Electronic Supplementary Information

Asymmetric formal synthesis of schulzeines A and C

Jaebong Jang,[a] Jong-Wha Jung,[b] Jaeseung Ahn,[a] Jaehoon Sim,[a] Dong-Jo Chang,[a] Dae-Duk Kim,[a] and Young-Ger Suh*[a]

[a]*College of Pharmacy, Seoul National University, Gwanak-ro 599, Gwanak-Gu, Seoul 151-742, Korea,* [b]*College of Pharmacy, Kyungpook National University, Daegu 702-701, Korea*

E-mail: ygsuh@snu.ac.kr

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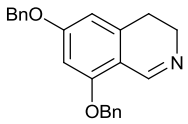
1. General Experimental

Unless noted otherwise, all starting materials and reagents were obtained from commercial suppliers and were used without further purification. Tetrahydrofuran and Et_2O were distilled from sodium benzophenone ketyl. Dichloromethane, chloroform, triethylamine, acetonitrile and pyridine were freshly distilled from calcium hydride. All solvents used for routine isolation of products and chromatography were reagent grade and glass distilled. Reaction flasks were dried at 100 ℃. Air and moisture sensitive reactions were performed under argon atmosphere. Flash column chromatography was performed using silica gel 60 (230-400 mesh, Merck) with the indicated solvents. Thin-layer chromatography was performed using 0.25 mm silica gel plates (Merck). Optical rotations were measured with JASCO P-2000 digital polarimeter using 100 mm cell of 1.5 – 2 mL capacity. Infrared spectra were recorded on a Perkin-Elmer 1710 FT-IR spectrometer. Mass spectra were obtained with VG Trio-2 GC-MS instrument. High resolution mass spectra were obtained with JEOL JMS-AX 505WA instrument. ^1H and ^{13}C NMR spectra were recorded on a Bruker Avance 500 (500 MHz), Avance 400 (400 MHz) or JEOL JNM-LA 300 spectrometer as solutions in deuteriochloroform ($CDCl_3$). Chemical shifts are expressed in parts per million (ppm, δ) downfield from tetramethylsilane and are referenced to the deuterated solvent ($CHCl_3$). ^1H-NMR data were reported in the order of chemical shift, multiplicity (s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet; br, broad and/or multiple resonances), number of protons, and coupling constant in hertz (Hz).

2. Experimental Section

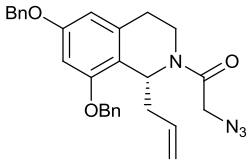
6,8-Bis(benzyloxy)-3,4-dihydroisoquinoline (7)



To the mixture of 2-(3,5-bis(benzyloxy)phenyl)ethylamine (2.0 g, 11.0 mmol), AcOH (12 mL) and TFA (3 mL) was added hexamethylenetetramine (3.1 g, 22.1 mmol) under argon and the mixture was stirred for 3 h at 90˚C. The reaction mixture was diluted with H_2O, basified with potassium carbonate and extracted with CH_2Cl_2. The combined organic layers were washed with brine, dried over $MgSO_4$ and concentrated in vacuo. The residue was purified by column chromatography on silica gel (5 to 10% EtOAc in hexane) to afford dihydroisoquinoline **7** (3.5 g, 95%) as yellow solid.

FT-IR (thin film, neat) ν_{max} 3062, 3032, 2935, 1736, 1620, 1603, 1575, 1497, 1442, 1377, 1351, 1309 cm^{-1}; ^{1}H-NMR ($CDCl_3$, 400 MHz) δ 8.69 (s, 1H), 7.43 – 7.29 (m, 10H), 6.45 (d, *J* = 1.88 Hz, 2H), 6.36 (s, 1H), 5.05 (s, 2H), 5.04 (s, 2H), 3.67 (t, 2H), 2.65 (t, 2H); ^{13}C-NMR ($CDCl_3$, 100 MHz) δ 161.9, 157.7, 155.2, 140.0, 136.3, 128.6, 128.5, 128.1, 128.0, 127.4, 127.1, 111.9, 105.3, 98.5, 70.1, 46.5, 26.0; LR-MS (FAB+) *m/z* 344 ($M+H^+$); HR-MS (FAB+) calcd for $C_{23}H_{22}NO_2$ ($M+H^+$) 344.1651; found 344.1658.

(*R*)-1-(1-Allyl-6,8-bis(benzyloxy)-3,4-dihydroisoquinolin-2(1*H*)-yl)-2-azidoethanone (11)



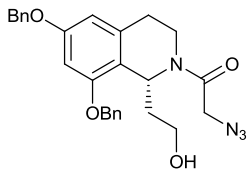
To a solution of bis-oxazoline (833.0 mg, 3.5 mmol) and 2,2`-dipyridyl (2.0 mg) in THF (3 mL) was added a 2.5 M soln. of *n*-BuLi in hexane (1.4 mL, 3.5 mmol) at 0˚C till the solution turned red. The reaction mixture was warmed to room temperature and stirred for 30 min. A 1.0 M soln. of allylzinc bromide in THF (2.4 mL, 2.4 mmol) was added to the reaction mixture. After stirring for 1h, dihydroisoquinoline **7** (300.0 mg, 0.87 mmol) in THF (9 mL) was added to the wine red reaction mixture at -78˚C by cannulation and then stirred for 4 h. The reaction mixture was quenched with MeOH at -78˚C and warmed to room temperature. The reaction mixture was extracted with EtOAc. The combined organic layers were washed with brine, dried over $MgSO_4$ and concentrated in vacuo to afford the crude homoallylic amine **9**.

To a solution of the resulting homoallylic amine **9** and 2-azidoacetic acid (265.0 mg, 2.6 mmol) in THF (9 mL) were added EDCI (502.0 mg, 2.6 mmol), DMAP (320.0 mg, 2.6 mmol) and HOBt (118.0 mg, 0.87 mmol). The reaction mixture was stirred for 12 h and was extracted with EtOAc. The combined organic layers were

washed with brine, dried over $MgSO_4$ and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel (25% EtOAc in hexane) to afford homoallylic amide **11** (299.0 mg, 73%) as a colorless oil**.**

$[\alpha]_D^{20}$ -96.0(c 1.0, $CHCl_3$); FT-IR (thin film, neat) ν_{max} 2106, 1739, 1656, 1607, 1496, 1453, 1376 cm^{-1}; ^{1}H-NMR ($CDCl_3$, 400 MHz, mixture of rotamers) δ 7.45 -7.28 (m, 10H), 6.49 (d, *J* = 11.7 Hz, 1H), 6.37 (d, *J* = 14.6 Hz, 1H), 5.94 (dd, *J* = 4.1, 9.7 Hz), 5.89 – 5.70 (m, 1H), 5.11 – 4.93 (m, 6H), 4.76 (dd, *J* = 2.6, 10.1 Hz, 1H), 4.70 (dd, *J* = 6.0, 13.2 Hz), 3.94 (m, 2H), 3.57 (m, 1H), 3.16 (td, *J* = 4.5, 12.6 Hz, 1H), 2.99 – 2.68 (m, 3H), 2.49 – 2.36 (m, 1H); ^{13}C-NMR ($CDCl_3$, 100 MHz, mixture of rotamers) δ 166.2, 165.6, 158.7, 158.3, 156.0, 155.4, 136.7, 136.6, 136.5, 136.4, 136.0, 135.3, 134.8, 134.1, 128.7, 128.6, 128.5, 128.5, 128.1, 128.0, 127.9, 127.8, 127.6, 127.4, 127.4, 127.2, 126.9, 118.8, 118.6, 117.9, 116.5, 105.9, 105.5, 98.6, 98.6, 70.2, 70.1, 70.0, 69.9, 51.9, 50.8, 50.5, 48.1, 38.7, 38.4, 38.0, 34.9, 29.1, 28.0; LR-MS (FAB+) *m/z* 469 ($M+H^+$); HR-MS (FAB+) calcd for $C_{28}H_{29}N_4O_3$ ($M+H^+$) 469.2240; found 469.2242.

(*R*)-2-Azido-1-(6,8-bis(benzyloxy)-1-(2-hydroxyethyl)-3,4-dihydroisoquinolin-2(1*H*)-yl)ethanone (12)



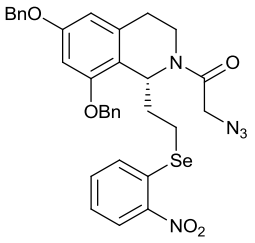
To a solution of homoallylamide **6** (378.0 mg, 0.81 mmol) and *N*-methylmorpholine *N*-oxide (284.0 mg, 2.4 mmol) in THF (6 mL) and H_2O (1.5 mL) was added OsO_4 (0.1 M in toluene, 0.4 mL, 0.04 mmol). The reaction mixture was stirred for 6 h, quenched with saturated Na_2CO_3 solution and extracted with Et_2O three times. The combined organic layers were concentrated in vacuo. The residue was dissolved in THF (6 mL) and H_2O (1.5 mL) and $NaIO_4$ (518.0 mg, 2.4 mmol) was added. The reaction mixture was stirred for 20 min and was extracted with Et_2O three times. The combined organic layers were washed with brine, dried over $MgSO_4$ and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel (50% EtOAc in hexane) to afford the aldehyde (354.0 mg, 93%) as a colorless oil.

To a solution of the aldehyde (574.0 mg, 1.2 mmol) in MeOH (12 ml) was added $NaBH_4$ (69.0 mg, 1.8 mmol). The reaction mixture was stirred for 5 min, quenched with saturated aqueous $NaHCO_3$ solution, and extracted with EtOAc. The combined organic layers were washed with brine, dried over $MgSO_4$ and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel (67% EtOAc in hexane) to afford alcohol **12** (548.0 mg, 95%) as a colorless oil.

$[\alpha]_D^{20}$ -45.9 (c 1.0, $CHCl_3$); FT-IR (thin film, neat) ν_{max} 3435, 2926, 2106, 1639, 1608, 1455 cm^{-1}; ^{1}H-NMR ($CDCl_3$, 400 MHz, mixture of rotamers) δ 7.46 – 7.26 (m, 10H), 6.48 (d, *J* = 6.52 Hz, 1H), 6.36 (d, *J* = 13.6 Hz, 1H), 5.88 (dd, *J* = 2.8, 10.7 Hz, 1H), 5.12 – 4.96 (m, 4H), 4.56 (dd, *J* = 4.5, 1.3 Hz), 4.28 (d, *J* = 15.7 Hz), 4.00 (q, *J* = 15.0 Hz, 2H), 3.96 (d, *J* = 16.0 Hz), 3.69 – 3.58 (m, 2H), 3.53 – 3.44 (m, 2H), 3.11 (td, *J* = 4.7, 12.2 Hz),

2.99 – 2.78 (m, 2H), 2.70 (d, *J* = 16.7 Hz), 2.36 – 2.26 (m, 1H), 2.17 – 2.08 (m), 1.88 – 1.78 (m), 1.72 – 1.63 (m, 1H); ^{13}C-NMR ($CDCl_3$, 100 MHz, mixture of rotamers) δ 167.2, 166.9, 158.7, 158.5, 155.7, 155.3, 136.7, 136.6, 136.4, 136.3, 136.2, 134.6, 128.7, 128.6, 128.3, 128.0, 128.0, 127.8, 127.5, 127.4, 127.3, 126.8, 118.8, 118.5, 106.1, 105.5, 99.0, 98.7, 70.3, 70.1, 70.1, 70.0, 59.1, 58.5, 50.8, 50.3, 47.7, 46.4, 39.5, 36.3, 35.5, 28.9, 28.0; LR-MS (FAB+) *m/z* 473 ($M+H^+$); HR-MS (FAB+) calcd for $C_{27}H_{29}N_4O_4$ ($M+H^+$) 473.2189; found 473.2183.

