

“A novel, concise and efficient protocol for non natural piperidine compounds”

Subhash P. Chavan,* Nilesh B. Dumare and Kailash P. Pawar

Division of Organic Chemistry, CSIR-NCL (National Chemical Laboratory), Pune-411008, India

sp.chavan@ncl.res.in

Supporting Information:

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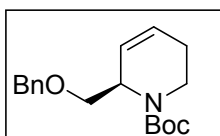
SI:-2	General
SI:-3-9	Experiemental
SI:-10-24	NMR spectra: copies of ^1H and ^{13}C
SI:-25-27	HPLC chromatograms

General:

Melting points are recorded using Buchi B-540 or M-560 melting point apparatus in capillary tubes and are uncorrected and the temperatures are in centigrade scale. IR spectra were recorded on a Perkin-Elmer Infrared Spectrophotometer Model 68B or on a Perkin-Elmer 1615 FT Infrared spectrophotometer. ^1H (200 and 400 MHz) and ^{13}C (50 and 100 MHz) NMR spectra were recorded on Bruker and Bruker Advance 400 spectrometers. The chemical shifts (δ ppm) and coupling constants (Hz) are reported in the standard fashion with reference to chloroform, δ 7.27 (for ^1H) or the central line (77.0 ppm) of CDCl_3 (for ^{13}C). In the ^{13}C NMR spectra, the nature of the carbons (C, CH, CH_2 , or CH_3) was determined by recording the DEPT-135 spectra. The reaction progress was monitored by the TLC analysis using thin layer plates precoated with silica gel 60 F254 (Merck) and visualized by fluorescence quenching or iodine or by charring after treatment with *p*-anisaldehyde. Merck's flash silica gel (300-400 mesh) was used for column chromatography. All small scale dry reactions were carried out using standard syringe-septum technique. Low temperature reactions were carried out using a bath made of sodium chloride and ice. Dry THF was obtained by distillation over sodium/benzophenone ketyl. Dry DCM was prepared by distillation over phosphorous pentoxide or calcium hydride. Dry benzene was obtained by distillation over sodium. All other reagents and solvents were used as received from the manufacturer, unless otherwise specified. All air and water sensitive reactions were performed in flasks flame dried under positive flow of argon and conducted under an argon atmosphere.

Experimental:

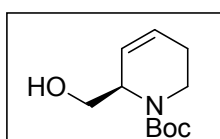
(*R*)-*tert*-Butyl 2-(benzyloxymethyl)-5,6-dihydropyridine-1(2*H*)-carboxylate (**16**)



A mixture of alcohol **9** (0.5 g, 1.55 mmol), PPh₃ (1.34 g, 5.11 mmol), imidazole (0.33 g, 4.96 mmol) and I₂ (0.86 g, 3.41 mmol) in anhydrous toluene (10 mL) was refluxed under nitrogen atmosphere for 30 minutes. After completion of the reaction (TLC), the reaction mixture was diluted with ethyl acetate (60 mL) and washed with Na₂S₂O₃ solution, water, followed by brine. The organic solvent was dried over anhydrous sodium sulphate, filtered and concentrated under reduced pressure. The residue was purified on silica gel by eluting with light petroleum: EtOAc (9:1) to afford **16** as colorless syrup (0.40 g, 85% yield).

Yield : 85%; [α]_D²⁵ : +106.31 (c 1.9, CHCl₃); IR (CHCl₃, cm⁻¹) : 1692, 1418, 1172; ¹H NMR (200 MHz, CDCl₃): δ 1.45 (s, 9H), 1.88-2.04 (m, 1H), 2.13-2.30 (m, 1H), 2.94 (bs, 1H), 3.47-3.61 (m, 2H), 4.15 (bs, 1H), 4.49-4.66 (m, 3H), 5.70-5.79 (m, 1H), 5.90-5.98 (m, 1H), 7.23-7.35 (m, 5H); ¹³C NMR (50 MHz, CDCl₃+CCl₄): δ 24.9, 28.5, 71.3, 73.0, 79.5, 126.7, 127.4, 128.3, 138.3, 154.5; ESIMS (*m/z*): 326.28(M+Na)⁺; HRMS calculated for [C₁₈H₂₅NO₃+Na]⁺ 326.1727; found: 326.1734.

(*R*)-*tert*-Butyl 2-(hydroxymethyl)-5,6-dihydropyridine-1(2*H*)-carboxylate (**8**)

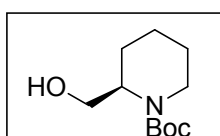


Benzyl ether **16** (0.100 g, 0.33 mmol, 1.0 eq.) was stirred in anhydrous THF (5 ml) at -78 °C under an atmosphere of nitrogen. Ammonia gas was condensed (3 mL) by passing into the precooled dewar flask, fitted with a dewar condenser (-78 °C). Sodium (0.023 g, 0.99 mmol, 3.0 eq.) was added and the deep blue mixture stirred for 10 min at -78 °C. After 10 min, solid ammonium chloride (5 g) was added to the reaction mixture. Ammonia was then allowed to evaporate by removing the cooling bath, and the product was extracted with ethyl acetate (4 x 50 mL). The organic layers were dried over anhydrous sodium sulfate, filtered and concentrated under reduced pressure to yield the crude product as yellow syrup. The residue was purified on silica gel by eluting with light petroleum: EtOAc (7:3) to afford homoallylic alcohol **8** as colorless oil (0.061 g, 88%).

Yield : 88%; $[\alpha]_{\text{D}}^{25}$: +200 (c 0.6, CHCl₃); lit.⁶ For comp-16 $[\alpha]_{\text{D}}^{24}$ +230.7 (c 1.75, CHCl₃);

IR (CHCl₃, cm⁻¹) : 3440, 2975, 1693, 1674, 1423; ¹H NMR (500 MHz, CDCl₃): δ 1.47 (s, 9H), 1.95-2.00 (m, 1H), 2.16-2.26 (m, 1H), 2.95 (br, 1H), 3.64 (m, 1H), 3.68-3.70 (m, 1H), 4.08 (bs, 1H), 4.53 (bs, 1H), 5.62 (dt, *J* = 10, 2.9 Hz, 1H), 5.95 (m, 1H); ¹³C NMR (125 MHz, CDCl₃+CCl₄): δ 24.9, 28.5, 38.1, 54.3, 64.9, 80.1, 124.8, 127.8, 156.2; ESIMS (*m/z*): 236.1 (M+Na)⁺.

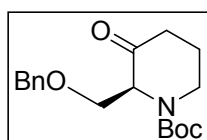
(*R*)-*tert*-Butyl 2-(hydroxymethyl)piperidine-1-carboxylate (17)



Olefin **8** (0.100 g, 0.46 mmol) and 10 % Pd/C in methanol (5 ml) were subjected under hydrogen atmosphere at 30 *psi* at room temperature (25 °C) for 3 h. The reaction mixture was filtered through celite and the celite layer was washed thoroughly with methanol (2 X 20 mL) and concentrated under reduced pressure. The residue thus obtained was purified by flash silica gel column chromatography using light petroleum: ethyl acetate (1:1) as an eluent to furnish alcohol **17** (0.092 g, 92% yield) as a colorless liquid.

Yield : 92%; $[\alpha]_{\text{D}}^{25}$: +38.9 (c 1, CHCl₃); lit.⁸ for *ent*-20 -40.1 (c 1, CHCl₃); IR (CHCl₃, cm⁻¹): 3442, 2940, 2890, 1655, 1422, 1370, 1280, 1170, 1150, 1060; ¹H NMR (200 MHz, CDCl₃): δ 1.46 (s, 9H), 1.55-1.68 (m, 4H), 2.11 (bs, 2H), 2.87 (m, 1H), 3.59 (dd, *J* = 10, 6 Hz, 1H), 3.75-3.85 (m, 1H), 3.93 (d, *J* = 12 Hz, 1H), 4.72-4.85 (m, 1H); ¹³C NMR (50 MHz, CDCl₃+CCl₄): δ 19.31, 24.8, 25.2, 28.3, 39.8, 52.1, 60.1, 79.5, 155.8; HRMS calculated for [C₁₁H₂₁NO₃+H]⁺ 216.1594; found: 216.1600.

(*S*)-*tert*-Butyl 2-((benzyloxy)methyl)-3-oxopiperidine-1-carboxylate (18)

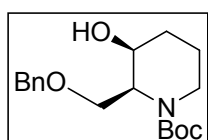


To a solution of alcohol **9** (0.100 g, 0.311 mmol) in ethyl acetate (5 mL) was added IBX (0.17 g, 0.622 mmol) at room temperature under nitrogen atmosphere. The reaction mixture was allowed to reflux for 3 h. After completion of reaction (monitored by TLC), the reaction mixture was allowed to cool at room temperature. The reaction mixture was filtered through a Whatmann filter paper. The combined organic layer was washed with saturated solution of NaHCO₃ (20 mL) followed by water (30 mL). The organic layer was dried over anhydrous sodium sulphate, filtered and concentrated under reduced pressure to provide crude residue. The residue was purified by

flash silica gel column chromatography using ethyl acetate and pet ether (3: 7) as an eluent to give keto carbamate **18** as colorless syrup (0.89 g, 90%).

Yield : 90%; $[\alpha]_{\text{D}}^{25}$: +30.2 (c 1.27, CHCl₃); IR (CHCl₃, cm⁻¹): 1717, 1693, 1404, 1117; ¹H NMR (200 MHz, CDCl₃): δ 1.50 (s, 9H), 1.89-2.17 (m, 2H), 2.45-2.60 (m, 2H), 3.49 (bs, 1H), 3.69-4.32 (m, 3H), 4.52 (s, 2H), 4.63 (bs, 1H), 7.25-7.42 (m, 5H); ¹³C NMR (50 MHz, CDCl₃+CCl₄): δ 22.1, 28.5, 38.8, 72.3, 73.4, 80.2, 127.4, 127.7, 128.5, 207.2.

(2S,3S)-*tert*-Butyl 2-((benzyloxy)methyl)-3-hydroxypiperidine-1-carboxylate (**19**)

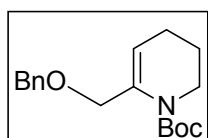


To a solution of keto carbamate **18** (0.1 g, 0.313 mmol) in methanol (5 mL) was added NaBH₄ (23.6 mg, 0.626 mmol) at 0 °C portionwise. The reaction mixture was allowed to stir for 30 min at room temperature. After completion of reaction, the reaction mixture was concentrated under reduced pressure. The aq. solution of NH₄Cl was added to the semisolid mass and allowed to stir for 30 min and the reaction mixture was extracted with DCM (3 X 30 mL). The combined organic layer was dried over anhydrous sodium sulphate, filtered and concentrated under reduced pressure to afford a residue. The residue was purified by flash silica gel column chromatography using ethyl acetate and pet. ether as an eluent to provide *cis* alcohol **19** (88.5 mg, 88%) as colorless syrup.

Yield : 88%; $[\alpha]_{\text{D}}^{25}$: +36.8 (c 0.5, CHCl₃); lit. ¹ for *ent*-17 $[\alpha]_{\text{D}}^{23}$ -38.9 (c = 0.76, CHCl₃).

¹H NMR (200 MHz, CDCl₃): δ 1.44 (s, 9H), 1.48-1.94 (m, 4H), 2.66 (br *t*, 1H), 3.61-3.69 (m, 1H), 3.76-3.97 (m, 3H), 4.53 (d, *J* = 1.5 Hz, 2H), 4.58-4.70 (m, 1H), 7.30 (s, 5H); ¹³C (100 MHz, CDCl₃+CCl₄): δ 24.0, 28.5, 28.8, 39.2, 53.2, 66.6, 69.5, 73.3, 79.8, 127.7, 127.8, 128.5, 137.7, 154.9; HRMS calculated for [C₁₈H₂₇NO₄+H]⁺ 322.2013; found: 322.2008.

tert-Butyl 6-((benzyloxy)methyl)-3,4-dihydropyridine-1(2*H*)-carboxylate (**20**)

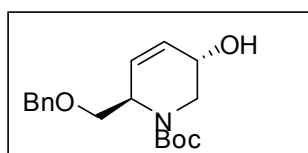


A mixture of alcohol **19** (0.05 g, 0.155 mmol), PPh₃ (0.134 g, 0.511 mmol), imidazole (0.033 g, 0.496 mmol) and I₂ (0.086 g, 0.341 mmol) in anhydrous toluene (10 mL) was refluxed under nitrogen atmosphere for 30 minutes. After completion of the reaction (TLC), the reaction mixture was diluted with ethyl acetate (60 mL) and washed with Na₂S₂O₃ solution followed by water. The organic layer was dried over anhydrous sodium sulphate, filtered and concentrated under reduced

pressure. The residue was purified on silica gel by eluting with light petroleum: EtOAc (9:1) to afford olefin **20** (0.011 g) and olefin **16** (0.017 g) in 60% yield.

Yield : 24%; IR (CHCl₃, cm⁻¹): 1698, 1654, 1400, 1162; ¹H NMR (200 MHz, CDCl₃): δ 1.48 (s, 9H), 1.78-1.81 (m, 2H), 2.09-2.18 (m, 2H), 3.50-3.57 (m, 2H), 4.33 (s, 2H), 4.48 (s, 2H), 5.27 (t, J = 3.5 Hz, 1H), 7.30-7.33 (m, 5H); ¹³C NMR (100 MHz, CDCl₃+CCl₄): δ 22.9, 23.5, 28.4, 44.4, 71.5, 71.9, 80.0, 112.9, 127.5, 127.8, 128.3, 136.8, 138.5, 153.; ; ESIMS (*m/z*): 326.28 (M+Na)⁺; HRMS calculated for [C₁₈H₂₅NO₃ +Na]⁺ 326.1727 found: 326.1734.

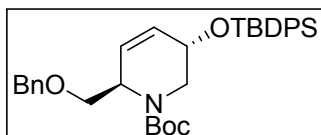
(2R,5S)-tert-Butyl 2-(benzyloxymethyl)-5-hydroxy-5,6-dihydropyridine-1(2H)-carboxylate (21)



To the solution of alkene **16** (0.100 g, 0.33 mmol) in dry 1,4-dioxane (20 mL) was added SeO₂ (0.054 g, 0.49 mmol). The reaction mixture was stirred for 3 h under reflux. After completion of reaction (monitored by TLC), the reaction mixture was allowed to cool at room temperature. The reaction mixture was filtered through a celite bed and the residue was washed with 1,4-dioxane (5 X 10 mL). The filtrate was concentrated *in vacuo* and the obtained residue was dissolved in ethyl acetate (30 mL). The organic layer was washed with saturated NaHCO₃ solution followed by water. The organic extract was dried over anhydrous Na₂SO₄, filtered and concentrated under reduced pressure to afford crude product. The crude residue was purified by flash silica gel column chromatography using 25% ethyl acetate in petroleum ether as an eluent to afford pure product **21** as colorless oil (0.031 g, 30%).

