Rh-Catalyzed Asymmetric Hydrogenation of γ-Phthalimido Substituted α,β-Unsaturated Carboxylic Acid Esters: An Efficient Enantioselective Synthesis of β-Aryl-γ-Amino Acids

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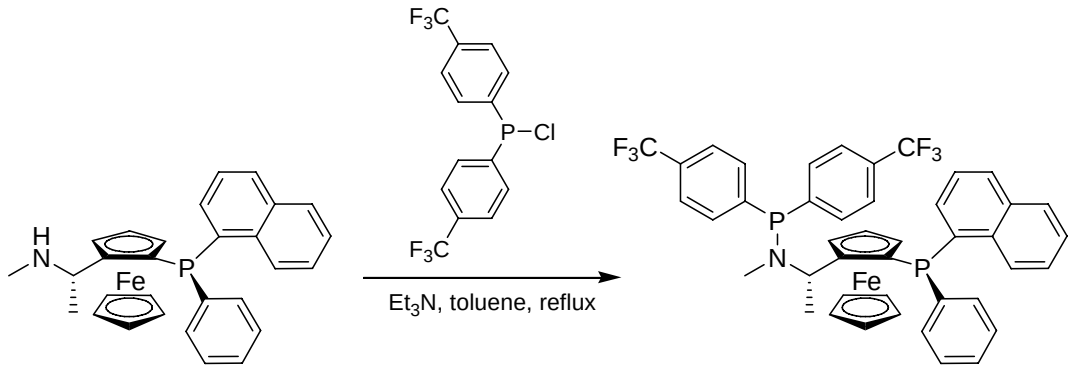
Instruments and Materials

All solvents were dried and degassed by standard methods and stored under nitrogen. All the commercially available catalysts and ligands were brought from Strem Chemicals and without further purification. Ligand **2a-e** were synthesized according to the procedures reported previously.[1,2] All reactions were carried out under an atmosphere of nitrogen using the standard Schlenk techniques or in a nitrogen-filled glovebox, unless otherwise noted.

NMR spectra were recorded on BRUKER DRX 400 spectrometers. Chemical shifts are reported in parts per million (ppm) down field from TMS with the solvent resonance as the internal standard. Coupling constants (*J*) are reported in Hz and refer to apparent peak multiplications. High resolution mass spectra (HRMS) were recorded on Q-Tof (Micromass) or Bruker APEX II spectrometer. Enantiomeric excess (ee) were determined by HPLC analysis on an Agilent HP-1100. Optical rotations were measured on a JASCO P-1020 high sensitive polarimeter.

Preparation of (S_C,R_{Fc},R_P)-3: (R_{Fc})-2-{(S_C)-1-*N*-methyl-*N*-[bis(4-trifluoromethylphenyl)phosphino] aminoethyl}-1-[(R_P)-(1-naphthyl)phenylphosphino]ferrocene:

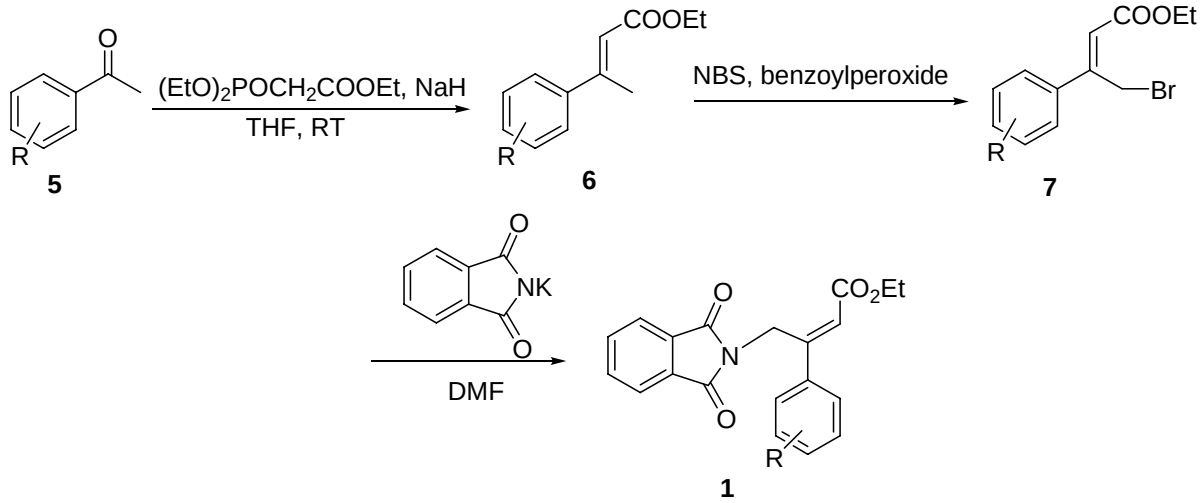


(S_C,R_{Fc},R_P)-PPFNHMe (S_C,R_{Fc},R_P)-**3**

To a solution of (S_c,R_{Fc},R_p)-PPFNHMe[4] (477 mg, 1.0 mmol) and Et_3N (0.28 mL, 2.0 mmol) in toluene (2.5 mL) was added dropwise bis[4-(trifluoromethyl)phenyl]chlorophosphine (0.374 g, 1.05 mmol) at 0ºC. The mixture was warmed to room temperature, and stirred at room temperature overnight. The reaction mixture was filtered through a pad of neutral aluminium oxide and eluted with hexane-EtOAc (10:1) to afford the title compound (717 mg, 90%) as orange foam. ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.58 (3H, d, *J* = 6.8 Hz; C**H**$_3$CH), 2.22 (3H, d, *J* = 3.3 Hz; C**H**$_3$N), 3.44 (5H, s; C_5**H**$_5$), 4.29 (1H, m; C_5**H**), 4.44 (1H, m; C_5**H**), 4.55 (1H, s; C_5**H**), 5.00-5.06 (1H, m; C**H**CH$_3$), 6.86 (1H, m; Ar-**H**),

7.02-7.07 (6H, m; Ar-**H**), 7.12-7.14 (1H, m; Ar-**H**), 7.34-7.36 (5H, m; Ar-**H**), 7.39-7.42 (3H, m; Ar-**H**), 7.56-7.58 (1H, m; Ar-**H**), 7.88-7.90 (2H, m; Ar-**H**), 9.42 (1H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 19.26, 19.32, 31.53, 31.64, 58.21, 58.30, 58.59, 58.68, 69.48, 69.94, 70.07, 71.96, 75.40, 75.54, 97.70, 124.64, 125.27, 125.55, 126.09, 126.40, 126.85, 126.97, 127.20, 127.79, 128.38, 128.75, 129.68, 130.19, 131.95, 132.11, 134.83, 136.34, 142.19 ppm; ^{31}P NMR (162 MHz , $CDCl_3$): δ = 55.44, -42.40 ppm; HRMS (EI) m/z calcd for $C_{43}H_{35}NF_6FeP_2^+$: 797.1498, found: 797.1493.

General Procedure for the Preparation of phthalimidobut-2-enoate 1.[5,6,7]



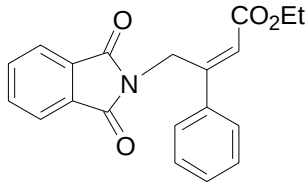
To a suspension of sodium hydride (60% dispersion in mineral oil, 4 mmol) in dry THF (200 mL) was added dropwise triethyl phosphonoacetate (4 mmol) at 0℃ under argon atmosphere. After 30 min, the appropriate ketone **5** (2.7 mmol) was added to the reaction mixture, which was then allowed to warm to room temperature and stirred for 24h and then cooled with a water bath. A saturated aqueous ammonium chloride solution (20 mL) was then added dropwise to the cold mixture. The aqueous phase was extracted with diethyl ether (4 x 50mL) and the combined organic phase was washed with brine (3 x 50mL), dried over sodium sulfate, and concentrated in vacuo. Flash chromatography (hexanes/ethyl acetate, 95:5) yielded ester **6** as a clear oil with 60-80% yields**.**

. A solution of α,β-unsaturated ester **6** (1 mmol), NBS (1.2 mmol) and benzoylperoxide (0.04 mmol) in dry CCl_4 (35 mL) was refluxed under nitrogen atmosphere for 10 h. The resulting reaction mixture was cooled to room temperature and then filtered through a sintered funnel to separate succinimide formed during the reaction. The filtrate was concentrated under reduced pressure to obtain bromoester **7**.

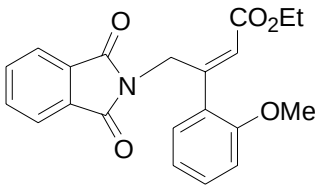
It was then purified by column chromatography packed with silica gel to give pure pale yellow colored gum, bromoester **7** with above 90% yields**.**

To the suspension of potassium phthalimide (10.0 mmol) in dry DMF (10 mL) was added a solution of bromoester **7** in dry DMF (10 mL). The reaction was run at room temperature and monitored by TLC. After the reaction was complete, the reaction mixture was poured into water and extracted with ether and concentrated in vacuum to leave a white solid. The residue can be easily purified by the recrystallization from ethyl acetate/ hexane (1/4) or by flash chromatography (hexanes/ethyl acetate, 5:1), recrystallized with ethyl acetate/ hexane (1/4), to give the desired compounds **1**.

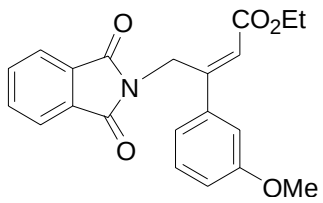
Ethyl (*Z*)-3-phenyl-4-phthalimidobut-2-enoate (1a). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.34 (3H, t, *J* = 7.2 Hz; C**H**$_3$CH$_2$), 4.25 (2H, q, *J* = 7.2 Hz; C**H**$_2$CH$_3$), 5.47 (2H, s; C**H**$_2$N), 6.17 (1H, s; =C**H**), 7.23-7.27 (3H, m; Ar-**H**), 7.36 (2H, m; Ar-**H**), 7.60-7.62 (2H, m; Ar-**H**), 7.68-7.71 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.3, 37.0, 60.5, 121.0, 123.2, 127.0,128.4, 129.0, 131.7, 133.9, 138.0, 153.9, 166.1, 167.8 ppm.



Ethyl (*Z*)-3-(2-methoxyphenyl)-4-phthalimidobut-2-enoate (1b). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.33 (3H, t, *J* = 7.2 Hz; C**H**$_3$CH$_2$), 3.74 (3H, s; C**H**$_3$O), 4.25 (2H, q, *J* = 7.2 Hz; C**H**$_2$CH$_3$), 5.41 (2H, d; C**H**$_2$N), 6.00 (1H, t; =C**H**), 6.68-6.70 (1H, m; Ar-**H**), 6.77-6.79 (1H, m; Ar-**H**), 7.06-7.08 (1H, m; Ar-**H**), 7.14 (1H, m; Ar-**H**), 7.59-7.61 (2H, m; Ar-**H**), 7.67-7.69 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.3, 38.5, 55.3, 60.4, 109.9, 120.3, 121.5, 122.9, 127.7, 129.9, 130.2, 131.8, 133.7, 154.2, 156.3, 166.0, 167.6 ppm.

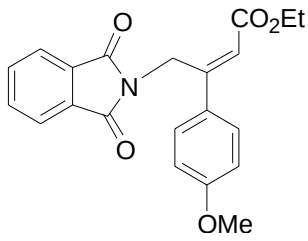


Ethyl (*Z*)-3-(3-methoxyphenyl)-4-phthalimidobut-2-enoate (1c). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.34 (3H, t, *J* = 7.2 Hz; C**H**$_3$CH$_2$), 3.75 (3H, s; C**H**$_3$O), 4.26 (2H, q, *J* = 7.2 Hz; C**H**$_2$CH$_3$), 5.46 (2H, d; C**H**$_2$N), 6.20 (1H, s; =C**H**), 6.76-6.78 (1H, m; Ar-**H**), 6.91 (1H, m; Ar-**H**), 6.95-6.97 (1H, m; Ar-**H**), 7.13-7.17 (1H, m; Ar-**H**), 7.61-7.63 (2H, m; Ar-**H**), 7.70-7.72 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.3, 37.0, 55.3, 60.5, 111.8, 115.6, 119.5, 121.0, 123.2, 129.4, 131.8, 133.9,

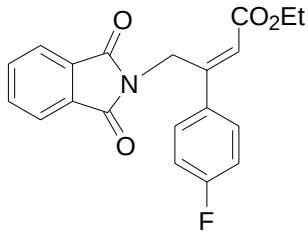


139.4, 153.7, 159.4, 166.1, 167.9 ppm.

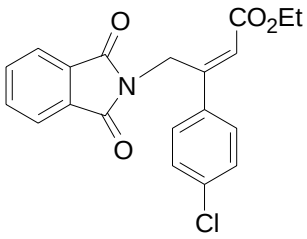
Ethyl (*Z*)-3-(4-methoxyphenyl)-4-phthalimidobut-2-enoate (1d). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.33 (3H, t, *J* = 7.2 Hz; C**H**$_3$CH$_2$), 3.72 (3H, s; C**H**$_3$O), 4.27 (2H, m, C**H**$_2$CH$_3$), 5.47 (2H, s; C**H**$_2$N), 6.15 (1H, s; =C**H**), 6.76-6.78 (2H, m; Ar-**H**), 7.33-7.36 (2H, m; Ar-**H**), 7.61-7. 63 (2H, m; Ar-**H**), 7.70-7.72 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.3, 36.6, 55.2, 60.3, 113.8, 119.7, 123.2, 128.4, 130.2, 131.8, 133.9, 153.2, 160.3, 166.2, 167.9 ppm.



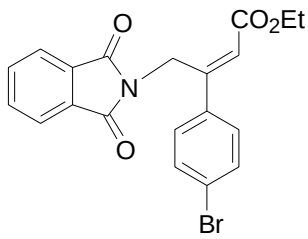
Ethyl (*Z*)-3-(4-fluorophenyl)-4-phthalimidobut-2-enoate (1e). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.34 (3H, t, *J* = 7.2 Hz; C**H**$_3$CH$_2$), 4.26 (2H, q, *J* = 7.2 Hz; C**H**$_2$CH$_3$), 5.45 (2H, s; C**H**$_2$N), 6.14 (1H, s; =C**H**), 6.91 (2H, m; Ar-**H**), 7.34-7.37 (2H, m; Ar-**H**), 7.62-7.65 (2H, m; Ar-**H**), 7.71-7.73 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.3, 37.0, 60.5, 115.3, 115.6, 120.6, 121.1, 123.2, 128.9, 129.0, 131.7, 134.0, 152.8, 161.9, 164.4, 165.9, 167.8 ppm.



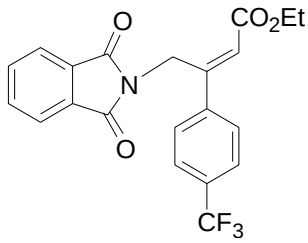
Ethyl (*Z*)-3-(4-chlorophenyl)-4-phthalimidobut-2-enoate (1f). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.34 (3H, t, *J* = 7.2 Hz; C**H**$_3$CH$_2$), 4.26 (2H, q, *J* = 7.2 Hz; C**H**$_2$CH$_3$), 5.44 (2H, s; C**H**$_2$N), 6.15 (1H, s; =C**H**), 7.21 (2H, m; Ar-**H**), 7.31 (2H, m; Ar-**H**), 7.65 (2H, m; Ar-**H**), 7.71-7.72 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.3, 36.8, 60.6, 121.4, 123.3, 128.4, 128.7, 131.7, 134.0, 135.1, 136.5, 152.6, 165.8, 167.8 ppm.



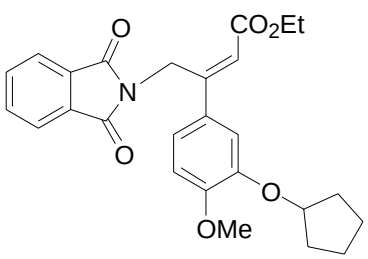
Ethyl (*Z*)-3-(4-bromophenyl)-4-phthalimidobut-2-enoate (1g). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.34 (3H, t, *J* = 7.2 Hz; C**H**$_3$CH$_2$), 4.27 (2H, q, *J* = 7.2 Hz; C**H**$_2$CH$_3$), 5.44 (2H, s; C**H**$_2$N), 6.15 (1H, s; =C**H**), 7.24 (2H, d, *J* = 8.4 Hz; Ar-**H**), 7.37 (2H, d, *J* = 8.4 Hz; Ar-**H**), 7.63-7.65 (2H, m; Ar-**H**), 7.71-7.73 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.3, 36.8, 60.6, 121.5, 123.3, 123.4, 128.7, 131.6, 134.0, 137.0, 152.6, 165.8, 167.8 ppm.



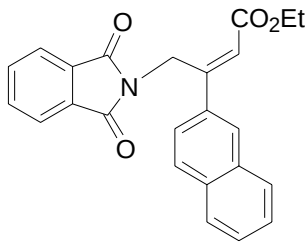
Ethyl (*Z*)-3-(4-trifluoromethylphenyl)-4-phthalimidobut-2-enoate (1h). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.35 (3H, t, *J* = 7.2 Hz; C**H**$_3$CH$_2$), 4.27 (2H, q, *J* = 7.2 Hz; C**H**$_2$CH$_3$), 5.47 (2H, s; C**H**$_2$N), 6.19 (1H, s; =C**H**), 7.48-7.53 (4H, m; Ar-**H**), 7.63-7.66 (2H, m; Ar-**H**), 7.71-7.73 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.3, 36.9, 60.7, 122.5, 123.3, 125.4, 127.5, 131.6, 134.1, 141.7, 152.3, 165.6, 167.8 ppm.



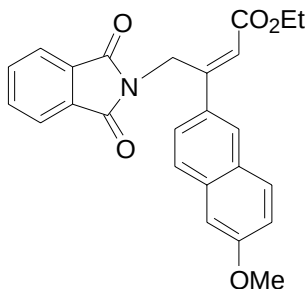
Ethyl (*Z*)-3-[3-(cyclopentyloxy)-4-methoxyphenyl]-4-phthalimidobut-2-enoate (1i). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.34 (3H, t, *J* = 7.2 Hz; C**H**$_3$CH$_2$), 1.56-1.59 (2H, m; C**H**$_2$CH$_2$), 1.78 (4H, m; C**H**$_2$CH$_2$), 1.91-1.93 (2H, m; C**H**$_2$CH$_2$), 3.75 (3H, s; C**H**$_3$O), 4.27 (2H, m; C**H**$_2$CH$_3$), 4.72 (1H, m; C**H**O), 5.45 (2H, s; C**H**$_2$N), 6.14 (1H, s; =C**H**), 6.71 (1H, d, *J* = 8.4 Hz; Ar-**H**), 6.87 (1H, d, *J* = 1.6 Hz; Ar-**H**), 6.94 (1 H, dd, *J* = 1.6 Hz, 8.4 Hz; Ar-**H**), 7.61-7.63 (2H, m; Ar-**H**), 7.70-7.71(2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.4, 24.1, 32.8, 36.9, 55.9, 60.4, 80.4, 111.4, 113.2, 119.7, 119.8, 123.2, 130.5, 131.8, 133.9, 147.2, 150.8, 153.8, 166.3, 167.9 ppm.



Ethyl (*Z*)-3-(2-Naphthyl)-4-phthalimidobut-2-enoate (1j). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.36 (3H, t, *J* = 7.2 Hz; C**H**$_3$CH$_2$), 4.28 (2H, q, *J* = 7.2 Hz; C**H**$_2$CH$_3$), 5.59 (2H, s; C**H**$_2$N), 6.32 (1H, s; =C**H**), 7.41-7.45 (2H, m; Ar-**H**), 7.49-7.55 (3H, m; Ar-**H**), 7.64-7.66 (2H, m; Ar-**H**), 7.71-7.73 (2H, m; Ar-**H**), 7.79-7.82 (1H, m; Ar-**H**), 7.91 (1H, s; Ar-**H**) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.3, 36.9, 60.5, 121.5, 123.2, 124.5, 126.4, 126.7, 126.8, 127.5, 128.1, 128.5, 131.7, 133.0, 133.4, 133.8, 135.4, 153.7, 166.1, 167.9 ppm.

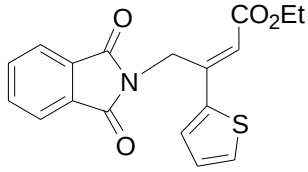


Ethyl (*Z*)-3-(6-methoxy-naphthalen-2-yl)-4-phthalimidobut-2-enoate (1k). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.35 (3H, t, *J* = 7.2 Hz; C**H**$_3$CH$_2$), 3.85 (3H, s; C**H**$_3$O), 4.28 (2H, q, *J* = 7.2 Hz; C**H**$_2$CH$_3$), 5.58 (2H, s; C**H**$_2$N), 6.30 (1H, s; =C**H**), 7.01 (1H, s; Ar-**H**), 7.07 (1H, d, *J* = 8.4 Hz; Ar-**H**), 7.46 (1H, d, *J* = 8.4 Hz; Ar-**H**), 7.54-7.55 (2H, m; Ar-**H**), 7.60-7.70 (4H, m; Ar-**H**), 7.84 (1H, s; Ar-**H**) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.3, 36.8, 55.3, 60.5, 105.5, 119.3, 120.8, 123.2, 125.0, 126.6, 126.9, 128.4, 130.0, 131.7, 133.1, 133.8,



134.8, 153.7, 158.4, 166.2, 167.9 ppm.

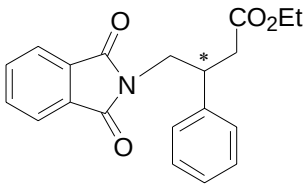
Ethyl (*Z*)-3-(2-thienyl)-4-phthalimidobut-2-enoate (1l). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.35 (3H, t, *J* = 7.2 Hz; C**H**$_3$CH$_2$), 4.28 (2H, q, *J* = 7.2 Hz; C**H**$_2$CH$_3$), 5.47 (2H, s; C**H**$_2$N), 6.43 (1H, d; =C**H**), 6.94 (1H, dd, *J* = 4.8, 4.0 Hz; Ar-**H**), 7.22 (1H, d, *J* = 4.8 Hz; Ar-**H**), 7.38 (1H, d, *J* = 4.0 Hz; Ar-**H**), 7.64-7.67 (2H, m; Ar-**H**), 7.76-7.78 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.3, 35.8, 60.5, 119.7, 123.3, 127.2, 127.6, 128.1, 131.9, 134.0, 141.3, 144.1, 166.0, 167.9 ppm.



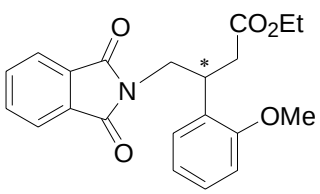
General procedure for asymmetric hydrogenation

In a nitrogen-filled glovebox, to a solution of $[Rh(COD)_2]BF_4$ (1.0 mg, 0.0025 mmol) in anhydrous and degassed CH_2Cl_2 (1 mL) was added ligand (0.00275 mmol). After stirring the mixture for 30 min, a substrate (0.25 mmol) dissolved in CH_2Cl_2 (1 mL) was added. The reaction mixture was transferred to a Par stainless autoclave. The autoclave was purged three times with hydrogen and the pressure was set to the desired pressure. The hydrogenation was performed at room temperature for 24 h. After carefully releasing the hydrogen, the solvent was removed. The enantiomeric excess was determined by HPLC after purification on silica gel.

Ethyl 3-phenyl-4-phthalimidobutanoate (4a). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.06 (3H, t, *J* = 7.2 Hz; C**H**$_3$CH$_2$), 2.72 (2H, m, *J* =8.0 Hz; C**H**$_2$CH), 3.70-3.78 (1H, m; C**H**CH$_2$), 3.89-3.95 (4H, m; OC**H**$_2$CH$_3$, C**H**$_2$N), 7.18-7.22 (1H, m; Ar-**H**), 7.25-7.28 (4H, m; Ar-**H**), 7.67-7.70 (2H, m; Ar-**H**), 7.79-7.81 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.0, 38.6, 40.8, 43.2, 60.5, 123.3, 127.3, 127.7, 128.6, 131.9, 134.0, 140.4, 168.1, 171.5 ppm; HRMS (ESI) m/z calcd for $C_{20}H_{19}NO_4Na^+$: 360.1212, found: 360.1223; $[\alpha]_D^{25}$ = -62.8 (c 1.00, $CHCl_3$) for 95% ee; HPLC (UV 254 nm, Chiralpak AD (0.46 cm x 25 cm), *i*-PrOH/Hexane = 3/97, 0.8 mL/min): t_1 = 47.0 min, t_2 = 56.3 min.

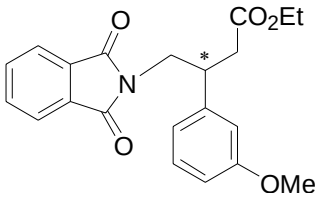


Ethyl 3-(2-methoxyphenyl)-4-phthalimidobutanoate (4b). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.08 (3H, t, *J* = 7.2 Hz; C**H**$_3$CH$_2$), 2.80-2.84 (2H, m; C**H**$_2$CH), 3.76 (3H, s; C**H**$_3$O), 3.94-3.99 (5H, m; C**H**$_2$O,



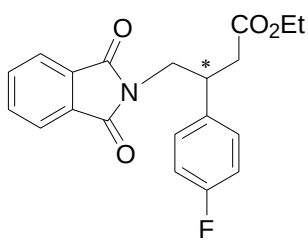
C**H**$_2$N, -C**H**-), 6.78-6.80 (1H, m; Ar-**H**), 6.83-6.86 (1H, m; Ar-**H**), 7.66-7.69 (2H, m; Ar-**H**), 7.77-7.79 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.3, 36.7, 37.2, 41.9, 55.5, 60.6, 110.9, 120.7, 123.3, 128.5, 128.7, 129.2, 132.2, 134.0, 157.9, 168.4, 172.3 ppm; HRMS (ESI) m/z calcd for $C_{21}H_{21}NO_5Na^+$: 390.1317, found: 390.1336; $[\alpha]_D^{25}$ = -81.9 (c 0.76, $CHCl_3$) for 96% ee; HPLC (UV 254 nm, Chiralcel OD-H (0.46 cm x 25 cm), *i*-PrOH/Hexane = 10/90, 1.0 mL/min): t_1 = 9.6 min, t_2 = 21.4 min.

Ethyl 3-(3-methoxyphenyl)-4-phthalimidobutanoate (4c). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.08 (3H, t, *J* = 7.2 Hz; C**H**$_3$CH$_2$), 2.70-2.72 (2H, d, *J* = 7.6 Hz; C**H**$_2$CH), 3.72-3.76 (4H, m; C**H**$_3$O, -C**H**-), 3.88-3.94 (4H, m; C**H**$_2$O, C**H**$_2$N), 6.76 (1H, m; Ar-**H**), 6.81 (1H, s; Ar-**H**), 6.86-6.88 (1H, m; Ar-**H**), 7.18-7.20 (1H, m; Ar-**H**), 7.69-7.71 (2H, m; Ar-**H**), 7.80-7.82 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.1, 38.6, 40.8, 43.2, 55.2, 60.5, 112.9, 113.3, 120.0, 123.3, 129.6, 131.9, 134.0, 142.1, 159.7, 168.2, 171.5 ppm; HRMS (ESI) m/z calcd for $C_{21}H_{21}NO_5Na^+$: 390.1317, found: 390.1328; $[\alpha]_D^{25}$ = -70.0 (c 1.00, $CHCl_3$) for 94% ee; HPLC (UV 254 nm, Chiralcel OD-H (0.46 cm x 25 cm), *i*-PrOH/Hexane = 3/97, 1.0 mL/min): t_1 = 22.3 min, t_2 = 35.1 min.



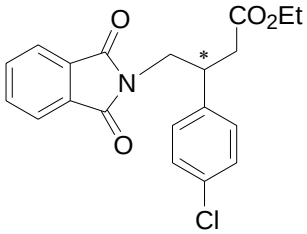
Ethyl 3-(4-methoxyphenyl)-4-phthalimidobutanoate (4d). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.07 (3H, t, *J* = 7.2 Hz; C**H**$_3$CH$_2$), 2.67-2.70 (2H, m; C**H**$_2$CH), 3.68-3.76 (1H, m; -C**H**-), 3.82 (3H, s; C**H**$_3$O), 3.85-3.88 (2H, m; C**H**$_2$N), 3.92-3.96 (2H, m; C**H**$_2$O), 6.80 (2H, d, *J* = 8.0 Hz; Ar-**H**), 7.18 (2H, d, *J* = 8.0 Hz; Ar-**H**), 7.67-7.71 (2H, m; Ar-**H**), 7.78-7.81 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.1, 38.9, 40.0, 43.3, 55.2, 60.5, 114.0, 123.3, 128.7, 131.9, 132.4, 134.0, 158.7, 168.2, 171.6 ppm; HRMS (ESI) m/z calcd for $C_{21}H_{21}NO_5Na^+$: 390.1317, found: 390.1328; $[\alpha]_D^{25}$ = -83.9 (c 0.94, $CHCl_3$) for 94% ee; HPLC (UV 254 nm, Chiralcel AD (0.46 cm x 25 cm), *i*-PrOH/Hexane = 5/95, 0.8 mL/min): t_1 = 54.4 min, t_2 = 74.6 min.

