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Electronic Supplementary Information

Catalyst- and Solvent-Free Aminolysis of the Asymmetric Derivatives of Evans' Chiral *N*-Acyloxazolidinones: Enantioselective Synthesis of Chiral Amides and Its Applications

Hui-Rong Lu,† Hui Geng,† Guan-Tian Ding,† and Pei-Qiang Huang*

Department of Chemistry, Fujian Provincial Key Laboratory of Chemical Biology, College of Chemistry and Chemical Engineering, Xiamen University, Xiamen, Fujian 361005, P. R. China.

E-mail: pqhuang@xmu.edu.cn

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† These authors contributed equally to this work.

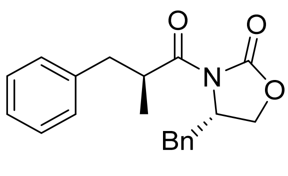
I. General Information

Unless otherwise noted, reactions were carried out in anhydrous and argon atmosphere. All glassware was dried in infrared rapid drying box before use. Unless otherwise specified, all reagents used in the experiment were purchased from the reagent supply platform (Adamas-beta®, Alfa Aesar, Sigma-Aldrich) and used directly without special treatment. All solvents were redistilled before use.

NMR spectra were measured by Bruker 400 MHz or Bruker 500 MHz spectrometer. Chemical shifts (δ) referenced to internal standard Me_4Si and solvent signals (Me_4Si, 0 ppm for ^1H NMR and $CDCl_3$, 77.0 ppm for ^{13}C NMR). HPLC analyses were carried out on an Agilent 1260 series system with Daicel Chiralpak® or Daicel Chiralcel® columns (4.6 × 250 mm, particle size 3 µm). Infrared spectra were measured with a Nicolet Avatar 360 FT-IR spectrometer using film KBr pellet techniques. Melting points were uncorrected. High resolution mass spectra (ESI) were measured by a Micromass QTOF2 Quadruple/Time-of-Flight Tandem mass spectrometer. Optical rotation data were obtained with an Anton Paar MCP 500 polarimeter at 589 nm. Silica gel (200–300 mesh) was used for flash column chromatography.

II. Synthesis of the Chiral *N*-Acyloxazolidinone Derivatives

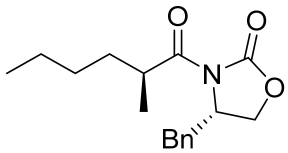
A series of chiral *N*-acyloxazolidones were prepared by asymmetric alkylation,[1] asymmetric Aldol reaction,[2,3] or asymmetric conjugate addition reaction[4] induced by Evans chiral auxiliaries, the experimental procedures were referred to the relevant literature.[1-4]



(*S*)-4-Benzyl-3-((*S*)-2-methyl-3-phenylpropanoyl)oxazolidin-2-one (2a).

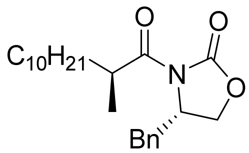
White solid (612 mg, 3.0 mmol, yield: 63%). Mp: 86-87.5 °C. ^1H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.24 (m, 5H), 7.26 – 7.14 (m, 5H), 4.57 – 4.46 (m, 1H), 4.17 – 4.09 (m, 1H), 4.09 – 4.03 (m, 1H), 3.95 (t, *J* = 8.4 Hz, 1H), 3.24 (dd, *J* = 13.4, 3.4 Hz, 1H), 3.03 (dd, *J* = 13.4, 7.7

Hz, 1H), 2.79 – 2.64 (m, 2H), 1.25 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.5, 153.0, 139.1, 135.1, 129.3, 129.3, 128.9, 128.3, 127.2, 126.4, 65.8, 55.0, 39.9, 39.5, 37.7, 16.7.



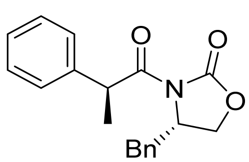
(*S*)-4-Benzyl-3-((*S*)-2-methylhexanoyl)oxazolidin-2-one (2b)

Colorless oil (816 mg, 3.6 mmol, yield: 78%). $[\alpha]_D^{25}$ +75.3 (*c* 1.0, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 – 7.29 (m, 2H), 7.31 – 7.25 (m, 1H), 7.28 – 7.19 (m, 2H), 4.68 (ddt, *J* = 9.7, 7.5, 3.3 Hz, 1H), 4.24 – 4.12 (m, 2H), 3.71 (h, *J* = 6.9 Hz, 1H), 3.27 (dd, *J* = 13.4, 3.3 Hz, 1H), 2.77 (dd, *J* = 13.4, 9.7 Hz, 1H), 1.80 – 1.70 (m, 1H), 1.48 – 1.37 (m, 1H), 1.38 – 1.26 (m, 4H), 1.22 (d, *J* = 6.8 Hz, 3H), 0.89 (t, *J* = 6.9 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 177.3, 153.0, 135.3, 129.4, 128.9, 127.3, 66.0, 55.3, 37.9, 37.6, 33.1, 29.4, 22.7, 17.3, 14.0.



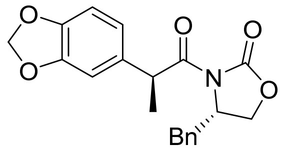
(*S*)-4-Benzyl-3-((*S*)-2-methyldodecanoyl)oxazolidin-2-one (2c)

Colorless oil (783 mg, 3.0 mmol, yield: 70%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 – 7.30 (m, 2H), 7.30 – 7.27 (m, 1H), 7.22 (dd, *J* = 6.9, 1.7 Hz, 2H), 4.72 – 4.63 (m, 1H), 4.23 – 4.13 (m, 2H), 3.70 (h, *J* = 6.9 Hz, 1H), 3.27 (dd, *J* = 13.4, 3.3 Hz, 1H), 2.77 (dd, *J* = 13.4, 9.6 Hz, 1H), 1.78 – 1.68 (m, 1H), 1.65 – 1.55 (m, 1H), 1.46 – 1.34 (m, 1H), 1.25 (s, 15H), 1.22 (d, *J* = 6.9 Hz, 3H), 0.88 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 177.4, 153.0, 135.3, 129.4, 128.9, 127.3, 66.0, 55.4, 37.9, 37.7, 33.4, 31.9, 29.7, 29.6, 29.5, 29.3, 27.3, 22.7, 17.4, 14.1.



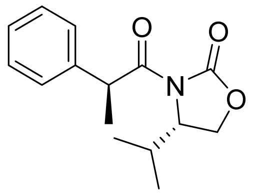
(*S*)-4-Benzyl-3-((*S*)-2-phenylpropanoyl)oxazolidin-2-one (2d)

White solid (807 mg, 3.4 mmol, yield: 77%). Mp: 76.5-78 °C. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 – 7.33 (m, 2H), 7.33 – 7.27 (m, 4H), 7.27 – 7.18 (m, 4H), 5.12 (q, *J* = 7.0 Hz, 1H), 4.63 – 4.49 (m, 1H), 4.08 (dd, *J* = 9.0, 2.4 Hz, 1H), 4.01 (t, *J* = 8.3 Hz, 1H), 3.33 (dd, *J* = 13.3, 3.4 Hz, 1H), 2.79 (dd, *J* = 13.3, 9.7 Hz, 1H), 1.54 (d, *J* = 7.0 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.5, 152.8, 140.2, 135.3, 129.4, 128.9, 128.6, 128.1, 127.3, 127.2, 65.8, 55.7, 43.1, 37.9, 19.4.



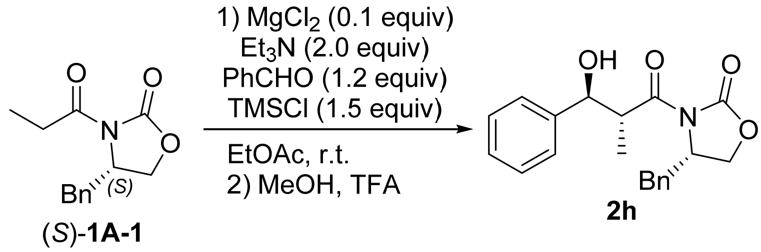
(*S*)-3-((*S*)-2-(Benzo[d][1,3]dioxol-5-yl)propanoyl)-4-benzyloxazolidin-2-one (2f)

Yellow oil, yield: 65%. $[\alpha]_D^{25}$ +105.5 (*c* 1.0, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 (t, *J* = 7.4 Hz, 2H), 7.30 – 7.24 (m, 1H), 7.24 – 7.18 (m, 2H), 6.88 (d, *J* = 1.8 Hz, 1H), 6.82 (dd, *J* = 8.0, 1.8 Hz, 1H), 6.73 (d, *J* = 8.0 Hz, 1H), 5.92 (s, 2H), 5.04 (q, *J* = 7.1 Hz, 1H), 4.62 – 4.55 (m, 1H), 4.14 – 4.09 (m, 1H), 4.09 – 4.03 (m, 1H), 3.33 (dd, *J* = 13.4, 3.2 Hz, 1H), 2.79 (dd, *J* = 13.4, 9.7 Hz, 1H), 1.50 (d, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 174.6, 152.9, 147.7, 146.7, 135.3, 134.0, 129.4, 128.9, 127.3, 121.4, 108.5, 108.3, 101.0, 65.9, 55.8, 42.6, 37.9, 19.5. HRMS calcd for $[C_{20}H_{19}NO_5Na]^+$ ($M+Na^+$): 376.1155, found: 376.1162.



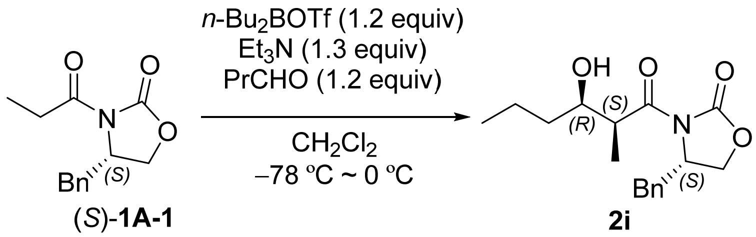
(*S*)-4-Isopropyl-3-((*S*)-2-phenylpropanoyl)oxazolidin-2-one (2g)

Colorless oil. (585 mg, 4.4 mmol, yield: 53%). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.39 – 7.31 (m, 2H), 7.31 – 7.24 (m, 2H), 7.24 – 7.18 (m, 1H), 5.14 (q, *J* = 7.0 Hz, 1H), 4.37 – 4.30 (m, 1H), 4.11 (dd, *J* = 9.2, 2.5 Hz, 1H), 4.05 (t, *J* = 8.7 Hz, 1H), 2.48 – 2.34 (m, 1H), 1.50 (d, *J* = 7.0, 3H), 0.92 (d, *J* = 4.6 Hz, 3H), 0.90 (d, *J* = 4.6 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 174.4, 153.4, 140.2, 128.4, 128.0, 127.0, 62.9, 58.8, 42.9, 28.4, 19.5, 17.8, 14.5.



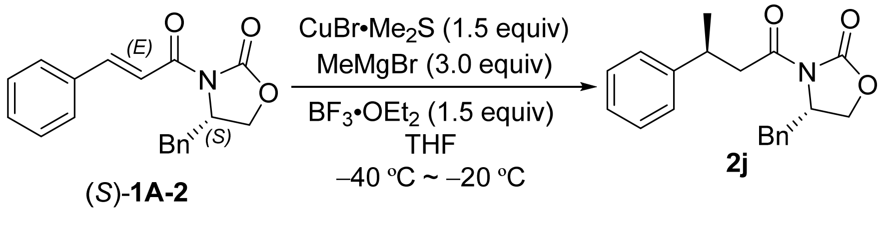
(*S*)-4-Benzyl-3-((2*R*,3*S*)-3-hydroxy-2-methyl-3-phenylpropanoyl)oxazolidin-2-one (2h)

White solid (523 mg, 2.0 mmol, yield: 77%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.47 – 7.37 (m, 2H), 7.37 – 7.31 (m, 2H), 7.31 – 7.21 (m, 4H), 7.19 – 7.09 (m, 2H), 4.80 (d, *J* = 8.1 Hz, 1H), 4.72 – 4.58 (m, 1H), 4.40 – 4.26 (m, 1H), 4.20 – 4.03 (m, 2H), 3.17 (dd, *J* = 13.6, 3.4 Hz, 1H), 3.13 (brs, 1H), 2.64 (dd, *J* = 13.6, 9.3 Hz, 1H), 1.07 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.5, 153.4, 142.0, 135.1, 129.3, 128.8, 128.4, 127.8, 127.1, 126.5, 77.2, 65.8, 55.2, 44.2, 37.4, 14.7.



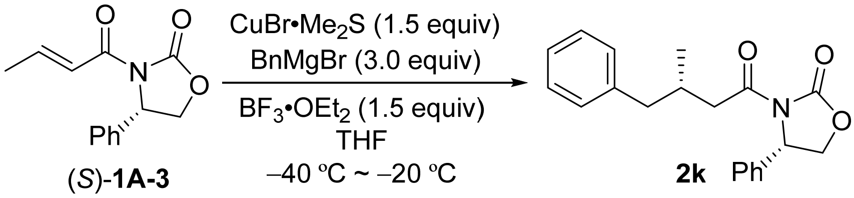
(*S*)-4-Benzyl-3-((2*S*,3*R*)-3-hydroxy-2-methylhexanoyl)oxazolidin-2-one (2i)

White solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.38 – 7.30 (m, 2H), 7.30 – 7.25 (m, 1H), 7.24 – 7.16 (m, 2H), 4.79 – 4.61 (m, 1H), 4.23 (dd, *J* = 9.1, 7.7 Hz, 1H), 4.18 (dd, *J* = 9.1, 2.9 Hz, 1H), 3.97 (ddd, *J* = 8.6, 4.3, 2.9 Hz, 1H), 3.77 (qd, *J* = 7.1, 2.9 Hz, 1H), 3.24 (dd, *J* = 13.5, 3.4 Hz, 1H), 3.02 (brs, 1H), 2.79 (dd, *J* = 13.5, 9.4 Hz, 1H), 1.61 – 1.45 (m, 2H), 1.44 – 1.31 (m, 2H), 1.25 (d, *J* = 7.1 Hz, 3H), 0.94 (t, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 177.4, 153.0, 135.0, 129.3, 128.9, 127.3, 71.2, 66.1, 55.0, 42.1, 37.7, 35.9, 19.1, 13.9, 10.3.



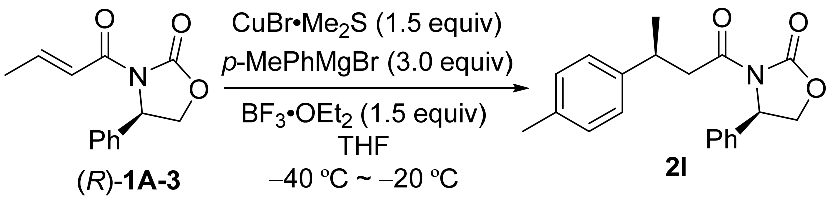
(*S*)-4-Benzyl-3-((*S*)-3-phenylbutanoyl)oxazolidin-2-one (2j)

White solid (386 mg, 2.0 mmol, yield: 60%). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.24 (m, 7H), 7.24 – 7.19 (m, 1H), 7.16 – 7.04 (m, 2H), 4.74 – 4.55 (m, 1H), 4.20 – 4.06 (m, 2H), 3.53 – 3.36 (m, 2H), 3.19 – 2.99 (m, 2H), 2.59 (dd, *J* = 13.5, 9.4 Hz, 1H), 1.36 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.9, 153.3, 145.6, 135.1, 129.3, 128.9, 128.4, 127.2, 127.0, 126.4, 66.0, 54.9, 43.2, 37.6, 36.0, 22.0.



(*S*)-3-((*S*)-3-Methyl-4-phenylbutanoyl)-4-phenyloxazolidin-2-one (2k)

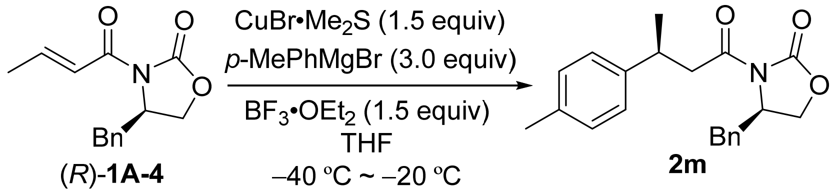
White solid (339 mg, 1.1 mmol, yield: 97%). Mp: 95-97 °C. $[\alpha]_D^{22}$ +48.5 (*c* 1.0, $CHCl_3$). ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.37 – 7.27 (m, 4H), 7.26 – 7.19 (m, 3H), 7.14 (dd, *J* = 8.4, 6.7 Hz, 1H), 7.12 – 7.05 (m, 2H), 5.31 (dd, *J* = 8.8, 3.8 Hz, 1H), 4.56 – 4.47 (m, 1H), 4.15 – 4.12 (m, 1H), 2.94 (ddd, *J* = 16.4, 5.6, 2.9 Hz, 1H), 2.77 (dd, *J* = 16.4, 8.2 Hz, 1H), 2.61 (ddd, *J* = 13.4 6.2, 2.0 Hz, 1H), 2.43 (dd, *J* = 13.4, 8.0 Hz, 1H), 2.35 – 2.28 (m, 1H), 0.84 (d, *J* = 6.7 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.8, 153.5, 140.0, 129.0, 128.9, 128.4, 128.2, 128.0, 126.7, 125.7, 69.6, 57.3, 42.7, 41.8, 31.3, 19.1. HRMS calcd for $[C_{20}H_{21}NO_3Na]^+$ ($M+Na^+$): 346.1414, found: 346.1418.



(*R*)-4-Phenyl-3-((*S*)-3-(*p*-tolyl)butanoyl)oxazolidin-2-one (2l)

White solid (900 mg, 3.7 mmol, yield: 76%). Mp: 71-72 °C. $[\alpha]_D^{22}$ +17.9 (*c* 1.0, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.31 – 7.19 (m, 3H), 7.15 – 6.98 (m, 6H), 5.34 (dd, *J* = 8.8, 4.0 Hz, 1H), 4.54 (t, *J* = 8.8 Hz, 1H), 4.10 (dd, *J* = 8.8, 4.0 Hz, 1H), 3.45 (dd, *J* = 15.7, 6.7 Hz, 1H), 3.32 – 3.25 (m, 1H), 3.00 (dd, *J* = 15.7, 7.9 Hz, 1H), 2.30 (s, 3H), 1.22 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.3, 153.5, 142.4, 138.7, 135.5, 129.0, 128.8, 128.2, 126.6, 125.5, 69.6, 57.3, 43.0, 35.5, 21.7, 20.8. HRMS calcd for $[C_{20}H_{21}NO_3Na]^+$ ($M+Na^+$):

346.1414, found: 346.1418.

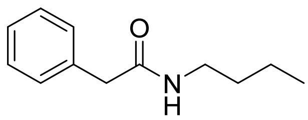


(*R*)-4-Bnezyl-3-((*S*)-3-(*p*-tolyl)butanoyl)oxazolidin-2-one (2m)

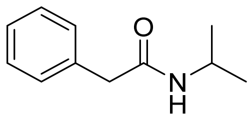
White solid (7.76 g, 23.0 mmol, yield: 81%). Mp: 85-86 °C. $[\alpha]_D^{20}$ +26.4 (*c* 1.0, $CHCl_3$). ^1H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.06 (m, 9H), 4.62 (ddt, *J* = 11.0, 7.5, 3.4 Hz, 1H), 4.18 – 4.05 (m, 2H), 3.49 – 3.33 (m, 2H), 3.13 – 3.00 (m, 2H), 2.60 (dd, *J* = 13.5, 9.3 Hz, 1H), 2.31 (s, 3H), 1.33 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.9, 153.3, 142.6, 135.8, 135.1, 129.3, 129.1, 128.8, 127.2, 126.8, 65.9, 54.9, 43.2, 37.5, 35.5, 22.1, 20.9. HRMS calcd for $[C_{21}H_{23}NO_3Na]^+$ ($M+Na^+$): 360.1570, found: 360.1572.

III. Aminolysis of the Chiral *N*-Acyloxazolidinone Derivatives

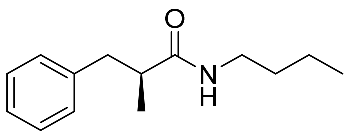
General Procedure 1 (GP-1): A mixture of *N*-Acyloxazolidinone (0.5 mmol, 1.0 equiv) and amine (1.5 mmol, 3.0 equiv) was stirred at room temperature under nitrogen atmosphere for 24 h. The resulting mixture was concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel eluting with petroleum ether/ethyl acetate (4/1, *v/v*) mixture.



***N*-Butyl-2-phenylacetamide (3A).** Following **GP-1**, **3A** was synthesized from **1a** and butan-1-amine. White solid. Yield: 98% (96 mg, 0.50 mmol). Spectral data match those previously reported[5a].



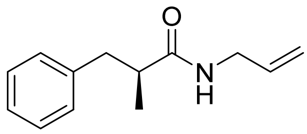
***N*-Isopropyl-2-phenylacetamide (3ac).** Following **GP-1**, **3ac** was synthesized from **1a** and propan-2-amine. White solid. Yield: 87% (62 mg, 0.4 mmol). Spectral data match those previously reported[5b].



(*S*)-*N*-Butyl-2-methyl-3-phenylpropanamide (3a).

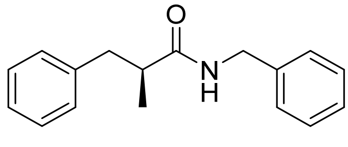
Following **GP-1**, **3a** was synthesized from **2a** and buthan-1-amine. Colorless oil. Yield: 80% (46 mg, 0.26 mmol). $[\alpha]_D^{25}$ +54.9 (*c* 0.99, $CHCl_3$); IR (film): 3294, 3085, 3064, 3028, 2960, 2930, 2872, 1645, 1557, 1496, 1455, 1260, 1080, 800, 744, 699 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 7.33 – 7.22 (m, 2H), 7.22 – 7.10 (m, 3H), 5.30 (s, 1H), 3.17 (dq, *J* = 13.0, 6.6 Hz, 1H), 3.10 (dq, *J* = 13.0, 6.6 Hz, 1H), 2.95 (dd, *J* = 13.4, 8.3 Hz, 1H), 2.67 (dd, *J* = 13.4, 6.2 Hz, 1H), 2.44 – 2.35 (m, 1H), 1.37 – 1.27 (m, 2H), 1.24 – 1.10 (m, 5H), 0.85 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.4, 139.9, 128.9, 128.3, 126.2, 43.9, 40.5, 39.0, 31.5, 19.9, 17.7, 13.6. HRMS calcd for $[C_{14}H_{21}NONa]^+$ $(M+Na^+)$: 242.1515, found: 242.1518.

The *ee* was determined via HPLC on a Chiralcel OD-3 column (15.0% 2-PrOH in hexanes, 1.0 mL/min); t_R: 5.3 min (major), 6.2 min (minor). The enantiomeric excess was determined to be 99%.



(*S*)-*N*-Allyl-2-methyl-3-phenylpropanamide (3b)

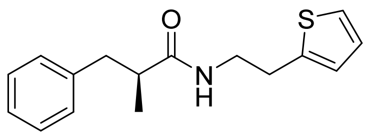
Following **GP-1**, **3b** was synthesized from **2a** and prop-2-en-1-amine. Colorless oil. Yield: 82% (72.4 mg, 0.43 mmol). $[\alpha]_D^{25}$ +48.8 (*c* 1.0, $CHCl_3$) {lit.[6a] $[\alpha]_D^{24}$ +59.0 (*c* 0.93, $CHCl_3$)}. IR (film): 3288, 3083, 3027, 2923, 2854, 1732, 1644, 1549, 1455, 1372, 1259, 1081, 1031, 918, 804, 745, 700, 587, 546 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 7.34 – 7.25 (m, 2H), 7.24 – 7.15 (m, 3H), 5.81 – 5.63 (m, 1H), 5.57 (s, 1H), 5.08 – 4.94 (m, 2H), 3.92 – 3.66 (m, 2H), 2.99 (dd, *J* = 13.4, 8.3 Hz, 1H), 2.69 (dd, *J* = 13.4, 6.6 Hz, 1H), 2.58 – 2.38 (m, 1H), 1.20 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 175.4, 139.8, 134.1, 128.9, 128.3, 126.2, 116.0, 43.8, 41.6, 40.4, 17.7. Spectral data match those previously reported[6b]. HRMS calcd for $[C_{13}H_{17}NONa]^+$ (M+Na^+): 226.1202, found: 226.1204. The *ee* was determined via HPLC on a Chiralcel OD-3 column (15.0% 2-PrOH in hexanes, 1.0 mL/min); t_R: 5.2 min (major), 5.8 min (minor). The enantiomeric excess was determined to be 98%.



(*S*)-*N*-Benzyl-2-methyl-3-phenylpropanamide (3c)

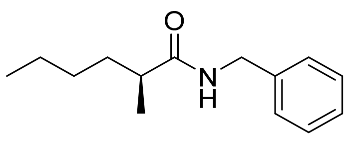
Following **GP-1**, **3c** was synthesized from **2a** and phenylmethanamine. White solid. Yield: 79% (57 mg, 0.28 mmol). $[\alpha]_D^{25}$ +41.5 (*c* 1.0, $CHCl_3$) {lit.[6a] $[\alpha]_D^{24}$ +52.3 (*c* 1.24, $CHCl_3$); lit.[7] $[\alpha]_D^{20}$ +23.2 (*c* 0.93, $CHCl_3$)}. IR (film): 3289, 3066, 3027, 3005, 2971, 2925, 2855, 1645, 1604, 1586, 1557, 1495, 1455, 1424, 1378, 1360, 1291, 1263, 1232, 1193, 1070, 1055, 1026, 1003, 780, 746, 734, 703, 695, 603 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 7.30 –

7.18 (m, 6H), 7.18 – 7.12 (m, 2H), 7.01 (dd, *J* = 7.3, 2.0 Hz, 2H), 5.63 (s, 1H), 4.37 (dd, *J* = 14.8, 5.7 Hz, 1H), 4.25 (dd, *J* = 14.8, 5.0 Hz, 1H), 2.97 (dd, *J* = 13.4, 8.6 Hz, 1H), 2.69 (dd, *J* = 13.4, 6.1 Hz, 1H), 2.50 – 2.43 (m, 1H), 1.21 (d, *J* = 6.7 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.3, 139.8, 138.1, 128.9, 128.5, 128.4, 127.6, 127.2, 126.2, 43.9, 43.3, 40.5, 17.8. Spectral data match those previously reported[6b]. HRMS calcd for $[C_{17}H_{19}NOH]^+$ ($M+H^+$): 254.1539, found: 254.1541. The *ee* was determined via HPLC on a Chiralcel OD-3 column (15.0% 2-PrOH in hexanes, 1.0 mL/min); t_R: 9.7 min (major), 10.7 min (minor). The enantiomeric excess was determined to be 99%.



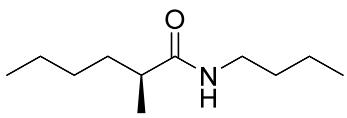
(*S*)-2-Methyl-3-phenyl-*N*-(2-(thiophen-2-yl)ethyl)propanamide (3d)

Following **GP-1**, **3d** was synthesized from **2a** and 2-(thiophen-2-yl)ethan-1-amine. Yellow solid. Yield: 83% (164 mg, 0.70 mmol). Mp: 67.8-68.9 °C. $[\alpha]_D^{20}$ +36.8 (*c* 1.0, $CHCl_3$); IR (film): 3294, 3084, 3027, 2965, 2929, 2872, 1647, 1549, 1495, 1453, 1368, 1247, 1079, 1031, 851, 806, 745, 699 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.30 – 7.23 (m, 2H), 7.20 (d, *J* = 7.3 Hz, 1H), 7.17 – 7.13 (m, 2H), 7.13 – 7.09 (m, 1H), 6.89 (dd, *J* = 5.1, 3.4 Hz, 1H), 6.63 (d, *J* = 3.4 Hz, 1H), 5.45 (s, 1H), 3.45 (dq, *J* = 13.2, 6.5 Hz, 1H), 3.37 (dq, *J* = 13.2, 6.4 Hz, 1H), 3.01 – 2.87 (m, 2H), 2.87 – 2.74 (m, 1H), 2.65 (dd, *J* = 13.4, 6.5 Hz, 1H), 2.38 (dt, *J* = 8.2, 6.7 Hz, 1H), 1.15 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.5, 141.2, 139.8, 128.9, 128.3, 126.9, 126.2, 125.2, 123.8, 43.8, 40.5, 40.4, 29.8, 17.7. HRMS calcd for $[C_{16}H_{19}NOSH]^+$ ($M+H^+$): 274.1260, found: 274.1259. The *ee* was determined via HPLC on a Chiralcel OJ-3 column (15.0% 2-PrOH in hexanes, 1.0 mL/min); t_R: 10.7 min (minor), 12.3 min (major). The enantiomeric excess was determined to be 99%.



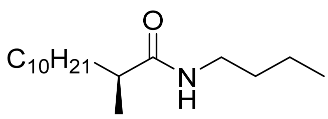
(*S*)-*N*-Benzyl-2-methylhexanamide (3e)

Following **GP-1**, **3e** was synthesized from **2b** and phenylmethanamine. White solid. Yield: 81% (65.8 mg, 0.37 mmol). Mp: 71.5-72.5 °C. $[\alpha]_D^{25}$ +7.6 (*c* 1.0, $CHCl_3$); IR (film): 3285, 3034, 2956, 2925, 2872, 2855, 1636, 1537, 1497, 1454, 1385, 1368, 1261, 1240, 1208, 1120, 1099, 1027, 799, 745, 695, 579, 541 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.29 (m, 2H), 7.26 (m, 3H), 5.92 (s, 1H), 4.42 (dd, *J* = 5.7, 2.1 Hz, 2H), 2.24 – 2.15 (m, 1H), 1.76 – 1.57 (m, 1H), 1.43 – 1.35 (m, 1H), 1.31 – 1.24 (m, 4H), 1.15 (d, *J* = 6.8 Hz, 3H), 0.87 (t, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.5, 138.6, 128.6, 127.7, 127.3, 43.3, 41.5, 34.0, 29.7, 22.6, 17.9, 13.9. HRMS calcd for $[C_{13}H_{19}NONa]^+$ $(M+Na^+)$: 220.1696, found: 220.1698. The *ee* was determined via HPLC on a Chiralpak IC-3 column (10.0% 2-PrOH in hexanes, 1.0 mL/min); t_R: 13.4 min (major), 14.4 min (minor). The enantiomeric excess was determined to be 99%.



(*S*)-*N*-Butyl-2-methylhexanamide (3f)

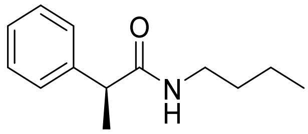
Following **GP-1**, **3f** was synthesized from **2b** and butan-1-amine. Colorless oil. Yield: 72% (60 mg, 0.45 mmol). $[\alpha]_D^{25}$ +14.0 (*c* 1.0, $CHCl_3$); IR (film): 3295, 3086, 2960, 2931, 2874, 1645, 1553, 1463, 1370, 1299, 1259, 1209, 1149, 1100, 1020, 802, 702 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 5.74 (s, 1H), 3.23 (tt, *J* = 7.1, 5.7 Hz, 2H), 2.19 – 2.08 (m, 1H), 1.70 – 1.55 (m, 1H), 1.52 – 1.42 (m, 2H), 1.40 – 1.29 (m, 3H), 1.29 – 1.18 (m, 4H), 1.10 (d, *J* = 6.9 Hz, 3H), 0.91 (t, *J* = 7.3 Hz, 3H), 0.86 (t, *J* = 7.0 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.5, 41.5, 39.0, 34.0, 31.7, 29.6, 22.6, 20.0, 17.9, 13.9, 13.7. HRMS calcd for $[C_{17}H_{35}NONa]^+$ $(M+Na^+)$: 208.1672, found: 208.1676. The *ee* was determined via HPLC on a Chiralcel OD-3 column (1.0% 2-PrOH in hexanes, 1.0 mL/min); t_R: 13.9 min (major), 15.9 min (minor). The enantiomeric excess was determined to be 99%.



(*S*)-*N*-Butyl-2-methyldodecanamide (3g)

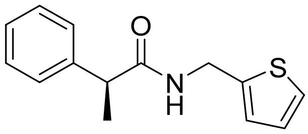
Following **GP-1**, **3g** was synthesized from **2c** and butan-1-amine. White solid. Yield: 67%

(53 mg, 0.3 mmol). Mp: 63.6-65.0 °C. $[\alpha]_D^{25}$ +9.94 (*c* 1.0, $CHCl_3$); IR (film): 3294, 2959, 2923, 2851, 1689, 1640, 1552, 1466, 1375, 1252, 1238, 1157, 1107, 1032, 802, 721 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 5.55 (s, 1H), 3.34 – 3.16 (m, 2H), 2.16 – 2.12 (m, 1H), 1.62 (dd, *J* = 14.4, 7.3 Hz, 1H), 1.52 – 1.44 (m, 2H), 1.40 – 1.31 (m, 3H), 1.25 (s, 16H), 1.12 (d, *J* = 6.8 Hz, 3H), 0.93 (t, *J* = 7.3 Hz, 3H), 0.88 (t, *J* = 6.7 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.5, 41.7, 39.0, 34.4, 31.9, 31.8, 29.6, 29.6, 29.5, 29.3, 27.5, 22.6, 20.0, 17.9, 14.1, 13.7. HRMS calcd for $[C_{17}H_{35}NOH]^+$ ($M+H^+$): 270.2791, found: 270.2793. The *ee* was determined via HPLC on a Chiralcel OD-3 column (1.0% 2-PrOH in hexanes, 1.0 mL/min); t_R: 11.0 min (minor), 12.3 min (major). The enantiomeric excess was determined to be 99%.



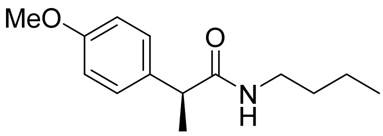
(*S*)-*N*-Butyl-2-phenylpropanamide (3h)

Following **GP-1**, **3h** was synthesized from **2d** and butan-1-amine. Yield: 93% (92 mg, 0.45 mmol); also synthesized from **2g** and butan-1-amine Yield: 89% (71 mg, 0.38 mmol). Colorless oil. $[\alpha]_D^{25}$ +20.6 (*c* 1.0, $CHCl_3$); IR (film): 3293, 3085, 3064, 3029, 2960, 2931, 2872, 1645, 1553, 1455, 1370, 1234, 698 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 7.36 – 7.31 (m, 2H), 7.31 – 7.28 (m, 2H), 7.28 – 7.23 (m, 1H), 5.47 (s, 1H), 3.54 (q, *J* = 7.2 Hz, 1H), 3.22 – 3.13 (m, 2H), 1.51 (d, *J* = 7.2 Hz, 3H), 1.44 – 1.33 (m, 2H), 1.27 – 1.20 (m, 2H), 0.86 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 174.2, 141.6, 128.8, 127.6, 127.1, 47.0, 39.4, 31.6, 20.0, 18.6, 13.7. HRMS calcd for $[C_{13}H_{19}NONa]^+$ ($M+Na^+$): 228.1359, found: 228.1362. The *ee* was determined via HPLC on a Chiralpak IC-3 column (15.0% 2-PrOH in hexanes, 1.0 mL/min); t_R: 10.3 min (major), 11.2 min (minor). The enantiomeric excess of was determined to be 98% and 95%, respectively.



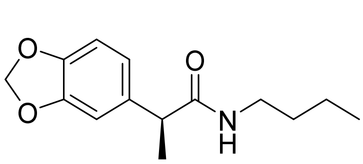
(*S*)-2-Phenyl-*N*-(thiophen-2-ylmethyl)propanamide (3i)

Following **GP-1**, **3i** was synthesized from **2d** and thiophen-2-ylmethanamine. White solid. yield: 98% (216 mg, 0.89 mmol). Mp: 56-57 °C. $[\alpha]_D^{25}$ +10.1 (*c* 1.0, $CHCl_3$); IR (film): 3290, 3070, 2978, 2929, 1648, 1601, 1541, 1497, 1452, 1370, 1229, 1187, 1156, 1069, 1012, 849, 832, 816, 739, 697, 670 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 7.39 – 7.23 (m, 5H), 7.16 (dd, *J* = 5.1, 1.3 Hz, 1H), 6.89 (dd, *J* = 5.1, 3.5 Hz, 1H), 6.83 (dd, *J* = 3.5, 1.3 Hz, 1H), 5.85 (s, 1H), 4.63 – 4.44 (m, 2H), 3.57 (q, *J* = 7.2 Hz, 1H), 1.53 (d, *J* = 7.2 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.8, 141.1, 141.0, 128.9, 127.6, 127.2, 126.7, 125.5, 124.9, 46.9, 38.4, 18.4. HRMS calcd for $[C_{14}H_{15}NOSH]^+$ ($M+H^+$): 246.0947, found: 246.0946. The *ee* was determined via HPLC on a Chiralpak IC-3 column (15.0% 2-PrOH in hexanes, 1.0 mL/min); t_R: 14.1 min (major), 15.1 min (minor). The enantiomeric excess was determined to be 99%.



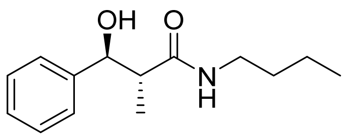
(*S*)-*N*-Butyl-2-(4-methoxyphenyl)propanamide (3j)

Following **GP-1**, **3j** was synthesized from **2e** and butan-1-amine. White solid. Yield: 90% (81.6 mg, 0.38 mmol). Mp: 75-77 °C. $[\alpha]_D^{25}$ +10.8 (*c* 1.0, $CHCl_3$); IR (film): 3299, 3085, 2960, 2928, 2860, 1645, 1557, 1463, 1455, 1369, 1251, 1235, 1034, 840, 791, 529 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 7.26 – 7.19 (m, 2H), 6.95 – 6.83 (m, 2H), 5.45 (s, 1H), 3.80 (s, 3H), 3.50 (q, *J* = 7.2 Hz, 1H), 3.22 – 3.12 (m, 2H), 1.50 (d, *J* = 7.2 Hz, 3H), 1.43 – 1.33 (m, 2H), 1.29 – 1.18 (m, 2H), 0.87 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.5, 158.7, 133.6, 128.7, 114.2, 55.3, 46.3, 39.3, 31.6, 20.0, 18.6, 13.7. HRMS calcd for $[C_{14}H_{21}NO_2H]^+$ ($M+H^+$): 236.1645, found: 236.1648. The *ee* was determined via HPLC on a Chiralpak IC-3 column (15.0% 2-PrOH in hexanes, 1.0 mL/min); t_R: 16.1 min (major), 17.5 min (minor). The enantiomeric excess was determined to be 97%.



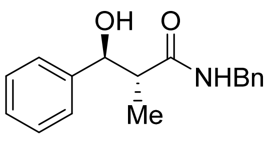
(*S*)-2-(Benzo[d][1,3]dioxol-5-yl)-*N*-butylpropanamide (3k)

Following **GP-1**, **3k** was synthesized from **2f** and butan-1-amine. Yellow oil. Yield: 95% (74 mg, 0.31 mmol). Mp: 62-63 °C. $[\alpha]_D^{25}$ +6.2 (*c* 1.0, $CHCl_3$); IR (film): 3299, 3085, 2930, 2873, 1645, 1549, 1505, 1488, 1440, 1367, 1246, 1185, 1105, 1040, 938, 814 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 6.80 (d, *J* = 1.6 Hz, 1H), 6.75 (s, 1H), 6.73 (dd, *J* = 7.9, 1.6 Hz, 1H), 5.94 (s, 2H), 5.55 (s, 1H), 3.46 (q, *J* = 7.2 Hz, 1H), 3.22 – 3.14 (m, 2H), 1.47 (d, *J* = 7.2 Hz, 3H), 1.44 – 1.35 (m, 2H), 1.31 – 1.18 (m, 2H), 0.87 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.2, 148.0, 146.7, 135.4, 120.8, 108.4, 107.9, 101.1, 46.7, 39.4, 31.6, 20.0, 18.7, 13.7. HRMS calcd for $[C_{14}H_{19}NO_3H]^+$ ($M+H^+$): 250.1438, found: 250.1440. The *ee* was determined via HPLC on a Chiralpak IC-3 column (15.0% 2-PrOH in hexanes, 1.0 mL/min); t_R: 19.5 min (major), 22.7 min (minor). The enantiomeric excess was determined to be 96%.



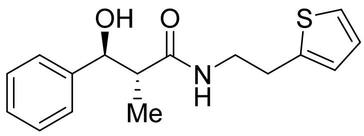
(2*R*,3*S*)-*N*-Butyl-3-hydroxy-2-methyl-3-phenylpropanamide (3l)

Following **GP-1**, **3l** was synthesized from **2h** and butan-1-amine. White solid. Yield: 80% (73 mg, 0.38 mmol). Mp : 117-118 °C. $[\alpha]_D^{20}$ –64.7 (*c* 1.0, $CHCl_3$); IR (film): 3316, 3064, 2960, 2922, 2873, 1747, 1641, 1549, 1454, 1373, 1212, 1156, 1088, 1053, 1015, 755, 703, 611, 546 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 7.31 (m, 4H), 7.28 – 7.23 (m, 1H), 5.93 (s, 1H), 4.69 (d, *J* = 6.3 Hz, 1H), 3.73 (brs, 1H), 3.15 (m, 2H), 2.55 – 2.43 (m, 1H), 1.40 – 1.32 (m, 2H), 1.29 – 1.16 (m, 2H), 1.13 (d, *J* = 7.0 Hz, 3H), 0.86 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.4, 142.7, 128.3, 127.6, 126.2, 76.4, 47.9, 39.1, 31.5, 19.9, 15.6, 13.7. HRMS calcd for $[C_{14}H_{21}NO_2H]^+$ ($M+H^+$): 236.1645, found: 236.1647.



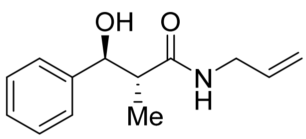
(2*R*,3*S*)-*N*-Benzyl-3-hydroxy-2-methyl-3-phenylpropanamide (3m)

Following **GP-1**, **3m** was synthesized from **2h** and phenylmethanamine. White solid. Yield: 78% (124 mg, 0.59 mmol). Mp: 127-128 °C. $[\alpha]_D^{25}$ –68.5 (*c* 1.0, $CHCl_3$); IR (film): 3323, 3031, 2917, 2849, 1646, 1554, 1496, 1453, 1428, 1384, 1350, 1252, 1217, 1094, 1019, 905, 841, 786, 758, 728, 702, 630, 619, 601, 557 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 7.37 – 7.31 (m, 4H), 7.31 – 7.27 (m, 2H), 7.27 – 7.26 (m, 1H), 7.26 – 7.22 (m, 1H), 7.13 – 7.07 (m, 2H), 6.17 (s, 1H), 4.70 (d, *J* = 6.5 Hz, 1H), 4.39 (dd, *J* = 14.9, 6.0 Hz, 1H), 4.28 (dd, *J* = 14.9, 5.5 Hz, 1H), 4.00 (brs, 1H), 2.65 – 2.44 (m, 1H), 1.14 (d, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.2, 142.6, 137.8, 128.7, 128.5, 127.7, 127.6, 127.5, 126.2, 76.4, 48.1, 43.3, 15.6. HRMS calcd for $[C_{17}H_{19}NO_2Na]^+$ (M+Na^+): 292.1308, found: 292.1306.



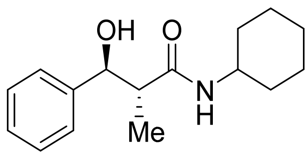
(2*R*,3*S*)-3-Hydroxy-2-methyl-3-phenyl-*N*-(2-(thiophen-2-yl)ethyl)propanamide(3n)

Following **GP-1**, **3n** was synthesized from **2h** and 2-(thiophen-2-yl)ethan-1-amine. White solid. Yield: 68% (115 mg, 0.59 mmol). Mp: 116-117 °C. $[\alpha]_D^{22}$ -50.3 (*c* 1.0, $CHCl_3$); IR (film): 3304, 2966, 2917, 2849, 1643, 1546, 1453, 1384, 1215, 1094, 1018, 912, 854, 824, 758, 692, 541 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 7.36 – 7.31 (m, 3H), 7.30 – 7.27 (m, 2H), 7.14 (dd, *J* = 5.1, 1.2 Hz, 1H), 6.91 (dd, *J* = 5.1, 3.4, Hz, 1H), 6.70 (dd, *J* = 3.4, 1.2 Hz, 1H), 5.80 (s, 1H), 4.71 (d, *J* = 6.5 Hz, 1H), 3.68 – 3.27 (m, 2H), 2.98 (dt, *J* = 15.1, 6.6 Hz, 1H), 2.89 (dt, *J* = 15.1, 6.6 Hz, 1H), 2.50 – 2.41 (m, 1H), 1.12 (d, *J* = 7.1 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.4, 142.5, 141.1, 128.4, 127.7, 127.0, 126.2, 125.4, 124.0, 76.5, 48.0, 40.6, 29.8, 15.5. HRMS calcd for $[C_{16}H_{19}NO_2SH]^+$ (M+H^+): 290.1209, found: 290.1207.



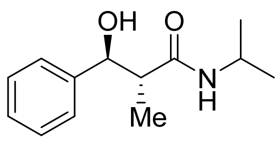
(2*R*,3*S*)-*N*-Allyl-3-hydroxy-2-methyl-3-phenylpropanamide (3o)

Following **GP-1**, **3o** was synthesized from **2h** and prop-2-en-1-amine.White solid. Yield: 66% (85 mg, 0.59 mmol). Mp: 109-111 °C. $[\alpha]_D^{22}$ –77.7 (*c* 1.0, $CHCl_3$); IR (film): 3294, 3084, 3031, 2971, 2917, 2850, 1748, 1644, 1550, 1453, 1377, 1258, 1214, 1092, 1057, 1018, 919, 758, 701 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 7.34 – 7.28 (m, 4H), 7.27 – 7.22 (m, 1H), 6.21 (t, *J* = 6.0 Hz, 1H), 5.76 – 5.62 (m, 1H), 5.04 (t, *J* = 1.7 Hz, 1H), 5.01 (dq, *J* = 7.1, 1.5 Hz, 1H), 4.68 (dd, *J* = 7.0, 3.1 Hz, 1H), 4.16 (d, *J* = 5.1 Hz, 1H), 3.85 – 3.70 (m, 2H), 2.54 (p, *J* = 7.0 Hz, 1H), 1.10 (d, *J* = 7.0 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 175.4, 142.5, 133.8, 128.2, 127.5, 126.2, 115.9, 76.2, 47.7, 41.5, 15.4. HRMS calcd for $[C_{13}H_{17}NO_2H]^+$ (M+H$^+$): 220.1332, found: 220.1332.



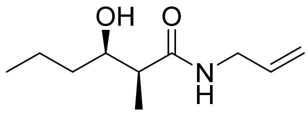
(2*R*,3*S*)-*N*-Cyclohexyl-3-hydroxy-2-methyl-3-phenylpropanamide (3p)

Following **GP-1**, **3p** was synthesized from **2h** and cyclohexanamine. White solid. Yield: 81% (62 mg, 0.29 mmol). Mp: 198-200 °C. $[\alpha]_D^{25}$ –91.0 (*c* 1.0, $CHCl_3$); IR (film): 3433, 3290, 2917, 2849, 1637, 1547, 1453, 1212, 1094, 1018, 753, 699, 648, 583 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 7.35 – 7.30 (m, 4H), 7.26 – 7.23 (m, 1H), 5.46 (d, *J* = 8.3 Hz, 1H), 4.72 (t, *J* = 4.7 Hz, 1H), 4.17 (d, *J* = 5.2 Hz, 1H), 3.77 – 3.61 (m, 1H), 2.47 – 2.40 (m, 1H), 1.89 – 1.79 (m, 1H), 1.79 – 1.69 (m, 2H), 1.69 – 1.53 (m, 2H), 1.35 – 1.25 (m, 2H), 1.18 (d, *J* = 7.1 Hz, 3H), 1.13 – 1.01 (m, 2H), 0.93 (tdd, *J* = 12.3, 10.6, 3.5 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 174.4, 142.8, 128.3, 127.5, 126.1, 76.4, 48.0, 32.8, 25.4, 24.7, 24.7, 15.6. HRMS calcd for $[C_{16}H_{23}NO_2H]^+$ (M+H$^+$): 262.1802, found: 262.1800.



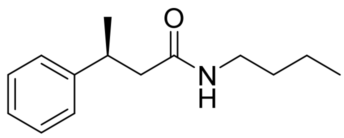
(2*R*,3*S*)-3-Hydroxy-*N*-isopropyl-2-methyl-3-phenylpropanamide (3q)

Following **GP-1**, **3q** was synthesized from **2h** and propan-2-amine. White solid. Yield: 70% (61 mg, 0.38 mmol). Mp: 135-136 °C. $[\alpha]_D^{20}$ –83.3 (*c* 1.0, $CHCl_3$); IR (film): 3440, 3304, 3030, 2973, 2922, 1640, 1548, 1454, 1366, 1216, 1132, 1055, 1017, 926, 757, 702, 610, 544 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.35 – 7.26 (m, 4H), 7.28 – 7.21 (m, 1H), 5.72 (d, *J* = 8.1 Hz, 1H), 4.68 (t, *J* = 6.0 Hz, 1H), 4.33 (d, *J* = 6.0 Hz, 1H), 4.04 – 3.90 (m, 1H), 2.49 – 2.39 (m, 1H), 1.13 (d, *J* = 7.1 Hz, 3H), 1.06 (d, *J* = 6.6 Hz, 3H), 0.97 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 174.4, 142.7, 128.2, 127.4, 126.0, 76.3, 47.7, 41.1, 22.4, 22.4, 15.5. HRMS calcd for $[C_{13}H_{19}NO_2H]^+$ ($M+H^+$): 222.1489, found: 222.1488.



(2*S*,3*R*)-*N*-Allyl-3-hydroxy-2-methylhexanamide (3r)

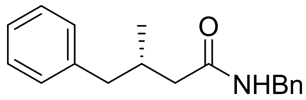
Following **GP-1**, **3r** was synthesized from **2i** and prop-2-en-1-amine**.** White solid. Yield: 70% (47 mg, 0.36 mmol). $[\alpha]_D^{20}$ +5.9 (*c* 1.0, $CHCl_3$) {lit.[10] $[\alpha]_D^{18}$ –11.2 (*c* 1.12, $CHCl_3$) for (2*R*,3*S*)-enantiomer}. IR (film): 3279, 2926, 1748, 1638, 1552, 1456, 1259, 1056, 971, 921, 803, 718 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.01 (s, 1H), 5.91 – 5.76 (m, 1H), 5.29 – 5.10 (m, 2H), 3.89 (t, *J* = 5.4 Hz, 3H), 2.66 (s, 1H), 2.40 – 2.27 (m, 1H), 1.59 – 1.41 (m, 2H), 1.40 – 1.29 (m, 2H), 1.18 (d, *J* = 7.0 Hz, 3H), 0.94 (t, *J* = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 176.3, 134.1, 116.4, 71.8, 44.8, 41.7, 35.8, 19.2, 14.0, 11.2. HRMS calcd for $[C_{10}H_{19}NO_2Na]^+$ ($M+Na^+$): 208.1308, found: 208.1311.



(*S*)-*N*-Butyl-3-phenylbutanamide (3s)

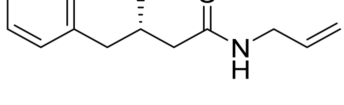
Following **GP-1**, **3s** was synthesized from **2j** and butan-1-amine. Colorless oil. Yield: 77% (61 mg, 0.36 mmol). $[\alpha]_D^{25}$ –25.3 (*c* 0.94, $CHCl_3$) {lit.[11] $[\alpha]_D^{25}$ –25.2 (*c* 1.13, $CHCl_3$)}. IR (film): 3302, 3085, 3028, 2961, 2930, 2872, 1643, 1556, 1494, 1453, 1376, 1260, 1225, 1082, 1019, 912, 803, 761, 699, 538 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.33 – 7.25 (m, 2H), 7.25 – 7.15 (m, 3H), 5.53 (s, 1H), 3.37 – 3.23 (m, 1H), 3.20 – 3.06 (m, 2H), 2.47 – 2.34 (m, 2H), 1.34 – 1.25 (m, 5H), 1.23 – 1.13 (m, 2H), 0.85 (t, *J* = 7.3 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.5, 145.9, 128.4, 126.7, 126.3, 45.8, 39.0, 37.0, 31.5, 21.6, 19.8, 13.6.

HRMS calcd for $[C_{14}H_{21}NOH]^+$ ($M+H^+$): 220.1696, found: 220.1698. The *ee* was determined via HPLC on a Chiralcel OD-3 column (15.0% 2-PrOH in hexanes, 1.0 mL/min); t_R: 6.2 min (major), 7.0 min (minor). The enantiomeric excess was determined to be 99%.



(*S*)-*N*-Benzyl-3-methyl-4-phenylbutanamide (3t)

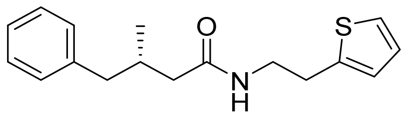
Following **GP-1**, **3t** was synthesized from **2k** and phenylmethanamine. Colorless oil. Yield: 74% (122 mg, 0.62 mmol). $[\alpha]_D^{25}$ +5.0 (*c* 1.0, $CHCl_3$). IR (film): 3288, 3063, 3028, 2959, 2924, 2870, 1644, 1549, 1496, 1454, 1378, 1261, 1164, 1093, 1029, 802, 738, 699 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 7.36 – 7.28 (m, 2H), 7.25 (tt, *J* = 6.3, 1.7 Hz, 5H), 7.20 – 7.15 (m, 1H), 7.15 – 7.11 (m, 2H), 5.76 (s, 1H), 4.47 – 4.35 (m, 2H), 2.65 (dd, *J* = 13.4, 6.5 Hz, 1H), 2.48 (dd, *J* = 13.4, 7.8 Hz, 1H), 2.40 – 2.27 (m, 1H), 2.24 (dd, *J* = 14.0, 5.8 Hz, 1H), 1.97 (dd, *J* = 14.0, 8.3 Hz, 1H), 0.94 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.1, 140.2, 138.3, 129.2, 128.6, 128.2, 127.7, 127.4, 125.9, 43.5, 43.5, 43.1, 32.6, 19.5. HRMS calcd for $[C_{18}H_{21}NOH]^+$ ($M+H^+$): 268.1696, found: 268.1691. The *ee* was determined via HPLC on a Chiralcel OJ-H column (15.0% 2-PrOH in hexanes, 1.0 mL/min); t_R: 3.672 min (major), 3.866 min (minor). The enantiomeric excess was determined to be 94%.



(*S*)-*N*-Allyl-3-methyl-4-phenylbutanamide (3u)

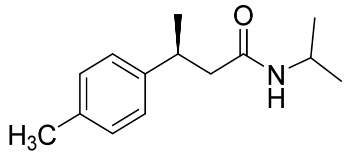
Following **GP-1**, **3u** was synthesized from **2k** and prop-2-en-1-amine. Colorless oil. Yield: 58% (78 mg, 0.62 mmol). $[\alpha]_D^{25}$ +3.5 (*c* 1.0, $CHCl_3$). IR (film): 3291, 3083, 3027, 2961, 2922, 2870, 2850, 1644, 1549, 1496, 1454, 1418, 1379, 1261, 1093, 1030, 918, 804, 741, 700 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 7.31 – 7.23 (m, 2H), 7.19 (d, *J* = 7.5 Hz, 1H), 7.18 – 7.13 (m, 2H), 5.96 – 5.75 (m, 1H), 5.68 (s, 1H), 5.24 – 5.04 (m, 2H), 3.97 – 3.77 (m, 2H), 2.65 (dd, *J* = 13.4, 6.5 Hz, 1H), 2.48 (dd, *J* = 13.4, 7.7 Hz, 1H), 2.39 – 2.25 (m, 1H), 2.23

(dd, *J* = 14.0, 5.7 Hz, 1H), 1.97 (dd, *J* = 14.0, 8.3 Hz, 1H), 0.94 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.1, 140.2, 134.3, 129.2, 128.2, 125.9, 116.3, 43.5, 43.1, 41.8, 32.5, 19.5. HRMS calcd for $[C_{14}H_{19}NOH]^+$ (M+H$^+$): 218.1539, found: 218.1539. The *ee* was determined via HPLC on a Chiralpak AD-H column (10.0% 2-PrOH in hexanes, 1.0 mL/min); t_R: 10.385 min (major), 15.288 min (minor). The enantiomeric excess was determined to be 95%.



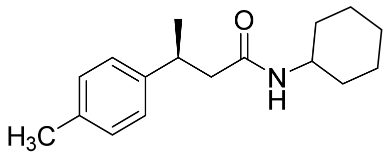
(*S*)-3-Methyl-4-phenyl-*N*-(2-(thiophen-2-yl)ethyl)butanamide (3v)

Following **GP-1**, **3v** was synthesized from **2k** and 2-(thiophen-2-yl)ethan-1-amine.Yellow oil. Yield: 58% (103 mg, 0.62 mmol). $[\alpha]_D^{25}$ +3.4 (*c* 1.0, $CHCl_3$). IR (film): 3296, 3066, 3026, 2957, 2921, 2850, 1644, 1548, 1496, 1454, 1378, 1261, 1167, 1095, 1029, 850, 803, 741, 699, 593 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.28 – 7.23 (m, 2H), 7.21 – 7.16 (m, 1H), 7.16 – 7.11 (m, 3H), 6.93 (dd, *J* = 5.2, 3.4 Hz, 1H), 6.85 – 6.75 (m, 1H), 5.65 (t, *J* = 6.2 Hz, 1H), 3.51 (q, *J* = 6.5 Hz, 2H), 3.01 (t, *J* = 6.6 Hz, 2H), 2.62 (dd, *J* = 13.4, 6.5 Hz, 1H), 2.45 (dd, *J* = 13.4, 7.8 Hz, 1H), 2.35 – 2.23 (m, 1H), 2.17 (dd, *J* = 14.0, 5.8 Hz, 1H), 1.91 (dd, *J* = 14.0, 8.3 Hz, 1H), 0.90 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 172.3, 141.3, 140.2, 129.2, 128.2, 127.0, 125.9, 125.3, 123.9, 43.6, 43.0, 40.7, 32.5, 29.9, 19.5. HRMS calcd for $[C_{17}H_{21}NOSH]^+$ (M+H$^+$): 288.1417, found: 288.1413. The *ee* was determined via HPLC on a Chiralpak AD-H column (15.0% 2-PrOH in hexanes, 1.0 mL/min); t_R: 5.966 min (major), 7.011 min (minor). The enantiomeric excess was determined to be 95%.



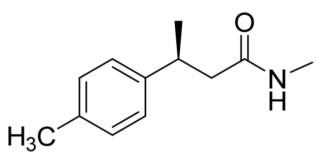
(*S*)-*N*-Isopropyl-3-(*p*-tolyl)butanamide (3w)

Following **GP-1**, **3w** was synthesized from **2l** and propan-2-amine. White solid. Yield: 54% (90 mg, 0.77 mmol). Mp: 95-97 °C. $[\alpha]_D^{25}$ +22.8 (*c* 1.0, $CHCl_3$). IR (film): 3301, 2972, 2916, 2849, 1634, 1541, 1516, 1375, 1094, 1019, 816, 692, 617, 602 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.10 (s, 4H), 5.23 (d, *J* = 7.2 Hz, 1H), 4.03 – 3.91 (m, 1H), 3.30 – 3.17 (m, 1H), 2.34 (dd, *J* = 7.5, 1.8 Hz, 2H), 2.31 (s, 3H), 1.28 (d, *J* = 7.0 Hz, 3H), 1.05 (d, *J* = 6.6 Hz, 3H), 0.94 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.8, 142.8, 135.8, 129.1, 126.6, 46.1, 41.0, 36.7, 22.6, 22.5, 21.6, 20.9. HRMS calcd for $[C_{14}H_{21}NOH]^+$ ($M+H^+$): 220.1696, found: 220.1695. The *ee* was determined via HPLC on a Chiralcel OD-H column (15.0% 2-PrOH in hexanes, 1.0 mL/min); t_R: 6.044 min (minor), 6.402 min (major). The enantiomeric excess was determined to be 97%.



