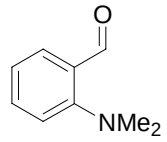
Supporting Information

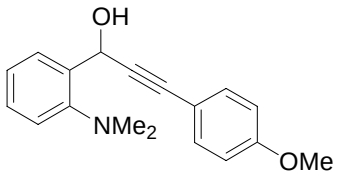
Selective *Endo*- and *Exo*-iodocyclisation in the Synthesis of Nitrogen Heterocycles.

*Karl O. Hessian and Bernard L. Flynn**



2-Dimethylamino benzaldehyde (9)

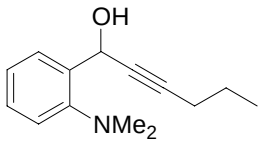
Prepared by the literature procedure of P. Damhaut *Tetrahedron.* **1997**, *53*, 5785.



1-(2-Dimethylaminophenyl)-3-(4-methoxyphenyl)prop-2-yn-1-ol (10a)

To a stirred solution of β,β-dibromo-4-methoxybenzene (848 mg, 2.92 mmol) in THF (20 mL) at -78 °C (dry-ice / acetone bath) was added dropwise a solution of *n*-butyllithium (2.97 mL in hexanes, 5.94 mmol), and the solution left to stir for 0.25 h. After this time, the solution was warmed to room temperature, and then recooled to -78 °C. A solution of **9** (429 mg, 2.88 mmol) in THF (5 mL) was added, and then warmed to room temperature. After stirring for a further 0.15 h, the solution was concentrated under reduced pressure to remove the greater part of solvent. The residue was quenched with $NH_4Cl_{(aq)}$ (50 mL, sat.), and extracted with diethyl ether (3 x 50 mL), dried over $MgSO_4$, filtered and concentrated to give the product as a pale yellow oil (810 mg, 100 %).

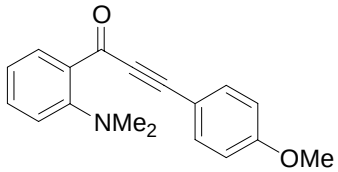
^{1}H-NMR ($CDCl_3$) δ 7.45 (d, *J* = 7.8 Hz, 1H), 7.36 (d, *J* = 8.4 Hz, 2H), 7.31 – 7.33 (m, 2H), 7.15 – 7.21 (m, 1H), 6.83 (d, *J* = 8.4 Hz, 2H), 5.84 (s, 1H), 3.78 (s, 3H), 2.80 (s, 6H). ^{13}C-NMR ($CDCl_3$) δ 159.5 (C), 151.4 (C), 135.8 (C), 132.9 (C-H), 128.7 (C-H), 128.1 (C-H), 125.5 (C-H), 121.7 (C-H), 114.9 (C), 113.7 (C-H), 88.5 (C), 86.1 (C), 64.9 (C-OH), 55.2 (CH_3), 45.6 (CH_3). LRMS *m/z*: 281.0 (94). HRMS *m/z*: M^+ calculated for $C_{18}H_{19}NO_2$: 281.1416. Found: 281.1414.



1-(2-Dimethylaminophenyl)hex-2-yn-ol (10b)

To a stirred solution of pentyne (520 mg, 7.65 mmol) in THF (15 mL) at 0 °C (ice bath) was added dropwise a solution of *n*-butyllithium (4.00 mL in hexanes, 6.96 mmol), and the solution left to stir for 0.25 h. After this time, a solution of **9** (1.00 g, 6.71 mmol) in THF (5 mL) was added, and the ice bath removed. After stirring for a further 0.15 h, the solution was concentrated under reduced pressure to remove the greater part of solvent. The residue was quenched with $NH_4Cl_{(aq)}$ (50 mL, sat.), and extracted with diethyl ether (3 x 50 mL), dried over $MgSO_4$, filtered and concentrated to give the product as a pale yellow oil (1.45 g, 100 %).

^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.39 (d, *J* = 7.2 Hz, 1H), 7.23 – 7.26 (m, 2H), 7.09 – 7.14 (m, 1H), 6.92 (bs, 1H), 5.65 (t, *J* = 1.8 Hz, 1H), 2.73 (s, 6H), 2.20 (dt, *J* = 1.2, 7.2 Hz, 2H), 1.52 (sextet, *J* = 7.2 Hz, 2H), 0.95 (t, *J* = 7.2 Hz, 3H). ^{13}C-NMR ($CDCl_3$) δ 151.3 (C), 136.3 (C), 128.5 (C-H), 128.0 (C-H), 125.1 (C-H), 121.1 (C-H), 86.7 (C), 81.2 (C), 63.6 (C-OH), 45.4 (CH_3), 21.9 (CH_2), 20.8 (CH_2), 13.4 (CH_3). LRESMS *m/z*: $C_{14}H_{20}NO^+$ = 218.2 (100).

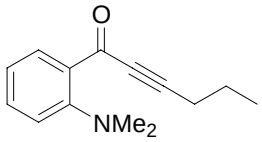


1-(2-Dimethylaminophenyl)-3-(4-methoxyphenyl)prop-2-yn-1-one (11a)

To a stirred solution of **10a** (709 mg, 2.52 mmol) in CH_2Cl_2 (80 mL), was added commercially available MnO_2 (6.96 g, 75 % activated) portionwise over 0.15 h. After stirring for a further 0.25 h, the solution was filtered through celite (4 cm) with CH_2Cl_2 (250 mL), and the yellow filtrate solution concentrated under reduced

pressure (without heating above 25 °C) onto silica gel (6 g). The residue was subjected to flash chromatography (silica gel, eluent: ethyl acetate, DCM, hexanes 1:2:7, then 2:2:6) to give the product as an orange resin (632 mg, 90 %).

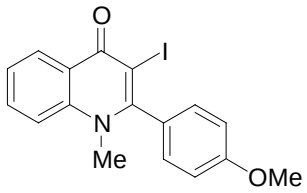
^{1}H-NMR ($CDCl_3$) δ 8.12 (dd, *J* = 1.2, 7.8 Hz, 1H), 7.59 (d, *J* = 8.7 Hz, 2H), 7.42 (dt, *J* = 1.2, 8.4 Hz, 1H), 7.02 (d, *J* = 8.1 Hz, 1H), 6.87 – 6.96 (m, 3H), 3.85 (s, 3H), 2.96 (s, 6H). ^{13}C-NMR ($CDCl_3$) δ 177.8 (C), 161.3 (C), 152.5 (C), 134.8 (C-H), 133.6 (C-H), 133.4 (C-H), 126.6 (C), 117.8 (C-H), 116.4 (C-H), 114.3 (C-H), 112.5 (C), 91.4 (C), 88.5 (C), 55.3 (CH_3), 43.9 (CH_3). LRESMS *m/z*: $C_{18}H_{18}NO_2^+$ = 280.2 (100). HRESMS *m/z*: M^+ calculated for $C_{18}H_{18}NO_2^+$: 280.1332. Found = 280.1329.



1-(2-Dimethylaminophenyl)hex-2-yn-one (11b)

To a stirred solution of **10b** (1.45 g, 6.68 mmol) in CH_2Cl_2 (100 mL), was added commercially available MnO_2 (14.5 g, 75 % activated) portionwise over 0.1 h. After stirring for a further 0.25 h, the solution was filtered through celite (4 cm) with CH_2Cl_2 (400 mL), and the yellow filtrate concentrated under reduced pressure onto silica gel (10 g). The residue was subjected to flash chromatography (silica gel, eluent: ethyl acetate, hexanes 1:19, then 1:9) to give the desired product as a yellow oil (1.38 g, 96 %).

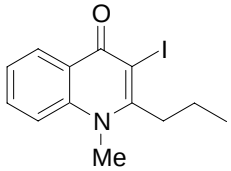
^{1}H-NMR (300 MHz, $CDCl_3$) δ 8.08 (d, *J* = 7.5 Hz, 1H), 7.41 (t, *J* = 8.4 Hz, 1H), 7.05 (m, 1H), 6.90 (t, *J* = 7.2 Hz), 2.93 (s, 6H), 2.44 (t, *J* = 6.9 Hz, 2H), 1.68 (sextet, *J* = 7.2 Hz, 2H), 1.07 (t, *J* = 7.2 Hz, 3H). ^{13}C-NMR ($CDCl_3$) δ 177.8 (C), 152.4 (C), 133.8 (C-H), 133.4 (C-H), 126.3 (C), 117.7 (C-H), 116.3 (C-H), 93.7 (C), 81.4 (C), 43.8 (CH_3), 21.3 (CH_2), 21.0 (CH_2), 13.5 (CH_3). LRESMS *m/z*: $C_{14}H_{18}NO^+$ = 216.1 (100). HRESMS *m/z*: M^+ calculated for $C_{14}H_{18}NO^+$: 216.1383. Found: 216.1379.



3-Iodo-2-(4-methoxyphenyl)-1-methylquinolin-4-one (13a)

To a stirred solution of **11a** (100 mg, 0.358 mmol) in CH_2Cl_2 (6 mL) was added a solution of iodine (91 mg, 0.358 mmol) in CH_2Cl_2 (4 mL) and stirred for 0.5 h. After this time, the solution was quenched with $Na_2S_2O_{5(aq)}$ (15 ml, 10 % sol.), extracted with CH_2Cl_2 (3 x 20 mL), dried over Na_2SO_4, and concentrated under reduced pressure onto silica gel. The residue was subjected to flash chromatography (silica gel, eluent: ethyl acetate, DCM, hexanes 2:1:7, then 4:1:5) to give the product as a yellow solid (131 mg, 94 %), mp = 179-181 °C.

^{1}H-NMR ($CDCl_3$) δ 8.55 (dd, *J* = 1.5, 8.1 Hz, 1H), 7.72 (dt, *J* = 1.5, 7.5 Hz, 1H), 7.54 (d, *J* = 8.7 Hz, 1H), 7.44 (t, *J* = 7.5 Hz, 1H), 7.21 (d, *J* = 8.7 Hz, 2H), 7.08 (d, *J* = 8.7 Hz, 2H), 3.91 (s, 3H), 3.61 (s, 3H). ^{13}C-NMR ($CDCl_3$) δ 174.0 (C), 160.3 (C), 155.3 (C), 140.7 (C), 132.5 (C-H), 131.7 (C), 129.7 (C-H), 127.6 (C-H), 124.3 (C-H), 122.8 (C), 115.7 (C-H), 114.4 (C-H), 89.8 (C-I), 55.4 (CH_3), 39.2 (CH_3). LRESMS *m/z*: $C_{17}H_{15}NO_2I^+$ = 392.1 (100). HRESMS *m/z*: M^+ calculated for $C_{17}H_{15}NO_2I^+$ = 392.0142. Found: 392.0137.

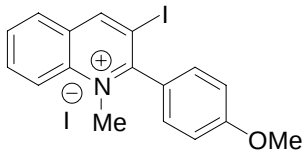


3-Iodo-1-methyl-2-propylquinolin-4-one (13b)

Iodine (175 mg, 0.689 mmol) was added to a solution of **11b** (145 mg, 0.674 mmol) in CH_2Cl_2 (10 mL), protected from light and left to stir for 1 h. After this time, the

solution was quenched with a solution of $Na_2S_2O_{5(aq)}$ (20 mL, sat %), and extracted with CH_2Cl_2 (2 x 10 mL). The combined organic layers were dried over Na_2SO_4, and concentrated under reduced pressure onto silica gel (2 g). The residue was quickly subjected to flash chromatography (silica gel, eluent diethyl ether : hexanes, 1 : 9, then 1 : 4) to give the desired product as an orange solid (190 mg, 86 %).

^{1}H-NMR (300 MHz, CD_3CN) δ 7.50 – 7.55 (m, 2H), 6.98 (d, *J* = 8.4 Hz, 1H), 6.88 (dt, *J* = 0.6, 6.9 Hz, 1H), 3.39 (s, 3H), 3.06 (d, *J* = 7.8 Hz, 2H), 1.69 (septet, *J* = 7.5 Hz, 2H), 0.97 (t, *J* = 9.0 Hz, 3H). ^{13}C-NMR (CD_3CN) δ 184.2 (C), 153.5 (C), 137.5 (C), 136.4 (C-H), 124.0 (C-H), 120.6 (C), 119.8 (C-H), 109.7 (C-H), 91.7 (C-I), 45.2 (CH_2), 35.8 (CH_3), 23.9 (CH_2), 12.6 (CH_3). LRESMS *m/z*: $C_{13}H_{15}NOI^+$ = 328.1 (100). HRESMS *m/z*: M^+ calculated for $C_{13}H_{15}NOI^+$: 328.0193. Found: 328.0191.

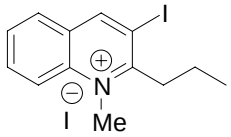


3-Iodo-2-(4-methoxyphenyl)-1-methylquinolinium iodide (15a)

A solution of iodine (86 mg, 0.339 mmol) in CH_3CN (3 mL) was added to a stirred solution of **10a** (95 mg, 0.338 mmol) in CH_3CN (2 mL), and stirred for 0.5 h. After this time, the solution was heated to 65 °C and stirred for 2 h. After this time, the solution was cooled to room temperature, and concentrated under reduced pressure to give a brown residue. The residue was recrystallised from DMF to give the product as red needles (151 mg, 89 %), mp = 136-138 °C.

^{1}H-NMR (D^6-DMSO) δ 9.89 (s, 1H), 8.51 (d, *J* = 7.7 Hz, 1H), 8.40 (d, *J* = 7.1 Hz, 1H), 8.30 (t, *J* = 7.1 Hz, 1H), 8.06 (t, *J* = 7.7 Hz, 1H), 7.51 (d, *J* = 7.5 Hz, 2H), 7.26 (d, *J* = 7.5 Hz, 2H), 4.29 (s, 3H), 3.91 (s, 3H). ^{13}C-NMR (D^6-DMSO) δ 161.4 (C), 161.3 (C), 155.2 (C-H), 139.2 (C), 136.3 (C-H), 130.5 (C-H), 130.4 (C-H), 129.8 (C), 129.6 (C-H), 129.1 (C), 120.3 (C-H), 115.1 (C-H), 97.0 (C-I), 56.0 (CH_3), 45.2

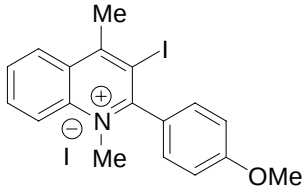
(CH_3). LRESMS *m/z*: $C_{17}H_{15}INO^+$ = 376.0 (12, M^+), 362 (100, M^+ - CH_2). HRESMS *m/z*: M^+ calculated for $C_{17}H_{15}INO^+$: 376.0193. Found: 376.0191.



3-Iodo-1-methyl-2-propylquinolinium iodide (15b)

Iodine (286 mg, 1.126 mmol) was added to a stirred solution of **10b** (122 mg, 0.562 mmol) in CH_3CN (3 mL) at room temperature and left to stir. After 0.5 h, the solution was heated to reflux and stirred for a further 10 h. After this time, the solution was cooled to room temperature and concentrated under reduced pressure. The residue was crystallised from ethanol to give the desired product as a yellow solid (175 mg, 71%), mp = 115-116 °C.

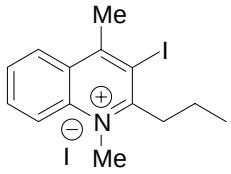
^{1}H-NMR (CD_3CN) δ 7.72 (s, 1H), 7.48 (d, *J* = 8.7 Hz, 1H), 7.38 – 7.41 (m, 2H), 7.15 (dt, *J* = 0.9, 6.9 Hz, 1H), 4.04 (s, 3H), 2.95 (t, *J* = 6.6 Hz, 2H), 1.74 (sextet, *J* = 6.7 Hz, 2H), 1.00 (t, *J* = 6.6 Hz, 3H). ^{13}C-NMR (CD_3CN) δ 151.6 (C-H), 139.2 (C), 130.5 (C-H), 130.4 (C-H), 129.8 (C), 129.6 (C-H), 129.1 (C), 120.3 (C-H), 102.3 (C-I), 47.5 (CH_3), 41.8 (CH_2), 19.8 (CH_2), 11.2 (CH_3). LRESMS *m/z*: $C_{13}H_{15}IN^+$ = 312.0 (100). HRESMS *m/z*: M^+ calculated for $C_{13}H_{15}$ IN^+: 312.0244. Found: 312.0241.



1,4-Dimethyl-3-iodo-2-(4-methoxyphenyl)quinolinium iodide (15c)

To a stirred solution of **11a** (55 mg, 0.197 mmol) in THF (4 mL) at -78 °C (dry-ice / acetone bath) was added methyl magnesium chloride (1.5 eq, sol. in THF) dropwise. After stirring for 0.15 h, the solution was warmed to room temperature, quenched with $NH_4Cl_{(aq)}$ (5 mL, 10% in H_2O), extracted with diethyl ether (2 x 10 mL), washed with water (10 mL), dried over Na_2SO_4, and concentrated under reduced pressure. The resultant oil was diluted with CH_3CN (2 mL) and a solution of iodine (50 mg, 0.197 mmol) in CH_3CN (3 mL) was added. The solution was heated to 65 °C and stirred for 18 h. After this time, the solution was cooled to room temperature, and concentrated under reduced pressure to give a brown residue. The residue was recrystallised from DMF to give the product as red needles (81 mg, 80 %), mp = 152-154 °C.

