Supporting information

Methane Monooxygenase Mimic Asymmetric Oxidation: Self-Assembling *μ*-Hydroxo, Carboxylate-Bridged Diiron(III) Catalyzed Enantioselective Dehydrogenation

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General Information

Proton (^{1}H NMR) and carbon (^{13}C NMR) nuclear magnetic resonance spectra were recorded at 500 MHz or 400 MHz and 126 MHz or 101 MHz, respectively. The chemical shifts are given in parts per million (ppm) on the delta (δ) scale. The solvent peak was used as a reference value, for ^{1}H NMR: $CDCl_3$ = 7.26 ppm; for ^{13}C NMR: $CDCl_3$ = 77.23 ppm. Analytical TLC was performed on precoated silica gel GF254 plates. Column chromatography was carried out on silica gel (200–300 mesh). Optical rotations were measured using a 2.5 mL cell with a 10 cm path length on Hanon P850 Automatic Polarimeter and concentrations (c) were reported in g × $(100\ mL)^{-1}$. HRMS were measured on the Q-TOF 6510 instruments. UV-vis spectra were carried on Agilent Cary 8454 UV-Visible spectrophotometer. Resonance Raman spectroscopy was measured on LabRAM HR Evolution in-situ UV laser confocal Raman Spectrometer. Enantiomeric excesses were determined by HPLC using a Daicel Chiralpak and Chiralcel column with hexane/*i*-PrOH as the eluent on Dionex instrument. All the solvents were freshly distilled prior to use according to the standard procedures.[1]

General Procedures

General procedure A: Dehydrogenative kinetic resolution of racemic substrates catalyzed by pre-synthesised diiron complexes

To a solution of racemic substrate (0.1 mmol, 1.0 equiv) in CH_2Cl_2 (1.0 mL), dimeric iron complex **C1-C8** (0.005 mmol, 5 mmol %) was added at -40 °C. Then 30% aqueous hydrogen peroxide (0.1 mmol, 10 μL, 1.0 equiv) was added and the reaction was then stirred at same tempreture for 24 h. Then the mixture was diluted with CH_2Cl_2 (20 mL), washed with water (10 mL), dried over $MgSO_4$, filtered and concentrated. The residue was purified by silica gel chromatography using ethyl acetate/petroleum ether as eluent to give the desired product.

General procedure B: Dehydrogenative kinetic resolution of racemic substrates catalyzed by self-assembled diiron complex

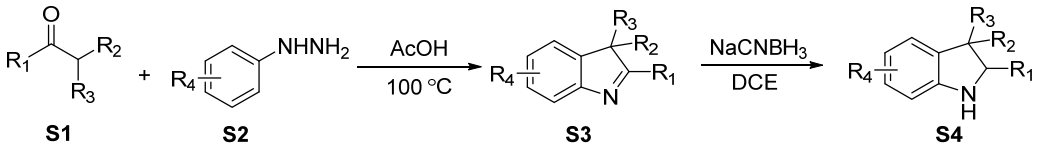
To a solution of racemic substrate (0.1 mmol, 1.0 equiv) in CH_2Cl_2 (1.0 mL), $\mathbf{C_{mono}8}$ (0.005 mmol, 3.7 mg, 5 mmol %) and sodium 6-methoxy-2-naphthoate (0.01 mmol, 2.2 mg, 10 mmol %) was added at -40 °C. Then 30% aqueous hydrogen peroxide (0.1 mmol, 10 μL, 1.0 equiv) was added as 4 portions in 2-hours intervals. The reaction was then stirred at same tempreture for 1-32 h. Then the mixture was diluted with CH_2Cl_2 (20 mL), washed with water (10 mL), dried over $MgSO_4$, filtered and concentrated. The residue was purified by silica gel chromatography using ethyl acetate/petroleum ether as eluent to give the desired product.

Synthesis of substrates

Substrates **1m**, **5b**, **5d**, **5e**, **5f**, **5g**, **8** and **11** were known compounds and prepared following the established procedures. [2-9]

General procedure C: Synthesis of racemic Substrates 1a-1l, 3a-3j, 5a, 5c, and 10:

Scheme S1. Preparation of substrates.

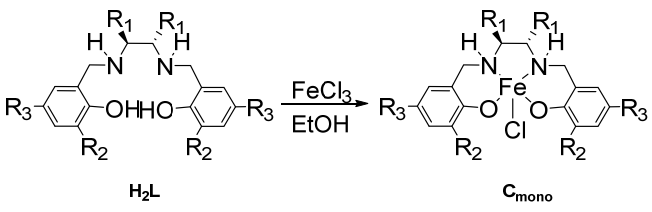


A mixture of arylhydrazine **S2** or its HCl salt (5.5 mmol) and **S1** (5 mmol) in AcOH (10 mL) was stirred at 100 ℃ for 1–6 h. The reaction was monitored by TLC. Upon completion, the reaction mixture was cooled with cold water and diluted with 1,2-dichloroethane (10 mL) followed by treatment with $NaCNBH_3$ (7.5 mmol, 1.5 equiv) in portions with cooling in cold water and was then stirred for 1 h at room temperature. The reaction was quenched with water, extracted with EtOAc and washed with sat. $NaHCO_3$. The organic layer was dried over $MgSO_4$, filtered, and concentrated. The residue was purified by chromatography with EtOAc/petroleum ether to provide the products.

Synthesis of monomeric Fe(salan) and Fe(salen) complexes

Fe(salan) complex $\mathbf{C_{mono}3}$ were prepared following established procedures.[10]

Scheme S2. Preparation of monomeric Fe(salan) complexes.



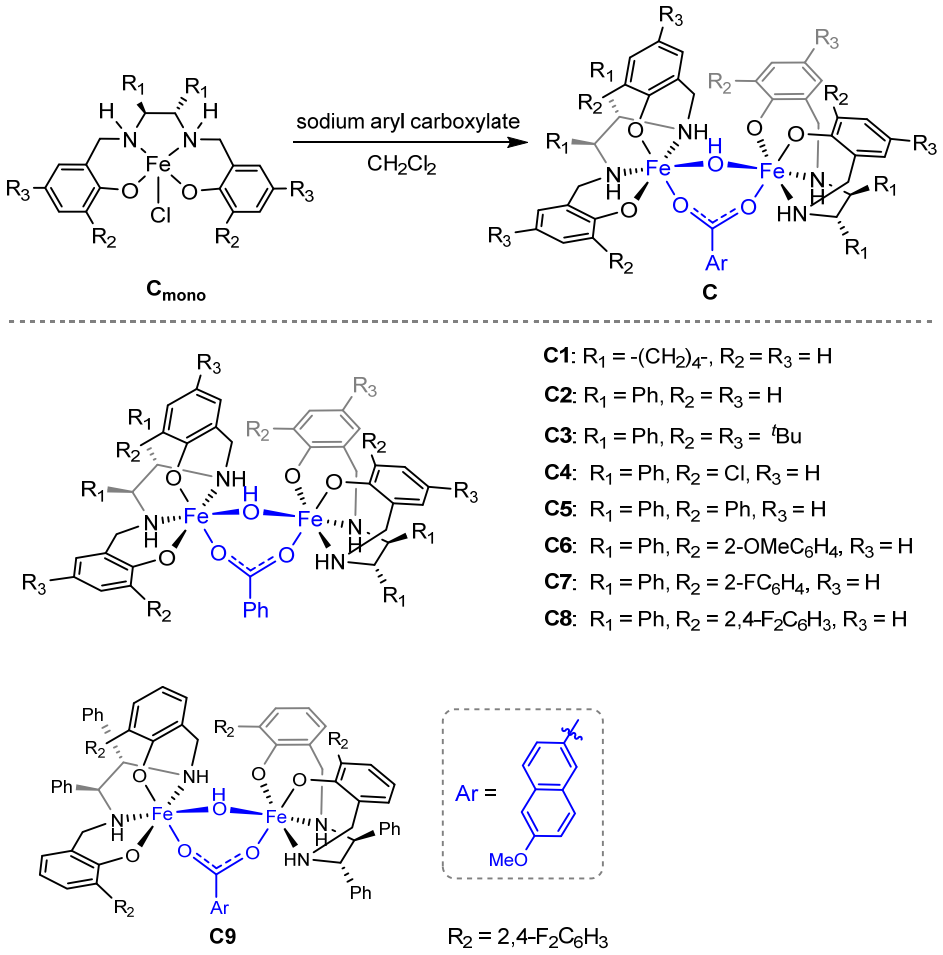
$\mathbf{C_{mono}1}$: R_1 = -$(CH_2)_4$-, R_2 = R_3 = H
$\mathbf{C_{mono}2}$: R_1 = Ph, R_2 = R_3 = H
$\mathbf{C_{mono}4}$: R_1 = Ph, R_2 = Cl, R_3 = H
$\mathbf{C_{mono}5}$: R_1 = Ph, R_2 = Ph, R_3 = H
$\mathbf{C_{mono}6}$: R_1 = Ph, R_2 = 2-$OMeC_6H_4$, R_3 = H
$\mathbf{C_{mono}7}$: R_1 = Ph, R_2 = 2-FC_6H_4, R_3 = H
$\mathbf{C_{mono}8}$: R_1 = Ph, R_2 = 2,4-$F_2C_6H_3$, R_3 = H

General procedure D: Synthesis of monomeric Fe(salan) complex $\mathbf{C_{mono}1}$, $\mathbf{C_{mono}2}$, $\mathbf{C_{mono}4}$-$\mathbf{C_{mono}8}$:

$FeCl_3$ (0.42 mmol, 1.05 equiv) was added to a solution of H_2L (0.4 mmol, 1.0 equiv) in ethanol (10 mL) giving a purplish solution which was refluxed for 4 h. Then the reaction mixture was evaporated in vacuo. The residue was chromatographed on silica gel (CH_2Cl_2 / MeOH = 19 : 1) to give the corresponding complex respectively.

Synthesis of carboxylate-bridged (*μ*-hydroxo) diiron(III) complexes C1-C10:

Scheme S3. Preparation of diiron(III) complexes.



General procedure E: Synthesis of diiron(III) complexes C1-C9:

Monomer complex $\mathbf{C_{mono}1}$-$\mathbf{C_{mono}8}$ (0.05 mmol, 1.0 equiv) dissolved in CH_2Cl_2-EtOH-acetone-H_2O (3 mL/ 3 mL/3 mL/ 1 drop) solution and additive sodium aryl carboxylate (20 equiv) was added. The mixure was maintained open-flask at room temperature for several days until the solid dimmeric iron complexes precipitated. UV-vis absorption spectra and ESI-MS was conducted to characterize these complexes. UV-vis absorption spectra used CH_2Cl_2 as solvent and the concentration is 10^{-5} mol/L.

Complex C1:

Reddish purple solid; ESI-MS *m/z* $[M - OH]^+$ calculated for $C_{47}H_{53}Fe_2N_4O_6$: 881.27, found 881.25; *m/z* $[M - H]^-$ calculated for $C_{47}H_{53}Fe_2N_4O_7$: 897.26, found 897.25.

UV-vis absorption features at 276, 311 and 498 nm and the corresponding monomer UV-vis absorption features at 275, 316 and 529 nm.

Complex C2:

Reddish purple solid; ESI-MS *m/z* $[M - OH]^+$ calculated for $C_{63}H_{57}Fe_2N_4O_6$: 1077.30, found 1077.30; *m/z* $[M - H]^-$ calculated for $C_{63}H_{57}Fe_2N_4O_7$: 1093.29, found 1093.32. UV-vis absorption features at 280, 310 and 491 nm and the corresponding monomer UV-vis absorption features at 316 and 504 nm.

Complex C3:

Purple solid; ESI-MS *m/z* $[M - OH]^+$ calculated for $C_{95}H_{121}Fe_2N_4O_6$: 1525.80, found 1525.76; *m/z* $[M - H]^-$ calculated for $C_{95}H_{121}Fe_2N_4O_7$: 1541.79, found 1541.80. UV-vis absorption features at 279, 329 and 543 nm and the corresponding monomer UV-vis absorption features at 281, 333 and 541 nm.

Complex C4:

Reddish purple solid; ESI-MS *m/z* $[M - OH]^+$ calculated for $C_{63}H_{53}Cl_4Fe_2N_4O_6$: 1213.14, found 1213.24; *m/z* $[M - H]^-$ calculated for $C_{63}H_{53}Cl_4Fe_2N_4O_7$: 1229.14, found 1229.17. UV-vis absorption features at 284 and 493 nm and the corresponding monomer UV-vis absorption features at 284, 317 and 525 nm.

Complex C5:

Purple solid; ESI-MS *m/z* $[M - OH]^+$ calculated for $C_{87}H_{73}Fe_2N_4O_6$: 1381.42, found 1381.37; *m/z* $[M - H]^-$ calculated for $C_{87}H_{73}Fe_2N_4O_7$: 1397.42, found 1397.41. UV-vis absorption features at 301 and 520 nm and the corresponding monomer UV-vis absorption features at 302 and 527 nm.

Complex C6:

Purple solid; ESI-MS *m/z* $[M - OH]^+$ calculated for $C_{91}H_{81}Fe_2N_4O_{10}$: 1501.46, found 1501.43; *m/z* $[M - H]^-$ calculated for $C_{91}H_{81}Fe_2N_4O_{11}$: 1517.46, found 1517.48. UV-vis absorption features at 301 and 518 nm and the corresponding monomer UV-vis absorption features at 300 and 541 nm.

Complex C7:

Purple solid; ESI-MS *m/z* $[M - OH]^+$ calculated for $C_{87}H_{69}F_4Fe_2N_4O_6$: 1453.38, found 1453.49; *m/z* $[M - H]^-$ calculated for $C_{87}H_{69}F_4Fe_2N_4O_7$: 1469.38, found 1469.38. UV-vis absorption features at 296 and 502 nm and the corresponding monomer UV-vis absorption features at 295 and 506 nm.

Complex C8:

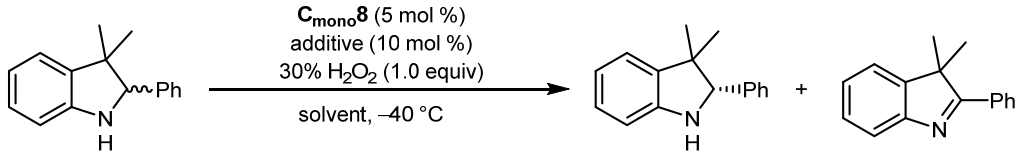
Purple solid; ESI-MS *m/z* $[M - OH]^+$ calculated for $C_{87}H_{65}F_8Fe_2N_4O_6$: 1525.35, found 1525.40; *m/z* $[M - H]^-$ calculated for $C_{87}H_{65}F_8Fe_2N_4O_7$: 1541.34, found 1541.34. UV-vis absorption features at 295 and 498 nm and the corresponding monomer UV-vis absorption features at 294 and 512 nm.

Complex C9:

Purple solid; ESI-MS *m/z* $[M - OH]^+$ calculated for $C_{92}H_{69}F_8Fe_2N_4O_7$: 1605.37, found 1605.37; *m/z* $[M - H]^-$ calculated for $C_{92}H_{69}F_8Fe_2N_4O_8$: 1621.37, found 1621.33. UV-vis absorption features at 302 and 521 nm.

Optimization of reaction conditions

Table S1. Solvent, additive and reaction temprature optimization of dehydrogenative kinetic resolution reaction[a]

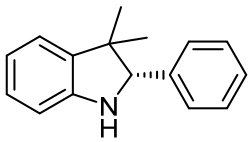


rac-**1a** (*S*)-**1a** **2a**

entry	additive	Solvent	conv. (%)[b]	ee (%)[c]	*s*[d]
1	PhCOONa	1,2-Dichloroethane	50	57	6.3
2	PhCOONa	Chloroform	55	22	1.7
3	PhCOONa	THF	45	0	n.d.
4	PhCOONa	Methanol	51	9	1.3
5	PhCOONa	Ethyl acetate	51	23	1.9
6	PhCOONa	Toluene	47	17	1.7
7	PhCOONa	Acetone	53	15	1.5
8	PhCOONa	Acetonitrile	47	13	1.5
9	PhCOONa	CH_2Cl_2	49	71	14
10[e]	PhCOONa	CH_2Cl_2	55	41	2.9
11[f]	PhCOONa	CH_2Cl_2	40	45	8.0
12	1-Naphthol	CH_2Cl_2	53	9	1.3

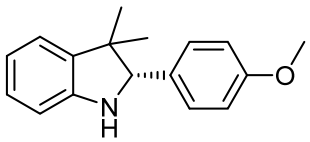
[a]Reaction condition: to rac-**1a** (0.1 mmol), monoiron $\mathbf{C_{mono}8}$ (5 mol %) and additive (10 mol %) in solvent (1.0 mL) at -40 ºC was added 30% aqueous H_2O_2 (0.1 mmol) as four portions in 2 h intervals for 6 h, and the mixture was stirred at -40 ºC for 18-24 h, unless otherwise noted. [b]Conversion was calculated from the isolated yield of recovered (*S*)-**1a**. [c]Determined by HPLC analysis on a chiral stationary phase. [d]Selectivity (*s*) values were calculated through the equation $s = \ln[(1 - C)(1 - ee)]/\ln[(1 - C)(1 + ee)]$, where C is the conversion. [e]Reaction temperature was -20 ºC. [f]Reaction temperature was -60 ºC.

Analytical data for products



(*S*)-3,3-Dimethyl-2-phenylindoline (1a)

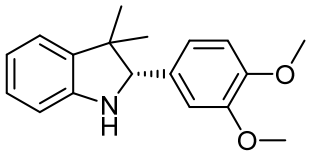
It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (1:9) as eluent to afford **1a** (11.2 mg, 50% yield). Yellow solid, m.p. 55-58 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.47 (dd, *J* = 5.3, 3.5 Hz, 2H), 7.39–7.30 (m, 3H), 7.12–7.06 (m, 2H), 6.82 (td, *J* = 7.4, 0.9 Hz, 1H), 6.75 (d, *J* = 7.7 Hz, 1H), 4.62 (s, 1H), 1.45 (s, 3H), 0.76 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 149.1, 139.9, 138.4, 128.3, 127.7, 127.7, 127.6, 122.7, 119.5, 109.7, 74.7, 45.6, 26.7, 24.7. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 296 nm), retention time: t_{major} = 9.723 min, t_{minor} = 5.240 min, ee = 94.10%; $[\alpha]_D^{20}$ = + 173.35 (c = 0.31, THF). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{16}H_{18}N$: 224.1434, found 224.1428. The absolute configuration was assigned as *S* by comparing the optical rotation and HPLC analysis with reported data.[11]



(*S*)-2-(4-Methoxyphenyl)-3,3-dimethylindoline (1b)

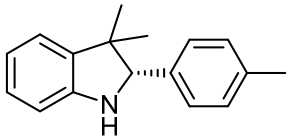
It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (1:9) as eluent to afford **1b** (12.5 mg, 49% yield) and **2b**. Yellow solid, m.p. 75-76 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40–7.34 (m, 2H), 7.11–7.04 (m, 2H), 6.92–6.87 (m, 2H), 6.80 (td, *J* = 7.4, 0.7 Hz, 1H), 6.73 (d, *J* = 7.7 Hz, 1H), 4.56 (s, 1H), 3.83 (s, 3H), 1.41 (s, 3H), 0.75 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.1, 149.0, 138.3, 131.7, 128.5, 127.4, 122.5, 119.2, 113.5, 109.4, 74.1, 55.3, 45.3, 26.4, 24.5. HPLC: the ee value was determined by

HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 215 nm), retention time: t_{major} = 7.863 min, t_{minor} = 5.160 min, ee = 97.96%; $[\alpha]_D^{20}$ = + 126.4 (c = 0.23, THF). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{17}H_{20}NO$: 254.1539, found 254.1551.



(*S*)-2-(3,4-Dimethoxyphenyl)-3,3-dimethylindoline (1c)

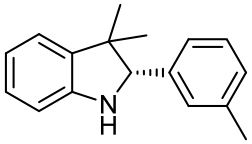
It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (2:8) as eluent to afford **1c** (13.6 mg, 48% yield). White solid, m.p. 98-99 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.08 (m, 3H), 6.96 (dd, *J* = 8.2, 1.8 Hz, 1H), 6.86 (d, *J* = 8.2 Hz, 1H), 6.80 (td, *J* = 7.4, 0.8 Hz, 1H), 6.73 (d, *J* = 7.7 Hz, 1H), 4.55 (s, 1H), 3.90 (s, 3H), 3.89 (s, 3H), 1.42 (s, 3H), 0.75 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 148.9, 148.6, 138.4, 132.5, 127.6, 122.7, 119.7, 119.4, 110.9, 110.8, 109.5, 74.5, 56.1, 45.5, 26.5, 24.7. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 254 nm), retention time: t_{major} = 13.733 min, t_{minor} = 7.470 min, ee = 96.04%; $[\alpha]_D^{20}$ = + 174.2 (c = 0.23, THF). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{18}H_{22}NO_2$: 284.1645, found 284.1633.



(*S*)-3,3-Dimethyl-2-(p-tolyl)indoline (1d)

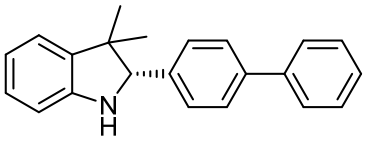
It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (1:9) as eluent to afford **1d** (12.1 mg, 51% yield). Yellow solid, m.p. 66-67 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.36 (d, *J* = 8.0 Hz, 2H), 7.18 (d, *J* = 8.0 Hz, 2H), 7.12–7.05 (m, 2H), 6.81 (t, *J* = 7.4 Hz, 1H), 6.74 (d, *J* = 7.7 Hz, 1H), 4.58 (s, 1H), 2.38 (s, 3H), 1.44 (s, 3H), 0.76 (s, 3H); ^{13}C

NMR (126 MHz, $CDCl_3$) δ 149.4, 138.4, 137.3, 136.9, 129.0, 127.6, 127.6, 122.7, 119.2, 109.5, 74.6, 45.5, 26.7, 24.7, 21.3. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 296 nm), retention time: t_{major} = 10.580 min, t_{minor} = 4.897 min, ee = 90.96%; $[\alpha]_D^{20}$ = + 86.44 (c = 0.31, THF). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{17}H_{20}N$: 238.1590 found 238.1595.



(*S*)-3,3-Dimethyl-2-(m-tolyl)indoline (1e)

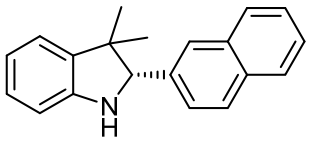
It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (1:9) as eluent to afford **1e** (11.7 mg, 49% yield). Pale yellow solid, m.p. 57-58 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.27–7.23 (m, 3H), 7.12–7.05 (m, 3H), 6.81 (td, *J* = 7.4, 0.9 Hz, 1H), 6.74 (d, *J* = 7.7 Hz, 1H), 4.57 (s, 1H), 4.24 (brs, 1H), 2.37 (s, 3H), 1.43 (s, 3H), 0.75 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 149.3, 139.9, 138.5, 137.9, 128.4, 128.3, 128.2, 127.6, 124.7, 122.7, 119.2, 109.4, 74.7, 45.5, 26.8, 24.7, 21.7. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 10/90, 1.0 mL/min, 243 nm), retention time: t_{major} = 7.320 min, t_{minor} =4.840 min, ee = 95.52%; $[\alpha]_D^{20}$ = + 100.7 (c = 0.33, $CHCl_3$). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{17}H_{20}N$: 238.1590, found 239.1597.



(*S*)-2-([1,1'-Biphenyl]-4-yl)-3,3-dimethylindoline (1f)

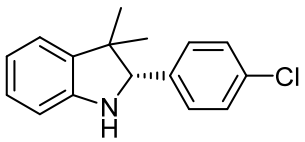
It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (1:9) as eluent to afford **1f** (14.4 mg, 48% yield). Yellow solid, m.p. 87-89 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.66–

7.59 (m, 4H), 7.54 (d, J = 8.2 Hz, 2H), 7.49–7.44 (m, 2H), 7.39–7.35 (m, 1H), 7.14–7.08 (m, 2H), 6.83 (td, J = 7.4, 0.8 Hz, 1H), 6.77 (d, J = 7.7 Hz, 1H), 4.66 (s, 1H), 1.48 (s, 3H), 0.81 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 149.3, 141.0, 140.6, 139.1, 138.3, 129.0, 128.1, 127.6, 127.4, 127.2, 127.0, 122.7, 119.4, 109.6, 74.5, 45.7, 26.7, 24.8. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 248 nm), retention time: t_{major} = 9.803 min, t_{minor} = 6.130 min, ee = 96.76%; $[\alpha]_D^{20}$ = + 176.2 (c = 0.11, THF). HRMS (EI) *m/z* [M + H]$^+$ calculated for $C_{22}H_{22}N$: 300.1747, found 300.1742.



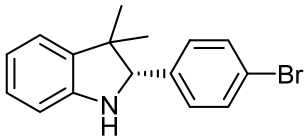
(*S*)-3,3-Dimethyl-2-(naphthalen-2-yl)indoline (1g)

It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (1:9) as eluent to afford **1g** (13.2 mg, 48% yield). White solid, m.p. 87-89 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.95 (s, 1H), 7.89–7.82 (m, 3H), 7.59 (dd, J = 8.5, 1.6 Hz, 1H), 7.53–7.47 (m, 2H), 7.17–7.08 (m, 2H), 6.84 (td, J = 7.4, 0.9 Hz, 1H), 6.78 (d, J = 7.7 Hz, 1H), 4.78 (s, 1H), 1.52 (s, 3H), 0.79 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 149.4, 138.3, 137.7, 133.4, 133.3, 128.1, 127.9, 127.8, 127.7, 126.3, 126.3, 126.0, 125.9, 122.7, 119.3, 109.5, 74.8, 45.8, 27.0, 24.9. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 215 nm), retention time: t_{major} = 12.497 min, t_{minor} = 5.613 min, ee = 93.78%; $[\alpha]_D^{20}$ = + 143.6 (c = 0.29, $CHCl_3$). HRMS (EI) *m/z* [M + H]$^+$ calculated for $C_{20}H_{20}N$: 274.1590, found 274.1597.



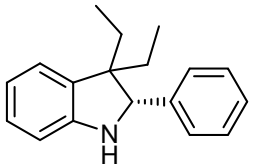
(*S*)-2-(4-Chlorophenyl)-3,3-dimethylindoline (1h)

It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (1:9) as eluent to afford **1h** (13.4 mg, 52% yield). Pale yellow solid, m.p. 93-94 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46–7.40 (m, 2H), 7.39–7.29 (m, 2H), 7.15–7.05 (m, 2H), 6.82 (td, *J* = 7.4, 0.9 Hz, 1H), 6.74 (d, *J* = 7.7 Hz, 1H), 4.59 (s, 1H), 4.12 (brs, 1H), 1.44 (s, 3H), 0.74 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 149.2, 138.6, 138.0, 133.3, 128.9, 128.4, 127.7, 122.7, 119.4, 109.5, 74.0, 45.5, 26.6, 24.7. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 254 nm), retention time: t_{major} = 12.657 min, t_{minor} = 5.570 min, ee = 84.22%; $[\alpha]_D^{20}$ = + 175.4 (c = 0.33, THF). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{16}H_{17}ClN$: 258.1044, found 258.1037.



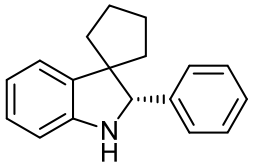
(*S*)-2-(4-Bromophenyl)-3,3-dimethylindoline (1i)

It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (1:9) as eluent to afford **1i** (15.7 mg, 52% yield). Yellow solid, m.p. 76-77 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.52–7.45 (m, 2H), 7.37–7.32 (m, 2H), 7.11–7.03 (m, 2H), 6.81 (td, *J* = 7.4, 0.8 Hz, 1H), 6.74 (d, *J* = 7.7 Hz, 1H), 4.56 (s, 1H), 1.42 (s, 3H), 0.73 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 148.9, 139.0, 138.1, 131.4, 129.3, 127.7, 122.7, 121.5, 119.6, 109.7, 74.1, 45.6, 26.6, 24.7. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 215 nm), retention time: t_{major} = 11.957 min, t_{minor} = 5.393 min, ee = 85.10%; $[\alpha]_D^{20}$ = + 91.6 (c = 0.31, THF). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{16}H_{17}BrN$: 302.0539, found 302.0543.



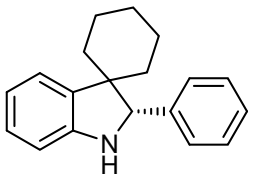
(*S*)-3,3-Diethyl-2-phenylindoline (1j)

It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (1:9) as eluent to afford **1j** (13.1 mg, 52% yield). Pale yellow solid, m.p. 36-39 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.45 (d, *J* = 7.2 Hz, 2H), 7.40–7.26 (m, 3H), 7.11 (td, *J* = 7.6, 0.9 Hz, 1H), 7.01 (d, *J* = 7.2 Hz, 1H), 6.80 (t, *J* = 7.4 Hz, 1H), 6.74 (d, *J* = 7.7 Hz, 1H), 4.89 (s, 1H), 4.09 (brs, 1H), 2.03 (dq, *J* = 14.9, 7.5 Hz, 1H), 1.65 (dq, *J* = 14.7, 7.4 Hz, 1H), 1.52 (dq, *J* = 14.9, 7.5 Hz, 1H), 1.00 (t, *J* = 7.5 Hz, 3H), 0.88 (dq, *J* = 14.5, 7.4 Hz, 1H), 0.64 (d, *J* = 7.5 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.4, 140.4, 133.7, 128.1, 127.7, 127.3, 127.3, 124.7, 118.3, 109.2, 70.2, 52.5, 27.4, 26.3, 9.4, 8.2. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 270 nm), retention time: t_{major} = 16.910 min, t_{minor} = 6.547 min, ee = 80.62%; $[\alpha]_D^{20}$ = + 53.2 (c = 0.16, THF). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{17}H_{22}N$: 252.1747, found 252.1753.



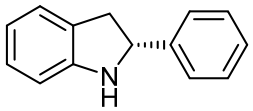
(*S*)-2'-Phenylspiro[cyclopentane-1,3'-indoline] (1k)

It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (1:9) as eluent to afford **1k** (11.8 mg, 47% yield). Yellow solid, m.p. 49-51 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 (dt, *J* = 3.8, 2.1 Hz, 2H), 7.35–7.27 (m, 3H), 7.13–7.04 (m, 2H), 6.80 (td, *J* = 7.4, 0.9 Hz, 1H), 6.72 (d, *J* = 7.6 Hz, 1H), 4.66 (s, 1H), 2.08–1.98 (m, 2H), 1.87–1.77 (m, 1H), 1.74–1.60 (m, 2H), 1.46 (t, *J* = 7.2 Hz, 2H), 1.29–1.21 (m, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 149.7, 141.2, 138.3, 128.4, 127.84, 127.82, 127.5, 123.0, 119.4, 109.2, 73.9, 57.5, 39.9, 35.1, 24.8, 24.8. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 300 nm), retention time: t_{major} = 7.987 min, t_{minor} = 5.530 min, ee = 96.04%; $[\alpha]_D^{20}$ = + 13.5 (c = 0.27, THF). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{18}H_{20}N$: 250.1590, found 250.1587.



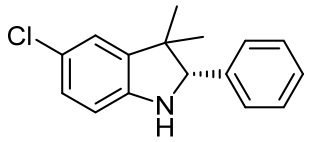
(*S*)-2'-Phenylspiro[cyclohexane-1,3'-indoline (1l)

It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (1:9) as eluent to afford **1l** (12.7 mg, 48% yield). White solid, m.p. 76-77 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.34–7.25 (m, 6H), 7.10 (td, *J* = 7.6, 1.2 Hz, 1H), 6.78 (td, *J* = 7.4, 0.7 Hz, 1H), 6.70 (d, *J* = 7.7 Hz, 1H), 4.58 (s, 1H), 1.90–1.78 (m, 2H), 1.71 (dd, *J* = 11.9, 6.6 Hz, 2H), 1.61–1.53 (m, 1H), 1.52–1.37 (m, 3H), 1.25–1.10 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.1, 141.3, 137.5, 128.3, 128.1, 127.8, 127.7, 124.5, 118.8, 109.1, 73.2, 49.4, 37.6, 32.0, 26.0, 23.2, 22.4. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 10/90, 1.0 mL/min, 305 nm), retention time: t_{major} = 8.070 min, t_{minor} = 5.913 min, ee = 95.26%; $[\alpha]_D^{20}$ = - 54.7 (c = 0.34, THF). HRMS (EI) *m/z* [M + H]$^+$ calculated for $C_{19}H_{22}N$: 264.1747, found 264.1738.



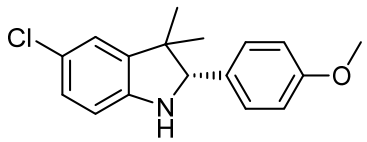
(*R*)-2-Phenylindoline (1m)

It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (1:9) as eluent to afford **1m** (10.4 mg, 53% yield). Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 (dd, *J* = 5.4, 3.4 Hz, 2H), 7.31–7.25 (m, 2H), 7.22 (dt, *J* = 5.1, 2.1 Hz, 1H), 7.02 (dd, *J* = 12.5, 7.4 Hz, 2H), 6.69 (td, *J* = 7.5, 0.8 Hz, 1H), 6.62 (d, *J* = 7.7 Hz, 1H), 4.90 (t, *J* = 9.0 Hz, 1H), 3.39 (dd, *J* = 15.6, 9.2 Hz, 1H), 2.94 (dd, *J* = 15.6, 8.8 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.9, 144.6, 128.8, 128.4, 127.8, 127.7, 126.5, 125.4, 124.8, 119.2, 109.3, 63.7, 39.7. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 10/90, 1.0 mL/min, 215 nm), retention time: t_{major} = 8.923 min, t_{minor} = 14.263 min, ee = 69.92%; $[\alpha]_D^{20}$ = + 31.6 (c = 0.18, THF). HRMS (EI) *m/z* [M + H]$^+$ calculated for $C_{14}H_{14}N$: 196.1121, found 196.1115.



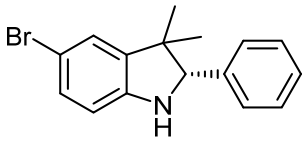
(*S*)-5-Chloro-3,3-dimethyl-2-phenylindoline (3a)

It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (1:9) as eluent to afford **3a** (12.4 mg, 48% yield). Yellow solid, m.p. 81-83 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49–7.40 (m, 2H), 7.40–7.29 (m, 3H), 7.03 (dd, *J* = 8.2, 2.2 Hz, 1H), 6.99 (d, *J* = 2.1 Hz, 1H), 6.63 (d, *J* = 8.2 Hz, 1H), 4.61 (s, 1H), 1.42 (s, 3H), 0.74 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 147.9, 140.3, 139.5, 128.4, 127.9, 127.6, 127.3, 123.8, 123.1, 110.2, 75.0, 45.9, 26.7, 24.6. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 215 nm), retention time: t_{major} = 17.350 min, t_{minor} = 6.153 min, ee = 93.52%; $[\alpha]_D^{20}$ = + 113.8 (c = 0.42, THF). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{16}H_{17}ClN$: 258.1044, found 258.1051.



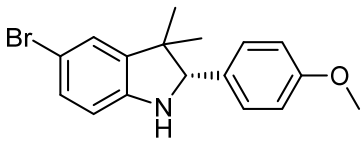
(*S*)-5-Chloro-2-(4-methoxyphenyl)-3,3-dimethylindoline (3b)

It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (2:8) as eluent to afford **3b** (13.8 mg, 48% yield). White solid, m.p. 106-107 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39–7.30 (m, 2H), 7.01 (dd, *J* = 8.2, 2.2 Hz, 1H), 6.98 (d, *J* = 2.1 Hz, 1H), 6.92–6.83 (m, 2H), 6.61 (d, *J* = 8.2 Hz, 1H), 4.54 (s, 1H), 3.82 (s, 3H), 1.38 (s, 3H), 0.73 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.4, 148.0, 140.4, 131.5, 128.6, 127.2, 123.7, 123.1, 113.7, 110.2, 74.5, 55.5, 45.8, 26.6, 24.5. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 255 nm), retention time: t_{major} = 12.883 min, t_{minor} = 5.277 min, ee = 95.36%; $[\alpha]_D^{20}$ = + 71.62 (c = 0.21, THF). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{17}H_{19}ClNO$: 288.1150, found 288.1152.



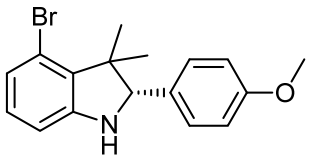
(*S*)-5-Bromo-3,3-dimethyl-2-phenylindoline (3c)

It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (1:9) as eluent to afford **3c** (14.2 mg, 47% yield). Yellow solid, m.p. 77-79 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.42 (dd, *J* = 8.2, 1.2 Hz, 2H), 7.38–7.31 (m, 3H), 7.17 (dd, *J* = 8.2, 2.0 Hz, 1H), 7.12 (d, *J* = 2.0 Hz, 1H), 6.59 (d, *J* = 8.2 Hz, 1H), 4.60 (s, 1H), 1.41 (s, 3 H), 0.73 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 148.4, 140.7, 139.5, 130.2, 128.4, 127.9, 127.5, 125.9, 110.8, 74.9, 45.9, 26.7, 24.6. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 320 nm), retention time: t_{major} = 16.910 min, t_{minor} = 6.030 min, ee = 95.72%; $[\alpha]_D^{20}$ = + 116.7 (c = 0.29, THF). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{16}H_{17}BrN$: 302.0539, found 302.0533.



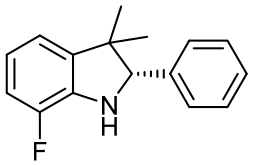
(*S*)-5-Bromo-2-(4-methoxyphenyl)-3,3-dimethylindoline (3d)

It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (2:8) as eluent to afford **3d** (15.6 mg, 47% yield). Yellow solid, m.p. 102-103 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35–7.30 (m, 2H), 7.15 (dd, *J* = 8.2, 2.0 Hz, 1H), 7.11 (d, *J* = 2.0 Hz, 1H), 6.91–6.85 (m, 2H), 6.57 (d, *J* = 8.2 Hz, 1H), 4.53 (s, 1H), 3.82 (s, 3H), 1.37 (s, 3H), 0.73 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.4, 148.5, 140.8, 131.5, 130.1, 128.6, 125.9, 113.8, 110.7, 74.5, 55.5, 45.7, 26.7, 24.5. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 307 nm), retention time: t_{major} = 13.857 min, t_{minor} = 5.370 min, ee = 96.00%; $[\alpha]_D^{20}$ = + 80.72 (c = 0.15, $CHCl_3$). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{17}H_{19}BrNO$: 332.0645, found 332.0634.



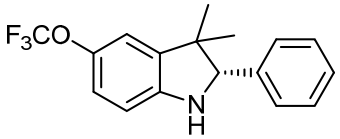
(*S*)-4-Bromo-2-(4-methoxyphenyl)-3,3-dimethylindoline(3e)

It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (2:8) as eluent to afford **3e** (16.3 mg, 49% yield). Yellow solid, m.p. 95-96 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 (d, *J* = 8.6 Hz, 2H), 7.00–6.84 (m, 4H), 6.62 (dd, *J* = 7.0, 1.6 Hz, 1H), 4.52 (s, 1H), 3.83 (s, 3H), 1.57 (s, 3H), 0.86 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.5, 151.6, 134.7, 131.0, 129.2, 129.1, 123.9, 119.6, 113.7, 108.4, 74.0, 55.5, 47.8, 25.7, 21.7. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 215 nm), retention time: t_{major} = 10.933 min, t_{minor} = 6.173 min, ee = 90.04%; $[\alpha]_D^{20}$ = + 83.1 (c = 0.17, THF). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{17}H_{19}BrNO$: 332.0645, found 332.0641.



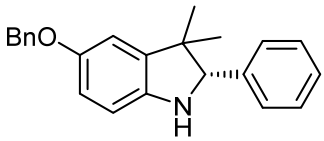
(*S*)-7-Fluoro-3,3-dimethyl-2-phenylindoline (3f)

It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (1:9) as eluent to afford **3f** (12.6 mg, 52% yield). Brown solid, m.p. 38-39 ºC.^{1}H NMR (500 MHz, $CDCl_3$) δ 7.49–7.45 (m, 2H), 7.41–7.29 (m, 3H), 6.92–6.81 (m, 2H), 6.75–6.70 (m, 1H), 4.65 (s, 1H), 1.44 (s, 3H), 0.75 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 149.1 (d, *J* = 240.3 Hz), 141.9 (d, *J* = 4.5 Hz), 139.4, 128.4, 127.9, 127.6, 119.7 (d, *J* = 5.6 Hz), 118.2 (d, *J* = 2.9 Hz), 114.2 (d, *J* = 17.4 Hz), 75.3, 46.3 (d, *J* = 2.4 Hz), 26.6, 24.6. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 240 nm), retention time: t_{major} = 5.840 min, t_{minor} = 4.547 min, ee = 74.36%; $[\alpha]_D^{20}$ = + 12.6 (c = 0.12, THF). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{16}H_{17}FN$: 242.1340, found 242.1331.



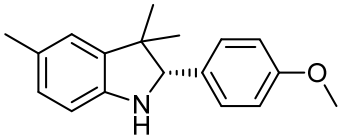
(*S*)-3,3-Dimethyl-2-phenyl-5-(trifluoromethoxy)indoline (3g)

It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (1:9) as eluent to afford **3g** (15.4 mg, 50% yield). Yellow solid, m.p. 67-69 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.46–7.42 (m, 2H), 7.41–7.30 (m, 3H), 6.93 (dd, *J* = 15.4, 7.0 Hz, 2H), 6.65 (d, *J* = 8.3 Hz, 1H), 4.65 (s, 1H), 1.44 (s, 3H), 0.75 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 148.1, 142.3, 139.8, 139.5, 128.4, 128.0, 127.6, 121.0 (q, *J* = 255.1 Hz), 120.6, 116.6, 109.2, 75.1, 45.8, 26.6, 24.6. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 304 nm), retention time: t_{major} = 15.567 min, t_{minor} = 6.167 min, ee = 86.68%; $[\alpha]_D^{20}$ = + 56.43 (c = 0.33, THF). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{17}H_{17}F_3NO$: 308.1257, found 308.1262.



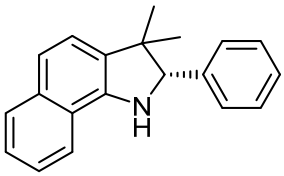
(*S*)-5-(Benzyloxy)-3,3-dimethyl-2-phenylindoline (3h)

It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (2:8) as eluent to afford **3h** (17.2 mg, 52% yield). Yellow solid, m.p. 93-95 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.47 (d, *J* = 7.3 Hz, 2H), 7.39 (m, 5H), 7.09 (dd, *J* = 14.9, 7.4 Hz, 2H), 6.99 (d, *J* = 8.6 Hz, 2H), 6.81 (t, *J* = 7.4 Hz, 1H), 6.73 (d, *J* = 7.7 Hz, 1H), 5.09 (s, 2H), 4.56 (s, 1H), 1.42 (s, 3H), 0.77 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 158.5, 149.4, 138.4, 137.3, 132.3, 128.8, 128.7, 128.2, 127.7, 127.5, 122.7, 119.2, 114.6, 109.4, 72.2, 70.2, 45.4, 26.6, 24.6. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 215 nm), retention time: t_{major} = 20.373 min, t_{minor} = 6.627 min, ee = 81.08%; $[\alpha]_D^{20}$ = + 166.6 (c = 0.41, THF). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{23}H_{24}NO$: 330.1852, found 330.1871.



(*S*)-2-(4-Methoxyphenyl)-3,3,5-trimethylindoline (3i)

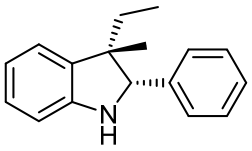
It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (2:8) as eluent to afford **3i** (13.1 mg, 49% yield). Pale yellow solid, m.p. 61-62 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 (d, *J* = 8.5 Hz, 2H), 6.90 (m, 4H), 6.64 (d, *J* = 7.7 Hz, 1H), 4.53 (s, 1H), 3.83 (s, 3H), 2.31 (s, 3H), 1.40 (s, 3H), 0.74 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.2, 147.0, 138.7, 132.2, 128.7, 128.5, 127.8, 123.5, 113.6, 109.3, 74.5, 55.5, 45.4, 26.5, 24.6, 21.2. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 215 nm), retention time: t_{major} = 9.197 min, t_{minor} = 5.367 min, ee = 92.34%; $[\alpha]_D^{20}$ = + +113.4 (c = 0.17, THF). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{18}H_{21}NO$: 268.1696, found 268.1704.



(*S*)-3,3-Dimethyl-2-phenyl-2,3-dihydro-1H-benzo[g]indole (3j)

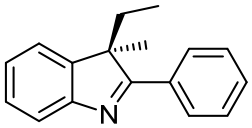
It was prepared following the general procedure B and purified by silica gel flash chromatography using CH_2Cl_2/petroleum ether (1:1) as eluent to afford **3j** (13.7 mg, 50% yield). Yellow solid, m.p. 84-85 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.97 (d, *J* = 8.6 Hz, 1H), 7.79 (d, *J* = 8.2 Hz, 1H), 7.65 (d, *J* = 8.5 Hz, 1H), 7.58–7.53 (m, 2H), 7.43–7.32 (m, 4H), 7.23 (m, 1H), 7.07 (d, *J* = 8.5 Hz, 1H), 4.72 (s, 1H), 1.77 (s, 3H), 1.01 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 139.7, 131.2, 130.0, 129.7, 129.1, 128.3, 128.3, 127.9, 126.4, 121.8, 113.1, 75.4, 47.4, 27.6, 23.2. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 248 nm), retention time: t_{major} = 9.923 min, t_{minor} = 6.053 min, ee = 80.50%; $[\alpha]_D^{20}$ = +

176.2 (c = 0.31, THF). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{20}H_{20}N$: 274.1590, found 274.1586.



(2*S*,3*S*)-3-Ethyl-3-methyl-2-phenylindoline (5a)

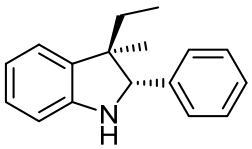
It was prepared following the general procedure B and purified by silica gel flash chromatography using CH_2Cl_2/petroleum ether (1:1) as eluent to afford **5a** (11.4 mg, 48% yield). Pale yellow solid, m.p. 63-64 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.65 (d, *J* = 7.3 Hz, 2H), 7.49 (dt, *J* = 25.6, 7.2 Hz, 3H), 7.24 (dd, *J* = 16.3, 7.7 Hz, 2H), 6.94 (t, *J* = 7.4 Hz, 1H), 6.86 (d, *J* = 7.7 Hz, 1H), 4.81 (s, 1H), 4.07 (brs, 1H), 1.60–1.52 (m, 4H), 0.95–0.87 (m, 1H), 0.80 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 149.9, 139.5, 136.2, 128.2, 127.8, 127.6, 127.5, 124.3, 118.6, 109.6, 76.0, 48.4, 28.0, 22.6, 8.4. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 301 nm), retention time: t_{major} = 12.993 min, t_{minor} = 6.150 min, ee = 93.04%; $[\alpha]_D^{20}$ = + 34.6 (c = 0.13, THF). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{17}H_{20}N$: 238.1590, found 238.1597. The absolute configuration was assigned as *S* by comparing the optical rotation and HPLC analysis with reported data[5]. The diastereomer was determined by comparing with reported data.[5]



(*R*)-3-Ethyl-3-methyl-2-phenyl-3H-indole (6a)

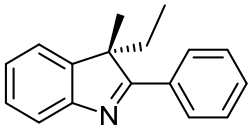
It was prepared following the general procedure B and purified by silica gel flash chromatography using CH_2Cl_2/petroleum ether (1:1) as eluent to afford **6a** (10.6 mg, 45% yield).Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.08–8.02 (m, 2H), 7.62 (d, *J* = 7.7 Hz, 1H), 7.44–7.39 (m, 3H), 7.29 (ddd, *J* = 7.7, 6.8, 2.0 Hz, 1H), 7.23–7.18 (m, 2H), 2.20 (dq, *J* = 14.7, 7.4 Hz, 1H), 2.06 (dq, *J* = 14.8, 7.4 Hz, 1H), 1.51 (s, 3H), 0.30 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 182.5, 145.7, 130.8, 128.9,

128.3, 128.0, 126.0, 121.2, 120.9, 59.6, 59.0, 32.2, 24.5, 8.9. HPLC: the ee value was determined by HPLC analysis (Chiralcel OD, *i*-PrOH/Hexane = 10/90, 1.0 mL/min, 331 nm), retention time: t_{major} = 4.163 min, t_{minor} = 5.743 min, ee = 81.32%; $[\alpha]_D^{20}$ = +12.2 (c = 0.12, THF). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{17}H_{18}N$: 236.1434, found 236.1439.



(2*S*,3*R*)-3-Ethyl-3-methyl-2-phenylindoline (5b)

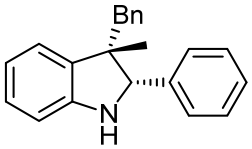
It was prepared following the general procedure B and purified by silica gel flash chromatography using CH_2Cl_2/petroleum ether (1:1) as eluent to afford **5b** (10.9 mg, 46% yield). Yellow solid, m.p. 37-39 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.40–7.26 (m, 5H), 7.09 (t, *J* = 7.6 Hz, 1H), 7.00 (d, *J* = 7.3 Hz, 1H), 6.79 (t, *J* = 7.4 Hz, 1H), 6.73 (d, *J* = 7.7 Hz, 1H), 4.73 (s, 1H), 1.89–1.63 (m, 2H), 1.00 (td, *J* = 7.4, 1.2 Hz, 3H), 0.77 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.1, 141.3, 135.9, 128.3, 127.6, 127.5, 123.5, 118.9, 109.0, 70.3, 49.7, 32.4, 23.7, 9.6. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 272 nm), retention time: t_{major} = 9.067 min, t_{minor} = 5.070 min, ee = 81.22%; $[\alpha]_D^{20}$ = + 87.6 (c = 0.26, THF). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{17}H_{19}N$: 238.1590, found 238.1597. The diastereomer was determined by comparing with reported data.[5]



(*S*)-3-Ethyl-3-methyl-2-phenyl-3H-indole (6b)

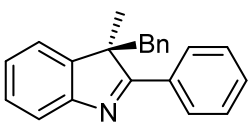
It was prepared following the general procedure B and purified by silica gel flash chromatography using CH_2Cl_2/petroleum ether (1:1) as eluent to afford **6b** (11.1 mg, 47% yield). Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.08–8.02 (m, 2H), 7.62 (d, *J*

= 7.7 Hz, 1H), 7.44–7.39 (m, 3H), 7.29 (ddd, *J* = 7.7, 6.8, 2.0 Hz, 1H), 7.23–7.18 (m, 2H), 2.20 (dq, *J* = 14.7, 7.4 Hz, 1H), 2.06 (dq, *J* = 14.8, 7.4 Hz, 1H), 1.51 (s, 3H), 0.30 (t, *J* = 7.4 Hz, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 182.5, 145.7, 130.8, 128.9, 128.3, 128.0, 126.0, 121.2, 120.9, 59.6, 59.0, 32.2, 24.5, 8.9. HPLC: the ee value was determined by HPLC analysis (Chiralcel OD, *i*-PrOH/Hexane = 10/90, 1.0 mL/min, 331 nm), retention time: t_{major} = 5.773 min, t_{minor} = 4.173 min, ee = 82.22%; $[\alpha]_D^{20}$ = -33.0 (c = 0.20, THF). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{17}H_{18}N$: 236.1434, found 236.1445.



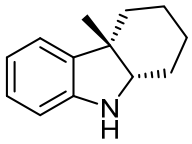
(2*S*,3*S*)-3-Benzyl-3-methyl-2-phenylindoline (5c)

It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (2:8) as eluent to afford **5c** (13.5 mg, 45% yield). White solid, m.p. 84-85 ºC. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.63 (d, *J* = 7.3 Hz, 2H), 7.43 (t, *J* = 7.4 Hz, 2H), 7.37 (dd, *J* = 8.3, 6.3 Hz, 1H), 7.17–7.07 (m, 4H), 6.82 (d, *J* = 7.7 Hz, 1H), 6.67–6.56 (m, 3H), 6.20 (d, *J* = 7.3 Hz, 1H), 4.82 (brs, 1H), 2.72 (d, *J* = 12.8 Hz, 1H), 1.80 (d, *J* = 12.8 Hz, 1H), 1.39 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.7, 139.0, 138.1, 134.9, 131.5, 128.4, 128.1, 127.9, 127.7, 127.3, 126.0, 125.8, 118.2, 109.6, 76.5, 49.0, 41.4, 22.5. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 215 nm), retention time: t_{major} = 15.097 min, t_{minor} = 5.897 min, ee = 81.16%; HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{22}H_{22}N$: 300.1747, found 300.1756. The diastereomer was determined by comparing with reported synthetic method.[5]



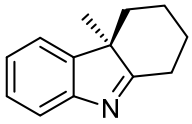
(R)-3-Benzyl-3-methyl-2-phenyl-3H-indole (6c)

It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (2:8) as eluent to afford **6c** (13.7 mg, 46% yield). White solid, m.p. 80-82 ºC. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.13 (dd, *J* = 6.7, 3.0 Hz, 2H), 7.45 (dd, *J* = 8.1, 4.7 Hz, 4H), 7.30–7.21 (m, 1H), 7.19–7.15 (m, 2H), 6.97–6.90 (m, 1H), 6.85 (dd, *J* = 10.2, 4.6 Hz, 2H), 6.49 (d, *J* = 7.2 Hz, 2H), 3.31 (q, *J* = 13.5 Hz, 2H), 1.68 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 181.5, 145.0, 136.1, 130.9, 129.5, 128.9, 128.7, 128.1, 127.7, 126.7, 125.7, 122.3, 121.0, 56.0, 44.7, 24.2. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 215 nm), retention time: t_{major} = 3.680 min, t_{minor} = 4.010 min, ee = 87.84%; HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{22}H_{20}N$: 298.1590, found 298.1584.



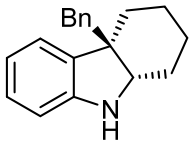
(4a*S*,9a*S*)-4a-Methyl-2,3,4,4a,9,9a-hexahydro-1H-carbazole (5d)

It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (1:9) as eluent to afford **5d** (8.8 mg, 47% yield). Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.09–7.02 (m, 2H), 6.77 (td, *J* = 7.5, 0.9 Hz, 1H), 6.70 (d, *J* = 7.6 Hz, 1H), 3.43 (t, *J* = 4.4 Hz, 1H), 1.75–1.58 (m, 4H), 1.45 (m, 4H), 1.33 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.6, 139.6, 127.1, 121.7, 119.0, 110.3, 66.1, 42.9, 35.2, 27.7, 23.8, 21.7, 21.3. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 3/97, 1.0 mL/min, 215 nm), retention time: t_{major} = 5.473 min, t_{minor} = 4.823 min, ee = 88.32%; $[\alpha]_D^{20}$ = - 26.4 (c = 0.35, THF). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{13}H_{18}N$: 188.1434, found 188.1427. The absolute configuration was assigned as *S* by comparing the optical rotation with reported data.[7] The diastereomer was determined by comparing with reported data.[7]



(*R*)-4a-Methyl-2,3,4,4a-tetrahydro-1H-carbazole (6d)

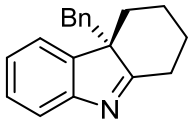
It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (1:9) as eluent to afford **6e** (8.4 mg, 45% yield). Yellow oil. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.60 (d, *J* = 7.7 Hz, 1H), 7.37–7.28 (m, 2H), 7.19 (td, *J* = 7.4, 0.8 Hz, 1H), 2.92–2.83 (m, 1H), 2.59 (td, *J* = 13.3, 5.7 Hz, 1H), 2.23 (ddd, *J* = 25.8, 13.2, 2.6 Hz, 2H), 1.85–1.68 (m, 2H), 1.42 (dt, *J* = 13.3, 4.3 Hz, 1H), 1.31 (s, 3H), 1.22–1.13 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 190.6, 154.2, 147.0, 127.7, 125.1, 121.6, 120.3, 54.0, 38.9, 29.9, 29.2, 21.6, 20.0. HPLC: the ee value was determined by HPLC analysis (Chiralcel OJ, *i*-PrOH/Hexane = 1/99, 1.0 mL/min, 253 nm), retention time: t_{minor} = 8.303 min, t_{major} = 10.370 min, ee = 80.86%; $[\alpha]_D^{20}$ = + 34.2 (c = 0.7, THF). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{13}H_{16}N$: 186.1277, found 186.1261. The absolute configuration was assigned as *R* by comparing the optical rotation and HPLC analysis with reported data.[7]



(4a*R*,9a*S*)-4a-Benzyl-2,3,4,4a,9,9a-hexahydro-1H-carbazole (5e)

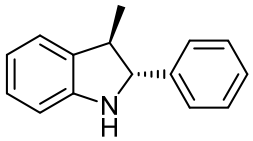
It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (1:9) as eluent to afford **5e** (12.1 mg, 46% yield). Pale brown solid. m.p. 68-69 ºC. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.25–7.17 (m, 3H), 7.07 (td, *J* = 7.5, 1.4 Hz, 1H), 7.00–6.93 (m, 2H), 6.79 (dd, *J* = 7.3, 0.9 Hz, 1H), 6.72 (ddd, *J* = 10.6, 5.8, 2.1 Hz, 2H), 3.46 (dd, *J* = 7.7, 5.5 Hz, 1H), 2.89 (dd, *J* = 40.1, 13.2 Hz, 2H), 1.93–1.83 (m, 1H), 1.79–1.70 (m, 1H), 1.69–1.49 (m, 3H), 1.40–1.18 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 149.9, 138.5, 135.0, 131.1, 127.7, 127.4, 126.2, 123.7, 118.6, 110.8, 63.4, 48.6, 45.0, 32.0, 29.8, 22.1, 22.0. HPLC: the ee value was determined by HPLC analysis (Chiralcel OD, *i*-PrOH/Hexane = 1/99, 1.0 mL/min, 215 nm), retention time: t_{major} = 8.877 min, t_{minor} = 9.953 min, ee =

82.84%; $[\alpha]_D^{20}$ = - 56.4 (c = 0.5, THF). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{19}H_{21}N$: 264.1747, found 264.1742.



(*S*)-4a-Benzyl-2,3,4,4a-tetrahydro-1H-carbazole (6e)

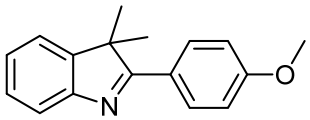
It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (1:9) as eluent to afford **6e** (12.6 mg, 48% yield). Brown oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.48 (d, *J* = 7.7 Hz, 1H), 7.28 (dd, *J* = 7.5, 1.2 Hz, 1H), 7.15 (dd, *J* = 7.7, 7.1 Hz, 1H), 7.10–7.05 (m, 4H), 6.77 (dd, *J* = 7.6, 1.7 Hz, 2H), 3.23 (d, *J* = 13.5 Hz, 1H), 3.00 (t, *J* = 12.1 Hz, 2H), 2.78 (td, *J* = 13.3, 5.7 Hz, 1H), 2.48 (dd, *J* = 13.5, 2.7 Hz, 1H), 2.30 (ddd, *J* = 11.7, 5.0, 2.9 Hz, 1H), 2.03 (dt, *J* = 13.8, 3.7 Hz, 1H), 1.81–1.77 (m, 1H), 1.53–1.45 (m, 1H), 1.23–1.17 (m, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 189.0, 154.6, 144.3, 136.2, 129.6, 128.0, 127.8, 126.8, 124.6, 122.9, 120.2, 58.9, 39.3, 37.3, 30.8, 29.4, 21.6. HPLC: the ee value was determined by HPLC analysis (Chiralcel OD, *i*-PrOH/Hexane = 3/97, 1.0 mL/min, 215 nm), retention time: t_{minor} = 17.190 min, t_{major} = 13.483 min, ee = 85.26%; $[\alpha]_D^{20}$ = + 43.2 (c = 0.35, THF). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{19}H_{20}N$: 262.1590, found 262.1576.



(2*R*,3*R*)-3-Methyl-2-phenylindoline (5f)

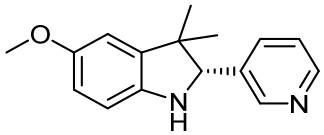
It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (1:9) as eluent to afford **5f** (10.7 mg, 51% yield). Yellow solid. m.p. 41-43 ºC. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.57–7.52 (m, 2H), 7.45–7.33 (m, 3H), 7.18–7.08 (m, 2H), 6.84 (td, *J* = 7.4, 0.9 Hz, 1H), 6.72 (d, *J* = 7.7 Hz, 1H), 4.44 (d, *J* = 9.9 Hz, 1H), 3.28–3.15 (m, 1H), 1.41 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 150.5, 143.5, 133.3, 128.7, 127.8, 127.8,

127.2, 123.4, 119.0, 109.1, 73.0, 46.6, 17.0. HPLC: the ee value was determined by HPLC analysis (Chiralcel OD, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 215 nm), retention time: t_{major} = 10.643 min, t_{minor} = 7.530 min, ee = 85.32%; HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{15}H_{15}N$: 210.1277, found 210.1285. The diastereomer was determined by comparing with reported data.[5]



2-(4-Methoxyphenyl)-3,3-dimethyl-3H-indole (2b)

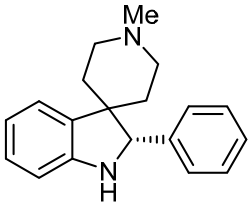
It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (1:9) as eluent to afford **2b** (12.3 mg, 49% yield). Yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.24–8.15 (m, 2H), 7.73 (d, *J* = 7.7 Hz, 1H), 7.41–7.31 (m, 2H), 7.25 (td, *J* = 7.4, 0.9 Hz, 1H), 7.04–6.99 (m, 2H), 3.84 (s, 3H), 1.59 (s, 6H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 182.5 161.5, 153.3, 147.5, 130.1, 127.7, 125.8, 125.4, 120.8, 120.4, 114.0, 55.3, 53.1, 25.0. HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{17}H_{18}NO$: 252.1383, found 252.1395.