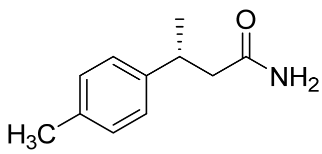
(*S*)-*N*-Cyclohexyl-3-(*p*-tolyl)butanamide (3x)

Following **GP-1**, **3x** was synthesized from **2l** and cyclohexanamine. White solid. Yield: 55% (44 mg, 0.31 mmol). Mp: 128-129 °C. $[\alpha]_D^{25}$ +22.8 (*c* 1.0, $CHCl_3$). IR (film): 3295, 3070, 2932, 2854, 1738, 1636, 1544, 1515, 1448, 1422, 1379, 1306, 1260, 1209, 1189, 1152, 1087, 1020, 987, 961, 891, 818, 721, 610, 568, 534 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.10 (s, 4H), 5.15 (d, *J* = 8.2 Hz, 1H), 3.76 – 3.60 (m, 1H), 3.27 – 3.17 (m, 1H), 2.36 (dd, *J* = 7.5, 4.9 Hz, 2H), 2.31 (s, 3H), 1.87 – 1.77 (m, 1H), 1.74 – 1.64 (m, 1H), 1.64 – 1.46 (m, 3H), 1.39 – 1.18 (m, 5H), 1.18 – 1.04 (m, 1H), 1.04 – 0.96 (m, 1H), 0.89 (tdd, *J* = 12.1, 10.5, 3.6 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 170.7, 142.8, 135.8, 129.1, 126.6, 47.8, 46.2, 36.7, 33.0, 32.8, 25.5, 24.7, 24.6, 21.7, 20.9. HRMS calcd for $[C_{17}H_{25}NOH]^+$ ($M+H^+$): 260.2009, found: 260.2008. The *ee* was determined via HPLC on a Chiralpak AD-H column (10.0% 2-PrOH in hexanes, 1.0 mL/min); t_R: 7.580 min (major), 9.004 min (minor). The enantiomeric excess was determined to be 98%.



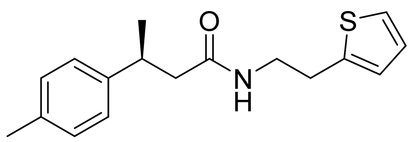
(*S*)-*N*-Methyl-3-(*p*-tolyl)butanamide (3y)

Following **GP-1, 3y** was synthesized from **2m** and methylamine as a white solid. Yield: 75% (2.79 g, 14.5 mmol). Mp: 85.3-86.3 °C. $[\alpha]_D^{25}$ +31.4 (*c* 0.5, $CHCl_3$). IR (film): 3457, 2961, 1644, 1560, 1515, 1455, 1411, 1159, 1105, 816, 702, 543 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 7.11 (s, 4H), 5.37 (s, 1H), 3.41 – 3.16 (m, 1H), 2.71 (d, *J* = 3.3 Hz, 3H), 2.50 – 2.33 (m, 2H), 2.31 (s, 3H), 1.28 (d, *J* = 6.8 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.5, 142.8, 135.9, 129.2, 126.6, 45.8, 36.5, 26.3, 21.7, 21.0. HRMS calcd for $[C_{12}H_{17}NOH]^+$ $(M+H^+)$: 192.1383, found: 192.1384. The *ee* was determined via HPLC on a Chiralpak AD-H column (5.0% 2-PrOH in hexanes, 1.0 mL/min); t_R: 12.9 min (major), 15.1 min (minor). The enantiomeric excess was determined to be 98%.



(*R*)-3-(*p*-Tolyl)butanamide (3z)

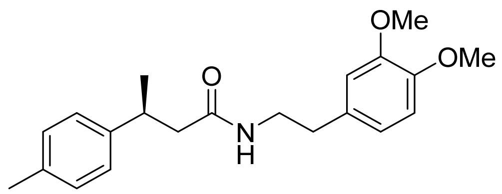
Following **GP-1, 3z** was synthesized from **2n** and ammonia. White solid. Yield: 71% (25 mg, 0.2 mmol). Mp: 103.4-103.9 °C. $[\alpha]_D^{25}$ –5.2 (*c* 1.0, $CHCl_3$). IR (film): 3393, 3199, 2957, 2920, 1651, 1409, 1130, 812 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 7.12 (d, *J* = 1.7 Hz, 4H), 5.35 (s, 1H), 5.23 (s, 1H), 3.35 – 3.14 (m, 1H), 2.50 (ddd, *J* = 14.3, 7.4, 2.7 Hz, 1H), 2.42 (ddd, *J* = 14.3, 7.4, 2.5 Hz, 1H), 2.31 (s, 3H), 1.31 (d, *J* = 7.0, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.2, 142.7, 136.0, 129.3, 126.6, 45.0, 36.4, 21.9, 21.0. HRMS calcd for $[C_{11}H_{15}NOH]^+$ $(M+H^+)$: 178.1226, found: 178.1228. The *ee* was determined via HPLC on a Chiralpak AD-H column (5.0% 2-PrOH in hexanes, 1.0 mL/min); t_R: 11.6 min (minor), 14.7 min (major). The enantiomeric excess was determined to be 97%.



(*S*)-*N*-(2-(Thiophen-2-yl)ethyl)-3-(*p*-tolyl)butanamide (3aa)

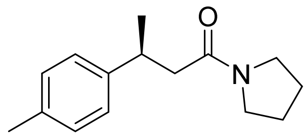
Following **GP-1**, **3aa** was synthesized from **2m** and 2-(thiophen-2-yl)ethan-1-amine. Yellow solid. Yield: 72% (41 mg, 0.2 mmol). Mp: 73.5-74.3 °C. $[\alpha]_D^{25}$ +22.4 (*c* 1.0, $CHCl_3$).

IR (film): 3301, 2960, 2920, 1644, 1551, 1514, 1434, 1350, 1017, 816, 695 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.14 (dd, *J* = 5.1, 1.1 Hz, 1H), 7.09 (s, 4H), 6.91 (dd, *J* = 5.1, 3.4 Hz, 1H), 6.66 (dd, *J* = 3.4, 1.1 Hz, 1H), 5.40 (s, 1H), 3.54 – 3.32 (m, 2H), 3.31 – 3.14 (m, 1H), 3.01 – 2.76 (m, 2H), 2.37 (dd, *J* = 7.5, 4.2 Hz, 2H), 2.31 (s, 3H), 1.27 (d, *J* = 7.0 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.7, 142.8, 141.3, 135.9, 129.2, 127.0, 126.6, 125.3, 123.8, 45.9, 40.6, 36.5, 29.8, 21.8, 21.0. HRMS calcd for $[C_{17}H_{21}NOSH]^+$ (M+H$^+$): 288.1417, found: 288.1418. The *ee* was determined via HPLC on a Chiralcel OD-H column (5.0% 2-PrOH in hexanes, 1.0 mL/min); t_R: 35.3 min (minor), 39.9 min (major). The enantiomeric excess was determined to be 95%.



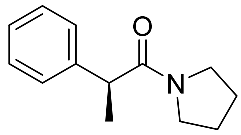
(*S*)-*N*-(3,4-Dimethoxyphenethyl)-3-(*p*-tolyl)butanamide (3ab)

Following **GP-1**, **3ab** was synthesized from **2m** and 2-(3,4-dimethoxyphenyl)ethan-1-amine as a white solid. Yield: 83% (141.9 mg, 0.42 mmol). $[\alpha]_D^{20}$ +5.0 (*c* 1.0, $CHCl_3$). IR (film): 3304, 3057, 3000, 2962, 2938, 2870, 2839, 1637, 1544, 1516, 1456, 1259, 1236, 1157, 1144, 1028, 963, 894, 850, 808, 765, 731, 630, 534 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.08 (s, 4H), 6.76 (d, *J* = 8.1 Hz, 1H), 6.65 (d, *J* = 2.1 Hz, 1H), 6.58 (dd, *J* = 8.1, 2.1 Hz, 1H), 5.51 (t, *J* = 5.9 Hz, 1H), 3.85 (s, 3H), 3.84 (s, 3H), 3.49 – 3.32 (m, 2H), 3.28 – 3.17 (m, 1H), 2.65 (dt, *J* = 14.0, 6.9 Hz, 1H), 2.59 (dt, *J* = 14.0, 7.2 Hz, 1H), 2.41 – 2.32 (m, 2H), 2.31 (s, 3H), 1.26 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 171.7, 148.9, 147.5, 142.7, 135.7, 131.3, 129.1, 126.5, 120.5, 111.7, 111.2, 55.8, 55.7, 45.7, 40.5, 36.4, 35.1, 21.6, 20.9. HRMS calcd for $[C_{21}H_{27}NO_3Na]^+$ (M+Na$^+$): 364.1883, found: 364.1885. The *ee* was determined via HPLC on a Chiralcel AD-H column (10.0% 2-PrOH in hexanes, 1.0 mL/min); t_R: 14.7 min (major), 16.6 min (minor). The enantiomeric excess was determined to be 99%.



(*S*)-1-(Pyrrolidin-1-yl)-3-(*p*-tolyl)butan-1-one (3ad)

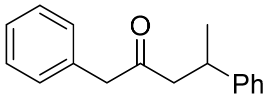
Following **GP-1**, **3ad** was synthesized from **2m** and pyrrolidine. Colorless oil. Yield: 85% (39 mg, 0.2 mmol). $[\alpha]_D^{25}$ +43.5 (*c* 0.5, $CHCl_3$). IR (film): 2944, 2912, 1646, 1547, 1434, 1291, 816, 714 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ ^1H NMR (400 MHz, $CDCl_3$) δ 7.24 – 7.06 (m, 4H), 3.50 – 3.28 (m, 4H), 3.20 – 3.10 (m, 1H), 2.57 – 2.40 (m, 2H), 2.31 (s, 2H), 1.90 – 1.75 (m, 4H), 1.32 (d, *J* = 7.0 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.7, 142.8, 141.3, 135.9, 129.2, 127.0, 126.6, 125.3, 123.8, 45.9, 40.6, 36.5, 29.8, 21.8, 21.0. HRMS calcd for $[C_{15}H_{21}NNaO]^+$ ($M+Na^+$): 254.1521, found: 254.1524. The *ee* was determined via HPLC on a Chiralcel AD-H column (10% 2-PrOH in hexanes, 1.0 mL/min); t_R: 15.4 min (major), 17.8 min (minor). The enantiomeric excess was determined to be 98%.



(*S*)-2-Phenyl-1-(pyrrolidin-1-yl)propan-1-one (3ae)

Following **GP-1**, **3ae** was synthesized from **2d** and pyrrolidine. Colorless oil. (30 mg, 0.34 mmol, yield: 50%). $[\alpha]_D^{25}$ +62.5 (*c* 1.0, $CHCl_3$); IR (film): 2972, 2927, 2870, 1640, 1489, 1457, 1424, 1373, 1339, 1303, 1257, 1192, 1061, 1034, 804, 756, 726, 702, 586, 575 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 7.30 (m, 4H), 7.28 – 7.17 (m, 1H), 3.73 (q, *J* = 6.9 Hz, 1H), 3.54 (dt, *J* = 12.2, 6.1 Hz, 1H), 3.51 – 3.36 (m, 2H), 3.21 – 3.10 (m, 1H), 1.88 (d, *J* = 6.1 Hz, 1H), 1.78 (m, 3H), 1.45 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.1, 141.7, 128.7, 127.5, 126.6, 46.2, 46.0, 45.0, 26.0, 24.1, 20.3. HRMS calcd for $[C_{13}H_{17}NOH]^+$ ($M+H^+$): 204.1383, found: 204.1383. The *ee* was determined via HPLC on a CHIRALPAK AD-3 column (15.0% 2-PrOH in hexanes, 1.0 mL/min); t_R: 5.3 min (major), 6.2 min (minor). The enantiomeric excess was determined to be 87%.

IV. Transformations of the transamidation products: (Chiral) amides

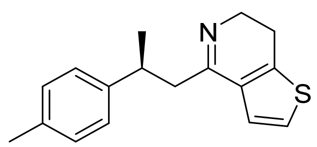


1,4-Diphenylpentan-2-one (4a)

Tf_2O (0.55 mmol, 1.1 equiv) was added dropwise to a solution of secondary amide **3ac** (0.5 mmol, 1.0 equiv) and 2-fluoropyridine (0.6 mmol, 1.2 equiv) in CH_2Cl_2 (2 mL, 0.25 mol/L) at 0 °C. After being stirred for 15 min at 0 °C, prop-1-en-2-ylbenzene (0.6 mmol, 1.2 equiv) was added dropwise to the solution. The mixture was warmed up to room temperature and stirred for 3 h. Quenched with saturated aqueous NH_4Cl solution (3 mL), the mixture was extracted with CH_2Cl_2 (3 × 10 mL). The combined organic layers were washed with saturated Na_2CO_3 aqueous solution (5 mL) and brine (5 mL), dried over anhydrous Na_2SO_4. The volatiles were removed under reduced pressure. The residue was purified by column chromatography on silica gel (Ether / Petroleum ether = 1 / 50) to give the desired ketone **4a** as a colorless oil in 65% yield (85 mg). IR (film): 3062, 3029, 2964, 2931, 1705, 1603, 1584, 1495, 1453, 1414, 1376, 1318, 1285, 1177, 1114, 1071, 1026, 762, 700 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 7.33 – 7.18 (m, 5H), 7.22 – 7.10 (m, 3H), 7.12 – 7.04 (m, 2H), 3.55 (s, 2H), 3.30 (h, *J* = 7.0 Hz, 1H), 2.76 (dd, *J* = 16.4, 6.6 Hz, 1H), 2.64 (dd, *J* = 16.4, 7.7 Hz, 1H), 1.21 (d, *J* = 7.0 Hz, 3H). ^{13}C NMR (151 MHz, $CDCl_3$) δ 207.1, 146.0, 133.9, 129.4, 128.6, 128.5, 126.9, 126.7, 126.2, 50.7, 50.2, 35.4, 21.8. HRMS calcd for $[C_{17}H_{18}OH]^+$ $(M+H^+)$: 239.1430, found: 239.1429.

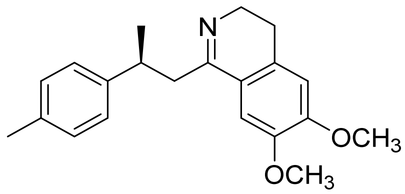
General procedure 2 (GP-2)

Tf_2O (1.1 equiv) was added dropwise to a solution of secondary amide (1.0 equiv) and 2-fluoropyridine (1.2 equiv) in CH_2Cl_2 (0.1 mol/L) at 0 °C. The mixture was stirred at 0 °C for about 1 h. The volatiles were removed under reduced pressure. The residue was purified by column chromatography on silica gel (elution Et_2O/Hexane and 0.10% triethylamine).



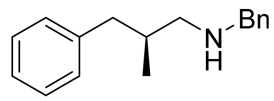
(*S*)-4-(2-(*p*-Tolyl)propyl)-6,7-dihydrothieno[3,2-*c*]pyridine (4b)

Following **GP-2**, **4b** was synthesized from **3aa**. $[\alpha]_D^{25}$ +45.0 (*c* 0.5, $CHCl_3$). IR (film): 2983, 2936, 1651, 1567, 1541, 1468, 1450, 1380, 1351, 1261, 1178, 1154, 771, 596, 530 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 7.09 (dd, *J* = 8.4, 2.7 Hz, 4H), 7.06 (s, 2H), 3.90 – 3.62 (m, 2H), 3.30 – 3.12 (m, 1H), 2.91 (dd, *J* = 13.8, 5.5 Hz, 1H), 2.81 – 2.67 (m, 3H), 2.31 (s, 3H), 1.27 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 163.1, 143.8, 143.3, 135.6, 131.7, 129.0, 126.7, 124.0, 121.9, 47.5, 45.9, 37.3, 22.2, 21.2, 21.0. HRMS calcd for $[C_{17}H_{19}NSH]^+$ ($M+H^+$): 270.1311, found 270.1311. The *ee* was determined via HPLC on a Chiralcel OD-H column (5.0% 2-PrOH in hexanes, 1.0 mL/min); t_R: 6.8 min (major), 7.0 min (minor). The enantiomeric excess was determined to be 90%.



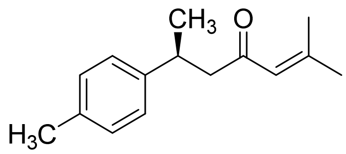
(*S*)-6,7-Dimethoxy-1-(2-(*p*-tolyl)propyl)-3,4-dihydroisoquinoline (4c)

Following **GP-2**, **4c** was synthesized from **3ab**. Yellow oil. Yield: 92% (43 mg, 0.15 mmol). $[\alpha]_D^{25}$ +32.7 (*c* 1.0, $CHCl_3$). IR (film): 3000, 2935, 2930, 2835, 1622, 1605, 1570, 1514, 1464, 1405, 1358, 1319, 1268, 1229, 1213, 1147, 1115, 1098, 1069, 1035, 963, 860, 815, 545 cm^{-1}. ^1H NMR (400 MHz, $CDCl_3$) δ 7.17 – 7.05 (m, 4H), 6.91 (s, 1H), 6.69 (s, 1H), 3.91 (s, 3H), 3.85 (s, 3H), 3.72 – 3.61 (m, 1H), 3.61 – 3.51 (m, 1H), 3.30 – 3.13 (m, 1H), 3.02 (dd, *J* = 14.0, 5.5 Hz, 1H), 2.81 (dd, *J* = 14.0, 9.1 Hz, 1H), 2.65 – 2.47 (m, 2H), 2.31 (s, 3H), 1.28 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 165.7, 150.6, 147.4, 144.0, 135.5, 131.7, 129.1, 126.7, 122.0, 110.3, 109.0, 56.2, 55.9, 46.9, 44.8, 37.6, 25.9, 21.2, 20.9. HRMS calcd for $[C_{21}H_{25}NO_2Na]^+$ ($M+Na^+$): 346.1778, found: 346.1783. The *ee* was determined by HPLC on a Chiralcel AD-H column (20.0% 2-PrOH in hexanes, 1.0 mL/min); t_R = 5.4 min (major), 7.1 min (minor). The enantiomeric excess was determined to be 98%.



(*S*)-*N*-Benzyl-2-methyl-3-phenylpropan-1-amine (4d)

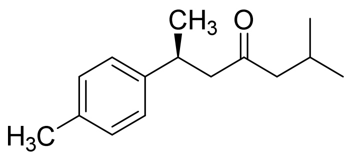
To a solution of secondary amide **3c** (0.32 mmol, 1.0 equiv) and $[Ir(COE)_2Cl]_2$ (1.6 μmol, 0.5 mol%) in CH_2Cl_2 (1.6 mL, 0.2 mol/L) was added Et_2SiH_2 (0.63 mmol, 4.0 equiv). The solution was stirred at room temperature for 1-2 h until completion of the reaction as indicated by TLC analysis. CH_2Cl_2 was removed and the resulting residue was purified by flash chromatography on silica gel eluting with petroleum ether/Et_2O/triethylamine (10/1/0.001, *v/v/v*) mixture to amine **4d** (61 mg, 0.32 mmol, yield: 81%) as a pale yellow oil. $[\alpha]_D^{20}$ +13.9 (*c* 1.0, $CHCl_3$). IR (film): 3331, 3084, 3062, 3026, 2954, 2923, 1675, 1603, 1495, 1453, 1377, 1308, 1188, 1119, 1029, 970, 739, 699 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 7.35 – 7.26 (m, 4H), 7.26 – 7.21 (m, 3H), 7.18 (d, *J* = 7.3 Hz, 1H), 7.14 (dd, *J* = 7.3, 5.7 Hz, 2H), 3.84 – 3.67 (m, 2H), 2.74 (dd, *J* = 13.4, 6.0 Hz, 1H), 2.59 (dd, *J* = 11.7, 6.0 Hz, 1H), 2.48 (dd, *J* = 11.7, 6.9 Hz, 1H), 2.38 (dd, *J* = 13.4, 8.3 Hz, 1H), 2.01 – 1.90 (m, 1H), 0.89 (d, *J* = 6.7 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 141.0, 140.6, 129.1, 128.3, 128.1, 128.1, 126.8, 125.7, 55.4, 54.1, 41.4, 35.4, 18.0. HRMS calcd for $[C_{17}H_{21}NH]^+$ ($M+H^+$): 240.1747, found: 240.1745. The *ee* was determined via HPLC on a Chiralcel OD-3 column (5.0% 2-PrOH in hexanes, 1.0 mL/min); t_R = 5.6 min (major), 6.6 min (minor). The enantiomeric excess was determined to be 99%.



(*S*)-2-Methyl-6-(*p*-tolyl)hept-2-en-4-one (K-1)

Tf_2O (0.22 mmol, 1.1 equiv) was added dropwise to a solution of secondary amide **3ap** (0.2 mmol, 1.0 equiv) and 2-fluoropyridine (0.24 mmol, 1.2 equiv) in CH_2Cl_2 (1 mL, 0.2 mol/L, newly distilled) at 0 °C. After being stirred for 30 minutes, 2-methylprop-1-ene (0.3 mmol, 1.5 equiv, 2.4 M THF solution) was added portion-wise over 1 hour. The solution was allowed to warm up to room temperature and stirred for 3 hours. To the mixture were added THF (2 mL), $TsOH \cdot H_2O$ (0.2 mmol, 1.0 equiv) and H_2O (0.1 mL), and the resulting mixtures was refluxed overnight. After being cooled to room temperature, the mixture was extracted with CH_2Cl_2 (3 × 5 mL). The combined organic phases were washed with an aq. Solution of Na_2CO_3, dried over anhydrous sodium sulfate, filtered, and concentrated under reduced pressure. The residue was purified by silica gel column chromatography eluting with hexane/ethyl acetate (30/1, *v/v*) mixture to an inseparable mixture of three ketones: (+)-(*S*)-*ar*-turmerone (**K-1**), (+)-(*S*)-dihydro-*ar*-turmerone (**K-2**), and non-conjugate enone **5** in 75% yield (28 mg). According to the ^{1}H NMR spectrum of the mixture, the yield of each ketone was 71%, 2% and 2%, respectively. (+)-(*S*)-Dihydro-*ar*-turmerone (**K-2**) could be removed from the mixture by repeated flash chromatographic separation.

(+)-(*S*)-*ar*-Turmerone (**K-1**): 98% *ee,* $[\alpha]_D^{20}$ +61.9 (*c* 0.5, $CHCl_3$) {lit.[8] $[\alpha]_D^{20}$ = +61.1 (*c* 1.50, $CHCl_3$), 98% *ee*}. IR (film): 2961, 2923, 1686, 1620, 1515, 1444, 1382, 1358, 1110, 1036, 1012, 815 cm^{-1}. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.10 (s, 4H), 6.02 (s, 1H), 3.29 (h, *J* = 6.9 Hz, 1H), 2.71 (dd, *J* = 15.7, 6.0 Hz, 1H), 2.61 (dd, *J* = 15.7, 8.3 Hz, 1H), 2.31 (s, 3H), 2.11 (s, 3H), 1.85 (s, 3H), 1.24 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 199.9, 155.1, 143.7, 135.5, 129.1, 126.6, 124.1, 52.7, 35.3, 27.6, 22.0, 21.0, 20.7. The *ee* was determined via HPLC on a Chiralcel OJ-H column (1.0% 2-PrOH in hexane, 1.0 mL/min); t_R: 9.5 min (major), 10.9 min (minor). The enantiomeric excess was determined to be 98%. HRMS Calcd for $[C_{15}H_{20}ONa]^+$ (M+Na$^+$): 239.1406, found 239.1407.



(*S*)-2-Methyl-6-(*p*-tolyl)heptan-4-one (K-2)

$[\alpha]_D^{25}$ +25.4 (*c* 1.0, $CHCl_3$) {lit.[9] $[\alpha]_D^{25}$ +30.3 (*c* 0.5, CH_2Cl_2)}. IR (film): 2960, 2926, 2871, 1713, 1646, 1515, 1464, 1408, 1365, 1310, 1261, 1185, 1101, 1023, 971, 863, 804, 706 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.02 (s, 4H), 3.27 – 3.15 (m, 1H), 2.60 (dd, *J* = 16.2, 6.4 Hz, 1H), 2.51 (dd, *J* = 16.2, 8.0 Hz, 1H), 2.24 (s, 3H), 2.24 – 2.05 (m, 2H), 2.05 – 1.95 (m, 1H), 1.16 (d, *J* = 6.9 Hz, 3H), 0.78 (d, *J* = 4.7 Hz, 3H), 0.77 (d, *J* = 4.7 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) *δ* 209.9, 143.3, 135.7, 129.1, 126.6, 52.5, 51.7, 34.9, 24.4, 22.5, 22.5, 22.0, 21.0. HRMS calcd for $[C_{15}H_{22}OH]^+$ (M+H$^+$): 219.1743, found: 219.1743.

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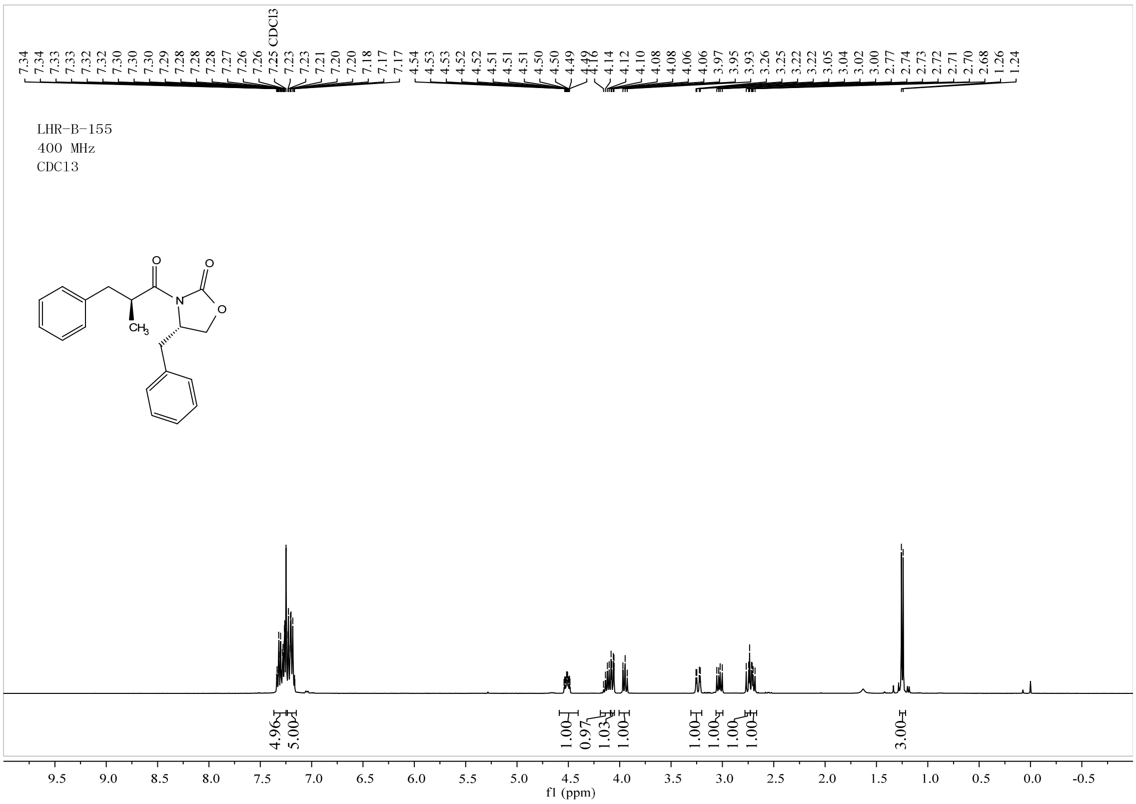
V. ^{1}H and ^{13}C NMR Spectra



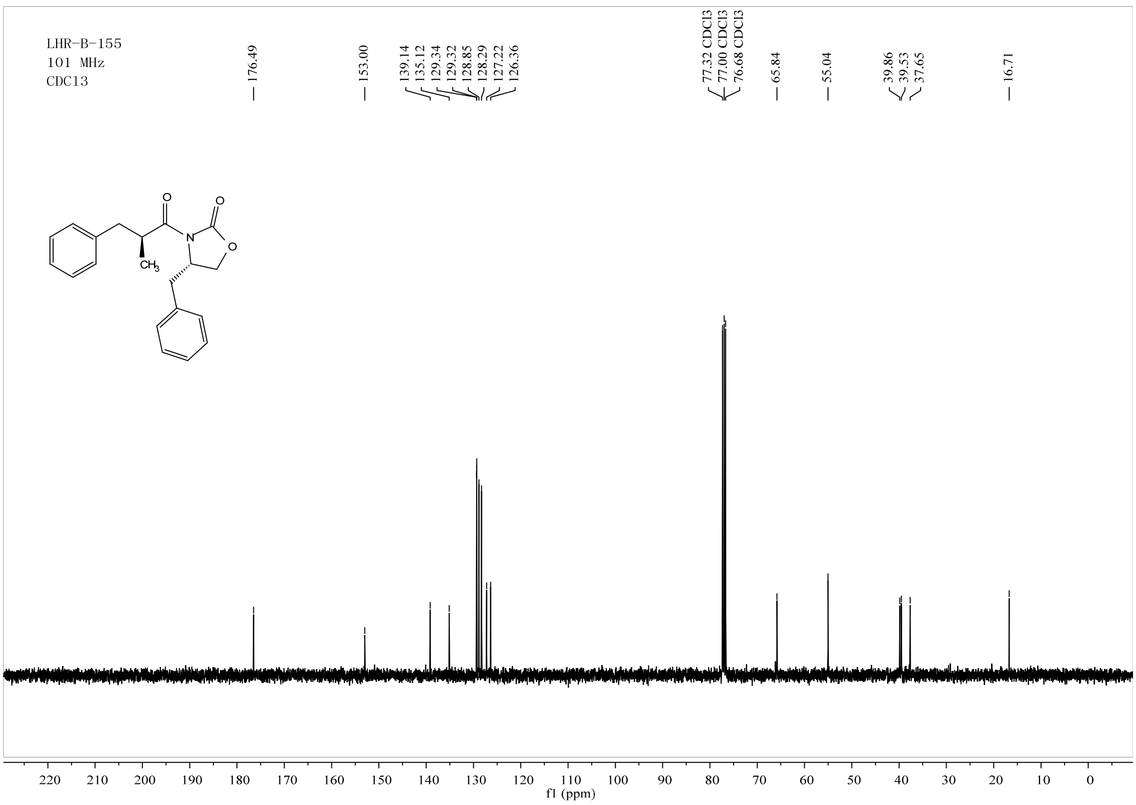
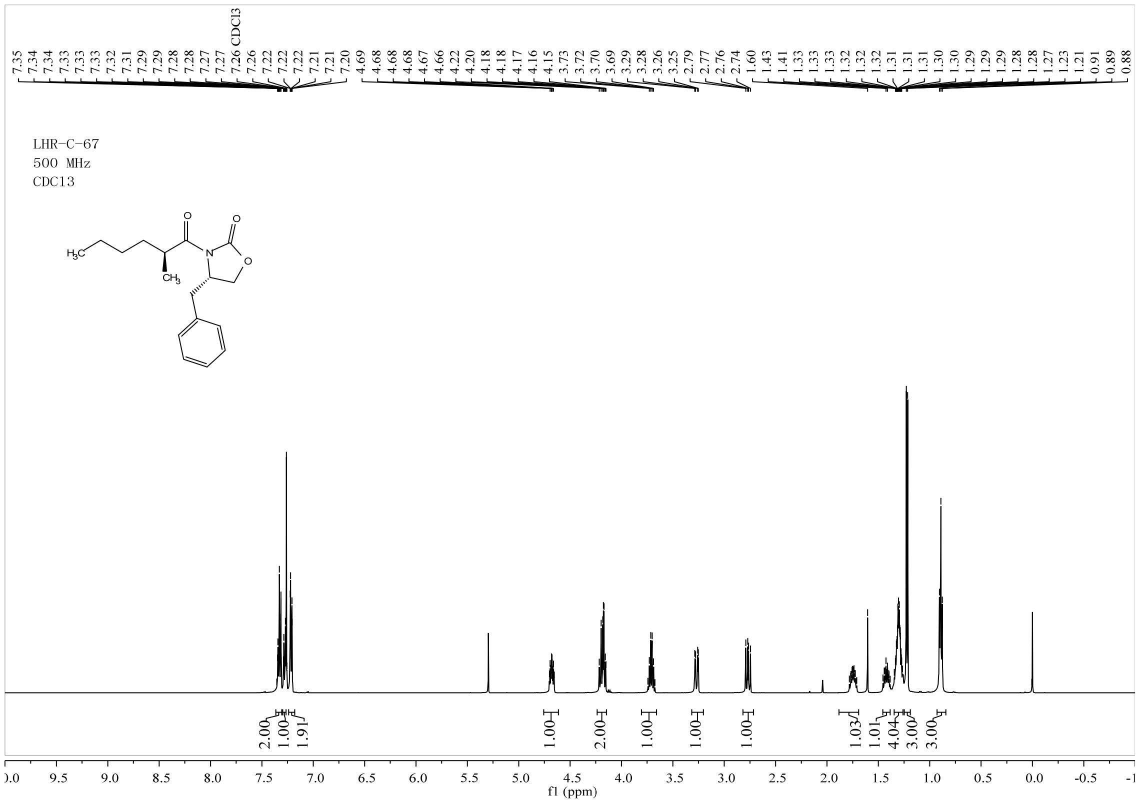


Figure S-1. ^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **2a.**



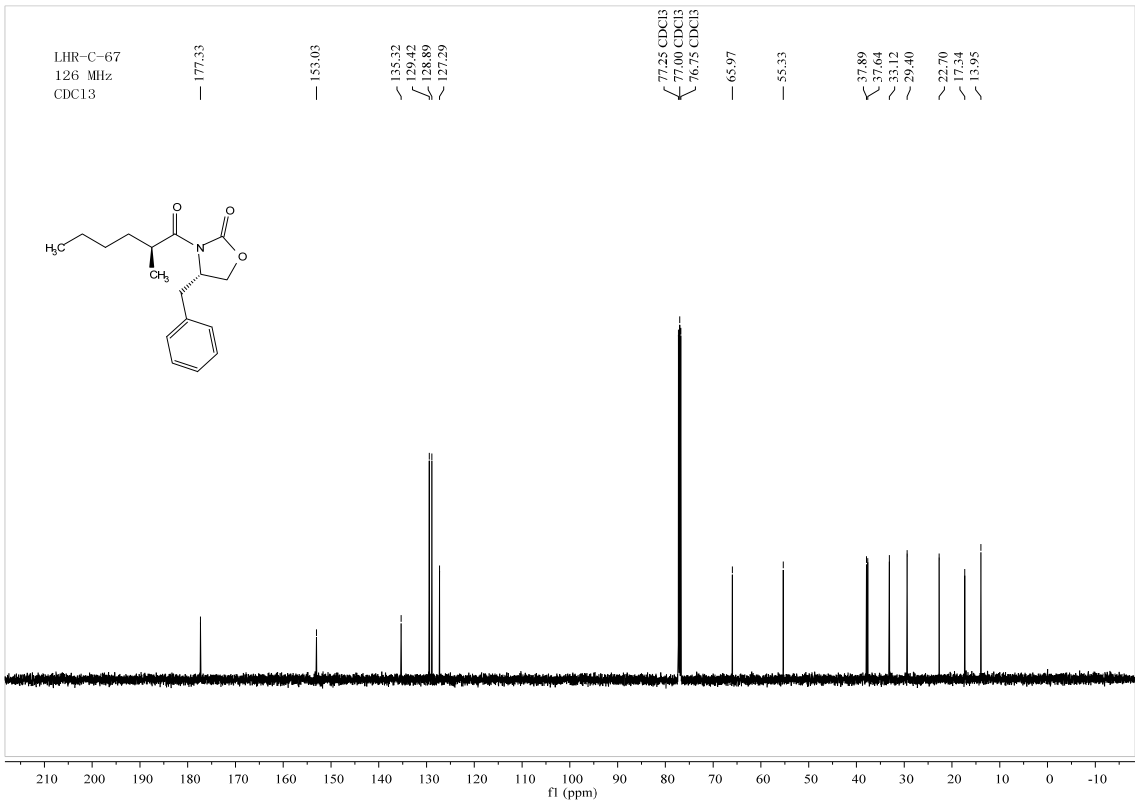
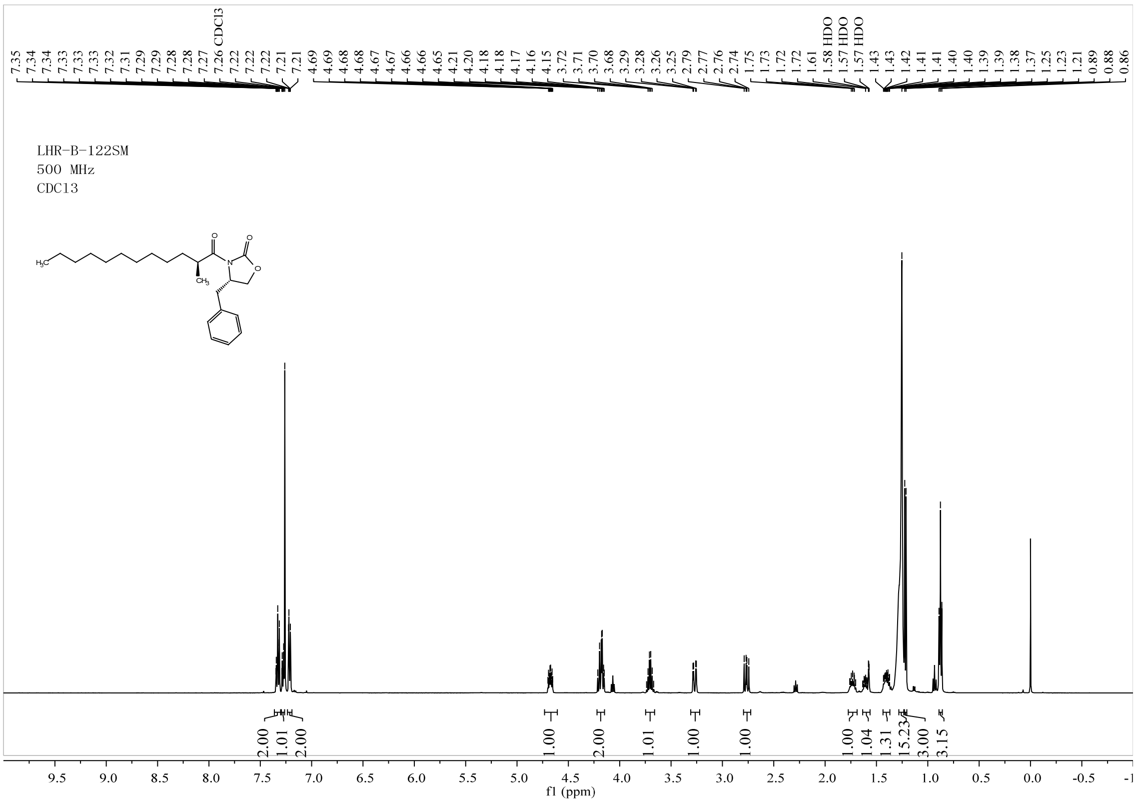


Figure S-2. ^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C NMR (126 MHz, $CDCl_3$) spectra of **2b.**



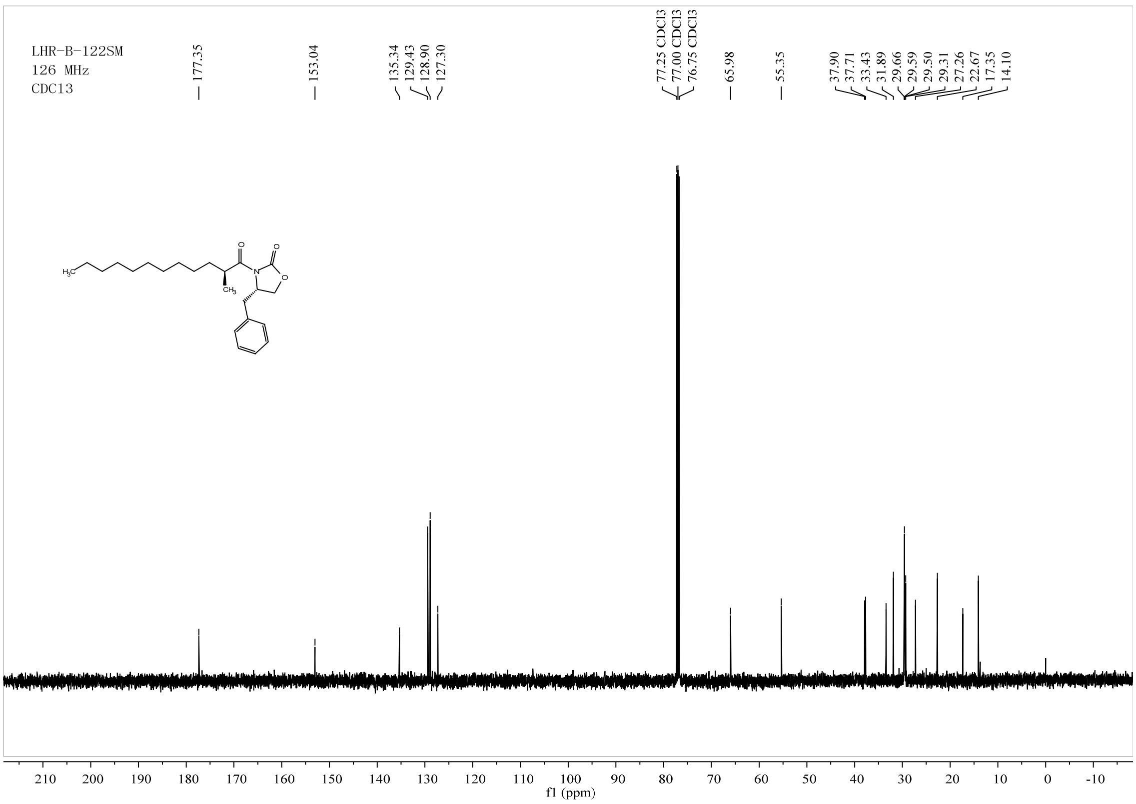
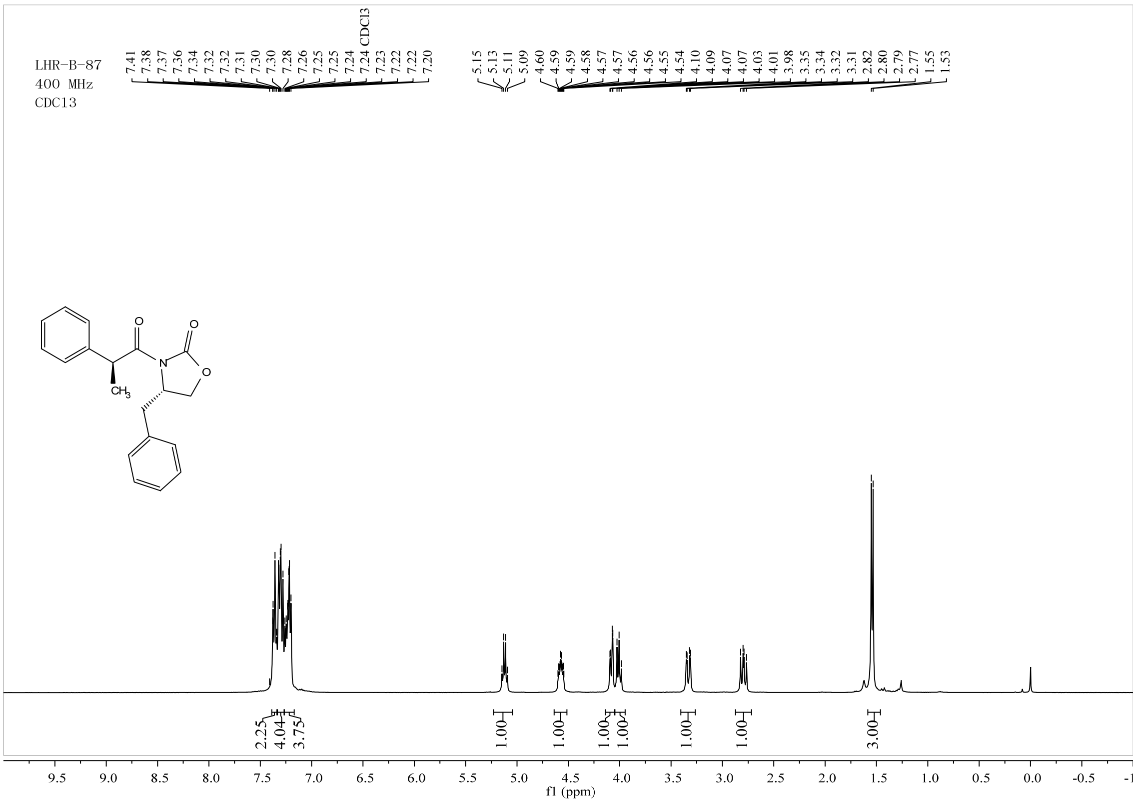


Figure S-3. ^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C NMR (126 MHz, $CDCl_3$) spectra of **2c.**



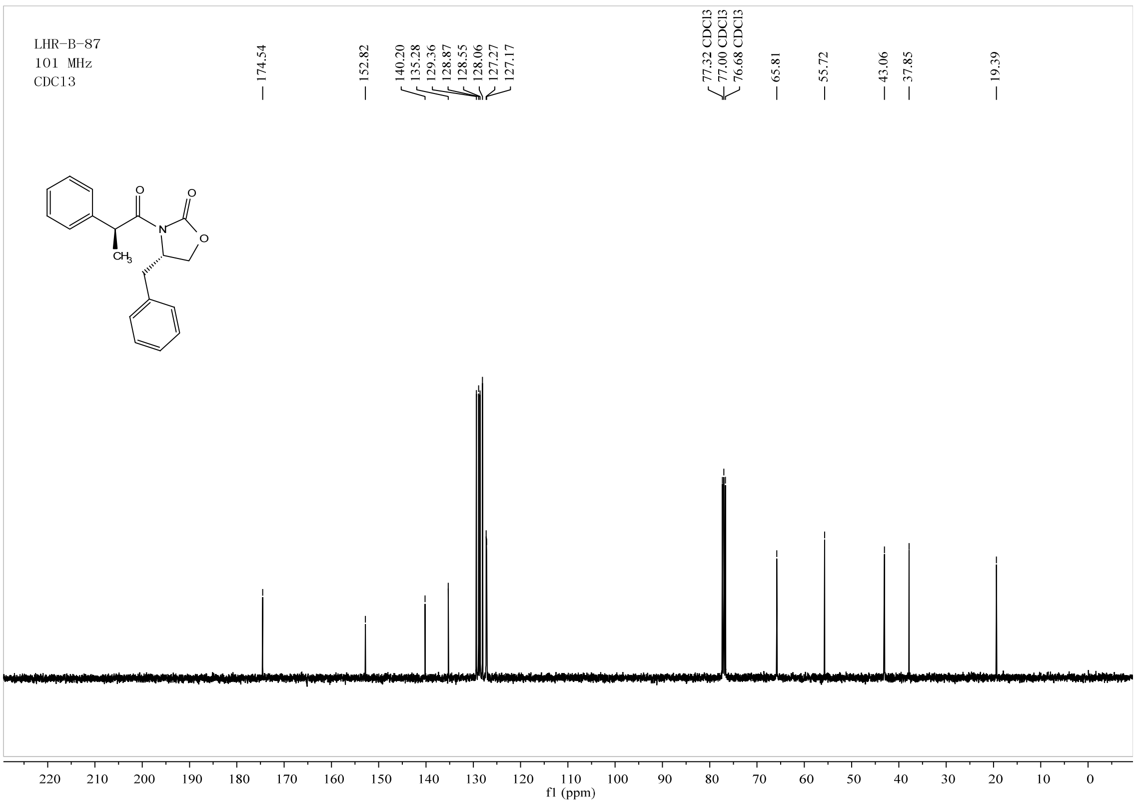
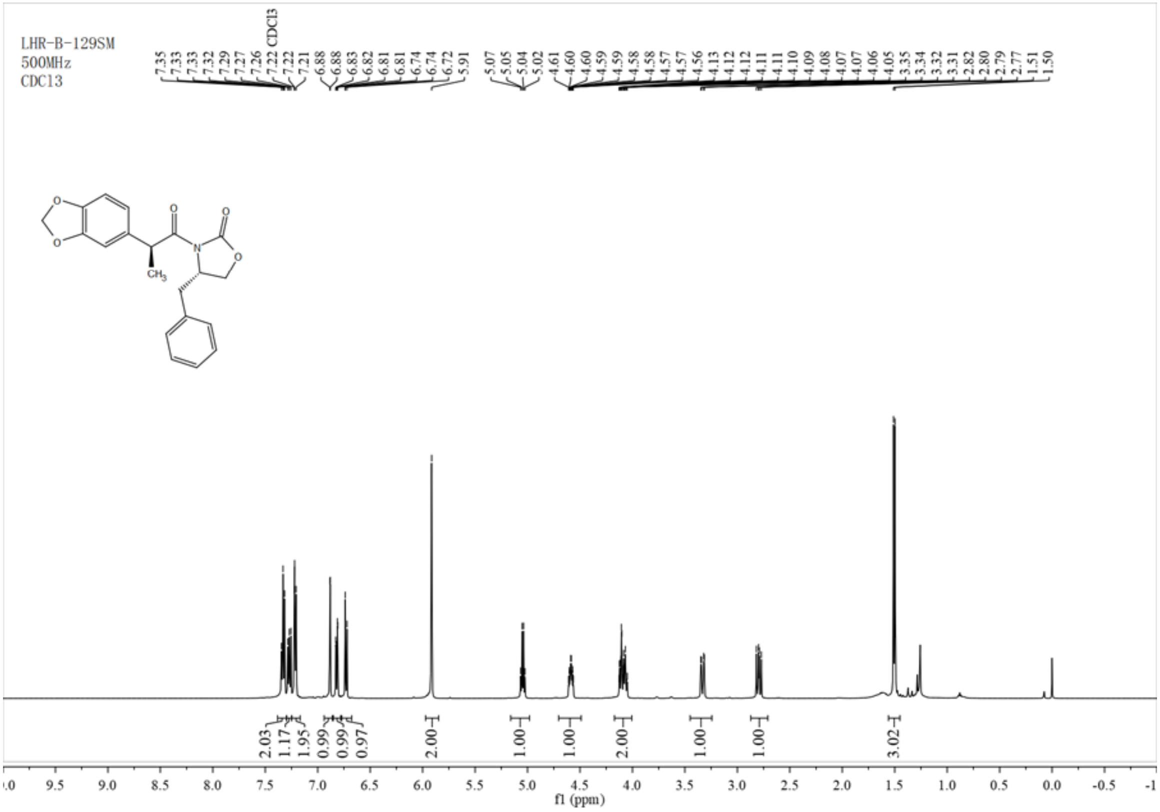


Figure S-4. ^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **2d**.



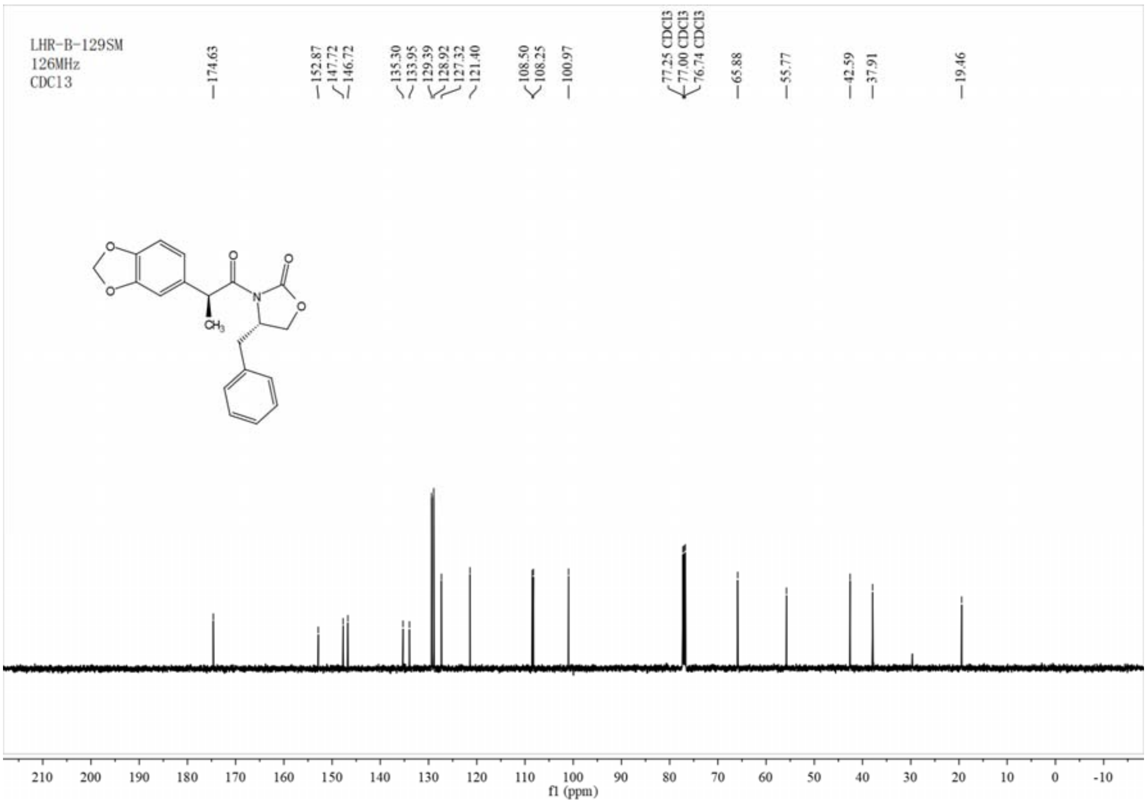
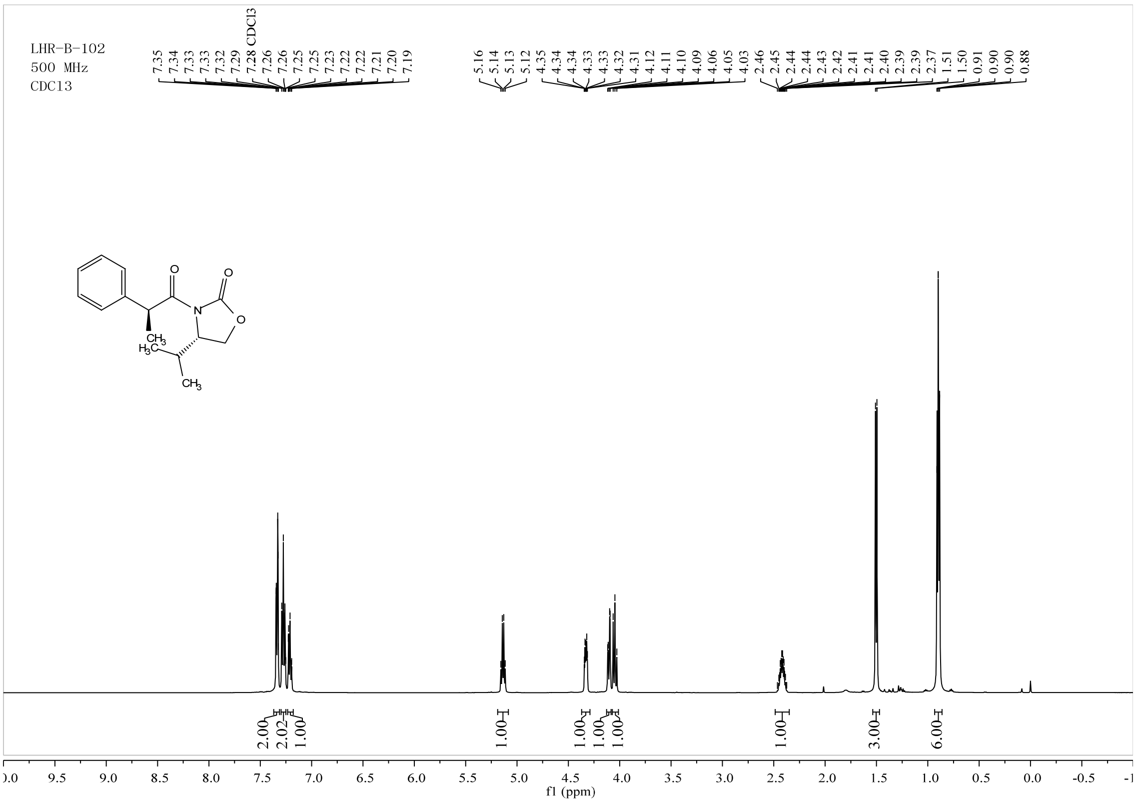


Figure S-5. ^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C NMR (126 MHz, $CDCl_3$) spectra of **2f**.



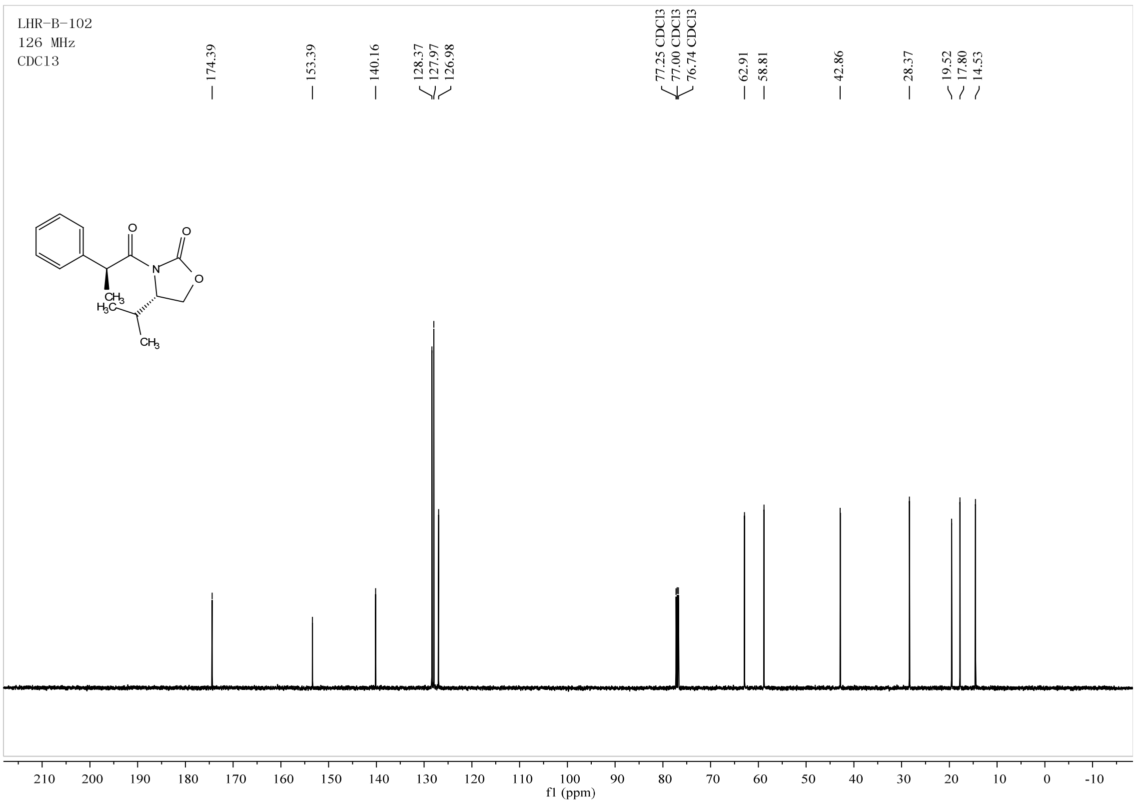
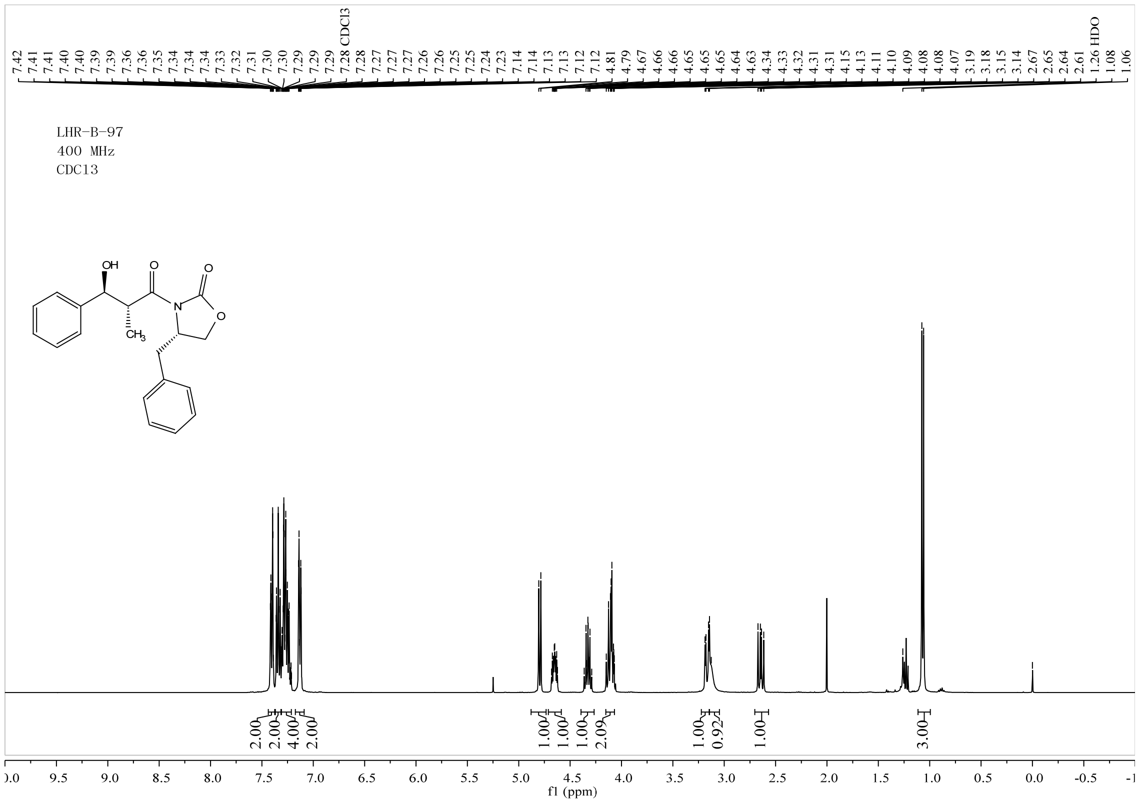


Figure S-6. ^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C NMR (126 MHz, $CDCl_3$) spectra of **2g**.