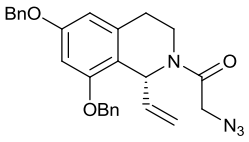
(*R*)-2-Azido-1-(6,8-bis(benzyloxy)-1-(2-(2-nitrophenylselanyl)ethyl)-3,4-dihydroisoquinolin-2(1*H*)-yl)ethanone



To a solution of alcohol **12** (546.0 mg, 1.2 mmol) in THF (12 ml) were added 2-nitrophenyl selenocyanate (880.0 mg, 3.9 mmol) and tributyl phosphine (0.63 mL, 3.9 mmol). The reaction mixture was stirred for 4 h and concentrated under reduced pressure. The residue was purified by flash column chromatography on silica gel (25% EtOAc in hexane) to afford the selenide (542.0 mg, 73%) as a yellow oil.

$[\alpha]_D^{20}$ -20.8 (c 0.2, $CHCl_3$); FT-IR (thin film, neat) ν_{max} 3063, 3031, 3007, 2925, 2857, 2106, 1954, 1812, 1737, 1655, 1607, 1591, 1566, 1510, 1453, 1377, 1332, 1304 cm^{-1}; ^{1}H-NMR ($CDCl_3$, 500 MHz, mixture of rotamers) δ 8.25 (d, *J* = 8.3 Hz), 8.21 (dd, *J* = 1.9, 7.8 Hz, 1H), 7.40 – 7.18 (m, 13H), 6.47 (d, *J* = 2.1 Hz, 1H), 6.36 (d, *J* = 1.8 Hz), 6.34 (d, *J* = 1.9 Hz, 1H), 5.92 (dd, *J* = 3.9, 9.3 Hz, 1H), 5.03 (s), 5.02 (s, 2H), 4.99 (s, 2H), 4.93 (dd, *J* = 3.7, 9.5 Hz), 4.59 (dd, *J* = 4.7, 13.4 Hz), 4.08 – 3.91 (m, 2H), 3.65 – 3.52 (m, 1H), 3.17 (td, *J* = 4.7, 12.3 Hz), 3.02 – 2.67 (m, 5H), 2.41 – 2.31 (m, 1H), 2.22 – 2.13 (m), 2.08 – 1.98 (m, 1H); ^{13}C-NMR ($CDCl_3$, 125 MHz, mixture of rotamers) δ 166.9, 166.5, 159.0, 158.6, 156.1, 155.4, 146.9, 146.7, 136.7, 136.6, 136.4, 136.2, 136.0, 134.8, 133.7, 133.6, 133.5, 132.6, 129.1, 128.8, 128.7, 128.6, 128.4, 128.1, 128.1, 127.5, 127.4, 127.3, 127.2, 126.5, 126.3, 125.6, 125.3, 118.2, 117.6, 106.3, 105.7, 98.9, 70.5, 70.2, 70.2, 51.6, 51.0, 50.7, 49.6, 39.4, 35.7, 33.4, 32.5, 29.1, 27.8, 23.0, 22.3; LR-MS (FAB+) *m/z* 658 ($M+H^+$); HR-MS (FAB+) calcd for $C_{33}H_{32}N_5O_5Se$ ($M+H^+$) 658.1572; found 658.1586.

(*R*)-2-Azido-1-(6,8-bis(benzyloxy)-1-vinyl-3,4-dihydroisoquinolin-2(1*H*)-yl)ethanone (13)

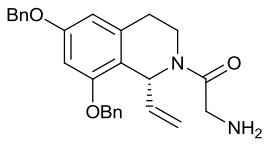


To a solution of the selenide (568.0 mg, 0.9 mmol) in THF (8 mL) was added 30% H_2O_2 (0.1 mL) and the reaction mixture was stirred at 45 ˚C for 6 h. The reaction mixture was cooled at room temperature, quenched

with saturated aqueous $NaHCO_3$ solution and extracted with EtOAc. The combined organic layers were washed with brine, dried over $MgSO_4$ and concentrated in vacuo. The residue was purified by column chromatography on silica gel (33% EtOAc in hexane) to afford allylic amide **13** (382.0 mg, 95%) as a colorless oil

$[\alpha]_D^{20}$ -55.6 (c 0.76, $CHCl_3$); FT-IR (thin film, neat) ν_{max} 2931, 2106, 1658, 1606, 1496, 1453, 1377, 1353 cm^{-1}; ^{1}H-NMR ($CDCl_3$, 400 MHz, mixture of rotamers) δ 7.42 – 7.26 (m, 10H), 6.49 (s, 1H), 6.47 (s), 6.40 (s, 1H), 6.36 (s), 6.02 – 5.90 (m, 1H), 5.38 (s), 5.19 – 4.96 (m, 6H), 4.90 (d, *J* = 17.2 Hz, 1H), 4.29 (dt, *J* = 5.3, 13.1 Hz), 4.04 – 3.89 (m, 2H), 3.58 – 3.52 (m), 3.40 – 3.31 (dq, *J* = 5.0, 13.0 Hz), 2.93 – 2.70 (m, 4H); ^{13}C-NMR ($CDCl_3$, 100 MHz, mixture of rotamers) δ 166.6, 165.6, 160.0, 158.7, 156.3, 155.6, 136.9, 136.7, 136.6, 136.6, 136.4, 135.9, 135.6, 128.7, 128.6, 128.6, 128.5, 128.1, 128.1, 128.0, 127.8, 127.5, 127.2, 127.0, 116.4, 115.6, 115.5, 105.9, 105.4, 98.8, 98.7, 70.3, 70.1, 70.1, 69.9, 52.8, 50.8, 50.5, 50.2, 39.6, 37.4, 29.0, 28.1; LR-MS (FAB+) *m/z* 455 ($M+H^+$); HR-MS (FAB+) calcd for $C_{27}H_{27}N_4O_3$ ($M+H^+$) 455.2083; found 455.2086.

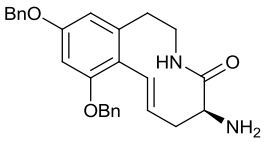
2-Amino-1-(6,8-bis(benzyloxy)-1-vinyl-3,4-dihydroisoquinolin-2(1*H*)-yl)ethanone (6)



To a solution of the allylic amide **13** (185.0 mg, 0.4 mmol) in THF (4 mL) and H_2O (0.2mL) was added triphenylphosphine (214.0 mg, 0.8 mmol). The reaction mixture was stirred for 12 h, quenched with saturated aqueous $NaHCO_3$ solution (2 mL) and stirred for an additional 2 h. The reaction mixture was diluted with EtOAc and extracted with EtOAc three times. The combined organic layers were washed with brine, dried over $MgSO_4$ and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel (7% MeOH in DCM) to afford the free amine **6** (160.0 mg, 90%) as a colorless oil

$[\alpha]_D^{20}$ -546.4 (c 1.0, $CHCl_3$); FT-IR (thin film, neat) ν_{max} 3374, 3032, 2928, 1650, 1606, 1496, 1454, 1375, 1353 cm^{-1}; ^{1}H-NMR ($CDCl_3$, 400 MHz, mixture of rotamers) δ 7.41 – 7.26 (m, 10H), 6.46 (d, *J* = 11.4 Hz, 1H), 6.36 (d, *J* = 12.2 Hz, 1H), 5.99 – 5.88 (m, 1H), 5.45 (s, 1H), 5.14 – 4.94 (m, 6H), 4.88 (d, *J* = 17.2 Hz), 4.32 (dt, *J* = 5.2, 12.6 Hz), 3.62 – 3.43 (m, 3H), 3.31 (dq, *J* = 4.9, 13.0 Hz), 2.90 – 2.68 (m, 3H), 1.86 (s, NH_2); ^{13}C-NMR ($CDCl_3$, 100 MHz, mixture of rotamers) δ 172.0, 171.0, 158.9, 158.6, 158.2, 155.6, 137.1, 136.8, 136.7, 136.7, 136.5, 136.3, 136.0, 135.8, 128.6, 128.6, 128.6, 128.5, 128.1, 128.0, 127.8, 127.5, 127.4, 127.1, 127.0, 116.8, 116.1, 115.0, 115.0, 105.8, 105.4, 98.7, 98.6, 70.2, 70.1, 70.1, 69.9, 52.1, 50.2, 43.4, 43.1, 38.9, 37.2, 29.0, 28.2; LR-MS (FAB+) *m/z* 429 ($M+H^+$); HR-MS (FAB+) calcd for $C_{27}H_{29}N_2O_3$ ($M+H^+$) 429.2178; found 429.2156.

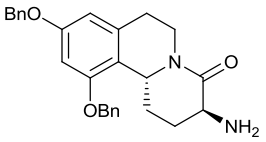
(*S*,*E*)-5-Amino-9,11-bis(benzyloxy)-2,3,5,6-tetrahydrobenzo[*d*]azecin-4(1*H*)-one(16)



To a solution of the free amine **6** (135.0 mg, 0.3 mmol) in benzene (30 mL) was added dropwise 2.0 M *i*PrMgCl solution in THF (0.6 mL, 1.2 mmol) at 50 ˚C. The reaction mixture was stirred for 1 h at the same temperature, and then cooled to ambient temperature. The reaction mixture was quenched with saturated aqueous $NaHCO_3$ solution and extracted with EtOAc. The combined organic layers were washed with brine, dried over $MgSO_4$ and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel (9% MeOH in EtOAc) to afford the lactam **16** (88.0 mg, 65%) as a white solid.

$[\alpha]_D^{30}$ = -13.8 (c 0.32 , MeOH); FT-IR (thin film, neat) ν_{max} 3317, 3065, 3032, 2926, 2869, 1639, 1602, 1573, 1544, 1498, 1484, 1454, 1434, 1380, 1362, 1333, 1308 cm^{-1}; ^{1}H-NMR (CD_3OD, 500 MHz) δ 7.42 – 7.25 (m, 10H), 6.57 (s, 1H), 6.51 (s, 1H), 6.19 (d, *J* = 16.3 Hz, 1H), 5.77 – 5.69 (m, 1H), 5.02 (s, 2H), 5.02 (s, 2H), 3.93 (d, *J* = 13.7 Hz, 1H), 3.38 – 3.29 (m, 1H), 3.04 (dd, *J* = 4.5, 11.5 Hz, 1H), 2.84 (t, *J* = 12.5 Hz, 1H), 2.57 – 2.52 (m, 1H), 2.29 (d, *J* = 14 Hz, 1H), 2.15 (q, *J* = 11.5 Hz 1H), 2.15 (s, NH_2); ^{13}C-NMR (CD_3OD, 125 MHz) δ 177.7, 160.4, 158.2, 143.3, 139.7, 139.6, 132.9, 131.7, 130.3, 130.2, 129.6, 129.5, 129.4, 129.1, 123.7, 110.0, 102.0, 72.3, 71.8, 62.3, 57.6, 45.3, 42.4, 33.6; LR-MS (FAB+) *m/z* 429 ($M+H^+$); HR-MS (FAB+) calcd for $C_{27}H_{29}N_2O_3$ ($M+H^+$) 429.2178; found 429.2177.