(*R*)-5-Methoxy-3,3-dimethyl-2-(pyridin-3-yl)indoline (8)

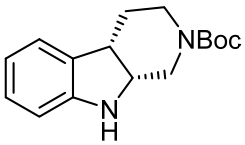
It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (1:9) as eluent to afford **8** (12.5 mg, 49% yield). Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 (d, *J* = 8.5 Hz, 2H), 6.90 (m, 4H), 6.64 (d, *J* = 7.7 Hz, 1H), 4.53 (s, 1H), 3.83 (s, 3H), 2.31 (s, 3H), 1.40 (s, 3H), 0.74 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 159.2, 147.0, 138.7, 132.2, 128.7, 128.5, 127.8, 123.5, 113.6, 109.3, 74.5, 55.5, 45.4, 26.5, 24.6, 21.2. HPLC: the ee value was determined by HPLC analysis (Chiralcel IB, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 215 nm), retention time: t_{major} = 24.073 min, t_{minor} = 13.037 min, ee =

89.52%; $[\alpha]_D^{20}$ = + 11.62 (c = 0.14, CH_2Cl_2). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{16}H_{19}N_2O$: 255.1492, found 255.1497.



(*S*)-1'-Methyl-2-phenylspiro[indoline-3,4'-piperidine] (10)

It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (2:1) as eluent to afford **10** (14.0 mg, 50% yield). Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31–7.26 (m, 6H), 7.11 (td, *J* = 7.6, 1.1 Hz, 1H), 6.78 (td, *J* = 7.4, 0.8 Hz, 1H), 6.69 (d, *J* = 7.7 Hz, 1H), 4.61 (s, 1H), 4.12 (s, 1H), 2.87–2.80 (m, 1H), 2.69–2.62 (m, 1H), 2.55–2.48 (m, 1H), 2.35 (d, *J* = 4.4 Hz, 3H), 2.12–2.05 (m, 2H), 2.01–1.94 (m, 1H), 1.84 (d, *J* = 8.4 Hz, 1H), 1.48–1.41 (m, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 150.4, 140.9, 135.8, 128.5, 128.1, 128.0, 124.4, 118.8, 108.9, 72.5, 52.7, 52.4, 46.8, 46.4, 36.8, 31.2. HPLC: the ee value was determined by HPLC analysis (Chiralcel OD, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 215 nm), retention time: t_{major} = 14.353 min, t_{minor} = 10.413 min, ee = 82.14%; HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{19}H_{23}N_2$: 279.1856, found 279.1859.

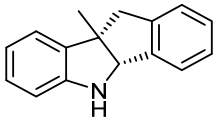


Tert-butyl

(4aS,9aR)-1,3,4,4a,9,9a-hexahydro-2H-pyrido[3,4-b]indole-2-carboxylate (5g)

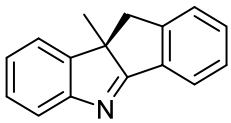
It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (1:1) as eluent to afford **5g** (12.9 mg, 46% yield). Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.04 (dd, *J* = 16.5, 8.2 Hz, 2H), 6.73 (t, *J* = 6.9 Hz, 1H), 6.62 (d, *J* = 7.7 Hz, 1H), 3.95 (s, 1H), 3.56–3.27 (m, 5H), 2.06–1.95 (m, 1H), 1.86 (s, 1H), 1.44 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ

155.8, 150.8, 131.2, 127.9, 123.8, 118.9, 109.8, 79.6, 57.6, 43.7, 41.4, 39.4, 28.6, 26.3. HPLC: the ee value was determined by HPLC analysis (Chiralcel IA, *i*-PrOH/Hexane = 1/99, 1.0 mL/min, 254 nm), retention time: t_{major} = 53.723 min, t_{minor} = 73.427 min, ee = 73.04%; HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{16}H_{23}N_2O_2$: 275.1754, found 275.1747. The diastereomer and absolute configuration was adetermined by comparing with reported data.[9]



(4bR,9bS)-9b-Methyl-4b,5,9b,10-tetrahydroindeno[1,2-b]indole (11)

It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (2:1) as eluent to afford **11** (10.9 mg, 49% yield). Colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.31 (d, *J* = 6.6 Hz, 1H), 7.23–7.13 (m, 4H), 7.00 (td, *J* = 7.6, 1.2 Hz, 1H), 6.75 (td, *J* = 7.4, 0.8 Hz, 1H), 6.61 (d, *J* = 7.8 Hz, 1H), 4.81 (s, 1H), 4.28 (brs, 1H), 3.39 (d, *J* = 16.2 Hz, 1H), 3.20 (d, *J* = 16.2 Hz, 1H), 1.55 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 149.4, 144.5, 142.8, 137.6, 128.2, 128.0, 127.3, 125.1, 124.2, 123.3, 119.6, 110.6, 74.7, 53.8, 47.0, 27.0. HPLC: the ee value was determined by HPLC analysis (Chiralcel OD, *i*-PrOH/Hexane = 10/90, 1.0 mL/min, 254 nm), retention time: t_{major} = 8.497 min, t_{minor} = 7.223 min, ee = 89.48%; $[\alpha]_D^{20}$ = - 1.6 (c = 0.08, $CHCl_3$). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{16}H_{16}N$: 222.1277, found 222.1269. The diastereomer was determined by comparing with reported data.[8]



(R)-9b-Methyl-9b,10-dihydroindeno[1,2-b]indole (12)

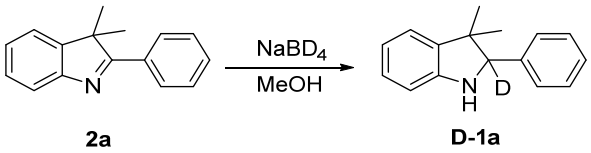
It was prepared following the general procedure B and purified by silica gel flash chromatography using ethyl acetate/petroleum ether (2:1) as eluent to afford **12** (10.1 mg, 46% yield). Yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.91 (d, *J* = 5.1 Hz, 1H), 7.65 (d, *J* = 7.7 Hz, 1H), 7.46–7.40 (m, 4H), 7.38–7.33 (m, 1H), 7.21 (t, *J* = 7.4 Hz,

1H), 3.12 (d, *J* = 14.5 Hz, 1H), 2.85 (d, *J* = 14.6 Hz, 1H), 1.40 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 196.4, 153.6, 144.3, 131.2, 128.4, 128.1, 127.2, 125.3, 124.0, 123.2, 121.6, 64.0, 39.0, 26.9. HPLC: the ee value was determined by HPLC analysis (Chiralcel OJ, *i*-PrOH/Hexane = 5/95, 1.0 mL/min, 311 nm), retention time: t_{major} = 28.283 min, t_{minor} = 24.640 min, ee = 85.70%; $[\alpha]_D^{20}$ = + 17.6 (c = 0.10, $CHCl_3$). HRMS (EI) *m/z* $[M + H]^+$ calculated for $C_{16}H_{14}N$: 220.1121, found 220.1127.

Mechanism studies

Kinetic isotope effect experiment

Scheme S4. Preparation of [D]-1a

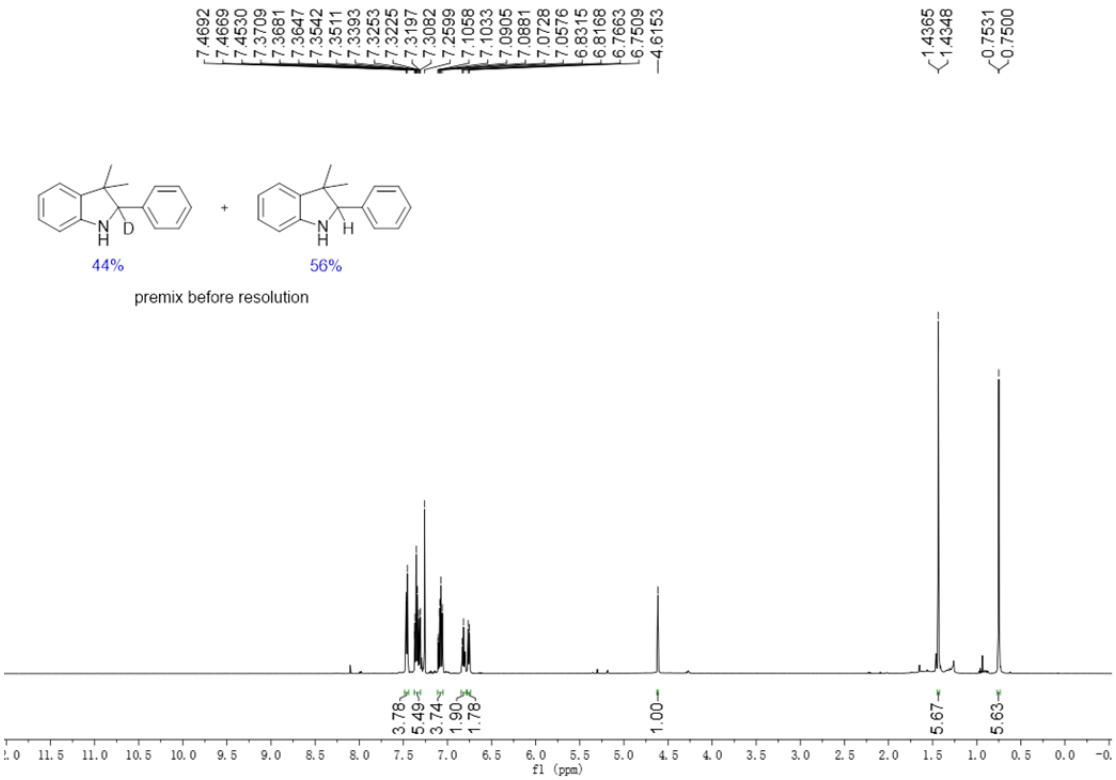


2a **D-1a**

[D]-1a was prepared through the reduction of **2a** by $NaBD_4$. In a 100 mL round bottom flask, **2a** was dissolved in MeOH (0.2 M) and the reaction mixture was cooled to 0 °C. $NaBD_4$ (1.2 equiv) was added and the reaction mixture was allowed to warm to room temperatue and stirred 5 h. The reaction mixture was concentrated by rotary evaporation under reduced pressure partitioned between DCM and water (20 mL each). The organic layer was removed and the aqueous layer extracted with DCM (2 x 20 mL). The combined organic layers were dried with $MgSO_4$, filtered, concentrated and purified by flash column chromatography, furnishing [**D]-1a** containing 6% of non-deuterated **1a**. The analytical data was as follows: ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.54–7.46 (m, 2H), 7.43–7.33 (m, 3H), 7.16–7.09 (m, 2H), 6.84 (td, J = 7.4, 0.9 Hz, 1H), 6.76 (d, J = 7.7 Hz, 1H), 1.48 (s, 3H), 0.79 (s, 3H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 149.5, 140.1, 138.2, 128.3, 127.7, 127.6, 127.6, 122.7, 119.2, 109.4, 74.2 (t, J = 21.2 Hz), 45.4, 26.7, 24.7.

The kinetic isotope effect experiment was conducted at 0.05 mmol scale following the general procedure using a mixture of **H-1a** and **D-1a** (11.2 mg, 44% D). It was stirred at -40 °C for 1 h and purified by silica gel flash chromatography (7 mg, 62.5% yield). The ratio of **H-1a** and **D-1a** was determined by ^{1}H NMR which shows 54% of the remaining product was **D-1a**. The KIE was calculated as follows:

$$\mathrm{KIE} = \frac{K_H}{K_D} = \frac{\frac{C_{H0} - C_{Ht}}{t}}{\frac{C_{D0} - C_{Dt}}{t}} = \frac{C_{H0} - C_{Ht}}{C_{D0} - C_{Dt}} = \frac{\frac{m_{H0} - m_{Ht}}{V}}{\frac{m_{D0} - m_{Dt}}{V}} = \frac{m_{H0} - m_{Ht}}{m_{D0} - m_{Dt}} = \frac{11.2*0.56 - 7*0.46}{11.2*0.44 - 7*0.54} = 2.7$$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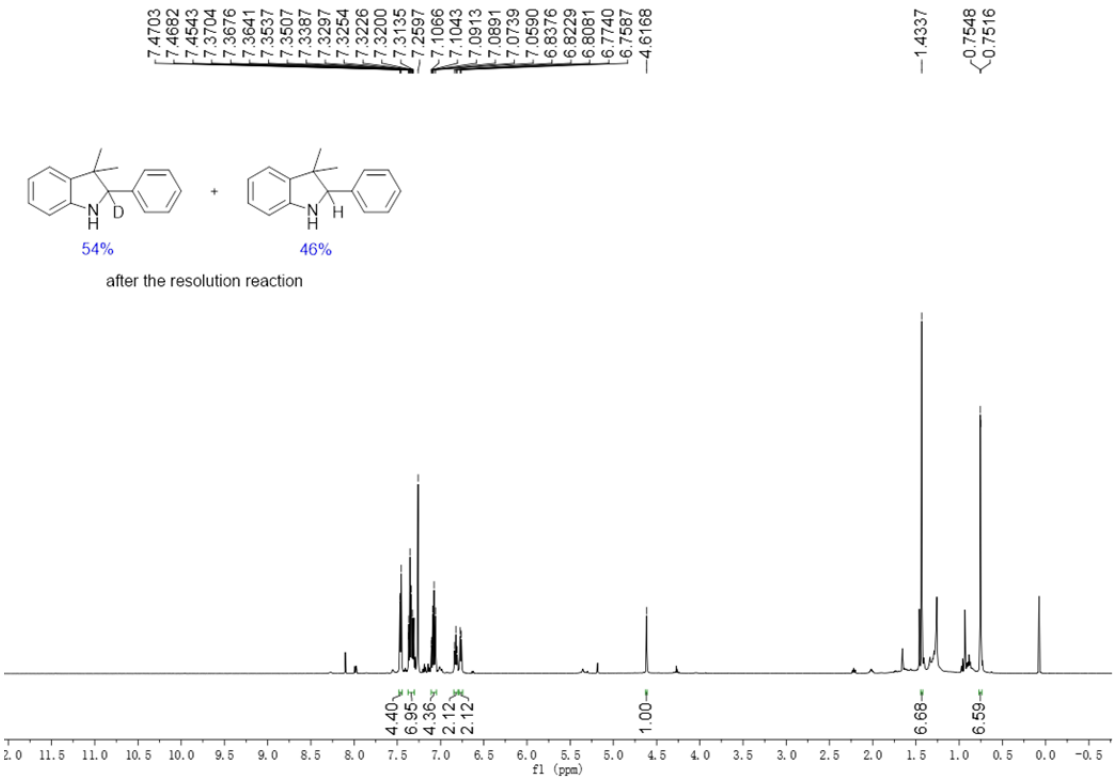
7.4530
7.3709
7.3681
7.3647
7.3542
7.3511
7.3393
7.3253
7.3225
7.3197
7.3082
7.2599
7.1058
7.1033
7.0905
7.0881
7.0728
7.0576
6.8315
6.8168
6.7663
6.7509
4.6153
1.4365
1.4348
0.7531
0.7500
N H D
N H H
44%
56%
premix before resolution
3.78
5.49
3.74
1.90
1.78
1.00
5.67
5.63
f1 (ppm)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7.3704
7.3676
7.3641
7.3537
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7.3226
7.3200
7.3135
7.2597
7.1066
7.1043
7.0913
7.0891
7.0739
7.0590
6.8376
6.8229
6.8081
6.7740
6.7587
4.6168
1.4337
0.7548
0.7516
N H D
N H H
54%
46%
after the resolution reaction
4.40
6.95
4.36
2.12
2.12
1.00
6.68
6.59
f1 (ppm)

Correlation of the enantiomeric excess of C_{mono}8 and 1a with sodium 6-methoxy-2-naphthoate additive

The CH_2Cl_2 solutions of **C_{mono}8** and ent-**C_{mono}8** (0.005 M, respectively) were prepared and mixed to regulate each (0% ee, 20% ee, 40% ee, 60% ee, 80% ee and 100% ee, 0.005 M, respectively) complex solution in an appropriate manner. To the solutions at -40 °C, **1a** (0.1 mmol, 22.3 mg) and sodium 6-methoxy-2-naphthoate (0.01 mmol, 2.2 mg, 10 mmol%) was added. Then 30% aqueous hydrogen peroxide (0.1 mmol, 10 μL) were added as 4 portions in 2-hours intervals. After stirring for 8 h at this temperature, the reaction mixture was diluted with CH_2Cl_2 (20 mL) at 50% conversion of **1a**, washed with water (10 mL), dried over $MgSO_4$, filtered and concentrated. The residue was purified by silica gel flash chromatography using EtOAc/petroleum ether (10:90) as eluent. The ee values of **1a** were determined by HPLC analysis on chiral phase column (Chiralpak IB-H, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 296 nm). A negative nonlinear effect was observed.

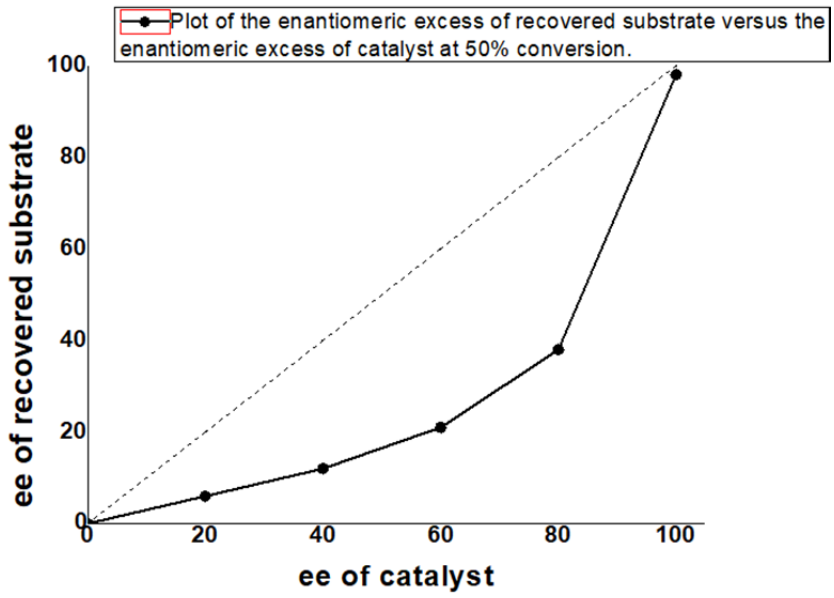


Figure S1. Plot of the ee of recovered **1a** versus the ee of **C_{mono}8** at 50% conversion. The dotted line symbolizes the linear correlation.

Correlation of the enantiomeric excess of C_{mono}8 and 1a without sodium 6-methoxy-2-naphthoate additive

The CH_2Cl_2 solutions of **C_{mono}8** and ent-**C_{mono}8** (0.005 M, respectively) were prepared and mixed to regulate each (0% ee, 20% ee, 40% ee, 60% ee, 80% ee and 100% ee, 0.005 M, respectively) complex solution in an appropriate manner. To the solutions at -40 °C, **1a** (0.1 mmol, 22.3 mg) was added. Then 30% aqueous hydrogen peroxide (0.1 mmol, 10 μL) were added as 4 portions in 2-hours intervals. After stirring for 8 h at this temperature, the reaction mixture was diluted with CH_2Cl_2 (20 mL) at 50% conversion of **1a**, washed with water (10 mL), dried over $MgSO_4$, filtered and concentrated. The residue was purified by silica gel flash chromatography using EtOAc/petroleum ether (10:90) as eluent. The ee values of **1a** were determined by HPLC analysis on chiral phase column (Chiralpak IB-H, *i*-PrOH/Hexane = 20/80, 1.0 mL/min, 296 nm). An approximate linear effect was observed.

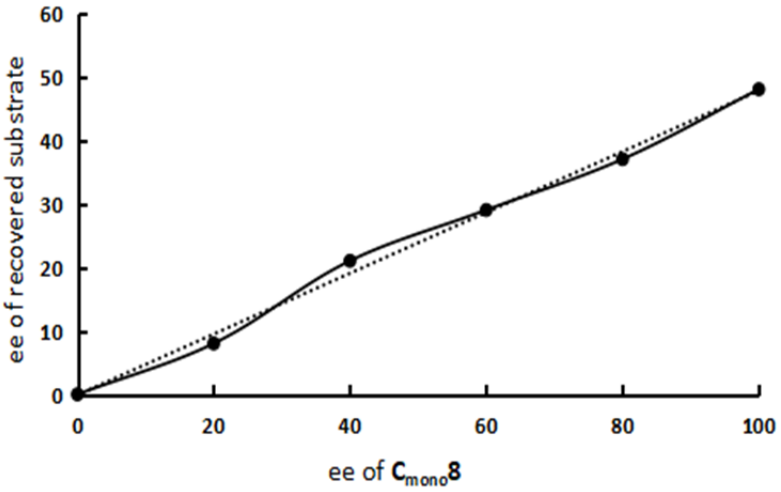
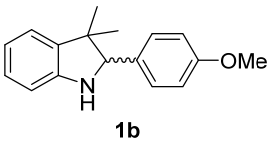


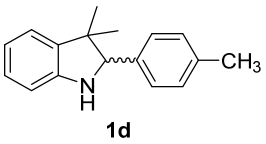
Figure S2. Plot of the ee of recovered **1a** versus the ee of **C_{mono}8** at 50% conversion without additive. The dotted line symbolizes the linear correlation.

Hammett polt for the competitive dehydrogenation experiments of substrates with a series of p-substituents (X) on α-aryl groups

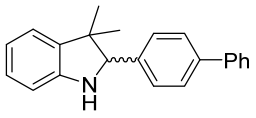
To a solution of a mixture two different *p*-substituted **1** (**1a** and **1b**; **1a** and **1d**; **1a** and **1f**; **1a** and **1i**; **1a** and **1n**; 0.1 mmol each) in $CHCl_3$ (1.0 mL), $\mathbf{C_{mono}8}$ (0.005 mmol, 3.7 mg, 5 mmol%) and sodium 6-methoxy-2-naphthoate (0.01 mmol, 2.2 mg, 10 mmol%) was added at -40 °C. Then 30% aqueous hydrogen peroxide (0.1 mmol, 10 μL, 1.0 eq) was added as 4 portions in 4-hours intervals and the reaction was quenched with water (10 mL) at 15-35% conversion and the mixture was extracted with CH_2Cl_2 (20 mL). The solvent was removed and the residue was purified by silica gel chromatography to give the desired product. The results were summarized as follows:.



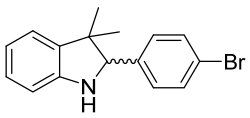
1b



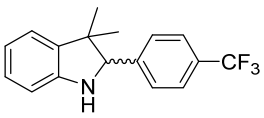
1d



1f



1i



1n

entry	*p*-substituted X	$\log(k_X/k_H)$ [a]	σ [b]	σ^+ [b]
1	OCH_3	0.385	-0.27	-0.78
2	CH_3	0.057	-0.14	-0.31
3	Ph	-0.042	0.05	-0.18
4	Br	-0.332	0.26	0.15
5	CF_3	-0.943	0.53	

[a]Average of three experiments at 15-35% conversion.

[b]Data from: Anslyn, E. V.; Dougherty, D. A. (**2006**). *Modern Physical Organic Chemistry*, University science books.

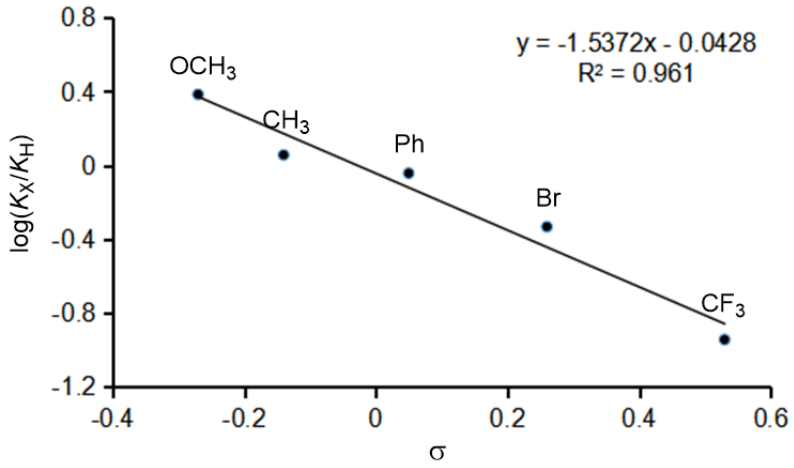


Figure S3. Hammett Plot of $\log(k_X/k_H)$ vs. σ for the competition experiments.

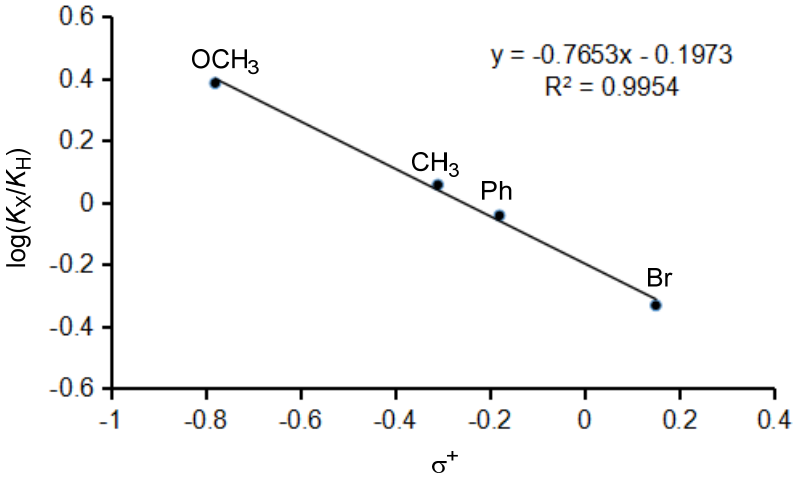
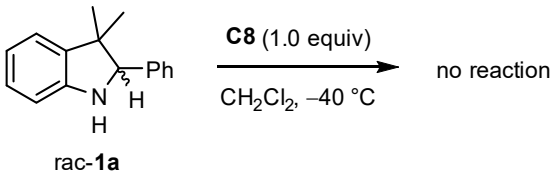


Figure S4. Hammett Plot of $\log(k_X/k_H)$ vs. σ^+ for the competition experiments

Control experiments

The oxidation reactivity of stoichiometric C8 without H_2O_2

Scheme S5. Control experiment using stoichiometric C8 without H_2O_2



To a solution of rac-**1a** (0.05 mmol, 11.2 mg) in CH_2Cl_2 (0.5 mL) was added **C8** (0.05 mmol, 77 mg) at -40 °C. The mixture was vigorously stirred for 12 h. No reaction occurred.

Resonance Raman spectroscopy

Resonance Raman spectra were measured with glass capillary tubes containing the complexes, maintained at room temperature, using a LabRAM HR Evolution raman spectrometer(HORIBA Scientific). An Helium-neon gas laser at 633nm was utilized as an excitation source. The laser power at the sample was about 5 mW and the acquisition time was 15s.

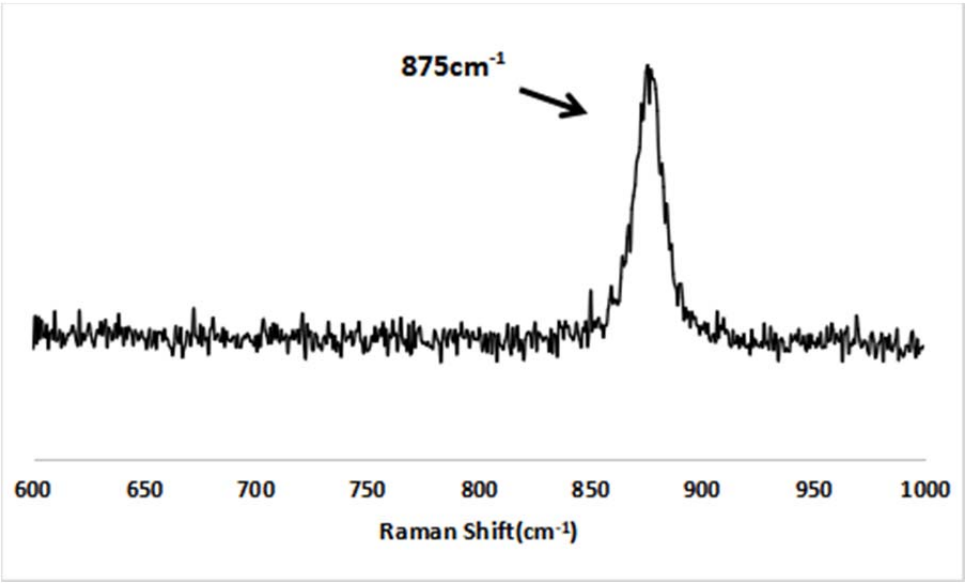


Figure S5. Raman spectrum of H_2O_2

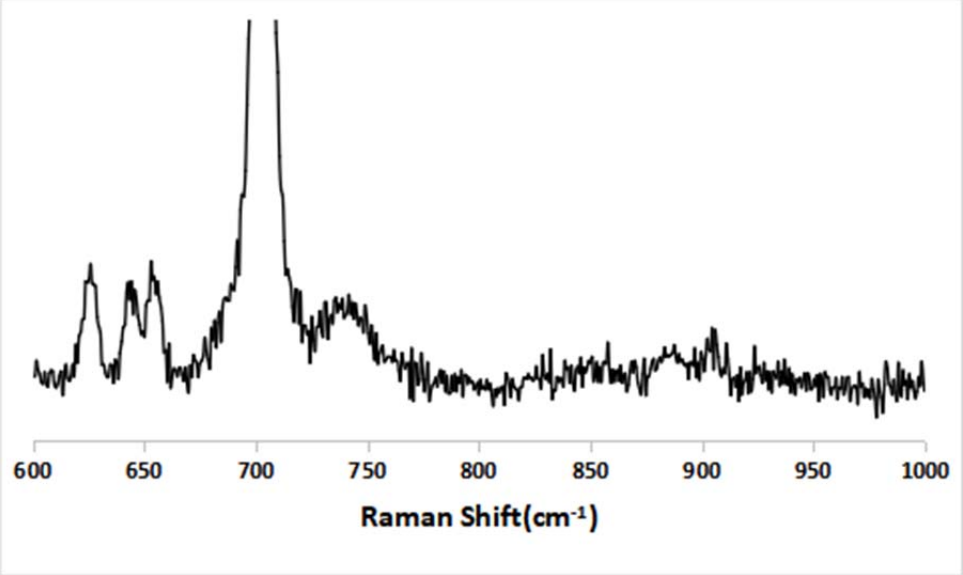


Figure S6. Raman spectrum of **C8** without H_2O_2

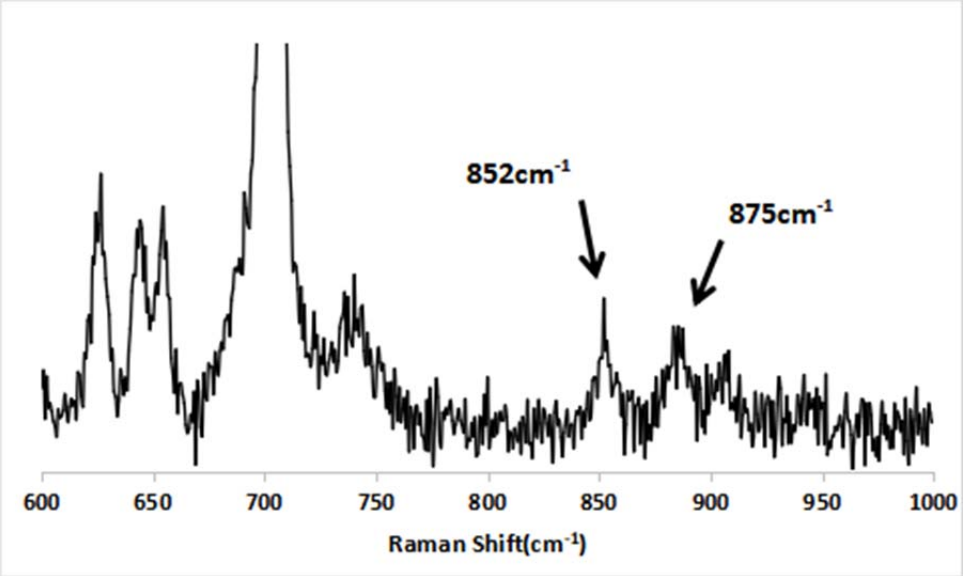
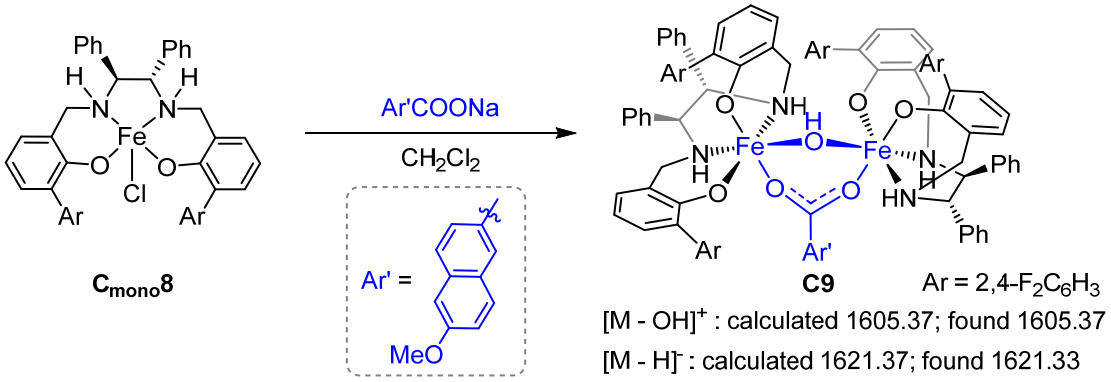
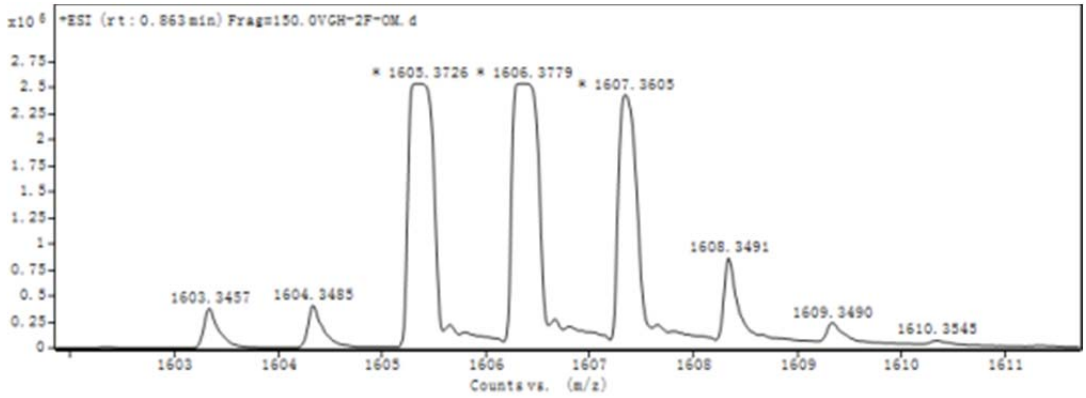


Figure S7. Raman spectrum of **C8** combining with 10 equiv of H_2O_2

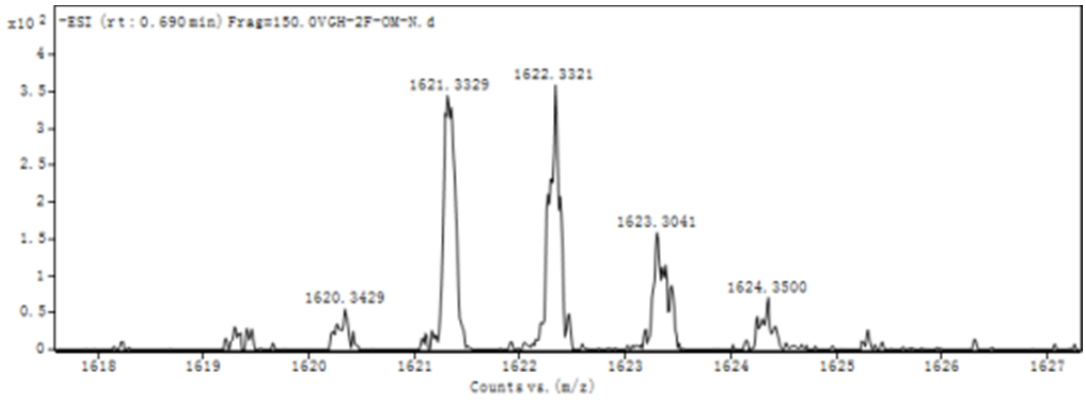
ESI-MS analysis

ESI-MS analysis was conducted to confirm the structure of **C9** and **13**. The isotope distribution patterns were calculated by EnviPat Web 2.4 site to compare the pattern and profile of the *m/z* peak(s) to the found ones. For complex **C9**, ESI-MS *m/z* $[M - OH]^+$ calculated for $C_{92}H_{69}F_8Fe_2N_4O_7$: 1605.37, found 1605.37; *m/z* $[M - H]^-$ calculated for $C_{92}H_{69}F_8Fe_2N_4O_8$: 1621.37, found 1621.33. For complex **13**, ESI-MS *m/z* $[M + H]^+$ calculated for $C_{87}H_{69}F_8Fe_2N_4O_8$: 1561.37, found 1561.36; The isotope distribution patterns of **C9** and **13** are identical to the calculated ones.

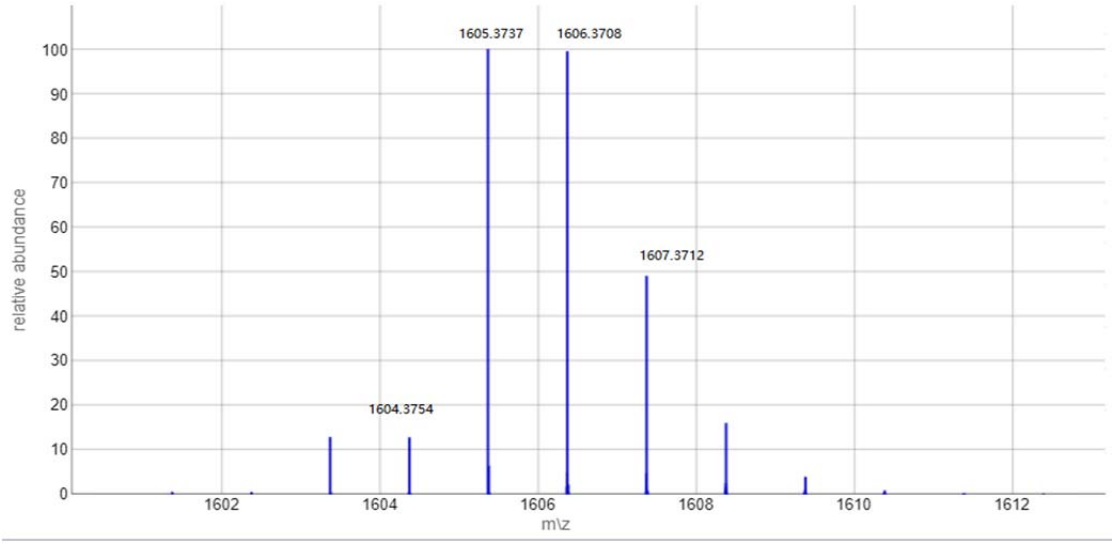




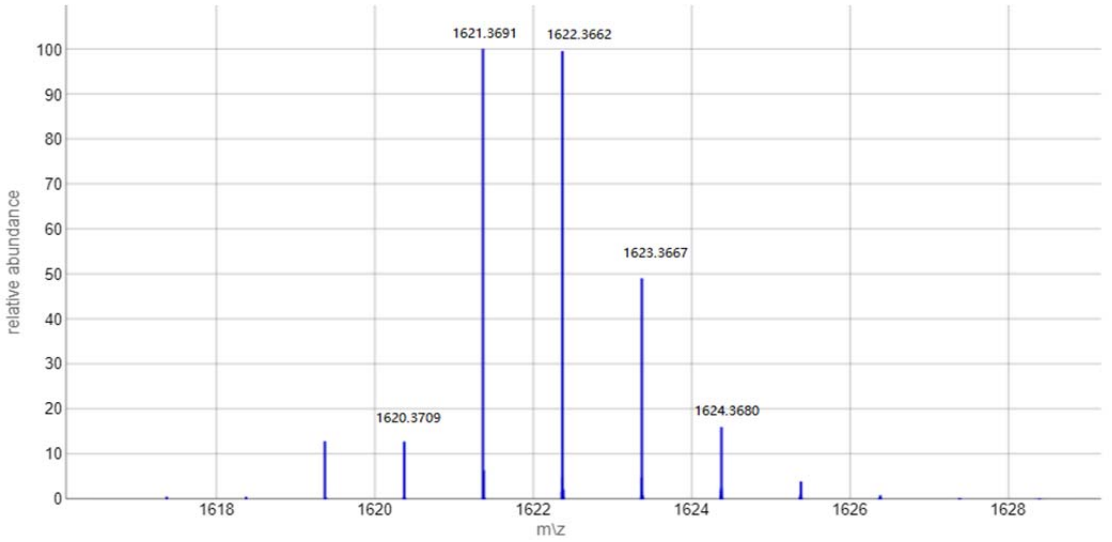
ESI-MS $[M - OH]^+$ for **C9**



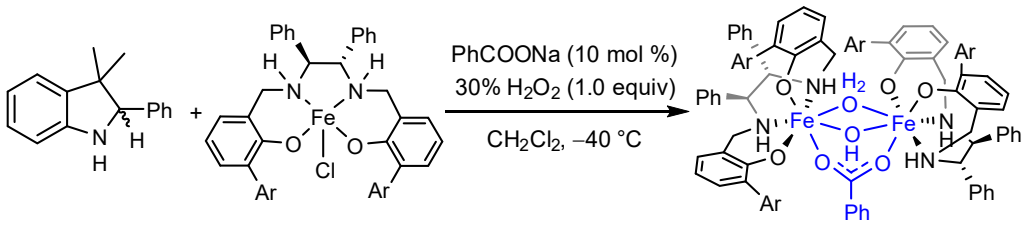
ESI-MS $[M - H]^-$ for **C9**



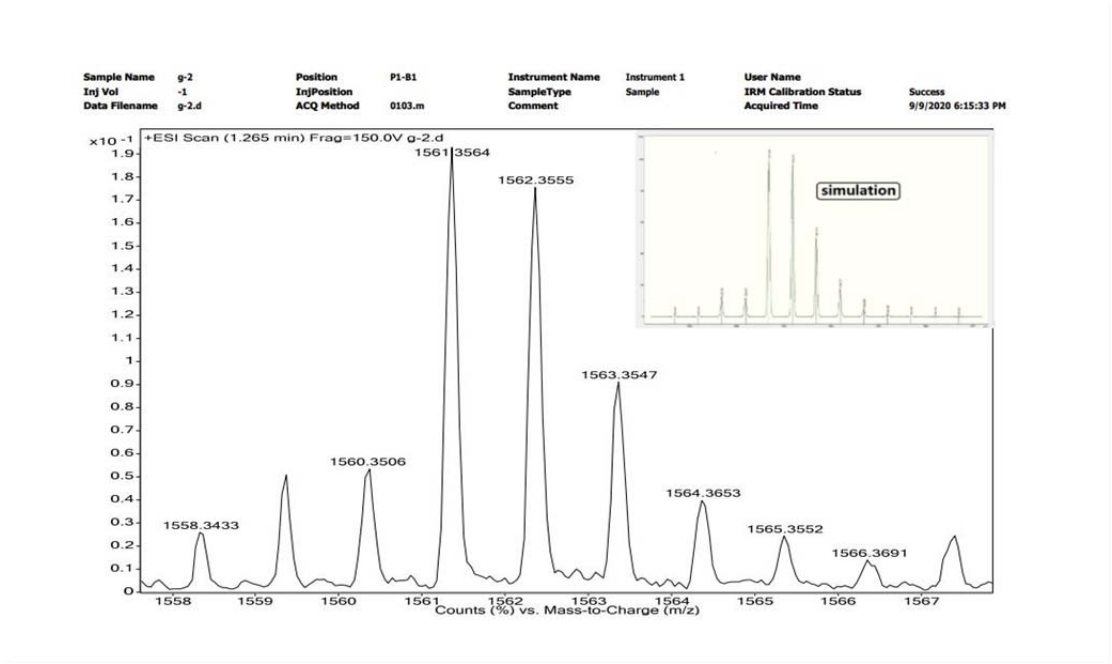
Simulation of ESI-MS $[M - OH]^+$ for **C9**



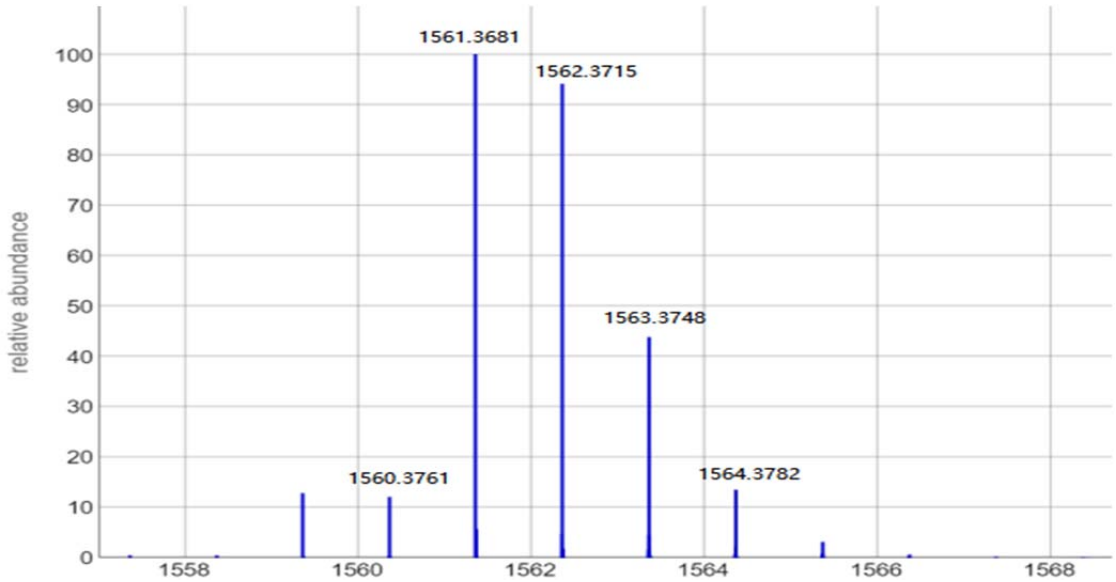
Simulation of ESI-MS $[M - H]^-$ for **C9**



$[M + H]^+$: calculated 1561.37
found 1561.36



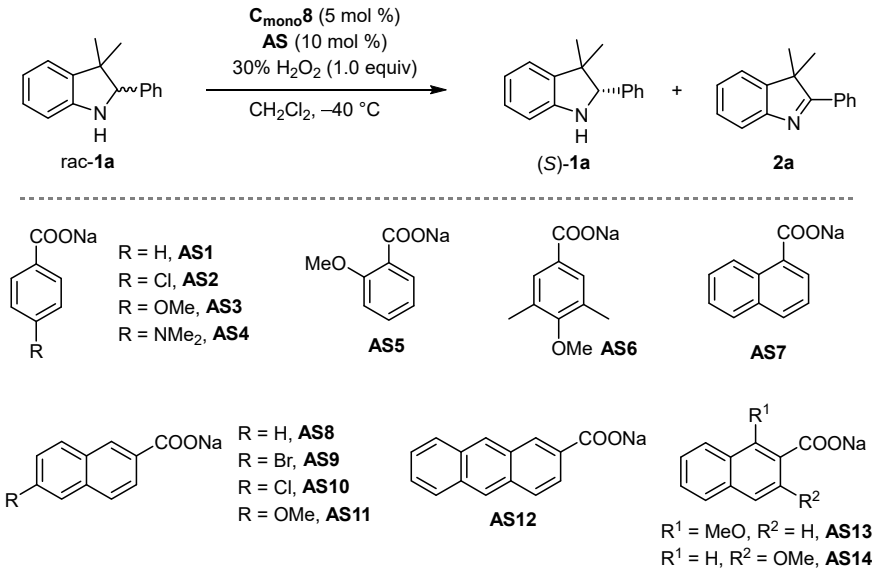
ESI-MS $[M + H]^+$ for **13**



Simulation of ESI-MS $[M + H]^+$ for **13**

Influence of various additives on selectivity

Table S2. The effect of different aryl carboxylic acid derivatives[a]



entry	acid derivative	conv. (%)[b]	ee (%)[c]	s[d]
1	**AS1**	49	71	14
2	**AS2**	52	61	8.3
3	**AS3**	53	83	17
4	**AS4**	51	82	21
5	**AS5**	52	67	8.3
6	**AS6**	50	65	9.1
7	**AS7**	47	49	5.5
8	**AS8**	50	87	41
9	**AS9**	51	94	70
10	**AS10**	52	85	22
11	**AS11**	50	94	115
12	**AS12**	50	70	12
13	**AS13**	53	95	43
14	**AS14**	49	67	11

[a]Reaction condition: to rac-**1a** (0.1 mmol), monoiron $\mathbf{C_{mono}8}$ (5 mol%) and carboxylic acid derivative (10 mol %) in CH_2Cl_2 (1.0 mL) at -40 ºC was added 30% aqueous H_2O_2 (0.1 mmol) as four portions in 2 h intervals for 6 h, and the mixture was stirred at -40 ºC for 18-24 h, unless otherwise noted.[b]Conversion was calculated from the isolated yield of recovered (S)-1a. [c]Determined by HPLC analysis on a chiral stationary phase. [d]Selectivity (s) values were calculated through the equation s = ln[(1 − C)(1 − ee)]/ln[(1 − C)(1 + ee)].

The effect of aryl carboxylic acid derivatives on the selectivity are systematically examined (Table S2). We found that the selectivity was highly dependent on the nature and the position of the substituents on the aryl ring of the additive. In general, aryl carboxylic acids bearing an electron-withdrawing group show inferior selectivity to those with electron-donating ones (e.g. entries 1-4; entries 8-11, Table S2). The observation suggested that the chelating properties of carboxylic acid moiety are essential to selectivity. The selectivity was also sensitive to the position of the substituents on the arene ring of the additive. While no obvious trend on the substituent pattern was concluded, the obvious variation on the selectivity implied that the substituent pattern on the arene ring might influence the chiral environment around the diiron through modulating the 2-benzoate-bridge.

X-ray crystallographic data

Single crystals of **C2 and C8** were prepared as follows:

$\mathbf{C_{mono}2}$ or $\mathbf{C_{mono}8}$ (0.05 mmol, 1.0 equiv) were dissolved in a mixture of CH_2Cl_2-EtOH-acetone-H_2O (3 mL/ 3 mL/3 mL/ 1 drop) solution and sodium benzoate (20 equiv) was added. The mixure was maintained open-flask at room temperature for several days until the crystal formed.

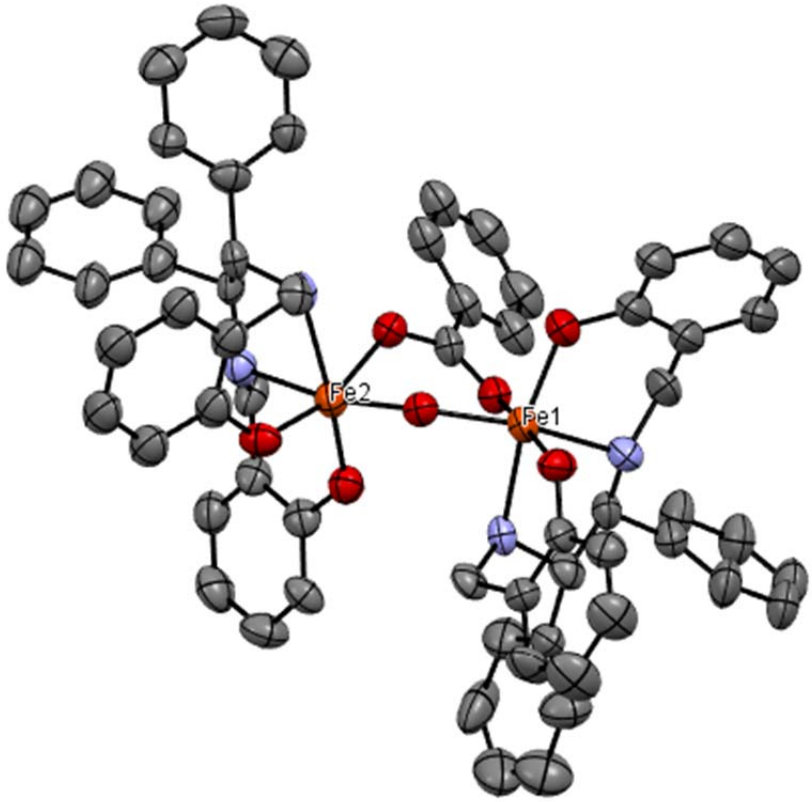


Figure S8. Molecular structure of complex **C2** (CCDC 2127388)

Crystal data and structure refinement for mo_211115G_2_0m.

Identification code	mo_211115G_2_0m
Empirical formula	$C_{67}H_{69}Fe_2N_4O_9$
Formula weight	1185.96
Temperature/K	173.0
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	11.486(2)
b/Å	19.300(3)
c/Å	28.572(5)
α/°	90
β/°	90
γ/°	90
Volume/Å^3	6334(2)
Z	4
ρ_{cal}cg/cm^3	1.244
μ/mm^{-1}	0.516
F(000)	2492.0
Crystal size/mm^3	0.15 × 0.02 × 0.02
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	3.822 to 52.878
Index ranges	$-14 \leq h \leq 14$, $-24 \leq k \leq 19$, $-32 \leq l \leq 35$
Reflections collected	29957
Independent reflections	12758 [R_{int} = 0.1522, R_{sigma} = 0.1959]
Data/restraints/parameters	12758/192/743
Goodness-of-fit on F^2	0.949
Final R indexes [I>=2σ (I)]	R_1 = 0.0857, wR_2 = 0.2026
Final R indexes [all data]	R_1 = 0.1757, wR_2 = 0.2616
Largest diff. peak/hole / e Å^{-3}	0.67/-0.77
Flack parameter	0.07(3)

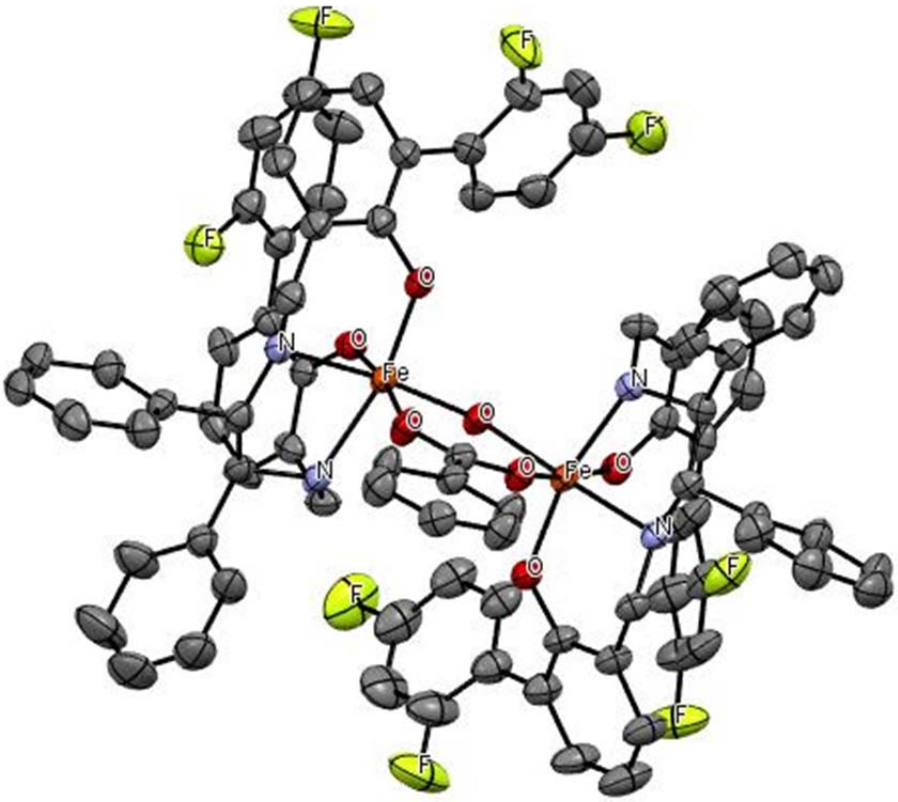


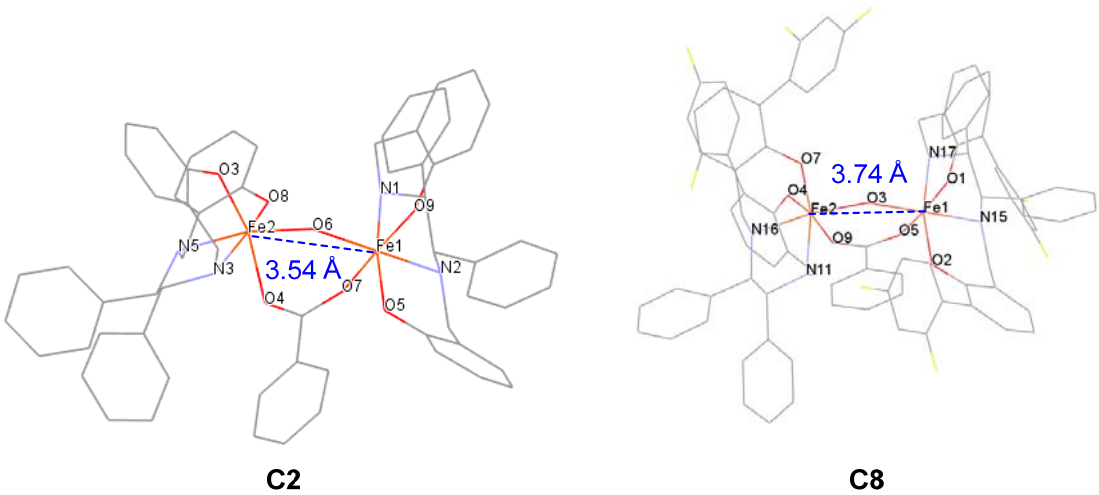
Figure S9. Molecular structure of complex **C8** (CCDC 2127389)

Compound	200828c
Formula	$C_{180}H_{148}Cl_4F_{16}Fe_4N_8O_{16}$
$D_{calc.}$/ g cm^{-3}	1.392
μ/mm^{-1}	4.193
Formula Weight	3348.26
Colour	clear light black
Shape	block
Size/mm^3	0.03 × 0.02 × 0.01
T/K	173.00(10)
Crystal System	orthorhombic
Flack Parameter	-0.009(2)
Hooft Parameter	-0.0065(17)
Space Group	$P2_12_12_1$
a/Å	15.2213(3)
b/Å	21.4654(5)
c/Å	24.4451(4)
α/°	90
β/°	90
γ/°	90
V/Å^3	7987.0(3)
Z	2
Z′	0.5
Wavelength/?	1.54184
Radiation type	Cu Kα
Θ_{min}/°	2.740
Θ_{max}/°	67.079
Measured Refl.	27176
Independent Refl.	13233
Reflections with I > 2(I)	11427
R_{int}	0.0407
Parameters	1029
Restraints	0
Largest Peak	0.433
Deepest Hole	-0.819
GooF	1.041
wR_2 (all data)	0.1538
wR_2	0.1454
R_1 (all data)	0.0667
R_1	0.0555

Selected key features in the crystal structures

X-ray diffraction studies revealed that complex **C2** and **C8** were dinuclear complexes mimiking the structure of the μ-hydroxo, carboxylate bridged non-heme diiron(III) core in the active site of MMO. A comparsion of the molecular structures of complexes **C2**, **C8** and MMO based on their X-ray crystallographic data was shown here.

The Fe–Fe distances in MMO, **C2**, and **C8** are 3.1 Å, 3.54 Å, and 3.74 Å, respectively. **C8** containing a bulkier salan basal ligand exhibits a longer Fe–Fe bond than **C2**, suggesting that varying the substituent on the basal salan ligand leads to an obvious change of the Fe–Fe bond length. Based on the selectivity difference of complex **C2** and **C8**, we persumed that the Fe–Fe bond distance in chiral diiron(III) dimer complexes might be crucial to the enantioselectivity.



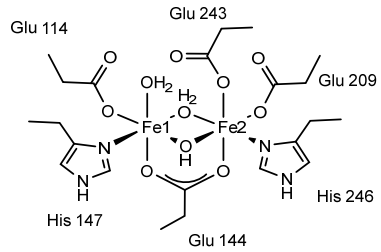
C2 **C8**

Figure S10. Stick figure of the X-ray crystal structure of **C2** and **C8**

Table S3. Selected bond distances (Å) of C2 and C8:

C2 Bond distances		**C8** Bond distances	
Atom-Atom	Bond distance (Å)	Atom-Atom	Bond distance (Å)
Fe(1)-Fe(2)	3.54	Fe(1)-Fe(2)	3.74
Fe(1)-O(5)	1.91	Fe(1)-O(2)	1.93
Fe(1)-O(7)	1.96	Fe(1)-O(5)	1.91
Fe(1)-O(6)	2.12	Fe(1)-O(3)	1.99
Fe(1)-O(9)	1.89	Fe(1)-O(1)	1.91
Fe(1)-N(1)	2.15	Fe(1)-N(17)	2.18
Fe(1)-N(2)	2.21	Fe(1)-N(15)	2.20
Fe(2)-O(3)	1.94	Fe(2)-O(4)	1.90
Fe(2)-O(6)	1.95	Fe(2)-O(3)	1.98
Fe(2)-O(8)	1.89	Fe(2)-O(7)	1.94
Fe(2)-O(4)	2.07	Fe(2)-O(9)	2.06
Fe(2)-N(3)	2.16	Fe(2)-N(11)	2.20
Fe(2)-N(4)	2.18	Fe(2)-N(16)	2.20

Table S4. Some key interatomic distances (Å) in $sMMO_{ox}$ according to known literature[12].



