Electronic Supplementary Material (ESI) for Organic Chemistry Frontiers.

Supporting Information

Iron-mediated highly diastereoselective allylation of carbonyl compounds with cyclic allylic halides

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General information

Unless otherwise stated, all reagents were purchased from commercial suppliers and used without further purification. All reactions were carried out under air using undistilled solvent, without the need of precautions to exclude air and moisture. IR spectra were recorded on a FT-IR spectrophotometer using KBr optics. ^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ on Bruker Avance or Joel 400 MHz spectrometers. Tetramethylsilane (TMS) served as an internal standard for ^{1}H NMR and ^{13}C NMR. High resolution mass spectra (HRMS) were obtained using a commercial apparatus (ESI or EI Source). 3-Chlorocyclohex-1-ene (**2b**),[1,2] 3-iodocyclohex-1-ene (**2c**),[3] 3-chloro-5,5-dimethylcyclohex-1-ene (**2d**),[4] and (*Z*)-3-bromocyclooct-1-ene (**2e**) [5] were synthesized according to reported methods.

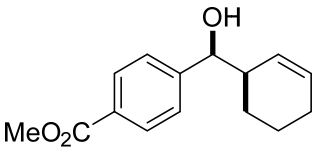
Experimental procedure

Typical procedures for bismuth(III) chloride-catalyzed iron-mediated highly diastereoselective allylation reactions of carbonyl compounds with cyclic allylic halides and cinnamyl bromide

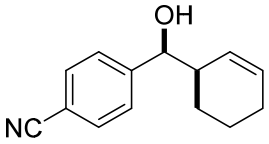
To a 10 mL Schlenk flask was sequentially added iron powder (83.8 mg, 1.5 mmol) and DMSO (1 mL). Then iron was activated by the addition of 1,2-dibromoethane (14.1 mg, 5 mol%) and TMSCl (8.1 mg, 5 mol%). After stirring for 5 min, $BiCl_3$ (31.5 mg, 0.1 mmol), allyl halides (1.5 mmol), and aldehyde or ketone (0.5 mmol) was sequentially added to the reaction mixture. The suspension was vigorously stirred at room temperature for 24 h before quenching with sat. NH_4Cl solution (30 mL) and extracting with ethyl acetate (20 mL×3). The combined extracts were washed with brine (30 mL), dried over anhydrous Na_2SO_4 and concentrated *in vacuo*. The residue obtained was purified by silica gel column chromatography using ethyl acetate/petroleum ether as eluent to give the pure products.

It should be noted that, in cases where iron was used as reaction mediator and NMR yield of product **3a** was provided (Table 1 and Scheme 1), the reaction mixture should be passed through a short column loaded with silica gel (and washed with ethyl acetate to remove any iron or iron salt in the reaction mixture because of its paramagnetism which is not suitable for NMR analysis) followed by normal work-up and subsequent ^{1}H NMR analysis. In a case where phenylglyoxylic acid was used as substrate, the reaction mixture was acidified by aq. HCl (0.1 M, 20 mL) before extracting with ethyl acetate.

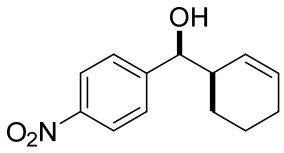
Characterization data of products



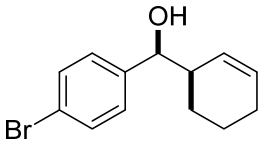
Methyl 4-(cyclohex-2-en-1-yl(hydroxy)methyl)benzoate (3a): 118.2 mg, 96% yield, >99:1 dr, white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.97-7.94 (m, 2H), 7.37-7.35 (m, 2H), 5.83-5.78 (m, 1H), 5.37-5.34 (m, 1H), 4.62 (d, J = 6.1 Hz, 1H), 3.87 (s, 3H), 2.50-2.43 (m, 2H), 2.00-1.92 (m, 2H), 1.75-1.67 (m, 1H), 1.61-1.54 (m, 1H), 1.53-1.39 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 167.0, 148.1, 130.8, 129.3, 128.9, 127.5, 126.3, 76.6, 52.0, 42.9, 25.0, 23.3, 21.0 ppm. IR (KBr): ν = 3508, 2936, 1707, 1436, 1290, 1119, 753 cm^{-1}. HRMS (m/z): calcd for $C_{15}H_{19}O_3$ $[M+H]^+$ 247.1334, found: 247.1330.



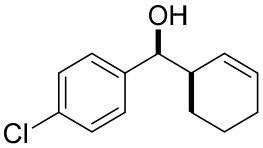
4-(Cyclohex-2-en-1-yl(hydroxy)methyl)benzonitrile (3b): 100.2 mg, 94% yield, >99:1 dr, white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.63-7.61 (m, 2H), 7.46-7.44 (m, 2H), 5.91-5.86 (m, 1H), 5.41 (dd, J = 10.2, 2.4 Hz, 1H), 4.70 (d, J = 5.5 Hz, 1H), 2.51-2.46 (m, 1H), 2.14 (s, 1H), 2.01-1.96 (m, 2H), 1.77-1.69 (m, 1H), 1.53-1.44 (m, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 148.1, 131.9, 131.6, 127.1, 127.1, 118.9, 110.8, 76.2, 42.9, 25.0, 22.9, 20.9 ppm. IR (KBr): ν = 3475, 2931, 2233, 1080, 563 cm^{-1}. HRMS (m/z): calcd for $C_{14}H_{16}NO$ $[M+H]^+$ 214.1232, found: 214.1231.



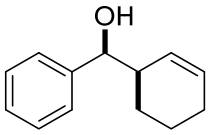
Cyclohex-2-en-1-yl (4-nitrophenyl)methanol (3c): 73.5 mg, 63% yield, >99:1 dr, yellow oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.19-8.17 (m, 2H), 7.52-7.49 (m, 2H), 5.93-5.88 (m, 1H), 5.45-5.41 (m, 1H), 4.77 (d, J = 5.4 Hz, 1H), 2.55-2.48 (m, 1H), 2.19 (s, 1H), 2.01-1.96 (m, 2H), 1.76-1.70 (m, 1H), 1.53-1.43 (m, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 150.1, 147.1, 131.9, 127.1, 127.0, 123.3, 76.0, 43.0, 25.0, 22.9, 21.0 ppm. IR (KBr): ν = 3355, 2923, 1603, 1518, 1347, 1030, 865, 725 cm^{-1}. HRMS (m/z): calcd for $C_{13}H_{16}NO_3$ $[M+H]^+$ 234.1130, found: 234.1162.



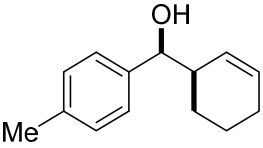
(4-Bromophenyl)(cyclohex-2-en-1-yl)methanol (3d): 113.5 mg, 85% yield, >99:1 dr, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.47-7.43 (m, 2H), 7.20-7.18 (m, 2H), 5.85-5.80 (m, 1H), 5.38-5.34 (m, 1H), 4.53 (d, J = 6.2 Hz, 1H), 2.47-2.40 (m, 1H), 2.13 (s, 1H), 2.00-1.95 (m, 2H), 1.76-1.68 (m, 1H), 1.66-1.59 (m, 1H), 1.53-1.42 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 141.7, 131.2, 130.8, 128.1, 127.5, 121.0, 76.5, 42.9, 25.1, 23.5, 21.0 ppm. IR (KBr): ν = 3319, 2932, 1483, 1009, 813, 672 cm^{-1}. HRMS (m/z): calcd for $C_{13}H_{16}BrO$ $[M+H]^+$ 267.0385, found: 267.0378.



4-Chlorophenyl(cyclohex-2-en-1-yl)methanol (3e): 89.1 mg, 80% yield, > 99:1 dr, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.33-7.26 (m, 4H), 5.87-5.82 (m, 1H), 5.41-5.36 (m, 1H), 4.59 (d, J = 6.2 Hz, 1H), 2.49-2.43 (m, 1H), 2.01-1.95 (m, 3H), 1.79-1.71 (m, 1H), 1.67-1.60 (m, 1H), 1.54-1.43 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 141.2, 132.9, 130.9, 128.3, 127.8, 127.5, 76.6, 43.0, 25.2, 23.5, 21.0 ppm. IR (KBr): ν = 3340, 2926, 1495, 1089, 1013, 815, 724 cm^{-1}. HRMS (m/z): calcd for $C_{13}H_{16}ClO$ $[M+H]^+$ 223.0890, found: 223.0885.

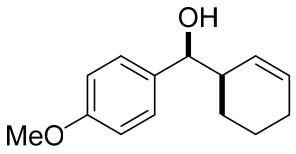


Cyclohex-2-en-1-yl(phenyl)methanol (3f): 84.5 mg, 90% yield, >99:1 dr, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.35-7.31 (m, 4H), 7.29-7.23 (m, 1H), 5.82-5.77 (m, 1H), 5.38-5.34 (m, 1H), 4.55 (d, J = 6.6 Hz, 1H), 2.51-2.45 (m, 1H), 2.04-1.95 (m, 3H), 1.80-1.64 (m, 2H), 1.57-1.44 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 142.8, 130.3, 128.1, 127.9, 127.3, 126.5, 77.3, 42.9, 25.2, 23.8, 21.1 ppm. IR (KBr): ν = 3385, 3026, 1453, 1017, 763, 701 cm^{-1}. HRMS (m/z): calcd for $C_{13}H_{17}O$ $[M+H]^+$ 189.1279, found: 189.1279.

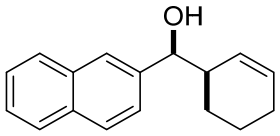


Cyclohex-2-en-1-yl(*p*-tolyl)methanol (3g): 65.7 mg, 65% yield, >99:1 dr, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.20 (d, J = 8.1 Hz, 2H), 7.13 (d, J = 8.1 Hz, 2H), 5.80-5.75 (m, 1H),

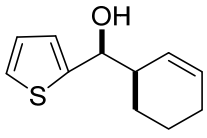
5.37-5.32 (m, 1H), 4.49 (d, J = 6.8 Hz, 1H), 2.49-2.42 (m, 1H), 2.33 (s, 3H), 2.01-1.94 (m, 3H), 1.80-1.67 (m, 2H), 1.54-1.44 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 139.9, 136.9, 130.0, 128.8, 128.0, 126.4, 77.3, 42.9, 25.2, 24.0, 21.1, 21.1 ppm. IR (KBr): ν = 3346, 2924, 2866, 1514, 1015, 807, 721, 691 cm^{-1}. HRMS (m/z): calcd for $C_{14}H_{19}O$ $[M+H]^+$ 203.1436, found: 203.1438.



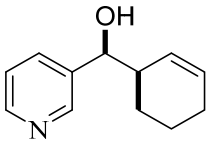
Cyclohex-2-en-1-yl(4-methoxyphenyl)methanol (3h): 71.3 mg, 67% yield, >99:1 dr, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.25-7.22 (m, 2H), 6.88-6.84 (m, 2H), 5.79-5.74 (m, 1H), 5.35-5.31 (m, 1H), 4.47 (d, J = 7.0 Hz, 1H), 3.79 (s, 3H), 2.48-2.42 (m, 1H), 2.06 (s, 1H), 1.99-1.95 (m, 2H), 1.78-1.71 (m, 2H), 1.57-1.44 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 158.8, 135.1, 130.0, 127.9, 127.7, 113.5, 77.1, 55.2, 42.9, 25.2, 24.2, 21.0 ppm. IR (KBr): ν = 3443, 3030, 2882, 1612, 1254, 1034, 814 cm^{-1}. HRMS (m/z): calcd for $C_{14}H_{19}O_2$ $[M+H]^+$ 219.1385, found: 219.1386



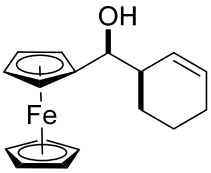
Cyclohex-2-en-1-yl(naphthalen-2-yl)methanol (3i): 110.8 mg, 93% yield, >99:1 dr, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.02-8.00 (m, 1H), 7.86-7.84 (m, 1H), 7.75 (d, J = 8.2 Hz, 1H), 7.64 (d, J = 7.2 Hz, 1H), 7.48-7.43 (m, 3H), 5.83-5.81 (d, J = 10.1 Hz, 1H), 5.45 (d, J = 10.3 Hz, 1H), 5.38 (d, J = 6.6 Hz, 1H), 2.76-2.72 (m, 1H), 2.27 (s, 1H), 2.03-1.94 (m, 2H), 1.76-1.72 (m, 1H), 1.64-1.52 (m, 2H), 1.45-1.39 (m, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 138.2, 133.6, 130.5, 130.4, 128.8, 128.5, 127.6, 125.7, 125.3, 125.2, 123.9, 123.1, 73.4, 41.8, 25.1, 23.4, 21.2 ppm. IR (KBr): ν = 3409, 2926, 2859, 1090, 993, 778, 724 cm^{-1}. HRMS (m/z): calcd for $C_{17}H_{19}O$ $[M+H]^+$ 239.1436, found: 239.1436.



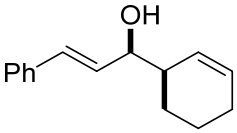
Cyclohex-2-en-1-yl(thiophen-2-yl)methanol (3j): 73.8 mg, 76% yield, >99:1 dr, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.28-7.26 (m, 1H), 7.01-6.98 (m, 2H), 5.87-5.82 (m, 1H), 5.49-5.45 (m, 1H), 4.81 (d, J = 7.0 Hz, 1H), 2.60-2.53 (m, 1H), 2.32 (s, 1H), 2.04-1.99 (m, 2H), 1.90-1.74 (m, 2H), 1.62-1.49 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 146.7, 130.3, 127.3, 126.3, 124.5, 124.4, 73.5, 43.3, 25.1, 24.5, 21.0 ppm. IR (KBr): ν = 3398, 3022, 2927, 1660, 1447, 1012, 853, 699 cm^{-1}. HRMS (m/z): calcd for $C_{11}H_{15}OS$ $[M+H]^+$ 195.0844, found: 195.0844.



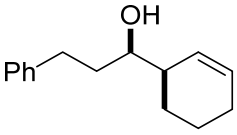
Cyclohex-2-en-1-yl(pyridin-3-yl)methanol (3k): 60.6 mg, 64% yield, >99:1 dr, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.47-8.42 (m, 2H), 7.73-7.70 (m, 1H), 7.28 (dd, J = 7.9, 4.8 Hz, 1H), 5.87-5.82 (m, 1H), 5.39 (dq, J = 10.2, 2.4 Hz, 1H), 4.63 (d, J = 6.3 Hz, 1H), 3.23 (s, 1H), 2.53-2.47 (m, 1H), 2.01-1.93 (m, 2H), 1.79-1.62 (m, 2H), 1.58-1.45 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 148.3, 148.1, 138.5, 134.4, 131.0, 127.2, 123.2, 74.8, 42.9, 25.1, 23.6, 20.9 ppm. IR (KBr): ν = 3216, 3022, 2928, 1580, 1428, 1027, 717 cm^{-1}. HRMS (m/z): calcd for $C_{12}H_{16}NO$ $[M+H]^+$ 190.1232, found: 190.1229.



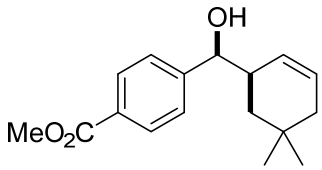
Cyclohex-2-en-1-yl(ferrocenyl)methanol (3l): 80.0 mg, 54% yield, >99:1 dr, yellow solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 5.75-5.70 (m, 1H), 5.45-5.41 (m, 1H), 4.27-4.26 (m, 1H), 4.21 (s, 5H), 4.18-4.15 (m, 3H), 2.27-2.21 (m, 1H), 2.14 (d, J = 1.5 Hz, 1H), 1.96-1.91 (m, 2H), 1.79-1.68 (m, 2H), 1.52-1.42 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 129.1, 128.1, 93.0, 73.0, 68.7, 68.2, 67.8, 67.5, 64.9, 42.6, 25.2, 24.8, 21.1 ppm. IR (KBr): ν = 3550, 3025, 2862, 1334, 1103, 820, 720 cm^{-1}. HRMS (m/z): calcd for $C_{17}H_{21}FeO$ $[M+H]^+$ 294.0910, found: 294.0916.



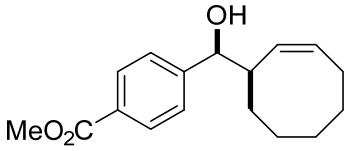
(*E*)-1-(Cyclohex-2-en-1-yl)-3-phenylprop-2-en-1-ol (3m): 87.9 mg, 82% yield, >99:1 dr, colorless oil; ^{1}HNMR (400 MHz, $CDCl_3$): δ = 7.40-7.37 (m, 2H), 7.34-7.30 (m, 2H), 7.26-7.22 (m, 1H), 6.60 (dd, J = 15.9, 1.0 Hz, 1H), 6.25 (dd, J = 15.9, 7.1 Hz, 1H), 5.88-5.83 (m, 1H), 5.65 (dq, J = 10.2, 2.4 Hz, 1H), 4.22-4.18 (m, 1H), 2.42-2.36 (m, 1H), 2.03-1.97 (m, 2H), 1.85-1.75 (m, 3H), 1.60-1.41 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 136.7, 131.4, 130.5, 130.2, 128.5, 127.6, 126.4, 76.1, 41.8, 25.2, 24.1, 21.3 ppm. IR (KBr): ν = 3439, 2924, 2856, 1648, 1448, 966, 751, 693 cm^{-1}. HRMS (m/z): calcd for $C_{15}H_{19}O$ $[M+H]^+$ 215.1436, found: 215.1437.



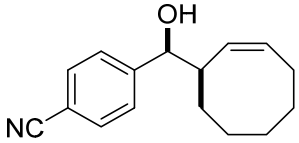
1-(Cyclohex-2-en-1-yl)-3-phenylpropan-1-ol (3n): 34.6 mg, 32% yield, >99:1 dr, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.31-7.25 (m, 2H), 7.23-7.16 (m, 3H), 5.89-5.84 (m, 1H), 5.54 (dq, *J* = 10.2, 2.4 Hz, 1H), 3.61 (dt, *J* = 7.4, 4.8 Hz, 1H), 2.88-2.81 (m, 1H), 2.70-2.62 (m, 1H), 2.27-2.21 (m, 1H), 2.01-1.96 (m, 2H), 1.83-1.70 (m, 4H), 1.57-1.46 (m, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 142.2, 130.6, 128.5, 128.4, 128.3, 125.8, 73.9, 41.4, 35.6, 32.5, 25.2, 22.7, 21.4 ppm. IR (KBr): ν = 3319, 3023, 2930, 1455, 1028, 914, 696 cm^{-1}. HRMS (m/z): calcd for $C_{15}H_{21}O$ $[M+H]^+$ 217.1592, found: 217.1587.



Methyl 4-((5,5-dimethylcyclohex-2-en-1-yl)(hydroxy)methyl)benzoate (3o): 115.2 mg, 84% yield, >99:1 dr, white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.01 (d, *J* = 8.0 Hz, 2H), 7.41 (d, *J* = 8.0 Hz, 2H), 5.82-5.77 (m, 1H), 5.47 (d, *J* = 10.1 Hz, 1H), 4.76 (d, *J* = 5.2 Hz, 1H), 3.91 (s, 3H), 2.54-2.52 (m, 1H), 2.02 (s, 1H), 1.88-1.83 (m, 1H), 1.73-1.67 (m, 1H), 1.29-1.24 (m, 1H), 1.21-1.15 (m, 1H), 0.92 (s, 3H), 0.82 (s, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 167.0, 147.8, 130.0, 129.5, 129.1, 126.3, 126.3, 76.7, 52.1, 41.8, 39.1, 36.0, 32.0, 29.3, 25.0 ppm. IR (KBr): ν = 3516, 3021, 2951, 1694, 1296, 1124, 685 cm^{-1}. HRMS (m/z): calcd for $C_{17}H_{23}O_3$ $[M+H]^+$ 275.1647, found: 275.1647.

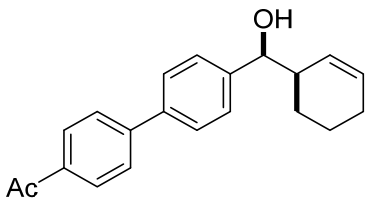


Methyl (*Z*)-4-(cyclooct-2-en-1-yl(hydroxy)methyl)benzoate (3p): 120.7 mg, 88% yield, >99:1 dr, white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.99 (d, *J* = 8.3 Hz, 2H), 7.40 (d, *J* = 8.3 Hz, 2H), 5.64-5.57 (m, 1H), 5.30-5.24 (m, 1H), 4.65 (dd, *J* = 6.9, 3.4 Hz, 1H), 3.90 (s, 3H), 2.94-2.86 (m, 1H), 2.15-2.10 (m, 1H), 2.02-1.95 (m, 1H), 1.90-1.86 (m, 1H), 1.65-1.50 (m, 3H), 1.55-1.48 (m, 2H), 1.42-1.36 (m, 1H), 1.32-1.19 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 167.0, 148.6, 130.7, 129.7, 129.5, 129.2, 126.6, 77.5, 52.1, 43.4, 31.3, 29.2, 26.7, 26.6, 25.4 ppm. IR (KBr): ν = 3485, 2924, 1723, 1435, 1283, 1105, 757 cm^{-1}. HRMS (m/z): calcd for $C_{17}H_{23}O_3$ $[M+H]^+$ 275.1647, found: 275.1647.

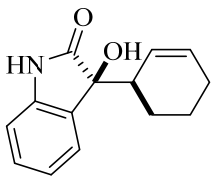


(*Z*)-4-(Cyclooct-2-en-1-yl(hydroxy)methyl)benzonitrile (3q): 118.3 mg, 98% yield, >99:1 dr, white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.64-7.60 (m, 2H), 7.44 (d, *J* = 8.2 Hz, 2H), 5.67-

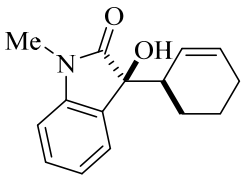
5.60 (m, 1H), 5.27-5.22 (m, 1H), 4.65 (dd, J = 6.9, 3.4 Hz, 1H), 2.91-2.83 (m, 1H), 2.18 (d, J = 3.8 Hz, 1H), 2.13-2.06 (m, 1H), 2.04-1.97 (m, 1H), 1.86-1.78 (m, 1H), 1.69-1.57 (m, 2H), 1.56-1.46 (m, 2H), 1.39-1.19 (m, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 148.8, 132.0, 131.1, 129.3, 127.4, 118.9, 111.1, 77.2, 43.4, 31.1, 29.2, 26.7, 26.6, 25.3 ppm. IR (KBr): ν = 3480, 2923, 2226, 1702, 1456, 758 cm^{-1}. HRMS (m/z): calcd for $C_{16}H_{20}NO$ $[M+H]^+$ 242.1545, found: 242.1545.



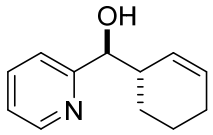
1-(4'-(Cyclohex-2-en-1-yl(hydroxy)methyl)-[1,1'-biphenyl]-4-yl)ethan-1-one (3r): 111.8 mg, 73% yield, >99:1 dr, white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.04-8.01 (m, 2H), 7.70-7.67 (m, 2H), 7.63-7.60 (m, 2H), 7.45 (d, J = 8.2 Hz, 2H), 5.88-5.83 (m, 1H), 5.44 (dd, J = 10.1, 2.4 Hz, 1H), 4.67 (dd, J = 6.6, 1.9 Hz, 1H), 2.64 (s, 3H), 2.57-2.51 (m, 1H), 2.03-1.98 (m, 3H), 1.81-1.69 (m, 2H), 1.61-1.47 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 197.8, 145.4, 143.0, 138.8, 135.7, 130.8, 128.9, 127.8, 127.1, 127.1, 127.0, 76.9, 43.0, 26.7, 25.2, 23.6, 21.1 ppm. IR (KBr): ν = 3531, 2923, 2866, 1672, 1600, 1358, 1270, 812, 674 cm^{-1}. HRMS (m/z): calcd for $C_{21}H_{23}O_2$ $[M+H]^+$ 307.1698, found: 307.1697.



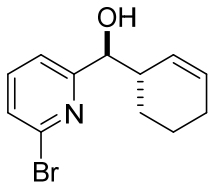
3-(Cyclohex-2-en-1-yl)-3-hydroxyindolin-2-one (3s): 92.8 mg, 81% yield, >99:1 dr, white solid; ^{1}H NMR (400 MHz, Methanol-d_4): δ = 7.29-7.21 (m, 2H), 6.97 (td, J = 7.7, 1.0 Hz, 1H), 6.86 (d, J = 7.7 Hz, 1H), 6.10-6.06 (m, 1H), 5.92-5.87 (m, 1H), 2.80-2.74 (m, 1H), 1.96-1.88 (m, 1H), 1.81-1.72 (m, 1H), 1.63-1.52 (m, 2H), 1.49-1.39 (m, 1H), 0.79-0.69 (m, 1H) ppm. ^{13}C NMR (100 MHz, Methanol-d_4): δ = 181.9, 143.3, 131.2, 131.1, 130.4, 126.5, 126.4, 123.2, 110.8, 79.7, 44.1, 26.0, 24.6, 22.5 ppm. IR (KBr): ν = 3342, 2944, 1705, 1472, 1049, 778, 621 cm^{-1} HRMS (m/z): calcd for $C_{14}H_{16}NO_2$ $[M+H]^+$ 230.1181, found: 230.1154.



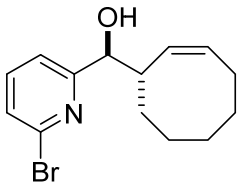
3-(Cyclohex-2-en-1-yl)-3-hydroxy-1-methylindolin-2-one (3t): 101.0 mg, 83% yield, >99:1 dr, white solid; ^{1}H NMR (400 MHz, Methanol-d_4): δ = 7.35-7.31 (m, 2H), 7.05 (td, J = 7.5, 1.0 Hz, 1H), 6.97 (dt, J = 7.5, 1.0 Hz, 1H), 6.09-6.04 (m, 1H), 5.91-5.86 (m, 1H), 3.18 (s, 3H), 2.84-2.77 (m, 1H), 1.95-1.88 (m, 1H), 1.80-1.70 (m, 1H), 1.55-1.37 (m, 3H), 0.71-0.61 (m, 1H) ppm. ^{13}C NMR (100 MHz, Methanol-d_4): δ = 178.5, 143.7, 129.8, 129.2, 129.2, 125.0, 124.6, 122.4, 108.0, 78.0, 42.9, 24.8, 24.6, 23.1, 21.0 ppm. IR (KBr): ν = 3330, 2927, 2836, 1698, 1087, 757, 664 cm^{-1}. HRMS (m/z): calcd for $C_{15}H_{18}NO_2$ $[M+H]^+$ 244.1338, found: 244.1320.



