Chiral Brønsted Acid-Catalyzed Friedel-Crafts Alkylation of Electron-Rich Arenes with *In Situ*-Generated *Ortho*-Quinone Methides: Highly Enantioselective Synthesis of Diarylindolylmethanes and Triarylmethanes

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Supporting Information

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General Aspects

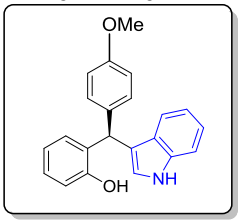
^{1}H and ^{13}C NMR spectra were recorded in $CDCl_3$ and solutions using a Varian Gemini 2000 spectrometer (300 MHz) and BruckerAvance DRX 400 (400 MHz). The signals were referenced to residual chloroform (7.26 ppm, ^{1}H, 77.00 ppm, ^{13}C). Chemical shifts are reported in ppm, multiplicities are indicated by s (singlet)brs (broad singlet), d (doublet), t (triplet), q (quartet), quint (quintet), sext (sextet) and m (multiplet). Melting points were determined uncorrected on a Boetius heating table. IR spectra were obtained with a FTIR spectrometer (Genesis ATI Mattson/Unicam). Optical rotations were measured using a Polarotronicpolarimeter (Schmidt &Haensch). All ESI mass spectra were recorded on a Brucker APEX II FT-ICR. HPLC analyses were carried out on a Jasco MD-2010 plus instrument with chiral stationary phase column (Daicel Chiralcel AD-H, OD-H, OJ-H column or Daicel Chiralpak IA column). The solvents were distilled from indicated drying reagents: dichloromethane (CaH_2), tetrahydrofuran (Na, benzophenone), diethyl ether (Na, benzophenone). Diethyl ether, ethyl acetate and hexane were technical grade and distilled from KOH. Flash column chromatography was performed by using Merck silica gel 60 230-400 mesh (0.040-0.063 mm). All reactions were monitored by thin- layer chromatography using precoated silica gel plates. Spots were visualized by UV and were treated with a solution of vanillin in methanol (technical grade).

General Procedure for the Brønsted Acid-Catalyzed Conjugate Addition of Indoles to *Ortho*-Hydroxybenzhydrols

The *ortho*-hydroxy benzhydryl alcohol **1** (0.21 mmol), the requisite indole **2** (0.21 mmol), and phosphoric acid **3d** (7 mg, 0.01 mmol) were dissolved in 3 mL of dry DCM under argon and the reaction mixture was stirred at rt for the indicated time. The progress of the reaction mixture was monitored by TLC. On disappearance of the starting material the reaction mixture was directly passed through silica gel column with ethyl acetate/hexanes to isolate the pure diarylindolylmethane which were further recrystallized from CH_2Cl_2/hexane.

Spectral Data of Diarylindolylmethanes 4-14:

Diarylindolylmethane 4a:



Yield 94%; yellow solid; **Mpt** = 94-96ºC (CH_2Cl_2/hexanes)

$[\alpha]_D^{24}$ = -12.0 (c = 0.5, $CHCl_3$)

Er = 91:9

IR (KBr) cm^{-1}757, 1243, 1509, 1608, 3405.

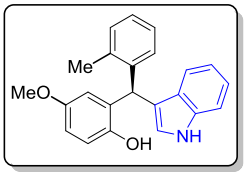
^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.83 (s, 3H), 5.12 (brs, 1H), 5.83 (s, 1H), 6.67 (s, 1H), 6.88-6.90 (m, 4H), 7.00-7.08 (m, 2H), 7.19-7.28 (m, 4H), 7.32-7.38 (m, 2H), 8.01 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 42.6, 55.3, 111.3, 114.1, 116.4, 118.0, 119.8, 119.9, 120.9, 122.5, 124.0, 126.9, 128.0, 130.0, 130.1, 134.4, 137.0, 153.9, 158.4.

ESI-MS+ m/z Calcd. for $C_{22}H_{19}NO_2$ 352.1313 [M+ Na]; found 352.1307.

HPLC OD-H Column (90% hexane: 10% *i*-propanol, 1 mL/min, 288 nm) R_{t1} = 14.3 min and R_{t2} = 18.0 min.

Diarylindolylmethane 4b:



Yield 87%; yellow solid; **Mpt** = 60-62 ºC (CH_2Cl_2/hexane)
[α]$_D^{24}$ = -9.0 (c = 0.1, $CHCl_3$)
Er = 95:5
IR (film) cm^{-1} 743, 1199, 1430, 1455, 1503, 3425.

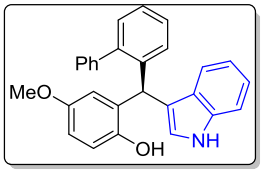
^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.31 (s, 3H), 3.63 (s, 3H), 4.60 (brs, 1H), 5.92 (s, 1H), 6.47 (d, 1H, *J* = 4.0 Hz), 6.60 (brs, 1H), 6.70 (dd, 1H, J_1 = 4.0 Hz, J_2 = 12.0 Hz), 6.79 (d, 1H, *J* = 12.0 Hz), 6.99-7.14 (m, 3H), 7.16-7.30 (m, 4H), 7.36 (d, 1H, *J* = 8.0 Hz), 8.01 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 19.6, 39.8, 55.6, 111.3, 112.1, 116.2, 116.9, 117.1, 119.8, 119.8 122.5, 124.2, 126.2, 126.8, 127.0, 128.6, 130.6, 130.9, 136.7, 137.0, 140.4, 148.0, 153.8.

ESI-MS+ m/z Calcd. for $C_{23}H_{21}NO_2$ 366.1470 [M+ Na]; found 366.1464.

HPLC AD-H Column (80% hexane: 20% *i*-propanol, 1 mL/min, 232 nm) R_{t1} = 11.2 min and R_{t2} = 14.4 min.

Diarylindolylmethane 4c:



Yield 85%; yellow solid; **Mpt** = 98-100 ºC (CH_2Cl_2/hexane)
[α]$_D^{24}$ = -15.5 (c = 0.16, $CHCl_3$)
Er = 95:5
IR (film) cm^{-1} 746, 1501, 3423.

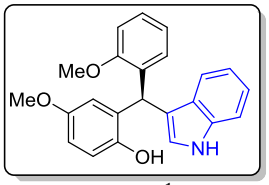
^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.56 (s, 3H), 4.26 (s, 1H), 5.75 (s, 1H), 6.55 (d, 1H, *J* = 4.0 Hz), 6.68-6.76 (m, 3H), 6.99 (t, 1H, *J* = 8.0 Hz), 7.11 (d, 1H, *J* = 8.0 Hz), 7.18-7.23 (m, 3H), 7.28-7.38 (m, 9H), 8.03 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 39.9, 55.5, 111.1, 112.0, 116.2, 116.9, 117.8, 119.6, 119.8, 122.4, 124.3, 126.5, 126.6, 127.2, 127.6, 128.1, 129.0, 129.5, 130.3, 131.5, 136.9, 139.6, 141.2, 141.9, 147.5, 153.6.

ESI-MS+ m/z Calcd. for $C_{28}H_{23}NO_2$ 428.1626 [M+ Na]; found 428.1619.

HPLC AD-H Column (80% hexane: 20% *i*-propanol, 1 mL/min, 288 nm) R_{t1} = 14.3 min and R_{t2} = 18.0 min.

Diarylindolylmethane 4d:



Yield 83%; pale yellow solid; **Mpt** = 80-82 ºC (CH_2Cl_2/hexane)

[α]$_D^{24}$ = -14.1 (c = 0.12, $CHCl_3$)

Er = 95:5

IR (film) cm^{-1} 754, 1241, 1489, 3418.

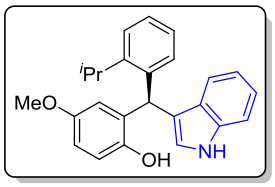
^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.67 (s, 3H), 3.85 (s, 3H), 5.31 (s, 1H), 6.19 (s, 1H), 6.63 (d, 1H, *J* = 4.0 Hz), 6.72 (dd, 1H, J_1= 4.0 Hz, J_2 = 8.0 Hz), 6.76 (brs, 1H), 6.85 (d, 1H, *J* = 8.0 Hz), 6.91 (t, 1H, *J* = 8.0 Hz), 6.97 (d, 1H, *J* = 8.0 Hz), 7.02 (t, 1H, *J* = 8.0 Hz), 7.16-7.21 (m, 2H), 7.25-7.28 (m, 2H), 7.36 (d, 1H, *J* = 8.0 Hz), 8.04 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 35.8, 55.8, 56.0, 111.0, 111.3, 112.3, 115.8, 117.0, 117.1, 119.8, 120.2, 121.2, 122.5, 124.2, 127.2, 128.1, 130.1, 130.6, 131.1, 137.2, 148.3, 153.7, 156.6.

ESI-MS+ m/z Calcd. for $C_{23}H_{21}NO_3$ 382.1419 [M+ Na]; found 382.1413

HPLC AD-H Column (70% hexane: 30% *i*-propanol, 1 mL/min, 225 nm) R_{t1} = 14.7 min and R_{t2} = 16.5 min.

Diarylindolylmethane 4e:



Yield 85%; white solid; **Mpt** = 140-142 ºC (CH_2Cl_2/hexane)

[α]$_D^{24}$ = -12.5 (c = 0.16, $CHCl_3$)

Er = 96:4

IR (film) cm^{-1} 743, 1503, 3419.

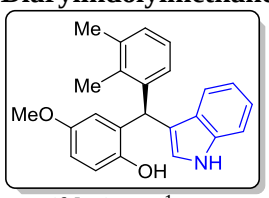
^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.17 (d, 3H, *J* = 8.0 Hz), 1.22 (d, 3H, *J* = 8.0 Hz), 3.25 (sep, 1H, *J* = 8.0 Hz), 3.64 (s, 3H), 4.54 (s, 1H), 6.07 (s, 1H), 6.47 (d, 1H, *J* = 4.0 Hz), 6.63 (brs, 1H), 6.72 (dd, 1H, J_1 = 4.0 Hz, J_2 = 8.0 Hz), 6.81 (d, 1H, *J* = 8.0 Hz), 6.99-7.09 (m, 3H), 7.20-7.30 (m, 3H), 7.38-7.41 (m, 2H), 8.03 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 23.9, 24.2, 28.5, 39.0, 55.6, 111.2, 112.1, 116.4, 116.9, 117.6, 119.7, 119.8, 122.5, 124.4, 125.7, 125.8, 126.8, 127.2, 128.7, 131.4, 137.0, 138.6, 147.2, 147.7, 153.7.

ESI-MS+ m/z Calcd. for $C_{25}H_{25}NO_2$ 394.1783 [M+ Na]; found 394.1775.

HPLC IA Column (85% hexane: 15% *i*-propanol, 1 mL/min, 232 nm) R_{t1} = 14.8 min and R_{t2} = 17.3 min.

Diarylindolylmethane 4f:



Yield 91%; yellow solid; **Mpt** = 76-78 ºC (CH_2Cl_2/hexane)

[α]$_D^{24}$ = -20.2 (c = 0.2, $CHCl_3$)

Er = 95:5

IR (film) cm^{-1} 743, 1504, 3418.

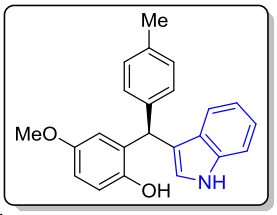
^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.21 (s, 3H), 2.34 (s, 3H), 3.65 (s, 3H), 4.55 (brs, 1H), 5.97 (s, 1H), 6.48 (d, 1H, *J* = 4.0Hz), 6.64 (brs, 1H), 6.73 (dd, 1H, J_1 = 4.0 Hz, J_2 = 8.0 Hz), 6.87 (d, 1H, *J* = 4.0 Hz), 6.97-7.10 (m, 3H), 7.21 (t, 1H, *J* = 8.0 Hz), 7.31 (d, 2H, *J* = 8.0 Hz), 7.39 (d, 1H, *J* = 8.0 Hz), 8.03 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 15.1, 21.0, 40.1, 55.6, 111.3, 112.0, 116.4, 116.8, 117.2, 119.6, 119.7, 122.4, 124.4, 125.5, 126.4, 127.0, 128.6, 131.3, 135.1, 136.9, 137.0, 140.3, 147.9, 153.7.

ESI-MS+ m/z Calcd. for $C_{24}H_{23}NO_2$ 380.1626 [M+ Na]; found 380.1620.

HPLC AD-H Column (80% hexane: 20% *i*-propanol, 1 mL/min, 265 nm) R_{t1} = 12.4 min and R_{t2} = 16.3 min.

Diarylindolylmethane 4g:



Yield 86%; reddish yellowsolid; **Mpt** = 66-68 ºC (CH_2Cl_2/hexane)
[α]$_D^{24}$ = -8.0 (c = 0.12, $CHCl_3$)
Er = 92:8
IR (KBr) cm^{-1}751, 1621, 3462.

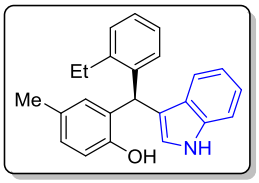
^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.35 (s, 3H), 3.67 (s, 3H), 4.64 (s, 1H), 5.77 (s, 1H), 6.57 (d, 1H, *J* = 4.0 Hz), 6.71-6.74 (m, 2H), 6.81 (d, 1H, *J* = 8.0 Hz), 7.04 (t, 1H, *J* = 8.0 Hz), 7.12-7.23 (m, 5H), 7.33 (d, 1H, *J* = 8.0 Hz), 7.39 (d, 1H, *J* = 8.0 Hz), 8.05 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 21.2, 43.3, 55.7, 111.3, 112.3, 116.3, 117.1, 117.6, 119.8, 119.9, 122.5, 124.0, 126.9, 128.9, 129.4, 131.4, 136.3, 137.0, 139.1, 148.0, 153.7.

ESI-MS+ m/z Calcd. for $C_{23}H_{21}NO_2$ 366.1470 [M+ Na]; found 366.1464.

HPLC AD-H Column (80% hexane: 20% *i*-propanol, 1 mL/min, 232 nm) R_{t1} = 15.3 min and R_{t2} = 17.9 min.

Diarylindolylmethane 4h:



Yield 92%; yellow solid; **Mpt** = 72-74 ºC (CH_2Cl_2/hexane)
[α]$_D^{24}$ = -7.5 (c = 0.14, $CHCl_3$)
Er = 95:5
IR (KBr) cm^{-1} 743, 1632, 3419.

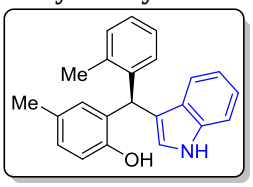
^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.22 (t, 3H, *J* = 8.0 Hz), 2.18 (s, 3H), 2.69 (dq, J_1 = 4.0 Hz, J_2 = 12.0 Hz, 1H), 4.76 (s, 1H), 6.01 (s, 1H), 6.61 (brs, 1H), 6.71 (brs, 1H), 6.76 (d, 1H, *J* = 8.0 Hz), 6.97 (d, 1H, *J* = 8.0 Hz), 7.05 (d, 1H, *J* = 8.0 Hz), 7.11 (t, 1H, *J* = 8.0 Hz), 7.20-7.31 (m, 5H), 7.40 (d, 1H, *J* = 8.0 Hz), 8.01 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 15.1, 27.8, 25.5, 39.4, 111.2, 116.1, 117.8, 119.7, 119.8, 122.4, 124.3, 125.9, 126.8, 126.9, 128.3, 128.7, 128.8, 129.4, 129.9, 130.5, 137.0, 140.0, 142.3, 151.6.

ESI-MS+ m/z Calcd. for $C_{24}H_{23}NO$ 364.1677 [M+ Na]; found 364.1671.

HPLC AD-H Column (80% hexane: 20% *i*-propanol, 1 mL/min, 250 nm) R_{t1} = 12.3 min and R_{t2} = 17.4 min.

Diarylindolylmethane 4i:



Yield 82%; yellow solid; **Mpt** = 86-88 ºC (CH_2Cl_2/hexane)

[α]$_D^{24}$ = -14.2 (c = 0.18, $CHCl_3$)

Er = 94:6

IR (film) cm^{-1} 744, 1094, 3417.

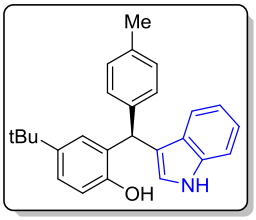
^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.19 (s, 3H), 2.32 (s, 3H), 4.76 (s, 1H), 5.92 (s, 1H), 6.63 (brs, 1H), 6.71 (brs, 1H), 6.76 (d, 1H, *J* = 8.0 Hz), 6.98 (d, 1H, *J* = 8.0 Hz), 7.02-7.06 (m, 2H), 7.11 (t, 1H, *J* = 4.0 Hz), 7.19-7.21 (m, 3H), 7.22-7.30 (m, 1H), 7.40 (d, 1H, *J* = 8.0 Hz), 8.03 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 35.7, 55.7, 55.9, 110.9, 111.2, 112.1, 115.7, 116.9, 117.0, 119.6, 120.1, 121.1, 122.4, 124.1, 127.1, 128.0, 130.0, 130.5, 131.0, 137.0, 148.1, 153.6, 156.5.

ESI-MS+ m/z Calcd. for $C_{23}H_{21}NO$ 350.1521 [M+ Na]; found 350.1515.

HPLC AD-H Column (80% hexane: 20% *i*-propanol, 1 mL/min, 232 nm) R_{t1} = 11.1 min and R_{t2} = 16.3 min.

Diarylindolylmethane 4j:



Yield 87%; reddish brown solid; **Mpt** = 138-140 ºC (CH_2Cl_2/hexane)

[α]$_D^{24}$ = +40.0 (c = 0.1, $CHCl_3$)

Er = 92:8

IR (film) cm^{-1} 741, 1508, 3419.

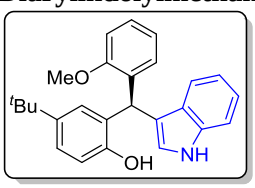
^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.20 (s, 9H), 2.36 (s, 3H), 4.88 (s, 1H), 5.76 (s, 1H), 6.73 (brs, 1H), 6.79 (d, 1H, *J* = 8.0 Hz), 7.01-7.06 (m, 2H), 7.13-7.23 (m, 6H), 7.28-7.31 (m, 1H), 7.39 (d, 1H, *J* = 8.0 Hz), 8.03 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 21.2, 31.6, 34.2, 44.0, 111.3, 116.0, 117.9, 119.7, 120.0, 122.5, 124.0, 124.6, 127.0, 127.4, 128.9, 129.0, 129.4, 136.3, 137.0, 139.4, 143.4, 151.8.

ESI-MS+ m/z Calcd. for $C_{26}H_{27}NO$ 392.1990[M+ Na]; found 392.1984.

HPLC AD-H Column (90% hexane: 10% *i*-propanol, 1 mL/min, 220 nm) R_{t1} = 24.0 min and R_{t2} = 26.1 min.

Diarylindolylmethane 4k:



Yield 82%; yellow solid; **Mpt** = 76-78ºC (CH_2Cl_2/hexane)

[α]$_D^{24}$ = -8.2 (c = 0.13, $CHCl_3$)

Er = 93:7

IR (KBr) cm^{-1}743, 1242, 1489, 2961, 3418.

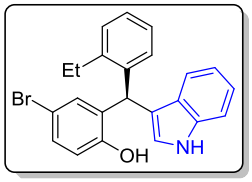
^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.20 (s, 9H), 3.85 (s, 3H), 5.61 (s, 1H), 6.16 (s, 1H), 6.77 (s, 1H), 6.84 (d, 1H, *J* = 8.0 Hz), 6.92 (t, 1H, *J* = 8.0 Hz), 6.96-7.02 (m, 2H), 7.13 (s, 1H), 7.18-7.22 (m, 4H), 7.25-7.29 (m, 1H), 7.37 (d, 1H, *J* = 8.0 Hz), 8.02 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 31.5, 34.1, 36.1, 55.8, 110.8, 111.1, 115.7, 117.1, 119.4, 120.2, 121.0, 122.2, 124.0, 124.2, 126.8, 127.0, 127.8, 128.5, 129.8, 130.8, 136.9, 143.0, 151.8, 156.4.

ESI-MS+ m/z Calcd. for $C_{26}H_{27}NO_2$408.1939 [M+ Na]; found 408.1933.

HPLC OD-H Column (95% hexane: 5% *i*-propanol, 1 mL/min, 232 nm) R_{t1} = 21.02 min and R_{t2} = 24.96 min.

Diarylindolylmethane 4l:



Yield 88%; yellow solid; **Mpt** = 80-82 ºC (CH_2Cl_2/hexane)

[α]$_D^{24}$ = -7.7 (c = 0.13, $CHCl_3$)

Er = 97:3

IR (film) cm^{-1} 744, 1486, 3420.

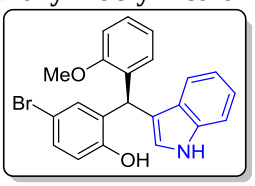
^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.22 (t, 3H, *J* = 8.0 Hz), 2.68 (q, 2H, *J* = 8.0 Hz), 4.95 (s, 1H), 5.99 (s, 1H), 6.62 (brs, 1H), 6.75 (d, 1H, *J* = 8.0 Hz), 7.01-7.14 (m, 4H), 7.22-7.30 (m, 5H), 7.41 (d, 1H, *J* = 8.0 Hz), 8.05 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 15.1, 25.5, 38.9, 111.3, 113.2, 116.9, 118.1, 119.5, 119.9, 122.7, 124.4, 126.1, 126.7, 127.2, 128.6, 128.9, 130.7, 132.2, 132.6, 136.9, 139.1, 142.3, 153.0.

ESI-MS+ m/z Calcd. for $C_{23}H_{20}BrNO$ 428.0626 [M+ Na]; found 426.0619.

HPLC IA Column (85% hexane: 15% *i*-propanol, 1 mL/min, 232 nm) R_{t1} = 9.8 min and R_{t2} = 12.1 min.

Diarylindolylmethane 4m:



Yield 84%; yellow solid; **Mpt** = 180-182 ºC (CH_2Cl_2/hexane)

[α]$_D^{24}$ = -13.3 (c = 0.24, $CHCl_3$)

Er = 99:1

IR (film) cm^{-1} 752, 1488, 3420.

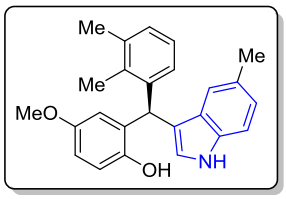
^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.86 (s, 3H), 5.68 (s, 1H), 6.14 (s, 1H), 6.78 (d, 2H, *J* = 8.0 Hz), 6.93 (t, 1H, *J* = 8.0 Hz), 6.98 (d, 1H, *J* = 8.0 Hz), 7.04 (t, 1H, *J* = 8.0 Hz), 7.14-7.28 (m, 6H), 7.40 (d, 1H, *J* = 8.0 Hz), 8.05 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 35.5, 55.8, 110.9, 111.3, 112.8, 116.1, 118.3, 119.7, 119.8, 121.2, 122.5, 124.1, 126.8, 128.2, 129.7, 130.6, 131.9, 136.9, 153.3, 156.3.

ESI-MS+ m/z Calcd. for $C_{22}H_{18}BrNO_2$ 430.0419 [M+ Na]; found 430.0414.

HPLC IA Column (85% hexane: 15% *i*-propanol, 1 mL/min, 232 nm) R_{t1} = 17.7 min and R_{t2} = 20.3 min.

Diarylindolylmethane 5a:



Yield 88%; pale yellow solid; **Mpt** = 90-92 °C (CH_2Cl_2/hexane)

[α]$_D^{25}$ = -8.0 (c = 0.25, $CHCl_3$)

Er = 94:6

IR (film) cm^{-1} 1645, 3420.

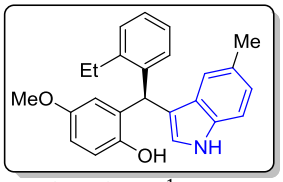
^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.23 (s, 3H), 2.37 (s, 3H), 2.40 (s, 3H), 3.68 (s, 3H), 4.77 (brs, 1H), 5.97 (s, 1H), 6.51 (s, 1H), 6.55 (s, 1H), 6.74 (d, 1H, *J* = 8.0 Hz), 6.81-6.83 (m, 1H), 6.88-6.90 (m, 1H), 7.00-7.06 (m, 2H), 7.11-7.15 (m, 2H), 7.24-7.28 (m, 1H), 7.95 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 15.2, 21.0, 21.6, 40.2, 55.6, 111.0, 112.0, 116.4, 116.7, 116.9, 119.2, 124.2, 124.6, 125.5, 126.4, 127.3, 128.7, 129.0, 131.5, 135.2, 135.3, 137.0, 140.0, 148.0, 153.8.

ESI-MS+ m/z Calcd. for $C_{25}H_{25}NO_2$ 394.1783 [M+ Na]; found 394.1776.

HPLC IA Column (85% hexane: 15% *i*-propanol, 1 mL/min, 274 nm) R_{t1} = 10.4 min and R_{t2} = 12.0 min.

Diarylindolylmethane 5b:



Yield 86%; reddish yellow solid; **Mpt** = 76-78 °C (CH_2Cl_2/hexane)

[α]$_D^{24}$ = -7.1 (c = 0.28, $CHCl_3$)

Er = 95:5

IR (KBr) cm^{-1} 798, 1504, 3419.

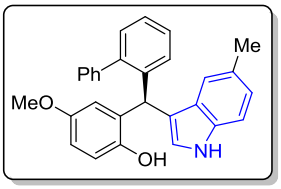
^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.22 (t, 3H, *J* = 8.0 Hz), 2.38 (s, 3H), 2.68 (q, 2H, *J* = 8.0 Hz), 3.65 (s, 3H), 4.57 (s, 1H), 5.96 (s, 1H), 6.47 (d, 1H, *J* = 4.0 Hz), 6.58 (brs, 1H), 6.72 (d, 1H, *J* = 8.0 Hz), 6.81 (d, 1H, *J* = 8.0 Hz), 7.03-7.12 (m, 4H), 7.22-7.29 (m, 3H), 7.94 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 15.1, 21.6, 25.6, 39.1, 55.6, 111.0, 112.1, 116.4, 116.9, 116.9, 119.2, 124.2, 124.6, 126.1, 127.0, 127.2, 128.7, 128.8, 129.0, 131.6, 135.3, 139.9, 142.4, 147.9, 153.8.

ESI-MS+ m/z Calcd. for $C_{25}H_{25}NO_2$ 394.1783 [M+ Na]; found 394.1775.

HPLC AD-H Column (80% hexane: 20% *i*-propanol, 1 mL/min, 234 nm) R_{t1} = 11.20 min and R_{t2} = 14.45 min.

Diarylindolylmethane 5c:



Yield 84%; yellow solid; **Mpt** 176-178°C (CH_2Cl_2/hexane)

[α]$_D^{25}$ = -11.2 (c = 0.15, $CHCl_3$)

Er = 92:8

IR (KBr) cm^{-1}750, 1662, 2924, 3419.

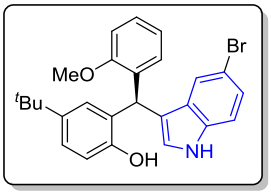
^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.33 (s, 3H), 3.66 (s, 3H), 4.29 (s, 1H), 5.71 (s, 1H), 6.54 (s, 1H), 6.68-6.75 (m, 3H), 6.88 (brs, 1H), 7.01 (d, 1H, *J* = 8.0 Hz), 7.23-7.33 (m, 10H), 7.95 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 21.5, 39.8, 55.6, 110.7, 111.9, 116.2, 116.8, 117.1, 119.3, 124.1, 124.3, 126.6, 126.8, 127.2, 127.7, 128.1, 128.8, 129.0, 129.4, 130.3, 131.6, 135.2, 139.6, 141.2, 141.9, 147.6, 153.5.

ESI-MS+ m/z Calcd. for $C_{29}H_{25}NO_2$ 442.1783 [M+ Na]; found 422.1725.

HPLC IA Column (85% hexane: 15% *i*-propanol, 1 mL/min, 232 nm) R_{t1} = 12.34min and R_{t2} = 20.81 min.

Diarylindolylmethane 6a:



Yield 84%; white solid; **Mpt** = 90-92 ºC (CH_2Cl_2/hexane)

$[\alpha]_D^{24}$ = -24.0 (c = 0.26, $CHCl_3$)

Er = 94:6

IR (KBr) cm^{-1}754, 1242, 1489, 2961, 3420.

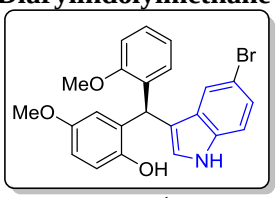
^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.18 (s, 9H), 3.86 (s, 3H), 5.43 (s, 1H), 6.09 (s, 1H), 6.78 (brs, 1H), 6.82 (d, 1H, *J* = 8.0 Hz), 6.90-6.98 (m, 2H), 7.07 (d, 1H, *J* = 4.0 Hz), , 7.12-7.19 (m, 2H), 7.23-7.29 (m, 4H), 8.04 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 31.7, 34.3, 36.0, 56.0, 111.1, 112.8, 112.9, 116.0, 117.2, 121.3, 122.9, 124.6, 125.3, 125.5, 126.9, 128.2, 128.4, 129.0, 129.8, 130.7, 135.7, 143.3, 151.7, 156.6.

ESI-MS+ m/z Calcd. for $C_{26}H_{26}BrNO_2$ 486.1045 [M+ Na]; found 486.1035

HPLC AD-H Column (90% hexane: 10% *i*-propanol, 1 mL/min, 220 nm) R_{t1} = 37.0 min and R_{t2} = 50.9 min.

Diarylindolylmethane 6b:



Yield 90%; yellow solid; **Mpt** = 102-104 ºC (CH_2Cl_2/hexane)

$[\alpha]_D^{24}$-30.3 (c = 0.20, $CHCl_3$)

Er = 95:5

IR (KBr) cm^{-1}754, 1242, 1489, 3419.

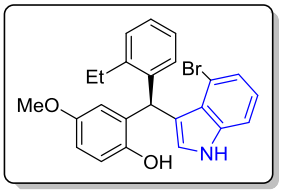
^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.67 (s, 3H), 3.86 (s, 3H), 5.11 (s, 1H), 6.11 (s, 1H), 6.58 (d, 1H, *J* = 4.0 Hz), 6.72 (dd, 1H, J_1 = 4.0 Hz, J_2 = 8.0 Hz), 6.78 (brs, 1H), 6.84 (d, 1H, *J* = 12.0 Hz), 6.91 (t, 1H, *J* = 8.0 Hz), 6.96 (d, 1H, *J* = 8.0 Hz), 7.11 (d, 1H, *J* = 4.0 Hz), 7.23-7.28 (m, 3H), 7.38 (brs, 1H), 8.05 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 35.3, 55.7, 55.9, 111.0, 112.1, 112.7, 112.9, 115.8, 116.8, 117.0, 121.1, 122.4, 125.3, 125.4, 128.1, 128.8, 129.7, 130.2, 13.8, 135.6, 147.8, 153.6, 156.5.

ESI-MS+ m/z Calcd. for $C_{23}H_{20}BrNO_3$ 460.0524[M+ Na]; found 460.0517.

HPLC AD-H Column (90% hexane: 10% *i*-propanol, 1 mL/min, 236 nm) R_{t1} = 77.0 min and R_{t2} = 86.2 min.

Diarylindolylmethane 7a:



Yield 87%; white solid; **Mpt** = 86-88 ºC (CH_2Cl_2/hexane)

[α]$_D^{24}$ = -20.0 (c = 0.2, $CHCl_3$)

Er = 96:4

IR (KBr) cm^{-1}741, 1041, 1183, 1503, 3418.

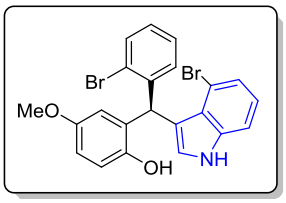
^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.20 (t, 3H, *J* = 8.0 Hz), 2.64-2.77 (m, 1H), 3.65 (s, 3H), 4.39 (s, 1H), 6.38 (d, 1H, *J* = 4.0 Hz), 6.60 (brs, 1H), 6.66 (s, 1H), 6.70 (dd, 2H, J_1 = 4.0 Hz, J_2 = 8.0 Hz), 6.79 (d, 1H, *J* = 8.0 Hz), 7.03 (t, 1H, *J* = 8.0 Hz), 7.05 (t, 1H, *J* = 8.0 Hz), 7.22-7.33 (m, 5H), 8.12 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 14.6, 25.6, 39.1, 55.7, 110.8, 111.5, 114.8, 116.7, 117.2, 118.5, 123.4, 124.6, 125.1, 126.0, 126.6, 127.1, 128.6, 128.8, 133.1, 128.3, 141.0, 142.5, 147.4, 153.8.

ESI-MS+ m/z Calcd. for $C_{24}H_{22}BrNO_2$ 458.0732 [M+ Na]; found 458.0725.

HPLC OD-H Column (80% hexane: 20% *i*-propanol, 1 mL/min, 262 nm) R_{t1} = 14.7 min and R_{t2} = 21.7 min.

Diarylindolylmethane 7b:



Yield 82%; brown solid; **Mpt** = 84-86 ºC (CH_2Cl_2/hexane)

[α]$_D^{24}$ = -24.1 (c = 0.18, $CHCl_3$)

Er = 92:8

IR (KBr) cm^{-1} 755, 1488, 1633, 3432.

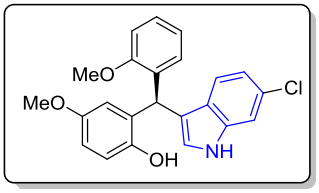
^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.86 (s, 3H), 5.53 (s, 1H), 6.08 (s, 1H), 6.77-6.79 (m, 2H), 6.92-6.99 (m, 2H), 7.09-7.10 (m, 2H), 7.25-7.36 (m, 4H), 7.36 (brs, 1H), 8.08 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 35.0, 55.8, 111.0, 112.7, 112.9, 113.0, 116.1, 118.2, 121.1, 122.2, 125.3, 125.4, 128.3, 128.5, 129.4, 129.6, 130.7, 131.7, 131.8, 135.5, 153.0, 156.2.

ESI-MS+ m/z Calcd. for $C_{22}H_{17}Br_2NO_2$ 507.9524 [M+ Na]; found 507.9518.

HPLC IA Column (85% hexane: 15% *i*-propanol, 1 mL/min, 236 nm) R_{t1} = 13.9 min and R_{t2} = 16.7 min.

Diarylindolylmethane 8a:



Yield 84%; yellow solid; **Mpt** = 142-144 ºC (CH_2Cl_2/hexane)

[α]$_D^{24}$ = -25.0 (c = 0.16, $CHCl_3$)

Er = 94:6

IR (KBr) cm^{-1} 804, 1508, 3420.

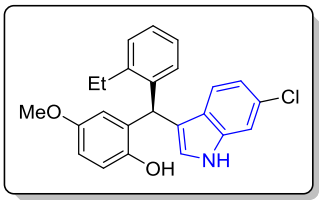
^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.67 (s, 3H), 3.81 (s, 3H), 4.54 (s, 1H), 5.73 (s, 1H), 6.53 (d, 1H, *J* = 4.0 Hz), 6.68 (brs, 1H), 6.73 (dd, 1H, J_1 = 4.0 Hz, J_2 = 8.0 Hz), 6.81 (d, 1H, *J* = 8.0 Hz), 6.87 (d, 2H, *J* = 8.0 Hz), 7.0 (d, 1H, *J* = 8.0 Hz), 7.16-7.20 (m, 2H), 7.28 (s, 1H), 7.36 (brs, 1H), 8.04 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 42.5, 55.4, 55.7, 111.2, 112.3, 114.2, 114.2, 116.3, 117.1, 118.4, 120.6, 120.9, 124.6, 125.5, 128.5, 130.0, 130.0,131.3, 134.0, 137.3, 147.7, 153.8, 158.5.

ESI-MS+ m/z Calcd. for $C_{23}H_{20}ClNO_3$ 416.1029 [M+ Na]; found 416.1023.

HPLC OD-H Column (80% hexane: 20% *i*-propanol, 1 mL/min, 278 nm) R_{t1} = 33.6 min and R_{t2} = 37.6 min.

Diarylindolylmethane 8b:



Yield 86%; yellow solid; **Mpt** = 74-76 ºC (CH_2Cl_2/hexane)

[α]$_D^{24}$ = -9.09 (c = 0.11, $CHCl_3$)

Er = 97:3

IR (KBr) cm^{-1} 807, 1503, 3421.

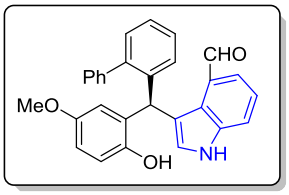
^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.21 (t, 3H, *J* = 8.0 Hz), 2.66-2.72 (m, 2H), 3.64 (s, 3H), 4.46 (s, 1H), 5.99 (s, 1H), 6.46 (d, 1H, *J* = 4.0 Hz), 6.59 (brs, 1H), 6.72 (dd, 1H, *J* = 4.0 Hz, J_2 = 8.0 Hz), 6.99-7.01 (m, 2H), 7.10 (t, 1H, *J* = 8.0 Hz), 7.18-7.28 (m, 4H), 7.38 (s, 1H), 8.00 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 15.2, 25.6, 38.8, 55.7, 111.3, 112.1, 116.5, 116.9, 118.1, 120.6, 120.7, 125.0, 125.6, 126.1, 127.2, 128.5, 128.7, 128.9, 131.2, 137.4, 139.6, 142.4, 147.7, 153.9.

ESI-MS+ m/z Calcd. for $C_{24}H_{22}ClNO_2$ 414.1237 [M+ Na]; found 414.1231.

HPLC OD-H Column (80% hexane: 20% *i*-propanol, 1 mL/min, 232 nm) R_{t1} = 20.8 min and R_{t2} = 26.0 min.

Diarylindolylmethane 9a:



Yield 82%; yellow solid; **Mpt** = 76-78ºC (CH_2Cl_2/hexane)

[α]$_D^{24}$ = -30.3 (c = 0.26, $CHCl_3$)

Er = 97:3

IR (KBr) cm^{-1}750, 1662, 2924, 3419.

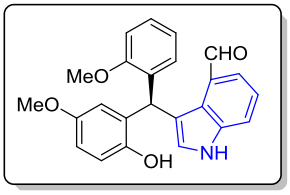
^{1}H NMR ($CDCl_3$, 300 MHz) δ 3.61 (s, 3H), 4.48 (s, 1H), 6.17 (s, 1H), 6.43 (d, 1H, *J* = 3.0 Hz), 6.61-6.71 (m, 2H), 6.85 (d, 1H, *J* = 3.0 Hz), 7.13-7.30 (m, 10H), 7.58-7.66 (m, 2H), 8.35 (brs, 1H), 9.92 (s, 1H).

^{13}C NMR ($CDCl_3$, 75 MHz) δ 41.9, 55.6, 111.8, 116.6, 117.0, 117.5, 118.9, 121.8, 123.6, 126.9, 127.2, 127.6, 128.0, 128.0, 128.6, 128.9, 129.0, 130.1, 130.6, 132.3, 138.4, 140.0, 141.2, 142.5, 147.3, 153.7.

ESI-MS+ m/z Calcd. for $C_{29}H_{23}NO_3$ 456.1576 [M+ Na]; found 456.1570.

HPLC IA Column (80% hexane: 20% *i*-propanol, 1 mL/min, 232 nm) R_{t1} = 9.6 min and R_{t2} = 13.3 min.

Diarylindolylmethane 9b:



Yield 89%; white solid; **Mpt** = 174-176 ºC (CH_2Cl_2/hexane)

[α]$_D^{24}$ = -28.8 (c = 0.25, $CHCl_3$)

Er = 93:7

IR (KBr) cm^{-1}751, 1044, 1242, 1489, 1662, 3409.

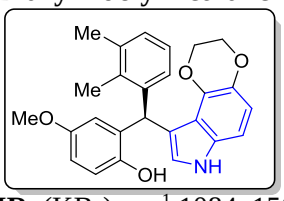
^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.63 (s, 3H), 3.80 (s, 3H), 5.32 (brs, 1H), 6.44 (d, 1H, *J* = 4.0 Hz), 6.57 (s, 1H), 6.67 (dd, 1H, J_1 = 4.0 Hz, J_2 = 8.0 Hz), 6.78-6.86 (m, 3H), 6.94 (d, 2H, *J* = 8.0 Hz), 7.22-7.28 (m, 2H), 7.59 (d, 1H, *J* = 8.0 Hz), 7.72 (d, 1H, J = 8.0 Hz), 8.48 (brs, 1H), 10.16 (s, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 37.7, 55.6, 56.0, 111.1, 111.8, 116.1, 116.9, 117.6, 118.0, 120.7, 121.8, 123.4, 125.9, 128.0, 128.3, 129.6, 130.1, 130.5, 131.5, 138.5, 147.8, 153.5, 156.9, 192.5.

ESI-MS+ m/z Calcd. for $C_{24}H_{21}NO_4$ 410.1368 [M+ Na]; found 410.1360.

HPLC AD-H Column (70% hexane: 30% *i*-propanol, 1 mL/min, 333 nm) R_{t1} = 10.6 min and R_{t2} = 21.3 min.

Diarylindolylmethane 10a:



Yield 90%; yellow solid; **Mpt** = 116-118 ºC (CH_2Cl_2/hexane)

[α]$_D^{24}$ = -18.8 (c = 0.2, $CHCl_3$)

Er = 96:4

IR (KBr) cm^{-1} 1084, 1503, 3419.

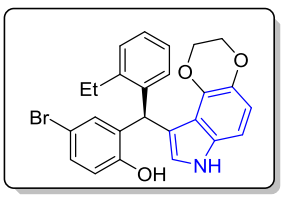
^{1}H NMR ($CDCl_3$, 400 MHz) δ 2.21 (s, 3H), 2.33 (s, 3H), 3.65 (s, 3H), 4.05-4.09 (m, 2H), 4.19 (brs, 2H), 4.66 (s, 1H), 6.28 (s, 1H), 6.41 (d, 1H, *J* = 4.0 H), 6.50 (brs, 1H), 6.70 (dd, 1H, J_1 = 4.0 Hz, J_2 = 8.0 Hz), 6.76 (d, 1H, *J* = 8.0 Hz), 6.80-6.84 (m, 2H), 6.98 (t, 1H, *J* = 8.0 Hz), 7.05 (d, 1H, *J* = 8.0 Hz), 7.28 (s, 1H), 7.87 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 15.2, 21.1, 40.8, 55.7, 64.4, 64.5, 104.0, 111.7, 113.8, 116.5, 116.5, 116.8, 117.0, 117.5, 124.3, 125.4, 126.5, 128.4, 132.8, 133.4, 135.2, 136.2, 136.9, 137.3, 147.9, 153.7.

ESI-MS+ m/z Calcd. for $C_{26}H_{25}NO_4$ 438.1681 [M+ Na]; found 438.1675.

HPLC AS-H Column (85% hexane: 15% *i*-propanol, 1 mL/min, 232 nm) R_{t1} = 21.5 min and R_{t2} = 27.2 min.

Diarylindolylmethane 10b:



Yield 89%; pale yellow solid; **Mpt** = 162-164 ºC (CH_2Cl_2/hexane)

[α]$_D^{24}$ = -14.4 (c = 0.22, $CHCl_3$)

Er = 94:6

IR (KBr) cm^{-1} 962, 1503, 3423.

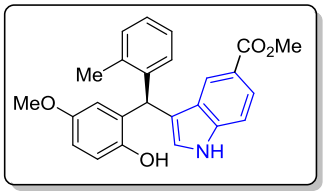
^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.21 (t, 3H, *J* = 8.0 Hz), 2.66 (q, 2H, *J* = 8.0 Hz), 4.01-4.10 (m, 2H), 4.18-4.20 (m, 2H), 5.03 (s, 1H), 6.27 (s, 1H), 6.50 (brs, 1H), 6.74-6.86 (m, 3H), 6.93 (brs, 1H), 6.97 (d, 1H, *J* = 4.0 Hz), 7.11 (t, 1H, *J* = 4.0 Hz), 7.21-7.28 (m, 3H), 7.90 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 15.0, 25.7, 39.8, 64.4, 64.4, 104.1, 113.1, 114.1, 116.4, 117.3, 118.2, 124.4, 126.1, 127.1, 128.7, 128.9, 130.5, 132.6, 133.4, 133.8, 136.3, 137.2, 140.2, 142.4, 153.1.

ESI-MS+ m/z Calcd. for $C_{25}H_{22}BrNO_3$ 486.0681 [M+ Na]; found 486.0671.

HPLC IA Column (90% hexane: 10% *i*-propanol, 1 mL/min, 232 nm) R_{t1} = 21.7 min and R_{t2} = 23.4 min.

Diarylindolylmethane 11a:



Yield 82%; white solid; **Mpt** = 112-114°C (CH_2Cl_2/hexane)

[α]$_D^{24}$ = +5.5 (c = 0.18, $CHCl_3$)

Er = 98:2

IR (KBr) cm^{-1}750, 1245, 1434, 1690, 3411

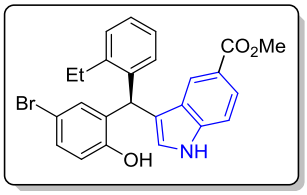
^{1}H NMR ($CDCl_3$, 400 MHz) δ2.32 (s, 3H), 3.64 (s, 3H), 3.88 (s, 3H), 4.57 (brs, 1H), 6.01 (s, 1H), 6.44 (s, 1H), 6.66-6.72 (m, 2H), 6.79 (d, 1H, *J* = 8.0 Hz), 6.96 (d, 1H, *J* = 8.0 Hz), 7.10 (t, 1H, *J* = 4.0 Hz), 7.13-7.24 (m, 2H), 7.37 (d, 1H, *J* = 8.0 Hz), 7.91 (d, 1H, *J* = 8.0 Hz), 8.11 (s, 1H), 8.28 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 19.9, 39.0, 51.9, 55.5, 110.9, 111.8, 116.3, 116.7, 118.9, 121.8, 122.4, 123.8, 125.6, 126.1, 126.6, 126.8, 128.1, 130.6, 130.9, 136.6, 139.4, 140.3, 147.5, 153.7, 168.1.

ESI-MS+ m/z Calcd. for $C_{25}H_{23}NO_4$ 424.1525 [M+ Na]; found 424.1519.

HPLC IA Column (90% hexane: 10% *i*-propanol, 1 mL/min, 232 nm) R_{t1} = 20.49 min and R_{t2} = 26.57 min.

Diarylindolylmethane 11b:



Yield 81%; yellow solid; **Mpt** = 114-116 °C (CH_2Cl_2/hexane)

[α]$_D^{24}$ = +7.19 (c = 0.1, $CHCl_3$)

Er = 93:7

IR (KBr) cm-^{1}1751, 1107, 1248, 1437, 1617, 1688, 3422.

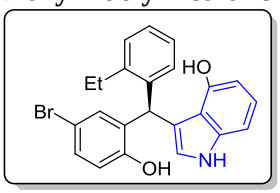
^{1}H NMR ($CDCl_3$, 400 MHz)δ1.19 (t, 3H, *J* = 8.0 Hz), 2.68 (m, 2H, *J* = 8.0 Hz), 3.88 (s, 3H), 5.56 (brs, 1H), 6.13 (s, 1H), 6.62 (s, 1H), 6.71 (d, 1H, *J* = 8.0 Hz), 6.98 (brs, 2H), 7.12 (t, 1H, *J* = 8.0 Hz), 7.22-7.28 (m, 3H), 7.35 (d, 1H, *J* = 12.0 Hz), 7.91 (d, 1H, *J* = 8.0 Hz), 8.11 (s, 1H), 8.39 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz)δ 15.1, 25.7, 38.2, 52.2, 111.3, 113.1, 118.0, 119.3, 121.9, 122.5, 124.1, 126.1, 126.2, 126.6, 127.4, 128.5, 129.1, 130.9, 132.6, 132.7, 139.6, 139.7, 142.5, 153.0, 168.7.

ESI-MS+ m/z Calcd. for $C_{25}H_{22}BrNO_3$ 486.0681 [M+ Na]; found 486.0675.

HPLC IA Column (80% hexane: 20% *i*-propanol, 1 mL/min, 232 nm) R_{t1} = 10.11 min and R_{t2} = 28.73 min.

Diarylindolylmethane 12:



Yield 70%; white solid; **Mpt** = 106-108°C (CH_2Cl_2/hexane)

$[\alpha]_D^{24}$ = -5.5 (c = 0.14, $CHCl_3$)

Er = 95:5

IR (KBr) cm^{-1}734, 1033, 1622, 3421.

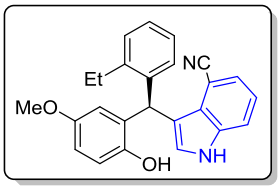
^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.20 (t, 3H, *J* = 4.0 Hz), 2.64-2.71 (m, 2H), 4.65 (s, 1H), 4.99 (s, 1H), 6.32 (s, 1H), 6.41 (d, 1H, *J* = 8.0 Hz), 6.47 (brs, 1H), 6.77 (d, 1H, *J* = 8.0 Hz), 6.96-7.06 (m, 4H), 7.15 (t, 1H, *J* = 4.0 Hz), 7.27-7.32 (m, 3H), 8.01 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 14.7, 25.4, 39.3, 104.5, 105.5, 113.2, 116.1, 116.5, 118.0, 123.5, 123.6, 126.2, 127.5, 128.8, 129.0, 130.8, 132.5, 132.8, 139.2, 139.5, 142.4, 150.1, 152.7.

ESI-MS+ m/z Calcd. for $C_{23}H_{20}BrNO_2$ 444.0575 [M+ Na]; found 444.0572

HPLC IA Column (90% hexane: 10% *i*-propanol, 1 mL/min, 232 nm) R_{t1} = 32.50 min and R_{t2} = 39.02 min.

Diarylindolylmethane 13a:



Yield 88%; white solid; **Mpt** = 124-126°C (CH_2Cl_2/hexane)

$[\alpha]_D^{24}$ = -56.2 (c = 0.15, $CHCl_3$)

Er = 96:4

IR (KBr) cm^{-1}749, 1044, 1201, 1505, 1614, 2218, 3408.

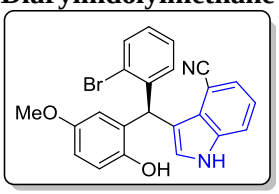
^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.14 (t, 3H, *J* = 8.0 Hz), 2.71 (dq, 2H, J_1 = 4.0 Hz, J_2 = 8.0 Hz), 3.62 (s, 3H), 6.40 (d, 1H, *J* = 4.0 Hz), 6.53 (s, 1H), 6.62-6.65 (m, 2H), 6.70 (d, 1H, *J* = 8.0 Hz), 6.91 (d, 1H, *J* = 8.0 Hz), 7.05 (t, 1H, *J* = 8.0 Hz), 7.13-7.26 (m, 3H), 7.35 (d, 1H, *J* = 8.0 Hz), 7.49 (d, 1H, *J* = 8.0 Hz), 8.64 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 14.4, 25.5, 37.9, 55.6, 102.0, 111.3, 116.5, 116.8, 117.1, 118.2, 118.6, 121.7, 125.7, 126.1, 126.6, 126.9, 128.0, 128.1, 128.6, 132.5, 137.1, 140.6, 142.5, 147.7, 153.4.

ESI-MS+ m/z Calcd. for $C_{25}H_{22}N_2O_2$ 405.1579 [M+ Na]; found 405.1574.

HPLC IA Column (90% hexane: 10% *i*-propanol, 1 mL/min, 232 nm) R_{t1} = 51.9 min and R_{t2} = 68.8 min.

Diarylindolylmethane 13b:



Yield 86%; white solid; **Mpt** = 218-220°C (CH_2Cl_2/hexane)

$[\alpha]_D^{24}$ = -66.67 (c = 0.12, $CHCl_3$)

Er = 96:4

IR (KBr) cm^{-1}746, 1105, 1241, 1489, 2218, 3419.

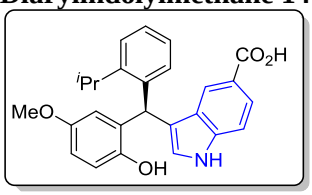
^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.76 (s, 3H), 6.59 (s, 1H), 6.70-6.73 (m, 1H), 6.79 (d, 1H, *J* = 4.0 Hz), 6.83-6.94 (m, 4H), 7.16-7.27 (m, 3H), 7.39 (d, 1H, *J* = 12.0 Hz), 7.57 (d, 1H, *J* = 8.0 Hz), 8.44 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 34.8, 56.1, 102.6, 111.4, 112.7, 116.6, 117.4, 118.5, 120.8, 122.1, 126.2, 127.0, 127.6, 128.4, 129.3, 130.6, 130.8, 132.0, 132.9, 137.3, 153.2, 157.0.

ESI-MS+ m/z Calcd. for $C_{23}H_{17}BrN_2O_2$ 455.0371 [M+ Na]; found 455.0367.

HPLC IA Column (80% hexane: 20% *i*-propanol, 1 mL/min, 244 nm) R_{t1} = 7.2 min and R_{t2} = 9.2 min.

Diarylindolylmethane 14:



Yield 83%; white solid; **Mpt** = 126-128ºC (CH_2Cl_2/hexane)

[α]$_D^{25}$ = -12.2 (c = 0.16, $CHCl_3$)

Er = 86:14

IR (KBr) cm^{-1}762, 1204, 1681, 3419.

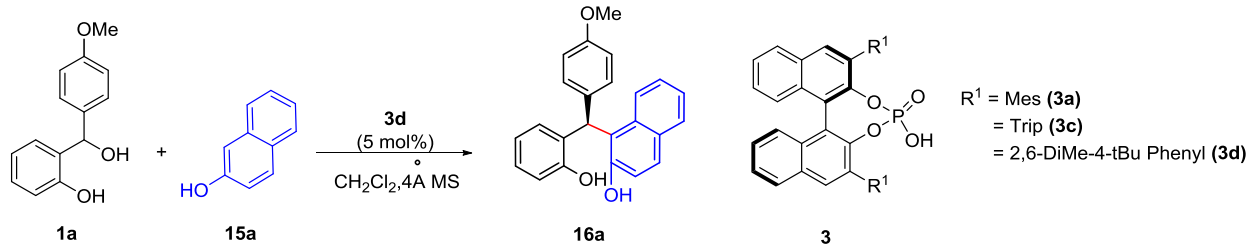
^{1}H NMR ($CDCl_3$, 400 MHz) δ 1.18 (t, 6H *J* = 8.0 Hz), 3.28 (q, 1H, *J* = 8.0 Hz), 3.64 (s, 3H), 6.20 (s, 1H), 6.46 (d, 1H, *J* = 4.0 H), 6.63 (s, 1H), 6.70 (dd, 1H, J_1 = 4.0 Hz, J_2 = 8.0 Hz), 6.78 (d, 1H, *J* = 8.0 Hz), 6.97 (d, 1H, *J* = 8.0 Hz), 7.06 (t, 1H, *J* = 4.0 Hz), 7.26-7.29 (m, 1H), 7.38 (t, 2H, *J* = 8.0 Hz), 7.95 (d, 1H, *J* = 8.0 Hz), 8.18 (s, 1H), 8.30 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 24.2, 28.8, 38.5, 55.8, 111.2, 112.2, 116.9, 116.9, 120.1, 121.0, 123.7, 124.5, 125.9, 126.0, 126.1, 126.8, 127.4, 128.6, 131.6, 138.9, 140.2, 147.4, 147.6, 153.9, 172.9.

ESI-MS+ m/z Calcd. for $C_{26}H_{25}NO_4$ 438.1681 [M+ Na]; found 438.1675.

HPLC IA Column (70% hexane: 30% *i*-propanol, 1 mL/min, 244 nm) R_{t1} = 6.0 min and R_{t2} = 10.3 min.

Optimization Studies for Brønsted Acid-Catalyzed Conjugate Addition of *β*-Naphthol (15a) to *Ortho*-Hydroxy Benzhydrol 1a[a]



1a **15a** **16a** **3**

Entry	Cat	Solvent	Yield [%][b]	Er[c]
1	**3a**	CH_2Cl_2	90	94:06
2	**3c**	CH_2Cl_2	92	95:05
3	**3d**	$CHCl_3$	94	96:04
4[d]	**3d**	$CHCl_3$	93	92:08
5[e]	**3d**	$CHCl_3$	92	95:05
6	**3d**	$PhCH_3$	90	98:02
7	**3d**	CCl_4	97	97:03
8	**3d**	DCE	90	98:02
9	**3d**	**CH_2Cl_2**	**95**	**99:01**

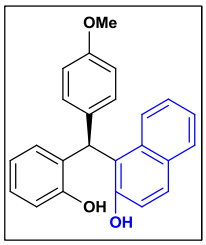
[a] All reactions were carried out with 0.23 mmol (1 equiv.) of **1a**, 0.27 mmol (1.2 equiv.) of **15a** and 5 mol% of catalyst **3a, 3c** and **3d** respectively in 2 mL of CH_2Cl_2 at rt using catalytic amount of 4Å MS. [b] Isolated yield of the product. [c] Er determined through chiral HPLC-analysis. [d] Reaction was carried out without using 4Å MS. [e] Reaction was carried out at 0 °C.

General Procedure for the Brønsted Acid-Catalyzed Conjugate Addition of Naphthols 15a-g and 16 to *Ortho*-Hydroxybenzhydrols 1.

Ortho-Hydroxybenzhydrol **1** (0.20 mmol), naphthol **15/16** (0.24 mmol), and catalyst **3d** (7 mg, 0.01 mmol) and 10 mg of 4 Å MS were dissolved in 2 mL of dry DCM under argon and the reaction mixture was stirred at rt for the indicated time. The progress of the reaction mixture was monitored by TLC. On disappearance of the starting material the reaction mixture was directly passed through silica gel column with ethyl acetate/hexanes to isolate the pure desired product which was further recrystallized from CH_2Cl_2/hexane.

Spectral Data of Substituted Triarylmethanes 17 and 18

Triarylmethane 17a:



Yield 95%; yellowish brown solid; **Mpt** = 86-88°C (CH_2Cl_2/hexane)

[α]$_D^{24}$ = -23.4 (c = 0.9, $CHCl_3$)

Er = 99:1

IR (KBr) cm^{-1}752, 1031, 1246, 1508, 1703, 3474.

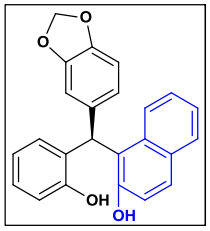
^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.99 (d, *J* = 8.7 Hz, 1H), 7.76 (dd, *J* = 18.0, 8.5 Hz, 2H), 7.42 (ddd, *J* = 8.6, 6.8, 1.6 Hz, 1H), 7.32 (t, *J* = 7.4 Hz, 1H), 7.22 – 7.12 (m, 3H), 7.06 (t, *J* = 8.5 Hz, 2H), 6.87 (dd, *J* = 8.3, 2.6 Hz, 4H), 6.50 (s, 1H), 3.78 (s, 3H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 158.9, 153.9, 153.3, 133.3, 132.4, 130.1, 130.0, 129.9, 129.8, 129.8, 128.9, 128.8, 127.9, 127.1, 123.5, 123.0, 121.8, 119.8, 118.7, 116.4, 114.9, 55.4, 42.4.

ESI-MS+ m/z Calcd. for $C_{24}H_{20}NaO_3$ 379.13101 [M+ Na]; found 379.13029

HPLC AS-H Column (95% hexane: 05% *i*-propanol, 1 mL/min, 230 nm) R_{t1} = 47.9 min and R_{t2} = 56.2 min.

Triarylmethane 17b:



Yield 95%; brown solid; **Mpt** = 85-88°C (CH_2Cl_2/hexane)

[α]$_D^{24}$ = -34.0 (c = 1, $CHCl_3$)

Er = 97:3

IR (KBr) cm^{-1}753, 813, 1039, 1244, 1501, 1702, 3465.

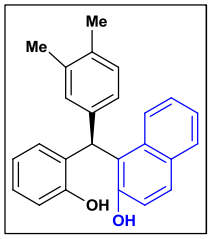
^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.97 (d, *J* = 8.6 Hz, 1H), 7.76 (dd, *J* = 16.5, 8.5 Hz, 2H), 7.42 (ddd, *J* = 8.6, 6.9, 1.5 Hz, 1H), 7.36 – 7.29 (m, 1H), 7.19 (td, *J* = 7.6, 1.7 Hz, 1H), 7.07 (dd, *J* = 8.2, 5.8 Hz, 2H), 6.88 (dd, *J* = 11.4, 7.8 Hz, 2H), 6.79 – 6.72 (m, 2H), 6.67 (dd, *J* = 8.0, 2.0 Hz, 1H), 6.46 (s, 1H), 6.00 – 5.88 (m, 2H), 5.56 (brs, 1H), 5.01 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 153.8, 153.4, 148.8, 147.1, 134.6, 133.3, 130.1, 130.0, 129.8, 129.0, 128.9, 127.7, 127.1, 123.5, 122.8, 121.8, 121.5, 119.8, 118.5, 116.4, 109.5, 108.9, 101.4, 42.8.

ESI-MS+ m/z Calcd. for $C_{24}H_{18}NaO_4$ 393.11028 [M+ Na]; found 393.10949

HPLC IA Column (90% hexane: 10% *i*-propanol, 1 mL/min, 230 nm) R_{t1} = 27.8 min and R_{t2} = 33.6 min.

Triarylmethane 17c:



Yield 95%; reddish brown solid; **Mpt** = 73-76°C (CH_2Cl_2/hexane)

[α]$_D^{24}$ = - 44.0 (c = 1.0, $CHCl_3$)

Er = 96:4

IR (KBr) cm^{-1}785, 1041, 1202, 1503, 1620, 2937, 3466.

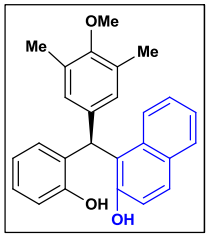
^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.98 (d, *J* = 8.7 Hz, 1H), 7.86 – 7.68 (m, 2H), 7.42 (ddd, *J* = 8.6, 6.8, 1.6 Hz, 1H), 7.32 (ddd, *J* = 8.0, 6.8, 1.1 Hz, 1H), 7.13 – 7.00 (m, 3H), 6.95 (dd, *J* = 7.7, 2.1 Hz, 1H), 6.83 (d, *J* = 8.6 Hz, 1H), 6.72 (dd, *J* = 8.7, 3.0 Hz, 1H), 6.61 (d, *J* = 3.0 Hz, 1H), 6.47 (s, 1H), 3.63 (s, 3H), 2.24 (s, 3H), 2.19 (s, 3H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 154.4, 153.5, 147.6, 138.1, 137.8, 136.2, 133.4, 130.8, 130.0, 129.9, 129.8, 129.1, 128.9, 127.1, 125.8, 123.4, 122.9, 120.0, 118.6, 117.1, 116.2, 113.0, 55.7, 43.0, 20.1, 19.6.

ESI-MS+ m/z Calcd. for $C_{26}H_{24}NaO_3$ 407.16231 [M+ Na]; found 407.16169

HPLC OD-H Column (90% hexane: 10% *i*-propanol, 1 mL/min, 240 nm) R_{t1} = 8.7 min and R_{t2} = 11.3 min.

Triarylmethane 17d:



Yield 90%; white solid; **Mpt** = 86-88°C (CH_2Cl_2/hexane)

[α]$_D^{24}$ = - 56.0 (c = 1.2, $CHCl_3$)

Er = 96:4

IR (KBr) cm^{-1}752, 1004, 1219, 1455, 1621, 2940, 3466.

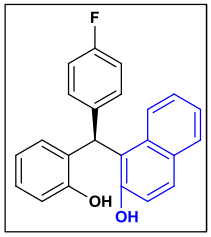
^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.99 (d, *J* = 8.6 Hz, 1H), 7.77 (dd, *J* = 17.2, 8.5 Hz, 2H), 7.43 (ddd, *J* = 8.6, 6.8, 1.5 Hz, 1H), 7.33 (dd, *J* = 8.2, 6.7 Hz, 1H), 7.19 (td, *J* = 7.7, 1.7 Hz, 1H), 7.06 (dd, *J* = 16.0, 8.0 Hz, 2H), 6.87 (t, *J* = 4.0 Hz, 4H), 6.46 (s, 1H), 5.55 (brs, 1H), 4.98 (brs, 1H), 3.71 (s, 3H), 2.21 (s, 6H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 156.3, 153.8, 153.3, 135.9, 133.4, 132.0, 130.2, 129.8, 129.8, 129.0, 128.8, 128.7, 127.8, 127.0, 123.2, 123.0, 121.6, 119.8, 119.1, 116.2, 59.8, 42.5, 16.4.

ESI-MS+ m/z Calcd. for $C_{26}H_{24}NaO_3$ 407.16231 [M+ Na]; found 407.16197

HPLC IA Column (90% hexane: 10% *i*-propanol, 1 mL/min, 230 nm) R_{t1} = 13.2 min and R_{t2} = 14.6 min.

Triarylmethane 17e:



Yield 90%; reddish brown solid; **Mpt** = 70-73°C (CH_2Cl_2/hexane)

[α]$_D^{24}$ = 26.7 (c = 0.3, $CHCl_3$)

Er = 87:13

IR (KBr) cm^{-1}753, 820, 1224, 1506, 1601, 1621, 1703, 3475.

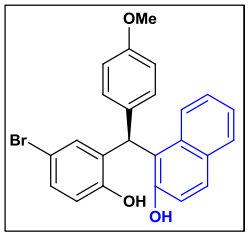
^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.02 (d, *J* = 8.6 Hz, 1H), 7.80 (dd, *J* = 20.8, 8.5 Hz, 2H), 7.45 (dd, *J* = 8.6, 6.8 Hz, 1H), 7.37 (t, *J* = 7.4 Hz, 1H), 7.23 (dd, *J* = 8.5, 5.2 Hz, 3H), 7.10 (dd, *J* = 8.6, 3.8 Hz, 2H), 7.04 (t, *J* = 8.4 Hz, 2H), 6.93 (t, *J* = 7.6 Hz, 1H), 6.88 (d, *J* = 8.0 Hz, 1H), 6.56 (s, 1H), 5.51 (brs, 2H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 163.2, 160.8, 153.9, 153.0, 136.5, 136.5, 133.2, 130.5, 130.4, 130.2, 130.0, 129.9, 129.0, 129.0, 127.8, 127.1, 123.6, 123.0, 121.8, 119.6, 118.4, 116.4, 116.2, 116.0, 42.5.