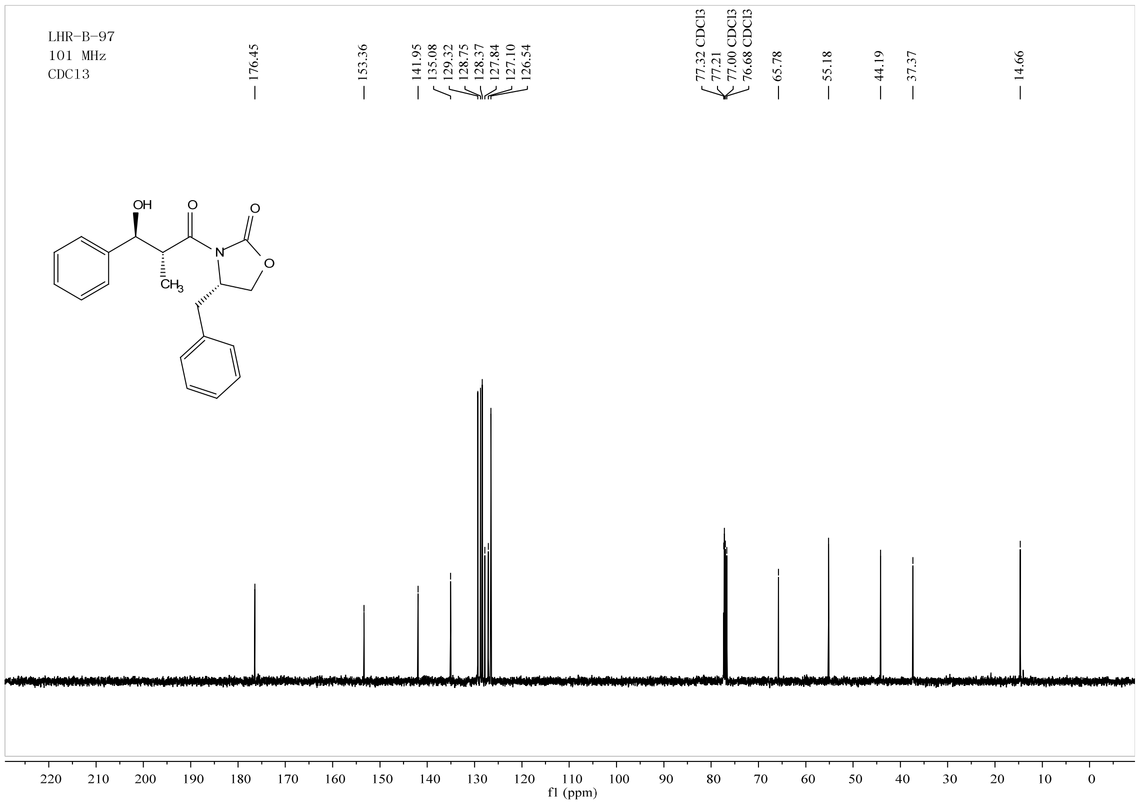
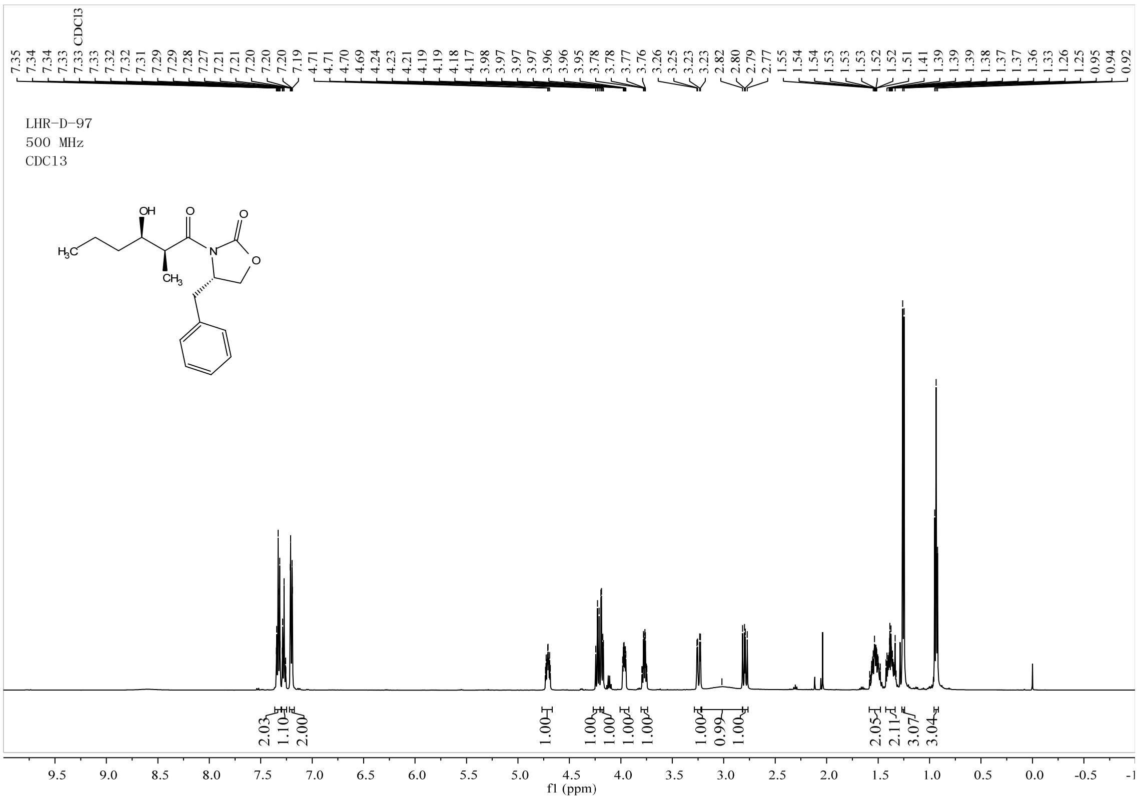


Figure S-7. ^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **2h**.



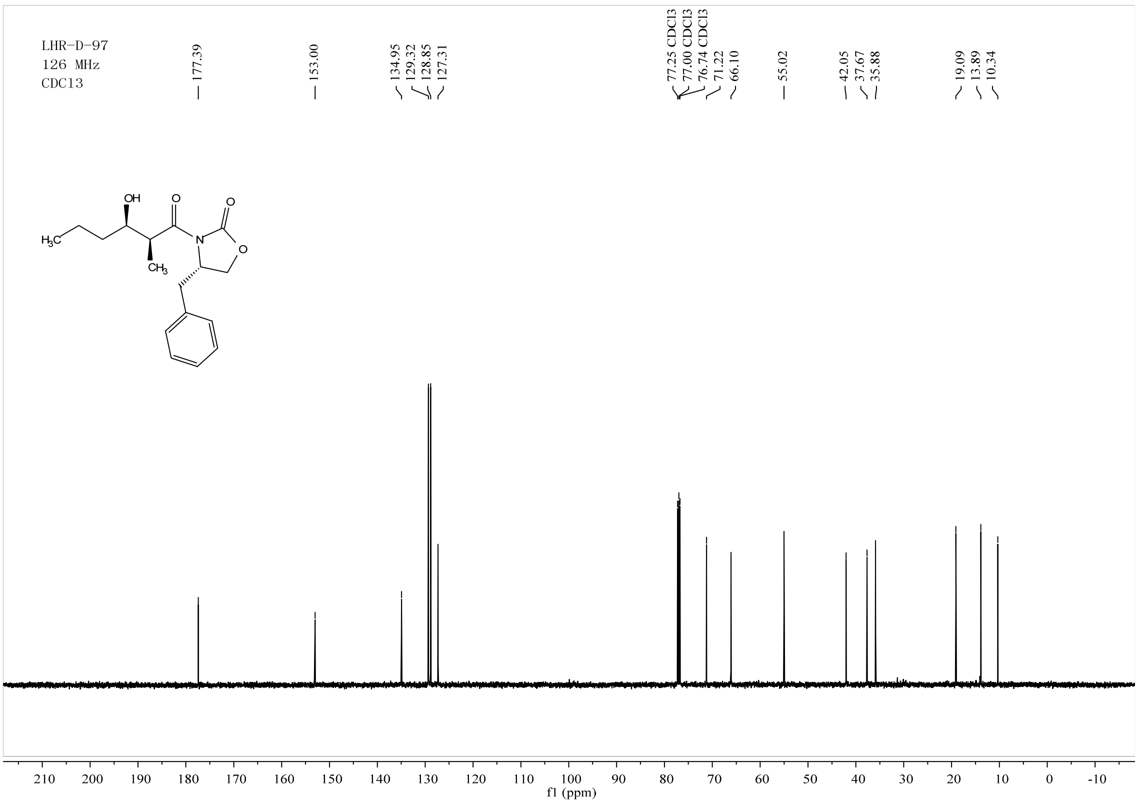
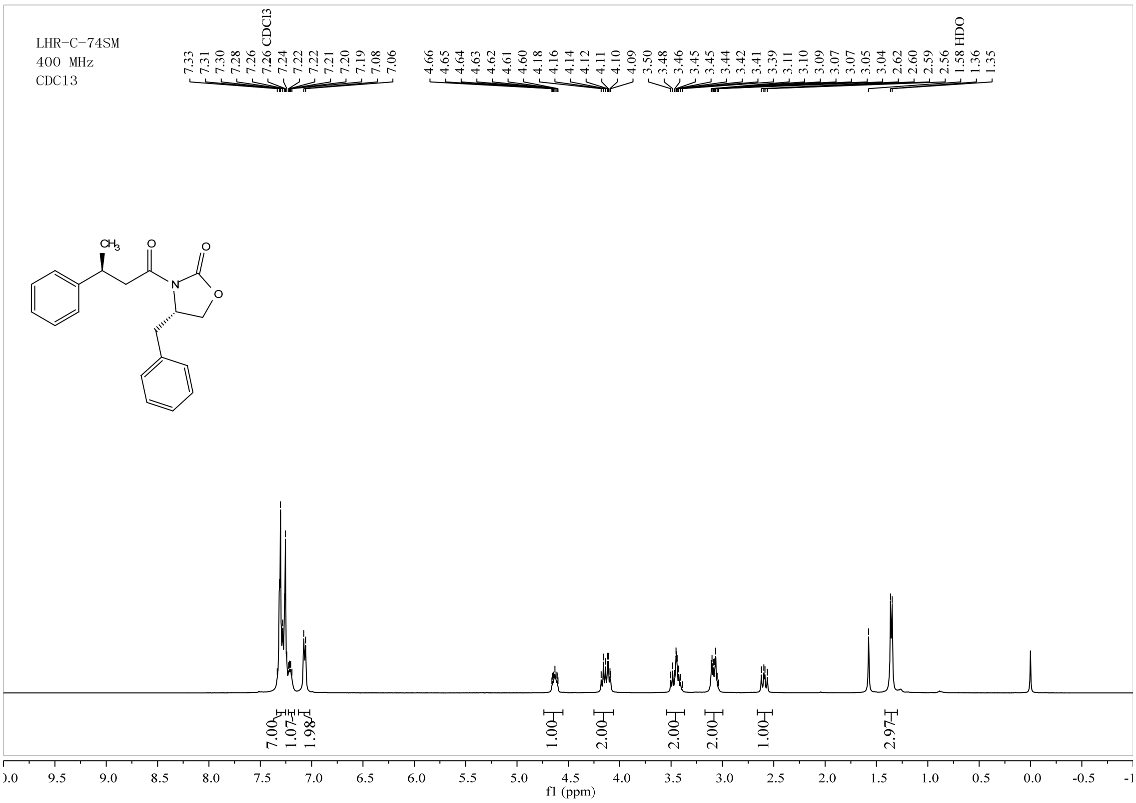


Figure S-8. ^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C NMR (126 MHz, $CDCl_3$) spectra of **2i**.



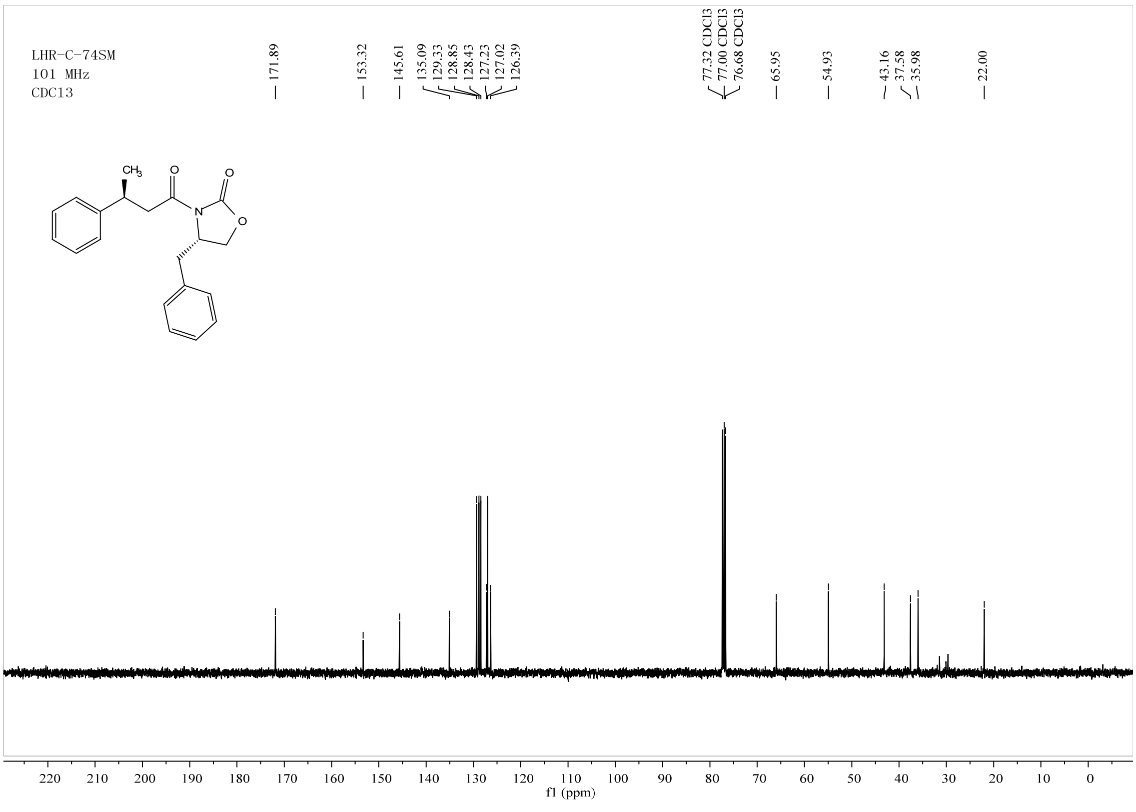
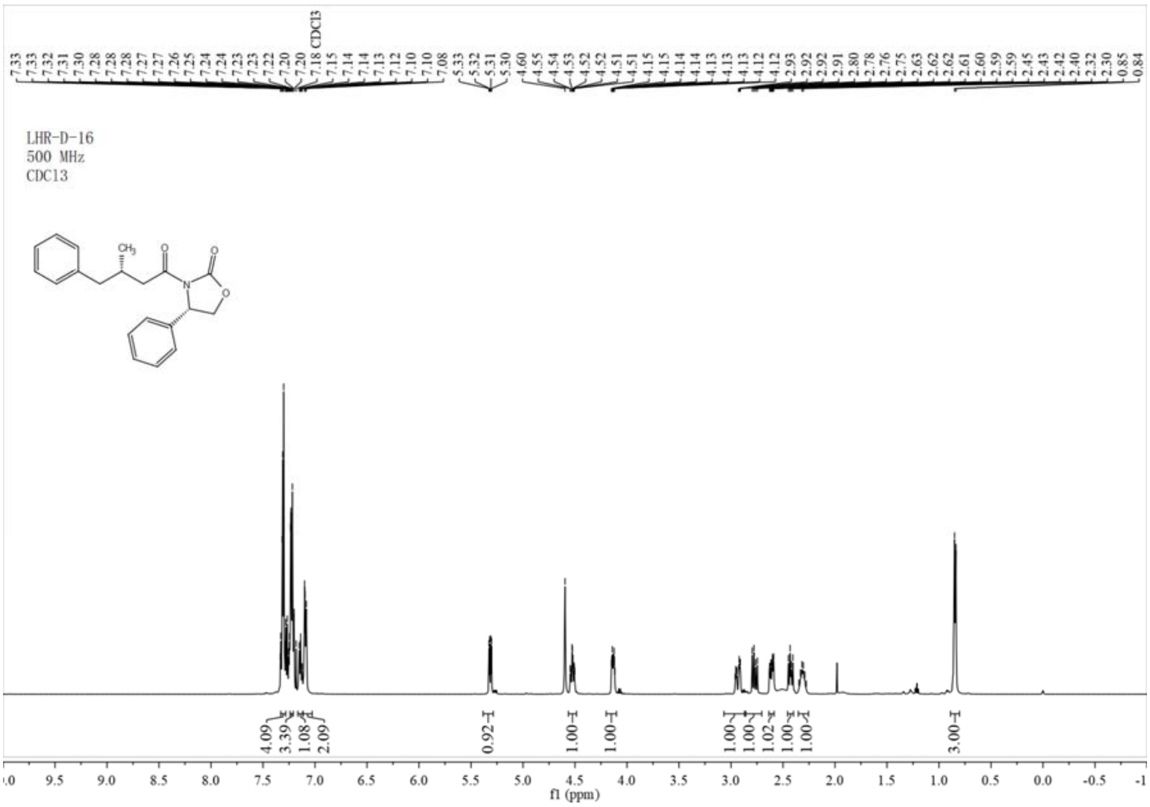


Figure S-9. ^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **2j**.



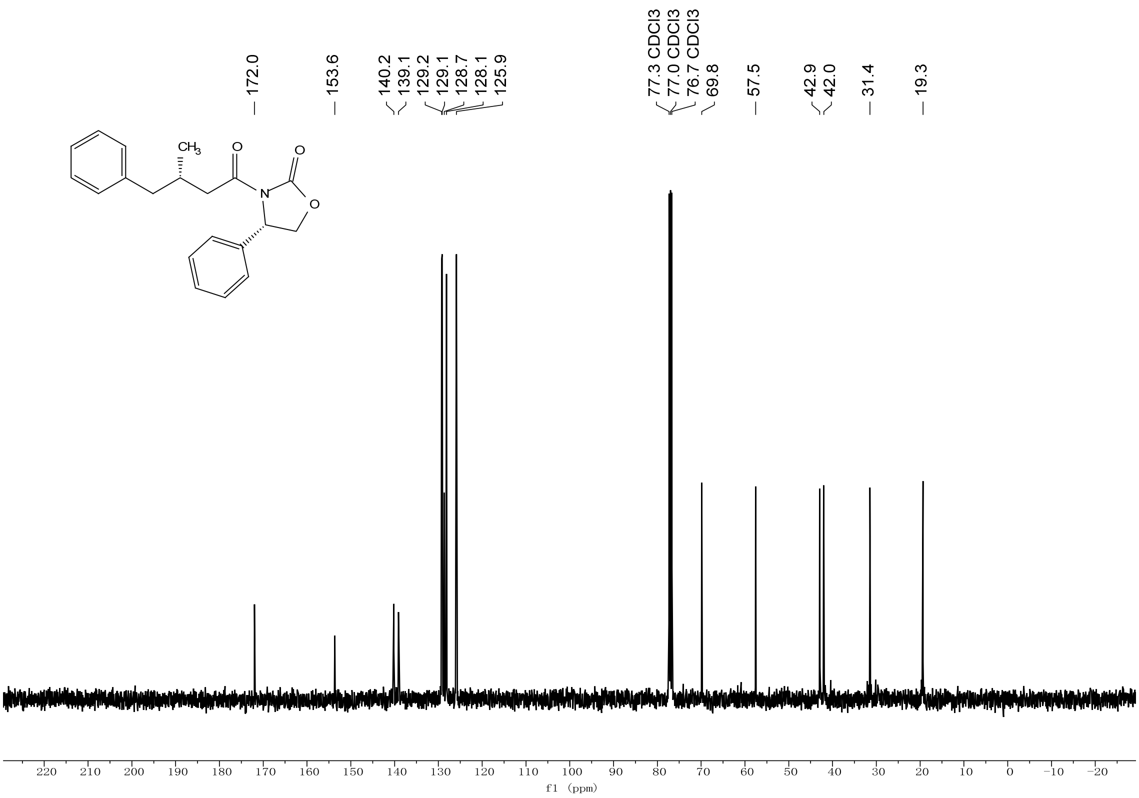
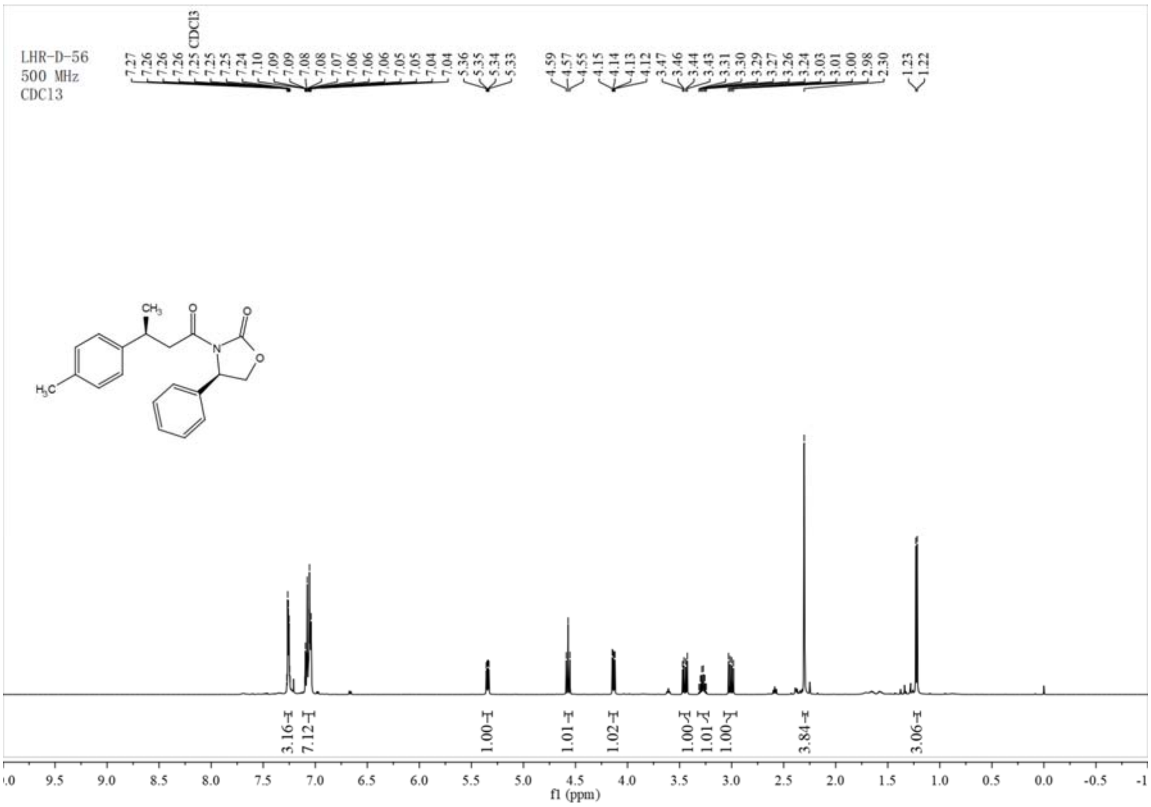


Figure S-10. ^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **2k**.



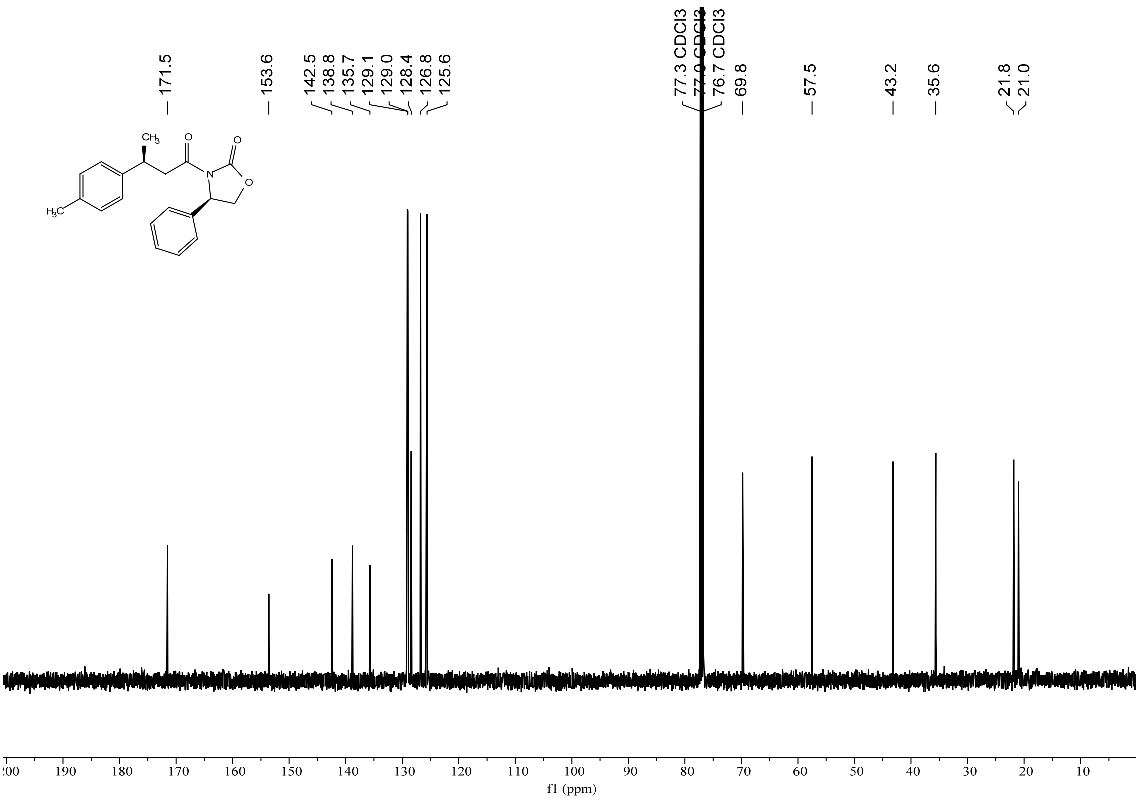
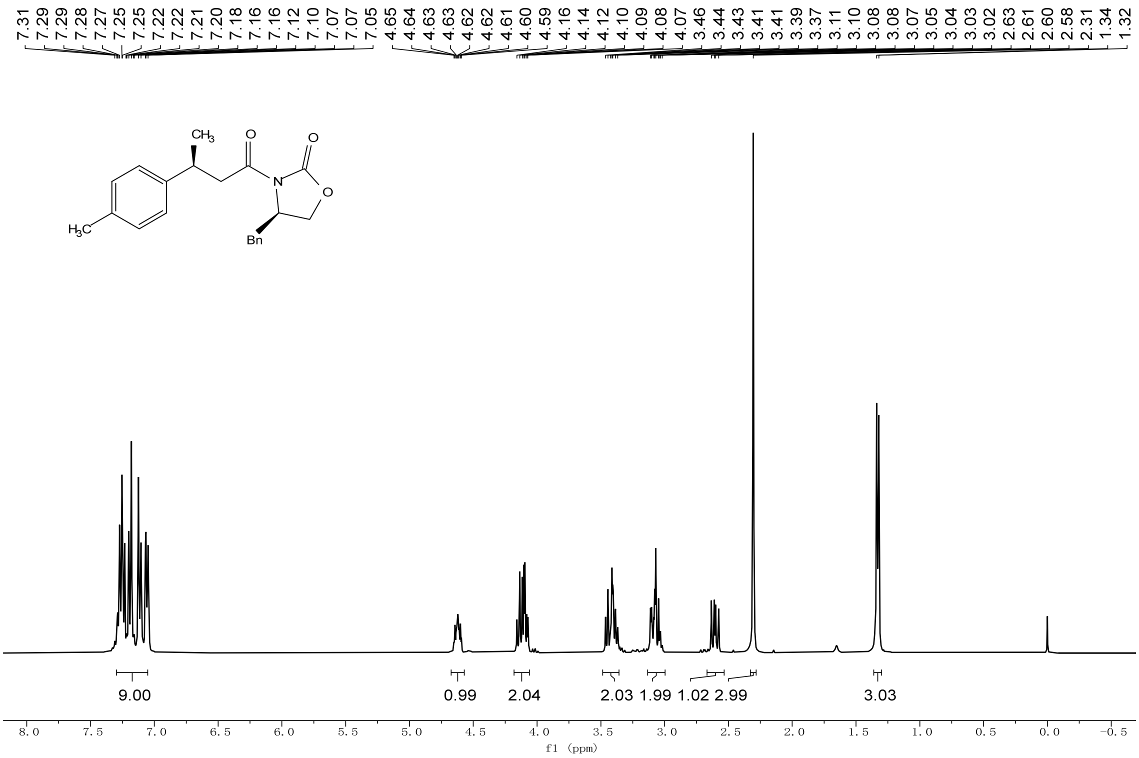


Figure S-11. ^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **2l**.



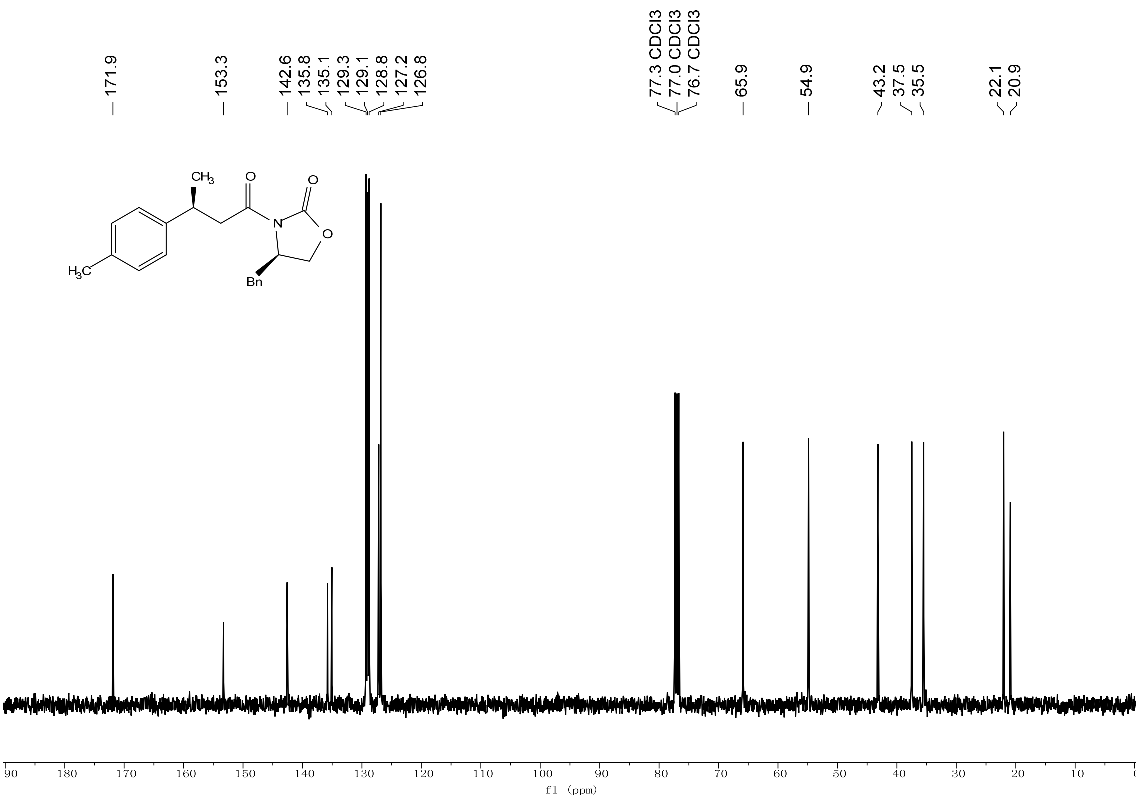
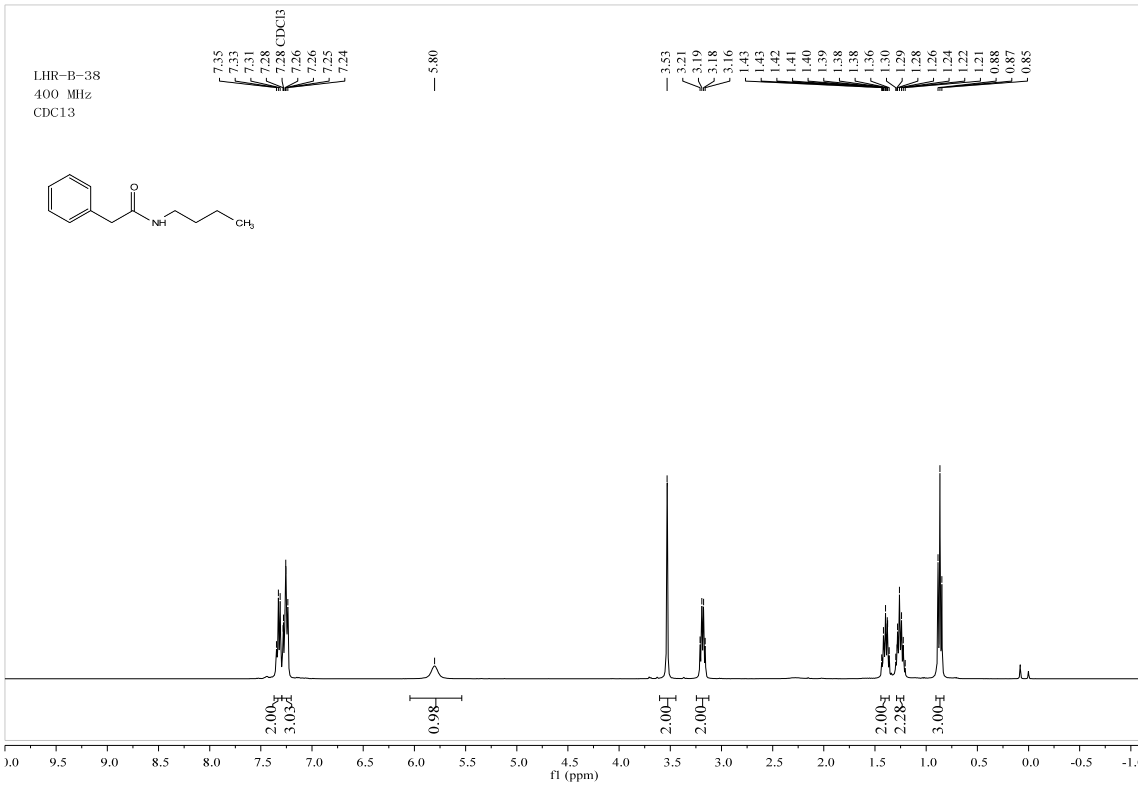


Figure S-12. ^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **2m**.



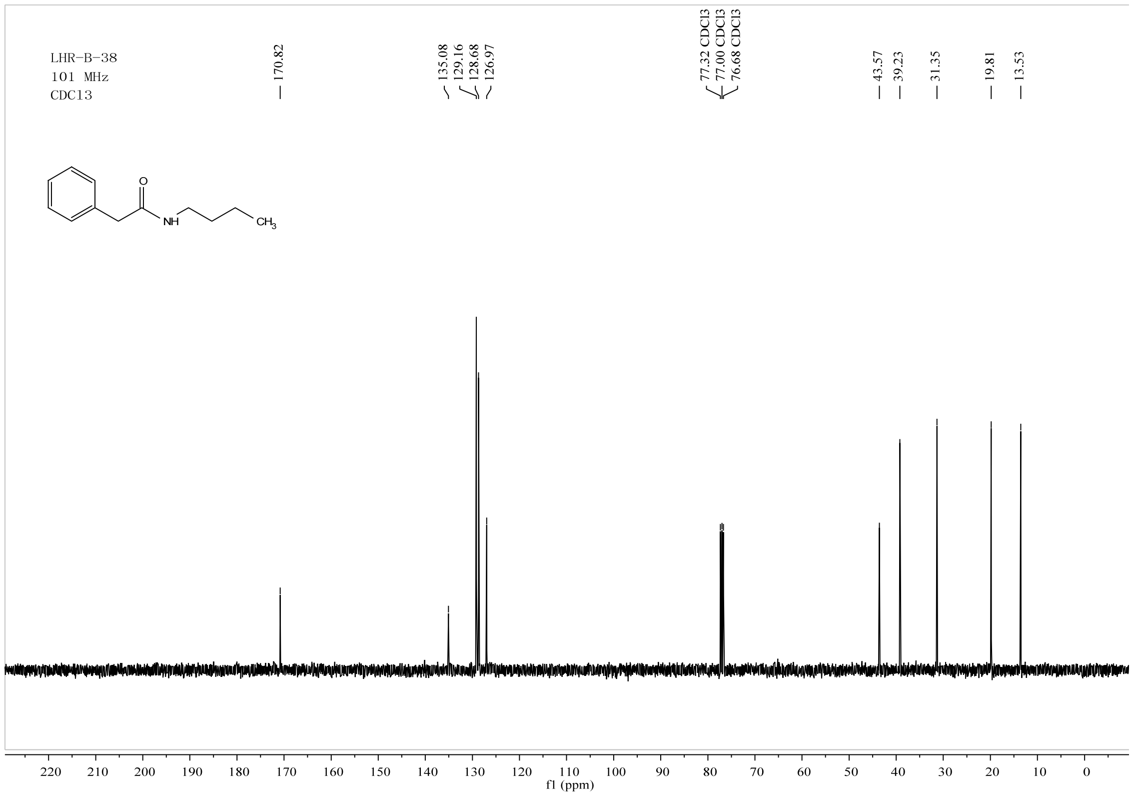
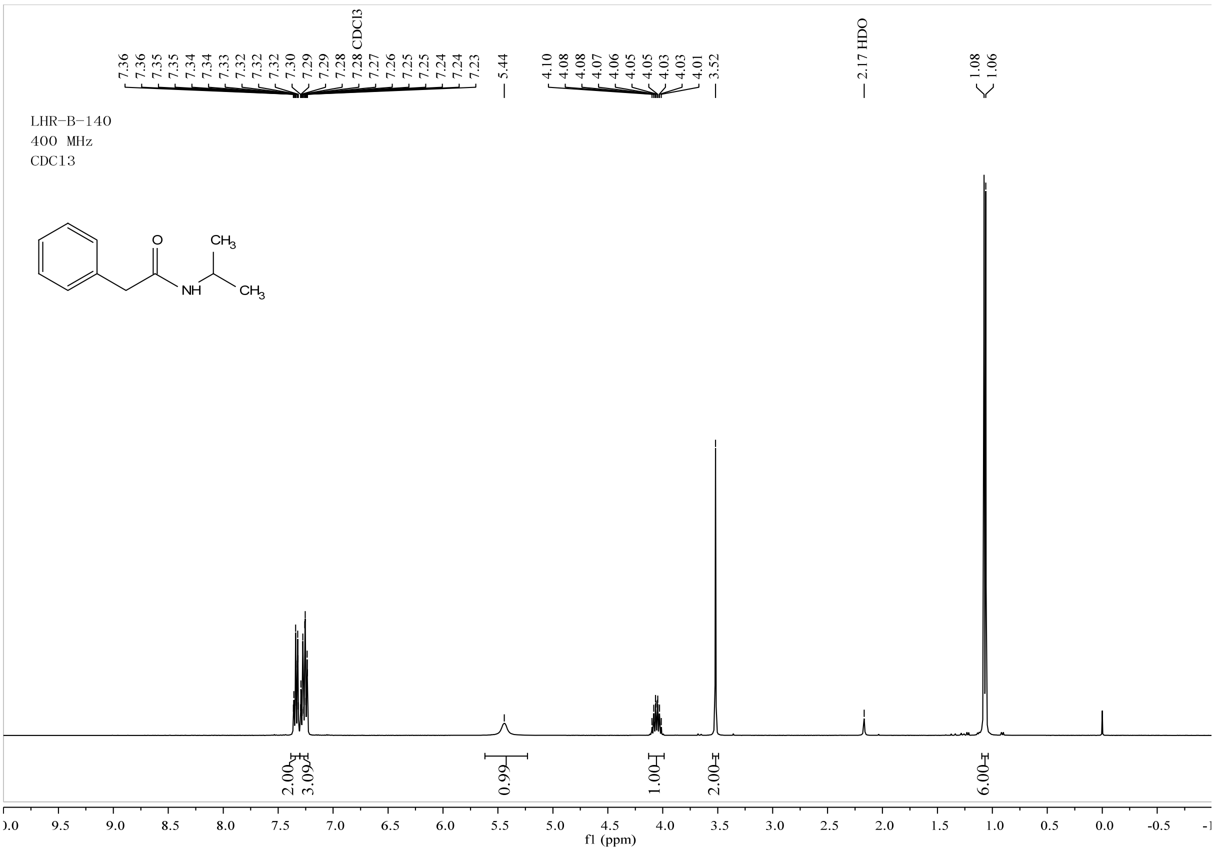


Figure S-13. ^1H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **3A**.



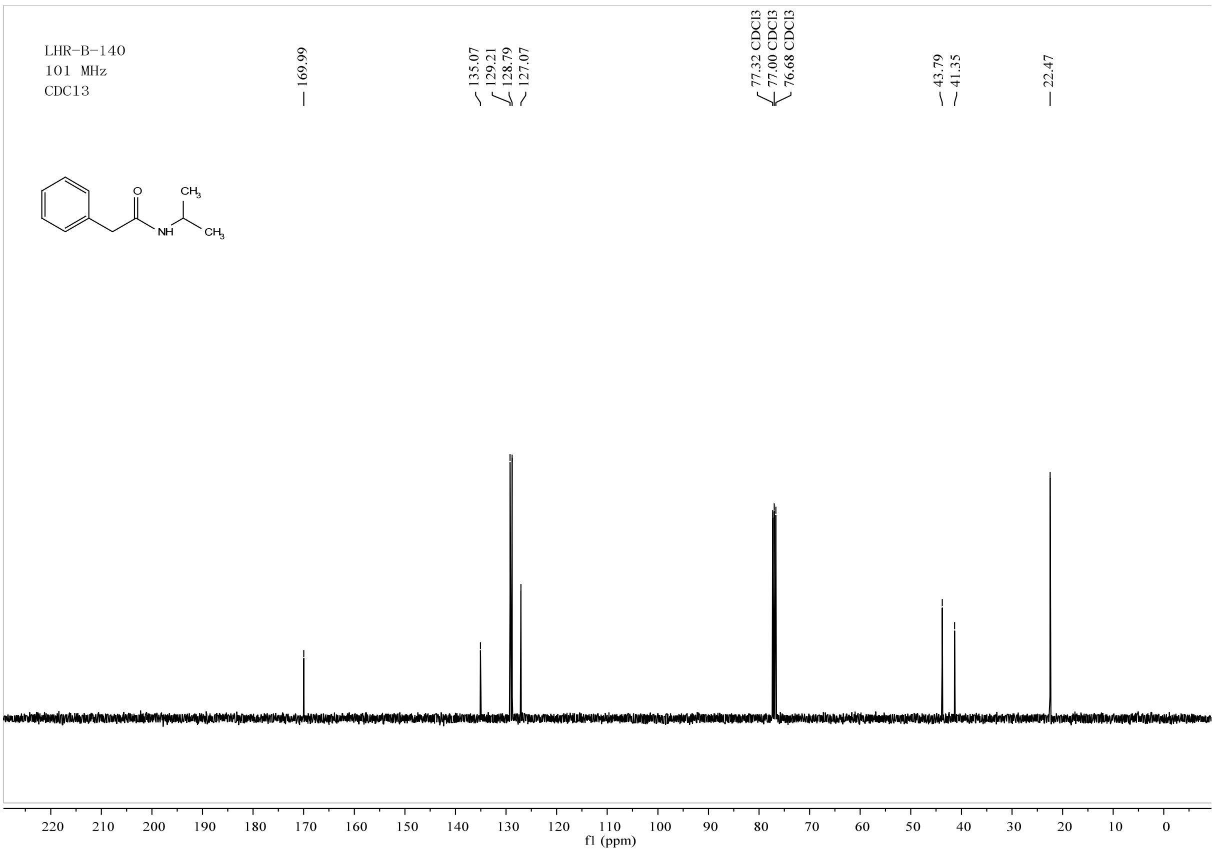
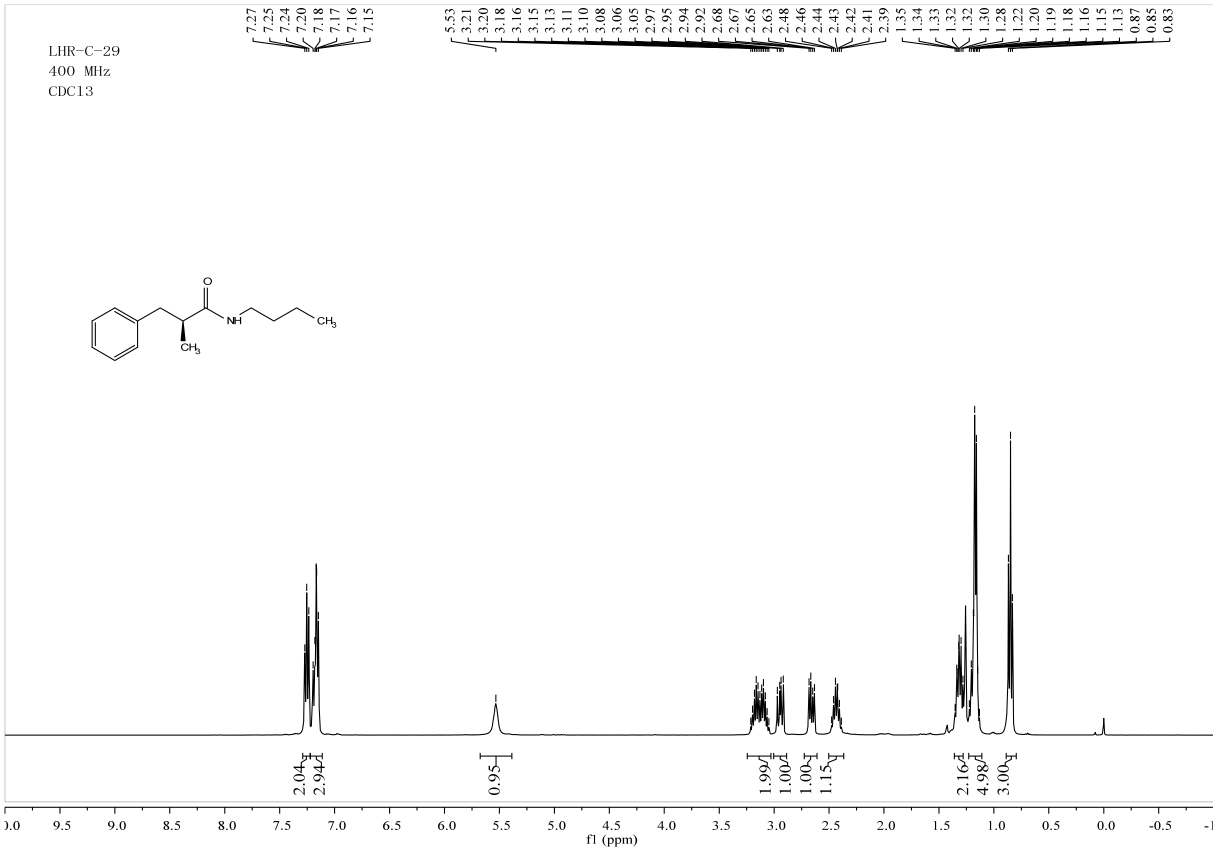


Figure S-14. ^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **3ac**.



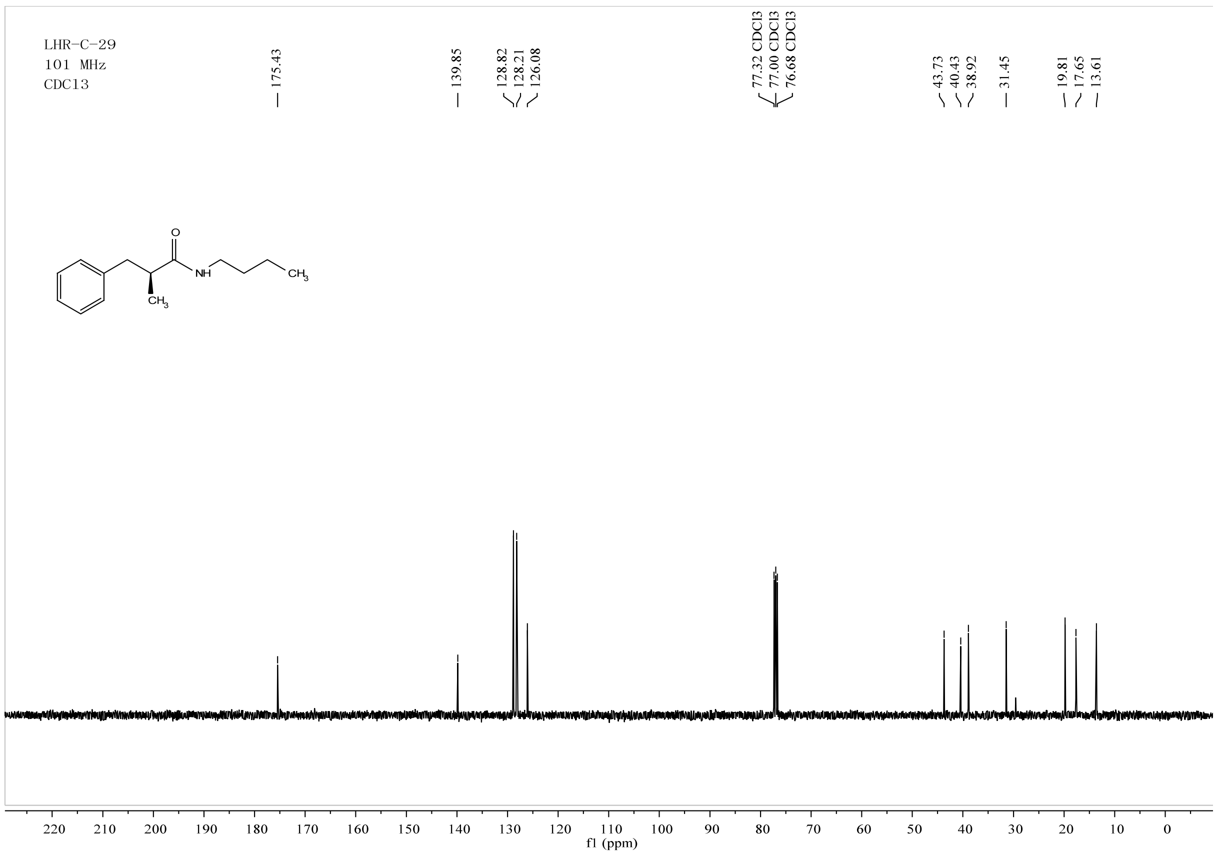
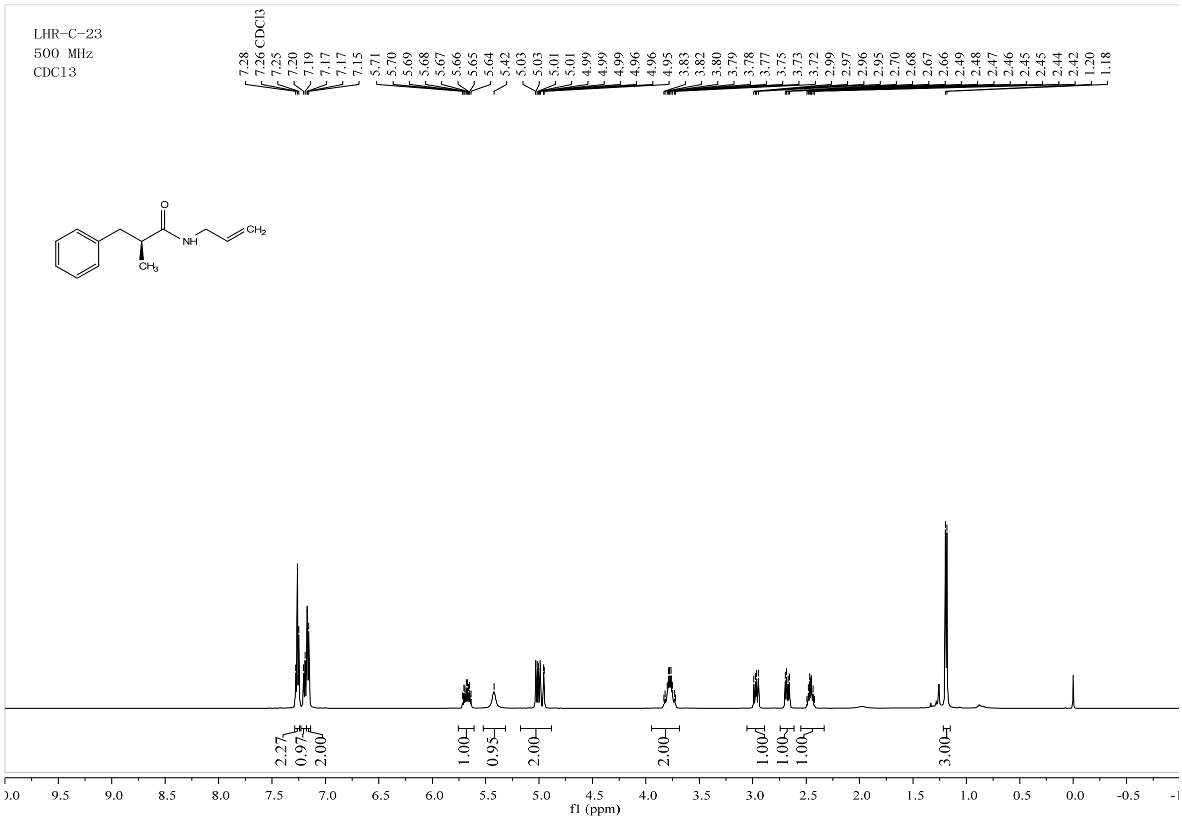


Figure S-15. ^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **3a**.



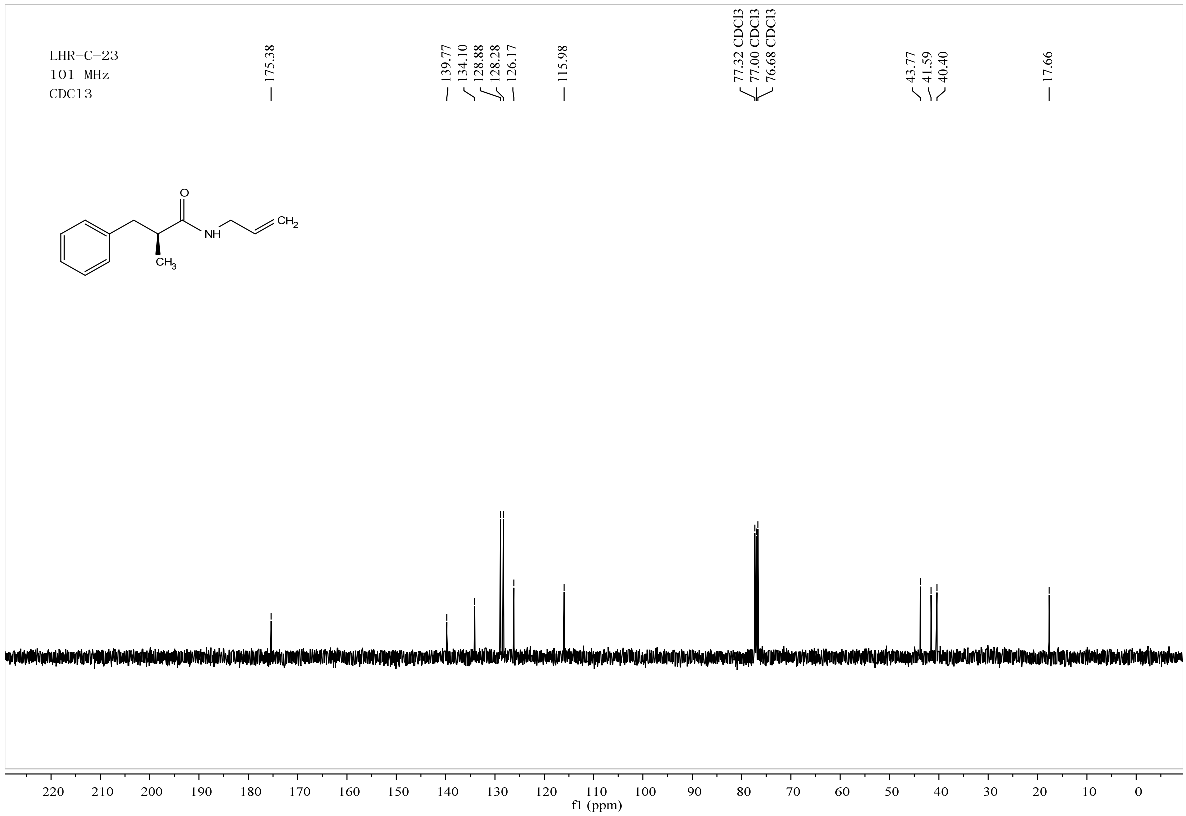
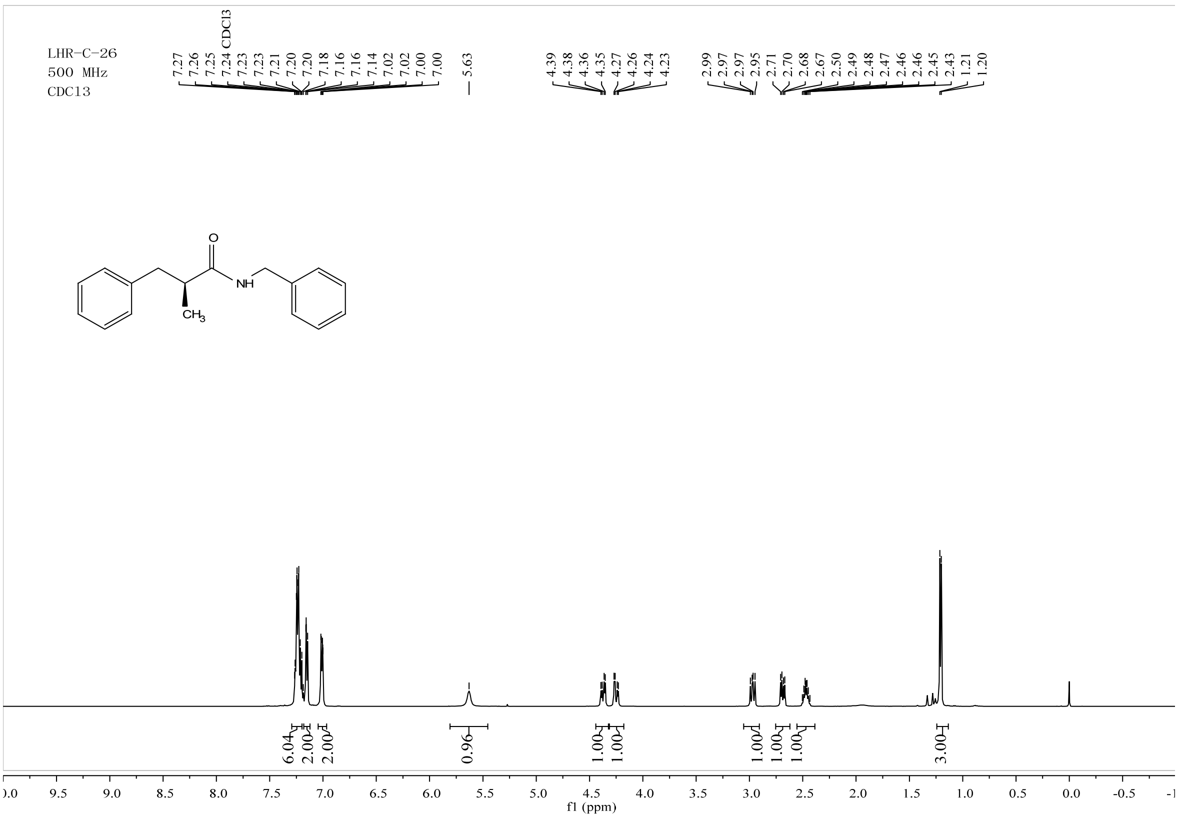


Figure S-16. ^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **3b**.



