*Supporting Information for*

# Rhodium-Catalyzed Direct Coupling of Biaryl Pyridine Derivatives with Internal Alkynes

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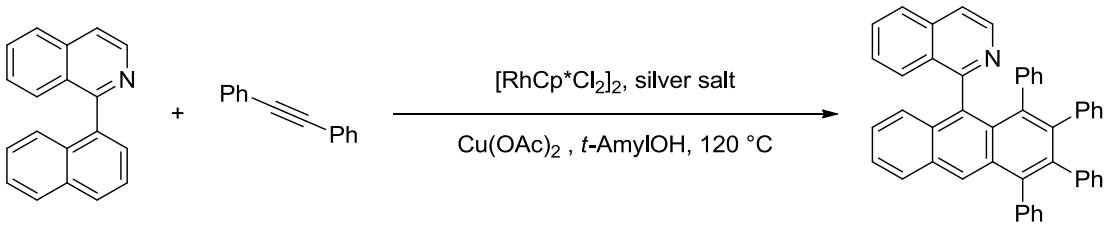
**General Methods**. Unless stated otherwise, all reactions were carried out in flame-dried glassware under a dry argon atmosphere. All solvents were purified and dried according to standard methods prior to use. $^{1}H$ and $^{19}F$ NMR spectra were recorded on a Varian or Agilent instrument (400 MHz and 376 MHz, respectively) and internally referenced to tetramethylsilane signal or residual protio solvent signals and $CFCl_3$, respectively. $^{13}C$ NMR spectra were recorded on a Varian instrument or Agilent instrument (100 MHz, respectively) and internally referenced to residual solvent signals. Data for $^{1}H$ NMR are recorded as follows: chemical shift (δ, ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet or unresolved, br = broad singlet, coupling constant (s) in Hz, integration). Data for $^{13}C$ NMR and $^{19}F$ NMR are reported in terms of chemical shift (δ, ppm).

Diarylacetylenes **2b-c**[1], **2e-f**[1], and biaryl compounds **1a-f**[2] were prepared according to the known procedures.

**Optimization Studies for Rh-catalyzed Dual C-H Functionalization/Cycloaromatization.**

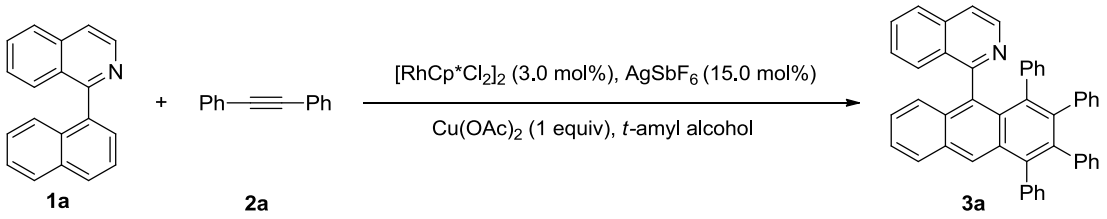
**Table S1: Effect of silver salt and other conditions.**[a]




| entry | silver salt | yield (%)[b] |
|---|---|---|
| 1 | AgOAc | 54 |
| 2 | AgOTf | 81 |
| 3 | $Ag_2CO_3$ | 55 |
| 4 | $AgPF_6$ | 78 |
| 5 | $AgSbF_6$ | 99 |
| 6 | - | 64 |
| 7[c] | $AgSbF_6$ | N.R. |
| 8[d] | $AgSbF_6$ | 85 |
| 9[e] | $AgSbF_6$ | 94 |

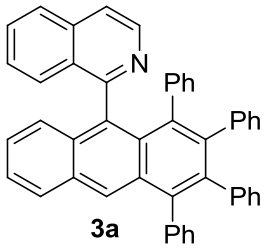
[a] Unless otherwise noted, all reactions were carried out as the following: $[RhCp^*Cl_2]_2$ (5 mol%), silver salt (25 mol%), **1a**/**2a**/ $Cu(OAc)_2$ = 1/ 2.2/ 2, 0.1 mol/L, and *t*-amyl alcohol (1 mL) in a sealed tube at 120 ℃ for 12 h. [b] Isolated yield. [c] $Cu(OTf)_2$ was used instead of $Cu(OAc)_2$. [d] reaction was run in reflux condition. [e] 100 ℃ was used.

**General Procedure for Rh-Catalyzed Direct Coupling of 1-(Naphthalen-1-yl)isoquinoline (1a) with Diphenylacetylene (2a):**




1a 2a 3a

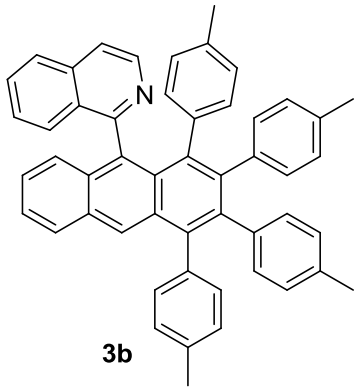
A flame-dried sealed tube was cooled to room temperature and filled with argon. To this flask were added 1-(naphthalen-1-yl)isoquinoline (**1a**) (0.2 mmol, 51.1 mg), diphenylacetylene (**2a**) (0.44 mmol, 78.4 mg), $[Cp^*RhCl_2]_2$ (0.006 mmol, 3.7 mg), silver hexafluoroantimonate(V) (0.03 mmol, 10.3 mg), $Cu(OAc)_2$ (0.2 mmol, 36.3 mg), and *t*-amylOH (2 mL). Then the sealed tube was heated at 120 $^oC$. After12 h, the reaction mixture was cooled to room temperature, $CH_2Cl_2$ (10 mL), water (10 mL) and ammonium hydroxide (15 M, 5 mL) were added. Then the organic layer was washed by ammonium hydroxide (15 M, 5 mL, two times) and brine, then separated, dried over $Na_2SO_4$ and filtered. The solvents were removed under reduced pressure. Then the residue was purified by silica gel column chromatography (PE/acetone = 30/1) to afford the desired product **3a**. Compounds **3b-3l** were prepared following the general procedure.




**3a**

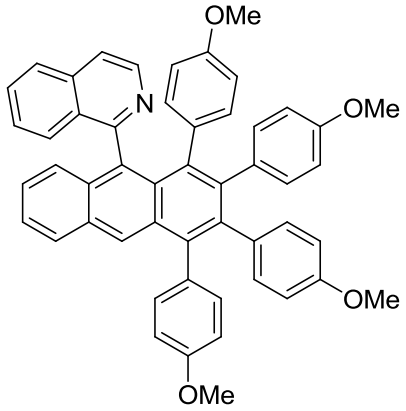
Yellow solid, m.p. = 152-154 $^o$C, 119.2 mg, 98% yield. $^1$H NMR (400 MHz, $CDCl_3$) δ 8.42 (s, 1H), 8.38 (d, *J* = 5.6 Hz, 1H), 7.84 (d, *J* = 8.4 Hz, 1H), 7.55 (d, *J* = 8.4 Hz, 1H), 7.47-7.44 (m, 2H), 7.40 (d, *J* = 8.4 Hz, 1H), 7.31-7.19 (m, 7H), 7.07-7.03 (m, 1H), 6.85-6.54 (m , 11H), 6.52-6.50 (m, 1H), 6.40 (t, *J* = 7.2 Hz, 1H), 6.36 (d, *J* = 7.6 Hz, 1 H), 6.18 (t, *J* = 7.6 Hz, 1H), 5.92 (t, *J* = 7.6 Hz, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 161.6, 141.9, 141.4, 140.56, 140.49, 139.9, 139.3, 138.35, 138.28, 137.7, 135.3, 134.0, 132.0, 131.7, 131.66, 131.64, 131.2, 131.0, 130.9, 130.8, 130.54, 130.50,

129.8, 129.5, 129.4, 128.6, 128.5, 128.1, 127.6, 127.5, 126.47, 126.43, 126.33, 126.28, 126.1, 126.0, 125.87, 125.84, 125.12, 125.09, 124.8, 124.7, 124.3, 124.0, 120.0; IR (thin film): $\nu_{max}(cm^{-1})$ = 3051, 3022, 2922, 2851, 1621, 1600, 1583, 1557, 1492, 1440, 746, 697; HRMS (ESI) calcd for $C_{47}H_{32}N$ $[M + H]^+$: 610.2529; Found: 610.2521.



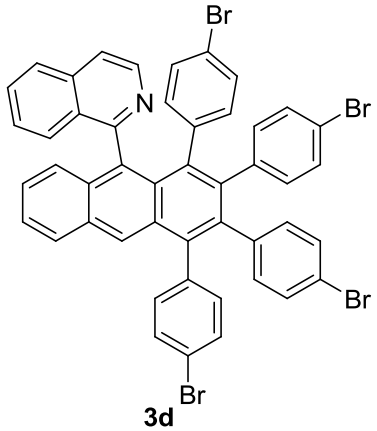

**3b**

Yellow solid, m.p. = 162-163 °C, 61.8 mg, 93% yield (0.1 mmol scale). $^1$H NMR (400 MHz, $CDCl_3$) δ 8.37-8.36 (m, 2H), 7.84 (d, *J* = 8.4 Hz, 1H), 7.58 (d, *J* = 8.4 Hz, 1H), 7.53-7.46 (m, 1H), 7.42 (d, *J* = 8.0 Hz, 1H), 7.33 (d, *J* = 5.6 Hz, 1H), 7.30-7.20 (m, 3H), 7.17-7.08 (m, 3H), 7.06-7.03 (m, 1H), 6.76 (d, *J* = 8.8 Hz, 1H), 6.72-6.70 (m, 1H), 6.67-6.58 (m, 4H), 6.51 (d, *J* = 7.6 Hz, 1H), 6.46-6.35 (m, 3H), 6.20 (dd, *J* = 7.6, 1.6 Hz, 1H), 6.15 (d, *J* = 8.0 Hz, 1H), 5.65 (d, *J* = 7.6 Hz, 1H), 2.36 (s, 3H), 2.05 (s, 3H), 1.95 (s, 3H), 1.74 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 162.1 , 142.0, 141.7 , 138.6 , 138.3 , 137.80, 137.79, 137.6, 137.2, 136.7, 135.7, 135.4, 134.1, 133.9, 133.7, 132.8, 132.0, 131.8, 131.7, 131.6, 131.1, 130.9, 130.8, 130.7, 130.4, 129.5, 129.3, 128.8, 128.7, 128.3, 128.2, 128.1, 127.1, 127.0, 126.7, 126.6, 126.24, 126.22, 126.15, 126.10, 125.6, 125.2, 124.6, 119.3, 21.3, 21.0, 20.9, 20.6; IR (thin film): $\nu_{max}(cm^{-1})$ = 3048, 2920, 1508, 1449, 1376, 1020, 818, 745; HRMS (ESI) calcd for $C_{51}H_{40}N$ $[M + H]^+$: 666.3155; Found: 666.3149.



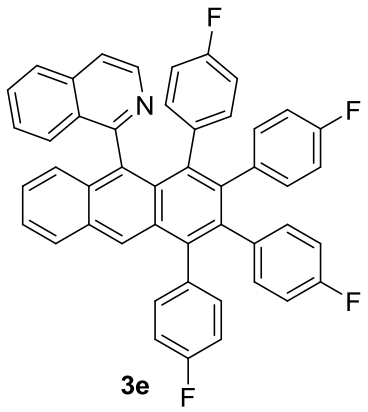

**3c**

Yellow solid, m.p. = 149-151 °C, 143.6 mg, 99% yield. $^{1}$H NMR (400 MHz, $CDCl_3$) δ 8.41-8.39 (m, 2H), 7.84 (d, *J* = 8.4 Hz, 1H), 7.63 (d, *J* = 8.0 Hz, 1H), 7.50 (t, *J* = 7.2 Hz, 1H), 7.44 (d, *J* = 8.4 Hz, 1H), 7.33 (d, *J* = 5.6 Hz, 1H), 7.28-7.22 (m, 3H), 7.20-7.15 (m, 1H), 7.07-7.01 (m, 1H), 6.85 (d, *J* = 8.4 Hz, 2H), 6.77 (d, *J* = 8.8 Hz, 1H), 6.77 (d, *J* = 7.6 Hz, 2H), 6.72 (d, *J* = 8.0 Hz, 2H), 6.61 (dd, *J* = 8.4, 1.6 Hz, 1H), 6.47-6.36 (m, 4H), 6.29-6.15 (m, 3H), 5.96 (dd, *J* = 8.4, 2.4 Hz, 1H), 5.43 (dd, *J* = 8.4, 2.4 Hz, 1H), 3.79 (s, 3H), 3.55 (s, 3H), 3.46 (s, 3H), 3.38 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 162.1, 157.8, 156.6, 156.3, 155.4, 142.0, 141.8, 138.5, 138.1, 137.5, 135.4, 133.7, 133.3, 132.9, 132.6, 132.3, 132.2, 132.1, 131.8, 131.6, 131.4, 131.3, 130.6, 130.4, 129.4, 128.63, 128.58, 128.0, 126.3, 126.2, 126.0, 125.7, 124.6, 119.7, 113.1, 113.0, 112.0, 111.9, 111.6, 111.5, 109.1, 55.0, 54.6, 54.5, 54.4; IR (thin film): $\nu_{max}$($cm^{-1}$) = 3046, 2932, 2833, 1609, 1575, 1513, 1463, 1285, 1244, 1175, 1033, 827; HRMS (ESI) calcd for $C_{51}H_{40}NO_4$ $[M + H]^+$: 730.2952; Found: 730.2944.




**3d**

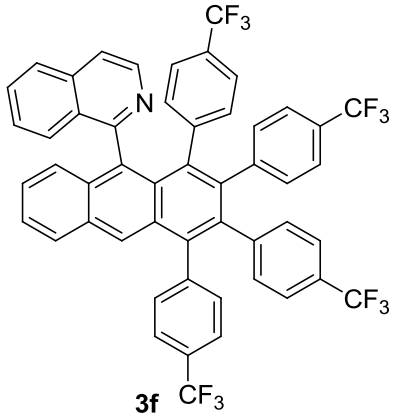
Yellow solid, m.p. > 300 °C, 174.1 mg, 95% yield. $^{1}$H NMR (400 MHz, $CDCl_3$) δ 8.39 (d, *J* = 5.6 Hz, 1H), 8.31 (s, 1H), 7.87 (d, *J* = 8.4 Hz, 1H), 7.72 (d, *J* = 8.4 Hz, 1H), 7.59-7.55 (m, 1H), 7.52-7.44 (m, 3H), 7.37-7.33 (m, 2H), 7.31-7.19 (m, 2H), 7.15-7.07 (m, 2H), 7.04-7.01 (m, 2H), 6.91 (dd, *J* = 8.0, 1.6 Hz, 1H), 6.87-6.81 (m, 2H), 6.68 (d, *J* = 8.4 Hz, 1H), 6.62 (d, *J* = 8.4 Hz, 1H), 6.56 (td, *J* = 8.4, 2.0 Hz, 2H), 6.40-6.33 (m, 2H), 6.17 (dd, *J* = 8.0, 2.0 Hz, 1H), 6.01 (dd, *J* = 8.0, 2.0 Hz, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 161.5, 142.0, 139.5, 138.8, 138.7, 138.2, 137.8, 137.6, 137.1, 136.5, 135.3, 134.2, 133.0, 132.9, 132.6, 132.35, 132.27, 132.25, 132.1, 131.9, 131.3, 131.2, 131.10, 131.08, 130.2, 130.1, 129.8, 129.74, 129.66, 129.5, 129.4, 128.8, 128.6, 128.1, 128.0, 127.6, 127.1, 126.7, 126.5, 126.1, 125.5, 121.2, 120.1, 119.9, 119.7, 118.9; IR (thin film): $\nu_{max}$($cm^{-1}$) = 3049, 1899, 1621, 1558, 1557, 1488, 1389, 1011, 822, 755, 746; HRMS (ESI) calcd for $C_{47}H_{28}Br_4N$ $[M + H]^+$: 921.8950; Found: 921.8938.




**3e**

Yellow solid, m.p. = 146-148 °C, 134.7 mg, 98% yield. $^{1}$H NMR (400 MHz, $CDCl_3$) δ 8.40 (d, *J* = 5.6 Hz, 1H), 8.36 (s, 1H), 7.87 (d, *J* = 8.4 Hz, 1H), 7.67 (d, *J* = 8.0 Hz, 1H), 7.54 (t, *J* = 7.2 Hz, 1H), 7.43-7.38 (m, 2H), 7.37-7.25 (m, 3H), 7.24-7.20 (m, 1H), 7.15-7.08 (m, 1H), 7.06-7.00 (m, 2H), 6.85 (d, *J* = 8.8 Hz, 1H), 6.80-6.68 (m, 2H), 6.66-6.62 (m, 1H), 6.60-6.54 (m, 2H), 6.53-6.41 (m, 3H), 6.37 (td, *J* = 8.4, 2.4 Hz, 1H), 6.28-6.25 (m, 1H), 6.14 (td, *J* = 8.8, 2.4 Hz, 1H), 5.62 (td, *J* = 8.8, 2.4 Hz, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 161.6 (d, *J* = 244.6 Hz), 161.5, 160.6 (d, *J* = 243.9 Hz), 160.3 (d, *J* = 242.2 Hz), 159.5 (d, *J* = 243.3 Hz), 142.0, 140.6, 138.1, 137.5, 137.4, 136.2 (d, *J* = 3.6 Hz), 136.1 (d, *J* = 3.4 Hz), 135.43 (d, *J* = 4.1 Hz),

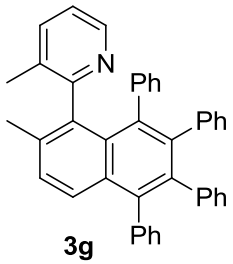
135.41, 135.1 (d, *J* = 3.5 Hz), 134.1, 133.2 (d, *J* = 8.2 Hz), 133.0 (d, *J* = 7.8 Hz), 132.5 (d, *J* = 7.9 Hz), 132.3-132.2 (m), 132.0, 131.8 (d, *J* = 7.8 Hz), 131.59, 131.57 (d, *J* = 7.5 Hz), 131.0, 129.8, 129.7, 129.5, 128.6, 128.1, 126.7, 126.6, 126.3, 126.1, 125.3, 120.1,115.0 (d, *J* = 21.1 Hz), 114.8 (d, *J* = 21.3 Hz), 113.9 (d, *J* = 12.1 Hz), 113.7 (d, *J* = 12.0 Hz), 113.5 (d, *J* = 10.5 Hz), 113.3 (d, *J* = 10.6 Hz), 112.7 (d, *J* = 21.4 Hz), 111.2 (d, *J* = 21.3 Hz); $^{19}$F NMR (386 Hz, $CDCl_3$) δ -115.1 (m), -116.4 (m), -116.8 (m), -117.7 (m); IR (thin film): $\nu_{max}(cm^{-1})$ = 3046, 2926,1604, 1557, 1509, 1224, 1157, 830, 745; HRMS (ESI) calcd for $C_{47}H_{28}F_4N$ $[M + H]^+$: 682.2152; Found: 682.2141.




**3f**

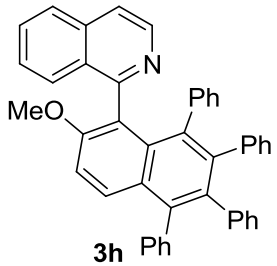
Yellow solid, m.p. = 149-151 °C, 159.5 mg, 90% yield. $^1$H NMR (400 MHz, $CDCl_3$) δ 8.43 (d, *J* = 5.6 Hz, 1H), 8.34 (s, 1H), 7.93 (d, *J* = 8.4 Hz, 1H), 7.67-7.63 (m, 3H), 7.58-7.56 (m, 2H), 7.45-7.33 (m, 5H), 7.23-7.12 (m, 3H), 7.05 (d, *J* = 8.0 Hz, 1H), 7.01-6.89 (m, 4H), 6.86 (d, *J* = 8.0 Hz, 1H), 6.74 (d, *J* = 7.6 Hz, 1H), 6.68 (d, *J* = 8.0 Hz, 1H), 6.64 (d, *J* = 8.4 Hz, 1H), 6.50 (d, *J* = 7.6 Hz, 1H), 6.22 (d, *J* = 8.0 Hz, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 160.8, 143.3 (q, *J* = 1.1 Hz), 143.2 (q, *J* = 1.1 Hz), 142.8 (q, *J* = 1.0 Hz), 142.2 (q, *J* = 1.2 Hz), 141.9, 139.3. 138.1, 137.4, 136.3, 135.3, 134.5, 132.3, 131.9, 131.8, 131.4, 131.3, 131.1, 131.01, 130.98, 130.7, 130.1, 129.9, 129.4 (q, *J* = 32.4 Hz), 129.5, 129.1, 128.7, 128.3 (q, *J* = 32.6 Hz), 128.2, 127.9 (q, *J* = 32.3 Hz), 127.8, 127.04, 127.01, 126.9, 126.6 (q, *J* = 32.1 Hz), 126.2, 125.9, 125.1-124.9 (m), 124.0 (q, *J* = 4.9 Hz), 123.9 (q, *J* = 3.6 Hz), 123.6 (q, *J* = 3.6 Hz), 123.4 (q, *J* = 3.7 Hz), 122.4-122.3 (m), 121.3 (q, *J* = 270.7 Hz), 121.1 (q, *J* = 3.8 Hz),

121.0 (q, *J* = 270.3 Hz), 120.9 (q, *J* = 270.7 Hz), 120.8 (q, *J* = 270.6 Hz), 120.7; $^{19}$F NMR (386 Hz, $CDCl_3$) δ -62.5, -62.9, -63.0, -63.2; IR (thin film): $\nu_{max}(cm^{-1})$ = 3046, 2926,1618, 1584, 1557, 1499, 1406,1325, 1167, 1123, 1066, 1019; HRMS (ESI) calcd for $C_{51}H_{28}F_{12}N$ $[M + H]^+$: 882.2025; Found: 882.2014 .




**3g**

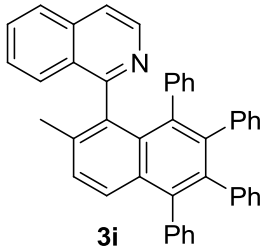
White solid, m.p. = 111-113 °C, 130.7 mg, 76% yield (0.32 mmol scale). $^1$H NMR (400 MHz, $CDCl_3$) δ 8.24 (d, *J* = 4.0 Hz, 1H), 7.62 (d, *J* = 8.8 Hz, 1H), 7.35-7.10 (m, 6H), 6.89 (d, *J* = 7.2 Hz, 1H), 6.83-6.56 (m, 15H), 6.49 (t, *J* = 7.2 Hz, 1H), 1.91 (s, 3H), 1.89 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 160.0, 146.1, 141.2, 140.7, 140.58, 140.55, 140.0, 138.4, 137.9, 137.5, 136.6, 136.4, 135.1, 132.1, 132.0, 131.8, 131.4, 131.1, 130.99, 130.96, 130.7, 130.4, 129.9, 128.5, 127.3, 127.2, 126.3, 126.2, 125.95, 125.93, 125.8, 125.2, 124.9, 124.64, 124.60, 121.5, 19.9, 19.5; IR (thin film): $\nu_{max}(cm^{-1})$ = 3054, 3023, 2920, 1601, 1581, 1493, 1441, 1376, 1027, 725, 697; HRMS (ESI) calcd for $C_{41}H_{32}N$ $[M + H]^+$: 538.2529; Found: 538.2508.




**3h**

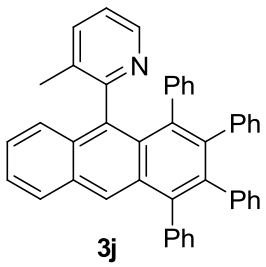
Yellow solid, m.p. = 150-151 °C, 112.3 mg, 95% yield. $^1$H NMR (400 MHz, $CDCl_3$) δ 8.27 (d, *J* = 5.6 Hz, 1H), 7.82 (d, *J* = 9.6 Hz, 1H), 7.69 (d, *J* = 8.0 Hz, 1H), 7.51-7.42 (m, 2H), 7.34-7.30 (m, 1H), 7.29-7.16 (m, 6H), 7.13 (d, *J* = 5.6 Hz, 1H), 6.83-6.64 (m, 7H), 6.62-6.55 (m, 2H), 6.46-6.43 (m, 2H), 6.39 (d, *J* = 7.6 Hz, 1H), 6.31 (t, *J* = 7.2 Hz, 1H), 6.12 (t, *J* = 7.6 Hz, 1H), 5.89 (t, *J* = 7.2 Hz, 1H), 3.54 (s, 3H);

$^{13}$C NMR (100 MHz, $CDCl_3$) δ 160.1, 156.1, 141.8, 141.6, 140.60, 140.55, 139.9, 139.2, 138.4, 137.2, 137.0, 135.3, 132.5, 132.0, 131.5, 131.12, 131.09, 131.06, 131.0, 130.52, 130.50, 130.1, 129.0, 128.9, 128.3, 127.4, 126.4, 126.3, 126.2, 126.1, 125.9, 125.8, 125.1, 125.0, 124.6, 124.2, 123.8, 123.4, 119.6, 113.1, 56.7; IR (thin film): $\nu_{max}$($cm^{-1}$) = 3050, 3022, 2933, 2838, 1599, 1500, 1441, 1266, 747, 697; HRMS (ESI) calcd for $C_{44}H_{32}NO$ $[M + H]^+$: 590.2478; Found: 590.2462.