^{1}H-NMR (D^6-DMSO) δ 8.40 (d, *J* = 7.8 Hz, 1H), 8.31 (d, *J* = 7.2 Hz, 1H), 8.16 (t, *J* = 7.2 Hz, 1H), 7.96 (t, *J* = 7.8 Hz, 1H), 7.49 (d, *J* = 7.5 Hz, 2H), 7.20 (d, *J* = 7.5 Hz, 2H), 4.45 (s, 3H), 3.96 (s, 3H), 2.92 (s, 3H). ^{13}C-NMR (D^6-DMSO) δ 161.2 (C), 161.1 (C), 146.8 (C), 137.6 (C), 135.9 (C-H), 131.0 (C-H), 130.5 (C-H), 129.8 (C), 129.6 (C-H), 129.3 (C), 122.8 (C-H), 114.9 (C-H), 94.5 (C-I), 55.8 (CH_3), 45.0 (CH_3), 14.3 (CH_3). LRESMS *m/z*: $C_{17}H_{15}INO^+$ = 390 (100). HRESMS *m/z*: M^+ calculated for $C_{17}H_{15}INO^+$: 390.0349. Found: 390.0352.

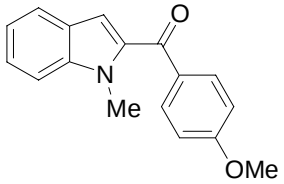


1,4-Dimethyl-3-iodo-2-propylquinolinium iodide (15d)

To a stirred solution of **11b** (70 mg, 0.326 mmol) in THF (4 mL) at -78 °C (dry-ice / acetone bath) was added methyl magnesium chloride (0.325 mL, 0.488 mmol) dropwise. After stirring for 0.15 h, the solution was warmed to room temperature, quenched with $NH_4Cl_{(aq)}$ (5 mL, 10% in H_2O), extracted with diethyl ether (2 x 10 mL), washed with water (10 mL), dried over Na_2SO_4, and concentrated under reduced

pressure. The resultant oil was diluted with CH_3CN (3 mL) and a solution of iodine (165 mg, 0.652 mmol) in CH_3CN (3 mL) was added at room temperature and left to stir. After 0.5 h, the solution was heated to reflux and stirred for a further 16 h. After this time, the solution was cooled to room temperature and concentrated under reduced pressure. The residue was crystallised from ethanol to give the desired product as a dark red solid (109 mg, 74%), mp = 120-122 °C.

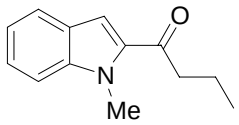
^{1}H-NMR (CD_3CN) δ 7.67 (d, *J* = 7.6 Hz, 1H), 7.41 – 7.53 (m, 2H), 7.03 (dt, *J* = 1.0, 7.4 Hz, 1H), 4.19 (s, 3H), 3.01 (s, 3H), 2.82 (t, *J* = 6.7 Hz, 2H), 1.72 (sextet, *J* = 6.7 Hz, 2H), 1.01 (t, *J* = 6.6 Hz, 3H). ^{13}C-NMR (CD_3CN) δ 148.7 (C), 138.9 (C), 130.6 (C-H), 130.4 (C-H), 129.3 (C), 129.2 (C-H), 128.9 (C), 120.3 (C-H), 104.5 (C-I), 48.2 (CH_3), 41.7 (CH_2), 19.4 (CH_2), 19.5 (CH_3), 11.2 (CH_3). LRESMS *m/z*: $C_{14}H_{17}IN^+$ = 326.0 (100). HRESMS *m/z*: M^+ calculated for $C_{14}H_{17}IN^+$: 326.0406. Found: 326.0402.



2-(4-Methoxybenzoyl)-1-methylindole (17a)

A solution of iodine (109, 0.429 mmol) in ethanol (3 mL) was added to a stirred solution of **10a** (120 mg, 0.427 mmol) in ethanol (3 mL), and stirred for 0.5 h. After this time, the solution was heated to 65 °C and stirred for 6 h. After this time, the solution was cooled to room temperature, diluted with $Na_2S_2O_{5(aq)}$ (10 mL, sat.), extracted with ethyl acetate (2 x 20 mL), dried over $MgSO_4$, and concentrated under reduced pressure onto silica gel (2 g). The residue was subjected to flash chromatography (silica gel, eluent: ethyl acetate, hexanes 1:99, 1:19) to give the product as a white solid (88 mg, 78 %), mp = 82-84 °C.

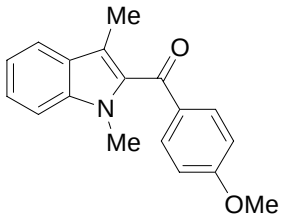
^{1}H-NMR ($CDCl_3$) δ 7.98 (d, *J* = 8.7 Hz, 2H), 7.70 (d, *J* = 8.0 Hz, 1H), 7.45 (d, *J* = 8.0 Hz, 1H), 7.41 (dt, *J* = 1.5, 6.5 Hz, 1H), 7.18 (dt, *J* = 1.5, 6.5 Hz, 1H), 7.00 (d, *J* = 8.7 Hz, 2H), 7.00 (s, 1H), 4.09 (s, 3H), 3.91 (s, 3H). ^{13}C-NMR ($CDCl_3$) δ 187.2 (C), 163.1 (C), 140.0 (C), 135.2 (C), 132.1 (C-H), 131.9 (C), 125.8 (C), 125.5 (C-H), 122.7 (C-H), 120.6 (C-H), 113.5 (C-H), 113.4 (C-H), 110.2 (C-H), 55.5 (CH_3), 31.8 (CH_3). LRESMS *m/z*: 265.1 (100). HRESMS *m/z*: M^+ calculated for $C_{17}H_{16}NO_2^+$: 266.1176. Found: 266.1176.



2-(1-Butanoyl)-1-methylindole (17b)

Iodine (176 mg, 0.693 mmol) in ethanol (5 mL) was added dropwise to a stirred solution of **10b** (150 mg, 0.691 mmol) in ethanol (5 mL, 95%) and the solution was then refluxed for 6 h, cooled and concentrated to remove the ethanol. The resultant crude was diluted with ethyl acetate (10 mL), quenched with $Na_2S_2O_{5(aq)}$ (15 ml, 10 % sol.), dried over Na_2SO_4, and concentrated under reduced pressure onto silica gel (2 g). The residue was subjected to flash chromatography (silica gel, eluent: diethyl ether, hexanes 1:19, then 1:9) to give the desired product as a cream solid (94 mg, 68 %), mp = 78-80 °C.

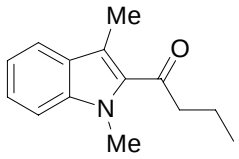
^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.70 (d, *J* = 8.1 Hz, 1H), 7.38 – 7.40 (m, 2H), 7.30 (s, 1H), 7.14 – 7.19 (m, 2H), 4.09 (s, 3H), 2.96 (t, *J* = 7.2 Hz, 2H), 1.82 (sextet, *J* = 7.5 Hz, 2H), 1.04 (t, *J* = 7.5 Hz, 3H). ^{13}C-NMR ($CDCl_3$) δ 194.5 (C), 140.0 (C), 134.9 (C), 125.8 (C), 125.7 (C-H), 122.8 (C-H), 120.6 (C-H), 111.0 (C-H), 110.3 (C-H), 41.8 (CH_2), 32.1 (CH_3), 18.6 (CH_2), 13.9 (CH_3). LRESMS *m/z*: $C_{13}H_{16}NO^+$ = 202.0 (40). HRESMS *m/z*: M^+ calculated for $C_{13}H_{16}NO^+$: 202.1226. Found: 202.1226.



1,3-Dimethyl-2-(4-methoxybenzoyl)indole (17c)

To a stirred solution of **11a** (50 mg, 0.179 mmol) in THF (4 mL) at -78 °C (dry-ice / acetone bath) was added methyl magnesium chloride (1.5 eq, sol. in THF) dropwise. After stirring for 0.15 h, the solution was warmed to room temperature, quenched with $NH_4Cl_{(aq)}$ (5 mL, 10% in H_2O), extracted with diethyl ether (2 x 10 mL), washed with water (10 mL), dried over Na_2SO_4, and concentrated under reduced pressure. The resultant oil was diluted with ethanol (5 mL, 95%) and a solution of iodine (45 mg, 0.179 mmol) in ethanol (3 mL) was added. The solution was then refluxed for 18 h, cooled and concentrated to remove the ethanol. The resultant crude was diluted with ethyl acetate (10 mL), quenched with $Na_2S_2O_{5(aq)}$ (10 ml, 10 % sol.), dried over Na_2SO_4, and concentrated under reduced pressure onto silica gel (1 g). The residue was subjected to flash chromatography (silica gel, eluent: ethyl acetate, hexanes 1:49, 1:19, then 1:9) to give the desired product as a yellow solid (36 mg, 72 %), mp = 88 - 90 °C.

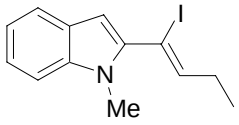
^{1}H-NMR ($CDCl_3$) δ 7.87 (d, *J* = 8.7 Hz, 2H), 7.65 (d, *J* = 7.8 Hz, 1H), 7.35 – 7.41 (m, 2H), 7.17 (t, *J* = 7.8, 1H), 6.98 (d, *J* = 8.7 Hz, 2H), 3.91 (s, 3H), 3.82 (s, 3H), 2.18 (s, 3H). ^{13}C-NMR ($CDCl_3$) δ 188.9 (C), 163.5 (C), 138.7 (C), 134.0 (C), 132.4 (C), 132.2 (C-H), 127.5 (C), 124.8 (C-H), 120.5 (C-H), 119.7 (C-H), 117.1 (C), 113.8 (C-H), 109.8 (C-H), 55.4 (CH_3), 31.4 (CH_3), 10.8 (CH_3). LRESMS *m/z*: $C_{18}H_{18}NO_2^+$ = 280.1 (100). HRESMS *m/z*: M^+ calculated for $C_{18}H_{18}NO_2^+$: 280.1332. Found: 280.1332.



2-(1-Butanoyl)-1,3-dimethylindole (17d)

To a stirred solution of **11b** (125 mg, 0.582 mmol) in THF (5 mL) at -78 °C (dry-ice / acetone) bath was added a solution of methyl magnesium bromide (0.30 mL in THF, 0.900 mmol). After 0.25 h, the solution was warmed to room temperature and quenched with $NH_4Cl_{(aq)}$ (10 mL, 10 % in H_2O), extracted with diethyl ether (3 x 10 mL), dried over $MgSO_4$ and concentrated under reduced pressure. The residue was taken up in ethanol (5 mL), and a solution of iodine (148 mg, 0.582 mmol) in ethanol (3 mL) was added dropwise. The solution was heated to reflux and left to stir for 6 h. After this time, the solution was cooled to room temperature and concentrated under reduced pressure to remove the greater part of solvent. The residue was quenched with $Na_2S_2O_{5(aq)}$ (10 mL, sat.) and extracted with diethyl ether (3 x 10 mL), dried over $MgSO_4$ and concentrated under reduced pressure onto silica gel (2 g). The residue was subjected to flash chromatography (silica gel, eluent) to give the desired product as a white solid (115 mg, 92 %), mp = 86-88 °C.

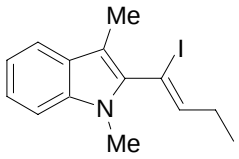
^{1}H-NMR (300 MHz, $CDCl_3$) δ 7.70 (d, *J* = 8.1 Hz, 1H), 7.33 – 7.42 (m, 2H), 7.16 (t, *J* = 1.5, 7.8 Hz, 1H), 3.97 (s, 3H), 2.94 (t, *J* = 7.2 Hz, 2H), 2.65 (s, 3H), 1.84 (sextet, *J* = 7.5 Hz, 2H), 1.06 (t, *J* = 7.5 Hz, 3H). ^{13}C-NMR ($CDCl_3$) δ 195.6 (C), 138.8 (C), 133.8 (C), 127.2 (C), 125.6 (C-H), 120.7 (C-H), 119.7 (C-H), 118.1 (C), 110.0 (C-H), 45.0 (CH_2), 32.5 (CH_3), 17.9 (CH_2), 13.9 (CH_3), 11.7 (CH_3). LRESMS *m/z*: $C_{14}H_{18}NO^+$ = 216.1 (100). HRESMS *m/z*: M^+ calculated for $C_{14}H_{18}NO^+$: 216.1383. Found: 216.1381.



2-[(*Z*)-1-Iodobut-1-enyl]-1-methylindole (24a)

n-Butyllithium (1.30 mL, 2.00 mmol) was added dropwise to a solution of pentyne (160 mg, 2.35 mmol) in THF (5 mL) at 0 °C (ice-bath) and stirred for 0.15 h. After this time a solution of **9** (260 mg, 1.75 mmol) in THF (2 mL) was added and the solution warmed to room temperature. After stirring for 0.15 h, acetic anhydride (250 mg, 2.45 mmol) was added and the solution left to stir for a further 0.5 h. After this time the solution was quenched with $NH_4Cl_{(aq)}$ (6 mL, 10 %) and extracted with diethyl ether (3 x 10 mL), dried over $MgSO_4$, and concentrated under reduced pressure to give a yellow resin. This residue was diluted with ethanol (15 mL), and a solution of iodine (443 mg, 1.75 mmol) in ethanol (6 mL) was added dropwise. After stirring for 0.1 h, the ethanol was removed under reduced pressure, taking care not to heat above 25 °C. The resultant brown residue was dried on a freeze-dryer pump for 48 h to remove any water, and then diluted with anhydrous dichloroethane (4 mL). The solution was heated to 65 °C and stirred for 2 h, then cooled to room temperature, quenched with $Na_2S_2O_{5(aq)}$ (10 mL, 10 %), extracted with CH_2Cl_2 (2 x 10 mL), dried over $MgSO_4$ and concentrated under reduced pressure onto silica gel (3 g). The residue was subjected to flash chromatography (silica gel, eluent: diethyl ether, hexanes 1:99, then 1:19) to give the desired product as an off-white solid (326 mg, 60 %), mp = 101-102 °C (this product underwent some *cis/trans*-isomerisation in $CDCl_3$).

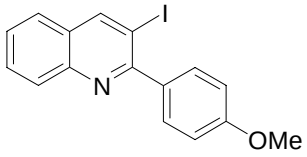
^{1}H-NMR (CD_3CN) δ 7.54 (d, *J* = 7.8 Hz, 1H), 7.37 (d, *J* = 7.8 Hz, 1H), 7.23 (dt, *J* = 1.1, 7.8 Hz, 1H), 7.09 (dt, *J* = 1.1, 7.8 Hz, 1H), 6.44 (s, 1H), 5.99 (t, *J* = 6.9 Hz, 1H), 2.35 (m, *J* = 7.5 Hz, 2H), 1.13 (t, *J* = 7.5, 3H). ^{13}C-NMR (CD_3CN) δ 145.7 (C-H), 141.9 (C), 137.6 (C), 126.9 (C), 122.3 (C-H), 120.6 (C-H), 119.8 (C-H), 109.7 (C-H), 101.9 (C-H), 89.7 (C-I), 30.4 (CH_3), 30.3 (CH_2), 11.8 (CH_3). LRESMS *m/z*: $C_{13}H_{15}NI^+$ = 312.1 (100). HRESMS *m/z*: M^+ calculated for $C_{13}H_{15}NI^+$: 312.0244. Found: 312.0241.



1,3-Dimethyl-2-[(*Z*)-1-Iodobut-1-enyl]indole (24b)

To a stirred solution of **11b** (54 mg, 0.251 mmol) in THF (5 mL) at -78 °C (dry-ice / acetone) bath was added a solution of methyl magnesium bromide (0.14 mL in THF, 0.420 mmol). After stirring for 0.15 h, acetic anhydride (85 mg, 0.83 mmol) was added and the solution left to stir for a further 0.5 h. After this time the solution was quenched with $NH_4Cl_{(aq)}$ (5 mL, 10 %) and extracted with diethyl ether (3 x 10 mL), dried over $MgSO_4$, and concentrated under reduced pressure to give a pale yellow resin. This residue was diluted with ethanol (3 mL), and a solution of iodine (64 mg, 0.252 mmol) in ethanol (2 mL) was added dropwise. After stirring for 0.1 h, the ethanol was removed under reduced pressure, taking care not to heat above 25 °C. The resultant brown residue was dried on a freeze-dryer pump for 36 h to remove any water, and then diluted with anhydrous dichloroethane (4 mL). The solution was heated to 65 °C and stirred for 1 h, then cooled to room temperature, quenched with $Na_2S_2O_{5(aq)}$ (5 mL, 10 %), extracted with CH_2Cl_2 (2 x 10 mL), dried over $MgSO_4$ and concentrated under reduced pressure onto silica gel (1 g). The residue was subjected to flash chromatography (silica gel, eluent: diethyl ether, hexanes 1:99, then 1:19) to give the desired product as a white solid (51 mg, 63 %), mp = 108-110 °C.

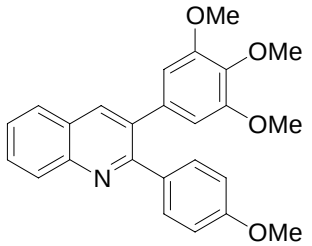
^{1}H-NMR (CD_3CN) δ 7.65 (d, *J* = 7.7 Hz, 1H), 7.41 (d, *J* = 7.7 Hz, 1H), 7.19 (dt, *J* = 1.1, 7.8 Hz, 1H), 7.04 (dt, *J* = 1.1, 7.8 Hz, 1H), 6.12 (t, *J* = 6.7 Hz, 1H), 3.56 (s, 3H), 2.40 (s, 3H), 2.33 (m, *J* = 7.5 Hz, 2H), 1.12 (t, *J* = 7.5, 3H). ^{13}C-NMR (CD_3CN) δ 141.2 (C), 138.6 (C), 136.9 (C), 126.8 (C), 122.3 (C-H), 119.1 (C-H), 116.5 (C-H), 110.0 (C-H), 101.9 (C-H), 89.8 (C-I), 31.2 (CH_3), 30.2 (CH_2), 12.9 (CH_3), 11.6 (CH_3). LRESMS *m/z*: $C_{14}H_{17}NI^+$ = 326.0 (100). HRESMS *m/z*: M^+ calculated for $C_{13}H_{15}NI^+$: 326.0400. Found: 326.0397.



