Supporting Information for:

Stereocontrolled synthesis of 3-hydroxy-2-piperidinone carboxamides by catalytic ring-opening aminolysis of bridged δ-lactam-γ-lactones

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Contents:

2. Experimental Section

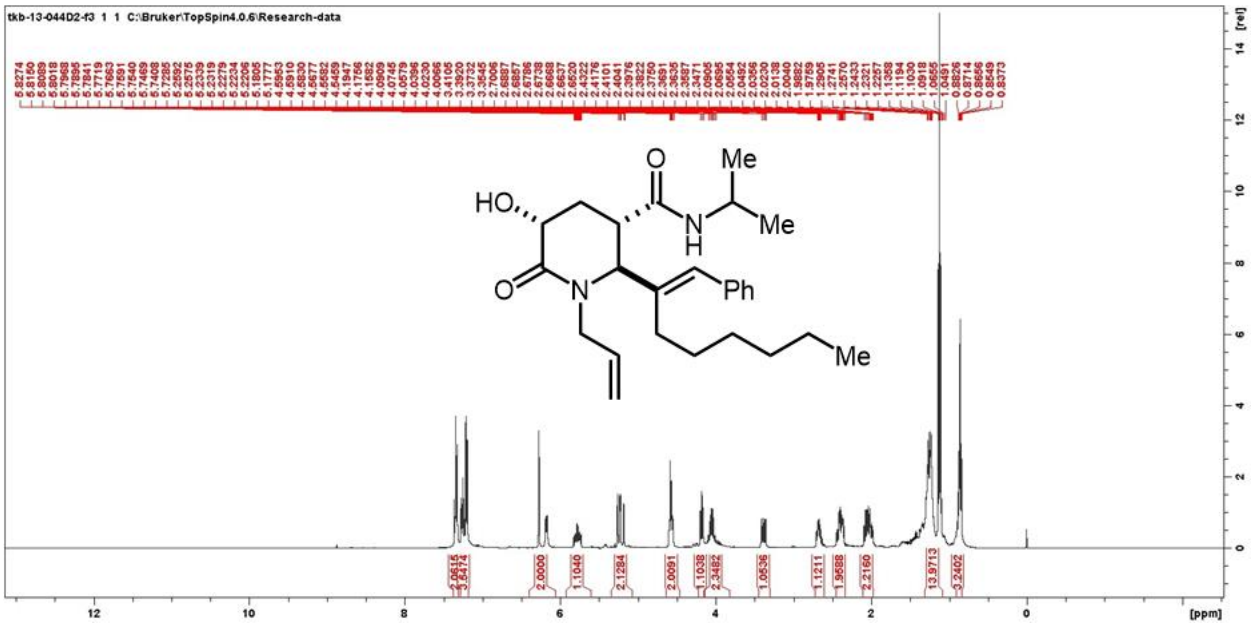
All experiments involving air and moisture-sensitive reagents were carried out under an inert atmosphere of nitrogen and using freshly distilled solvents. Freshly purchased toluene and DMF were stored under 4 A^{o} molecular sieves for several days prior to use. THF and 2-MeTHF were distilled from sodium benzophenone ketyl. Other reagents were purchased at the highest commercial quality and used without further purification, unless otherwise stated. All amines, enals, Grignard reagents, *N*-bromosuccinimide, and potassium carbonate were newly purchased and used without further purification. Column chromatography was performed on silica gel (230-400 mesh). Thin-layer chromatography (TLC) was performed using Silicycle SiliaplateTM glass backed plates (250 μm thickness, 60 Å porosity, F-254 indicator) and visualized using UV (254 nm). Unless otherwise indicated, ^{1}H NMR spectral data were acquired using $CDCl_3$ as solvent, at room temperature. Chemical shifts are quoted in parts per million (ppm) referenced to 0.00 ppm for TMS. The following abbreviations (or combinations thereof) were used to explain multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, br = broad, dq = double quartet, dp = double pentet. Coupling constants, J, were reported in Hertz unit (Hz). ^{13}C NMR spectra were recorded on Bruker DRX-400 was fully decoupled by broad band proton decoupling. Chemical shifts were reported in ppm referenced to the center line of a triplet at 77.16 ppm of $CDCl_3$. HRMS-EI^{+} data were obtained using either electron spray ionization (ESI) or electron impact (EI) techniques. High-resolution ESI was obtained on an LTQ-FT (ion trap; analyzed using Excalibur). High resolution EI was obtained on an Autospec (magnetic sector; analyzed using MassLynx). The bridged lactam-lactones were prepared using our developed procedure.[1]

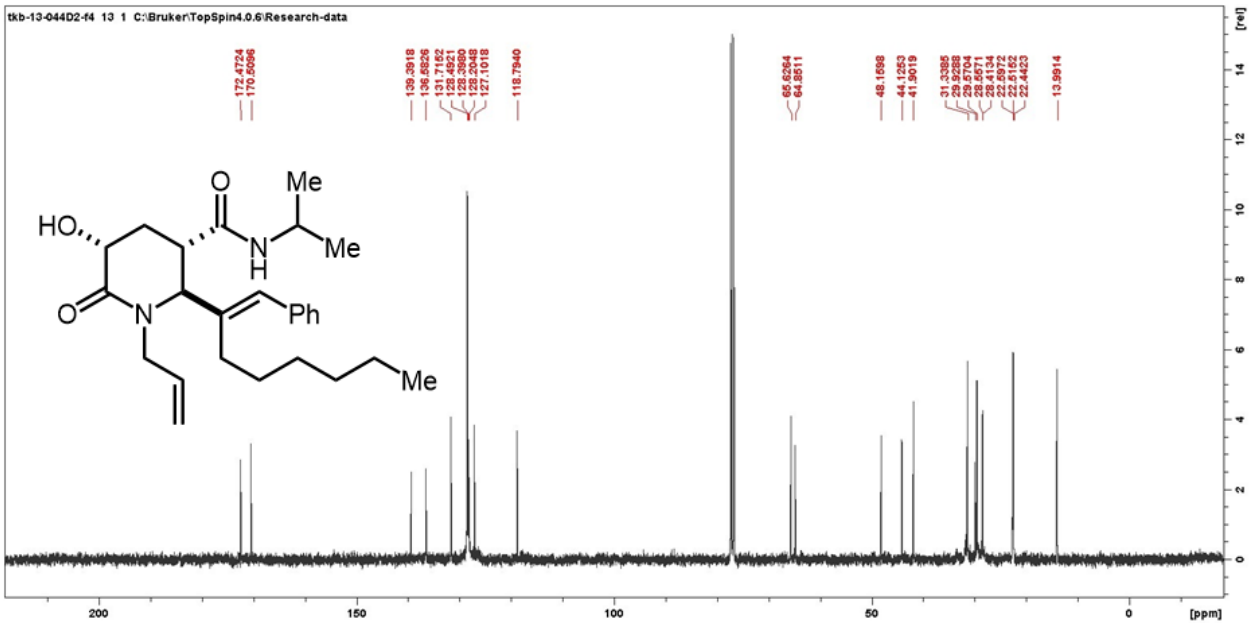
General Procedure A: Ring-opening aminolysis

To an oven-dried reaction tube equipped with a stir bar was added $Pd(TFA)_2$ (0.02 mmol, 6.6 mg, 2 mol%), PPh_3 (524.6 mg, 0.20 mmol, 20 mol%), and anhydrous 2-MeTHF (5.0 mL) at room temperature under a nitrogen atmosphere. The contents were stirred for 20 minutes and the bridged lactone (1.0 mmol, 1.0 equiv) dissolved in 2-MeTHF (5.0 mL) was added via syringe. The amine was then added (2 mmol, 2 equiv, solid amines were dissolved in 2-MeTHF). The reaction mixture was stirred for 18 h at 40 ^{o}C. The reaction mixture was filtered through a short pad of silica, which was rinsed with EtOAc (2 x 20 mL). The crude mixture was concentrated under reduced pressure and purified by flash column chromatography on silica gel eluting with hexanes/acetone.

Compound 7a

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 50:50). Yellowish oil. Yield = 375.4 mg, 88%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.43 – 7.11 (m, 5H), 6.26 (s, 1H), 6.17 (d, *J* = 5.2 Hz, 1H), 5.78 (dddd, *J* = 17.4, 10.2, 7.4, 4.9 Hz, 1H), 5.31 – 5.14 (m, 2H), 4.63 – 4.49 (m, 2H), 4.22 – 4.14 (m, 1H), 4.13 – 3.97 (m, 1H), 3.38 (dd, *J* = 15.0, 7.4 Hz, 1H), 2.74 – 2.59 (m, 1H), 2.49 – 2.29 (m, 2H), 2.11 – 1.92 (m, 2H), 1.29 – 1.09 (m, 14H), 0.86 (t, *J* = 6.1 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.47, 170.51, 139.40, 136.59, 131.72, 128.49, 128.47, 128.40, 128.21, 127.11, 118.80, 65.63, 64.85, 48.16, 44.13, 41.90, 31.34, 29.93, 29.57, 28.56, 28.42, 22.60, 22.52, 22.45, 14.00. **HRMS-EI$^+$** (*m/z*): calc for $C_{26}H_{38}N_2O_3$ $[M]^+$ 426.2882, found 426.2887.