Atom	Atom	Distance (Å)
Fe1	Fe2	3.1
Fe1	Glu 114 O	1.9
Fe1	His 147 N	2.1
Fe1	Glu 144 O	2.1
Fe1	μOH O	1.7
Fe1	OH_2 O	2.3
Fe1	μOH_2 O	2.3
Fe2	Glu 209 O	1.9
Fe2	His 246 N	2.2
Fe2	Glu 243 O	2.0
Fe2	Glu 144 O	2.5
Fe2	μOH O	2.0
Fe2	OH_2 O	2.5

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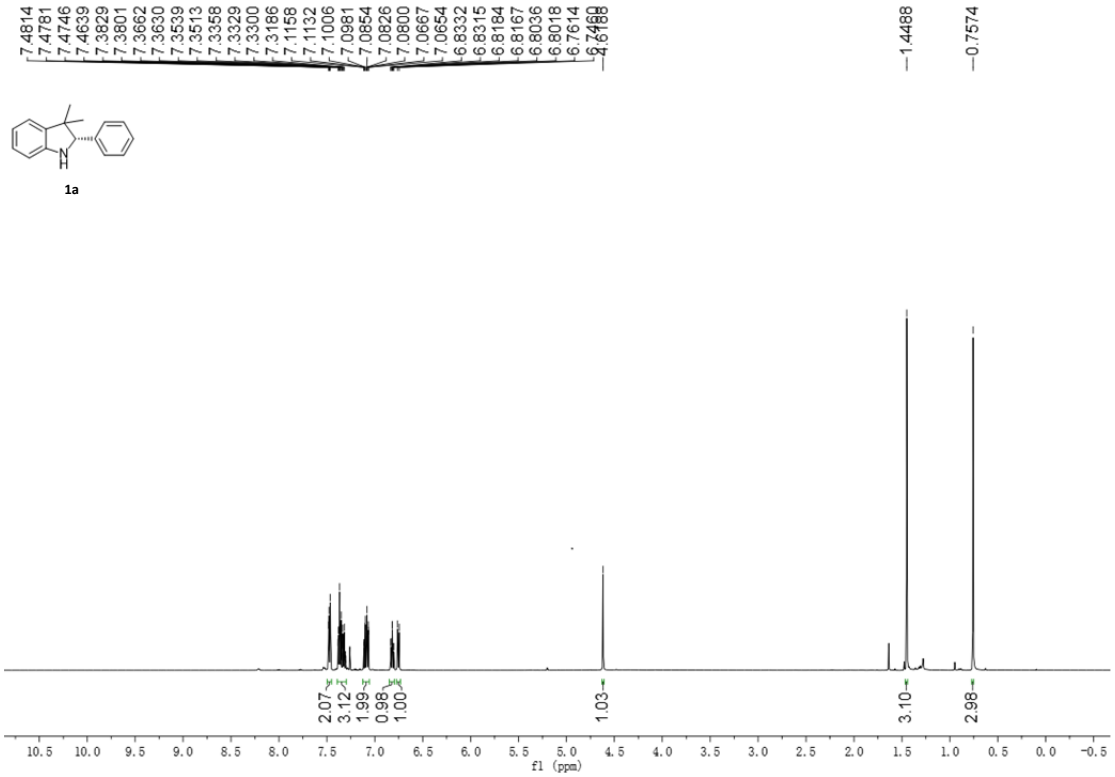
NMR spectra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7.3300
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6.8036
6.8018
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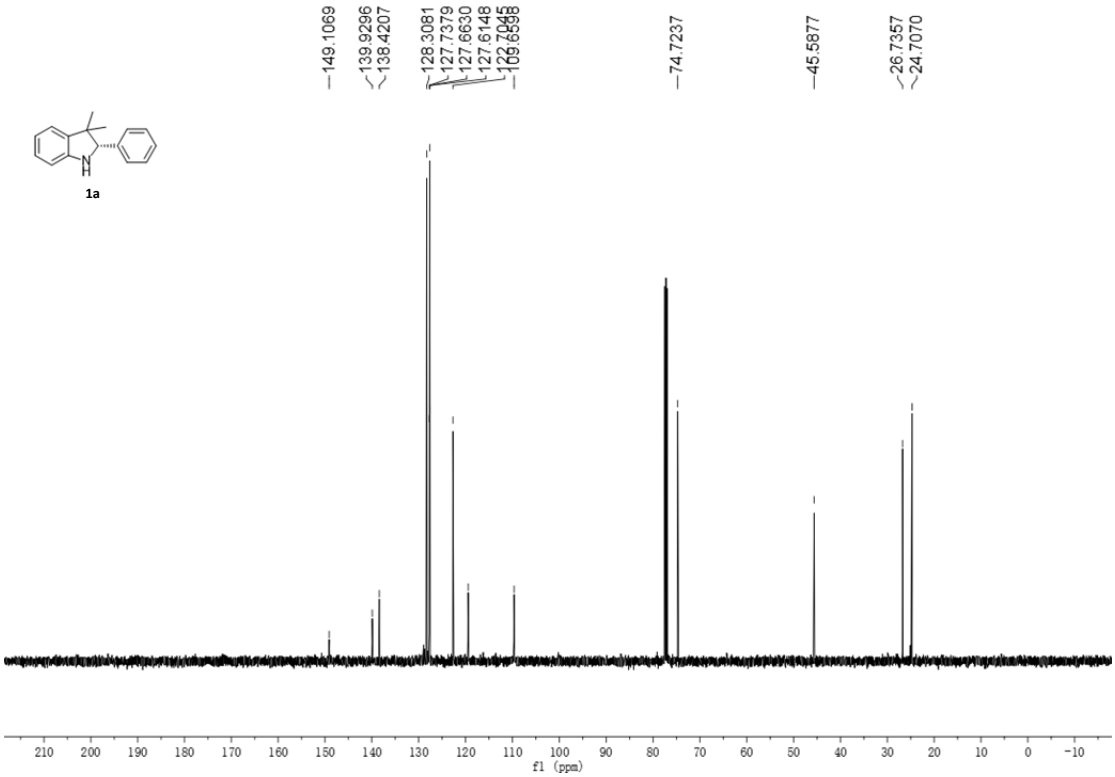
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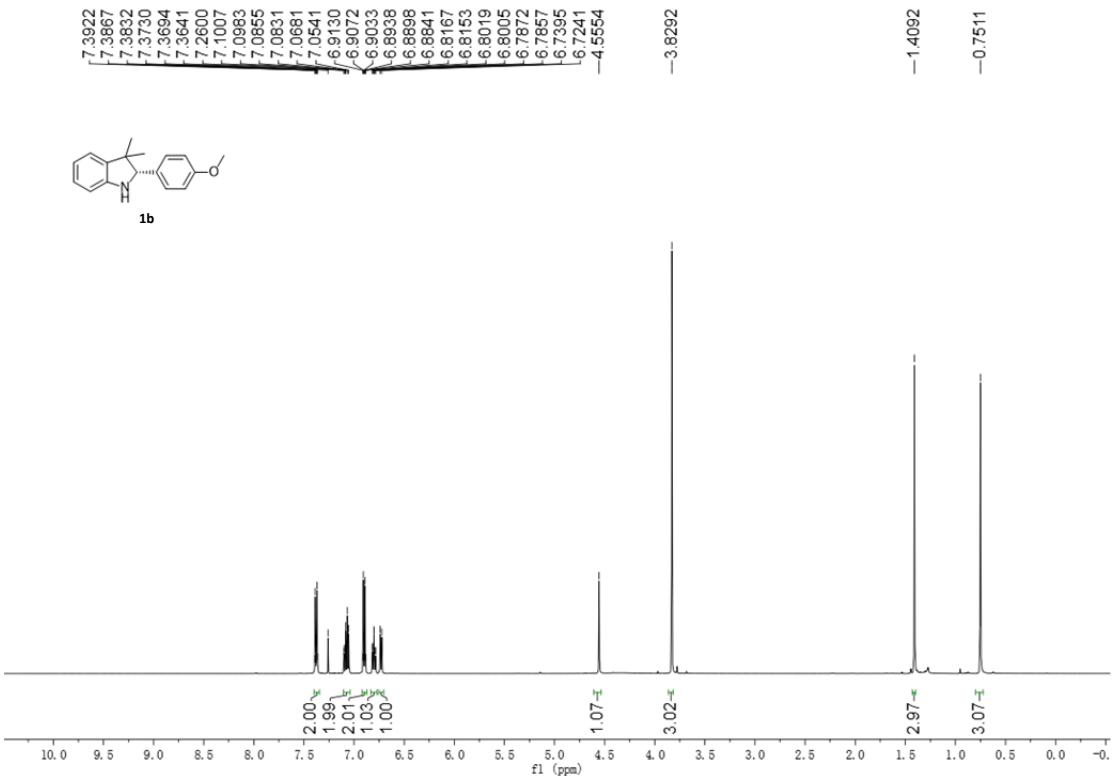
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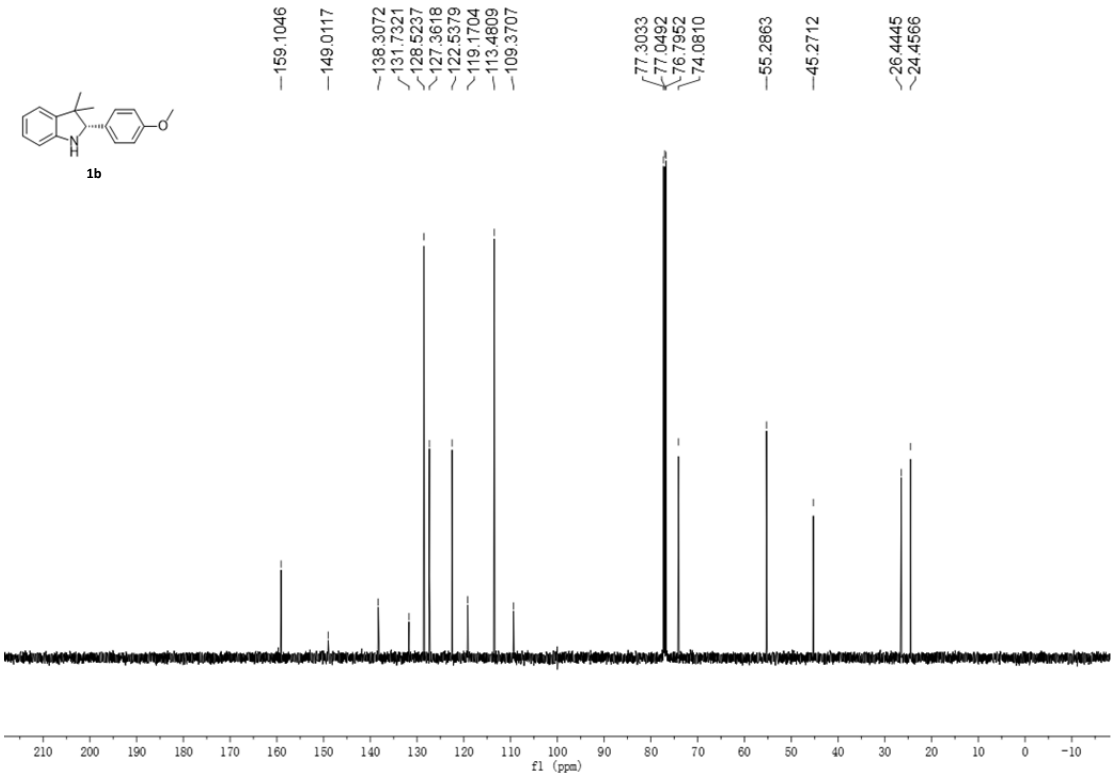
f1 (ppm)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113.4809
109.3707
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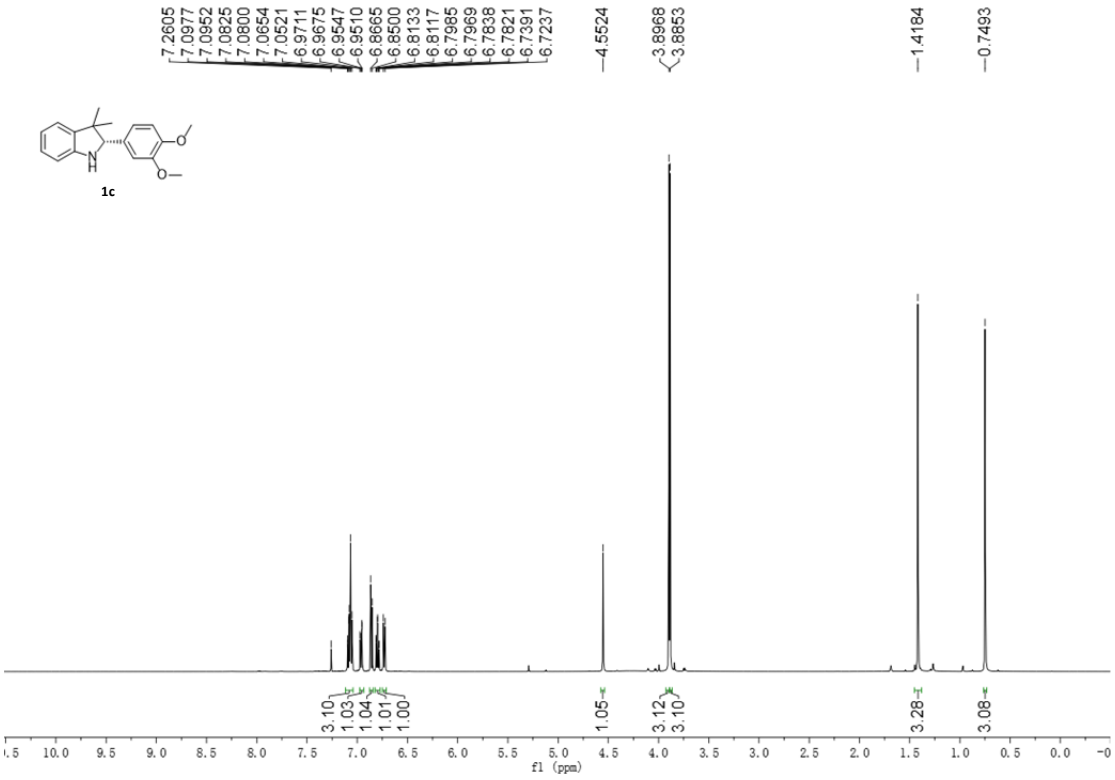
74.0810
55.2863
45.2712
26.4445
24.4566
1b
f1 (ppm)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7.0521
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6.9547
6.9510
6.8665
6.8500
6.8133
6.8117
6.7985
6.7969
6.7838
6.7821
6.7391
6.7237
4.5524
3.8968
3.8853
1.4184
0.7493
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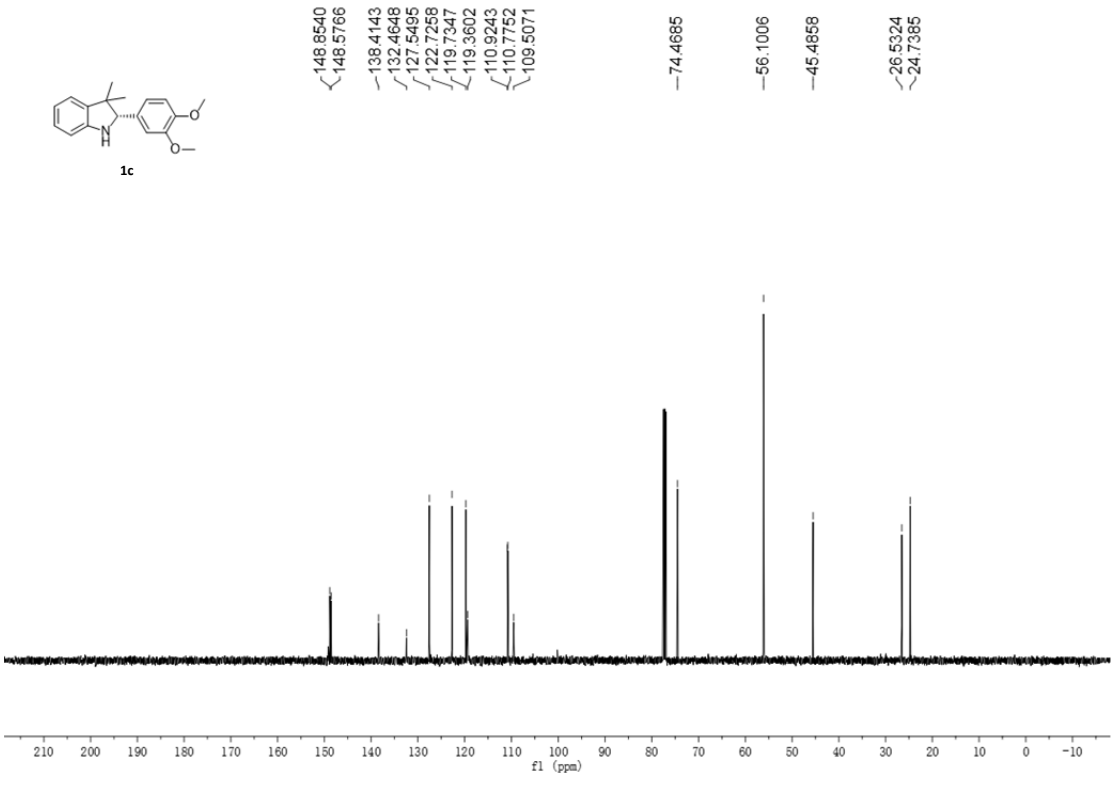
1.04
1.01
1.00
1.05
3.12
3.10
3.28
3.08
f1 (ppm)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56.1006
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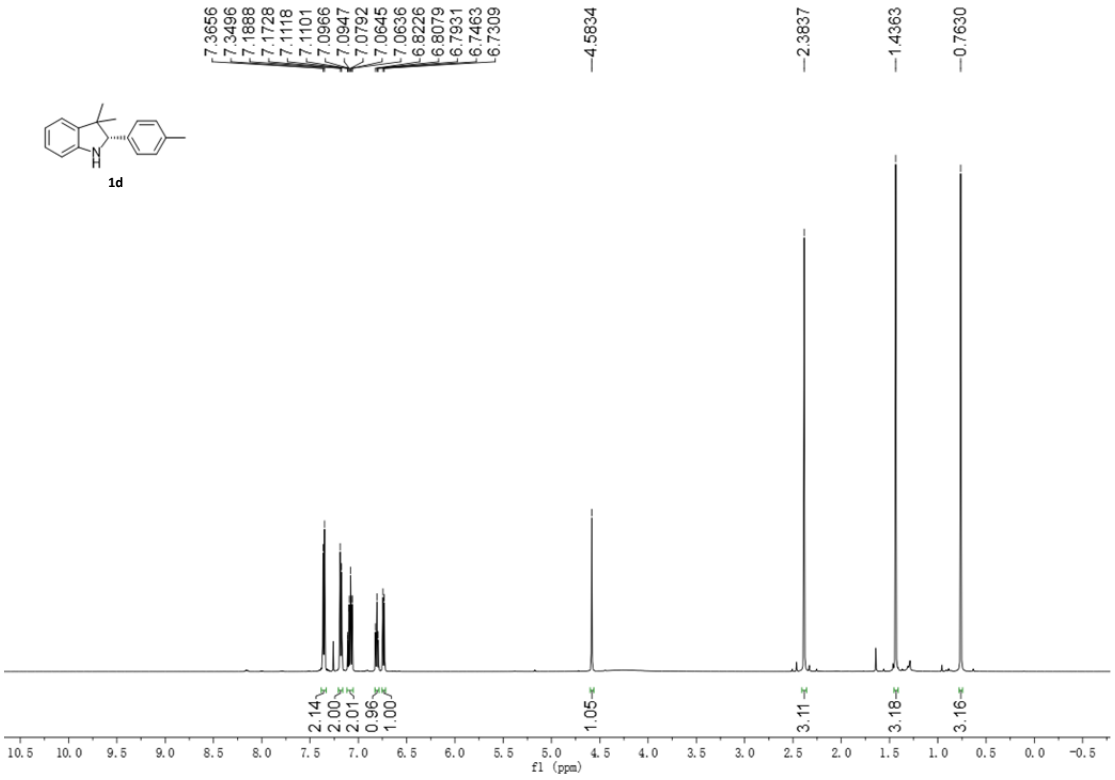
26.5324
24.7385
1c
f1 (ppm)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7.0792
7.0645
7.0636
6.8226
6.8079
6.7931
6.7463
6.7309
4.5834
2.3837
1.4363
0.7630
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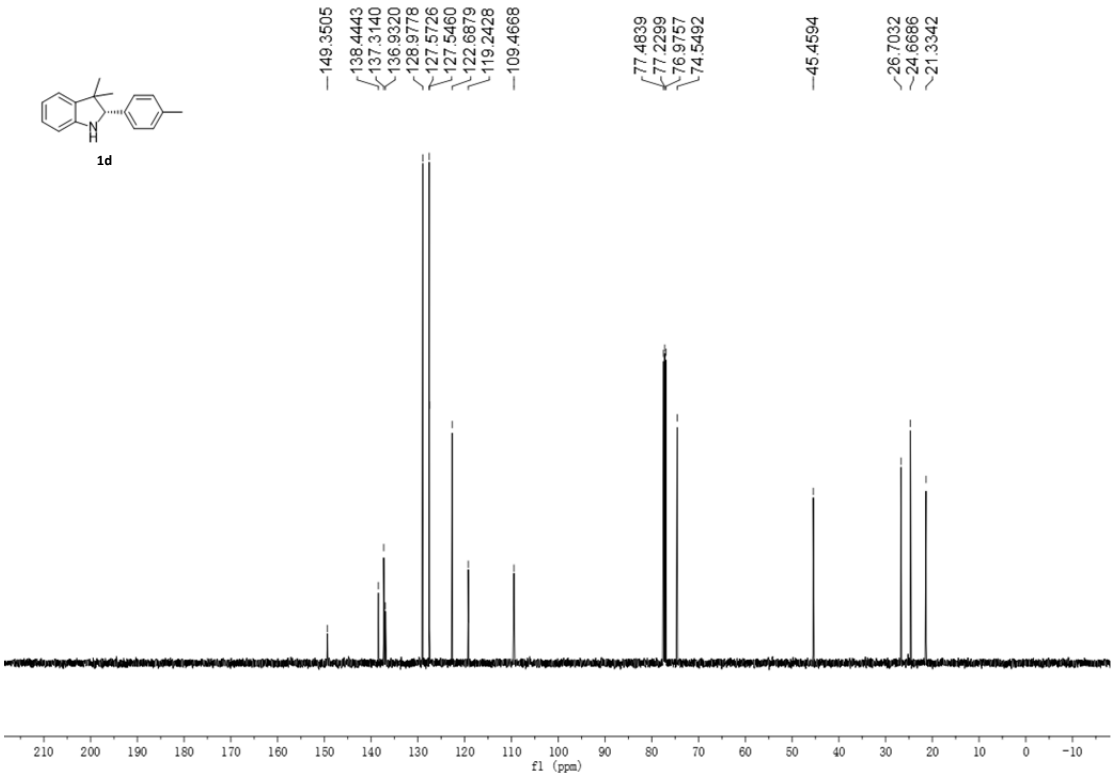
2.00
2.01
0.96
1.00
1.05
3.11
3.18
3.16
f1 (ppm)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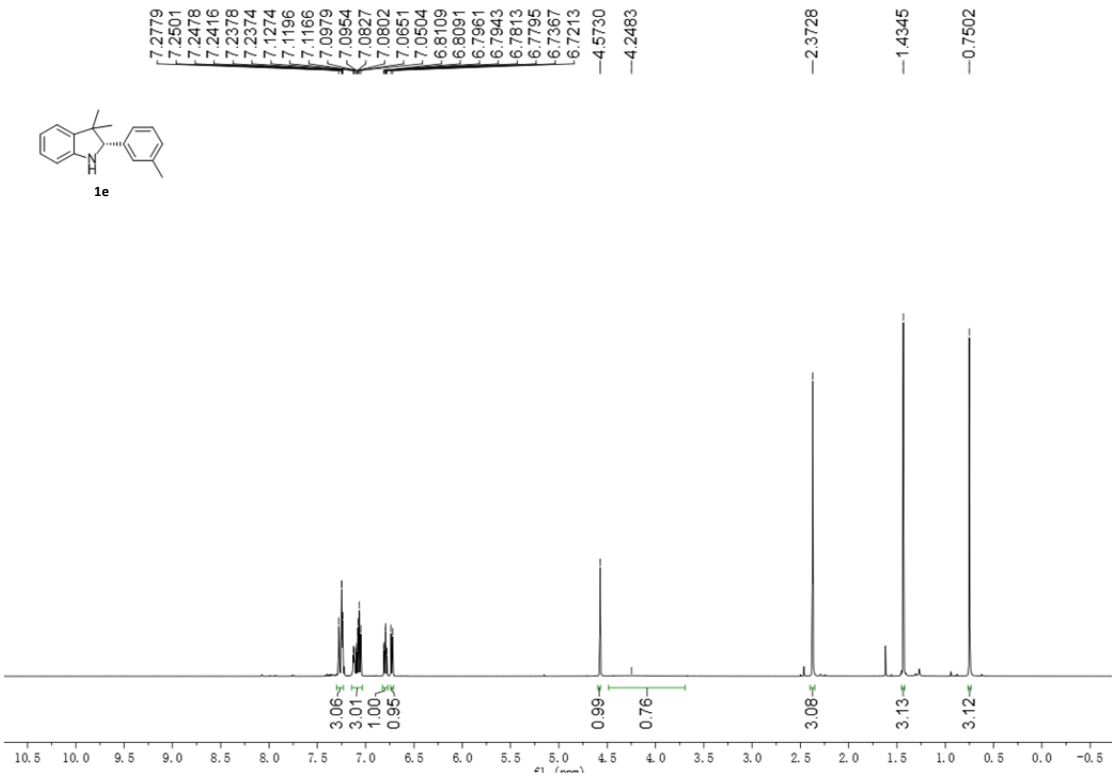
24.6686
21.3342
1d
f1 (ppm)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7.0954
7.0827
7.0802
7.0651
7.0504
6.8109
6.8091
6.7961
6.7943
6.7813
6.7795
6.7367
6.7213
4.5730
4.2483
2.3728
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0.99
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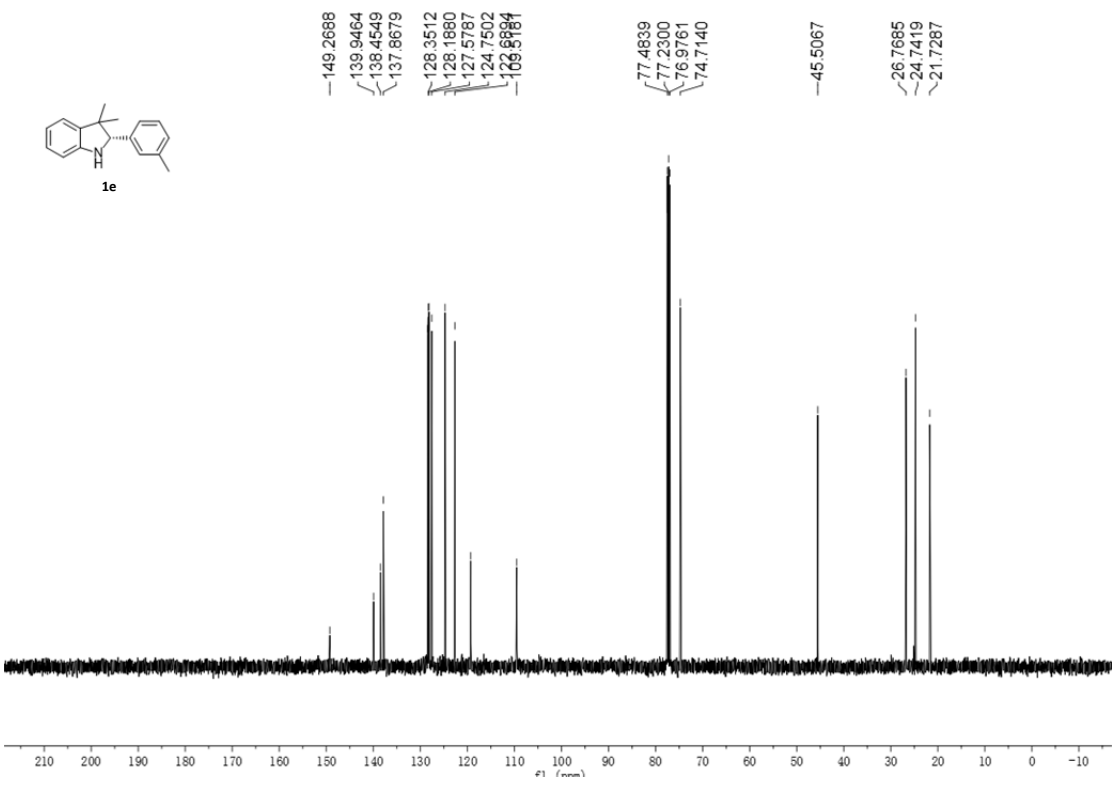
3.08
3.13
3.12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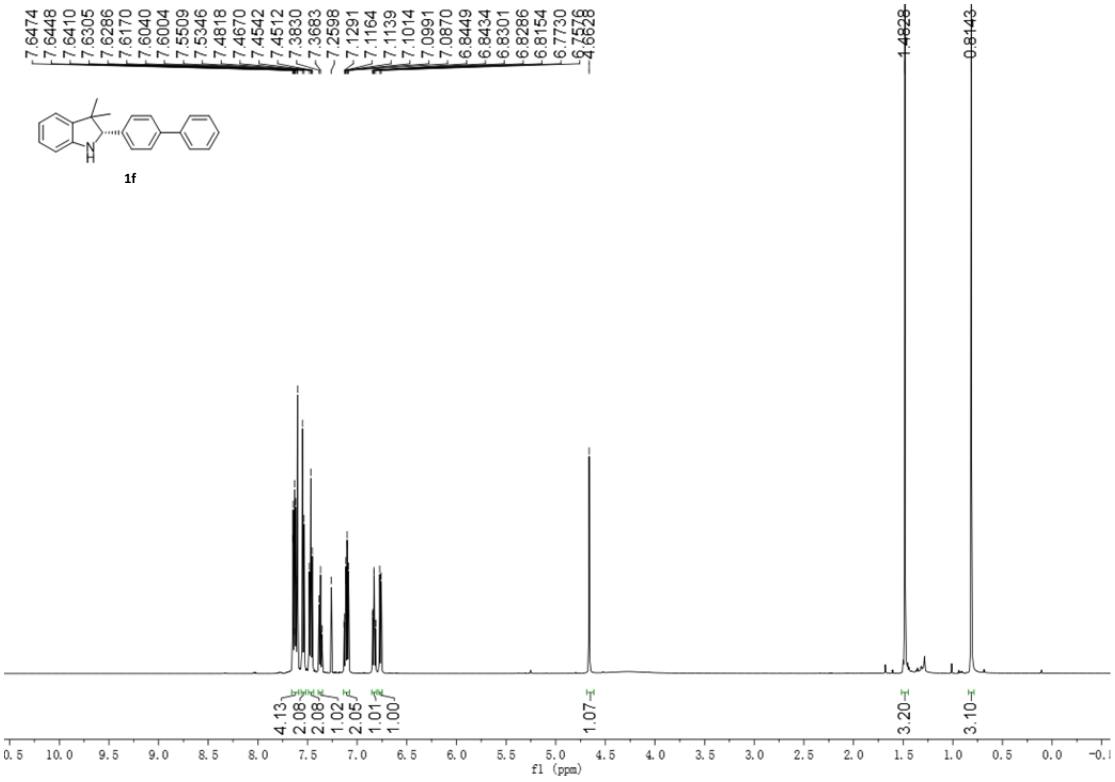
21.7287
1e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7.6410
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7.6040
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7.5346
7.4818
7.4670
7.4542
7.4512
7.3830
7.3683
7.2598
7.1291
7.1164
7.1139
7.1014
7.0991
7.0870
6.8449
6.8434
6.8301
6.8286
6.8154
6.7730
6.7578
6.6628
1.4828
0.8143
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4.13
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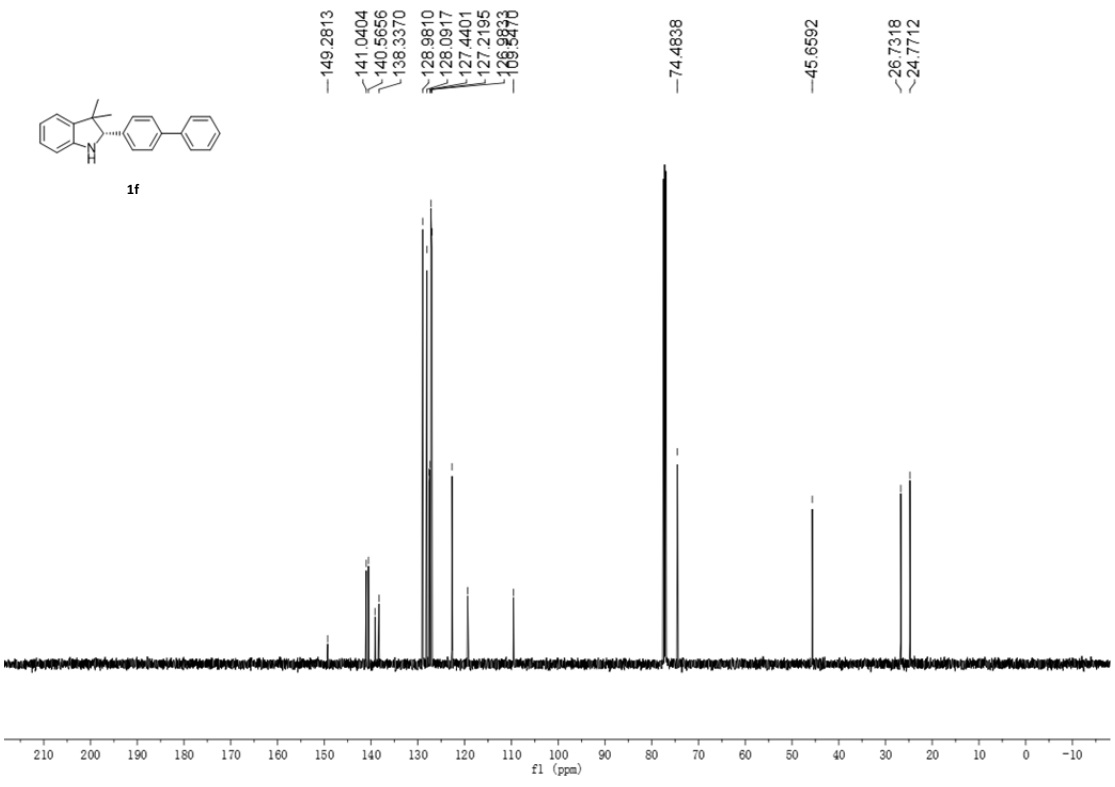
2.08
1.02
2.05
1.01
1.00
1.07
3.20
3.10
f1 (ppm)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138.3370
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127.4401
127.2195
74.4838
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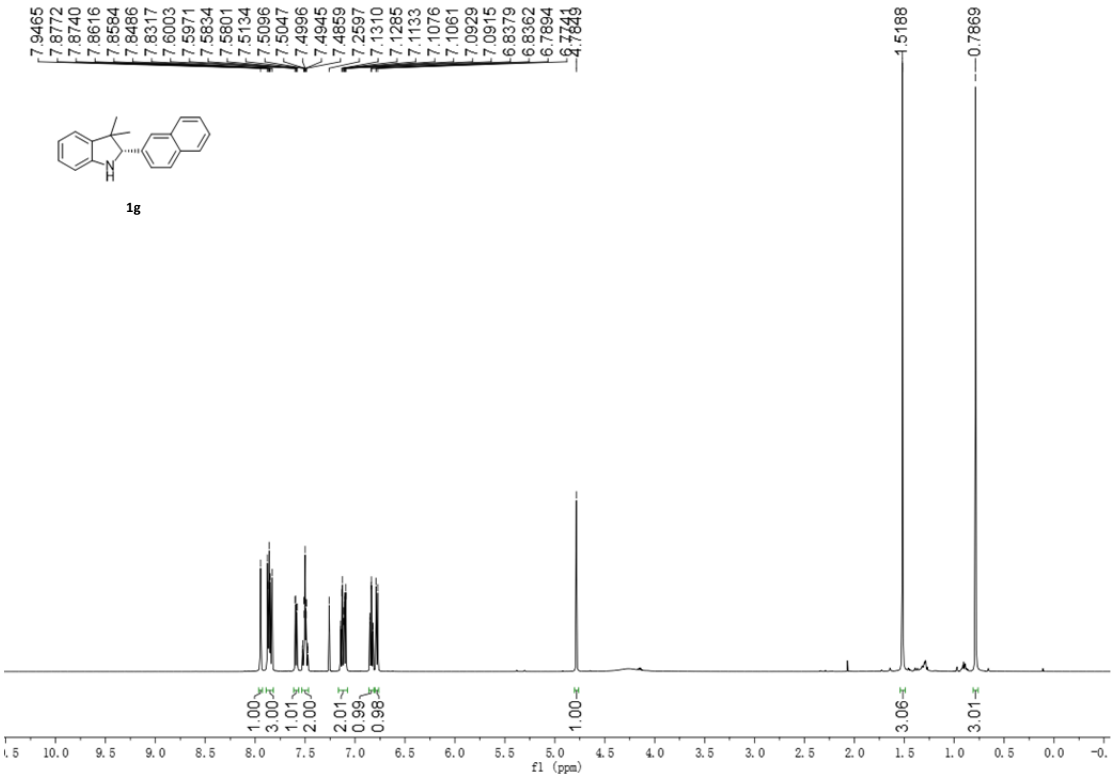
26.7318
24.7712
1f
f1 (ppm)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7.8616
7.8584
7.8486
7.8317
7.6003
7.5971
7.5834
7.5801
7.5134
7.5096
7.5047
7.4996
7.4945
7.4859
7.2597
7.1310
7.1285
7.1133
7.1076
7.1061
7.0929
7.0915
6.8379
6.8362
6.7894
1.5188
0.7869
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3.00
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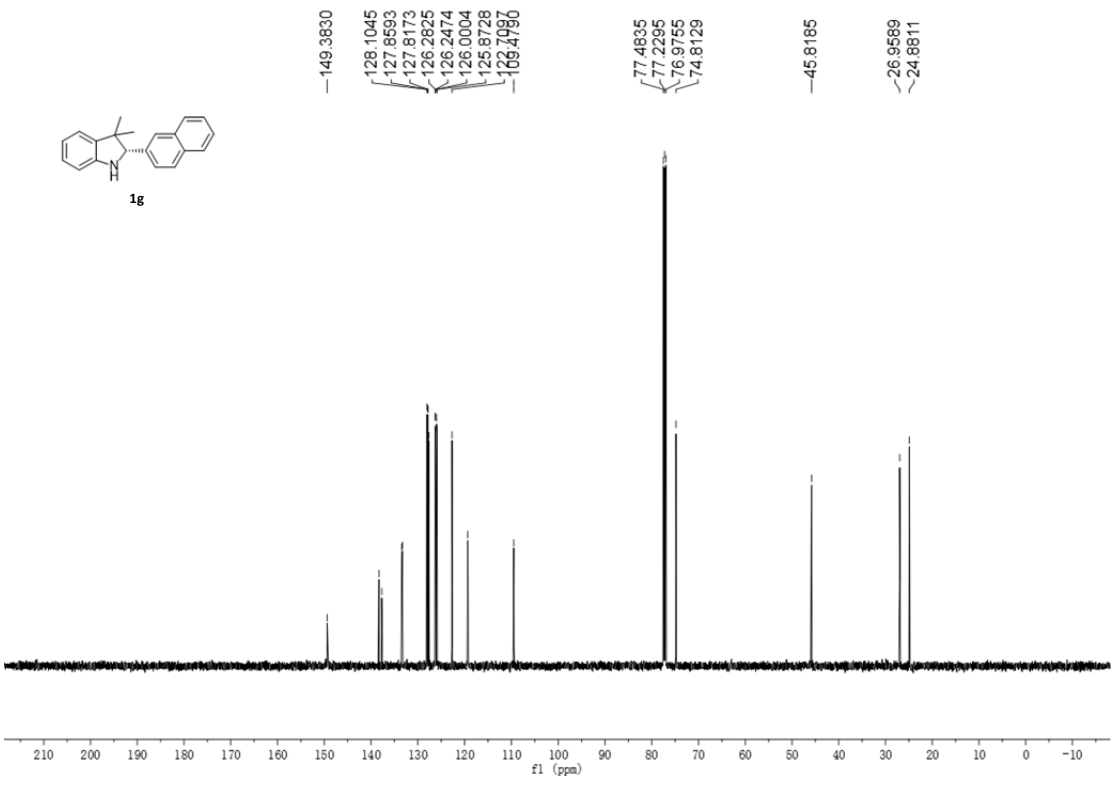
2.00
2.01
0.99
0.98
1.00
3.06
3.01
f1 (ppm)

1g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127.8173
126.2825
126.2474
126.0004
125.8728
122.7097
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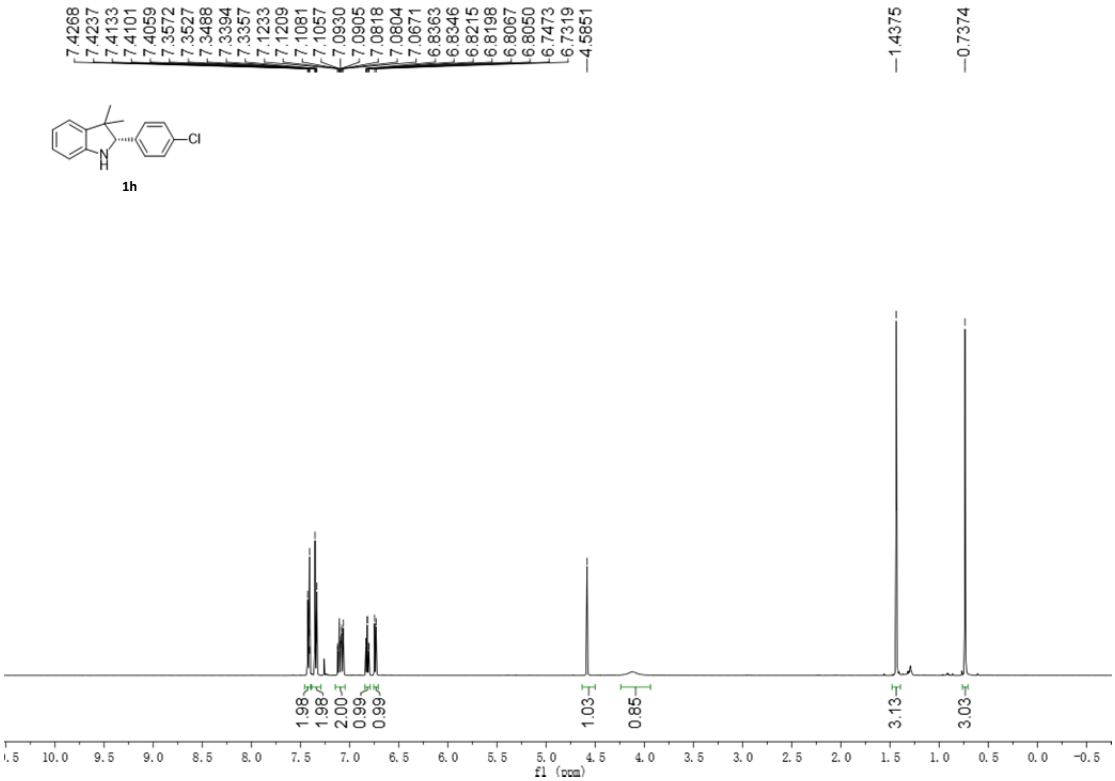
77.2295
76.9755
74.8129
45.8185
26.9589
24.8811
f1 (ppm)

1g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7.3394
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7.1209
7.1081
7.1057
7.0930
7.0905
7.0818
7.0804
7.0671
6.8363
6.8346
6.8215
6.8198
6.8067
6.8050
6.7473
6.7319
4.5851
1.4375
0.7374
Cl
1h
1.98
1.98
2.00
0.99
0.99
1.03
0.85
3.13
3.03
f1 (ppm)
149.1768
138.5948
137.9967
133.3045
128.9198
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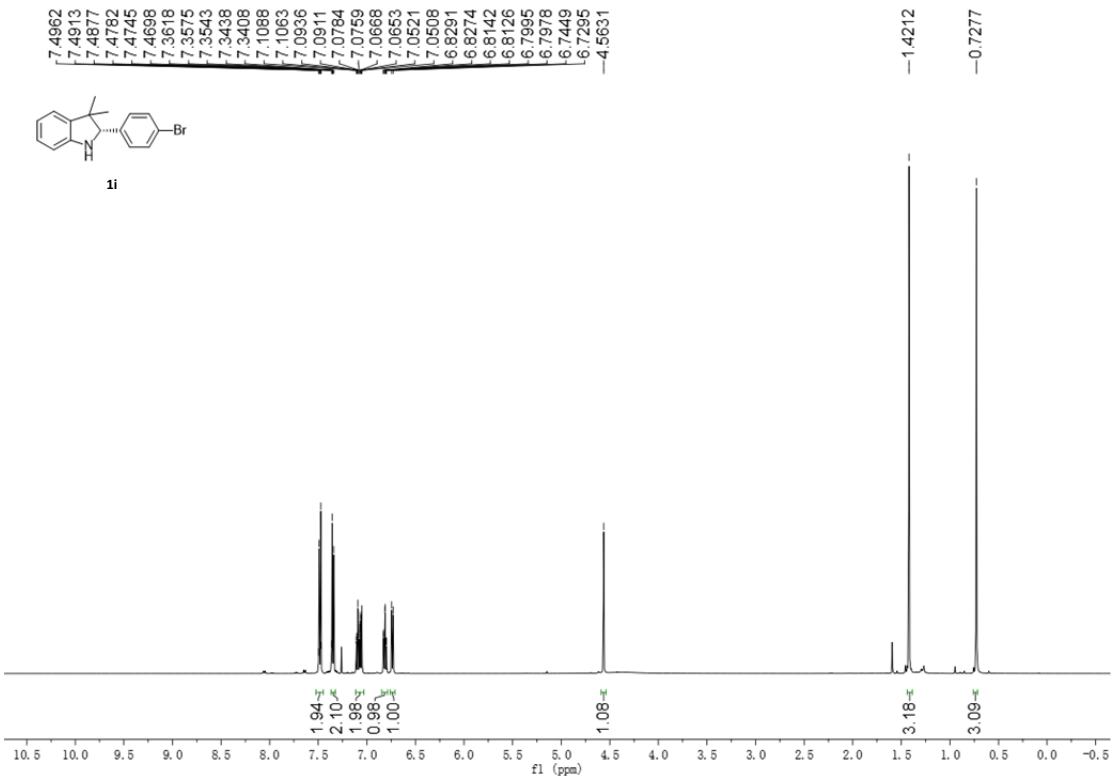
24.7027
Cl
1h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7.3408
7.1088
7.1063
7.0936
7.0911
7.0784
7.0759
7.0668
7.0653
7.0521
7.0508
6.8291
6.8274
6.8142
6.8126
6.7995
6.7978
6.7449
6.7295
4.5631
1.4212
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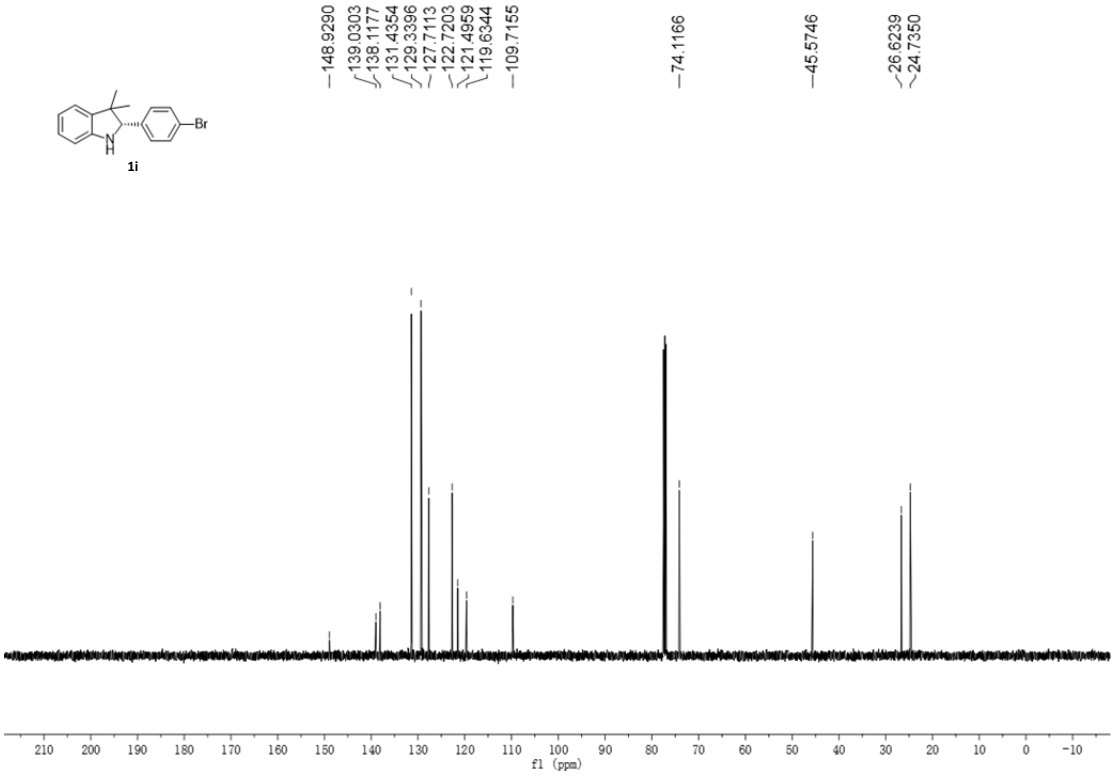
Br
1i
1.94
2.10
1.98
0.98
1.00
1.08
3.18
3.09
f1 (ppm)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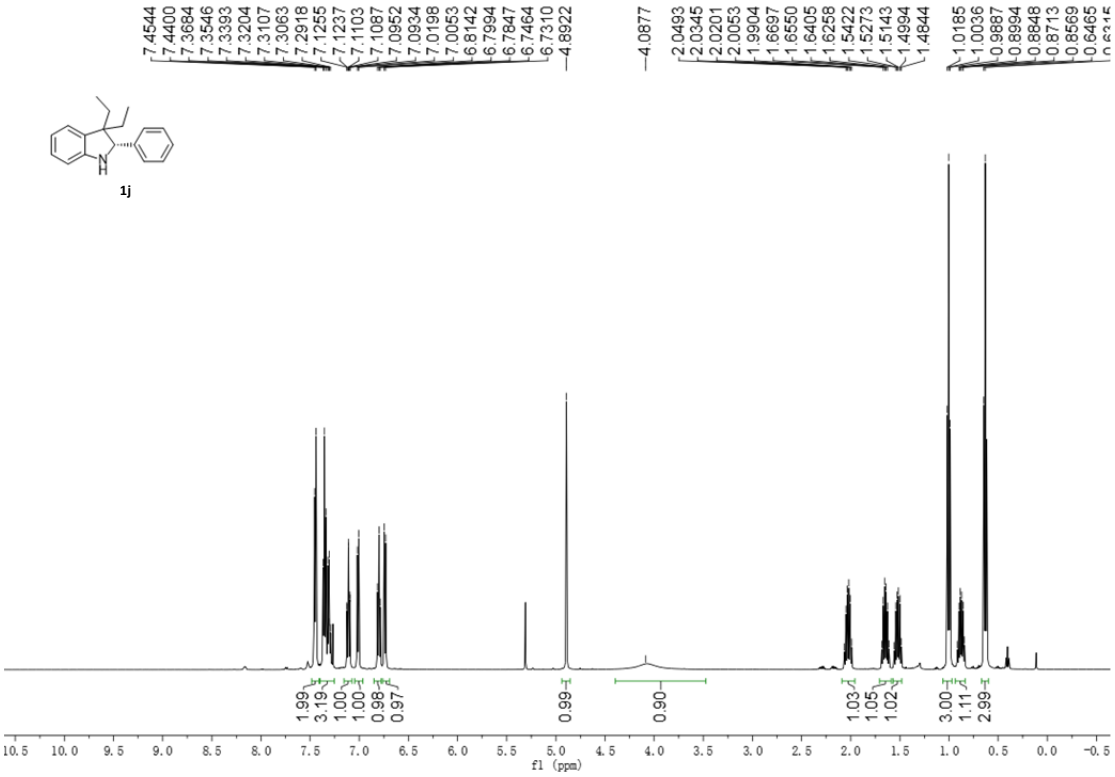
109.7155
74.1166
45.5746
26.6239
24.7350
Br
1i
f1 (ppm)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7.2918
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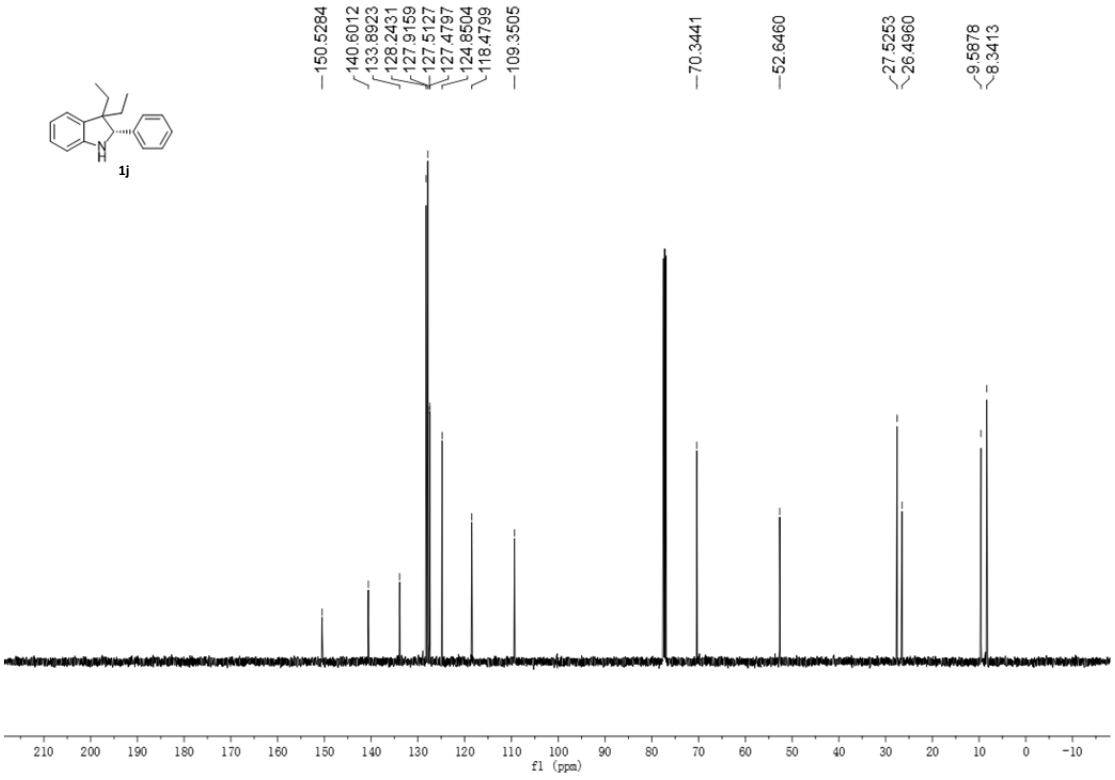
7.1237
7.1103
7.1087
7.0952
7.0934
7.0198
7.0053
6.8142
6.7994
6.7847
6.7464
6.7310
4.8922
4.0877
2.0493
2.0345
2.0201
2.0053
1.9904
1.6697
1.6550
1.6405
1.6258
1.5422
1.5273
1.5143
1.4994
1.4844
1.0185
1.0036
0.9887
0.8994
0.8848
0.8713
0.8569
0.6465
1j
1.99
3.19
1.00
1.00
0.98
0.97
0.99
0.90
1.03
1.05
1.02
3.00
1.11
2.99
f1 (ppm)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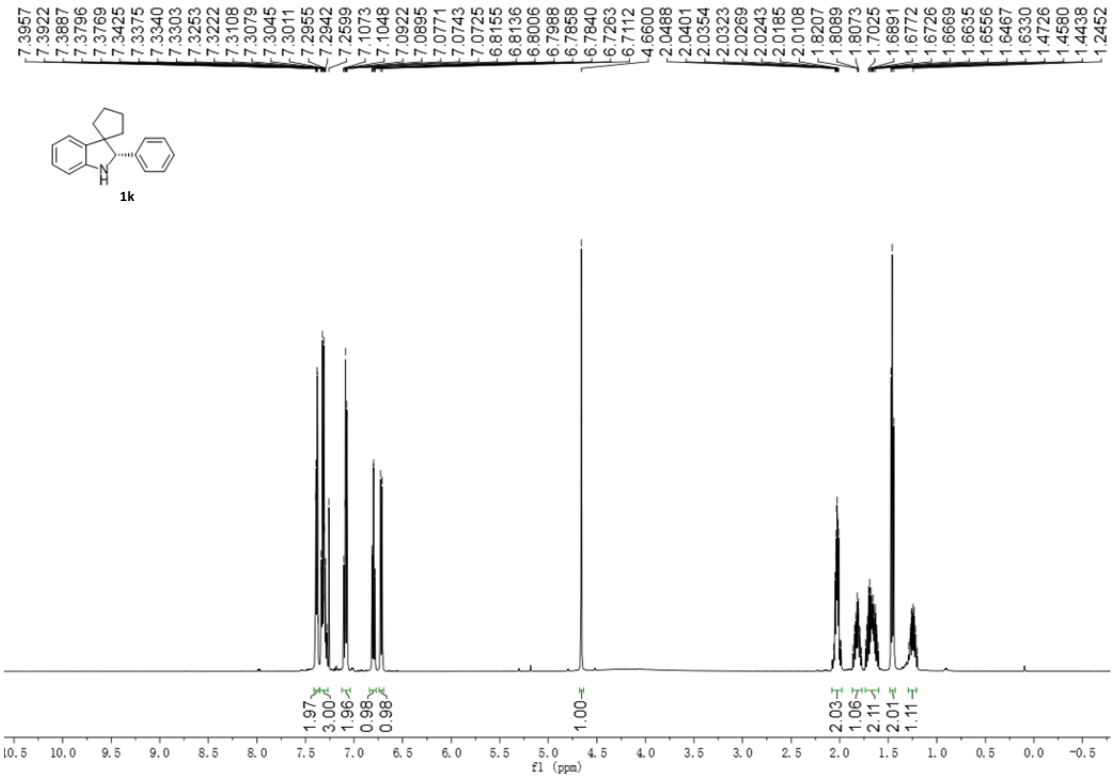
52.6460
27.5253
26.4960
9.5878
8.3413
1j
f1 (ppm)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7.3375
7.3340
7.3303
7.3253
7.3222
7.3108
7.3079
7.3045
7.3011
7.2955
7.2942
7.2599
7.1073
7.1048
7.0922
7.0895
7.0771
7.0743
7.0725
6.8155
6.8136
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6.7988
6.7858
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2.0354
2.0323
2.0269
2.0243
2.0185
2.0108
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1.8089
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1.7025
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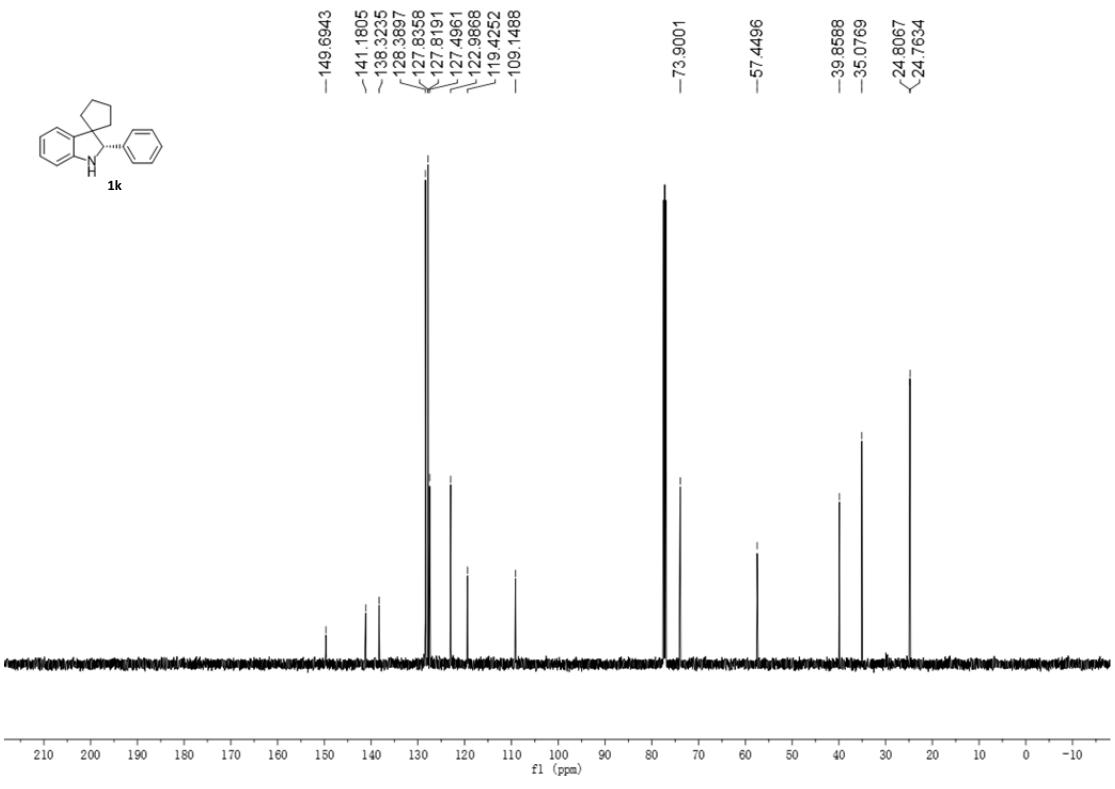
1.6772
1.6726
1.6669
1.6635
1.6556
1.6467
1.6330
1.4726
1.4580
1.4438
1.2452
1k
1.97
3.00
1.96
0.98
0.98
1.00
2.03
1.06
2.11
2.01
1.11
f1 (ppm)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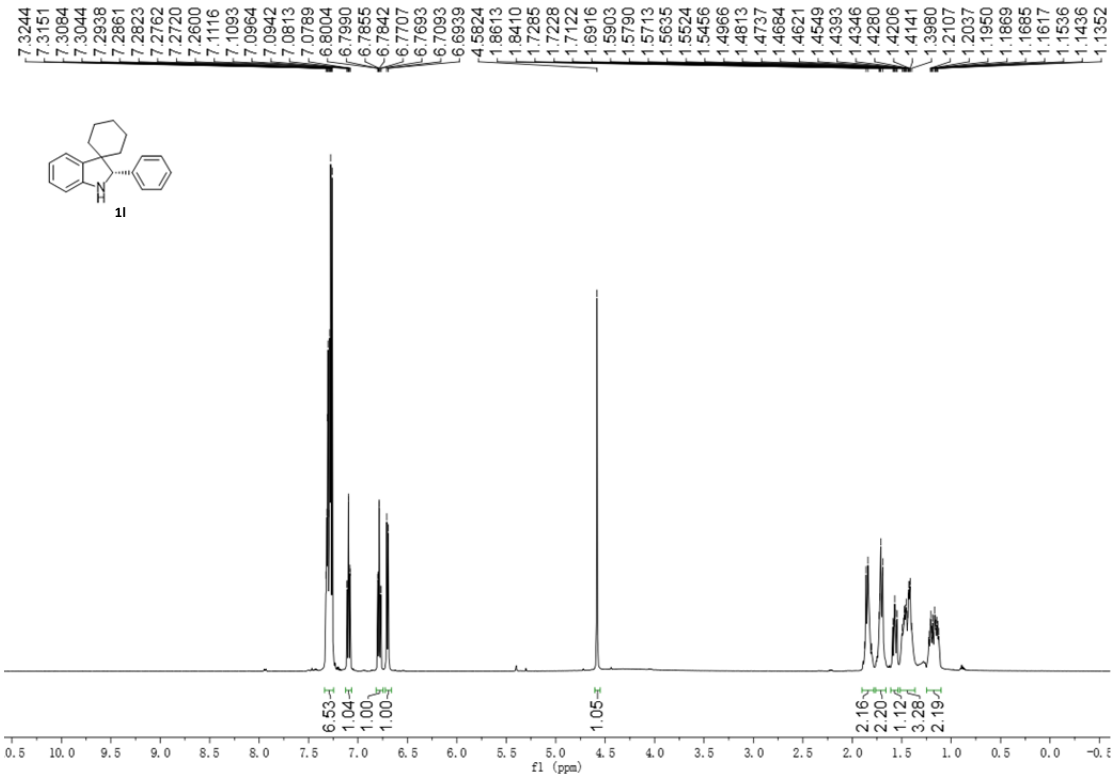
35.0769
24.8067
24.7634
1k
f1 (ppm)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7.1093
7.0964
7.0942
7.0813
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1.4346
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1.4206
1.4141
1.3980
1.2107
1.2037
1.1950
1.1869
1.1685
1.1617
1.1536
1.1436
1.1352
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1.00
1.00
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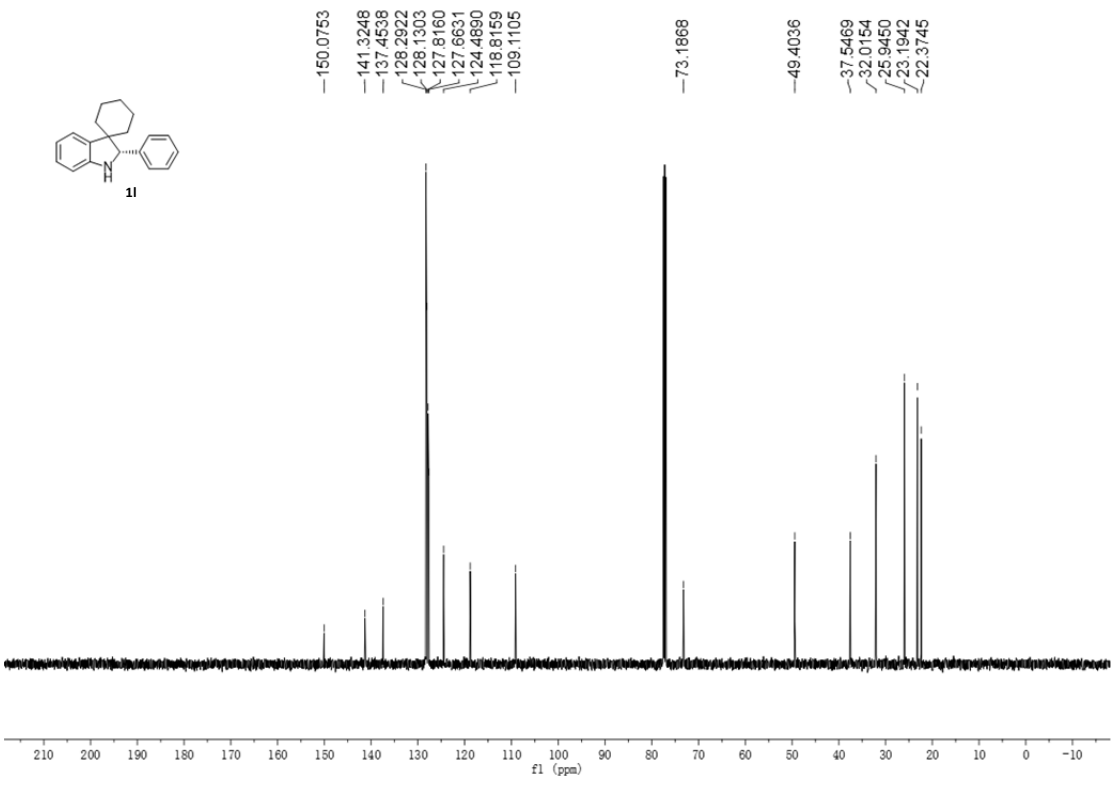
2.19
f1 (ppm)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118.8159
109.1105
73.1868
49.4036
37.5469
32.0154
25.9450
23.1942
22.3745
1l
f1 (ppm)