Yield: 30% [α]²⁵ : +284.94 (c 0.97, CHCl₃); For compound 20 lit.¹⁰[α]²⁰ +270 (c 1.0, CHCl₃); IR (CHCl₃, cm⁻¹): 3421, 1690, 1171; found: 342.1690; ¹H NMR (400 MHz, CDCl₃): δ 1.46 (s, 9H), 3.06-3.29 (m, 1H), 3.53 (bs, 2H), 3.98-4.31 (m, 2H), 4.49 (d, J = 12 Hz, 1H), 4.54 (d, J = 12 Hz, 1H), 4.60-4.70 (m, 1H), 5.92-5.96 (m, 1H), 6.06-7.10 (m, 1H), 7.26-7.34 (m, 5H); ¹³C NMR (100 MHz, CDCl₃+CCl₄): δ 28.46, 62.9, 70.3, 73.2, 80.1, 127.5, 127.7, 128.4, 138.1; ESIMS (*m/z*) : 342.1 (M+Na)⁺; HRMS calculated for [C₁₈H₂₅NO₄+Na]⁺ 342.1676.

(2R,5S)-tert-Butyl 2-((benzyloxy)methyl)-5-((tert-butyldiphenylsilyl)oxy)-5,6-dihydropyridine-1(2H)-carboxylate (22)



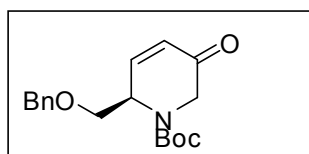
To a solution of alcohol **21** (0.020 g, 0.062 mmol) in anhydrous DCM (5 mL) was added imidazole (0.0085 g, 0.124 mmol) followed by addition of TBDPSCl (0.024 mL, 0.093 mmol) and DMAP (cat.) at 0 °C under nitrogen atmosphere. The reaction was allowed to stir at room temperature for 15 h. After completion of reaction (monitored by TLC), the reaction mixture was diluted with water (30 mL) and extracted with DCM (3 X 30 mL). The combined organic layer was dried over anhydrous sodium sulphate, filtered and concentrated under reduced pressure. The residue was purified by flash silica gel column chromatography using ethyl acetate: pet. ether (5: 95) as an eluent to afford TBS ether as colorless syrup **22** (0.020 g, 60%).

Yield : 60%; $[\alpha]_{\text{D}}^{25}$: +170 (c 0.7, CHCl₃), lit.¹¹ for *ent*-**21** $[\alpha]_{\text{D}}^{21}$ -178 (c 1, CHCl₃);

IR (CHCl₃, cm⁻¹) : 1695, 1417, 1111; ¹H NMR (500 MHz, CDCl₃): δ 1.05 (s, 9H), 1.49 (s, 9H), 2.76- 3.15 (m, 1H), 3.51 (s, 2H), 4.06 (s, 1H), 4.12- 4.41 (m, 1H), 4.47 (d, *J* = 12.2 Hz, 1H), 4.52 (d, *J* = 12.2 Hz, 1H), 4.55- 4.85 (m, 1H), 5.66 (bs, 1H), 5.81 (dd, *J* = 10.1, 3.8 Hz, 1H), 7.23- 7.31 (m, 5H), 7.32- 7.44 (m, 6H), 7.66 (d, *J* = 6.6 Hz, 2H), 7.72 (d, *J* = 6.8 Hz, 2H); ¹³C NMR (125 MHz, CDCl₃+CCl₄): δ 19.3, 26.9, 28.5, 63.9, 70.6, 73.0, 79.5, 127.4, 127.5, 127.7, 128.3, 129.6, 129.7, 133.9, 135.8, 138.2, 154.9.

(R)-tert-Butyl 2-(benzyloxymethyl)-5-oxo-5,6-dihydropyridine-1(2H)-carboxylate (24)

To a solution of alcohol **21** (0.030 g, 0.094 mmol) in ethyl acetate (5 mL) was added IBX (0.052 g, 0.19 mmol). The reaction mixture was refluxed for 2h.

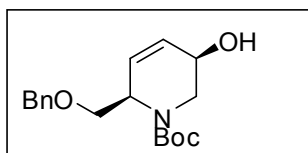


After completion of reaction, the reaction mixture was allowed to cool to room temperature. The reaction mixture was filtered through a Whatman filter paper and the residue was washed with ethyl acetate (3 X 10 mL). The combined organic layer was washed with NaHCO₃, dried over anhydrous sodium sulphate, filtered and concentrated under reduced pressure to obtain crude enone. The residue thus obtained was purified by flash silica gel column chromatography using light petroleum: ethyl acetate (8:2) as an eluent to furnish colorless enone compound **24** (0.026 g, 90% yield).

Yield: 90%; $[\alpha]_{\text{D}}^{25}$: +119.4 (c 0.94, CHCl₃); IR (CHCl₃, cm⁻¹): 3020, 1705, 1693, 1150;

^1H NMR (200 MHz, CDCl_3): δ 1.46 (s, 9H), 3.61-3.91 (m, 3H), 4.45-4.58 (m, 3H), 4.91 (bs, 1H), 6.20 (dd, $J = 10, 1.5$ Hz, 1H), 6.99 (dd, $J = 10, 5$ Hz, 1H), 7.22-7.33 (m, 5H); ^{13}C (100 MHz, $\text{CDCl}_3 + \text{CCl}_4$): δ 28.4, 70.1, 73.3, 80.9, 127.2, 127.5, 127.8, 128.3, 128.4, 128.5, 193.1; ESIMS (m/z): 340.3 ($\text{M} + \text{Na}$) $^+$.

(2*R*,5*R*)-*tert*-Butyl 2-(benzyloxymethyl)-5-hydroxy-5,6-dihydropyridine-1(2*H*)-carboxylate (23)

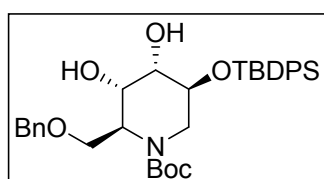


To a stirred solution of enone **24** (0.025 g, 0.078 mmol) and cerium chloride heptahydrate (0.046 g, 0.18 mmol) in methanol (5 mL) was added sodium borohydride (0.006 g, 0.17 mmol) portion wise at 0 °C. The reaction mixture was stirred for one hour at that temperature, after which NH_4Cl was added to destroy excess NaBH_4 . All volatiles were evaporated *in vacuo* and the residue was partitioned between water (20 mL) and CH_2Cl_2 (20 mL). The aqueous phase was extracted with CH_2Cl_2 (2 X 20 mL), the combined organic phase was washed with brine, dried over anhydrous Na_2SO_4 , filtered and concentrated *in vacuo* to give the crude alcohol. The residue was purified by flash silica gel column chromatography using light petroleum: ethyl acetate (7:3) as an eluent to furnish colorless alcohol **23** (0.020 g, 80% yield).

Yield : 80%; $[\alpha]_{\text{D}}^{25}$: +140.76 (c 0.43, CHCl_3); lit¹⁰ for *ent*-22 $[\alpha]_{\text{D}}^{21}$ -146 ($c = 1.0$, CHCl_3);

IR (CHCl_3 , cm^{-1}) : 3421, 1696, 1670, 1455, 1416, 1116; ^1H NMR (500 MHz, $\text{CDCl}_3 + \text{CCl}_4$): δ 1.45 (s, 9H), 1.97 (s, 1H), 2.85 (bs, 1H), 3.47-3.72 (m, 2H), 4.22 (bs, 2H), 4.45 (bs, 1H), 4.53 (d, $J = 12$ Hz, 1H), 4.57 (d, $J = 12$ Hz, 1H), 5.82 (dd, $J = 10, 3$ Hz, 1H), 5.95 (d, $J = 10$ Hz, 1H), 7.26-7.35 (m, 5H); ^{13}C NMR (125 MHz, $\text{CDCl}_3 + \text{CCl}_4$): δ 28.5, 51.6, 63.7, 70.6, 73.3, 80.1, 127.6, 127.7, 128.4, 131.6, 138.0, 154.4; ESIMS (m/z) : 342 ($\text{M} + \text{Na}$) $^+$; HRMS calculated for $[\text{C}_{18}\text{H}_{25}\text{NO}_4 + \text{Na}]^+$ 342.1676; found: 342.1690.

(2*S*,3*S*,4*S*,5*S*)-*tert*-Butyl 2-((benzyloxy)methyl)-5-((*tert*-butyldiphenylsilyl)oxy)-3,4-dihydropiperidine-1-carboxylate (25)



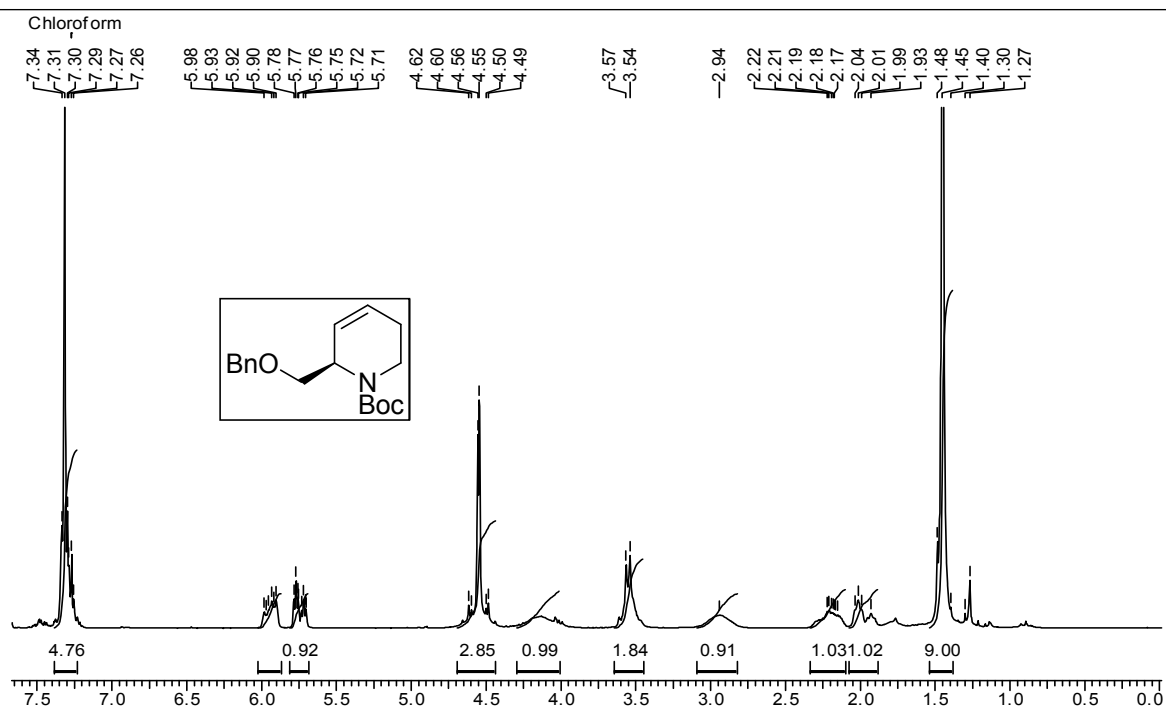
To a solution of alcohol **23** (0.020 g, 0.062 mmol) in anhydrous DCM was added imidazole (0.0085 g, 0.124 mmol) followed by addition of TBDPSCl (0.024 mL, 0.093 mmol) at 0 °C under nitrogen atmosphere. The reaction mixture was allowed to stir at room temperature for 6 h. After completion of reaction (monitored by TLC), reaction mixture was diluted with water. The reaction mixture was extracted with DCM (3 X 30

mL). The combined organic layer was dried over anhydrous sodium sulphate, filtered and concentrated under reduced pressure. The residue was filtered by flash silica gel chromatography using ethyl acetate: pet. ether as an eluent to afford impure TBS ether. To a solution of crude olefin (0.034 mg, 60.95 μmol) in CH_3CN : EtOAc (2 mL: 2 mL) at 0 $^\circ\text{C}$ was added a solution of $\text{RuCl}_3\cdot\text{H}_2\text{O}$ (cat.) and NaIO_4 (19.5 mg, 91.43 μmol) in distilled water (2 mL). The mixture was stirred vigorously for 2 min. and quenched with saturated aqueous solution of $\text{Na}_2\text{S}_2\text{O}_3$ (30 mL). The aqueous phase was separated and extracted with EtOAc (3 X 20 mL). The combined organic extracts were dried over anhydrous sodium sulphate, filtered and concentrated under reduced pressure. The residue thus obtained was purified by flash silica gel column chromatography using light pet. ether: ethyl acetate (6:4) as an eluent to furnish colorless alcohol **25** (0.02 g, 54% over two steps).

