Supporting Information for

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

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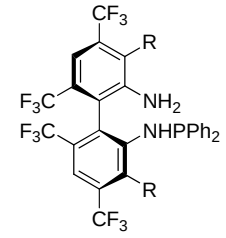
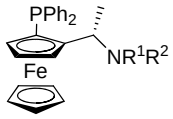
I. General Remarks.

^{1}H 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 ^{1}H 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 $Cu(CN)_4BF_4/PPh_3$ as the catalyst. The absolute (1*R*,3*R*)-**5b** achieved by $Cu(CN)_4BF_4$/(S,R_p)-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]

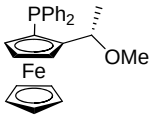
II. Ligand Screening for One Pot and Sequential Catalytic Asymmetric 1,3-DC/Aromatization Reaction



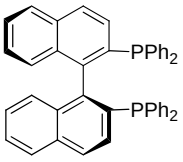
(*S*)-TF-BiphamPhos
L1: R = H; **L2:** R = Br



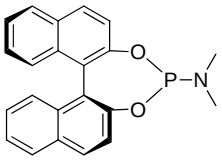
(S,R_p)-**L3**: R^1 = Me, R^2 = Me
(S,R_p)-**L4**: R^1 = H, R^2 = Me
(S,R_p)-**L5**: R^1 = H, R^2 = H



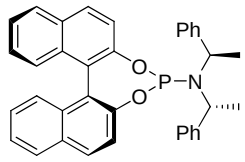
(S,R_p)-**L6**



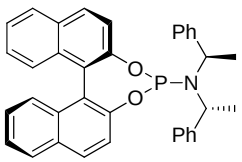
(*S*)-BINAP **L7**



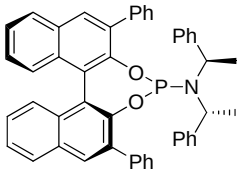
(*S*)-Monophos **L8**



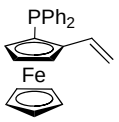
(*S*,*R*,*R*)-**L9**



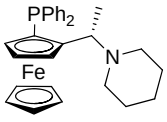
(*R*,*R*,*R*)-**L10**



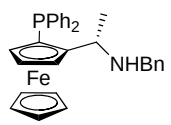
(*S*,*R*,*R*)-**L11**



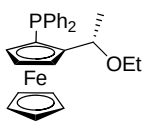
(R_p)-**L12**



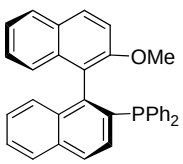
(S,R_p)-**L13**



(S,R_p)-**L14**

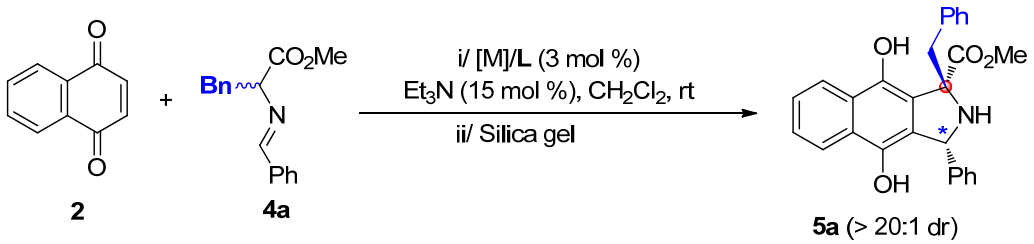


(S,R_p)-**L15**



(*S*)-MOP **L16**

Scheme 1. Screened chiral ligands.



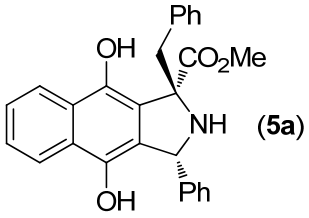
entry	L	[M]	time/h	yield (%)[b]	ee (%)[c]
1	**L1**	AgOAc	6	81	20
2	**L1**	$Cu(CH_3CN)_4BF_4$	6	87	9
3	**L2**	AgOAc	6	86	27
4	**L2**	$Cu(CH_3CN)_4BF_4$	6	85	37
5	**L3**	AgOAc	4	76	9
6	**L3**	$Cu(CH_3CN)_4BF_4$	4	85	71
7	**L4**	$Cu(CH_3CN)_4BF_4$	4	69	69
8	**L5**	$Cu(CH_3CN)_4BF_4$	4	74	22
9	**L6**	$Cu(CH_3CN)_4BF_4$	4	85	78
10	**L7**	AgOAc	12	31	2
11	**L7**	$Cu(CH_3CN)_4BF_4$	12	34	4
12	**L8**	AgOAc	4	65	7
13	**L8**	$Cu(CH_3CN)_4BF_4$	4	50	13
14	**L9**	$Cu(CH_3CN)_4BF_4$	4	70	66
15	**L10**	$Cu(CH_3CN)_4BF_4$	4	71	51
16	**L11**	$Cu(CH_3CN)_4BF_4$	4	82	14
17	**L12**	$Cu(CH_3CN)_4BF_4$	4	78	63
18	**L13**	$Cu(CH_3CN)_4BF_4$	4	67	71
19	**L14**	$Cu(CH_3CN)_4BF_4$	4	75	37
20	**L15**	$Cu(CH_3CN)_4BF_4$	4	82	65
21	**L16**	$Cu(CH_3CN)_4BF_4$	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 CH_2Cl_2. [b] Isolated yield. [c] Determined by HPLC analysis.

III. General Procedure for Cu(I)/(S,R_p)-PPFOMe-Catalyzed Asymmetric 1,3-Dipolar Cycloaddition/Aromatization

Under argon atmosphere, (S,R_p)-PPFOMe (3.1 mg, 0.0072 mmol) and $Cu(CH_3CN)_4BF_4$ (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 naphthoquione (0.2 mmol) were added sequentially, after that the mixture was dropped to -20 ^oC, TEA (3 mg, 0.03 mmol) was added. Once starting material was

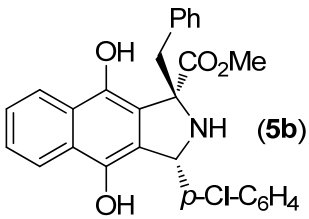
consumed (monitored by TLC), The reaction mixture was treated with silica gel for a short time, then the organic solvent was removed and the residue was purified by column chromatography to give the product, which was then directly analyzed by chiral HPLC to determine the enantiomeric excess.



(**5a**)

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

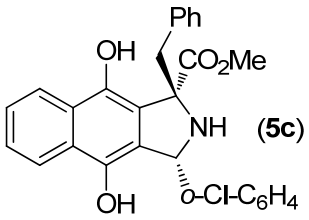
The title compound was prepared according to the general procedure as described above in 86% yield. m.p. 118-121 ^{o}C; $[\alpha]^{25}_{D}$ = +128.2 (*c* 1.36, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 8.16 (d, *J* =7.8 Hz, 1H), 7.88 (d, *J* = 7.8 Hz, 1H), 7.76-7.68 (m, 2H), 7.34-7.15 (m, 10H), 4.88 (s, 1H), 3.85 (s, 3H), 3.63 (d, *J* = 14.1 Hz, 1H), 3.46 (d, *J* = 14.1 Hz, 1H); ^{13}C NMR ($CDCl_3$, 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; ^{13}C 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 $C_{27}H_{22}NO_4^+$: 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.



(**5b**)

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

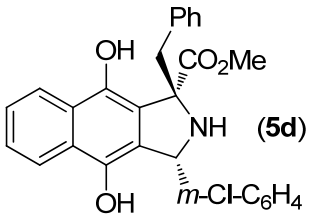
The title compound was prepared according to the general procedure as described above in 89% yield. m.p. 112-115 ^{o}C; $[\alpha]^{25}_{D}$ = +97.4 (*c* 1.60, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 8.16 (d, *J* =7.5 Hz, 1H), 7.88 (d, *J* = 7.5 Hz), 7.79-7.68 (m, 2H), 7.31-7.19 (m, 7H), 7.12 (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); ^{13}C NMR ($CDCl_3$, 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 $C_{27}H_{21}ClNO_4^+$: 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-dihydroxy-2,3-dihydro-1H-benzo[f]isoindole-1-carboxylate

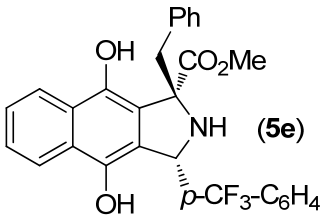
The title compound was prepared according to the general procedure as described above in 87% yield. m.p. 129-132 ^{o}C; $[\alpha]^{25}_{D}$ = +75.4 (*c* 1.54, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 8.18 (d, *J* =7.5 Hz, 1H), 7.92 (d, *J* = 7.5 Hz, 1H), 7.90-7.71 (m, 2H), 7.35-7.32 (m, 1H), 7.24-7.15 (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); ^{13}C NMR ($CDCl_3$, 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; ^{13}C 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 $C_{27}H_{21}ClNO_4^+$: 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.



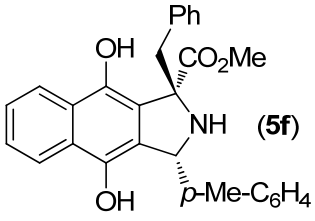
(1*R*,3*R*)-methyl 1-benzyl-3-(3-chlorophenyl)-4,9-dihydroxy-2,3-dihydro-1H-benzo[f]isoindole-1-carboxylate

The title compound was prepared according to the general procedure as described above in 87% yield. m.p. 176-179 ^oC; $[\alpha]^{25}_D$ = +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); ^{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 $C_{27}H_{21}ClNO_4^+$: 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.



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

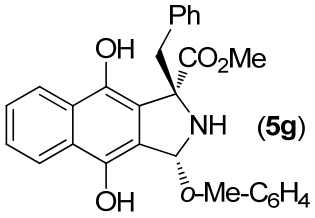
The title compound was prepared according to the general procedure as described above in 87% yield. m.p. 187-190 ^{o}C; $[\alpha]^{25}_{D}$ = +72.6 (*c* 1.84, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 8.17 (d, *J* =7.5 Hz, 1H), 7.88 (d, *J* = 7.5 Hz, 1H), 7.80-7.68 (m, 2H), 7.55-7.47 (m, 4H), 7.22-7.10 (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); ^{13}C NMR ($CDCl_3$, 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 ($J_{C\text{-}F}$ = 32.4 Hz), 128.4, 128.3, 127.3, 126.6, 126.4, 125.3 ($J_{C\text{-}F}$ = 3.7 Hz), 124.0 ($J_{C\text{-}F}$ = 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 $C_{28}H_{21}F_3NO_4^+$: 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.



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

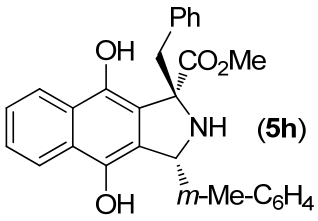
The title compound was prepared according to the general procedure as described above in 93% yield. m.p. 176-178 ^{o}C; $[\alpha]^{25}_{D}$ = +156.3 (*c* 1.62, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 8.15 (d, *J* = 7.8 Hz, 1H), 7.87 (d, *J* = 7.8 Hz, 1H), 7.86-7.67 (m, 2H), 7.21-7.07 (m, 9H), 4.85 (s, 1H), 3.84 (s, 3H), 3.62 (d, *J* = 13.8 Hz,

1H), 3.44 (d, *J* = 13.8 Hz, 1H), 2.29 (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 $C_{28}H_{24}NO_4^+$: 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, λ = 220 nm); t_r = 7.57 and 20.44 min.



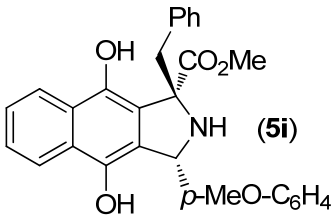
(1*R*,3*R*)-methyl 1-benzyl-4,9-dihydroxy-3-(o-tolyl)-2,3-dihydro-1H-benzo[f] isoindole-1-carboxylate

The title compound was prepared according to the general procedure as described above in 81% yield. m.p. 135-138 ^{o}C; $[\alpha]^{25}_D$ = +107.4 (*c* 1.50, $CHCl_3$); ^{1}H 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), 2.34 (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 $C_{28}H_{24}NO_4^+$: 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, λ = 220 nm); t_r = 6.33 and 15.03 min.



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

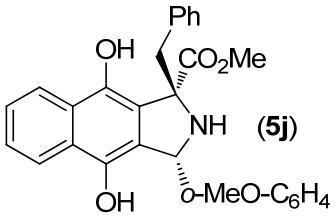
The title compound was prepared according to the general procedure as described above in 86% yield. m.p. 179-182 ^{o}C; $[\alpha]^{25}_{D}$ = +113.5 (*c* 1.48, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 8.16 (d, *J* = 7.2 Hz, 1H), 7.88 (d, *J* = 7.2 Hz, 1H), 7.76-7.69 (m, 2H), 7.19-7.06 (m, 9H), 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); ^{13}C NMR ($CDCl_3$, 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 $C_{28}H_{24}NO_4^+$: 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.



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

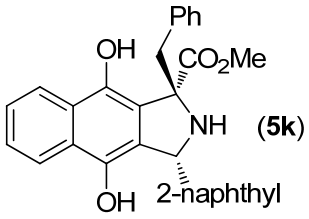
The title compound was prepared according to the general procedure as described above in 81% yield. m.p. 143-145 ^{o}C; $[\alpha]^{25}_{D}$ = +215.9 (*c* 1.44, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 8.16 (d, *J* =7.8 Hz, 1H), 7.88 (d, *J* = 7.8 Hz, 1H), 7.78-7.66 (m, 2H), 7.23-7.15 (m, 7H), 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); ^{13}C NMR

($CDCl_3$, 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 $C_{28}H_{24}NO_5^+$: 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.



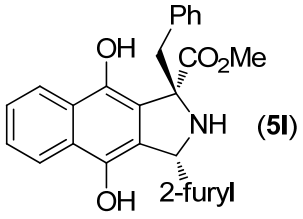
(1*R*,3*R*)-methyl 1-benzyl-4,9-dihydroxy-3-(2-methoxyphenyl)-2,3-dihydro-1H-benzo[f]isoindole-1-carboxylate

The title compound was prepared according to the general procedure as described above in 85% yield. m.p. 149-152 ^oC; $[\alpha]^{25}_D$ = +49.3 (*c* 0.93, $CHCl_3$); ^1H NMR ($CDCl_3$, 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); ^{13}C NMR ($CDCl_3$, 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 $C_{28}H_{25}NO_5 + H^+$: 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-4,9-dihydroxy-3-(naphthalen-2-yl)-2,3-dihydro-1H-benzo[f]isoindole-1-carboxylate

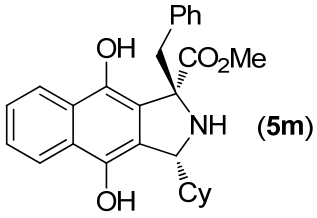
The title compound was prepared according to the general procedure as described above in 79% yield. m.p. 198-201 ^{o}C; $[\alpha]^{25}_{D}$ = +43.1 (*c* 1.46, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 8.17 (d, *J* =7.5 Hz, 1H), 7.84 (d, *J* = 7.5 Hz, 1H), 7.77-7.67 (m, 5H), 7.47-7.42 (m, 3H), 7.22-7.17 (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); ^{13}C NMR ($CDCl_3$, 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 $C_{31}H_{24}NO_4^+$: 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-dihydroxy-2,3-dihydro-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 ^{o}C; ^{1}H NMR ($CDCl_3$, 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

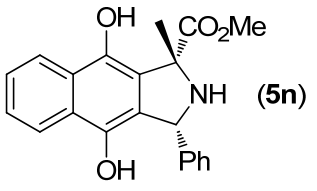
Hz, 1H), 2.79 (br, 1H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 182.2, 181.6, 172.8, 152.9, 148.3, 146.7, 142.2, 135.5, 133.9, 132.6, 130.5, 128.1, 127.0, 126.6, 126.3, 110.6, 107.5, 75.7, 59.4, 42.0; IR (KBr) ν 3374, 2926, 1736, 1637, 1594, 1436, 1337, 1293, 1270, 1148, 1046, 770, 732, 702 cm^{-1}. HRMS: calcd. for $C_{25}H_{20}NO_5^+$: 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.



(**5m**)

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

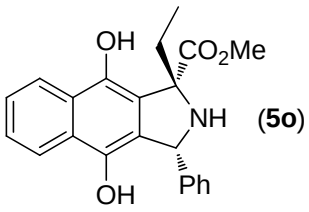
The title compound was prepared according to the general procedure as described above in 70% yield. m.p. 138-141 ^{o}C; $[\alpha]^{25}_{D}$ = +83.7 (*c* 1.28, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 8.14 (d, *J* = 7.2 Hz, 1H), 8.02 (d, *J* = 7.2 Hz, 1H), 7.77-7.73 (m, 2H), 7.14-7.13 (m, 3H), 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 $C_{27}H_{28}NO_4^+$: 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.



(**5n**)

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

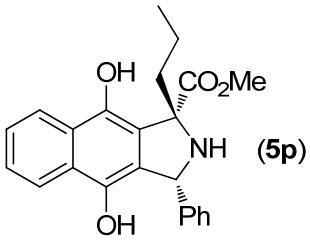
The title compound was prepared according to the general procedure as described above in 82% yield. m.p. 95-98 °C; $[\alpha]^{25}_{D}$ = +31.5 (*c* 1.20, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 8.11 (d, *J* = 8.4 Hz, 1H), 7.97 (d, *J* = 8.4 Hz, 1H), 7.74-7.70 (m, 2H), 7.44 (d, *J* = 6.9 Hz, 1H), 7.37-7.27 (m, 3H), 5.69 (s, 1H), 3.79 (s, 3H), 2.60 (br, 1H), 1.81 (s, 3H); ^{13}C NMR ($CDCl_3$, 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 $C_{21}H_{18}NO_4^+$: 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.



(**5o**)

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

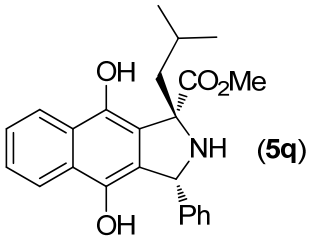
The title compound was prepared according to the general procedure as described above in 90% yield. m.p. 102-105 °C; $[\alpha]^{25}_{D}$ = +5.5 (*c* 1.40, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 8.12 (d, *J* = 7.2 Hz, 1H), 7.97 (d, *J* = 7.2 Hz, 1H), 7.74-7.70 (m, 2H), 7.42-7.30 (m, 5H), 5.64 (s, 1H), 3.79 (s, 3H), 2.35-2.31 (m, 1H), 2.22-2.17 (m, 1H), 0.96-0.91 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 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 $C_{22}H_{20}NO_4^+$: 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.



(**5p**)

(1*R*,3*R*)-methyl 4,9-dihydroxy-3-phenyl-1-propyl-2,3-dihydro-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 ^{o}C; $[\alpha]^{25}_{D}$ = +6.3 (*c* 1.20, $CHCl_3$); ^{1}H NMR ($CDCl_3$, 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); ^{13}C NMR ($CDCl_3$, 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 $C_{23}H_{22}NO_4^+$: 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.

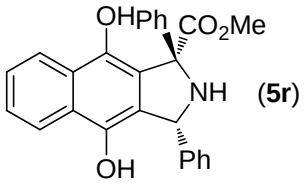


(**5q**)

(1*R*,3*R*)-methyl 4,9-dihydroxy-1-isobutyl-3-phenyl-2,3-dihydro-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 ^{o}C; $[\alpha]^{25}_{D}$ = +26.5 (*c* 1.56, $CHCl_3$); ^{1}H NMR ($CDCl_3$, TMS, 300 MHz) δ 8.12 (d, *J* = 9.0 Hz, 1H), 7.97 (d, *J* = 9.0 Hz, 1H),

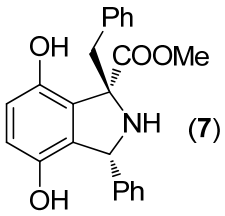
7.77-7.70 (m, 2H), 7.41-7.29 (m, 5H), 5.65 (s, 1H), 3.77 (s, 3H), 2.61 (br, 1H), 2.25-2.11 (m, 2H), 1.78-1.74 (m, 1H), 1.02 (d, J = 6.6 Hz, 3H), 0.86 (d, J = 6.6 Hz, 3H); ^{13}C NMR ($CDCl_3$, TMS, 75 MHz) δ 182.1, 173.5, 149.9, 147.2, 141. 3, 133.8, 133.7, 133.0, 132.8, 128.6, 128.0, 127.6, 126.5, 126.3, 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 $C_{24}H_{24}NO_4^+$: 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.



(**5r**)

(1*R*,3*R*)-methyl 4,9-dihydroxy-1,3-diphenyl-2,3-dihydro-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 ^{o}C; $[\alpha]^{25}_{D}$ = +48.6 (*c* 0.42, $CHCl_3$); ^{1}H 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, 8H), 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 $C_{26}H_{20}NO_4^+$: 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.

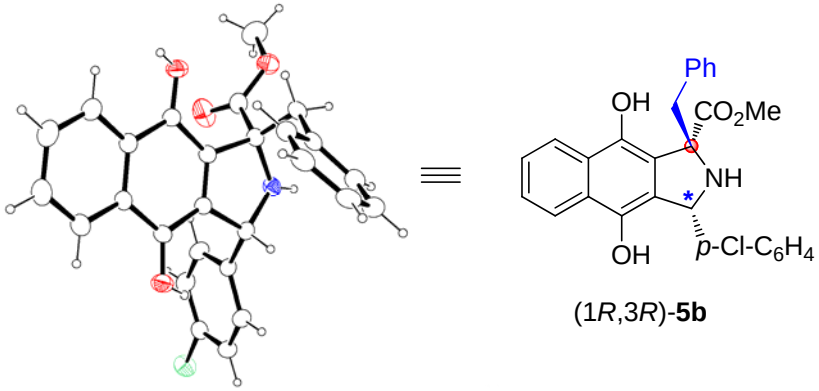


(7)

(1*R*,3*R*)-methyl 1-benzyl-4,7-dihydroxy-3-phenylisoindoline-1-carboxylate

The title compound was prepared according to the general procedure as described above in 82% yield. $[\alpha]^{25}_{D}$ = +53.7 (*c* 0.48, $CHCl_3$); ^{1}H 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 $C_{23}H_{22}NO_4^+$: 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, λ = 220 nm); t_r = 14.89 and 30.87 min.

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



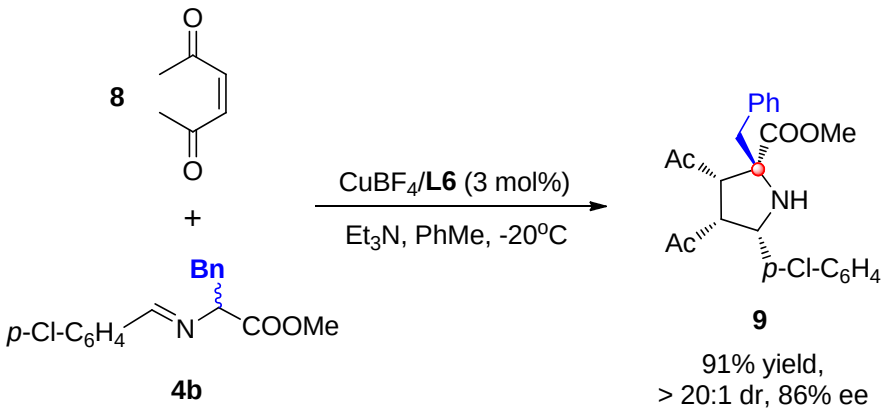
(1*R*,3*R*)-**5b**

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

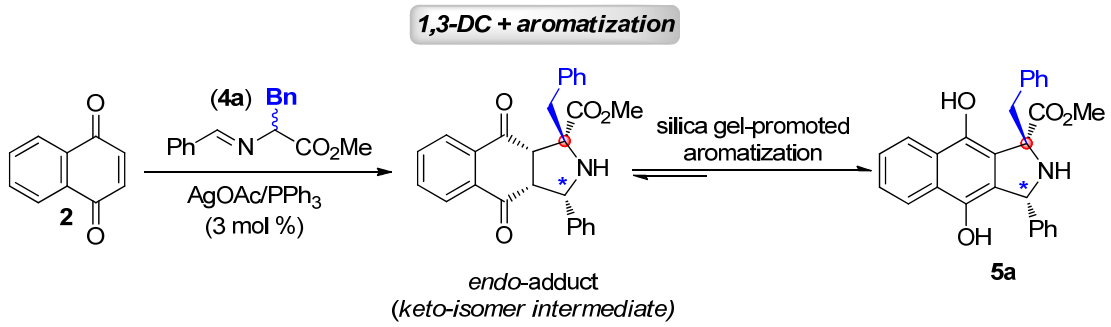
Crystal data for (1*R*,3*R*)-**5b**: $C_{27}H_{22}ClNO_4$, M_r = 459.91, *T* = 293 K, tetragonal, space group *P*4(3), *a* = 12.7827(8), *b* = 12.7827(8), *c* = 13.7419(18) Å, *V* = 2245.4(4) Å^3, *Z* = 4, 3391 unique reflections, final R_1 = 0.0343 and wR_2 = 0.0908 for 4129 observed [$I>2\sigma(I)$] reflections, Flack χ = -0.05(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).

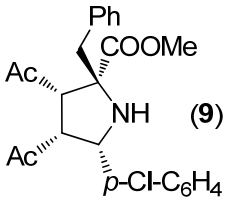
V. Proposed Relative Configuration of the Labile Keto-Isomer Intermidiate in This One-Pot Sequential Catalytic Asymmetric 1,3-DC/Aromatization



To further investigate the relative configuration of the labile keto-isomer intermediate of this one-pot sequential catalytic asymmetric 1,3-DC/Aromatization 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 keto-isomer intermediate in this one-pot sequential 1,3-DC/Aromatization reaction were tentatively proposed to be *endo* on the basis of these results (Scheme 2).



Scheme 2. Proposed relative configuration of the labile keto-isomer intermidiate in this one-pot sequential catalytic asymmetric 1,3-DC/Aromatization.



(2*R*,3S,4*R*,5*S*)-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 $Cu(CH_3CN)_4BF_4$ (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 ^{o}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. ^{1}H NMR ($CDCl_3$, 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); ^{13}C NMR ($CDCl_3$, 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 $C_{23}H_{25}ClNO_4^+$: 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

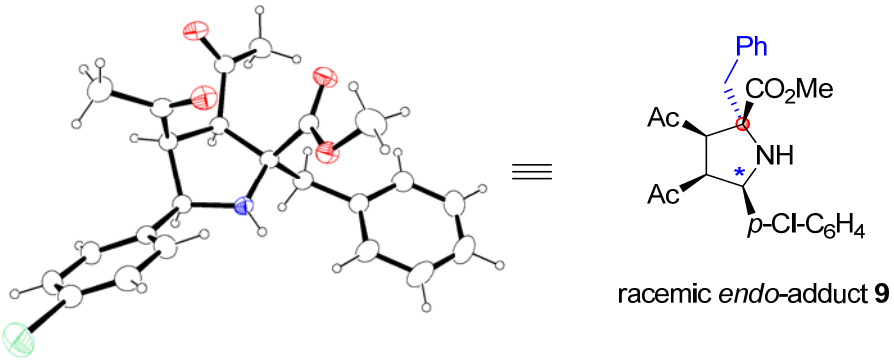


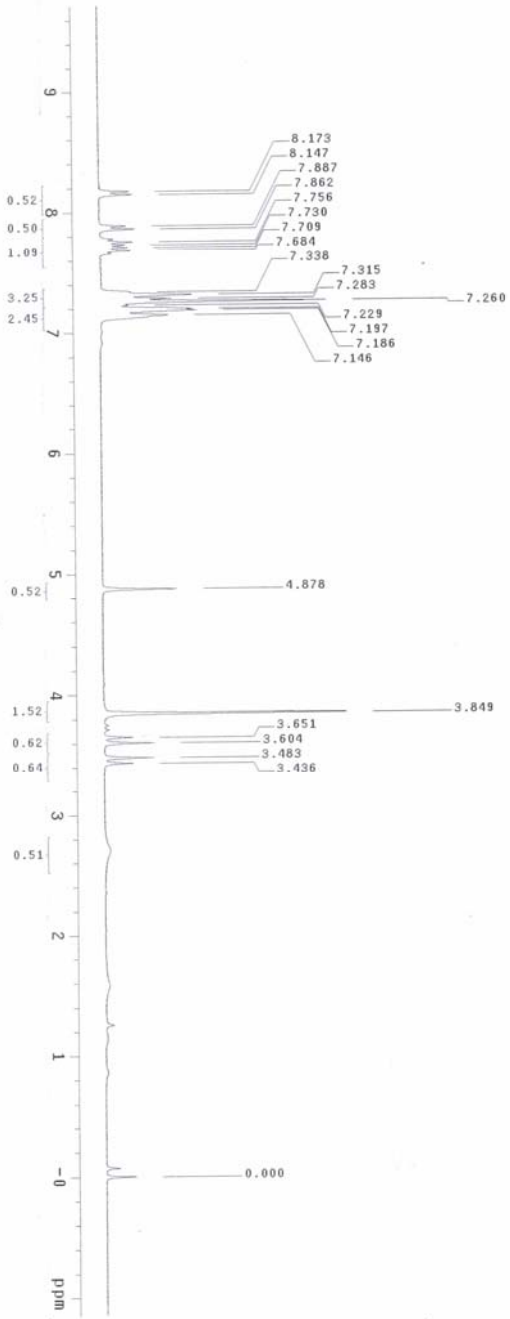
Figure 2. X-ray structure of racemic *endo*-**9**.