7.3858
7.3751
7.3711
7.3662
7.3531
7.2977
7.2949
7.2903
7.2774
7.2734
7.2663
7.2617
7.2585
7.2346
7.2312
7.2276
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7.2134
7.0421
7.0240
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6.6692
6.6672
6.6330
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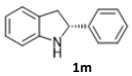
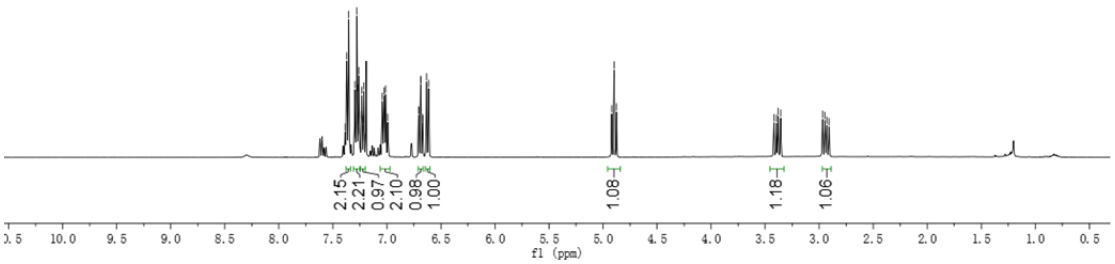


1m


2.15
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2.10
0.98
1.00
1.08
1.18
1.06
f1 (ppm)


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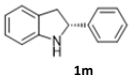
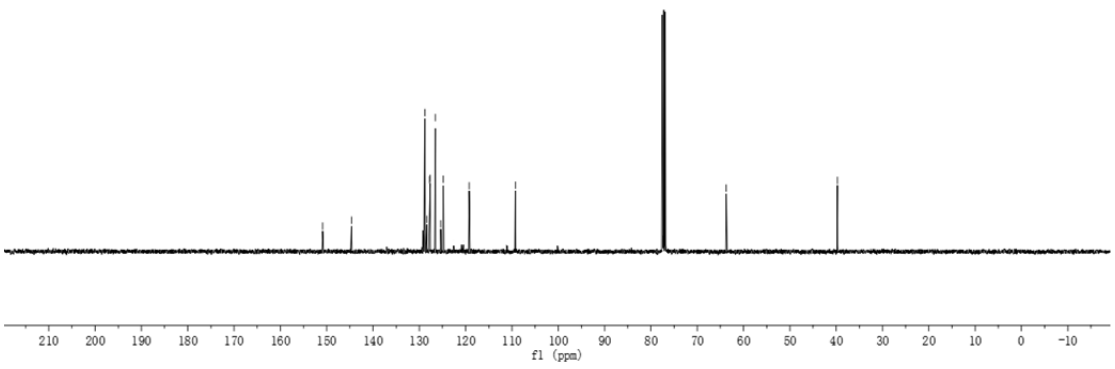
128.8179
128.4057
127.7894
127.6485
126.5271
124.8168
63.7355
39.7265



1m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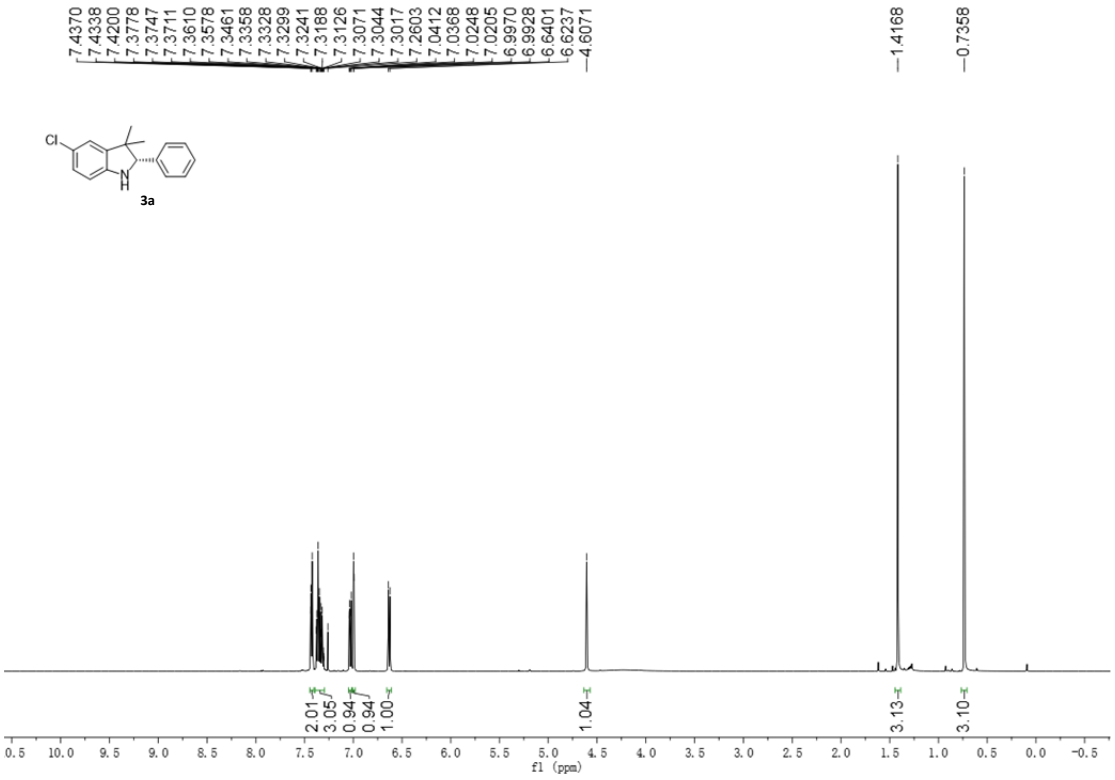
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7.3188
7.3126
7.3071
7.3044
7.3017
7.2603
7.0412
7.0368
7.0248
7.0205
6.9970
6.9928
6.6401
6.6237
4.6071
1.4168
0.7358
Cl
N
H
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3.05
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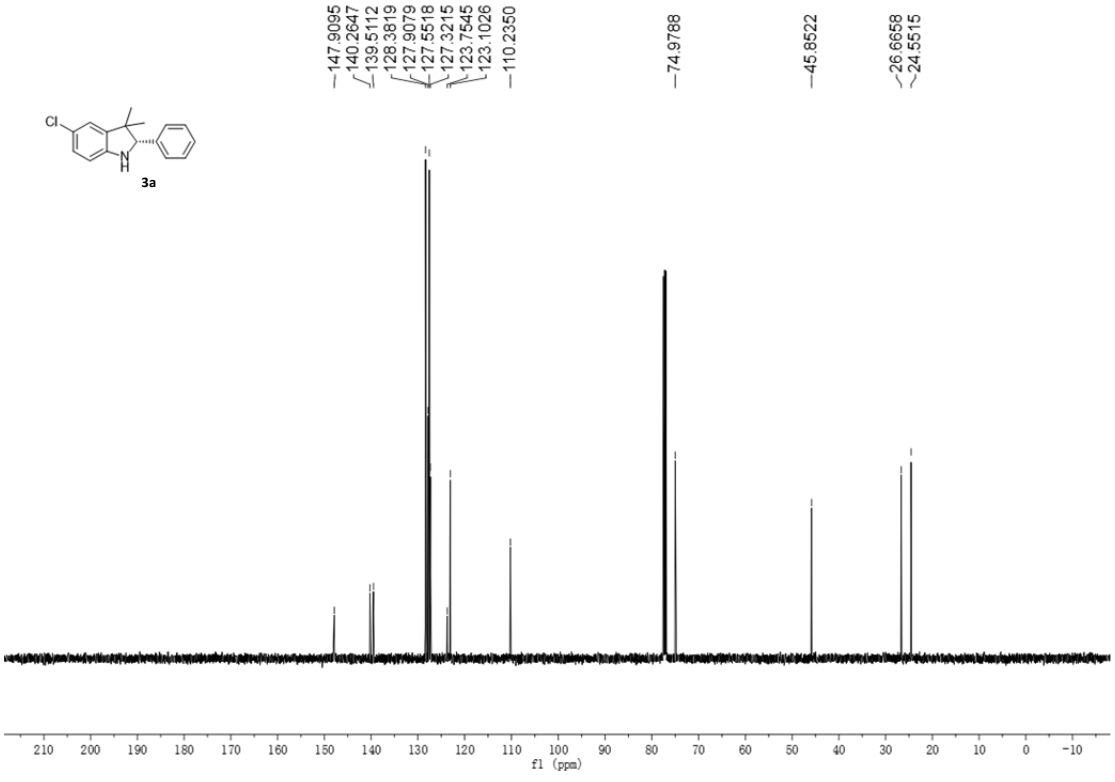
0.94
1.00
1.04
3.13
3.10
f1 (ppm)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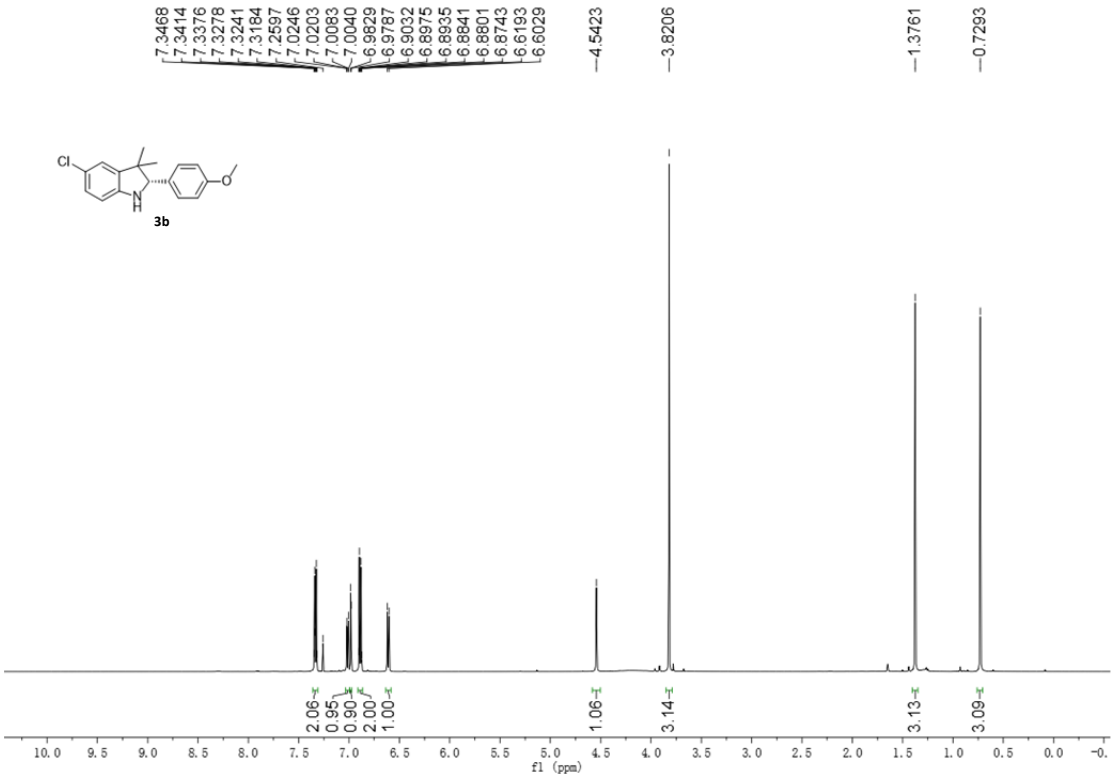
127.5518
127.3215
123.7545
123.1026
110.2350
74.9788
45.8522
26.6658
24.5515
Cl
N
H
3a
f1 (ppm)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7.0203
7.0083
7.0040
6.9829
6.9787
6.9032
6.8975
6.8935
6.8841
6.8801
6.8743
6.6193
6.6029
4.5423
3.8206
1.3761
0.7293
Cl
O
H
3b
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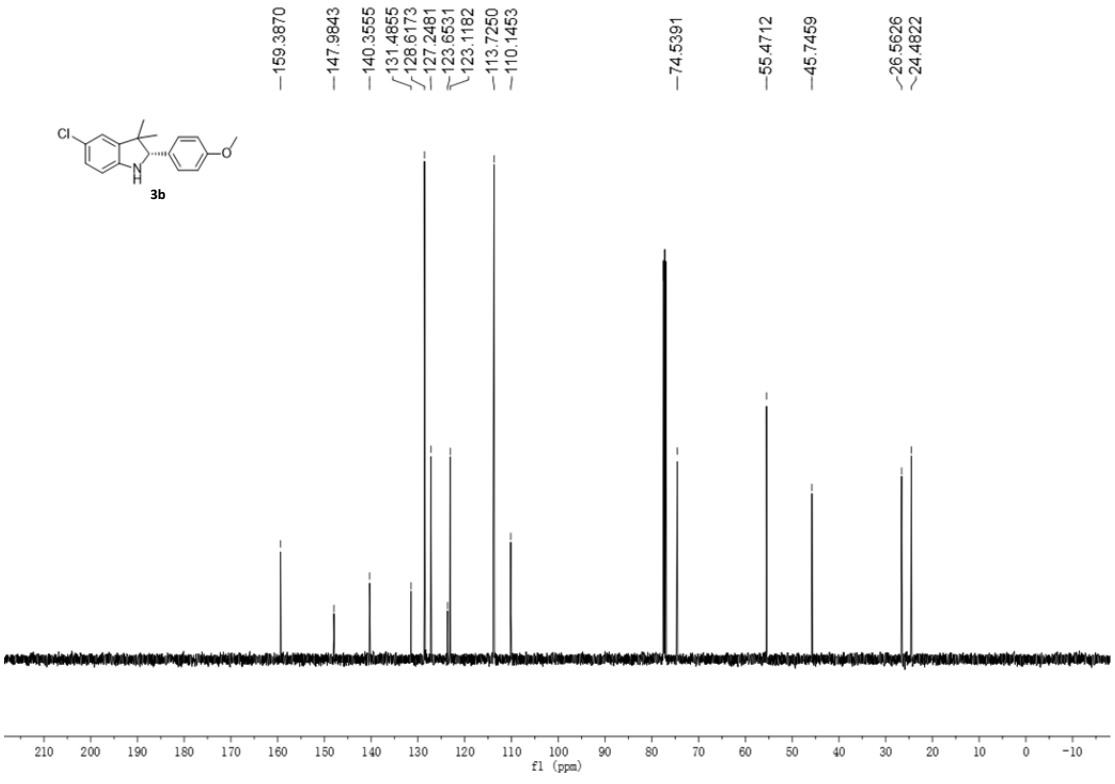
0.90
2.00
1.00
1.06
3.14
3.13
3.09
f1 (ppm)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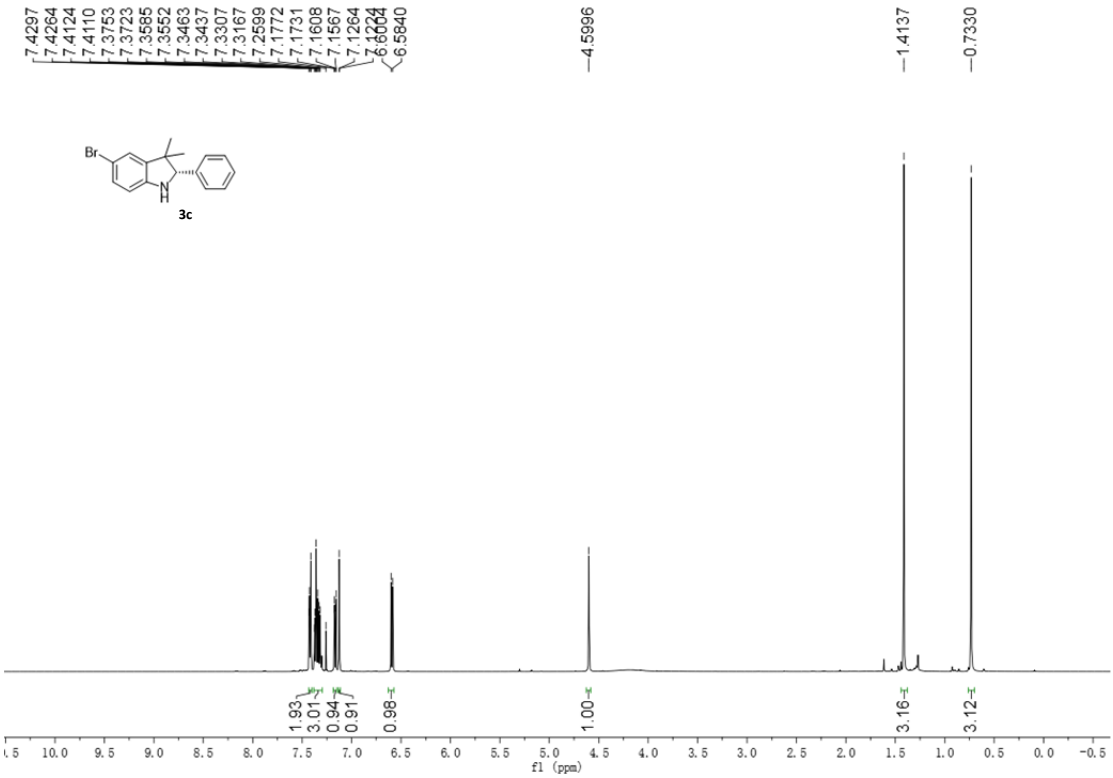
55.4712
45.7459
26.5626
24.4822
Cl
O
H
3b
f1 (ppm)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7.3307
7.3167
7.2599
7.1772
7.1731
7.1608
7.1567
7.1264
6.6004
6.5840
4.5996
1.4137
0.7330
Br
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0.94
0.91
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1.00
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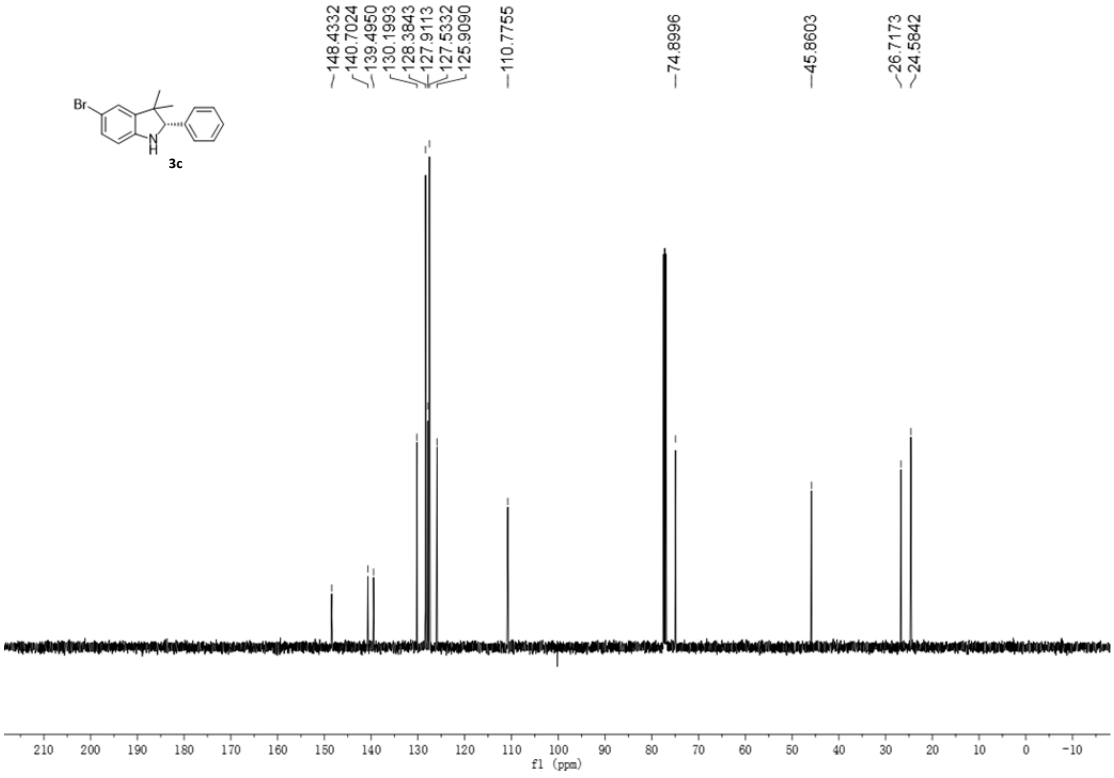
3.12
f1 (ppm)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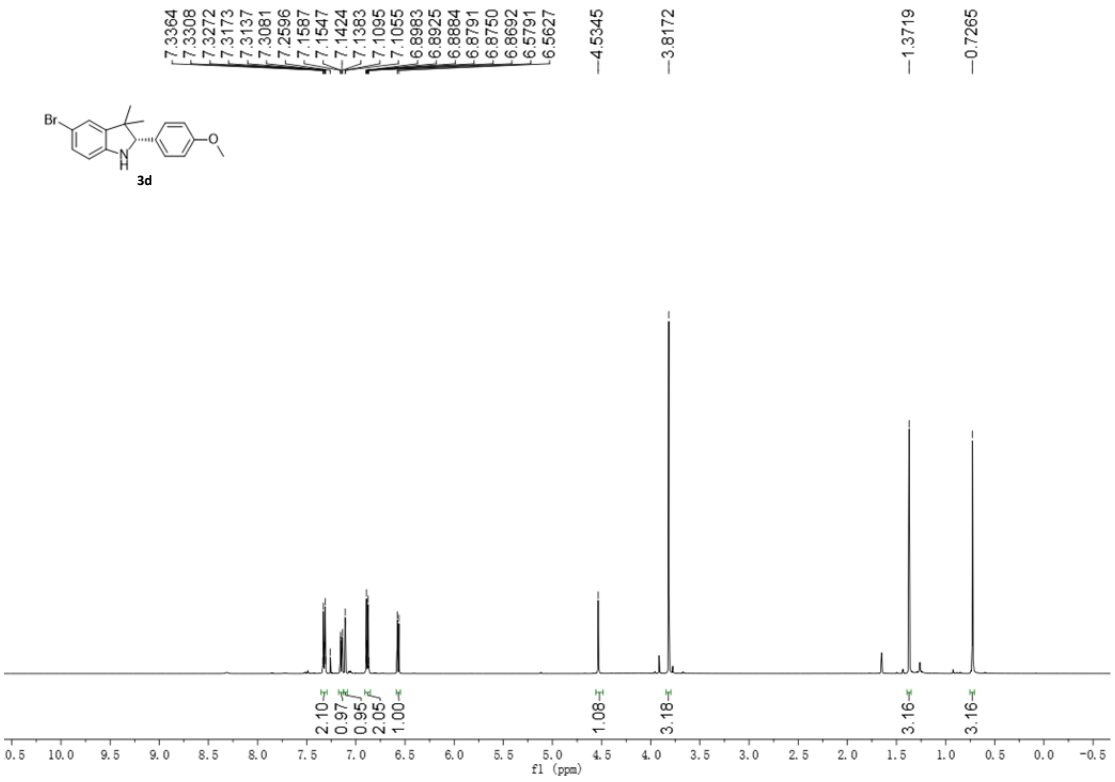
Br
N
H
3c
f1 (ppm)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7.1547
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7.1055
6.8983
6.8925
6.8884
6.8791
6.8750
6.8692
6.5791
6.5627
4.5345
3.8172
1.3719
0.7265
Br
O
N
H
3d
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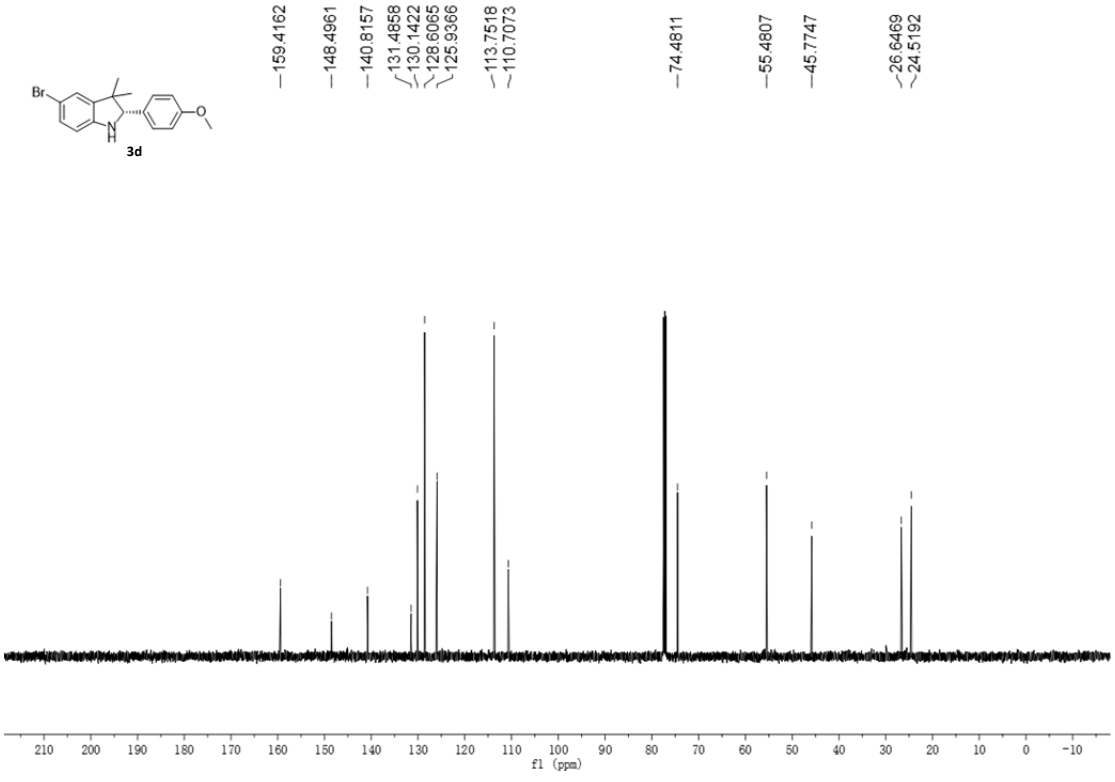
0.97
0.95
2.05
1.00
1.08
3.18
3.16
3.16
f1 (ppm)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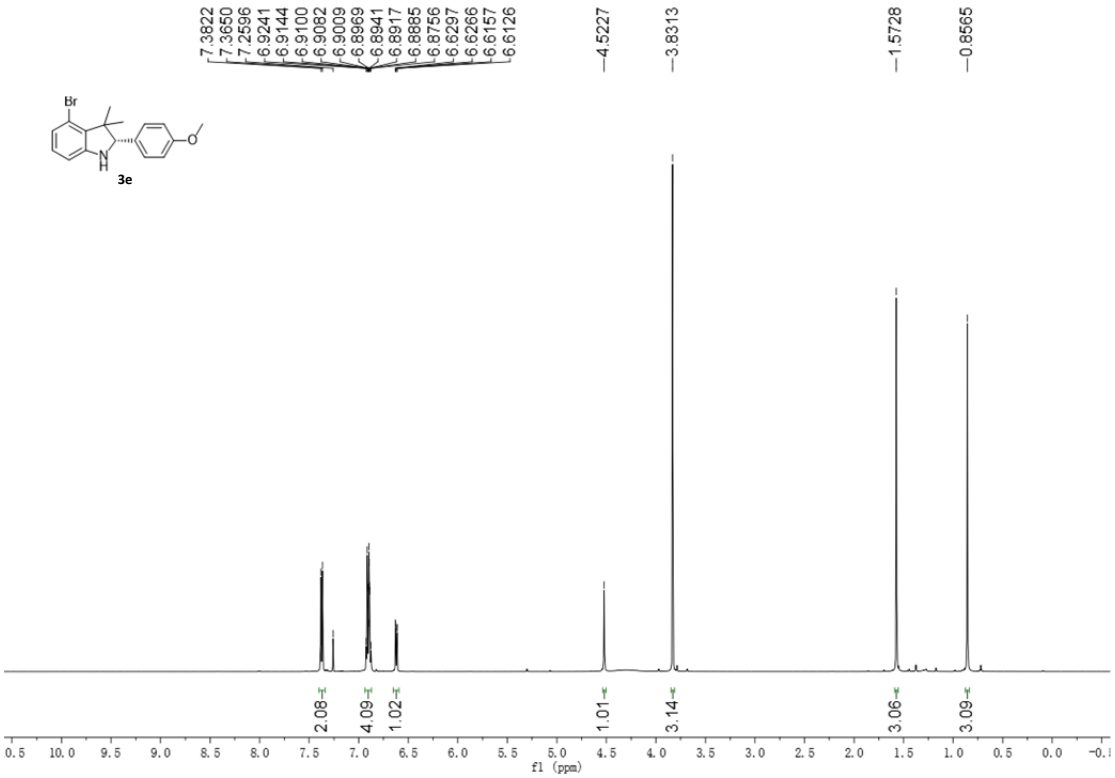
24.5192
Br
O
N
H
3d
f1 (ppm)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6.8917
6.8885
6.8756
6.6297
6.6266
6.6157
6.6126
4.5227
3.8313
1.5728
0.8565
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O
H
N
3e
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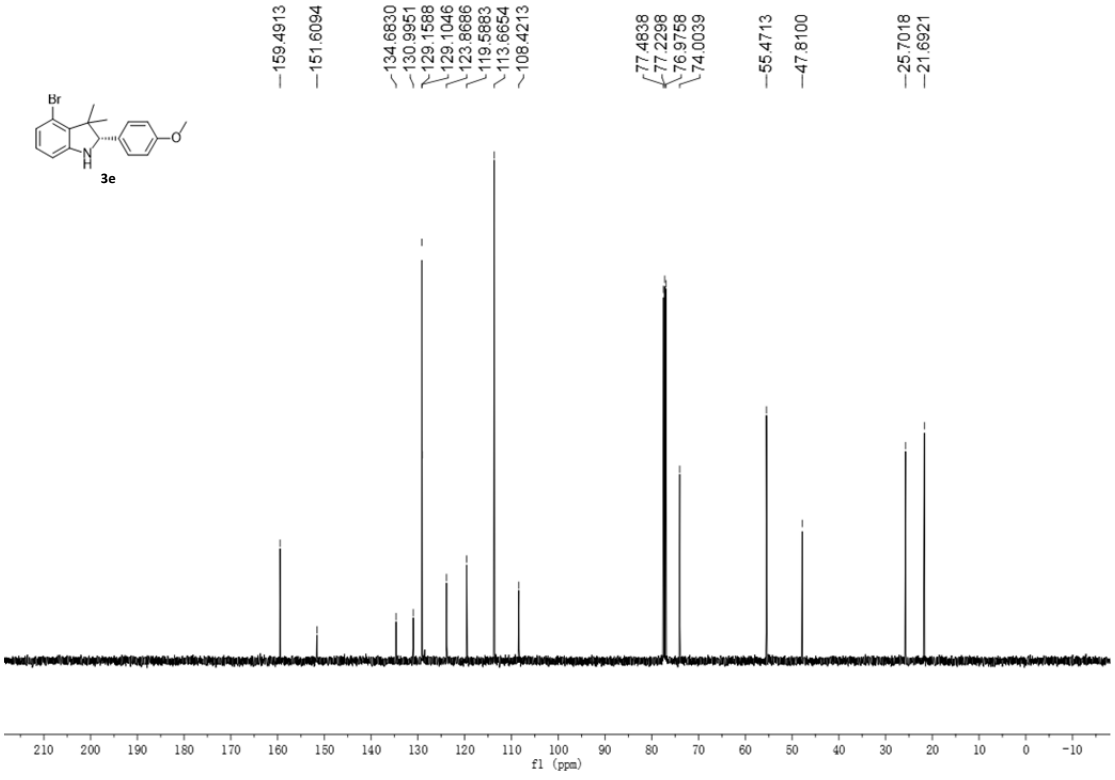
4.09
1.02
1.01
3.14
3.06
3.09
f1 (ppm)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77.2298
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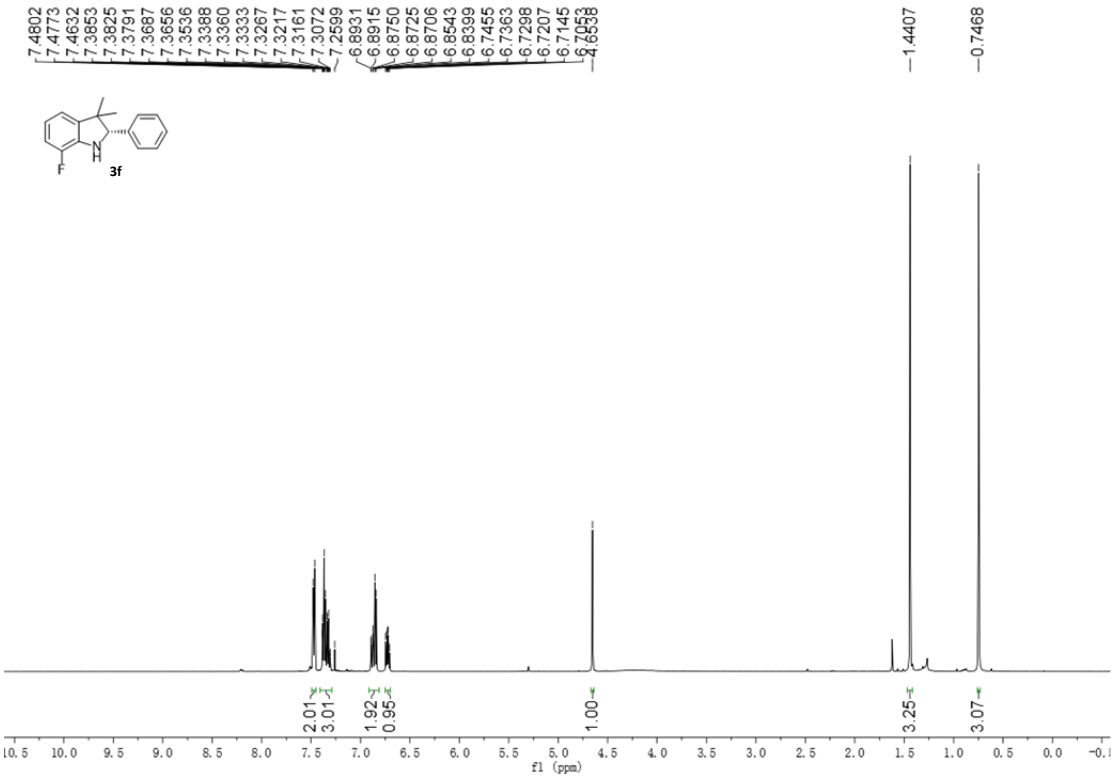
21.6921
Br
O
H
N
3e
f1 (ppm)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7.3656
7.3536
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7.3360
7.3333
7.3267
7.3217
7.3161
7.3072
7.2599
6.8931
6.8915
6.8750
6.8725
6.8706
6.8543
6.8399
6.7455
6.7363
6.7298
6.7207
6.7145
6.6538
1.4407
0.7468
3f
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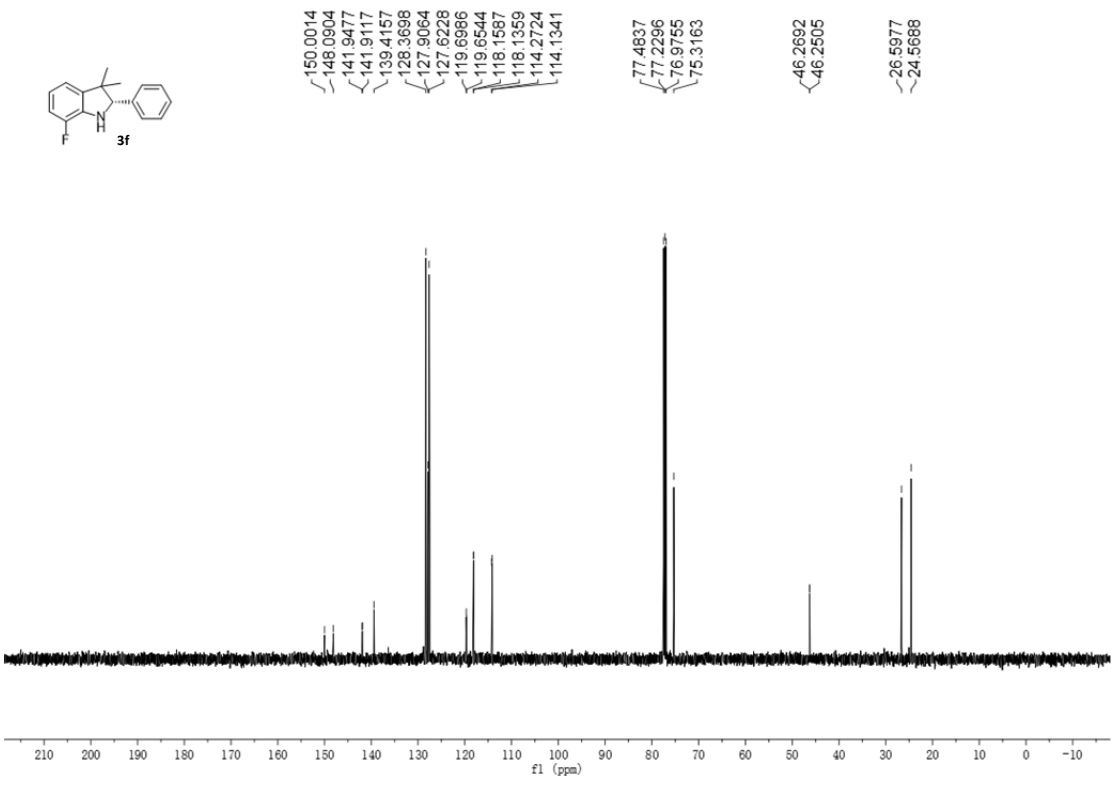
3.01
1.92
0.95
1.00
3.25
3.07
f1 (ppm)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114.2724
114.1341
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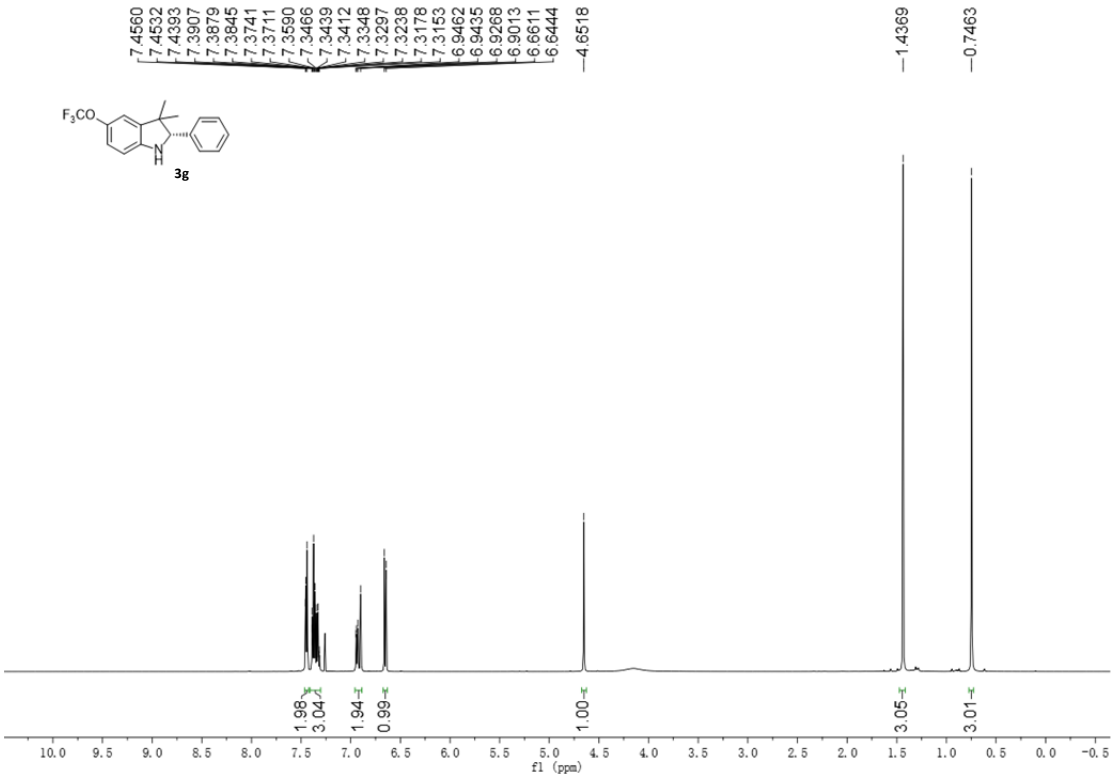
46.2692
46.2505
26.5977
24.5668
3f
f1 (ppm)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7.3879
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7.3439
7.3412
7.3348
7.3297
7.3238
7.3178
7.3153
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6.9435
6.9268
6.9013
6.6611
6.6444
4.6518
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0.7463
F3CO
3g
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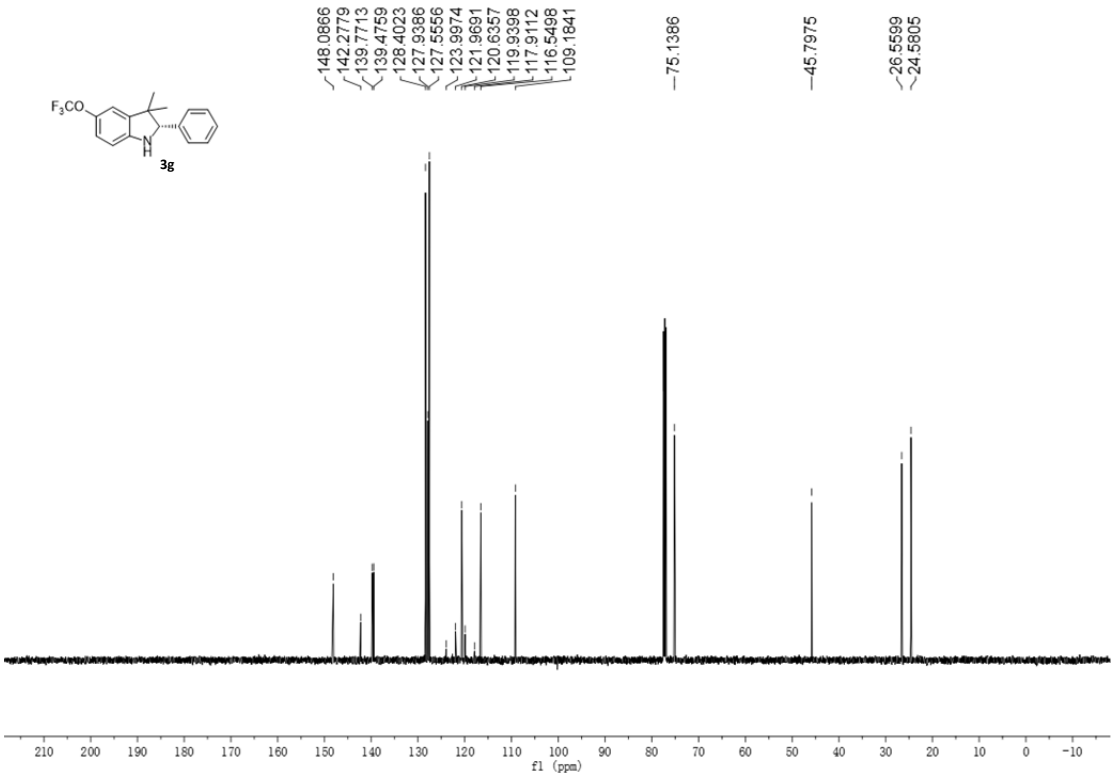
3.04
1.94
0.99
1.00
3.05
3.01
f1 (ppm)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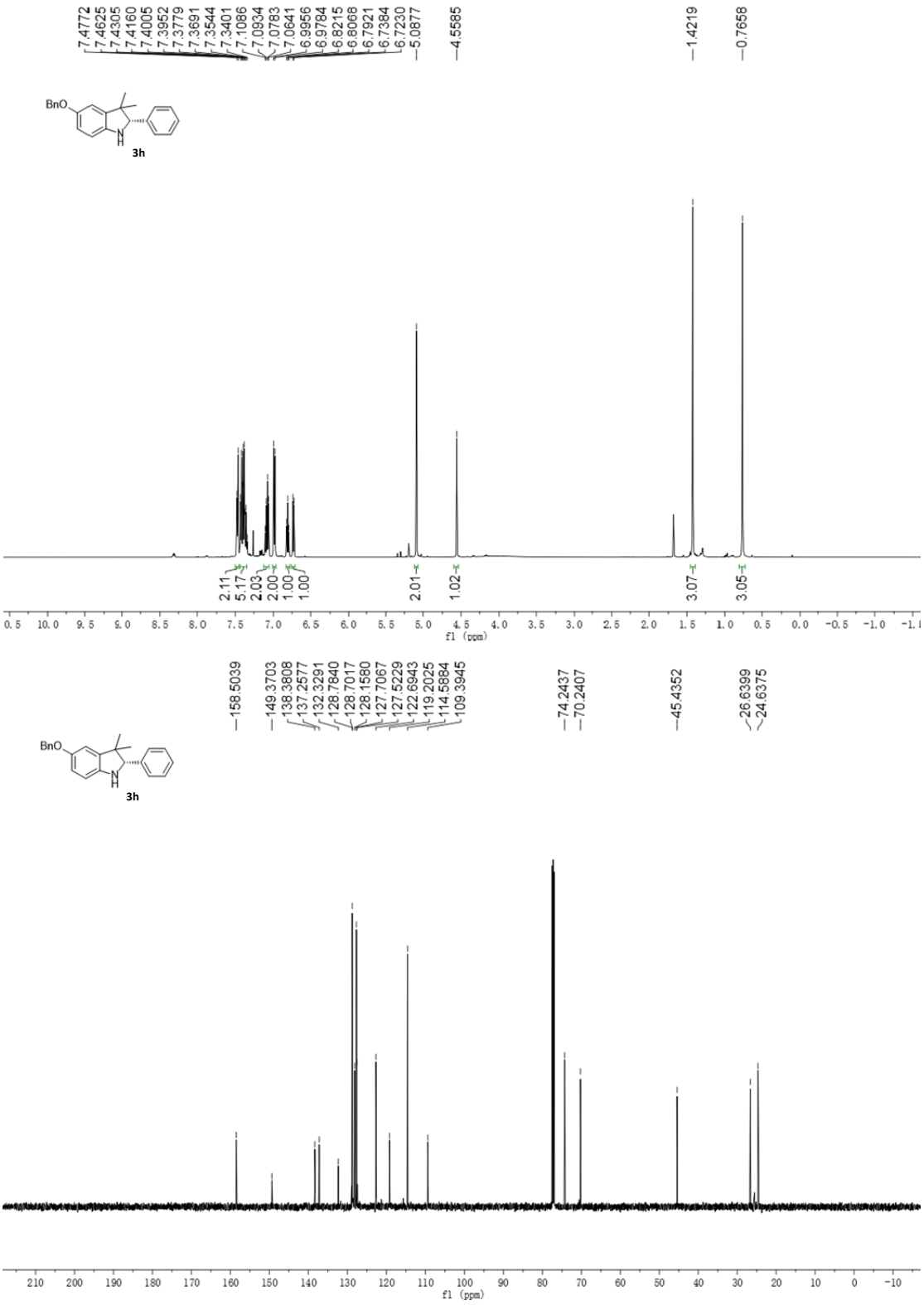
26.5599
24.5805
F3CO
3g
f1 (ppm)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6.9956
6.9784
6.8215
6.8068
6.7921
6.7384
6.7230
5.0877
4.5585
1.4219
0.7658
BnO
3h
2.11
5.17
2.03
2.00
1.00
1.00
2.01
1.02
3.07
3.05
f1 (ppm)
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149.3703
138.3808
137.2577
132.3291
128.7840
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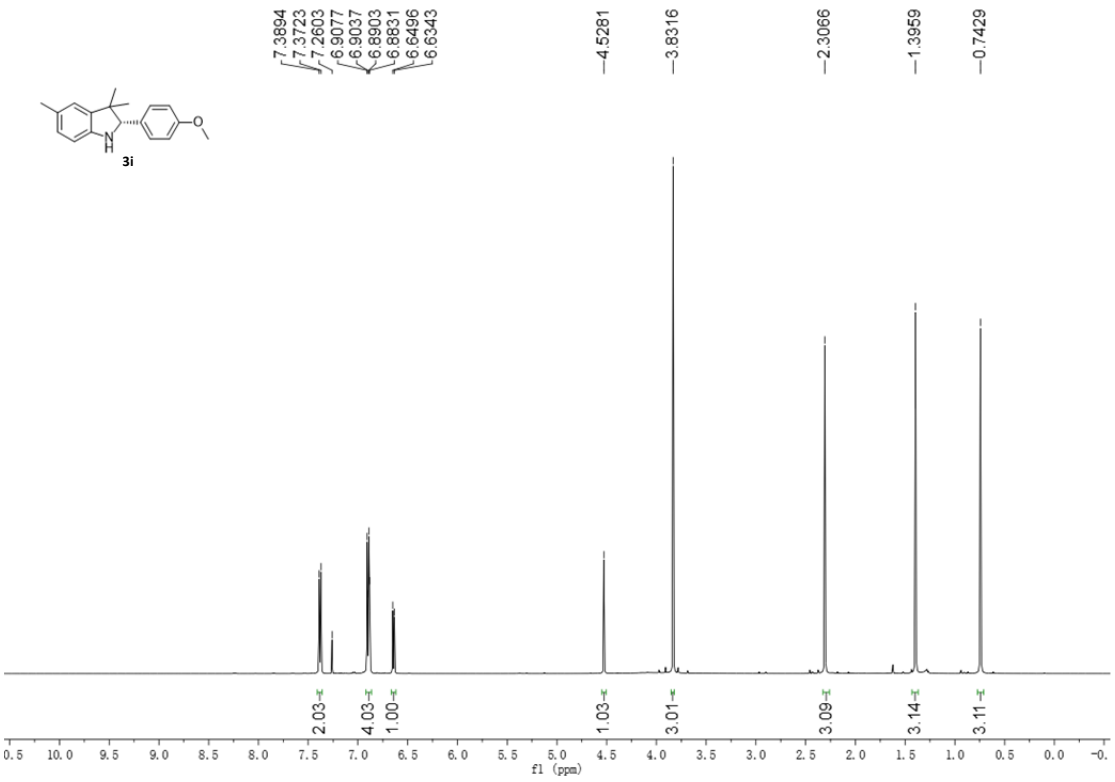
BnO
3h
f1 (ppm)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2.3066
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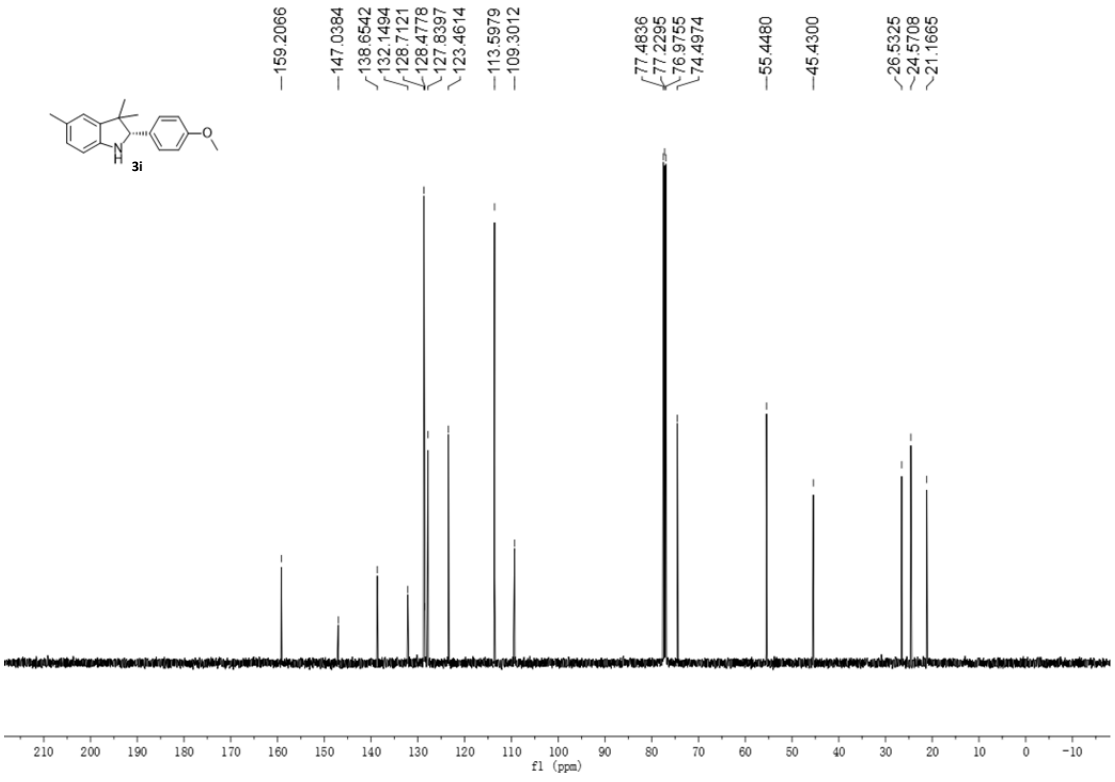
4.03
1.00
1.03
3.01
3.09
3.14
3.11
f1 (ppm)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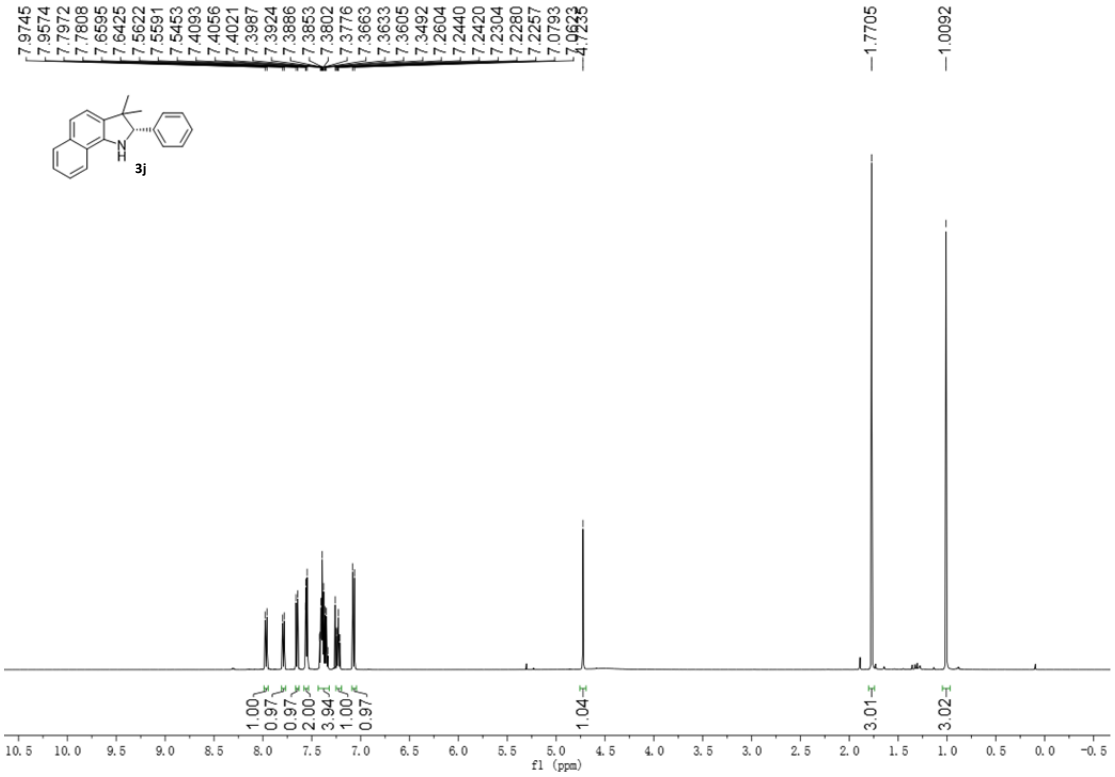
3i
f1 (ppm)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7.3853
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7.3776
7.3663
7.3633
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7.3492
7.2604
7.2440
7.2420
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7.2280
7.2257
7.0793
7.0623
4.1238
1.7705
1.0092
3j
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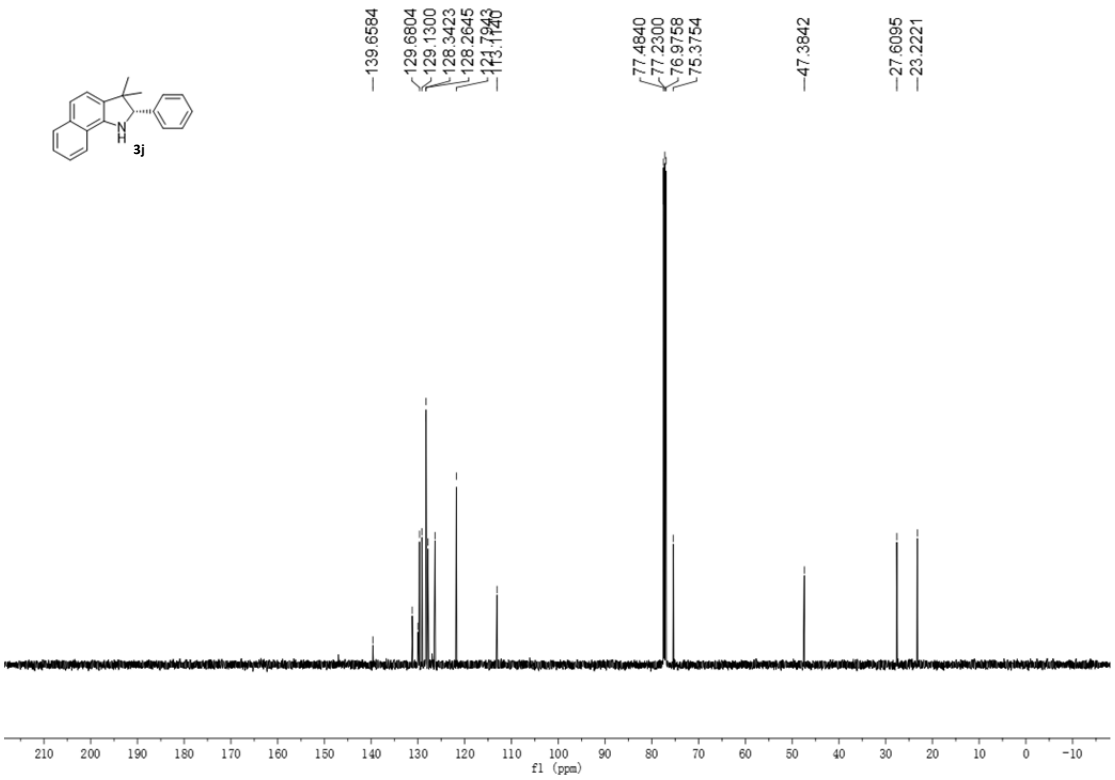
0.97
2.00
3.94
1.00
0.97
1.04
3.01
3.02
f1 (ppm)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27.6095
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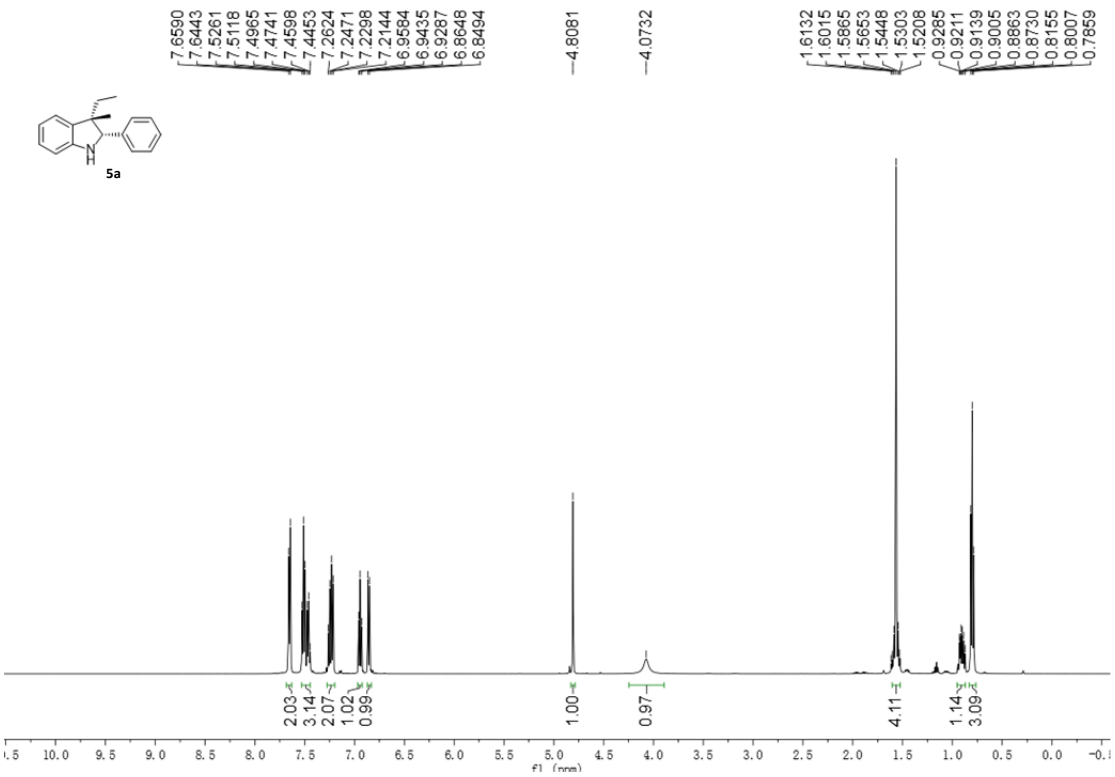
3j
f1 (ppm)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1.6015
1.5865
1.5653
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1.5303
1.5208
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0.9211
0.9139
0.9005
0.8863
0.8730
0.8155
0.8007
0.7859
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2.07
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0.99
1.00
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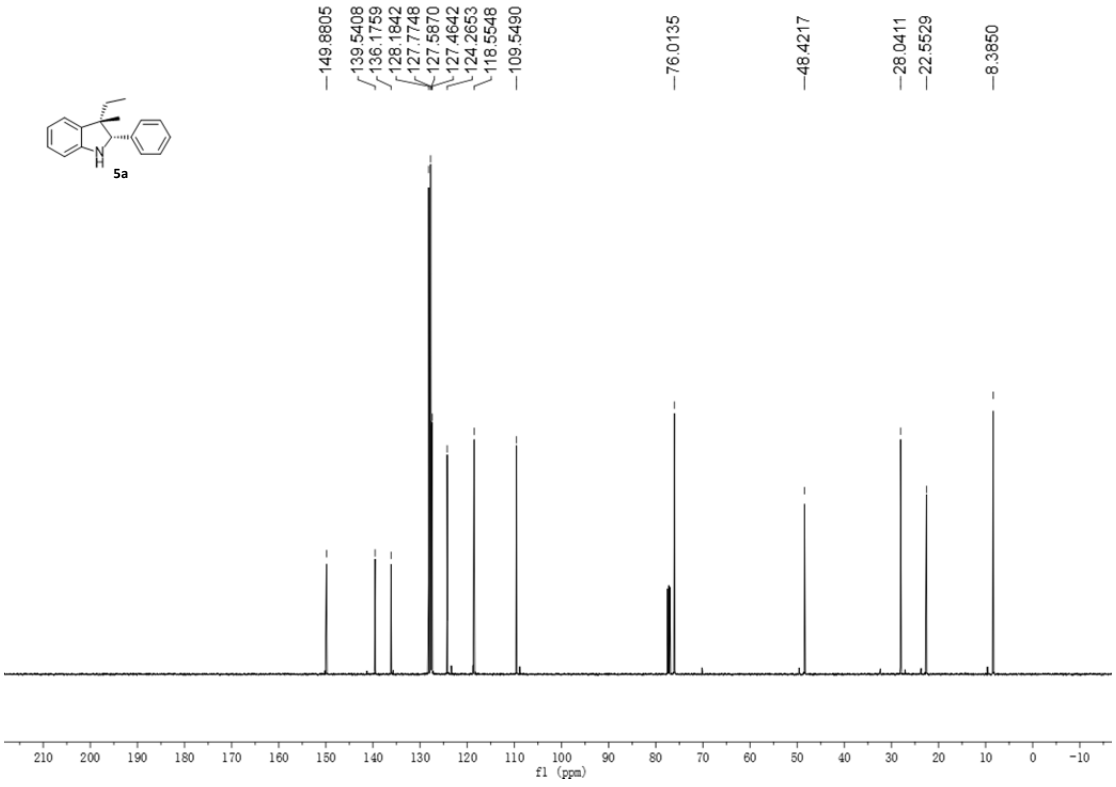
3.09
f1 (ppm)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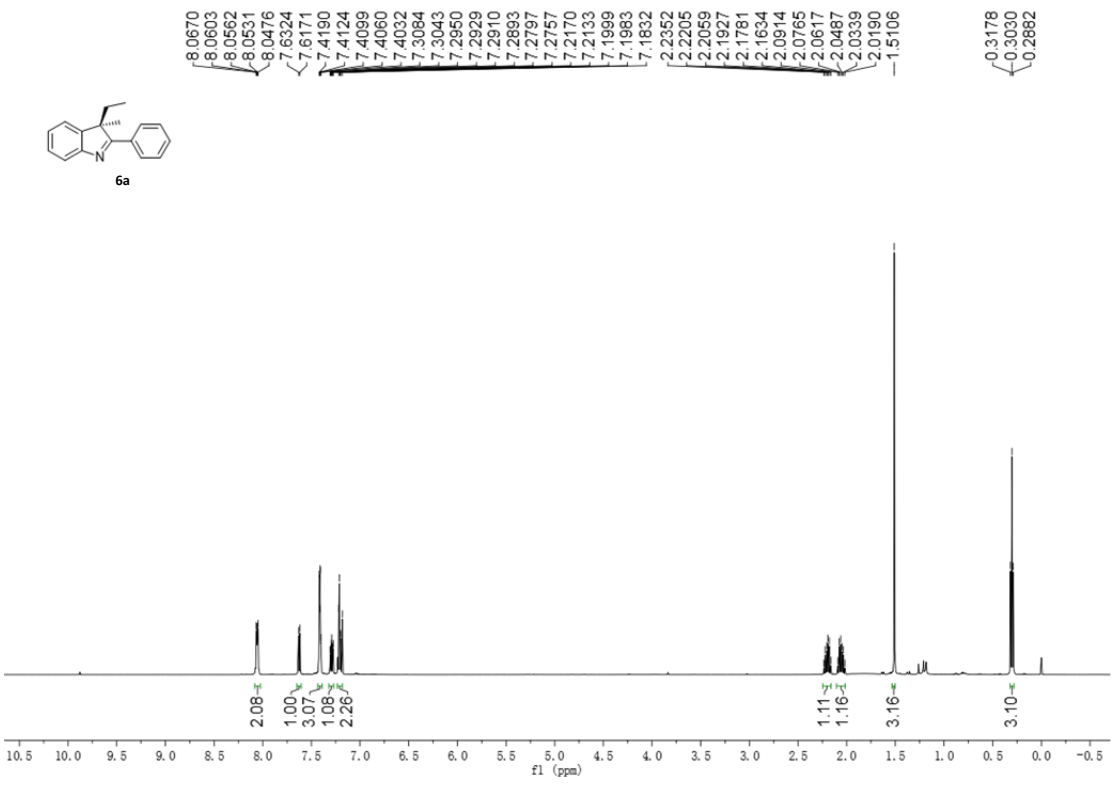
5a
f1 (ppm)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7.3084
7.3043
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7.2133
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7.1983
7.1832
2.2352
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2.2059
2.1927
2.1781
2.1634
2.0914
2.0765
2.0617
2.0487
2.0339
2.0190
1.5106
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0.3030
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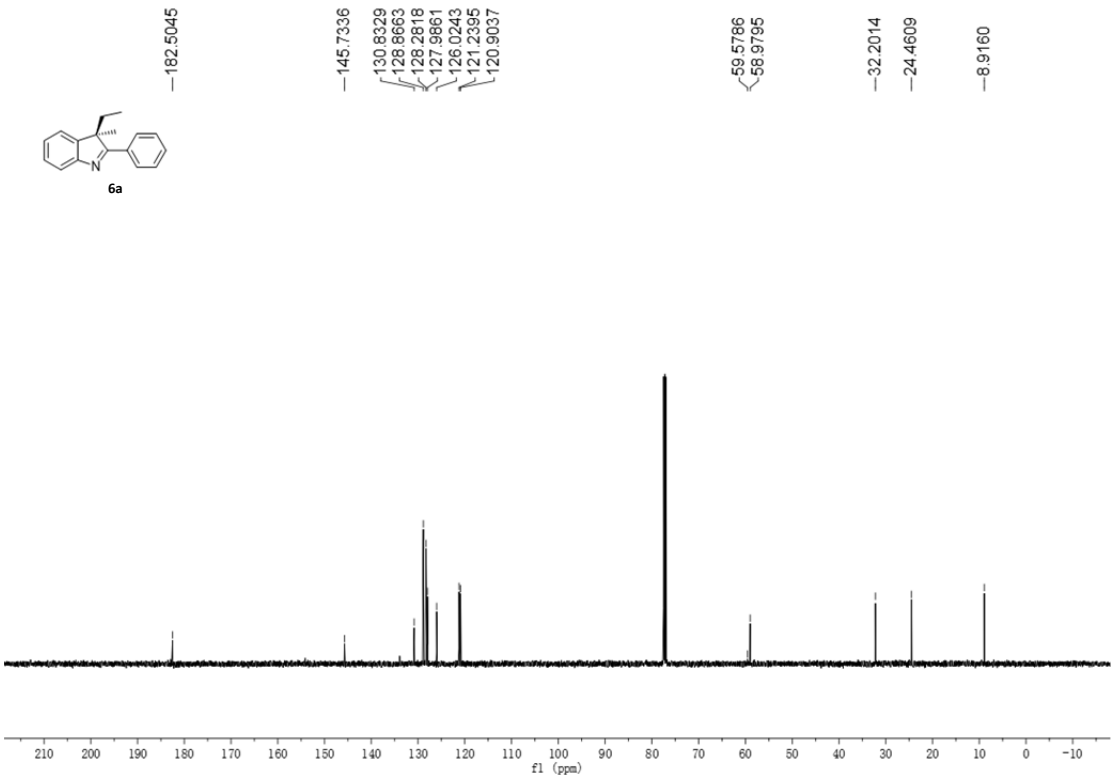
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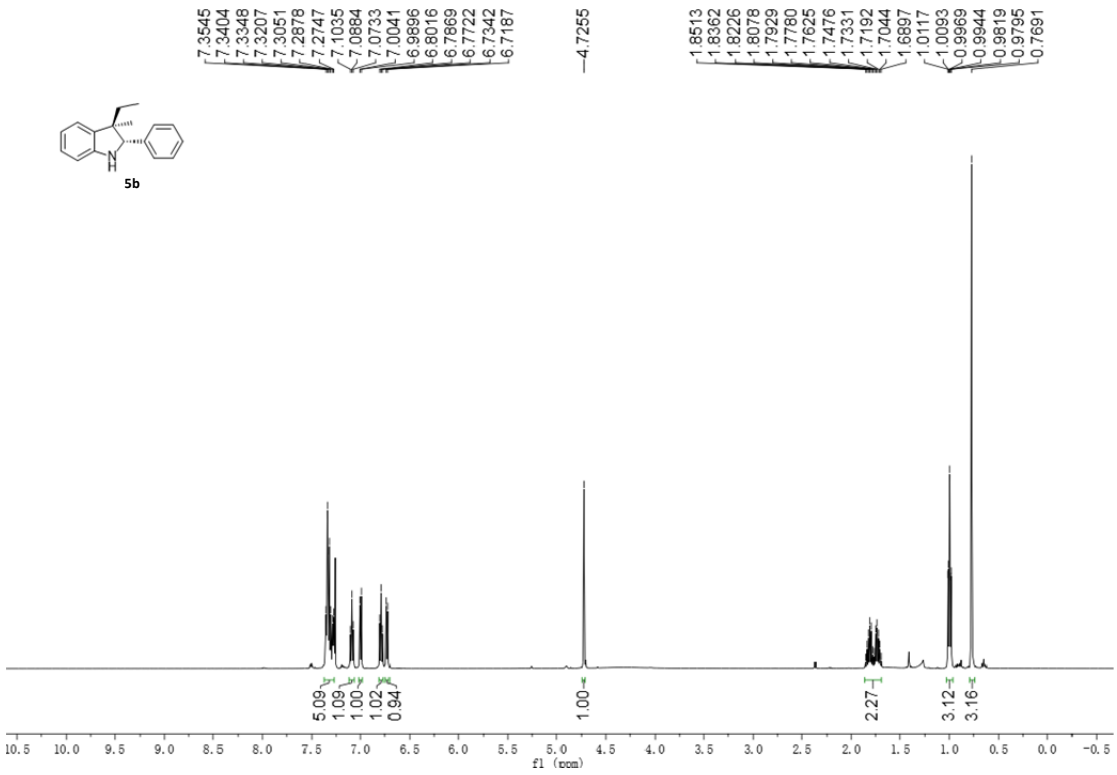
f1 (ppm)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1.7476
1.7331
1.7192
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0.9795
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1.00
2.27
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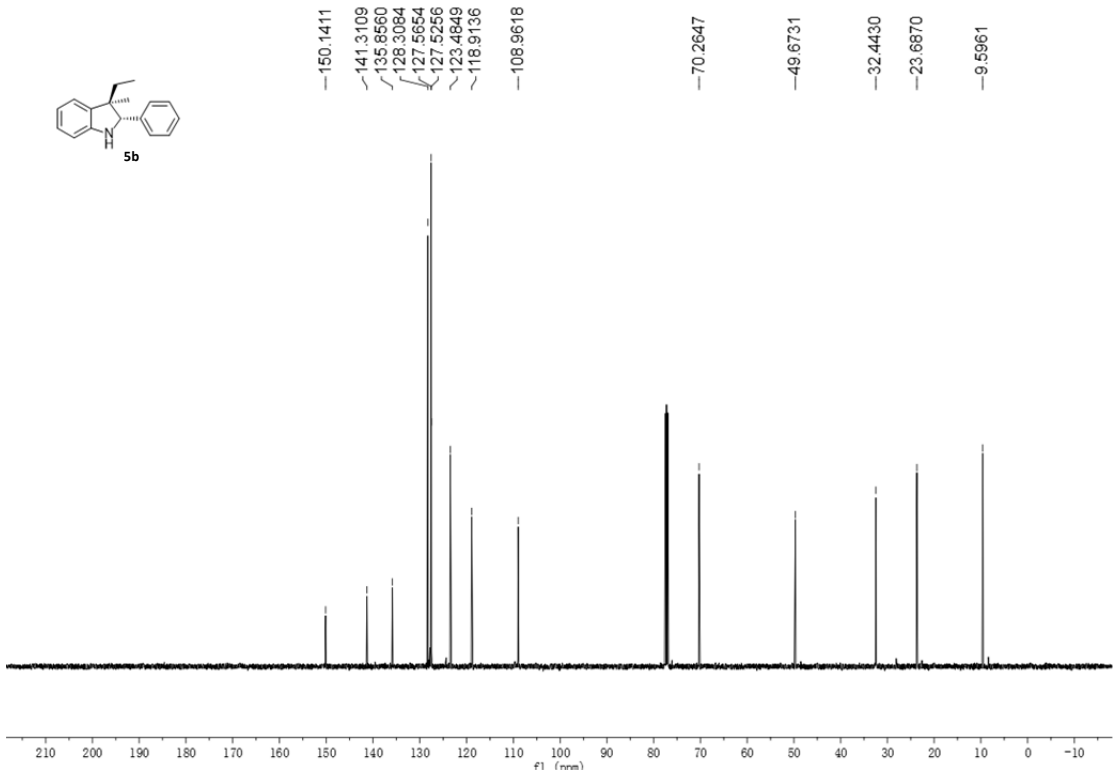
3.16
f1 (ppm)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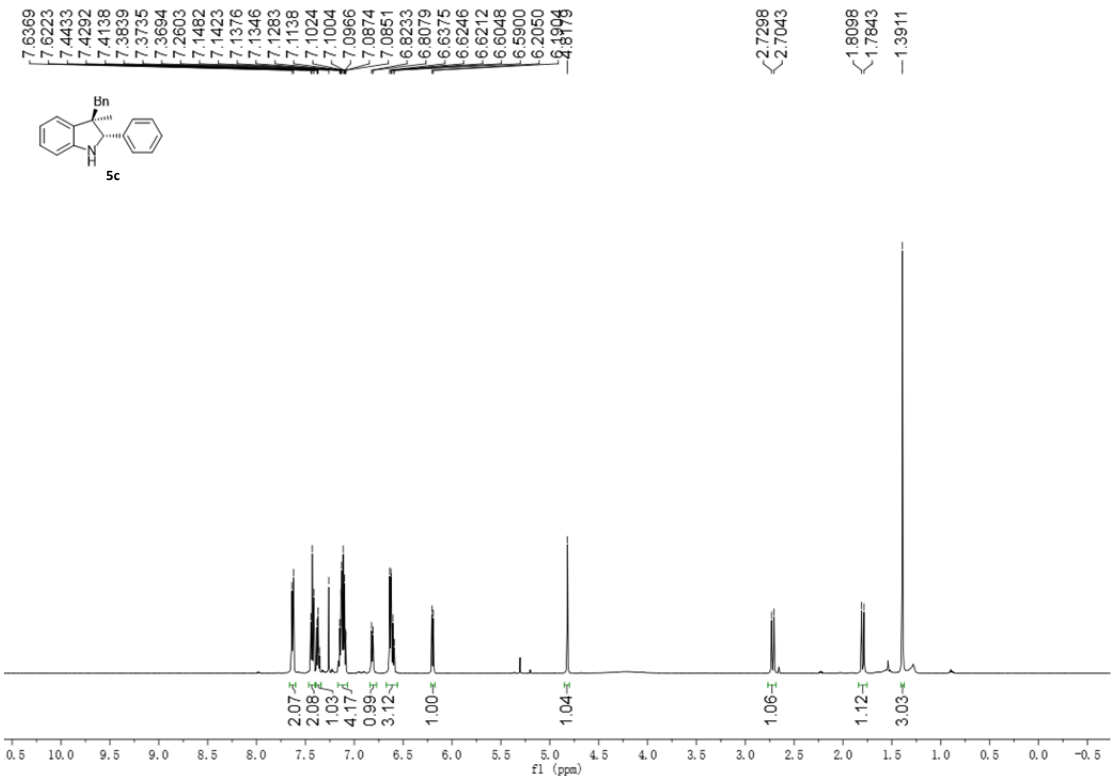
5b
f1 (ppm)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7.1346
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6.6246
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6.2050
6.1904
4.8179
2.7298
2.7043
1.8098
1.7843
1.3911
Bn
5c
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2.08
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4.17
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3.12
1.00
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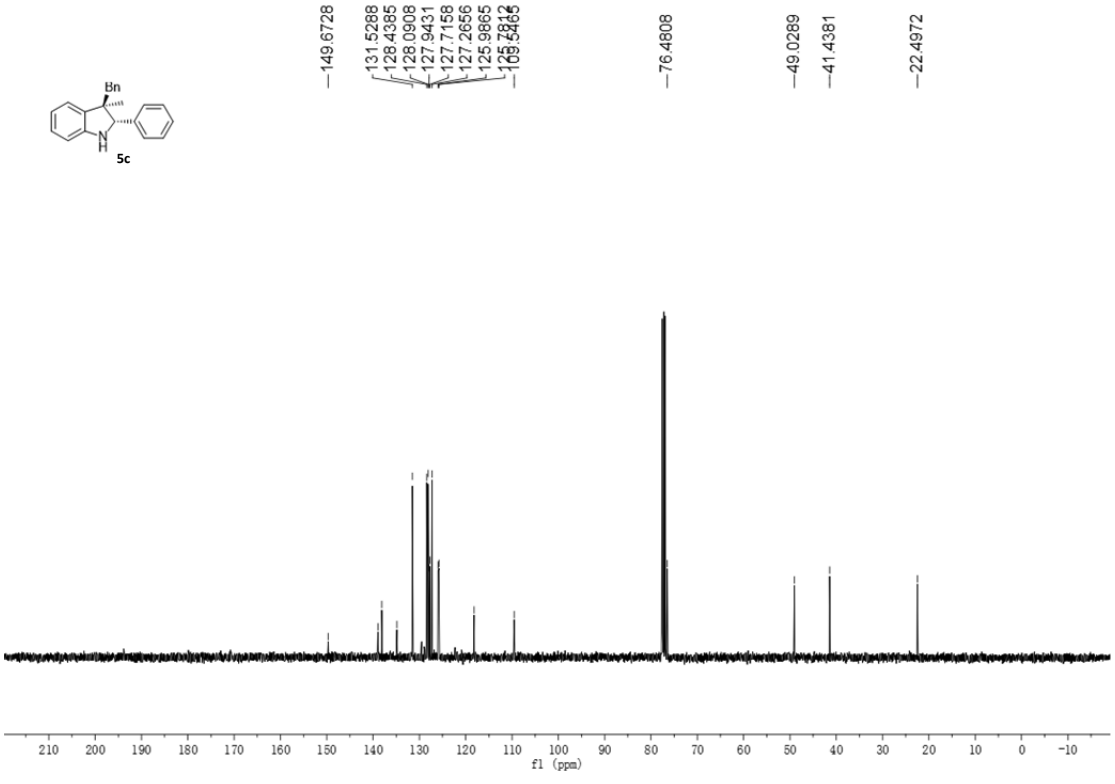
1.06
1.12
3.03
f1 (ppm)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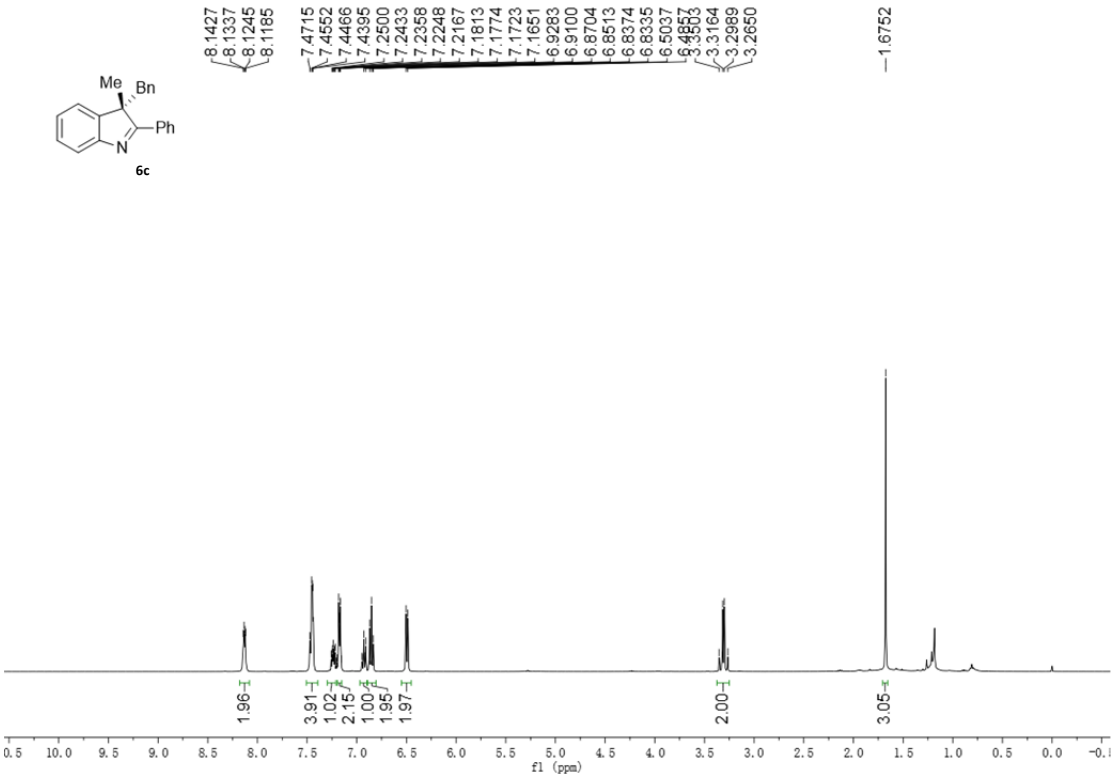
Bn
5c
f1 (ppm)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6.8374
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6.4857
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3.2989
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Me
Bn
Ph
N
6c
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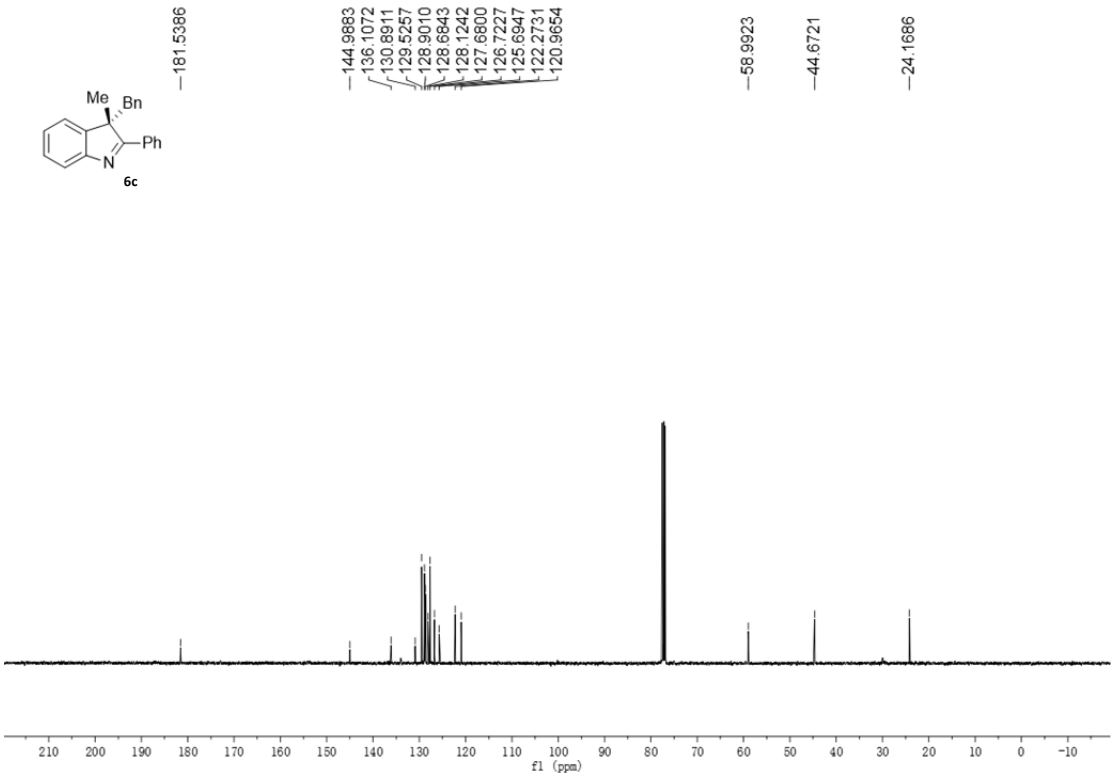
3.91
1.02
2.15
1.00
1.95
1.97
2.00
3.05
f1 (ppm)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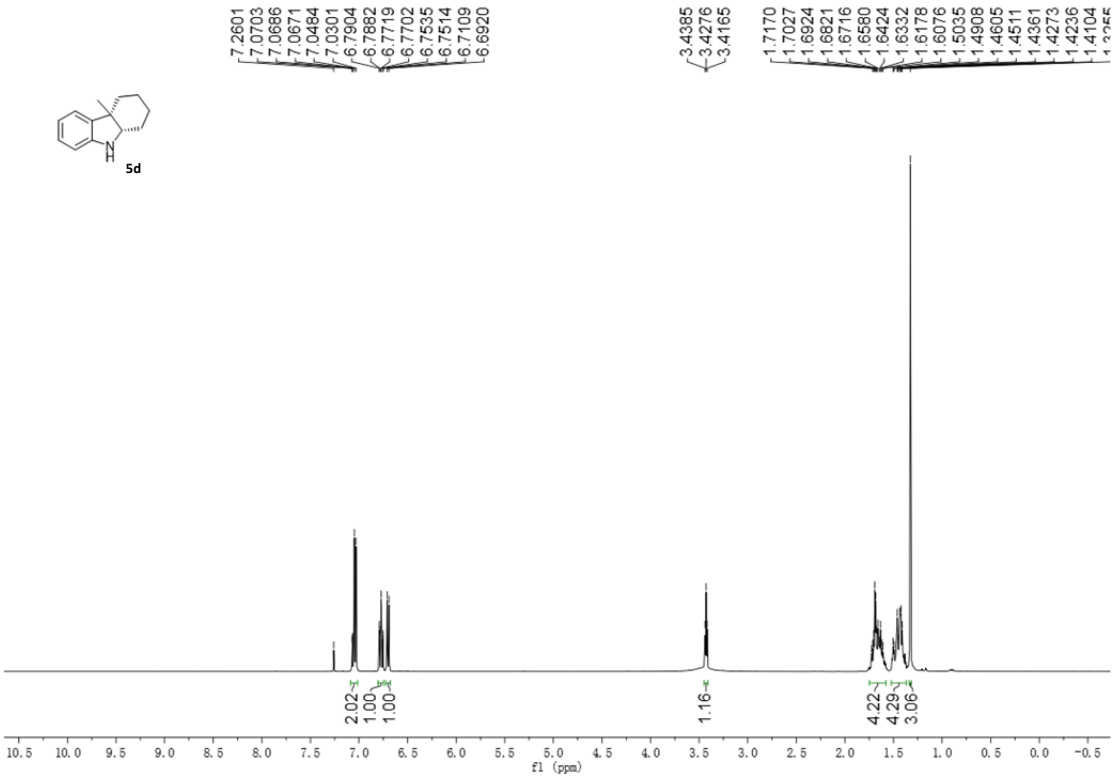
144.9883
136.1072
130.8911
129.5257
128.9010
128.6843
128.1242
127.6800
126.7227
125.6947
122.2731
120.9654
58.9923
44.6721
24.1686
Me
Bn
Ph
N
6c
f1 (ppm)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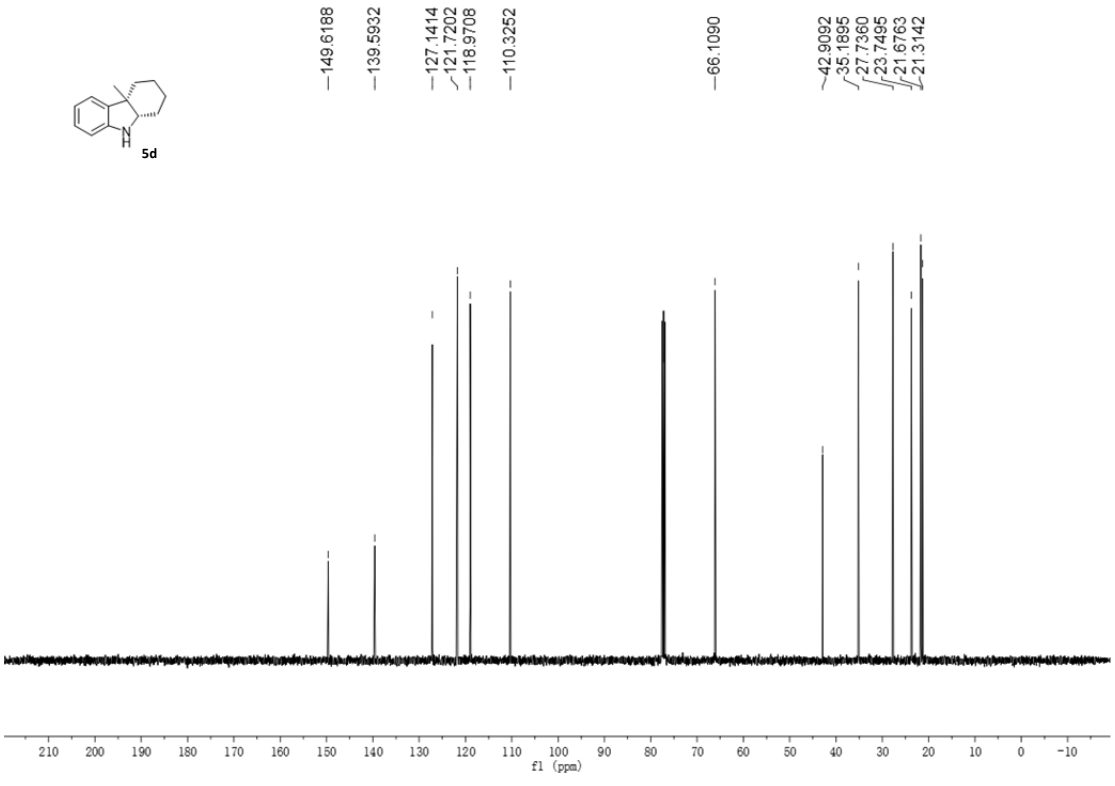
7.0686
7.0671
7.0484
7.0301
6.7904
6.7882
6.7719
6.7702
6.7535
6.7514
6.7109
6.6920
3.4385
3.4276
3.4165
1.7170
1.7027
1.6924
1.6821
1.6716
1.6580
1.6424
1.6332
1.6178
1.6076
1.5035
1.4908
1.4605
1.4511
1.4361
1.4273
1.4236
1.4104
5d
2.02
1.00
1.00
1.16
4.22
4.29
3.06
f1 (ppm)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127.1414
121.7202
118.9708
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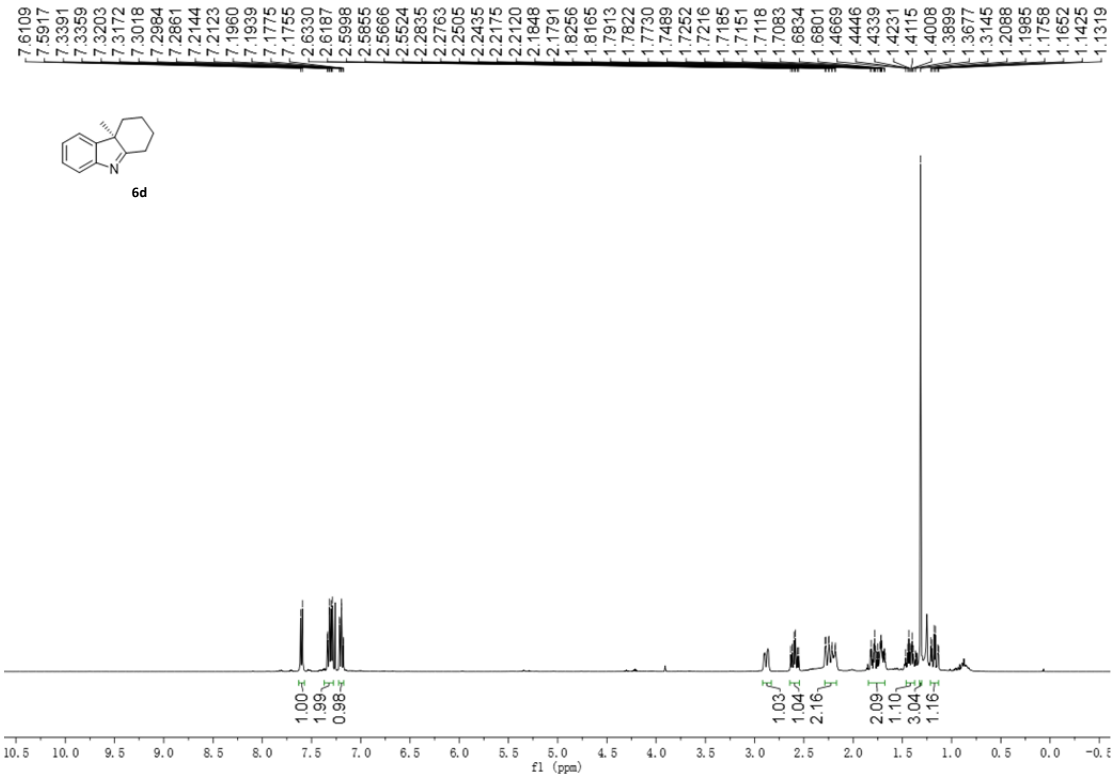
66.1090
42.9092
35.1895
27.7360
23.7495
21.6763
21.3142
5d
f1 (ppm)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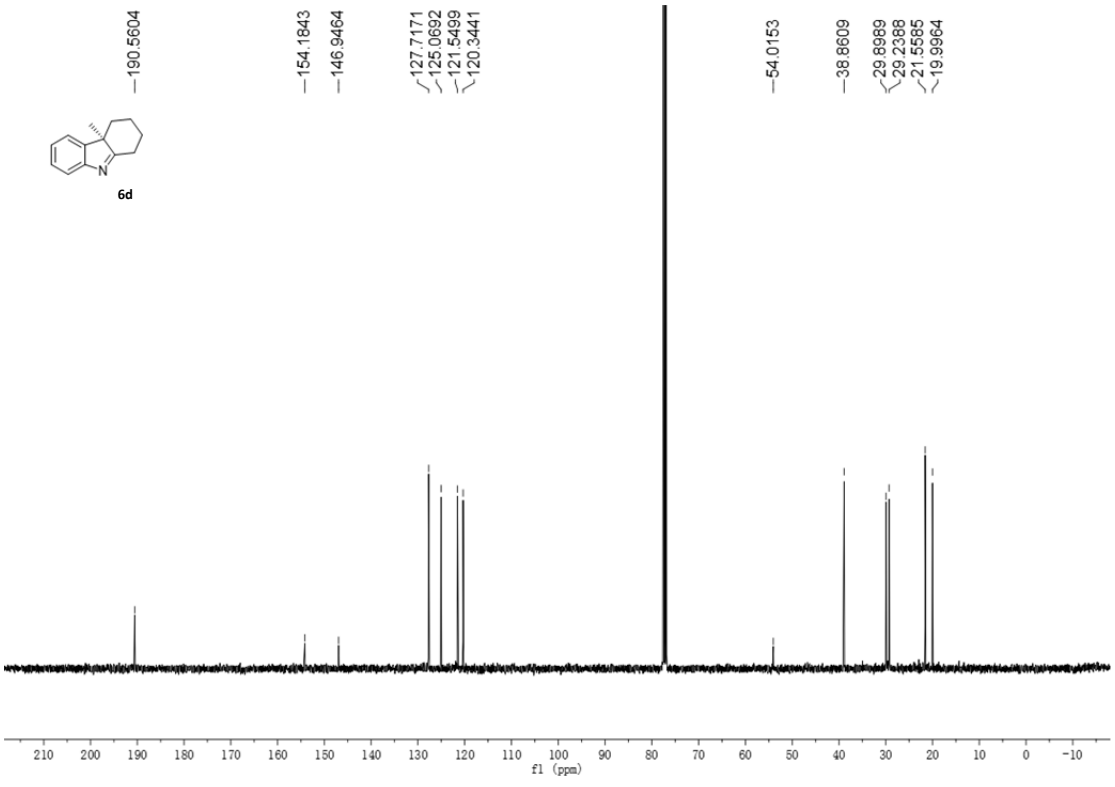
7.3359
7.3203
7.3172
7.3018
7.2984
7.2861
7.2144
7.2123
7.1960
7.1939
7.1775
7.1755
2.6330
2.6187
2.5998
2.5855
2.5666
2.5524
2.2835
2.2763
2.2505
2.2435
2.2175
2.2120
2.1848
2.1791
1.8256
1.8165
1.7913
1.7822
1.7730
1.7489
1.7252
1.7216
1.7185
1.7151
1.7118
1.7083
1.6834
1.6801
1.4669
1.4446
1.4339
1.4231
1.4115
1.4008
1.3899
1.3677
1.3145
1.2088
1.1985
1.1758
1.1652
1.1425
1.1319
6d
1.00
1.99
0.98
1.03
1.04
2.16
2.09
1.10
3.04
1.16
f1 (ppm)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146.9464
127.7171
125.0692
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120.3441
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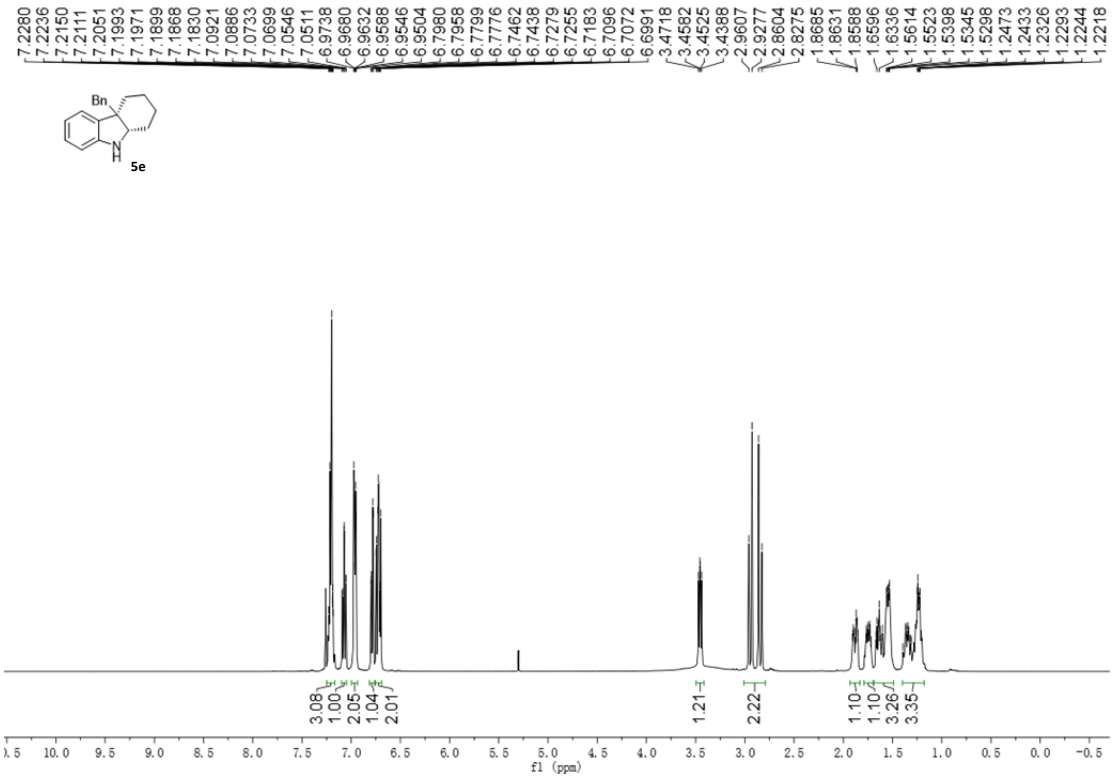
38.8609
29.8989
29.2388
21.5585
19.9964
6d
f1 (ppm)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7.2236
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7.2111
7.2051
7.1993
7.1971
7.1899
7.1868
7.1830
7.0921
7.0886
7.0733
7.0699
7.0546
7.0511
6.9738
6.9680
6.9632
6.9588
6.9546
6.9504
6.7980
6.7958
6.7799
6.7776
6.7462
6.7438
6.7279
6.7255
6.7183
6.7096
6.7072
6.6991
3.4718
3.4582
3.4525
3.4388
2.9607
2.9277
2.8604
2.8275
1.8685
1.8631
1.8588
1.6596
1.6336
1.5614
1.5523
1.5398
1.5345
1.5298
1.2473
1.2433
1.2326
1.2293
1.2244
1.2218
Bn
N
H
5e
3.08
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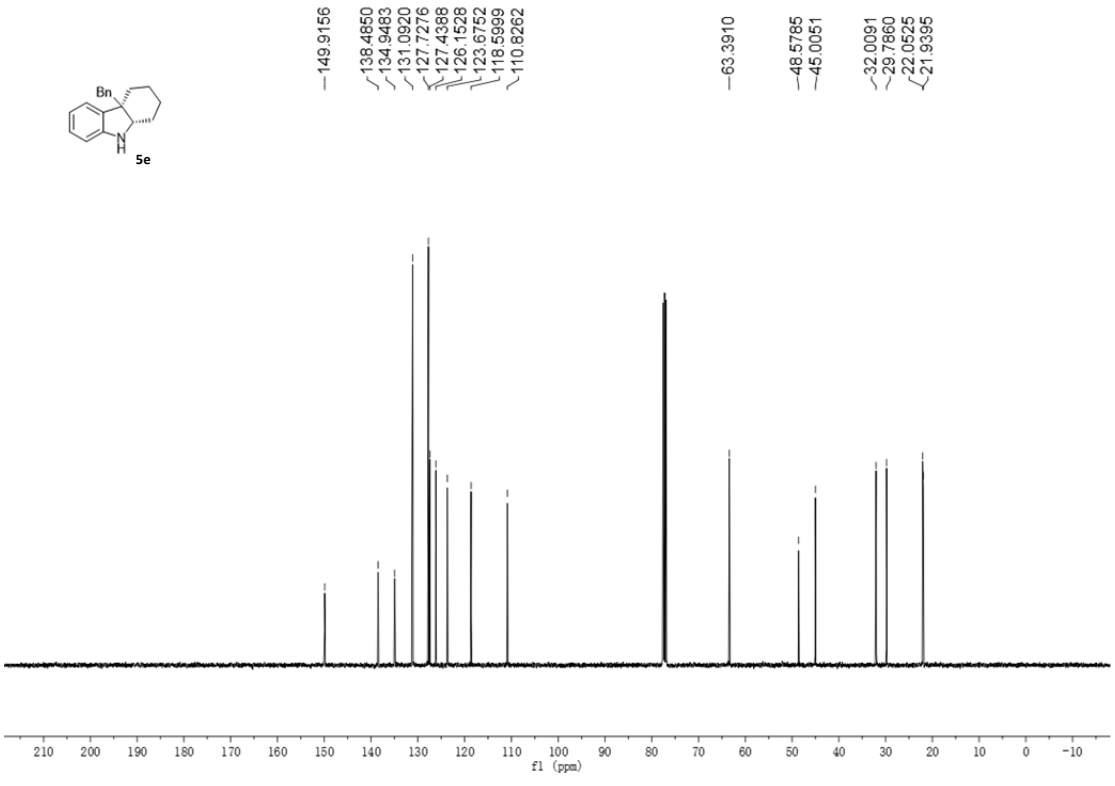
1.10
1.10
3.26
3.35
f1 (ppm)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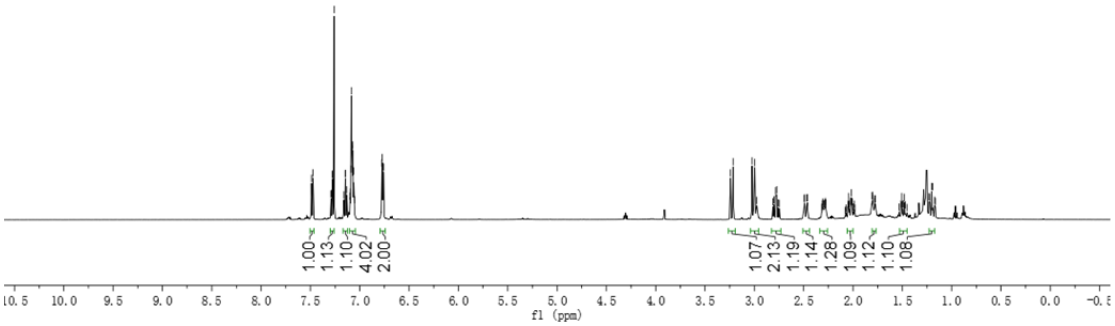
134.9483
131.0920
127.7276
127.4388
126.1528
123.6752
118.5999
110.8262
63.3910
48.5785
45.0051
32.0091
29.7860
22.0525
21.9395
Bn
N
H
5e
f1 (ppm)


