a**	**2b**	**4e**	*n*-hexane	**3b**	trace	-
60[d]	**1a**	**2b**	**4e**	1,1,1-TCA	**3b**	90	75
61[d]	**1a**	**2b**	**4e**	1,1,2-TCA+*i*-PrOH (3:1)	**3b**	65	78
62[d]	**1a**	**2b**	**4e**	1,1,2-TCA +*i*-PrOH (1:1)	**3b**	nd[e]	76
63[d]	**1a**	**2b**	**4e**	1,1,2-TCA +*t*-BuOH (3:1)	**3b**	<50	83
64[d]	**1c**	**2b**	**4e**	1,1,2-TCA	**3t**	<20	52
65[d]	**1d**	**2b**	**4e**	1,1,2-TCA	**3u**	trace	-
66[d]	**1b**	**2b**	**4e**	CH_2Cl_2	**3e**	94	89
67[d]	**1b**	**2b**	**4e**	Et_2O	**3e**	83	53
68[d]	**1b**	**2b**	**4e**	DCE	**3e**	88	90
69[d]	**1b**	**2b**	**4e**	1,1,1-TCE	**3e**	74	86
70[d]	**1b**	**2b**	**4e**	1,1,2-TCA	**3g**	99	93
71[d]	**1b**	**2b**	**4f**	1,1,2-TCA	**3e**	99	-91
72[d]	**1b**	**2b**	**4e**	1,1,2,2-TCE	**3e**	35	86
73[d]	**1b**	**2b**	**4e**	1,1,2-TCA +*i*-PrOH (3:1)	**3e**	nd[e]	91
74[d]	**1b**	**2b**	**4e**	1,1,2-TCA +*t*-BuOH (3:1)	**3e**	<10	nd[e]
75[d,h]	**1b**	**2b**	**4e**	1,1,2-TCA	**3e**	95	91
76[d,i]	**1b**	**2b**	**4e**	1,1,2-TCA	**3e**	90	90
77[d,j]	**1b**	**2b**	**4e**	1,1,2-TCA	**3e**	83	79

[a] Reaction conditions (unless otherwise noted): **1** (0.2 mmol), **2** (0.2 mmol), solvent (0.2 mL, 1M), catalyst (10 mmol%), room temperature. [b] Isolated yields. [c] Determined by chiral HPLC analysis. [d] Solvent (2 mL, 0.1 M). [e] nd: not determined. [f] 4Å molecular sieves was added. [g] Reaction performed at 0 °C. [h] Catalyst loading: 5 mol%. [i] Catalyst loading: 2 mol%. [j] Catalyst loading: 1 mol%.

DCE: 1,2-dichloroethane. 1,1,1-TCA: 1,1,1-trichloroethane. 1,1,2-TCA: 1,1,2-trichloroethane. 1,1,2,2-TCE: 1,1,2,2-tetrachloroethane.

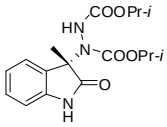
3.2 General procedure:



A mixture of 3-methyl-2-oxindole **1a** (30 mg, 0.2 mmol) and DIAD **2b** (40 mg, 0.2 mmol) in 1,1,2-trichloroethane (2 mL) at room temperature was added the catalyst $(DHQD)_2PHAL$ **4e** (15 mg, 0.02 mmol). The resulting mixture was stirred for 48 hours and directly purified by column chromatography on silica gel (petroleum ether/ethyl acetate 1/1) to afford the (*S*)-diisopropyl 1-(3-methyl-2-oxoindolin-3-yl)hydrazine-1,2-dicarboxylate **3b** (81 mg, yield 99%) as a white solid.

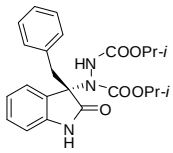
Adducts **3e-3s** were synthesized analogously, the structures were confirmed by the X-Ray analysis of the single crystal of racemic adduct **3f**, the absolute configure of the newly formed chiral center was further established by the X-Ray analysis of the single crystal of enantiopure adduct **3r**.

3.2.1 Characterization of compound **3b**



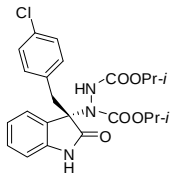
(*S*)-diisopropyl 1-(3-methyl-2-oxoindolin-3-yl)hydrazine-1,2-dicarboxylate (3b): White power, mp 84-86 °C. $[\alpha]_D^{20}$ +6.0 (*c* 0.5, $CHCl_3$). FTIR ν_{max} (Film, cm^{-1}): 3734, 1716, 1508, 829, 602. ^1H NMR (300 MHz, $CDCl_3$) δ 0.90 (s, 3H), 1.06-1.09 (d, 3H, *J* = 5.7 Hz), 1.32-1.34 (d, 6H, *J* = 6.2 Hz), 1.55 (s, 3H), 4.71-4.77 (m, 1H), 5.01-5.09 (m, 1H), 6.84-6.86 (d, 1H, *J* = 7.3 Hz), 7.00-7.05 (t, 1H, *J* = 7.3 Hz), 7.15-7.27 (m, 2H), 7.77-7.79 (d, 1H, *J* = 7.3 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.4, 21.6, 21.9, 22.0, 23.1, 66.3, 70.0, 70.7, 109.8, 123.0, 124.2, 128.4, 133.0, 139.6, 153.8, 156.9, 180.0. HRMS (ESI): calcd for $C_{17}H_{23}N_3O_5$ $[M+H]^+$ 350.1710; found 350.1712. HPLC (Chiralcel AD-H, 90:10 *n*-hexane/2-propanol, 25 °C, 1 mL·min^{-1}) t_R = 13.4 min (minor), 18.0 min (major).

3.2.2 Characterization of compound **3e**



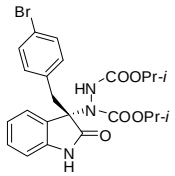
(*S*)-diisopropyl 1-(3-benzyl-2-oxoindolin-3-yl)hydrazine-1,2-dicarboxylate (3e): White solid, mp 176-177 °C. $[\alpha]_D^{20}$ +41.2 (*c* 1.0, $CHCl_3$). FTIR ν_{max} (Film, cm^{-1}): 3272, 2979, 1726, 1623, 1473, 1383, 1250, 1107, 754. ^1H NMR (300 MHz, $CDCl_3$) δ 0.91 (s, 3H), 1.05-1.07 (d, 3H, *J* = 6.0 Hz), 1.35-1.37 (d, 6H, *J* = 6.4 Hz), 3.14-3.18 (d, 1H, *J* = 12.0 Hz), 3.23-3.27 (d, 1H, *J* = 12.0 Hz), 4.70-4.74 (m, 1H), 5.08-5.12 (m, 1H), 6.45-6.46 (m, 1H), 6.66-6.68 (m, 2H), 6.95 (m, 3H), 7.07-7.08 (m, 2H), 7.46-7.53 (m, 1H), 7.93-7.96 (m, 1H), 8.32 (br, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.5, 21.6, 22.0, 22.1, 41.8, 70.1, 70.4, 70.8, 109.4, 122.7, 125.0, 126.8, 127.4, 128.6, 130.5, 130.6, 132.7, 140.6, 153.8, 157.2, 178.2. HRMS (EI): calcd for $C_{23}H_{27}N_3O_5$ $[M]^+$ 425.1951; found 425.1954. HPLC (Sino-Chiral OD, 90:10 *n*-hexane/2-propanol, 25 °C, 1 mL·min^{-1}) t_R = 6.5 min (minor), 5.0 min (major).

3.2.3 Characterization of compound **3f**



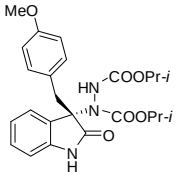
(*S*)-diisopropyl-1-(3-(4-chlorobenzyl)-2-oxoindolin-3-yl)hydrazine-1,2-dicarboxylate (3f):
White solid, mp 119-120 °C. $[\alpha]_D^{20}$ +60.4 (*c* 0.6, $CHCl_3$). FTIR ν_{max} (Film, cm^{-1}): 3272, 2981, 1726, 1622, 1473, 1384, 1250, 1180, 1107, 754. ^1H NMR (300 MHz, $CDCl_3$) δ 0.93 (m, 3H), 1.07-1.09 (d, 3H, *J* = 6.0 Hz), 1.35-1.37 (d, 6H, *J* = 6.0 Hz), 3.11-3.15 (d, 1H, *J* = 11.9 Hz), 3.22-3.26 (d, 1H, *J* = 11.9 Hz), 4.70-4.78 (m, 1H), 5.05-5.13 (m, 1H), 6.50-6.52 (d, 1H, *J* = 7.7 Hz), 6.60-6.62 (d, 2H, *J* = 8.5 Hz), 6.92-6.94 (d, 2H, *J* = 8.5 Hz), 7.07-7.16 (m, 2H), 7.27-7.28 (d, 1H, *J* = 4.3 Hz), 7.81 (s, 1H), 7.92-7.94 (d, 1H, *J* = 6.8 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.5, 21.6, 22.0, 22.1, 41.1, 70.0, 70.3, 71.0, 109.4, 123.0, 125.0, 127.7, 128.9, 130.0, 131.2, 131.7, 133.0, 140.3, 153.7, 157.1, 177.6. HRMS (ESI): calcd for $C_{23}H_{26}ClN_3O_5$ $[M+H]^+$ 460.1634; found 460.1637. HPLC (Chiralcel OD-H, 90:10 *n*-hexane/2-propanol, 10 °C, 1 mL·min^{-1}) t_R = 8.4 min (minor), 4.8 min (major).

3.2.4 Characterization of compound **3g**



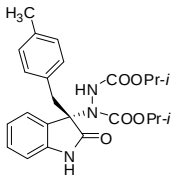
(*S*)-diisopropyl-1-(3-(4-bromobenzyl)-2-oxoindolin-3-yl)hydrazine-1,2-dicarboxylate (3g):
Yellow solid, mp 109-110 °C. $[\alpha]_D^{20}$ +54.7 (*c* 1.0, $CHCl_3$). FTIR ν_{max} (Film, cm^{-1}): 3276, 1724, 1473, 1384, 1249, 1106, 754. ^1H NMR (300 MHz, $CDCl_3$) δ 0.91-0.93 (d, 3H, *J* = 5.3 Hz), 1.06-1.08 (d, 3H, *J* = 6.0 Hz), 1.34-1.36 (d, 6H, *J* = 6.3 Hz), 3.10-3.14 (d, 1H, *J* = 12.3 Hz), 3.26-3.30 (d, 1H, *J* = 11.9 Hz), 4.68-4.76 (m, 1H), 5.04-5.12 (m, 1H), 6.47-6.50 (d, 1H, *J* = 7.0 Hz), 6.57-6.60 (d, 2H, *J* = 8.5 Hz), 7.09-7.12 (m, 4H), 7.31 (s, 1H), 7.89 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.5, 21.6, 21.9, 22.1, 41.2, 70.0, 70.3, 70.9, 109.4, 121.1, 122.9, 124.9, 128.9, 130.0, 130.6, 131.8, 132.1, 140.3, 153.7, 157.1, 177.8. HRMS (EI): calcd for $C_{23}H_{26}N_3O_5Br$ $[M]^+$ 503.1056; found 503.1053. HPLC (Chiralcel OD-H, 90:10 *n*-hexane/2-propanol, 15 °C, 1 mL·min^{-1}) t_R = 7.4 min (minor), 4.7 min (major).

3.2.5 Characterization of compound **3h**



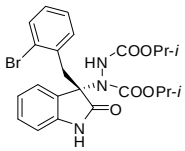
(*S*)-diisopropyl-1-(3-(4-methoxybenzyl)-2-oxoindolin-3-yl)hydrazine-1,2-dicarboxylate (3h):
White solid, mp 97-99 °C. $[\alpha]_D^{20}$ +50.5 (*c* 1.0, $CHCl_3$). FTIR ν_{max} (Film, cm^{-1}): 3435, 1626, 1384, 1251, 1107, 578. ^1H NMR (300 MHz, $CDCl_3$) δ 0.85-0.87 (d, 3H, *J* = 4.5 Hz), 1.00-1.02 (d, 3H, *J* = 6.3 Hz), 1.27-1.30 (d, 6H, *J* = 6.3 Hz), 3.02-3.06 (d, 1H, *J* = 11.8 Hz), 3.13-3.17 (d, 1H, *J* = 12.7 Hz), 3.52 (s, 3H), 4.63-4.71 (m, 1H), 4.98-5.06 (m, 1H), 6.40-6.43 (d, 3H, *J* = 8.2 Hz), 6.51-6.53 (d, 2H, *J* = 9.1 Hz), 7.01-7.12 (m, 3H), 7.45 (s, 1H), 7.85-7.87 (d, 1H, *J* = 6.3 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.5, 21.6, 21.9, 22.1, 41.0, 54.9, 70.2, 70.8, 109.2, 112.9, 122.8, 124.5, 125.0, 128.6, 130.5, 131.4, 140.4, 153.8, 157.1, 158.4, 178.0. HRMS (ESI): calcd for $C_{24}H_{29}N_3O_6$ $[M+H]^+$ 456.2129; found 456.2127. HPLC (Chiralcel OD-H, 90:10 *n*-hexane/2-propanol, 10 °C, 1 mL·min^{-1}) t_R = 9.6 min (minor), 5.7 min (major).

3.2.6 Characterization of compound **3i**



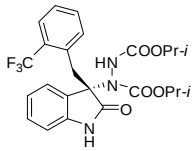
(*S*)-diisopropyl-1-(3-(4-methylbenzyl)-2-oxoindolin-3-yl)hydrazine-1,2-dicarboxylate (3i):
White solid, mp 114-116 °C. $[\alpha]_D^{20}$ +53.2 (*c* 1.0, $CHCl_3$). FTIR ν_{max} (Film, cm^{-1}): 3435, 1642, 608. ^1H NMR (300 MHz, $CDCl_3$) δ 0.83-0.85 (d, 3H, *J* = 5.2 Hz), 0.99-1.01 (d, 3H, *J* = 6.2 Hz), 1.28-1.30 (d, 6H, *J* = 6.2 Hz), 1.98 (s, 3H), 3.02-3.06 (d, 1H, *J* = 12.0 Hz), 3.10-3.14 (d, 1H, *J* = 12.3 Hz), 4.61-4.64 (m, 1H), 4.99-5.07 (m, 1H), 6.39-6.47 (m, 3H), 6.64-6.67 (d, 2H, *J* = 7.8 Hz), 7.01-7.03 (m, 2H), 7.37 (s, 1H), 7.86-7.88 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 20.8, 21.5, 21.6, 22.0, 22.1, 41.4, 70.1, 70.3, 70.8, 109.3, 122.7, 125.0, 128.2, 128.6, 129.5, 130.3, 130.6, 136.3, 140.5, 153.8, 157.1, 178.1. HRMS (EI): calcd for $C_{24}H_{29}N_3O_5$ $[M]^+$ 439.2107; found 439.2111. HPLC (Chiralcel OD-H, 90:10 *n*-hexane/2-propanol, 10 °C, 1 mL·min^{-1}) t_R = 6.8 min (minor), 4.8 min (major).

3.2.7 Characterization of compound **3j**



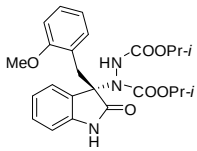
(*S*)-diisopropyl-1-(3-(2-bromobenzyl)-2-oxoindolin-3-yl)hydrazine-1,2-dicarboxylate (3j):
White solid, mp 172-173 °C. $[\alpha]_D^{20}$ +2.2 (*c* 1.0, $CHCl_3$). FTIR ν_{max} (Film, cm^{-1}): 3278, 1726, 1473, 1384, 1248, 1107, 752. ^1H NMR (300 MHz, $CDCl_3$) δ 0.94-0.96 (d, 3H, *J* = 7.0 Hz), 1.08-1.10 (d, 3H, *J* = 6.4 Hz), 1.36-1.38 (d, 6H, *J* = 6.4 Hz), 3.49-3.53 (d, 1H, *J* = 12.8 Hz), 3.61-3.66 (d, 1H, *J* = 12.1 Hz), 4.70-4.79 (m, 1H), 5.05-5.13 (m, 1H), 6.46-6.49 (d, 1H, *J* = 7.7 Hz), 6.90-7.10 (m, 7H), 7.29-7.31 (m, 2H), 8.01-8.03 (d, 1H, *J* = 7.0 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.5, 21.7, 22.0, 22.1, 39.8, 69.8, 70.4, 70.9, 108.7, 122.5, 126.1, 126.46, 126.52, 128.6, 128.8, 129.2, 131.7, 132.6, 132.8, 140.0, 153.6, 156.9, 177.7. HRMS (ESI): calcd for $C_{23}H_{26}BrN_3O_5$ $[M+H]^+$ 504.1129; found 504.1138. HPLC (Chiralcel OD-H, 90:10 *n*-hexane/2-propanol, 10 °C, 1 mL·min^{-1}) t_R = 16.6 min (minor), 6.8 min (major).

3.2.8 Characterization of compound **3k**



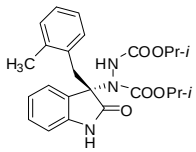
(*S*)-diisopropyl-1-(2-oxo-3-(2-(trifluoromethyl)benzyl)indolin-3-yl)hydrazine-1,2-dicarboxylate (3k):
White solid, mp 105-106 °C. $[\alpha]_D^{20}$ +27.6 (*c* 3.9, $CHCl_3$). FTIR ν_{max} (Film, cm^{-1}): 3275, 1727, 1473, 1385, 1310, 1109, 1040, 767. ^1H NMR (300 MHz, $CDCl_3$) δ 0.83-0.84 (d, 3H, *J* = 6.0 Hz), 0.97-0.99 (d, 3H, *J* = 6.0 Hz), 1.27-1.29 (d, 6H, *J* = 6.6 Hz), 3.43-3.47 (d, 1H, *J* = 13.8 Hz), 3.56-3.61 (d, 1H, *J* = 13.8 Hz), 4.63-4.67 (m, 1H), 4.99-5.05 (m, 1H), 6.41-6.43 (d, 1H, *J* = 6.9 Hz), 6.91-6.95 (m, 2H), 7.04-7.06 (d, 3H, *J* = 7.7 Hz), 7.23 (s, 1H), 7.31-7.33 (d, 1H, *J* = 7.7 Hz), 7.79-7.81 (d, 1H, *J* = 6.2 Hz), 8.52 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.4, 21.6, 21.8, 22.0, 36.8, 70.1, 70.3, 70.9, 109.2, 122.3, 122.6, 126.0, 126.2, 126.3, 127.1, 128.7, 129.6, 130.7, 131.6, 131.8, 140.5, 153.7, 156.8, 179.0. HRMS (ESI): calcd for $C_{24}H_{26}F_3N_3O_5$ $[M+H]^+$ 494.1897; found 494.1900. HPLC (Sino-Chiral OD, 90:10 *n*-hexane/2-propanol, 15 °C, 1 mL·min^{-1}) t_R = 7.6 min (minor), 5.6 min (major).

3.2.9 Characterization of compound **3l**



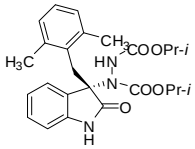
(*S*)-diisopropyl-1-(3-(2-methoxybenzyl)-2-oxoindolin-3-yl)hydrazine-1,2-dicarboxylate (3l):
White solid, mp 95-97 °C. $[\alpha]_D^{20}$ +13.5 (*c* 0.5, $CHCl_3$). FTIR ν_{max} (Film, cm^{-1}): 3259, 2981, 1724, 1622, 1471, 1384, 1250, 1181, 118, 752. ^1H NMR (300 MHz, $CDCl_3$) δ 0.83-0.84 (d, 3H, *J* = 5.5 Hz), 0.98-1.00 (d, 3H, *J* = 6.2 Hz), 1.27-1.29 (d, 6H, *J* = 6.2 Hz), 3.16-3.20 (d, 1H, *J* = 12.3 Hz), 3.37 (s, 3H), 3.43-3.47 (d, 1H, *J* = 12.3 Hz), 4.61-4.69 (m, 1H), 4.98-5.06 (m, 1H), 6.34-6.45 (m, 2H), 6.52-6.56 (t, 1H, *J* = 7.7 Hz), 6.76-6.79 (d, 1H, *J* = 7.7 Hz), 6.90-6.92 (m, 3H), 7.19 (s, 1H), 7.79-7.81 (d, 1H, *J* = 6.4 Hz), 7.89 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.5, 21.6, 22.0, 22.1, 34.4, 54.7, 70.0, 70.2, 70.7, 108.7, 109.6, 119.6, 121.3, 121.9, 125.9, 128.1, 128.3, 130.4, 131.8, 140.3, 153.8, 156.9, 157.7, 178.7. HRMS (EI): calcd for $C_{24}H_{29}N_3O_6$ $[M]^+$ 455.2056; found 455.2060. HPLC (Sino-Chiral OD, 90:10 *n*-hexane/2-propanol, 15 °C, 0.5 mL·min^{-1}) t_R = 13.0 min (minor), 11.3 min (major).

3.2.10 Characterization of compound **3m**



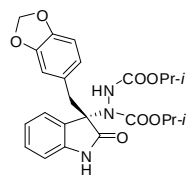
(*S*)-diisopropyl-1-(3-(2-methylbenzyl)-2-oxoindolin-3-yl)hydrazine-1,2-dicarboxylate (3m):
Waxy oil. $[\alpha]_D^{20}$ +44.0 (*c* 1.5, $CHCl_3$). FTIR ν_{max} (Film, cm^{-1}): 3266, 2981, 1725, 1622, 1472, 1384, 1250, 1180, 1107, 755. ^1H NMR (300 MHz, $CDCl_3$) δ 0.81-0.82 (d, 3H, *J* = 5.1 Hz), 0.97-0.99 (d, 3H, *J* = 6.2 Hz), 1.28-1.30 (d, 6H, *J* = 6.2 Hz), 1.99 (s, 3H), 3.16-3.21 (d, 1H, *J* = 12.7 Hz), 3.31-3.35 (d, 1H, *J* = 12.9 Hz), 4.60-4.68 (m, 1H), 4.98-5.07 (m, 1H), 6.35-6.37 (d, 1H, *J* = 6.8 Hz), 6.53-6.56 (d, 1H, *J* = 7.6 Hz), 6.68-6.71 (m, 1H), 6.80-6.82 (m, 2H), 6.95-6.99 (m, 2H), 7.38 (s, 1H), 7.91-7.93 (d, 1H, *J* = 6.5 Hz), 8.16 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 19.8, 21.4, 21.6, 22.0, 22.1, 37.5, 70.2, 70.3, 70.8, 109.3, 122.5, 124.8, 125.4, 126.9, 128.7, 130.0, 130.5, 131.0, 137.7, 140.6, 153.7, 157.2, 178.9. HRMS (ESI): calcd for $C_{24}H_{29}N_3O_5$ $[M+H]^+$ 440.2180; found 440.2183. HPLC (Sino-Chiral OD, 90:10 *n*-hexane/2-propanol, 10 °C, 1 mL·min^{-1}) t_R = 8.7 min (minor), 4.0 min (major).

3.2.11 Characterization of compound **3n**



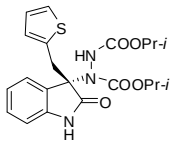
(*S*)-diisopropyl-1-(3-(2,6-dimethylbenzyl)-2-oxoindolin-3-yl)hydrazine-1,2-dicarboxylate (3n):
Waxy oil. $[\alpha]_D^{20}$ +32.7 (*c* 1.9, $CHCl_3$). FTIR ν_{max} (Film, cm^{-1}): 3259, 2979, 1727, 1622, 1472, 1383, 1250, 1108, 754. ^1H NMR (300 MHz, $CDCl_3$) δ 0.81-0.83 (d, 3H, *J* = 5.4 Hz), 0.97-0.99 (d, 3H, *J* = 6.2 Hz), 1.27-1.29 (d, 6H, *J* = 6.2 Hz), 1.87 (s, 3H), 1.98 (s, 3H), 3.06-3.11 (d, 1H, *J* = 12.4 Hz), 3.29-3.33 (d, 1H, *J* = 12.4 Hz), 4.60-4.68 (m, 1H), 4.98-5.06 (m, 1H), 6.21 (s, 1H), 6.38-6.43 (d, 1H, *J* = 7.0 Hz), 6.60-6.62 (d, 1H, *J* = 6.2 Hz), 6.68-6.70 (d, 1H, *J* = 7.7 Hz), 6.97-7.01 (m, 2H), 7.32 (s, 1H), 7.90-7.93 (m, 2H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 19.3, 20.6, 21.4, 21.6, 22.0, 22.1, 37.4, 70.1, 70.3, 70.7, 109.2, 122.4, 125.5, 127.5, 128.6, 129.8, 130.7, 130.8, 131.8, 133.9, 134.4, 140.7, 153.7, 157.2, 178.7. HRMS (ESI): calcd for $C_{25}H_{31}N_3O_5$ $[M+H]^+$ 454.2336; found 454.2335. HPLC (Sino-Chiral OD, 90:10 *n*-hexane/2-propanol, 10 °C, 1 mL·min^{-1}) t_R = 7.7 min (minor), 3.5 min (major).