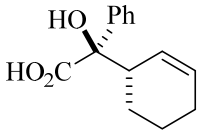
Cyclohex-2-en-1-yl)(pyridin-2-yl)methanol (3u): 68.1 mg, 72% yield, 90:10 dr, white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 8.54 (dt, J = 5.0, 1.4 Hz, 1H), 7.65 (td, J = 7.7, 1.4 Hz, 1H), 7.26 (d, J = 7.7 Hz, 1H), 7.19 (ddd, J = 7.7, 5.0, 1.4 Hz, 1H), 5.83-5.78 (m, 1H), 5.55-5.50 (m, 1H), 4.60 (s, 1H), 4.20 (d, J = 6.0 Hz, 1H), 2.64-2.58 (m, 1H), 1.98-1.93 (m, 2H), 1.73-1.65 (m, 2H), 1.53-1.45 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 160.8, 148.1, 136.3, 130.0, 126.5, 122.2, 121.1, 75.9, 42.9, 25.9, 25.1, 21.7 ppm. IR (KBr): ν = 3156, 2858, 1596, 1111, 760 cm^{-1}. HRMS (m/z): calcd for $C_{12}H_{16}NO$ $[M+H]^+$ 190.1232, found: 190.1232.



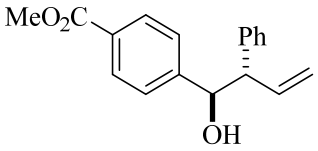
(*S*)-*(*6-Bromopyridin-2-yl)((*S*)-cyclohex-2-en-1-yl)methanol (3v): 107.3 mg, 80% yield, >99:1 dr, white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.53 (t, J = 7.8 Hz, 1H), 7.37 (d, J = 7.8 Hz, 1H), 7.30-7.28 (m, 1H), 5.90-5.85 (m, 1H), 5.52-5.47 (m, 1H), 4.68 (t, J = 5.3 Hz, 1H), 3.08 (d, J = 5.5 Hz, 1H), 2.64-2.59 (m, 1H), 1.99-1.98 (m, 2H), 1.71-1.68 (m, 1H), 1.55-1.40 (m, 3H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 162.9, 141.1, 138.6, 130.8, 127.8, 126.5, 119.9, 75.9, 42.4, 25.0, 22.5, 21.0 ppm. IR (KBr): ν = 3385, 2927, 1585, 1409, 990, 696 cm^{-1}. HRMS (m/z): calcd for $C_{12}H_{15}NOBr$ $[M+H]^+$ 268.0337, found: 268.0337.



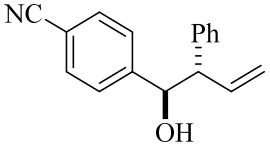
(*S*)-(6-Bromopyridin-2-yl)((*S*,*Z*)-cyclooct-2-en-1-yl)methanol (3w): 125.9 mg, 85% yield, >99:1 dr, white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.52 (t, J = 7.7 Hz, 1H), 7.37 (d, J = 7.8 Hz, 1H), 7.23 (d, J = 7.6 Hz, 1H), 5.70-5.63 (m, 1H), 5.50-5.45 (m, 1H), 4.62 (t, J = 6.2 Hz, 1H), 3.49 (d, J = 6.8 Hz, 1H), 2.91-2.83 (m, 1H), 2.20-2.11 (m, 1H), 2.04-1.96 (m, 1H), 1.71-1.59 (m, 4H), 1.54-1.46 (m, 2H), 1.41-1.24 (m, 4H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 163.2, 141.0, 138.7, 130.7, 130.1, 126.7, 120.1, 76.1, 43.4, 30.4, 29.3, 26.7, 26.5, 25.3 ppm. IR (KBr): ν = 3330, 2919, 2853, 1556, 1404, 1129, 753 cm^{-1}. HRMS (m/z): calcd for $C_{14}H_{19}NOBr$ $[M+H]^+$ 296.0650, found: 296.0650.



2-(Cyclohex-2-en-1-yl)-2-hydroxy-2-phenylacetic acid (3x): 75.5 mg, 65% yield, >99:1 dr, white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.72-7.62 (m, 2H), 7.45-7.24 (m, 3H), 6.51 (s, 1H), 6.00 (d, J = 10.6 Hz, 1H), 5.59-5.49 (s, 1H), 3.26 (s, 1H), 2.00 (s, 2H), 1.75-1.71 (m, 1H), 1.49-1.42 (m, 1H), 1.31-1.28 (m, 2H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 178.9, 139.1, 132.7, 128.2, 127.7, 125.9, 125.8, 80.4, 43.7, 25.0, 22.7, 21.6 ppm. IR (KBr): ν = 3411, 2923, 1723, 1227, 719 cm^{-1}. HRMS (m/z): calcd for $C_{14}H_{17}O_3$ $[M+H]^+$ 233.1178, found: 233.1172.

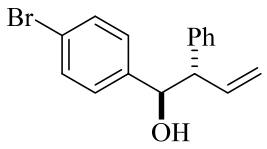


Methyl 4-(1-hydroxy-2-phenylbut-3-en-1-yl)benzoate (4a): 94.6 mg, 67% yield, >99:1 dr, white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.88-7.85 (m, 2H), 7.23-7.13 (m, 5H), 7.05-7.02 (m, 2 H), 6.28-6.19 (m, 1H), 5.30-5.21 (m, 2H), 4.88 (dd, J = 7.8, 2.4 Hz, 1H), 3.87 (s, 3H), 3.51 (t, J = 8.4 Hz, 1H), 2.48 (d, J = 2.5 Hz, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 166.9, 147.0, 140.0, 137.3, 129.2, 129.1, 128.5, 128.2, 126.8, 126.6, 118.9, 76.8, 59.3, 52.0 ppm. IR (KBr): ν = 3508, 2958, 2898, 1694, 1431, 1289, 1113, 924, 721 cm^{-1}. HRMS (m/z): calcd for $C_{18}H_{19}O_3$ $[M+H]^+$ 283.1334, found: 283.1339.

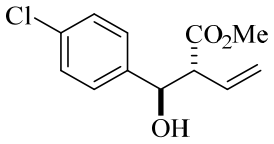


4-(1-Hydroxy-2-phenylbut-3-en-1-yl)benzonitrile (4b): 91.0 mg, 73% yield, >99:1 dr, white solid; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.47 (d, J = 7.9 Hz, 2H), 7.22-7.20 (m, 5H), 7.02 (d, J = 7.9 Hz, 2H), 6.26-6.17 (m, 1H), 5.31-5.22 (m, 2H), 4.85 (dd, J = 7.8, 2.1 Hz, 1H), 3.44 (t, J = 8.6

Hz, 1H), 2.56 (d, J = 2.4 Hz, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 147.1, 139.6, 136.9, 131.6, 128.6, 128.1, 127.3, 127.0, 119.3, 118.8, 111.0, 76.5, 59.4 ppm. IR (KBr): ν = 3508, 3083, 2887, 2226, 1605, 1391, 1067, 920, 704 cm^{-1}. HRMS (m/z): calcd for $C_{17}H_{16}NO$ $[M+H]^+$ 250.1232, found: 250.1232.



1-(4-Bromophenyl)-2-phenylbut-3-en-1-ol (4c): 75.8 mg, 50% yield, >99:1 dr, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.32-7.30 (m, 2H), 7.24-7.15 (m, 3H), 7.03-6.97 (m, 4H), 6.25-6.16 (m, 1H), 5.28-5.21 (m, 2H), 4.77 (d, J = 9.4 Hz, 1H), 3.48-3.43 (m, 1H), 2.40 (s, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 140.7, 140.1, 137.5, 130.9, 128.5, 128.3, 128.2, 126.8, 121.2, 118.8, 76.5, 59.3 ppm. IR (KBr): ν = 3432, 3028, 2897, 1487, 1070, 1010, 819, 701 cm^{-1}. HRMS (m/z): calcd for $C_{16}H_{16}BrO$ $[M+H]^+$ 303.0385, found: 303.0381.



Methyl 2-((4-chlorophenyl)(hydroxy)methyl)but-3-enoate (4d): 66.2 mg, 55% yield, 90:10 dr, colorless oil; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.25-7.22 (m, 2H), 7.19-7.16 (m, 2H), 5.60 (ddd, J = 17.2, 10.2, 8.7 Hz, 1H), 5.03 (d, J = 10.2 Hz, 1H), 4.95 (d, J = 17.2 Hz, 1H), 4.84 (dd, J = 8.2, 4.9 Hz, 1H), 3.66 (s, 3H), 3.31 (t, J = 8.4 Hz, 1H), 2.98-2.97 (m, 1H) ppm. ^{13}C NMR (100 MHz, $CDCl_3$): δ = 173.2, 139.5, 133.7, 131.7, 128.5, 128.0, 120.0, 74.5, 57.8, 52.2 ppm. IR (KBr): ν = 3689, 3429, 2952, 1712, 1435, 1068, 828 cm^{-1}. HRMS (m/z): calcd for $C_{12}H_{14}ClO_3$ $[M+H]^+$ 241.0631, found: 241.0631.

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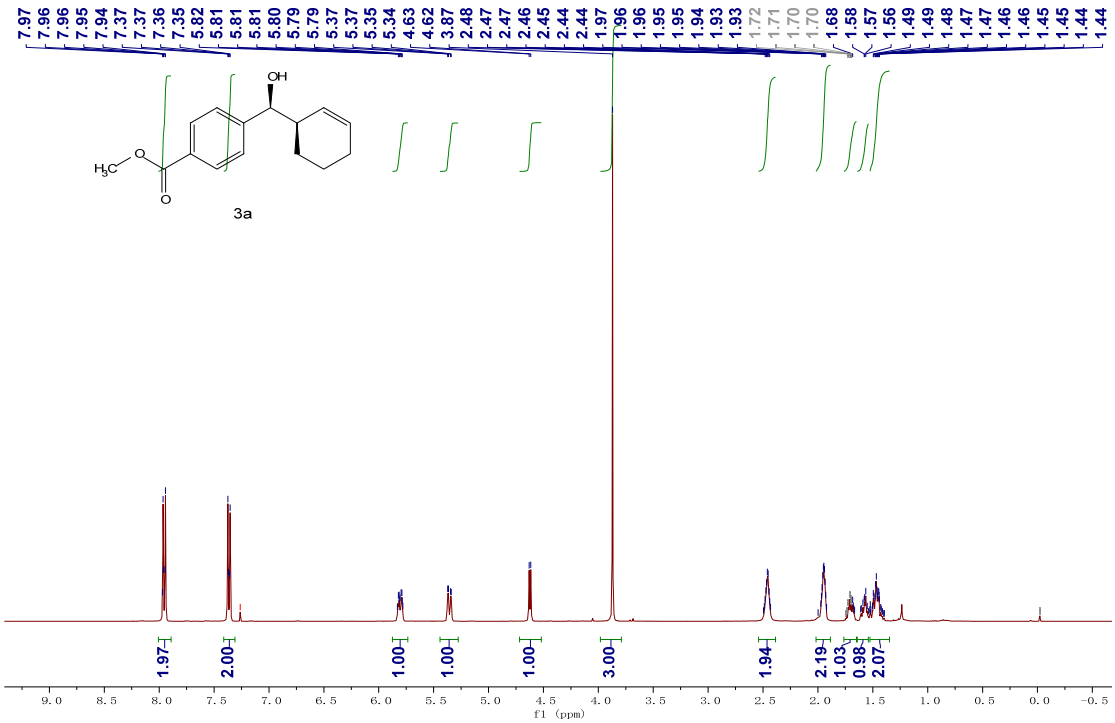
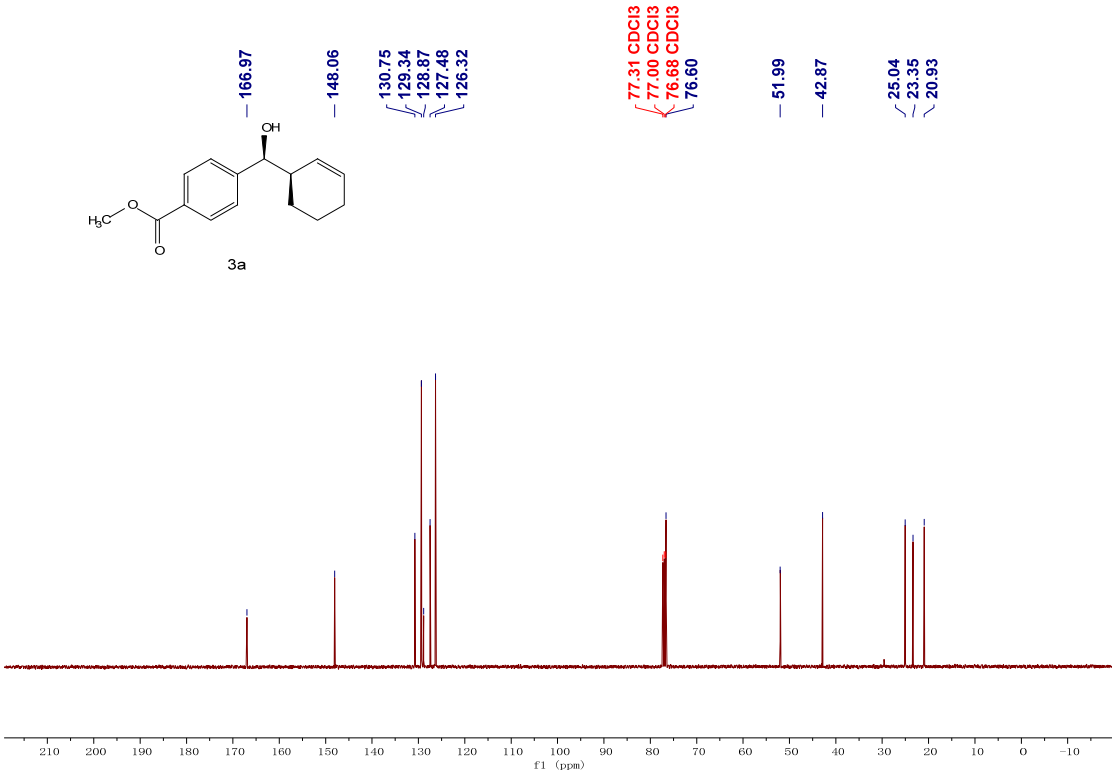
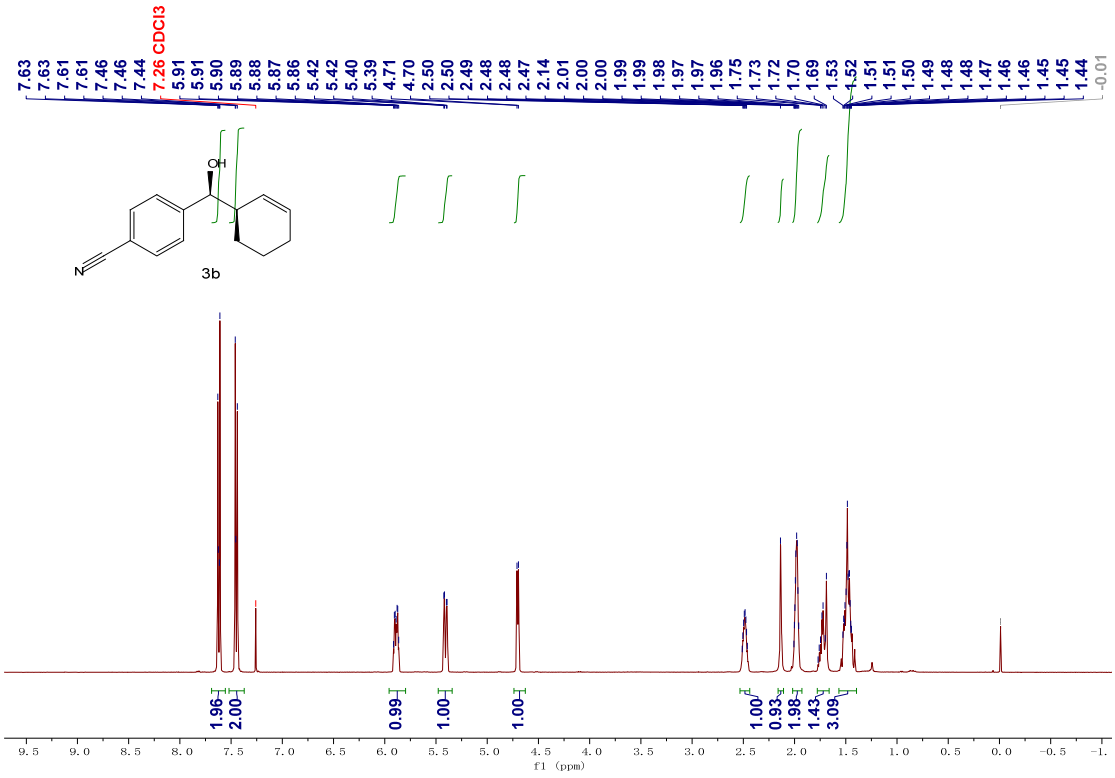
^{1}H and ^{13}C NMR spectra of products