ESI-MS+ m/z Calcd. for $C_{23}H_{17}FnaO_2$ 367.11103 [M+ Na]; found 367.11014.

HPLC IA Column (80% hexane: 20% *i*-propanol, 1 mL/min, 240 nm) R_{t1} = 8.3 min and R_{t2} = 9.0 min.

Triarylmethane 17f:



Yield 94%; yellowish brown solid; **Mpt** = 84-87°C (CH_2Cl_2/hexane)

[α]$_D^{24}$ = -34.9 (c = 10.1, $CHCl_3$)

Er = 96:4

IR (KBr) cm^{-1}746, 812, 1249, 1509, 1700, 3464.

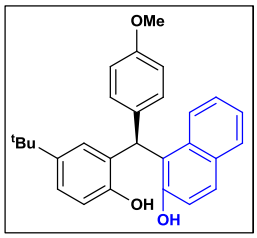
^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.93 (d, *J* = 8.6 Hz, 1H), 7.78 (dd, *J* = 13.2, 8.3 Hz, 2H), 7.43 (ddt, *J* = 8.5, 6.8, 1.3 Hz, 1H), 7.39 – 7.31 (m, 1H), 7.31 – 7.25 (m, 1H), 7.21 – 6.97 (m, 4H), 6.93 – 6.82 (m, 2H), 6.75 (dd, *J* = 8.6, 1.0 Hz, 1H), 6.47 (s, 1H), 5.20 (brs, 2H), 3.79 (s, 3H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 159.1, 153.1, 153.0, 133.3, 132.8, 131.7, 131.6, 130.4, 130.3, 129.9, 129.7, 129.0, 127.2, 123.6, 122.8, 119.8, 118.4, 118.1, 115.0, 113.8, 55.4, 42.2.

ESI-MS+ m/z Calcd. for $C_{24}H_{19}BrNaO_3$ 457.04153 [M+ Na]; found 457.04080

HPLC IA Column (90% hexane: 10% *i*-propanol, 1 mL/min, 236 nm) R_{t1} = 23.9 min and R_{t2} = 31.1 min.

Triarylmethane 17g:



Yield 94%; yellowish brown solid; **Mpt** = 80-82°C (CH_2Cl_2/hexane)

[α]$_D^{24}$ = 20 (c (= 1, $CHCl_3$)

Er = 96:4

IR (KBr) cm^{-1}745, 816, 1032, 1248, 1509, 1622, 2960, 3465.

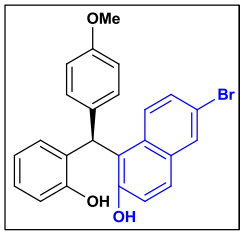
^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.03 (d, *J* = 8.6 Hz, 1H), 7.82 – 7.67 (m, 2H), 7.42 (ddd, *J* = 8.6, 7.0, 1.5 Hz, 1H), 7.35 – 7.29 (m, 1H), 7.22 – 7.12 (m, 3H), 7.12 – 7.05 (m, 2H), 6.87 (d, *J* = 8.5 Hz, 2H), 6.79 (d, *J* = 8.4 Hz, 1H), 6.46 (s, 1H), 3.79 (s, 3H), 1.16 (s, 9H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 158.8, 153.3, 151.7, 144.5, 133.3, 132.5, 130.0, 129.9, 129.9, 128.9, 127.4, 127.0, 126.9, 125.6, 123.4, 123.1, 119.8, 118.5, 116.0, 114.7, 55.4, 42.8, 34.3, 31.5.

ESI-MS+ m/z Calcd. for $C_{28}H_{28}NaO_3$ 435.19361 [M+ Na]; found 435.19295

HPLC IA Column (90% hexane: 10% *i*-propanol, 1 mL/min, 242 nm) R_{t1} = 15.0 min and R_{t2} = 23.1 min.

Triarylmethane 17h:



Yield 93%; reddish brown solid; **Mpt** = 108-110°C (CH_2Cl_2/hexane)

[α]$_D^{24}$ = -37.7 (c = 1.06, $CHCl_3$)

Er = 97:3

IR (KBr) cm^{-1}758, 1030, 1248, 1508, 1608, 3467.

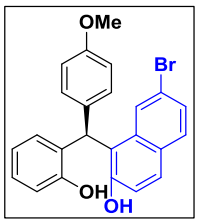
^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.91 (d, *J* = 2.1 Hz, 1H), 7.84 (d, *J* = 9.2 Hz, 1H), 7.63 (d, *J* = 8.9 Hz, 1H), 7.45 (dd, *J* = 9.2, 2.1 Hz, 1H), 7.23 – 7.16 (m, 1H), 7.16 – 6.99 (m, 4H), 6.93 – 6.83 (m, 4H), 6.45 (s, 1H), 5.53 (brs, 1H), 4.97 (brs, 1H), 3.79 (s, 3H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 159.1, 153.6, 153.6, 132.1, 132.0, 131.1, 130.7, 130.2, 130.0, 129.8, 129.0, 128.9, 127.7, 125.0, 121.9, 121.0, 119.3, 117.2, 116.3, 115.0, 55.4, 42.3.

ESI-MS+ m/z Calcd. for $C_{24}H_{19}BrNaO_3$ 457.04153 [M+ Na]; found 457.04058

HPLC IA Column (80% hexane: 20% *i*-propanol, 1 mL/min, 240 nm) R_{t1} = 10.6 min and R_{t2} = 12.5 min.

Triarylmethane 17i:



Yield 94%; reddish brown solid; **Mpt** = 78-81°C (CH_2Cl_2/hexane)

[α]$_D^{24}$ = -43.75 (c = 0.96, $CHCl_3$)

Er = 97:3

IR (KBr) cm^{-1}756, 835, 1031, 1248, 1455, 1508, 1615, 3463.

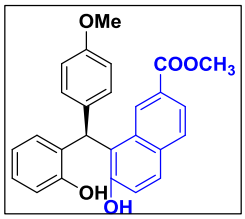
^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.17 (s, 1H), 7.65 (dd, *J* = 19.6, 8.7 Hz, 2H), 7.39 (dt, *J* = 8.6, 1.6 Hz, 1H), 7.20 (td, *J* = 7.6, 1.7 Hz, 1H), 7.13 (d, *J* = 8.4 Hz, 2H), 7.06 (dd, *J* = 8.5, 6.1 Hz, 2H), 6.88 (q, *J* = 8.2 Hz, 4H), 6.42 (s, 1H), 5.69 (brs, 1H), 5.23 (brs, 1H), 3.79 (s, 3H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 159.0, 154.1, 153.7, 134.8, 132.1, 130.4, 130.0, 129.8, 129.7, 129.0, 128.2, 127.5, 126.8, 125.5, 121.8, 121.7, 120.3, 118.6, 116.3, 114.9, 55.4, 42.2.

ESI-MS+ m/z Calcd. for $C_{24}H_{19}BrNaO_3$ 457.04153 [M+ Na]; found 457.04087

HPLC IA Column (80% hexane: 20% *i*-propanol, 1 mL/min, 230 nm) R_{t1} = 8.9 min and R_{t2} = 10.1 min.

Triarylmethane 17j:



Yield 90%; white solid; **Mpt** = 82-85°C (CH_2Cl_2/hexane)

[α]$_D^{24}$ = -37.2 (c = 0.94, $CHCl_3$)

Er = 99:1

IR (KBr) cm^{-1}754, 1035, 1205, 1292, 1509, 1620, 1694, 3454.

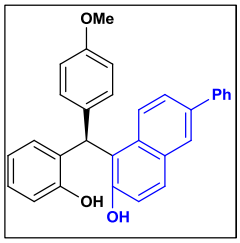
^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.52 (s, 1H), 8.07 – 7.92 (m, 2H), 7.83 (d, J = 8.9 Hz, 1H), 7.23 – 7.09 (m, 4H), 7.03 (d, J = 7.6 Hz, 1H), 6.88 (dt, J = 9.3, 2.7 Hz, 4H), 6.52 (s, 1H), 5.75 (brs, 1H), 5.14 (brs, 1H), 3.94 (s, 3H), 3.79 (s, 3H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 167.5, 159.1, 155.5, 153.7, 136.0, 132.1, 131.9, 131.3, 130.0, 129.8, 129.0, 128.8, 127.6, 126.5, 124.9, 123.3, 121.8, 120.8, 119.4, 116.3, 115.0, 55.4, 52.3, 42.3.

ESI-MS+ m/z Calcd. for $C_{26}H_{22}NaO_5$ 437.13649 [M+ Na]; found 437.13617

HPLC IA Column (80% hexane: 20% *i*-propanol, 1 mL/min, 240 nm) R_{t1} = 13.0 min and R_{t2} = 16.0 min.

Triarylmethane 17k:



Yield 96%; reddish brown solid; **Mpt** = 95-97°C (CH_2Cl_2/hexane)

[α]$_D^{24}$ = -21.6 (c = 1.02, $CHCl_3$)

Er = 97:3

IR (KBr) cm^{-1} 756, 1030, 1248, 1508, 1606, 3464.

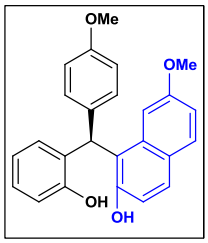
^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.07 (d, *J* = 8.9 Hz, 1H), 8.01 – 7.96 (m, 1H), 7.79 (d, *J* = 8.9 Hz, 1H), 7.67 (dd, *J* = 8.2, 4.9 Hz, 3H), 7.46 (t, *J* = 7.6 Hz, 2H), 7.38 – 7.32 (m, 1H), 7.19 (dd, *J* = 11.4, 7.9 Hz, 3H), 7.10 (t, *J* = 7.6 Hz, 2H), 6.89 (d, *J* = 8.2 Hz, 4H), 6.54 (s, 1H), 5.62 (brs, 1H), 5.22 (brs, 1H), 3.79 (s, 3H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 159.0, 153.9, 153.5, 141.0, 136.2, 132.5, 132.4, 130.3, 130.1, 130.1, 129.9, 129.0, 128.9, 128.0, 127.3, 127.3, 126.7, 126.6, 123.6, 121.8, 120.3, 118.8, 116.4, 114.9, 55.4, 42.5.

ESI-MS+ m/z Calcd. for $C_{30}H_{24}NaO_3$ 455.16231 [M+ Na]; found 455.16157

HPLC IA Column (80% hexane: 20% *i*-propanol, 1 mL/min, 240 nm) R_{t1} = 12.4 min and R_{t2} = 14.5 min.

Triarylmethane 17l:



Yield 92%; reddish brown solid; **Mpt** = 84-86°C (CH_2Cl_2/hexane)

[α]$_D^{24}$ = -18.2 (c = 1, $CHCl_3$)

Er = 94:6

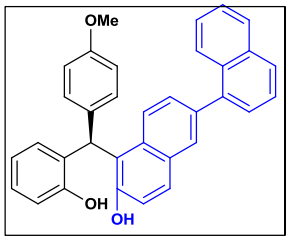
IR (KBr) cm^{-1} 756, 833, 1031, 1248, 1509, 1624, 3464.

^{1}H NMR ($CDCl_3$, 400 MHz) 7.68 (dd, *J* = 8.9, 7.0 Hz, 2H), 7.31 – 7.28 (m, 1H), 7.19 (d, *J* = 8.5 Hz, 3H), 7.12 (d, *J* = 7.6 Hz, 1H), 7.00 (dd, *J* = 9.0, 2.2 Hz, 1H), 6.96 – 6.92 (m, 1H), 6.90 (dd, *J* = 8.8, 2.4 Hz, 4H), 6.44 (s, 1H), 3.81 (s, 3H), 3.77 (s, 3H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 158.9, 158.6, 153.9, 153.7, 134.7, 132.5, 130.3, 130.1, 129.9, 129.7, 128.8, 128.1, 125.2, 121.7, 117.9, 117.0, 116.3, 115.4, 114.8, 103.0, 55.4, 55.3, 42.5.

ESI-MS+ m/z Calcd. for $C_{25}H_{22}NaO_4$ 409.14158 [M+ Na]; found 409.14090

Triarylmethane 17m:



Yield 94%; reddish brown solid; **Mpt** = 95-97ºC (CH_2Cl_2/hexane)

$[\alpha]_D^{24}$ = -21.6 (c = 1.02, $CHCl_3$)

Er = 97:3

IR (KBr) cm^{-1}756, 1033, 1508, 1606, 3466.

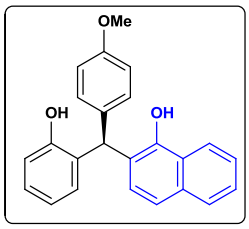
^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.10 (d, *J* = 8.9 Hz, 1H), 7.97 – 7.85 (m, 4H), 7.79 (d, *J* = 8.8 Hz, 1H), 7.63 – 7.37 (m, 6H), 7.22 (dd, *J* = 7.6, 1.2 Hz, 2H), 7.16 – 7.10 (m, 2H), 6.94 – 6.87 (m, 4H), 6.58 (s, 1H), 5.54 (brs, 1H), 5.01 (brs, 1H), 3.81 (s, 3H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 159.1, 153.9, 153.6, 140.1, 135.9, 131.0, 132.5, 132.4, 131.9, 130.2, 130.1, 129.9, 129.8, 129.7, 129.6, 129.0, 128.4, 127.9, 127.8, 127.3, 126.3, 126.2, 125.9, 125.6, 122.8, 121.9, 120.3, 118.8, 116.4, 115.0, 55.4, 42.5.

ESI-MS+ m/z Calcd. for $C_{34}H_{26}NaO_3$ 505.17796 [M+ Na]; found 505.17748

HPLC IA Column (90% hexane: 10% *i*-propanol, 1 mL/min, 240 nm) R_{t1} = 27.4 min and R_{t2} = 31.1 min.

Triarylmethane 18:



Yield 90%; yellowish brown solid; **Mpt** = 94-96ºC (CH_2Cl_2/hexane)

$[\alpha]_D^{24}$ = -17.5 (c = 0.8, $CHCl_3$)

Er = 93:7

IR (KBr) cm^{-1}756, 1032, 1244, 1509, 1625, 3442.

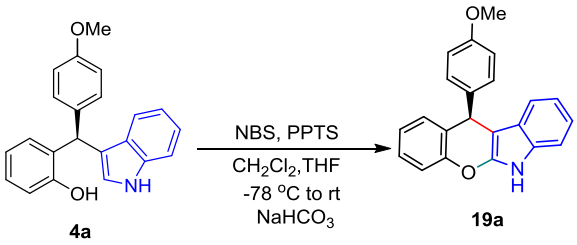
^{1}H NMR ($CDCl_3$, 400 MHz) δ 8.28 – 8.20 (m, 1H), 7.94 – 7.86 (m, 1H), 7.44 (dtd, J = 8.3, 6.8, 5.3 Hz, 2H), 7.15 (ddd, J = 8.0, 7.1, 1.9 Hz, 1H), 7.09 – 7.03 (m, 2H), 6.88 – 6.72 (m, 6H), 6.67 (d, J = 7.8 Hz, 1H), 6.26 (s, 1H), 3.79 (s, 3H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 158.4, 153.4, 151.0, 134.6, 133.0, 131.1, 130.8, 130.7, 130.6, 128.0, 127.0, 126.9, 125.2, 125.1, 124.3, 122.4, 121.0, 116.3, 114.2, 107.9, 55.4, 46.2.

ESI-MS+ m/z Calcd. for $C_{24}H_{20}NaO_3$ 379.13101 [M+ Na]; found 379.13033

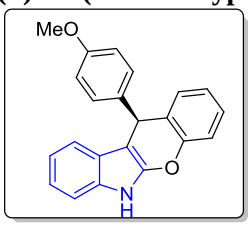
HPLC IA Column (80% hexane: 20% *i*-propanol, 1 mL/min, 230 nm) R_{t1} = 8.7 min and R_{t2} = 11.1 min.

General Procedure for the Synthesis of Dihydrochromeno[2,3-*b*]indoles 19a and 19b[1],[2]



Indole **4a** (0.057 g, 0.17 mmol) was dissolved in 2 mL of dry DCM and (0.047 g, 0.18 mmol) PPTS was added to it and the reaction mixture was cooled to -78 °C. At this temperature (0.033 g, 0.18 mmol) NBS dissolved in 2 mL of dry THF was added dropwise to the reaction mixture. The reaction mixture was allowed to stir at -78 °C for 45 min and then quenched with saturated $NaHCO_3$ solution The organic phase was diluted with CH_2Cl_2 and extracted to get hold of the crude product which was finally purified by column chromatography.

(S)-11-(4-Methoxyphenyl)-6,11-dihydrochromeno[2,3-*b*]indole (19a):



Yield 65%; pale yellow solid; **Mpt** = 206-208°C (CH_2Cl_2/hexane)

[α]$_D^{25}$ = +54.19 (c = 0.15, $CHCl_3$)

Er = 91:9

IR (KBr) cm^{-1}755, 2470, 3465.

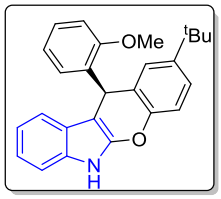
^{1}H NMR ($CDCl_3$, 400 MHz) δ 3.79 (s, 3H), 5.44 (s, 1H), 6.84 (d, 2H, *J* = 8.0 Hz), 6.98-7.09 (m, 4H), 7.15-7.17 (m, 2H), 7.28-7.30 (m, 4H), 7.86 (brs, 1H).

^{13}C NMR ($CDCl_3$, 100 MHz) δ 40.0, 55.2, 90.3, 110.4, 113.9, 116.9, 118.1, 120.2, 120.5, 124.0, 125.3, 126.2, 127.5, 129.6, 130.9, 131.1, 138.0, 144.5, 150.3, 158.2.

ESI-MS+ m/z Calcd. for $C_{22}H_{17}NO_2$ 350.1157 [M+ Na]; found 350.1551.

HPLC IA Column (98% hexane: 2% *i*-propanol, 1 mL/min, 228 nm) R_{t1} = 15.9 min and R_{t2} = 18.8 min.

(S)-2-(tert-Butyl)-11-(2-methoxyphenyl)-6,11-dihydrochromeno[2,3-*b*]indole (19b):



Yield 60%; off-white solid; **Mpt** = 222-224°C (CH_2Cl_2/hexane)

[α]$_D^{25}$ = +37.2 (c = 0.11, $CHCl_3$)

Er = 92:8

IR (KBr) cm^{-1}753, 1489, 3420.

^{1}H NMR ($CDCl_3$, 300 MHz) δ 1.24 (s, 9H), 4.10 (s, 3H), 6.04 (s, 1H), 6.74-6.80 (m, 2H), 6.86-6.87 (m, 1H), 6.97-7.08 (m, 6H), 7.13-7.24 (m, 2H), 7.85 (brs, 1H).

^{13}C NMR ($CDCl_3$, 75 MHz) δ31.6, 34.5, 55.8, 90.4, 110.4, 110.9, 116.2, 118.3, 120.2, 120.4, 121.3, 124.6, 125.1, 126.5, 127.5, 127.6, 130.5, 131.1, 134.5, 145.8, 146.9, 148.8, 156.8, 162.8.

ESI-MS+ m/z Calcd. for $C_{26}H_{25}NO_2$ 406.1783 [M+ Na]; found 406.1776.

HPLC OD-H Column (98% hexane: 2% *i*-propanol, 1 mL/min, 221 nm) R_{t1} = 35.2 min and R_{t2} = 47.0 min.

References

1. T. P. Pathak, K. M. Gligorich, B. E. Welm, M. S. Sigman, *J. Am. Chem. Soc*. **2010**, *132*, 7870.
2. R. Jana, T. P. Pathak, K. H. Jensen, M. S. Sigman, *Org. Lett.* **2012**, *14*, 4074

^{1}H And ^{13}C NMR Spectral Reproductions of F-C products (4, 5-14, 17-19)

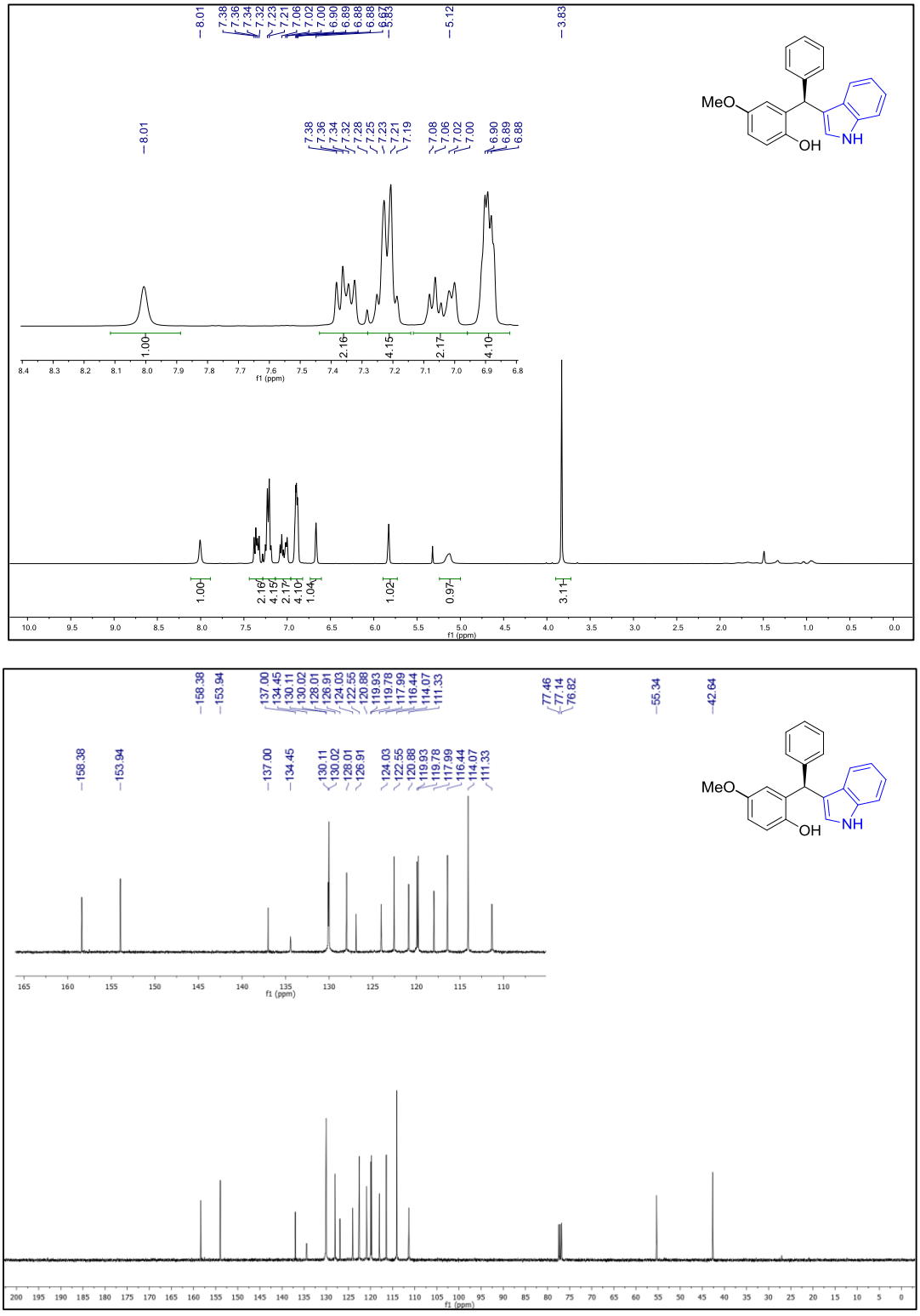


Figure 1.1. ^{1}H (400 MHz, $CDCl_3$) and^{13}C (100 MHz, $CDCl_3$) NMR of(S)-2-((1H-indol-3-yl)(4-methoxyphenyl)methyl)phenol (**4a**).

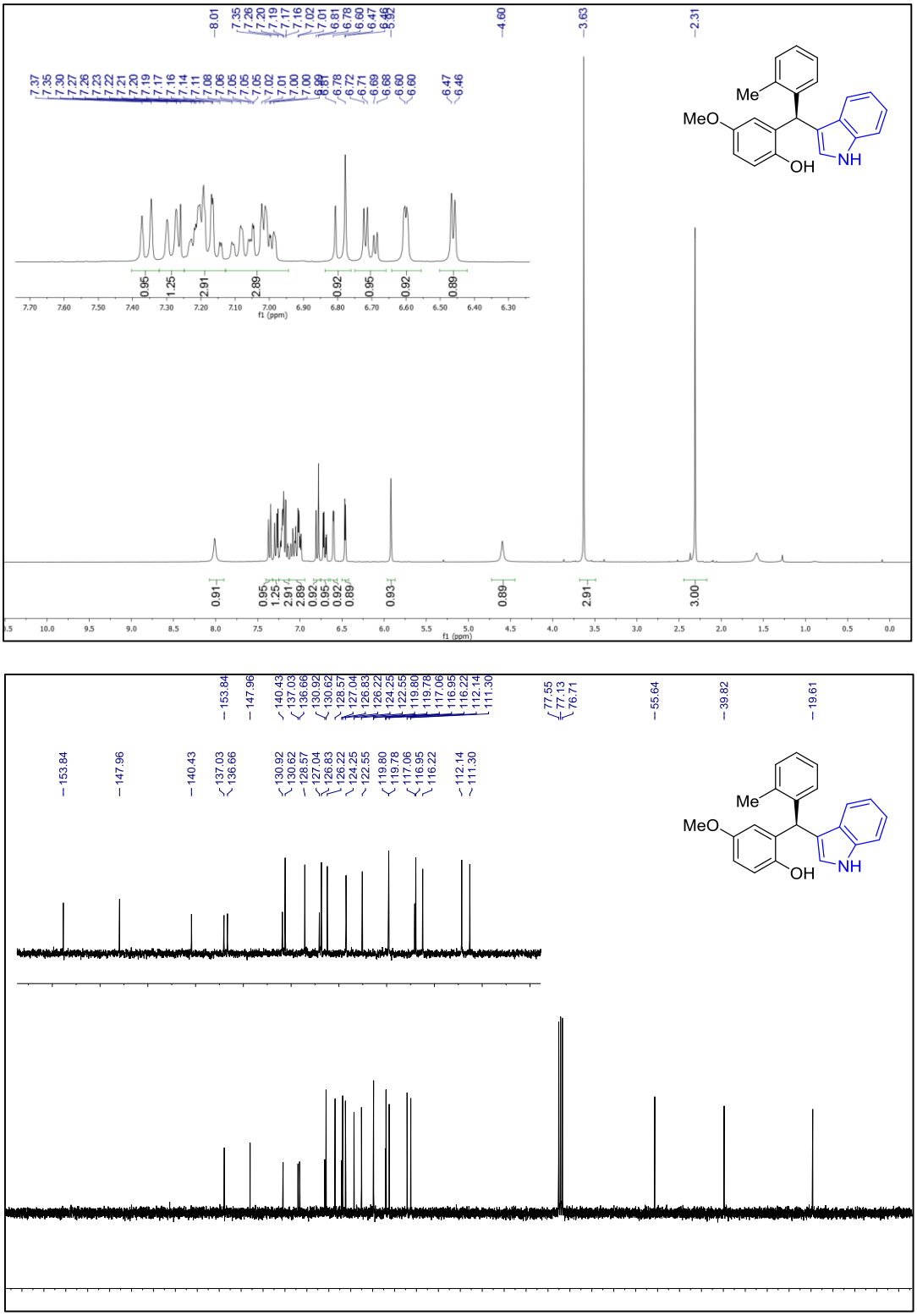


Figure 1.2. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-2-((1H-indol-3-yl)(*o*-tolyl)methyl)-4-methoxyphenol (**4b**).

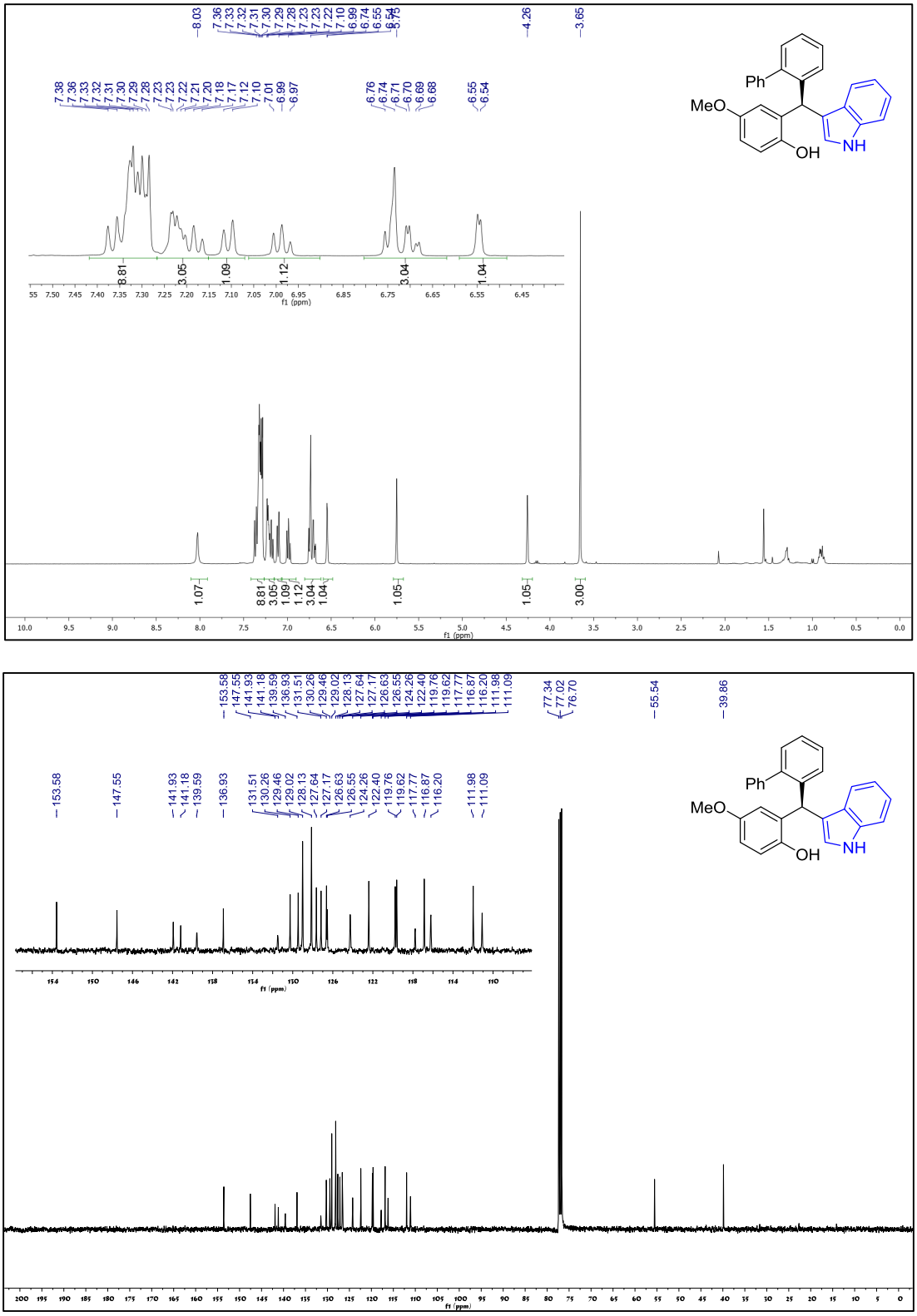


Figure 1.3. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-2-([1,1'-biphenyl]-2-yl(1H-indol-3-yl)methyl)-4-methoxyphenol (**4c**).

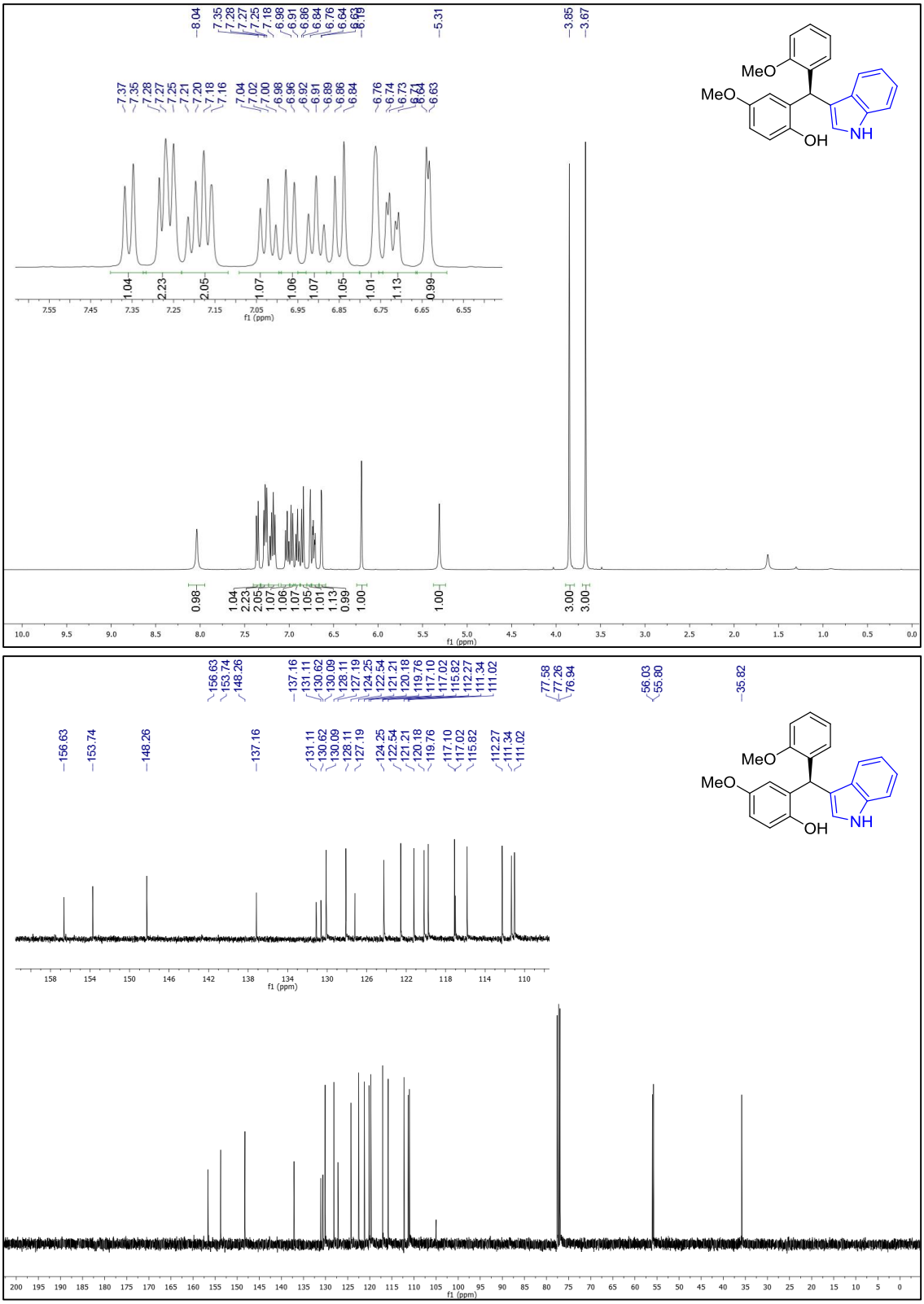


Figure 1.4. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-2-((1H-indol-3-yl)(2-methoxyphenyl)methyl)-4-methoxyphenol (**4d**).

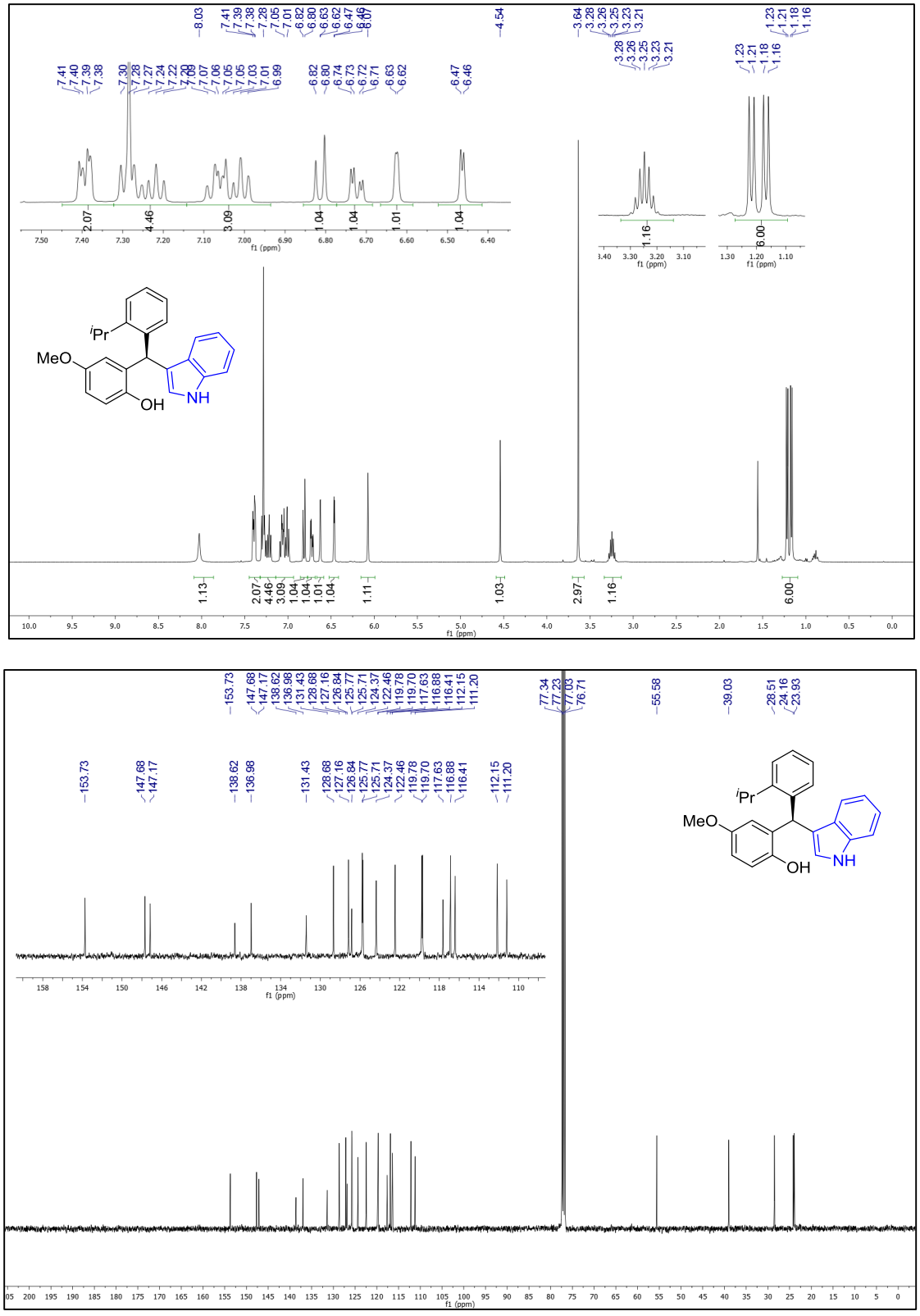


Figure 1.5. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-2-((1H-indol-3-yl)(2-isopropylphenyl)methyl)-4-methoxyphenol (**4e**).

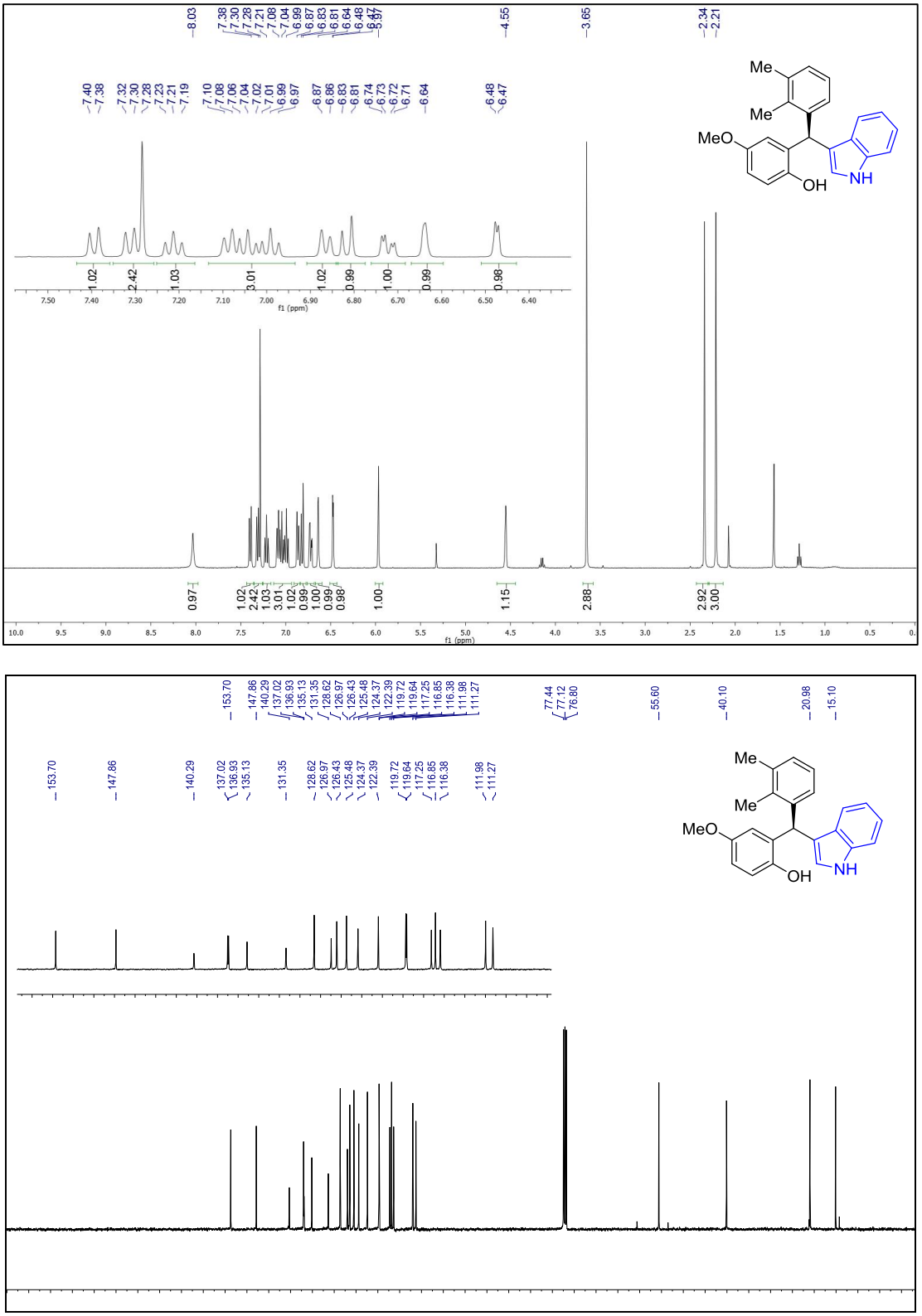


Figure 1.6. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of (S)-2-((2,3-dimethylphenyl)(1H-indol-3-yl)methyl)-4-methoxyphenol (**4f**).

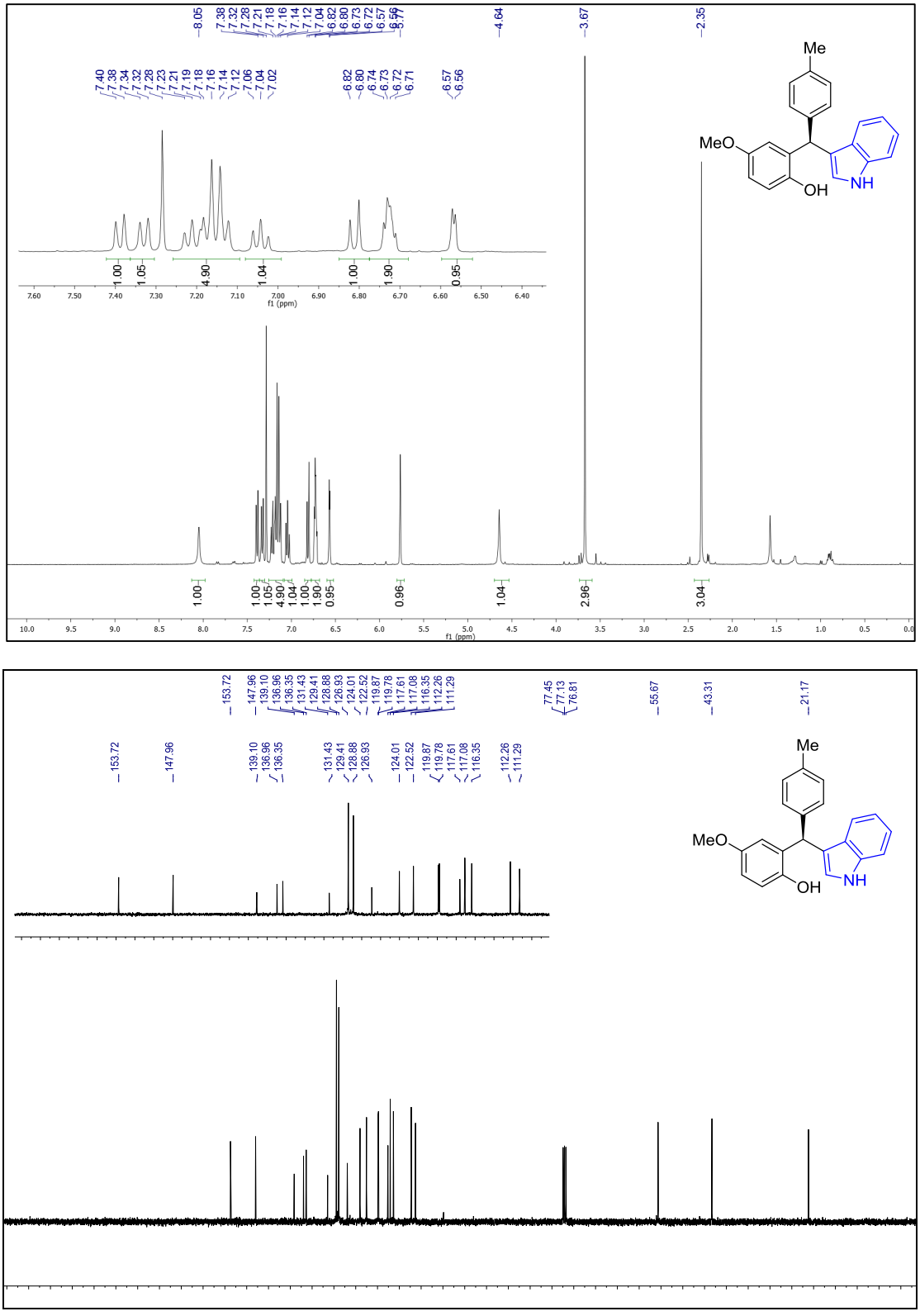


Figure 1.7. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-2-((1H-indol-3-yl)(*p*-tolyl)methyl)-4-methoxyphenol (**4g**).

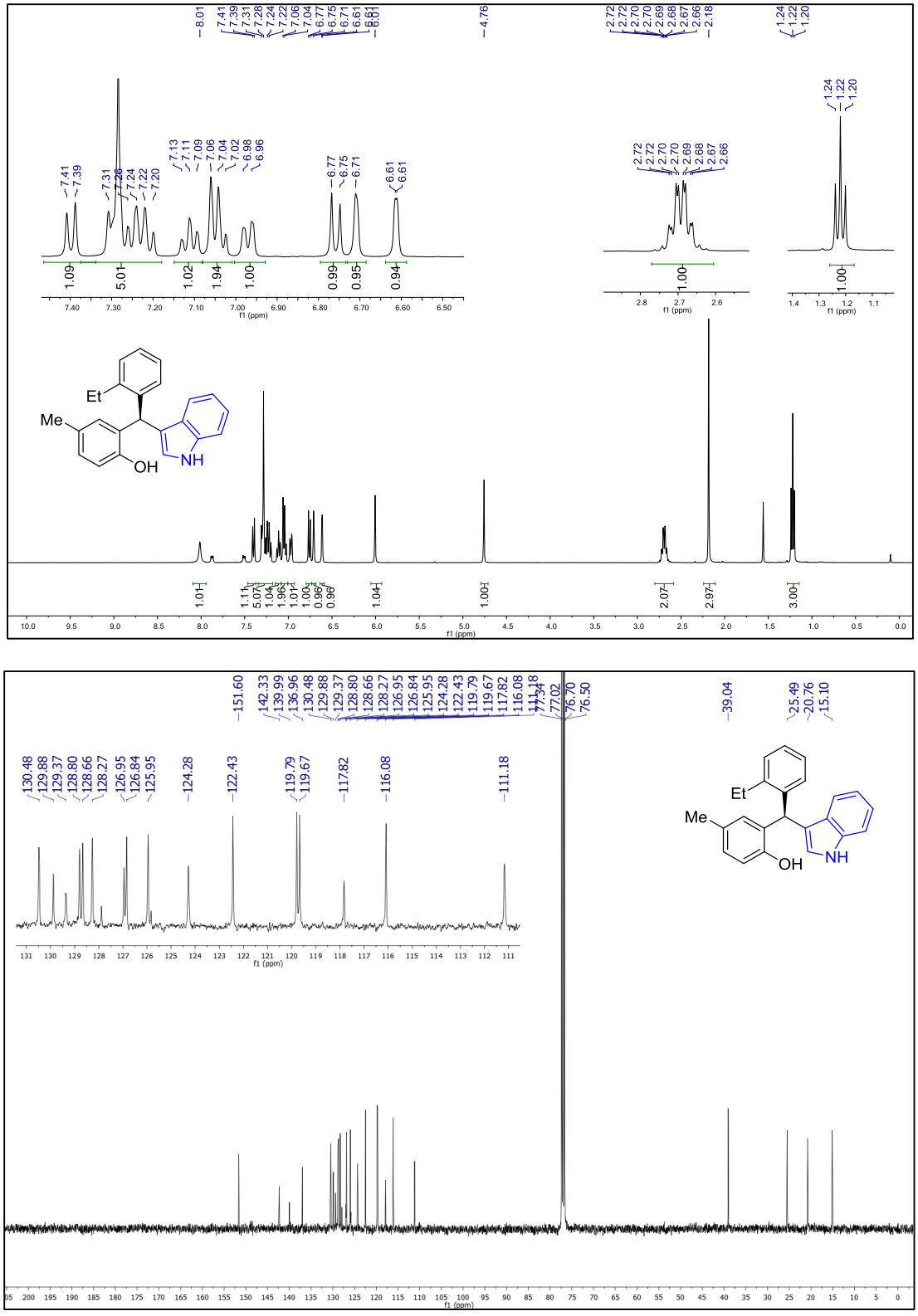


Figure 1.8. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-2-((2-ethylphenyl)(1H-indol-3-yl)methyl)-4-methylphenol (**4h**).

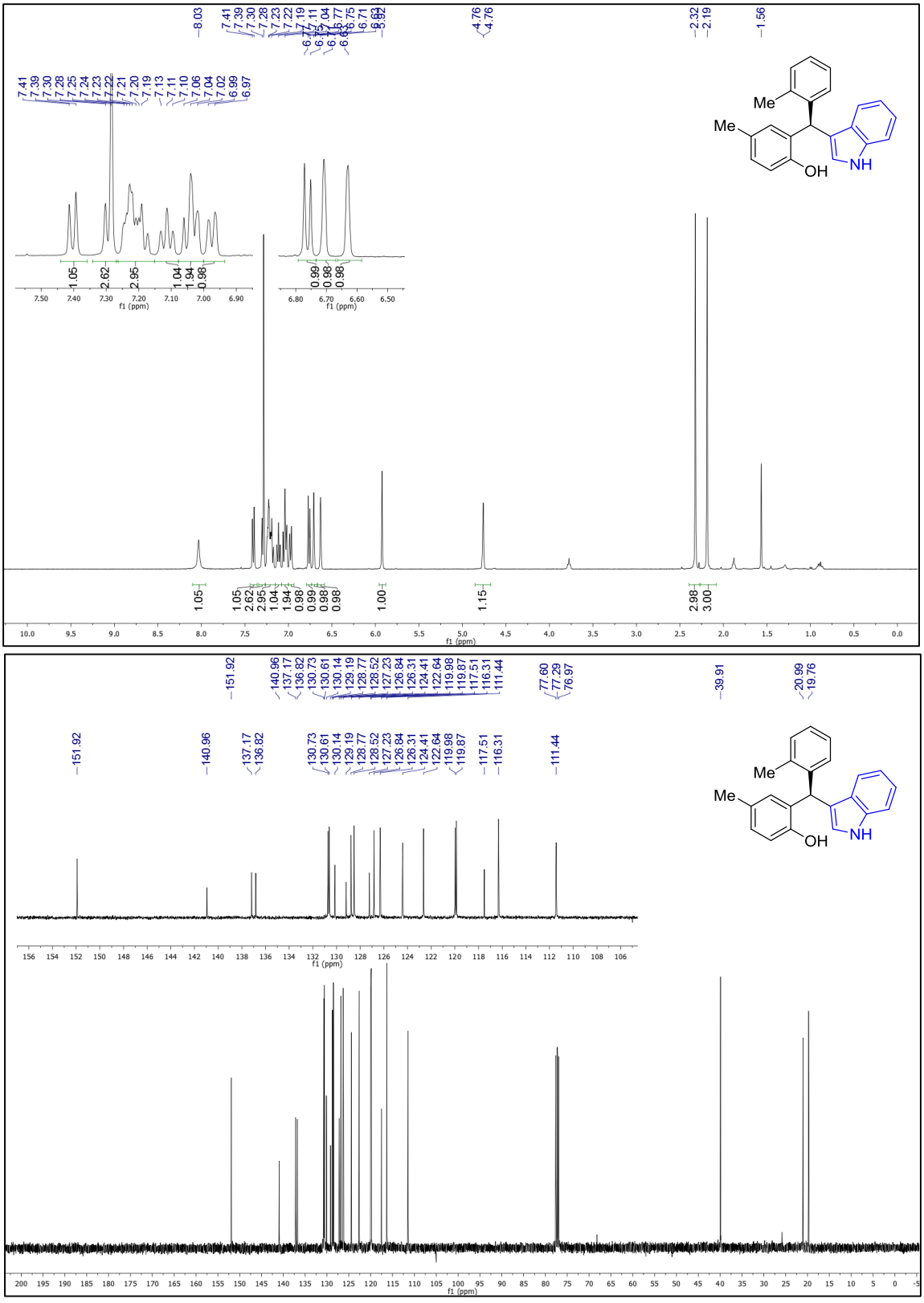


Figure 1.9. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-2-((1H-indol-3-yl)(*o*-tolyl)methyl)-4-methylphenol (**4i**).

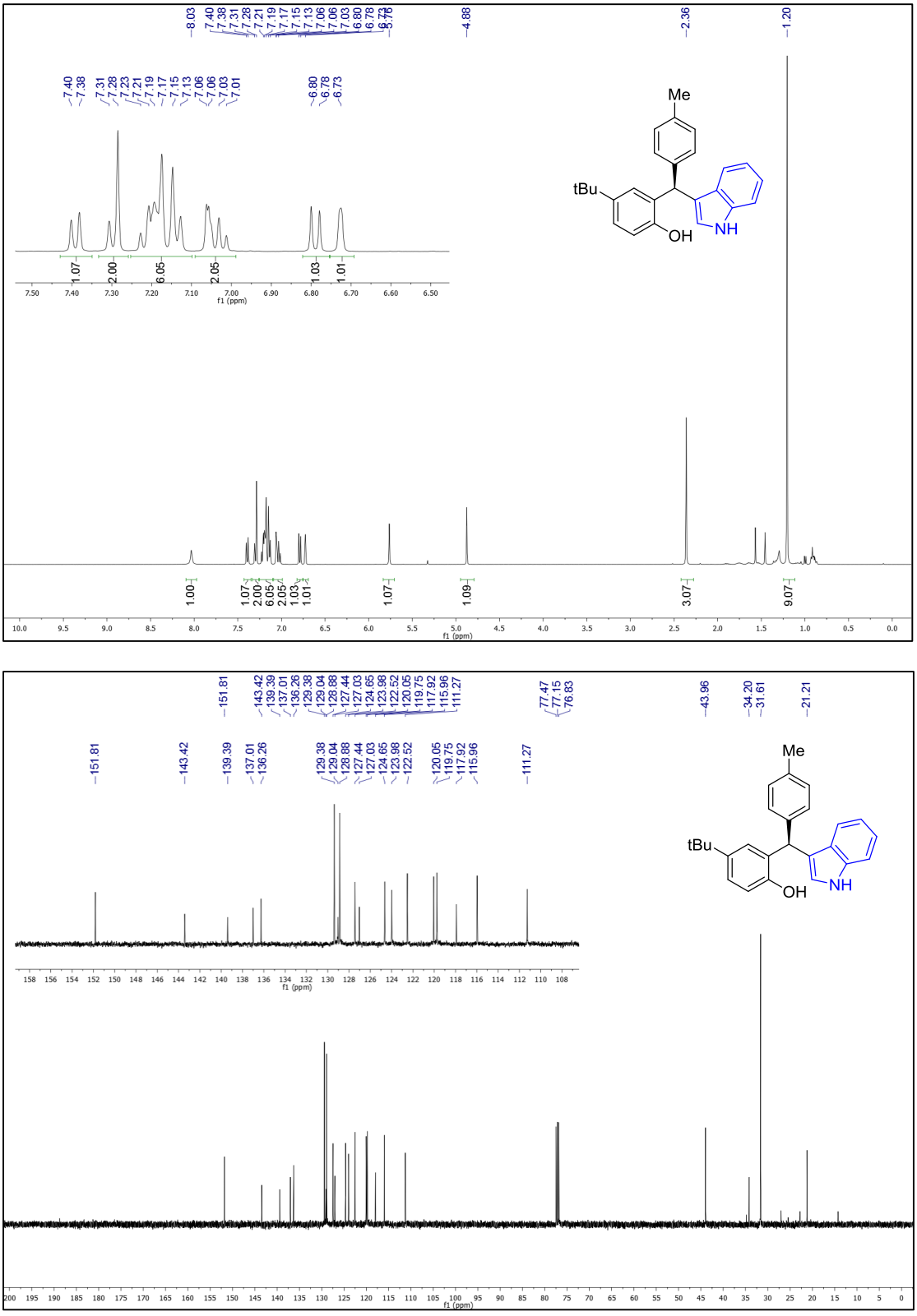


Figure 1.10. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-2-((1H-indol-3-yl)(*p*-tolyl)methyl)-4-(tert-butyl)phenol (**4j**).

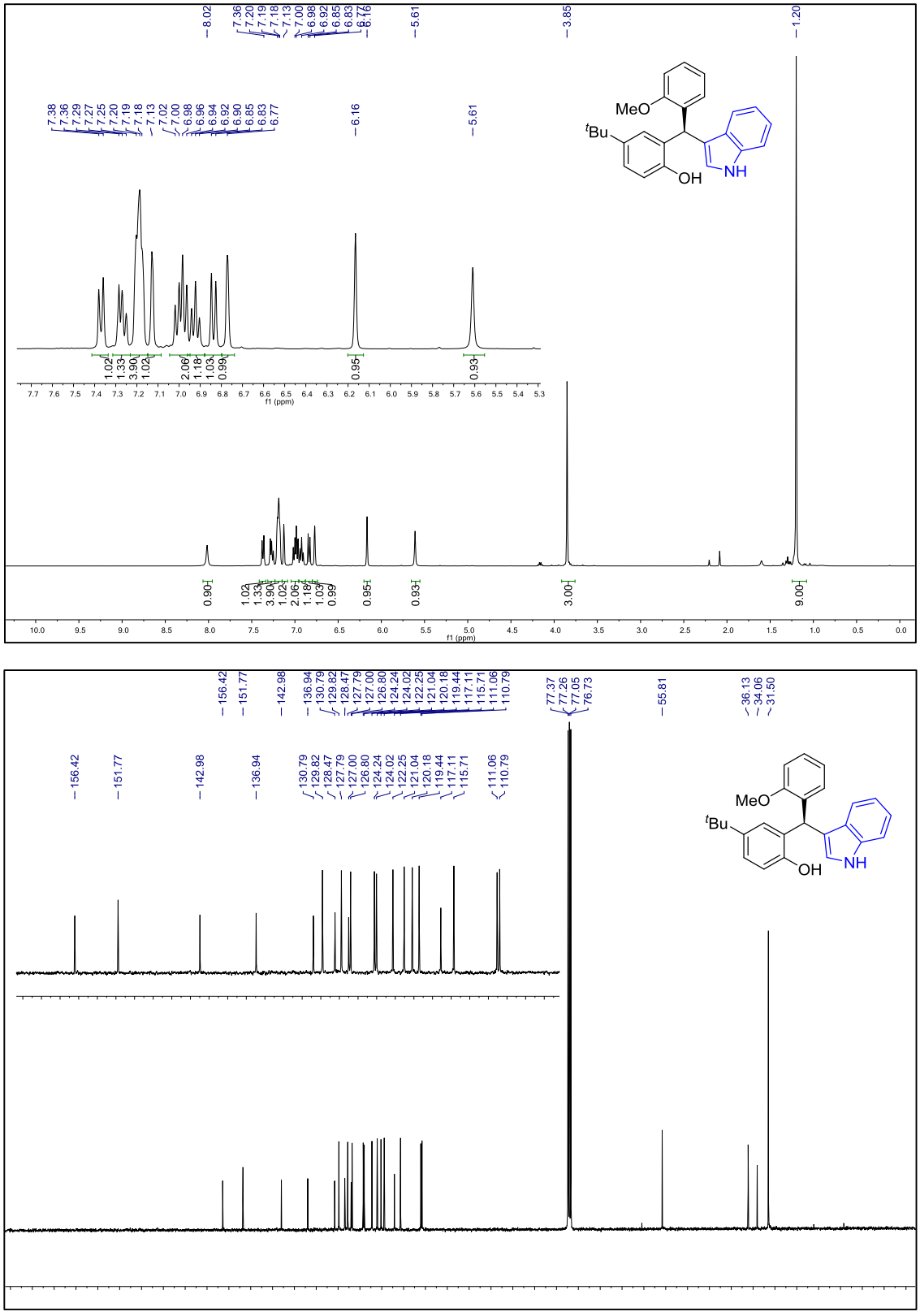


Figure 1.11. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of (S)-2-((1H-indol-3-yl)(2-methoxyphenyl)methyl)-4-(tert-butyl)phenol (**4k**).

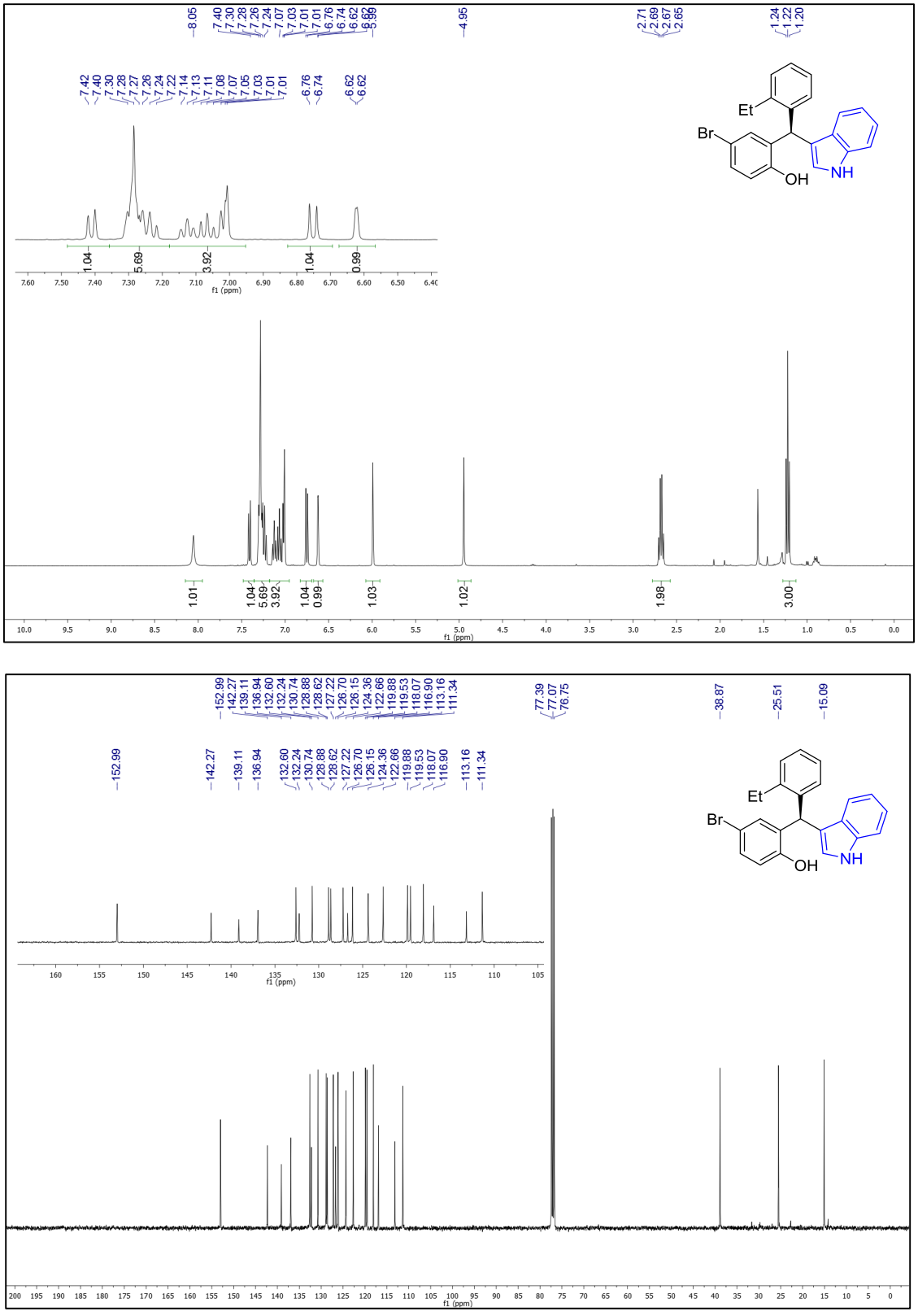


Figure 1.12. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-4-bromo-2-((2-ethylphenyl)(1H-indol-3-yl)methyl)phenol (**4l**).