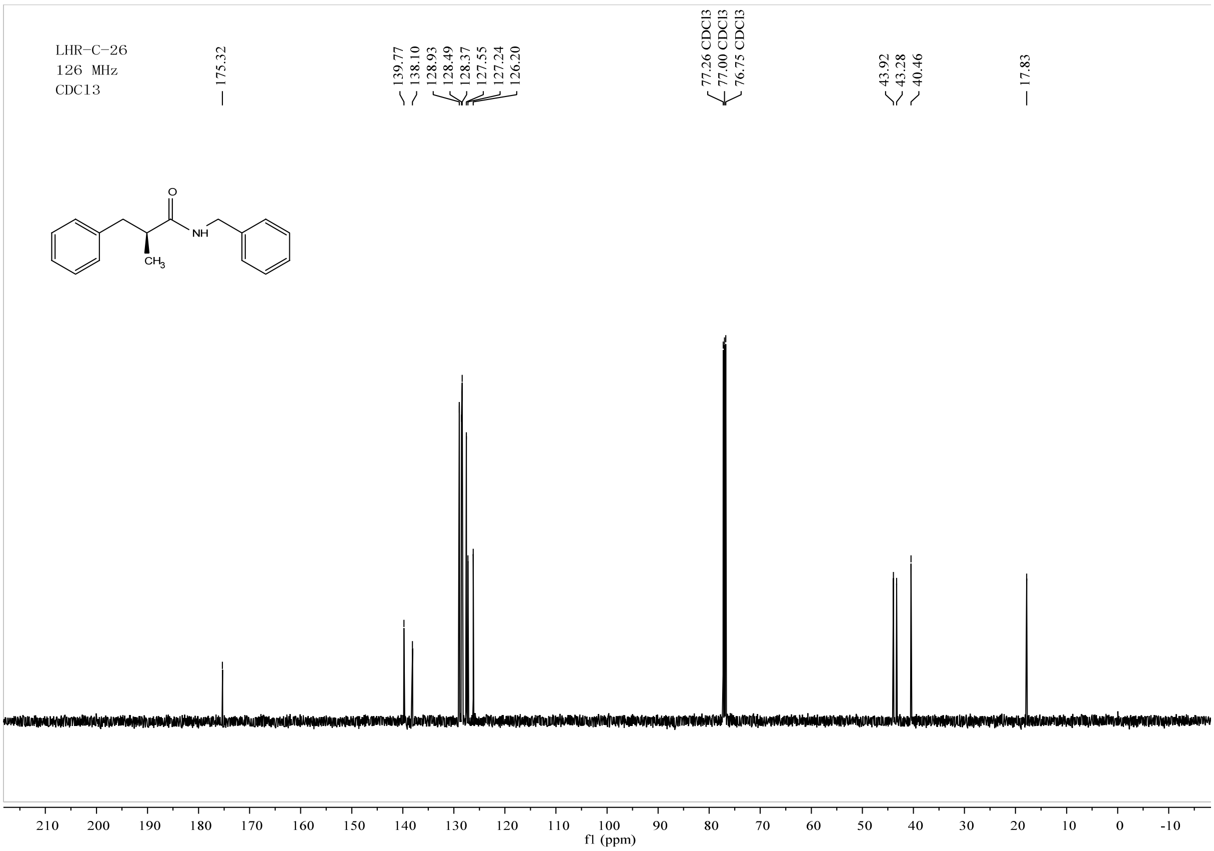
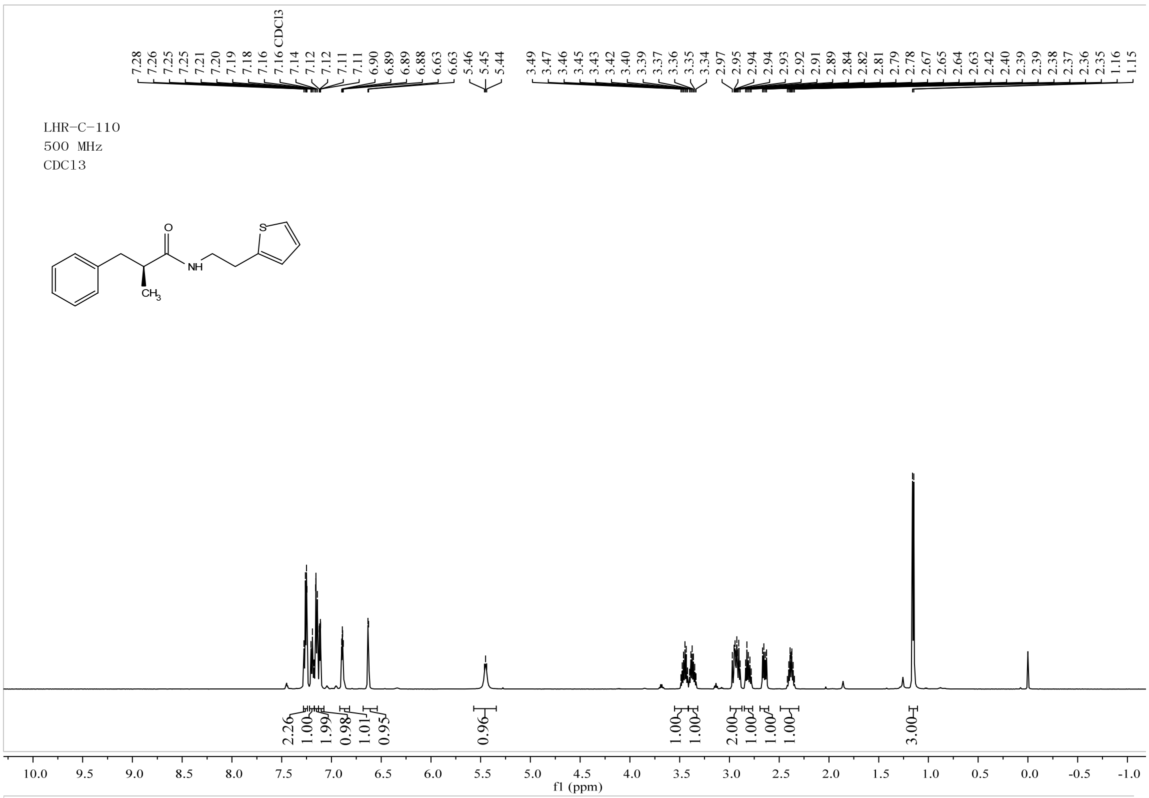


Figure S-17. ^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C NMR (126 MHz, $CDCl_3$) spectra of **3c**.



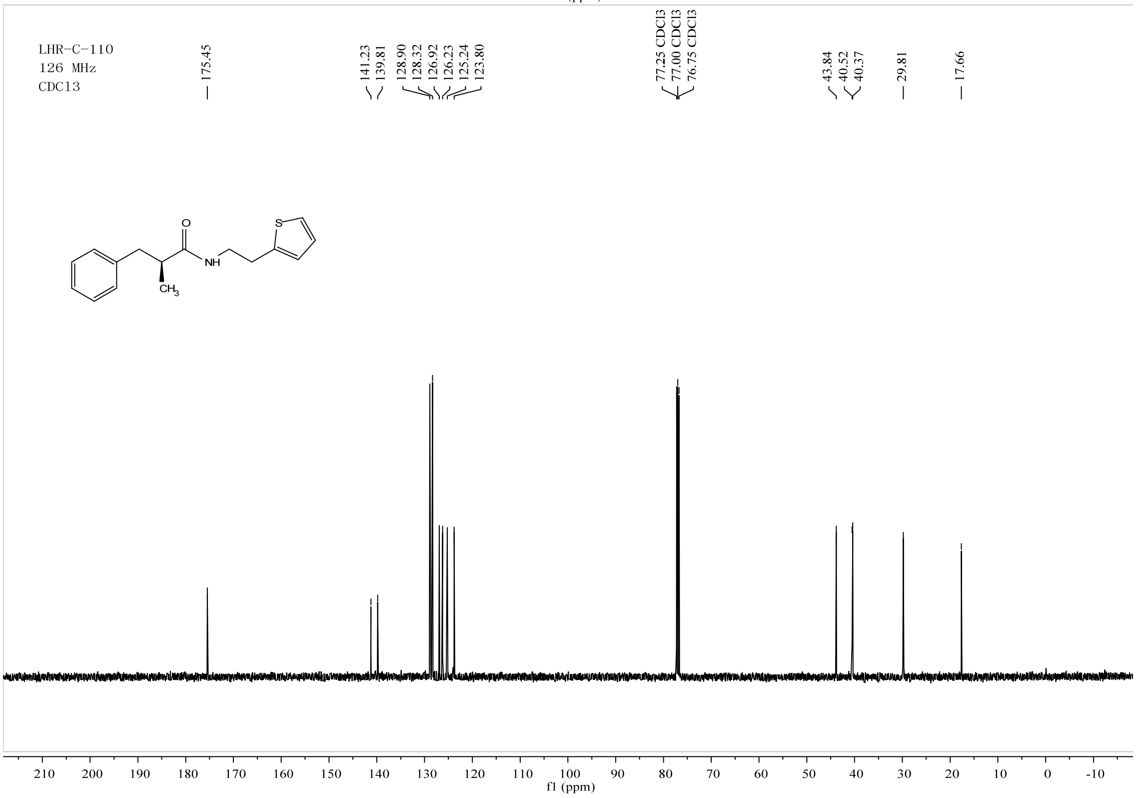
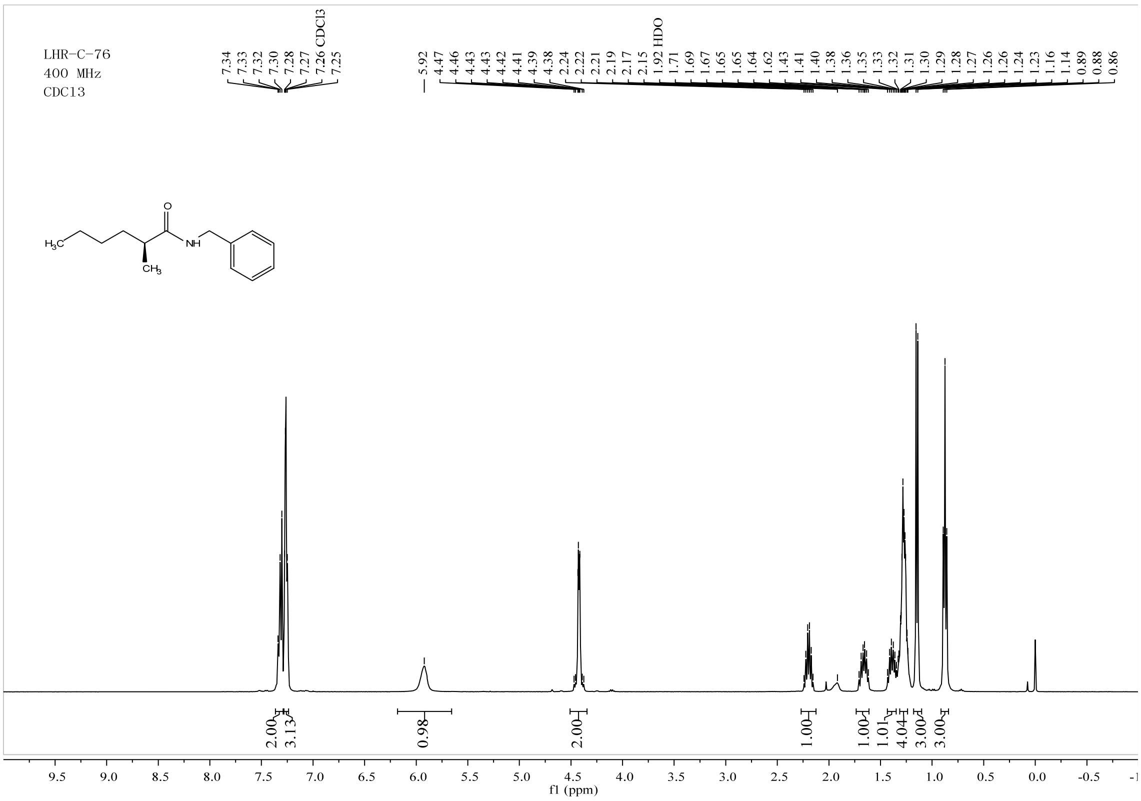


Figure S-18. ^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C NMR (126 MHz, $CDCl_3$) spectra of **3d**.



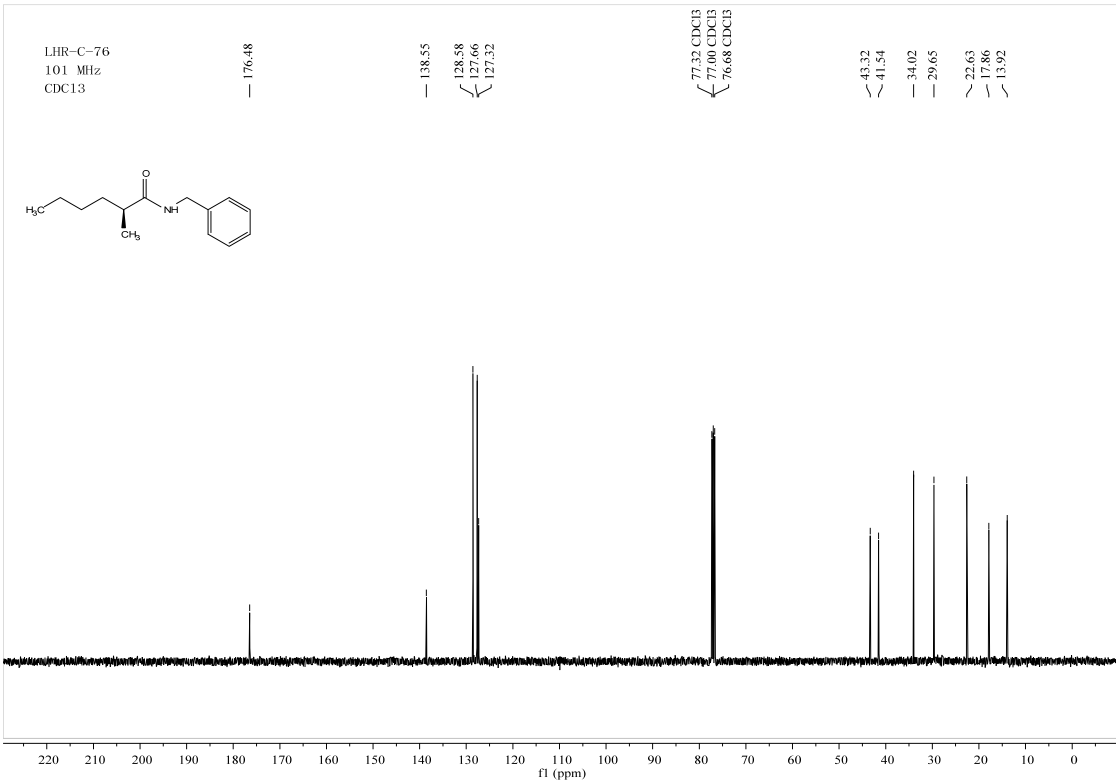
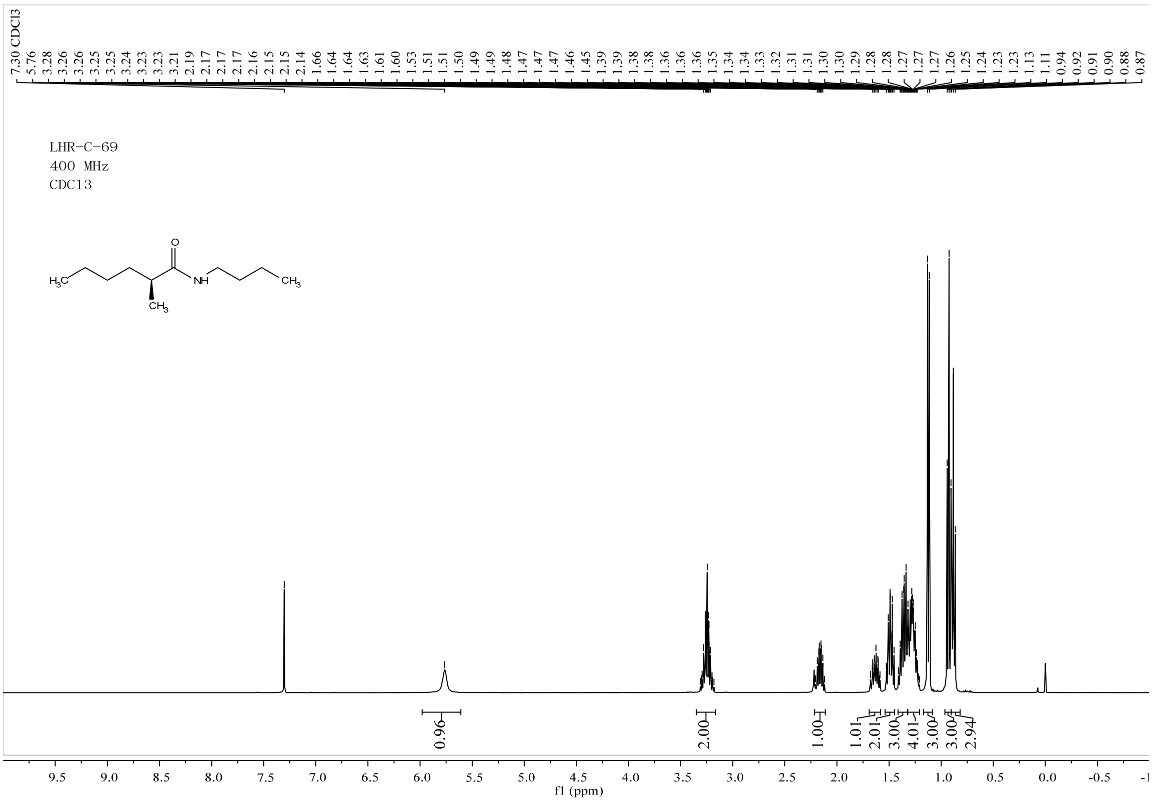


Figure S-19. ^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **3e**.



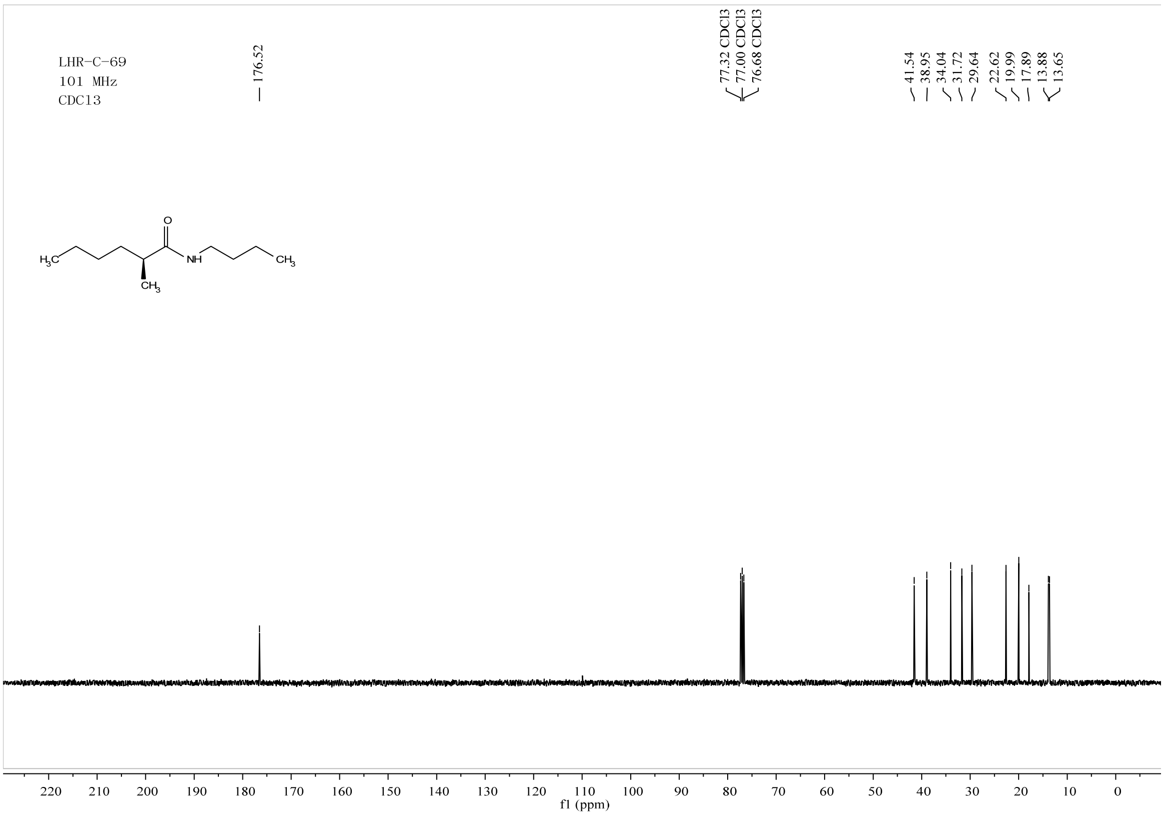
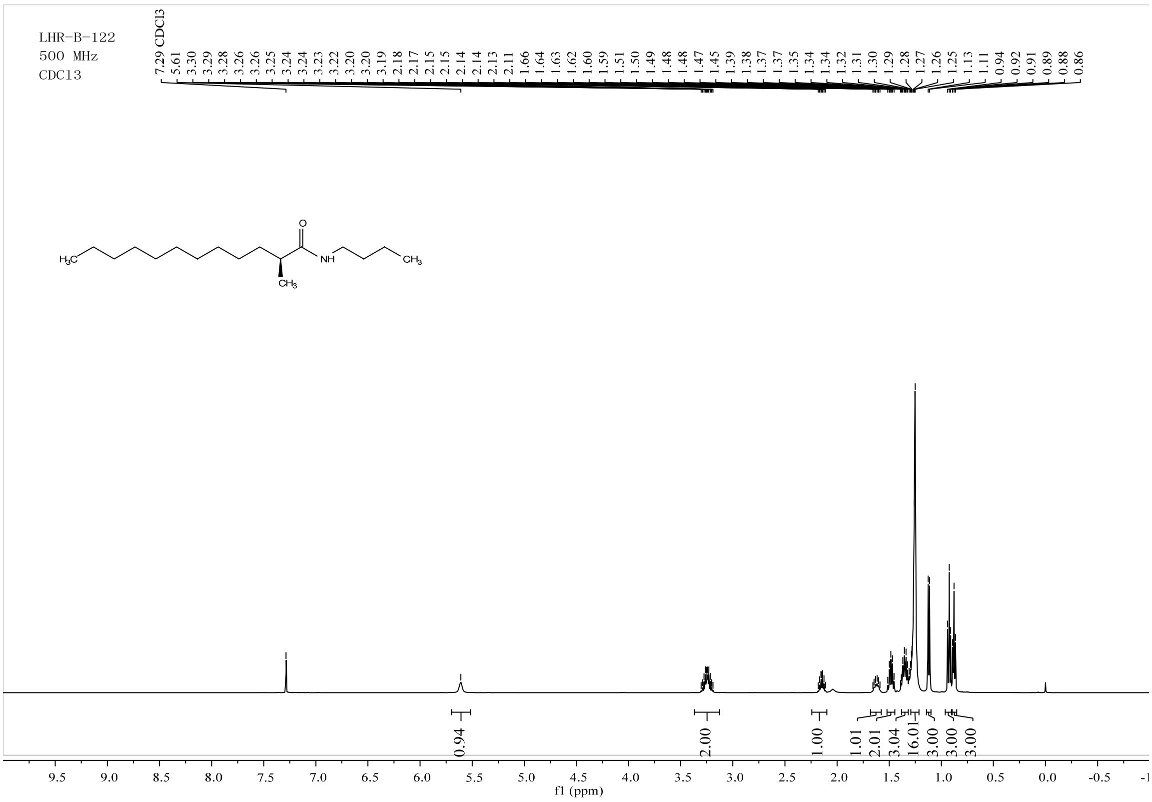


Figure S-20. ^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **3f**.



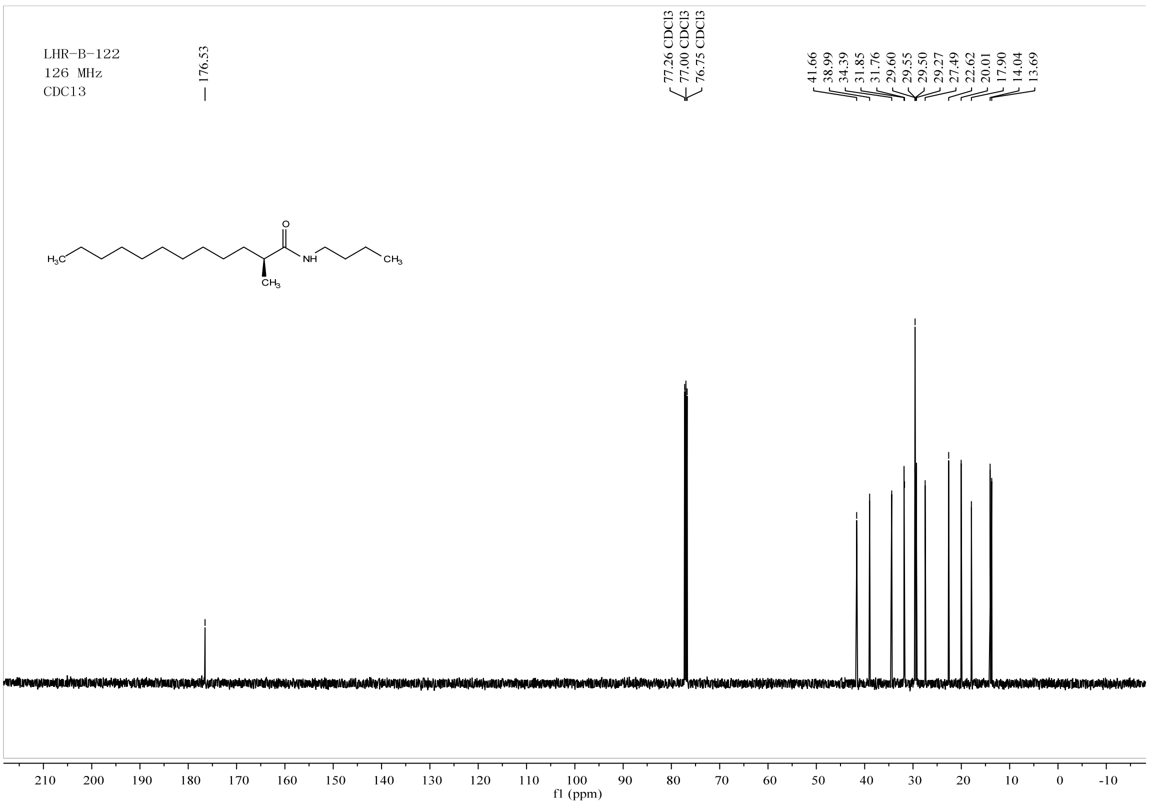
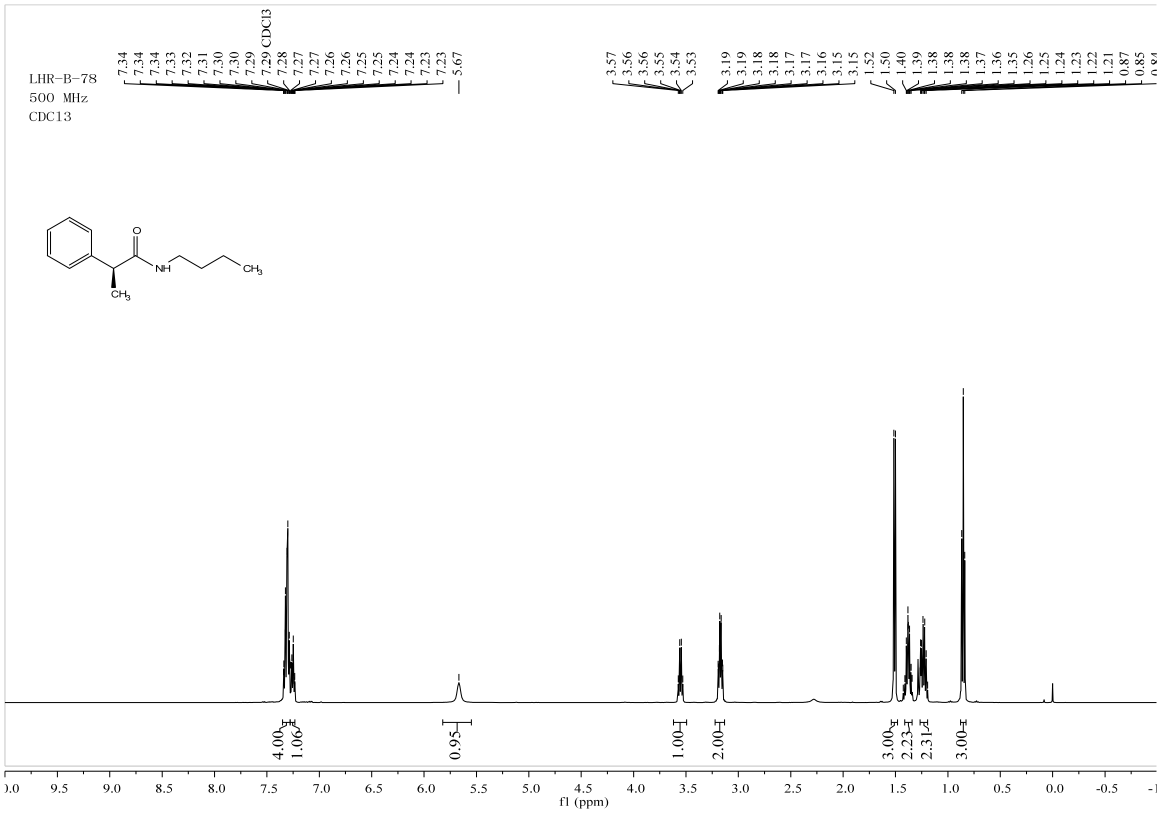


Figure S-21. ^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C NMR (126 MHz, $CDCl_3$) spectra of **3g**.



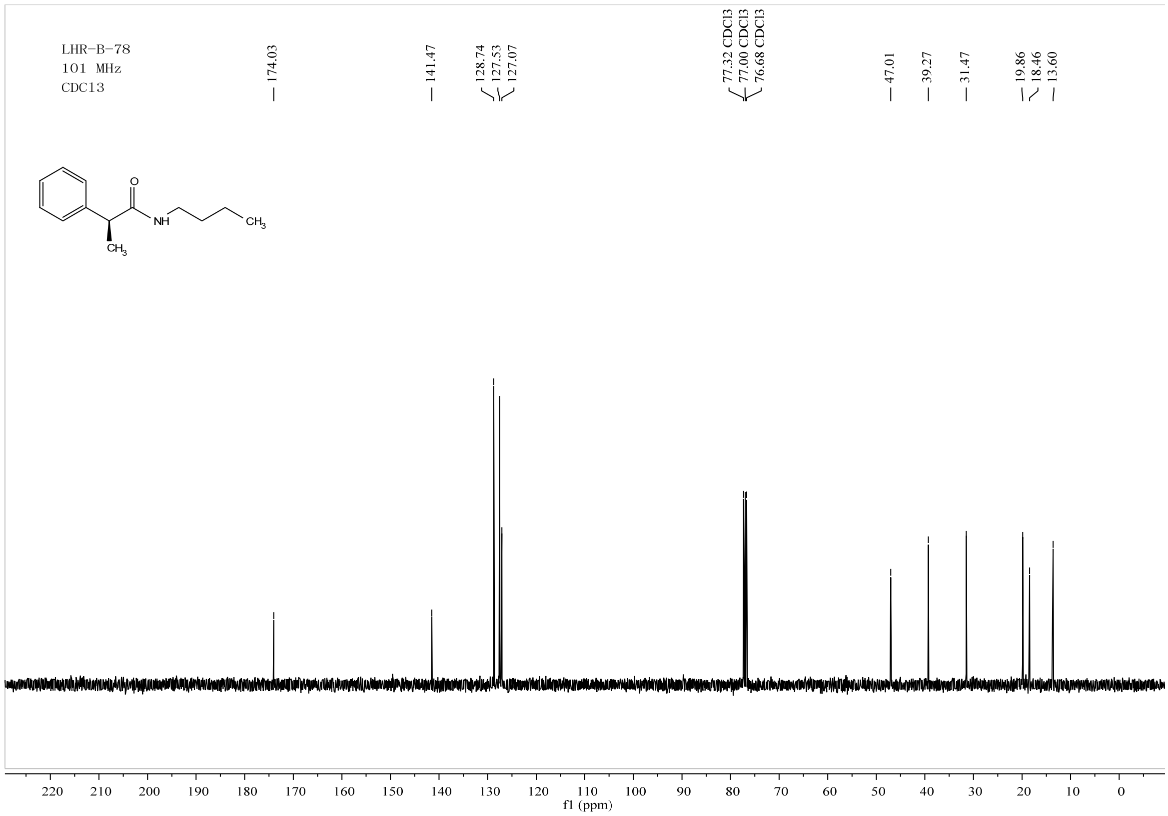
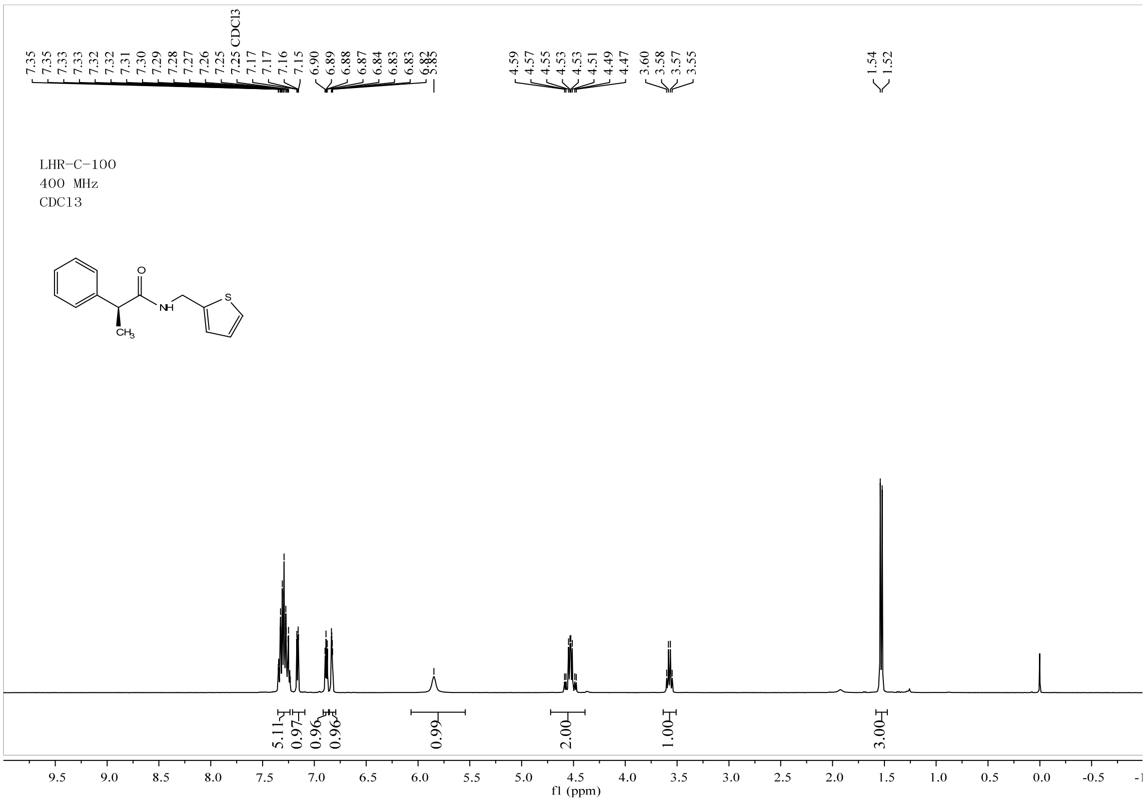


Figure S-22. ^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **3h**.



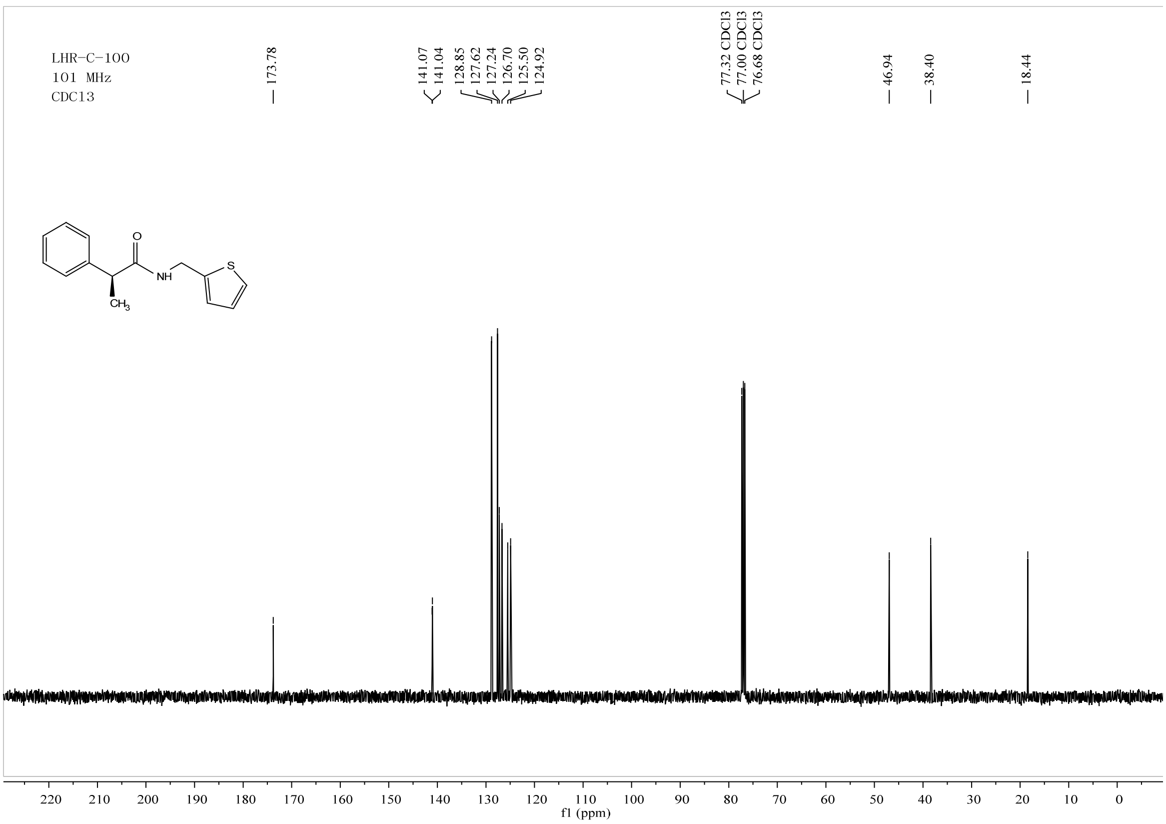
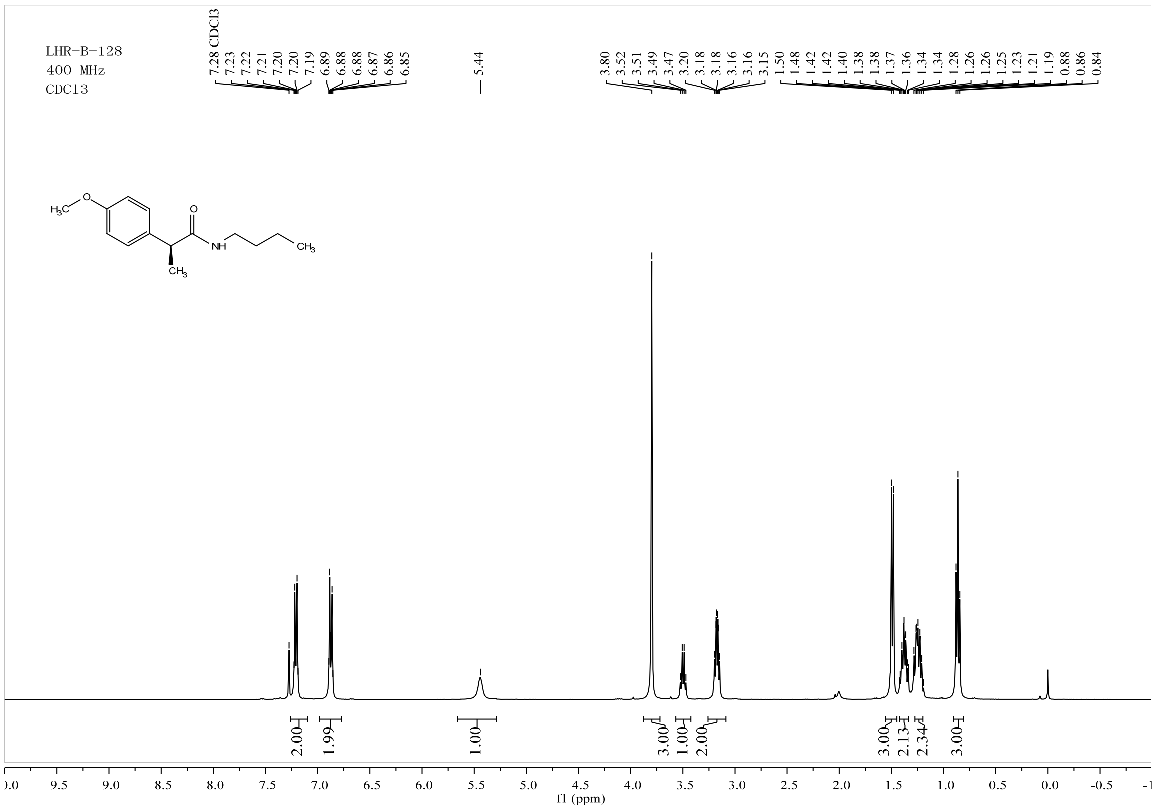


Figure S-23. ^1H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **3i**.



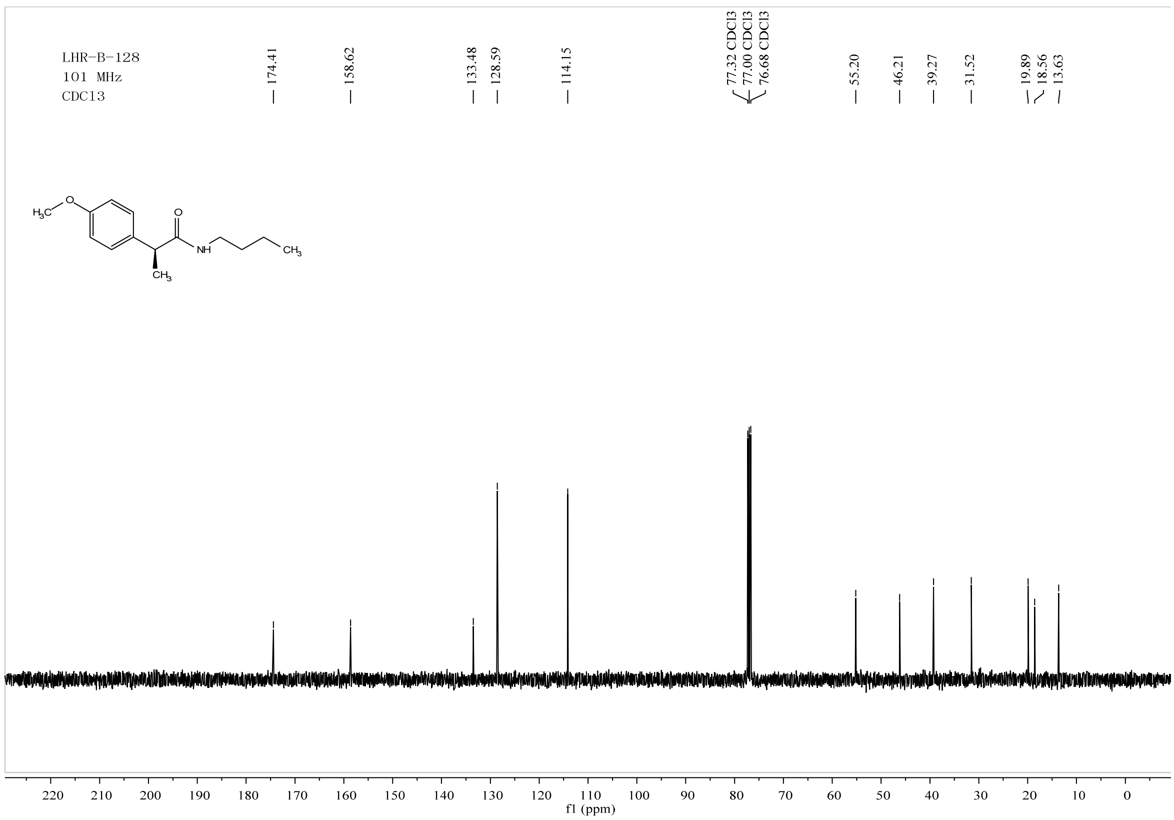
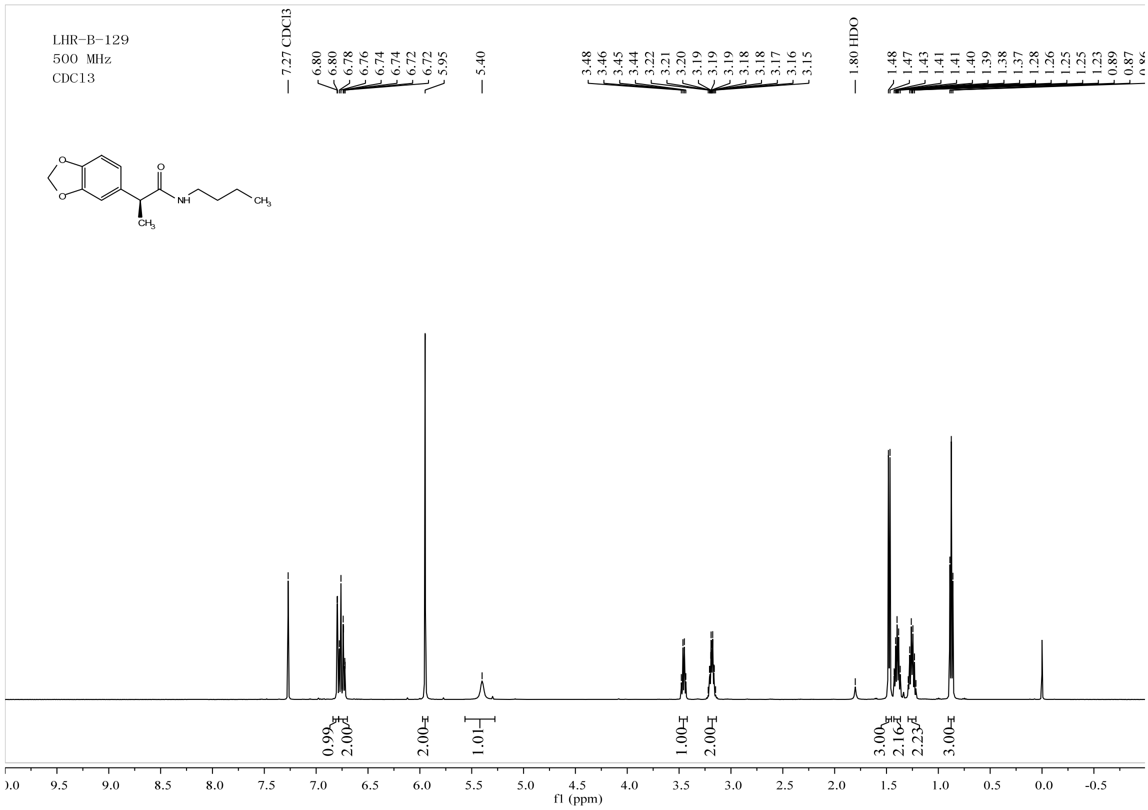


Figure S-24. ^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **3j**.



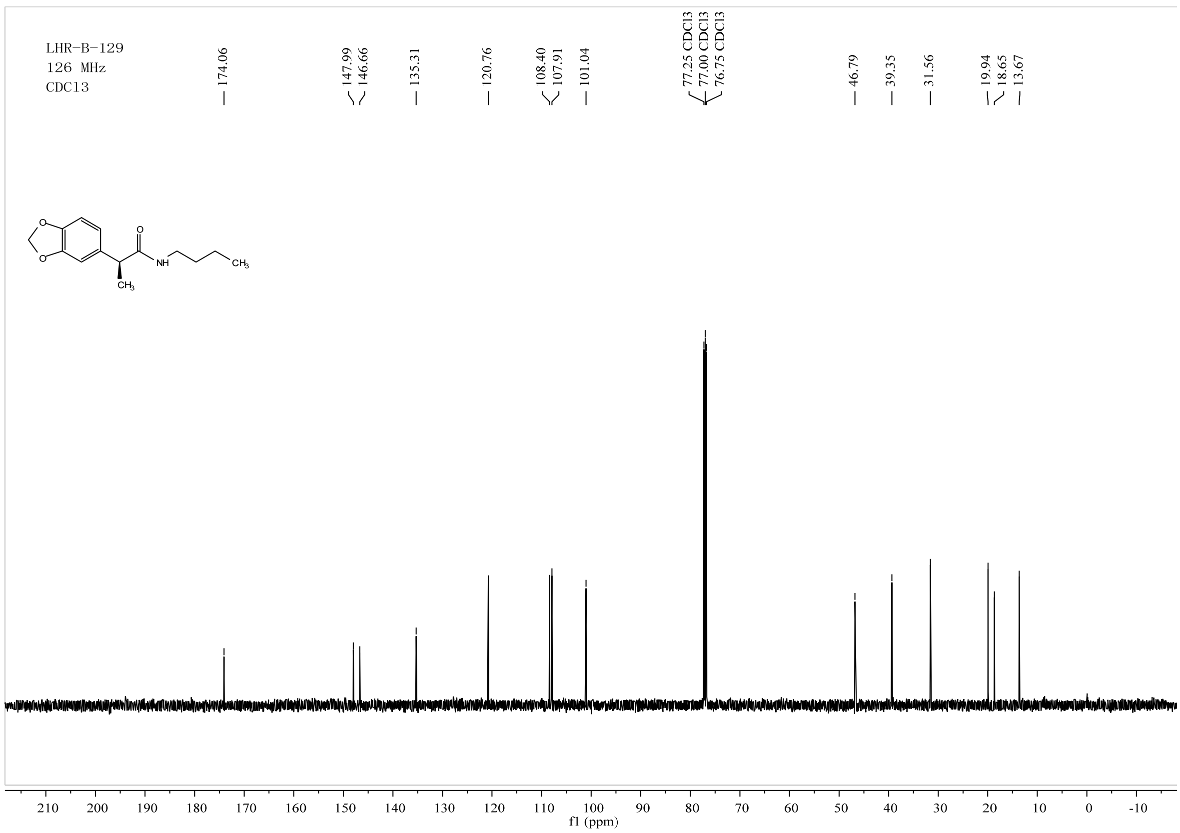
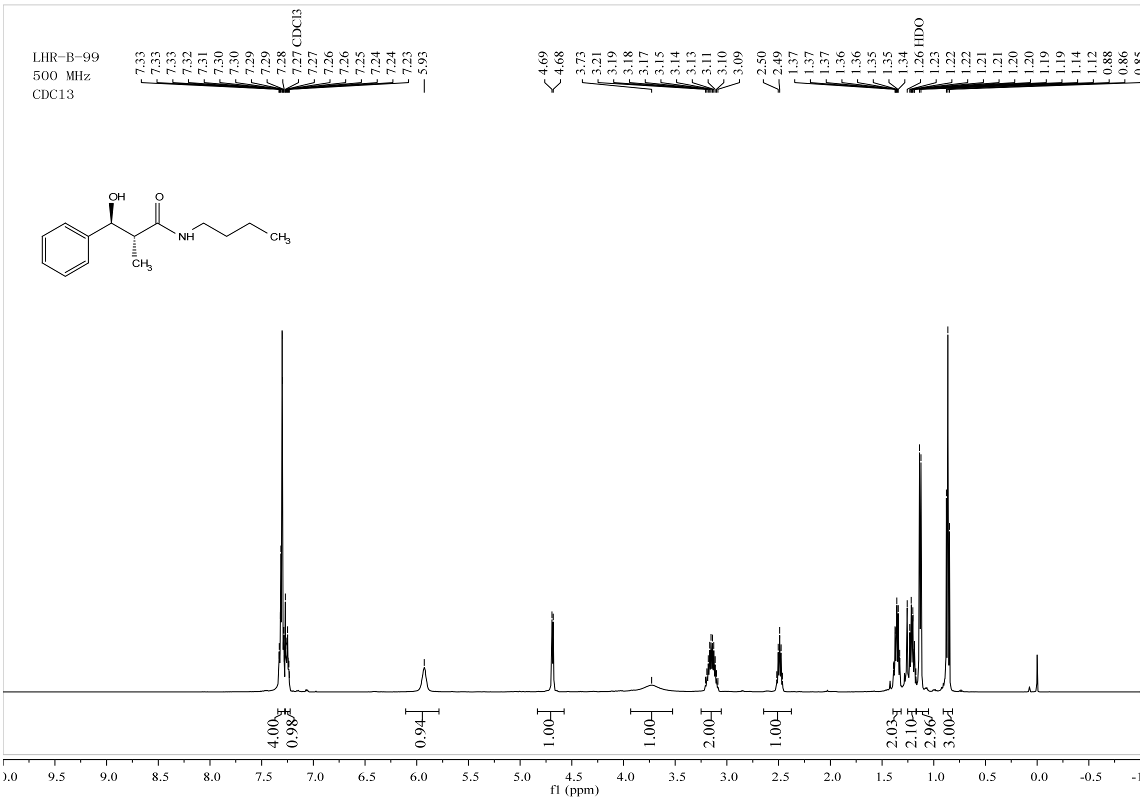


Figure S-25. ^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C NMR (126 MHz, $CDCl_3$) spectra of **3k**.



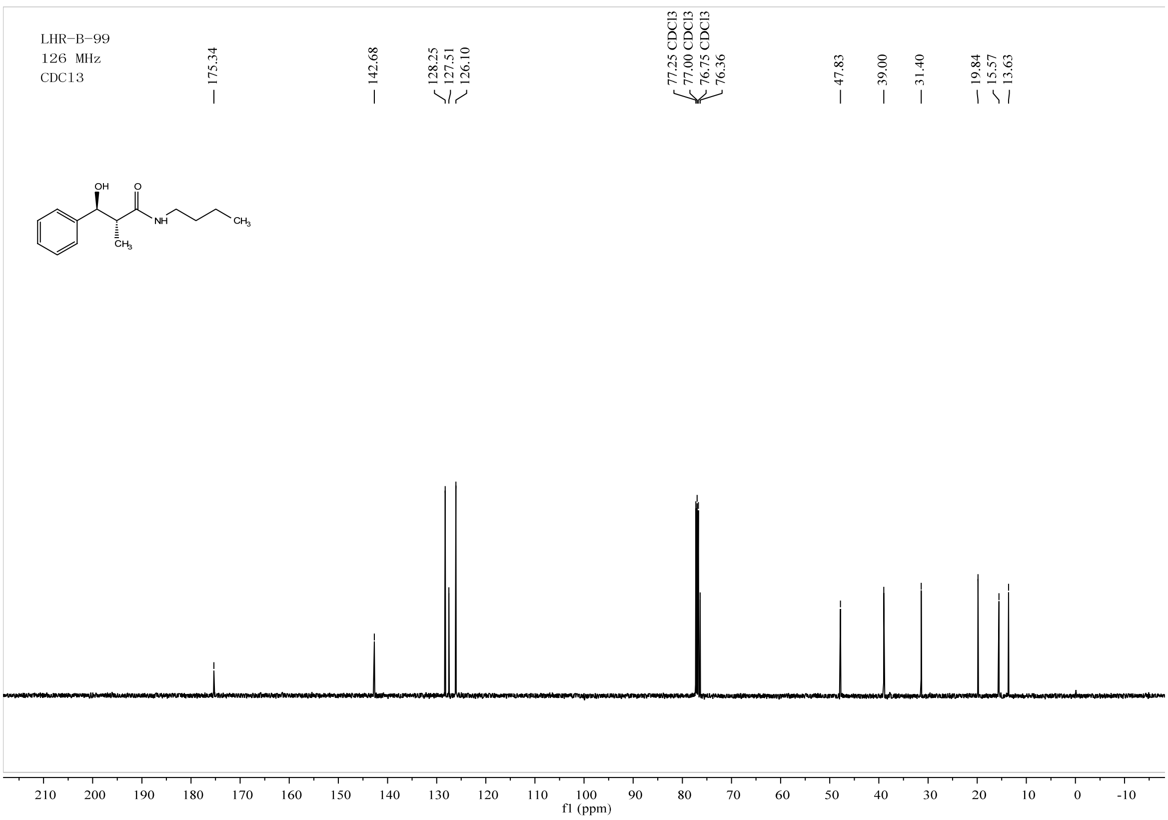
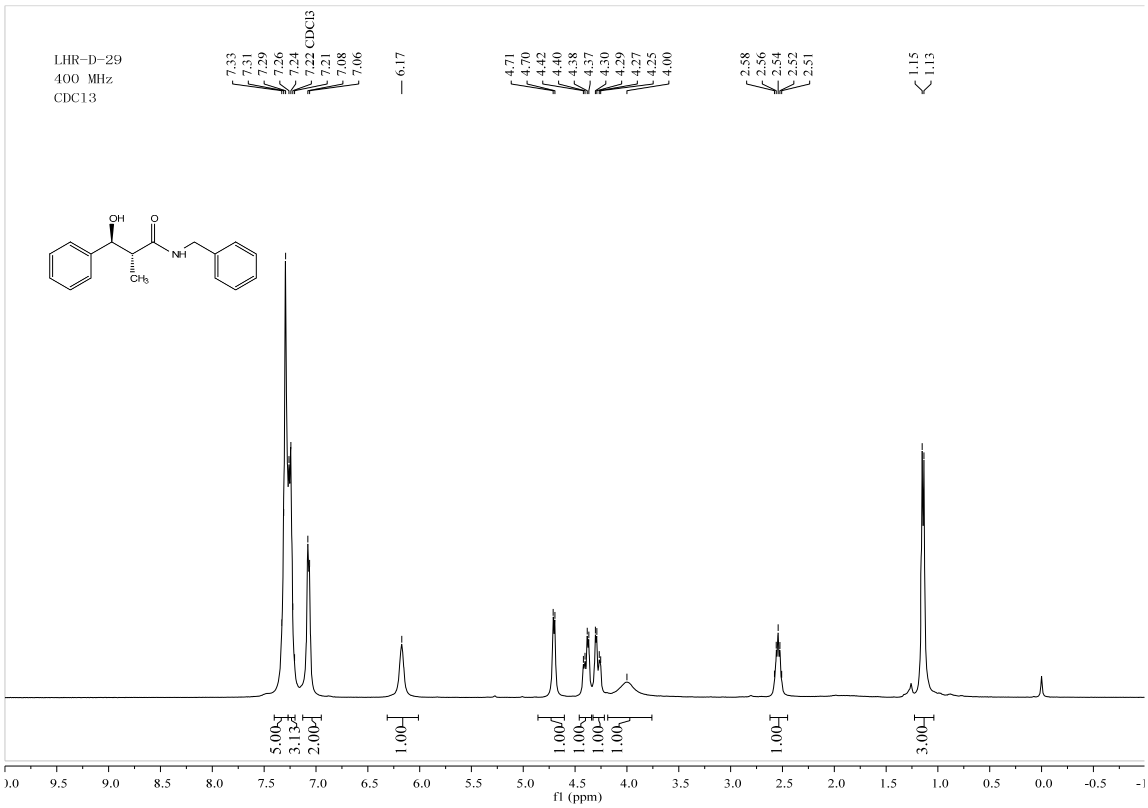


Figure S-26. ^1H NMR (500 MHz, $CDCl_3$) and ^{13}C NMR (126 MHz, $CDCl_3$) spectra of **3l**.