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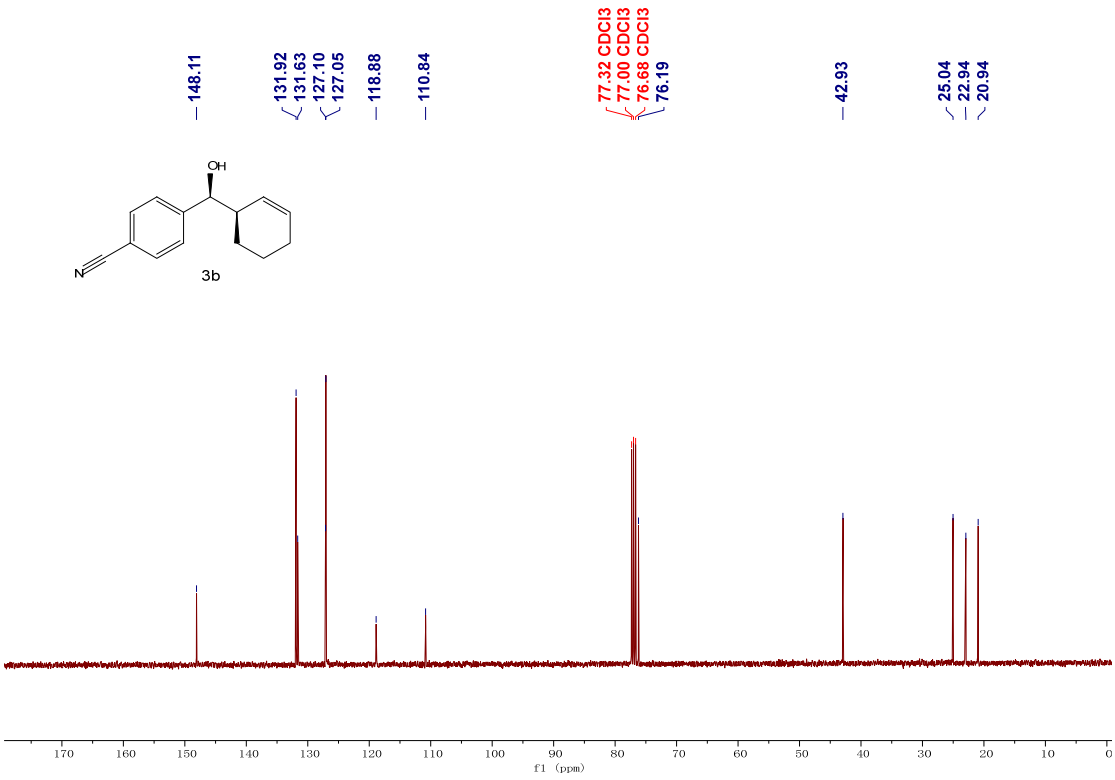
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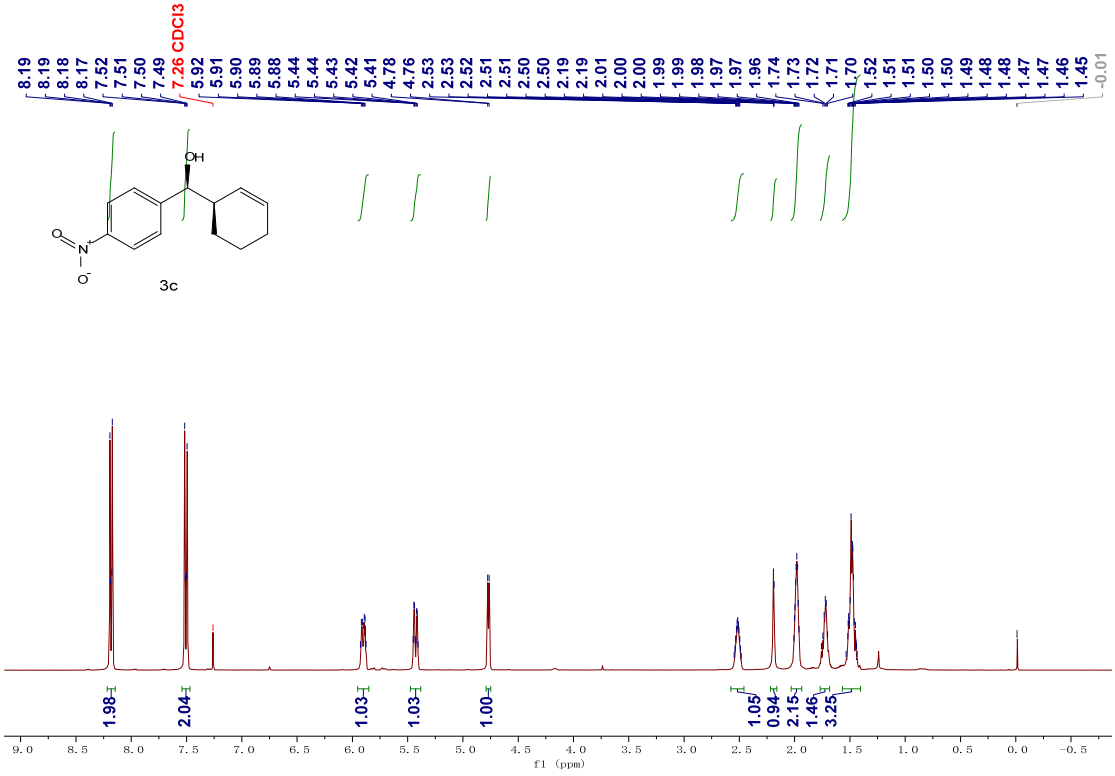
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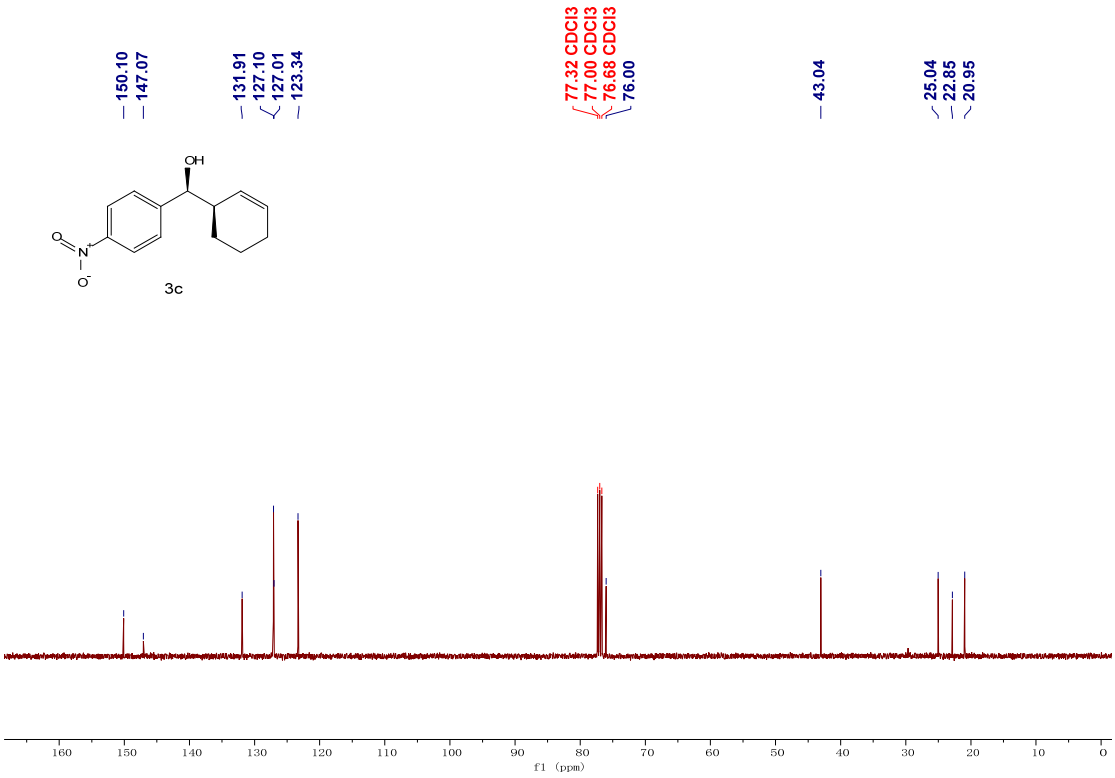
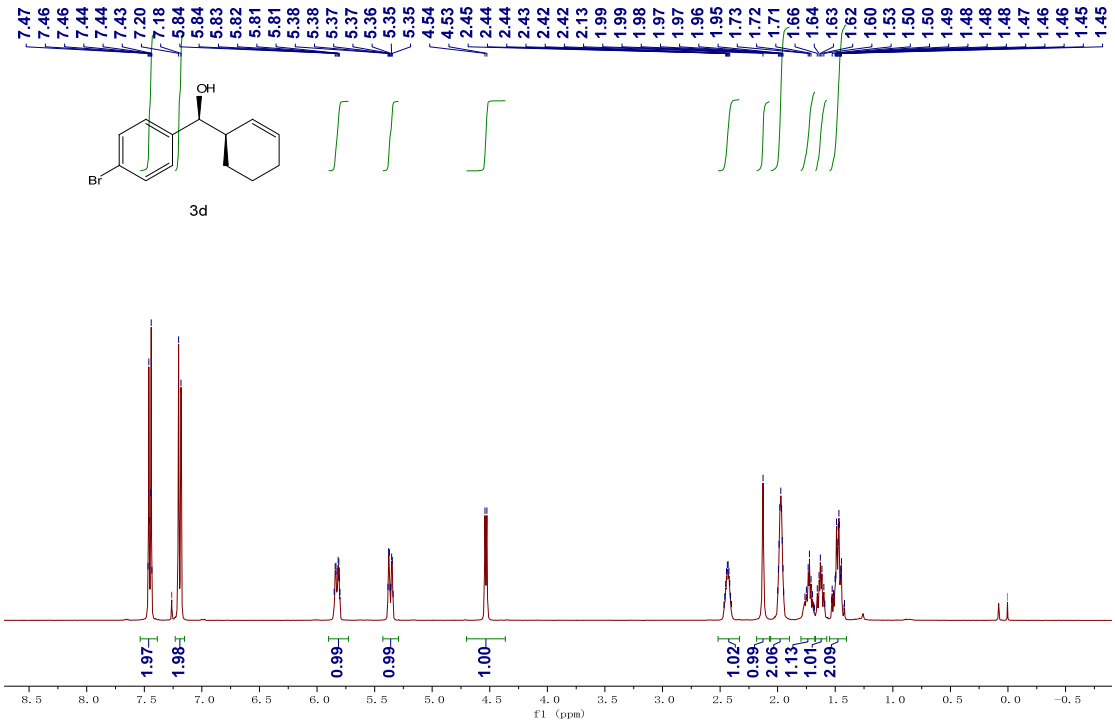
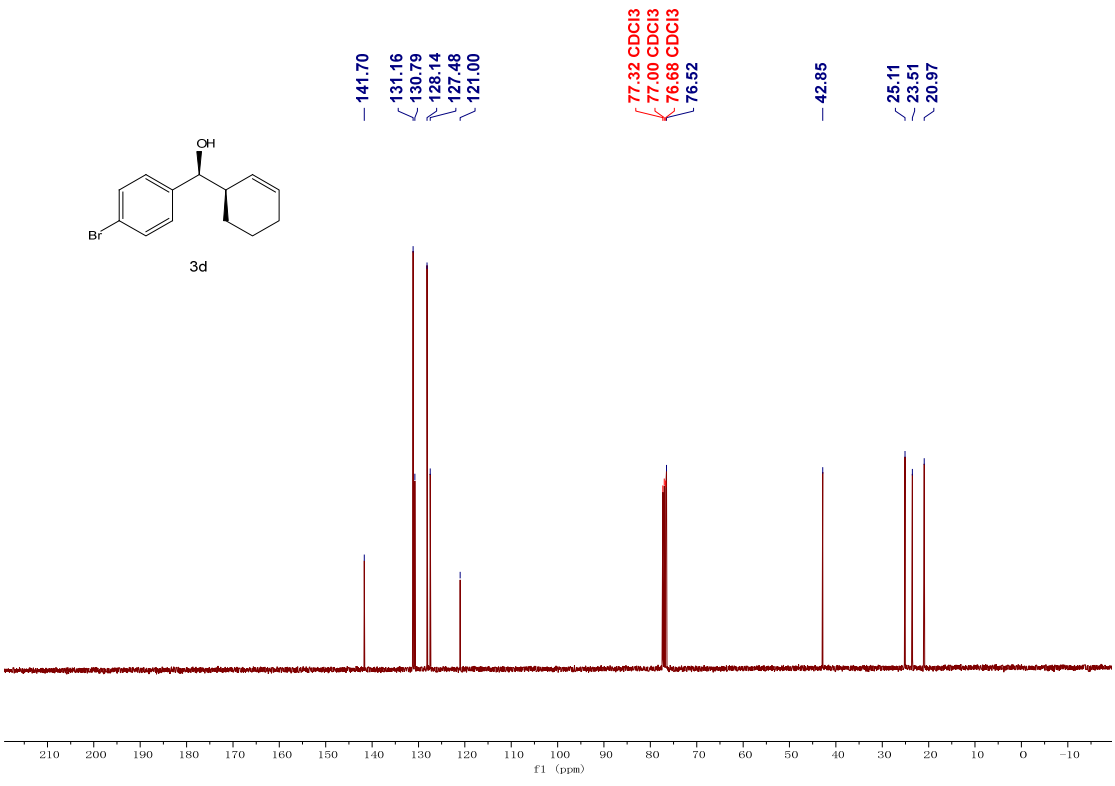
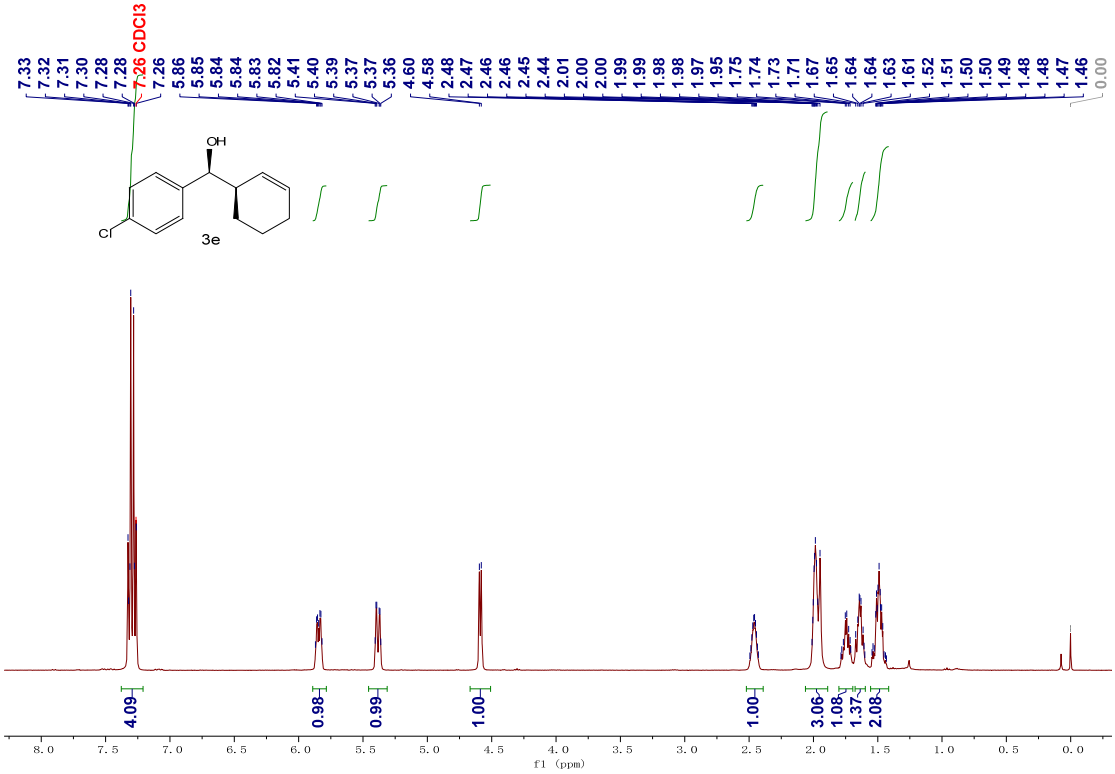
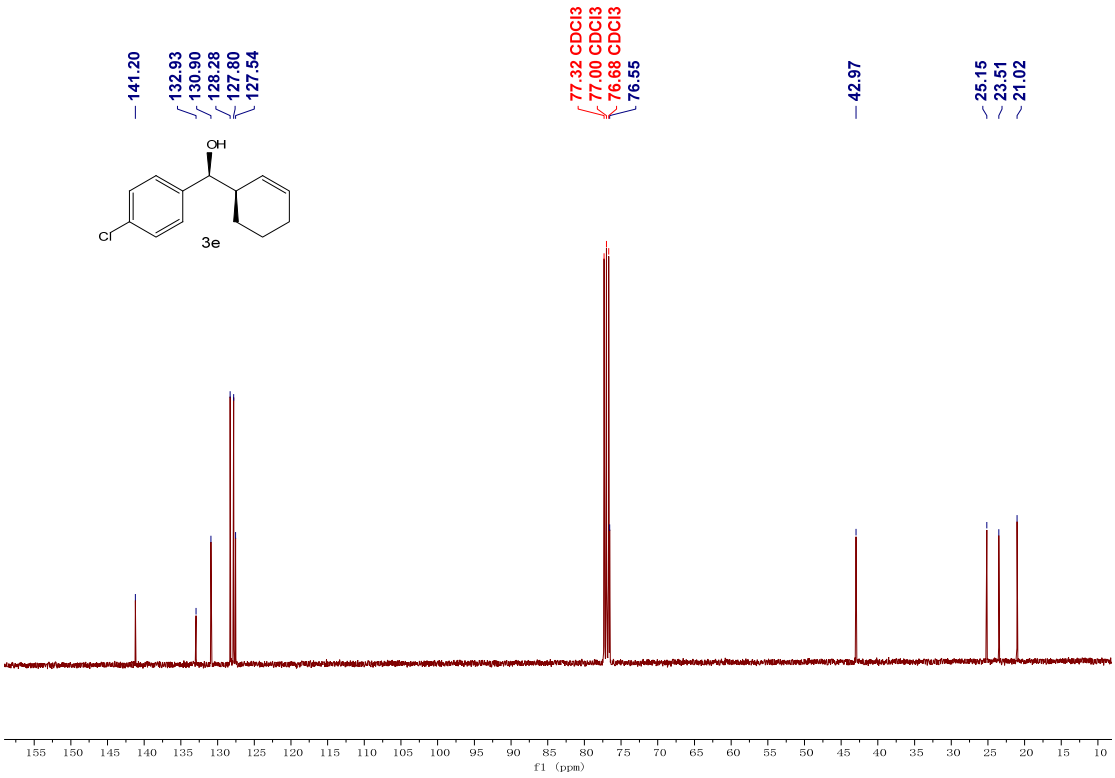
1.70
1.52
1.51
1.51
1.50
1.50
1.49
1.48
1.48
1.47
1.47
1.46
1.45
-0.01
OH
O
N+
O-
3c
1.98
2.04
1.03
1.03
1.00
1.05
0.94
2.15
1.46
3.25
f1 (ppm)

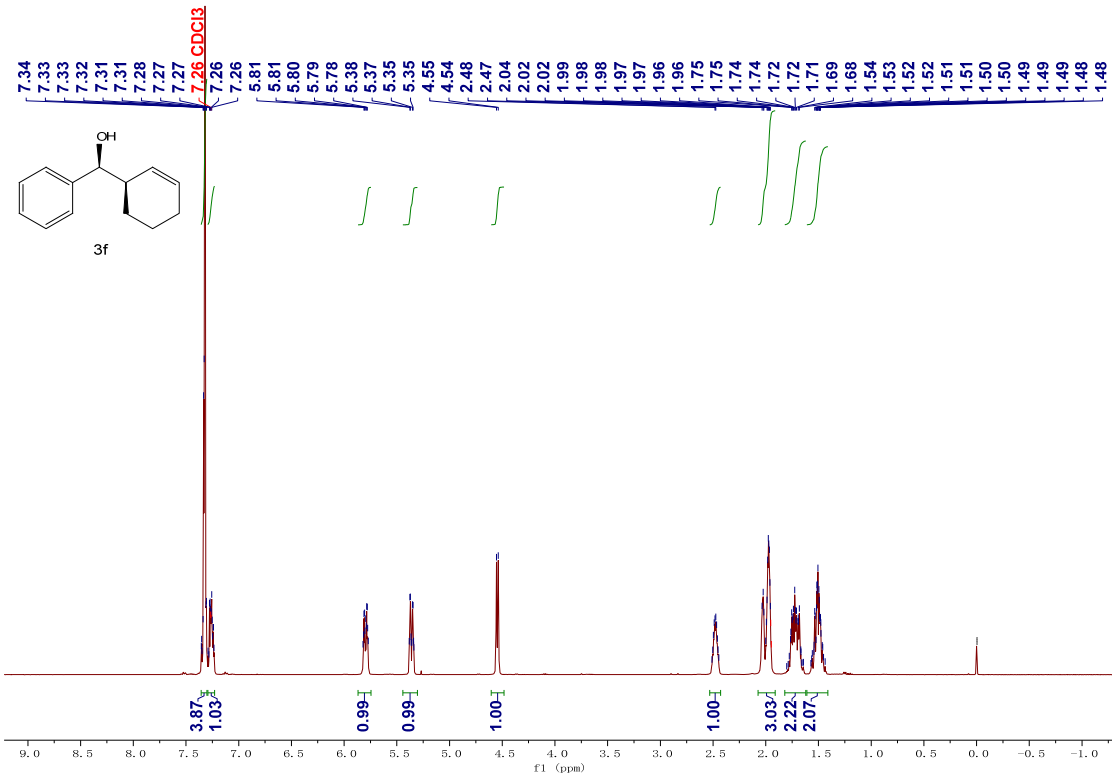

150.10
147.07
131.91
127.10
127.01
123.34
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
76.00
43.04
25.04
22.85
20.95
OH
O
N+
O-
3c
f1 (ppm)

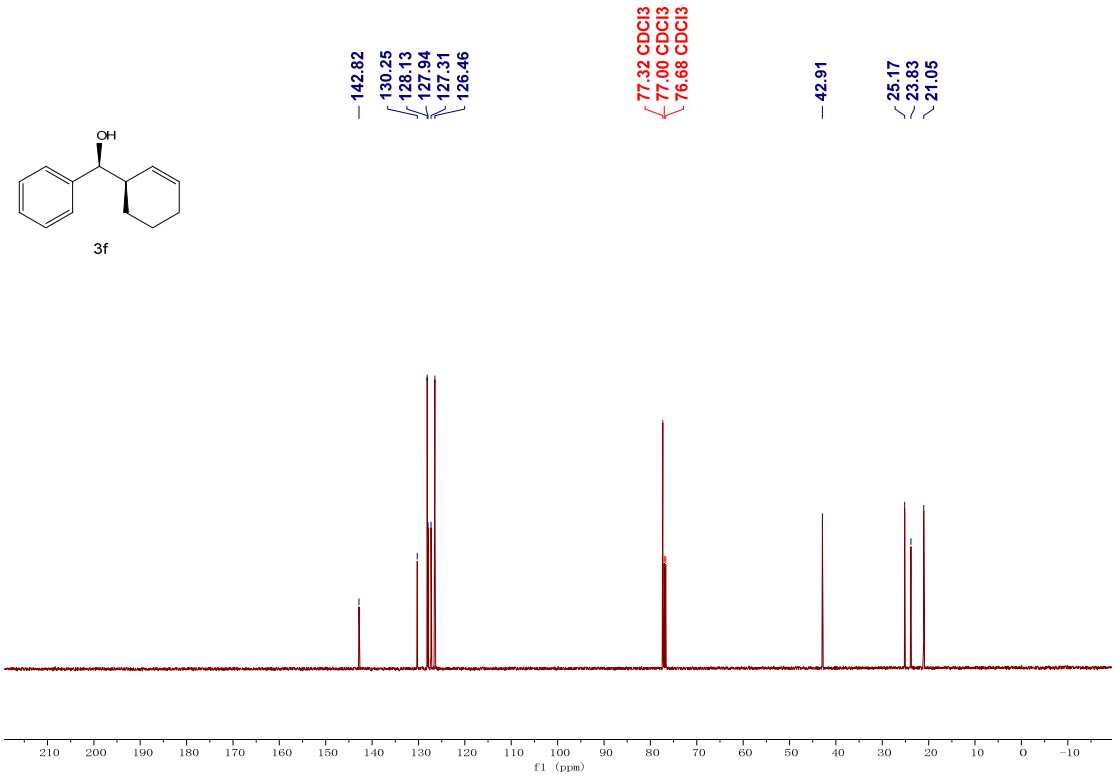

7.47 7.46 7.46 7.44 7.44 7.43 7.20 7.18 5.84 5.84 5.83 5.82 5.81 5.81 5.38 5.38 5.37 5.37 5.36 5.35 5.35 4.54 4.53 2.45 2.44 2.44 2.43 2.42 2.42 2.13 1.99 1.99 1.98 1.97 1.97 1.96 1.95 1.73 1.72 1.71 1.66 1.64 1.63 1.62 1.60 1.53 1.50 1.50 1.49 1.48 1.48 1.48 1.47 1.46 1.46 1.45 1.45
OH
Br
3d
1.97 1.98 0.99 0.99 1.00 1.02 0.99 2.06 1.13 1.01 2.09
f1 (ppm)

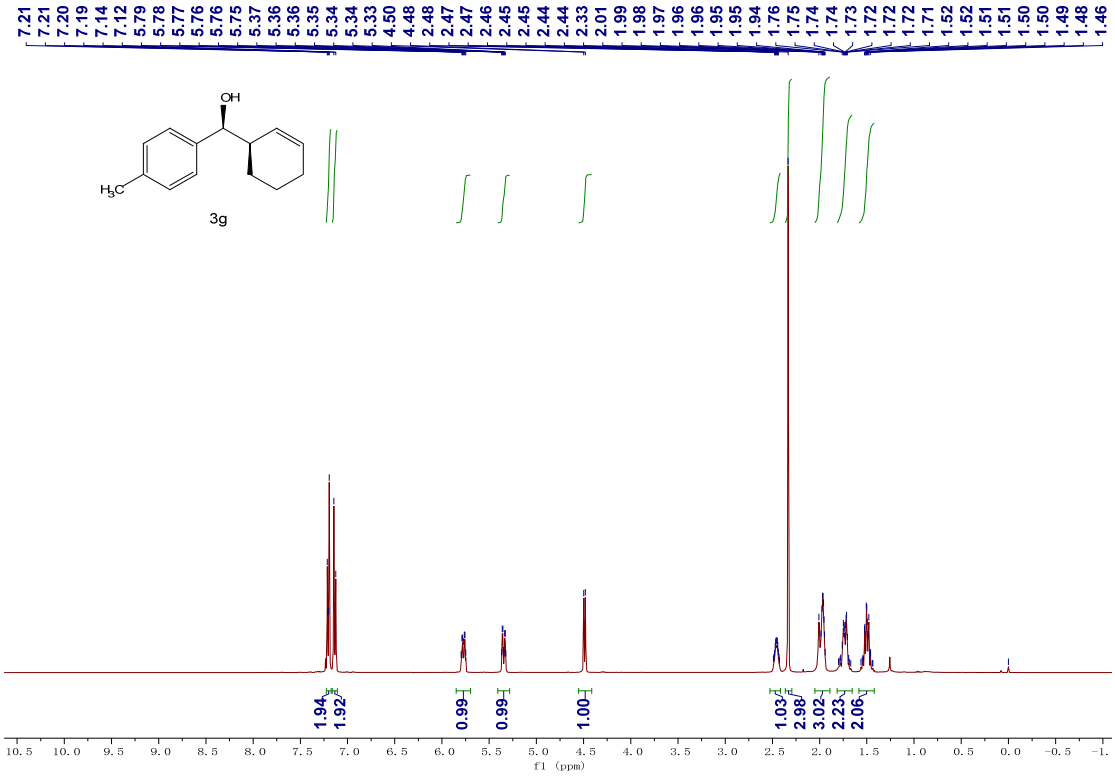

141.70
131.16
130.79
128.14
127.48
121.00
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
76.52
42.85
25.11
23.51
20.97
OH
Br
3d
f1 (ppm)

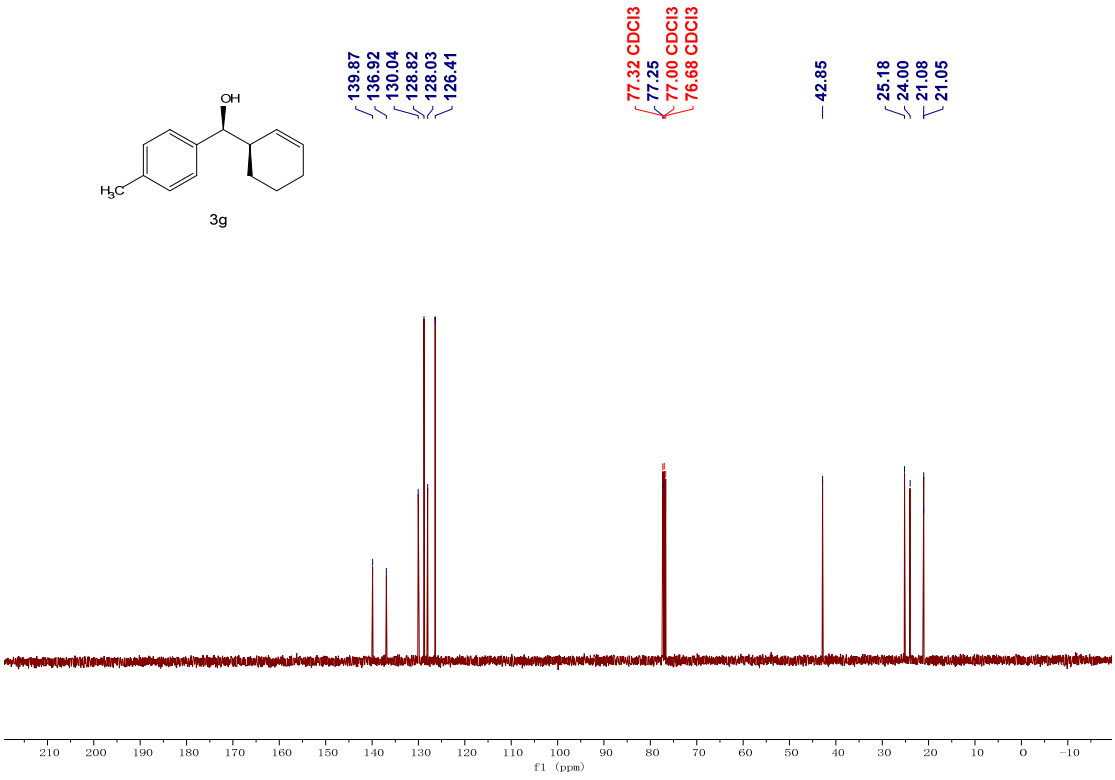

7.33 7.32 7.31 7.30 7.28 7.28 7.26 CDCl3 7.26
5.86 5.85 5.84 5.84 5.83 5.82 5.41 5.40 5.39 5.37 5.37 5.36
4.60 4.58 2.48 2.47 2.46 2.46 2.45 2.44 2.01 2.00 2.00 1.99 1.99 1.98 1.98 1.97 1.95 1.75 1.74 1.73 1.71 1.67 1.65 1.64 1.64 1.63 1.61 1.52 1.51 1.50 1.50 1.49 1.48 1.48 1.47 1.46 0.00
OH
Cl
3e
4.09
0.98
0.99
1.00
1.00
3.06
1.08
1.37
2.08
f1 (ppm)

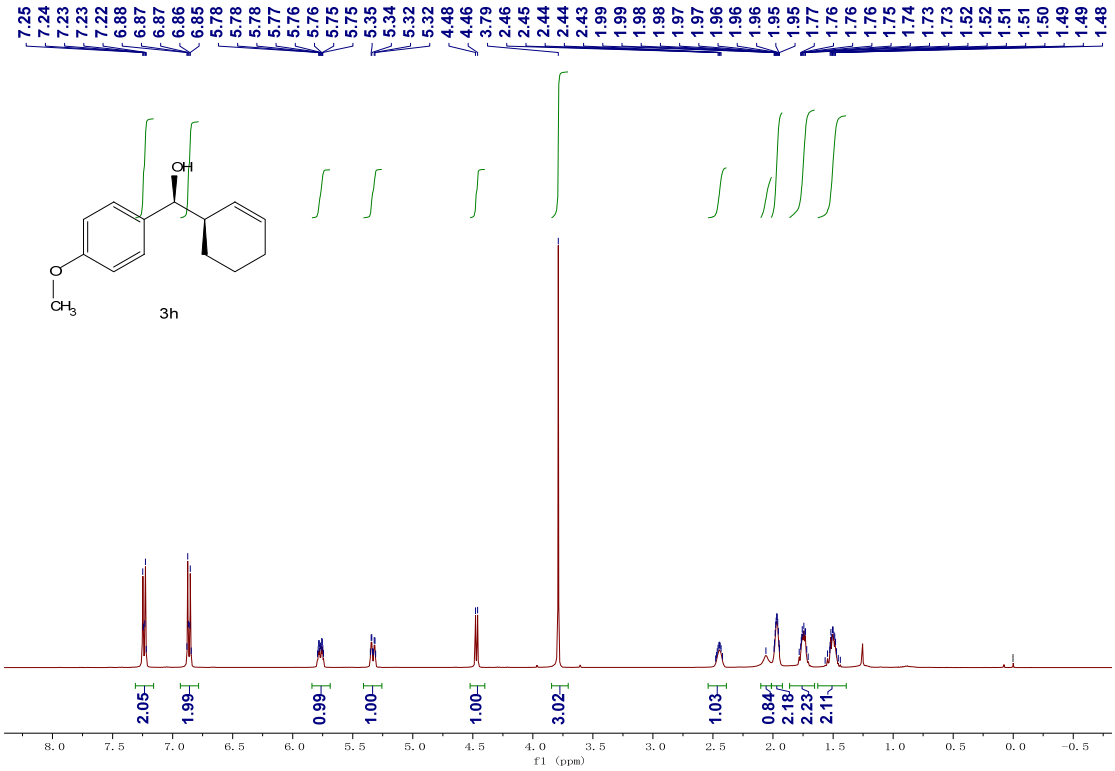

141.20
132.93
130.90
128.28
127.80
127.54
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
76.55
42.97
25.15
23.51
21.02
OH
Cl
3e
f1 (ppm)