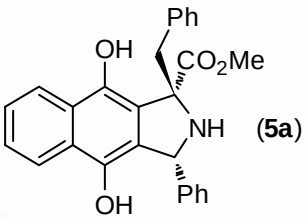
Crystal data for racemic *endo*-adduct **9**: $C_{23}H_{24}ClNO_4$, M_r = 413.88, T = 293 K, Monoclinic, space group $P2(1)/c$, a = 8.9427(14), b = 24.682(4), c = 9.8606(16) Å, V = 2156.1(6) Å^3, Z = 4, 3082 unique reflections, final R_1 = 0.0450 and wR_2 = 0.1077 for 4233 observed [$I>2\sigma(I)$] reflections. 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

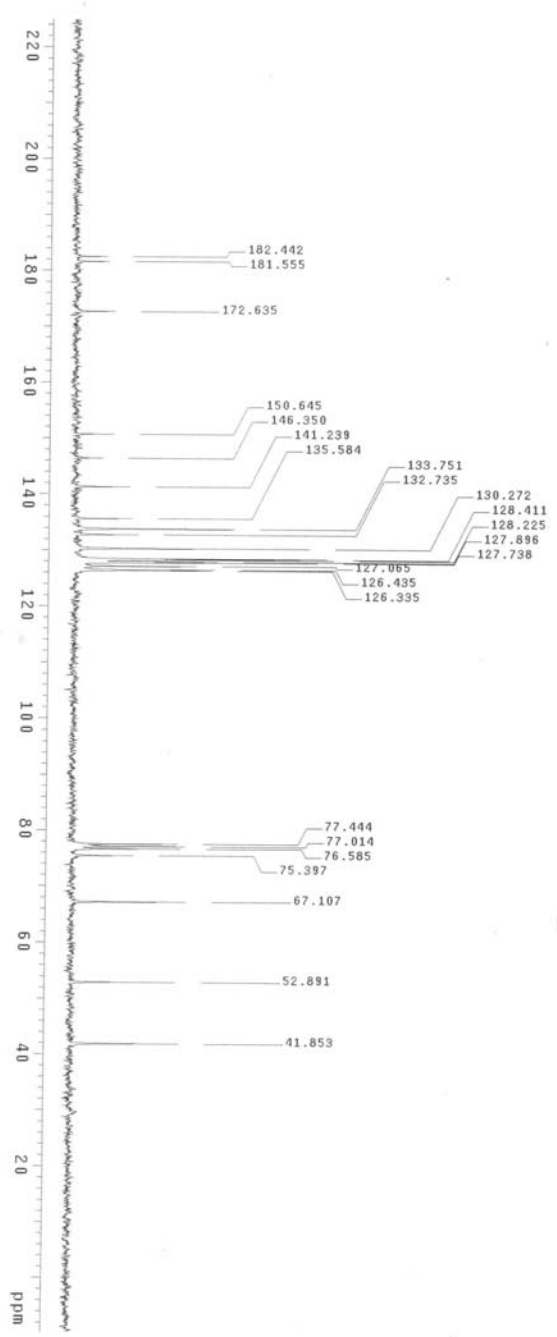
1. Hayashi, T; Fukushima, T, M; Kagotani, M; Nagashima, N; Hamada, Y; Matsumoto, A; Kawakami, S; Konishi, M; Yamamoto, K; Kumada, M. *Bull. Chem. Soc. Jpn.*, **1980**, 53, 1138.
2. CCDC 904693 (**5b**) and CCDC 904694 (**9**) contain the supplementary crystallographic data for this paper. These data 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. ^{1}H NMR and ^{13}C NMR Spectra





(**5a**)



c13hz1-4-29

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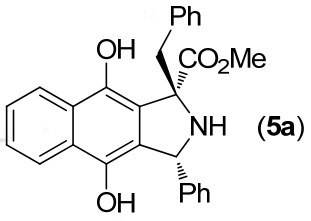
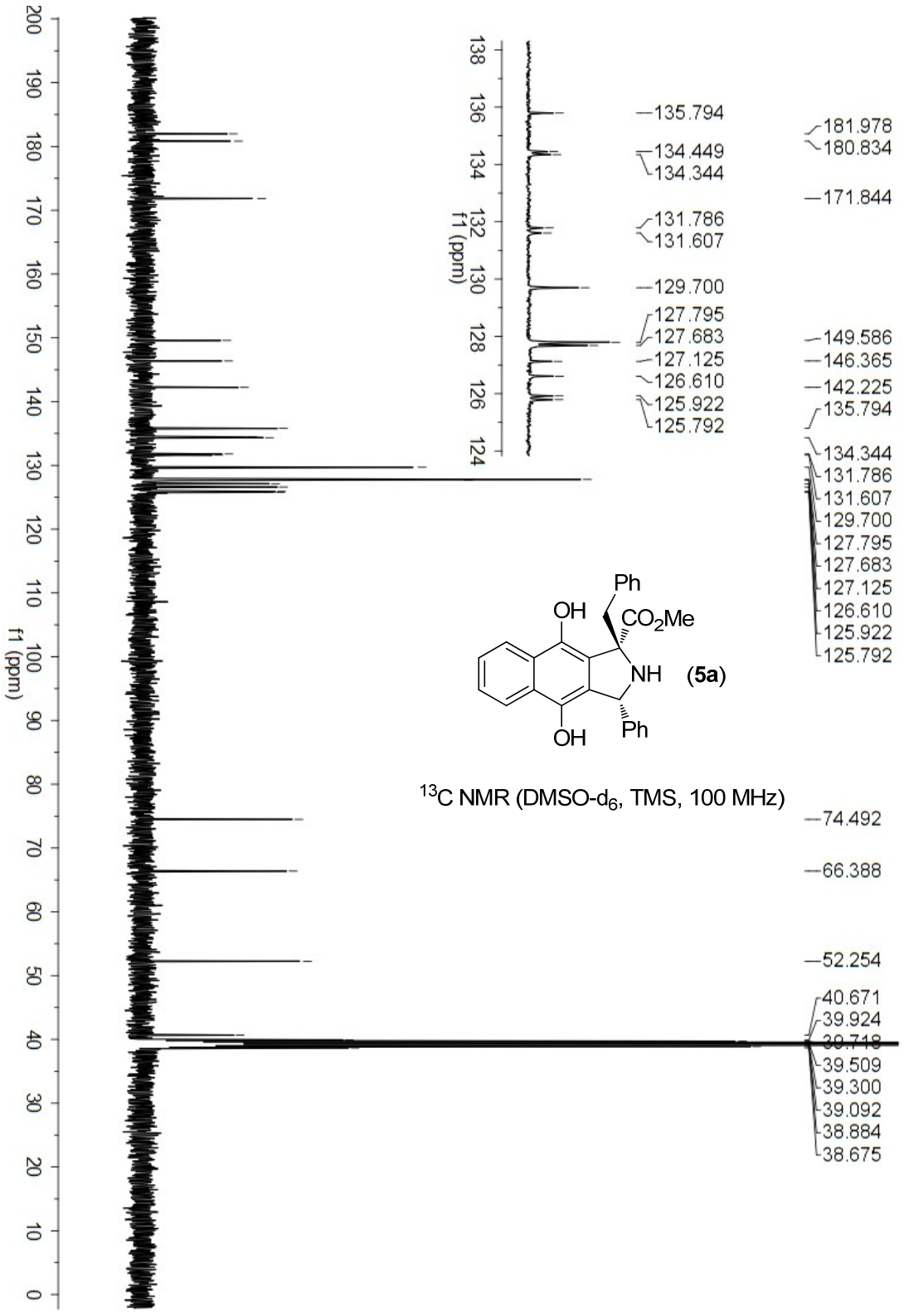
DECOUPLE H1, 300.0815382 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec



(**5a**)

^{13}C NMR ($CDCl_3$, TMS, 75 MHz)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134.344
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131.607
129.700
127.795
127.683
127.125
126.610
125.922
125.792
181.978
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171.844
149.586
146.365
142.225
135.794
134.344
131.786
131.607
129.700
127.795
127.683
127.125
126.610
125.922
125.792
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40.671
39.924
39.718
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39.300
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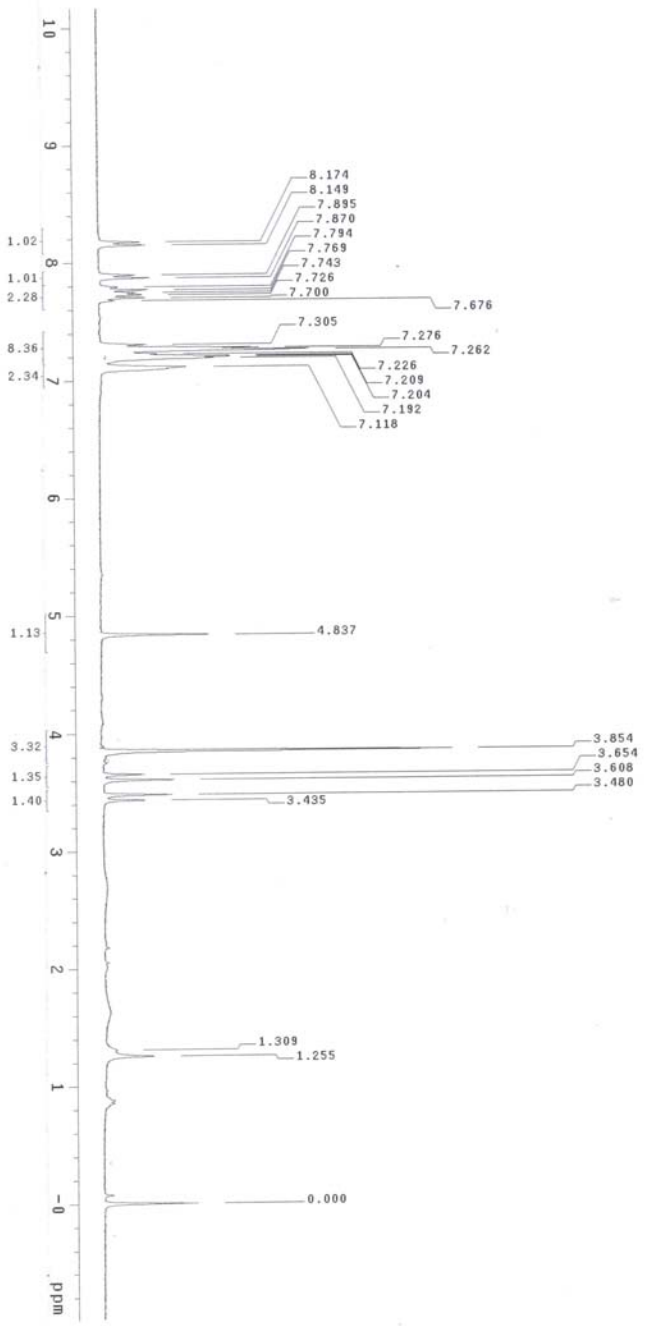
(5a)
OH
Ph
13C NMR (DMSO-d6, TMS, 100 MHz)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7.794
7.769
7.743
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7.305
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7.262
7.226
7.209
7.204
7.192
7.118
4.837
3.854
3.654
3.608
3.480
3.435
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1.01
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1.13
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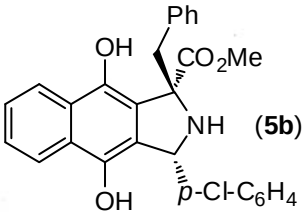
1.35
1.40
ppm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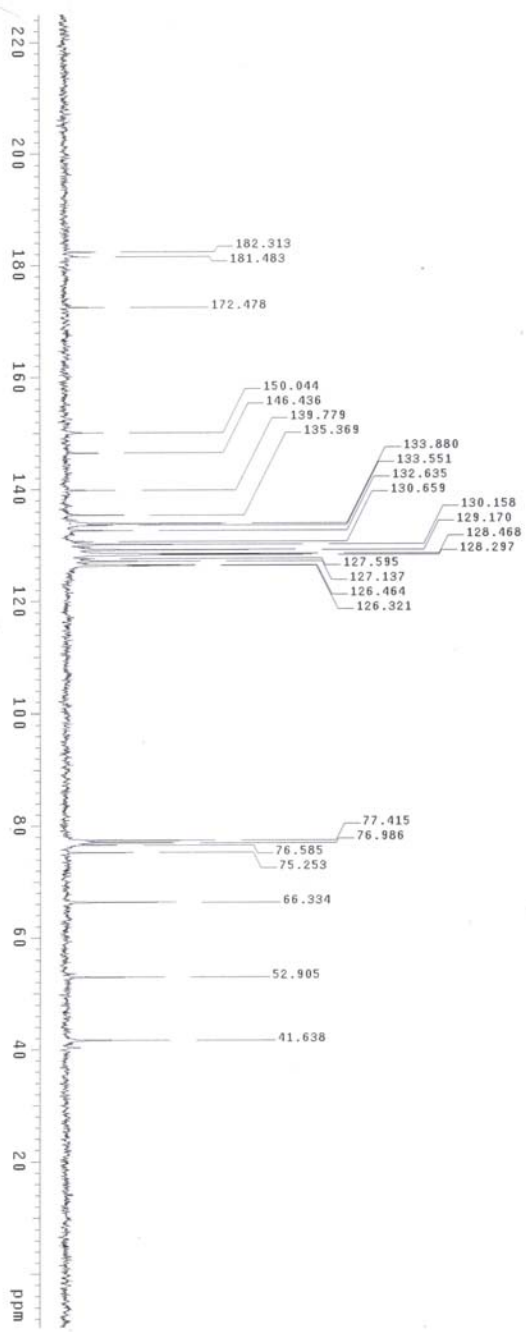
p-Cl-C6H4

(**5b**)



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Ambient temperature
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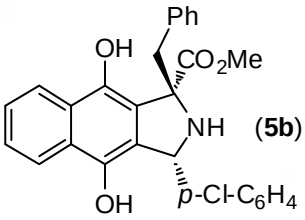
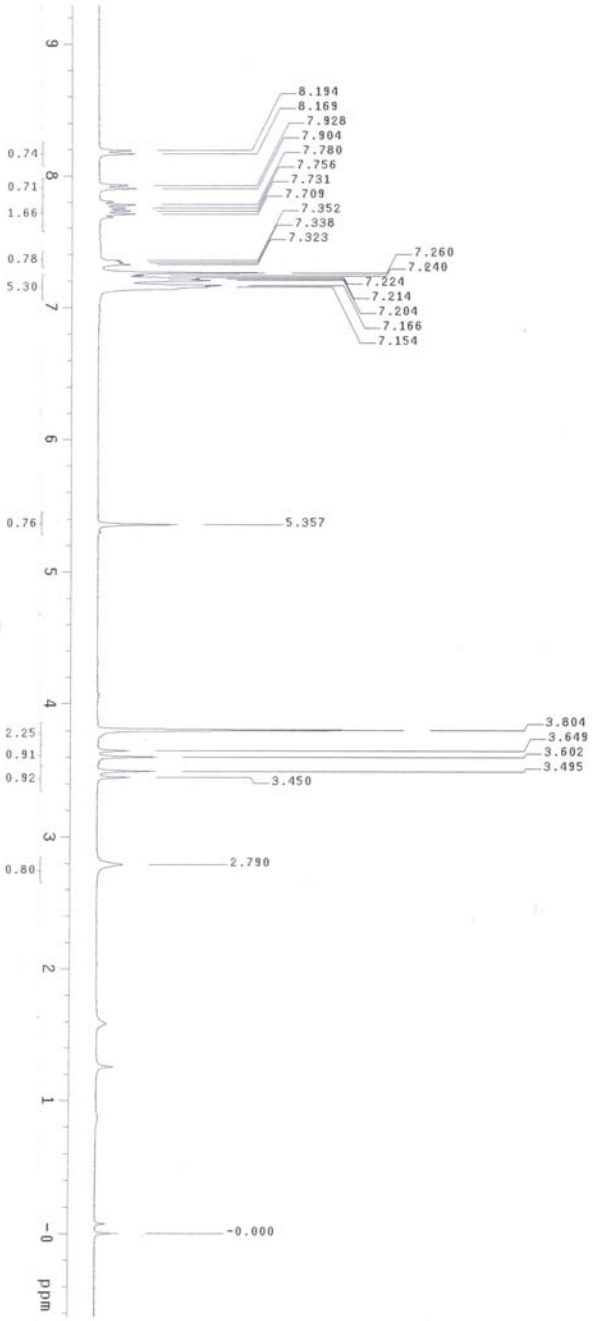
Power 40 dB
continuously on
WALTZ16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec



(**5b**)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7.214
7.204
7.166
7.154
5.357
3.804
3.649
3.602
3.495
3.450
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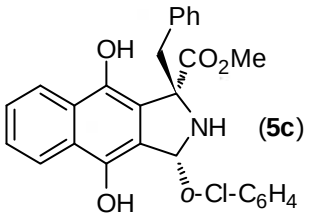
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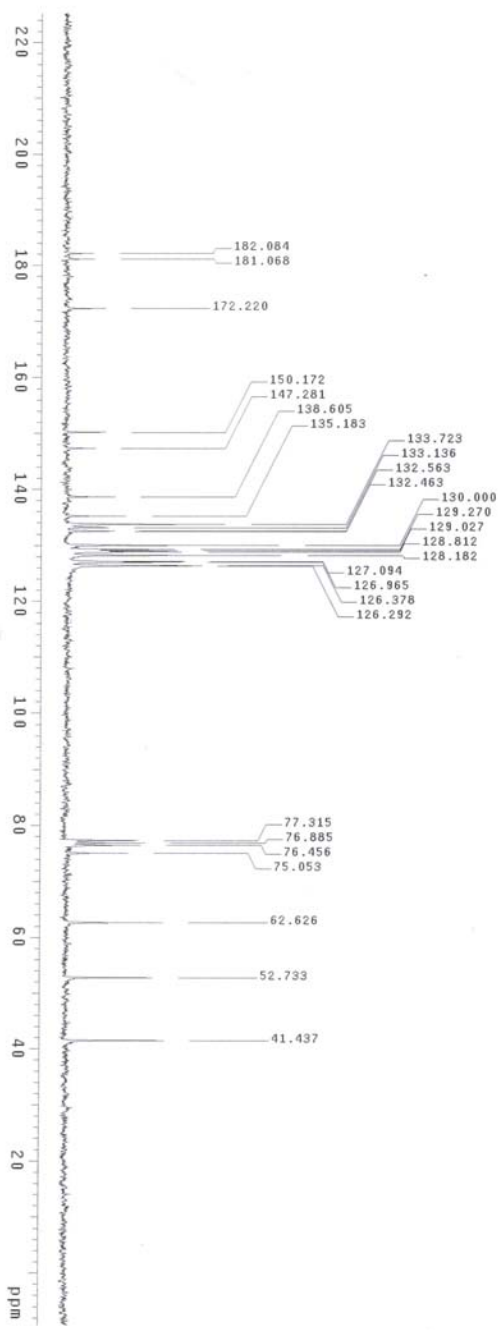
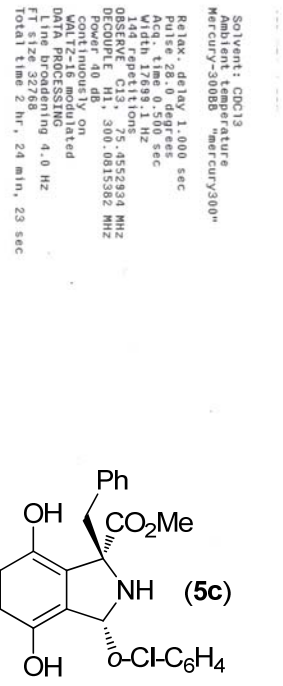
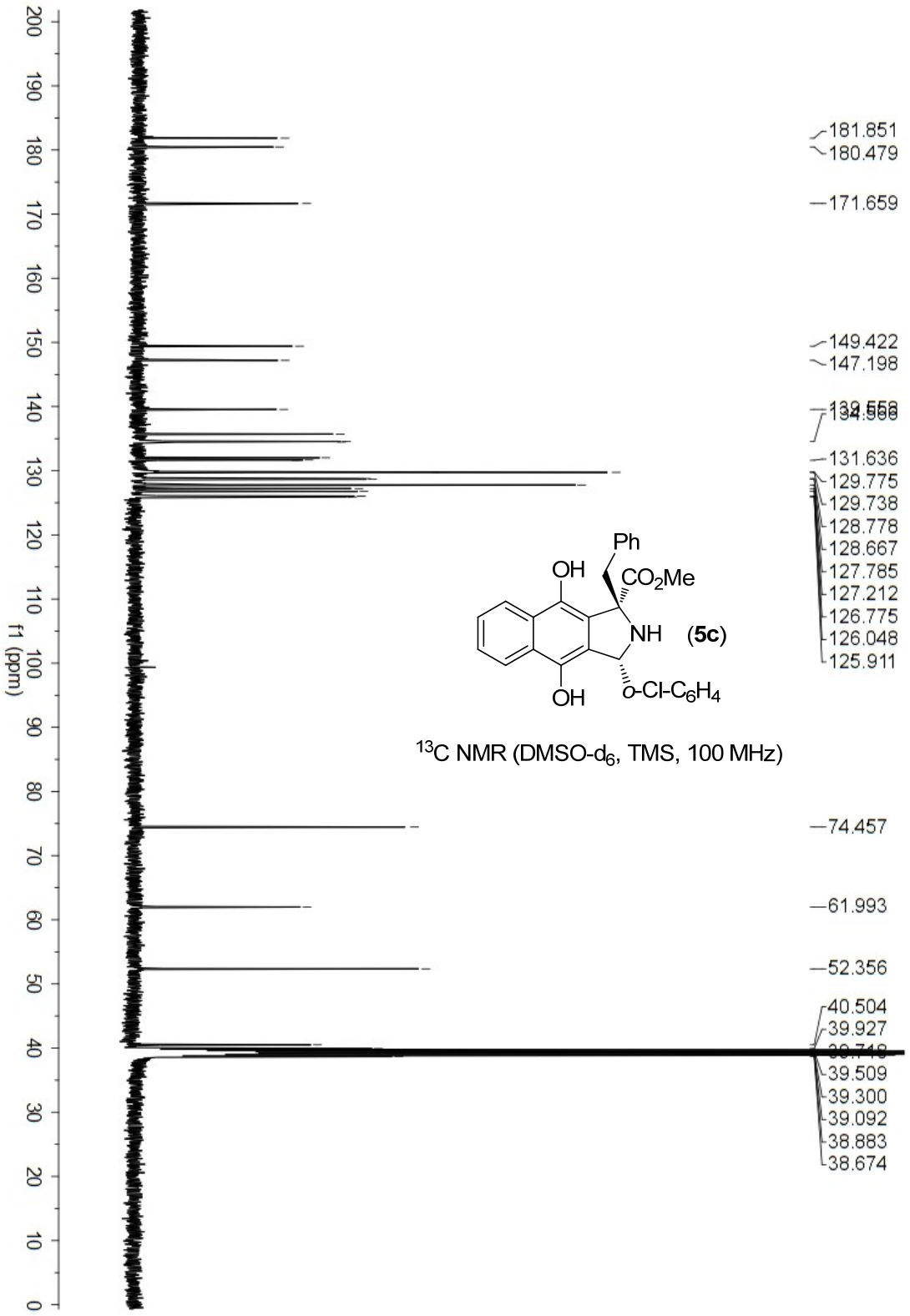
OH
o-Cl-C6H4

(**5c**)





^{13}C NMR ($CDCl_3$, TMS, 75 MHz)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61.993
52.356
40.504
39.927
39.509
39.300
39.092
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Ph
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CO2Me
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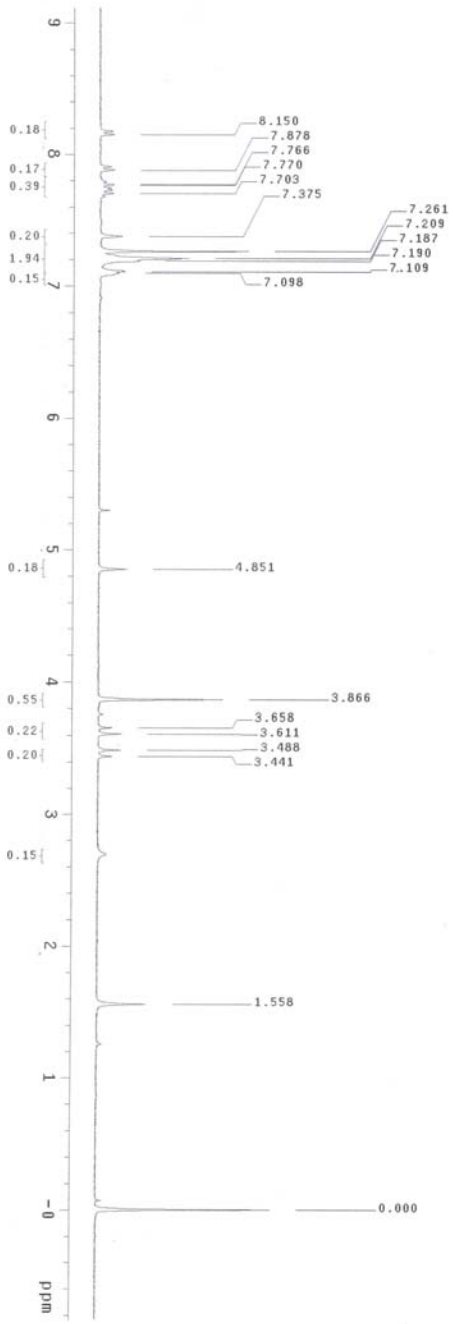
(5c)
OH
o-Cl-C6H4
13C NMR (DMSO-d6, TMS, 100 MHz)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3.866
3.658
3.611
3.488
3.441
1.558
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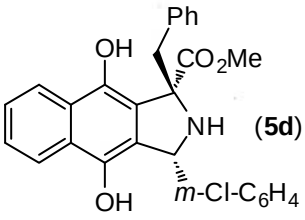
0.15
ppm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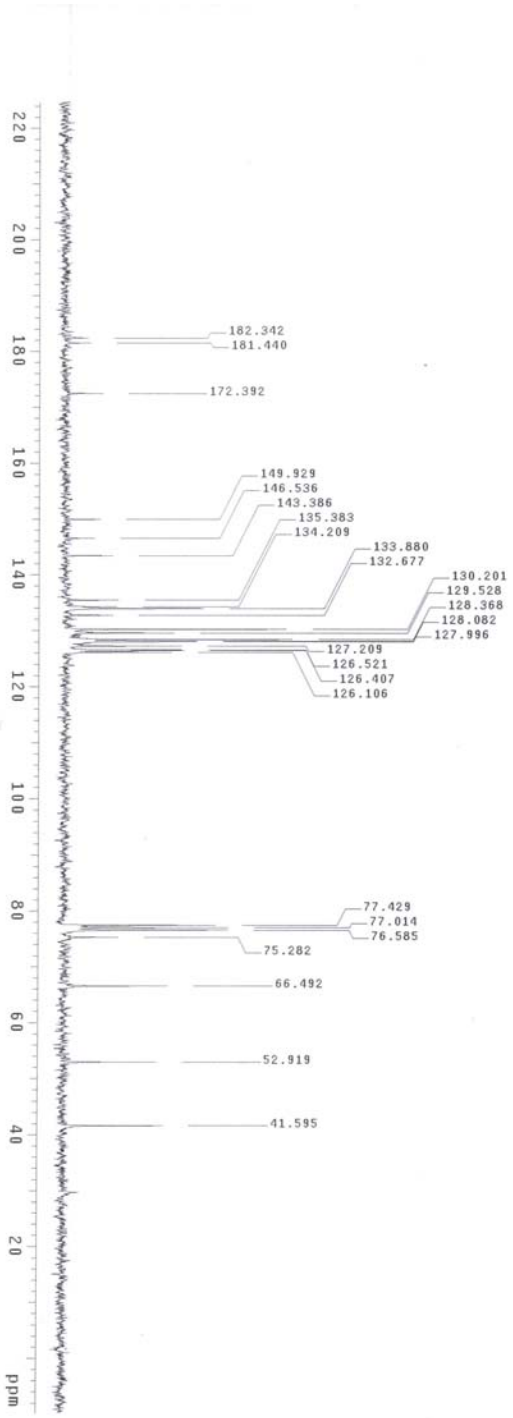
OH
m-Cl-C6H4

(**5d**)



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Ambient temperature
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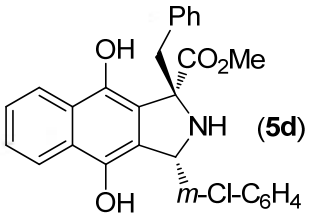
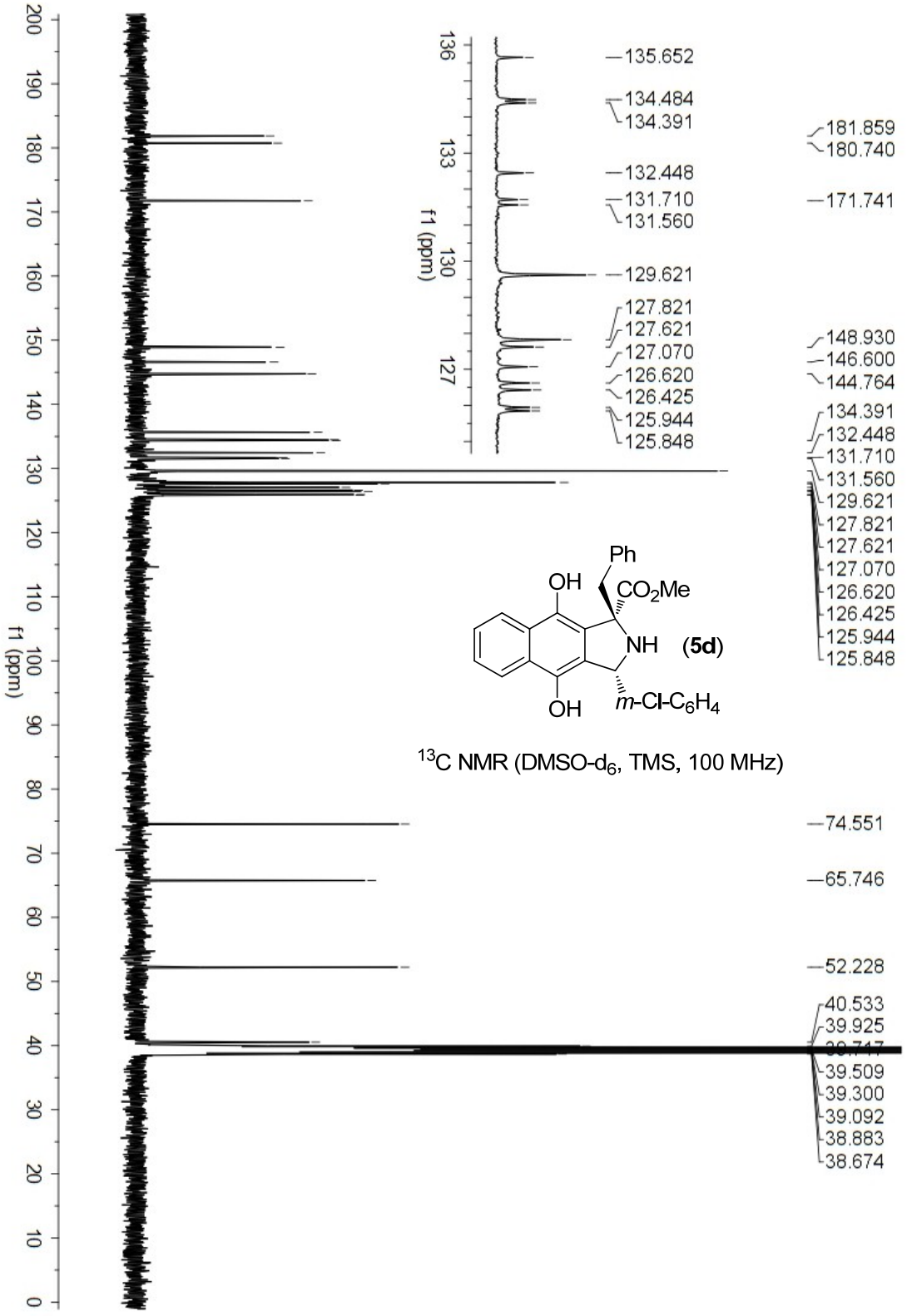
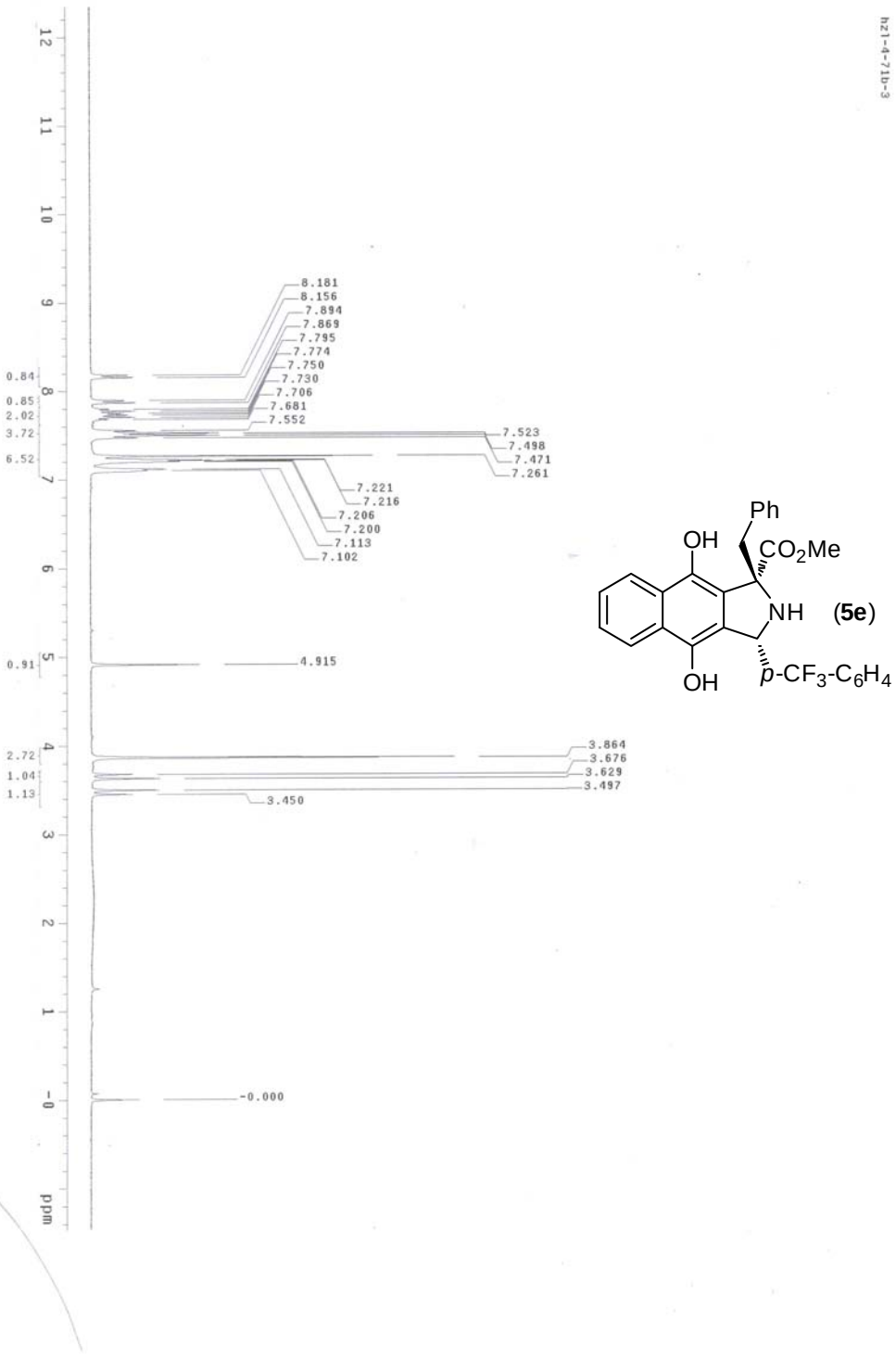
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
208 repetitions
OBSERVE C13, 75.4553247 MHz
DECOUPLE H1, 300.0815382 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec