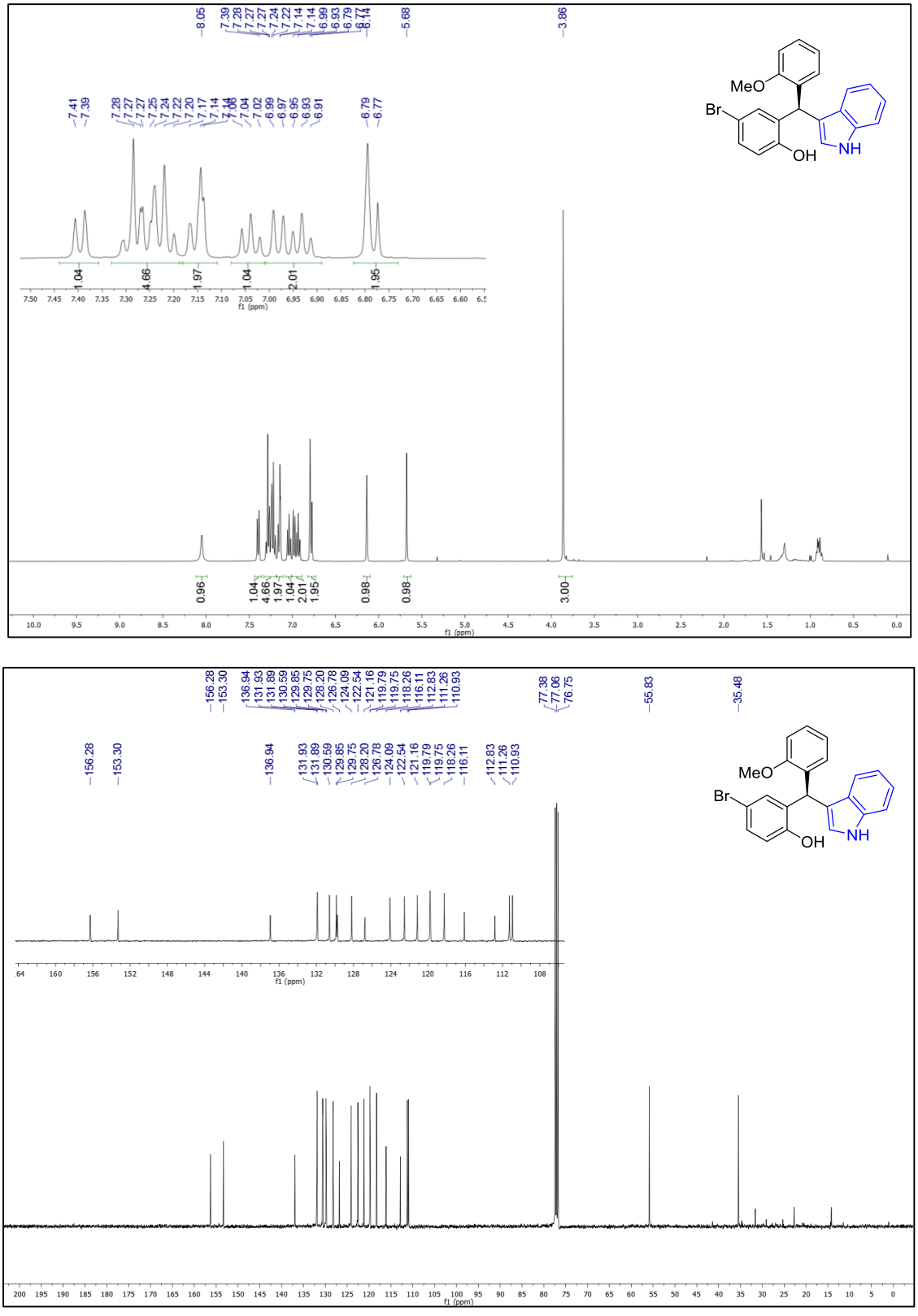


Figure 1.13. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-2-((1H-indol-3-yl)(2-methoxyphenyl)methyl)-4-bromophenol (**4m**).

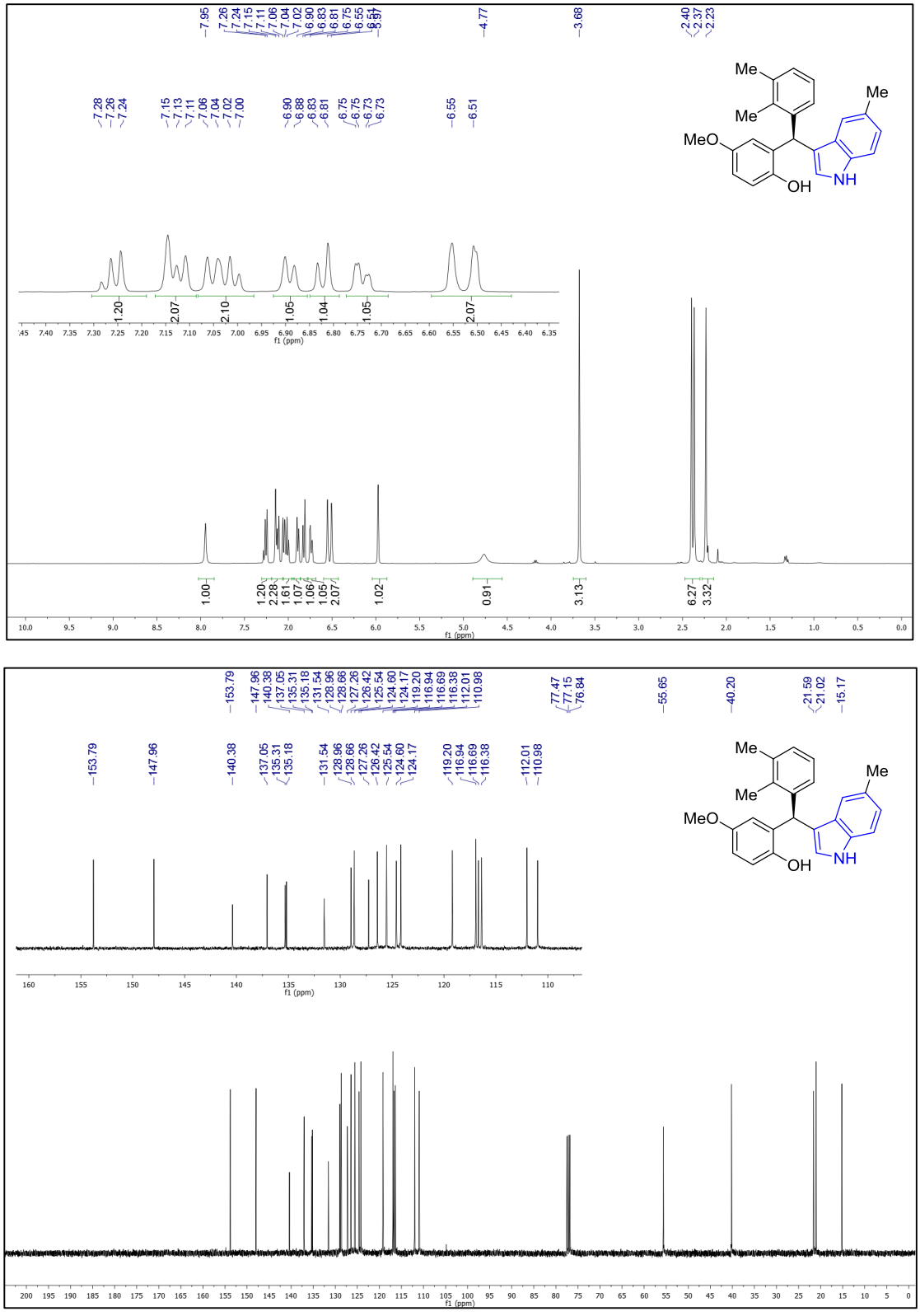


Figure 1.14. ^{1}H (400 MHz, $CDCl_3$) and^{13}C (100 MHz, $CDCl_3$) NMR of(S)-2-((2,3-dimethylphenyl)(5-methyl-1H-indol-3-yl)methyl)-4-methoxyphenol (**5a**).

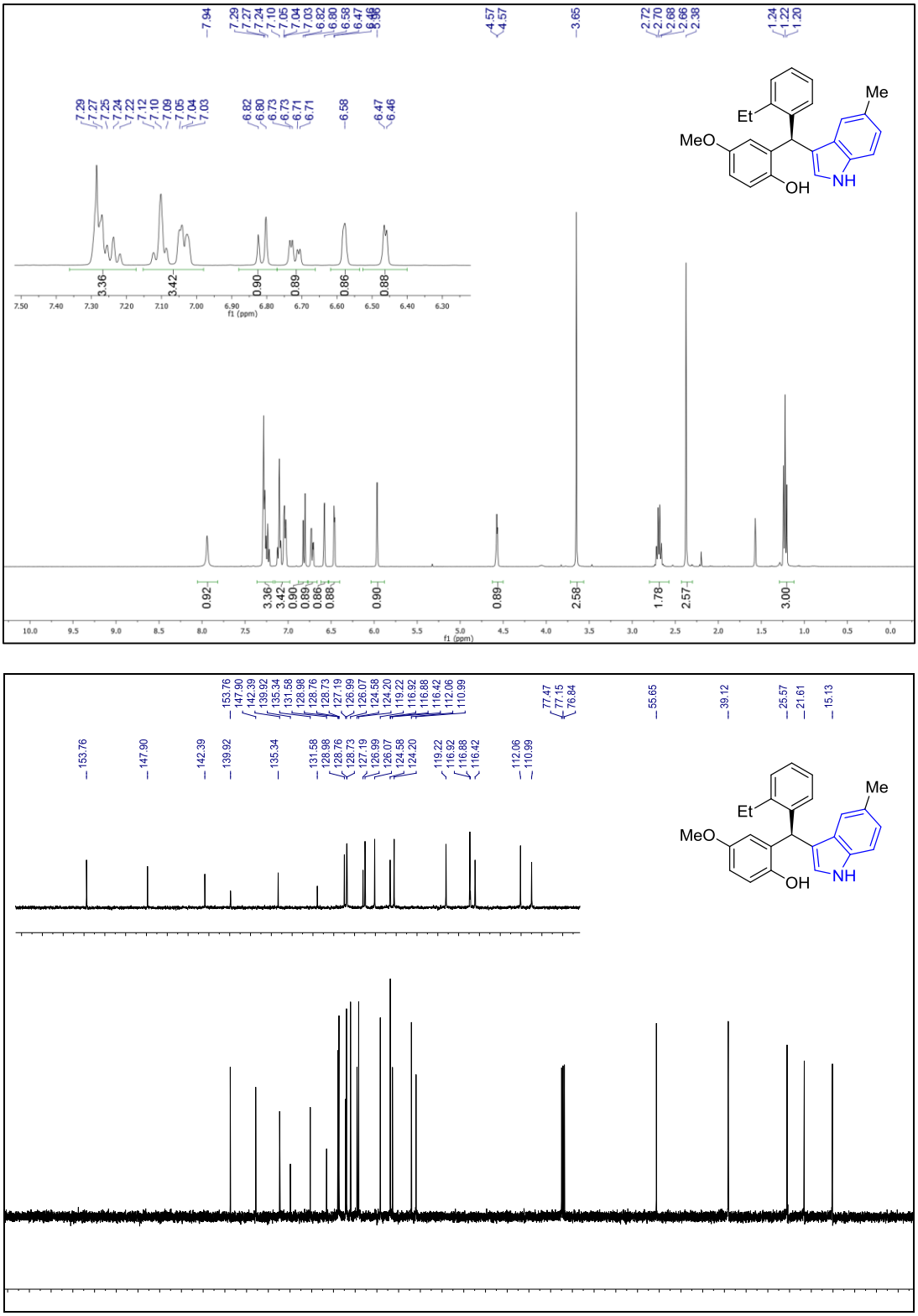


Figure 1.15. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-2-((2-ethylphenyl)(5-methyl-1H-indol-3-yl)methyl)-4-methoxyphenol (**5b**).

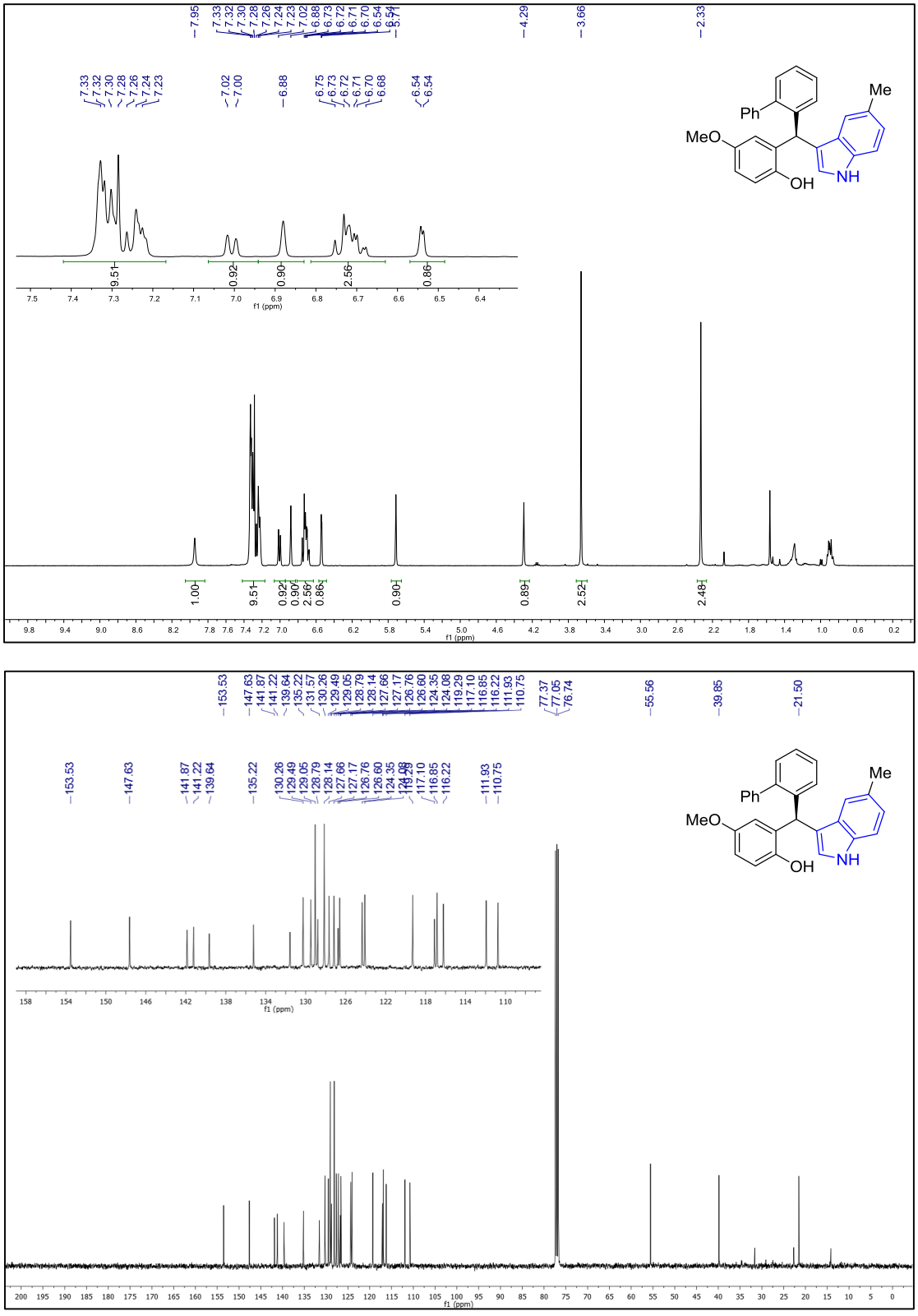


Figure 1.16. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-2-([1,1'-biphenyl]-2-yl(5-methyl-1H-indol-3-yl)methyl)-4-methoxyphenol (**5c**).

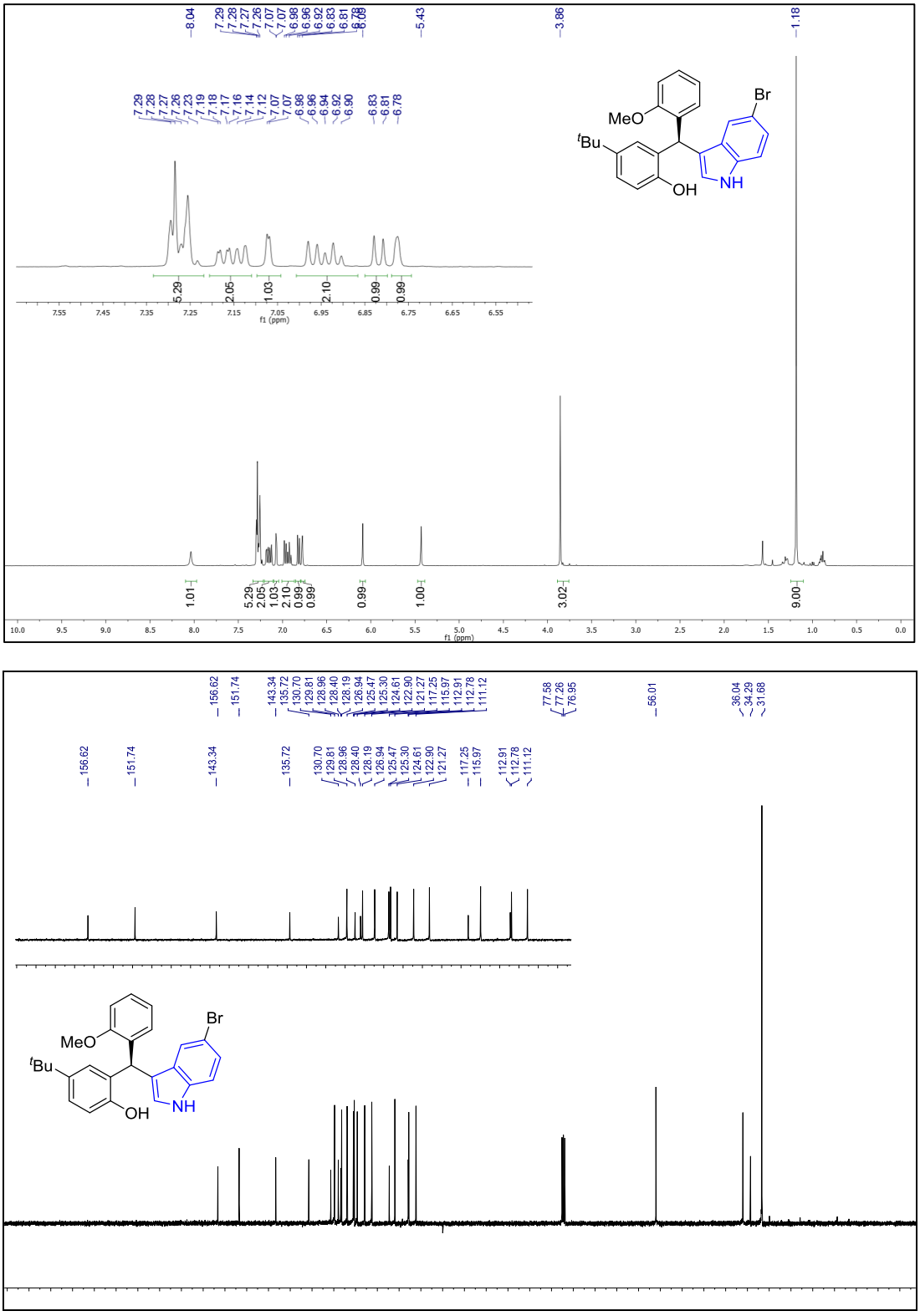


Figure 1.17. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-2-((5-bromo-1H-indol-3-yl)(2-methoxyphenyl)methyl)-4-(tert-butyl)phenol (**6a**).

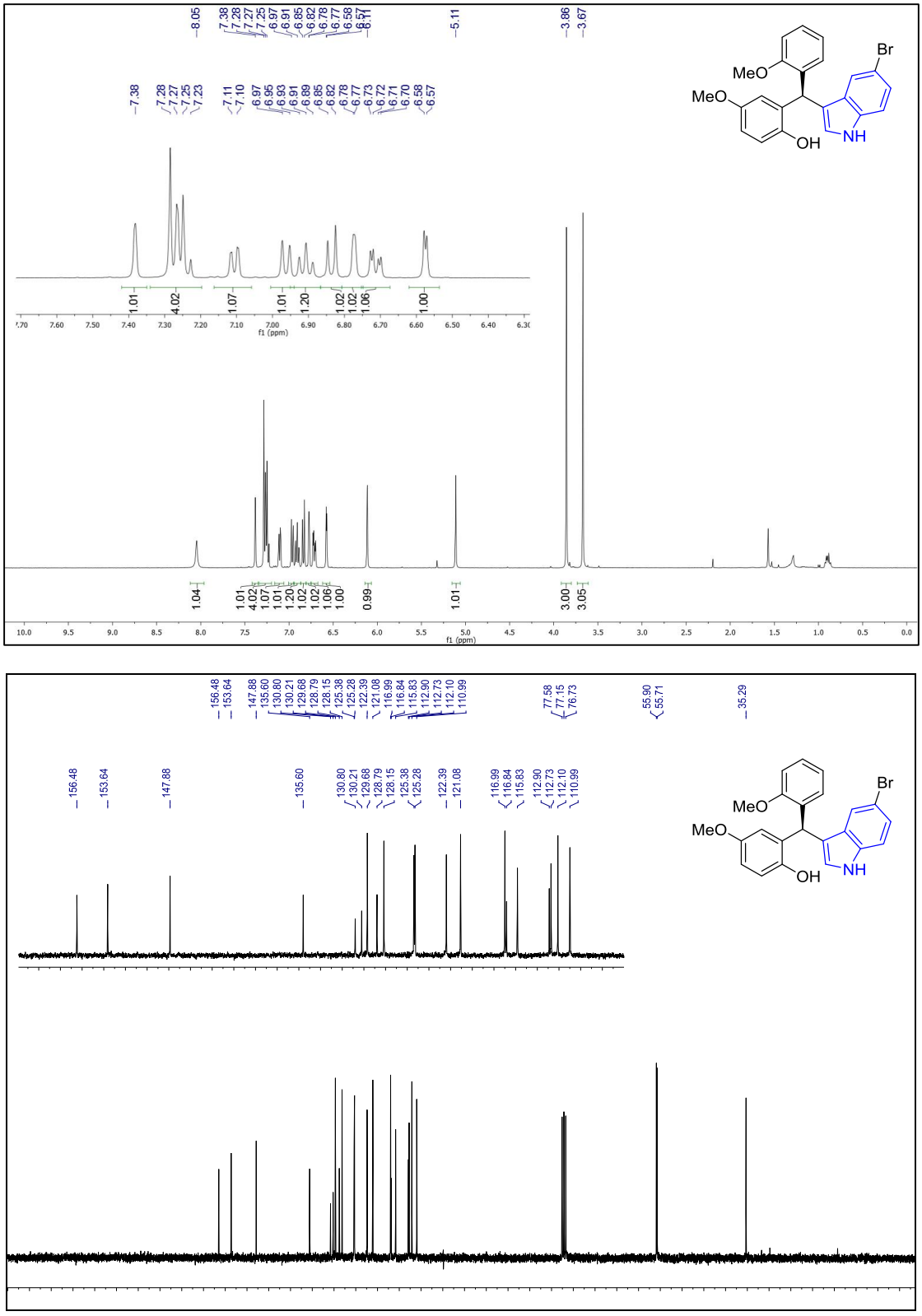


Figure 1.18. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-2-((5-bromo-1H-indol-3-yl)(2-methoxyphenyl)methyl)-4-methoxyphenol (**6b**).

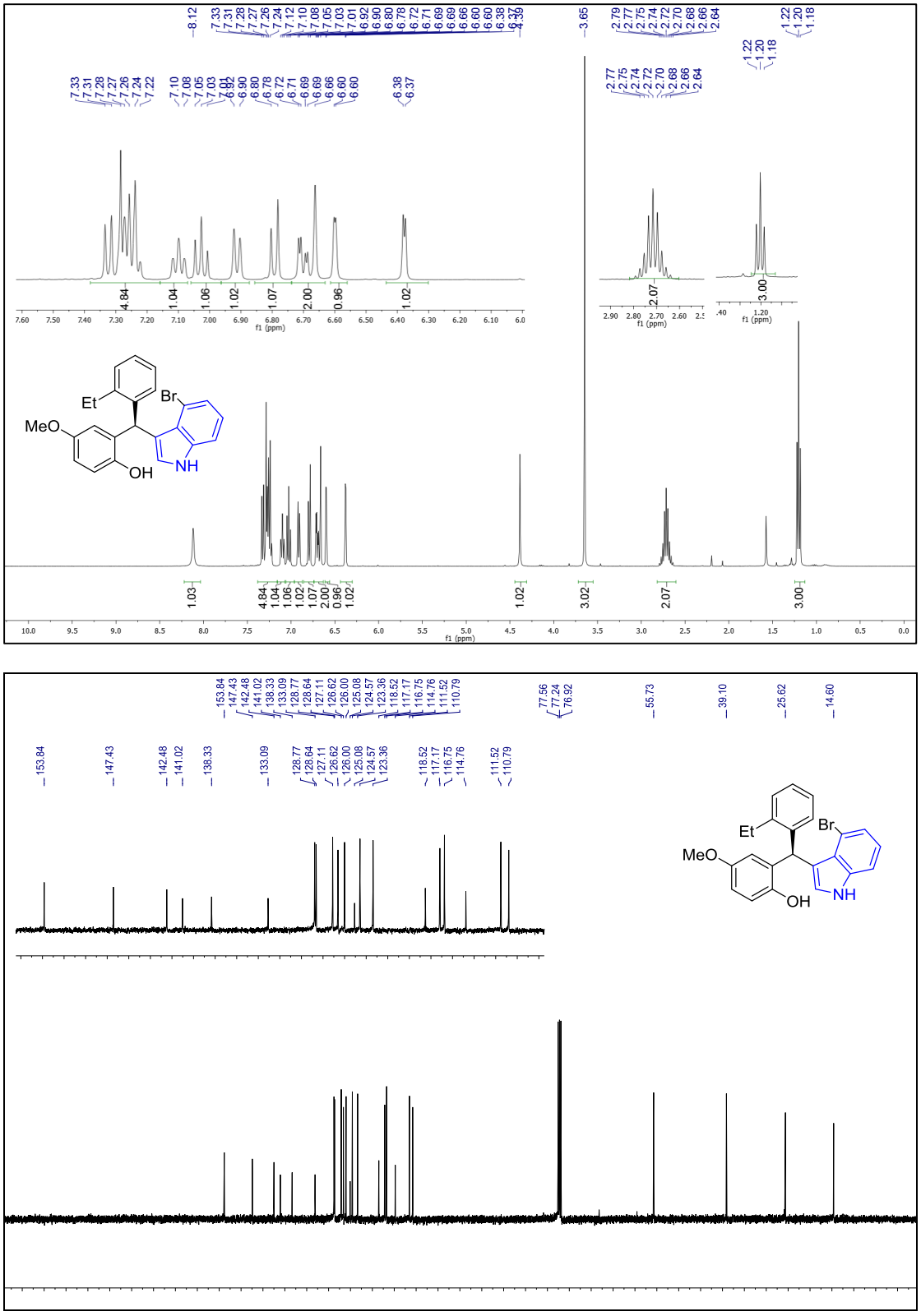


Figure 1.19. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-2-((4-bromo-1H-indol-3-yl)(2-ethylphenyl)methyl)-4-methoxyphenol (**7a**).

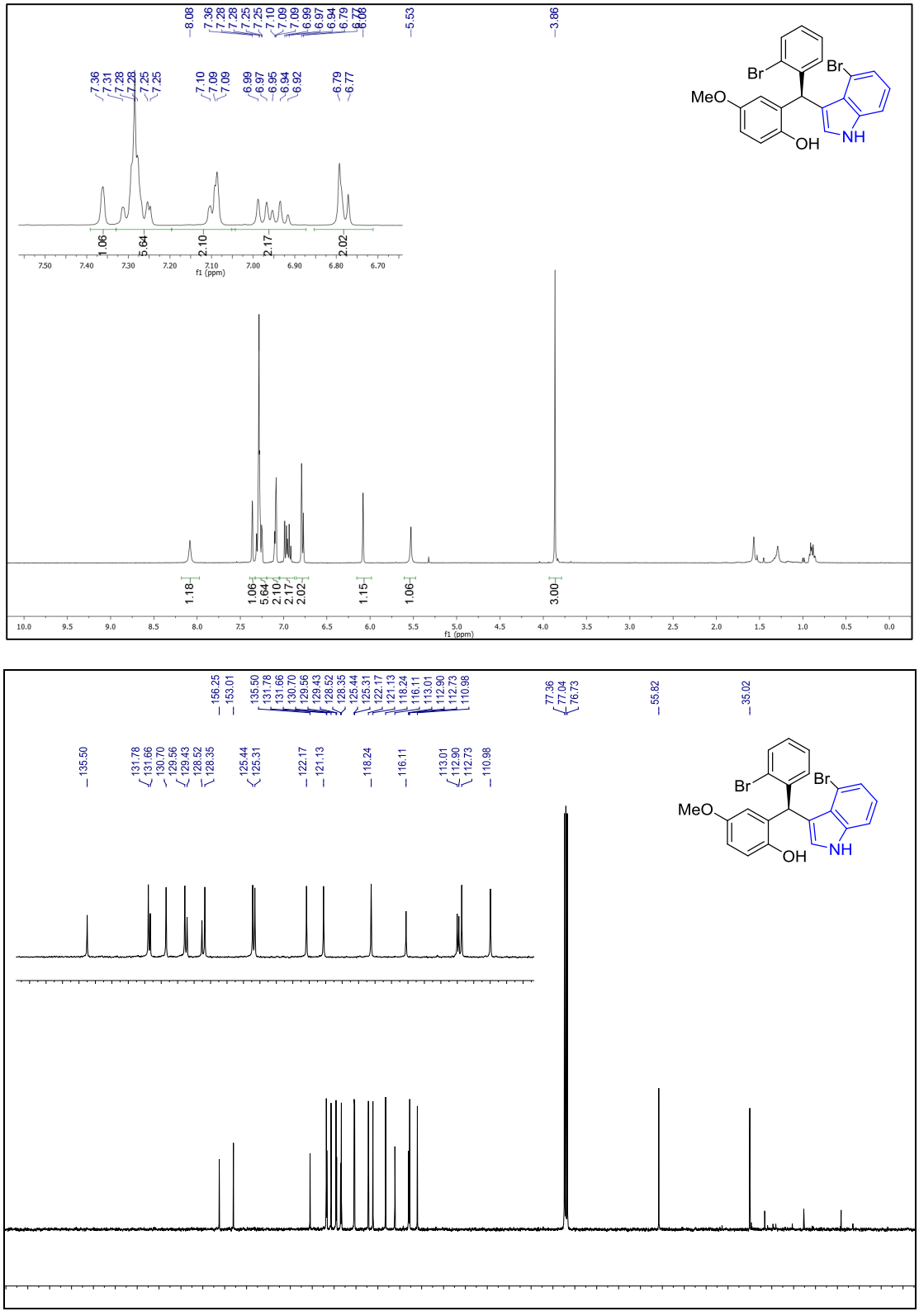


Figure 1.20. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-2-((4-bromo-1H-indol-3-yl)(2-bromophenyl)methyl)-4-methoxyphenol (**7b**).

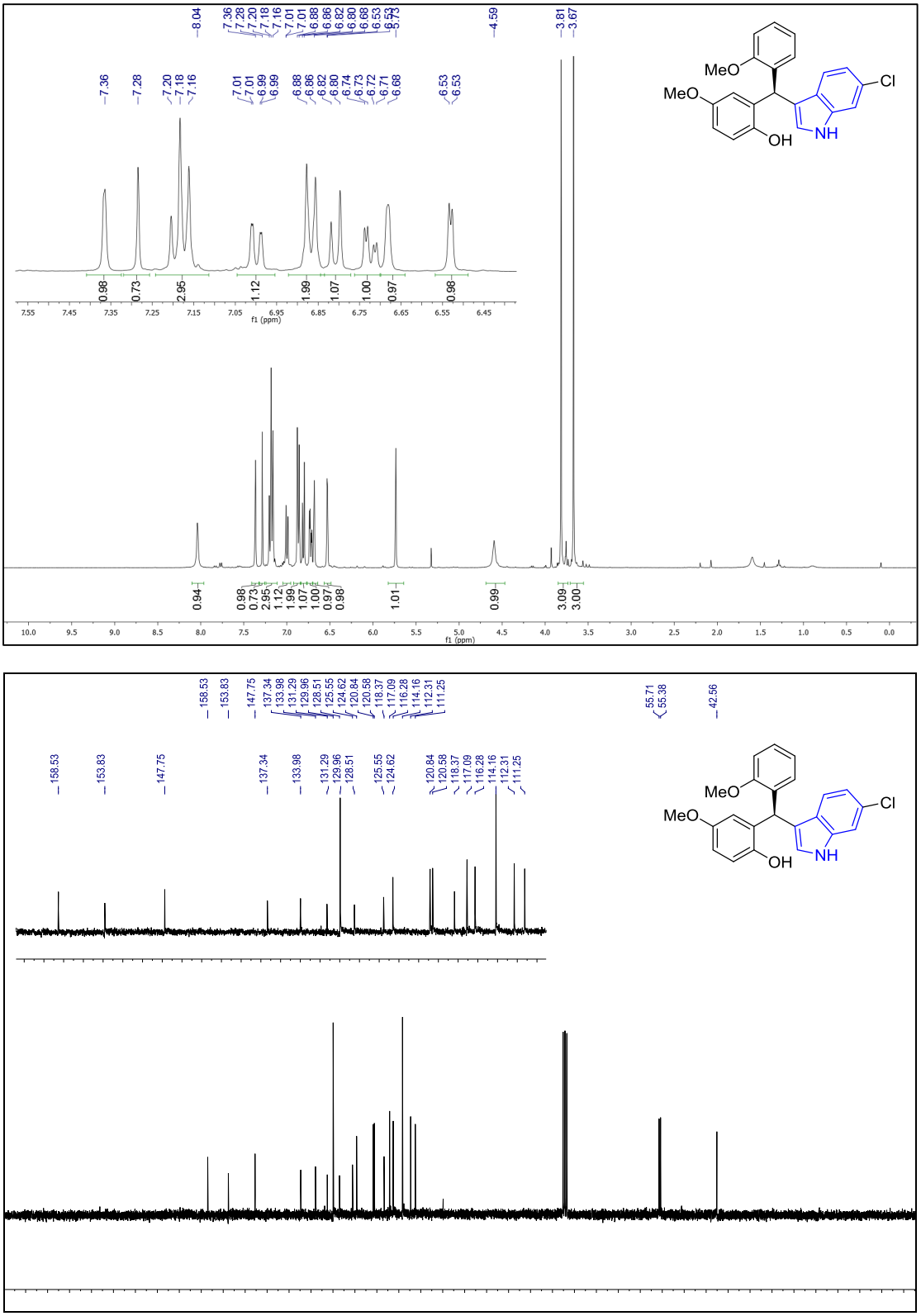


Figure 1.21. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-2-((6-chloro-1H-indol-3-yl)(2-methoxyphenyl)methyl)-4-methoxyphenol (**8a**).

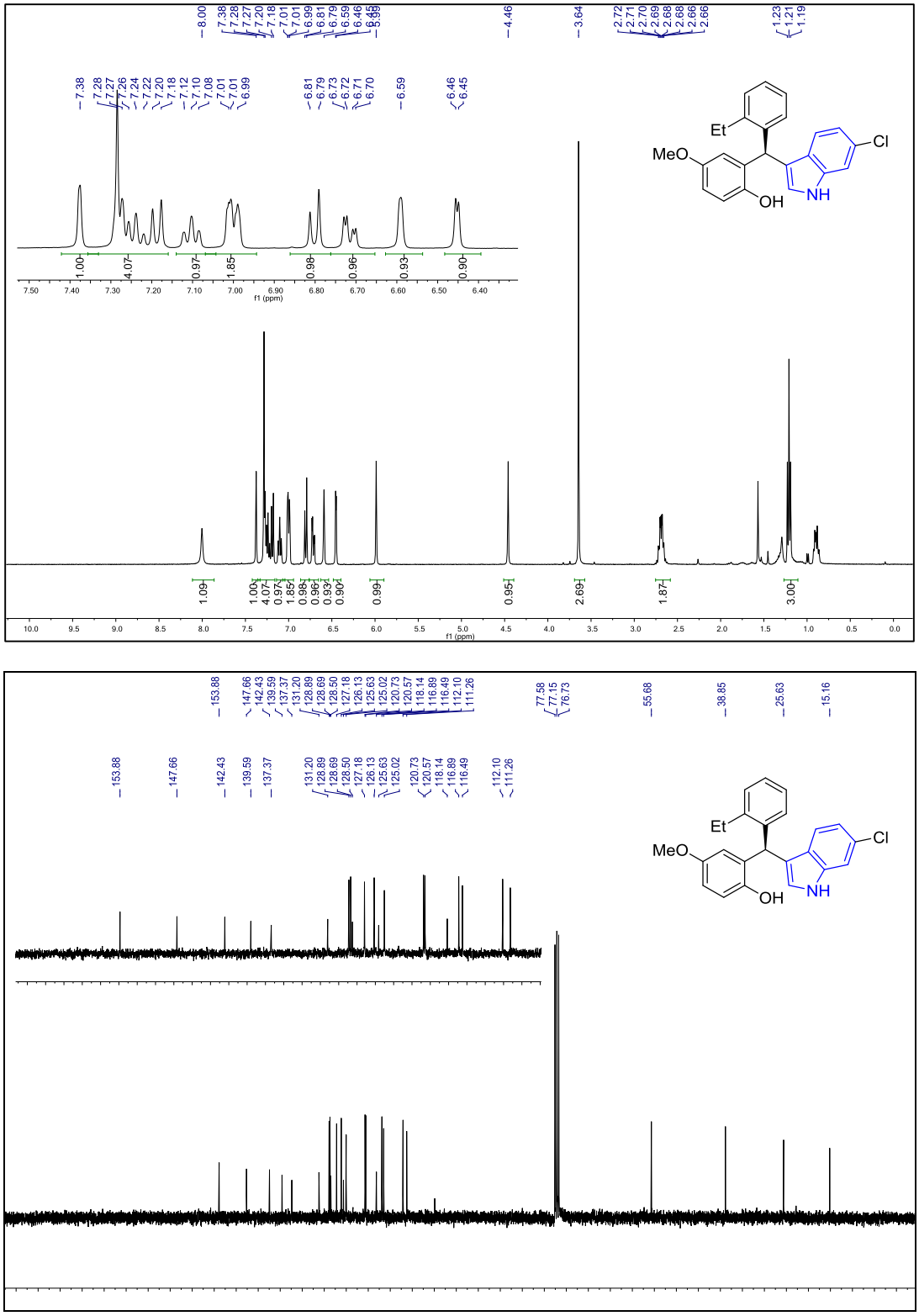


Figure 1.22. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-2-((6-chloro-1H-indol-3-yl)(2-ethylphenyl)methyl)-4-methoxyphenol (**8b**).

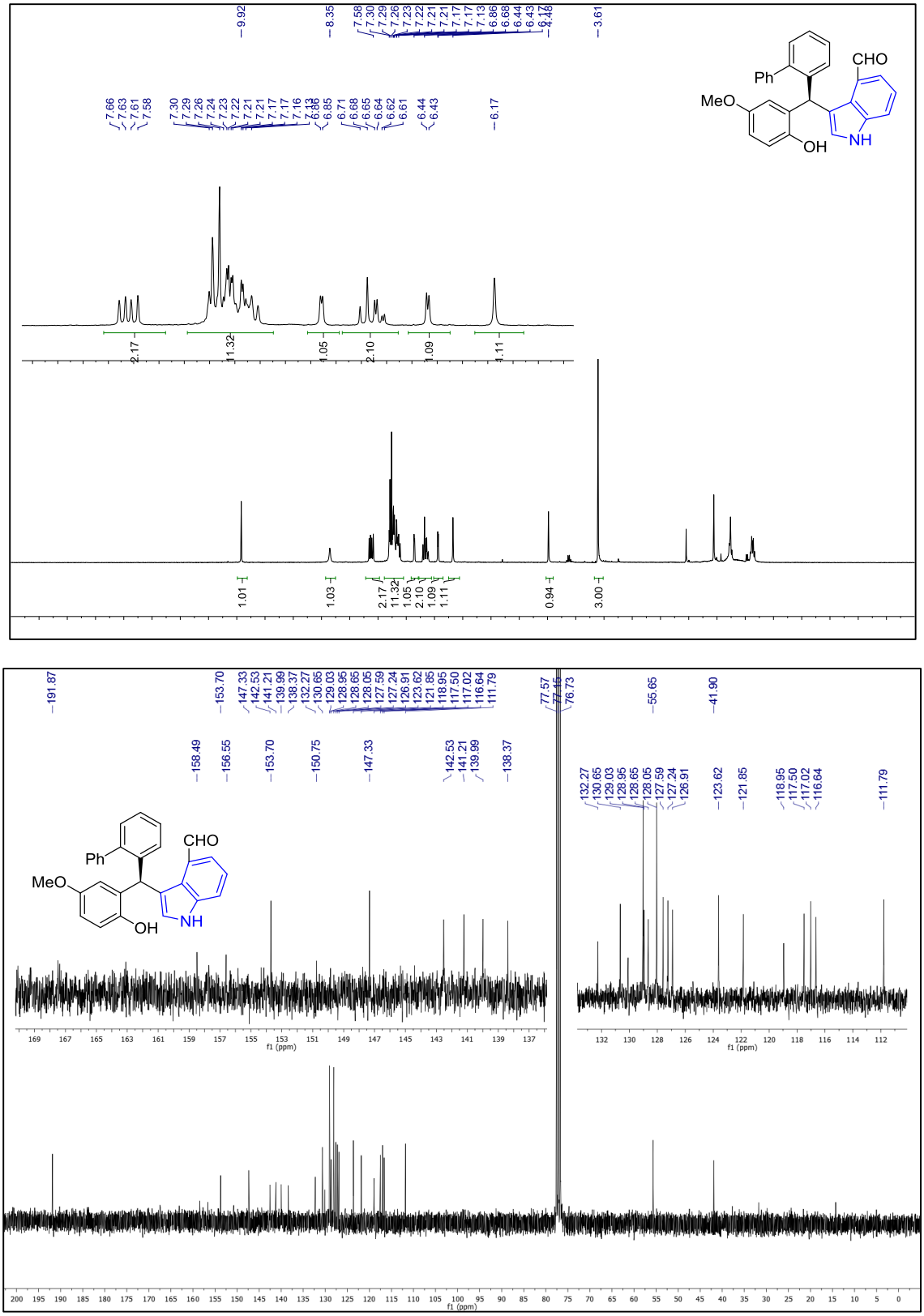


Figure 1.23. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of (S)-3-([1,1'-biphenyl]-2-yl(2-hydroxy-5-methoxyphenyl)methyl)-1H-indole-4-carbaldehyde (**9a**).

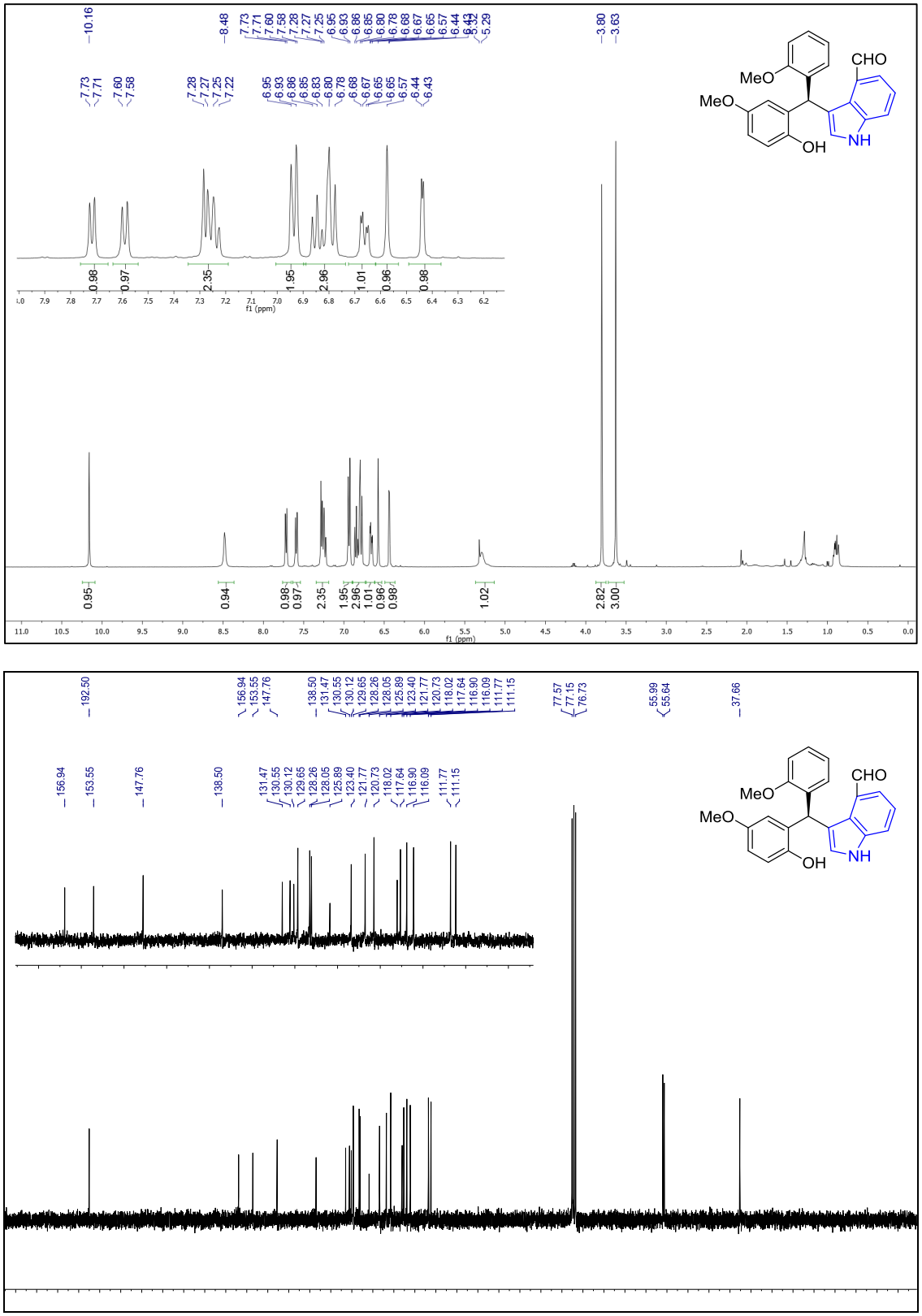


Figure 1.24. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-3-((2-hydroxy-5-methoxyphenyl)(2-methoxyphenyl)methyl)-1H-indole-4-carbaldehyde (**9b**).

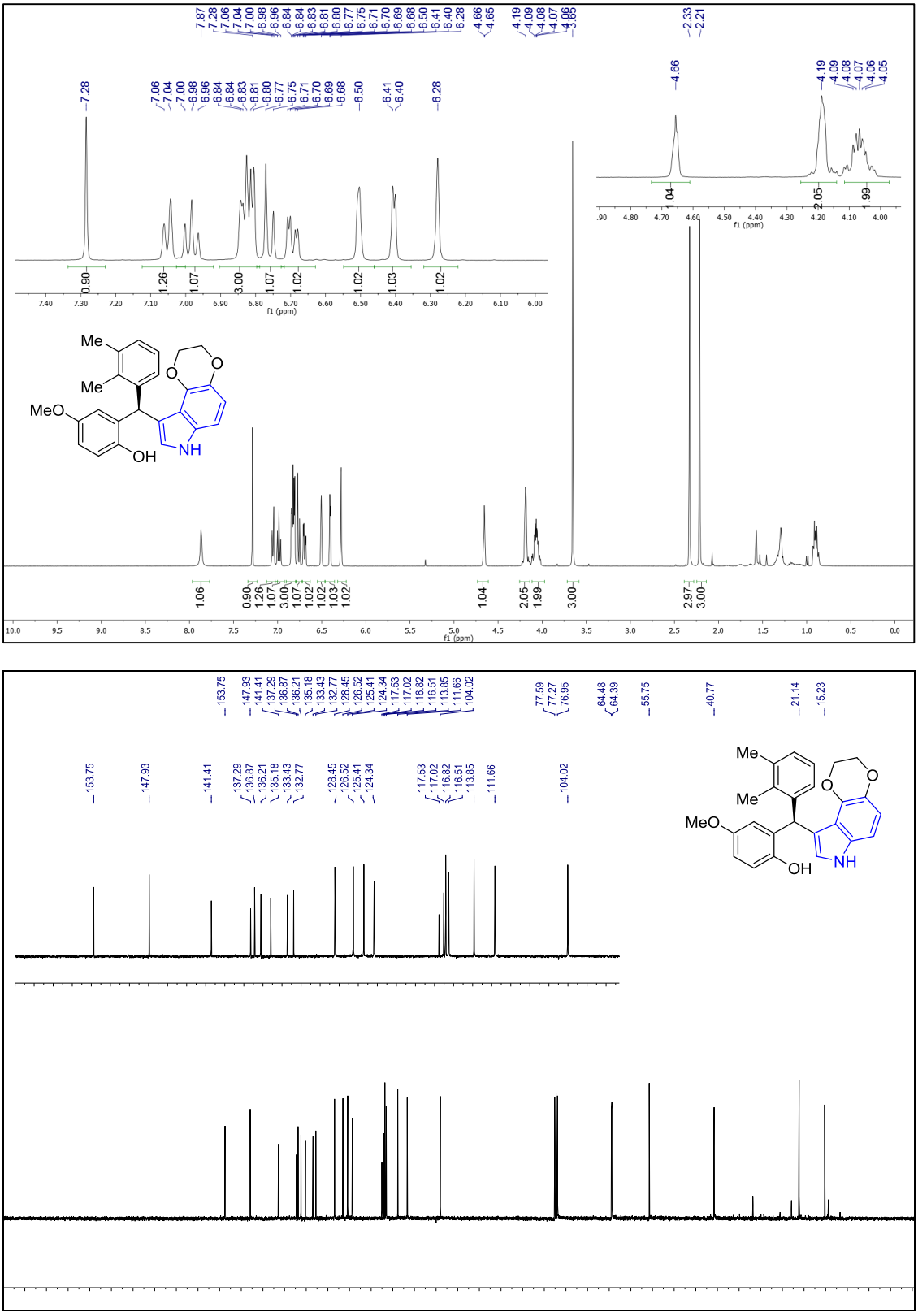


Figure 1.25. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-2-((2,3-dihydro-7H-[1,4]dioxino[2,3-e]indol-9-yl)(2,3-dimethylphenyl)methyl)-4-methoxyphenol (**10a**).

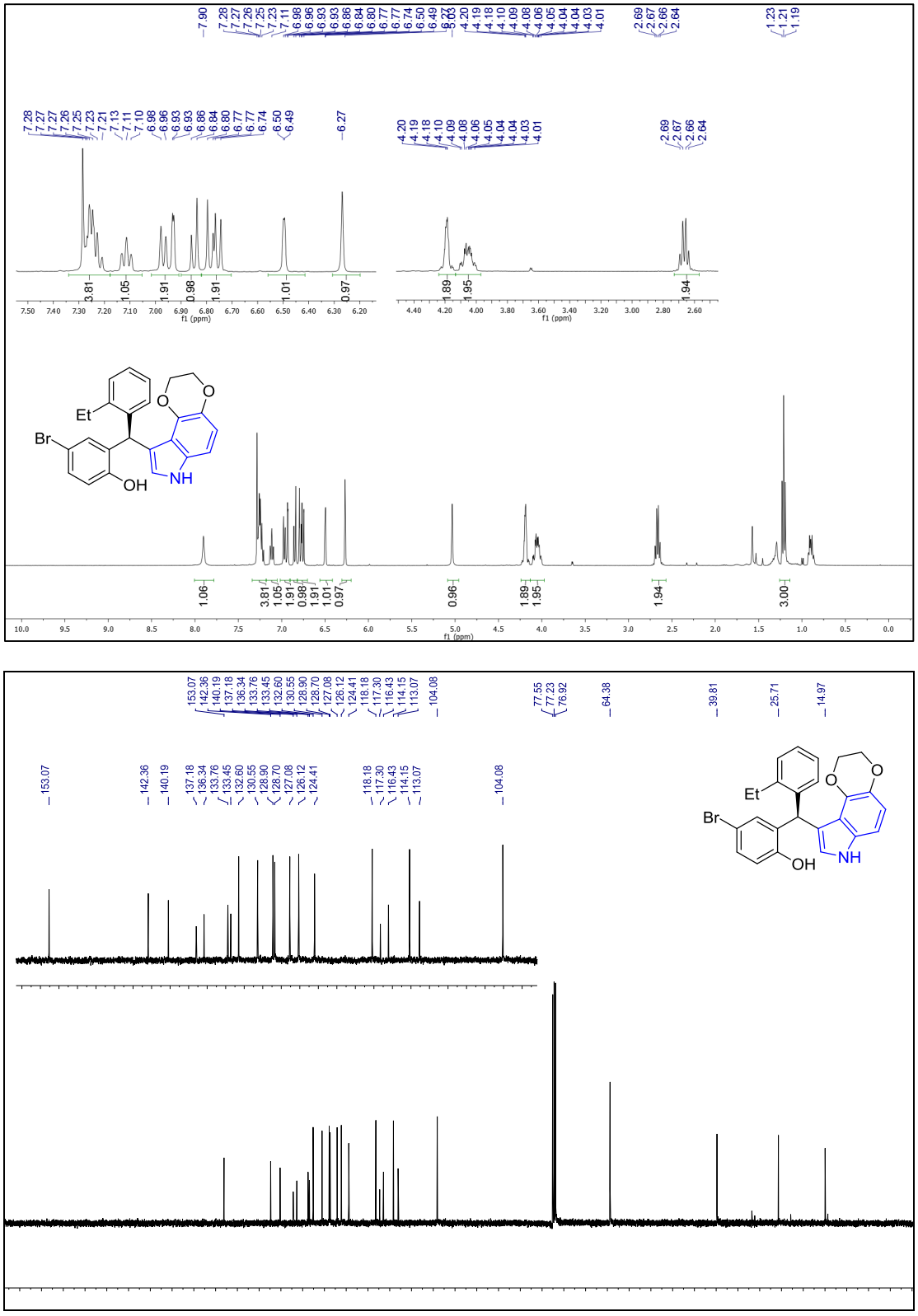


Figure 1.26. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-4-bromo-2-((2,3-dihydro-7H-[1,4]dioxino[2,3-e]indol-9-yl)(2-ethylphenyl)methyl)phenol (**10b**).

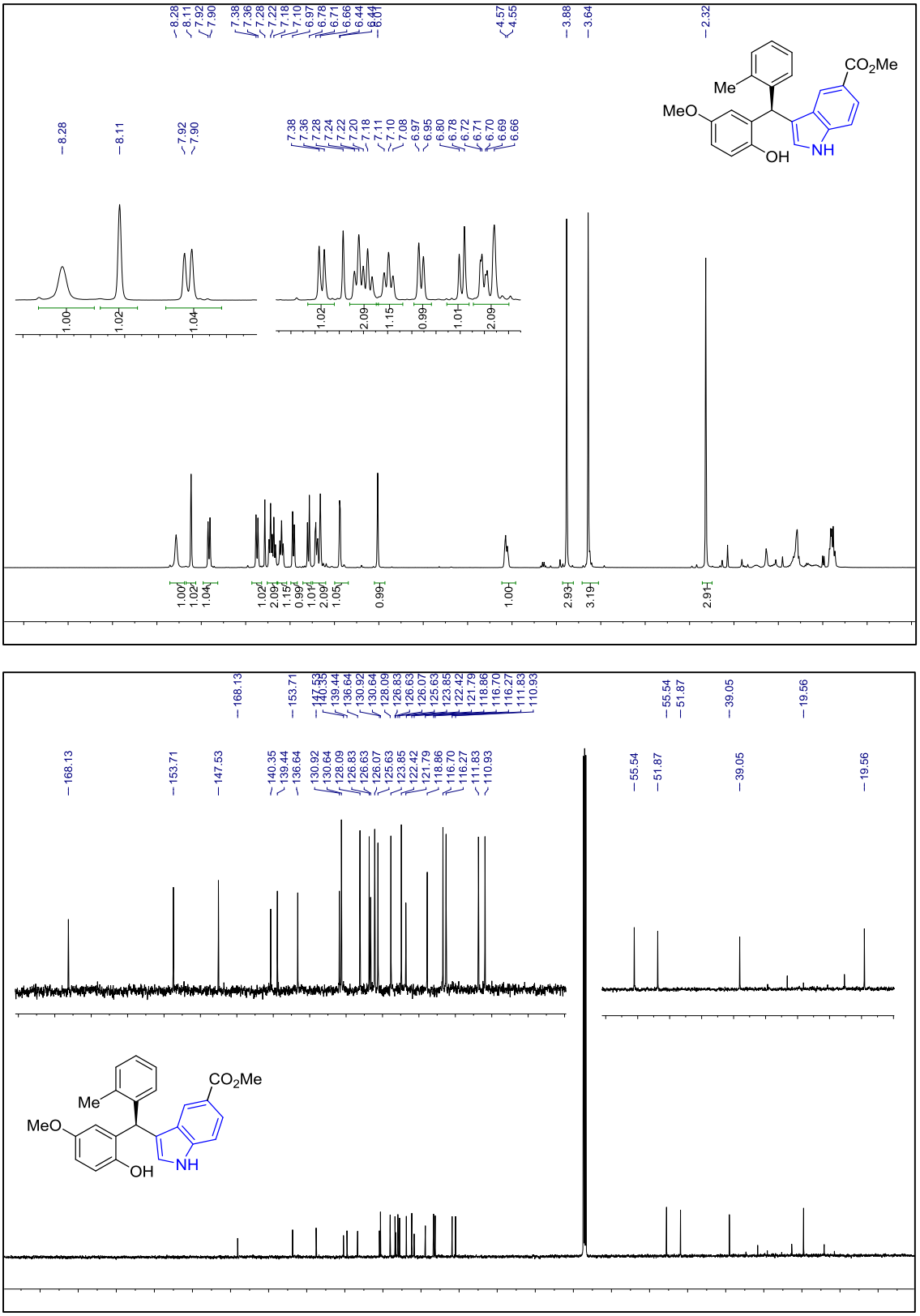


Figure 1.27. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR ofMethyl (S)-3-((2-hydroxy-5-methoxyphenyl)(*o*-tolyl)methyl)-1H-indole-5-carboxylate (**11a**).

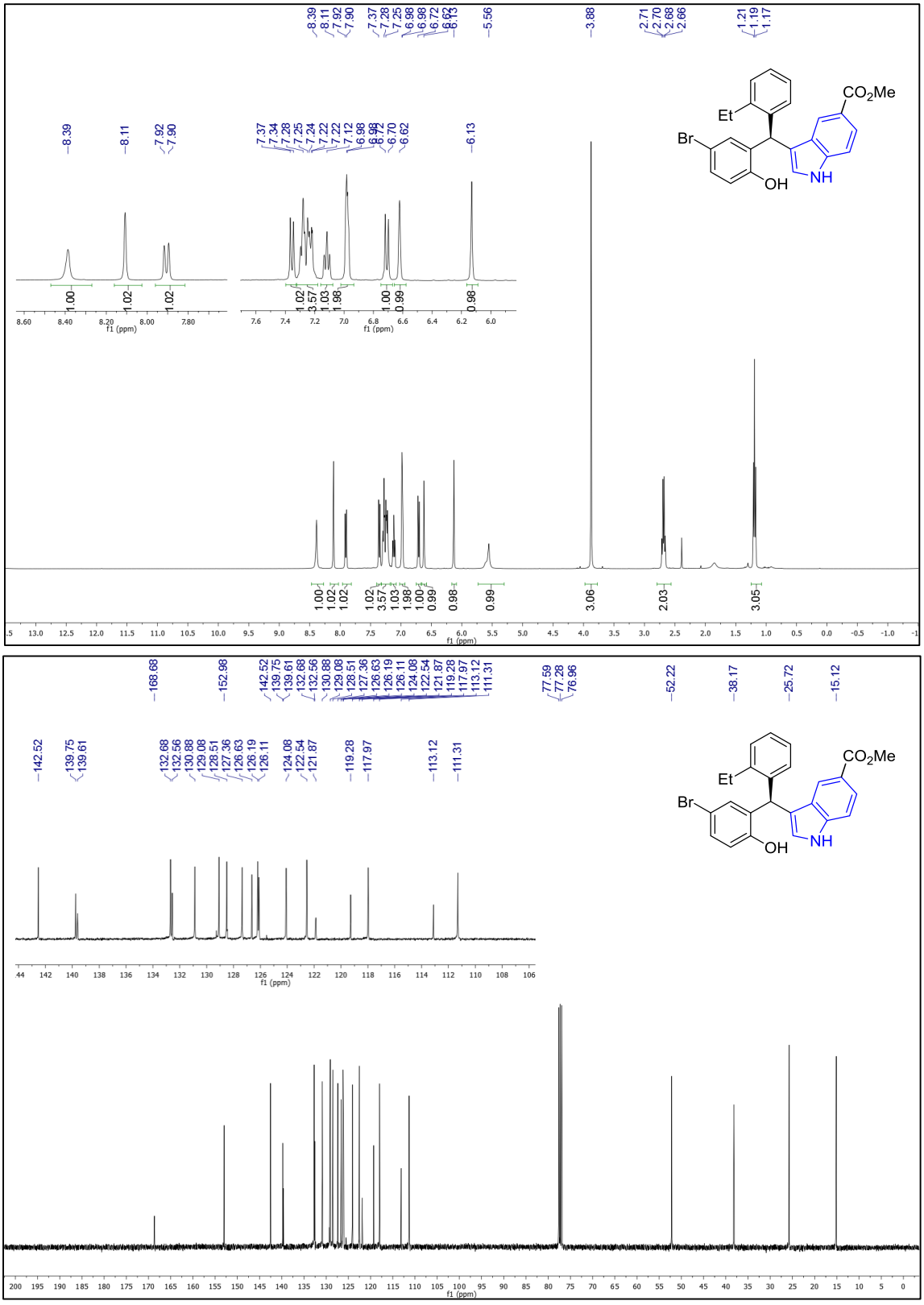
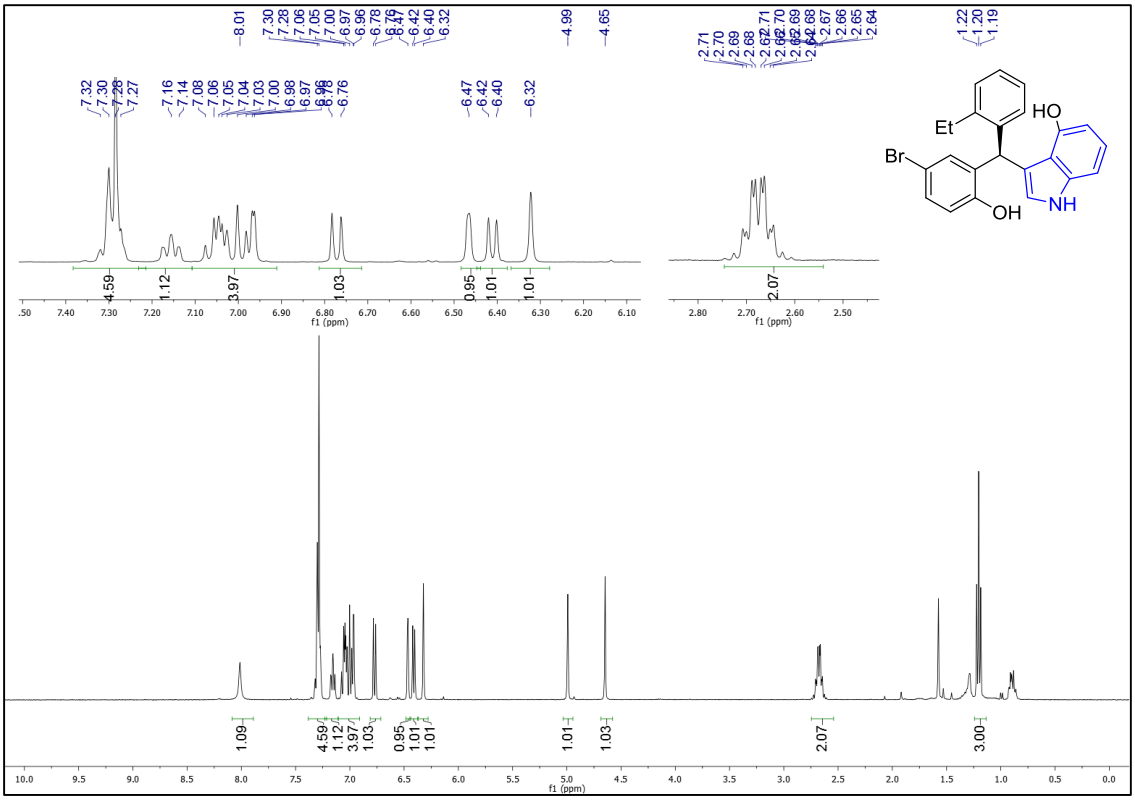


Figure 1.28. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR ofMethyl (S)-3-((5-bromo-2-hydroxyphenyl)(2-ethylphenyl)methyl)-1H-indole-5-carboxylate (**11b**).