7.4889
7.4735
7.2925
7.2901
7.2774
7.2751
7.2600
7.1606
7.1469
7.1458
7.1311
7.0978
7.0954
7.0876
7.0844
7.0803
7.0769
7.0699
7.0624
7.0522
6.7767
6.7738
6.7620
6.7581
3.2428
3.2157
3.0235
2.9964
2.9751
2.8116
2.8002
2.7849
2.7735
2.4954
2.4902
2.4685
2.4630
2.3063
2.2842
2.2799
2.0434
2.0232
2.0159
2.0085
1.8056
1.8030
1.7981
1.7825
1.7774
1.7749
1.5064
1.4797
1.2286
1.2205
1.2012
1.1932
1.1738
1.1658

Bn
N

6e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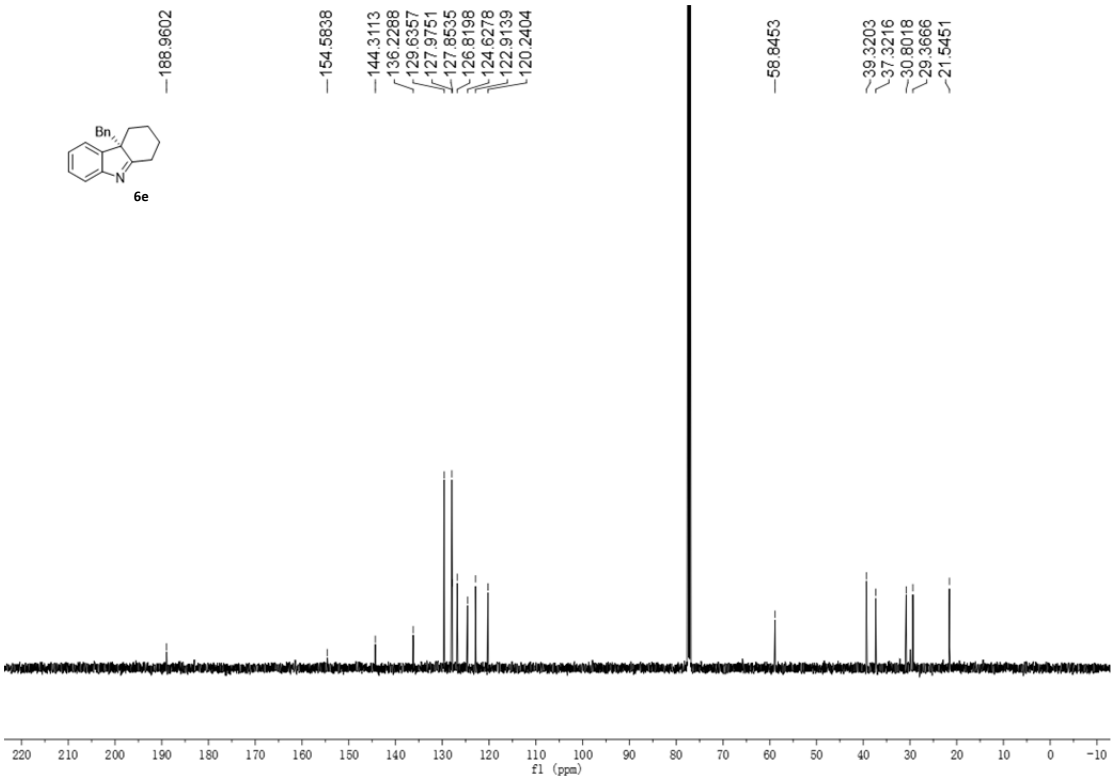
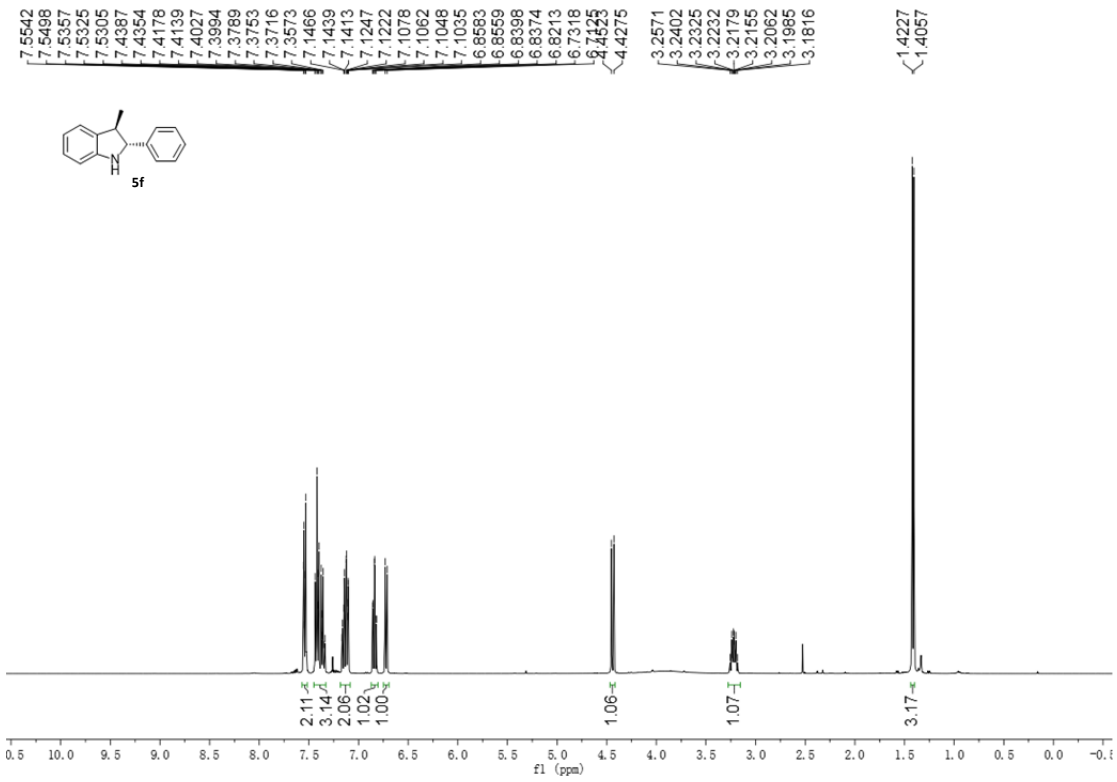
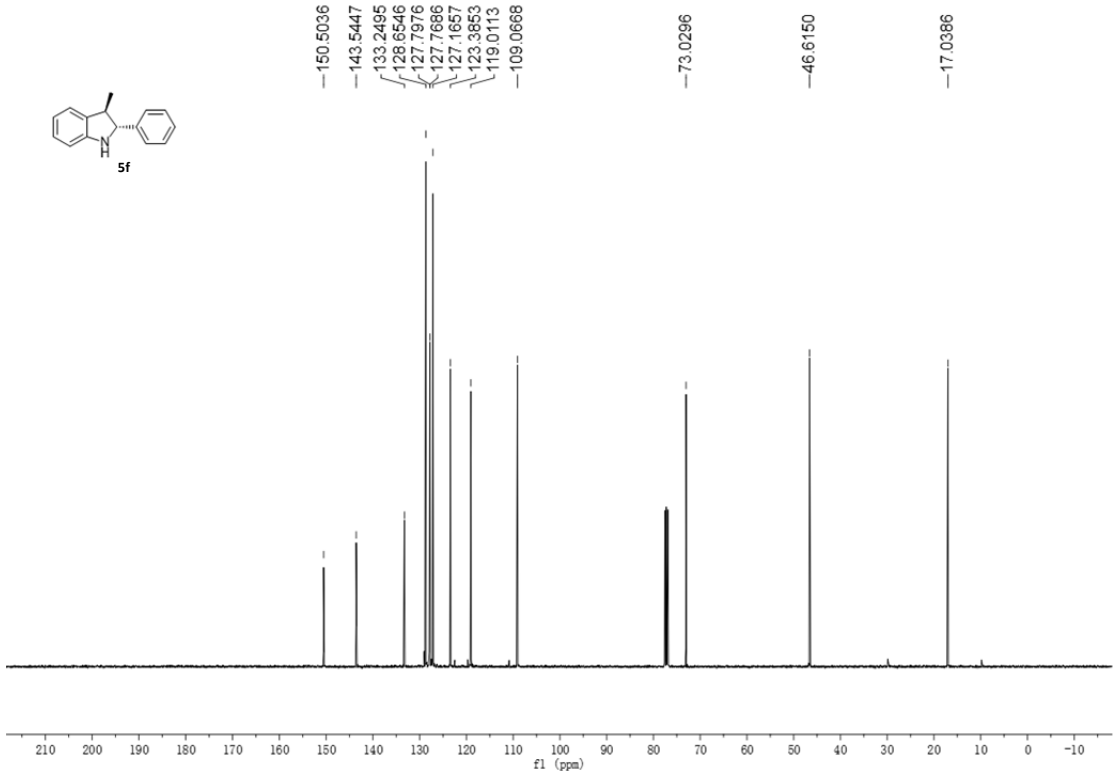
1.13
1.10
4.02
2.00
1.07
2.13
1.19
1.14
1.28
1.09
1.12
1.10
1.08
f1 (ppm)


188.9602
154.5838
144.3113
136.2288
129.6357
127.9751
127.8535
126.8198
124.6278
122.9139
120.2404
58.8453
39.3203
37.3216
30.8018
29.3666
21.5451
f1 (ppm)