(3*S*,11b*R*)-3-Amino-9,11-bis(benzyloxy)-2,3,6,7-tetrahydro-1*H*-pyrido[2,1-a]isoquinolin-4(11b*H*)-one (3)

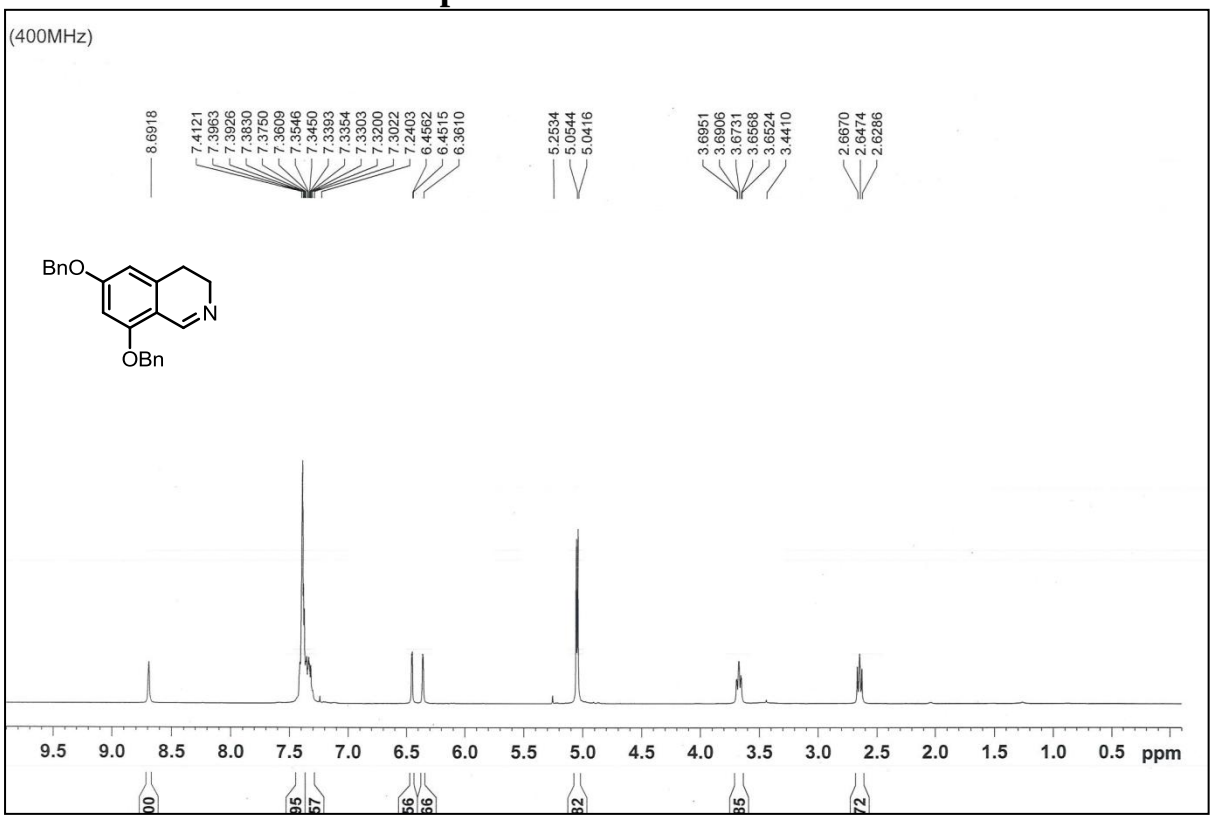


To a solution of lactam **16** (23.0 mg, 0.05 mmol) in benzene (5 mL) was added *p*-toluenesulfonic acid (46.0 mg, 0.27 mmol) and the mixture was stirred for 24 h. The reaction mixture was quenched with saturated aqueous $NaHCO_3$ solution and extracted with EtOAc three times. The combined organic layers were washed with brine, dried over $MgSO_4$ and concentrated in vacuo. The residue was purified by flash column chromatography on silica gel (6% NH_3-MeOH in DCM) to afford benzo[α]quinolizidine **3** (22.0 mg, 95%) as a colorless oil.

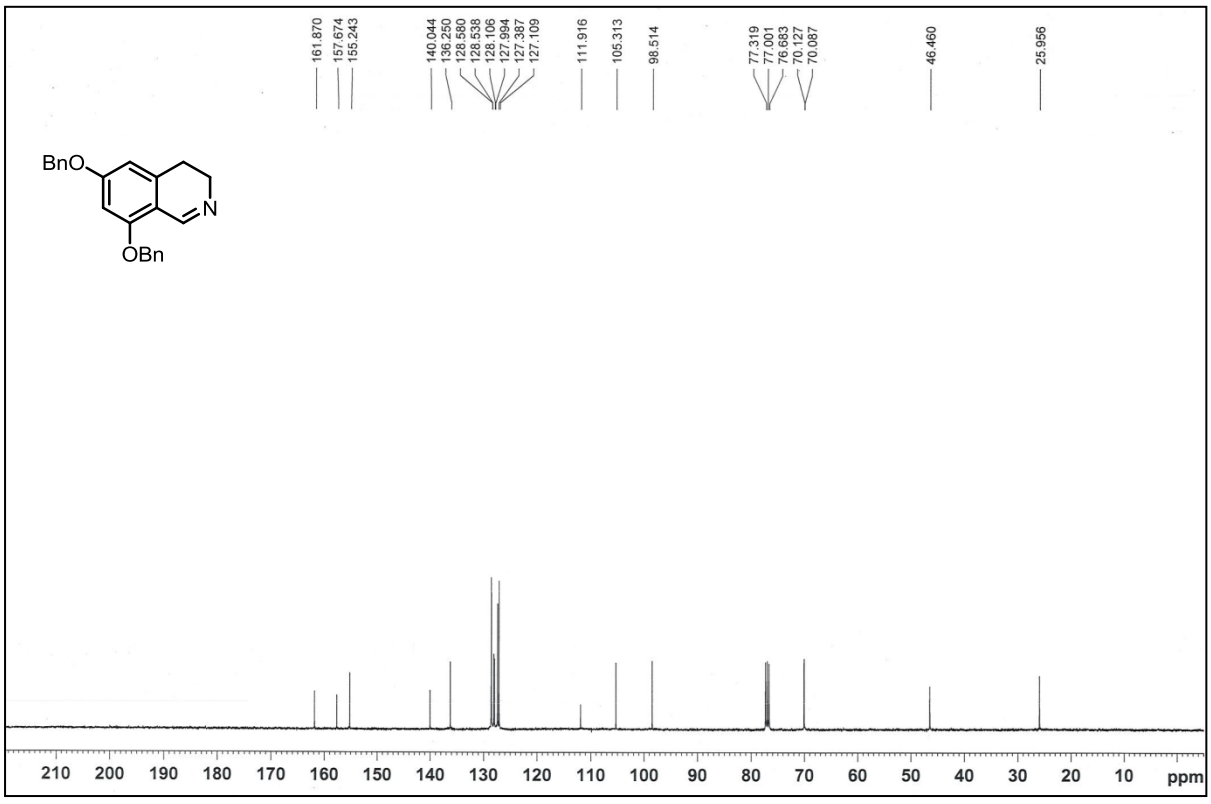
$[\alpha]_D^{24}$ +170.8 (c 0.50, $CHCl_3$), [lit.[1] $[\alpha]_D^{24}$ +182.4 (c 2.10, $CHCl_3$)]; FT-IR (thin film, neat) ν_{max} 2929, 1635, 1606, 1497, 1455, 1433, 1377, 1356, 1306 cm^{-1}; ^{1}H-NMR ($CDCl_3$, 500 MHz) δ 7.41 – 7.29 (m, 10H), 6.49 (d, *J* = 2.0 Hz, 1H), 6.36 (d, *J* = 1.6 Hz, 1H), 5.07 – 4.99 (m, 4H), 4.94 – 4.88 (m, 1H), 4.78 (dd, *J* = 2.3, 10.8 Hz, 1H), 3.33 (dd, *J* = 6.5, 11.7 Hz, 1H), 2.98 (dq, *J* = 3.2, 10.4 Hz, 1H), 2.88 – 2.79 (m, 1H), 2.63 – 2.55 (m, 1H), 2.23 –

2.17 (m, 1H), 1.84 (bs, NH_2), 1.67 – 1.58 (m, 1H), 1.44 – 1.35 (m, 1H); ^{13}C-NMR ($CDCl_3$, 125 MHz) δ 172.3, 158.1, 156.7, 138.0, 136.7, 136.5, 128.6, 128.5, 128.0, 128.0, 127.4, 126.9, 118.5, 106.1, 99.1, 70.1, 70.1, 56.0, 52.6, 39.0, 30.5, 29.4, 28.5; LR-MS (FAB+) *m/z* 429 ($M+H^+$); HR-MS (FAB+) calcd for $C_{27}H_{29}N_2O_3$ ($M+H^+$) 429.2178; found 429.2182.