^{13}C NMR ($CDCl_3$, TMS, 75 MHz)



^{13}C NMR (DMSO-d_6, TMS, 100 MHz)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7.200
7.113
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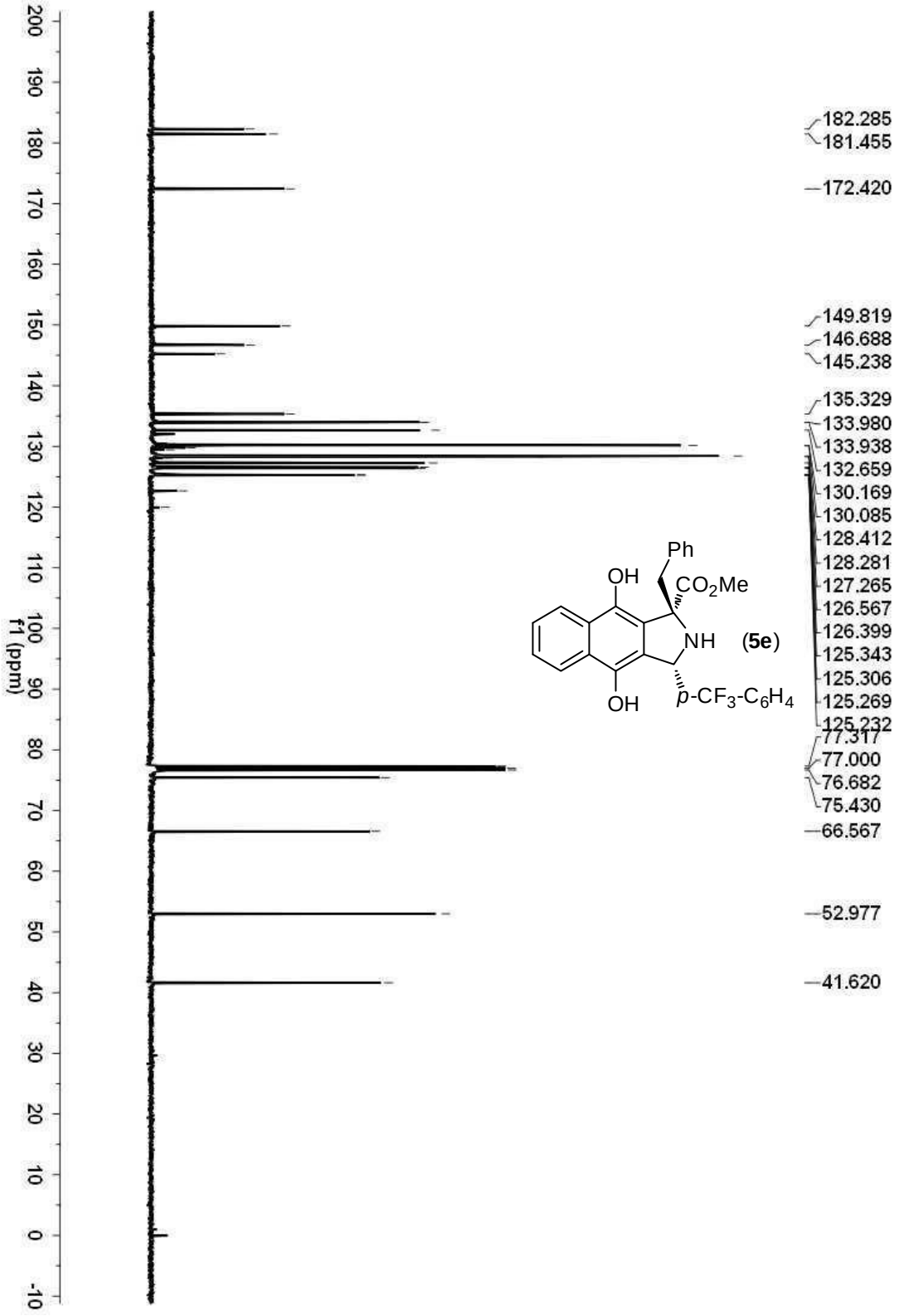
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20
10
0
-10
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181.455
172.420
149.819
146.688
145.238
135.329
133.980
133.938
132.659
130.169
130.085
128.412
128.281
127.265
126.567
126.399
125.343
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77.000
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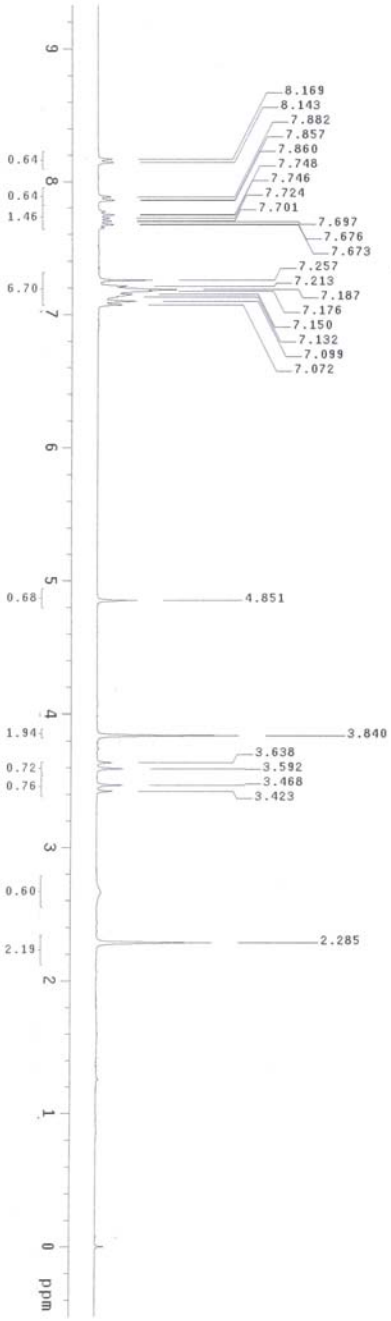
p-CF3-C6H4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7.213
7.187
7.176
7.150
7.132
7.099
7.072
4.851
3.840
3.638
3.592
3.468
3.423
2.285
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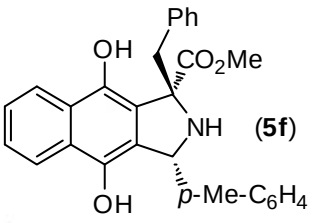
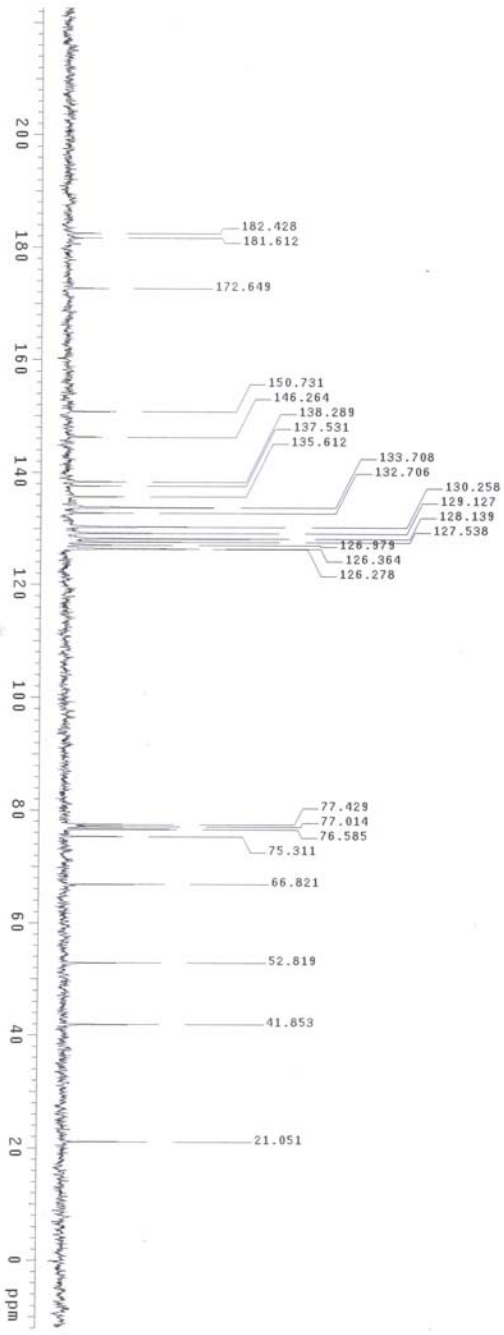
0.64
1.46
6.70
0.68
1.94
0.72
0.76
0.60
2.19
ppm

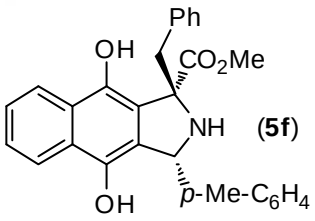

Ph
OH
CO2Me
NH
OH
p-Me-C6H4

(**5f**)

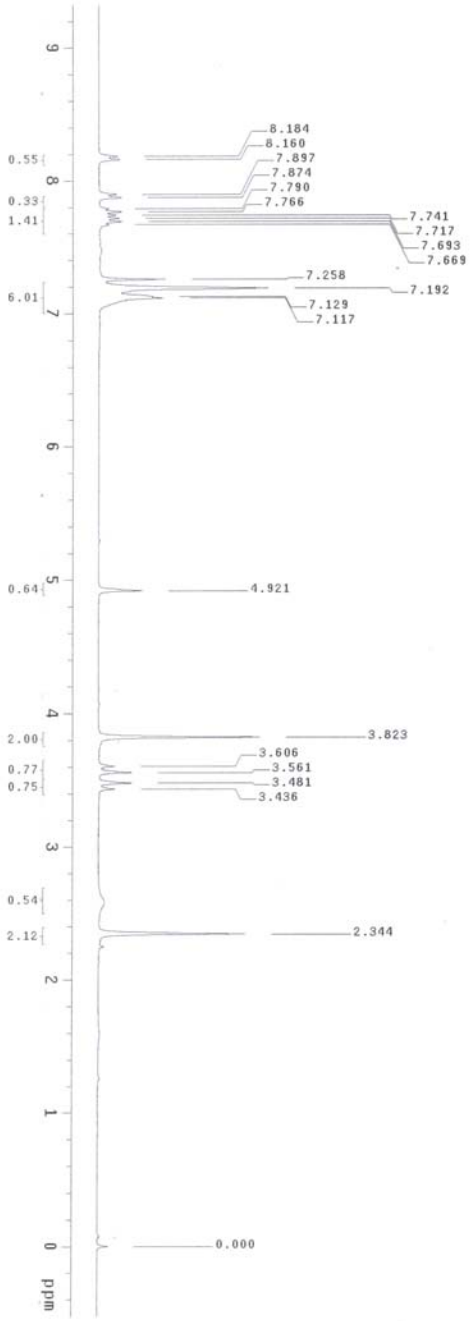


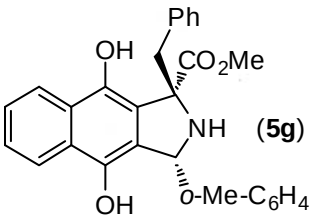
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"

Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
56 repetitions
OBSERVE C13, 75.4554921 MHz
DECOUPLE H1, 300.0815382 MHz
Power 40 dB
continuously on
WALTZ16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec

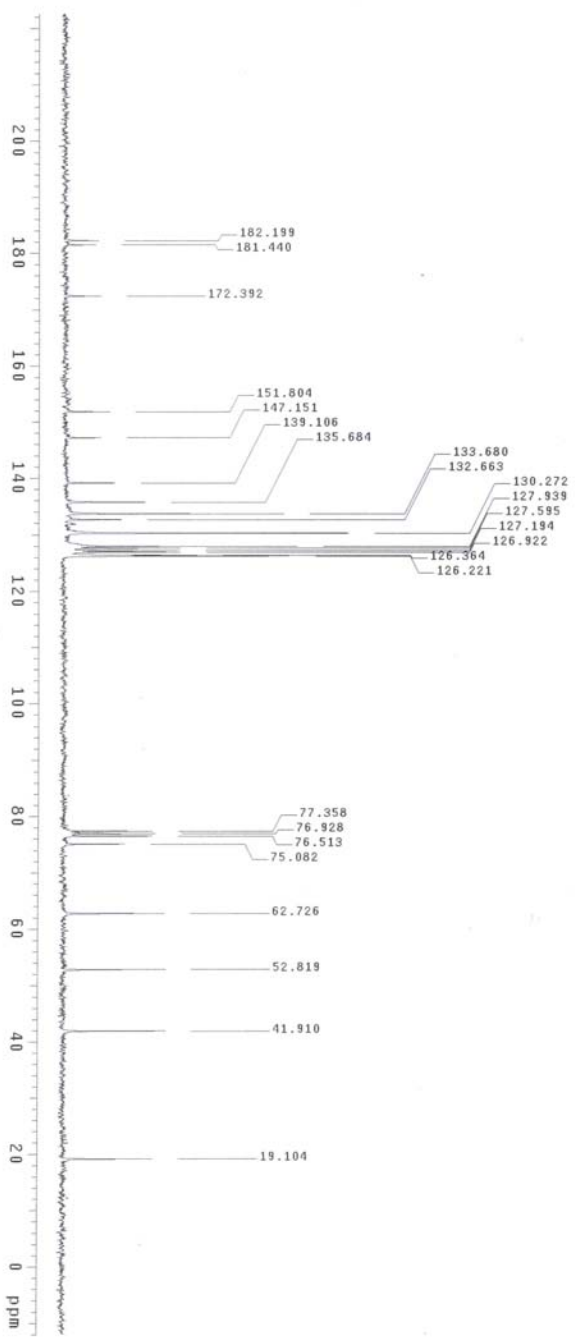


(**5f**)


8.184
8.160
7.897
7.874
7.790
7.766
7.741
7.717
7.693
7.669
7.258
7.192
7.129
7.117
4.921
3.823
3.606
3.561
3.481
3.436
2.344
0.000
0.55
0.33
1.41
6.01
0.64
2.00
0.77
0.75
0.54
2.12
ppm

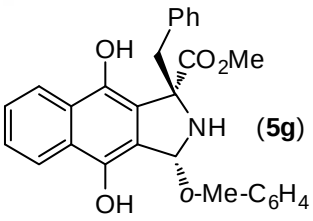

Ph
OH
CO2Me
NH
OH
o-Me-C6H4

(**5g**)

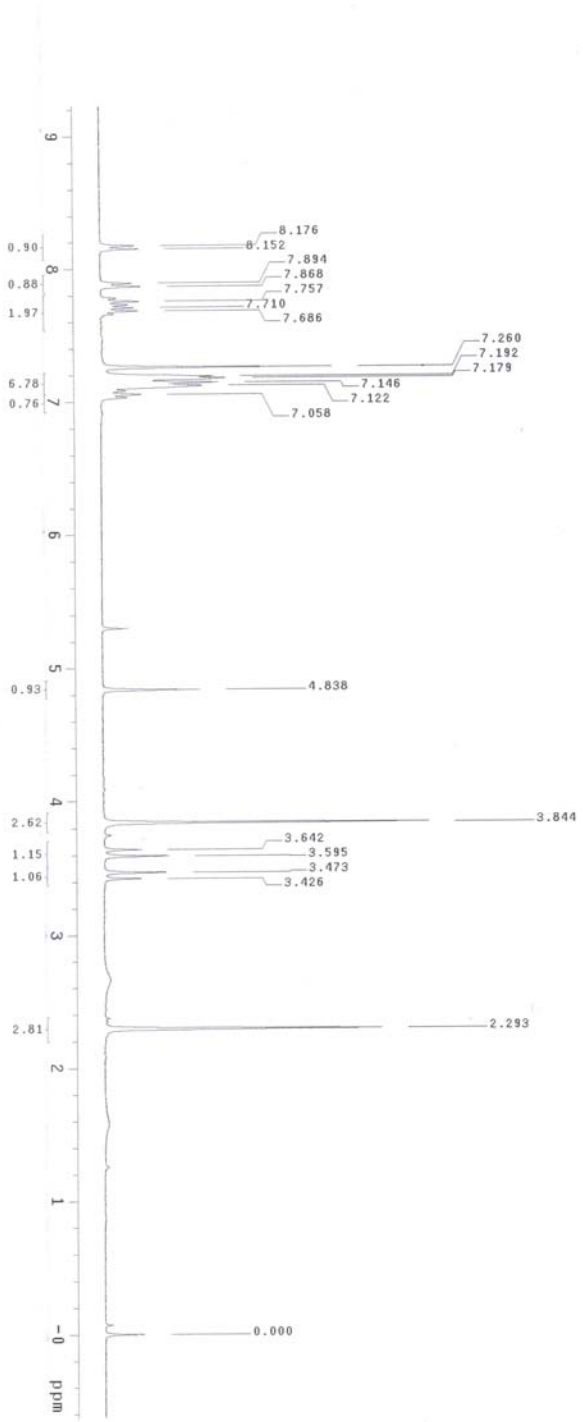


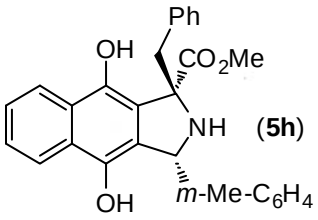
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"

Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
152 repetitions
OBSERVE C13, 75.4554921 MHz
DECOUPLE H1, 300.0815382 MHz
Power 40 dB
continuously on
WALTZ16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec

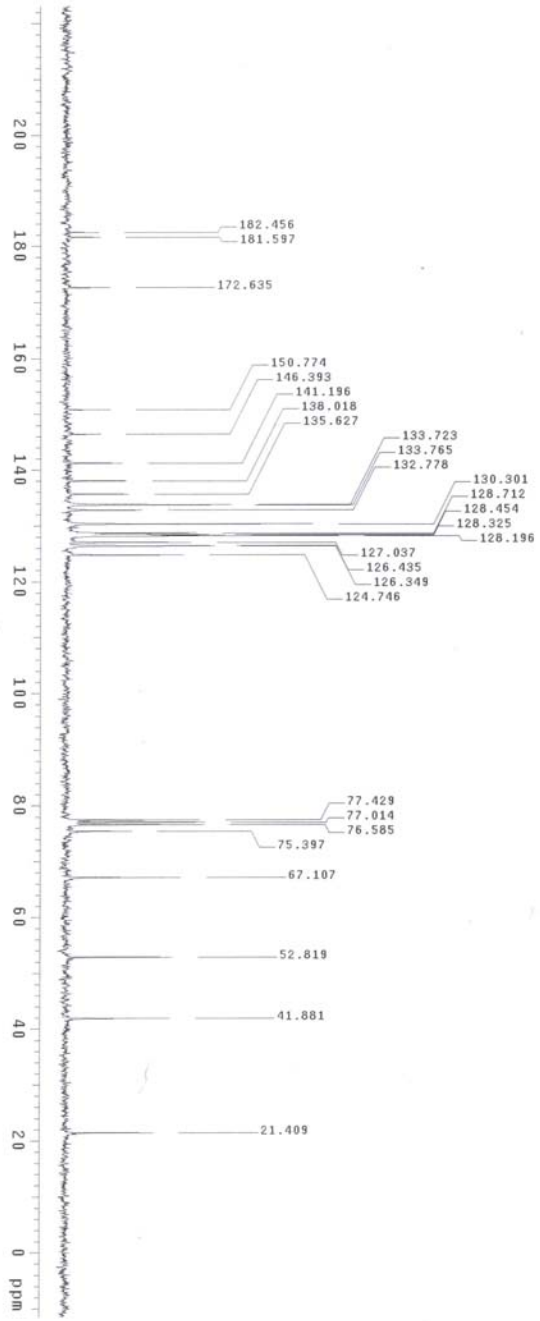


(**5g**)

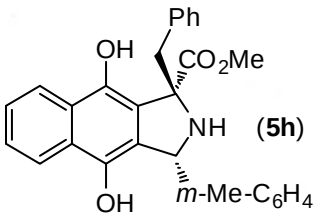

hz1-4-78b-3
8.176
8.152
7.894
7.868
7.757
7.710
7.686
7.260
7.192
7.179
7.146
7.122
7.058
4.838
3.844
3.642
3.595
3.473
3.426
2.293
0.000
0.90
0.88
1.97
6.78
0.76
0.93
2.62
1.15
1.06
2.81
ppm

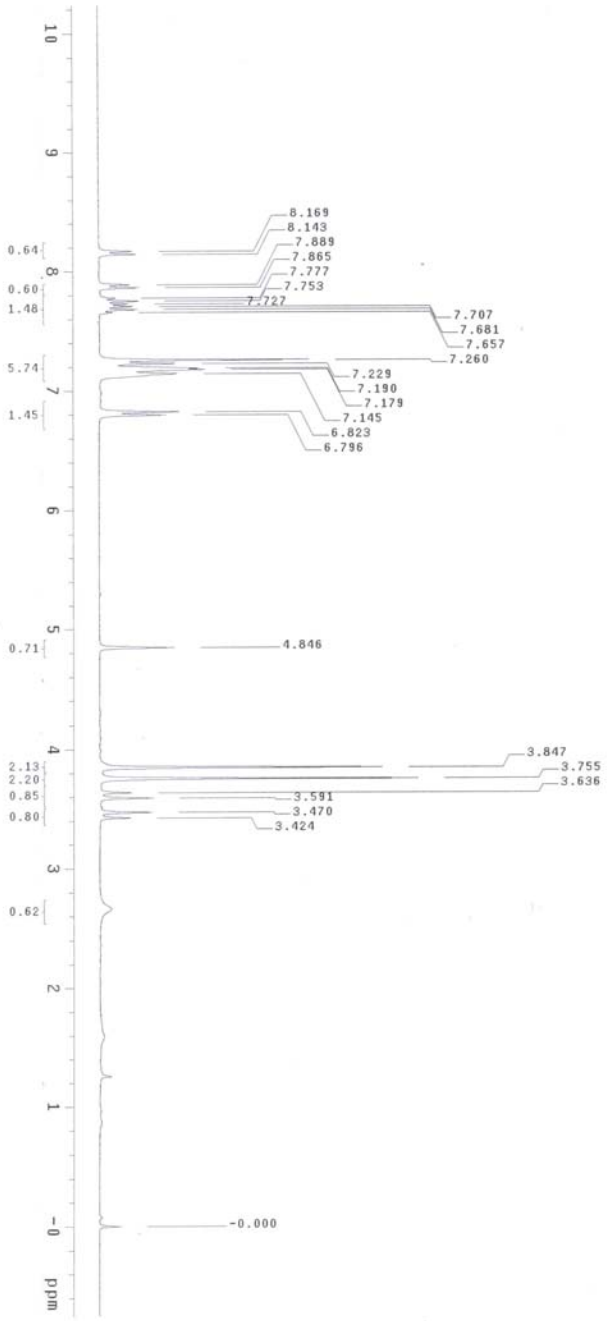

Ph
OH
CO2Me
NH
OH
m-Me-C6H4

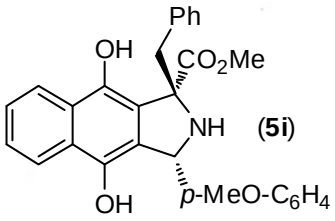
(**5h**)


182.456
181.597
172.635
150.774
146.393
141.196
138.018
135.627
133.723
133.765
132.778
130.301
128.712
128.454
128.325
128.196
127.037
126.435
126.349
124.746
77.429
77.014
76.585
75.397
67.107
52.819
41.881
21.409
200
180
160
140
120
100
80
60
40
20
0 ppm

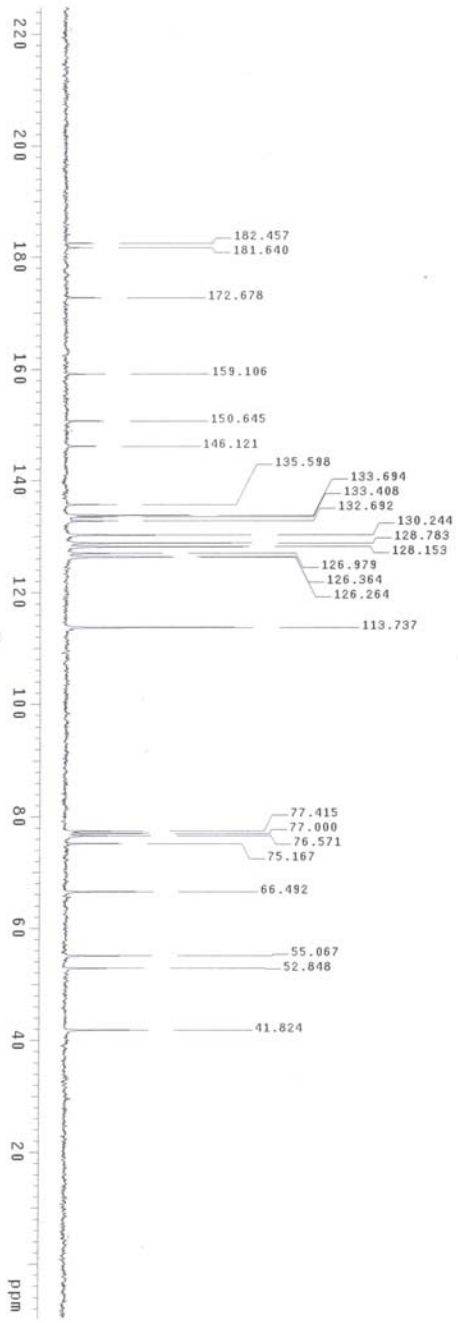

Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
208 repetitions
OBSERVE C13, 75.4554543 MHz
DECOUPLE H1, 300.0815382 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec


Ph
OH
CO2Me
NH
(5h)
m-Me-C6H4
OH

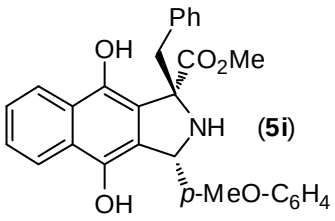

hzl-4-33b
8.169
8.143
7.889
7.865
7.777
7.753
7.727
7.707
7.681
7.657
7.260
7.229
7.190
7.179
7.145
6.823
6.796
4.846
3.847
3.755
3.636
3.591
3.470
3.424
-0.000
0.64
0.60
1.48
5.74
1.45
0.71
2.13
2.20
0.85
0.80
0.62
ppm

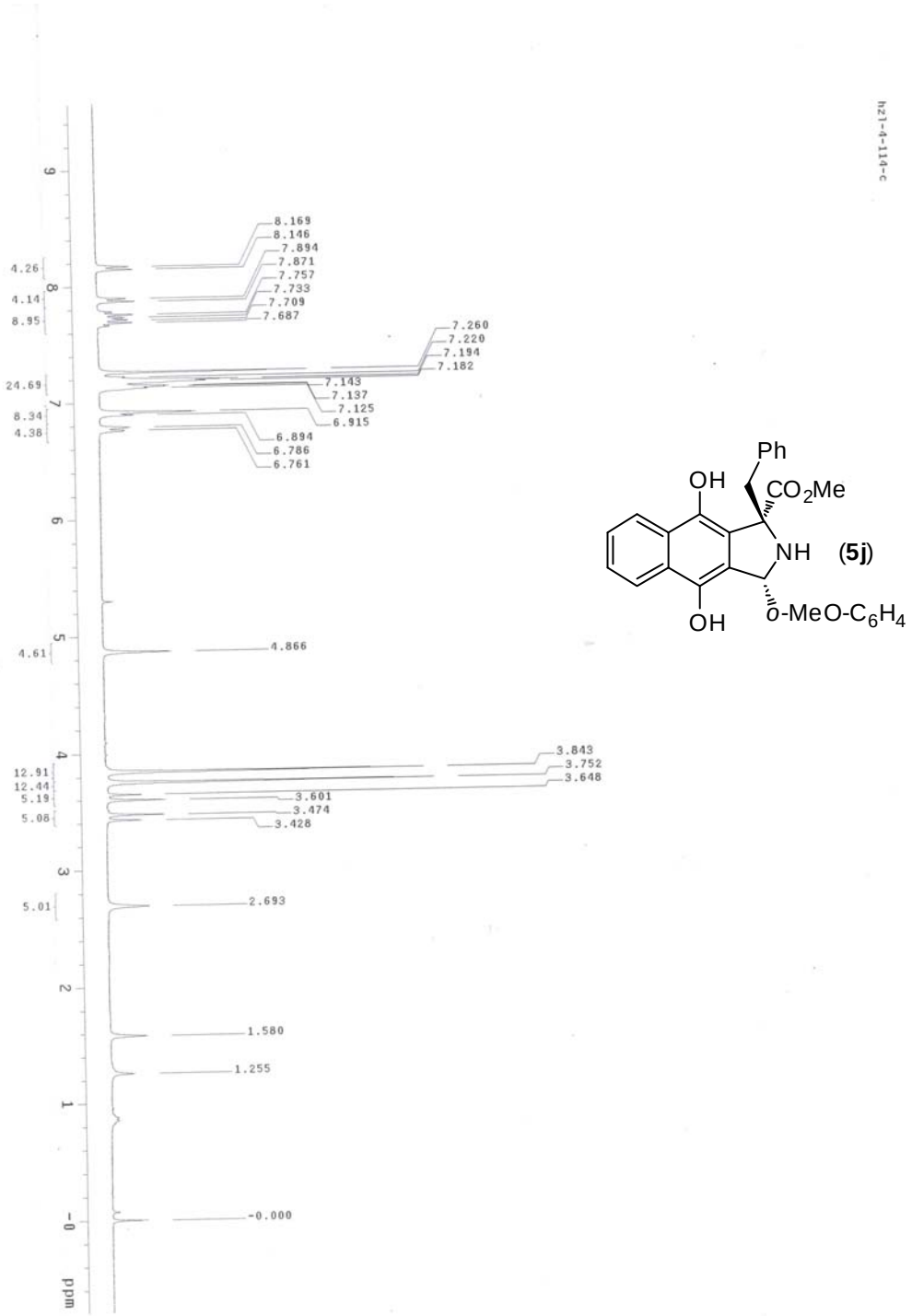

Ph
OH
CO2Me
NH
OH
p-MeO-C6H4

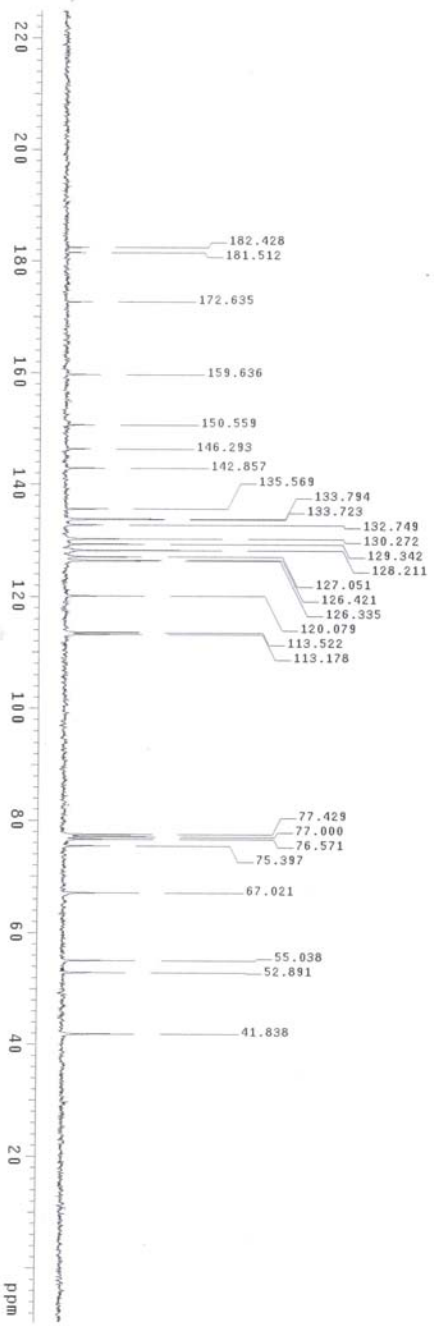
(**5i**)