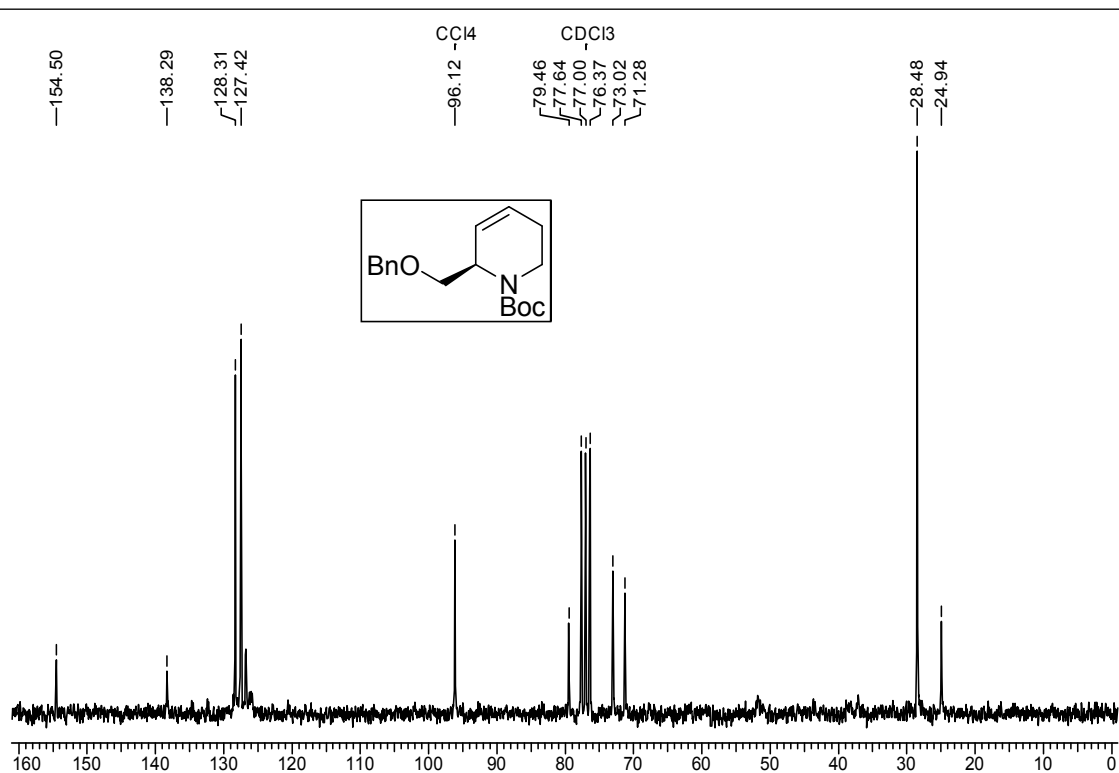
Yield: 54%; IR (CHCl_3 , cm^{-1}): 3434, 2929, 2856, 1693, 1668, 1427, 1111; $[\alpha]_{\text{D}}^{25}$: +50 (c 1, CHCl_3), lit.¹¹ +52 (c 1, CHCl_3); ^1H NMR (500 MHz, $\text{CDCl}_3+\text{CCl}_4$): δ 1.08 (s, 9H), 1.34 (s, 9H), 2.10 (bs, 1H), 2.25 (s, 1H), 2.49 (s, 1H), 2.83 (bs, 1H), 3.55 (s, 2H), 3.76 (bs, 1H), 3.87- 3.95 (m, 1H), 4.01 (s, 1H), 4.36 (broad s, 1H), 4.48 (d, $J = 12.0$ Hz, 1H), 4.53 (d, $J = 12.0$ Hz, 1H), 7.25- 7.45 (m, 11H), 7.63 (d, $J = 7.6$ Hz, 2H), 7.72 (d, $J = 7.6$ Hz, 2H); ^{13}C NMR (125 MHz, $\text{CDCl}_3+\text{CCl}_4$): δ 19.4, 27.1, 28.4, 29.8, 68.9, 69.7, 69.9, 73.2, 74.1, 79.9, 127.5, 127.7, 127.9, 128.5, 130.1, 133.7, 135.8, 137.9, 155.1.

NMR spectra: copies of ^1H and ^{13}C

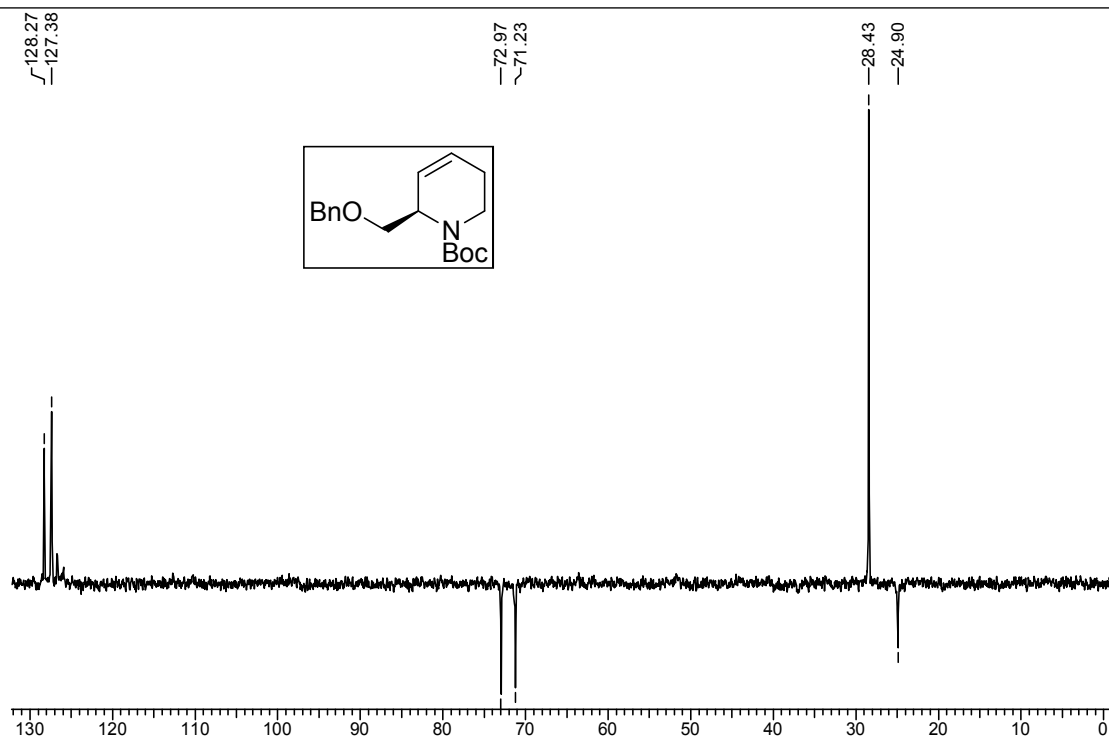
^1H NMR Spectrum of compound 16 ($\text{CDCl}_3+\text{CCl}_4$, 200 MHz)



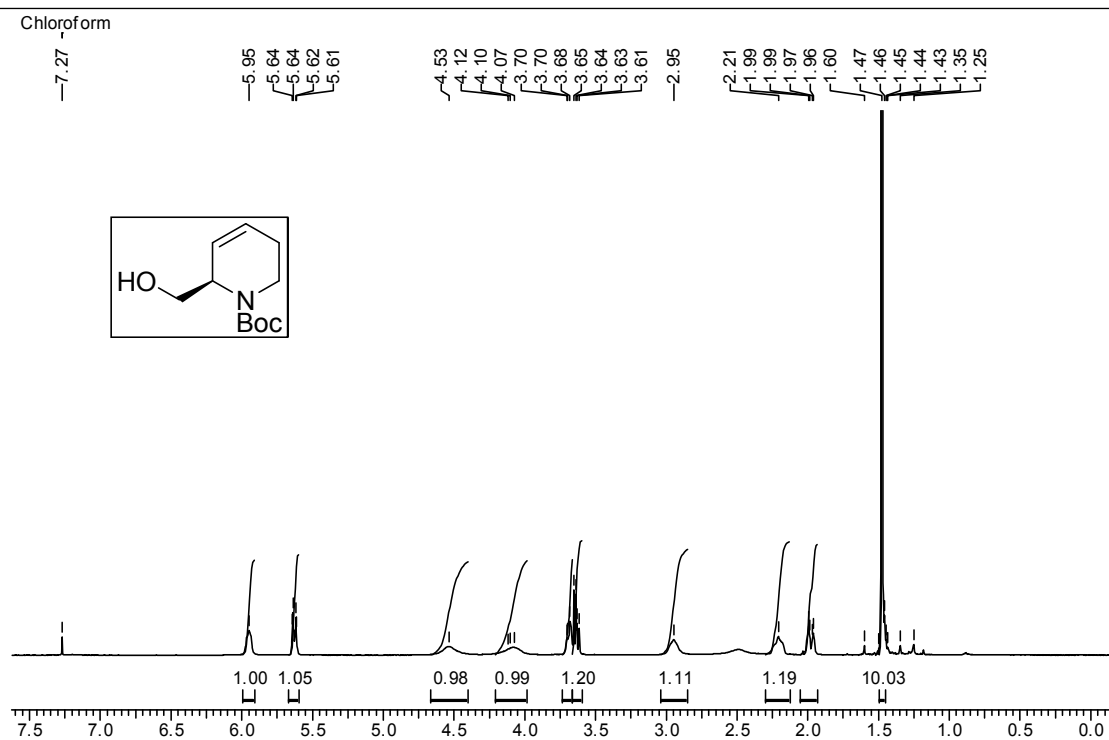
^{13}C NMR Spectrum of compound 16 ($\text{CDCl}_3+\text{CCl}_4$, 50 MHz)



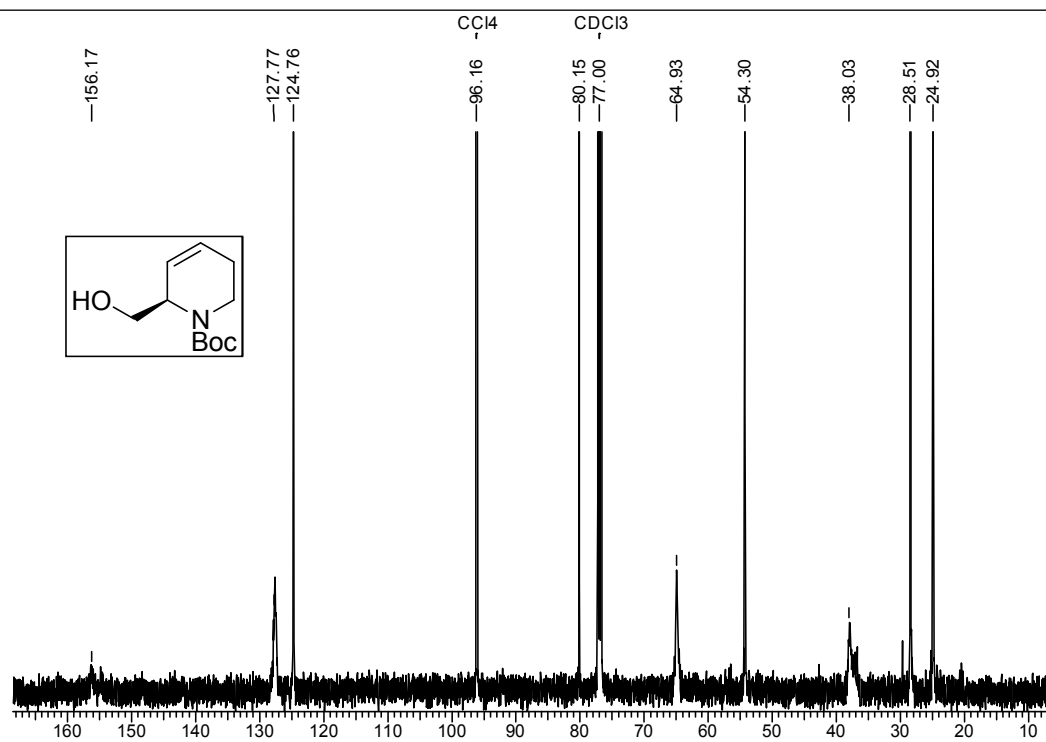
DEPT Spectrum of compound 16 (CDCl₃+CCl₄, 50 MHz)



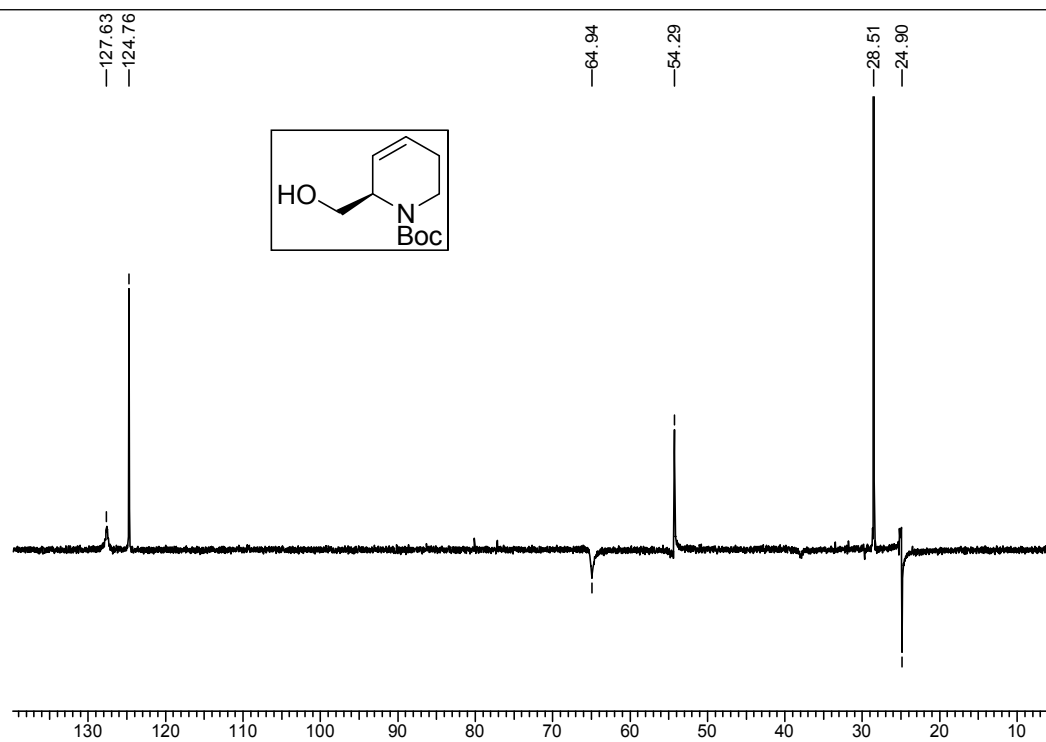
¹H NMR Spectrum of compound 8 (CDCl₃+CCl₄, 500 MHz)