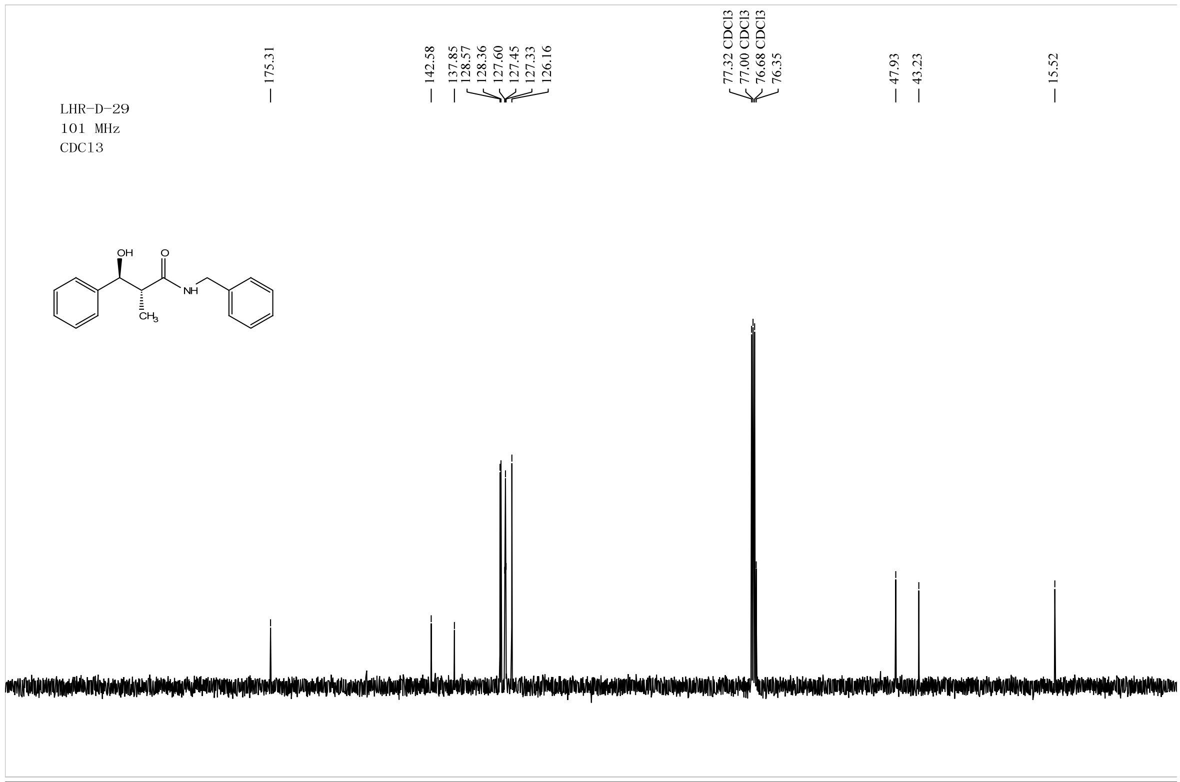
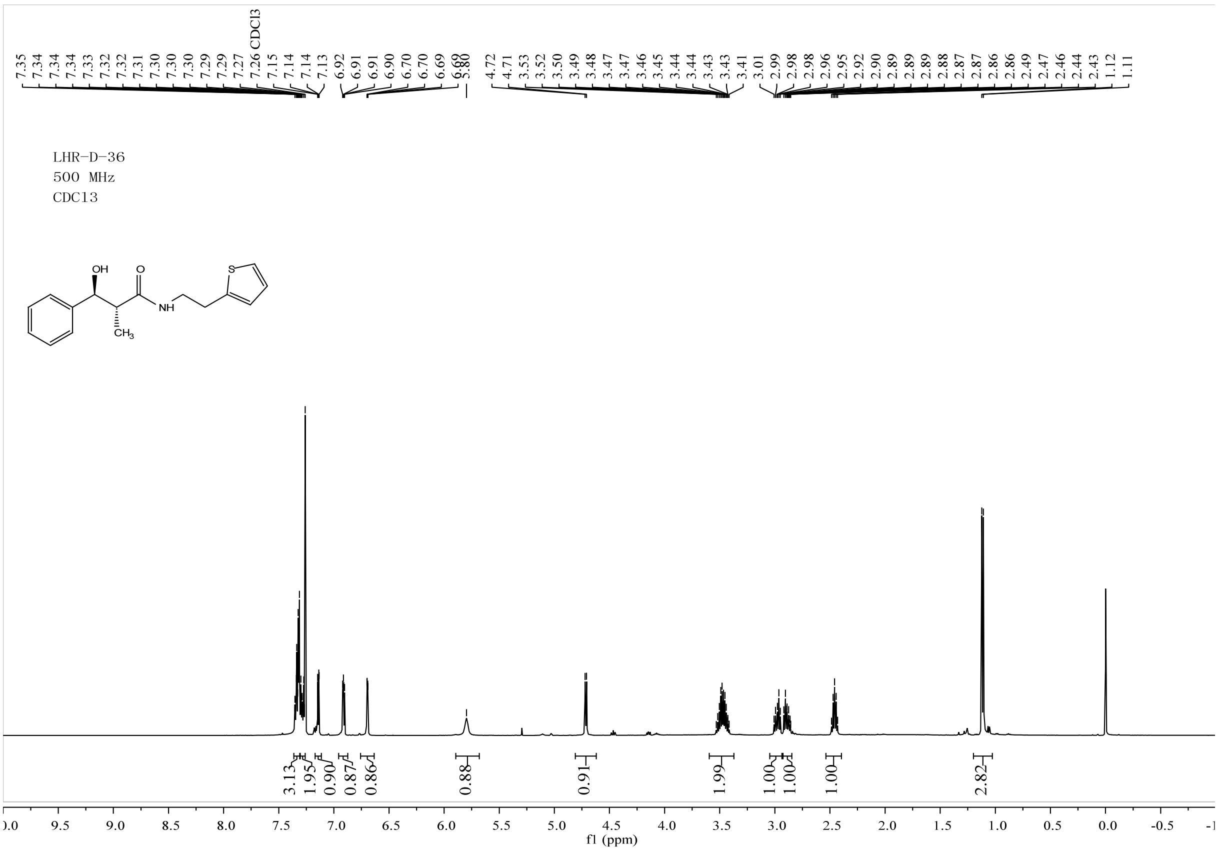


Figure S-27. ^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **3m**.



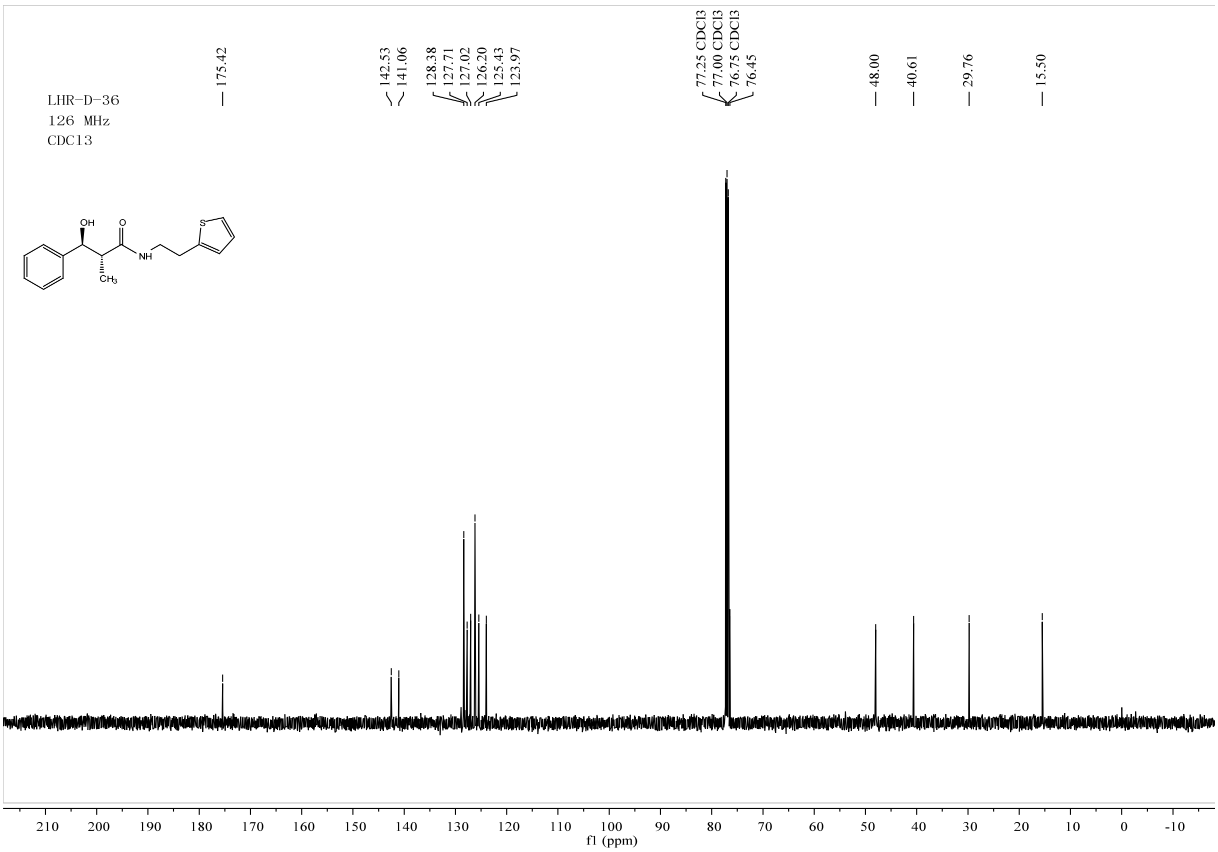
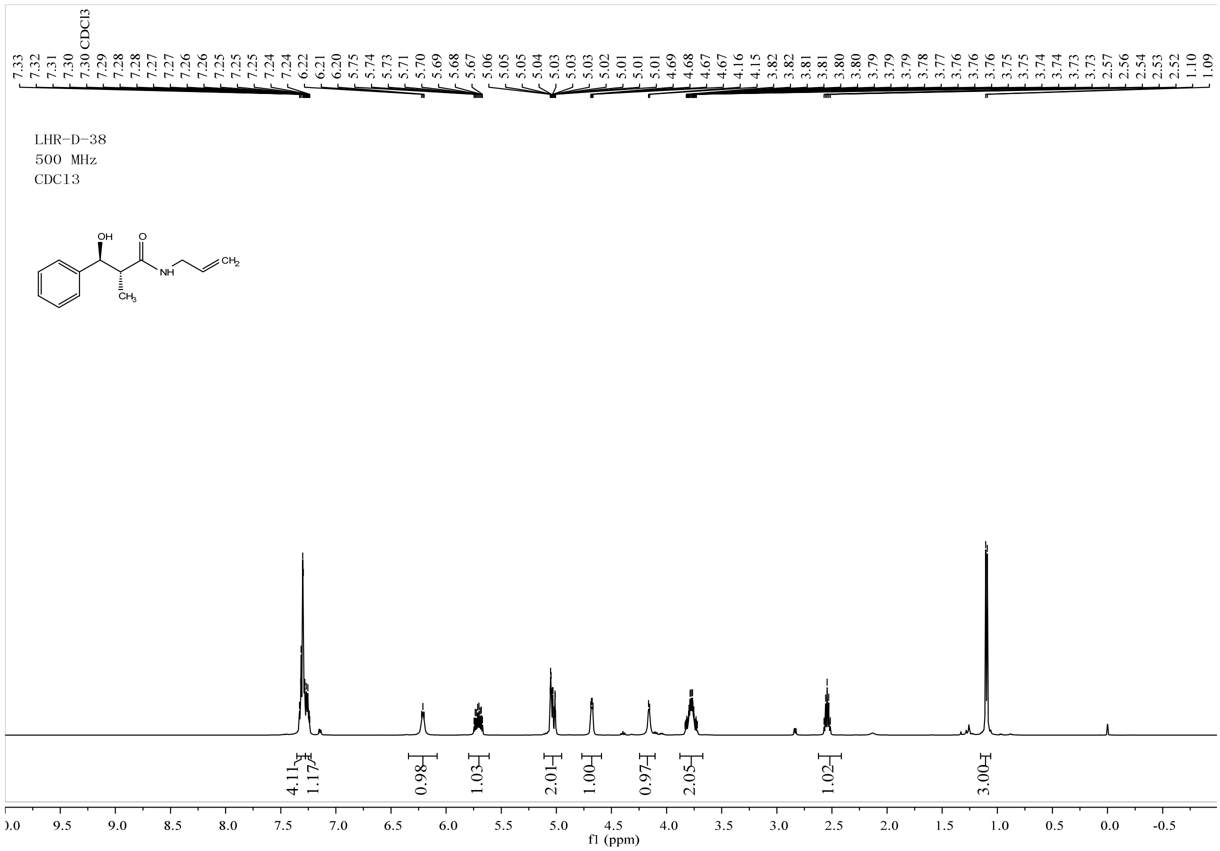


Figure S-28. ^1H NMR (500 MHz, $CDCl_3$) and ^{13}C NMR (126 MHz, $CDCl_3$) spectra of **3n**.



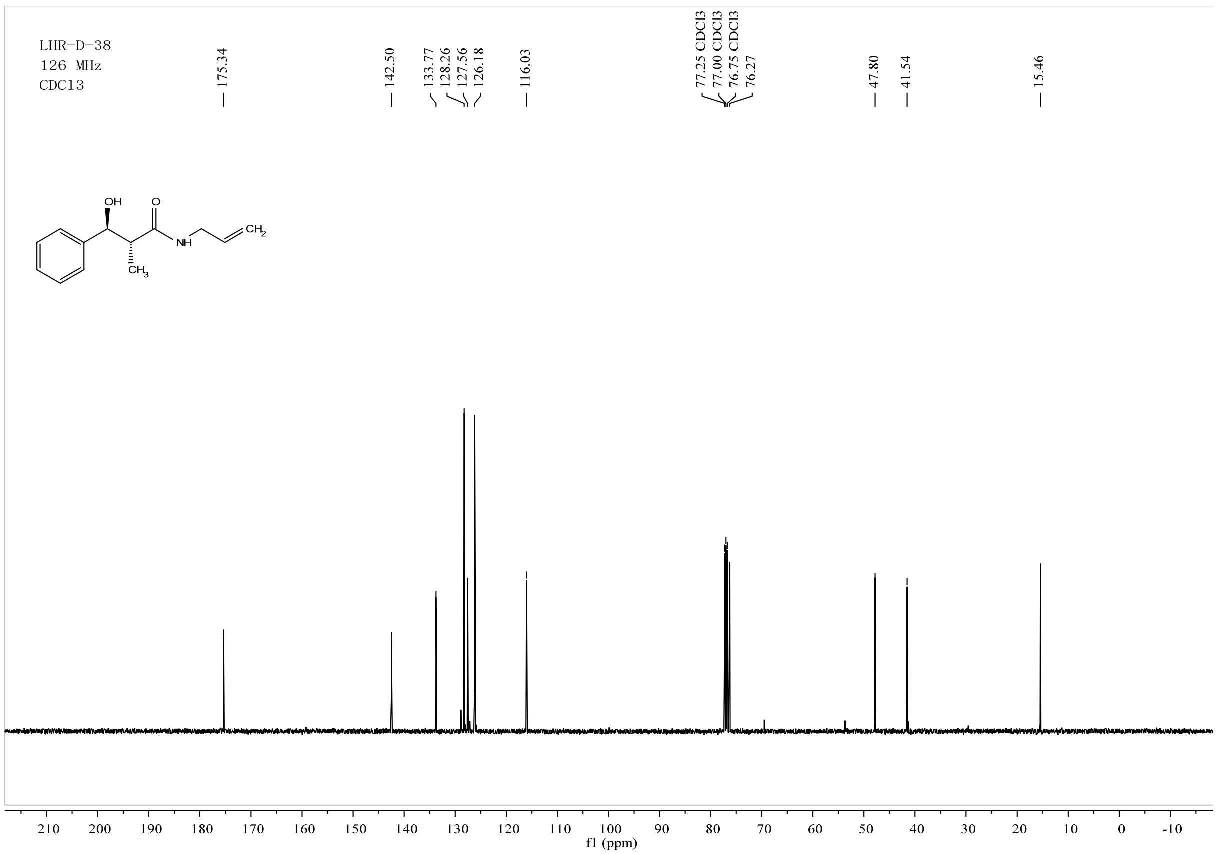
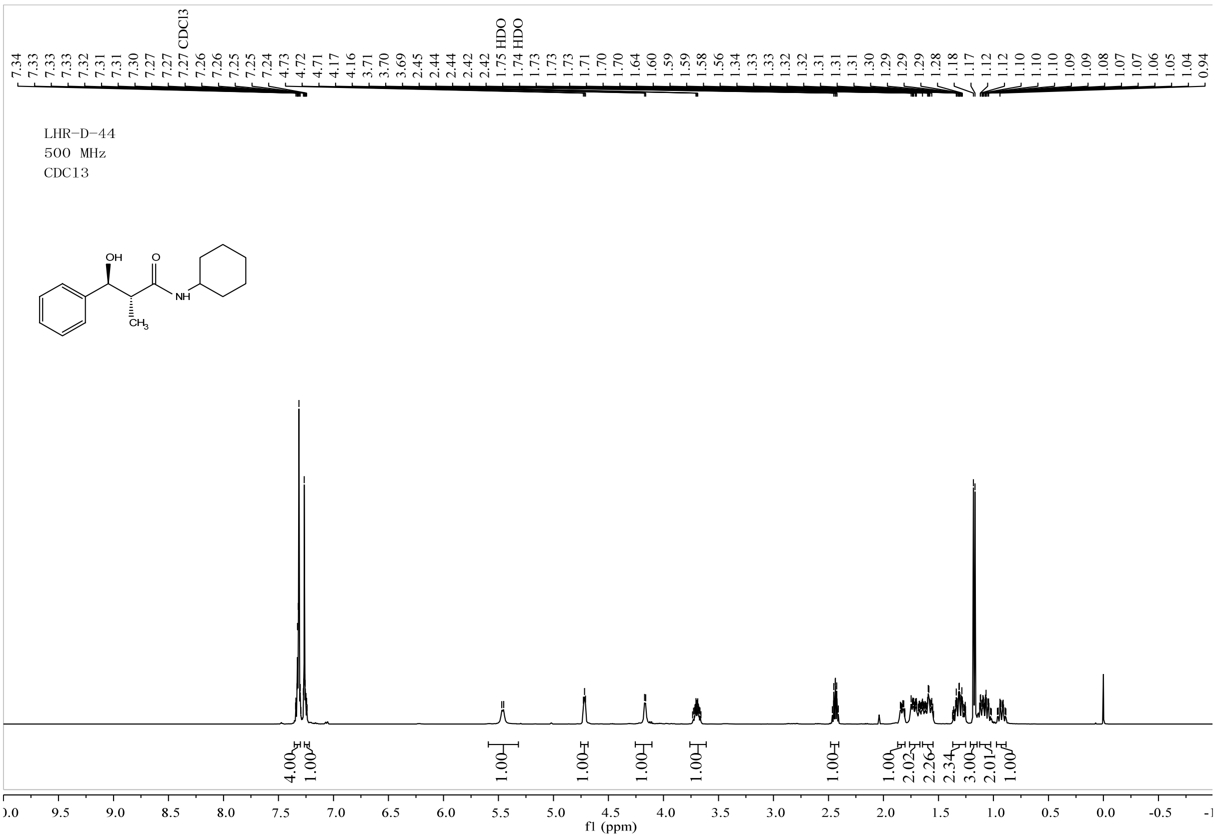


Figure S-29. ^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C NMR (126 MHz, $CDCl_3$) spectra of **3o**.



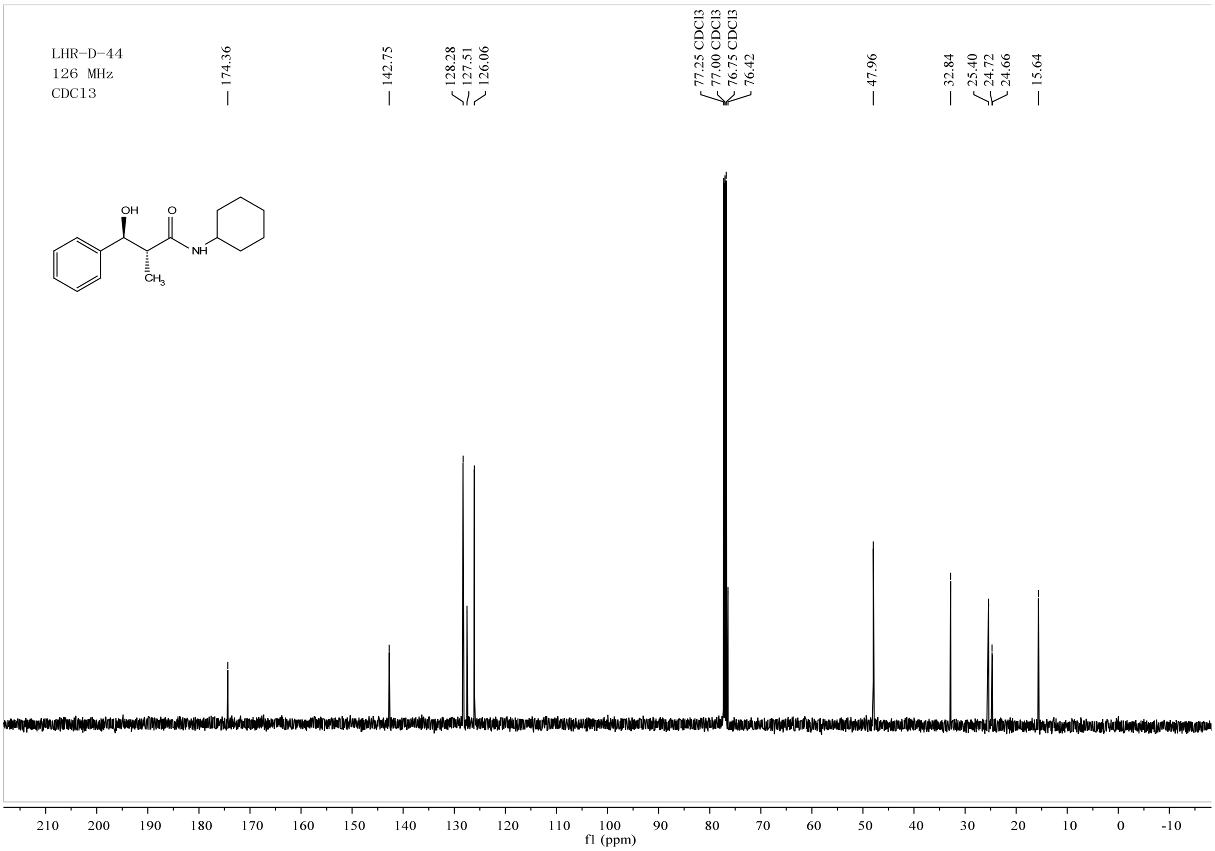
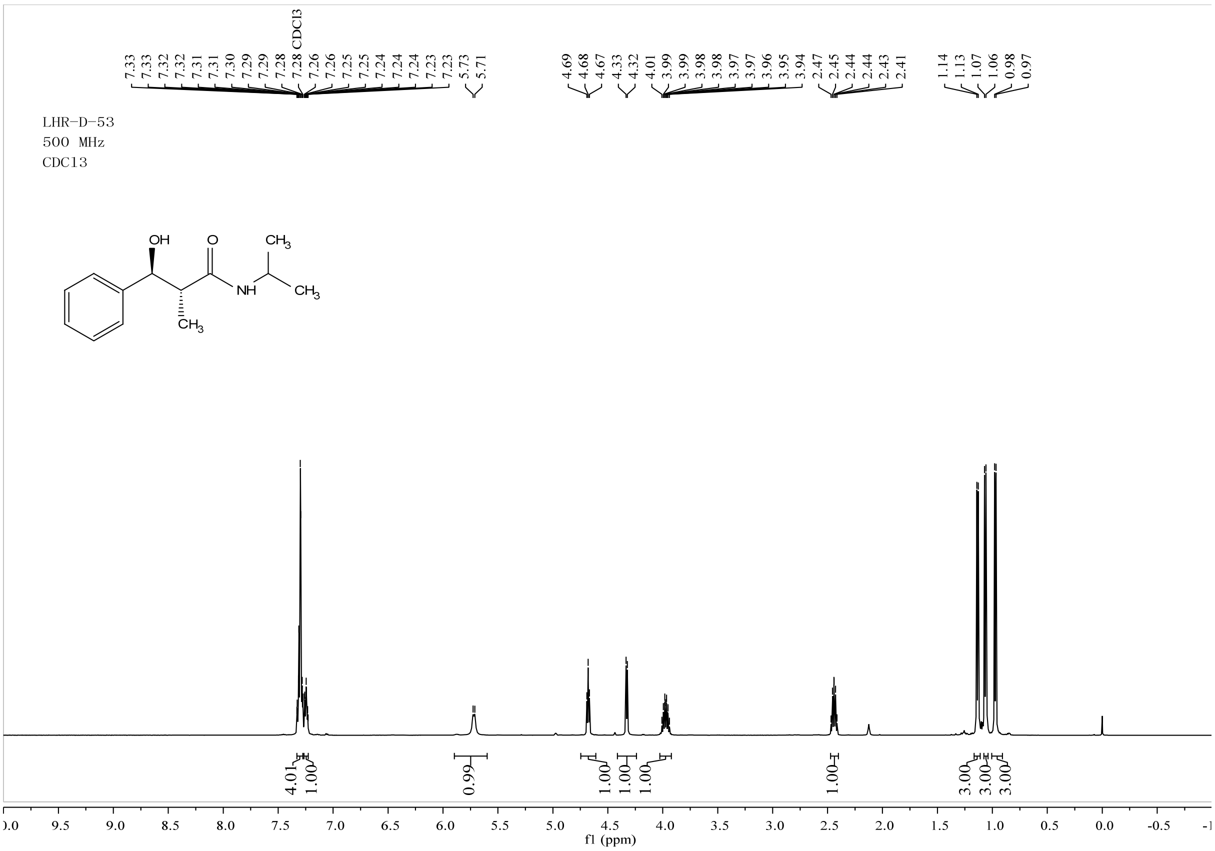


Figure S-30. ^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C NMR (126 MHz, $CDCl_3$) spectra of **3p**.



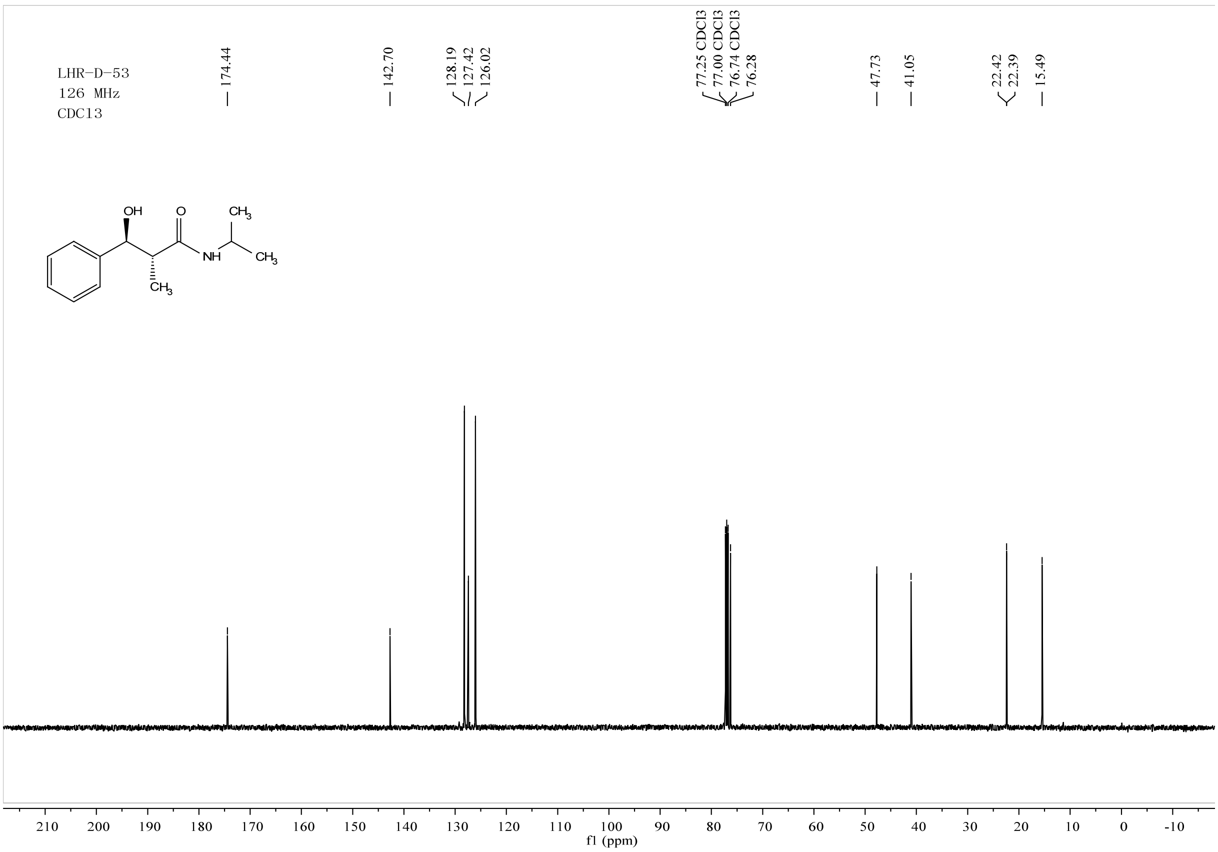
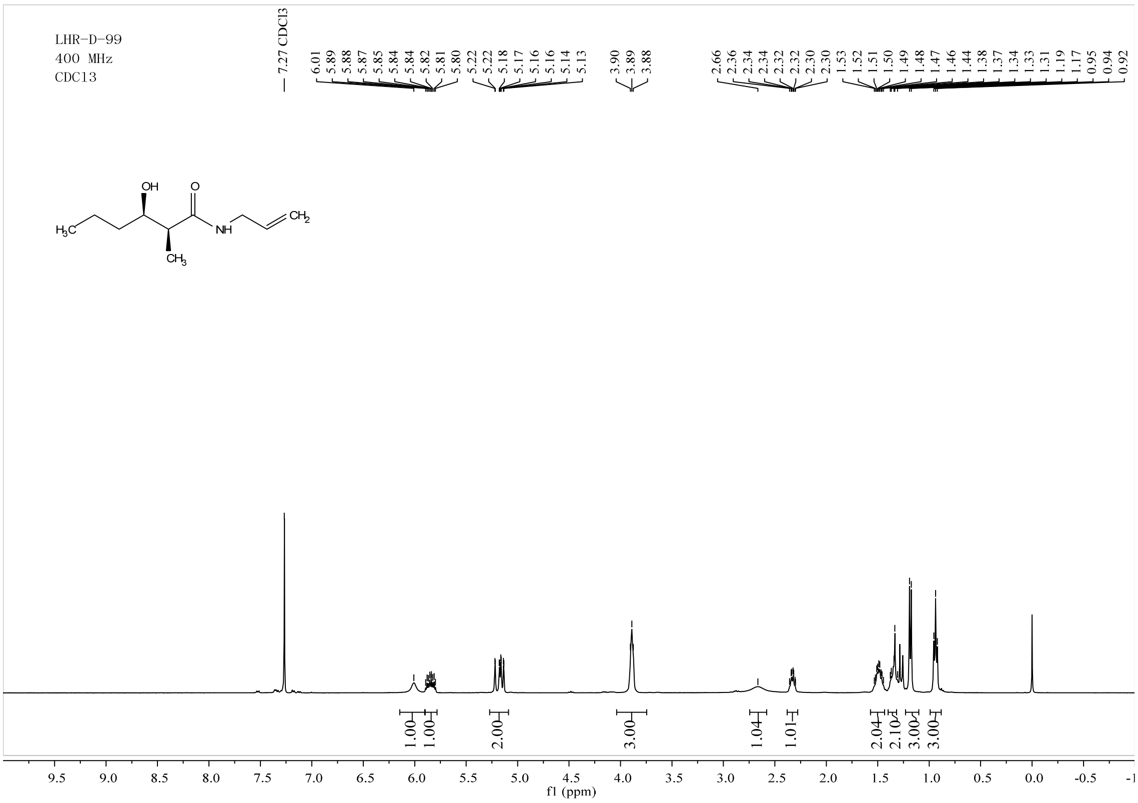


Figure S-31. ^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C NMR (126 MHz, $CDCl_3$) spectra of **3q**.



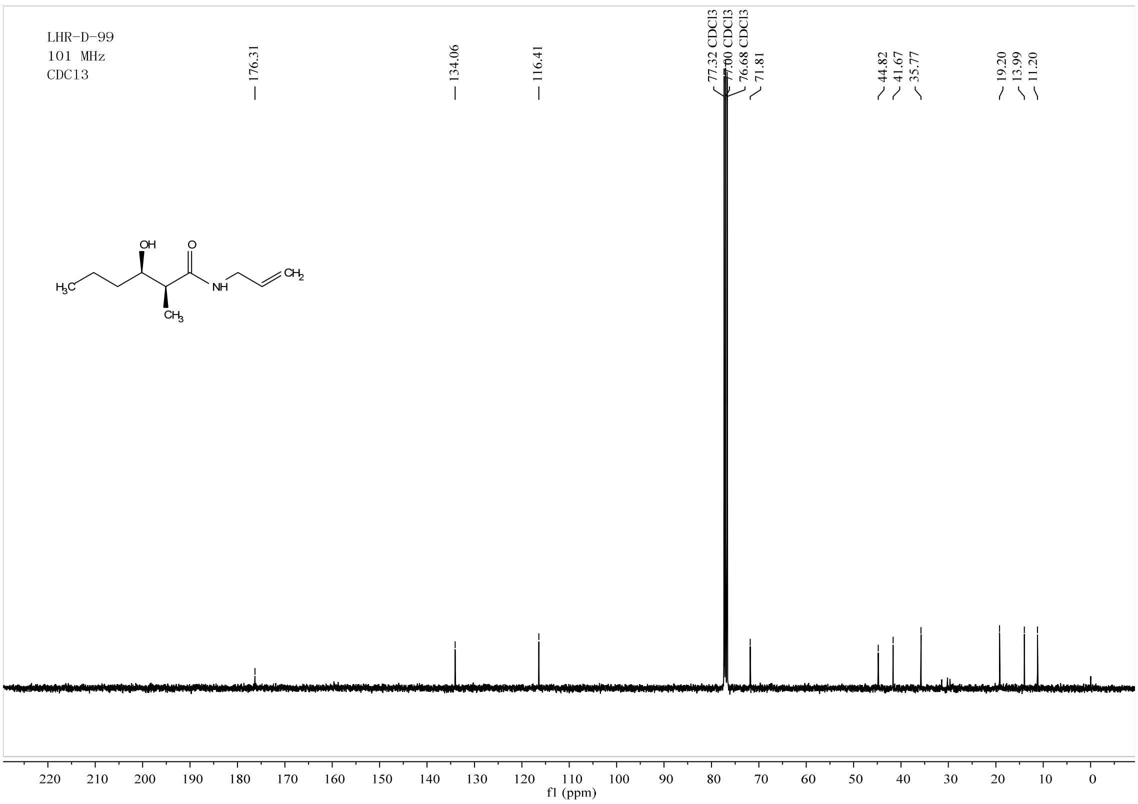
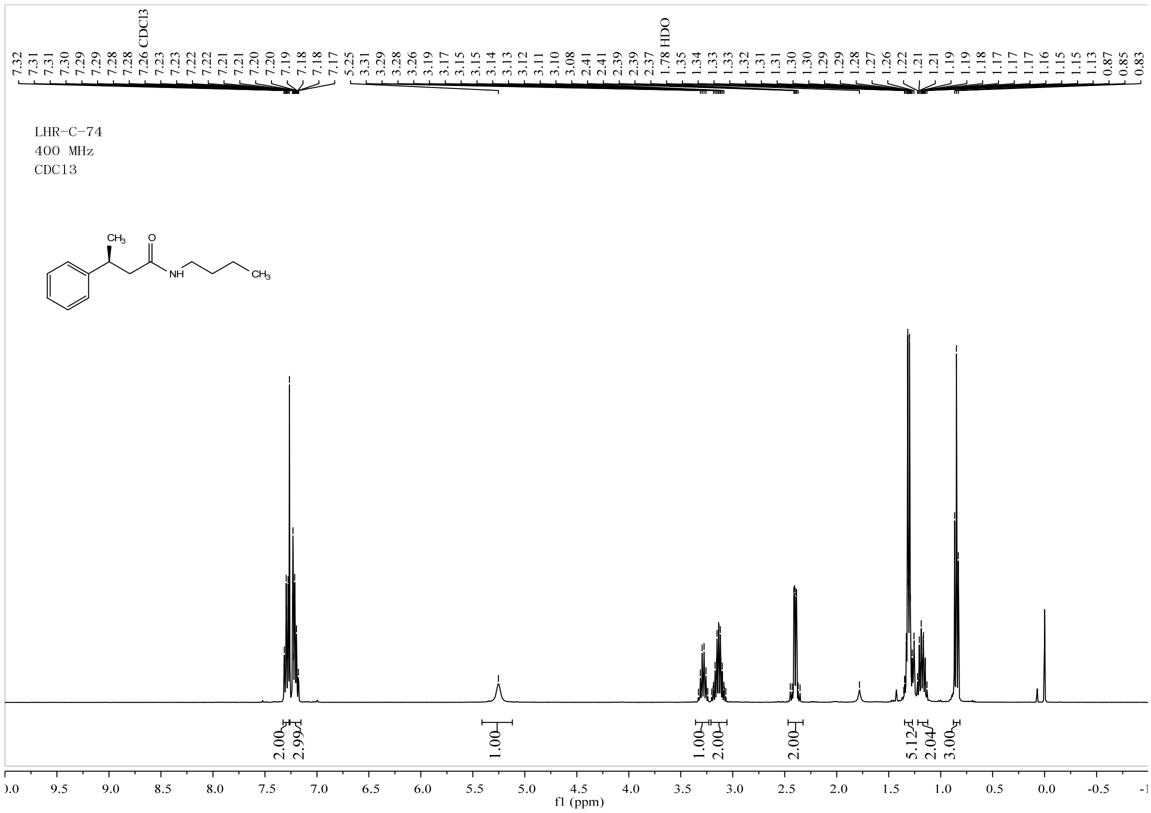


Figure S-32. ^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **3r**.



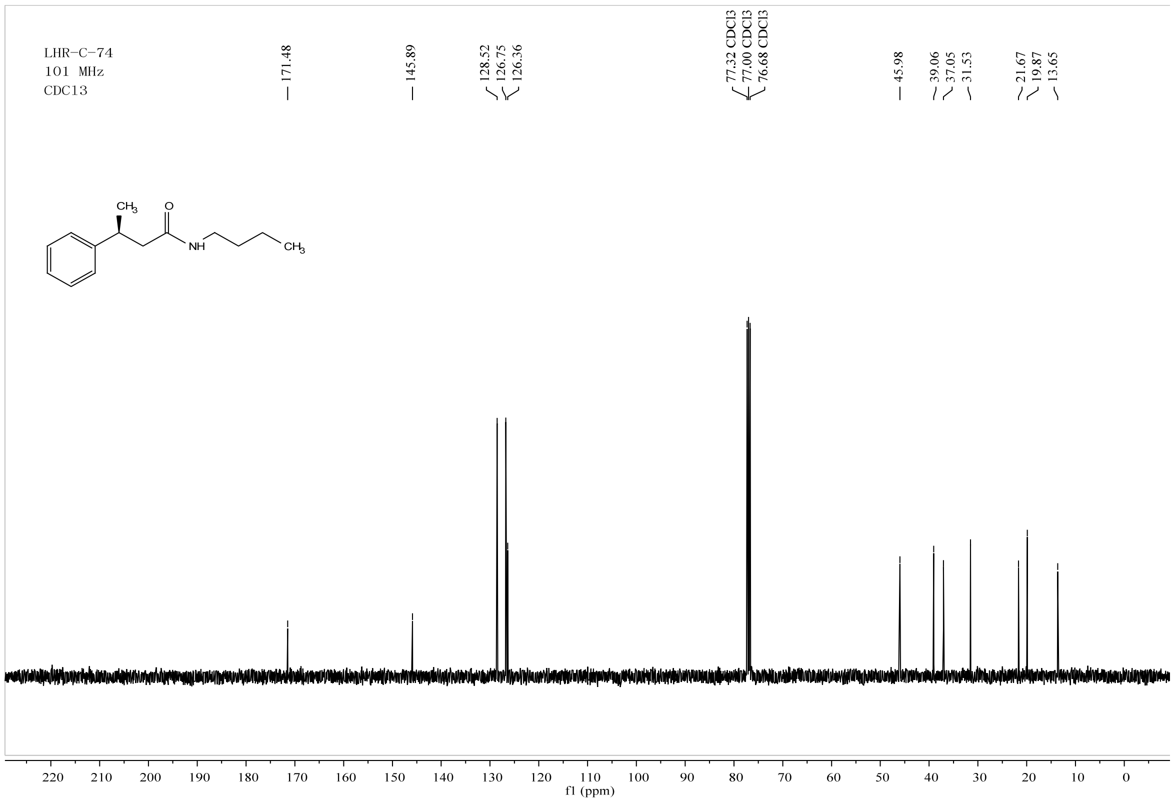
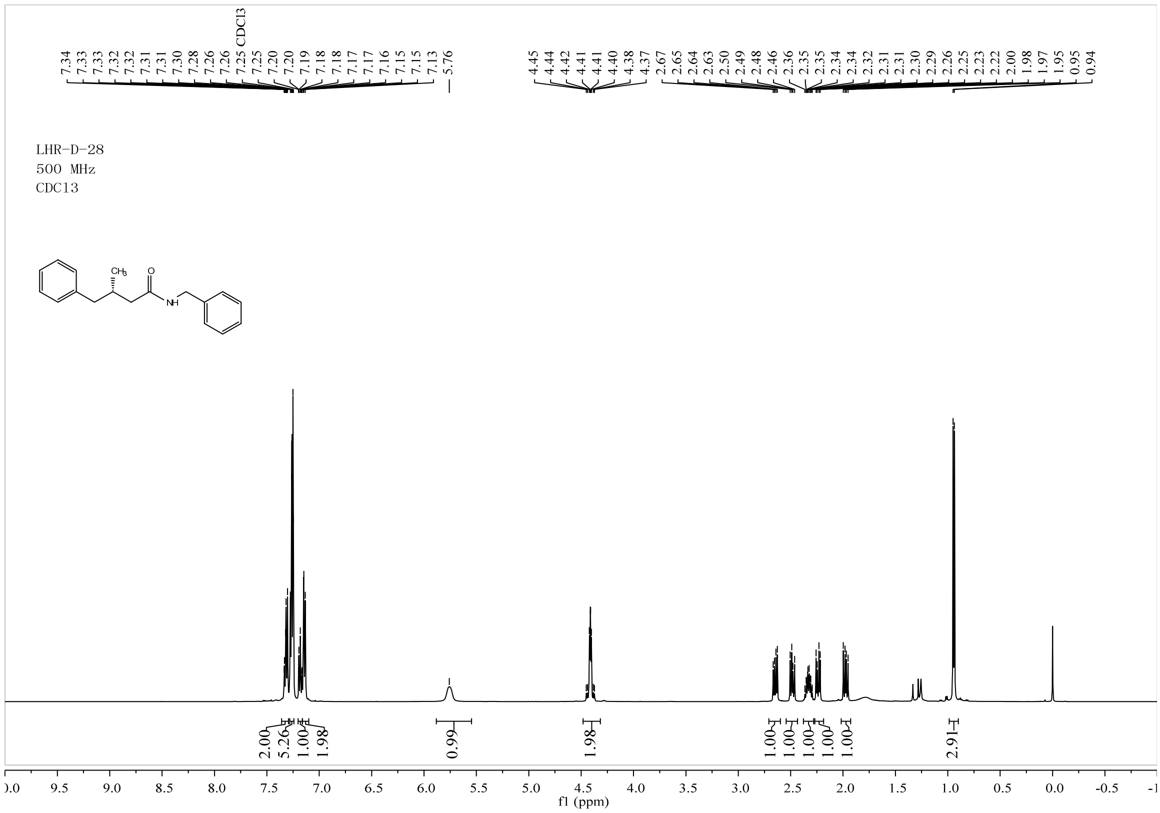


Figure S-33. ^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **3s**.



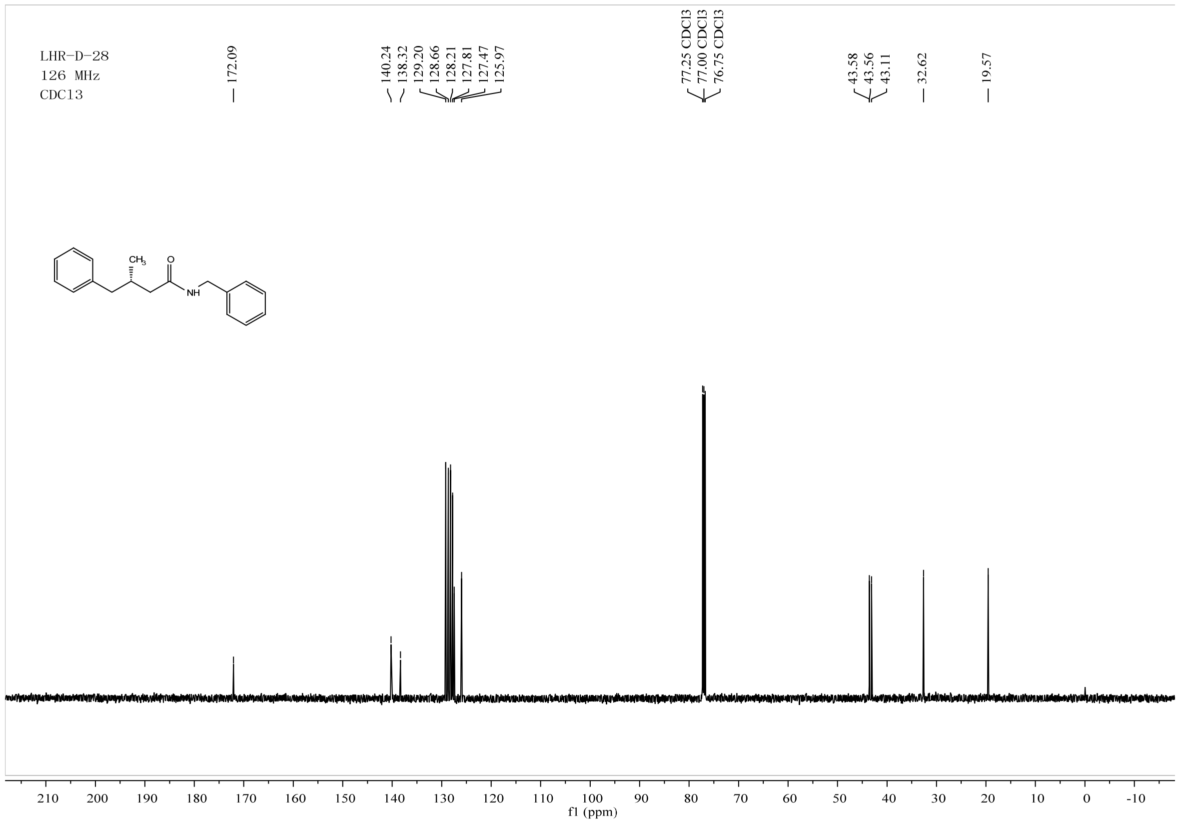
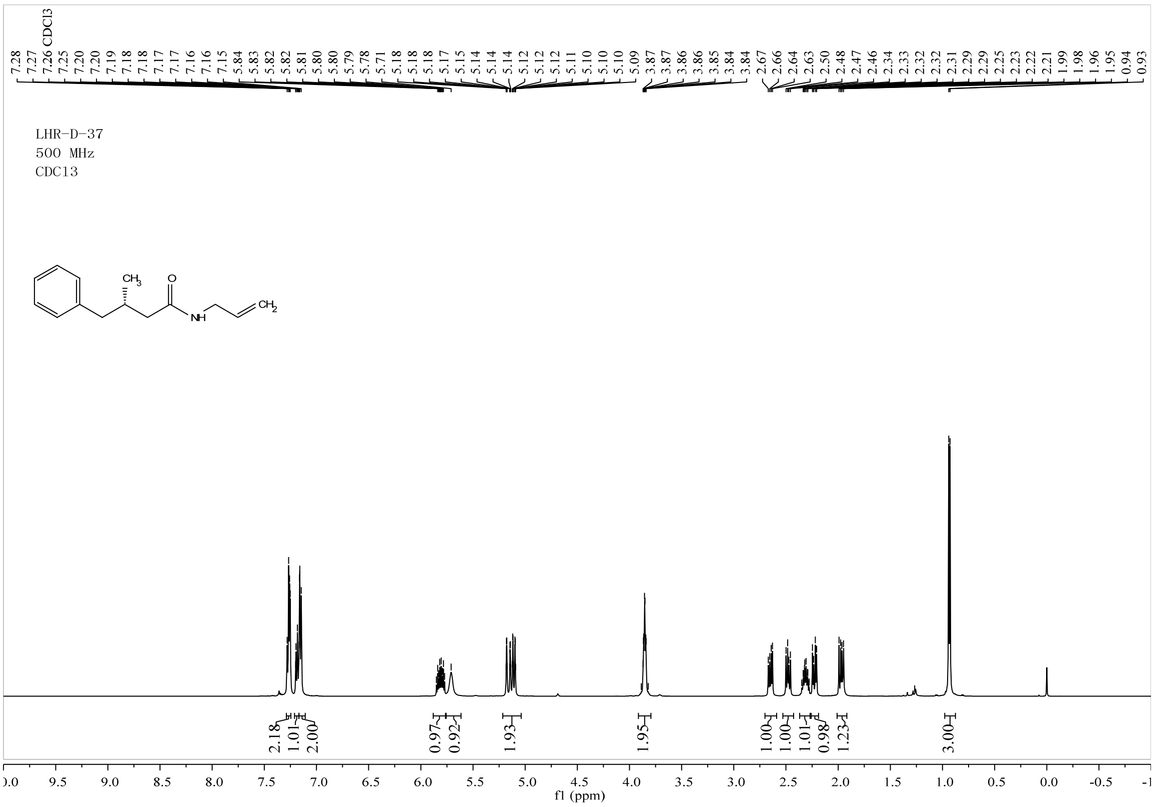


Figure S-34. ^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C NMR (126 MHz, $CDCl_3$) spectra of **3t**.



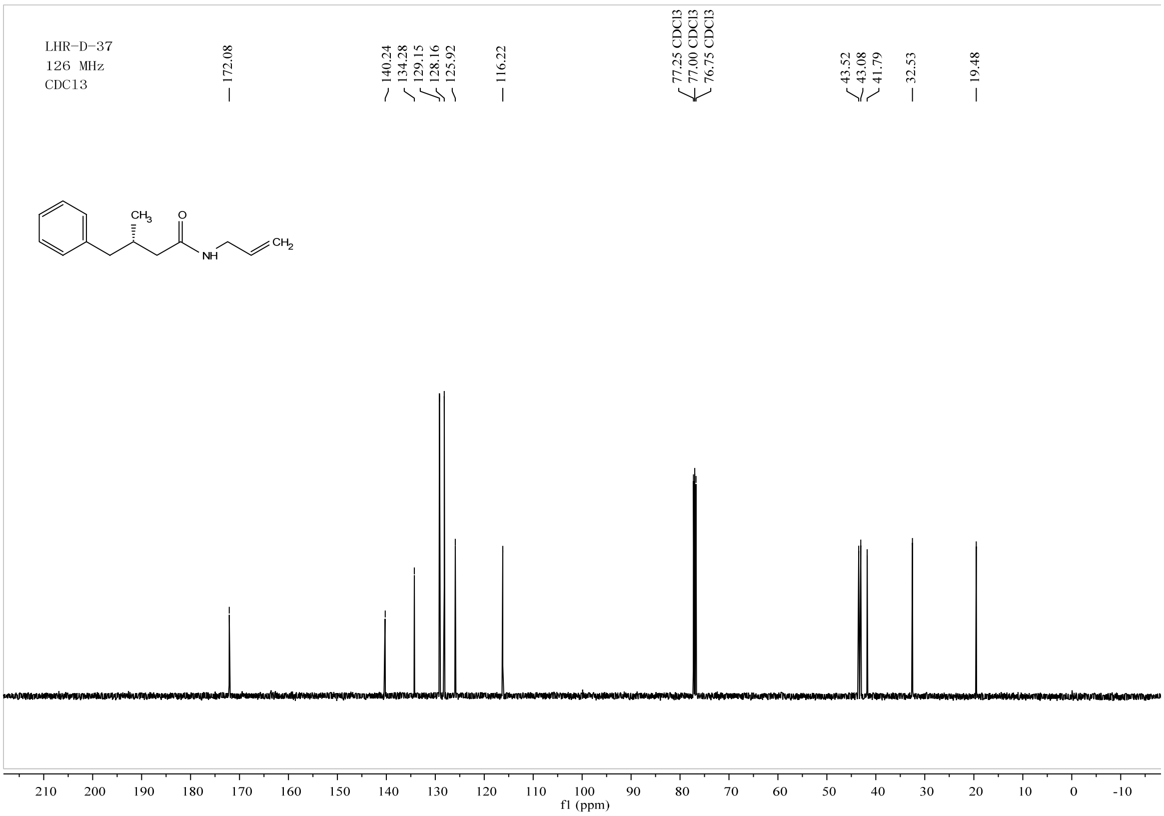
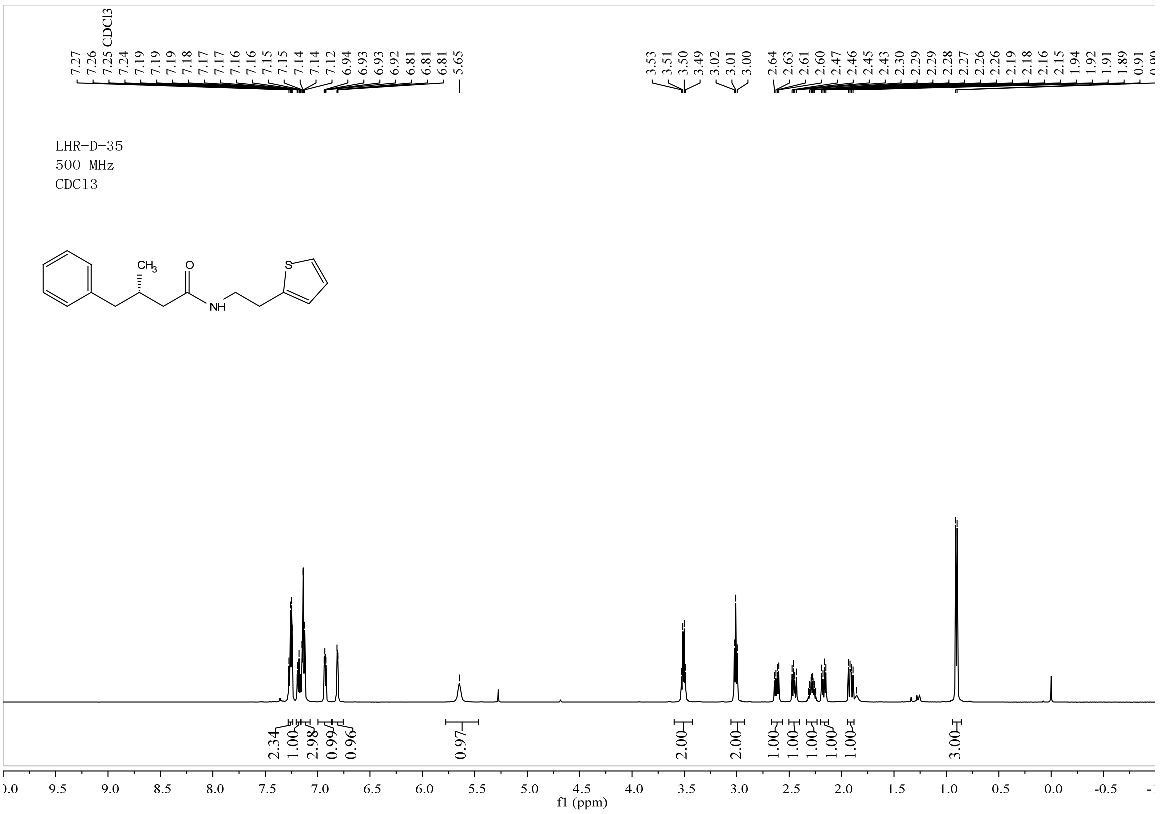


Figure S-35. ^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C NMR (126 MHz, $CDCl_3$) spectra of **3u**.



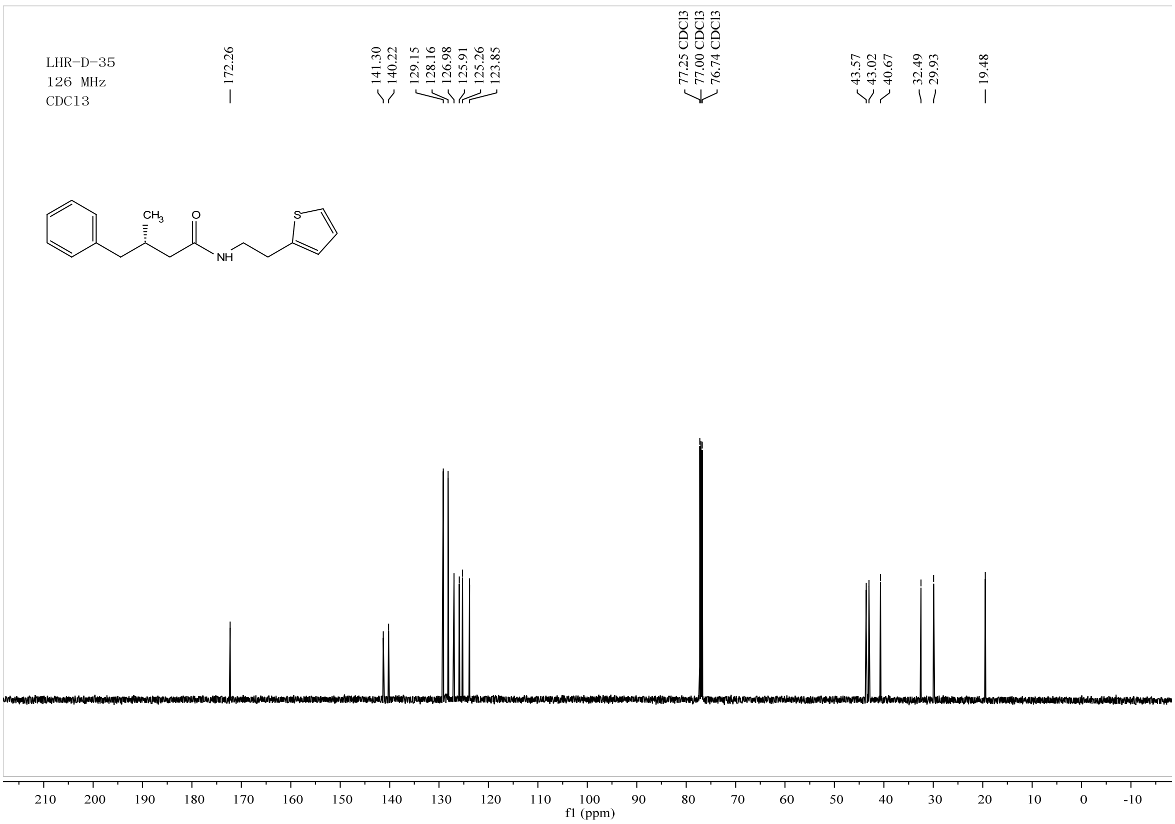
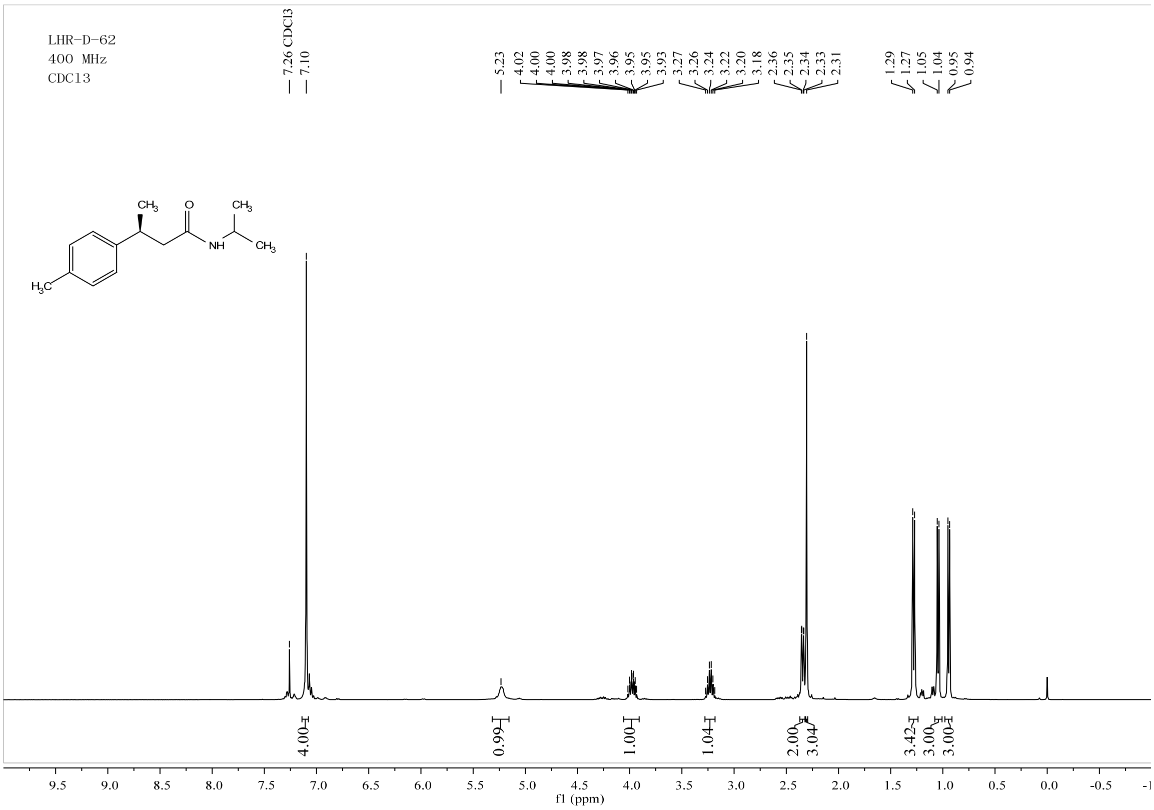


Figure S-36. ^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C NMR (126 MHz, $CDCl_3$) spectra of **3v**.