182.457
181.640
172.678
159.106
150.645
146.121
135.598
133.694
133.408
132.692
130.244
128.783
128.153
126.979
126.364
126.264
113.737
77.415
77.000
76.571
75.167
66.492
55.067
52.848
41.824
ppm


Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
192 repetitions
OBSERVE C13, 75.4553172 MHz
DECOUPLE H1, 300.0815382 MHz
Power 40 dB
continuously on
WALTZ16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec

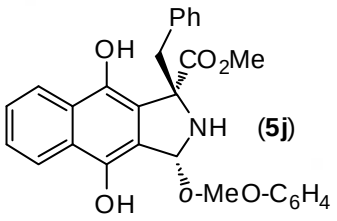

Ph
OH
CO2Me
NH
(5i)
OH
p-MeO-C6H4


hz1-4-114-c
8.169
8.146
7.894
7.871
7.757
7.733
7.709
7.687
7.260
7.220
7.194
7.182
7.143
7.137
7.125
6.915
6.894
6.786
6.761
4.866
3.843
3.752
3.648
3.601
3.474
3.428
2.693
1.580
1.255
-0.000
4.26
4.14
8.95
24.69
8.34
4.38
4.61
12.91
12.44
5.19
5.08
5.01
ppm
Ph
OH
CO2Me
NH
(5j)
o-MeO-C6H4
OH

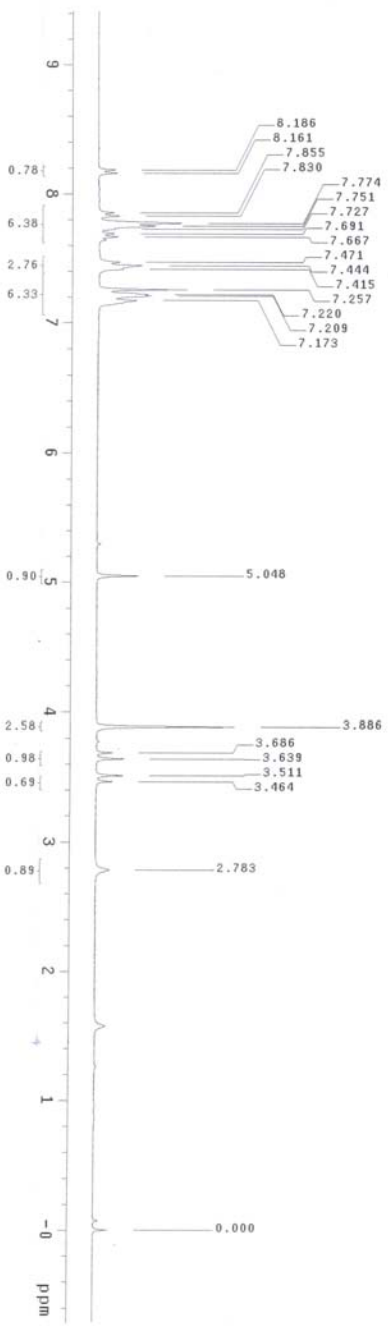


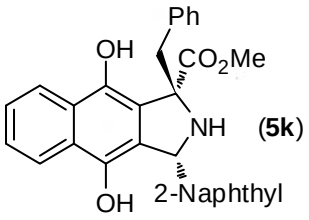
Solvent: CDCl3
Ambient temperature
Mercury-300B "mercury300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.580 sec
Width 17869.1 Hz
208 repetitions
OBSERVE C13, 75.4553161 MHz
DECOUPLE H1, 300.0815382 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec

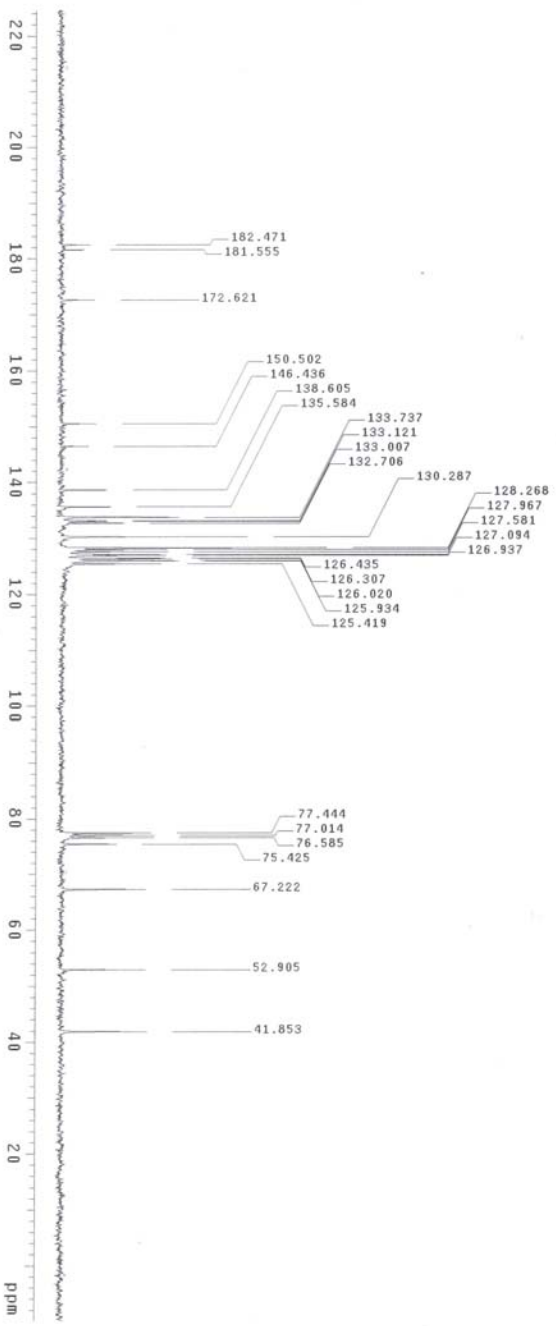


(**5j**)


8.186
8.161
7.855
7.830
7.774
7.751
7.727
7.691
7.667
7.471
7.444
7.415
7.257
7.220
7.209
7.173
5.048
3.886
3.686
3.639
3.511
3.464
2.783
0.000
0.78
6.38
2.76
6.33
0.90
2.58
0.98
0.69
0.89
ppm

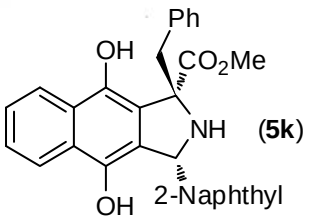

Ph
OH
CO2Me
NH
OH
2-Naphthyl

(**5k**)

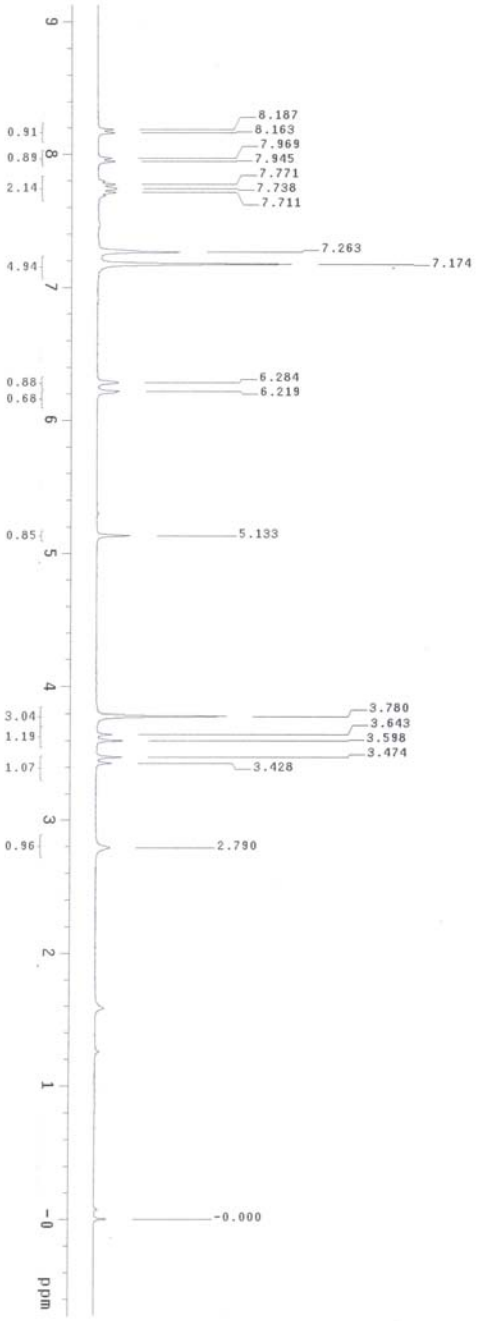


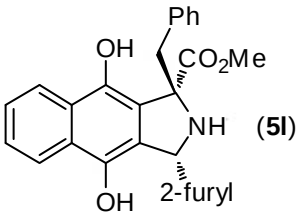
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"

Relax. delay 1.000 sec
Pulse 30.0 degrees
Acq. time 0.350 sec
Width 17699.1 Hz
192 repetitions
OBSERVE C13, 75.4553388 MHz
DECOUPLE H1, 300.0815382 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec

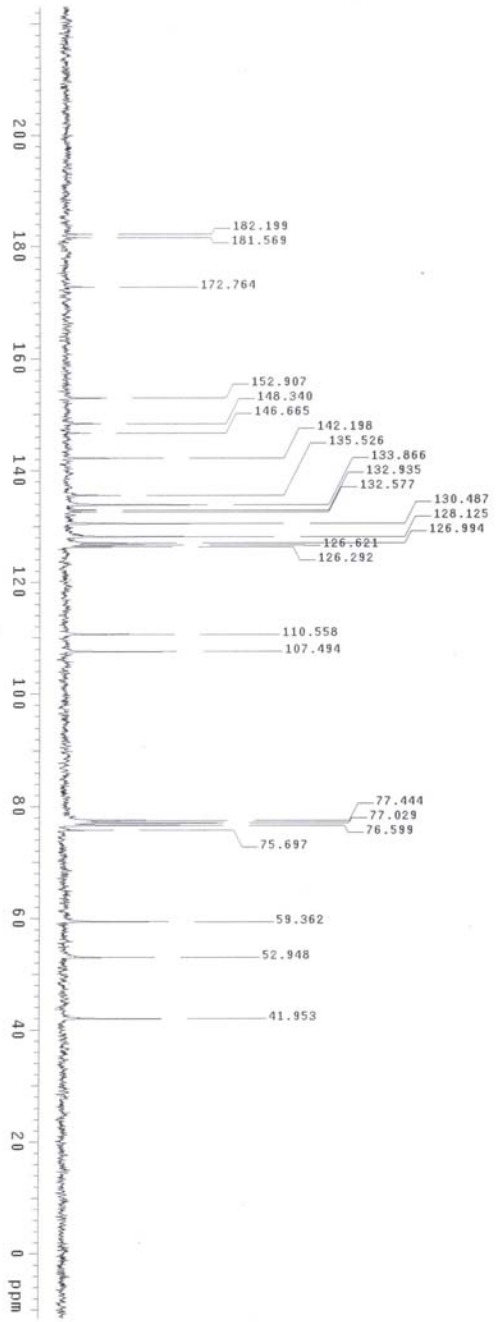


(**5k**)

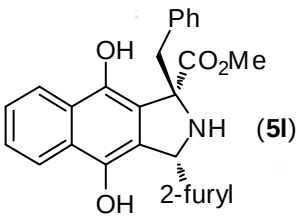

8.187
8.163
7.969
7.945
7.771
7.738
7.711
7.263
7.174
6.284
6.219
5.133
3.780
3.643
3.598
3.474
3.428
2.790
-0.000
ppm


Ph
OH
CO2Me
NH
OH
2-furyl

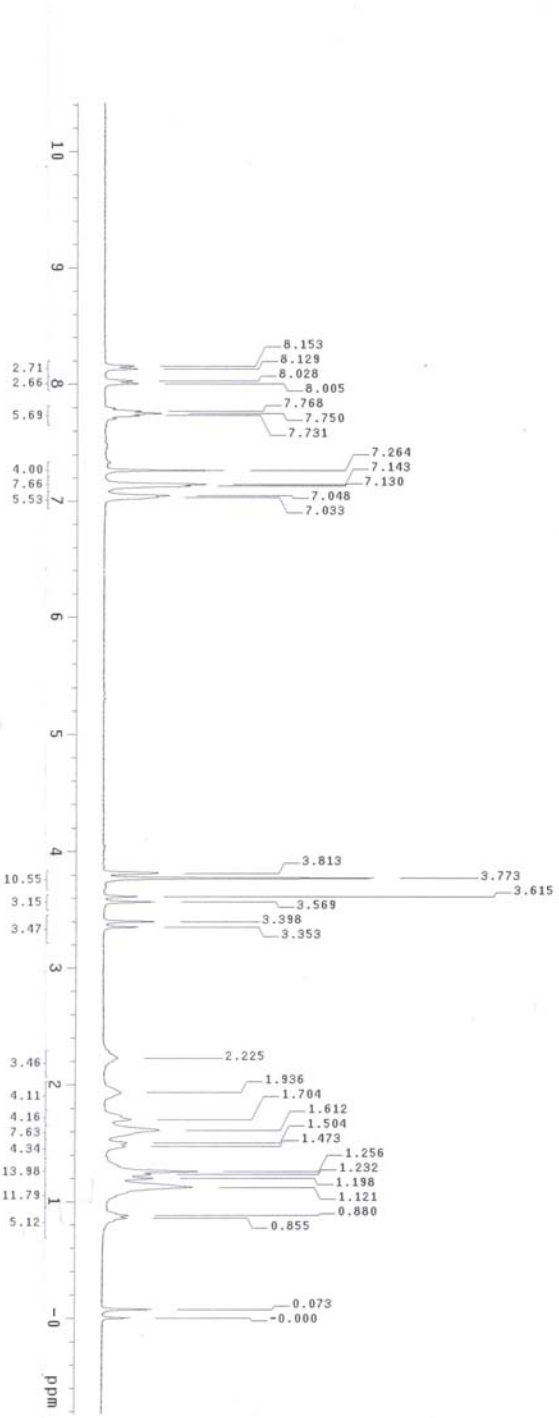
(**5l**)

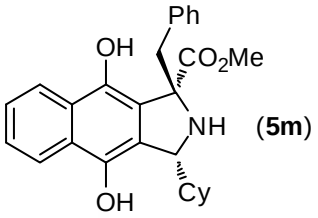

182.199
181.569
172.764
152.907
148.340
146.665
142.198
135.526
133.866
132.935
132.577
130.487
128.125
126.994
126.621
126.292
110.558
107.494
77.444
77.029
76.599
75.697
59.362
52.948
41.953
200
180
160
140
120
100
80
60
40
20
0
ppm


Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
248 repetitions
OBSERVE C13, 75.4554597 MHz
DECOUPLE H1, 300.0815382 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec

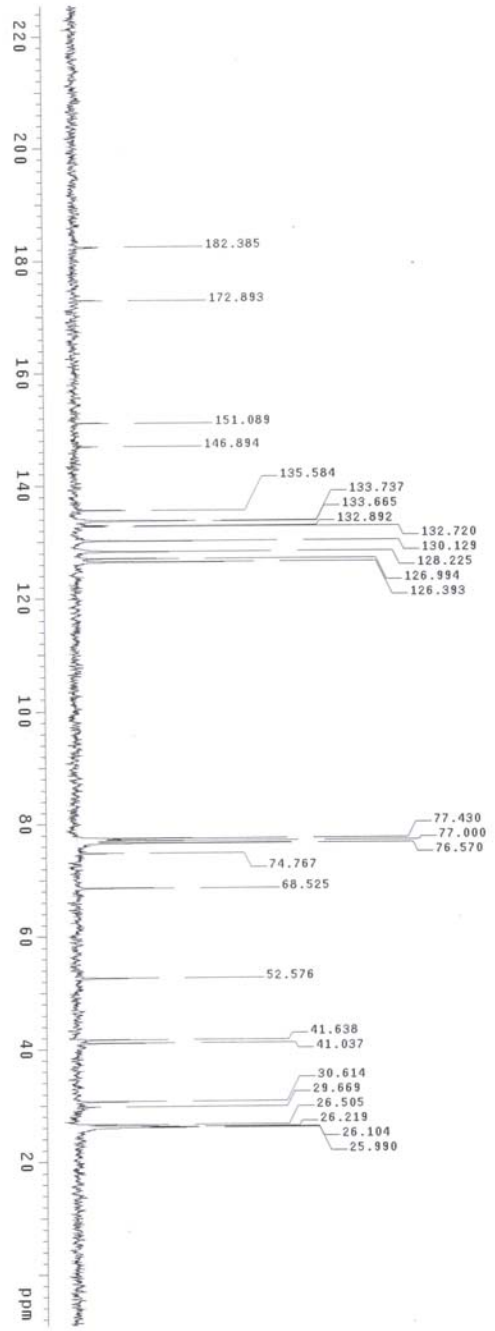

Ph
OH
CO2Me
NH
OH
2-furyl

(**5l**)


hz1-4-123
Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
8.153
8.129
8.028
8.005
7.768
7.750
7.731
7.264
7.143
7.130
7.048
7.033
3.813
3.773
3.615
3.569
3.398
3.353
2.225
1.936
1.704
1.612
1.504
1.473
1.256
1.232
1.198
1.121
0.880
0.855
0.073
-0.000
2.71
2.66
5.69
4.00
7.66
5.53
10.55
3.15
3.47
3.46
4.11
4.16
7.63
4.34
13.98
11.79
5.12
ppm


Ph
OH
CO2Me
NH
Cy
OH

(**5m**)

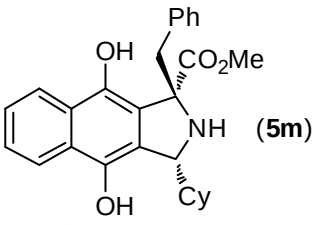


Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: CARBON

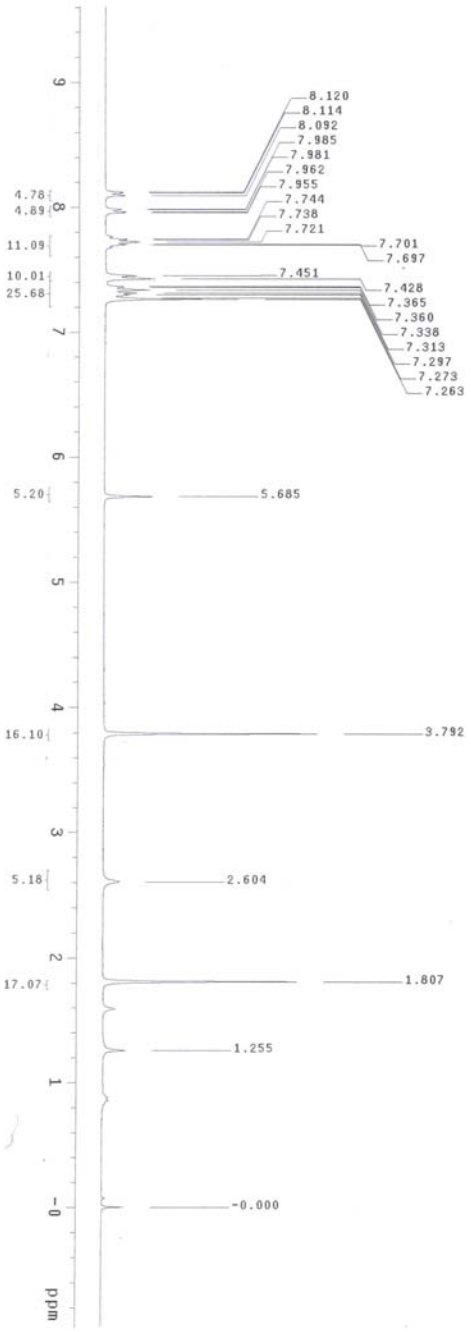
Pulse Sequence: s2pul

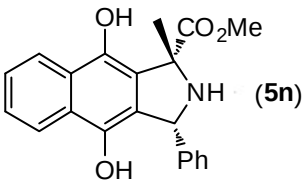
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"

Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.499 sec
Width 17699.1 Hz
600 repetitions
OBSERVE C13, 75.4552272 MHz
DECOUPLE H1, 300.0820522 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 19 sec

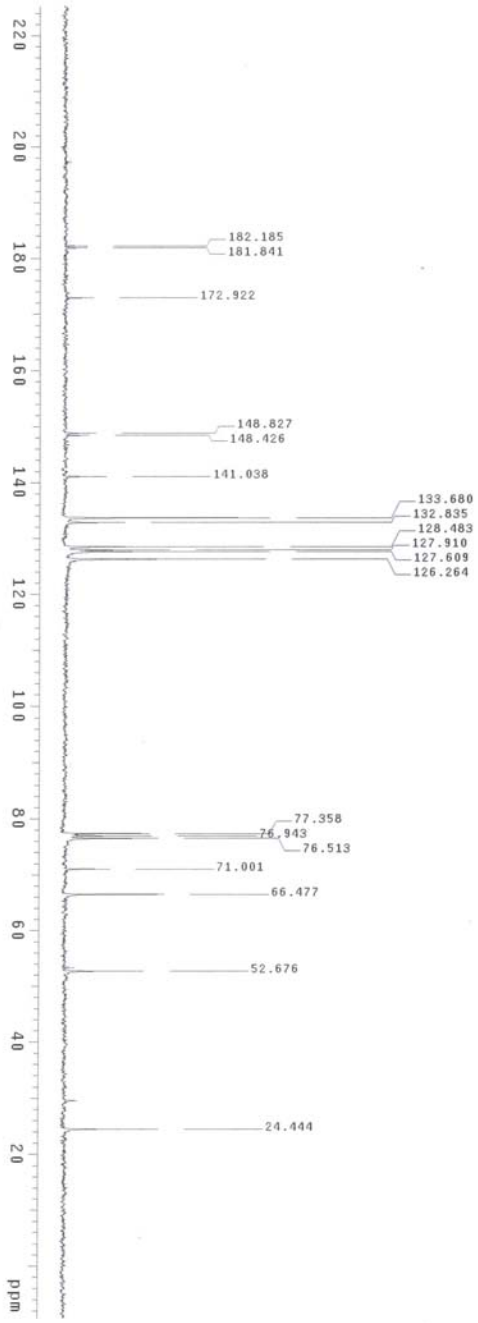


(**5m**)

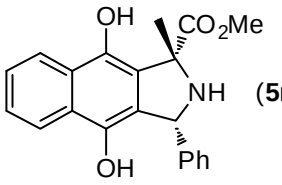

8.120
8.114
8.092
7.985
7.981
7.962
7.955
7.744
7.738
7.721
7.701
7.697
7.451
7.428
7.365
7.360
7.338
7.313
7.297
7.273
7.263
5.685
3.792
2.604
1.807
1.255
-0.000
4.78
4.89
11.09
10.01
25.68
5.20
16.10
5.18
17.07
ppm


OH
CO2Me
NH
Ph
OH

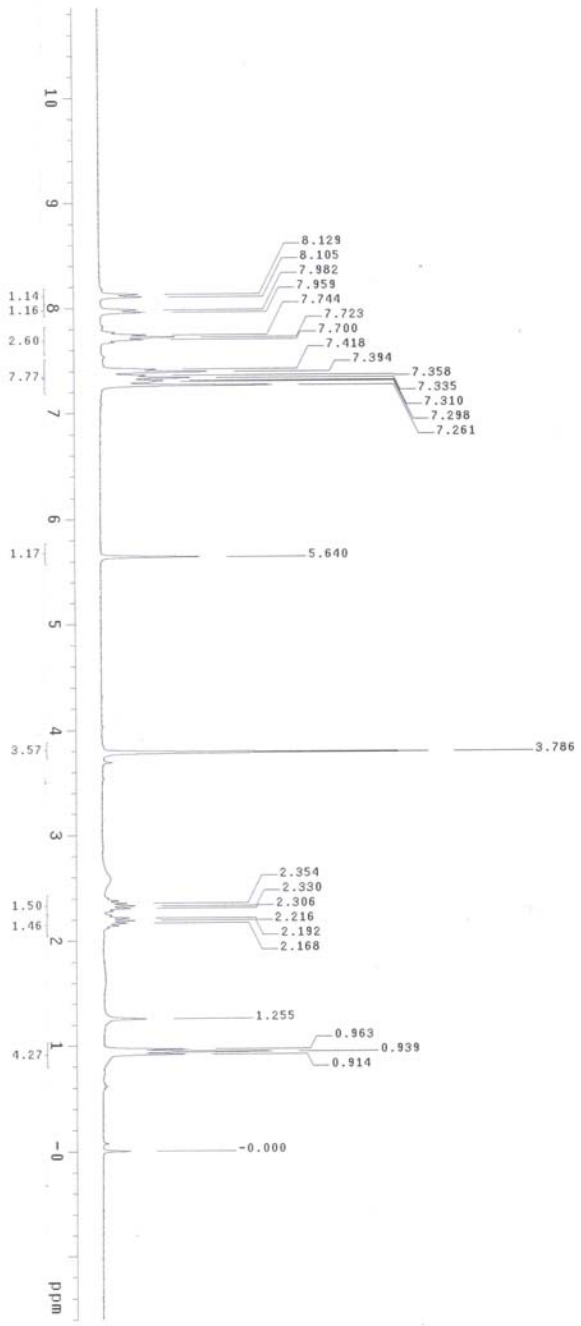
(**5n**)

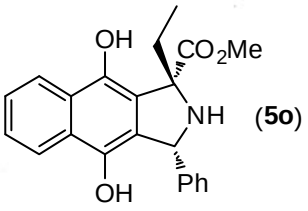

182.185
181.841
172.922
148.827
148.426
141.038
133.680
132.835
128.483
127.910
127.609
126.264
77.358
76.943
76.513
71.001
66.477
52.676
24.444
220
200
180
160
140
120
100
80
60
40
20
ppm

Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
368 repetitions
OBSERVE C13, 75.4552891 MHz
DECOUPLE H1, 300.0815382 MHz
Power 40 dB
Continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec

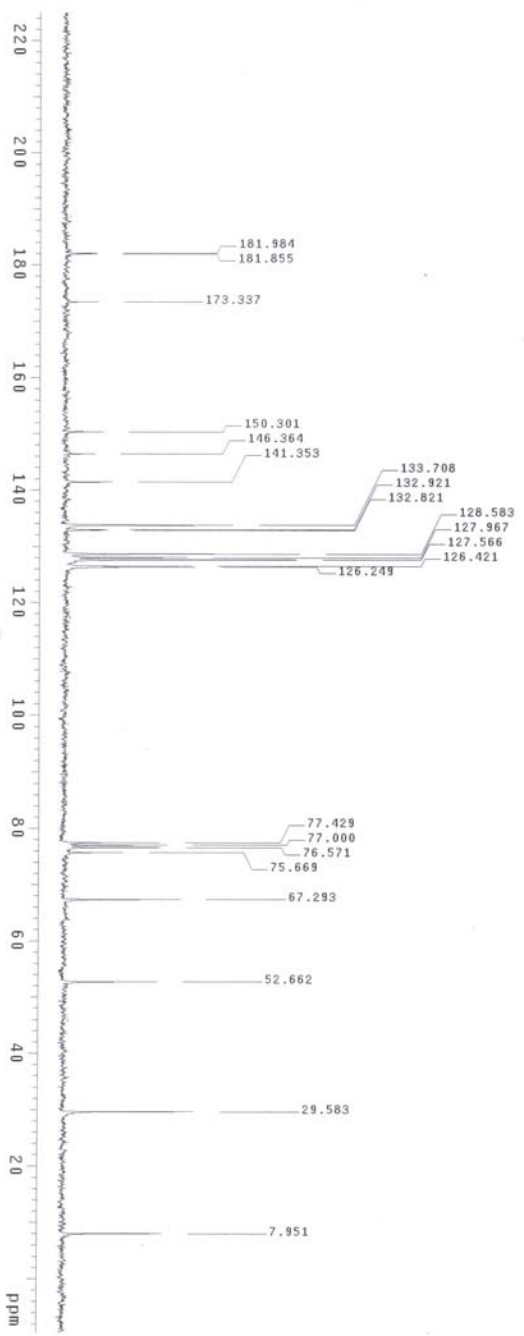

OH
CO2Me
NH
Ph
OH

(**5n**)

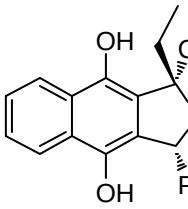

hz1-3-106b-2
8.129
8.105
7.982
7.959
7.744
7.723
7.700
7.418
7.394
7.358
7.335
7.310
7.298
7.261
5.640
3.786
2.354
2.330
2.306
2.216
2.192
2.168
1.255
0.963
0.939
0.914
-0.000
1.14
1.16
2.60
7.77
1.17
3.57
1.50
1.46
4.27
ppm


OH
CO2Me
NH
Ph
OH

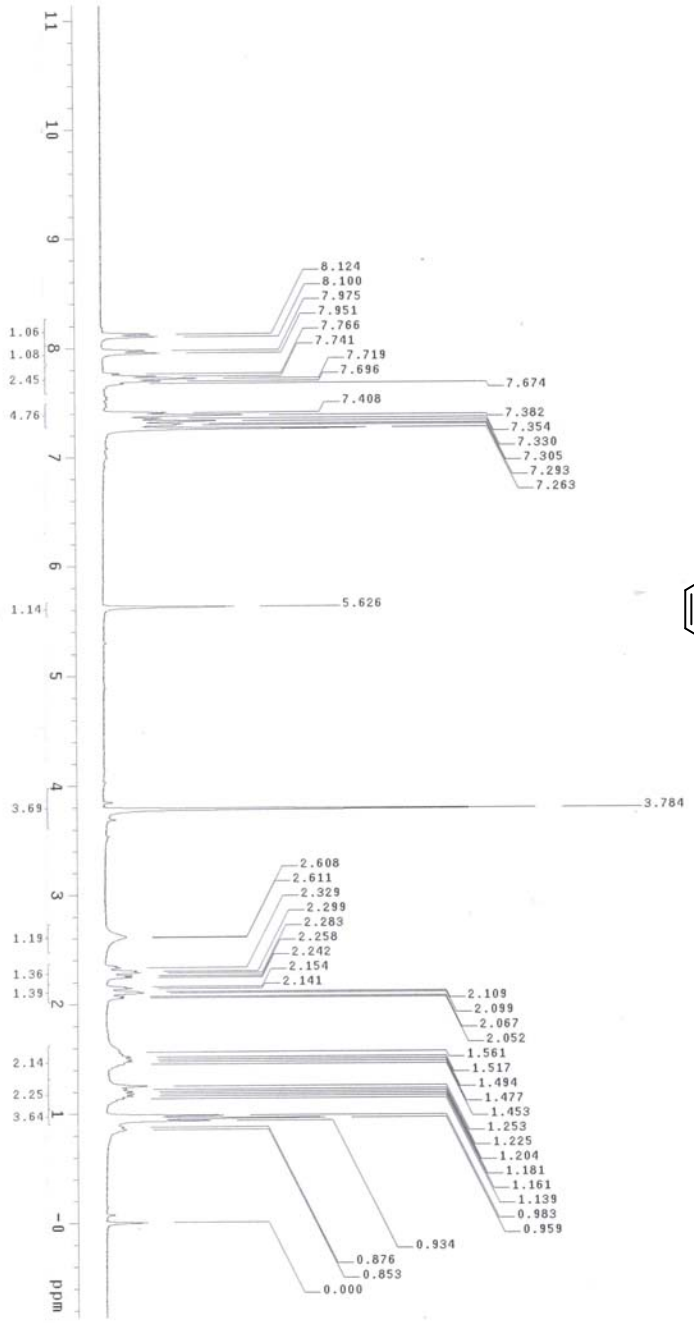
(**5o**)