3.2.12 Characterization of compound **3o**



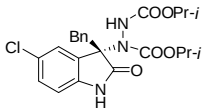
(*S*)-diisopropyl-1-(3-(benzo[*d*][1,3]dioxol-5-ylmethyl)-2-oxoindolin-3-yl)hydrazine-1,2-dicarboxylate (3o):
White solid, mp 115-116 °C. $[\alpha]_D^{20}$ +50.4 (*c* 1.1, $CHCl_3$). FTIR ν_{max} (Film, cm^{-1}): 3276, 1724, 1489, 1384, 1252, 1106, 1038, 754. ^1H NMR (300 MHz, $CDCl_3$) δ 0.86 (s, 3H), 1.00-1.02 (d, 3H, *J* = 5.8 Hz), 1.28-1.30 (d, 6H, *J* = 5.8 Hz), 3.00-3.04 (d, 1H, *J* = 11.6 Hz), 3.09-3.14 (d, 1H, *J* = 11.6 Hz), 4.65-4.71 (m, 1H), 5.00-5.06 (m, 1H), 5.64 (s, 2H), 6.06 (s, 2H), 6.33-6.35 (d, 1H, *J* = 8.2 Hz), 6.47-6.49 (d, 1H, *J* = 5.8 Hz), 7.02-7.07 (m, 2H), 7.19 (s, 1H), 7.66 (s, 1H), 7.84-7.86 (d, 1H, *J* = 5.8 Hz); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.5, 21.6, 21.9, 22.1, 41.4, 70.1, 70.2, 70.8, 100.7, 107.4, 109.3, 110.5, 122.9, 123.9, 124.9, 126.2, 128.8, 130.4, 140.4, 146.4, 146.7, 153.7, 157.1, 177.8. HRMS (ESI): calcd for $C_{24}H_{27}N_3O_7$ $[M+H]^+$ 470.1922; found 470.1922. HPLC (Sino-Chiral OD, 90:10 *n*-hexane/2-propanol, 10 °C, 1 mL·min^{-1}) t_R = 6.9 min (minor), 7.9 min (major).

3.2.13 Characterization of compound **3p**



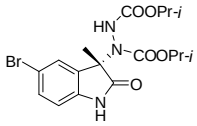
(*S*)-diisopropyl-1-(2-oxo-3-(thiophen-2-ylmethyl)indolin-3-yl)hydrazine-1,2-dicarboxylate (3p):
Pale yellow solid, mp 109-110 °C. $[\alpha]_D^{20}$ +10.8 (*c* 1.3, $CHCl_3$). FTIR ν_{max} (Film, cm^{-1}): 3259, 2981, 1726, 1473, 1383, 1250, 1107, 753. ^1H NMR (300 MHz, $CDCl_3$) δ 0.92-0.93 (d, 3H, *J* = 5.2 Hz), 1.06-1.08 (d, 3H, *J* = 6.4 Hz), 1.34-1.36 (d, 6H, *J* = 6.4 Hz), 3.35-3.39 (d, 1H, *J* = 13.4 Hz), 3.53-3.58 (d, 1H, *J* = 13.3 Hz), 4.69-4.78 (m, 1H), 5.04-5.12 (m, 1H), 6.47-6.48 (d, 1H, *J* = 2.8 Hz), 6.58-6.60 (d, 1H, *J* = 7.0 Hz), 6.64-6.67 (m, 1H), 6.90-6.91 (d, 1H, *J* = 4.9 Hz), 7.10-7.19 (m, 2H), 7.46 (s, 1H), 7.89-7.91 (d, 1H, *J* = 7.0 Hz), 8.40 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.5, 21.6, 22.0, 22.1, 36.2, 69.7, 70.2, 70.9, 109.6, 123.0, 125.0, 126.0, 128.2, 129.0, 130.3, 134.0, 141.2, 153.7, 157.1, 178.1. HRMS (EI): calcd for $C_{21}H_{25}N_3O_5S$ $[M]^+$ 431.1515; found 431.1518. HPLC (Chiralcel OG, 90:10 *n*-hexane/2-propanol, 10 °C, 1 mL·min^{-1}) t_R = 42.4 min (minor), 14.3 min (major).

3.2.14 Characterization of compound **3q**



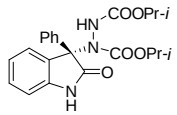
(*S*)-diisopropyl-1-(3-benzyl-5-chloro-2-oxoindolin-3-yl)hydrazine-1,2-dicarboxylate (3q):
White solid, mp 104-105 °C. $[\alpha]_D^{20}$ +87.1 (*c* 3.3, $CHCl_3$). FTIR ν_{max} (Film, cm^{-1}): 3259, 2979, 1728, 1470, 1384, 1252, 699. ^1H NMR (300 MHz, $CDCl_3$) δ 0.98 (s, 3H), 1.09-1.11 (d, 3H, *J* = 6.0 Hz), 1.35-1.37 (d, 6H, *J* = 6.0 Hz), 3.11-3.15 (d, 1H, *J* = 12.3 Hz), 3.23-3.27 (d, 1H, *J* = 12.1 Hz), 4.70-4.78 (m, 1H), 5.08-5.16 (m, 1H), 6.36-6.39 (m, 1H), 6.69-6.71 (d, 2H, *J* = 5.5 Hz), 6.97-7.00 (m, 3H), 7.06-7.09 (dd, 1H, *J* = 1.8, 8.2 Hz), 7.33 (s, 1H), 7.98 (s, 1H), 8.03 (s, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.6, 21.7, 22.0, 22.1, 41.7, 70.3, 71.1, 110.2, 125.4, 127.0, 127.6, 128.1, 128.7, 130.4, 132.1, 132.2, 139.0, 153.7, 157.0, 177.7. HRMS (ESI): calcd for $C_{23}H_{26}ClN_3O_5$ $[M+H]^+$ 460.1634; found 460.1638. HPLC (Chiralcel OG, 90:10 *n*-hexane/2-propanol, 10 °C, 1 mL·min^{-1}) t_R = 21.3 min (minor), 8.9 min (major).

3.2.15 Characterization of compound **3r**



(*S*)-diisopropyl-1-(5-bromo-3-methyl-2-oxoindolin-3-yl)hydrazine-1,2-dicarboxylate (3r):
White crystal, mp 153-154 °C. $[\alpha]_D^{20}$ -15.3 (*c* 1.1, $CHCl_3$). FTIR ν_{max} (Film, cm^{-1}): 3284, 2980, 1731, 1385, 1253, 1107. ^1H NMR (300 MHz, $CDCl_3$) δ 1.00 (m, 3H), 1.10-1.12 (d, 3H, *J* = 5.5 Hz), 1.32-1.33 (d, 6H, *J* = 4.3 Hz), 1.52 (s, 3H), 4.74-4.78 (m, 1H), 5.05-5.09 (m, 1H), 6.71-6.74 (d, 1H, *J* = 8.0 Hz), 6.87-6.96 (m, 1H), 7.32-7.34 (d, 1H, *J* = 7.4 Hz), 7.92 (s, 1H), 8.29 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.6, 21.7, 21.9, 22.1, 23.0, 66.0, 70.2, 71.1, 111.2, 115.8, 127.5, 131.4, 134.9, 138.5, 153.7, 156.7, 179.0. HRMS (EI): calcd for $C_{17}H_{22}N_3O_5Br$ $[M]^+$ 427.0743; found 427.0746. HPLC (Chiralcel OD-H, 90:10 *n*-hexane/2-propanol, 10 °C, 1 mL·min^{-1}) t_R = 5.9 min (minor), 7.0 min (major).

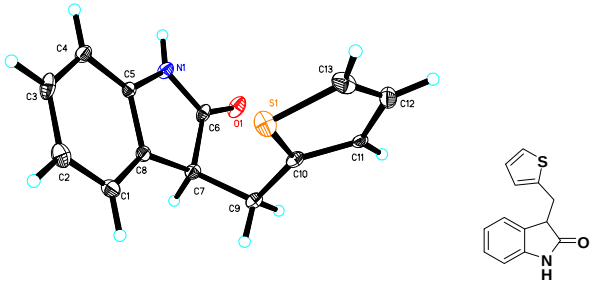
3.2.16 Characterization of compound **3s**



(*S*)-diisopropyl-1-(2-oxo-3-phenylindolin-3-yl)hydrazine-1,2-dicarboxylate (3s):
White solid, mp 121-123 °C. FTIR ν_{max} (Film, cm^{-1}): 3312, 2983, 1727, 1469, 1376, 1264, 1181, 1087, 764. ^1H NMR (300 MHz, $CDCl_3$) δ 0.91-0.93 (d, 3H, *J* = 6.2 Hz), 1.00-1.02 (d, 3H, *J* = 6.2 Hz), 1.07-1.09 (d, 3H, *J* = 6.2 Hz), 1.17-1.19 (d, 3H, *J* = 6.2 Hz), 4.73-4.86 (m, 2H), 6.80-6.83 (m, 1H), 7.11-7.16 (t, 1H, *J* = 7.4 Hz), 7.22-7.24 (m, 1H), 7.28-7.30 (m, 3H), 7.61-7.63 (m, 2H), 8.07-8.10 (m, 1H); ^{13}C NMR (75 MHz, $CDCl_3$) δ 21.5, 21.65, 21.70, 21.9, 69.7, 71.0, 72.7, 109.9, 122.6, 127.1, 128.3, 128.8, 128.9, 129.6, 130.0, 133.3, 140.4, 154.3, 155.6, 177.7. HRMS (EI): calcd for $C_{22}H_{25}N_3O_5$ $[M]^+$ 411.1794; found 411.1797. HPLC (Chiralcel OD-H, 90:10 *n*-hexane/2-propanol, 15 °C, 1 mL·min^{-1}) t_R = 9.3 min (minor), 16.3 min (major).

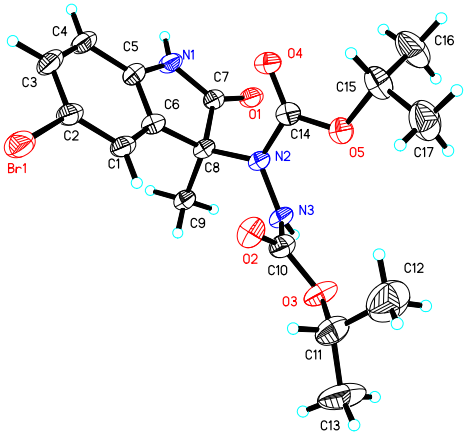
4. CRYSTAL DATA

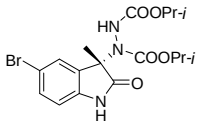
4.1 ORTEP drawing of 2-oxindole **1o**



Crystal data and structure refinement for sx2655		
Identification code	sx2655	
CCDC number	734758	
Empirical formula	$C_{13}H_{11}NOS$	
Formula weight	229.29	
Temperature	173(2) K	
Wavelength	0.71073 A	
Crystal system, space group	Monoclinic, P2(1)	
Unit cell dimensions	a = 5.8503(12) Å	α = 90 deg.
	b = 8.4929(17) Å	β = 95.88(3) deg.
	c = 10.957(2) Å	γ = 90 deg.
Volume	541.54(19) $Å^3$	
Z, Calculated density	2, 1.406 mg • m^{-3}	
Absorption coefficient	0.273 mm^{-1}	
F(000)	240	
Crystal size	0.40 mm×0.35 mm×0.14 mm	
Theta range for data collection	1.87 to 27.44 deg.	
Limiting indices	-7<=h<=7, -11<=k<=11, -14<=l<=14	
Reflections collected / unique	2460 / 2460 [R(int) = 0.0000]	
Completeness to theta = 27.44	99.8 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9627 and 0.8985	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2460 / 1 / 145	
Goodness-of-fit on F^2	1.039	
Final R indices [I>2sigma(I)]	R1 = 0.0444, wR2 = 0.0921	
R indices (all data)	R1 = 0.0591, wR2 = 0.1142	
Absolute structure parameter	-0.02(11)	
Largest diff. peak and hole	0.345 and -0.233 e. $Å^{-3}$	

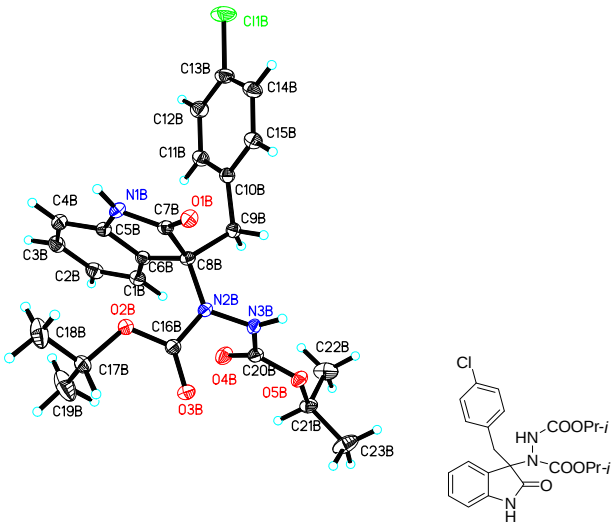
4.2 ORTEP drawing of (*S*)-**3r**





Crystal data and structure refinement for sa119		
Identification code	sa119	
CCDC number	734756	
Empirical formula	$C_{17}H_{22}BrN_3O_5$	
Formula weight	428.29	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system, space group	Trigonal, R3	
Unit cell dimensions	a = 21.619(3) Å	α = 90 deg.
	b = 21.619(3) Å	β = 90 deg.
	c = 12.828(3) Å	γ = 120 deg.
Volume	5192.4(15) $Å^3$	
Z, Calculated density	9, 1.233 mg • m^{-3}	
Absorption coefficient	1.808 mm^{-1}	
F(000)	1980	
Crystal size	0.30 mm×0.26 mm×0.23 mm	
Theta range for data collection	2.69 to 27.48 deg.	
Limiting indices	-27<=h<=28, -28<=k<=28, -16<=l<=16	
Reflections collected / unique	20929 / 5305 [R(int) = 0.0546]	
Completeness to theta = 25.02	99.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.6812 and 0.6131	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5305 / 124 / 291	
Goodness-of-fit on F^2	1.081	
Final R indices [I>2sigma(I)]	R1 = 0.0473, wR2 = 0.1265	
R indices (all data)	R1 = 0.0518, wR2 = 0.1310	
Absolute structure parameter	0.052(10)	
Largest diff. peak and hole	0.461 and -0.307 e. $Å^{-3}$	

4.3 ORTEP drawing of racemic **3f**



Crystal data and structure refinement for sa155		
Identification code	sa155	
CCDC number	734757	
Empirical formula	$C_{23}H_{26}ClN_3O_5$	
Formula weight	459.92	
Temperature	173(2) K	
Wavelength	0.71073 Å	
Crystal system, space group	Triclinic, P-1	
Unit cell dimensions	a = 8.9418(18) Å	α = 115.51(3) deg.
	b = 12.020(2) Å	β = 92.14(3) deg.
	c = 12.756(3) Å	γ = 107.65(3) deg.
Volume	1156.4(4) $Å^3$	
Z, Calculated density	2, 1.321 mg • m^{-3}	
Absorption coefficient	0.204 mm^{-1}	
F(000)	484	
Crystal size	0.35 mm×0.32 mm×0.32 mm	
Theta range for data collection	2.73 to 27.48 deg.	
Limiting indices	-11<=h<=11, -15<=k<=15, -16<=l<=16	
Reflections collected / unique	18945 / 5296 [R(int) = 0.0316]	
Completeness to theta = 25.02	99.7 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9376 and 0.9320	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	5296 / 0 / 293	
Goodness-of-fit on F^2	1.124	
Final R indices [I>2sigma(I)]	R1 = 0.0549, wR2 = 0.1270	
R indices (all data)	R1 = 0.0615, wR2 = 0.1315	
Largest diff. peak and hole	0.526 and -0.523 e. $Å^{-3}$	

5. REFERENCES

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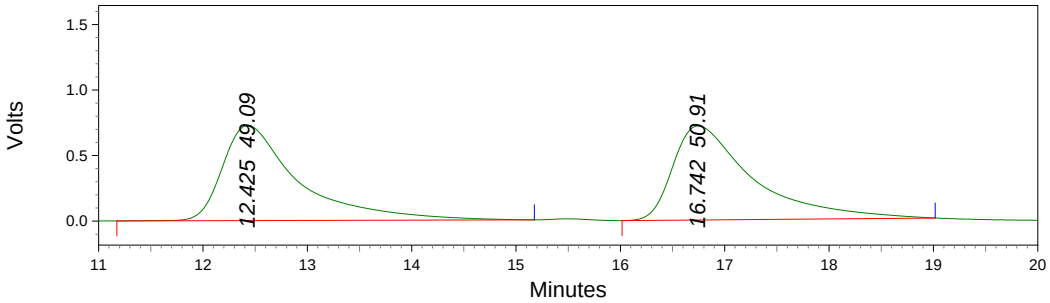
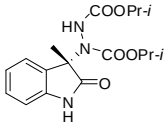
6. HPLC TRACES

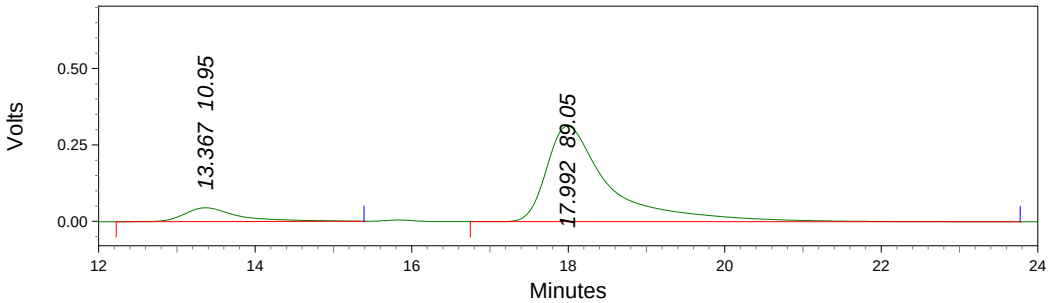
6.1 HPLC trace for compound **3b**

racemic sample



Retention Time	Area	Area Percent
12.425	36548226	49.09
16.742	37902237	50.91
Totals	74450463	100.00

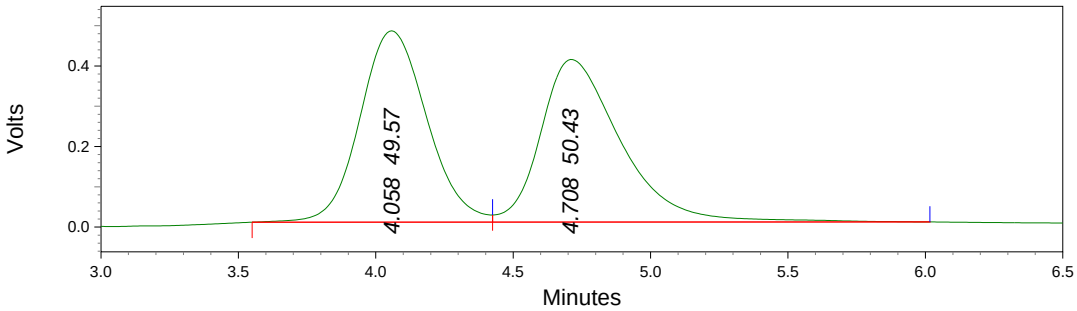




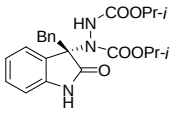
Retention Time	Area	Area Percent
13.367	2134564	10.95
17.992	17365673	89.05
Totals	19500237	100.00

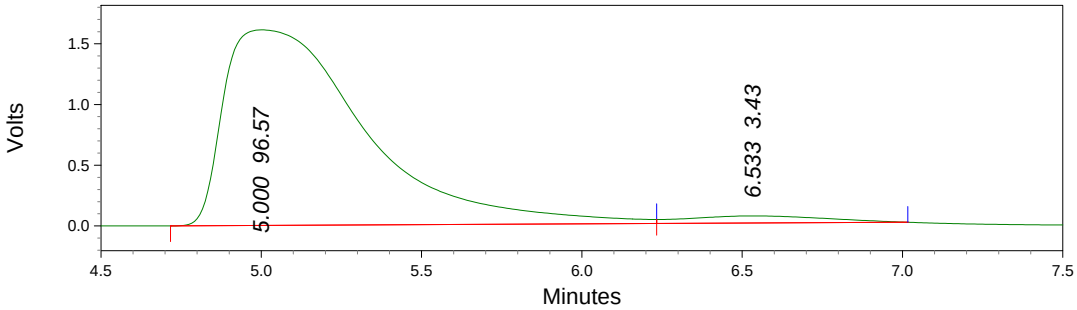
6.2 HPLC trace for compound **3e**

racemic sample



Retention Time	Area	Area Percent
4.058	8045880	49.57
4.708	8185118	50.43
Totals	16230998	100.00

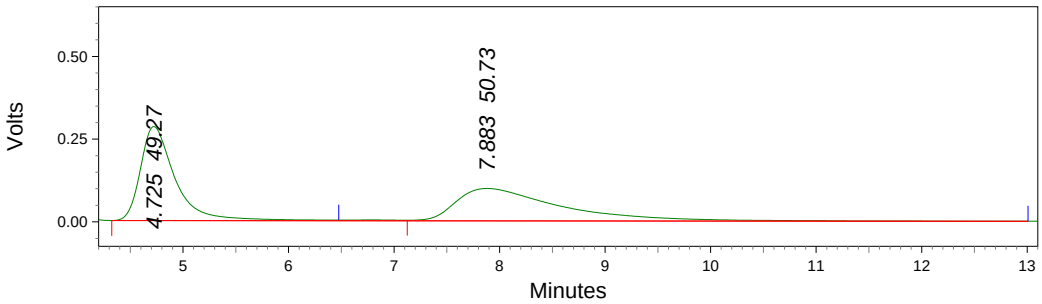




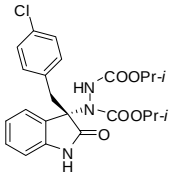
Retention Time	Area	Area Percent
5.000	49453926	96.57
6.533	1754840	3.43
Totals	51208766	100.00

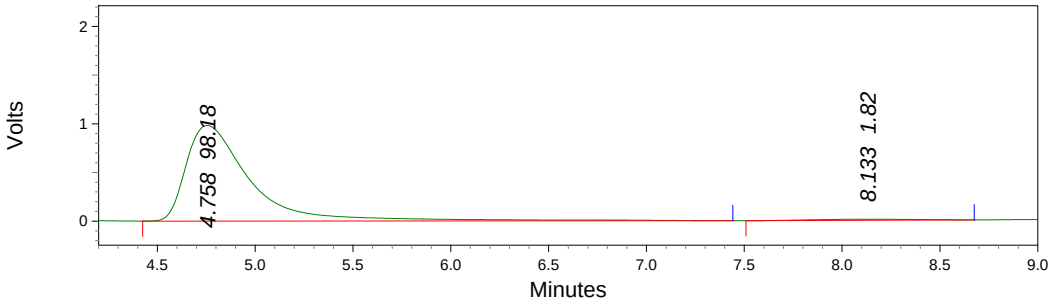
6.3 HPLC trace for compound **3f**

racemic sample



Retention Time	Area	Area Percent
4.725	6498367	49.27
7.883	6692103	50.73
Totals	13190470	100.00

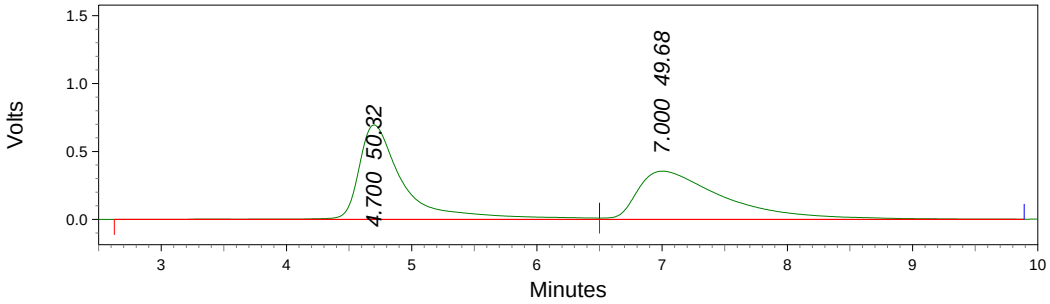




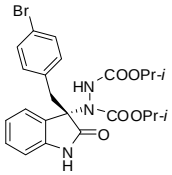
Retention Time	Area	Area Percent
4.758	22411737	98.18
8.133	416244	1.82
Totals	22827981	100.00

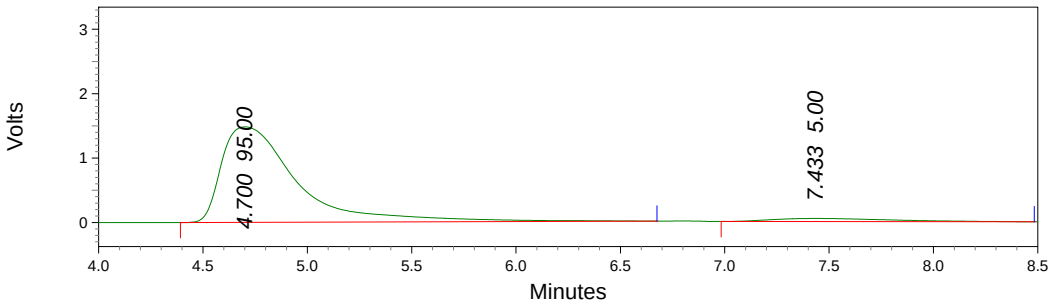
6.4 HPLC trace for compound **3g**

racemic sample



Retention Time	Area	Area Percent
4.700	17227334	50.32
7.000	17009018	49.68
Totals	34236352	100.00