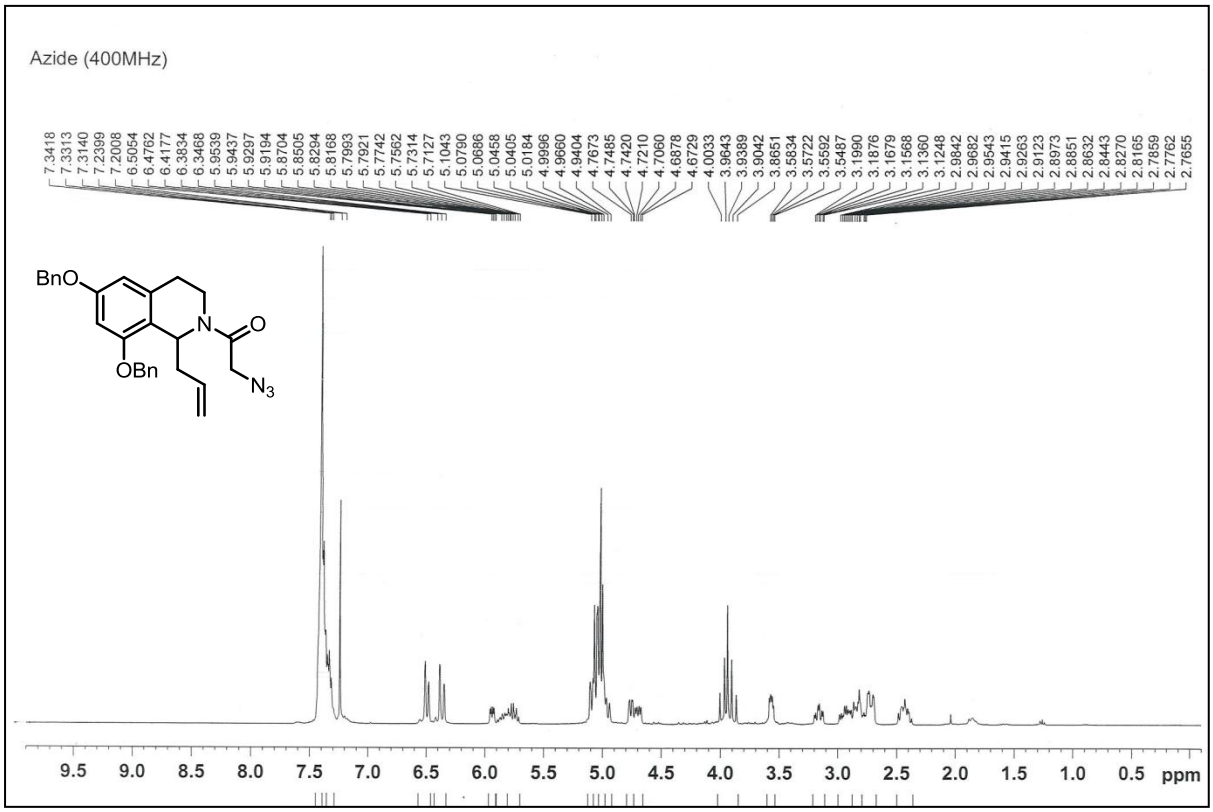
3. ^{1}H-NMR and ^{13}C-NMR Spectra



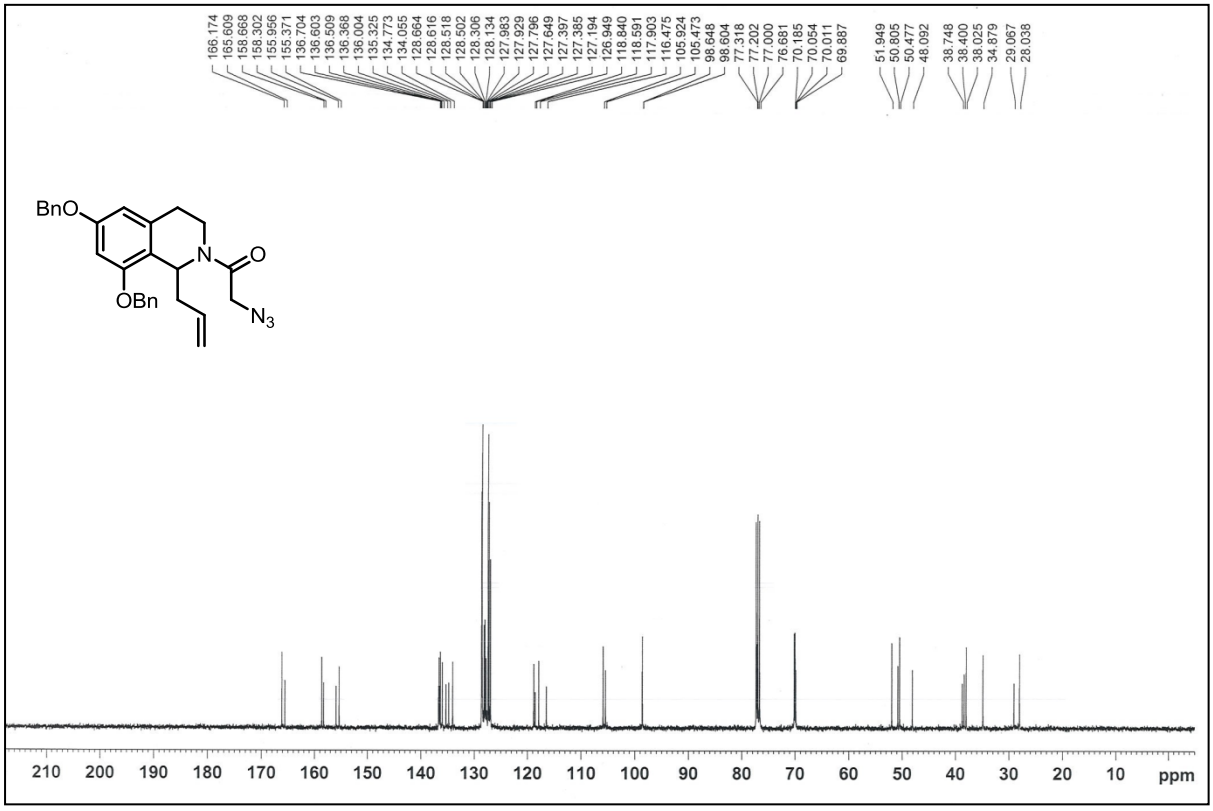
▲ ^{1}H-NMR (400 MHz, $CDCl_3$)



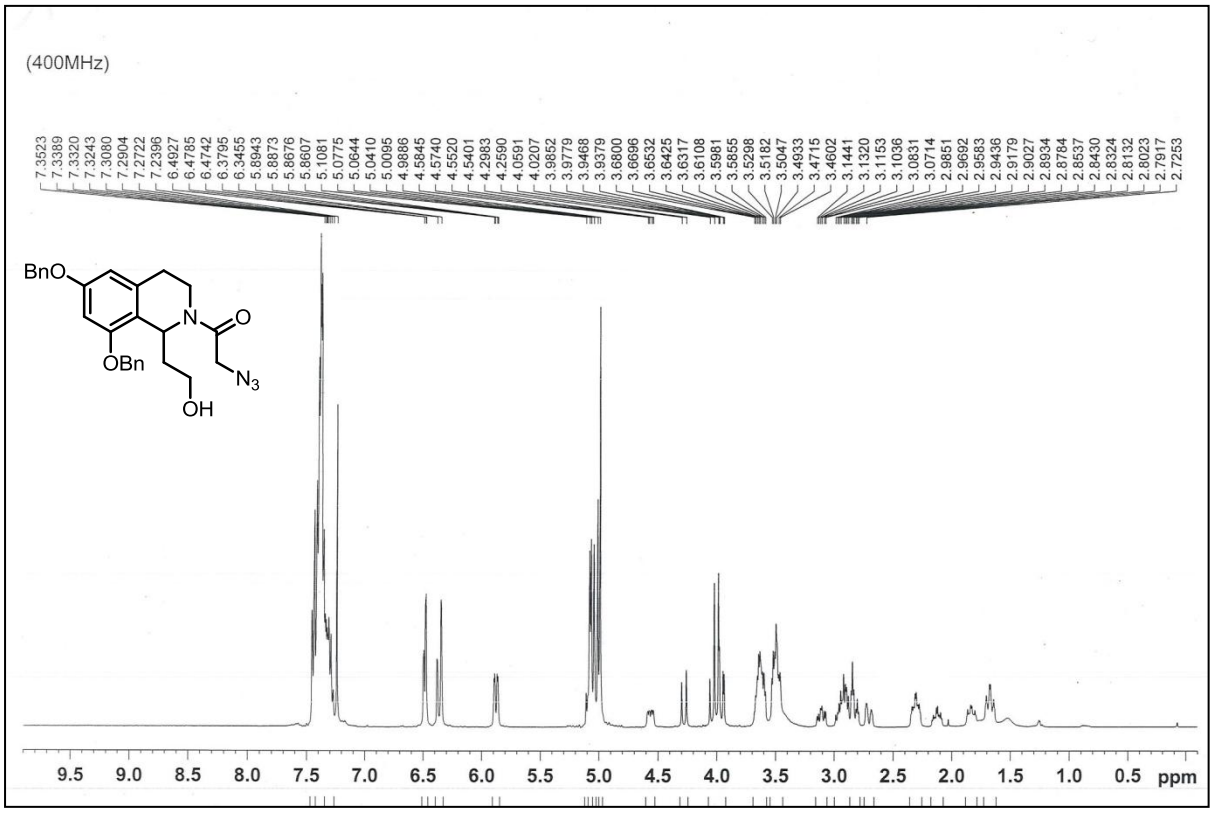
▲ ^{13}C-NMR (100 MHz, $CDCl_3$)



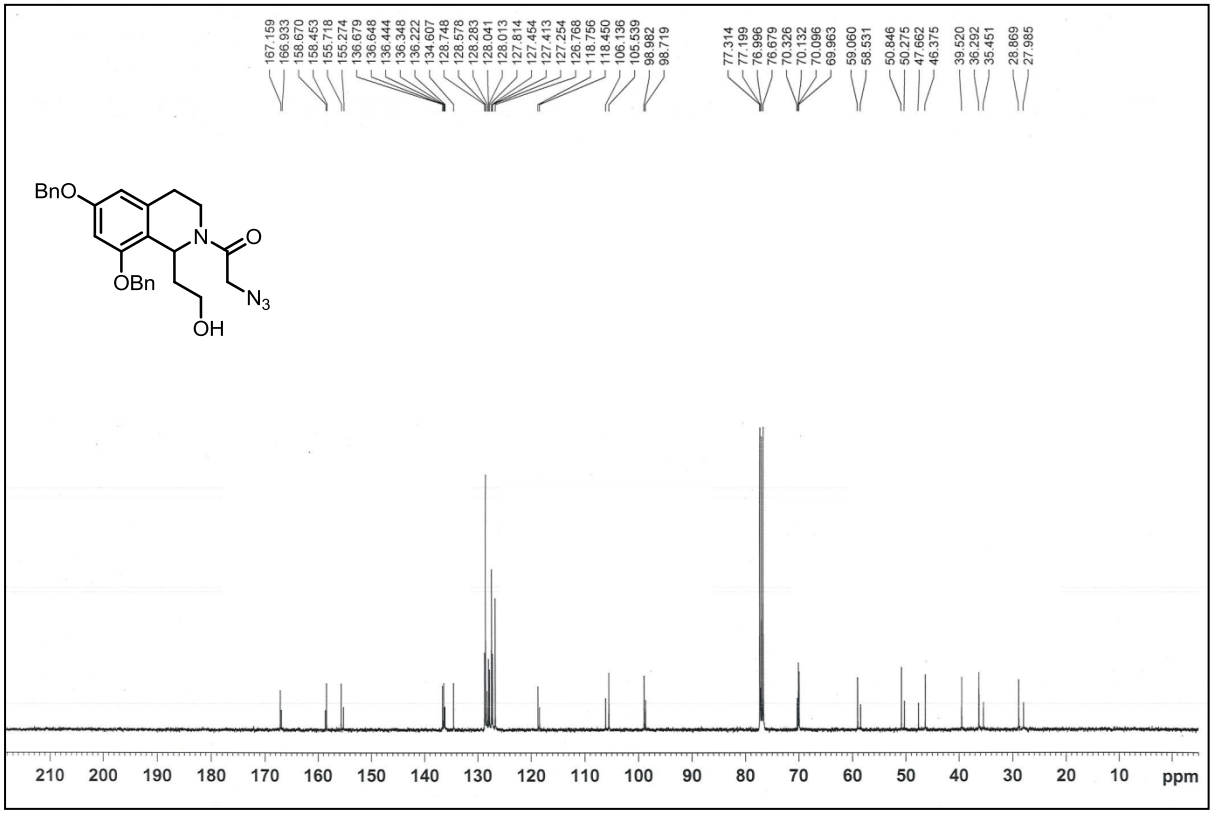
▲ ^{1}H-NMR (400 MHz, $CDCl_3$)