**3i**

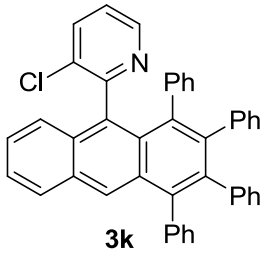
Yellow solid, m.p. = 139-140 °C, 97.7 mg, 84% yield. $^1$H NMR (400 MHz, $CDCl_3$) δ 8.29 (d, *J* = 6.0 Hz, 1H), 7.71 (d, *J* = 8.8 Hz, 1H), 7.60 (d, *J* = 8.4 Hz, 1H), 7.52 (d, *J* = 8.0 Hz, 1H), 7.47 (td, *J* = 6.8, 0.8 Hz, 1H), 7.35-7.14 (m, 7H), 6.84-6.45 (m, 12H), 6.37-6.31 (m, 2H), 6.15 (t, *J* = 7.2 Hz, 1H), 5.89 (t, *J* = 7.2 Hz, 1H), 1.81 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 162.4, 141.8, 141.3, 140.7, 140.6, 140.0, 139.3, 138.4, 138.1, 137.8, 136.1, 135.47, 135.46, 132.1, 131.9, 131.5, 131.4, 131.10, 131.07, 131.05, 131.00, 130.7, 129.3, 128.5, 128.4, 127.9, 127.42, 127.36, 126.43, 126.38, 126.3, 126.2, 125.9, 125.8, 125.02, 124.98, 124.6, 124.2, 123.9, 119.6, 20.6; IR (thin film): $\nu_{max}$($cm^{-1}$) = 3052, 3023, 1601, 1583, 1557, 1494, 1441, 748, 696; HRMS (ESI) calcd for $C_{44}H_{32}N$ $[M + H]^+$: 574.2529; Found: 574.2510.




**3j**

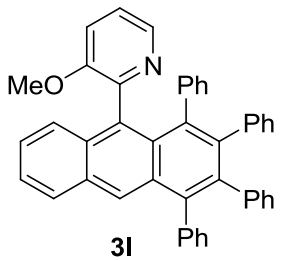
Yellow solid, m.p. = 135-137 °C, 115.0 mg, 99% yield. $^1$H NMR (400 MHz, $CDCl_3$) δ 8.33-8.32 (m, 2H), 7.81 (d, *J* = 8.4 Hz, 1H), 7.38 (d, *J* = 8.0 Hz, 1H), 7.33-7.16 (m,

6H), 6.97 (t, *J* = 8.4 Hz, 2H), 6.86-6.71 (m, 10H), 6.69-6.58 (m, 5H), 6.53 (t, *J* = 7.6 Hz, 1H), 1.86 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 159.1, 146.2, 141.3, 140.7, 140.6, 140.5, 139.9, 138.4, 138.2, 137.6, 136.5, 135.3, 133.3, 131.9, 131.64, 131.61, 131.2, 131.1, 130.94, 130.90, 130.87, 130.64, 130.62, 129.7, 128.84, 128.77, 127.58, 127.56, 127.44, 126.4, 126.3, 126.2, 126.1, 126.0, 125.9, 125.33, 125.30, 125.1, 124.8, 124.7, 122.0, 20.0; IR (thin film): $\nu_{max}$($cm^{-1}$) = 3052, 3023, 2921, 1732, 1601, 1582, 1493, 1441, 751, 698; HRMS (ESI) calcd for $C_{44}H_{32}N$ $[M + H]^+$: 574.2529; Found: 574.2523.




**3k**

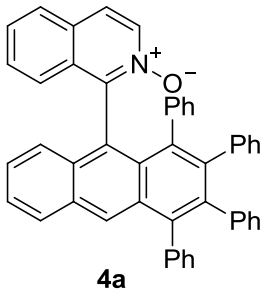
Yellow solid, m.p. = 104-106 °C, 119.0 mg, 99% yield. $^1$H NMR (400 MHz, $CDCl_3$) δ 8.38-8.36 (m, 2H), 7.84 (d, *J* = 8.4 Hz, 1H), 7.43-7.16 (m, 8H), 7.07 (d, *J* = 7.2 Hz, 1H), 6.99 (d, *J* = 8.8 Hz, 1H), 6.90-6.72 (m, 9H), 6.71-6.51 (m, 6H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 158.2, 146.9, 141.7, 140.6, 140.5, 140.3, 139.8, 138.5, 138.3, 137.2, 136.2, 133.4, 133.1, 132.1, 131.6, 131.5, 131.2, 131.1, 130.98, 130.93, 130.8, 130.69, 130.66, 130.5, 128.9, 128.7, 128.5, 127.6, 126.5, 126.4, 126.35, 126.32, 126.06, 126.03, 126.02, 126.00, 125.2, 125.1, 125.04, 124.98, 124.8, 122.9; IR (thin film): $\nu_{max}$($cm^{-1}$) = 3053, 3023, 2922, 1601, 1571, 1441, 1362, 735, 699; HRMS (ESI) calcd for $C_{43}H_{29}ClN$ $[M + H]^+$: 594.1983; Found: 594.1960.




**3l**

Yellow solid, m.p. = 159-161 °C, 104.6 mg, 89% yield. $^1$H NMR (400 MHz, $CDCl_3$) δ 8.29 (s, 1H), 8.09 (dd, *J* = 4.8, 0.8 Hz, 1H), 7.81 (d, *J* = 8.4 Hz, 1H), 7.38-7.16 (m, 7H), 7.03 (d, *J* = 8.8 Hz, 1H), 6.90-6.87 (m, 2H), 6.86-6.71 (m, 8H), 6.71-6.63 (m, 3H), 6.59-6.48 (m, 4H), 3.49 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 153.2, 150.1, 141.0, 140.72, 140.68, 140.65, 140.4, 140.0, 138.3, 138.1, 137.5, 133.6, 132.2, 131.75, 131.69, 131.28, 131.24, 131.03, 131.00, 130.9, 130.6, 130.5, 129.5, 128.8, 127.7, 127.53, 127.49, 126.42, 126.39, 126.3, 126.0, 125.9, 125.8, 125.6, 125.1, 124.8, 124.7, 124.64, 124.61, 123.2, 115.7, 53.9; IR (thin film): $\nu_{max}$($cm^{-1}$) = 3053, 3023, 2932, 1601, 1582, 1492, 1456, 1441, 1426, 1279, 753, 698; HRMS (ESI) calcd for $C_{44}H_{32}NO[M + H]^+$: 590.2478; Found: 590.2466.

**General Procedure for the Preparation of Chiral Isoquinoline *N*-oxides:**

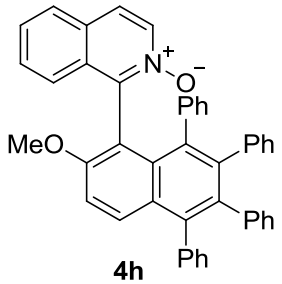



**4a**

**(±)-4a**. *m*-Chloroperoxybenzoic acid (152.8 mg, 1.2 mmol) was added to a solution of **3a (**365.9 mg, 0.6 mmol**)** in $CH_2Cl_2$ at 0 °C, and the mixture was stirred at room temperature. After the reaction was complete (monitored by TLC), the mixture was then quenched by saturated aqueous $NaHCO_3$ (5 mL), the organic solution was washed with brine, dried over $Na_2SO_4$, filtered and evaporated in vacuo. The crude product was purified by column chromatography on silica gel with ethyl acetate to afford **(±)-4a** as yellow solid (348.5 mg, 93% yield), m.p. = 244-245 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 8.50 (s, 1H), 7.82-7.80 (m, 2H), 7.56 (d, *J* = 7.2 Hz, 1H), 7.49 (d, *J* = 7.6 Hz, 1H), 7.41 (d, *J* = 7.2 Hz, 1H), 7.31-7.10 (m, 10H), 6.90 - 6.89 (m, 2H), 6.83-6.57 (m, 10H), 6.43 (t, *J* = 7.2 Hz, 1H), 6.18 (d, *J* = 7.2 Hz, 1H), 5.98 (t, *J* = 7.2 Hz, 1H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 146.0, 141.9, 140.4, 140.2, 139.6, 138.9, 138.5, 138.4, 136.8, 136.7, 131.4, 131.3, 131.2, 131.1, 131.0, 130.8, 130.70, 130.66,

130.5, 129.9, 129.7, 129.0, 128.9, 128.7, 128.5, 127.8, 127.6, 127.5, 127.3, 127.0, 126.4, 126.3, 126.2, 126.1, 125.96, 125.93, 125.7, 125.6, 125.1, 125.0, 124.9, 124.7, 124.4, 123.4; IR (thin film): $\nu_{max}(cm^{-1})$ = 3053, 3022, 1619, 1599, 1557, 1491, 1440, 1324, 753, 698; HRMS (ESI) calcd for $C_{47}H_{32}NO$ $[M + H]^+$: 626.2478; Found: 626.2454.

A solution of the racemic **4a** (60.0 mg in 20.0 mL of $^{i}$PrOH) was separated at a time by semi-preparative HPLC (Daicel CHIRALCEL IC, 2 cm x 25 cm, 60% $^{i}$PrOH in hexanes, 5 mL/min). Enantiomer **(+)-4a** was collected from 83 minute to 100.0 minute (27.5 mg, 46%, $[\alpha]_D^{20}$ = +7.7, c = 0.2, $CHCl_3$), enantiomerically pure by HPLC analysis. Enantiomer **(-)-4a** was collected from 105 minute to 120 minute (27.5 mg, 46%, $[\alpha]_D^{20}$ = -7.8, c = 0.22, $CHCl_3$), enantiomerically pure by HPLC analysis.



**4h**

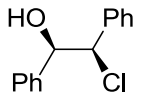
**(±)-4h**. *m*-Chloroperoxybenzoic acid (40.1 mg, 0.23 mmol) was added to a solution of **3h (**138.2 mg, 0.23 mmol**)** in $CH_2Cl_2$ at 0 °C, and the mixture was stirred at room temperature. After the reaction was complete (monitored by TLC), the mixture was then quenched by saturated aqueous $NaHCO_3$ (2 mL), the organic solution was washed with brine, dried over $Na_2SO_4$, filtered and evaporated in vacuo. The crude product was purified by column chromatography on silica gel (EtOAc, then 10% methanol in DCM) to afford **(±)-4h** as white solid (123.8 mg, 87% yield), m.p. = 179-180 °C. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.91 (d, *J* = 9.6 Hz, 1H), 7.79 (d, *J* = 7.2 Hz, 1H), 7.51-7.49 (m, 1H), 7.46 (d, *J* = 7.6 Hz, 1H), 7.41-7.18 (m, 9H), 7.09 (d, *J* = 7.2 Hz, 1H), 6.88-6.58 (m, 11H), 6.38 (t, *J* = 7.2 Hz, 1H), 6.15 (d, *J* = 7.6 Hz, 1H), 5.92 (t, *J* = 7.2 Hz, 1 H), 3.66 (s, 3H); $^{13}$C NMR (100 MHz, $CDCl_3$) δ 156.1, 145.2,

142.2, 140.6, 140.5, 139.9, 139.1, 138.4, 137.3, 136.7, 136.2, 131.7, 131.4, 131.2, 131.1, 131.00, 130.97, 130.8, 130.7, 130.2, 128.9, 128.5, 128.3, 128.2, 127.6, 127.4, 127.3, 126.5, 126.4, 126.3, 126.2, 126.1, 126.0, 125.9, 125.8, 125.6, 125.1, 124.7, 124.3, 122.9, 114.2, 112.9, 56.7; IR (thin film): $\nu_{max}(cm^{-1})$ = 3424, 3054, 2924, 1600, 1502, 1272, 1114, 697; HRMS (ESI) calcd for $C_{44}H_{32}NO_2$ $[M + H]^+$: 606.2428; Found: 606.2429.

A solution of the racemic **4h** (60.0 mg in 20.0 mL of $^{i}$PrOH) was separated at a time by semi-preparative HPLC (Daicel CHIRALCEL IC, 2 cm x 25 cm, 40% $^{i}$PrOH in hexanes, 5 mL/min). Enantiomer **(-)-4h** was collected from 42.5 minute to 46.5 minute (29.5 mg, 49%, $[\alpha]_D^{20}$ = -89.7, c = 0.2, $CHCl_3$), enantiomerically pure by HPLC analysis. Enantiomer **(+)-4h** was collected from 89.5 minute to 92.5 minute (29.5 mg, 49%, $[\alpha]_D^{20}$ = +89.7, c = 0. 25, $CHCl_3$), enantiomerically pure by HPLC analysis.

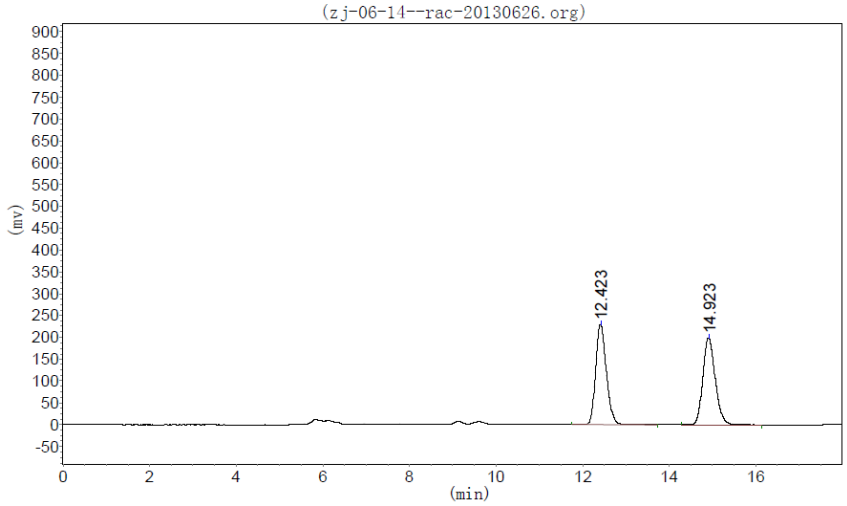
**General Procedure for Desymmetrization of meso Epoxide by Axially Chiral Pyridine *N*-Oxides**

The reaction was carried out by following the procedures reported by Takenaka[3], Denmark[4] and Fu[5]. The catalysts were recovered in >85% yield without racemization.

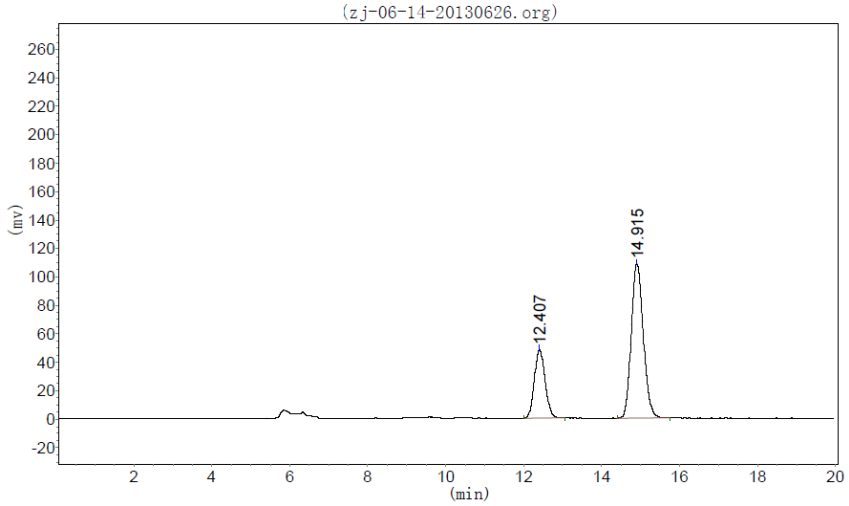



To a cooled (-78 ℃) solution of catalyst **(+)-4a** (9.4 mg, 0.015 mmol), *cis*-stilbene oxide (30.0 mg, 0.153 mmol) and $^{i}Pr_2NEt$ (35 μL, 0.200 mmol) in $CH_2Cl_2$ (2 mL) was added $SiCl_4$ (1.0 M solution in $CH_2Cl_2$; 200 μL) over 10 min. After 48 hours at -78 ℃, the reaction was quenched by drop-wise addition of $^{i}Pr_2NEt$ (0.2 mL) and 2-dimethylaminoethanol (0.1 mL) and then stirred for 30 min at -78 ℃. The mixture was transferred via a cannula into a cold (0 ℃), rapidly stirring mixture of saturated aqueous KF/1M $KH_2PO_4$ solution (1/1, 20 mL). The resulting mixture was stirred for

5 min, and then extracted with DCM (3 x 25 mL). The combined organic layers were dried over $Na_2SO_4$, filtered, and concentrated in vacuo. The crude material was purified by flash chromatography on silica gel (5% petroleum ether in EtOAc) to afford (1*R*,2*R*)-2-chloro-1,2-diphenylethanol (23.9 mg, 78%) with 43% ee. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.25-7.16 (m, 8H), 7.10-7.09 (m, 2H), 5.00 (d, *J* = 8.4 Hz, 1H), 4.95 (d, *J* = 8.4 Hz, 1H), 3.06 (s, 1H); All spectral data were identical to the literature values.[3] Enantiomeric excess was determined by HPLC with a Daicel Chiralpak AS-H, n-hexane/2-propanol = 90/10, v = 0.5 mL · min$^{-1}$, λ = 220 nm, t (minor) = 12.41 min, t (major) = 14.92 min; $[\alpha]_D^{20}$ = -11.2 (c = 0.33, EtOH).

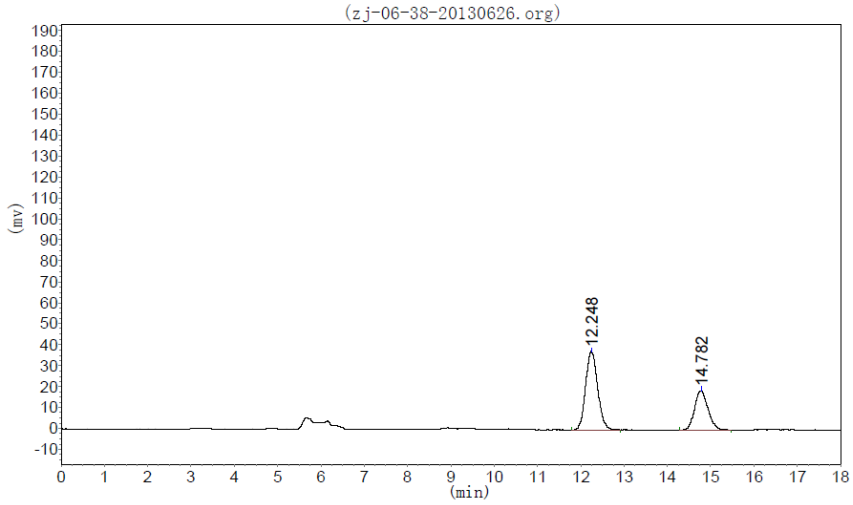



| Peak No. | R. Time | Peak Height | Peak Area | Percent |
|---|---|---|---|---|
| 1 | 12.423 | 228728.813 | 3949327.500 | 49.8506 |
| 2 | 14.923 | 198312.031 | 3972994.250 | 50.1494 |
| Total | | 427040.844 | 7922321.750 | 100.0000 |




| Peak No. | R. Time | Peak Height | Peak Area | Percent |
|---|---|---|---|---|
| 1 | 12.407 | 48225.102 | 925779.375 | 28.3151 |
| 2 | 14.915 | 108381.023 | 2343778.750 | 71.6849 |
| **Total** | | 156606.125 | 3269558.125 | 100.0000 |

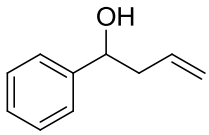
When **(-)-4h** was used as catalyst, the general procedure was followed with **(+)-4a** (9.1 mg, 0.015 mmol) and the epoxide (30.0 mg, 0.153 mmol) to give (1*R*,2*R*)-2-chloro-1,2-diphenylethanol (15.7 mg, 45%) with -28% ee. Enantiomeric excess was determined by HPLC with a Daicel Chiralpak AS-H, n-hexane/2-propanol = 90/10, v = 0.5 mL $\cdot$ min$^{-1}$, $\lambda$ = 220 nm, t (minor) = 14.78 min, t (major) = 12.25 min; $[\alpha]_D^{20}$ = +6.2 (c = 0.27, EtOH).



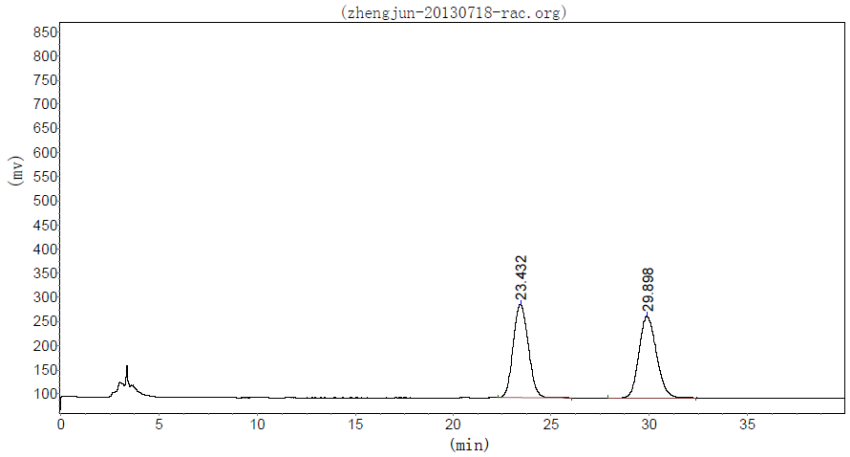

| Peak No. | R. Time | Peak Height | Peak Area | Percent |
|---|---|---|---|---|
| 1 | 12.248 | 37401.000 | 721707.625 | 64.0949 |
| 2 | 14.782 | 18583.918 | 404291.500 | 35.9051 |
| **Total** | | 55984.918 | 1125999.125 | 100.0000 |

**General Procedure for the Asymmetric Allylation of Benzaldehyde with Allyltrichlorosilane by Axially Chiral Pyridine *N*-Oxides**

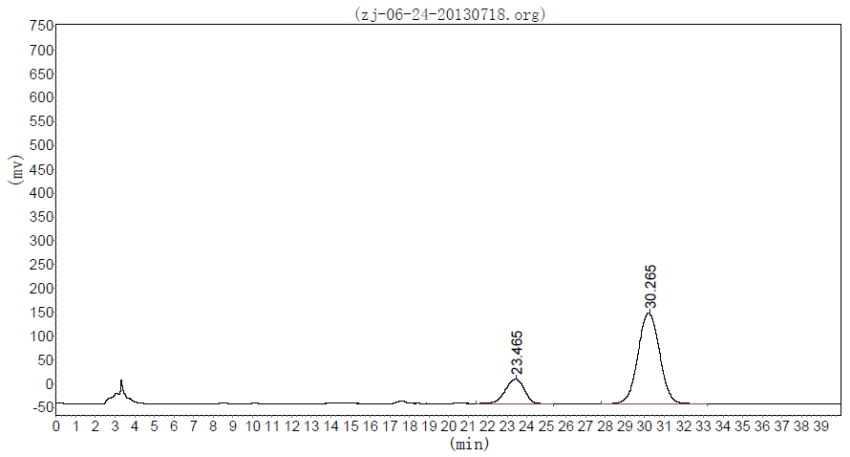
The reaction was carried out by following the procedures reported by Kočovský.[6] The catalysts were recovered in >85% yield without racemization.




Allyltrichlorosilane (56 μL, 0.35 mmol) was added to a solution of catalyst **(+)-4a** (18.7 mg, 0.03 mmol), $^{i}Pr_2NEt$ (52 μL, 0.375 mmol), and benzaldehyde (31.9 mg, 0.3 mmol) in MeCN (2 mL) under argon at -40 ℃. The reaction mixture was stirred at -40 ℃ for 48 h. The reaction was quenched with saturated aqueous $NaHCO_3$ (1 mL). The organic layers were separated, and the aqueous layer was extracted with DCM (2 x 5 mL). The combined organic extracts were washed with brine (3 mL) and dried over $Na_2SO_4$. The solvent was removed in vacuo. The residue was purified by column chromatography on silica gel with a petroleum ether/ethyl acetate mixture (15:1) to afford (*S*)-1-phenylbut-3-en-1-ol (12.7 mg, 29% yield, 62% ee). $^1$H NMR (400 MHz, $CDCl_3$) δ 7.35-7.31 (m, 4H), 7.28-7.22 (m, 1H), 5.82-5.72 (m, 1H), 5.15-5.09 (m, 2H), 4.67 (t, *J* = 6.8 Hz, 1H), 2.49-2.46(m, 2H), 2.33 (s, 1H); All spectral data were identical to the literature values.[6] Enantiomeric excess was determined by HPLC with a Daicel Chiralpak OD-H, n-hexane/2-propanol = 99/1, v = 1.0 mL · min$^{-1}$, λ = 220 nm, t (minor) = 23.47 min, t (major) = 30.27 min; $[\alpha]_D^{20}$ = -26.9 (c = 0.21, $CHCl_3$).