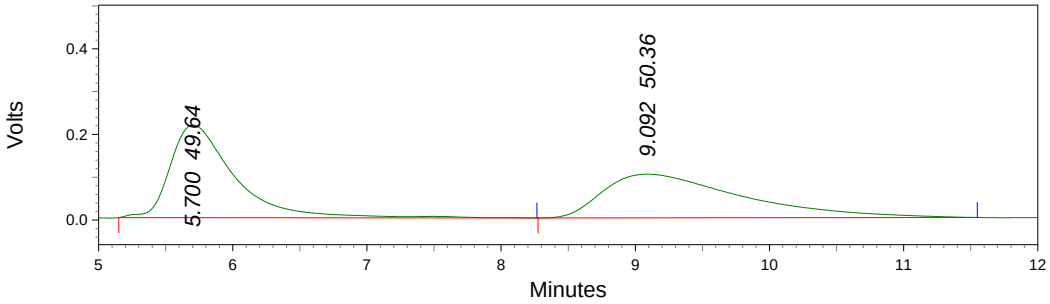




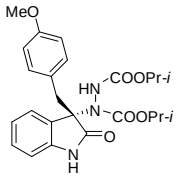
Retention Time	Area	Area Percent
4.700	37209870	95.00
7.433	1959969	5.00
Totals	39169839	100.00

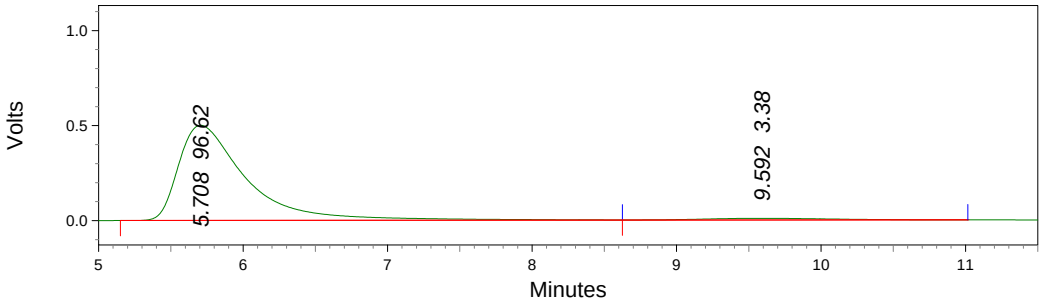
6.5 HPLC trace for compound **3h**

racemic sample



Retention Time	Area	Area Percent
5.700	7193554	49.64
9.092	7298255	50.36
Totals	14491809	100.00

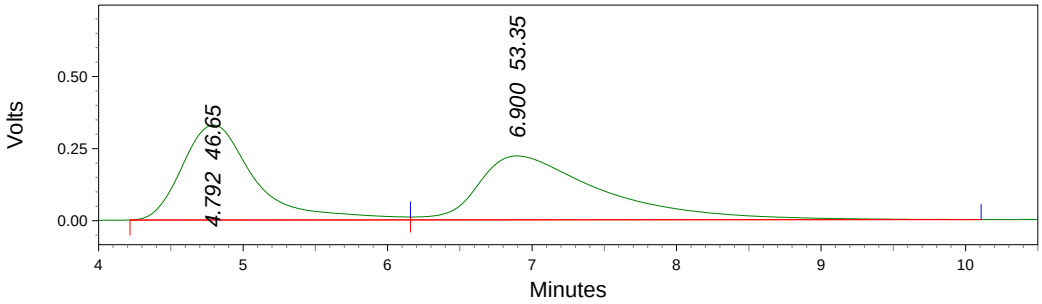




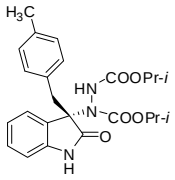
Retention Time	Area	Area Percent
5.708	16324479	96.62
9.592	570520	3.38
Totals	16894999	100.00

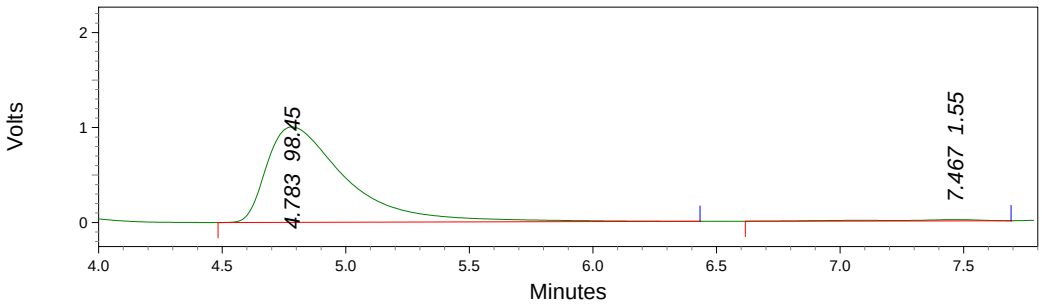
6.6 HPLC trace for compound **3i**

racemic sample



Retention Time	Area	Area Percent
4.792	11454695	46.65
6.900	13099170	53.35
Totals	24553865	100.00

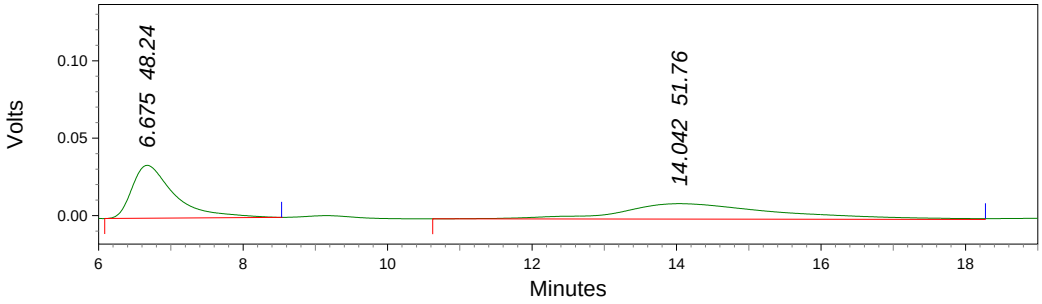




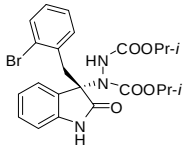
Retention Time	Area	Area Percent
4.783	22387319	98.45
7.467	352078	1.55
Totals	22739397	100.00

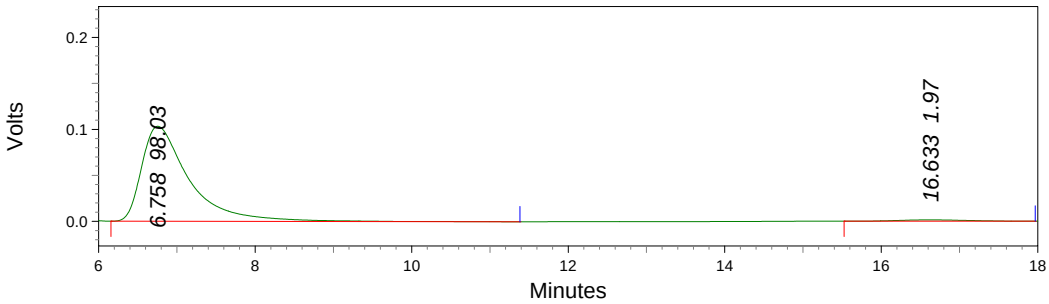
6.7 HPLC trace for compound **3j**

racemic sample



Retention Time	Area	Area Percent
6.675	1391738	48.24
14.042	1493071	51.76
Totals	2884809	100.00

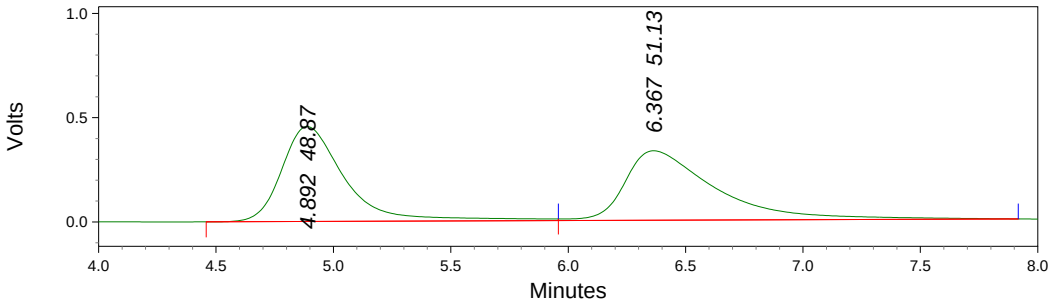




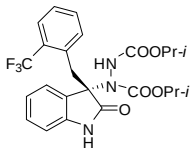
Retention Time	Area	Area Percent
6.758	4350240	98.03
16.633	87623	1.97
Totals	4437863	100.00

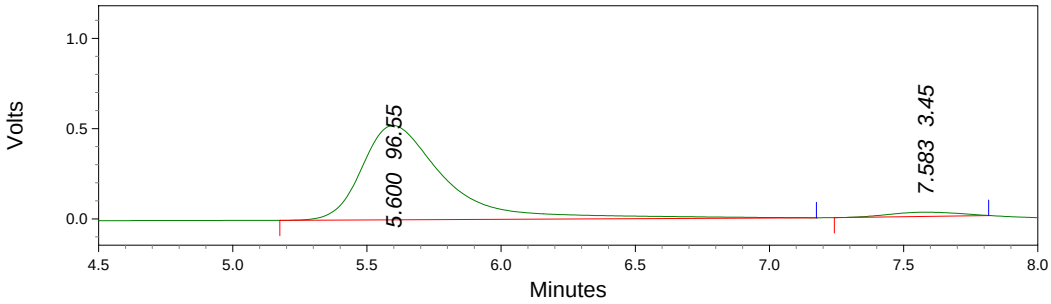
6.8 HPLC trace for compound **3k**

racemic sample



Retention Time	Area	Area Percent
4.892	8546418	48.87
6.367	8942442	51.13
Totals	17488860	100.00

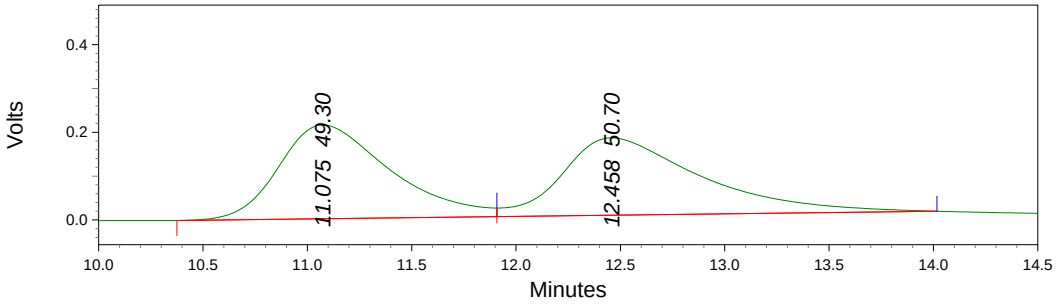




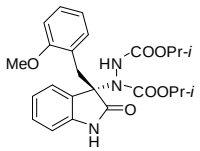
Retention Time	Area	Area Percent
5.600	11269952	96.55
7.583	403013	3.45
Totals	11672965	100.00

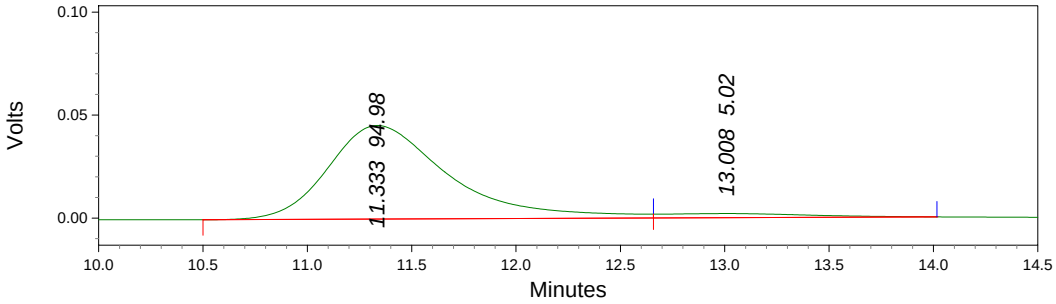
6.9 HPLC trace for compound **3l**

racemic sample



Retention Time	Area	Area Percent
11.075	7923948	49.30
12.458	8149428	50.70
Totals	16073376	100.00

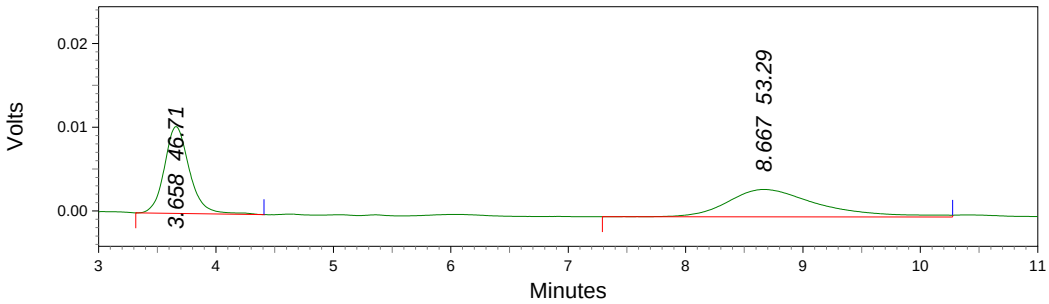




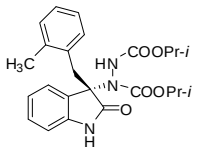
Retention Time	Area	Area Percent
11.333	1826161	94.98
13.008	96428	5.02
Totals	1922589	100.00

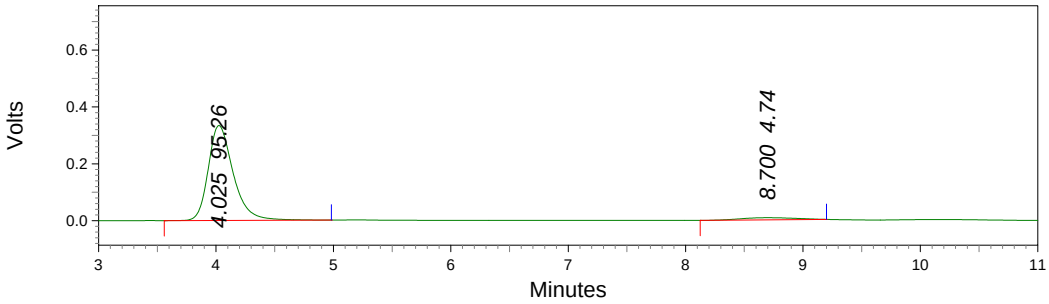
6.10 HPLC trace for compound **3m**

racemic sample



Retention Time	Area	Area Percent
3.658	153847	46.71
8.667	175548	53.29
Totals	329395	100.00

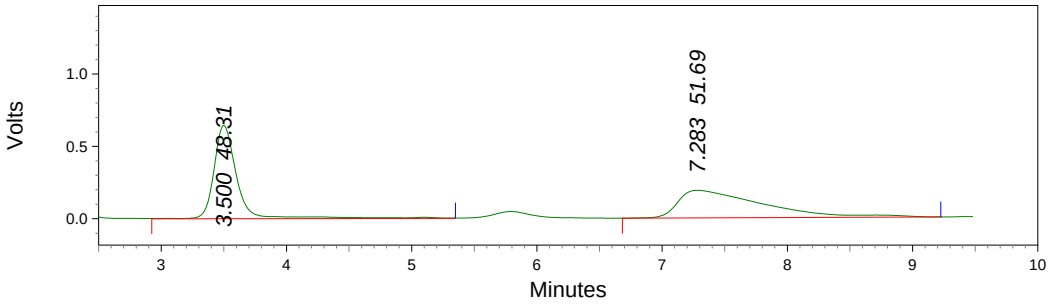




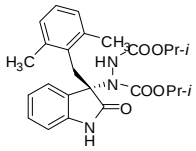
Retention Time	Area	Area Percent
4.025	4916989	95.26
8.700	244394	4.74
Totals	5161383	100.00

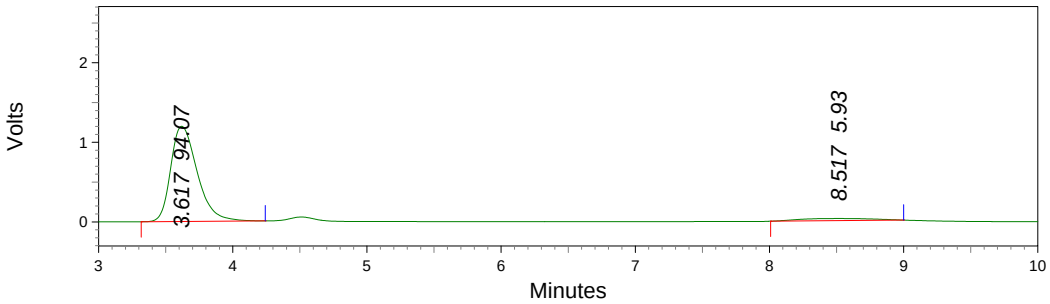
6.11 HPLC trace for compound **3n**

racemic sample



Retention Time	Area	Area Percent
3.500	8360658	48.31
7.283	8945414	51.69
Totals	17306072	100.00

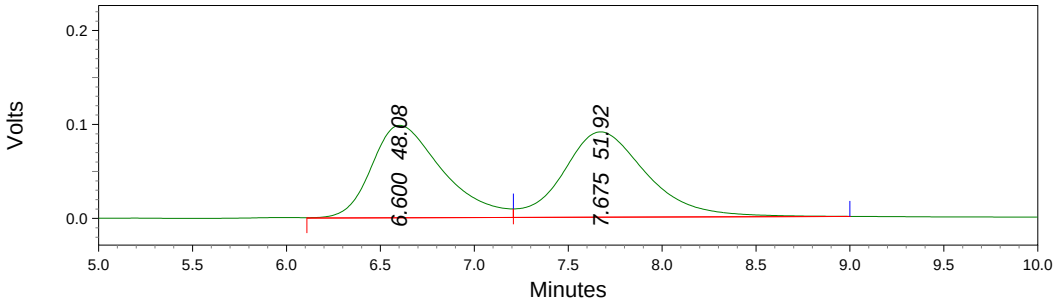




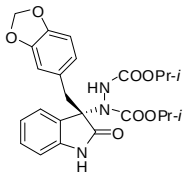
Retention Time	Area	Area Percent
3.617	16395793	94.07
8.517	1033034	5.93
Totals	17428827	100.00

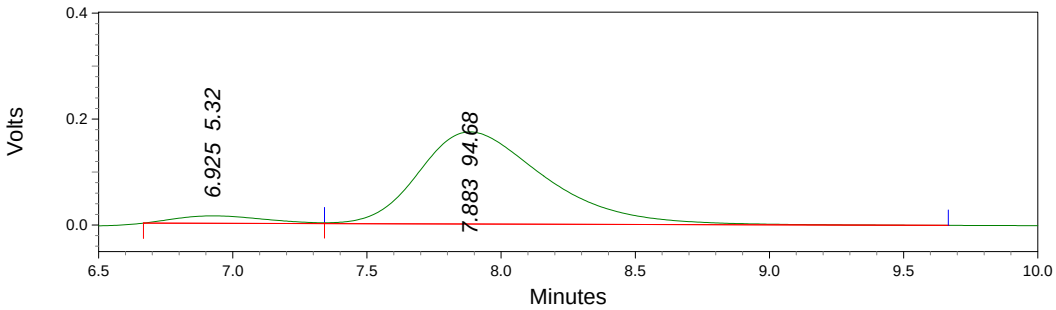
6.12 HPLC trace for compound **3o**

racemic sample



Retention Time	Area	Area Percent
6.600	2582546	48.08
7.675	2789142	51.92
Totals	5371688	100.00

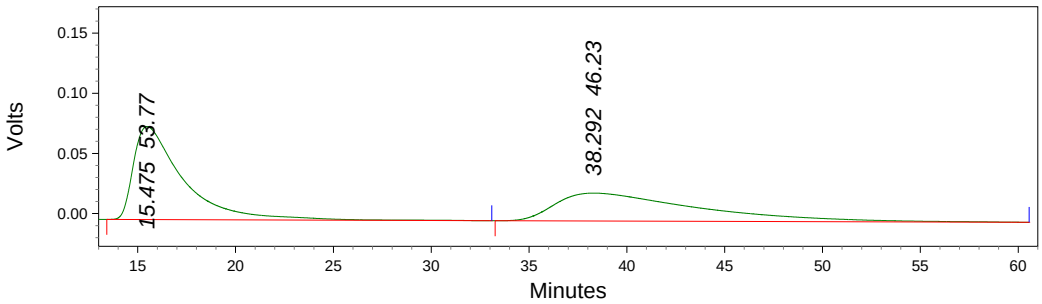




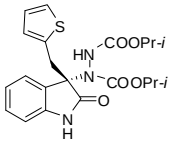
Retention Time	Area	Area Percent
6.925	320988	5.32
7.883	5711317	94.68
Totals	6032305	100.00

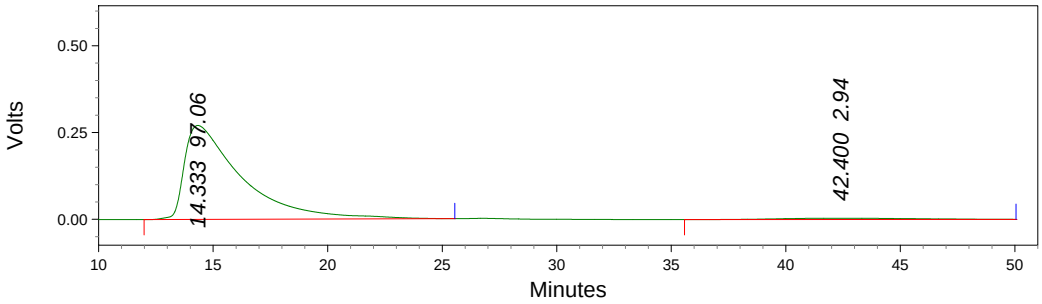
6.13 HPLC trace for compound **3p**

racemic sample



Retention Time	Area	Area Percent
15.475	13681970	53.77
38.292	11765060	46.23
Totals	25447030	100.00

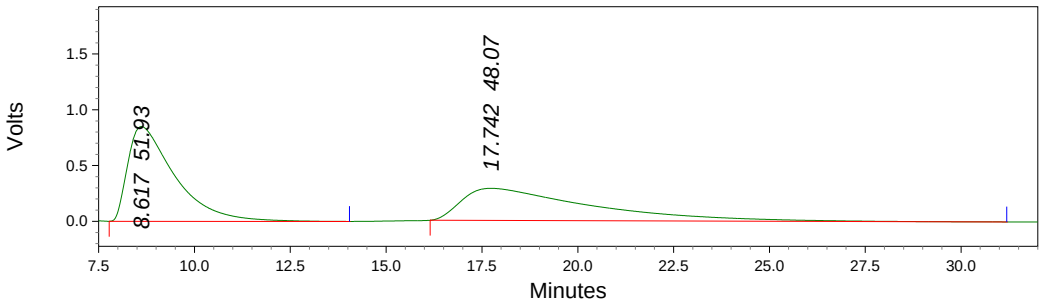




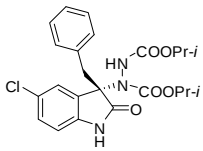
Retention Time	Area	Area Percent
14.333	46925521	97.06
42.400	1421097	2.94
Totals	48346618	100.00