3-Iodo-2-(4-methoxyphenyl)quinoline

Triphenylphosphine (86 mg, 0.328 mmol) and **15a** (93 mg, 0.185 mmol) were diluted in sulfolane (1.26 mL), and heated to 150 ºC for 0.5 h, and then recooled to room temperature. After this time, the solution was quenched with water (5 mL) and extracted with ethyl acetate (2 x 5 mL), dried over $MgSO_4$, and concentrated under reduced pressure onto silica gel. The residue was subjected to a small silica gel plug, washed with eluent (5 % diethyl ether in hexanes, then 20 % diethyl ether in hexanes) to substantially remove the triphenylphosphine and sulfolane, and the collected solution was concentrated under reduced pressure onto silica gel. The residue was subjected to flash chromatography (silica gel, eluent ethyl acetate : hexanes, 1 : 4) to give the desired product as a yellow solid (51 mg, 77 %), mp = 108-110 °C.

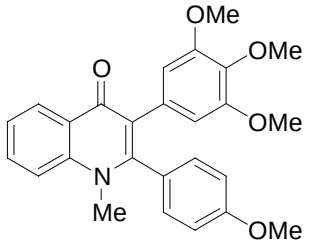
^{1}H-NMR ($CDCl_3$) δ 8.90 (s, 1H), 8.39 (d, *J* = 8.1 Hz, 1H), 7.78 – 7.83 (m, 2H), 7.70 (d, *J* = 8.7 Hz, 2H), 7.61 (dt, *J* = 1.5, 8.4 Hz, 1H), 7.04 (d, *J* = 8.1 Hz, 2H), 3.90 (s, 3H). ^{13}C-NMR ($CDCl_3$) δ 160.2 (C), 160.0 (C), 147.1 (C-H), 146.9 (C), 134.6 (C), 130.8 (C-H), 130.1 (C-H), 129.3 (C-H), 128.3 (C), 127.0 (C-H), 126.2 (C-H), 113.3 (C-H), 91.4 (C-I), 55.3 (CH_3). LRESMS *m/z*: $C_{16}H_{13}NIO^+$ = 362.1 (100). HRESMS *m/z*: M^+ calculated for $C_{16}H_{13}INO^+$ = 362.0036. Found = 362.0032.



2-(4-Methoxyphenyl)-3-(3,4,5-trimethoxyphenyl)quinoline (25)

A solution of Na_2CO_3 (205 mg, 1.93 mmol) in H_2O (1 mL) was added to a solution of 3-Iodo-2-(4-methoxyphenyl)quinoline (80 mg, 0.222 mmol), 3,4,5-trimethoxybenzene boronic acid (142 mg, 0.670 mmol), $Pd(dba)_2$ (3.3 mg, 5.7 μmol), and triphenylphosphine (7.0 mg, 26.7 μmol) in DMF (4 mL). The solution was degassed by exposure to vacuum and backfilling with $N_{2(g)}$ (3 cycles) to remove dissolved oxygen. The solution was then heated to 120 °C, and left to stir for 10 h. After cooling to room temperature the reaction mixture was diluted with HCl (0.05 M in H_2O, 20 mL), and extracted with ethyl acetate (3 x 30 mL), dried over Na_2SO_4, and concentrated onto silica gel (4 g). The residue was subjected to flash chromatography (silica gel, eluent: ethyl acetate, hexanes 1:9, 1:4, then 1:1) to give the product as a yellow solid (72 mg, 81 %), mp = 138-142 °C.

^{1}H-NMR ($CDCl_3$) δ 8.16 – 8.19 (m, 2H), 7.85 (dd, *J* = 1.2, 7.5 Hz, 1H), 7.72 (dt, *J* = 1.2, 7.5 Hz, 1H), 7.55 (dt, *J* = 1.2, 7.5 Hz, 1H), 7.42 (d, *J* = 8.7 Hz, 2H), 6.84 (d, *J* = 8.7 Hz, 2H), 6.47 (s, 2H), 3.88 (s, 3H), 3.81 (s, 3H), 3.70 (s, 6H). ^{13}C-NMR ($CDCl_3$) δ 159.6 (C), 157.9 (C), 152.9 (C), 147.3 (C), 136.8 (C-H), 135.4 (C), 134.2 (C), 133.0 (C), 131.1 (C-H), 129.4 (C-H), 129.3 (C-H), 127.3 (C-H), 126.9 (C), 126.5 (C-H), 113.4 (C-H), 107.2 (C-H), 60.9 (CH_3), 56.2, (CH_3), 56.0 (CH_3). LRESMS *m/z*: $C_{25}H_{24}NO_4^+$ = 402.3 (100). HRESMS *m/z*: M^+ calculated for $C_{25}H_{24}NO_4^+$: 402.1700. Found: 402.1695.

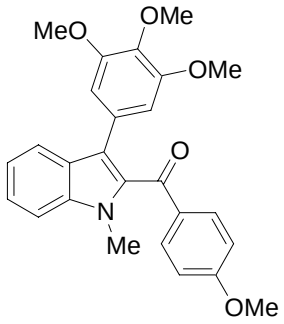


2-(4-Methoxyphenyl)-1-methyl-3-(3,4,5-trimethoxyphenyl)quinolin-4-one (26)

A solution of Na_2CO_3 (213 mg, 2.01 mmol) in H_2O (1 mL) was added to a solution of **KH-3-69** (168 mg, 0.448 mmol), 3,4,5-trimethoxybenzene boronic acid (142 mg, 0.670 mmol), DBA (7.7 mg, 13.4 μmol), and triphenylphosphine (14.1 mg, 53.8

μmol) in DMF (4 mL). The solution was degassed by exposure to vacuum and backfilling with $N_{2(g)}$ (3 cycles) to remove dissolved oxygen. The solution was then heated to 110 °C, and left to stir for 8 h. After cooling to room temperature the reaction mixture was diluted with HCl (0.05 M in H_2O, 20 mL), and extracted with ethyl acetate (3 x 30 mL), dried over Na_2SO_4, and concentrated onto silica gel (3 g). The residue was subjected to flash chromatography (silica gel, eluent: ethyl acetate, DCM, hexanes 3:1:4, 3:1:2, then 3:1:0) to give the product as a yellow solid (160 mg, 83 %), mp = 192-194 °C.

^{1}H-NMR (300 MHz, $CDCl_3$) δ 8.57 (dd, *J* = 1.5, 7.8 Hz, 1H), 7.73 (dt, *J* = 1.5, 7.5 Hz, 1H), 7.57 (d, *J* = 8.4 Hz, 1H), 7.42 (t, *J* = 7.5 Hz, 1H), 7.08 (d, *J* = 8.7 Hz, 2H), 6.81 (d, *J* = 8.7 Hz, 2H), 6.27 (s, 2H), 3.77 (s, 3H), 3.76 (s, 3H), 3.65 (s, 6H), 3.58 (s, 3H). ^{13}C-NMR ($CDCl_3$) δ 175.9 (C), 159.7 (C), 152.2 (C), 141.3 (C), 136.2 (C), 132.2 (C-H), 131.3 (C), 130.8 (C-H), 127.2 (C), 127.1 (C-H), 126.4 (C), 124.1 (C), 123.5 (C-H), 115.8 (C-H), 113.7 (C-H), 109.0 (C-H), 60.6 (CH_3), 55.8 (CH_3), 55.2 (CH_3), 37.7 (CH_3). LRESMS *m/z*: $C_{26}H_{26}NO_5^+$ = 432.3 (100). HRESMS *m/z*: M^+ calculated for $C_{26}H_{26}NO_5^+$: 432.1806. Found: 432.1803.



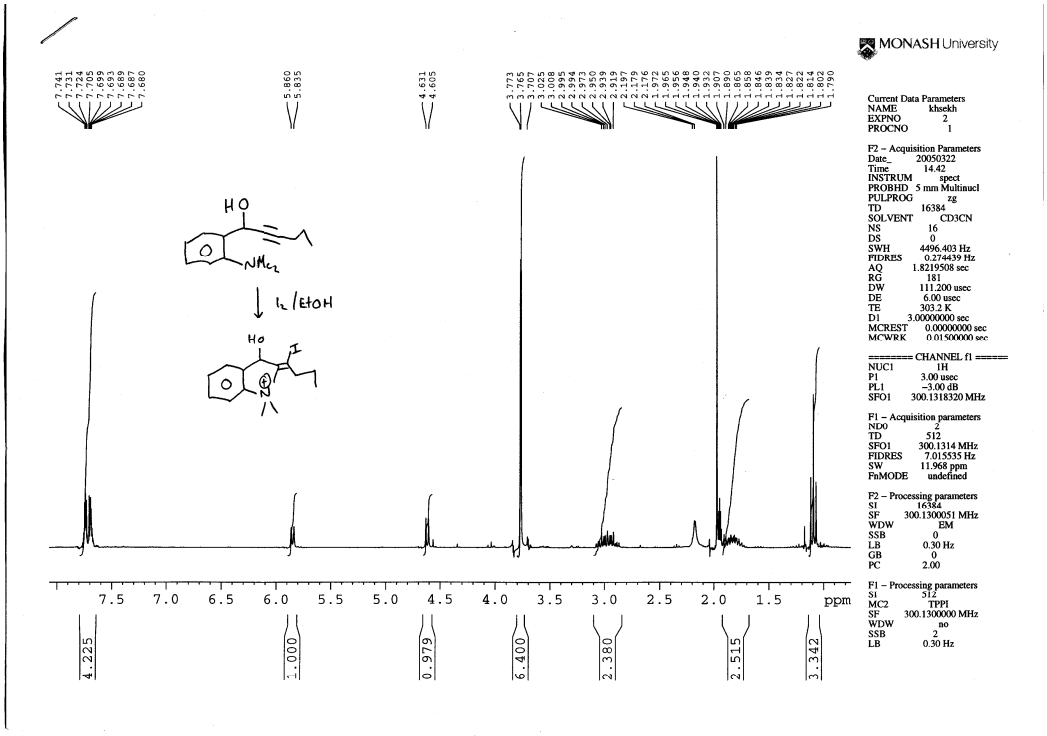
2-(4-Methoxybenzoyl)-1-methyl-3-(3,4,5-trimethoxyphenyl)indole (27)

t-Butyllithium (0.65 mL, 0.824 mmol) was added dropwise to a solution of 3,4,5-trimethoxy iodobenzene (117 mg, 0.400 mmol) in THF (3 mL) at -78 °C (dry-ice / acetone bath) and stirred for 0.25 h. After this time, the solution was warmed to room temperature, and then recooled to -78 °C. A solution of **11a** (85 mg, 0.305 mmol) in THF (2 mL) was added dropwise, and the solution left to stir for a further 0.1 h, and

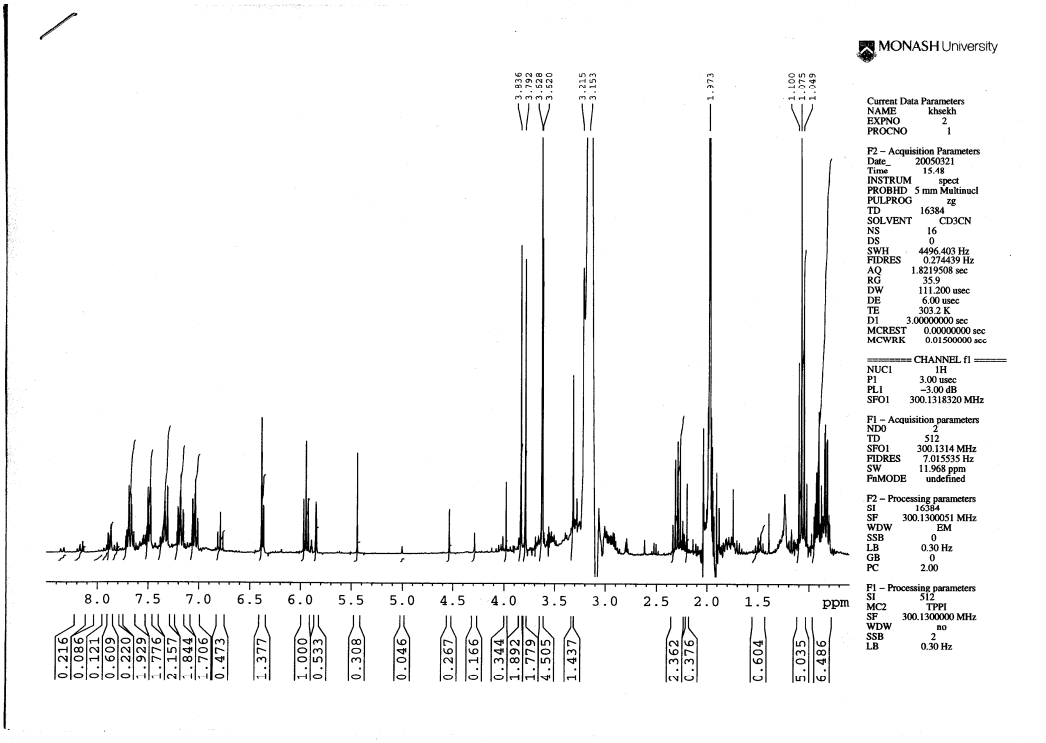
then warmed to room temperature. The solution was then quenched with $NH_4Cl_{(aq)}$ (5 mL, sat.), extracted with ethyl acetate (2 x 10 mL), dried over Na_2SO_4, and concentrated under reduced pressure to give an orange residue. The residue was diluted with ethanol (8 mL, 95 %), and a solution of iodine (78 mg, 0.307 mmol) in ethanol (2 mL) was added dropwise. After stirring at room temperature for 1 h, water (1 mL) was added and the solution was heated to 65 °C and left to stir. After 10 h, the solution was cooled to room temperature and concentrated under reduced pressure to remove the greater part of solvent. The residue was quenched with $Na_2S_2O_{5(aq)}$ (10 mL, sat.), extracted with ethyl acetate (2 x 10 mL), dried over Na_2SO_4, and concentrated under reduced pressure onto silica gel (2 g). The residue was subjected to flash chromatography (silica gel, eluent ethyl acetate, dichloromethane, hexanes 1:2:17, 1:1:8, then 2:1:7) to give the product as a yellow solid (84 mg, 63 %), mp = 146-148 °C.

^{1}H-NMR ($CDCl_3$) δ 7.84 (d, *J* = 8.1 Hz, 1H), 7.71 (d, *J* = 8.7 Hz, 2H), 7.40 – 7.49 (m, 2H), 7.24 (dt, *J* = 1.5, 6.6 Hz, 1H), 6.67 (d, *J* = 8.7 Hz, 2H), 6.53 (s, 2H), 3.92 (s, 3H), 3.77 (s, 6H), 3.73 (s, 6H). ^{13}C-NMR ($CDCl_3$) δ 187.5 (C), 165.2 (C), 159.4 (C), 156.8 (C), 146.8 (C), 136.2 (C), 134.4 (C), 132.1 (C), 131.6 (C-H), 131.3 (C-H), 127.0 (C), 125.1 (C-H), 120.4 (C-H), 119.7 (C-H), 117.0 (C), 112.2 (C-H), 109.7 (C-H), 60.5 (CH_3), 56.1 (CH_3), 55.4 (CH_3), 31.4 (CH_3). LRESMS *m/z*: $C_{26}H_{26}NO_5^+$ = 432.2 (100). HRESMS *m/z*: M^+ calculated for $C_{26}H_{26}NO_5^+$: 432.1805. Found: 432.1807.