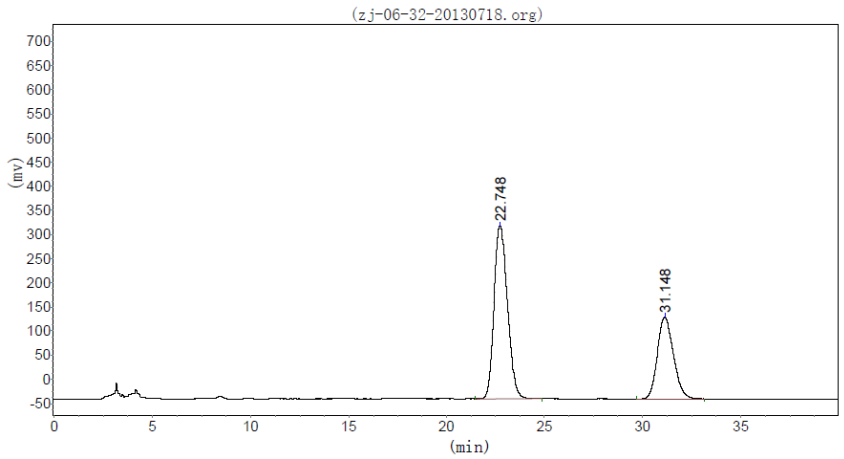



| Peak No. | R. Time | Peak Height | Peak Area | Percent |
|---|---|---|---|---|
| 1 | 23.432 | 193763.156 | 10379736.000 | 49.9334 |
| 2 | 29.898 | 168826.438 | 10407441.000 | 50.0666 |
| **Total** | | 362589.594 | 20787177.000 | 100.0000 |




| Peak No. | R. Time | Peak Height | Peak Area | Percent |
|---|---|---|---|---|
| 1 | 23.465 | 50921.457 | 3437527.750 | 19.1691 |
| 2 | 30.265 | 189366.859 | 14495110.000 | 80.8309 |
| **Total** | | 240288.316 | 17932637.750 | 100.0000 |

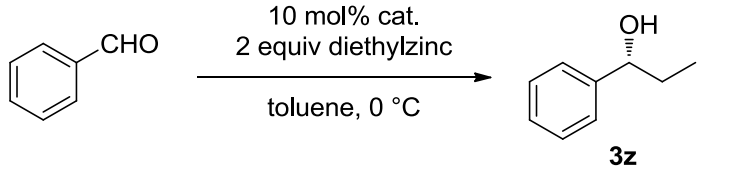
When **(-)-4h** was used as catalyst, the general procedure was followed with **(+)-4a** (18.7 mg, 0.03 mmol) and the benzaldehyde (31.9 mg, 0.3 mmol) to give (*R*)-1-phenylbut-3-en-1-ol (27.3 mg, 62% yield, 28% ee). Enantiomeric excess was determined by HPLC with a Daicel Chiralpak OD-H, n-hexane/2-propanol = 99/1, v = 1.0 mL·min$^{-1}$, λ = 220 nm, t (minor) = 31.15 min, t (major) = 22.75 min; $[\alpha]_D^{20}$ = +16.7 (c = 0.49, $CHCl_3$).




| Peak No. | R. Time | Peak Height | Peak Area | Percent |
|---|---|---|---|---|
| 1 | 22.748 | 359178.375 | 17081162.000 | 63.8298 |
| 2 | 31.148 | 169854.828 | 9679334.000 | 36.1702 |
| **Total** | | 529033.203 | 26760496.000 | 100.0000 |

**General Procedure for the Asymmetric Addition of Diethylzinc to Benzaldehyde Catalyzed by Axially Chiral Pyridine *N*-Oxides**

The catalysts were recovered in >85% yield without racemization.

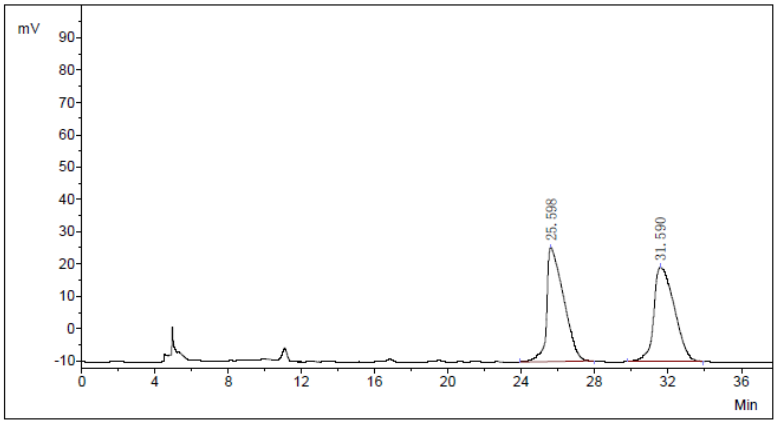



**3z**

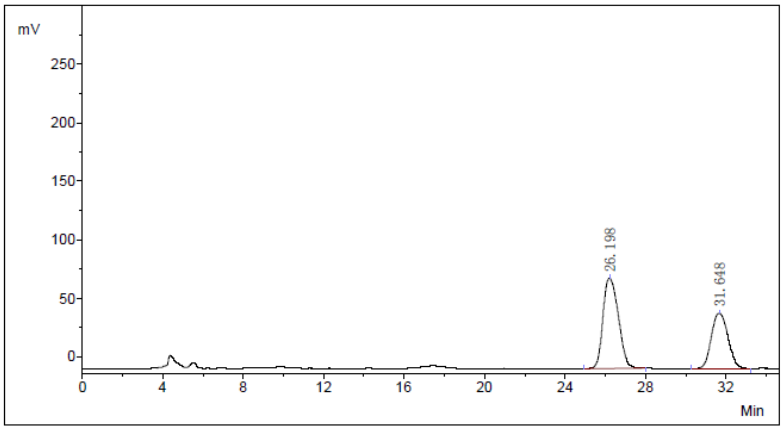
16% yield, 20% ee

To a solution of **(*R*)-(+)-4h** (20.6 mg, 0.034 mmol) in dry toluene (2 mL) at -78 °C under $N_2$, $Et_2Zn$ (1 M in hexane, 0.68 mL,0.68 mmol) was added. The mixture was stirred for 10 min. Benzaldehyde (0.034 mL, 0.34 mmol) was then added. The reaction mixture was then immediately cooled to 0 °C and stirred for 48 h under $N_2$. Saturated aqueous $NH_4Cl$ (5 mL) was added and the mixture was extracted with EtOAc (10 mL x 3). The extracts were washed with brine and dried with $Na_2SO_4$. The solvent was removed in vacuo. Purification by column chromatography on silica gel with a mixture of hexane/EtOAc (15:1) as the eluent afforded 1-phenyl-1-propanol (**3z**) (7.5 mg, 16% yield, 20% ee) as a colorless liquid. $^1H$ NMR (400 MHz, $CDCl_3$) δ

7.34-7.23 (m, 5H), 4.54 (t, *J* = 6.4 Hz, 1H), 2.17 (s, 1H), 1.90-1.63 (m, 2H), 0.89 (t, *J* = 7.2 Hz, 3H).[7] Enantiomeric excess was determined by HPLC with a Daicel Chiralpak OD-H, n-hexane/2-propanol = 99/1, v = 1.0 mL·min$^{-1}$, λ = 220 nm, t (major) = 26.20 min, t (minor) = 31.65 min; $[\alpha]_D^{20}$ = 3.1 (c = 0.13, $CHCl_3$).



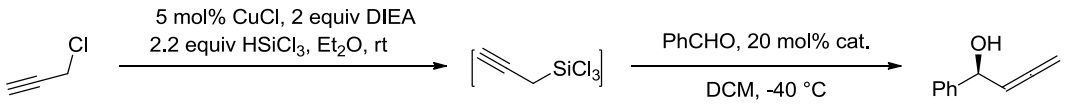

| No. | PeakNo | ID.Name | R.Time | PeakHeight | PeakArea | PerCent |
|---|---|---|---|---|---|---|
| 1 | 1 | Unknown | 25.598 | 35296.2 | 2266459.1 | 49.9393 |
| 2 | 2 | Unknown | 31.590 | 29107.8 | 2271969.1 | 50.0607 |
| Total | | | | 64404.1 | 4538428.3 | 100.0000 |




| No. | PeakNo | ID.Name | R.Time | PeakHeight | PeakArea | PerCent |
|---|---|---|---|---|---|---|
| 1 | 1 | Unknown | 26.198 | 77222.0 | 4106792.7 | 59.9734 |
| 2 | 2 | Unknown | 31.648 | 47665.1 | 2740895.9 | 40.0266 |
| Total | | | | 124887.1 | 6847688.6 | 100.0000 |

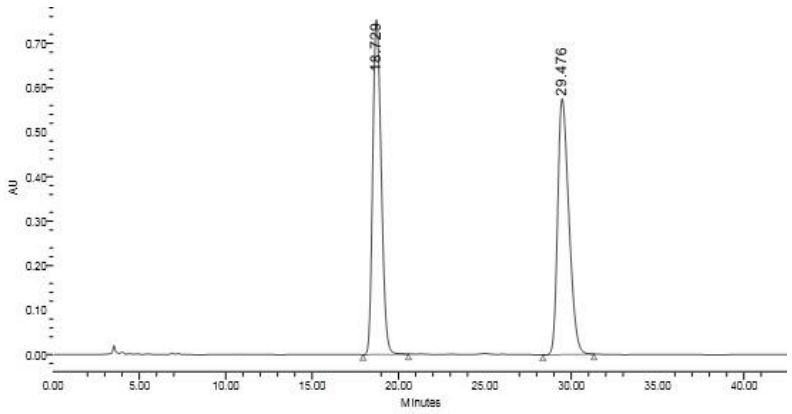
**General Procedure for the Enantioselective Allenylation of Aldehydes with Propargyltrichlorosilane Catalyzed by Axially Chiral Pyridine *N*-Oxides**

The catalysts were recovered in >85% yield without racemization.

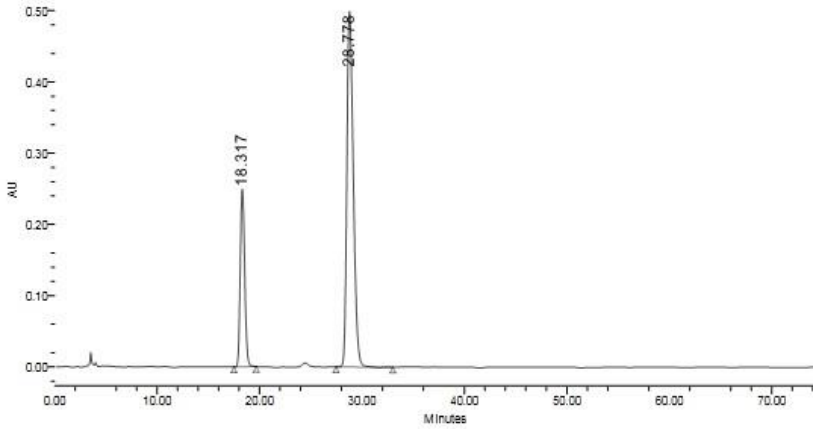



21.1% yield, 49% ee

Trichlorosilane (0.44 mL, 2.2 equiv.) was added to a stirred solution of propargyl chloride (149.2 mg, 1.0 equiv.) and cuprous chloride (10 mg, 5 mol%) in diethyl ether (4 mL) at rt for 6 h under Ar. The solvent was evaporated by rotary evaporator in vacuo. The residue was dissolved in dichloromethane (1 mL) and cooled to −40 ℃. To this solution, a solution of benzaldehyde (26.5 mg) and **(*R*)-(+)-4h** (30.3 mg) in dichloromethane (1 mL) was added via Syringe and the whole mixture was stirred for 48 h at the same temperature. The reaction was quenched with saturated $NaHCO_3$. The mixture was extracted with diethyl ether, and the organic extract was successively washed with brine and dried over anhydrous $Na_2SO_4$. After filtration and evaporation in vacuo, purification by column chromatography on silica gel with a mixture of hexane/EtOAc (15:1) as the eluent afforded 1-Phenyl-2,3-butadien-1-ol (**3w**) (7.7 mg, 21 % yield, 49 % ee) as a colorless liquid. $^1$H NMR (400 MHz, $CDCl_3$) δ 7.46-7.18 (m, 5 H), 5.43 (q, J = 6.4 Hz, 1 H), 5.25 (d, J = 6.4 Hz, 1 H), 4.89-4.98 (m, 2 H), 2.31 (s, 1 H).[8] Enantiomeric excess was determined by HPLC with a Daicel Chiralpak OD-H, n-hexane/2-propanol = 98/2, v = 1.0 mL·min$^{-1}$, λ = 220 nm, t (minor) = 18.32 min, t (major) = 28.78 min; $[\alpha]_D^{20}$ = 6.6 (c = 0.23, $CHCl_3$).




| | RT (min) | Area (µV*sec) | % Area | Height (µV) | % Height |
|---|---|---|---|---|---|
| 1 | 18.729 | 25799463 | 49.82 | 753424 | 56.69 |
| 2 | 29.476 | 25982018 | 50.18 | 575701 | 43.31 |




| | RT (min) | Area (µV*sec) | % Area | Height (µV) | % Height |
|---|---|---|---|---|---|
| 1 | 18.317 | 7463368 | 25.54 | 249219 | 33.30 |
| 2 | 28.778 | 21763999 | 74.46 | 499192 | 66.70 |

X-ray of **5a**

**5a** was prepared by adding acetyl chloride into the solution of **3a** in $Et_2O$. Yellow solid was then formed. The solid was collected by filtration, washed three times with $Et_2O$, and finally dried in vacuo. The crystalline was then obtained by adding one drop of methanol in the saturated solution of **5a** in DCM.

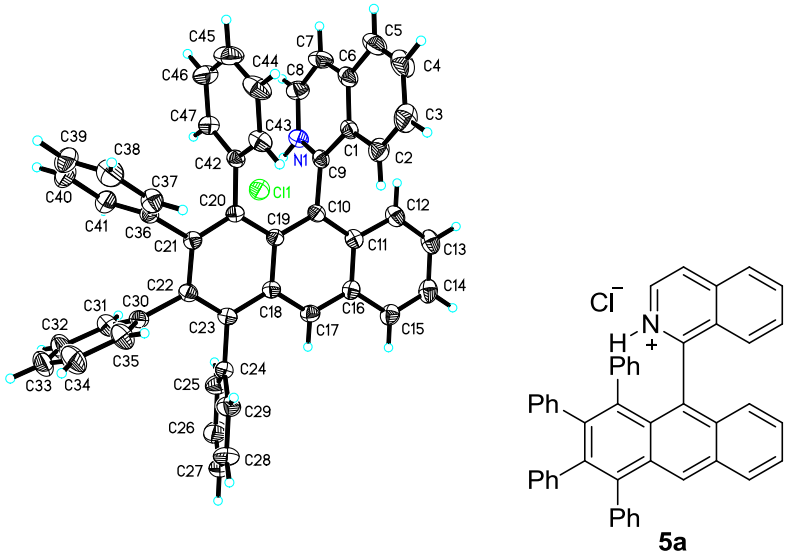
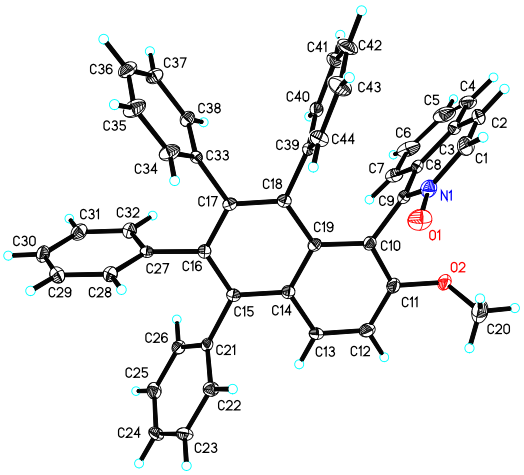


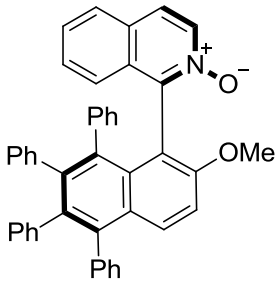

Table 1. Crystal data and structure refinement for cd213199.

| | |
|---|---|
| Identification code | cd213199 |
| Empirical formula | C47 H32 Cl N |
| Formula weight | 646.19 |
| Temperature | 293(2) K |
| Wavelength | 0.71073 A |
| Crystal system, space group | Triclinic, P-1 |
| Unit cell dimensions | a = 8.7922(9) A alpha = 96.760(2) deg. |
| | b = 9.5339(10) A beta = 99.441(3) deg. |
| | c = 21.234(2) A gamma = 90.583(2) deg. |
| Volume | 1742.8(3) A^3 |
| Z, Calculated density | 2, 1.231 Mg/m^3 |
| Absorption coefficient | 0.144 mm^-1 |
| F(000) | 676 |
| Crystal size | 0.211 x 0.148 x 0.112 mm |
| Theta range for data collection | 1.96 to 26.00 deg. |
| Limiting indices | -10<=h<=10, -11<=k<=11, -13<=l<=26 |
| Reflections collected / unique | 10628 / 6824 [R(int) = 0.0275] |
| Completeness to theta = 26.00 | 99.8 % |

| | |
|---|---|
| Absorption correction | Empirical |
| Max. and min. transmission | 1.00000 and 0.37002 |
| Refinement method | Full-matrix least-squares on F^2 |
| Data / restraints / parameters | 6824 / 1 / 446 |
| Goodness-of-fit on F^2 | 1.037 |
| Final R indices [I>2sigma(I)] | R1 = 0.0511, wR2 = 0.1238 |
| R indices (all data) | R1 = 0.0842, wR2 = 0.1402 |
| Largest diff. peak and hole | 0.213 and -0.177 e.A^-3 |

X-ray of **(*aR*)-(+)-4h**



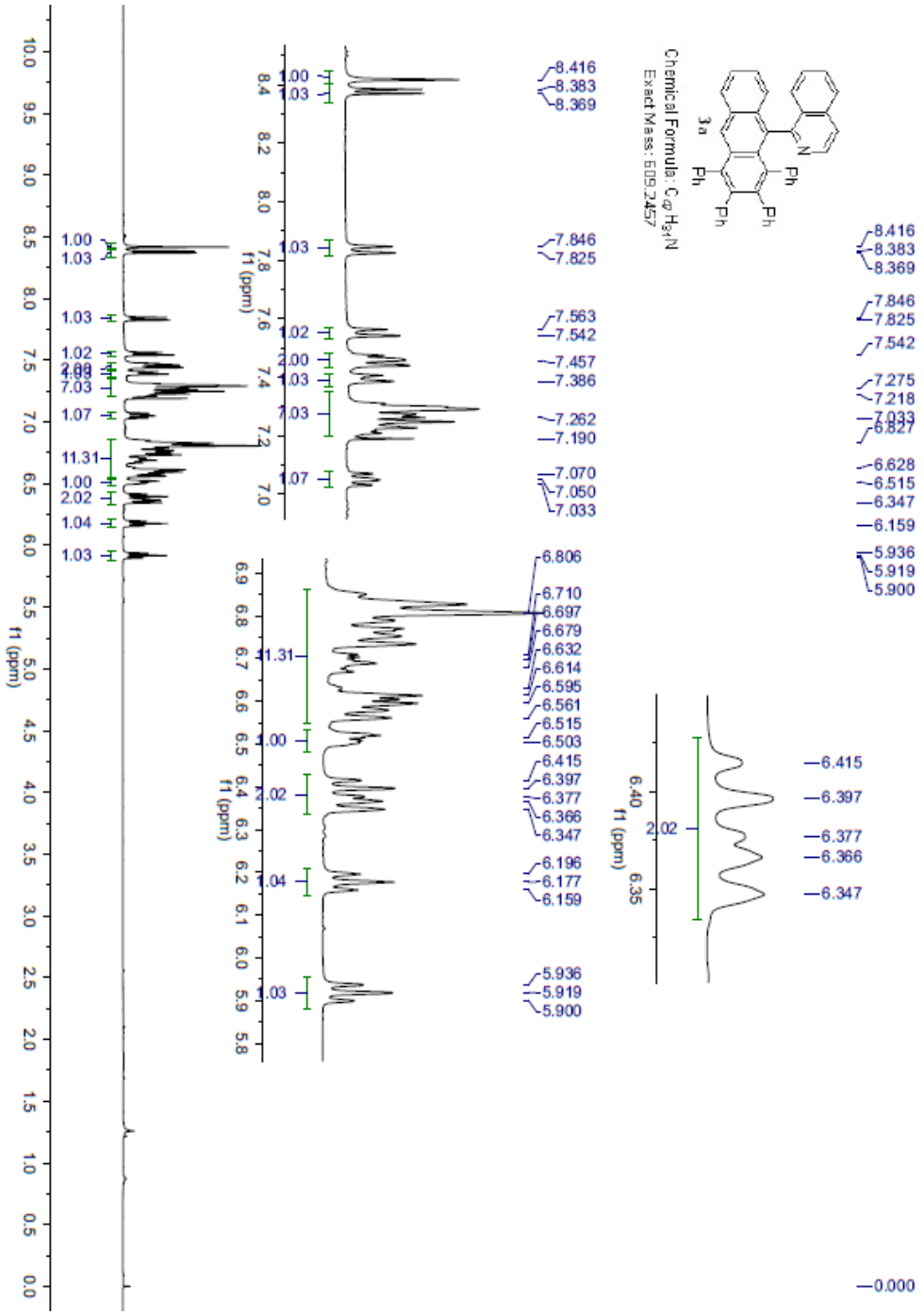

(*aR*)-(+)-**4h**

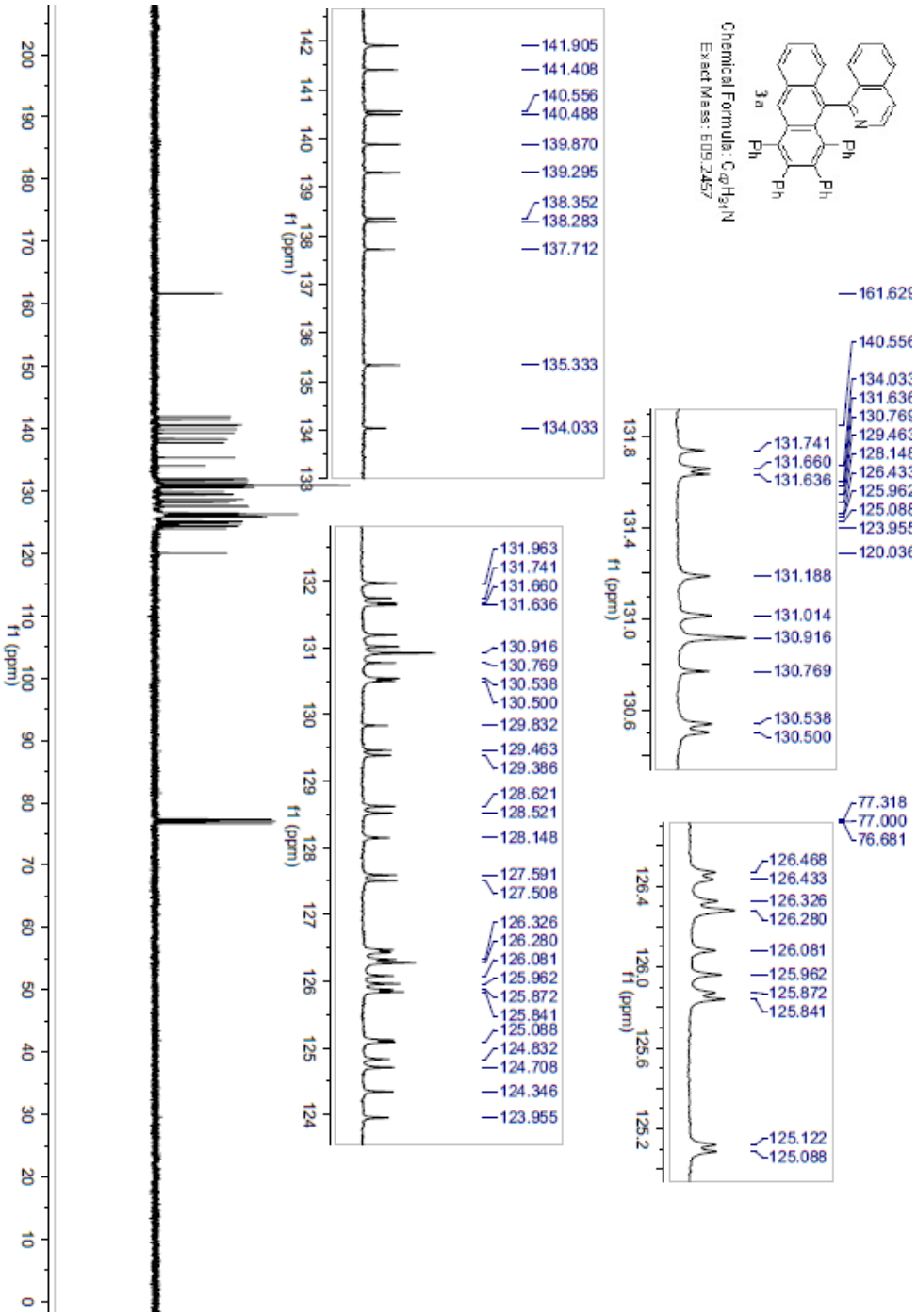
Table 2. Crystal data and structure refinement for mo_dm13272_0m.

| | |
|---|---|
| Identification code | mo_dm13272_0m |
| Empirical formula | C48 H39 N O4 |
| Formula weight | 693.80 |
| Temperature | 140(2) K |
| Wavelength | 0.71073 A |
| Crystal system, space group | Orthorhombic, P2(1)2(1)2(1) |
| Unit cell dimensions | a = 11.0511(18) A alpha = 90 deg.<br>b = 14.426(2) A beta = 90 deg.<br>c = 22.603(4) A gamma = 90 deg. |
| Volume | 3603.5(10) A^3 |
| Z, Calculated density | 4, 1.279 Mg/m^3 |
| Absorption coefficient | 0.081 mm^-1 |
| F(000) | 1464 |
| Crystal size | 0.23 x 0.13 x 0.08 mm |
| Theta range for data collection | 1.67 to 27.48 deg. |

| | |
|---|---|
| Limiting indices | -14<=h<=14, -18<=k<=18, -26<=l<=29 |
| Reflections collected / unique | 29121 / 8265 [R(int) = 0.0943] |
| Completeness to theta = 27.48 | 99.9 % |
| Absorption correction | Semi-empirical from equivalents |
| Max. and min. transmission | 0.9936 and 0.9817 |
| Refinement method | Full-matrix least-squares on F^2 |
| Data / restraints / parameters | 8265 / 0 / 481 |
| Goodness-of-fit on F^2 | 0.975 |
| Final R indices [I>2sigma(I)] | R1 = 0.0544, wR2 = 0.1027 |
| R indices (all data) | R1 = 0.1024, wR2 = 0.1184 |
| Absolute structure parameter | 1.9(14) |
| Largest diff. peak and hole | 0.219 and -0.228 e.A^-3 |