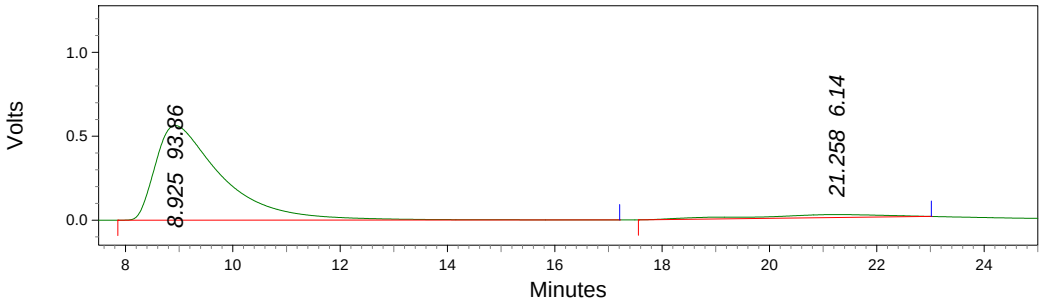
6.14 HPLC trace for compound **3q**

racemic sample



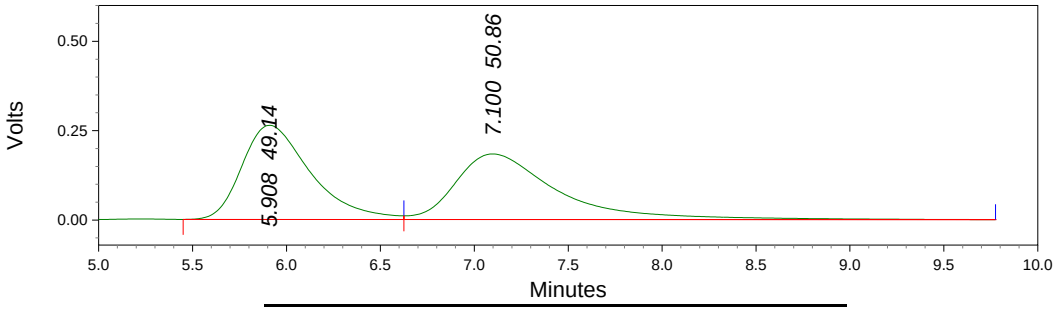
Retention Time	Area	Area Percent
8.617	72318942	51.93
17.742	66933285	48.07
Totals	139252227	100.00



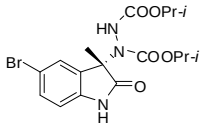


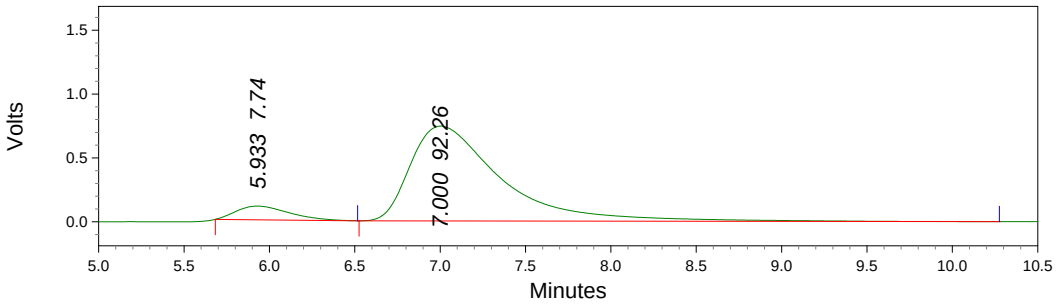
Retention Time	Area	Area Percent
8.925	48402288	93.86
21.258	3168511	6.14
Totals	51570799	100.00

6.15 HPLC trace for compound **3r**
racemic sample



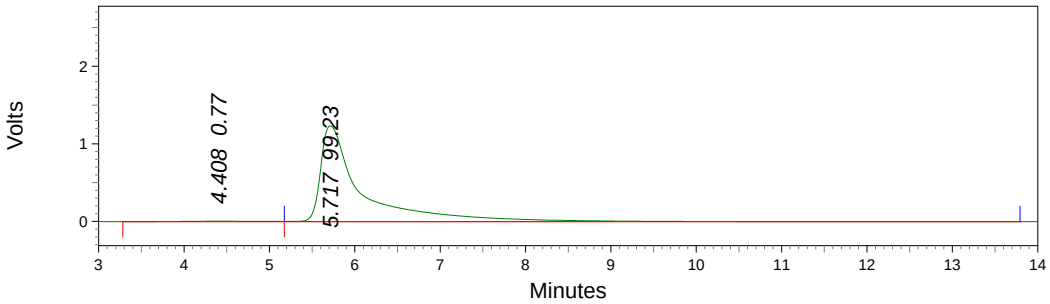
Retention Time	Area	Area Percent
5.908	6678489	49.14
7.100	6913046	50.86
Totals	13591535	100.00





Retention Time	Area	Area Percent
5.933	2292776	7.74
7.000	27348436	92.26
Totals	29641212	100.00

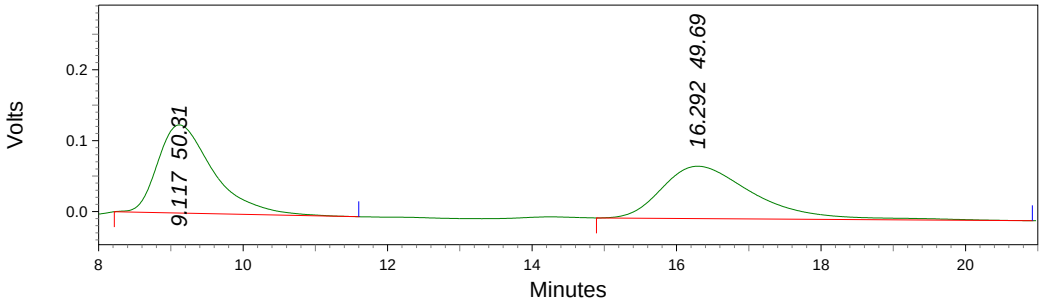
HPLC trace for the single crystal



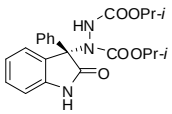
Retention Time	Area	Area Percent
4.408	319910	0.77
5.717	41305696	99.23
Totals	41625606	100.00

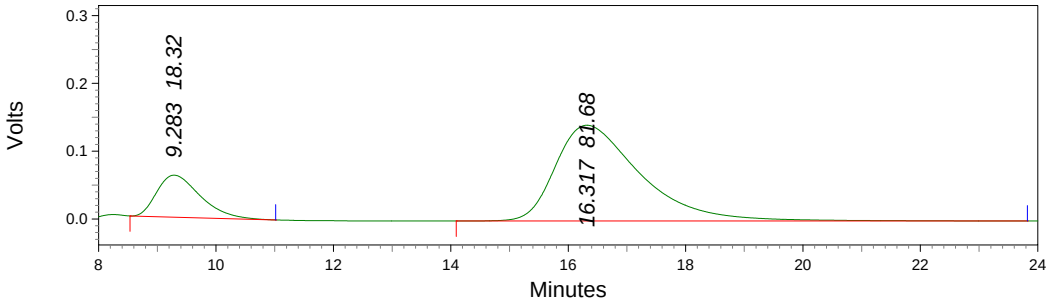
6.16 HPLC trace for compound **3s**

racemic sample



Retention Time	Area	Area Percent
9.117	6618250	50.31
16.292	6536624	49.69
Totals	13154874	100.00

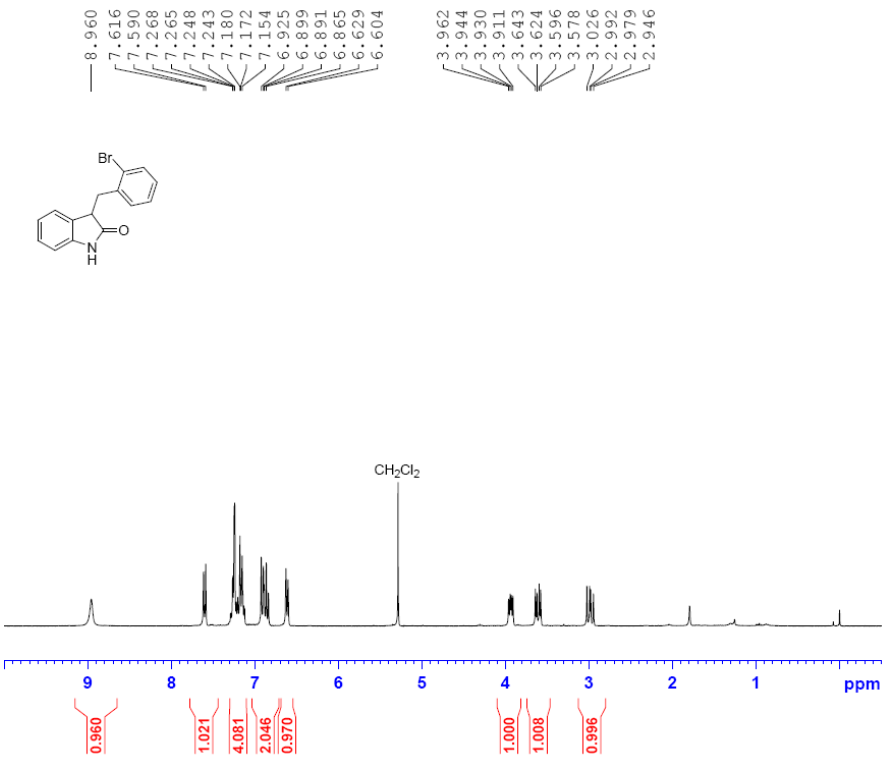




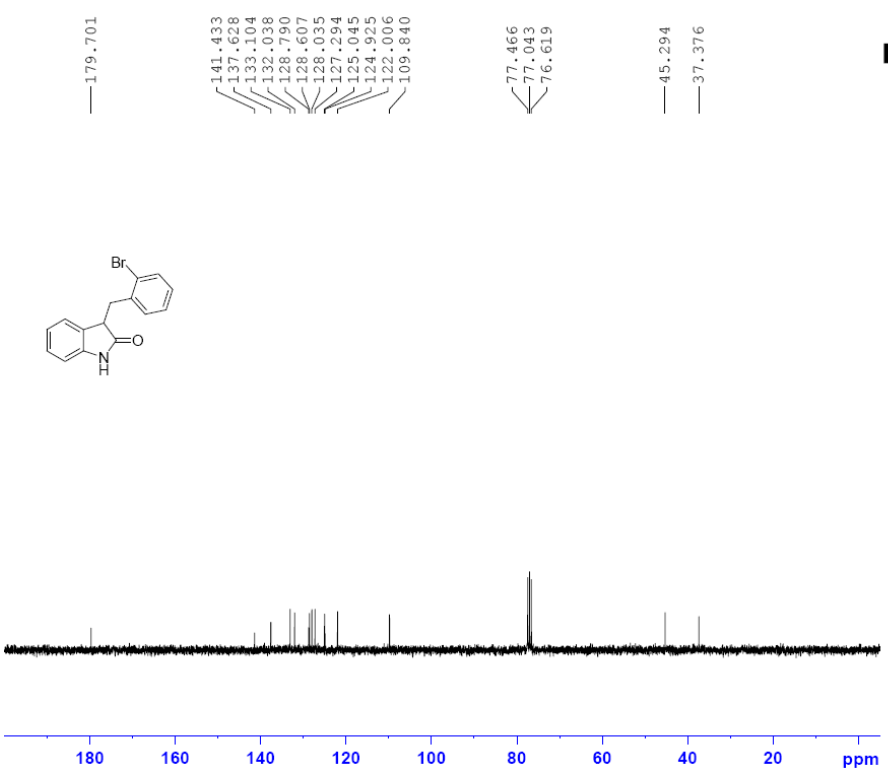
Retention Time	Area	Area Percent
9.283	3189325	18.32
16.317	14222075	81.68
Totals	17411400	100.00

7. NMR SPECTRUM

7.1 ^{1}H NMR and ^{13}C NMR spectrum of **1i**

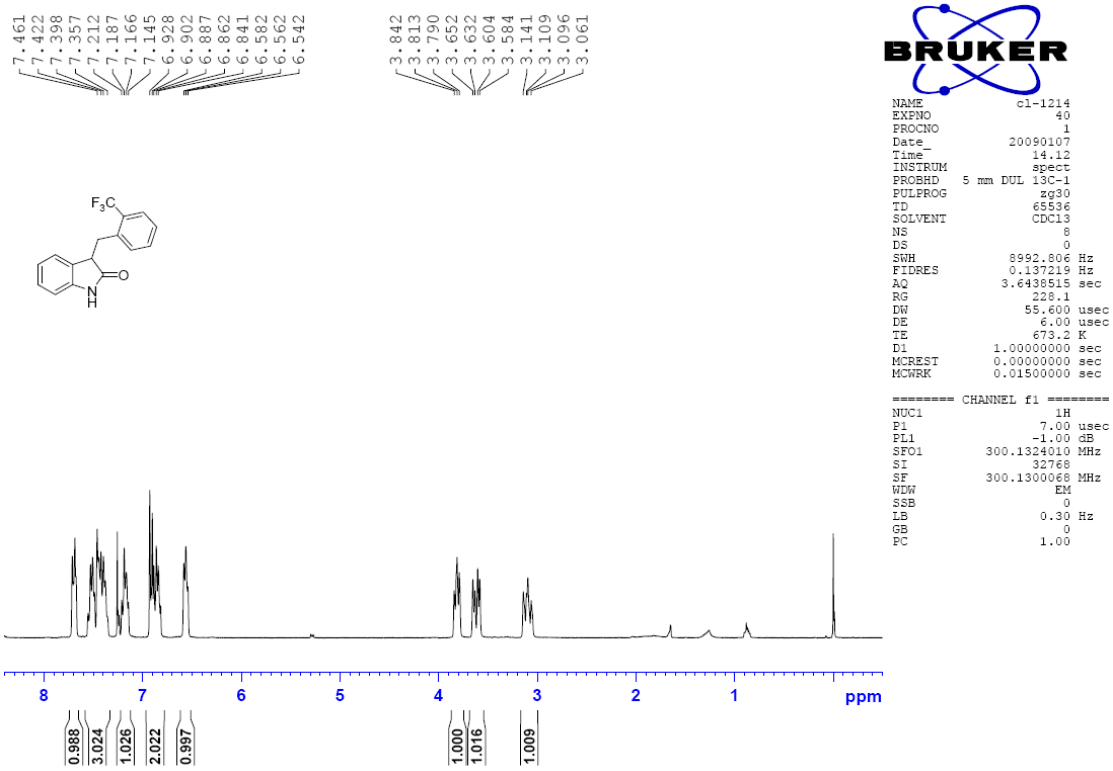


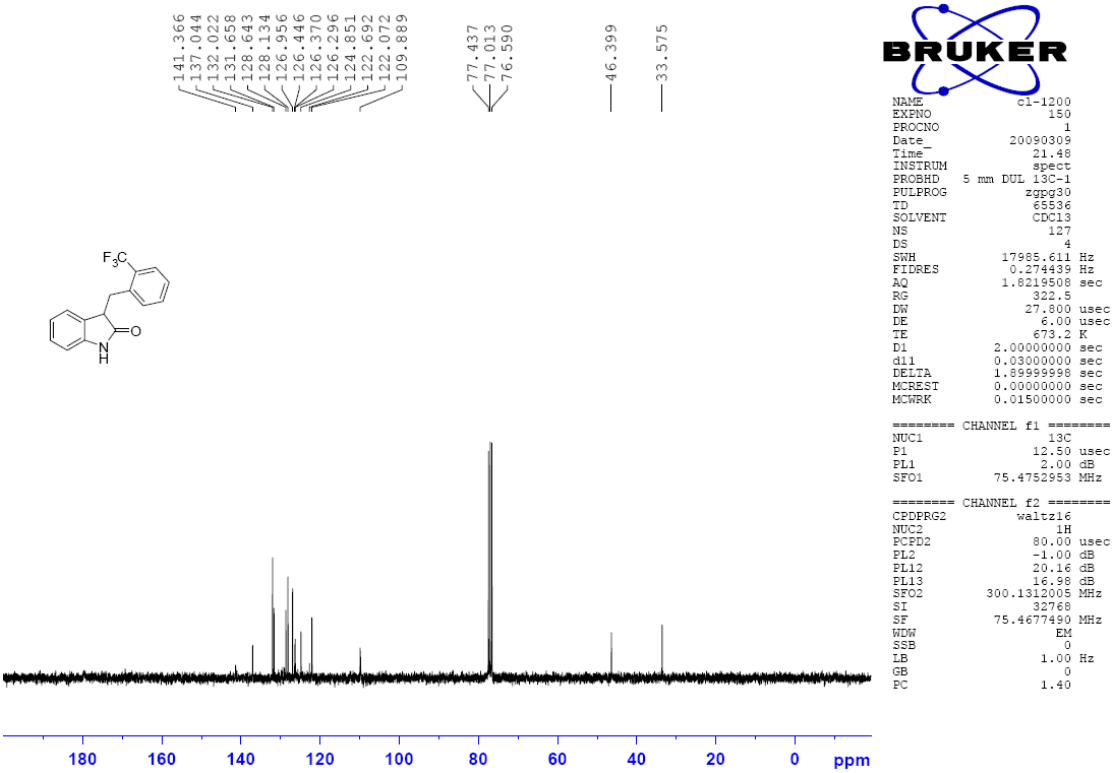

```
NAME            cl-1200
EXPNO               111
PROCNO                1
Date_          20081211
Time              10.06
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    4
DS                    0
SWH            8992.806 Hz
FIDRES         0.137219 Hz
AQ           3.6438515 sec
RG               161.3
DW              55.600 usec
DE               6.00 usec
TE              297.2 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                7.00 usec
PL1              -1.00 dB
SFO1        300.1324010 MHz
SI                32768
SF          300.1300080 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



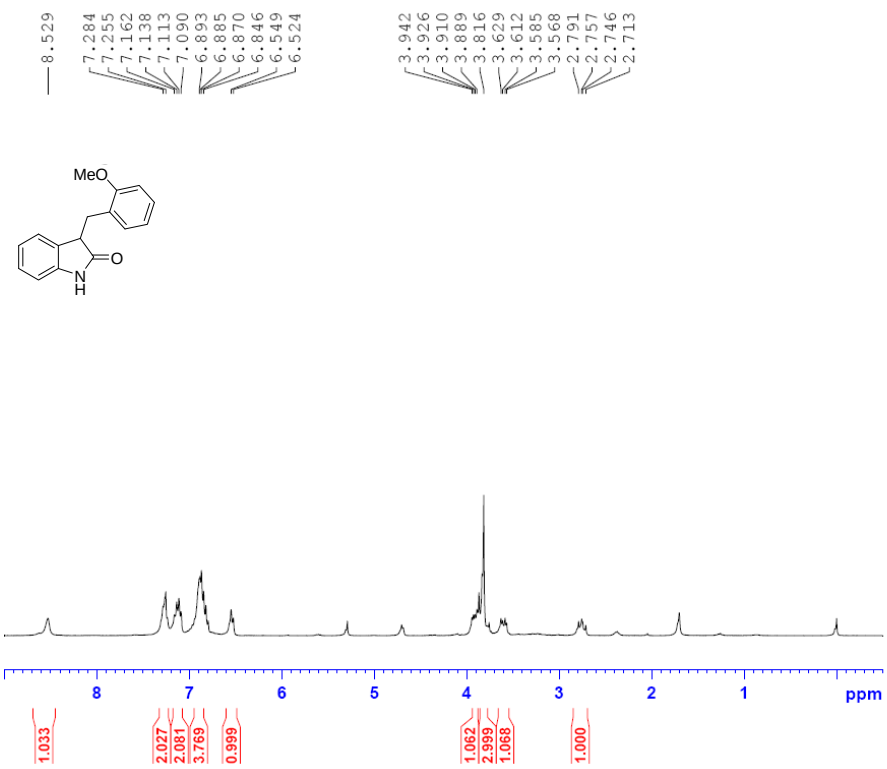

```
NAME            cl-1200
EXPNO               112
PROCNO                1
Date_          20081211
Time              10.08
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   16
DS                    4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ           1.8219508 sec
RG               645.1
DW              27.800 usec
DE               6.00 usec
TE              296.8 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1               12.50 usec
PL1               2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            80.00 usec
PL2              -1.00 dB
PL12             20.16 dB
PL13             16.98 dB
SFO2        300.1312005 MHz
SI                32768
SF           75.4677490 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

7.2 ^{1}H NMR and ^{13}C NMR spectrum of **1j**

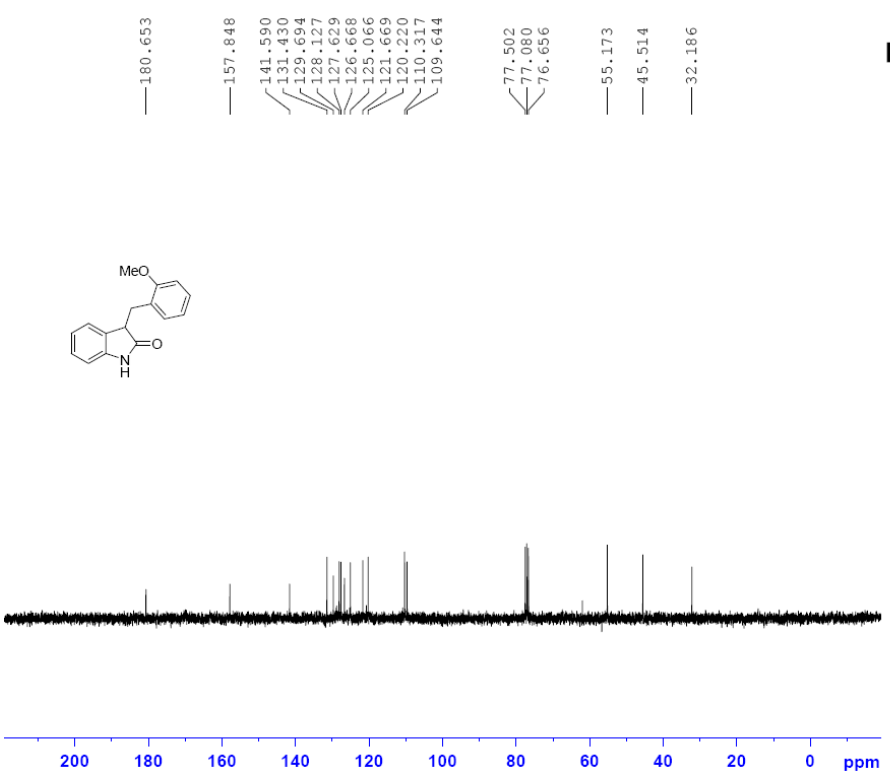




7.3 ^{1}H NMR and ^{13}C NMR spectrum of **1k**

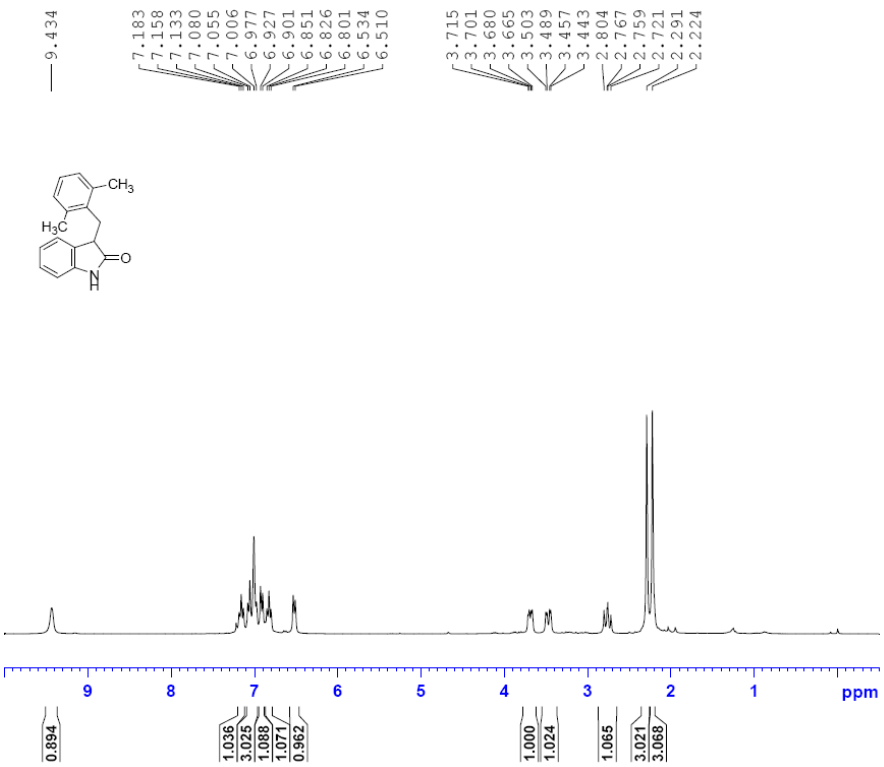



```
NAME            cl-1200
EXPNO                80
PROCNO                1
Date_          20081129
Time              10.43
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   12
DS                    0
SWH            8992.806 Hz
FIDRES         0.137219 Hz
AQ           3.6438515 sec
RG               203.2
DW              55.600 usec
DE               6.00 usec
TE              297.9 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                7.00 usec
PL1              -1.00 dB
SFO1        300.1324010 MHz
SI                32768
SF          300.1300064 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                1.00
```