Ethyl 3-(4-fluorophenyl)-4-phthalimidobutanoate (4e). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.07 (3H, t, *J* = 7.2 Hz; C**H**$_3$CH$_2$), 2.68-2.72 (2H, m; C**H**$_2$CH), 3.72-3.76 (1H, m; -C**H**-), 3.86-3.88 (2H, m; C**H**$_2$N),



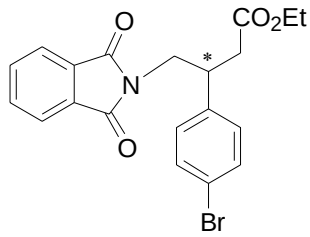
3.89-3.98 (2H, m; C**H**$_2$O), 6.94 (2H, t, J = 8.4 Hz; Ar-**H**), 7.22-7.28 (2H, m; Ar-**H**), 7.69-7.71 (2H, m; Ar-**H**), 7.79-7.81 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.0, 38.4, 38.7, 40.1, 43.1, 60.5, 115.4, 115.6, 123.3, 129.2, 129.3, 131.8, 134.0, 136.1, 160.7, 163.2, 168.1, 171.3 ppm; HRMS (ESI) m/z calcd for $C_{20}H_{18}NO_4FNa^+$: 378.1118, found: 378.1126; $[\alpha]_D^{25}$ = -88.8 (c 1.00, $CHCl_3$) for 96% ee; HPLC (UV 254 nm, Chiralpak AD (0.46 cm x 25 cm), *i*-PrOH/Hexane = 5/95, 0.8 mL/min): t_1 = 37.3 min, t_2 = 50.4 min.

Ethyl 3-(4-chlorophenyl)-4-phthalimidobutanoate (4f). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.08 (3H, t,



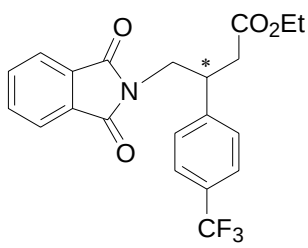
J = 7.2 Hz; C**H**$_3$CH$_2$), 2.68-2.72 2H, m; C**H**$_2$CH), 3.72-3.75 (1H, m; -C**H**-), 3.86-3.91 (2H, m; C**H**$_2$N), 3.92 (2H, q, J = 7.2 Hz; C**H**$_2$O), 7.20-7.26 (4H, m; Ar-**H**), 7.69-7.71 (2H, m; Ar-**H**), 7.79-7.81 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.1, 38.6, 40.2, 42.9, 60.6, 123.3, 128.8, 129.1, 131.8, 133.0, 134.1, 138.9, 168.1, 171.2 ppm; HRMS (ESI) m/z calcd for $C_{20}H_{18}NO_4ClNa^+$: 394.0822, found: 394.0836; $[\alpha]_D^{25}$ = -96.5 (c 0.80, $CHCl_3$) for 95% ee; HPLC (UV 254 nm, Chiralpak AD (0.46 cm x 25 cm), *i*-PrOH/Hexane =5/95, 0.8 mL/min): t_1 = 42.8 min, t_2 = 62.1 min.

Ethyl 3-(4-bromophenyl)-4-phthalimidobutanoate (4g). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.08 (3H, t,



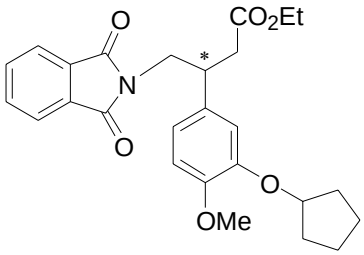
J = 7.2 Hz; C**H**$_3$CH$_2$), 2.63-2.74 (2H, m; C**H**$_2$CH), 3.70-3.74 (1H, m; -C**H**-), 3.82-3.90 (2H, m; C**H**$_2$N), 3.92 (2H, q, J = 7.2 Hz; C**H**$_2$O), 7.15 (2H, d, J = 8.0 Hz; Ar-**H**), 7.39 (2H, d, J = 8.0 Hz; Ar-**H**), 7.69-7.71 (2H, m; Ar-**H**), 7.79-7.82 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.1, 38.5, 40.3, 42.9, 60.6, 121.2, 123.3, 129.5, 131.7, 134.1, 139.5, 168.1, 171.1 ppm; HRMS (ESI) m/z calcd for $C_{20}H_{18}NO_4Br\ Na^+$: 438.0317, found: 438.0327; $[\alpha]_D^{25}$ = -106.7 (c 0.96, $CHCl_3$) for 94% ee; HPLC (UV 254 nm, Chiralpak AD (0.46 cm x 25 cm), *i*-PrOH/Hexane = 5/95, 1.0 mL/min): t_1 = 38.0 min, t_2 = 56.4 min.

Ethyl 3-(4-trifluoromethylphenyl)-4-phthalimidobutanoate (4h). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.06-1.11 (3H, m; C**H**$_3$CH$_2$), 2.72-2.76 (2H, m; C**H**$_2$CH), 3.81-3.87 (1H, m; -C**H**-), 3.90-3.98 (4H, m;



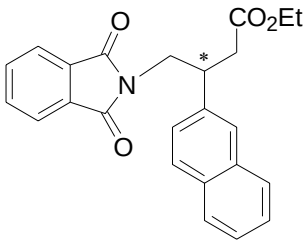
C**H**$_2$N, C**H**$_2$O), 7.40 (2H, d, *J* = 8.0 Hz; Ar-**H**), 7.54 (2H, d, *J* = 8.0 Hz; Ar-**H**), 7.69-7.72 (2H, m; Ar-**H**), 7.80-7.83 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.0, 38.4, 40.7, 42.8, 60.7, 123.4,125.6, 128.2, 131.7, 134.2, 144.6, 168.1, 171.1 ppm; HRMS (ESI) m/z calcd for $C_{21}H_{18}NO_4F_3Na$+: 428.1086, found: 428.1102; $[\alpha]_D^{25}$ = -73.0 (c 1.10, $CHCl_3$) for 93% ee; HPLC (UV 254 nm, Chiralpak AD (0.46 cm x 25 cm), *i*-PrOH/Hexane = 10/90, 1.0 mL/min): t_1 = 15.9 min, t_2 = 24.4 min.

Ethyl 3-[3-(cyclopentyloxy)-4-methoxyphenyl]-4-phthalimidobutanoate (4i). ^{1}H NMR (400 MHz,



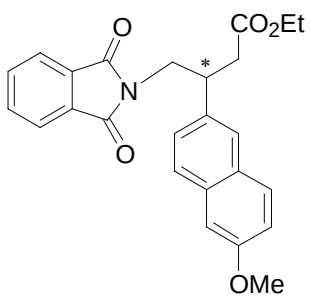
$CDCl_3$): δ = 1.09 (3H, t, *J* = 7.2 Hz; C**H**$_3$CH$_2$), 1.55-1.57 (2H, m; C**H**$_2$CH$_2$), 1.73-1.89 (6H, m; C**H**$_2$CH$_2$), 2.68 (2H, d, *J* = 7.2 Hz; C**H**$_2$CH), 3.68 (1H, m; -C**H**-), 3.78 (3H, s; C**H**$_3$O), 3.85-3.89 (2H, m; C**H**$_2$N), 3.93-3.98 (2H, m; C**H**$_2$O), 4.70 (1H, m; -C**H**-), 6.73-6.80 (3H, m; Ar-**H**), 7.67-7.71 (2H, m; Ar-**H**), 7.78-7.80 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.1, 24.1, 32.7, 32.9, 38.9, 40.2, 43.3, 56.0, 60.5, 80.3, 111.9, 114.7, 119.6, 123.3, 131.9, 132.7, 134.0, 147.4, 149.0, 168.1, 171.6 ppm; HRMS (ESI) m/z calcd for $C_{26}H_{29}NO_6Na^+$: 474.1893, found: 474.1916; $[\alpha]_D^{25}$ = -80.4 (c 0.9, $CHCl_3$) for 97% ee; HPLC (UV 254 nm, Chiralpak AD (0.46 cm x 25 cm), *i*-PrOH/Hexane = 10/90, 0.8 mL/min): t_1 = 22.5 min, t_2 = 30.3 min.

Ethyl 3-(2-Naphthyl)-4-phthalimidobutanoate (4j). ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.02-1.07 (3H,



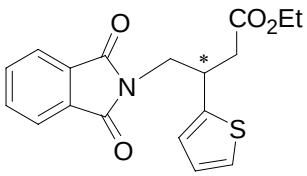
m; C**H**$_3$CH$_2$), 2.80-2.82 (2H, m; C**H**$_2$CH), 3.88-3.93 (3H, m; -C**H**-, C**H**$_2$N), 3.95-4.01 (2H, m; C**H**$_2$O), 7.42-7.47 (3H, m; Ar-**H**), 7.65-7.68 (2H, m; Ar-**H**), 7.72 (1H, s; Ar-**H**), 7.77-7.81 (5H, m; Ar-**H**), 7.73 (2H, m; Ar-**H**), 7.79 (1H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.0, 38.8, 40.9, 43.1, 60.5, 123.3, 125.7, 125.8, 126.1, 126.7, 127.7, 127.8, 128.4, 131.9, 132.7, 133.4, 134.0, 137.9, 168.2, 171.5 ppm; HRMS (ESI) m/z calcd for $C_{24}H_{21}NO_4Na^+$: 410.1368, found: 410.1374. $[\alpha]_D^{25}$ = -98.7 (c 0.94, $CHCl_3$) for 94% ee; HPLC (UV 254 nm, Chiralcel OD-H (0.46 cm x 25 cm), *i*-PrOH/Hexane =3/97, 1.0 mL/min): t_1 = 27.9 min, t_2 = 34.9 min.

Ethyl 3-(6-methoxy-naphthalen-2-yl)-4-phthalimidobutanoate (4k). ^{1}H NMR (400 MHz, $CDCl_3$): δ

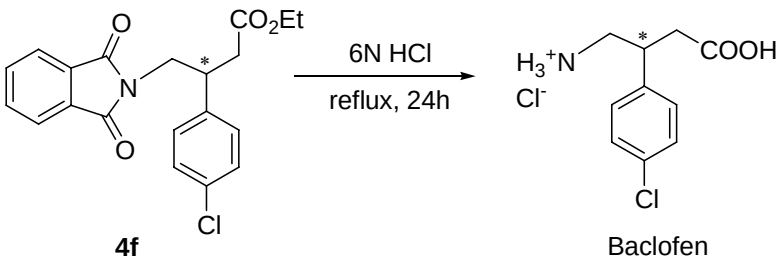


= 1.03 (3H, t, J = 7.2 Hz; C**H**$_3$CH$_2$), 2.78 (2H, d, J = 7.2 Hz; C**H**$_2$CH), 3.88-3.94 (6H, m; C**H**$_3$O, -C**H**-, C**H**$_2$N), 3.96 (2H, m; C**H**$_2$O), 7.08-7.11 (2H, m; Ar-**H**), 7.40-7.42 (1H, m; Ar-**H**), 7.64-7.70 (5H, m; Ar-**H**), 7.78-7.80 (2H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, CDCl$_3$): δ = 14.1, 38.8, 40.8, 43.2, 55.3, 60.5, 105.6, 118.9, 123.3, 126.2, 126.5, 127.2, 128.9, 129.3, 131.9, 133.8, 134.0, 135.6, 157.6, 168.2, 171.6 ppm; HRMS (ESI) calcd for $C_{25}H_{23}NO_5Na^+$: 440.1474, found: 440.1485; $[\alpha]_D^{25}$ = -151.2 (c 0.80, CHCl$_3$) for 94% ee; HPLC (UV 254 nm, Chiralcel OD-H (0.46 cm x 25 cm), *i*-PrOH/Hexane = 2/98, 1.0 mL/min): t_1 = 53.3 min, t_2 = 60.7 min.

Ethyl 3-(2-thienyl)-4-phthalimidobutanoate (4l). ^{1}H NMR (400 MHz, CDCl$_3$): δ = 1.12 (3H, t, J = 7.2 Hz; C**H**$_3$CH$_2$), 2.75 (2H, d, J = 7.2 Hz; C**H**$_2$CH), 3.88-4.07 (5H, m; C**H**$_2$O, -C**H**-, C**H**$_2$N), 6.90 (2H, d, J = 4.0 Hz; Ar-**H**), 7.14 (1H, m; Ar-**H**), 7.70-7.72 (2H, m; Ar-**H**), 7.82 (2H, s; Ar-**H**) ppm; ^{13}C NMR (100 MHz, CDCl$_3$): δ = 14.1, 36.3, 39.6, 43.7, 60.7, 123.4, 124.2, 124.9, 126.8, 131.9, 134.1, 143.5, 168.1, 171.1 ppm; HRMS (ESI) m/z calcd for $C_{18}H_{17}NO_4SNa^+$: 366.0776, found: 366.0784; $[\alpha]_D^{25}$ = -62.8 (c 0.2, CHCl$_3$) for 97% ee; HPLC (UV 254 nm, Chiralpak AD (0.46 cm x 25 cm), *i*-PrOH/Hexane = 5/95, 1.0 mL/min): t_1 = 28.3 min, t_2 = 34.0 min.



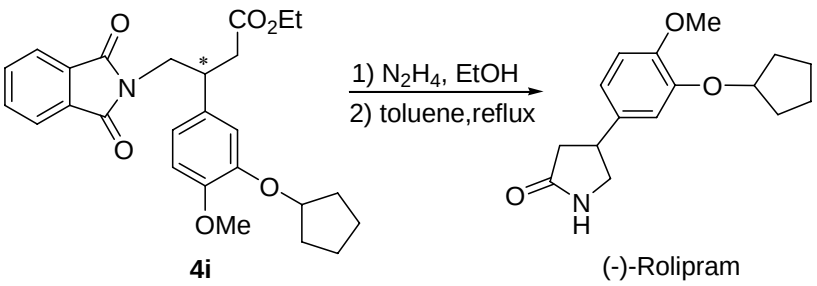
General Procedure for Hydrolysis of Ethyl 3-(4-chlorophenyl)-4-phthalimidobutanoate to Form Baclofen.[8]



A mixture of **4f** (0.5 mmol, 261mg), 6 M HCl (10 mL) was heated under reflux for 24 h. The solution was cooled and the precipitated phthalic acid was filtered off. The filtrate was evaporated to dryness and the result solid was resuspended in water (10 mL). Filtered, and the filtrate was evaporated to dryness under reduced pressure. The solid dried under vacuum: 95% yield. ^{1}H NMR (400 MHz, D$_2$O):

δ = 2.73-2.83 (2H, m; C**H**$_2$CH), 3.24-3.40 (3H, m; C**H**$_2$N, -C**H**-), 7.34-7.43 (4H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, D_2O): δ = 38.4, 39.5, 43.6, 129.3, 129.4, 133.4, 137.0, 175.5 ppm.

Synthesis of (-)-Rolipram.[9,10,11]



To a solution of **4i** (0.17 mmol) in THF (10 mL) was added hydrazine hydrate (0.5 mL) and the solution was stirred for 24 h. After complete consumption of the starting material, the reaction mixture was extracted with dichloromethane and concentrated in vacuum, the residue was dissolved in a mixture of Et_3N (0.5 mL) and toluene (2 mL) and the resultant solution heated at 110 °C for 20 h. After cooling to RT, the solvents were evaporated under reduced pressure. The residue was purified by flash chromatography (silica gel, EtOAc) to yield colorless crystals 36.5 mg (78%); ^{1}H NMR (400 MHz, $CDCl_3$): δ = 1.60-1.63 (2H, m; C**H**$_2$CH$_2$), 1.83-1.92 (6 H, m; -C-C**H**$_2$-CH$_2$), 2.45 (1 H, dd, J = 8.8, 16.9 Hz; C**H**$_2$CO), 2.69 (1H, dd, J = 8.8, 16.9 Hz; C**H**$_2$CO), 3.37-3.41 (1H, m; C**H**$_2$N), 3.63-3.65 (1H, m; -C**H**-), 3.74-3.79 (1H, m; C**H**$_2$N), 3.83 (3H, s; C**H**$_3$O), 4.75-4.78 (1H, m; C**H**O), 6.21 (1H, br; N-**H**), 6.76-6.84 (3H, m; Ar-**H**) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 24.1, 32.8, 38.2, 40.0, 49.9, 56.2, 80.6, 112.2, 113.9, 118.8, 134.6, 147.9, 149.2, 177.9 ppm.

References:

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1904.

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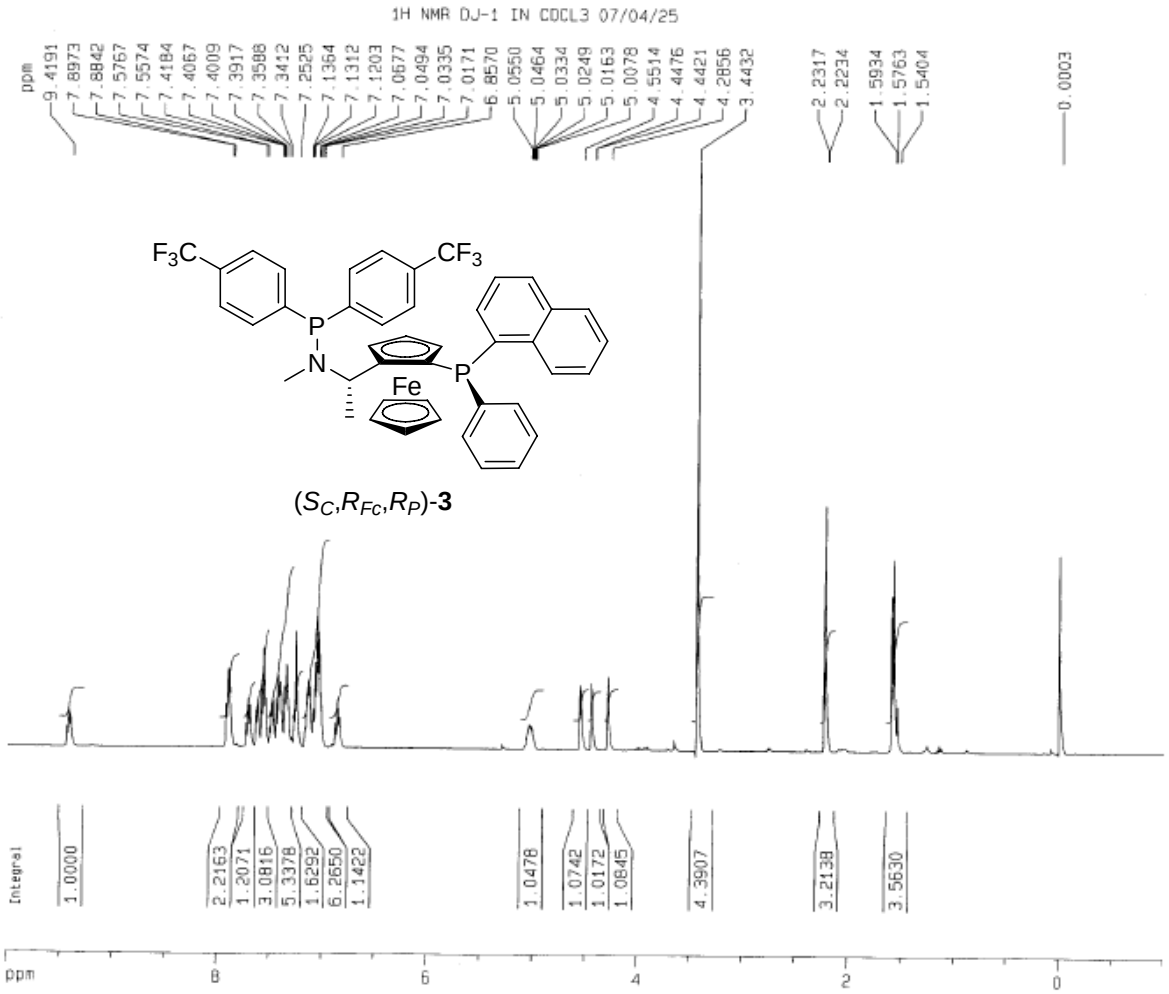

(S_C,R_{Fc},R_P)-**3**

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PROCNO                 1

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PULPROG               zg
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SOLVENT            CDCl3
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DS                     0
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ            2.0447731 sec
RG                  90.5
DW                62.400 usec
DE                  6.00 usec
TE                 295.2 K
D1            1.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

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P1                  8.20 usec
PL1                 5.00 dB
SFO1         400.1332521 MHz

F2 - Processing parameters
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SSB                    0
LB                 -0.50 Hz
GB                   0.1
PC                  1.40

1D NMR plot parameters
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CY                 10.00 cm
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F1               4001.30 Hz
F2P               -1.000 ppm
F2               -400.13 Hz
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HZCM           220.07152 Hz/cm
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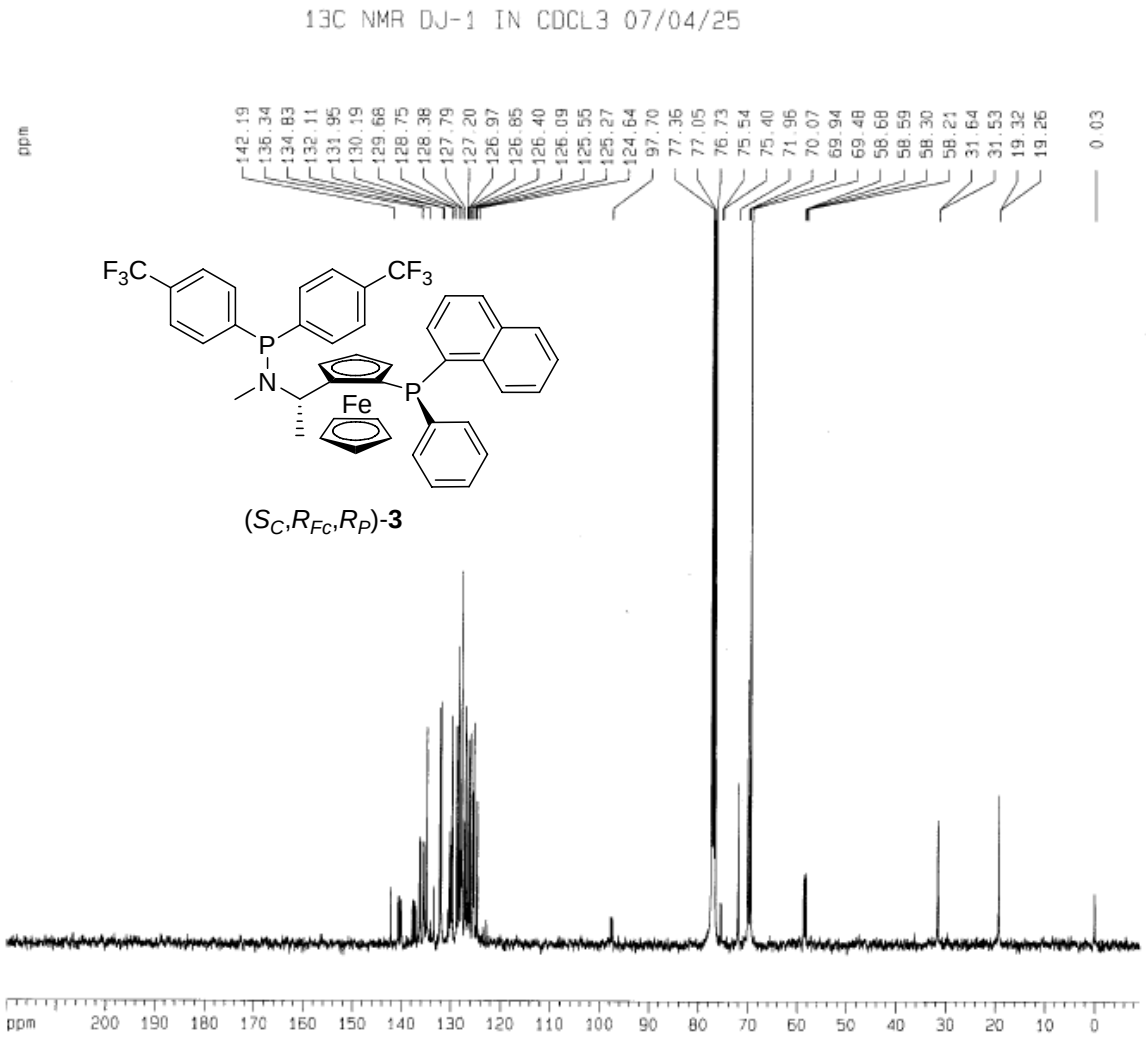


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Current Data Parameters
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EXPNO               412
PROCNO                1

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SOLVENT             D2O
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DS                   16
SWH           23148.148 Hz
FIDRES         0.353213 Hz
AQ            1.4156276 sec
RG                16384
DW               21.600 usec
DE                6.00 usec
TE               296.2 K
D1           1.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 8.20 usec
PL1                1.50 dB
SFO1        100.6234215 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             80.00 usec
PL2                5.00 dB
PL12              23.00 dB
SFO2        400.1320007 MHz

F2 - Processing parameters
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SF          100.6127690 MHz
WDW                 EM
SSB                  0
LB                4.00 Hz
GB                   0
PC                1.40

1D NMR plot parameters
CX                20.00 cm
CY                20.00 cm
F1P              220.000 ppm
F1             22134.81 Hz
F2P              -10.000 ppm
F2             -1006.13 Hz
PPMCM           11.50000 ppm/cm
HZCM          1157.04688 Hz/cm
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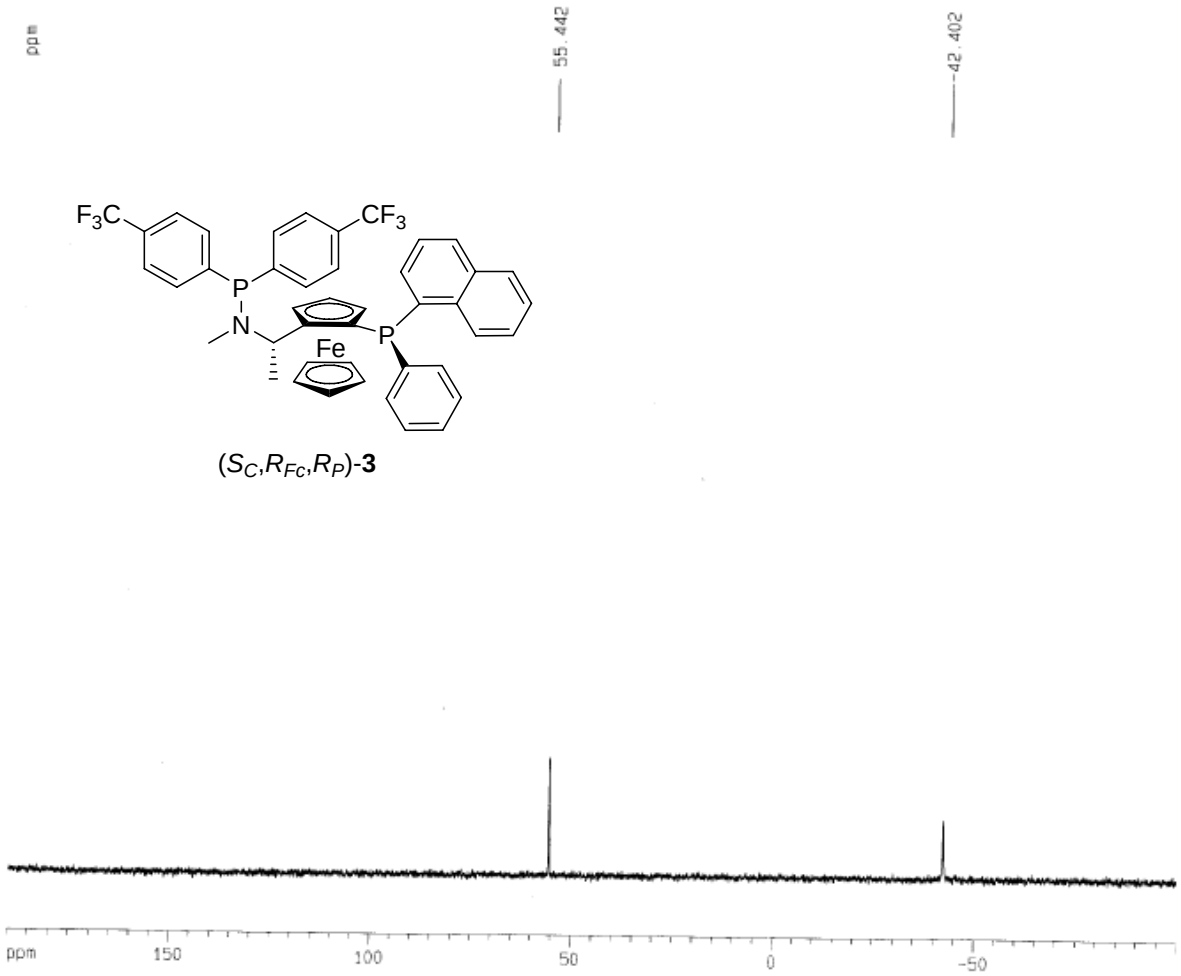
31P NMR DJ-1 IN CDCL37/04/23