Bn
N

6e


7.5542
7.5498
7.5357
7.5325
7.5305
7.4387
7.4354
7.4178
7.4139
7.4027
7.3994
7.3789
7.3753
7.3716
7.3573
7.1466
7.1439
7.1413
7.1247
7.1222
7.1078
7.1062
7.1048
7.1035
6.8583
6.8559
6.8398
6.8374
6.8213
6.7318
4.4275
3.2571
3.2402
3.2325
3.2232
3.2179
3.2155
3.2062
3.1985
3.1816
1.4227
1.4057
5f
2.11
3.14
2.06
1.02
1.00
1.06
1.07
3.17
f1 (ppm)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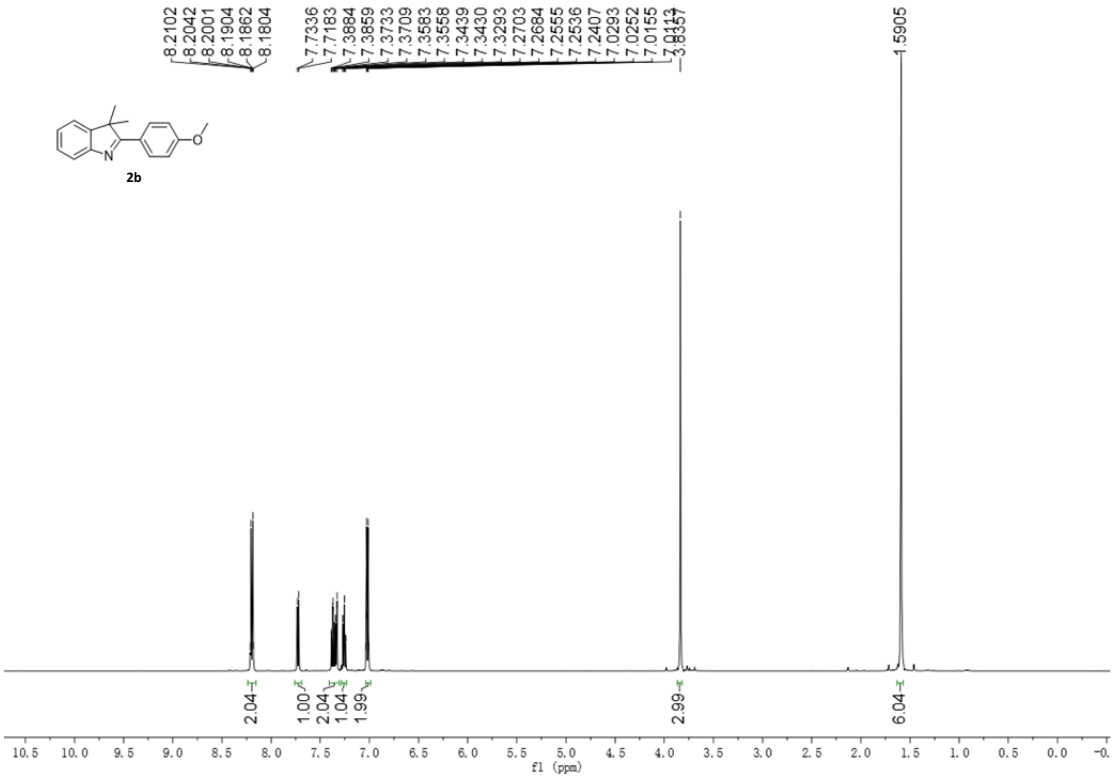
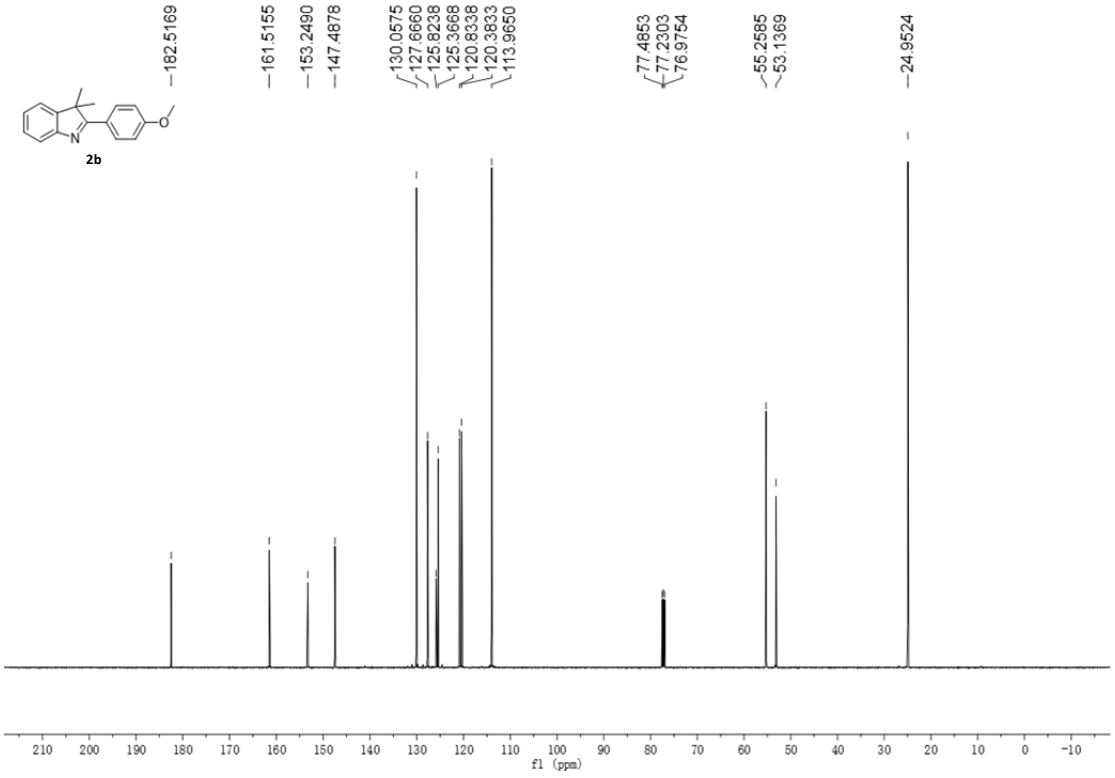
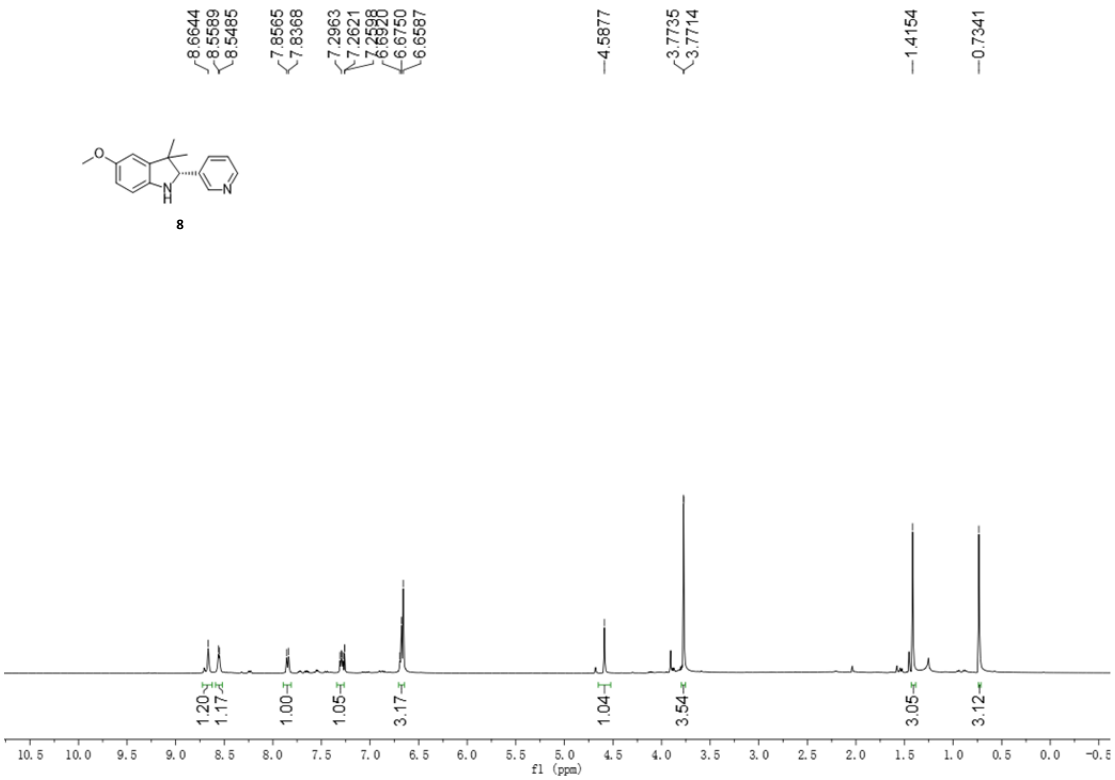
133.2495
128.6546
127.7976
127.7686
127.1657
123.3853
119.0113
109.0668
73.0296
46.6150
17.0386
5f
f1 (ppm)


8.2102
8.2042
8.2001
8.1904
8.1862
8.1804
7.7336
7.7183
7.3884
7.3859
7.3733
7.3709
7.3583
7.3558
7.3439
7.3430
7.3293
7.2703
7.2684
7.2555
7.2536
7.2407
7.0293
7.0252
7.0155
3.8357
1.5905
2b
2.04
1.00
2.04
1.04
1.99
2.99
6.04
f1 (ppm)


182.5169
161.5155
153.2490
147.4878
130.0575
127.6660
125.8238
125.3668
120.8338
120.3833
113.9650
77.4853
77.2303
76.9754
55.2585
53.1369
24.9524
2b
f1 (ppm)


8.6644
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8.5485
7.8565
7.8368
7.2963
7.2621
7.2598
6.6920
6.6750
6.6587
4.5877
3.7735
3.7714
1.4154
0.7341
1.20
1.17
1.00
1.05
3.17
1.04
3.54
3.05
3.12
f1 (ppm)
8
154.1979
149.1220
149.0837
142.8878
139.4456
135.7787
135.5549
123.4220
112.3970
110.1504
109.8601
77.5471
77.2296
76.9118
56.1800
46.0746
26.2062
24.5839
f1 (ppm)
8


7.2999
7.2931
7.2803
7.2719
7.2627
7.2598
7.1274
7.1252
7.1121
7.1100
7.0969
7.0948
6.7989
6.7974
6.7840
6.7825
6.7692
6.7676
6.6941
6.6787
4.6139
4.1246
2.8508
2.8467
2.8369
2.8276
2.8230
2.8155
2.6644
2.6475
2.5238
2.5175
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2.5004
2.4945
2.4873
2.3536
2.3448
2.0961
2.0909
2.0886
2.0850
2.0675
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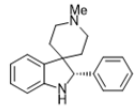
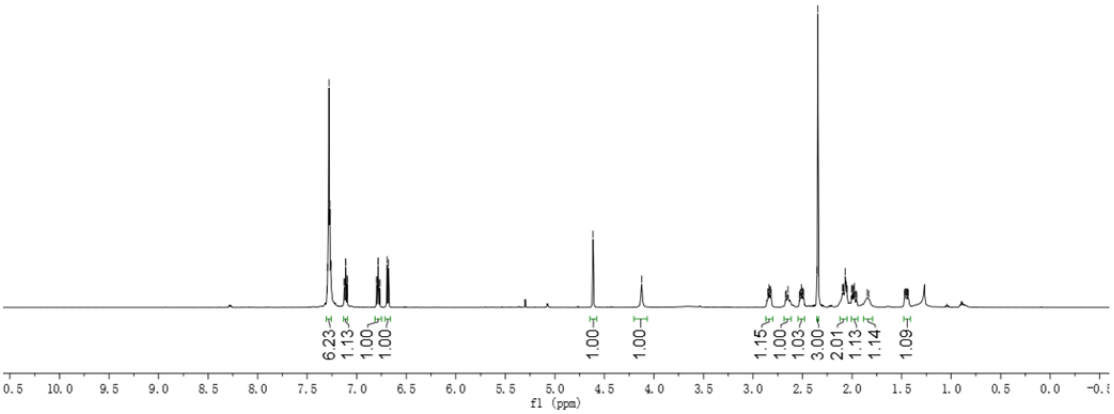

Me
N
H

10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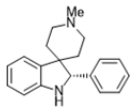
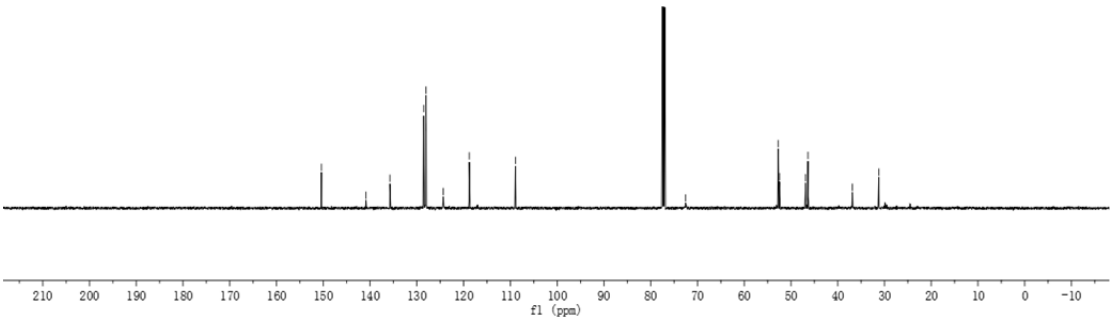
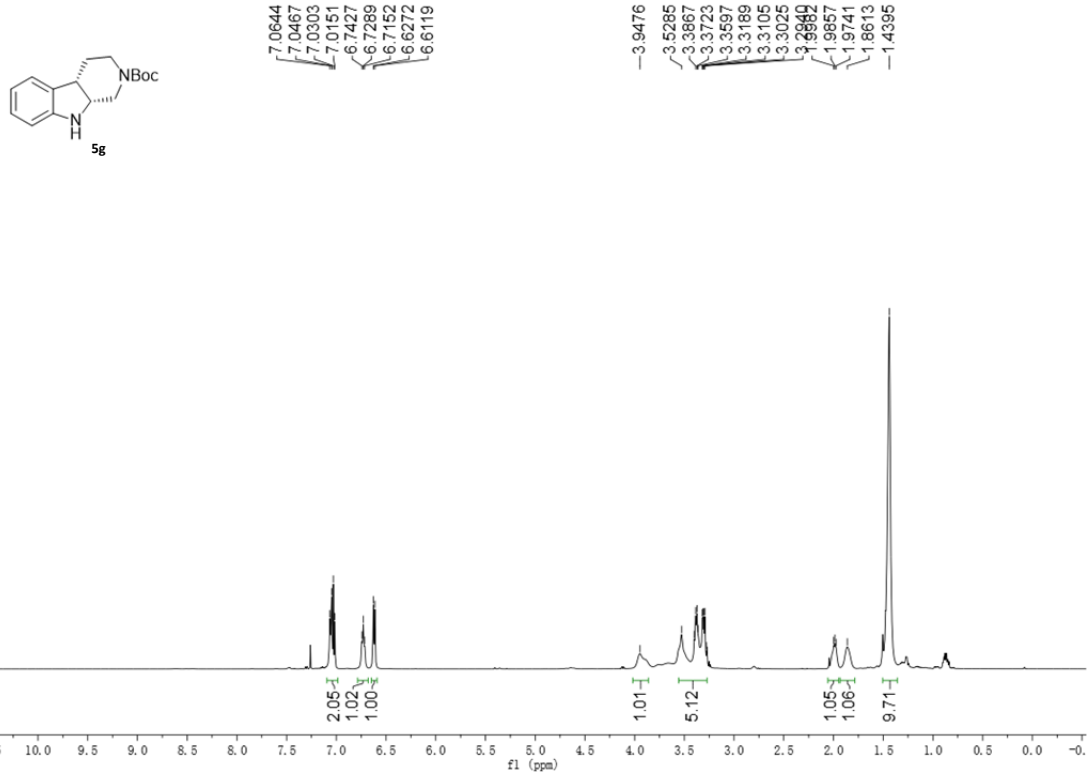
1.00
1.00
1.00
1.00
1.15
1.00
1.03
3.00
2.01
1.13
1.14
1.09
f1 (ppm)


150.3935
140.8751
135.7627
128.5448
128.0738
128.0439
124.3479
118.7906
108.9181
72.5135
52.7045
52.4364
46.8419
46.3470
36.8439
31.2113


Me
N
H

10


f1 (ppm)


NBoc
N
H
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6.7289
6.7152
6.6272
6.6119
3.9476
3.5285
3.3867
3.3723
3.3597
3.3189
3.3105
3.3025
1.9857
1.9741
1.8613
1.4395
2.05
1.02
1.00
1.01
5.12
1.05
1.06
9.71
f1 (ppm)
155.7980
150.7727
131.2199
127.8687
123.7974
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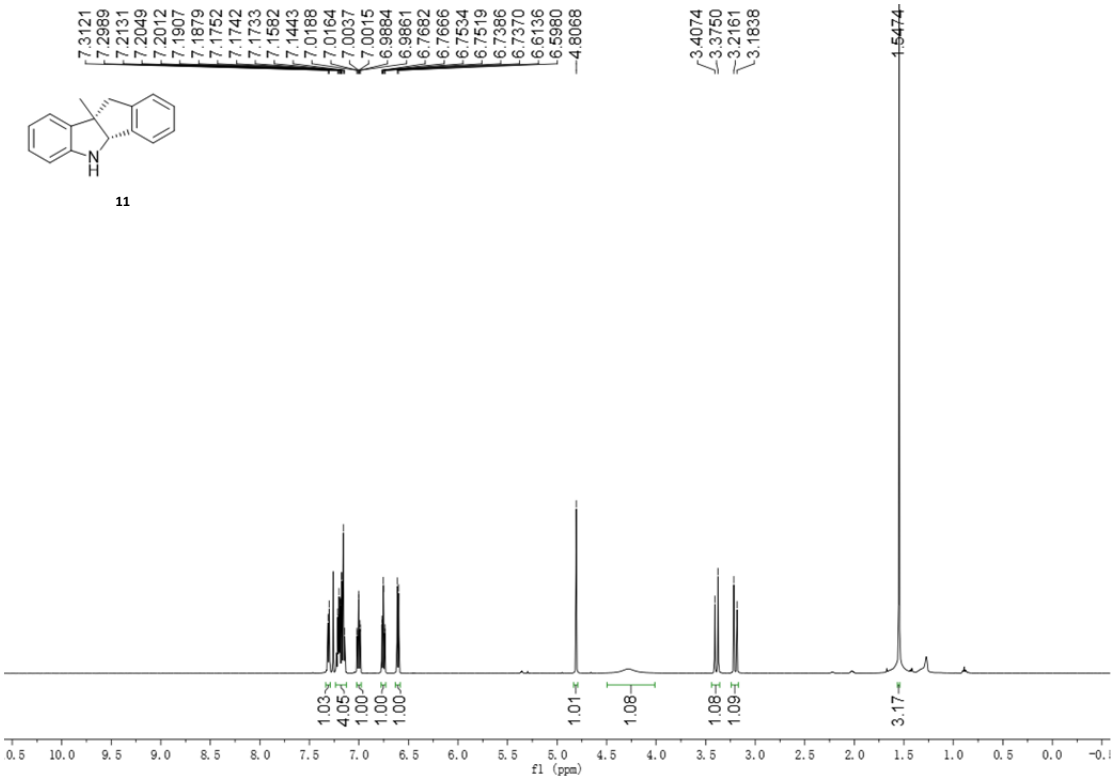
79.6109
57.6078
43.7209
41.4258
39.4313
28.6105
26.2547
f1 (ppm)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7.2131
7.2049
7.2012
7.1907
7.1879
7.1752
7.1742
7.1733
7.1582
7.1443
7.0188
7.0164
7.0037
7.0015
6.9884
6.9861
6.7682
6.7666
6.7534
6.7519
6.7386
6.7370
6.6136
6.5980
4.8068
3.4074
3.3750
3.2161
3.1838
1.5474
11
1.03
4.05
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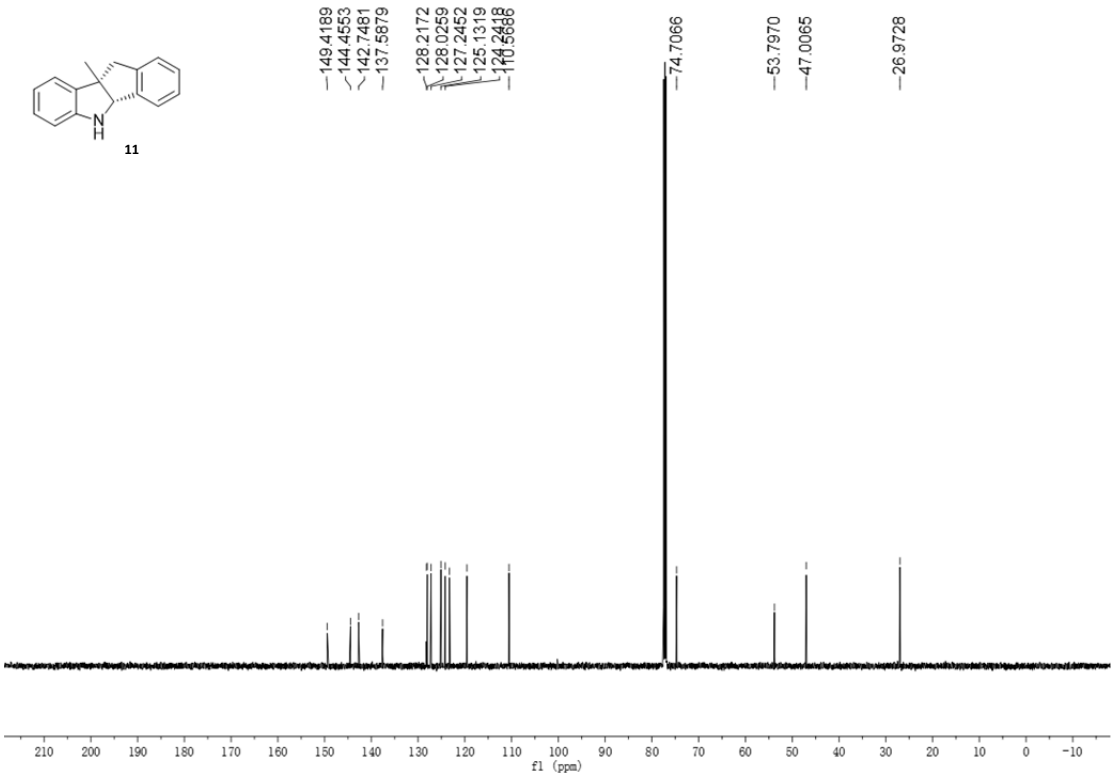
1.08
1.08
1.09
3.17
f1 (ppm)


149.4189
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142.7481
137.5879
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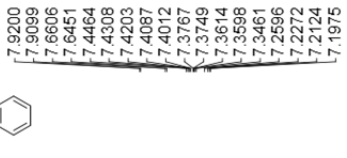
128.0259
127.2452
125.1319
74.7066
53.7970
47.0065
26.9728
11
f1 (ppm)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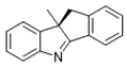
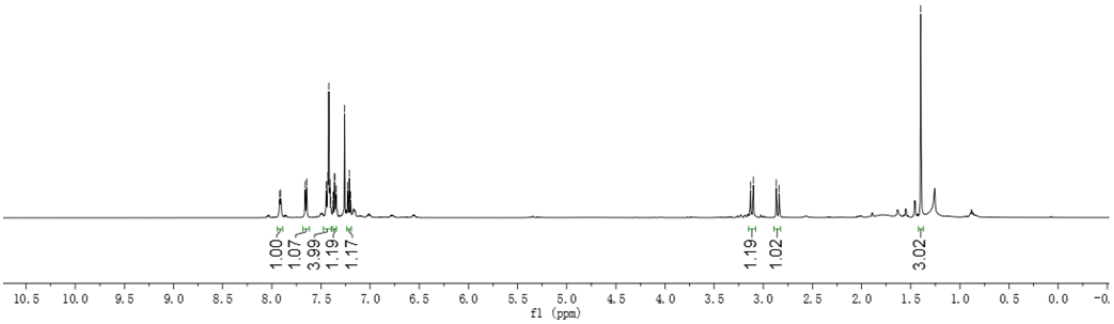
7.4087
7.4012
7.3767
7.3749
7.3614
7.3598
7.3461
7.2596
7.2272
7.2124
7.1975
3.1308
3.1017
2.8675
2.8383
1.3979



12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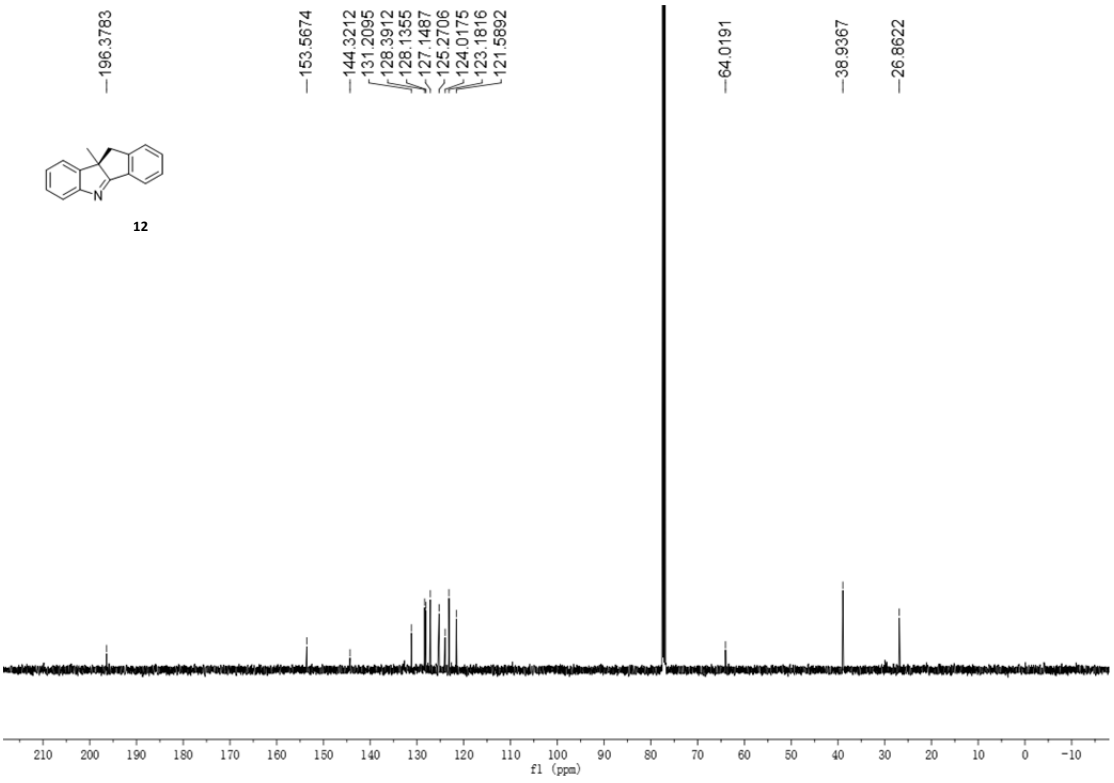
1.17
1.19
1.02
3.02
f1 (ppm)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144.3212
131.2095
128.3912
128.1355
127.1487
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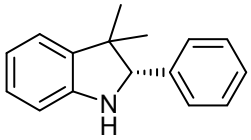
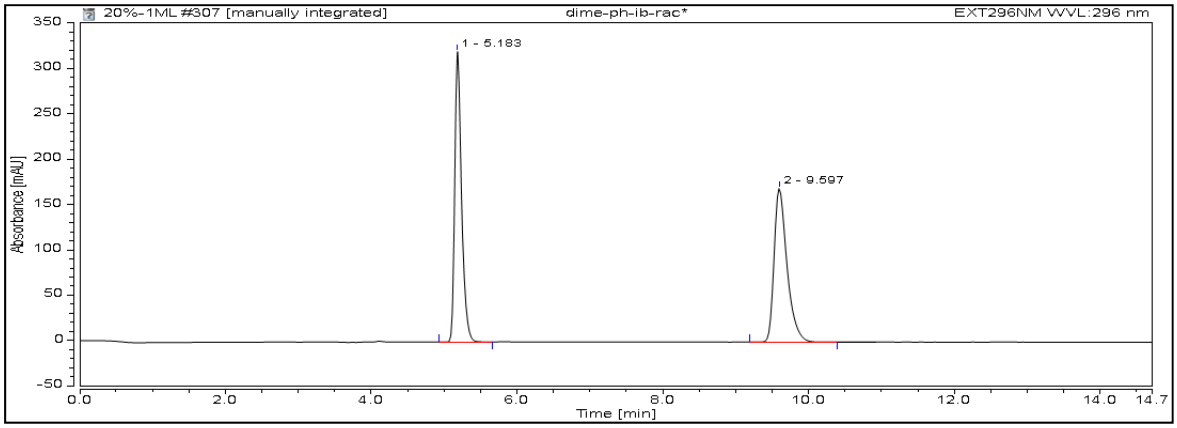
124.0175
123.1816
121.5892
64.0191
38.9367
26.8622
f1 (ppm)

12

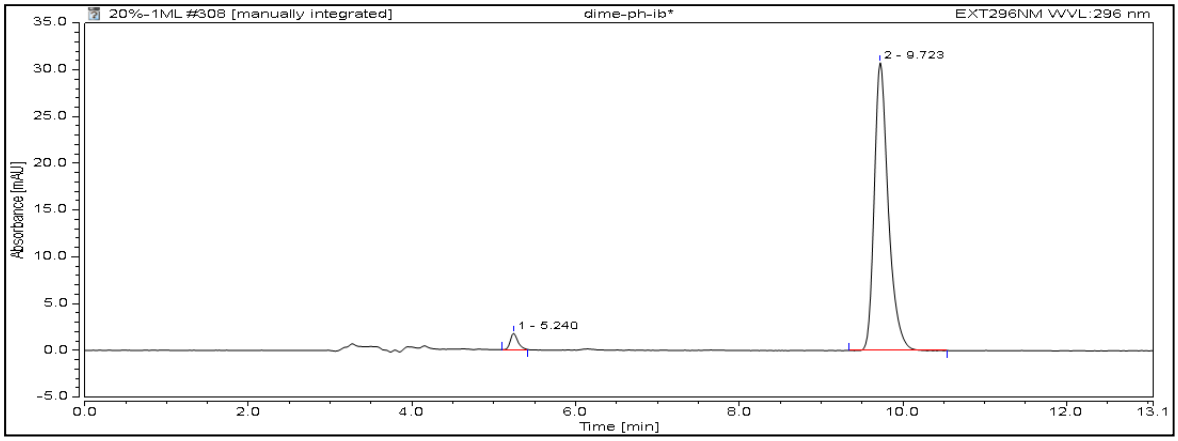
HPLC



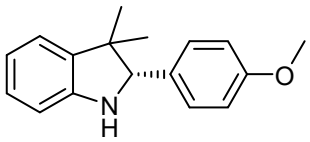
1a



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.183	34.677	49.92	n.a.
2		9.597	34.794	50.08	n.a.
Total:			**69.471**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.240	0.186	2.95	n.a.
2		9.723	6.117	97.05	n.a.
Total:			**6.303**	**100.00**	



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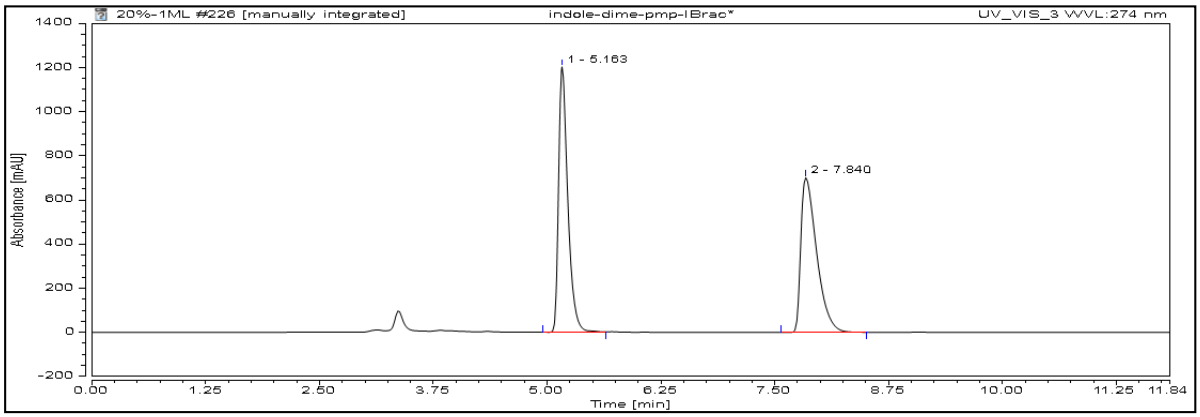
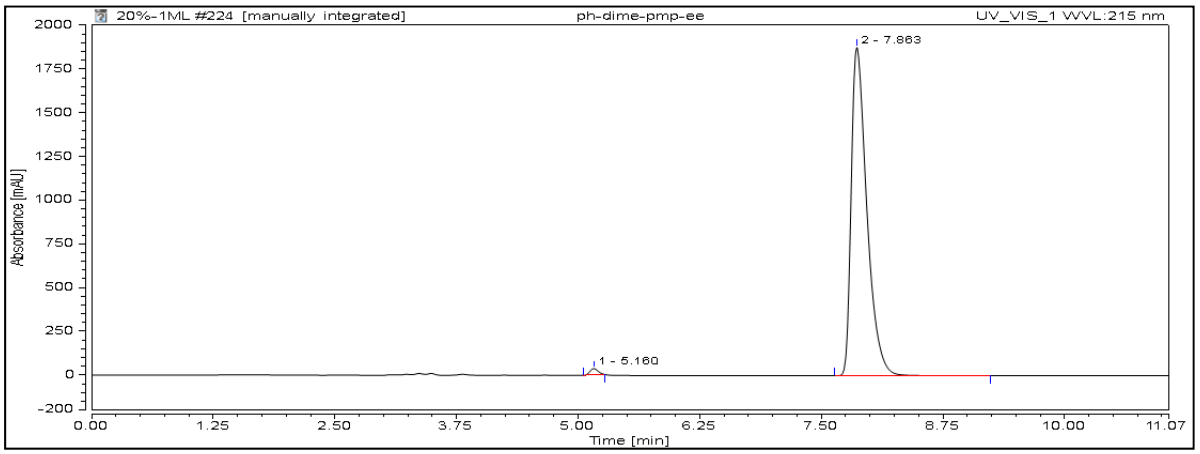
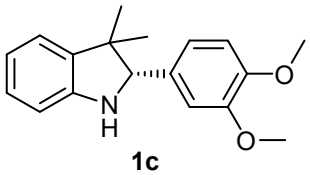


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.163	141.140	50.06	n.a.
2		7.840	140.817	49.94	n.a.
Total:			**281.957**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.160	3.587	1.02	n.a.
2		7.863	349.349	98.98	n.a.
Total:			**352.936**	**100.00**	



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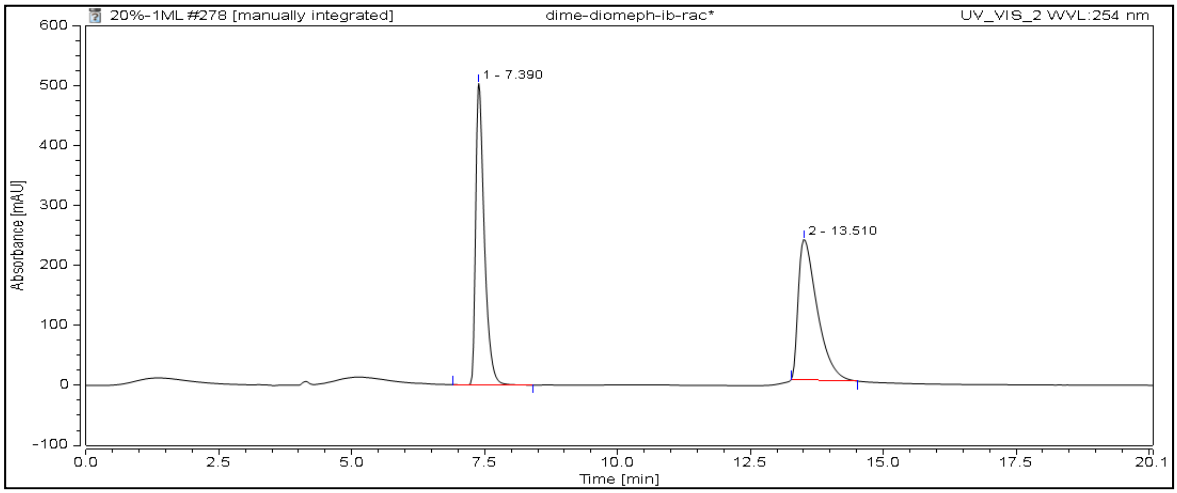
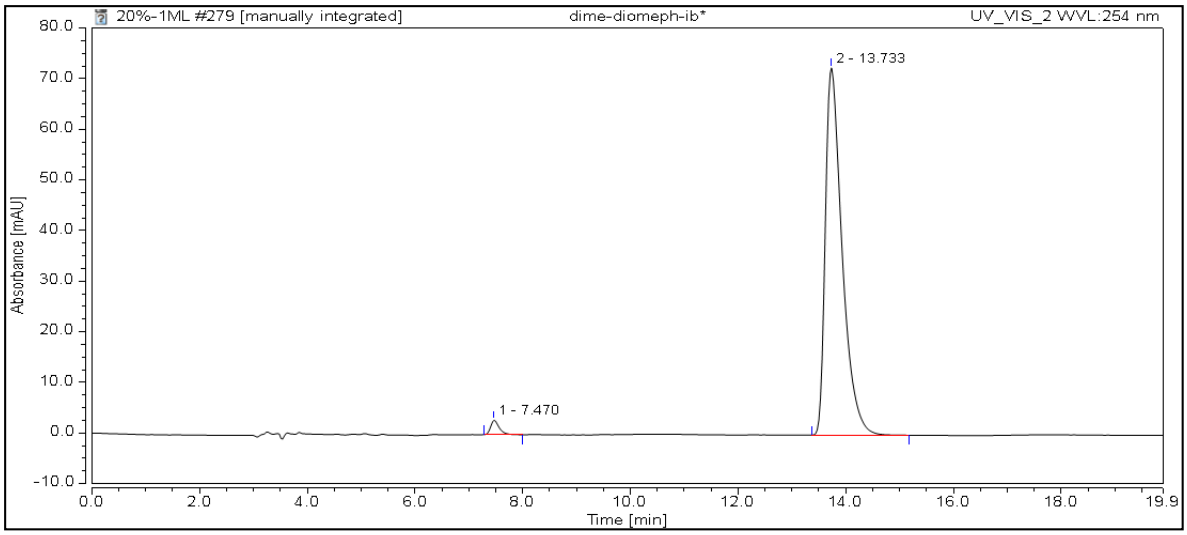
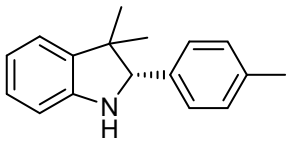


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		7.390	94.399	49.42	n.a.
2		13.510	96.622	50.58	n.a.
Total:			**191.021**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		7.470	0.530	1.98	n.a.
2		13.733	26.216	98.02	n.a.
Total:			**26.746**	**100.00**	



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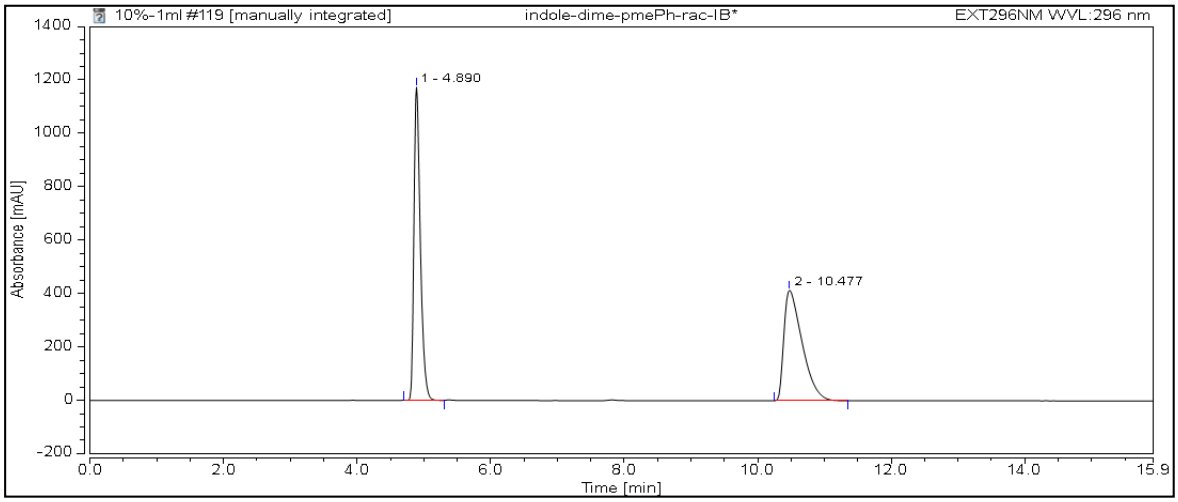
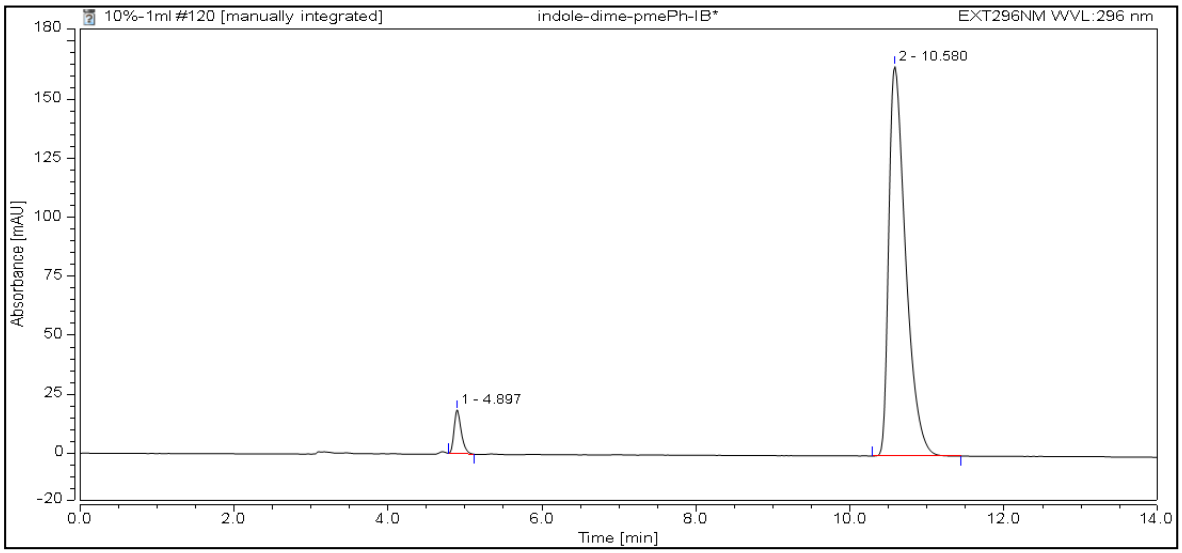
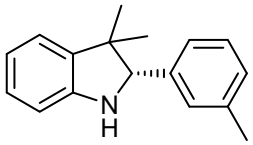


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		4.890	129.476	49.82	n.a.
2		10.477	130.438	50.18	n.a.
Total:			**259.914**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		4.897	1.989	4.52	n.a.
2		10.580	41.975	95.48	n.a.
Total:			**43.964**	**100.00**	



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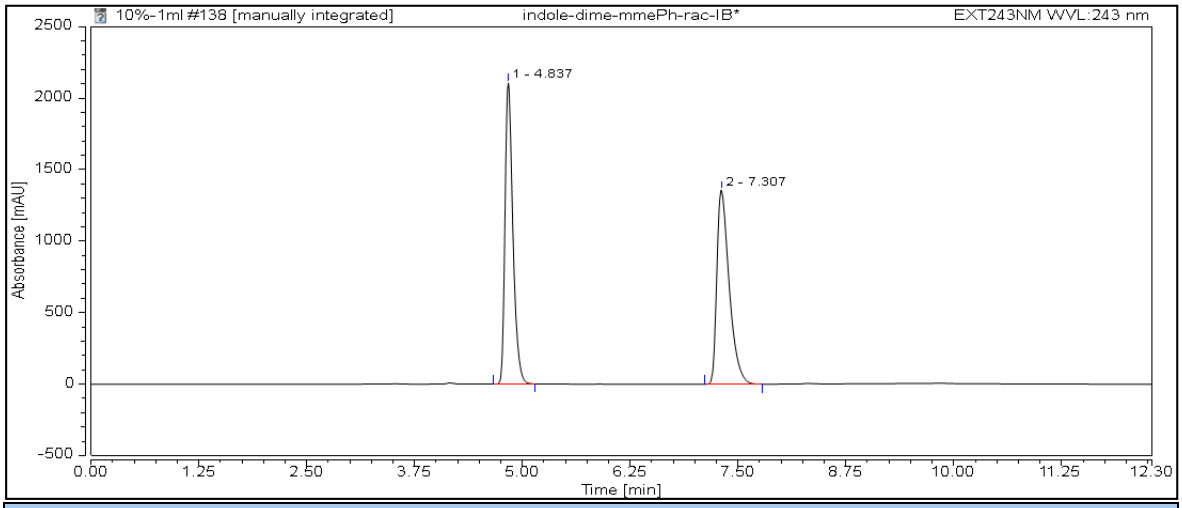
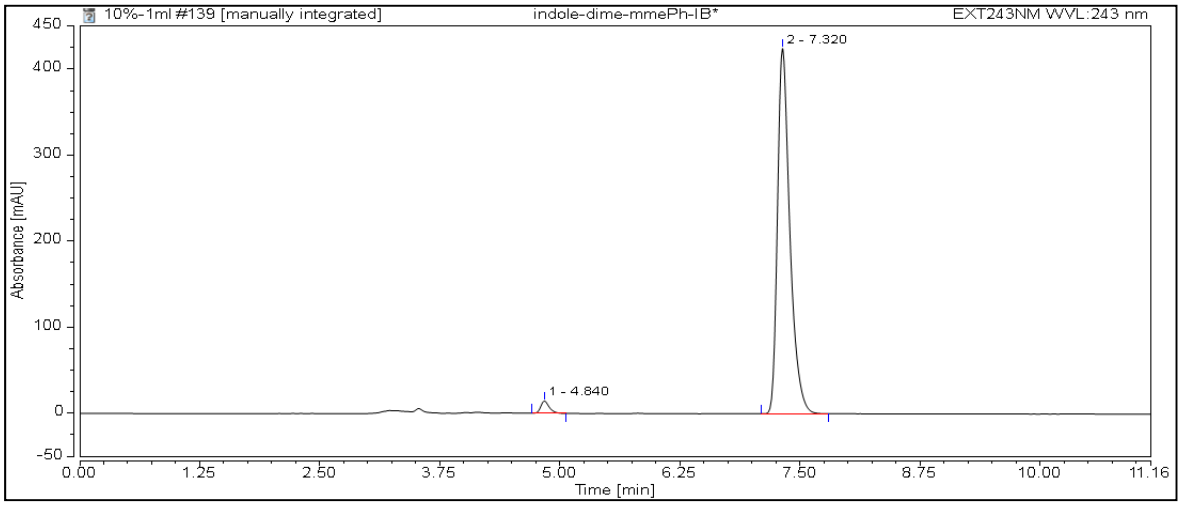
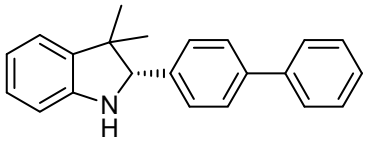


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		4.837	222.599	49.76	n.a.
2		7.307	224.783	50.24	n.a.
Total:			**447.382**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		4.840	1.455	2.24	n.a.
2		7.320	63.639	97.76	n.a.
Total:			**65.095**	**100.00**	



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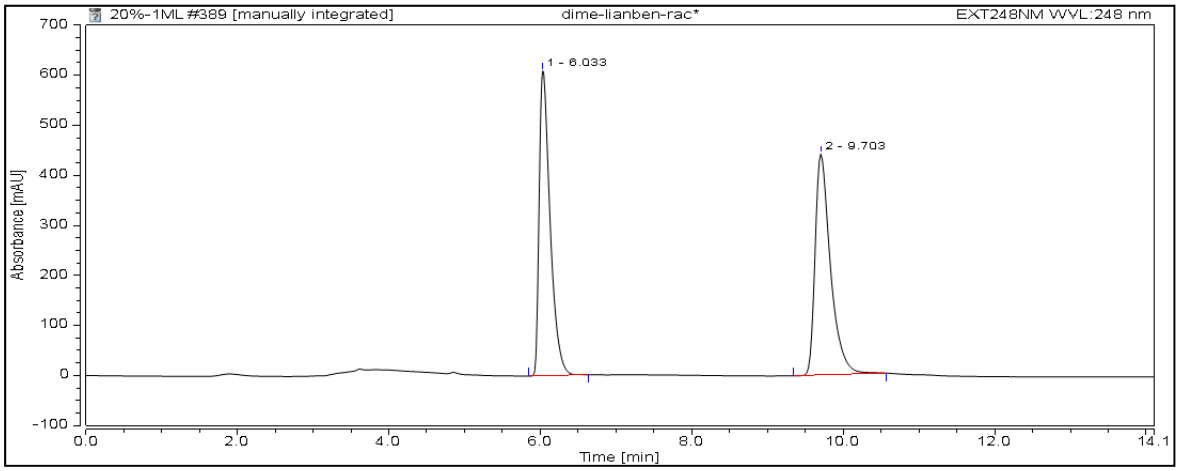
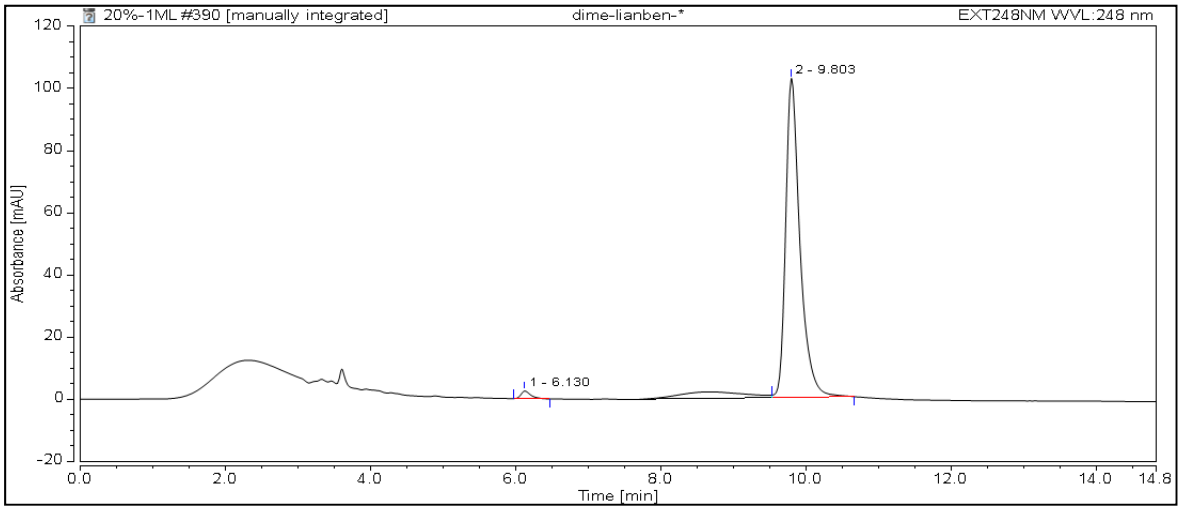
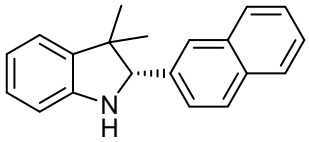


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		6.033	101.840	49.53	n.a.
2		9.703	103.791	50.47	n.a.
Total:			**205.631**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		6.130	0.386	1.62	n.a.
2		9.803	23.483	98.38	n.a.
Total:			**23.869**	**100.00**	



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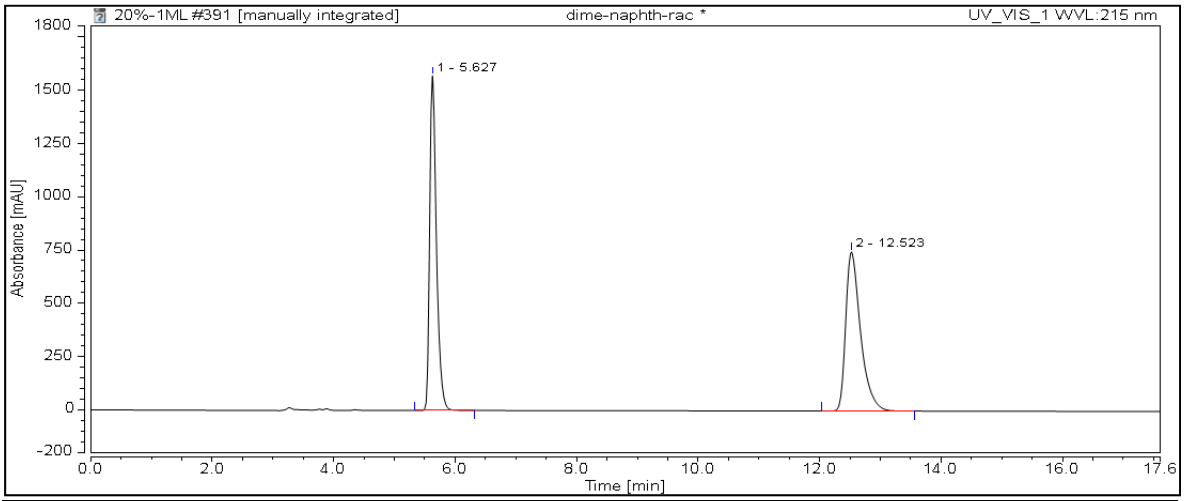
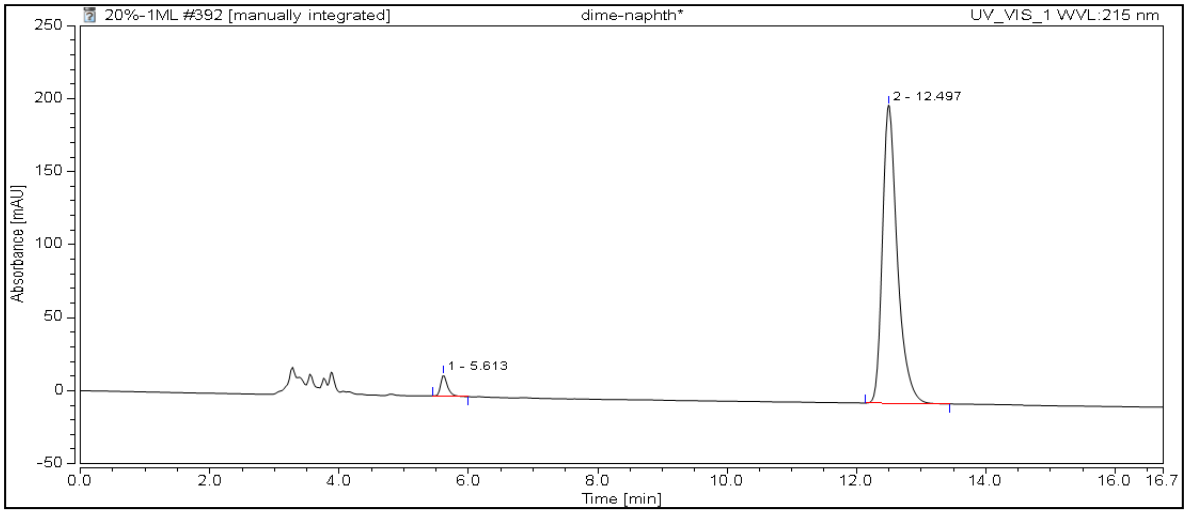
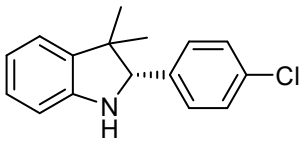


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.627	202.858	49.03	n.a.
2		12.523	210.875	50.97	n.a.
Total:			**413.732**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.613	1.756	3.11	n.a.
2		12.497	54.637	96.89	n.a.
Total:			**56.393**	**100.00**	



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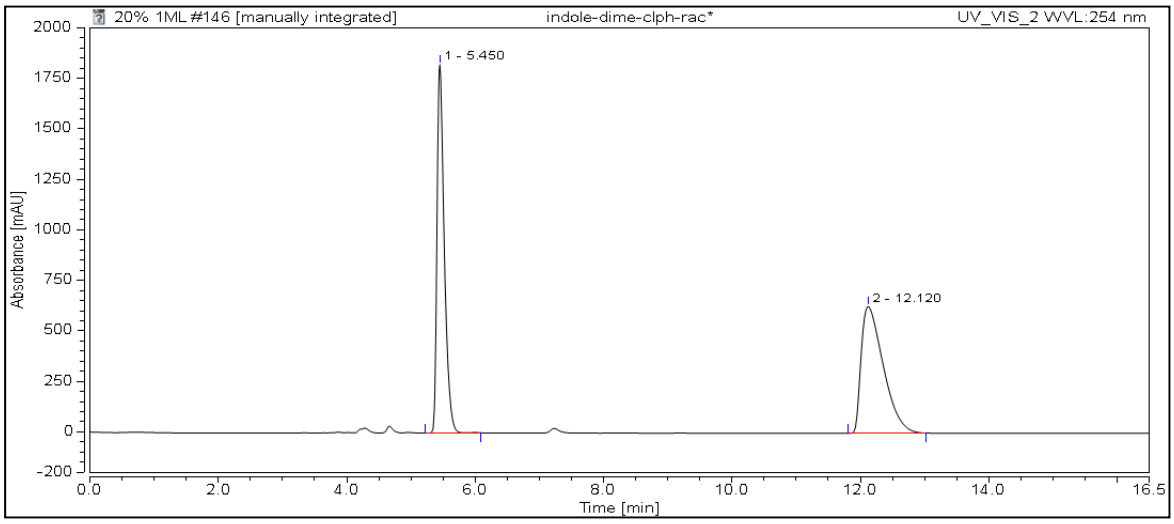
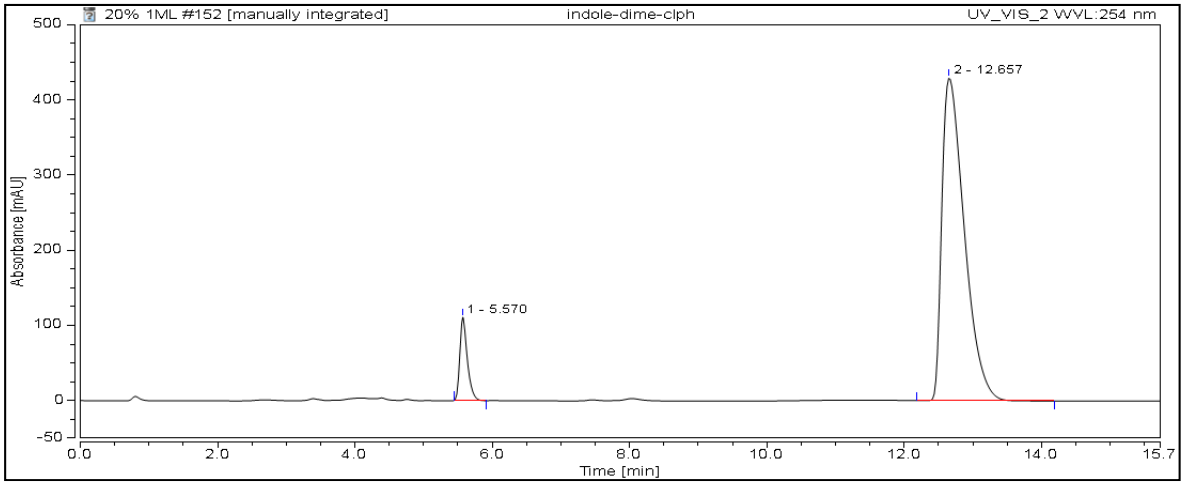
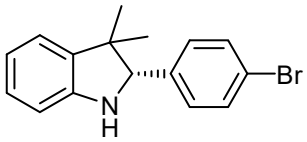


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.450	238.753	48.89	n.a.
2		12.120	249.638	51.11	n.a.
Total:			**488.391**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.570	13.925	7.89	n.a.
2		12.657	162.511	92.11	n.a.
Total:			**176.436**	**100.00**	



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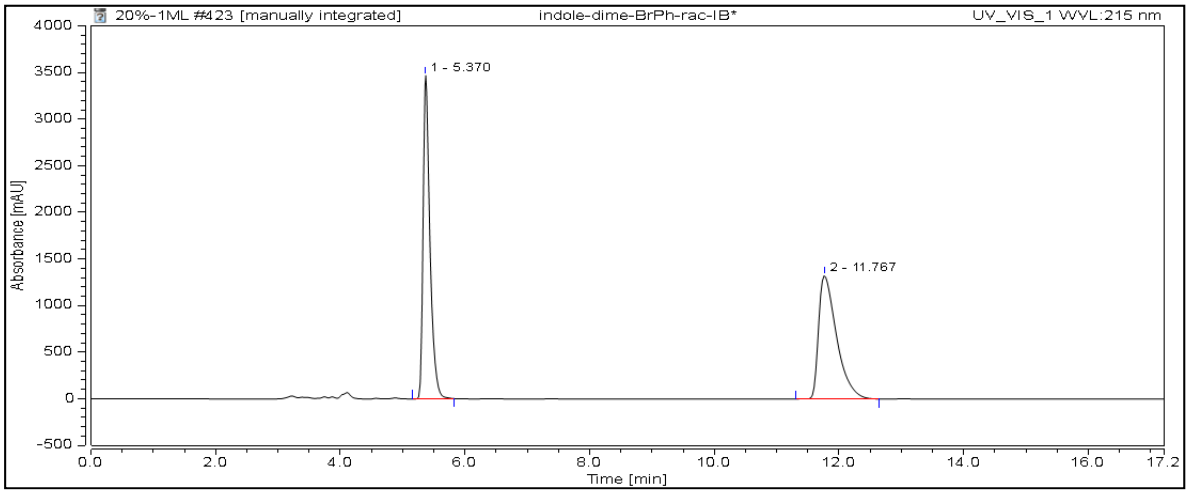
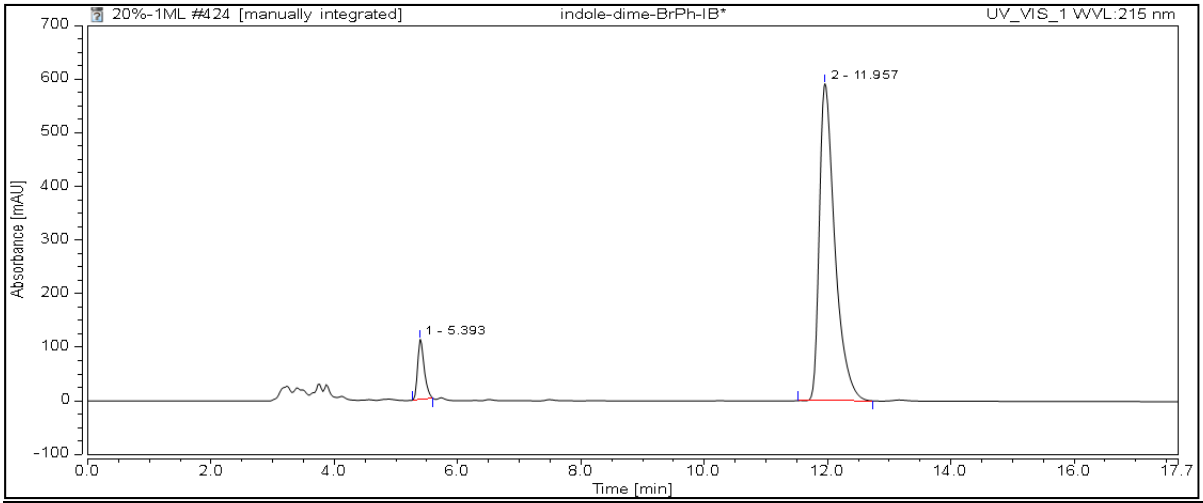
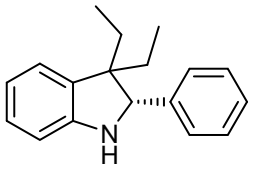


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.370	438.707	49.86	n.a.
2		11.767	441.161	50.14	n.a.
Total:			**879.868**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.393	13.856	7.45	n.a.
2		11.957	172.233	92.55	n.a.
Total:			**186.089**	**100.00**	