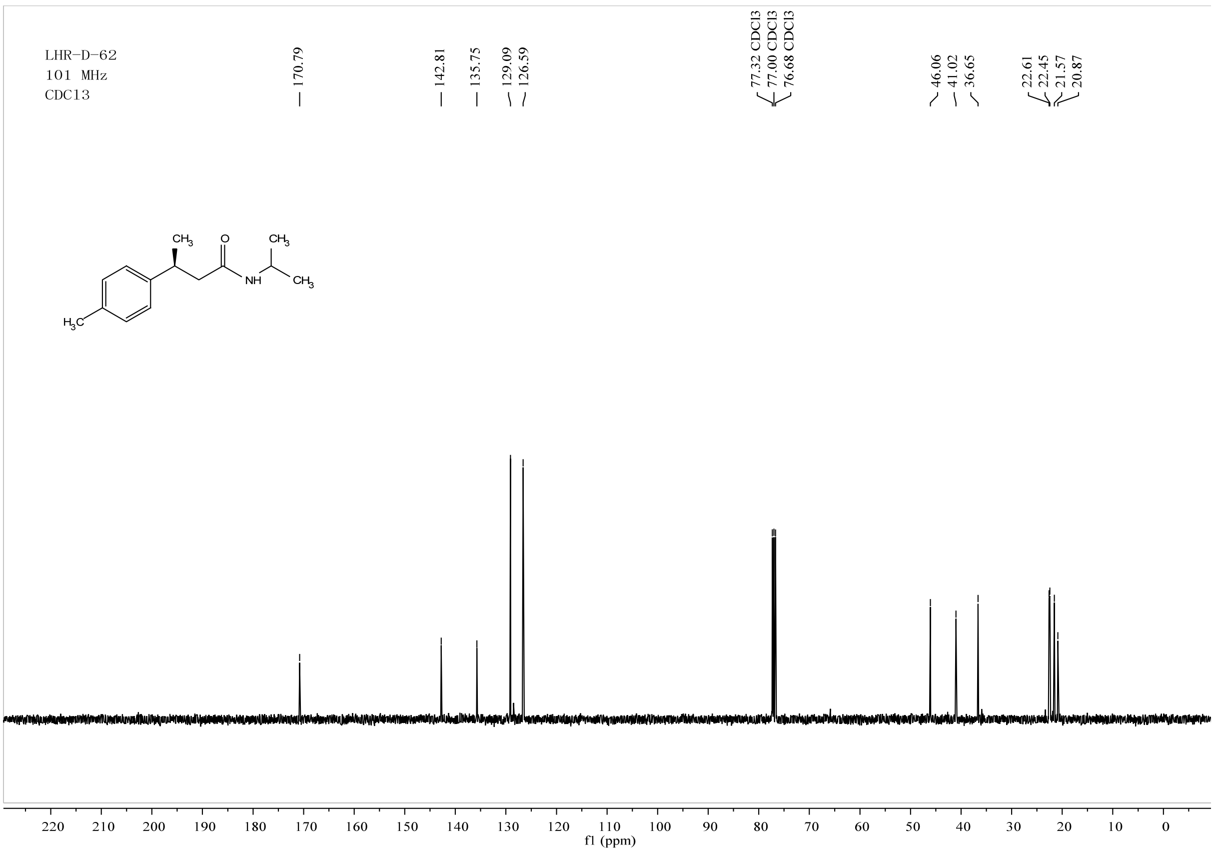


Figure S-37. ^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **3w**.

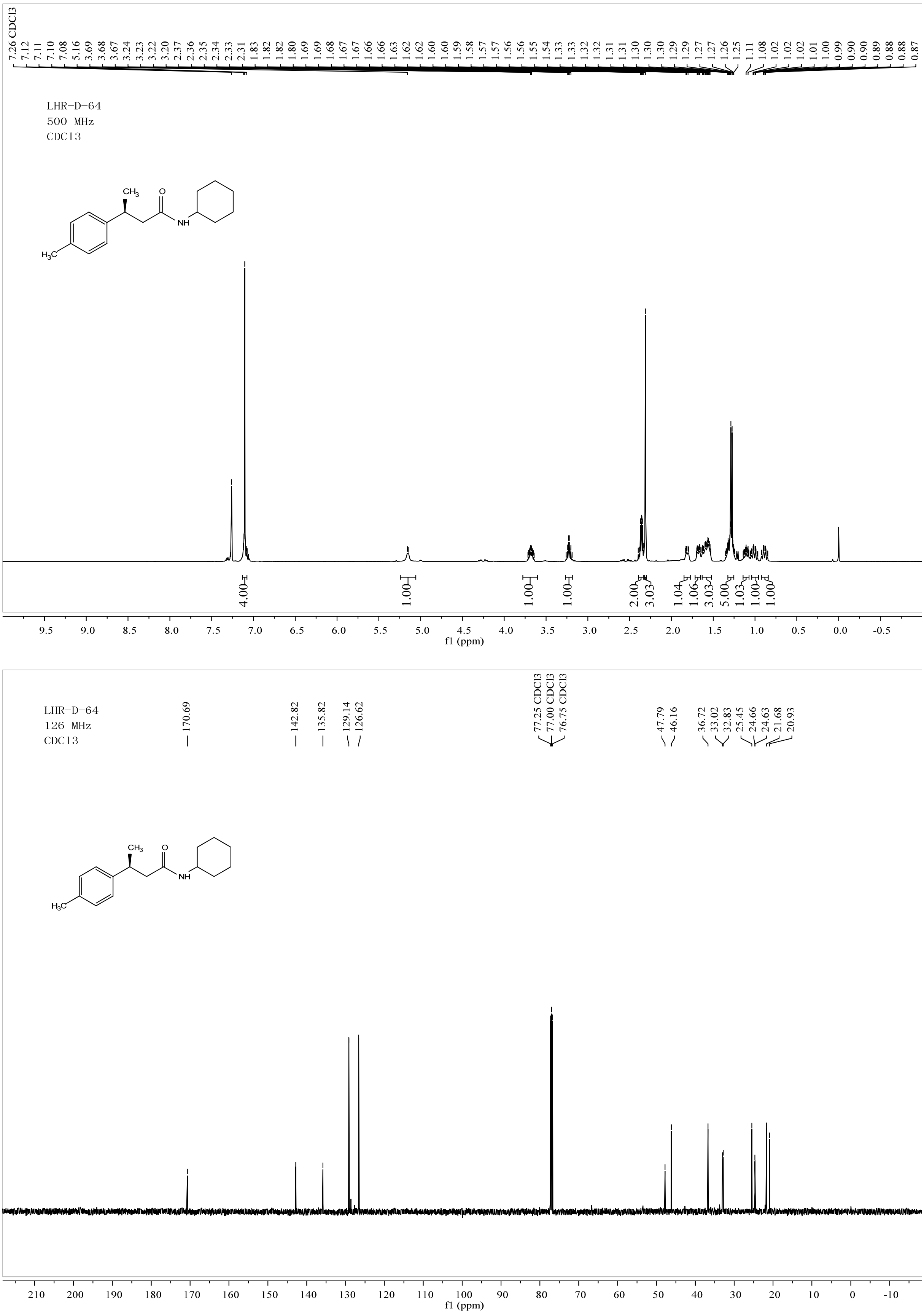
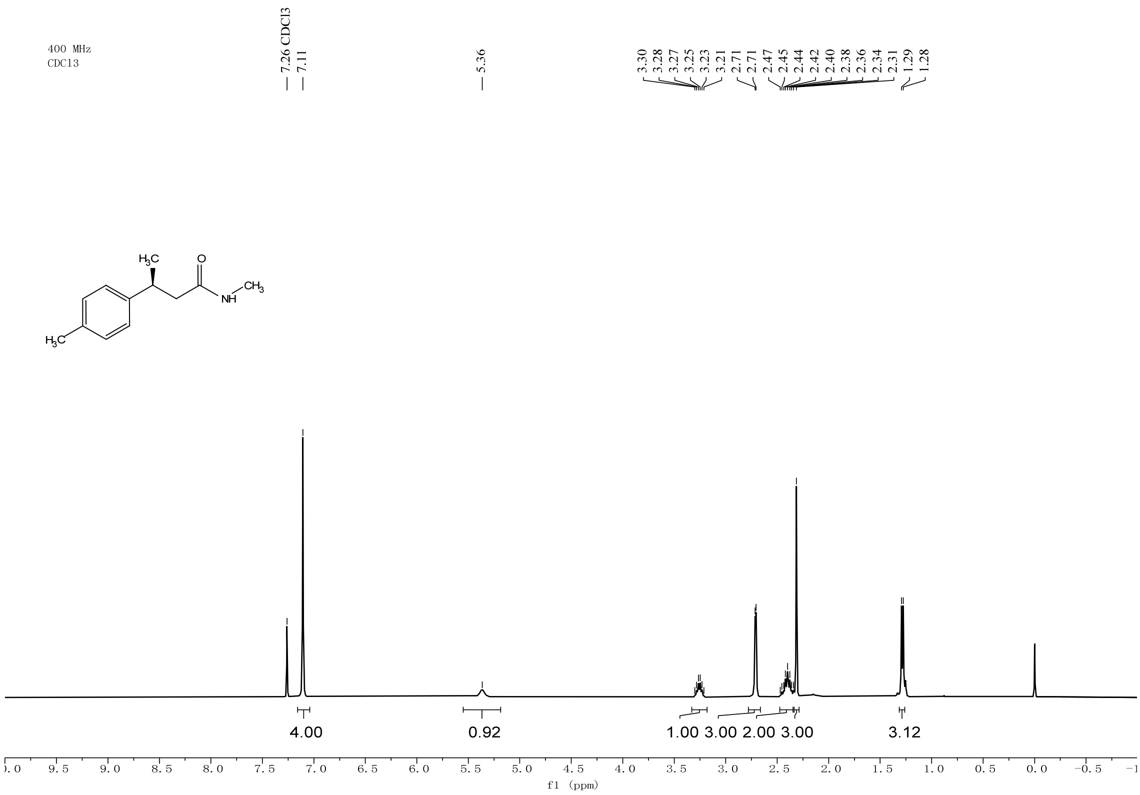


Figure S-38. ^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C NMR (126 MHz, $CDCl_3$) spectra of **3x**.



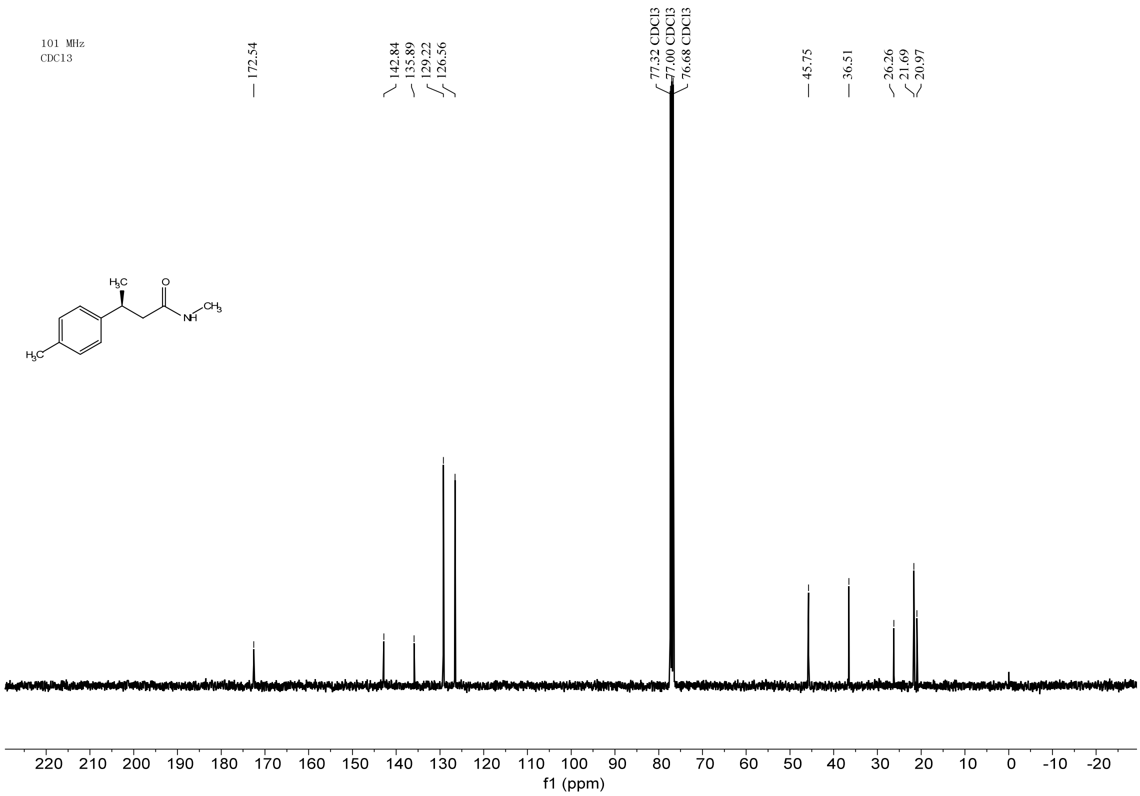


Figure S-39. ^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **3y**.

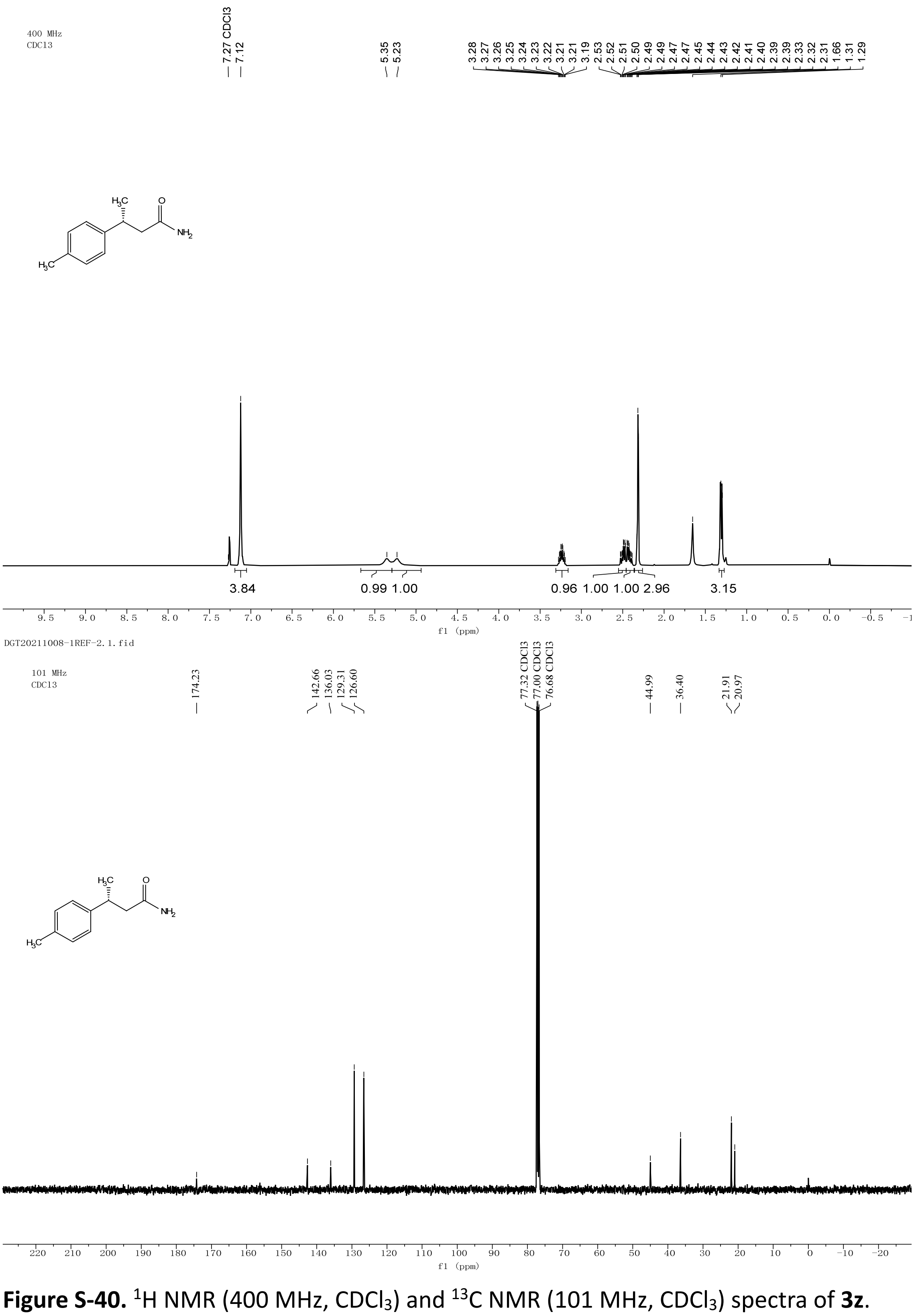
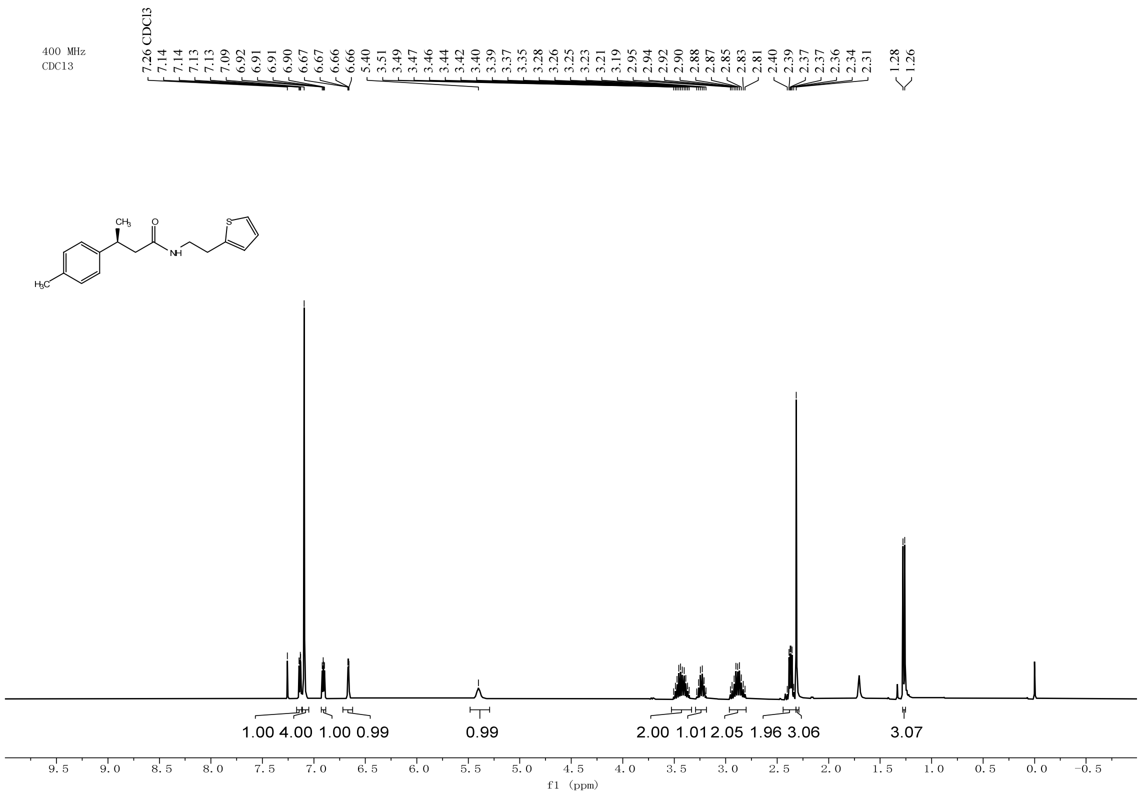


Figure S-40. ^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **3z**.



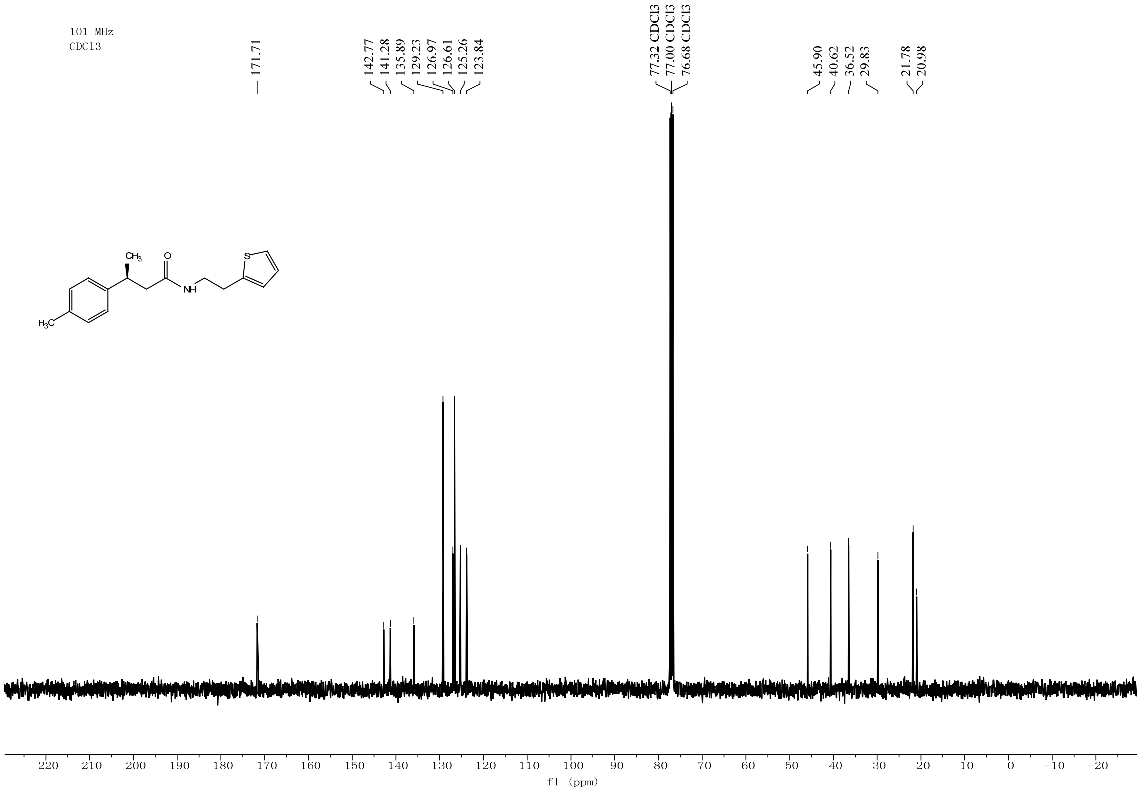
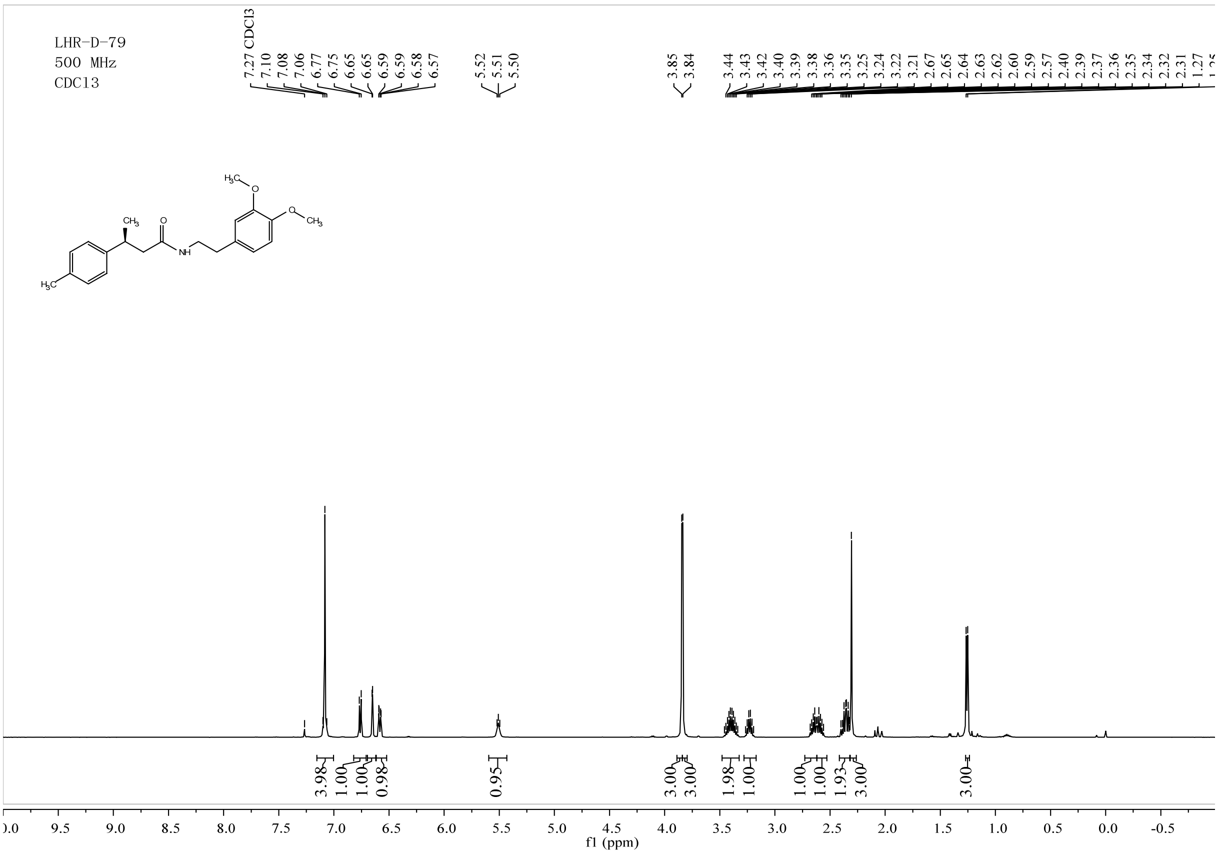


Figure S-41. ^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **3aa**.



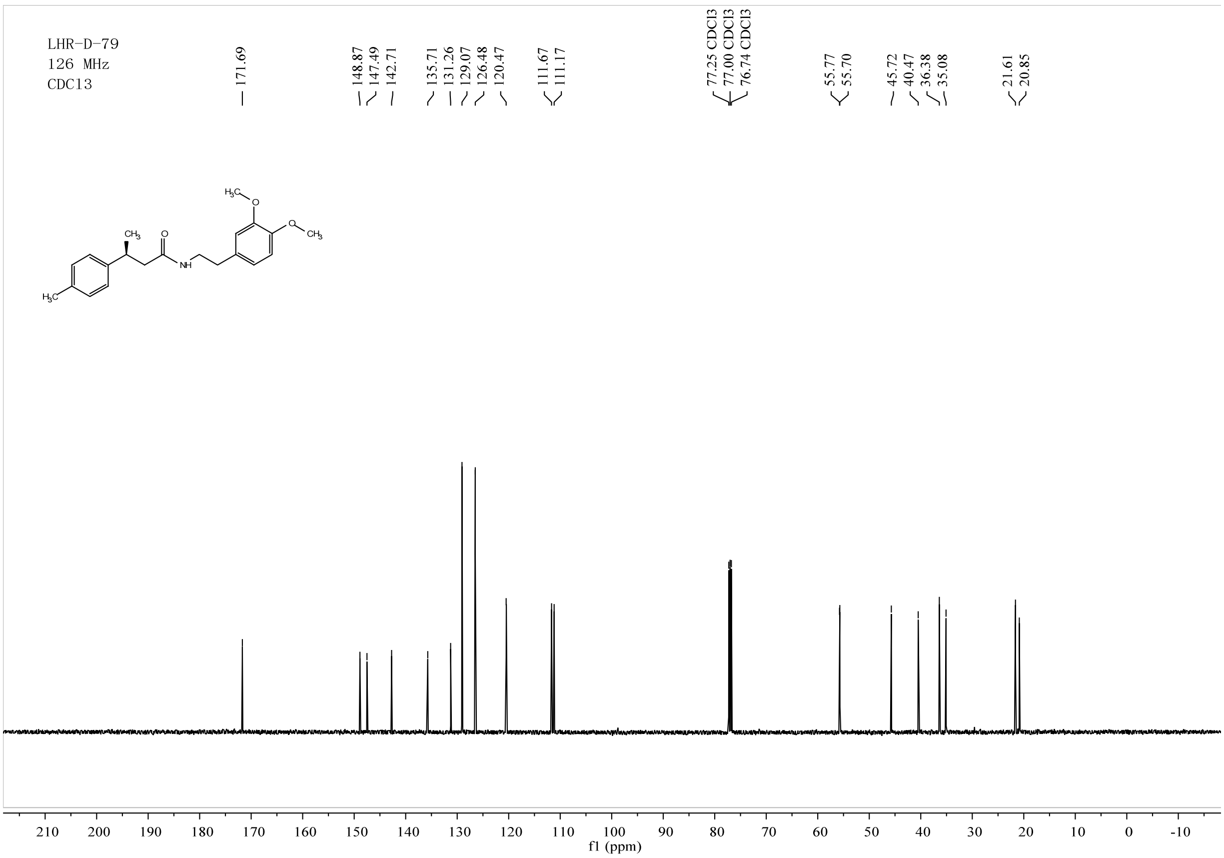
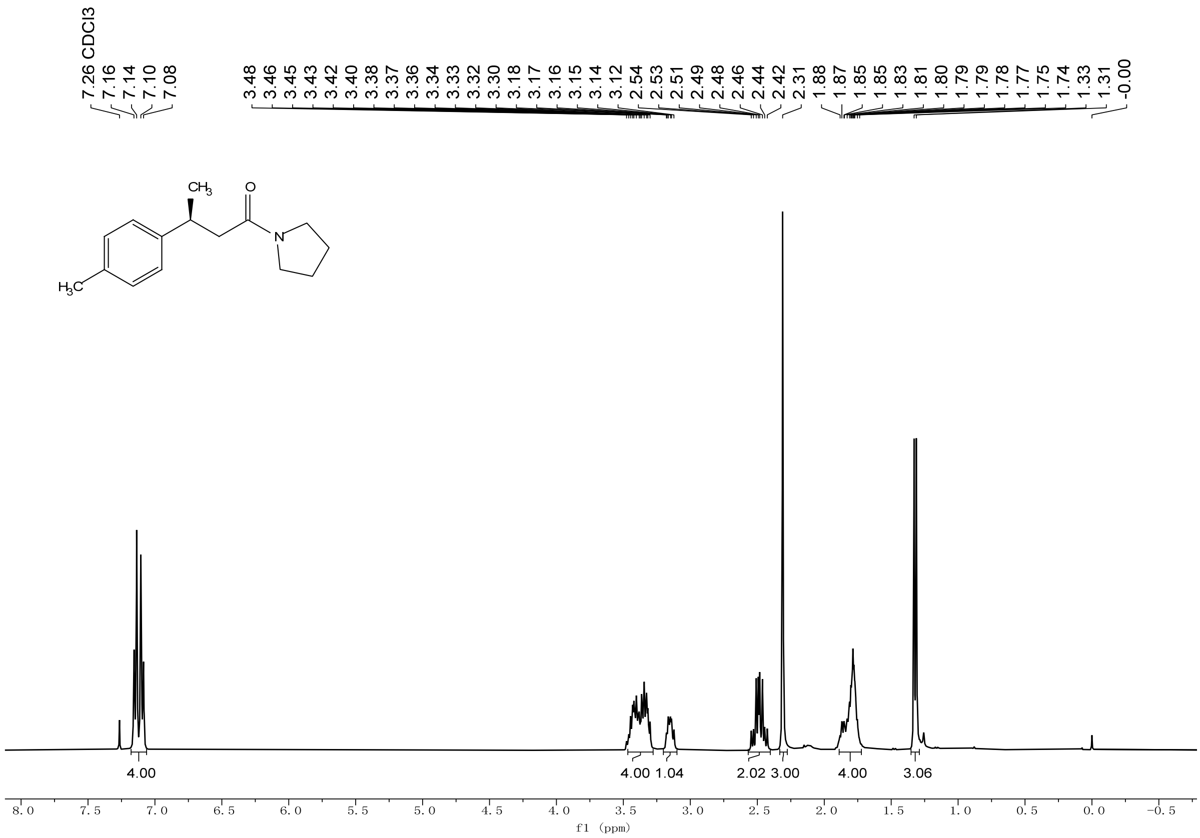


Figure S-42. ^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C NMR (126 MHz, $CDCl_3$) spectra of **3ab**.



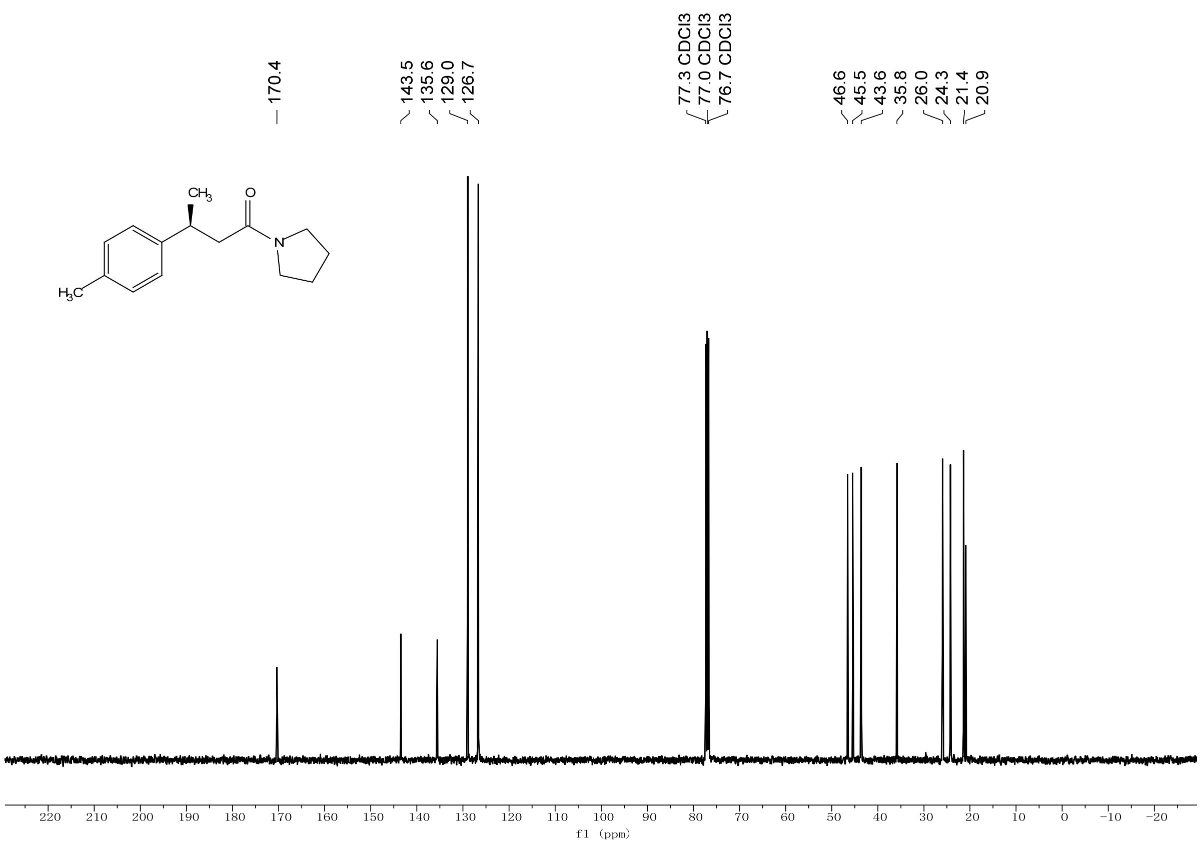
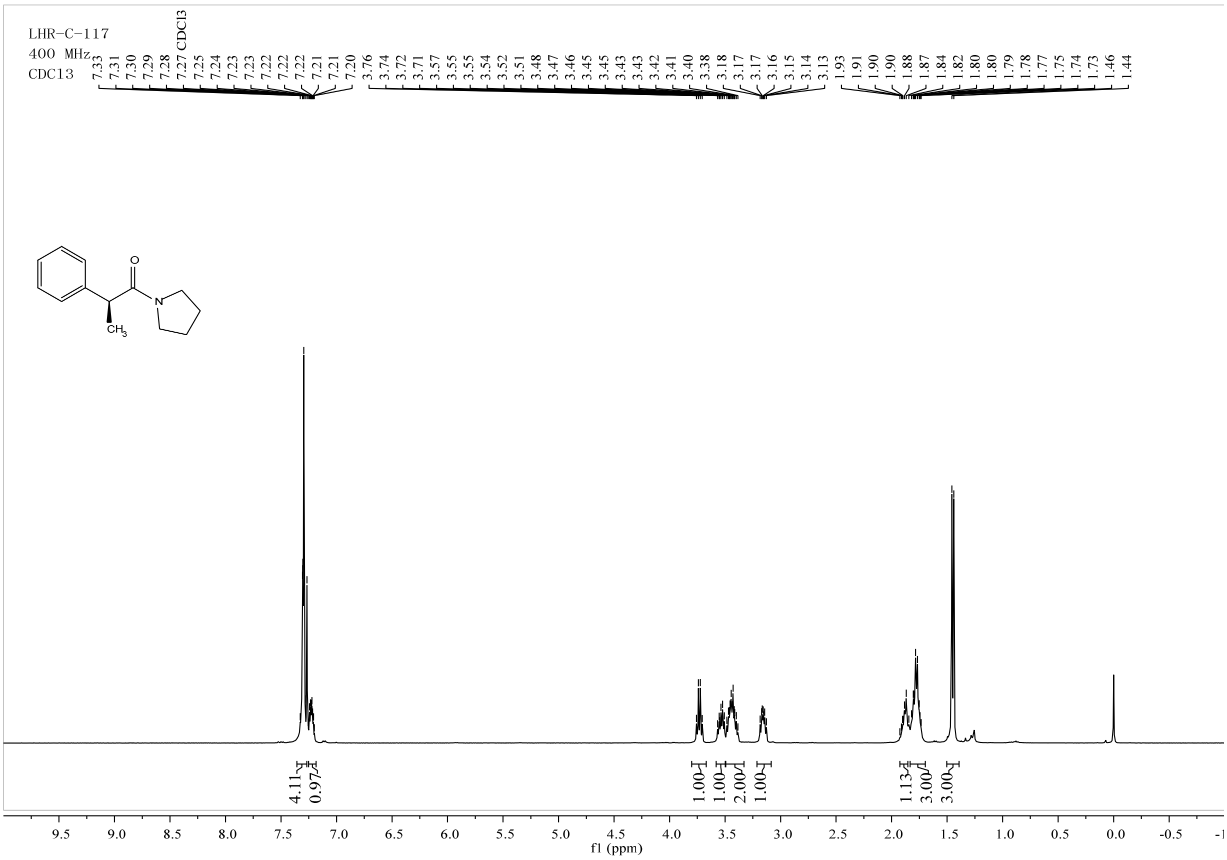


Figure S-43. ^1H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **3ad**.



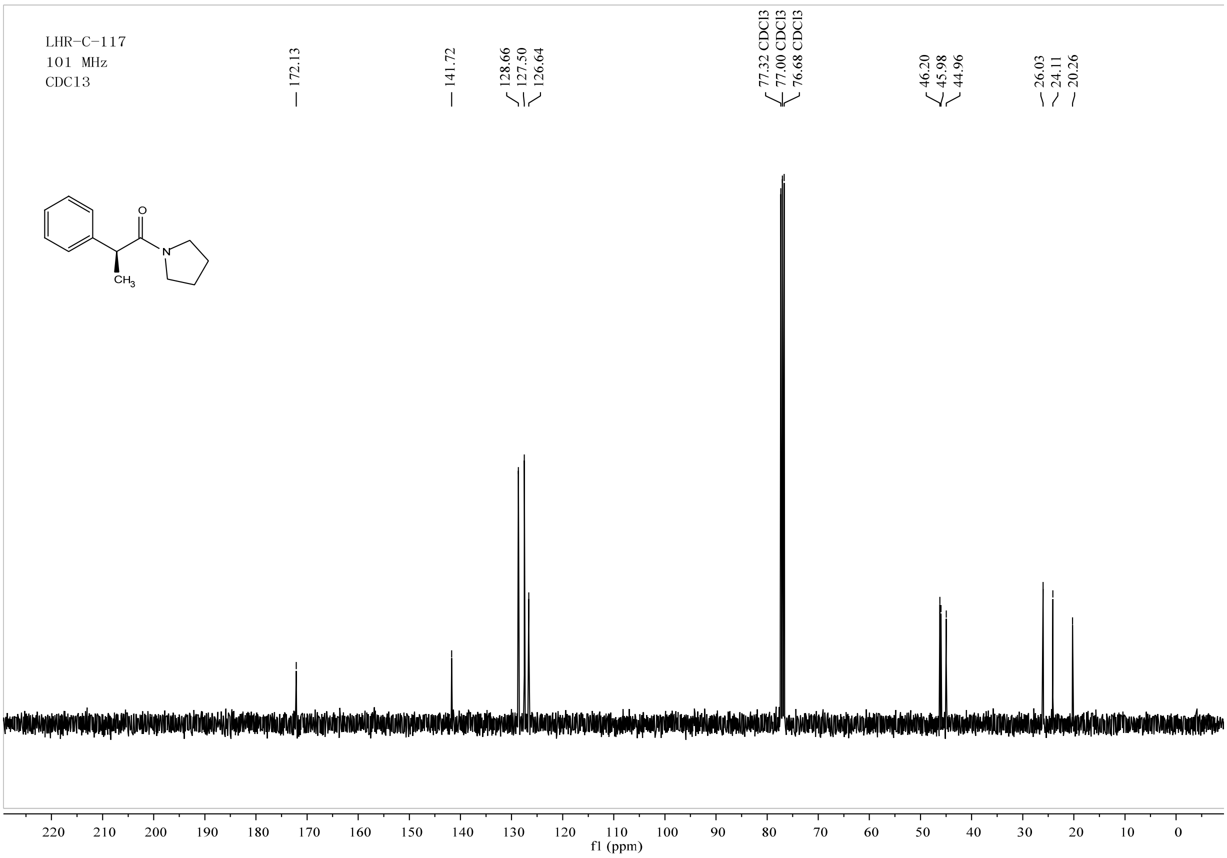
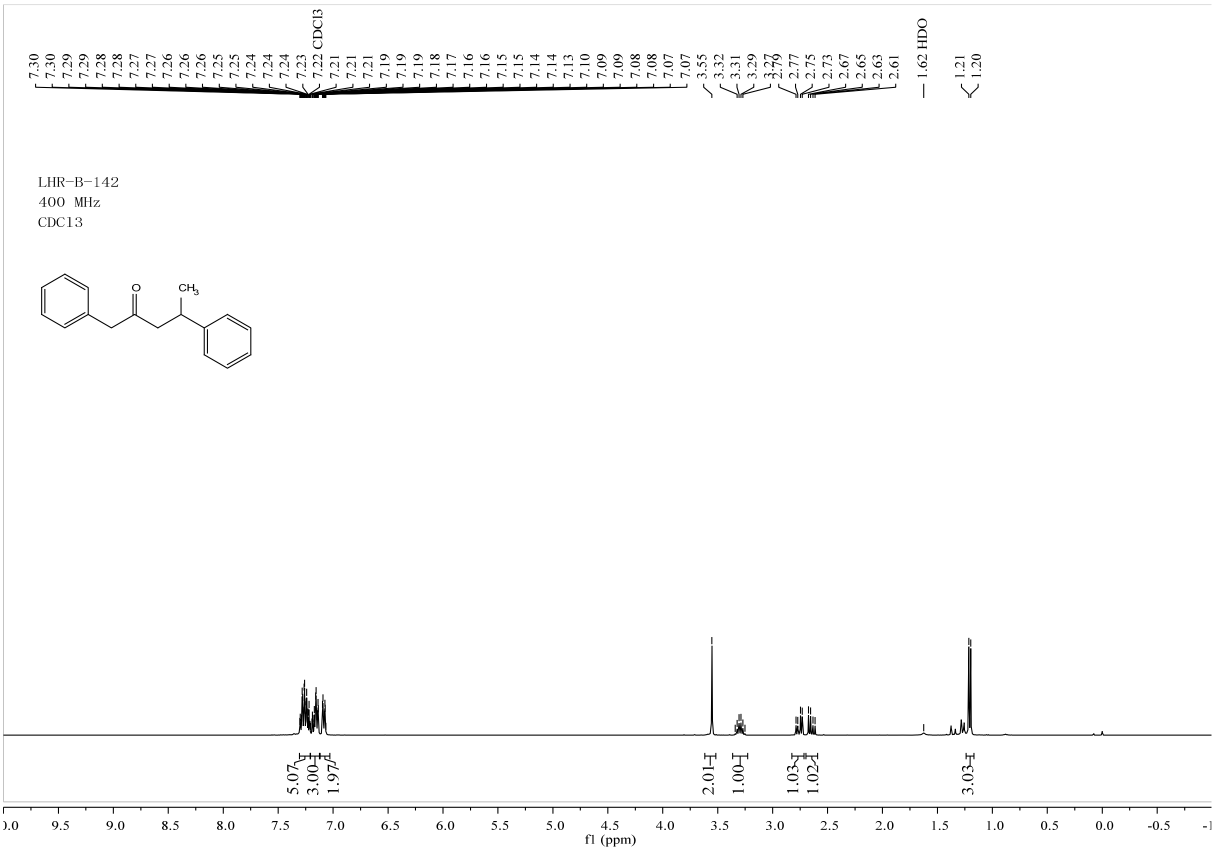


Figure S-44. ^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **3ae**.



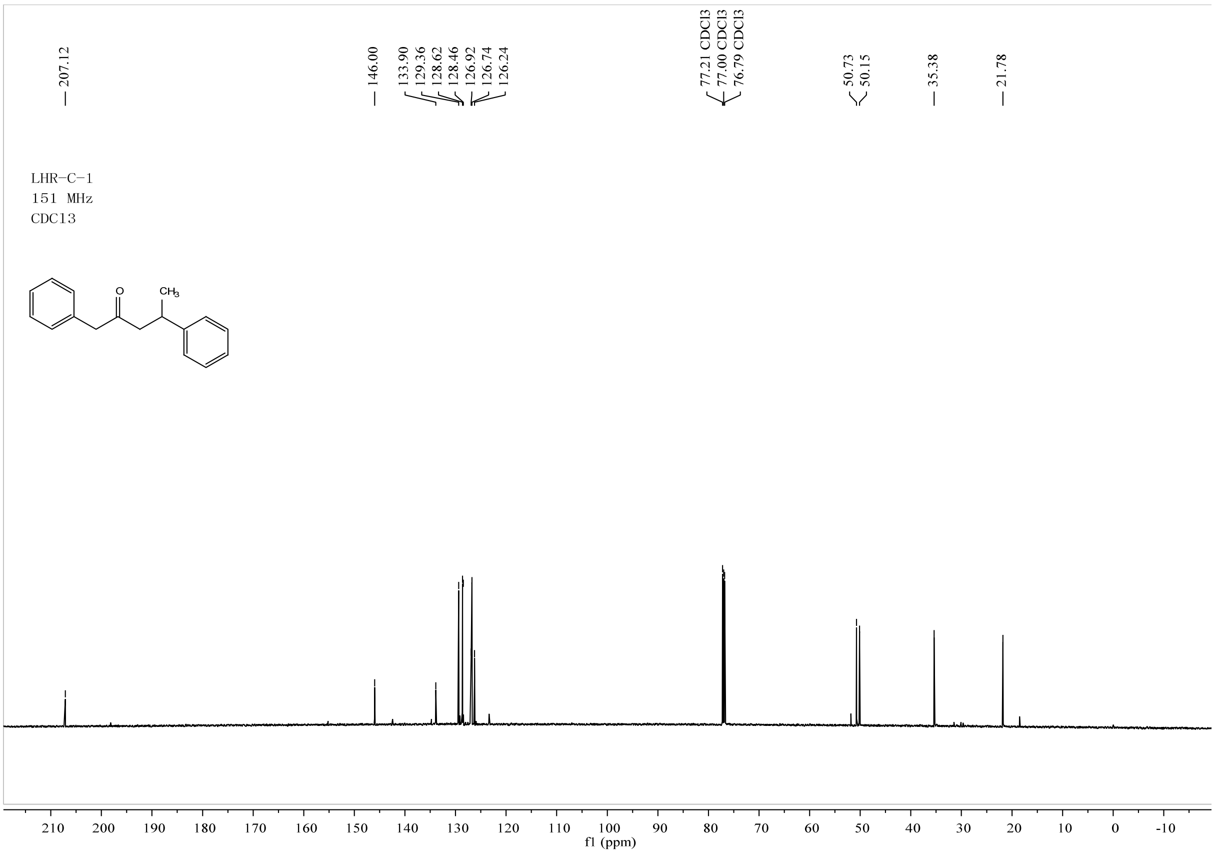
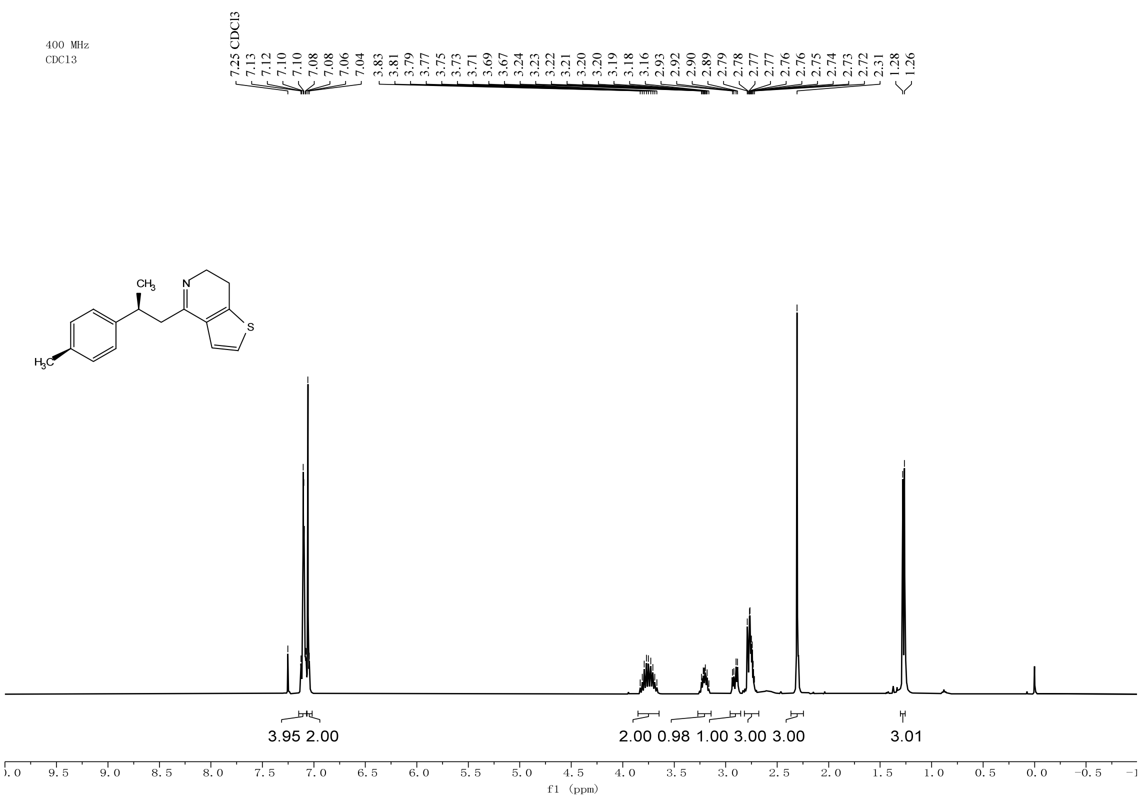


Figure S-45. ^1H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (126 MHz, $CDCl_3$) spectra of **4a**.



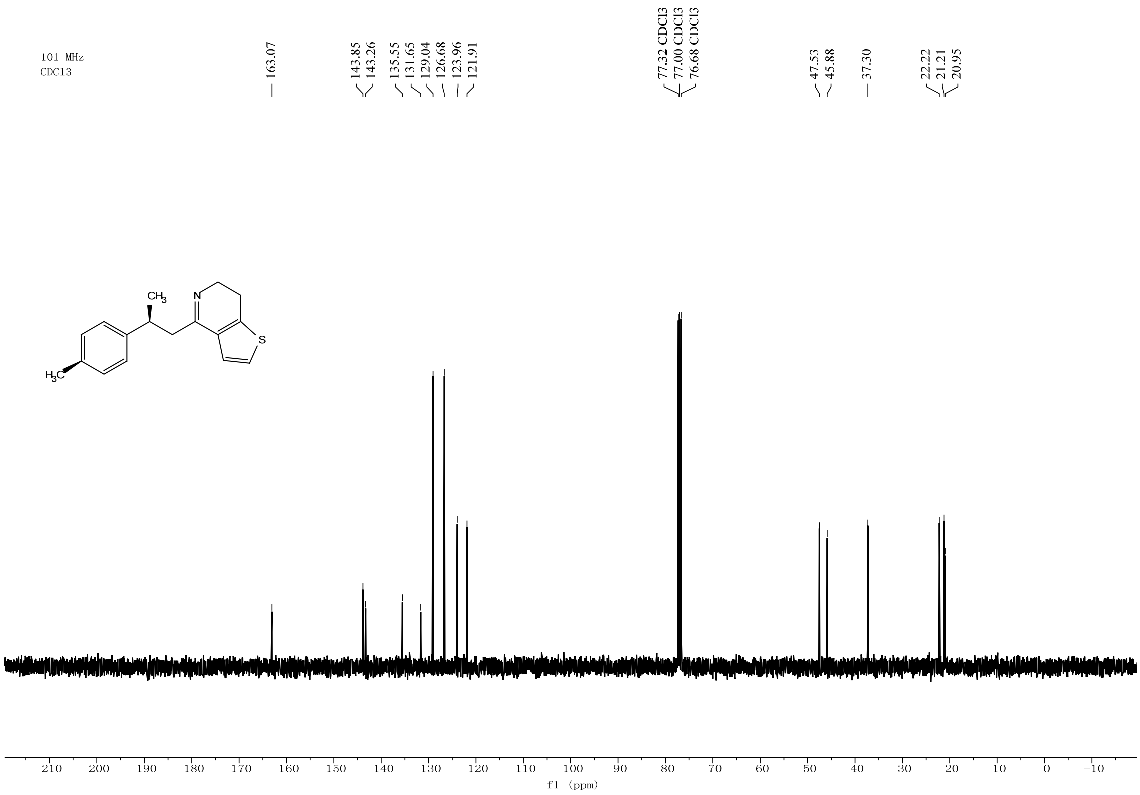
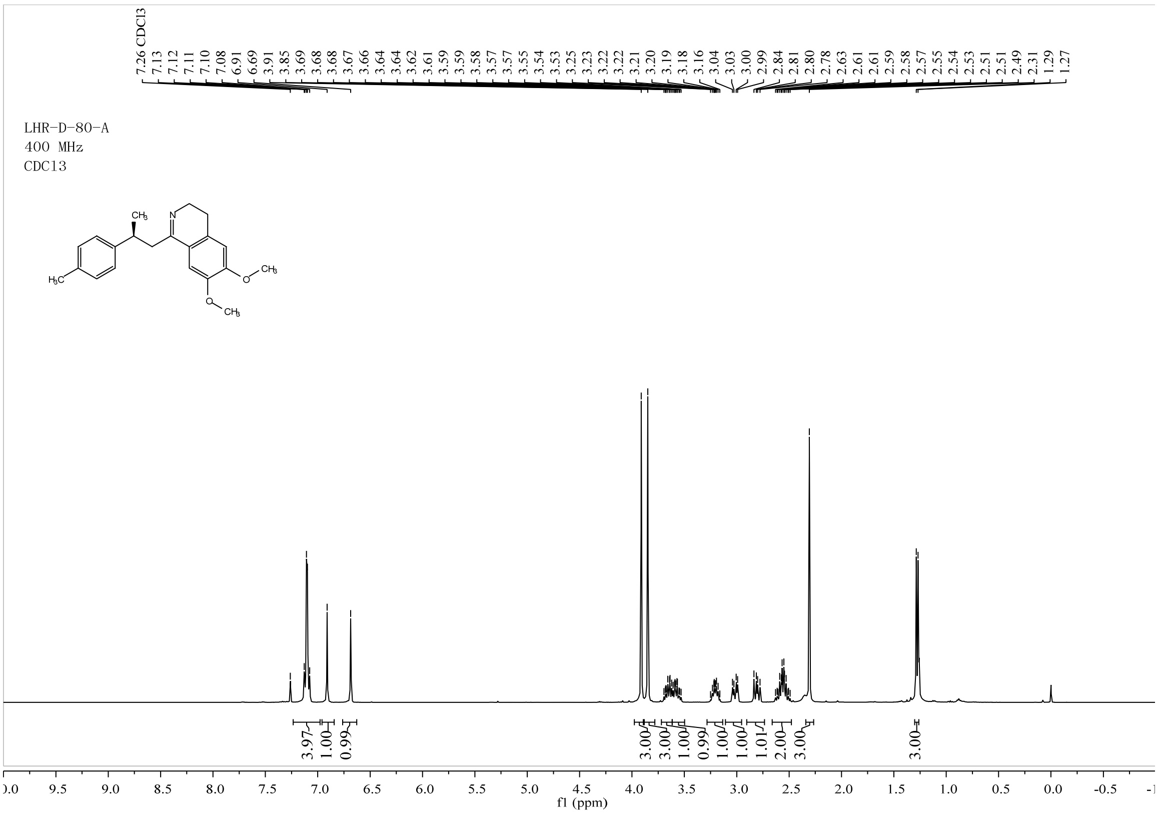


Figure S-46. ^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **4b**.



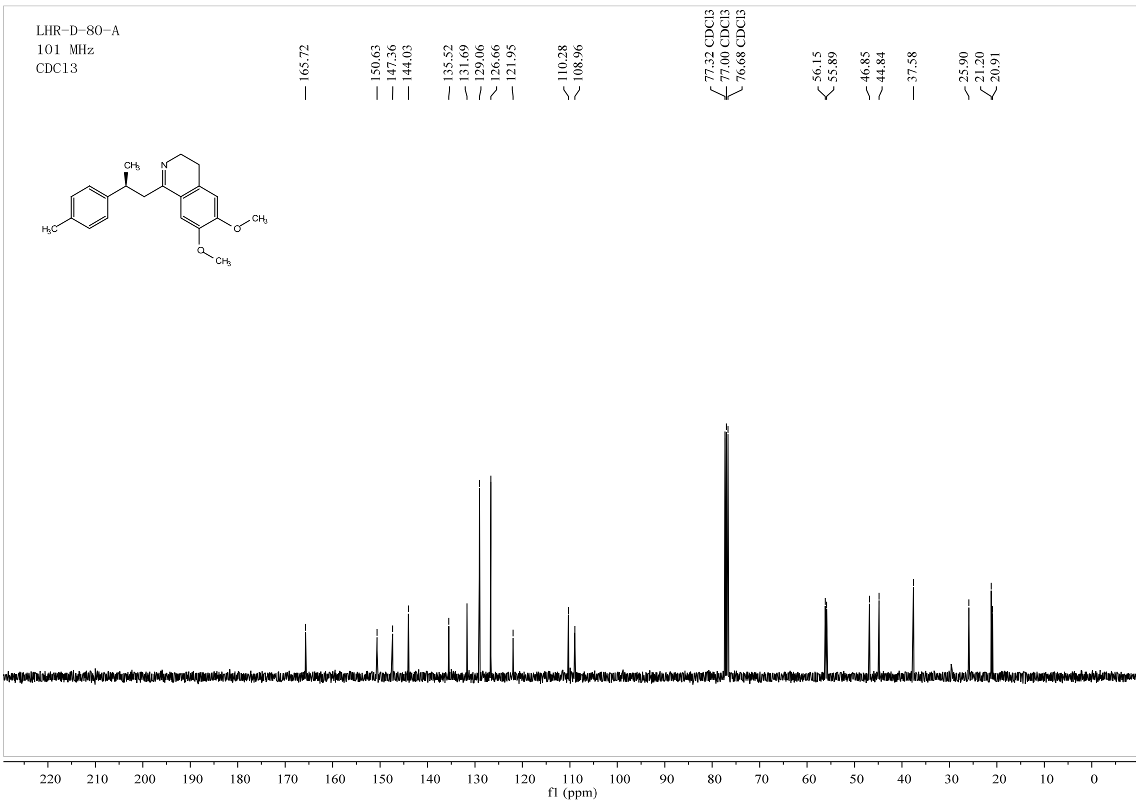
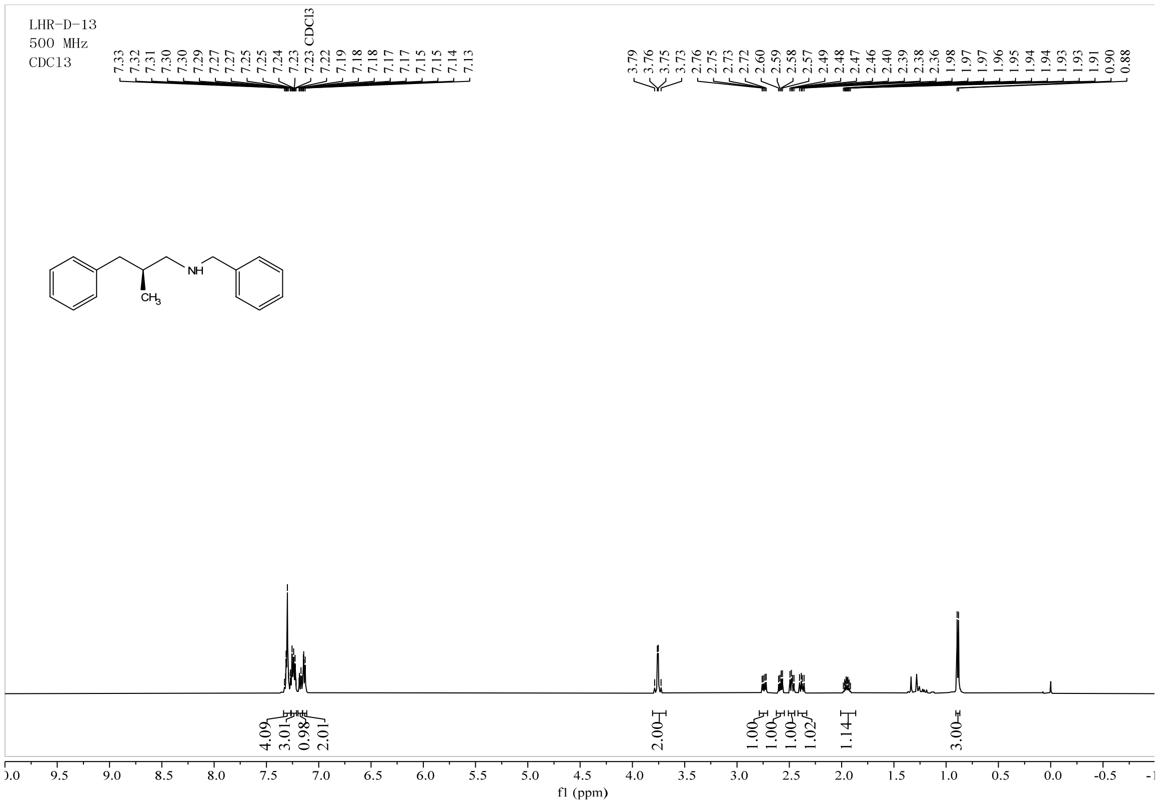


Figure S-47. ^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **4c**.