(S_C,R_{Fc},R_P)-**3**

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Current Data Parameters
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EXPNO               409
PROCNO                1

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PULPROG               zg
TD                 32768
SOLVENT            CDCl3
NS                    19
DS                     0
SWH            48543.688 Hz
FIDRES          1.481436 Hz
AQ            0.3375604 sec
RG              10321.3
DW                10.300 usec
DE                  6.00 usec
TE                 295.2 K
D1           2.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
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P1                  8.00 usec
PL1                 0.00 dB
SFO1         161.9820520 MHz

F2 - Processing parameters
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SF          161.9755337 MHz
WDW                   EM
SSB                    0
LB                  5.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
CY                  2.00 cm
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F1               30775.35 Hz
F2P              -100.000 ppm
F2              -16197.55 Hz
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HZCM           2348.64526 Hz/cm
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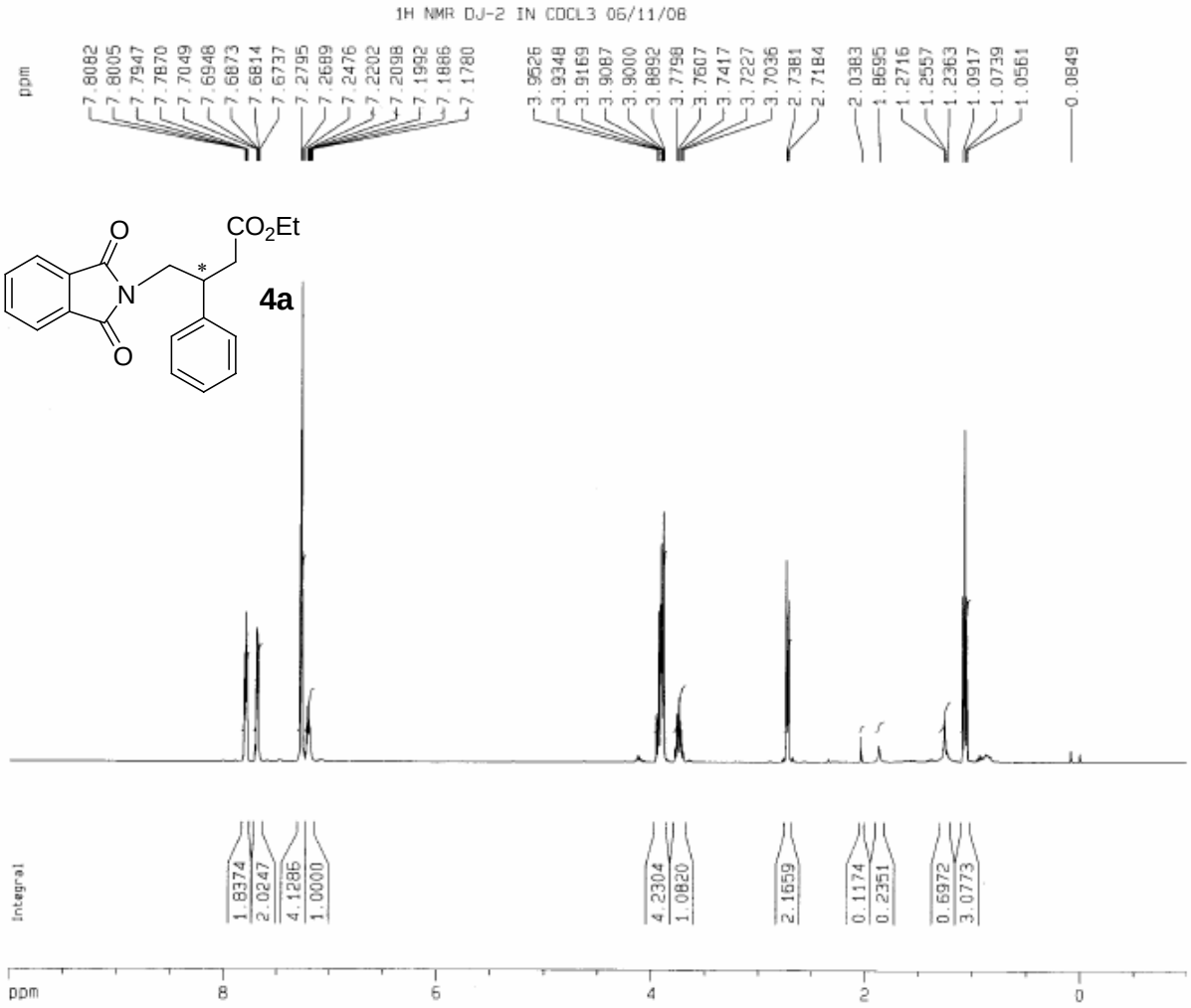


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EXPNO               217
PROCNO                1

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PULPROG              zg
TD                32768
SOLVENT           CDCl3
NS                    8
DS                    0
SWH            8012.820 Hz
FIDRES         0.244532 Hz
AQ            2.0447731 sec
RG                   32
DW               62.400 usec
DE                6.00 usec
TE               295.2 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 8.20 usec
PL1                5.00 dB
SFO1        400.1332521 MHz

F2 - Processing parameters
SI                32768
SF          400.1300076 MHz
WDW                  GM
SSB                   0
LB                -0.50 Hz
GB                  0.1
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
CY                 8.00 cm
F1P               10.000 ppm
F1               4001.30 Hz
F2P               -1.000 ppm
F2               -400.13 Hz
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HZCM           220.07150 Hz/cm
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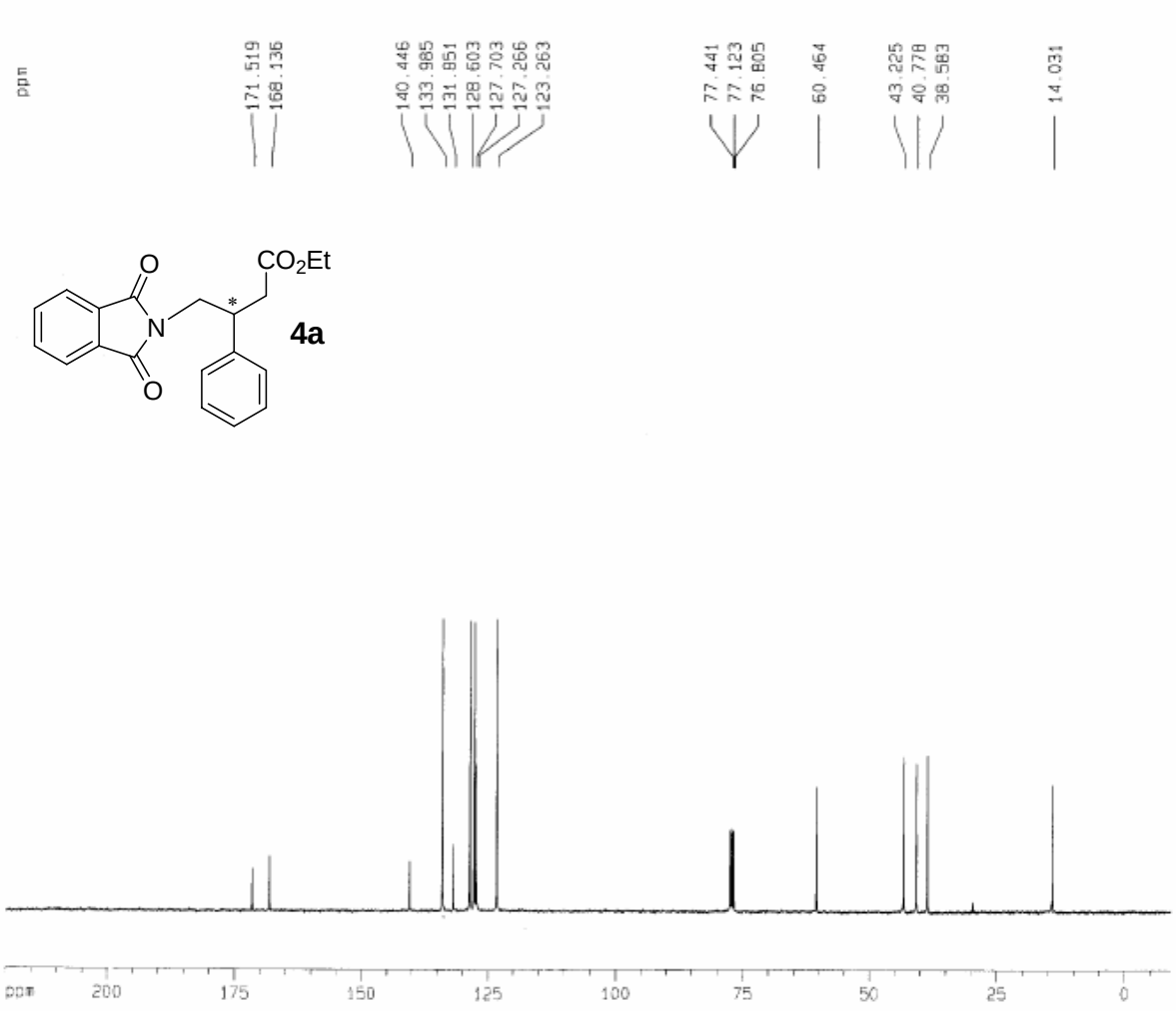
13C NMR DJ-2 IN CDCL3 06/11/08

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Current Data Parameters
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EXPNO                216
PROCNO                 1

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Time               14.57
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PULPROG             zgpg
TD                 65536
SOLVENT            CDCl3
NS                   162
DS                    16
SWH            23148.148 Hz
FIDRES          0.353213 Hz
AQ              1.4156276 sec
RG                  16384
DW                 21.600 usec
DE                  6.00 usec
TE                 295.2 K
D1              1.00000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                   8.20 usec
PL1                  1.50 dB
SFO1          100.6234215 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                  5.00 dB
PL12                23.00 dB
SFO2          400.1320007 MHz

F2 - Processing parameters
SI                   32768
SF            100.6127690 MHz
WDW                     EM
SSB                      0
LB                    4.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   20.00 cm
CY                    5.00 cm
F1P                 220.000 ppm
F1                22134.81 Hz
F2P                -10.000 ppm
F2                -1006.13 Hz
PPMCM              11.50000 ppm/cm
HZCM            1157.04688 Hz/cm
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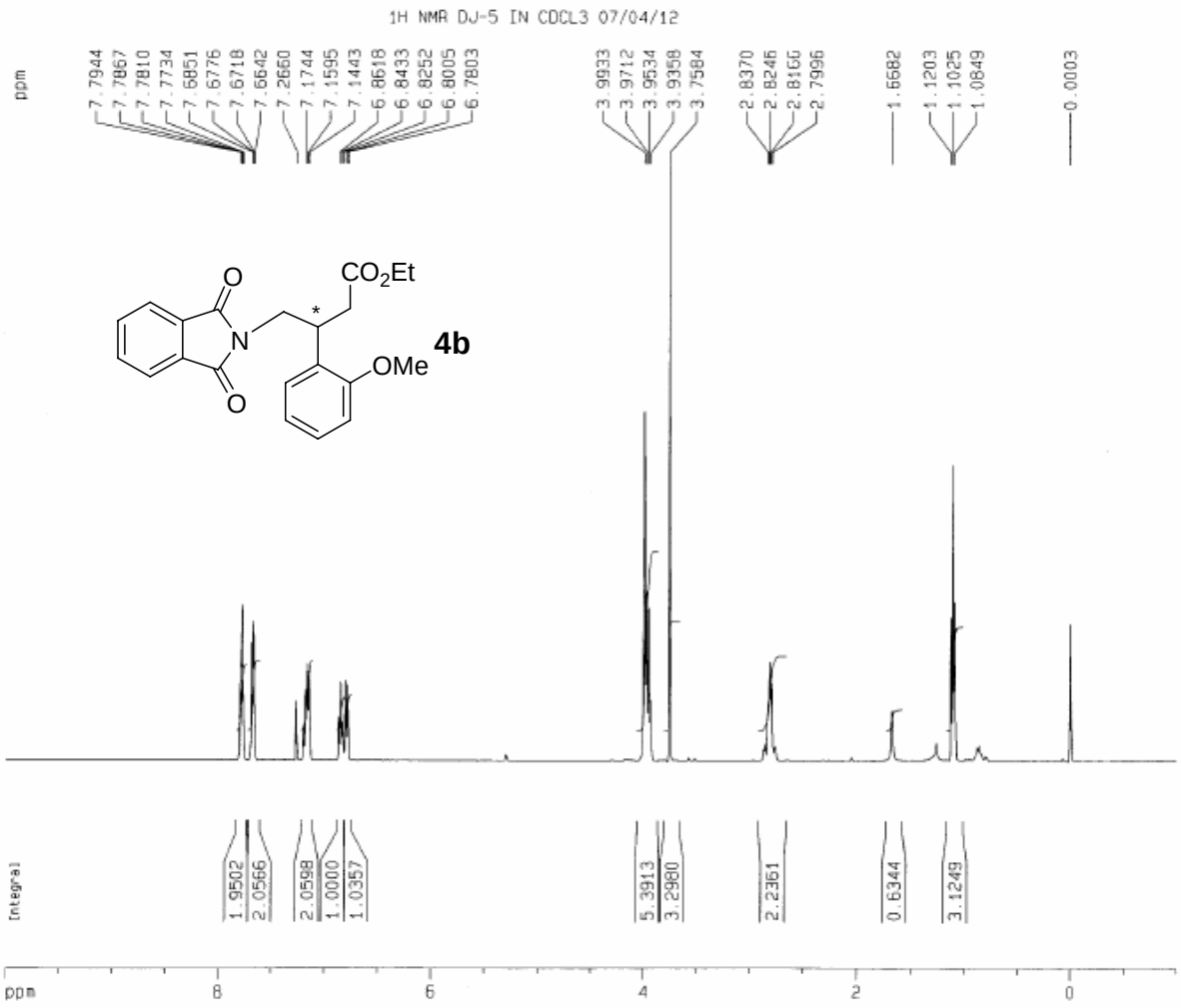


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EXPNO               368
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PULPROG              zg
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SOLVENT           CDCl3
NS                    8
DS                    0
SWH            8012.820 Hz
FIDRES         0.244532 Hz
AQ           2.0447731 sec
RG               101.6
DW              62.400 usec
DE               6.00 usec
TE              293.2 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                8.20 usec
PL1               5.00 dB
SFO1        400.1332521 MHz

F2 - Processing parameters
SI                32768
SF          400.1300157 MHz
WDW                  GM
SSB                   0
LB               -0.50 Hz
GB                  0.1
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
CY                10.00 cm
F1P              10.000 ppm
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F2P              -1.000 ppm
F2              -400.13 Hz
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HZCM          220.07150 Hz/cm
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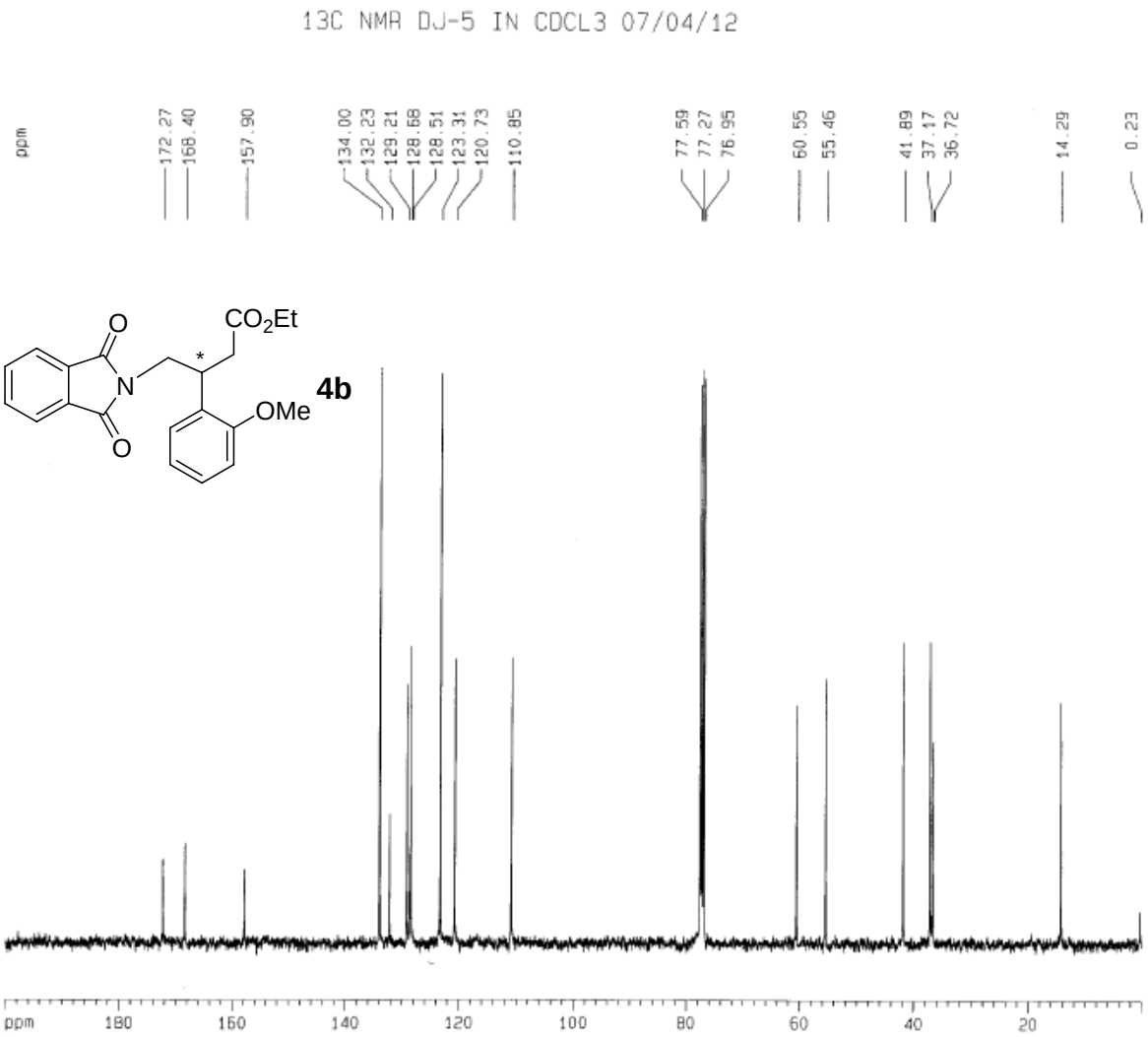


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PROCNO                1

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PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   748
DS                    16
SWH           23148.148 Hz
FIDRES         0.353213 Hz
AQ            1.4156276 sec
RG                 16384
DW               21.600 usec
DE                 6.00 usec
TE                295.2 K
D1           1.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.20 usec
PL1                 1.50 dB
SFO1        100.6234215 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                5.00 dB
PL12              23.00 dB
SFO2        400.1320007 MHz

F2 - Processing parameters
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SF          100.6127487 MHz
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SSB                    0
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1D NMR plot parameters
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CY                 10.00 cm
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F2                  0.00 Hz
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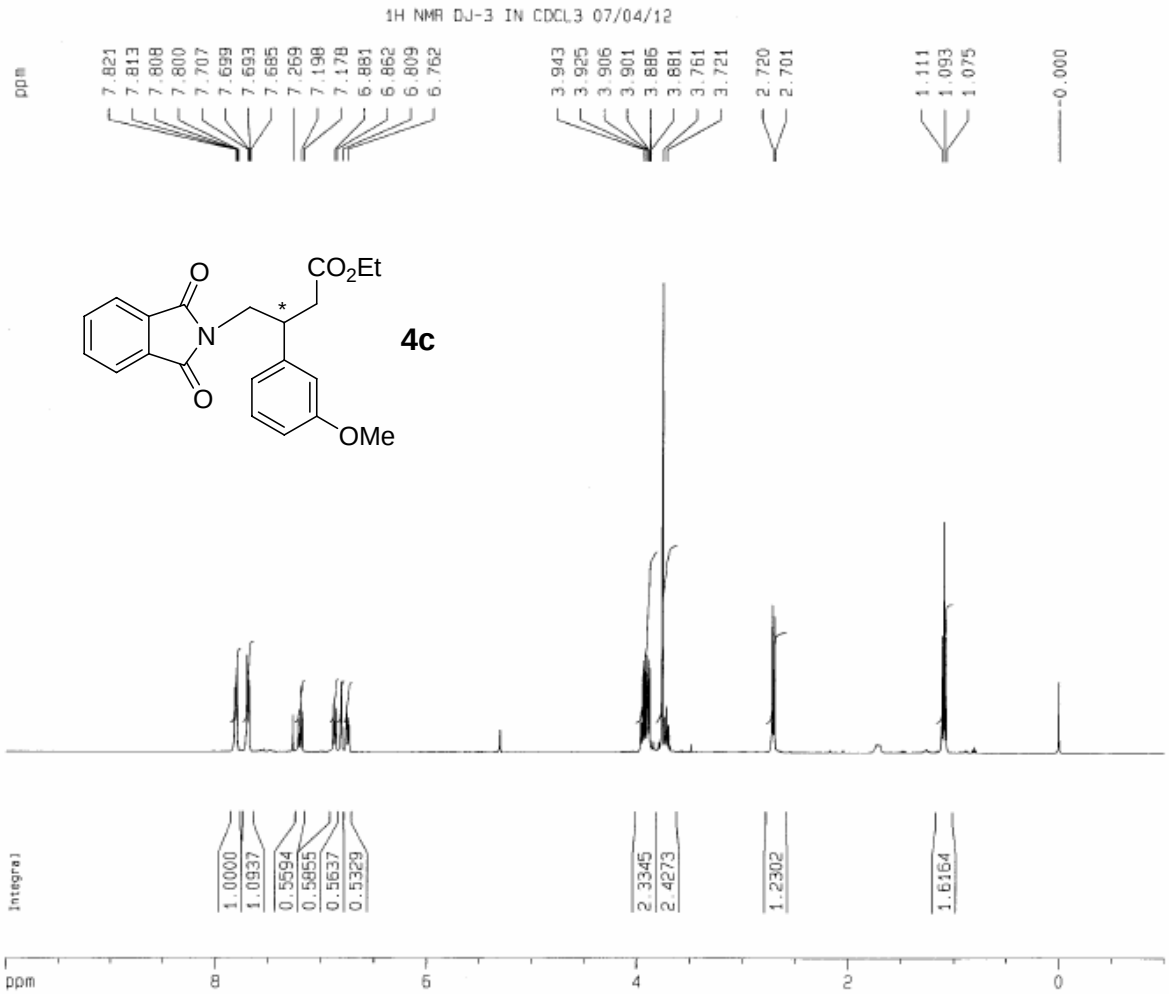


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PROCNO                1

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PULPROG              zg
TD                32768
SOLVENT           CDCl3
NS                    8
DS                    0
SWH            8012.820 Hz
FIDRES         0.244532 Hz
AQ           2.0447731 sec
RG                   64
DW               62.400 usec
DE                6.00 usec
TE               294.2 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 8.20 usec
PL1                5.00 dB
SFO1        400.1332521 MHz

F2 - Processing parameters
SI                32768
SF          400.1300135 MHz
WDW                  GM
SSB                   0
LB                -0.50 Hz
GB                  0.1
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
CY                 8.00 cm
F1P               10.000 ppm
F1               4001.30 Hz
F2P               -1.000 ppm
F2               -400.13 Hz
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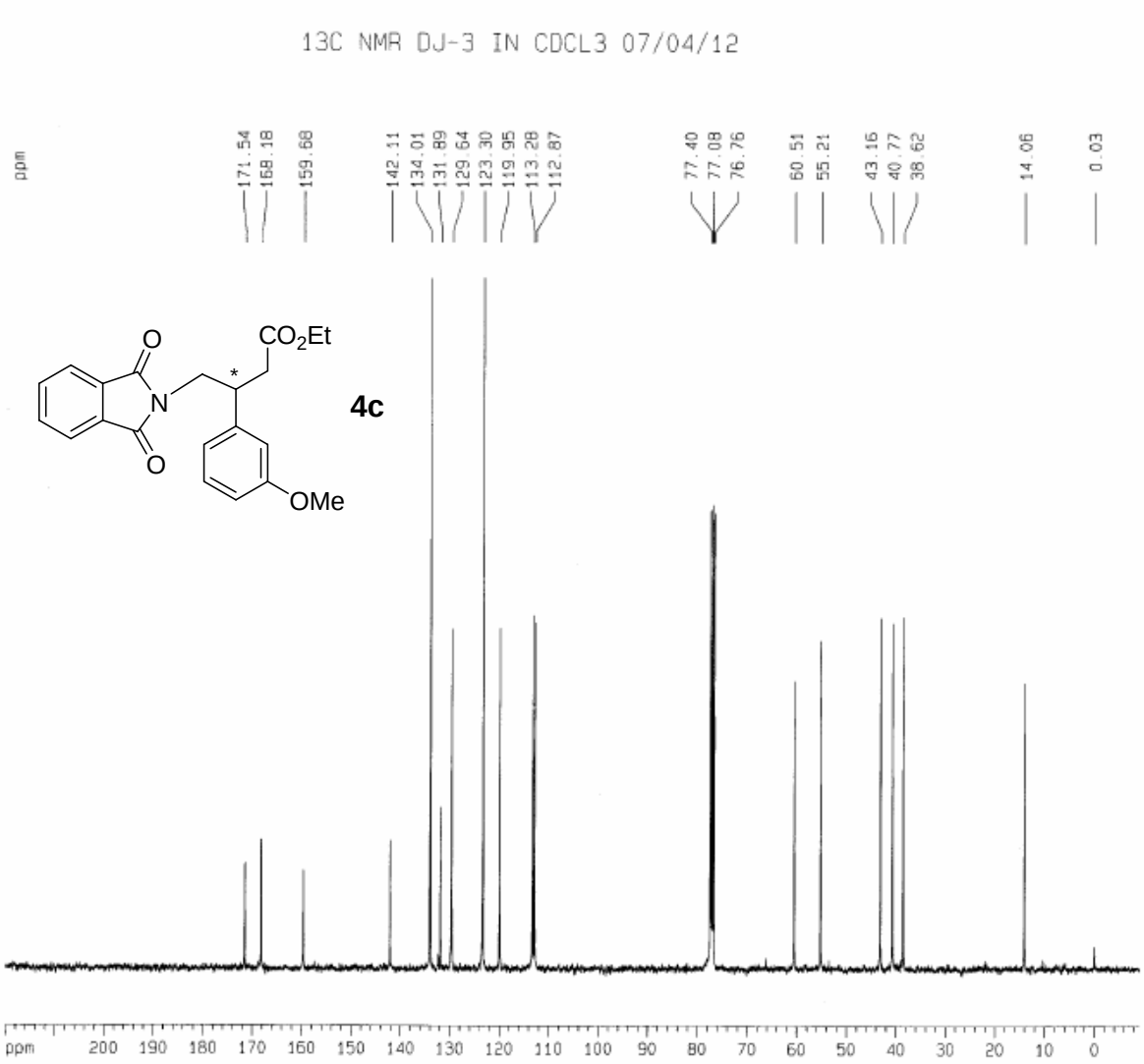


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SOLVENT            CDCl3
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FIDRES          0.353213 Hz
AQ             1.4156276 sec
RG                 16384
DW                21.600 usec
DE                 6.00 usec
TE                294.2 K
D1            1.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.20 usec
PL1                 1.50 dB
SFO1         100.6234215 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                 5.00 dB
PL12               23.00 dB
SFO2         400.1320007 MHz