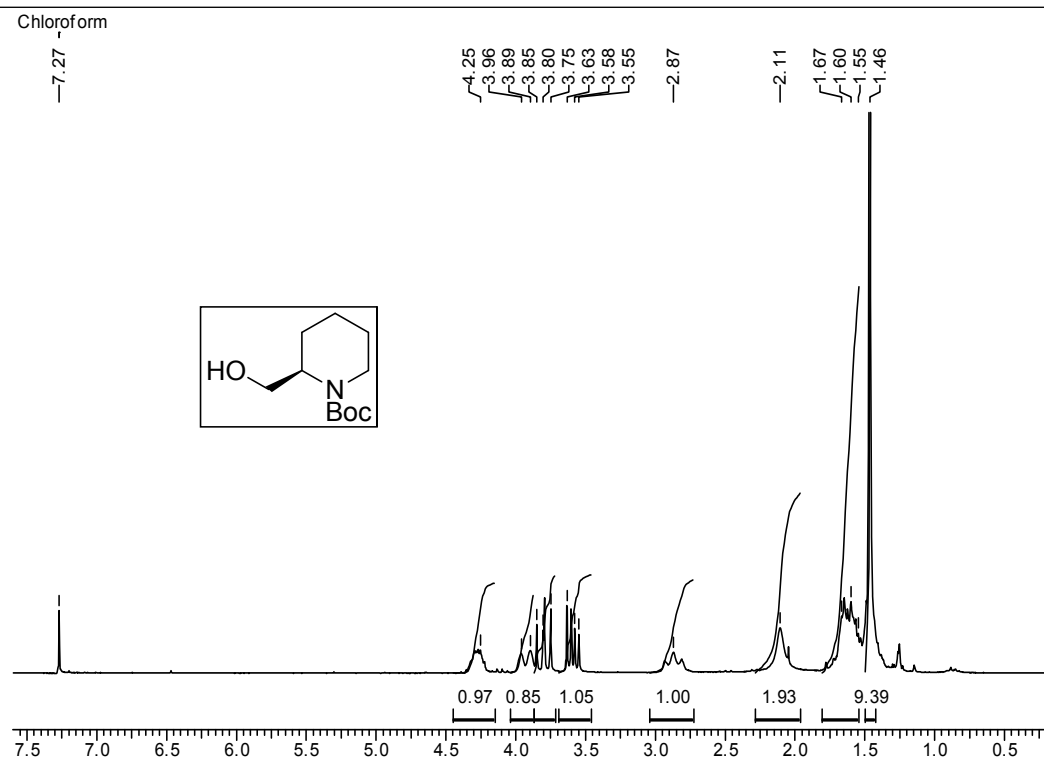
¹³C NMR Spectrum of compound 8 (CDCl₃+CCl₄, 125 MHz)



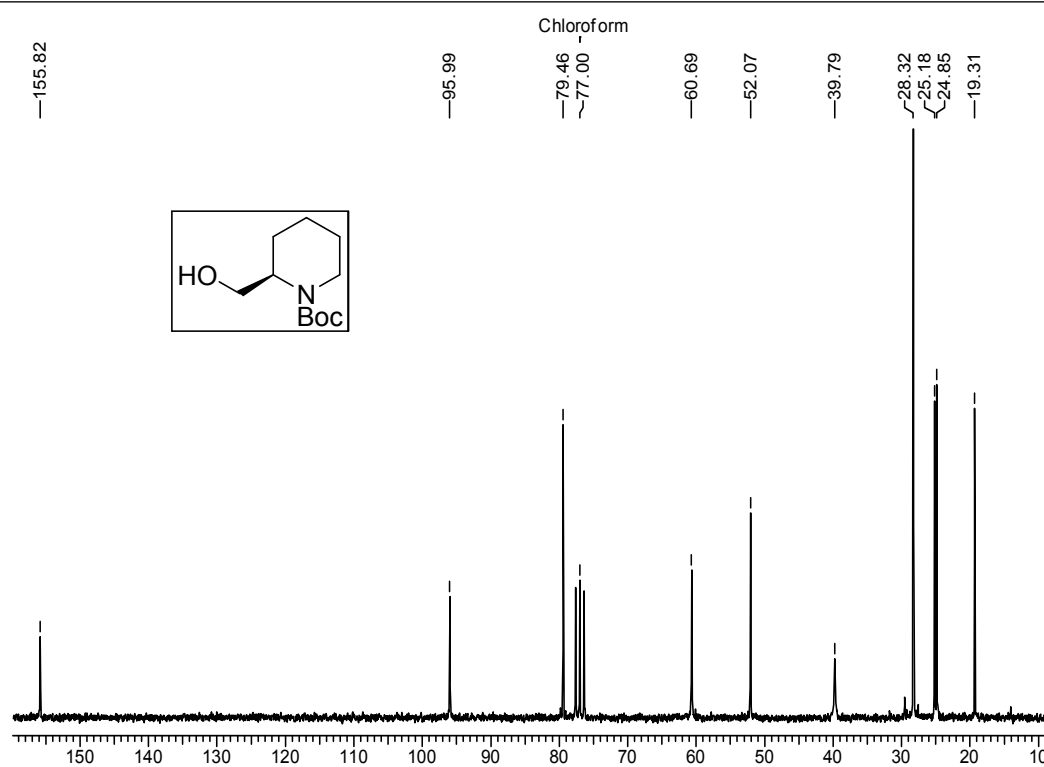
DEPT Spectrum of compound 8 (CDCl₃+CCl₄, 125 MHz)



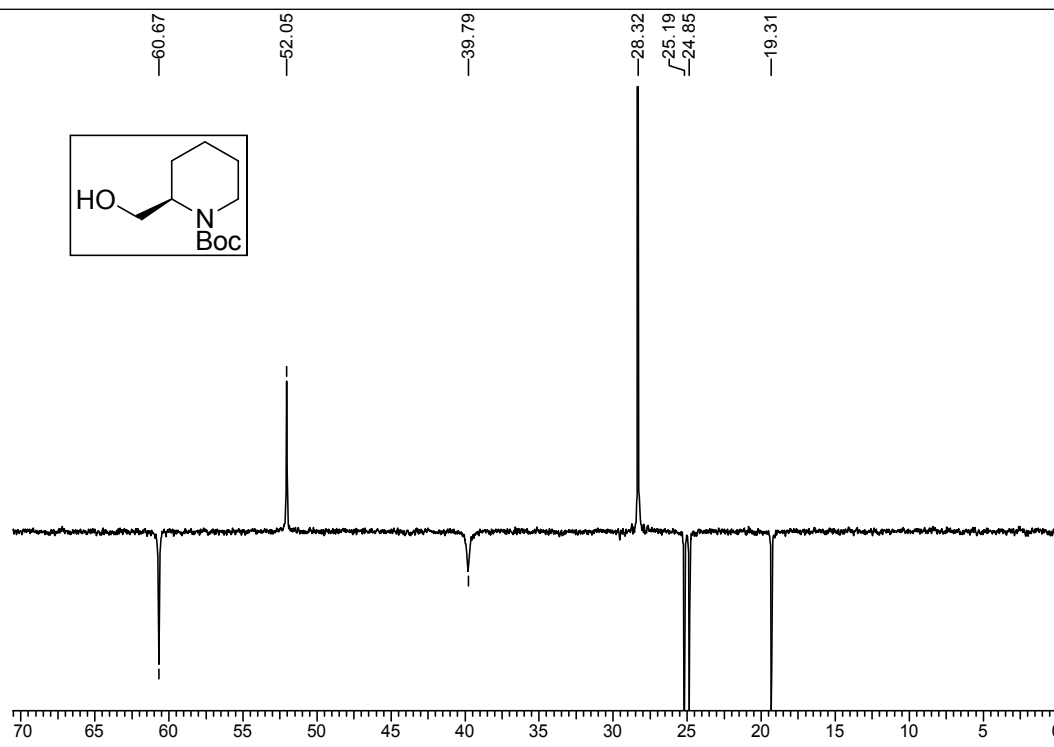
¹H NMR Spectrum of compound 17 (CDCl₃+CCl₄, 200 MHz)



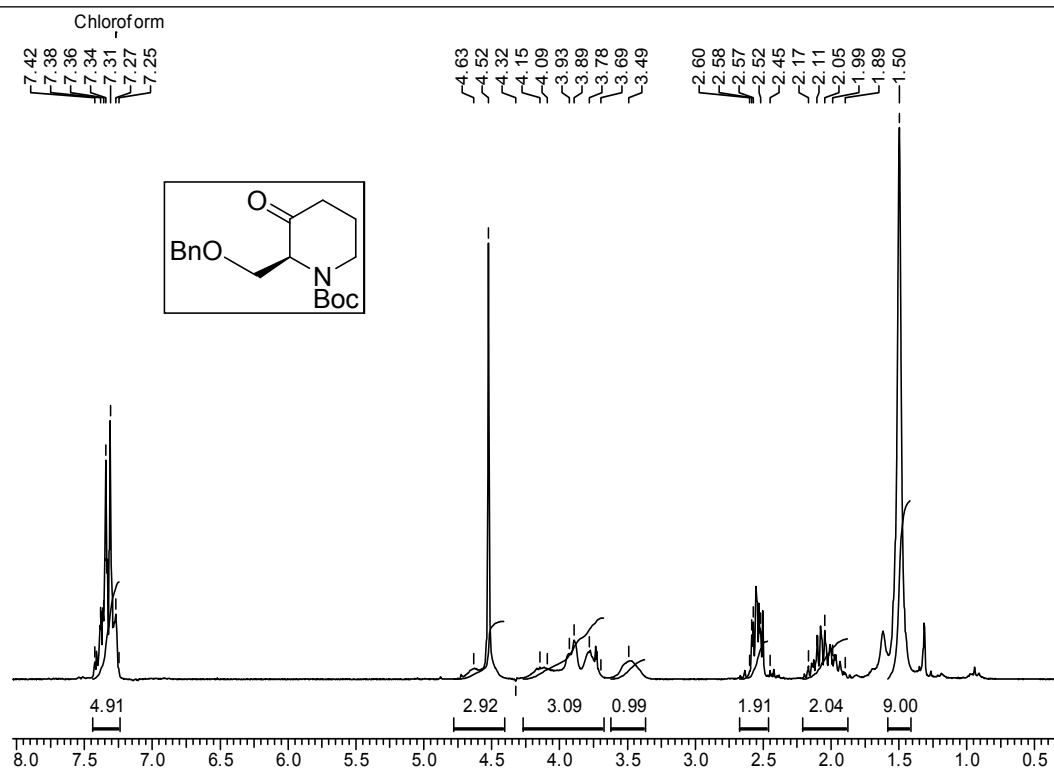
¹³C NMR Spectrum of compound 17 (CDCl₃+CCl₄, 50 MHz)



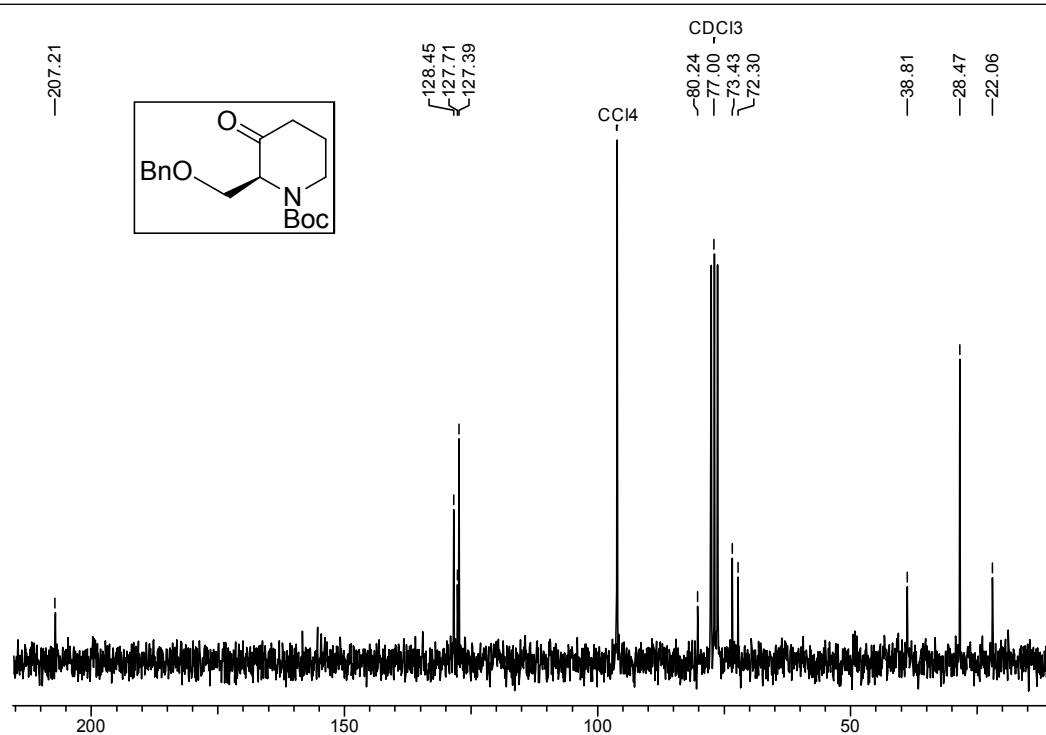
DEPT Spectrum of compound 17 (CDCl₃+CCl₄, 50 MHz)



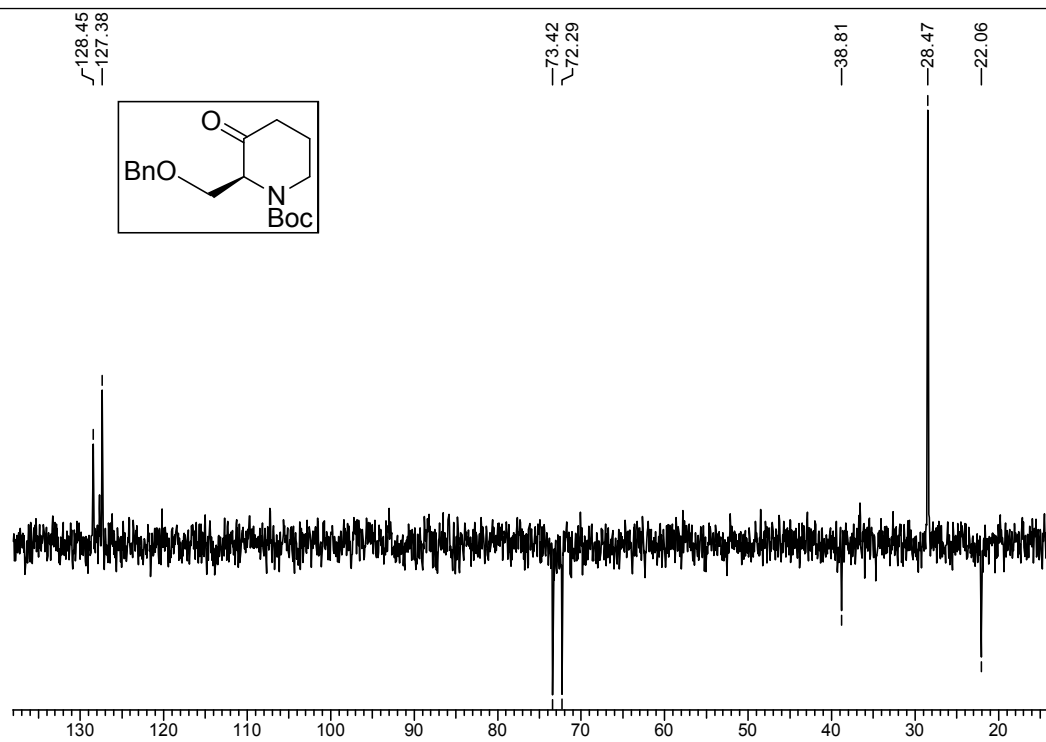
¹H NMR Spectrum of compound 18 (CDCl₃+CCl₄, 200 MHz)