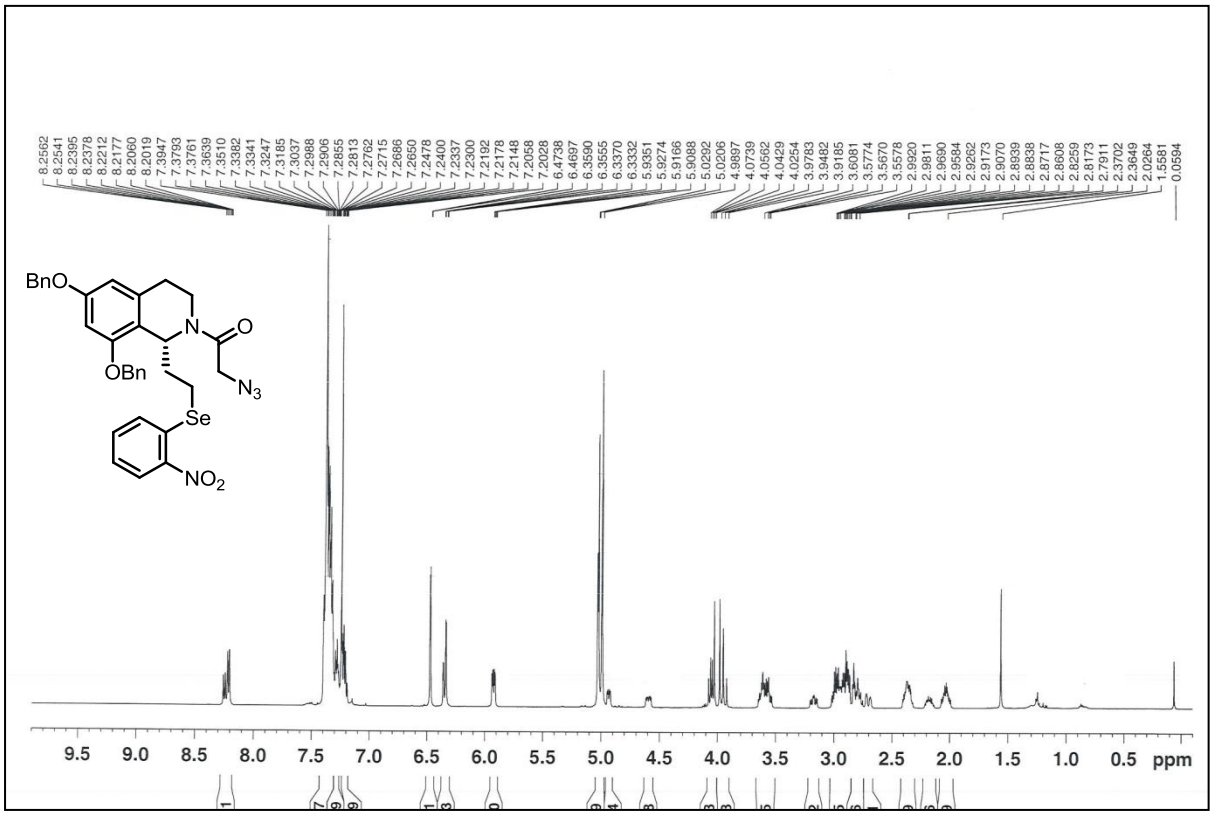
▲ ^{13}C-NMR (100 MHz, $CDCl_3$)



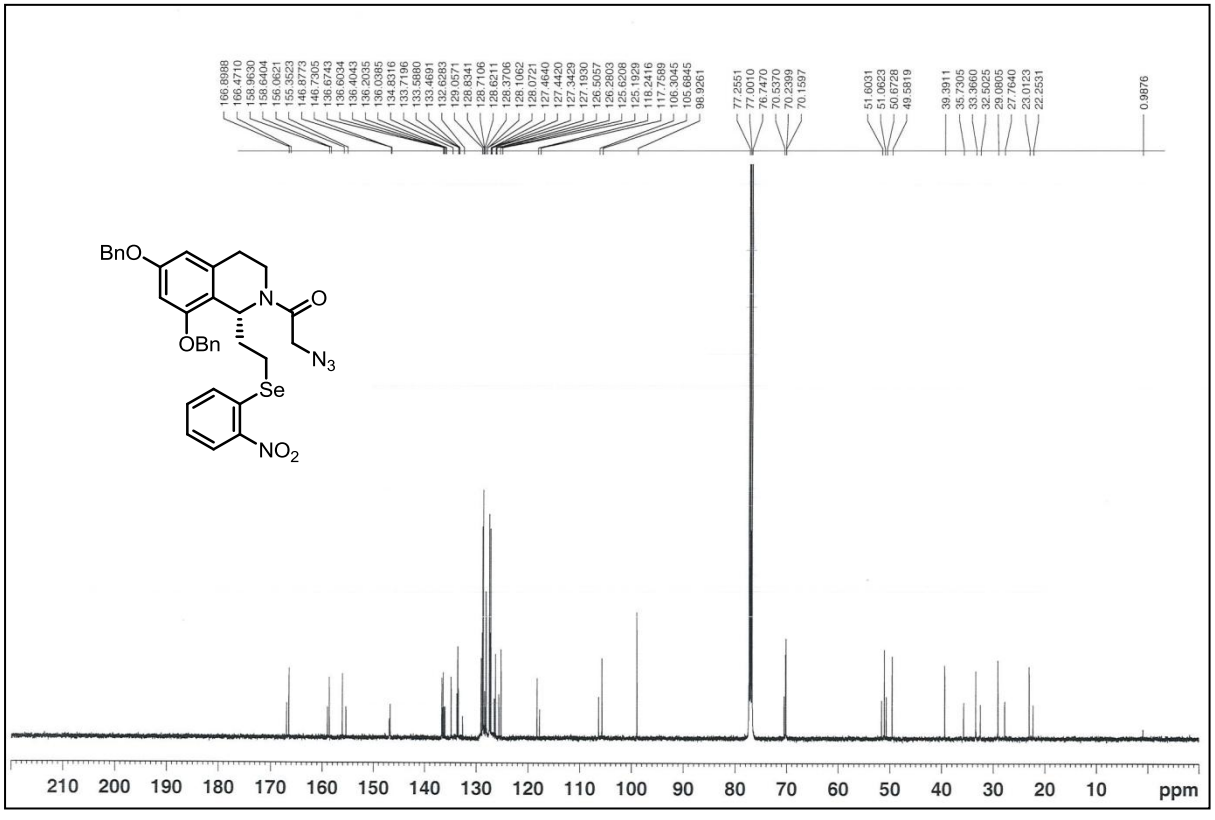
▲ ^{1}H-NMR (400 MHz, $CDCl_3$)



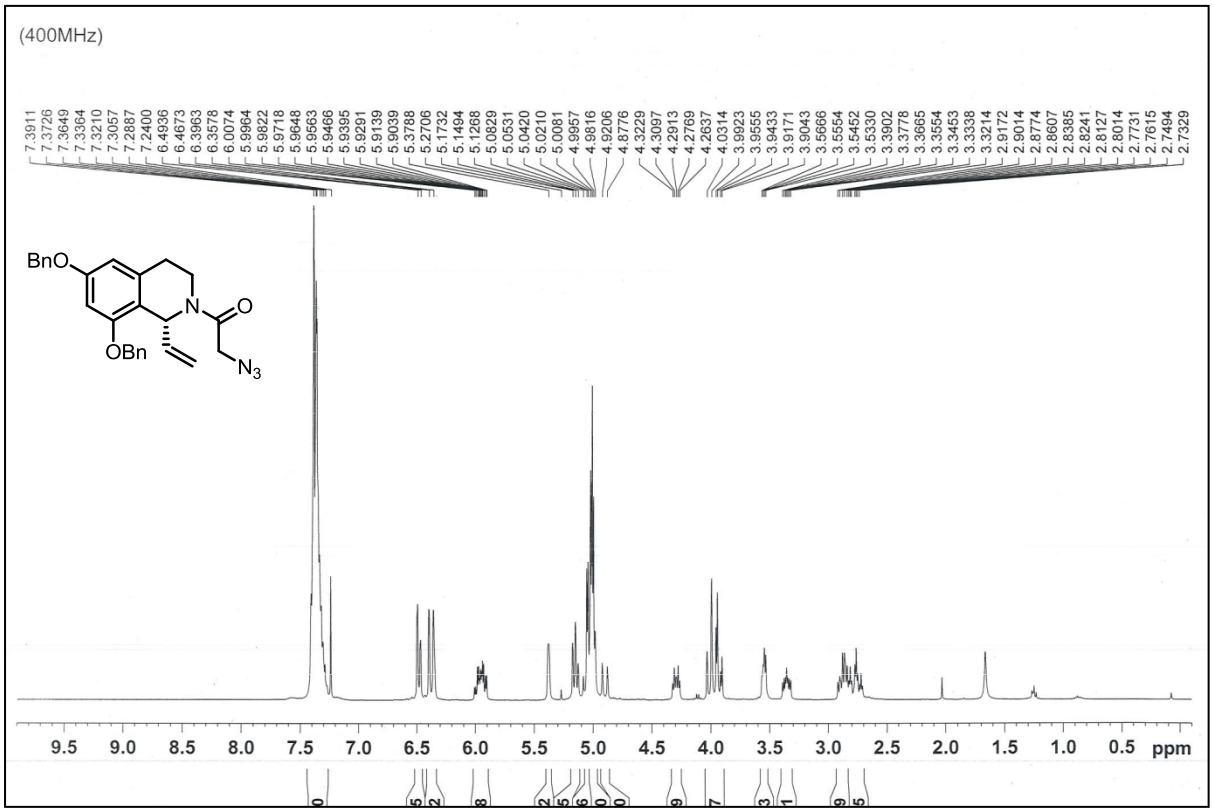
▲ ^{13}C-NMR (100 MHz, $CDCl_3$)



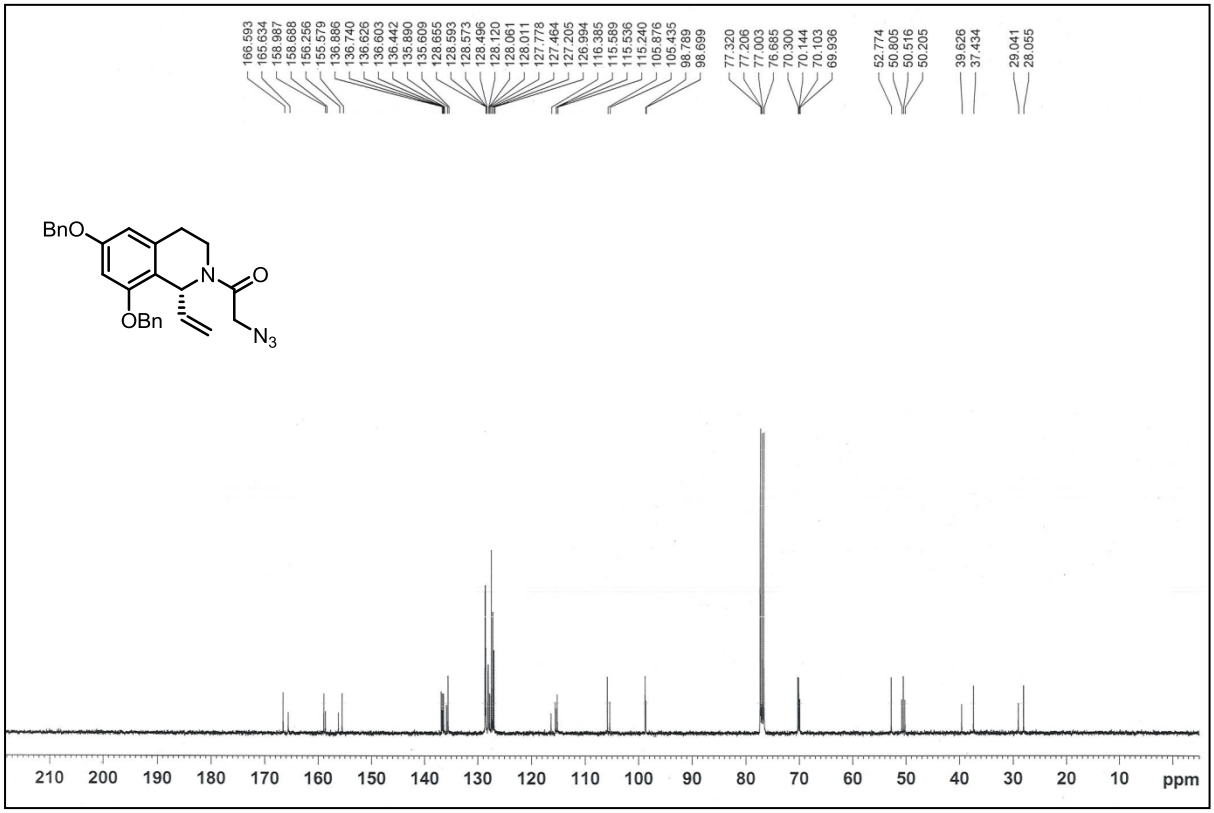
▲ ^{1}H-NMR (500 MHz, $CDCl_3$)



