

**A Facile Access to Enantioenriched Isoindolines *via* One-Pot
Sequential Cu(I)-Catalyzed Asymmetric 1,3-Dipolar
Cycloaddition/Oxidation**

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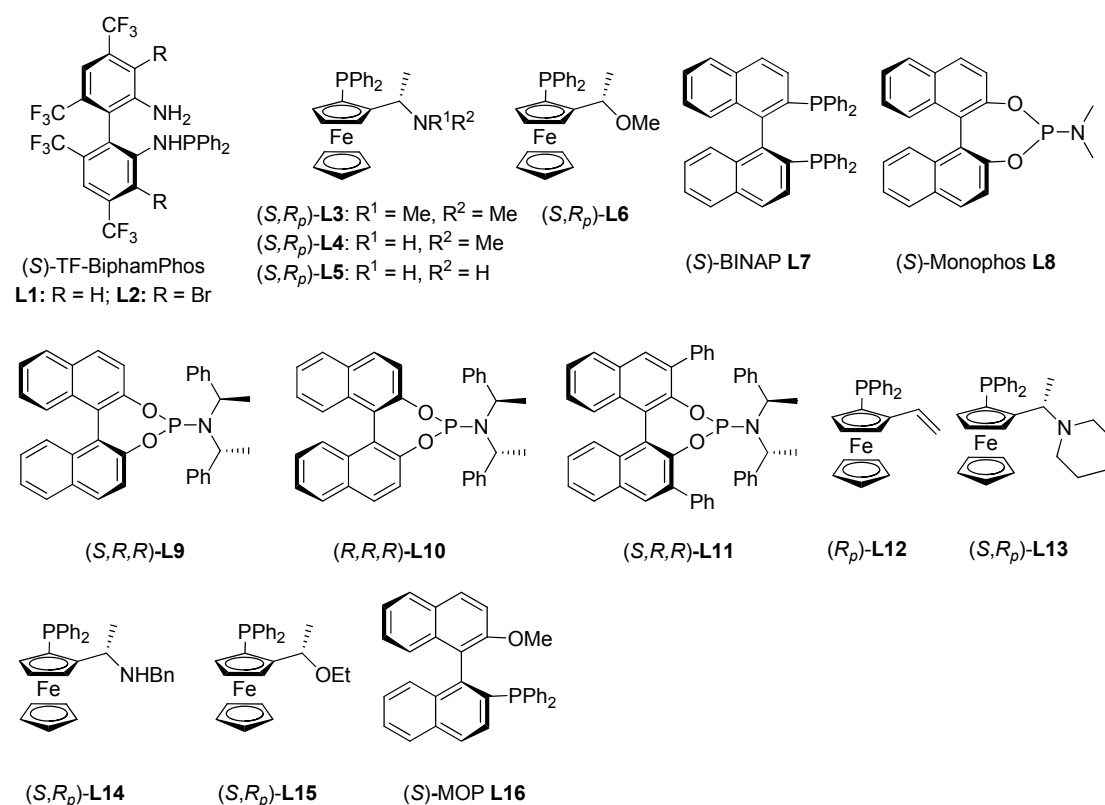
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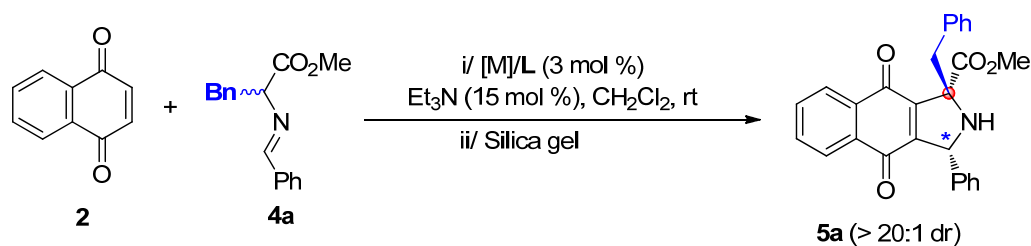
I. General Remarks.

^1H NMR spectra were recorded on a VARIAN Mercury 300 MHz or Bruker 400 MHz spectrometer in CDCl_3 . ^{13}C NMR spectra were recorded on a VARIAN Mercury 75 MHz or Bruker 100 MHz spectrometer in CDCl_3 . Commercially obtained reagents were used without further purification. All reactions were monitored by TLC with silica gel-coated plates. Diastereomeric ratios were determined from crude ^1H NMR or HPLC analysis. Enantiomeric excesses were determined by HPLC, using a chiralpak AD-H column, a chiralpak AS-H column or a chiralcel OD-H column with hexane and *i*-PrOH as solvents. (*S,R_p*)-**L6** were prepared according to the literature procedure.¹ The racemic adducts were attained by using $\text{Cu}(\text{CN})_4\text{BF}_4/\text{PPh}_3$ as the catalyst. The absolute (1*R*,3*R*)-**5b** achieved by $\text{Cu}(\text{CN})_4\text{BF}_4/(\textit{S,R}_p)\text{-PPFOMe}$ was determined unequivocally according to the X-ray diffraction analysis, and those of other adducts were deduced on the basis of these results.²

II. Ligand Screening for One Pot and Sequential Catalytic Asymmetric 1,3-DC/Oxidation Reaction



Scheme 1. Screened chiral ligands.



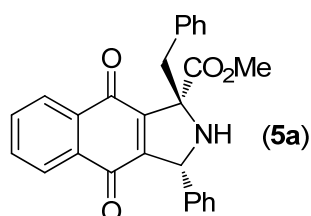
entry	L	[M]	time/h	yield (%) ^b	ee (%) ^c
1	L1	AgOAc	6	81	20
2	L1	Cu(CH ₃ CN) ₄ BF ₄	6	87	9
3	L2	AgOAc	6	86	27
4	L2	Cu(CH ₃ CN) ₄ BF ₄	6	85	37
5	L3	AgOAc	4	76	9
6	L3	Cu(CH ₃ CN) ₄ BF ₄	4	85	71
7	L4	Cu(CH ₃ CN) ₄ BF ₄	4	69	69
8	L5	Cu(CH ₃ CN) ₄ BF ₄	4	74	22
9	L6	Cu(CH ₃ CN) ₄ BF ₄	4	85	78
10	L7	AgOAc	12	31	2
11	L7	Cu(CH ₃ CN) ₄ BF ₄	12	34	4
12	L8	AgOAc	4	65	7
13	L8	Cu(CH ₃ CN) ₄ BF ₄	4	50	13
14	L9	Cu(CH ₃ CN) ₄ BF ₄	4	70	66
15	L10	Cu(CH ₃ CN) ₄ BF ₄	4	71	51
16	L11	Cu(CH ₃ CN) ₄ BF ₄	4	82	14
17	L12	Cu(CH ₃ CN) ₄ BF ₄	4	78	63
18	L13	Cu(CH ₃ CN) ₄ BF ₄	4	67	71
19	L14	Cu(CH ₃ CN) ₄ BF ₄	4	75	37
20	L15	Cu(CH ₃ CN) ₄ BF ₄	4	82	65
21	L16	Cu(CH ₃ CN) ₄ BF ₄	4	78	13

^a All reactions were carried out with 0.26 mmol of **4a** and 0.20 mmol of **2** in 2 mL of CH₂Cl₂. ^b Isolated yield. ^c Determined by HPLC analysis.

III. General Procedure for Cu(I)/(*S,R*_p)-PPFOMe-Catalyzed Asymmetric 1,3-Dipolar Cycloaddition/Oxidation

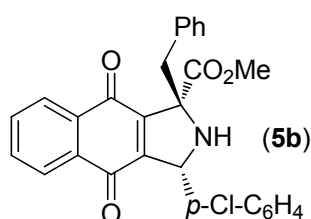
Under argon atmosphere, (*S,R*_p)-PPFOMe (3.1 mg, 0.0072 mmol) and Cu(CH₃CN)₄BF₄ (1.9 mg, 0.006 mmol) were dissolved in toluene (2 mL), and stirred at room temperature for about 1 h. Then, imine substrate (0.26 mmol), and naphthoquinone (0.2 mmol) were added sequentially, after that the mixture was dropped to -20 °C, TEA (3 mg, 0.03 mmol) was added. Once starting material was

consumed (monitored by TLC), The reaction mixture was treated with silica gel for a short time, then the organic solvent was removed and the residue was purified by column chromatography to give the product, which was then directly analyzed by chiral HPLC to determine the enantiomeric excess.



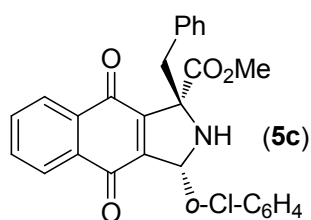
(1R,3R)-methyl 1-benzyl-4,9-dioxo-3-phenyl-2,3,4,9-tetrahydro-1H-benzo[f]isoindole-1-carboxylate

The title compound was prepared according to the general procedure as described above in 86% yield. m.p. 118-121 °C; $[\alpha]_D^{25} = +128.2$ (*c* 1.36, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 8.16 (d, *J* = 7.8 Hz, 1H), 7.88 (d, *J* = 7.8 Hz, 1H), 7.76-7.68 (m, 2H), 7.34-7.15 (m, 10H), 4.88 (s, 1H), 3.85 (s, 3H), 3.63 (d, *J* = 14.1 Hz, 1H), 3.46 (d, *J* = 14.1 Hz, 1H), 2.60-2.80 (br, 1H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 182.4, 181.6, 172.6, 150.6, 146.4, 141.2, 135.6, 133.8, 132.7, 130.3, 128.4, 128.2, 127.9, 127.7, 127.1, 126.4, 126.3, 75.4, 67.1, 52.9, 41.9; ¹³C NMR (DMSO-*d*₆, TMS, 100 MHz) δ 182.0, 180.8, 171.8, 149.6, 146.4, 142.2, 135.8, 134.4, 134.3, 131.8, 131.6, 129.7, 127.8, 127.7, 127.1, 126.6, 125.9, 125.8, 74.5, 66.4, 52.3, 40.7; IR (KBr) ν 3382, 3061, 3026, 2957, 2848, 2167, 1740, 1635, 1593, 1494, 1454, 1635, 1297, 1247, 1045, 909, 774, 736, 702 cm⁻¹. HRMS: calcd. for C₂₇H₂₂NO₄⁺: 424.1543, found 424.1537. The product was analyzed by HPLC to determine the enantiomeric excess: 96% *ee* (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min, λ = 220 nm); *t*_r = 11.59 and 33.88 min.



(1*R*,3*R*)-methyl 1-benzyl-3-(4-chlorophenyl)-4,9-dioxo-2,3,4,9-tetrahydro-1H-benzo[*f*]isoindole-1-carboxylate

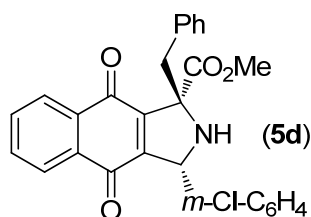
The title compound was prepared according to the general procedure as described above in 89% yield. m.p. 112-115 °C; $[\alpha]_D^{25} = +97.4$ (*c* 1.60, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 8.16 (d, *J* = 7.5 Hz, 1H), 7.82 (d, *J* = 7.5 Hz, 1H), 7.79-7.68 (m, 2H), 7.31-7.19 (m, 7H), 7.12 (m, 2H), 4.84 (s, 1H), 3.85 (s, 3H), 3.63 (d, *J* = 13.8 Hz, 1H), 3.46 (d, *J* = 13.8 Hz, 1H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 182.3, 181.5, 172.5, 150.0, 146.4, 139.8, 135.4, 133.9, 133.6, 132.6, 130.7, 130.2, 129.2, 128.5, 128.3, 127.6, 127.1, 126.5, 126.3, 75.3, 66.3, 52.9, 41.6; IR (KBr) ν 3380, 1741, 1667, 1637, 1594, 1491, 1339, 1219, 1089, 704 cm⁻¹. HRMS: calcd. for C₂₇H₂₁ClNO₄⁺: 458.1154, found. 458.1148. The product was analyzed by HPLC to determine the enantiomeric excess: 95% *ee* (Chiralpak AS-H, *i*-propanol/hexane = 40/60, flow rate 1.0 mL/min, λ = 220 nm); t_r = 8.07 and 20.86 min.



(1*R*,3*S*)-methyl 1-benzyl-3-(2-chlorophenyl)-4,9-dioxo-2,3,4,9-tetrahydro-1H-benzo[*f*]isoindole-1-carboxylate

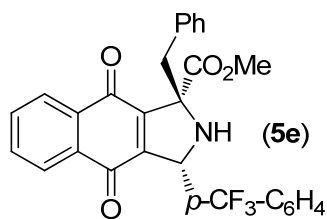
The title compound was prepared according to the general procedure as described above in 87% yield. m.p. 129-132 °C; $[\alpha]_D^{25} = +75.4$ (*c* 1.54, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 8.18 (d, *J* = 7.5 Hz, 1H), 7.92 (d, *J* = 7.5 Hz, 1H), 7.90-7.71 (m, 2H), 7.35-7.32 (m, 1H), 7.24-7.15 (m, 8H), 5.36 (s, 1H), 3.80 (s, 3H), 3.63 (d, *J* = 14.1 Hz, 1H), 3.47 (d, *J* = 14.1 Hz, 1H), 2.79 (br, 1H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 182.1, 181.1, 172.2, 150.2, 147.3, 138.6, 135.2, 133.7, 133.1, 132.6, 132.5, 130.0, 129.3, 129.0, 128.8, 128.2, 127.1, 127.0, 126.4, 126.3, 75.1, 62.6, 52.7, 41.4; ¹³C NMR (DMSO-*d*₆, TMS, 100 MHz) δ 181.9, 180.5, 171.7, 149.4, 147.2, 139.6, 135.7, 134.6, 134.5, 132.0, 131.7, 131.6, 129.8, 129.7, 128.8, 128.7, 127.8, 127.2, 126.8, 126.0, 125.9, 74.5, 62.0, 52.4, 40.5; IR (KBr) ν 3374, 2951, 2168, 1740,

1637, 1593, 1496, 1474, 1438, 1368, 1340, 1297, 1253, 1050, 906, 798, 755, 742, 705 cm^{-1} . HRMS: calcd. for $\text{C}_{27}\text{H}_{21}\text{ClNO}_4^+$: 458.1154, found. 458.1150. The product was analyzed by HPLC to determine the enantiomeric excess: 94% *ee* (Chiralpak AS-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 8.79 and 26.77 min.



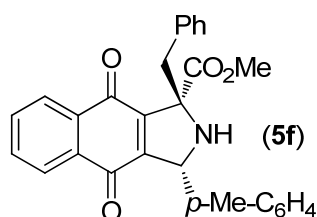
(1*R*,3*R*)-methyl 1-benzyl-3-(3-chlorophenyl)-4,9-dioxo-2,3,4,9-tetrahydro-1H-benzo[f]isoindole-1-carboxylate

The title compound was prepared according to the general procedure as described above in 87% yield. m.p. 176-179 °C; $[\alpha]_D^{25} = +59.0$ (*c* 1.52, CHCl_3); ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 8.15 (m, 1H), 7.88 (m, 1H), 7.77-7.70 (m, 2H), 7.38 (m, 1H), 7.21-7.19 (m, 7H), 7.11-7.10 (m, 1H), 4.85 (s, 1H), 3.87 (s, 3H), 3.63 (d, J = 14.1 Hz, 1H), 3.46 (d, J = 14.1 Hz, 1H), 2.60-2.80 (br, 1H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 182.3, 181.4, 172.4, 149.9, 146.5, 143.4, 135.4, 134.2, 133.9, 132.7, 130.2, 129.5, 128.4, 128.1, 128.0, 127.2, 126.5, 126.4, 126.1, 75.3, 66.5, 52.9, 41.6; ^{13}C NMR (DMSO-d_6 , TMS, 100 MHz) δ 181.9, 180.7, 171.7, 148.9, 146.6, 144.8, 135.7, 134.5, 134.4, 132.4, 131.7, 131.6, 129.6, 127.8, 127.6, 127.1, 126.6, 126.4, 125.9, 125.8, 74.6, 65.8, 52.2, 40.5; IR (KBr) ν 3382, 2168, 1741, 1637, 1594, 1433, 1369, 1340, 1297, 1249, 1048, 777, 740, 705, 587 cm^{-1} . HRMS: calcd. for $\text{C}_{27}\text{H}_{21}\text{ClNO}_4^+$: 458.1154, found. 458.1146. The product was analyzed by HPLC to determine the enantiomeric excess: 94% *ee* (Chiralpak AS-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 9.51 and 31.36 min.



(1R,3R)-methyl 1-benzyl-4,9-dioxo-3-(4-(trifluoromethyl)phenyl)-2,3,4,9-tetrahydro-1H-benzo[f]isoindole-1-carboxylate

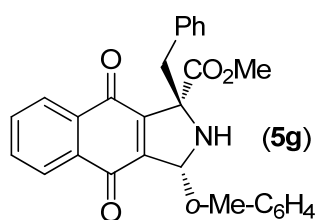
The title compound was prepared according to the general procedure as described above in 87% yield. m.p. 187-190 °C; $[\alpha]_D^{25} = +72.6$ (*c* 1.84, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 8.17 (d, *J* = 7.5 Hz, 1H), 7.88 (d, *J* = 7.5 Hz, 1H), 7.80-7.68 (m, 2H), 7.55-7.47 (m, 4H), 7.22-7.10 (m, 5H), 4.92 (s, 1H), 3.86 (s, 3H), 3.65 (d, *J* = 14.1 Hz, 1H), 3.47 (d, *J* = 14.1 Hz, 1H); ¹³C NMR (CDCl₃, TMS, 100 MHz) δ 182.3, 181.5, 172.4, 149.8, 146.7, 145.2, 135.3, 134.0, 133.9, 132.7, 130.2, 129.9 (*J*_{C-F} = 32.4 Hz), 128.4, 128.3, 127.3, 126.6, 126.4, 125.3 (*J*_{C-F} = 3.7 Hz), 124.0 (*J*_{C-F} = 270.3 Hz), 75.4, 66.6, 53.0, 41.6; IR (KBr) ν 3384, 2953, 1742, 1668, 1637, 1618, 1594, 1496, 1436, 1369, 1325, 1249, 1219, 1165, 1124, 1067, 1017, 849, 735, 704, 642, 601 cm⁻¹. HRMS: calcd. for C₂₈H₂₁F₃NO₄⁺: 492.1409, found. 492.1417. The product was analyzed by HPLC to determine the enantiomeric excess: 92% *ee* (Chiralpak AD-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); *t*_r = 8.85 and 10.63 min.



(1R,3R)-methyl 1-benzyl-4,9-dioxo-3-(p-tolyl)-2,3,4,9-tetrahydro-1H-benzo[f]isoindole-1-carboxylate

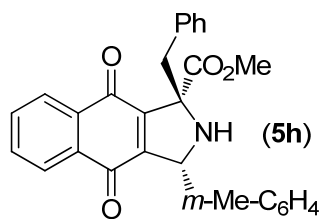
The title compound was prepared according to the general procedure as described above in 93% yield. m.p. 176-178 °C; $[\alpha]_D^{25} = +156.3$ (*c* 1.62, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 8.15 (d, *J* = 7.8 Hz, 1H), 7.87 (d, *J* = 7.8 Hz, 1H), 7.86-7.67 (m, 2H), 7.21-7.07 (m, 9H), 4.85 (s, 1H), 3.84 (s, 3H), 3.62 (d, *J* = 13.8 Hz,

1H), 3.44 (d, $J = 13.8$ Hz, 1H), 2.60-2.80 (br, 1H), 2.29 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 182.4, 181.6, 172.6, 150.7, 146.3, 138.3, 137.5, 135.6, 133.7, 132.7, 130.3, 129.1, 128.1, 127.5, 127.0, 126.4, 126.3, 75.3, 66.8, 52.8, 41.8, 21.1; IR (KBr) ν 3381, 3029, 2951, 1740, 1634, 1593, 1453, 1339, 1297, 1246, 1045, 816, 771, 735, 704 cm^{-1} . HRMS: calcd. for $\text{C}_{28}\text{H}_{24}\text{NO}_4^+$: 438.1670, found. 438.1695. The product was analyzed by HPLC to determine the enantiomeric excess: 95% *ee* (Chiralpak AS-H, *i*-propanol/hexane = 30/70, flow rate 1.2 mL/min, $\lambda = 220$ nm); $t_r = 7.57$ and 20.44 min.



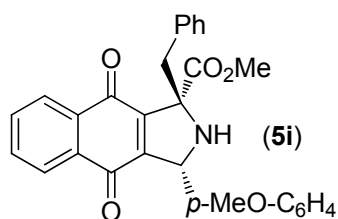
(1R,3R)-methyl 1-benzyl-4,9-dioxo-3-(*o*-tolyl)-2,3,4,9-tetrahydro-1H-benzo[f]isoindole-1-carboxylate

The title compound was prepared according to the general procedure as described above in 81% yield. m.p. 135-138 °C; $[\alpha]_D^{25} = +107.4$ (c 1.50, CHCl_3); ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 8.17 (d, $J = 7.2$ Hz, 1H), 7.89 (d, $J = 7.2$ Hz, 1H), 7.79-7.67 (m, 2H), 7.19-7.12 (m, 9H), 4.92 (s, 1H), 3.82 (s, 3H), 3.58 (d, $J = 13.5$ Hz, 1H), 3.46 (d, $J = 13.5$ Hz, 1H), 2.60-2.80 (br, 1H), 2.34 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 182.2, 181.4, 172.4, 151.8, 147.2, 139.1, 135.7, 133.7, 132.7, 130.3, 127.9, 127.6, 127.2, 126.9, 126.4, 126.2, 75.1, 62.7, 52.8, 41.9, 19.1; IR (KBr) ν 3358, 3029, 2950, 1740, 1667, 1593, 1219, 1050, 735 cm^{-1} . HRMS: calcd. for $\text{C}_{28}\text{H}_{24}\text{NO}_4^+$: 438.1670, found. 438.1693. The product was analyzed by HPLC to determine the enantiomeric excess: 93% *ee* (Chiralcel OD-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, $\lambda = 220$ nm); $t_r = 6.33$ and 15.03 min.



(1R,3R)-methyl 1-benzyl-4,9-dioxo-3-(m-tolyl)-2,3,4,9-tetrahydro-1H-benzo[f]isoindole-1-carboxylate

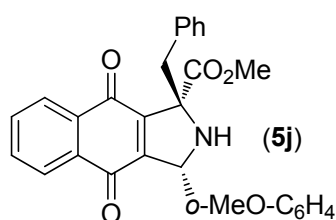
The title compound was prepared according to the general procedure as described above in 86% yield. m.p. 179-182 °C; $[\alpha]_D^{25} = +113.5$ (*c* 1.48, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 8.16 (d, *J* = 7.2 Hz, 1H), 7.88 (d, *J* = 7.2 Hz, 1H), 7.76-7.69 (m, 2H), 7.19-7.06 (m, 9H), 4.84 (s, 1H), 3.84 (s, 3H), 3.62 (d, *J* = 14.1 Hz, 1H), 3.45 (d, *J* = 14.1 Hz, 1H), 2.29 (s, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 182.5, 181.6, 172.6, 150.8, 146.4, 141.2, 138.0, 135.6, 133.8, 133.7, 132.8, 130.3, 128.7, 128.5, 128.3, 128.2, 127.0, 126.4, 126.3, 124.8, 75.4, 67.1, 52.8, 41.9, 21.4; IR (KBr) ν 3383, 3026, 2951, 1741, 1667, 1643, 1593, 1454, 1339, 1296, 1246, 1169, 1044, 733, 704 cm⁻¹. HRMS: calcd. for C₂₈H₂₄NO₄⁺: 438.1670, found. 438.1695. The product was analyzed by HPLC to determine the enantiomeric excess: 97% *ee* (Chiralpak AS-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); *t*_r = 7.95 and 19.35 min.



(1R,3R)-methyl 1-benzyl-3-(4-methoxyphenyl)-4,9-dioxo-2,3,4,9-tetrahydro-1H-benzo[f]isoindole-1-carboxylate

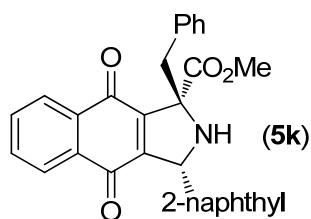
The title compound was prepared according to the general procedure as described above in 81% yield. m.p. 143-145 °C; $[\alpha]_D^{25} = +215.9$ (*c* 1.44, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 8.16 (d, *J* = 7.8 Hz, 1H), 7.88 (d, *J* = 7.8 Hz, 1H), 7.78-7.66 (m, 2H), 7.23-7.15 (m, 7H), 6.81 (d, *J* = 8.1 Hz, 2H), 4.85 (s, 1H), 3.85 (s, 3H), 3.76 (s, 3H), 3.61 (d, *J* = 13.5 Hz, 1H), 3.45 (d, *J* = 13.5 Hz, 1H), 2.60-2.80 (br,

¹H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 182.5, 181.6, 172.7, 159.1, 150.6, 146.1, 135.6, 133.7, 133.4, 132.7, 130.2, 128.8, 128.2, 127.0, 126.4, 126.3, 113.7, 75.2, 66.5, 55.1, 52.9, 41.8; IR (KBr) ν 3381, 3029, 2952, 2837, 1740, 1633, 1593, 1511, 1454, 1367, 1338, 1301, 1246, 1174, 1034, 915, 833, 771, 736, 712, 641, 586, 554 cm⁻¹. HRMS: calcd. for C₂₈H₂₄NO₅⁺: 454.1649, found. 454.1641. The product was analyzed by HPLC to determine the enantiomeric excess: 96% *ee* (Chiralpak AS-H, *i*-propanol/hexane = 30/70, flow rate 1.2 mL/min, λ = 220 nm); t_r = 13.51 and 39.34 min.



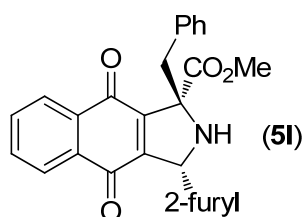
(1*R*,3*R*)-methyl 1-benzyl-3-(2-methoxyphenyl)-4,9-dioxo-2,3,4,9-tetrahydro-1H-benzo[f]isoindole-1-carboxylate

The title compound was prepared according to the general procedure as described above in 85% yield. m.p. 149-152 °C; [α]_D²⁵ = +49.3 (*c* 0.93, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 8.16 (d, *J* = 6.9 Hz, 1H), 7.88 (d, *J* = 6.9 Hz, 1H), 7.87-7.69 (m, 2H), 7.22-7.13 (m, 6H), 6.92-6.89 (m, 2H), 6.77 (d, *J* = 7.5 Hz, 1H), 4.87 (s, 1H), 3.84 (s, 3H), 3.75 (s, 3H), 3.62 (d, *J* = 14.1 Hz, 1H), 3.45 (d, *J* = 14.1 Hz, 1H), 2.69 (br, 1H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 182.4, 181.5, 172.6, 159.6, 150.6, 146.3, 142.9, 135.6, 133.8, 133.7, 132.7, 130.3, 129.3, 128.2, 127.1, 126.4, 126.3, 120.1, 113.5, 113.2, 75.4, 67.0, 55.0, 52.9, 41.8; IR (KBr) ν 3382, 3027, 2950, 1740, 1634, 1593, 1508, 1495, 1453, 1434, 1368, 1337, 1296, 1245, 1169, 1045, 860, 819, 771, 731, 704 cm⁻¹. HRMS: calcd. for C₂₈H₂₅NO₅ + H⁺: 456.1806, found. 456.1790. The product was analyzed by HPLC to determine the enantiomeric excess: 94% *ee* (Chiralpak AS-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 11.87 and 33.47 min.



(1*R*,3*R*)-methyl 1-benzyl-3-(naphthalen-2-yl)-4,9-dioxo-2,3,4,9-tetrahydro-1H-benzo[f]isoindole-1-carboxylate

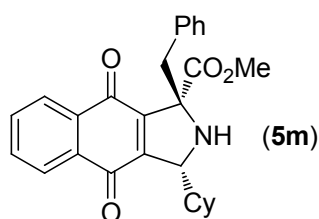
The title compound was prepared according to the general procedure as described above in 79% yield. m.p. 198-201 °C; $[\alpha]_D^{25} = +43.1$ (*c* 1.46, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 8.17 (d, *J* = 7.5 Hz, 1H), 7.84 (d, *J* = 7.5 Hz, 1H), 7.77-7.67 (m, 5H), 7.47-7.42 (m, 3H), 7.22-7.17 (m, 6H), 5.05 (s, 1H), 3.89 (s, 3H), 3.66 (d, *J* = 14.1 Hz, 1H), 3.49 (d, *J* = 14.1 Hz, 1H), 2.78 (br, 1H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 182.5, 181.6, 172.6, 150.5, 146.4, 138.6, 135.6, 133.7, 133.1, 133.0, 132.7, 130.3, 128.3, 128.0, 127.6, 127.1, 126.9, 126.4, 126.3, 126.0, 125.9, 125.4, 75.4, 67.2, 52.9, 41.9; IR (KBr) ν 3382, 3060, 2951, 2360, 1741, 1633, 1593, 1496, 1454, 1434, 1336, 1296, 1244, 1169, 1125, 1045, 860, 818, 770, 731, 703, 668 cm⁻¹. HRMS: calcd. for C₃₁H₂₄NO₄⁺: 474.1700, found. 474.1689. The product was analyzed by HPLC to determine the enantiomeric excess: 97% *ee* (Chiralpak AS-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); *t_r* = 12.75 and 28.66 min.



(1*R*,3*S*)-methyl 1-benzyl-3-(furan-2-yl)-4,9-dioxo-2,3,4,9-tetrahydro-1H-benzo[f]isoindole-1-carboxylate

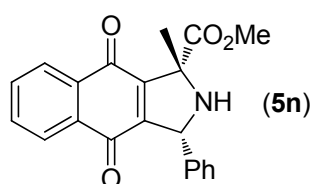
The title compound was prepared according to the general procedure as described above in 76% yield. m.p. 155-158 °C; ¹H NMR (CDCl₃, TMS, 300 MHz) δ 8.18 (d, *J* = 7.2 Hz, 1H), 7.96 (d, *J* = 7.2 Hz, 1H), 7.77-7.71 (m, 2H), 7.17 (s, 5H), 6.28 (m, 1H), 6.21 (m, 1H), 5.13 (s, 1H), 3.78 (s, 3H), 3.62 (d, *J* = 13.5 Hz, 1H), 3.45 (d, *J* = 13.5

Hz, 1H), 2.79 (br, 1H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 182.2, 181.6, 172.8, 152.9, 148.3, 146.7, 142.2, 135.5, 133.9, 132.6, 130.5, 128.1, 127.0, 126.6, 126.3, 110.6, 107.5, 75.7, 59.4, 42.0; IR (KBr) ν 3374, 2926, 1736, 1637, 1594, 1436, 1337, 1293, 1270, 1148, 1046, 770, 732, 702 cm^{-1} . HRMS: calcd. for $\text{C}_{25}\text{H}_{20}\text{NO}_5^+$: 414.1336, found. 414.1330. The product was analyzed by HPLC to determine the enantiomeric excess: 94% *ee* (Chiralpak AS-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 11.18 and 14.19 min.



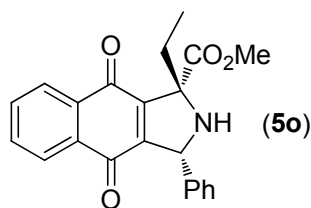
(1*R*,3*R*)-methyl 1-benzyl-3-cyclohexyl-4,9-dioxo-2,3,4,9-tetrahydro-1H-benzo[f]isoindole-1-carboxylate

The title compound was prepared according to the general procedure as described above in 70% yield. m.p. 138-141 °C; $[\alpha]_D^{25} = +83.7$ (*c* 1.28, CHCl_3); ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 8.14 (d, J = 7.2 Hz, 1H), 8.02 (d, J = 7.2 Hz, 1H), 7.77-7.73 (m, 2H), 7.14-7.13 (m, 3H), 7.05-7.03 (m, 2H), 3.81 (s, 1H), 3.77 (s, 3H), 3.59 (d, J = 13.8 Hz, 1H), 3.38 (d, J = 13.8 Hz, 1H), 2.23 (m, 1H), 1.94 (m, 1H), 1.70 (m, 1H), 1.61 (m, 3H), 1.50-1.47 (m, 1H), 1.23-1.12 (m, 4H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 182.4, 172.9, 151.1, 146.9, 135.6, 133.7, 133.6, 132.9, 132.7, 130.1, 128.2, 127.0, 126.4, 74.8, 68.5, 52.6, 41.6, 41.0, 30.6, 26.5, 26.2, 26.1, 26.0; IR (KBr) ν 3358, 2977, 1735, 1594, 1424, 1215, 1047, 878, 773, 669, 626 cm^{-1} . HRMS: calcd. for $\text{C}_{27}\text{H}_{28}\text{NO}_4^+$: 430.2018, found. 430.2012. The product was analyzed by HPLC to determine the enantiomeric excess: 94% *ee* (Chiralcel AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min, λ = 220 nm); t_r = 7.28 and 10.16 min.



(1*R*,3*R*)-methyl 1-methyl-4,9-dioxo-3-phenyl-2,3,4,9-tetrahydro-1*H*-benzo[*f*]isoindole-1-carboxylate

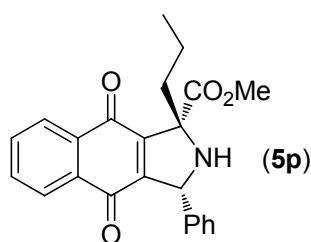
The title compound was prepared according to the general procedure as described above in 82% yield. m.p. 95-98 °C; $[\alpha]_D^{25} = +31.5$ (*c* 1.20, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 8.11 (d, *J* = 8.4 Hz, 1H), 7.97 (d, *J* = 8.4 Hz, 1H), 7.74-7.70 (m, 2H), 7.44 (d, *J* = 6.9 Hz, 2H), 7.37-7.27 (m, 3H), 5.69 (s, 1H), 3.79 (s, 3H), 2.60 (br, 1H), 1.81 (s, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 182.2, 181.8, 172.9, 148.8, 148.4, 141.0, 133.7, 132.8, 128.5, 127.9, 127.6, 126.3, 71.0, 66.5, 52.7, 24.4; IR (KBr) ν 3368, 2951, 1740, 1634, 1593, 1492, 1454, 1372, 1332, 1267, 1171, 1106, 1027, 901, 776, 730, 716, 704, 641, 554 cm⁻¹. HRMS: calcd. for C₂₁H₁₈NO₄⁺: 348.1230, found. 348.1220. The product was analyzed by HPLC to determine the enantiomeric excess: 89% *ee* (Chiralcel OD-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min, λ = 220 nm); *t_r* = 17.20 and 30.03 min.



(1*R*,3*R*)-methyl 1-ethyl-4,9-dioxo-3-phenyl-2,3,4,9-tetrahydro-1*H*-benzo[*f*]isoindole-1-carboxylate

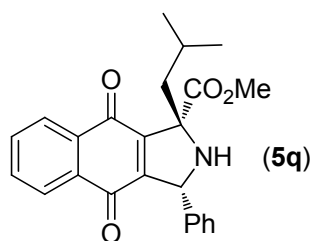
The title compound was prepared according to the general procedure as described above in 90% yield. m.p. 102-105 °C; $[\alpha]_D^{25} = +5.5$ (*c* 1.40, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 8.12 (d, *J* = 7.2 Hz, 1H), 7.97 (d, *J* = 7.2 Hz, 1H), 7.74-7.70 (m, 2H), 7.42-7.30 (m, 5H), 5.64 (s, 1H), 3.79 (s, 3H), 2.50-2.70 (br, 1H), 2.35-2.31 (m, 1H), 2.22-2.17 (m, 1H), 0.96-0.91 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 182.0, 181.9, 173.3, 150.3, 146.4, 141.4, 133.7, 132.9, 132.8, 128.6, 128.0, 127.6, 126.4, 126.3, 75.7, 67.3, 52.7, 29.6, 8.0; IR (KBr) ν 3374, 2966, 1736, 1632, 1594, 1492, 1456, 1368, 1334, 1290, 1261, 1170, 1082, 1026, 772, 742, 715, 701 cm⁻¹. HRMS: calcd. for C₂₂H₂₀NO₄⁺: 362.1387, found. 362.1382. The product was analyzed by HPLC to determine the enantiomeric excess: 94% *ee* (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min, λ = 220 nm); *t_r* = 17.17 and 33.97

min.



**(1R,3R)-methyl 4,9-dioxo-3-phenyl-1-propyl-2,3,4,9-tetrahydro-1H-benzo[f]
isoindole-1-carboxylate**

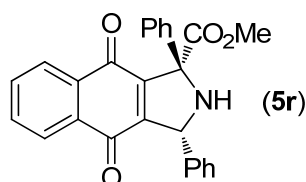
The title compound was prepared according to the general procedure as described above in 77% yield. m.p. 91-94 °C; $[\alpha]_D^{25} = +6.3$ (*c* 1.20, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 8.11 (d, *J* = 7.2 Hz, 1H), 7.96 (d, *J* = 7.2 Hz, 1H), 7.77-7.67 (m, 2H), 7.41-7.29 (m, 5H), 5.63 (s, 1H), 3.78 (s, 3H), 2.61 (br, 1H), 2.39-2.24 (m, 1H), 2.15-2.05 (m, 1H), 1.52-1.45 (m, 1H), 1.23-1.14 (m, 1H), 0.98-0.93 (t, *J* = 7.2 Hz, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 182.0, 181.9, 173.3, 150.0, 146.7, 141.3, 133.7, 132.9, 132.8, 128.6, 128.0, 127.6, 126.4, 126.3, 75.2, 67.3, 52.7, 38.9, 17.1, 14.2; IR (KBr) ν 3375, 3065, 3030, 2958, 2929, 2872, 1736, 1633, 1594, 1493, 1455, 1434, 1368, 1333, 1289, 1249, 1170, 1109, 1044, 1028, 941, 777, 747, 715, 701, 645, 574 cm⁻¹. HRMS: calcd. for C₂₃H₂₂NO₄⁺: 376.1530, found. 376.1525. The product was analyzed by HPLC to determine the enantiomeric excess: 90% *ee* (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min, λ = 220 nm); *t_r* = 23.21 and 43.83 min.