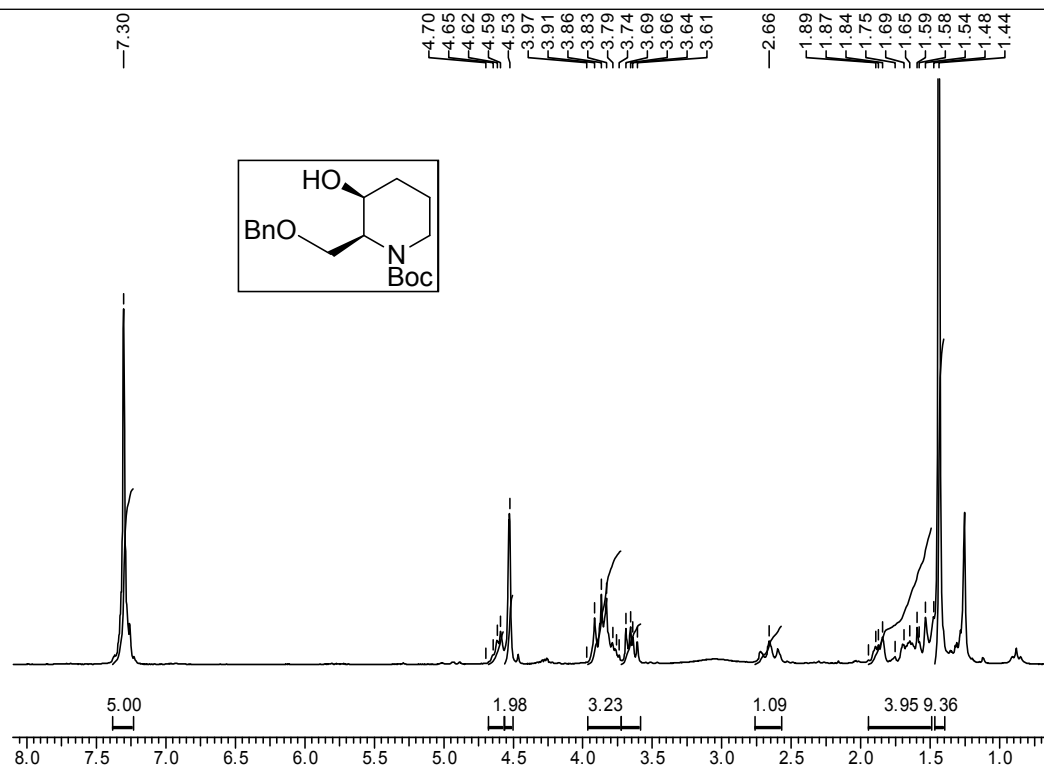
^{13}C NMR Spectrum of compound 18 ($\text{CDCl}_3+\text{CCl}_4$, 50 MHz)



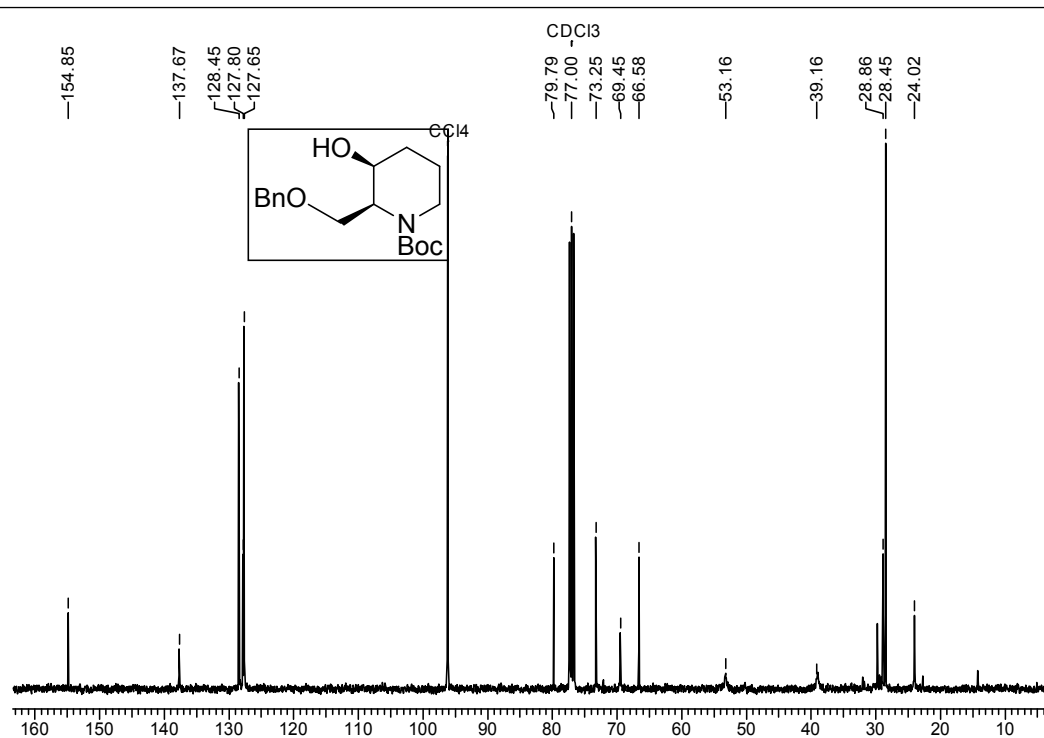
DEPT Spectrum of compound 18 ($\text{CDCl}_3+\text{CCl}_4$, 50 MHz)



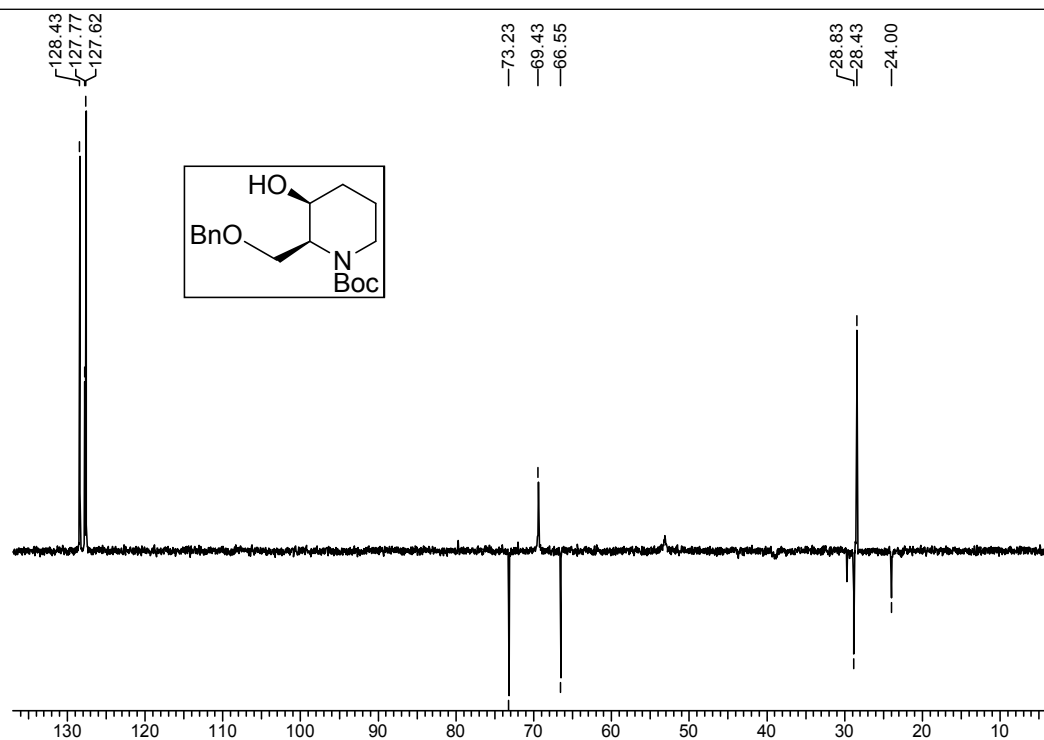
¹H NMR Spectrum of compound 19 (CDCl₃+CCl₄, 200 MHz)



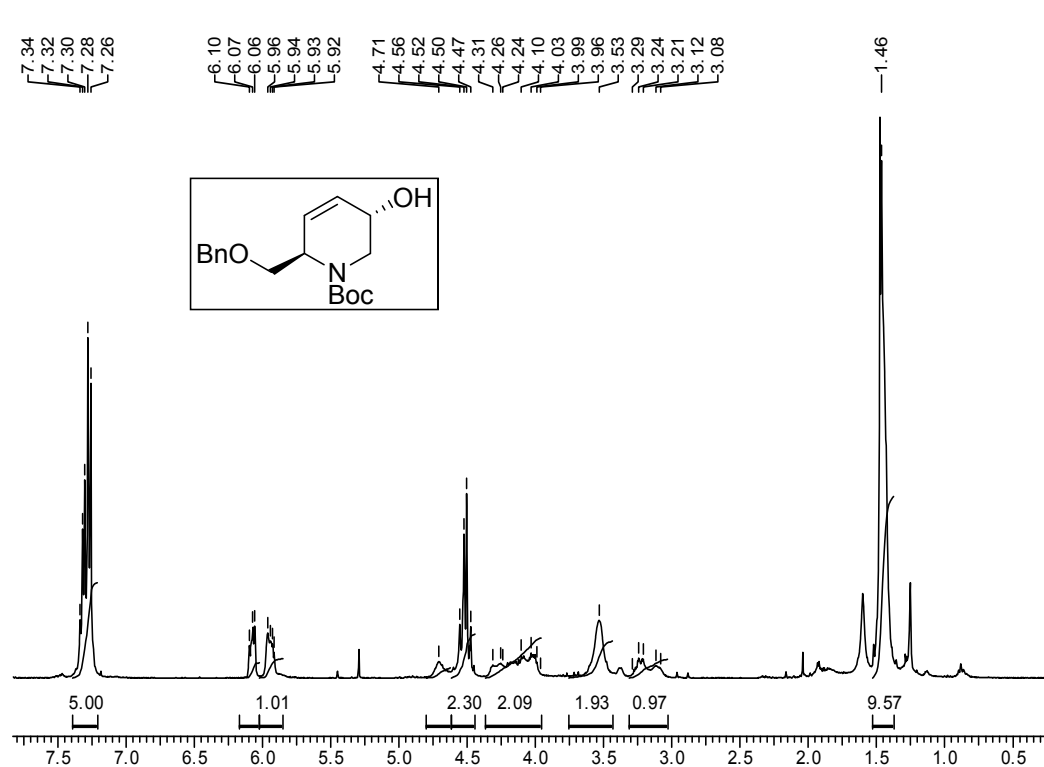
¹³C NMR Spectrum of compound 19 (CDCl₃+CCl₄, 100 MHz)



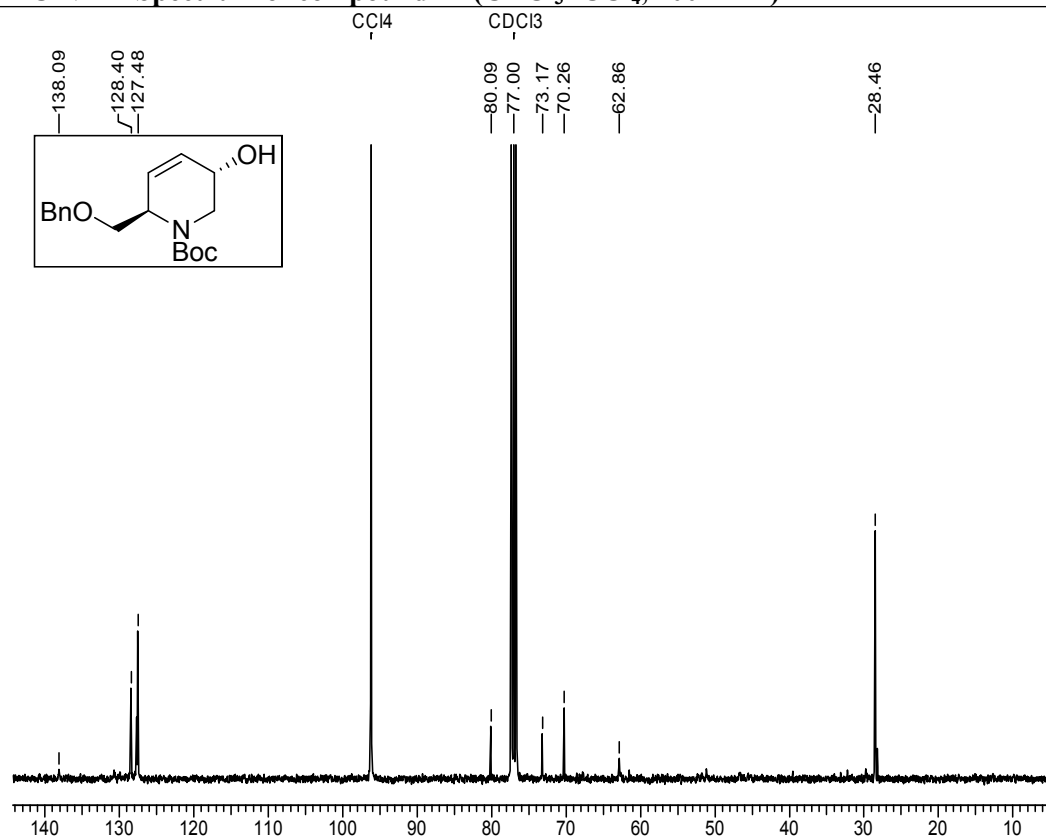
DEPT Spectrum of compound 19 (CDCl₃+CCl₄, 100 MHz)



