

Construction of α -Amido-indanones via Formal Allenamide Hydroacylation /Nazarov Cyclization

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Supporting Information: Experimental procedures, characterization data for all compounds and synthetic intermediates.

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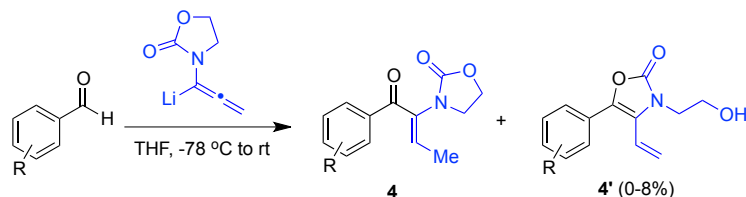
General Information. Reactions were carried out in flame-dried glassware under a positive argon atmosphere unless otherwise stated. Transfer of anhydrous solvents and reagents was accomplished with oven-dried syringes. Solvents were distilled before use: methylene chloride and 1,2-dichloroethane from calcium hydride, tetrahydrofuran from sodium/benzophenone ketyl. All other solvents and commercially available reagents were either purified by standard procedures or used without further purification. Thin layer chromatography was performed on glass plates precoated with 0.25 mm silica gel; the stains for TLC analysis were conducted with 2.5 % *p*-anisaldehyde in AcOH-H₂SO₄-EtOH (1:3:85) and further heating until development of color. Flash chromatography was performed on 230-400 mesh silica gel with the indicated eluents. Nuclear magnetic resonance (NMR) spectra were recorded in indicated deuterated solvents and are reported in ppm in the presence of TMS as internal standard and coupling constants (*J*) are reported in Hertz (Hz). Infrared (IR) spectra were recorded neat and reported in cm⁻¹. Mass spectra were recorded by

using EI or ESI as specified in each case.

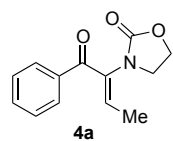
Standard Procedure for the Synthesis of 4:

To a flame-dried round bottom flask containing a magnetic stirring bar was added allenamide **1** (0.5 mmol) and anhydrous THF (6 mL) under Ar. The temperature of the solution was reduced to $-78\text{ }^{\circ}\text{C}$ (acetone/dry ice bath). *n*-BuLi (1.2 equiv, 2.27 M in hexane, 0.26 mL) was added dropwise using 1 mL syringe. After stirring for 45 min, a solution of aldehydes **2** (1.1 equiv, 0.55 mmol) was added. The reaction mixture was stirred at $-78\text{ }^{\circ}\text{C}$ for 5 min, then allowed to warm to rt for 10 min. The reaction was quenched with sat. aq NH_4Cl solution. The organic layer was separated and the aqueous layer was extracted with Et_2O (3 x 20 mL). The combined organic layer was washed with brine (1 x 20 mL), dried over anhydrous MgSO_4 , and then filtered through a pad of celite. The solvent was removed under reduced pressure and the residue was purified on a silica gel column to afford the desired product.

In several cases, especially those reactions with lower yields, side product **4'** inconsistently appeared in the crude mixture and could not be obtained in pure form. The crude ^1H NMR spectrum clearly indicated the presence of vinyl protons and ethylene unit. The ^{13}C NMR spectrum showed the existence of only two aliphatic carbons and several other sp^2 carbons. Also, the IR spectrum revealed the presence of a hydroxy group. The structure of **4'** was tentatively assigned based on these data. This side reaction will be further explored in detail.

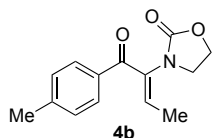


Characterization Data for 4a-i and d-4a:



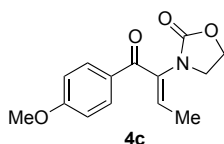
(*Z*)-3-(1-Oxo-1-phenylbut-2-en-2-yl) oxazolidin-2-one (**4a**)

Following the representative procedure, flash chromatography (hexane/EtOAc = 1/1) gave **4a** in 65% yield as a colorless oil: IR (film) 3062, 2960, 2916, 1756, 1654, 1598, 1413, 1247 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 7.74-7.71 (m, 2H), 7.58-7.53 (m, 1H), 7.47-7.27 (m, 2H), 6.63 (q, $J = 6.8$ Hz, 1H), 4.52-4.48 (m, 2H), 3.86-3.82 (m, 2H), 1.96 (d, $J = 6.8$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 192.0, 156.6, 142.0, 137.0, 135.6, 132.0, 128.8, 128.0, 62.5, 45.5, 14.1; HRMS (EI, M^+) Calcd for $\text{C}_{13}\text{H}_{13}\text{NO}_3$ 231.0895; found m/z 231.0889.



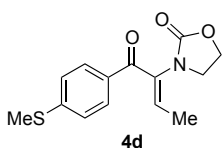
(Z)-3-(1-Oxo-1-(p-tolyl)but-2-en-2-yl) oxazolidin-2-one (4b)

Following the representative procedure, flash chromatography (hexane/EtOAc = 1/1) gave **4b** in 44% yield as a colorless oil: IR (film) 2959, 2921, 2852, 1755, 1653, 1606, 1412, 1246 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 7.65-7.63 (m, 2H), 7.27-7.23 (m, 2H), 6.60 (q, $J = 7.2$ Hz, 1H), 4.50-4.46 (m, 2H), 3.84-3.80 (m, 2H), 2.41 (s, 3H), 1.95 (d, $J = 6.8$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 191.7, 156.5, 142.8, 141.1, 135.5, 134.3, 129.1, 128.7, 62.4, 45.5, 21.2, 14.0; HRMS (EI, M^+) Calcd for $\text{C}_{14}\text{H}_{15}\text{NO}_3$ 245.1052; found m/z 245.1050.



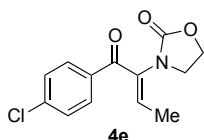
(Z)-3-(1-(4-Methoxyphenyl)-1-oxobut-2-en-2-yl) oxazolidin-2-one (4c)

Following the representative procedure, flash chromatography (hexane/EtOAc = 1/1) gave **4c** in 18% yield as a pale yellow oil: IR (film) 2915, 2842, 1755, 1648, 1600, 1510, 1414, 1252 cm^{-1} ; ^1H NMR (400 MHz, CDCl_3) δ 7.78-7.74 (m, 2H), 6.95-6.92 (m, 2H), 6.55 (q, $J = 6.8$ Hz, 1H), 4.50-4.46 (m, 2H), 3.87 (s, 3H), 3.84-3.80 (m, 2H), 1.95 (d, $J = 7.2$ Hz, 3H); ^{13}C NMR (100 MHz, CDCl_3) δ 190.7, 162.9, 156.5, 139.7, 135.4, 131.3, 129.4, 113.3, 62.4, 55.1, 45.6, 13.9; HRMS (EI, M^+) Calcd for $\text{C}_{14}\text{H}_{15}\text{NO}_4$ 261.1001; found m/z 261.0999.



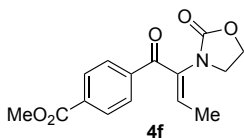
(Z)-3-(1-(4-(Methylthio) phenyl)-1-oxobut-2-en-2-yl) oxazolidin-2-one (4d)

Following the representative procedure, flash chromatography (hexane/EtOAc = 1/1) gave **4d** in 52% yield as a pale yellow oil: IR (film) 2958, 2920, 2850, 1753, 1649, 1589, 1411, 1246 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.71-7.69 (m, 2H), 7.30-7.27 (m, 2H), 6.61 (q, $J = 7.0$ Hz, 1H), 4.53-4.50 (m, 2H), 3.87-3.83 (m, 2H), 2.55 (s, 3H), 1.98 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 191.4, 156.9, 145.4, 141.0, 135.8, 133.4, 129.9, 125.0, 62.8, 46.0, 14.9, 14.4; HRMS (EI, M^+) Calcd for $\text{C}_{14}\text{H}_{15}\text{NO}_3\text{S}$ 277.0773; found m/z 277.0772.



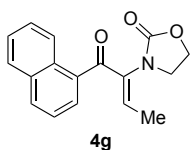
(Z)-3-(1-(4-Chlorophenyl)-1-oxobut-2-en-2-yl) oxazolidin-2-one (4e)

Following the representative procedure, flash chromatography (hexane/EtOAc = 1/1) gave **4e** in 46% yield as a colorless oil: IR (film) 3068, 2960, 2917, 1755, 1657, 1588, 1413, 1247 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.71-7.70 (m, 2H), 7.47-7.44 (m, 2H), 6.61 (q, $J = 7.0$ Hz, 1H), 4.54-4.51 (m, 2H), 3.87-3.84 (m, 2H), 1.99 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 191.3, 157.0, 142.2, 138.9, 135.9, 135.7, 130.7, 128.8, 62.9, 46.0, 14.5; HRMS (EI, M^+) Calcd for $\text{C}_{13}\text{H}_{12}\text{ClNO}_3$ 265.0506; found m/z 265.0504.



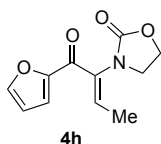
(Z)-Methyl 4-(2-(2-oxooxazolidin-3-yl) but-2-enoyl) benzoate (4f)

Following the representative procedure, flash chromatography (hexane/EtOAc = 1/1) gave **4f** in 46% yield as a colorless oil: IR (film) 2954, 2916, 1755, 1724, 1659, 1610, 1408, 1281 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 8.15-8.13 (m, 2H), 7.80-7.77 (m, 2H), 6.65 (q, $J = 7.0$ Hz, 1H), 4.56-4.53 (m, 2H), 3.98 (s, 3H), 3.90-3.87 (m, 2H), 2.00 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 191.9, 166.2, 156.9, 143.6, 141.2, 136.1, 133.3, 129.6, 129.1, 62.9, 52.5, 45.9, 14.6; HRMS (EI, M^+) Calcd for $\text{C}_{15}\text{H}_{15}\text{NO}_5$ 289.0950; found m/z 289.0949.



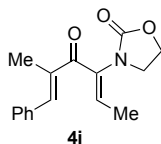
(Z)-3-(1-(Naphthalen-2-yl)-1-oxobut-2-en-2-yl) oxazolidin-2-one (4g)

Following the representative procedure, flash chromatography (hexane/EtOAc = 1/1) gave **4g** in 52% yield as a colorless oil: IR (film) 3053, 2960, 2915, 1755, 1662, 1591, 1414, 1246 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 8.12-8.01 (m, 1H), 7.98 (d, $J = 8.5$ Hz, 1H), 7.91-7.89 (m, 1H), 7.63-7.61 (m, 1H), 7.56-7.54 (m, 2H), 7.52-7.50 (m, 1H), 6.64 (q, $J = 7.0$ Hz, 1H), 4.58-4.55 (m, 2H), 3.98-3.95 (m, 2H), 1.93 (d, $J = 7.5$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 194.0, 157.2, 145.5, 138.1, 135.6, 133.6, 131.2, 130.7, 128.4, 127.4, 127.0, 126.6, 125.4, 124.3, 63.0, 46.1, 14.6; HRMS (EI, M^+) Calcd for $\text{C}_{17}\text{H}_{15}\text{NO}_3$ 281.1052; found m/z 281.1050.



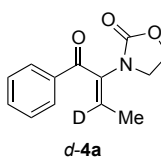
(Z)-3-(1-(Furan-2-yl)-1-oxobut-2-en-2-yl) oxazolidin-2-one (4h)

Following the representative procedure, flash chromatography (hexane/EtOAc = 1/1) gave **4h** in 33% yield as a yellow oil: IR (film) 2920, 1754, 1644, 1564, 1466, 1414, 1252 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) 7.65-7.64 (m, 1H), 7.27-7.26 (m, 1H), 7.12 (q, $J = 7.0$ Hz, 1H), 6.58-6.57 (m, 1H), 4.54-4.50 (m, 2H), 3.85-3.82 (m, 2H), 2.00 (d, $J = 7.5$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 177.4, 157.0, 151.7, 146.9, 140.9, 135.0, 119.9, 112.3, 62.8, 46.0, 14.5; HRMS (EI, M^+) Calcd for $\text{C}_{11}\text{H}_{11}\text{NO}_4$ 221.0688; found m/z 221.0688.



3-((2Z, 5E)-5-Methyl-4-oxo-6-phenylhexa-2, 5-dien-3-yl) oxazolidin-2-one (4i)

Following the representative procedure, flash chromatography (hexane/EtOAc = 1/1) gave **4i** in 35% yield as a colorless oil: IR (neat) 3056, 2985, 2917, 1757, 1642, 1575, 1411, 1243 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.46-7.42 (m, 4H), 7.38-7.36 (m, 1H), 7.29 (s, 1H), 6.67 (q, $J = 7.0$ Hz, 1H), 4.54-4.50 (m, 2H), 3.87-3.84 (m, 2H), 2.18 (br s, 3H), 1.97 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 194.8, 157.1, 139.9, 139.4, 136.2, 135.7, 135.5, 129.7, 128.6, 128.5, 62.9, 46.1, 14.6, 14.3; HRMS (EI, M^+) Calcd for $\text{C}_{16}\text{H}_{17}\text{NO}_3$ 271.1209; found m/z 271.1205.



Deuterated 4a

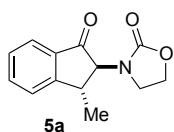
Following the representative procedure, flash chromatography (hexane/EtOAc = 1/1) gave **d-4a** in 44% yield as colorless oil: IR (film) 3060, 2958, 2924, 2854, 1756, 1656, 1598, 1411, 1250 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.74-7.70 (m, 2H), 7.58-7.53 (m, 1H), 7.47-7.42 (m, 2H), 4.52-4.48 (m, 2H), 3.86-3.83 (m, 2H), 1.96 (s, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 192.4, 156.9, 142.1 (t, $J_{\text{CD}} = 23.9$ Hz), 137.5, 135.9, 132.4, 129.3, 128.4, 62.8, 45.9, 14.4; HRMS (ESI, $[\text{M}+\text{Na}]^+$) Calcd for $\text{C}_{13}\text{H}_{12}\text{DNNaO}_3$ 255.0850; found m/z 255.0848.

Standard Procedure for the Synthesis of 5:

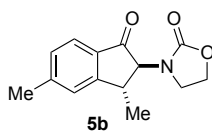
To a stirred solution of **4** (0.1 mmole) in 1,2-dichloroethane (10 mL) at the indicated temperature

was added Lewis or Brønsted acid (reagent and equivalent specified below) under argon atmosphere. After starting material was completely consumed as monitored by TLC, the reaction was quenched with sat. aq NaHCO₃ (5 mL). The organic layer was separated and the aqueous layer was extracted with dichloromethane (2 x 5 mL). The combined organic layer was washed with brine, dried over anhydrous MgSO₄, and then filtered through a pad of cellite. The solvent was removed under reduced pressure and the residue was purified on a silica gel column to afford the desired product.

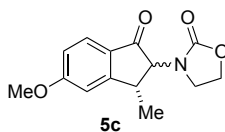
Characterization Data for 5a-i



Following the representative procedure using 5 equiv triflic acid at 65 °C (oil bath), flash chromatography (hexane/EtOAc = 1/1) gave **5a** in 98% yield (cis:trans >20:1): IR (film) 2963, 2924, 1748, 1722, 1607, 1429, 1261, 1207 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 7.81-7.79 (m, 1H), 7.75-7.71 (m, 1H), 7.57-7.55 (m, 1H), 7.49-7.46 (m, 1H), 4.50 (ddd, *J* = 8.5, 8.5, 7.0 Hz, 1H), 4.45 (ddd, *J* = 9.0, 9.0, 7.0 Hz, 1H), 4.42 (d, *J* = 6.5 Hz, 1H), 3.67 (ddd, *J* = 9.0, 8.0, 7.0 Hz, 1H), 3.52 (ddd, *J* = 9.0, 8.0, 7.0 Hz, 1H), 3.36 (app. quintet, *J* = 6.5 Hz, 1H), 1.64 (d, *J* = 6.5 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 199.9, 158.9, 154.8, 135.9, 134.3, 128.2, 124.9, 124.0, 67.0, 62.3, 41.9, 36.6, 17.7; HRMS (EI, M⁺) Calcd for C₁₃H₁₃NO₃ 231.0895; found *m/z* 231.0900.