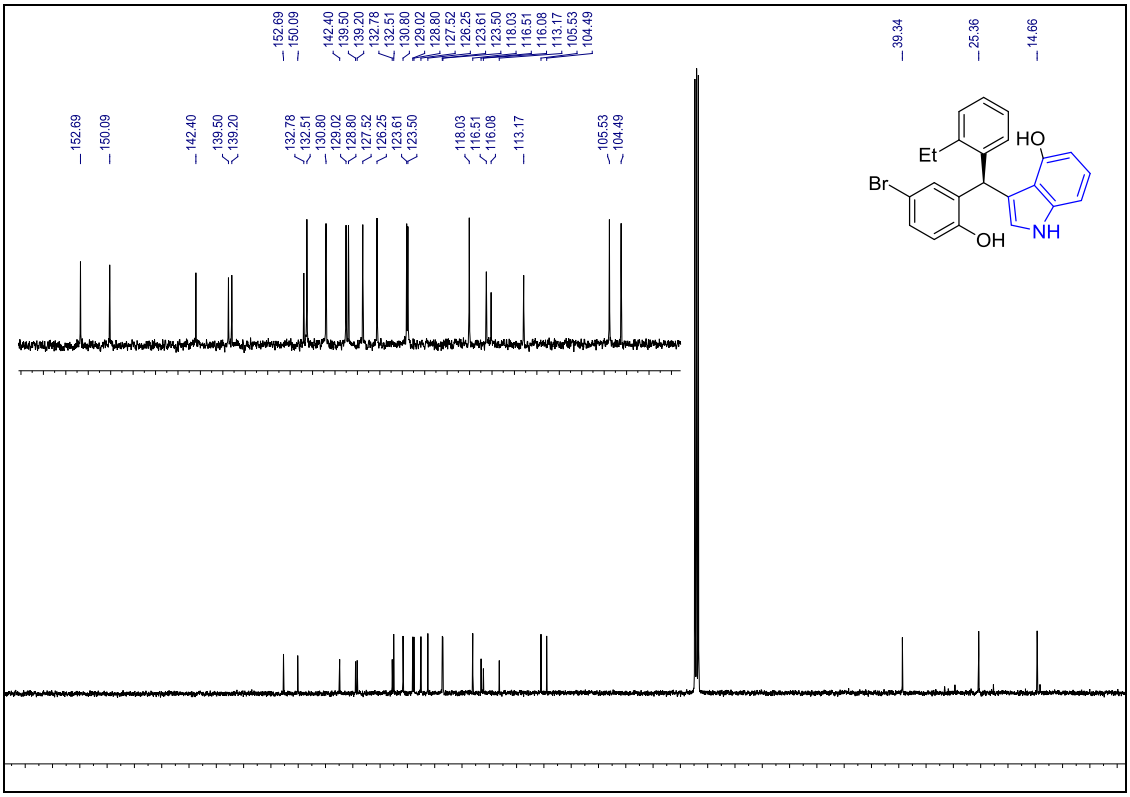


Figure 1.29. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-3-((5-bromo-2-hydroxyphenyl)(2-ethylphenyl)methyl)-1H-indol-4-ol (**12**).

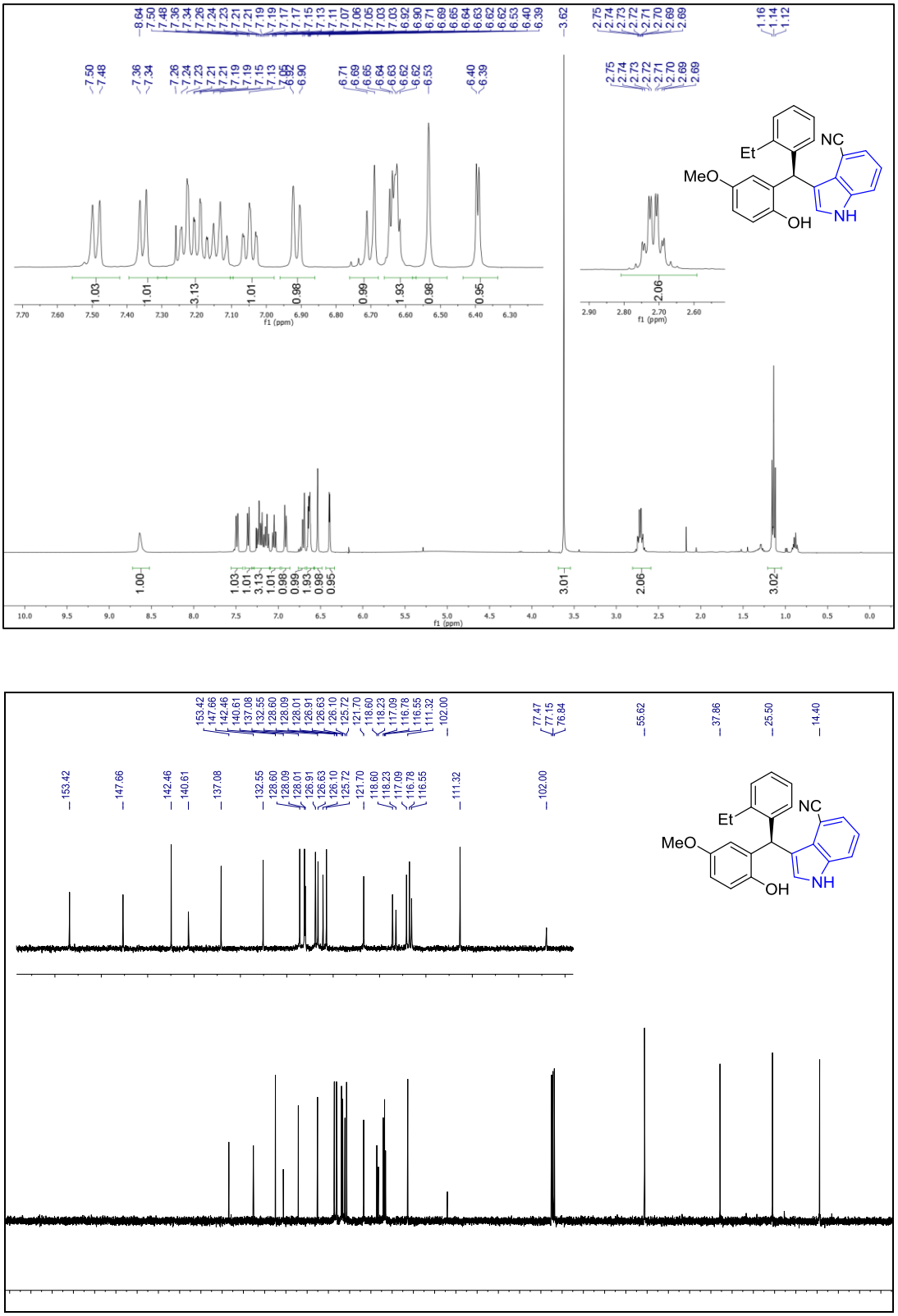


Figure 1.30. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-3-((2-ethylphenyl)(2-hydroxy-5-methoxyphenyl)methyl)-1H-indole-4-carbonitrile (**13a**).

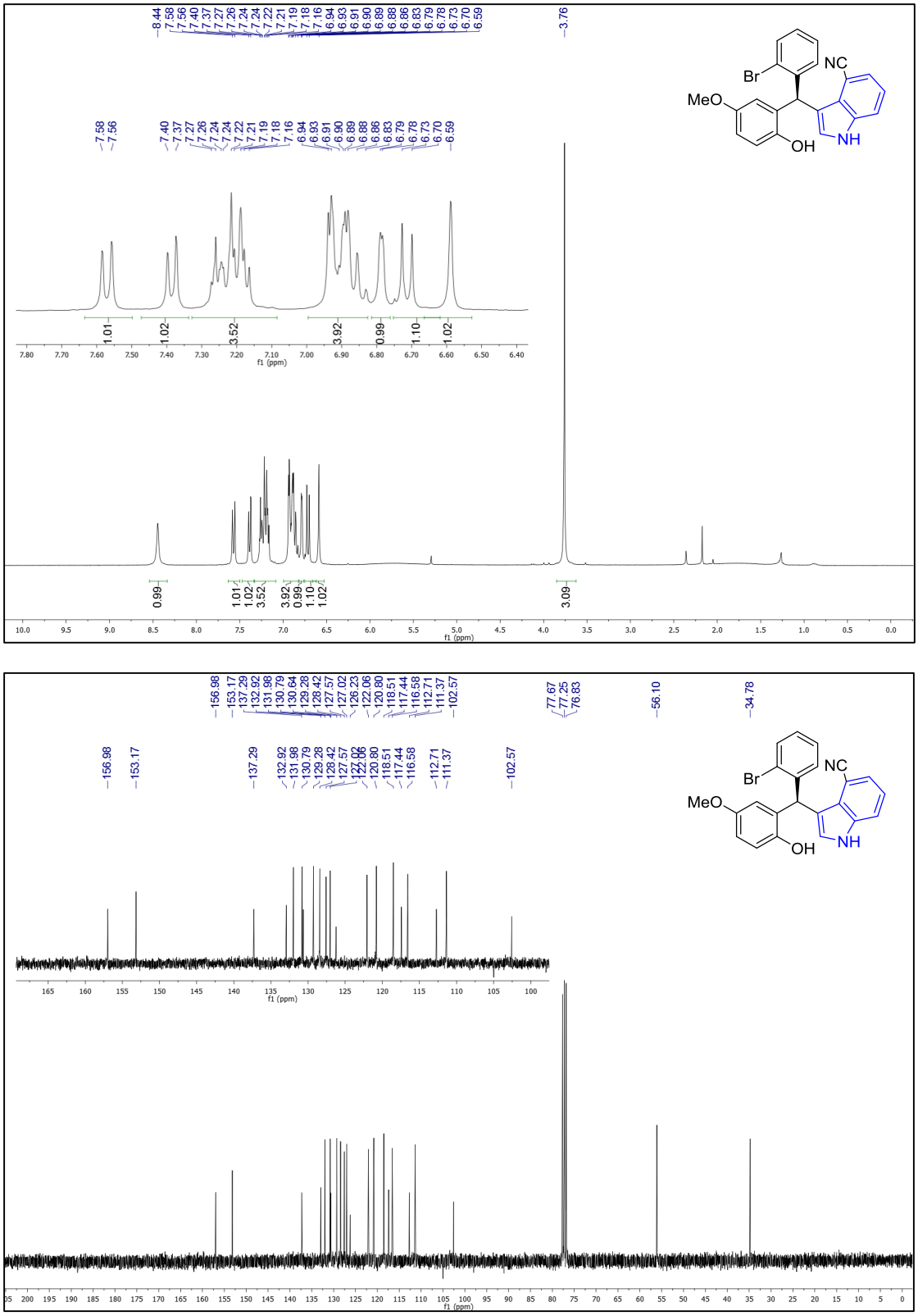


Figure 1.31. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-3-((2-bromophenyl)(2-hydroxy-5-methoxyphenyl)methyl)-1H-indole-4-carbonitrile (**13b**).

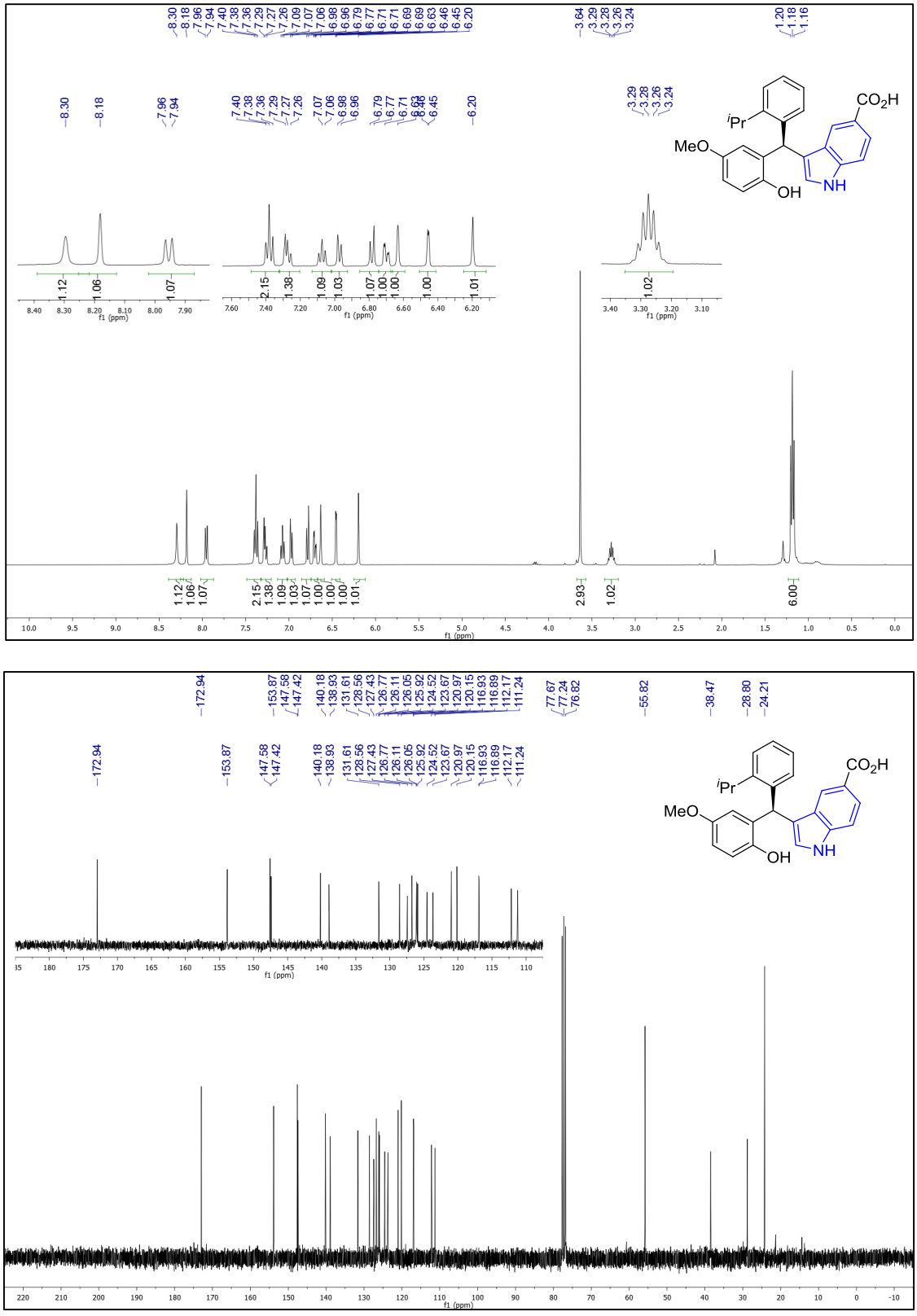


Figure 1.32. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-3-((2-hydroxy-5-methoxyphenyl)(2-isopropylphenyl)methyl)-1H-indole-5-carboxylic acid (**14**).

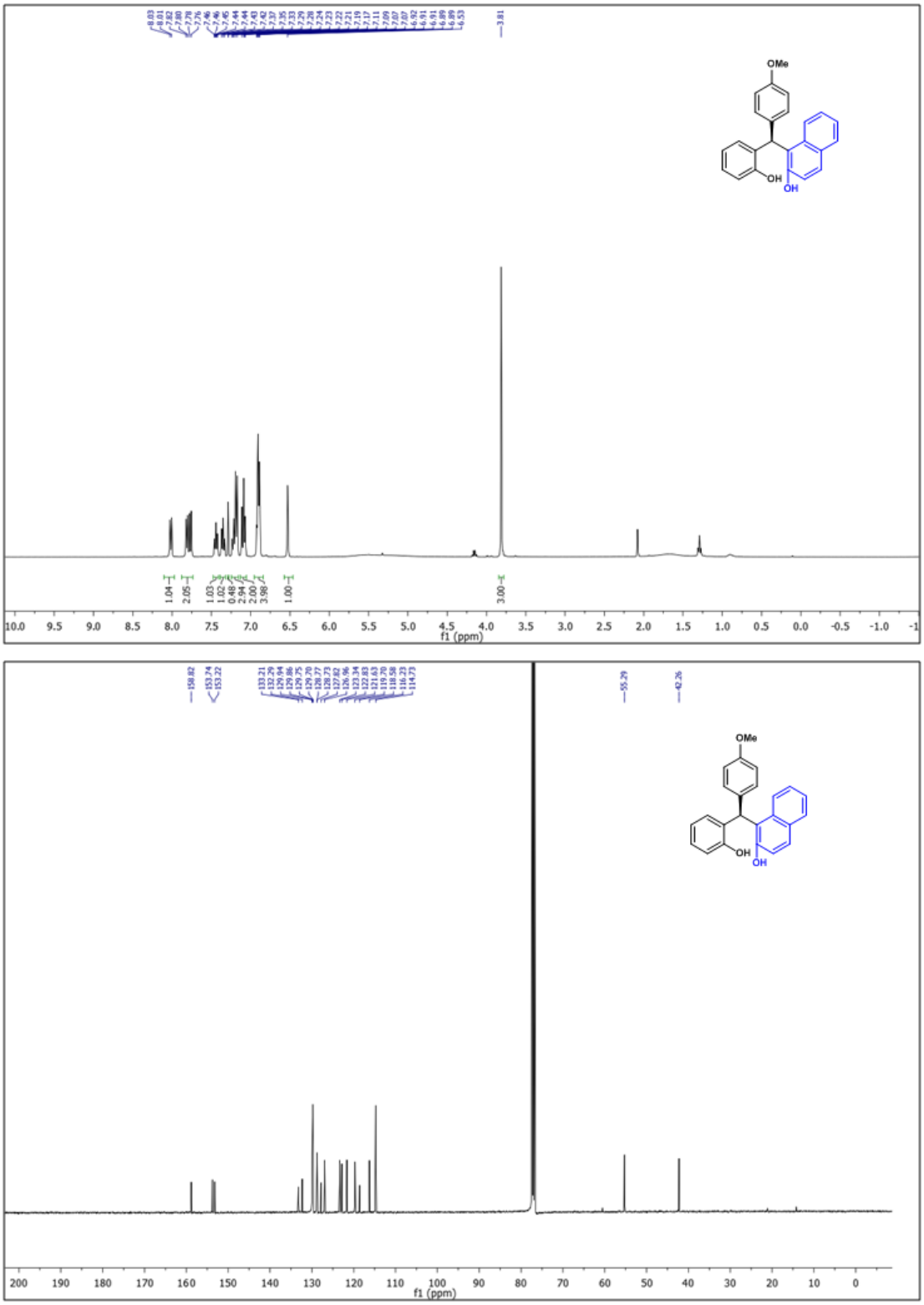


Figure 1.33. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of **(***R***)**-1-((2-hydroxyphenyl)(4-methoxyphenyl)methyl)naphthalen-2-ol (**17a**).

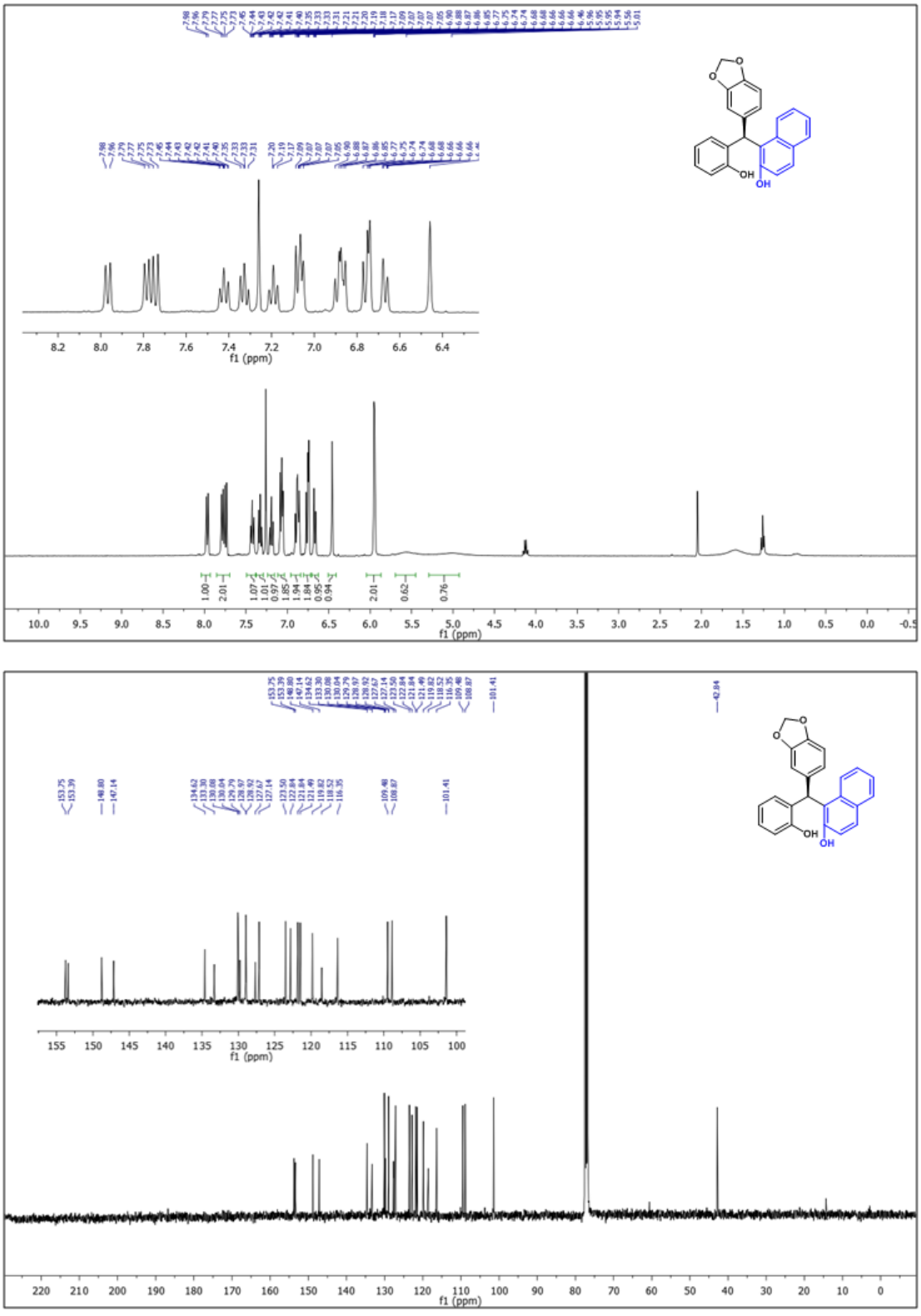


Figure 1.34. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of (*R*)-1-(benzo[d][1,3]dioxol-5-yl(2-hydroxyphenyl)methyl)naphthalen-2-ol (**17b**).

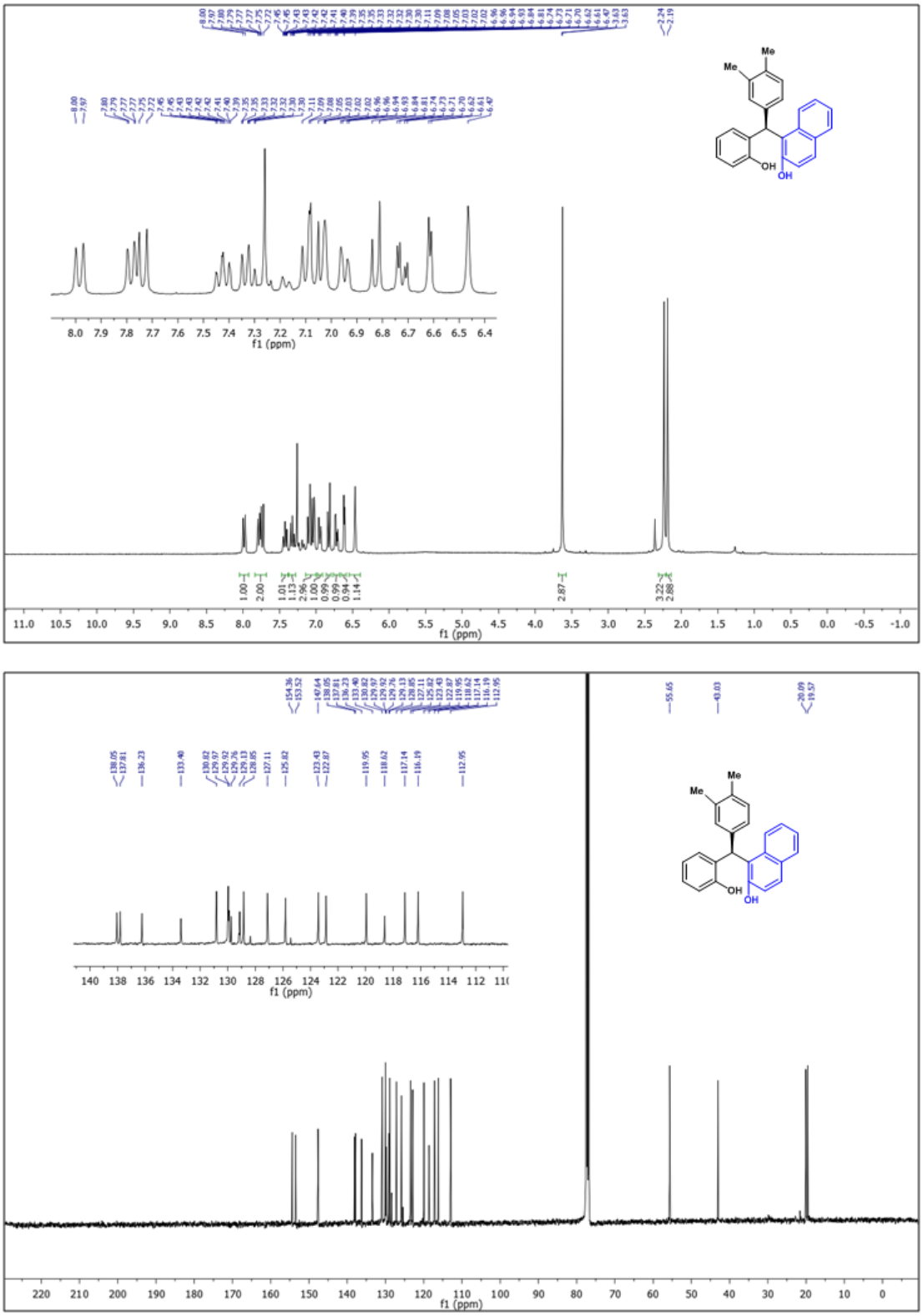


Figure 1.35. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of (*R*)-1-((3,4-dimethylphenyl)(2-hydroxy-5-methoxyphenyl)methyl)naphthalen-2-ol (**17c**).

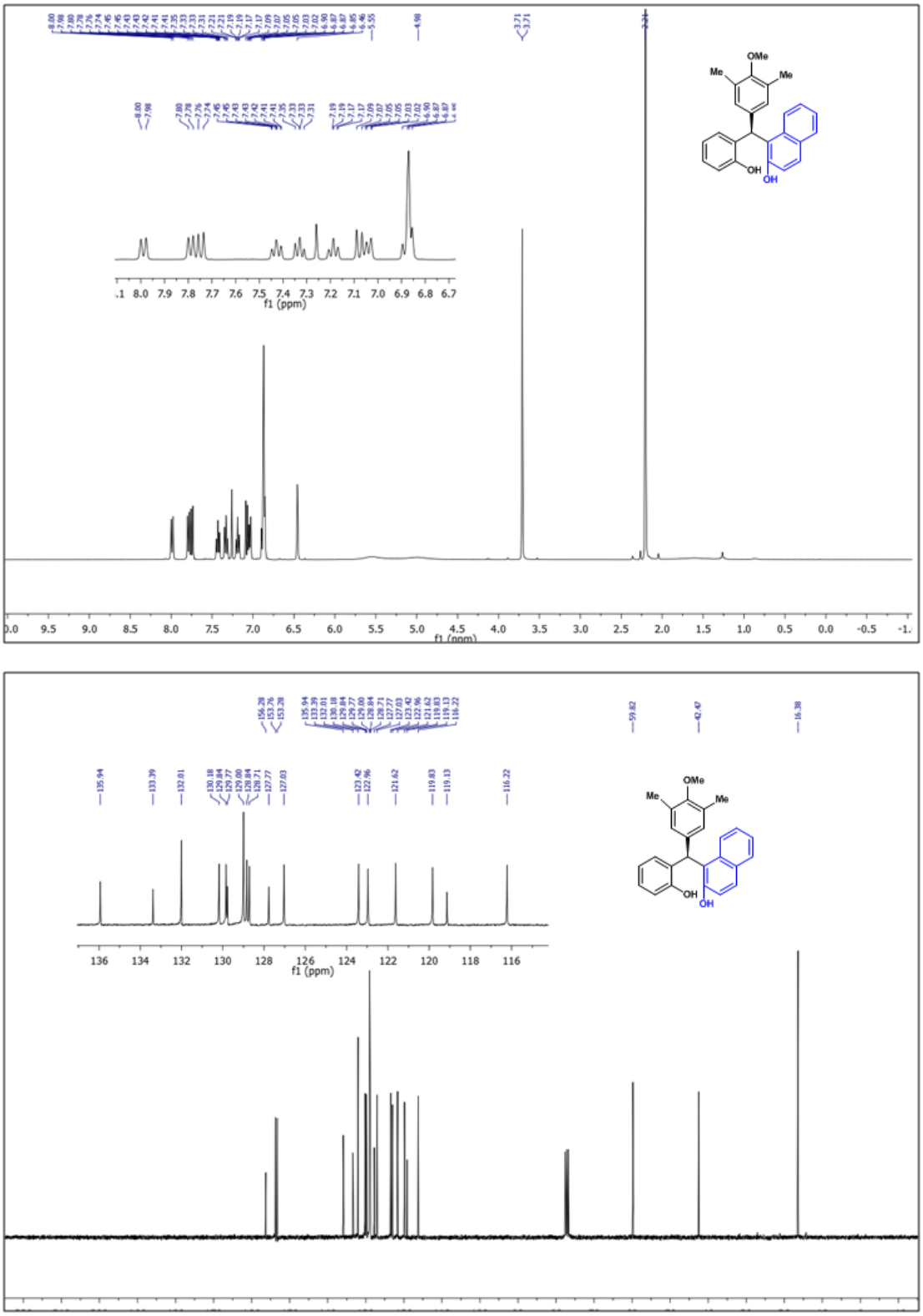


Figure 1.36. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of (*R*)-1-((2-hydroxyphenyl)(4-methoxy-3,5-dimethylphenyl)methyl)naphthalen-2-ol (**17d**).

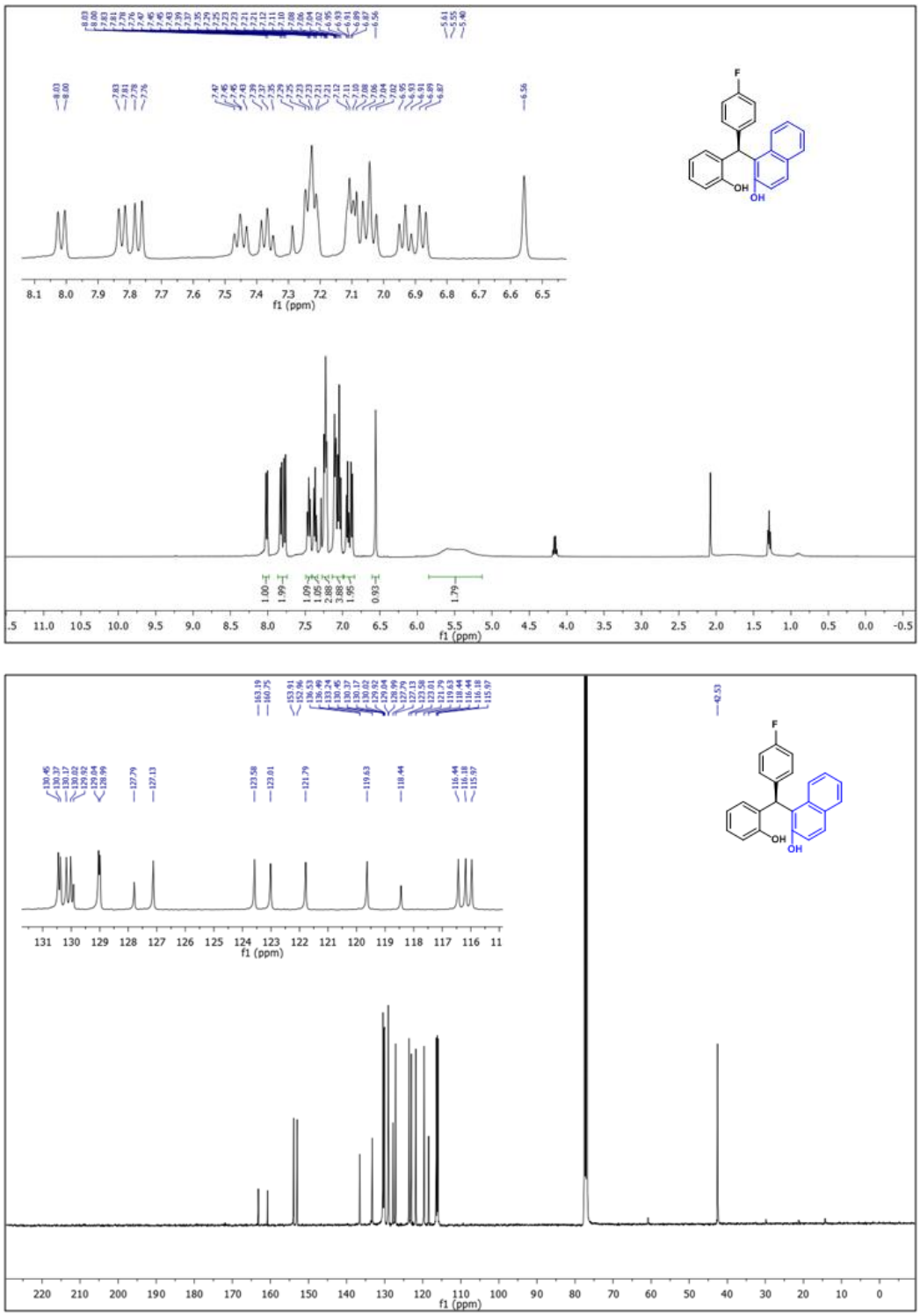


Figure 1.37. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of (*R*)-1-((4-fluorophenyl)(2-hydroxyphenyl)methyl)naphthalen-2-ol (**17e**).

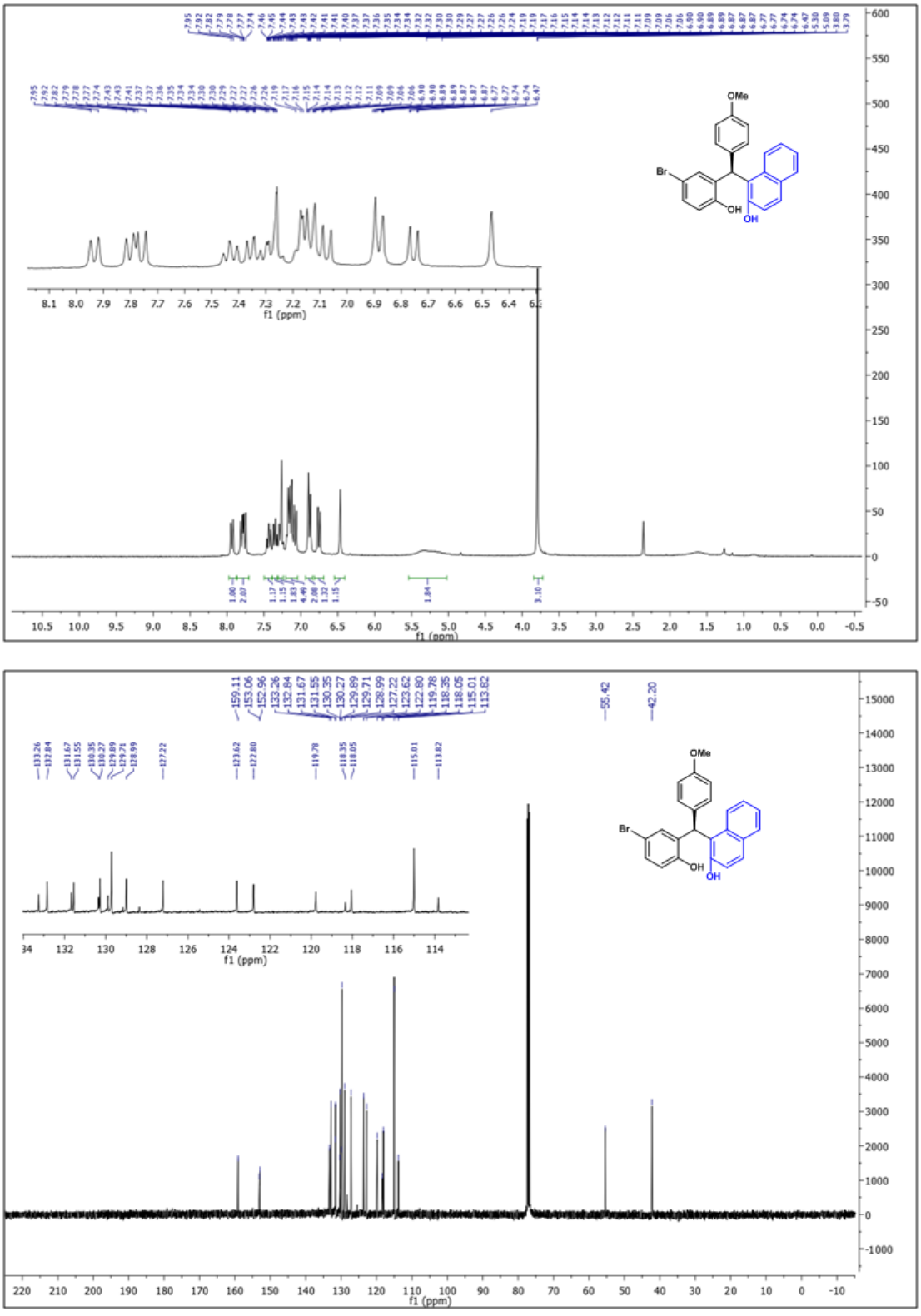


Figure 1.38. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of (*R*)-1-((5-bromo-2-hydroxyphenyl)(4-methoxyphenyl)methyl)naphthalen-2-ol (**17f**).

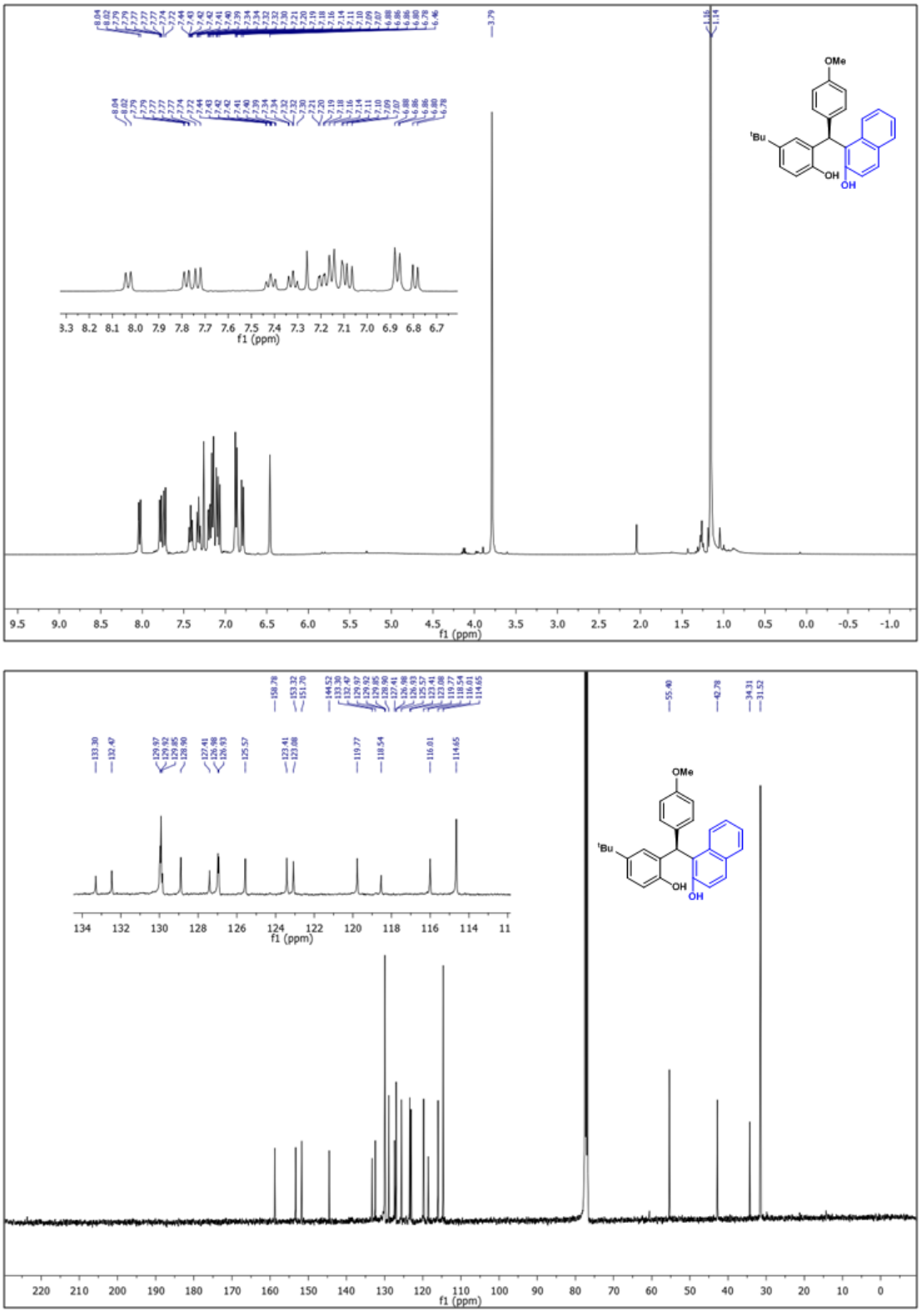


Figure 1.39. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of (*R*)-1-((5-(tert-butyl)-2-hydroxyphenyl)(4-methoxyphenyl)methyl)naphthalen-2-ol (**17g**).

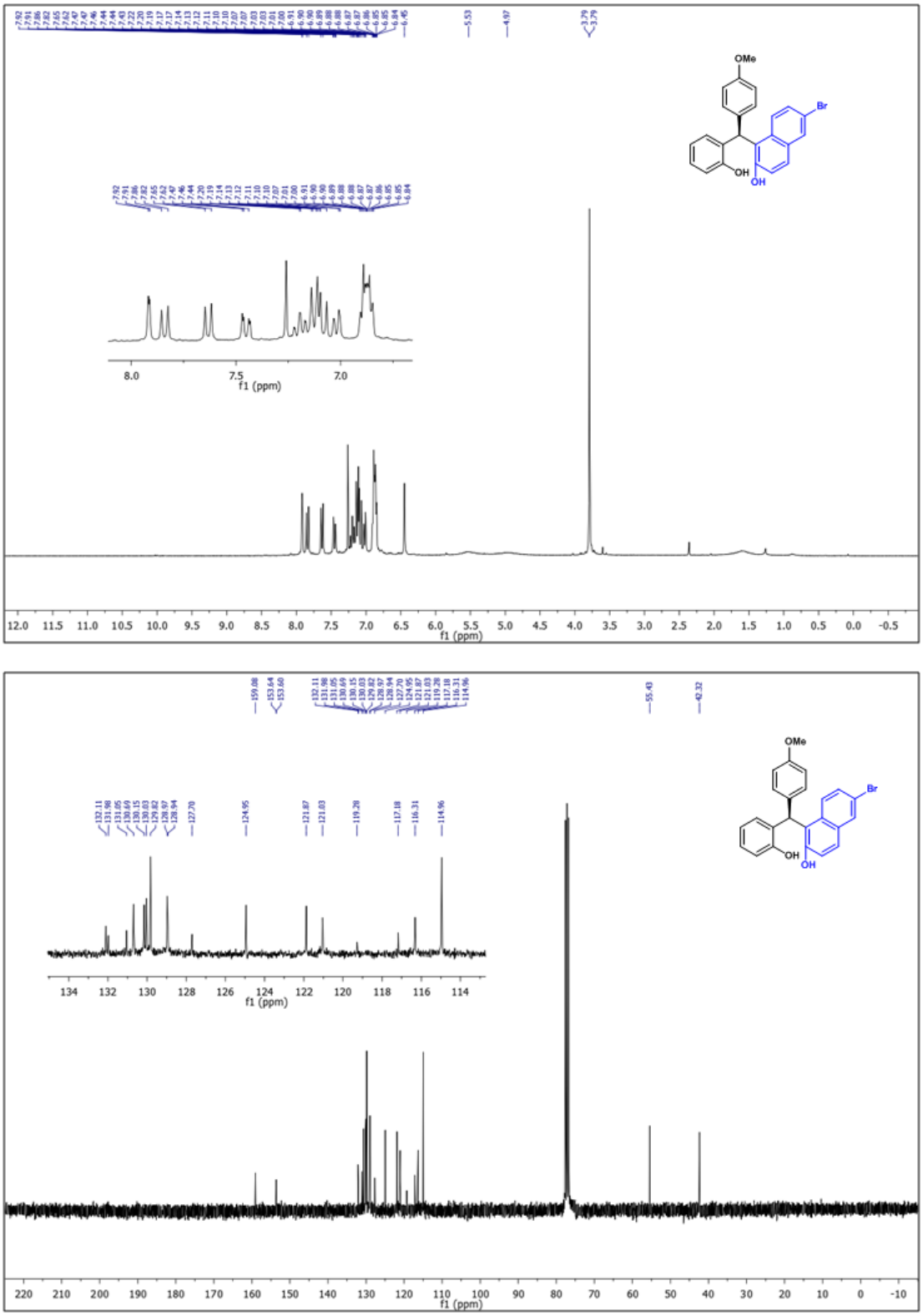


Figure 1.40. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of (*R*)-6-bromo-1-((2-hydroxyphenyl)(4-methoxyphenyl)methyl)naphthalen-2-ol (**17h**).

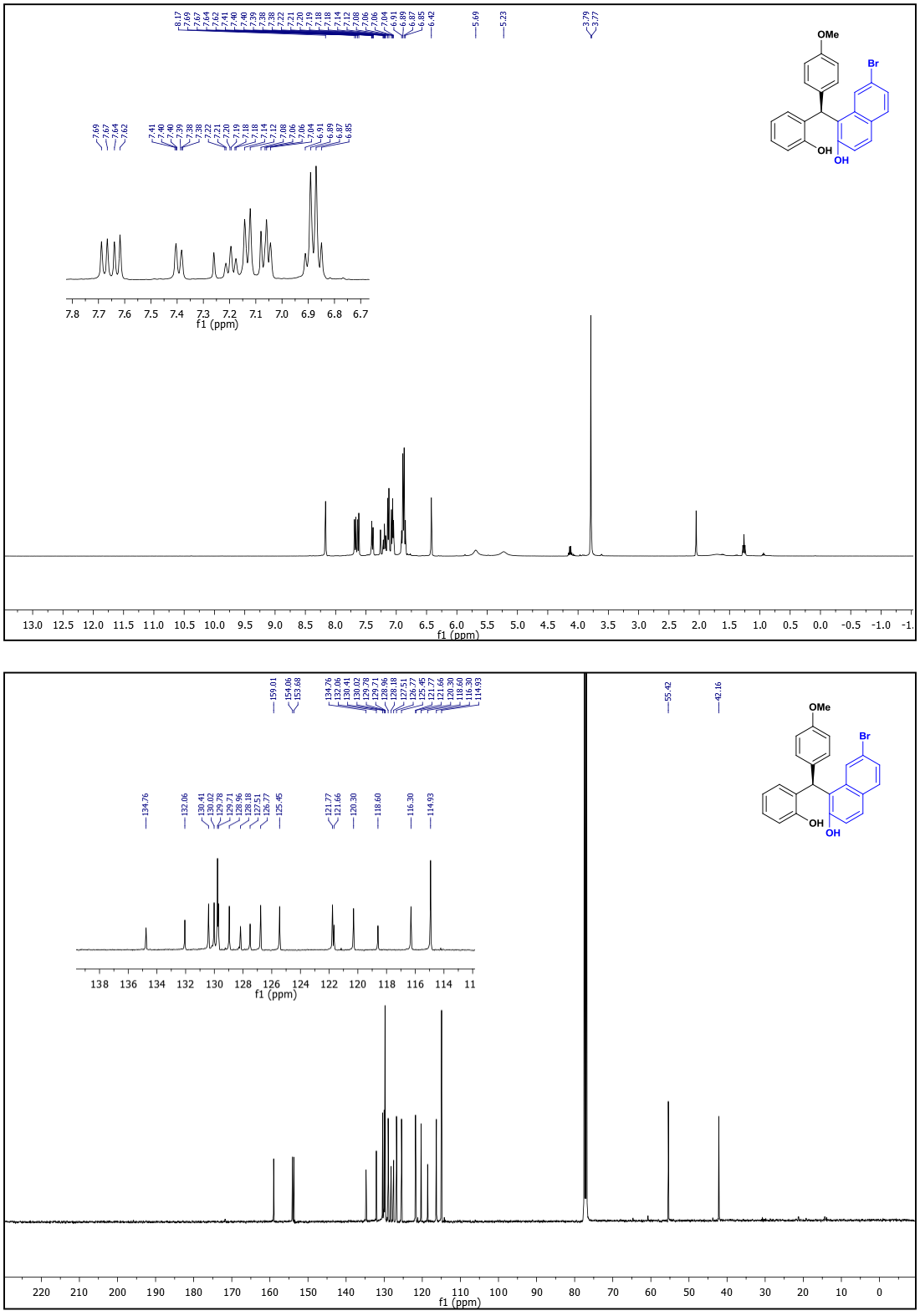


Figure 1.41. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of (*R*)-7-bromo-1-((2-hydroxyphenyl)(4-methoxyphenyl)methyl)naphthalen-2-ol (**17i**).

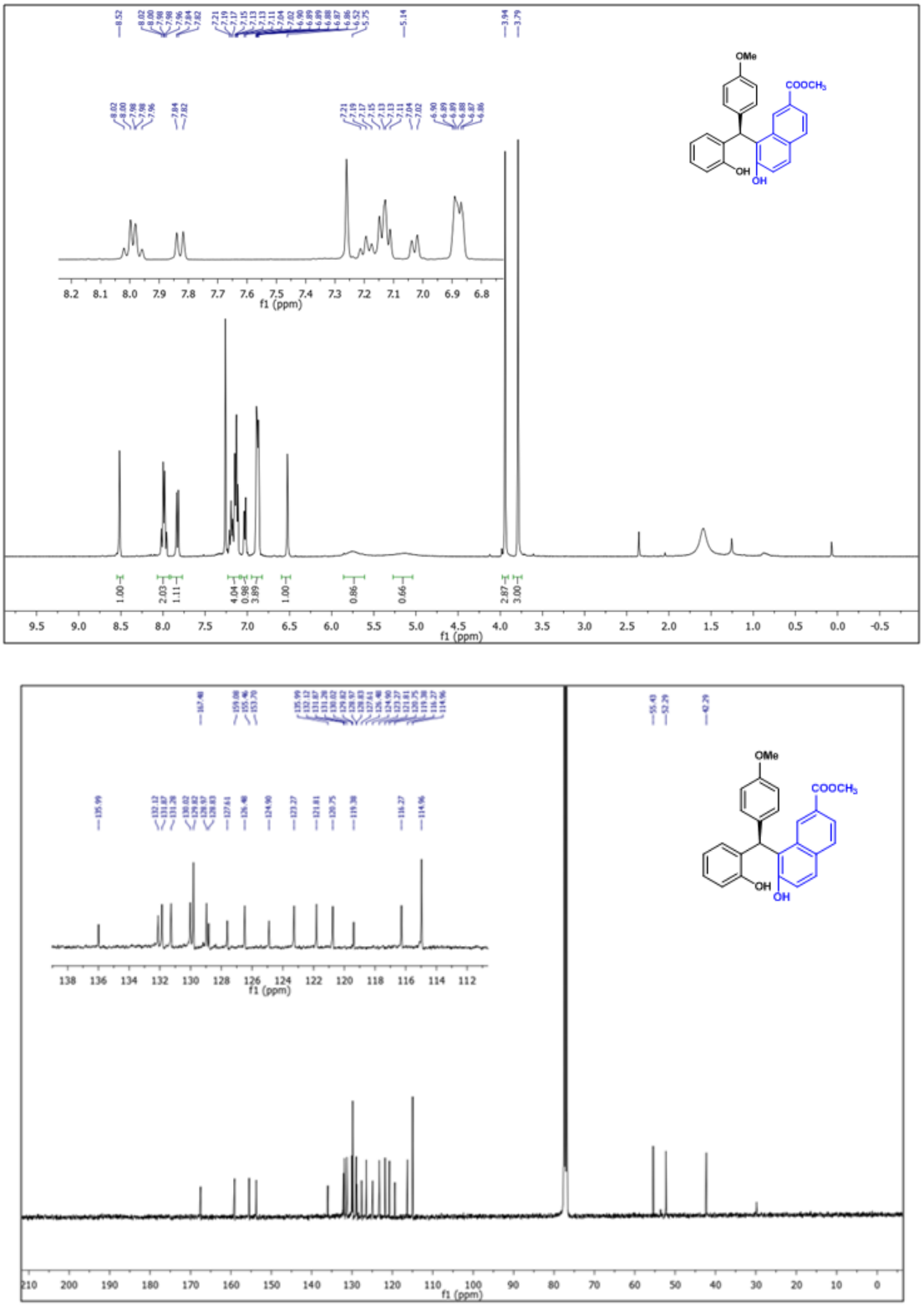


Figure 1.42. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of (*R*)-methyl 7-hydroxy-8-((2-hydroxyphenyl)(4-methoxyphenyl)methyl)-2-naphthoate (**17j**).

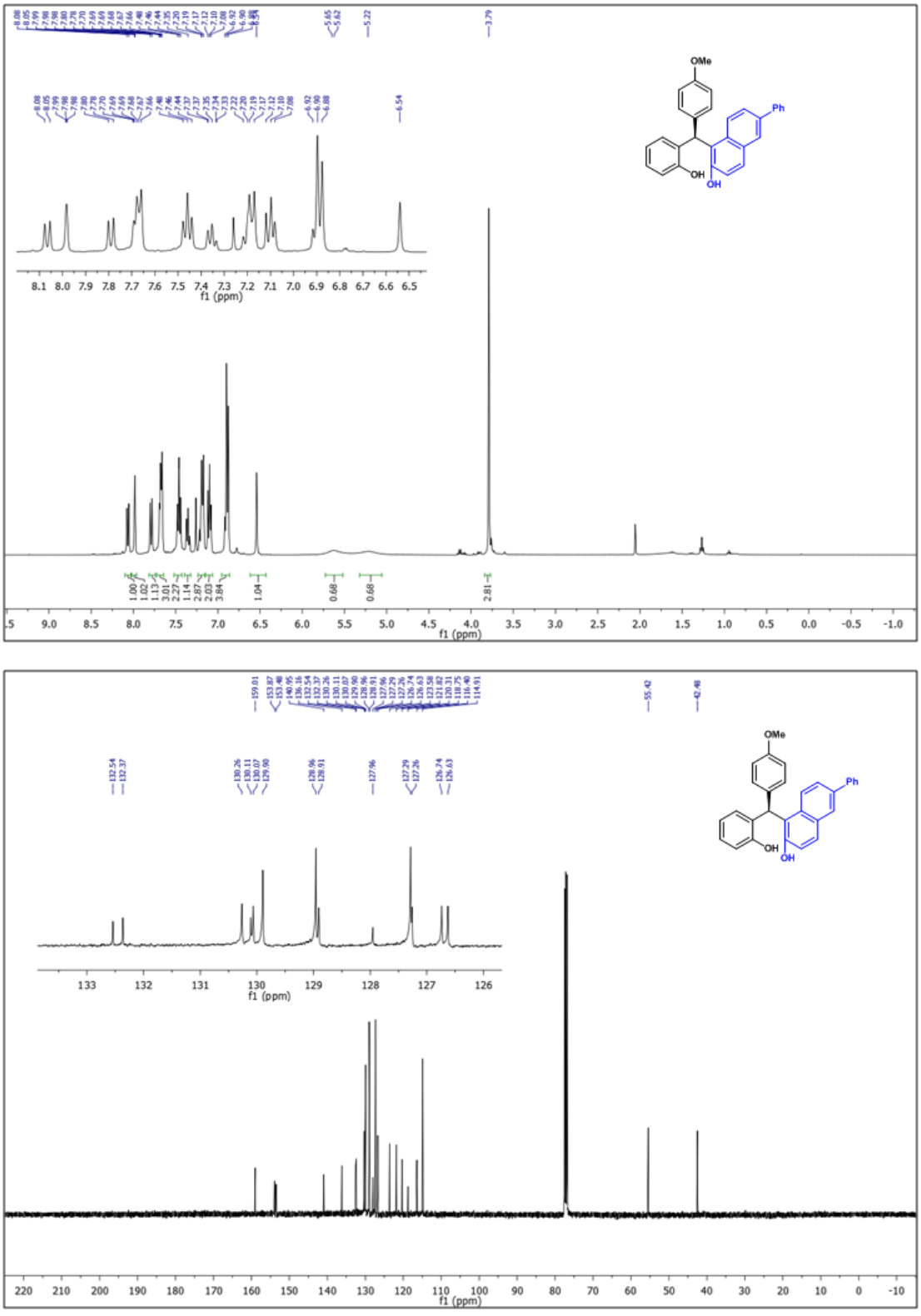


Figure 1.43. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of (*R*)-1-((2-hydroxyphenyl)(4-methoxyphenyl)methyl)-6-phenylnaphthalen-2-ol (**17k**).

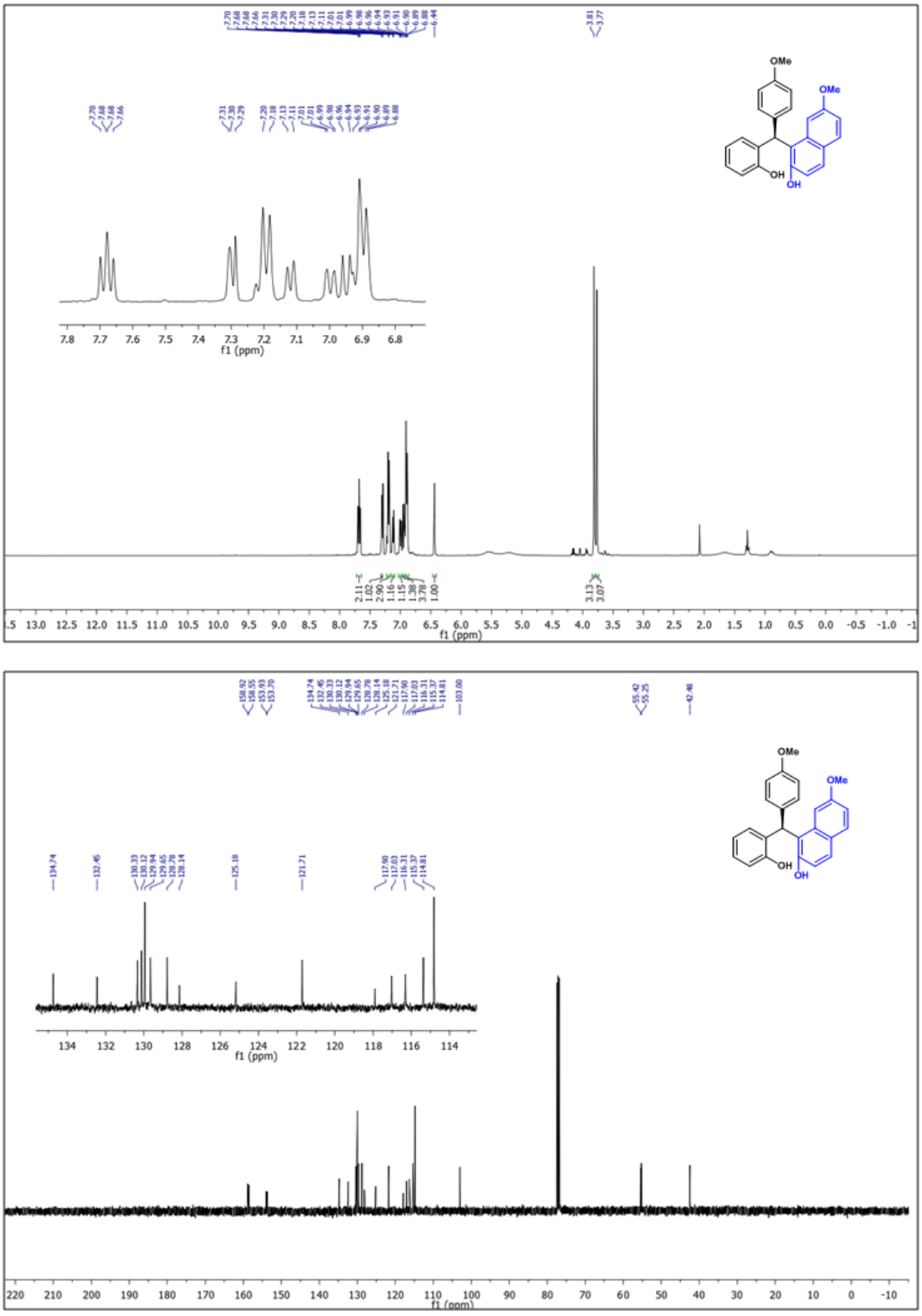


Figure 1.44. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of (*R*)-1-((2-hydroxyphenyl)(4-methoxyphenyl)methyl)-7-methoxynaphthalen-2-ol (**17l**).

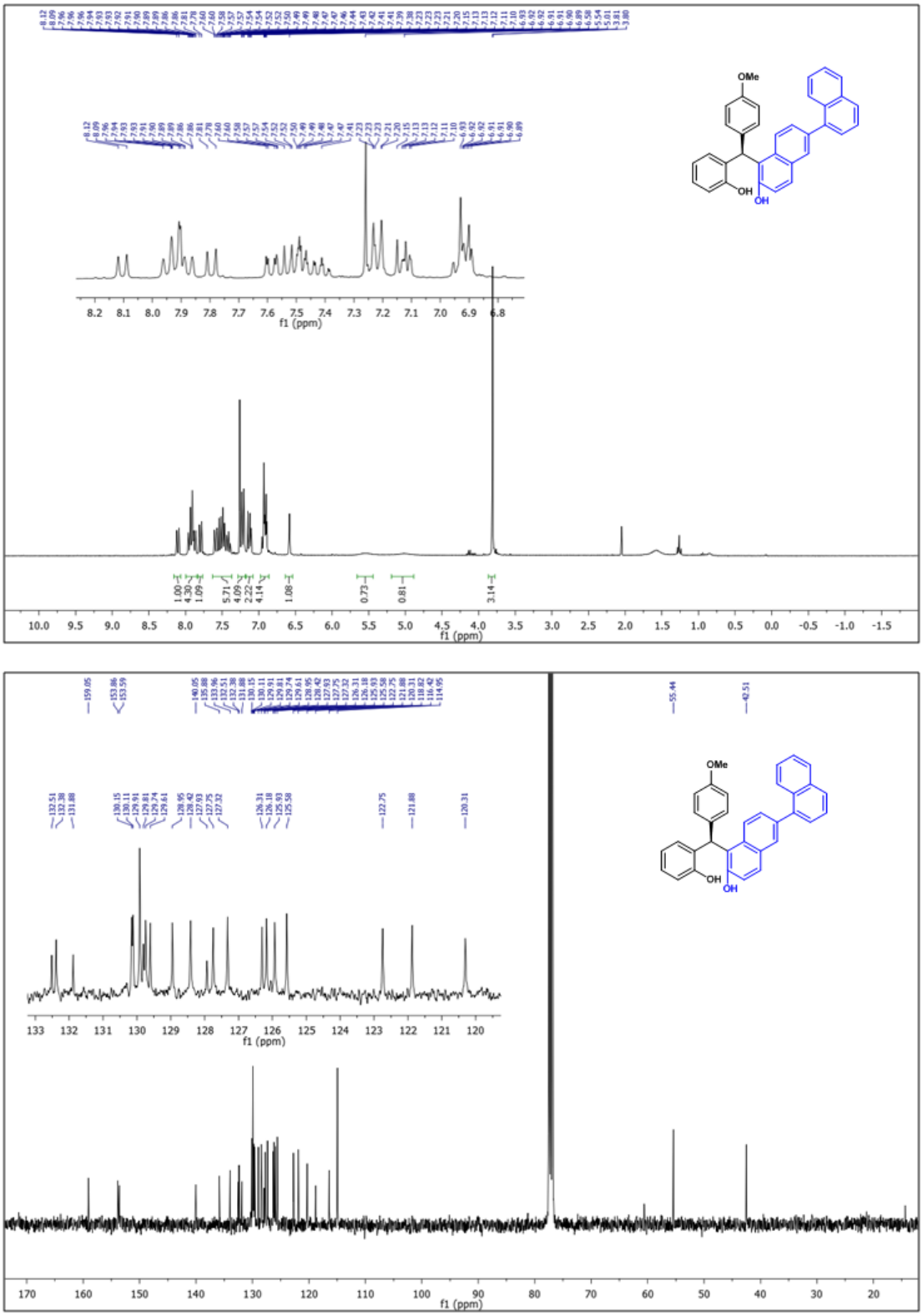


Figure 1.45. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of (*R*)-5'-((2-hydroxyphenyl)(4-methoxyphenyl)methyl)-[1,2'-binaphthalen]-6'-ol (**17m**).