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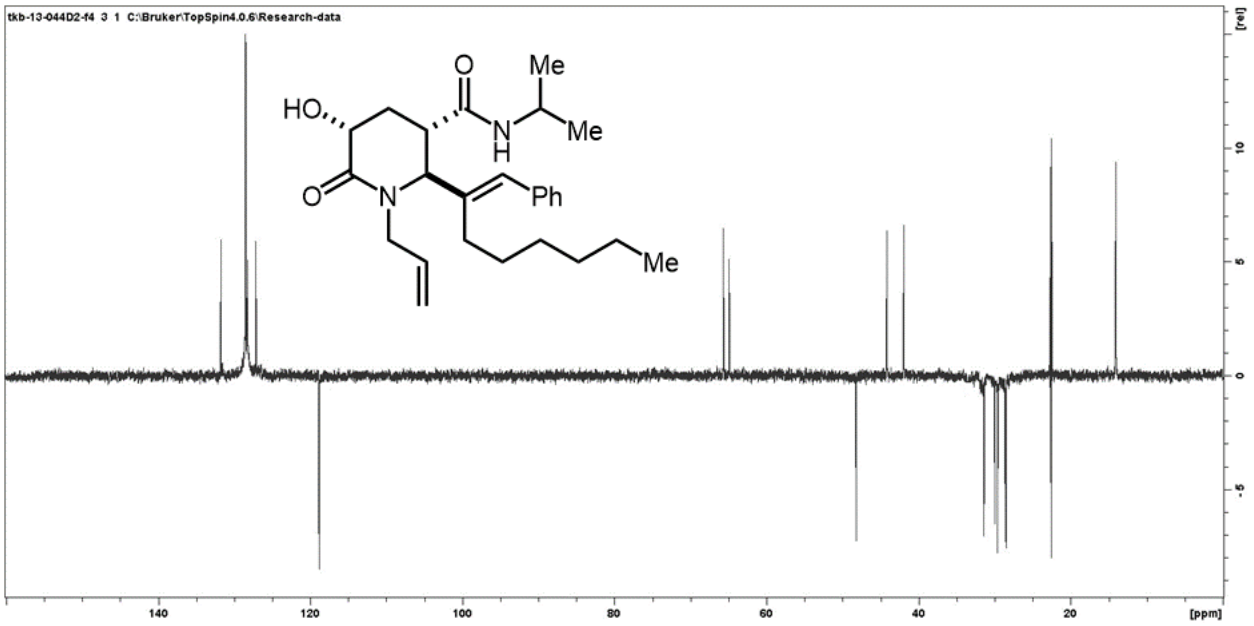
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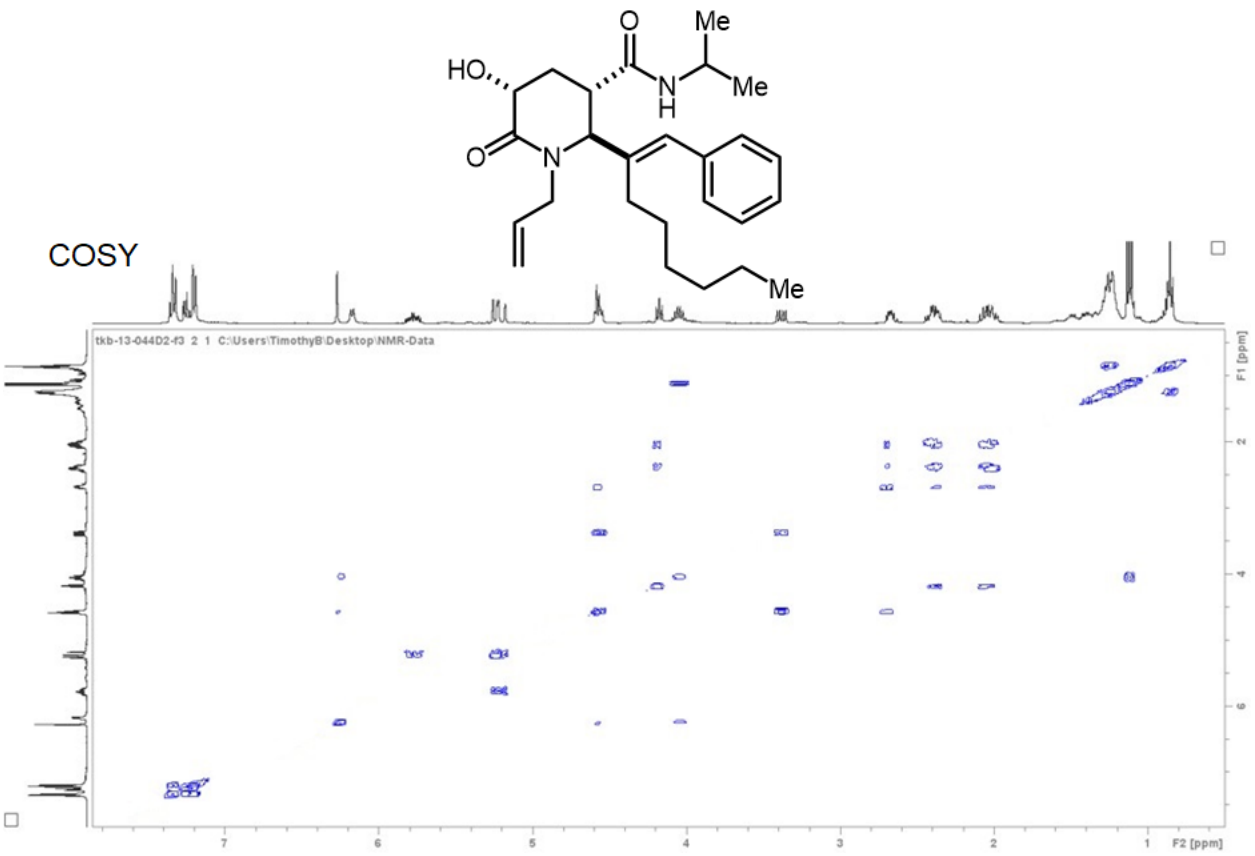
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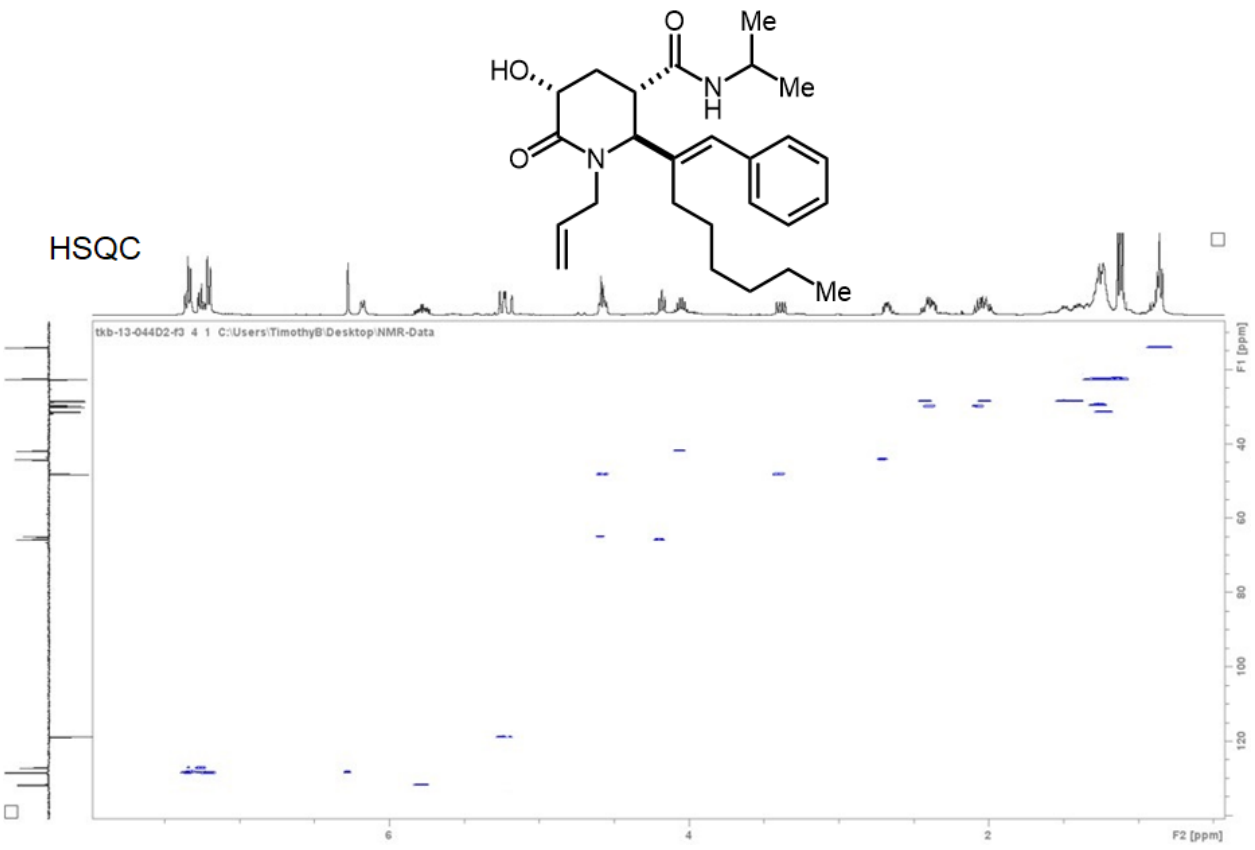
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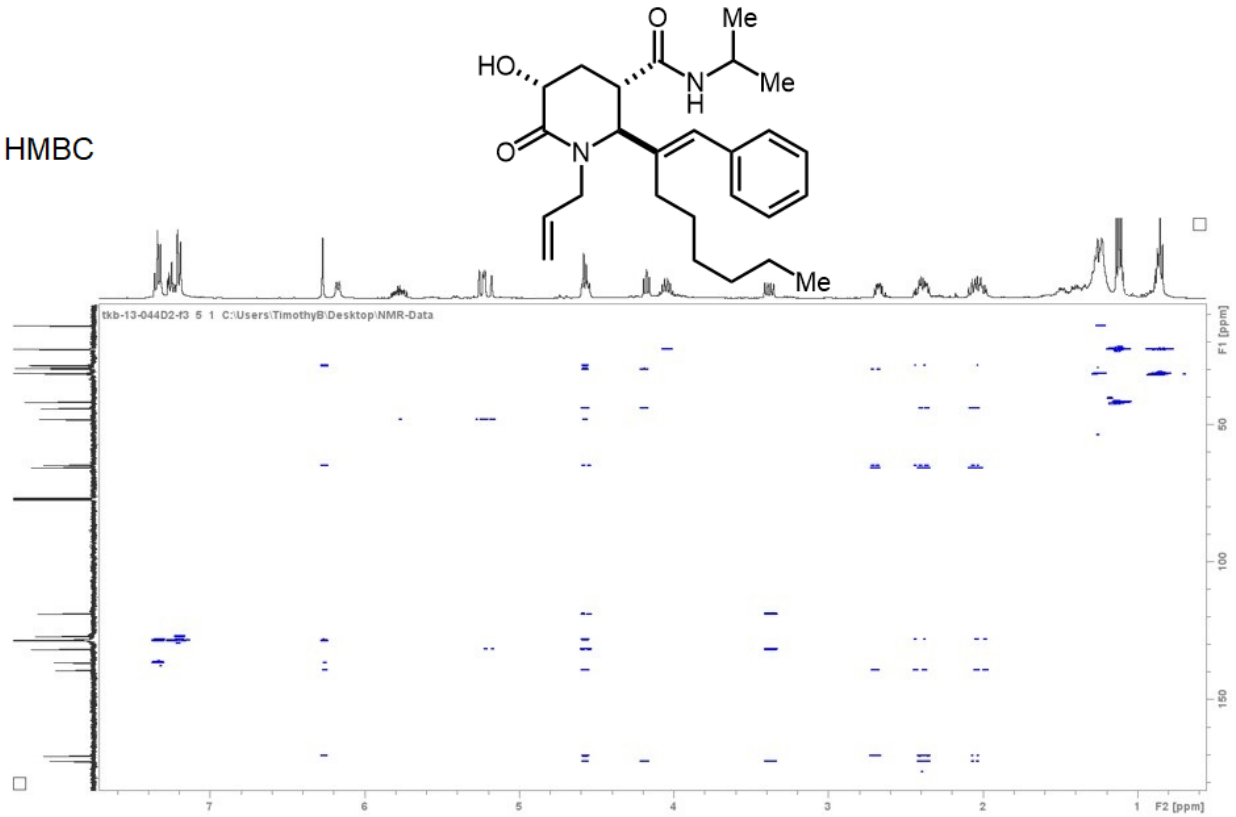
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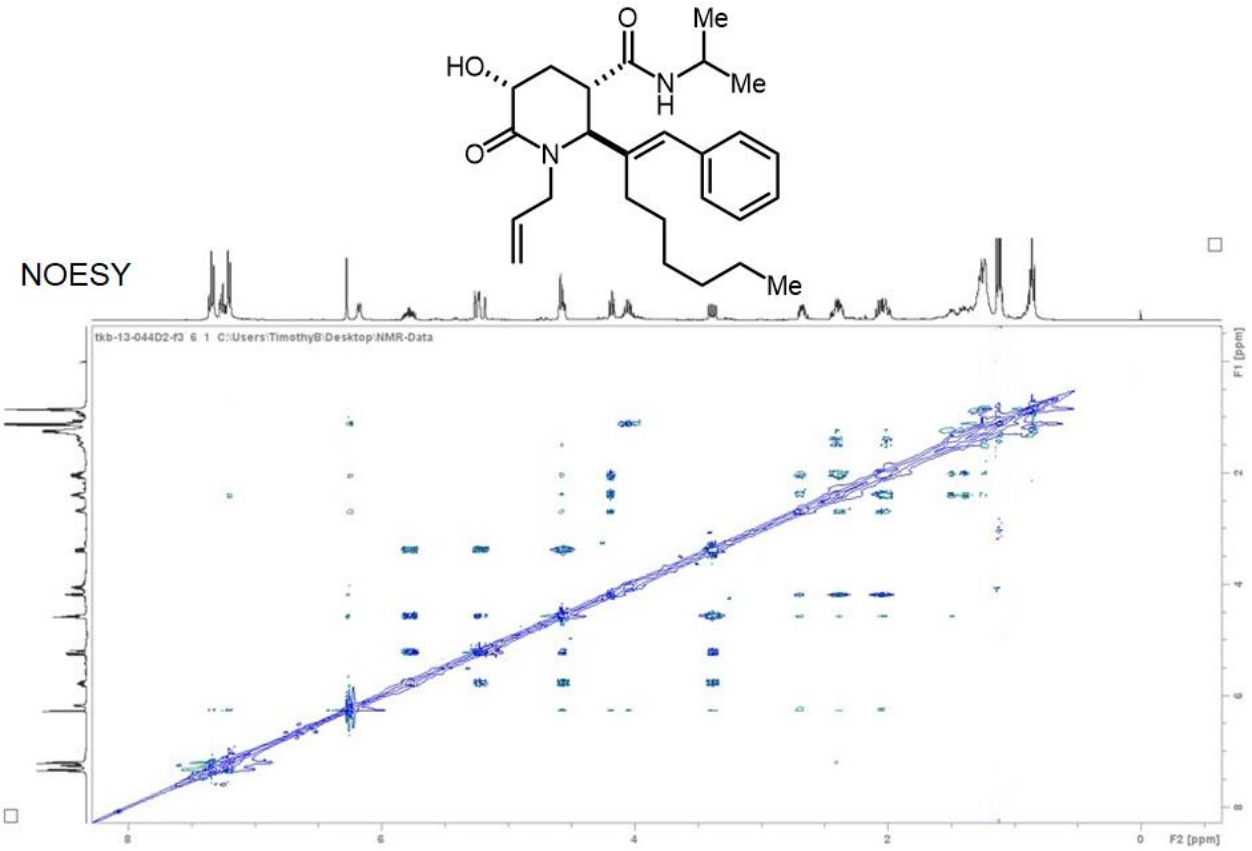
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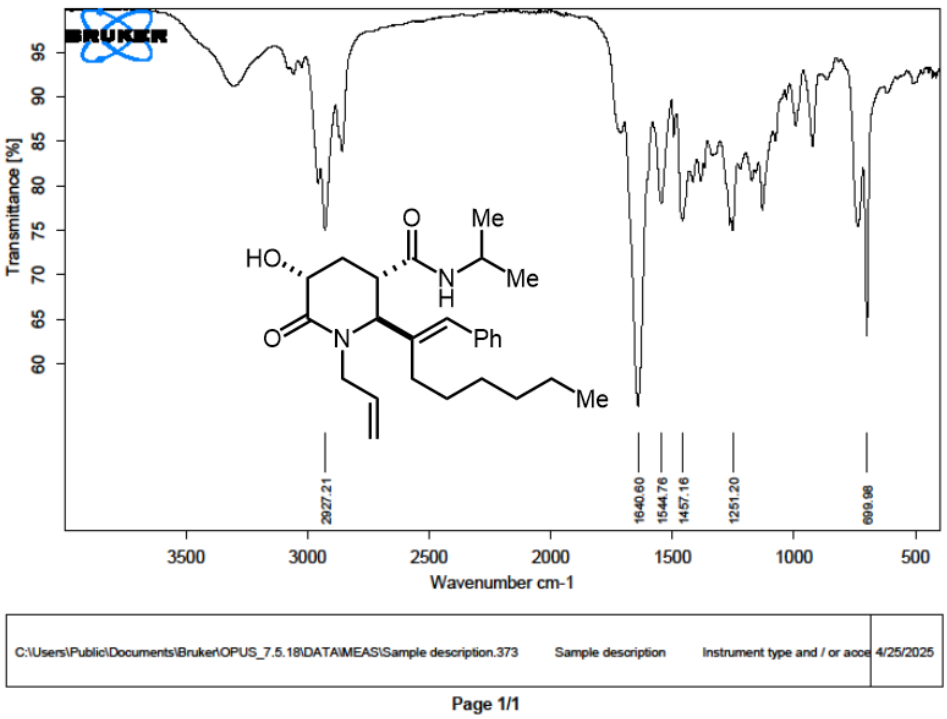
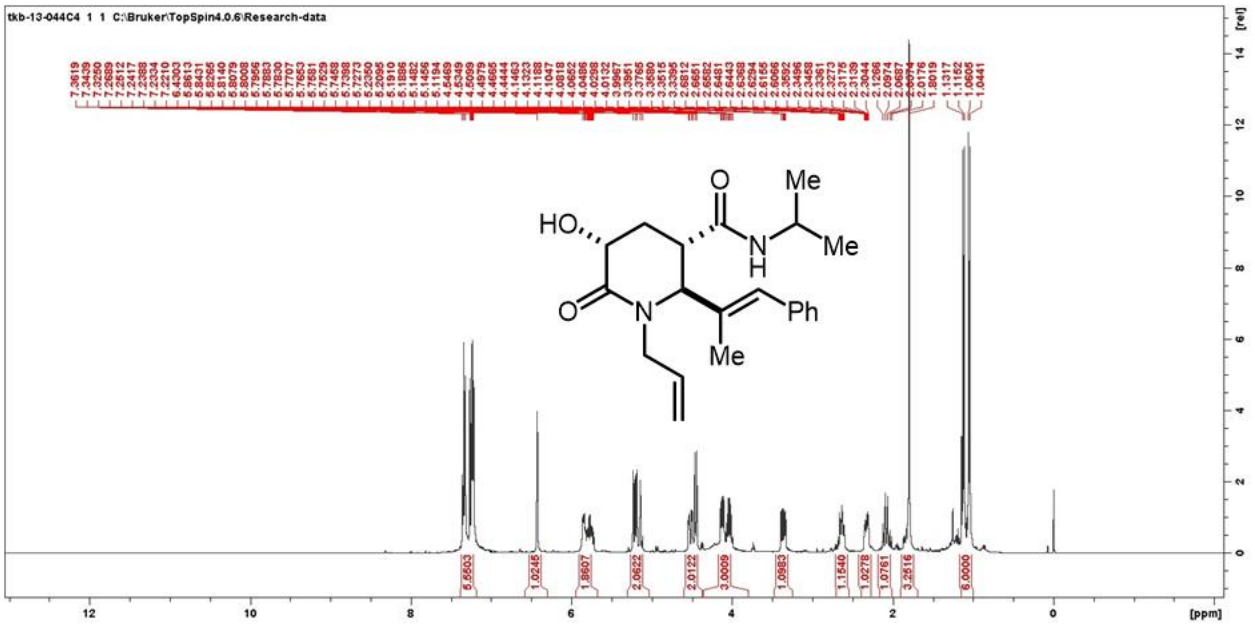
IR spectrum