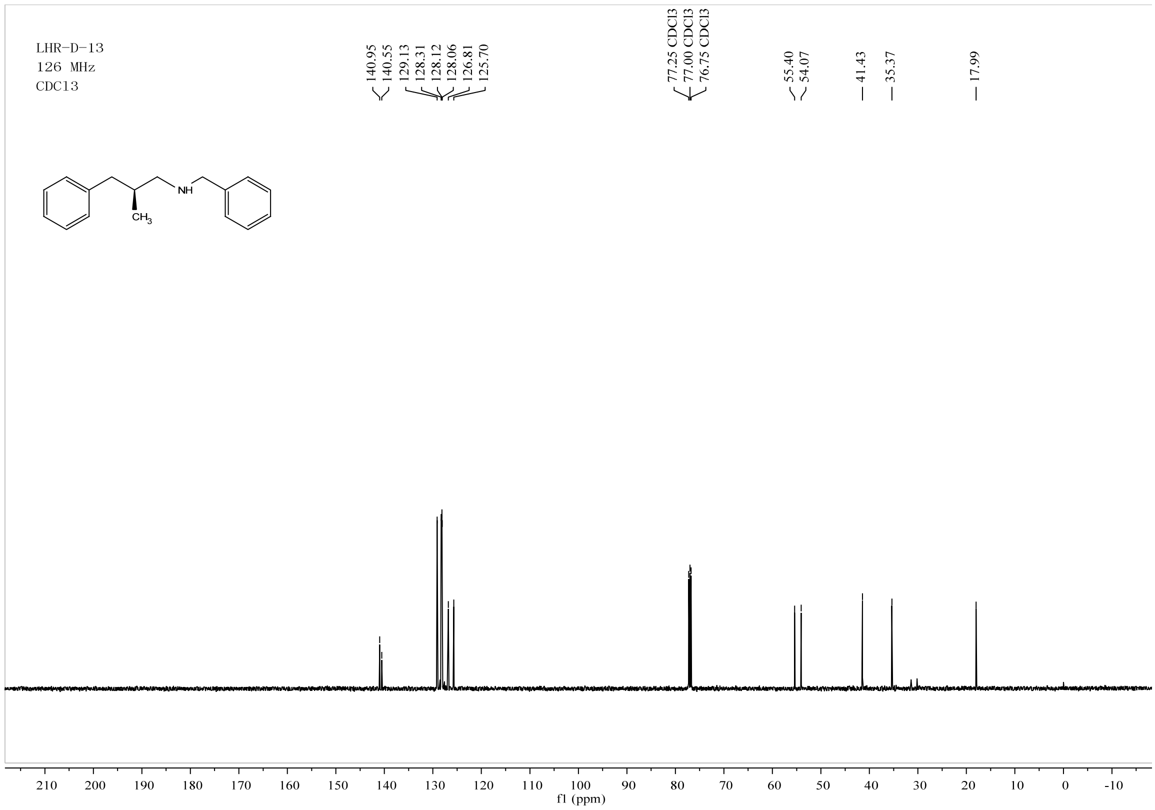
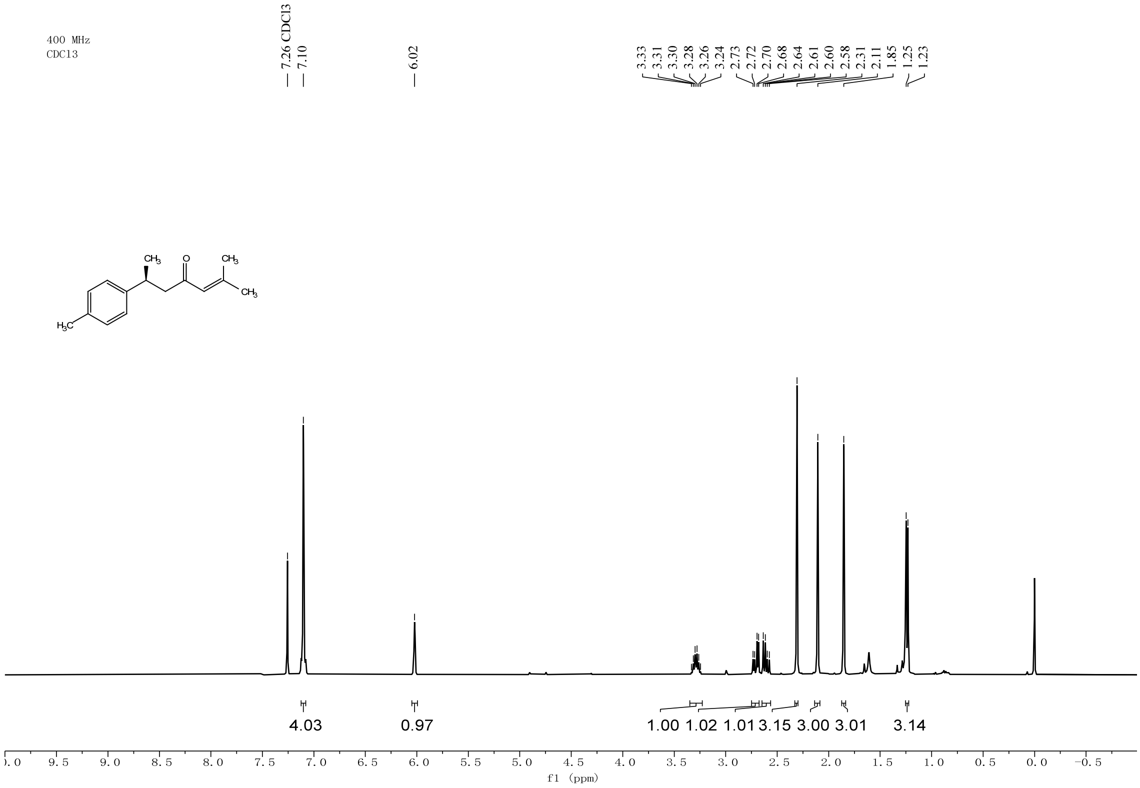


Figure S-48. ^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C NMR (126 MHz, $CDCl_3$) spectra of **4d**.



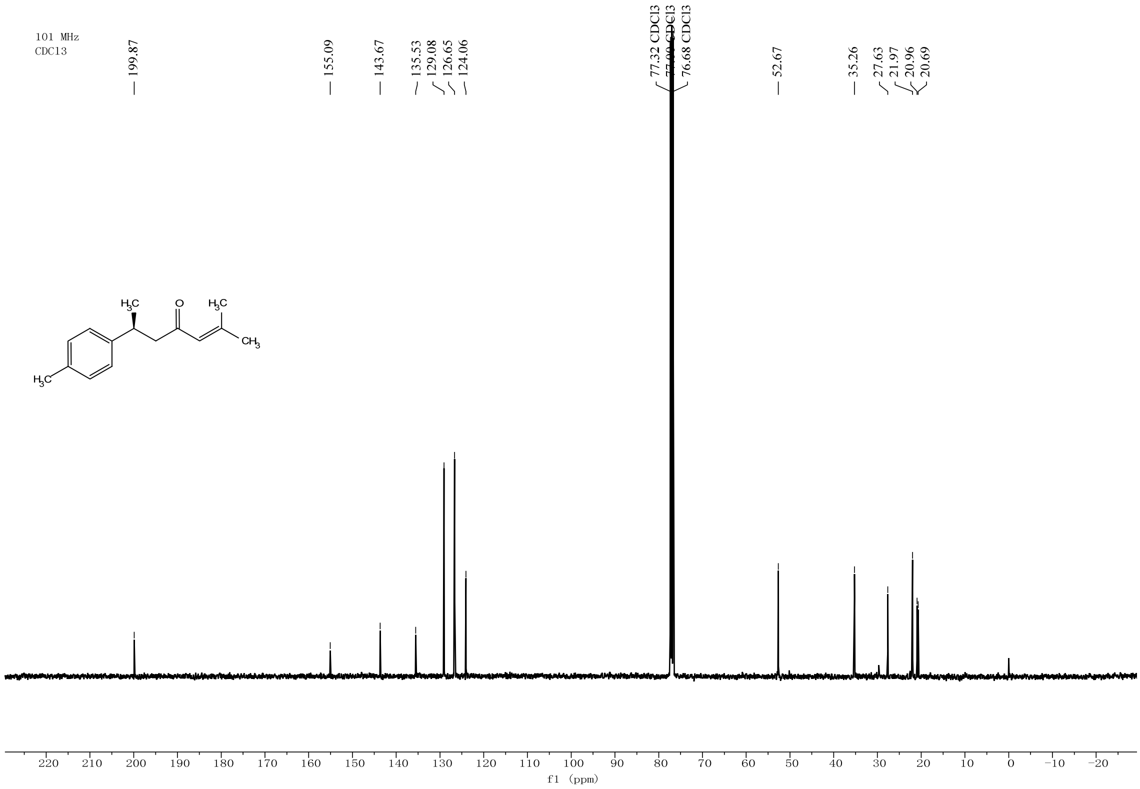
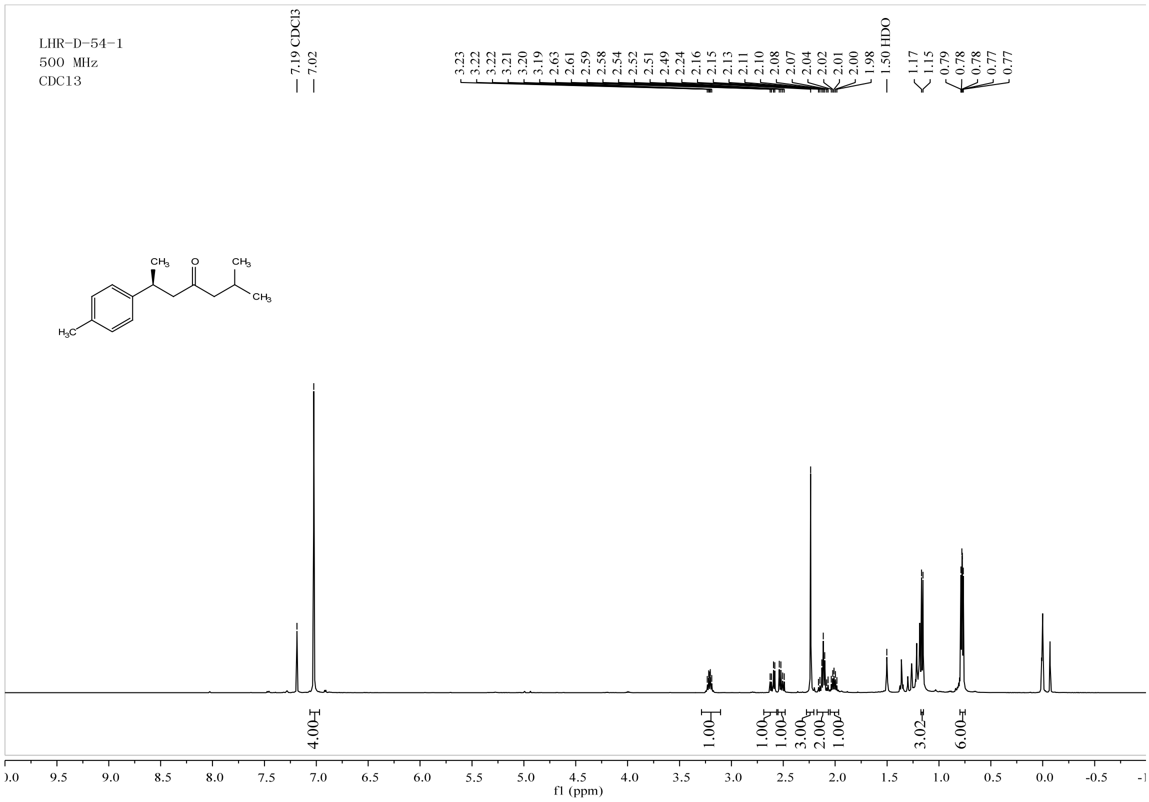


Figure S-49. ^{1}H NMR (400 MHz, $CDCl_3$) and ^{13}C NMR (101 MHz, $CDCl_3$) spectra of **K-1**.



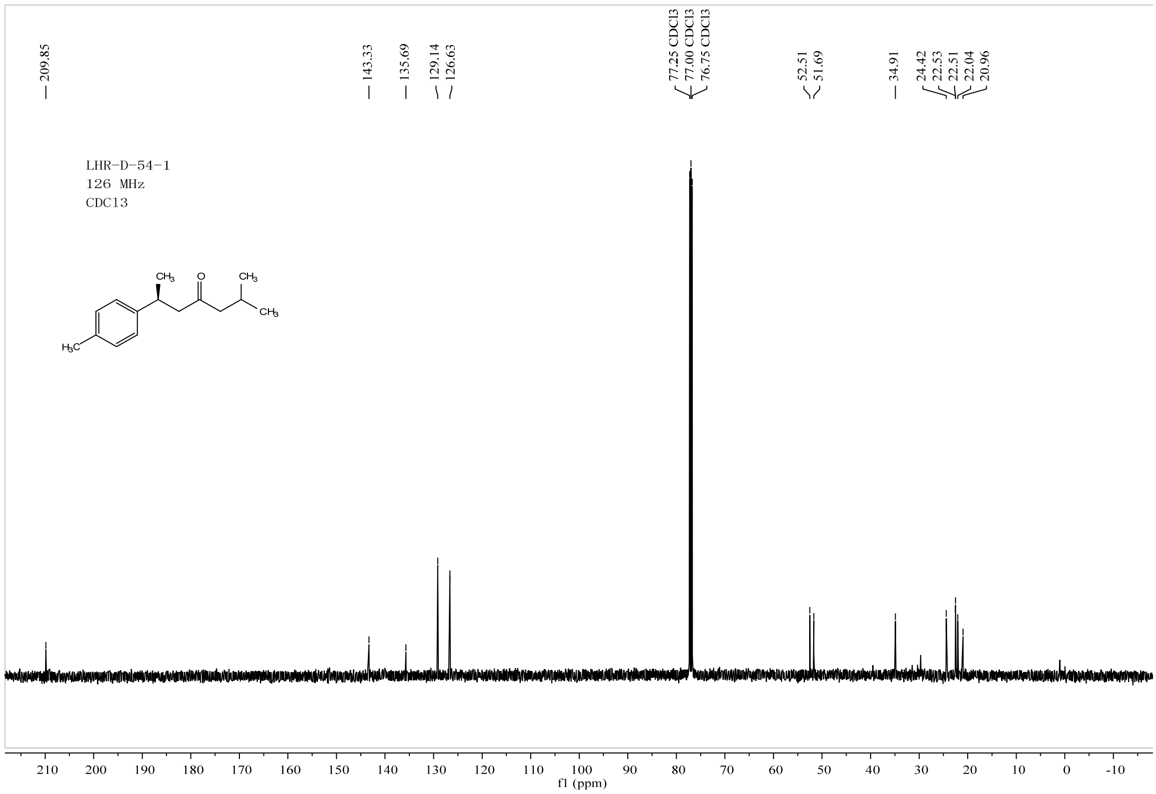
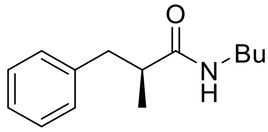


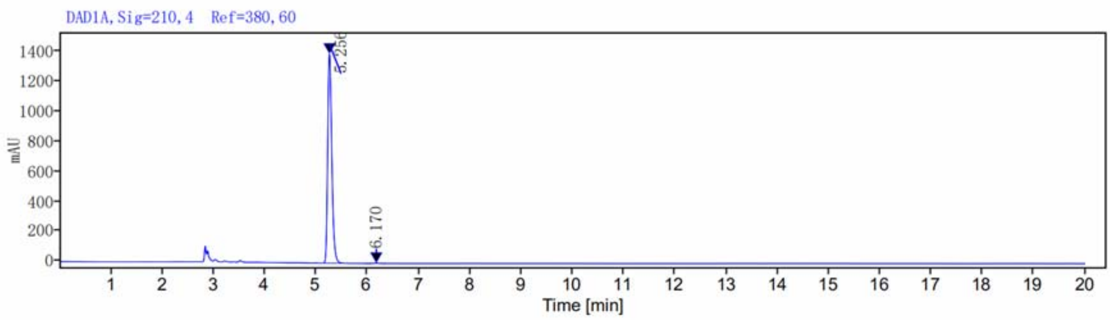
Figure S-50. ^{1}H NMR (500 MHz, $CDCl_3$) and ^{13}C NMR (126 MHz, $CDCl_3$) spectra of **K-2**.

Determination of enantiomeric excess (*ee*) of the products by chiral HPLC



3a, 99% *ee*

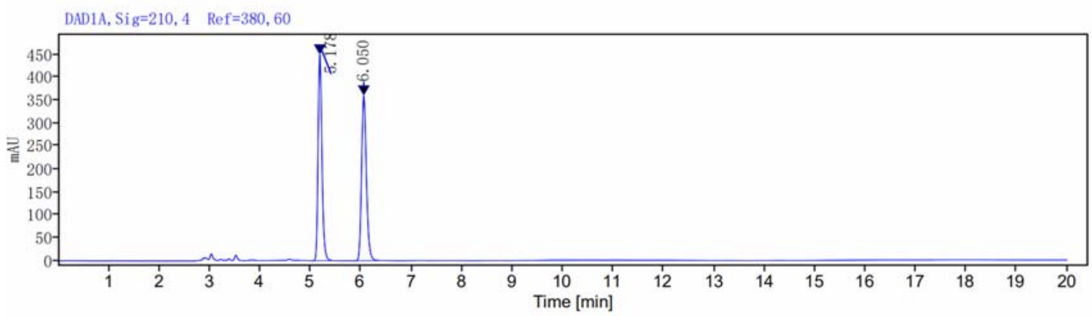
<Chromatogram>



Signal: DAD1A, Sig=210, 4 Ref=380, 60

RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
5.256	MV m	0.72408	7743.28121	1393.79674	99.7202
6.170	MM m	0.40954	21.72783	3.07190	0.2798
		Totals	7765.00905		

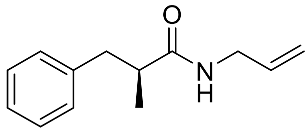
<Chromatogram>



Signal: DAD1A, Sig=210, 4 Ref=380, 60

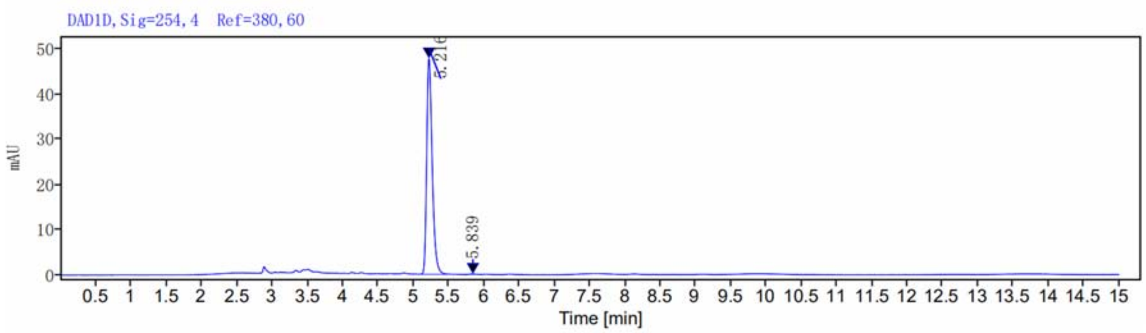
RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
5.178	BV	0.41614	2392.98147	449.65936	49.9164
6.050	BV	0.51080	2400.99479	359.90040	50.0836
		Totals	4793.97626		

Figure S-51. HPLC Data of (*S*)-**3a** (from **2a**) and racemic **3a**



3b, 98% *ee*

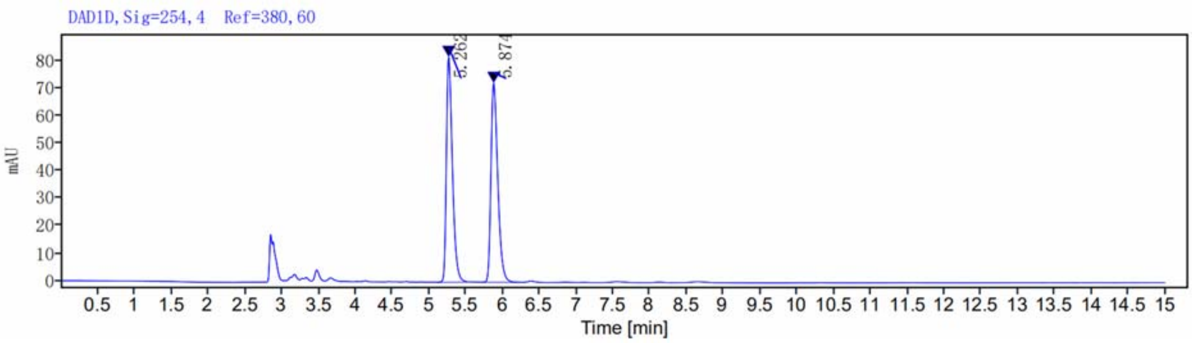
<Chromatogram>



Signal: DAD1D, Sig=254, 4 Ref=380, 60

RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
5.216	VV	0.37352	259.99705	47.76991	99.8221
5.839	MM m	0.18116	0.46342	0.10768	0.1779
		Totals	260.46047		

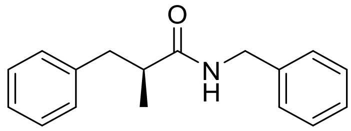
<Chromatogram>



Signal: DAD1D, Sig=254, 4 Ref=380, 60

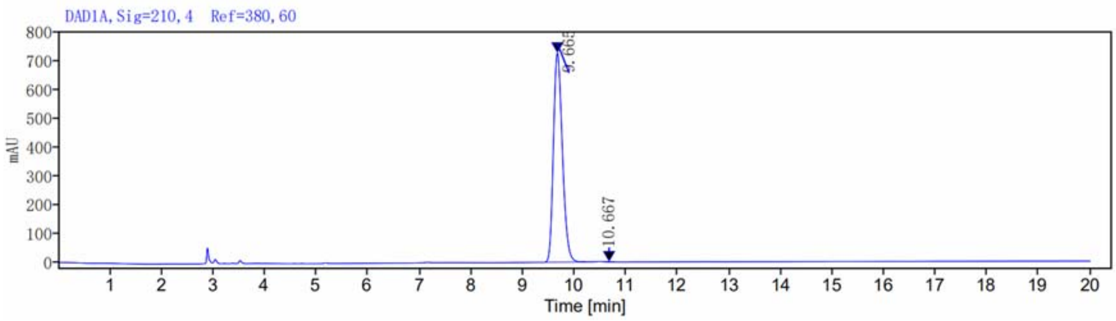
RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
5.262	BV	0.42088	466.69096	81.86246	49.9223
5.874	VV	0.44184	468.14343	72.43735	50.0777
		Totals	934.83440		

Figure S-52. HPLC Data of (*S*)-**3b** (from **2a**) and racemic **3b**



3c, 99% *ee*

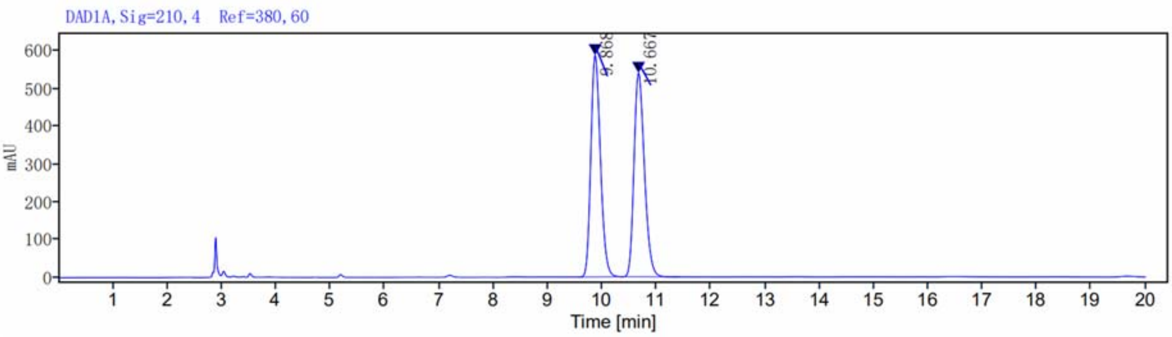
<Chromatogram>



Signal: DAD1A, Sig=210, 4 Ref=380, 60

RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
9.665	BV	0.83445	8820.10214	730.94057	99.9740
10.667	MM n	0.30033	2.29124	0.37485	0.0260
		Totals	8822.39337		

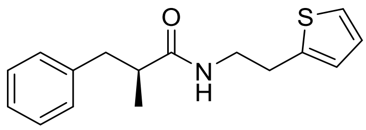
<Chromatogram>



Signal: DAD1A, Sig=210, 4 Ref=380, 60

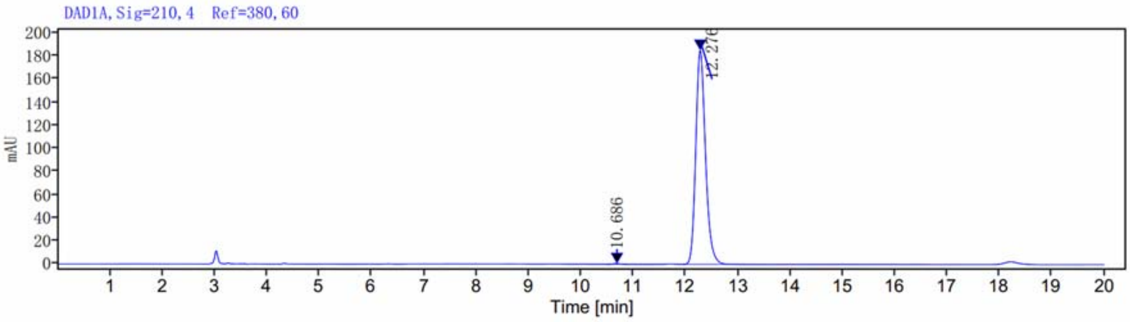
RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
9.868	VB	0.79105	7207.21482	587.13319	50.0265
10.667	BV	1.04987	7199.58892	539.74531	49.9735
		Totals	14406.80374		

Figure S-53. HPLC Data of (*S*)-**3c** (from **2a**) and racemic **3c**



3d, 99% *ee*

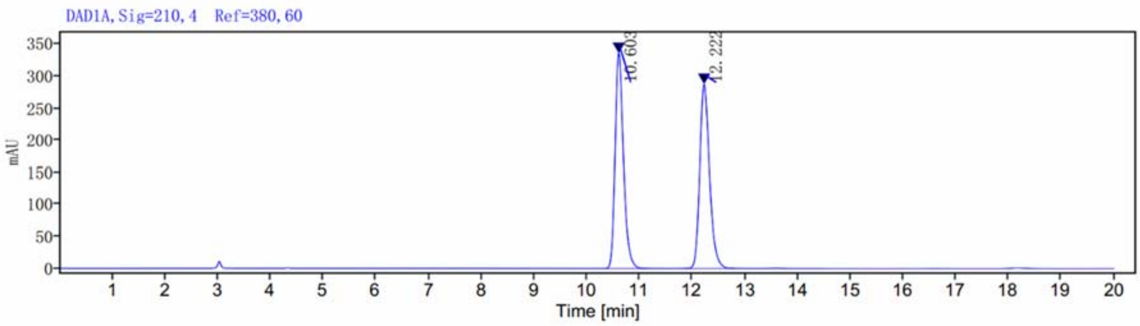
<Chromatogram>



Signal: DAD1A, Sig=210, 4 Ref=380, 60

RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
10.686	MM m	0.37885	5.38661	0.53651	0.2251
12.276	BV	0.84790	2387.53486	185.25042	99.7749
		Totals	2392.92147		

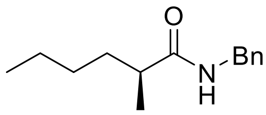
<Chromatogram>



Signal: DAD1A, Sig=210, 4 Ref=380, 60

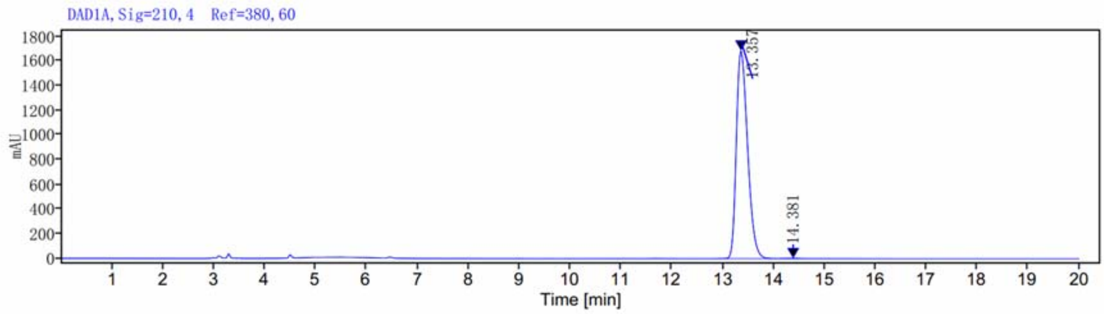
RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
10.603	VV	0.80967	3717.33852	336.63845	49.9472
12.222	VV	0.98372	3725.19125	287.98791	50.0528
		Totals	7442.52976		

Figure S-54. HPLC Data of (*S*)-**3d** (from **2a**) and racemic **3d**



3e, 99% *ee*

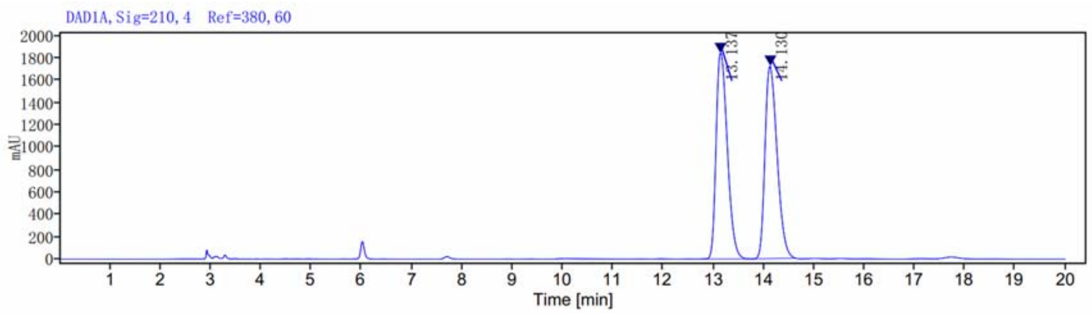
<Chromatogram>



Signal: DAD1A, Sig=210, 4 Ref=380, 60

RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
13.357	VV	0.90026	25845.36840	1687.31356	99.9785
14.381	MM m	0.36955	5.55029	0.67512	0.0215
		Totals	25850.91868		

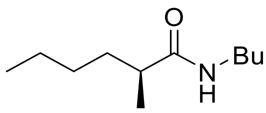
<Chromatogram>



Signal: DAD1A, Sig=210, 4 Ref=380, 60

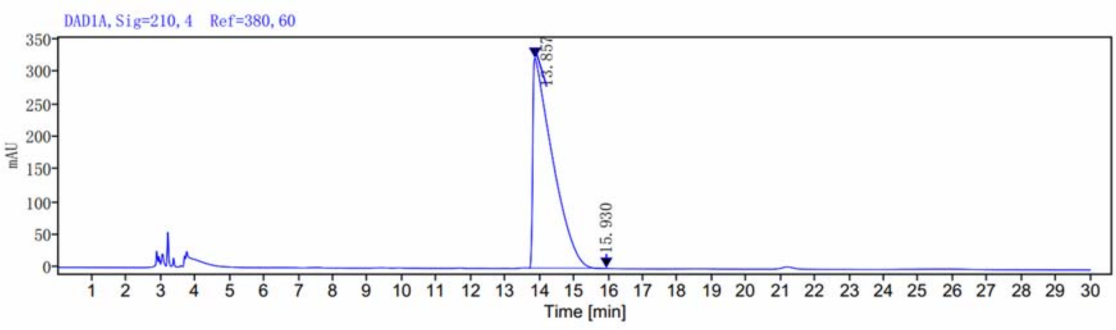
RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
13.137	MM m	0.98376	28688.95822	1845.78374	49.8798
14.130	MM m	0.86958	28827.20550	1728.17045	50.1202
		Totals	57516.16372		

Figure S-55. HPLC Data of (*S*)-**3e** (from **2b**) and racemic **3e**



3f, 99% *ee*

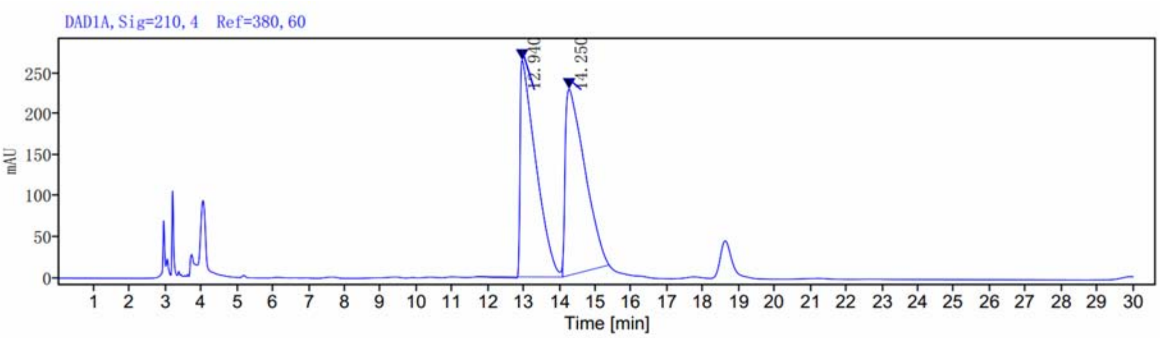
<Chromatogram>



Signal: DAD1A, Sig=210, 4 Ref=380, 60

RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
13.857	BM m	1.91654	12801.52540	322.25501	99.9407
15.930	MM n	0.78850	7.59839	0.40060	0.0593
		Totals	12809.12379		

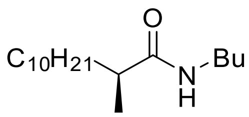
<Chromatogram>



Signal: DAD1A, Sig=210, 4 Ref=380, 60

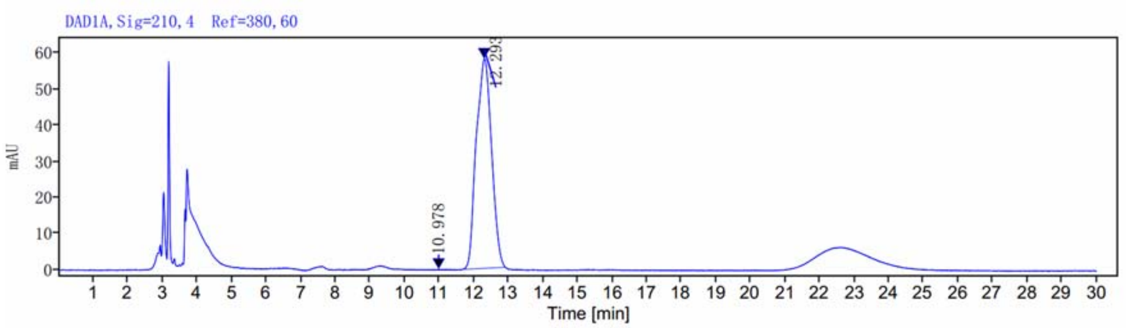
RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
12.940	MM m	2.38103	7969.90326	264.35727	48.1868
14.250	MM m	1.30084	8569.71227	227.15375	51.8132
		Totals	16539.61553		

Figure S-56. HPLC Data of (*S*)-**3f** (from **2b**) and racemic **3f**



3g, 99% *ee*

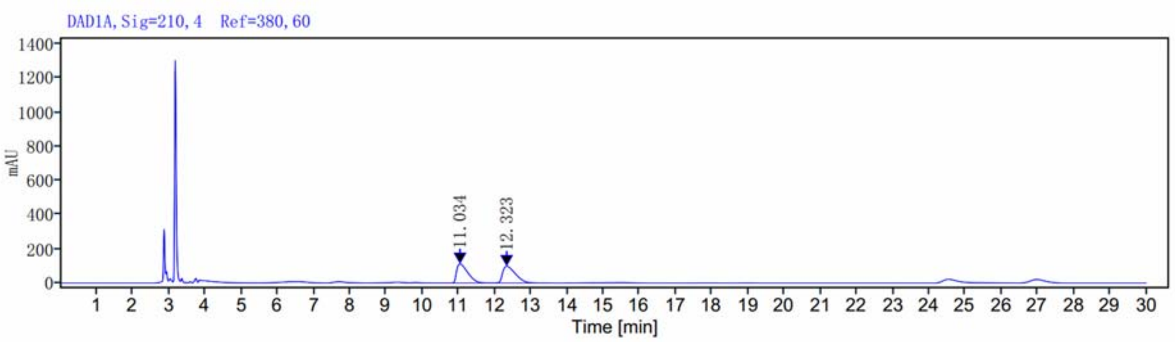
<Chromatogram>



Signal: DAD1A, Sig=210, 4 Ref=380, 60

RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
10.978	MM m	0.83576	2.21971	0.23422	0.1244
12.293	VM m	1.17024	1781.80580	58.06325	99.8756
		Totals	1784.02551		

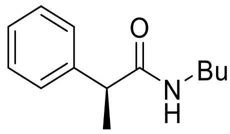
<Chromatogram>



Signal: DAD1A, Sig=210, 4 Ref=380, 60

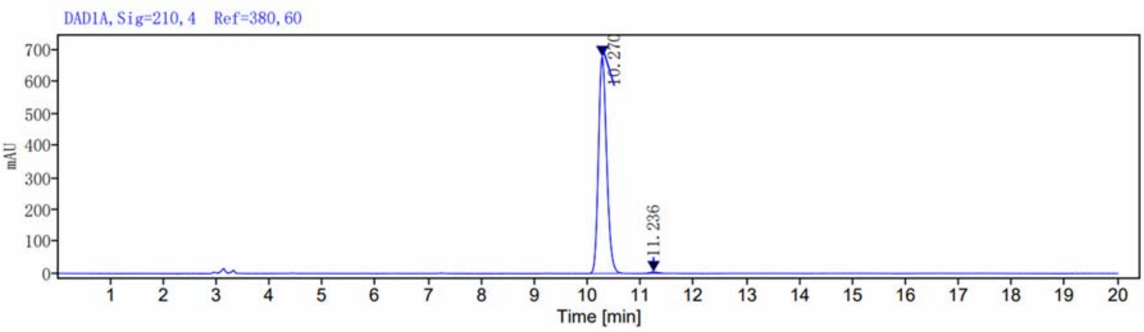
RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
11.034	BV	0.92804	2357.26133	111.61976	49.9018
12.323	VV	1.10855	2366.53768	97.41087	50.0982
		Totals	4723.79901		

Figure S-57. HPLC Data of (*S*)-**3g** (from **2c**) and racemic **3g**



3h, 98% *ee*

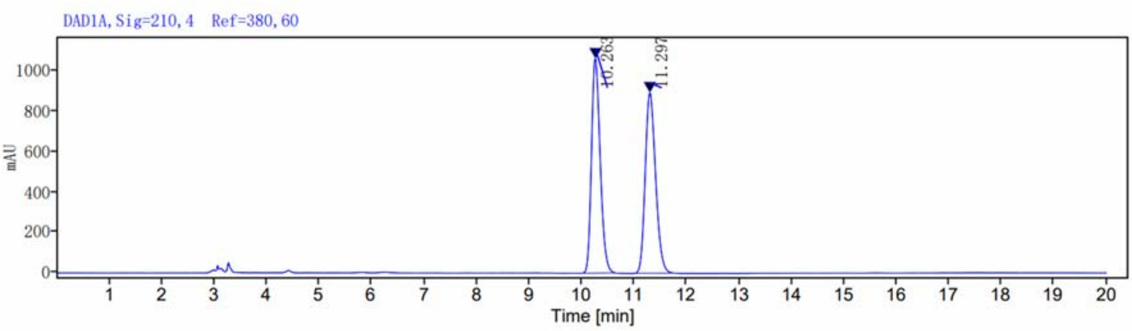
<Chromatogram>



Signal: DAD1A, Sig=210, 4 Ref=380, 60

RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
10.270	VV	0.54225	7312.73319	680.20639	99.1837
11.236	MM m	0.44344	60.18257	5.32873	0.8163
		Totals	7372.91575		

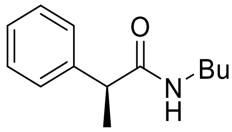
<Chromatogram>



Signal: DAD1A, Sig=210, 4 Ref=380, 60

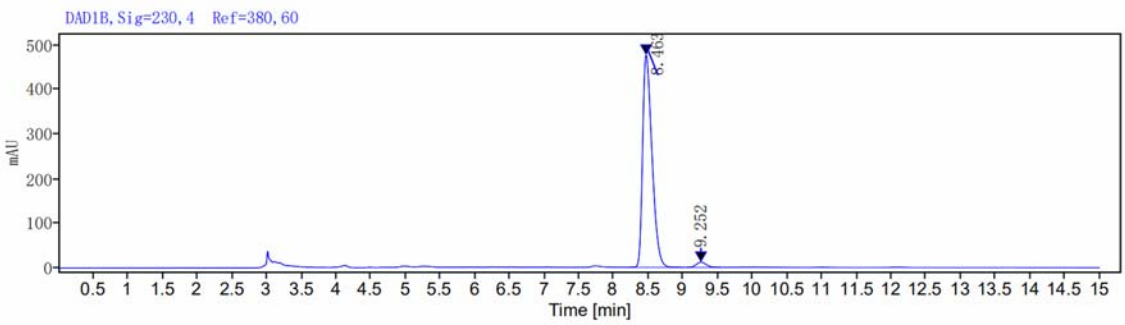
RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
10.263	BV m	0.62168	12168.43032	1056.30095	49.9347
11.297	BV m	0.70275	12200.26900	889.65273	50.0653
		Totals	24368.69932		

Figure S-58. HPLC Data of (*S*)-**3h** (from **2d**) and racemic **3h**



3h, 95% *ee*

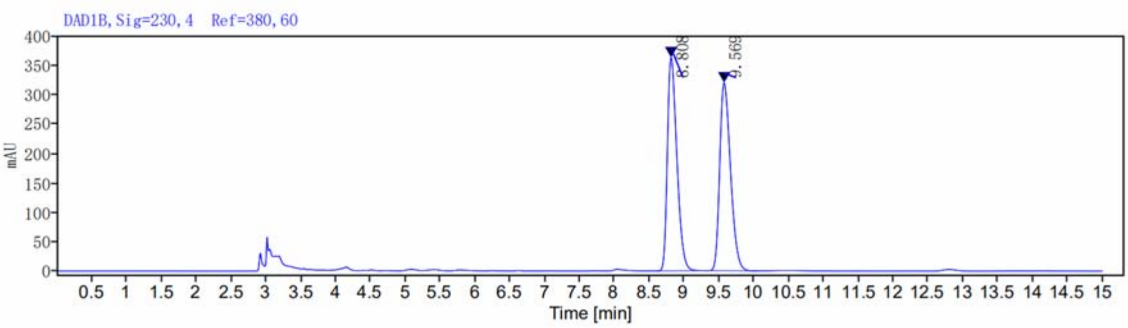
<Chromatogram>



Signal: DAD1B, Sig=230, 4 Ref=380, 60

RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
8.463	VV	0.76115	4463.48544	476.34449	97.4538
9.252	VV	0.48983	116.61908	11.60847	2.5462
		Totals	4580.10451		

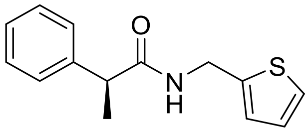
<Chromatogram>



Signal: DAD1B, Sig=230, 4 Ref=380, 60

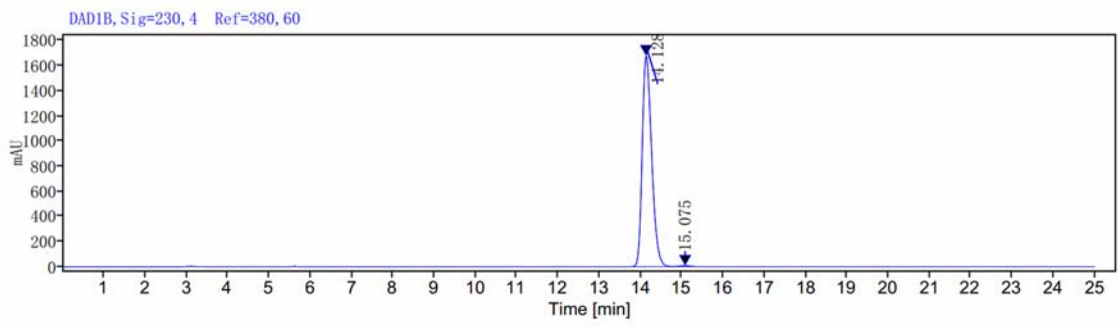
RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
8.808	BB	0.73000	3463.42117	365.52884	49.9960
9.569	BB	0.70487	3463.96981	321.97130	50.0040
		Totals	6927.39098		

Figure S-59. HPLC Data of (*S*)-**3h** (from **2g**) and racemic **3h**



3i, 99% *ee*

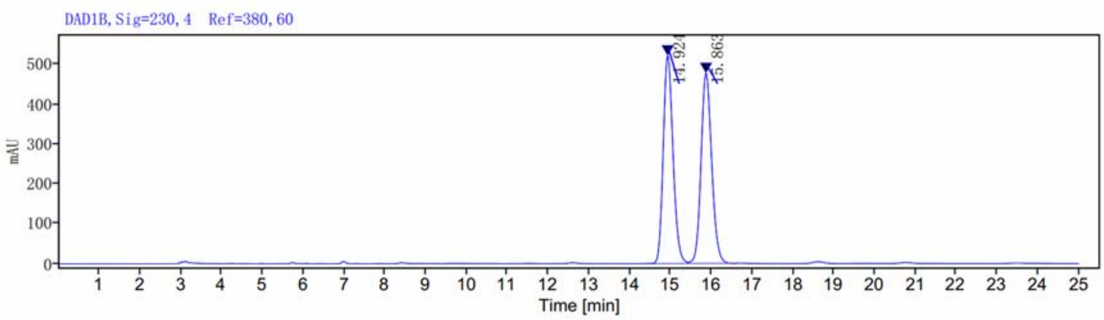
<Chromatogram>



Signal: DAD1B, Sig=230, 4 Ref=380, 60

RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
14.128	VM m	0.95945	26696.85491	1680.44643	99.5228
15.075	MM m	0.73907	128.01348	8.54419	0.4772
		Totals	26824.86839		

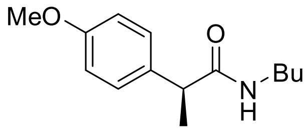
<Chromatogram>



Signal: DAD1B, Sig=230, 4 Ref=380, 60

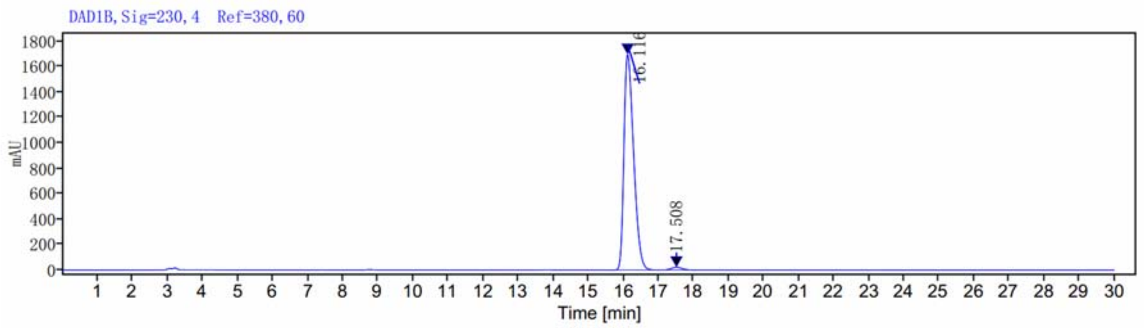
RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
14.924	VV	0.85821	8680.61103	521.15970	50.1360
15.863	VV	0.96463	8633.50568	476.90723	49.8640
		Totals	17314.11671		

Figure S-60. HPLC Data of (*S*)-**3i** (from **2d**) and racemic **3i**



3j, 97% *ee*

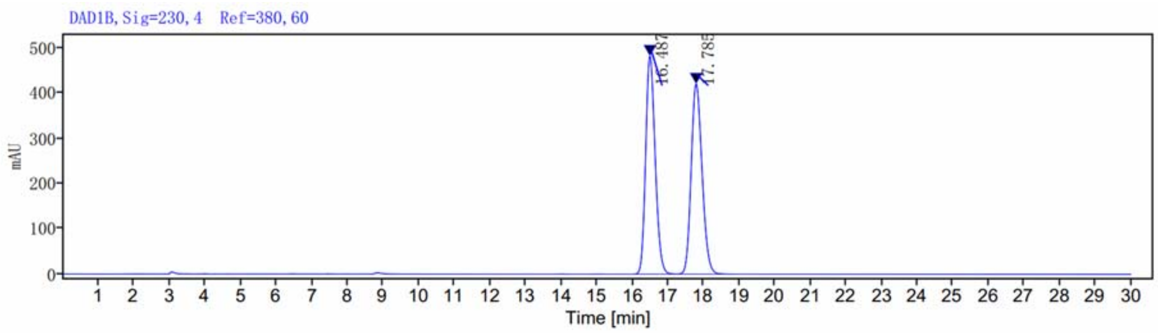
<Chromatogram>



Signal: DAD1B, Sig=230, 4 Ref=380, 60

RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
16.116	VV	1.40667	32531.38208	1695.76849	98.5642
17.508	VV	0.98995	473.88330	22.07644	1.4358
		Totals	33005.26538		

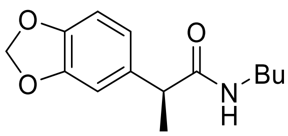
<Chromatogram>



Signal: DAD1B, Sig=230, 4 Ref=380, 60

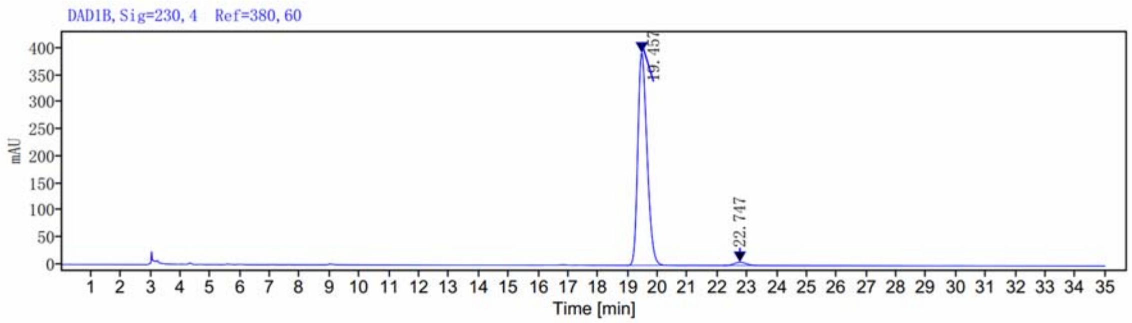
RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
16.487	VV	1.14224	8944.11694	482.78844	49.9870
17.785	BV	1.34458	8948.75425	421.05607	50.0130
		Totals	17892.87119		

Figure S-61. HPLC Data of (*S*)-**3j** (from **2e**) and racemic **3j**



3k, 96% *ee*

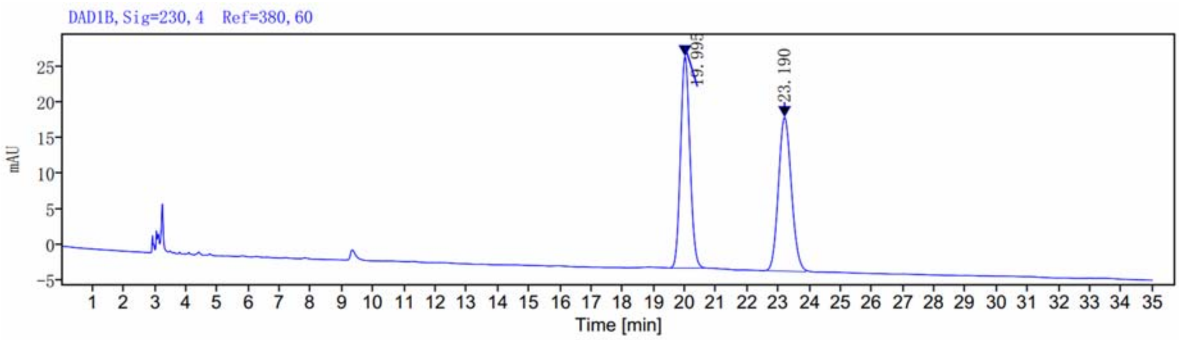
<Chromatogram>



Signal: DAD1B, Sig=230, 4 Ref=380, 60

RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
19.457	VV	1.35307	8469.37349	393.68731	98.0449
22.747	VV	1.07922	168.88511	5.96738	1.9551
		Totals	8638.25860		

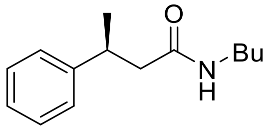
<Chromatogram>



Signal: DAD1B, Sig=230, 4 Ref=380, 60

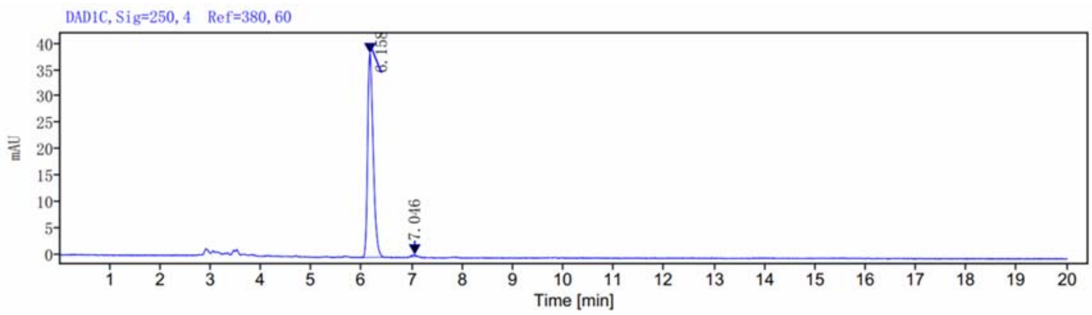
RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
19.995	VV	1.06206	626.10826	29.79597	50.0015
23.190	VV	1.23833	626.07109	21.63212	49.9985
		Totals	1252.17935		

Figure S-62. HPLC Data of (*S*)-**3k** (from **2f**) and racemic **3k**



3s, 99% *ee*

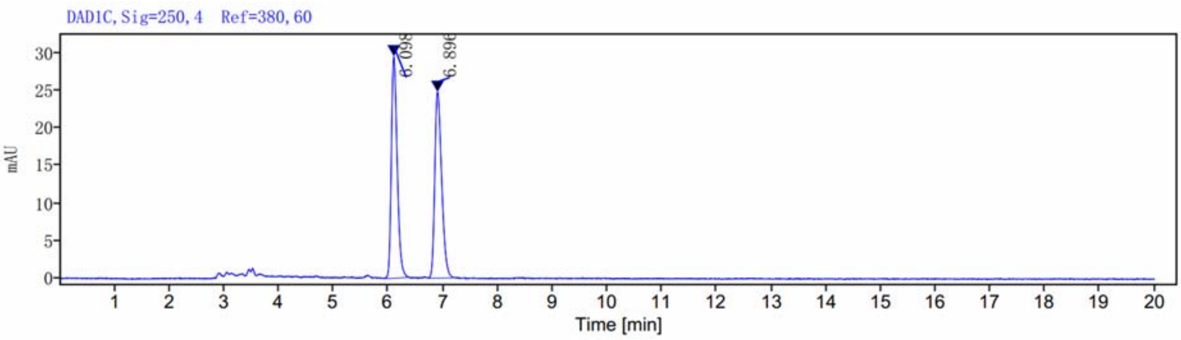
<Chromatogram>



Signal: DAD1C, Sig=250, 4 Ref=380, 60

RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
6.158	MM m	0.61000	294.93647	38.63150	99.3963
7.046	MM m	0.18603	1.79123	0.34547	0.6037
		Totals	296.72770		

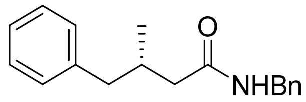
<Chromatogram>



Signal: DAD1C, Sig=250, 4 Ref=380, 60

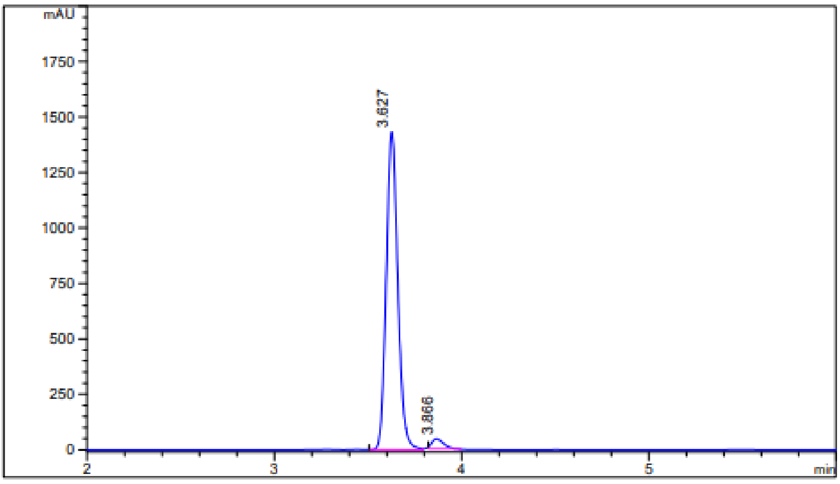
RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
6.098	VV	0.37655	218.53107	29.57186	50.1174
6.896	VV	0.43264	217.50707	24.80577	49.8826
		Totals	436.03814		

Figure S-63. HPLC Data of (*S*)-**3s** (from **2j**) and racemic **3s**



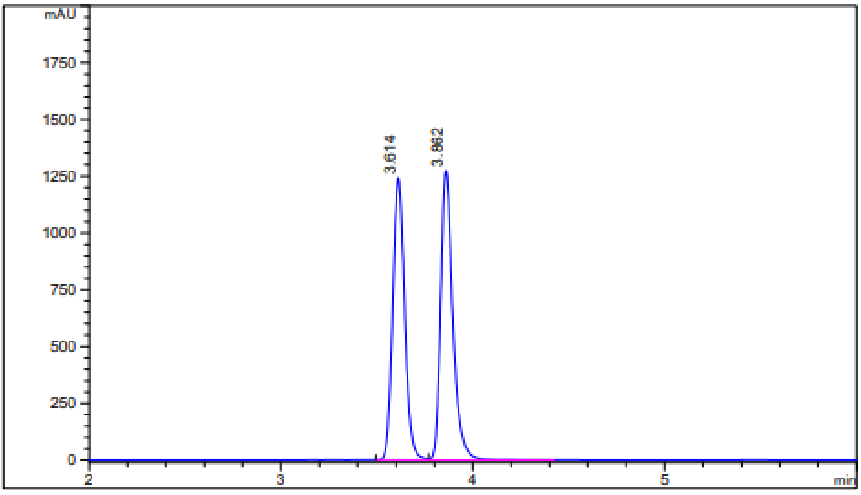
3t, 94% *ee*

<Chromatogram>



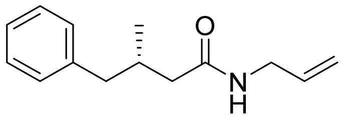
编号	峰高	峰类型	峰面积	峰面积 %	实测保留时间
1	1.432e3	VV	6.179e3	96.920	3.627
2	44.827	MM	196.347	3.080	3.866