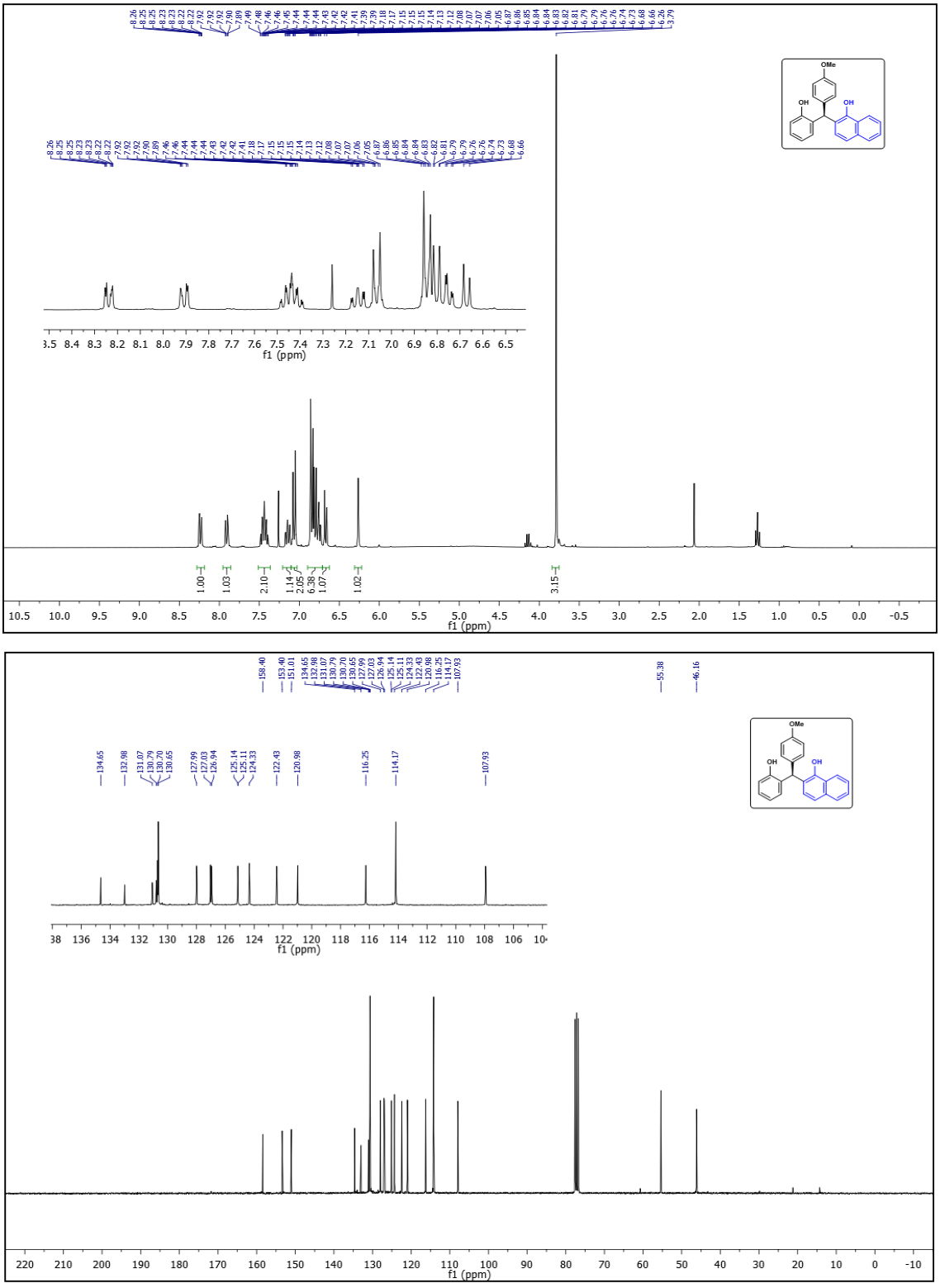


Figure 1.46. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of (*R*)-2-((2-hydroxyphenyl)(4-methoxyphenyl)methyl)naphthalen-1-ol (**18)**.

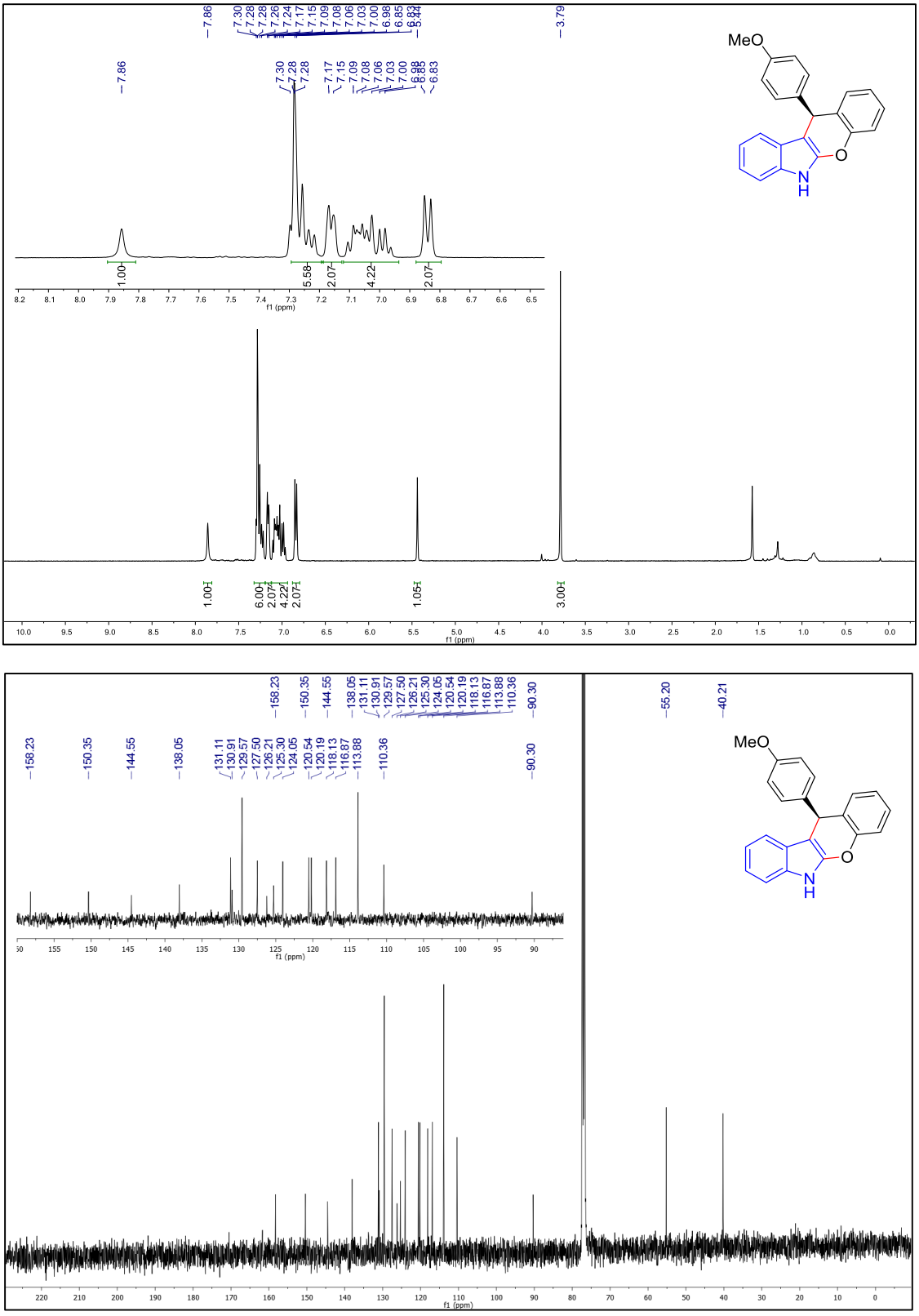


Figure 1.47. ^{1}H (400 MHz, $CDCl_3$) and ^{13}C (100 MHz, $CDCl_3$) NMR of(S)-11-(4-methoxyphenyl)-6,11-dihydrochromeno[2,3-b]indole (**19a**).

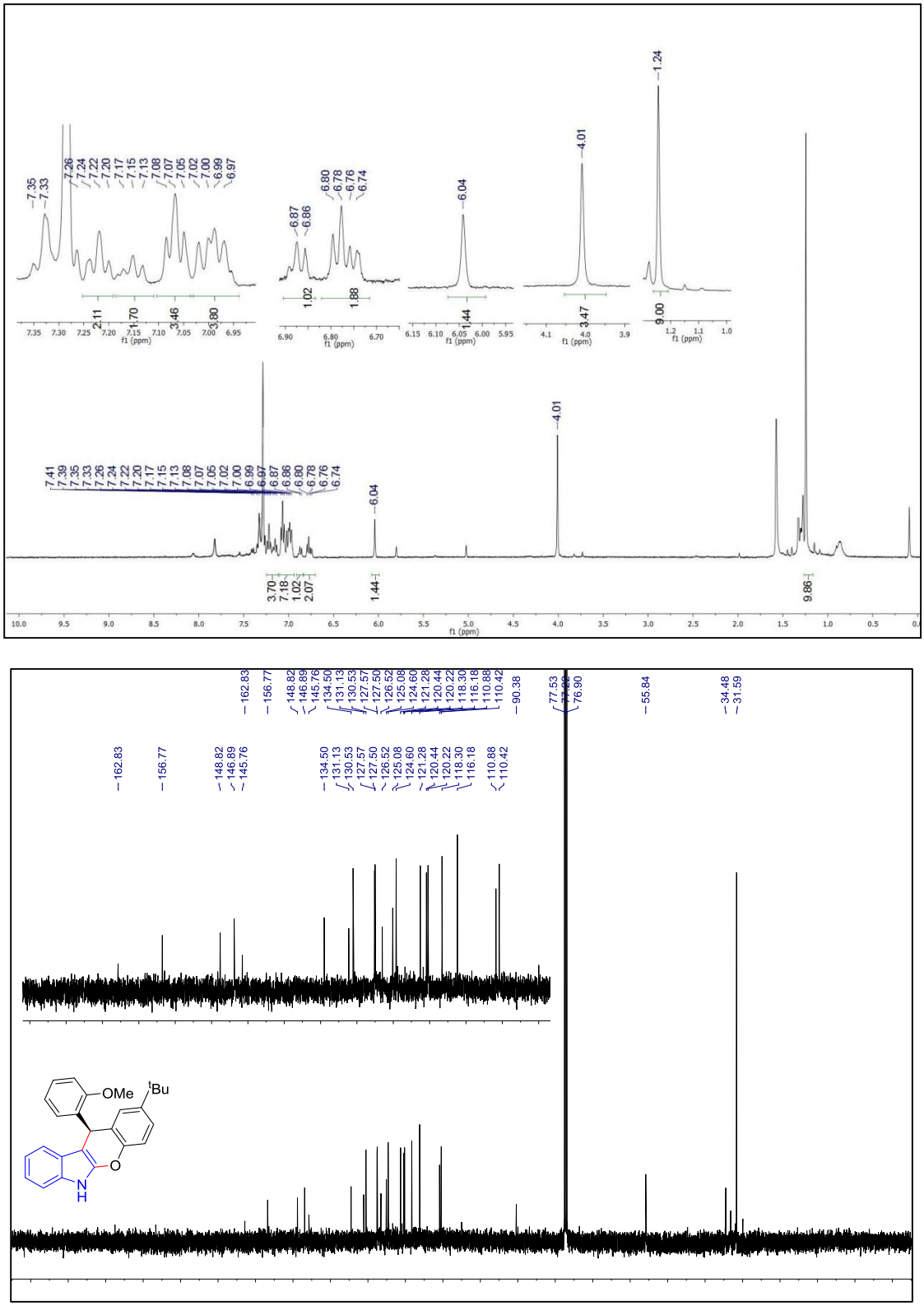
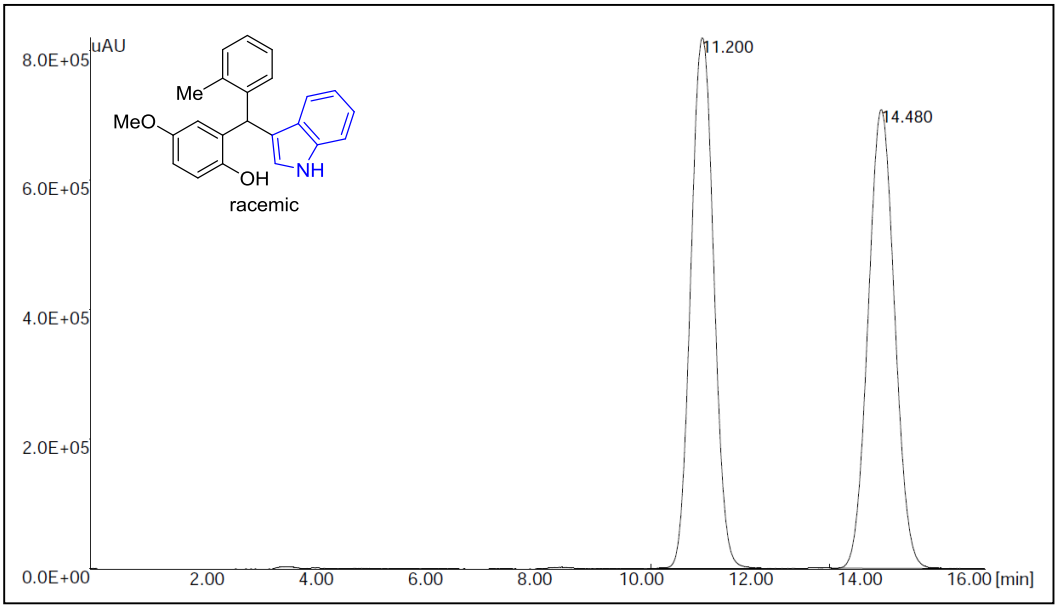


Figure 1.48. ^{1}H (300 MHz, $CDCl_3$) and ^{13}C (75 MHz, $CDCl_3$) NMR of(S)-2-(*tert*-butyl)-11-(2-methoxyphenyl)-6,11-dihydrochromeno[2,3-b]indole (**19b**).

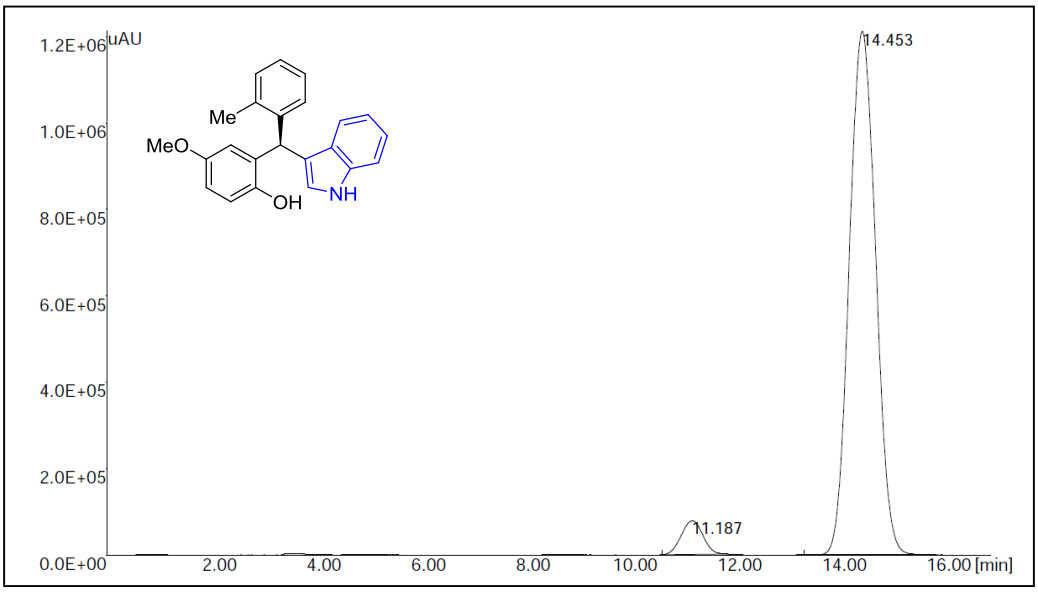
HPLC Profiles Of Racemic AndChiral 3-Substituted Indoles and Substituted Napthols (4-14 and 17-19)

(Table 2, entry 1)AD-H column, 80:20 (Hex:IPA), 1 mL/min, 232 nm



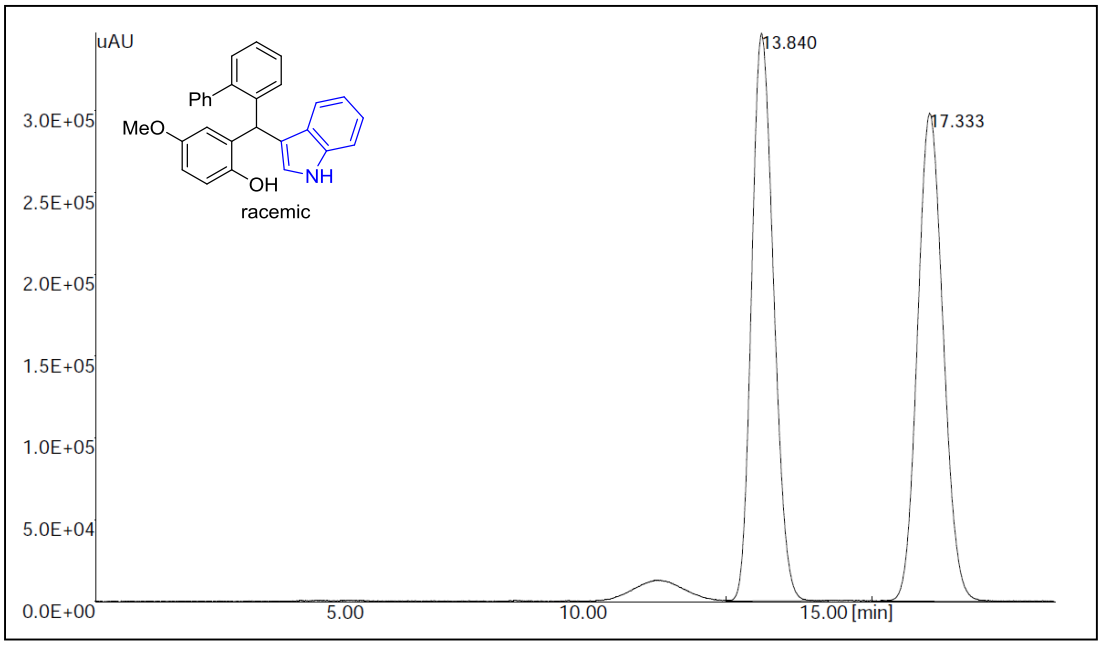
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		11.200	820049	24003427.158	50.07
2		14.480	708882	23939512.980	49.93

AD-H column, 80:20 (Hex:IPA), 1 mL/min, 232 nm(er = 95:5)



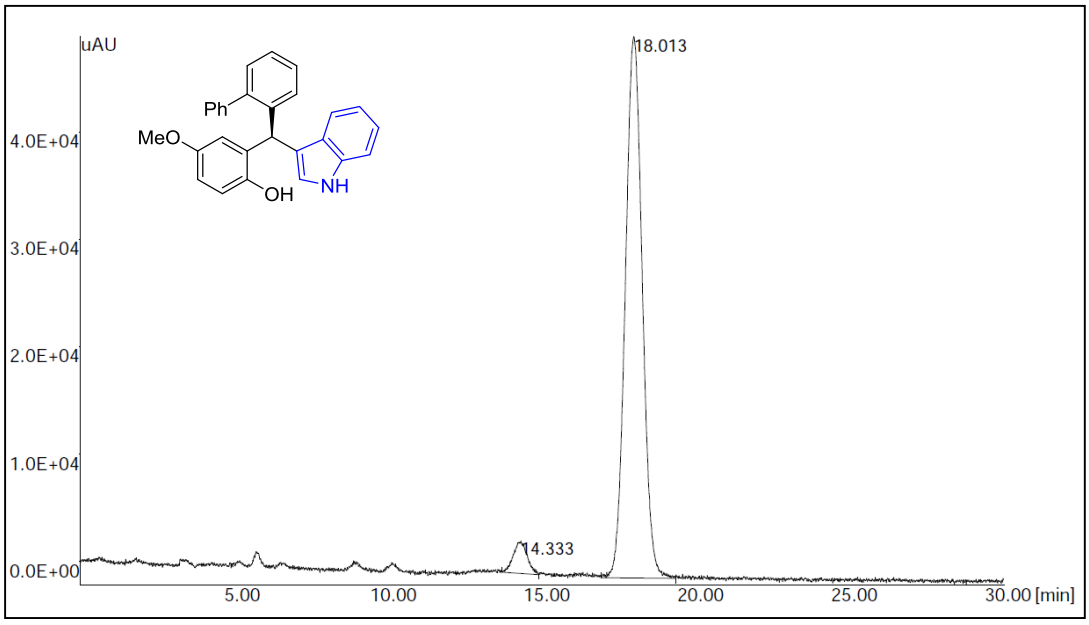
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		11.187	77937	2238985.633	5.01
2		14.453	1213149	42487957.066	94.99

(Table 2, entry 2)AD-H column, 80:20 (Hex:IPA), 1 mL/min, 288 nm



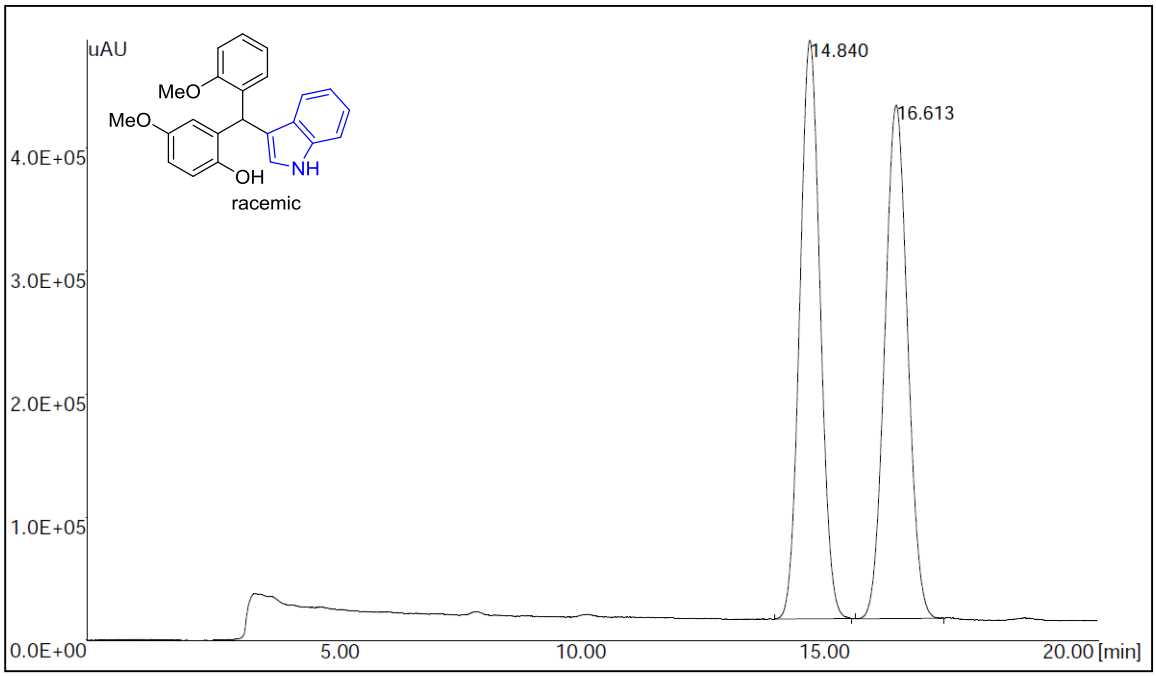
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		13.840	347351	11044541.901	49.91
2		17.333	298771	11085212.994	50.09

AD-H column, 80:20 (Hex:IPA), 1 mL/min, 288 nm(er = 95:5)



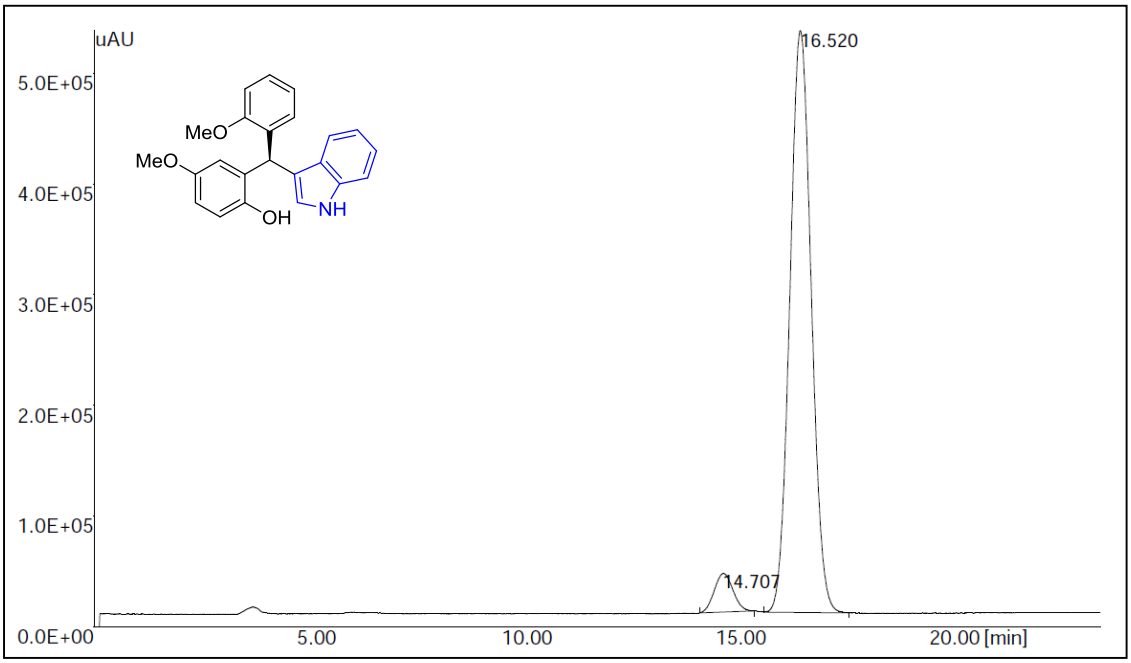
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		14.333	3015	93174.401	4.61
2		18.013	50421	1928583.229	95.39

(Table 2, entry 3)AD-H column, 70:30 (Hex:IPA), 1 mL/min, 225 nm



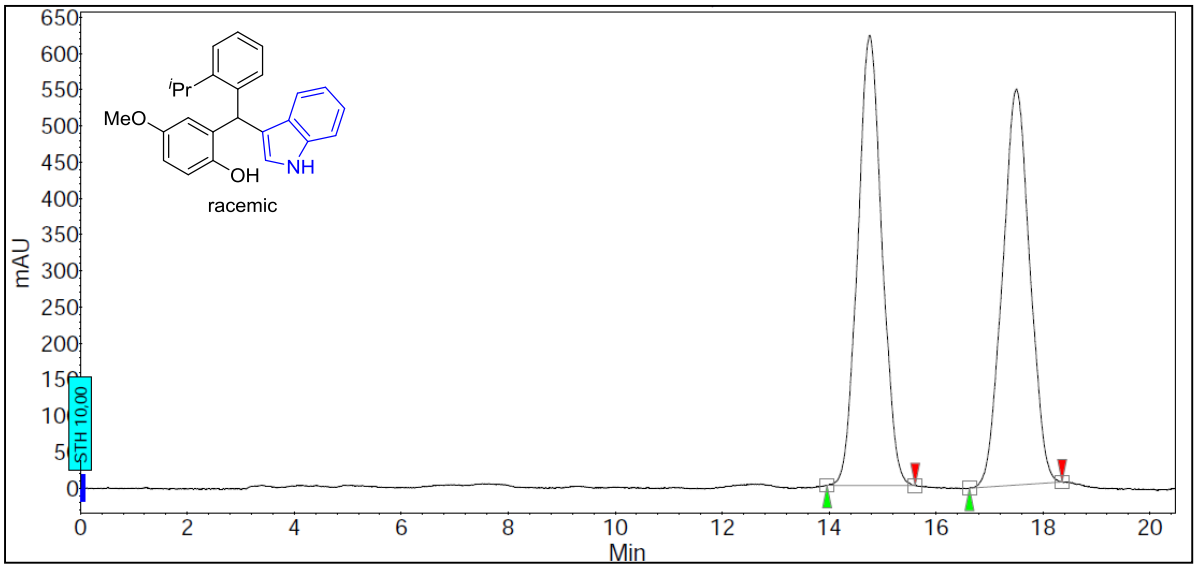
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		14.840	470358	13674964.204	50.14
2		16.613	417590	13597217.803	49.86

AD-H column, 70:30 (Hex:IPA), 1 mL/min, 225 nm(er = 95:5)



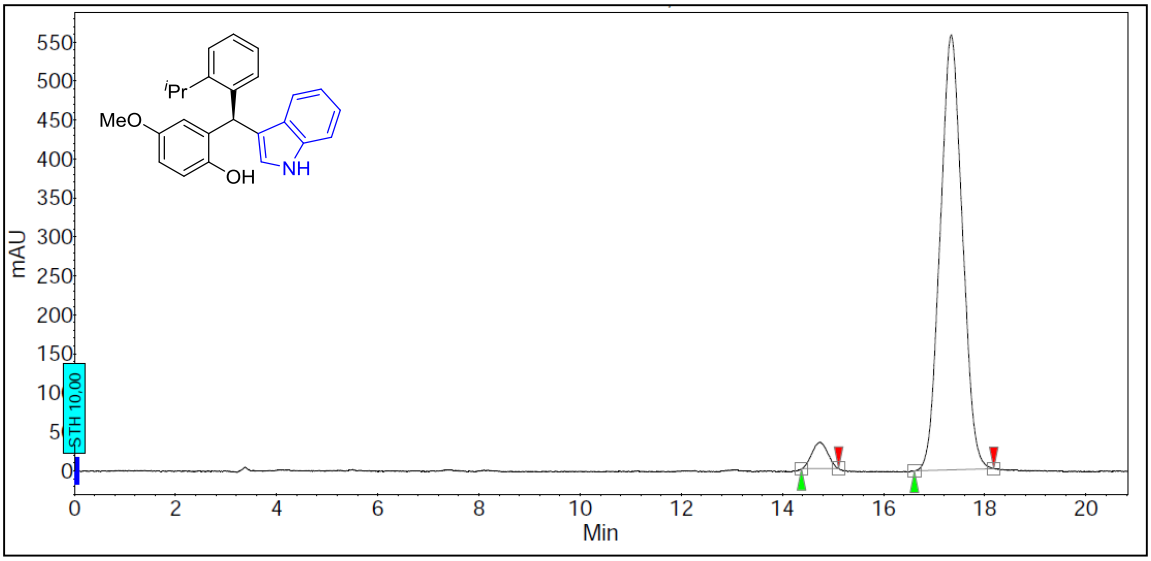
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		14.707	34819	1060851.216	5.49
2		16.520	525673	18269778.672	94.51

(Table 2, entry 4)IA column, 70:30 (Hex:IPA), 1 mL/min, 232 nm



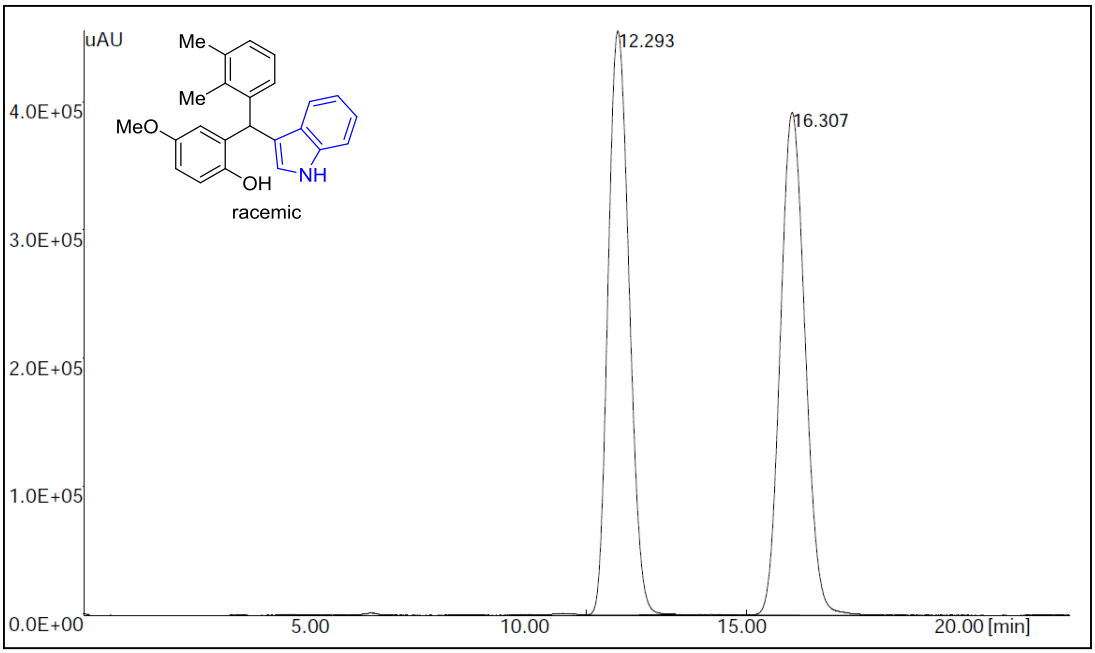
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	14,759	50,19	621,7	326,2	50,189
2	UNKNOWN	17,505	49,81	546,8	323,8	49,811
Total			100,00	1168,5	650,0	100,000

IA column, 70:30 (Hex:IPA), 1 mL/min, 232 nm(er = 96:4)



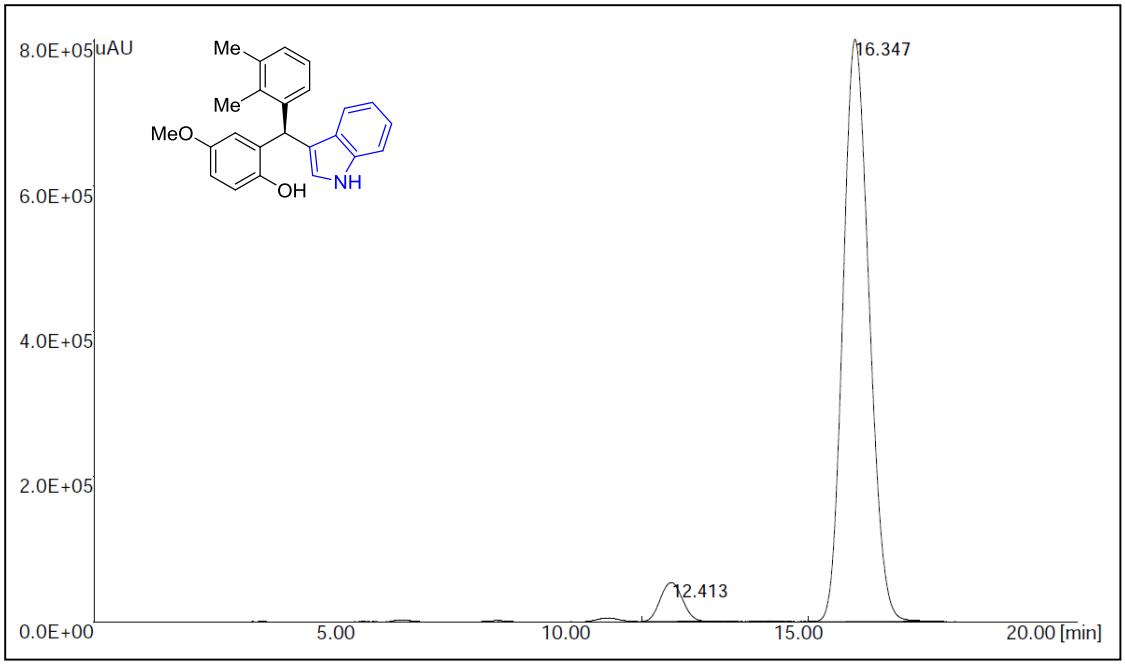
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	14,746	4,13	33,9	12,2	4,127
2	UNKNOWN	17,345	95,87	558,0	283,2	95,873
Total			100,00	591,9	295,4	100,000

(Table 2, entry 5)AD-H column, 80:20 (Hex:IPA), 1 mL/min, 265 nm



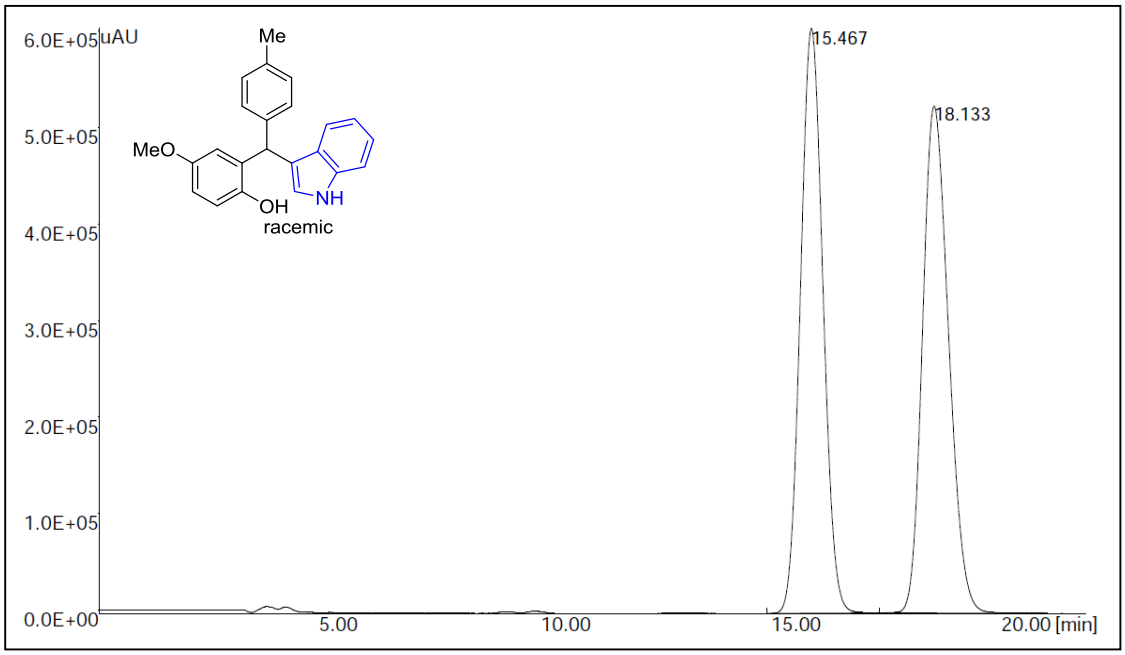
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		12.293	455659	15367151.829	49.81
2		16.307	392369	15483681.831	50.19

AD-H column, 80:20 (Hex:IPA), 1 mL/min, 265 nm(er = 95:5)



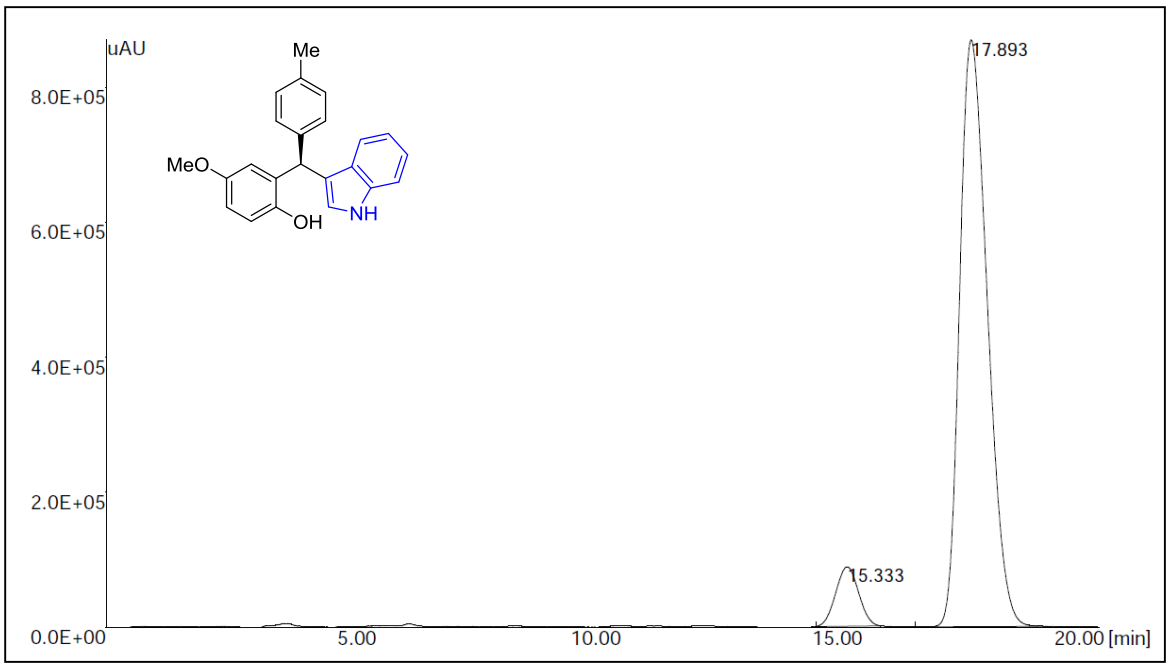
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		12.413	53486	1763612.826	5.35
2		16.347	801769	31202110.065	94.65

(Table 2, entry 6)AD-H column, 80:20 (Hex:IPA), 1 mL/min, 232 nm



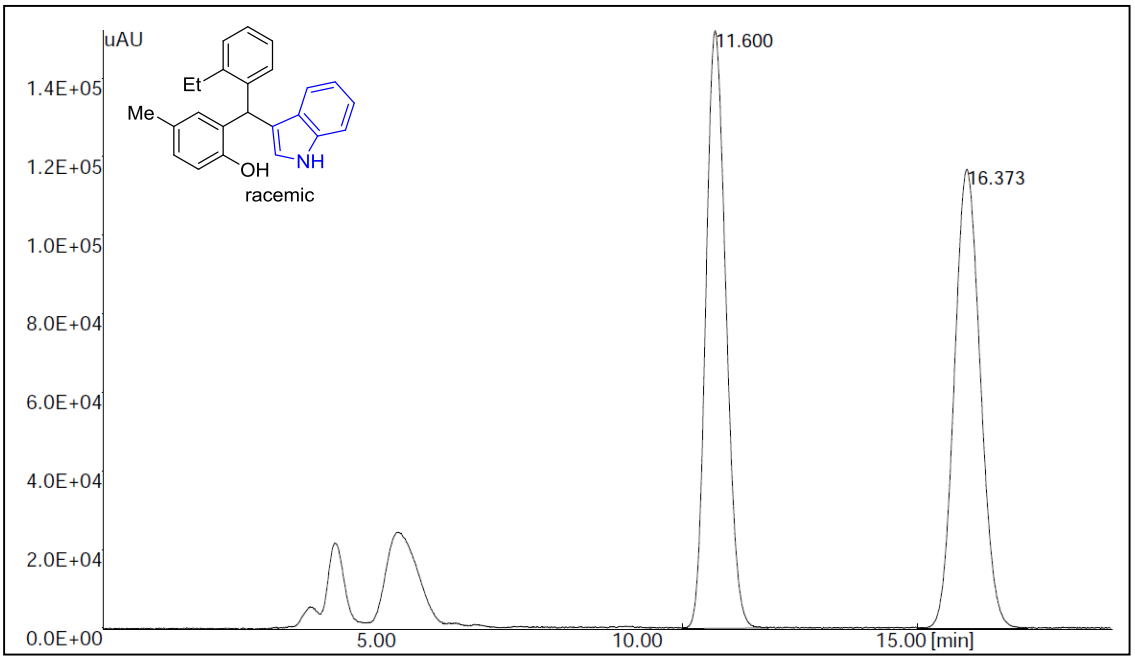
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		15.467	606062	20569183.507	49.97
2		18.133	524947	20593646.307	50.03

AD-H column, 80:20 (Hex:IPA), 1 mL/min, 232 nm(er = 92:8)



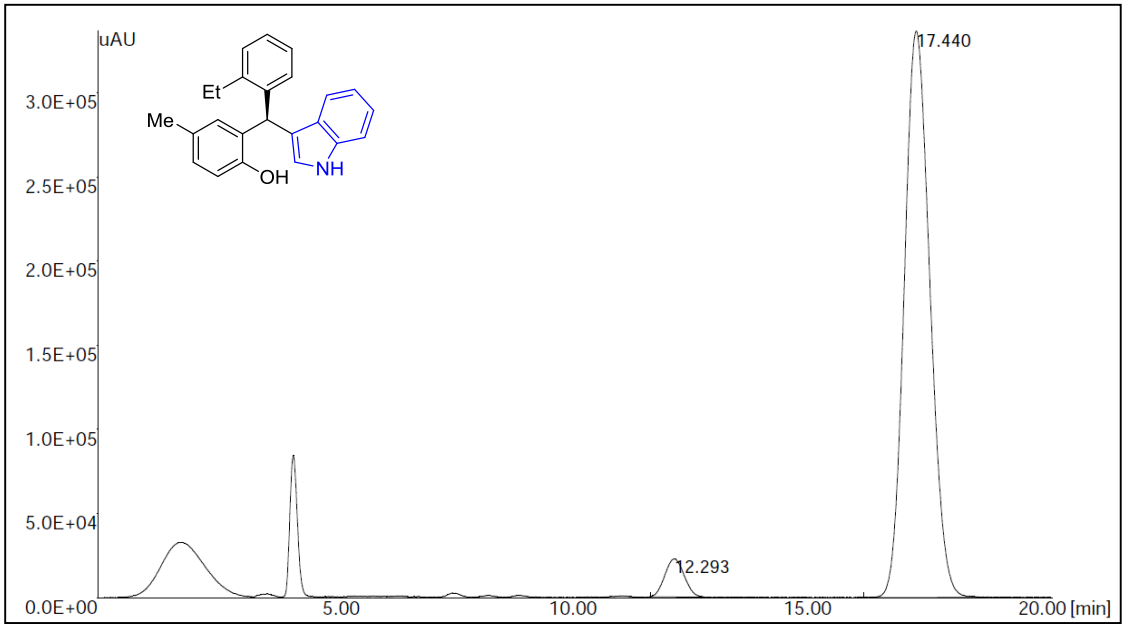
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		15.333	88143	2896336.043	7.71
2		17.893	869877	34658986.571	92.29

(Table 2, entry 7)AD-H column, 80:20 (Hex:IPA), 1 mL/min, 250 nm



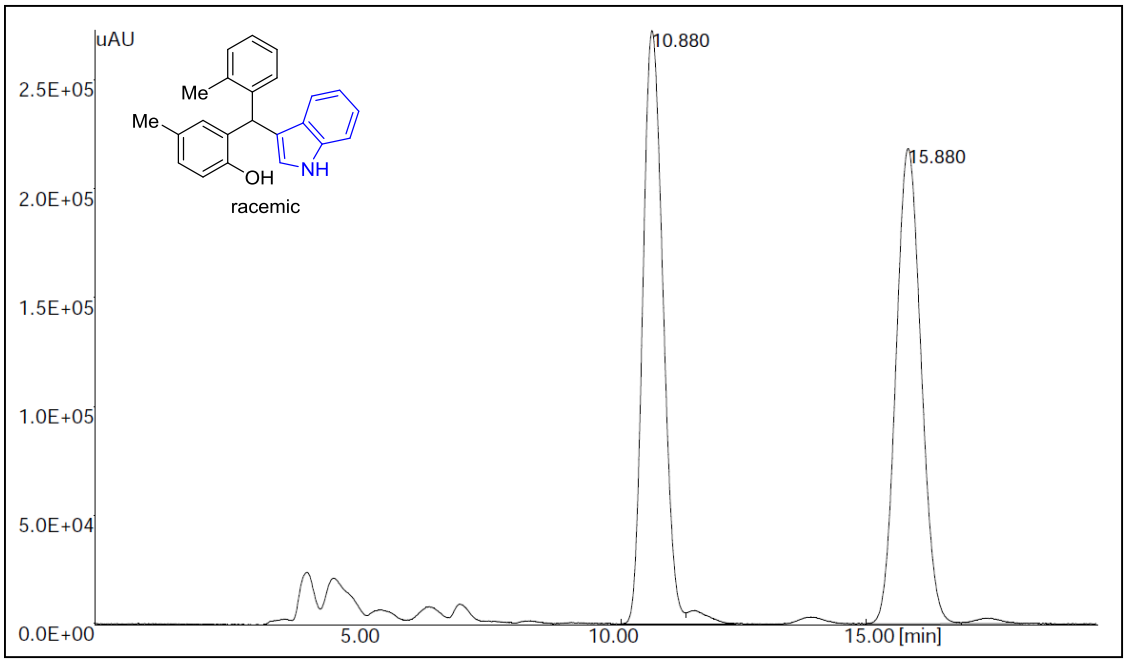
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		11.600	152066	4023261.260	50.13
2		16.373	116807	4002578.460	49.87

AD-H column, 80:20 (Hex:IPA), 1 mL/min, 250 nm(er = 95:5)



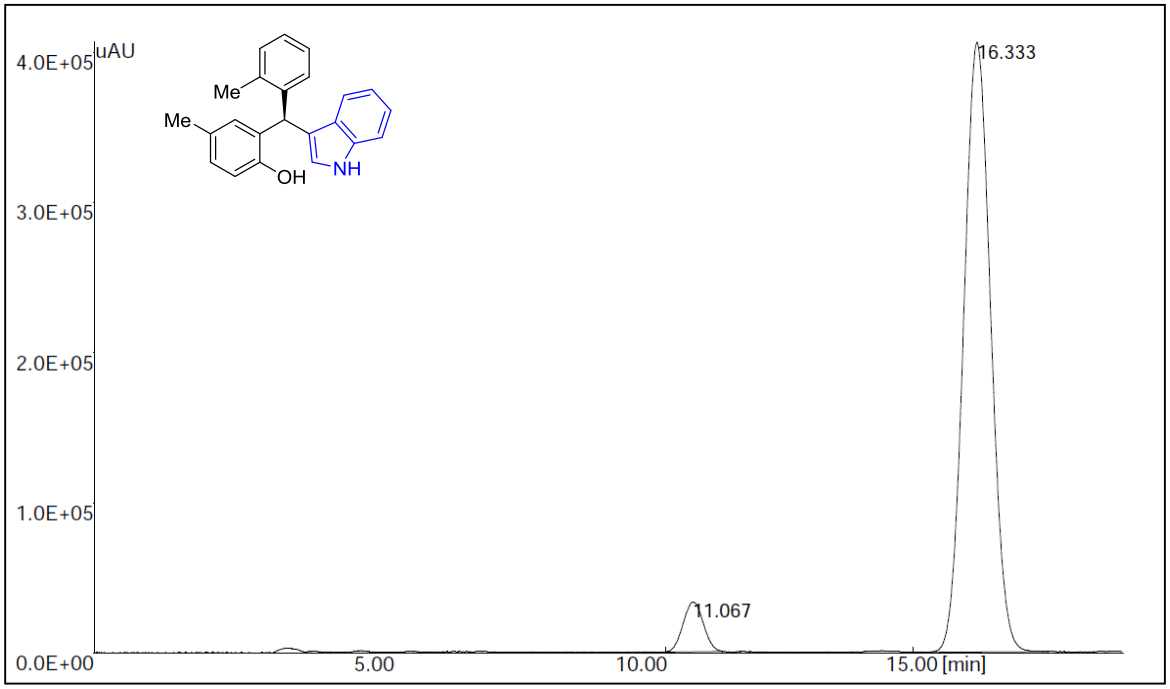
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		12.293	22649	648703.210	4.84
2		17.440	336321	12748629.773	95.16

(Table 2, entry 8)AD-H column, 80:20 (Hex:IPA), 1 mL/min, 232 nm



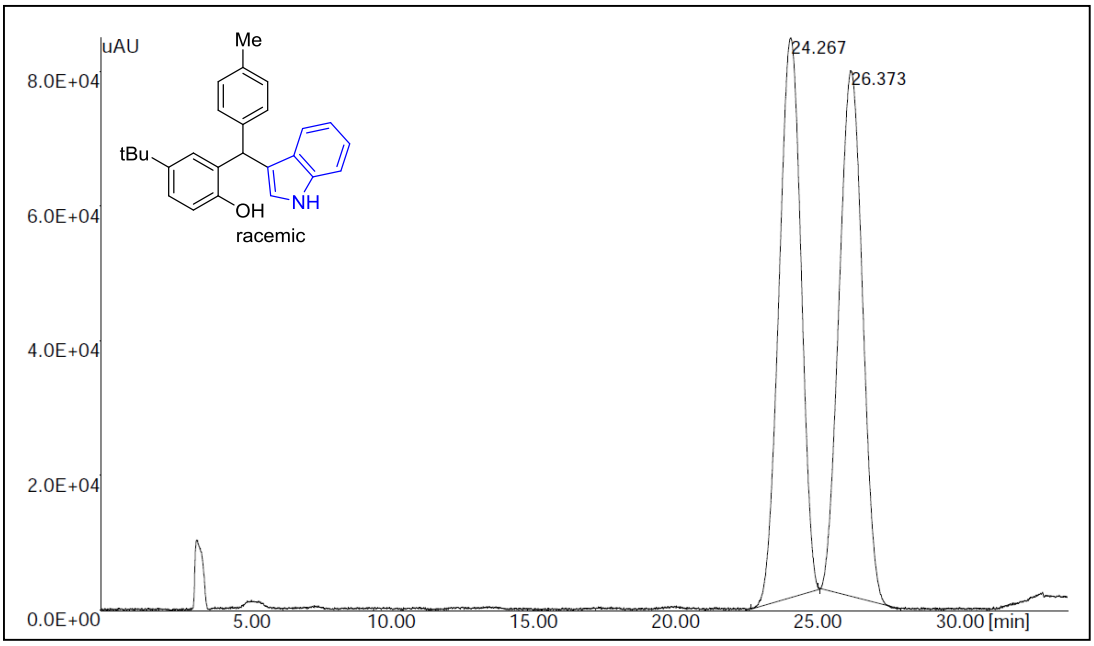
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		10.880	272825	7423204.568	49.97
2		15.880	218611	7433541.131	50.03

AD-H column, 80:20 (Hex:IPA), 1 mL/min, 232 nm(er = 94:6)



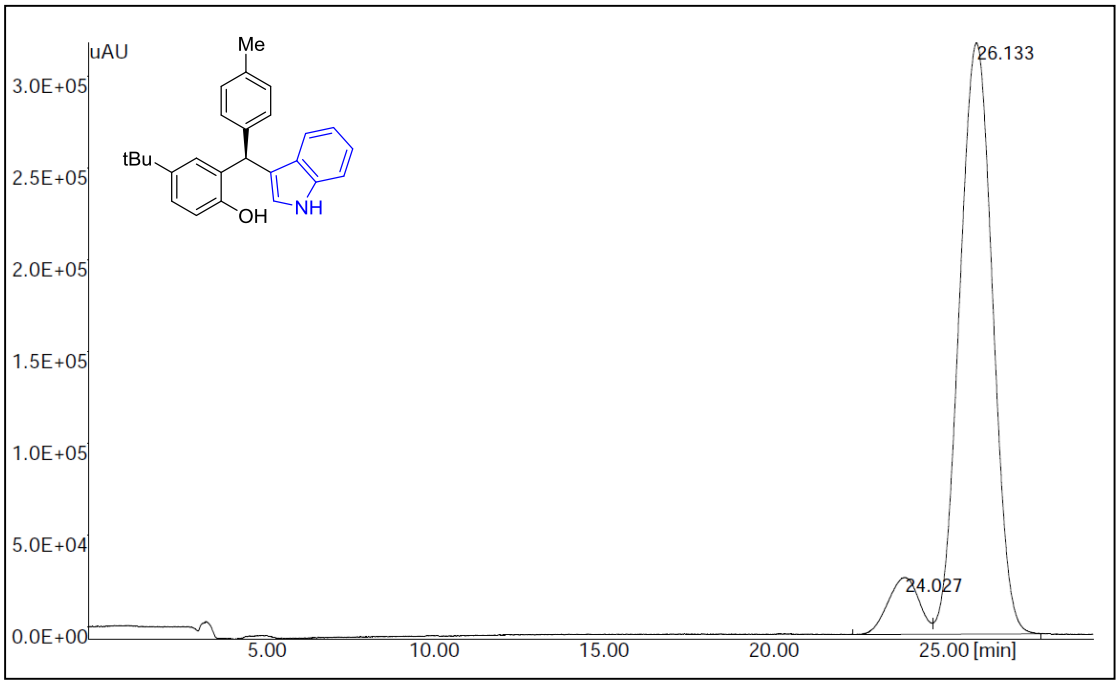
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		11.067	33120	860924.013	5.78
2		16.333	406460	14031268.864	94.22

(Table 2, entry 9)AD-H column, 90:10 (Hex:IPA), 1 mL/min, 220 nm



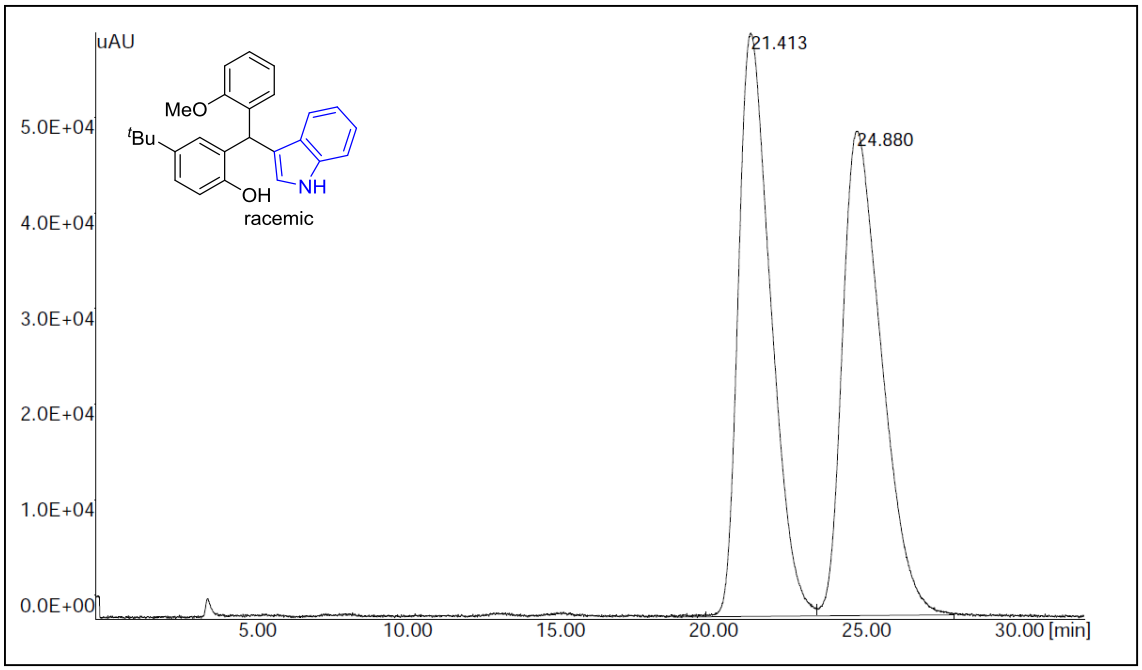
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		24.267	83147	4515318.867	50.25
2		26.373	78057	4471004.081	49.75

AD-H column, 90:10 (Hex:IPA), 1 mL/min, 220 nm (er = 92:8)



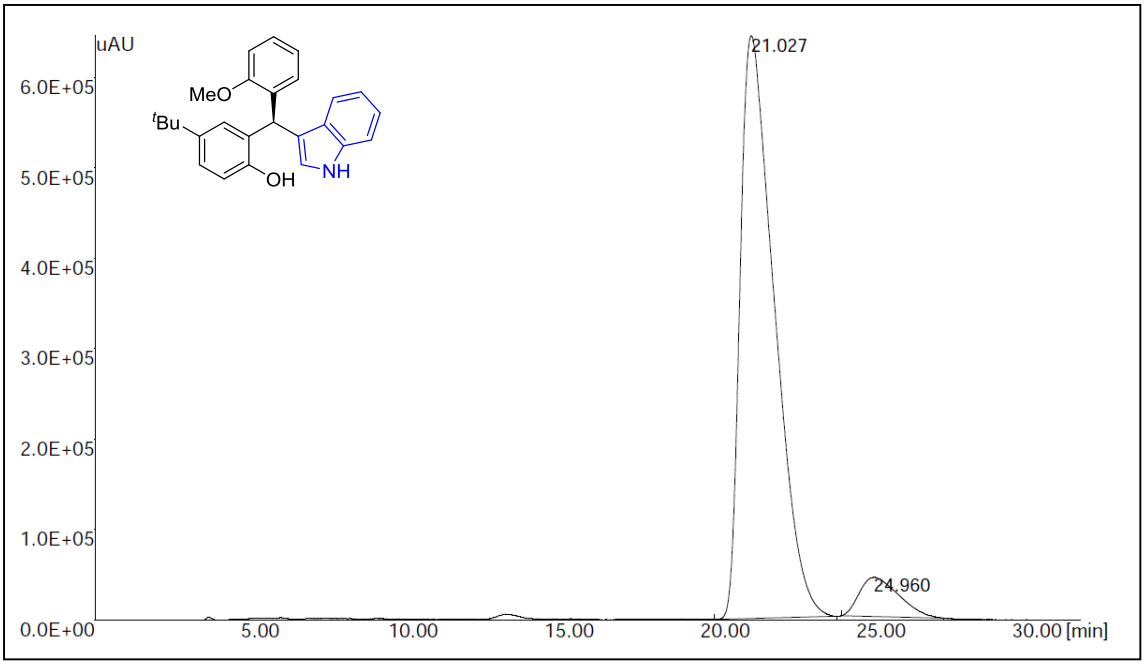
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		24.027	31080	1977403.608	8.31
2		26.133	322056	21826004.373	91.69

(Table 2, entry 10)OD-H column, 95:5 (Hex:IPA), 1 mL/min, 232 nm



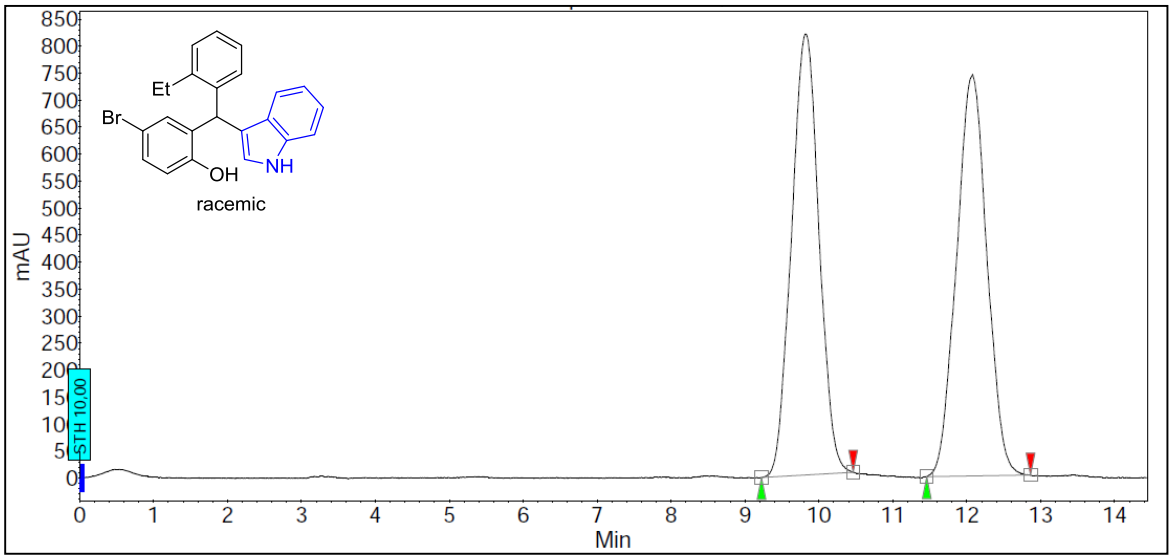
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		21.413	61031	4501064.535	49.97
2		24.880	50699	4506287.138	50.03

OD-H column, 95:5 (Hex:IPA), 1 mL/min, 232 nm(er = 93:7)



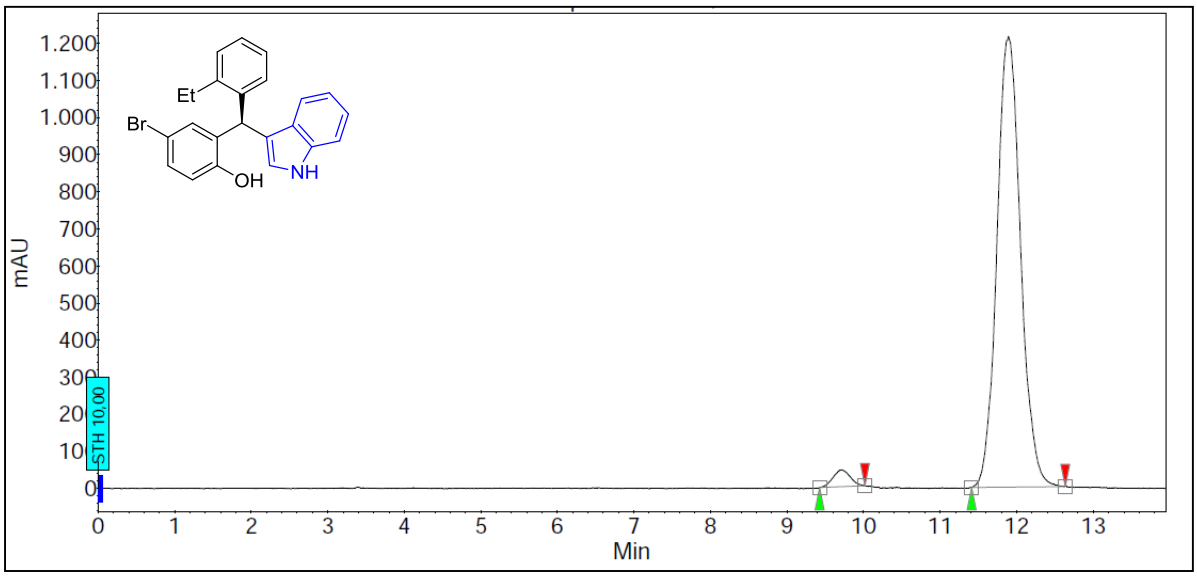
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		21.027	644639	48951191.930	92.77
2		24.960	43386	3812697.657	7.23