**Copies of NMR Spectra**

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Ph
Ph
Ph
N
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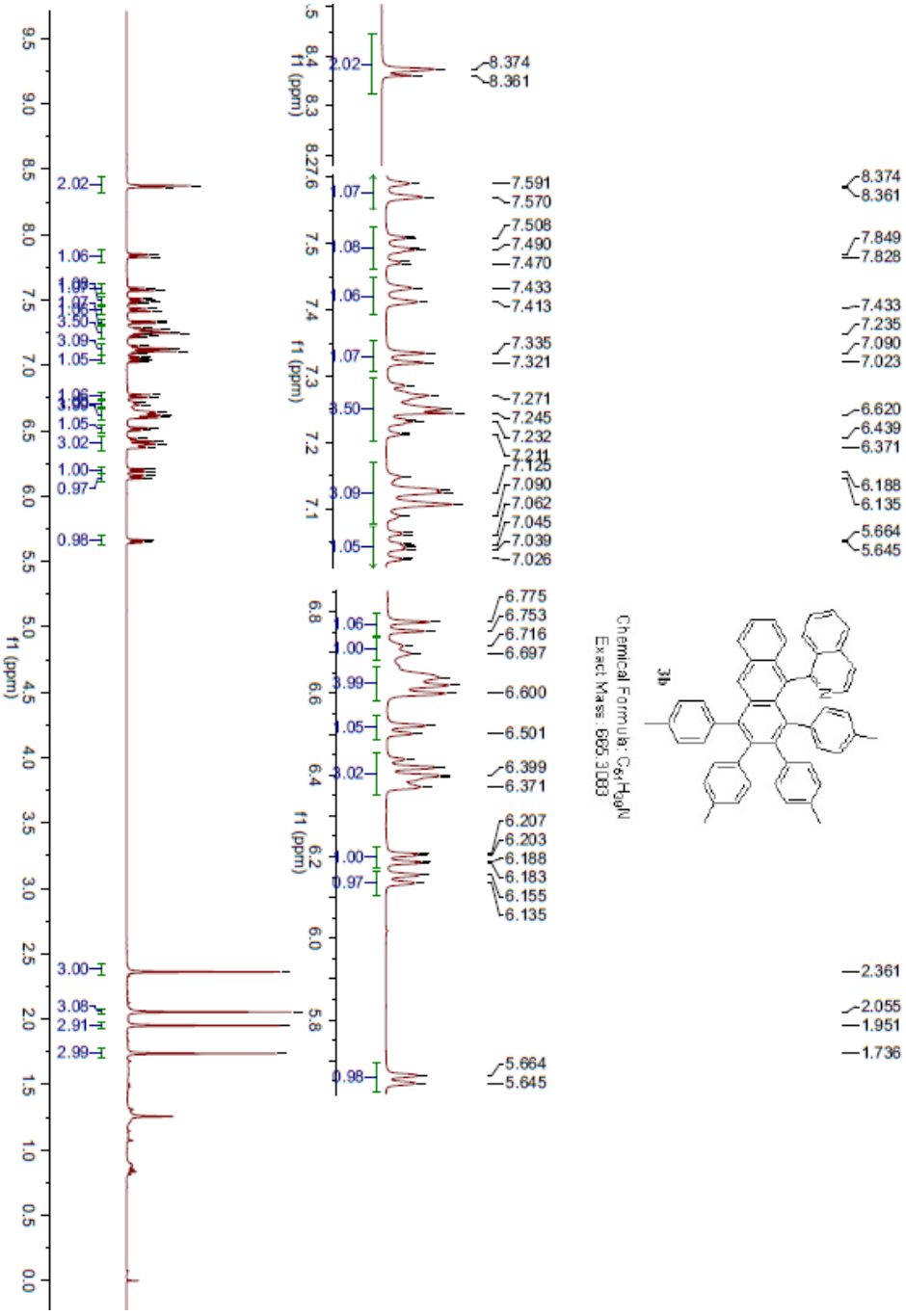
f1 (ppm)
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3.09
1.05
3.02
1.00
0.97
0.98
3.00
3.08
2.91
2.99
8.374
8.361
7.591
7.570
7.508
7.490
7.470
7.433
7.413
7.335
7.321
7.271
7.245
7.232
7.125
7.090
7.062
7.045
7.039
7.026
6.775
6.753
6.716
6.697
6.600
6.501
6.399
6.371
6.207
6.203
6.188
6.183
6.155
6.135
5.664
5.645
7.849
7.828
7.235
7.023
6.620
6.439
6.371
6.188
6.135
2.361
2.055
1.951
1.736
3b
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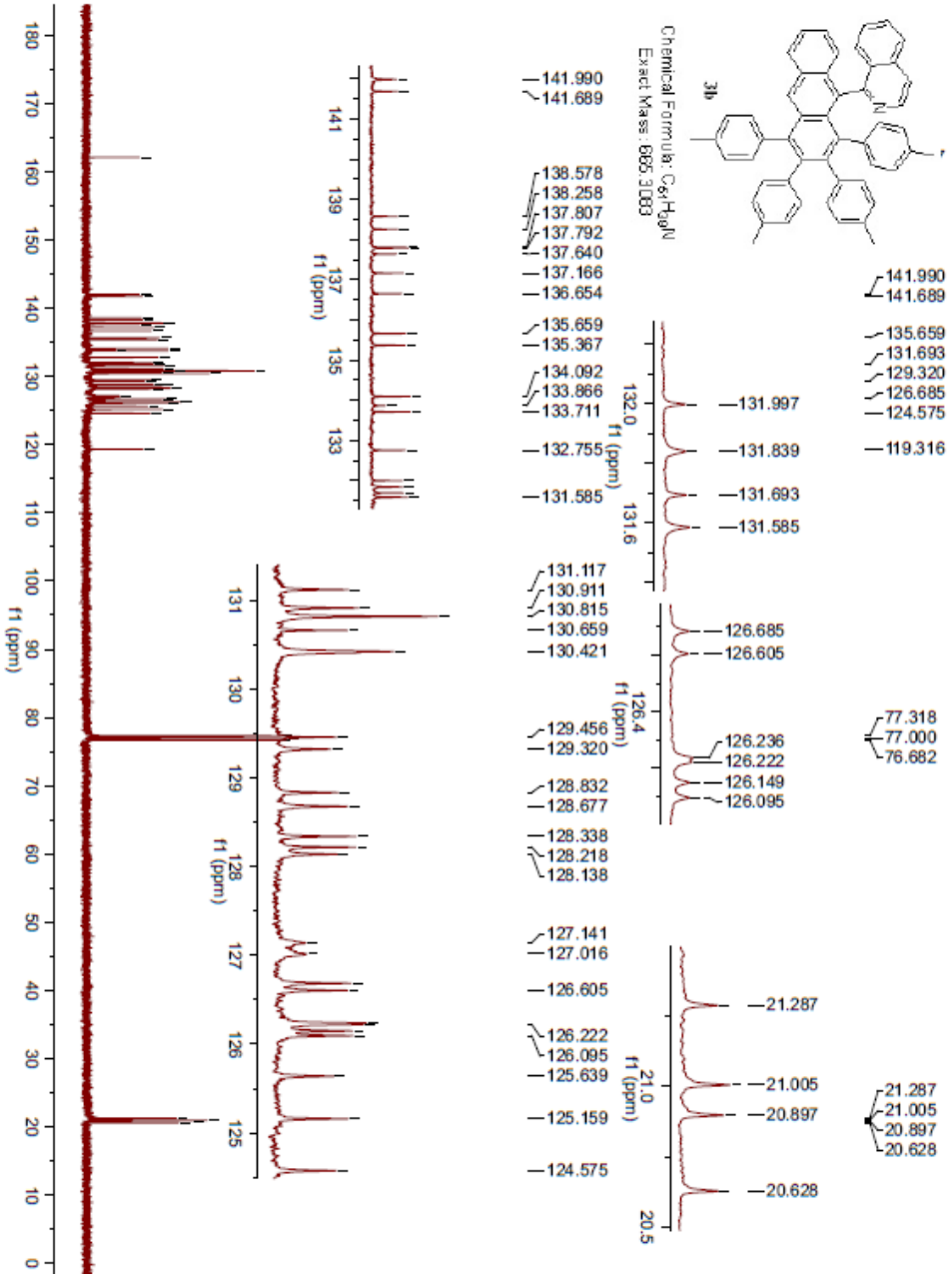
Exact Mass: 665.3083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137.166
136.654
135.659
135.367
134.092
133.866
133.711
132.755
131.585
131.117
130.911
130.815
130.659
130.421
129.456
129.320
128.832
128.677
128.338
128.218
128.138
127.141
127.016
126.605
126.222
126.095
125.639
125.159
124.575
131.997
131.839
131.693
131.585
126.685
126.605
126.236
126.222
126.149
126.095
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21.005
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135.659
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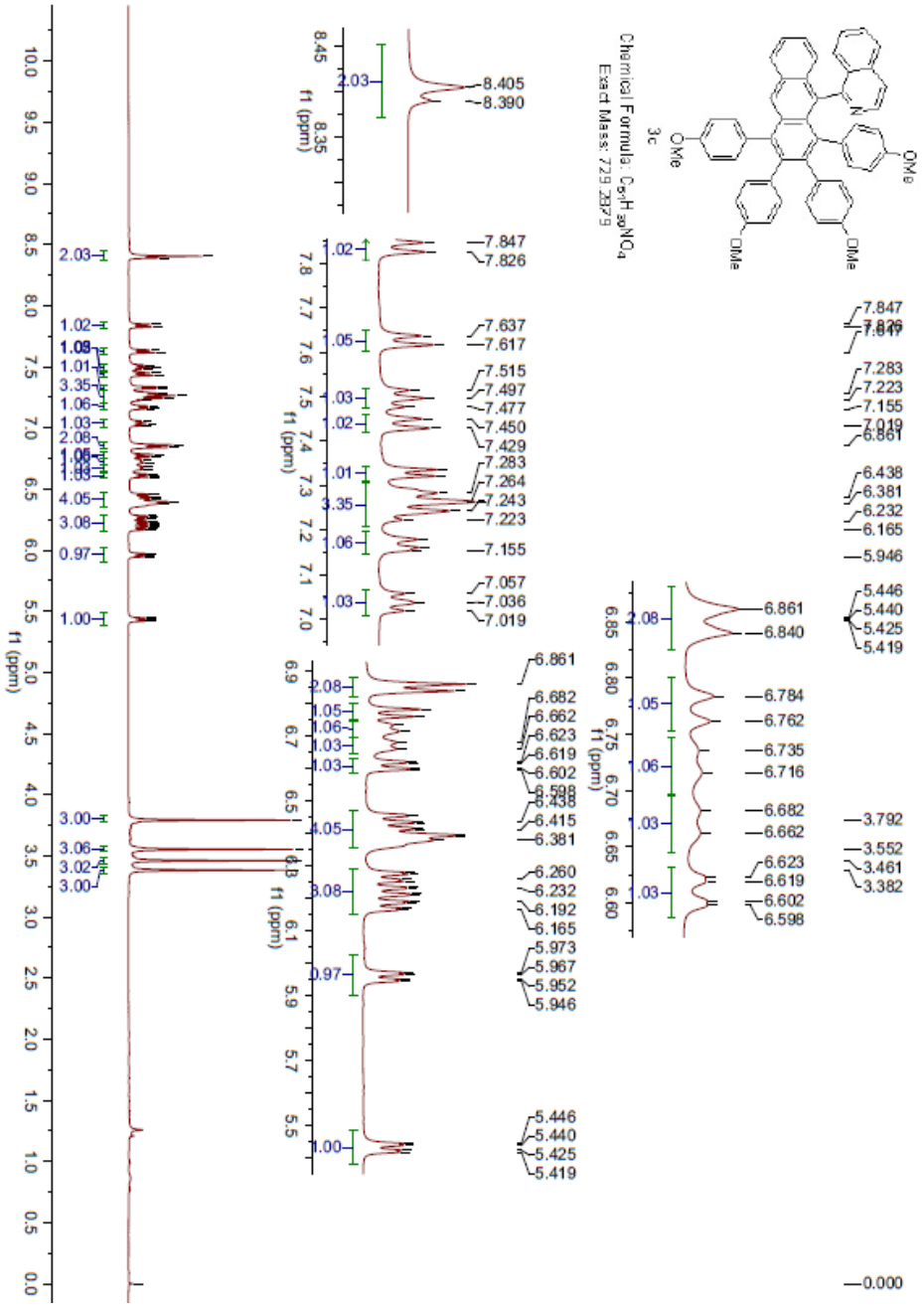
124.575
119.316
f1 (ppm)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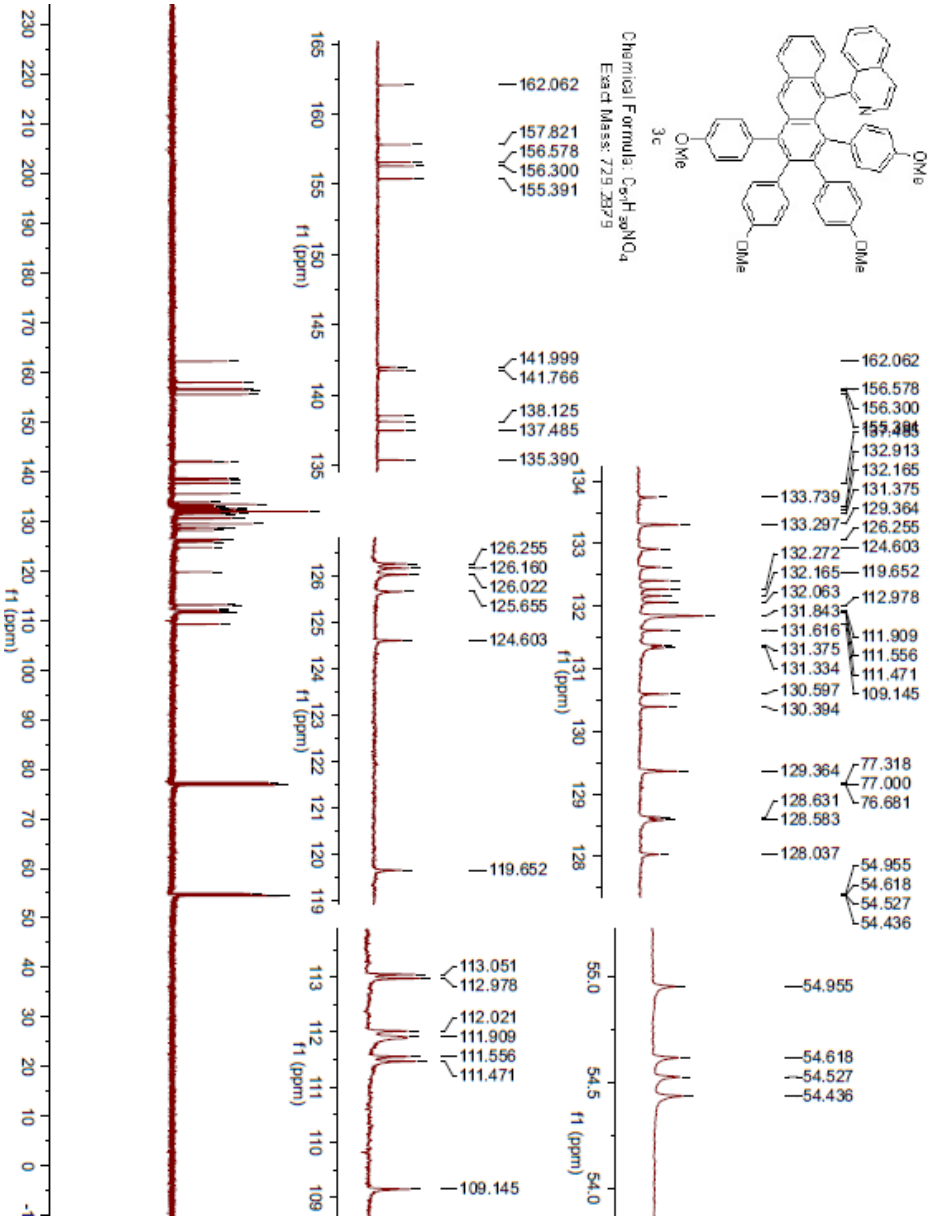
OMe
OMe
N
f1 (ppm)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OMe
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141.999
141.766
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137.485
135.390
126.255
126.160
126.022
125.655
124.603
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113.051
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111.471
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54.527
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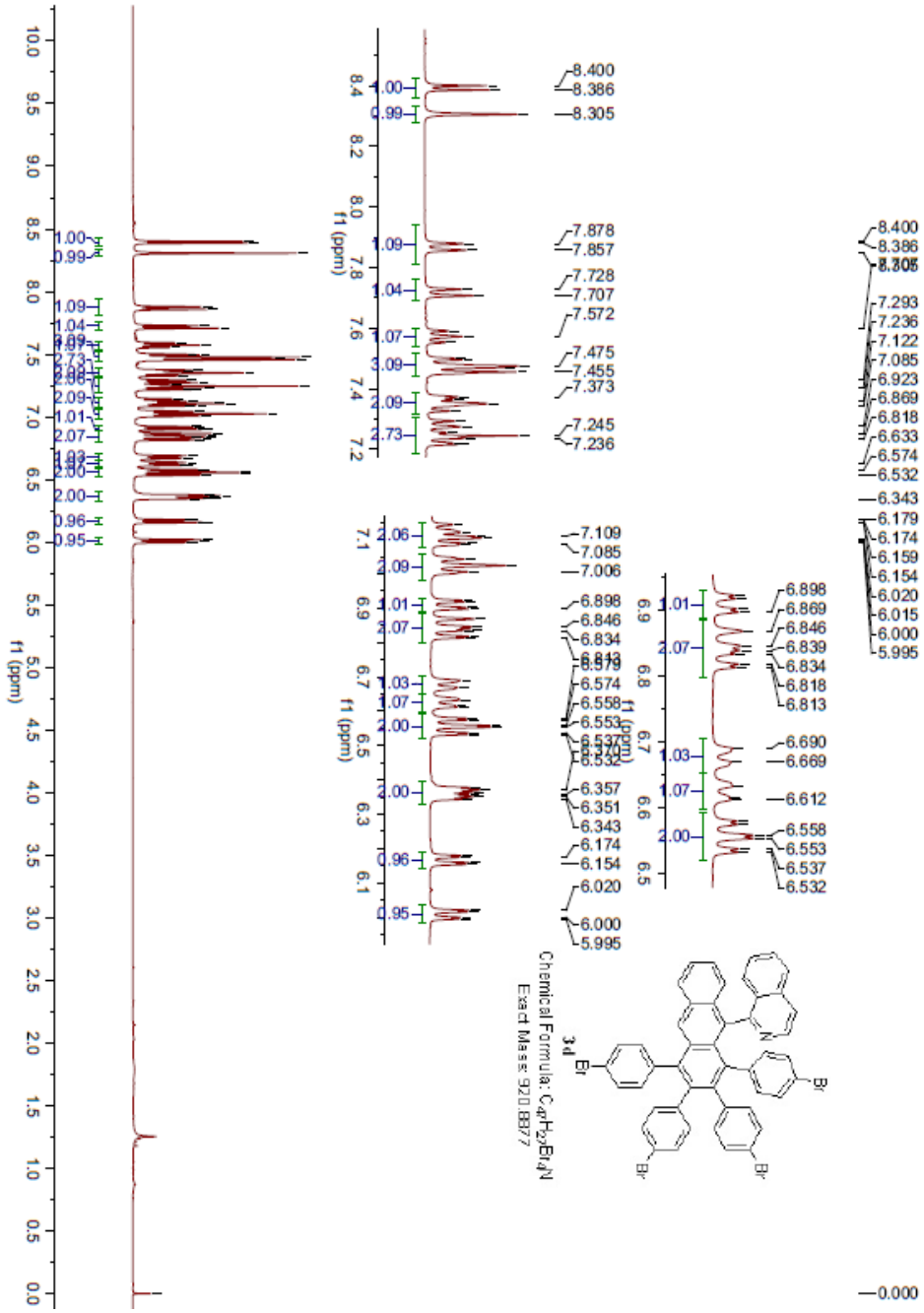
54.955
54.618
54.527
54.436
f1 (ppm)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7.109
7.085
7.006
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6.846
6.834