Compound 7b

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 30:70). Pale yellow oil. Yield = 320.4 mg, 90%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.22 (m, 5H), 6.43 (s, 1H) 5.86 – 5.73 (m, 2H), 5.23 – 5.11 (m, 2H), 4.48 (ddt, *J* = 15.0, 4.7, 1.7 Hz, 1H), 4.37 (dt, *J* = 5.8, 1.5 Hz, 1H), 4.25 (dd, *J* = 11.1, 5.7 Hz, 1H), 3.92 (dp, *J* = 7.9, 6.5 Hz, 1H), 3.09 (dd, *J* = 15.0, 7.8 Hz, 1H), 2.71 – 2.59 (m, 2H), 2.36 – 2.25 (m, 1H), 2.24 – 2.05 (m, 1H), 1.80 (s, 3H), 1.03 (d, *J* = 6.6 Hz, 3H), 0.87 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.5, 170.0, 136.4, 133.6, 131.7, 130.6, 128.8, 128.3, 127.2, 118.7, 67.3, 66.6, 47.0, 43.9, 41.7, 31.3, 22.8, 22.5, 13.33. **HRMS-EI$^+$** (*m/z*): calc for $C_{21}H_{28}N_2O_3$ $[M]^+$ 356.2100, found 356.2107.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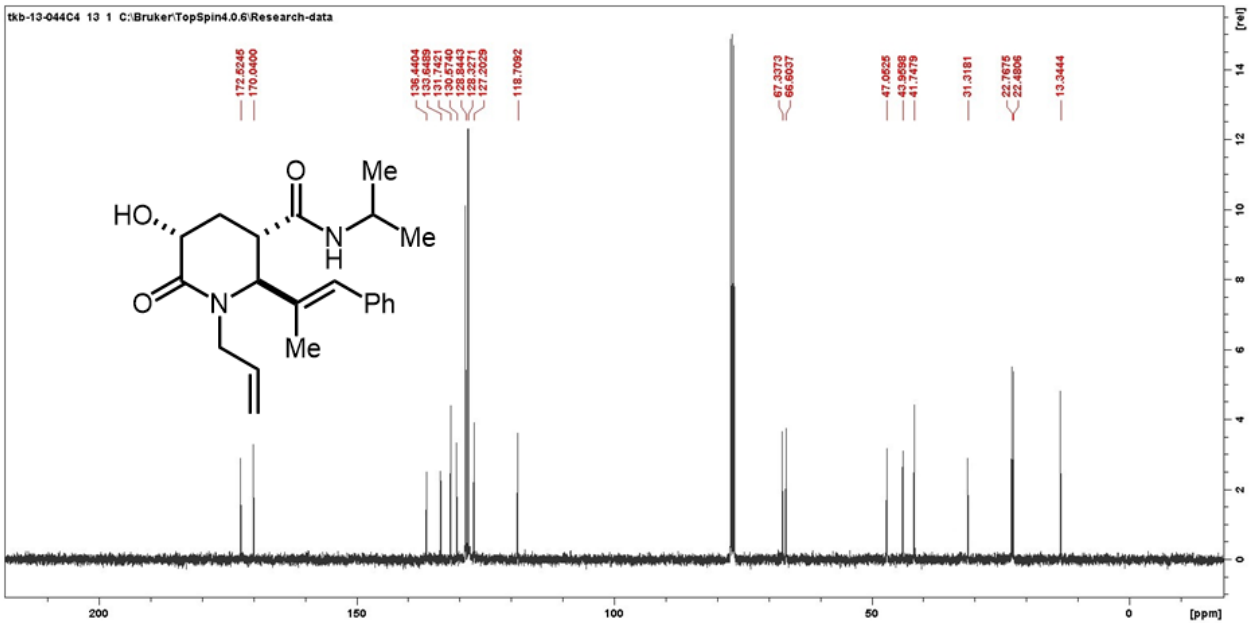
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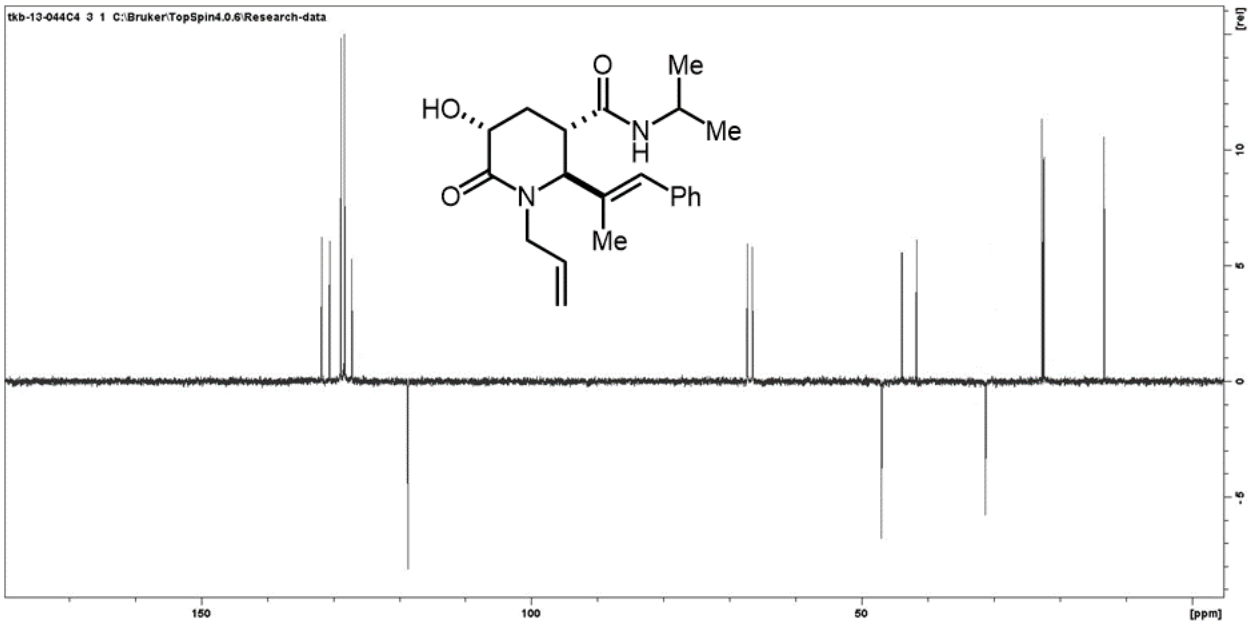
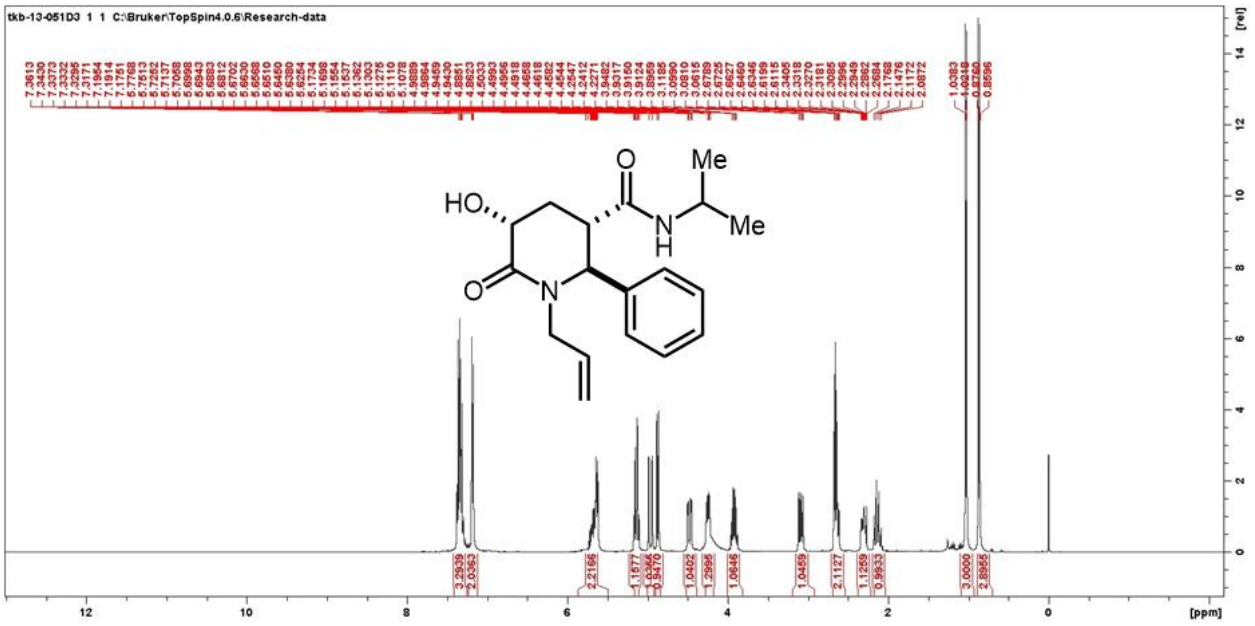