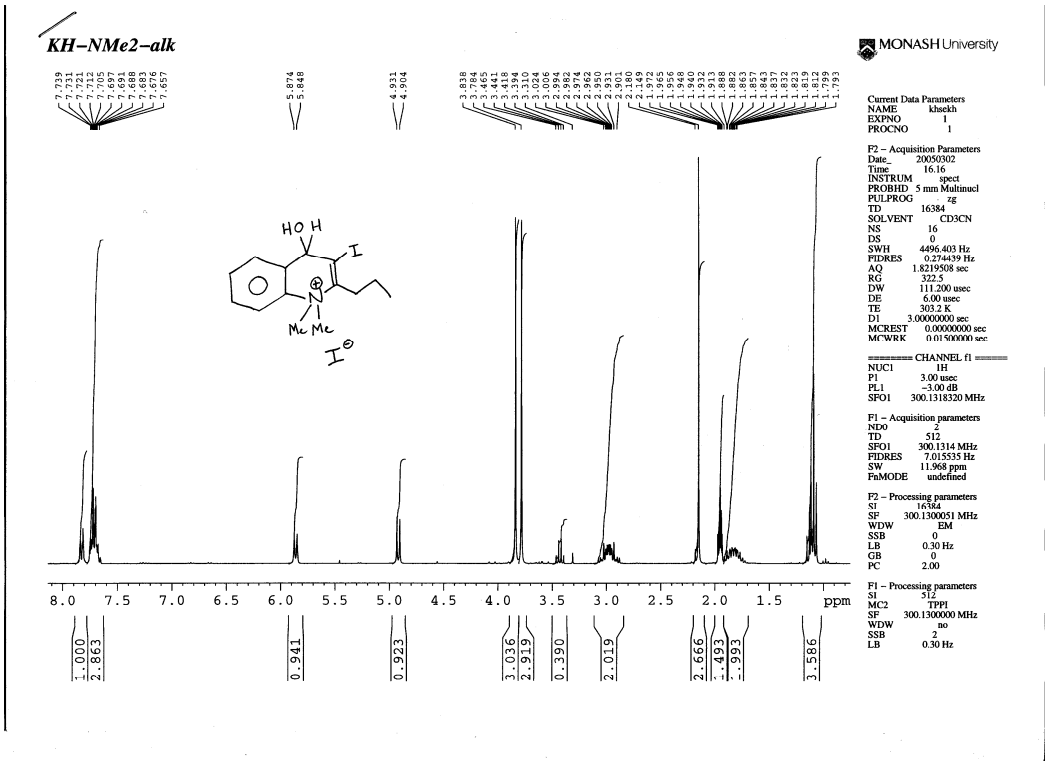
Cyclisation in ethanol: Initial Product



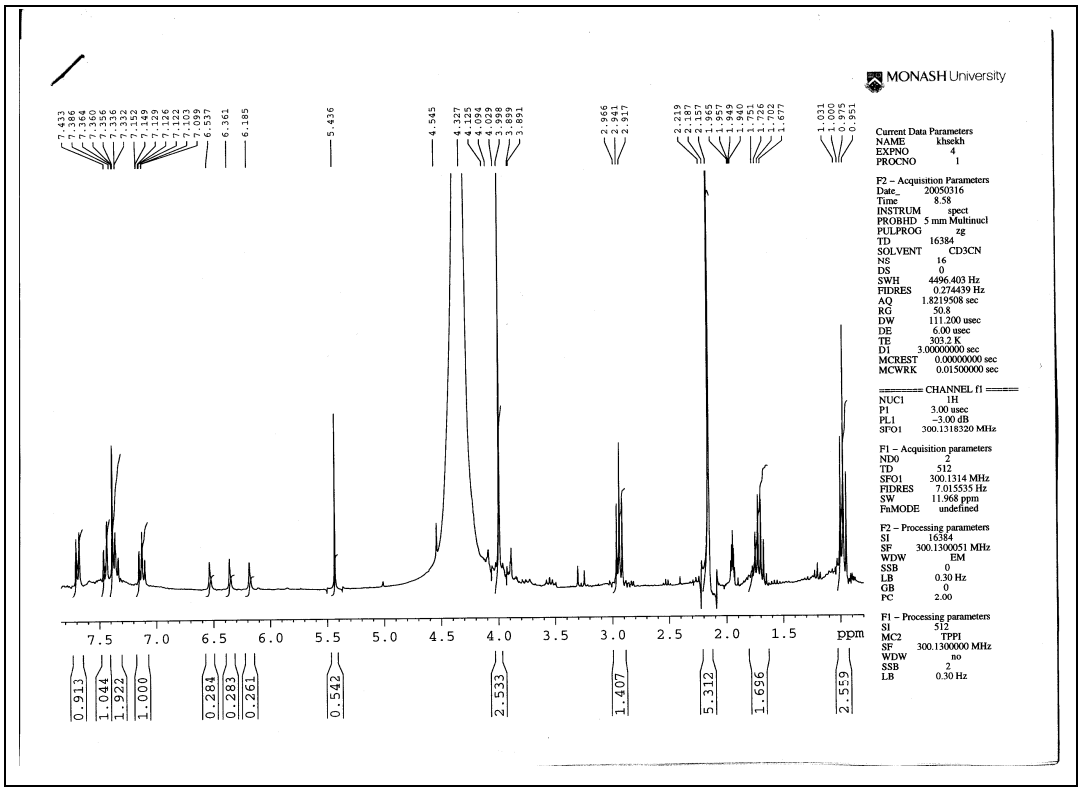
Cyclisation in ethanol: Product after heating in acetonitrile



Cyclisation in acetonitrile: Initial Product



Cyclisation in acetonitrile: Product after heating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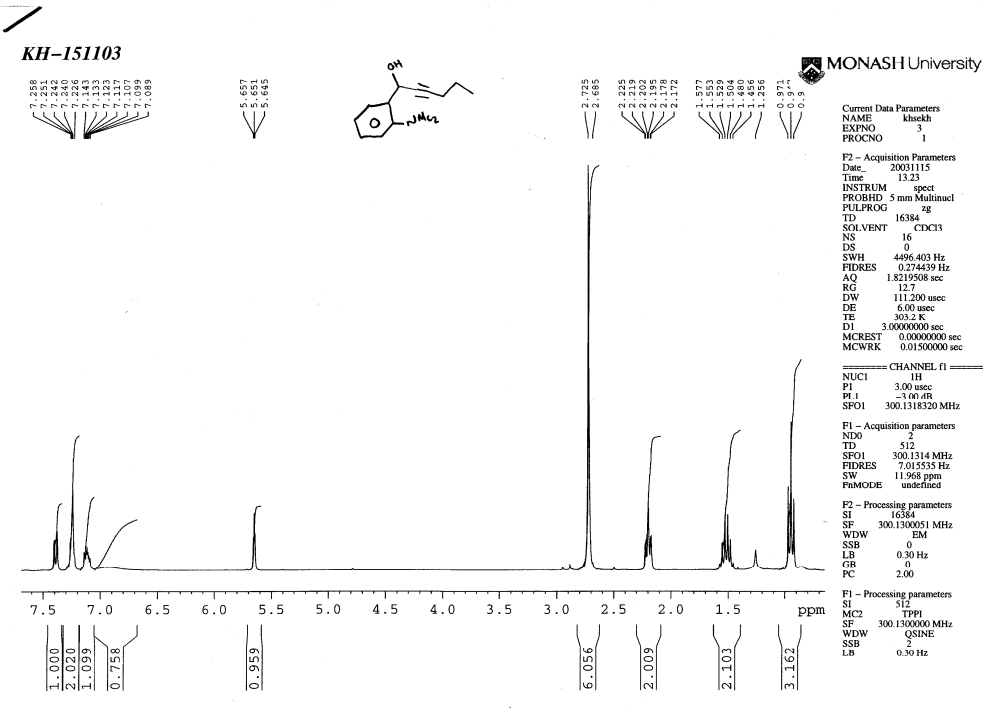
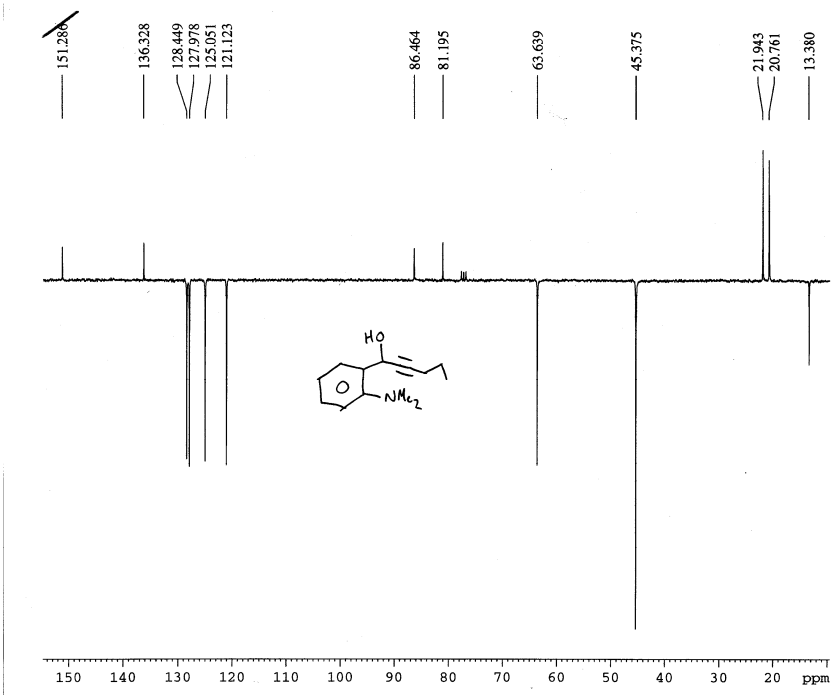
F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined
F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00
F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 ppm
0.913
1.044
1.922
1.000
0.284
0.283
0.261
0.542
2.533
1.407
5.312
1.696
2.559

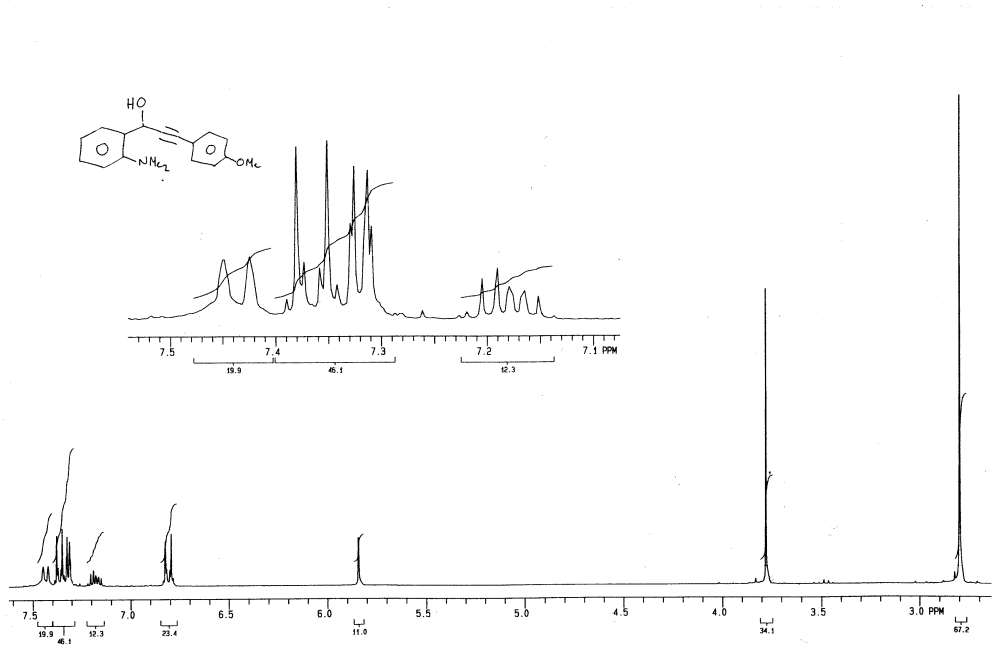
KH–151103

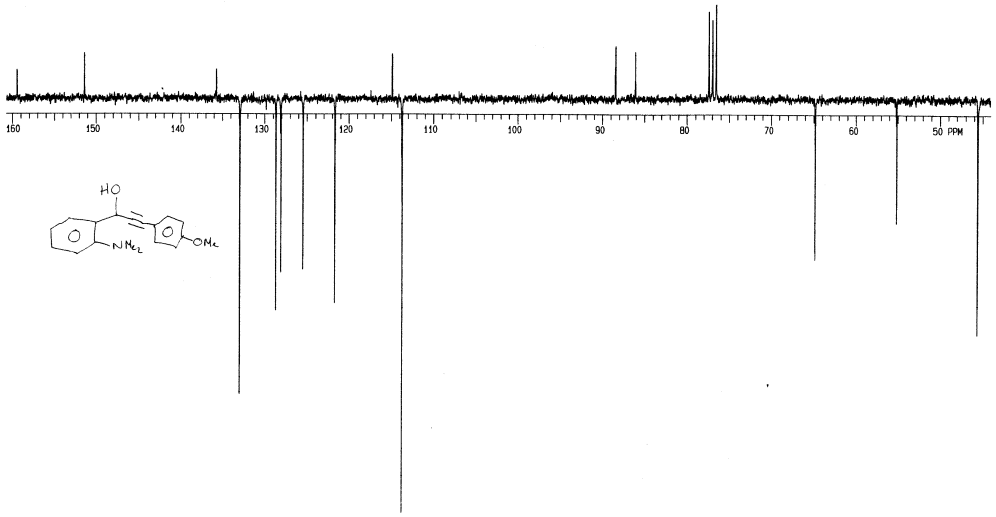

MONASH University
7.258
7.251
7.242
7.240
7.226
7.143
7.133
7.123
7.117
7.107
7.099
7.089
5.657
5.651
5.645
OH
NMe2
2.725
2.685
2.225
2.219
2.202
2.195
2.178
2.172
1.577
1.553
1.529
1.504
1.480
1.456
1.256
0.971
0.9
0.9
Current Data Parameters
NAME khsekh
EXPNO 3
PROCNO 1
F2 – Acquisition Parameters
Date_ 20031115
Time 13.23
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 12.7
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 -3.00 dB
SFO1 300.1318320 MHz
F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined
F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00
F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW QSINE
SSB 2
LB 0.30 Hz
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 ppm
1.000
2.020
1.099
0.758
0.959
6.056
2.009
2.103
3.162



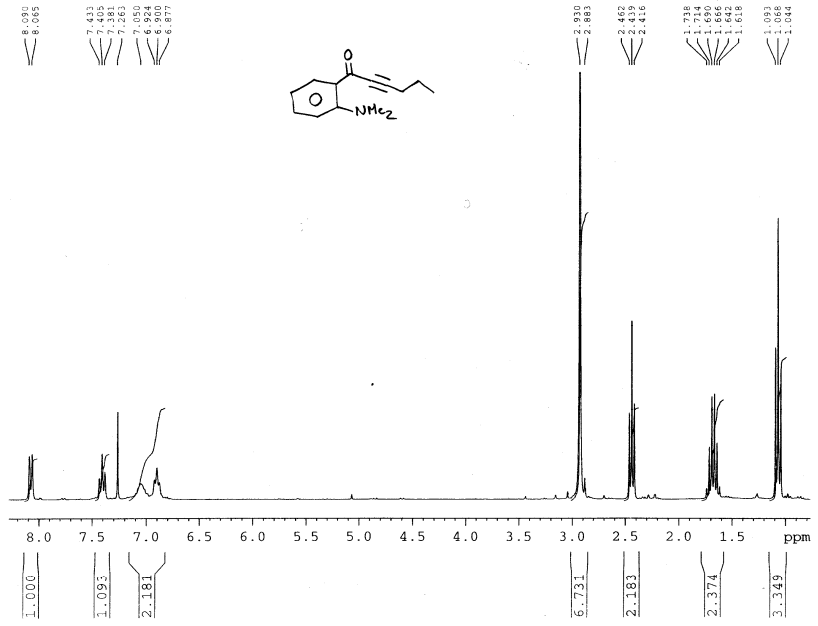
KH-3-71-a

```
Current Data Parameters
NAME                  khsekh
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_              20050307
Time                  13.42
INSTRUM               spect
PROBHD   5 mm Multinucl
PULPROG               jmod
TD                   32768
SOLVENT              CDCl3
NS                      86
DS                       4
SWH              20325.203 Hz
FIDRES           0.620276 Hz
AQ               0.8061428 sec
RG                  1824.6
DW                  24.600 usec
DE                  20.00 usec
TE                  303.2 K
CNST2           145.0000000
CNST11            1.0000000
D1              4.00000000 sec
d20             0.00689655 sec
DELTA           0.00001426 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                  13C
P1                  11.20 usec
p2                  22.40 usec
PL1                  0.00 dB
SFO1           75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2           waltz16
NUC2                  1H
PCPD2              100.00 usec
PL2                 -3.00 dB
PL12                19.40 dB
SFO2          300.1320000 MHz

F1 - Acquisition parameters
ND0                       2
TD                      512
SFO1           300.1314 MHz
FIDRES           7.015535 Hz
SW                 11.968 ppm
FnMODE           undefined

F2 - Processing parameters
SI                    32768
SF             75.4677556 MHz
WDW                      EM
SSB                       0
LB                     3.00 Hz
GB                        0
PC                     1.00

F1 - Processing parameters
```





KH–3–71: low Rf



MONASH University

Current Data Parameters
NAME khaleh
EXPNO 1
PROCNO 1

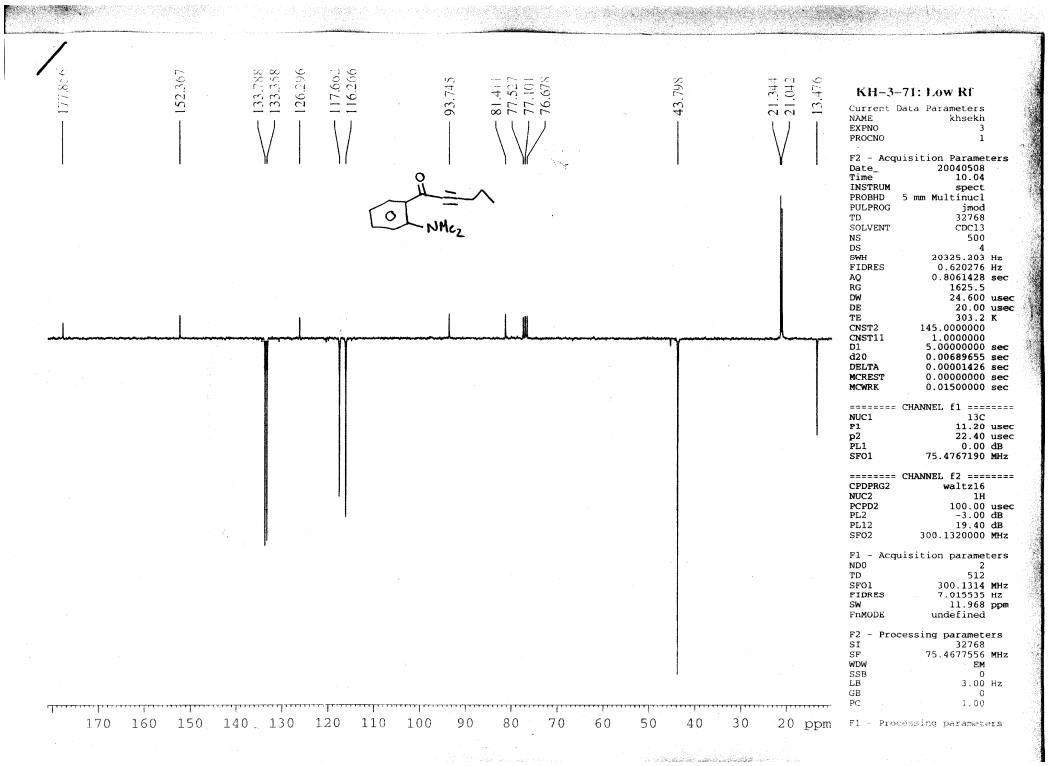
F2 – Acquisition Parameters
Date_ 20040508
Time 9.39
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 203.2
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 –3.00 dB
SFO1 300.1318320 MHz