6.574
6.558
6.553
6.537
6.357
6.351
6.343
6.174
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6.633
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6.532
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6.174
6.159
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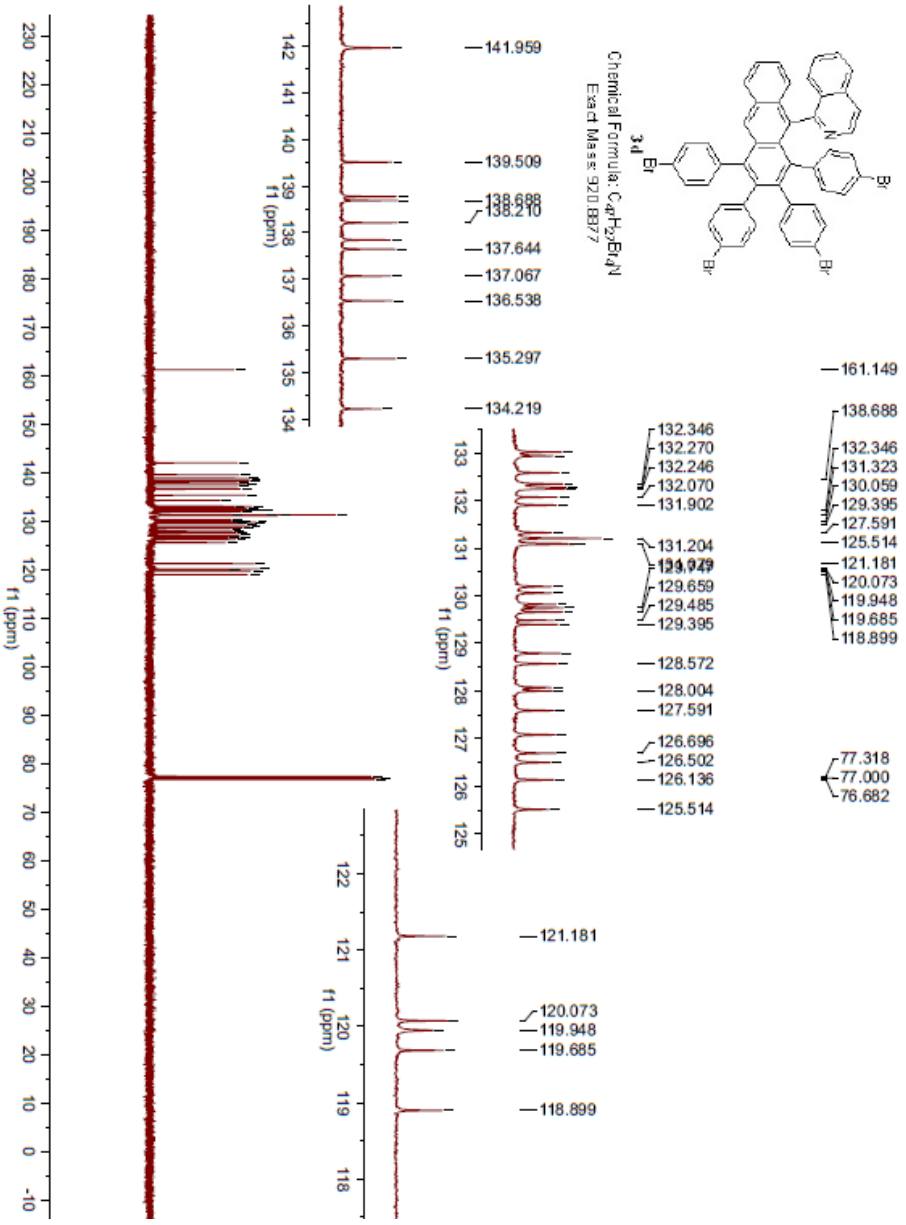
Exact Mass: 920.8877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132.246
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131.204
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125.514
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139.509
138.688
138.210
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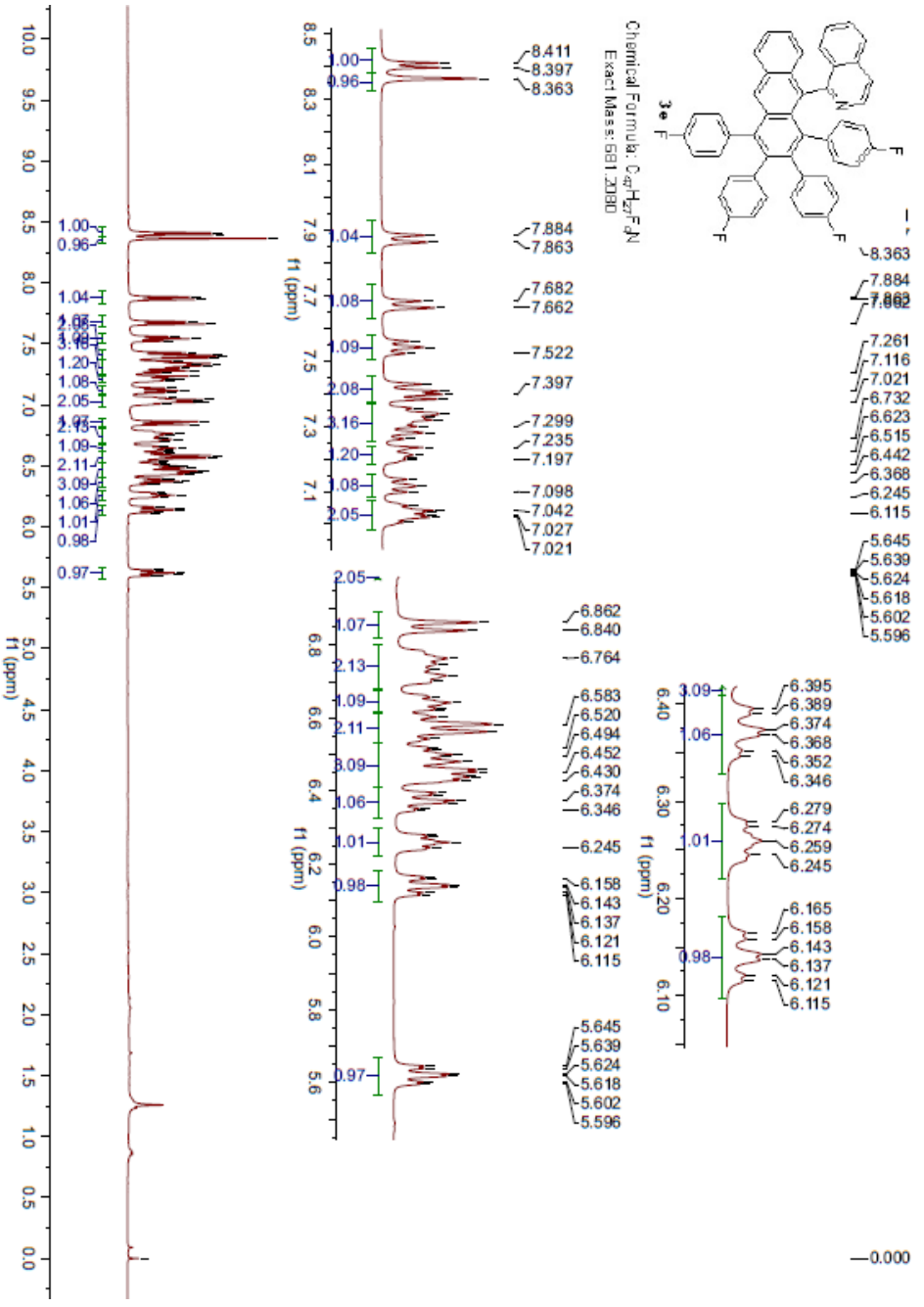
119.948
119.685
118.899
f1 (ppm)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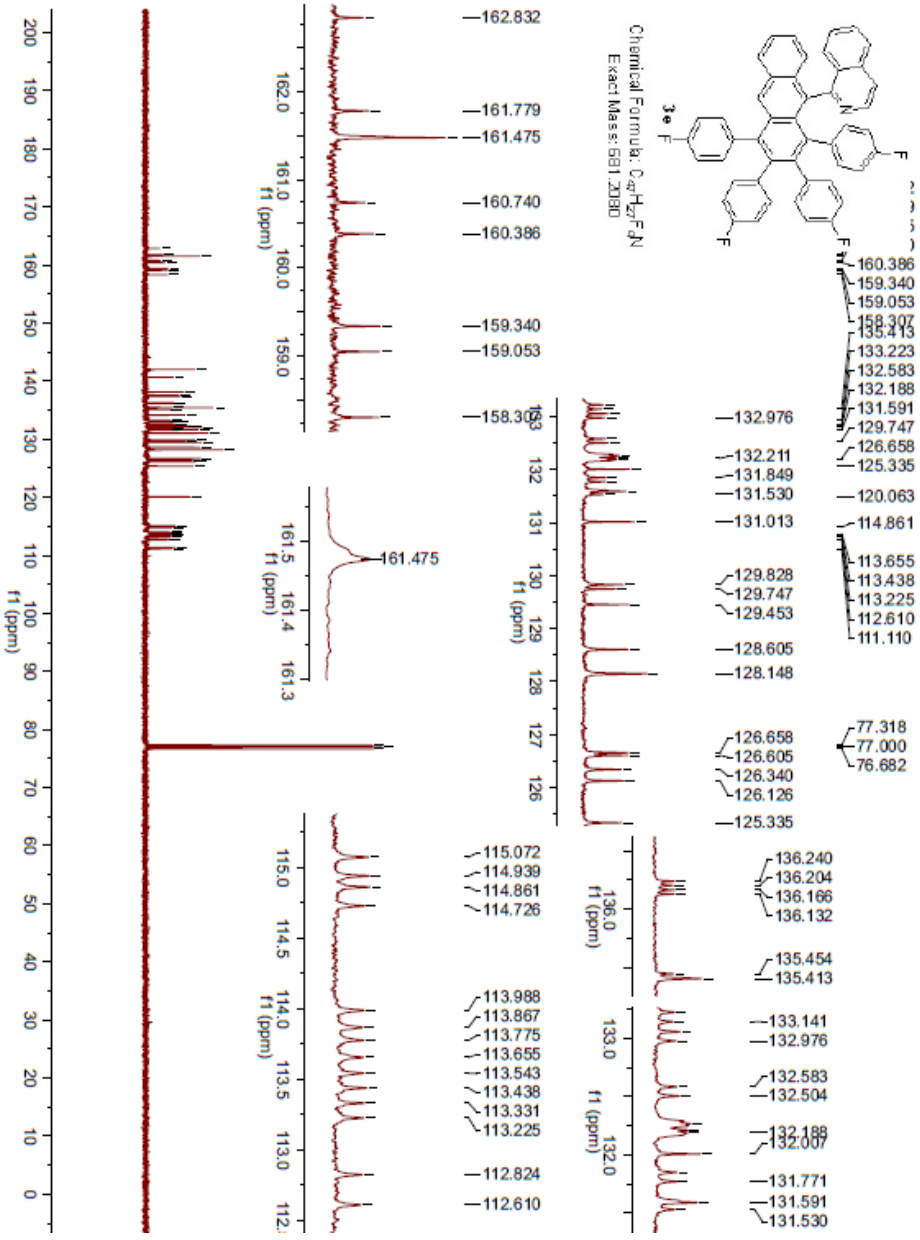
3e
f1 (ppm)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132.583
132.188
131.591
129.747
126.658
125.335
120.063
114.861
113.655
113.438
113.225
112.610
111.110
77.318
77.000
76.682
132.976
132.211
131.849
131.530
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129.828
129.747
129.453
128.605
128.148
126.658
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126.340
126.126
125.335
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136.166
136.132
135.454
135.413
133.141
132.976
132.583
132.504
132.188
132.007
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131.591
131.530
162.832
161.779
161.475
160.740
160.386
159.340
159.053
158.307
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114.861
114.726
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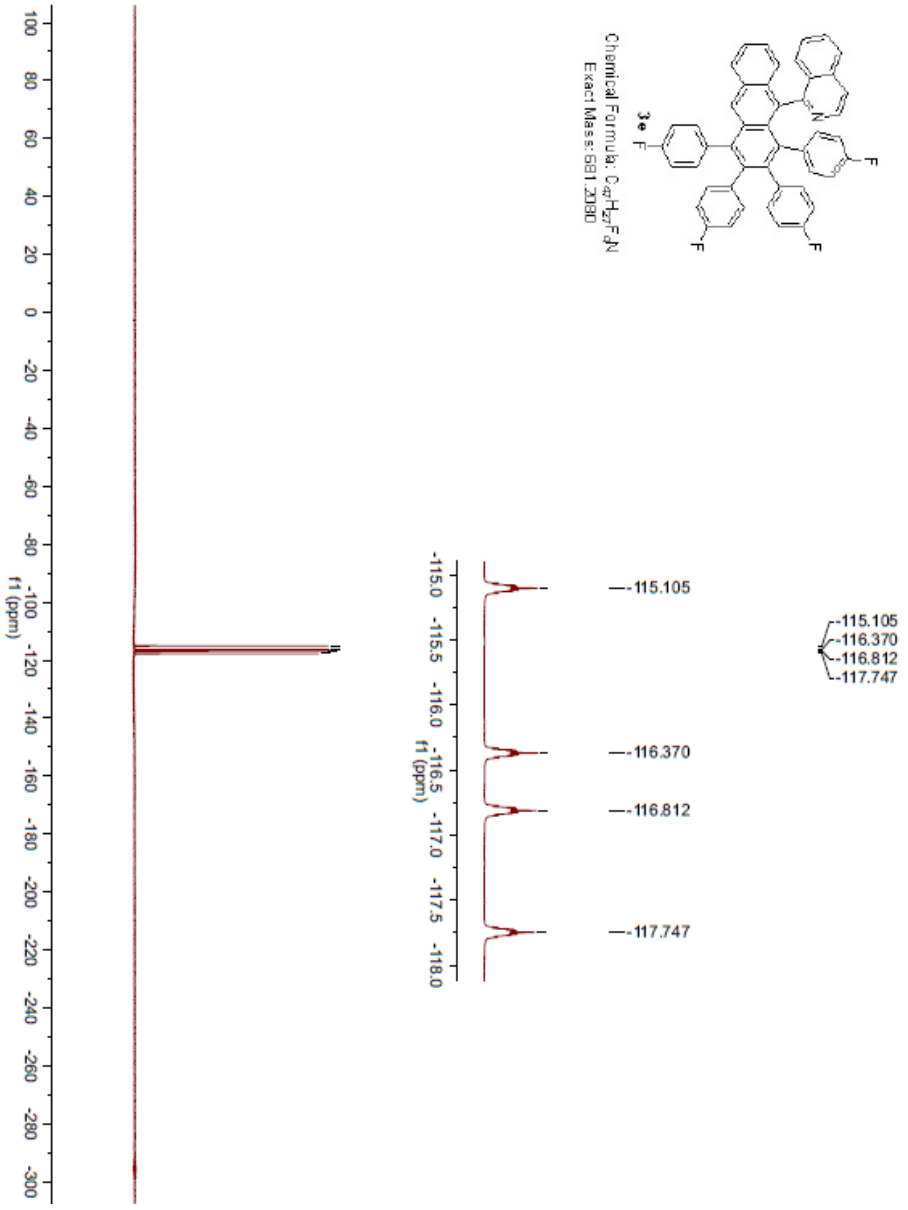
112.610
f1 (ppm)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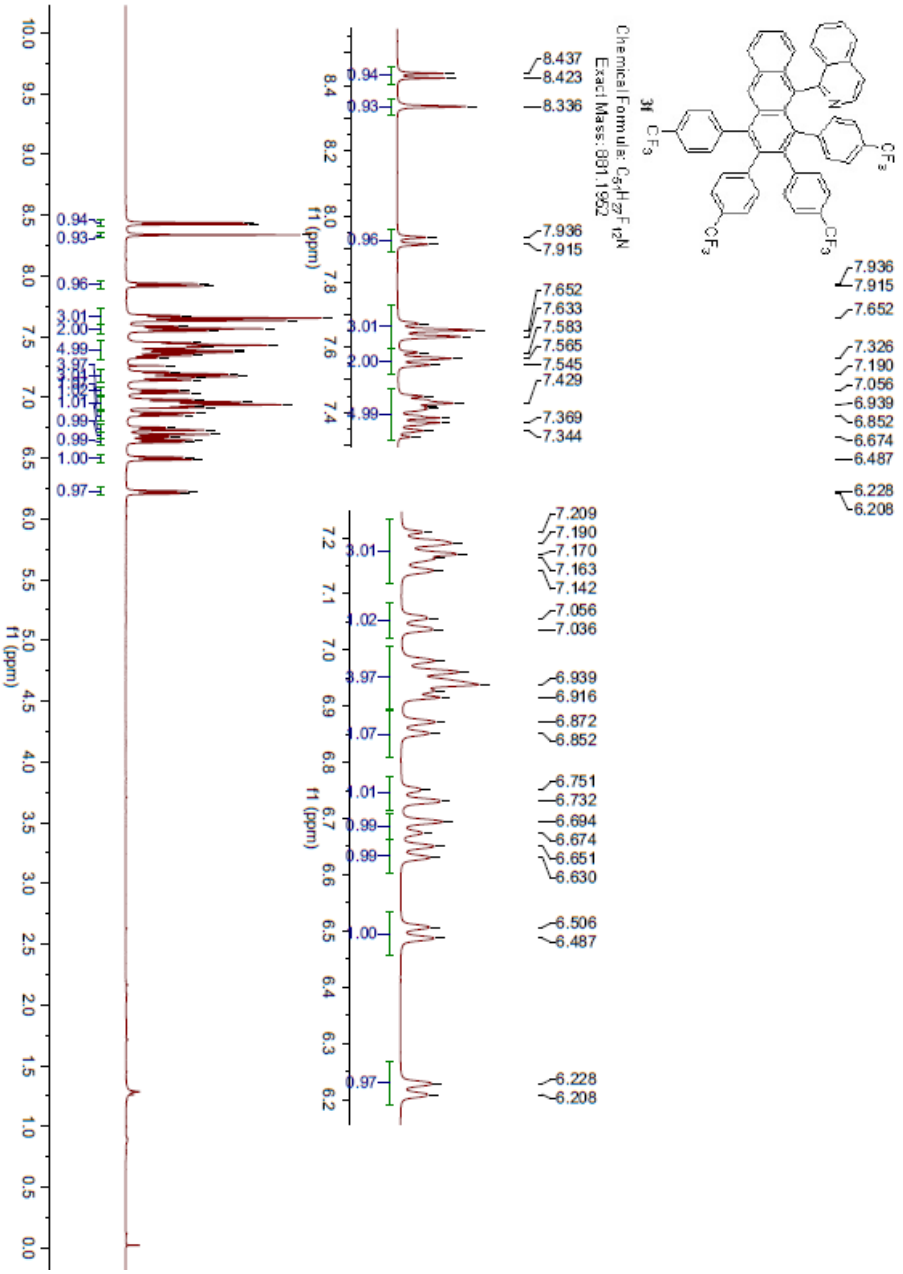
f1 (ppm)
f1 (ppm)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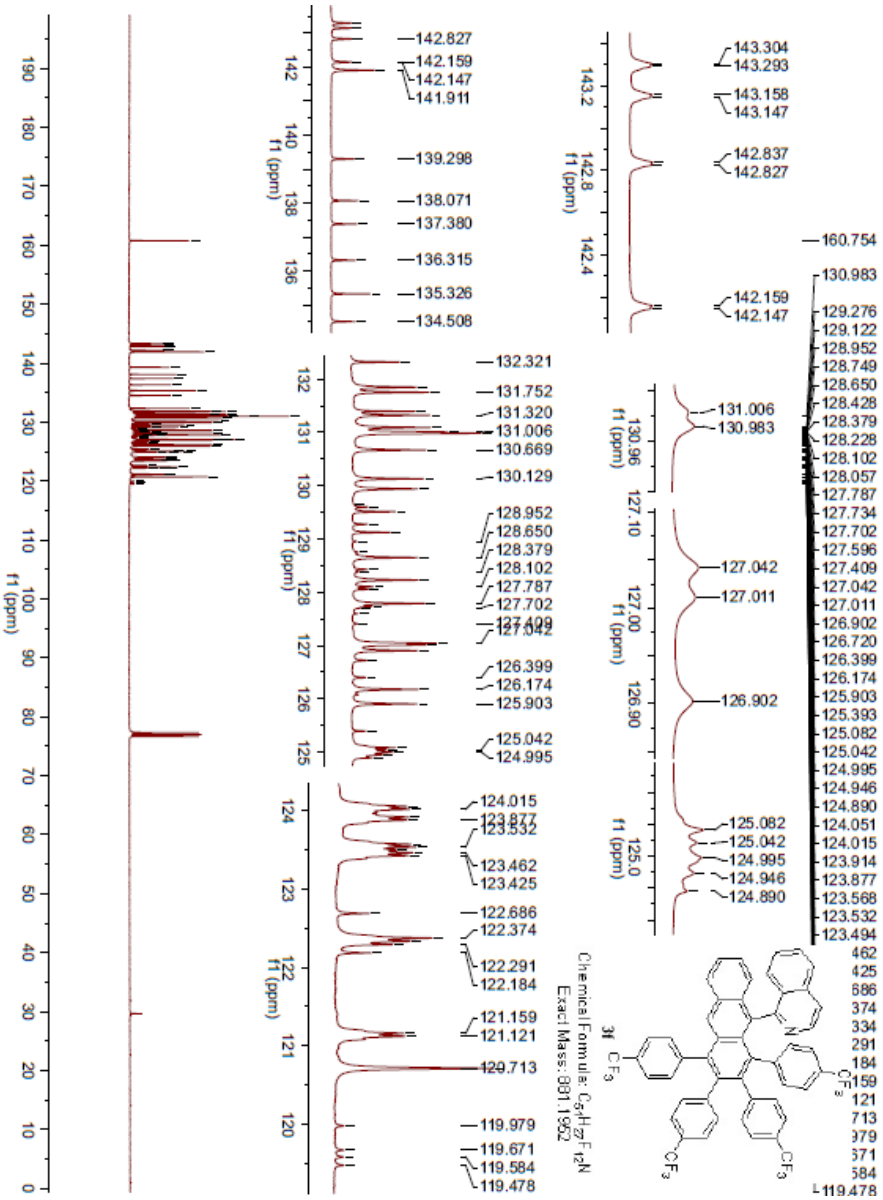
6.228
6.208
f1 (ppm)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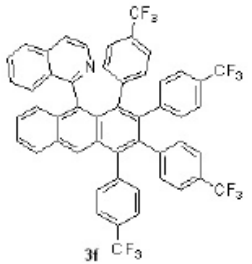
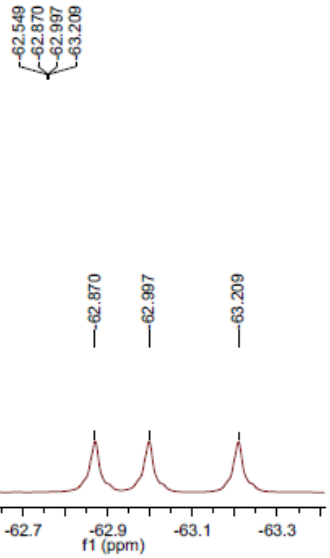
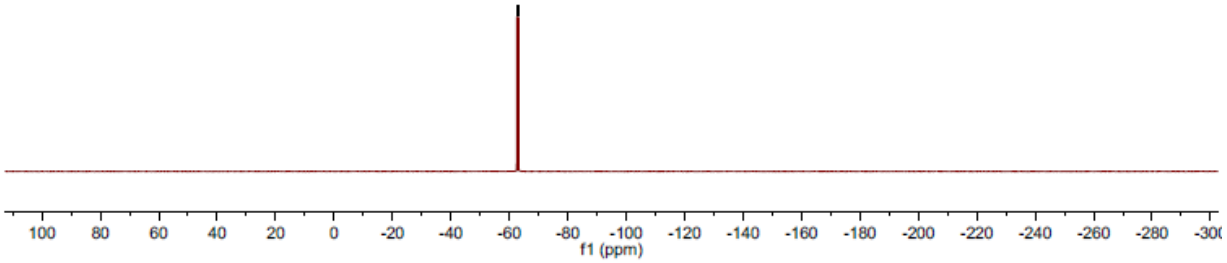
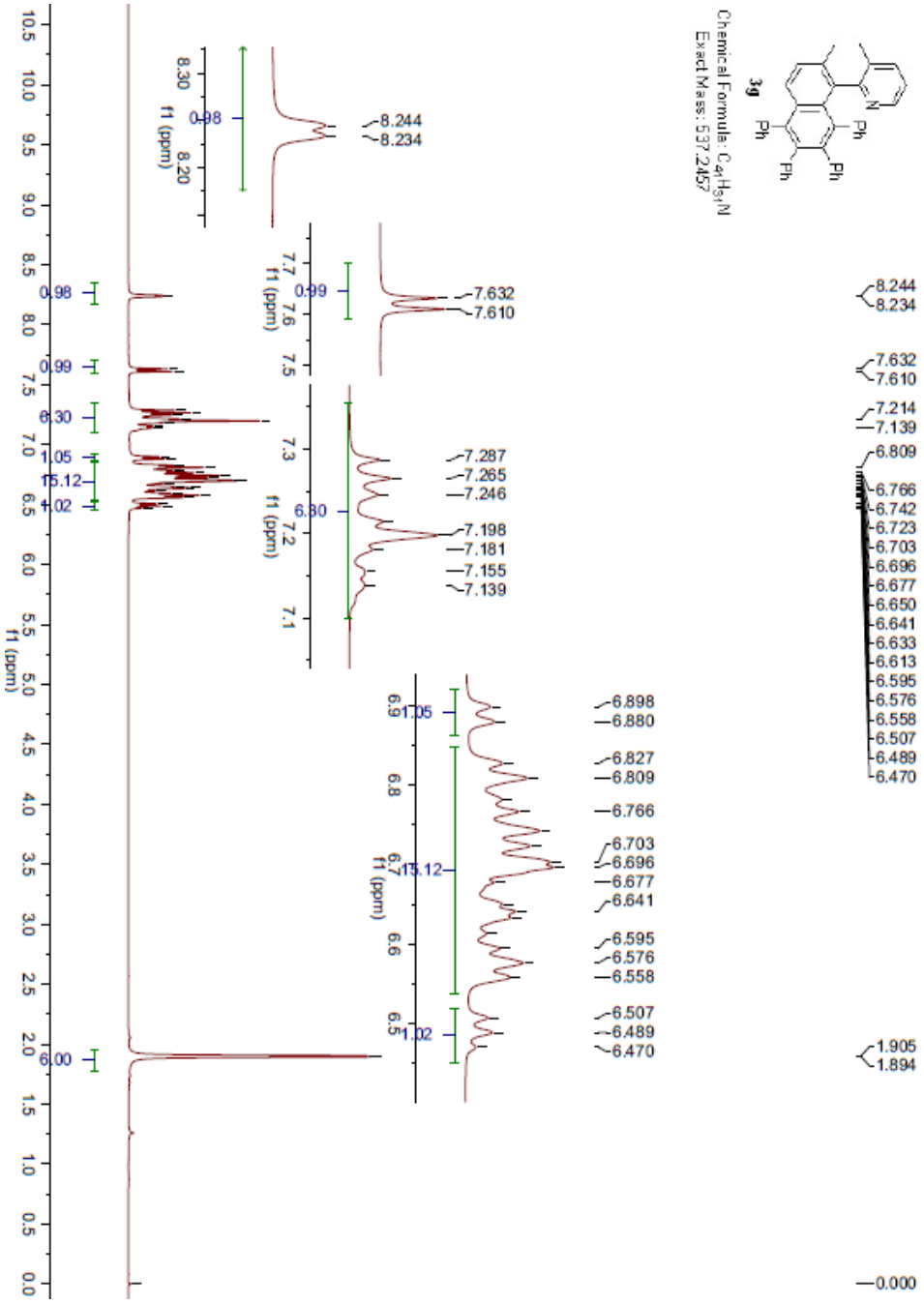