F2 - Processing parameters
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SF           100.6127690 MHz
WDW                    EM
SSB                     0
LB                   4.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  20.00 cm
CY                  12.00 cm
F1P                220.000 ppm
F1               22134.81 Hz
F2P                -10.000 ppm
F2                -1006.13 Hz
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HZCM           1157.04688 Hz/cm
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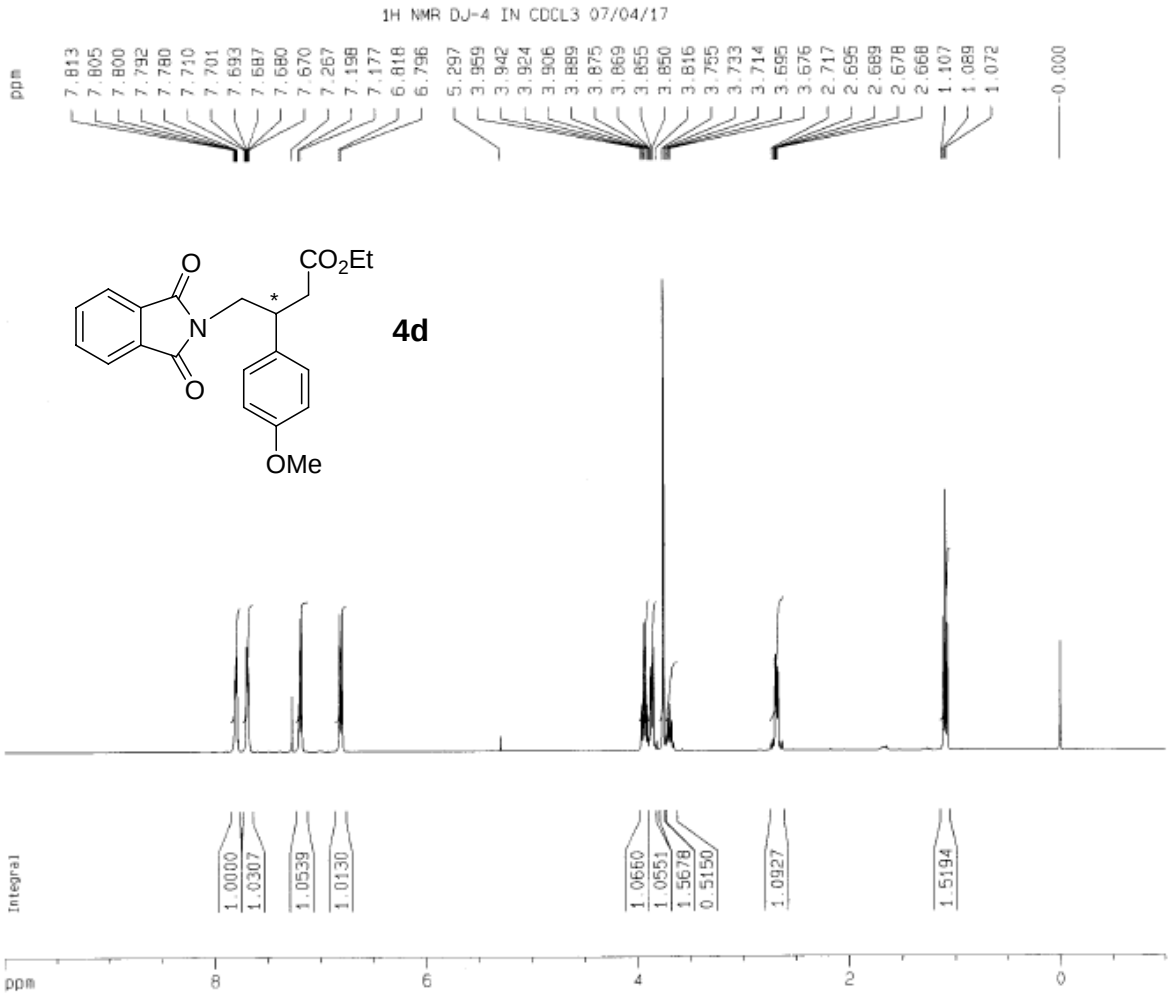


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Current Data Parameters
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EXPNO                386
PROCNO                 1

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PULPROG               zg
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SOLVENT            CDCl3
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DS                     0
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ             2.0447731 sec
RG                    64
DW                62.400 usec
DE                  6.00 usec
TE                 295.2 K
D1            1.00000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                  8.20 usec
PL1                 5.00 dB
SFO1          400.1332521 MHz

F2 - Processing parameters
SI                 32768
SF           400.1300145 MHz
WDW                   GM
SSB                    0
LB                 -0.50 Hz
GB                   0.1
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
CY                  8.00 cm
F1P               10.000 ppm
F1               4001.30 Hz
F2P               -1.000 ppm
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HZCM           220.07150 Hz/cm
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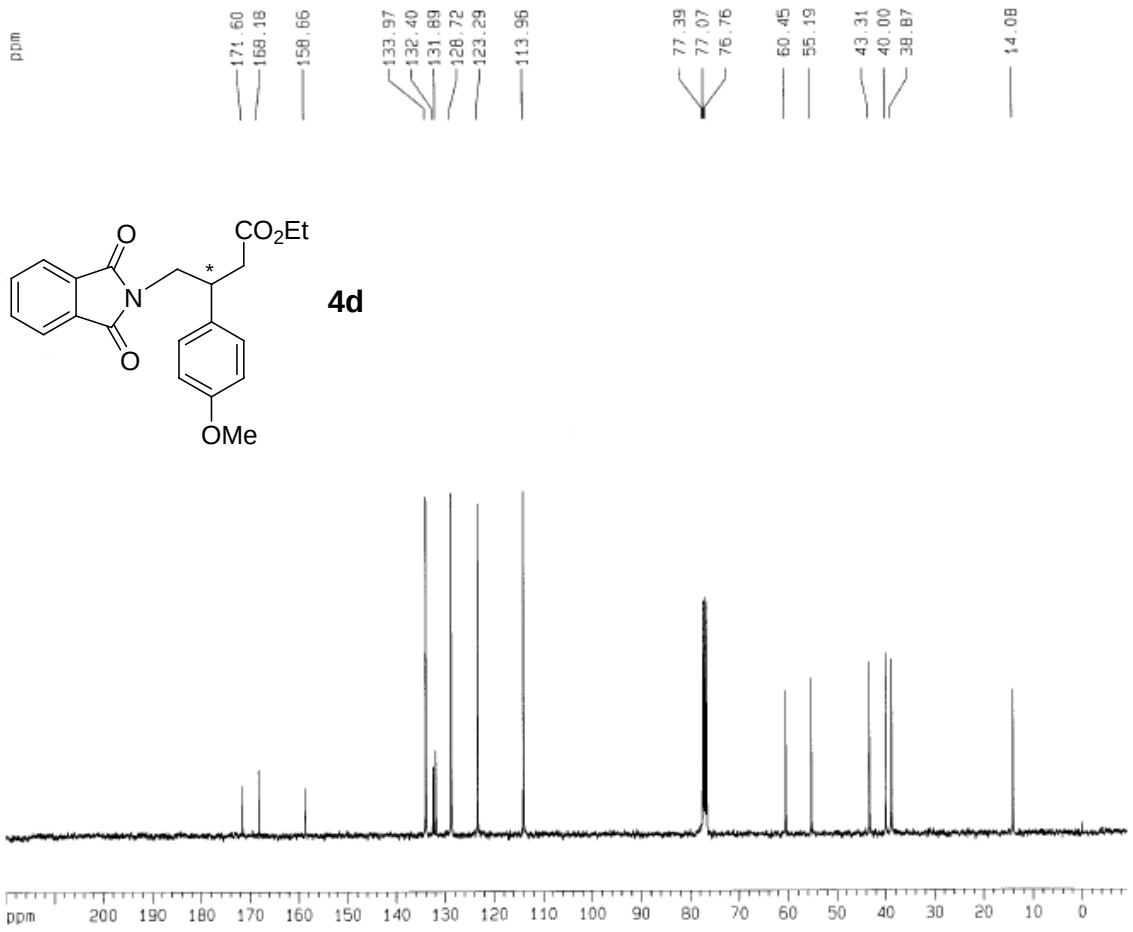
13C NMR DJ-4 IN CDCL3 07/04/17

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Current Data Parameters
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PROCNO                1

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PULPROG            zgdc
TD                65536
SOLVENT           CDCl3
NS                  507
DS                   16
SWH            23148.148 Hz
FIDRES          0.353213 Hz
AQ             1.4156276 sec
RG                16384
DW                21.600 usec
DE                 6.00 usec
TE                296.2 K
D1            1.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.20 usec
PL1                 1.50 dB
SFO1          100.6234215 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              80.00 usec
PL2                 5.00 dB
PL12               23.00 dB
SFO2          400.1320007 MHz

F2 - Processing parameters
SI                 32768
SF            100.6127690 MHz
WDW                   EM
SSB                    0
LB                  4.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
CY                  6.00 cm
F1P               220.000 ppm
F1              22134.81 Hz
F2P               -10.000 ppm
F2              -1006.13 Hz
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HZCM          1157.04688 Hz/cm
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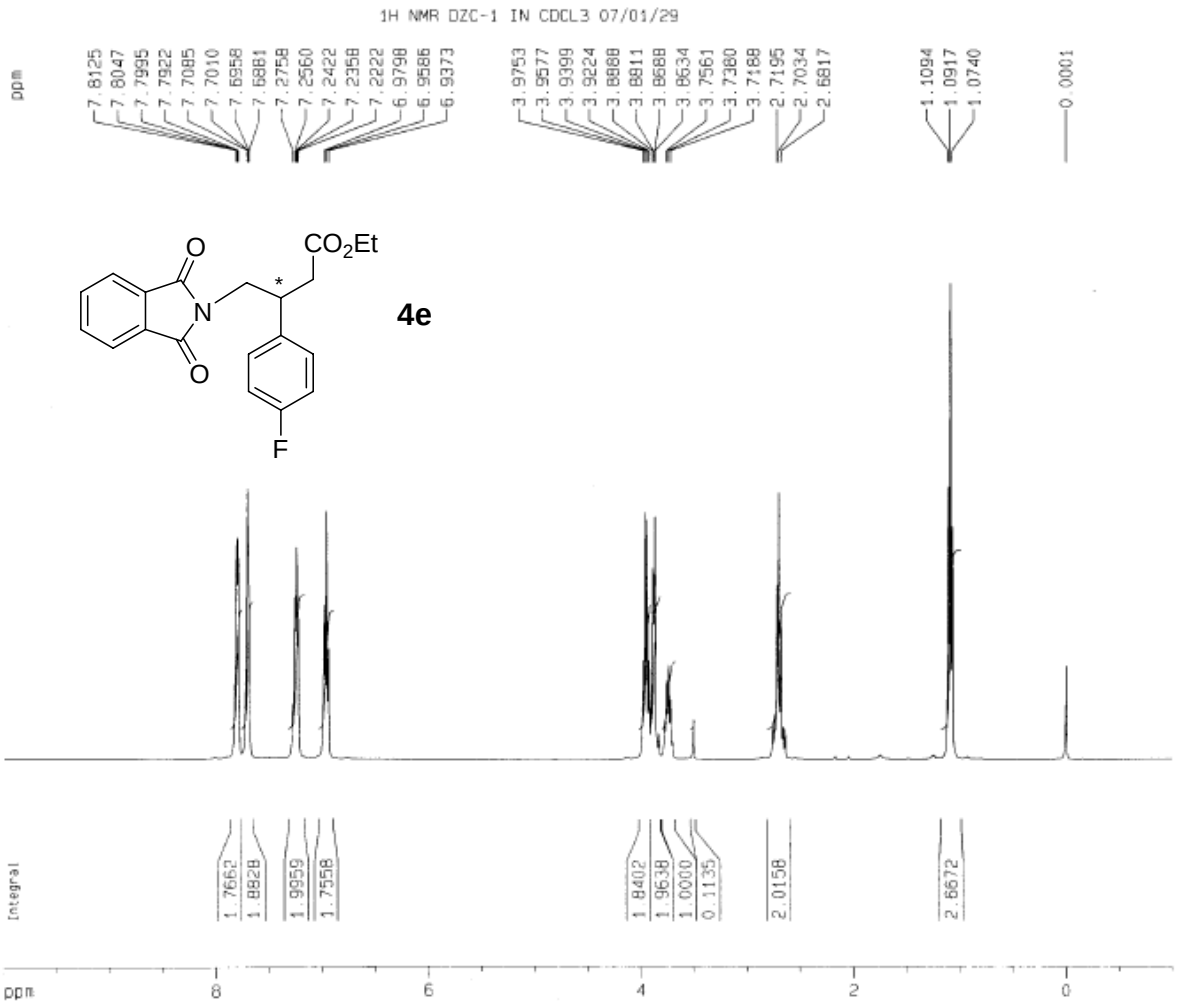


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Current Data Parameters
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EXPNO                 101
PROCNO                  1

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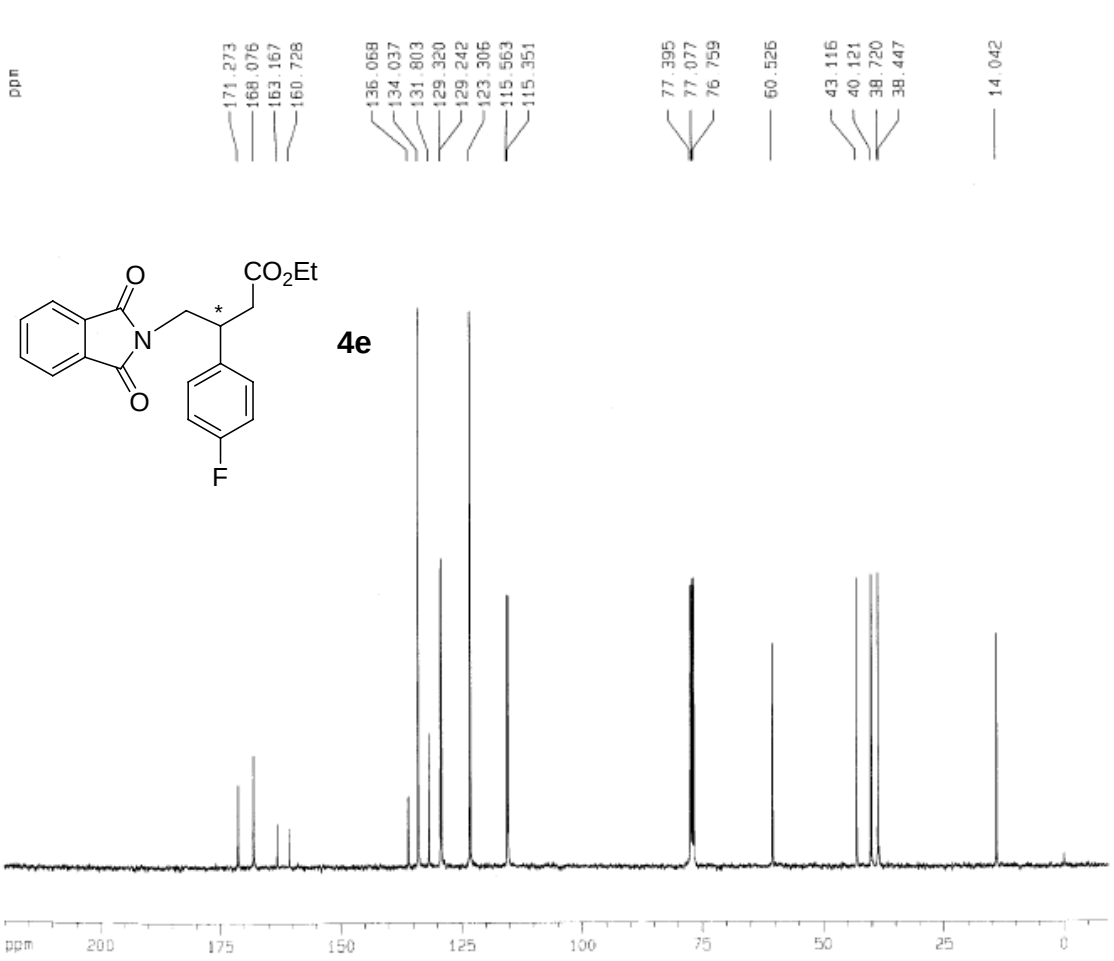
PULPROG                zg
TD                  32768
SOLVENT             CDCl3
NS                      8
DS                      0
SWH              8012.820 Hz
FIDRES           0.244532 Hz
AQ             2.0447731 sec
RG                   50.8
DW                 62.400 usec
DE                   6.00 usec
TE                    0.0 K
D1             1.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   8.20 usec
PL1                  5.00 dB
SFO1          400.1332521 MHz

F2 - Processing parameters
SI                  32768
SF            400.1300101 MHz
WDW                    GM
SSB                     0
LB                  -0.50 Hz
GB                    0.1
PC                   1.40

1D NMR plot parameters
CX                  20.00 cm
CY                   8.00 cm
F1P                 10.000 ppm
F1                 4001.30 Hz
F2P                 -1.000 ppm
F2                 -400.13 Hz
PPMCM              0.55000 ppm/cm
HZCM             220.07150 Hz/cm
```

13C NMR DZC-1 IN COCL3 07/01/29




```
Current Data Parameters
NAME                 duan
EXPNO                 102
PROCNO                  1

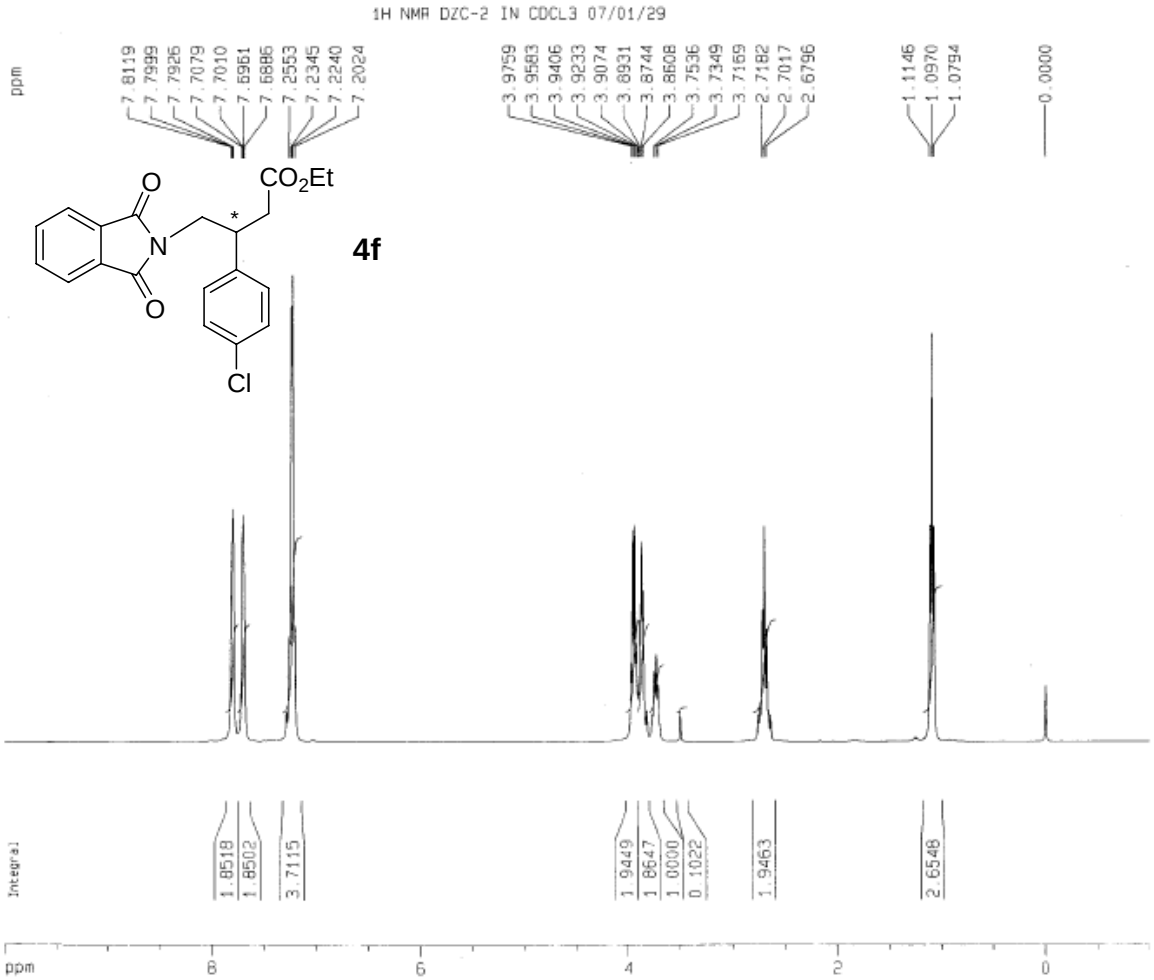
F2 - Acquisition Parameters
Date_            20070129
Time                17.30
INSTRUM           drx400
PROBHD   5 mm PABBO BB-
PULPROG             zgdc
TD                  65536
SOLVENT              DMSO
NS                    498
DS                     16
SWH             23148.148 Hz
FIDRES           0.353213 Hz
AQ              1.4156276 sec
RG                  16384
DW                 21.600 usec
DE                  6.00 usec
TE                   0.0 K
D1              1.00000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                   8.20 usec
PL1                  1.50 dB
SFO1           100.6234215 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                   1H
PCPD2                80.00 usec
PL2                   5.00 dB
PL12                 23.00 dB
SFO2           400.1320007 MHz

F2 - Processing parameters
SI                   32768
SF             100.6127690 MHz
WDW                     EM
SSB                      0
LB                    4.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   20.00 cm
CY                   10.00 cm
F1P                 220.000 ppm
F1                 22134.81 Hz
F2P                 -10.000 ppm
F2                 -1006.13 Hz
PPMCM              11.50000 ppm/cm
HZCM             1157.04688 Hz/cm
```



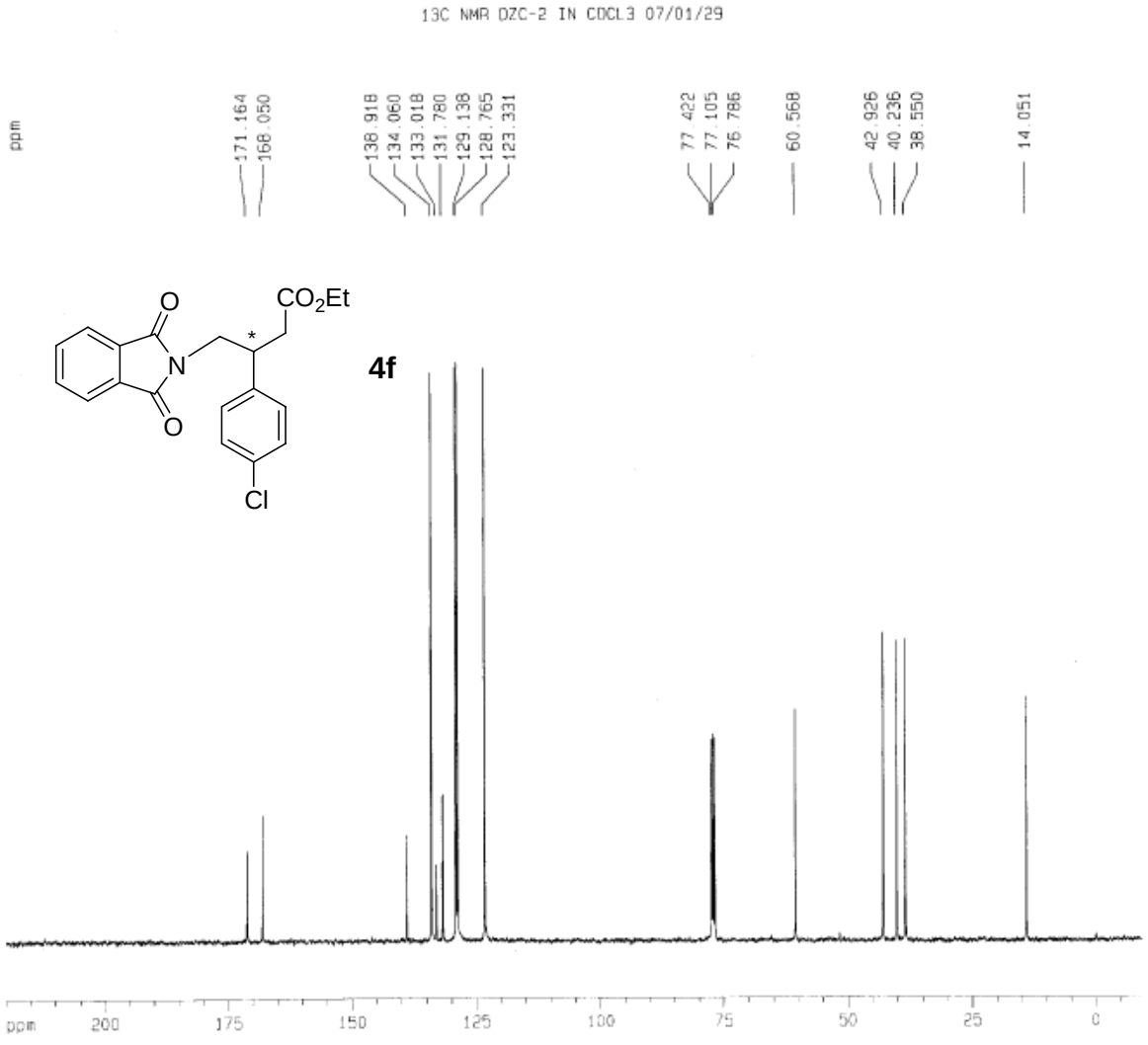

```
Current Data Parameters
NAME                 duan
EXPNO                 103
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070129
Time                17.40
INSTRUM           drx400
PROBHD   5 mm PABBO BB-
PULPROG                zg
TD                  32768
SOLVENT             CDCl3
NS                      8
DS                      0
SWH             8012.820 Hz
FIDRES          0.244532 Hz
AQ             2.0447731 sec
RG                   45.3
DW                 62.400 usec
DE                   6.00 usec
TE                    0.0 K
D1             1.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   8.20 usec
PL1                  5.00 dB
SFO1          400.1332521 MHz

F2 - Processing parameters
SI                  32768
SF            400.1300086 MHz
WDW                    EM
SSB                     0
LB                  -0.50 Hz
GB                    0.1
PC                   1.40

1D NMR plot parameters
CX                  20.00 cm
CY                   8.00 cm
F1P                 10.000 ppm
F1                4001.30 Hz
F2P                 -1.000 ppm
F2                -400.13 Hz
PPMCM             0.55000 ppm/cm
HZCM            220.07150 Hz/cm
```




```
Current Data Parameters
NAME                 duan
EXPNO                 104
PROCNO                  1