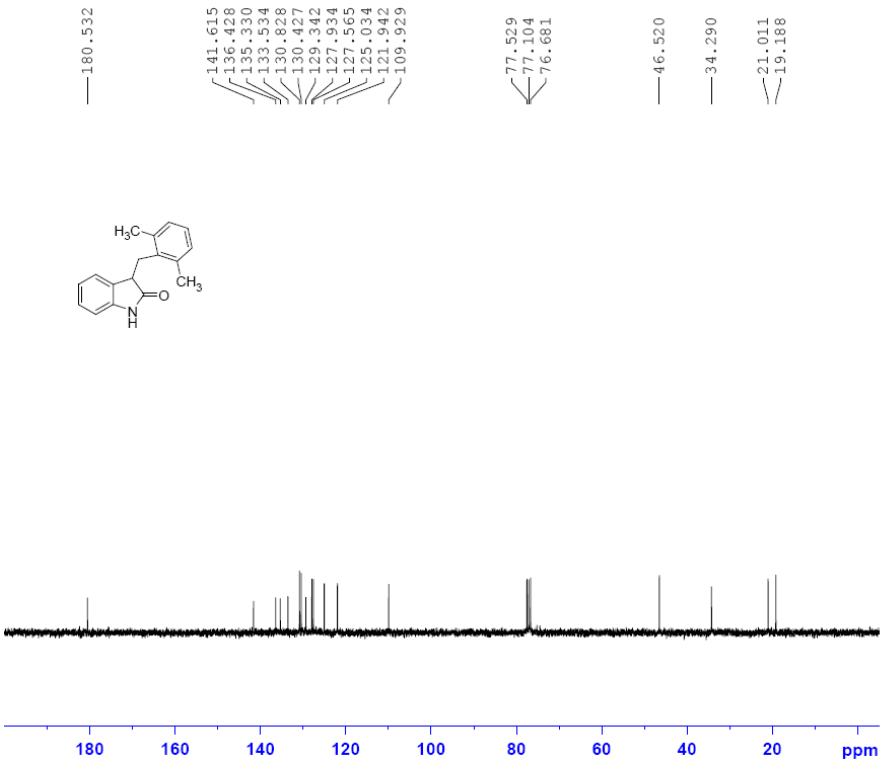



```
NAME            cl-1200
EXPNO               161
PROCNO                1
Date_          20090309
Time              22.01
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   12
DS                    4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ           1.8219508 sec
RG              1448.2
DW              27.800 usec
DE               6.00 usec
TE              673.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1               12.50 usec
PL1               2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            80.00 usec
PL2              -1.00 dB
PL12             20.16 dB
PL13             16.98 dB
SFO2        300.1312005 MHz
SI                32768
SF          75.4677490 MHz
WDW                  EM
SSB                   0
LB                1.00 Hz
GB                    0
PC                1.40
```

7.4 ^{1}H NMR and ^{13}C NMR spectrum of **1m**

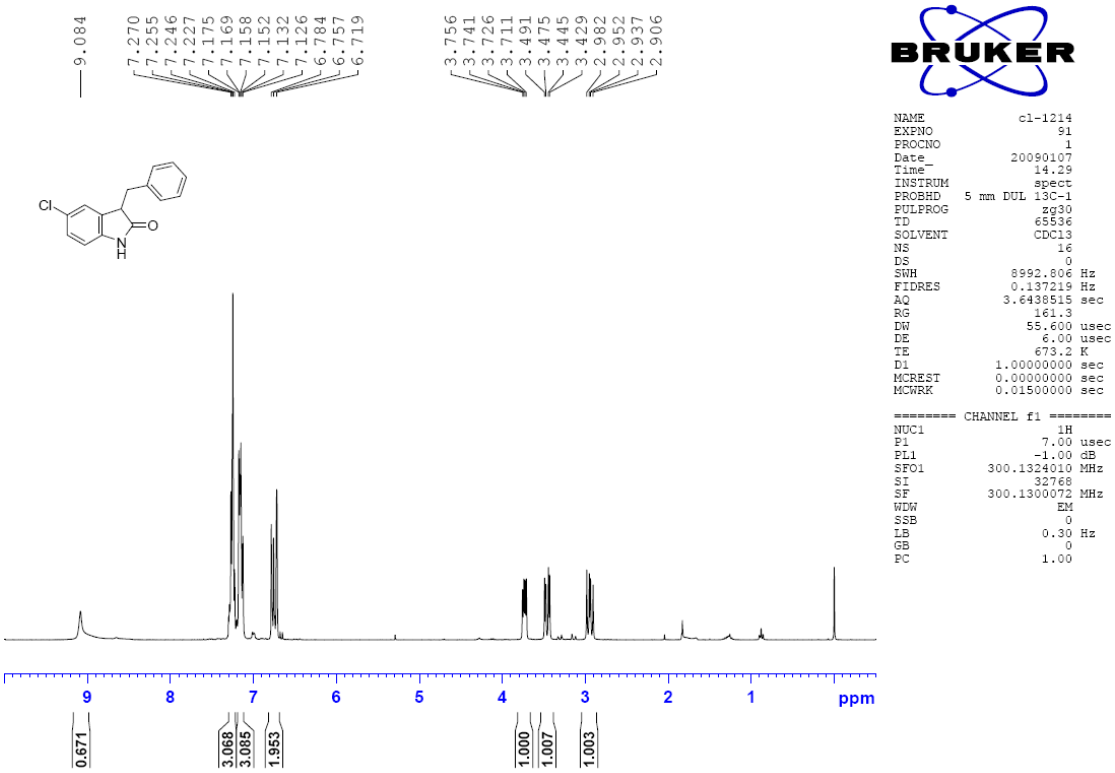


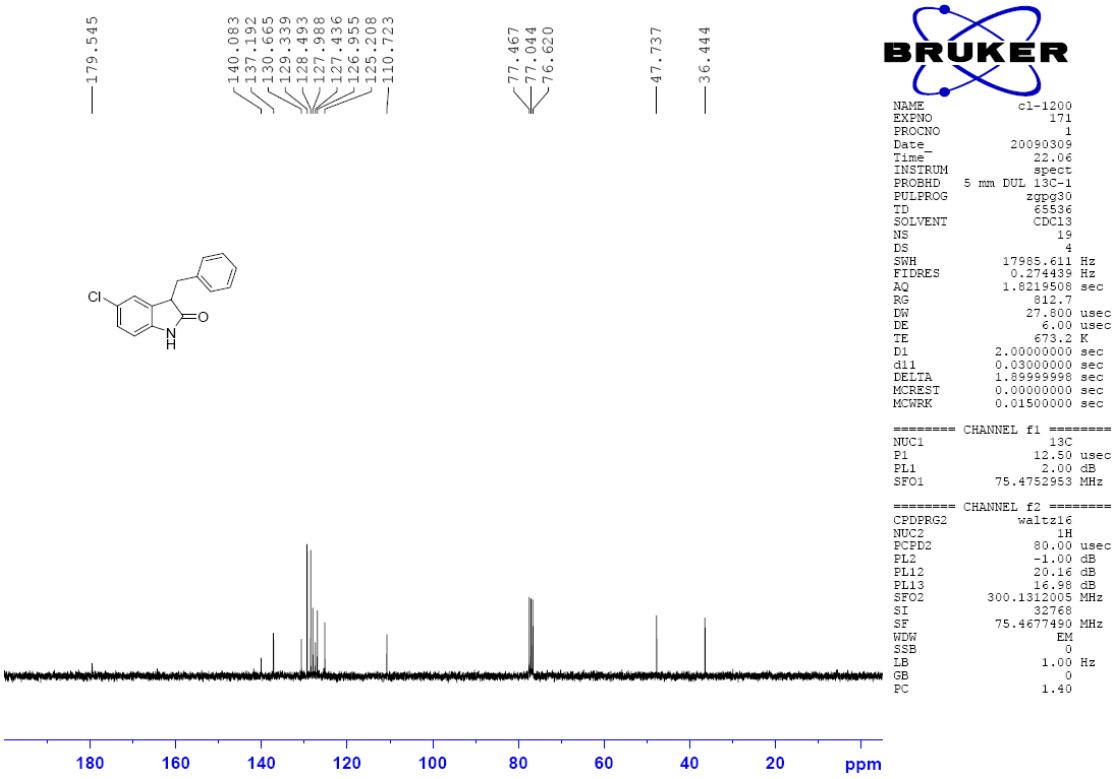

```
NAME            c1-1214
EXPNO                20
PROCNO                1
Date_          20090106
Time              21.38
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    0
SWH            8992.806 Hz
FIDRES         0.137219 Hz
AQ            3.6438515 sec
RG                 80.6
DW               55.600 usec
DE                6.00 usec
TE               673.2 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 7.00 usec
PL1               -1.00 dB
SFO1        300.1324010 MHz
SI                32768
SF          300.1300180 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```



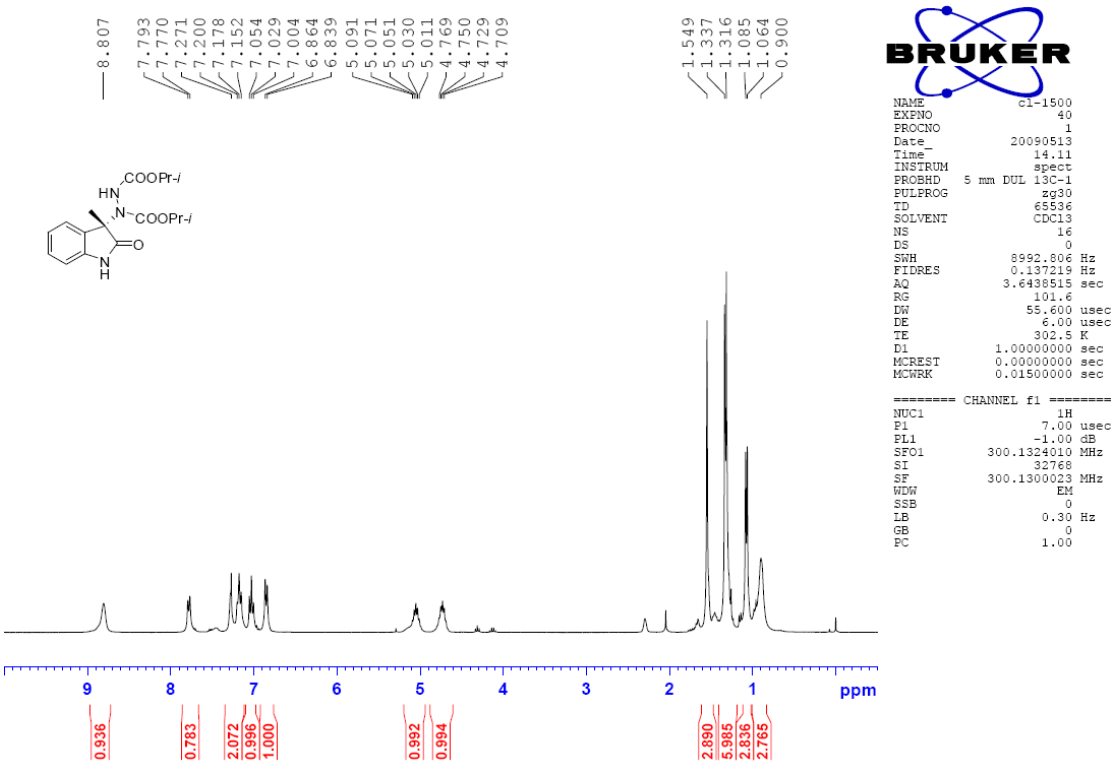

```
NAME            c1-1214
EXPNO                30
PROCNO                1
Date_          20090106
Time              21.40
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                   14
DS                    4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ            1.8219508 sec
RG               1448.2
DW               27.800 usec
DE                6.00 usec
TE               673.2 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -1.00 dB
PL12              20.16 dB
PL13              16.98 dB
SFO2        300.1312005 MHz
SI                32768
SF           75.4677490 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

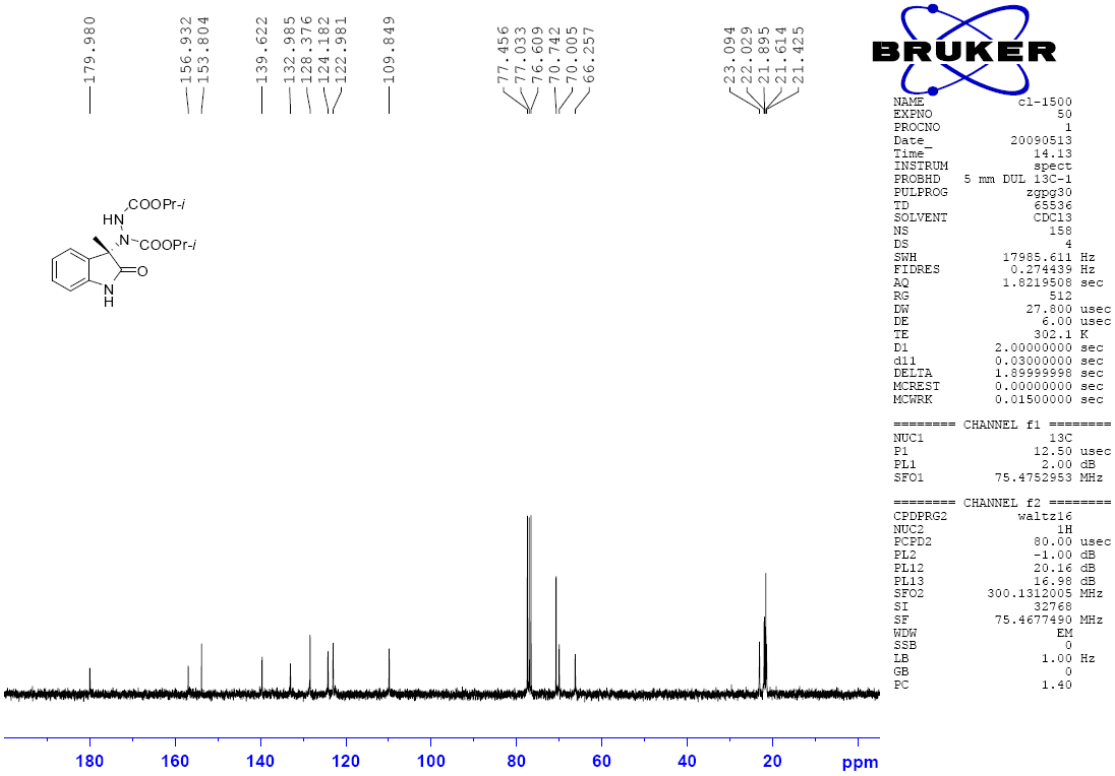
7.5 ^{1}H NMR and ^{13}C NMR spectrum of **1p**



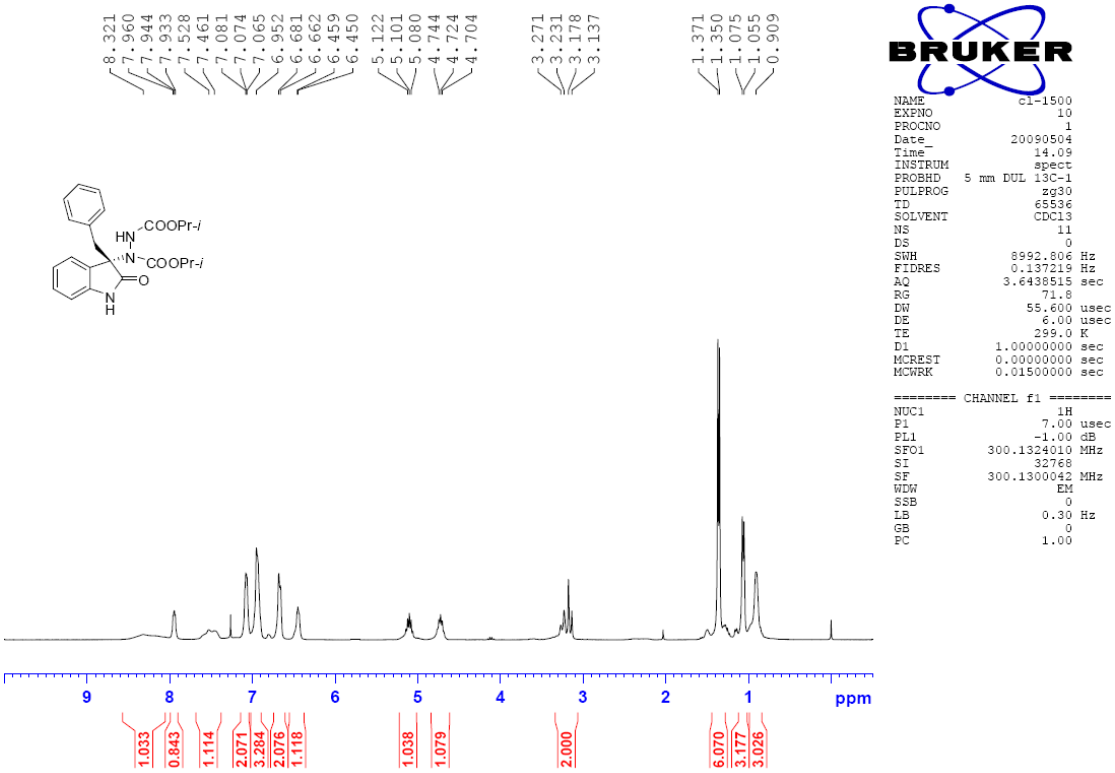


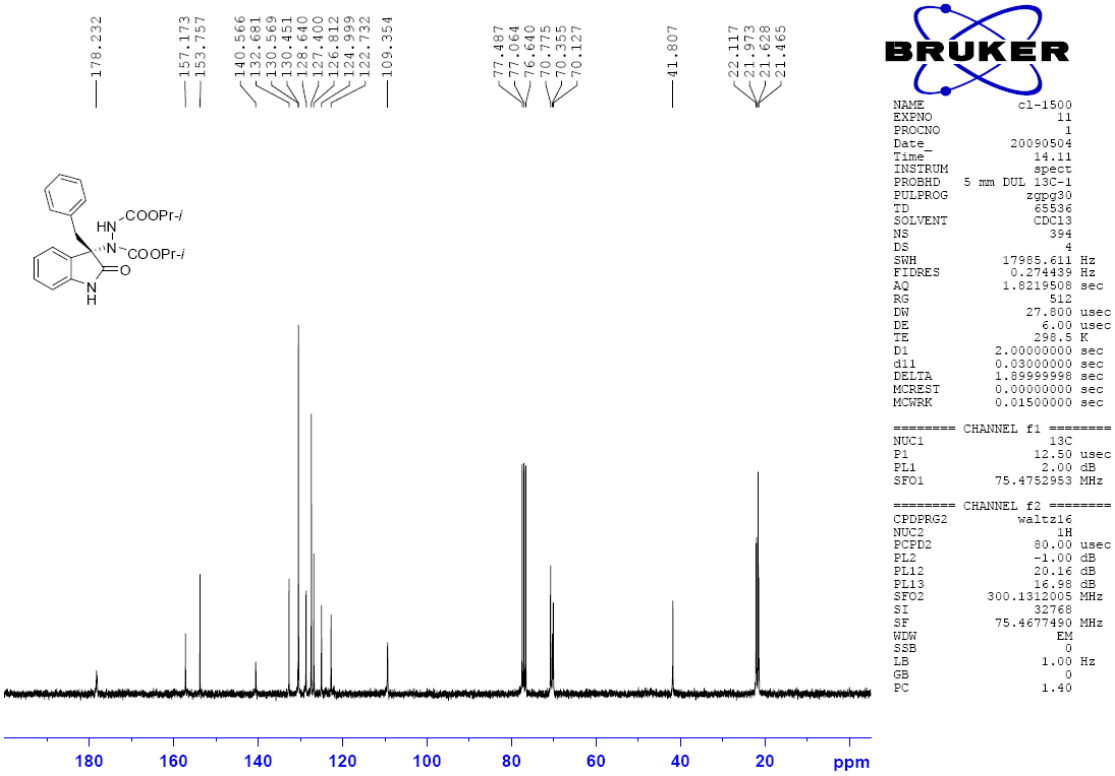
7.6 ^{1}H NMR and ^{13}C NMR spectrum of **3b**