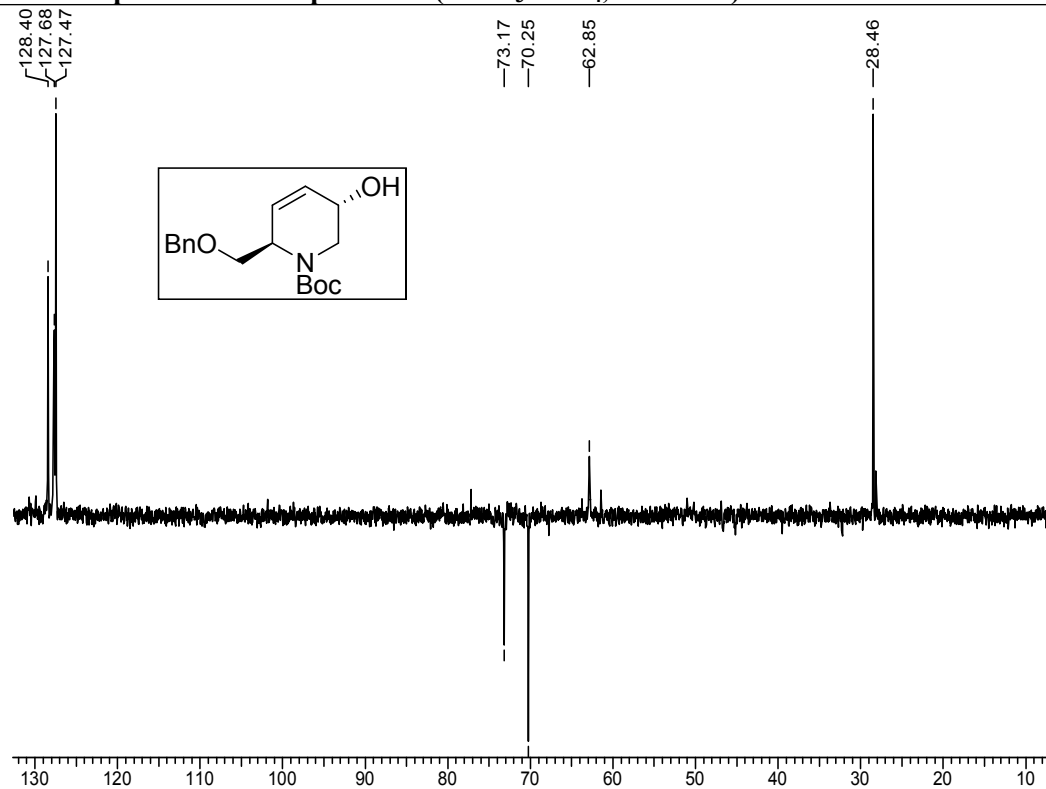
¹H NMR Spectrum of compound 21 (CDCl₃+CCl₄, 400 MHz)



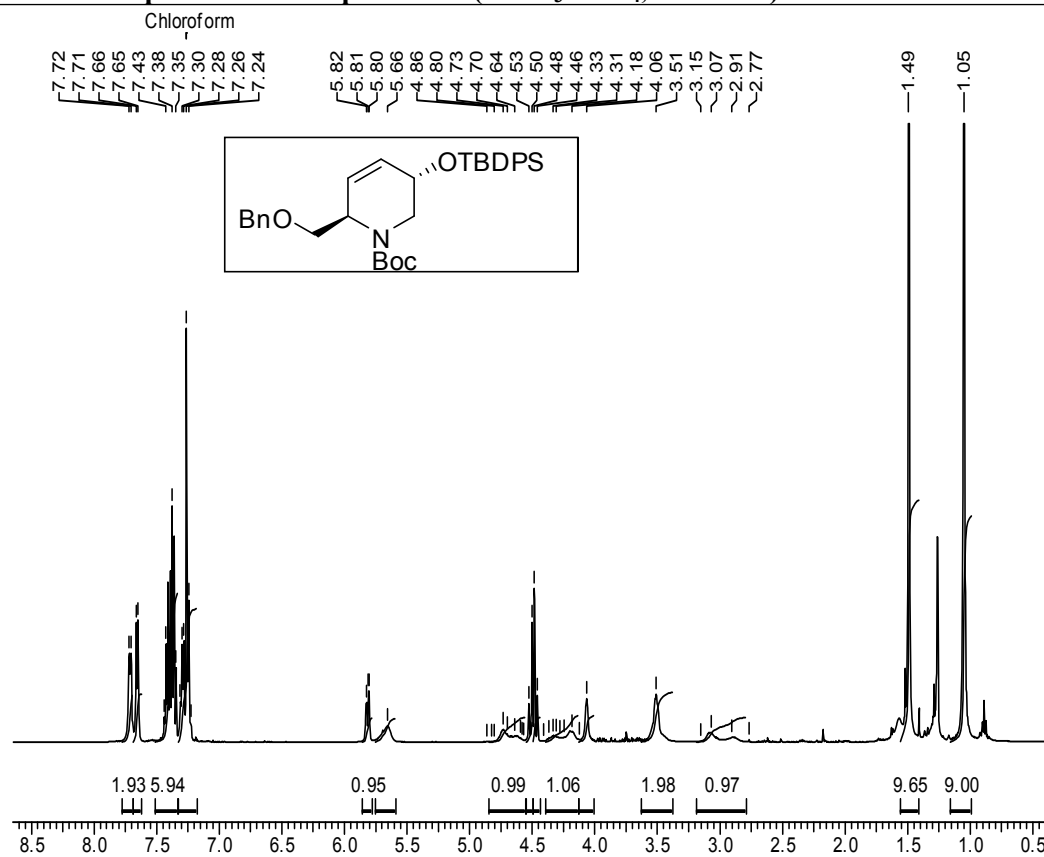
¹³C NMR Spectrum of compound 21 (CDCl₃+CCl₄, 100 MHz)



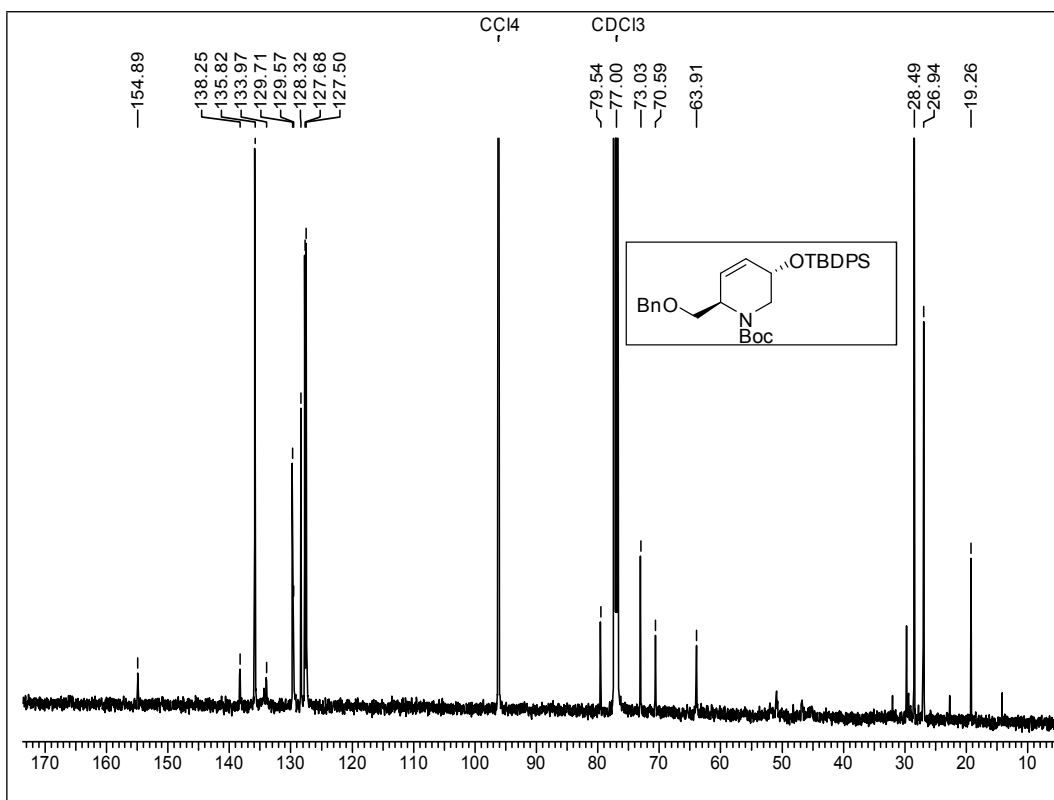
DEPT Spectrum of compound 21 (CDCl₃+CCl₄, 100 MHz)



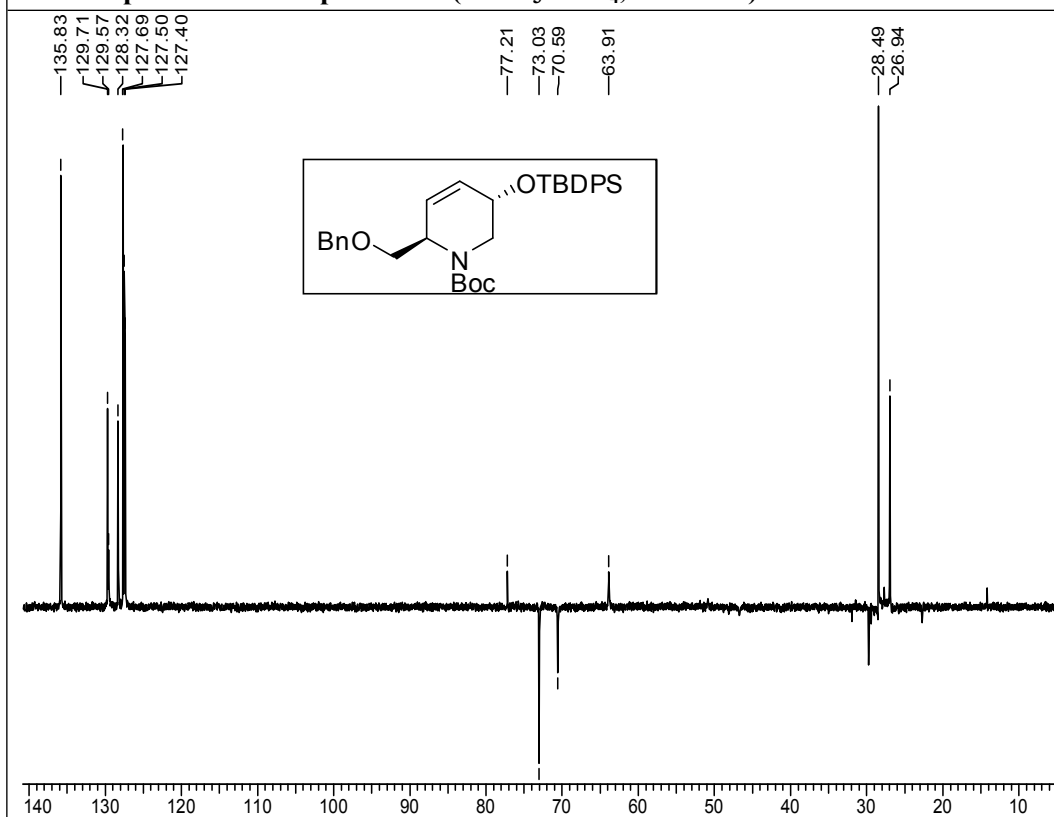
¹H NMR Spectrum of compound 22 (CDCl₃+CCl₄, 500 MHz)



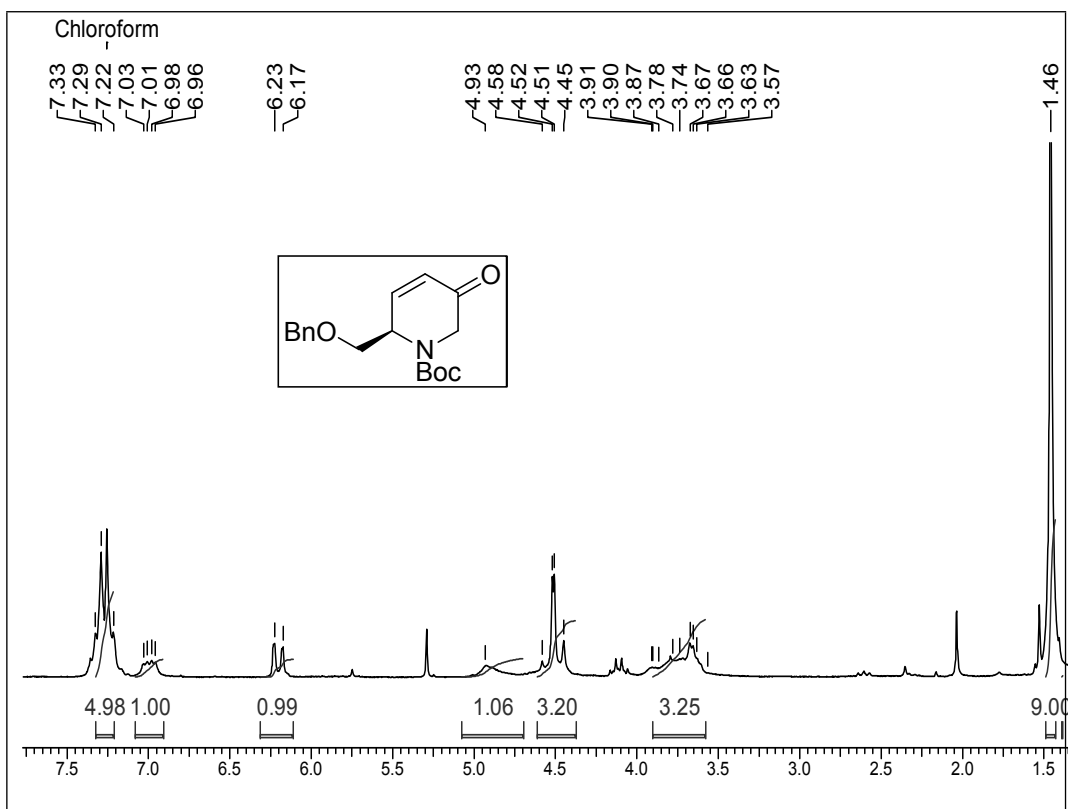
¹³C NMR Spectrum of compound 22 (CDCl₃+CCl₄, 125 MHz)