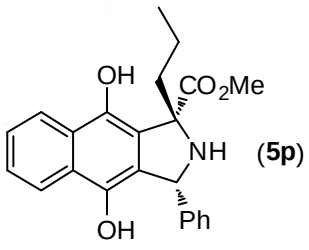

220
200
180
160
140
120
100
80
60
40
20
ppm
181.984
181.855
173.337
150.301
146.364
141.353
133.708
132.921
132.821
128.583
127.967
127.566
126.421
126.249
77.429
77.000
76.571
75.669
67.293
52.662
29.583
7.951


Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
184 repetitions
OBSERVE C13, 75.4553064 MHz
DECOUPLE H1, 300.0815382 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec

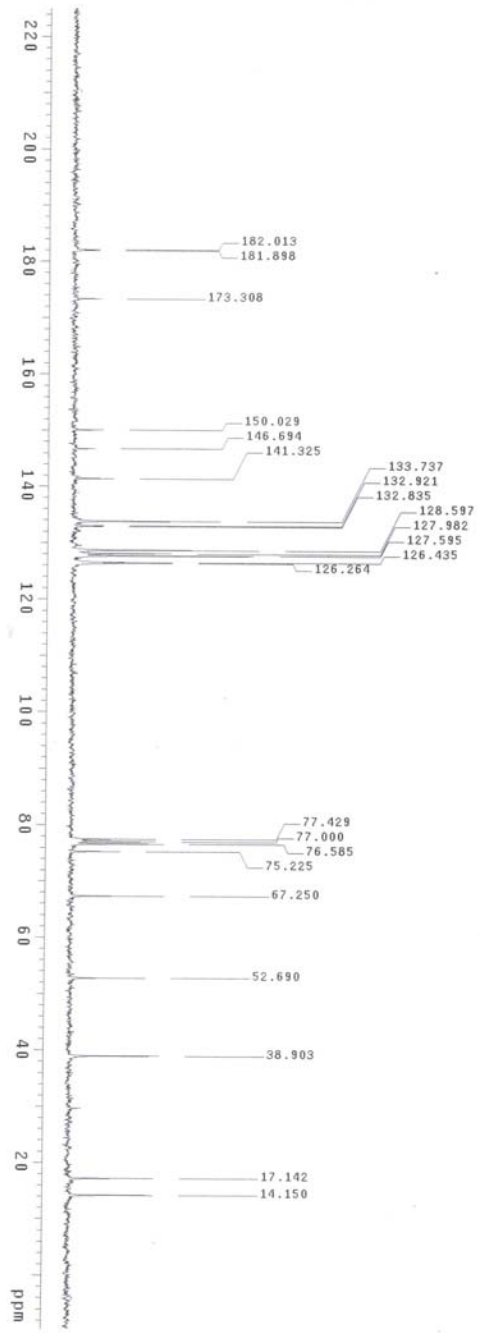

OH
CO2Me
NH
Ph
OH

(**5o**)


hzl-4-112b-2
8.124
8.100
7.975
7.951
7.766
7.741
7.719
7.696
7.674
7.408
7.382
7.354
7.330
7.305
7.293
7.263
5.626
3.784
2.608
2.611
2.329
2.299
2.283
2.258
2.242
2.154
2.141
2.109
2.099
2.067
2.052
1.561
1.517
1.494
1.477
1.453
1.253
1.225
1.204
1.181
1.161
1.139
0.983
0.959
0.934
0.876
0.853
0.000
ppm

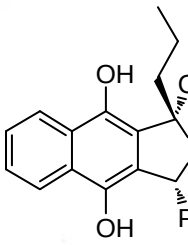

OH
CO2Me
NH
Ph
OH

(**5p**)

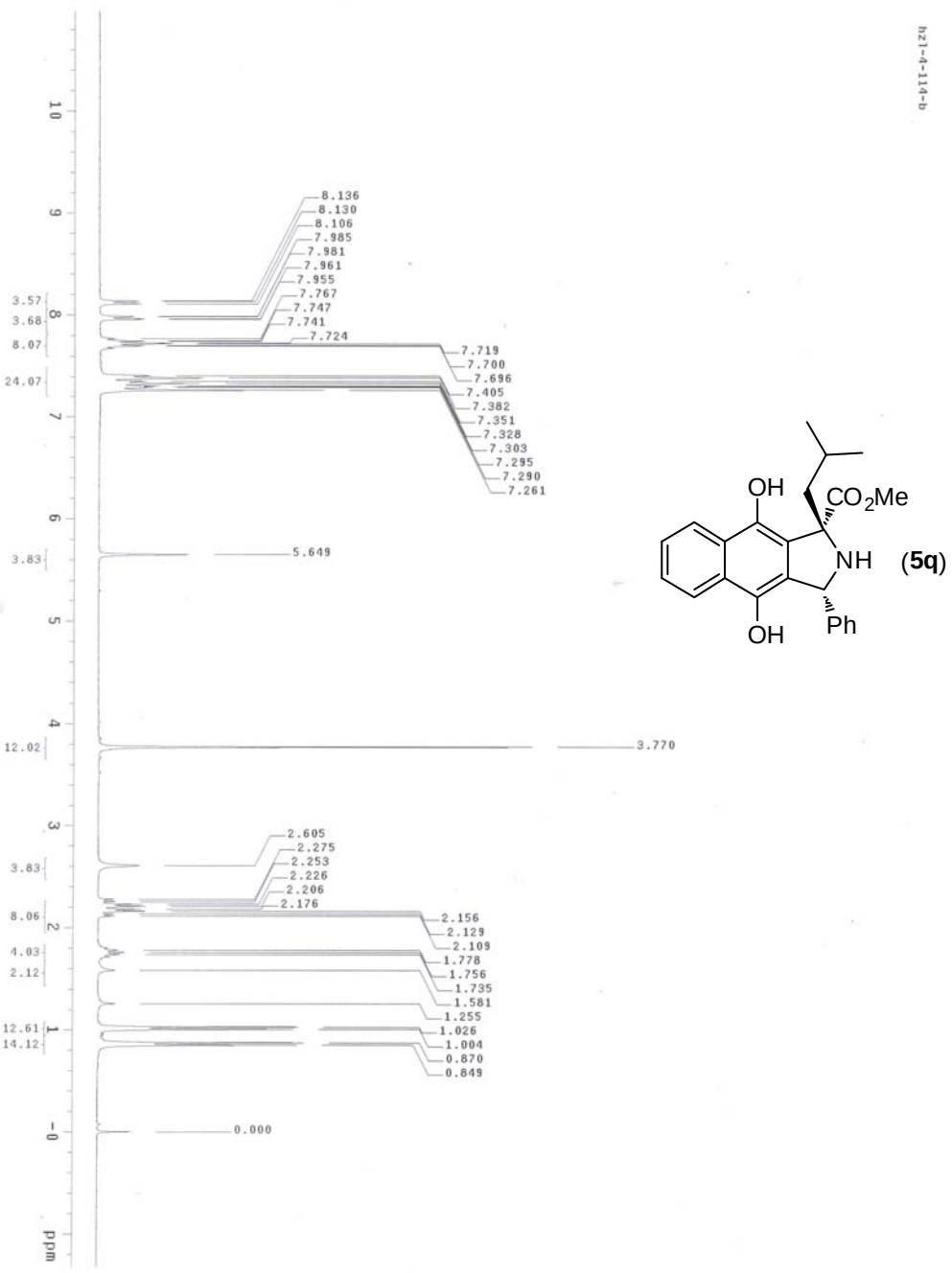


Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 0.500 sec
Width 17699.1 Hz
192 repetitions
OBSERVE C13, 75.4553053 MHz
DECOUPLE H1, 300.0815382 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec



(**5p**)


hz1-4-114-b
8.136
8.130
8.106
7.985
7.981
7.961
7.955
7.767
7.747
7.741
7.724
7.719
7.700
7.696
7.405
7.382
7.351
7.328
7.303
7.295
7.290
7.261
5.649
3.770
2.605
2.275
2.253
2.226
2.206
2.176
2.156
2.129
2.109
1.778
1.756
1.735
1.581
1.255
1.026
1.004
0.870
0.849
0.000
3.57
3.68
8.07
24.07
3.83
12.02
3.83
8.06
4.03
2.12
12.61
14.12
ppm

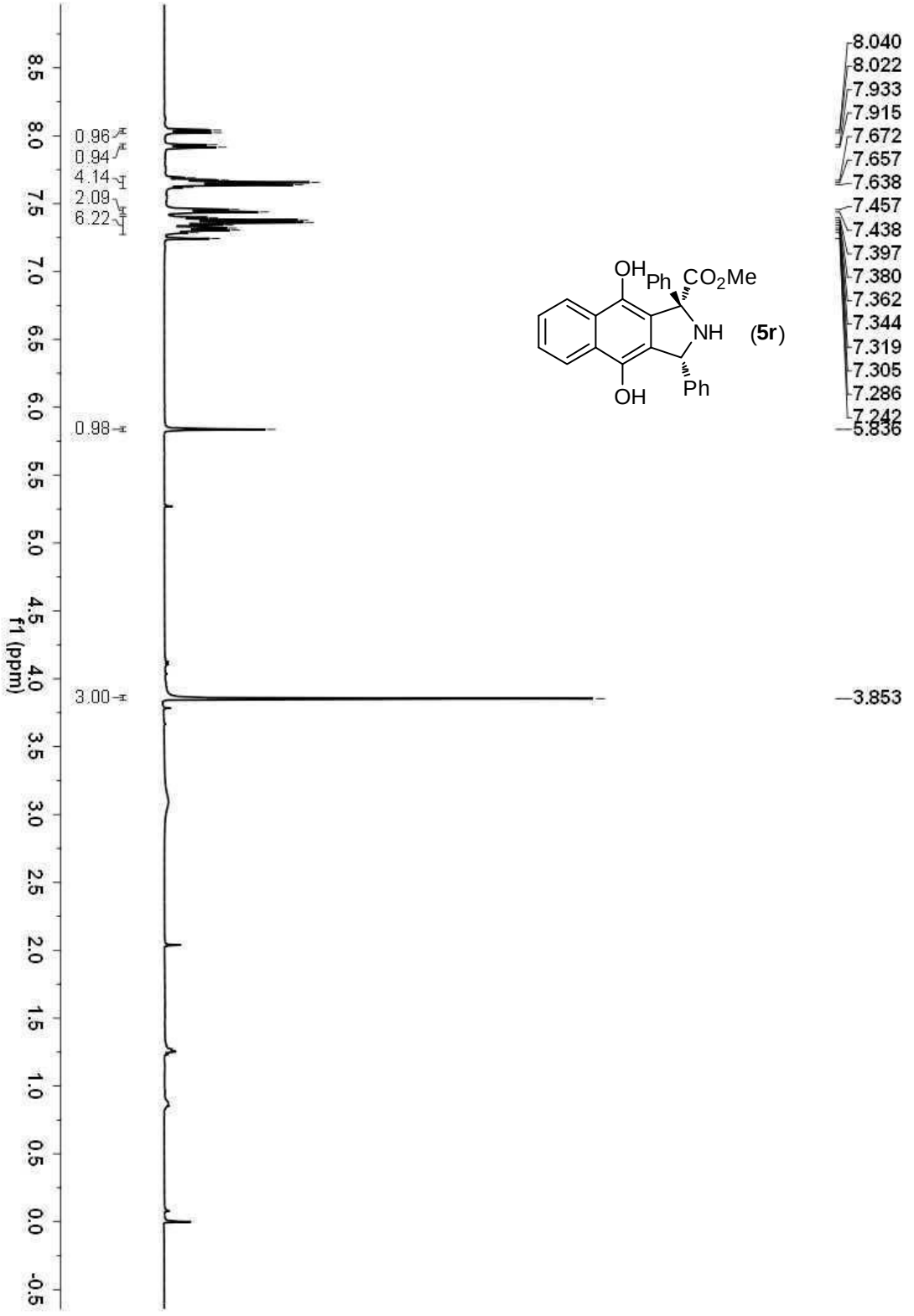
OH
CO2Me
NH
Ph
OH

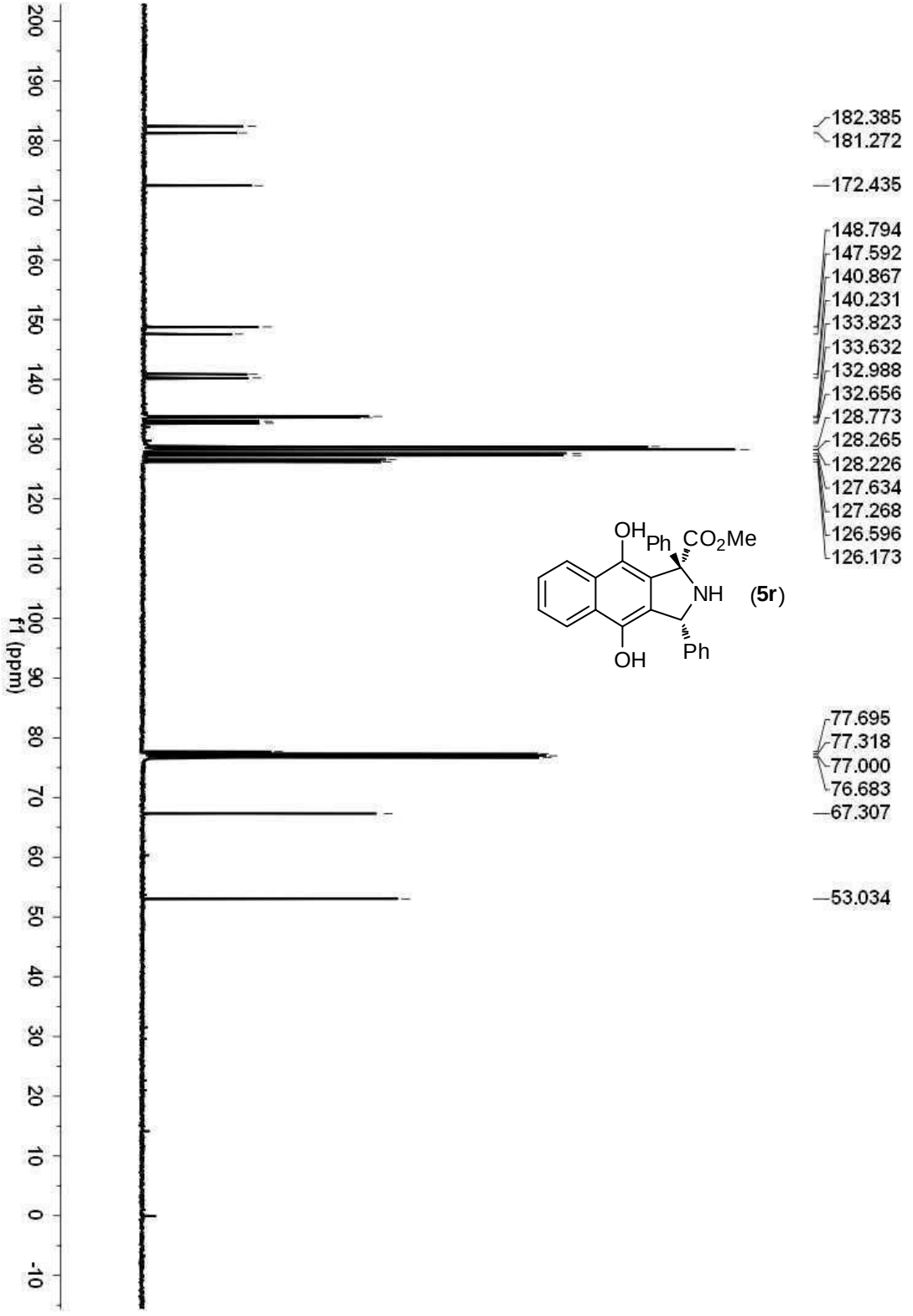
(**5q**)

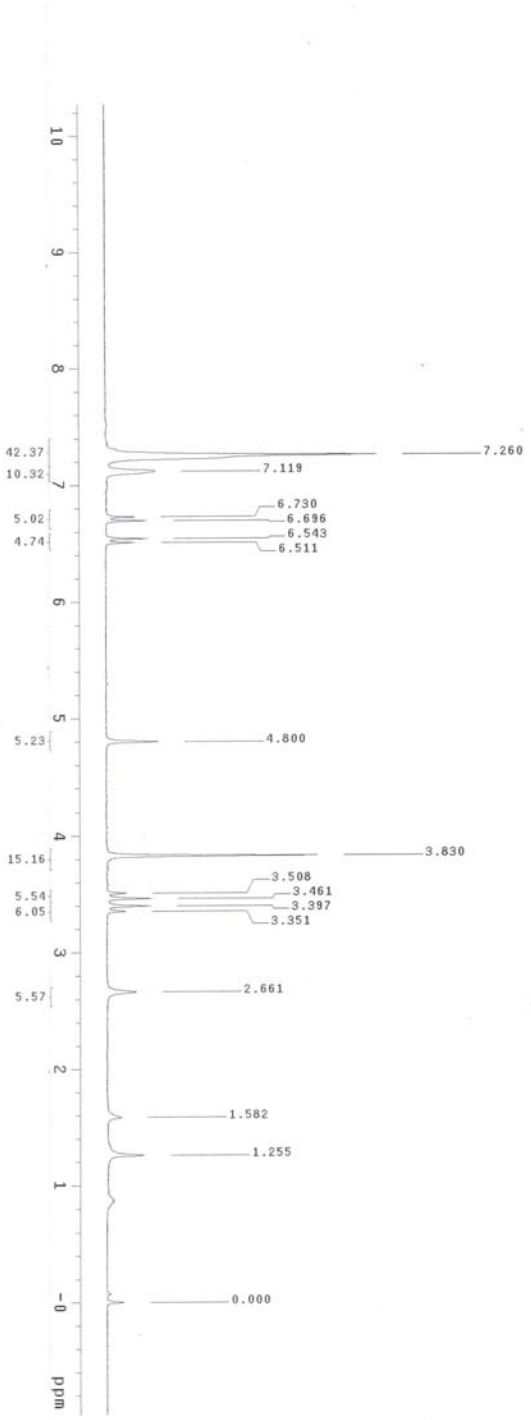
Solvent: CDCl3
Ambient temperature
Mercury-300BB "mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.500 sec
Width 19531.2 Hz
280 repetitions
OBSERVE C13, 75.4553085 MHz
DECOUPLE H1, 300.0815382 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 23 sec
182.084
173.537
149.943
147.166
141.339
133.780
133.708
132.992
132.806
128.583
127.967
127.638
126.521
126.278
77.429
77.000
76.585
75.311
67.036
52.690
44.745
24.544
24.444
24.329
220
200
180
160
140
120
100
80
60
40
20
ppm

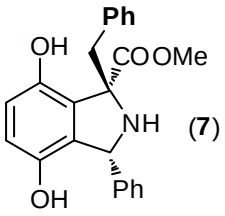
OH
CO2Me
NH
Ph
OH

(**5q**)

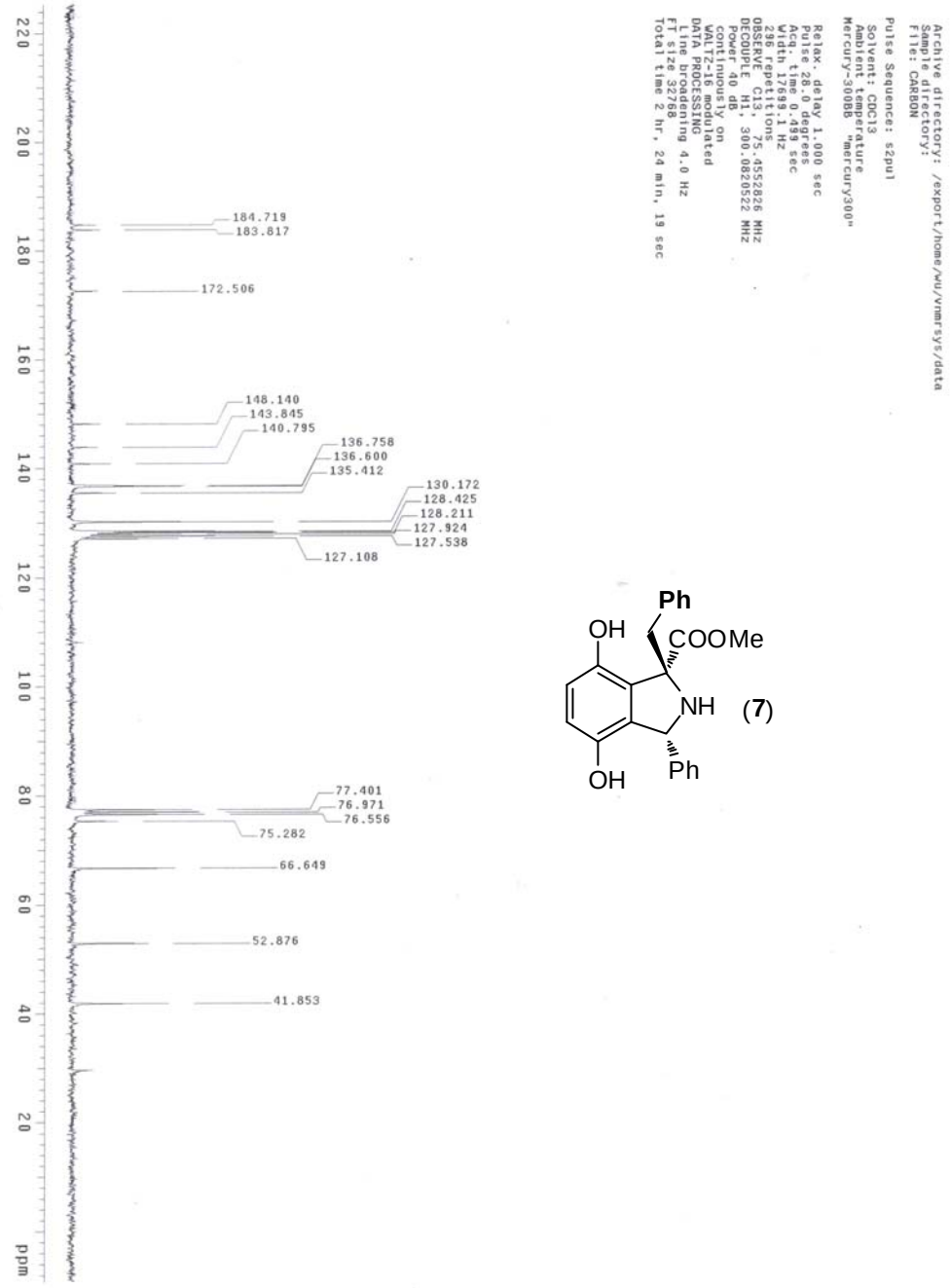

8.040
8.022
7.933
7.915
7.672
7.657
7.638
7.457
7.438
7.397
7.380
7.362
7.344
7.319
7.305
7.286
7.242
5.836
3.853
0.96
0.94
4.14
2.09
6.22
0.98
3.00
f1 (ppm)
OH
Ph
CO2Me
NH
Ph
OH
(5r)


182.385
181.272
172.435
148.794
147.592
140.867
140.231
133.823
133.632
132.988
132.656
128.773
128.265
128.226
127.634
127.268
126.596
126.173
77.695
77.318
77.000
76.683
67.307
53.034
f1 (ppm)
OH
Ph
CO2Me
NH
Ph
OH
(5r)


hz1-4-120
Archive directory: /export/home/vm/vnmrsys/data
Sample directory:
File: PROTON
Pulse Sequence: s2pul
42.37
10.32
5.02
4.74
5.23
15.16
5.54
6.05
5.57
7.260
7.119
6.730
6.696
6.543
6.511
4.800
3.830
3.508
3.461
3.397
3.351
2.661
1.582
1.255
0.000
ppm

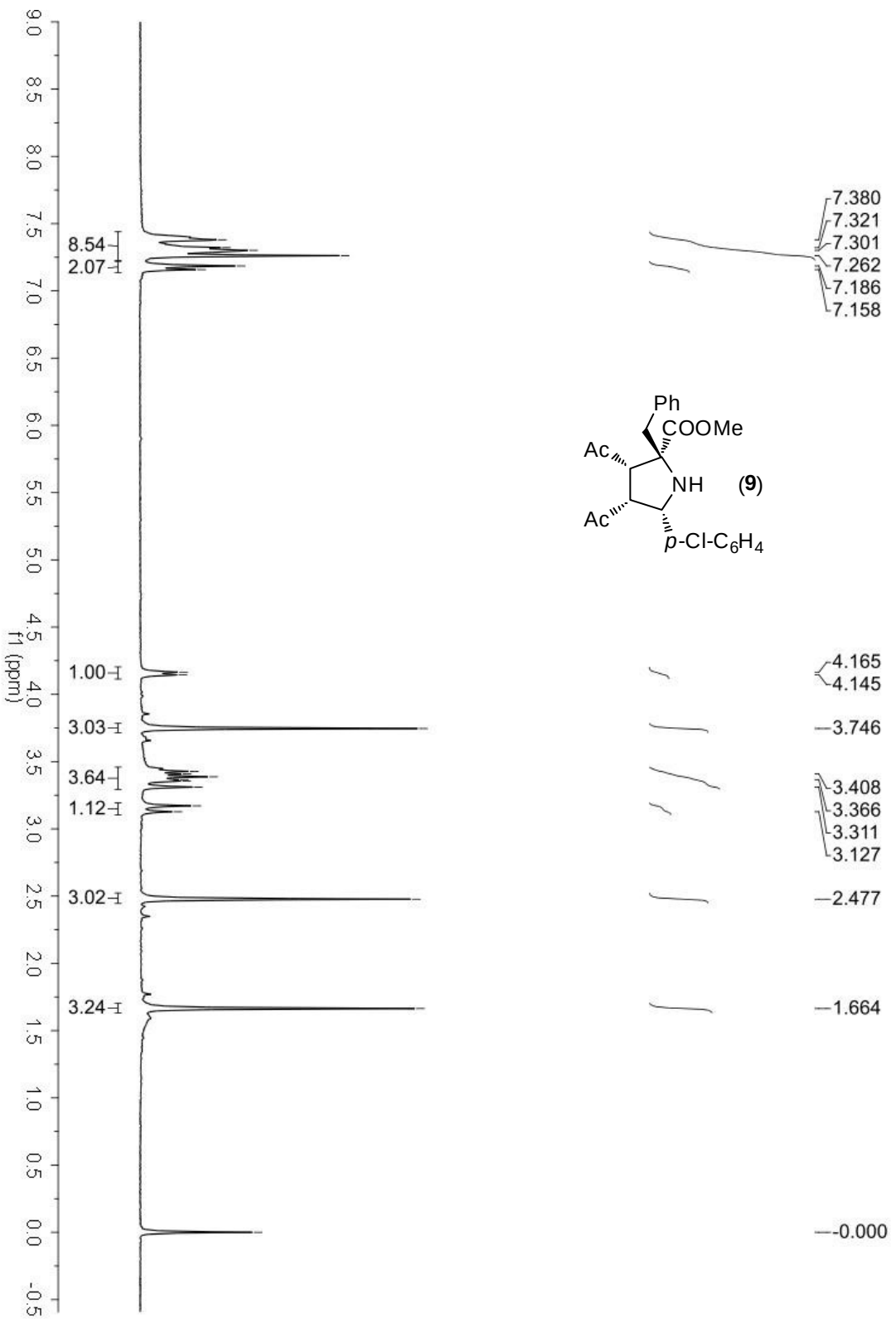

Ph
OH
COOMe
NH
Ph
OH

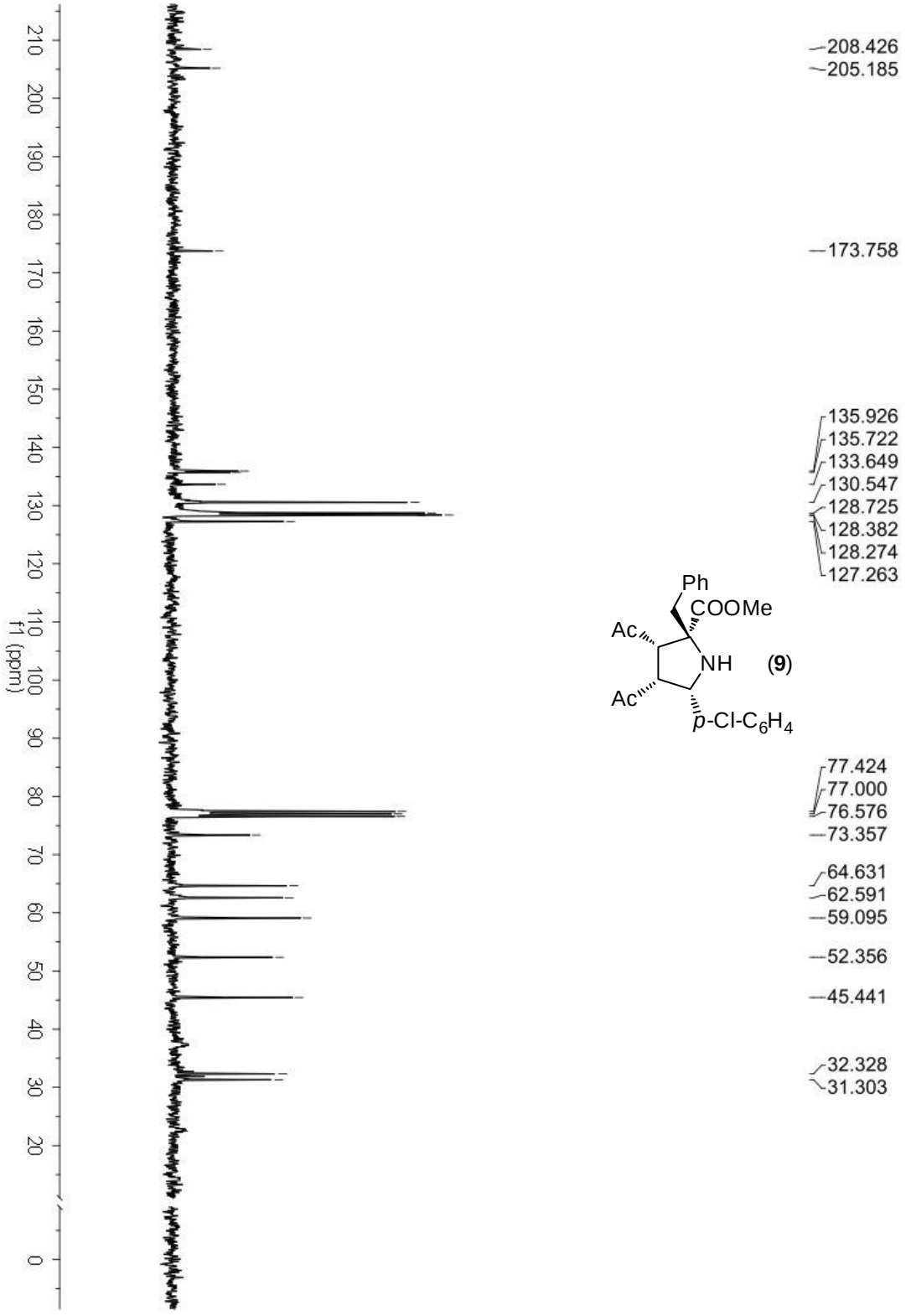
(**7**)