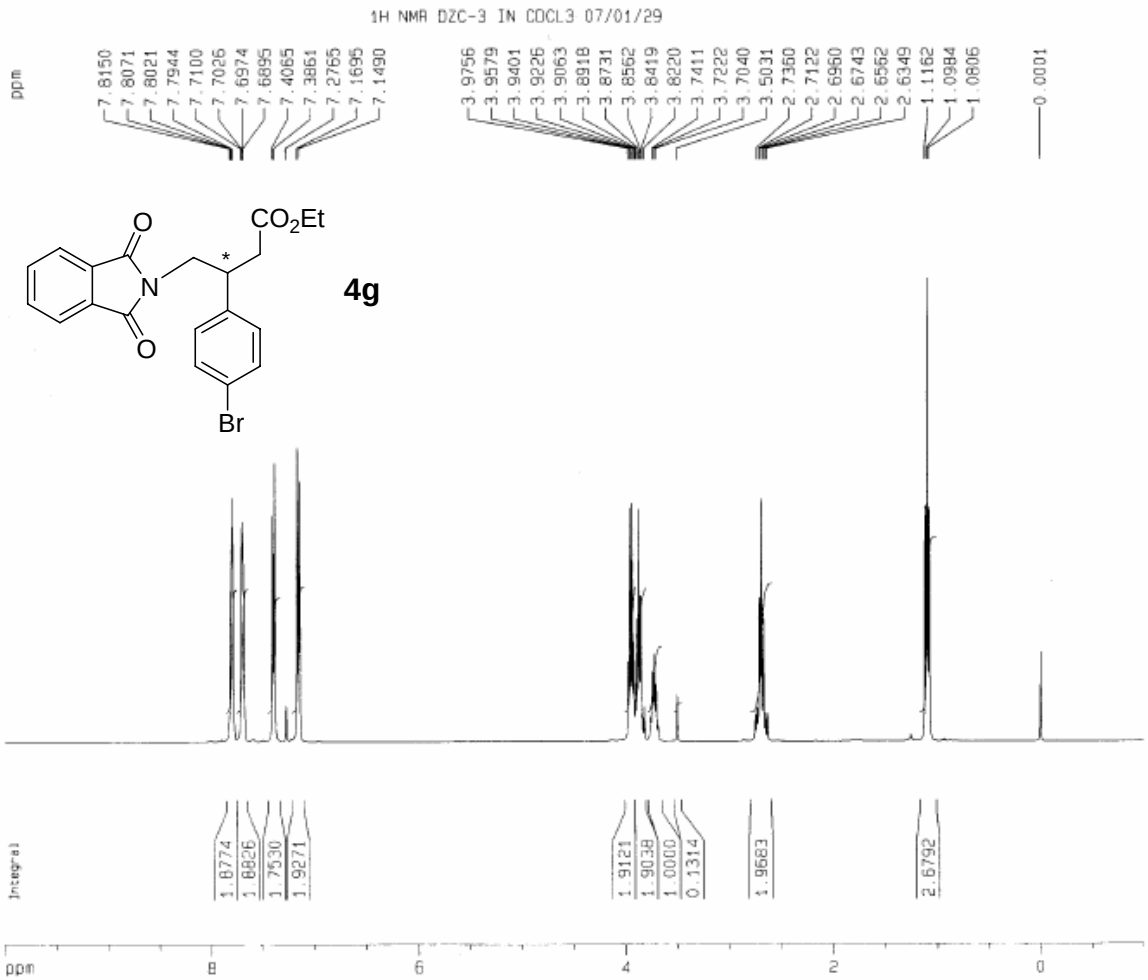
F2 - Acquisition Parameters
Date_            20070129
Time                17.46
INSTRUM           drx400
PROBHD   5 mm PABBO BB-
PULPROG              zgdc
TD                  65536
SOLVENT              DMSO
NS                    398
DS                     16
SWH            23148.148 Hz
FIDRES          0.353213 Hz
AQ             1.4156276 sec
RG                 16384
DW                21.600 usec
DE                  6.00 usec
TE                   0.0 K
D1             1.00000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.20 usec
PL1                 1.50 dB
SFO1          100.6234215 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 5.00 dB
PL12               23.00 dB
SFO2          400.1320007 MHz

F2 - Processing parameters
SI                  32768
SF            100.6127690 MHz
WDW                    EM
SSB                     0
LB                   4.00 Hz
GB                      0
PC                    1.40

1D NMR plot parameters
CX                  20.00 cm
CY                  10.00 cm
F1P                220.000 ppm
F1               22134.81 Hz
F2P                -10.000 ppm
F2               -1006.13 Hz
PPMCM              11.50000 ppm/cm
HZCM            1157.04688 Hz/cm
```



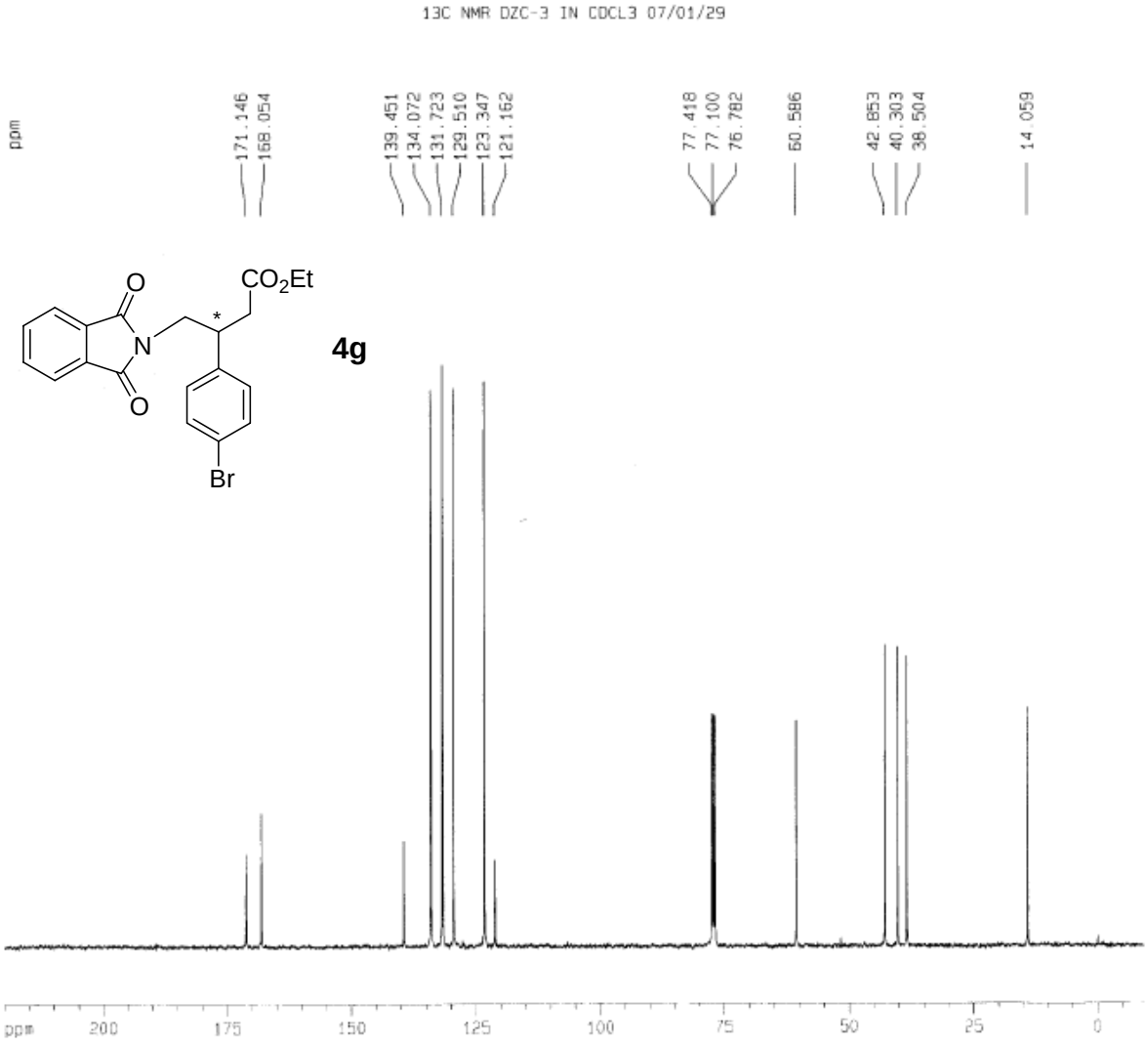

```
Current Data Parameters
NAME                 duan
EXPNO                 105
PROCNO                  1

F2 - Acquisition Parameters
Date_            20070129
Time                18.05
INSTRUM           drx400
PROBHD   5 mm PABBO BB-
PULPROG                zg
TD                  32768
SOLVENT             CDCl3
NS                      8
DS                      0
SWH              8012.820 Hz
FIDRES           0.244532 Hz
AQ             2.0447731 sec
RG                   40.3
DW                 62.400 usec
DE                  6.00 usec
TE                   0.0 K
D1             1.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                   1H
P1                   8.20 usec
PL1                  5.00 dB
SFO1          400.1332521 MHz

F2 - Processing parameters
SI                  32768
SF            400.1300108 MHz
WDW                    GM
SSB                     0
LB                  -0.50 Hz
GB                    0.1
PC                   1.40

1D NMR plot parameters
CX                  20.00 cm
CY                   8.00 cm
F1P                 10.000 ppm
F1                 4001.30 Hz
F2P                 -1.000 ppm
F2                 -400.13 Hz
PPMCM              0.55000 ppm/cm
HZCM             220.07150 Hz/cm
```




```
Current Data Parameters
NAME                 duan
EXPNO                 106
PROCNO                  1

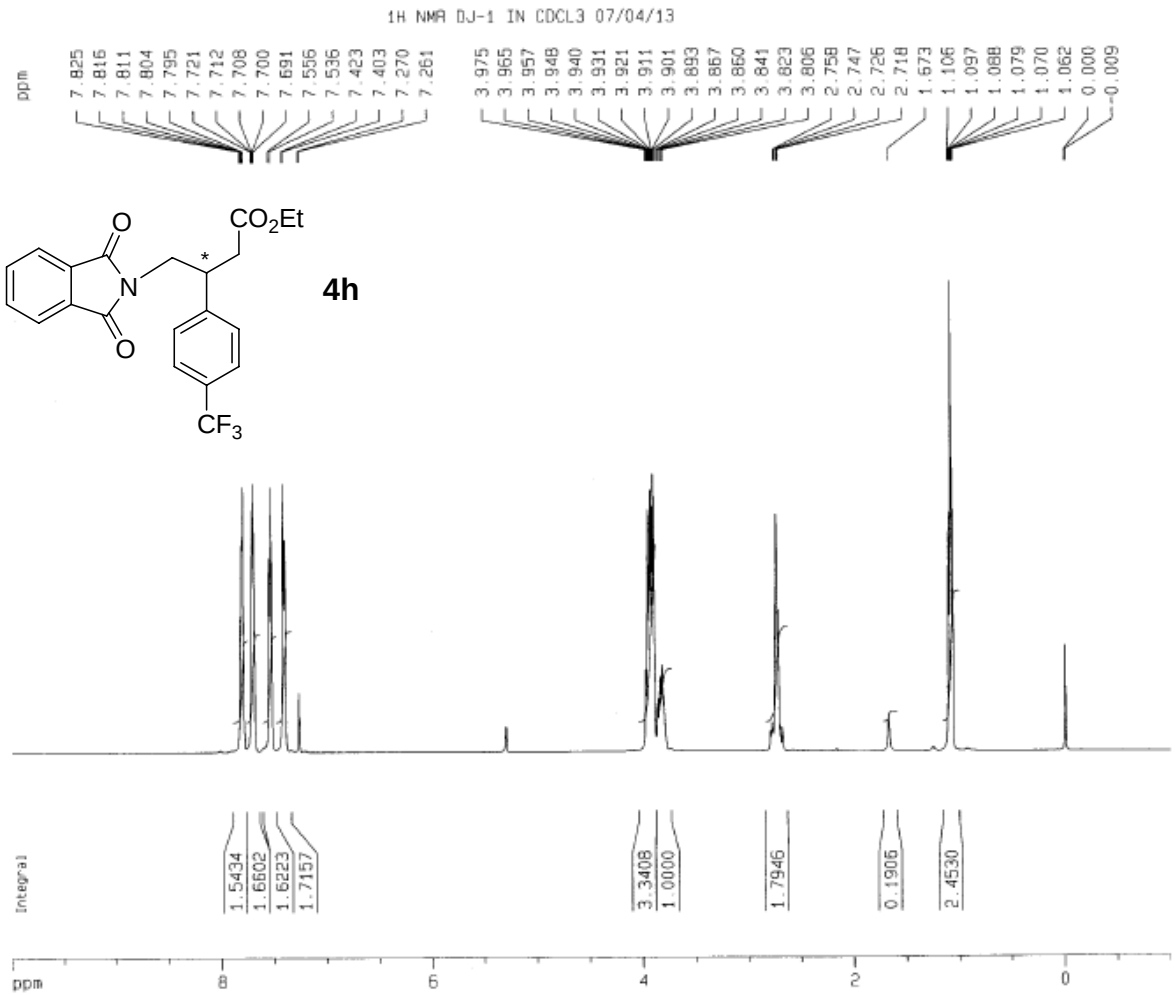
F2 - Acquisition Parameters
Date_           20070129
Time               18.10
INSTRUM          drx400
PROBHD   5 mm PABBO BB-
PULPROG             zgdc
TD                 65536
SOLVENT             DMSO
NS                    507
DS                     16
SWH            23148.148 Hz
FIDRES          0.353213 Hz
AQ             1.4156276 sec
RG                 16384
DW                21.600 usec
DE                 6.00 usec
TE                  0.0 K
D1             1.00000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.20 usec
PL1                 1.50 dB
SFO1          100.6234215 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              80.00 usec
PL2                 5.00 dB
PL12               23.00 dB
SFO2          400.1320007 MHz

F2 - Processing parameters
SI                  32768
SF            100.6127690 MHz
WDW                    EM
SSB                     0
LB                   4.00 Hz
GB                      0
PC                    1.40

1D NMR plot parameters
CX                  20.00 cm
CY                  10.00 cm
F1P               220.000 ppm
F1               22134.81 Hz
F2P               -10.000 ppm
F2               -1006.13 Hz
PPMCM            11.50000 ppm/cm
HZCM           1157.04688 Hz/cm
```



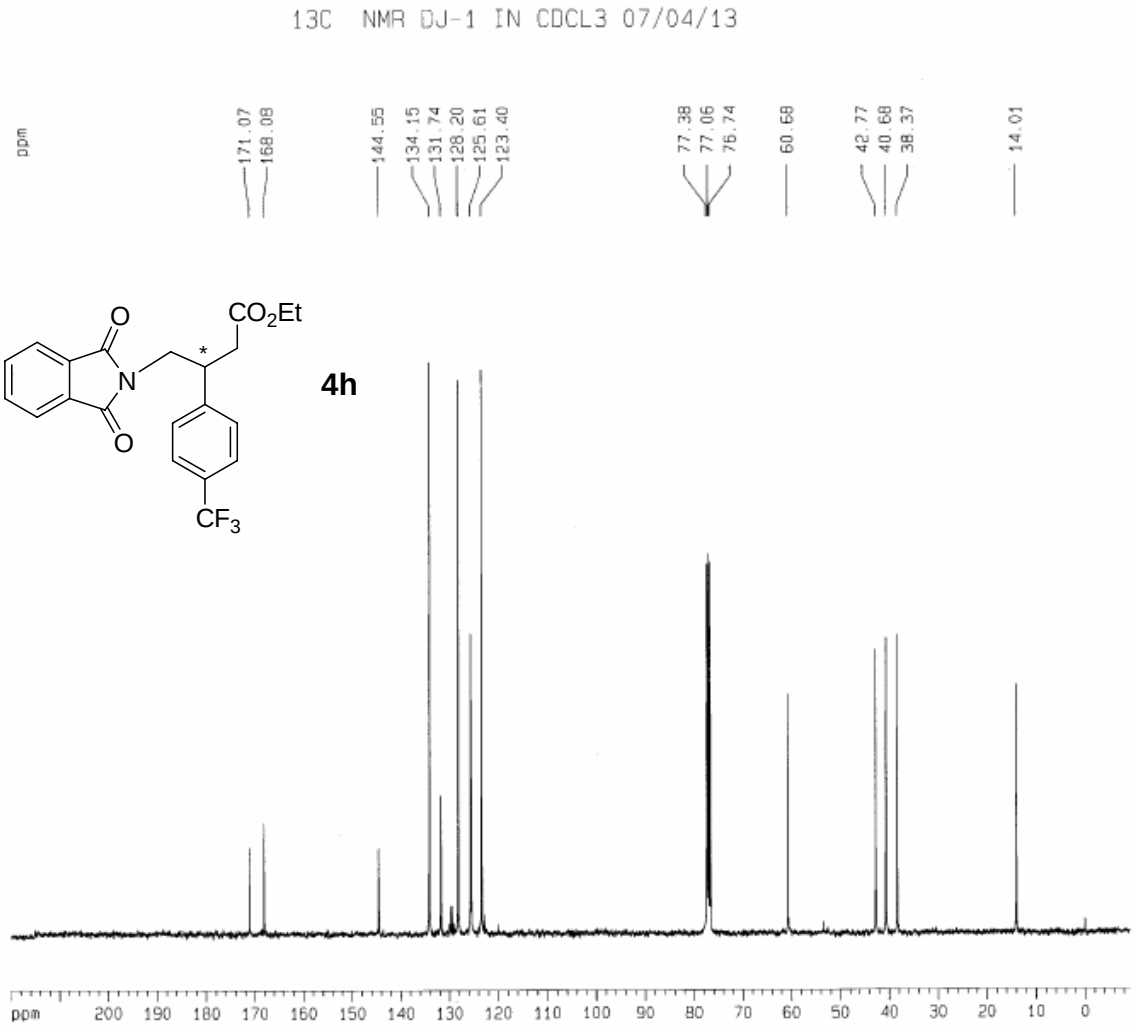

```
Current Data Parameters
NAME                 dj
EXPNO              376
PROCNO               1

F2 - Acquisition Parameters
Date_          20070413
Time              11.34
INSTRUM          drx400
PROBHD   5 mm PABBO BB-
PULPROG              zg
TD                32768
SOLVENT           CDCl3
NS                    8
DS                    0
SWH            8012.820 Hz
FIDRES         0.244532 Hz
AQ           2.0447731 sec
RG                   64
DW               62.400 usec
DE                6.00 usec
TE               295.2 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 8.20 usec
PL1                5.00 dB
SFO1        400.1332521 MHz

F2 - Processing parameters
SI                32768
SF          400.1300142 MHz
WDW                  GM
SSB                   0
LB                -0.50 Hz
GB                  0.1
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
CY                 8.00 cm
F1P               10.000 ppm
F1               4001.30 Hz
F2P               -1.000 ppm
F2               -400.13 Hz
PPMCM            0.55000 ppm/cm
HZCM           220.07150 Hz/cm
```




```
Current Data Parameters
NAME                  dj
EXPNO                377
PROCNO                 1

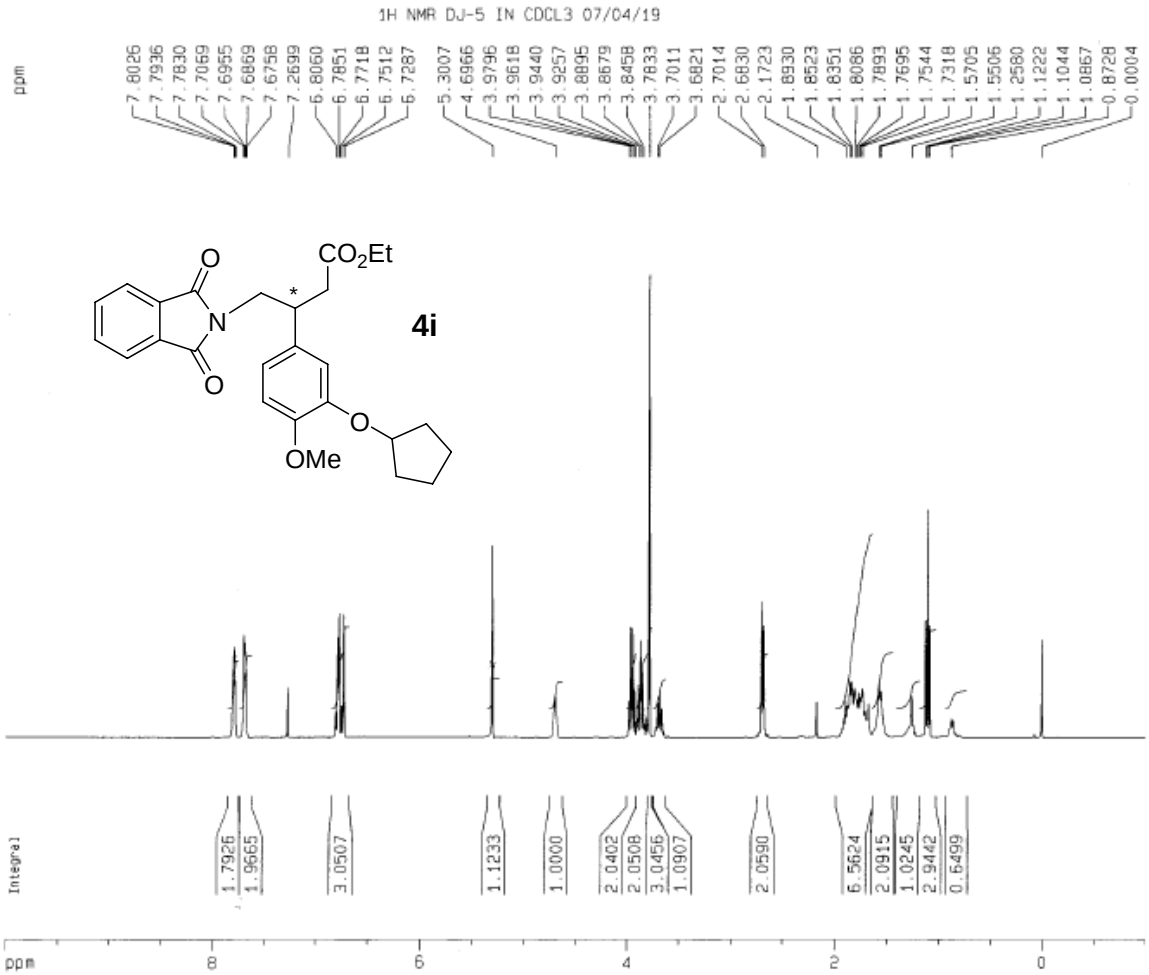
F2 - Acquisition Parameters
Date_           20070413
Time               12.30
INSTRUM          drx400
PROBHD   5 mm PABBO BB-
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                  1288
DS                    16
SWH            23148.148 Hz
FIDRES          0.353213 Hz
AQ             1.4156276 sec
RG                 16384
DW                21.600 usec
DE                 6.00 usec
TE                 296.2 K
D1             1.00000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.20 usec
PL1                 1.50 dB
SFO1          100.6234215 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                 5.00 dB
PL12               23.00 dB
SFO2          400.1320007 MHz

F2 - Processing parameters
SI                 32768
SF            100.6127690 MHz
WDW                   EM
SSB                    0
LB                  4.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
CY                 10.00 cm
F1P               220.000 ppm
F1              22134.81 Hz
F2P               -10.000 ppm
F2              -1006.13 Hz
PPMCM             11.50000 ppm/cm
HZCM            1157.04688 Hz/cm
```



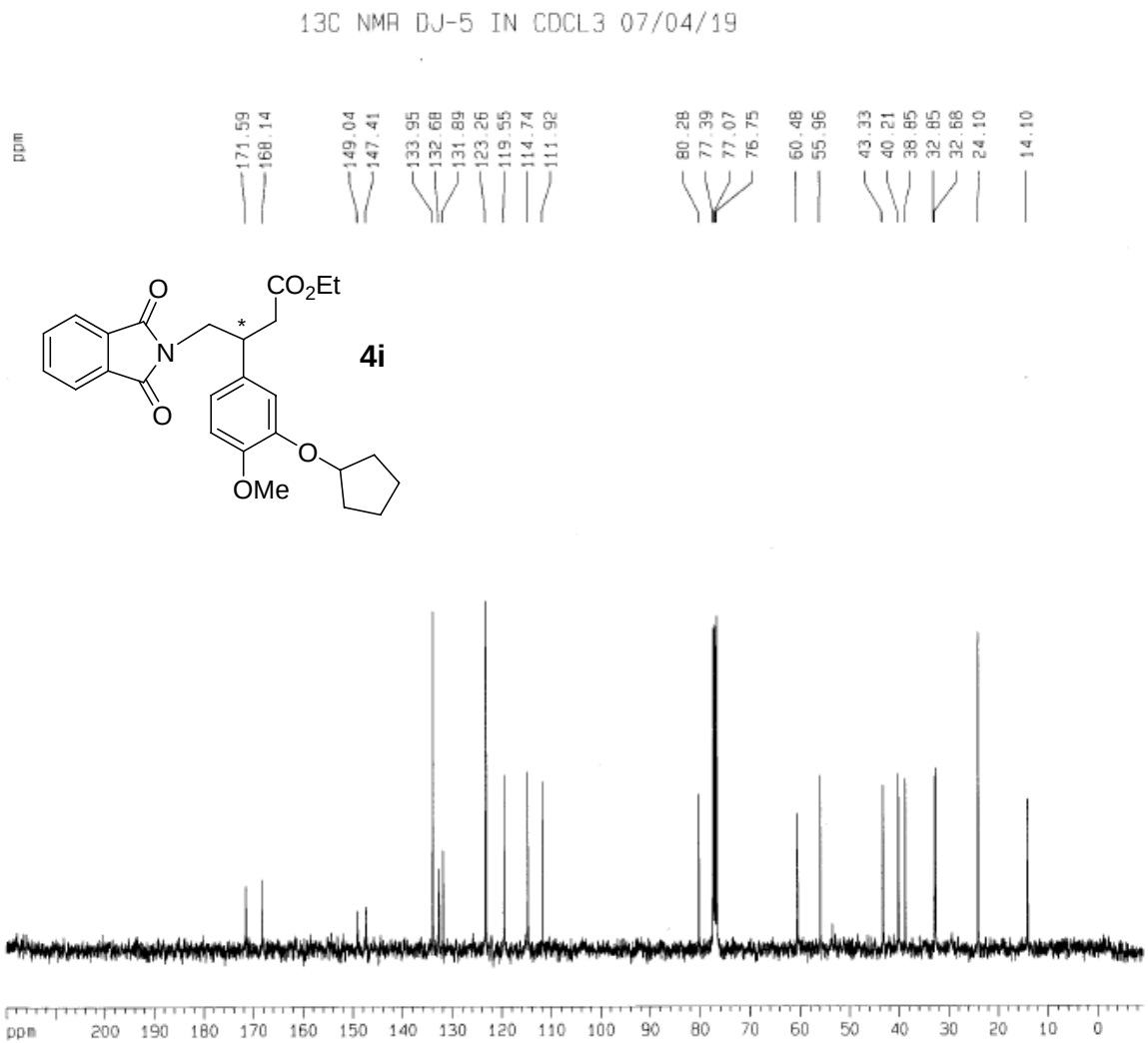

```
Current Data Parameters
NAME                  dj
EXPNO                400
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070419
Time               12.49
INSTRUM          drx400
PROBHD   5 mm PABBO BB-
PULPROG               zg
TD                32768
SOLVENT           CDCl3
NS                    8
DS                    0
SWH            8012.820 Hz
FIDRES         0.244532 Hz
AQ           2.0447731 sec
RG                 71.8
DW               62.400 usec
DE                6.00 usec
TE               295.2 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 8.20 usec
PL1                5.00 dB
SFO1        400.1332521 MHz

F2 - Processing parameters
SI                32768
SF          400.1300133 MHz
WDW                  GM
SSB                   0
LB                -0.50 Hz
GB                  0.1
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
CY                 8.00 cm
F1P               10.000 ppm
F1               4001.30 Hz
F2P               -1.000 ppm
F2               -400.13 Hz
PPMCM            0.55000 ppm/cm
HZCM           220.07150 Hz/cm
```




```
Current Data Parameters
NAME                  dj
EXPNO                401
PROCNO                 1

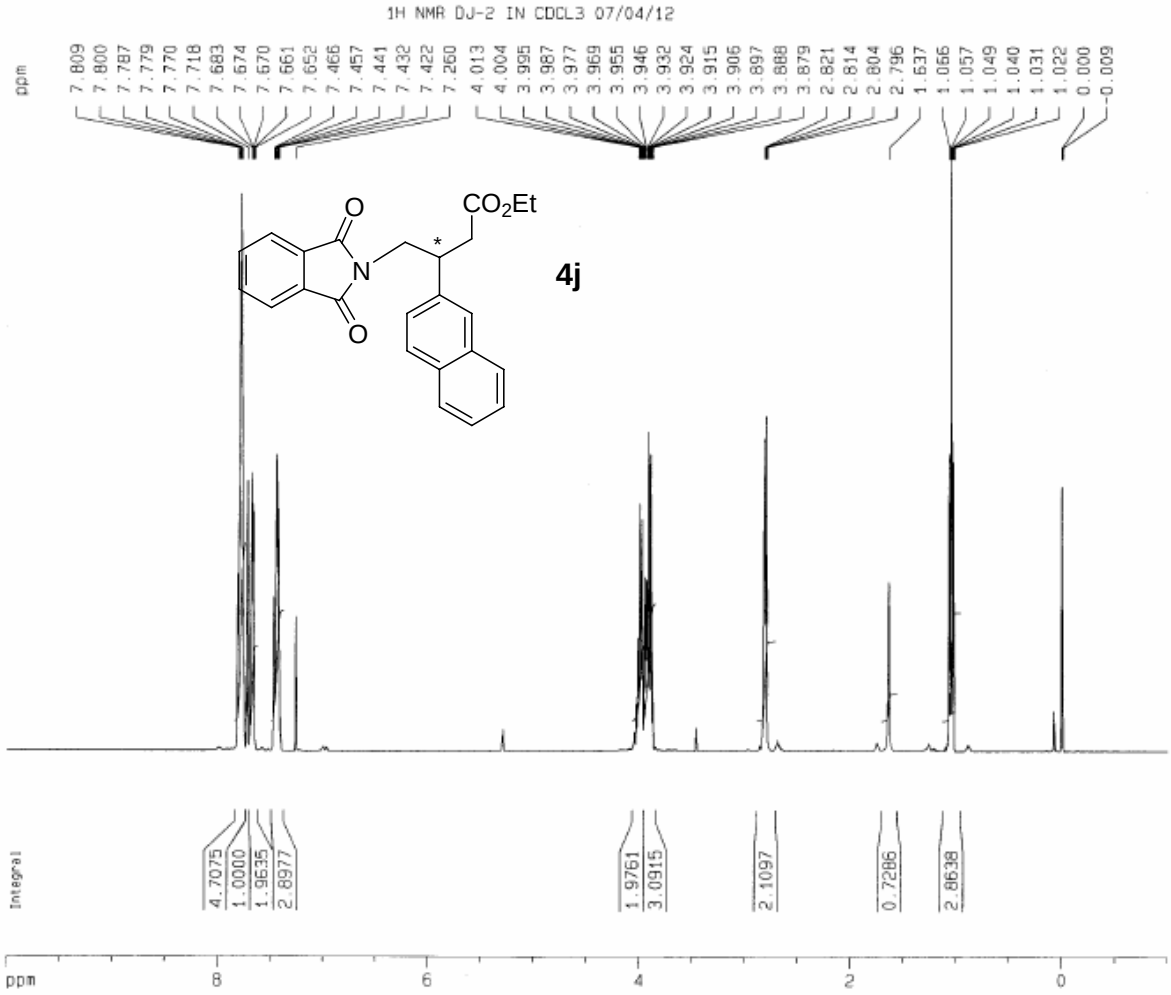
F2 - Acquisition Parameters
Date_           20070419
Time               12.57
INSTRUM          drx400
PROBHD   5 mm PABBO BB-
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                   102
DS                    16
SWH            23148.148 Hz
FIDRES          0.353213 Hz
AQ             1.4156276 sec
RG                 16384
DW                21.600 usec
DE                 6.00 usec
TE                296.2 K
D1             1.00000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.20 usec
PL1                 1.50 dB
SFO1          100.6234215 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                5.00 dB
PL12              23.00 dB
SFO2          400.1320007 MHz