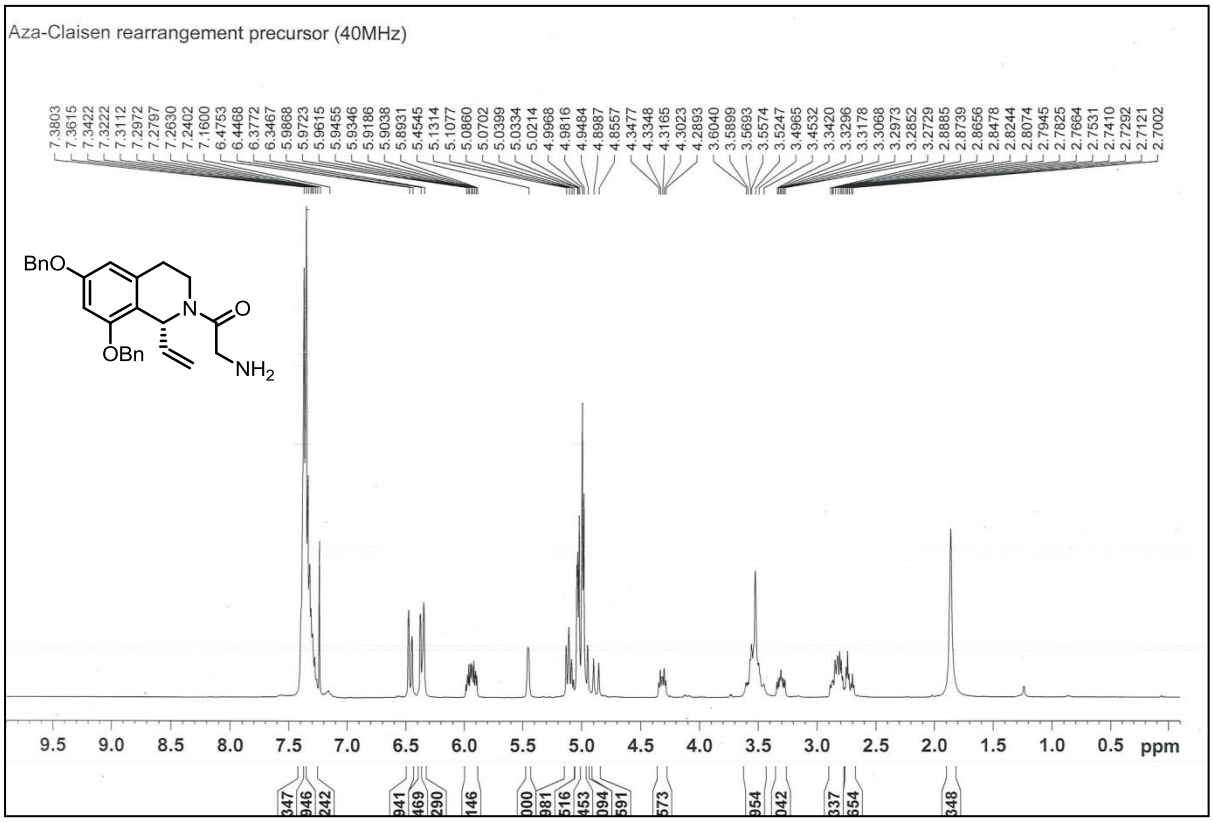
▲ ^{13}C-NMR (125 MHz, $CDCl_3$)



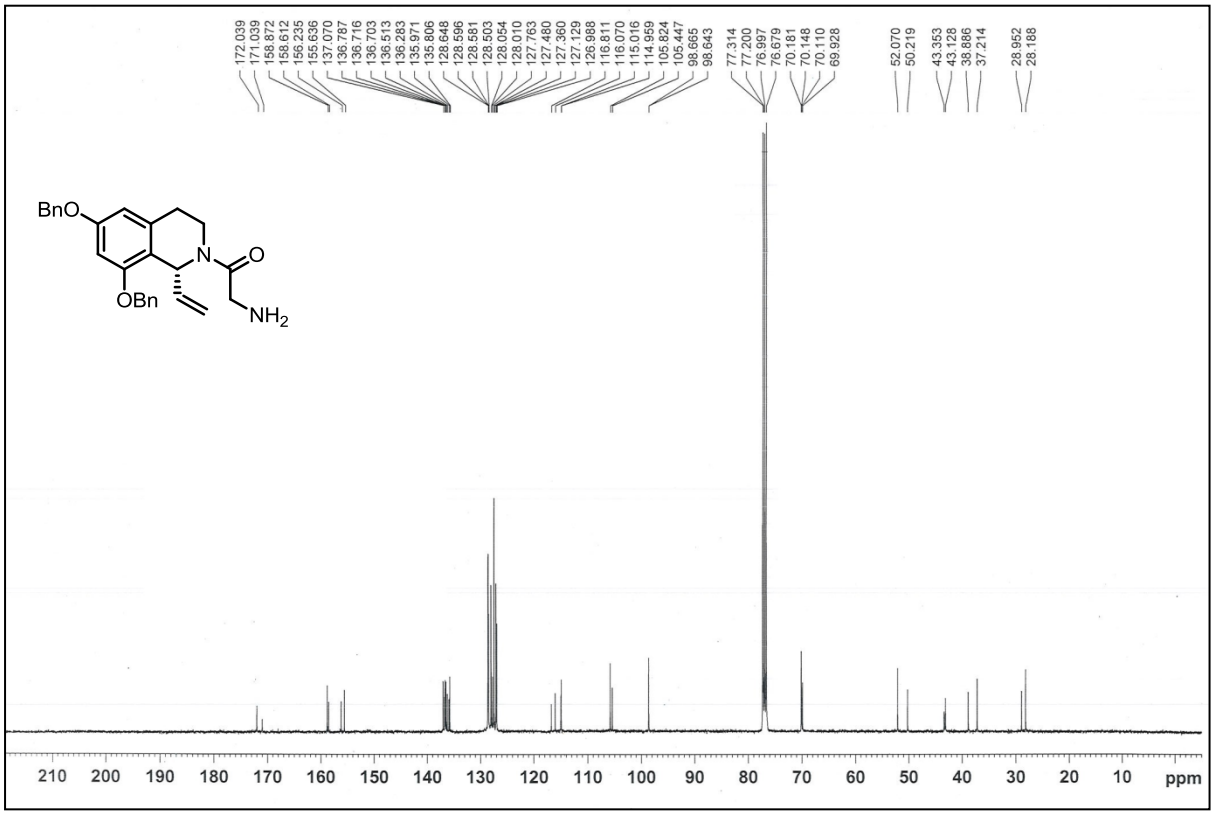
▲ ^{1}H-NMR (400 MHz, $CDCl_3$)



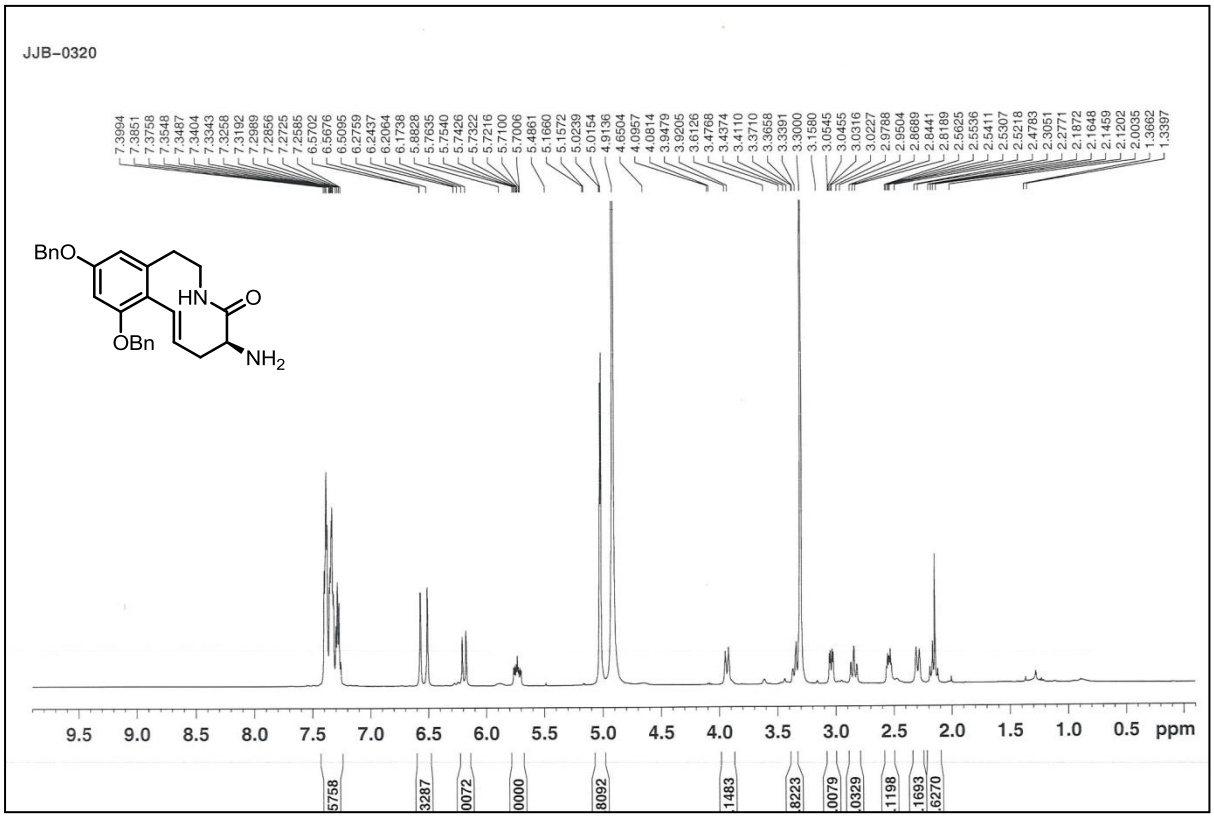
▲ ^{13}C-NMR (100 MHz, $CDCl_3$)