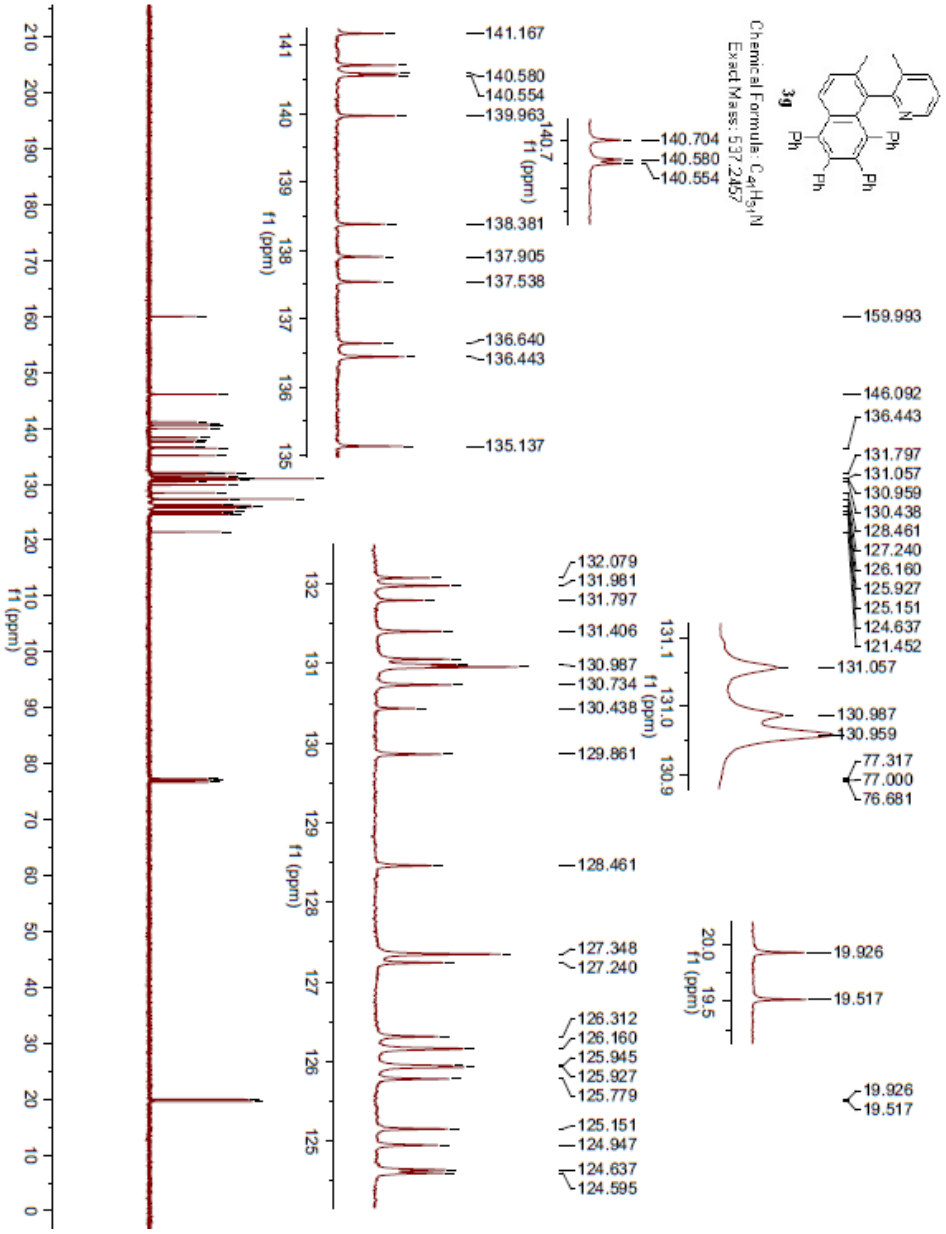

3f

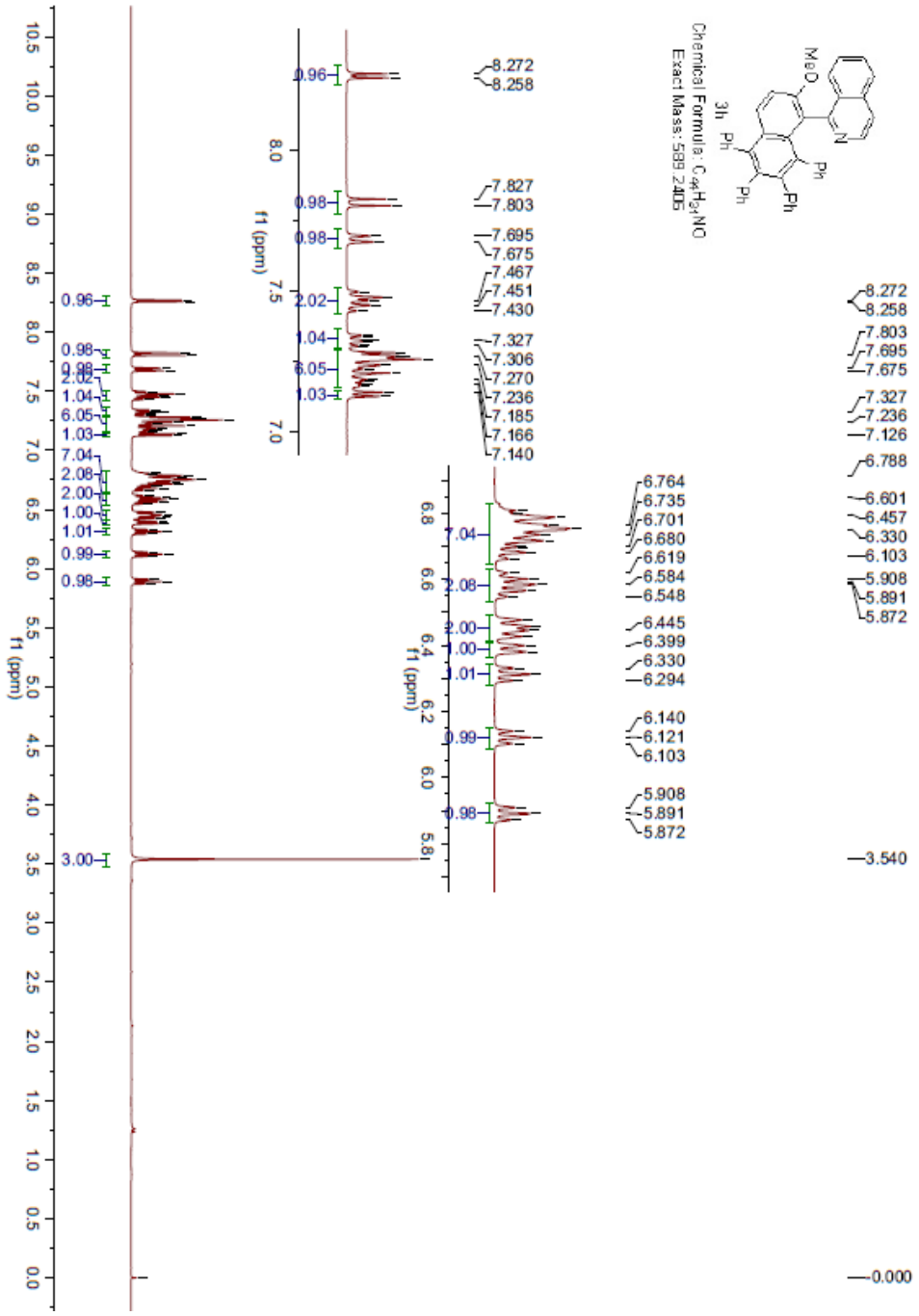
Chemical Formula: $C_{51}H_{27}F_{12}N$
Exact Mass: 881.1952

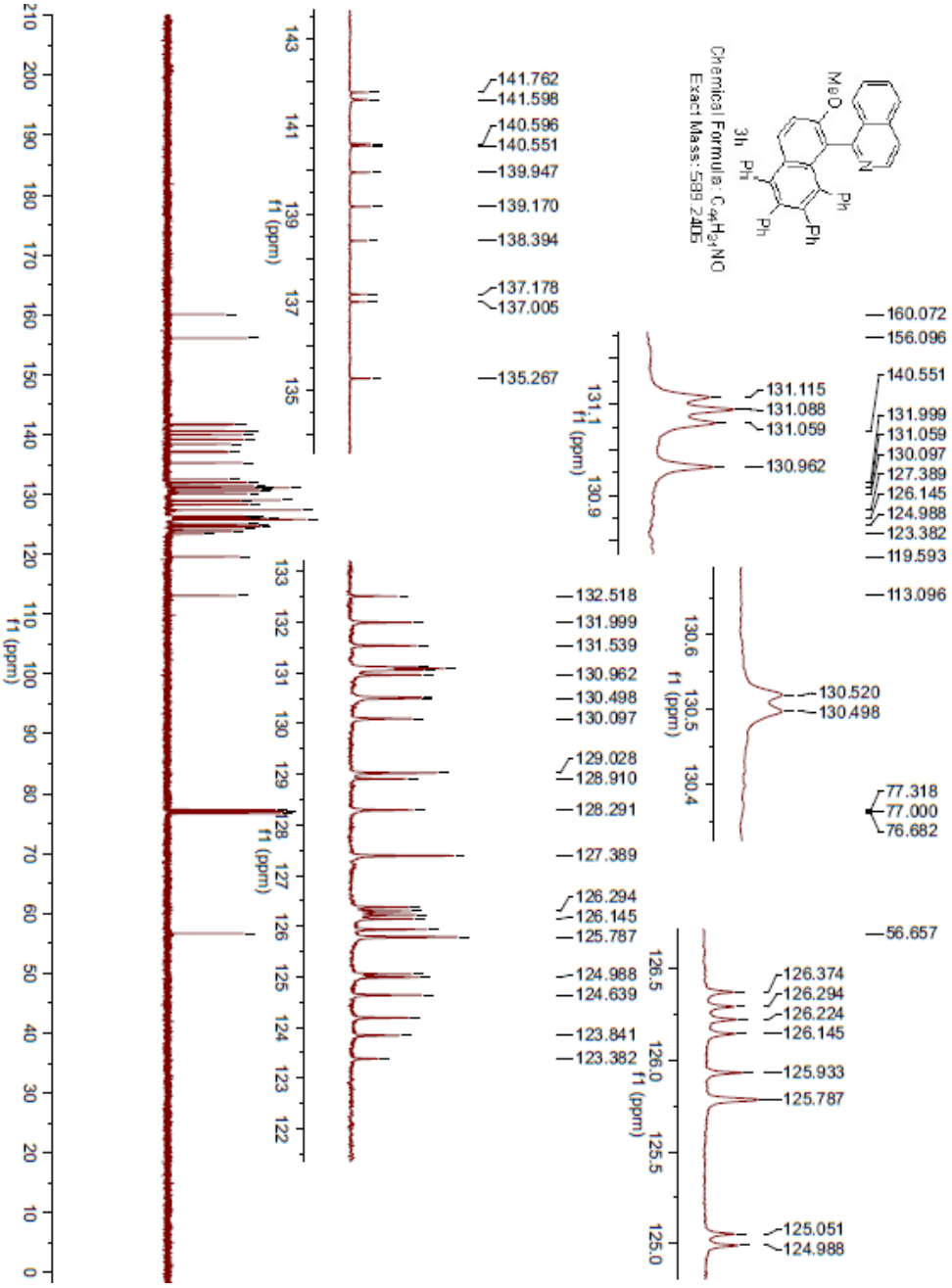


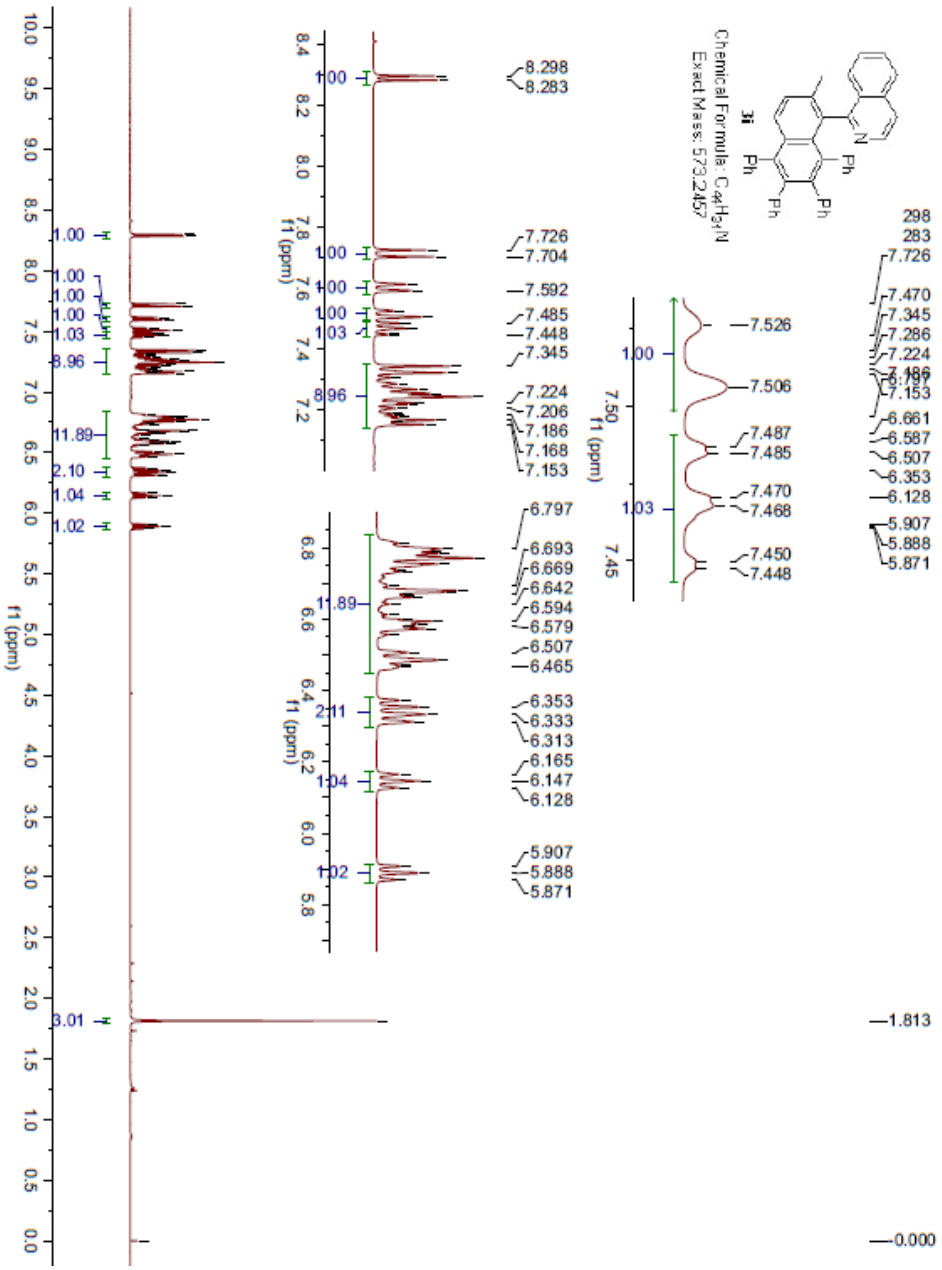



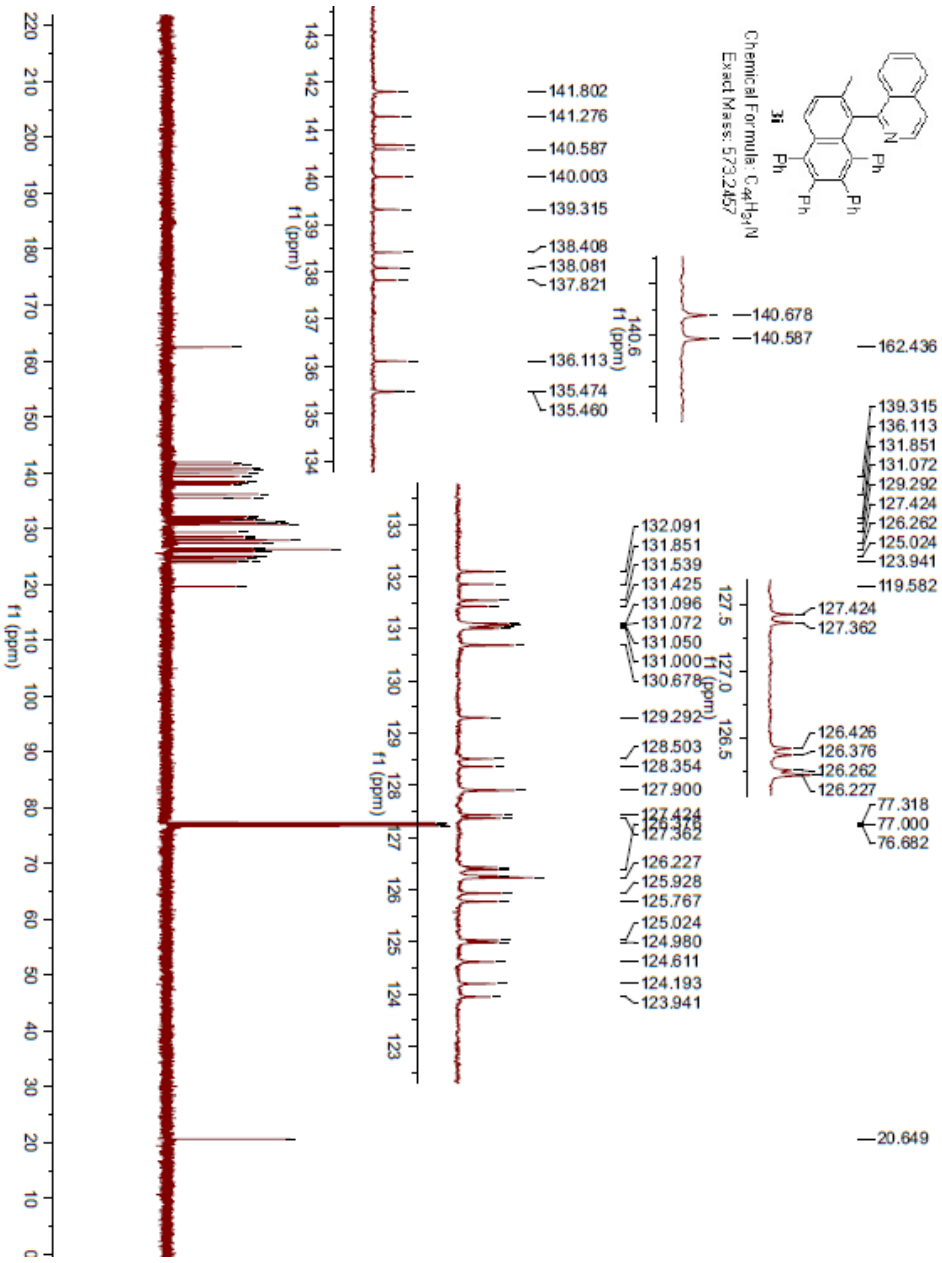

Chemical Formula: C41H31N
Exact Mass: 537.2457
3ag
Ph
Ph
Ph
Ph
N
8.244
8.234
7.632
7.610
7.214
7.139
6.809
6.766
6.742
6.723
6.703
6.696
6.677
6.650
6.641
6.633
6.613
6.595
6.576
6.558
6.507
6.489
6.470
1.905
1.894
0.000
f1 (ppm)

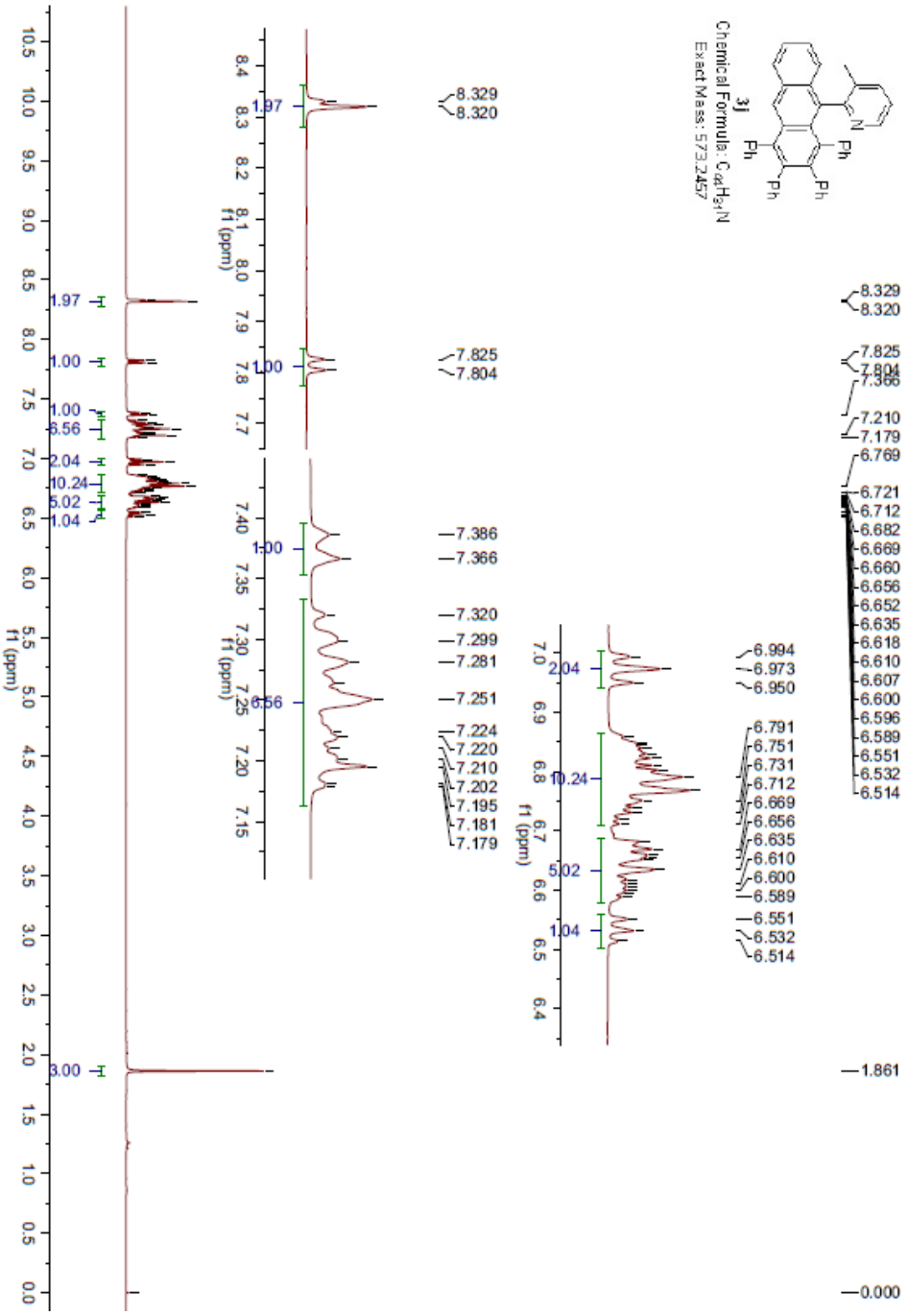

3g
Chemical Formula: C41H31N
Exact Mass: 537.2457
Ph
Ph
Ph
Ph
N
159.993
146.092
136.443
131.797
131.057
130.959
130.438
128.461
127.240
126.160
125.927
125.151
124.637
121.452
77.317
77.000
76.681
141.167
140.580
140.554
139.963
138.381
137.905
137.538
136.640
136.443
135.137
132.079
131.981
131.797
131.406
130.987
130.734
130.438
129.861
128.461
127.348
127.240
126.312
126.160
125.945
125.927
125.779
125.151
124.947
124.637
124.595
19.926
19.517
f1 (ppm)

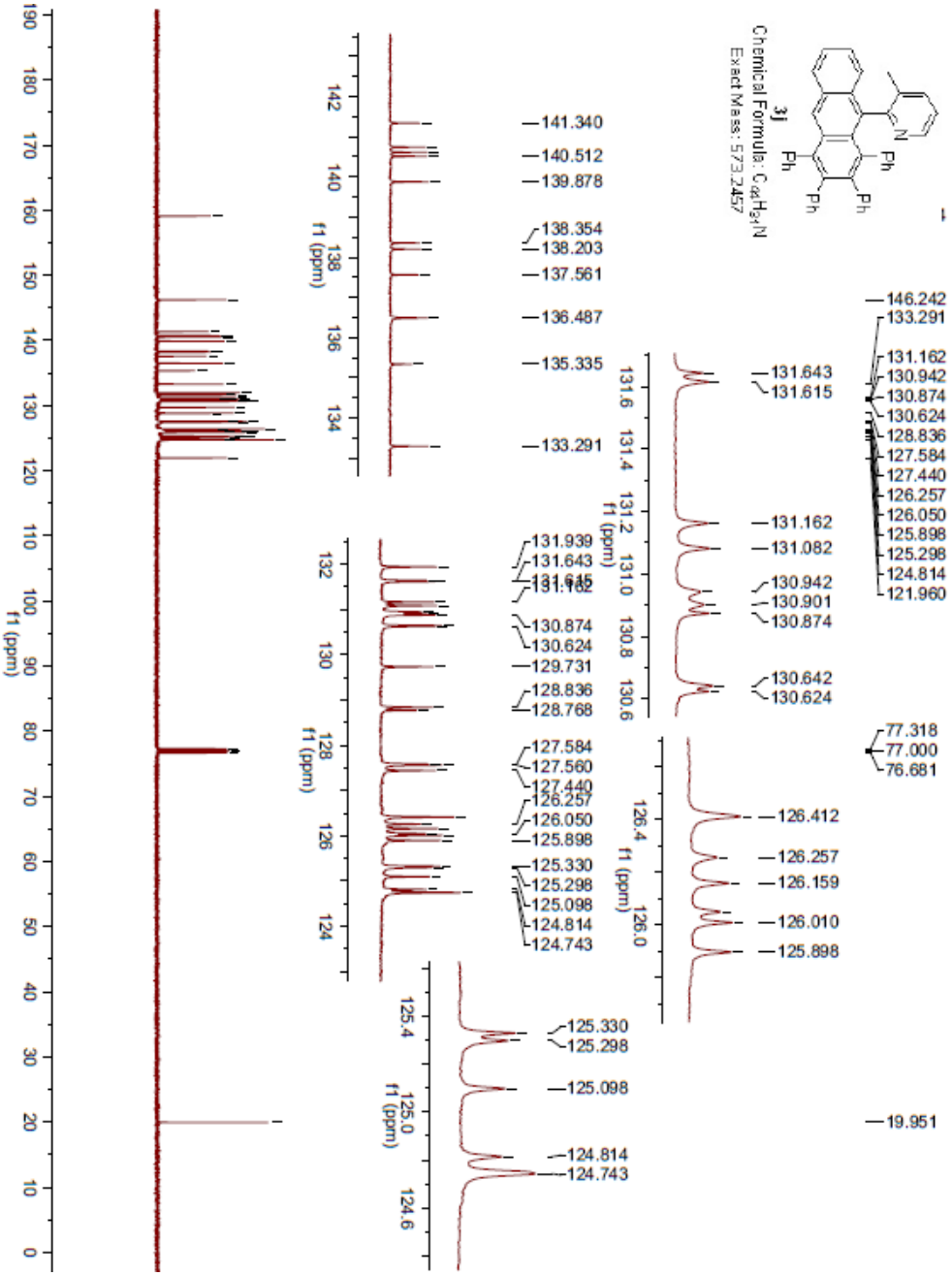

Chemical Formula: C46H31NO
Exact Mass: 589.2405
3h
MeO
Ph
Ph
Ph
Ph
N
f1 (ppm)
8.272
8.258
7.827
7.803
7.695
7.675
7.467
7.451
7.430
7.327
7.306
7.270
7.236
7.185
7.166
7.140
6.764
6.735
6.701
6.680
6.619
6.584
6.548
6.445
6.399
6.330
6.294
6.140
6.121
6.103
5.908
5.891
5.872
3.540
0.000