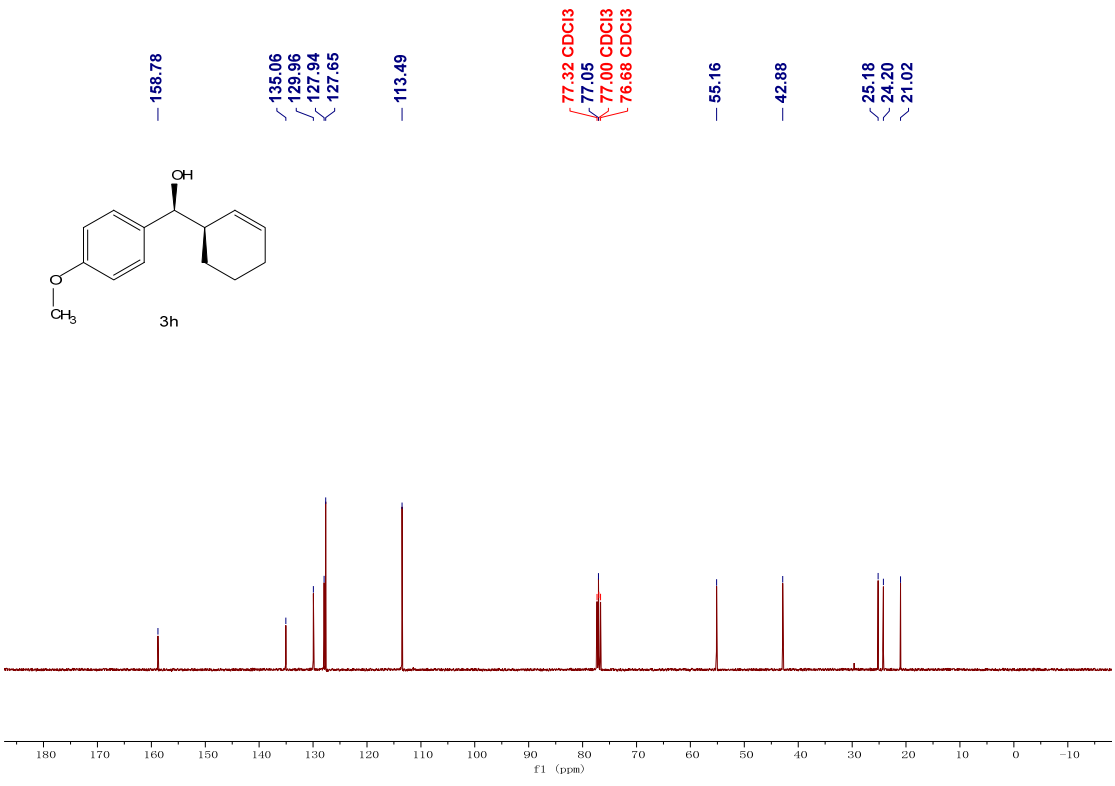

7.34
7.33
7.33
7.32
7.31
7.31
7.28
7.27
7.27
7.26 CDCl3
7.26
7.26
5.81
5.80
5.79
5.78
5.38
5.37
5.35
5.35
4.55
4.54
2.48
2.47
2.04
2.02
2.02
1.99
1.98
1.98
1.97
1.97
1.96
1.96
1.75
1.75
1.74
1.74
1.72
1.72
1.71
1.69
1.68
1.54
1.53
1.52
1.52
1.51
1.51
1.50
1.50
1.49
1.49
1.49
1.48
1.48
OH
3f
3.87
1.03
0.99
0.99
1.00
1.00
3.03
2.22
2.07
f1 (ppm)

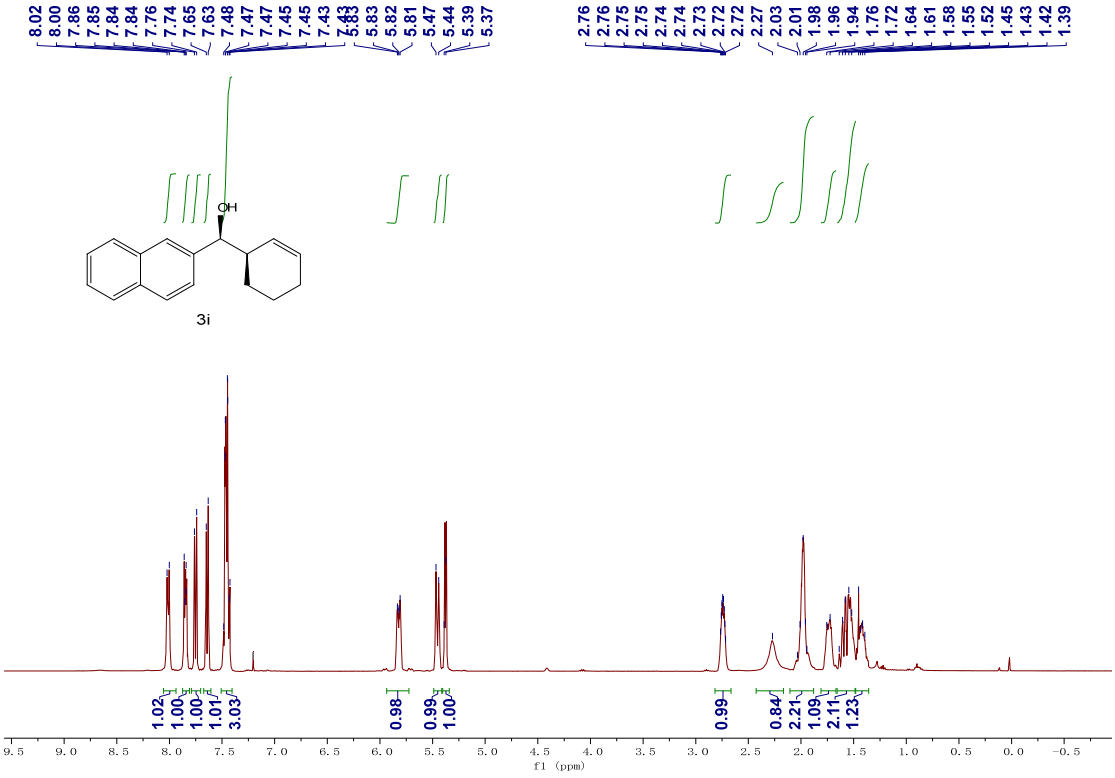

142.82
130.25
128.13
127.94
127.31
126.46
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
42.91
25.17
23.83
21.05
OH
3f
f1 (ppm)

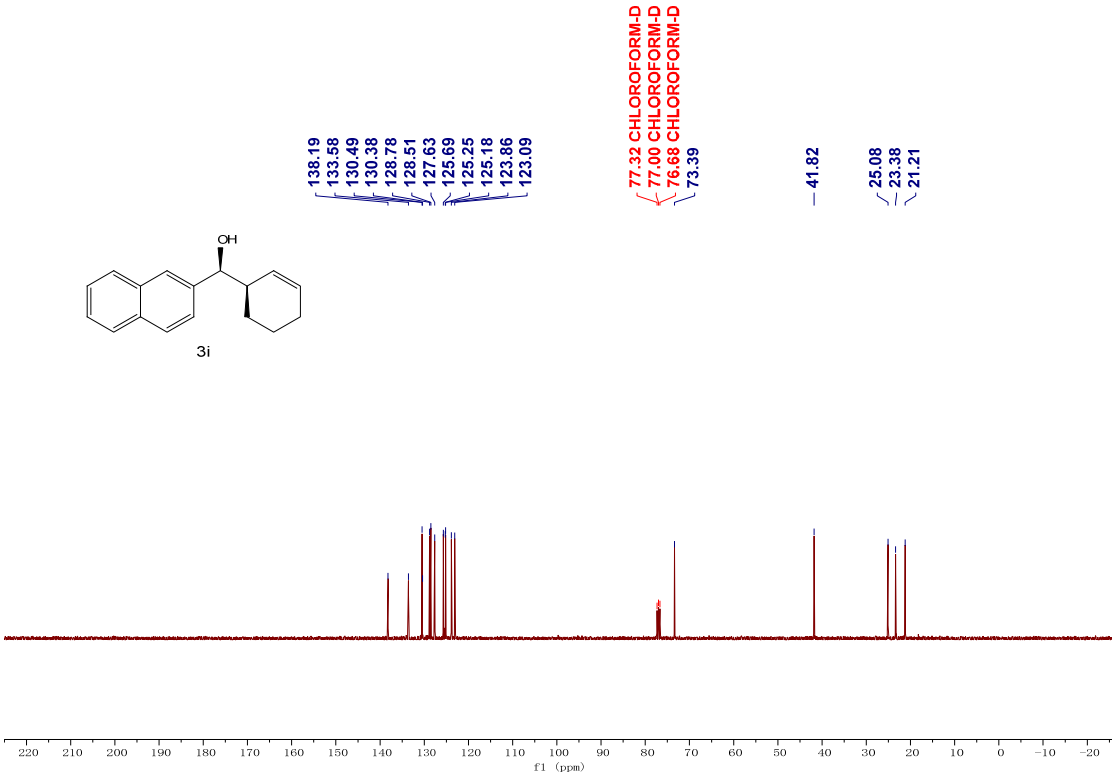

7.21 7.21 7.20 7.19 7.14 7.12 5.79 5.78 5.77 5.76 5.76 5.75 5.37 5.36 5.36 5.35 5.34 5.34 5.33 4.50 4.48 2.48 2.47 2.47 2.46 2.45 2.45 2.44 2.44 2.33 2.01 1.99 1.98 1.97 1.96 1.96 1.95 1.95 1.94 1.76 1.75 1.74 1.74 1.73 1.72 1.72 1.72 1.71 1.52 1.52 1.51 1.51 1.50 1.50 1.49 1.48 1.46
OH
H3C
3g
1.94
1.92
0.99
0.99
1.00
1.03
2.98
3.02
2.23
2.06
f1 (ppm)

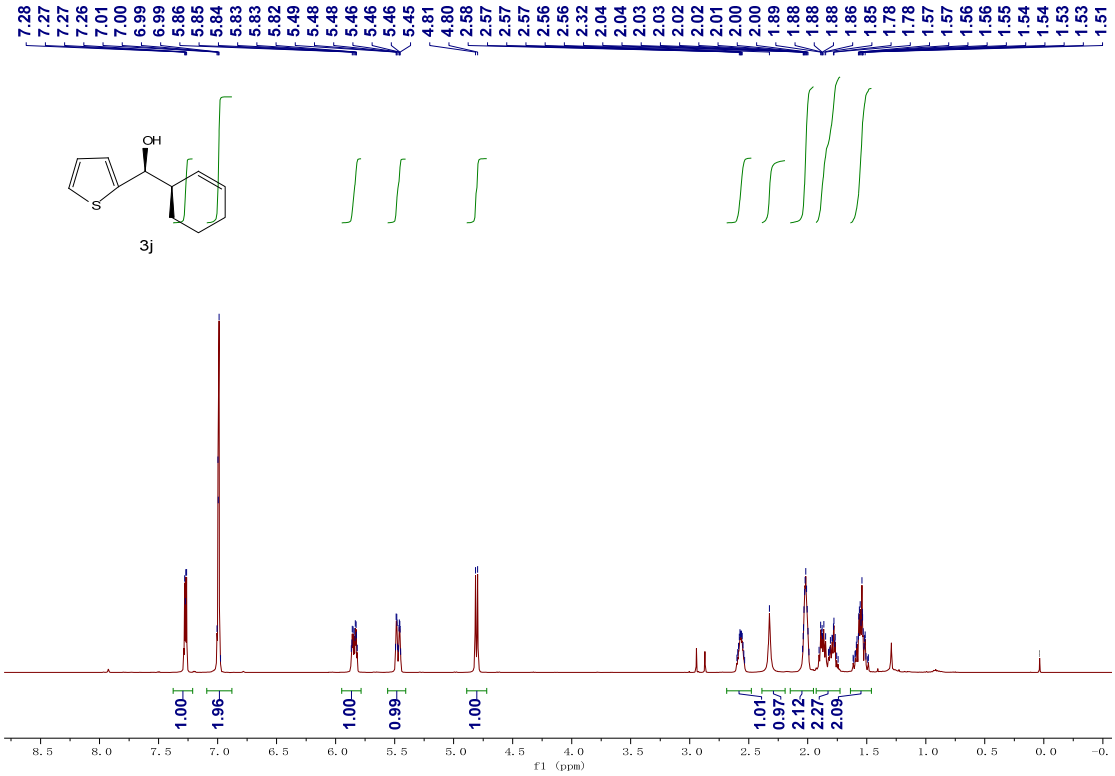

139.87
136.92
130.04
128.82
128.03
126.41
77.32 CDCl3
77.25
77.00 CDCl3
76.68 CDCl3
42.85
25.18
24.00
21.08
21.05
OH
H3C
3g
f1 (ppm)

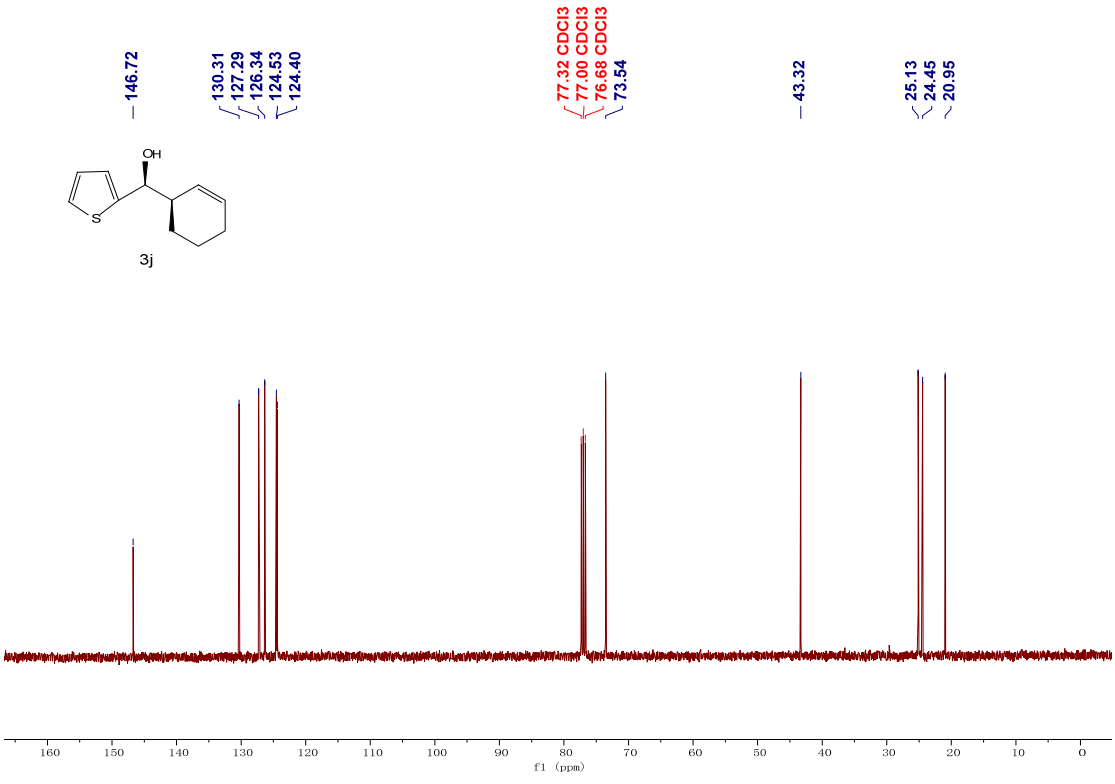

7.25 7.24 7.23 7.23 7.22 6.88 6.87 6.86 6.85 5.78 5.78 5.77 5.76 5.76 5.75 5.75 5.35 5.34 5.32 5.32 4.48 4.46 3.79 2.46 2.45 2.44 2.44 2.43 1.99 1.99 1.98 1.98 1.97 1.97 1.96 1.96 1.96 1.95 1.95 1.77 1.76 1.76 1.76 1.75 1.74 1.73 1.73 1.52 1.52 1.51 1.51 1.50 1.49 1.49 1.48
OH
O
CH3
3h
2.05 1.99 0.99 1.00 1.00 3.02 1.03 0.84 2.18 2.23 2.11
8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0 -0.5
f1 (ppm)

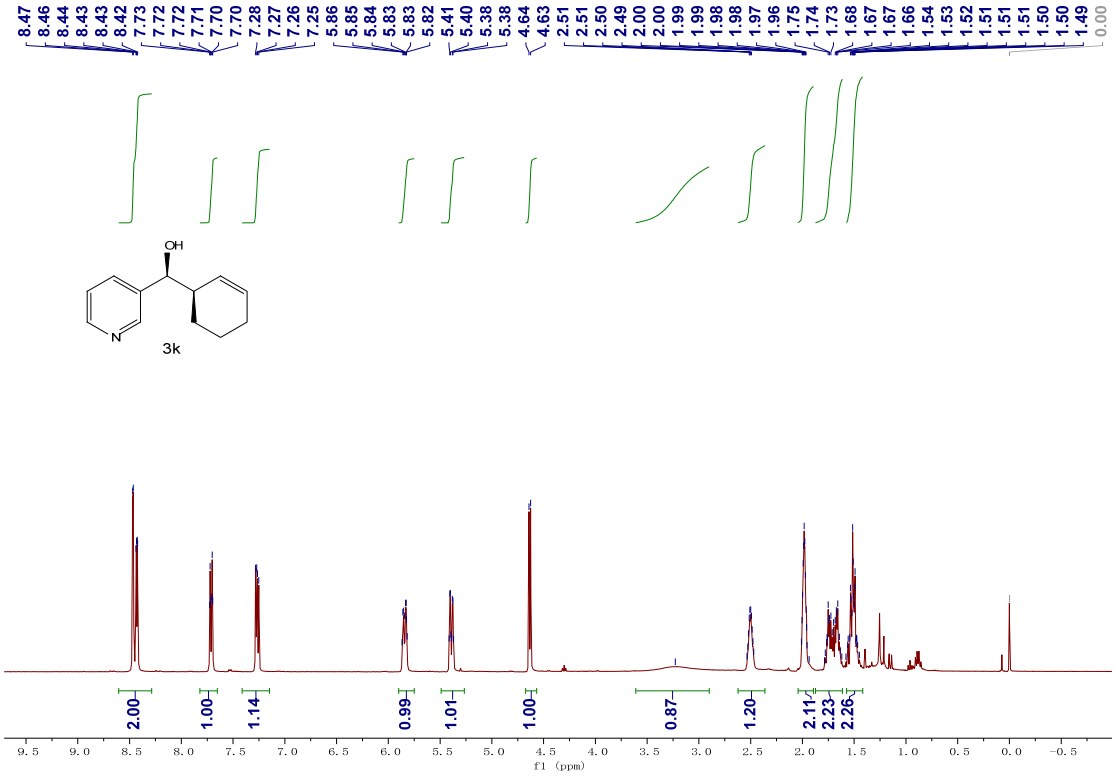

158.78
135.06 129.96 127.94 127.65
113.49
77.32 CDCl3 77.05 77.00 CDCl3 76.68 CDCl3
55.16
42.88
25.18 24.20 21.02
OH
O
CH3
3h
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 -10
f1 (ppm)

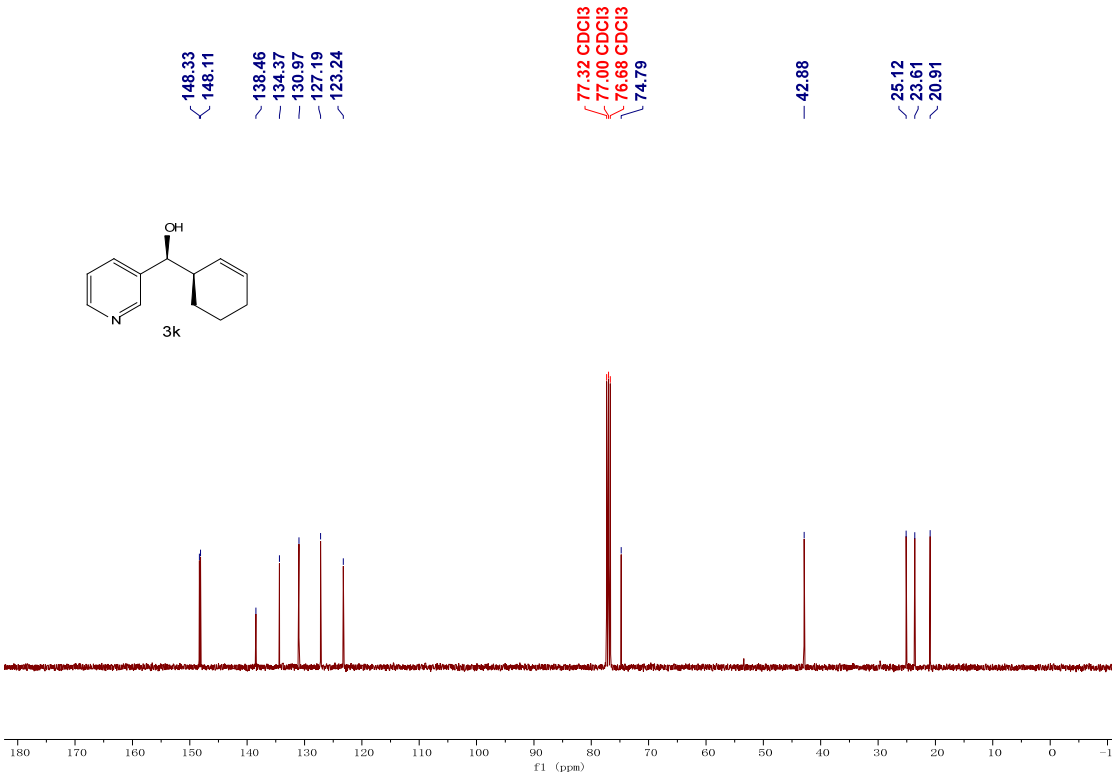

8.02 8.00 7.86 7.85 7.84 7.84 7.76 7.74 7.65 7.63 7.48 7.47 7.47 7.45 7.45 7.43 5.83 5.82 5.81 5.47 5.44 5.39 5.37
2.76 2.76 2.75 2.75 2.74 2.74 2.73 2.72 2.72 2.27 2.03 2.01 1.98 1.96 1.94 1.76 1.72 1.64 1.61 1.58 1.55 1.52 1.45 1.43 1.42 1.39
OH
3i
1.02 1.00 1.00 1.01 3.03 0.98 0.99 1.00 0.99 0.84 2.21 1.09 2.11 1.23
f1 (ppm)

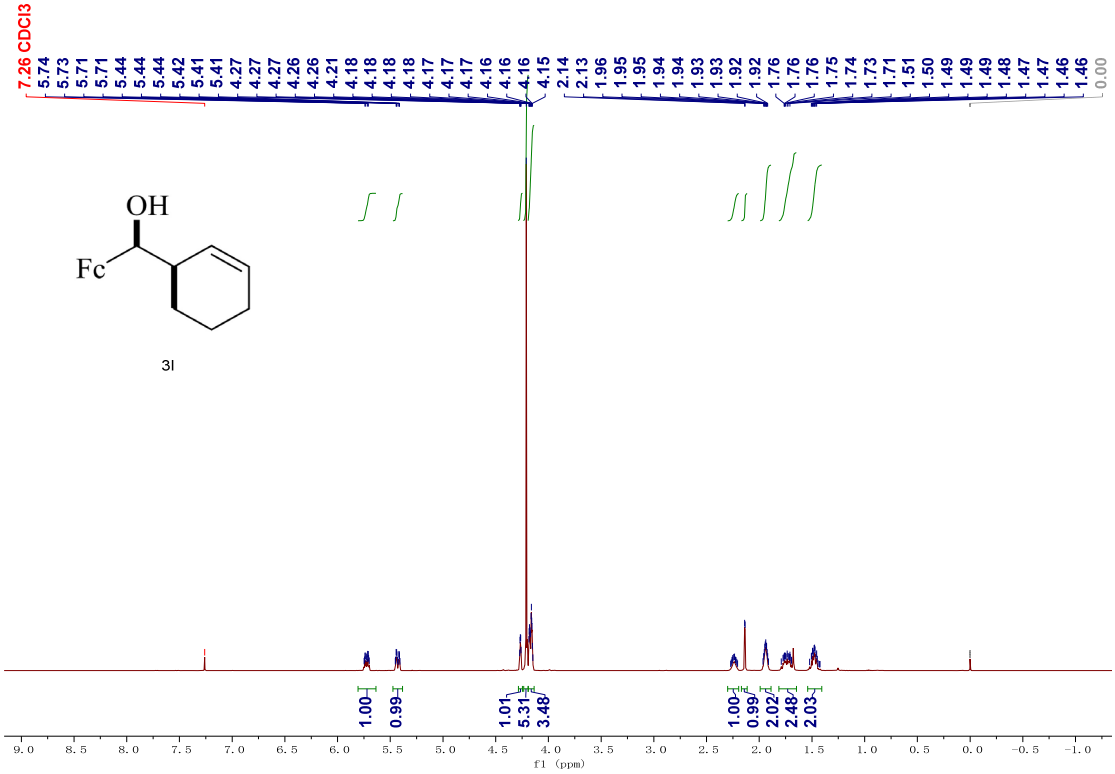

138.19 133.58 130.49 130.38 128.78 128.51 127.63 125.69 125.25 125.18 123.86 123.09
77.32 CHLOROFORM-D
77.00 CHLOROFORM-D
76.68 CHLOROFORM-D
73.39
41.82
25.08 23.38 21.21
OH
3i
f1 (ppm)


7.28 7.27 7.27 7.26 7.01 7.00 6.99 6.99 5.86 5.85 5.84 5.83 5.83 5.82 5.49 5.48 5.48 5.46 5.46 5.46 5.45
4.81 4.80 2.58 2.57 2.57 2.57 2.56 2.56 2.32 2.04 2.04 2.03 2.03 2.02 2.02 2.01 2.00 2.00 1.89 1.88 1.88 1.88 1.86 1.85 1.78 1.78 1.57 1.57 1.56 1.56 1.55 1.54 1.54 1.53 1.51
OH
S
3j
1.00 1.96 1.00 0.99 1.00 1.01 0.97 2.12 2.27 2.09
f1 (ppm)