F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz



KH–3–71: Low Rf

Current Data Parameters
NAME khsekh
EXPNO 3
PROCNO 1

F2 - Acquisition Parameters
Date_ 20040508
Time 10.04
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG jmod
TD 32768
SOLVENT CDCl3
NS 500
DS 4
SWH 20325.203 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 1625.5
DW 24.600 usec
DE 20.00 usec
TE 303.2 K
CNST2 145.00000000
CNST11 1.0000000
D1 5.00000000 sec
d20 0.00689655 sec
DELTA 0.00001426 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

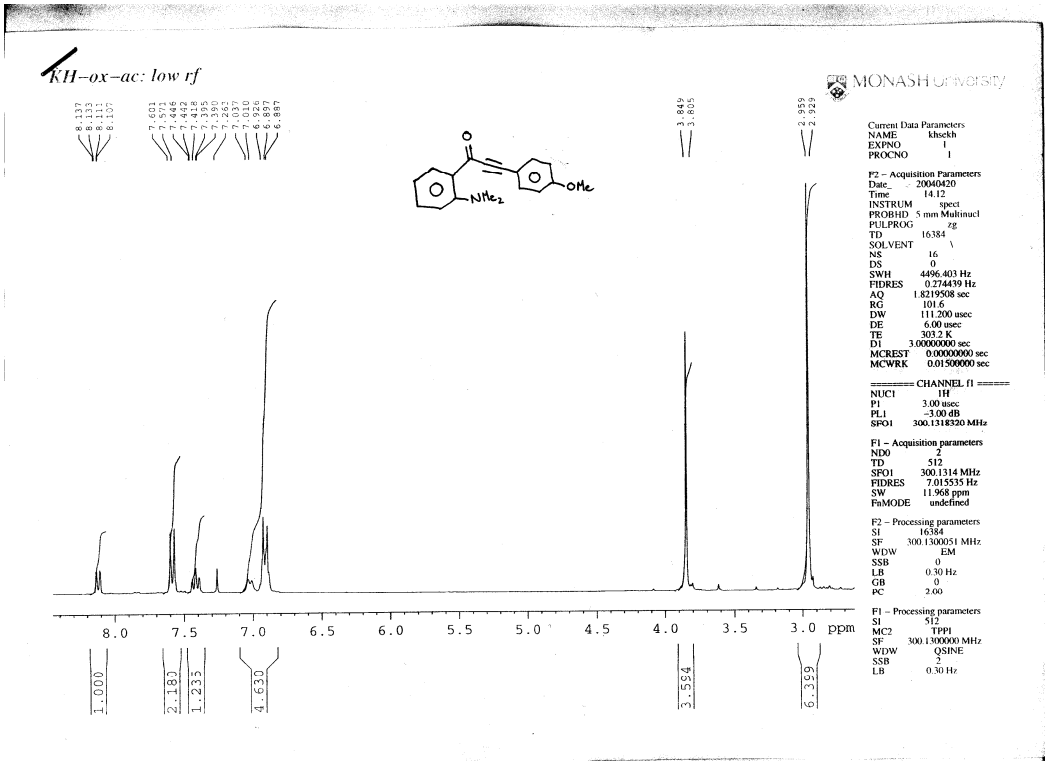
======== CHANNEL f1 ========
NUC1 13C
P1 11.20 usec
p2 22.40 usec
PL1 0.00 dB
SFO1 75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL12 19.40 dB
SFO2 300.1320000 MHz

F1 - Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 - Processing parameters
SI 32768
SF 75.4677556 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

F1 - Processing parameters



Current Data Parameters
NAME khsekh
EXPNO 1
PROCNO 1

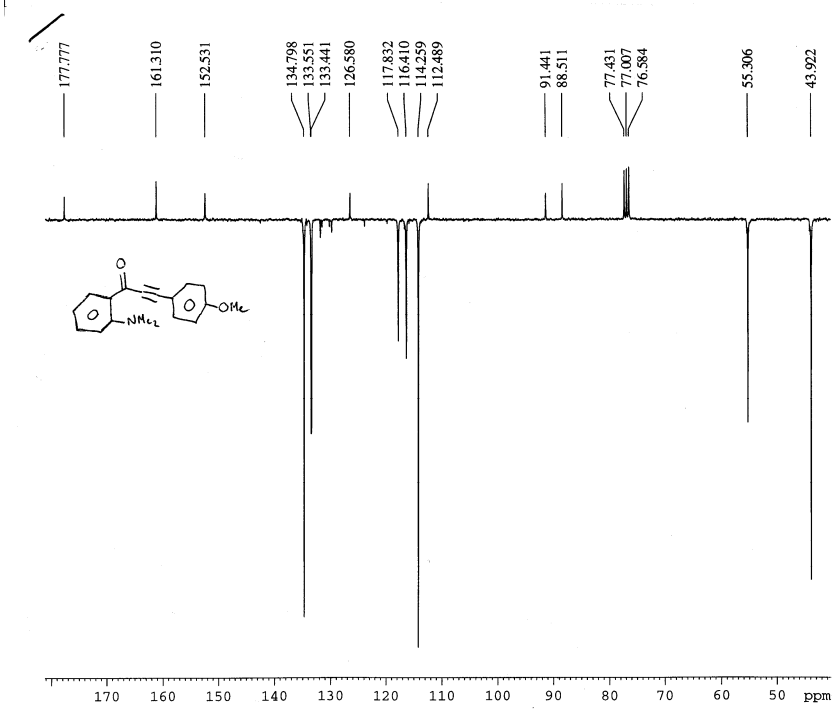
F2 – Acquisition Parameters
Date_ 20040420
Time 14.12
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT \
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 101.6
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======= CHANNEL f1 =====
NUC1 1H
P1 3.00 usec
PL1 -3.00 dB
SFO1 300.1318320 MHz

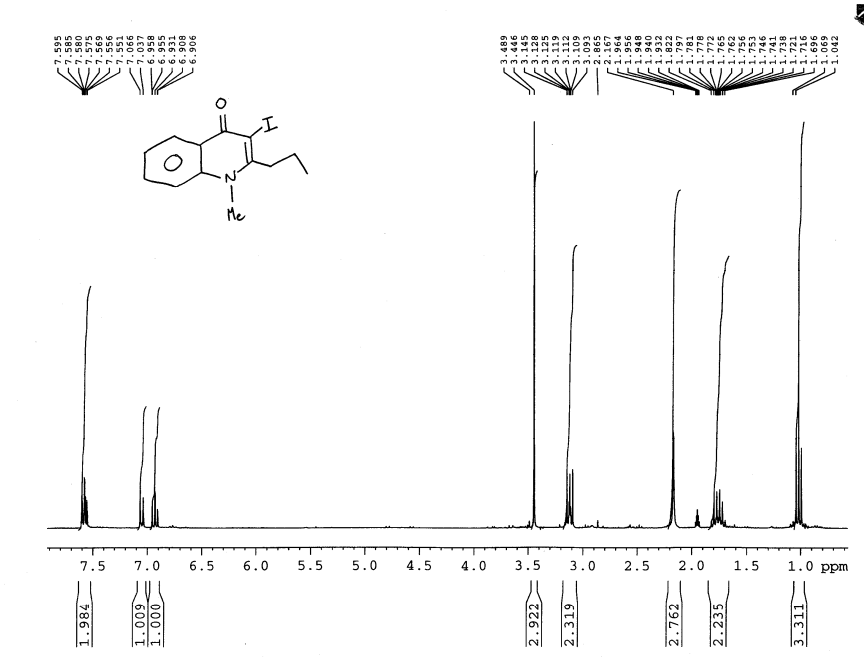
F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW QSINE
SSB 2
LB 0.30 Hz

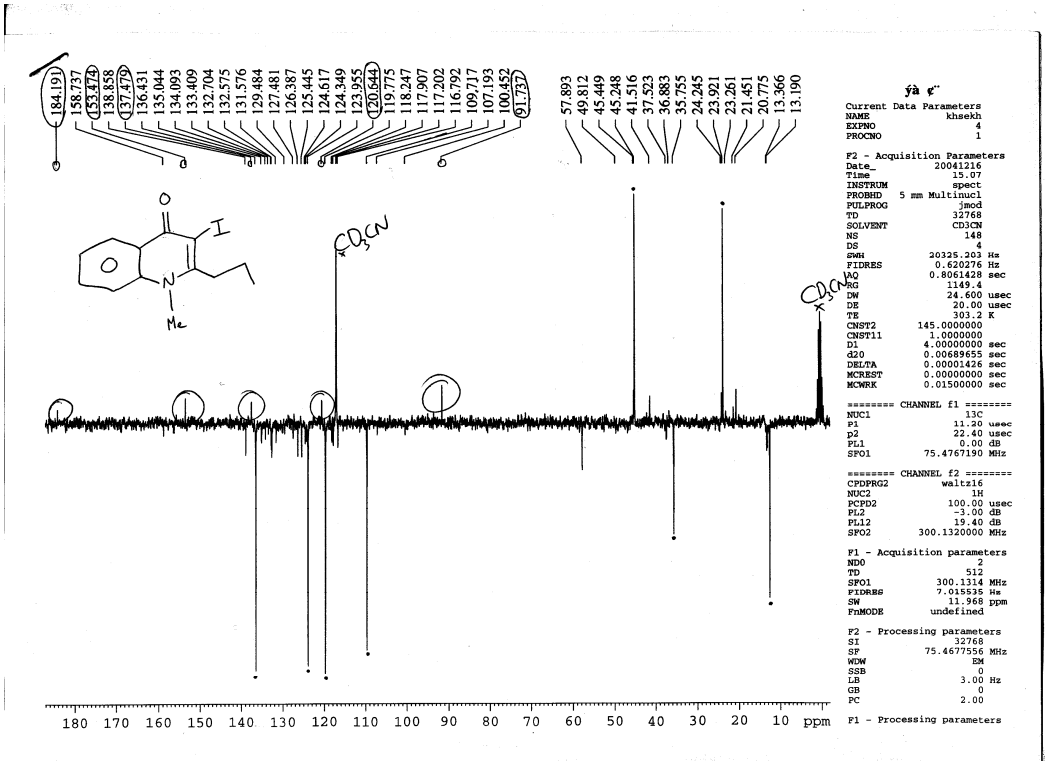


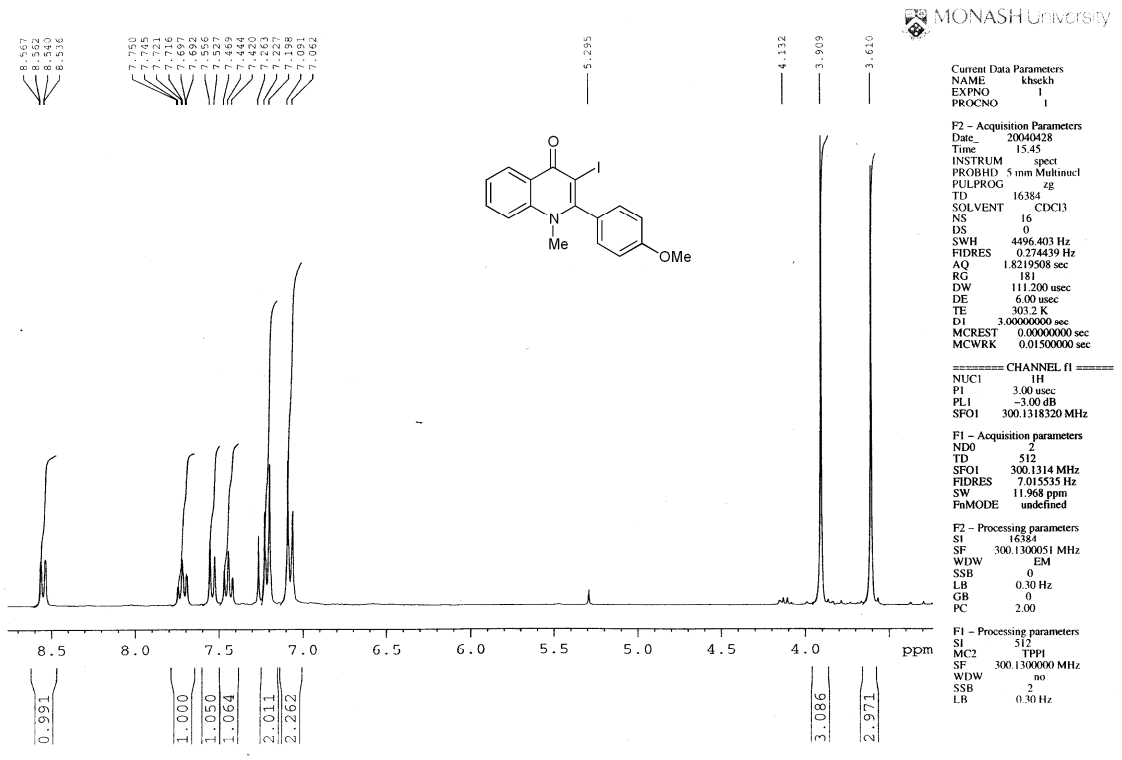

```
Current Data Parameters
NAME                  khsekh
EXPNO                      6
PROCNO                     1

F2 - Acquisition Parameters
Date_              20040916
Time                   8.07
INSTRUM               spect
PROBHD    5 mm Multinucl
PULPROG                jmod
TD                    32768
SOLVENT               CDCl3
NS                     5313
DS                        4
SWH               20325.203 Hz
FIDRES             0.620276 Hz
AQ                 0.8061428 sec
RG                      2048
DW                  24.600 usec
DE                  20.00 usec
TE                   303.2 K
CNST2             145.0000000
CNST11              1.0000000
D1                  6.00000000 sec
d20                 0.00689655 sec
DELTA               0.00001426 sec
MCREST              0.00000000 sec
MCWRK               0.01500000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                     11.20 usec
p2                     22.40 usec
PL1                     0.00 dB
SFO1              75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2               waltz16
NUC2                      1H
PCPD2                100.00 usec
PL2                    -3.00 dB
PL12                   19.40 dB
SFO2             300.1320000 MHz

F2 - Processing parameters
SI                        32768
SF                75.4677556 MHz
WDW                         EM
SSB                          0
LB                      3.00 Hz
GB                           0
PC                        1.00
```

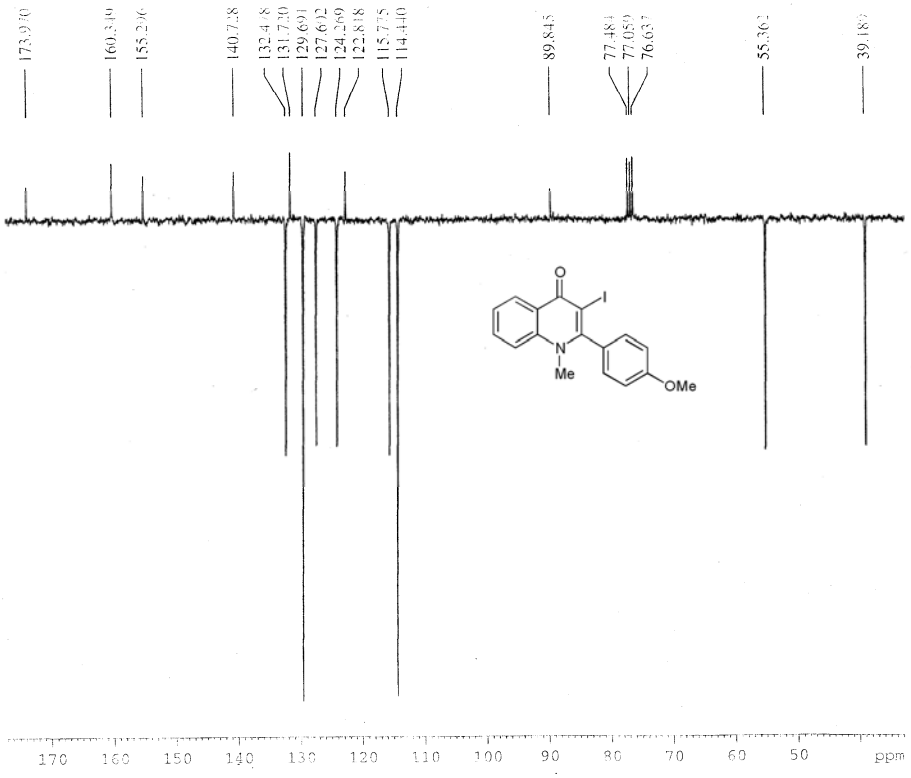


MONASH University

```
Current Data Parameters
NAME                  khsekh
EXPNO                      3
PROCNO                     1

F2 - Acquisition Parameters
Date_              20041217
Time                  14.11
INSTRUM               spect
PROBHD    5 mm Multinucl
PULPROG                  zg
TD                    16384
SOLVENT               CD3CN
NS                        16
DS                         0
SWH                4496.403 Hz
FIDRES             0.274439 Hz
AQ                 1.8219508 sec
RG                      90.5
DW                  111.200 usec
DE                    6.00 usec
TE                   303.2 K
D1                 3.00000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     3.00 usec
PL1                   -3.00 dB
SFO1             300.1318320 MHz

F1 - Acquisition parameters
ND0                        2
TD                       512
SFO1             300.1314 MHz
FIDRES             7.015535 Hz
SW                 11.968 ppm
FnMODE            undefined

F2 - Processing parameters
SI                        16384
SF               300.1300051 MHz
WDW                        EM
SSB                         0
LB                     0.30 Hz
GB                          0
PC                      2.00

F1 - Processing parameters
SI                        512
MC2                      TPPI
SF               300.1300000 MHz
WDW                        no
SSB                         2
LB                     0.30 Hz
```