Archive directory: /export/home/wu/vnmrsys/data
Sample directory:
File: CARBON
Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
Mercury-300BB "Mercury300"
Relax. delay 1.000 sec
Pulse 28.0 degrees
Acq. time 0.999 sec
Width 17669.1 Hz
296 repetitions
OBSERVE C13, 75.4552826 MHz
DECOUPLE H1, 300.0820522 MHz
Power 40 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 4.0 Hz
FT size 32768
Total time 2 hr, 24 min, 19 sec
184.719
183.817
172.506
148.140
143.845
140.795
136.758
136.600
135.412
130.172
128.425
128.211
127.924
127.538
127.108
77.401
76.971
76.556
75.282
66.649
52.876
41.853
220
200
180
160
140
120
100
80
60
40
20
ppm

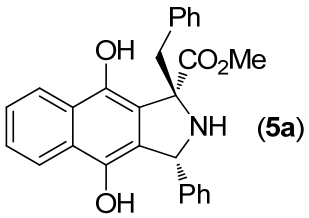
Ph
OH
COOMe
NH
Ph
OH

(**7**)


7.380
7.321
7.301
7.262
7.186
7.158
Ph
COOMe
Ac
NH
(9)
Ac
p-Cl-C6H4
8.54
2.07
1.00
3.03
3.64
1.12
3.02
3.24
f1 (ppm)
4.165
4.145
3.746
3.408
3.366
3.311
3.127
2.477
1.664
-0.000


208.426
205.185
173.758
135.926
135.722
133.649
130.547
128.725
128.382
128.274
127.263
77.424
77.000
76.576
73.357
64.631
62.591
59.095
52.356
45.441
32.328
31.303
f1 (ppm)
Ph
COOMe
Ac
NH
Ac
p-Cl-C6H4
(9)

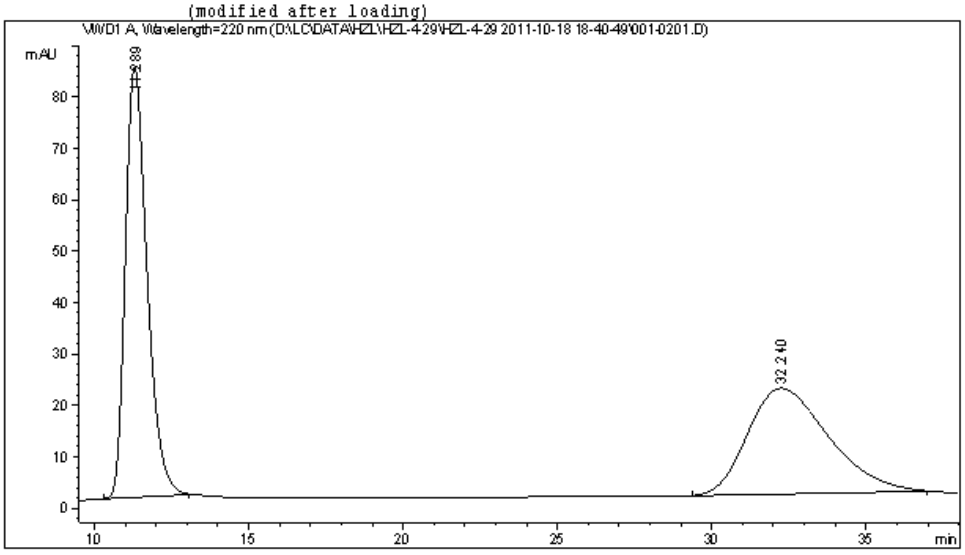
IX. HPLC Chromatograms



(**5a**)

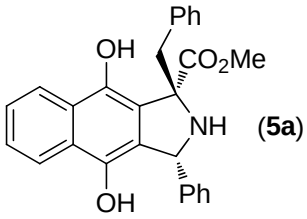
Data File D:\LC\DATA\HZL\HZL-4-29\HZL-4-29 2011-10-18 18-40-49\001-0201.D
Sample Name: HZL-4-29

```
    ====================================================================
    Acq. Operator   : HZL                          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\HZL\Data\HZL-4-29\HZL-4-29 2011-10-18 18-40-49\ASH-20-80-10ML-220NM.
                      M
    Last changed    : 8/29/2011 3:56:33 PM by HZL
    Analysis Method : D:\LC\DATA\HZL\HZL-4-29\HZL-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: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s    [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       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


    ====================================================================
```

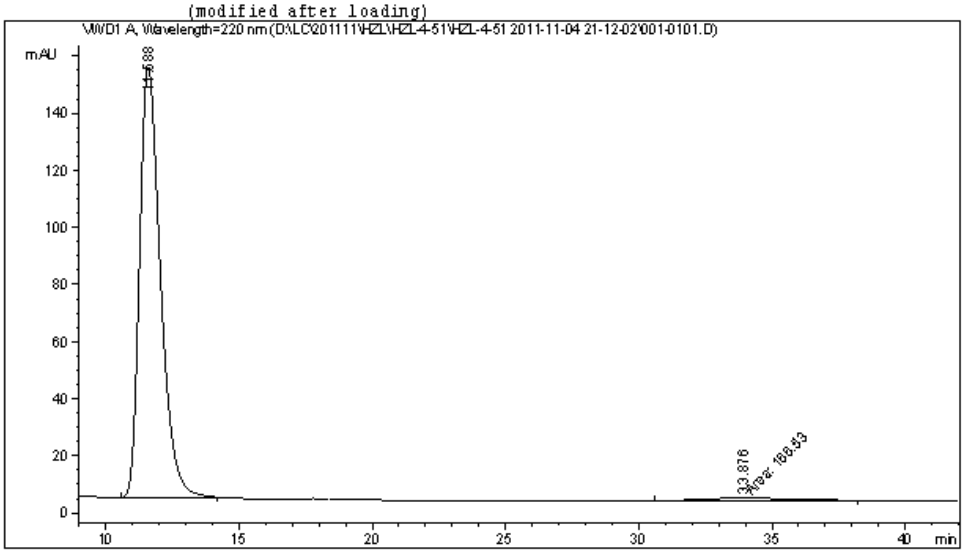
Instrument 1 9/24/2012 9:57:19 AM FX Page 1 of 1



(**5a**)

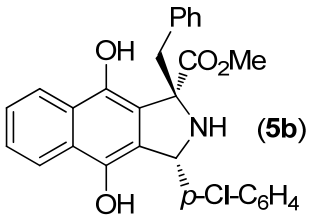
Data File D:\LC\201111\HZL\HZL-4-51\HZL-4-51 2011-11-04 21-12-02\001-0101.D
Sample Name: HZL-4-51B

```
    ====================================================================
    Acq. Operator   : HZL                          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\HZL\HZL-4-51\HZL-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 HZL
    Analysis Method : D:\LC\201111\HZL\HZL-4-51\HZL-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: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       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

    ====================================================================
```

Instrument 1 12/12/2011 4:39:09 PM yl Page 1 of 1



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

```
    ====================================================================
    Acq. Operator   : HZL                          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\HZL\HZL-4-21\HZL-4-21C 2011-10-20 20-05-18\ASH-40-60-10ML-
                      220NM.M
    Last changed    : 10/20/2011 8:03:32 PM by HZL
    Analysis Method : D:\LC\201111\HZL\HZL-4-21\HZL-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 HZL
                      (modified after loading)
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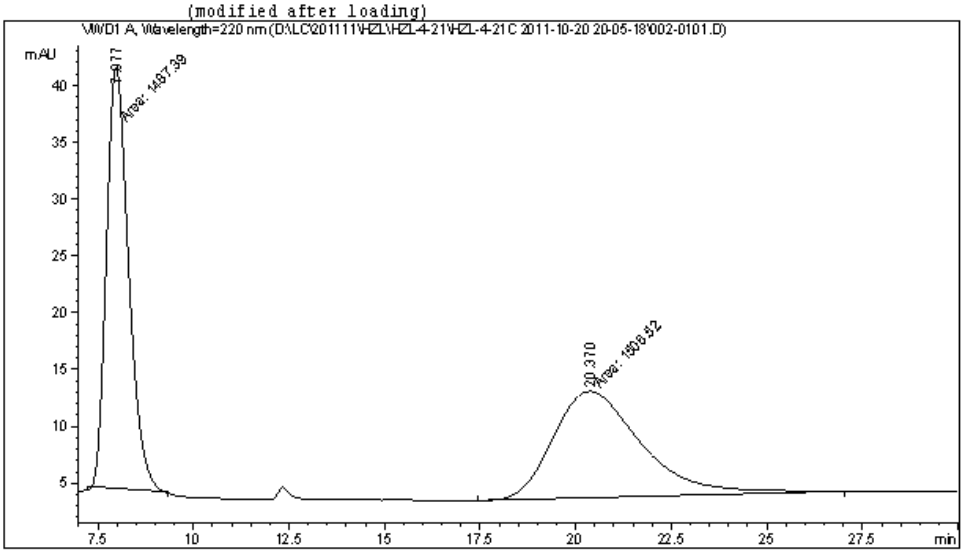



```
    ====================================================================
                            Area Percent Report
    ====================================================================

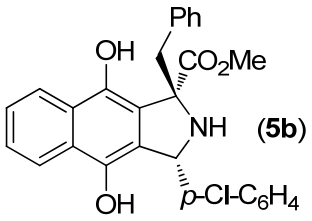
    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

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

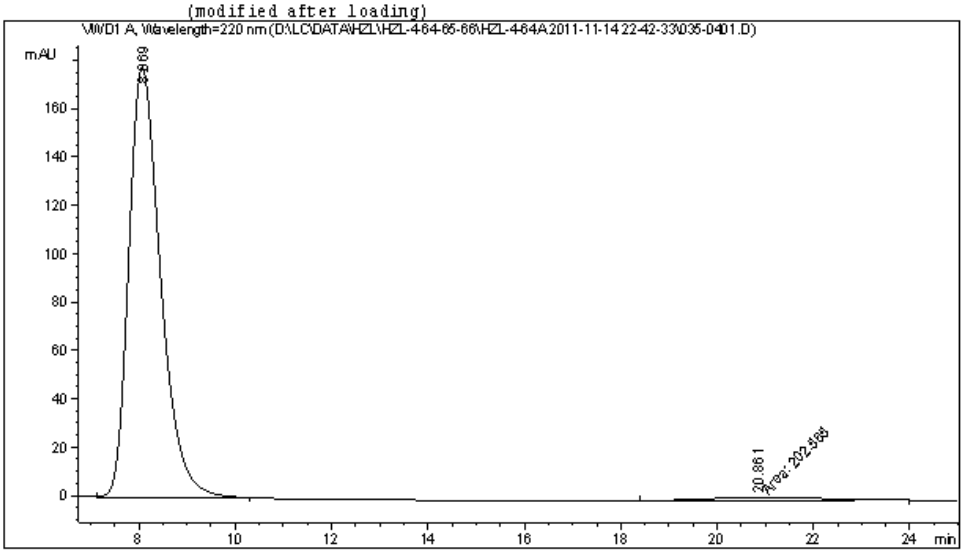
    Totals :                  2993.90796   46.39268

    ====================================================================
```



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

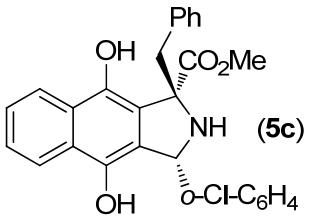


Area Percent Report

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

Signal 1: VWD1 A, Wavelength=220 nm

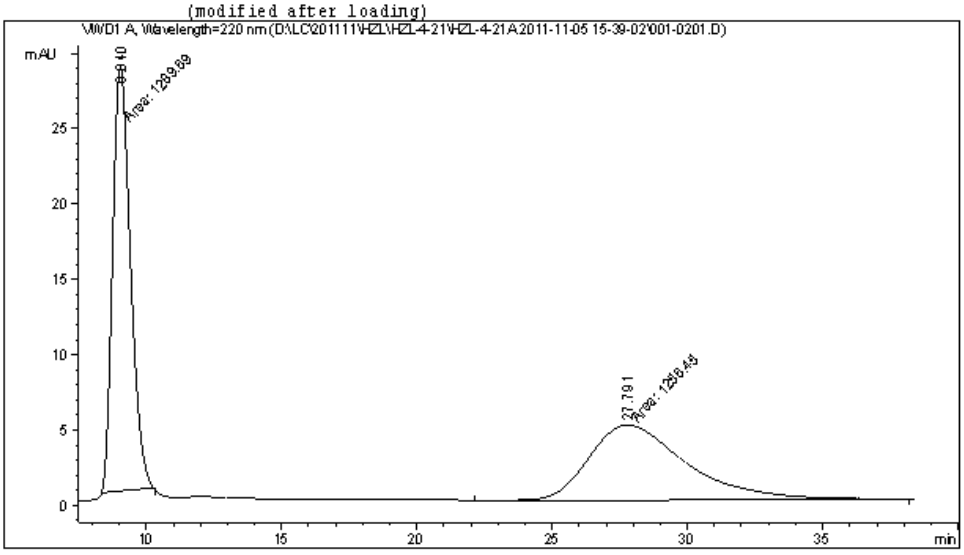
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	8.069	BB	0.7045	8175.20117	178.06204	97.5821
2	20.861	MM	2.6940	202.56479	1.25320	2.4179
Totals :				8377.76596	179.31524	



(5c)

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



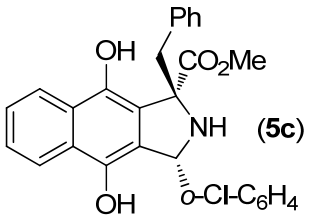
Area Percent Report

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	

Instrument 1 11/5/2011 4:34:27 PM HZL



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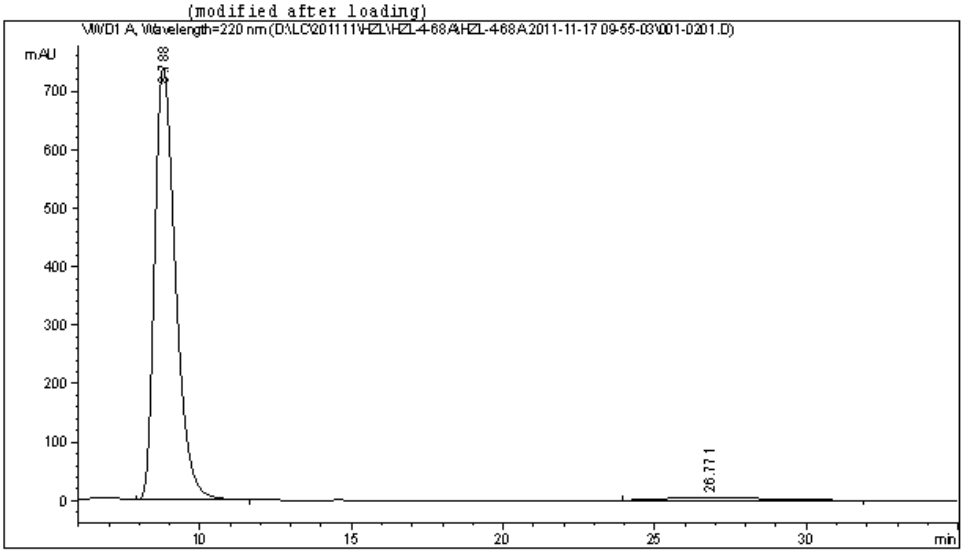
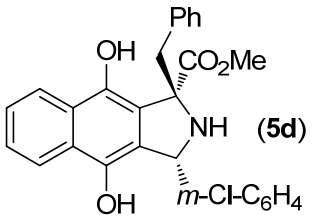


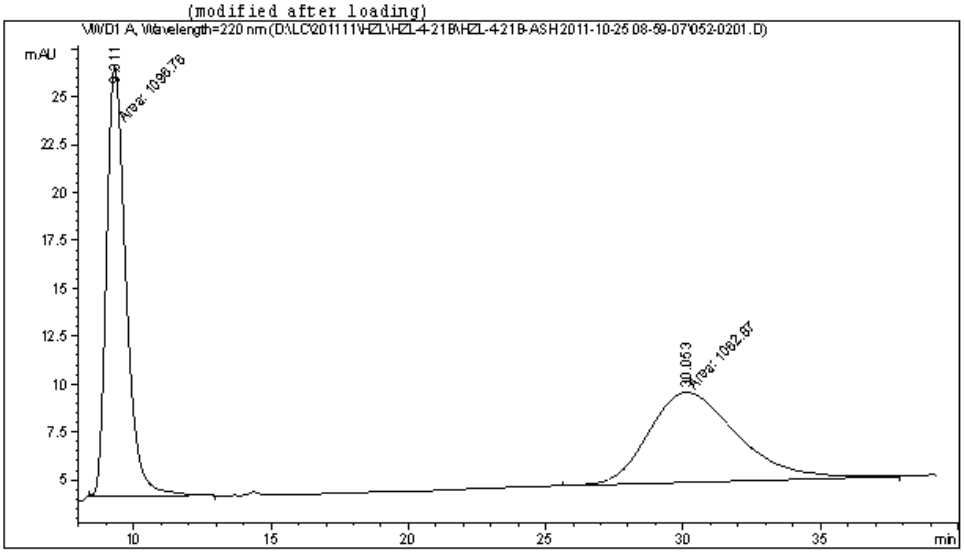

```
    ====================================================================
                            Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|------|----|-------|----------|----------|--------|
       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

    ====================================================================
```

Instrument 1 11/17/2011 11:07:50 AM hzl Page 1 of 1

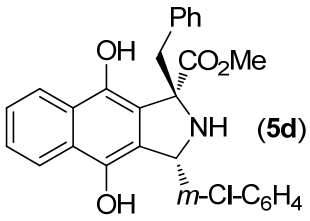


Data File D:\LC\201111\HZL\HZL-4-21B\HZL-4-21B-ASH 2011-10-25 08-59-07\052-0201.D
Sample Name: HZL-4-21B

```
    ====================================================================
    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\HZL\HZL-4-21B\HZL-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\HZL\HZL-4-21B\HZL-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)
```



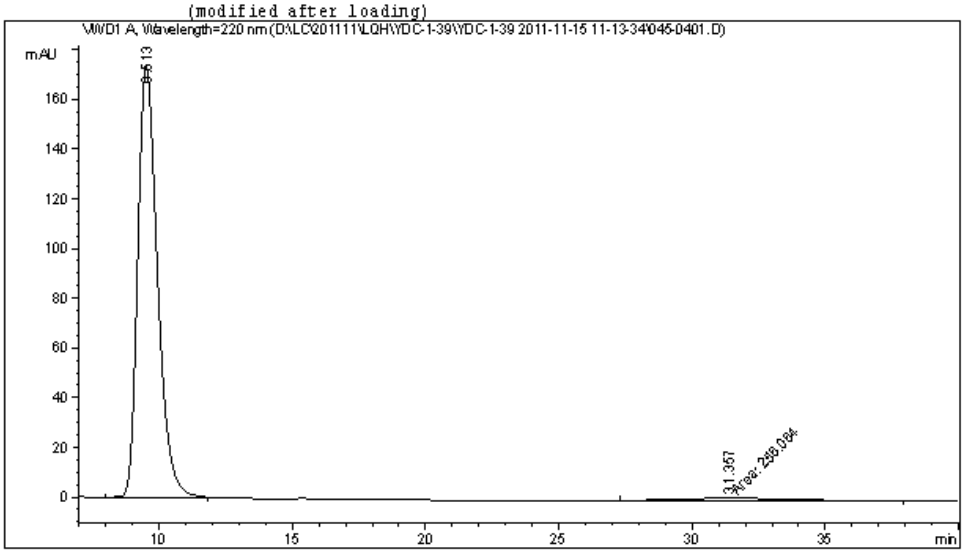

```
    ====================================================================
                            Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|------|----|-------|----------|----------|--------|
       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

    ====================================================================
```



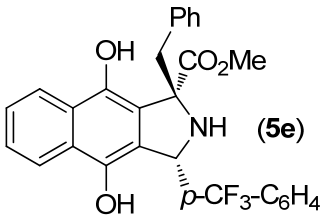
(5d)

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: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|-------|
       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\HZL\HZL-4-71\HZL-4-71B-ADH 2011-11-18 10-07-52\001-0201.D
Sample Name: HZL-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\HZL\HZL-4-71\HZL-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\HZL\HZL-4-71\HZL-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)
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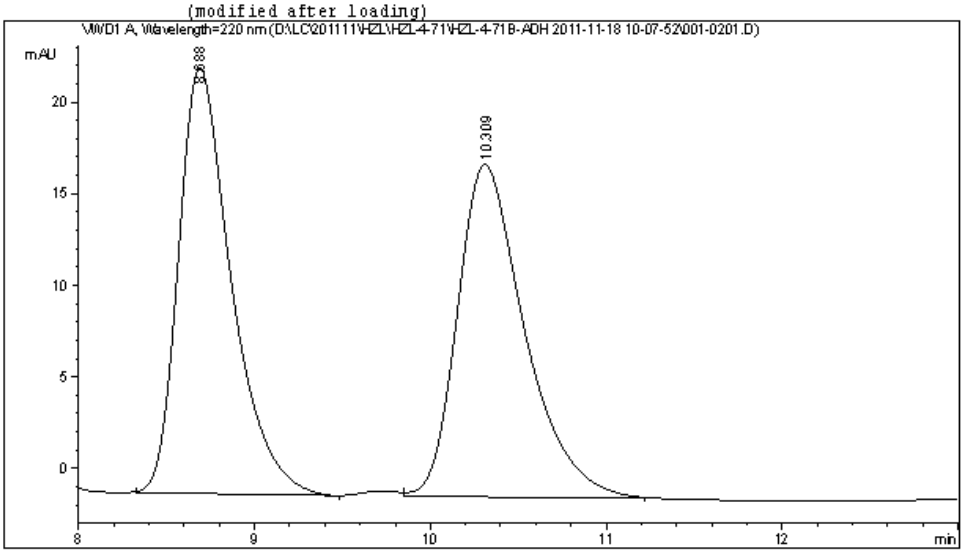



```
    ====================================================================
                            Area Percent Report
    ====================================================================

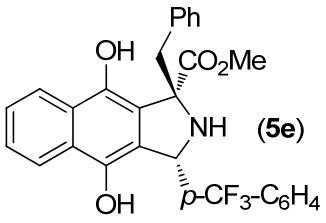
    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|------|----|-------|----------|----------|--------|
       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

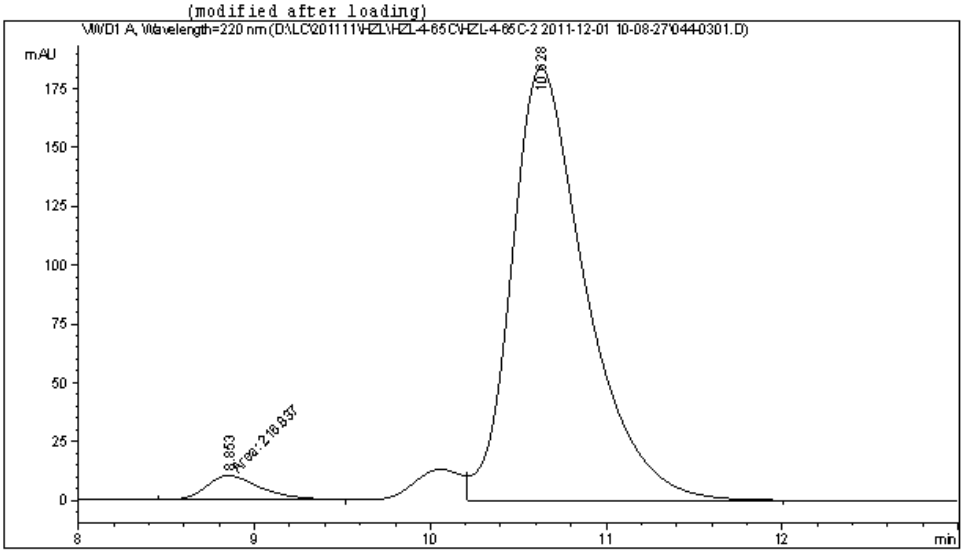
    ====================================================================
```



(5e)

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

```
    ====================================================================
    Acq. Operator   : HZL                          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\HZL\HZL-4-65C\HZL-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\HZL\HZL-4-65C\HZL-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

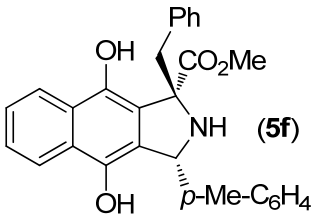

    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|------|----|-------|----------|----------|-------|
       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

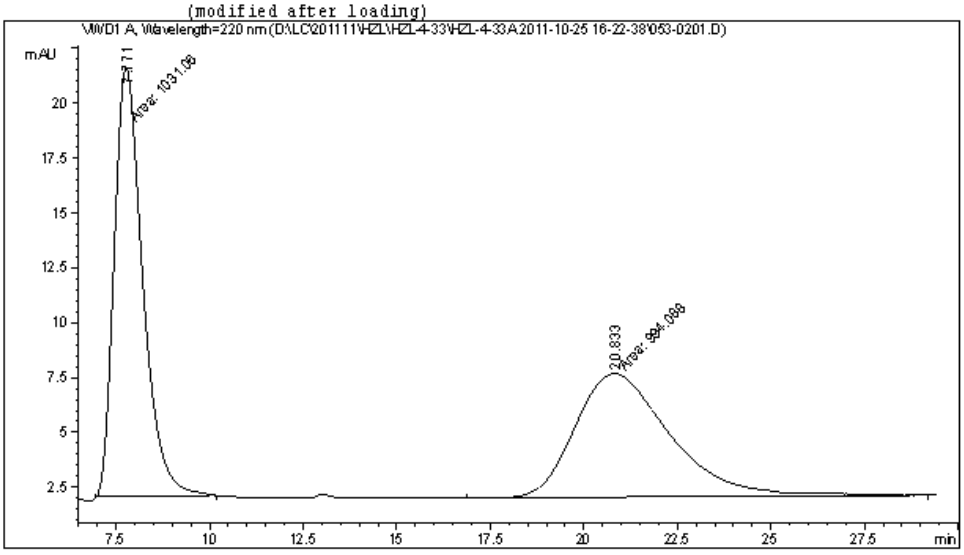
    ====================================================================
```

Instrument 1 12/15/2011 4:49:56 PM FX Page 1 of 1



Data File D:\LC\201111\HZL\HZL-4-33\HZL-4-33A 2011-10-25 16-22-38\053-0201.D
Sample Name: HZL-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\HZL\HZL-4-33\HZL-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\HZL\HZL-4-33\HZL-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 HZL (modified after loading)		

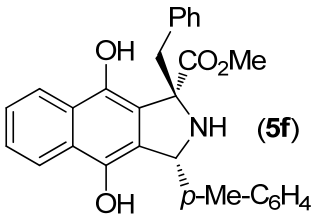


Area Percent Report

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

Signal 1: VWD1 A, Wavelength=220 nm

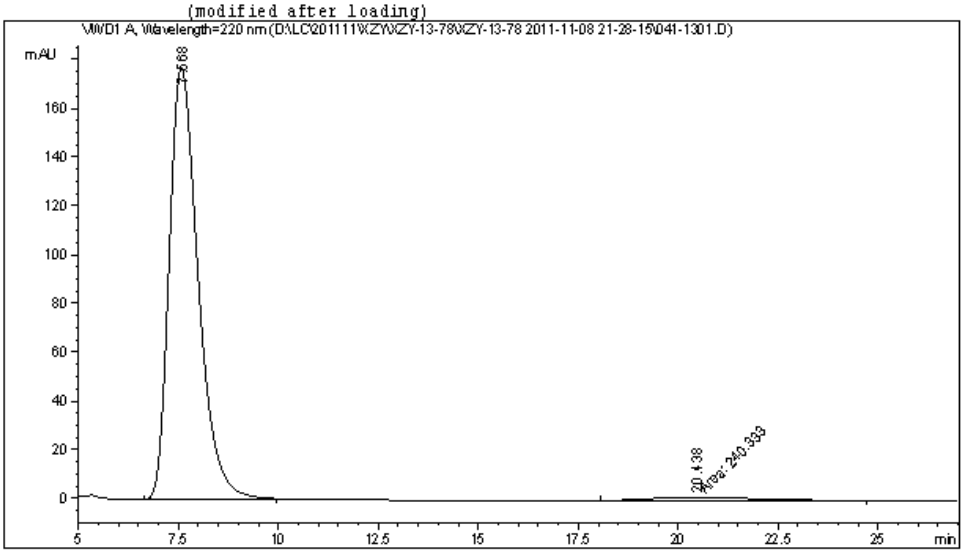
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.771	MM	0.8797	1031.05908	19.53331	50.9128
2	20.833	MM	2.9494	994.08826	5.61755	49.0872
Totals :				2025.14734	25.15086	



(5f)

Data File D:\LC\201111\XZY\XZY-13-78\XZY-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\XZY\XZY-13-78\XZY-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\XZY\XZY-13-78\XZY-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

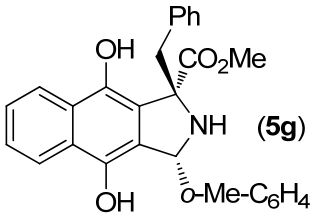

    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|------|----|-------|----------|----------|-------|
       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

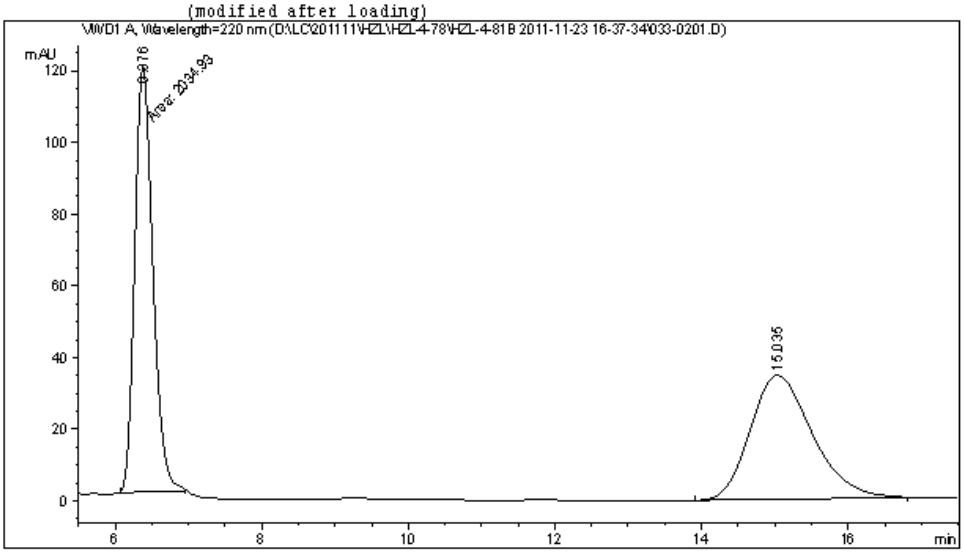
    ==================================================================
```

Instrument 1 11/9/2011 2:42:06 PM THL Page 1 of 1



Data File D:\LC\201111\HZL\HZL-4-78\HZL-4-81B 2011-11-23 16-37-34\033-0201.D
Sample Name: HZL-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\HZL\HZL-4-78\HZL-4-81B 2011-11-23 16-37-34\ODH-30-70-10ML-220NM.M
Last changed	: 9/14/2011 10:11:46 AM by HZL
Analysis Method	: D:\LC\201111\HZL\HZL-4-78\HZL-4-81B 2011-11-23 16-37-34\033-0201.D\DA.M (ODH-30-70-10ML-220NM.M)
Last changed	: 12/15/2011 10:07:15 AM by FX (modified after loading)