Compound 7c

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 25:75). Pale yellow oil. Yield = 291.1 mg, 92%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.23 (m, 3H), 7.22 – 7.11 (m, 2H), 5.71 – 5.60 (m, 2H), 5.20 – 5.08 (m, 1H), 4.97 (dq, *J* = 17.1, 1.4 Hz, 1H), 4.87 (d, *J* = 9.1 Hz, 1H), 4.48 (ddt, *J* = 15.0, 4.7, 1.7 Hz, 1H), 4.37 (dt, *J* = 5.8, 1.5 Hz, 1H), 4.25 (dd, *J* = 11.1, 5.7 Hz, 1H), 3.92 (dp, *J* = 7.9, 6.5 Hz, 1H), 3.09 (dd, *J* = 15.0, 7.8 Hz, 1H), 2.71 – 2.59 (m, 2H), 2.36 – 2.25 (m, 1H), 2.24 – 2.05 (m, 1H), 1.03 (d, *J* = 6.6 Hz, 3H), 0.87 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.57, 169.69, 139.56, 131.56, 129.02, 128.36, 127.03, 118.68, 66.91, 63.54, 49.01, 47.22, 41.43, 31.72, 22.33. **HRMS-EI$^+$** (*m/z*): calc for $C_{18}H_{24}N_2O_3$ $[M]^+$ 316.1787, found 316.1794.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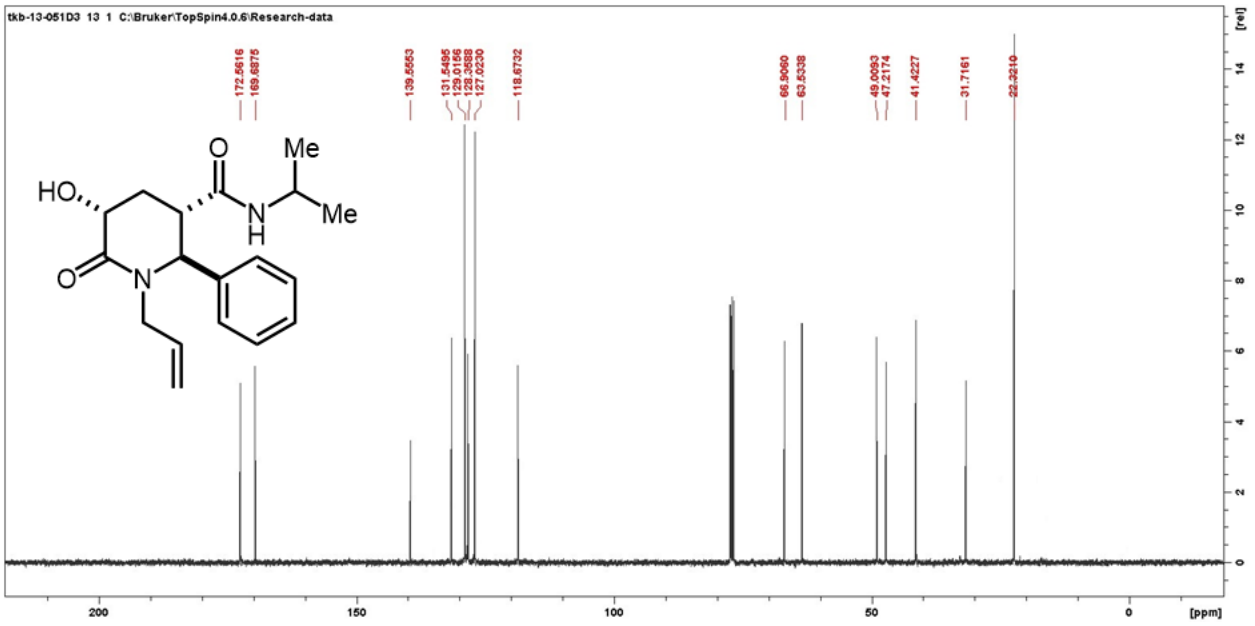
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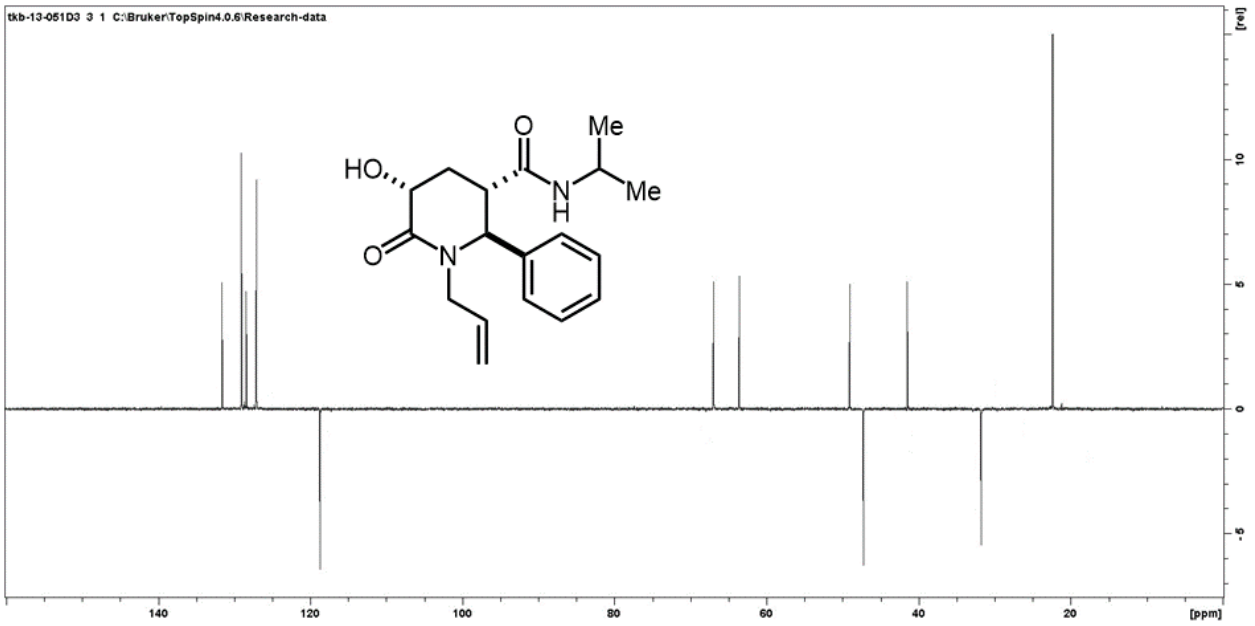
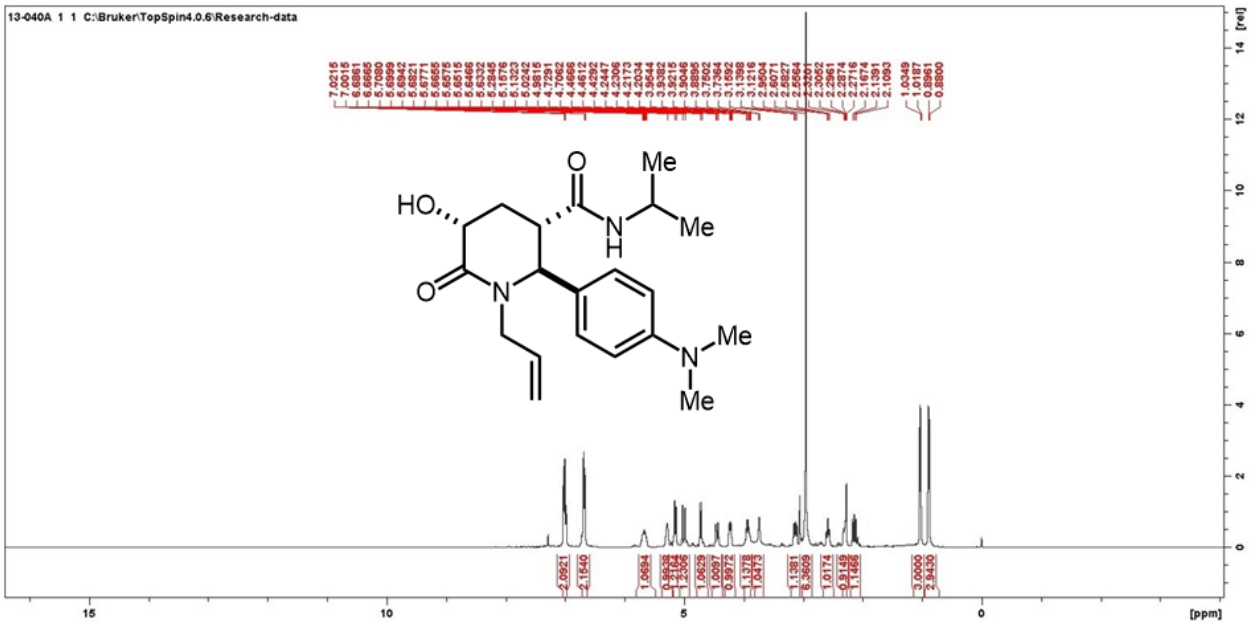