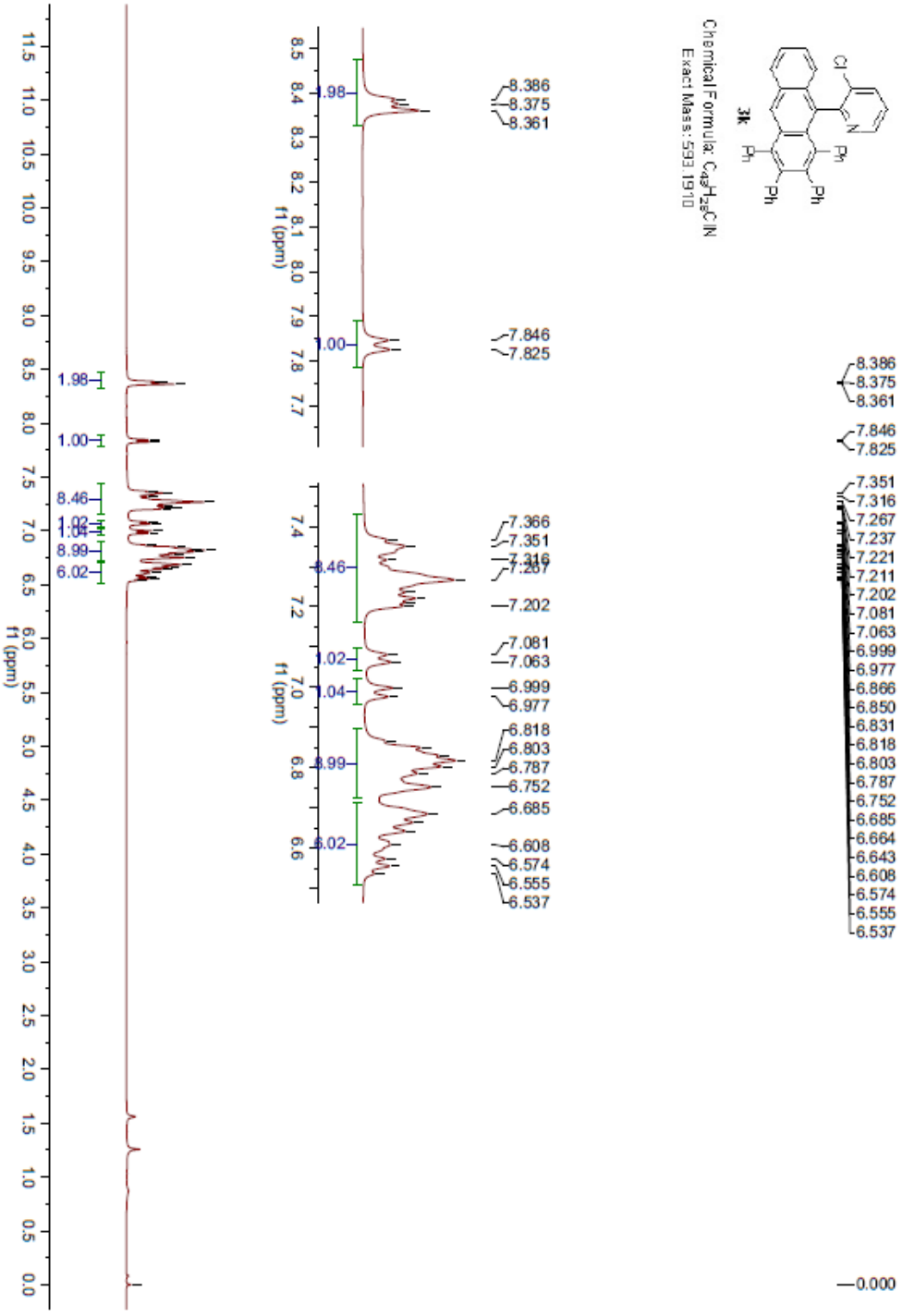

3h
MeO
Ph
Ph
Ph
Ph
N
Chemical Formula: C48H33NO
Exact Mass: 639.2405
141.762
141.598
140.596
140.551
139.947
139.170
138.394
137.178
137.005
135.267
160.072
156.096
140.551
131.999
131.059
130.097
127.389
126.145
124.988
123.382
119.593
113.096
131.115
131.088
131.059
130.962
132.518
131.999
131.539
130.962
130.498
130.097
129.028
128.910
128.291
127.389
126.294
126.145
125.787
124.988
124.639
123.841
123.382
130.520
130.498
77.318
77.000
76.682
56.657
126.374
126.294
126.224
126.145
125.933
125.787
125.051
124.988
f1 (ppm)

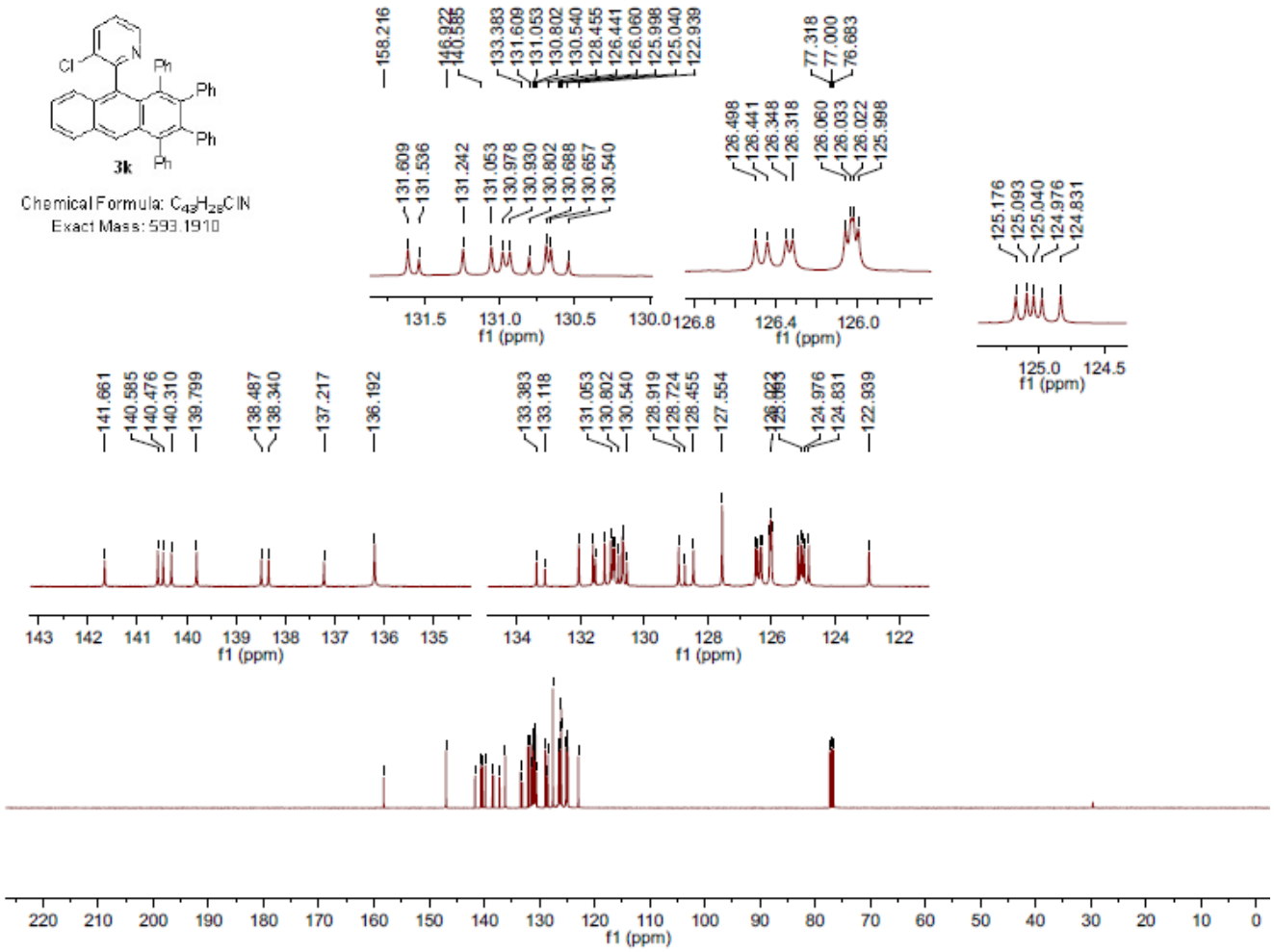

3i
Chemical Formula: C44H31N
Exact Mass: 573.2457
Ph
Ph
Ph
Ph
N
f1 (ppm)
1.00
1.00
1.00
1.00
1.03
8.96
11.89
2.10
1.04
1.02
3.01
8.298
8.283
7.726
7.704
7.592
7.485
7.448
7.345
7.224
7.206
7.186
7.168
7.153
6.797
6.693
6.669
6.642
6.594
6.579
6.507
6.465
6.353
6.333
6.313
6.165
6.147
6.128
5.907
5.888
5.871
7.526
7.506
7.487
7.485
7.470
7.468
7.450
7.448
1.813
0.000

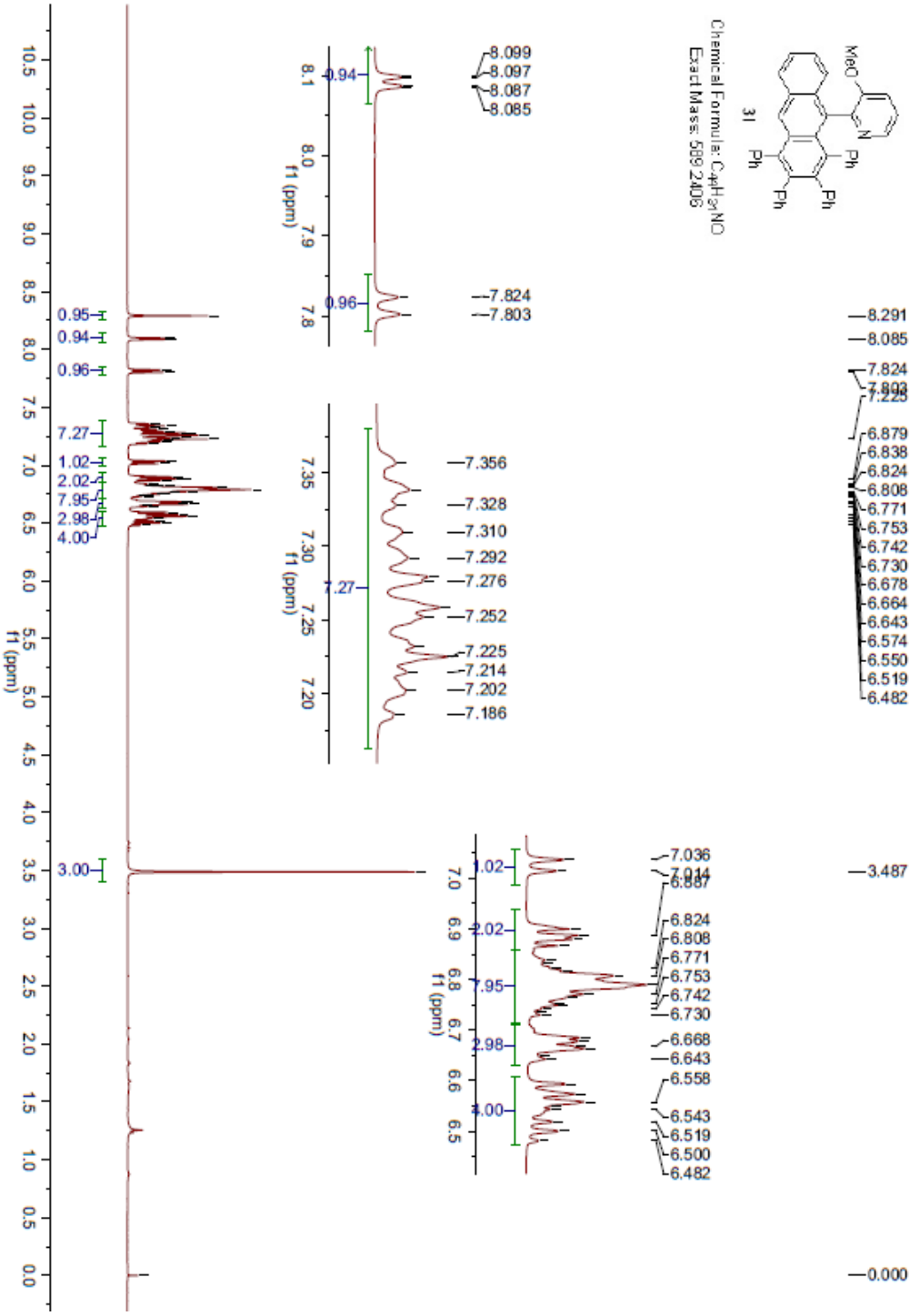

3i
Chemical Formula: C44H31N
Exact Mass: 573.2457
Ph
Ph
Ph
Ph
N
141.802
141.276
140.587
140.003
139.315
138.408
138.081
137.821
136.113
135.474
135.460
140.678
140.587
162.436
139.315
136.113
131.851
131.072
129.292
127.424
126.262
125.024
123.941
119.582
132.091
131.851
131.539
131.425
131.096
131.072
131.050
131.000
130.678
129.292
128.503
128.354
127.900
127.424
127.362
126.227
125.928
125.767
125.024
124.980
124.611
124.193
123.941
127.424
127.362
126.426
126.376
126.262
126.227
77.318
77.000
76.682
20.649
f1 (ppm)

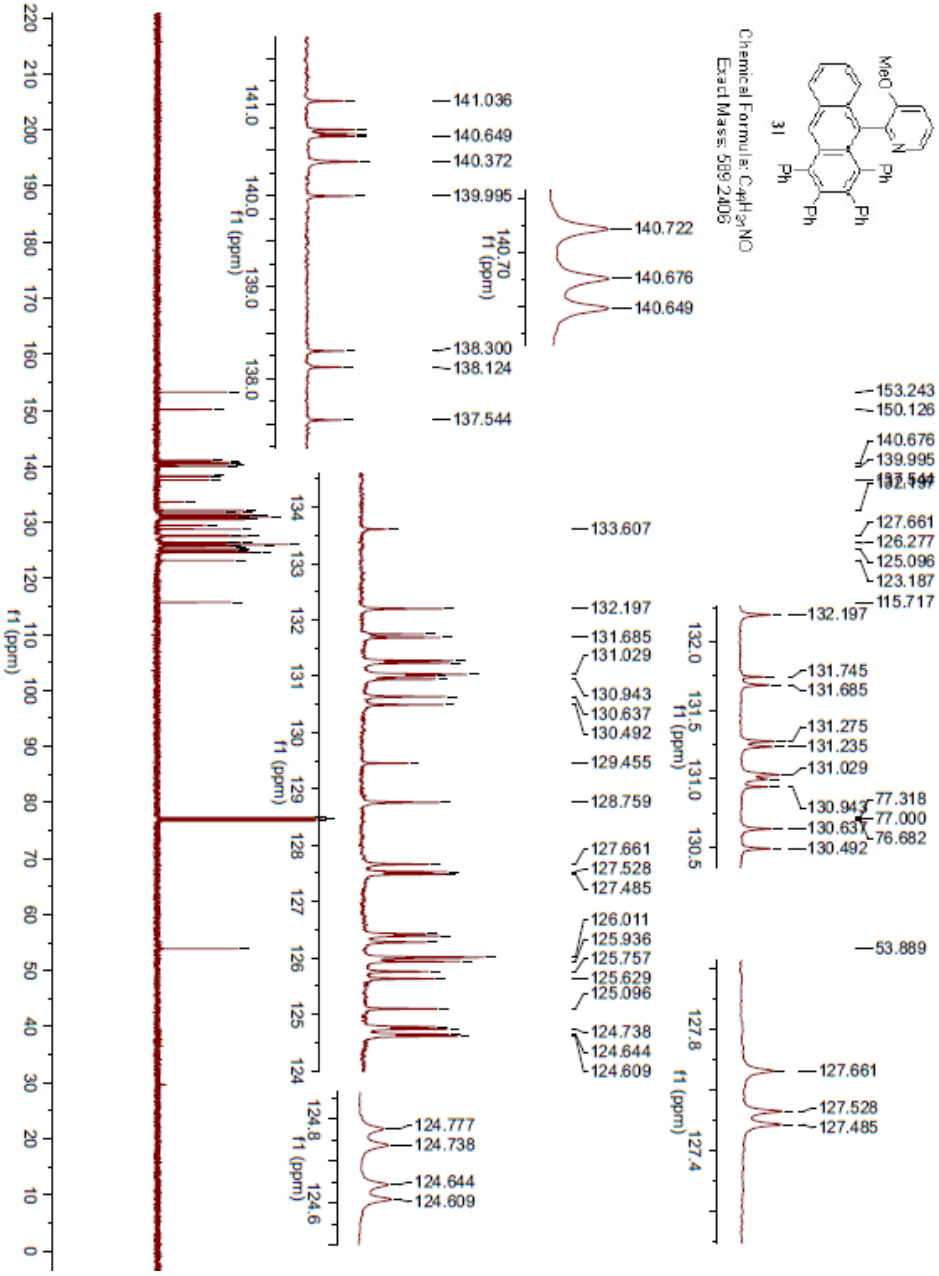

3j
Chemical Formula: C44H31N
Exact Mass: 573.2457
Ph
Ph
Ph
Ph
N
8.329
8.320
7.825
7.804
7.386
7.366
7.320
7.299
7.281
7.251
7.224
7.220
7.210
7.202
7.195
7.181
7.179
6.994
6.973
6.950
6.791
6.751
6.731
6.712
6.669
6.656
6.635
6.610
6.600
6.589
6.551
6.532
6.514
1.861
0.000
1.97
1.00
1.00
6.56
2.04
10.24
5.02
1.04
3.00
f1 (ppm)


3j
Chemical Formula: C42H29N
Exact Mass: 547.2457
Ph
Ph
Ph
Ph
N
146.242
133.291
131.162
130.942
130.874
130.624
128.836
127.584
127.440
126.257
126.050
125.898
125.298
124.814
121.960
131.643
131.615
131.162
131.082
130.942
130.901
130.874
130.642
130.624
77.318
77.000
76.681
126.412
126.257
126.159
126.010
125.898
19.951
141.340
140.512
139.878
138.354
138.203
137.561
136.487
135.335
133.291
131.939
131.643
130.874
130.624
129.731
128.836
128.768
127.584
127.560
127.440
126.257
126.050
125.898
125.330
125.298
125.098
124.814
124.743
f1 (ppm)


Chemical Formula: C43H28ClN
Exact Mass: 593.1910
3k
Cl
N
Ph
Ph
Ph
Ph
f1 (ppm)
1.98
1.00
8.46
1.02
1.04
8.99
6.02
8.386
8.375
8.361
7.846
7.825
7.366
7.351
7.316
7.267
7.202
7.081
7.063
6.999
6.977
6.818
6.803
6.787
6.752
6.685
6.608
6.574
6.555
6.537
0.000


Cl
N
Ph
Ph
Ph
Ph
3k
Chemical Formula: C43H28ClN
Exact Mass: 593.1910
158.216
146.922
140.585
133.383
131.609
131.053
130.802
130.540
128.455
126.441
126.060
125.998
125.040
122.939
77.318
77.000
76.683
131.609
131.536
131.242
131.053
130.978
130.930
130.802
130.688
130.657
130.540
126.498
126.441
126.348
126.318
126.060
126.033
126.022
125.998
125.176
125.093
125.040
124.976
124.831
131.5
131.0
130.5
130.0
f1 (ppm)
126.8
126.4
126.0
f1 (ppm)
125.0
124.5
f1 (ppm)
141.661
140.585
140.476
140.310
139.799
138.487
138.340
137.217
136.192
133.383
133.118
131.053
130.802
130.540
128.919
128.724
128.455
127.554
124.976
124.831
122.939
143
142
141
140
139
138
137
136
135
f1 (ppm)
134
132
130
128
126
124
122
f1 (ppm)
220
210
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
f1 (ppm)


Chemical Formula: C44H31NO
Exact Mass: 589.2406
31
MeO
N
Ph
Ph
Ph
Ph
f1 (ppm)
8.099
8.097
8.087
8.085
7.824
7.803
7.356
7.328
7.310
7.292
7.276
7.252
7.225
7.214
7.202
7.186
8.291
8.085
7.824
6.879
6.838
6.824
6.808
6.771
6.753
6.742
6.730
6.678
6.664
6.643
6.574
6.550
6.519
6.482
7.036
6.824
6.808
6.771
6.753
6.742
6.730
6.668
6.643
6.558
6.543
6.519
6.500
6.482
3.487
0.000