**(1R,3R)-methyl 1-isobutyl-4,9-dioxo-3-phenyl-2,3,4,9-tetrahydro-1H-benzo[f]
isoindole-1-carboxylate**

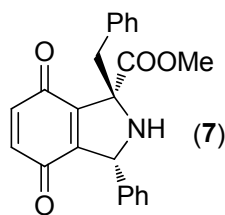
The title compound was prepared according to the general procedure as described above in 74% yield. m.p. 112-115 °C; $[\alpha]_D^{25} = +26.5$ (*c* 1.56, CHCl₃); ¹H NMR (CDCl₃, TMS, 300 MHz) δ 8.12 (d, *J* = 9.0 Hz, 1H), 7.97 (d, *J* = 9.0 Hz, 1H),

7.77-7.70 (m, 2H), 7.41-7.29 (m, 5H), 5.65 (s, 1H), 3.77 (s, 3H), 2.61 (br, 1H), 2.25-2.11 (m, 2H), 1.78-1.74 (m, 1H), 1.02 (d, $J = 6.6$ Hz, 3H), 0.86 (d, $J = 6.6$ Hz, 3H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 182.1, 173.5, 149.9, 147.2, 141.3, 133.8, 133.7, 133.0, 132.8, 128.6, 128.0, 127.6, 126.5, 126.3, 75.3, 67.0, 52.7, 44.8, 24.5, 24.4, 24.3; IR (KBr) ν 3380, 2954, 1735, 1668, 1631, 1594, 1492, 1455, 1367, 1329, 1220, 1169, 1125, 1029, 715, 701 cm^{-1} . HRMS: calcd. for $\text{C}_{24}\text{H}_{24}\text{NO}_4^+$: 390.1700, found. 390.1703. The product was analyzed by HPLC to determine the enantiomeric excess: 93% *ee* (Chiralpak AS-H, *i*-propanol/hexane = 30/70, flow rate 1.0 mL/min, λ = 220 nm); t_r = 7.77 and 26.04 min.



(1R,3R)-methyl 4,9-dioxo-1,3-diphenyl-2,3,4,9-tetrahydro-1H-benzo[f]isoindole-1-carboxylate

The title compound was prepared according to the general procedure as described above in 86% yield. m.p. 120-123 °C; $[\alpha]_D^{25} = +48.6$ (c 0.42, CHCl_3); ^1H NMR (CDCl_3 , TMS, 400 MHz) δ 8.03 (d, $J = 7.2$ Hz, 1H), 7.92 (d, $J = 7.2$ Hz, 1H), 7.67-7.64 (m, 4H), 7.45 (d, $J = 7.6$ Hz, 2H), 7.40-7.24 (m, 6H), 5.84 (s, 1H), 3.85 (s, 3H); ^{13}C NMR (CDCl_3 , TMS, 100 MHz) δ 182.3, 182.2, 172.4, 148.7, 147.5, 140.9, 140.2, 133.8, 133.6, 132.9, 132.6, 129.7, 128.7, 128.2, 128.0, 127.6, 127.2, 126.5, 126.1, 76.6, 67.3, 53.0; IR (KBr) ν 3384, 3065, 3030, 2958, 1736, 1669, 1593, 1457, 1219, 1070, 754, 698 cm^{-1} . HRMS: calcd. for $\text{C}_{26}\text{H}_{20}\text{NO}_4^+$: 410.1387, found. 410.1377. The product was analyzed by HPLC to determine the enantiomeric excess: 93% *ee* (Chiralpak AD-H, *i*-propanol/hexane = 15/85, flow rate 1.0 mL/min, λ = 220 nm); t_r = 31.13 and 34.41 min.



(1*R*,3*R*)-methyl 1-benzyl-4,7-dioxo-3-phenyl-2,3,4,7-tetrahydro-1*H*-isoindole-1-carboxylate

The title compound was prepared according to the general procedure as described above in 82% yield. $[\alpha]_D^{25} = +53.7$ (c 0.48, CHCl_3); ^1H NMR (CDCl_3 , TMS, 300 MHz) δ 7.26 (m, 3H), 7.12 (m, 2H), 6.71 (d, $J = 10.2$ Hz, 1H), 6.53 (d, $J = 10.2$ Hz, 1H), 4.80 (s, 1H), 3.83 (s, 3H), 3.49 (d, $J = 14.1$ Hz, 1H), 3.37 (d, $J = 14.1$ Hz, 1H), 2.66 (br, 1H); ^{13}C NMR (CDCl_3 , TMS, 75 MHz) δ 184.7, 183.8, 172.5, 148.1, 143.9, 140.8, 136.8, 136.6, 135.4, 130.2, 128.4, 128.2, 127.9, 127.5, 127.1, 75.3, 66.7, 52.9, 41.9; IR (KBr) ν 2956, 1736, 1669, 1593, 1452, 1215, 1047, 669, cm^{-1} . HRMS: calcd. for $\text{C}_{23}\text{H}_{22}\text{NO}_4^+$: 376.1549, found. 376.1530. The product was analyzed by HPLC to determine the enantiomeric excess: 86% *ee* (Chiralpak AS-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min, $\lambda = 220$ nm); $t_r = 14.89$ and 30.87 min.

IV. The Absolute Configuration Determination of (1*R*,3*R*)-5b

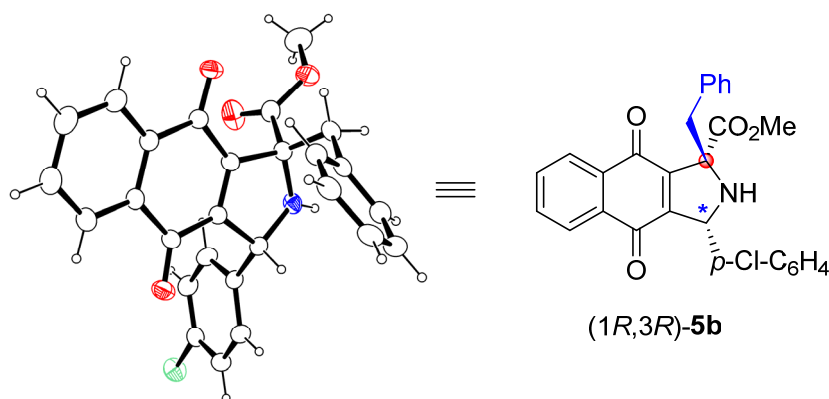
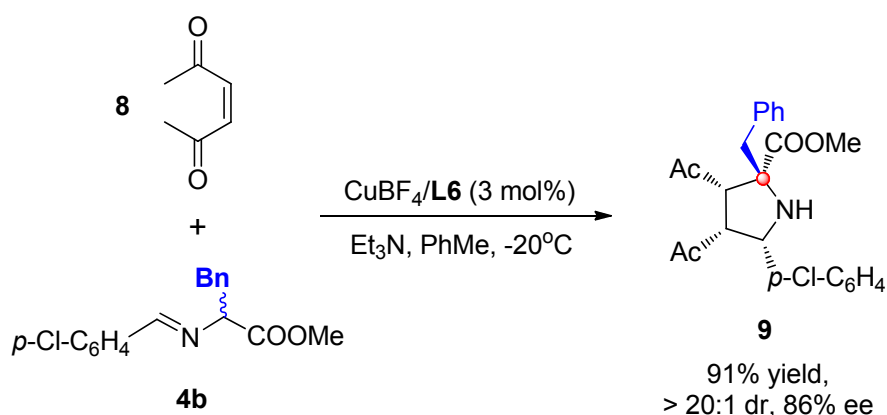


Figure 1. X-ray structure of (1*R*,3*R*)-5b.

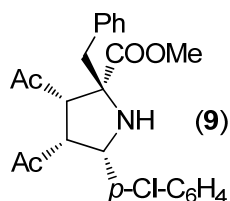
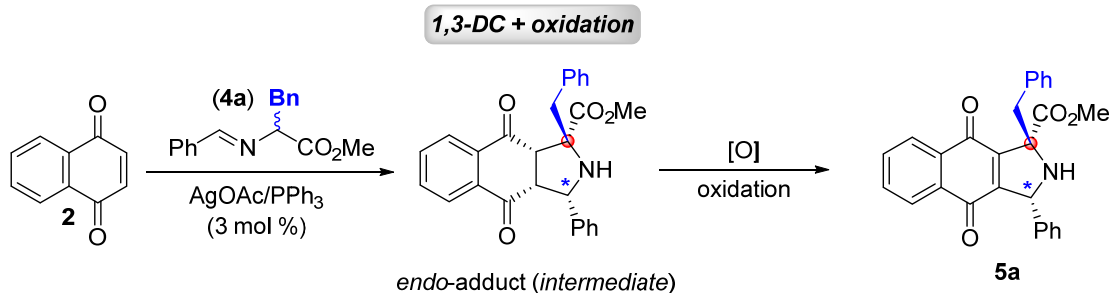
Crystal data for (1*R*,3*R*)-5b: $\text{C}_{27}\text{H}_{22}\text{ClNO}_4$, $M_r = 459.91$, $T = 293$ K, tetragonal, space group $P4(3)$, $a = 12.7827(8)$, $b = 12.7827(8)$, $c = 13.7419(18)$ Å, $V = 2245.4(4)$ Å³, $Z = 4$, 3391 unique reflections, final $R_1 = 0.0320$ and $wR_2 = 0.0806$ for 4129 observed [$I > 2\sigma(I)$] reflections, Flack $\chi = -0.04(6)$. CCDC 904693 contains the

supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB21EZ, UK; fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk).

V. Proposed Relative Configuration of the Labile Intermediate in This One-Pot Sequential Catalytic Asymmetric 1,3-DC/Oxidation



To further investigate the relative configuration of the labile intermediate of this one-pot sequential catalytic asymmetric 1,3-DC/oxidation reaction, (Z)-hex-3-ene-2,5-dione **8** was employed as the dipolarophile and **4b** was employed as imino ester to study the stereochemistry of the 1,3-dipolar cycloaddition under the optimized reaction condition. As expected, the normal 1,3-DC adduct **9** was obtained in 91% yield with excellent diastereoselectivity and 86% *ee* (Scheme 1), and the relative configuration of racemic adduct **9** was determined unambiguously to be *endo* by single X-ray crystallographic analysis (Figure 2). Hence, the relative configuration of the labile intermediate in this one-pot sequential 1,3-DC/oxidation reaction were tentatively proposed to be *endo* on the basis of these results.



(2R,3S,4R,5S)-methyl 3,4-diacetyl-2-benzyl-5-(4-chlorophenyl)pyrrolidine-2-carboxylate

Under argon atmosphere, (*S,R_p*)-PPFOMe (3.1 mg, 0.0072 mmol) and Cu(CH₃CN)₄BF₄ (1.9 mg, 0.006 mmol) were dissolved in toluene (2mL), and stirred at room temperature for about 1 h. Then, imine substrate **4b** (78.3 mg, 0.26 mmol), and (*Z*)-hex-3-ene-2,5-dione (22.4 mg, 0.2 mmol) were added sequentially, after that the mixture was dropped to -20 °C, TEA (3 mg, 0.03 mmol) was added. Once starting material was consumed (monitored by TLC), the residue was purified by column chromatography to give **9** in 91% yield, which was then directly analyzed by chiral HPLC to determine the enantiomeric excess. ¹H NMR (CDCl₃, TMS, 300 MHz) δ 7.38-7.26 (m, 7H), 7.17 (d, *J* = 8.4 Hz, 2H), 4.16 (d, *J* = 6.0 Hz, 1H), 3.75 (s, 3H), 3.43-3.37 (m, 2H), 3.33 (d, *J* = 13.5 Hz, 1H), 3.15 (d, *J* = 13.5 Hz, 1H), 2.48 (s, 3H), 1.66 (s, 3H); ¹³C NMR (CDCl₃, TMS, 75 MHz) δ 208.4, 205.2, 173.8, 135.9, 135.7, 133.7, 130.5, 128.7, 128.4, 128.3, 127.3, 73.4, 64.6, 62.6, 59.1, 52.4, 45.4, 32.3, 31.3; HRMS: calcd. for C₂₃H₂₅ClNO₄⁺: 414.1467, found. 414.1446. The product was analyzed by HPLC to determine the enantiomeric excess: 86% *ee* (Chiralpak AD-H, *i*-propanol/hexane = 20/80, flow rate 1.0 mL/min, λ = 220 nm); t_r = 8.16 and 10.99 min.

VI. The Relative Configuration Determination of Racemic *endo*-9

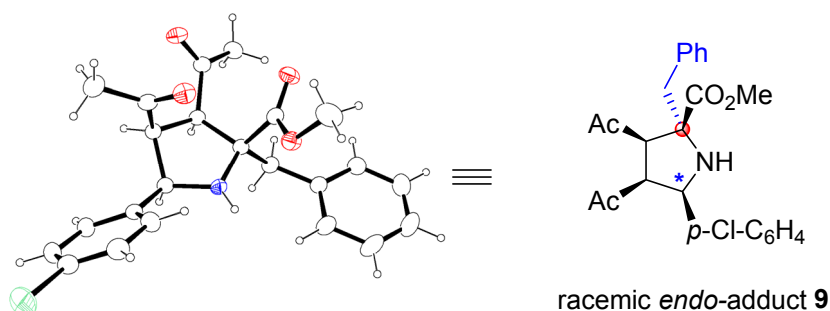


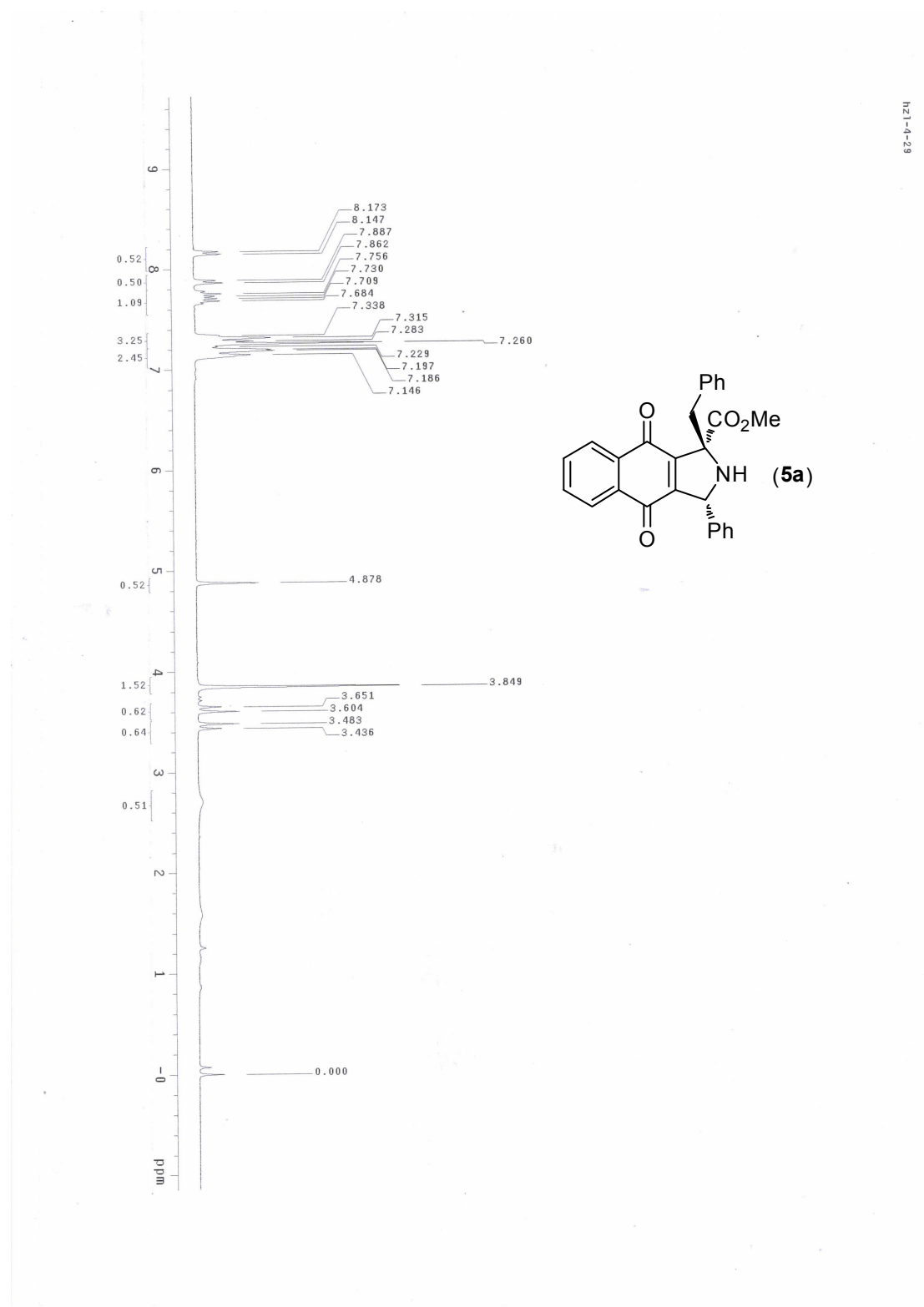
Figure 2. X-ray structure of racemic *endo*-9.

Crystal data for racemic *endo*-adduct **9**: $C_{23}H_{24}ClNO_4$, $M_r = 413.88$, $T = 293$ K, Monoclinic, space group $P2(1)/c$, $a = 8.9427(14)$, $b = 24.682(4)$, $c = 9.8606(16)$ Å, $V = 2156.1(6)$ Å³, $Z = 4$, 3082 unique reflections, final $R_1 = 0.0450$ and $wR_2 = 0.1077$ for 4233 observed [$I > 2\sigma(I)$] reflections. CCDC 904694 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB21EZ, UK; fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk).

VII. References

1. Hayashi, T; Fukushima, T, M; Kagotani, M; Nagashima, N; Hamada, Y; Matsumoto, A; Kawakami, S; Konishi, M; Yamamoto, K; Kumada, M. *Bull. Chem. Soc. Jpn.*, **1980**, 53, 1138.
2. CCDC 904693 (**5b**) and CCDC 904694 (**9**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB21EZ, UK; fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk).

VIII. ^1H NMR and ^{13}C NMR Spectra



C13H21-4-29

Solvent: CDCl₃
Ambient temperature
Mercury-300BB "mercury300"

Relax. delay 1.000 sec

Acq. 128.0 deg/sec

Acq. 128.0 deg/sec

Width 17699.1 Hz

200 Repetitions

OBSERVE C13, 75.4553150 MHz

Power 40 dB, 500.0615362 MHz

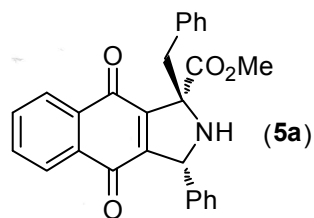
Continuously on

WALTZ-16 modulated

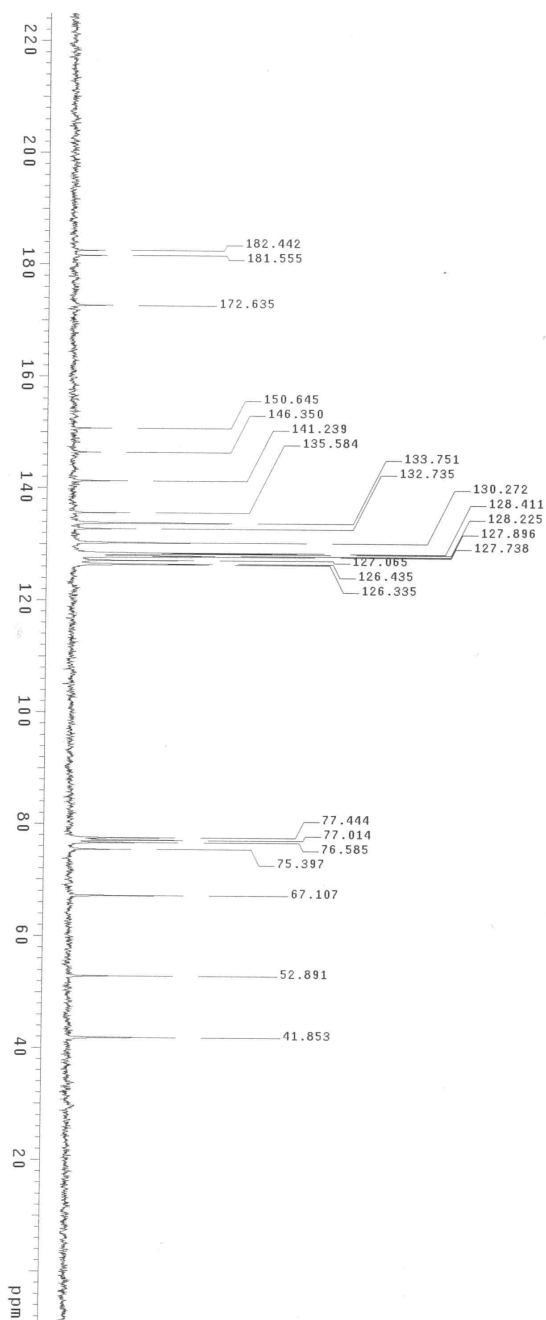
DATA PROCESSING

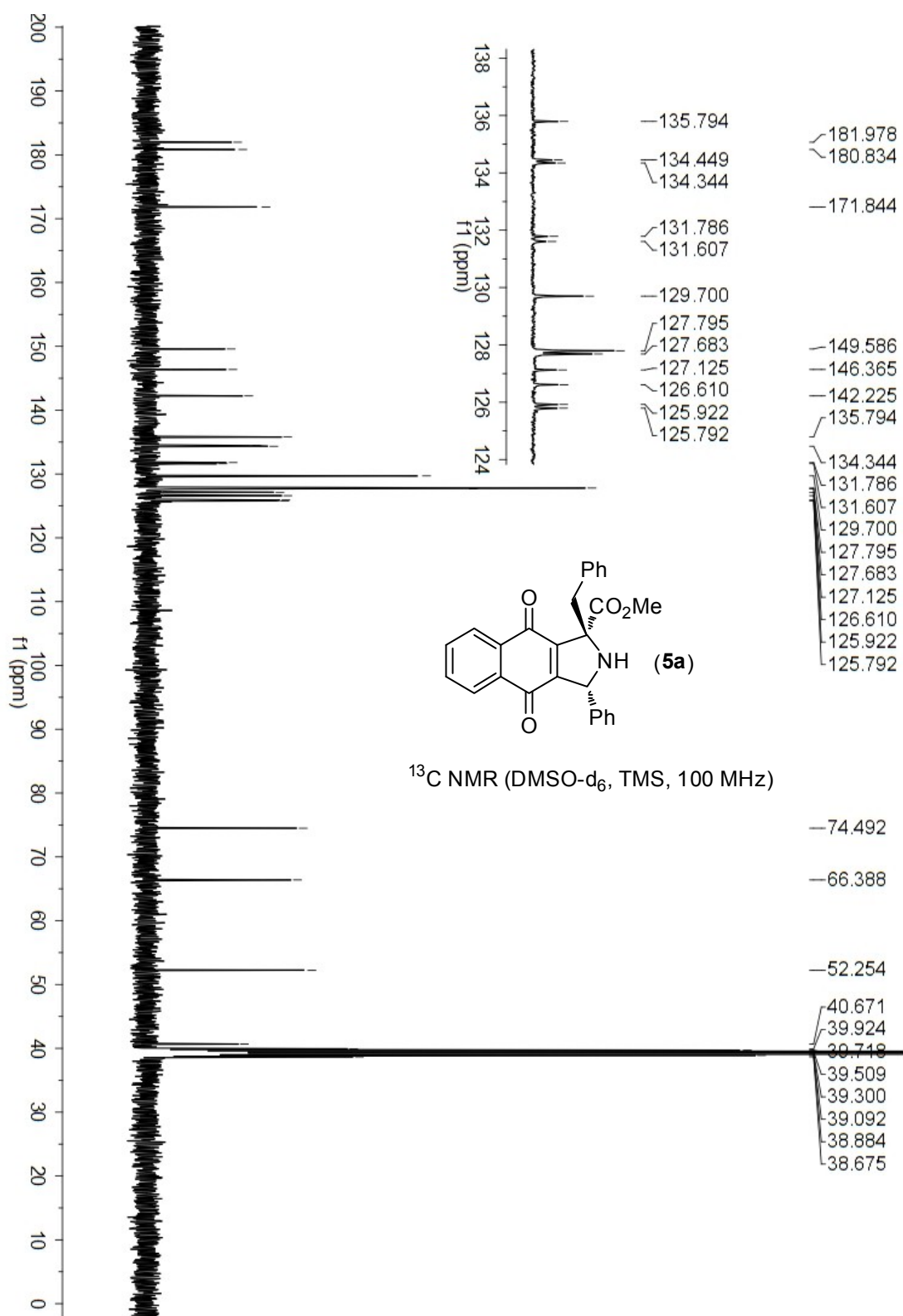
FT size 32768

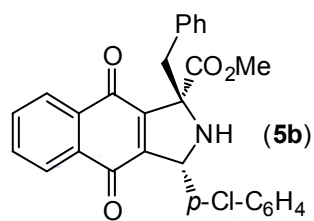
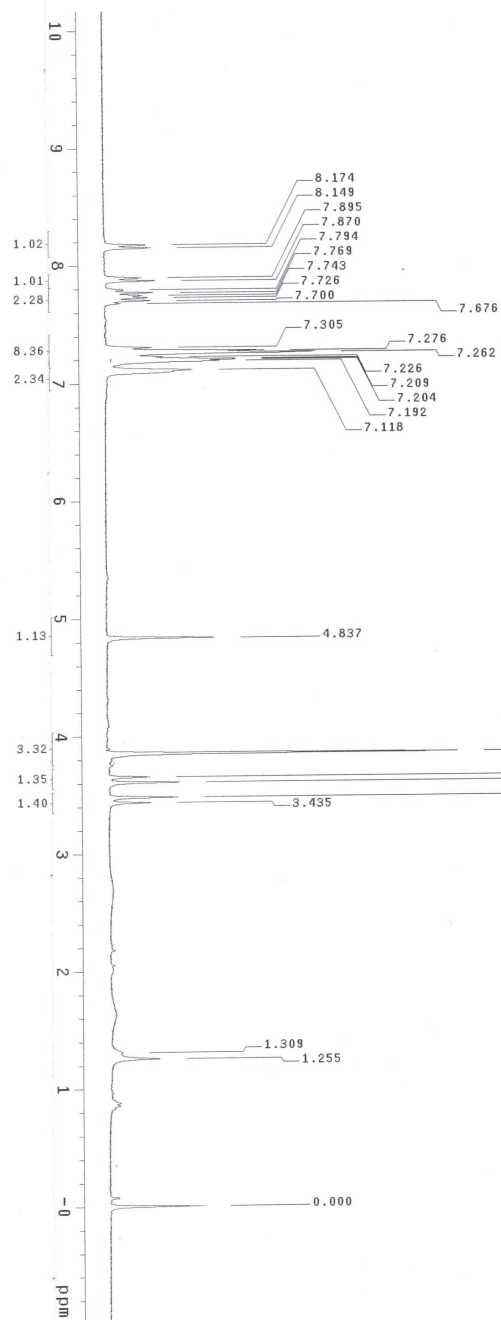
Total time 2 hr, 24 min, 23 sec



¹³C NMR (CDCl₃, TMS, 75 MHz)

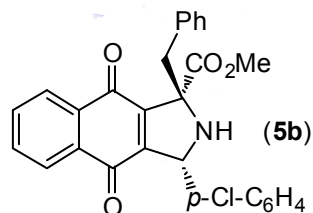
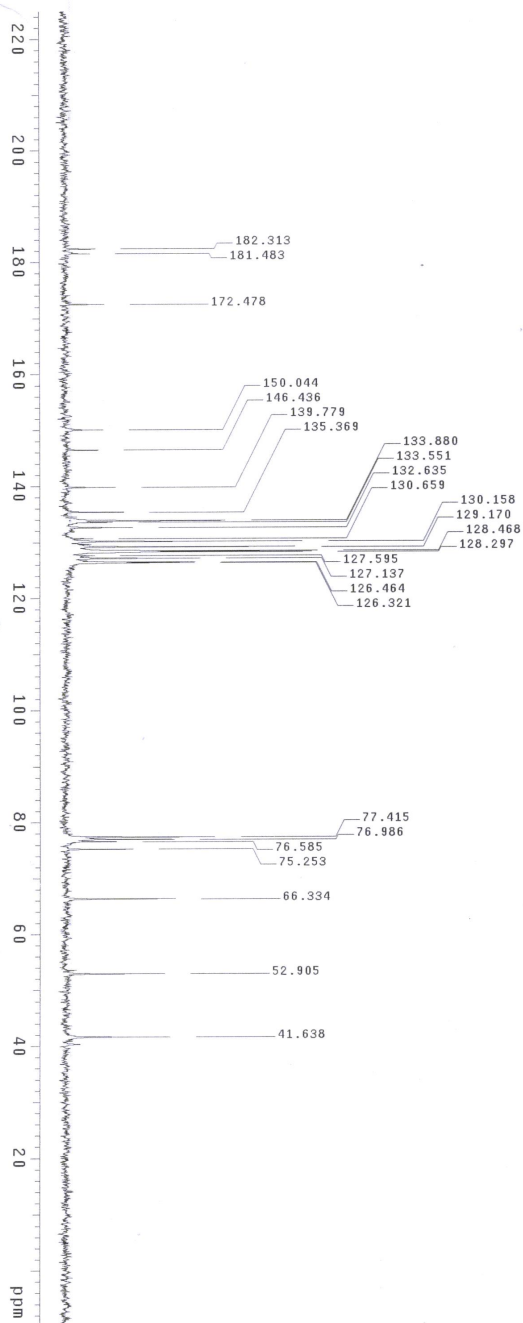


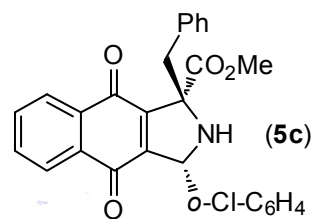
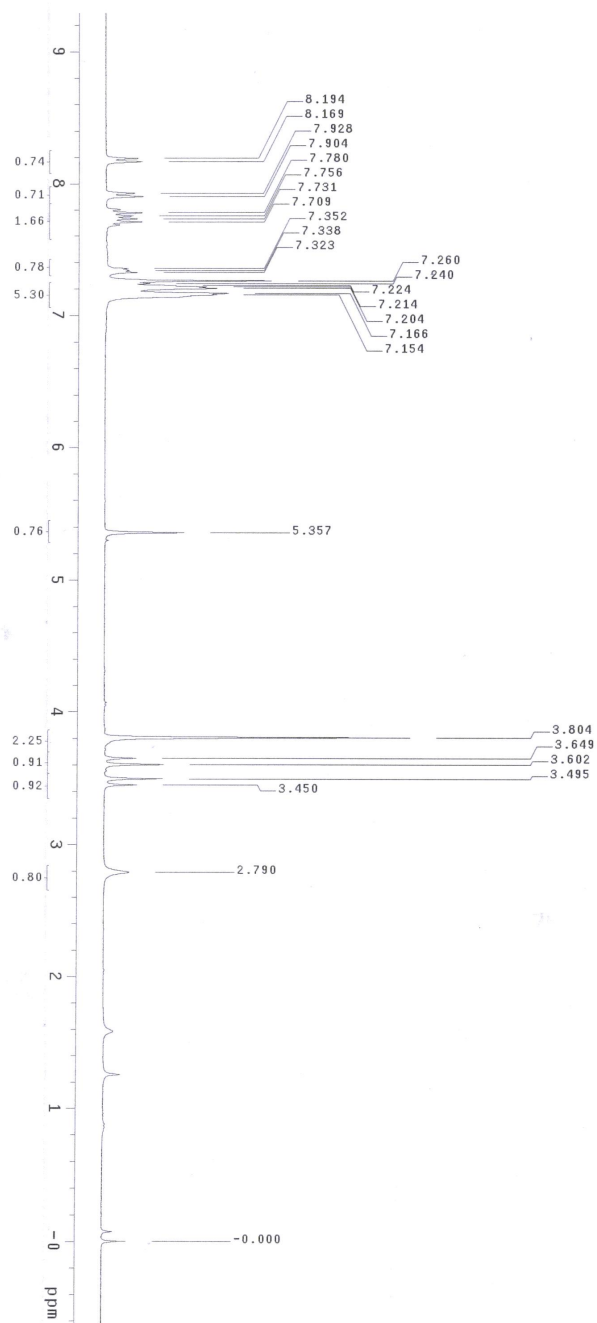




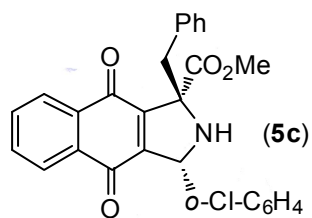
0172-b-120

Solvent: CDCl₃
 Acquisition: 13C NMR
 Mercury-3008B "mercury300"
 Relax. delay 1.000 sec
 Pulse 28.0 degrees
 Acq. time 0.500 sec
 Shift 125.763 MHz
 128 Repetitions
 OBSERVE C13, 75.453118 MHz
 DECOUPLE H1, 300.0815382 MHz
 Power 40.0 dB
 Continuity on
 WAITZ-16 modulated
 DATA PROCESSING
 Line broadening 4.0 Hz
 F2 size 32768
 Total time 2 hr, 24 min, 23 sec