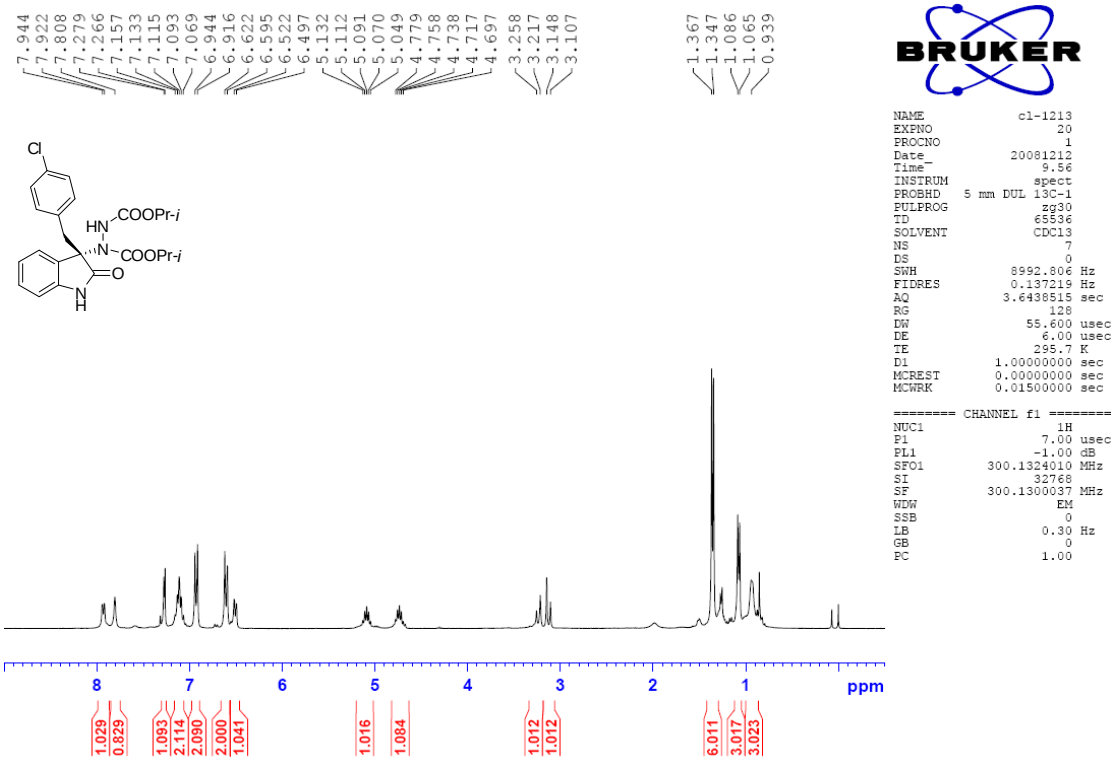


7.7 ^{1}H NMR and ^{13}C NMR spectrum of **3e**

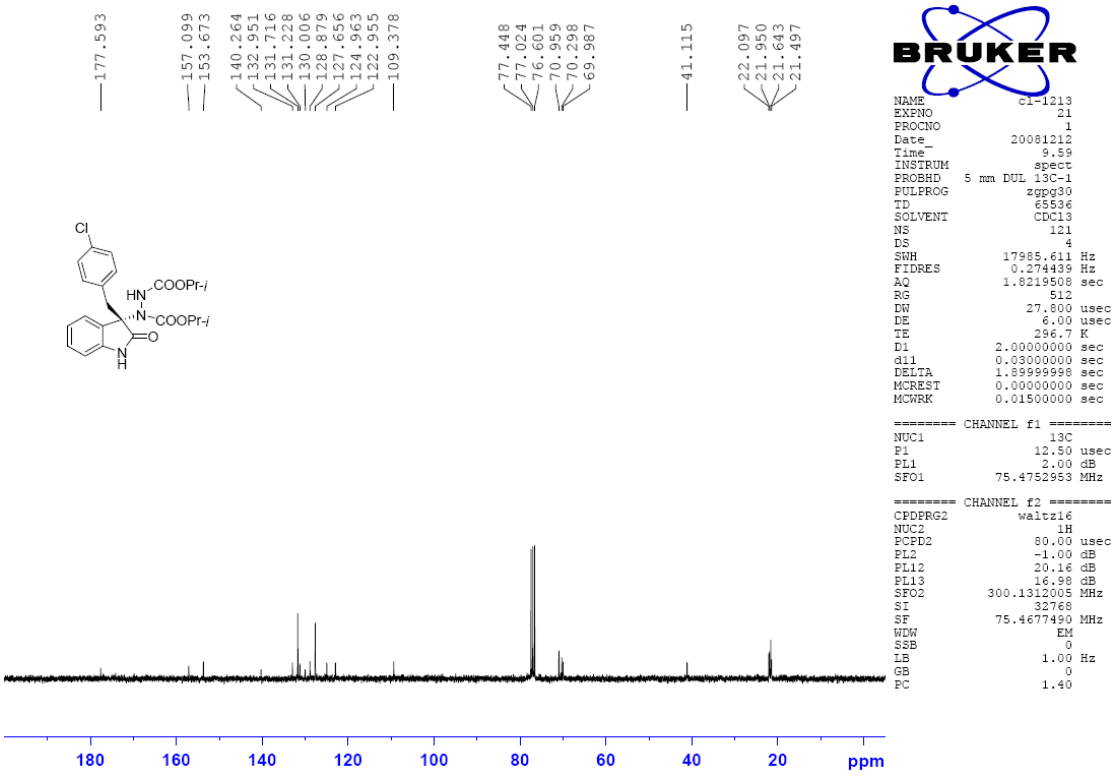




7.8 ^{1}H NMR and ^{13}C NMR spectrum of **3f**

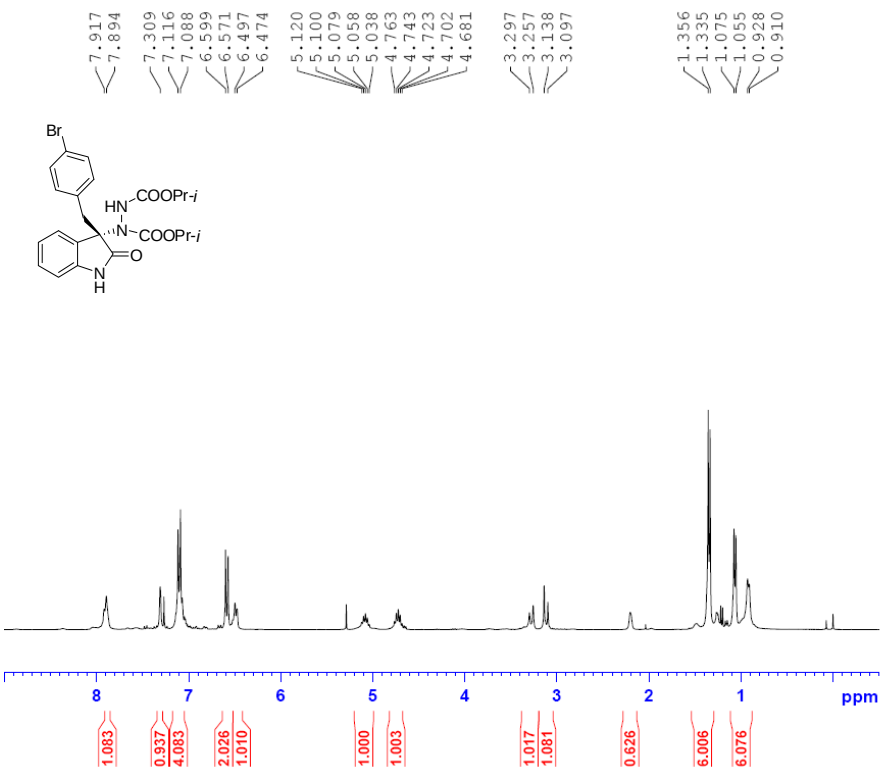



```
NAME              c1-1213
EXPNO                  20
PROCNO                  1
Date            20081212
Time                 9.56
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG              zg30
TD                  65536
SOLVENT             CDCl3
NS                      7
DS                      0
SWH              8992.806 Hz
FIDRES           0.137219 Hz
AQ              3.6438515 sec
RG                    128
DW                 55.600 usec
DE                  6.00 usec
TE                 295.7 K
D1             1.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   7.00 usec
PL1                 -1.00 dB
SFO1          300.1324010 MHz
SI                    32768
SF            300.1300037 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

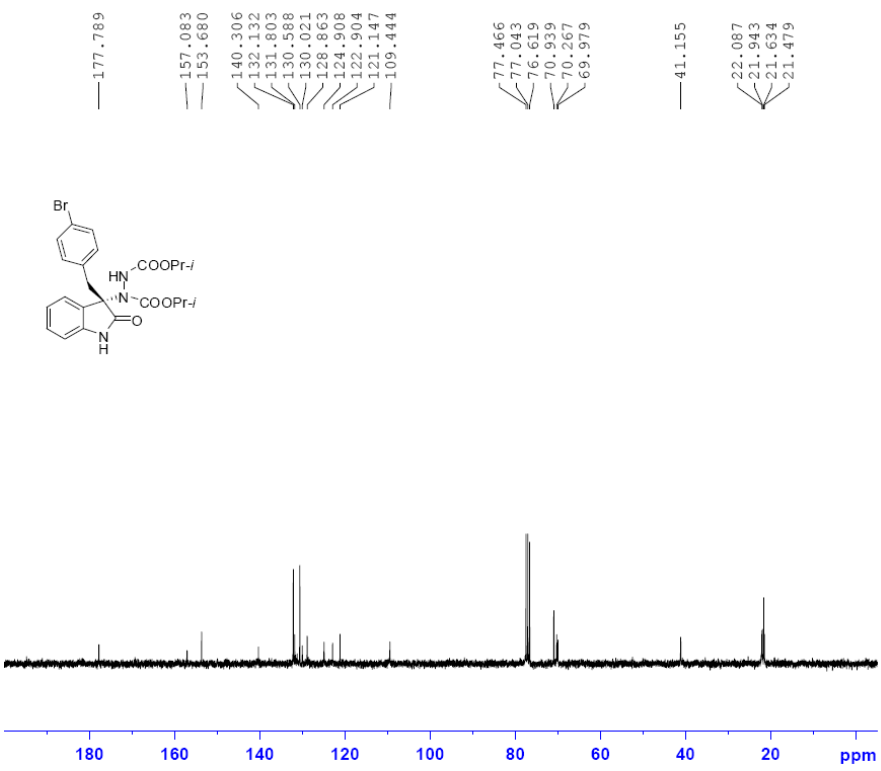



```
NAME              c1-1213
EXPNO                  21
PROCNO                  1
Date            20081212
Time                 9.59
INSTRUM             spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                  65536
SOLVENT             CDCl3
NS                    121
DS                      4
SWH             17985.611 Hz
FIDRES           0.274439 Hz
AQ              1.8219508 sec
RG                    512
DW                 27.800 usec
DE                  6.00 usec
TE                 296.7 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                  12.50 usec
PL1                  2.00 dB
SFO1           75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                 -1.00 dB
PL12                20.16 dB
PL13                16.98 dB
SFO2          300.1312005 MHz
SI                    32768
SF             75.4677490 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```

7.9 ^{1}H NMR and ^{13}C NMR spectrum of **3g**

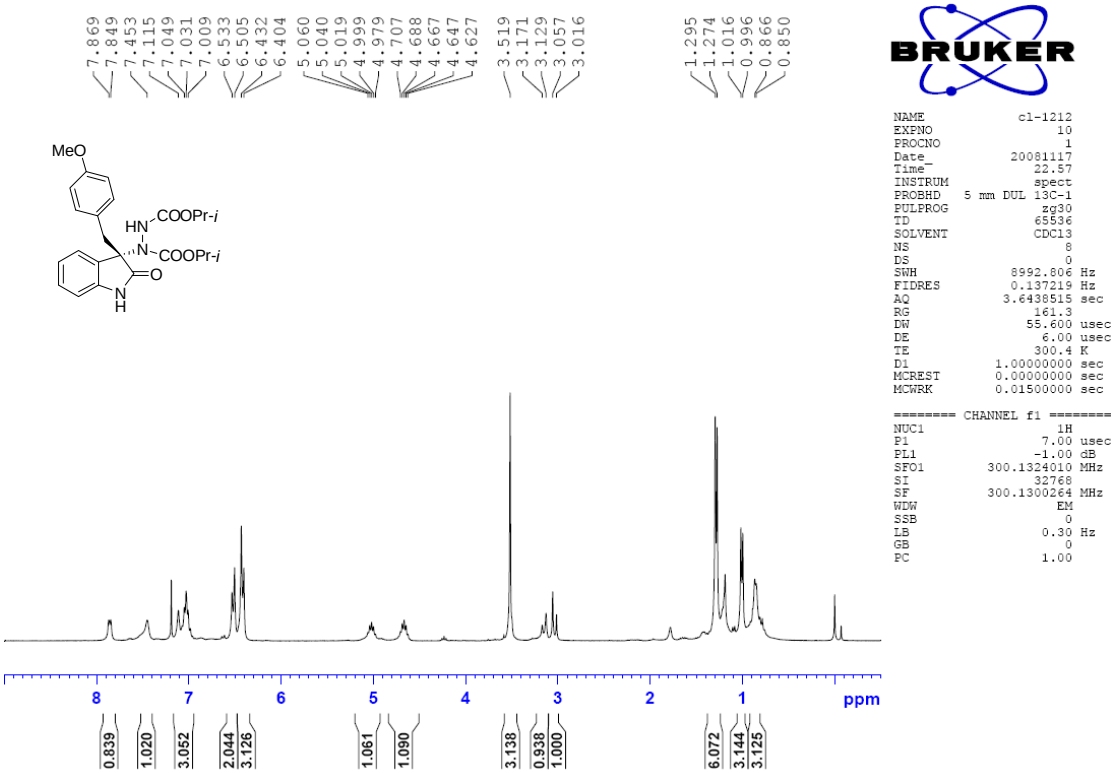



```
NAME            cl-1211
EXPNO                21
PROCNO                1
Date_          20081114
Time              18.07
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   12
DS                    0
SWH            8992.806 Hz
FIDRES         0.137219 Hz
AQ           3.6438515 sec
RG               101.6
DW              55.600 usec
DE                6.00 usec
TE               301.2 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 7.00 usec
PL1               -1.00 dB
SFO1        300.1324010 MHz
SI                32768
SF          300.1300033 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

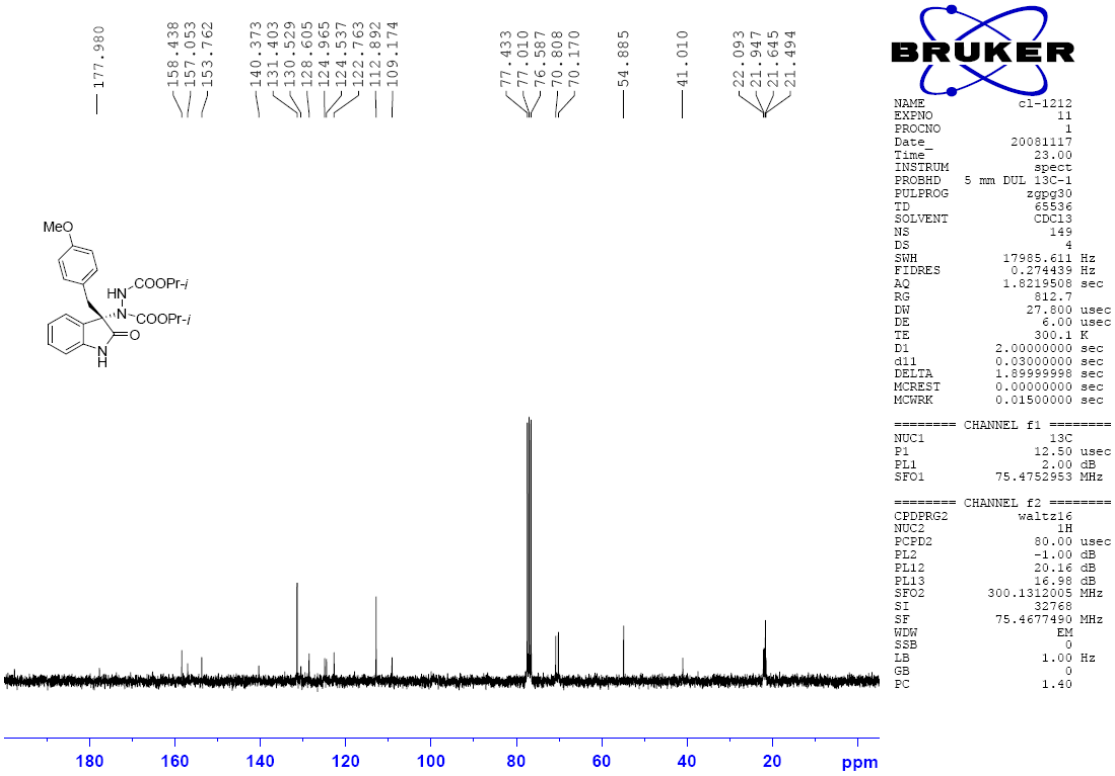



```
NAME            cl-1211
EXPNO                22
PROCNO                1
Date_          20081114
Time              18.10
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   62
DS                    4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ           1.8219508 sec
RG              3649.1
DW              27.800 usec
DE                6.00 usec
TE               300.8 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                12.50 usec
PL1                2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2               -1.00 dB
PL12              20.16 dB
PL13              16.98 dB
SFO2        300.1312005 MHz
SI                32768
SF           75.4677490 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

7.10 ^{1}H NMR and ^{13}C NMR spectrum of **3h**

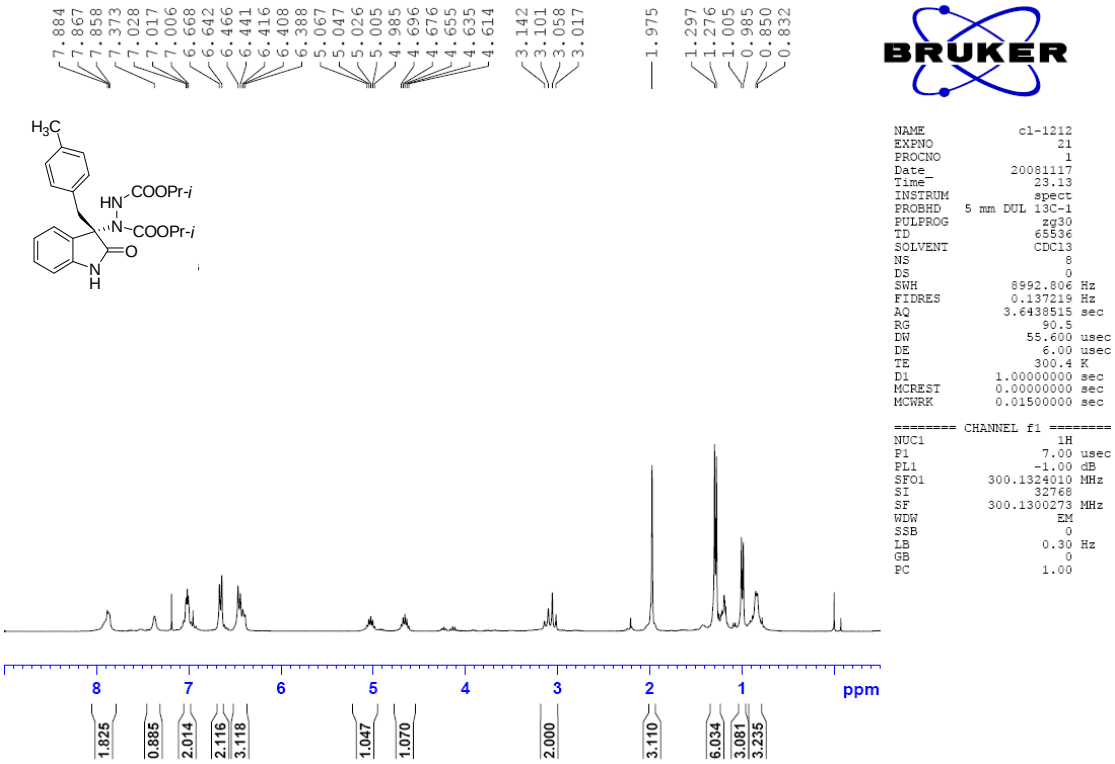


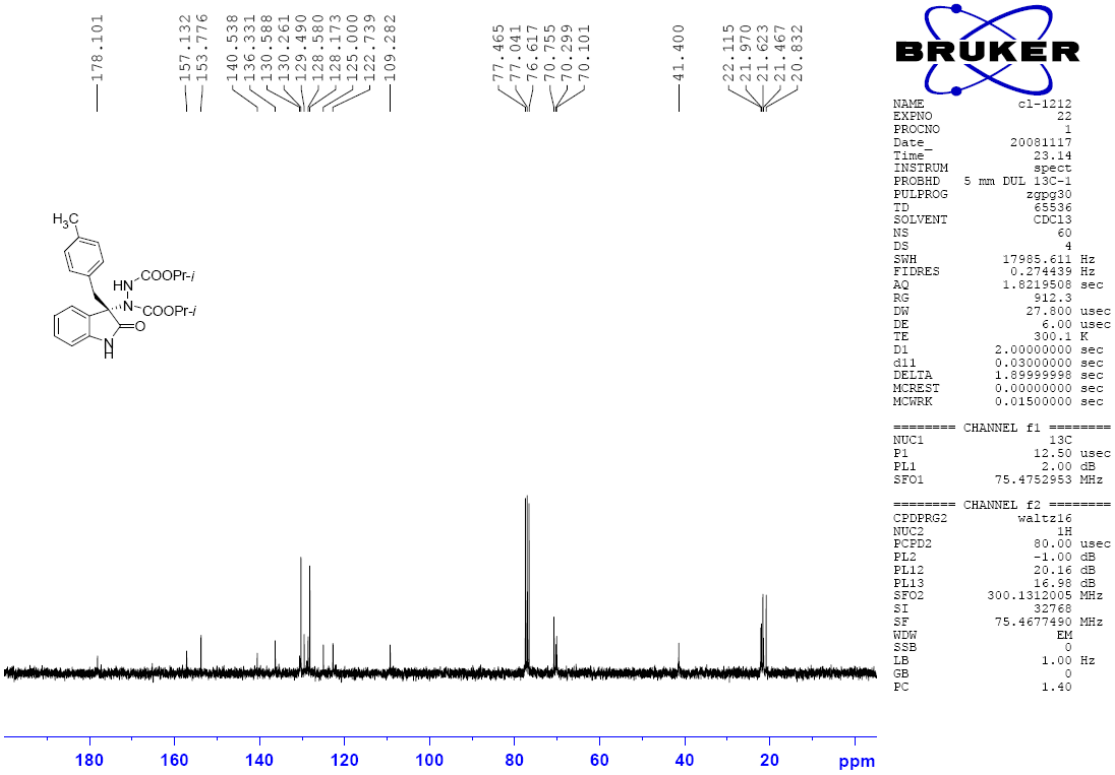

```
NAME            cl-1212
EXPNO                10
PROCNO                1
Date_          20081117
Time               22.57
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     0
SWH             8992.806 Hz
FIDRES          0.137219 Hz
AQ            3.6438515 sec
RG                 161.3
DW                55.600 usec
DE                 6.00 usec
TE                 300.4 K
D1            1.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                  7.00 usec
PL1                -1.00 dB
SFO1         300.1324010 MHz
SI                 32768
SF           300.1300264 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



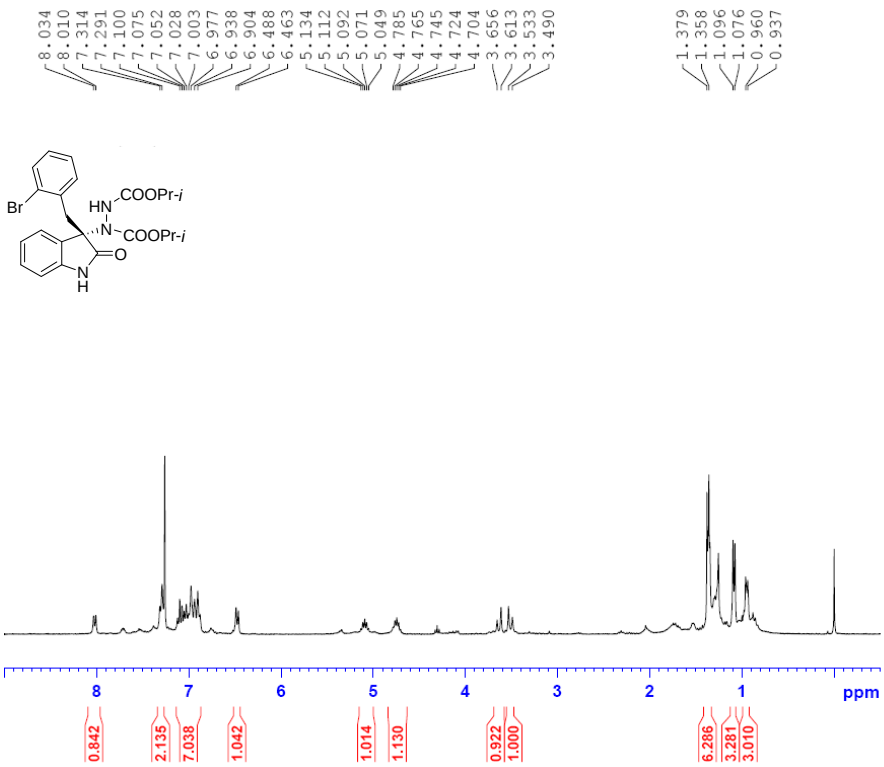

```
NAME            cl-1212
EXPNO                11
PROCNO                1
Date_          20081117
Time               23.00
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG           zgpg30
TD                 65536
SOLVENT            CDCl3
NS                   149
DS                     4
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ            1.8219508 sec
RG                 812.7
DW                27.800 usec
DE                 6.00 usec
TE                 300.1 K
D1            2.00000000 sec
d11           0.03000000 sec
DELTA         1.89999998 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 12.50 usec
PL1                 2.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -1.00 dB
PL12               20.16 dB
PL13               16.98 dB
SFO2         300.1312005 MHz
SI                 32768
SF            75.4677490 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