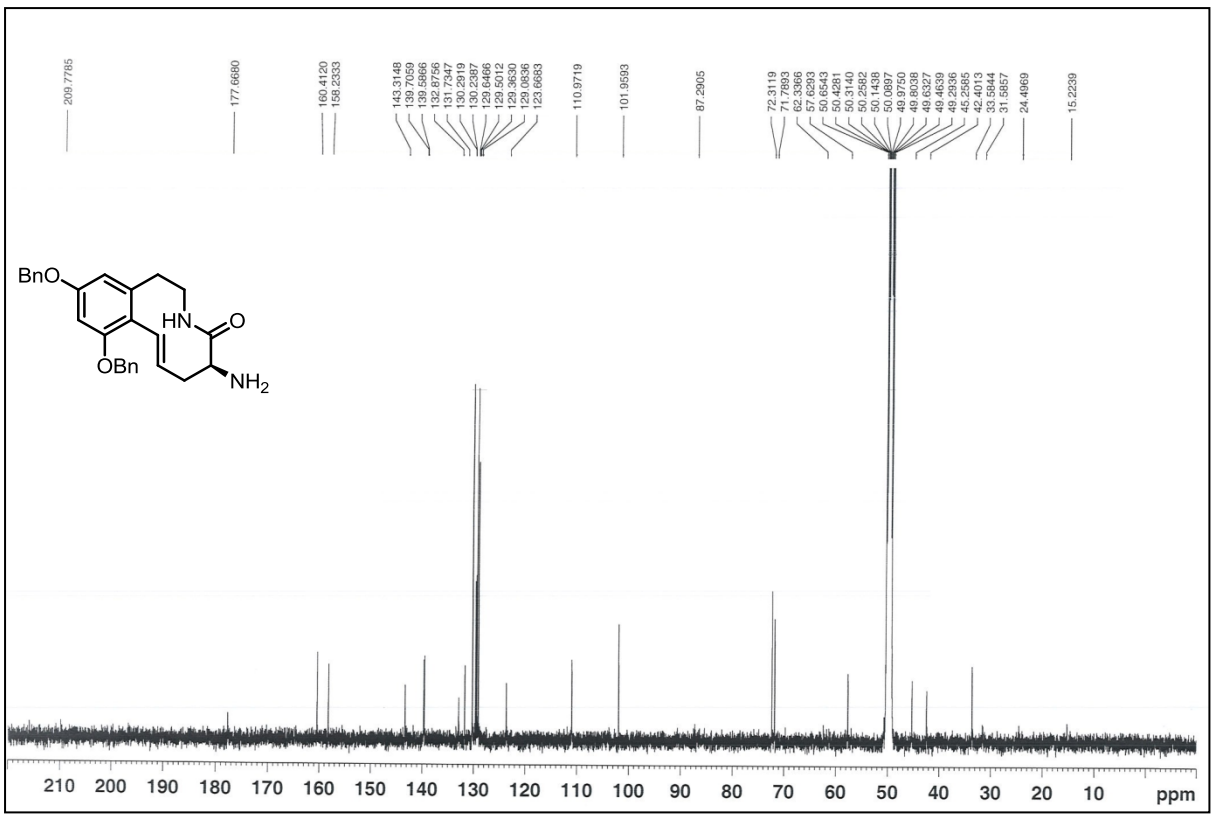
▲ ^{1}H-NMR (400 MHz, $CDCl_3$)



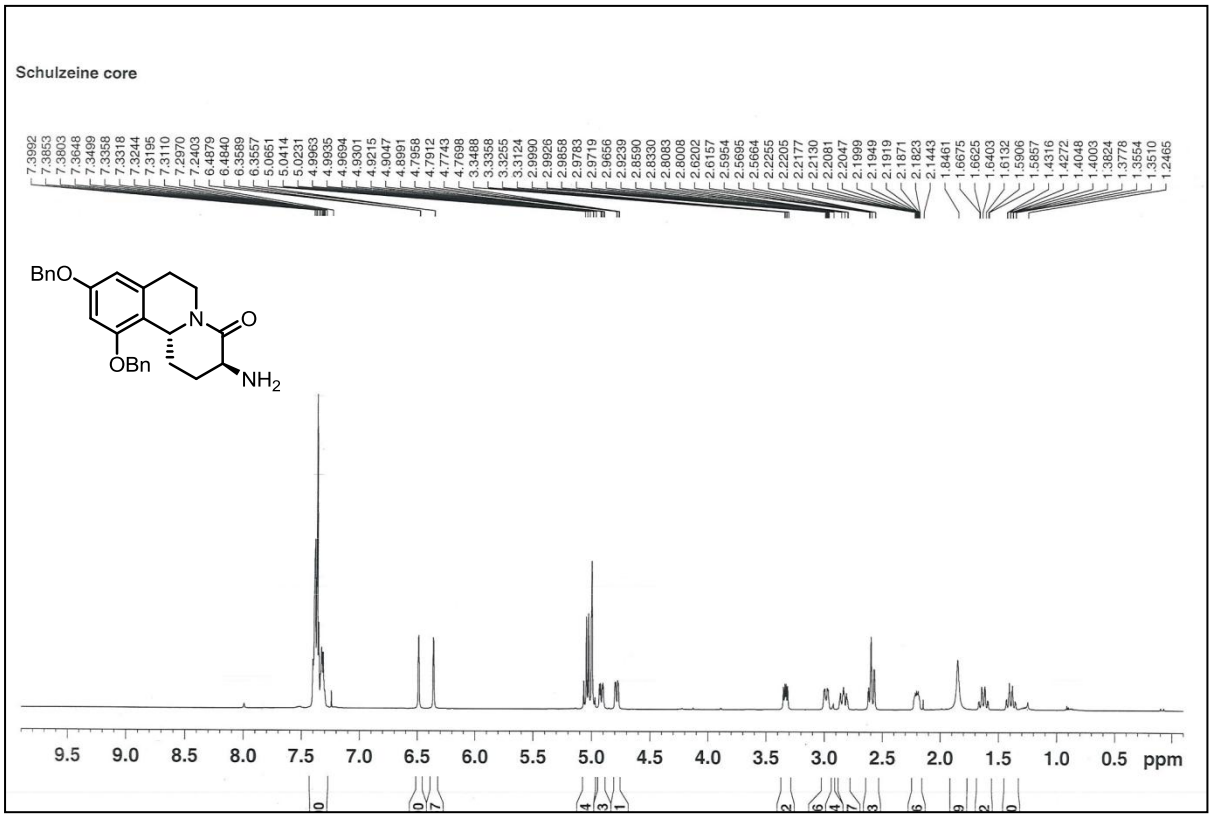
▲ ^{13}C-NMR (100 MHz, $CDCl_3$)



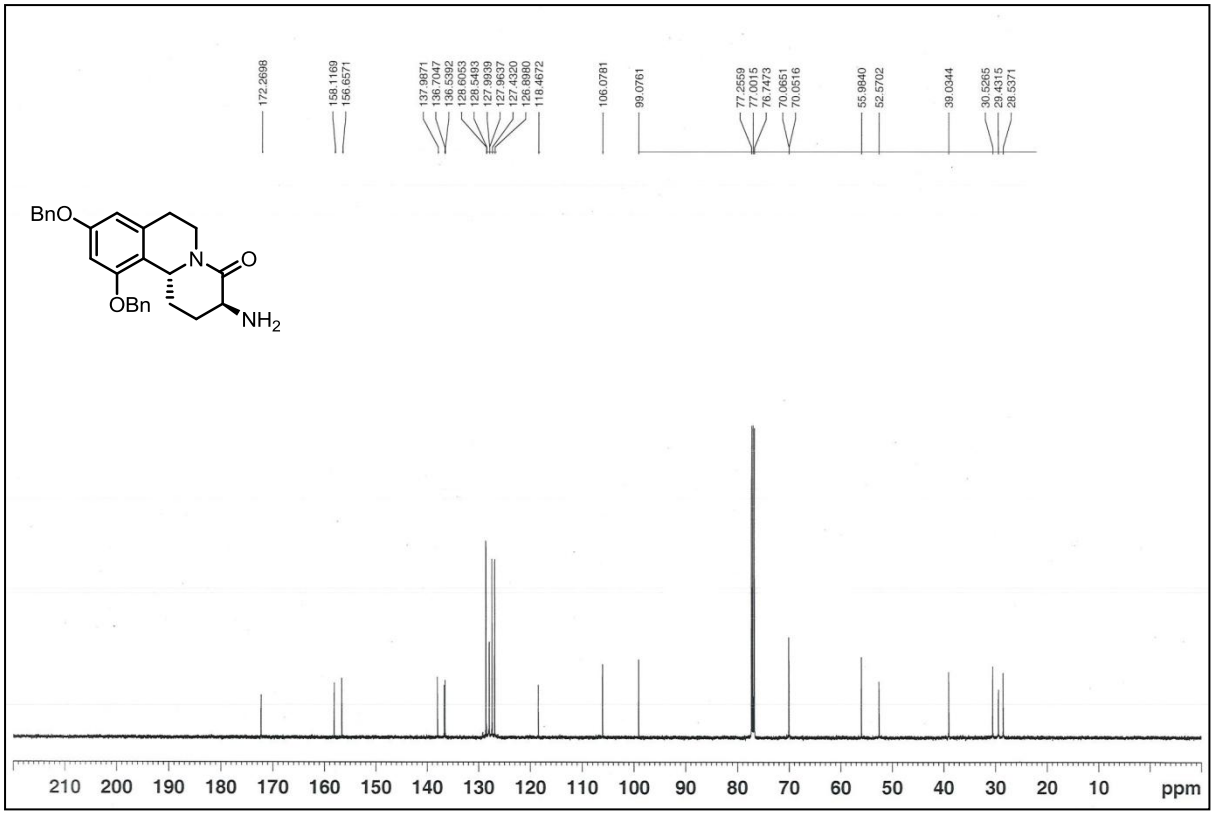
▲ ^{1}H-NMR (500 MHz, $CDCl_3$)



▲ ^{13}C-NMR (125 MHz, $CDCl_3$)



▲ ^{1}H-NMR (500 MHz, $CDCl_3$)



▲ ^{13}C-NMR (125 MHz, $CDCl_3$)

4. HPLC data

Data File: c:\gilson\unipoint\proh-hex.316\proh-hex.gdt
Date acquired: Mon Dec 26 2011 12:30:46
Control Method: C:\GILSON\UNIPOINT\PROH-HEX.GCT
Analysis Method: c:\gilson\unipoint\proh-hex.gan

Analysis Method Events
0.00 Enable Integration initial default
0.00 Disable Negative Peak Integration initial default
0.00 Peak Width 1 initial default
0.00 Peak Sensitivity 50 initial default
Channel Scales
<Auto range all channels>
uv/vis-1510.00 to 10.00 mVolt (0 %offset), Data rate: 20.00 (points/second) <Auto zero> <Analysis Channel>

Peak Table
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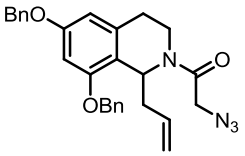
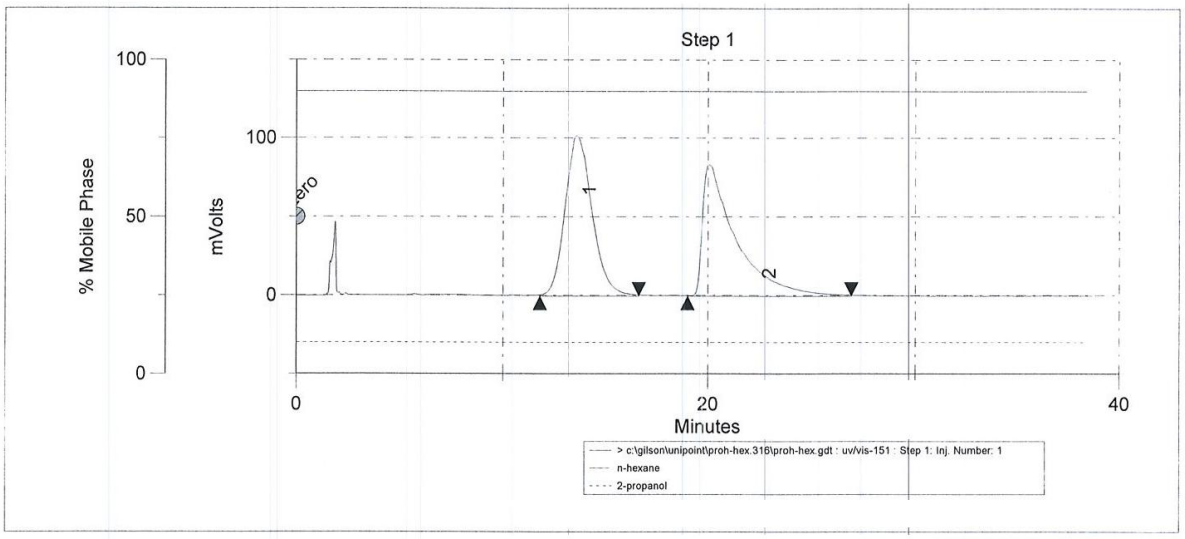
Peak Detection Parameters
Relative Error: 5 (%) Absolute Error: 0.1 (min)