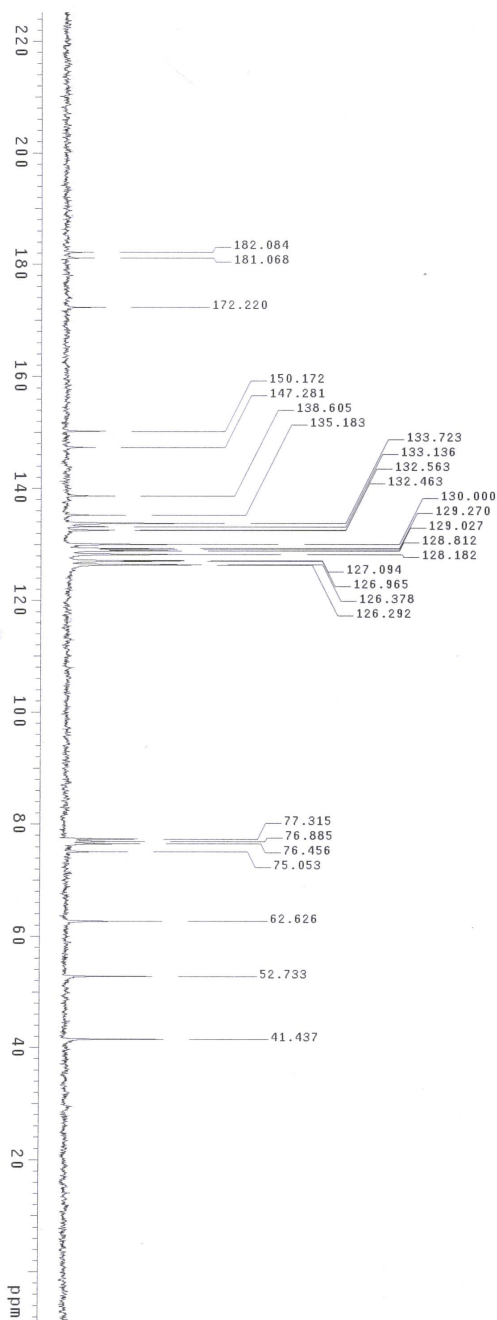


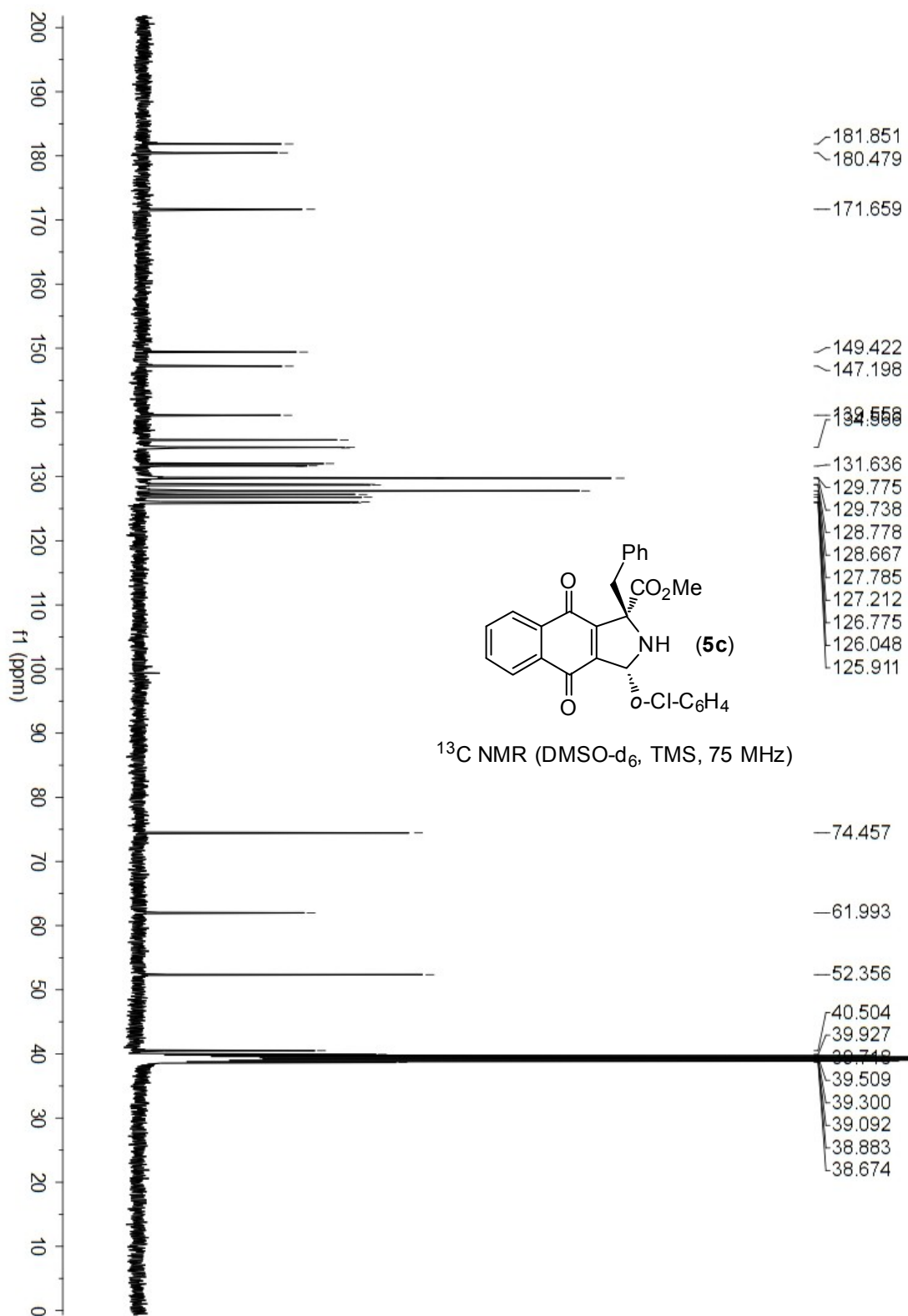


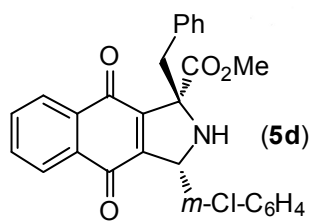
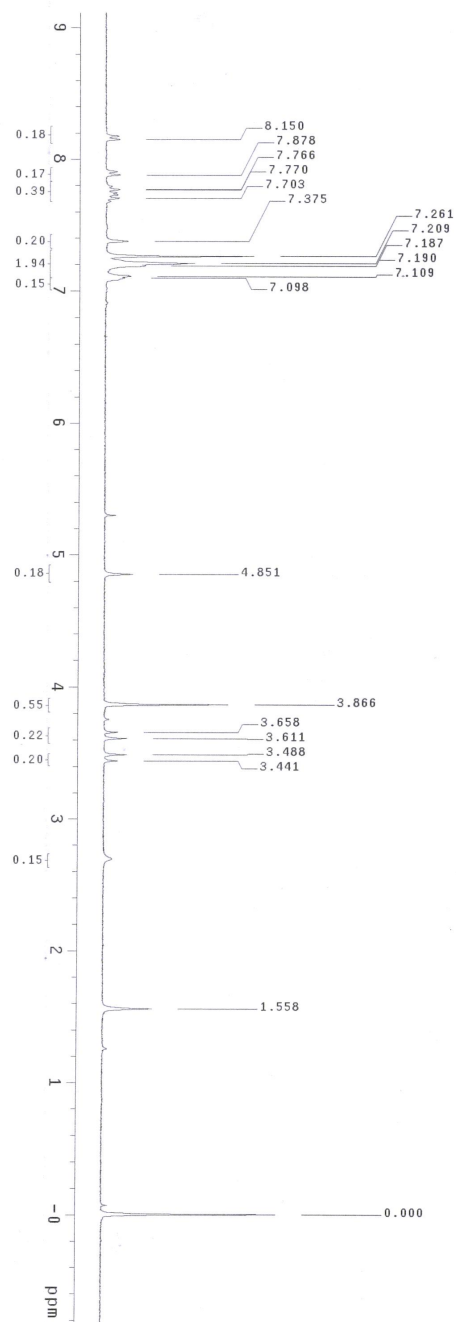
Solvent: CDCl₃
 Acquisition temperature: 29.9 °C
 Mercury-500MHz
 Relax. delay: 1.000 sec
 Pulse: 29.0 degrees
 Acq. time: 0.500 sec
 Nuclei: 13C
 144 repetitions
 OBSERVE: C13, 75.4552934 MHz
 DECOUPLE: H1, 300.0815382 MHz
 13C, 75.4552934 MHz
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 4.0 Hz
 F2 size 228
 Total time 2 hr, 24 min, 23 sec



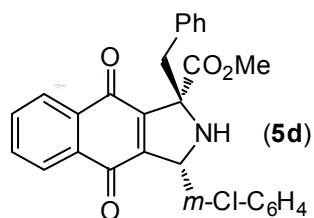
¹³C NMR (CDCl₃, TMS, 75 MHz)



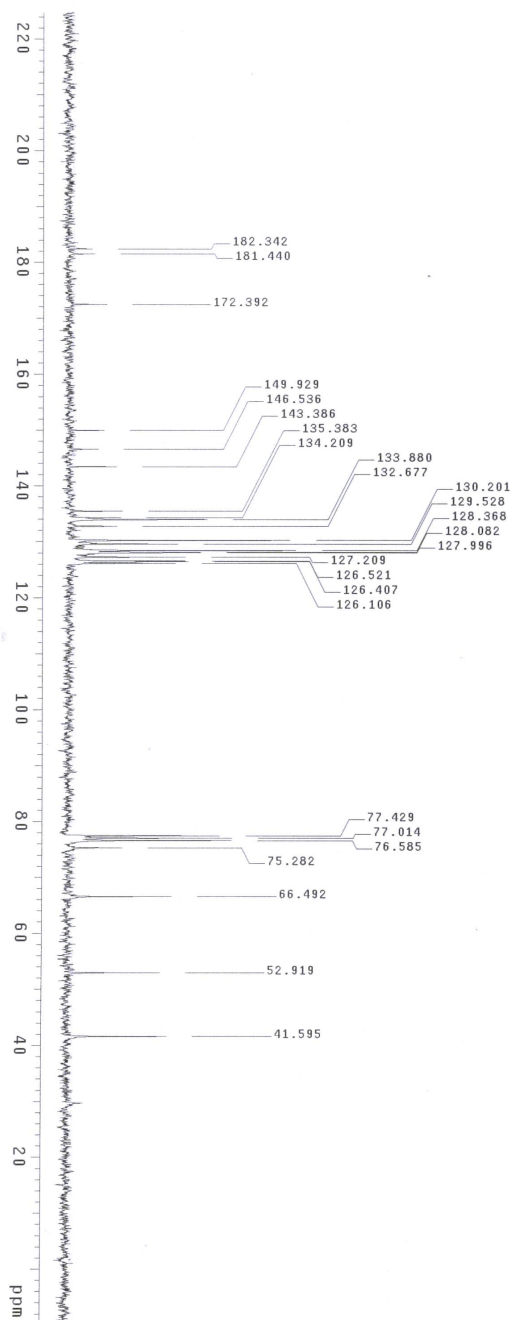


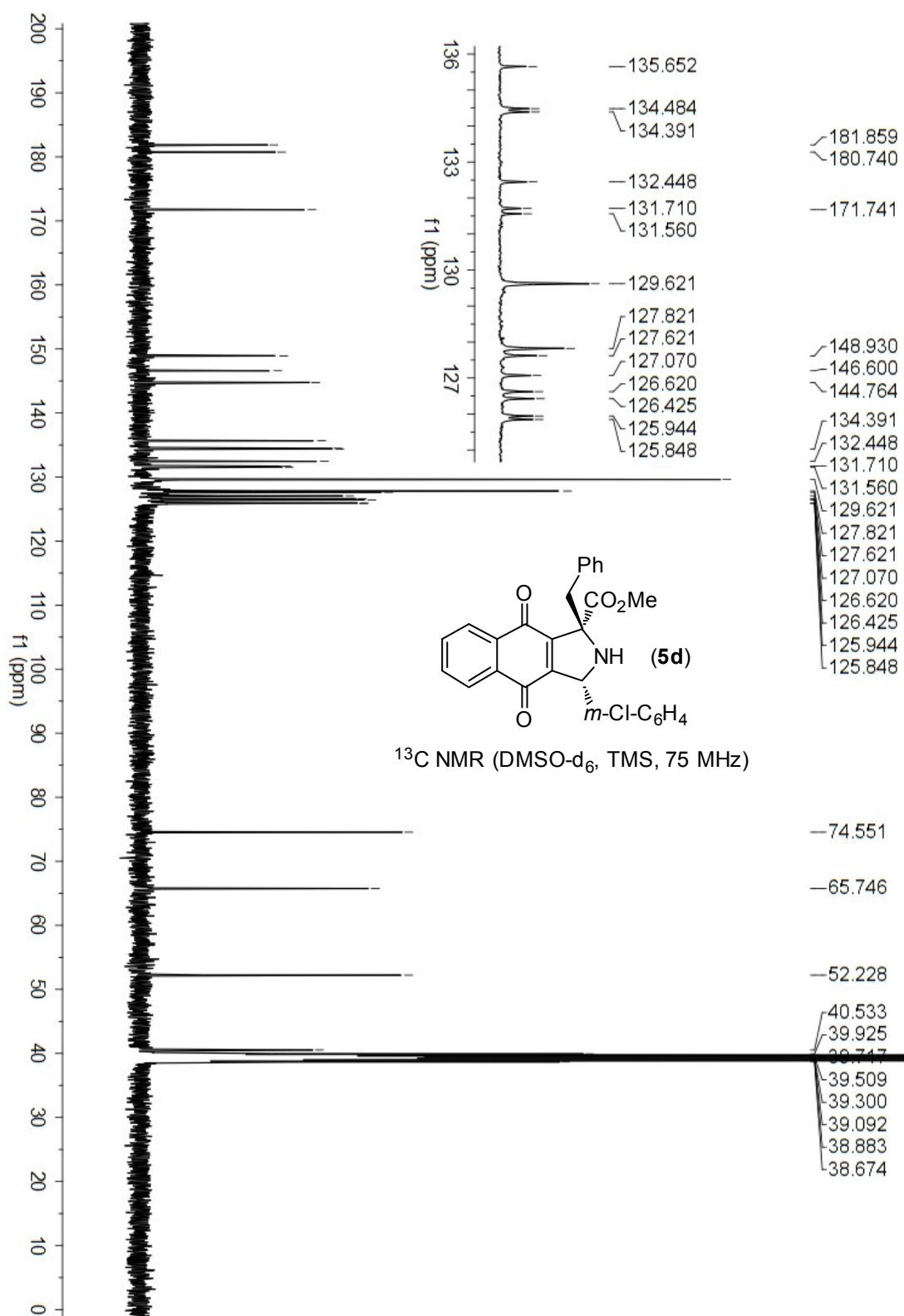


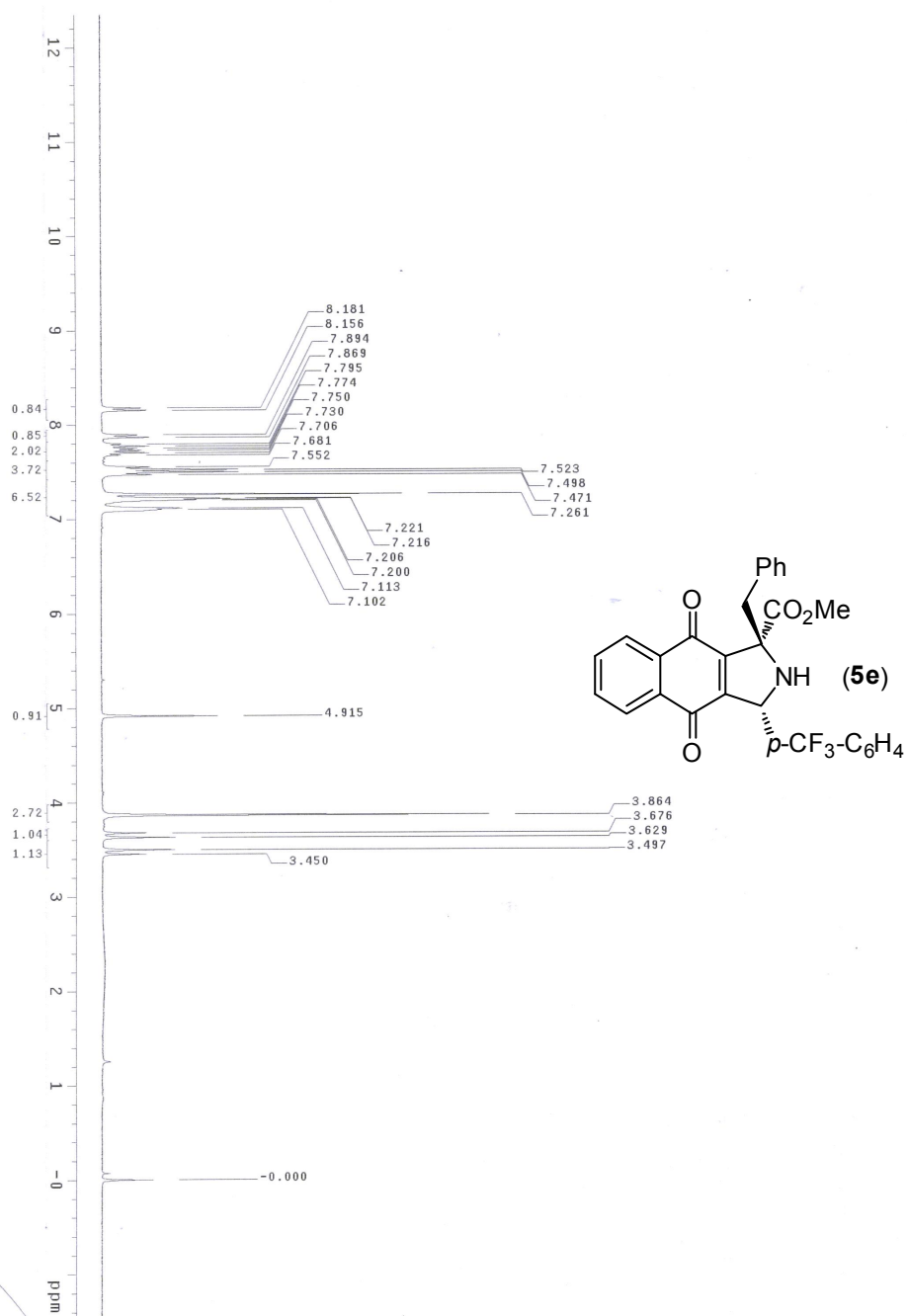
Solvent: CDCl₃
 Acquisition Date: 11/11/2008
 Mercury-300BB "mercury300"
 Relax. delay: 1.000 sec
 Pulse: 28.0 degrees
 Acq. time: 0.500 sec
 Observed: 126.913 MHz
 208 F₂ pulses
 OBSERVE: C13, 75.4553247 MHz
 DECOUPLE: H1, 300.0815382 MHz
 Power: 40 dB
 Presat: 100% on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening: 4.0 Hz
 FT size: 32768
 Total time: 2 hr, 24 min, 23 sec

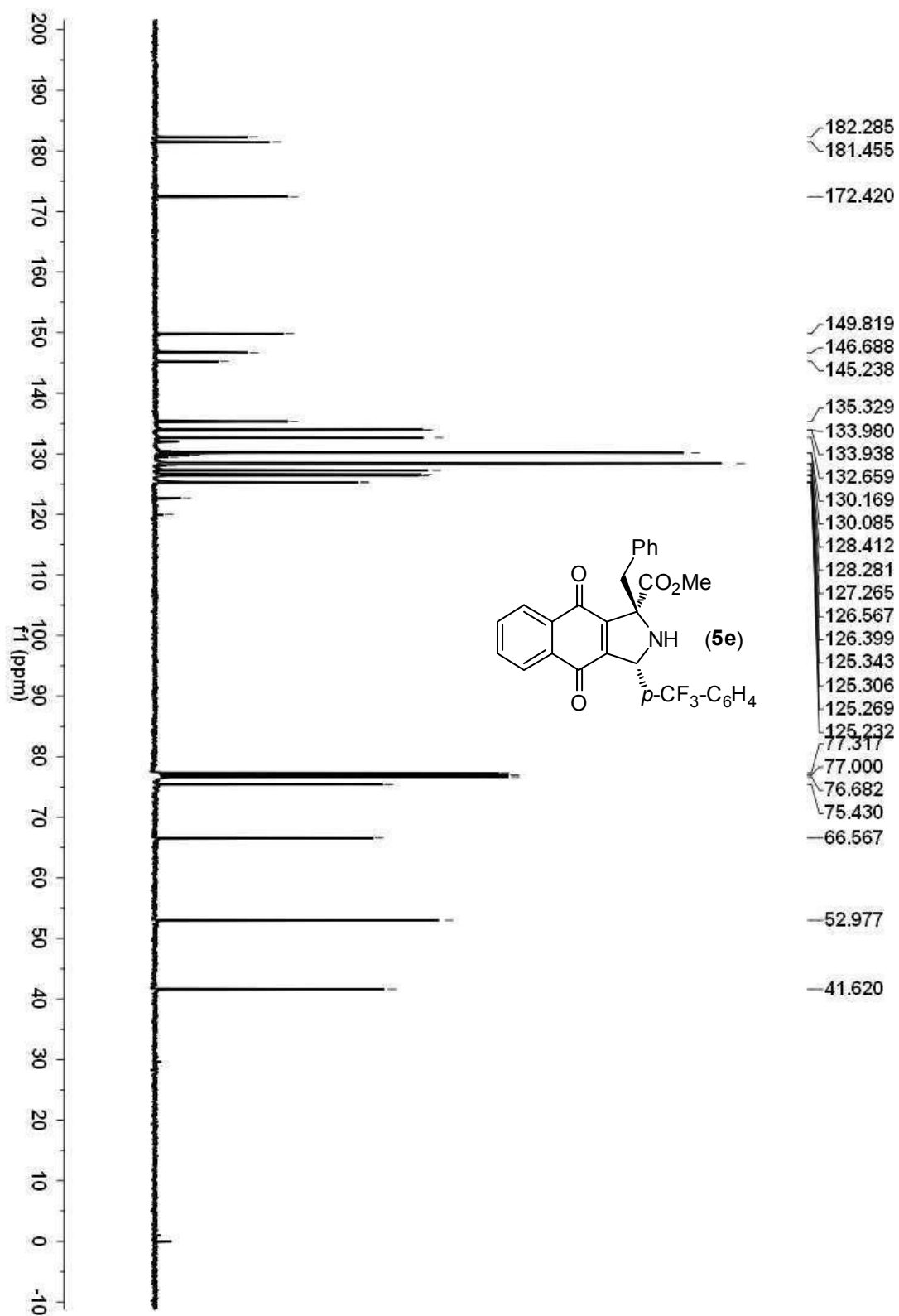


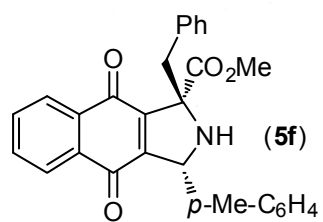
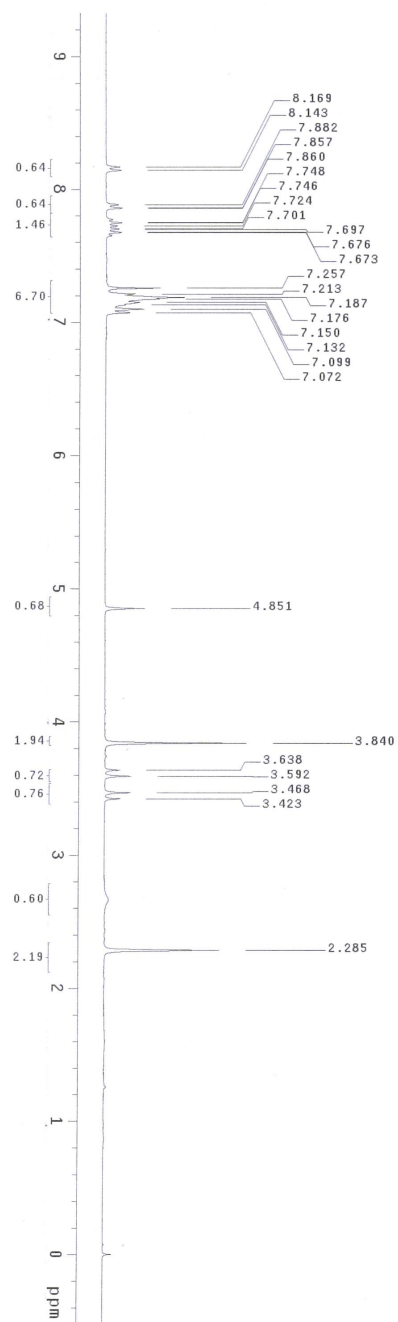
¹³C NMR (CDCl₃, TMS, 75 MHz)



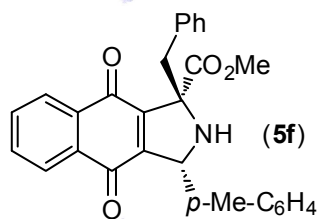
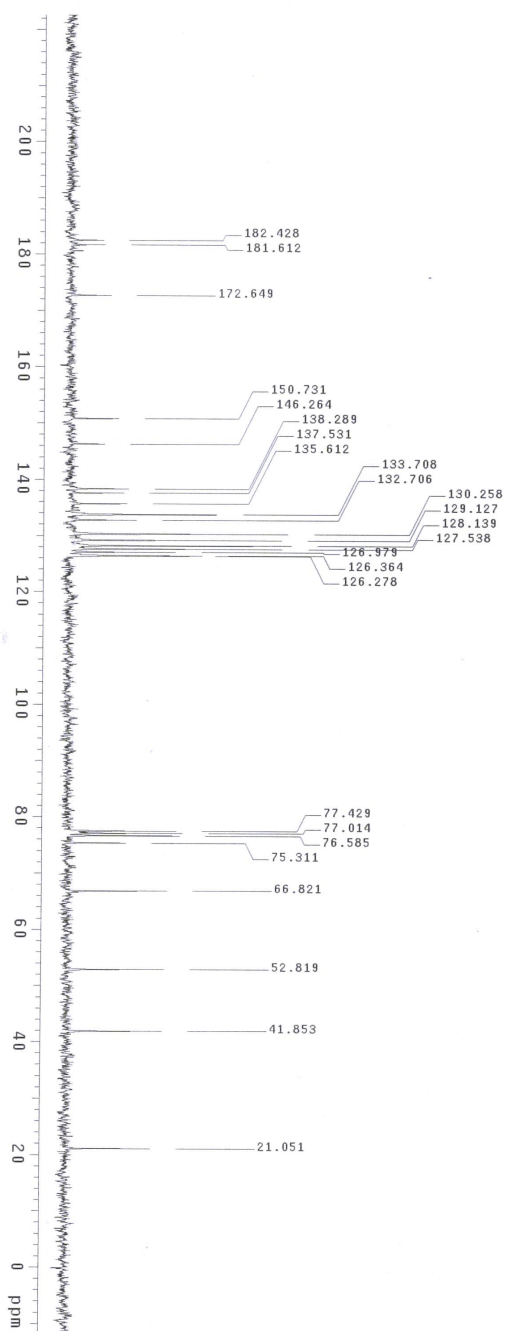


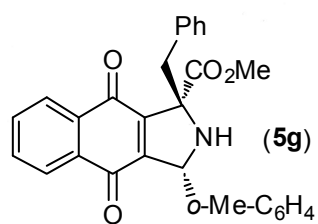
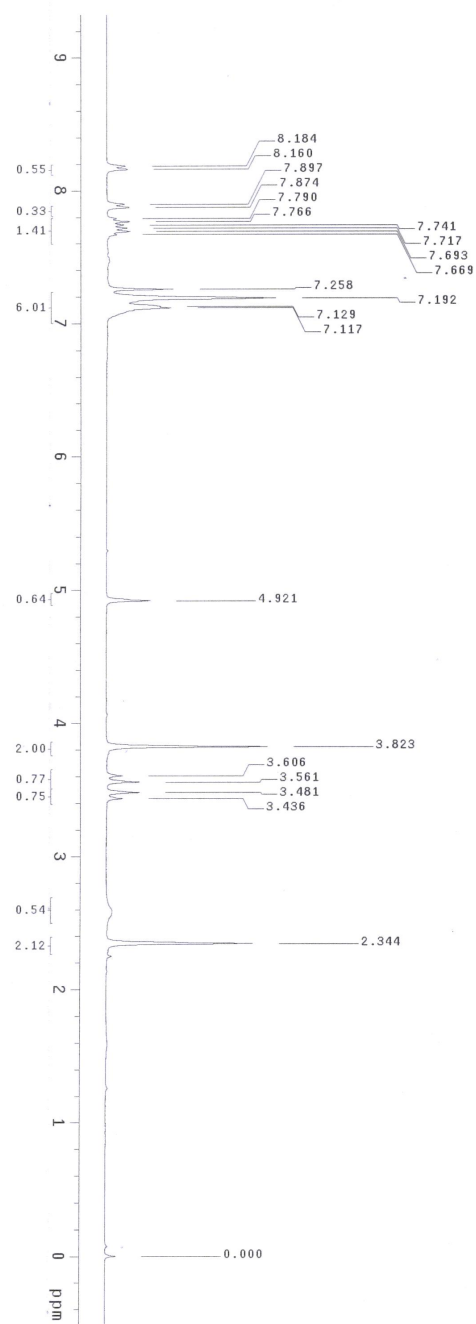




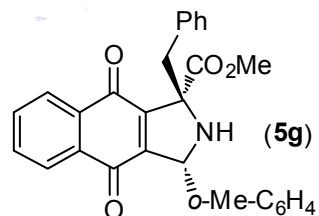
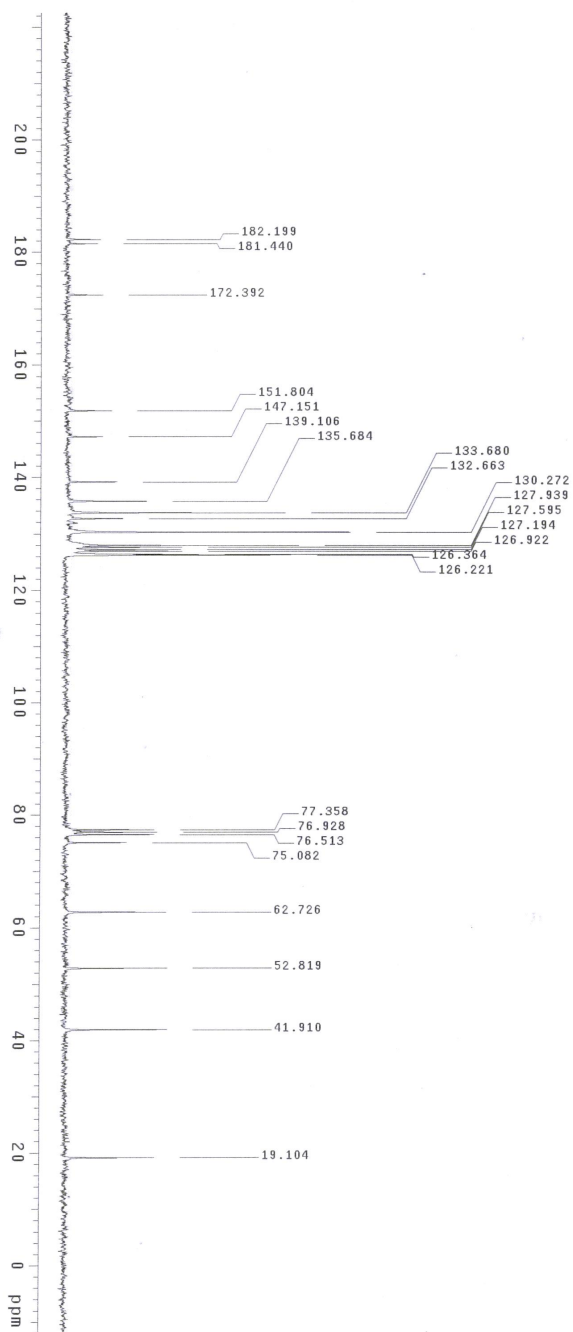


Solvent: CDCl₃
 Acquisition Date: 12/11/2008
 Mercury-300BB "mercury300"
 Relax. delay 1.000 sec
 Pulse 28.0 degrees
 Acq. time 0.500 sec
 F1 300.136389 MHz
 56 repetitions
 OBSERVE C13, 75.4554921 MHz
 DECOUPLE H1, 300.10815382 MHz
 Power 40.00 dB
 Locking on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 4.0 Hz
 F1 size 32768
 Total time 2 hr, 24 min, 23 sec

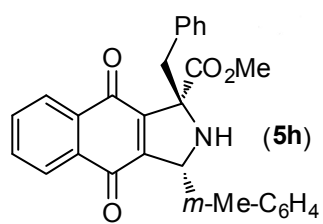
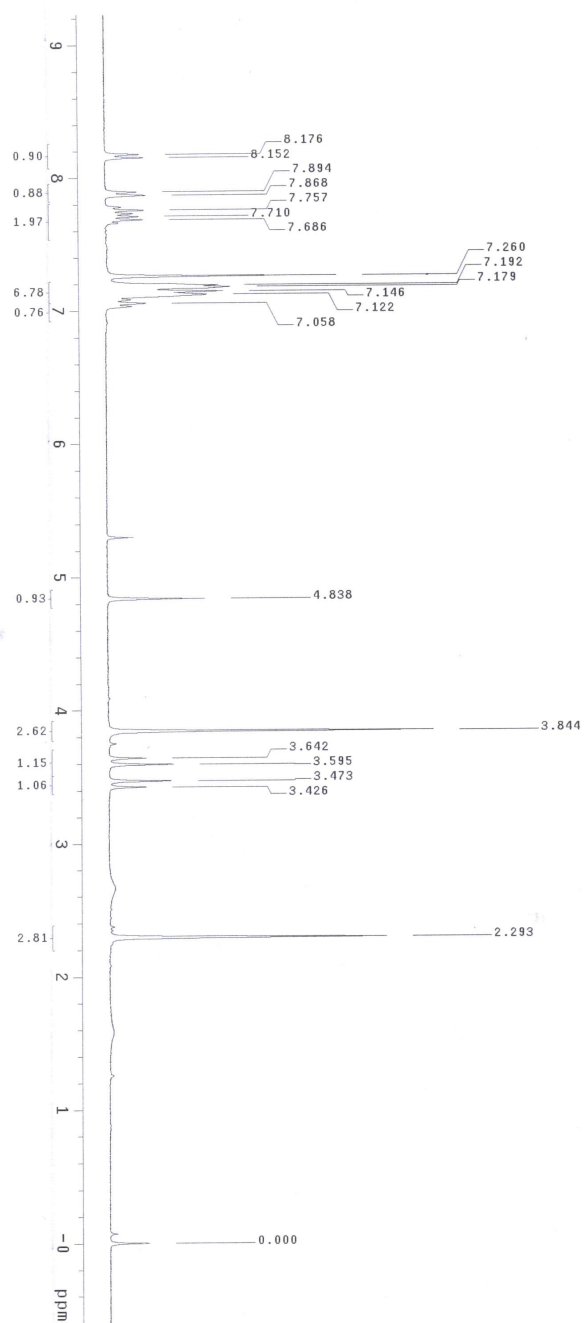




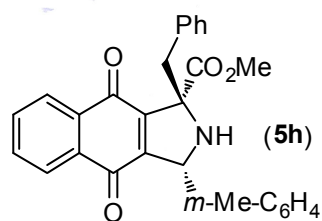
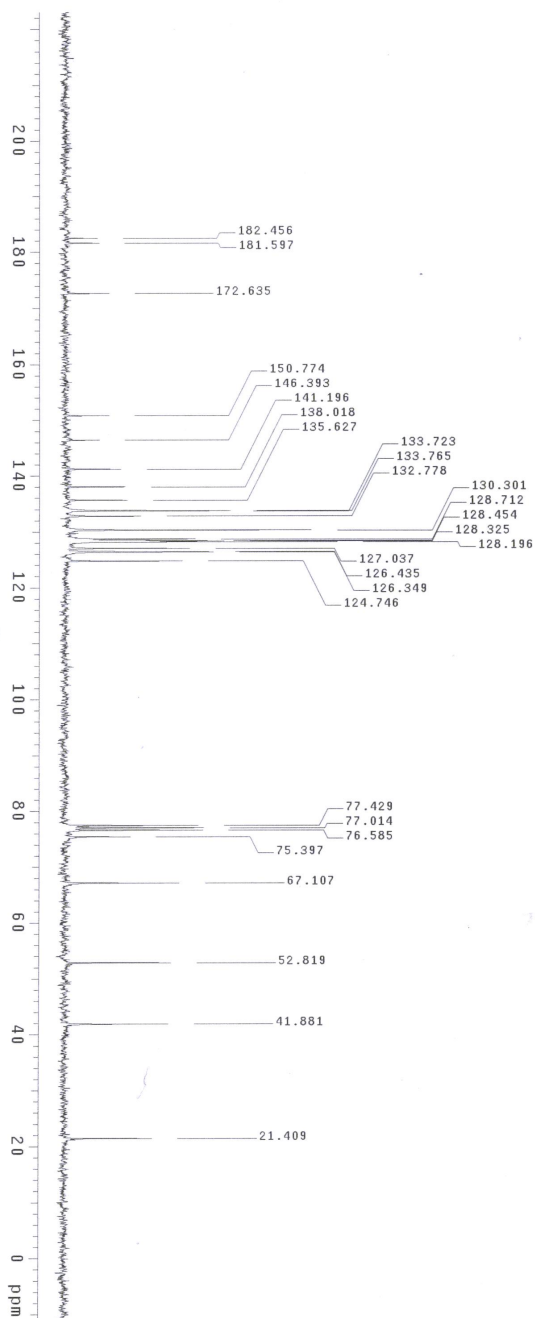
Solvent: CDCl₃
 Acquisition temperature: 300.2 K
 Mercury-300BB "mercury300"
 Relax. delay: 1.000 sec
 Pulse: 28.0 degrees
 Acq. time: 0.500 sec
 Nuclei: 13C
 152 repetitions
 OBSERVE: C13, 75.4554921 MHz
 DECOUPLE: H1, 300.0815382 MHz
 CONTINUOUS on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 4.0 Hz
 F1 size 225
 Total time 2 hr, 24 min, 23 sec