184.191
158.737
153.474
138.858
137.479
136.431
135.044
134.093
133.409
132.704
132.575
131.576
129.484
127.481
126.387
125.445
124.617
124.349
123.955
120.644
119.775
118.247
117.907
117.202
116.792
109.717
107.193
100.452
91.737
57.893
49.812
45.449
45.248
41.516
37.523
36.883
35.755
24.245
23.921
23.261
21.451
20.775
13.366
13.190
CD3CN
CD3CN
Me
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
Current Data Parameters
NAME khsekh
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20041216
Time 15.07
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG jmod
TD 32768
SOLVENT CD3CN
NS 148
DS 4
SWH 20325.203 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 1149.4
DW 24.600 usec
DE 20.00 usec
TE 303.2 K
CNST2 145.0000000
CNST11 1.0000000
D1 4.00000000 sec
d20 0.00689655 sec
DELTA 0.00001426 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 13C
P1 11.20 usec
p2 22.40 usec
PL1 0.00 dB
SFO1 75.4767190 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL12 19.40 dB
SFO2 300.1320000 MHz
F1 - Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined
F2 - Processing parameters
SI 32768
SF 75.4677556 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 2.00
F1 - Processing parameters


MONASH University
8.567
8.562
8.540
8.536
7.750
7.745
7.721
7.716
7.697
7.692
7.546
7.527
7.469
7.444
7.420
7.263
7.238
7.228
7.091
7.062
5.295
4.132
3.909
3.610
O
I
N
Me
OMe
8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 ppm
0.991
1.000
1.050
1.064
2.011
2.262
3.086
2.971
Current Data Parameters
NAME khsekh
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20040428
Time 15.45
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 181
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 -3.00 dB
SFO1 300.1318320 MHz
F1 - Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined
F2 - Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00
F1 - Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz



KH-3-69

Current Data Parameters

NAME	khsekh
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20040518
Time	15.01
INSTRUM	spect
PROBHD	5 mm Multinucl
PULPROG	jmod
TD	32768
SOLVENT	CDCl3
NS	321
DS	4
SWH	20325.203 Hz
FIDRES	0.620276 Hz
AQ	0.8061428 sec
RG	1824.6
DW	24.600 usec
DE	20.00 usec
TE	303.2 K
CNST2	145.0000000
CNST11	1.0000000
D1	5.00000000 sec
d20	0.00689655 sec
DELTA	0.00001426 sec
MCREST	0.00000000 sec
MCWRK	0.01500000 sec

======== CHANNEL f1 ========

NUC1	13C
P1	11.20 usec
p2	22.40 usec
PL1	0.00 dB
SFO1	75.4767190 MHz

======== CHANNEL f2 ========

CPDPRG2	waltz16
NUC2	1H
PCPD2	100.00 usec
PL2	-3.00 dB
PL12	19.40 dB
SFO2	300.1320000 MHz

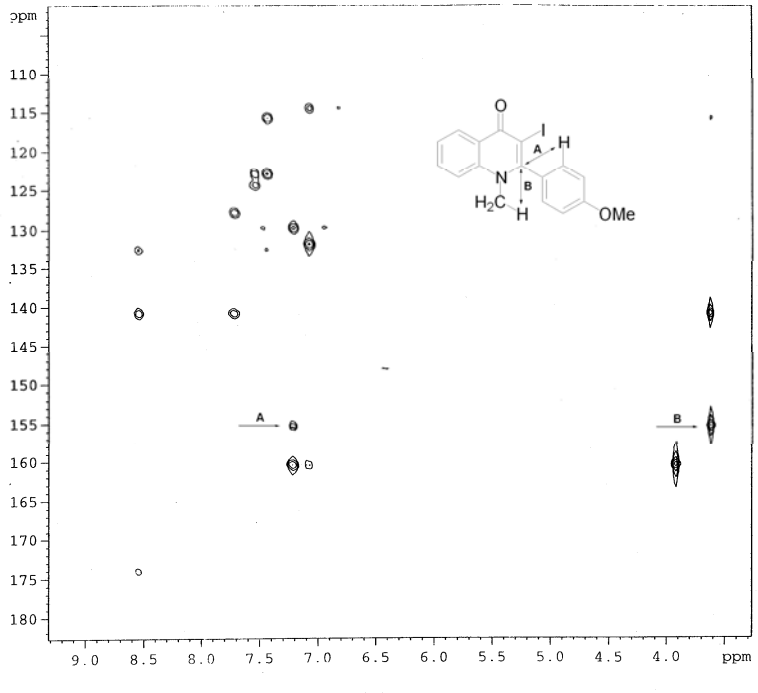
F1 - Acquisition parameters

ND0	2
TD	512
SFO1	300.1314 MHz
FIDRES	7.015535 Hz
SW	11.968 ppm
FnMODE	undefined

F2 - Processing parameters

SI	32768
SF	75.4677556 MHz
WDW	EM
SSB	0
LB	3.00 Hz
GB	0
PC	1.00

F1 - Processing parameters



MONASH University

Current Data Parameters

NAME	stsekh
EXPNO	1
PROCNO	1

F2 - Acquisition Parameters

Date_	20040519
Time	10.41
INSTRUM	spect
PROBHD	5 mm Multinucl
PULPROG	inv4gplplrnd
TD	2048
SOLVENT	CDCl3
NS	8
DS	16
SWH	4496.403 Hz
FIDRES	2.195509 Hz
AQ	0.2277876 sec
RG	4096
DW	111.200 usec
DE	6.00 usec
TE	303.2 K
CNST2	145.0000000
d0	0.00000300 sec
D1	2.00000000 sec
d2	0.00344828 sec
D6	0.06000000 sec
d13	0.00000300 sec
D16	0.00020000 sec
IN0	0.00002650 sec

======== CHANNEL f1 ========

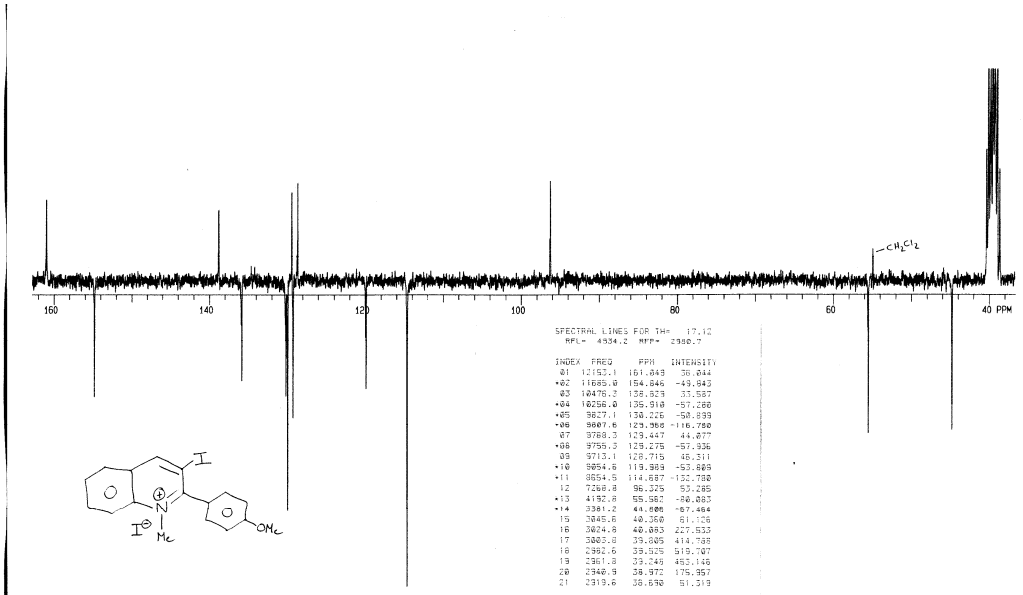
NUC1	1H
P1	7.25 usec
p2	14.50 usec
PL1	-3.00 dB
SFO1	300.1313506 MHz

======== CHANNEL f2 ========

NUC2	13C
P3	9.60 usec
PL2	-3.00 dB
SFO2	75.4763978 MHz

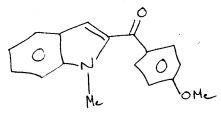
====== GRADIENT CHANNEL ====

GPNAM1	sine.100
GPNAM2	sine.100
GPNAM3	sine.100
GPX1	0.00 %
GPX2	0.00 %
GPX3	0.00 %
GPY1	0.00 %
GPY2	0.00 %
GPY3	0.00 %
GPZ1	50.00 %
GPZ2	30.00 %
GPZ3	40.10 %
P16	1000.00 usec



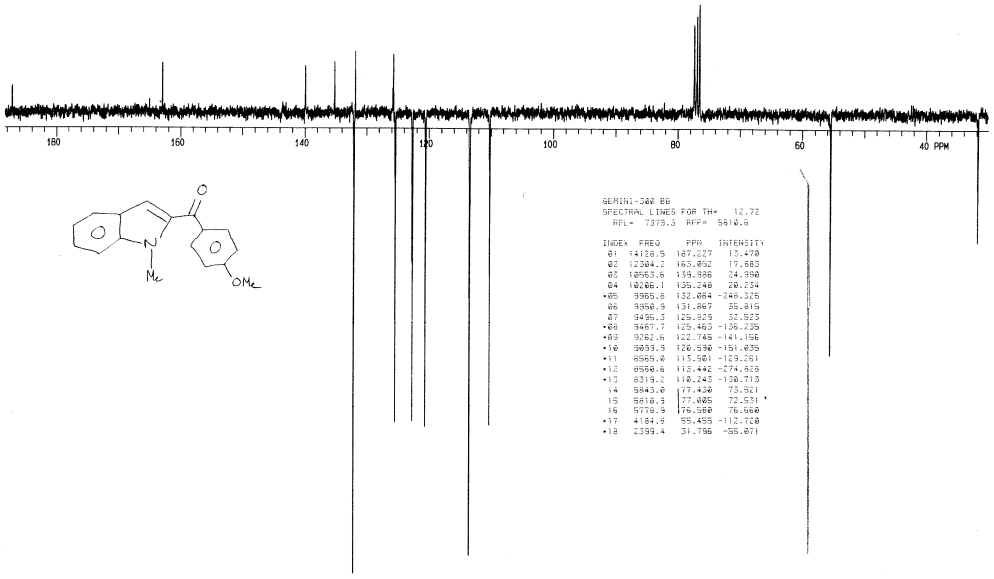
SPECTRAL LINES FOR TH= 17.12
RFL= 4934.2 RFP= 2980.7

INDEX	FREQ	PPM	INTENSITY
01	12157.1	161.843	36.044
*02	11685.0	154.846	-49.843
03	10476.2	138.829	33.587
*04	10256.0	135.910	-57.260
*05	9827.1	130.226	-58.899
*06	9607.6	129.366	-116.780
07	9788.3	129.447	44.077
*08	9758.3	129.275	-57.936
09	9713.1	128.715	46.311
*10	9054.6	119.989	-53.809
*11	8654.5	114.687	-132.780
12	7268.8	96.325	53.285
*13	4152.8	55.562	-84.083
*14	3381.2	44.806	-67.464
15	3045.6	40.360	81.126
16	3024.8	40.083	227.653
17	3003.8	39.805	414.798
18	2982.6	39.525	519.707
19	2961.8	39.246	463.146
20	2940.9	38.972	175.957
21	2919.6	38.690	51.519



GEMINI-300 BB
SPECTRAL LINES FOR TH= 7.16
RFL= 2733.6 RFP= 2178.5

INDEX	FREQ	PPM	INTENSITY
01	2398.68	7.954	51.147
02	2396.73	7.987	22.458
03	2389.95	7.965	51.446
04	2312.85	7.708	20.357
05	2304.75	7.681	21.405
06	2239.59	7.463	18.192
07	2231.90	7.438	30.857
08	2229.94	7.431	30.909
09	2223.23	7.409	18.797
10	2181.81	7.284	13.832
11	2155.24	7.163	22.428
12	2153.75	7.177	15.202
13	2146.81	7.161	10.312
14	2147.34	7.156	9.877
15	2105.89	7.018	56.160
16	2101.58	7.004	54.864
17	2097.16	6.989	53.092
18	1228.44	4.094	214.275
19	1172.70	3.908	200.851

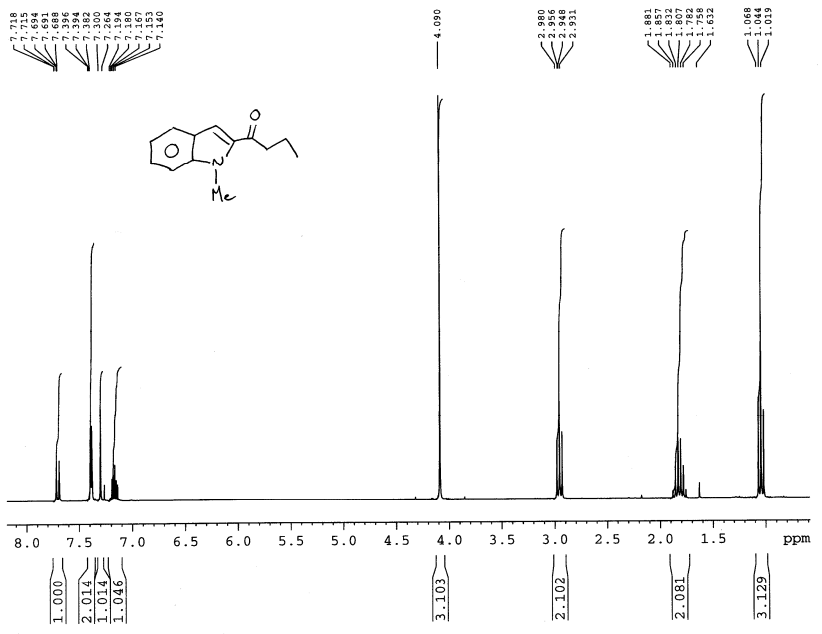


GEMINI-300 BB
SPECTRAL LINES FOR TH= 12.72
RFL= 7373.3 RPP= 5810.6

INDEX	FREQ	PPM	INTENSITY
01	14128.5	187.227	13.470
02	12594.2	163.852	17.882
03	10563.6	139.986	24.890
04	10266.1	136.248	26.234
*05	9965.8	132.064	-248.325
06	9950.9	131.867	35.819
07	9495.3	125.829	32.923
*08	9467.7	125.463	-156.235
*09	9262.6	122.745	-141.156
*10	9033.9	120.590	-191.435
*11	8565.8	113.501	-129.251
*12	8550.8	113.442	-274.826
*13	8319.2	110.243	-130.715
14	5843.8	77.420	73.521
15	5810.9	77.005	72.531
16	5776.9	76.560	76.660
*17	4184.8	55.455	-112.728
*18	2399.4	31.796	-55.671

KH–1612

MONASH University



Current Data Parameters
NAME khsekh
EXPNO 1
PROCNO 1

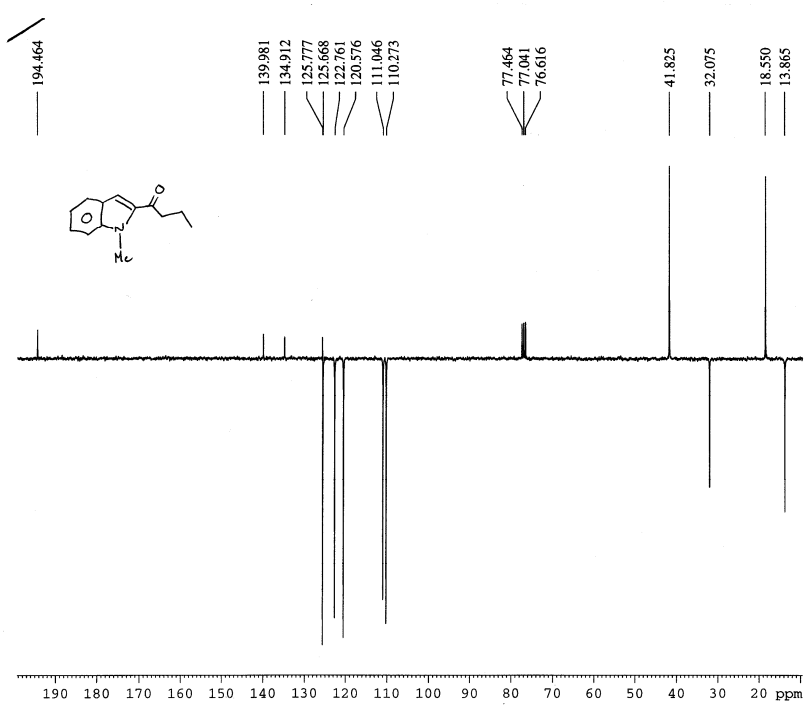
F2 – Acquisition Parameters
Date_ 20041117
Time 17.43
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 64
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 3.00 usec
PL1 -3.00 dB
SFO1 300.1318320 MHz