Area Percent Report

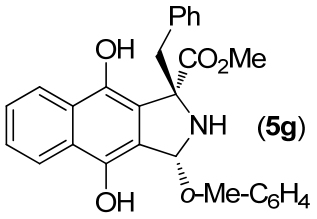
Sorted By : Signal
Multiplier : 1.0000
Dilution : 1.0000
Use Multiplier & Dilution Factor with ISTDs

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	

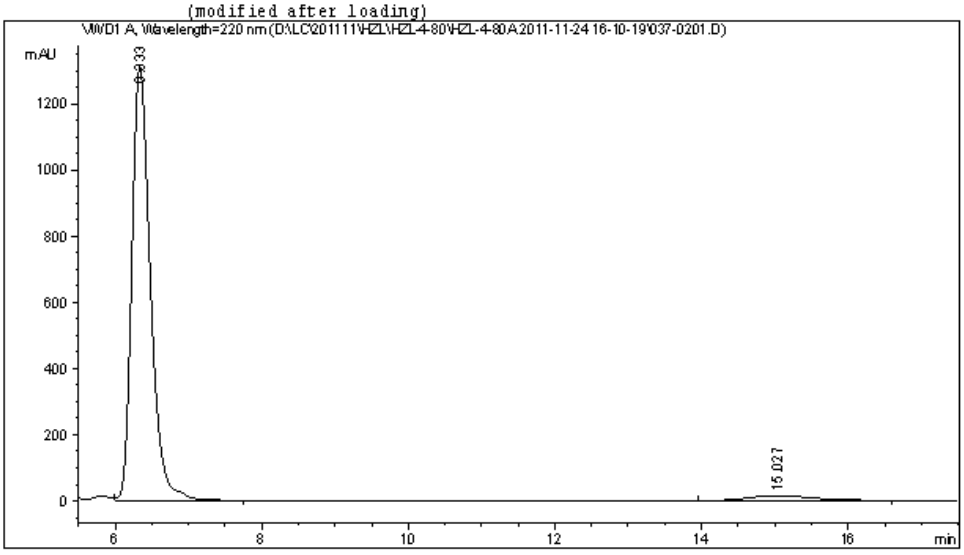
Instrument 1 12/15/2011 10:07:29 AM FX

Page 1 of 1



Data File D:\LC\201111\HZL\HZL-4-80\HZL-4-80A 2011-11-24 16-10-19\037-0201.D
Sample Name: HZL-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\HZL\HZL-4-80\HZL-4-80A 2011-11-24 16-10-19\ODH-30-70-10ML-
                  220NM-20MIN.M
Last changed    : 11/24/2011 3:51:31 PM by HZL
Analysis Method : D:\LC\201111\HZL\HZL-4-80\HZL-4-80A 2011-11-24 16-10-19\037-0201.D\DA.M (
                  ODH-30-70-10ML-220NM-20MIN.M)
Last changed    : 12/15/2011 10:09:25 AM by FX
                  (modified after loading)
```




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

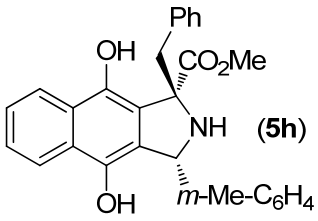
Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs


Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|------|----|-------|----------|----------|-------|
   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

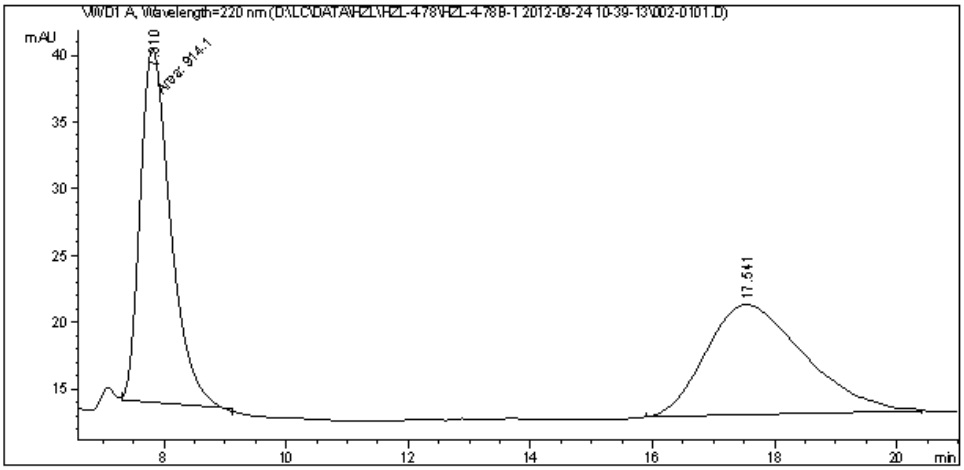
==================================================================
```



(5h)

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

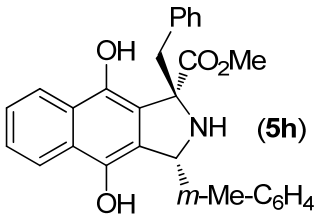

    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       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

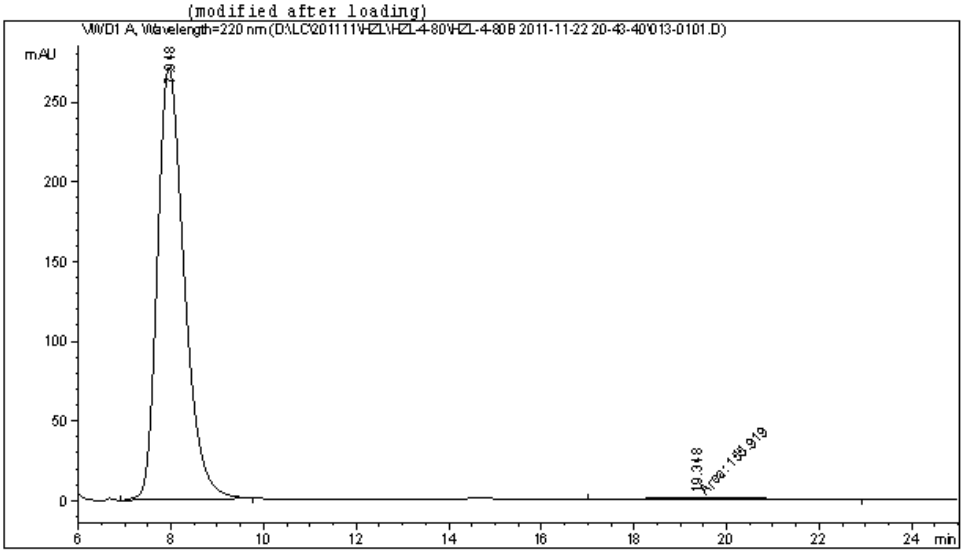

    ==================================================================
                            *** End of Report ***
```

Instrument 1 9/24/2012 11:21:11 AM FX Page 1 of 1



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

Acq. Operator	: HZL	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\HZL\HZL-4-80\HZL-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\HZL\HZL-4-80\HZL-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)		

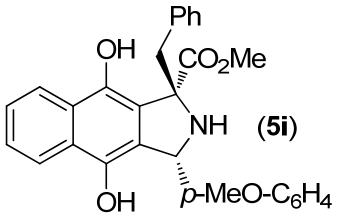


Area Percent Report

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

Signal 1: VWD1 A, Wavelength=220 nm

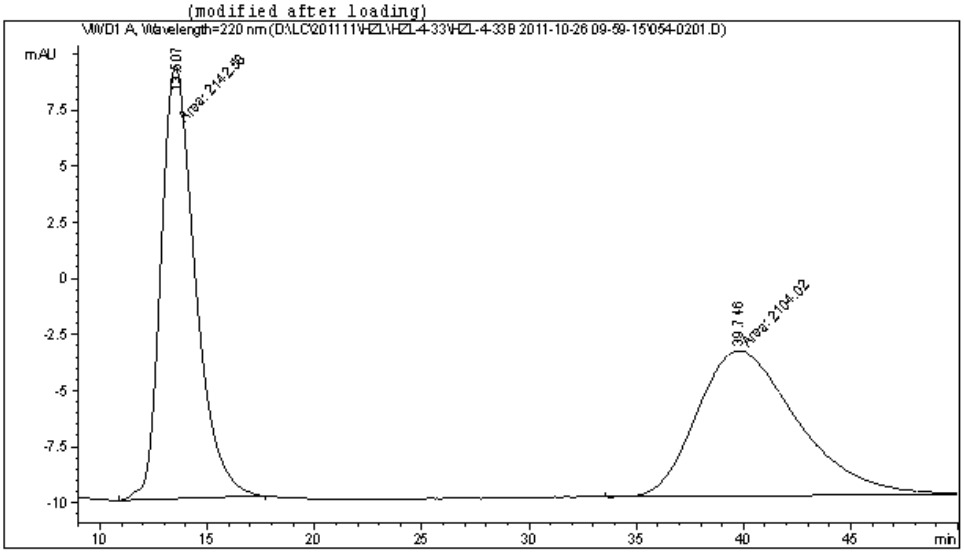
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	7.948	VB	0.6202	1.09292e4	271.14075	98.5934
2	19.348	MM	2.3333	155.91867	1.11373	1.4066
Totals :				1.10851e4	272.25447	



(5i)

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

Acq. Operator	: HZL	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\HZL\HZL-4-33\HZL-4-33B 2011-10-26 09-59-15\ASH-30-70-12ML-220NM.M		
Last changed	: 10/25/2011 4:20:13 PM by hzl		
Analysis Method	: D:\LC\201111\HZL\HZL-4-33\HZL-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 HZL (modified after loading)		

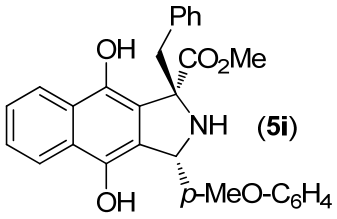


Area Percent Report

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	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	



(5i)

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

```
==================================================================
Acq. Operator   : HZL                          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\HZL\HZL-4-59\HZL-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 HZL
Analysis Method : D:\LC\201111\HZL\HZL-4-59\HZL-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)
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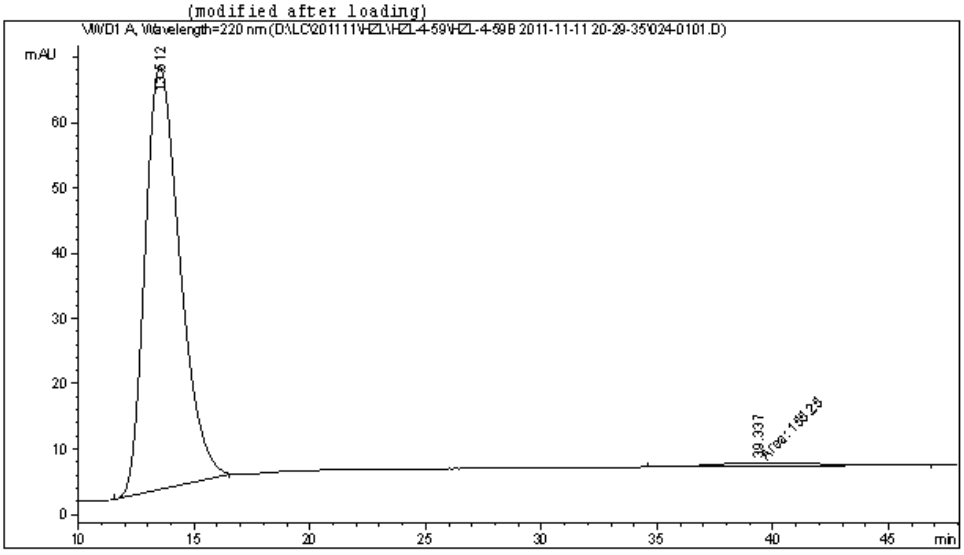
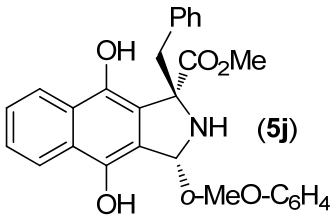



```
==================================================================
                        Area Percent Report
==================================================================

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


Signal 1: VWD1 A, Wavelength=220 nm

Peak RetTime Type  Width     Area      Height     Area
  #   [min]        [min]  mAU   *s   [mAU   ]      %
----|------|----|-------|----------|----------|-------|
   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

==================================================================
```

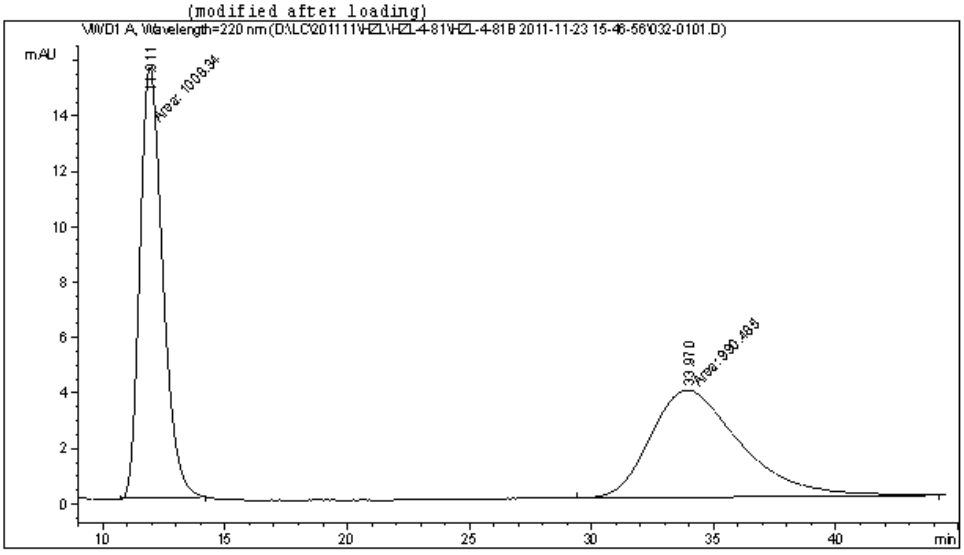
Instrument 1 11/11/2011 9:30:38 PM THL Page 1 of 1



(5j)

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

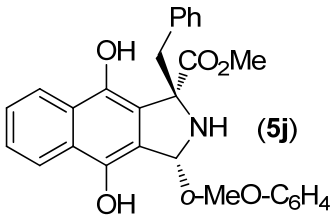


Area Percent Report

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

Signal 1: VWD1 A, Wavelength=220 nm

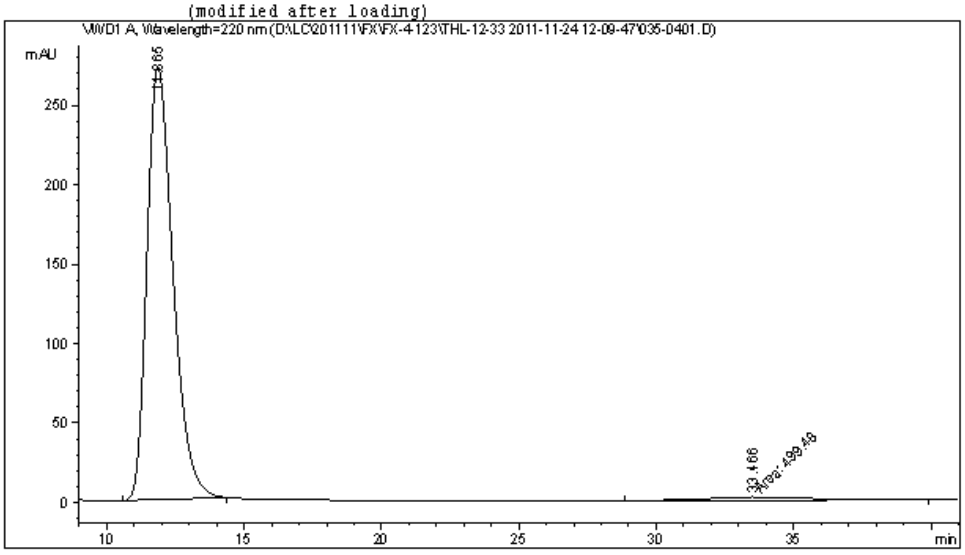
Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	11.911	MM	1.0821	1008.33783	15.53055	50.4466
2	33.970	MM	4.2712	990.48456	3.86496	49.5534
Totals :				1998.82239	19.39551	



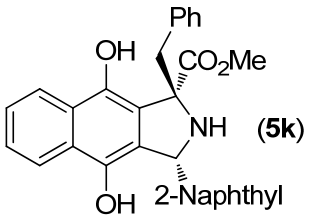
(5j)

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)
```

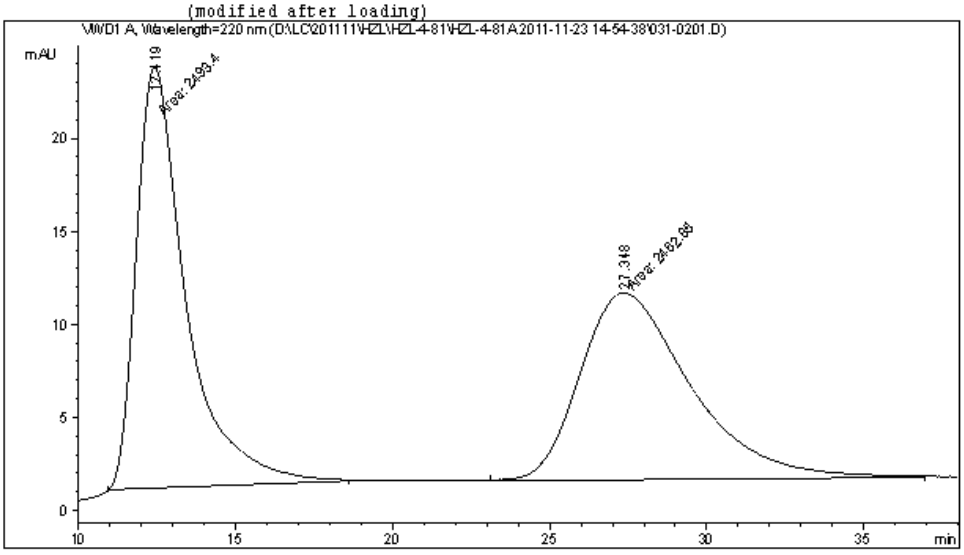



```
    ====================================================================
                            Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       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\HZL\HZL-4-81\HZL-4-81A 2011-11-23 14-54-38\031-0201.D
Sample Name: HZL-4-81A

Acq. Operator	: HZL	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\HZL\HZL-4-81\HZL-4-81A 2011-11-23 14-54-38\ASH-30-70-10ML-220NM.M		
Last changed	: 10/20/2011 6:42:48 PM by HZL		
Analysis Method	: D:\LC\201111\HZL\HZL-4-81\HZL-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 hzl (modified after loading)		

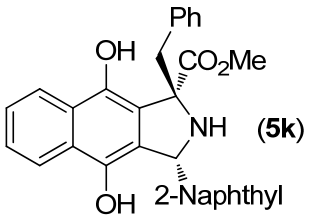


Area Percent Report

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	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)
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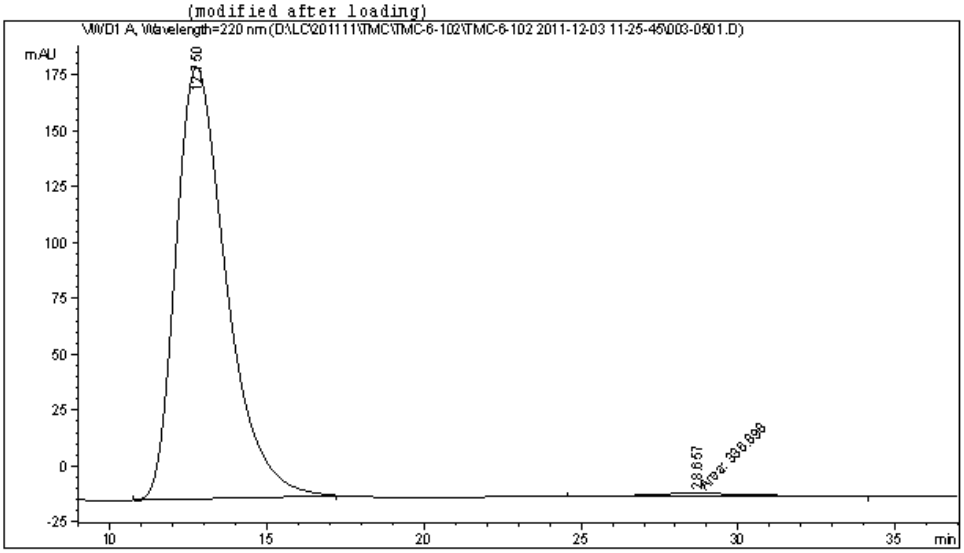



```
    ====================================================================
                            Area Percent Report
    ====================================================================

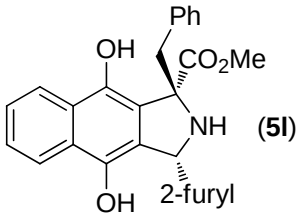
    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|------|----|-------|----------|----------|--------|
       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

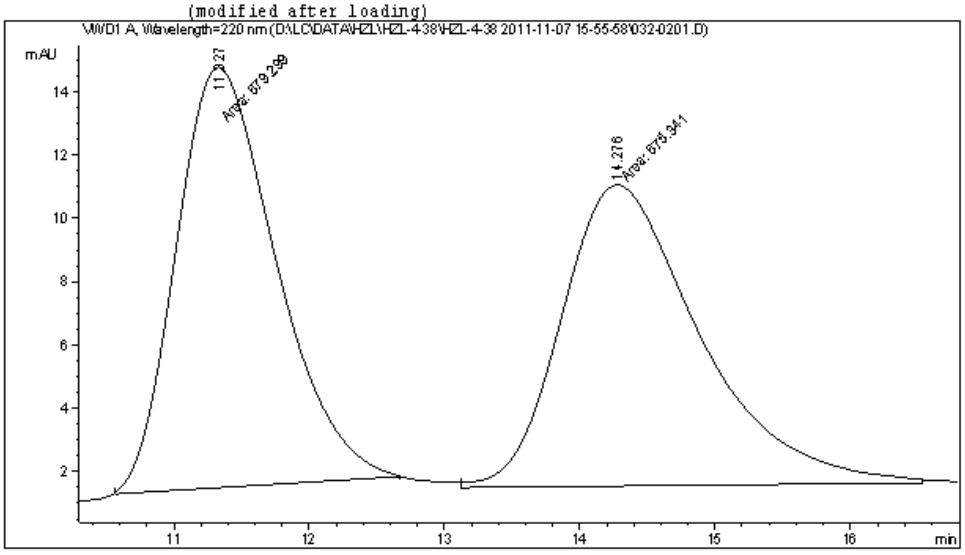
    ====================================================================
```



(**5l**)

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

Acq. Operator	: HZL	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\HZL\HZL-4-38\HZL-4-38 2011-11-07 15-55-58\ASH-30-70-10ML-220NM.M		
Last changed	: 10/20/2011 6:42:48 PM by HZL		
Analysis Method	: D:\LC\DATA\HZL\HZL-4-38\HZL-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)		

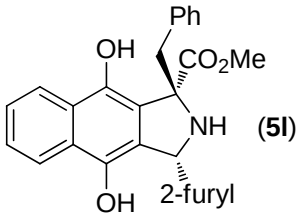


Area Percent Report

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

Signal 1: VWD1 A, Wavelength=220 nm

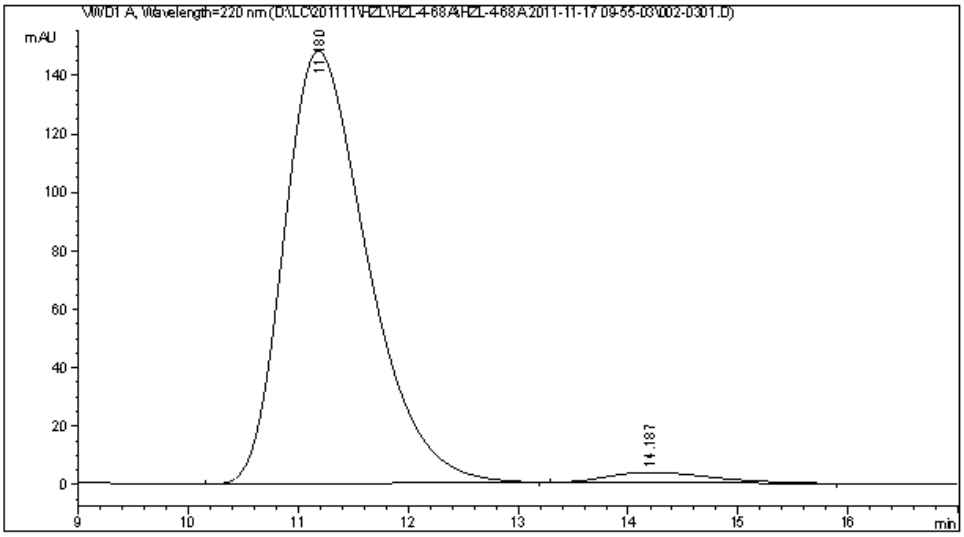
Peak #	RetTime [min]	Type	Width [min]	Area mAU*s	Height [mAU]	Area %
1	11.327	MM	0.8527	679.29883	13.27685	50.1461
2	14.276	MM	1.1835	675.34106	9.51031	49.8539
Totals :				1354.63989	22.78717	



(**5l**)

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



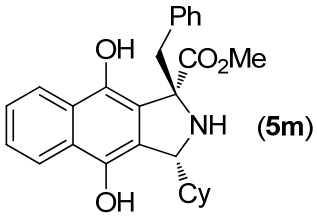

```
    ====================================================================
                           Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=220 nm

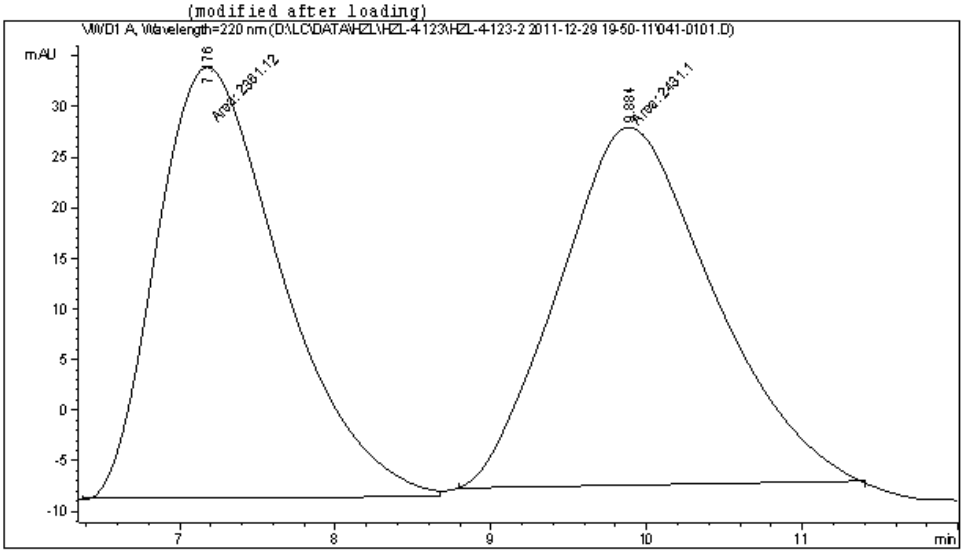
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  11.180 VB    0.8114 7800.07568  148.01219  97.0599
       2  14.187 BB    0.8576  236.27974    3.60615   2.9401

    Totals :                  8036.35542  151.61835
```



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

```
    ====================================================================
    Acq. Operator   : HZL                          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\HZL\HZL-4-123\HZL-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 HZL
    Analysis Method : D:\LC\DATA\HZL\HZL-4-123\HZL-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
    ====================================================================

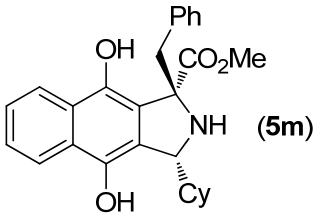
    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|------|----|-------|----------|----------|--------|
       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

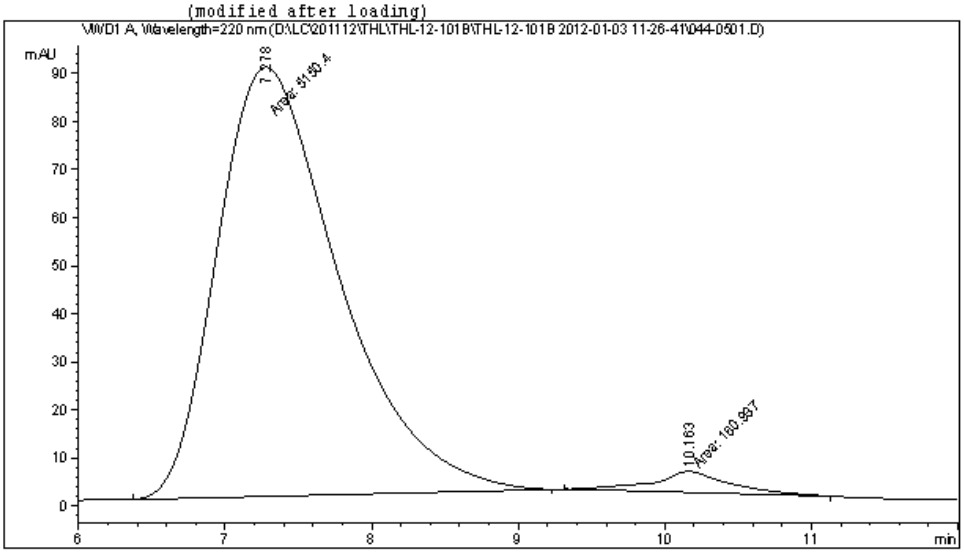
    ====================================================================
```



(5m)

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
    ==================================================================

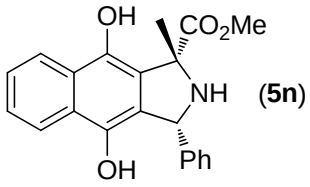
    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

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

    Totals :                  5311.33469   93.66722

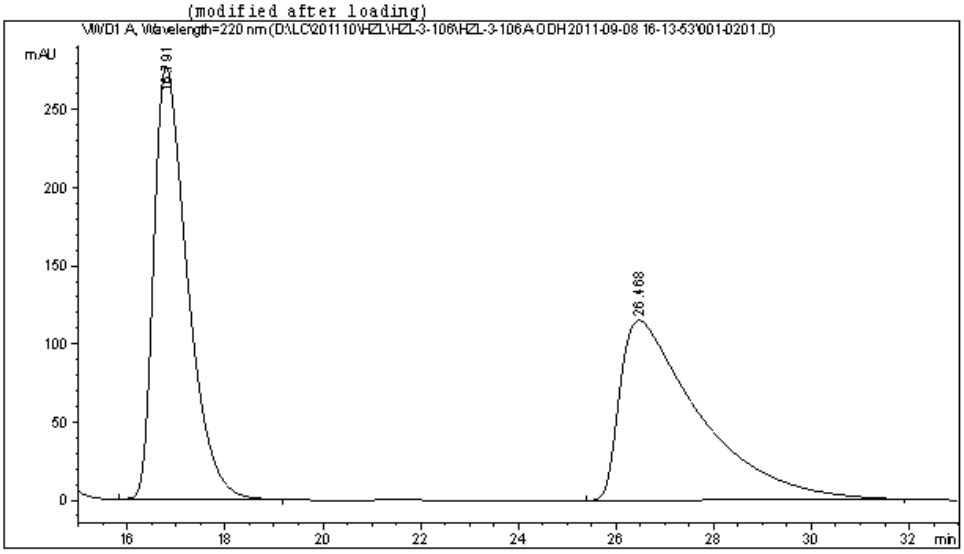
    ==================================================================
```



(**5n**)

Data File D:\LC\20111O\HZL\HZL-3-106\HZL-3-106A-ODH 2011-09-08 16-13-53\001-0201.D
Sample Name: HZL-3-106A