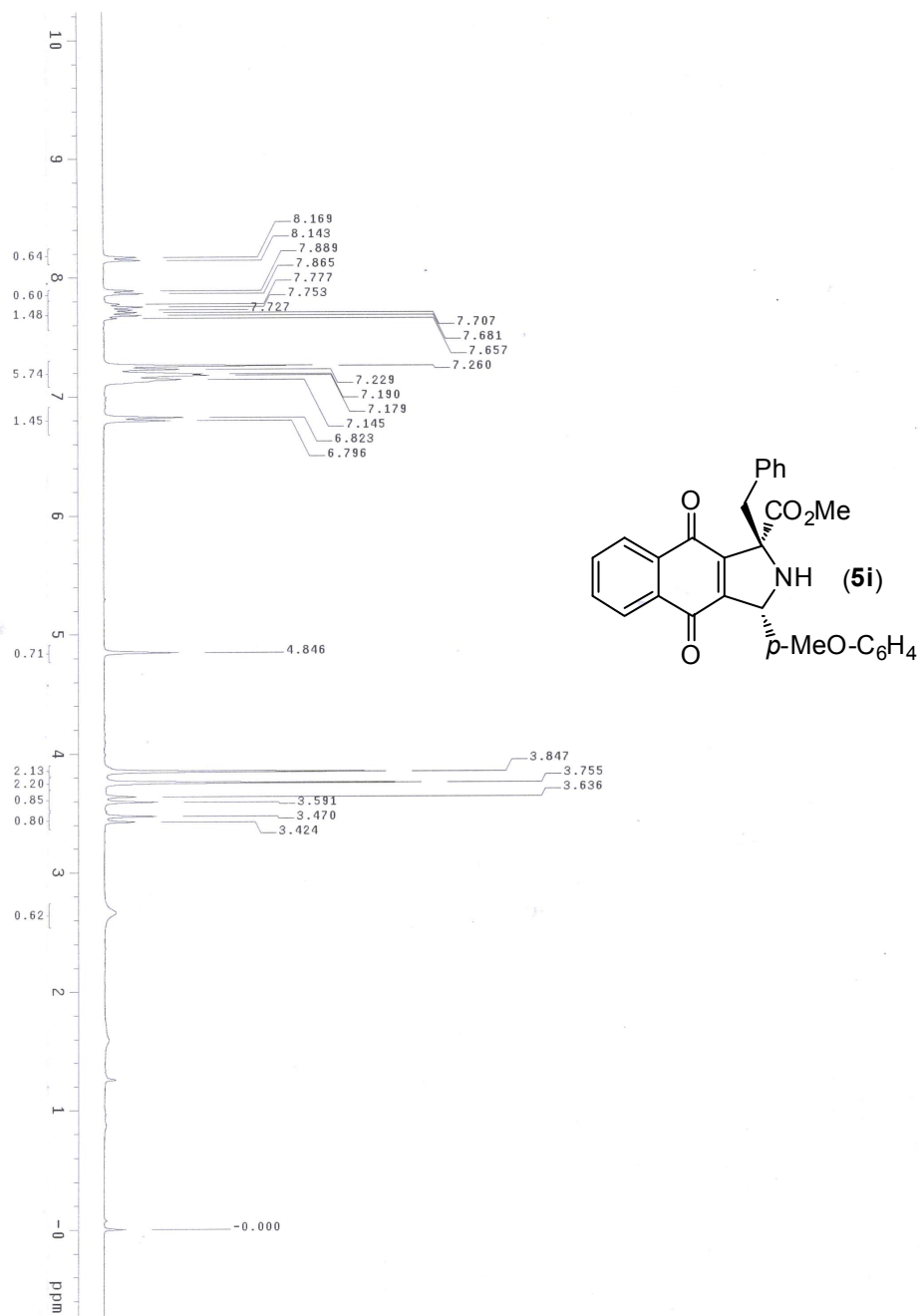


h21-4-78b-3

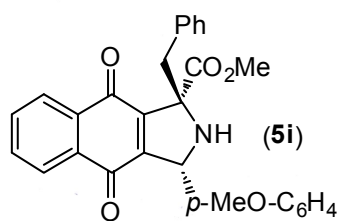
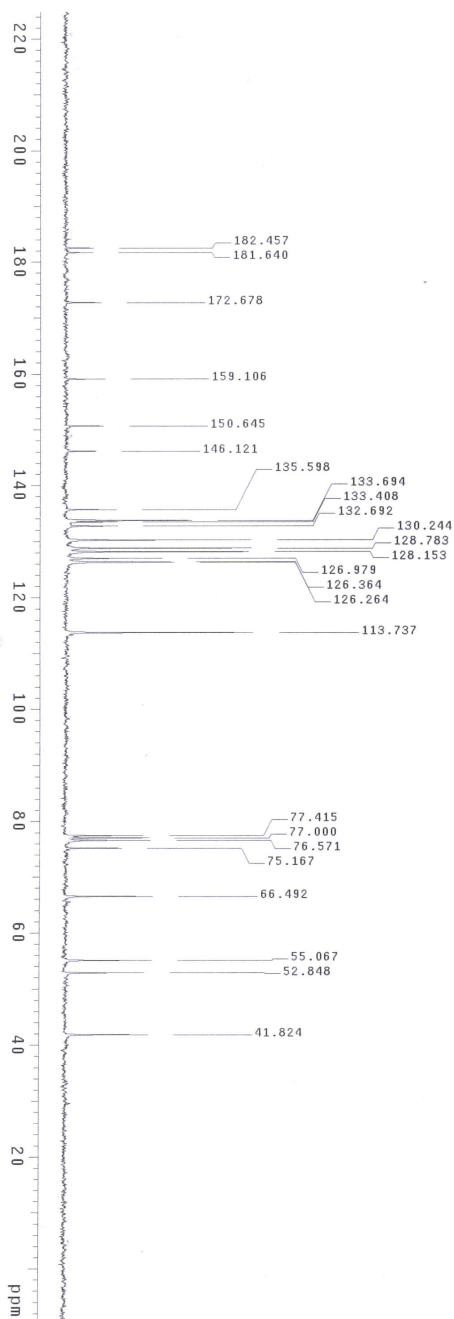


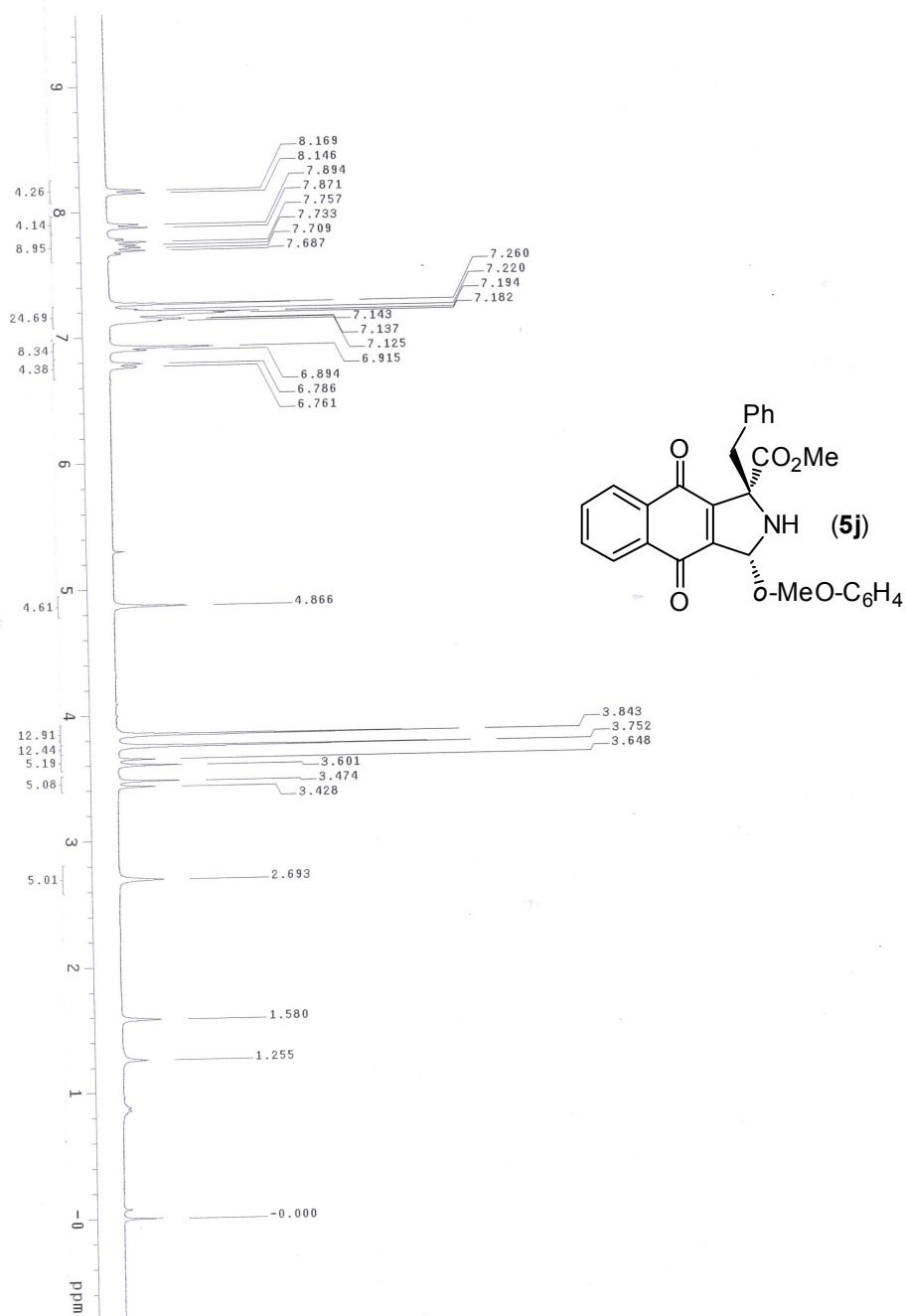
Solvent: CDCl₃
 Ambient temperature
 Mercury-300BB "mercury300"
 Relax. delay 1.000 sec
 Pulse 28.0 degrees
 Acq. time 0.50 sec
 Width 163.1 Hz
 Freq 125.13 MHz
 208 repetitions
 OBSERVE C13, 75.4554543 MHz
 DECOUPLE H1, 300.0815362 MHz
 Decoupling on
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 4.0 Hz
 F1 125.13 MHz
 F2 125.13 MHz
 Total time 2 hr, 24 min, 23 sec



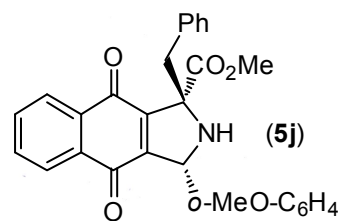
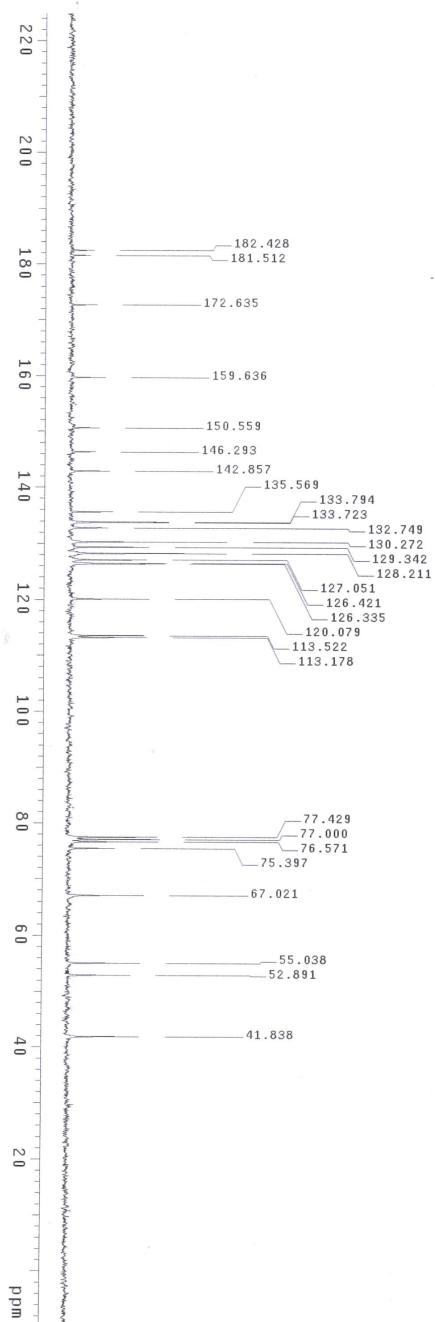


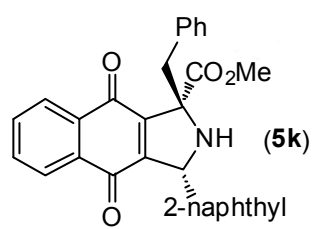
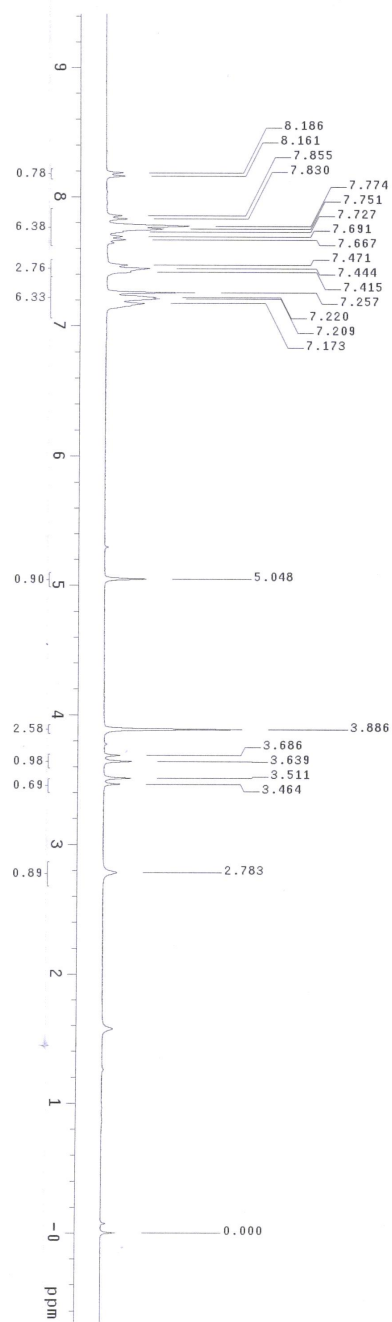
Solvent: CDCl₃
 Acquisition: 13C NMR
 Mercury-300MB "mercury300"
 Relax. delay 1.000 sec
 Pulse: 28.0 degrees
 Acq. time: 0.500 sec
 F1: 125.761 MHz
 192 Repetitions
 OBSERVE: C13, 75.4559172 MHz
 DECOUPLE: H1, 300.0815382 MHz
 Power: 40 dB on
 CONTINUOUS
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 4.0 Hz
 F1 size 32768
 Total time 2.00 hr, 24 min, 23 sec



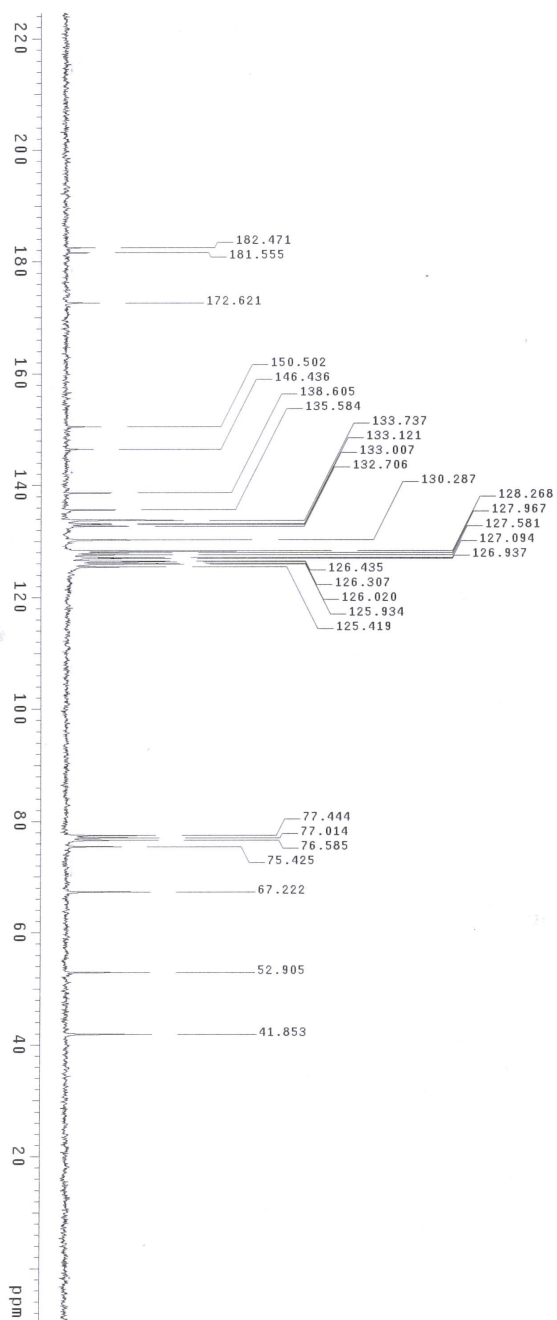
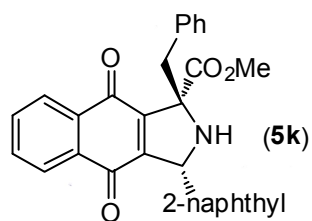


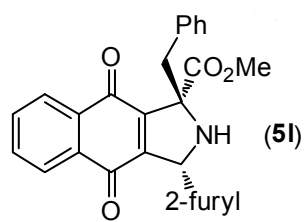
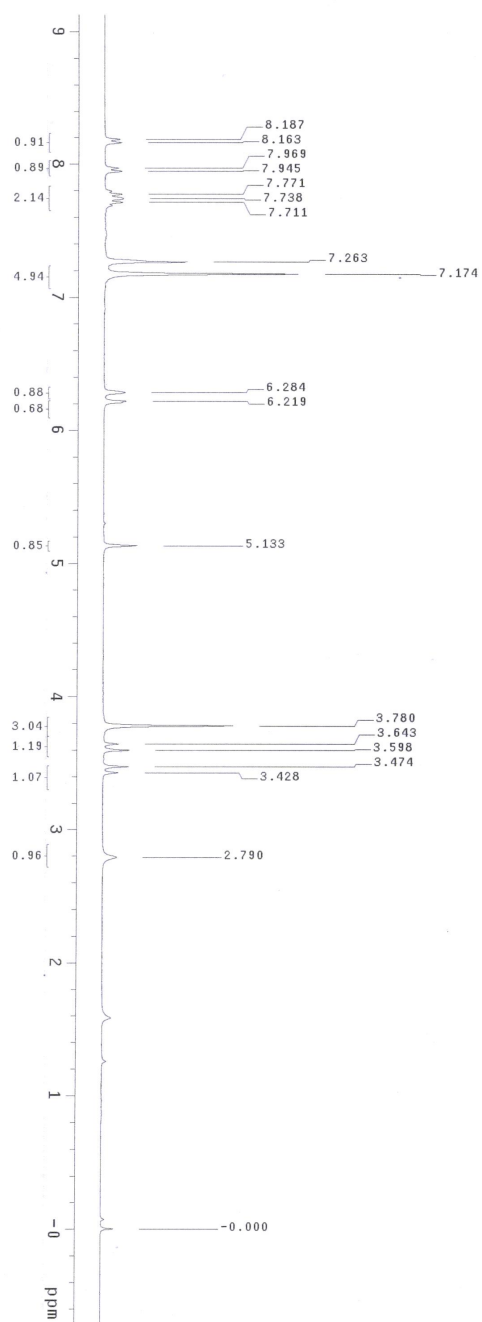
Solvent: CDCl₃
 Name: 5j
 Temperature: 300.2 K
 Mercury-300MHz
 Relax. delay: 1.000 sec
 Pulse: 28.0 degrees
 Acq. time: 0.500 sec
 F₂: 125.761 MHz
 F₁: 125.761 MHz
 OBSERVE C13, 75.4553161 MHz
 DECOUPLE H1, 300.0815382 MHz
 Power: 40.0 dB
 H1 channel: ON
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening: 4.0 Hz
 FT size: 32768
 Total time: 2 hr, 24 min, 23 sec



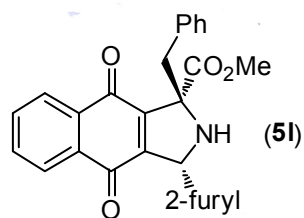
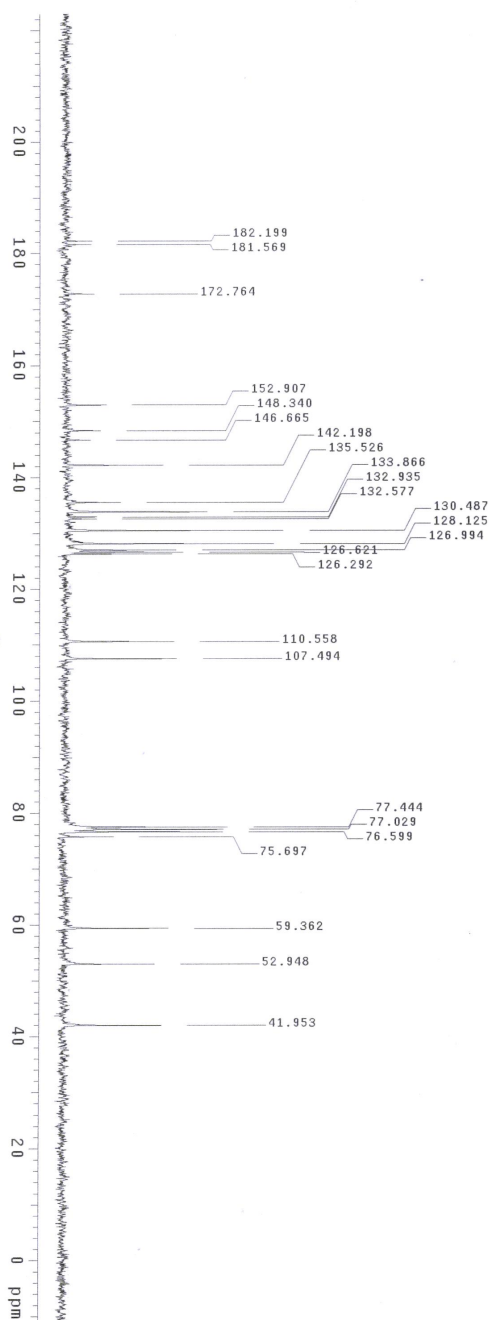


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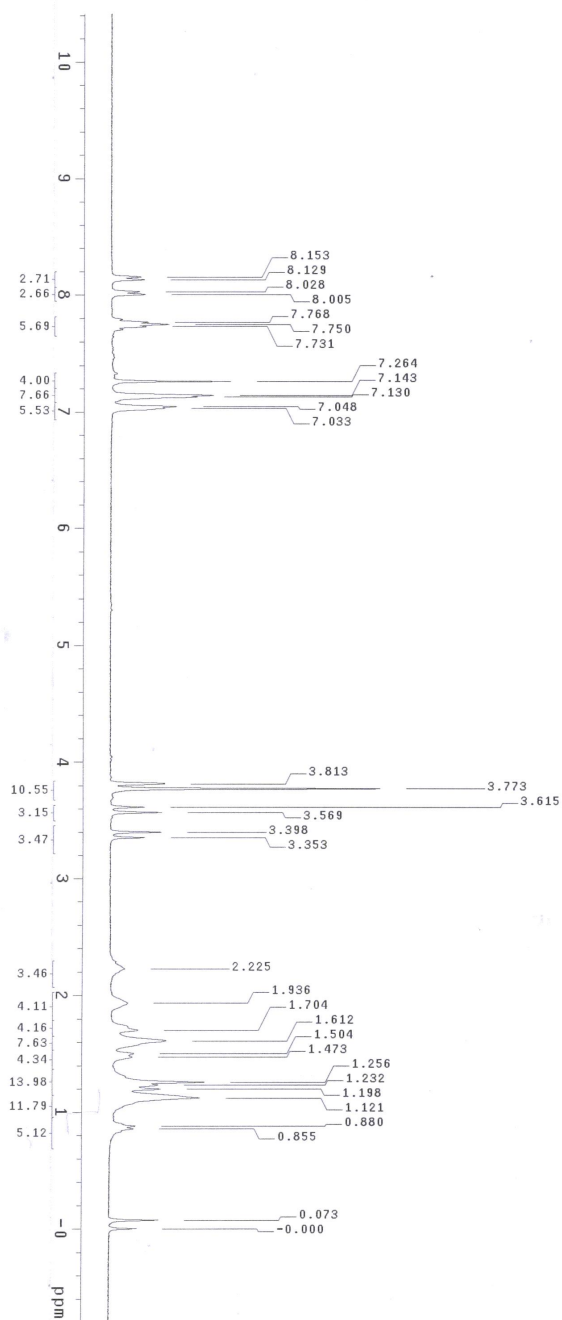
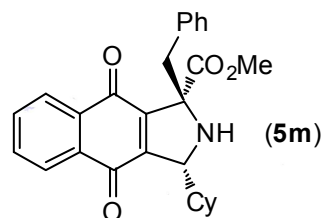




Solvent: CDCl₃
 Acquired temperature
 Mercury-300B "mercury300"
 Relax. delay 1.000 sec
 Pulse 28.0 degrees
 Acq. time 3.000 sec
 F2 101.625 MHz
 248 repetitions
 OBSERVE C13, 75.4554537 MHz
 DECOUPLE H1, 300.0815362 MHz
 CONTINUOUSLY on
 WALTZ-16 modulated
 DATA PROCESSING
 F1 line broadening 4.0 Hz
 F2 101.625 MHz
 Total time 2 hr, 24 min, 23 sec



h21-4-123
 Archive directory: /export/home/vu/vmr/sys/data
 Sample directory:
 File: PROTON
 Pulse Sequence: szpu1



THE ORDER

Andze sequence: szpu

Ambient temperature

.....

Relax. delay 1.000 sec

Acq. time 0.499 sec

600 repetitions

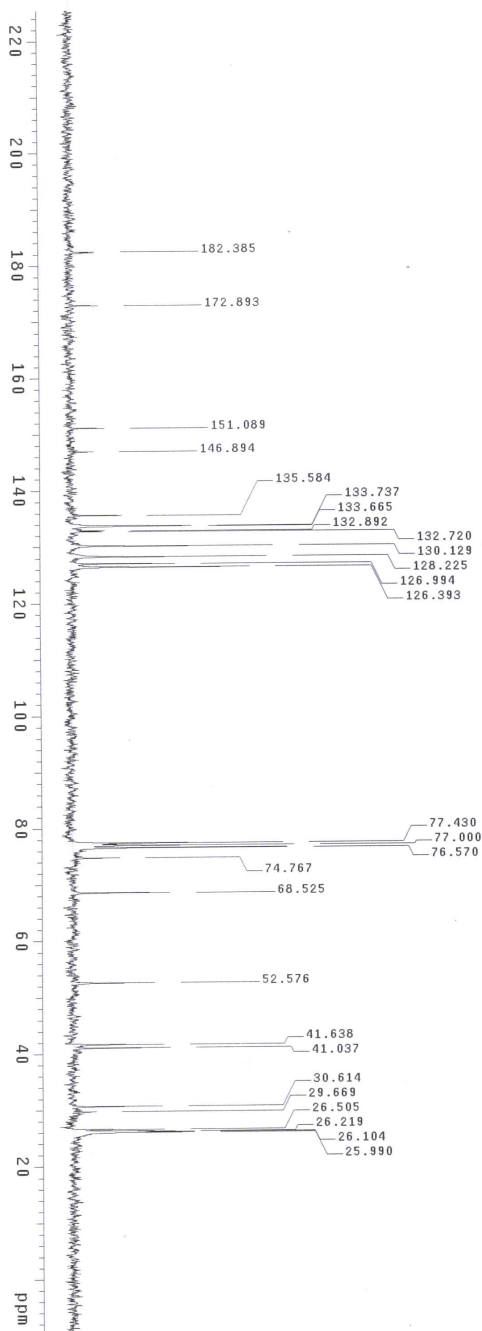
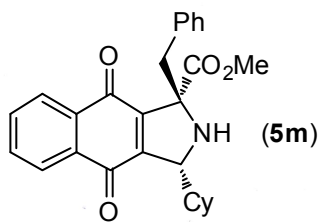
DECOUPLE H1, 300.0820522 MHz

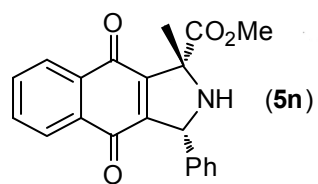
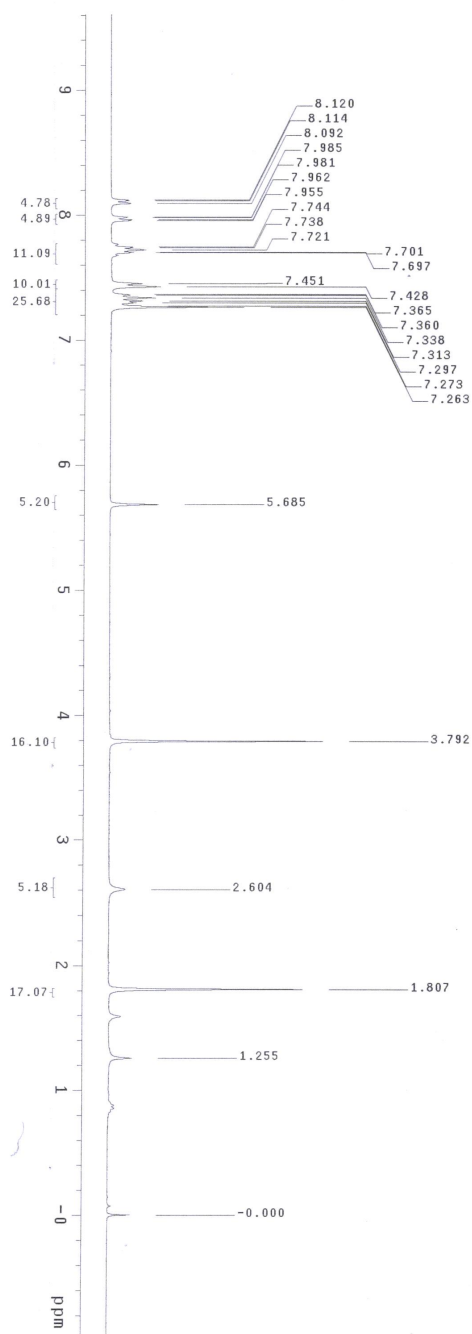
continuously on

DATA PROCESSING

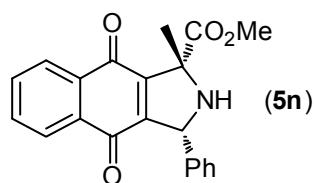
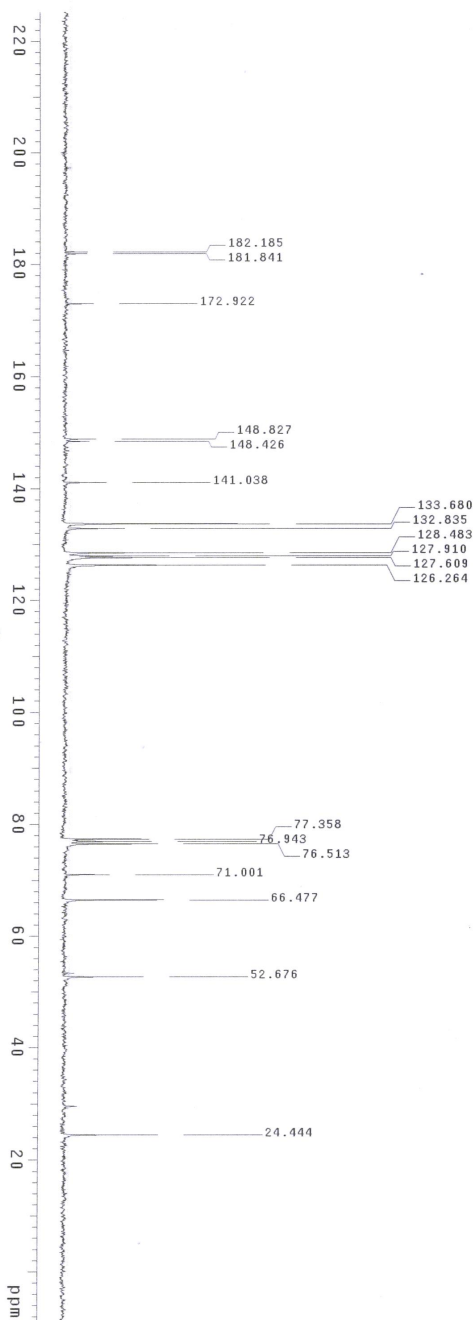
Line numbering
FT size 32768

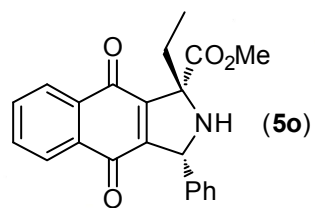
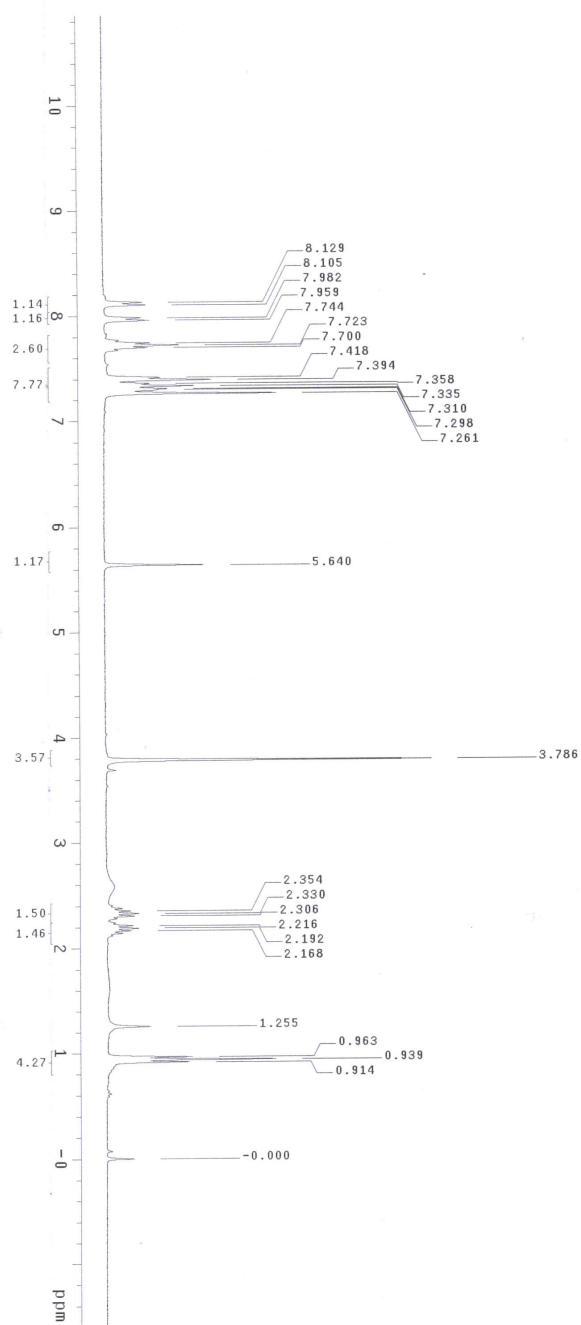
LOCAL CLIMATE, 2000-2009



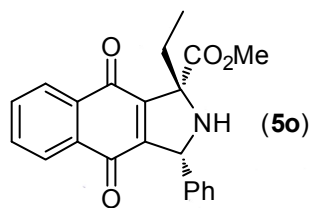
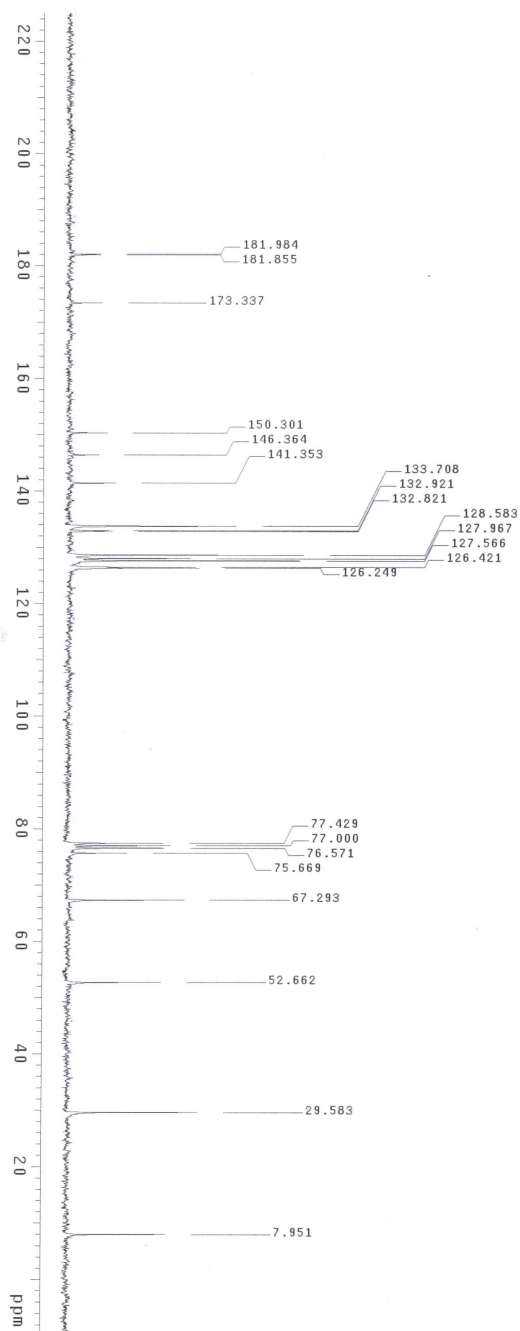


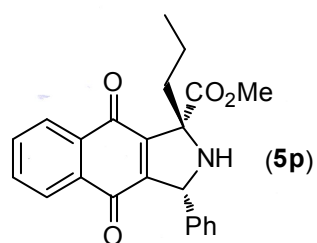
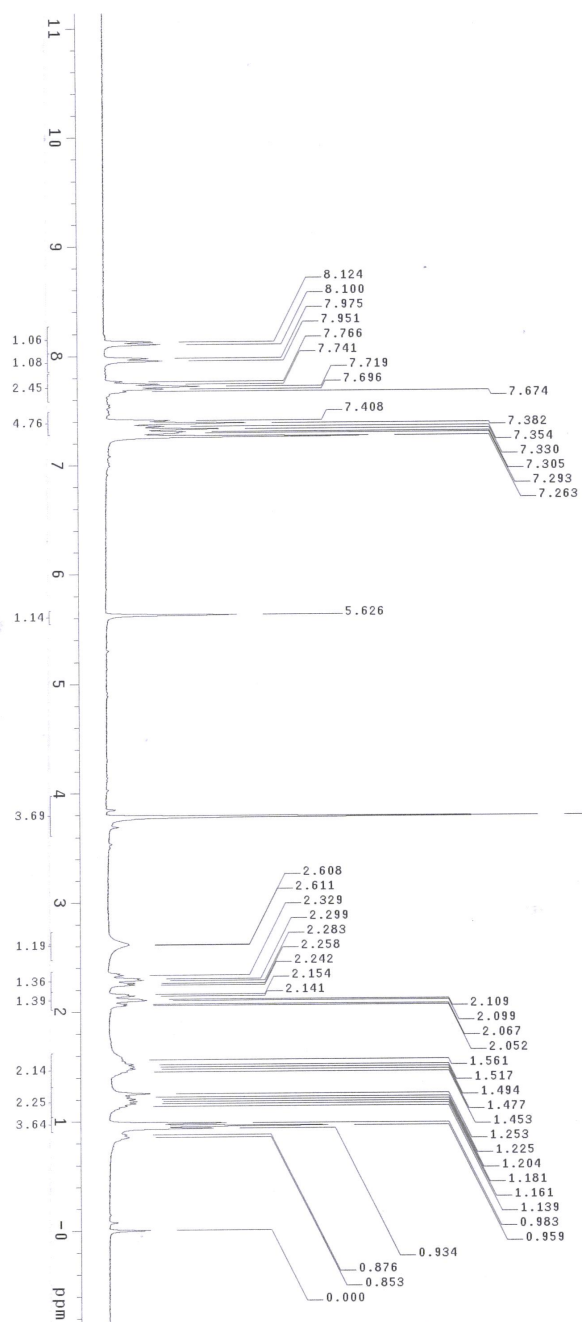
Solvent: CDCl₃
 Acquisition temperature
 Mercury-300BB "mercury300"
 Relax delay 1.000 sec
 Pulse 28.0 degrees
 Acq. time 0.50 sec
 Width 163.1 Hz
 Freq 125.76 MHz
 368 repetitions
 OBSERVE C13, 75.4552891 MHz
 DECOUPLE H1, 300.0815382 MHz
 100.0000000 Hz
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 4.0 Hz
 F1 25.0000000 Hz
 Total time 2 hr, 24 min, 23 sec