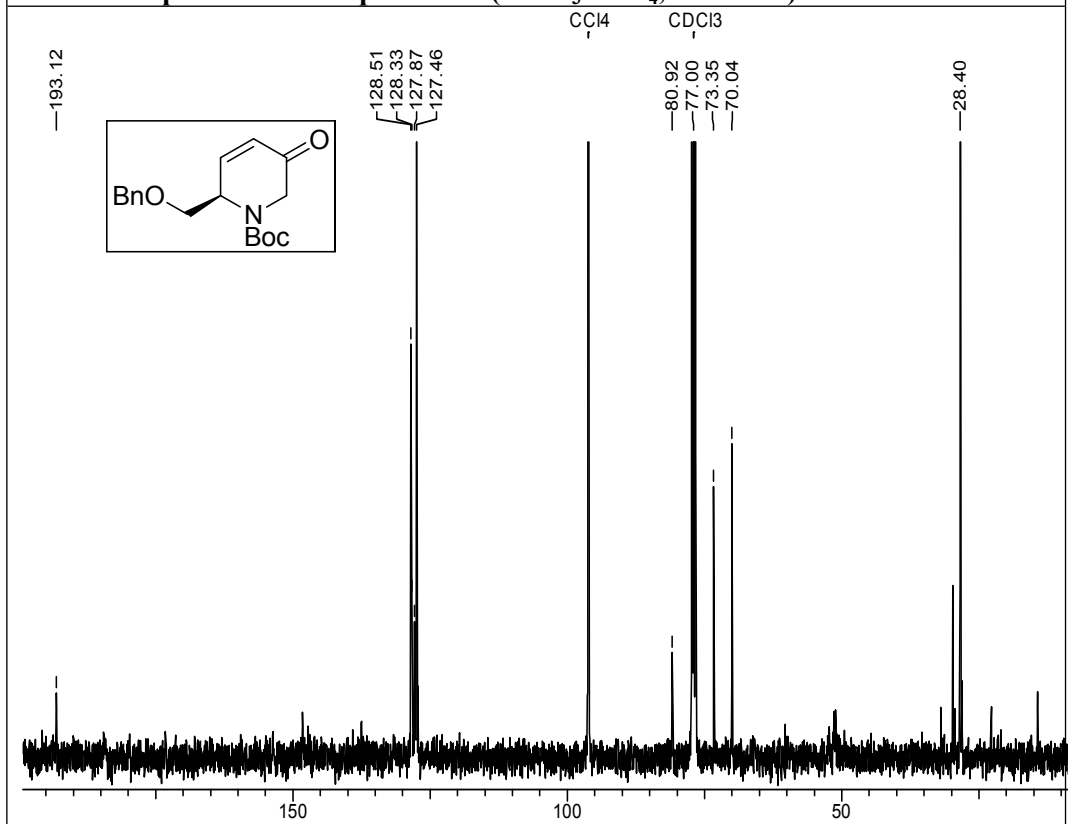
DEPT Spectrum of compound 22 (CDCl₃+CCl₄, 125 MHz)



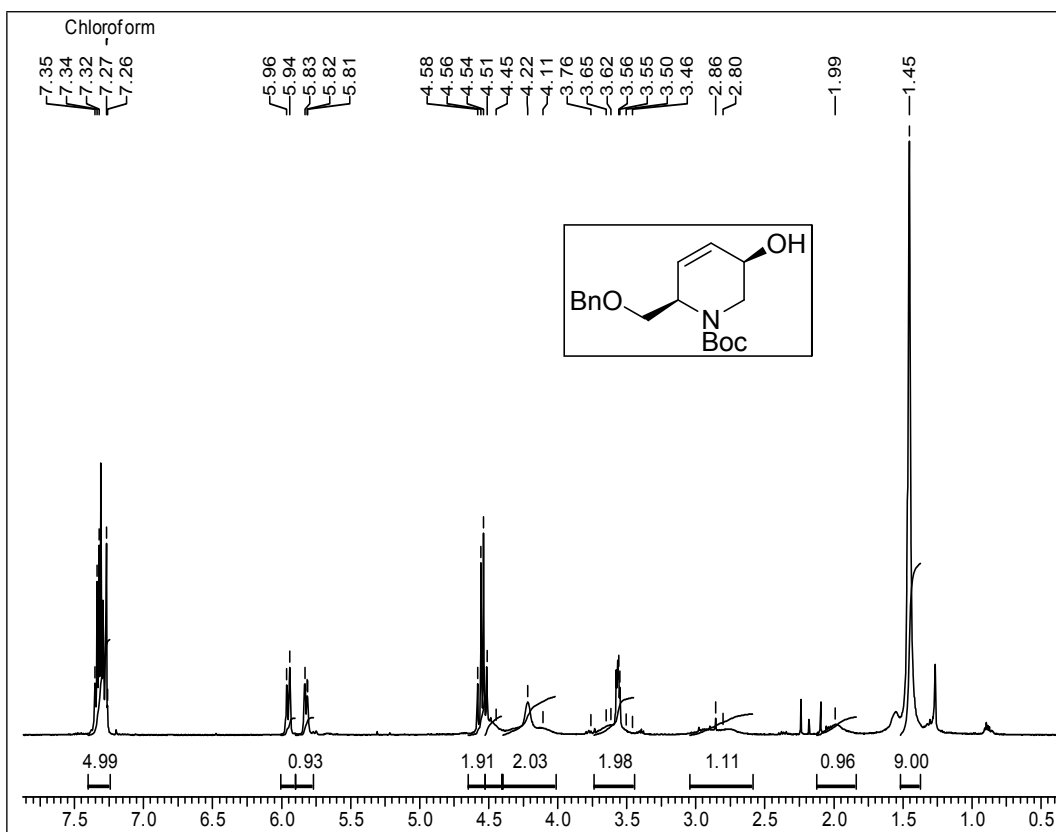
¹H NMR Spectrum of compound 24 (CDCl₃+CCl₄, 200 MHz)



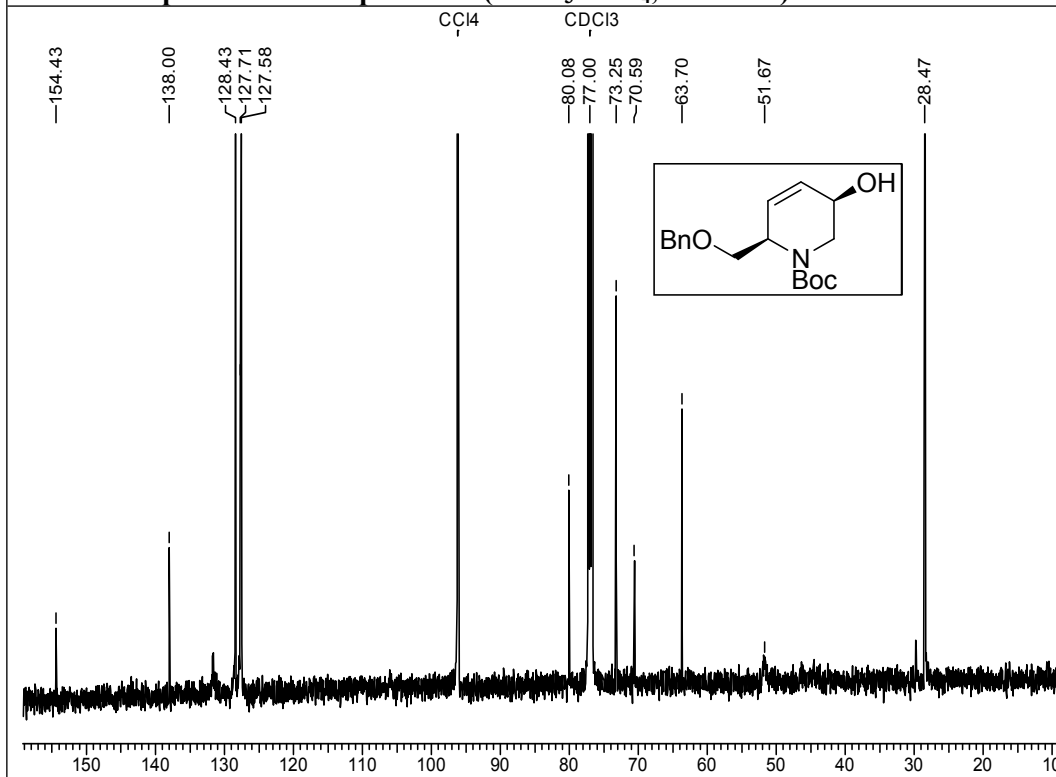
^{13}C NMR Spectrum of compound 24 ($\text{CDCl}_3 + \text{CCl}_4$, 100 MHz)



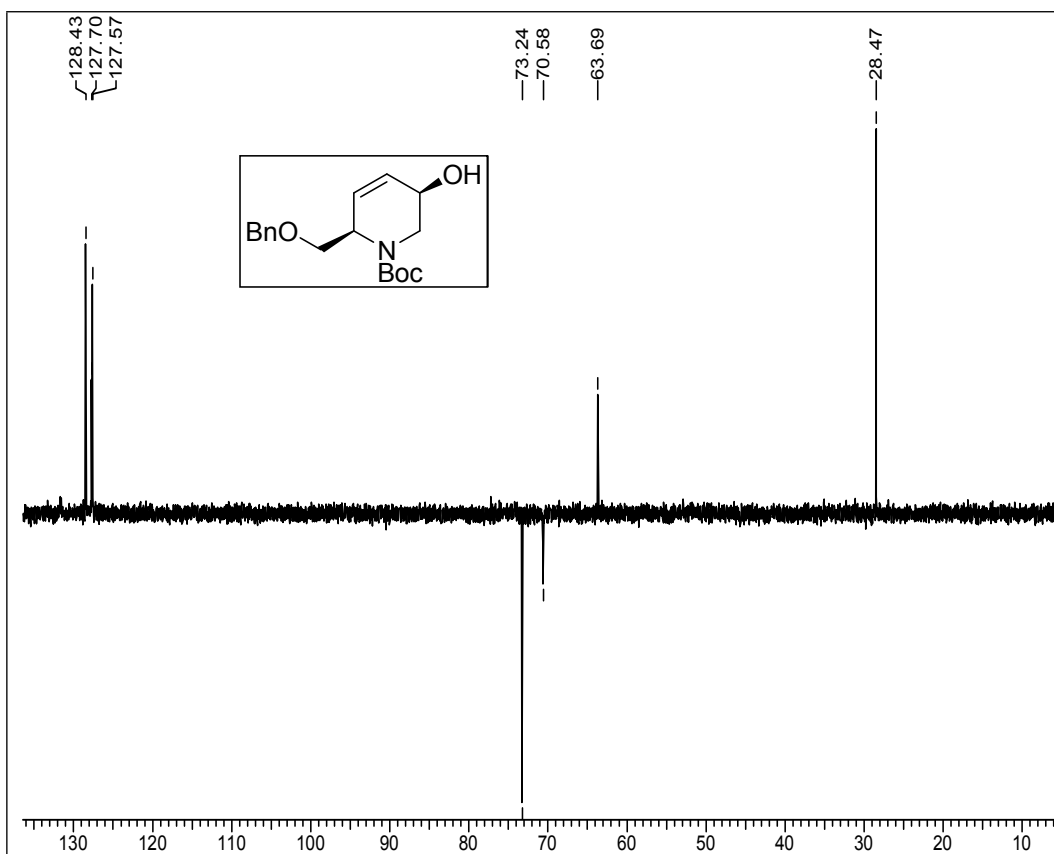
^1H NMR Spectrum of compound 23 ($\text{CDCl}_3 + \text{CCl}_4$, 500 MHz)



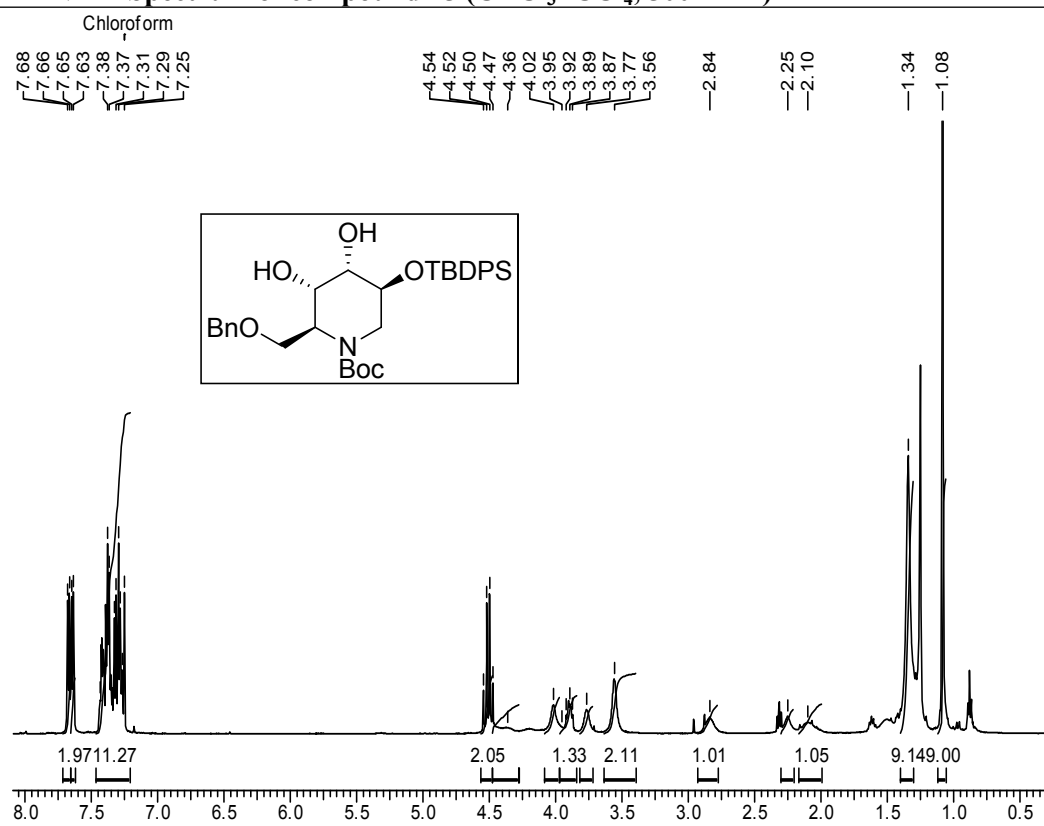
^{13}C NMR Spectrum of compound 23 ($\text{CDCl}_3 + \text{CCl}_4$, 125 MHz)