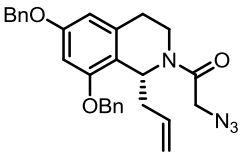
	R. Time	Area	Area %	Height
1	13.59	15698958.00	49.91	102.17
2	20.04	15758231.00	50.09	83.74

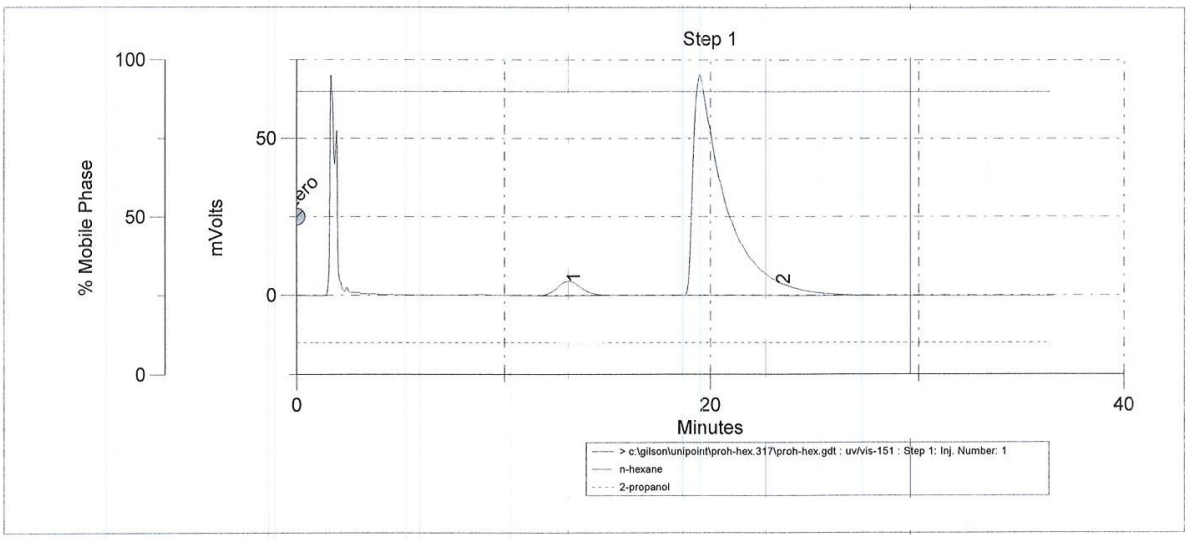
Data File: c:\gilson\unipoint\proh-hex.317\proh-hex.gdt
Date acquired: Mon Dec 26 2011 15:38:14
Control Method: C:\GILSON\UNIPOINT\PROH-HEX.GCT
Analysis Method: c:\gilson\unipoint\proh-hex.gan

Analysis Method Events
0.00 Enable Integration initial default
0.00 Disable Negative Peak Integration initial default
0.00 Peak Width 1 initial default
0.00 Peak Sensitivity 50 initial default
Channel Scales
<Auto range all channels>
uv/vis-1510.00 to 10.00 mVolt (0 %offset), Data rate: 20.00 (points/second) <Auto zero> <Analysis Channel>

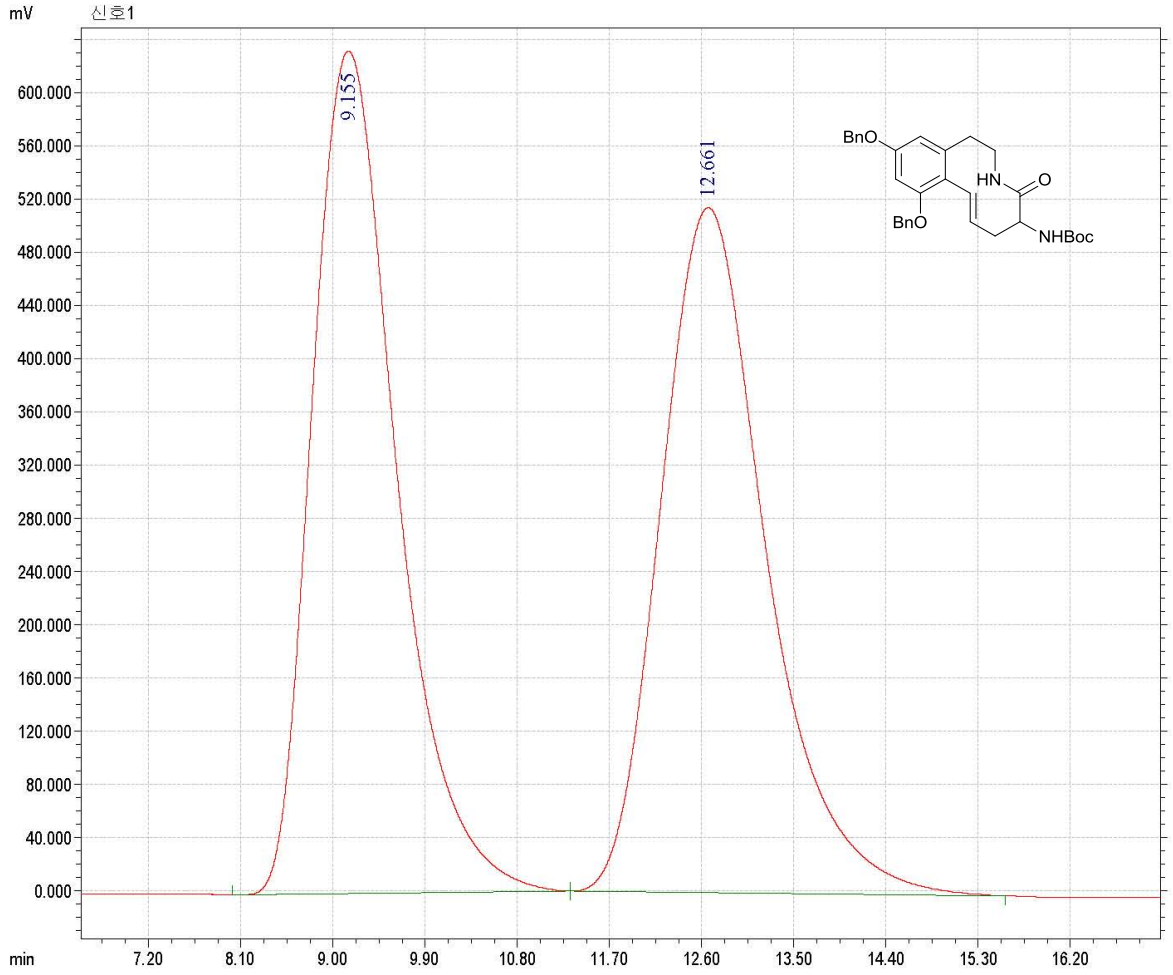
Peak Table
<None>

Peak Detection Parameters
Relative Error: 5 (%) Absolute Error: 0.1 (min)



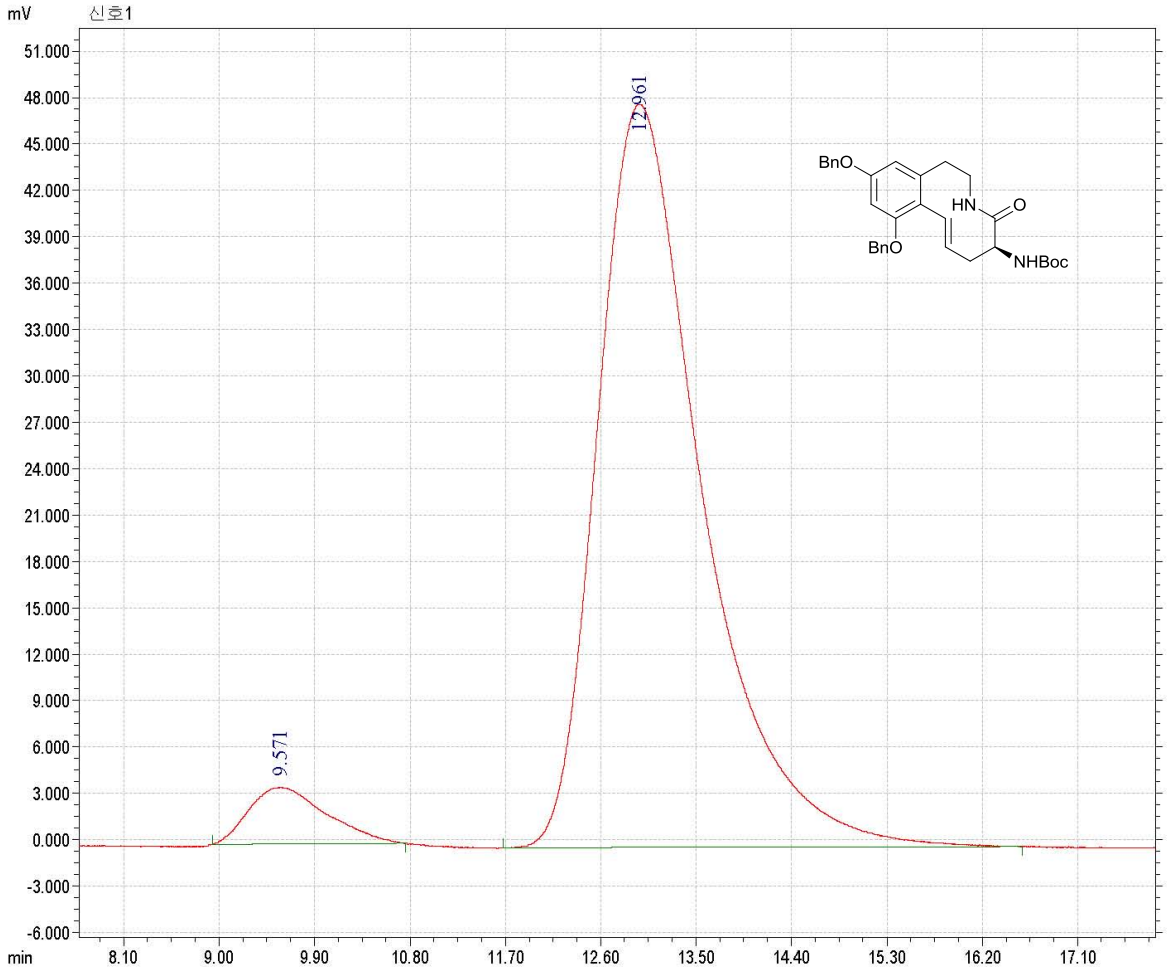


	R. Time	Area	Area %	Height
1	13.21	647997.88	4.95	4.64
2	19.48	12435641.00	95.05	70.45



	R. Time	Area	Area%	
	9.155	36242934.89	49.633	BMB
	12.661	36778251.90	50.367	BMB
Σ		73021186.80	100.000	

데이터비고:
CHIRALPAK AD-H, Hexane:i-PrOH = 90:10, Flow = 1.0ml/min, pressure = 710



	R. Time	Area	Area%	
	9.571	185005.74	5.317	BMB
	12.961	3294280.49	94.683	BMB
Σ		3479286.24	100.000	

데이터비고:
CHIRALPAK AD-H, Hexane:i-PrOH = 90:10, Flow = 1.0ml/min, pressure = 710

5. Reference

1. E. G. Bowen, D. J. Wardrop, *J. Am. Chem. Soc.* 2009, **131**, 6062