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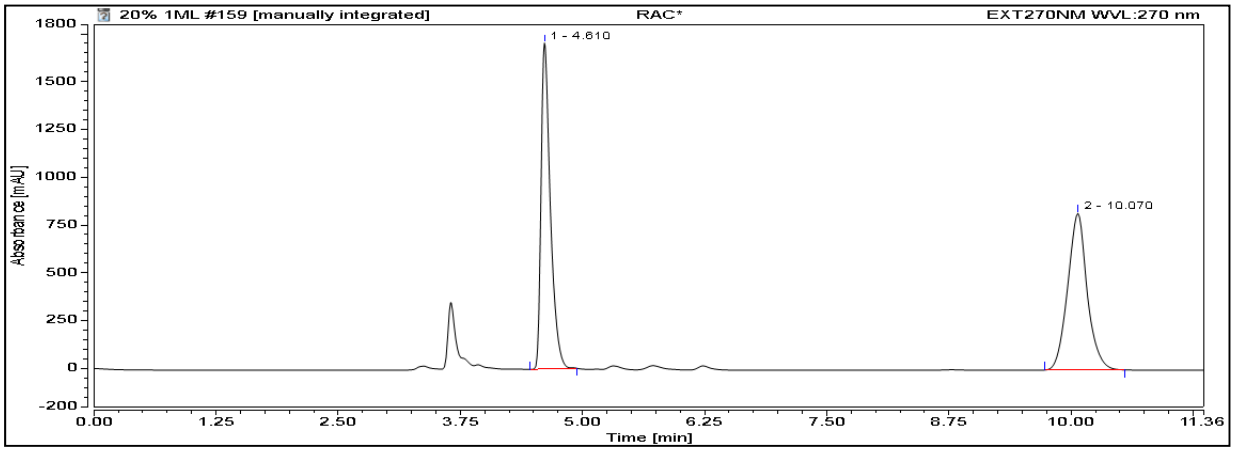
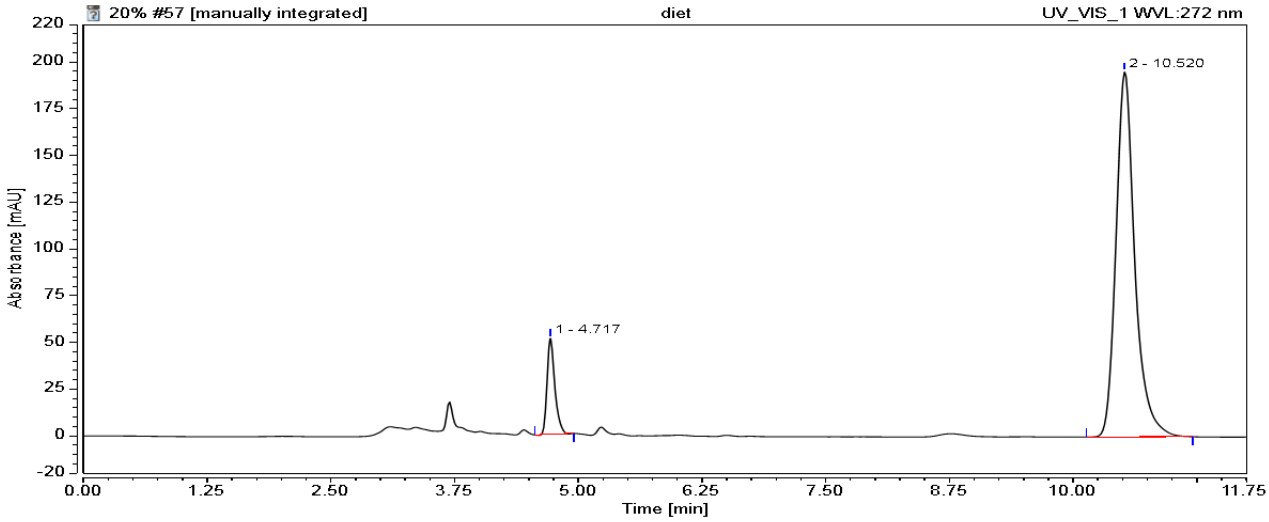
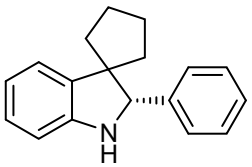


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		4.610	187.923	49.80	n.a.
2		10.070	189.424	50.20	n.a.
Total:			**377.347**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		4.717	4.620	9.69	n.a.
2		10.520	43.077	90.31	n.a.
Total:			**47.698**	**100.00**	



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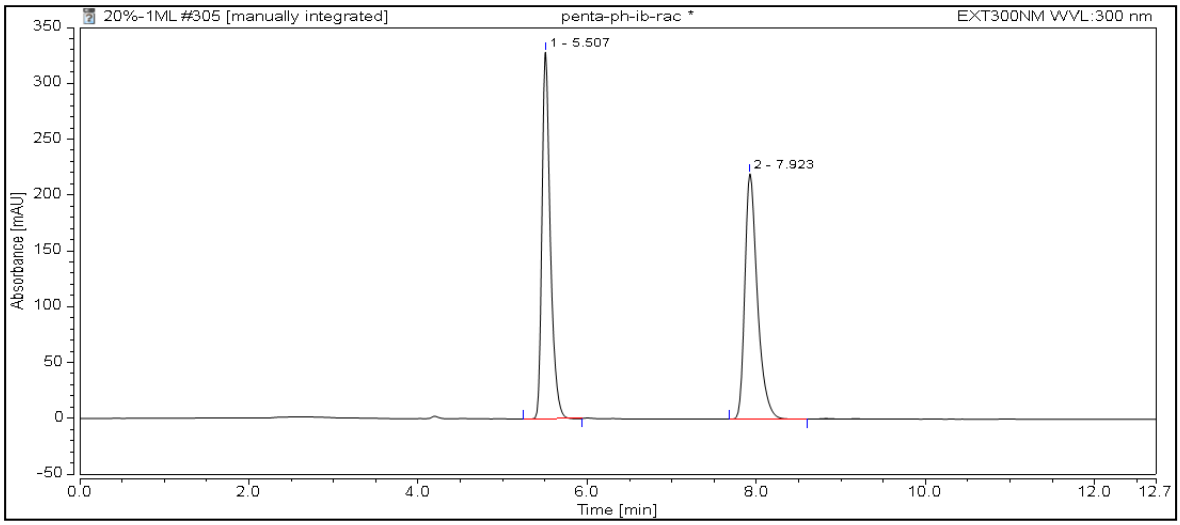
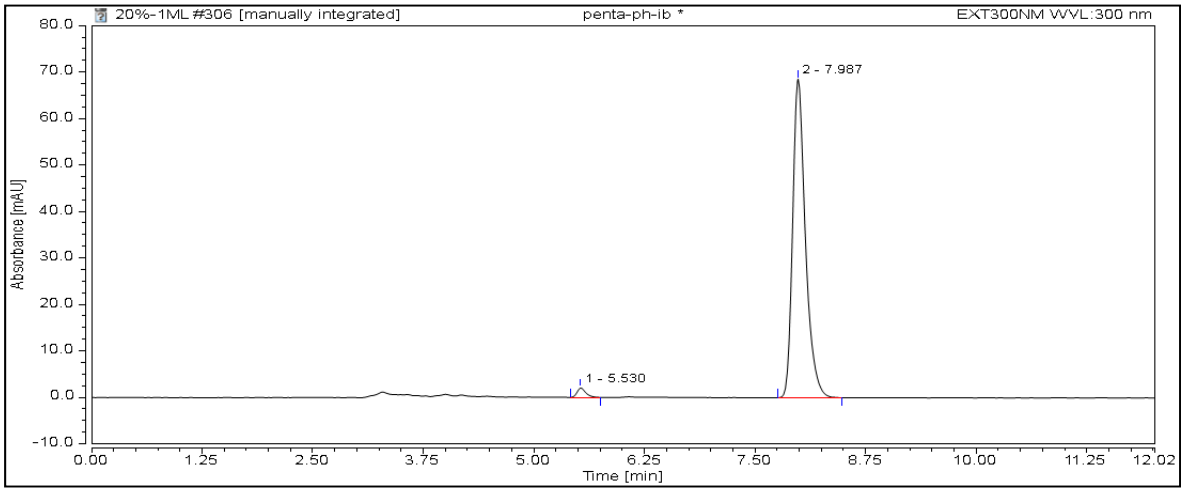
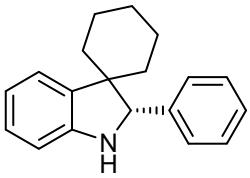


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.507	37.474	49.87	n.a.
2		7.923	37.673	50.13	n.a.
Total:			**75.147**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.530	0.232	1.98	n.a.
2		7.987	11.482	98.02	n.a.
Total:			**11.714**	**100.00**	



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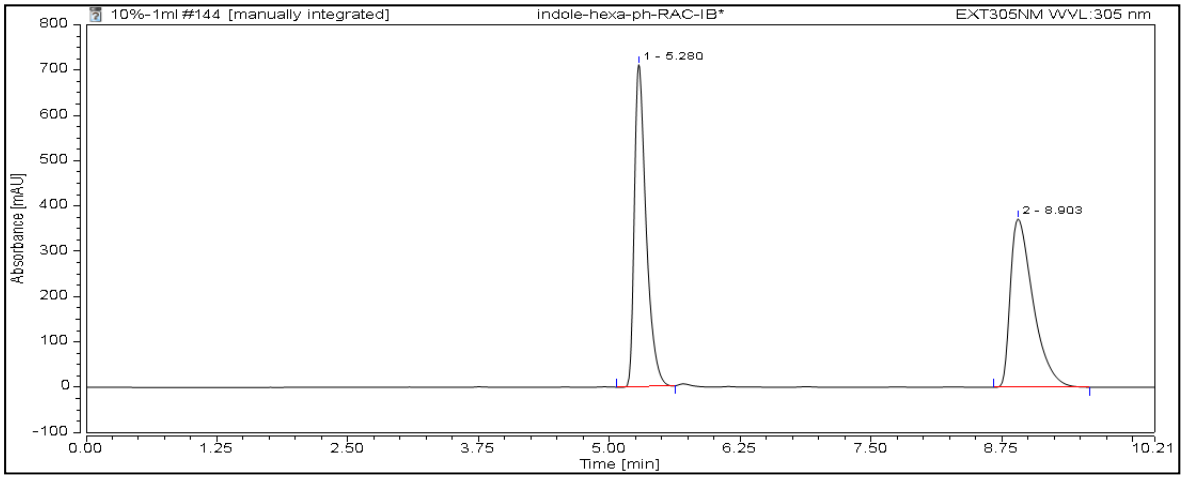
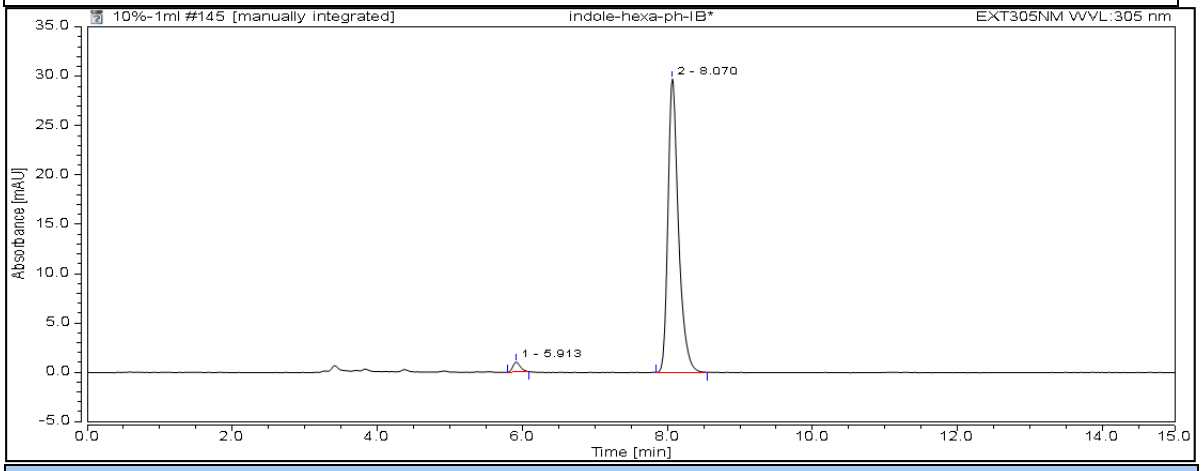
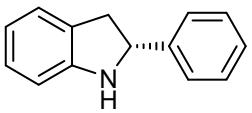


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.280	92.296	49.66	n.a.
2		8.903	93.543	50.34	n.a.
Total:			**185.839**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.913	0.122	2.37	n.a.
2		8.070	5.018	97.63	n.a.
Total:			**5.140**	**100.00**	



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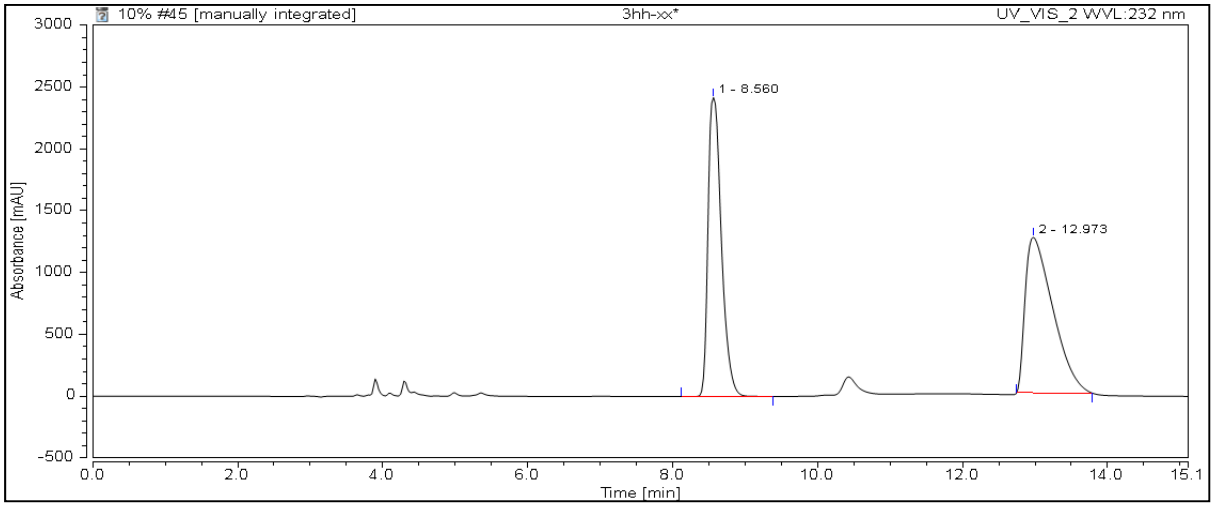
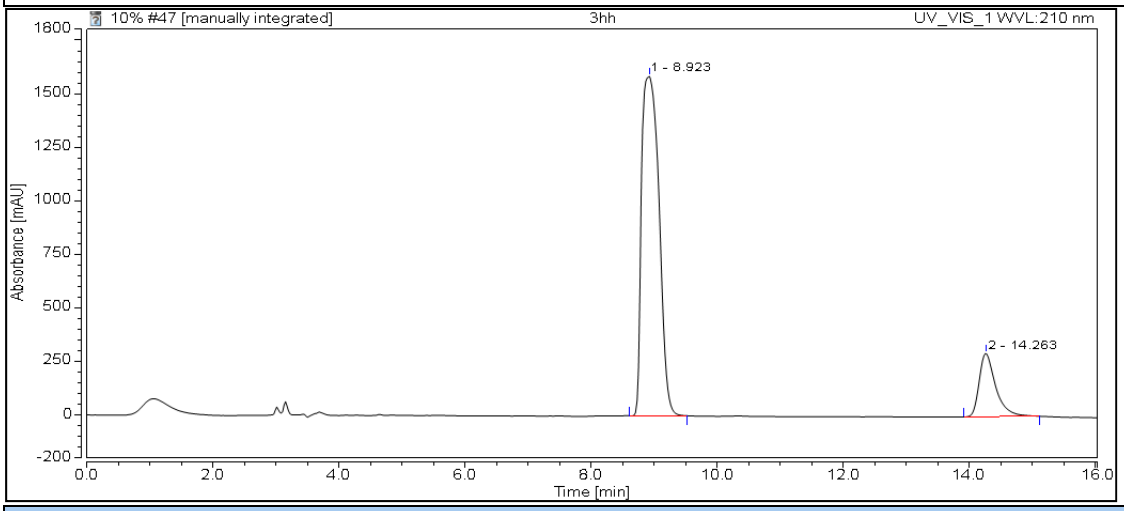
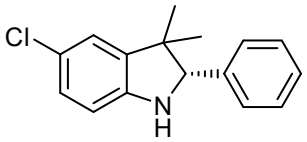


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		8.560	515.362	48.08	n.a.
2		12.973	556.413	51.92	n.a.
Total:			**1071.774**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		8.923	508.146	84.96	n.a.
2		14.263	89.926	15.04	n.a.
Total:			**598.071**	**100.00**	



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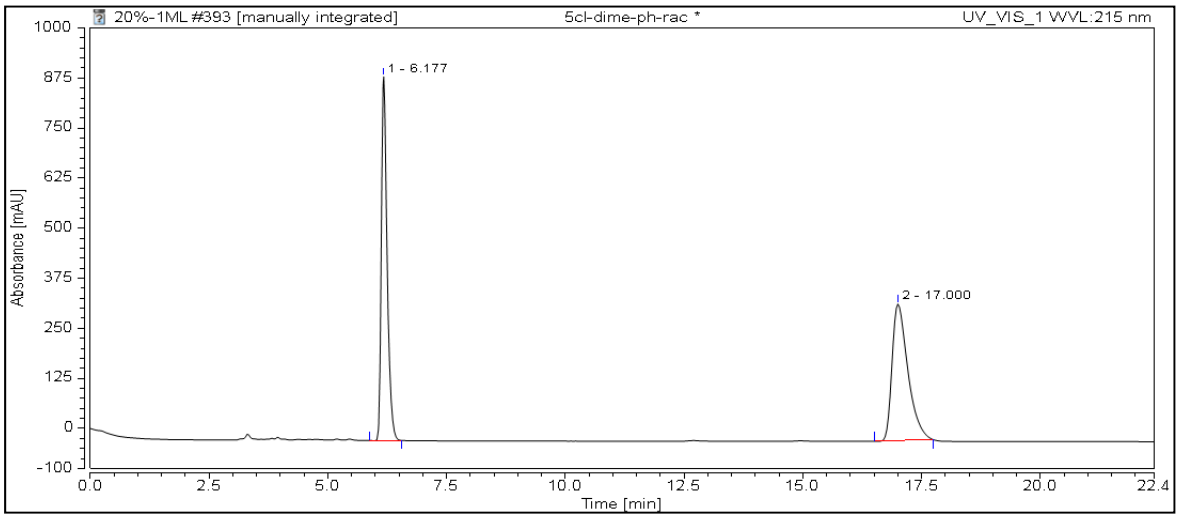
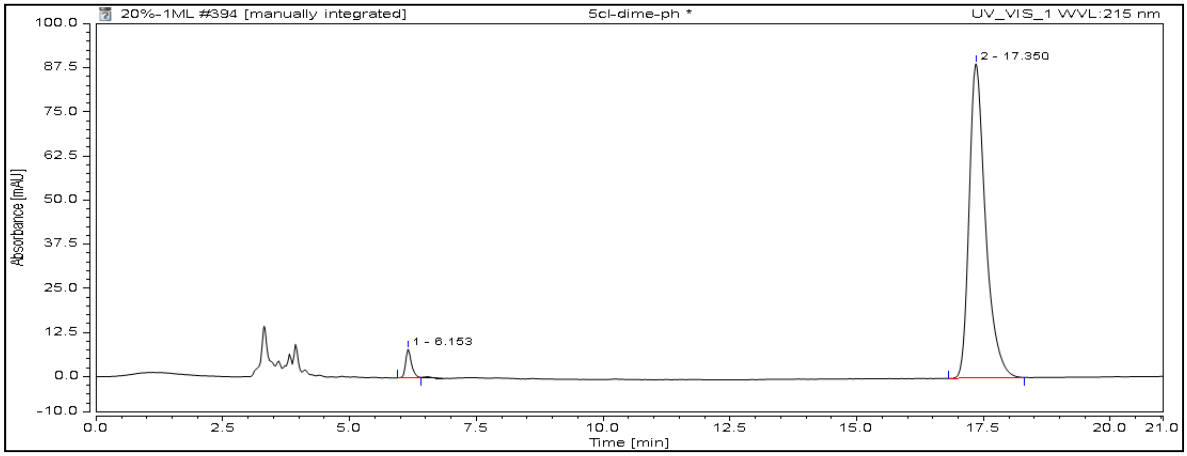
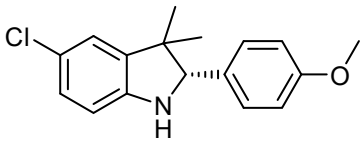


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		6.177	127.293	49.15	n.a.
2		17.000	131.679	50.85	n.a.
Total:			**258.972**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		6.153	1.117	3.24	n.a.
2		17.350	33.379	96.76	n.a.
Total:			**34.496**	**100.00**	



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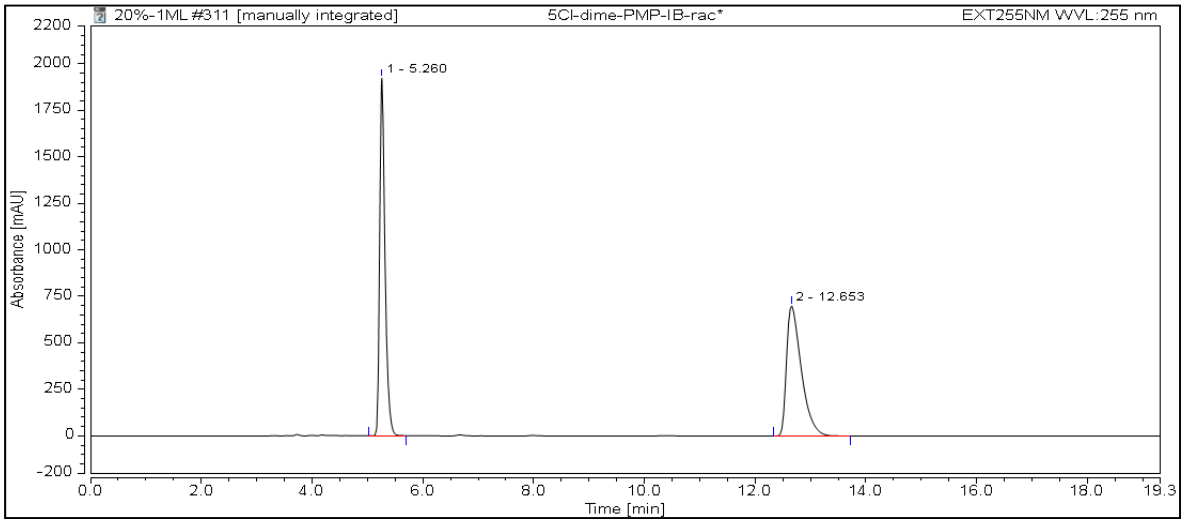
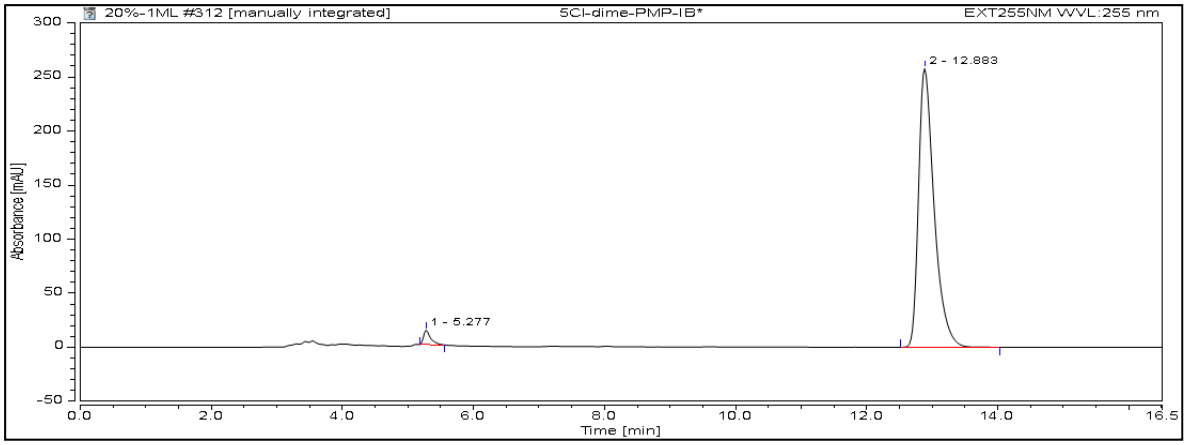
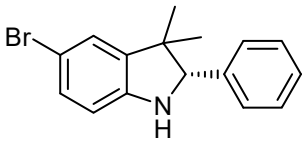


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.260	215.373	49.97	n.a.
2		12.653	215.625	50.03	n.a.
Total:			**430.998**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.277	1.687	2.32	n.a.
2		12.883	71.146	97.68	n.a.
Total:			**72.833**	**100.00**	



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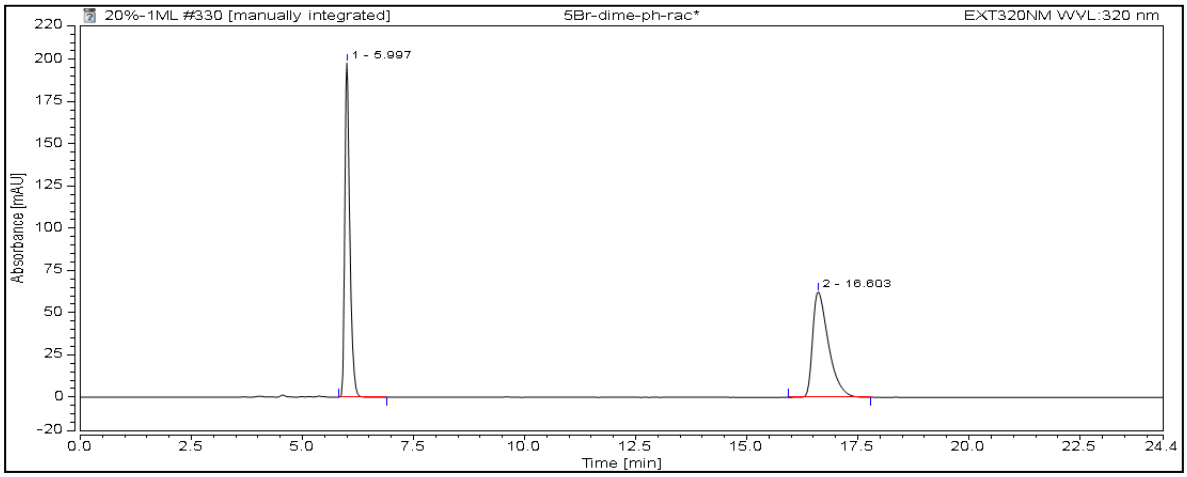
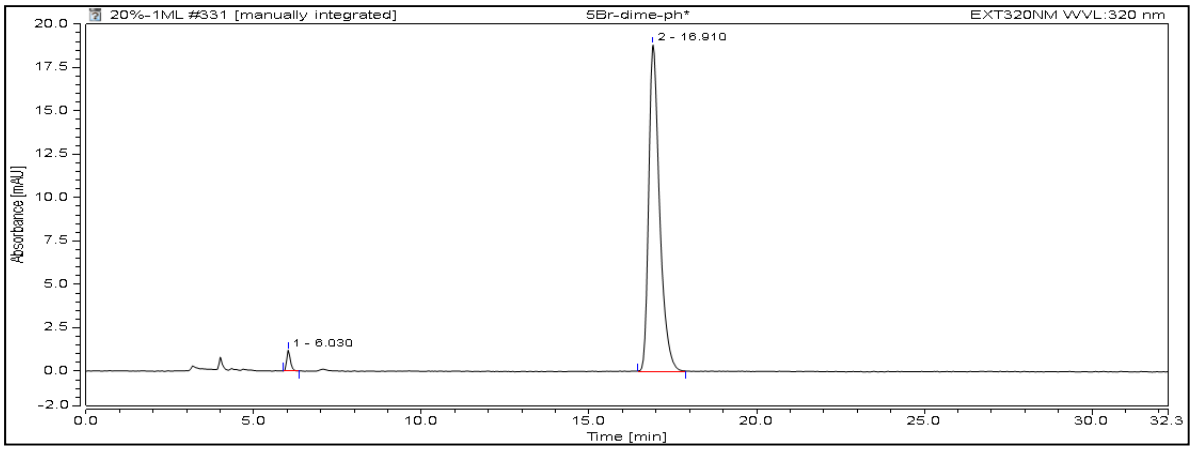
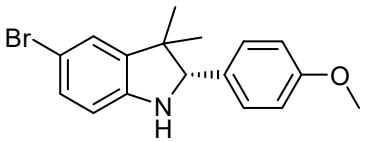


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.997	25.644	49.91	n.a.
2		16.603	25.734	50.09	n.a.
Total:			**51.378**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		6.030	0.155	2.14	n.a.
2		16.910	7.072	97.86	n.a.
Total:			**7.226**	**100.00**	



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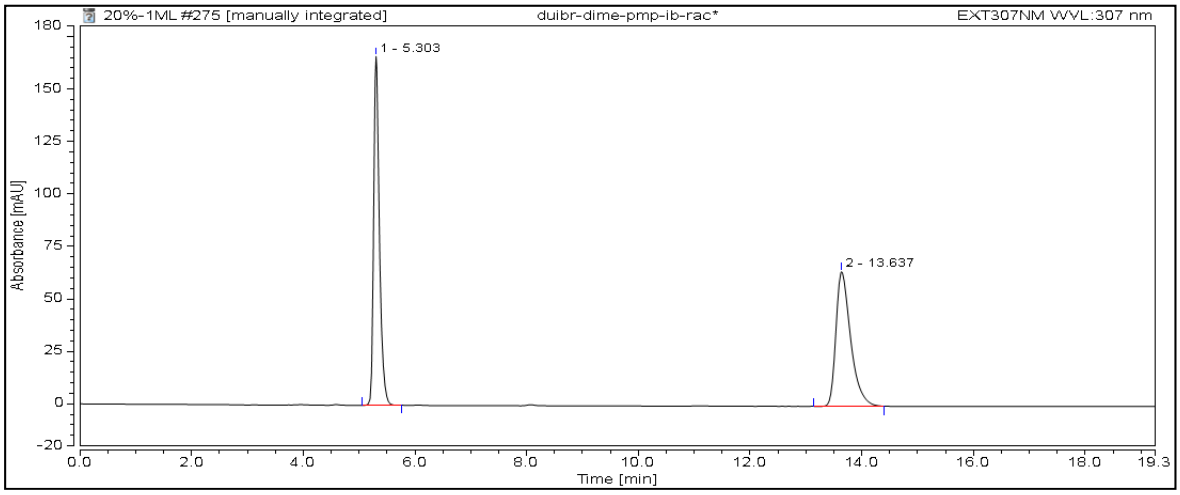
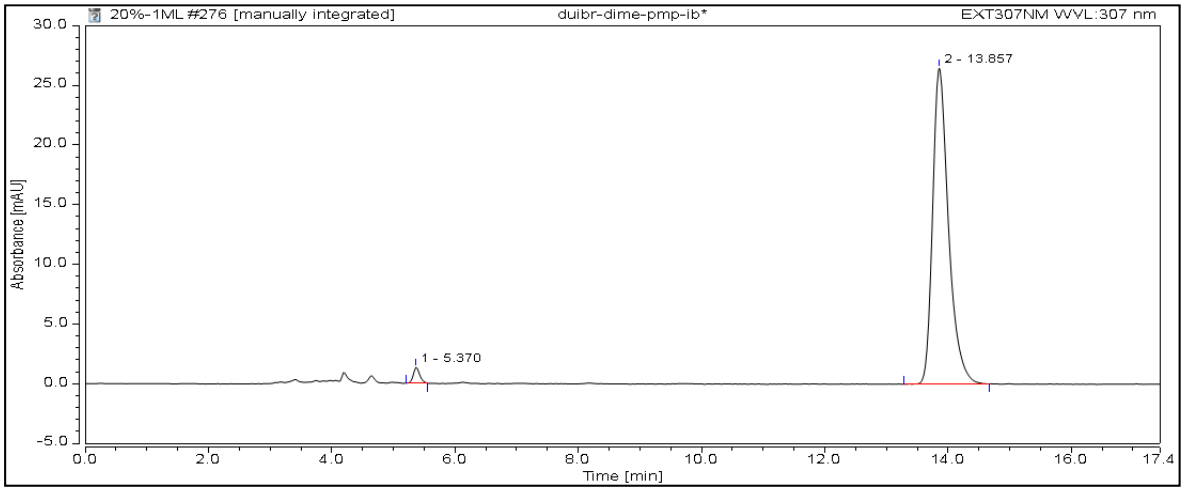
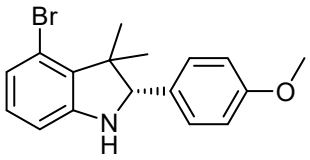


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.303	19.804	50.36	n.a.
2		13.637	19.522	49.64	n.a.
Total:			**39.326**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.370	0.161	2.00	n.a.
2		13.857	7.899	98.00	n.a.
Total:			**8.060**	**100.00**	



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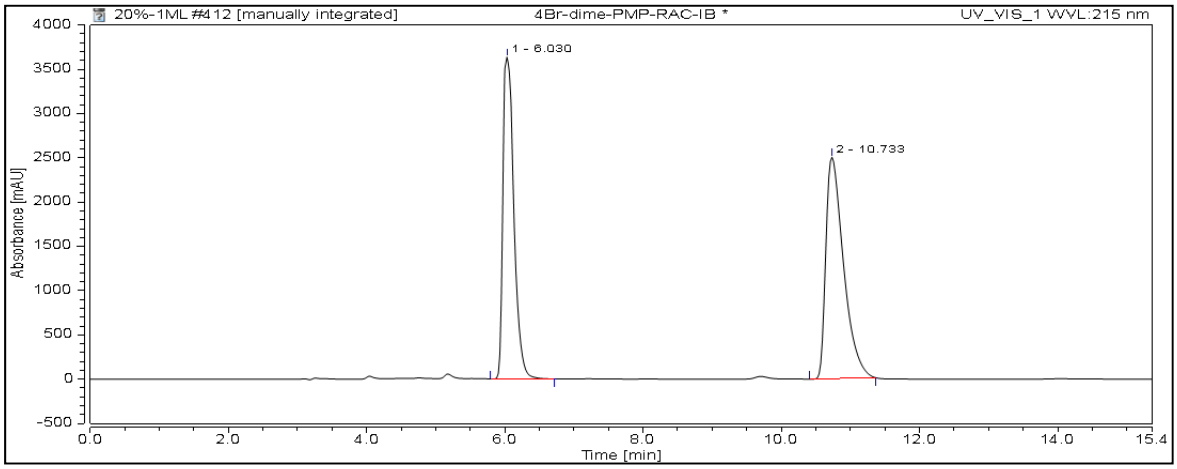
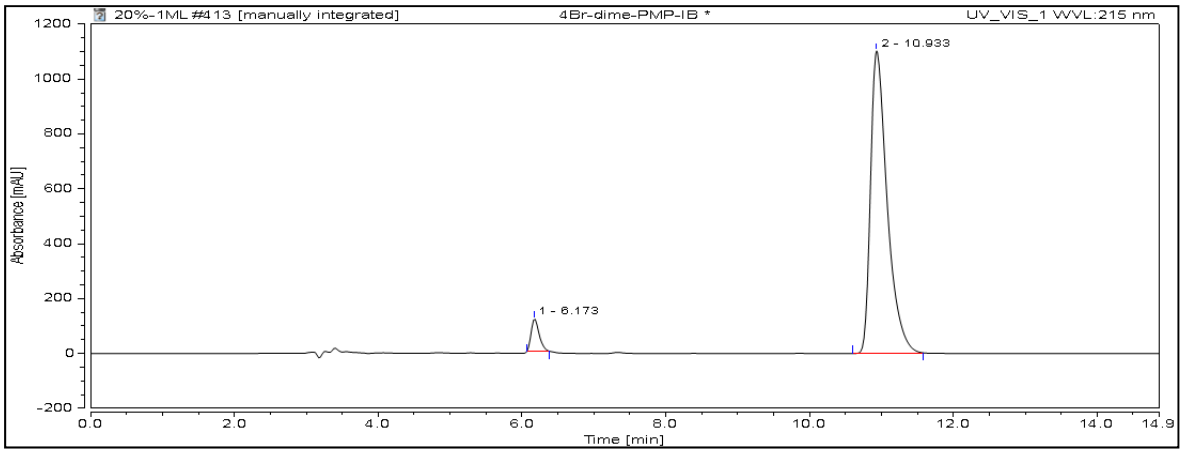
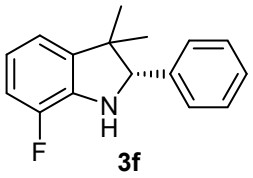


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		6.030	667.885	47.37	n.a.
2		10.733	742.144	52.63	n.a.
Total:			**1410.029**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		6.173	15.485	4.98	n.a.
2		10.933	295.613	95.02	n.a.
Total:			**311.098**	**100.00**	



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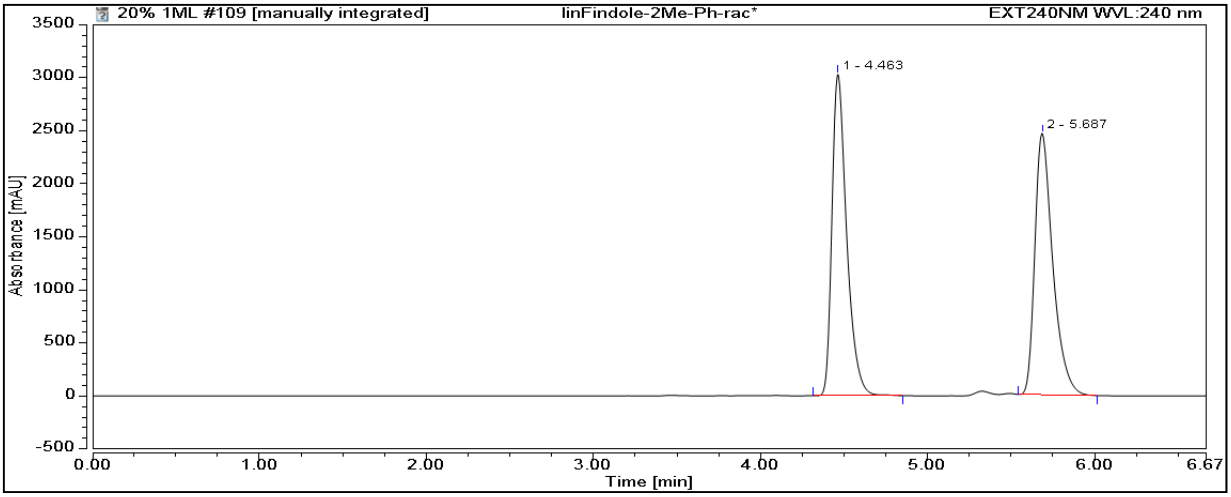
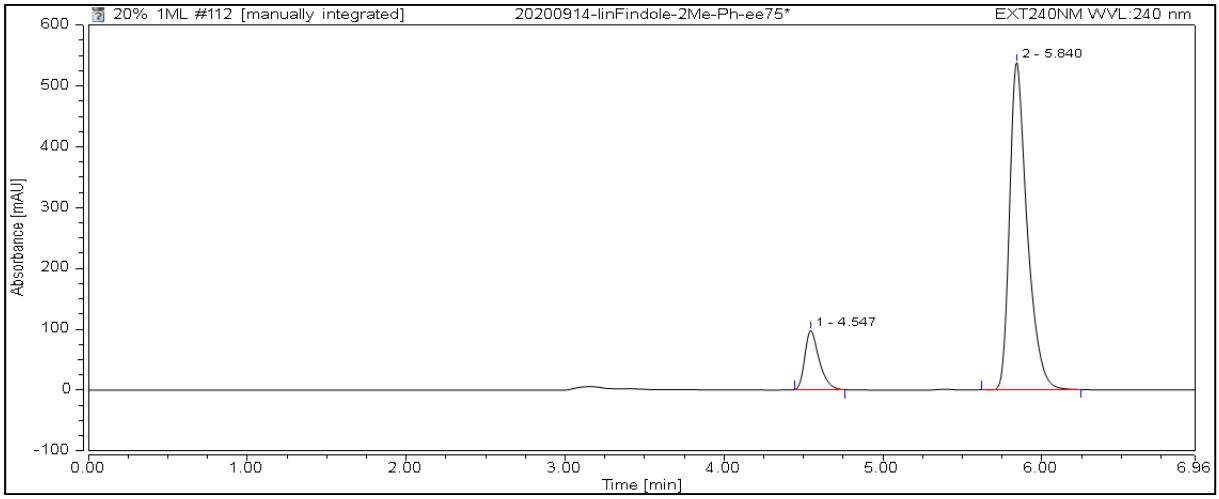
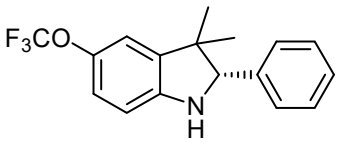


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		4.463	308.375	50.09	n.a.
2		5.687	307.212	49.91	n.a.
Total:			**615.587**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		4.547	10.152	12.82	n.a.
2		5.840	69.038	87.18	n.a.
Total:			**79.190**	**100.00**	



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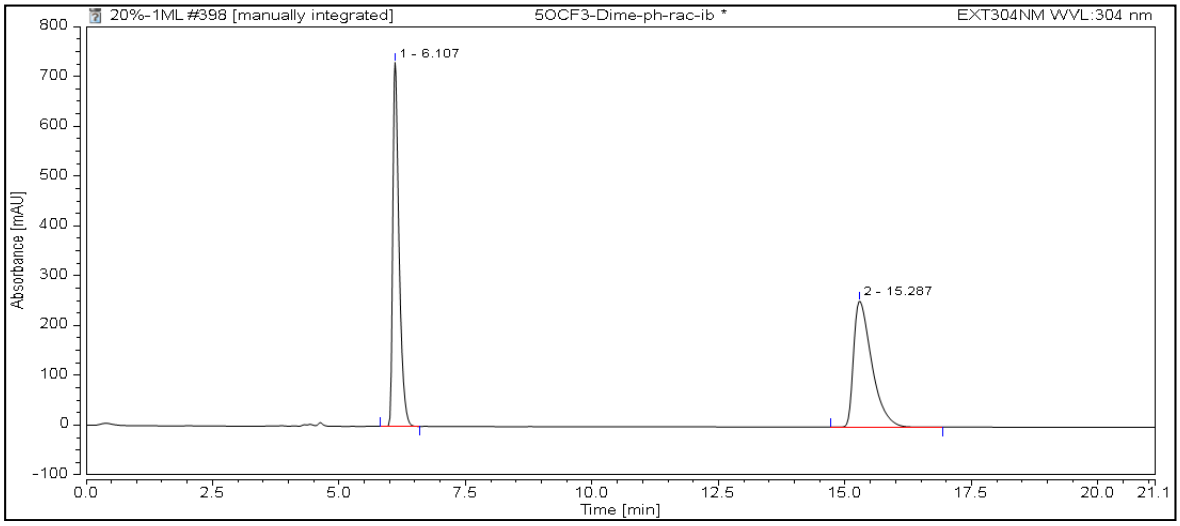
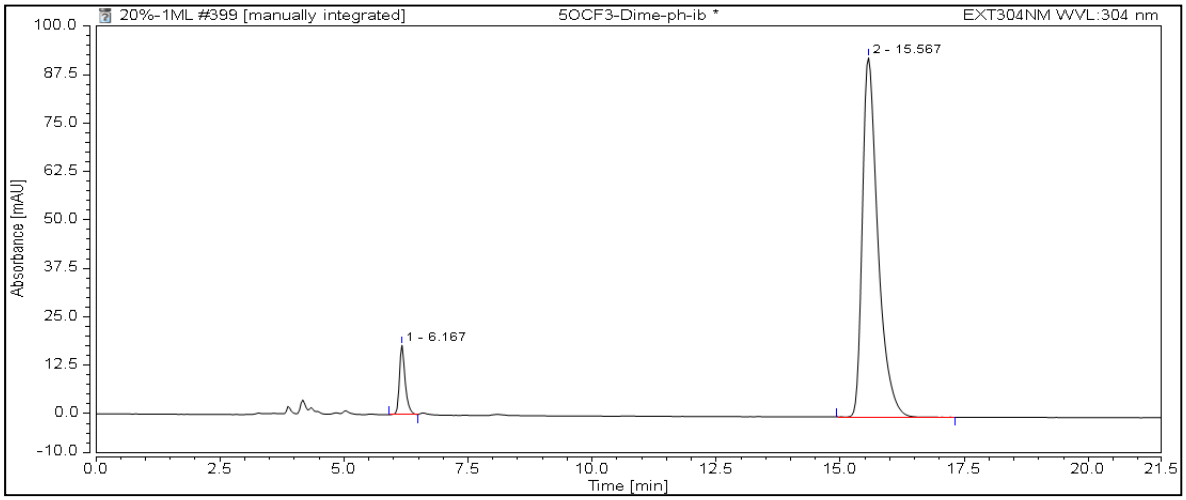
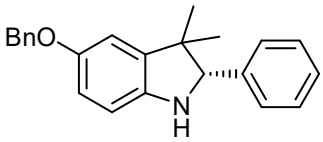


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		6.107	104.778	49.94	n.a.
2		15.287	105.010	50.06	n.a.
Total:			**209.787**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		6.167	2.404	6.66	n.a.
2		15.567	33.714	93.34	n.a.
Total:			**36.117**	**100.00**	



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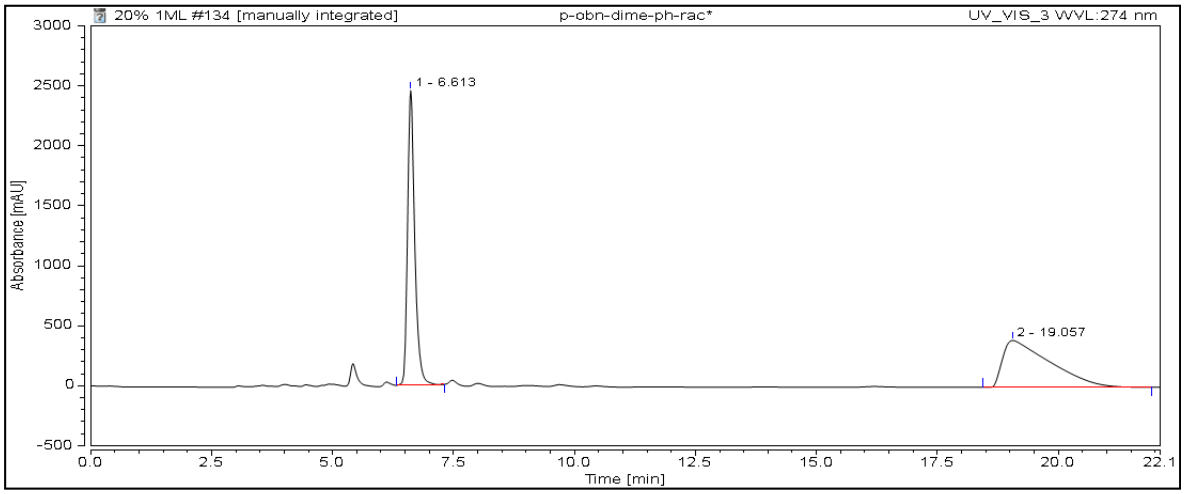
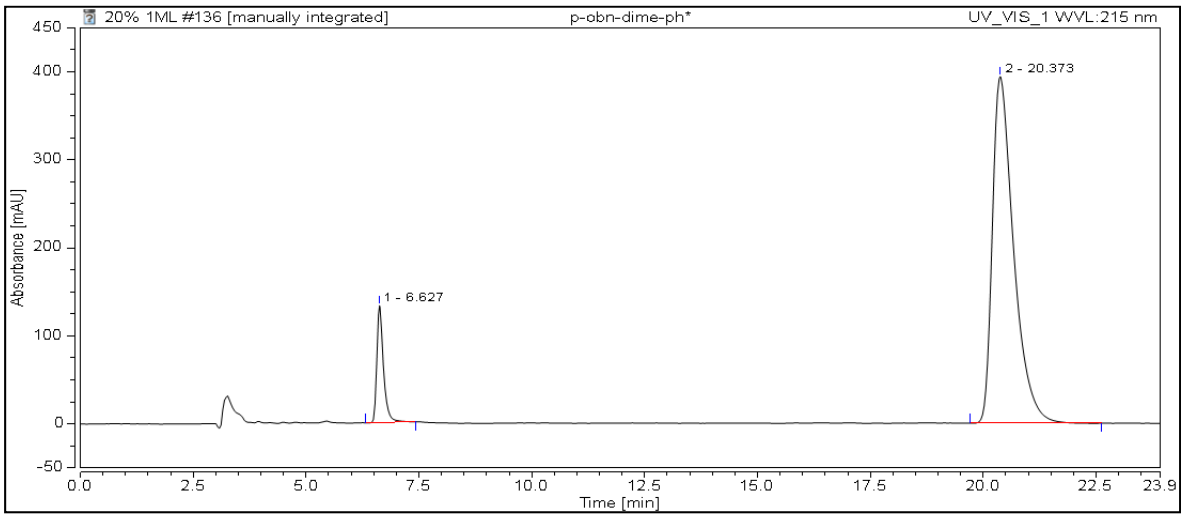
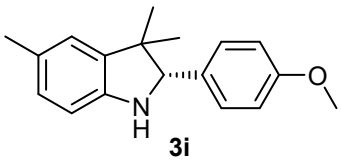


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		6.613	420.573	48.88	n.a.
2		19.057	439.878	51.12	n.a.
Total:			**860.450**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		6.627	22.490	9.46	n.a.
2		20.373	215.156	90.54	n.a.
Total:			**237.646**	**100.00**	



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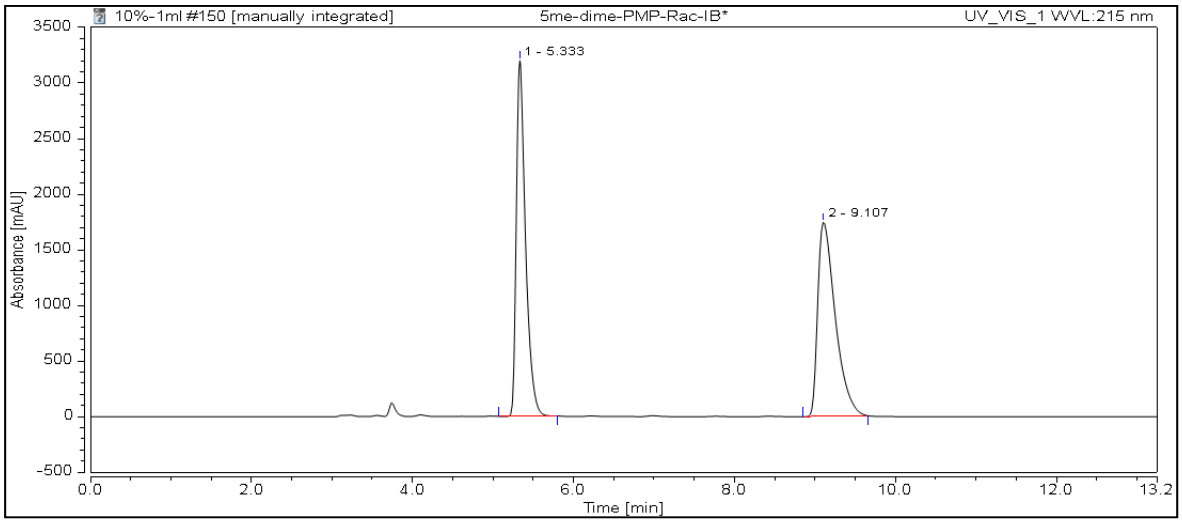
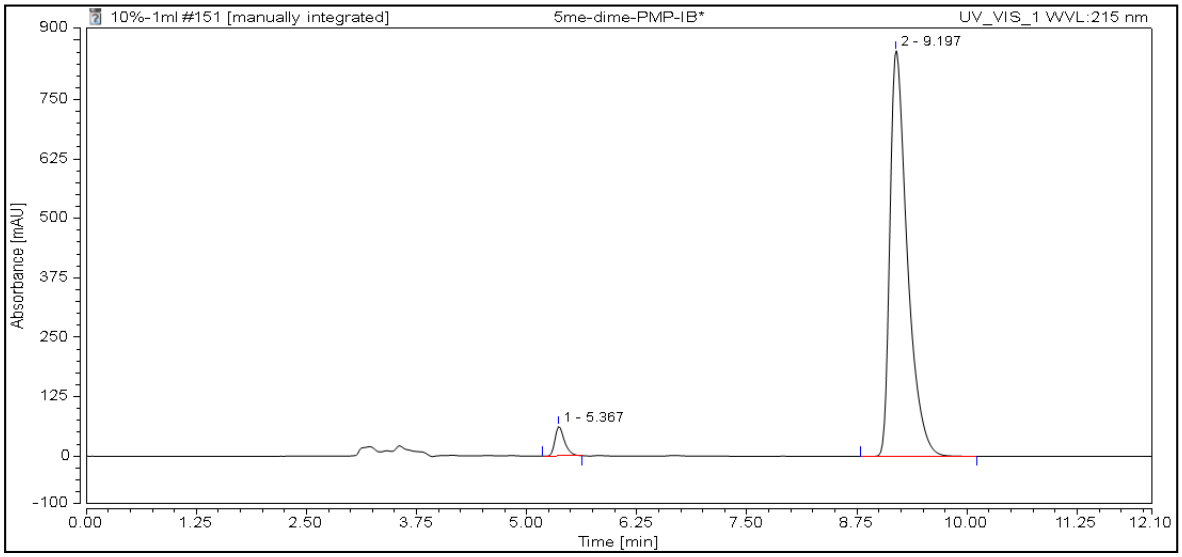
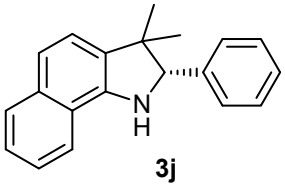


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.333	422.748	49.02	n.a.
2		9.107	439.615	50.98	n.a.
Total:			**862.362**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.367	7.762	3.83	n.a.
2		9.197	195.119	96.17	n.a.
Total:			**202.881**	**100.00**	



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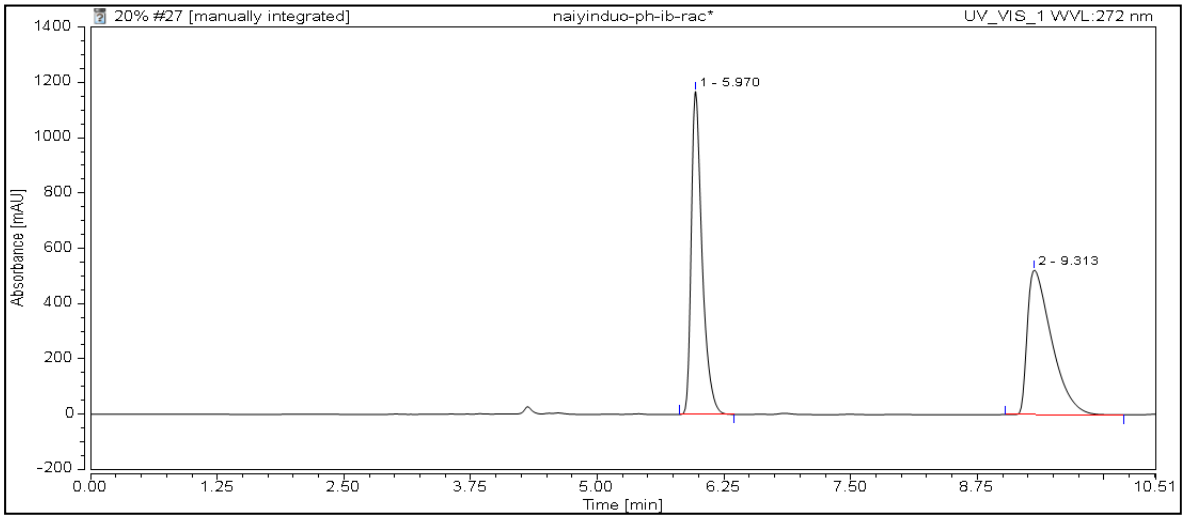
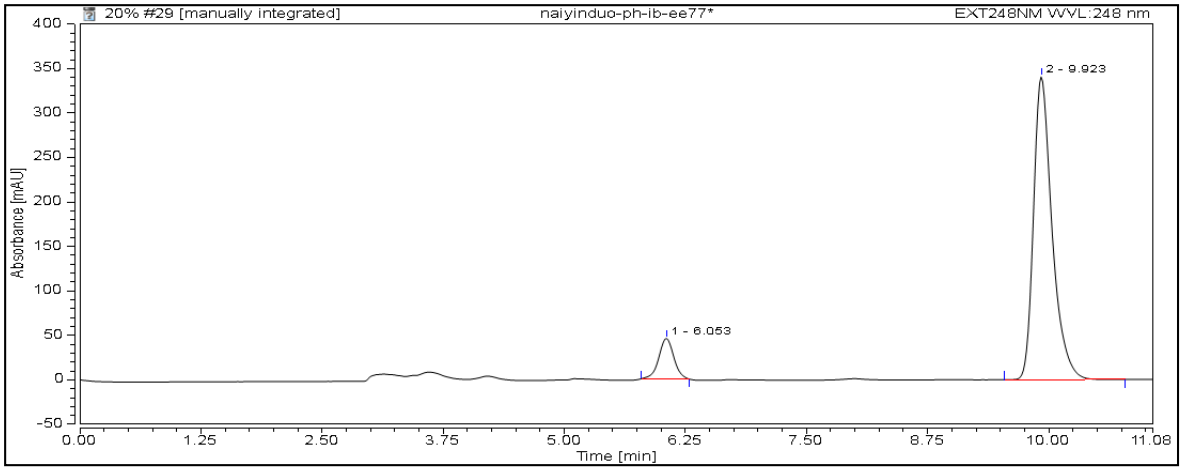
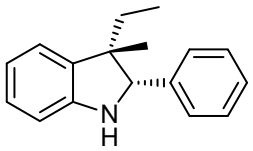


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.970	144.679	49.95	n.a.
2		9.313	144.977	50.05	n.a.
Total:			**289.656**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		6.053	8.167	9.75	n.a.
2		9.923	75.621	90.25	n.a.
Total:			**83.787**	**100.00**	



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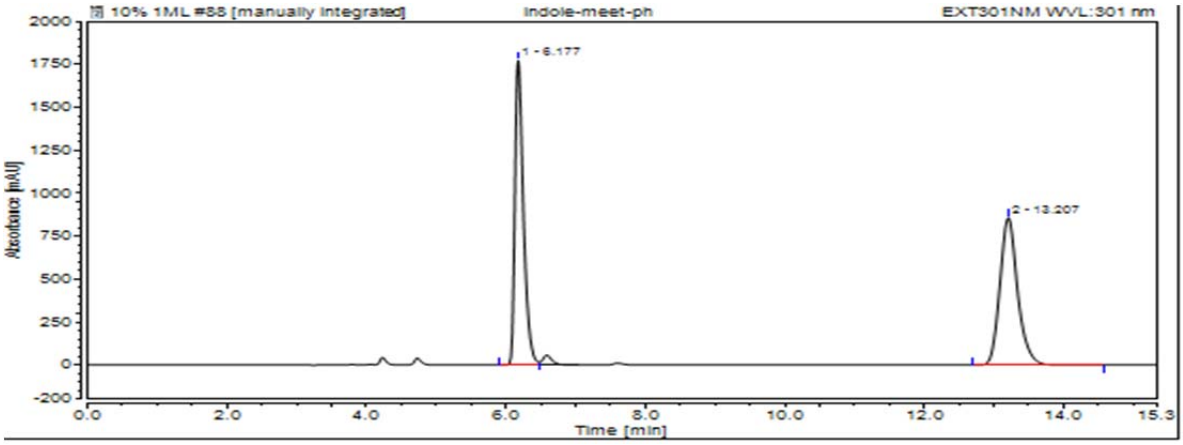
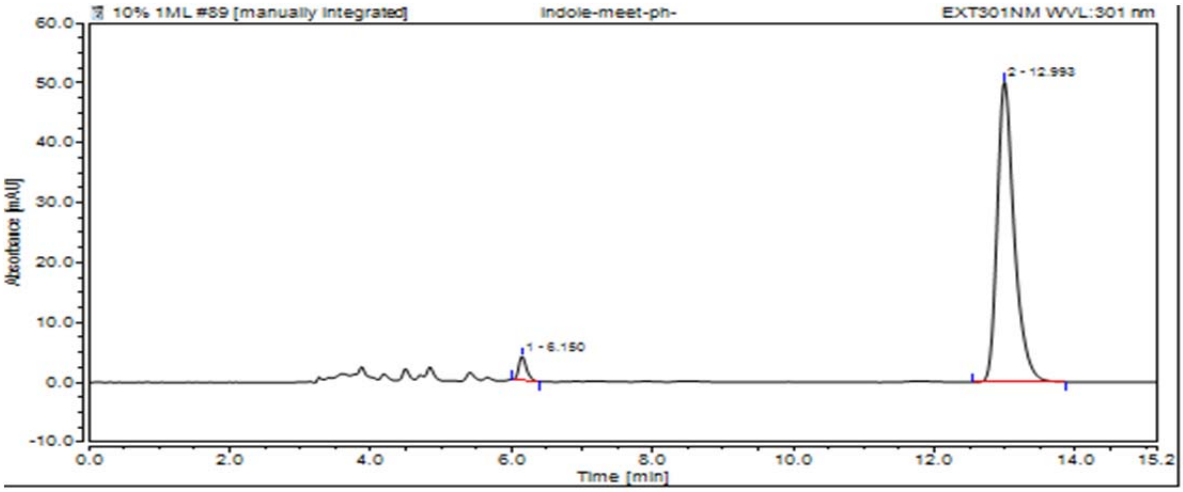
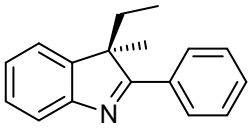


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		6.177	249.239	49.77	n.a.
2		13.207	251.567	50.23	n.a.
Total:			**500.806**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		6.150	0.507	3.48	n.a.
2		12.993	14.065	96.52	n.a.
Total:			**14.572**	**100.00**	