Chemical Formula: C44H29NO
Exact Mass: 587.2406
MeO
N
Ph
Ph
Ph
Ph
31
153.243
150.126
140.676
139.995
127.661
126.277
125.096
123.187
115.717
132.197
131.745
131.685
131.275
131.235
131.029
130.943
130.637
130.492
77.318
77.000
76.682
53.889
127.661
127.528
127.485
141.036
140.649
140.372
139.995
138.300
138.124
137.544
140.722
140.676
140.649
133.607
132.197
131.685
131.029
130.943
130.637
130.492
129.455
128.759
127.661
127.528
127.485
126.011
125.936
125.757
125.629
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124.738
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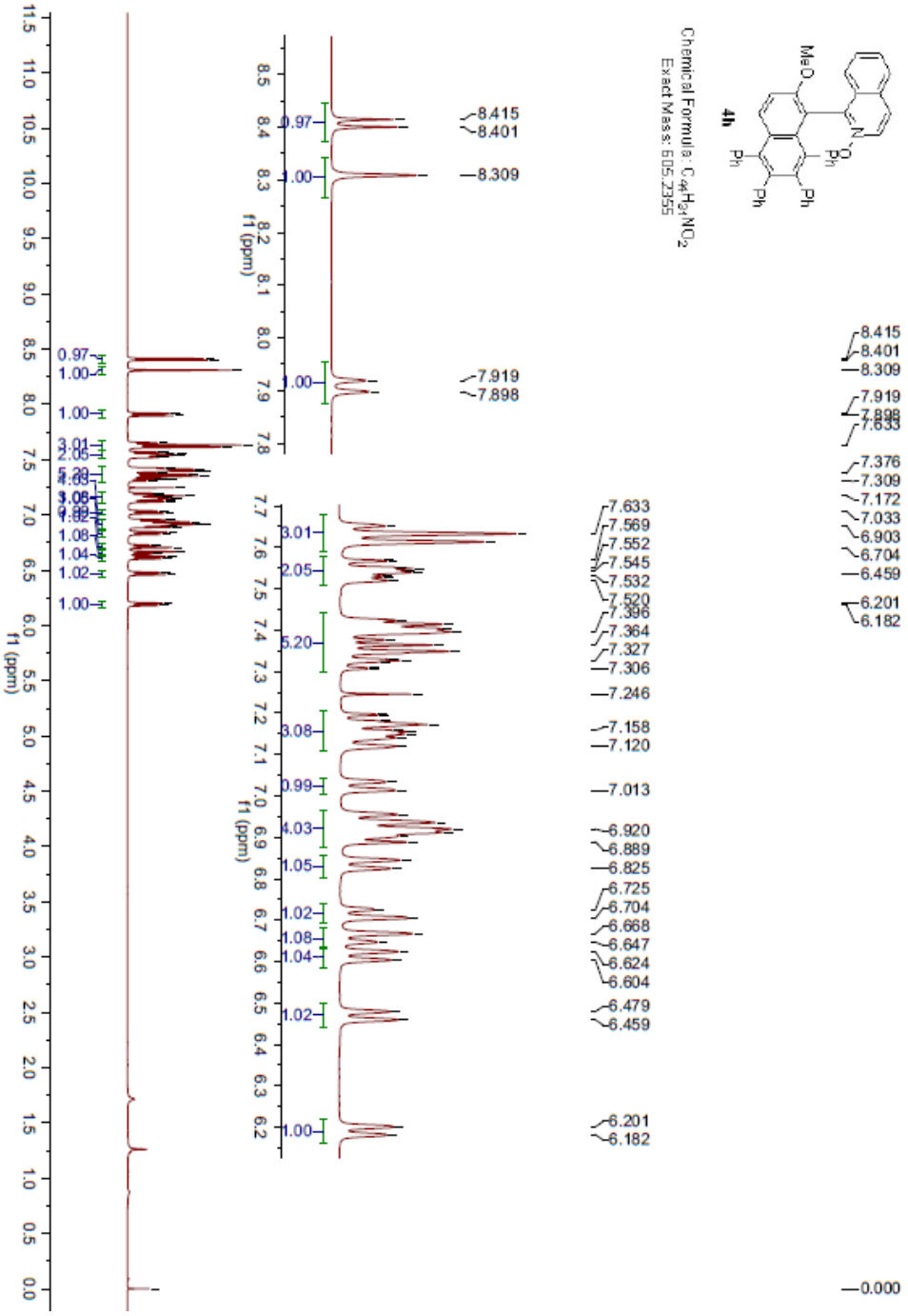
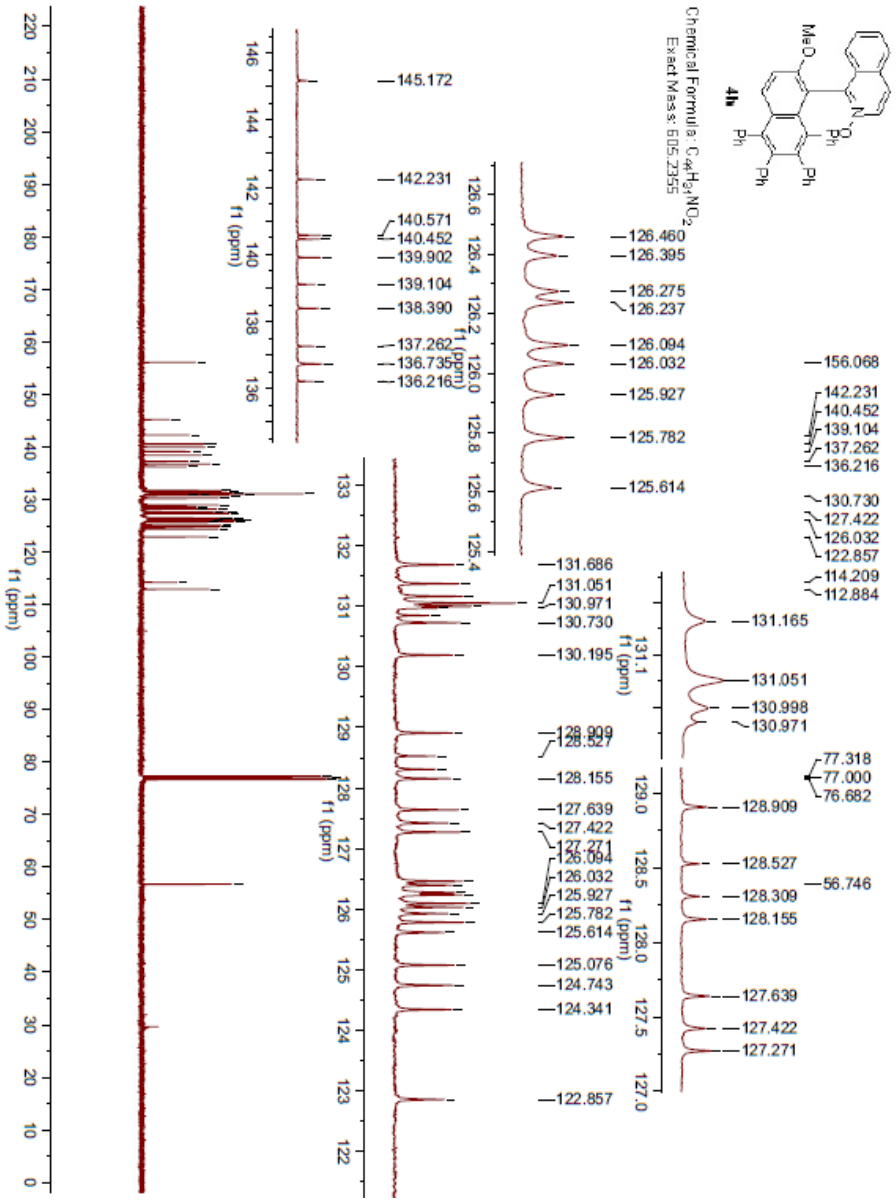
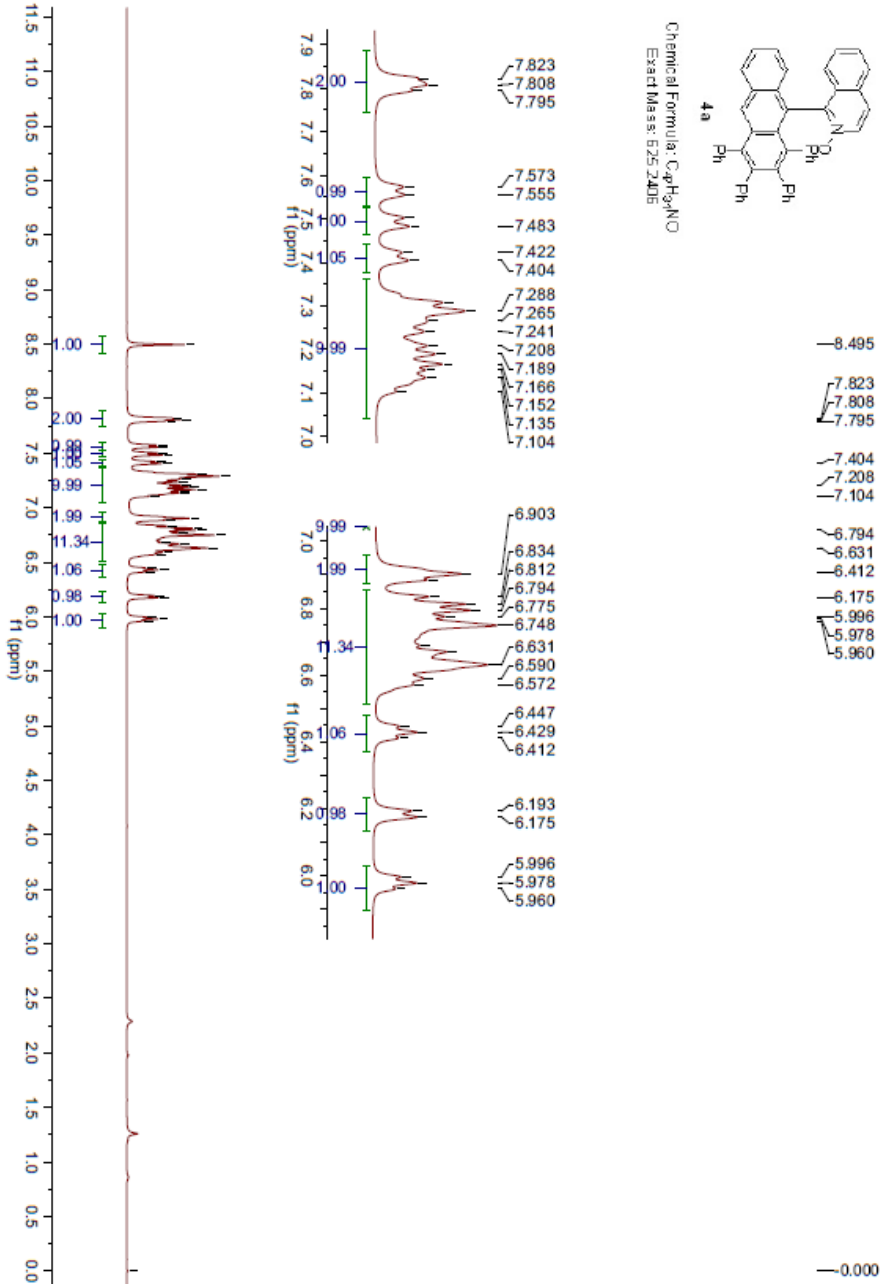
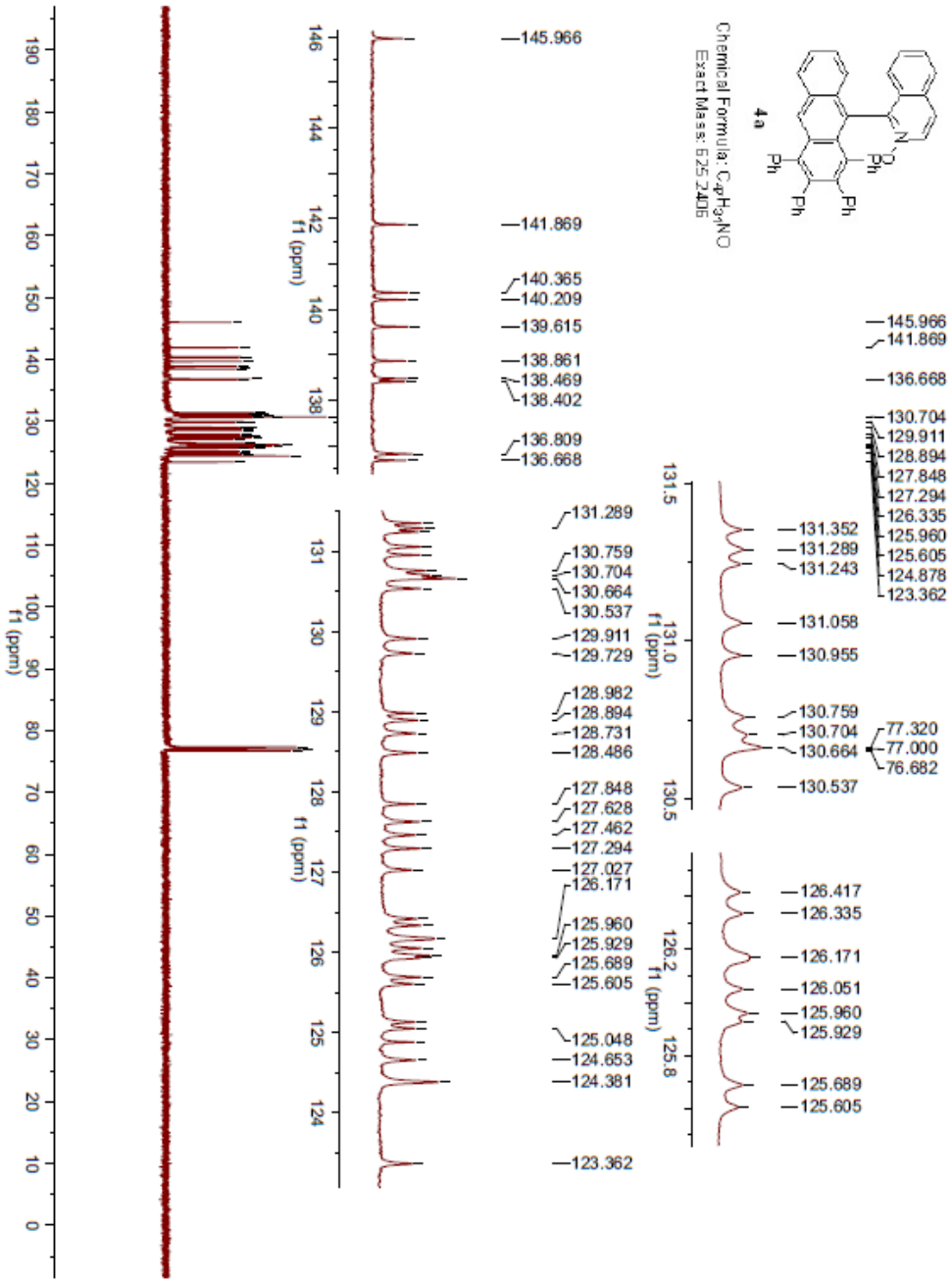
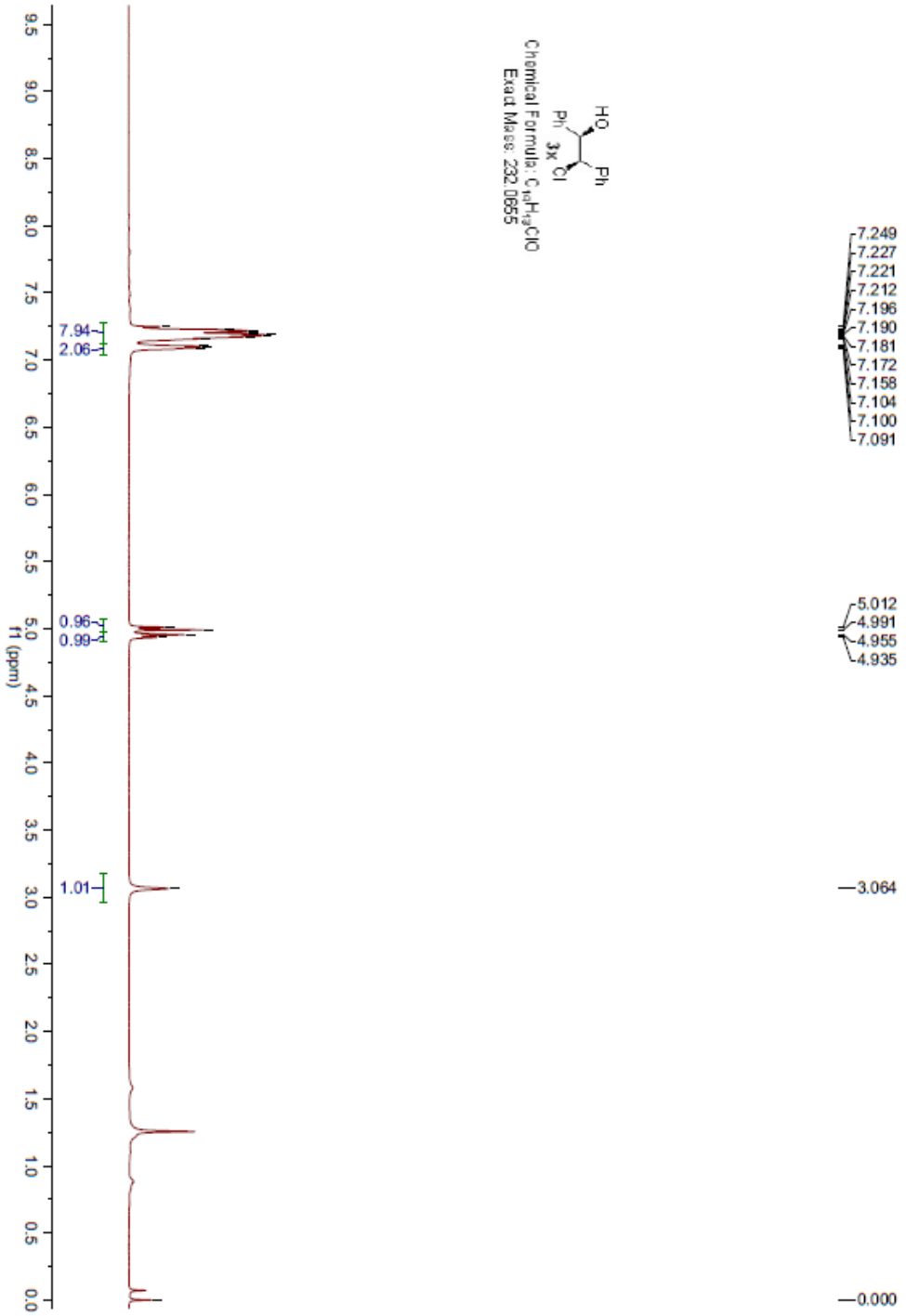
124.777
124.738
124.644
124.609
f1 (ppm)

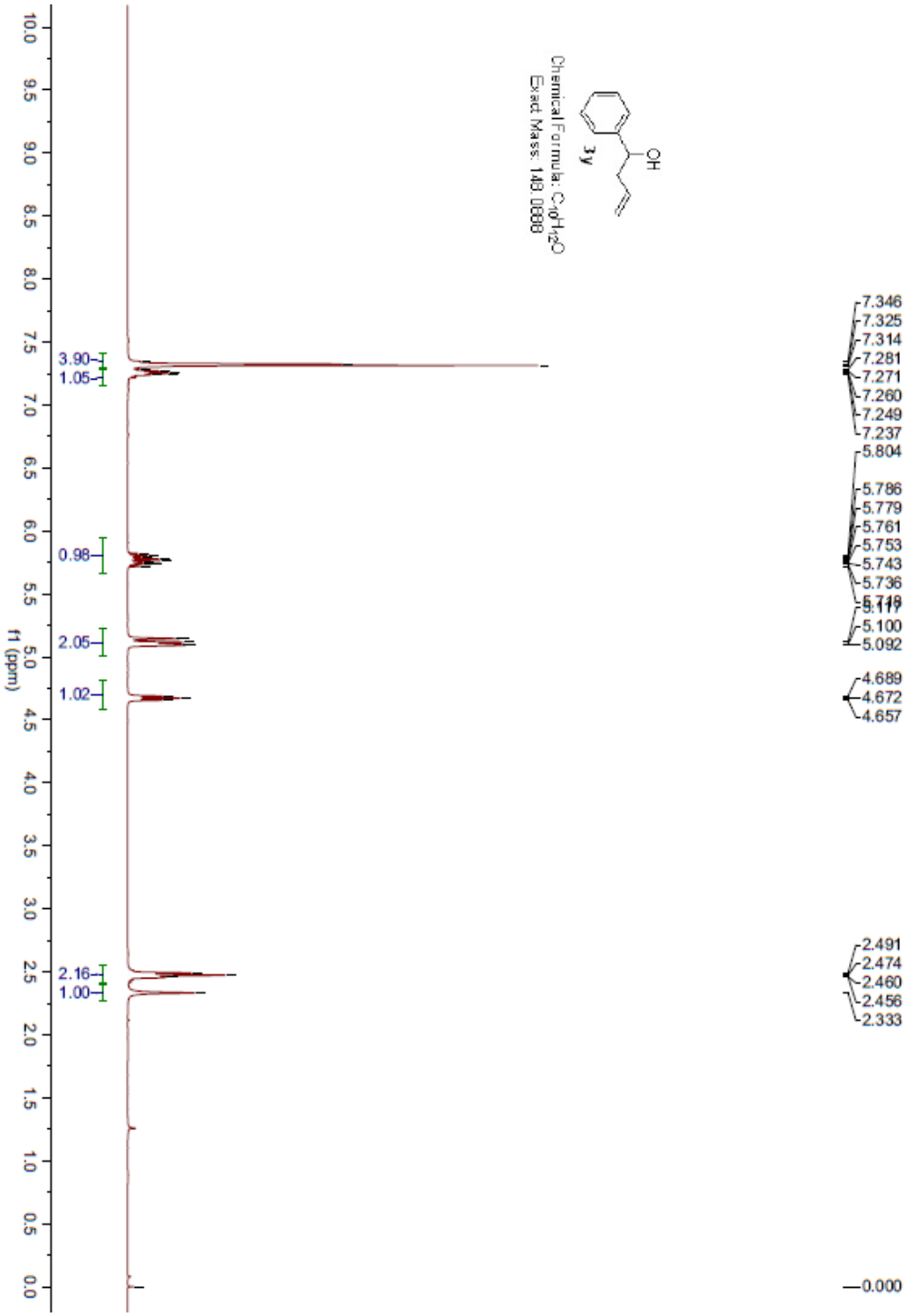

4h
Chemical Formula: C46H33NO2
Exact Mass: 615.2355
MeO
Ph
Ph
Ph
Ph
N
O
8.415
8.401
8.309
7.919
7.898
7.633
7.376
7.309
7.172
7.033
6.903
6.704
6.459
6.201
6.182
0.000
f1 (ppm)

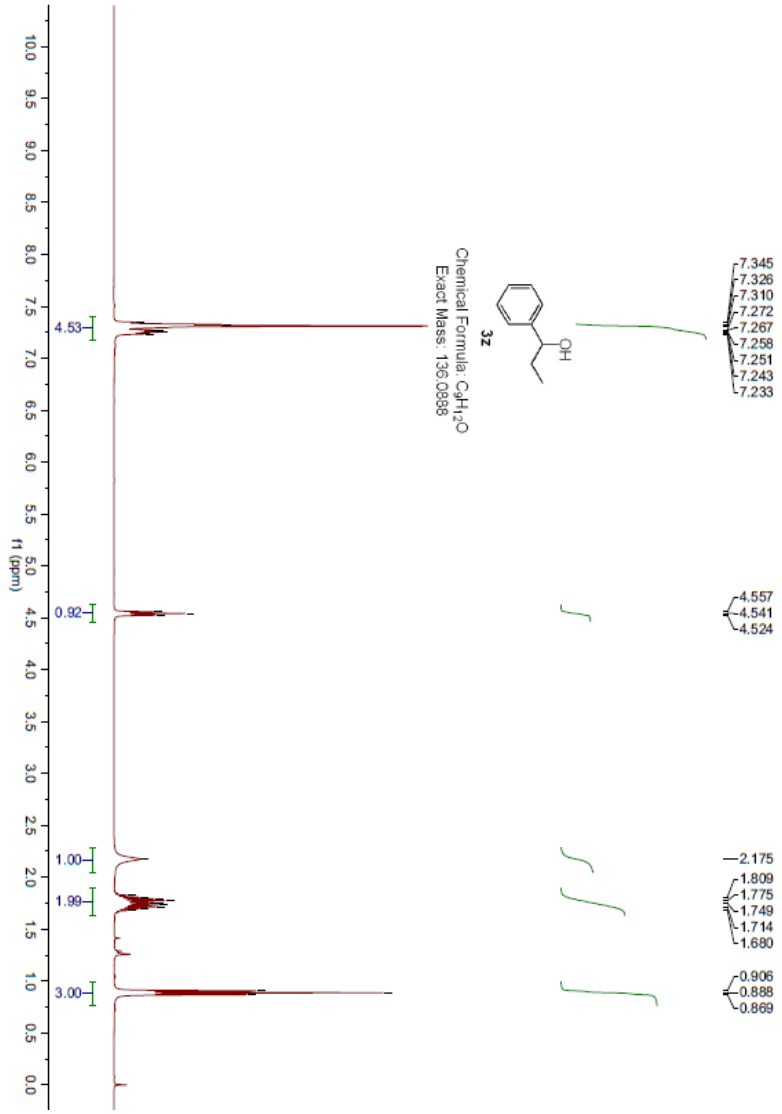

4h
Chemical Formula: C46H33NO2
Exact Mass: 615.2355
MeO
Ph
Ph
Ph
Ph
N
O
156.068
142.231
140.452
139.104
137.262
136.216
130.730
127.422
126.032
122.857
114.209
112.884
131.165
131.051
130.998
130.971
77.318
77.000
76.682
128.909
128.527
128.309
128.155
56.746
127.639
127.422
127.271
145.172
142.231
140.571
140.452
139.902
139.104
138.390
137.262
136.735
136.216
126.460
126.395
126.275
126.237
126.094
126.032
125.927
125.782
125.614
131.686
131.051
130.971
130.730
130.195
128.909
128.527
128.155
127.639
127.422
127.271
126.094
126.032
125.927
125.782
125.614
125.076
124.743
124.341
122.857
f1 (ppm)

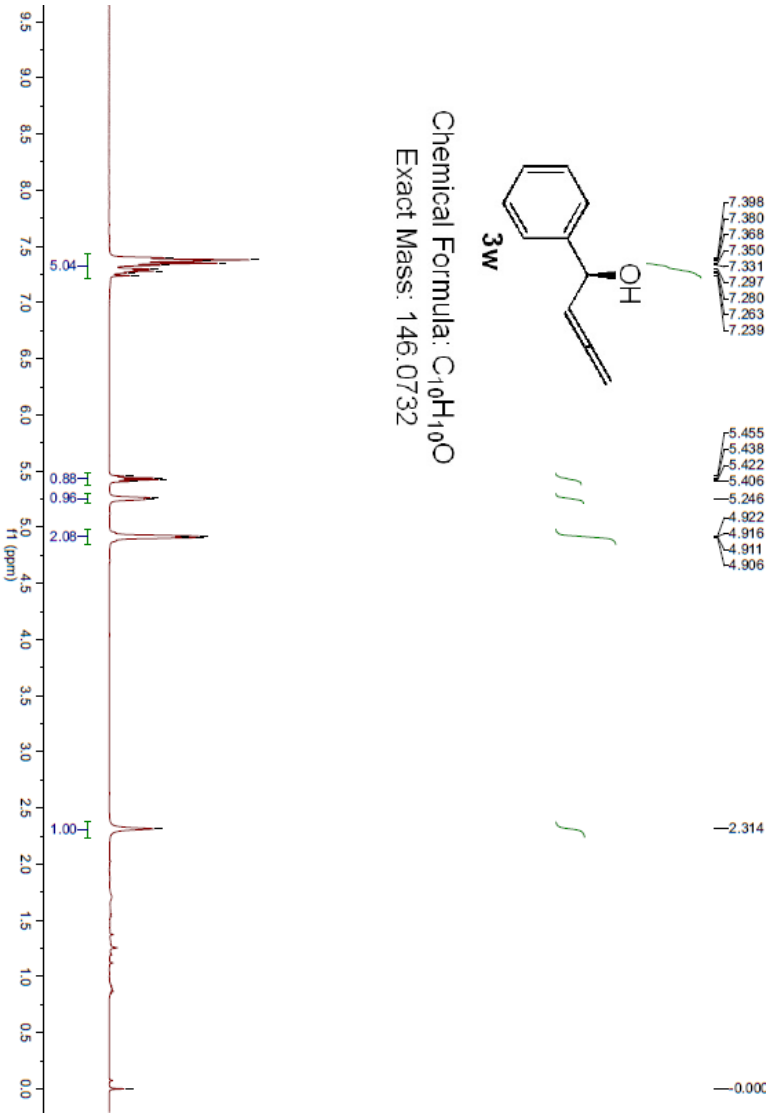

Chemical Formula: C42H29NO
Exact Mass: 563.2406
4-a
Ph
Ph
Ph
Ph
O
N
f1 (ppm)
1.00
2.00
9.99
1.99
11.34
1.06
0.98
1.00
7.823
7.808
7.795
7.573
7.555
7.483
7.422
7.404
7.288
7.265
7.241
7.208
7.189
7.166
7.152
7.135
7.104
6.903
6.834
6.812
6.794
6.775
6.748
6.631
6.590
6.572
6.447
6.429
6.412
6.193
6.175
5.996
5.978
5.960
8.495
7.823
7.808
7.795
7.404
7.208
7.104
6.794
6.631
6.412
6.175
5.996
5.978
5.960
0.000


4a
Chemical Formula: C40H29NO
Exact Mass: 635.2406
145.966
141.869
140.365
140.209
139.615
138.861
138.469
138.402
136.809
136.668
131.289
130.759
130.704
130.664
130.537
129.911
129.729
128.982
128.894
128.731
128.486
127.848
127.628
127.462
127.294
127.027
126.171
125.960
125.929
125.689
125.605
125.048
124.653
124.381
123.362
131.352
131.289
131.243
131.058
130.955
130.759
130.704
130.664
130.537
126.417
126.335
126.171
126.051
125.960
125.929
125.689
125.605
77.320
77.000
76.682
f1 (ppm)


Chemical Formula: $C_{16}H_{17}ClO$
Exact Mass: 232.0955
HO
Ph
Ph
3x
Cl
7.249
7.227
7.221
7.212
7.196
7.190
7.181
7.172
7.158
7.104
7.100
7.091
5.012
4.991
4.955
4.935
3.064
0.000
7.94
2.06
0.96
0.99
1.01
f1 (ppm)


Chemical Formula: C10H12O
Exact Mass: 148.0888
3y
OH
f1 (ppm)
3.90
1.05
0.98
2.05
1.02
2.16
1.00
7.346
7.325
7.314
7.281
7.271
7.260
7.249
7.237
5.804
5.786
5.779
5.761
5.753
5.743
5.736
5.100
5.092
4.689
4.672
4.657
2.491
2.474
2.460
2.456
2.333
0.000


3z
Chemical Formula: C9H12O
Exact Mass: 136.0888
OH
7.345
7.326
7.310
7.272
7.267
7.258
7.251
7.243
7.233
4.557
4.541
4.524
2.175
1.809
1.775
1.749
1.714
1.680
0.906
0.888
0.869
4.53
0.92
1.00
1.99
3.00
f1 (ppm)


3w
OH
Chemical Formula: C10H10O
Exact Mass: 146.0732
7.398
7.380
7.368
7.350
7.331
7.297
7.280
7.263
7.239
5.455
5.438
5.422
5.406
5.246
4.922
4.916
4.911
4.906
2.314
0.000
5.04
0.88
0.96
2.08
1.00
f1 (ppm)

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