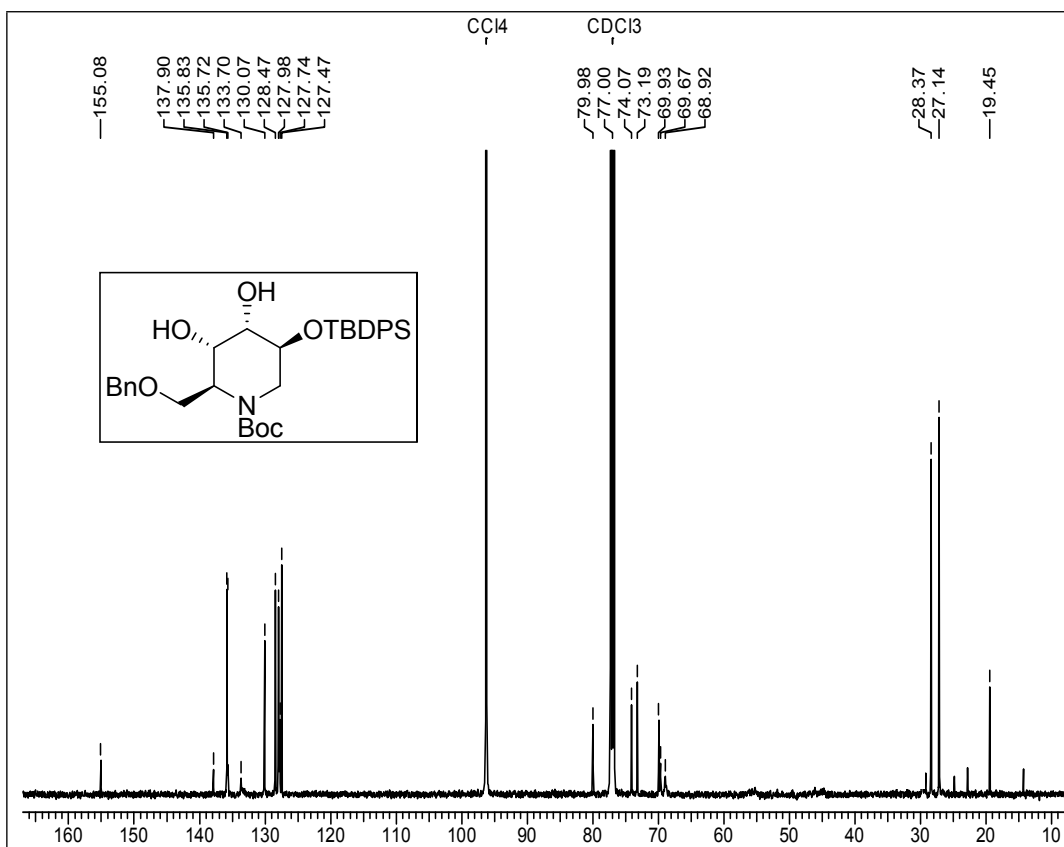
DEPT Spectrum of compound 23 ($\text{CDCl}_3 + \text{CCl}_4$, 125 MHz)



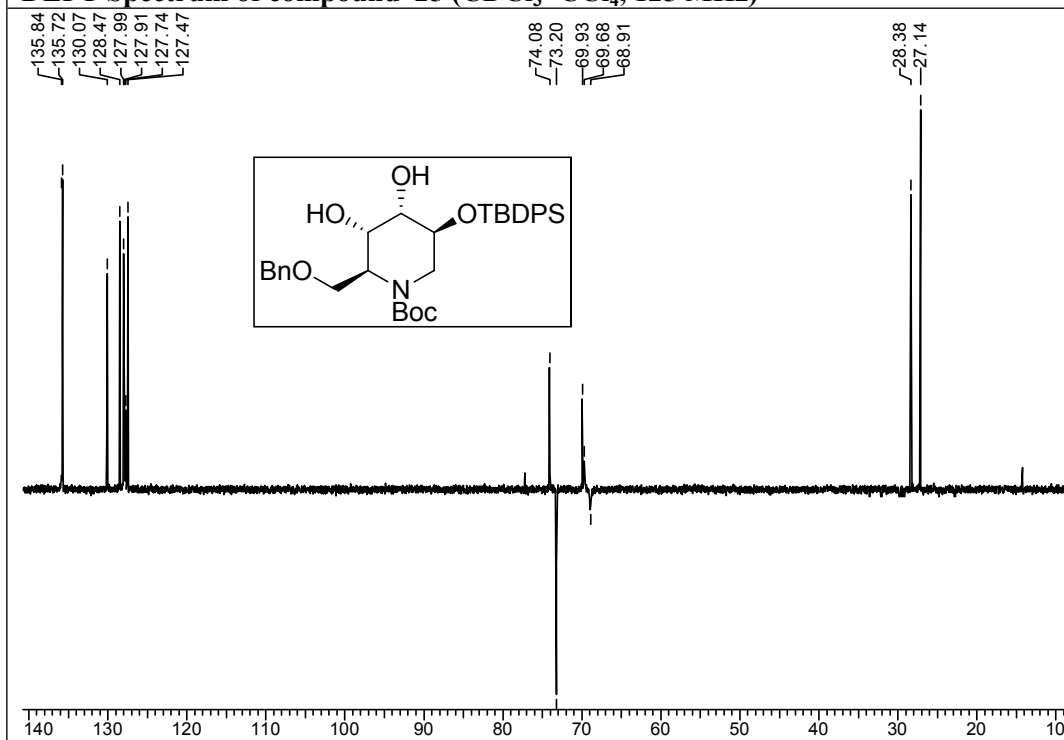
¹H NMR Spectrum of compound 25 (CDCl₃+CCl₄, 500 MHz)



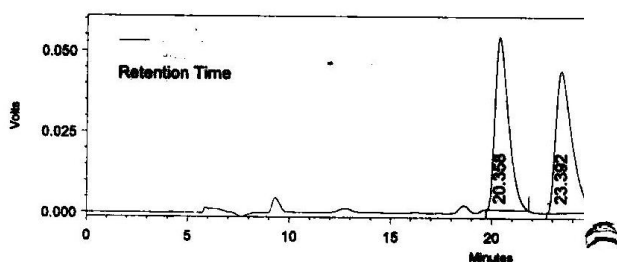
¹³C NMR Spectrum of compound 25 (CDCl₃+CCl₄, 125 MHz)



DEPT Spectrum of compound 25 (CDCl₃+CCl₄, 125 MHz)



HPLC Chromatograms

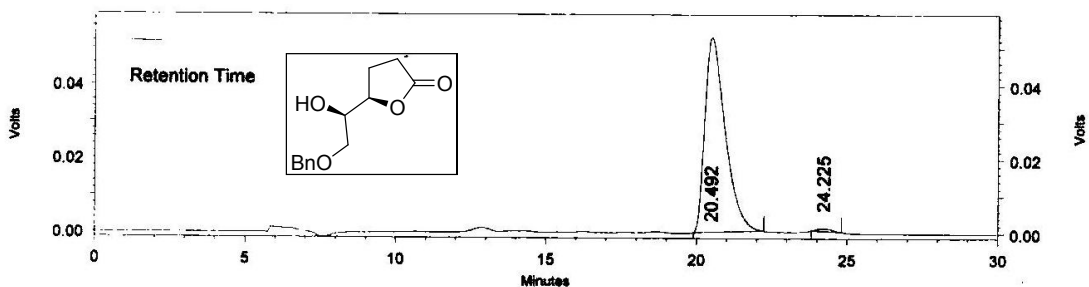


Project Leader : Dr.S P Chavan
 Column : Chiralcel OD-H (250x4.6mm)
 Mobile Phase :IPA:Petether: (20:80)
 Wavelength : 254nm
 Flow Rate : 0.5ml/min (26kgf)
 Sample Con : 1mg/1ml Inj vol-20ul

Kunte

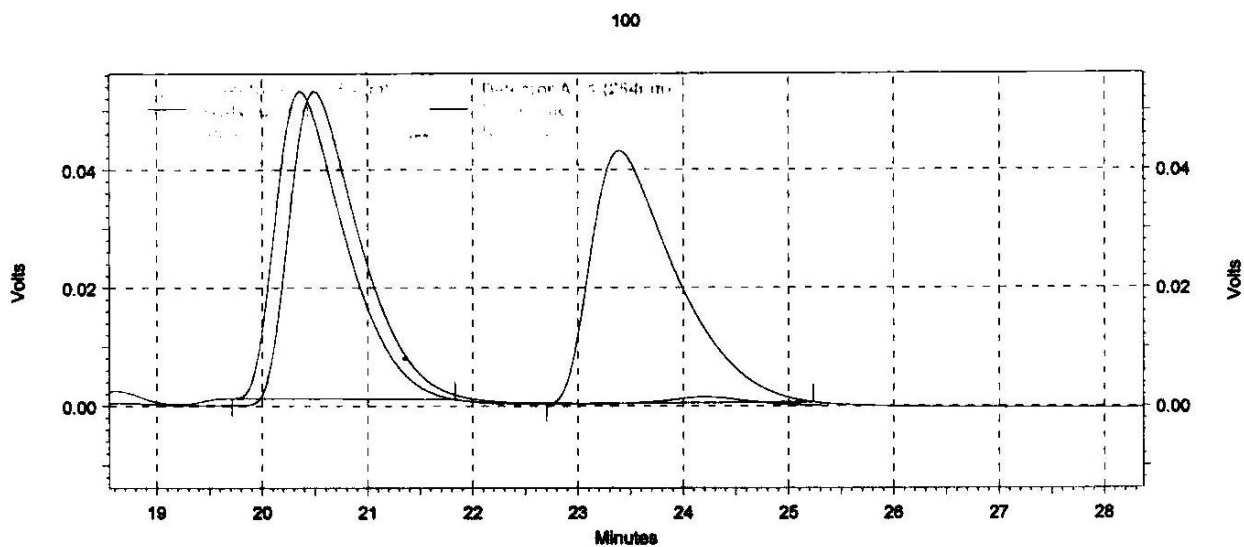
Detector A - 1 (254nm)

PK #	Retention Time	Area	Area %
1	20.358	2452872	49.993
2	23.392	2453535	50.007
		4906407	100.000



Detector A - 1 (254nm)

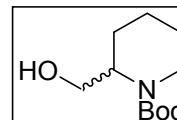
PK #	Retention Time	Area	Area %
1	20.492	2487393	98.885
2	24.225	28051	1.115
Totals		2515444	100.000

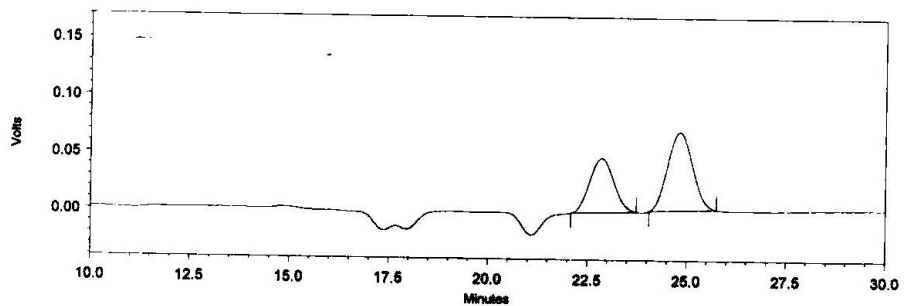


— C:\CLASS-VP\Data\Dr. CHAVAN S. PWB 1013, Detector A - 1 (254nm)

— C:\CLASS-VP\Data\Dr. CHAVAN S. PWB 1012, Detector A - 1 (254nm)

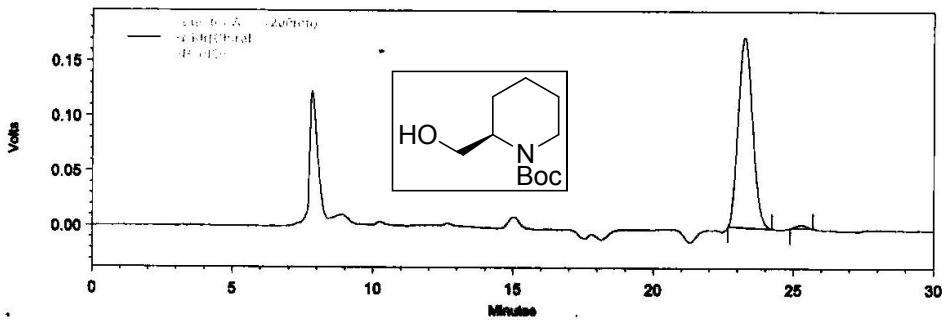
Project Leader :- Dr. S P Chavan
 Column :-Kromasil 5-AmyCoat (250 x 4.6mm)
 M.Phase :-IPA:PE (4:96)
 Wavelength :- 200 nm
 Flow :- 0.5ml/min (288psi)
 conc. :- 4mg/ 1 ml
 Injection vol :- 5 ul





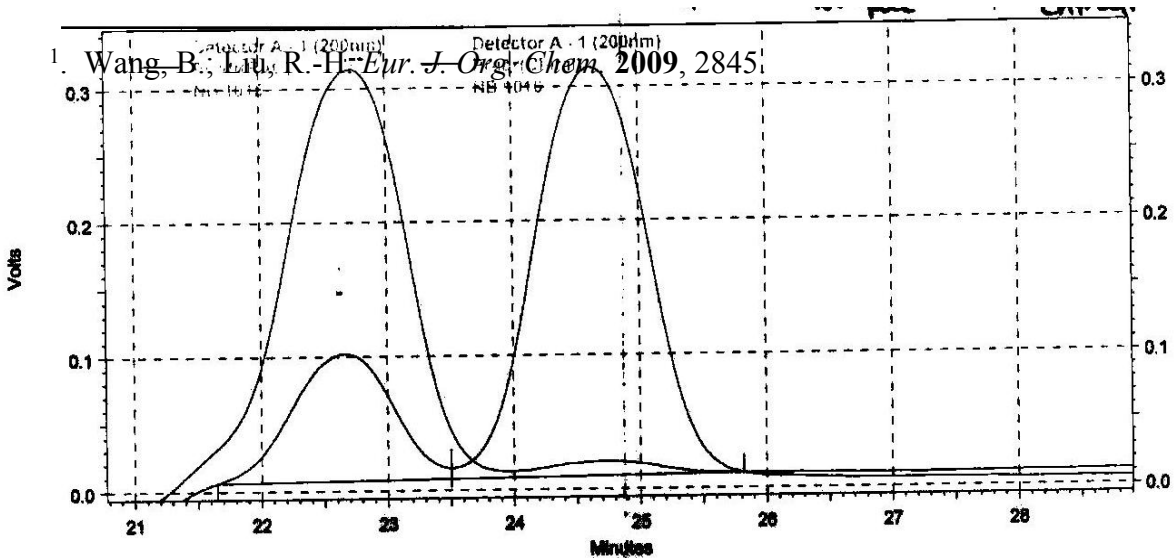
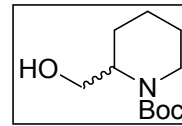
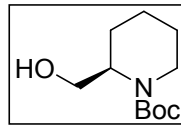
Detector A - 1 (200nm)

Pk #	Retention Time	Area	Area %
1	22.858	2005440	40.105
2	24.825	2994974	59.895
Totals		5000414	100.000



Detector A - 1 (200nm)

Pk #	Retention Time	Area	Area %
1	23.250	6194327	98.565
2	25.258	90176	1.435



— C:\CLASS-VP\Data\Dr. CHAVAN S. PNB 1015, Detector A - 1 (200nm)

— C:\CLASS-VP\Data\Dr. CHAVAN S. PNB 1016, Detector A - 1 (200nm)