7.11 ^{1}H NMR and ^{13}C NMR spectrum of **3i**

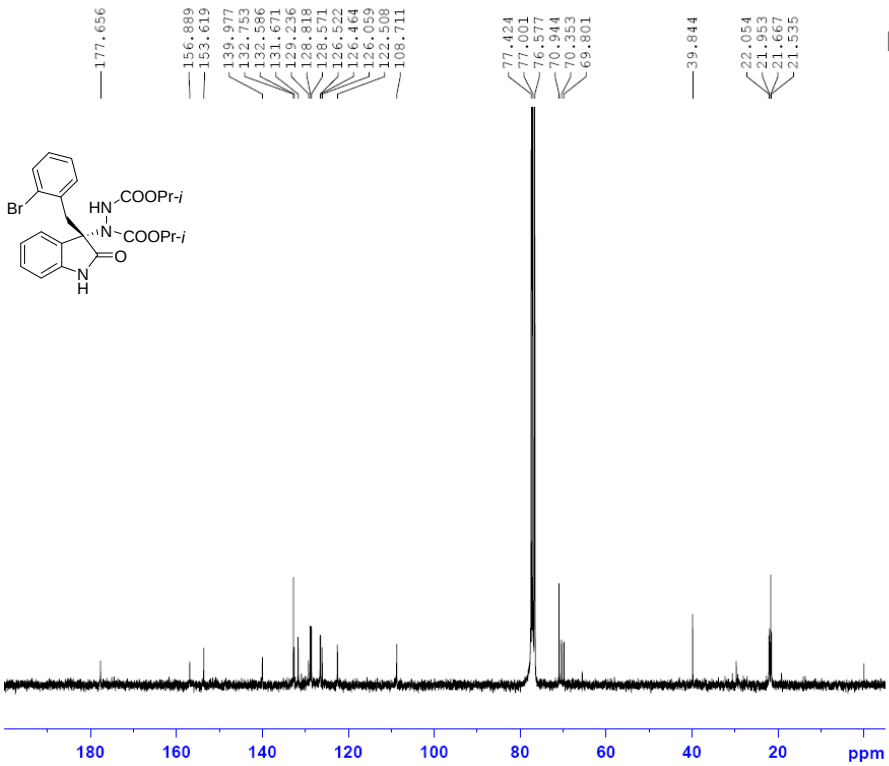




7.12 ^{1}H NMR and ^{13}C NMR spectrum of **3j**

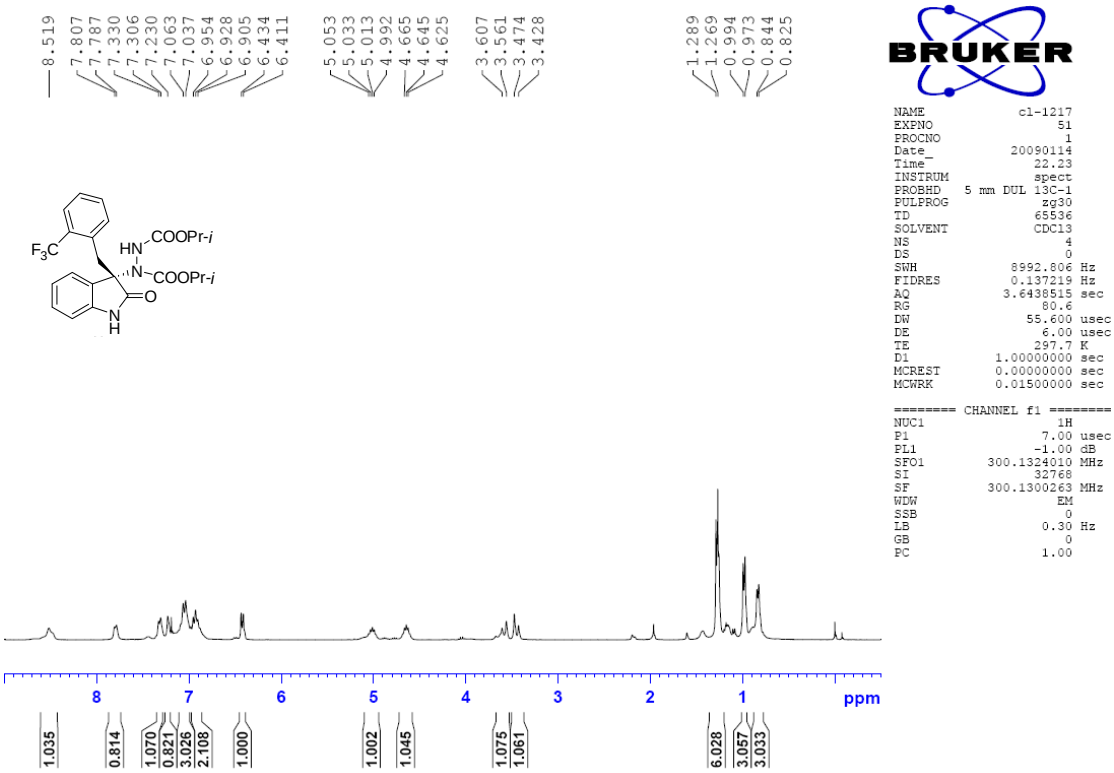


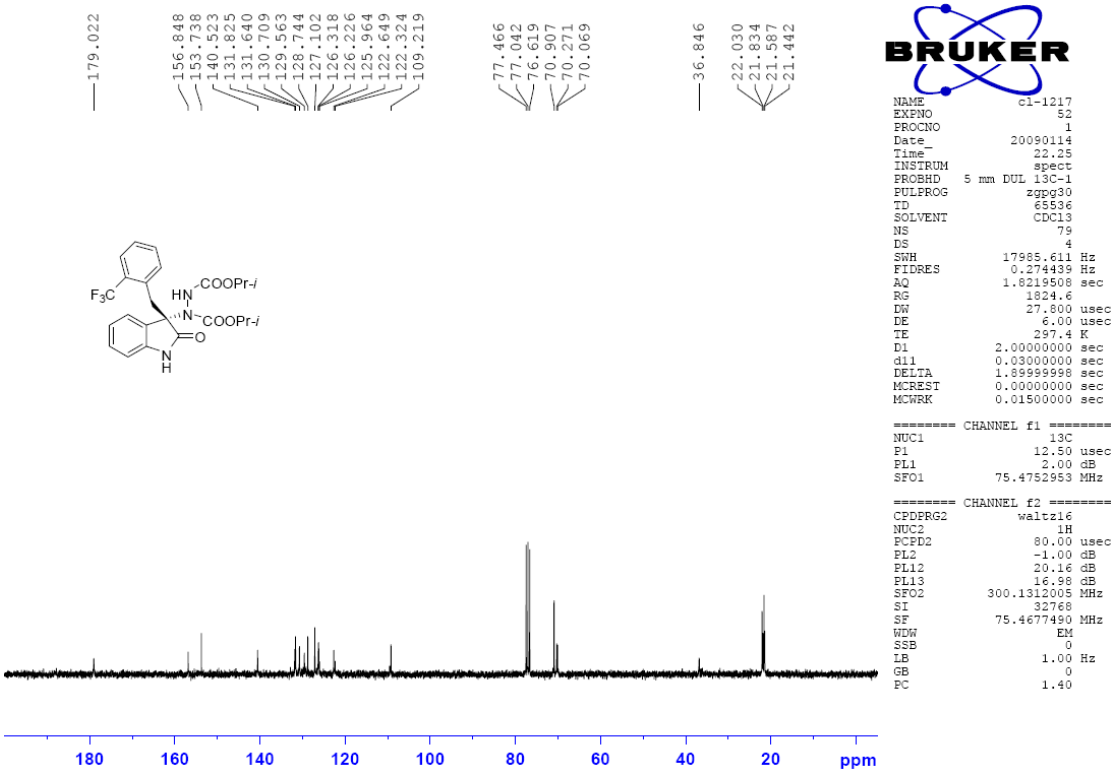

```
NAME            cl-1213
EXPNO                30
PROCNO                1
Date_          20081215
Time               20.25
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     9
DS                     0
SWH             8992.806 Hz
FIDRES          0.137219 Hz
AQ             3.6438515 sec
RG                   362
DW                55.600 usec
DE                  6.00 usec
TE                 296.2 K
D1             1.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                  7.00 usec
PL1                -1.00 dB
SFO1          300.1324010 MHz
SI                 32768
SF            300.1300052 MHz
WDW                   EM
SSB                    0
LB                  0.30 Hz
GB                     0
PC                  1.00
```



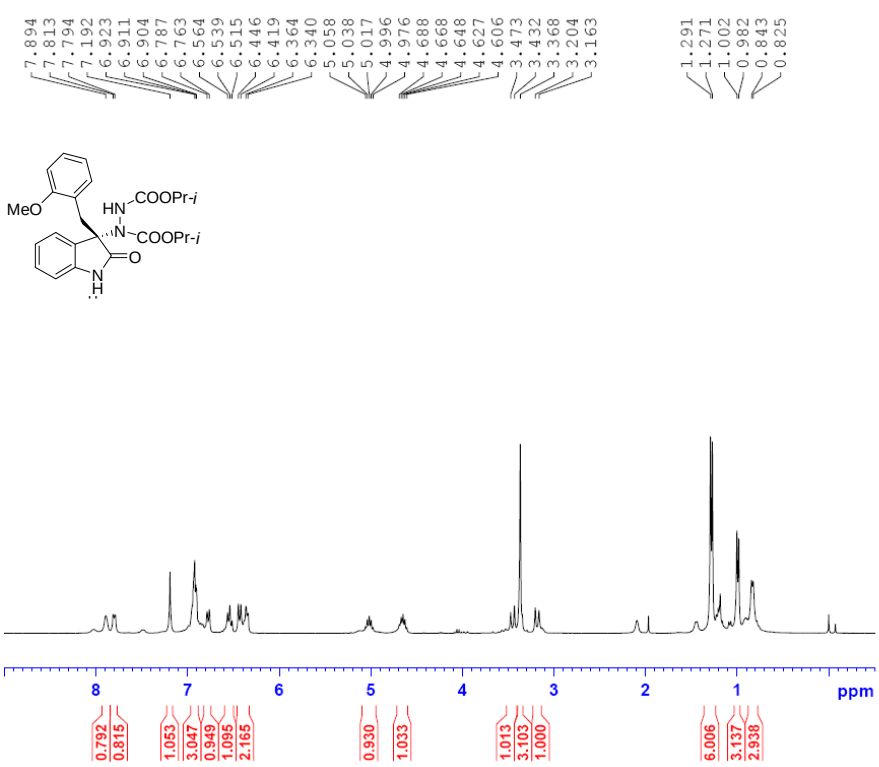

```
NAME            cl-1213
EXPNO                60
PROCNO                1
Date_          20081216
Time                7.58
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                  7839
DS                     4
SWH            17985.611 Hz
FIDRES          0.274439 Hz
AQ             1.8219508 sec
RG                 574.7
DW                27.800 usec
DE                  6.00 usec
TE                 297.6 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                 12.50 usec
PL1                 2.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                -1.00 dB
PL12               20.16 dB
PL13               16.98 dB
SFO2          300.1312005 MHz
SI                 32768
SF             75.4677490 MHz
WDW                   EM
SSB                    0
LB                  1.00 Hz
GB                     0
PC                  1.40
```

7.13 ^{1}H NMR and ^{13}C NMR spectrum of **3k**

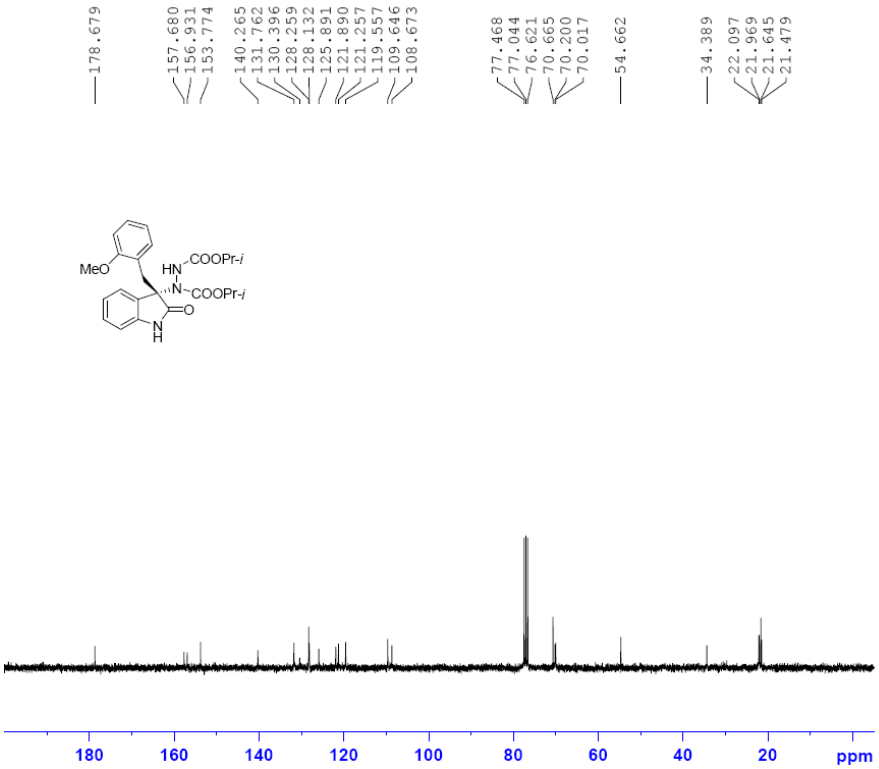




7.14 ^{1}H NMR and ^{13}C NMR spectrum of **3l**

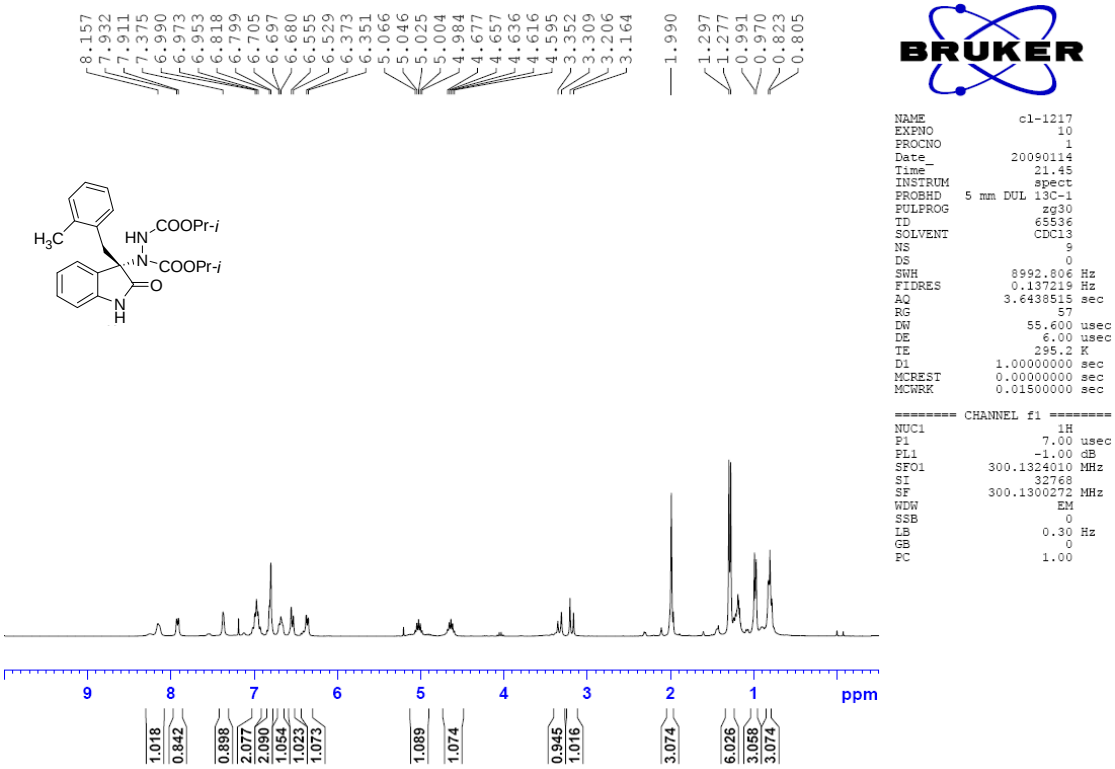


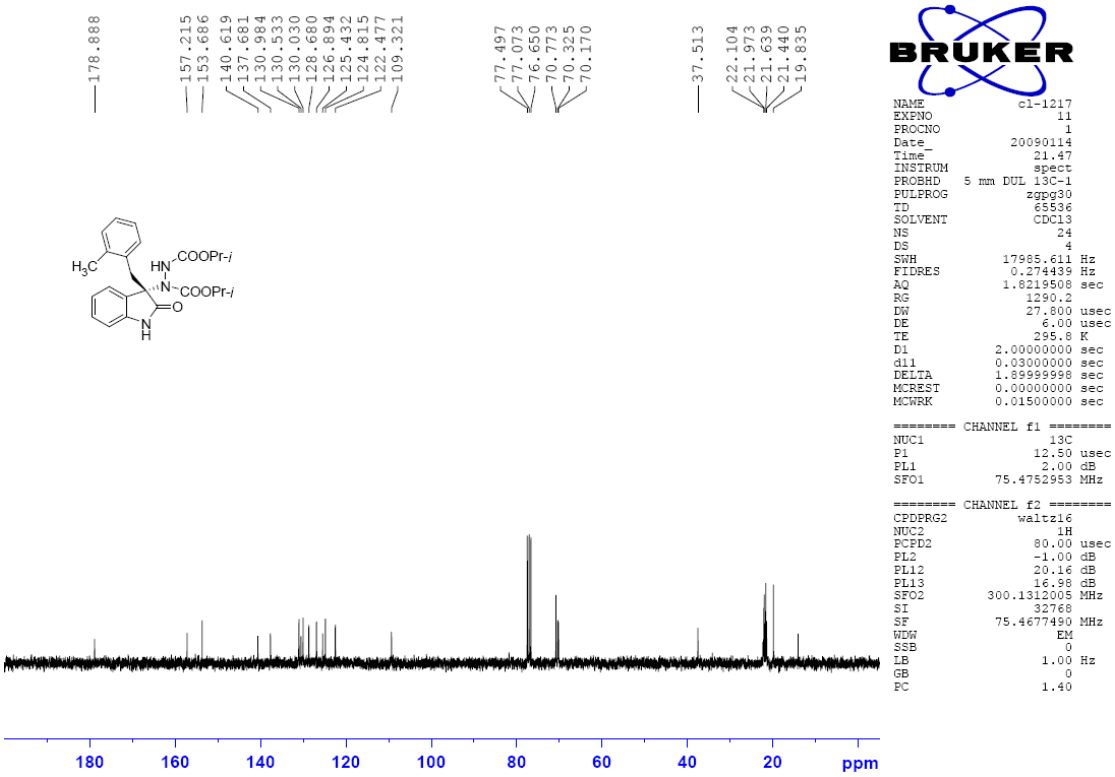

```
NAME            cl-1213
EXPNO                31
PROCNO                1
Date_          20081212
Time              10.09
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                   12
DS                    0
SWH            8992.806 Hz
FIDRES         0.137219 Hz
AQ           3.6438515 sec
RG                90.5
DW              55.600 usec
DE               6.00 usec
TE              297.4 K
D1          1.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                7.00 usec
PL1              -1.00 dB
SFO1        300.1324010 MHz
SI                32768
SF          300.1300258 MHz
WDW                  EM
SSB                   0
LB                0.30 Hz
GB                    0
PC                1.00
```



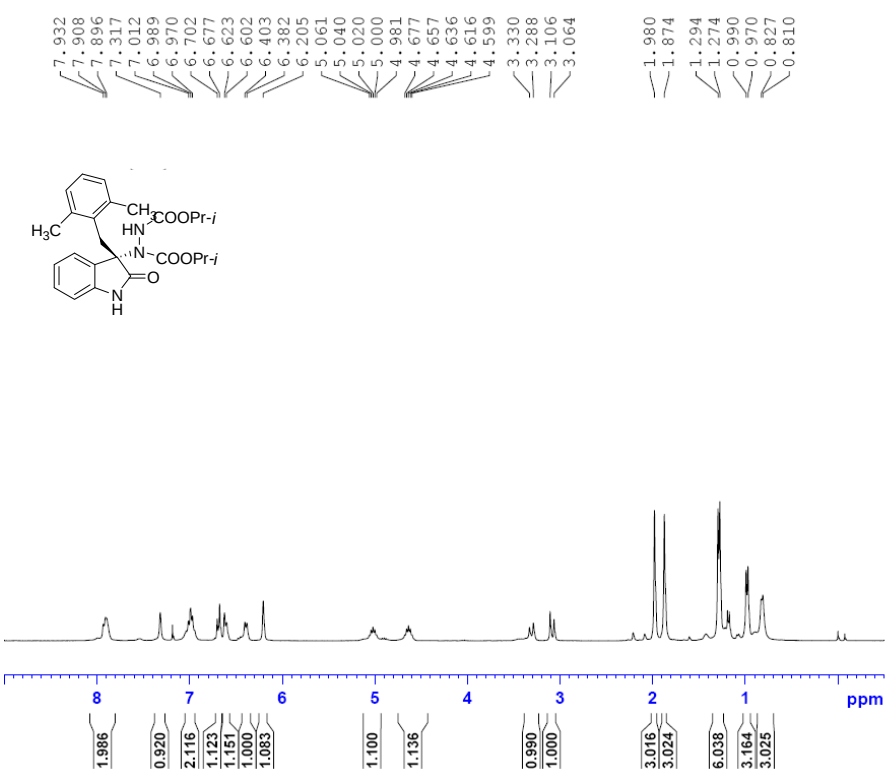

```
NAME            cl-1213
EXPNO                32
PROCNO                1
Date_          20081212
Time              10.12
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                   77
DS                    4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ           1.8219508 sec
RG               1448.2
DW              27.800 usec
DE               6.00 usec
TE              297.0 K
D1          2.00000000 sec
d11         0.03000000 sec
DELTA       1.89999998 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1               12.50 usec
PL1               2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                 1H
PCPD2            80.00 usec
PL2              -1.00 dB
PL12             20.16 dB
PL13             16.98 dB
SFO2        300.1312005 MHz
SI                32768
SF          75.4677490 MHz
WDW                  EM
SSB                   0
LB                1.00 Hz
GB                    0
PC                1.40
```

7.15 ^{1}H NMR and ^{13}C NMR spectrum of **3m**

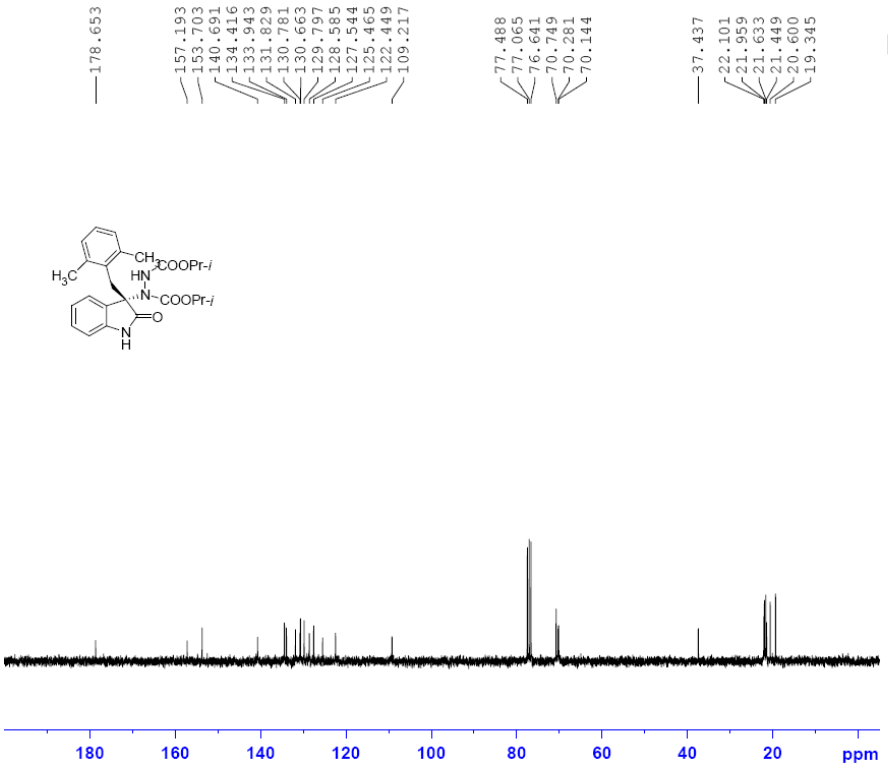




7.16 ^{1}H NMR and ^{13}C NMR spectrum of **3n**

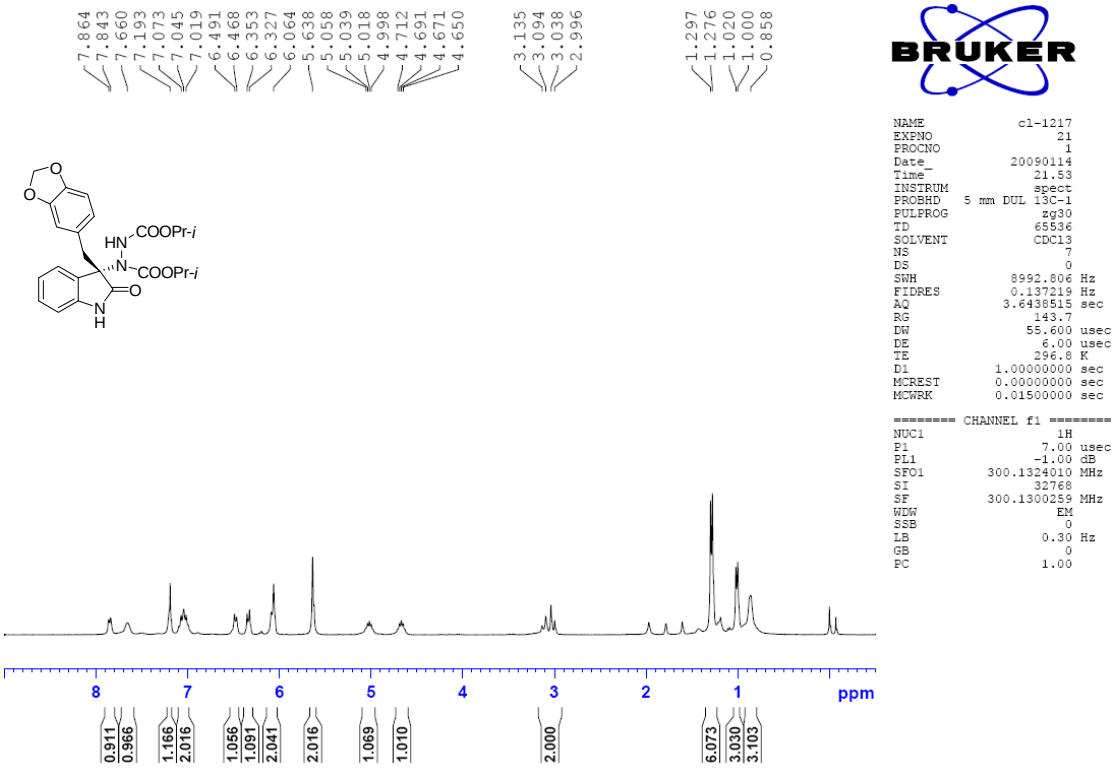


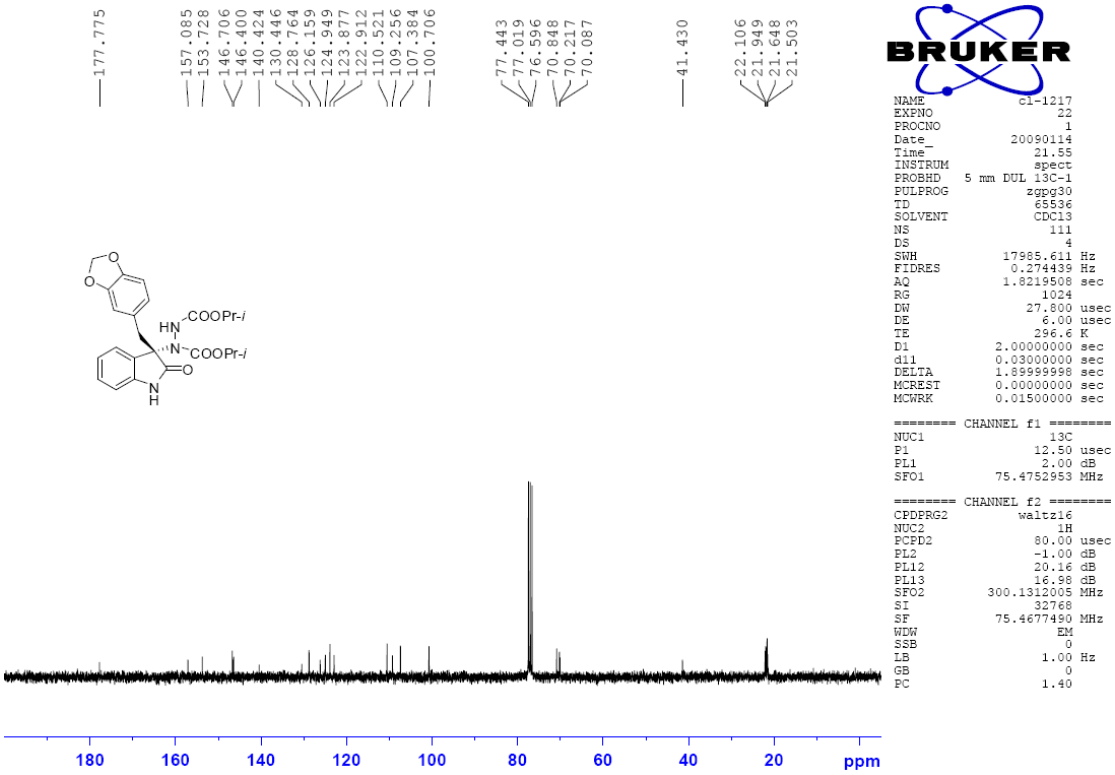

```
NAME                cl-1217
EXPNO                    60
PROCNO                    1
Date_            20090114
Time                 22.55
INSTRUM              spect
PROBHD    5 mm DUL 13C-1
PULPROG               zg30
TD                    65536
SOLVENT               CDCl3
NS                        4
DS                        0
SWH               8992.806 Hz
FIDRES            0.137219 Hz
AQ               3.6438515 sec
RG                     71.8
DW                   55.600 usec
DE                     6.00 usec
TE                    297.8 K
D1               1.00000000 sec
MCREST           0.00000000 sec
MCWRK            0.01500000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                     7.00 usec
PL1                   -1.00 dB
SFO1            300.1324010 MHz
SI                    32768
SF              300.1300280 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```



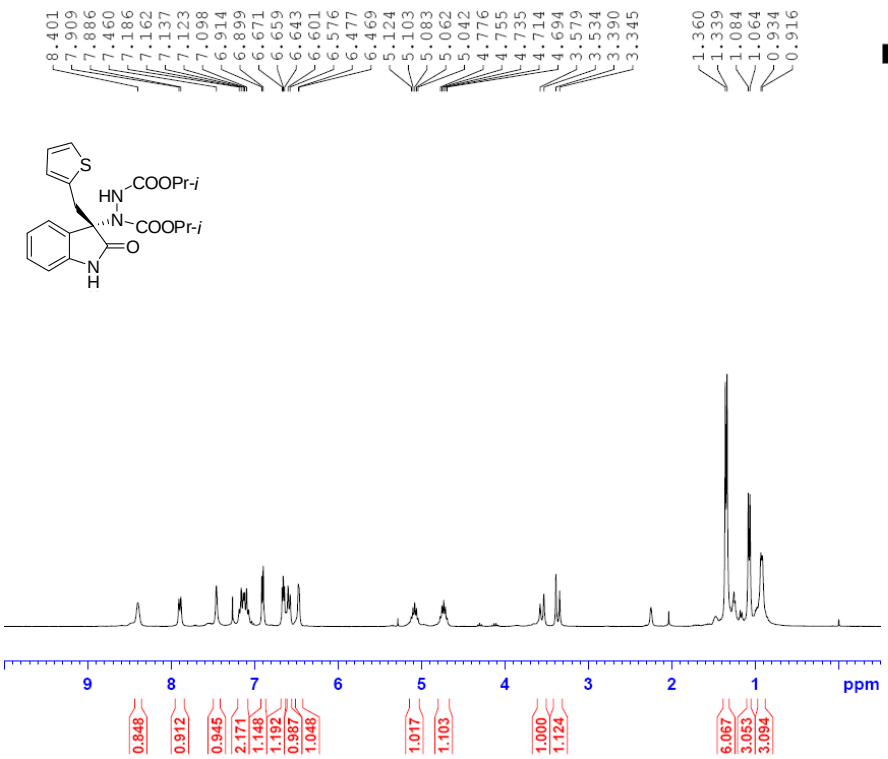