(Table 2, entry 11)IA column, 85:15 (Hex:IPA), 1 mL/min, 232 nm



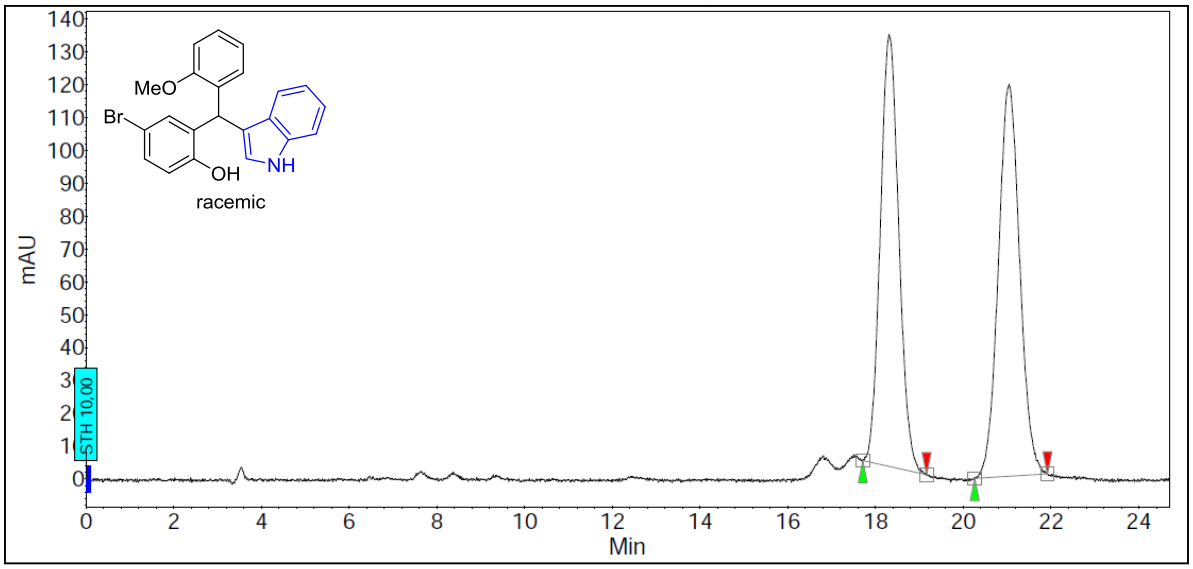
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	9,827	49,84	816,8	351,4	49,843
2	UNKNOWN	12,080	50,16	743,8	353,6	50,157
Total			100,00	1560,6	705,0	100,000

IA column, 85:15 (Hex:IPA), 1 mL/min, 232 nm(er = 97:3)



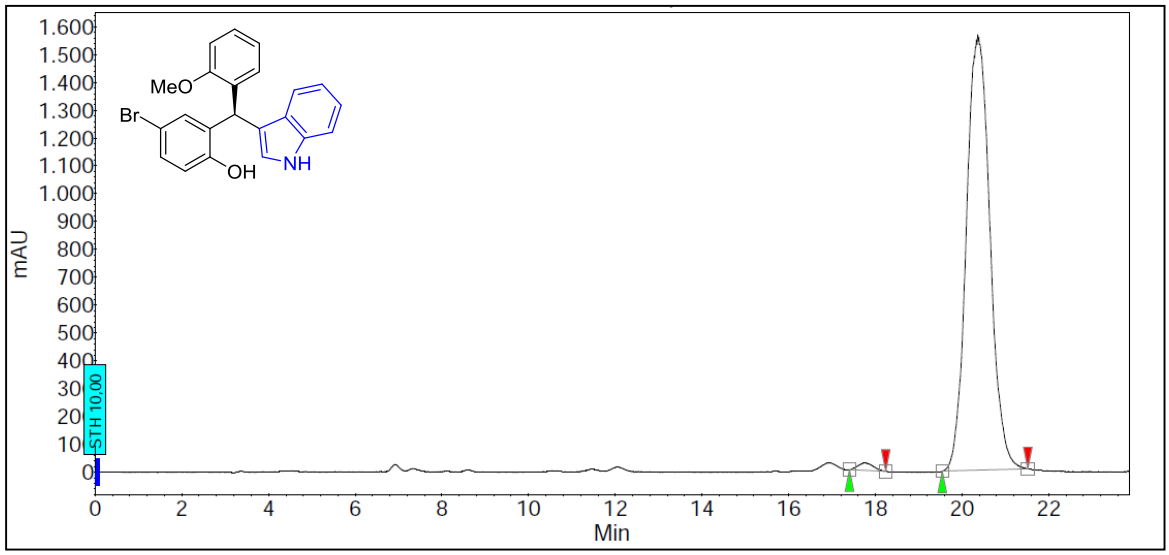
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	9,707	2,64	44,6	11,8	2,638
2	UNKNOWN	11,893	97,36	1215,2	434,8	97,362
Total			100,00	1259,8	446,5	100,000

(Table 2, entry 12)IA column, 85:15 (Hex:IPA), 1 mL/min, 232 nm



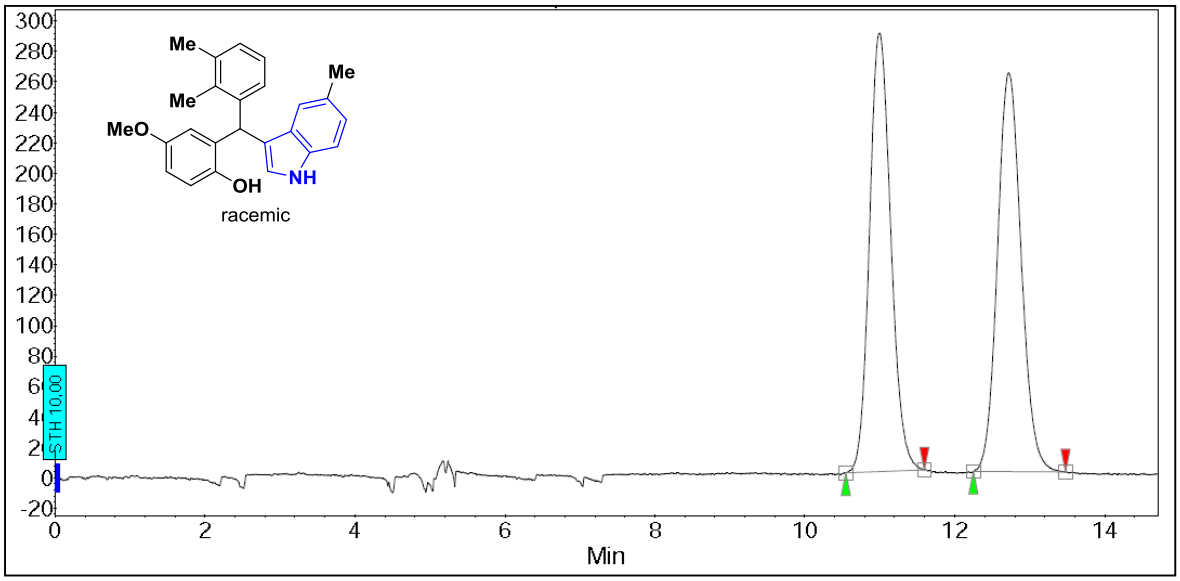
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	18,319	48,80	130,8	64,2	48,798
2	UNKNOWN	21,038	51,20	119,4	67,4	51,202
Total			100,00	250,2	131,5	100,000

IA column, 85:15 (Hex:IPA), 1 mL/min, 232 nm(er = 99:1)



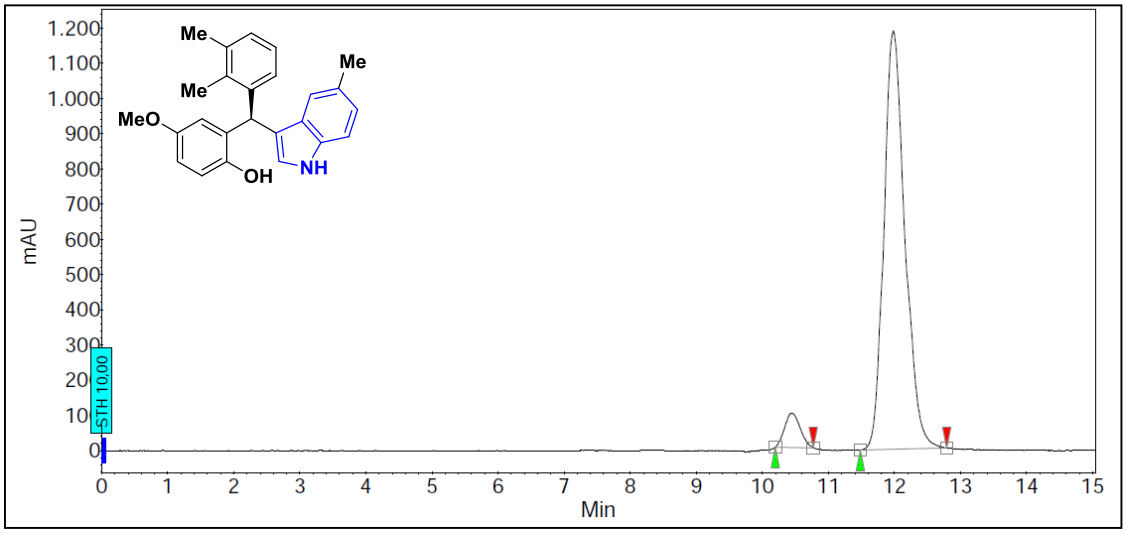
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	17,772	1,08	26,6	10,8	1,084
2	UNKNOWN	20,359	98,92	1564,0	983,4	98,916
Total			100,00	1590,5	994,2	100,000

(Table 3, entry1)IA column, 85:15 (Hex:IPA), 1 mL/min, 274 nm



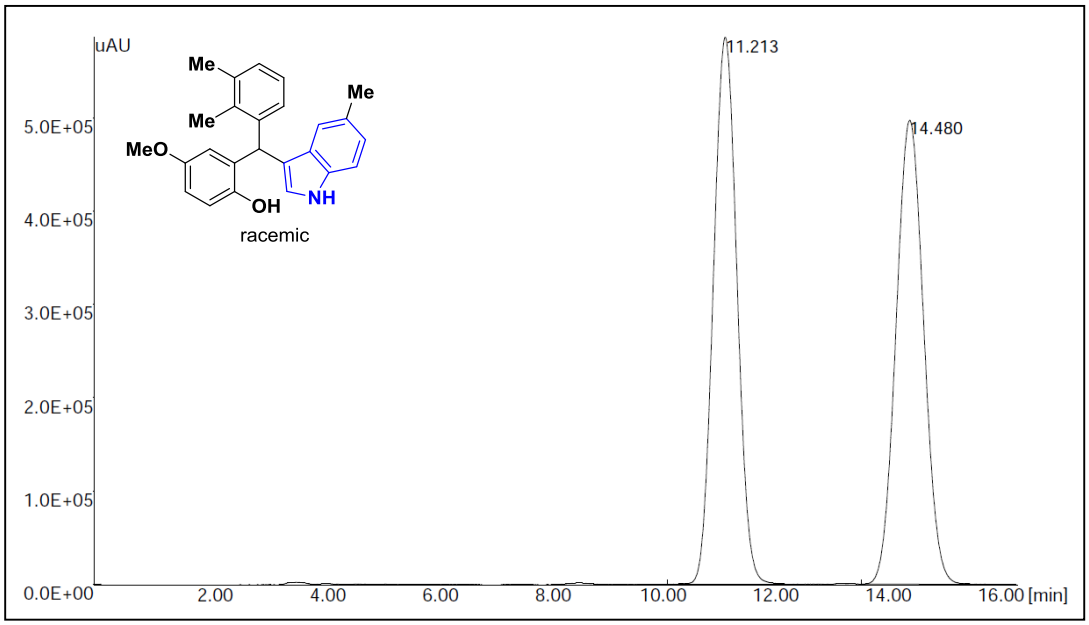
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	11,000	49,72	287,7	95,1	49,721
2	UNKNOWN	12,720	50,28	261,9	96,1	50,279
Total			100,00	549,7	191,2	100,000

IA column, 85:15 (Hex:IPA), 1 mL/min, 274 nm(er = 94:6)



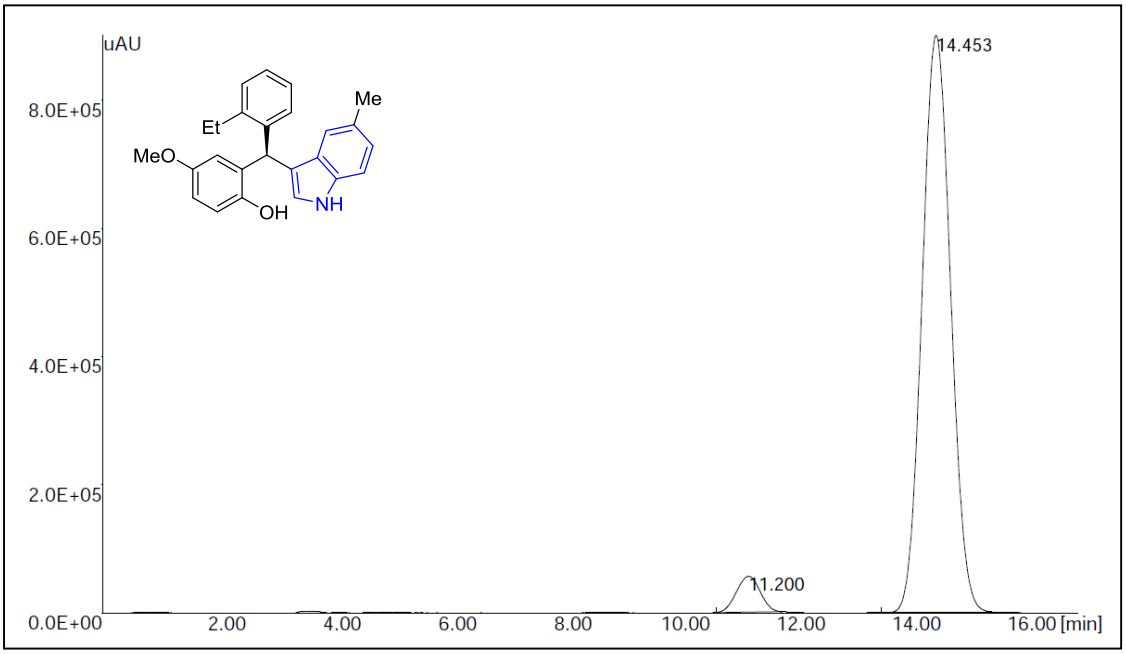
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
2	UNKNOWN	10,453	5,75	97,6	26,7	5,748
1	UNKNOWN	11,987	94,25	1188,5	437,1	94,252
Total			100,00	1286,1	463,8	100,000

(Table 3, entry2)AD-H column, 80:20 (Hex:IPA), 1 mL/min, 234 nm



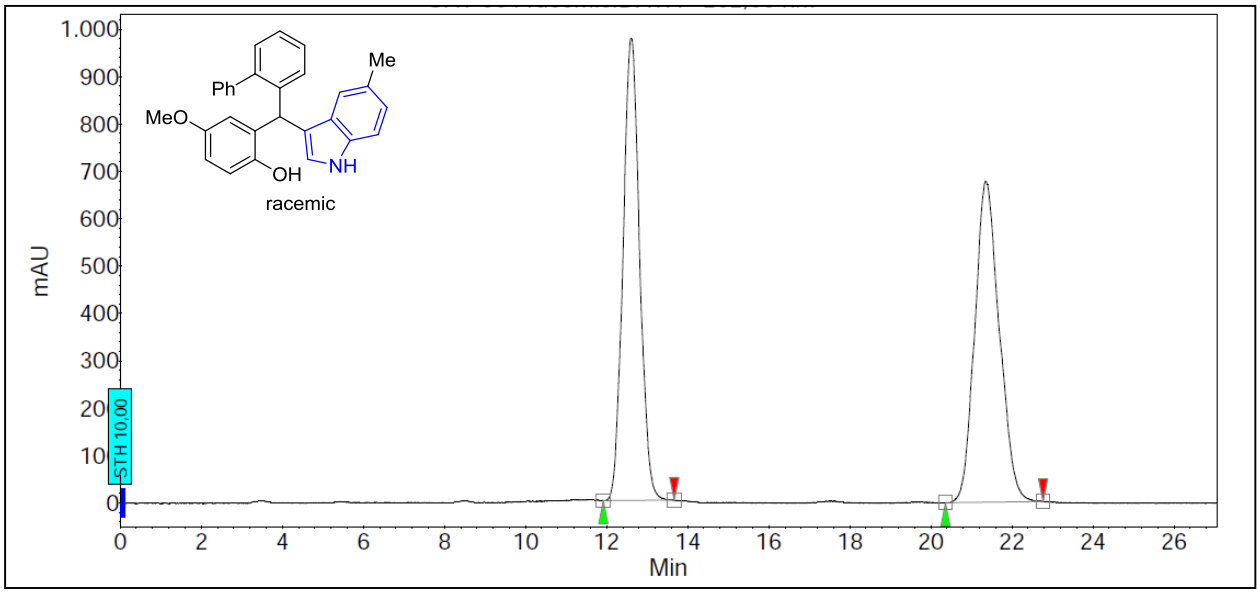
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		11.213	586356	17048371.454	50.13
2		14.480	497352	16960008.253	49.87

AD-H column, 80:20 (Hex:IPA), 1 mL/min, 234 nm(er = 95:5)



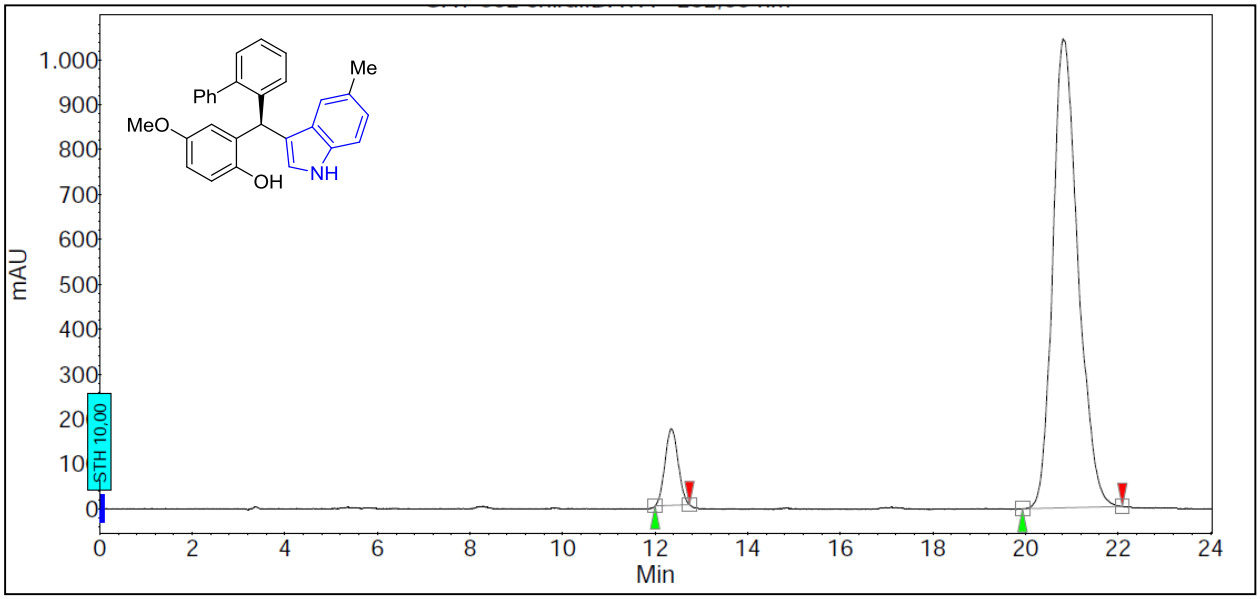
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		11.200	55823	1586979.224	4.88
2		14.453	901049	30954424.461	95.12

(Table 3, entry 3)IA column, 85:15 (Hex:IPA), 1 mL/min, 232 nm



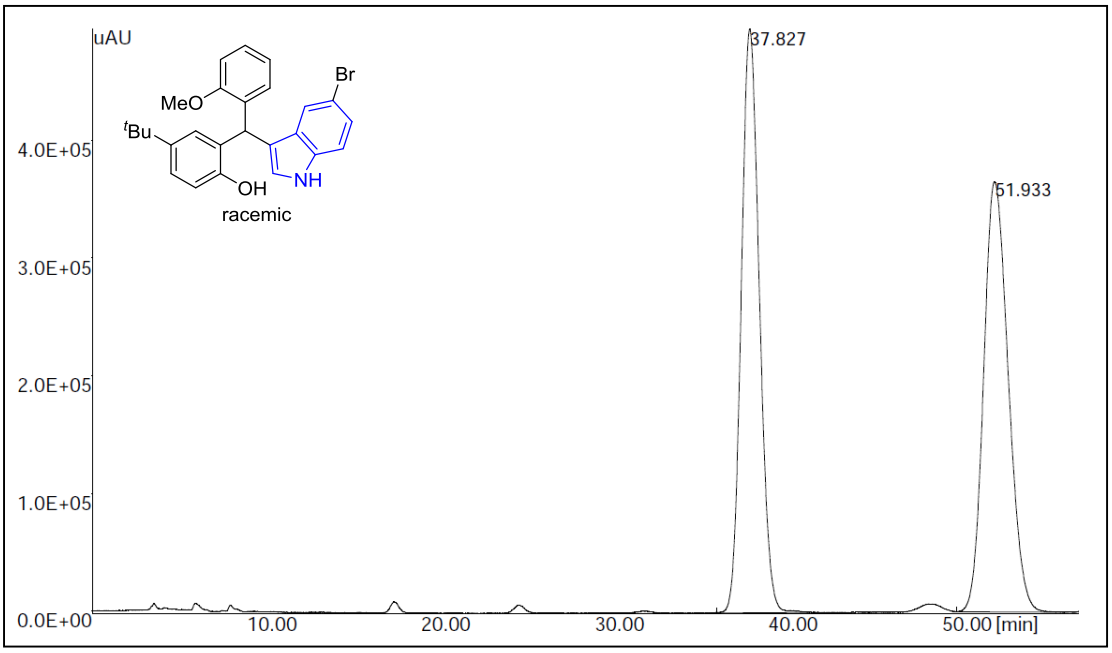
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	12,599	49,38	975,7	463,0	49,382
2	UNKNOWN	21,345	50,62	678,0	474,6	50,618
Total			100,00	1653,6	937,6	100,000

IA column, 85:15 (Hex:IPA), 1 mL/min, 232 nm(er = 92:8)



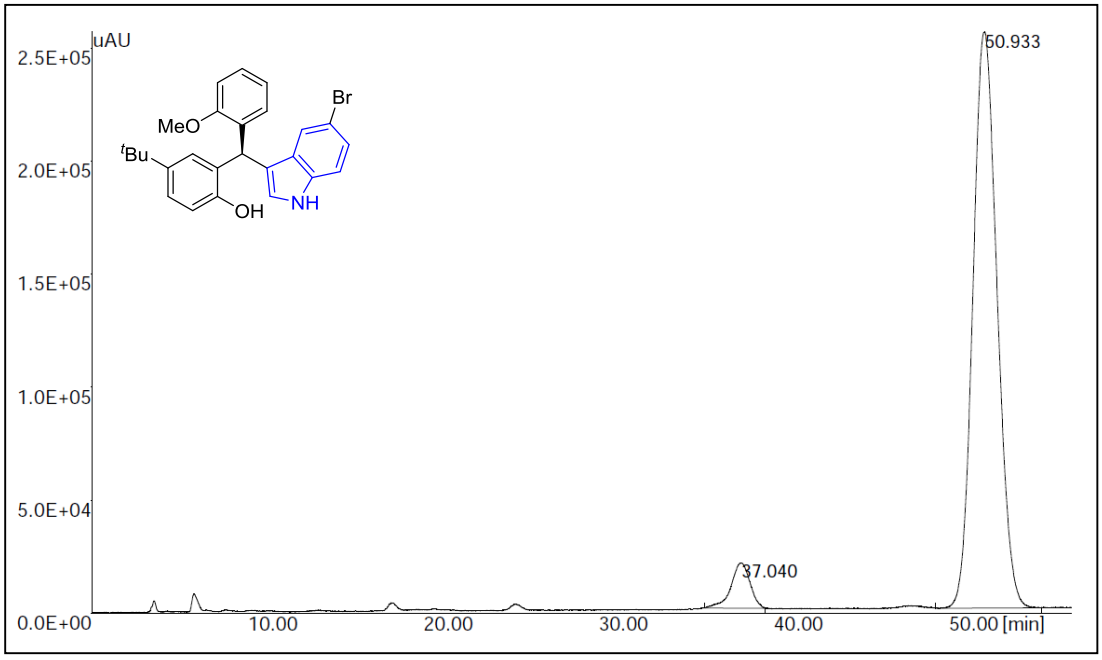
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
2	UNKNOWN	12,346	7,82	170,9	56,1	7,823
1	UNKNOWN	20,812	92,18	1045,3	661,2	92,177
Total			100,00	1216,2	717,3	100,000

(Table 3, entry 4)AD-H column, 90:10 (Hex:IPA), 1 mL/min, 220 nm



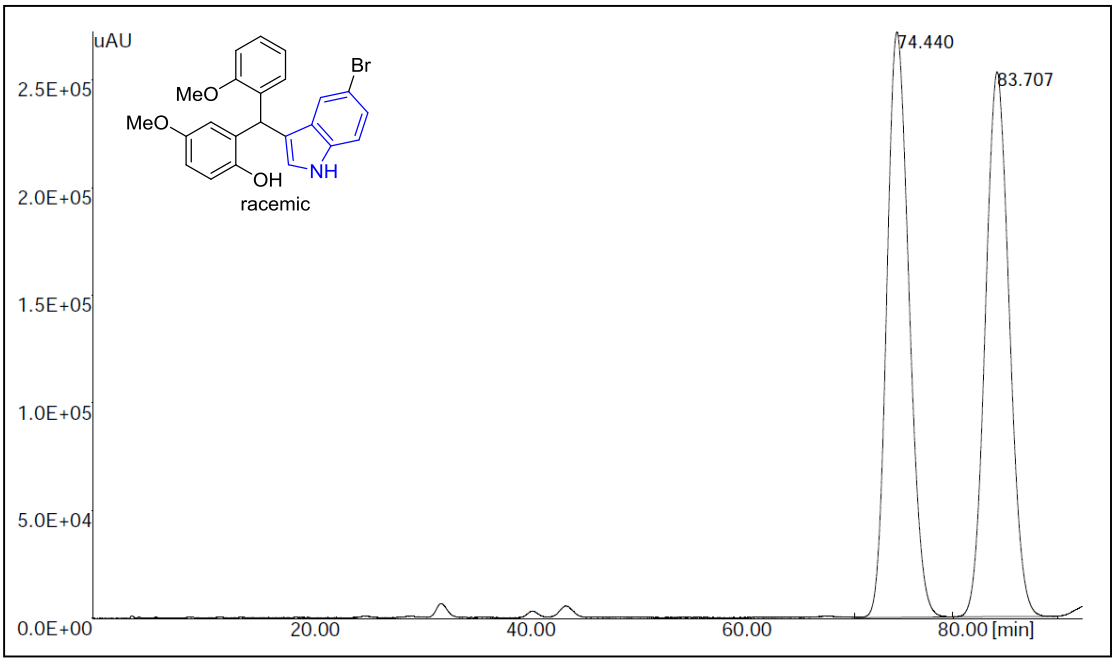
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		37.827	496156	35462136.928	49.86
2		51.933	365936	35666074.728	50.14

AD-H column, 90:10 (Hex:IPA), 1 mL/min, 220 nm(er = 94:6)



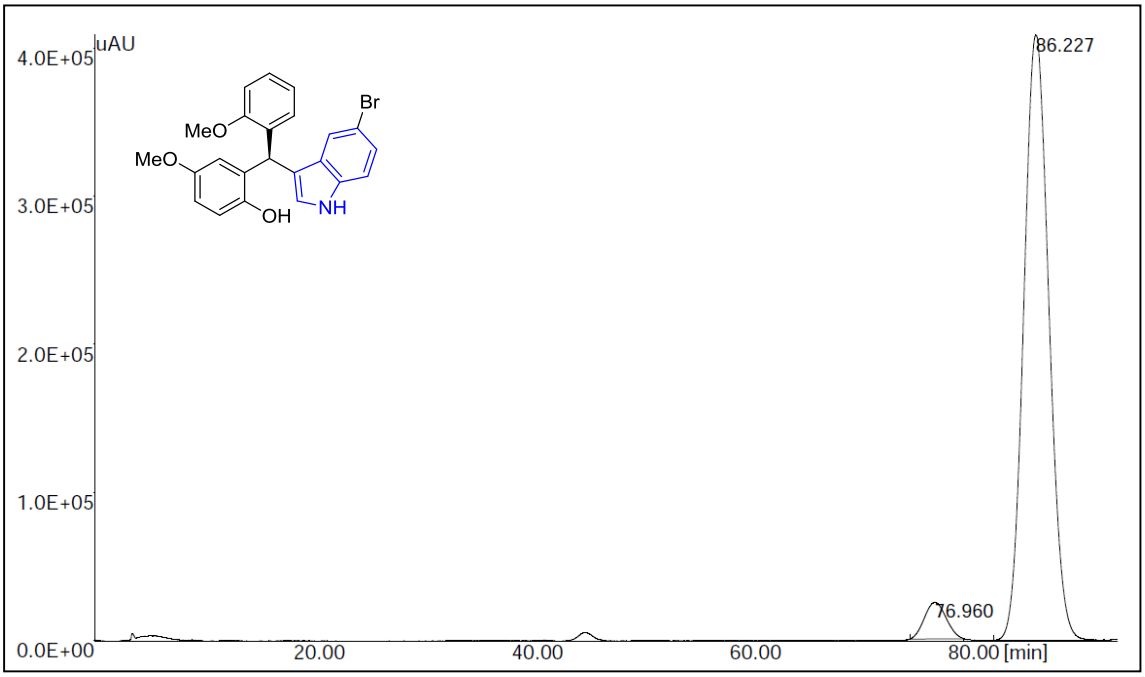
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		37.040	20059	1493926.822	5.82
2		50.933	254926	24195514.913	94.18

(Table 3, entry5)AD-H column, 90:10 (Hex:IPA), 1 mL/min, 236 nm



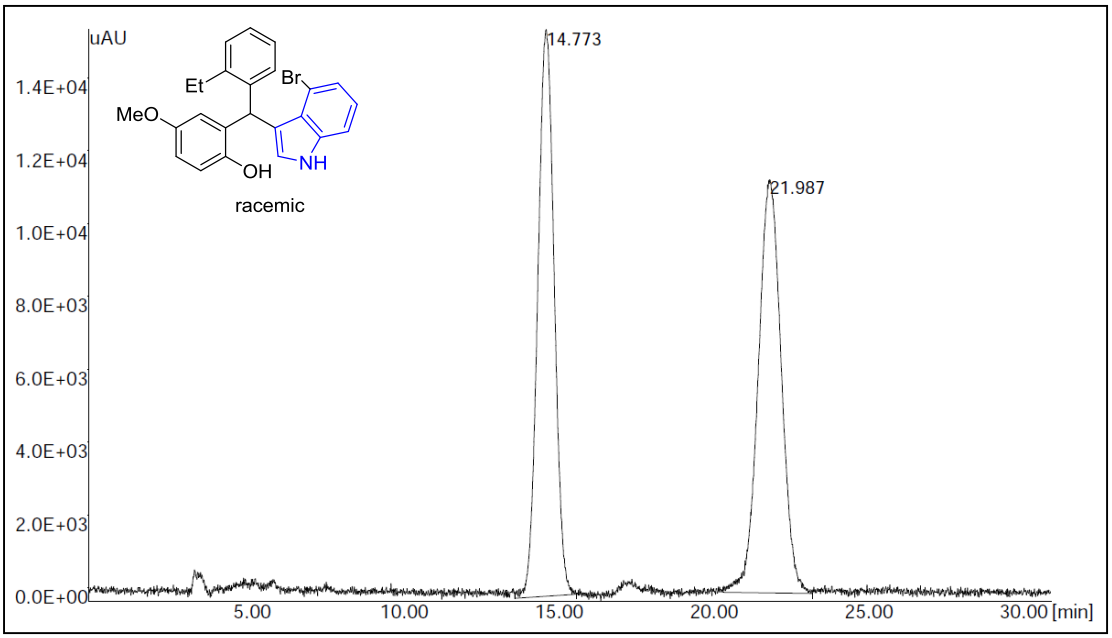
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		74.440	271782	38603109.775	50.02
2		83.707	253064	38568022.820	49.98

AD-H column, 90:10 (Hex:IPA), 1 mL/min, 236 nm(er = 95:5)



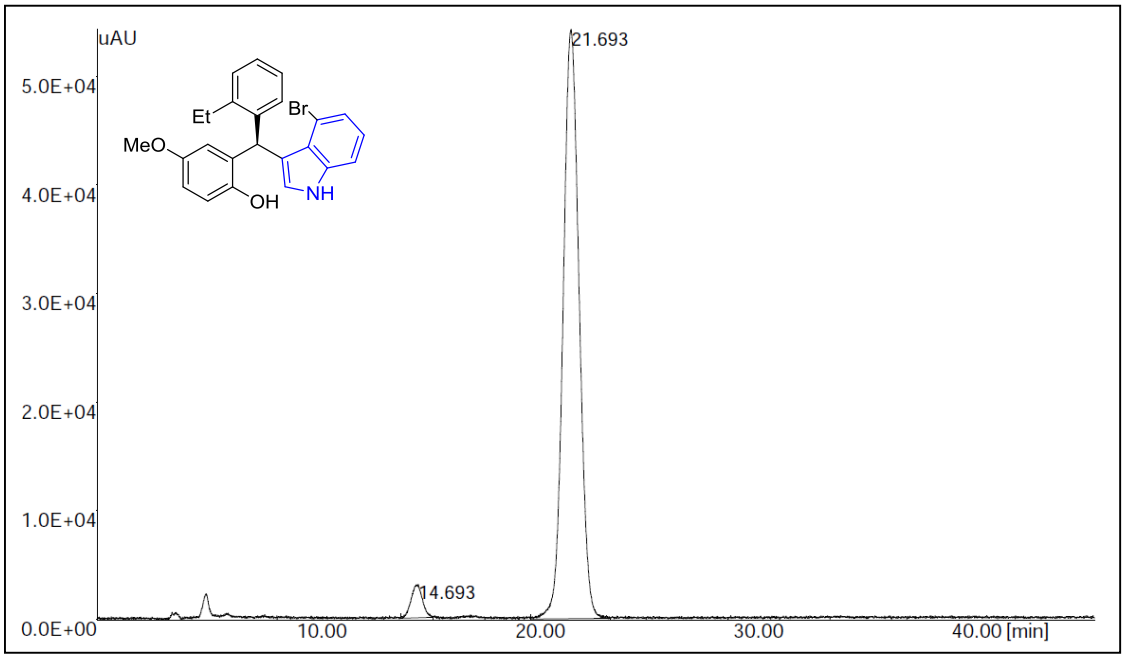
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		76.960	24864	3412681.651	5.01
2		86.227	408945	64687401.764	94.99

(Table 3, entry6)OD-H column, 80:20 (Hex:IPA), 1 mL/min, 262nm



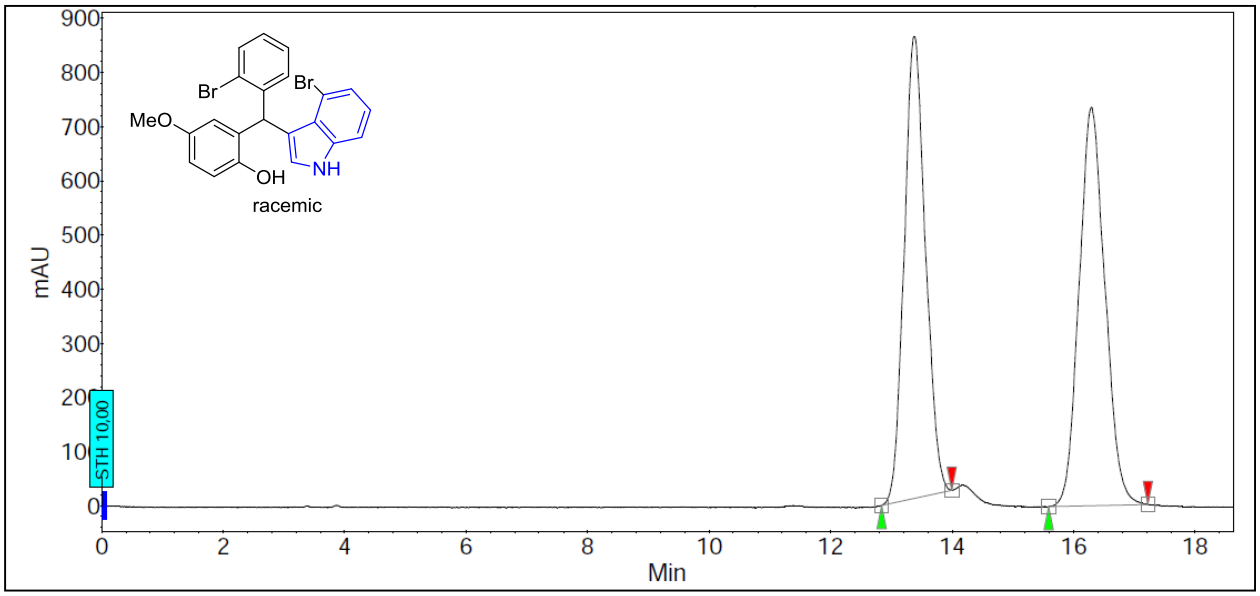
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		14.773	15550	574073.609	49.47
2		21.987	11343	586257.609	50.53

OD-H column, 80:20 (Hex:IPA), 1 mL/min, 262 nm(er = 96:4)



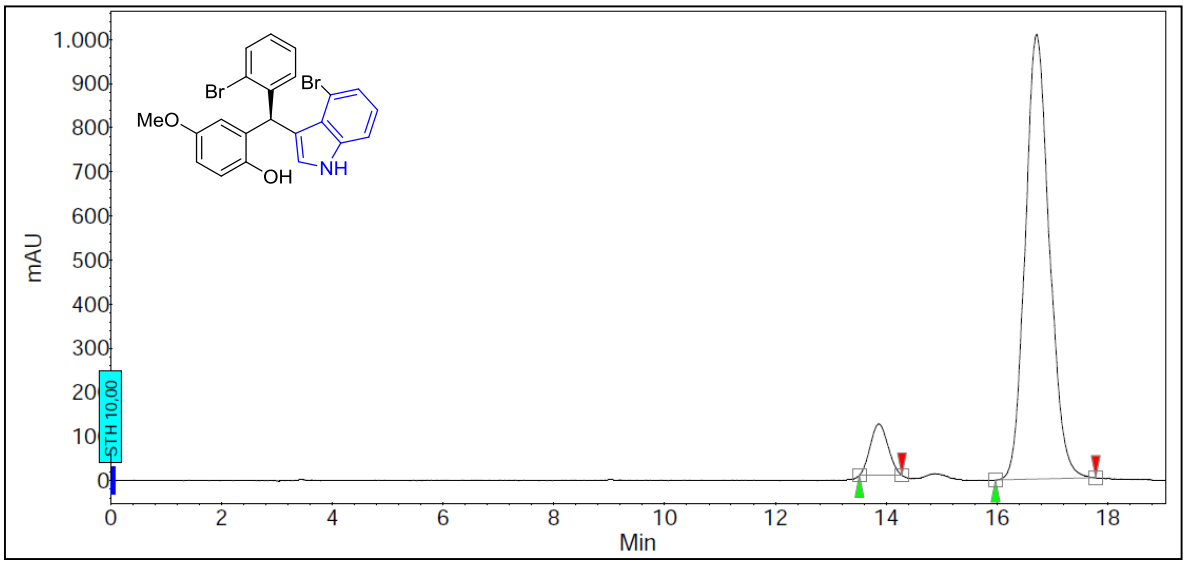
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		14.693	3054	110736.002	3.85
2		21.693	54299	2763694.051	96.15

(Table 3, entry 7)IA column, 85:15 (Hex:IPA), 1 mL/min, 236 nm



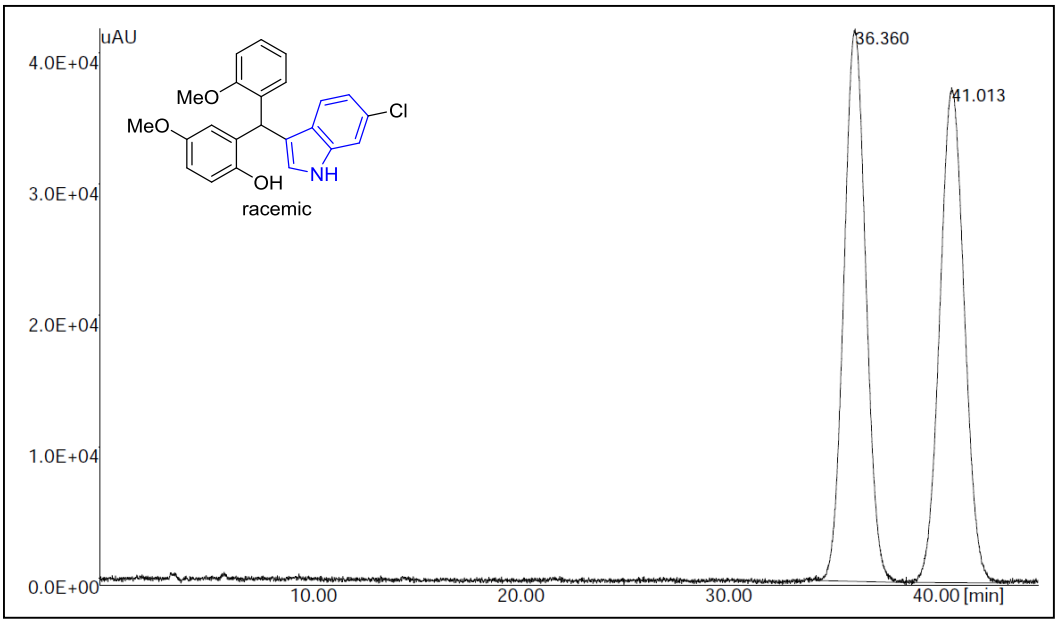
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	13,372	48,79	852,8	345,5	48,793
2	UNKNOWN	16,292	51,21	735,7	362,6	51,207
Total			100,00	1588,5	708,0	100,000

IA column, 85:15 (Hex:IPA), 1 mL/min, 236 nm(er = 92:8)



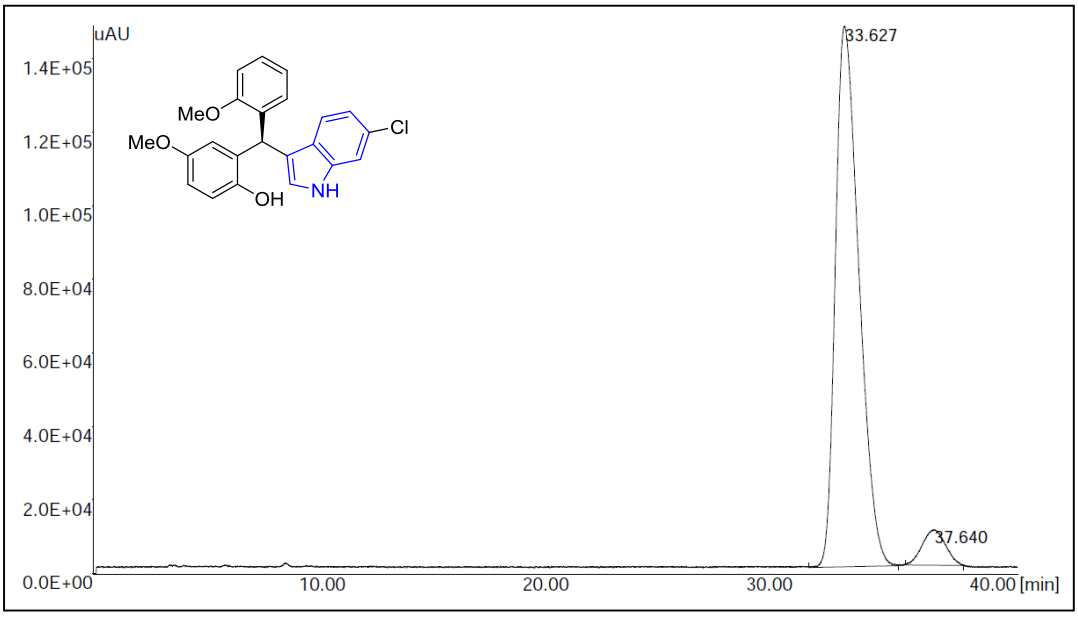
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
2	UNKNOWN	13,866	7,75	116,6	42,8	7,747
1	UNKNOWN	16,719	92,25	1009,2	509,2	92,253
Total			100,00	1125,8	551,9	100,000

(Table 3, entry8)OD-H column, 80:20 (Hex:IPA), 1 mL/min, 278 nm



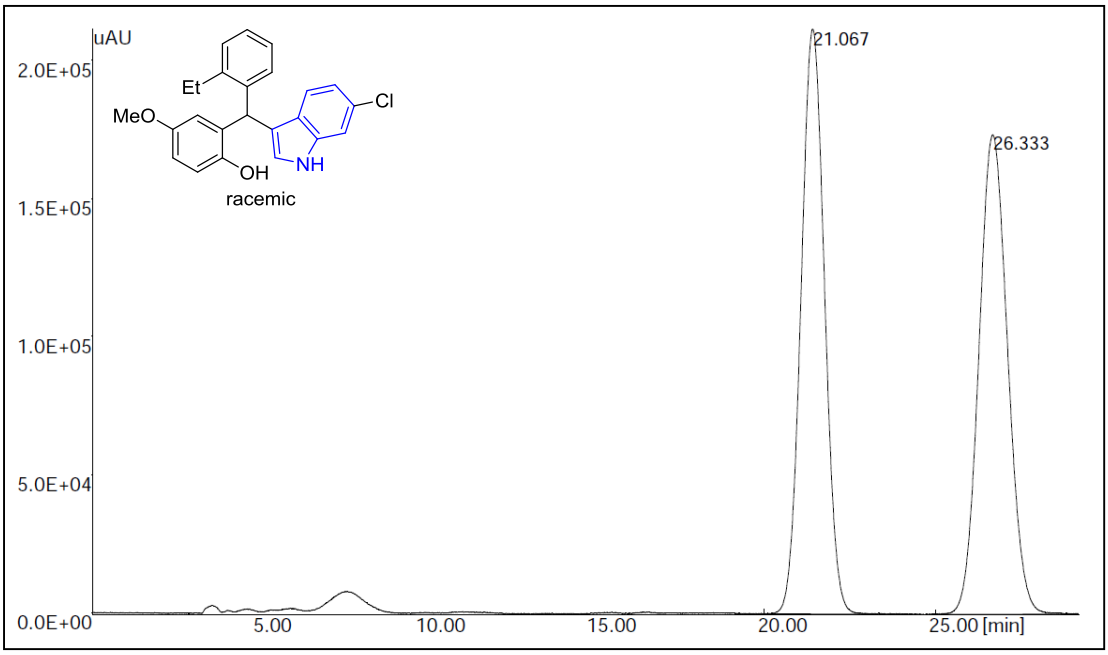
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		36.360	42086	3089398.305	49.91
2		41.013	37725	3101062.392	50.09

OD-H column, 80:20 (Hex:IPA), 1 mL/min, 278 nm(er = 94:6)



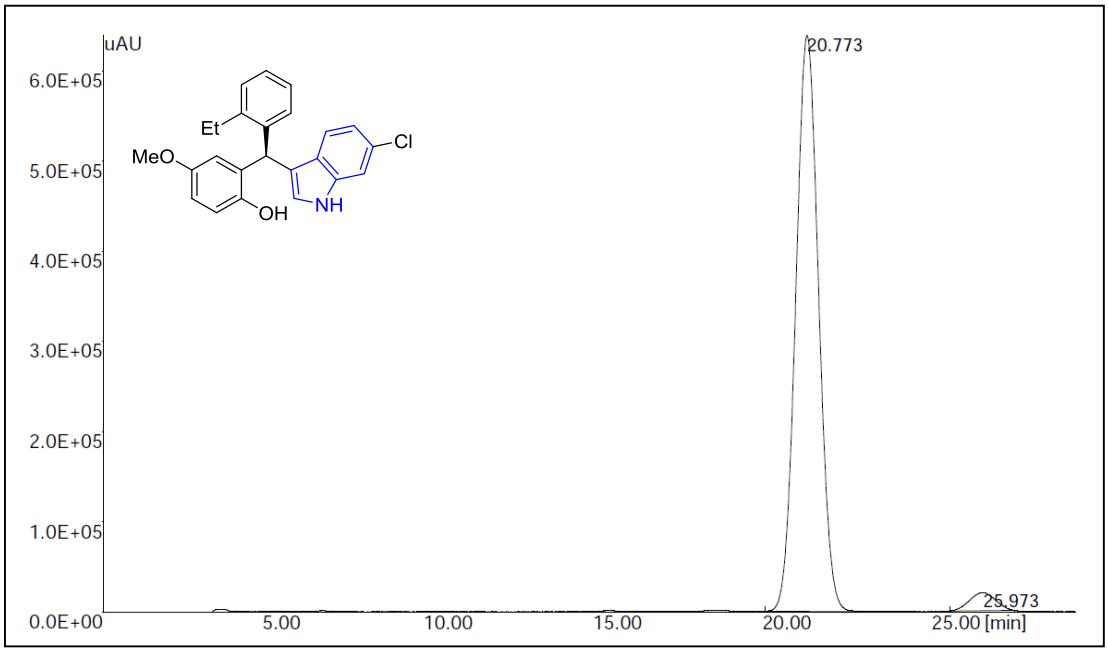
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		33.627	147044	10762398.740	93.90
2		37.640	9684	699712.010	6.10

(Table 3, entry9)OD-H column, 80:20 (Hex:IPA), 1 mL/min, 232 nm



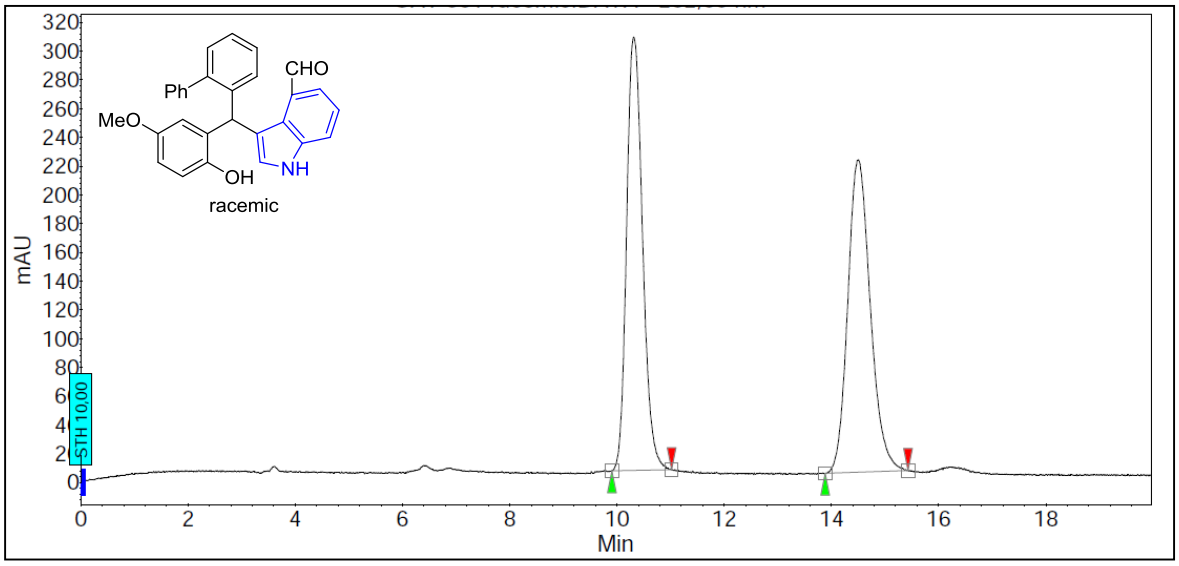
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		21.067	211659	10097041.580	49.96
2		26.333	173542	10112766.551	50.04

OD-H column, 80:20 (Hex:IPA), 1 mL/min, 232 nm (er = 97:3)



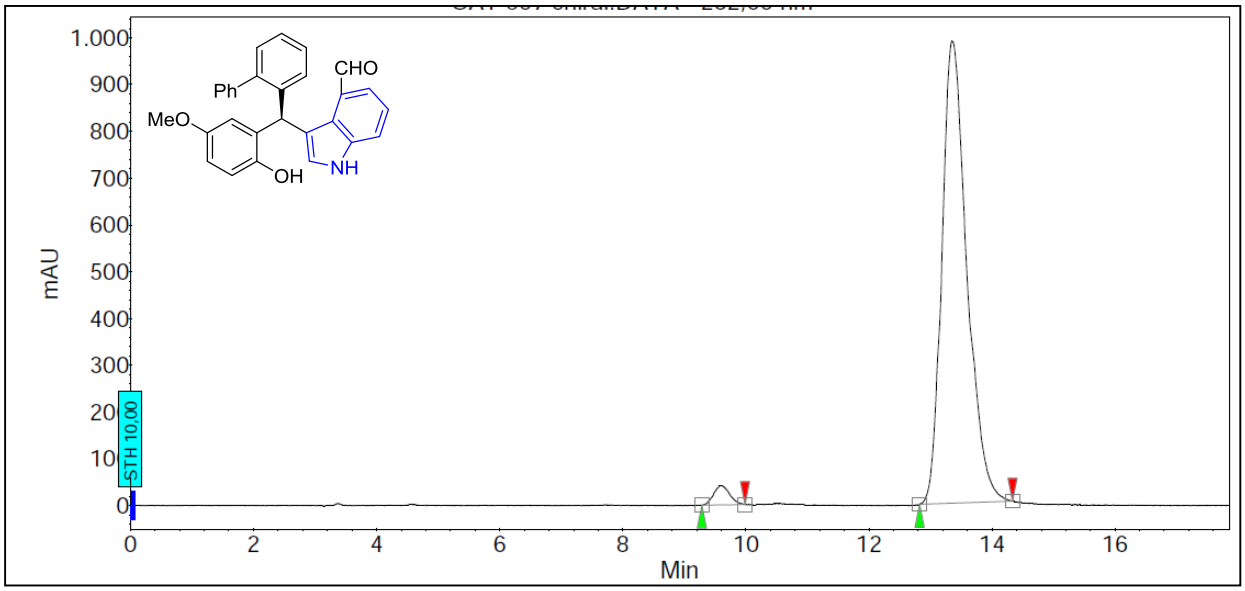
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		20.773	638802	29901555.646	96.50
2		25.973	20086	1084248.016	3.50

(Table 3, entry 10) IA column, 80:20 (Hex:IPA), 1 mL/min, 232 nm



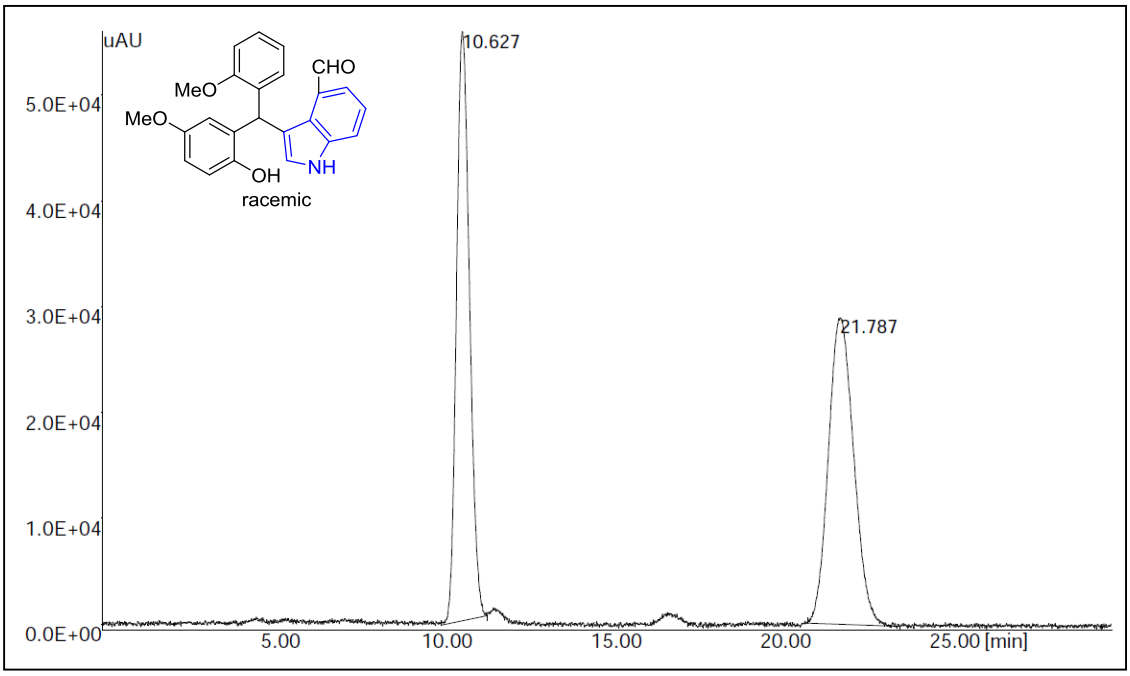
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	10,306	49,47	301,4	103,8	49,469
2	UNKNOWN	14,492	50,53	216,9	106,0	50,531
Total			100,00	518,3	209,7	100,000

IA column, 80:20 (Hex:IPA), 1 mL/min, 232 nm(er = 97:3)



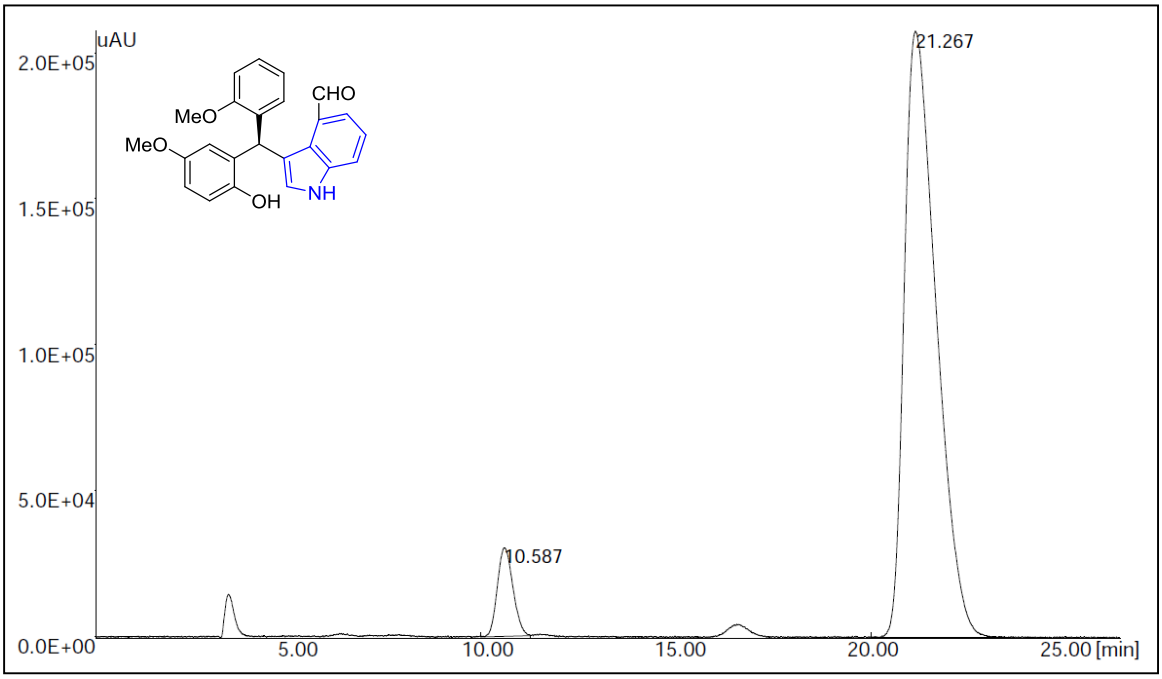
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	9,599	2,58	41,1	12,3	2,583
2	UNKNOWN	13,346	97,42	988,4	463,3	97,417
Total			100,00	1029,6	475,6	100,000

(Table 3, entry11)AD-H column, 70:30 (Hex:IPA), 1 mL/min, 333 nm



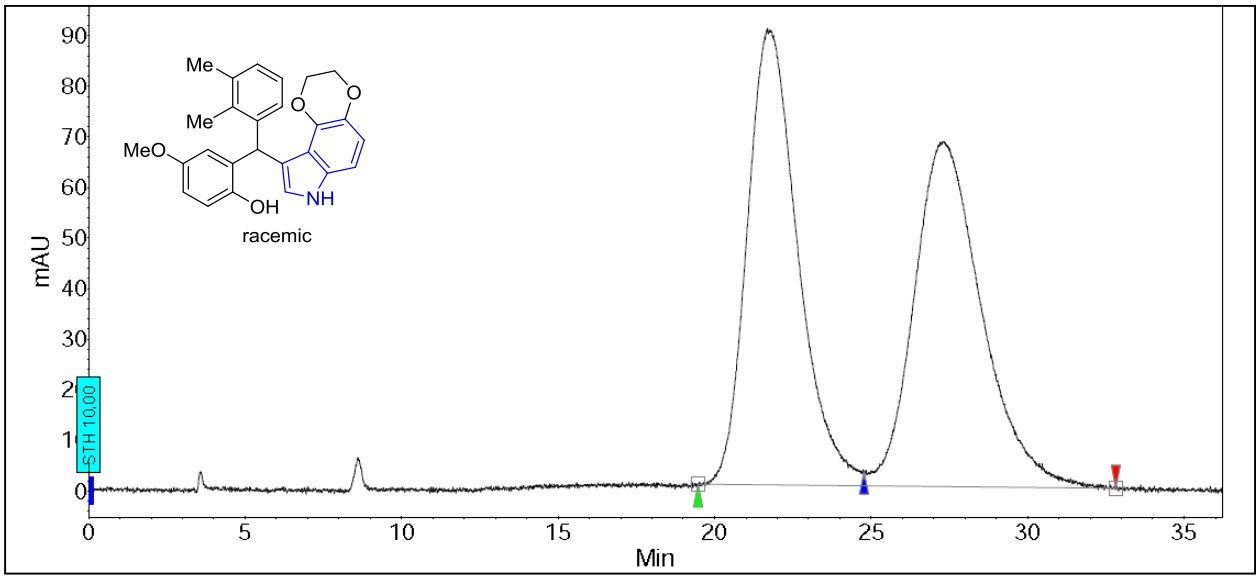
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		10.627	55651	1493829.222	49.75
2		21.787	28927	1508647.471	50.25

AD-H column, 70:30 (Hex:IPA), 1 mL/min, 333 nm (er = 93:7)



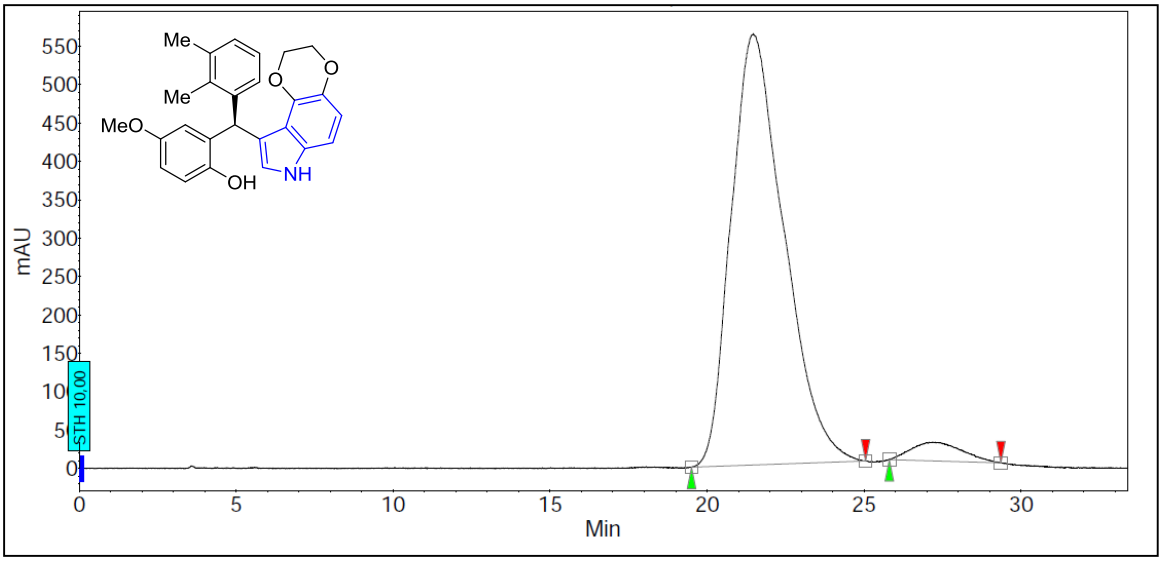
#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		10.587	30345	858566.013	6.71
2		21.267	207915	11945422.178	93.29