Compound 7d

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 10:90). Amorphous solid. Yield = 345.1 mg, 96%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.05 (d, *J* = 7.2 Hz, 2H), 6.74 (d, *J* = 6.2 Hz, 2H), 5.67 (dddd, *J* = 17.2, 9.4, 7.6, 4.4 Hz, 1H), 5.28 (t, *J* = 6.2 Hz, 1H), 5.15 (d, *J* = 10.2 Hz, 1H), 5.00 (d, *J* = 17.0 Hz, 1H), 4.72 (d, *J* = 9.4 Hz, 1H), 4.44 (ddd, *J* = 14.9, 4.5, 2.1 Hz, 1H), 4.22 (dd, *J* = 11.3, 5.7 Hz, 1H), 3.93 (dq, *J* = 12.9, 6.5 Hz, 1H), 3.74 (td, *J* = 5.4, 3.1 Hz, 1H), 3.13 (dd, *J* = 15.2, 7.8 Hz, 1H), 3.08 – 3.01 (m, 1H), 3.00 – 2.94 (m, 7H), 2.64 – 2.53 (m, 1H), 2.35 – 2.25 (m, 1H), 1.03 (dd, *J* = 6.6, 1.9 Hz, 3H), 0.89 (dd, *J* = 6.6, 1.9 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.42, 169.98, 150.47, 131.78, 127.95, 126.62, 125.49, 118.47, 112.58, 66.95, 63.24, 49.26, 46.94, 41.43, 40.45, 30.33, 22.43, 22.41. **HRMS-EI^{+}** (*m/z*): calc for $C_{20}H_{29}N_3O_3$ $[M]^{+}$ 359.2209, found 359.2214.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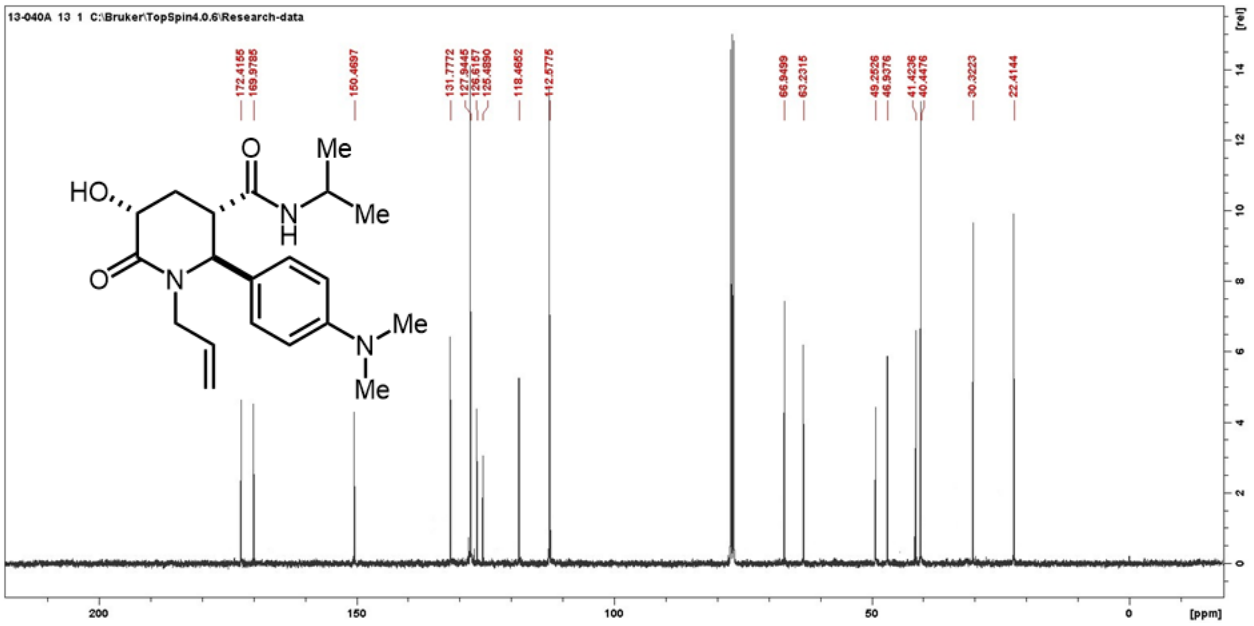
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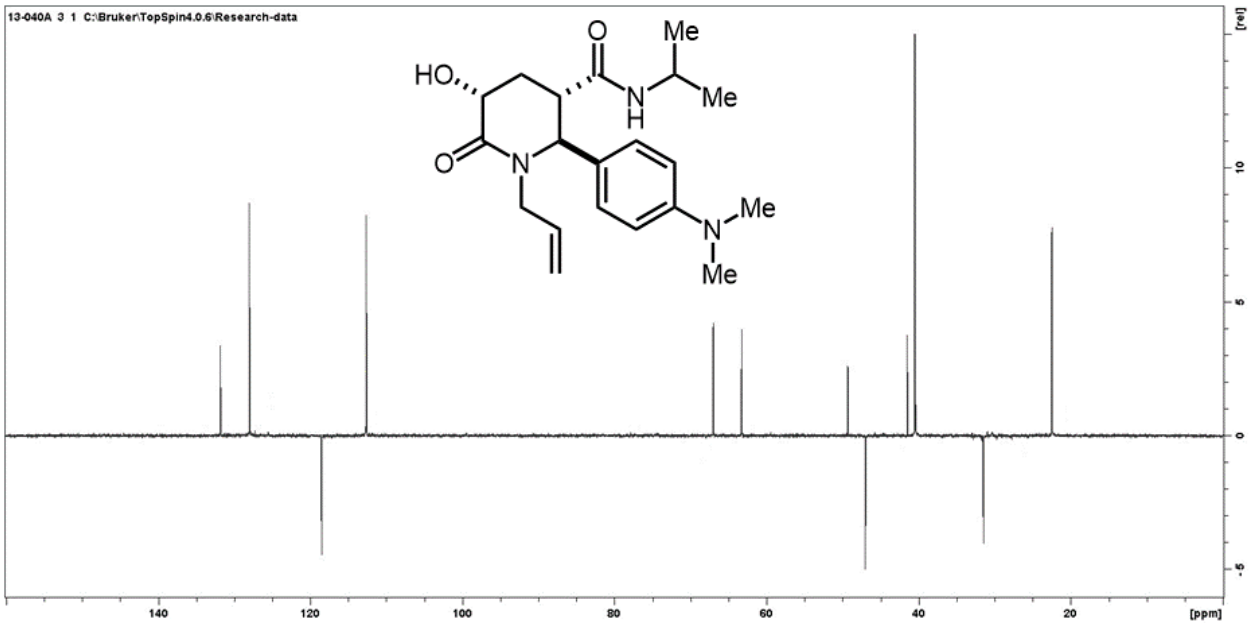
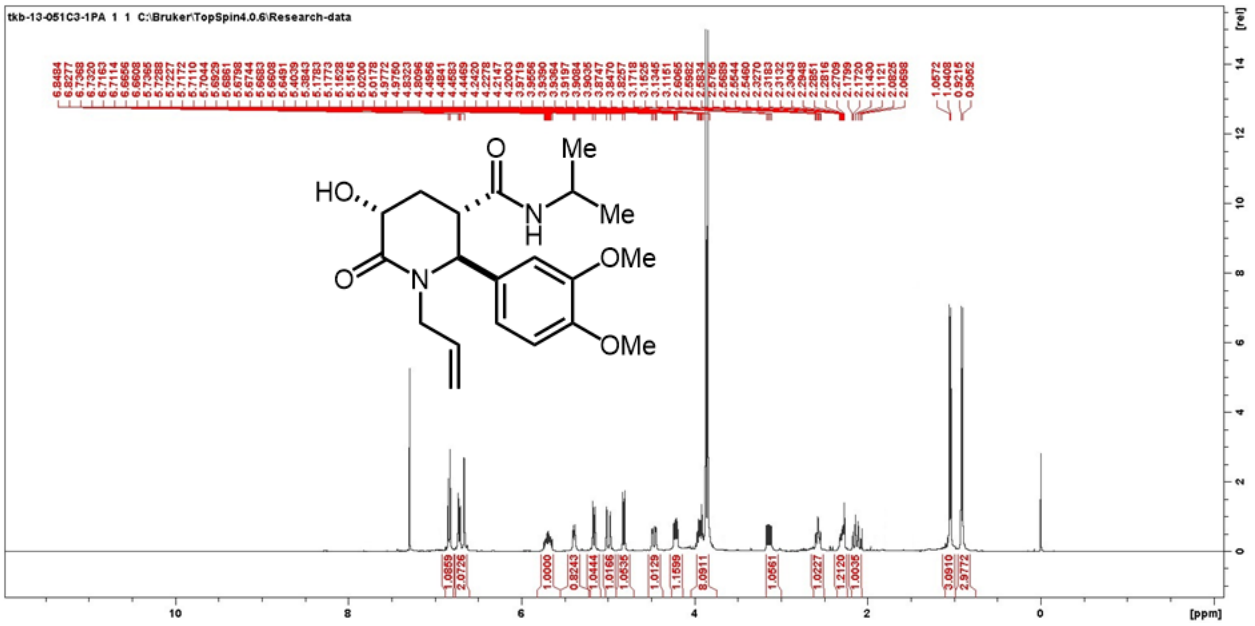