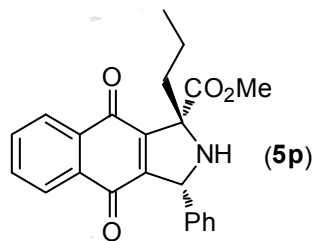
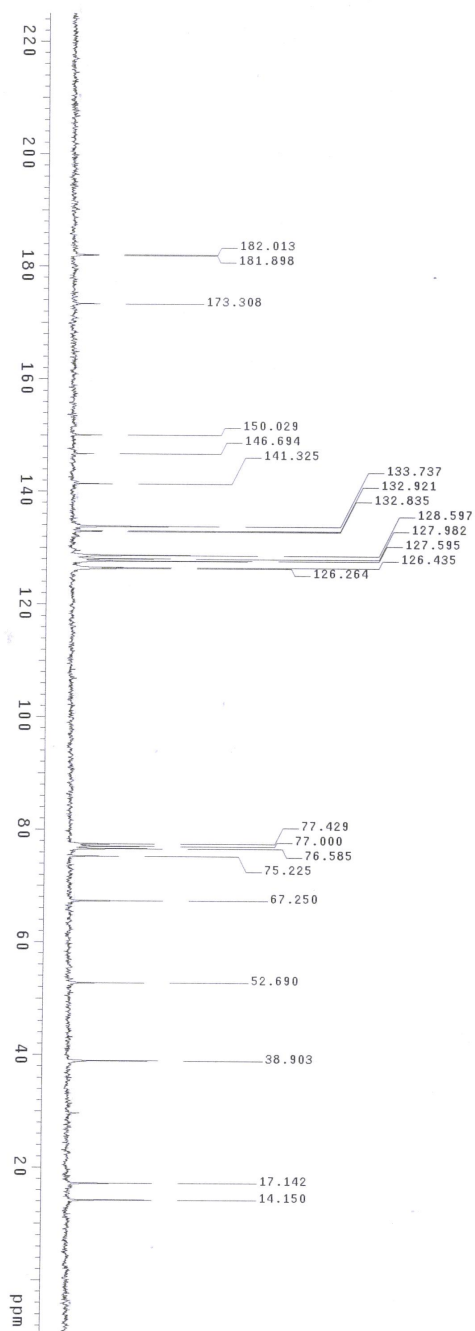




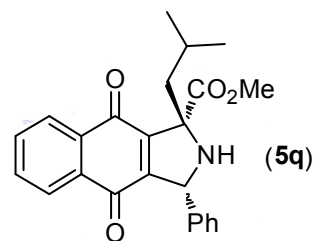
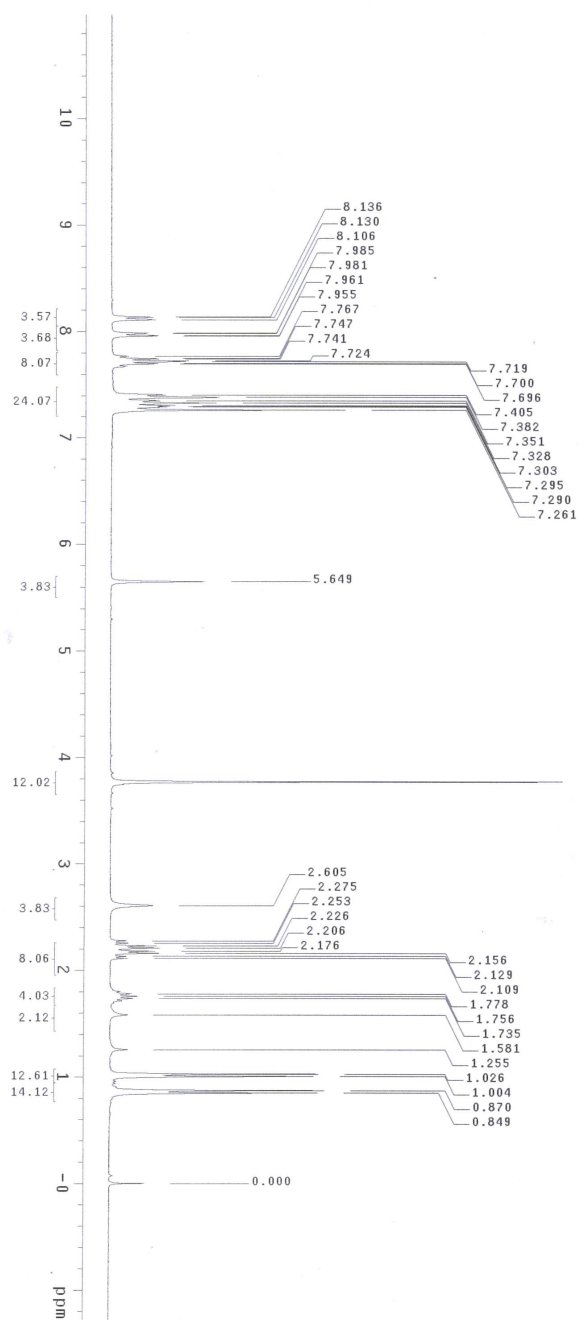
SOLVENT: CDCl₃
 Acquisition temperature
 Mercury-300BB "mercury300"
 Relax. delay 1.000 sec
 Pulse 29.0 degrees
 Acq. time 0.500 sec
 Nucleus 13C
 Observed 128.583 MHz
 184 repetitions
 OBSERVE C13, 75.4553064 MHz
 DECOUPLE H1, 300.0815382 MHz
 POWER 40.0 dB
 SFO 125.761 MHz
 CONTINUOUSLY on
 DATA PROCESSING
 Line broadening 4.0 Hz
 F1 size 32768
 Total time 2 hr, 24 min, 23 sec





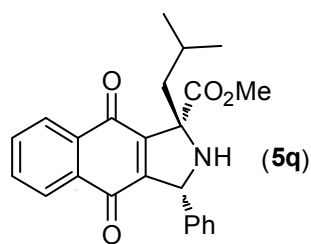
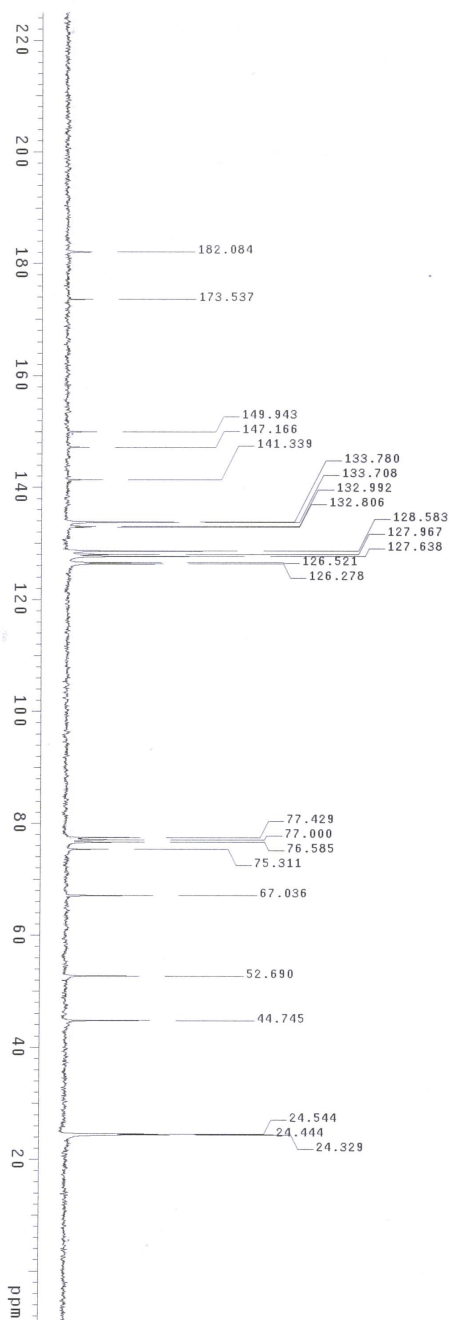


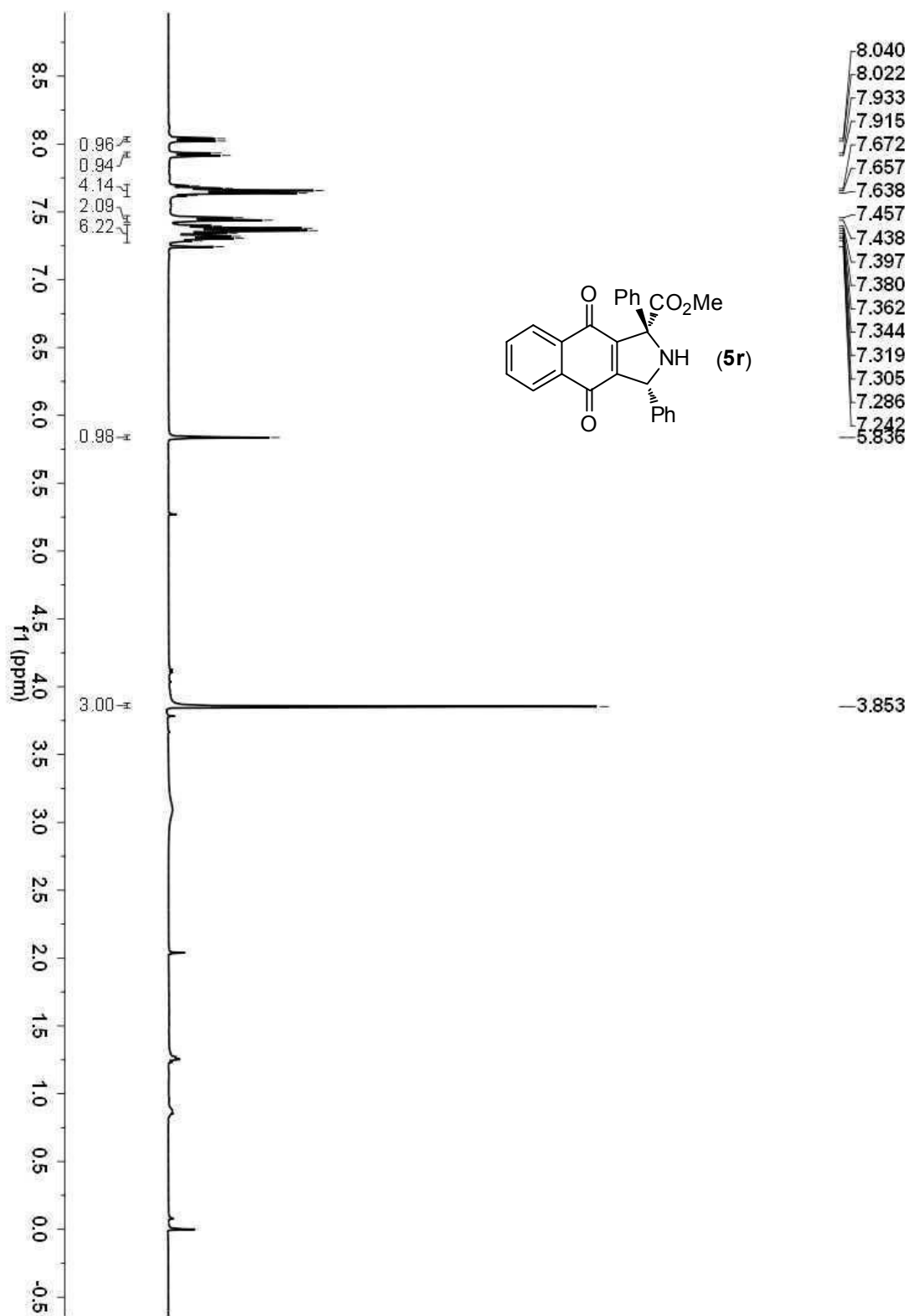
Solvent: CDCl₃
 Relax delay: 1.000 sec
 Pulse: 28.0 degrees
 Acq. time: 0.500 sec
 V. In: 1013.1 Hz
 V. Out: 1013.1 Hz
 OBSERVE: C13, 75.453053 MHz
 DECOUPLE: H1, 300.10815382 MHz
 Power: 40 dB
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening: 4.0 Hz
 FT size: 32768
 Total time: 2 hr, 24 min, 23 sec

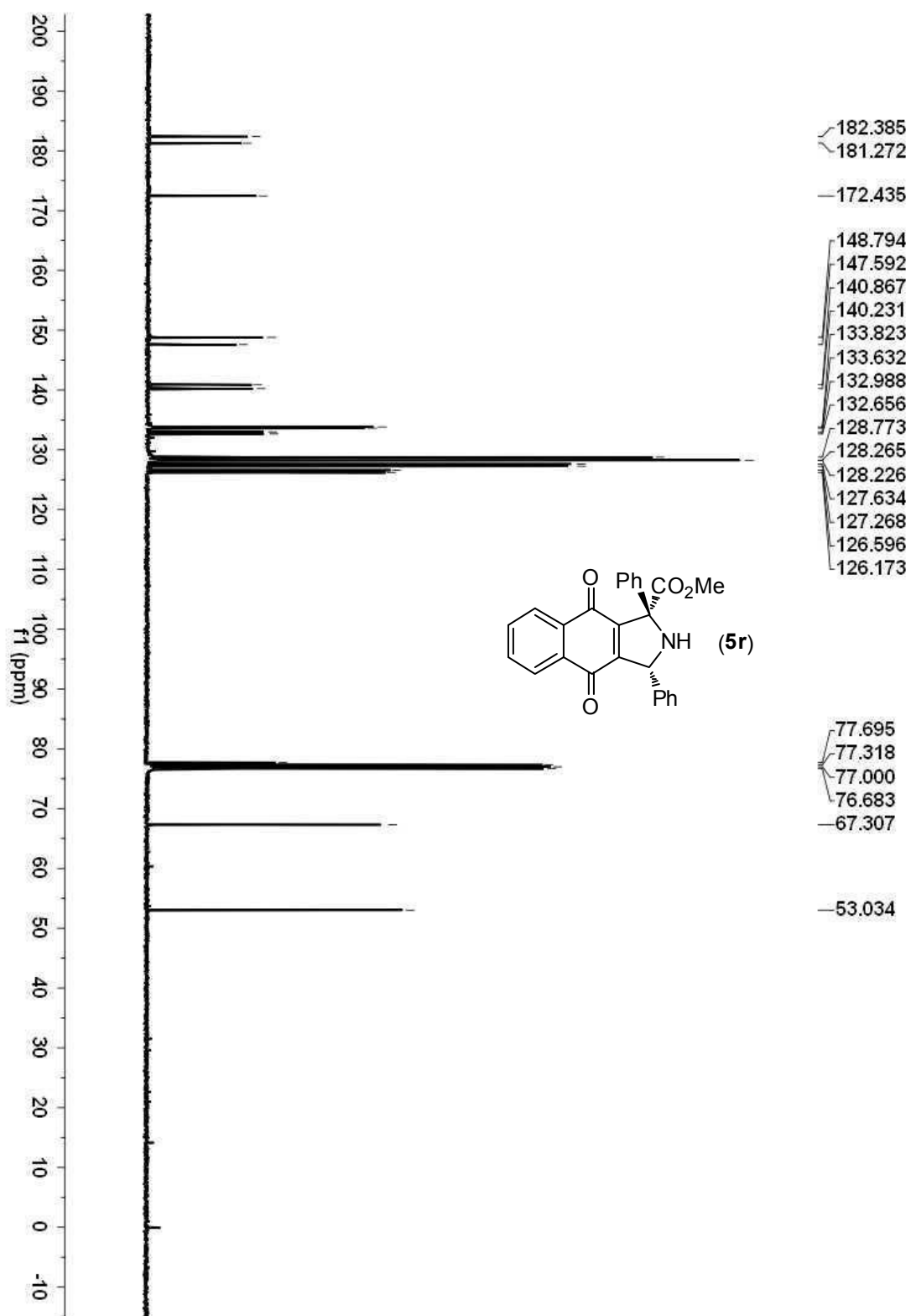


CSMD1-14-1140

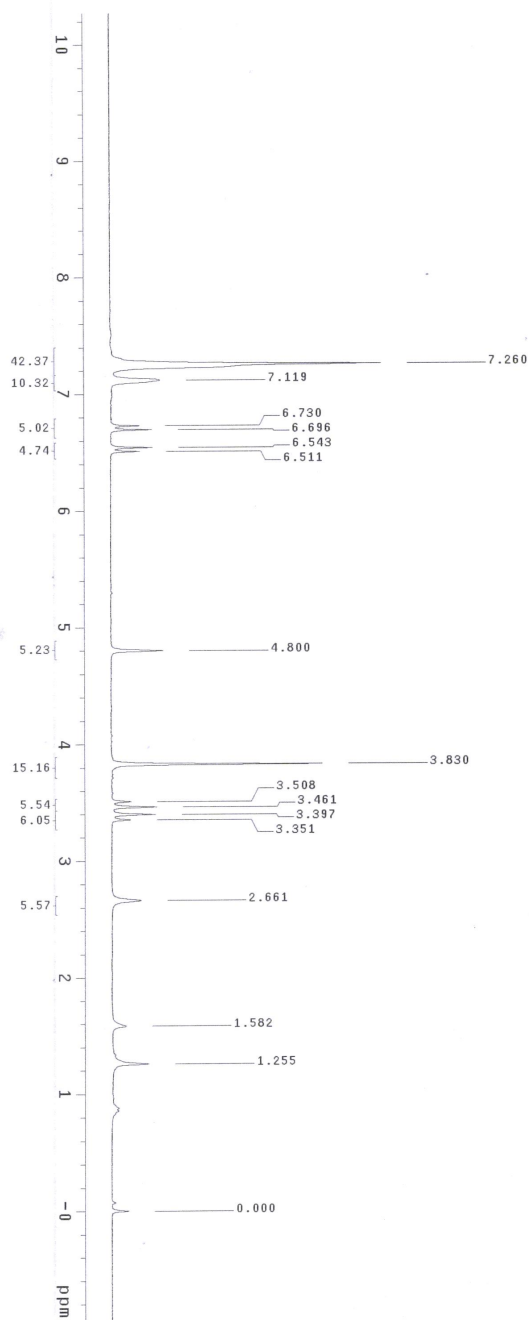
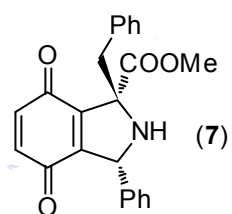
Solvent: CDCl₃
 Ambient temperature
 Mercury-500B8 Mercury3000
 Relax delay 1.000 sec
 Pulse 28.0 degrees
 Acq. time 0.500 sec
 Width 17699.1 Hz
 240.000 MHz
 OBSERVE C13 101.75, 4553085 MHz
 DECOUPLE H1, 300.0815382 MHz
 Power 40 dB
 continuously on
 MATHS integrated
 DATA PROCESSING
 Line broadening 4.0 Hz
 FT size 32768
 Total time 2 hr, 24 min, 23 sec



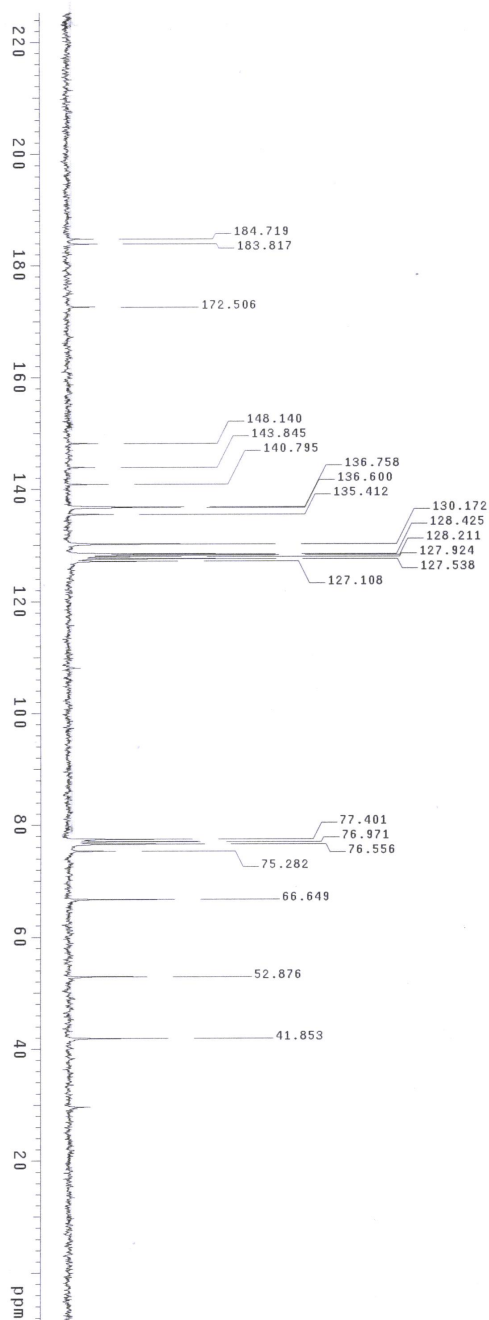
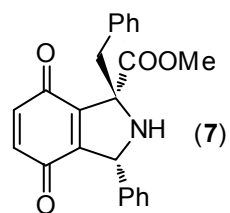


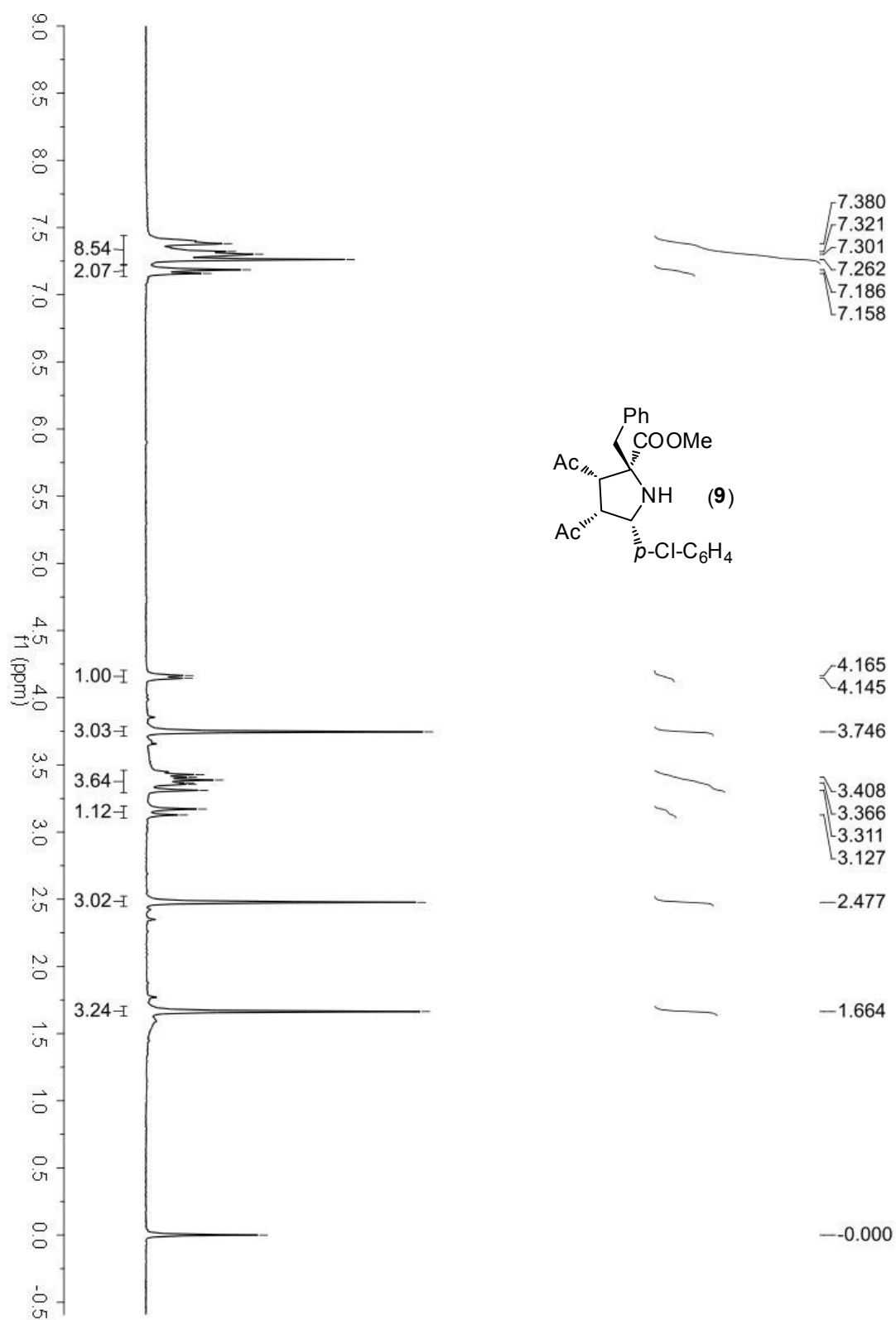


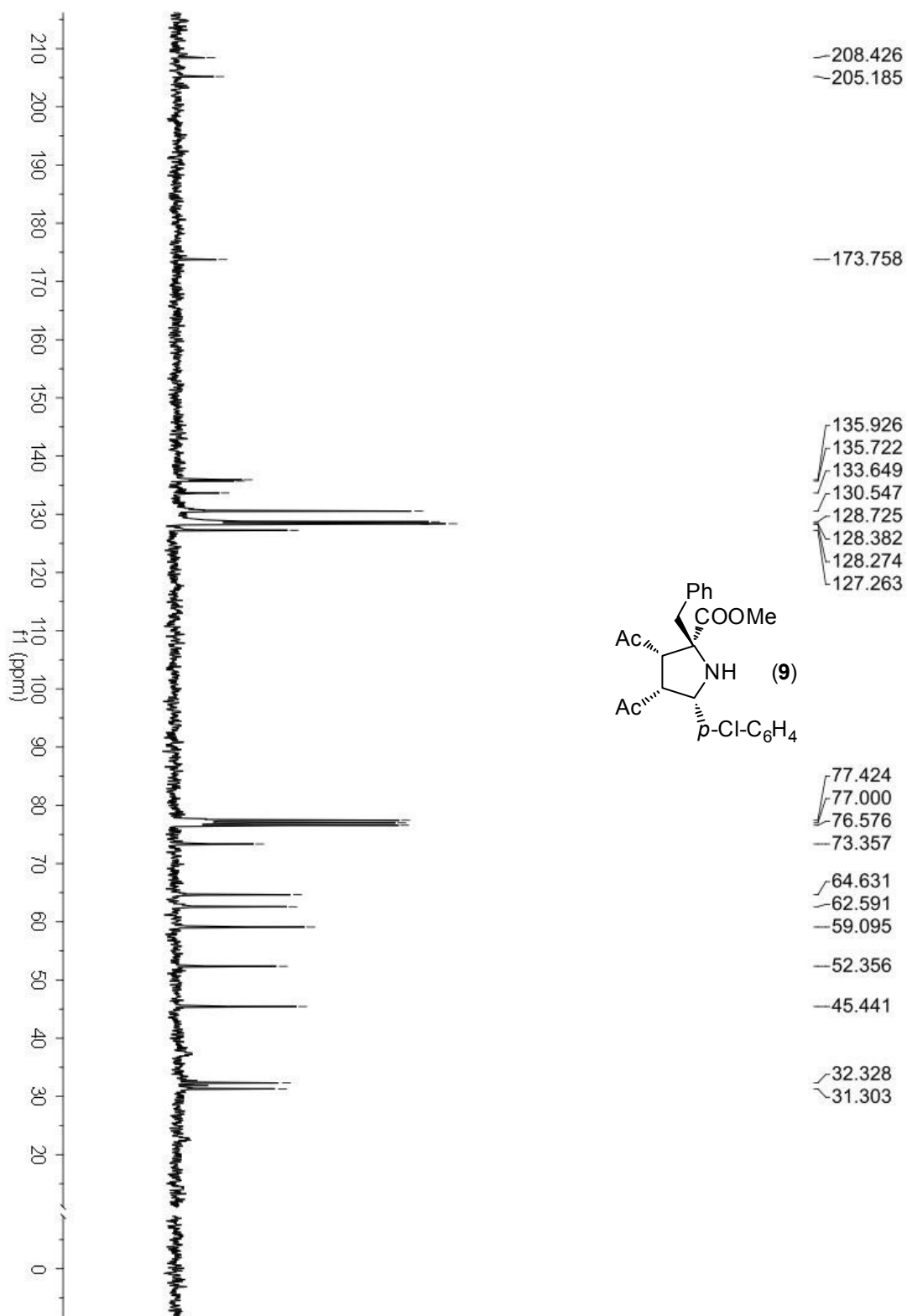
h21-4-120
 Archive directory: /export/home/vu/vnmr/sys/data
 Sample directory:
 File: PROTON
 Pulse Sequence: szpu1



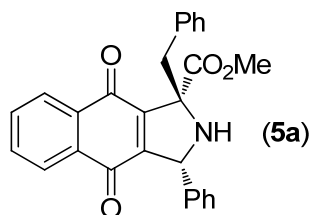
Archive directory: /export/home/jw/vnmr/sys/data
 Sample directory:
 File: CARBON
 Pulse Sequence: s2pul1
 Solvent: CDCl3
 Ambient temperature
 Mercury-300BB "mercury300"
 Relax: delay 1.000 sec
 Pulse 28.0 degrees
 Width 1769.4 Hz
 Width 1769.4 Hz
 296 repetitions
 OBSERVE C13, 75.4552828 MHz
 DECOUPLE H1, 300.0620522 MHz
 296 repetitions
 continuously on
 WALTZ-16 modulated
 DATA PROCESSING
 Line broadening 4.0 Hz
 F1 152.2446
 F2 152.2446
 Total time 2 hr, 24 min, 19 sec





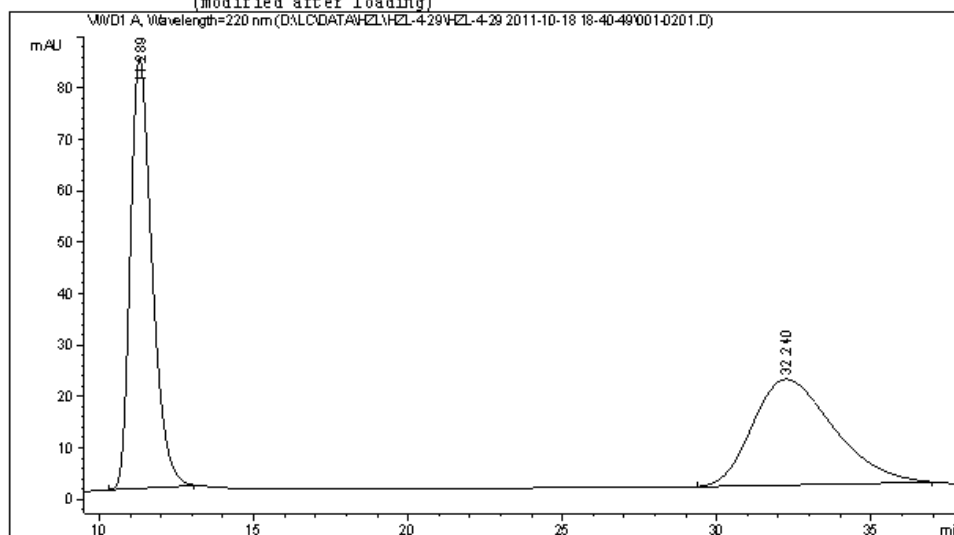


IX. HPLC Chromatograms



Data File D:\LC\DATA\H2L\H2L-4-29\H2L-4-29 2011-10-18 18-40-49\001-0201.D
 Sample Name: H2L-4-29

```
=====
Acq. Operator   : H2L                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : Vial 1
Injection Date  : 10/18/2011 6:52:46 PM     Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\H2L\Data\H2L-4-29\H2L-4-29 2011-10-18 18-40-49\ASH-20-80-10ML-220NM.
M
Last changed    : 8/29/2011 3:56:33 PM by H2L
Analysis Method : D:\LC\DATA\H2L\H2L-4-29\H2L-4-29 2011-10-18 18-40-49\001-0201.D\DA.M (ASH-
20-80-10ML-220NM.M)
Last changed     : 9/24/2012 9:57:12 AM by FX
                  (modified after loading)
=====
```



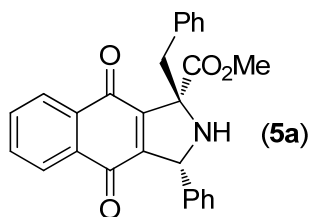
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VMD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.289	BB	0.7515	4096.40576	83.69995	52.1176
2	32.240	BB	2.1436	3763.51929	20.59521	47.8824

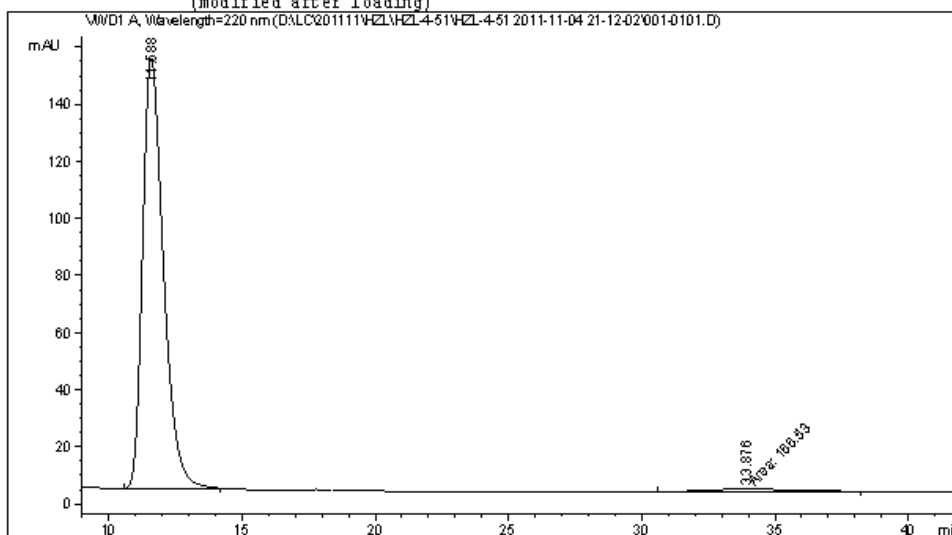
Totals : 7859.92505 104.29516



Data File D:\LC\201111\H2L\H2L-4-51\H2L-4-51 2011-11-04 21-12-02\001-0101.D
Sample Name: H2L-4-51B

```
=====
Acq. Operator   : H2L                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 1
Injection Date  : 11/4/2011 9:13:17 PM      Inj       :    1
                                           Inj Volume: 5 µl

Acq. Method     : D:\LC\201111\H2L\H2L-4-51\H2L-4-51 2011-11-04 21-12-02\ASH-20-80-10ML-
                  220NM-45MIN.M
Last changed    : 8/29/2011 3:55:38 PM by H2L
Analysis Method : D:\LC\201111\H2L\H2L-4-51\H2L-4-51 2011-11-04 21-12-02\001-0101.D\DA.M (
                  ASH-20-80-10ML-220NM-45MIN.M)
Last changed    : 12/12/2011 4:39:01 PM by yl
                  (modified after loading)
=====
```



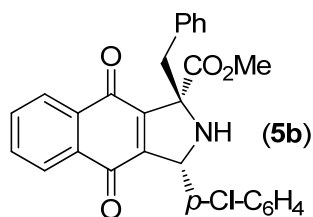
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VMD1 A, Wavelength=220 nm

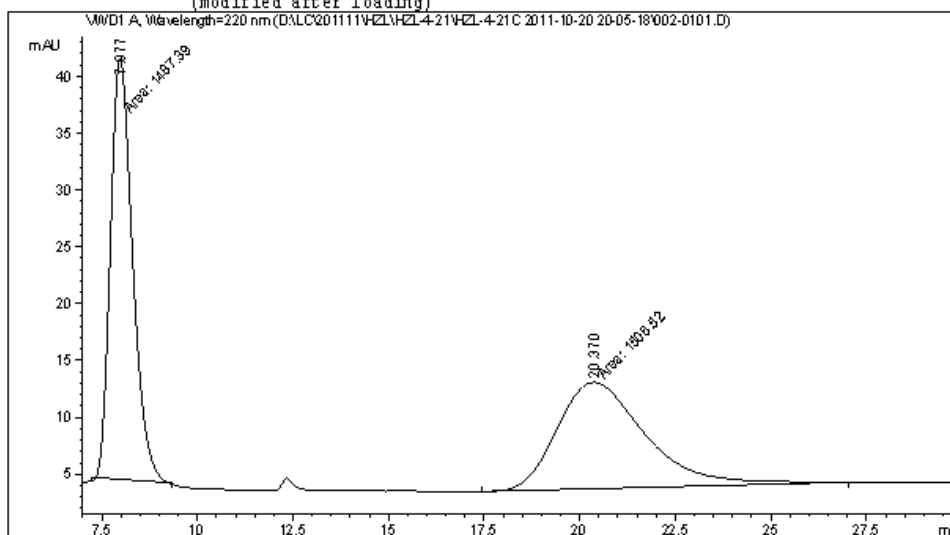
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.588	BB	0.8335	8191.35303	150.73788	98.0075
2	33.876	MM	3.3027	166.53038	8.40367e-1	1.9925