(Table 3, entry12)AS-H column, 85:15 (Hex:IPA), 1 mL/min, 232 nm



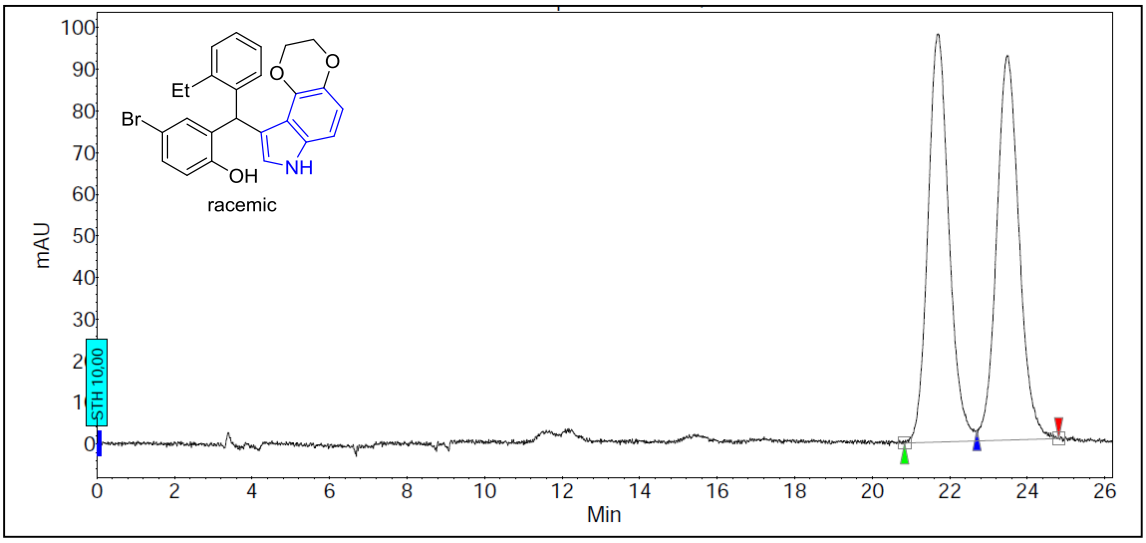
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	21,745	49,75	89,9	169,0	49,747
2	UNKNOWN	27,305	50,25	68,3	170,7	50,253
Total			100,00	158,2	339,7	100,000

AS-H column, 85:15 (Hex:IPA), 1 mL/min, 232 nm(er = 96:4)



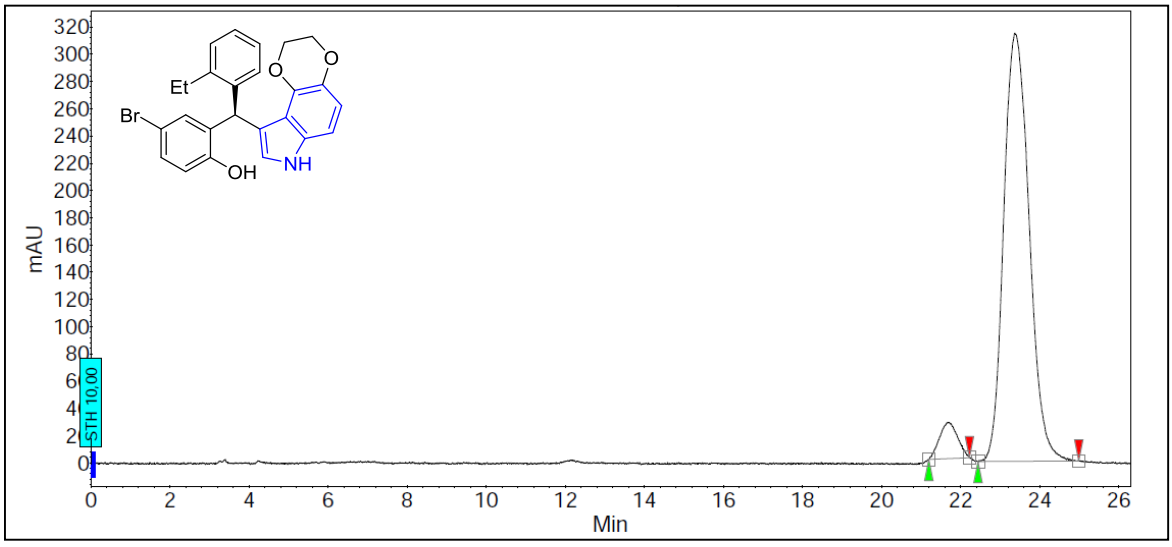
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	21,465	95,96	562,3	1096,4	95,963
2	UNKNOWN	27,238	4,04	24,1	46,1	4,037
Total			100,00	586,4	1142,5	100,000

(Table 3, entry13)IA column, 90:10 (Hex:IPA), 1 mL/min, 232 nm



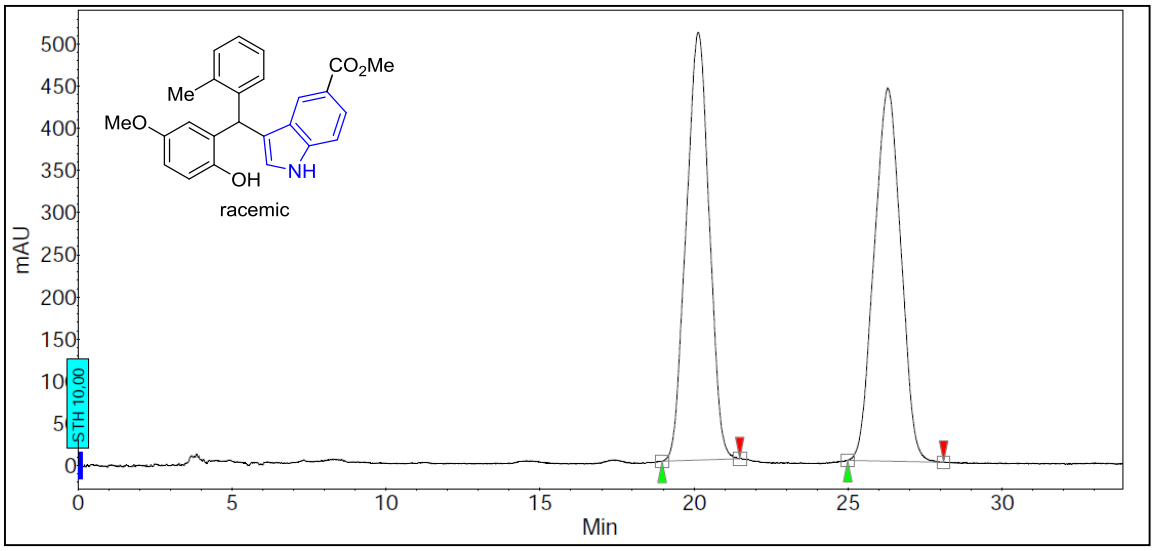
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	21,692	49,80	98,0	61,5	49,804
2	UNKNOWN	23,478	50,20	92,3	62,0	50,196
Total			100,00	190,4	123,5	100,000

IA column, 90:10 (Hex:IPA), 1 mL/min, 232 nm(er = 94:6)



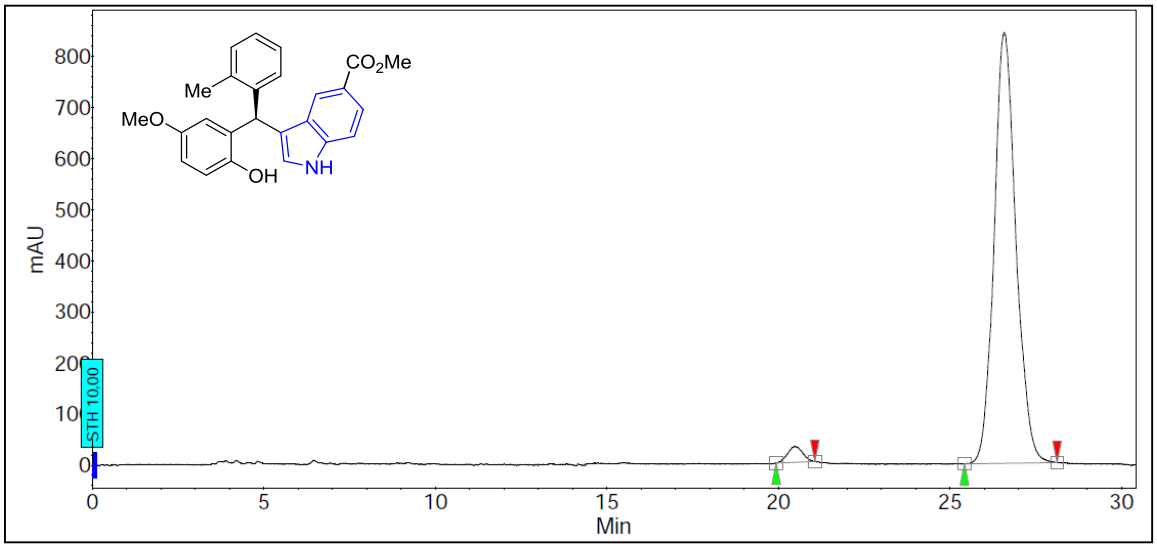
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
2	UNKNOWN	21,692	5,68	26,4	14,1	5,685
1	UNKNOWN	23,372	94,32	314,3	234,1	94,315
Total			100,00	340,7	248,2	100,000

(Table 3, entry14)IA column, 90:10 (Hex:IPA), 1 mL/min, 232 nm



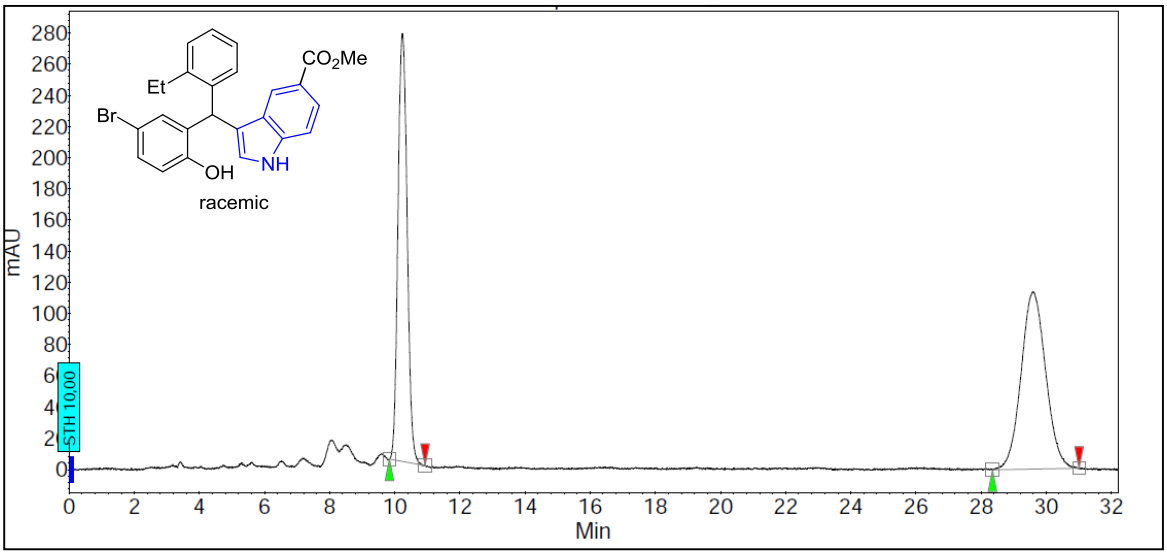
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	20,132	49,72	507,5	439,0	49,724
2	UNKNOWN	26,278	50,28	443,1	443,9	50,276
Total			100,00	950,6	882,9	100,000

IA column, 90:10 (Hex:IPA), 1 mL/min, 232 nm(er = 98:2)



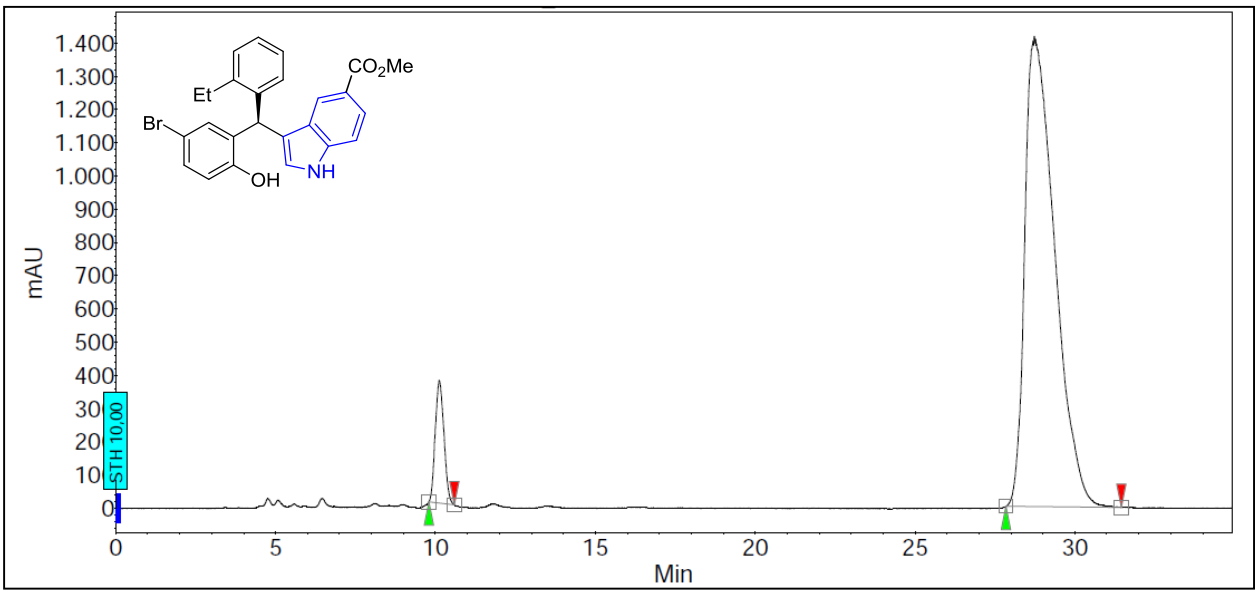
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	20,492	2,46	30,4	15,7	2,458
2	UNKNOWN	26,571	97,54	843,7	621,7	97,542
Total			100,00	874,1	637,4	100,000

(Table 3, entry15)IA column, 80:20 (Hex:IPA), 1 mL/min, 232 nm



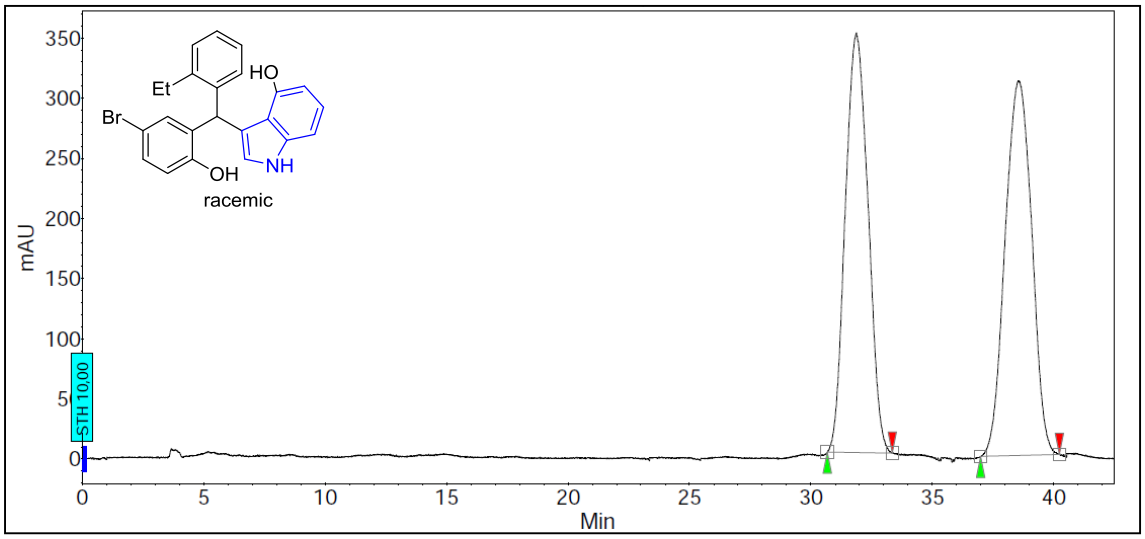
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	10,239	47,39	274,3	89,7	47,385
2	UNKNOWN	29,585	52,61	113,2	99,6	52,615
Total			100,00	387,5	189,2	100,000

IA column, 80:20 (Hex:IPA), 1 mL/min, 232 nm(er = 93:7)



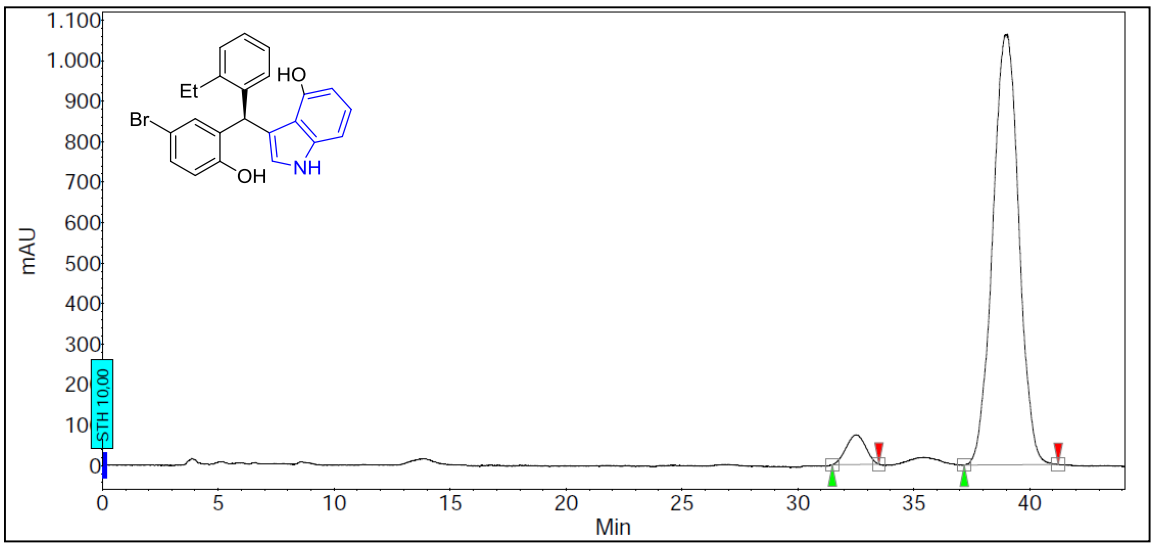
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	10,119	7,07	369,0	116,6	7,073
2	UNKNOWN	28,731	92,93	1415,9	1531,9	92,927
Total			100,00	1784,9	1648,5	100,000

(Table 3, entry16)IA column, 90:10 (Hex:IPA), 1 mL/min, 232 nm



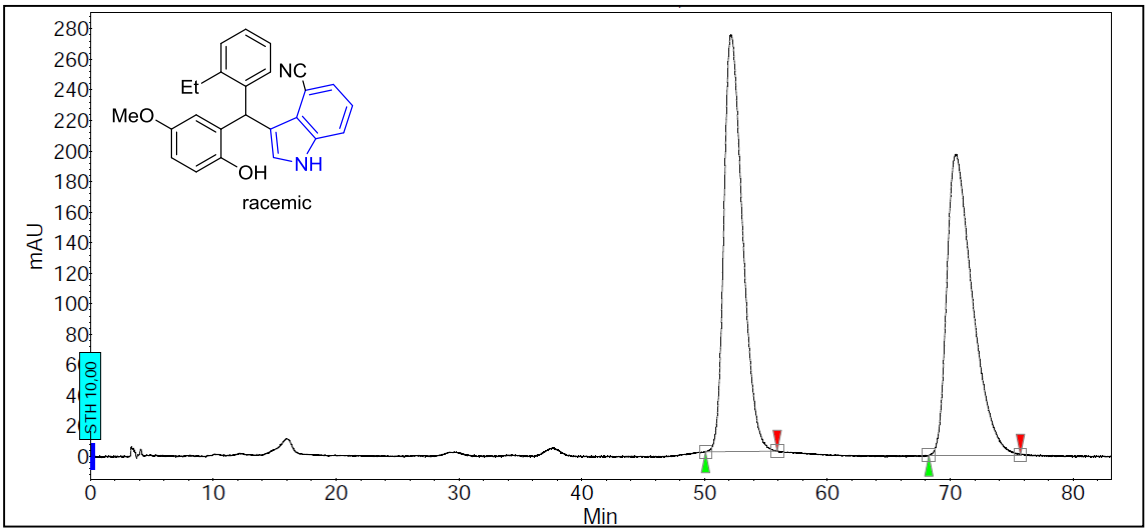
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	31,864	48,88	348,5	378,8	48,885
2	UNKNOWN	38,544	51,12	311,3	396,1	51,115
Total			100,00	659,9	774,8	100,000

IA column, 90:10 (Hex:IPA), 1 mL/min, 232 nm (er = 95:5)



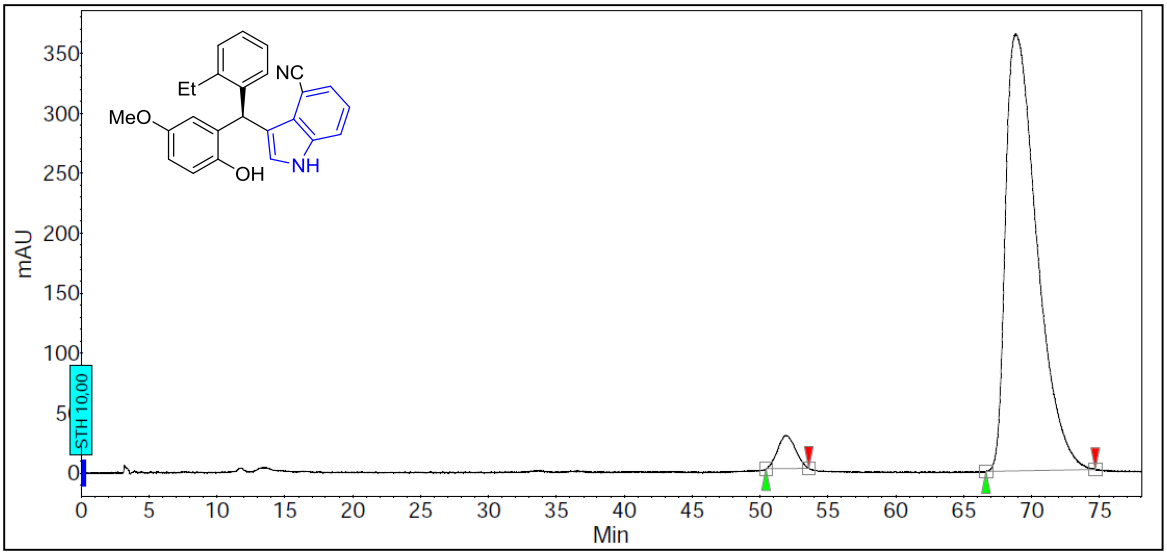
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	32,504	4,87	73,0	70,9	4,874
2	UNKNOWN	39,024	95,13	1063,3	1384,1	95,126
Total			100,00	1136,3	1455,0	100,000

(Table 3, entry17)IA column, 90:10 (Hex:IPA), 1 mL/min, 232 nm



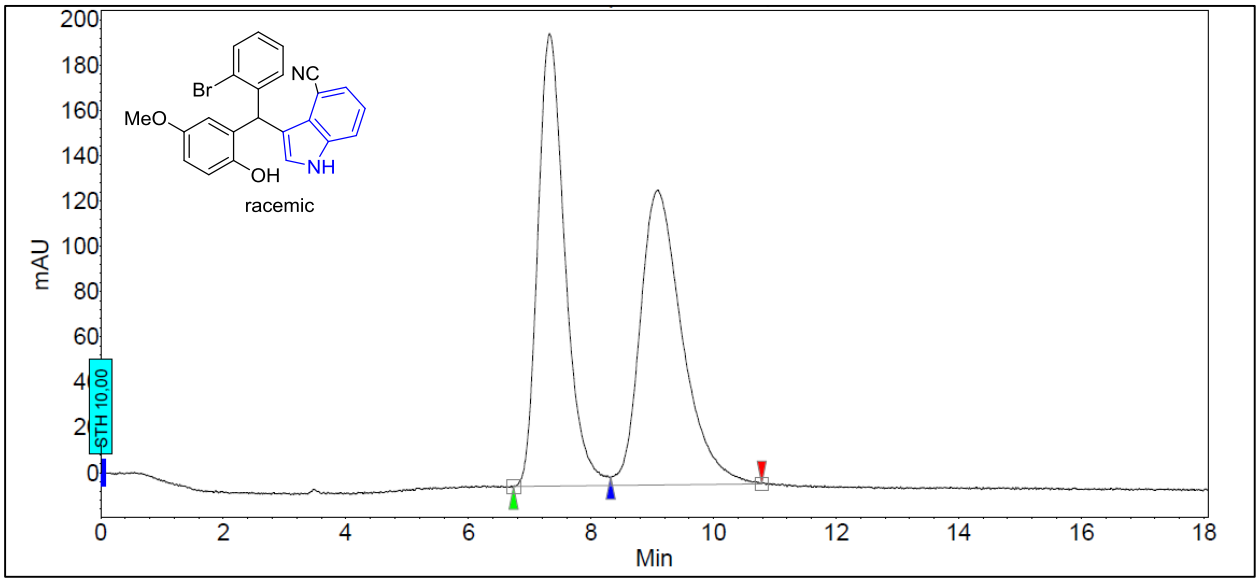
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	52,155	50,05	273,2	480,2	50,049
2	UNKNOWN	70,468	49,95	197,1	479,2	49,951
Total			100,00	470,3	959,4	100,000

IA column, 90:10 (Hex:IPA), 1 mL/min, 232 nm(er = 96:4)



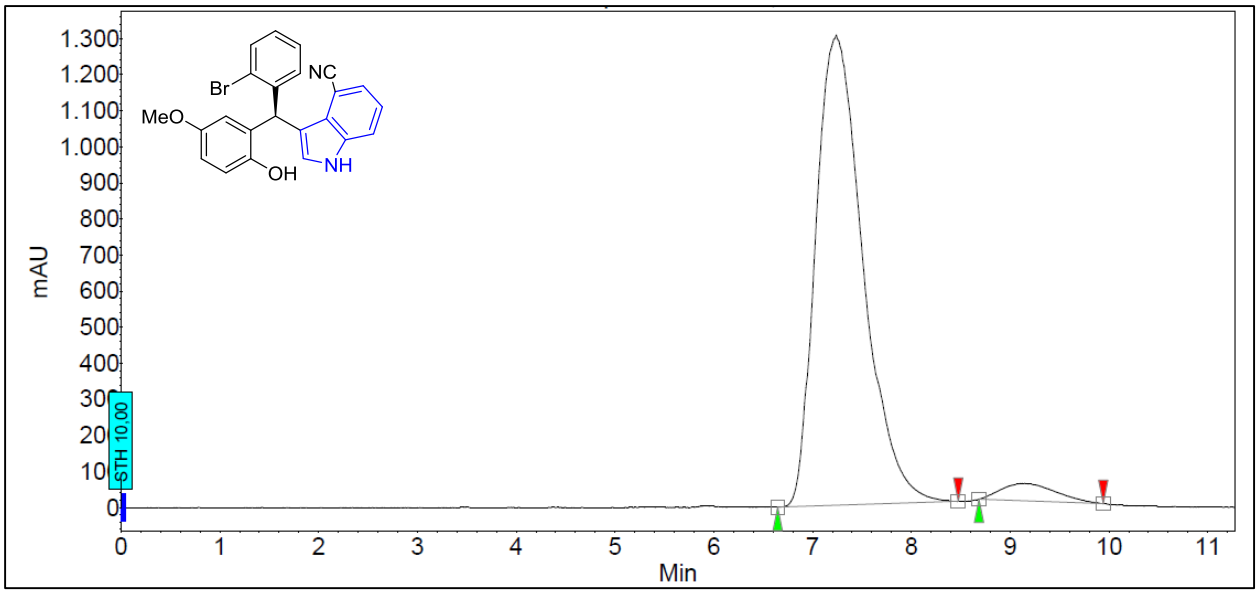
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	51,915	4,13	27,4	41,9	4,133
2	UNKNOWN	68,842	95,87	364,6	971,3	95,867
Total			100,00	392,0	1013,1	100,000

(Table 3, entry 18)OD-Hcolumn, 80:20 (Hex:IPA), 1 mL/min, 244nm



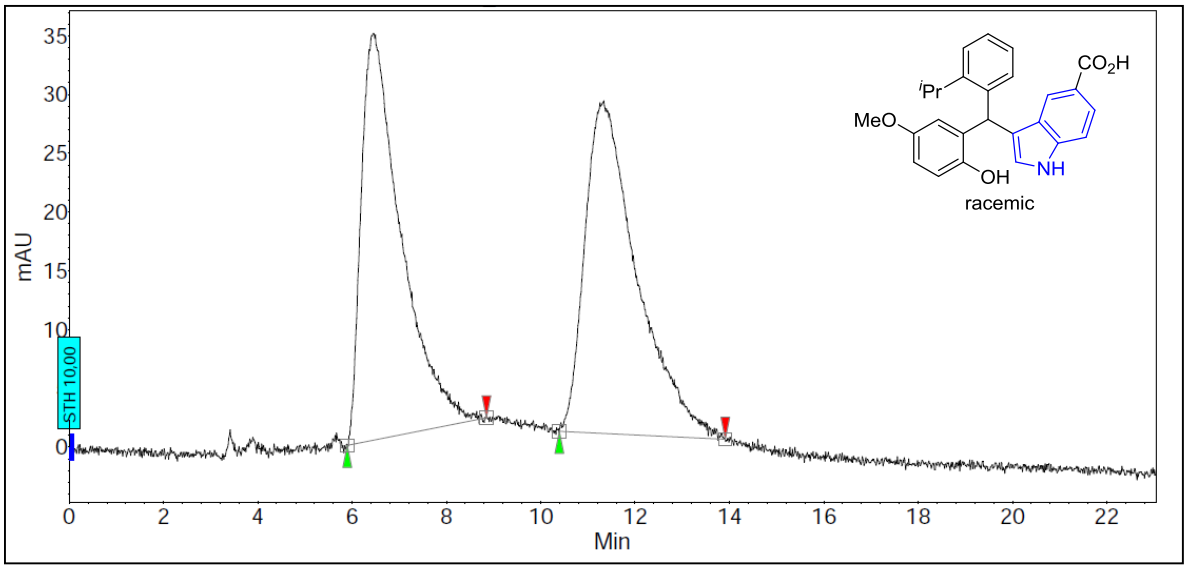
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	7,319	49,77	199,7	102,2	49,775
2	UNKNOWN	9,079	50,23	129,9	103,1	50,225
Total			100,00	329,5	205,3	100,000

OD-H column, 80:20 (Hex:IPA), 1 mL/min, 244 nm(er = 96:4)



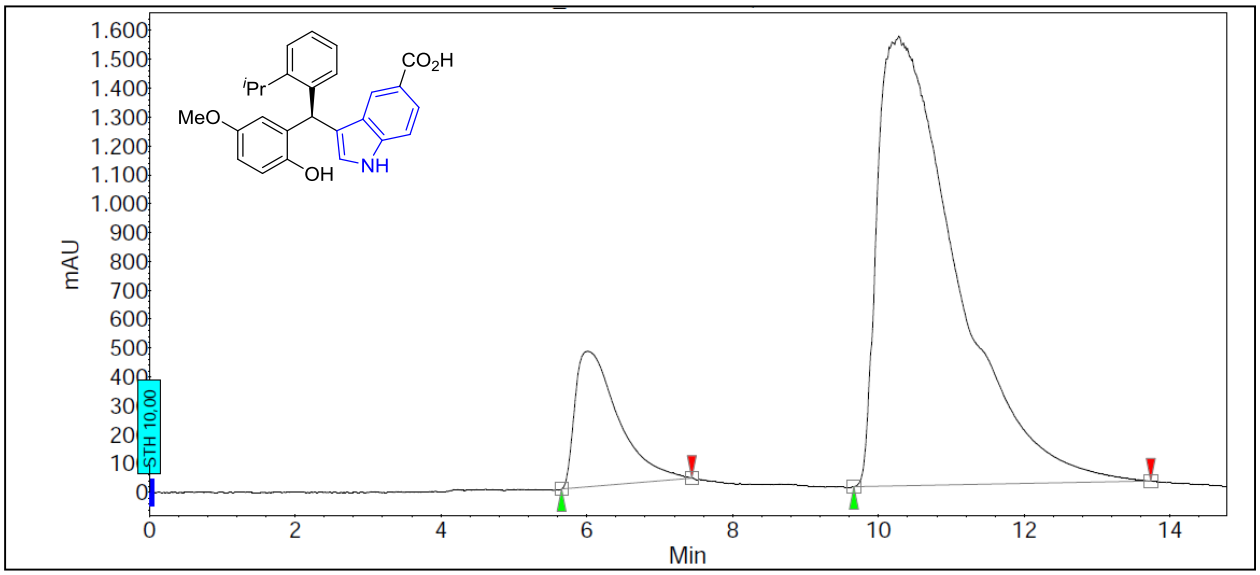
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	7,240	95,89	1303,0	719,3	95,889
2	UNKNOWN	9,160	4,11	47,9	30,8	4,111
Total			100,00	1350,9	750,2	100,000

(Table 3, entry19)IA column, 70:30 (Hex:IPA), 1 mL/min, 244 nm



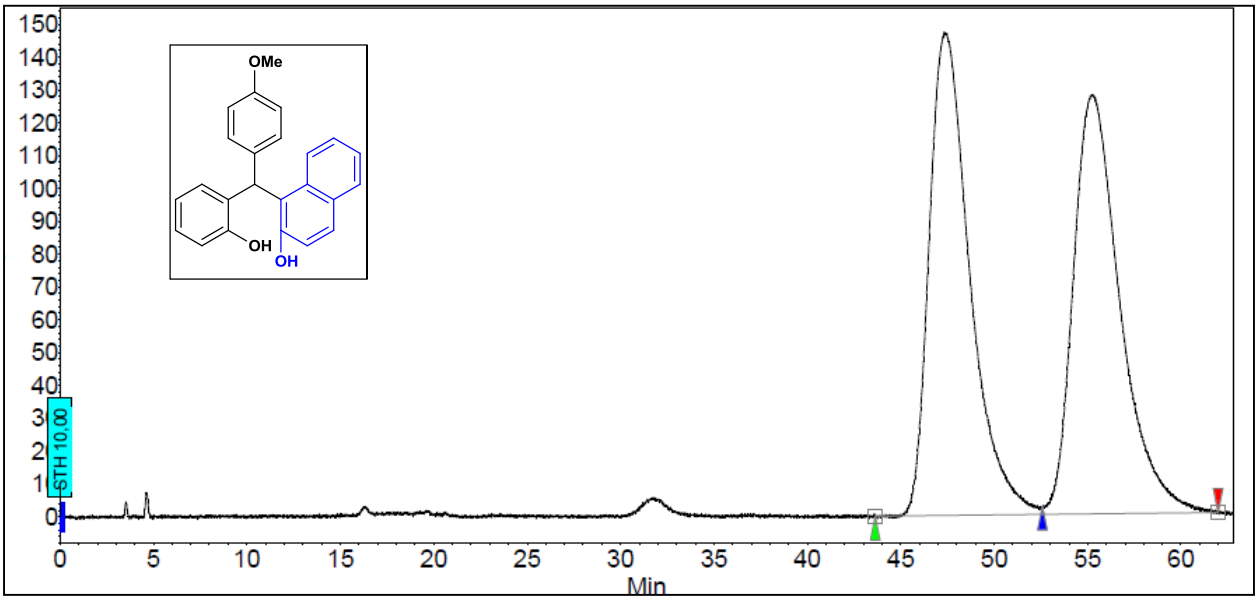
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
2	UNKNOWN	6,440	48,41	34,6	33,9	48,408
1	UNKNOWN	11,306	51,59	28,1	36,1	51,592
Total			100,00	62,7	70,0	100,000

IA column, 70:30 (Hex:IPA), 1 mL/min, 244 nm(er = 86:14)

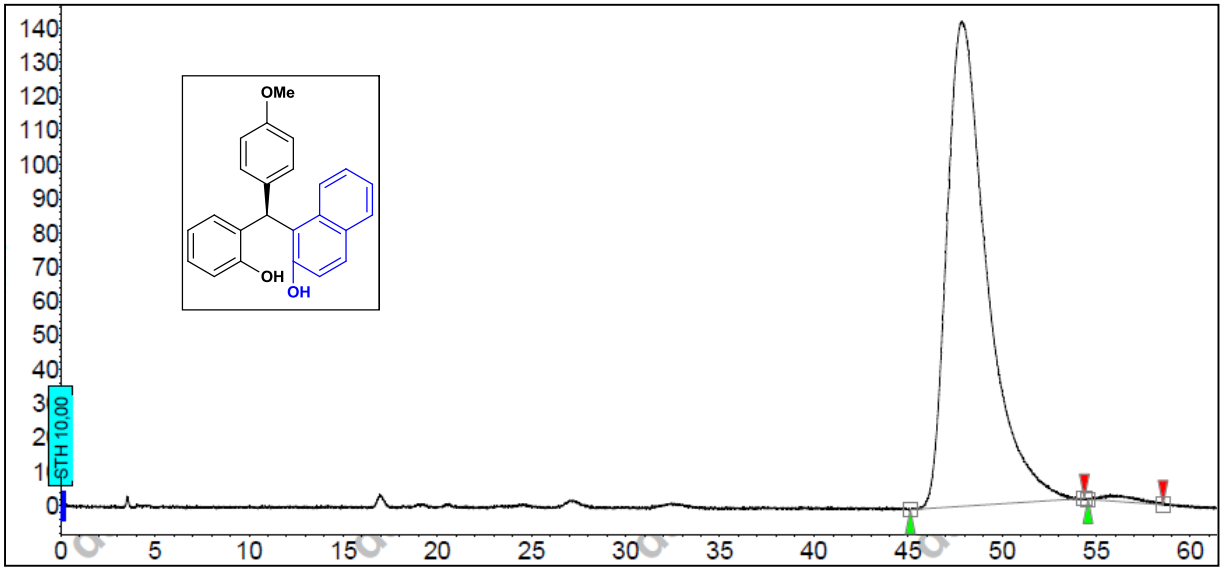


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	6,000	14,14	469,0	321,7	14,142
2	UNKNOWN	10,280	85,86	1558,5	1953,1	85,858
Total			100,00	2027,5	2274,8	100,000

(Table 4, entry 1) AS-H column, 95:5 (Hex:IPA), 1 mL/min, 230 nm

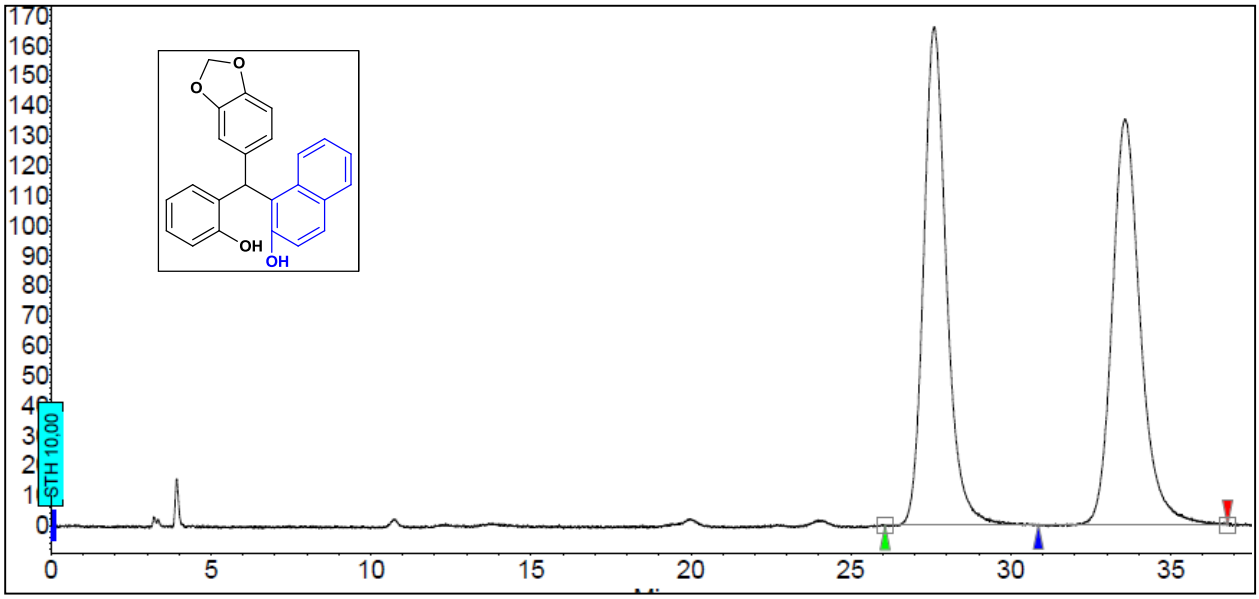


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	47,383	50,03	146,8	370,1	50,027
2	UNKNOWN	55,249	49,97	127,6	369,7	49,973
Total			100,00	274,4	739,8	100,000

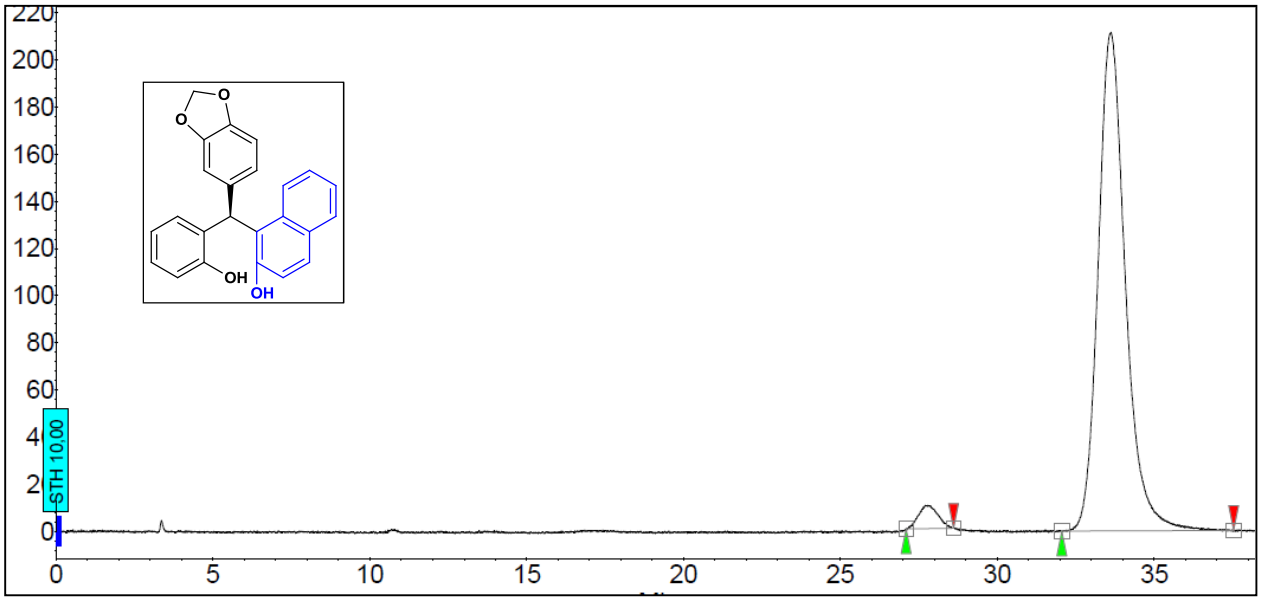


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	47,877	98,92	142,1	350,2	98,917
2	UNKNOWN	56,223	1,08	1,6	3,8	1,083
Total			100,00	143,7	354,0	100,000

(Table 4, entry 2), IA column, 90:10 (Hex:IPA), 1 mL/min, 230 nm

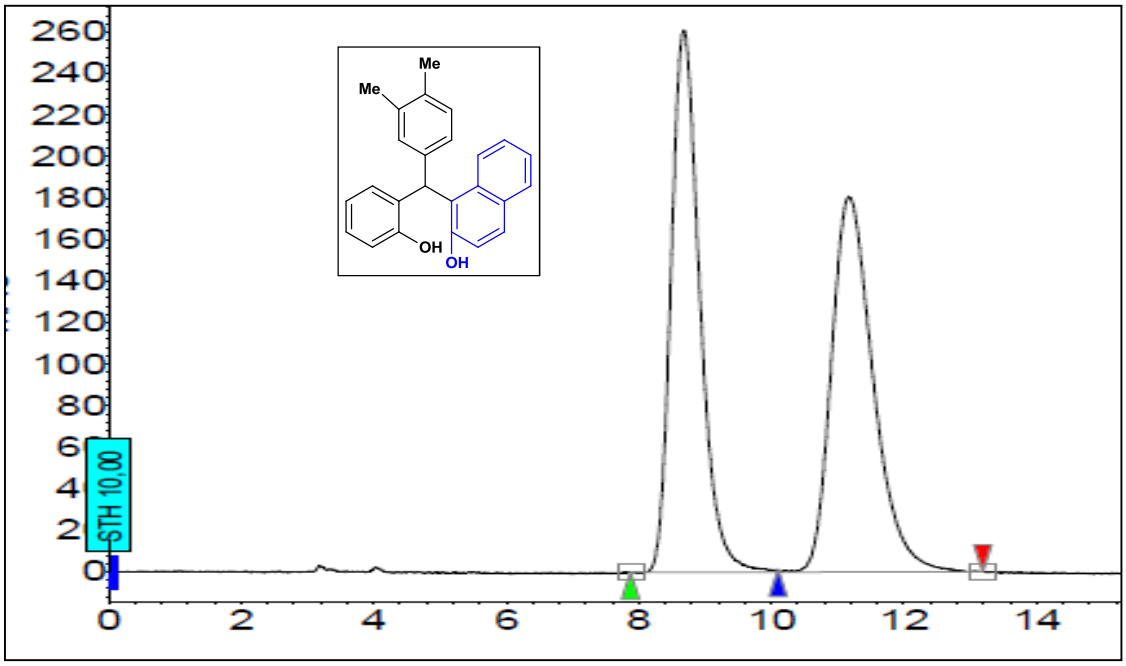


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	27,598	49,82	166,0	137,5	49,819
2	UNKNOWN	33,571	50,18	134,9	138,5	50,181
Total			100,00	300,9	276,0	100,000

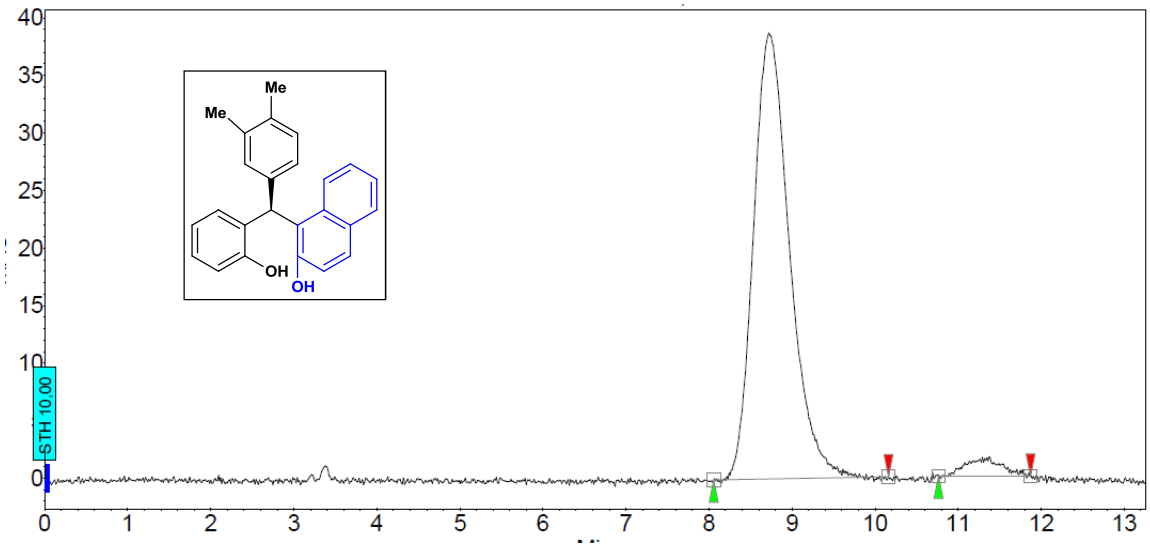


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	27,771	3,23	9,5	7,1	3,227
2	UNKNOWN	33,611	96,77	211,3	213,5	96,773
Total			100,00	220,8	220,6	100,000

(Table 4, entry 3), OD-H column, 90:10 (Hex:IPA), 1 mL/min, 240 nm

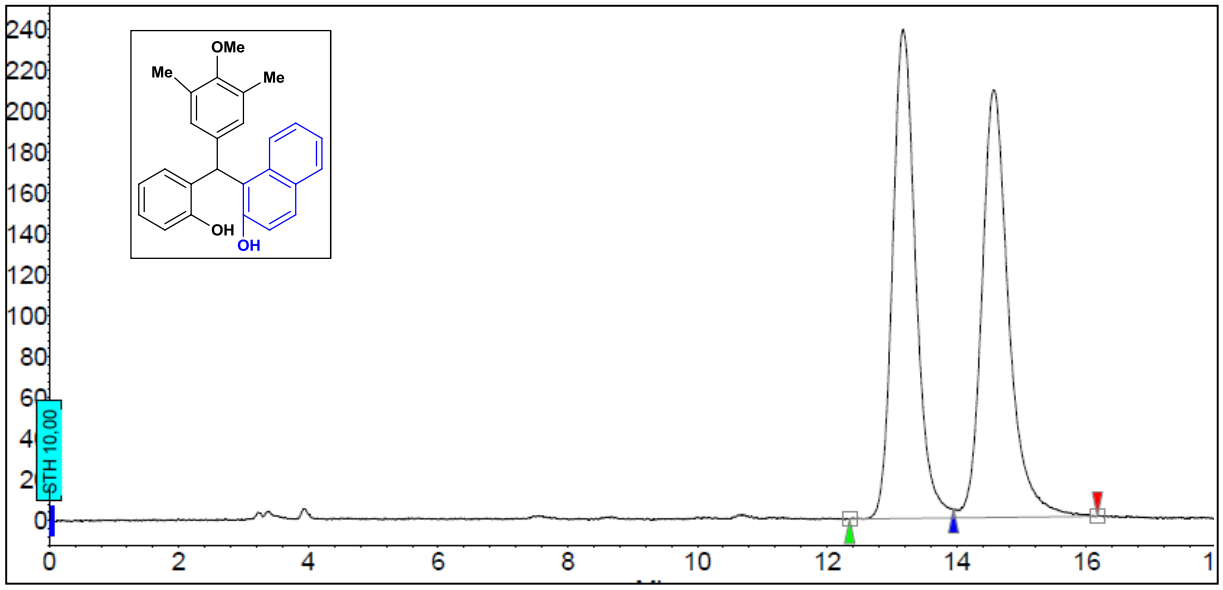


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	8,679	49,84	261,2	131,3	49,838
2	UNKNOWN	11,173	50,16	180,6	132,1	50,162
Total			100,00	441,8	263,4	100,000

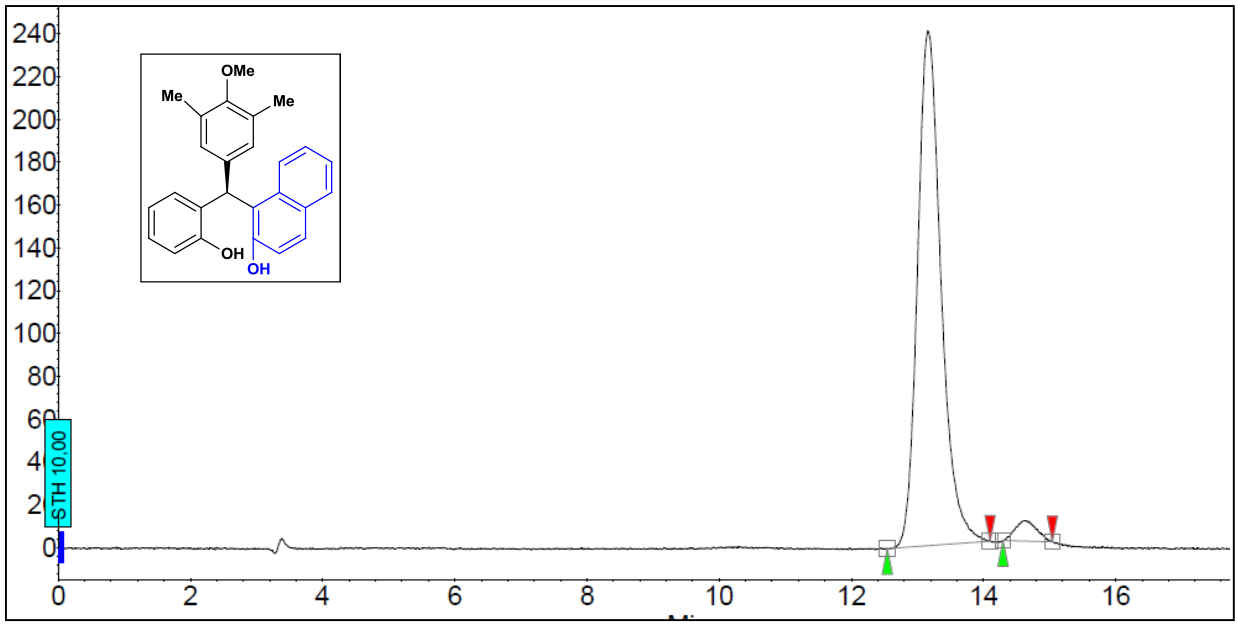


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	8,733	96,05	38,6	19,5	96,052
2	UNKNOWN	11,307	3,95	1,2	0,8	3,948
Total			100,00	39,8	20,3	100,000

(Table 4, entry 4), IA column, 90:10 (Hex:IPA), 1 mL/min, 230 nm

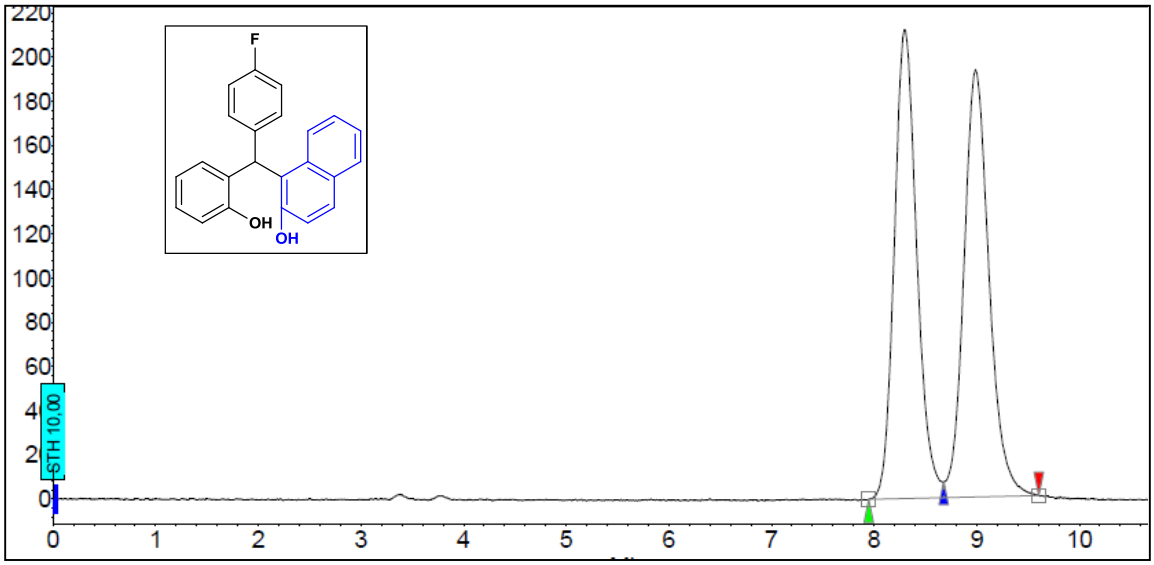


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	13,172	49,49	239,1	98,6	49,488
2	UNKNOWN	14,572	50,51	208,8	100,6	50,512
Total			100,00	447,9	199,1	100,000

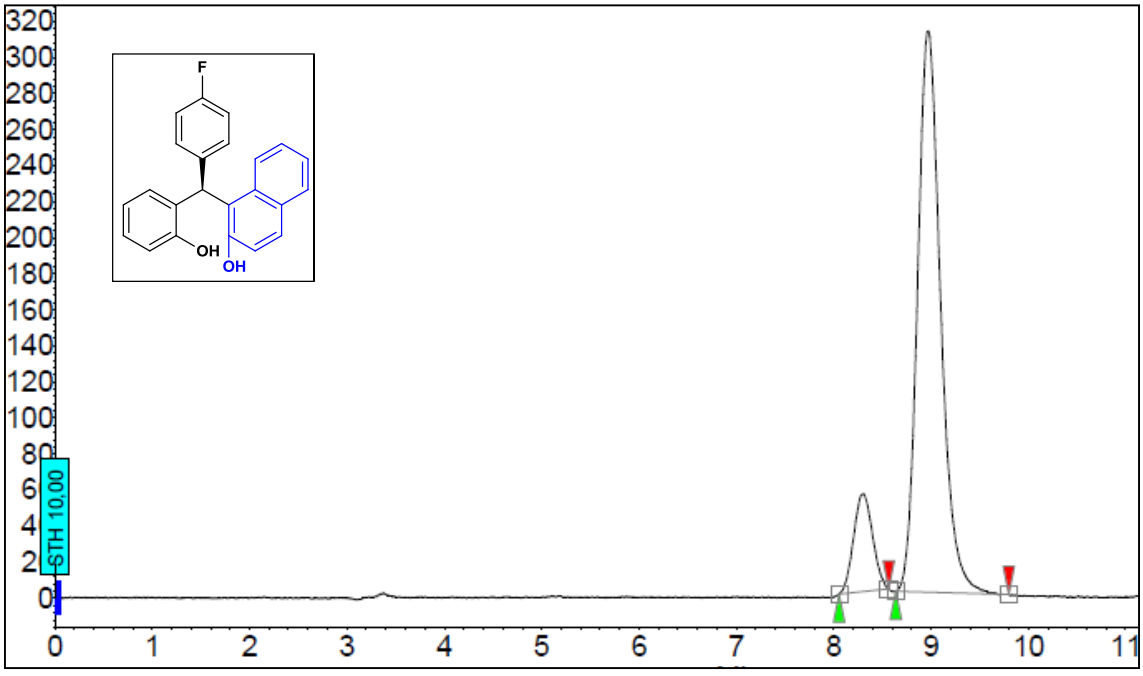


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	13,159	96,49	240,5	99,0	96,490
2	UNKNOWN	14,639	3,51	9,6	3,6	3,510
Total			100,00	250,0	102,6	100,000

(Table 4, entry 5), IA column, 80:20 (Hex:IPA), 1 mL/min, 240 nm

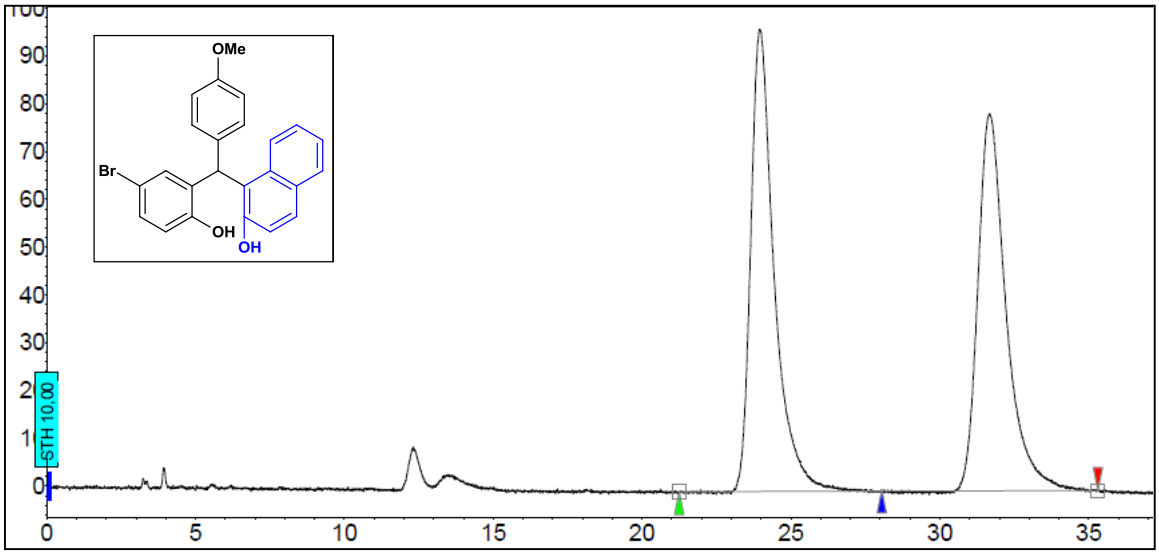


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	8,293	49,44	212,1	53,7	49,440
2	UNKNOWN	8,987	50,56	193,3	55,0	50,560
Total			100,00	405,4	108,7	100,000

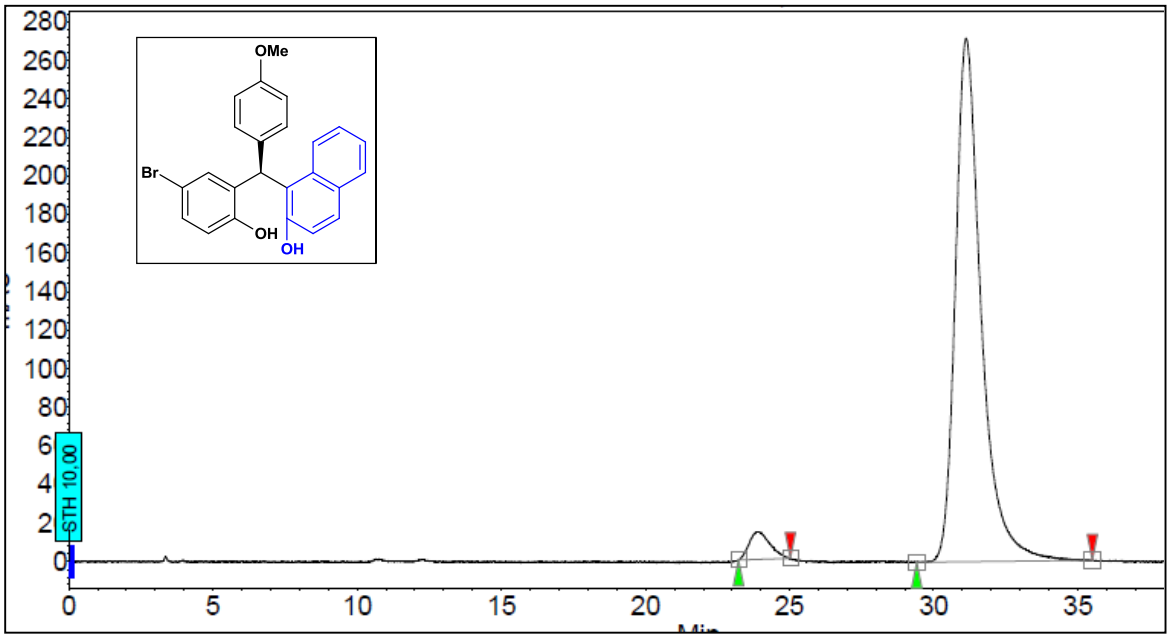


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	8,293	12,52	54,1	12,3	12,523
2	UNKNOWN	8,973	87,48	311,2	85,6	87,477
Total			100,00	365,3	97,9	100,000

(Table 4, entry 6), IA column, 90:10 (Hex:IPA), 1 mL/min, 236 nm

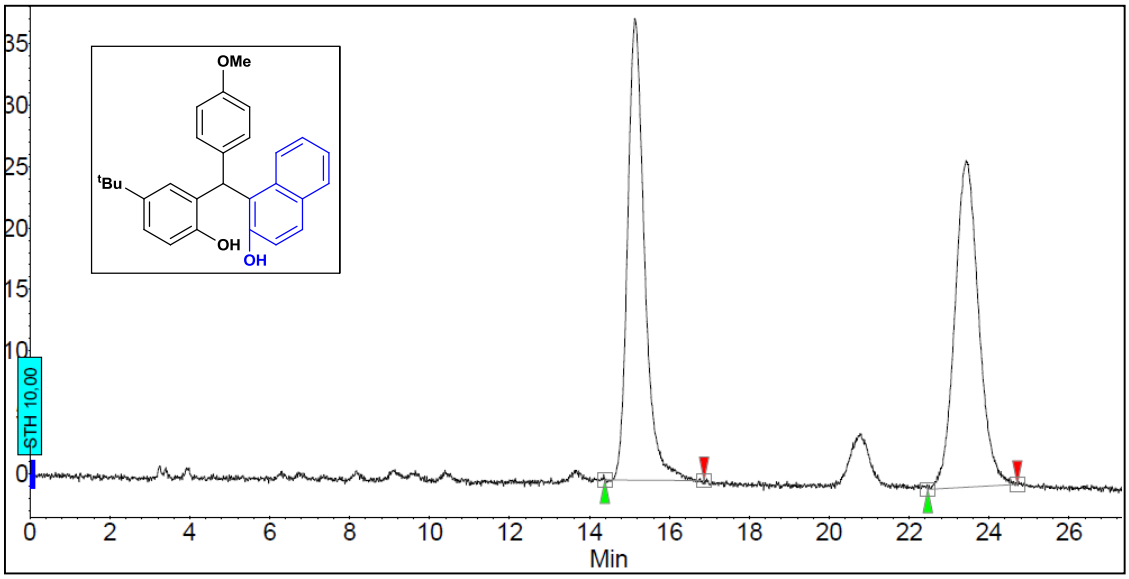


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	23,958	50,56	96,6	88,1	50,559
2	UNKNOWN	31,664	49,44	78,8	86,1	49,441
Total			100,00	175,4	174,2	100,000