<Chromatogram>



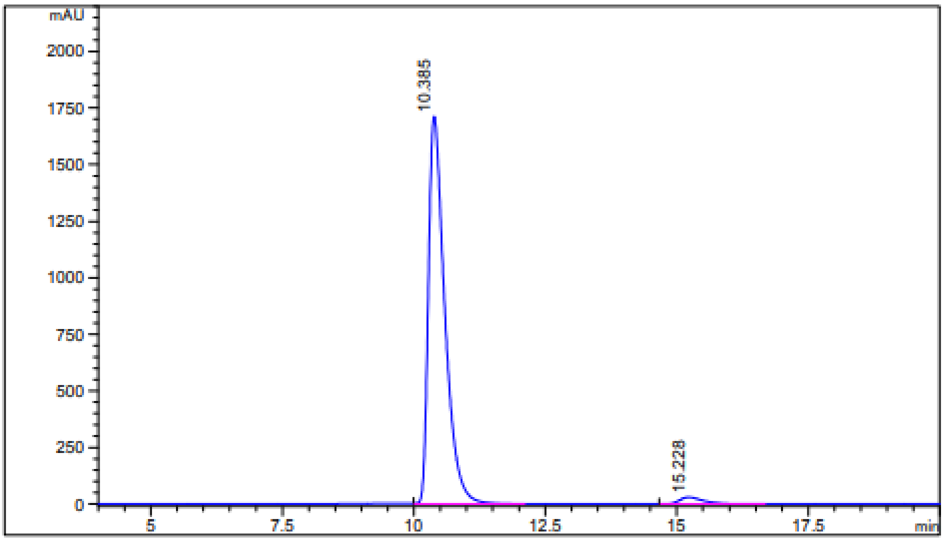
编号	峰高	峰类型	峰面积	峰面积 %	实测保留时间
1	1.242e3	VV	5.361e3	48.839	3.614
2	1.275e3	VV	5.615e3	51.161	3.862

Figure S-64. HPLC Data of (*S*)-**3t** (from **2k**) and racemic **3t**



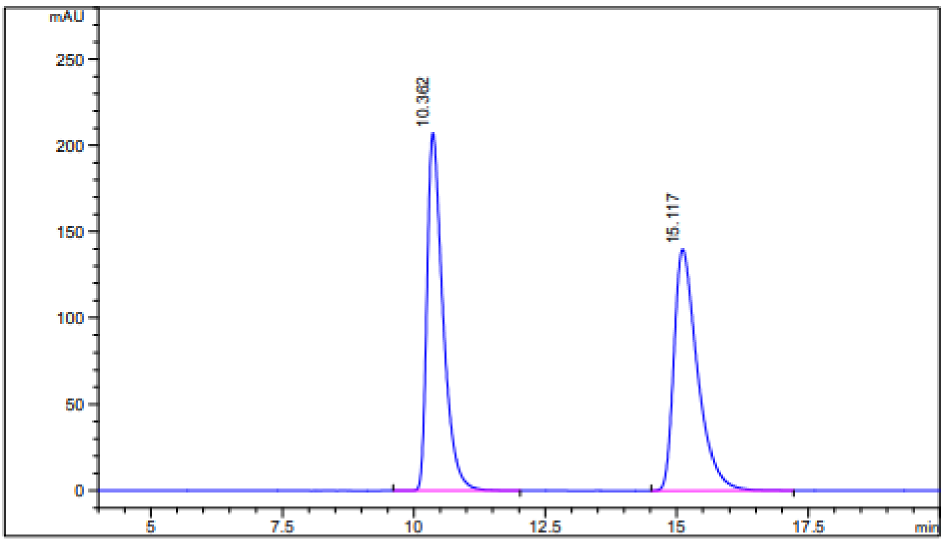
3u, 95% *ee*

<Chromatogram>



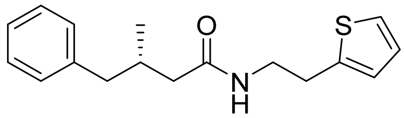
编号	峰高	峰类型	峰面积	峰面积 %	实测保留时间
1	1.711e3	VB	3.705e4	97.560	10.385
2	30.026	BB	926.802	2.440	15.228

<Chromatogram>



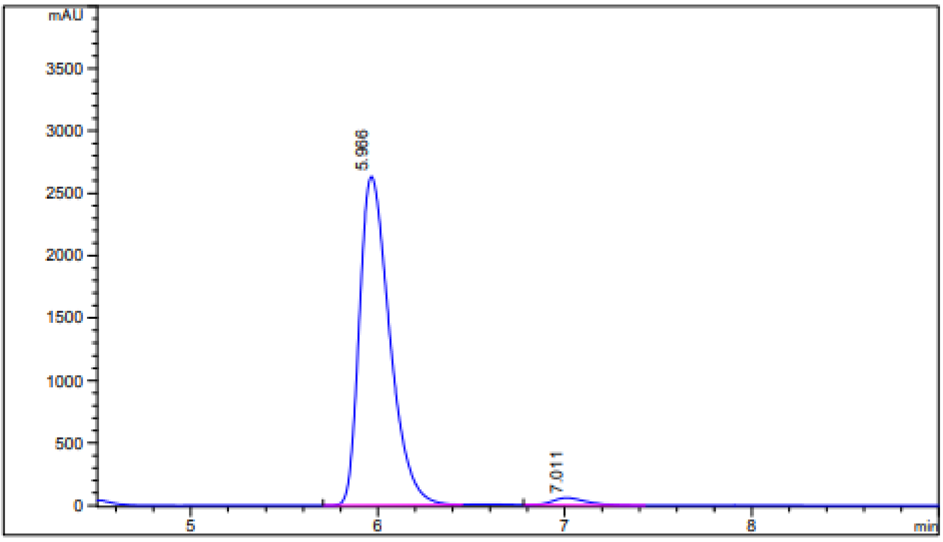
编号	峰高	峰类型	峰面积	峰面积 %	实测保留时间
1	207.290	BB	4.361e3	50.100	10.362
2	139.730	BB	4.343e3	49.900	15.117

Figure S-65. HPLC Data of (*S*)-**3u** (from **2k**) and racemic **3u**



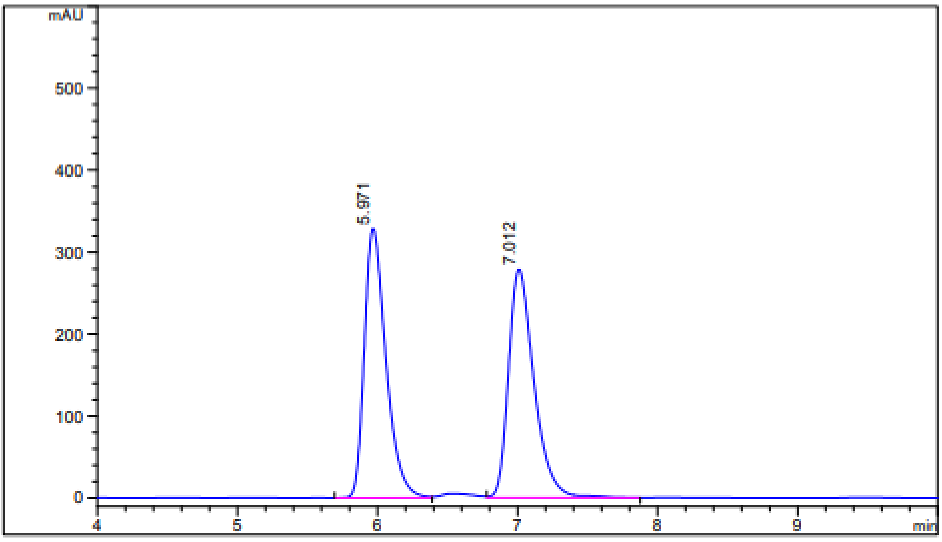
3v, 95% *ee*

<Chromatogram>



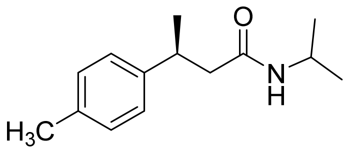
编号	峰高	峰类型	峰面积	峰面积 %	实测保留时间
1	2.632e3	BV	2.964e4	97.734	5.966
2	56.684	BB	687.023	2.266	7.011

<Chromatogram>



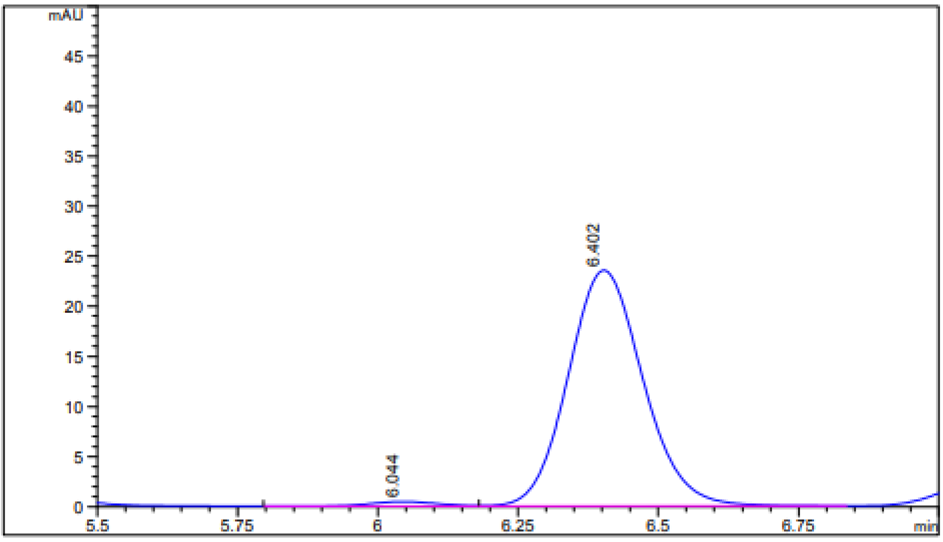
编号	峰高	峰类型	峰面积	峰面积 %	实测保留时间
1	328.771	BV	3.530e3	49.925	5.971
2	278.823	VB	3.540e3	50.075	7.012

Figure S-66. HPLC Data of (*S*)-**3v** (from **2k**) and racemic **3v**



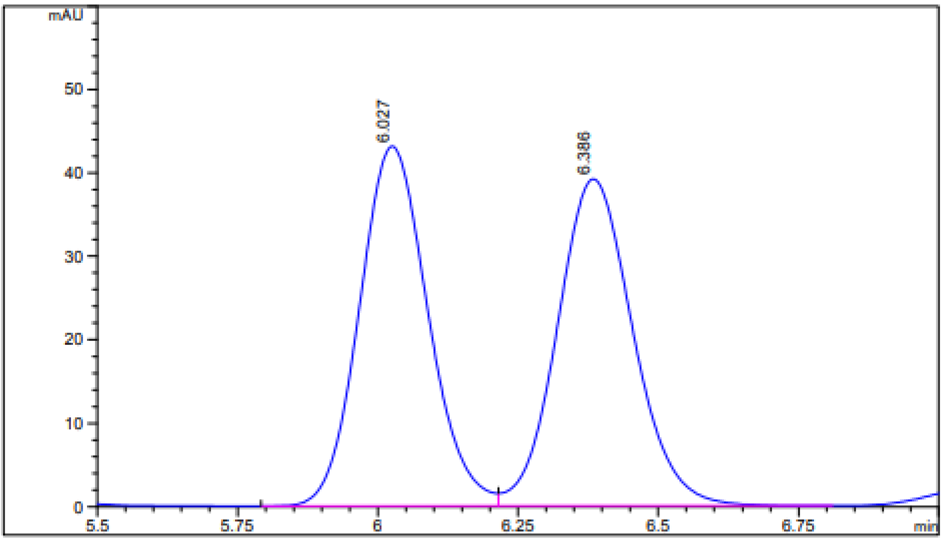
3w, 97% *ee*

<Chromatogram>



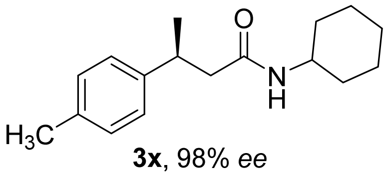
编号	峰高	峰类型	峰面积	峰面积 %	实测保留时间
1	0.443	BV	3.669	1.645	6.044
2	23.467	VB	219.316	98.355	6.402

<Chromatogram>



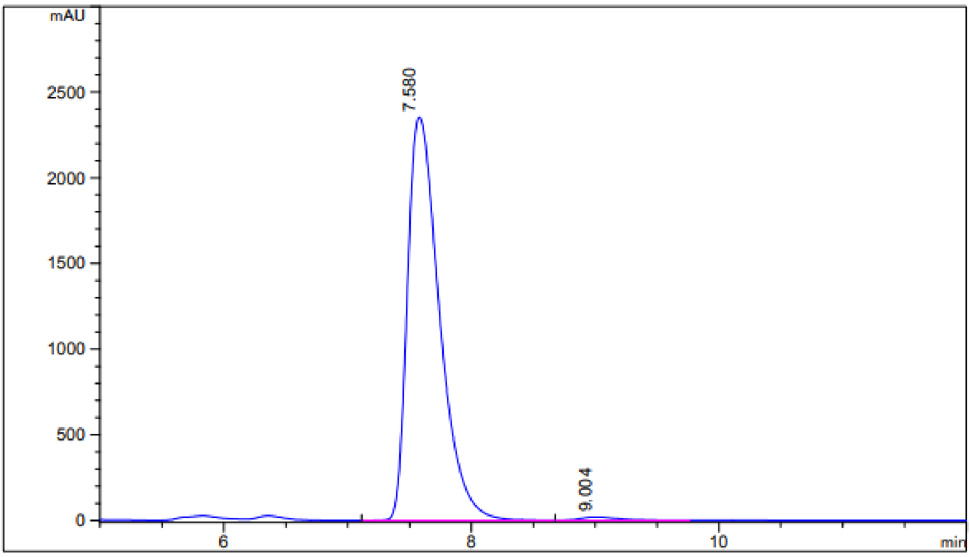
编号	峰高	峰类型	峰面积	峰面积 %	实测保留时间
1	43.081	BV	366.762	49.948	6.027
2	39.161	VB	367.524	50.052	6.386

Figure S-67. HPLC Data of (*S*)-**3w** (from **2l**) and racemic **3w**



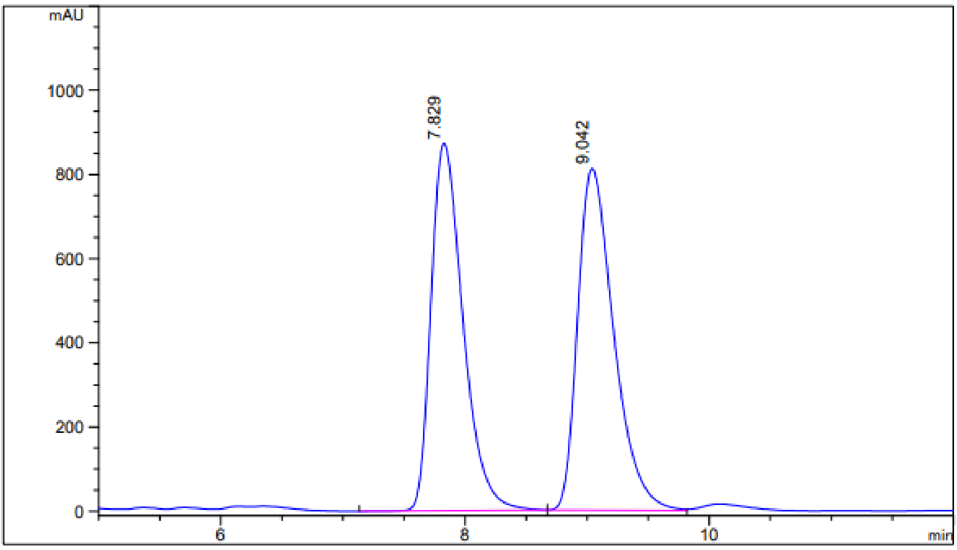
3x, 98% *ee*

<Chromatogram>



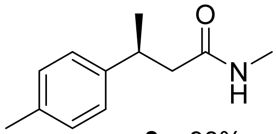
编号	峰高	峰类型	峰面积	峰面积 %	实测保留时间
1	2.352e3	BV	4.232e4	98.953	7.580
2	19.863	VV	447.829	1.047	9.004

<Chromatogram>



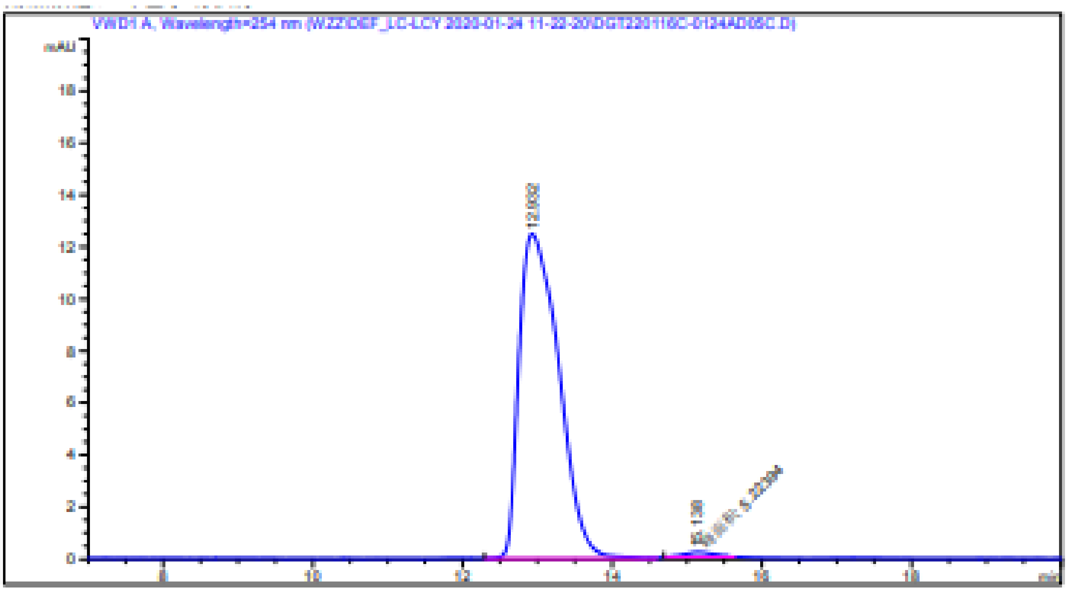
编号	峰高	峰类型	峰面积	峰面积 %	实测保留时间
1	871.621	BB	1.585e4	48.772	7.829
2	811.411	BV	1.665e4	51.228	9.042

Figure S-68. HPLC Data of (*S*)-**3x** (from **2l**) and racemic **3x**



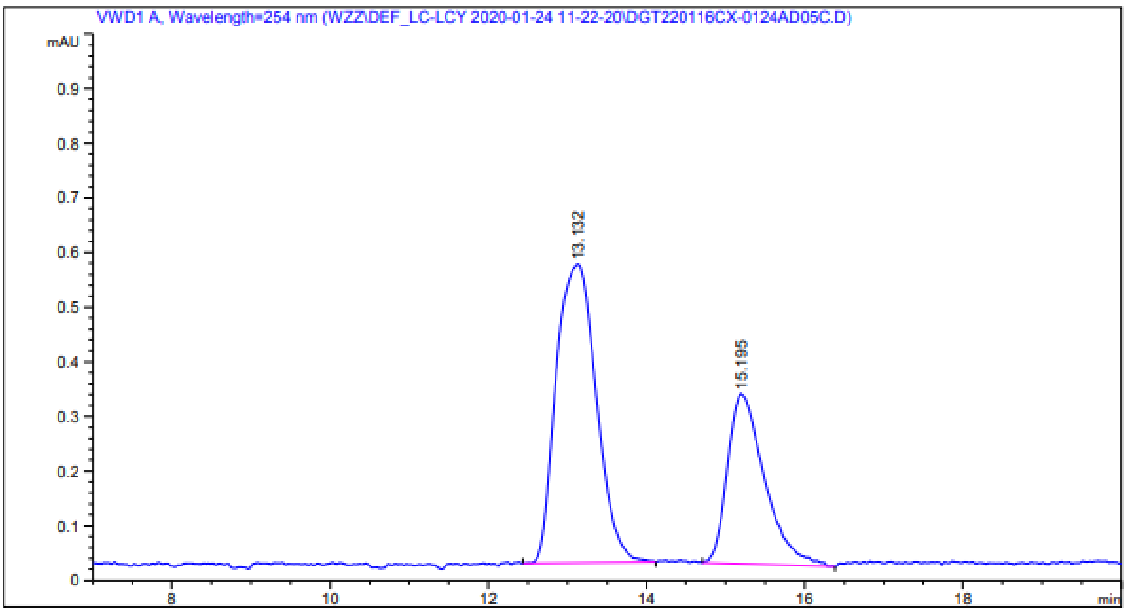
3y, 98% *ee*

<Chromatogram>



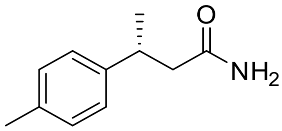
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	12.932	BV	0.6111	459.07980	12.48140	98.8749
2	15.138	MM	0.4864	5.22394	1.79001e-1	1.1251

<Chromatogram>



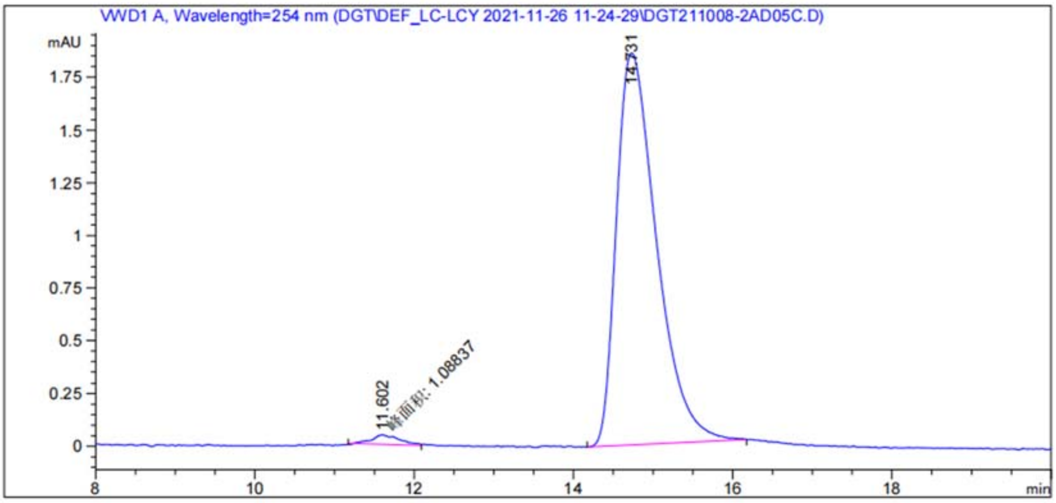
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	13.132	BB	0.4818	19.25915	5.45658e-1	65.5074
2	15.195	BB	0.4765	10.14083	3.10954e-1	34.4926

Figure S-69. HPLC Data of (*S*)-**3y** (from **2m**) and racemic **3y**



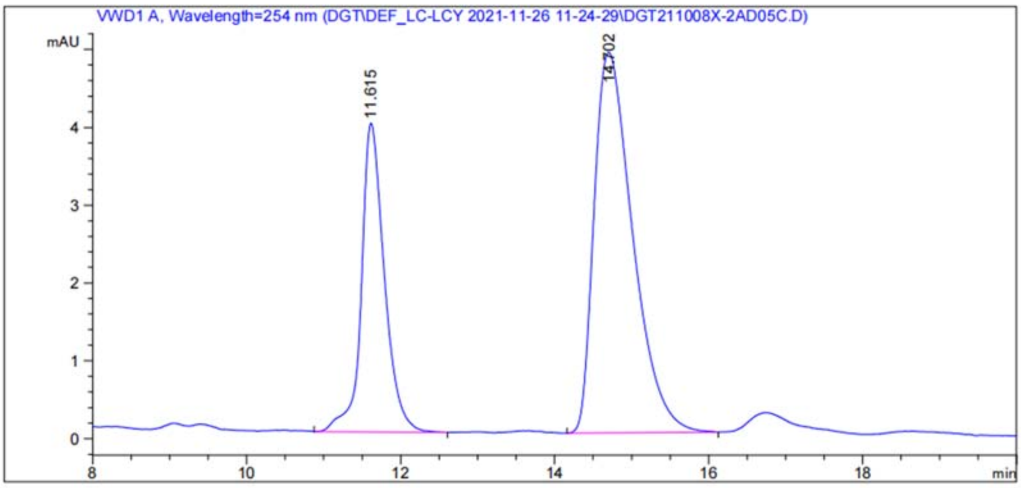
3z, 97% *ee*

<Chromatogram>



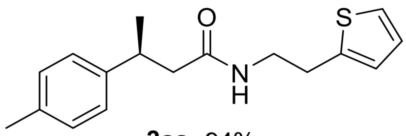
峰 #	保留时间 [min]	信号	类型	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	11.602	1	MM	1.08837	4.43774e-2	1.6459
2	14.731	1	BB	65.03825	1.85761	98.3541
总量 :				66.12661	1.90199	

<Chromatogram>



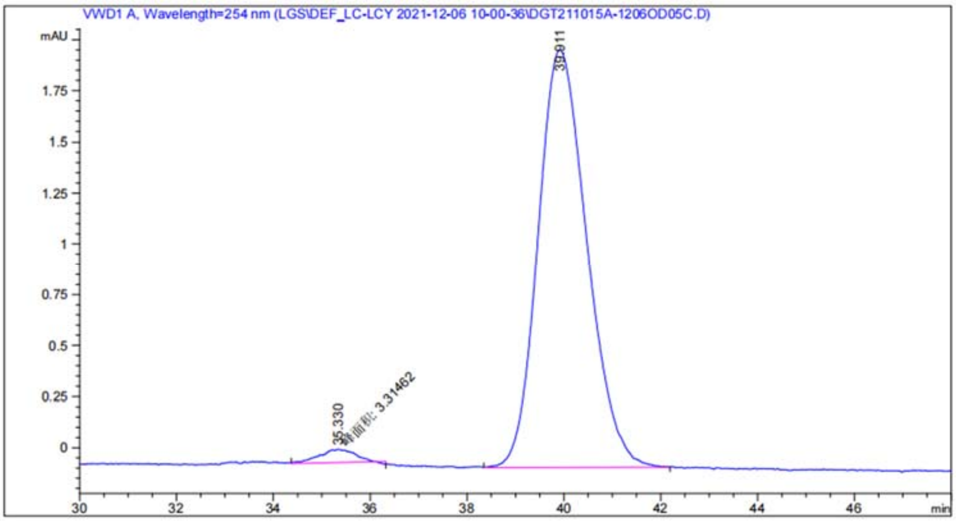
峰 #	保留时间 [min]	信号	类型	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	11.615	1	BB	82.54096	3.96518	32.6894
2	14.702	1	BB	169.96002	4.88664	67.3106
总量 :				252.50098	8.85182	

Figure S-70. HPLC Data of (*R*)-**3z** (from **2n**) and racemic **3z**



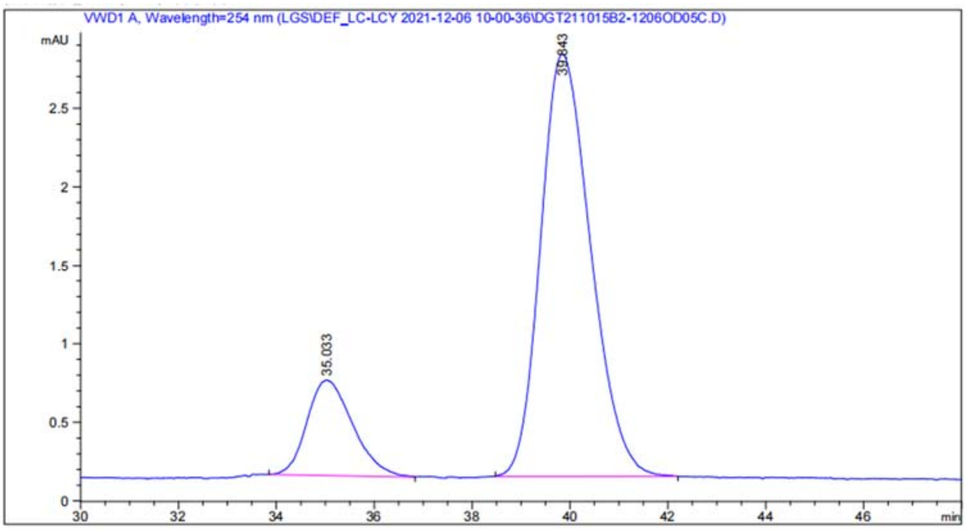
3aa, 94% *ee*

<Chromatogram>



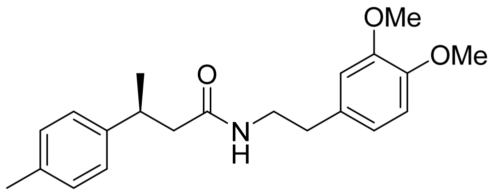
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	35.330	MM	0.8630	3.31462	6.40160e-2	2.2609
2	39.911	BB	0.9329	143.29047	2.04639	97.7391
总量 :				146.60508	2.11041	

<Chromatogram>



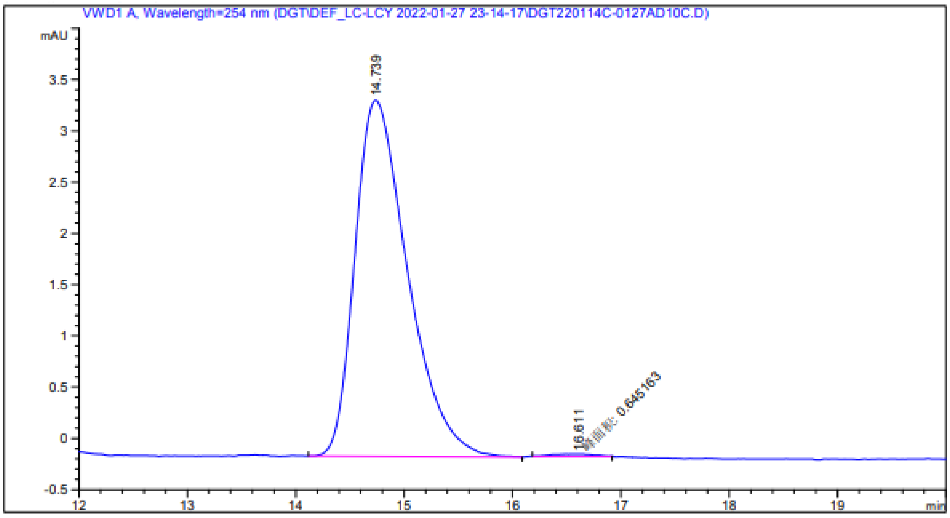
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	35.033	BB	0.7732	39.47625	6.08245e-1	17.1022
2	39.843	BB	1.0466	191.34972	2.68768	82.8978
总量 :				230.82597	3.29592	

Figure S-71. HPLC Data of (*S*)-**3aa** (from **2l**) and racemic **3aa**



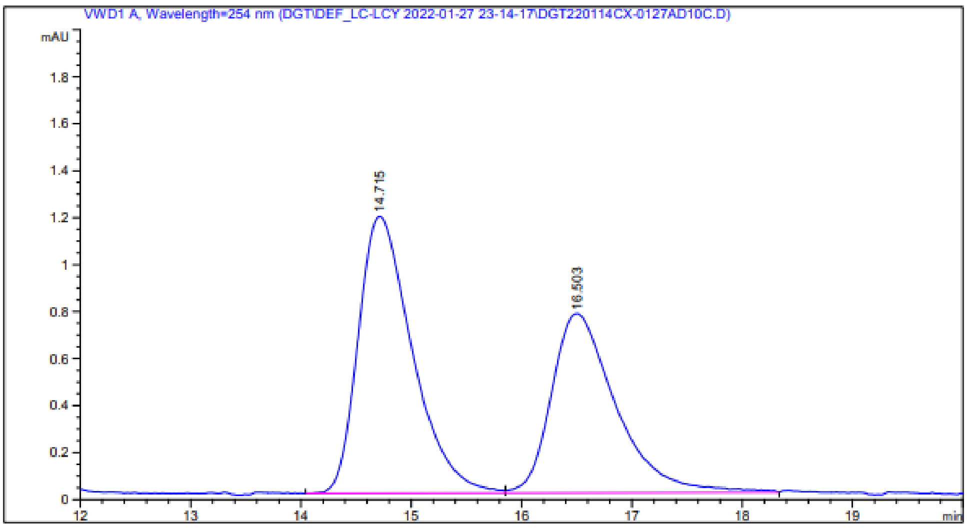
3ab, 99% *ee*

<Chromatogram>



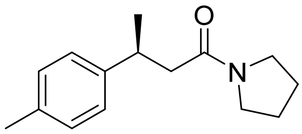
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	14.739	BB	0.5151	117.65303	3.47426	99.4546
2	16.611	MM	0.4470	6.45163e-1	2.40545e-2	0.5454

<Chromatogram>



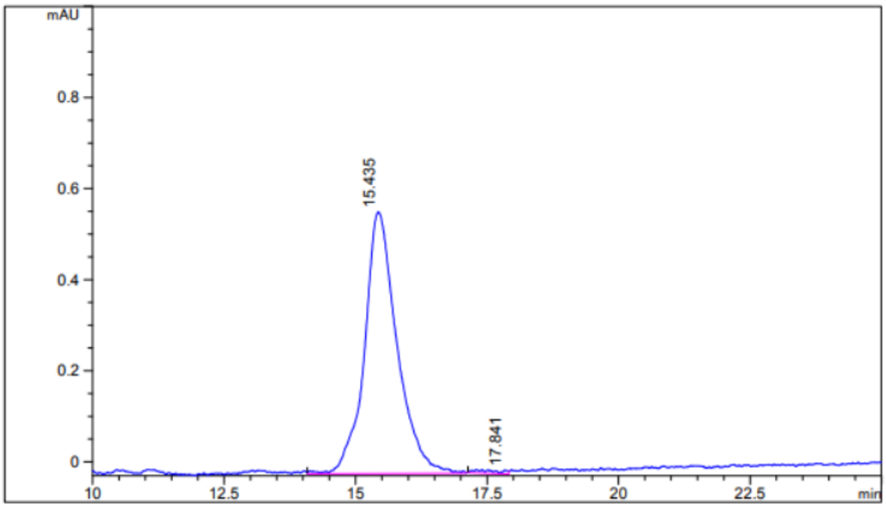
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	14.715	BV	0.5128	40.08183	1.17855	56.4087
2	16.503	VB	0.5898	30.97428	7.62768e-1	43.5913

Figure S-72. HPLC Data of (*S*)-**3ab** (from **2m**) and racemic **3ab**



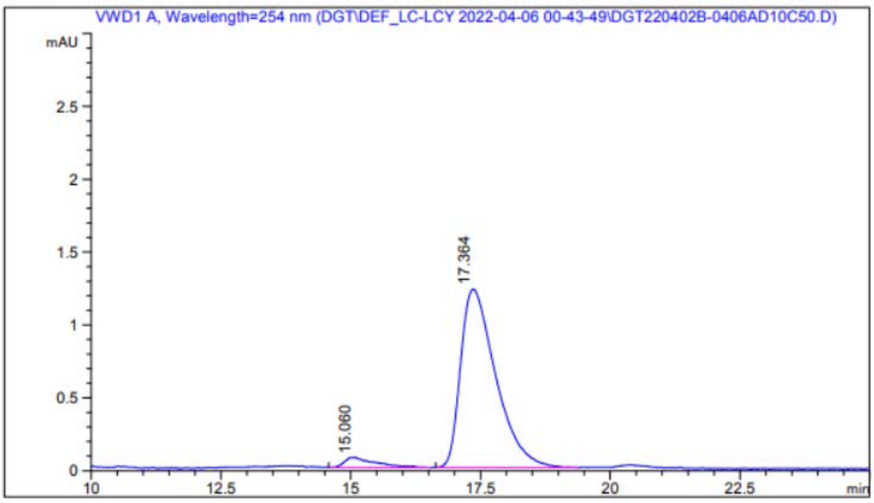
3ad, 98% *ee*

<Chromatogram>



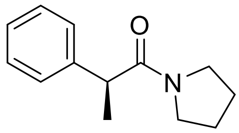
编号	峰高	峰类型	峰面积	峰面积 %	实测保留时间
1	0.574	VV	24.682	98.997	15.435
2	0.008	MM	0.250	1.003	17.841

<Chromatogram>



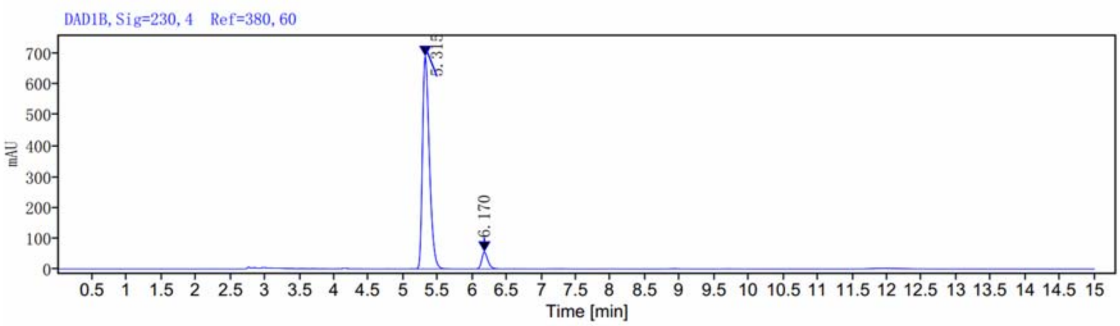
编号	峰高	峰类型	峰面积	峰面积 %	实测保留时间
1	0.071	BB	3.058	5.027	15.060
2	1.225	BB	57.780	94.973	17.364

Figure S-73. HPLC Data of (*S*)-**3ad** (from **2m**) and (*R*)-**3ad** (from **2n**)



3ae, 87% *ee*

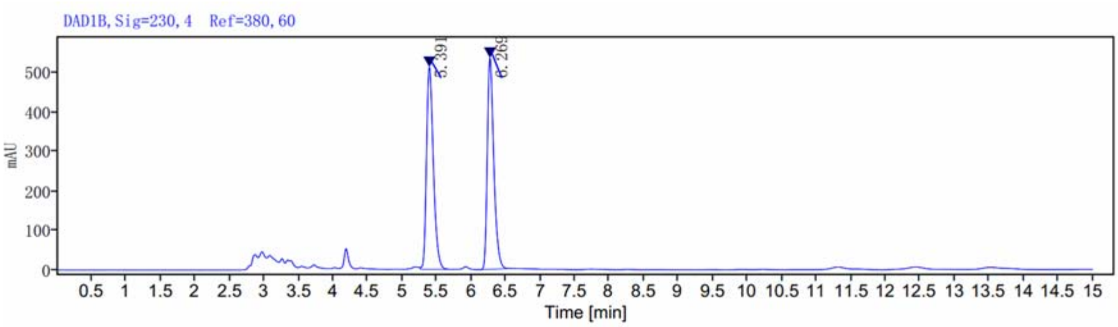
<Chromatogram>



Signal: DAD1B, Sig=230, 4 Ref=380, 60

RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
5.315	BV	0.41023	4813.74732	692.36288	93.4637
6.170	BV	0.31603	336.64425	55.12180	6.5363
		Totals	5150.39157		

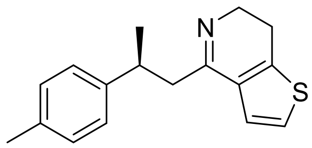
<Chromatogram>



Signal: DAD1B, Sig=230, 4 Ref=380, 60

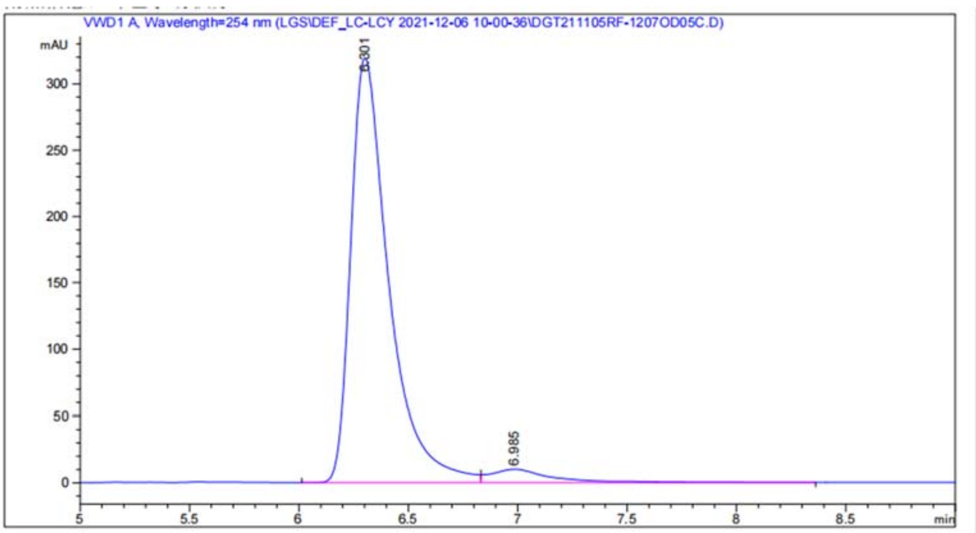
RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
5.391	VB	0.42499	3562.48280	514.27071	50.4734
6.269	BV	0.37753	3495.65376	537.43112	49.5266
		Totals	7058.13657		

Figure S-74. HPLC Data of (*S*)-**3ae** (from **2d**)and racemic **3ae**



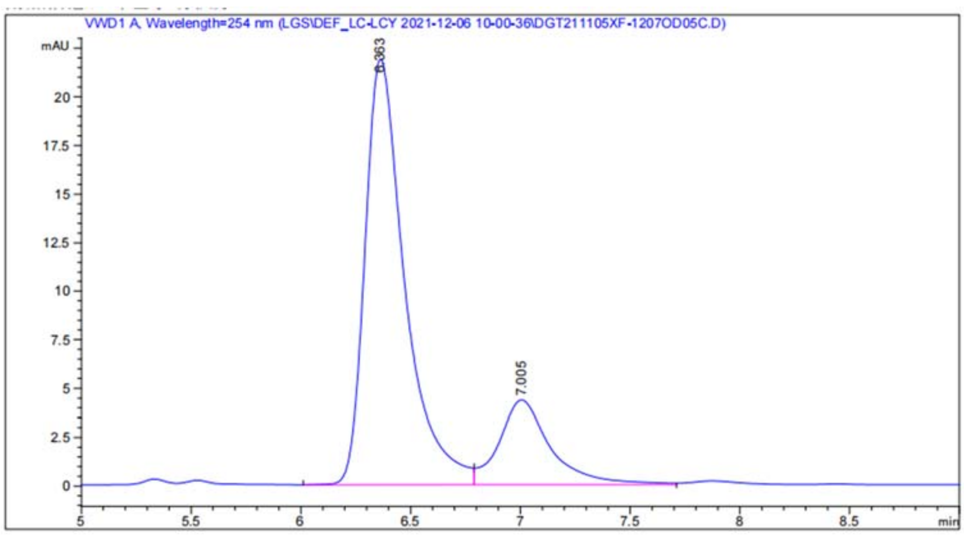
4b, 90% *ee*

<Chromatogram>



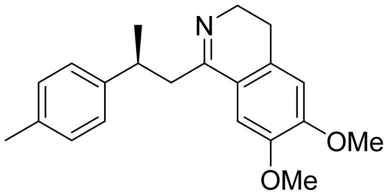
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	6.301	BV	0.1810	3858.81519	319.20117	94.9661
2	6.985	VV	0.2834	204.54344	10.05312	5.0339
总量：				4063.35863	329.25430	

<Chromatogram>



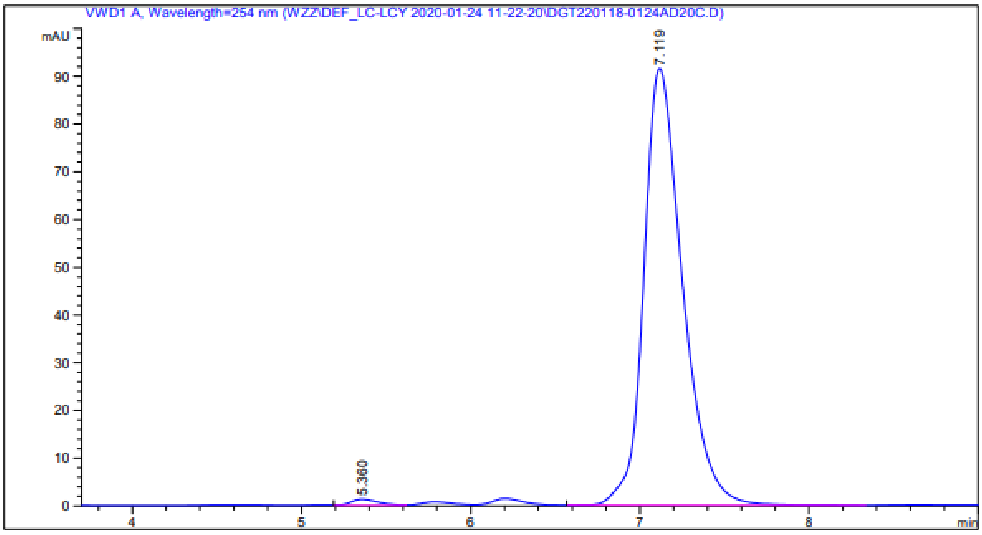
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	6.363	BV	0.1873	276.10880	21.86767	79.5963
2	7.005	VV	0.2368	70.77776	4.32677	20.4037
总量：				346.88656	26.19444	

Figure S-75. HPLC Data of (*S*)-**4b** (from **3aa**) and racemic **4b**



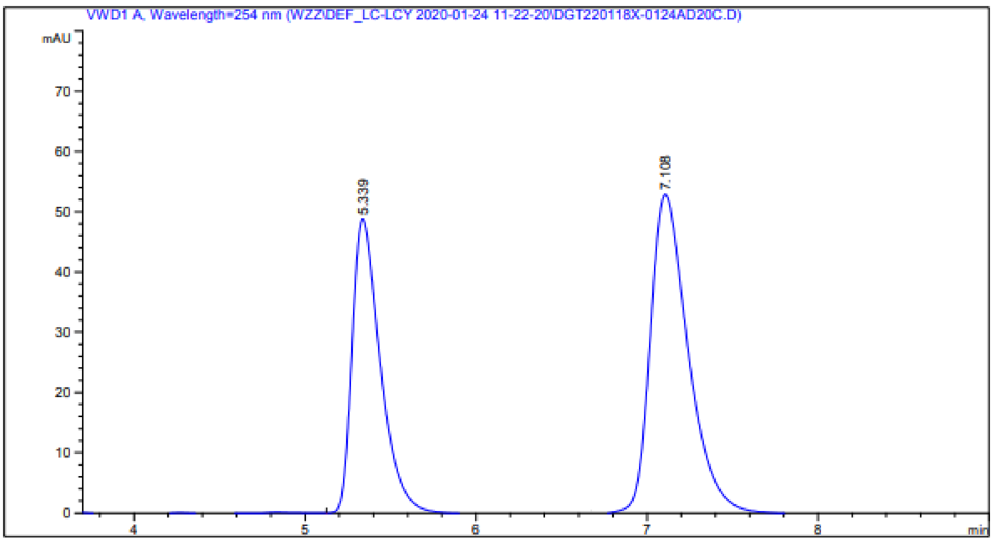
4c, 98% *ee*

<Chromatogram>



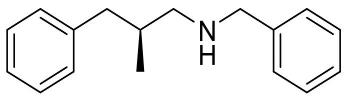
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	5.360	VV	0.1733	14.61541	1.29913	0.9935
2	7.119	BV	0.2396	1456.49365	91.55421	99.0065

<Chromatogram>



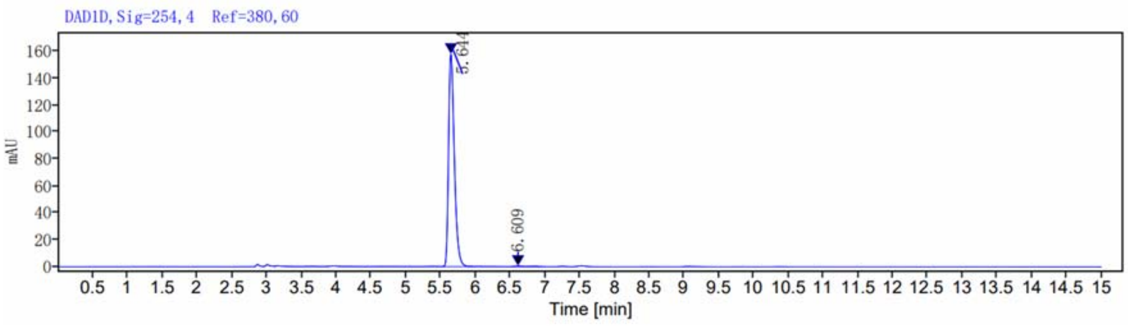
峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	5.339	VB	0.1746	564.02307	48.87109	40.8290
2	7.108	BB	0.2340	817.40607	52.97548	59.1710

Figure S-76. HPLC Data of (*S*)-**4c** (from **3ab**) and racemic **4c**



4d, 99% *ee*

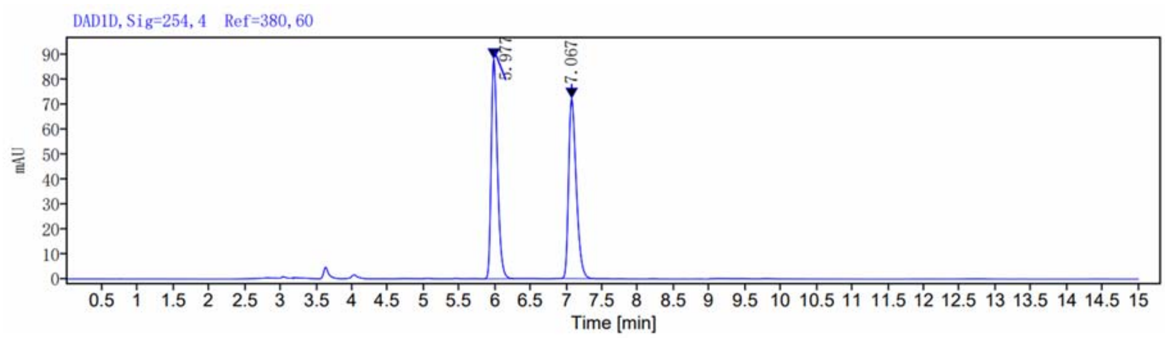
<Chromatogram>



Signal: DAD1D, Sig=254, 4 Ref=380, 60

RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
5.644	BV	0.43354	907.11244	158.12002	99.4396
6.609	MM m	0.46169	5.11218	0.52709	0.5604
		Totals	912.22463		

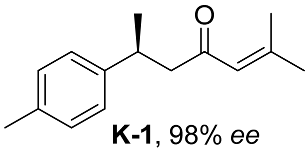
<Chromatogram>



Signal: DAD1D, Sig=254, 4 Ref=380, 60

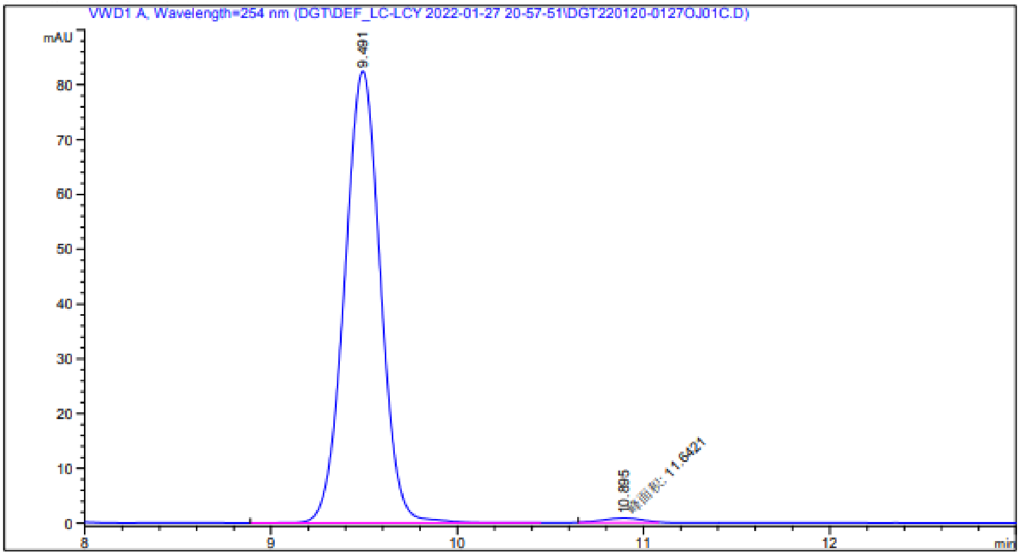
RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
5.977	BV	0.40751	538.27319	87.95378	49.9991
7.067	VV	0.43580	538.29245	72.07830	50.0009
		Totals	1076.56564		

Figure S-77. HPLC Data of (*S*)-**4d** (from **3c**) and racemic **4d**



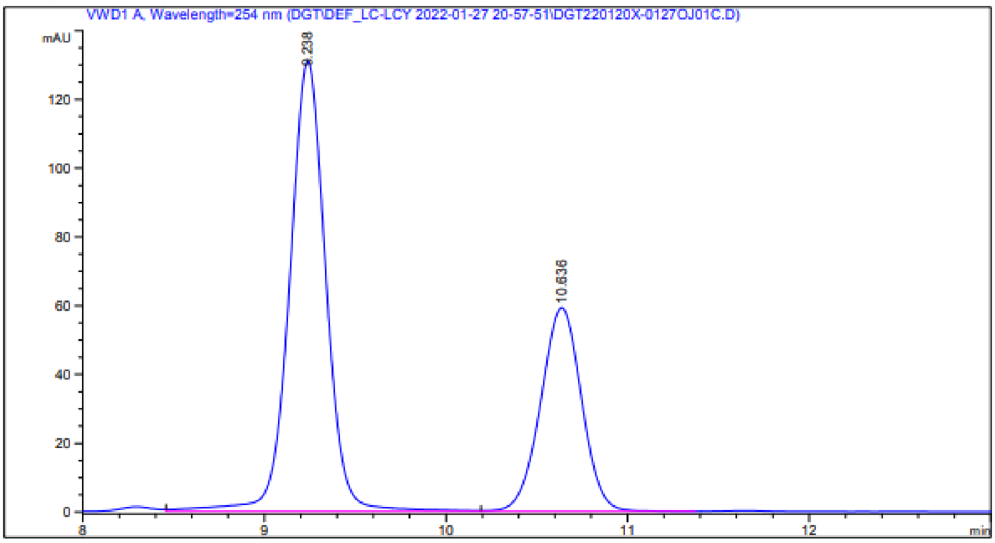
K-1, 98% *ee*
(+)-(*S*)-ar-turmerone

<Chromatogram>



峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	9.491	BB	0.2056	1099.45422	82.43847	98.9522
2	10.895	MM	0.2362	11.64207	8.21571e-1	1.0478

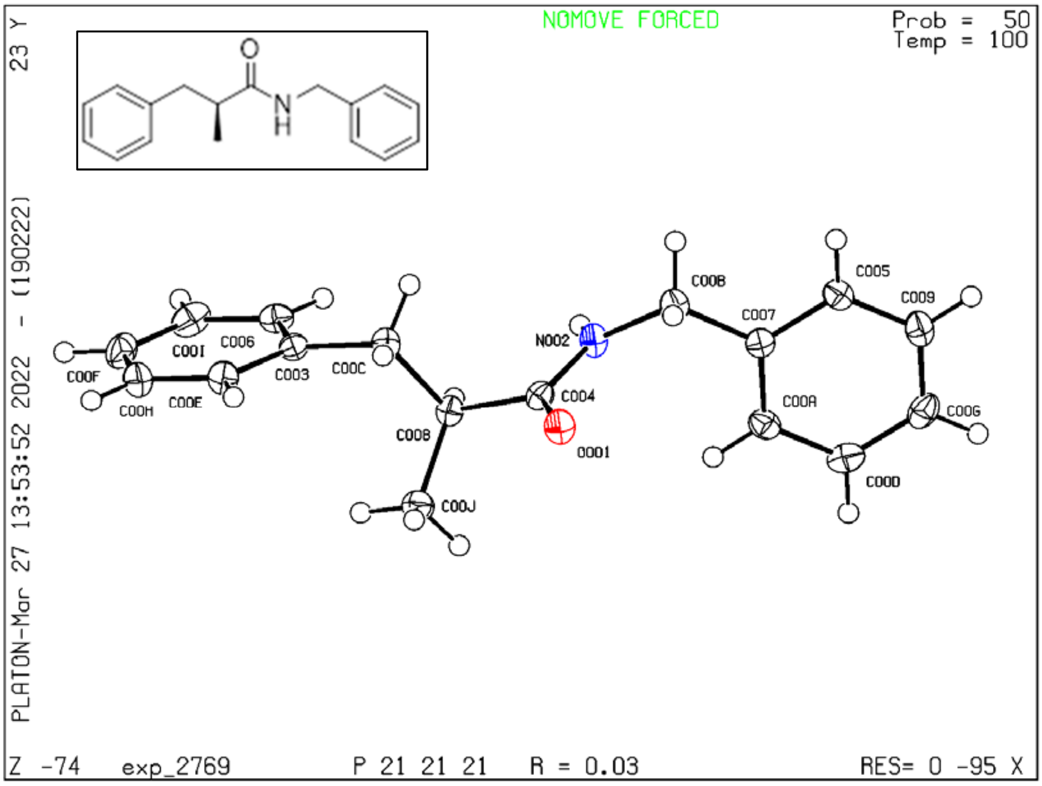
<Chromatogram>



峰 #	保留时间 [min]	类型	峰宽 [min]	峰面积 [mAU*s]	峰高 [mAU]	峰面积 %
1	9.238	VV	0.2147	1824.38293	130.79854	66.3171
2	10.636	VB	0.2427	926.61670	59.16216	33.6829

Figure S-78. HPLC Data of (+)-(*S*)-*ar*-turmerone (*S*)-**K-1** (from **3y**) and racemic **K-1**

X-Ray crystallography of 3c: CCC Deposition Number: 2162378



Single crystals of $C_{17}H_{19}NO$ (**3c**) were recrystallized by vapor diffusion method (ethyl acetate and *n*-hexane) at 25 °C. A suitable crystal was selected and **mounted** on a **XtaLAB Synergy, Dualflex, HyPix** diffractometer. The crystal was kept at 99.99(10) K during data collection. Using Olex2[1], the structure was solved with the SHELXT[2] structure solution program using Intrinsic Phasing and refined with the SHELXL[3] refinement package using Least Squares minimisation.

Identification code	**exp_2769**
Empirical formula	$C_{17}H_{19}NO$
Formula weight	253.33
Temperature/K	99.99(10)
Crystal system	orthorhombic
Space group	$P2_12_12_1$
a/Å	9.74880(10)

b/Å	9.78030(10)
c/Å	14.75820(10)
α **/°**	90
β **/°**	90
γ **/°**	90
Volume/Å^3	1407.14(2)
Z	4
ρ $_{calc}$**g/cm^3**	1.196
μ **/mm^{-1}**	0.574
F(000)	544.0
Crystal size/mm^3	0.2 × 0.2 × 0.2
Radiation	Cu Kα (λ = 1.54184)
2Θ range for data collection/°	10.852 to 148.558
Index ranges	-12 ⩽ h ⩽ 11, -10 ⩽ k ⩽ 12, -17 ⩽ l ⩽ 18
Reflections collected	14481
Independent reflections	2818 [R_{int} = 0.0332, R_{sigma} = 0.0205]
Data/restraints/parameters	2818/0/174
Goodness-of-fit on F^2	1.061
Final R indexes [I>=2σ (I)]	R_1 = 0.0257, wR_2 = 0.0652
Final R indexes [all data]	R_1 = 0.0259, wR_2 = 0.0654
Largest diff. peak/hole / e Å^{-3}	0.17/-0.14
Flack parameter	-0.08(9)

Table 1 Crystal data and structure refinement for **3c**

1. O. V. Dolomanov, L. J. Bourhis, R. J. Gildea, J. A. K. Howard and H. Puschmann, *J. Appl. Cryst*., 2009, **42**, 339-341.

2. G. M. Sheldrick, *Acta Cryst.*, 2015, **A71**, 3-8.

3. G. M. Sheldrick, *Acta Cryst*., 2015, **C71**, 3-8.