Totals : 8357.88341 151.57825



Data File D:\LC\201111\HZZ\HZZ-4-21\HZZ-4-21C 2011-10-20 20-05-18\002-0101.D
Sample Name: HZZ-4-21C

```
=====
Acq. Operator   : HZZ                               Seq. Line :    1
Acq. Instrument : Instrument 1                       Location  : Vial 2
Injection Date  : 10/20/2011 8:07:18 PM              Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method     : D:\LC\201111\HZZ\HZZ-4-21\HZZ-4-21C 2011-10-20 20-05-18\ASH-40-60-10ML-
                  220NM.M
Last changed    : 10/20/2011 8:03:32 PM by HZZ
Analysis Method : D:\LC\201111\HZZ\HZZ-4-21\HZZ-4-21C 2011-10-20 20-05-18\002-0101.D\DA.M (
                  ASH-40-60-10ML-220NM.M)
Last changed    : 10/29/2011 3:19:44 PM by HZZ
                  (modified after loading)
=====
```



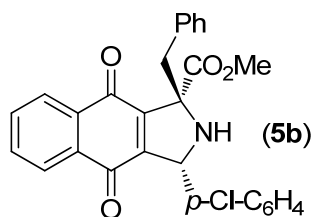
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.977	MM	0.6694	1487.38757	37.03318	49.6805
2	20.370	MM	2.6827	1506.52039	9.35950	50.3195

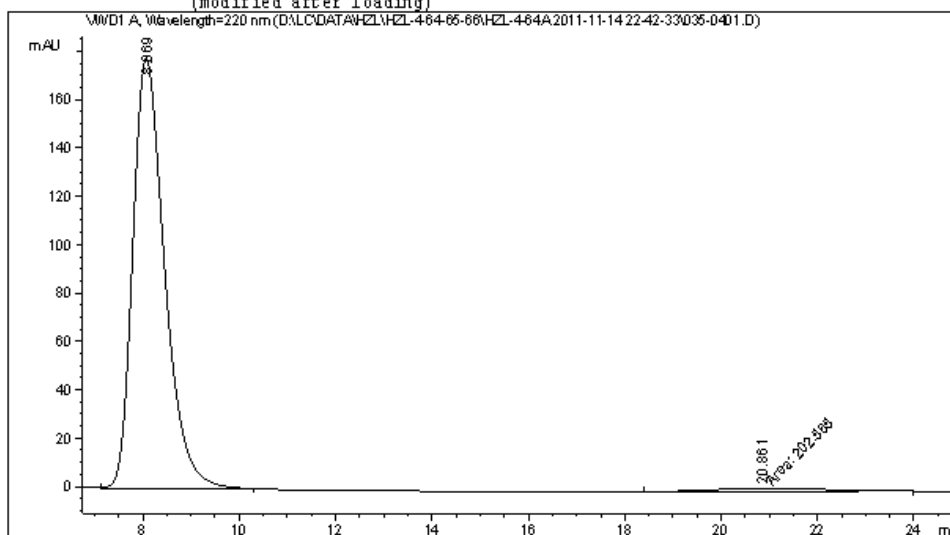
Totals : 2993.90796 46.39268



Data File D:\LC\DATA\H2L\H2L-4-64-65-66\H2L-4-64A 2011-11-14 22-42-33\035-0401.D
Sample Name: H2L-4-66B

```
=====
Acq. Operator   : hzl                      Seq. Line :    4
Acq. Instrument : Instrument 1              Location  : Vial 35
Injection Date  : 11/14/2011 11:42:09 PM    Inj       :    1
                                           Inj Volume: 5 µl

Acq. Method     : D:\LC\201111\H2L\H2L-4-64-65-66\H2L-4-64A 2011-11-14 22-42-33\ASH-40-60-
                  10ML-220NM-30MIN.M
Last changed    : 10/31/2011 7:25:18 PM by HZL
Analysis Method : D:\LC\DATA\H2L\H2L-4-64-65-66\H2L-4-64A 2011-11-14 22-42-33\035-0401.D\DA.
                  M (ASH-40-60-10ML-220NM-30MIN.M)
Last changed    : 9/24/2012 10:07:24 AM by FX
                  (modified after loading)
=====
```



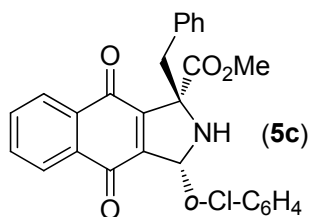
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=220 nm

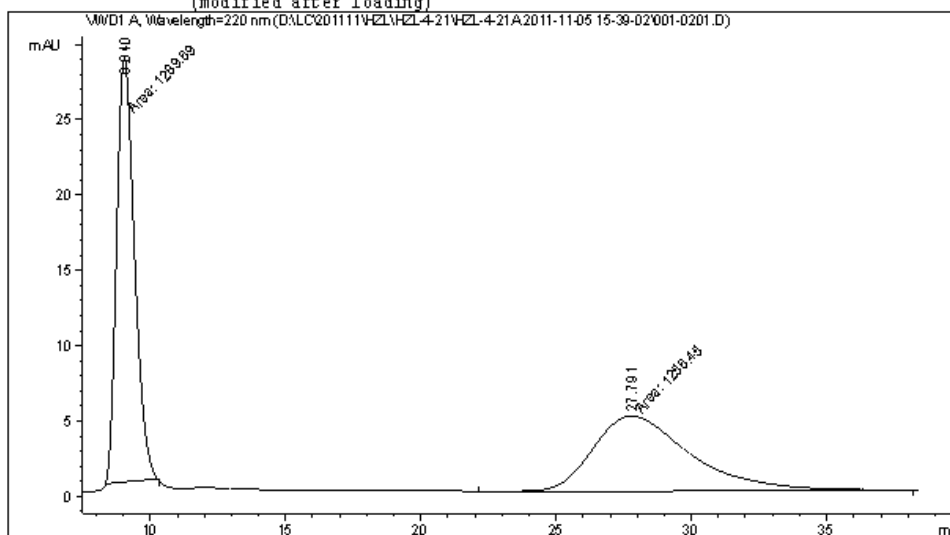
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.069	BB	0.7045	8175.20117	178.06204	97.5821
2	20.861	MM	2.6940	202.56479	1.25320	2.4179

Totals : 8377.76596 179.31524



Data File D:\LC\201111\H2L\H2L-4-21\H2L-4-21A 2011-11-05 15-39-02\001-0201.D
Sample Name: H2L-4-21A

```
=====
Acq. Operator   : HZL                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : Vial 1
Injection Date  : 11/5/2011 3:51:26 PM      Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\201111\H2L\H2L-4-21\H2L-4-21A 2011-11-05 15-39-02\ASH-30-70-10ML-
220NM.M
Last changed    : 10/20/2011 6:42:48 PM by HZL
Analysis Method : D:\LC\201111\H2L\H2L-4-21\H2L-4-21A 2011-11-05 15-39-02\001-0201.D\DA.M (
ASH-30-70-10ML-220NM.M)
Last changed    : 11/5/2011 4:34:18 PM by HZL
(modified after loading)
=====
```



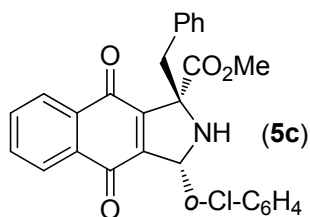
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=220 nm

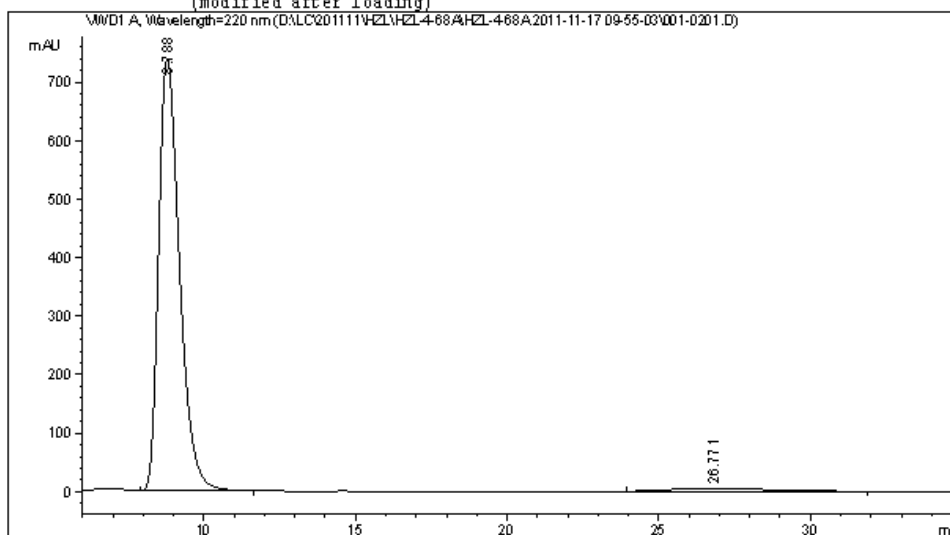
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.040	MM	0.7646	1289.69067	28.11200	50.6529
2	27.791	MM	4.1969	1256.44556	4.98963	49.3471

Totals : 2546.13623 33.10163



Data File D:\LC\201111\H2L\H2L-4-68A\H2L-4-68A 2011-11-17 09-55-03\001-0201.D
Sample Name: H2L-4-68A

```
=====
Acq. Operator   : hzl                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : Vial 1
Injection Date  : 11/17/2011 10:07:30 AM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\201111\H2L\H2L-4-68A\H2L-4-68A 2011-11-17 09-55-03\ASH-30-70-10ML-
220NM-40MIN.M
Last changed    : 10/29/2011 2:58:53 PM by HZL
Analysis Method : D:\LC\201111\H2L\H2L-4-68A\H2L-4-68A 2011-11-17 09-55-03\001-0201.D\DA.M (
ASH-30-70-10ML-220NM-40MIN.M)
Last changed    : 11/17/2011 11:07:45 AM by hzl
(modified after loading)
=====
```



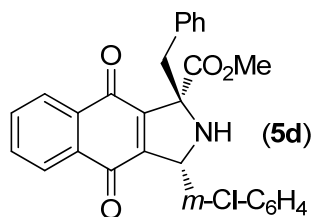
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=220 nm

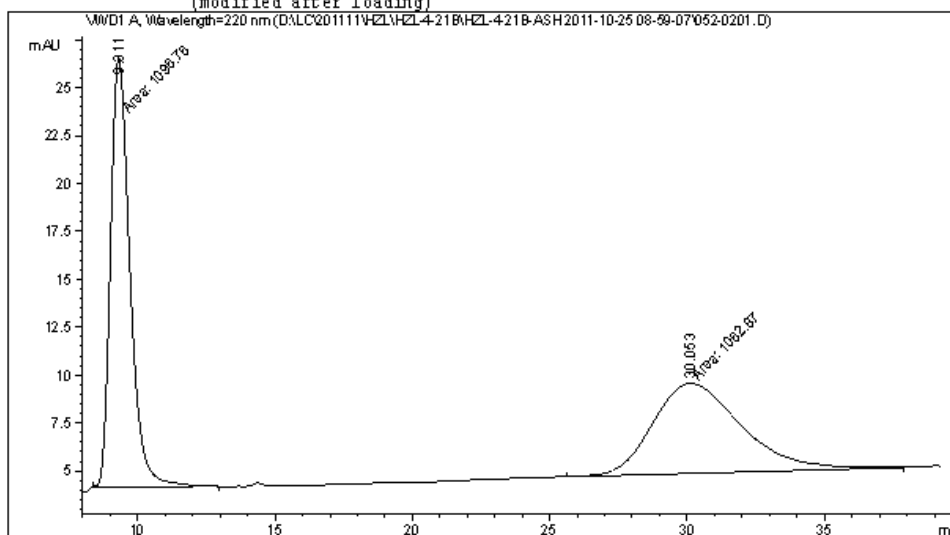
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.788	VB	0.7369	3.53488e4	739.33594	96.8285
2	26.771	BB	2.5743	1157.79492	5.28305	3.1715

Totals : 3.65066e4 744.61899



Data File D:\LC\201111\H2L\H2L-4-21B\H2L-4-21B-ASH 2011-10-25 08-59-07\052-0201.D
Sample Name: H2L-4-21B

```
=====
Acq. Operator   : hzl                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : Vial 52
Injection Date  : 10/25/2011 9:11:52 AM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\201111\H2L\H2L-4-21B\H2L-4-21B-ASH 2011-10-25 08-59-07\ASH-30-70-
                  10ML-220NM.M
Last changed    : 10/20/2011 6:42:48 PM by HZL
Analysis Method : D:\LC\201111\H2L\H2L-4-21B\H2L-4-21B-ASH 2011-10-25 08-59-07\052-0201.D\
                  DA.M (ASH-30-70-10ML-220NM.M)
Last changed    : 10/29/2011 2:51:53 PM by HZL
                  (modified after loading)
=====
```



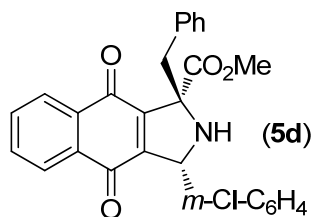
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=220 nm

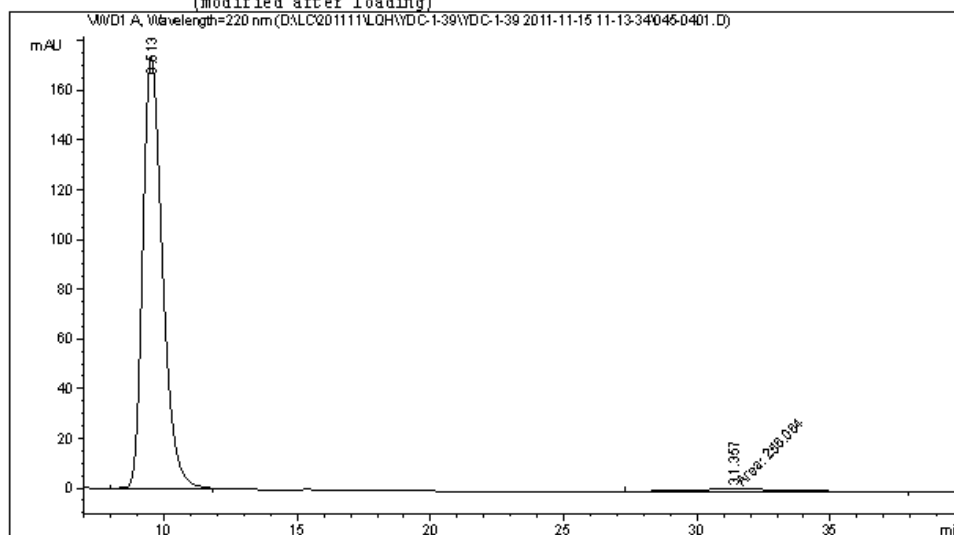
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	9.311	MM	0.8174	1096.75818	22.36259	50.7846
2	30.053	MM	3.7439	1062.87061	4.73154	49.2154

Totals : 2159.62878 27.09412



Data File D:\LC\201111\LQH\YDC-1-39\YDC-1-39 2011-11-15 11-13-34\045-0401.D
 Sample Name: HZL-4-66A

```
=====
Acq. Operator   : LQH                      Seq. Line :    4
Acq. Instrument : Instrument 1              Location  : Vial 45
Injection Date  : 11/15/2011 12:38:40 PM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\201111\LQH\YDC-1-39\YDC-1-39 2011-11-15 11-13-34\ASH-30-70-10ML-
                  220NM-40MIN.M
Last changed    : 10/29/2011 2:58:53 PM by HZL
Analysis Method : D:\LC\201111\LQH\YDC-1-39\YDC-1-39 2011-11-15 11-13-34\045-0401.D\DA.M (
                  ASH-30-70-10ML-220NM-40MIN.M)
Last changed    : 12/15/2011 4:58:16 PM by FX
                  (modified after loading)
=====
```



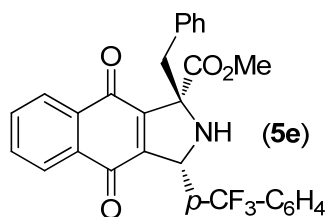
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VMD1 A, Wavelength=220 nm

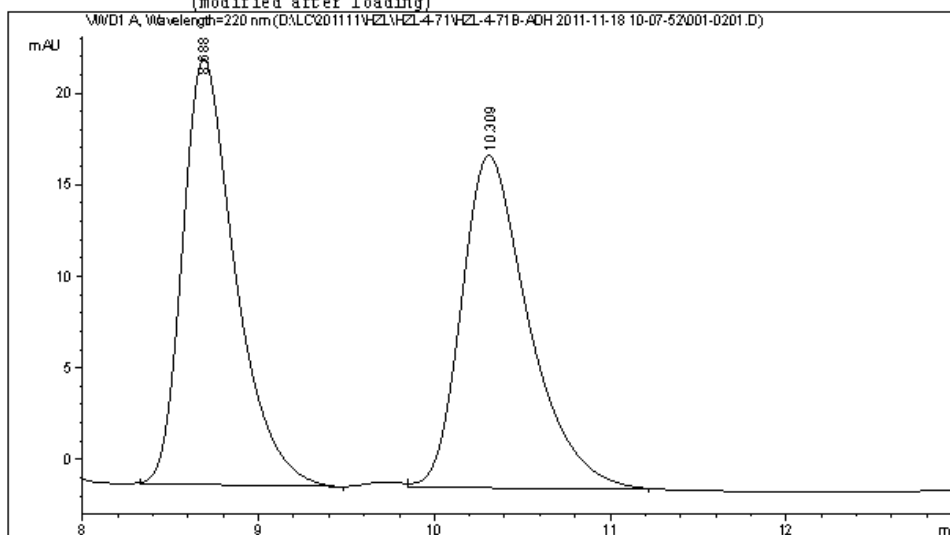
Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	9.513	VB	0.7973	8909.80176	173.02185	97.2063
2	31.357	MM	4.3574	256.06387	9.79429e-1	2.7937

Totals : 9165.86563 174.00128



Data File D:\LC\201111\H2L\H2L-4-71\H2L-4-71B-ADH 2011-11-18 10-07-52\001-0201.D
Sample Name: H2L-4-71B

```
=====
Acq. Operator   : hzl                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : Vial 1
Injection Date  : 11/18/2011 10:19:43 AM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\201111\H2L\H2L-4-71\H2L-4-71B-ADH 2011-11-18 10-07-52\ADH-30-70-
                  10ML-220NM.M
Last changed    : 9/15/2011 8:42:49 AM by THL
Analysis Method : D:\LC\201111\H2L\H2L-4-71\H2L-4-71B-ADH 2011-11-18 10-07-52\001-0201.D\DA.
                  M (ADH-30-70-10ML-220NM.M)
Last changed    : 12/15/2011 9:44:56 AM by FX
                  (modified after loading)
=====
```



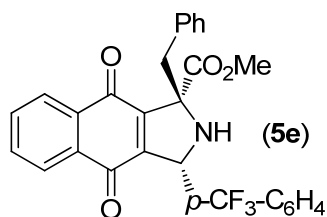
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.688	BB	0.3125	483.74359	23.26646	50.1151
2	10.309	VB	0.4000	481.52161	18.14019	49.8849

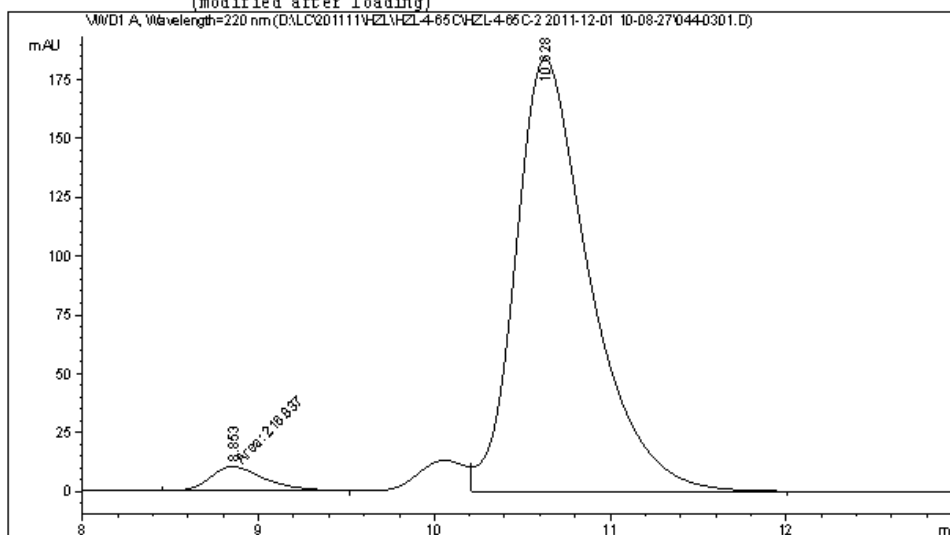
Totals : 965.26520 41.40665



Data File D:\LC\201111\H2L\H2L-4-65C\H2L-4-65C-2 2011-12-01 10-08-27\044-0301.D
Sample Name: H2L-4-88

```
=====
Acq. Operator   : H2L                      Seq. Line :    3
Acq. Instrument : Instrument 1              Location  : Vial 44
Injection Date  : 12/1/2011 10:42:22 AM     Inj       :    1
                                           Inj Volume: 5 µl

Acq. Method     : D:\LC\201111\H2L\H2L-4-65C\H2L-4-65C-2 2011-12-01 10-08-27\ADH-30-70-10ML-
220NM-20MIN.M
Last changed    : 11/19/2011 10:39:21 AM by THL
Analysis Method : D:\LC\201111\H2L\H2L-4-65C\H2L-4-65C-2 2011-12-01 10-08-27\044-0301.D\DA.M
(ADH-30-70-10ML-220NM-20MIN.M)
Last changed    : 12/15/2011 4:49:40 PM by FX
(modified after loading)
=====
```



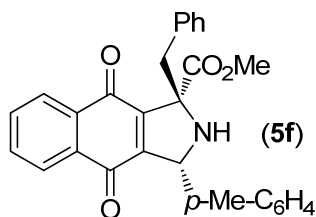
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=220 nm

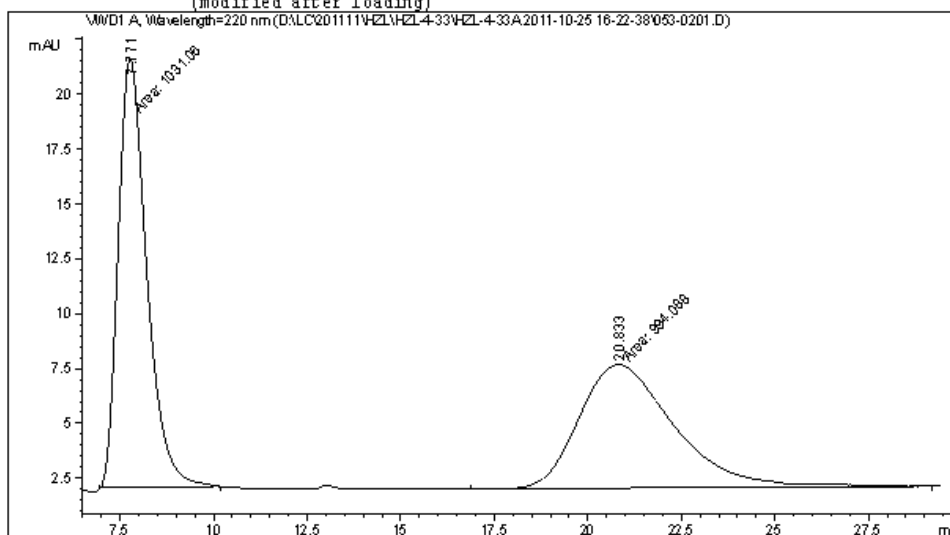
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.853	MM	0.3541	216.83698	10.20610	3.8809
2	10.628	VB	0.4364	5370.42090	184.09636	96.1191

Totals : 5587.25787 194.30246



Data File D:\LC\201111\HZZ\HZZ-4-33\HZZ-4-33A 2011-10-25 16-22-38\053-0201.D
Sample Name: HZZ-4-33A

```
=====
Acq. Operator   : hzl                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : Vial 53
Injection Date  : 10/25/2011 4:35:14 PM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\201111\HZZ\HZZ-4-33\HZZ-4-33A 2011-10-25 16-22-38\ASH-30-70-12ML-
220NM.M
Last changed    : 10/25/2011 4:20:13 PM by hzl
Analysis Method : D:\LC\201111\HZZ\HZZ-4-33\HZZ-4-33A 2011-10-25 16-22-38\053-0201.D\DA.M (
ASH-30-70-12ML-220NM.M)
Last changed    : 10/29/2011 3:04:44 PM by HZZ
                  (modified after loading)
=====
```



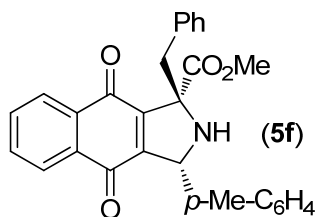
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.771	MM	0.8797	1031.05908	19.53331	50.9128
2	20.833	MM	2.9494	994.08826	5.61755	49.0872

Totals : 2025.14734 25.15086

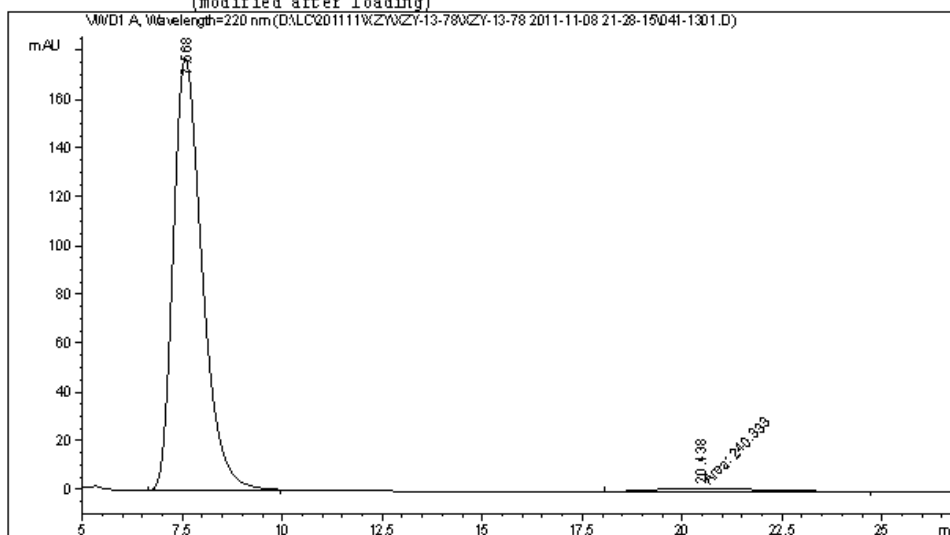


Data File D:\LC\201111\XYZY\XYZY-13-78\XYZY-13-78 2011-11-08 21-28-15\041-1301.D
Sample Name: HZL-4-59A

```

=====
Acq. Operator   : FX                      Seq. Line :   13
Acq. Instrument : Instrument 1             Location  : Vial 41
Injection Date  : 11/9/2011 8:02:49 AM     Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\201111\XYZY\XYZY-13-78\XYZY-13-78 2011-11-08 21-28-15\ASH-30-70-12ML-
220NM-30MIN.M
Last changed    : 10/29/2011 3:08:59 PM by HZL
Analysis Method : D:\LC\201111\XYZY\XYZY-13-78\XYZY-13-78 2011-11-08 21-28-15\041-1301.D\DA.M (
ASH-30-70-12ML-220NM-30MIN.M)
Last changed    : 11/9/2011 2:41:59 PM by THL
(modified after loading)
=====

```



Area Percent Report

```

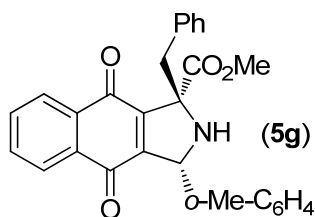
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: WVD1 A, Wavelength=220 nm

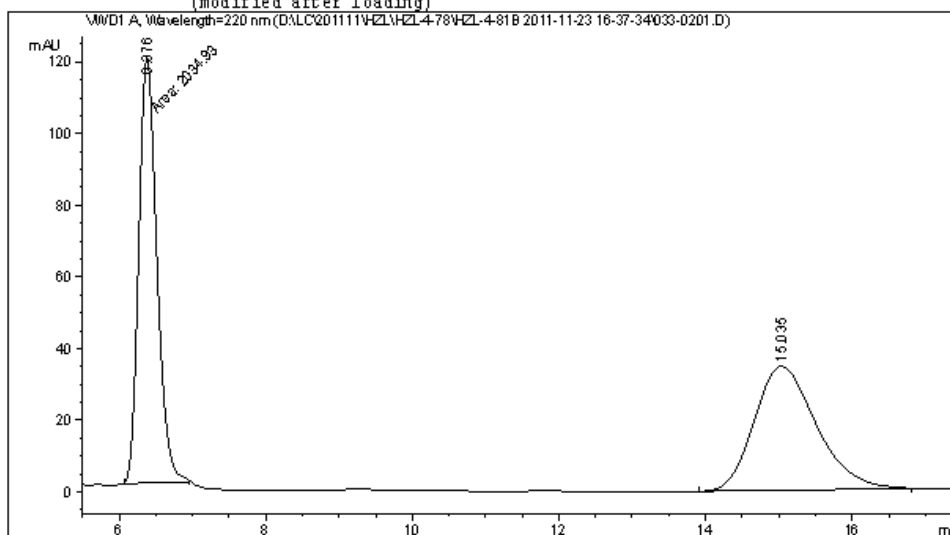
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.568	BB	0.7705	8895.69824	177.21434	97.3694
2	20.438	MM	2.7972	240.33339	1.43199	2.6306

Totals : 9136.03163 178.64633



Data File D:\LC\201111\H2L\H2L-4-78\H2L-4-81B 2011-11-23 16-37-34\033-0201.D
Sample Name: H2L-4-78A

```
=====
Acq. Operator   : HZL                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : Vial 33
Injection Date  : 11/23/2011 4:49:34 PM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\201111\H2L\H2L-4-78\H2L-4-81B 2011-11-23 16-37-34\0DH-30-70-10ML-
220NM.M
Last changed    : 9/14/2011 10:11:46 AM by HZL
Analysis Method : D:\LC\201111\H2L\H2L-4-78\H2L-4-81B 2011-11-23 16-37-34\033-0201.D\DA.M (
0DH-30-70-10ML-220NM.M)
Last changed    : 12/15/2011 10:07:15 AM by FX
(modified after loading)
=====
```



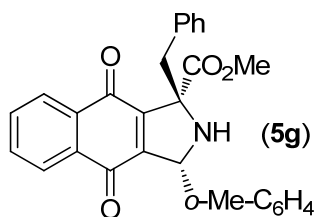
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=220 nm

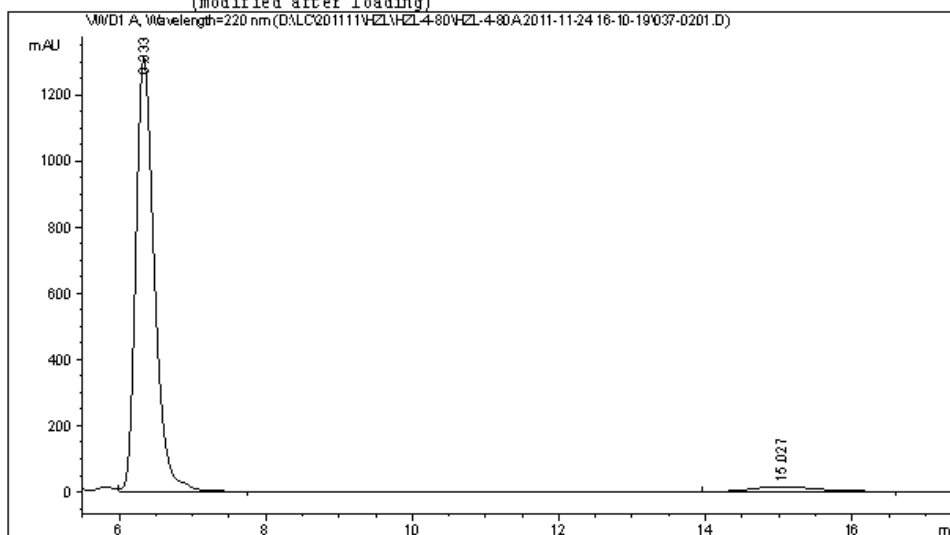
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.376	MM	0.2861	2034.92603	118.52831	49.8982
2	15.035	BB	0.9118	2043.22571	34.59540	50.1018

Totals : 4078.15173 153.12370



Data File D:\LC\201111\H2L\H2L-4-80\H2L-4-80A 2011-11-24 16-10-19\037-0201.D
Sample Name: H2L-4-80A

```
=====
Acq. Operator   : HZL                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : Vial 37
Injection Date  : 11/24/2011 4:22:20 PM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\201111\H2L\H2L-4-80\H2L-4-80A 2011-11-24 16-10-19\0DH-30-70-10ML-
                  220NM-20MIN.M
Last changed    : 11/24/2011 3:51:31 PM by HZL
Analysis Method : D:\LC\201111\H2L\H2L-4-80\H2L-4-80A 2011-11-24 16-10-19\037-0201.D\DA.M (
                  0DH-30-70-10ML-220NM-20MIN.M)
Last changed    : 12/15/2011 10:09:25 AM by FX
                  (modified after loading)
=====
```



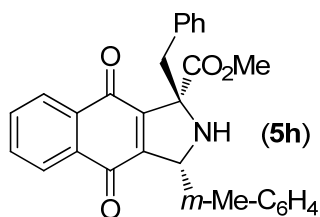
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=220 nm

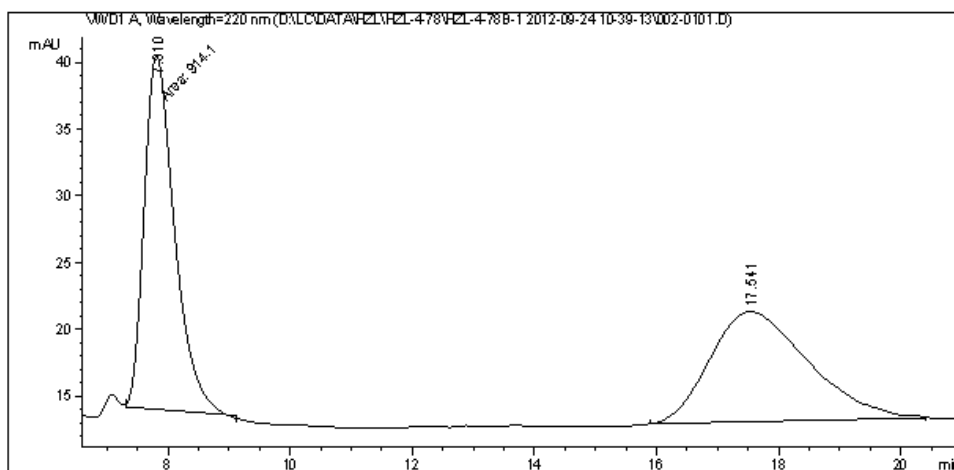
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	6.333	VB	0.2688	2.29704e4	1308.67627	96.3848
2	15.027	BB	0.8856	861.57837	14.93419	3.6152

Totals : 2.38320e4 1323.61046



Data File D:\LC\DATA\H2L\H2L-4-78\H2L-4-78B-1 2012-09-24 10-39-13\002-0101.D
Sample Name: H2L-4-78B

```
=====
Acq. Operator   : FX                               Seq. Line :    1
Acq. Instrument : Instrument 1                     Location  : Vial 2
Injection Date  : 9/24/2012 10:40:38 AM            Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method     : D:\LC\DATA\H2L\H2L-4-78\H2L-4-78B-1 2012-09-24 10-39-13\ASH-30-70-1ML-
220NM.M
Last changed    : 1/13/2012 10:19:59 AM by LOH
Analysis Method : D:\LC\DATA\H2L\H2L-4-78\H2L-4-78B-1 2012-09-24 10-39-13\002-0101.D\DA.M (
ASH-30-70-1ML-220NM.M)
Last changed    : 9/24/2012 11:21:06 AM by FX
                  (modified after loading)
Method Info     : ASH-50-50-1ML-254NM-50MIN
=====
```



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

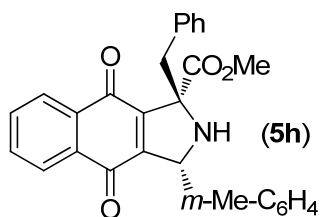
```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VMD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	7.810	MM	0.5742	914.09973	26.53284	49.7468
2	17.541	BB	1.3159	923.40363	8.26015	50.2532