Compound 7e

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 20:80). Amorphous solid. Yield = 350.1 mg, 93%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.84 (d, *J* = 8.3 Hz, 1H), 6.72 (dd, *J* = 8.2, 2.1 Hz, 1H), 6.66 (d, *J* = 2.0 Hz, 1H), 5.70 (dddd, *J* = 17.5, 10.1, 7.7, 4.7 Hz, 1H), 5.39 (d, *J* = 8.0 Hz, 1H), 5.20 – 5.11 (m, 1H), 5.00 (dq, *J* = 17.2, 1.5 Hz, 1H), 4.82 (d, *J* = 9.1 Hz, 1H), 4.47 (ddt, *J* = 15.0, 4.8, 1.6 Hz, 1H), 4.22 (dd, *J* = 11.0, 5.7 Hz, 1H), 4.01 – 3.79 (m, 8H), 3.14 (dd, *J* = 14.9, 7.8 Hz, 1H), 2.58 (ddd, *J* = 12.2, 9.1, 3.4 Hz, 1H), 2.30 (ddd, *J* = 13.3, 5.8, 3.7 Hz, 1H), 2.20 – 2.04 (m, 1H), 1.05 (d, *J* = 6.6 Hz, 3H), 0.91 (d, *J* = 6.5 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.50, 169.75, 149.47, 148.97, 131.98, 131.67, 119.37, 118.66, 111.40, 109.83, 66.84, 63.37, 56.05, 55.97, 49.26, 47.23, 41.55, 31.59, 30.30, 22.45, 22.43, 22.35, 22.32. **HRMS-EI^{+}** (*m/z*): calc for $C_{20}H_{28}N_2O_5$ $[M]^{+}$ 376.1998, found 376.1995.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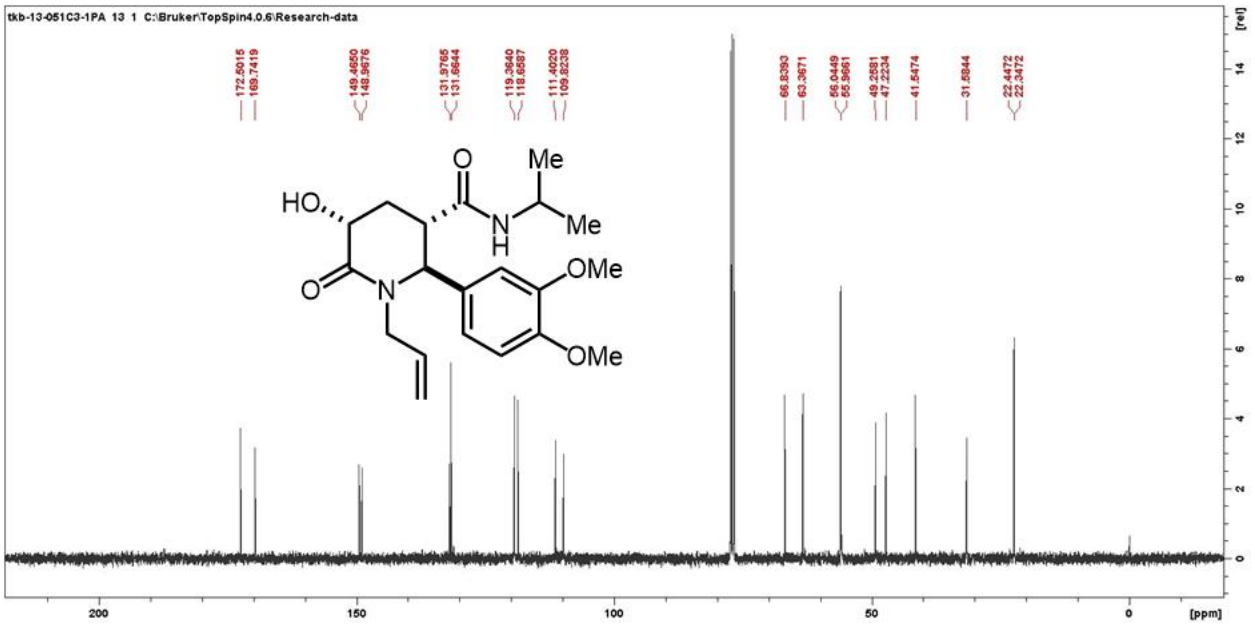
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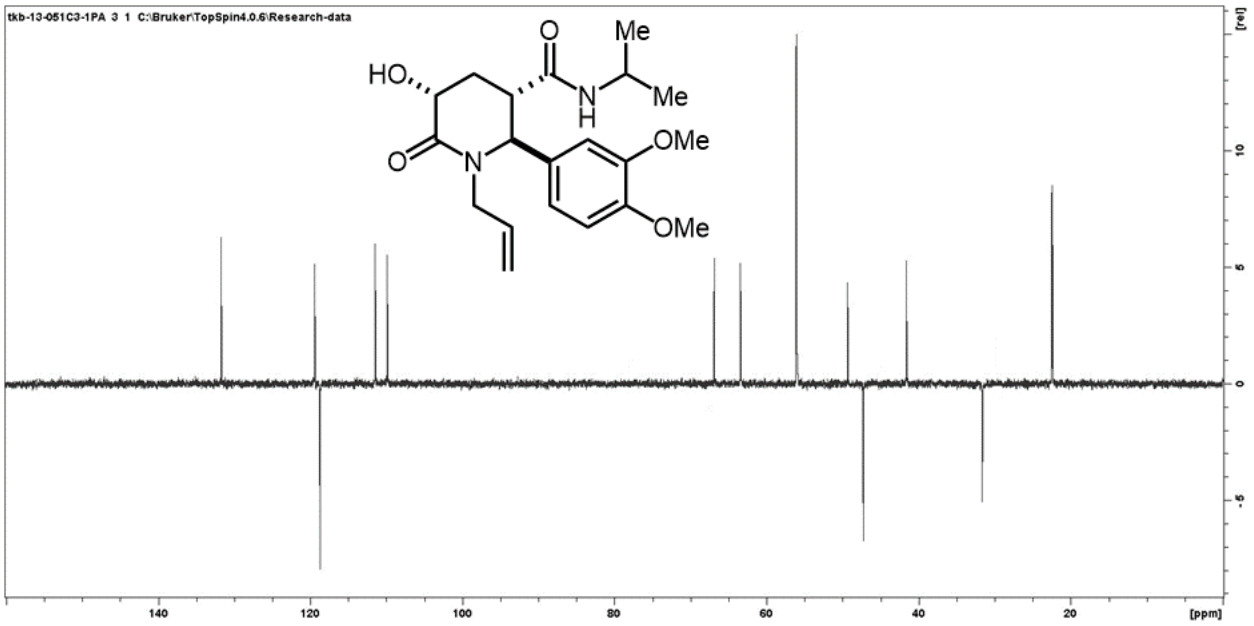
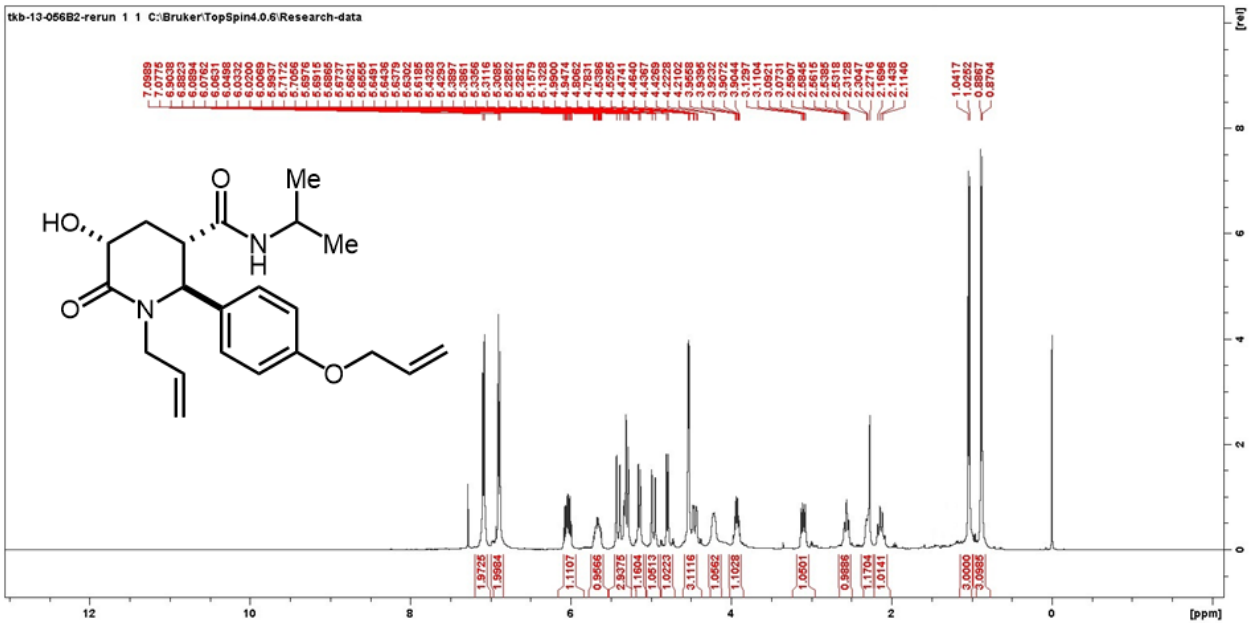


Compound 7f

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 50:50). Yellowish oil. Yield = 335.3 mg, 90%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.16 (d, *J* = 8.0 Hz, 2H), 6.86 (d, *J* = 8.0 Hz, 2H), 6.04 (ddt, *J* = 17.2, 10.5, 5.2 Hz, 1H), 5.67 (dddd, *J* = 17.5, 9.9, 7.9, 4.6 Hz, 1H), 5.41 (dq, *J* = 17.3, 1.7 Hz, 1H), 5.35 – 5.24 (m, 2H), 5.15 (d, *J* = 10.2 Hz, 1H), 5.01 – 4.90 (m, 1H), 4.79 (d, *J* = 9.2 Hz, 1H), 4.53 (dt, *J* = 5.4, 1.5 Hz, 2H), 4.45 (dd, *J* = 15.0, 4.6 Hz, 1H), 4.22 (dd, *J* = 11.3, 5.6 Hz, 1H), 4.00 – 3.85 (m, 1H), 3.10 (dd, *J* = 15.0, 7.7 Hz, 1H), 2.57 (ddt, *J* = 12.5, 9.6, 4.3 Hz, 1H), 2.28 (d, *J* = 6.0 Hz, 1H), 2.13 (q, *J* = 12.0 Hz, 1H), 1.03 (d, *J* = 6.5 Hz, 3H), 0.88 (d, *J* = 6.5 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.46, 169.69, 158.55, 132.93, 131.62, 131.57, 128.30, 125.51, 118.68, 117.87, 115.19, 68.87, 66.93, 63.10, 49.32, 47.08, 41.48, 31.59, 22.44, 22.39. **HRMS-EI$^+$** (*m/z*): calc for $C_{21}H_{28}N_2O_4$ $[M]^+$ 372.2049, found 372.2045.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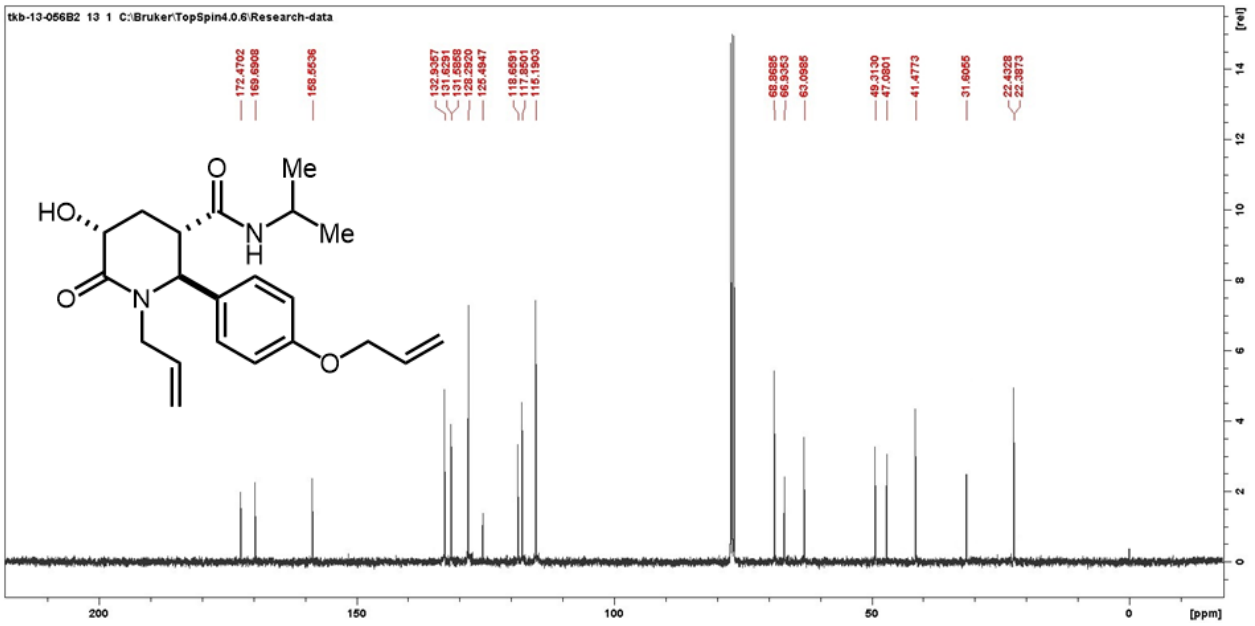
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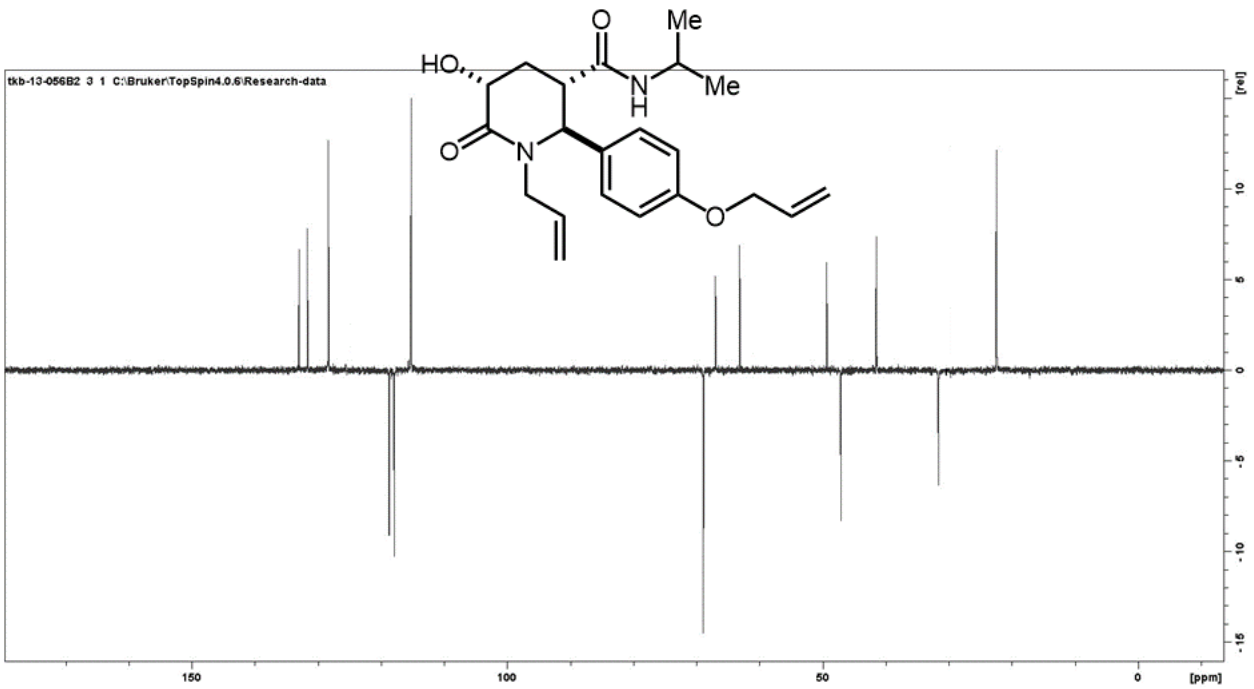
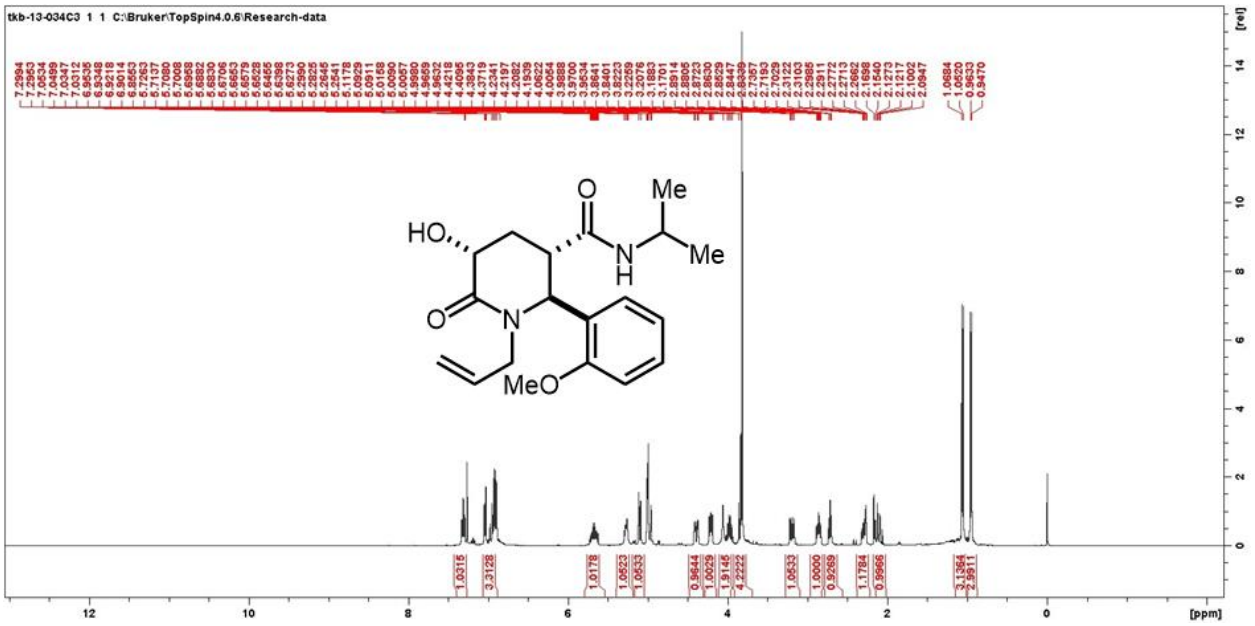