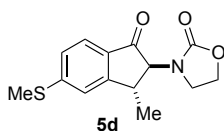


Following the representative procedure using 5 equiv triflic acid at 65 °C (oil bath), flash chromatography (hexane/EtOAc = 1/1) gave **5b** in 87% yield (cis:trans >20:1); IR (film) 3018, 2966, 2919, 1749, 1719, 1611, 1430, 1263, 1216 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 7.66 (d, *J* = 8.5 Hz, 1H), 7.31 (br s, 1H), 7.24 (d, *J* = 8.5 Hz, 1H), 4.48-4.39 (m, 2H), 4.37 (d, *J* = 6.0 Hz, 1H), 3.62 (ddd, *J* = 9.5, 8.0, 7.0 Hz, 1H), 3.46 (ddd, *J* = 9.5, 8.0, 7.0 Hz, 1H), 3.26 (app. quintet, *J* = 6.5 Hz, 1H), 2.47 (s, 3H), 1.58 (d, *J* = 6.5 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 199.3, 159.0, 155.3, 147.4, 132.0, 129.4, 125.3, 123.8, 67.0, 62.3, 41.8, 36.5, 22.3, 17.6; HRMS (ESI, [M+Na]⁺) Calcd for C₁₄H₁₅NNaO₃ 268.0944; found *m/z* 268.0941.

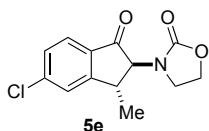


Following the representative procedure using 5 equiv triflic acid at 65 °C (oil bath), flash chromatography (hexane/EtOAc = 1/1) gave **5c** in 88% yield (cis:trans 1:4, inseparable mixture): IR

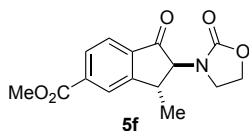
(film) 3019, 2969, 2917, 1750, 1713, 1601, 1429, 1263, 1217 cm^{-1} ; trans isomer: ^1H NMR (500 MHz, CDCl_3) δ 7.70 (d, $J = 8.5$ Hz, 1H), 6.94 (dd, $J = 8.5, 2.0$ Hz, 1H), 6.92 (br s, 1H), 4.48-4.37 (m, 2H), 4.36 (d, $J = 6.0$ Hz, 1H), 3.91 (s, 3H), 3.64-3.59 (m, 1H), 3.48-3.43 (m, 1H), 3.25 (app. quintet, $J = 7.0$ Hz, 1H), 1.57 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 197.9, 166.3, 158.9, 157.9, 127.6, 125.9, 115.8, 108.5, 66.8, 62.2, 55.8, 41.8, 36.7, 17.7; cis isomer: ^1H NMR (500 MHz, CDCl_3) δ 7.72 (d, $J = 8.5$ Hz, 1H), 6.94 (dd, $J = 8.5, 2.5$ Hz, 1H), 6.9 (d, $J = 2.5$ Hz, 1H), 4.81 (d, $J = 7.5$ Hz, 1H), 4.45-4.35 (m, 2H), 4.04 (ddd, $J = 7.5, 7.5, 7.0$ Hz, 1H), 3.91 (s, 3H), 3.78 (app. quintet, $J = 8.0$ Hz, 1H), 3.39 (ddd, $J = 8.5, 8.5, 7.0$ Hz, 1H), 1.28 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 198.3, 166.3, 160.6, 159.2, 126.9, 126.2, 116.2, 109.1, 62.7, 62.0, 43.5, 37.8, 17.6 [one aliphatic carbon is missing due to peak overlapping.]; HRMS (ESI, $[\text{M}+\text{Na}]^+$) Calcd for $\text{C}_{14}\text{H}_{15}\text{NNaO}_4$ 284.0893; found m/z 284.0889.



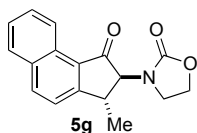
Following the representative procedure using 5 equiv triflic acid at 65 $^{\circ}\text{C}$ (oil bath), flash chromatography (hexane/EtOAc = 1/1) gave **5d** in 97% yield (cis:trans >20:1): IR (film) 3058, 2965, 2924, 1747, 1713, 1594, 1431, 1262, 1198 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.68 (d, $J = 8.5$ Hz, 1H), 7.31 (br s, 1H), 7.28 (d, $J = 8.5$ Hz, 1H), 4.49 (ddd, $J = 8.5, 8.5, 6.5$ Hz, 1H), 4.44 (ddd, $J = 9.0, 9.0, 6.5$ Hz, 1H), 4.41 (d, $J = 6.0$ Hz, 1H), 3.65 (ddd, $J = 9.5, 8.0, 6.5$ Hz, 1H), 3.51 (ddd, $J = 9.0, 8.0, 7.0$ Hz, 1H), 3.30 (app. quintet, $J = 6.5$ Hz, 1H), 2.59 (s, 3H), 1.62 (d, $J = 6.5$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 198.5, 158.9, 155.4, 150.1, 130.9, 125.1, 124.0, 120.4, 66.9, 62.3, 41.9, 36.6, 17.6, 14.9; HRMS (ESI, $[\text{M}+\text{Na}]^+$) Calcd for $\text{C}_{14}\text{H}_{15}\text{NNaO}_3\text{S}$ 300.0665; found m/z 300.0660.



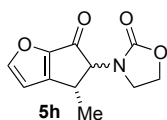
Following the representative procedure using 5 equiv triflic acid at 65 $^{\circ}\text{C}$ (oil bath), flash chromatography (hexane/EtOAc = 1/1) gave **5e** in 94% yield (cis:trans >20:1): IR (film) 3061, 2965, 2923, 1748, 1725, 1600, 1431, 1259, 1210 cm^{-1} ; ^1H NMR (500 MHz, CDCl_3) δ 7.70 (d, $J = 8.5$ Hz, 1H), 7.51 (br s, 1H), 7.42 (d, $J = 8.5$ Hz, 1H), 4.47 (ddd, $J = 9.0, 9.0, 7.0$ Hz, 1H), 4.42 (ddd, $J = 8.5, 8.5, 7.0$ Hz, 1H), 4.36 (d, $J = 6.5$ Hz, 1H), 3.66-3.61 (m, 1H), 3.53-3.48 (m, 1H), 3.32 (app. quintet, $J = 6.5$ Hz, 1H), 1.59 (d, $J = 7.0$ Hz, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 198.5, 158.8, 156.2, 142.6, 132.7, 129.0, 125.4, 125.2, 67.0, 62.3, 42.1, 36.6, 17.5; HRMS (ESI, $[\text{M}+\text{Na}]^+$) Calcd for $\text{C}_{13}\text{H}_{12}\text{ClNNaO}_3$ 288.0398; found m/z 288.0395.



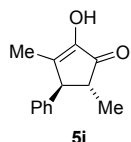
Following the representative procedure using 5 equiv triflic acid at 65 °C (oil bath), flash chromatography (hexane/EtOAc = 1/1) gave **5f** in 91% yield (cis:trans >20:1): IR (film) 3020, 2955, 2927, 1751, 1726, 1615, 1436, 1262, 1216 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 8.23 (br s, 1H), 8.14-8.12 (m, 1H), 7.86-7.85 (m, 1H), 4.55-4.46 (m, 2H), 4.44 (d, *J* = 6.5 Hz, 1H), 4.01 (s, 3H), 3.69 (ddd, *J* = 9.0, 8.0, 7.0 Hz, 1H), 3.56 (ddd, *J* = 9.0, 8.0, 7.0 Hz, 1H), 3.41 (app. quintet, *J* = 7.0 Hz, 1H), 1.68 (d, *J* = 7.0 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 199.6, 166.0, 158.8, 154.5, 137.4, 136.6, 129.3, 126.3, 123.9, 67.4, 62.3, 52.7, 42.2, 36.6, 17.6; HRMS (ESI, [M+Na]⁺) Calcd for C₁₅H₁₅NNaO₅ 312.0842; found *m/z* 312.0841.



Following the representative procedure using 5 equiv triflic acid at 65 °C (oil bath), flash chromatography (hexane/EtOAc = 1/1) gave **5g** in 97% yield (cis:trans >20:1): IR (film) 3014, 2966, 2925, 1744, 1704, 1628, 1428, 1269, 1219 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 9.07 (d, *J* = 8.5 Hz, 1H), 8.20 (d, *J* = 8.5 Hz, 1H), 7.96-7.94 (m, 1H), 7.76-7.73 (m, 1H), 7.65-7.61 (m, 2H), 4.55-4.44 (m, 2H), 4.54 (d, *J* = 5.5 Hz, 1H), 3.71 (ddd, *J* = 9.0, 8.0, 7.0, 1H), 3.55 (ddd, *J* = 9.5, 7.5, 6.5 Hz, 1H), 3.46 (app. quintet, *J* = 6.0 Hz, 1H), 1.70 (d, *J* = 7.0 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 200.2, 159.1, 157.8, 137.1, 132.8, 129.6, 128.9, 128.8, 128.3, 127.2, 124.3, 121.8, 67.1, 62.3, 42.0, 36.8, 17.9; HRMS (EI, M⁺) Calcd for C₁₇H₁₅NO₃ 281.1052; found *m/z* 281.1049.



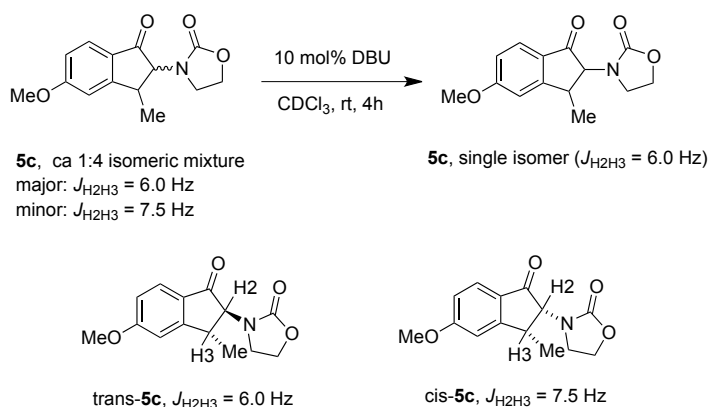
Following the representative procedure using 5 equiv triflic acid at 65 °C (oil bath), flash chromatography (hexane/EtOAc = 1/1) gave **5h** in 65% yield (cis:trans 1:3, inseparable mixture): IR (film) 3059, 2972, 2918, 1749, 1715, 1579, 1426, 1268, 1246 cm⁻¹; trans isomer: ¹H NMR (500 MHz, CDCl₃) δ 7.87 (d, *J* = 2.0 Hz, 1H), 6.59 (d, *J* = 2.0 Hz, 1H), 4.52 (d, *J* = 3.5 Hz, 1H), 4.50-4.40 (m, 2H), 3.66-3.61 (m, 1H), 3.57-3.52 (m, 1H), 3.21 (qd, *J* = 7.5, 3.5 Hz, 1H), 1.53 (d, *J* = 7.5 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 182.3, 158.5, 157.6, 154.5, 152.0, 109.2, 70.1, 62.2, 42.2, 33.1, 18.0; cis isomer: ¹H NMR (500 MHz, CDCl₃) δ 7.87 (d, *J* = 2.0 Hz, 1H), 6.60 (d, *J* = 2.0 Hz, 1H), 4.50-4.40 (m, 3H), 4.00-3.95 (m, 1H), 3.69 (app. quintet, *J* = 7.0 Hz, 1H), 3.52-3.47 (m, 1H), 1.31 (d, *J* = 7.5 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 181.9, 160.0, 158.7, 154.6, 151.8, 109.6, 65.3, 62.6, 42.1, 33.0, 16.1; HRMS (ESI, [M+Na]⁺) Calcd for C₁₁H₁₁NNaO₄ 244.0580; found *m/z* 244.0583.



Following the representative procedure using 20 mol% Sc(OTf)₃ at 80 °C (oil bath), flash chromatography (hexane/EtOAc = 1/1) gave **5i** in 96% yield: IR (film) 3336 (br), 3027, 2977, 1700, 1656, 1600, 1406, 1360, 1206 cm⁻¹; ¹H NMR (500 MHz, CDCl₃) δ 7.38-7.35 (m, 2H), 7.32-7.29 (m, 1H), 7.16-7.14 (m, 2H), 5.37 (br s, 1H), 3.32 (app. quintet, *J* = 1.9 Hz, 1H), 2.32 (qdd, *J* = 8.0, 2.2, 0.8 Hz, 1H), 1.82 (dd, *J* = 2.3, 0.8 Hz, 3H), 1.29 (d, *J* = 7.5 Hz, 3H); ¹³C NMR (125 MHz, CDCl₃) δ 204.3, 148.2, 143.3, 141.2, 128.9, 127.5, 127.2, 54.7, 48.6, 15.0, 12.5; HRMS (EI, M⁺) Calcd for C₁₃H₁₄O₂ 202.0994; found *m/z* 202.0995.

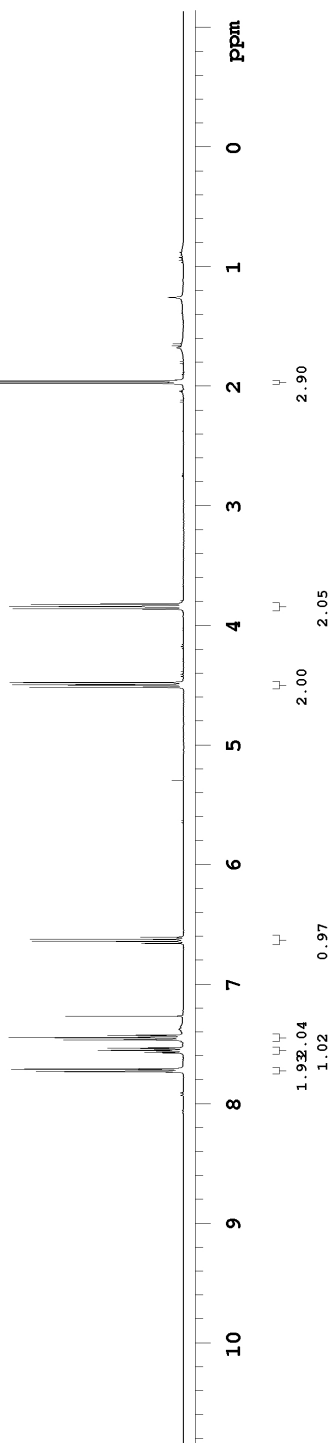
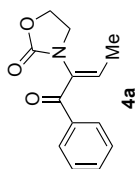
Stereochemical Assignment for Nazarov Products **5a-5h**

The trans isomers of 2,3-substituted-1-indanone were generally considered as thermodynamically favored products over the cis counterpart. In the case of **5c** obtained as isomeric products (ratio ca 1:4), the coupling constant (*J*) between H-2 and H-3 for the major isomer is 6.0 Hz and for the minor isomer is 7.5 Hz. That mixture was subjected to 10 mol% DBU in CDCl₃ (0.01M in **5c**) at room temperature, where the epimerization was envisioned to be guided by the thermodynamic control. In the event, the minor isomer was no longer detected in the proton NMR after 4h; therefore the disappearing isomer was assigned as cis-**5c** and the predominant isomer as trans-**5c**. With that, we presumed that *J*_{H₂H₃} for trans isomers of closely related indanones would be similar to the value of 6.0 Hz. Based on the evaluation of *J*_{H₂H₃}, **5a**, **5b**, **5c-g** were referred to as trans. Similar approach was then applied to the stereochemical assignment for heteroaromatic substrate **5h** isolated as a ca 1:3 isomeric mixture, and the trans isomer could be readily assigned, whose *J*_{H₂H₃} equals 3.5 Hz.