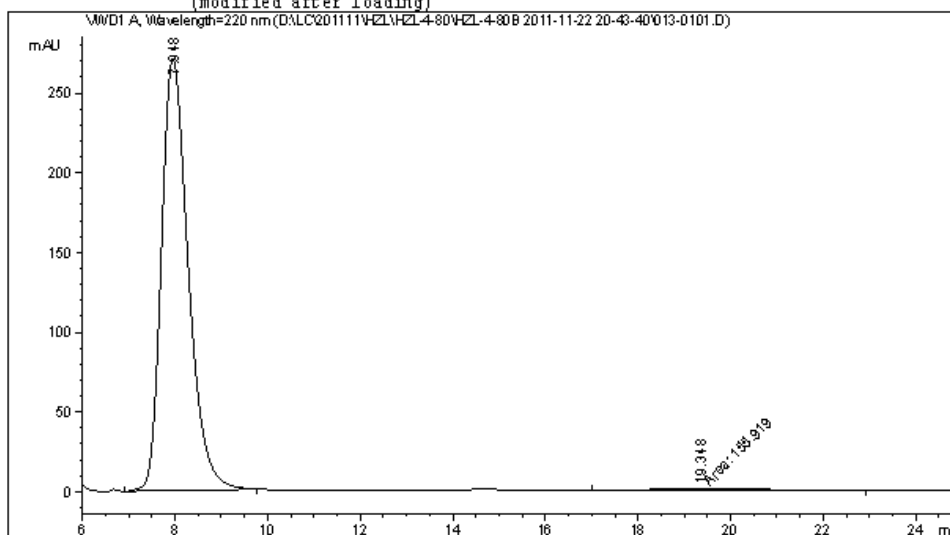
```
Totals :                      1837.50336  34.79298
```

```
=====
*** End of Report ***
```



Data File D:\LC\201111\H2L\H2L-4-80\H2L-4-80B 2011-11-22 20-43-40\013-0101.D
Sample Name: H2L-4-80B

```
=====
Acq. Operator   : H2L                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 13
Injection Date  : 11/22/2011 8:44:57 PM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\201111\H2L\H2L-4-80\H2L-4-80B 2011-11-22 20-43-40\ASH-30-70-10ML-
220NM-30MIN.M
Last changed    : 11/22/2011 8:35:29 PM by THL
Analysis Method : D:\LC\201111\H2L\H2L-4-80\H2L-4-80B 2011-11-22 20-43-40\013-0101.D\DA.M (
ASH-30-70-10ML-220NM-30MIN.M)
Last changed    : 12/15/2011 10:16:49 AM by FX
                  (modified after loading)
=====
```



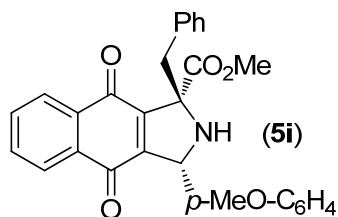
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.948	VB	0.6202	1.09292e4	271.14075	98.5934
2	19.348	MM	2.3333	155.91867	1.11373	1.4066

Totals : 1.10851e4 272.25447

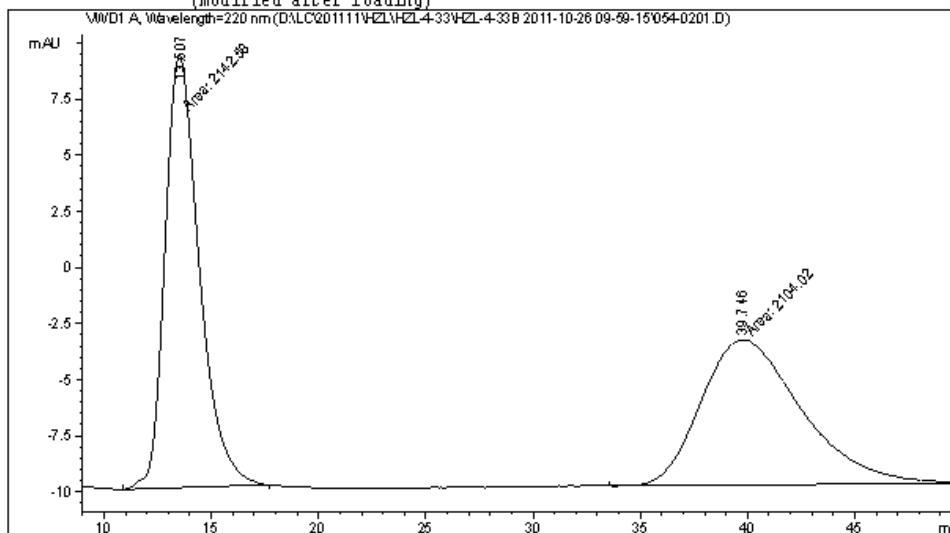


Data File D:\LC\201111\HZZ\HZZ-4-33\HZZ-4-33B 2011-10-26 09-59-15\054-0201.D
Sample Name: HZZ-4-33B

```

=====
Acq. Operator   : HZZ                               Seq. Line :    2
Acq. Instrument : Instrument 1                       Location  : Vial 54
Injection Date  : 10/26/2011 10:11:44 AM             Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method     : D:\LC\201111\HZZ\HZZ-4-33\HZZ-4-33B 2011-10-26 09-59-15\ASH-30-70-12ML-
220NM.M
Last changed    : 10/25/2011 4:20:13 PM by hzz
Analysis Method : D:\LC\201111\HZZ\HZZ-4-33\HZZ-4-33B 2011-10-26 09-59-15\054-0201.D\DA.M (
ASH-30-70-12ML-220NM.M)
Last changed    : 10/29/2011 3:28:05 PM by HZZ
(modified after loading)
=====

```



Area Percent Report

```

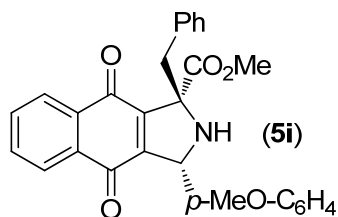
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs

```

Signal 1: VWD1 A, Wavelength=220 nm

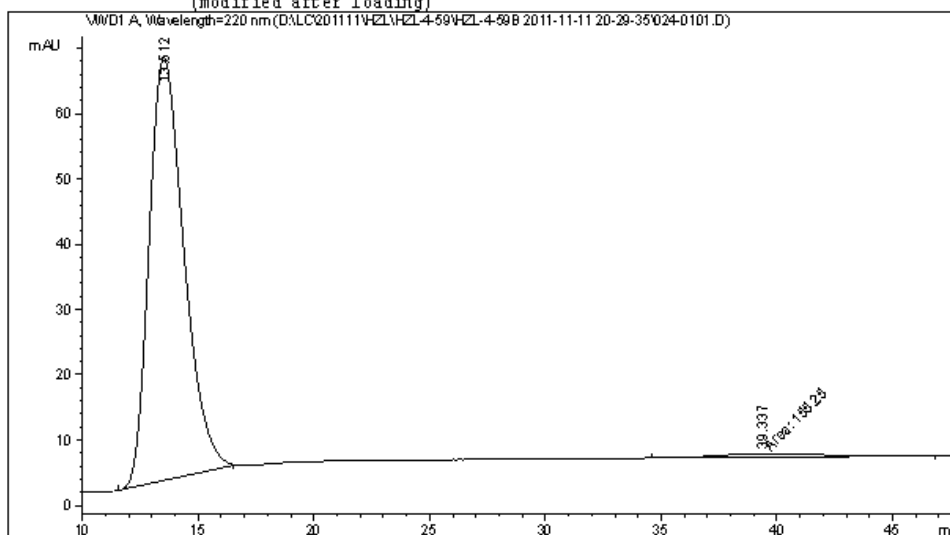
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.507	MM	1.8584	2142.58325	19.21558	50.4540
2	39.746	MM	5.4205	2104.02490	6.46932	49.5460

Totals : 4246.60815 25.68490



Data File D:\LC\201111\HZZ\HZZ-4-59\HZZ-4-59B 2011-11-11 20-29-35\024-0101.D
Sample Name: HZZ-4-59B

```
=====
Acq. Operator   : HZZ                               Seq. Line :    1
Acq. Instrument : Instrument 1                       Location  : Vial 24
Injection Date  : 11/11/2011 8:30:54 PM              Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method     : D:\LC\201111\HZZ\HZZ-4-59\HZZ-4-59B 2011-11-11 20-29-35\ASH-30-70-12ML-
220NM-50MIN.M
Last changed    : 10/29/2011 3:52:23 PM by HZZ
Analysis Method : D:\LC\201111\HZZ\HZZ-4-59\HZZ-4-59B 2011-11-11 20-29-35\024-0101.D\DA.M (
ASH-30-70-12ML-220NM-50MIN.M)
Last changed    : 11/11/2011 9:30:27 PM by THL
(modified after loading)
=====
```



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

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

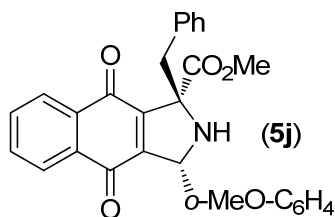
Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	13.512	BB	1.6053	6780.93555	64.64053	97.7617
2	39.337	MM	4.9252	155.24968	5.25358e-1	2.2383

Totals : 6936.18523 65.16589

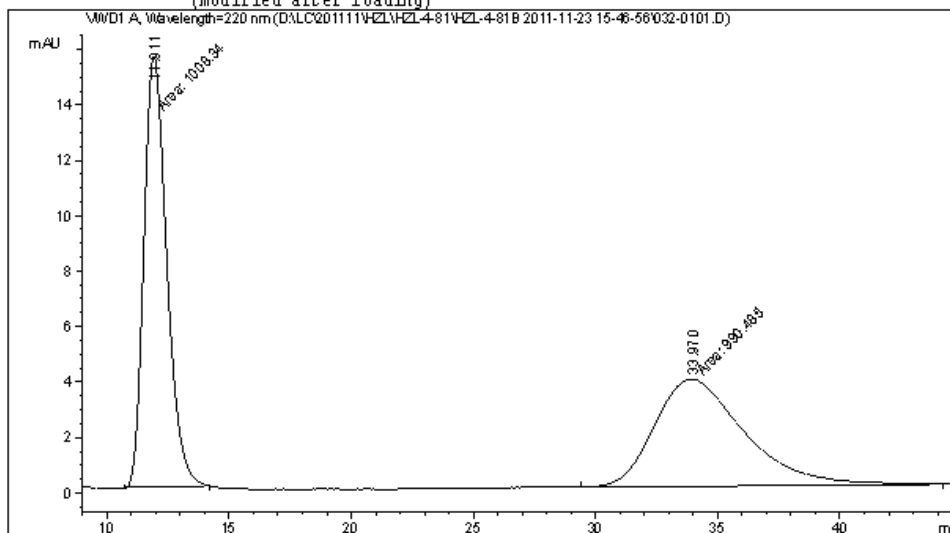
Instrument 1 11/11/2011 9:30:38 PM THL

Page 1 of 1



Data File D:\LC\201111\H2L\H2L-4-81\H2L-4-81B 2011-11-23 15-46-56\032-0101.D
Sample Name: H2L-4-81B

```
=====
Acq. Operator   : HZL                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 32
Injection Date  : 11/23/2011 3:48:45 PM     Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\201111\H2L\H2L-4-81\H2L-4-81B 2011-11-23 15-46-56\ASH-30-70-10ML-
220NM.M
Last changed    : 10/20/2011 6:42:48 PM by HZL
Analysis Method : D:\LC\201111\H2L\H2L-4-81\H2L-4-81B 2011-11-23 15-46-56\032-0101.D\DA.M (
ASH-30-70-10ML-220NM.M)
Last changed    : 12/15/2011 10:22:14 AM by FX
                  (modified after loading)
=====
```



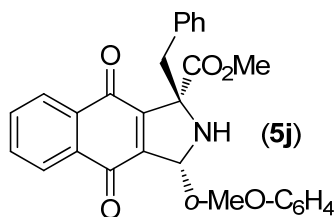
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=220 nm

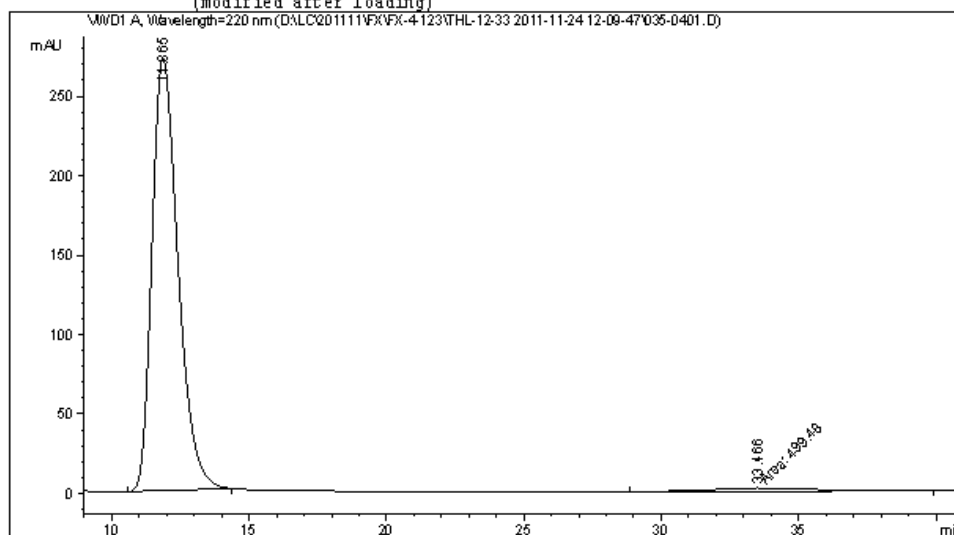
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.911	MM	1.0821	1008.33783	15.53055	50.4466
2	33.970	MM	4.2712	990.48456	3.86496	49.5534

Totals : 1998.82239 19.39551



Data File D:\LC\201111\FX\FX-4-123\THL-12-33 2011-11-24 12-09-47\035-0401.D
 Sample Name: HZL-4-83A

```
=====
Acq. Operator   : FX                               Seq. Line :    4
Acq. Instrument : Instrument 1                     Location  : Vial 35
Injection Date  : 11/24/2011 12:55:19 PM           Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method     : D:\LC\201111\FX\FX-4-123\THL-12-33 2011-11-24 12-09-47\ASH-30-70-10ML-
                  220NM-45MIN.M
Last changed    : 11/24/2011 12:08:10 PM by hzl
Analysis Method : D:\LC\201111\FX\FX-4-123\THL-12-33 2011-11-24 12-09-47\035-0401.D\DA.M (
                  ASH-30-70-10ML-220NM-45MIN.M)
Last changed    : 11/24/2011 2:27:08 PM by hzl
                  (modified after loading)
=====
```



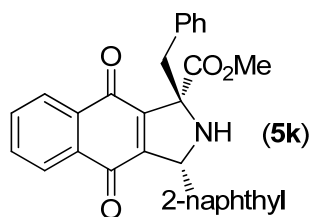
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VMD1 A, Wavelength=220 nm

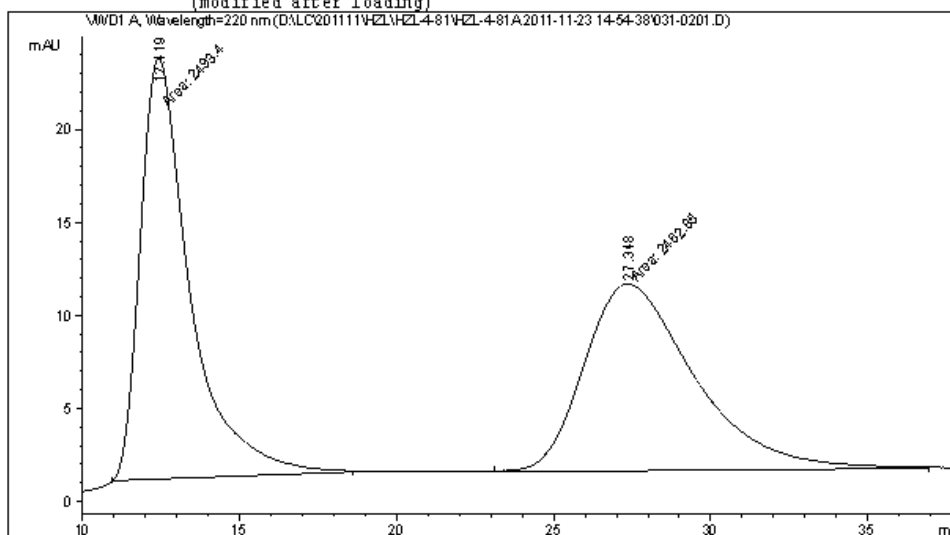
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.865	BB	1.0140	1.79072e4	272.05200	97.2864
2	33.466	MM	4.0919	499.47961	2.03443	2.7136

Totals : 1.84067e4 274.08644



Data File D:\LC\201111\H2L\H2L-4-81\H2L-4-81A 2011-11-23 14-54-38\031-0201.D
Sample Name: H2L-4-81A

```
=====
Acq. Operator   : H2L                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : Vial 31
Injection Date  : 11/23/2011 3:07:03 PM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\201111\H2L\H2L-4-81\H2L-4-81A 2011-11-23 14-54-38\ASH-30-70-10ML-
220NM.M
Last changed    : 10/20/2011 6:42:48 PM by H2L
Analysis Method : D:\LC\201111\H2L\H2L-4-81\H2L-4-81A 2011-11-23 14-54-38\031-0201.D\DA.M (
ASH-30-70-10ML-220NM.M)
Last changed    : 11/23/2011 3:49:03 PM by h2l
                  (modified after loading)
=====
```



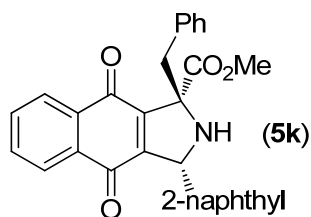
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=220 nm

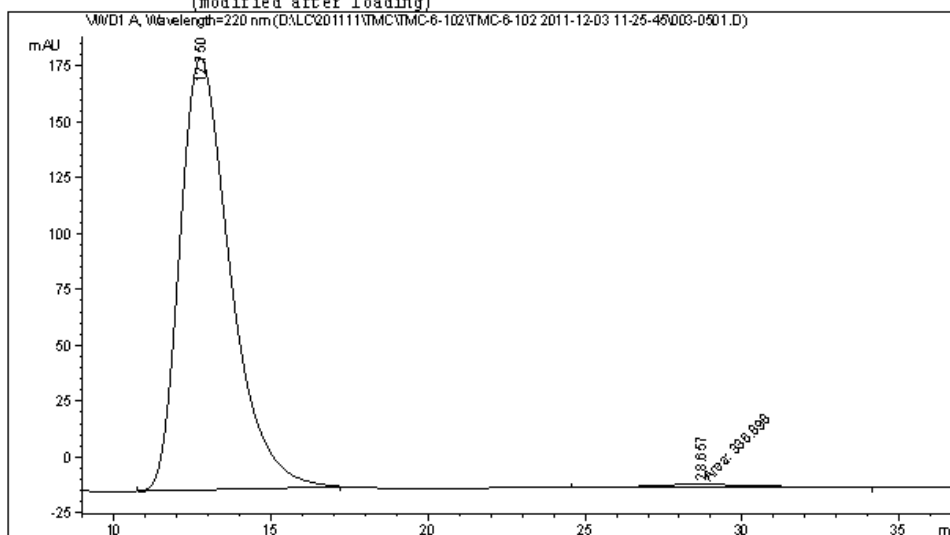
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.419	MM	1.8377	2493.40479	22.61336	50.3082
2	27.348	MM	4.0914	2462.84985	10.03253	49.6918

Totals : 4956.25464 32.64589



Data File D:\LC\201111\TMC\TMC-6-102\TMC-6-102 2011-12-03 11-25-45\003-0501.D
Sample Name: HZL-4-95C

```
=====
Acq. Operator   : TMC                      Seq. Line :    5
Acq. Instrument : Instrument 1              Location  : Vial 3
Injection Date  : 12/3/2011 12:52:22 PM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\201111\TMC\TMC-6-102\TMC-6-102 2011-12-03 11-25-45\ASH-30-70-10ML-
                                           220NM-40MIN.M
Last changed    : 10/29/2011 2:58:53 PM by HZL
Analysis Method : D:\LC\201111\TMC\TMC-6-102\TMC-6-102 2011-12-03 11-25-45\003-0501.D\DA.M (
                                           ASH-30-70-10ML-220NM-40MIN.M)
Last changed    : 12/3/2011 3:15:56 PM by LQH
                                           (modified after loading)
=====
```



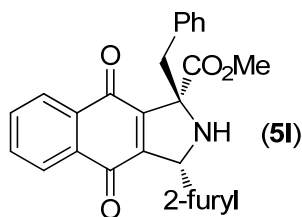
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	12.750	BB	1.6962	2.15630e4	193.26617	98.4616
2	28.657	MM	4.0378	336.89764	1.39061	1.5384

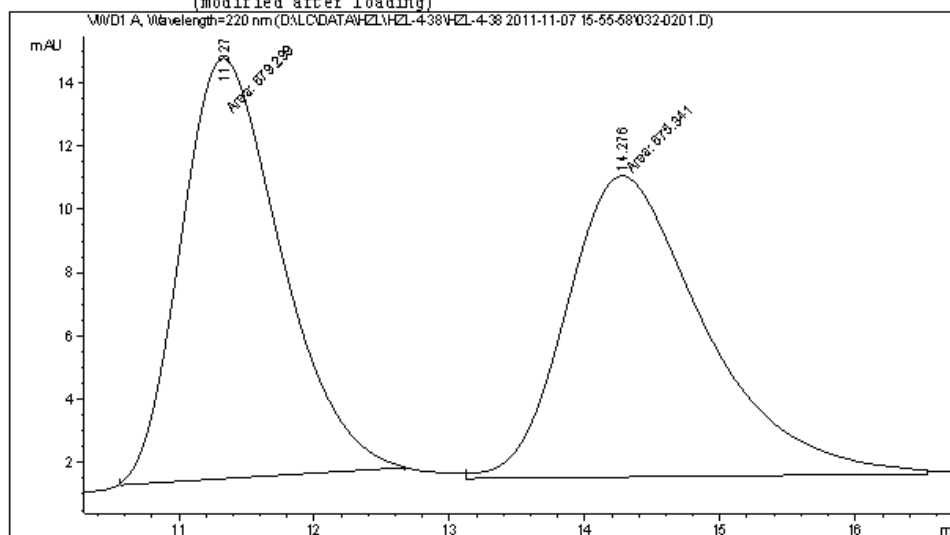
Totals : 2.18999e4 194.65679



Data File D:\LC\DATA\H2L\H2L-4-38\H2L-4-38 2011-11-07 15-55-58\032-0201.D
 Sample Name: H2L-4-38A

```
=====
Acq. Operator   : H2L                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : Vial 32
Injection Date  : 11/7/2011 4:08:24 PM      Inj       :    1
                                           Inj Volume: 5 µl

Acq. Method     : D:\LC\201111\H2L\H2L-4-38\H2L-4-38 2011-11-07 15-55-58\ASH-30-70-10ML-
                  220NM.M
Last changed    : 10/20/2011 6:42:48 PM by H2L
Analysis Method : D:\LC\DATA\H2L\H2L-4-38\H2L-4-38 2011-11-07 15-55-58\032-0201.D\DA.M (ASH-
                  30-70-10ML-220NM.M)
Last changed    : 9/24/2012 10:00:52 AM by FX
                  (modified after loading)
=====
```



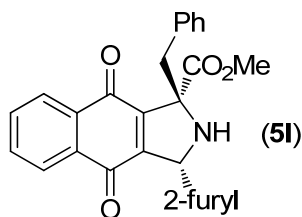
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VMD1 A, Wavelength=220 nm

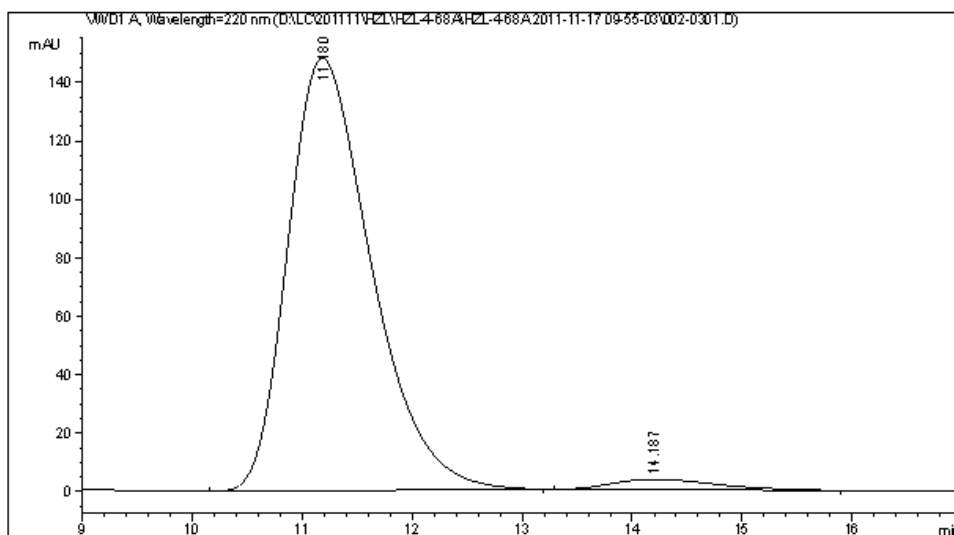
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	11.327	MM	0.8527	679.29883	13.27685	50.1461
2	14.276	MM	1.1835	675.34106	9.51031	49.8539

Totals : 1354.63989 22.78717



Data File D:\LC\201111\H2L\H2L-4-68A\H2L-4-68A 2011-11-17 09-55-03\002-0301.D
Sample Name: H2L-4-68B

```
=====
Acq. Operator   : hzl                      Seq. Line :    3
Acq. Instrument : Instrument 1              Location  : Vial 2
Injection Date  : 11/17/2011 10:49:23 AM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\201111\H2L\H2L-4-68A\H2L-4-68A 2011-11-17 09-55-03\ASH-30-70-10ML-
220NM-20MIN.M
Last changed    : 11/10/2011 9:40:34 PM by THL
Analysis Method : D:\LC\201111\H2L\H2L-4-68A\H2L-4-68A 2011-11-17 09-55-03\002-0301.D\DA.M (
ASH-30-70-10ML-220NM-20MIN.M)
Last changed    : 11/17/2011 11:18:16 AM by hzl
                  (modified after loading)
Method Info     : ASH-50-50-1ML-254NM-50MIN
=====
```



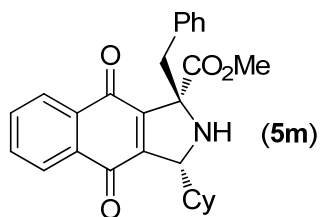
```
=====
                        Area Percent Report
=====
```

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VMD1 A, Wavelength=220 nm

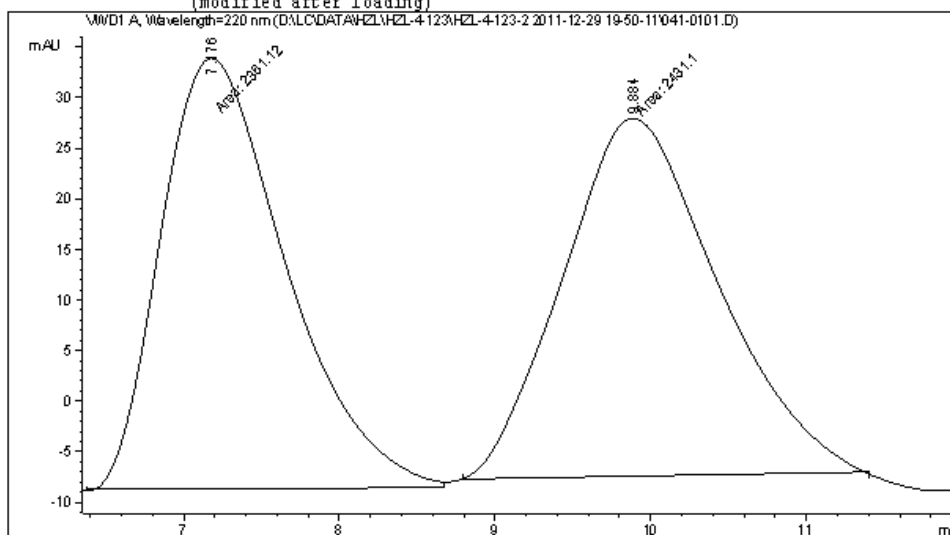
Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]
1	11.180	VB	0.8114	7800.07568	97.0599	148.01219
2	14.187	BB	0.8576	236.27974	2.9401	3.60615

Totals : 8036.35542 151.61835



Data File D:\LC\DATA\H2L\H2L-4-123\H2L-4-123-2 2011-12-29 19-50-11\041-0101.D
 Sample Name: H2L-4-123

```
=====
Acq. Operator   : H2L                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 41
Injection Date  : 12/29/2011 7:51:00 PM     Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\201112\H2L\H2L-4-123\H2L-4-123-2 2011-12-29 19-50-11\ASH-20-80-10ML-
                  220NM.M
Last changed    : 8/29/2011 3:56:33 PM by H2L
Analysis Method : D:\LC\DATA\H2L\H2L-4-123\H2L-4-123-2 2011-12-29 19-50-11\041-0101.D\DA.M (
                  ASH-20-80-10ML-220NM.M)
Last changed    : 9/24/2012 9:58:45 AM by FX
                  (modified after loading)
=====
```



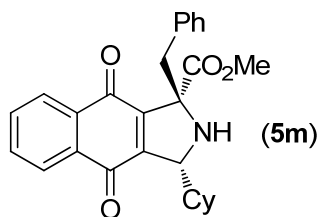
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=220 nm

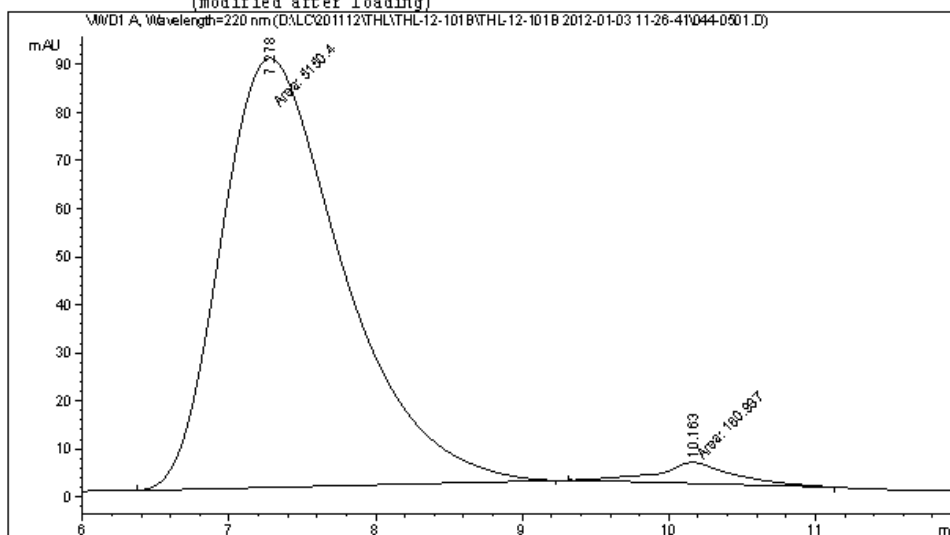
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.176	MM	0.9249	2361.12451	42.54876	49.2699
2	9.884	MM	1.1467	2431.10254	35.33603	50.7301

Totals : 4792.22705 77.88478



Data File D:\LC\201112\THL\THL-12-101B\THL-12-101B 2012-01-03 11-26-41\044-0501.D
Sample Name: HZL-4-129

```
=====
Acq. Operator   : THL                      Seq. Line :    5
Acq. Instrument : Instrument 1              Location  : Vial 44
Injection Date  : 1/3/2012 1:22:49 PM      Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\201112\THL\THL-12-101B\THL-12-101B 2012-01-03 11-26-41\ASH-20-80-
                  10ML-220NM-15MIN.M
Last changed    : 12/15/2011 4:27:37 PM by FX
Analysis Method : D:\LC\201112\THL\THL-12-101B\THL-12-101B 2012-01-03 11-26-41\044-0501.D\
                  DA.M (ASH-20-80-10ML-220NM-15MIN.M)
Last changed    : 1/3/2012 2:52:14 PM by LQH
                  (modified after loading)
=====
```



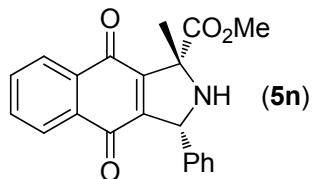
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=220 nm

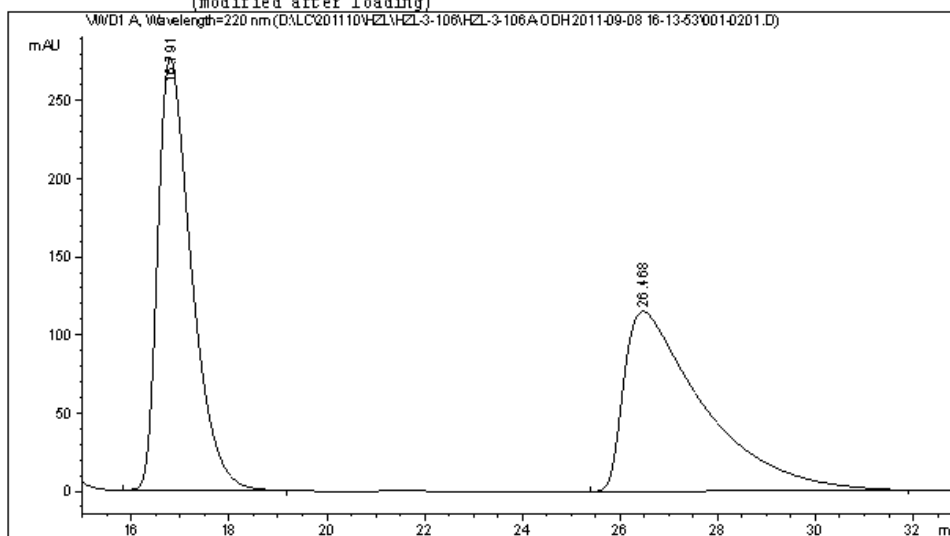
Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]
1	7.278	MM	0.9613	5150.39746	96.9699	89.29688
2	10.163	MM	0.6137	160.93723	3.0301	4.37035

Totals : 5311.33469 93.66722



Data File D:\LC\201110\H2L\H2L-3-106\H2L-3-106A-ODH 2011-09-08 16-13-53\001-0201.D
 Sample Name: H2L-3-106A

```
=====
Acq. Operator   : H2L                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : Vial 1
Injection Date  : 9/8/2011 4:25:43 PM      Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\H2L\Data\H2L-3-106\H2L-3-106A-ODH 2011-09-08 16-13-53\ODH-20-80-
                                           10ML-220NM.M
Last changed    : 9/8/2011 4:11:48 PM by H2L
Analysis Method : D:\LC\201110\H2L\H2L-3-106\H2L-3-106A-ODH 2011-09-08 16-13-53\001-0201.D\
                                           DA.M (ODH-20-80-10ML-220NM.M)
Last changed    : 11/8/2011 5:11:36 PM by THL
                                           (modified after loading)
=====
```



Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

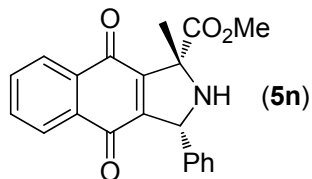
Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Height [mAU]	Area %
1	16.791	BB	0.7278	1.32454e4	276.51071	50.4540
2	26.468	BB	1.6016	1.30070e4	115.03537	49.5460

Totals : 2.62525e4 391.54608

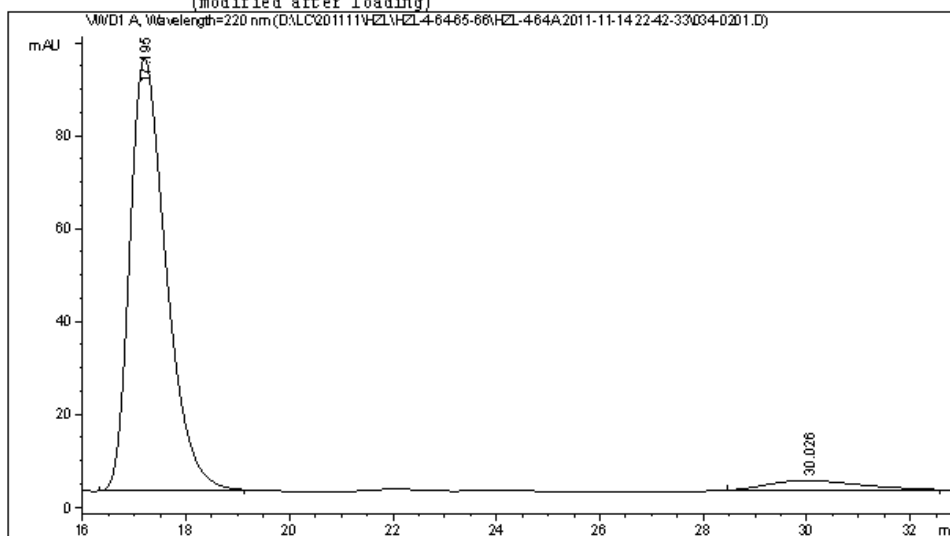
Instrument 1 11/8/2011 5:11:44 PM THL

Page 1 of 1



Data File D:\LC\201111\H2L\H2L-4-64-65-66\H2L-4-64A 2011-11-14 22-42-33\034-0201.D
 Sample Name: H2L-4-64A

```
=====
Acq. Operator   : hzl                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : Vial 34
Injection Date  : 11/14/2011 10:54:30 PM   Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\201111\H2L\H2L-4-64-65-66\H2L-4-64A 2011-11-14 22-42-33\ODH-20-80-
                  10ML-220NM-35MIN.M
Last changed    : 9/13/2011 6:04:32 PM by HZL
Analysis Method : D:\LC\201111\H2L\H2L-4-64-65-66\H2L-4-64A 2011-11-14 22-42-33\034-0201.D\
                  DA.M (ODH-20-80-10ML-220NM-35MIN.M)
Last changed    : 12/15/2011 4:35:18 PM by FX
                  (modified after loading)
=====
```