Compound 7g

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 50:50). Yellowish oil. Yield = 301.4 mg, 87%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 (td, *J* = 7.8, 1.8 Hz, 1H), 7.01 – 6.81 (m, 3H), 5.68 (dddd, *J* = 17.3, 10.2, 7.3, 5.0 Hz, 1H), 5.28 (q, *J* = 5.4 Hz, 1H), 5.10 (dt, *J* = 10.1, 1.3 Hz, 1H), 5.04 – 4.92 (m, 2H), 4.40 (ddt, *J* = 15.0, 5.0, 1.7 Hz, 1H), 4.21 (dd, *J* = 10.4, 5.7 Hz, 1H), 4.06 (s, 1H), 3.98 (ddt, *J* = 13.0, 7.8, 6.4 Hz, 1H), 3.85 (d, *J* = 9.6 Hz, 1H), 3.82 (s, 3H), 3.25 – 3.06 (m, 1H), 2.86 (ddd, *J* = 11.6, 7.6, 4.4 Hz, 1H), 2.72 (t, *J* = 6.6 Hz, 1H), 2.35 – 2.24 (m, 1H), 2.19 – 2.05 (m, 1H), 1.06 (d, *J* = 6.5 Hz, 3H), 0.96 (d, *J* = 6.5 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.39, 170.67, 156.98, 131.95, 129.77, 129.08, 126.97, 120.86, 118.18, 111.28, 66.37, 60.33, 55.40, 47.55, 45.56, 41.55, 31.37, 22.51. **HRMS-EI$^+$** (*m/z*): calc for $C_{19}H_{26}N_2O_4$ $[M]^+$ 346.1893, found 346.1899.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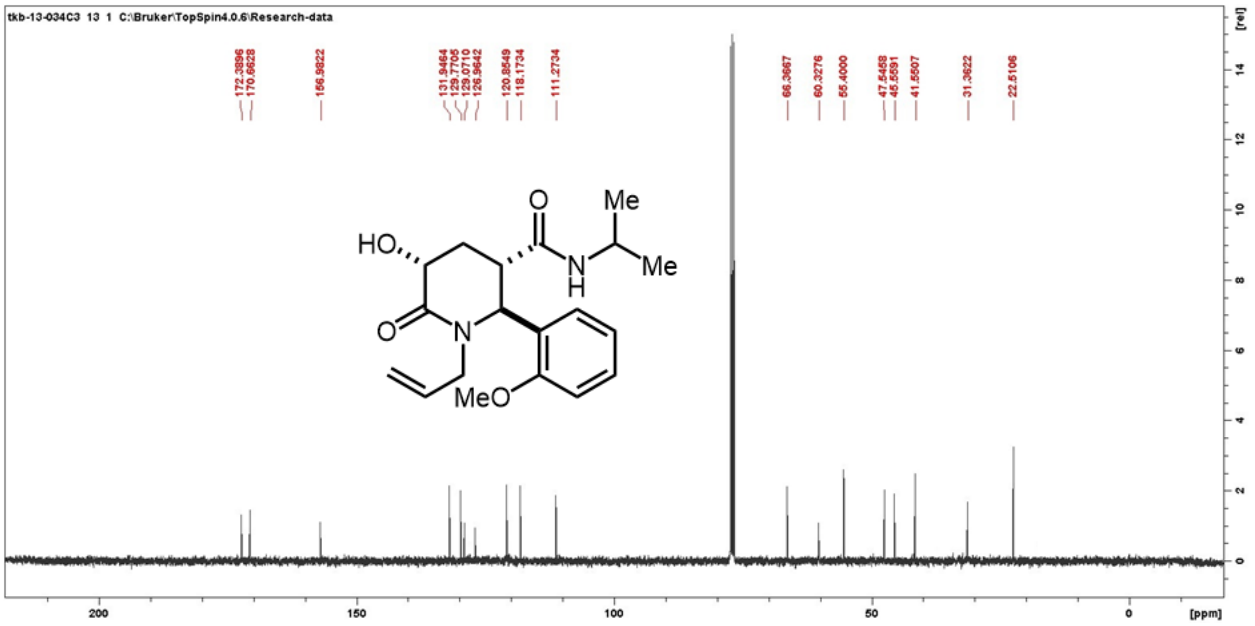
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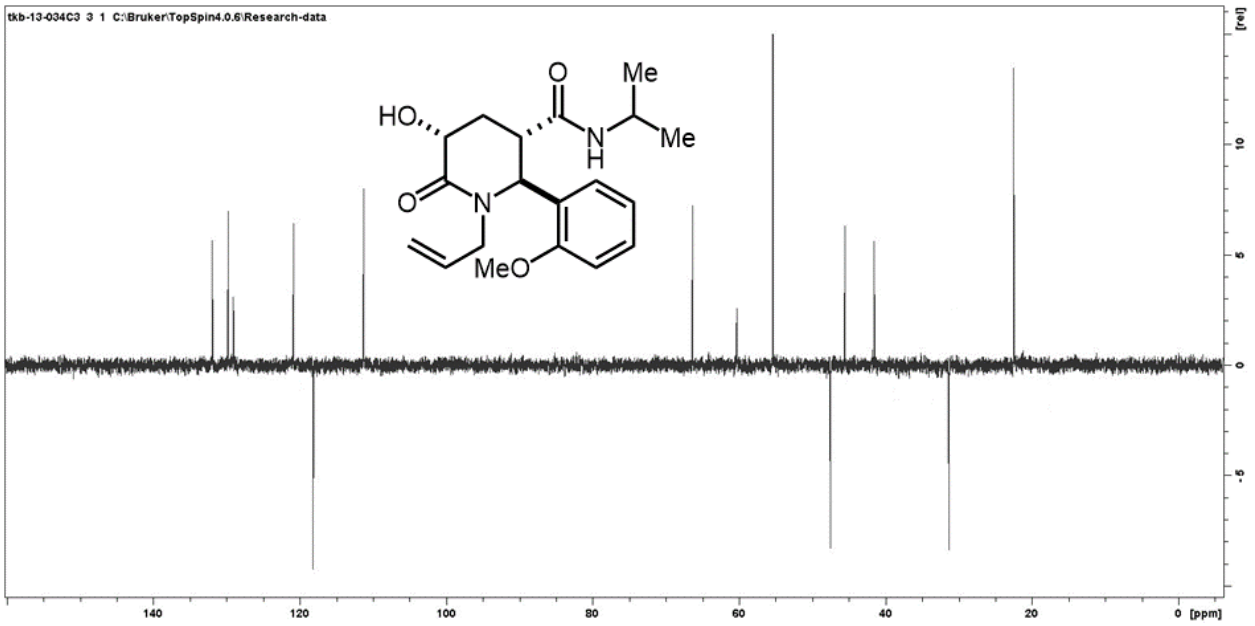
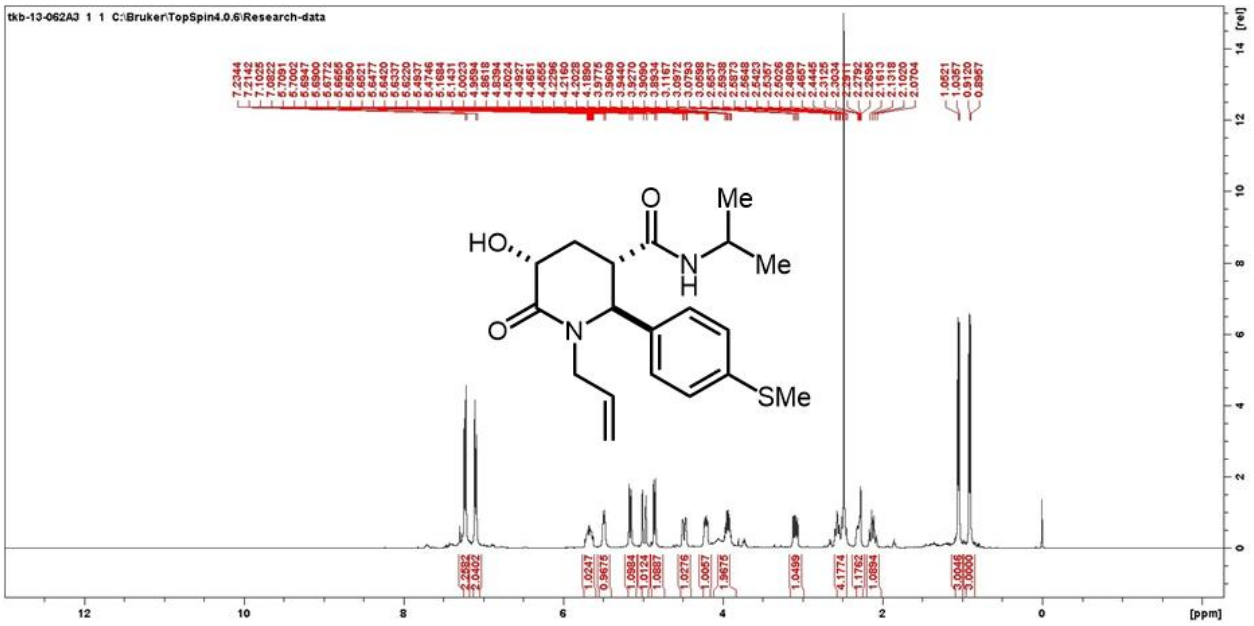