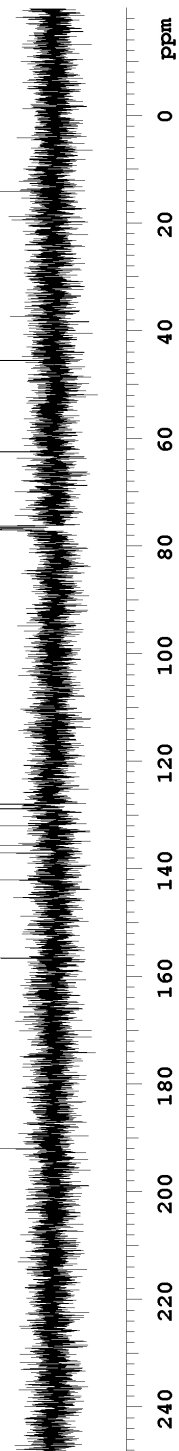
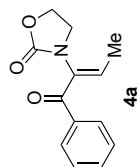


^1H and ^{13}C NMR Spectra

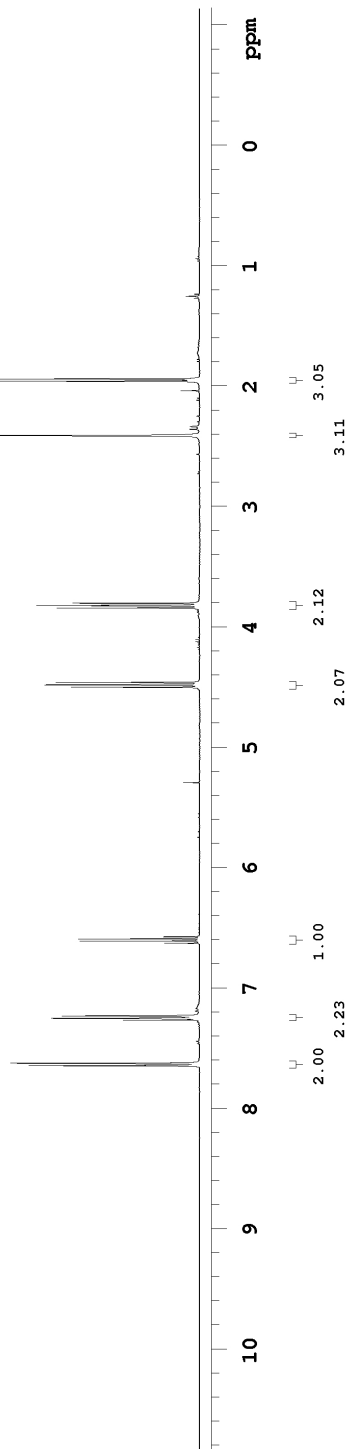
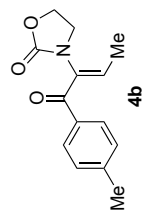
399.794 MHz H1 ID in cdcl3 (ref. to CDC13 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, autoxdb probe
date: Sep 13 2011 sweep width: 4799Hz acq.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt hz/mm:20.0 Pulse sequence: s2pul
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spectrometer:ibdw



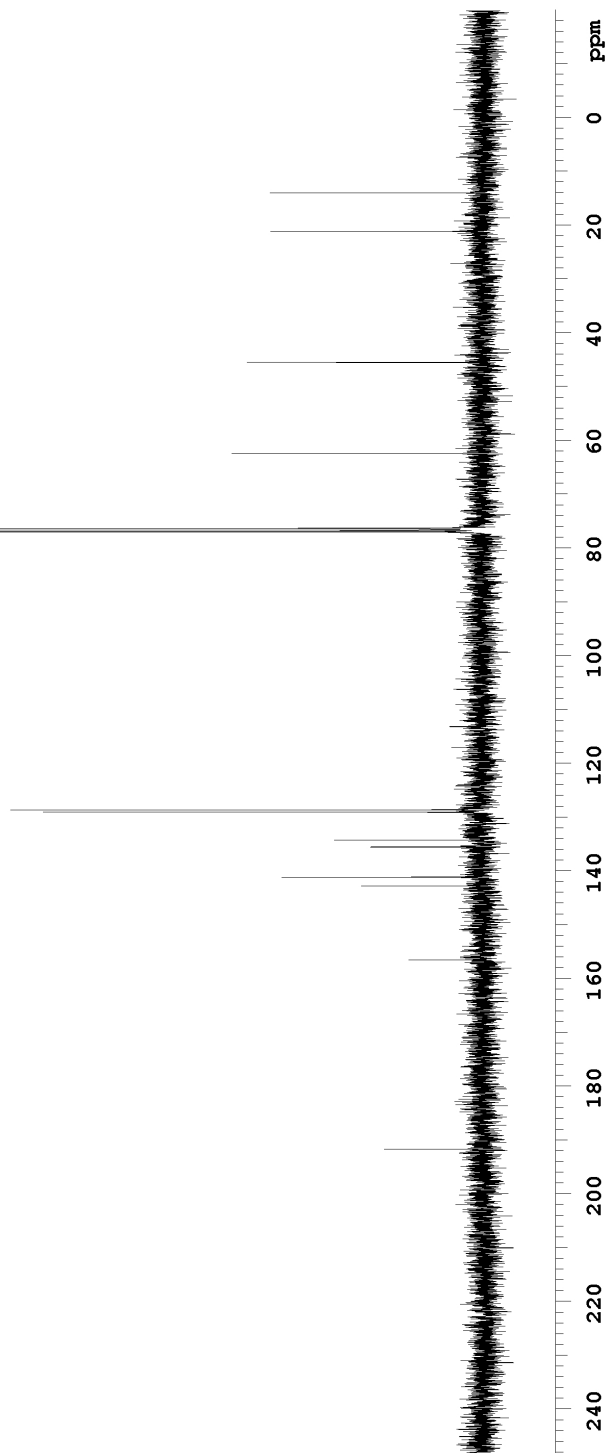
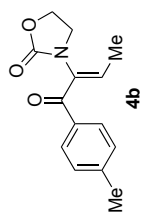
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date: Sep 13 2011 sweep width: 26991Hz acq.time: 2.5s relax.time: 0.1s # scans: 56 dig.res.: 0.2 Hz/pt
spectrometer:ibdw file://mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmin/4a-13C
hz/mm:112.5 Pulse Sequence: s2pul



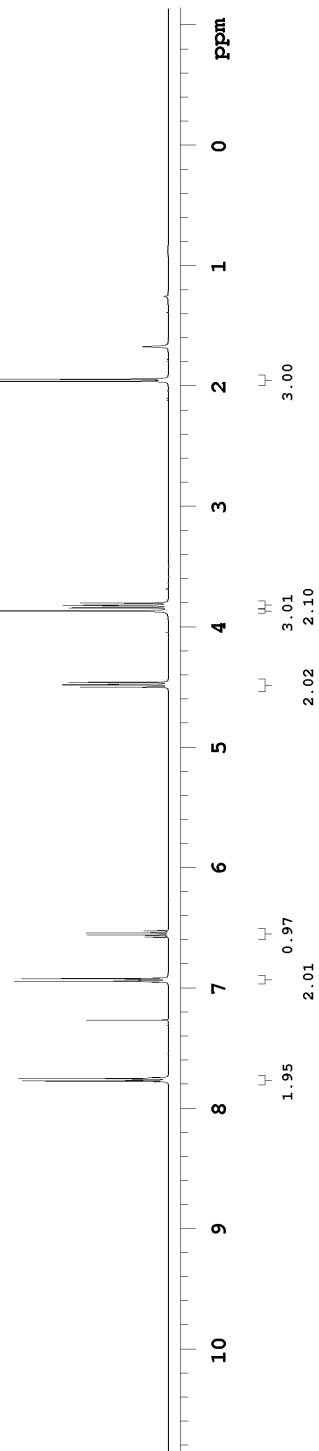
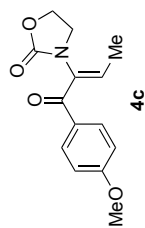
399.794 MHz H1 ID in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, autoxdb probe
date: Sep 13 2011 sweep width: 4799Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt
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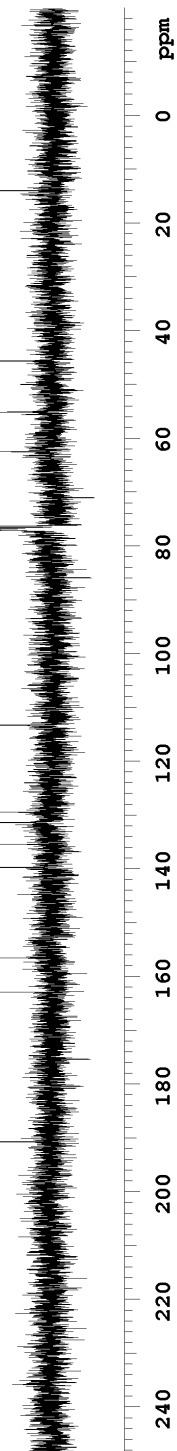
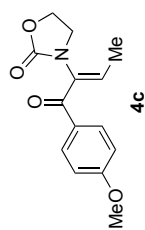
100.537 MHz C13[HL] 1D in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.0 C -> actual temp = 27.0 C, autoxdb probe
date: Sep 13 2011 sweep width: 26991Hz acq.time: 2.5s relax.time: 0.1s # scans: 44 dig.res.: 0.2 Hz/pt
spectrometer:ibdw file://mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmin/4b-13C
hz/mm:112.5 Pulse Sequence: s2pul



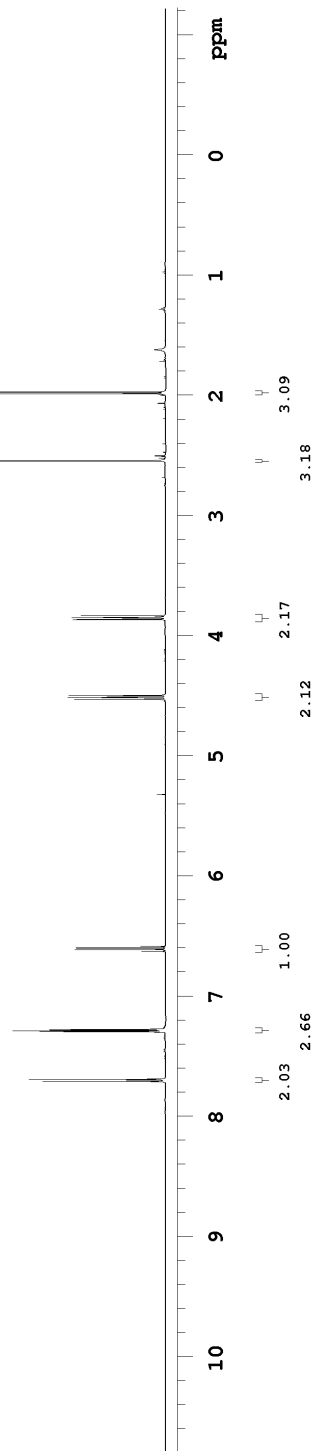
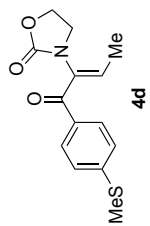
399.794 MHz H1 ID in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.0 C -> actual temp = 27.0 C, autoxdb probe
date: Oct 3 2011 sweep width: 4799Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt
spectrometer:ibdw file://mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmin/4c-1H



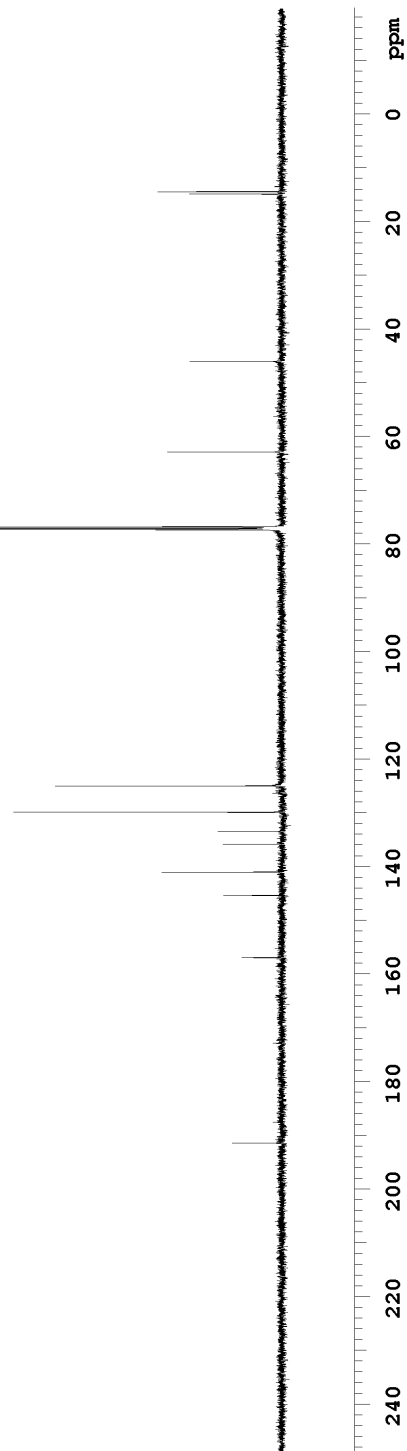
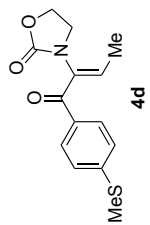
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date: Oct 3 2011 sweep width: 26991Hz acq.time: 2.5s relax.time: 0.1s # scans: 116 dig.res.: 0.2 Hz/pt
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hz/mm:112.5 Pulse Sequence: s2pul



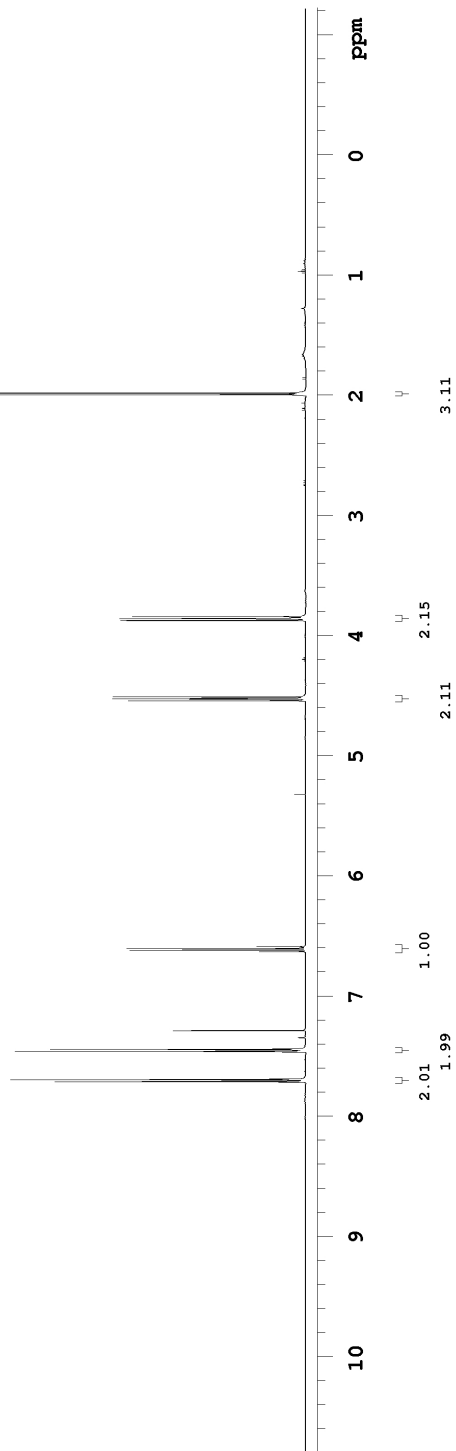
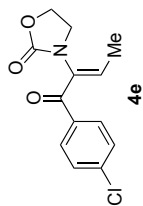
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date: Sep 14 2011 sweep width: 6010Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt
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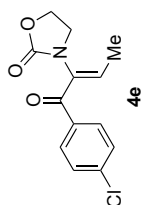
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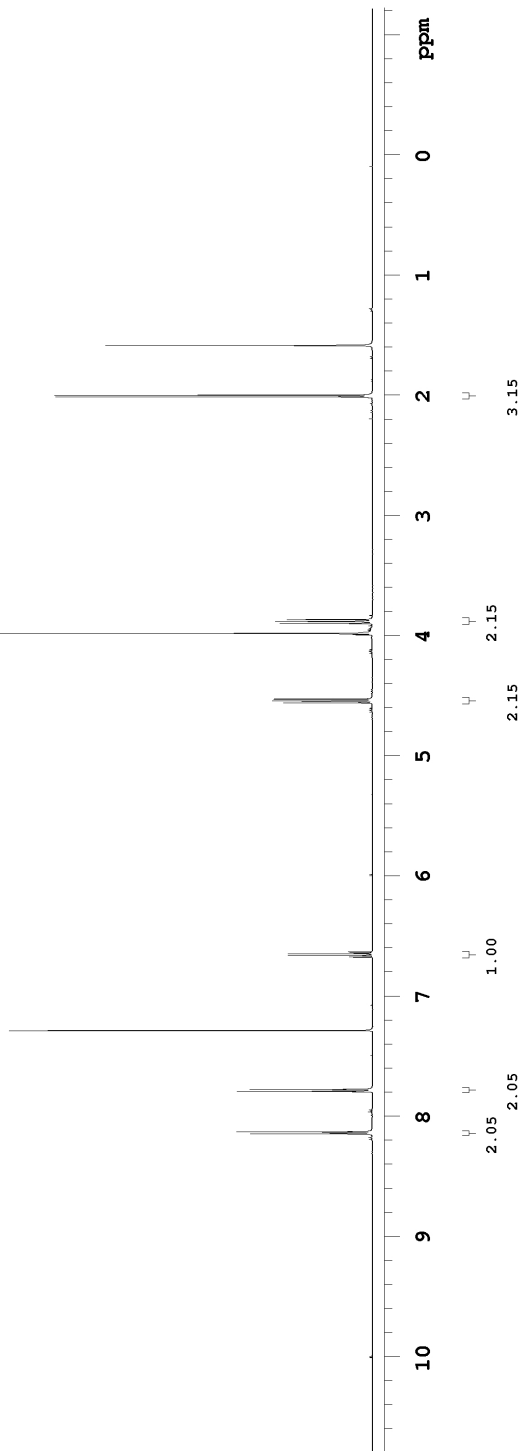
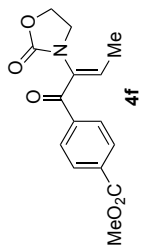
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date: Sep 14 2011 sweep width: 6010Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt
spectrometer:ibdw file://mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmin/4e-1H
hz/mm:25.0 Pulse Sequence: s2pul