F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

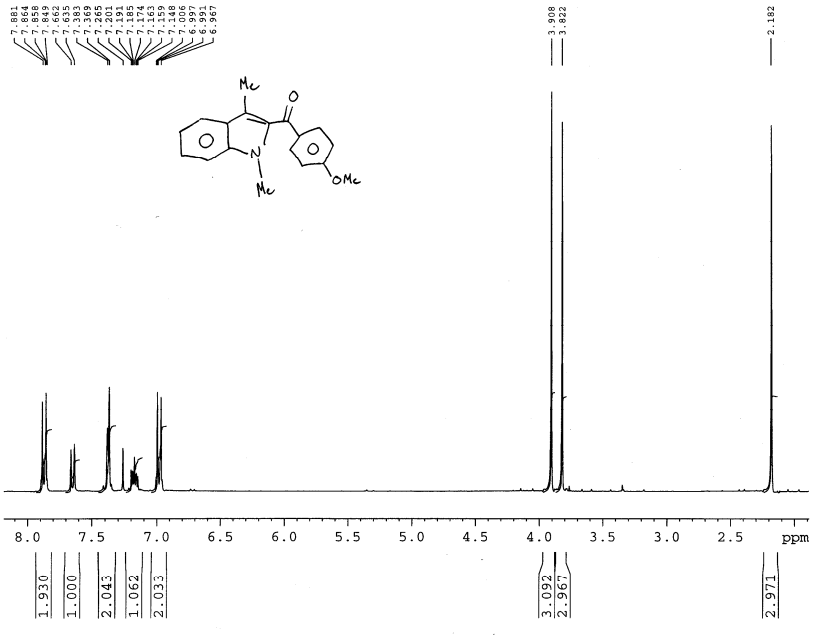
F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz



KH–1612

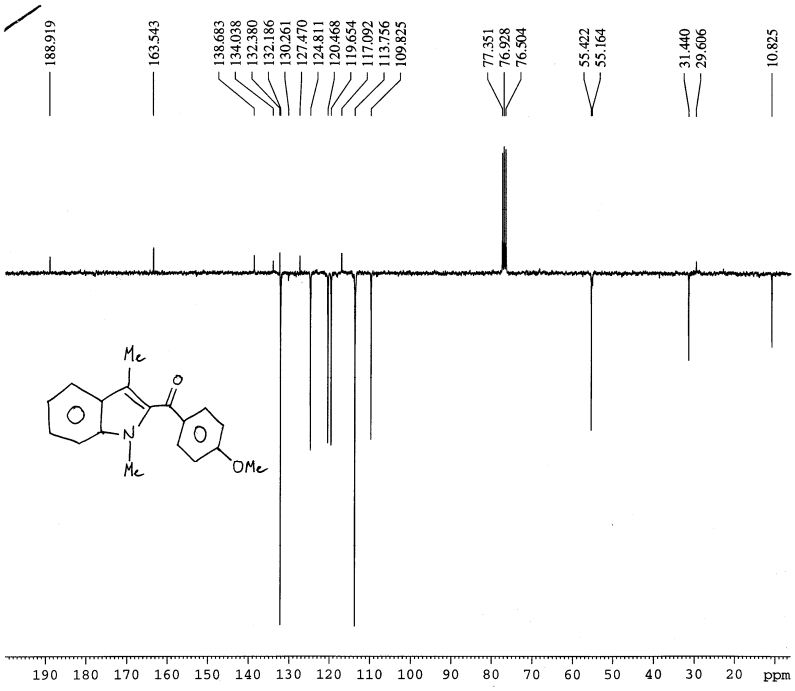
```
Current Data Parameters
NAME                  khsekh
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_              20041117
Time                  17.59
INSTRUM               spect
PROBHD    5 mm Multinucl
PULPROG                jmod
TD                    32768
SOLVENT               CDCl3
NS                      682
DS                        4
SWH               20325.203 Hz
FIDRES             0.620276 Hz
AQ                0.8061428 sec
RG                   1625.5
DW                  24.600 usec
DE                  20.00 usec
TE                   303.2 K
CNST2            145.0000000
CNST11             1.0000000
D1                 5.00000000 sec
d20                0.00689655 sec
DELTA              0.00001426 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                    11.20 usec
p2                    22.40 usec
PL1                    0.00 dB
SFO1             75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2                100.00 usec
PL2                   -3.00 dB
PL12                  19.40 dB
SFO2            300.1320000 MHz

F1 - Acquisition parameters
ND0                        2
TD                       512
SFO1              300.1314 MHz
FIDRES              7.015535 Hz
SW                   11.968 ppm
FnMODE             undefined

F2 - Processing parameters
SI                       32768
SF                75.4677556 MHz
WDW                        EM
SSB                         0
LB                       3.00 Hz
GB                          0
PC                       1.00

F1 - Processing parameters
```

KH–3–89



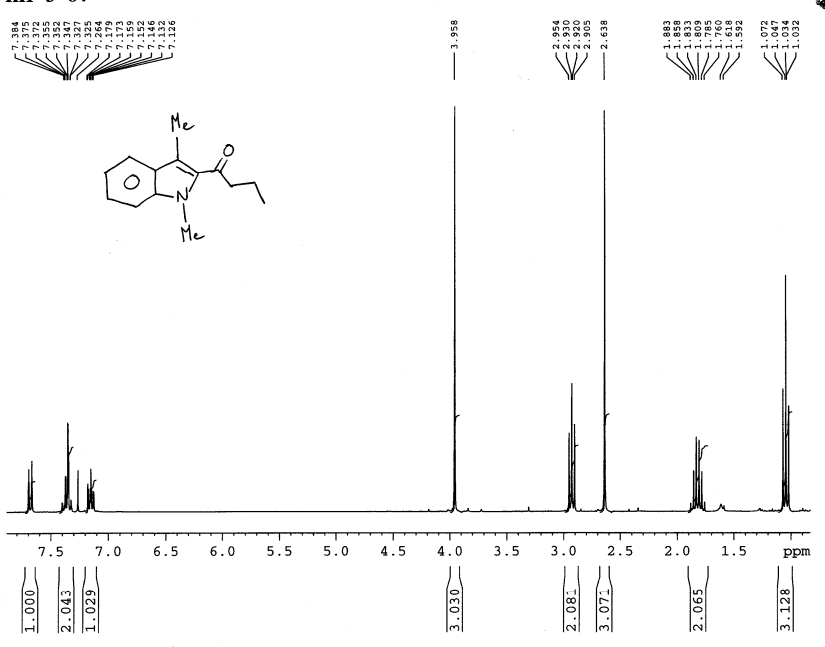
MONASH University

```
Current Data Parameters
NAME          khsekh
EXPNO              1
PROCNO             1

F2 – Acquisition Parameters
Date_       20041025
Time           15.28
INSTRUM         spect
PROBHD   5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT         CDCl3
NS                 16
DS                  0
SWH         4496.403 Hz
FIDRES      0.274439 Hz
AQ          1.8219508 sec
RG              161.3
DW          111.200 usec
DE            6.00 usec
TE            303.2 K
D1          3.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ======
NUC1               1H
P1              3.00 usec
PL1            -3.00 dB
SFO1     300.1318320 MHz

F1 – Acquisition parameters
ND0                2
TD               512
SFO1      300.1314 MHz
FIDRES      7.015535 Hz
SW          11.968 ppm
FnMODE      undefined

F2 – Processing parameters
SI               16384
SF        300.1300051 MHz
WDW               EM
SSB                0
LB              0.30 Hz
GB                 0
PC              2.00

F1 – Processing parameters
SI               512
MC2              TPPI
SF        300.1300000 MHz
WDW              no
SSB                2
LB              0.30 Hz
```



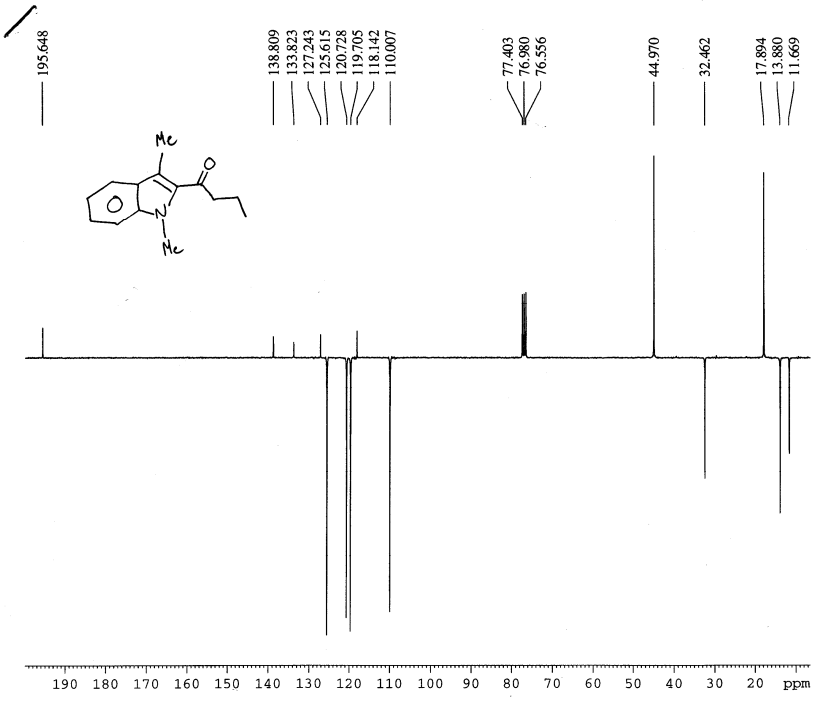
KH–3–89

```
Current Data Parameters
NAME                  khsekh
EXPNO                      7
PROCNO                     1

F2 - Acquisition Parameters
Date_              20040714
Time                  17.26
INSTRUM               spect
PROBHD   5 mm Multinucl
PULPROG                jmod
TD                    32768
SOLVENT               CDCl3
NS                    11551
DS                        4
SWH               20325.203 Hz
FIDRES            0.620276 Hz
AQ                0.8061428 sec
RG                     2048
DW                   24.600 usec
DE                   20.00 usec
TE                   303.2 K
CNST2            145.0000000
CNST11             1.0000000
D1                 4.00000000 sec
d20                0.00689655 sec
DELTA              0.00001426 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                     11.20 usec
p2                     22.40 usec
PL1                     0.00 dB
SFO1              75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2              waltz16
NUC2                     1H
PCPD2                100.00 usec
PL2                   -3.00 dB
PL12                  19.40 dB
SFO2             300.1320000 MHz

F1 - Acquisition parameters
ND0                        2
TD                       512
SFO1             300.1314 MHz
FIDRES             7.015535 Hz
SW                  11.968 ppm
FnMODE             undefined

F2 - Processing parameters
SI                       32768
SF               75.4677556 MHz
WDW                         EM
SSB                         0
LB                       3.00 Hz
GB                          0
PC                       1.00

F1 - Processing parameters
```



MONASH University

```
Current Data Parameters
NAME          khsekh
EXPNO              2
PROCNO             1

F2 – Acquisition Parameters
Date_        20041125
Time            15.43
INSTRUM         spect
PROBHD  5 mm Multinucl
PULPROG            zg
TD              16384
SOLVENT          CDCl3
NS                 16
DS                  0
SWH         4496.403 Hz
FIDRES      0.274439 Hz
AQ          1.8219508 sec
RG                90.5
DW            111.200 usec
DE              6.00 usec
TE              303.2 K
D1          3.00000000 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1               1H
P1              3.00 usec
PL1            -3.00 dB
SFO1    300.1318320 MHz

F1 – Acquisition parameters
ND0                2
TD               512
SFO1    300.1314 MHz
FIDRES    7.015535 Hz
SW         11.968 ppm
FnMODE     undefined

F2 – Processing parameters
SI             16384
SF      300.1300051 MHz
WDW             EM
SSB             0
LB           0.30 Hz
GB              0
PC           2.00

F1 – Processing parameters
SI               512
MC2             TPPI
SF      300.1300000 MHz
WDW             no
SSB              2
LB           0.30 Hz
```



KH–3–84

Current Data Parameters
NAME khsekh
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041126
Time 17.16
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG jmod
TD 32768
SOLVENT CDCl3
NS 30000
DS 4
SWH 20325.203 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 1824.6
DW 24.600 usec
DE 20.00 usec
TE 303.2 K
CNST2 145.0000000
CNST11 1.0000000
D1 5.00000000 sec
d20 0.00689655 sec
DELTA 0.00001426 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 11.20 usec
p2 22.40 usec
PL1 0.00 dB
SFO1 75.4767190 MHz

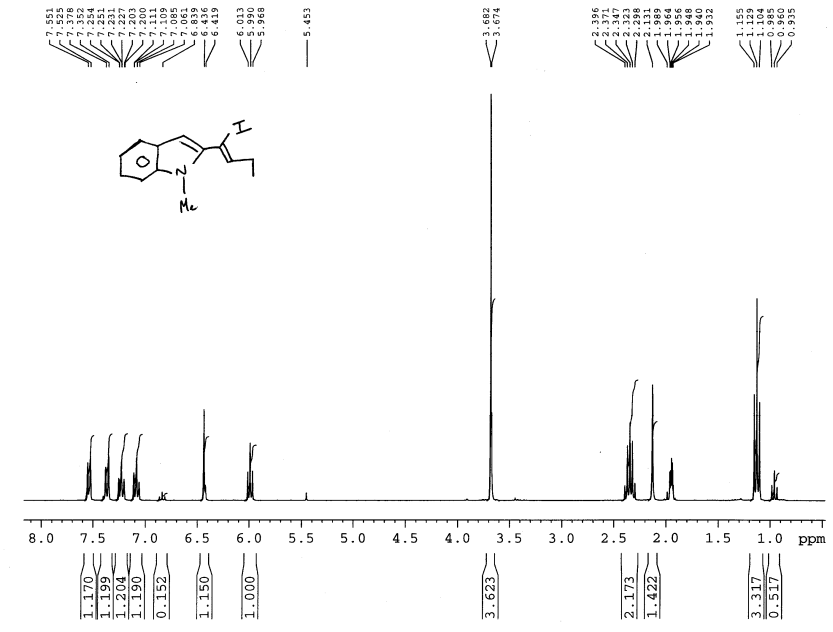
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL12 19.40 dB
SFO2 300.1320000 MHz

F1 - Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 - Processing parameters
SI 32768
SF 75.4677556 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

F1 - Processing parameters

4–vi



MONASH University

Current Data Parameters
NAME khsekh
EXPNO 5
PROCNO 1

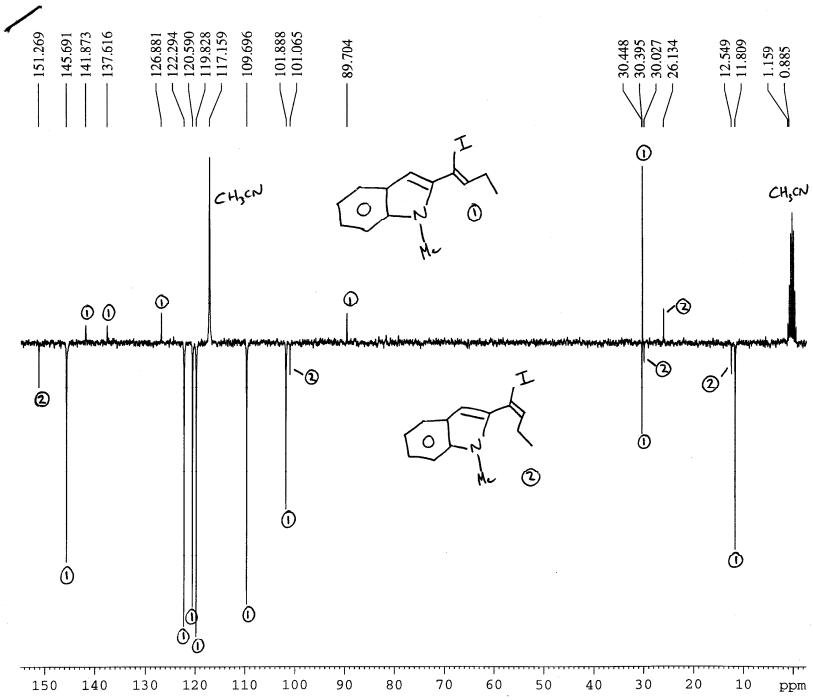
F2 – Acquisition Parameters
Date_ 20040923
Time 19.37
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CD3CN
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 161.3
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 -3.00 dB
SFO1 300.1318320 MHz