F2 - Processing parameters
SI                 32768
SF            100.6127690 MHz
WDW                   EM
SSB                    0
LB                  4.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
CY                  6.00 cm
F1P               220.000 ppm
F1               22134.81 Hz
F2P               -10.000 ppm
F2               -1006.13 Hz
PPMCM            11.50000 ppm/cm
HZCM           1157.04688 Hz/cm
```




```
Current Data Parameters
NAME                  dj
EXPNO                371
PROCNO                 1

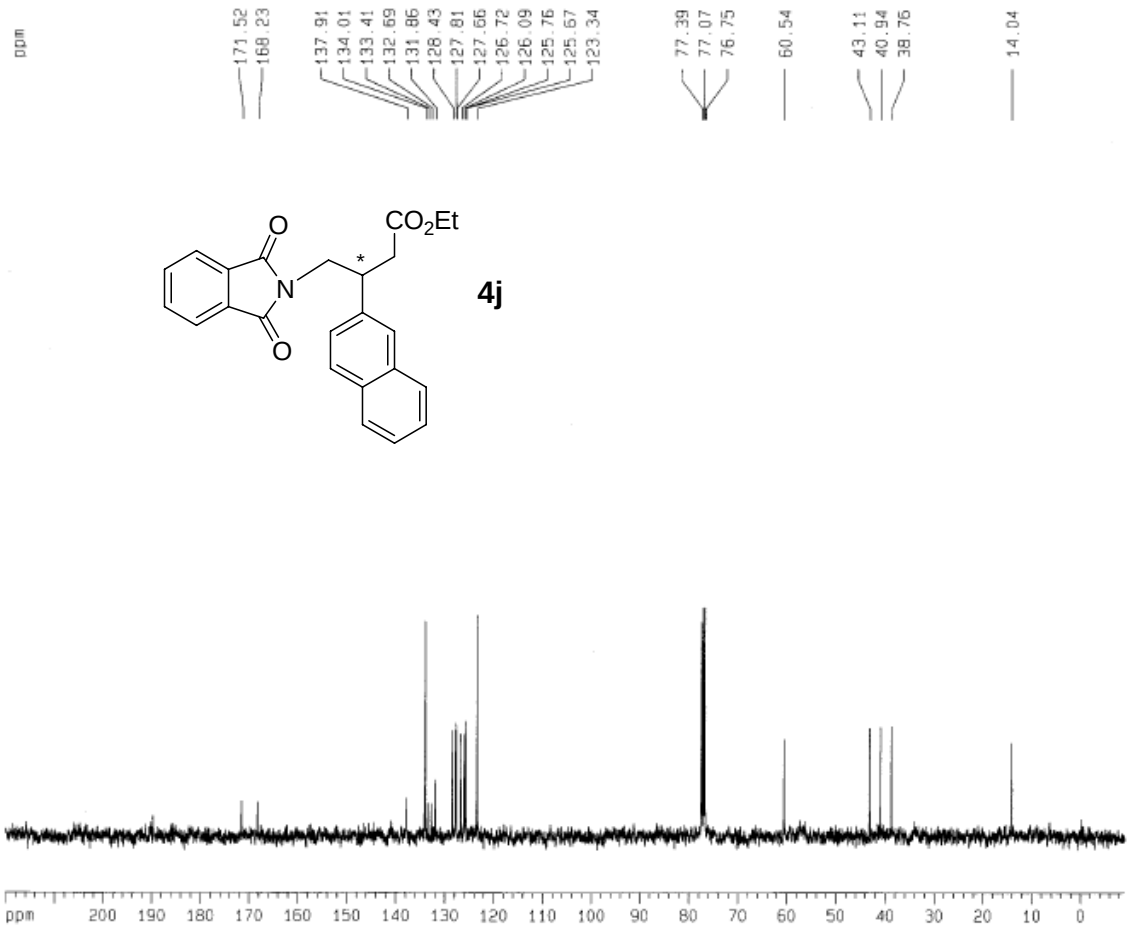
F2 - Acquisition Parameters
Date_           20070412
Time               10.44
INSTRUM          drx400
PROBHD   5 mm PABBO BB-
PULPROG               zg
TD                 32768
SOLVENT            CDCl3
NS                     8
DS                     0
SWH            8012.820 Hz
FIDRES         0.244532 Hz
AQ           2.0447731 sec
RG                   128
DW               62.400 usec
DE                6.00 usec
TE               293.2 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

-------- CHANNEL f1 --------
NUC1                  1H
P1                 8.20 usec
PL1                5.00 dB
SFO1        400.1332521 MHz

F2 - Processing parameters
SI                 32768
SF          400.1300184 MHz
WDW                   GM
SSB                    0
LB                 -0.50 Hz
GB                   0.1
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
CY                 10.00 cm
F1P               10.000 ppm
F1               4001.30 Hz
F2P               -1.000 ppm
F2               -400.13 Hz
PPMCM            0.55000 ppm/cm
HZCM           220.07150 Hz/cm
```

13C NMR DJ-2 IN CDCL3 07/04/12




```
Current Data Parameters
NAME                  dj
EXPNO                372
PROCNO                 1

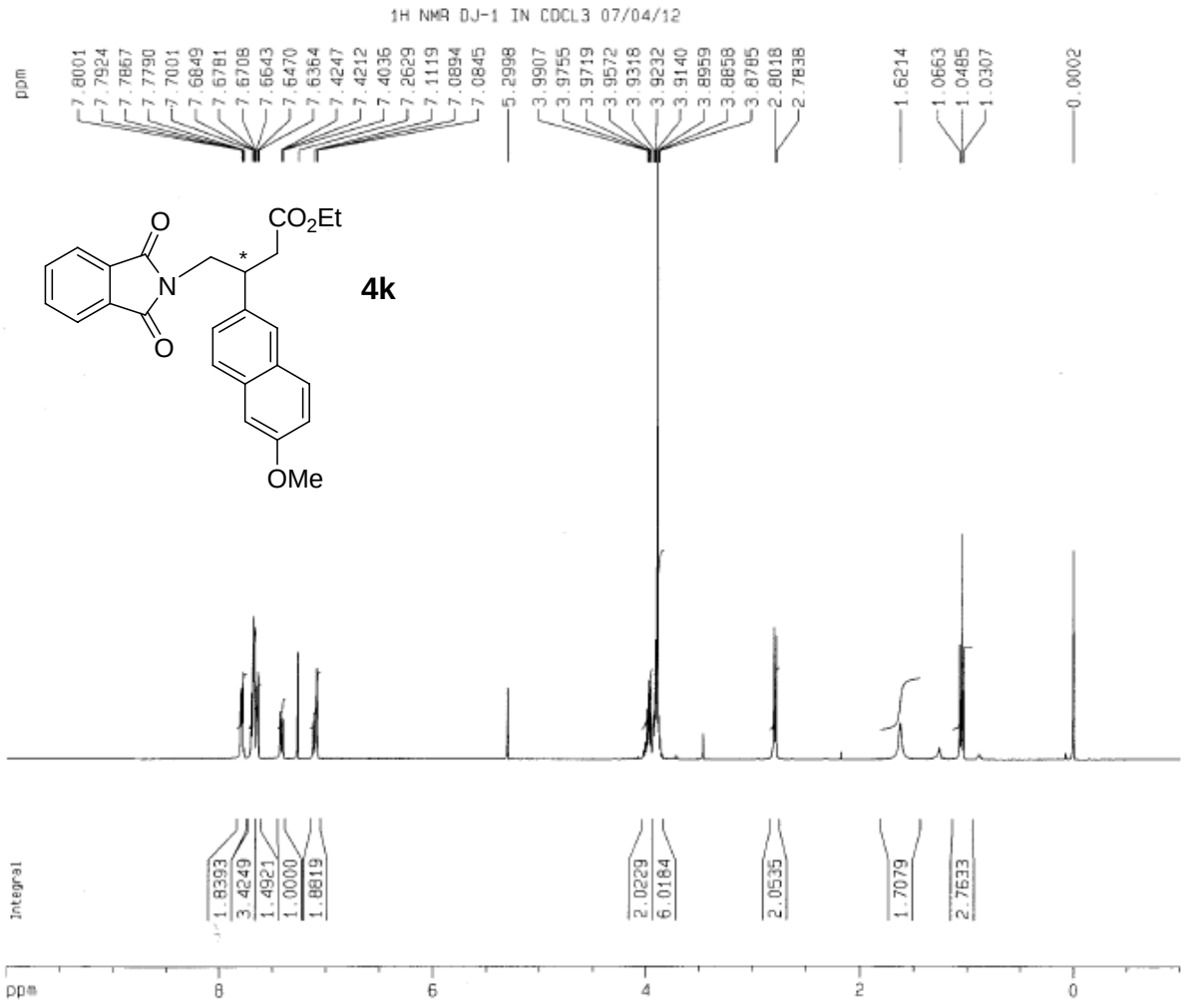
F2 - Acquisition Parameters
Date_           20070412
Time               10.49
INSTRUM          drx400
PROBHD   5 mm PABBO BB-
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                    67
DS                    16
SWH            23148.148 Hz
FIDRES          0.353213 Hz
AQ             1.4156276 sec
RG                 16384
DW                21.600 usec
DE                  6.00 usec
TE                 294.2 K
D1             1.00000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                   8.20 usec
PL1                  1.50 dB
SFO1          100.6234215 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                   1H
PCPD2               80.00 usec
PL2                  5.00 dB
PL12                23.00 dB
SFO2          400.1320007 MHz

F2 - Processing parameters
SI                    32768
SF            100.6127690 MHz
WDW                      EM
SSB                       0
LB                     4.00 Hz
GB                        0
PC                     1.40

1D NMR plot parameters
CX                    20.00 cm
CY                     4.00 cm
F1P                  220.000 ppm
F1                 22134.81 Hz
F2P                 -10.000 ppm
F2                 -1006.13 Hz
PPMCM               11.50000 ppm/cm
HZCM              1157.04688 Hz/cm
```



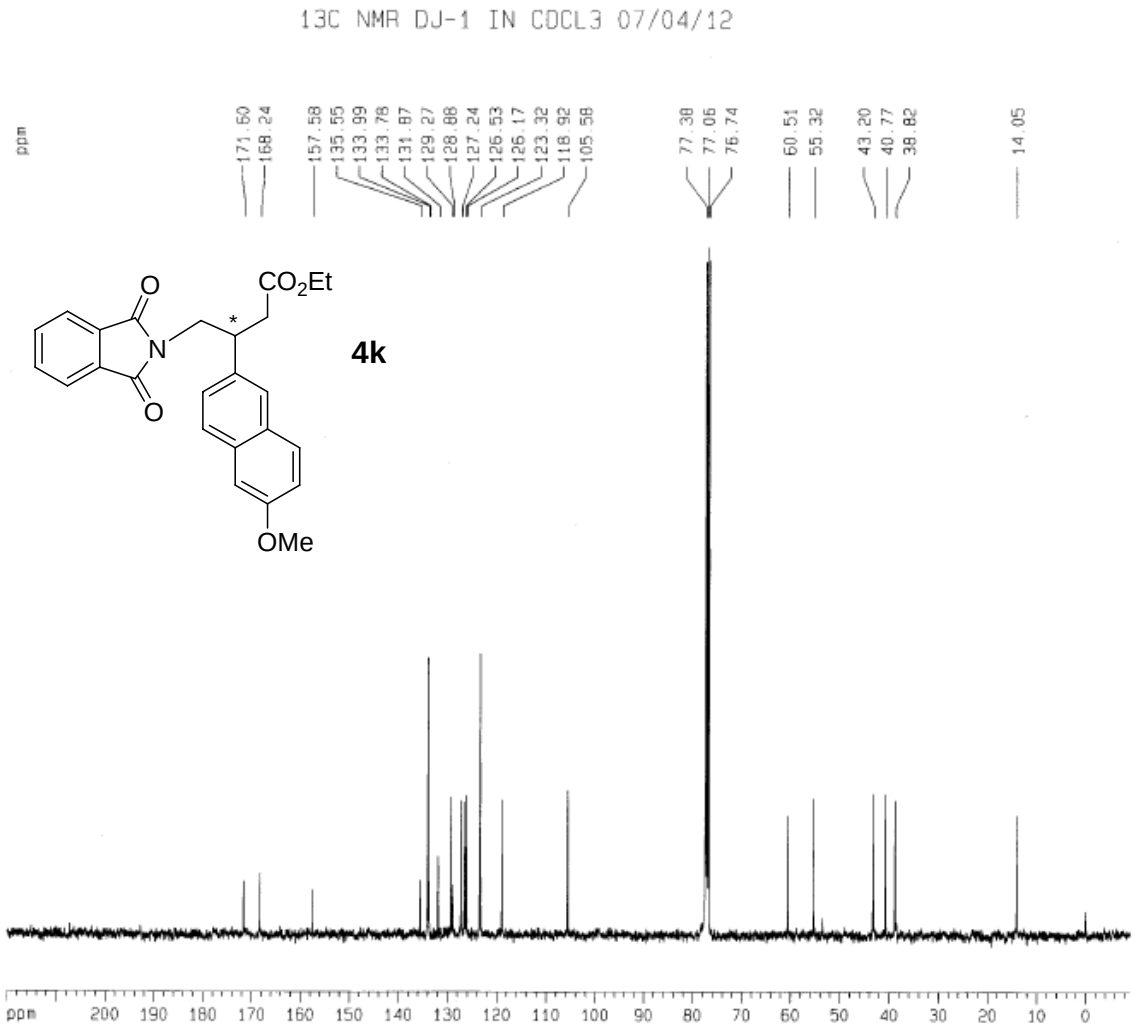

```
Current Data Parameters
NAME                 dj
EXPNO               369
PROCNO                1

F2 - Acquisition Parameters
Date_          20070412
Time               9.54
INSTRUM          drx400
PROBHD   5 mm PABBO BB-
PULPROG              zg
TD                32768
SOLVENT           CDCl3
NS                    8
DS                    0
SWH            8012.820 Hz
FIDRES         0.244532 Hz
AQ           2.0447731 sec
RG                  128
DW               62.400 usec
DE                6.00 usec
TE               293.2 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

-------- CHANNEL f1 --------
NUC1                 1H
P1                8.20 usec
PL1               5.00 dB
SFO1        400.1332521 MHz

F2 - Processing parameters
SI                32768
SF          400.1300162 MHz
WDW                  GM
SSB                   0
LB                -0.50 Hz
GB                  0.1
PC                 1.40

1D NMR plot parameters
CX               20.00 cm
CY               10.00 cm
F1P             10.000 ppm
F1             4001.30 Hz
F2P             -1.000 ppm
F2             -400.13 Hz
PPMCM          0.55000 ppm/cm
HZCM         220.07150 Hz/cm
```




```
Current Data Parameters
NAME                    d)
EXPNO                  370
PROCNO                   1

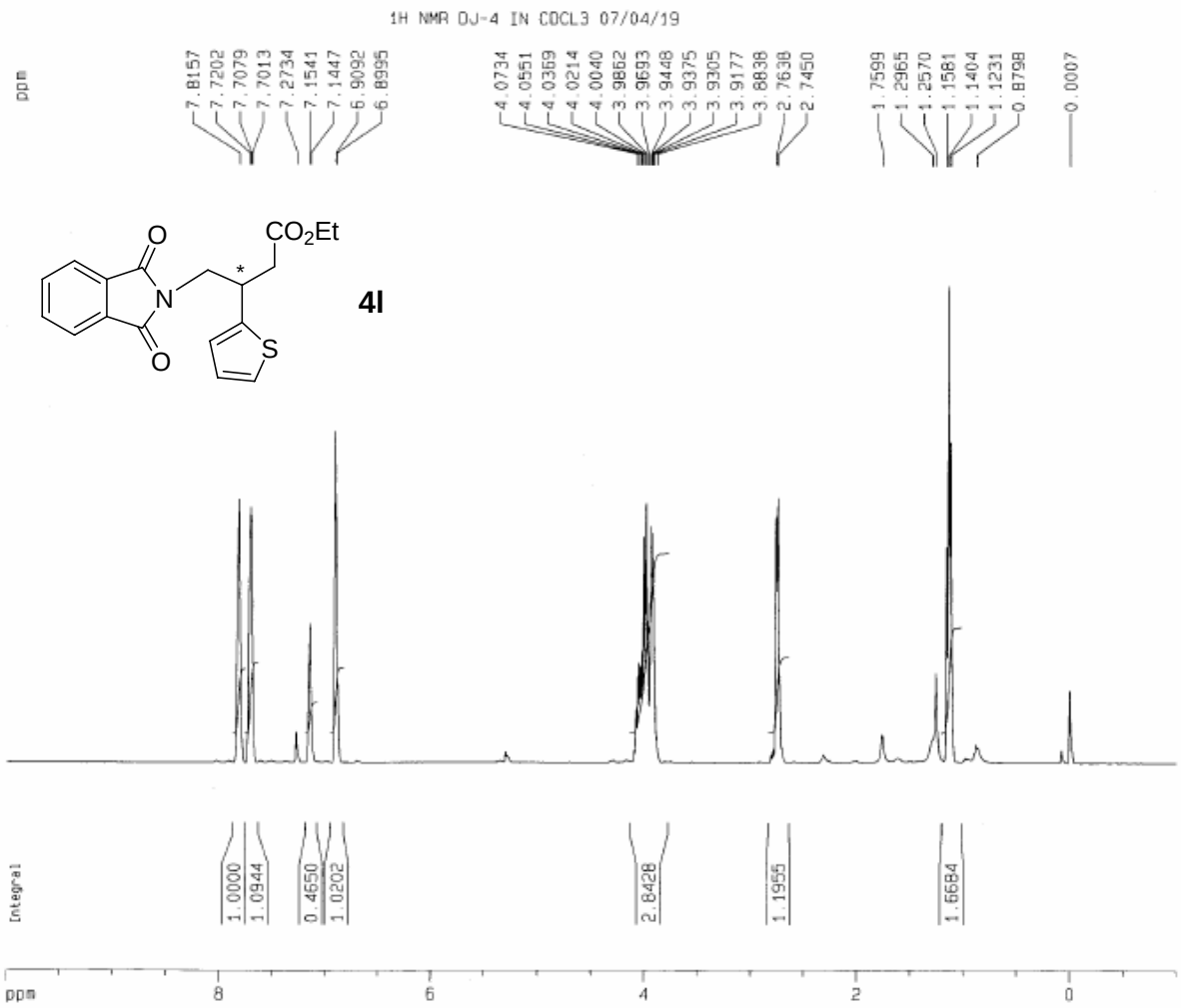
F2 - Acquisition Parameters
Date_            20070412
Time                 10.10
INSTRUM             drx400
PROBHD   5 mm PABBO BB-
PULPROG               zgdc
TD                    65536
SOLVENT               CDCl3
NS                      995
DS                       16
SWH             23148.148 Hz
FIDRES           0.353213 Hz
AQ              1.4156276 sec
RG                   16384
DW                 21.600 usec
DE                  6.00 usec
TE                 294.2 K
D1              1.00000000 sec
d11             0.03000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                   13C
P1                    8.20 usec
PL1                   1.50 dB
SFO1          100.6234215 MHz

======== CHANNEL f2 ========
CPDPRG2            waltz16
NUC2                    1H
PCPD2               80.00 usec
PL2                  5.00 dB
PL12                23.00 dB
SFO2          400.1320007 MHz

F2 - Processing parameters
SI                   32768
SF            100.6127690 MHz
WDW                     EM
SSB                      0
LB                    4.00 Hz
GB                       0
PC                    1.40

1D NMR plot parameters
CX                   20.00 cm
CY                   12.00 cm
F1P                 220.000 ppm
F1                22134.81 Hz
F2P                -10.000 ppm
F2                -1006.13 Hz
PPMCM              11.50000 ppm/cm
HZCM            1157.04688 Hz/cm
```




```
Current Data Parameters
NAME                  dj
EXPNO                 398
PROCNO                  1

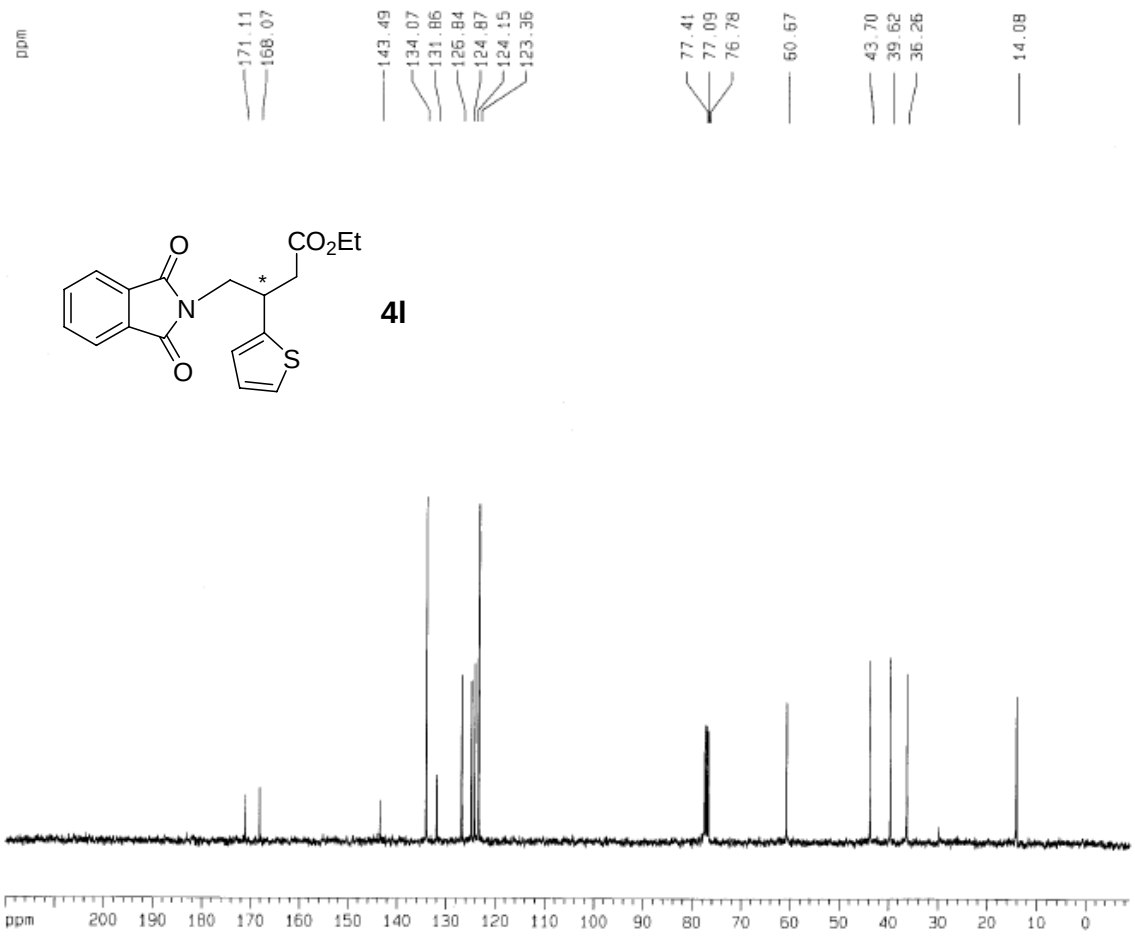
F2 - Acquisition Parameters
Date_            20070419
Time                12.38
INSTRUM           drx400
PROBHD   5 mm PABBO BB-
PULPROG                zg
TD                  32768
SOLVENT             CDCl3
NS                      8
DS                      0
SWH              8012.820 Hz
FIDRES           0.244532 Hz
AQ             2.0447731 sec
RG                   50.8
DW                 62.400 usec
DE                   6.00 usec
TE                  295.2 K
D1             1.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

-------- CHANNEL f1 --------
NUC1                   1H
P1                   8.20 usec
PL1                  5.00 dB
SFO1          400.1332521 MHz

F2 - Processing parameters
SI                  32768
SF            400.1300128 MHz
WDW                    GM
SSB                     0
LB                  -0.50 Hz
GB                    0.1
PC                   1.40

1D NMR plot parameters
CX                  20.00 cm
CY                   8.00 cm
F1P                 10.000 ppm
F1                 4001.30 Hz
F2P                 -1.000 ppm
F2                 -400.13 Hz
PPMCM              0.55000 ppm/cm
HZCM             220.07150 Hz/cm
```

13C NMR DJ-4 IN CDCL3 07/04/19



4l

```
Current Data Parameters
NAME                  dj
EXPNO                399
PROCNO                 1

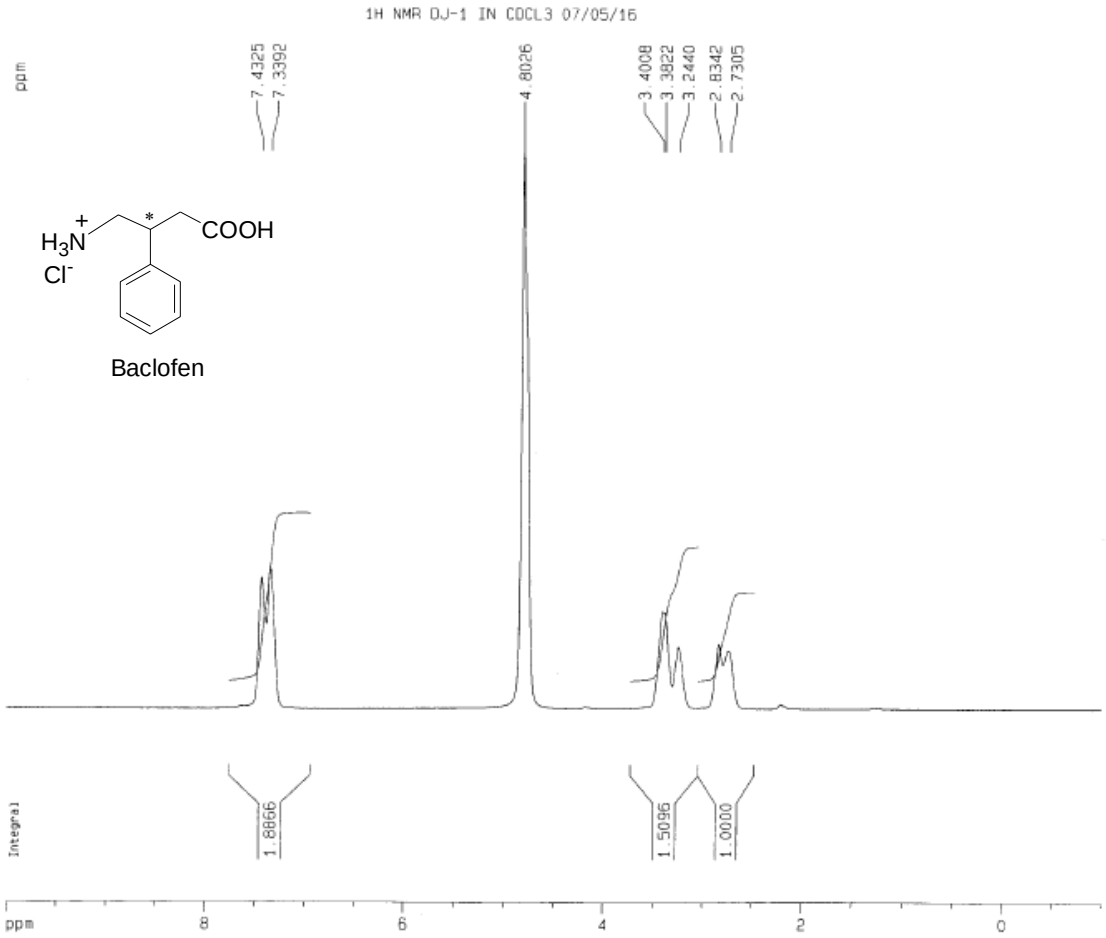
F2 - Acquisition Parameters
Date_           20070419
Time               12.44
INSTRUM          drx400
PROBHD   5 mm PABBO BB-
PULPROG             zgdc
TD                 65536
SOLVENT            CDCl3
NS                    90
DS                    16
SWH            23148.148 Hz
FIDRES          0.353213 Hz
AQ             1.4156276 sec
RG                 16384
DW               21.600 usec
DE                 6.00 usec
TE                296.2 K
D1             1.00000000 sec
d11            0.03000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  8.20 usec
PL1                 1.50 dB
SFO1          100.6234215 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                 5.00 dB
PL12               23.00 dB
SFO2          400.1320007 MHz