```
NAME                cl-1217
EXPNO                    63
PROCNO                    1
Date_            20090114
Time                 22.57
INSTRUM              spect
PROBHD    5 mm DUL 13C-1
PULPROG             zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       37
DS                        4
SWH              17985.611 Hz
FIDRES            0.274439 Hz
AQ               1.8219508 sec
RG                    574.7
DW                   27.800 usec
DE                     6.00 usec
TE                    297.3 K
D1               2.00000000 sec
d11              0.03000000 sec
DELTA            1.89999998 sec
MCREST           0.00000000 sec
MCWRK            0.01500000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                    12.50 usec
PL1                    2.00 dB
SFO1            75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                 80.00 usec
PL2                   -1.00 dB
PL12                  20.16 dB
PL13                  16.98 dB
SFO2            300.1312005 MHz
SI                    32768
SF               75.4677490 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

7.17 ^{1}H NMR and ^{13}C NMR spectrum of **3o**

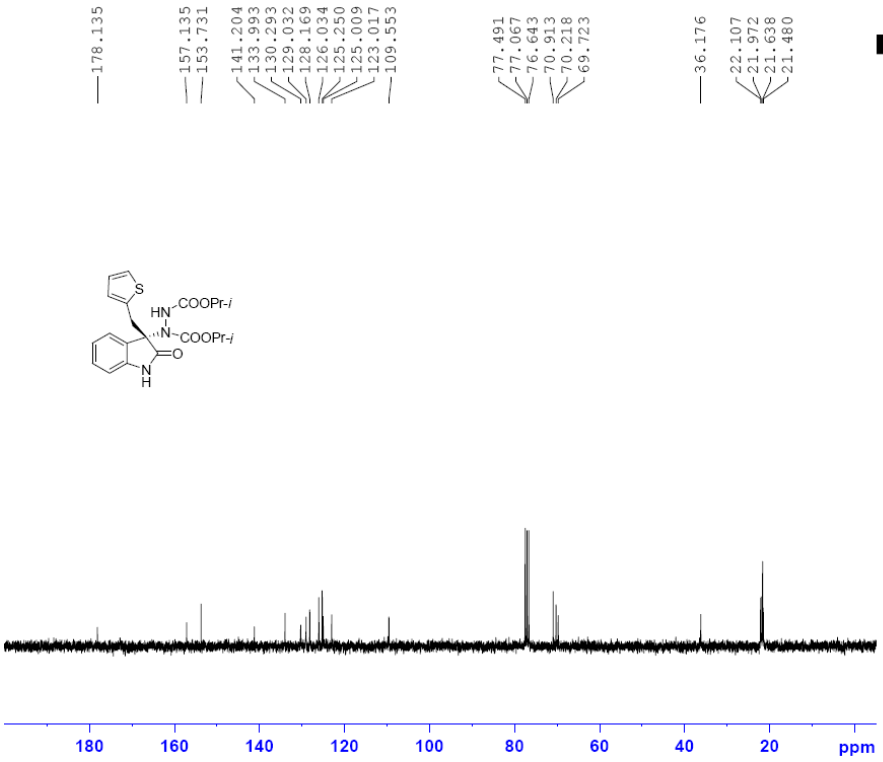




7.18 ^{1}H NMR and ^{13}C NMR spectrum of **3p**

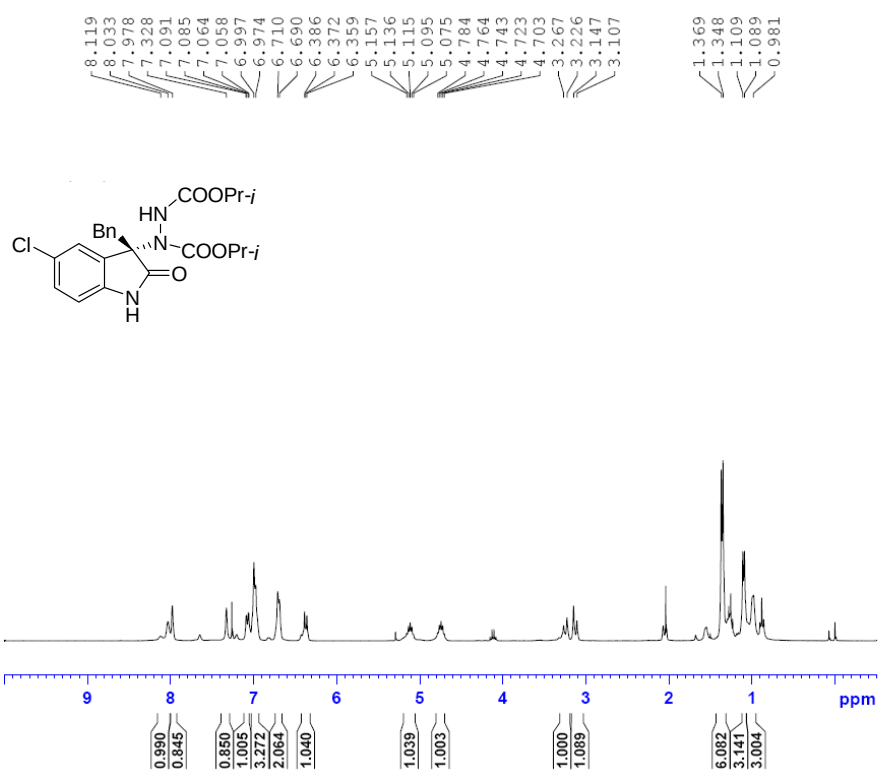



```
NAME            cl-1213
EXPNO                41
PROCNO                1
Date_          20081212
Time              10.20
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    6
DS                    0
SWH            8992.806 Hz
FIDRES         0.137219 Hz
AQ            3.6438515 sec
RG                 80.6
DW               55.600 usec
DE                6.00 usec
TE                297.5 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                7.00 usec
PL1              -1.00 dB
SFO1        300.1324010 MHz
SI                32768
SF          300.1300036 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

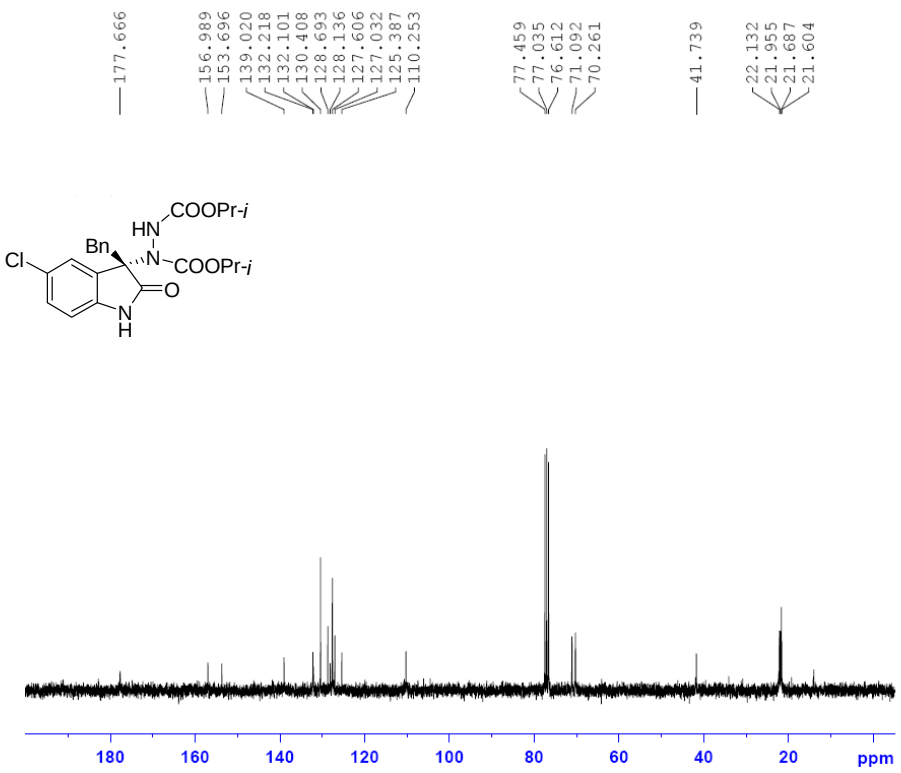



```
NAME            cl-1213
EXPNO                42
PROCNO                1
Date_          20081212
Time              10.22
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG          zgpg30
TD                65536
SOLVENT           CDCl3
NS                   25
DS                    4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ            1.8219508 sec
RG                912.3
DW               27.800 usec
DE                6.00 usec
TE                297.0 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1               12.50 usec
PL1               2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            80.00 usec
PL2              -1.00 dB
PL12             20.16 dB
PL13             16.98 dB
SFO2        300.1312005 MHz
SI                32768
SF          75.4677490 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

7.19 ^{1}H NMR and ^{13}C NMR spectrum of **3q**

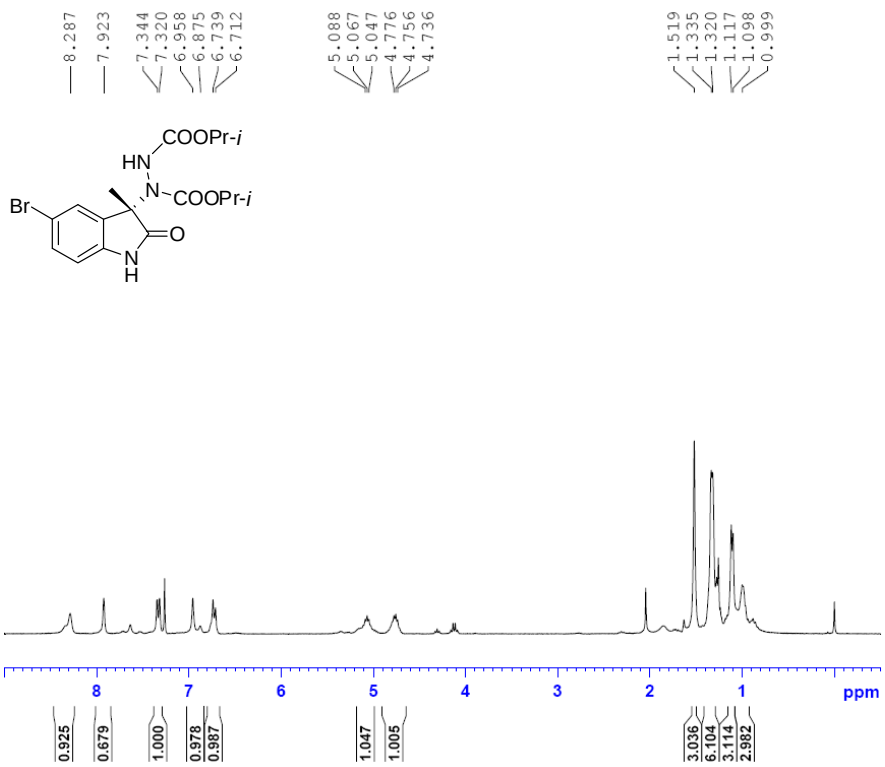



```
NAME                cl-1217
EXPNO                    31
PROCNO                    1
Date_            20090114
Time                 22.05
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG               zg30
TD                    65536
SOLVENT               CDCl3
NS                        7
DS                        0
SWH               8992.806 Hz
FIDRES            0.137219 Hz
AQ                3.6438515 sec
RG                      114
DW                   55.600 usec
DE                    6.00 usec
TE                   297.2 K
D1              1.00000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    7.00 usec
PL1                  -1.00 dB
SFO1            300.1324010 MHz
SI                    32768
SF              300.1300049 MHz
WDW                      EM
SSB                       0
LB                     0.30 Hz
GB                        0
PC                     1.00
```

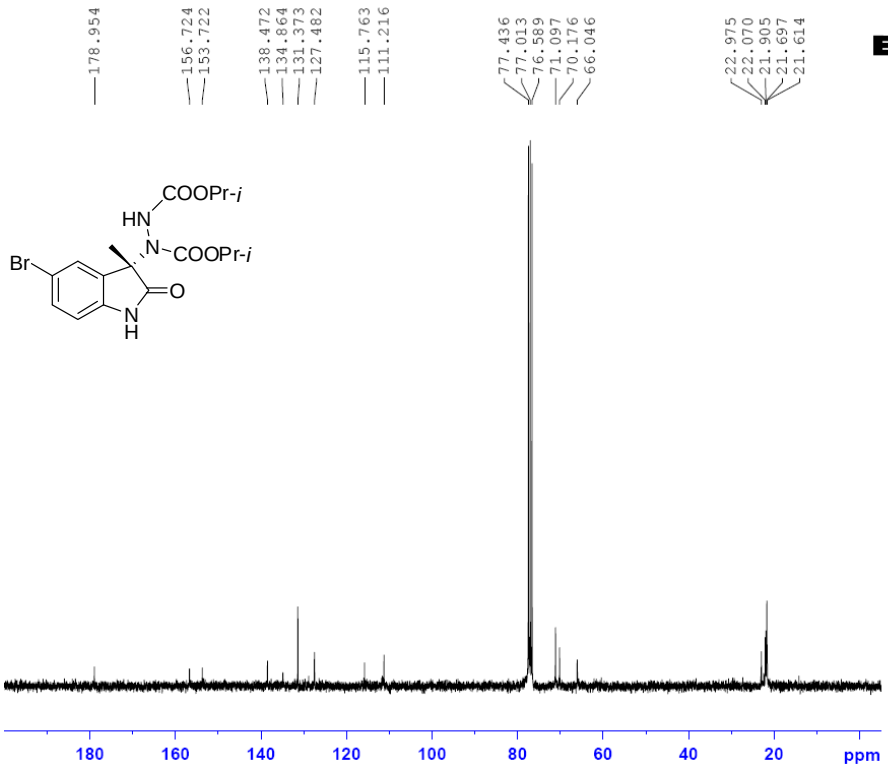



```
NAME                cl-1217
EXPNO                    32
PROCNO                    1
Date_            20090114
Time                 22.08
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG            zgpg30
TD                    65536
SOLVENT               CDCl3
NS                       81
DS                        4
SWH              17985.611 Hz
FIDRES            0.274439 Hz
AQ                1.8219508 sec
RG                      512
DW                   27.800 usec
DE                    6.00 usec
TE                   297.1 K
D1              2.00000000 sec
d11             0.03000000 sec
DELTA           1.89999998 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                   12.50 usec
PL1                   2.00 dB
SFO1            75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                     1H
PCPD2                80.00 usec
PL2                  -1.00 dB
PL12                 20.16 dB
PL13                 16.98 dB
SFO2            300.1312005 MHz
SI                    32768
SF              75.4677490 MHz
WDW                      EM
SSB                       0
LB                     1.00 Hz
GB                        0
PC                     1.40
```

7.20 ^{1}H NMR and ^{13}C NMR spectrum of **3r**

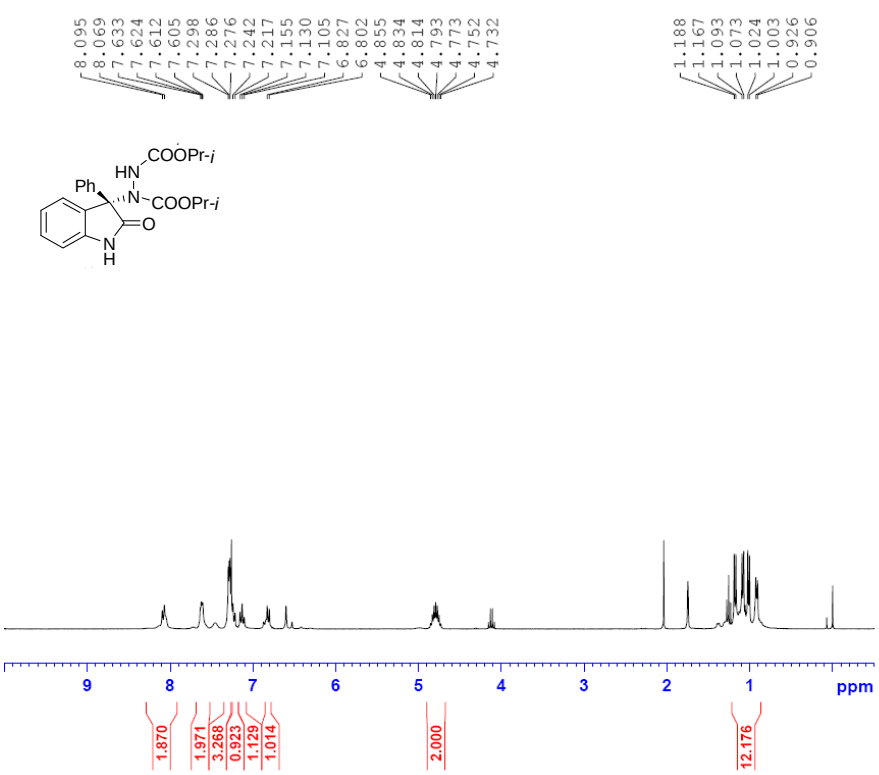



```
NAME            cl-1213
EXPNO                10
PROCNO                1
Date_          20081211
Time              10.14
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    5
DS                    0
SWH            8992.806 Hz
FIDRES         0.137219 Hz
AQ           3.6438515 sec
RG                  362
DW               55.600 usec
DE                6.00 usec
TE               297.5 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                7.00 usec
PL1              -1.00 dB
SFO1        300.1324010 MHz
SI                32768
SF          300.1300044 MHz
WDW                  EM
SSB                   0
LB                 0.30 Hz
GB                    0
PC                 1.00
```

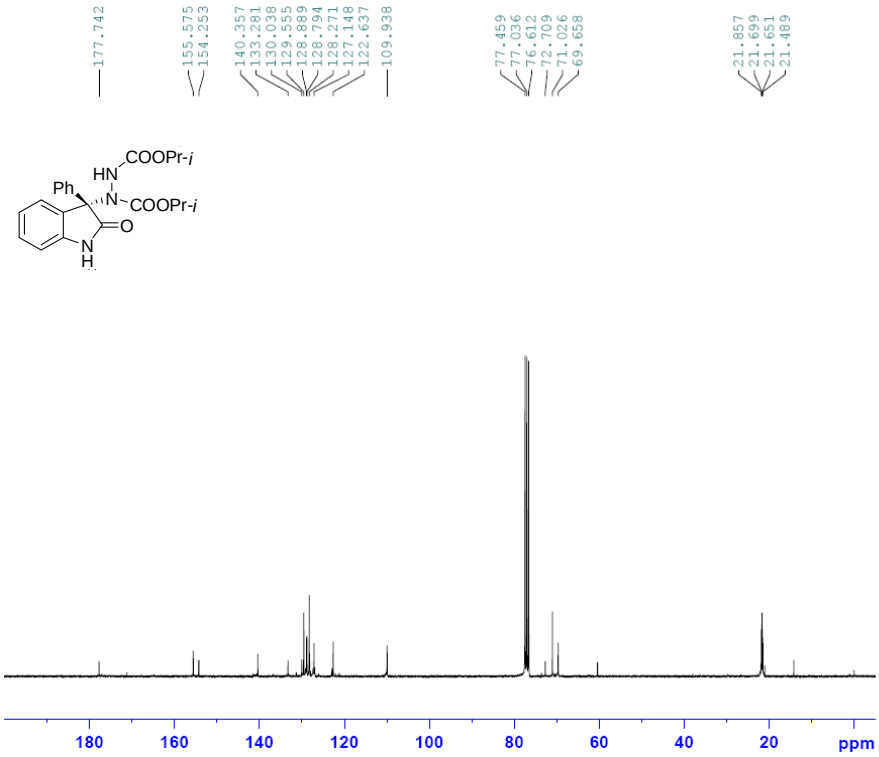



```
NAME            cl-1213
EXPNO                11
PROCNO                1
Date_          20081211
Time              10.16
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG         zgpg30
TD                65536
SOLVENT           CDCl3
NS                  973
DS                    4
SWH           17985.611 Hz
FIDRES         0.274439 Hz
AQ           1.8219508 sec
RG                406.4
DW               27.800 usec
DE                6.00 usec
TE               297.1 K
D1           2.00000000 sec
d11          0.03000000 sec
DELTA        1.89999998 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1               12.50 usec
PL1               2.00 dB
SFO1         75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2            80.00 usec
PL2              -1.00 dB
PL12             20.16 dB
PL13             16.98 dB
SFO2        300.1312005 MHz
SI                32768
SF          75.4677490 MHz
WDW                  EM
SSB                   0
LB                 1.00 Hz
GB                    0
PC                 1.40
```

7.21 ^{1}H NMR and ^{13}C NMR spectrum of **3s**




```
NAME              cl-1211
EXPNO                  10
PROCNO                  1
Date_            20081114
Time                 17.58
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                       8
DS                       0
SWH               8992.806 Hz
FIDRES            0.137219 Hz
AQ              3.6438515 sec
RG                     181
DW                  55.600 usec
DE                    6.00 usec
TE                   301.1 K
D1             1.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   7.00 usec
PL1                 -1.00 dB
SFO1          300.1324010 MHz
SI                   32768
SF            300.1300062 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```




```
NAME            CL-1211-ph
EXPNO                   10
PROCNO                   1
Date_            20081115
Time                 0.33
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG            zgpg30
TD                   65536
SOLVENT              CDCl3
NS                    6869
DS                       4
SWH              17985.611 Hz
FIDRES            0.274439 Hz
AQ              1.8219508 sec
RG                    2048
DW                  27.800 usec
DE                    6.00 usec
TE                   300.6 K
D1             2.00000000 sec
d11            0.03000000 sec
DELTA          1.89999998 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                  12.50 usec
PL1                  2.00 dB
SFO1          75.4752953 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                 -1.00 dB
PL12                20.16 dB
PL13                16.98 dB
SFO2          300.1312005 MHz
SI                   32768
SF             75.4677466 MHz
WDW                     EM
SSB                      0
LB                    1.00 Hz
GB                       0
PC                    1.40
```