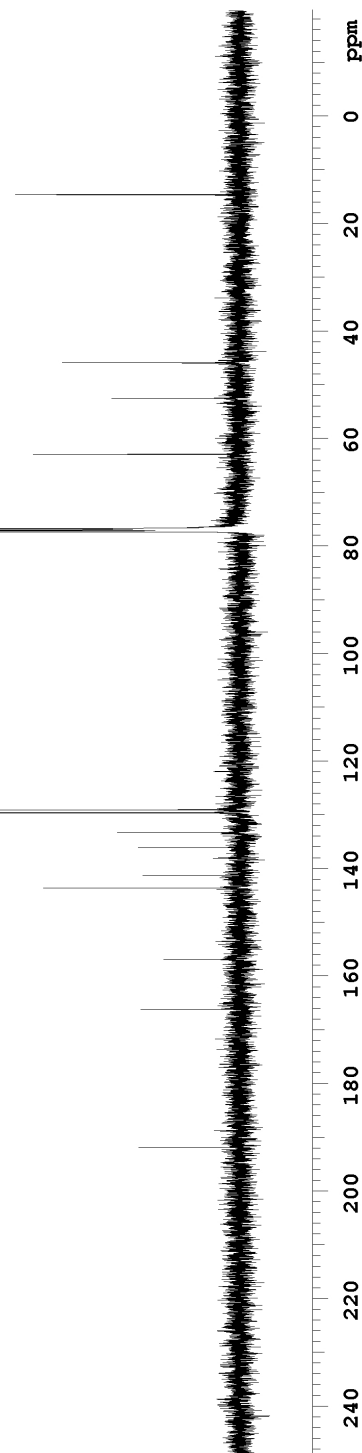
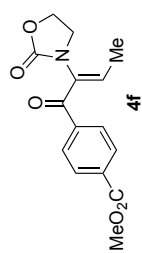
125.690 MHz C13[HL] ID in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, coldddual probe
date: Sep 14 2011 sweep width: 33784Hz acq.time: 2.5s relax.time: 0.1s # scans: 8 dig.res.: 0.3 Hz/pt
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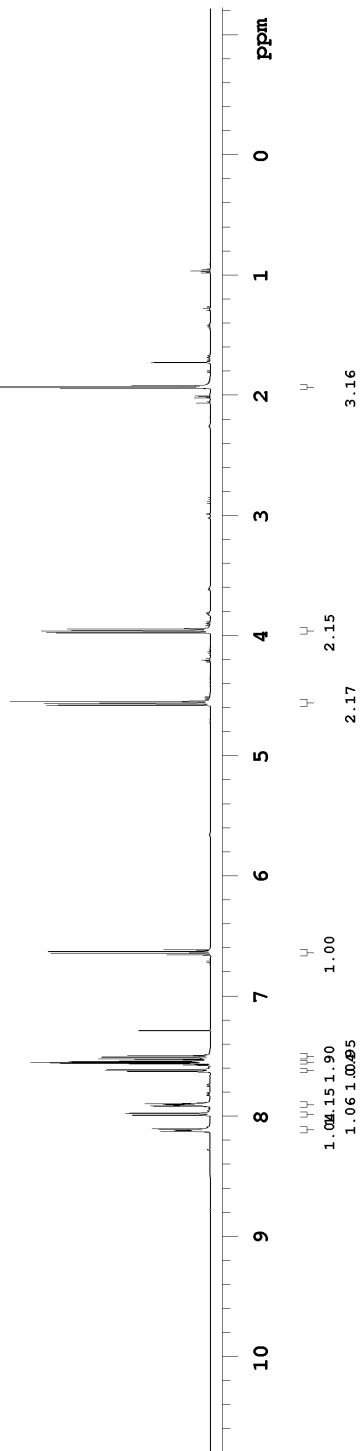
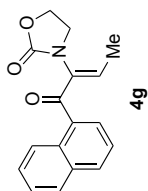
499.815 MHz H1 ID in cdcl3 (ref. to cdcl3 @ 7.26 ppm), temp 27.7 C -> actual temp = 27.0 C, coldlual probe
date: Sep 21 2011 sweep width: 6010Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt
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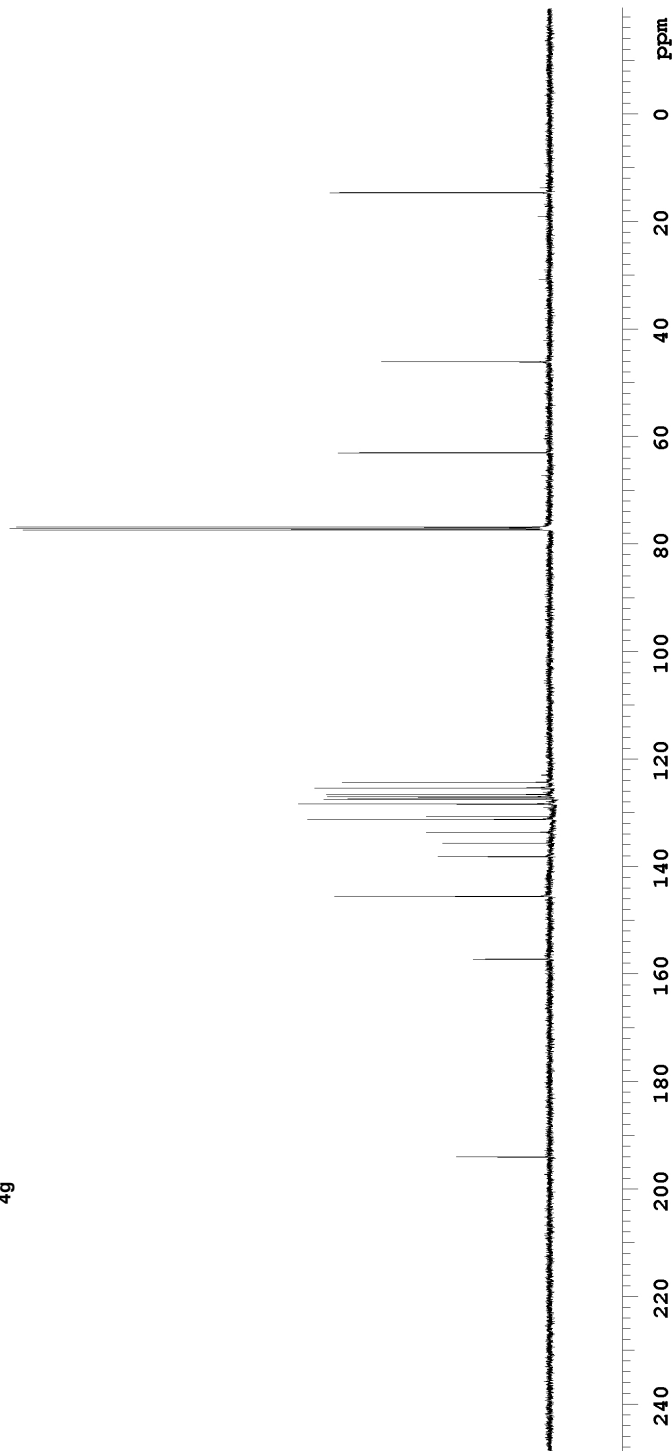
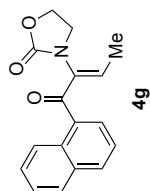
125.690 MHz C13[HL] ID in cdcl3 (ref. to cdcl3 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, coldddual probe
date: Sep 21 2011 sweep width: 33784Hz acq.time: 2.5s relax.time: 0.1s # scans: 76 dig.res.: 0.3 Hz/pt
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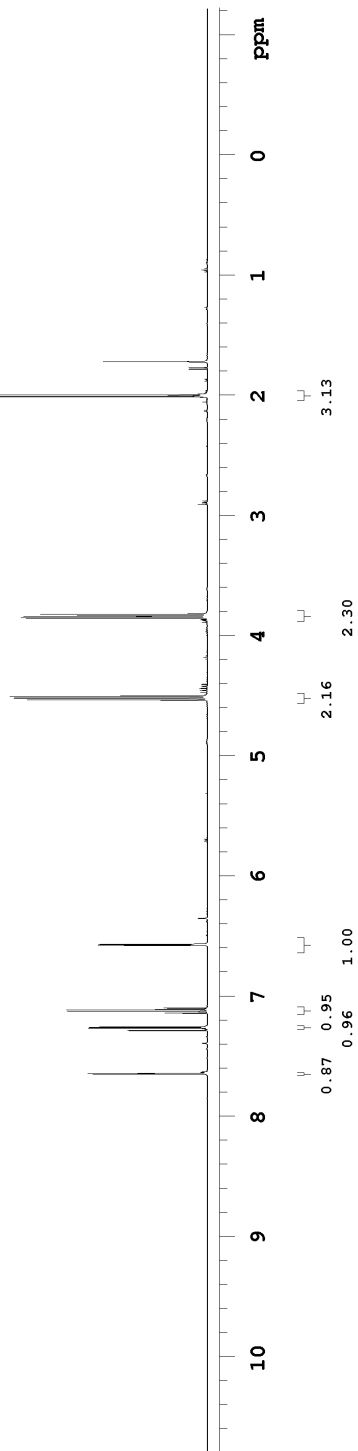
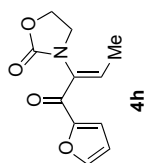
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date: Sep 30 2011 sweep width: 6010Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt
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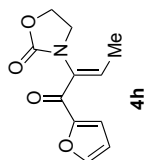
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date: Sep 30 2011 sweep width: 33784Hz acq.time: 2.5s relax.time: 0.1s # scans: 20 dig.res.: 0.3 Hz/pt
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hz/mm:140.8 Pulse Sequence: s2pul



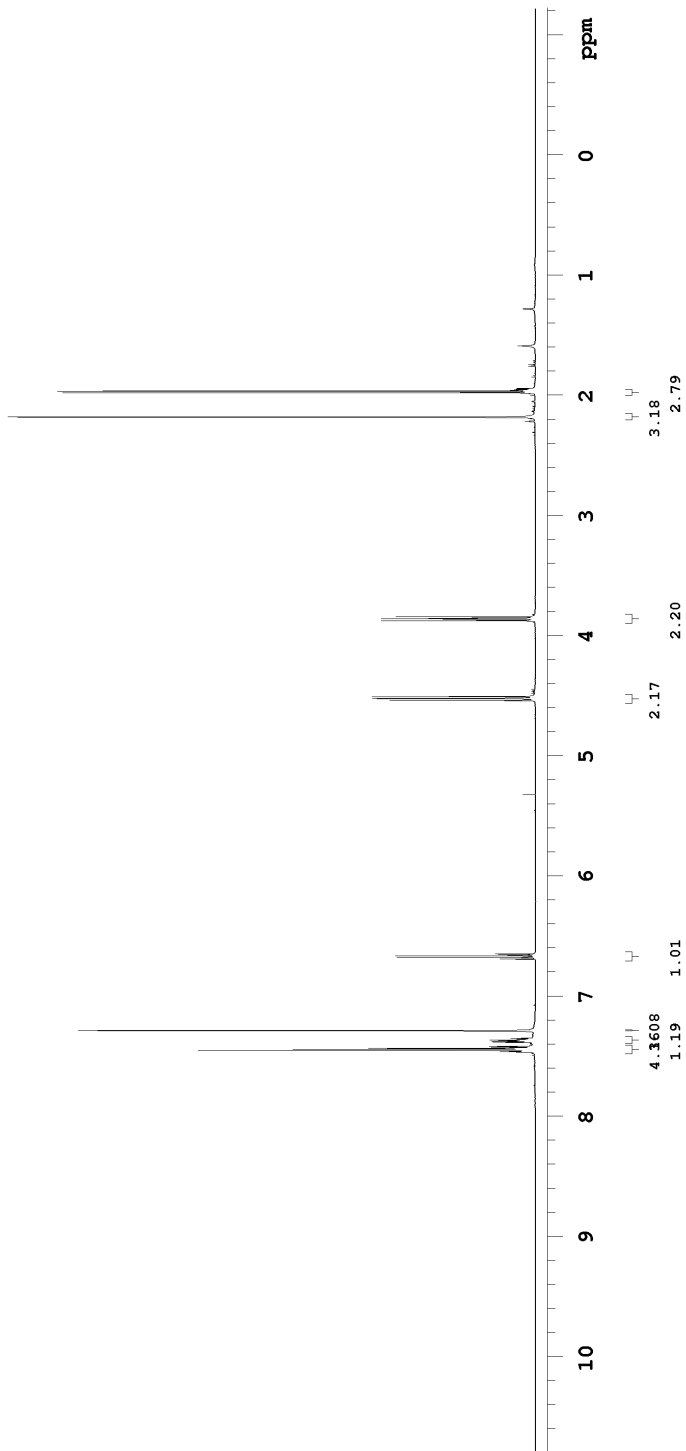
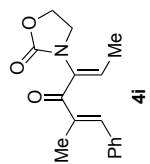
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date: Oct 5 2011 sweep width: 6010Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt
spectrometer:ibdw file://mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmin/4h-1H



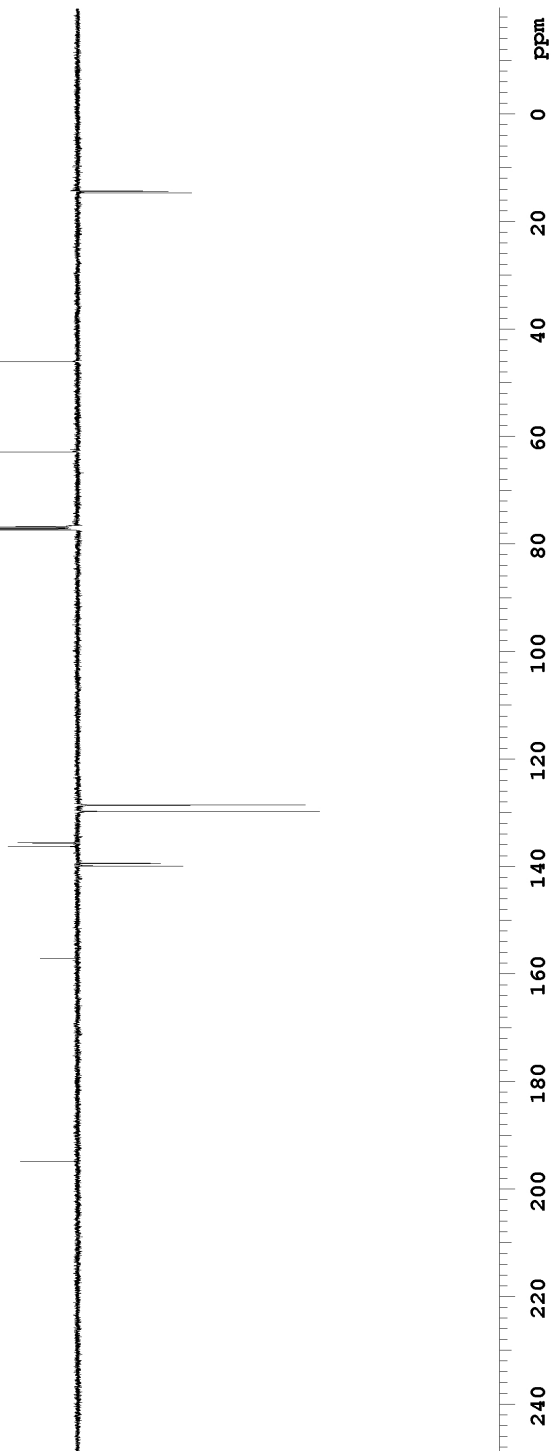
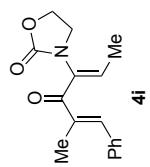
125.690 MHz C13[HL] ID in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, coldddual probe
date: Oct 5 2011 sweep width: 33784Hz acq.time: 2.5s relax.time: 0.1s # scans: 12 dig.res.: 0.3 Hz/pt
spectrometer:ibdw file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmin/4h-13C