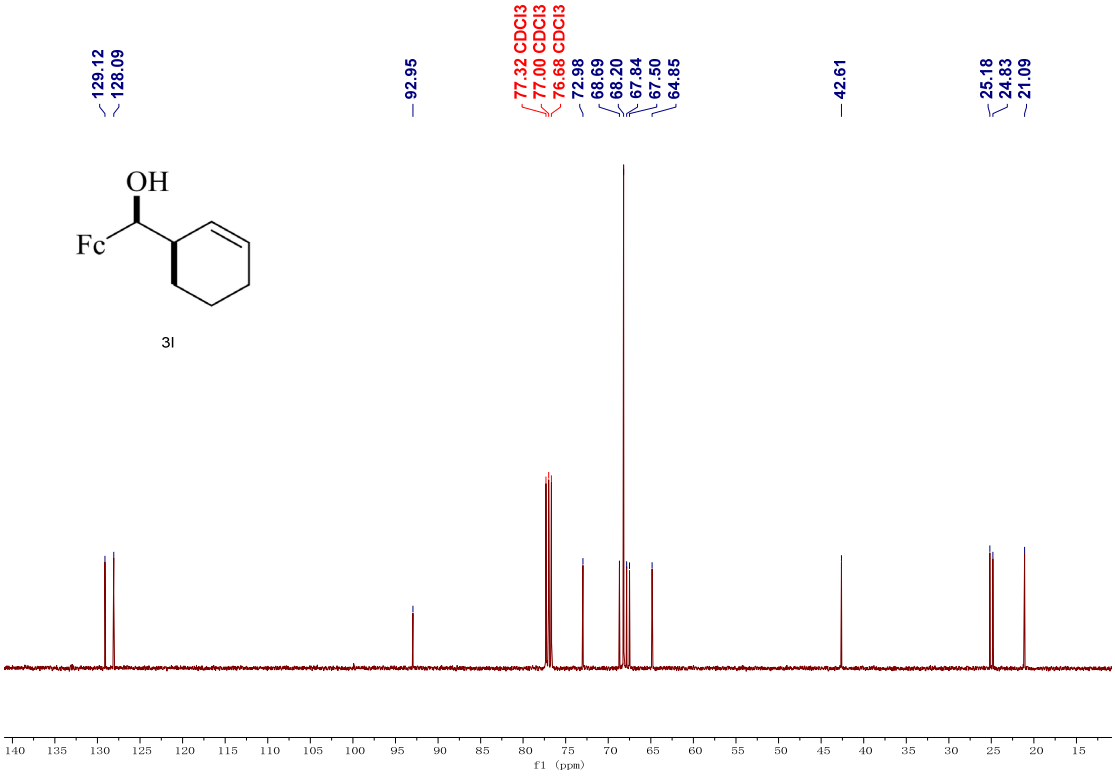

146.72
130.31 127.29 126.34 124.53 124.40
77.32 CDCl3 77.00 CDCl3 76.68 CDCl3 73.54
43.32
25.13 24.45 20.95
OH
S
3j
f1 (ppm)


8.47 8.46 8.44 8.43 8.43 8.42 7.73 7.72 7.72 7.71 7.70 7.70 7.28 7.27 7.26 7.25 5.86 5.85 5.84 5.83 5.83 5.82 5.41 5.40 5.38 5.38 4.64 4.63 2.51 2.51 2.50 2.49 2.00 2.00 1.99 1.99 1.98 1.98 1.97 1.96 1.75 1.74 1.73 1.68 1.67 1.67 1.66 1.54 1.53 1.52 1.51 1.51 1.51 1.50 1.50 1.49 0.00
OH
N
3k
2.00 1.00 1.14 0.99 1.01 1.00 0.87 1.20 2.11 2.23 2.26
f1 (ppm)

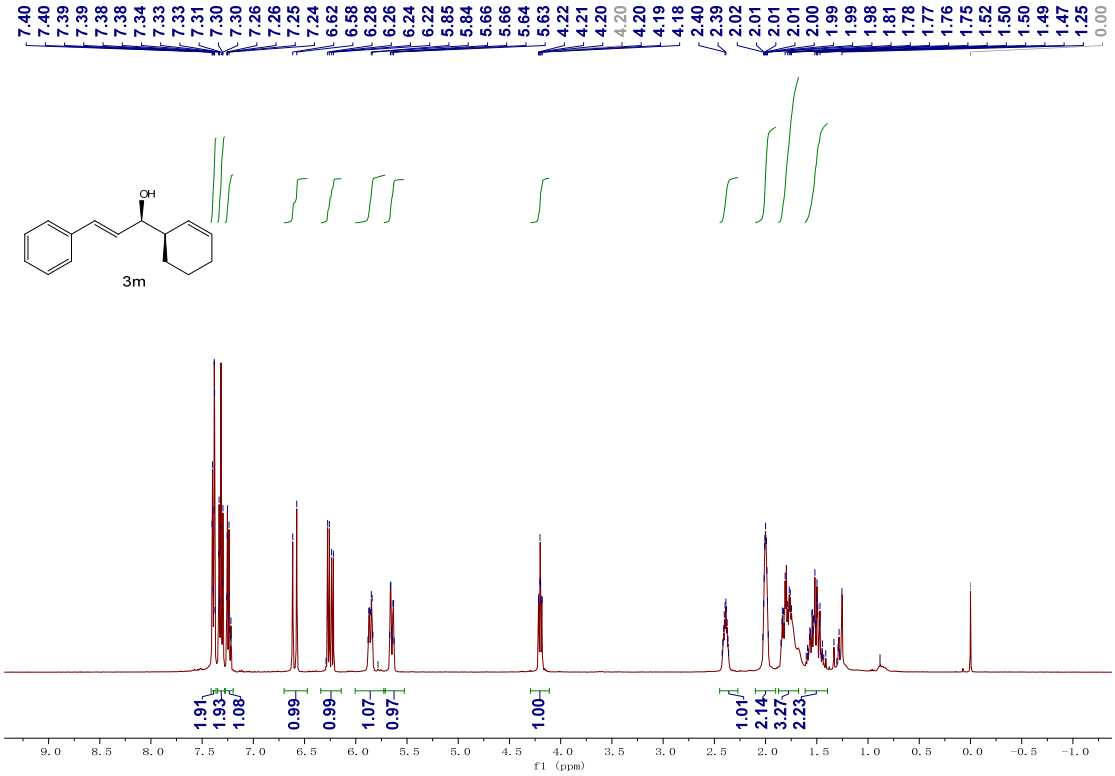

148.33 148.11 138.46 134.37 130.97 127.19 123.24
77.32 CDCl3 77.00 CDCl3 76.68 CDCl3 74.79
42.88
25.12 23.61 20.91
OH
N
3k
f1 (ppm)

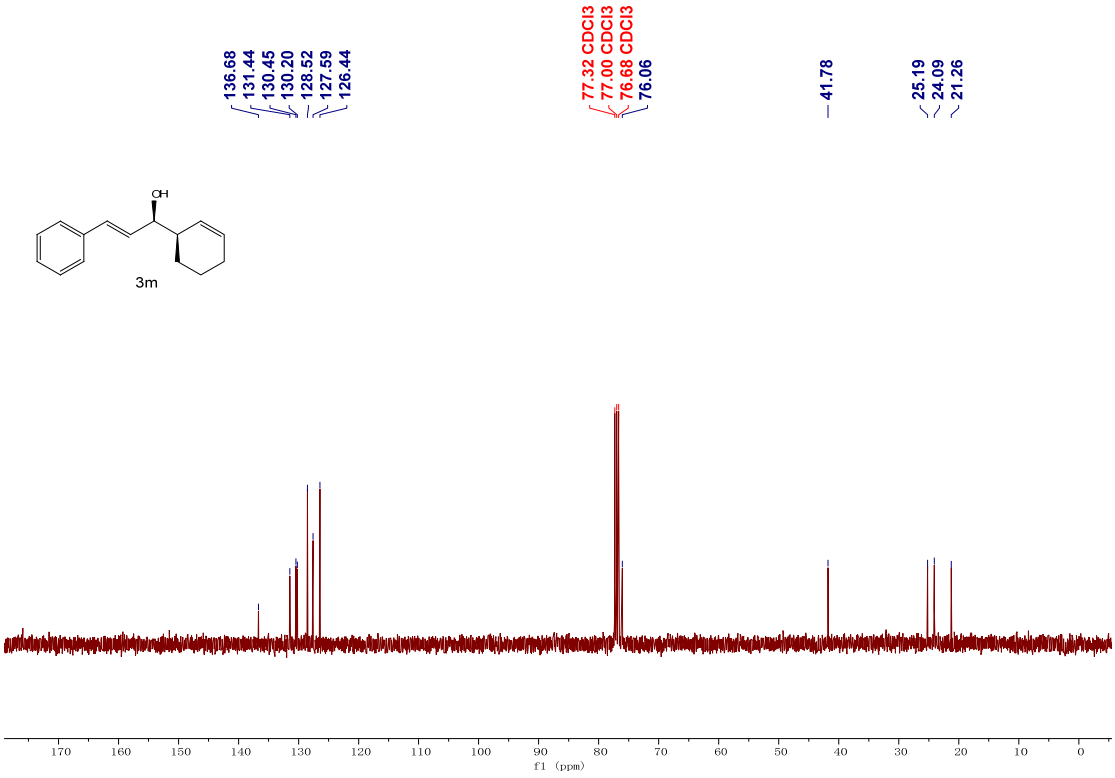

7.26 CDCl3
5.74
5.73
5.71
5.71
5.44
5.44
5.44
5.42
5.41
5.41
4.27
4.27
4.26
4.26
4.21
4.18
4.18
4.18
4.17
4.17
4.17
4.16
4.16
4.15
2.14
2.13
1.96
1.95
1.95
1.94
1.94
1.93
1.93
1.92
1.92
1.76
1.76
1.76
1.75
1.74
1.73
1.71
1.51
1.50
1.49
1.49
1.49
1.48
1.47
1.47
1.46
1.46
0.00
OH
Fc
1.00
0.99
1.01
5.31
3.48
1.00
0.99
2.02
2.48
2.03
f1 (ppm)

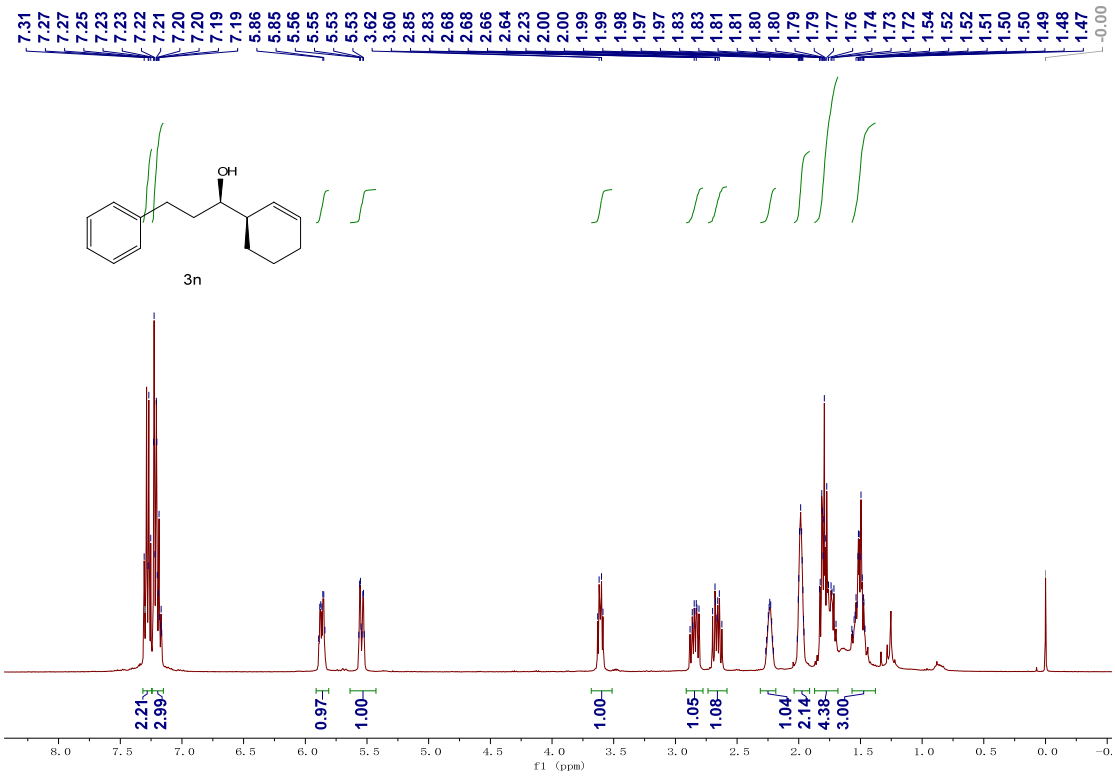
3l

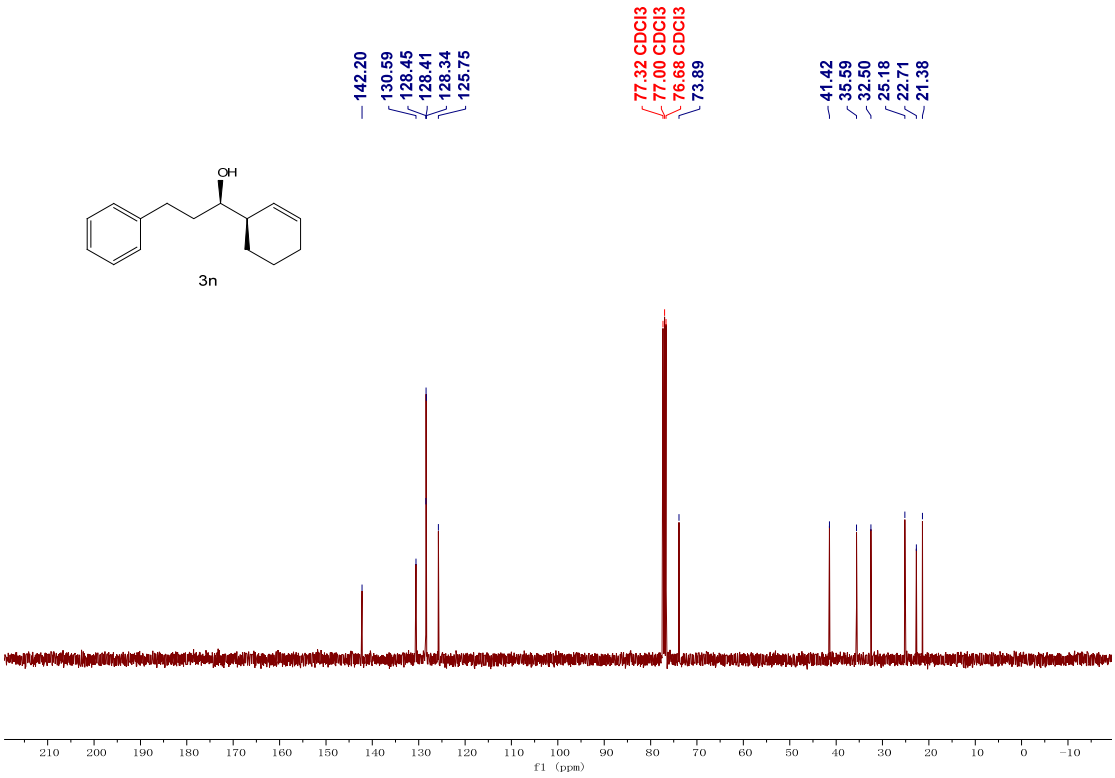

129.12
128.09
92.95
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
72.98
68.69
68.20
67.84
67.50
64.85
42.61
25.18
24.83
21.09
OH
Fc
f1 (ppm)

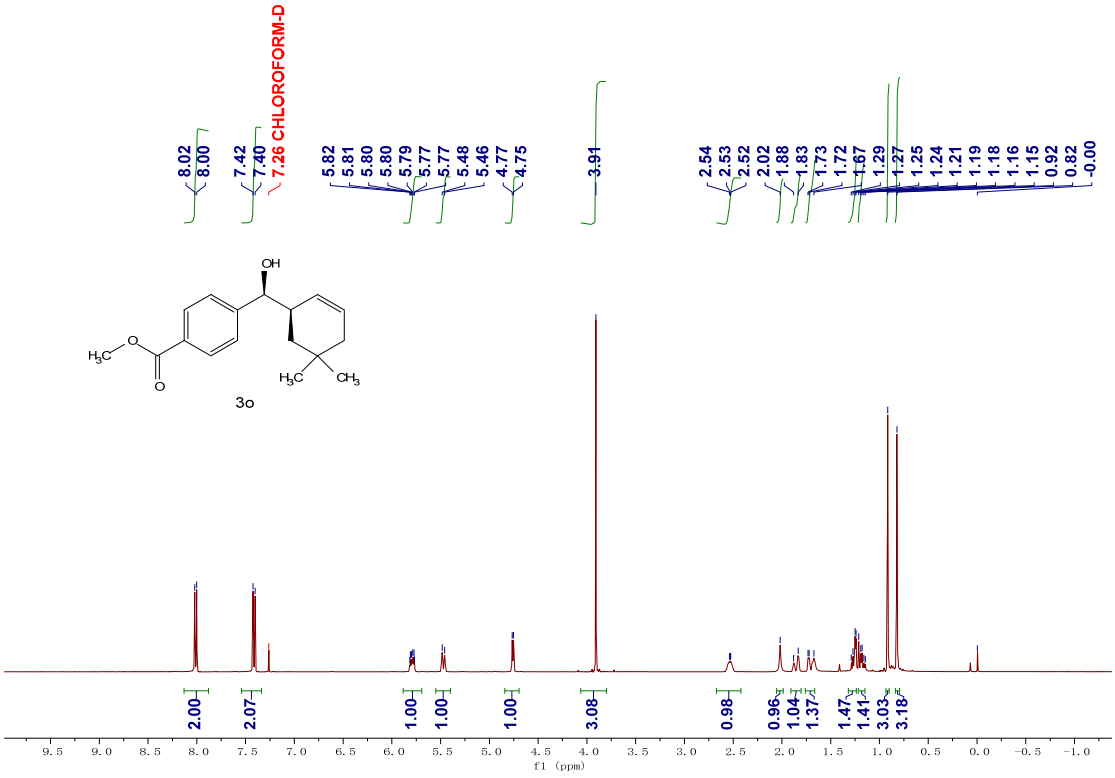
3l

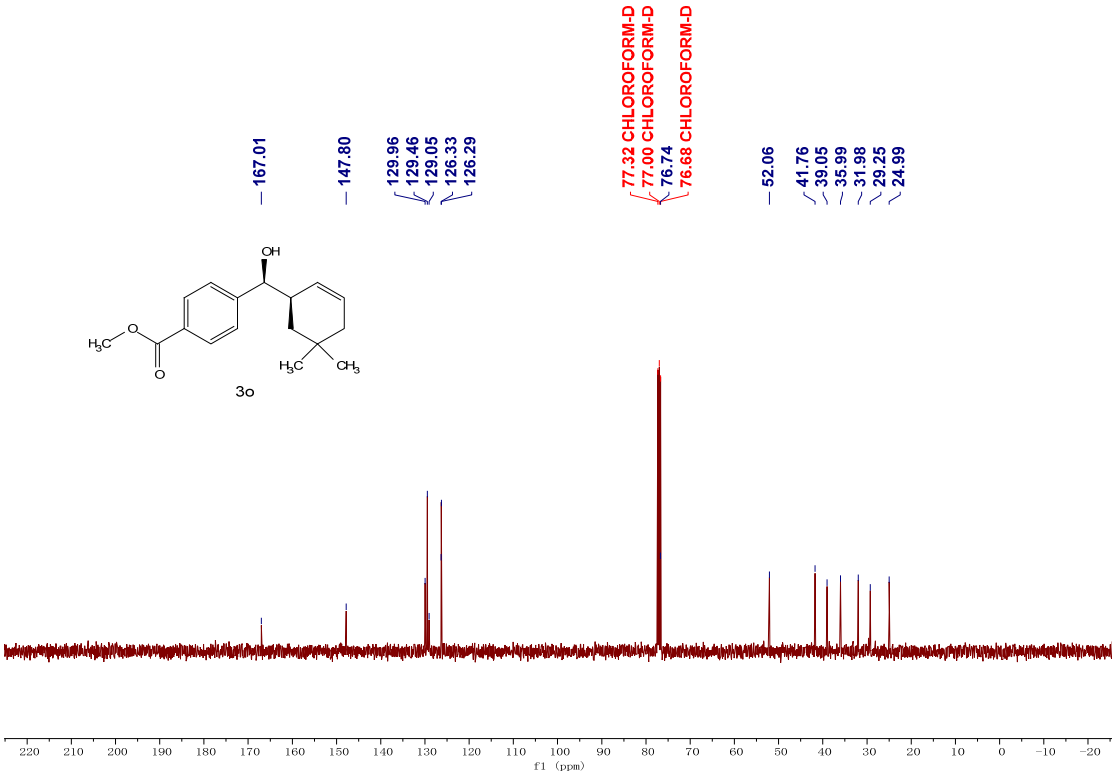

7.40 7.40 7.39 7.39 7.38 7.38 7.34 7.33 7.33 7.31 7.30 7.30 7.26 7.26 7.25 7.24 6.62 6.58 6.28 6.26 6.24 6.22 5.85 5.84 5.66 5.66 5.64 5.63 4.22 4.21 4.20 4.20 4.19 4.18 2.40 2.39 2.02 2.01 2.01 2.01 2.00 1.99 1.99 1.98 1.81 1.78 1.77 1.76 1.75 1.52 1.50 1.50 1.49 1.47 1.25 0.00
OH
3m
1.91 1.93 1.08 0.99 0.99 1.07 0.97 1.00 1.01 2.14 3.27 2.23
f1 (ppm)

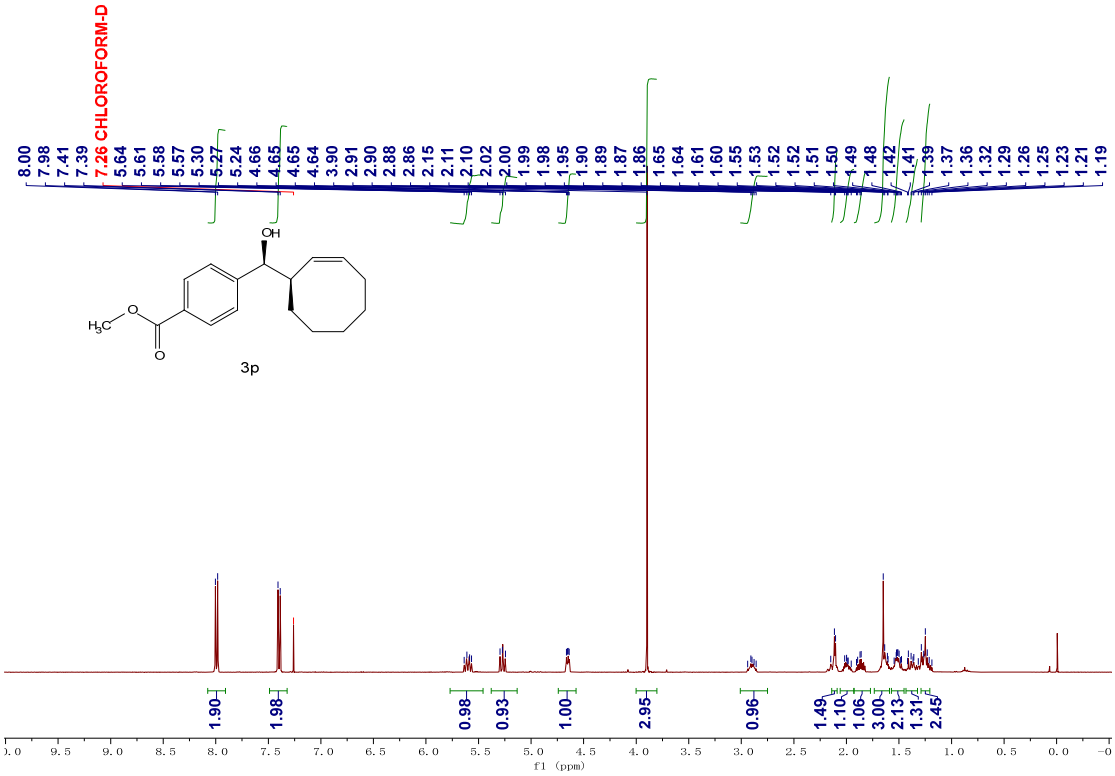

136.68 131.44 130.45 130.20 128.52 127.59 126.44
77.32 CDCl3 77.00 CDCl3 76.68 CDCl3 76.06
41.78
25.19 24.09 21.26
OH
3m
f1 (ppm)

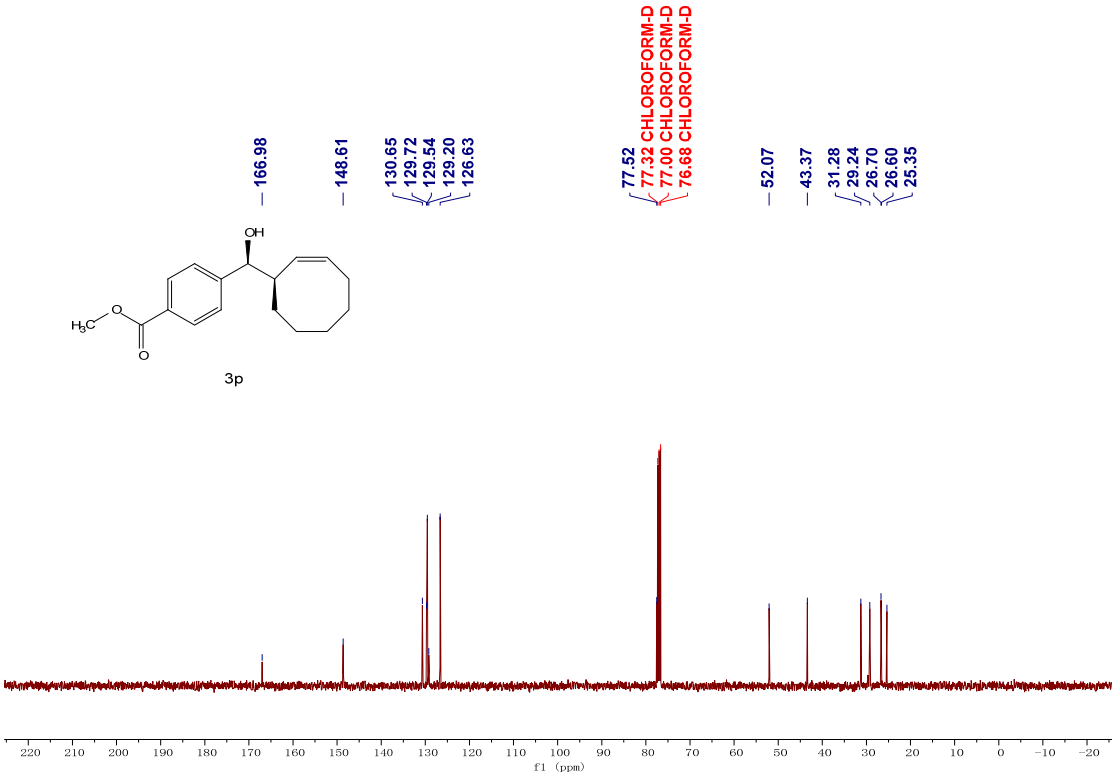

7.31
7.27
7.27
7.25
7.23
7.23
7.22
7.21
7.20
7.20
7.19
7.19
5.86
5.85
5.56
5.55
5.53
5.53
3.62
3.60
2.85
2.83
2.68
2.68
2.66
2.64
2.23
2.00
2.00
1.99
1.99
1.98
1.97
1.97
1.83
1.83
1.81
1.81
1.80
1.80
1.79
1.79
1.77
1.76
1.74
1.73
1.72
1.54
1.52
1.52
1.51
1.50
1.50
1.49
1.48
1.47
-0.00
OH
3n
2.21
2.99
0.97
1.00
1.00
1.05
1.08
1.04
2.14
4.38
3.00
f1 (ppm)