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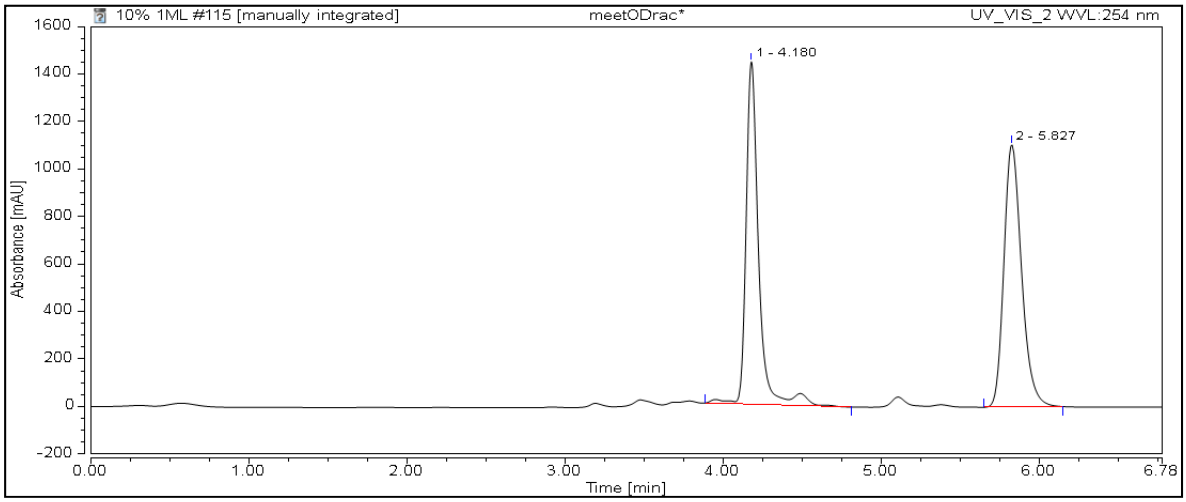
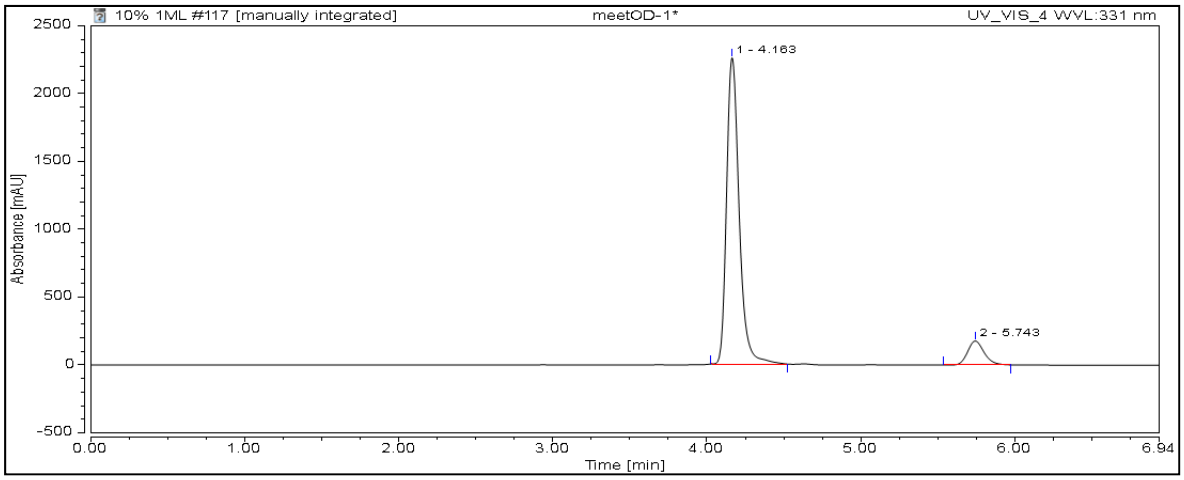
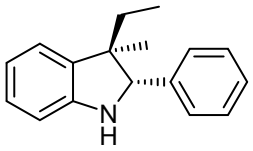


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		4.180	133.769	48.33	n.a.
2		5.827	143.001	51.67	n.a.
Total:			**276.770**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		4.163	213.500	90.66	n.a.
2		5.743	22.007	9.34	n.a.
Total:			**235.507**	**100.00**	



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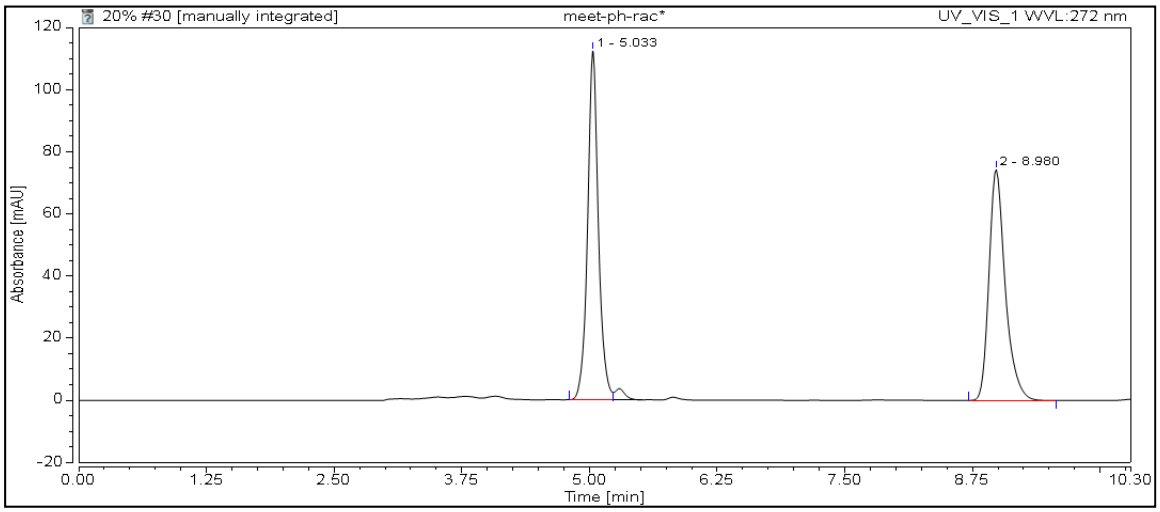
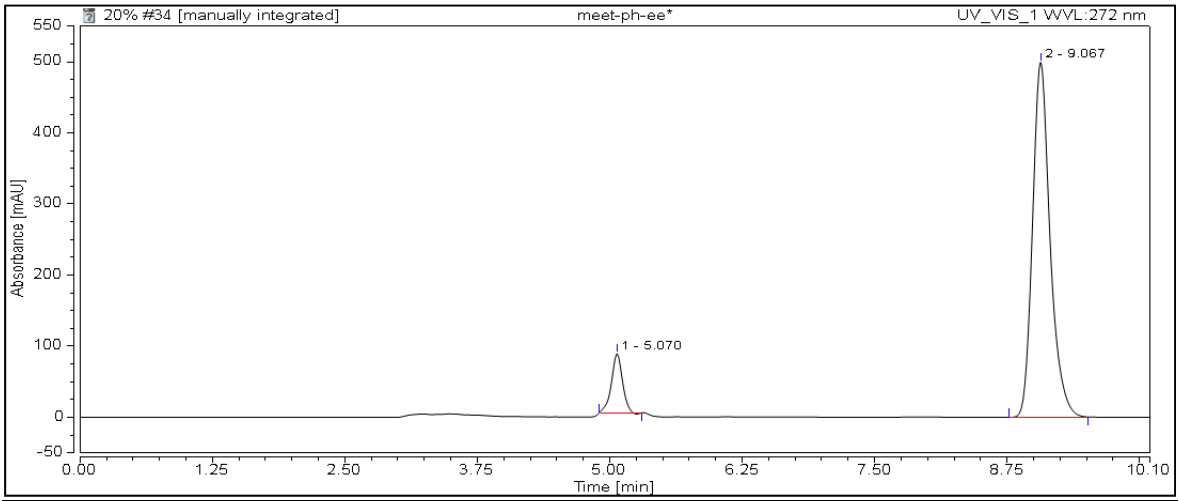
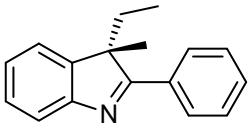


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.033	13.741	50.03	n.a.
2		8.980	13.727	49.97	n.a.
Total:			**27.469**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.070	9.930	9.39	n.a.
2		9.067	95.818	90.61	n.a.
Total:			**105.748**	**100.00**	



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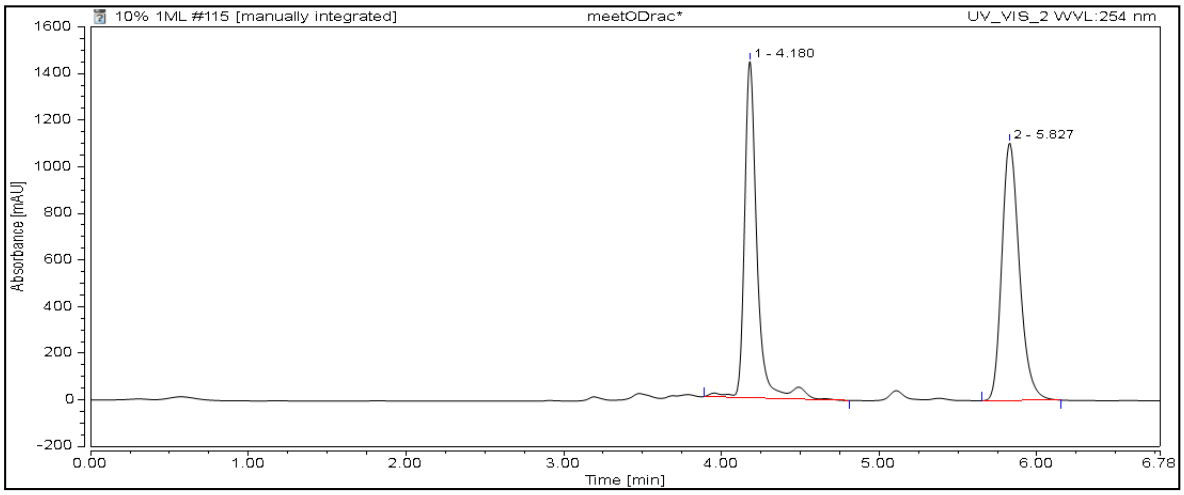
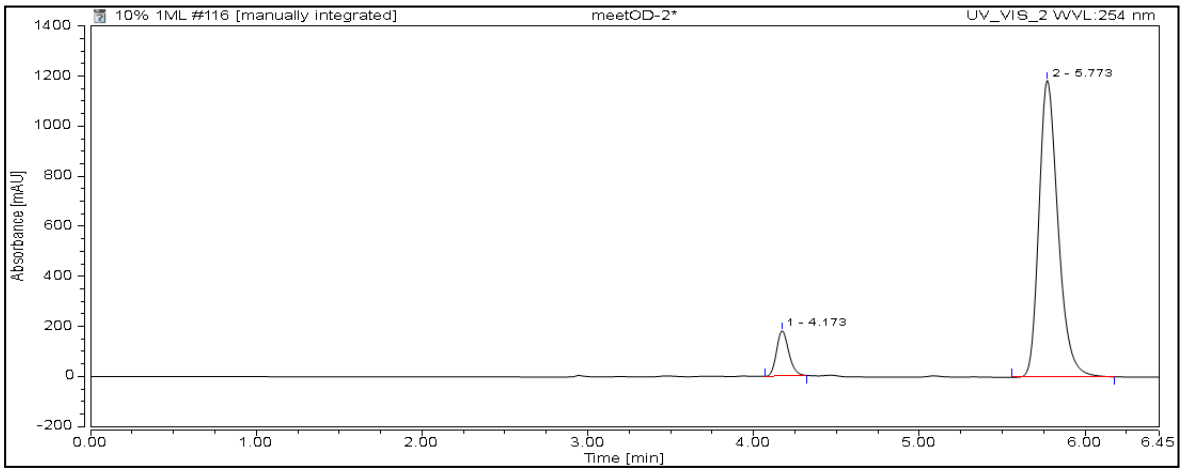
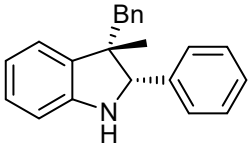


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		4.180	133.769	48.33	n.a.
2		5.827	143.001	51.67	n.a.
Total:			**276.770**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		4.173	15.443	8.89	n.a.
2		5.773	158.253	91.11	n.a.
Total:			**173.697**	**100.00**	



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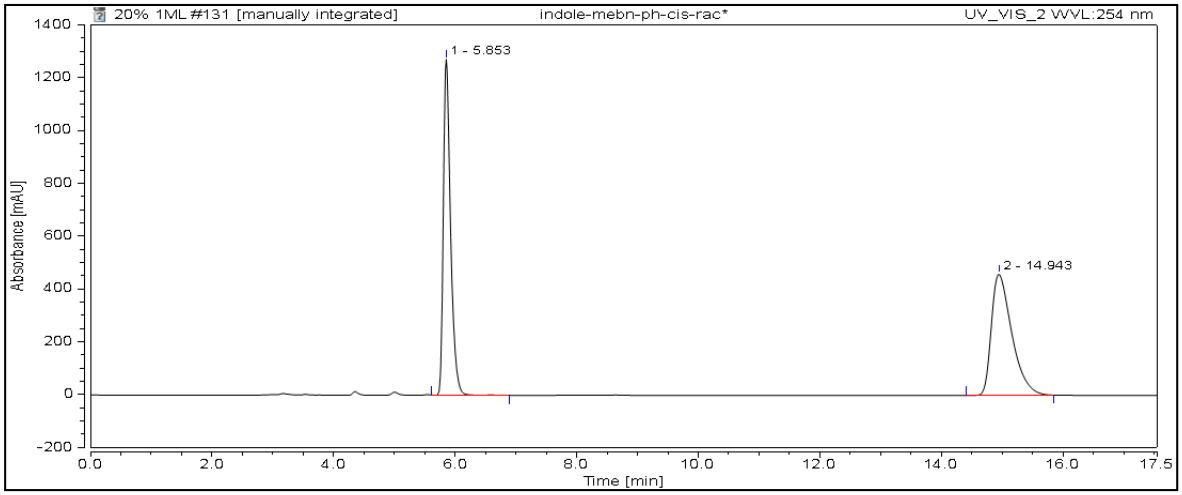
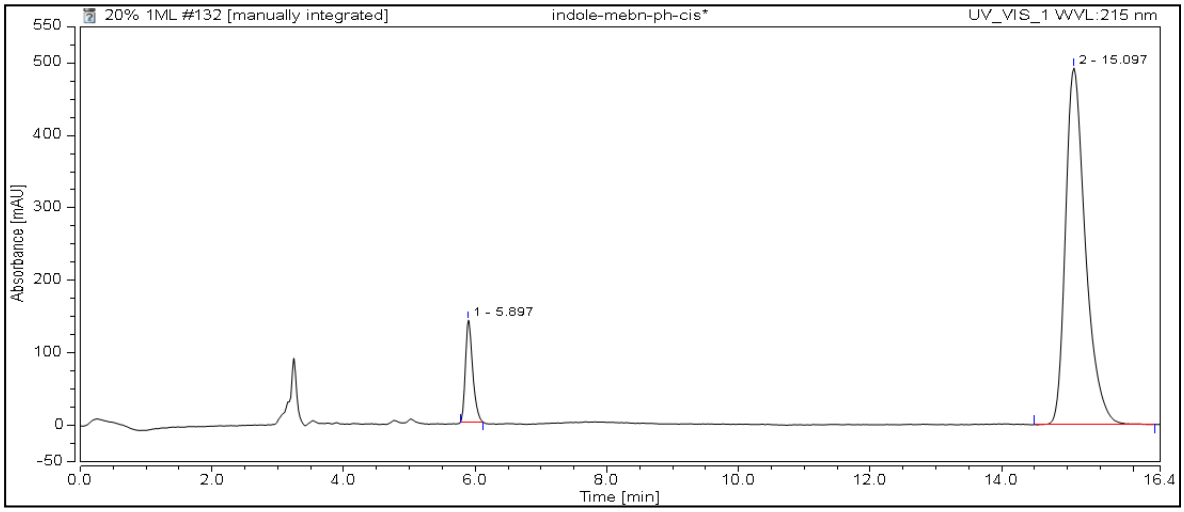
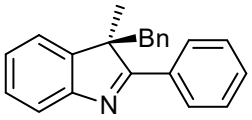


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.853	174.208	49.47	n.a.
2		14.943	177.937	50.53	n.a.
Total:			**352.145**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		5.897	17.960	9.42	n.a.
2		15.097	172.760	90.58	n.a.
Total:			**190.720**	**100.00**	



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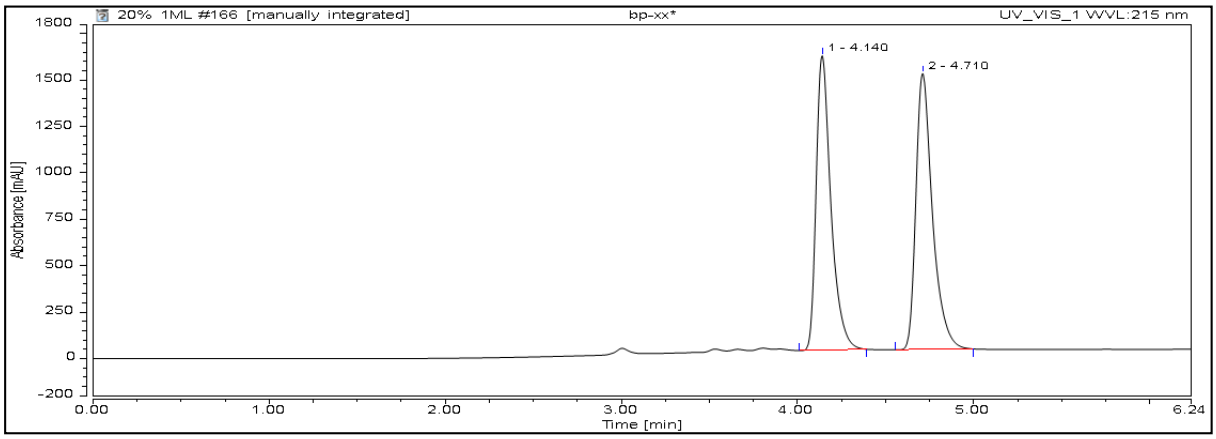
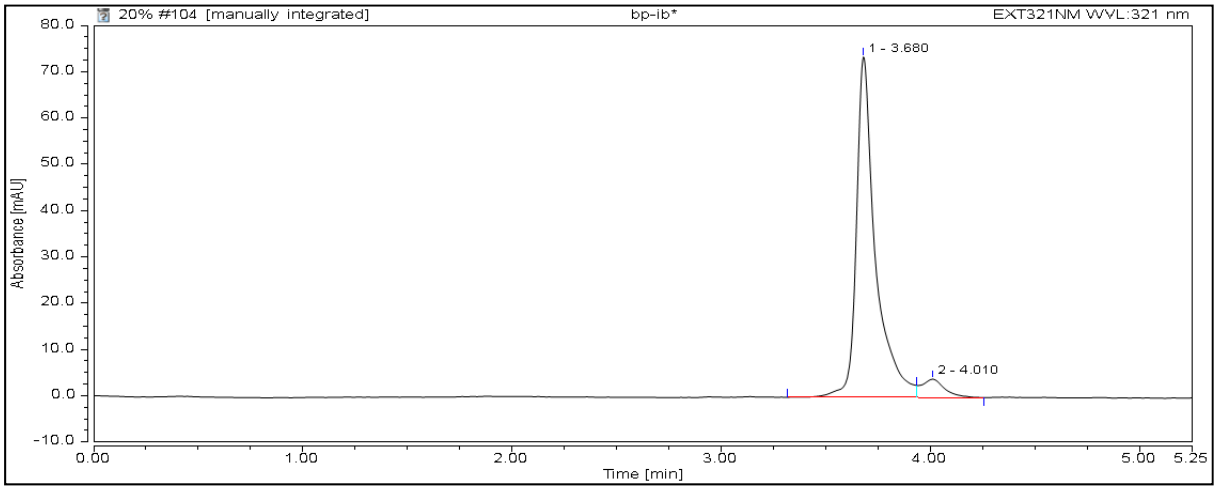
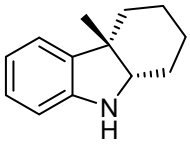


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		4.140	153.921	48.78	n.a.
2		4.710	161.627	51.22	n.a.
Total:			**315.548**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		3.680	7.772	93.92	n.a.
2		4.010	0.503	6.08	n.a.
Total:			**8.275**	**100.00**	



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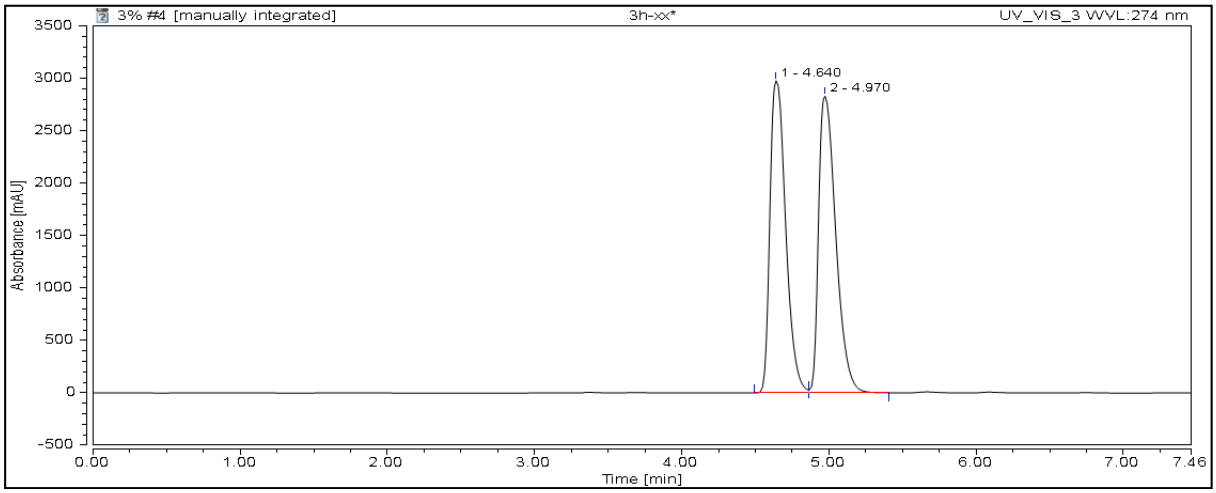
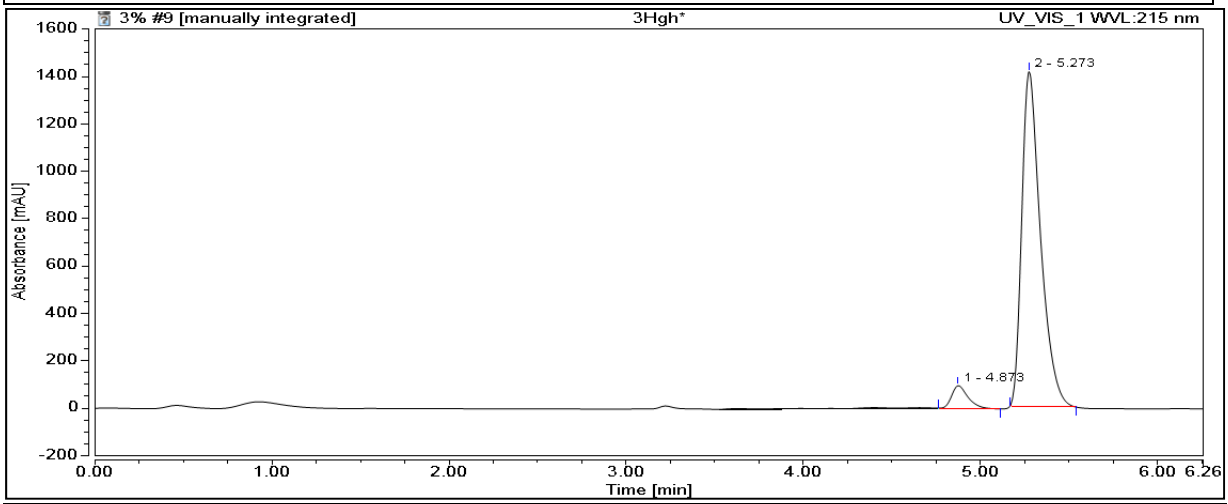
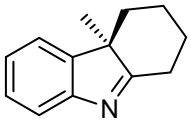


Integration Results

No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		4.640	369.246	49.08	n.a.
2		4.970	383.055	50.92	n.a.
Total:			**752.301**	**100.00**	



No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		4.873	10.754	5.84	n.a.
2		5.273	173.387	94.16	n.a.
Total:			**184.142**	**100.00**	



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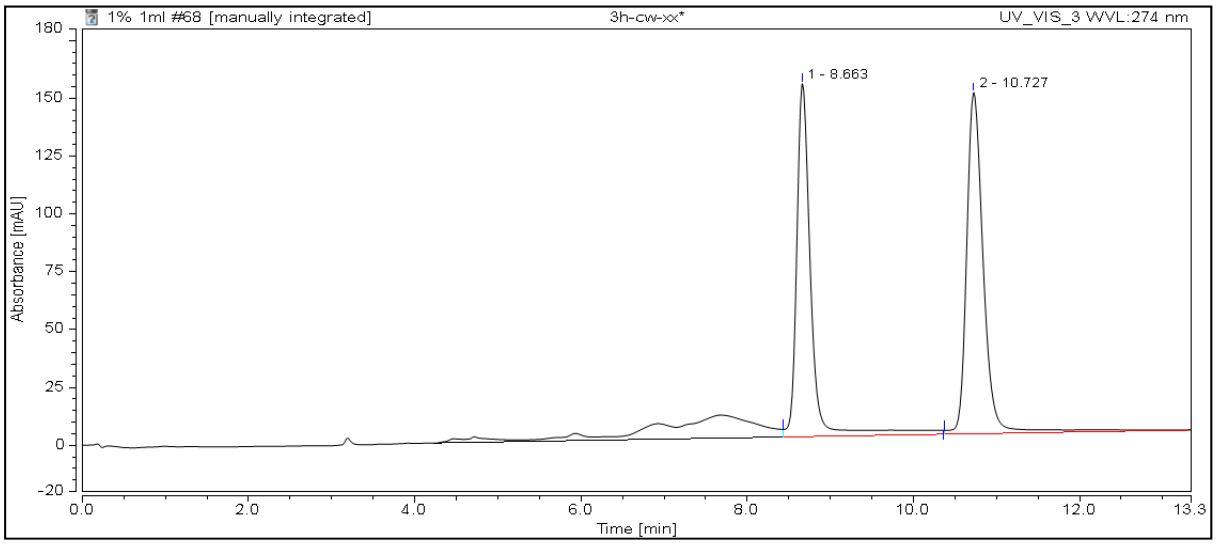
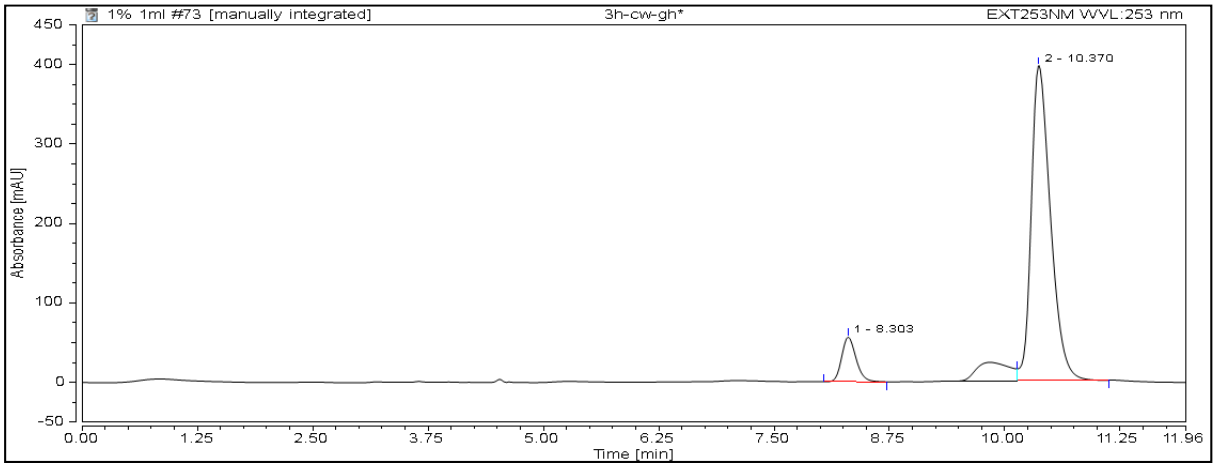
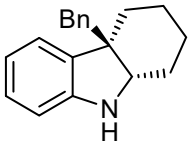


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		8.663	30.716	46.80	n.a.
2		10.727	34.922	53.20	n.a.
Total:			**65.639**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		8.303	10.126	9.57	n.a.
2		10.370	95.682	90.43	n.a.
Total:			**105.809**	**100.00**	



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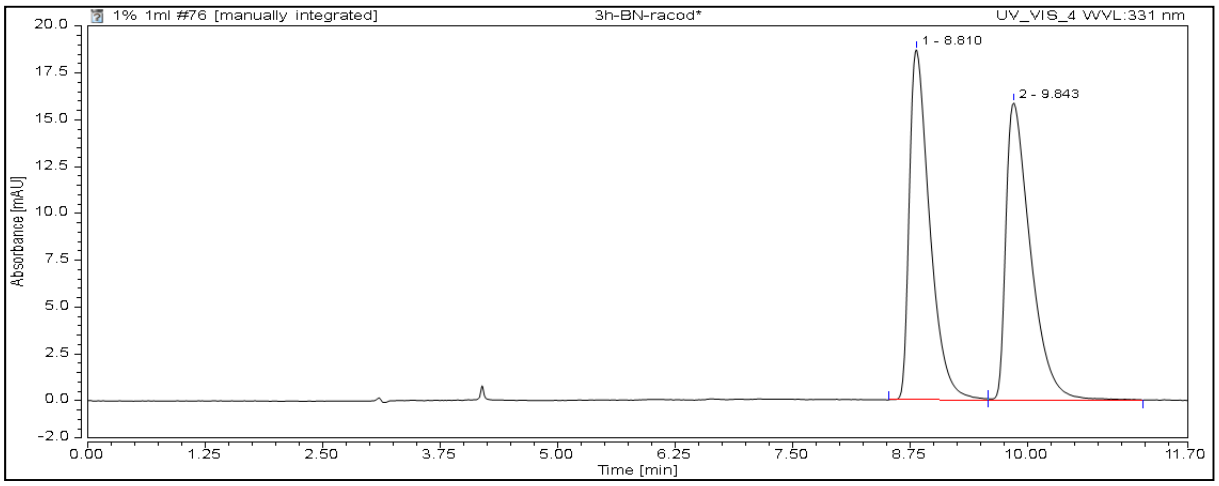
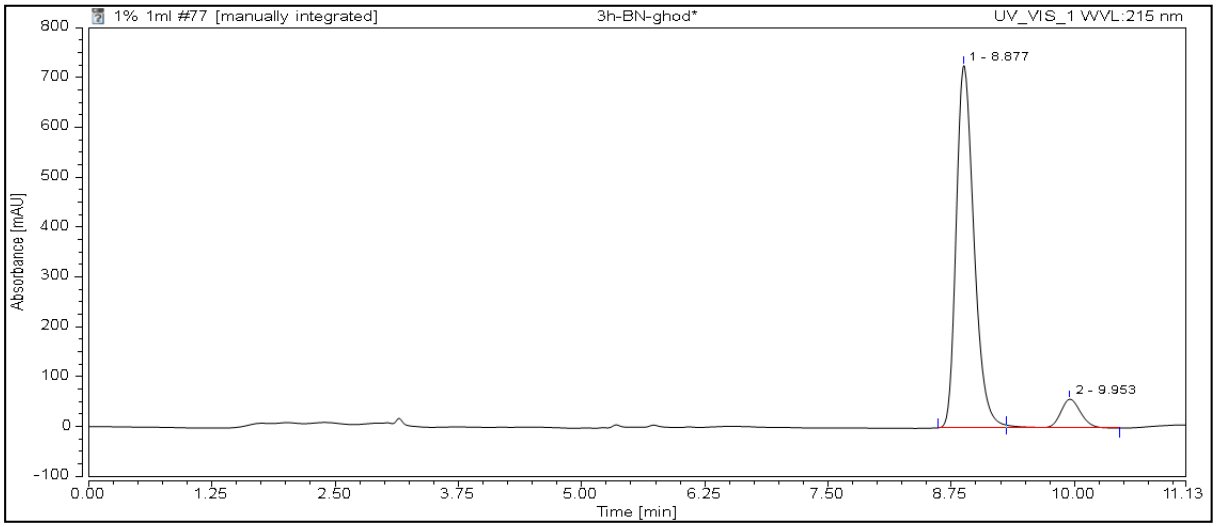
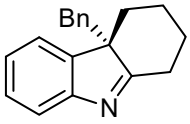


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		8.810	4.707	49.47	n.a.
2		9.843	4.808	50.53	n.a.
Total:			**9.515**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		8.877	152.120	91.42	n.a.
2		9.953	14.270	8.58	n.a.
Total:			**166.390**	**100.00**	



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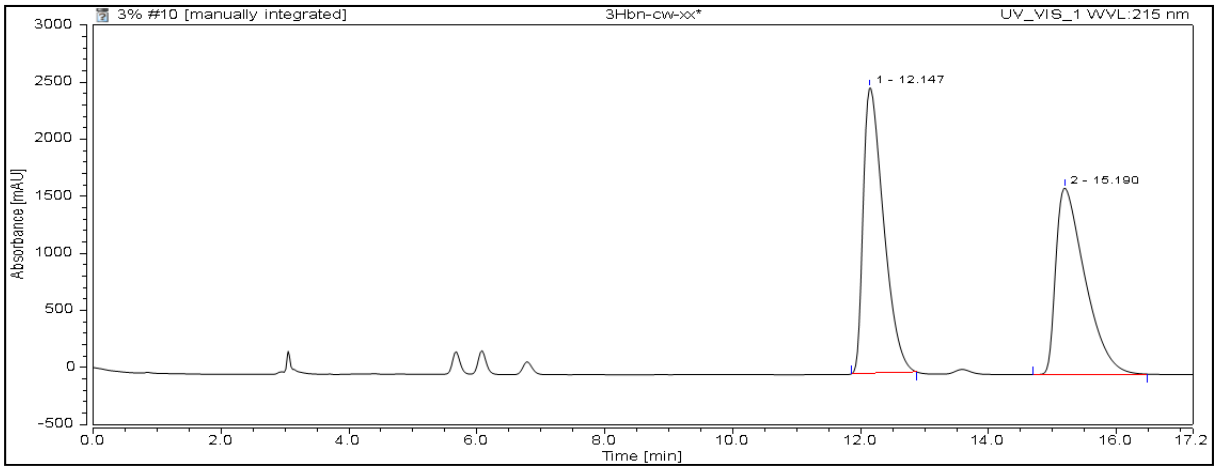
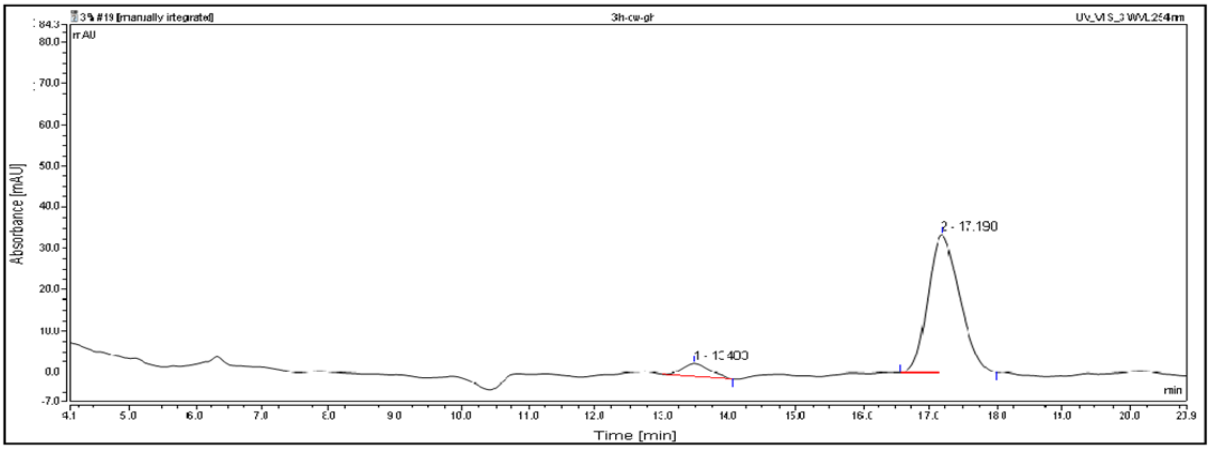
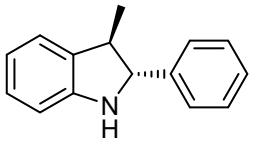


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		12.147	924.048	51.82	n.a.
2		15.190	859.231	48.18	n.a.
Total:			**1783.280**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		13.483	1.4577	7.37	n.a.
2		17.190	18.3214	92.63	n.a.
Total:			**19.7791**	**100.00**	



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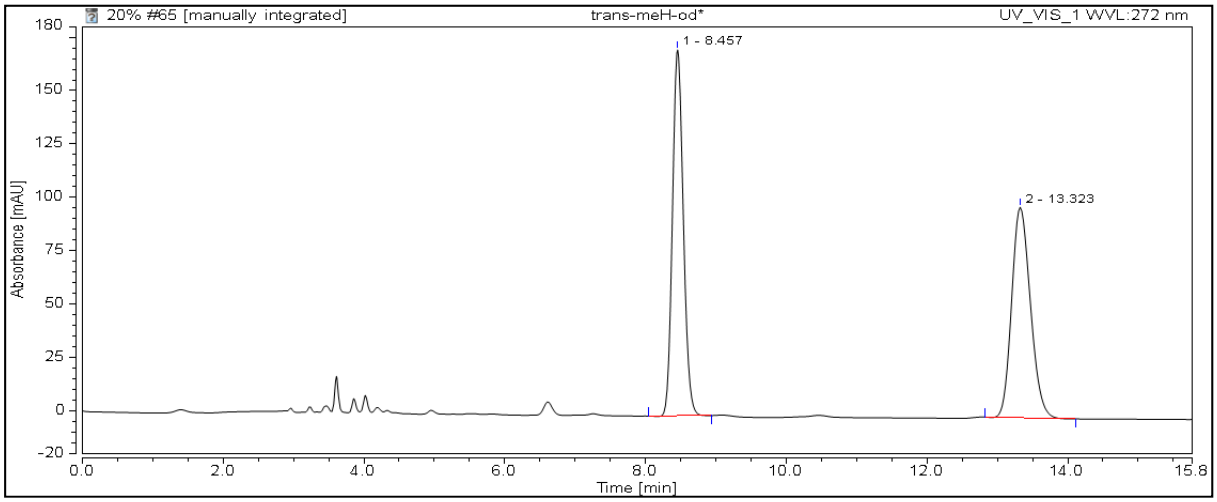
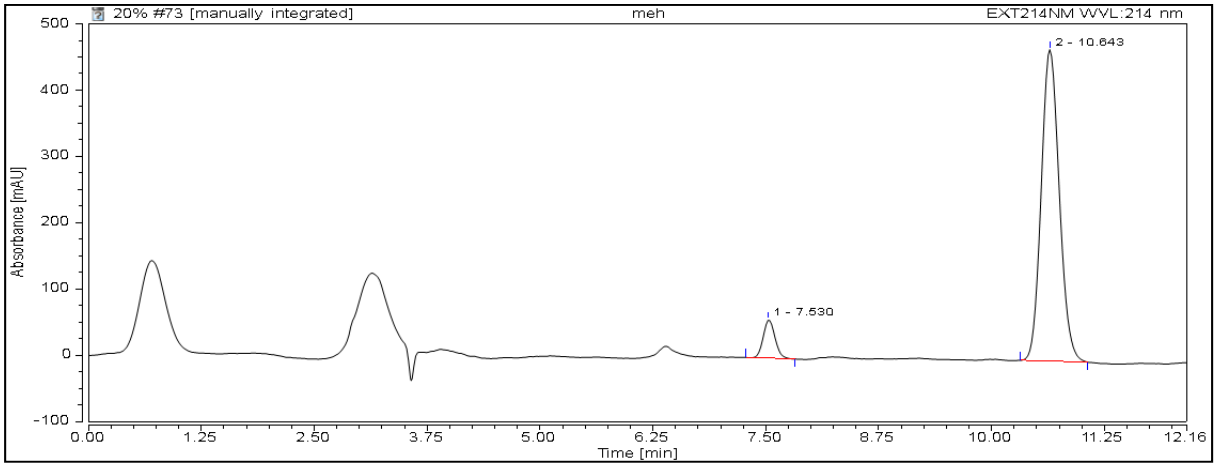
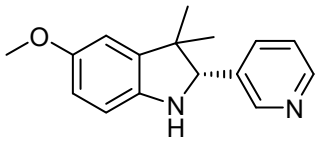


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		8.457	30.600	50.19	n.a.
2		13.323	30.364	49.81	n.a.
Total:			**60.964**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		7.530	8.651	7.34	n.a.
2		10.643	109.164	92.66	n.a.
Total:			**117.815**	**100.00**	



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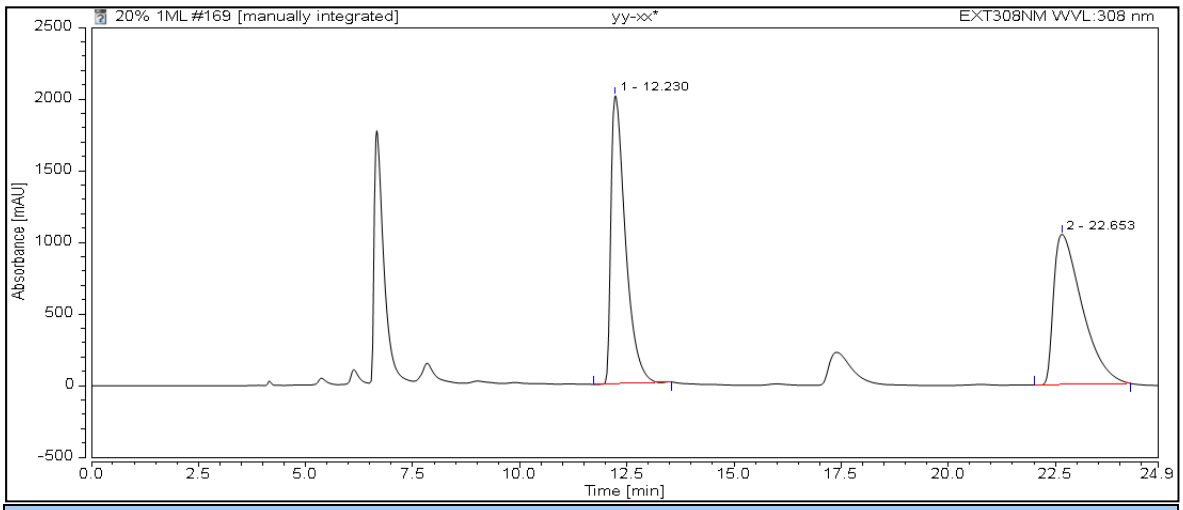
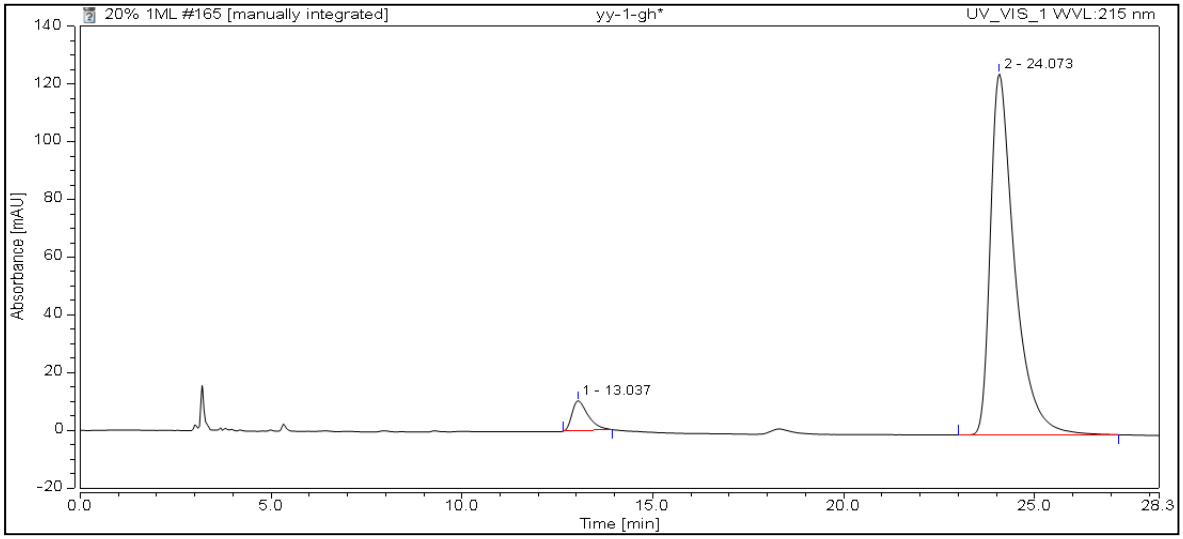
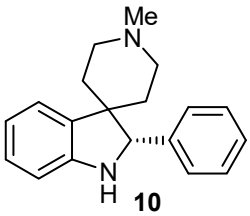


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		12.230	787.497	49.41	n.a.
2		22.653	806.309	50.59	n.a.
Total:			**1593.806**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		13.037	5.074	5.24	n.a.
2		24.073	91.823	94.76	n.a.
Total:			**96.897**	**100.00**	



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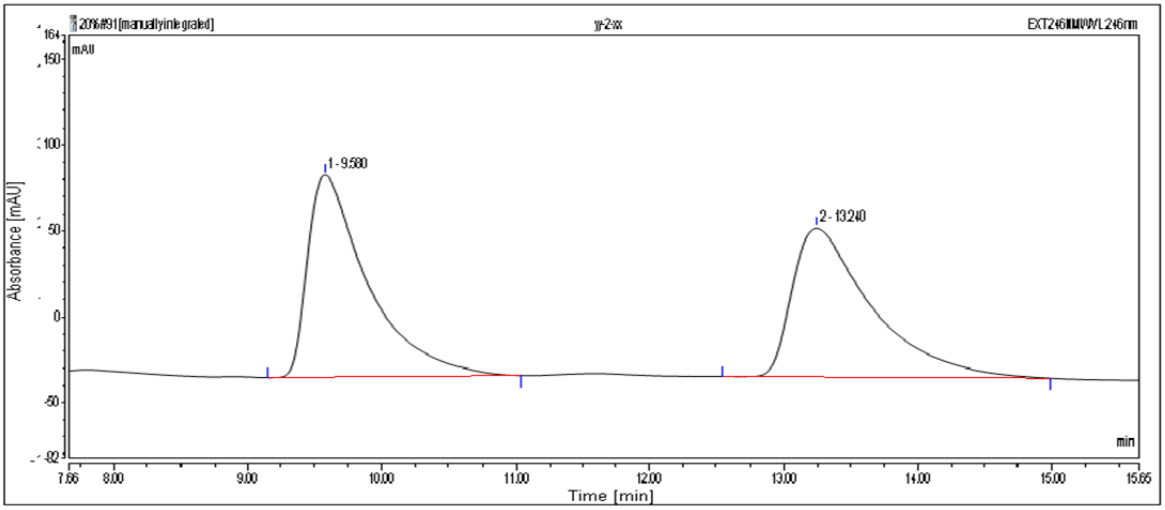
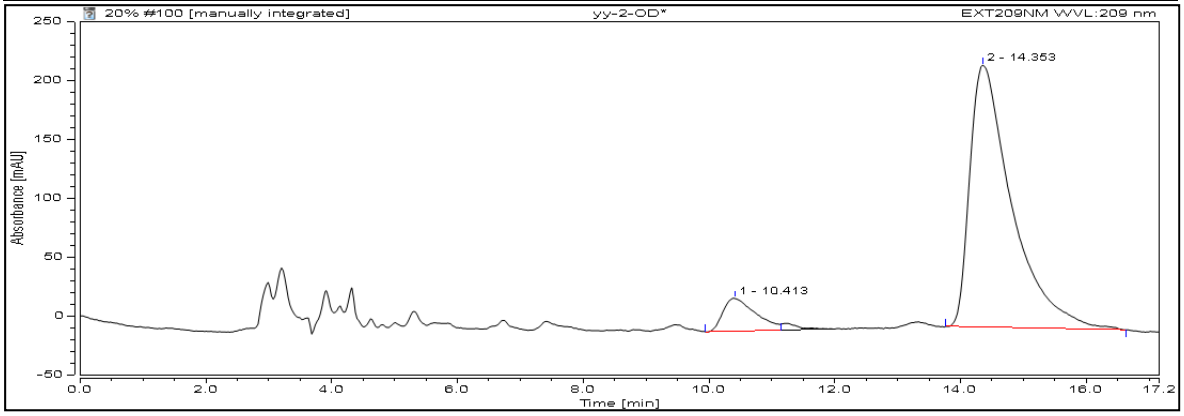
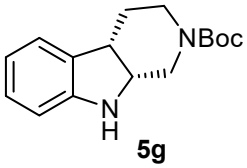


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		9.580	61.578	50.62	n.a.
2		13.240	60.078	49.38	n.a.
Total:			**121.656**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		10.413	16.871	8.93	n.a.
2		14.353	172.116	91.07	n.a.
Total:			**188.987**	**100.00**	



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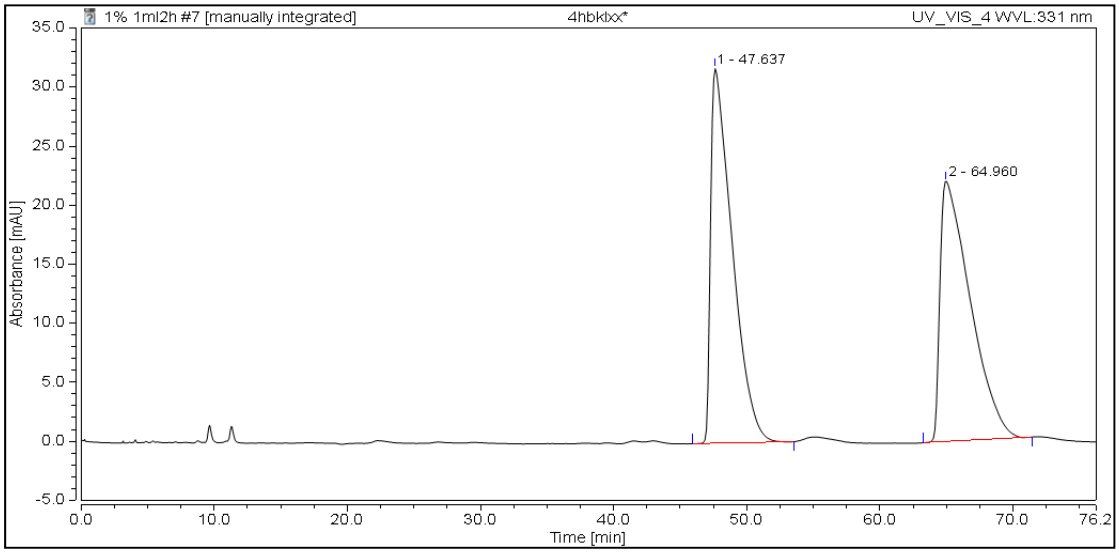
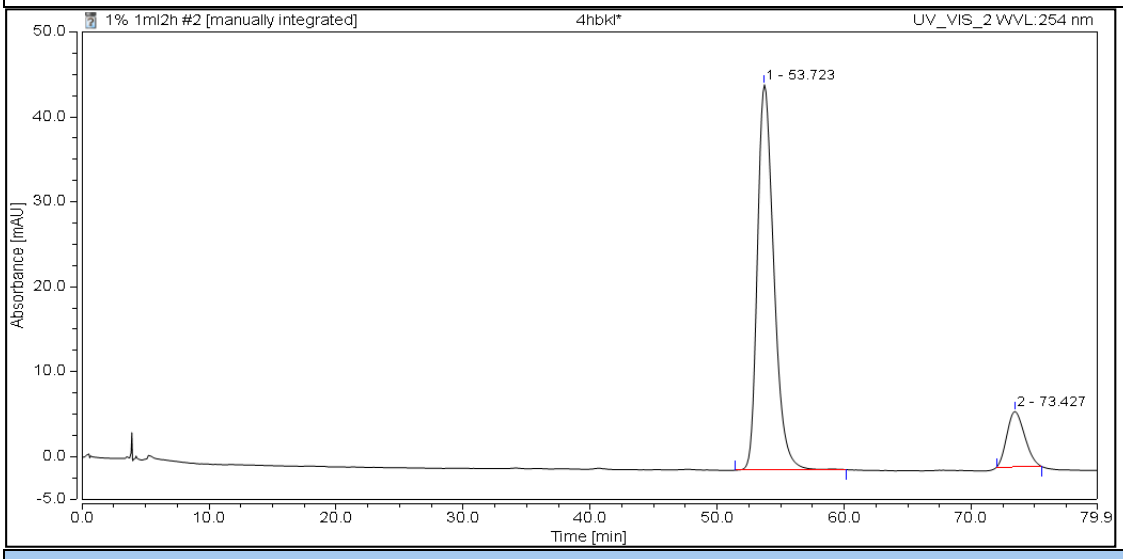
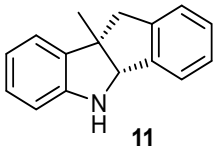


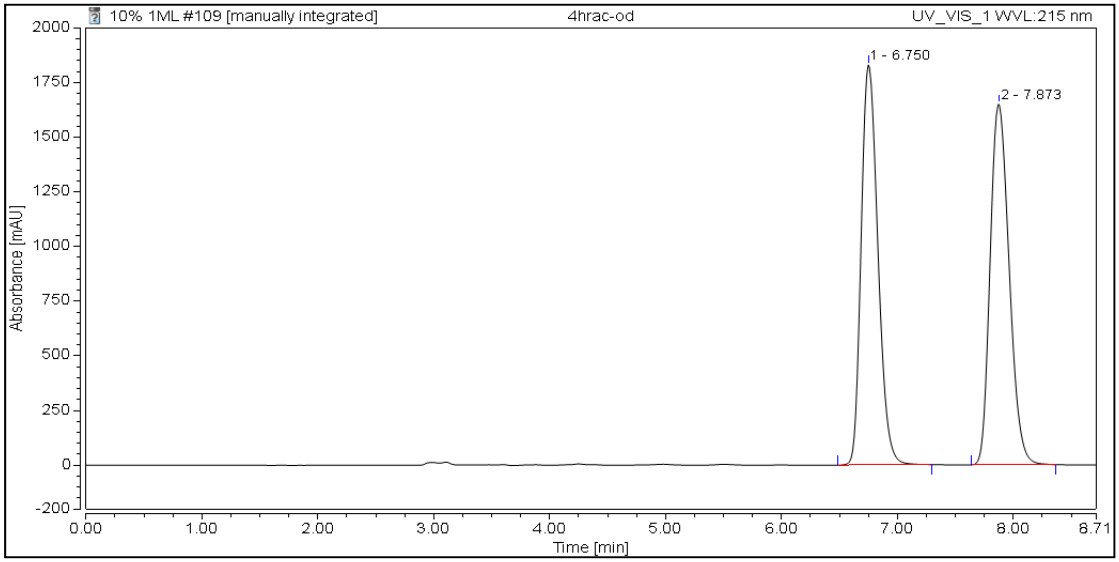
Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		47.637	59.414	50.66	n.a.
2		64.960	57.876	49.34	n.a.
Total:			**117.290**	**100.00**	



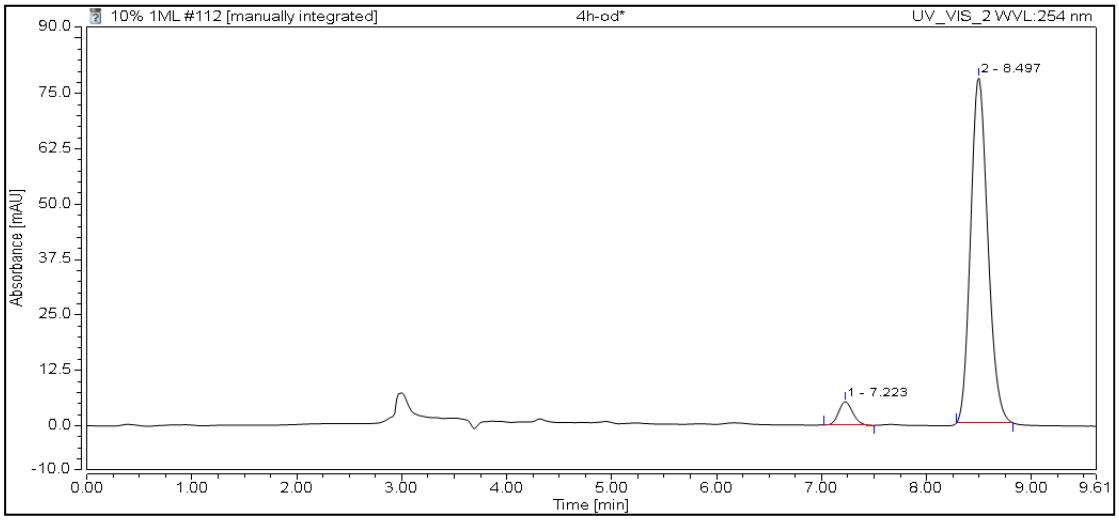
Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		53.723	68.183	86.52	n.a.
2		73.427	10.620	13.48	n.a.
Total:			**78.803**	**100.00**	



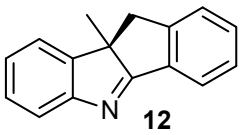
11



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		6.750	306.254	49.44	n.a.
2		7.873	313.208	50.56	n.a.
Total:			**619.462**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		7.223	0.822	5.26	n.a.
2		8.497	14.805	94.74	n.a.
Total:			**15.627**	**100.00**	



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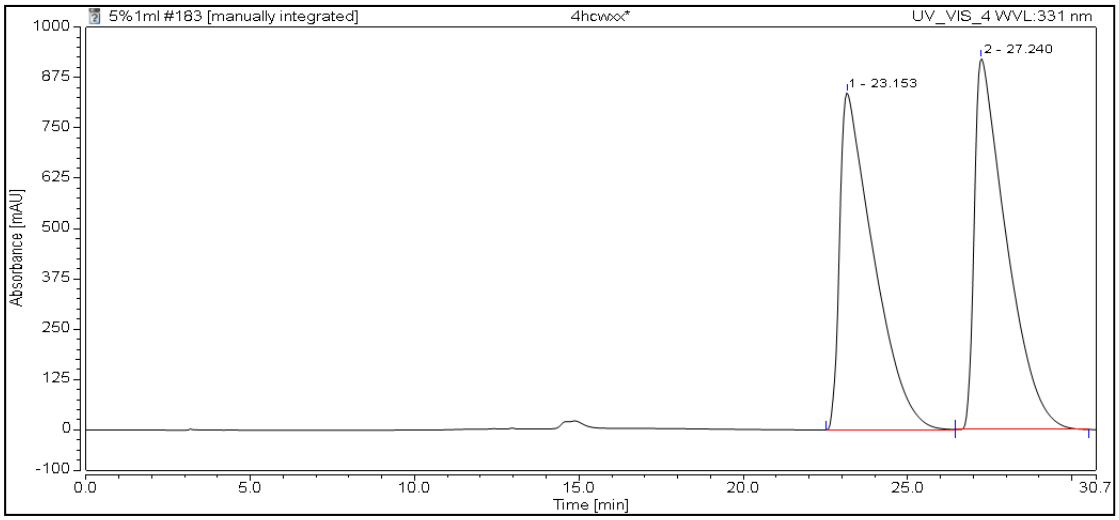
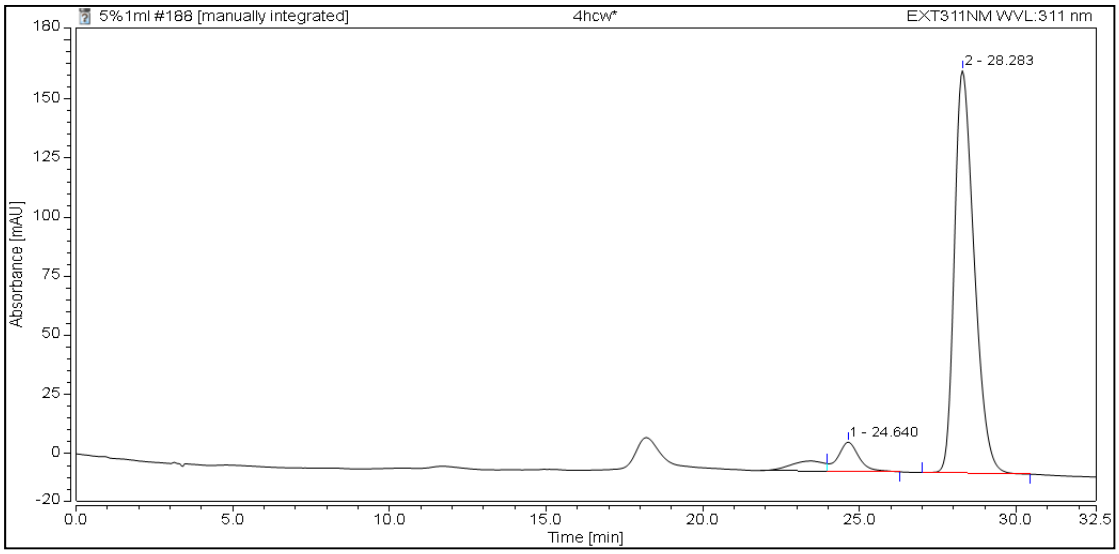


Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		23.153	997.821	48.97	n.a.
2		27.240	1039.921	51.03	n.a.
Total:			**2037.742**	**100.00**	



Integration Results					
No.	Peak Name	Retention Time min	Area mAU*min	Relative Area %	Amount n.a.
1		24.640	9.589	7.15	n.a.
2		28.283	124.486	92.85	n.a.
Total:			**134.074**	**100.00**	