F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz

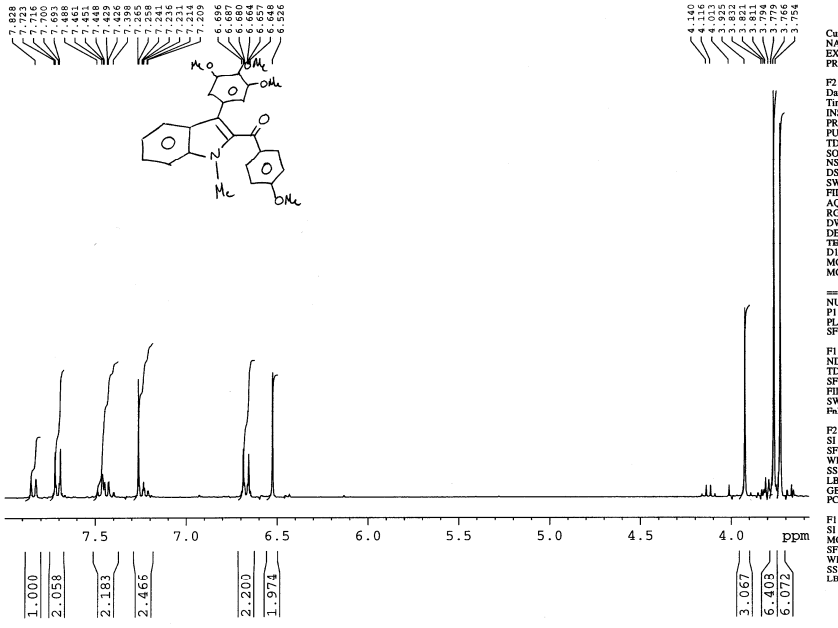


4–vi

```
Current Data Parameters
NAME                  khsekh
EXPNO                      6
PROCNO                     1

F2 - Acquisition Parameters
Date_              20040923
Time                  19.49
INSTRUM               spect
PROBHD   5 mm Multinucl
PULPROG                jmod
TD                    32768
SOLVENT               CD3CN
NS                      406
DS                        4
SWH               20325.203 Hz
FIDRES             0.620276 Hz
AQ                0.8061428 sec
RG                    1824.6
DW                  24.600 usec
DE                  20.00 usec
TE                    303.2 K
CNST2             145.00000000
CNST11              1.0000000
D1                5.00000000 sec
d20               0.00689655 sec
DELTA             0.00001426 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                     11.20 usec
p2                     22.40 usec
PL1                     0.00 dB
SFO1              75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2               waltz16
NUC2                       1H
PCPD2                 100.00 usec
PL2                    -3.00 dB
PL12                   19.40 dB
SFO2             300.1320000 MHz

F1 - Acquisition parameters
ND0                        2
TD                       512
SFO1             300.1314 MHz
FIDRES             7.015535 Hz
SW                    11.968 ppm
FnMODE              undefined

F2 - Processing parameters
SI                       32768
SF               75.4677556 MHz
WDW                         EM
SSB                          0
LB                    3.00 Hz
GB                           0
PC                        1.00

F1 - Processing parameters
```

KH–4–58 lrf

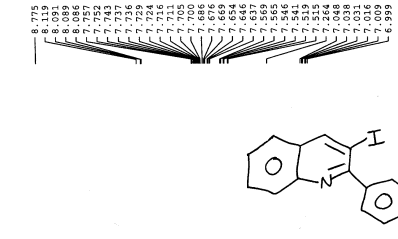
MONASH University

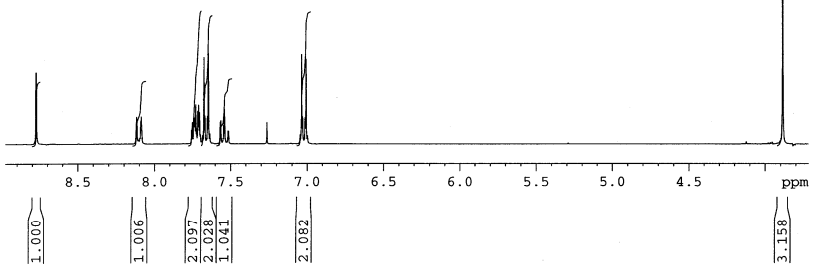



```
Current Data Parameters
NAME                  khsekh
EXPNO                      1
PROCNO                     1

F2 – Acquisition Parameters
Date_              20041029
Time                  10.02
INSTRUM               spect
PROBHD   5 mm Multinucl
PULPROG                  zg
TD                    16384
SOLVENT               CDCl3
NS                       16
DS                        0
SWH               4496.403 Hz
FIDRES             0.274439 Hz
AQ                1.8219508 sec
RG                      362
DW                  111.200 usec
DE                    6.00 usec
TE                    303.2 K
D1                3.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ======
NUC1                       1H
P1                     3.00 usec
PL1                   -3.00 dB
SFO1              300.1318320 MHz

F1 – Acquisition parameters
ND0                        2
TD                       512
SFO1             300.1314 MHz
FIDRES             7.015535 Hz
SW                    11.968 ppm
FnMODE              undefined

F2 – Processing parameters
SI                       16384
SF               300.1300051 MHz
WDW                         EM
SSB                          0
LB                    0.30 Hz
GB                           0
PC                        2.00

F1 – Processing parameters
SI                         512
MC2                       TPPI
SF               300.1300000 MHz
WDW                      QSINE
SSB                          2
LB                    0.30 Hz
```

KH–4–66

MONASH University





Current Data Parameters
NAME khsekh
EXPNO 7
PROCNO 1

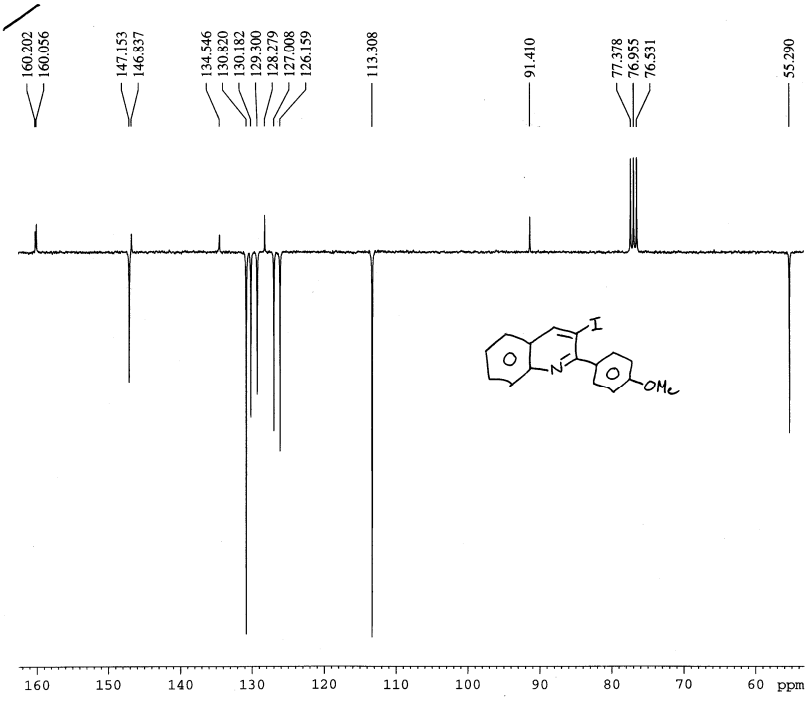
F2 – Acquisition Parameters
Date_ 20050303
Time 18.01
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 101.6
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 −3.00 dB
SFO1 300.1318320 MHz

F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz



KH–4–66

Current Data Parameters
NAME khsekh
EXPNO 8
PROCNO 1

F2 - Acquisition Parameters
Date_ 20050303
Time 18.09
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG jmod
TD 32768
SOLVENT CDCl3
NS 9253
DS 4
SWH 20325.203 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 1824.6
DW 24.600 usec
DE 20.00 usec
TE 303.2 K
CNST2 145.0000000
CNST11 1.0000000
D1 5.00000000 sec
d20 0.00689655 sec
DELTA 0.00001426 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 11.20 usec
p2 22.40 usec
PL1 0.00 dB
SFO1 75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL12 19.40 dB
SFO2 300.1320000 MHz

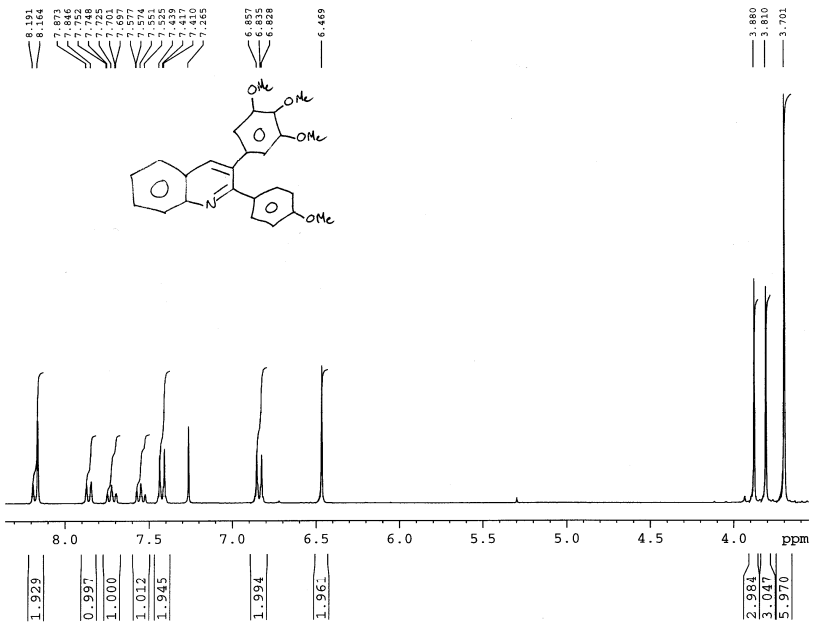
F1 - Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 - Processing parameters
SI 32768
SF 75.4677556 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

F1 - Processing parameters

KH–4–61

MONASH University



Current Data Parameters
NAME khsekh
EXPNO 3
PROCNO 1

F2 – Acquisition Parameters
Date_ 20041020
Time 16.28
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 64
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 287.4
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

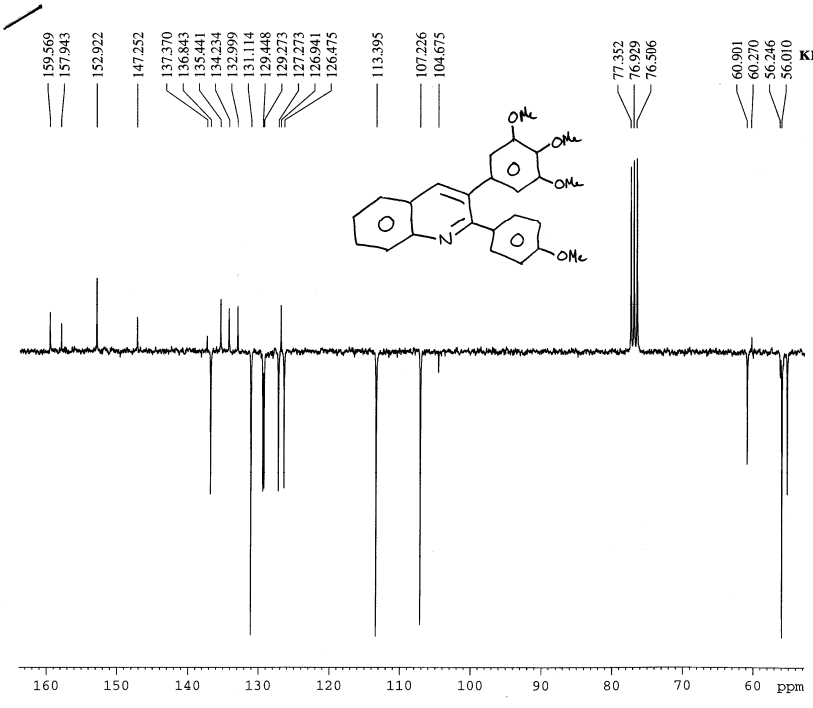
======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 −3.00 dB
SFO1 300.1318320 MHz

F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz

KH–Suzuki Coupling: ac #2



Current Data Parameters
NAME khsekh
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041020
Time 9.00
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG jmod
TD 32768
SOLVENT CDCl3
NS 6751
DS 4
SWH 20325.203 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 1824.6
DW 24.600 usec
DE 20.00 usec
TE 303.2 K
CNST2 145.0000000
CNST11 1.0000000
D1 5.00000000 sec
d20 0.00689655 sec
DELTA 0.00001426 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 11.20 usec
p2 22.40 usec
PL1 0.00 dB
SFO1 75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL12 19.40 dB
SFO2 300.1320000 MHz

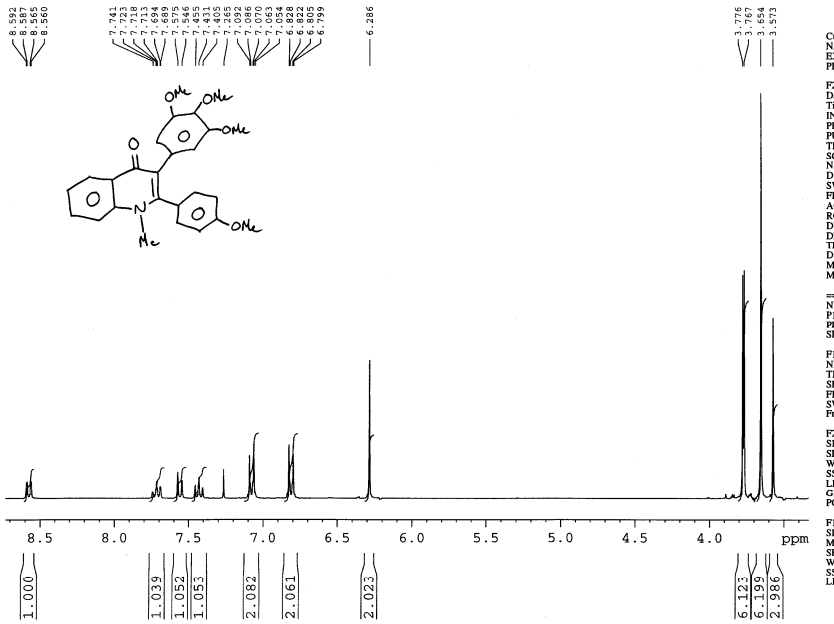
F1 - Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 - Processing parameters
SI 32768
SF 75.4677556 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

F1 - Processing parameters

KH–4–56

MONASH University



Current Data Parameters
NAME khsekh
EXPNO 1
PROCNO 1

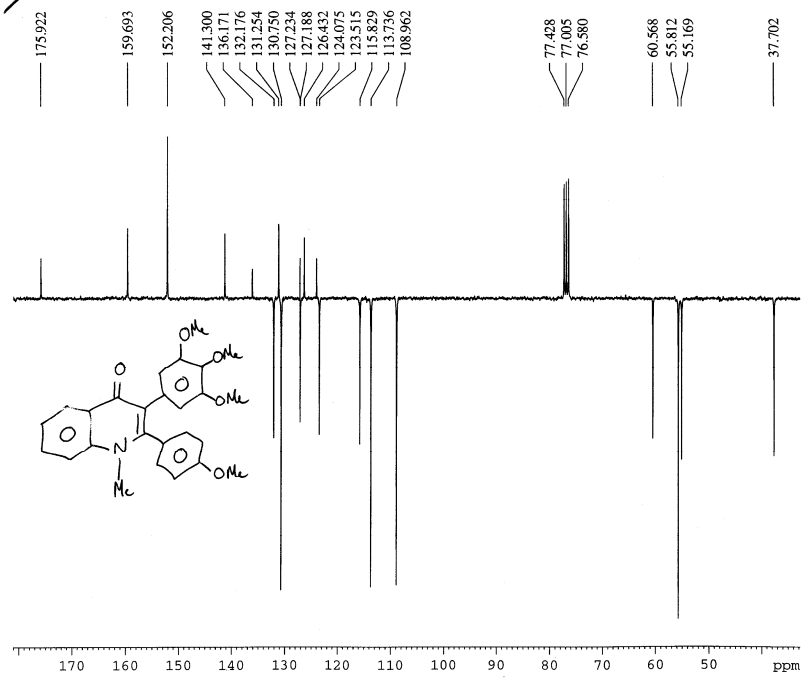
F2 – Acquisition Parameters
Date_ 20041018
Time 13.42
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG zg
TD 16384
SOLVENT CDCl3
NS 16
DS 0
SWH 4496.403 Hz
FIDRES 0.274439 Hz
AQ 1.8219508 sec
RG 114
DW 111.200 usec
DE 6.00 usec
TE 303.2 K
D1 3.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ======
NUC1 1H
P1 3.00 usec
PL1 -3.00 dB
SFO1 300.1318320 MHz

F1 – Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 – Processing parameters
SI 16384
SF 300.1300051 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 2.00

F1 – Processing parameters
SI 512
MC2 TPPI
SF 300.1300000 MHz
WDW no
SSB 2
LB 0.30 Hz



KH–4–56

Current Data Parameters
NAME khsekh
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20041006
Time 19.57
INSTRUM spect
PROBHD 5 mm Multinucl
PULPROG jmod
TD 32768
SOLVENT CDCl3
NS 5138
DS 4
SWH 20325.203 Hz
FIDRES 0.620276 Hz
AQ 0.8061428 sec
RG 2048
DW 24.600 usec
DE 20.00 usec
TE 303.2 K
CNST2 145.0000000
CNST11 1.0000000
D1 8.00000000 sec
d20 0.00689655 sec
DELTA 0.00001426 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ========
NUC1 13C
P1 11.20 usec
p2 22.40 usec
PL1 0.00 dB
SFO1 75.4767190 MHz

======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 -3.00 dB
PL12 19.40 dB
SFO2 300.1320000 MHz

F1 - Acquisition parameters
ND0 2
TD 512
SFO1 300.1314 MHz
FIDRES 7.015535 Hz
SW 11.968 ppm
FnMODE undefined

F2 - Processing parameters
SI 32768
SF 75.4677556 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00

F1 - Processing parameters