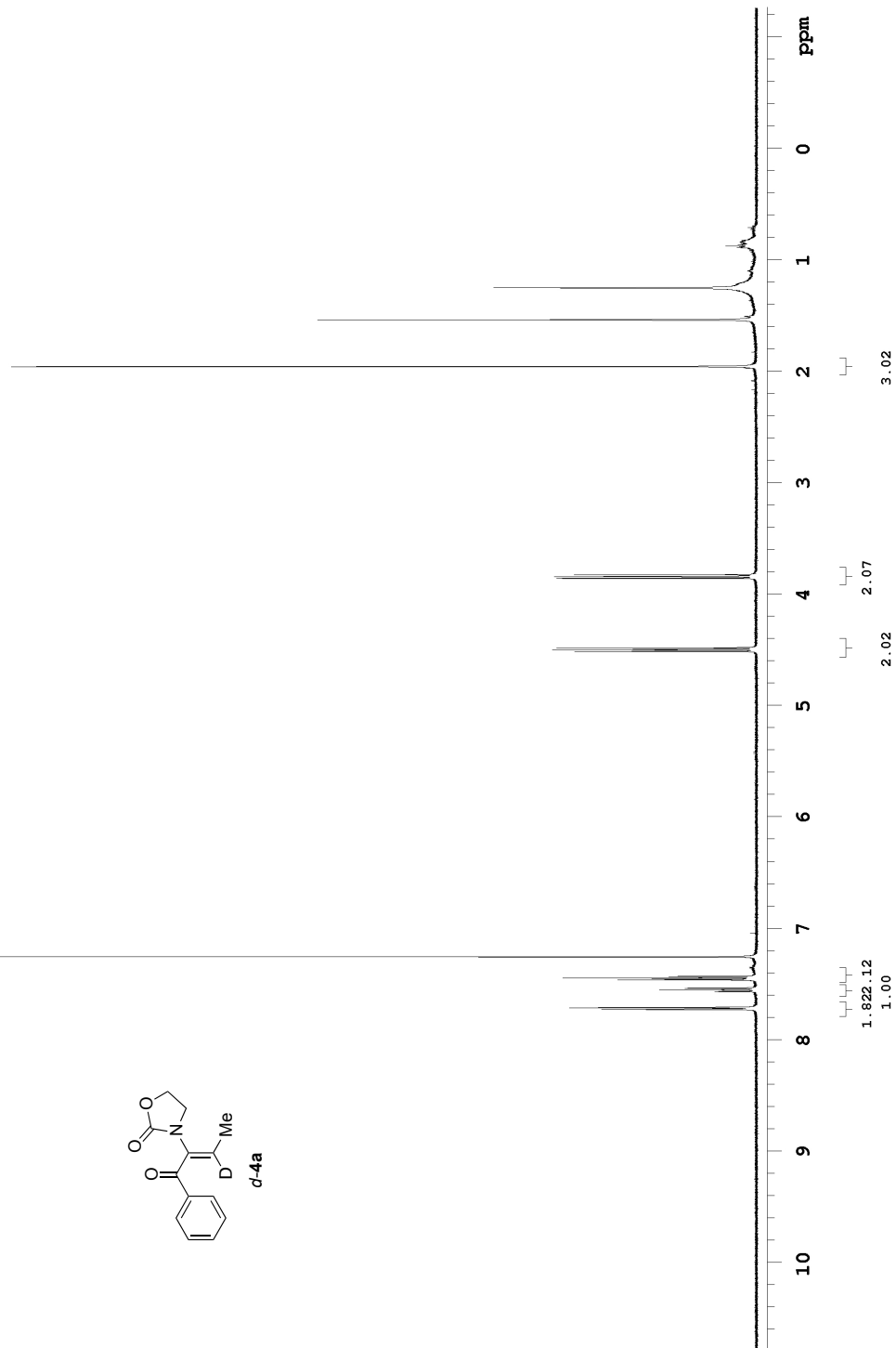
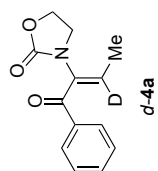
499.815 MHz H1 ID in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.7 C -> actual temp = 27.0 C, coldlual probe
date: Feb 24 2011 sweep width: 6010Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt
spectrometer:ibdw file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmin/41-1H
hz/mm:25.0 Pulse Sequence: s2pul



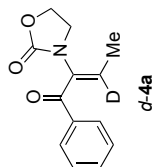
125.692 Mhz C13[HL] APT_ad in cdc13 (ref. to CDC13 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, coldludal probe
date: 2011-08-28, time: 10:28:49, file: /mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmin/41-13C
relax.time: 0.1s # scans: 60 dig.res.: 0.3 Hz/pt
spectrometer: ibdw Pulse Sequence: APT_ad



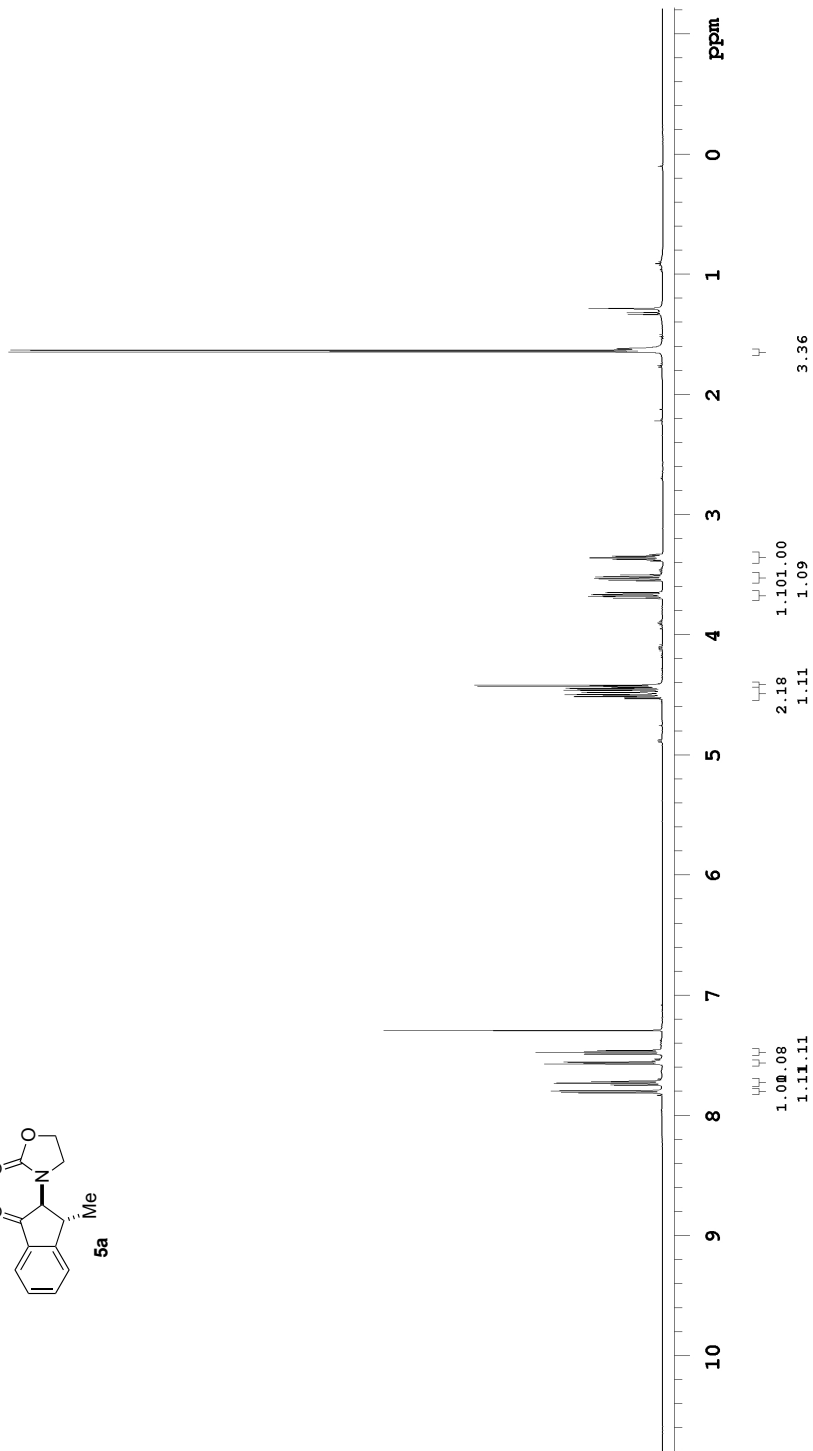
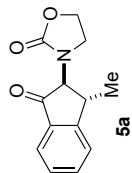
498.122 MHz H1 ID in cdcl3 (ref. to cdcl3 @ 7.26 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe
date: Dec 23 2011 sweep width: 6001Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt
spectrometer:ibdw file://mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmin/d-4a-1H
hz/mm:25.0 Pulse Sequence: s2pul



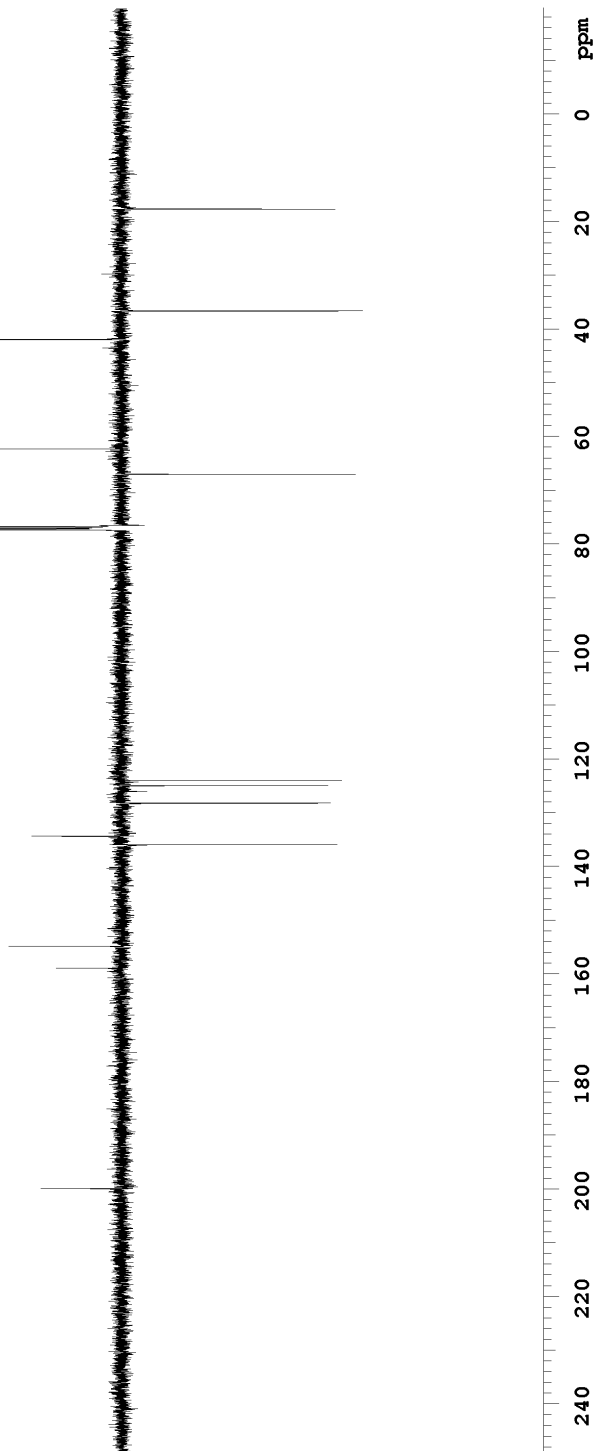
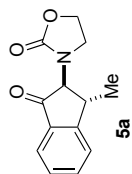
125.690 MHz C13[HL] ID in cdcl3 (ref. to CDCl3 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, coldddual probe
date: Dec 22 2011 sweep width: 33784Hz acq.time: 2.5s relax.time: 0.1s # scans: 18136 dig.res.: 0.3 Hz/pt
spectrometer:ibdw file://mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmin/d-4a-13C
hz/mm:140.8 Pulse Sequence: s2pul



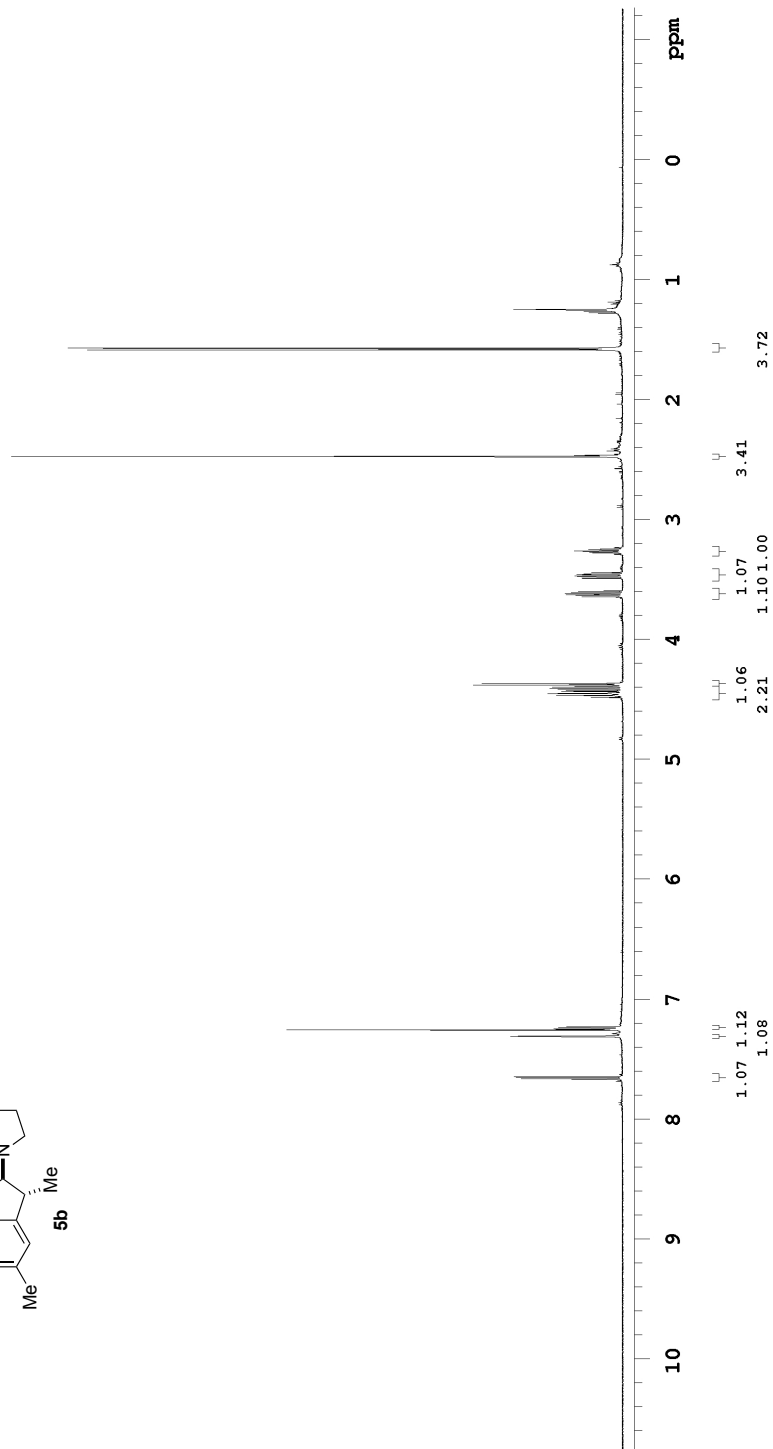
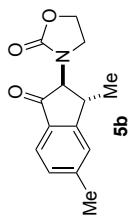
499.815 MHz H1 ID in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.7 C -> actual temp = 27.0 C, coldlial probe
date: Apr 26 2012 sweep width: 6010Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt
spectrometer:ibdw file://mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmin/5a-1H