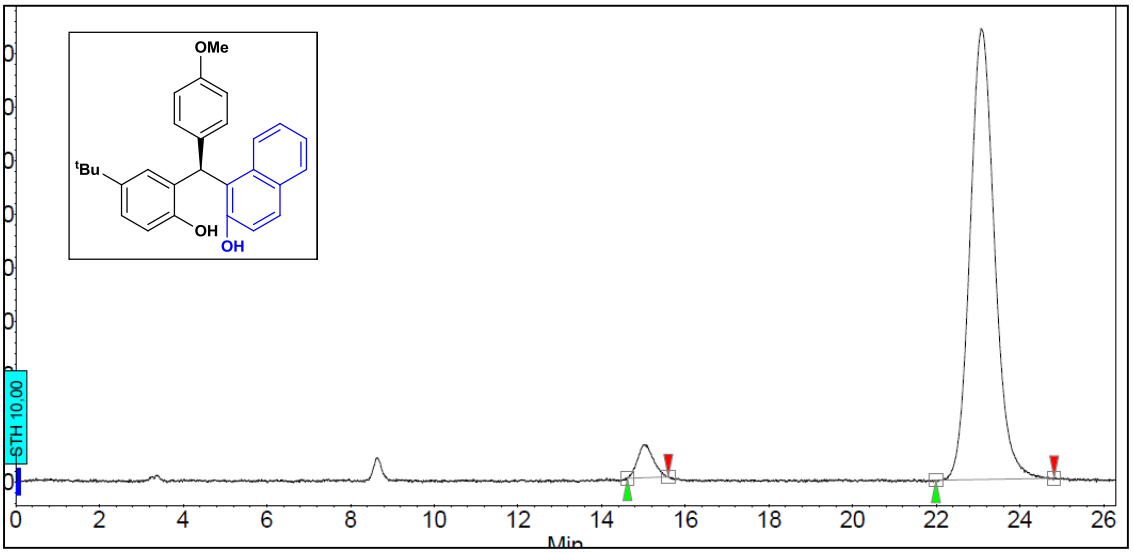


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	23,905	4,03	14,3	11,9	4,032
2	UNKNOWN	31,118	95,97	271,5	282,3	95,968
Total			100,00	285,9	294,1	100,000

(Table 4, entry 7), IA column, 90:10 (Hex:IPA), 1 mL/min, 242 nm

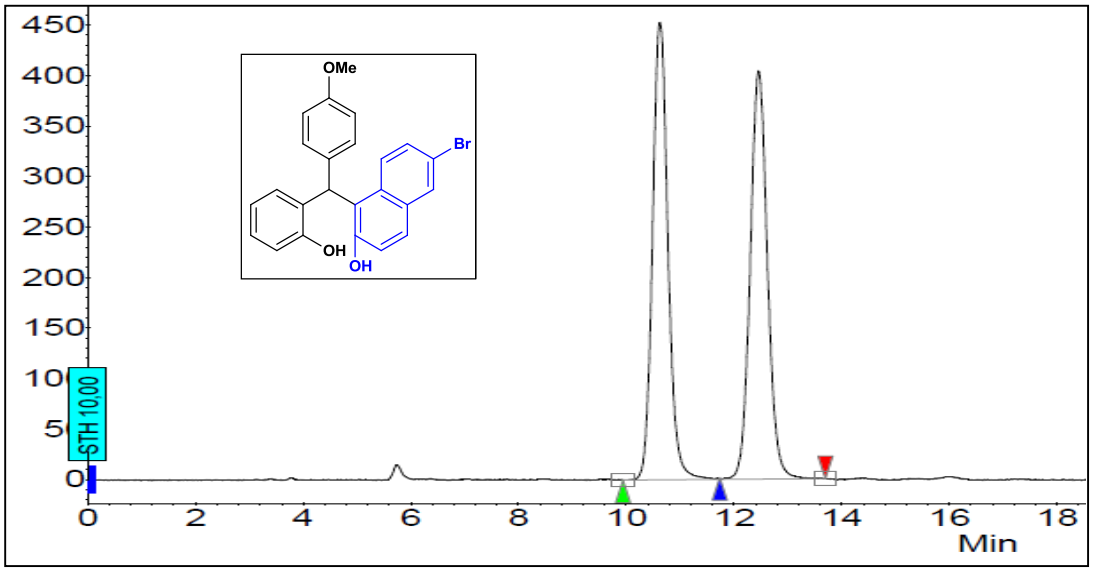


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	15,146	49,94	37,5	18,3	49,935
2	UNKNOWN	23,425	50,06	26,6	18,4	50,065
Total			100,00	64,0	36,7	100,000

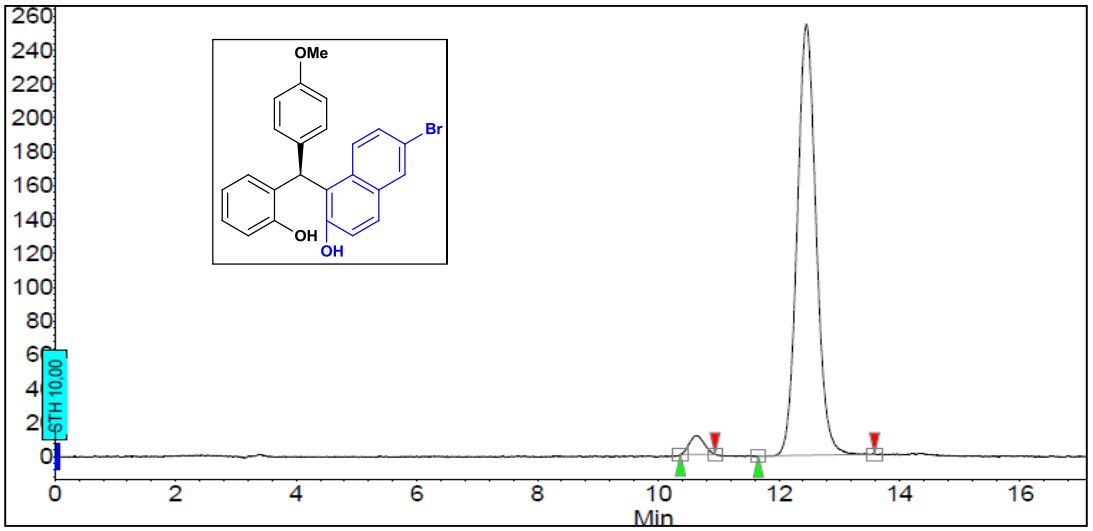


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	15,039	4,48	6,1	2,7	4,481
2	UNKNOWN	23,078	95,52	84,1	56,9	95,519
Total			100,00	90,2	59,6	100,000

(Table 4, entry 8), IA column, 80:20 (Hex:IPA), 1 mL/min, 244 nm

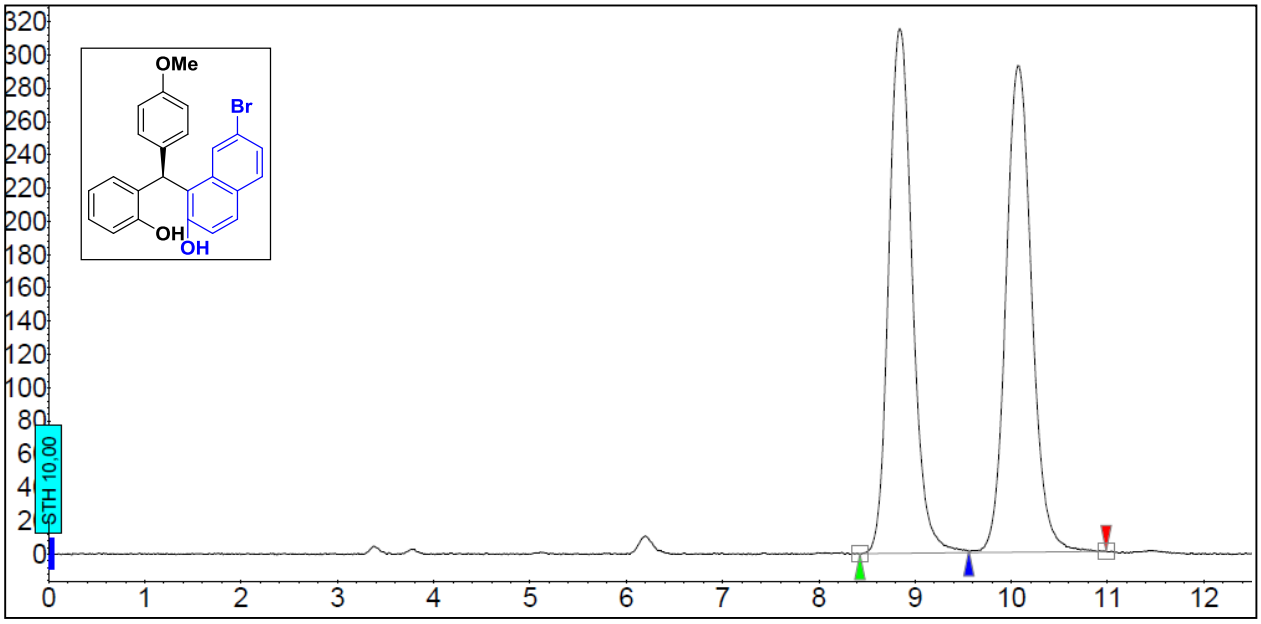


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	10,626	50,08	452,5	150,4	50,077
2	UNKNOWN	12,452	49,92	404,0	149,9	49,923
Total			100,00	856,5	300,3	100,000

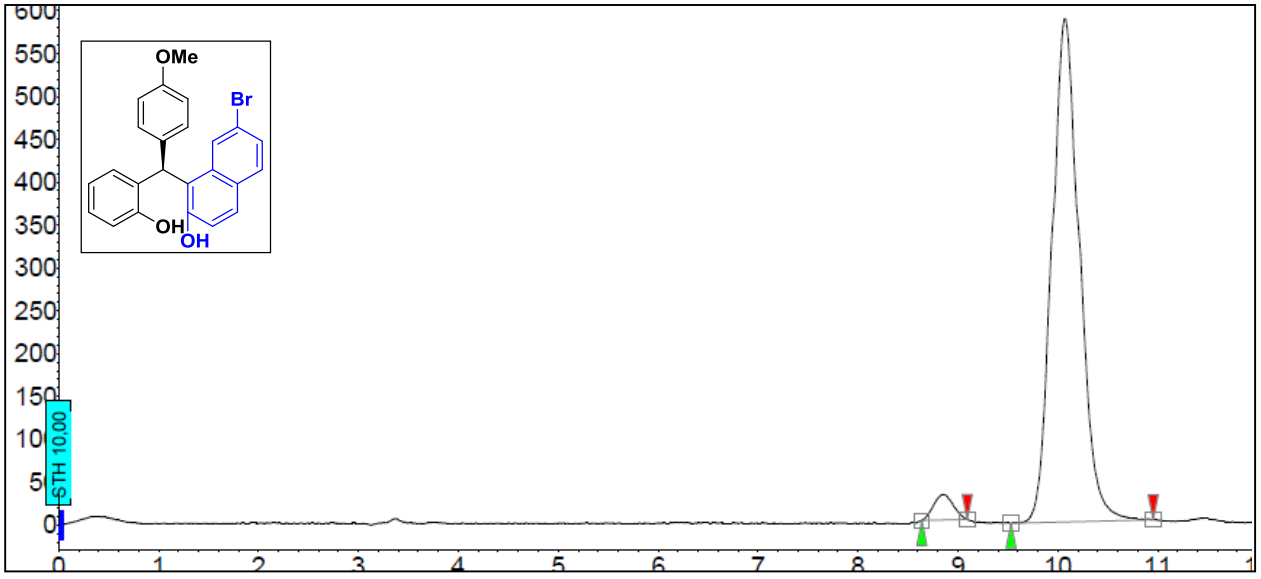


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
2	UNKNOWN	10,626	3,29	11,2	3,1	3,287
1	UNKNOWN	12,452	96,71	254,5	92,3	96,713
Total			100,00	265,7	95,5	100,000

(Table 4, entry 9), IA column, 80:20 (Hex:IPA), 1 mL/min, 230 nm

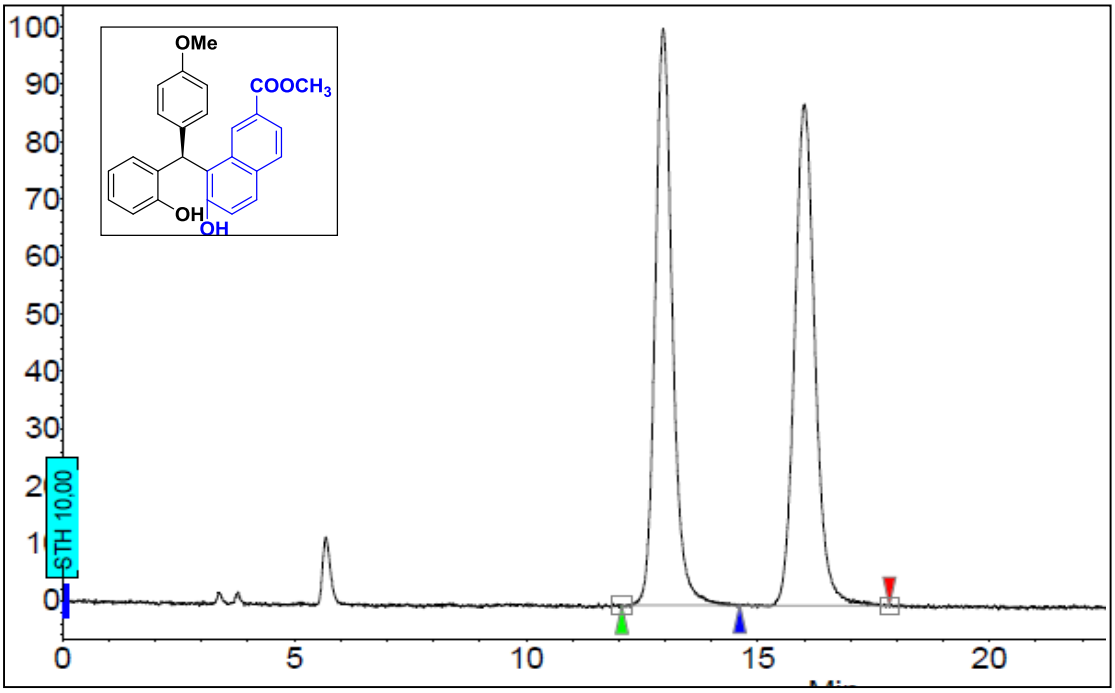


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	8,840	49,59	315,4	89,5	49,592
2	UNKNOWN	10,067	50,41	292,4	91,0	50,408
Total			100,00	607,8	180,5	100,000

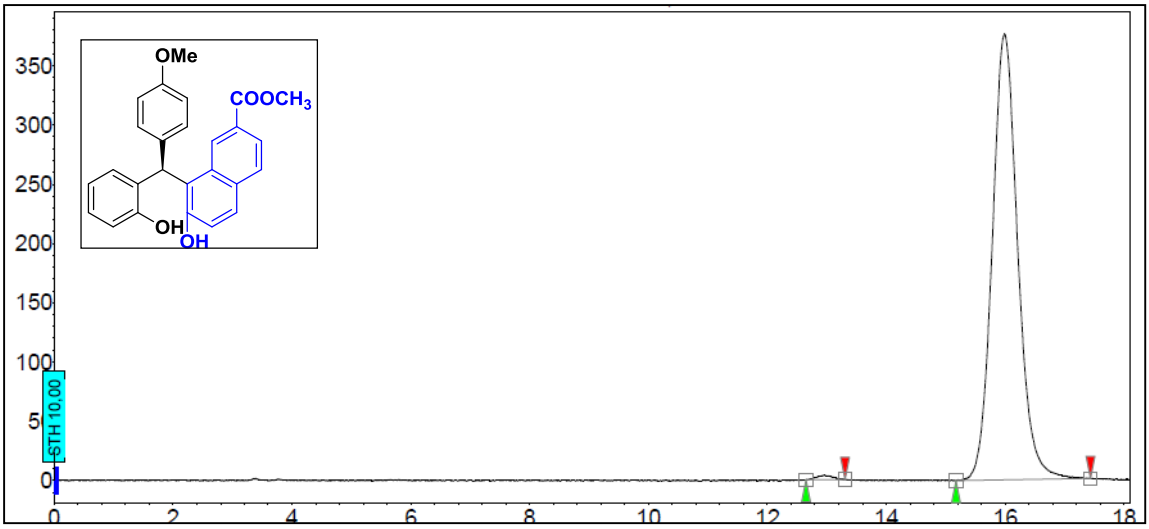


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
2	UNKNOWN	8,853	3,47	29,8	6,8	3,468
1	UNKNOWN	10,067	96,53	587,1	188,1	96,532
Total			100,00	617,0	194,9	100,000

(Table 4, entry 10), IA column, 80:20 (Hex:IPA), 1 mL/min, 240 nm

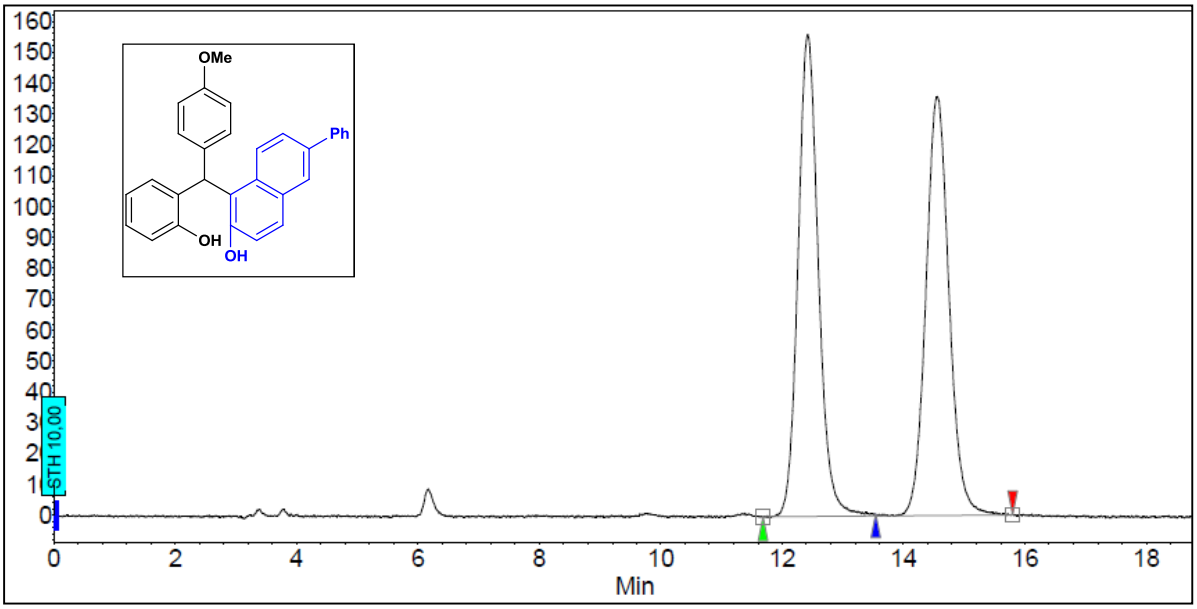


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	12,959	49,36	100,6	43,4	49,361
2	UNKNOWN	15,999	50,64	87,3	44,5	50,639
Total			100,00	187,9	87,9	100,000

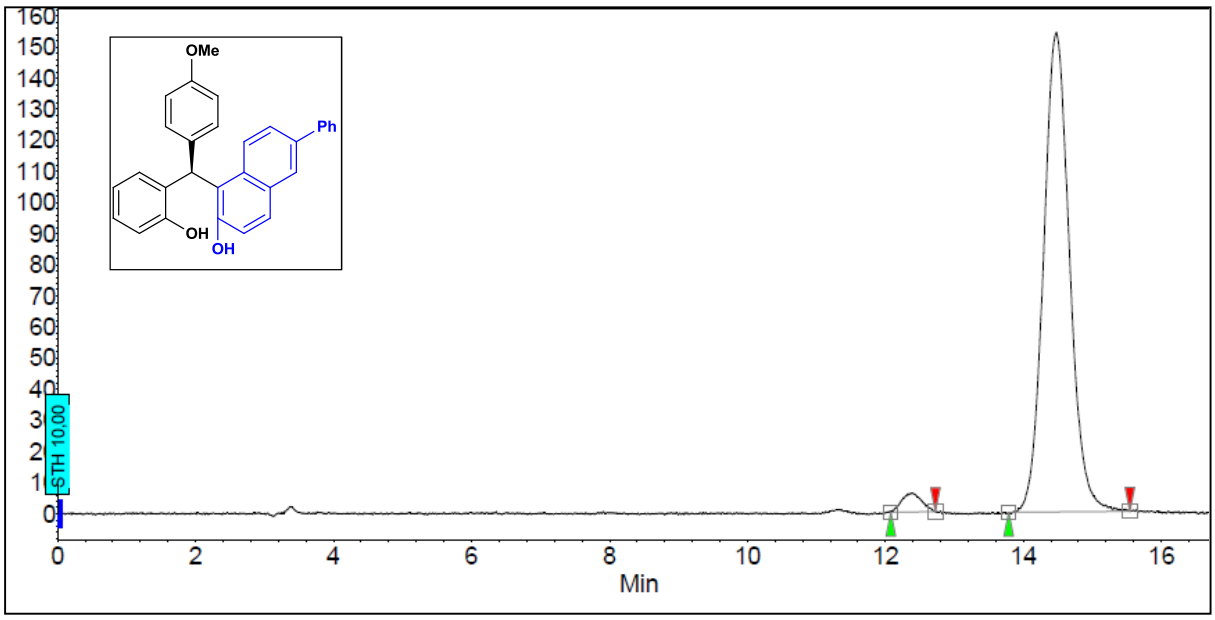


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
2	UNKNOWN	12,972	0,59	3,4	1,1	0,587
1	UNKNOWN	15,986	99,41	376,8	186,7	99,413
Total			100,00	380,2	187,8	100,000

(Table 4, entry 11), IA column, 80:20 (Hex:IPA), 1 mL/min, 240 nm

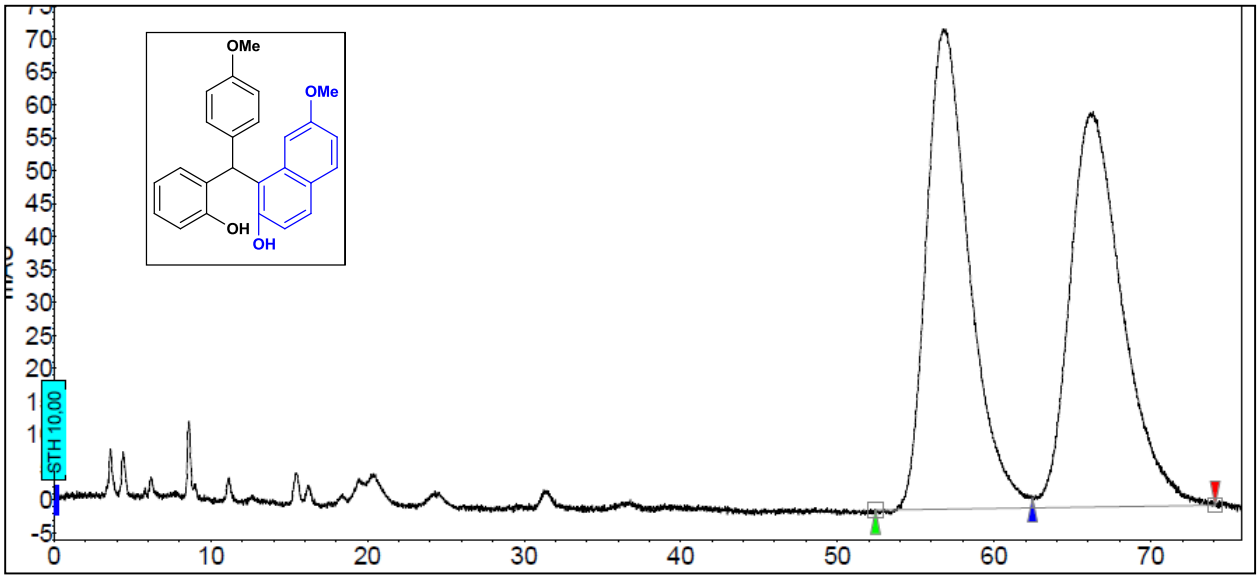


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	12,426	50,30	156,1	60,7	50,297
2	UNKNOWN	14,559	49,70	135,7	60,0	49,703
Total			100,00	291,9	120,8	100,000

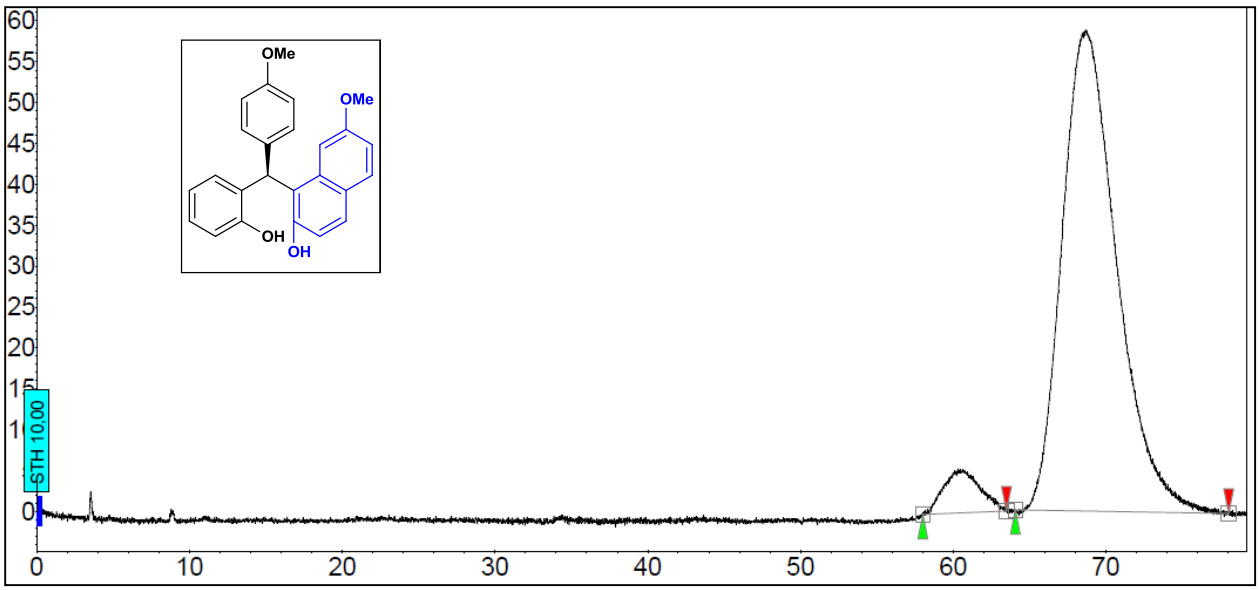


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	12,372	2,92	6,0	2,0	2,922
2	UNKNOWN	14,479	97,08	154,1	67,3	97,078
Total			100,00	160,1	69,3	100,000

(Table 4, entry 12), AS-H column, 95:05 (Hex:IPA), 1 mL/min, 230 nm

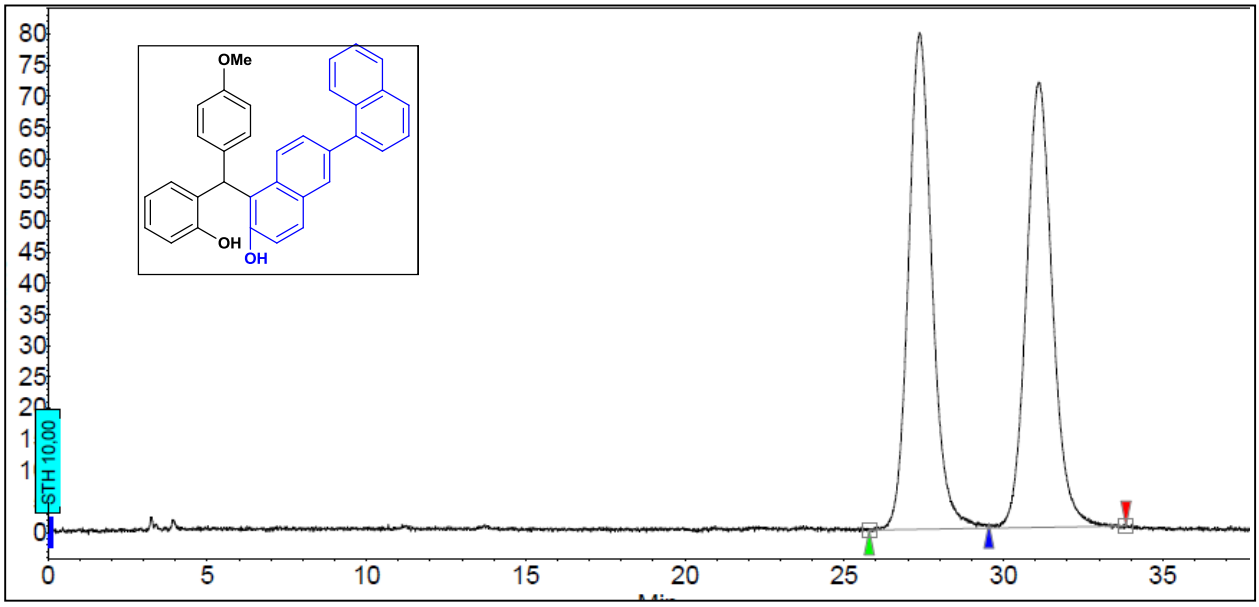


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	56,806	49,75	72,9	230,4	49,749
2	UNKNOWN	66,203	50,25	59,2	232,7	50,251
Total			100,00	132,1	463,1	100,000

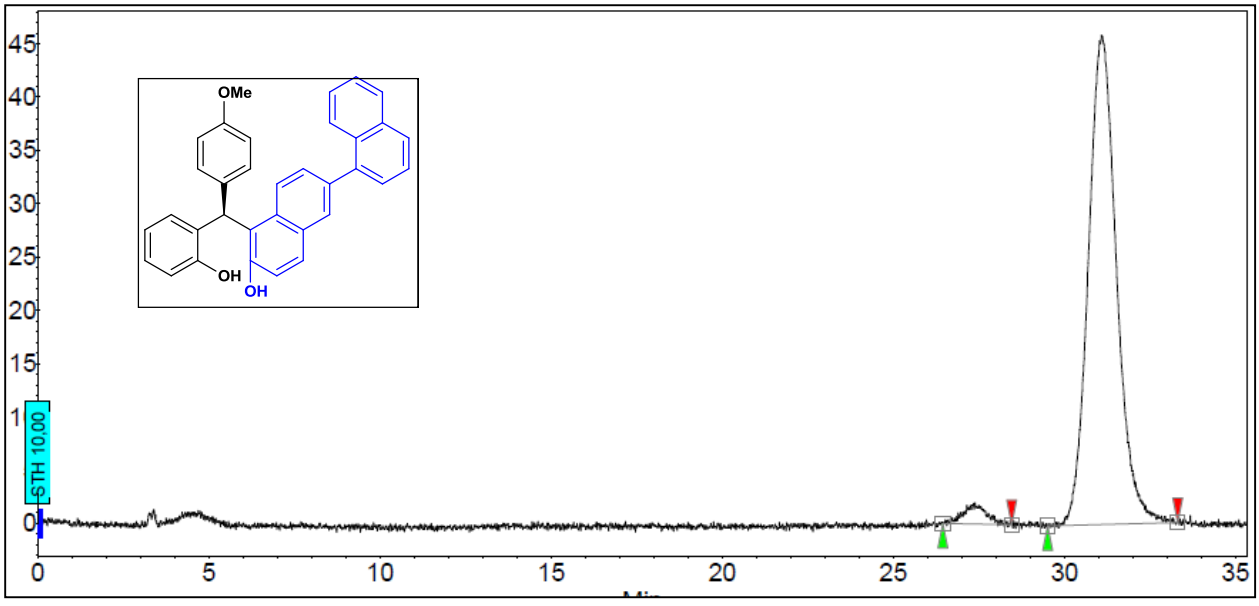


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
2	UNKNOWN	60,312	5,65	5,4	14,6	5,653
1	UNKNOWN	68,682	94,35	58,9	243,8	94,347
Total			100,00	64,2	258,4	100,000

(Table 4, entry 13), IA column, 90:10 (Hex:IPA), 1 mL/min, 240 nm

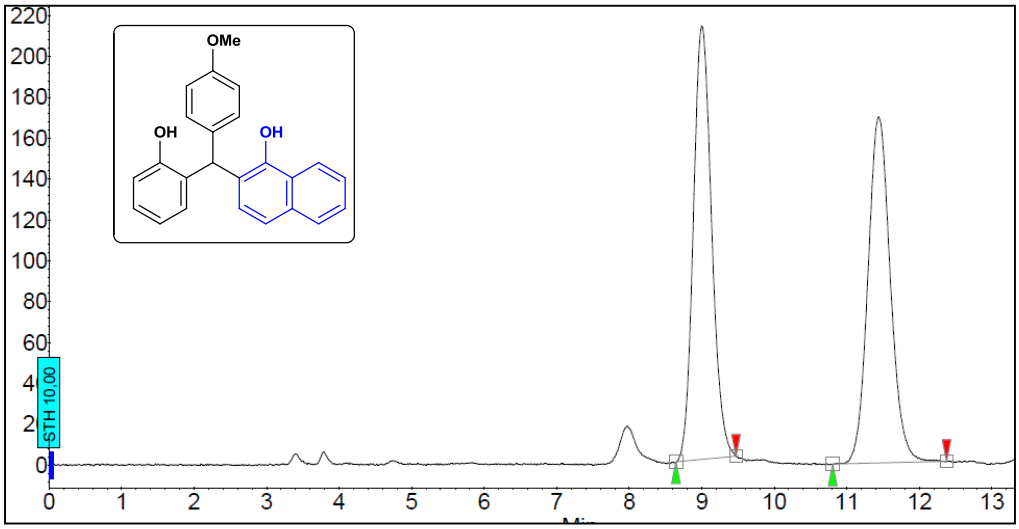


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	27,371	50,18	79,4	68,6	50,175
2	UNKNOWN	31,104	49,82	71,4	68,1	49,825
Total			100,00	150,8	136,7	100,000

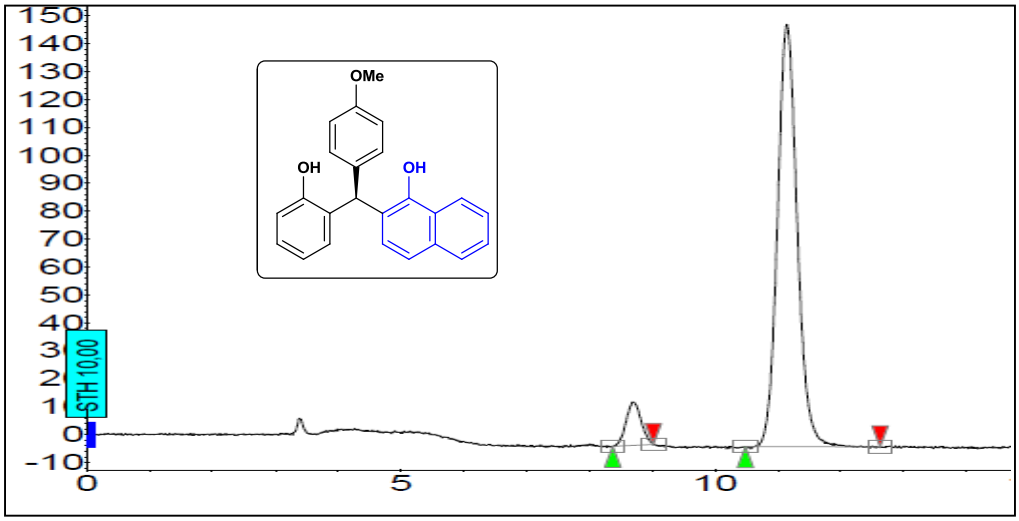


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
2	UNKNOWN	27,371	3,22	1,8	1,5	3,217
1	UNKNOWN	31,078	96,78	45,9	43,7	96,783
Total			100,00	47,7	45,2	100,000

(Table 4, entry 15), IA column, 80:20 (Hex:IPA), 1 mL/min, 230 nm

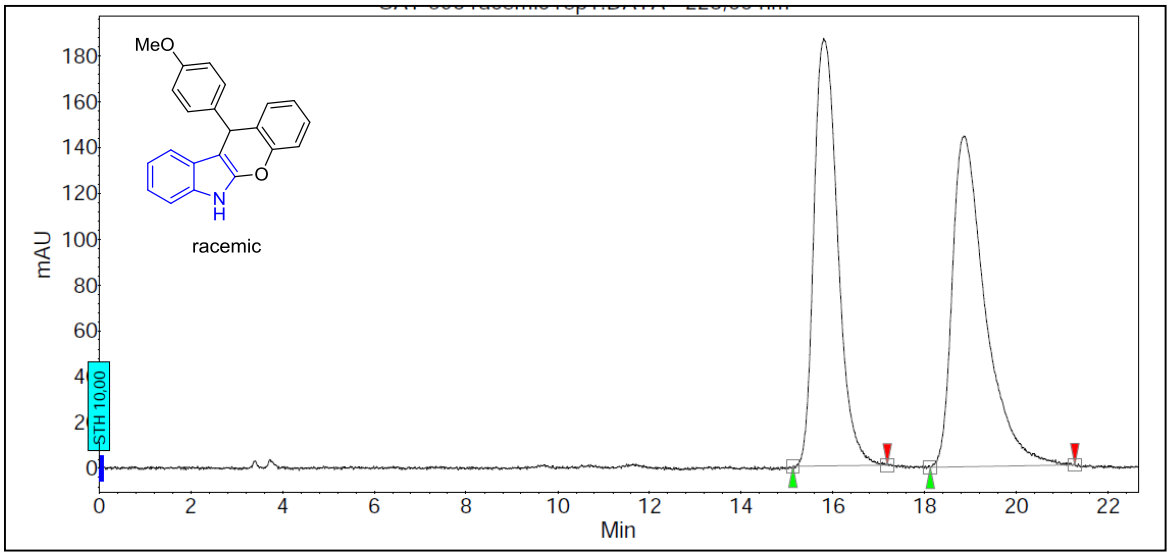


Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	9.000	50.53	212.1	61.9	50.525
2	UNKNOWN	11.440	49.47	169.3	60.6	49.475
Total			100.00	381.4	122.6	100.000



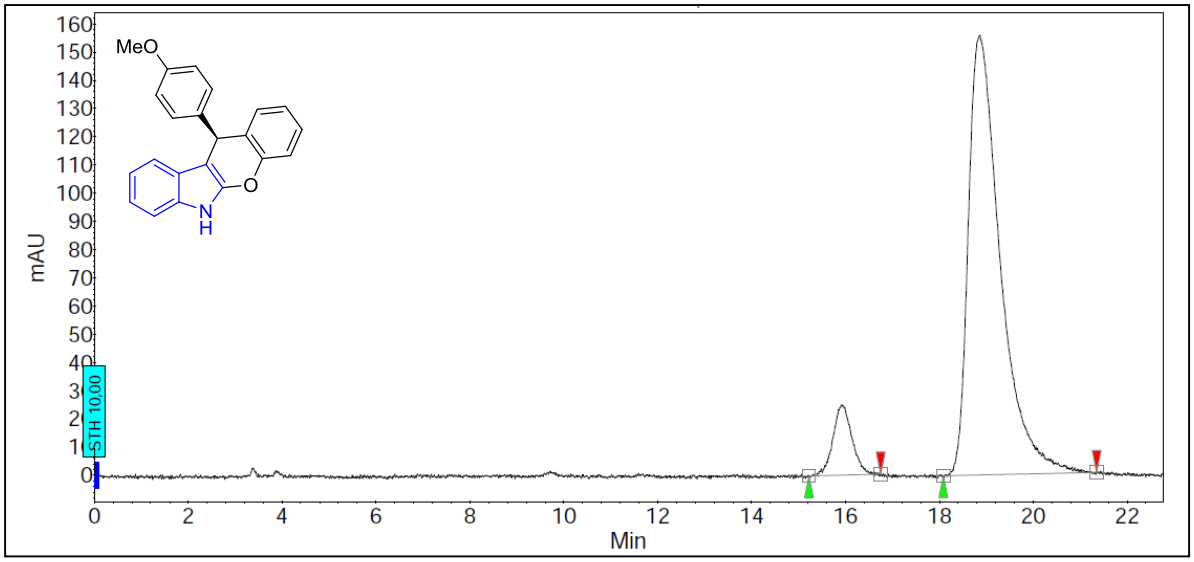
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	8.706	7.25	15.5	4.3	7.253
2	UNKNOWN	11.146	92.75	151.3	55.1	92.747
Total			100.00	166.8	59.4	100.000

(19a)IA column, 98:2 (Hex:IPA), 1 mL/min, 228 nm



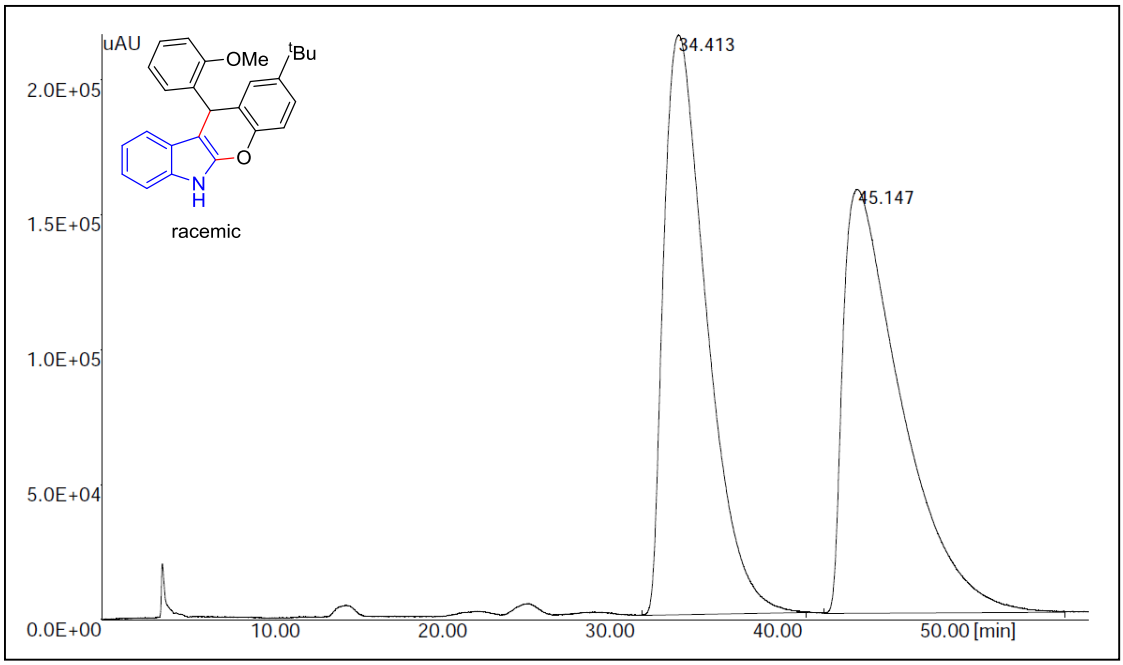
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	15,799	48,16	186,5	111,7	48,157
2	UNKNOWN	18,865	51,84	143,8	120,2	51,843
Total			100,00	330,2	231,9	100,000

(19a)IA column, 98:2 (Hex:IPA), 1 mL/min, 228 nm(er = 91:9)



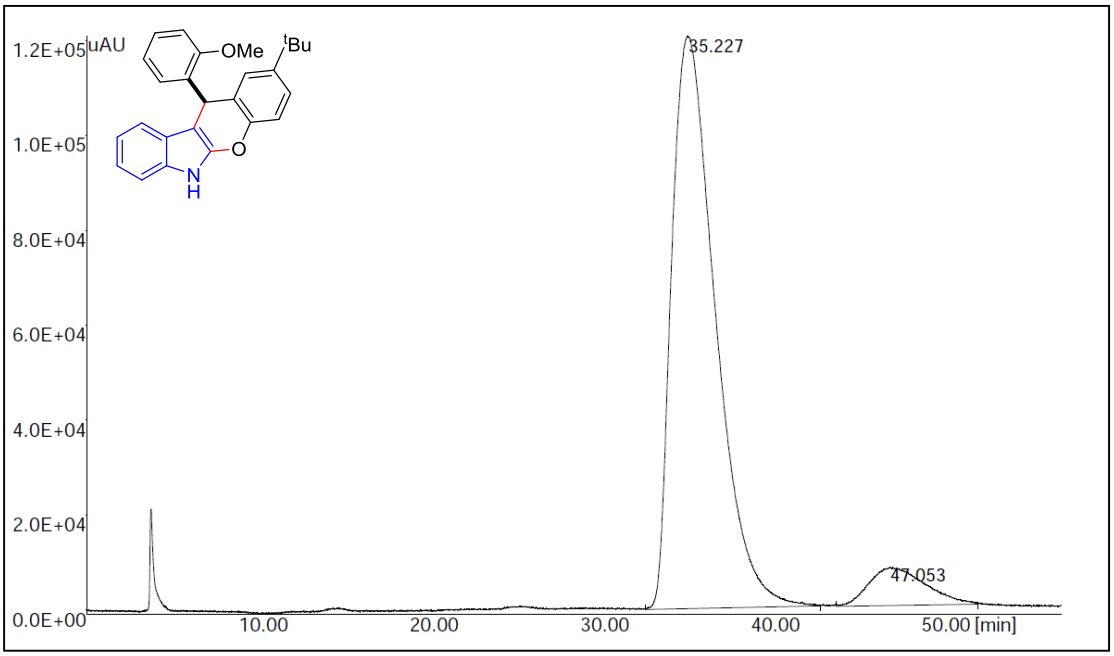
Index	Name	Time [Min]	Quantity [% Area]	Height [mAU]	Area [mAU.Min]	Area % [%]
1	UNKNOWN	15,932	8,63	24,8	11,6	8,634
2	UNKNOWN	18,852	91,37	155,8	122,7	91,366
Total			100,00	180,5	134,3	100,000

(19b)OD-H column, 98:2 (Hex:IPA), 1 mL/min, 221 nm



#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		34.413	215004	38170110.569	50.16
2		45.147	157164	37921829.365	49.84

(19b)OD-H column, 98:2 (Hex:IPA), 1 mL/min, 221 nm(er = 92:8)



#	Name	RT	Height [uAU]	Area [uAU.Sec]	%Area
1		35.227	120579	21286135.517	92.14
2		47.053	8068	1815970.427	7.86

X-ray Crystallographic Analysis

The data were collected on a Gemini diffractometer (Agilent Technologies) using Cu-K_α radiation (λ = 1.5418 Å), ω-scan rotation. Data reduction was performed with the CrysAlisPro (CrysAlisPro: Data collection and data reduction software package, Agilent Technologies) including the program SCALE3 ABSPACK (SCALE3 ABSPACK: Empirical absorption correction using spherical harmonics) for empirical absorption correction. The structures were solved by direct methods with SIR2004. The refinement of all non-hydrogen atoms was performed anisotropically, the hydrogen atoms isotropically with SHELXL-97. The structure figure was generated with ORTEP

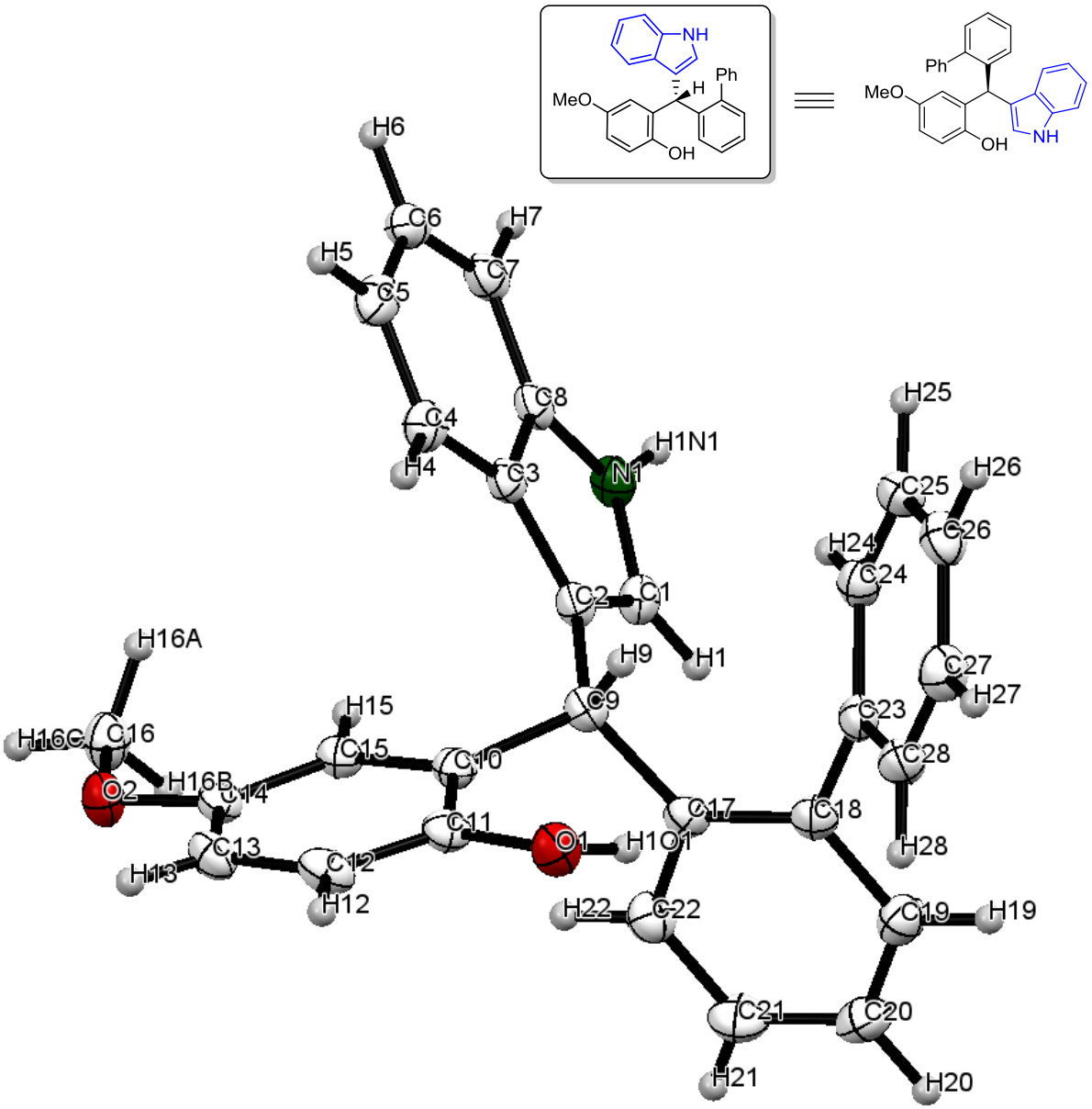


Table 1. Crystal data and structure refinement for x2068fin.**(Cu-radiation)**

Identification code	x2068fin	
Empirical formula	C28 H23 N O2	
Formula weight	405.47	
Temperature	130(2) K	
Wavelength	154.184 pm	
Crystal system	Orthorhombic	
Space group	P 21 21 21	
Unit cell dimensions	a = 943.230(10) pm	α= 90°.
	b = 1040.300(10) pm	β= 90°.
	c = 2119.50(2) pm	γ = 90°.
Volume	2.07974(4) nm^3	
Z	4	
Density (calculated)	1.295 Mg/m^3	
Absorption coefficient	0.637 mm^{-1}	
F(000)	856	
Crystal size	0.4 x 0.35 x 0.2 mm^3	
Theta range for data collection	4.172 to 67.716°.	
Index ranges	-11<=h<=11, -12<=k<=12, -25<=l<=25	
Reflections collected	31480	
Independent reflections	3765 [R(int) = 0.0227]	
Completeness to theta = 67.684°	100.0 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	1 and 0.60413	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3765 / 0 / 372	
Goodness-of-fit on F^2	1.054	
Final R indices [I>2sigma(I)]	R1 = 0.0266, wR2 = 0.0680	
R indices (all data)	R1 = 0.0269, wR2 = 0.0684	
Absolute structure parameter	-0.01(4)	
Extinction coefficient	n/a	
Largest diff. peak and hole	0.129 and -0.190 e.$Å^{-3}$	

Comments: Structure solution with SHELXS-2014 (direct method). Anisotropic refinement of all non-hydrogen atoms with SHELXL-2014. All H atoms were located on difference Fourier maps calculated at the final stage of the structure refinement.

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (pm^2x 10^{-1}) for x2068fin. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	5126(1)	-320(1)	4161(1)	28(1)
O(2)	7133(1)	1400(1)	1865(1)	26(1)
N(1)	273(2)	1827(1)	2203(1)	25(1)
C(1)	1071(2)	2023(2)	2739(1)	22(1)
C(2)	2050(2)	1069(2)	2803(1)	21(1)
C(3)	1842(2)	209(2)	2279(1)	20(1)
C(4)	2497(2)	-937(2)	2083(1)	24(1)
C(5)	1994(2)	-1550(2)	1552(1)	27(1)
C(6)	841(2)	-1058(2)	1210(1)	26(1)
C(7)	181(2)	72(2)	1386(1)	26(1)
C(8)	706(2)	703(2)	1920(1)	22(1)
C(9)	3045(2)	836(2)	3349(1)	20(1)
C(10)	4606(2)	732(2)	3162(1)	20(1)
C(11)	5538(2)	117(2)	3574(1)	23(1)
C(12)	6917(2)	-140(2)	3392(1)	25(1)
C(13)	7412(2)	264(2)	2812(1)	25(1)
C(14)	6523(2)	959(2)	2415(1)	22(1)
C(15)	5123(2)	1174(2)	2585(1)	21(1)
C(16)	6240(2)	2086(2)	1442(1)	29(1)
C(17)	2823(2)	1794(2)	3888(1)	22(1)
C(18)	2069(2)	1454(2)	4439(1)	22(1)
C(19)	1926(2)	2364(2)	4922(1)	27(1)
C(20)	2516(2)	3578(2)	4870(1)	31(1)
C(21)	3266(2)	3903(2)	4332(1)	31(1)
C(22)	3410(2)	3019(2)	3847(1)	27(1)
C(23)	1477(2)	140(2)	4544(1)	22(1)
C(24)	504(2)	-426(2)	4133(1)	24(1)
C(25)	-15(2)	-1650(2)	4251(1)	26(1)
C(26)	420(2)	-2331(2)	4779(1)	28(1)
C(27)	1380(2)	-1777(2)	5191(1)	29(1)

C(28)	1900(2)	-555(2)	5078(1)	27(1)

Table 3. Bond lengths [pm] and angles [°] for x2068fin.

O(1)-C(11)	138.1(2)
O(1)-H(1O1)	85(3)
O(2)-C(14)	137.7(2)
O(2)-C(16)	142.3(2)
N(1)-C(8)	137.6(2)
N(1)-C(1)	137.8(2)
N(1)-H(1N1)	89(2)
C(1)-C(2)	136.1(2)
C(1)-H(1)	97(2)
C(2)-C(3)	143.9(2)
C(2)-C(9)	150.9(2)
C(3)-C(4)	140.5(2)
C(3)-C(8)	141.1(2)
C(4)-C(5)	137.8(2)
C(4)-H(4)	99(2)
C(5)-C(6)	140.4(3)
C(5)-H(5)	96(2)
C(6)-C(7)	138.1(3)
C(6)-H(6)	99(2)
C(7)-C(8)	139.9(2)
C(7)-H(7)	100(2)
C(9)-C(10)	152.8(2)
C(9)-C(17)	153.1(2)
C(9)-H(9)	98(2)
C(10)-C(11)	139.4(2)
C(10)-C(15)	139.4(2)
C(11)-C(12)	138.3(3)
C(12)-C(13)	138.2(3)
C(12)-H(12)	98(2)
C(13)-C(14)	139.0(2)
C(13)-H(13)	93(2)

C(14)-C(15)	138.8(2)
C(15)-H(15)	96.2(19)
C(16)-H(16A)	102(2)
C(16)-H(16B)	97(2)
C(16)-H(16C)	98(2)
C(17)-C(22)	139.3(2)
C(17)-C(18)	141.3(2)
C(18)-C(19)	140.0(2)
C(18)-C(23)	149.4(2)
C(19)-C(20)	138.4(3)
C(19)-H(19)	97(2)
C(20)-C(21)	138.4(3)
C(20)-H(20)	98(3)
C(21)-C(22)	138.6(3)
C(21)-H(21)	95(2)
C(22)-H(22)	96(2)
C(23)-C(24)	139.6(2)
C(23)-C(28)	140.0(2)
C(24)-C(25)	138.6(3)
C(24)-H(24)	98(2)
C(25)-C(26)	138.7(3)
C(25)-H(25)	97(2)
C(26)-C(27)	138.4(3)
C(26)-H(26)	99(2)
C(27)-C(28)	138.4(3)
C(27)-H(27)	96(2)
C(28)-H(28)	95(3)

C(11)-O(1)-H(1O1)	110.5(18)
C(14)-O(2)-C(16)	116.91(13)
C(8)-N(1)-C(1)	108.84(14)
C(8)-N(1)-H(1N1)	123.7(14)
C(1)-N(1)-H(1N1)	125.6(14)
C(2)-C(1)-N(1)	110.13(15)
C(2)-C(1)-H(1)	128.2(12)
N(1)-C(1)-H(1)	121.7(12)

C(1)-C(2)-C(3)	106.53(14)
C(1)-C(2)-C(9)	127.93(15)
C(3)-C(2)-C(9)	125.13(14)
C(4)-C(3)-C(8)	118.91(15)
C(4)-C(3)-C(2)	134.09(15)
C(8)-C(3)-C(2)	107.00(14)
C(5)-C(4)-C(3)	118.88(16)
C(5)-C(4)-H(4)	119.7(11)
C(3)-C(4)-H(4)	121.4(11)
C(4)-C(5)-C(6)	121.34(17)
C(4)-C(5)-H(5)	120.5(13)
C(6)-C(5)-H(5)	118.2(13)
C(7)-C(6)-C(5)	121.24(16)
C(7)-C(6)-H(6)	119.4(12)
C(5)-C(6)-H(6)	119.3(12)
C(6)-C(7)-C(8)	117.33(16)
C(6)-C(7)-H(7)	122.0(13)
C(8)-C(7)-H(7)	120.7(13)
N(1)-C(8)-C(7)	130.25(16)
N(1)-C(8)-C(3)	107.46(14)
C(7)-C(8)-C(3)	122.28(15)
C(2)-C(9)-C(10)	114.37(13)
C(2)-C(9)-C(17)	112.49(13)
C(10)-C(9)-C(17)	111.79(13)
C(2)-C(9)-H(9)	104.9(11)
C(10)-C(9)-H(9)	106.1(11)
C(17)-C(9)-H(9)	106.4(10)
C(11)-C(10)-C(15)	118.69(15)
C(11)-C(10)-C(9)	118.56(14)
C(15)-C(10)-C(9)	122.68(14)
O(1)-C(11)-C(12)	116.85(15)
O(1)-C(11)-C(10)	122.56(15)
C(12)-C(11)-C(10)	120.51(15)
C(13)-C(12)-C(11)	120.45(16)
C(13)-C(12)-H(12)	119.8(14)
C(11)-C(12)-H(12)	119.8(14)

C(12)-C(13)-C(14)	119.57(16)
C(12)-C(13)-H(13)	122.7(13)
C(14)-C(13)-H(13)	117.7(13)
O(2)-C(14)-C(15)	124.36(15)
O(2)-C(14)-C(13)	115.64(15)
C(15)-C(14)-C(13)	120.00(15)
C(14)-C(15)-C(10)	120.54(15)
C(14)-C(15)-H(15)	121.4(11)
C(10)-C(15)-H(15)	118.1(11)
O(2)-C(16)-H(16A)	110.3(12)
O(2)-C(16)-H(16B)	110.3(13)
H(16A)-C(16)-H(16B)	107.6(18)
O(2)-C(16)-H(16C)	107.1(14)
H(16A)-C(16)-H(16C)	111.2(17)
H(16B)-C(16)-H(16C)	110.4(18)
C(22)-C(17)-C(18)	118.76(15)
C(22)-C(17)-C(9)	119.60(15)
C(18)-C(17)-C(9)	121.61(15)
C(19)-C(18)-C(17)	118.96(16)
C(19)-C(18)-C(23)	118.29(15)
C(17)-C(18)-C(23)	122.68(14)
C(20)-C(19)-C(18)	121.30(17)
C(20)-C(19)-H(19)	120.2(12)
C(18)-C(19)-H(19)	118.5(12)
C(21)-C(20)-C(19)	119.62(16)
C(21)-C(20)-H(20)	121.2(14)
C(19)-C(20)-H(20)	119.2(14)
C(20)-C(21)-C(22)	119.95(17)
C(20)-C(21)-H(21)	119.7(14)
C(22)-C(21)-H(21)	120.3(14)
C(21)-C(22)-C(17)	121.40(17)
C(21)-C(22)-H(22)	120.7(12)
C(17)-C(22)-H(22)	117.9(12)
C(24)-C(23)-C(28)	118.34(16)
C(24)-C(23)-C(18)	122.59(15)
C(28)-C(23)-C(18)	119.07(15)

C(25)-C(24)-C(23)	120.37(16)
C(25)-C(24)-H(24)	120.6(12)
C(23)-C(24)-H(24)	119.0(12)
C(24)-C(25)-C(26)	120.76(17)
C(24)-C(25)-H(25)	120.2(12)
C(26)-C(25)-H(25)	119.0(12)
C(27)-C(26)-C(25)	119.31(16)
C(27)-C(26)-H(26)	120.8(12)
C(25)-C(26)-H(26)	119.9(12)
C(28)-C(27)-C(26)	120.32(16)
C(28)-C(27)-H(27)	120.7(15)
C(26)-C(27)-H(27)	118.9(15)
C(27)-C(28)-C(23)	120.89(16)
C(27)-C(28)-H(28)	118.7(15)
C(23)-C(28)-H(28)	120.4(15)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($pm^2 x\ 10^{-1}$)for x2068fin. The anisotropic displacement factor exponent takes the form: $-2p^2[\ h^2a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	27(1)	32(1)	24(1)	7(1)	-1(1)	1(1)
O(2)	21(1)	33(1)	26(1)	5(1)	3(1)	3(1)
N(1)	19(1)	29(1)	26(1)	2(1)	-1(1)	6(1)
C(1)	22(1)	24(1)	21(1)	0(1)	4(1)	2(1)
C(2)	17(1)	24(1)	21(1)	2(1)	3(1)	-1(1)
C(3)	19(1)	22(1)	19(1)	3(1)	3(1)	-3(1)
C(4)	26(1)	24(1)	23(1)	3(1)	2(1)	1(1)
C(5)	34(1)	22(1)	25(1)	0(1)	5(1)	-2(1)
C(6)	30(1)	30(1)	20(1)	1(1)	2(1)	-10(1)
C(7)	21(1)	34(1)	22(1)	6(1)	1(1)	-6(1)
C(8)	18(1)	25(1)	22(1)	5(1)	4(1)	-3(1)
C(9)	20(1)	20(1)	21(1)	2(1)	0(1)	1(1)
C(10)	19(1)	19(1)	23(1)	-2(1)	-1(1)	-1(1)

C(11)	24(1)	20(1)	24(1)	2(1)	-2(1)	-3(1)
C(12)	22(1)	23(1)	32(1)	6(1)	-7(1)	0(1)
C(13)	17(1)	25(1)	35(1)	1(1)	1(1)	1(1)
C(14)	21(1)	21(1)	25(1)	0(1)	1(1)	-3(1)
C(15)	20(1)	20(1)	22(1)	1(1)	-2(1)	1(1)
C(16)	28(1)	34(1)	24(1)	5(1)	3(1)	4(1)
C(17)	20(1)	24(1)	21(1)	0(1)	-3(1)	2(1)
C(18)	19(1)	26(1)	22(1)	1(1)	-3(1)	1(1)
C(19)	29(1)	31(1)	22(1)	-2(1)	-1(1)	3(1)
C(20)	36(1)	29(1)	29(1)	-8(1)	-4(1)	3(1)
C(21)	34(1)	22(1)	38(1)	-1(1)	-5(1)	-2(1)
C(22)	28(1)	25(1)	28(1)	3(1)	1(1)	0(1)
C(23)	20(1)	26(1)	21(1)	-1(1)	3(1)	1(1)
C(24)	21(1)	29(1)	21(1)	1(1)	0(1)	2(1)
C(25)	21(1)	30(1)	27(1)	-1(1)	-1(1)	-2(1)
C(26)	27(1)	26(1)	31(1)	3(1)	6(1)	-2(1)
C(27)	29(1)	33(1)	24(1)	7(1)	0(1)	2(1)
C(28)	24(1)	33(1)	22(1)	0(1)	-2(1)	-2(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (pm^2x 10^{-1}) for x2068fin.

	x	y	z	U(eq)
H(1N1)	-530(30)	2230(20)	2107(10)	29(5)
H(1O1)	4400(30)	90(30)	4291(13)	54(8)
H(1)	900(20)	2749(19)	3018(10)	24(5)
H(4)	3290(20)	-1320(19)	2325(9)	25(5)
H(5)	2420(20)	-2340(20)	1410(10)	31(5)
H(6)	490(20)	-1530(20)	835(10)	26(5)
H(7)	-650(20)	420(20)	1151(10)	29(5)
H(9)	2777(19)	-6(19)	3513(8)	16(4)
H(12)	7550(30)	-600(20)	3679(11)	37(6)
H(13)	8330(20)	110(20)	2675(9)	28(5)

H(15)	4470(20)	1609(18)	2307(9)	20(5)
H(16A)	5410(20)	1530(20)	1308(9)	28(5)
H(16B)	5860(20)	2850(20)	1647(10)	29(5)
H(16C)	6820(30)	2330(20)	1080(11)	35(6)
H(19)	1390(20)	2131(19)	5295(10)	22(5)
H(20)	2420(30)	4180(20)	5222(12)	45(7)
H(21)	3670(20)	4730(20)	4296(10)	39(6)
H(22)	3930(20)	3235(19)	3470(10)	26(5)
H(24)	200(20)	51(19)	3758(9)	23(5)
H(25)	-710(20)	-2030(20)	3974(10)	25(5)
H(26)	30(20)	-3200(20)	4862(9)	26(5)
H(27)	1660(30)	-2240(20)	5558(11)	41(6)
H(28)	2580(30)	-200(20)	5362(11)	41(6)