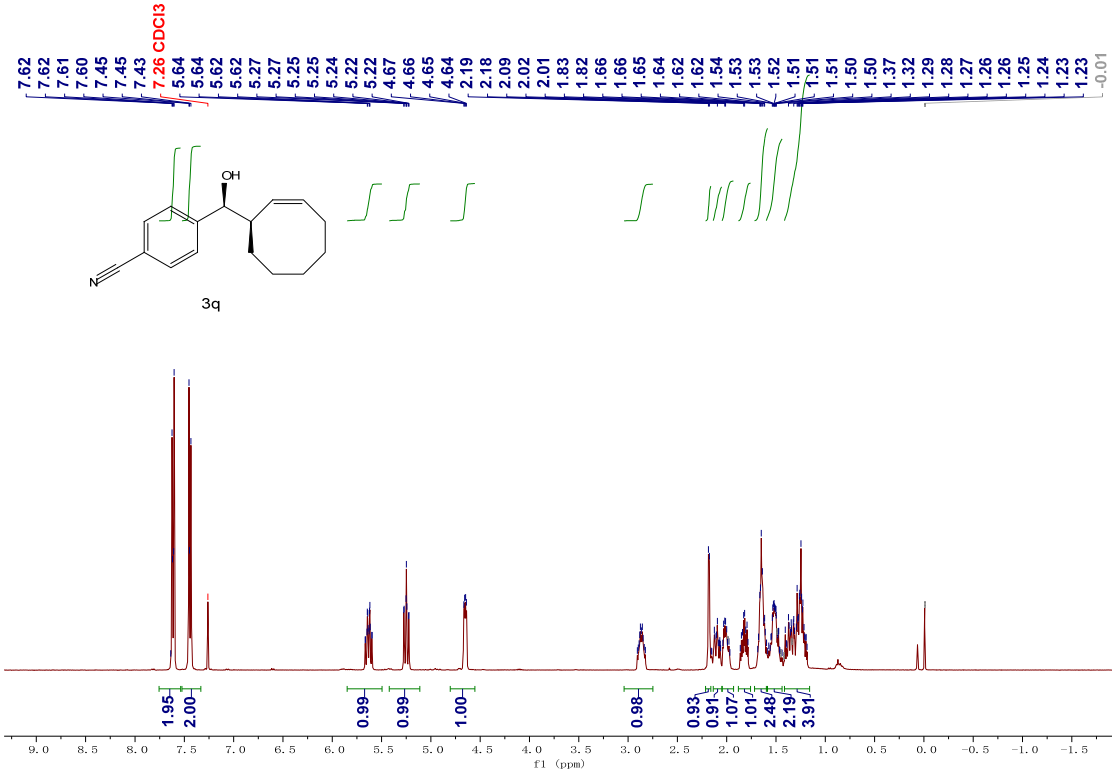

142.20
130.59
128.45
128.41
128.34
125.75
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
73.89
41.42
35.59
32.50
25.18
22.71
21.38
OH
3n
f1 (ppm)

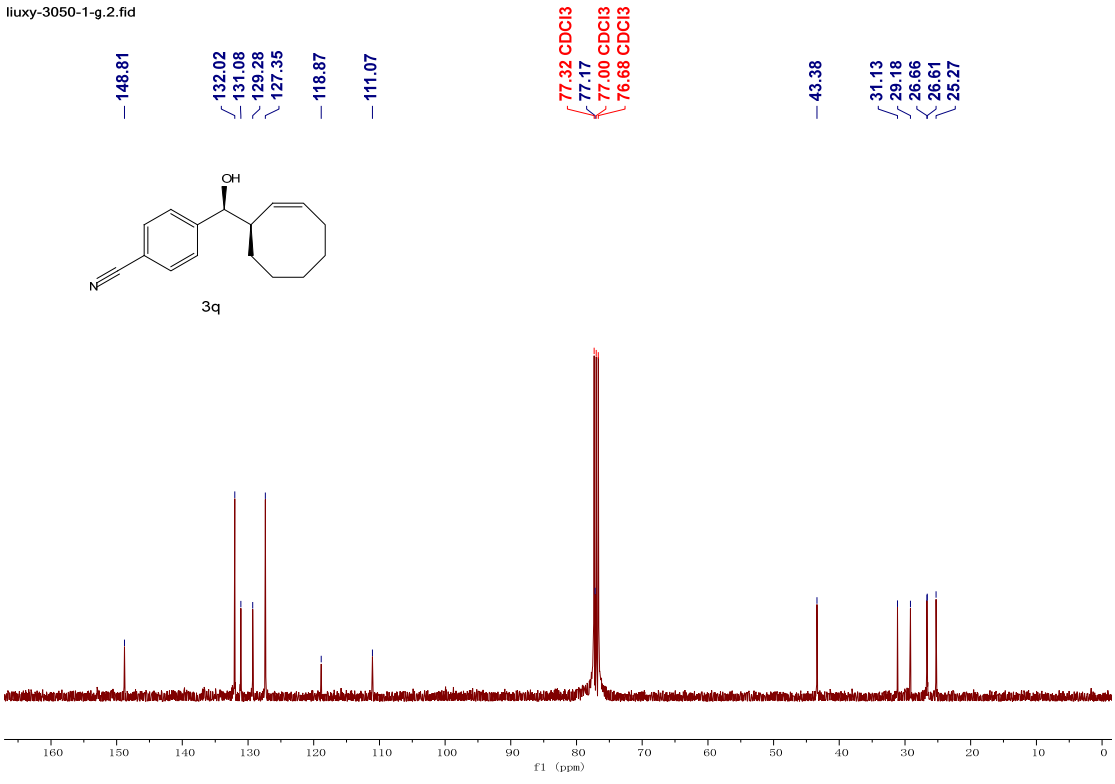

8.02
8.00
7.42
7.40
7.26 CHLOROFORM-D
5.82
5.81
5.80
5.80
5.79
5.77
5.77
5.48
5.46
4.77
4.75
3.91
2.54
2.53
2.52
2.02
1.88
1.83
1.73
1.72
1.67
1.29
1.27
1.25
1.24
1.21
1.19
1.18
1.16
1.15
0.92
0.82
-0.00
OH
O
H3C
O
H3C
CH3
3o
2.00
2.07
1.00
1.00
1.00
3.08
0.98
0.96
1.04
1.37
1.47
1.41
3.03
3.18
f1 (ppm)

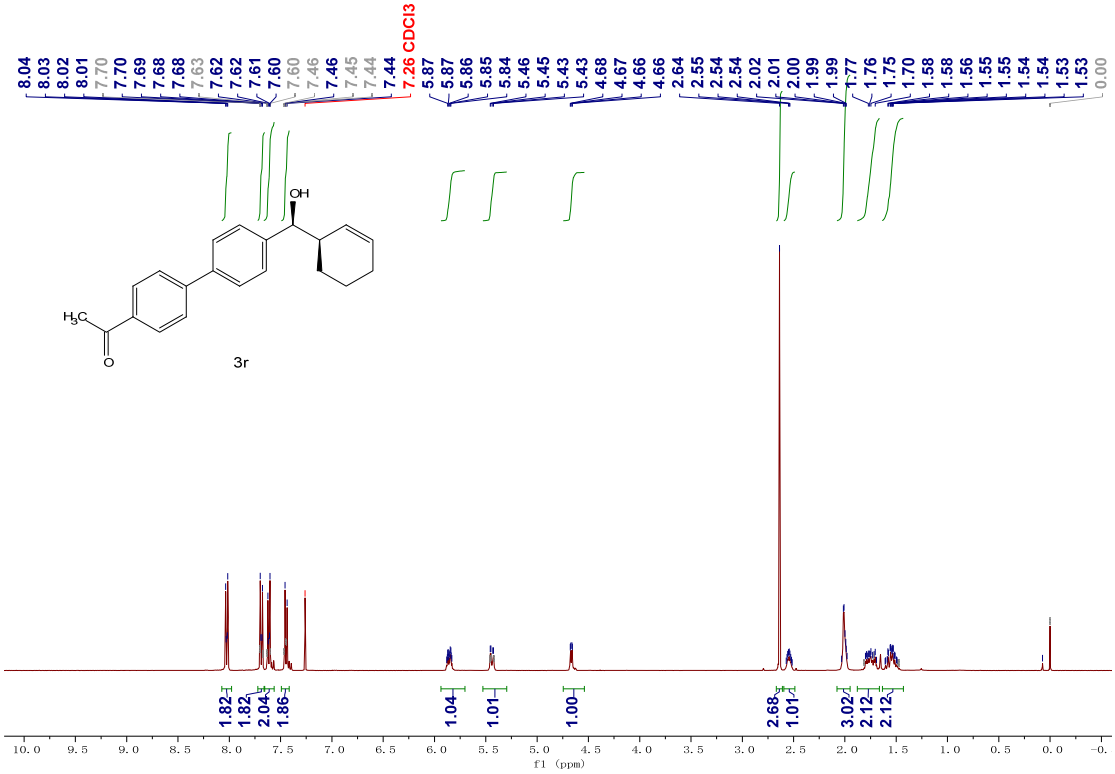

167.01
147.80
129.96
129.46
129.05
126.33
126.29
77.32 CHLOROFORM-D
77.00 CHLOROFORM-D
76.74
76.68 CHLOROFORM-D
52.06
41.76
39.05
35.99
31.98
29.25
24.99
OH
O
H3C
O
H3C
CH3
3o
f1 (ppm)


7.26 CHLOROFORM-D
8.00 7.98 7.41 7.39 5.64 5.61 5.58 5.57 5.30 5.27 5.24 4.66 4.65 4.64 3.90 2.91 2.90 2.88 2.86 2.15 2.11 2.10 2.02 2.00 1.99 1.98 1.95 1.90 1.89 1.87 1.86 1.65 1.64 1.61 1.60 1.55 1.53 1.52 1.52 1.51 1.50 1.49 1.48 1.42 1.41 1.39 1.37 1.36 1.32 1.29 1.26 1.25 1.23 1.21 1.19
OH
O
H3C
O
3p
1.90 1.98 0.98 0.93 1.00 2.95 0.96 1.49 1.10 1.06 3.00 2.13 1.31 2.45
f1 (ppm)


166.98 148.61 130.65 129.72 129.54 129.20 126.63
77.52 77.32 CHLOROFORM-D 77.00 CHLOROFORM-D 76.68 CHLOROFORM-D
52.07 43.37 31.28 29.24 26.70 26.60 25.35
OH
O
H3C
O
3p
f1 (ppm)


7.62 7.62 7.61 7.60 7.45 7.45 7.43 7.26 CDCl3 5.64 5.64 5.62 5.62 5.27 5.25 5.25 5.24 5.22 5.22 4.67 4.66 4.65 4.64 2.19 2.18 2.09 2.02 2.01 1.83 1.82 1.66 1.66 1.65 1.64 1.62 1.62 1.54 1.53 1.53 1.52 1.51 1.51 1.51 1.50 1.50 1.37 1.32 1.29 1.28 1.27 1.26 1.26 1.25 1.24 1.23 1.23 -0.01
OH
N
3q
1.95 2.00 0.99 0.99 1.00 0.98 0.93 0.91 1.07 1.01 2.48 2.19 3.91
f1 (ppm)


liuxy-3050-1-g.2.fid
148.81 132.02 131.08 129.28 127.35 118.87 111.07 77.32 CDCl3 77.17 77.00 CDCl3 76.68 CDCl3 43.38 31.13 29.18 26.66 26.61 25.27
OH
N
3q
f1 (ppm)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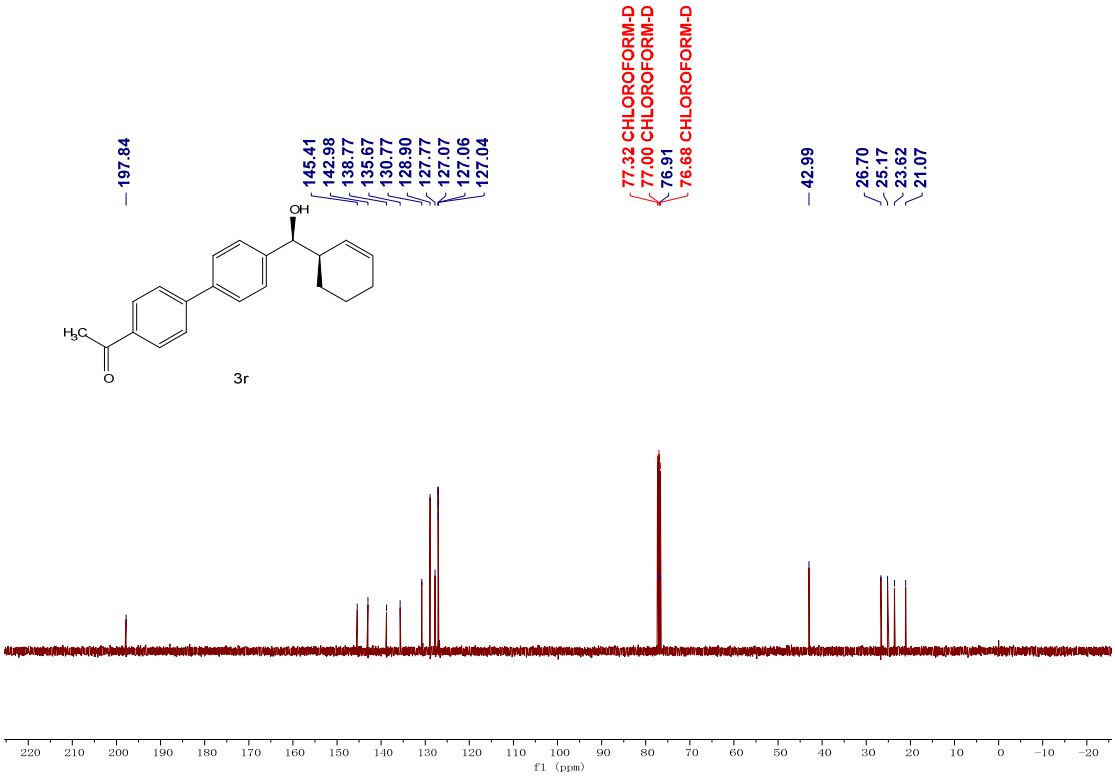
5.87 5.87 5.86 5.85 5.84 5.46 5.45 5.43 5.43 4.68 4.67 4.66 4.66
2.64 2.55 2.54 2.54 2.54 2.02 2.01 2.00 1.99 1.99 1.77 1.76 1.75 1.70 1.58 1.58 1.56 1.55 1.55 1.54 1.54 1.53 1.53 0.00
OH
H3C
O
3r
1.82 1.82 2.04 1.86 1.04 1.01 1.00 2.68 1.01 3.02 2.12 2.12
f1 (ppm)