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

Sorted By : Signal
 Multiplier : 1.0000
 Dilution : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

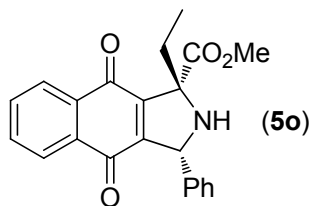
Signal 1: WWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]
1	17.195	BB	0.7464	4539.73438	94.5845	93.12305
2	30.026	BB	1.4737	259.92557	5.4155	2.07204

Totals : 4799.65994 95.19509

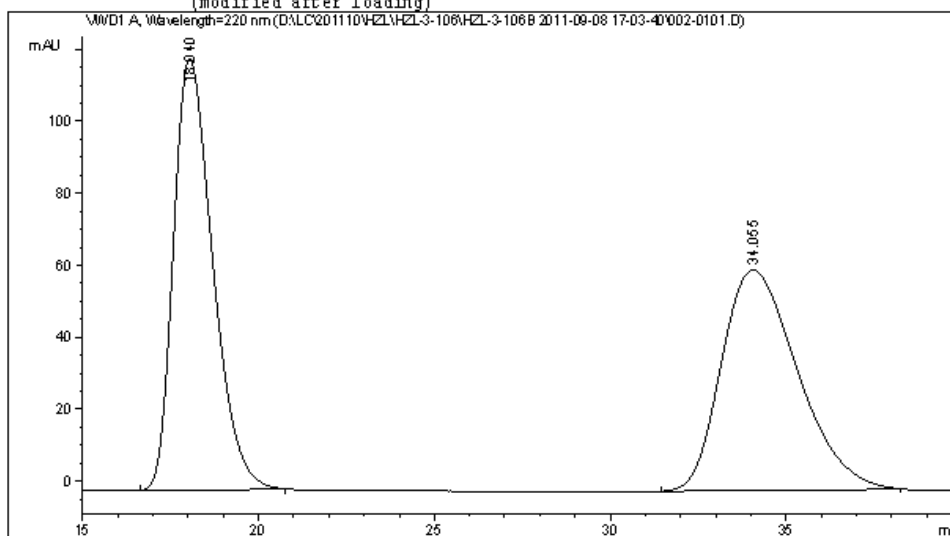
Instrument 1 12/15/2011 4:35:23 PM FX

Page 1 of 1



Data File D:\LC\201110\HZZ\HZZ-3-106\HZZ-3-106B 2011-09-08 17-03-40\002-0101.D
 Sample Name: HZZ-3-106B

```
=====
Acq. Operator   : HZZ                               Seq. Line :    1
Acq. Instrument : Instrument 1                       Location  : Vial 2
Injection Date  : 9/8/2011 5:04:54 PM                Inj       :    1
                                                    Inj Volume: 5 µl
Acq. Method     : D:\LC\HZZ\Data\HZZ-3-106\HZZ-3-106B 2011-09-08 17-03-40\ASH-20-80-10ML-
                  220NM.M
Last changed    : 8/29/2011 3:56:33 PM by HZZ
Analysis Method : D:\LC\201110\HZZ\HZZ-3-106\HZZ-3-106B 2011-09-08 17-03-40\002-0101.D\DA.M
                  (ASH-20-80-10ML-220NM.M)
Last changed    : 11/8/2011 5:10:33 PM by THL
                  (modified after loading)
=====
```

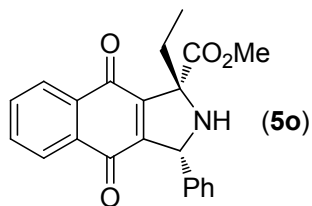


Area Percent Report

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

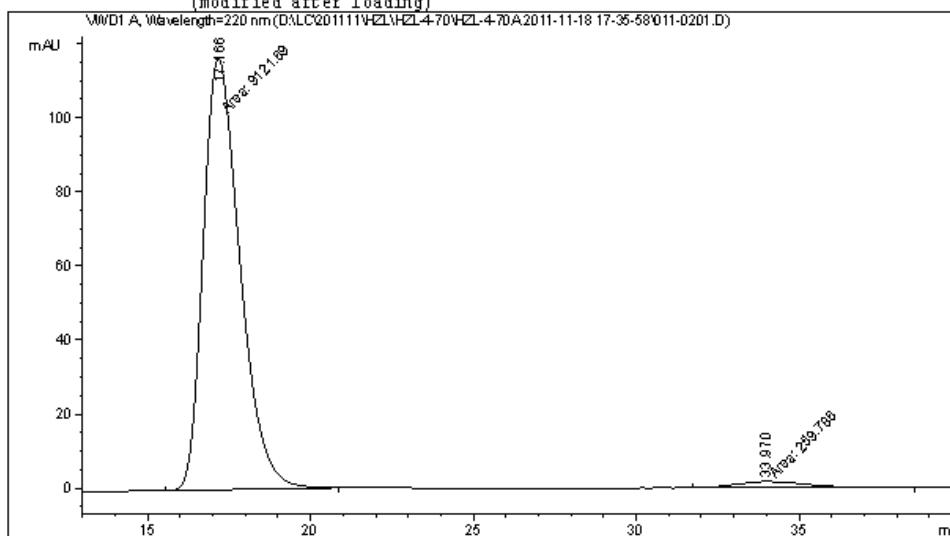
Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]
1	18.040	BB	1.1901	9286.13867	50.2003	119.93852
2	34.055	BB	2.2053	9212.01758	49.7997	61.32467
Totals :				1.84982e4	181.26320	



Data File D:\LC\201111\H2L\H2L-4-70\H2L-4-70A 2011-11-18 17-35-58\011-0201.D
Sample Name: H2L-4-70A

```
=====
Acq. Operator   : H2L                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : Vial 11
Injection Date  : 11/18/2011 5:47:53 PM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\201111\H2L\H2L-4-70\H2L-4-70A 2011-11-18 17-35-58\ASH-20-80-10ML-
                  220NM-45MIN.M
Last changed    : 8/29/2011 3:55:38 PM by H2L
Analysis Method : D:\LC\201111\H2L\H2L-4-70\H2L-4-70A 2011-11-18 17-35-58\011-0201.D\DA.M (
                  ASH-20-80-10ML-220NM-45MIN.M)
Last changed    : 11/18/2011 6:35:11 PM by h2l
                  (modified after loading)
=====
```



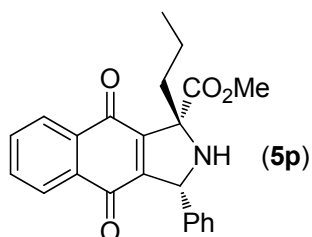
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=220 nm

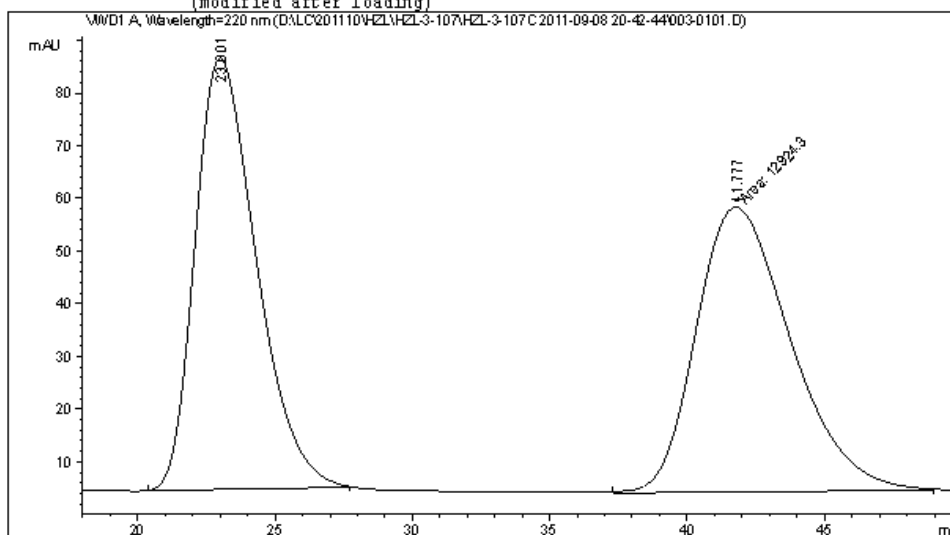
Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]
1	17.166	MM	1.3060	9121.68848	97.2309	116.40973
2	33.970	MM	2.5427	259.78558	2.7691	1.70282

Totals : 9381.47406 118.11255



Data File D:\LC\201110\H2L\H2L-3-107\H2L-3-107C 2011-09-08 20-42-44\003-0101.D
Sample Name: H2L-3-107C

```
=====
Acq. Operator   : H2L                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 3
Injection Date  : 9/8/2011 8:44:06 PM      Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\H2L\Data\H2L-3-107\H2L-3-107C 2011-09-08 20-42-44\ASH-20-80-10ML-
220NM.M
Last changed    : 8/29/2011 3:56:33 PM by H2L
Analysis Method : D:\LC\201110\H2L\H2L-3-107\H2L-3-107C 2011-09-08 20-42-44\003-0101.D\DA.M
(ASH-20-80-10ML-220NM.M)
Last changed    : 11/8/2011 5:09:48 PM by THL
(modified after loading)
=====
```



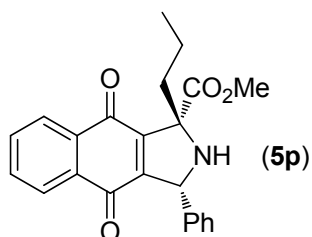
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: WVD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	23.001	BB	2.3299	1.26838e4	81.81721	49.5305
2	41.777	MM	3.9866	1.29243e4	54.03196	50.4695

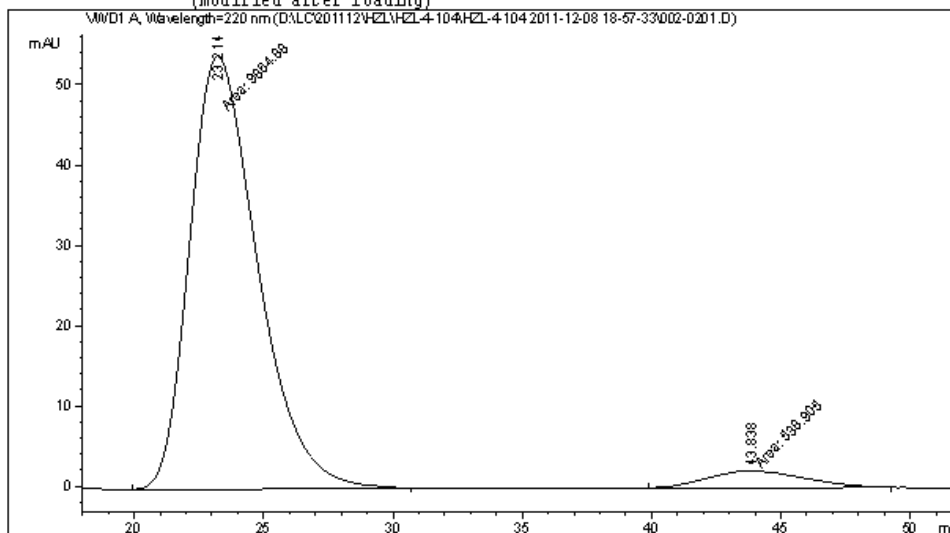
Totals : 2.56081e4 135.84917



Data File D:\LC\201112\H2L\H2L-4-104\H2L-4-104 2011-12-08 18-57-33\002-0201.D
Sample Name: H2L-4-104

```
=====
Acq. Operator   : H2L                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : Vial 2
Injection Date  : 12/8/2011 7:10:08 PM      Inj       :    1
                                           Inj Volume: 5 µl

Acq. Method     : D:\LC\201112\H2L\H2L-4-104\H2L-4-104 2011-12-08 18-57-33\ASH-20-80-10ML-
220NM-55MIN.M
Last changed    : 9/13/2011 11:19:37 AM by FX
Analysis Method : D:\LC\201112\H2L\H2L-4-104\H2L-4-104 2011-12-08 18-57-33\002-0201.D\DA.M (
ASH-20-80-10ML-220NM-55MIN.M)
Last changed    : 12/15/2011 10:42:24 AM by FX
                  (modified after loading)
=====
```



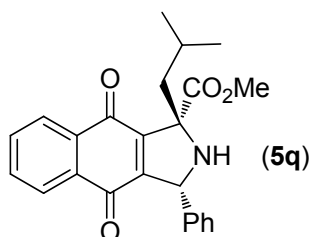
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=220 nm

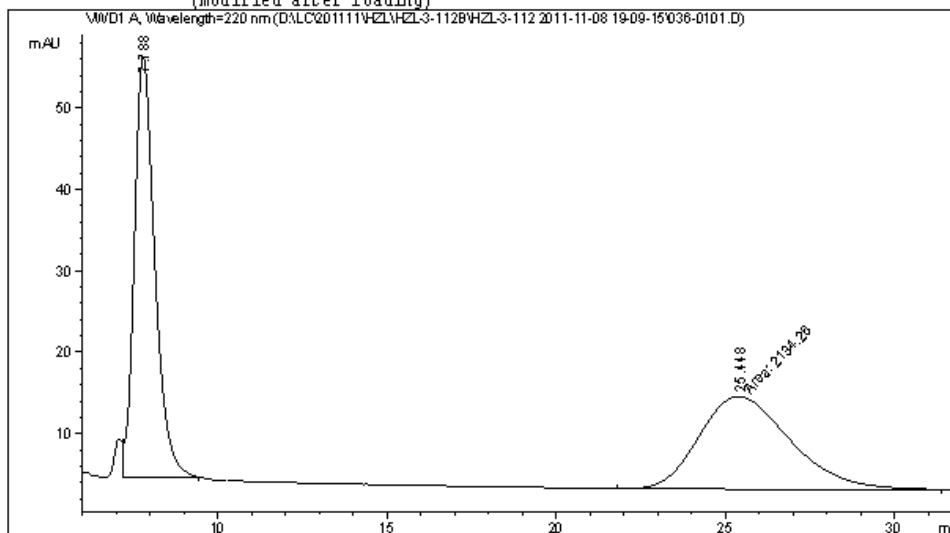
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	23.214	MM	3.0470	9864.87988	53.95921	94.8201
2	43.838	MM	4.3166	538.90503	2.08077	5.1799

Totals : 1.04038e4 56.03997



Data File D:\LC\201111\H2L\H2L-3-112B\H2L-3-112 2011-11-08 19-09-15\036-0101.D
Sample Name: H2L-3-112B

```
=====
Acq. Operator   : hzl                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 36
Injection Date  : 11/8/2011 7:11:11 PM      Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\201111\H2L\H2L-3-112B\H2L-3-112 2011-11-08 19-09-15\ASH-30-70-10ML-
                  220NM.M
Last changed    : 10/20/2011 6:42:48 PM by HZL
Analysis Method : D:\LC\201111\H2L\H2L-3-112B\H2L-3-112 2011-11-08 19-09-15\036-0101.D\DA.M
                  (ASH-30-70-10ML-220NM.M)
Last changed    : 11/8/2011 7:44:47 PM by THL
                  (modified after loading)
=====
```



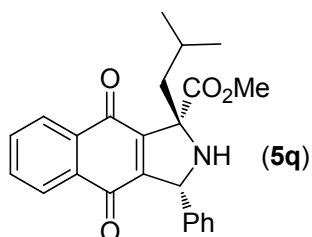
=====
Area Percent Report
=====

Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: VWD1 A, Wavelength=220 nm

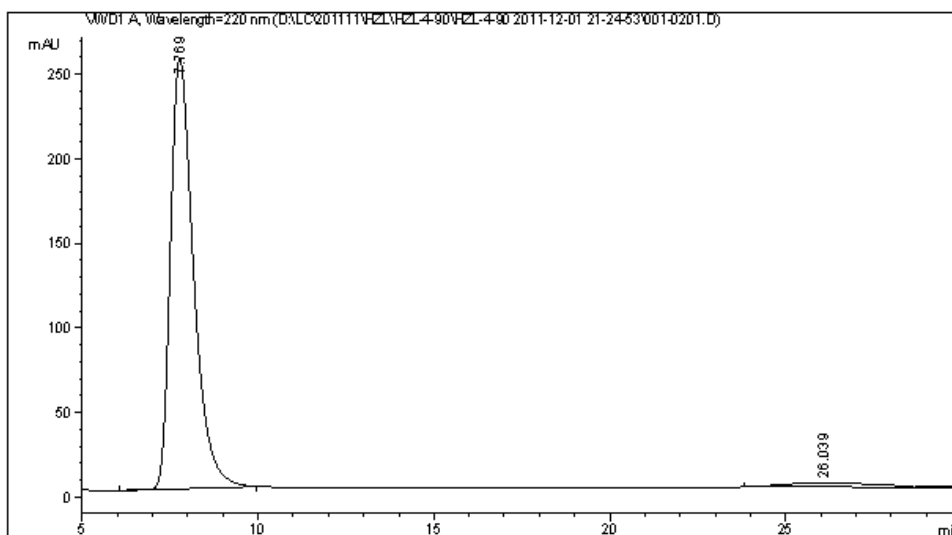
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.788	VB	0.6386	2180.77393	51.76181	50.5390
2	25.448	MM	3.1224	2134.25659	11.39201	49.4610

Totals : 4315.03052 63.15382



Data File D:\LC\201111\H2L\H2L-4-90\H2L-4-90 2011-12-01 21-24-53\001-0201.D
Sample Name: H2L-4-90

```
=====
Acq. Operator   : H2L                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : Vial 1
Injection Date  : 12/1/2011 9:37:28 PM      Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\201111\H2L\H2L-4-90\H2L-4-90 2011-12-01 21-24-53\ASH-30-70-10ML-
                  220NM-35MIN.M
Last changed    : 11/12/2011 3:56:16 PM by LTL
Analysis Method : D:\LC\201111\H2L\H2L-4-90\H2L-4-90 2011-12-01 21-24-53\001-0201.D\DA.M (
                  ASH-30-70-10ML-220NM-35MIN.M)
Last changed    : 12/15/2011 10:29:53 AM by FX
                  (modified after loading)
Method Info     : ASH-50-50-1ML-254NM-50MIN
=====
```



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

```
Sorted By      :      Signal
Multiplier     :      1.0000
Dilution       :      1.0000
Use Multiplier & Dilution Factor with ISTDs
```

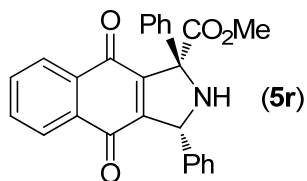
Signal 1: VMD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.769	VB	0.7003	1.16301e4	254.63065	96.2474
2	26.039	BBA	2.1365	453.44806	2.48461	3.7526

```
Totals :                      1.20835e4  257.11526
```

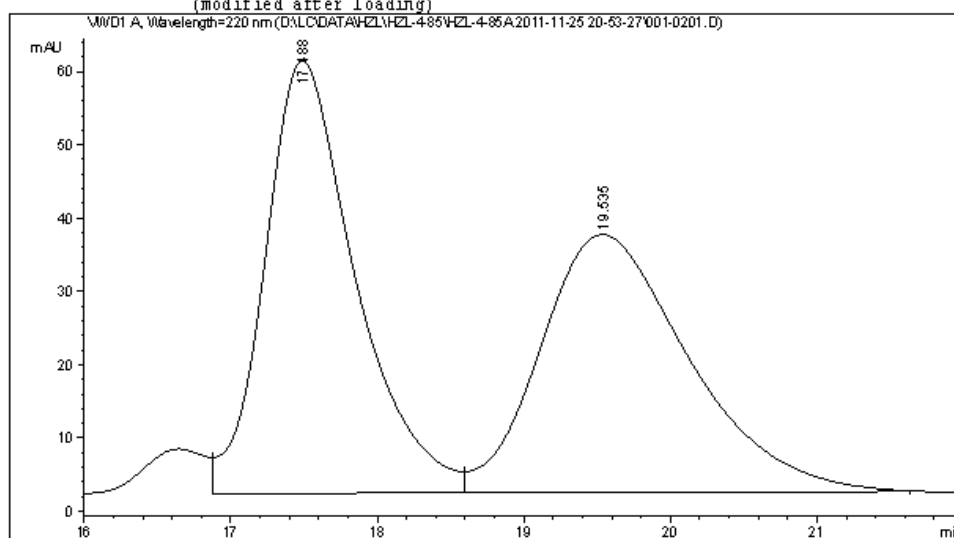
Instrument 1 12/15/2011 10:30:02 AM FX

Page 1 of 2



Data File D:\LC\DATA\H2L\H2L-4-85\H2L-4-85A 2011-11-25 20-53-27\001-0201.D
 Sample Name: H2L-4-85A

```
=====
Acq. Operator   : H2L                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : Vial 1
Injection Date  : 11/25/2011 9:05:19 PM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\201111\H2L\H2L-4-85\H2L-4-85A 2011-11-25 20-53-27\ADH-30-70-10ML-
                  220NM.M
Last changed    : 9/15/2011 8:42:49 AM by THL
Analysis Method : D:\LC\DATA\H2L\H2L-4-85\H2L-4-85A 2011-11-25 20-53-27\001-0201.D\DA.M (
                  ADH-30-70-10ML-220NM.M)
Last changed    : 9/24/2012 10:15:51 AM by FX
                  (modified after loading)
=====
```



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

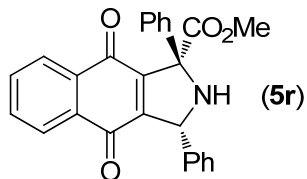
Sorted By : Signal
 Multiplier : 1.0000
 Dilution : 1.0000
 Use Multiplier & Dilution Factor with ISTDs

Signal 1: VMD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	17.488	WV	0.6332	2528.17090	59.08281	50.5948	
2	19.535	VB	1.0471	2468.73096	35.18646	49.4052	
Totals :				4996.90186	94.26928		

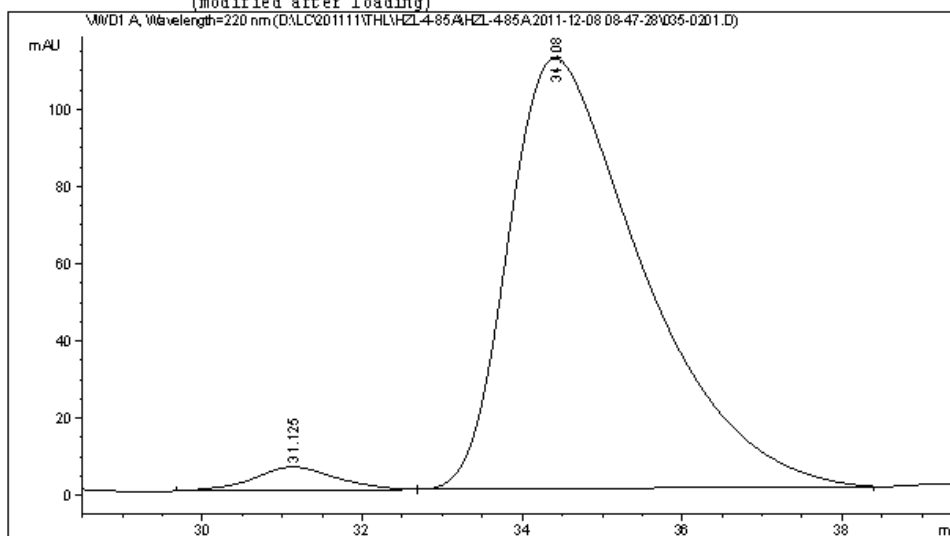
Instrument 1 9/24/2012 10:15:59 AM FX

Page 1 of 1



Data File D:\LC\201111\THL\H2L-4-85A\H2L-4-85A 2011-12-08 08-47-28\035-0201.D
 Sample Name: H2L-4-103A

```
=====
Acq. Operator   : HZL                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : Vial 35
Injection Date  : 12/8/2011 9:50:21 AM      Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\201111\thl\H2L-4-85A\H2L-4-85A 2011-12-08 08-47-28\ADH-15-85-10ML-
                                           220NM-60MIN.M
Last changed    : 12/7/2011 9:20:12 PM by TMC
Analysis Method : D:\LC\201111\THL\H2L-4-85A\H2L-4-85A 2011-12-08 08-47-28\035-0201.D\DA.M (
                                           ADH-15-85-10ML-220NM-60MIN.M)
Last changed    : 12/15/2011 9:55:50 AM by FX
                                           (modified after loading)
=====
```



Area Percent Report

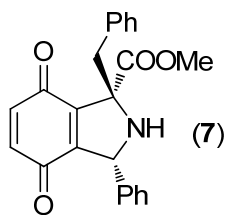
```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]
1	31.125	BV	0.9243	440.21582	3.2697	6.08338
2	34.408	VB	1.7225	1.30233e4	96.7303	111.48381
Totals :				1.34635e4		117.56719

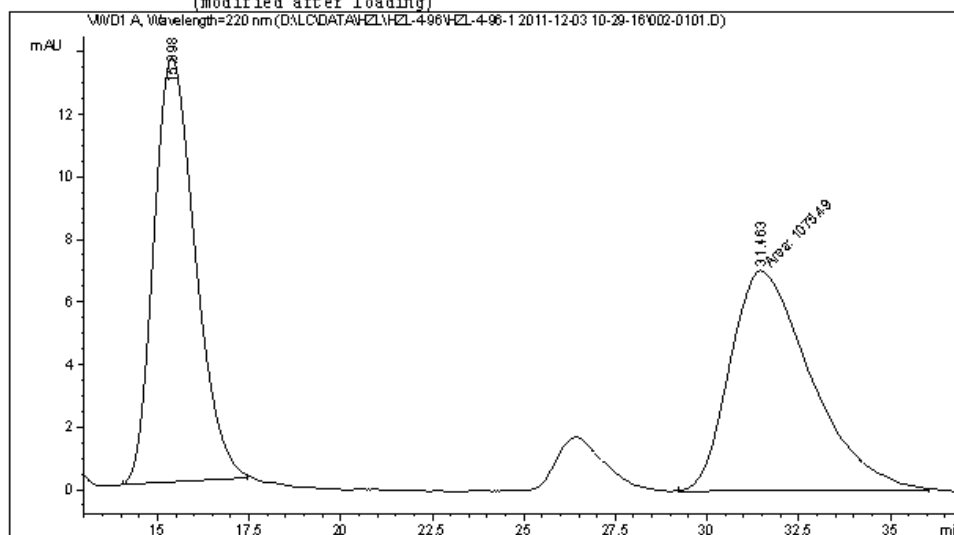
Instrument 1 12/15/2011 9:56:00 AM FX

Page 1 of 1



Data File D:\LC\DATA\H2L\H2L-4-96\H2L-4-96-1 2011-12-03 10-29-16\002-0101.D
 Sample Name: H2L-4-96-1

```
=====
Acq. Operator   : H2L                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 2
Injection Date  : 12/3/2011 10:30:18 AM     Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\201112\H2L\H2L-4-96\H2L-4-96-1 2011-12-03 10-29-16\ASH-20-80-10ML-
220NM.M
Last changed    : 8/29/2011 3:56:33 PM by H2L
Analysis Method : D:\LC\DATA\H2L\H2L-4-96\H2L-4-96-1 2011-12-03 10-29-16\002-0101.D\DA.M (
ASH-20-80-10ML-220NM.M)
Last changed    : 8/14/2012 5:40:15 PM by THL
(modified after loading)
=====
```



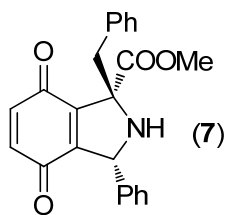
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VMD1 A, Wavelength=220 nm

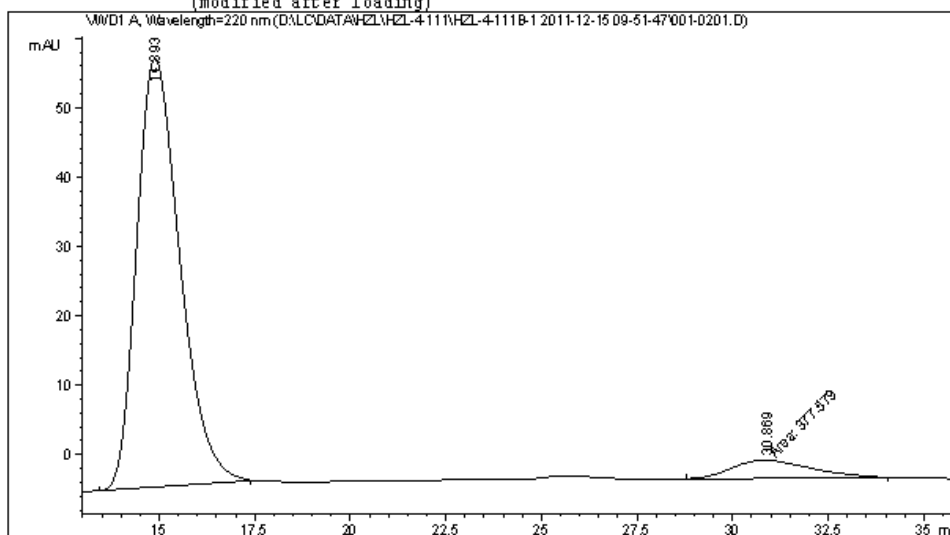
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	15.398	BB	1.2078	1065.41504	13.53992	49.7646
2	31.463	MM	2.5478	1075.49463	7.03542	50.2354

Totals : 2140.90967 20.57534



Data File D:\LC\DATA\HZL\HZL-4-111\HZL-4-111B-1 2011-12-15 09-51-47\001-0201.D
Sample Name: HZL-4-111B-1

```
=====
Acq. Operator   : hzl                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : Vial 1
Injection Date  : 12/15/2011 10:03:57 AM    Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\201112\HZL\HZL-4-111\HZL-4-111B-1 2011-12-15 09-51-47\ASH-20-80-
                  10ML-220NM-40MIN.M
Last changed    : 8/29/2011 6:08:43 PM by HZL
Analysis Method : D:\LC\DATA\HZL\HZL-4-111\HZL-4-111B-1 2011-12-15 09-51-47\001-0201.D\DA.M
                  (ASH-20-80-10ML-220NM-40MIN.M)
Last changed    : 8/14/2012 5:42:05 PM by THL
                  (modified after loading)
=====
```



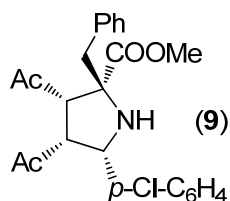
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VWD1 A, Wavelength=220 nm

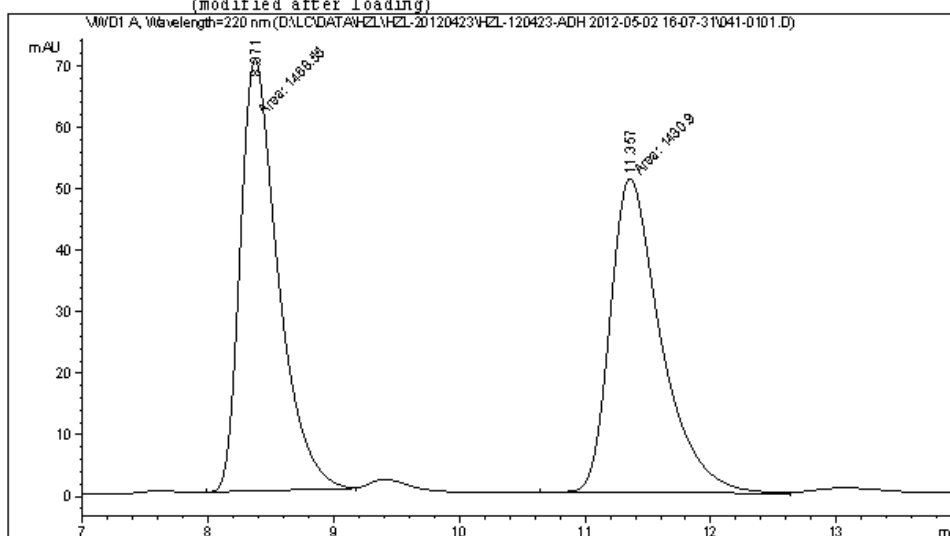
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.893	BB	1.2117	4852.64600	61.79405	92.7808
2	30.869	MM	2.4113	377.57892	2.60980	7.2192

Totals : 5230.22491 64.40385



Data File D:\LC\DATA\H2L\H2L-20120423\H2L-120423-ADH 2012-05-02 16-07-31\041-0101.D
 Sample Name: h2l-5-120423

```
=====
Acq. Operator   : h2l                      Seq. Line :    1
Acq. Instrument : Instrument 1              Location  : Vial 41
Injection Date  : 5/2/2012 4:09:01 PM      Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\DATA\H2L\H2L-20120423\H2L-120423-ADH 2012-05-02 16-07-31\ADH-20-80-
                  IML-220NM.M
Last changed    : 5/2/2012 3:53:21 PM by h2l
Analysis Method : D:\LC\DATA\H2L\H2L-20120423\H2L-120423-ADH 2012-05-02 16-07-31\041-0101.D\
                  DA.M (ADH-20-80-IML-220NM.M)
Last changed    : 5/16/2012 7:38:12 PM by FX
                  (modified after loading)
=====
```



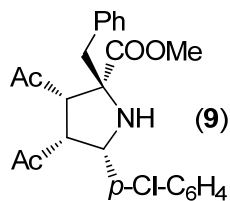
Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution       : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: WVD1 A, Wavelength=220 nm

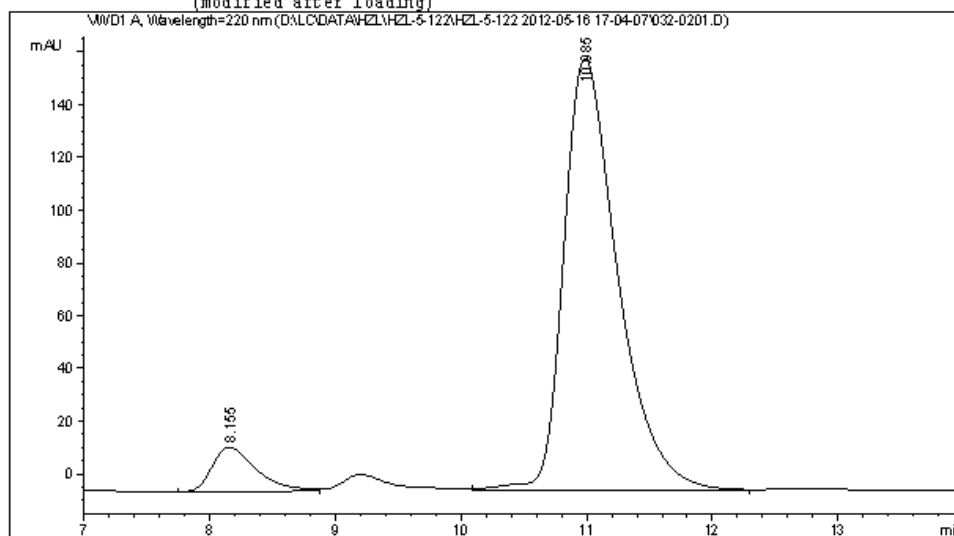
Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area %	Height [mAU]
1	8.371	MM	0.3496	1468.54810	50.6492	70.00198
2	11.357	MM	0.4667	1430.90125	49.3508	51.10359

Totals : 2899.44934 121.10557



Data File D:\LC\DATA\H2L\H2L-5-122\H2L-5-122 2012-05-16 17-04-07\032-0201.D
 Sample Name: H2L-5-122

```
=====
Acq. Operator   : H2L                      Seq. Line :    2
Acq. Instrument : Instrument 1              Location  : Vial 32
Injection Date  : 5/16/2012 5:16:41 PM      Inj       :    1
                                           Inj Volume: 5 µl
Acq. Method     : D:\LC\DATA\H2L\H2L-5-122\H2L-5-122 2012-05-16 17-04-07\ADH-20-80-1ML-
                  220NM-20MIN.M
Last changed    : 5/16/2012 5:03:19 PM by H2L
Analysis Method : D:\LC\DATA\H2L\H2L-5-122\H2L-5-122 2012-05-16 17-04-07\032-0201.D\DA.M (
                  ADH-20-80-1ML-220NM-20MIN.M)
Last changed    : 5/16/2012 7:36:53 PM by FX
                  (modified after loading)
=====
```



Area Percent Report

```
Sorted By      : Signal
Multiplier     : 1.0000
Dilution      : 1.0000
Use Multiplier & Dilution Factor with ISTDs
```

Signal 1: VMD1 A, Wavelength=220 nm

Peak #	RetTime [min]	Type	Width [min]	Area mAU	Area *s	Height [mAU]	Area %
1	8.155	BV	0.3757	410.32269	16.68796	7.7467	
2	10.985	BB	0.4499	4886.43262	163.83717	92.2533	

Totals : 5296.75531 180.52513