F2 - Processing parameters
SI                  32768
SF            100.6127690 MHz
WDW                    EM
SSB                     0
LB                   4.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  20.00 cm
CY                   6.00 cm
F1P               220.000 ppm
F1               22134.81 Hz
F2P               -10.000 ppm
F2               -1006.13 Hz
PPMCM            11.50000 ppm/cm
HZCM           1157.04688 Hz/cm
```



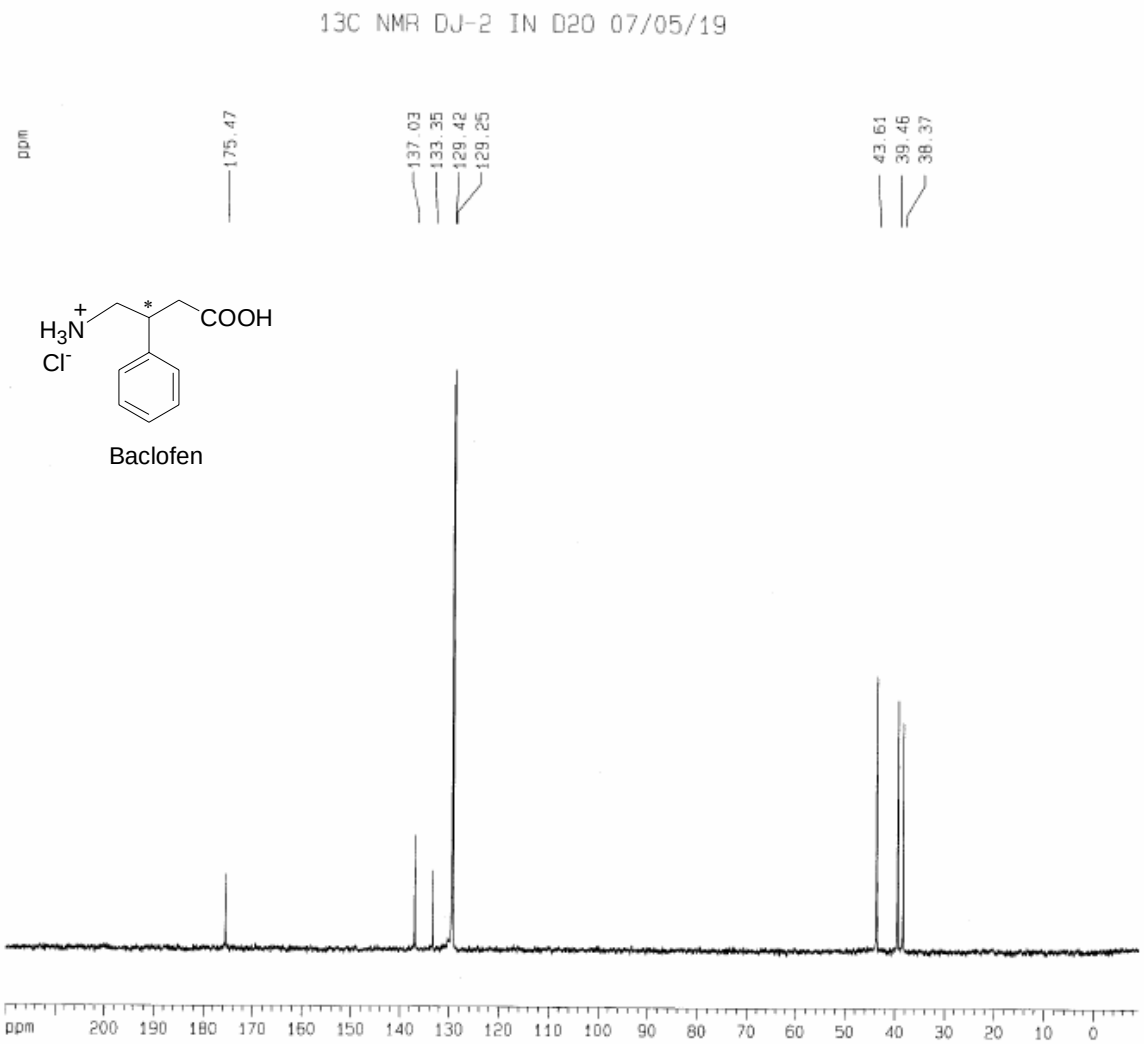

```
Current Data Parameters
NAME                  dj
EXPNO                431
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070516
Time                9.53
INSTRUM           drx400
PROBHD   5 mm PABBO BB-
PULPROG               zg
TD                 32768
SOLVENT              D2O
NS                     8
DS                     0
SWH            8012.820 Hz
FIDRES         0.244532 Hz
AQ           2.0447731 sec
RG                   128
DW               62.400 usec
DE                 6.00 usec
TE                295.2 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                  8.20 usec
PL1                 5.00 dB
SFO1        400.1332521 MHz

F2 - Processing parameters
SI                 32768
SF          400.1299592 MHz
WDW                   GM
SSB                    0
LB                 -0.50 Hz
GB                   0.1
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
CY                 10.00 cm
F1P               10.000 ppm
F1               4001.30 Hz
F2P               -1.000 ppm
F2                -400.13 Hz
PPMCM            0.55000 ppm/cm
HZCM           220.07149 Hz/cm
```




```
Current Data Parameters
NAME                 dj
EXPNO               434
PROCNO                1

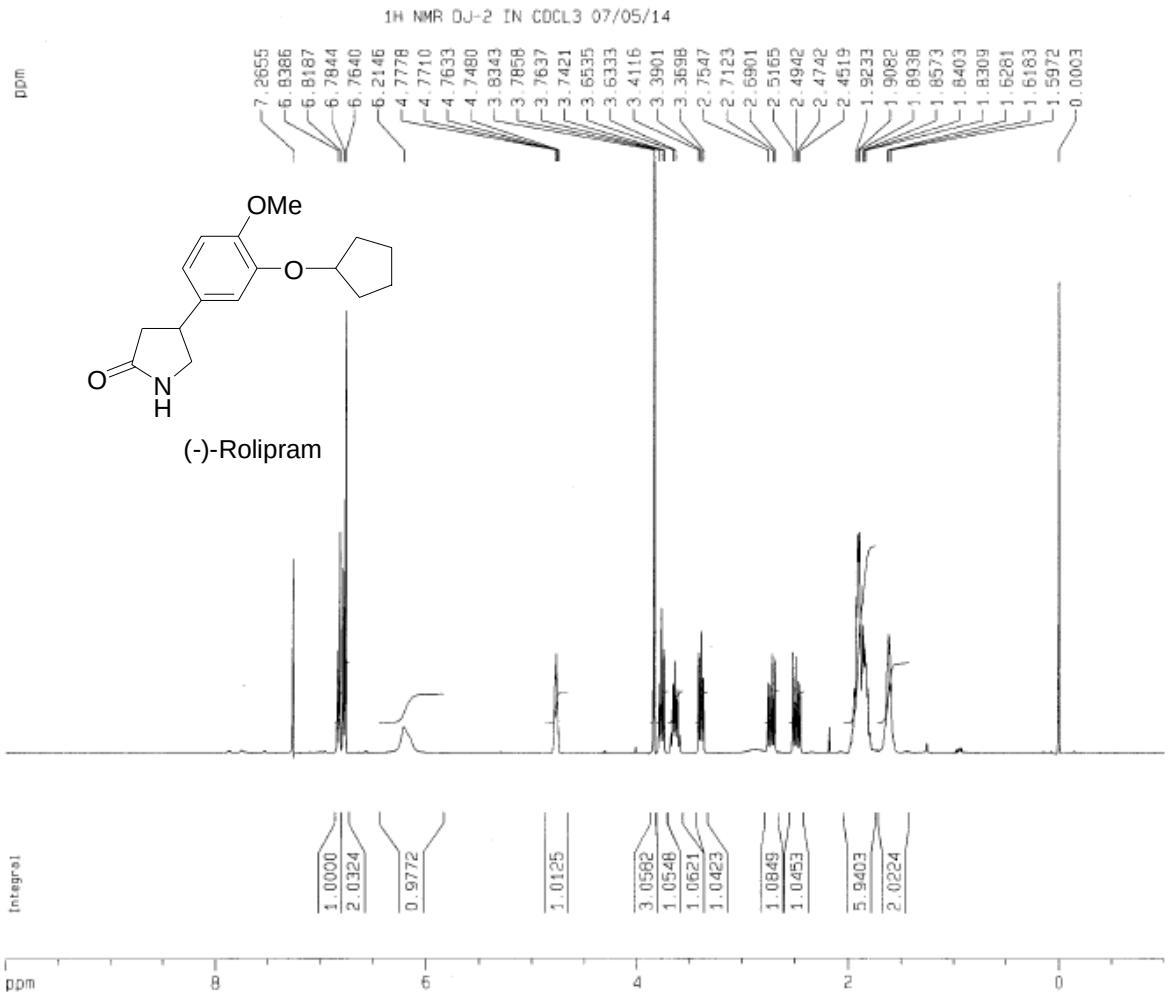
F2 - Acquisition Parameters
Date_          20070519
Time              13.19
INSTRUM          drx400
PROBHD   5 mm PABBO BB-
PULPROG            zgdc
TD                65536
SOLVENT             D2O
NS                 2159
DS                   16
SWH           23148.148 Hz
FIDRES         0.353213 Hz
AQ            1.4156276 sec
RG                16384
DW               21.600 usec
DE                6.00 usec
TE               298.2 K
D1           1.00000000 sec
d11          0.03000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 8.20 usec
PL1                1.50 dB
SFO1        100.6234215 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2             80.00 usec
PL2                5.00 dB
PL12              23.00 dB
SFO2        400.1320007 MHz

F2 - Processing parameters
SI                 32768
SF          100.6127690 MHz
WDW                   EM
SSB                    0
LB                  4.00 Hz
GB                     0
PC                  1.40

1D NMR plot parameters
CX                 20.00 cm
CY                 10.00 cm
F1P               220.000 ppm
F1              22134.81 Hz
F2P               -10.000 ppm
F2              -1006.13 Hz
PPMCM            11.50000 ppm/cm
HZCM          1157.04688 Hz/cm
```



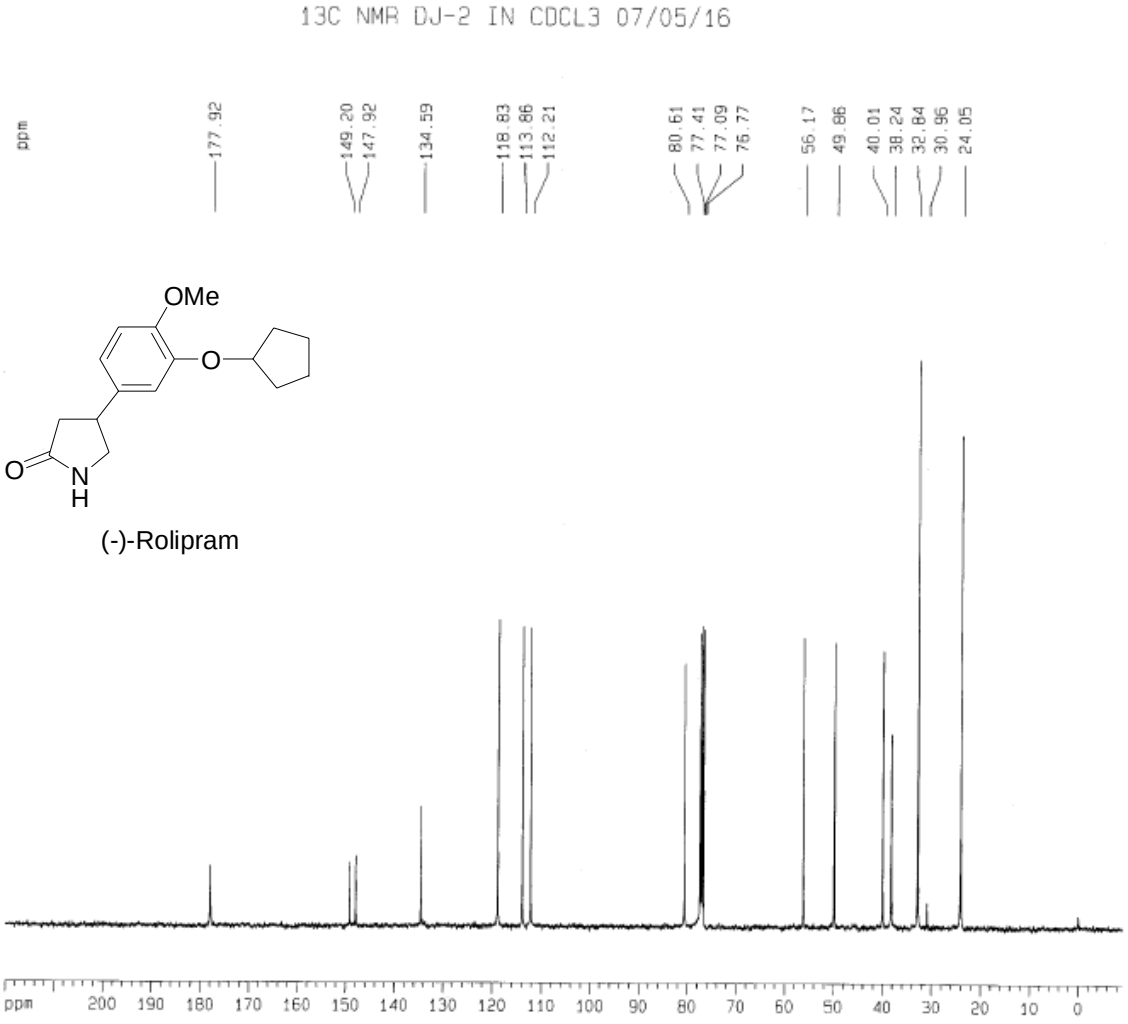

```
Current Data Parameters
NAME                 dj
EXPNO               428
PROCNO                1

F2 - Acquisition Parameters
Date_          20070514
Time              13.39
INSTRUM          drx400
PROBHD   5 mm PABBO BB-
PULPROG              zg
TD                32768
SOLVENT           CDCl3
NS                    8
DS                    0
SWH            8012.820 Hz
FIDRES         0.244532 Hz
AQ            2.0447731 sec
RG                  128
DW               62.400 usec
DE                6.00 usec
TE               295.2 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 8.20 usec
PL1                5.00 dB
SFO1        400.1332521 MHz

F2 - Processing parameters
SI                32768
SF          400.1300155 MHz
WDW                  EM
SSB                   0
LB                -0.50 Hz
GB                  0.1
PC                 1.40

1D NMR plot parameters
CX                20.00 cm
CY                20.00 cm
F1P              10.000 ppm
F1              4001.30 Hz
F2P              -1.000 ppm
F2              -400.13 Hz
PPMCM           0.55000 ppm/cm
HZCM          220.07150 Hz/cm
```




```
Current Data Parameters
NAME                  dj
EXPNO                432
PROCNO                 1

F2 - Acquisition Parameters
Date_           20070516
Time               10.53
INSTRUM           drx400
PROBHD   5 mm PABBO BB-
PULPROG              zgdc
TD                  65536
SOLVENT             CDCl3
NS                   1287
DS                     16
SWH            23148.148 Hz
FIDRES          0.353213 Hz
AQ             1.4156276 sec
RG                 16384
DW               21.600 usec
DE                 6.00 usec
TE                297.2 K
D1            1.00000000 sec
d11           0.03000000 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

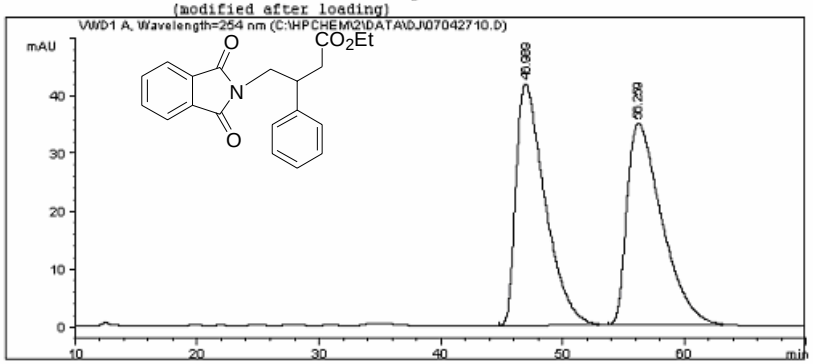
======== CHANNEL f1 ========
NUC1                 13C
P1                  8.20 usec
PL1                 1.50 dB
SFO1         100.6234215 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             80.00 usec
PL2                 5.00 dB
PL12               23.00 dB
SFO2         400.1320007 MHz

F2 - Processing parameters
SI                  32768
SF           100.6127690 MHz
WDW                    EM
SSB                     0
LB                   4.00 Hz
GB                      0
PC                   1.40

1D NMR plot parameters
CX                  20.00 cm
CY                  10.00 cm
F1P                220.000 ppm
F1               22134.81 Hz
F2P                -10.000 ppm
F2               -1006.13 Hz
PPMCM             11.50000 ppm/cm
HZCM           1157.04688 Hz/cm
```

```
==================================================================
Injection Date  : 4/27/2007 11:02:29 PM
Sample Name     : sample                          Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/28/2007 12:12:15 AM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 8:36:38 PM by bayer
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :   1.80400e-1  [mg/ml]   (not used in calc.)

Signal 1: VWD1 A, Wavelength=254 nm

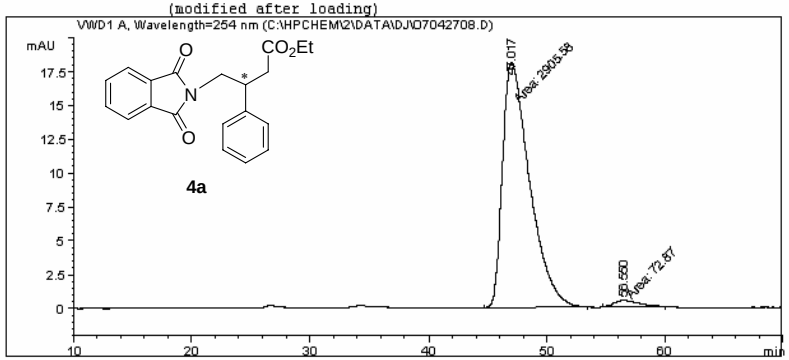
Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  46.989 BB    2.3733 6927.28271   41.79746  49.9369
   2  56.259 BB    2.7509 6944.79639   34.92500  50.0631

Totals :                  1.38721e4    76.72246

 Results obtained with enhanced integrator!
==================================================================
                        Summed Peaks Report
==================================================================

Signal 1: VWD1 A, Wavelength=254 nm
```

```
==================================================================
Injection Date  : 4/27/2007 8:37:36 PM
Sample Name     : sample                          Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/27/2007 8:23:26 PM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 10:28:01 PM by bayer
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :   1.80400e-1  [mg/ml]   (not used in calc.)

Signal 1: VWD1 A, Wavelength=254 nm

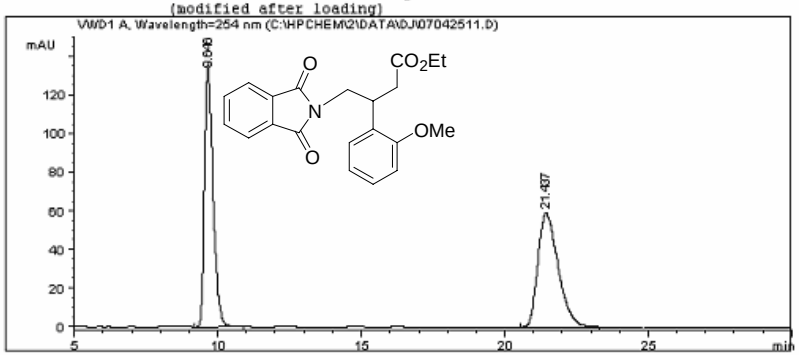
Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  47.017 MM    2.6687 2905.57812   18.14620  97.5534
   2  56.550 MM    2.6206   72.86999 4.63451e-1   2.4466

Totals :                  2978.44811   18.60965

 Results obtained with enhanced integrator!
==================================================================
                        Summed Peaks Report
==================================================================

Signal 1: VWD1 A, Wavelength=254 nm
```

```
==================================================================
Injection Date  : 4/26/2007 9:25:09 AM
Sample Name     : sample                          Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/26/2007 9:33:21 AM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 9:20:05 PM by bayer
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :   1.80400e-1  [mg/ml]   (not used in calc.)

Signal 1: VWD1 A, Wavelength=254 nm

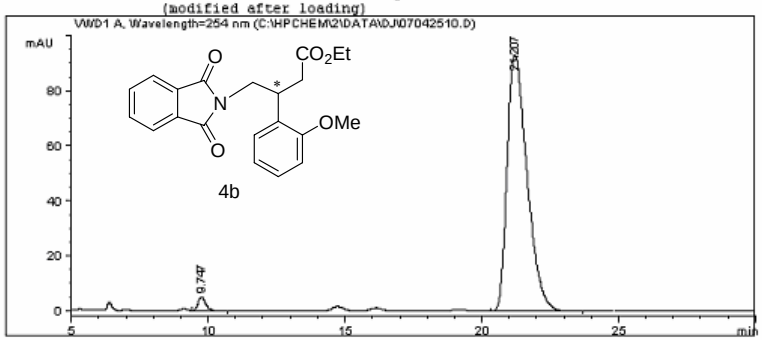
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|-------|
   1   9.646 VB    0.3185 2822.03467  135.59676  49.3745
   2  21.437 BB    0.7589 2893.53101   58.96656  50.6255

Totals :                  5715.56567  194.56332

 Results obtained with enhanced integrator!
==================================================================
                        Summed Peaks Report
==================================================================

Signal 1: VWD1 A, Wavelength=254 nm
```

```
==================================================================
Injection Date  : 4/26/2007 8:48:29 AM
Sample Name     : sample                          Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/26/2007 9:21:29 AM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 10:54:34 PM by bayer
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :   1.80400e-1  [mg/ml]   (not used in calc.)

Signal 1: VWD1 A, Wavelength=254 nm

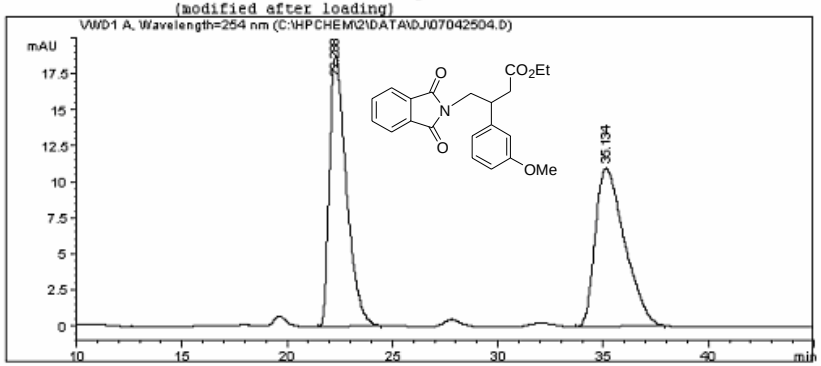
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|-------|
   1   9.747 VB    0.3091   99.15491    4.89565   2.0538
   2  21.207 BB    0.7670 4728.65869   93.14376  97.9462

Totals :                  4827.81360   98.03941

 Results obtained with enhanced integrator!
==================================================================
                        Summed Peaks Report
==================================================================

Signal 1: VWD1 A, Wavelength=254 nm
```

```
==================================================================
Injection Date  : 4/25/2007 4:32:35 PM
Sample Name     : sample                          Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/25/2007 4:16:29 PM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 9:17:54 PM by bayer
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :   1.80400e-1  [mg/ml]   (not used in calc.)

Signal 1: VWD1 A, Wavelength=254 nm

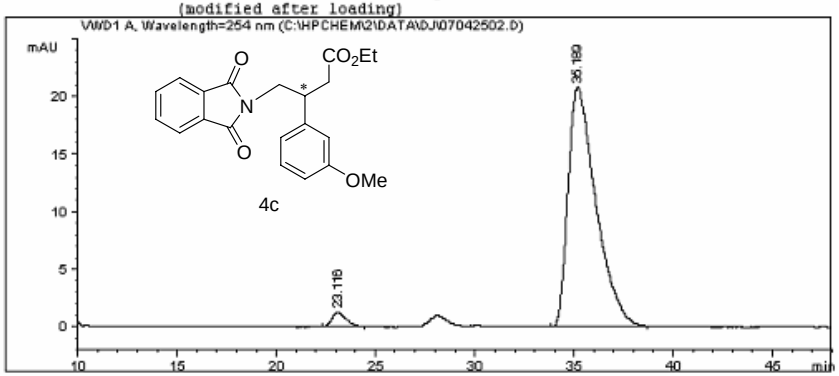
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  22.288 PB    0.8357 1050.99048   18.71658  49.5314
   2  35.134 PB    1.3970 1070.87659   10.96646  50.4686

Totals :                  2121.86707   29.68304

 Results obtained with enhanced integrator!
==================================================================
                        Summed Peaks Report
==================================================================

Signal 1: VWD1 A, Wavelength=254 nm
```

```
==================================================================
Injection Date  : 4/25/2007 2:48:13 PM
Sample Name     : sample                          Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/25/2007 1:18:49 PM by wdy
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 10:56:54 PM by bayer
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :   1.80400e-1  [mg/ml]   (not used in calc.)

Signal 1: VWD1 A, Wavelength=254 nm

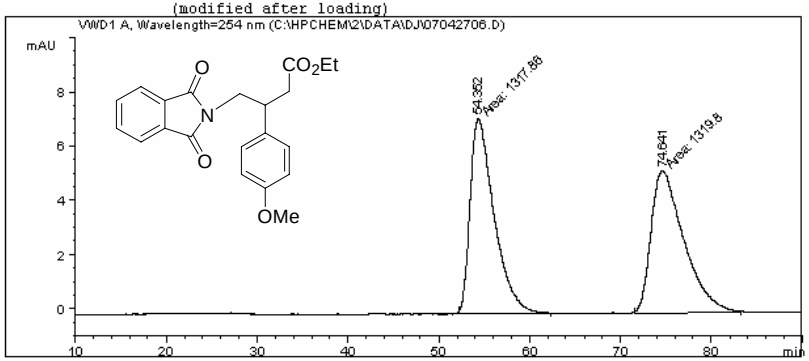
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  23.116 BB    0.7508   60.53914    1.17844   2.8994
   2  35.189 BB    1.4614 2027.42590   20.78243  97.1006

Totals :                  2087.96505   21.96087

 Results obtained with enhanced integrator!
==================================================================
                        Summed Peaks Report
==================================================================

Signal 1: VWD1 A, Wavelength=254 nm
```

```
==================================================================
Injection Date  : 4/27/2007 6:07:53 PM
Sample Name     : sample                          Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/27/2007 1:43:53 PM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 9:16:16 PM by bayer
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :   1.80400e-1  [mg/ml]   (not used in calc.)


Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  54.352 MM    3.0580 1317.85986    7.18251  49.9632
   2  74.641 MM    4.1931 1319.80042    5.24587  50.0368

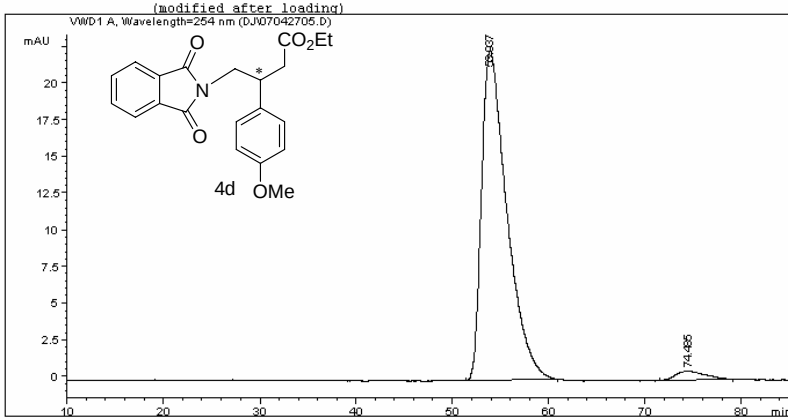
Totals :                  2637.66028   12.42838

 Results obtained with enhanced integrator!
==================================================================
                        Summed Peaks Report
==================================================================

Signal 1: VWD1 A, Wavelength=254 nm
```

```
Data File C:\HPCHEM\2\DATA\DJ\07042705.D                    Sample Name: sample


==================================================================
Injection Date  : 4/27/2007 4:34:30 PM
Sample Name     : sample                          Location : Vial 1
Acq. Operator   : dj
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/27/2007 1:43:53 PM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 5/16/2007 8:04:18 PM by hjd
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000


Signal 1: VWD1 A, Wavelength=254 nm

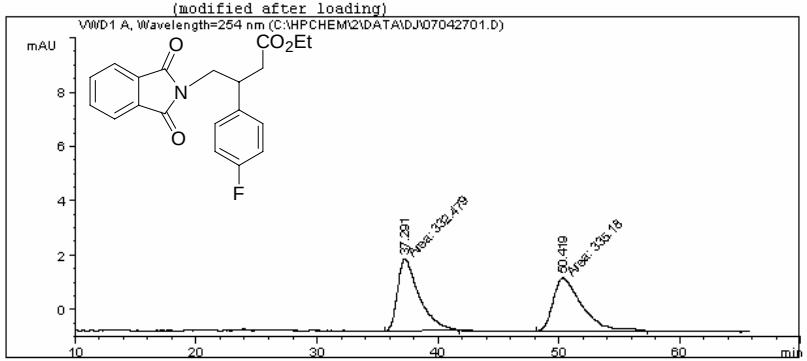
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  53.937 BB    2.5940 4128.97363   22.51291  96.8695
   2  74.485 BB    2.5963  133.43394 6.02974e-1   3.1305

Totals :                  4262.40758   23.11589

 Results obtained with enhanced integrator!
==================================================================
                        *** End of Report ***


Instrument 2 5/16/2007 8:04:28 PM hjd                       Page 1 of 1
```

```
==================================================================
Injection Date  : 4/27/2007 9:58:30 AM
Sample Name     : sample                          Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/27/2007 11:01:31 AM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 8:39:24 PM by bayer
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :   1.80400e-1  [mg/ml]   (not used in calc.)

Signal 1: VWD1 A, Wavelength=254 nm

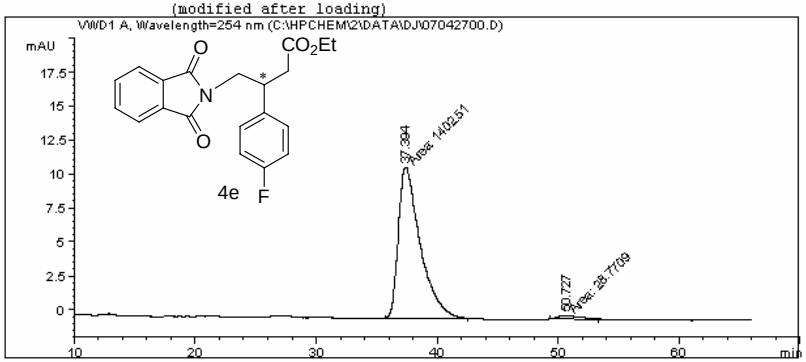
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  37.291 MM    2.0963  332.47919    2.64341  49.7977
   2  50.419 MM    2.8279  335.18005    1.97547  50.2023

Totals :                   667.65924    4.61887

 Results obtained with enhanced integrator!
==================================================================
                        Summed Peaks Report
==================================================================

Signal 1: VWD1 A, Wavelength=254 nm
```

```
==================================================================
Injection Date  : 4/27/2007 8:48:25 AM
Sample Name     : sample                          Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/27/2007 9:42:51 AM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 10:32:57 PM by bayer
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :   1.80400e-1  [mg/ml]   (not used in calc.)

Signal 1: VWD1 A, Wavelength=254 nm

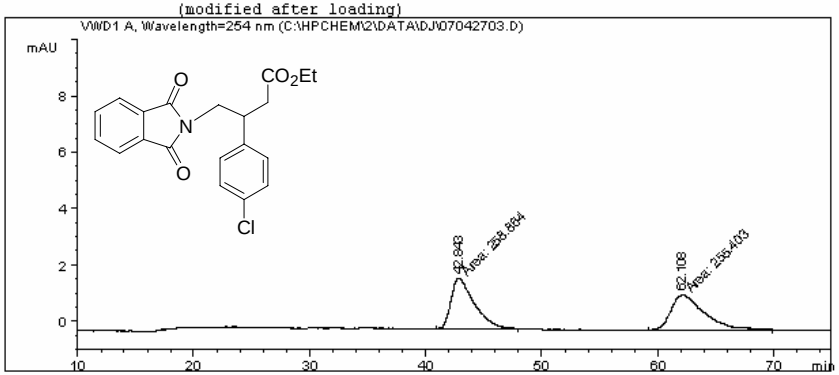
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  37.394 MM    2.1008 1402.50891   11.12665  97.9899
   2  50.727 MM    2.1828   28.77086 2.19679e-1   2.0101

Totals :                  1431.27977   11.34633

 Results obtained with enhanced integrator!
==================================================================
                        Summed Peaks Report
==================================================================

Signal 1: VWD1 A, Wavelength=254 nm
```

```
==================================================================
Injection Date  : 4/27/2007 1:49:23 PM
Sample Name     : sample                          Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/27/2007 1:43:53 PM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 8:41:28 PM by bayer
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :   1.80400e-1  [mg/ml]   (not used in calc.)


Signal 1: VWD1 A, Wavelength=254 nm

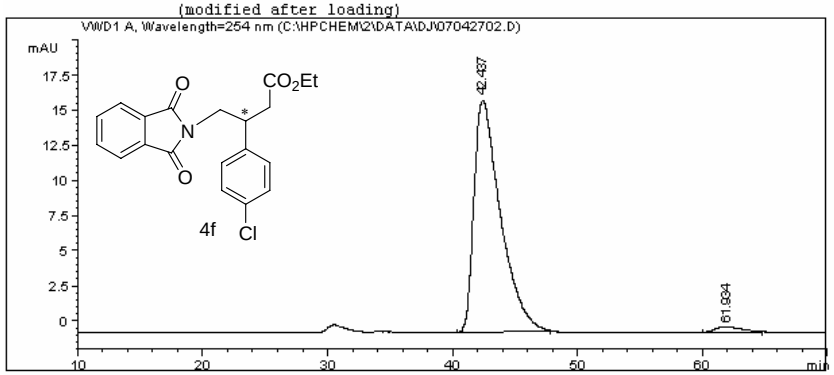
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  42.843 MM    2.3475  258.86438    1.83790  50.3366
   2  62.108 MM    3.4173  255.40279    1.24564  49.6634

Totals :                   514.26717    3.08354

 Results obtained with enhanced integrator!
==================================================================
                        Summed Peaks Report
==================================================================

Signal 1: VWD1 A, Wavelength=254 nm
```

```
==================================================================
Injection Date  : 4/27/2007 11:06:53 AM
Sample Name     : sample                          Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/27/2007 11:01:31 AM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 10:32:57 PM by bayer
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :   1.80400e-1  [mg/ml]   (not used in calc.)


Signal 1: VWD1 A, Wavelength=254 nm

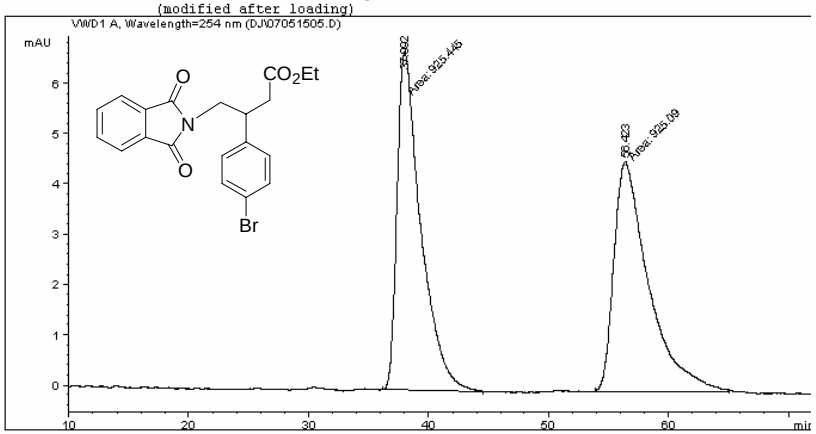
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  42.437 BB    2.1624 2436.85229   16.50326  97.7808
   2  61.934 BB    1.8104   55.30472 3.60449e-1   2.2192

Totals :                  2492.15702   16.86371

 Results obtained with enhanced integrator!
==================================================================
                        Summed Peaks Report
==================================================================

Signal 1: VWD1 A, Wavelength=254 nm
```

```
==================================================================
Injection Date  : 5/16/2007 8:50:19 AM
Sample Name     : samp                          Location : Vial 1
Acq. Operator   : dj
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 5/16/2007 9:59:59 AM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 5/16/2007 7:54:31 PM by hjd
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000

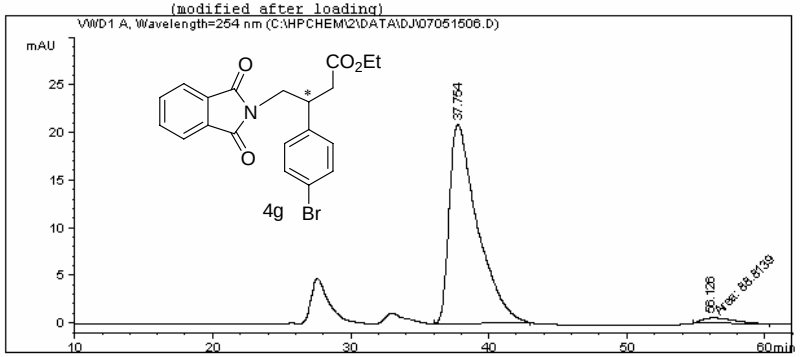
Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  37.992 MM    2.2956  925.44507    6.71894  50.0096
   2  56.423 MM    3.3648  925.08972    4.58220  49.9904

Totals :                  1850.53479   11.30114

 Results obtained with enhanced integrator!
==================================================================
                        *** End of Report ***
```

```
==================================================================
Injection Date  : 5/16/2007 10:02:56 AM
Sample Name     : samp                          Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 5/16/2007 9:59:59 AM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/31/2007 9:35:46 AM by wyh
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :   1.80400e-1  [mg/ml]   (not used in calc.)

Signal 1: VWD1 A, Wavelength=254 nm

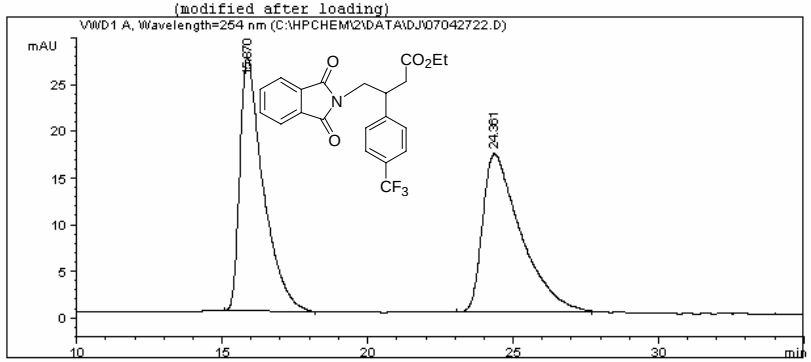
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  37.754 BB    1.9298 2961.13745   20.89705  97.0880
   2  56.126 MM    2.5572   88.81391 5.78845e-1   2.9120

Totals :                  3049.95136   21.47590

 Results obtained with enhanced integrator!
==================================================================
                        Summed Peaks Report
==================================================================

Signal 1: VWD1 A, Wavelength=254 nm
```

```
==================================================================
Injection Date  : 4/29/2007 10:21:53 AM
Sample Name     : sample                          Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/29/2007 9:02:04 AM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 9:59:27 PM by bayer
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :   1.80400e-1  [mg/ml]   (not used in calc.)


Signal 1: VWD1 A, Wavelength=254 nm

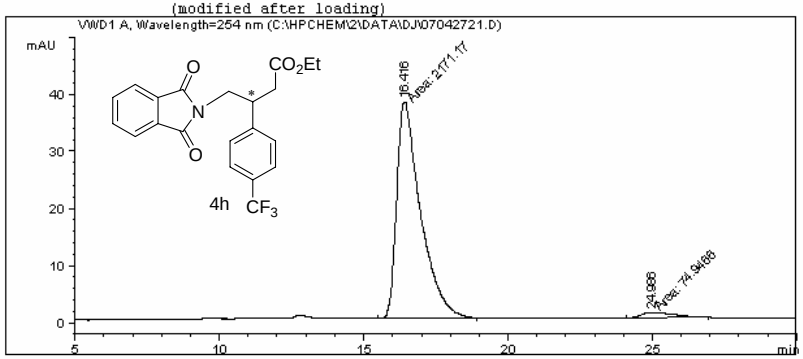
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  15.870 PB    0.8070 1515.77112   27.15453  49.5519
   2  24.361 PB    1.2815 1543.18640   16.93499  50.4481

Totals :                  3058.95752   44.08952

 Results obtained with enhanced integrator!
==================================================================
                        Summed Peaks Report
==================================================================

Signal 1: VWD1 A, Wavelength=254 nm
```

```
==================================================================
Injection Date  : 4/29/2007 9:41:42 AM
Sample Name     : sample                          Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/29/2007 9:02:04 AM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 11:16:56 PM by bayer
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :   1.80400e-1  [mg/ml]   (not used in calc.)


Signal 1: VWD1 A, Wavelength=254 nm

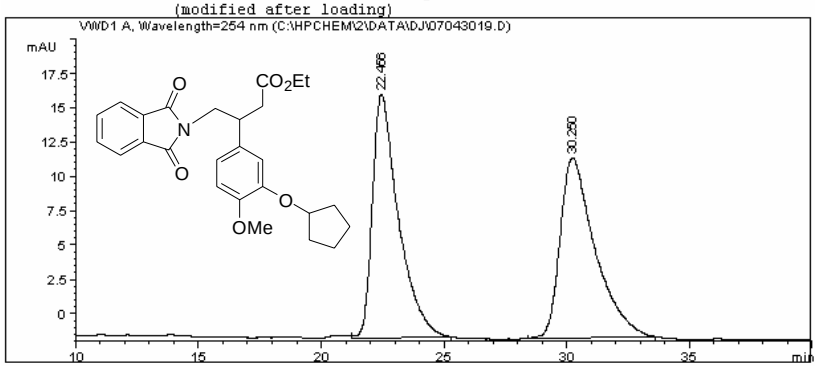
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  16.416 MM    0.9525 2171.16895   37.99152  96.6633
   2  24.986 MM    1.3222   74.94664 9.44756e-1   3.3367

Totals :                  2246.11559   38.93627

 Results obtained with enhanced integrator!
==================================================================
                        Summed Peaks Report
==================================================================

Signal 1: VWD1 A, Wavelength=254 nm
```

```
==================================================================
Injection Date  : 5/1/2007 3:47:27 PM
Sample Name     : sample                          Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 5/1/2007 4:23:34 PM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 10:02:56 PM by bayer
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :   1.80400e-1  [mg/ml]   (not used in calc.)

Signal 1: VWD1 A, Wavelength=254 nm

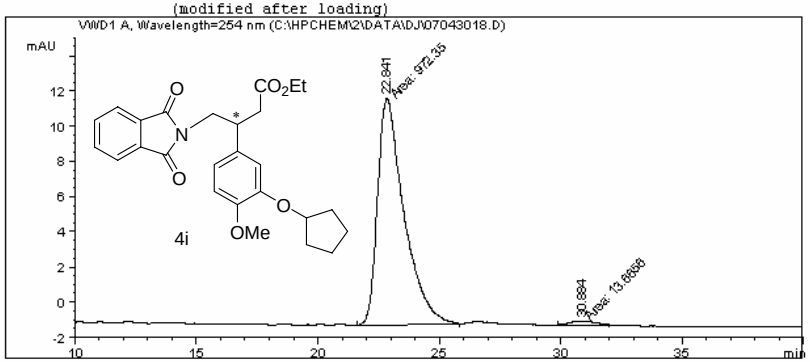
Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  22.456 VB    1.0568 1312.91663   17.68629  50.1832
   2  30.250 BB    1.3612 1303.33203   13.13865  49.8168

Totals :                  2616.24866   30.82494

 Results obtained with enhanced integrator!
==================================================================
                        Summed Peaks Report
==================================================================

Signal 1: VWD1 A, Wavelength=254 nm
```

```
==================================================================
Injection Date  : 5/1/2007 2:55:29 PM
Sample Name     : sample                          Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 5/1/2007 2:42:33 PM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 11:11:58 PM by bayer
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :   1.80400e-1  [mg/ml]   (not used in calc.)

Signal 1: VWD1 A, Wavelength=254 nm

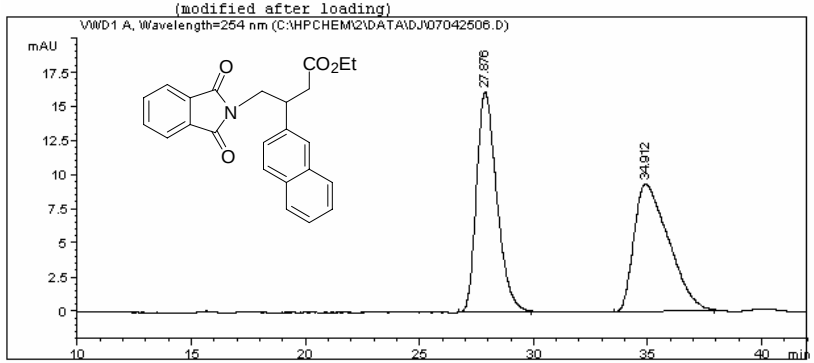
Peak RetTime Type  Width     Area      Height     Area
  #   [min]       [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  22.841 MM    1.2616  972.34998   12.84551  98.6141
   2  30.884 MM    1.2443   13.66558 1.83038e-1   1.3859

Totals :                   986.01555   13.02855

 Results obtained with enhanced integrator!
==================================================================
                        Summed Peaks Report
==================================================================

Signal 1: VWD1 A, Wavelength=254 nm
```

```
==================================================================
Injection Date  : 4/25/2007 6:29:02 PM
Sample Name     : sample                          Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/25/2007 7:13:25 PM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 9:45:34 PM by bayer
                  (modified after loading)
```




```
==================================================================
                      Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :   1.80400e-1  [mg/ml]   (not used in calc.)

Signal 1: VWD1 A, Wavelength=254 nm

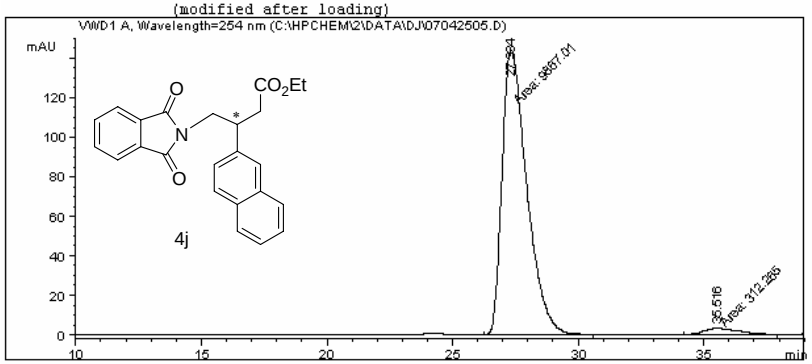
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  27.876 BB    0.9404  991.31299   16.08426  50.2068
   2  34.912 BB    1.4725  983.14655    9.32990  49.7932

Totals :                  1974.45953   25.41416

 Results obtained with enhanced integrator!
==================================================================
                      Summed Peaks Report
==================================================================

Signal 1: VWD1 A, Wavelength=254 nm
```

```
==================================================================
Injection Date  : 4/25/2007 5:21:00 PM
Sample Name     : sample                          Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/25/2007 4:16:29 PM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 11:03:38 PM by bayer
                  (modified after loading)
```




```
==================================================================
                      Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :   1.80400e-1  [mg/ml]   (not used in calc.)

Signal 1: VWD1 A, Wavelength=254 nm

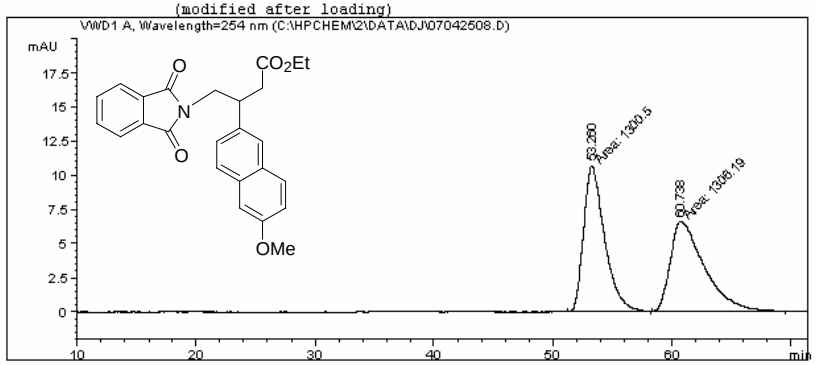
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  27.334 MM    1.1399 9867.00684  144.26749  96.9323
   2  35.516 MM    1.7268  312.26526    3.01387   3.0677

Totals :                  1.01793e4   147.28136

 Results obtained with enhanced integrator!
==================================================================
                      Summed Peaks Report
==================================================================

Signal 1: VWD1 A, Wavelength=254 nm
```

```
==================================================================
Injection Date  : 4/25/2007 8:42:28 PM
Sample Name     : sample                          Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/25/2007 9:53:22 PM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 9:47:11 PM by bayer
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :   1.80400e-1  [mg/ml]   (not used in calc.)


Signal 1: VWD1 A, Wavelength=254 nm

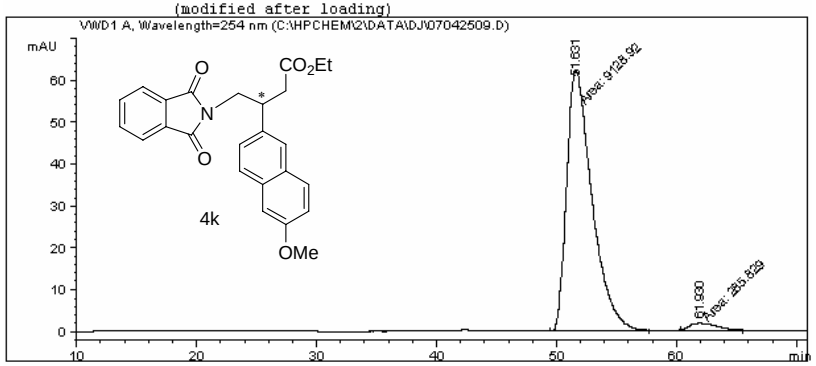
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  53.260 MM    2.0304 1300.49597   10.67517  49.8907
   2  60.738 MM    3.3159 1306.19397    6.56522  50.1093

Totals :                  2606.68994   17.24039

 Results obtained with enhanced integrator!
==================================================================
                        Summed Peaks Report
==================================================================

Signal 1: VWD1 A, Wavelength=254 nm
```

```
==================================================================
Injection Date  : 4/25/2007 10:06:38 PM
Sample Name     : sample                          Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/25/2007 9:53:22 PM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 11:07:06 PM by bayer
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :   1.80400e-1  [mg/ml]   (not used in calc.)


Signal 1: VWD1 A, Wavelength=254 nm

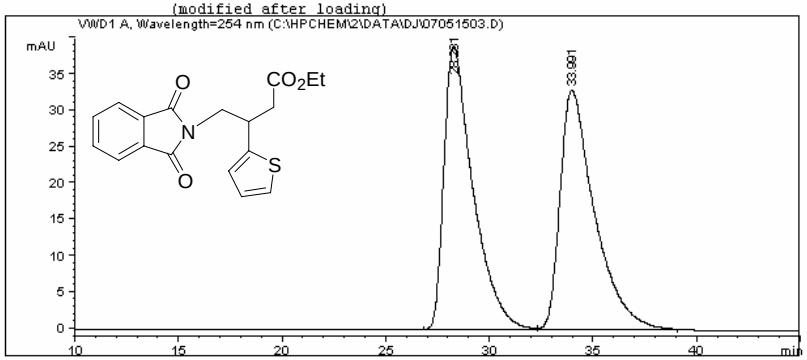
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  51.631 MM    2.4550 9128.91602   61.97523  96.9640
   2  61.930 MM    2.6937  285.82910    1.76848   3.0360

Totals :                  9414.74512   63.74371

 Results obtained with enhanced integrator!
==================================================================
                        Summed Peaks Report
==================================================================

Signal 1: VWD1 A, Wavelength=254 nm
```

```
==================================================================
Injection Date  : 5/15/2007 10:26:58 PM
Sample Name     : samp                          Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 5/15/2007 11:20:20 PM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 11:34:08 PM by bayer
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :   1.80400e-1  [mg/ml]   (not used in calc.)

Signal 1: VWD1 A, Wavelength=254 nm

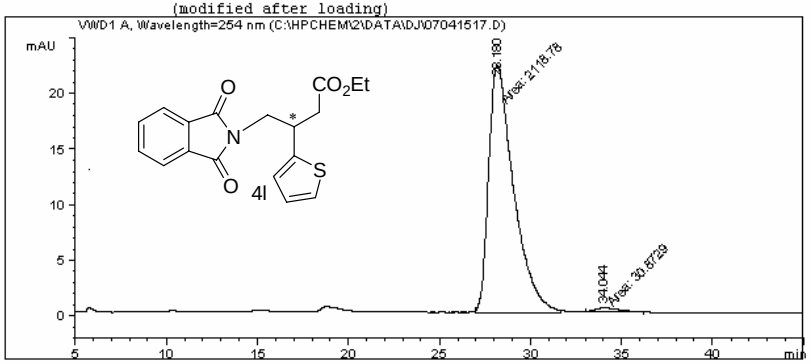
Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  28.281 BB    1.4209 3817.57275   39.02330  50.0238
   2  33.991 BB    1.6710 3813.93945   32.94393  49.9762

Totals :                   7631.51221   71.96723

 Results obtained with enhanced integrator!
==================================================================
                        Summed Peaks Report
==================================================================

Signal 1: VWD1 A, Wavelength=254 nm
```

```
==================================================================
Injection Date  : 4/16/2007 7:40:20 PM
Sample Name     :                               Location : Vial 1
Acq. Operator   : dj
Acq. Instrument : Instrument 2
Acq. Method     : C:\HPCHEM\2\METHODS\DZHCH.M
Last changed    : 4/16/2007 8:27:38 PM by dj
                  (modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\KCM-1D.M
Last changed    : 5/15/2007 11:29:20 PM by bayer
                  (modified after loading)
```




```
==================================================================
                        Area Percent Report
==================================================================

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Sample Amount         :   1.80400e-1  [mg/ml]   (not used in calc.)

Signal 1: VWD1 A, Wavelength=254 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|-------|----|-------|----------|----------|--------|
   1  28.180 MM    1.5850 2118.77832   22.27925  98.5638
   2  34.044 MM    1.6199   30.87293 3.17643e-1   1.4362

Totals :                   2149.65125   22.59690

 Results obtained with enhanced integrator!
==================================================================
                        Summed Peaks Report
==================================================================

Signal 1: VWD1 A, Wavelength=254 nm
```