```
    ================================================================
    Acq. Operator   : HZL                          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\HZL\Data\HZL-3-106\HZL-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 HZL
    Analysis Method : D:\LC\20111O\HZL\HZL-3-106\HZL-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

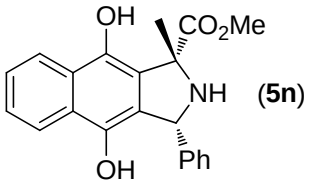
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|------|----|-------|----------|----------|--------|
       1  16.791 BB    0.7278 1.32454e4   276.51071  50.4540
       2  26.468 BB    1.6016 1.30070e4   115.03537  49.5460

    Totals :                  2.62525e4   391.54608

    ================================================================
```

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

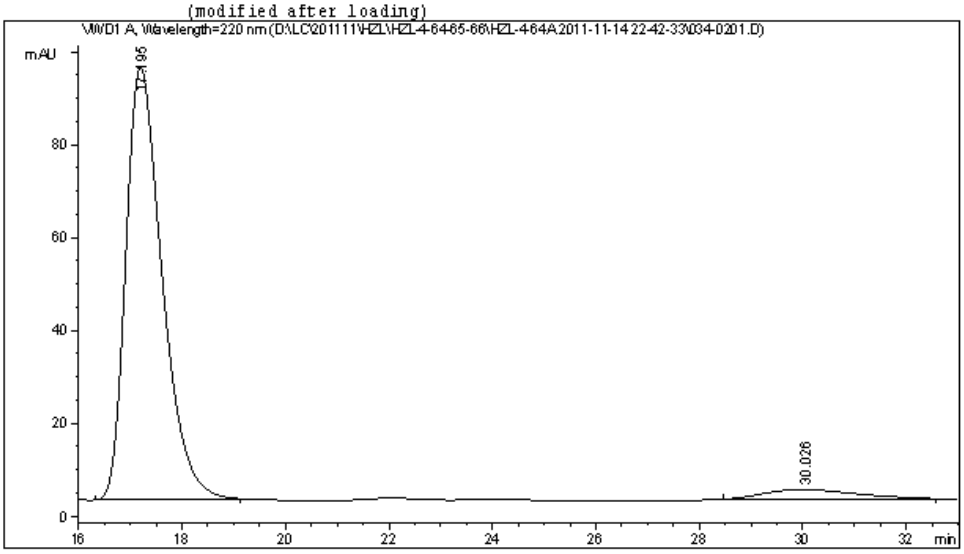
Page 1 of 1



(**5n**)

Data File D:\LC\201111\HZL\HZL-4-64-65-66\HZL-4-64A 2011-11-14 22-42-33\034-0201.D
Sample Name: HZL-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\HZL\HZL-4-64-65-66\HZL-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\HZL\HZL-4-64-65-66\HZL-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: VWD1 A, Wavelength=220 nm

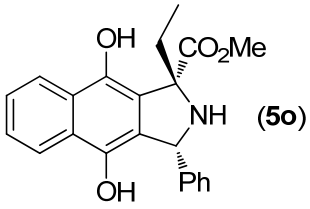
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|------|----|-------|----------|----------|--------|
       1  17.195 BB    0.7464 4539.73438   93.12305  94.5845
       2  30.026 BB    1.4737  259.92557    2.07204   5.4155

    Totals :                  4799.65994   95.19509

    ==================================================================
```

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

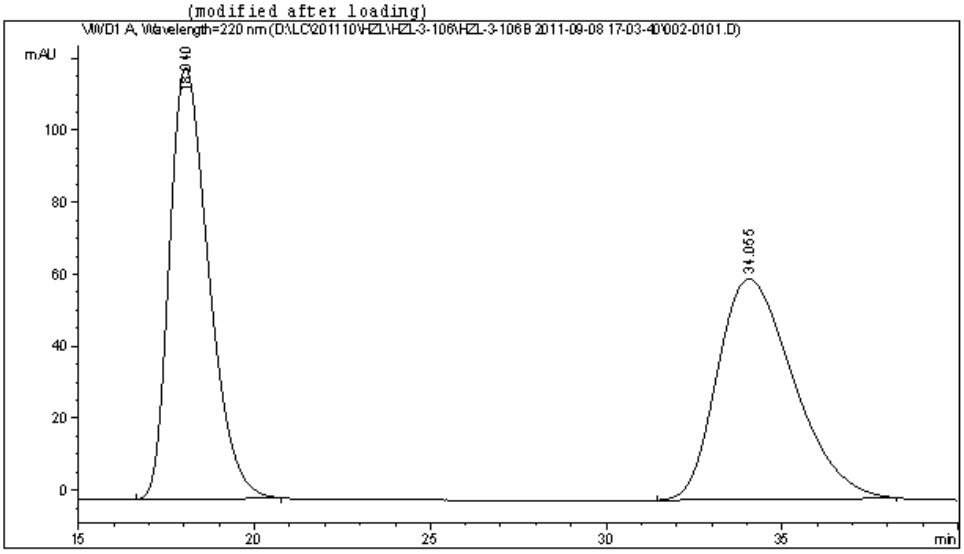
Page 1 of 1



(**5o**)

Data File D:\LC\20111O\HZL\HZL-3-106\HZL-3-106B 2011-09-08 17-03-40\002-0101.D
Sample Name: HZL-3-106B

```
    ================================================================
    Acq. Operator   : HZL                          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\HZL\Data\HZL-3-106\HZL-3-106B 2011-09-08 17-03-40\ASH-20-80-10ML-
                      220NM.M
    Last changed    : 8/29/2011 3:56:33 PM by HZL
    Analysis Method : D:\LC\20111O\HZL\HZL-3-106\HZL-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
    ================================================================

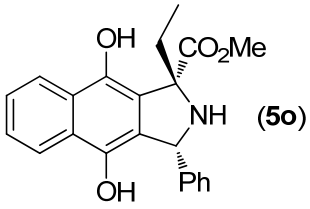
    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  18.040 BB    1.1901 9286.13867  119.93852  50.2003
       2  34.055 BB    2.2053 9212.01758   61.32467  49.7997

    Totals :                  1.84982e4   181.26320

    ================================================================
```



(**5o**)

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

```
    ======================================================================
    Acq. Operator   : HZL                          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\HZL\HZL-4-70\HZL-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 HZL
    Analysis Method : D:\LC\201111\HZL\HZL-4-70\HZL-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 hzl
                      (modified after loading)
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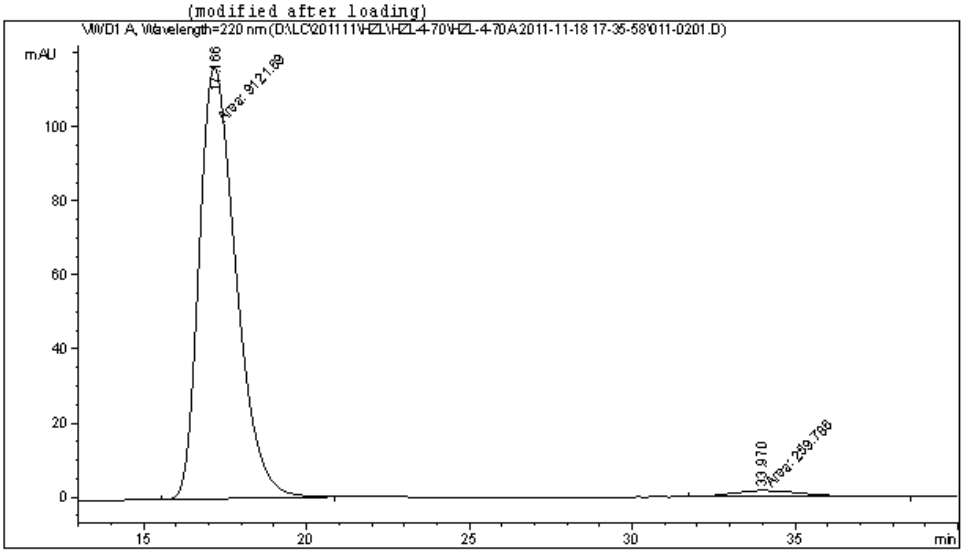
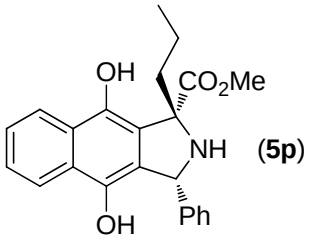



```
    ======================================================================
                            Area Percent Report
    ======================================================================

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


    Signal 1: VWD1 A, Wavelength=220 nm

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

    Totals :                  9381.47406  118.11255

    ======================================================================
```

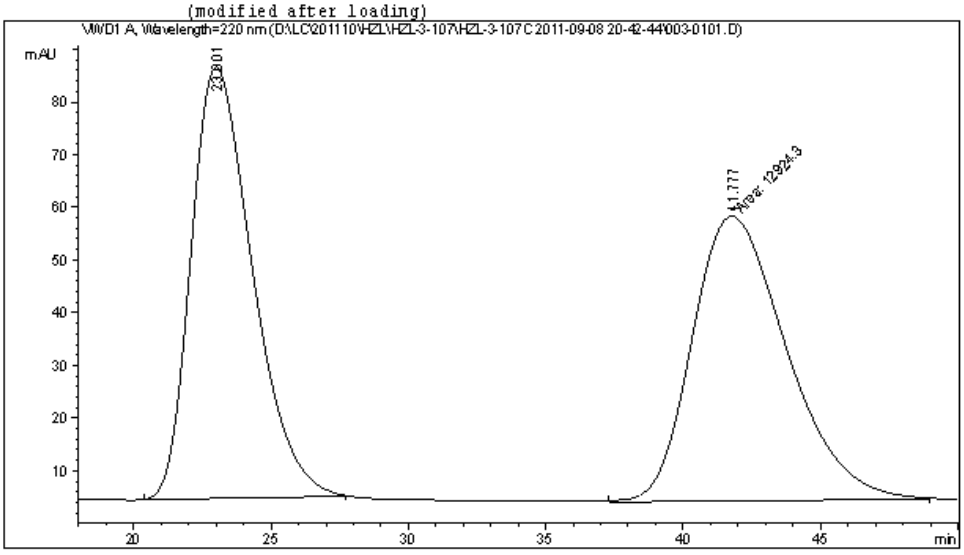
Instrument 1 11/18/2011 6:35:20 PM hzl



(**5p**)

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

Acq. Operator	: HZL	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\HZL\Data\HZL-3-107\HZL-3-107C 2011-09-08 20-42-44\ASH-20-80-10ML-220NM.M		
Last changed	: 8/29/2011 3:56:33 PM by HZL		
Analysis Method	: D:\LC\201110\HZL\HZL-3-107\HZL-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)		

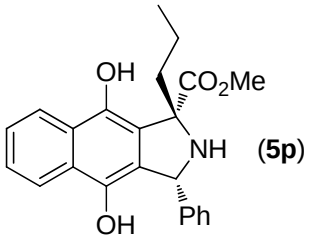


Area Percent Report

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

Signal 1: VWD1 A, Wavelength=220 nm

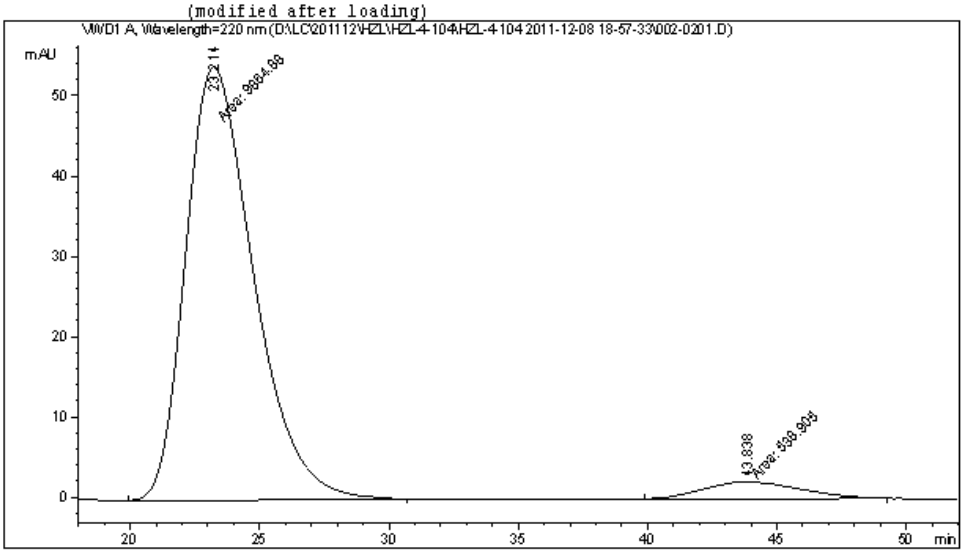
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	23.001	BB	2.3299	1.26838e4	81.81721	49.5305
2	41.777	MM	3.9866	1.29243e4	54.03196	50.4695
Totals :				2.56081e4	135.84917	



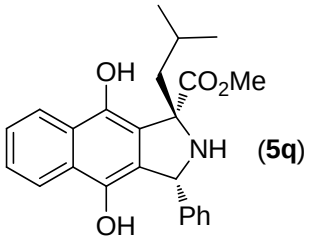
(**5p**)

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

```
    ====================================================================
    Acq. Operator   : HZL                          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\HZL\HZL-4-104\HZL-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\HZL\HZL-4-104\HZL-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)
```



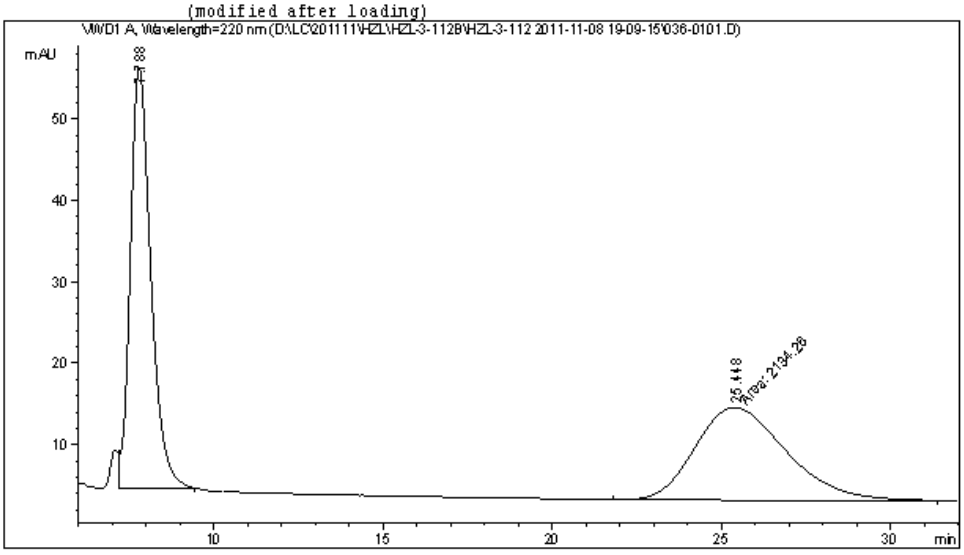

```
    ====================================================================
                            Area Percent Report
    ====================================================================

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


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|------|----|-------|----------|----------|--------|
       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

    ====================================================================
```



(**5q**)

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

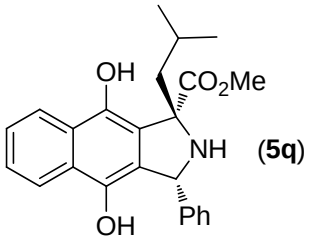
```
    ==================================================================
    Acq. Operator   : hzl                          Seq. Line :   1
    Acq. Instrument : Instrument 1                  Location : Vial 36
    Injection Date  : 11/8/2011 7:11:11 PM             Inj :   1
                                                   Inj Volume : 5 µl
    Acq. Method     : D:\LC\201111\HZL\HZL-3-112B\HZL-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\HZL\HZL-3-112B\HZL-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 Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|------|----|-------|----------|----------|--------|
       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

    ==================================================================
```

Instrument 1 11/8/2011 7:44:54 PM THL

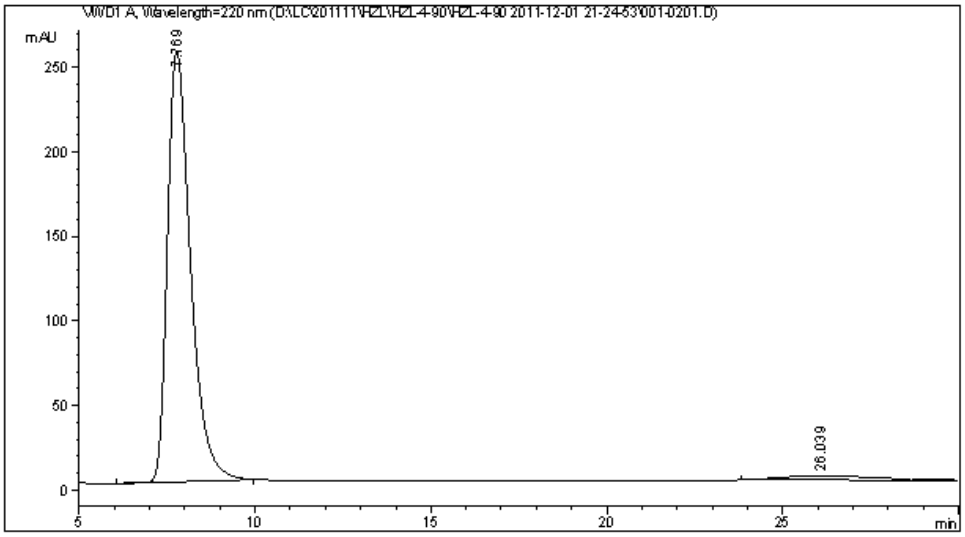
Page 1 of 1



(**5q**)

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

Acq. Operator	: HZL
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\HZL\HZL-4-90\HZL-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\HZL\HZL-4-90\HZL-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

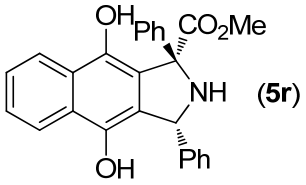


Area Percent Report

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

Signal 1: VWD1 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	



(**5r**)

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

```
    ==================================================================
    Acq. Operator   : HZL                          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\HZL\HZL-4-85\HZL-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\HZL\HZL-4-85\HZL-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)
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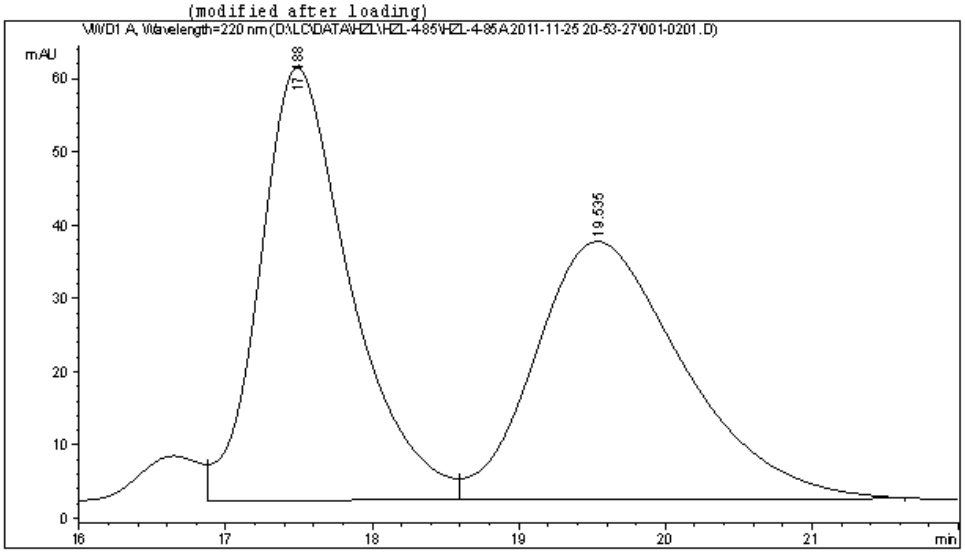



```
    ==================================================================
                            Area Percent Report
    ==================================================================

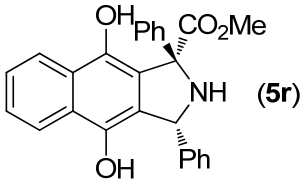
    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       1  17.488 VV    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

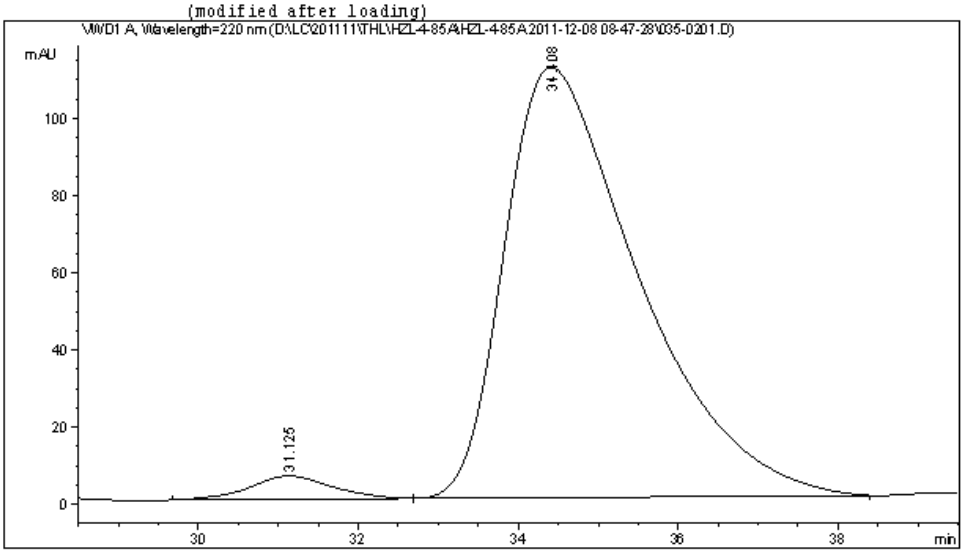
    ==================================================================
```



(**5r**)

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

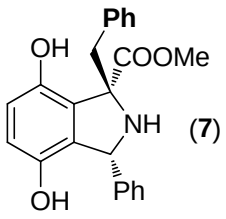
    Sorted By             :      Signal
    Multiplier            :      1.0000
    Dilution              :      1.0000
    Use Multiplier & Dilution Factor with ISTDs


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|------|----|-------|----------|----------|-------|
       1  31.125 BV    0.9243  440.21582    6.08338   3.2697
       2  34.408 VB    1.7225 1.30233e4   111.48381  96.7303

    Totals :                  1.34635e4   117.56719

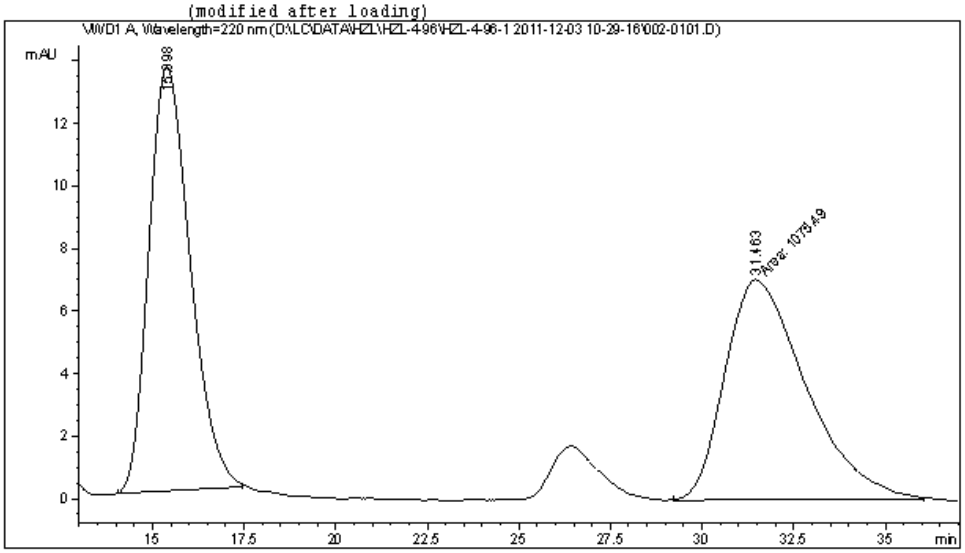
    ==================================================================
```



(**7**)

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

```
    ====================================================================
    Acq. Operator   : HZL                          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\HZL\HZL-4-96\HZL-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 HZL
    Analysis Method : D:\LC\DATA\HZL\HZL-4-96\HZL-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

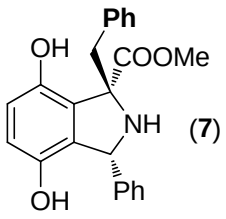

    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|--------|
       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

    ====================================================================
```

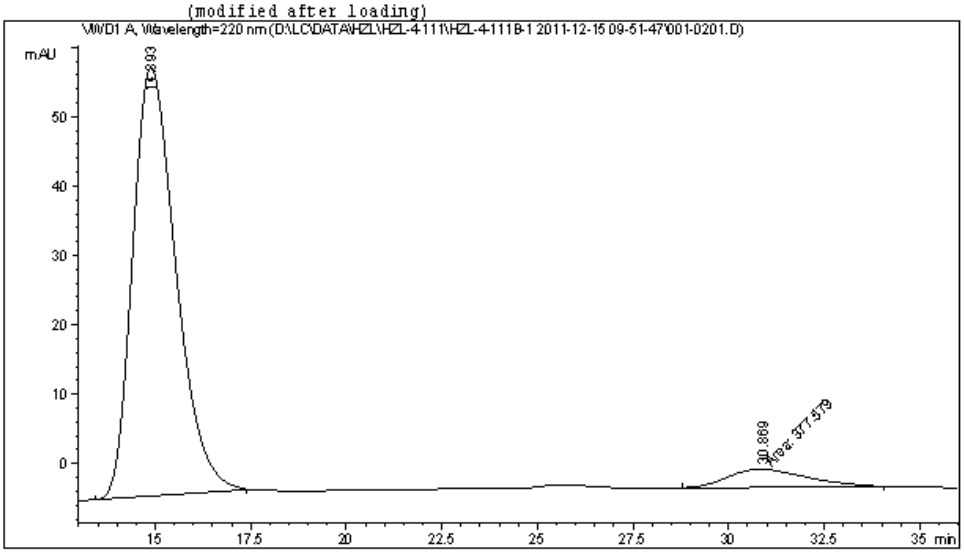
Instrument 1 8/14/2012 5:40:20 PM THL Page 1 of 1



(**7**)

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)

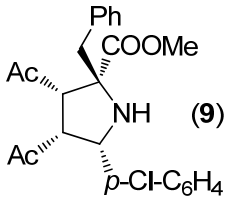


Area Percent Report

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

Signal 1: VWD1 A, Wavelength=220 nm

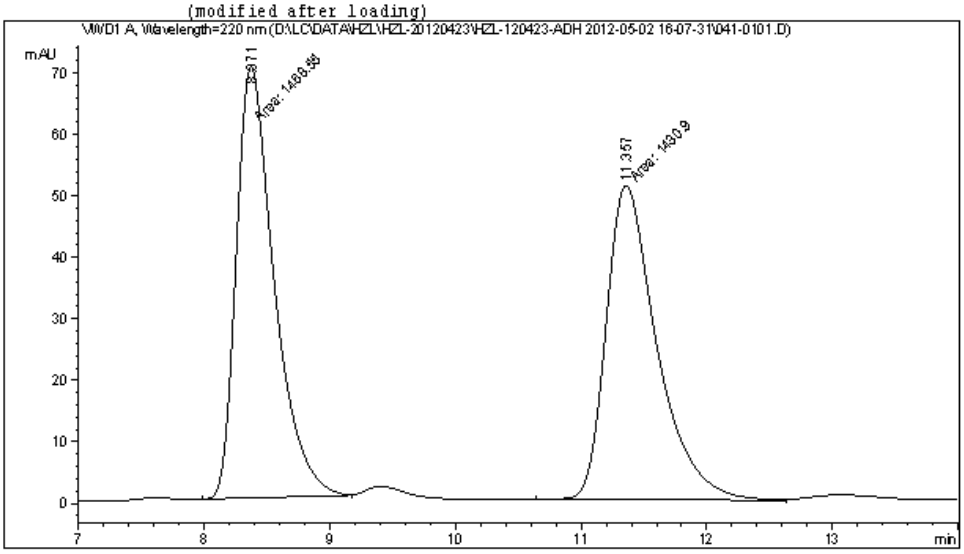
Peak #	RetTime [min]	Type	Width [min]	Area mAU *s	Height [mAU]	Area %
1	14.893	BB	1.2117	4852.64600	61.79405	92.7808
2	30.869	MM	2.4113	377.57892	2.60980	7.2192
Totals :				5230.22491	64.40385	



(9)

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

```
    ====================================================================
    Acq. Operator   : hzl                          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\HZL\HZL-20120423\HZL-120423-ADH 2012-05-02 16-07-31\ADH-20-80-
                      1ML-220NM.M
    Last changed    : 5/2/2012 3:53:21 PM by hzl
    Analysis Method : D:\LC\DATA\HZL\HZL-20120423\HZL-120423-ADH 2012-05-02 16-07-31\041-0101.D\
                      DA.M (ADH-20-80-1ML-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: VWD1 A, Wavelength=220 nm

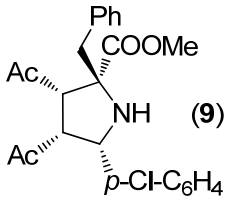
    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|------|----|-------|----------|----------|--------|
       1   8.371 MM    0.3496 1468.54810   70.00198  50.6492
       2  11.357 MM    0.4667 1430.90125   51.10359  49.3508

    Totals :                  2899.44934  121.10557

    ====================================================================
```

Instrument 1 5/16/2012 7:38:18 PM FX

Page 1 of 1



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

```
    ==================================================================
    Acq. Operator   : HZL                          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\HZL\HZL-5-122\HZL-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 HZL
    Analysis Method : D:\LC\DATA\HZL\HZL-5-122\HZL-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)
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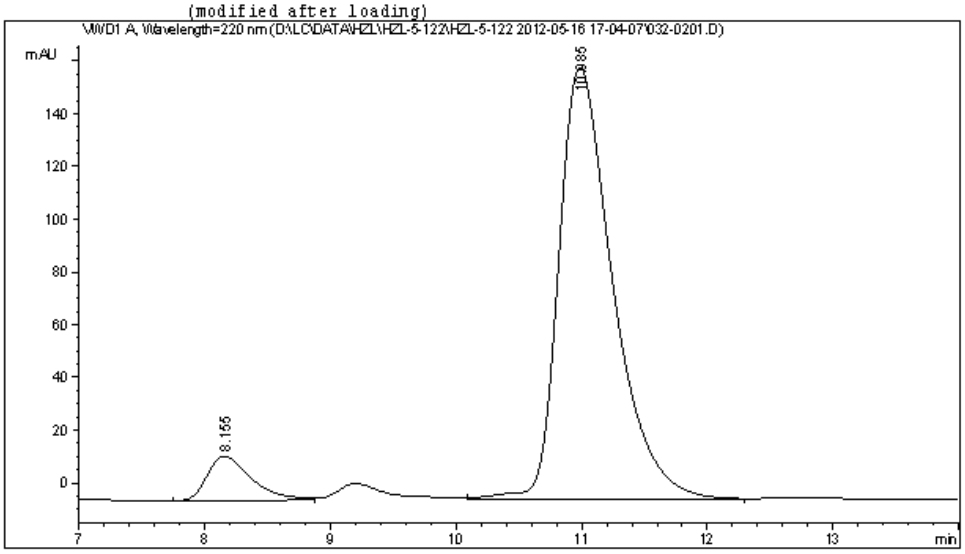


```
    ==================================================================
                          Area Percent Report
    ==================================================================

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


    Signal 1: VWD1 A, Wavelength=220 nm

    Peak RetTime Type  Width     Area      Height     Area
      #   [min]        [min]  mAU   *s   [mAU   ]      %
    ----|-------|----|-------|----------|----------|-------|
       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


    ==================================================================
```