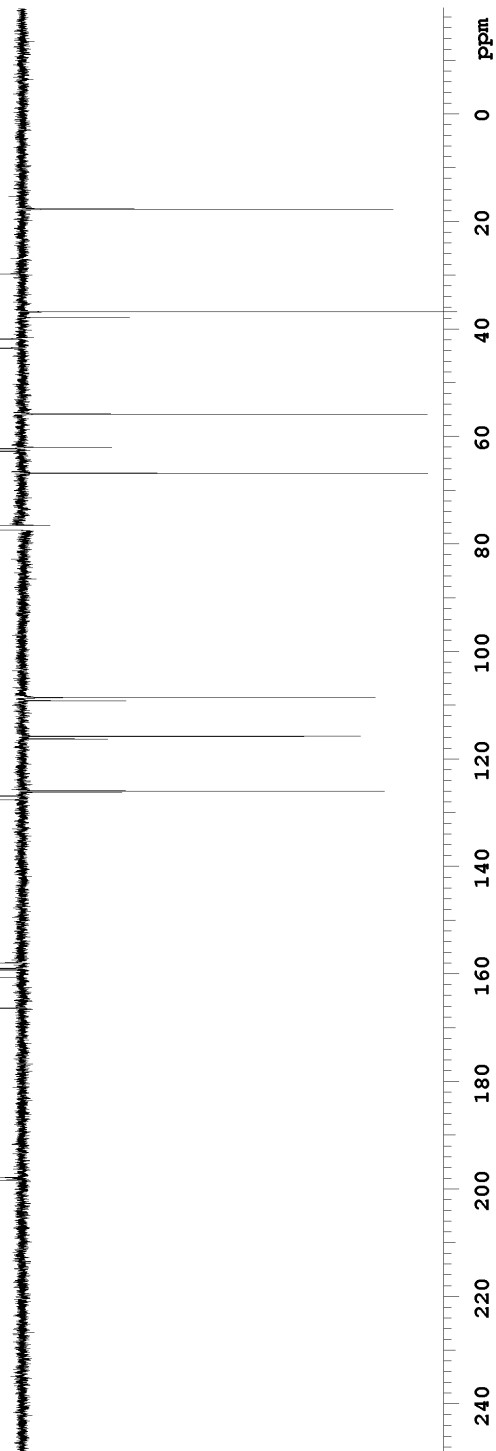
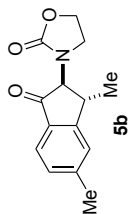
125.692 MHz C13[HL] APT_ad in cdc13 (ref. to CDC13 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, coldludal probe
dateCHNPSM: 2012.04.25 # scans: 60 dig.res.: 0.3 Hz/pt relax.time: 0.1s # scans: 60 dig.res.: 0.3 Hz/pt
spectrometer:ibdw file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmin/2012.04.25.u5_YKW-8-111_C13_apt_apt_ad



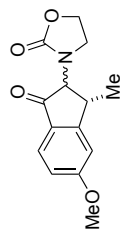
498.122 MHz H1 ID in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe
date: Apr 2 2012 sweep width: 6001Hz acq.time: 5.0s relax.time: 0.1s # scans: 12 dig.res.: 0.1 Hz/pt
spectrometer:ibdw file://mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmin/5b-1H



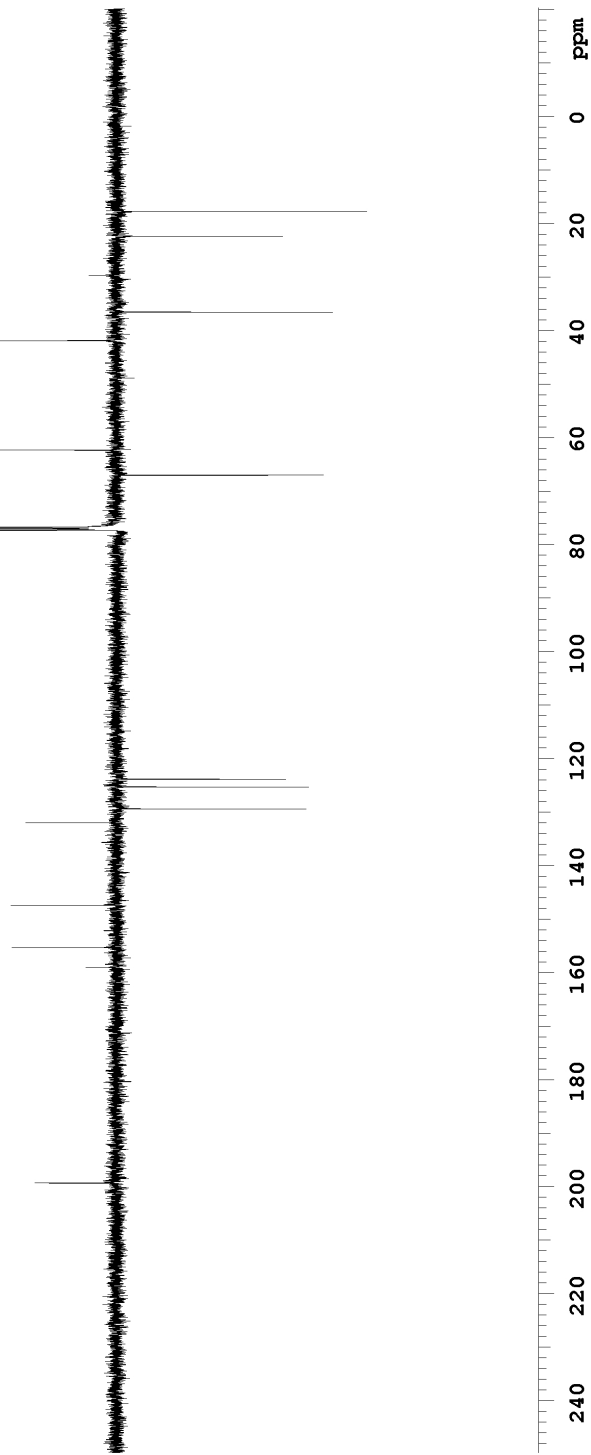
125.692 MHz C13[HL] APT_ad in cdc13 (ref. to CDC13 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, coldludal probe
date: 20120824, time: 20:28:49, file: /mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmin/5c-13C
Pulse Sequence: APT_ad



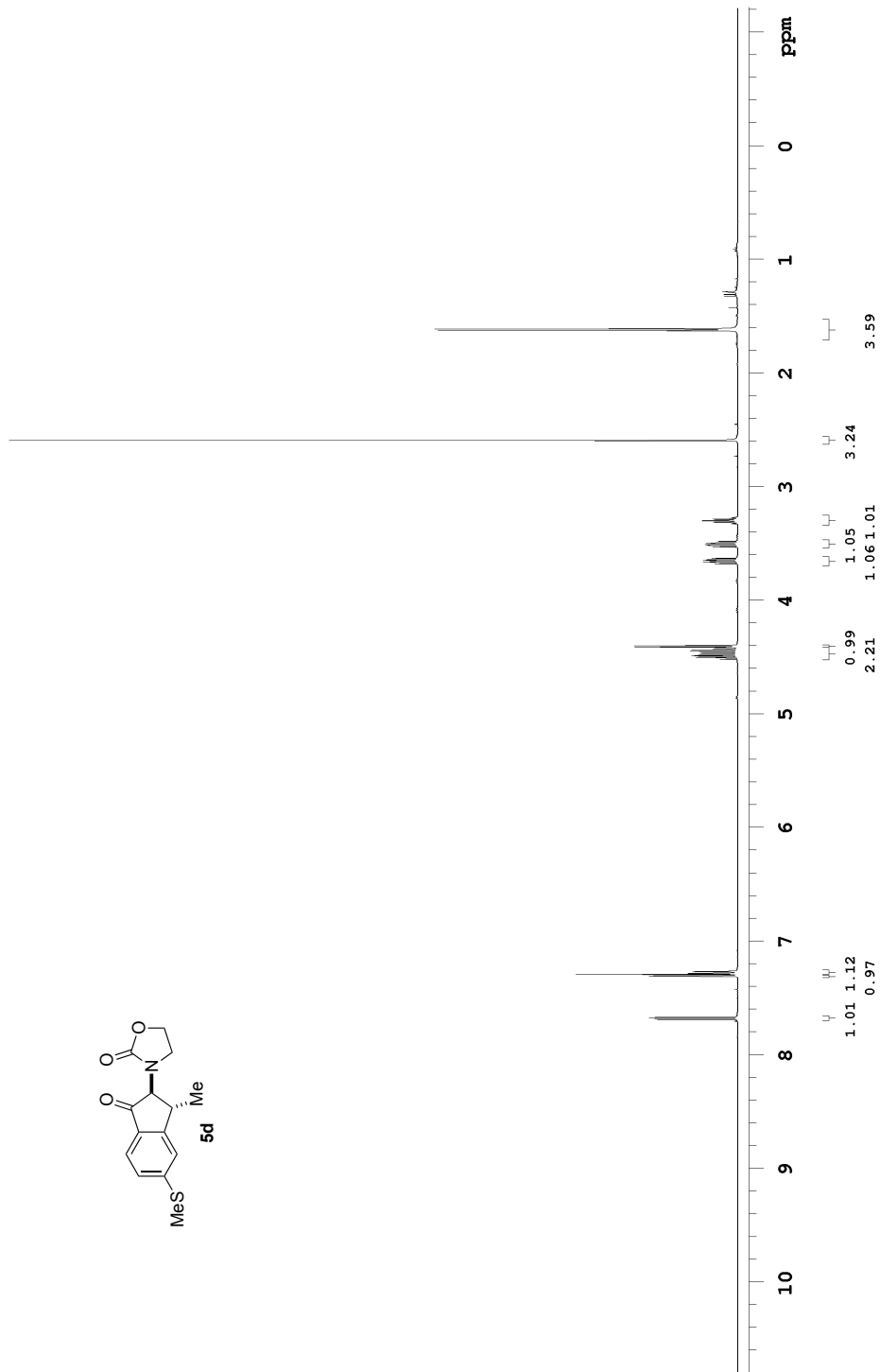
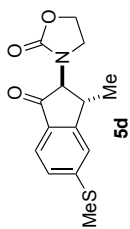
125.267 MHz C13[HL] APT_ad in cdc13 (ref. to CDC13 @ 77.06 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe
dateCHPam2_28Hz_0790ppm1be_3327g_sagehime1gms relax.time: 0.1s # scans: 1244 dig.res.: 0.3 Hz/pt hz/mm:140.9
spectrometer:ibdw file://mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmin/5b-13c
Pulse Sequence: APT_ad



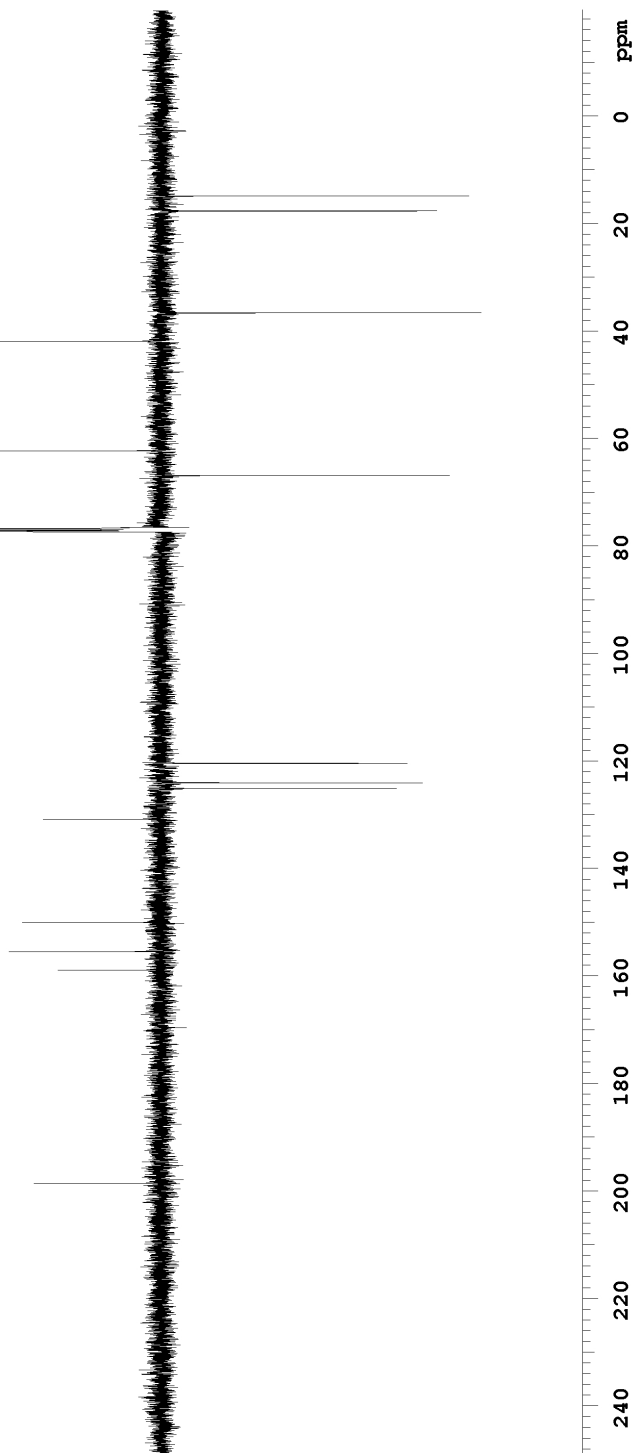
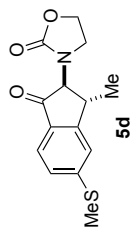
5c, cis:trans 1:4