197.84
145.41 142.98 138.77 135.67 130.77 128.90 127.77 127.07 127.06 127.04
77.32 CHLOROFORM-D
77.00 CHLOROFORM-D
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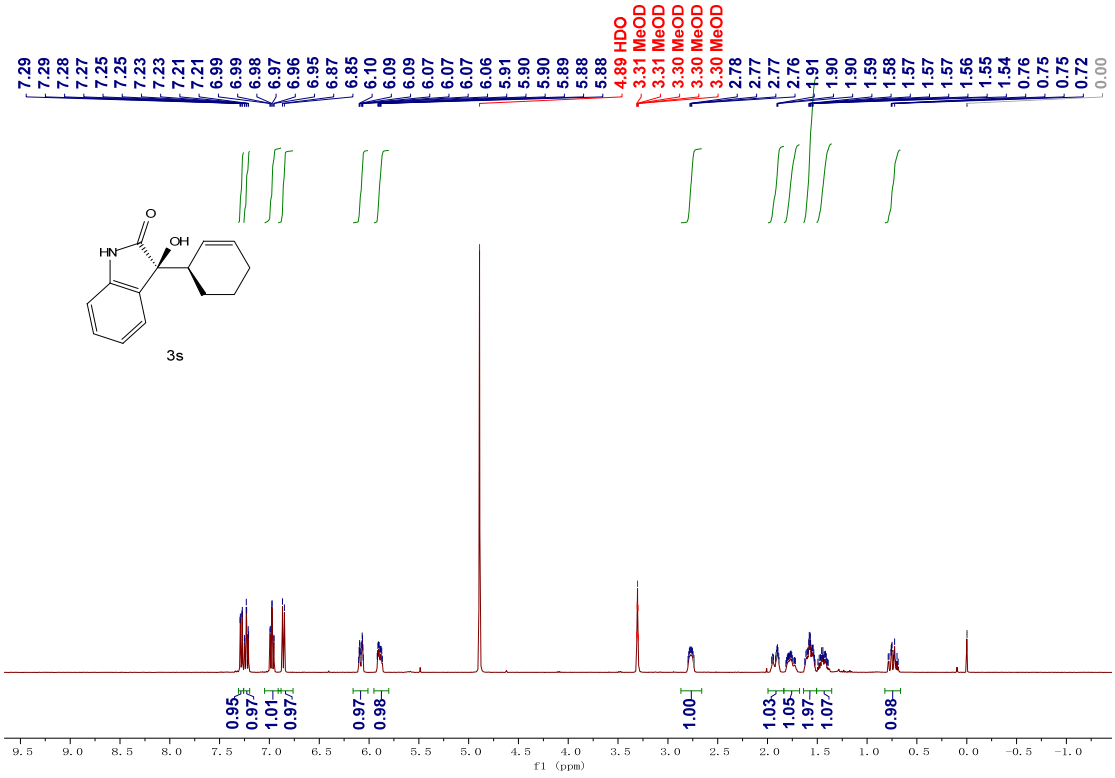
76.68 CHLOROFORM-D
42.99
26.70 25.17 23.62 21.07
OH
H3C
O
3r
f1 (ppm)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7.28
7.27
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7.23
7.23
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7.21
6.99
6.99
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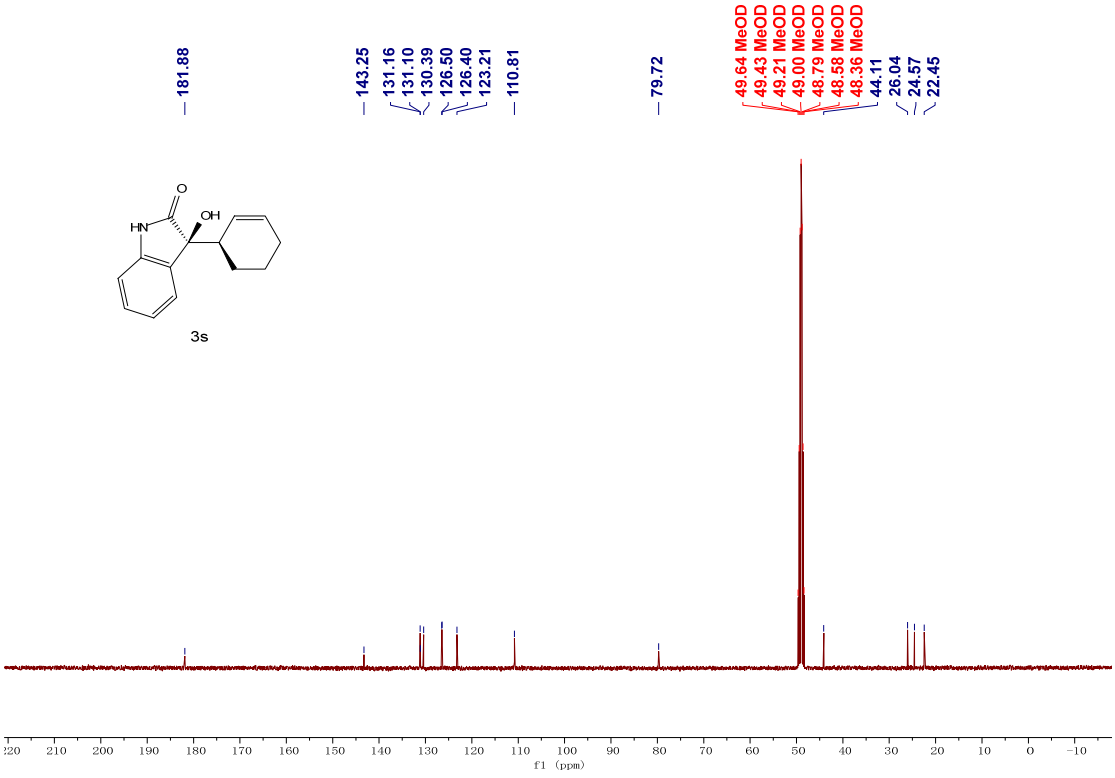
6.97
6.96
6.95
6.87
6.85
6.10
6.09
6.09
6.07
6.07
6.07
6.06
5.91
5.90
5.90
5.89
5.88
5.88
4.89 HDO
3.31 MeOD
3.31 MeOD
3.30 MeOD
3.30 MeOD
3.30 MeOD
2.78
2.77
2.77
2.76
1.91
1.90
1.90
1.59
1.58
1.57
1.57
1.57
1.56
1.55
1.54
0.76
0.75
0.75
0.72
0.00
O
HN
OH
3s
0.95
0.97
1.01
0.97
0.97
0.98
1.00
1.03
1.05
1.97
1.07
0.98
f1 (ppm)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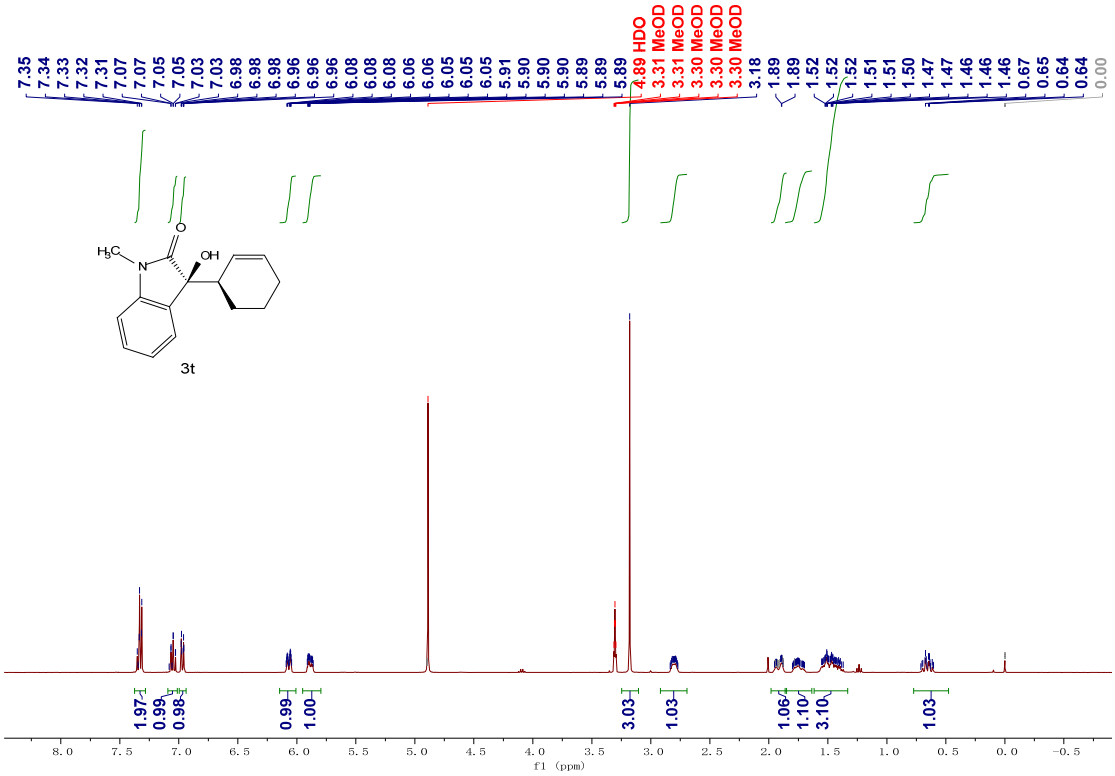
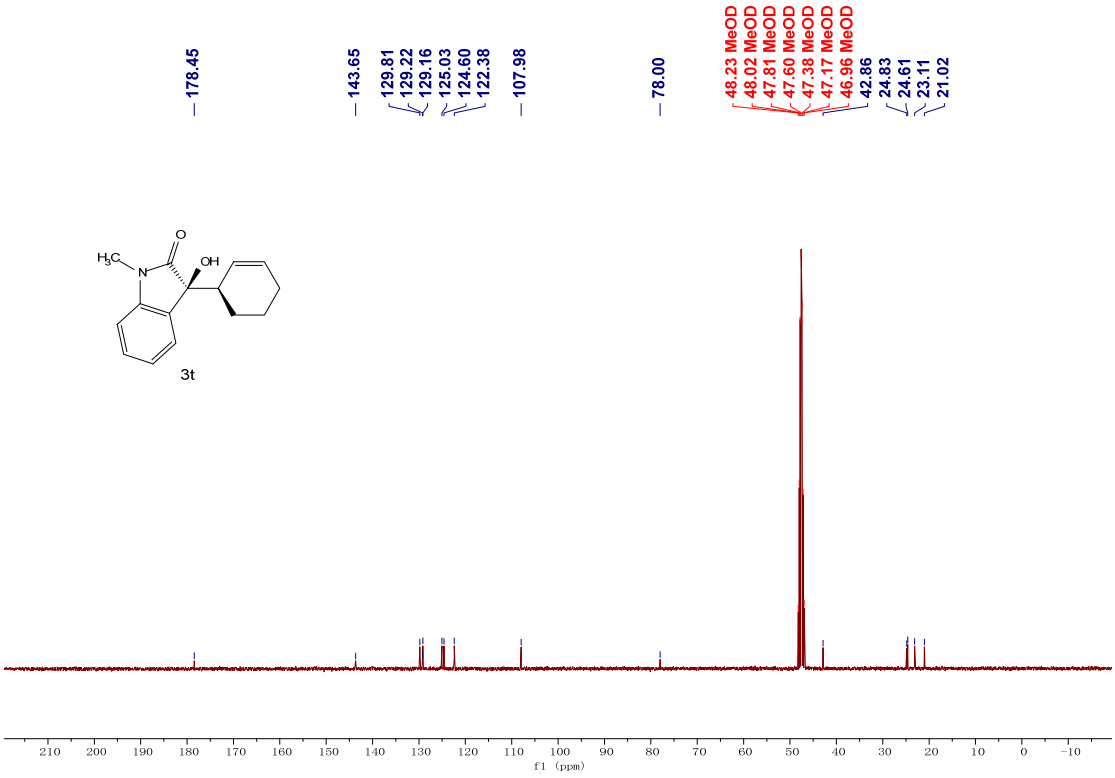
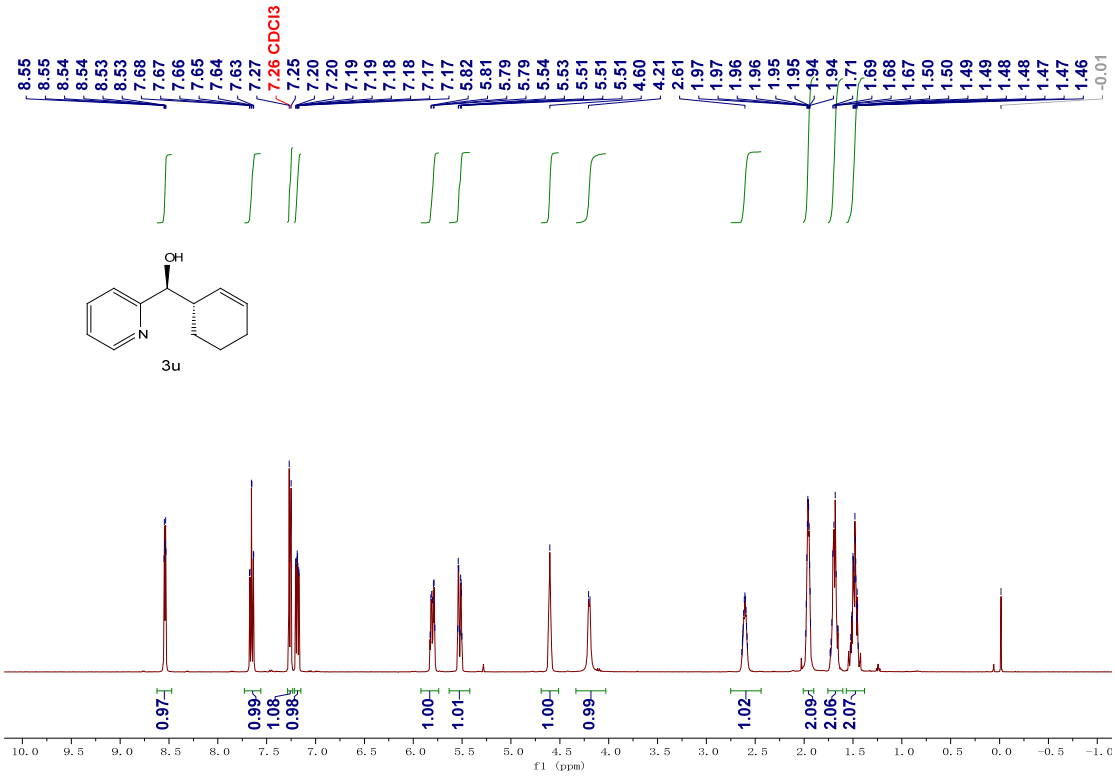
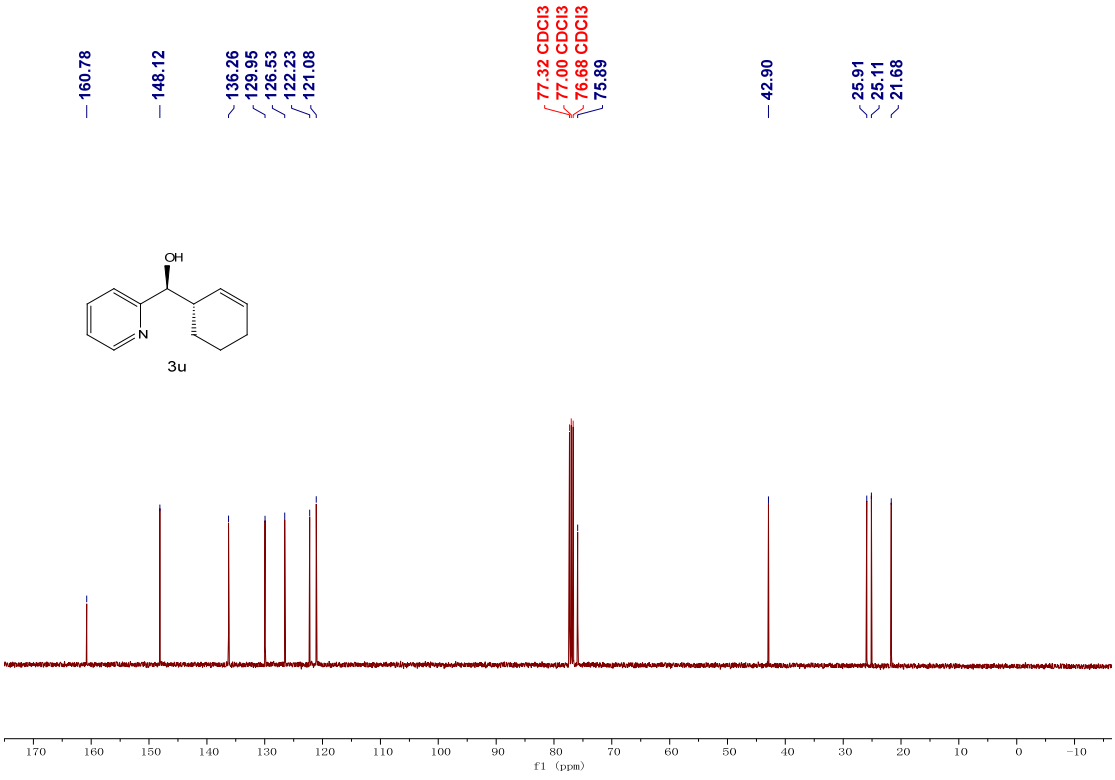
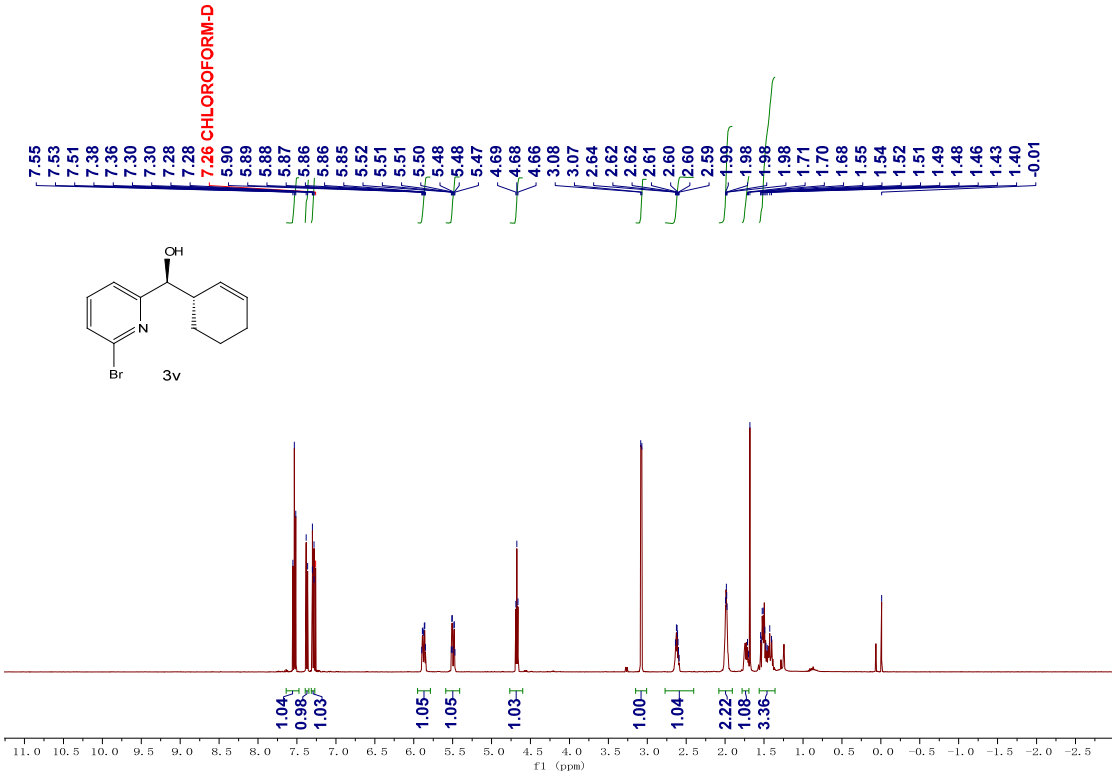
143.25
131.16
131.10
130.39
126.50
126.40
123.21
110.81
79.72
49.64 MeOD
49.43 MeOD
49.21 MeOD
49.00 MeOD
48.79 MeOD
48.58 MeOD
48.36 MeOD
44.11
26.04
24.57
22.45
O
HN
OH
3s
f1 (ppm)


7.35 7.34 7.33 7.32 7.31 7.07 7.07 7.05 7.05 7.03 7.03 6.98 6.98 6.96 6.96 6.96 6.08 6.08 6.08 6.06 6.06 6.05 6.05 6.05 5.91 5.90 5.90 5.89 5.89 5.89 4.89 HDO 3.31 MeOD 3.31 MeOD 3.30 MeOD 3.30 MeOD 3.18 1.89 1.89 1.52 1.52 1.52 1.51 1.51 1.50 1.47 1.47 1.46 1.46 1.46 0.67 0.65 0.64 0.64 0.00
3t
1.97 0.99 0.98 0.99 1.00 3.03 1.03 1.06 1.10 3.10 1.03
f1 (ppm)


178.45 143.65 129.81 129.22 129.16 125.03 124.60 122.38 107.98 78.00 48.23 MeOD 48.02 MeOD 47.81 MeOD 47.60 MeOD 47.38 MeOD 47.17 MeOD 46.96 MeOD 42.86 24.83 24.61 23.11 21.02
3t
f1 (ppm)


8.55 8.55 8.54 8.54 8.53 8.53 7.68 7.67 7.66 7.65 7.64 7.63 7.27 7.26 CDCl3 7.25 7.20 7.20 7.19 7.19 7.18 7.18 7.17 7.17 5.82 5.81 5.79 5.79 5.54 5.53 5.51 5.51 5.51 4.60 4.21 2.61 1.97 1.97 1.96 1.96 1.95 1.95 1.94 1.94 1.71 1.69 1.68 1.67 1.50 1.50 1.49 1.49 1.48 1.48 1.47 1.47 1.46 -0.01
OH
N
3u
0.97 0.99 1.08 0.98 1.00 1.01 1.00 0.99 1.02 2.09 2.06 2.07
f1 (ppm)


160.78 148.12 136.26 129.95 126.53 122.23 121.08 77.32 CDCl3 77.00 CDCl3 76.68 CDCl3 75.89 42.90 25.91 25.11 21.68
OH
N
3u
f1 (ppm)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7.53
7.51
7.38
7.36
7.30
7.30
7.28
7.28
7.26 CHLOROFORM-D
5.90
5.89
5.88
5.87
5.86
5.86
5.85
5.52
5.51
5.51
5.50
5.48
5.48
5.47
4.69
4.68
4.66
3.08
3.07
2.64
2.62
2.62
2.61
2.60
2.60
2.59
1.99
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1.98
1.98
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1.54
1.52
1.51
1.49
1.48
1.46
1.43
1.40
-0.01
OH
N
Br
3v
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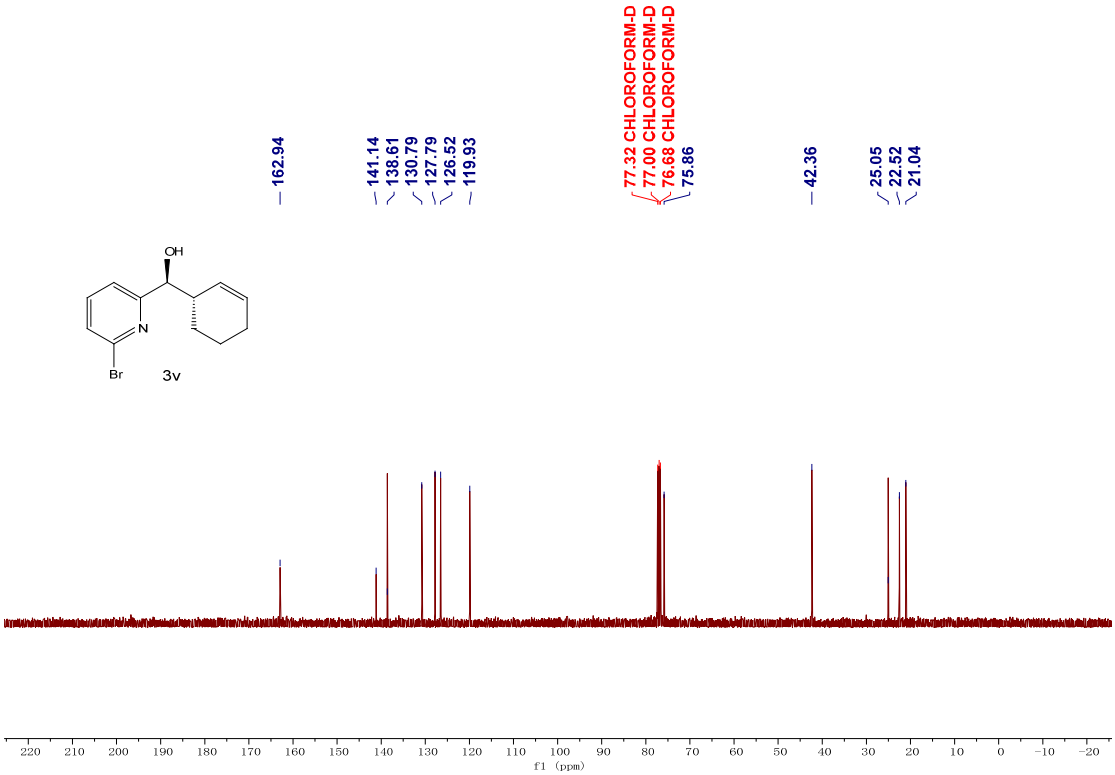
0.98
1.03
1.05
1.05
1.03
1.00
1.04
2.22
1.08
3.36
f1 (ppm)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141.14
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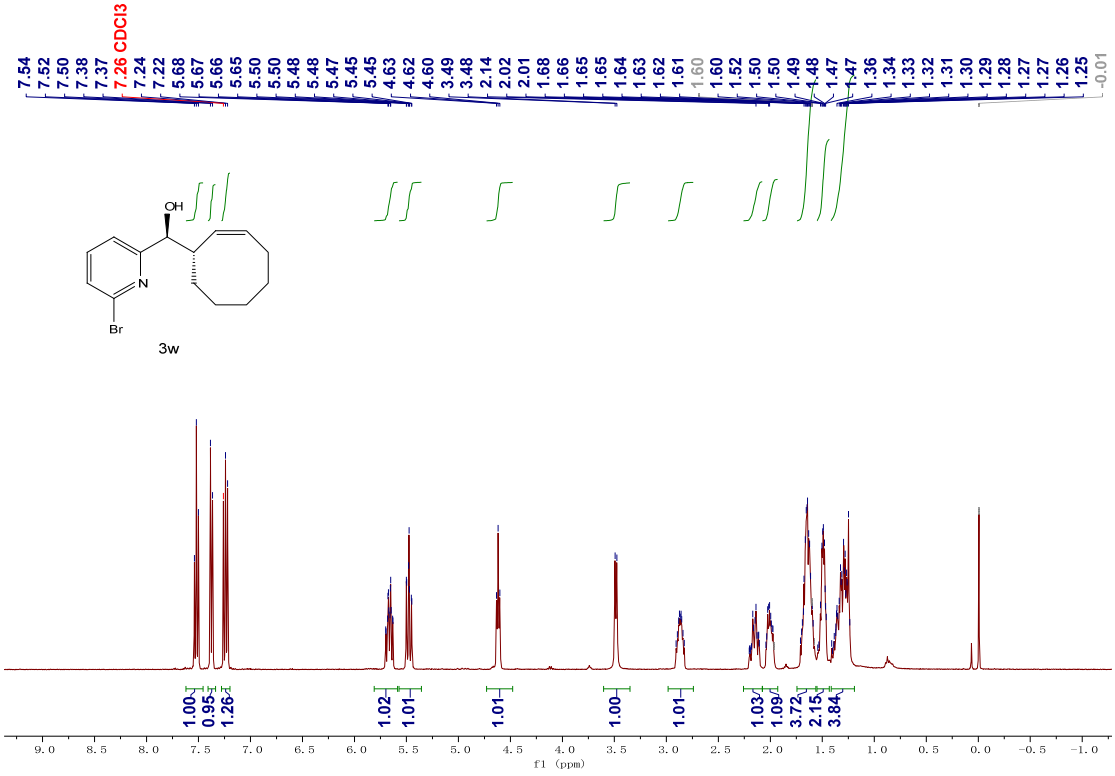
130.79
127.79
126.52
119.93
77.32 CHLOROFORM-D
77.00 CHLOROFORM-D
76.68 CHLOROFORM-D
75.86
42.36
25.05
22.52
21.04
OH
N
Br
3v
f1 (ppm)


7.54
7.52
7.50
7.38
7.37
7.26 CDCl3
7.24
7.22
5.68
5.67
5.66
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5.50
5.50
5.48
5.48
5.47
5.45
5.45
4.63
4.62
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1.33
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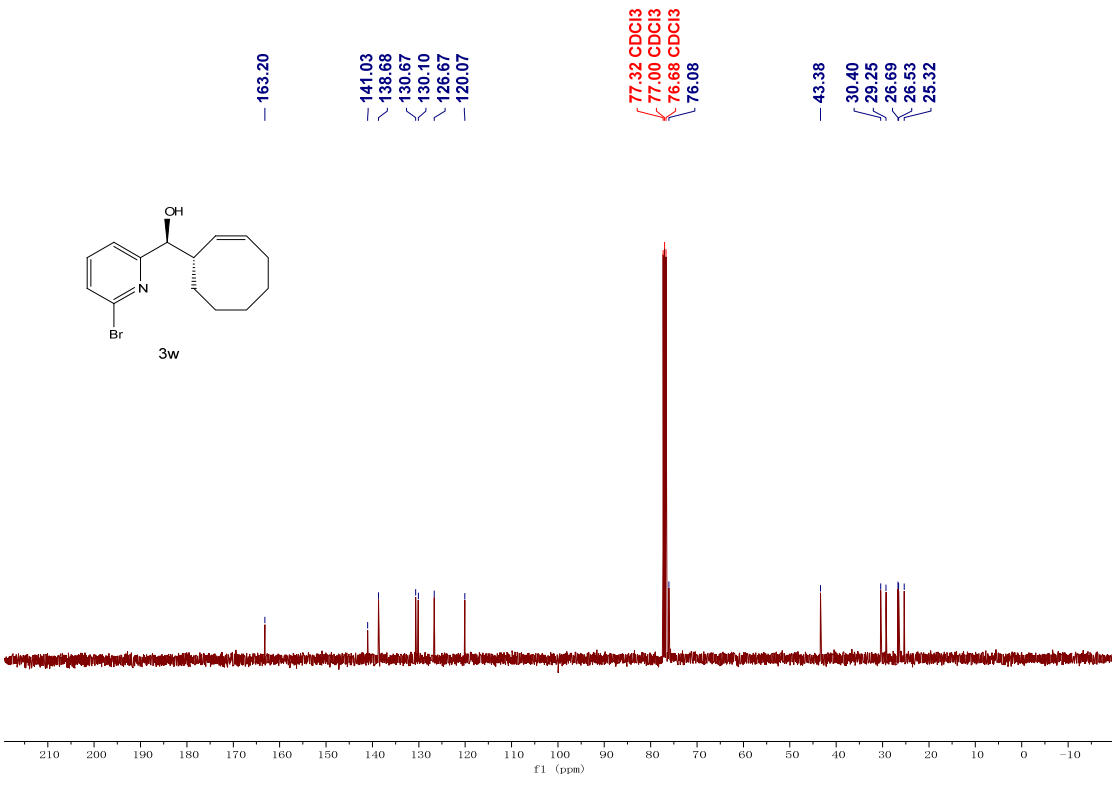
1.31
1.30
1.29
1.28
1.27
1.27
1.26
1.25
-0.01
OH
N
Br
3w
1.00
0.95
1.26
1.02
1.01
1.01
1.00
1.01
1.03
1.09
3.72
2.15
3.84
f1 (ppm)