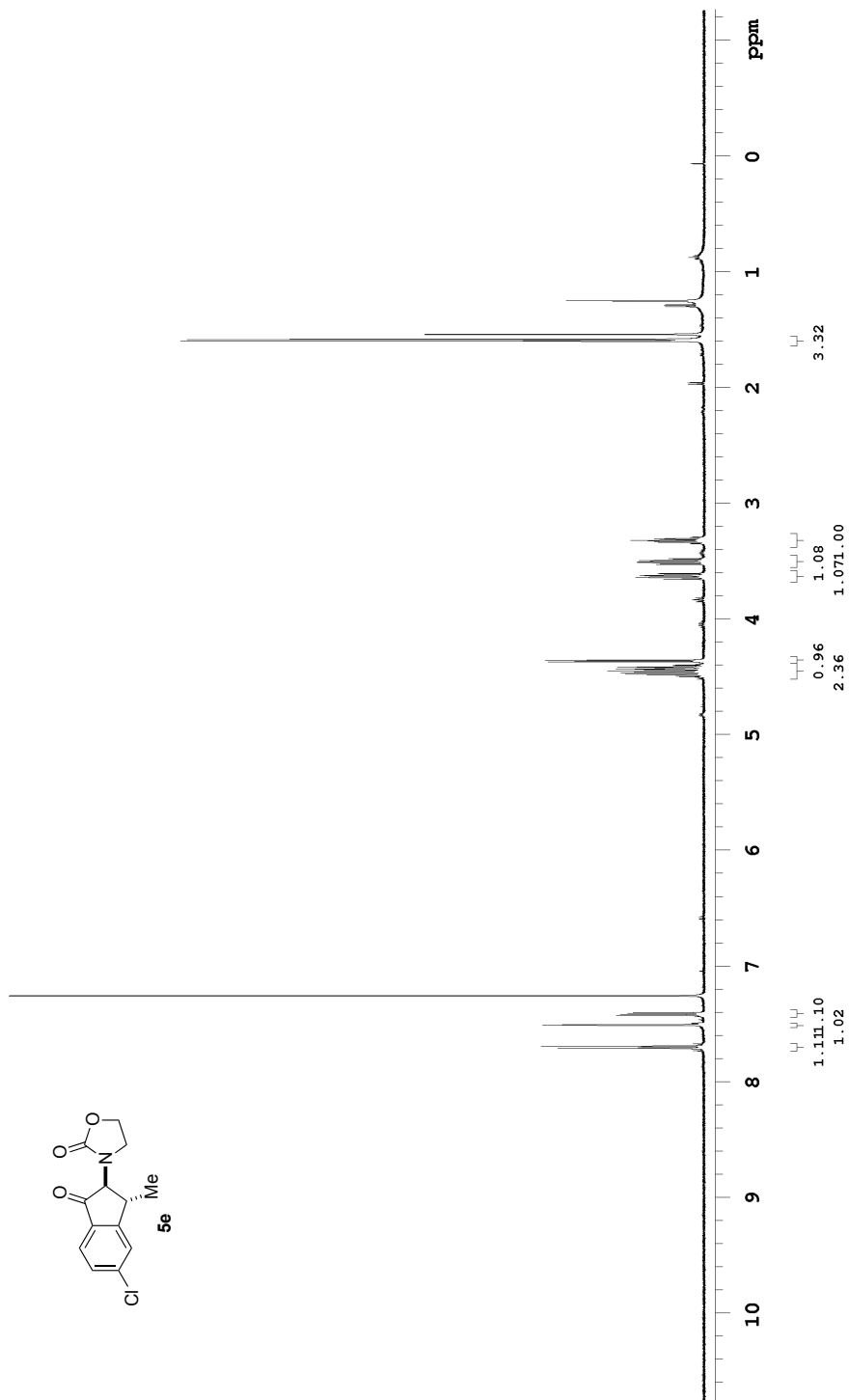
499.815 MHz H1 ID in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.7 C -> actual temp = 27.0 C, coldhdal probe
date: Apr 6 2012 sweep width: 6010Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt
spectrometer:ibdw file://mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmin/5d-1H



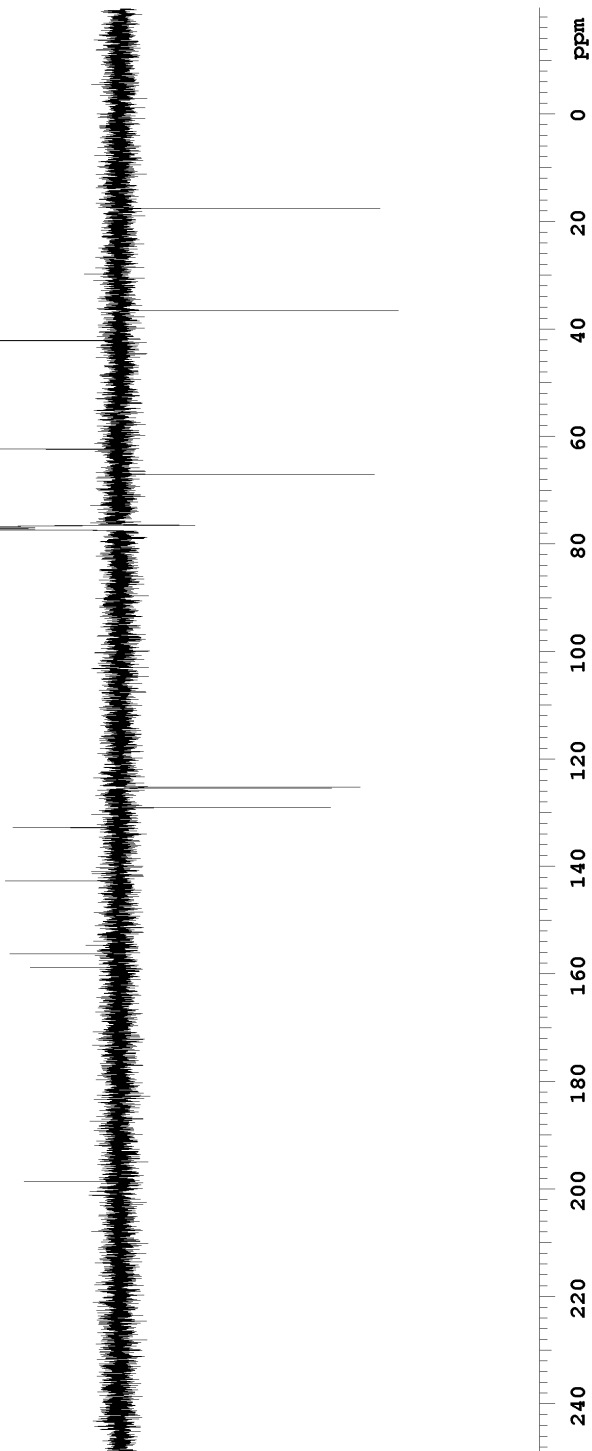
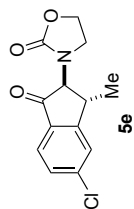
125.692 MHz C13[HL] APT_ad in cdc13 (ref. to CDC13 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, coldludal probe
dateCHPam6 28Hz, 0.1s # scans: 72 dig.res.: 0.3 Hz/pt hz/mm:140.8
spectrometer:ibdw file://mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmi/5d-13C
Pulse Sequence: APT_ad



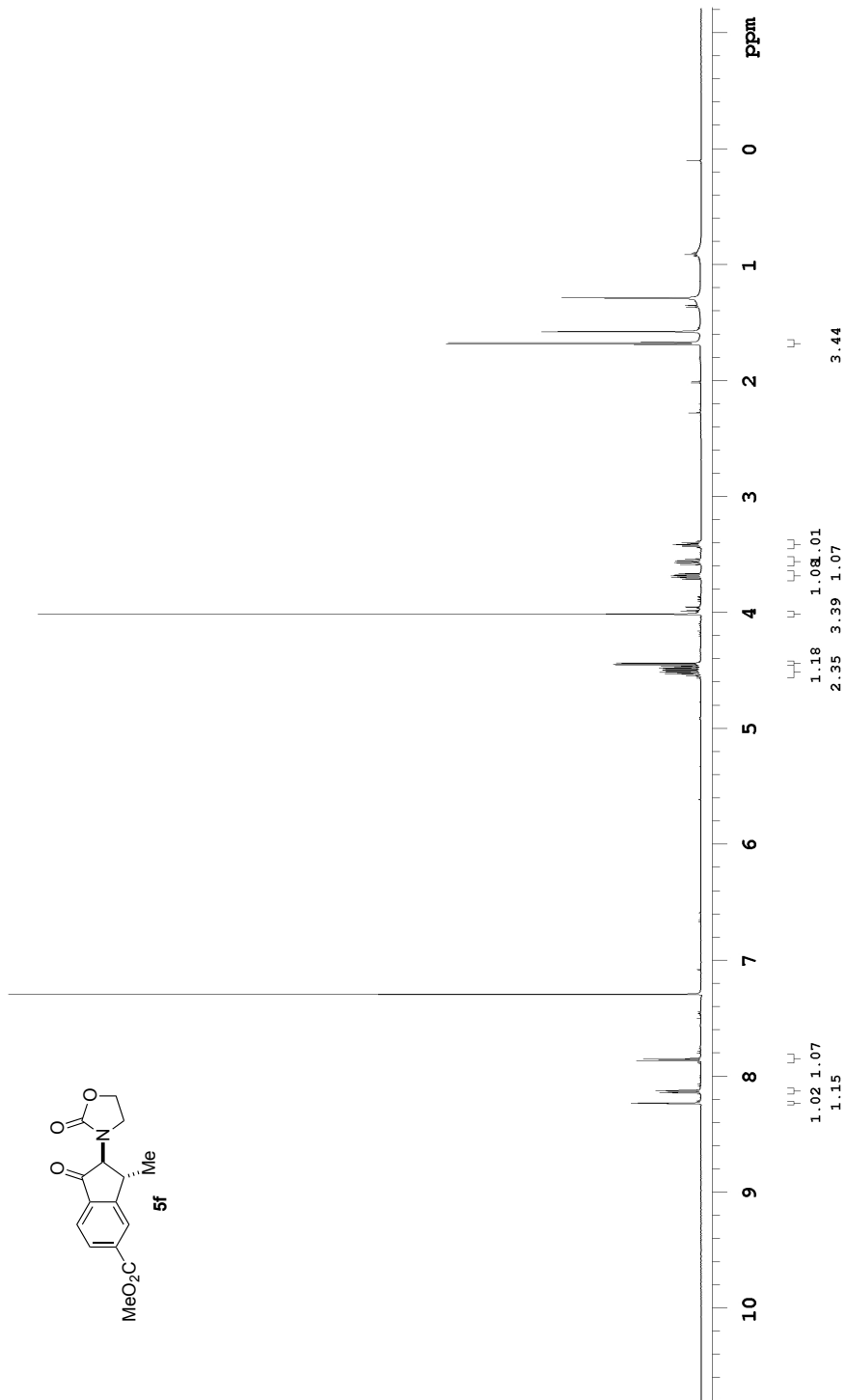
498.122 MHz H1 ID in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.2 C -> actual temp = 27.0 C, autoxdb probe
date: Apr 16 2012 sweep width: 6001Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt
spectrometer:ibdw file://mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmin/5e-1H
hz/mm:25.0 Pulse Sequence: s2pul



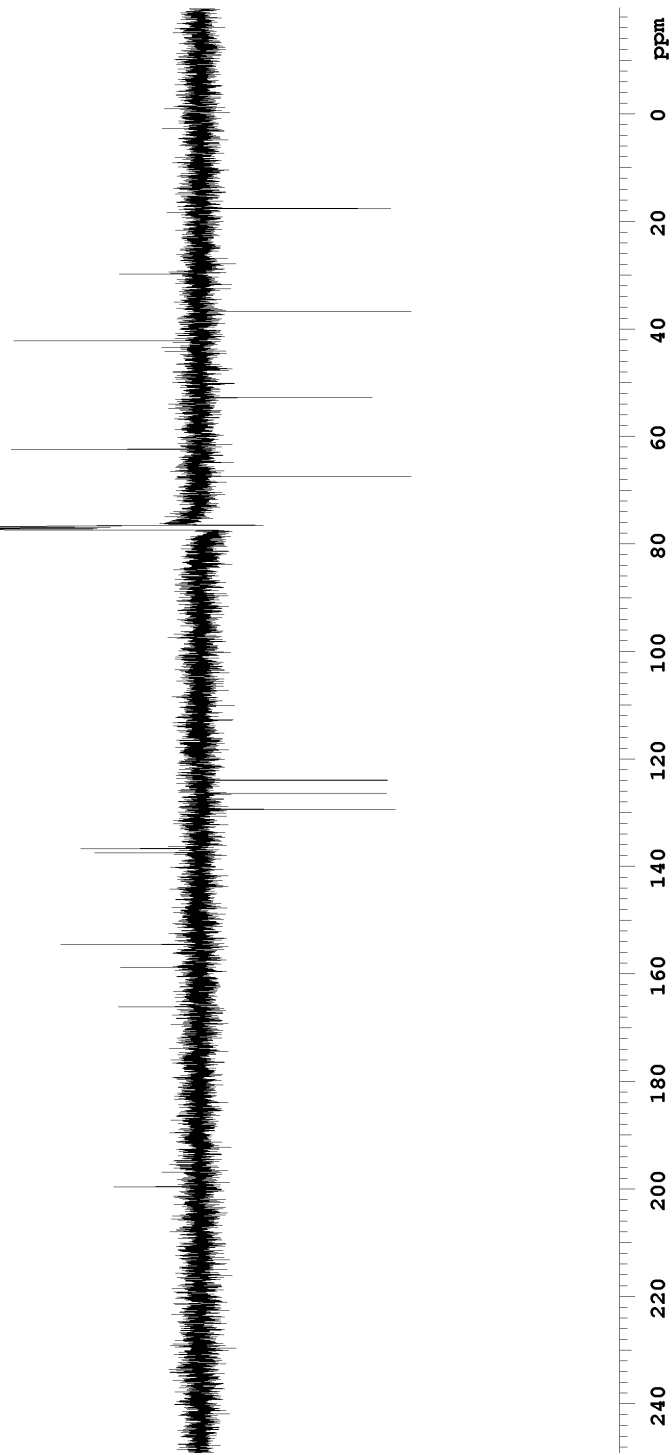
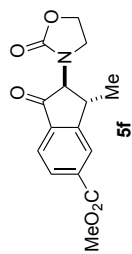
125.692 MHz C13[HL] APT_ad in cdc13 (ref. to CDC13 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, coldludal probe
date: 2012-08-28, 09:59:56, 333.49g, 333.49g, 333.49g, relax.time: 0.1s # scans: 188 dig.res.: 0.3 Hz/pt hz/mm: 140.8
spectrometer: ibdw file: /mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmin/5e-13C
Pulse Sequence: APT_ad



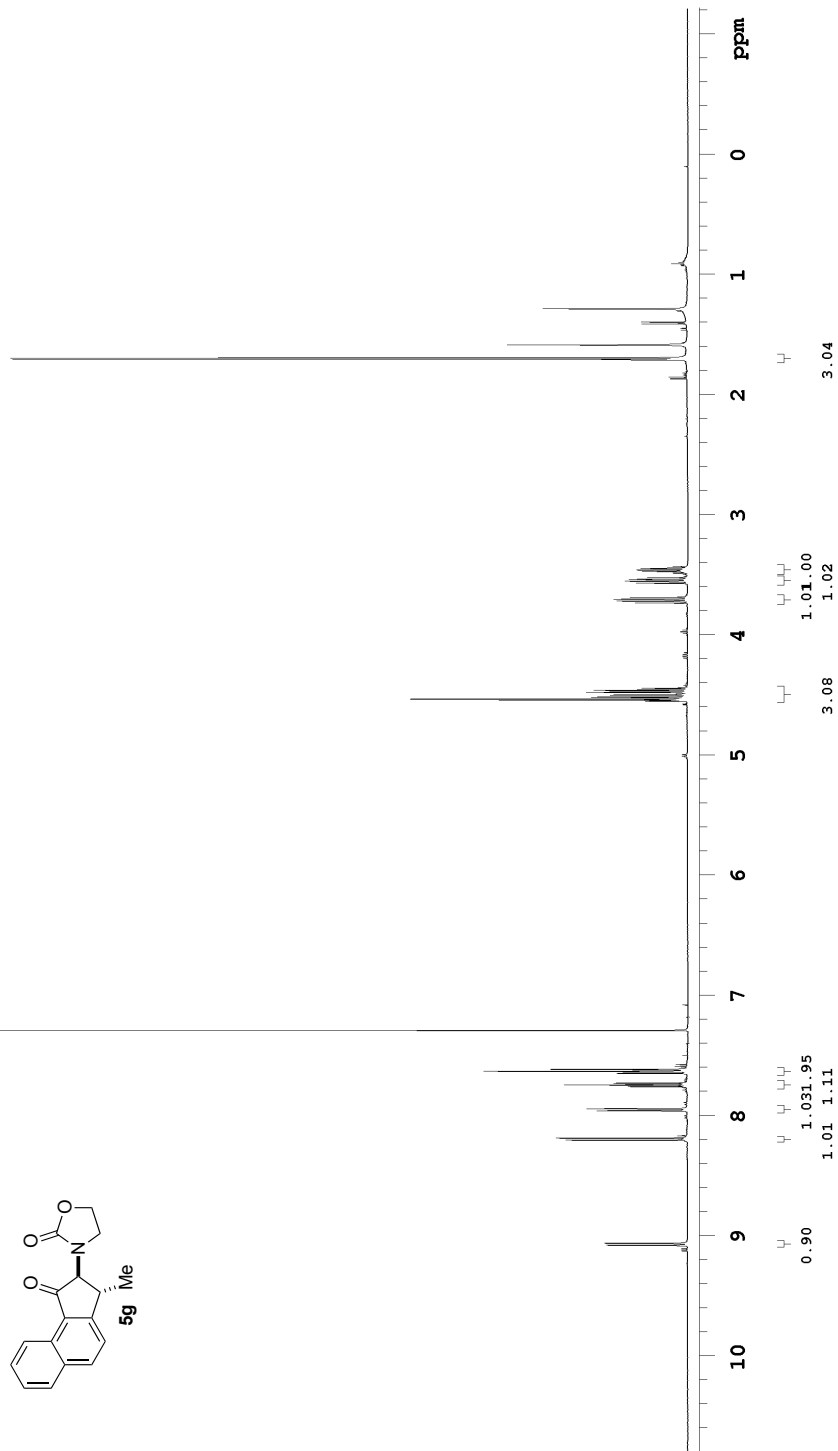
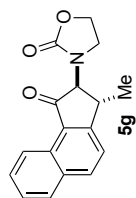
499.815 MHz H1 ID in cdcl3 (ref. to cdcl3 @ 7.26 ppm), temp 27.7 C -> actual temp = 27.0 C, coldlial probe
date: Apr 19 2012 sweep width: 6010Hz acq.time: 5.0s relax.time: 0.1s # scans: 4 dig.res.: 0.1 Hz/pt
spectrometer:ibdw file://mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmin/5f-1H



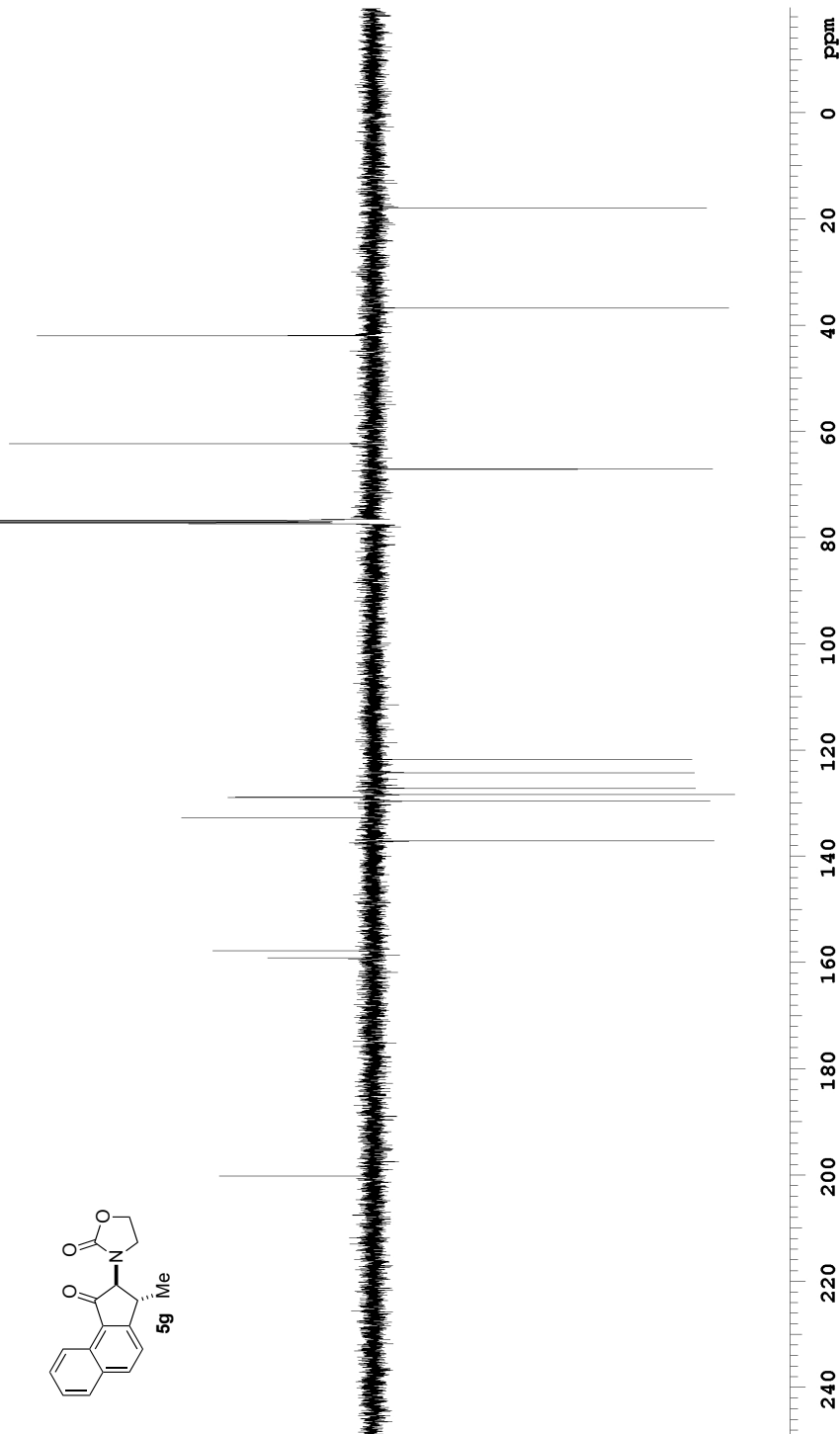
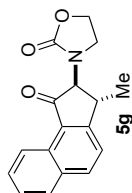
125.692 MHz C13[HL] APT_ad in cdc13 (ref. to CDC13 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, coldludal probe
date: 20120328 20:22:48, 28/03/2012 20:22:48, 28/03/2012 20:22:48, relax.time: 0.1s # scans: 220 dig.res.: 0.3 Hz/pt
spectrometer: ibdw file: /mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmin/5f-13C
Pulse Sequence: APT_ad



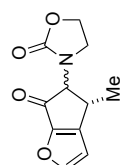
499.815 MHz H1 ID in cdcl3 (ref. to cdcl3 @ 7.26 ppm), temp 27.7 C -> actual temp = 27.0 C, coldlual probe
date: Apr 26 2012 sweep width: 6010Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt
spectrometer:ibdw file://mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmin/5g-1H



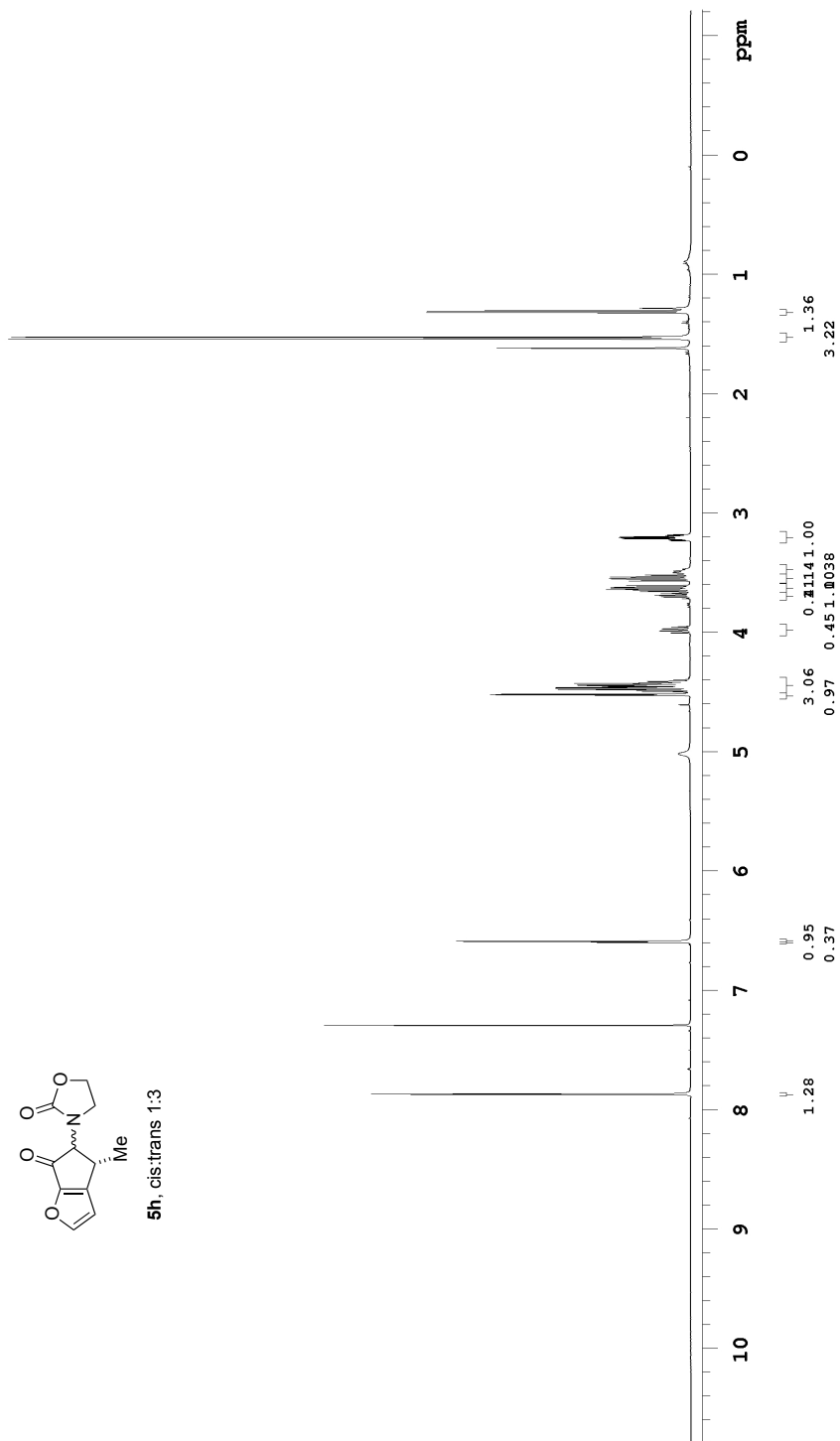
125.692 Mhz C13[HL] APT_ad in cdc13 (ref. to CDC13 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, coldludal probe
date: 08/08/2012, 20:22:00, 0.3 Hz/pt, 0.1 s # scans: 120, dig.res.: 0.3 Hz/pt, 140.8
spectrometer: ibdw
file: /mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmin/5g-13C
Pulse Sequence: APT_ad



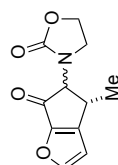
499.815 MHz H1 ID in cdcl3 (ref. to cdcl3 @ 7.26 ppm), temp 27.7 C -> actual temp = 27.0 C, coldlial probe
date: May 3 2012 sweep width: 6010Hz acq.time: 5.0s relax.time: 0.1s # scans: 16 dig.res.: 0.1 Hz/pt
spectrometer:ibdw file:/mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmin/5h-1H



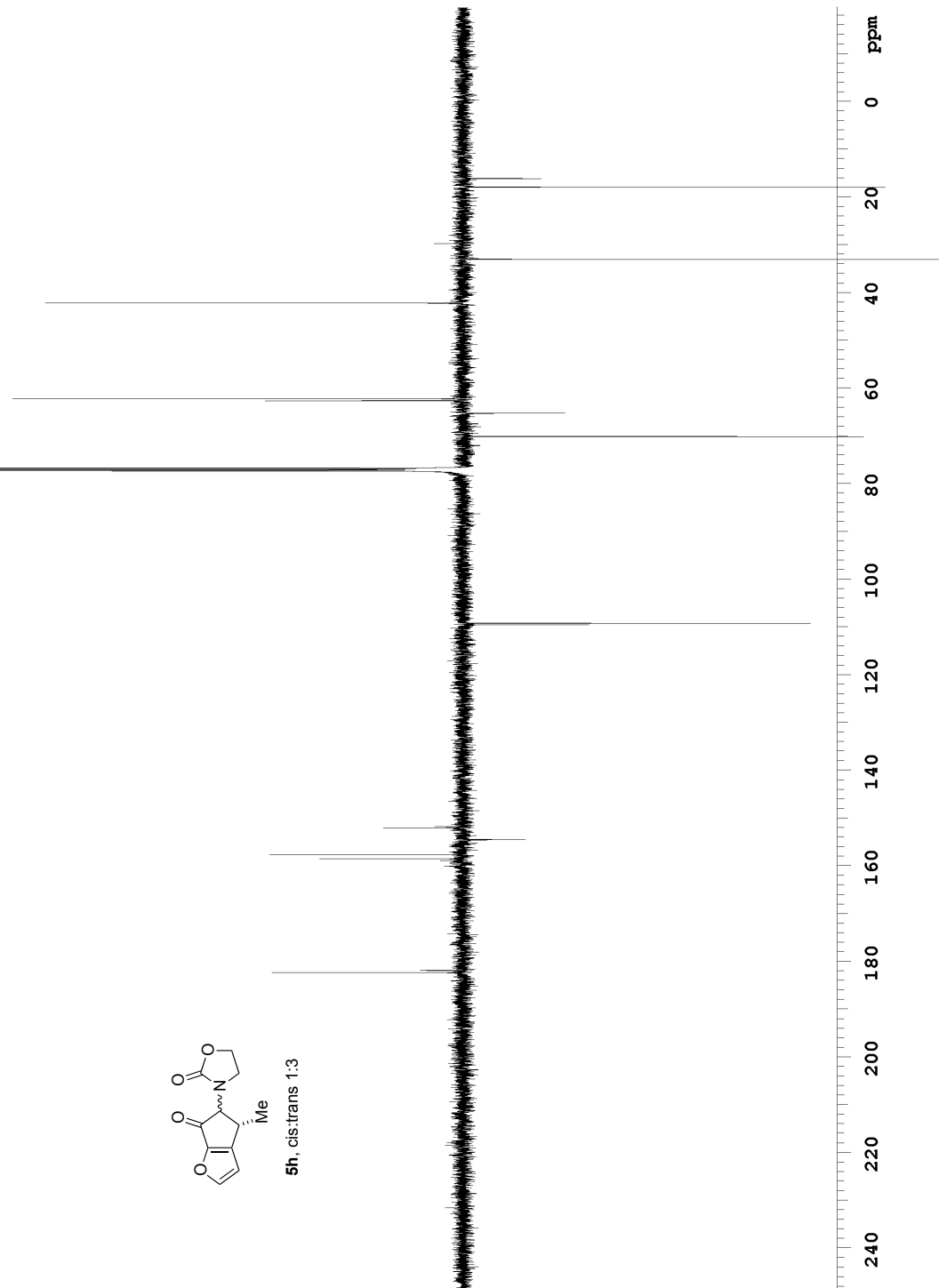
5h, cis:trans 1:3



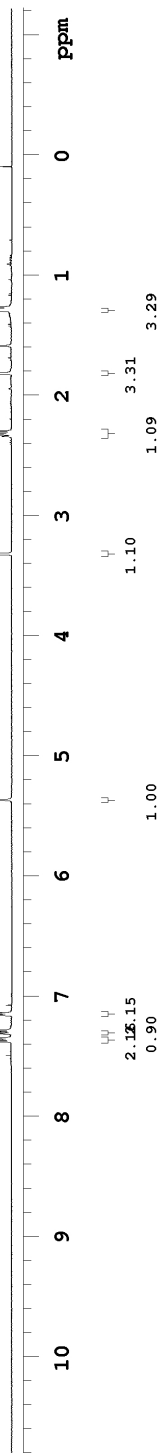
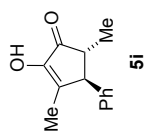
125.692 MHz C13[HL] APT_ad in cdc13 (ref. to CDC13 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, coldludal probe
dateCHM5am8_28Hz_0790ppm130 33349g segtime1.06s relax.time: 0.1s # scans: 228 dig.res.: 0.3 Hz/pt hz/mm:140.8
spectrometer:ibdw file://mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmi/5h-13C
Pulse Sequence: APT_ad



5h, cis:trans 1:3



499.815 MHz H1 ID in cdcl3 (ref. to CDCl3 @ 7.26 ppm), temp 27.7 C -> actual temp = 27.0 C, coldlual probe
date: Feb 25 2011 sweep width: 6010Hz acq.time: 5.0s relax.time: 0.1s # scans: 0 dig.res.: 0.1 Hz/pt
spectrometer:ibdw file://mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmin/51-1H



125.692 Mhz C13[HL] APT_ad in cdc13 (ref. to CDC13 @ 77.06 ppm), temp 27.7 C -> actual temp = 27.0 C, coldludal probe
date: 08/08/2011 20:11:48, 08/08/2011 20:11:48, 08/08/2011 20:11:48, relax.time: 0.1s # scans: 200 dig.res.: 0.3 Hz/pt
spectrometer: ibdw file: /mnt/d600/home13/westnmr/nmrdata/DATA_FROM_NMRSERVICE/Yen-Ku/03-Tianmin/51-13C
Pulse Sequence: APT_ad