163.20
141.03
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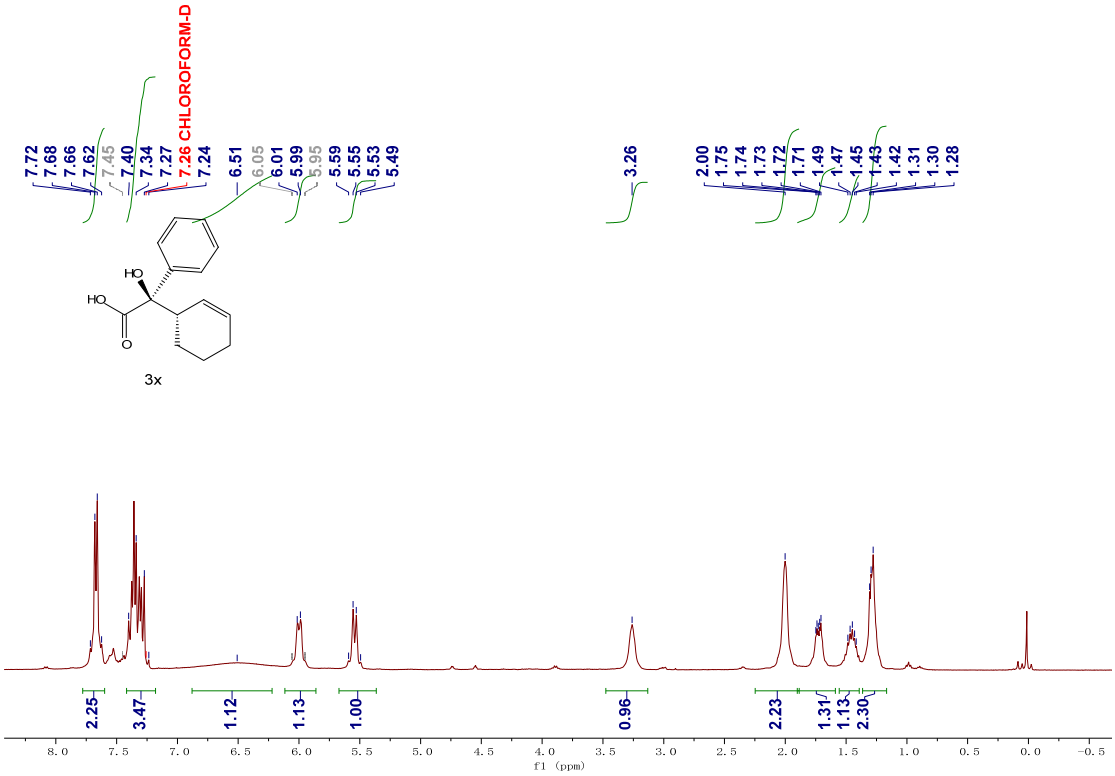
130.10
126.67
120.07
77.32 CDCl3
77.00 CDCl3
76.68 CDCl3
76.08
43.38
30.40
29.25
26.69
26.53
25.32
OH
N
Br
3w
f1 (ppm)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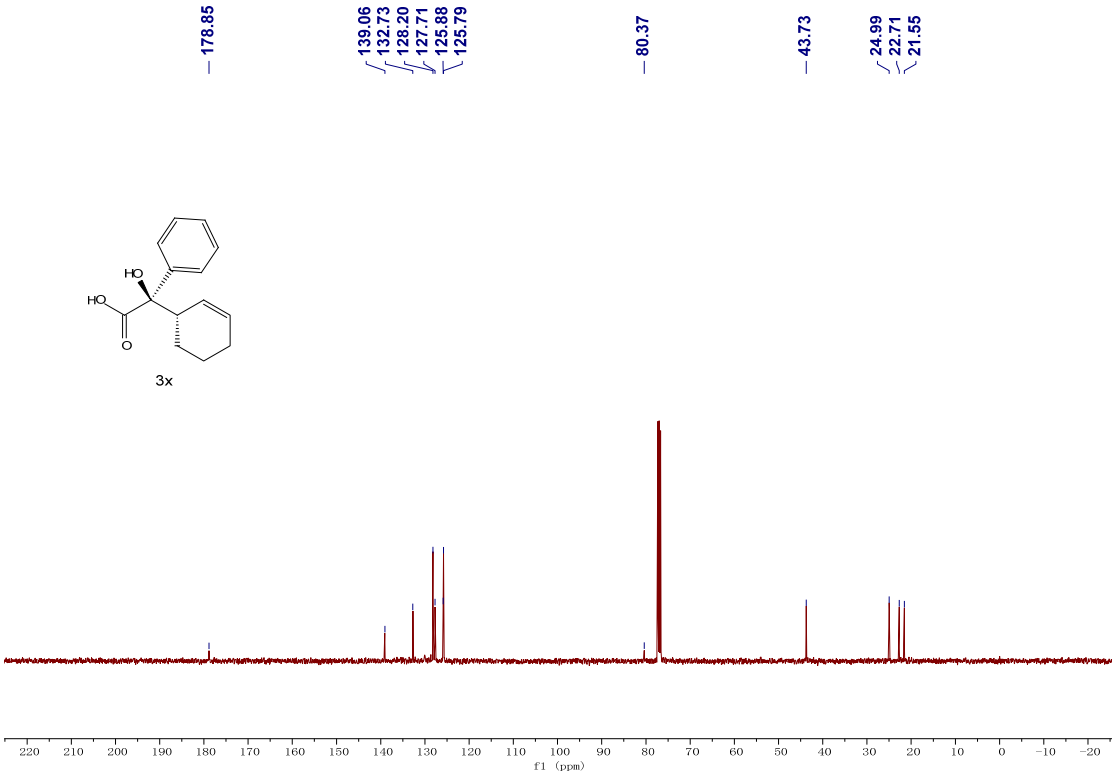
7.40
7.34
7.27
7.26 CHLOROFORM-D
7.24
6.51
6.05
6.01
5.99
5.95
5.59
5.55
5.53
5.49
3.26
2.00
1.75
1.74
1.73
1.72
1.71
1.49
1.47
1.45
1.43
1.42
1.31
1.30
1.28
HO
HO
O
3x
2.25
3.47
1.12
1.13
1.00
0.96
2.23
1.31
1.13
2.30
f1 (ppm)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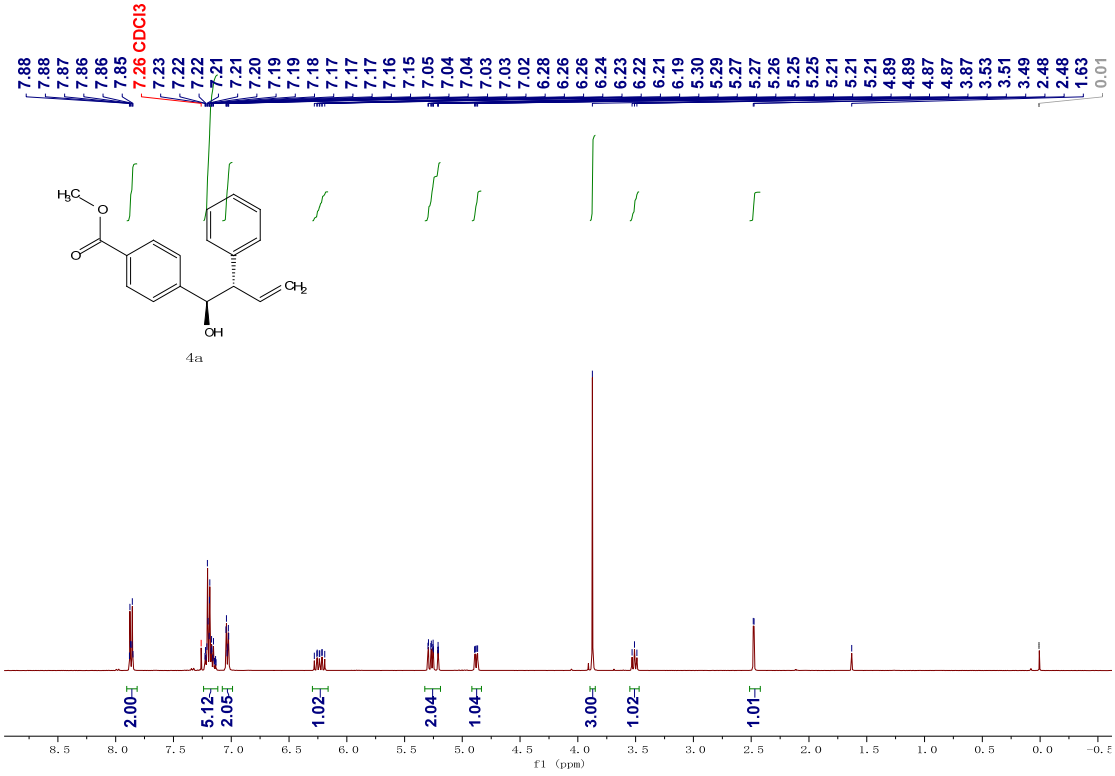
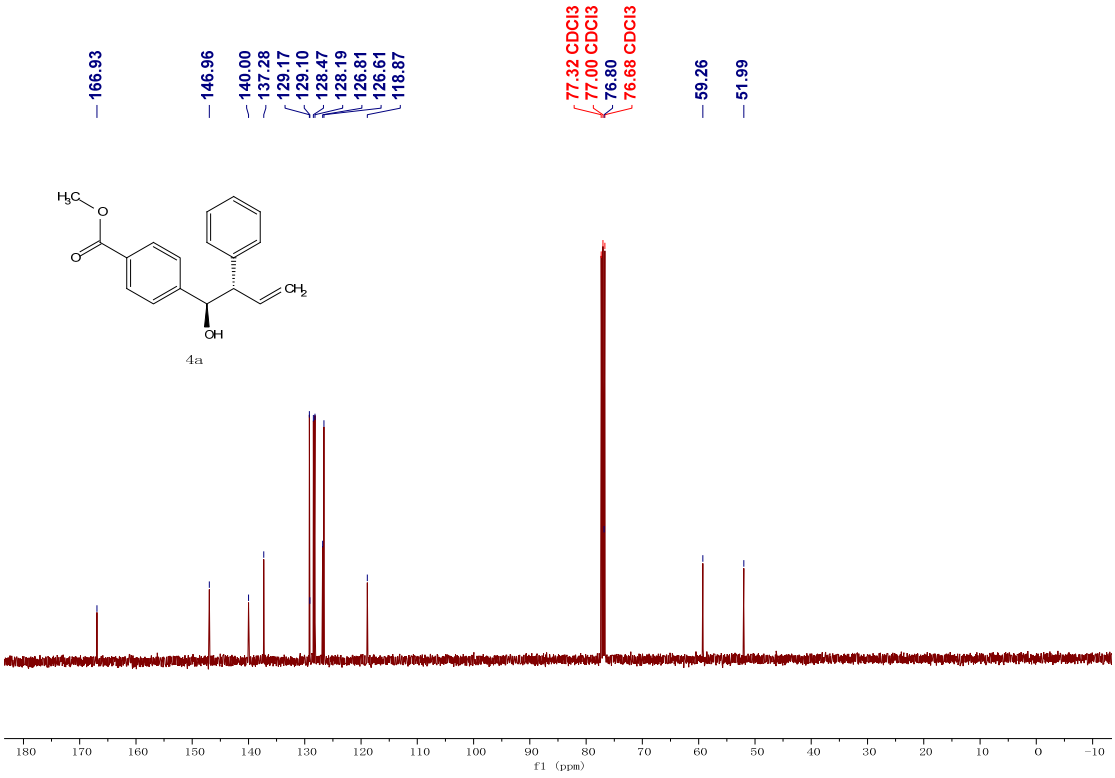
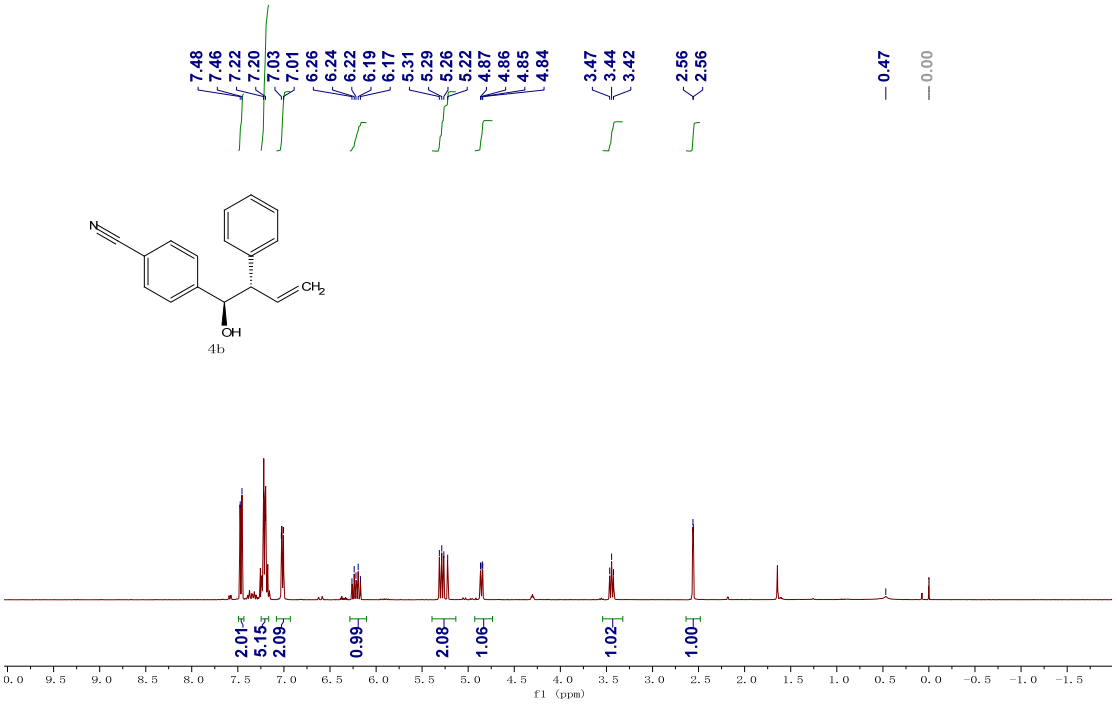
80.37
43.73
24.99
22.71
21.55
HO
HO
O
3x
f1 (ppm)


7.88 7.88 7.87 7.86 7.86 7.85 7.26 CDCl3 7.23 7.22 7.22 7.21 7.21 7.20 7.19 7.19 7.18 7.17 7.17 7.17 7.16 7.15 7.05 7.04 7.04 7.03 7.03 7.02 6.28 6.26 6.26 6.24 6.23 6.22 6.21 6.19 5.30 5.29 5.27 5.27 5.26 5.25 5.25 5.21 5.21 5.21 4.89 4.89 4.87 4.87 3.87 3.53 3.51 3.49 2.48 2.48 1.63 0.01
H3C
O
O
OH
CH2
4a
2.00 5.12 2.05 1.02 2.04 1.04 3.00 1.02 1.01
f1 (ppm)


166.93 146.96 140.00 137.28 129.17 129.10 128.47 128.19 126.81 126.61 118.87
77.32 CDCl3 77.00 CDCl3 76.80 76.68 CDCl3
59.26 51.99
H3C
O
O
OH
CH2
4a
f1 (ppm)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7.01
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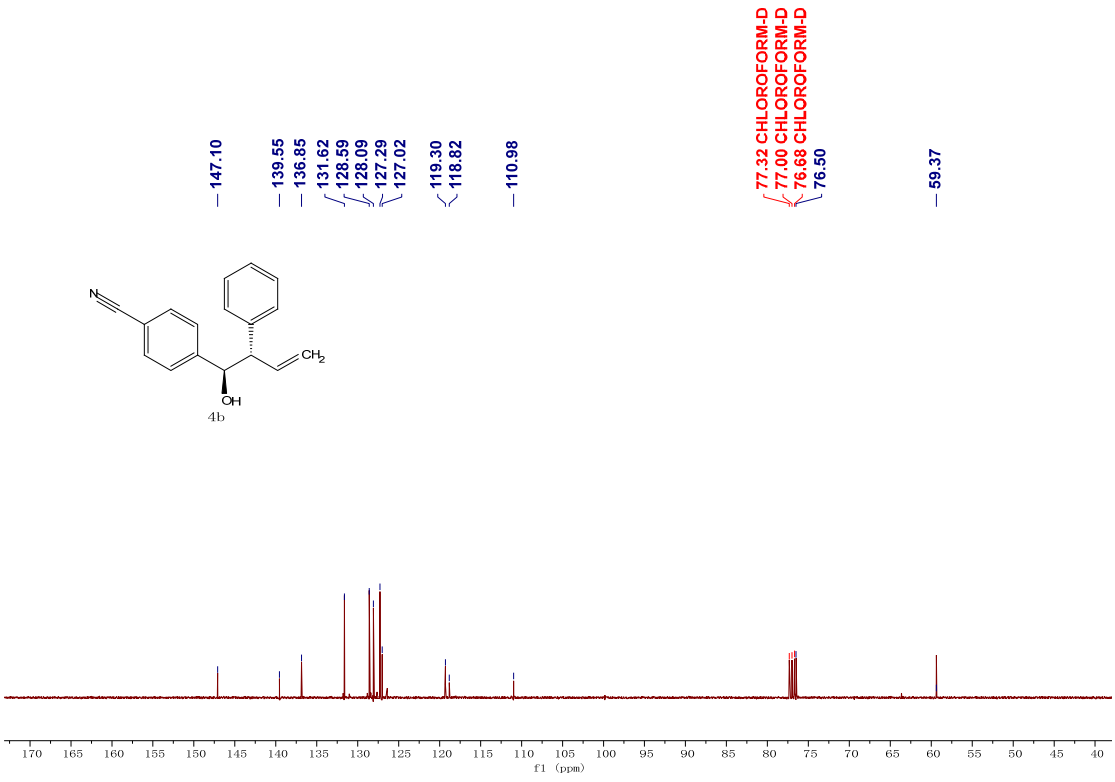
6.24
6.22
6.19
6.17
5.31
5.29
5.26
5.22
4.87
4.86
4.85
4.84
3.47
3.44
3.42
2.56
2.56
0.47
0.00
N
CH2
OH
4b
2.01
5.15
2.09
0.99
2.08
1.06
1.02
1.00
f1 (ppm)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128.09
127.29
127.02
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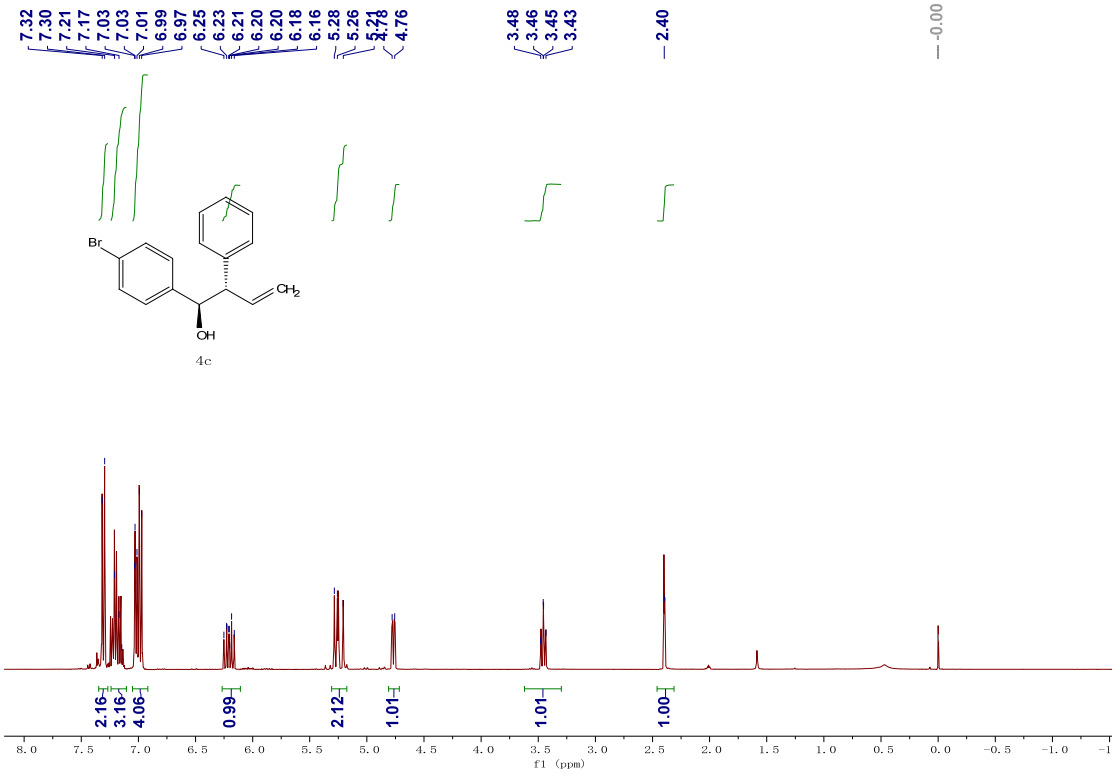
118.82
110.98
77.32 CHLOROFORM-D
77.00 CHLOROFORM-D
76.68 CHLOROFORM-D
76.50
59.37
N
CH2
OH
4b
f1 (ppm)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7.03
7.01
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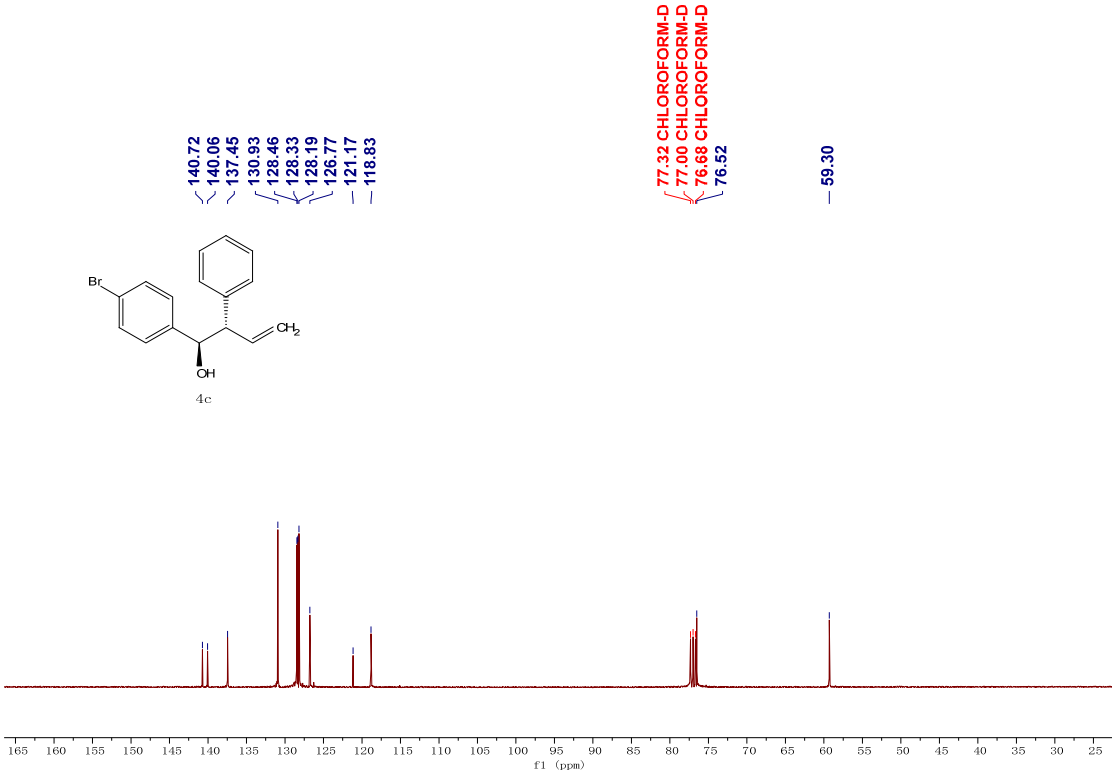
6.97
6.25
6.23
6.21
6.20
6.20
6.18
6.16
5.28
5.26
5.21
4.78
4.76
3.48
3.46
3.45
3.43
2.40
-0.00
Br
CH2
OH
4c
2.16
3.16
4.06
0.99
2.12
1.01
1.01
1.00
f1 (ppm)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126.77
121.17
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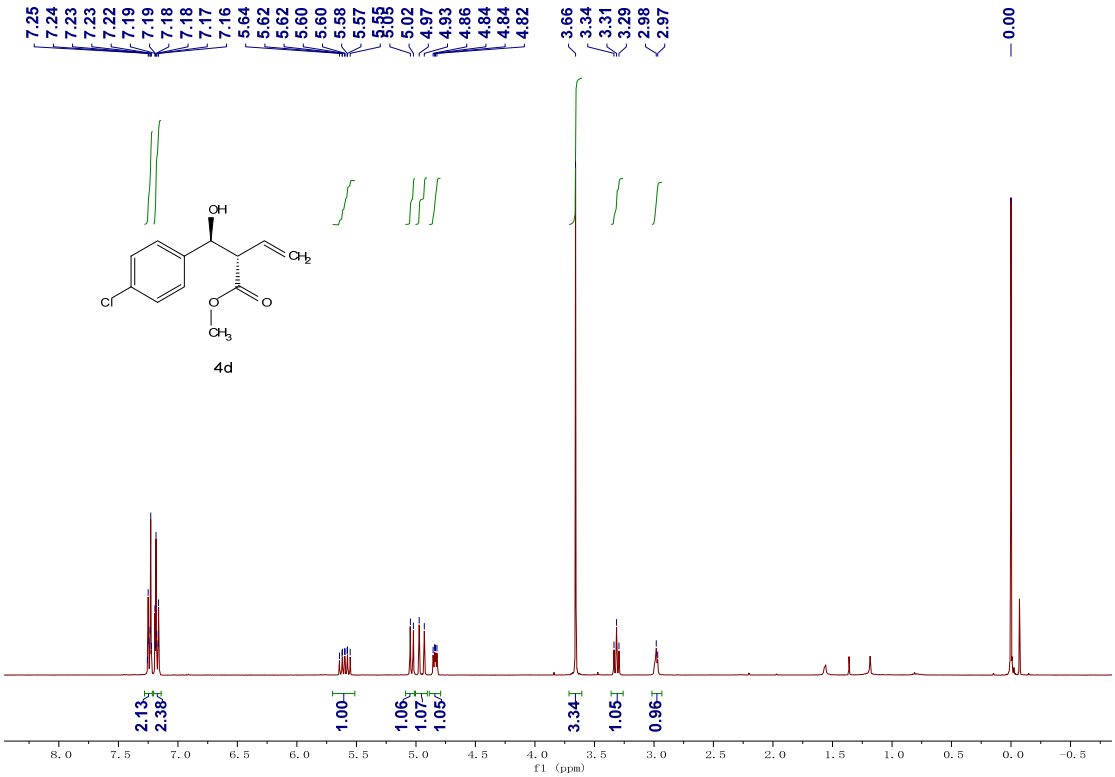
77.32 CHLOROFORM-D
77.00 CHLOROFORM-D
76.68 CHLOROFORM-D
76.52
59.30
Br
CH2
OH
4c
f1 (ppm)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5.62
5.60
5.60
5.58
5.57
5.55
5.05
5.02
4.97
4.93
4.86
4.84
4.84
4.82
3.66
3.34
3.31
3.29
2.98
2.97
0.00
OH
CH2
Cl
O
O
CH3
4d
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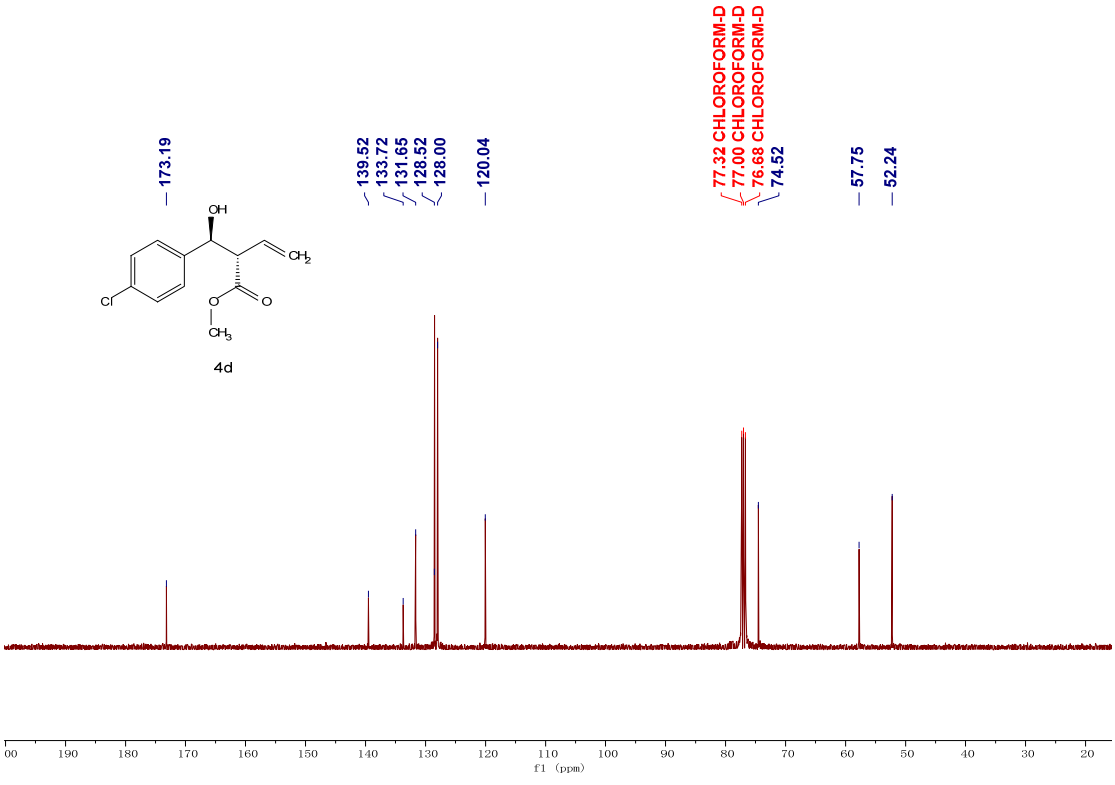
2.38
1.00
1.06
1.07
1.05
3.34
1.05
0.96
f1 (ppm)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131.65
128.52
128.00
120.04
77.32 CHLOROFORM-D
77.00 CHLOROFORM-D
76.68 CHLOROFORM-D
74.52
57.75
52.24
OH
CH2
Cl
O
O
CH3
4d
f1 (ppm)