Compound 7h

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 50:50). Yellowish oil. Yield = 337.1 mg, 93%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (d, *J* = 8.0 Hz, 2H), 7.09 (d, *J* = 8.0 Hz, 2H), 5.67 (dddd, *J* = 17.5, 10.0, 7.7, 4.5 Hz, 1H), 5.48 (d, *J* = 7.9 Hz, 1H), 5.16 (d, *J* = 10.1 Hz, 1H), 4.98 (d, *J* = 17.2 Hz, 1H), 4.85 (d, *J* = 9.0 Hz, 1H), 4.48 (dd, *J* = 15.1, 4.5 Hz, 1H), 4.21 (dd, *J* = 11.0, 5.6 Hz, 1H), 4.06 (s, 1H), 4.00 – 3.85 (m, 1H), 3.09 (dd, *J* = 15.0, 7.8 Hz, 1H), 2.62 – 2.42 (m, 2H), 2.48 (s, 3H), 2.35 – 2.25 (m, 1H), 2.12 (q, *J* = 12.1 Hz, 1H), 1.04 (d, *J* = 6.6 Hz, 3H), 0.90 (d, *J* = 6.5 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.50, 169.59, 139.03, 136.21, 131.51, 127.50, 126.78, 118.83, 66.86, 63.04, 49.06, 47.23, 41.56, 31.65, 30.32, 22.41, 22.37, 15.57. **HRMS-EI$^+$** (*m/z*): calc for $C_{19}H_{26}N_2O_3S$ $[M]^+$ 362.1664, found 362.1669.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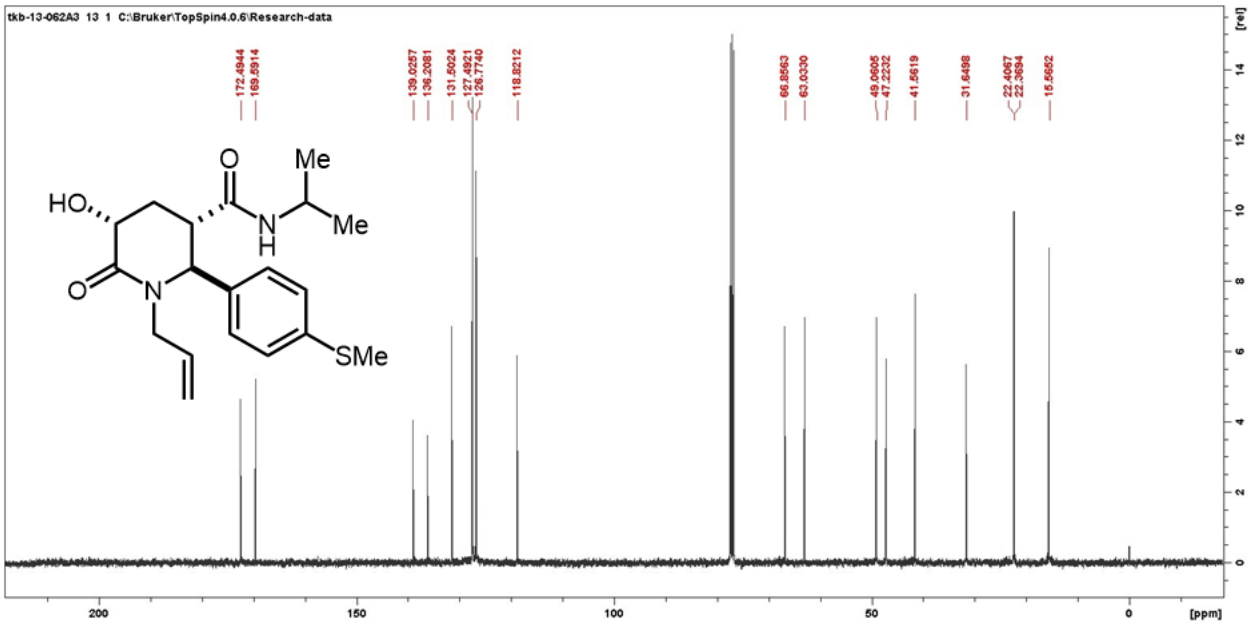
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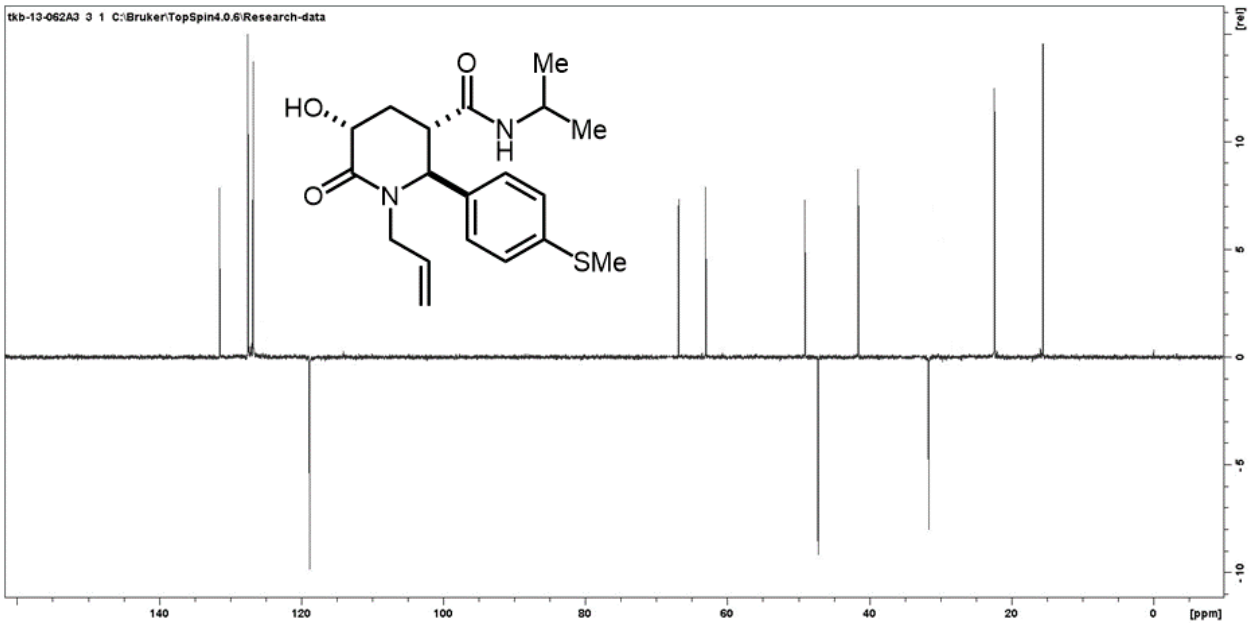
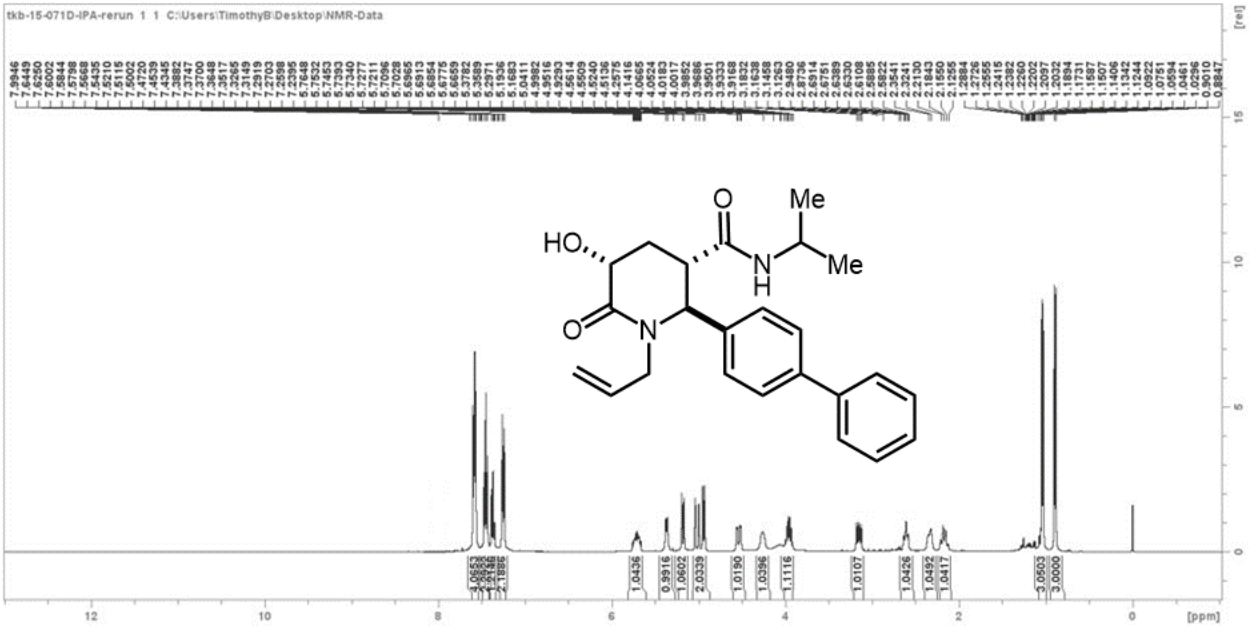


Compound 7i

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 30:70). Yellowish oil. Yield = 376.8 mg, 96%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.62 – 7.54 (m, 4H), 7.45 (dd, *J* = 8.5, 6.8 Hz, 2H), 7.41 – 7.33 (m, 1H), 7.25 (d, *J* = 8.0 Hz, 2H), 5.72 (dddd, *J* = 17.5, 10.1, 7.7, 4.6 Hz, 1H), 5.37 (d, *J* = 7.9 Hz, 1H), 5.22 – 5.14 (m, 1H), 5.02 (dd, *J* = 17.3, 1.7 Hz, 1H), 4.94 (d, *J* = 8.9 Hz, 1H), 4.54 (dd, *J* = 15.0, 4.6 Hz, 1H), 4.26 (s, 1H), 4.02 – 3.89 (m, 1H), 3.15 (dd, *J* = 15.0, 7.8 Hz, 1H), 2.95 (s, 2H), 2.87 (s, 2H), 2.61 (ddd, *J* = 12.1, 8.9, 3.2 Hz, 1H), 2.17 (q, *J* = 11.7 Hz, 1H), 1.04 (d, *J* = 6.6 Hz, 3H), 0.89 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.57, 169.61, 162.51, 141.35, 140.17, 138.56, 131.52, 128.92, 127.72, 127.70, 127.45, 127.00, 118.88, 66.89, 63.26, 49.23, 47.38, 41.59, 36.47, 31.56, 31.43, 22.43, 22.42. **HRMS-EI^{+}** (*m/z*): calc for $C_{24}H_{28}N_2O_3$ $[M]^{+}$ 392.2110, found 392.2114.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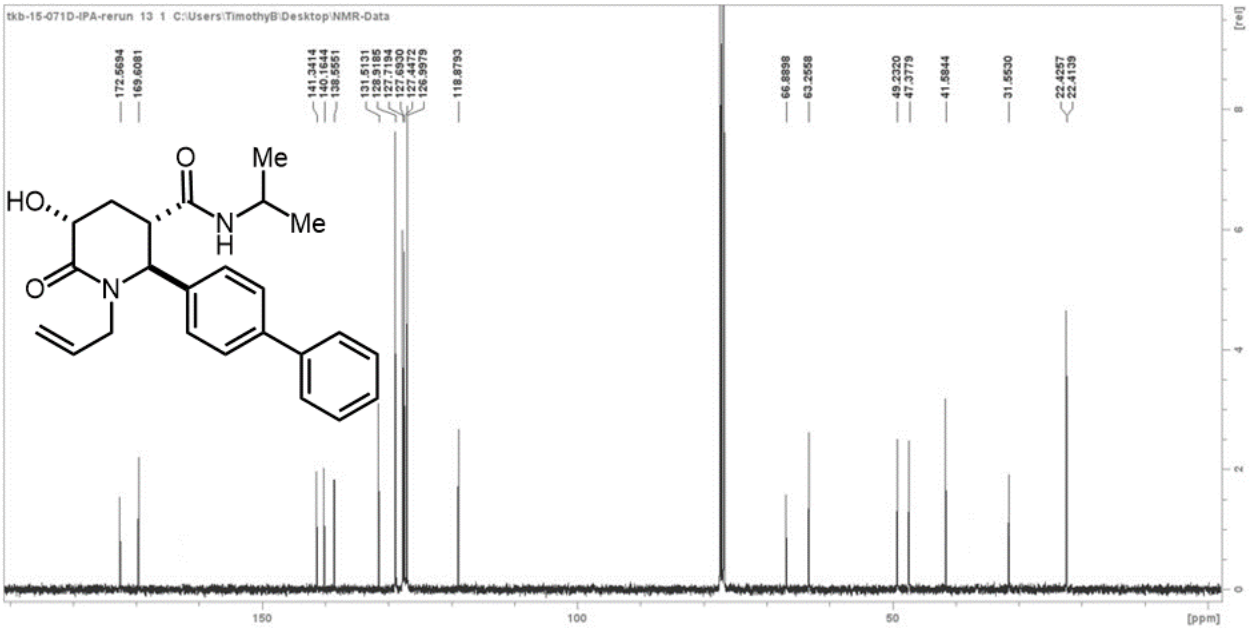
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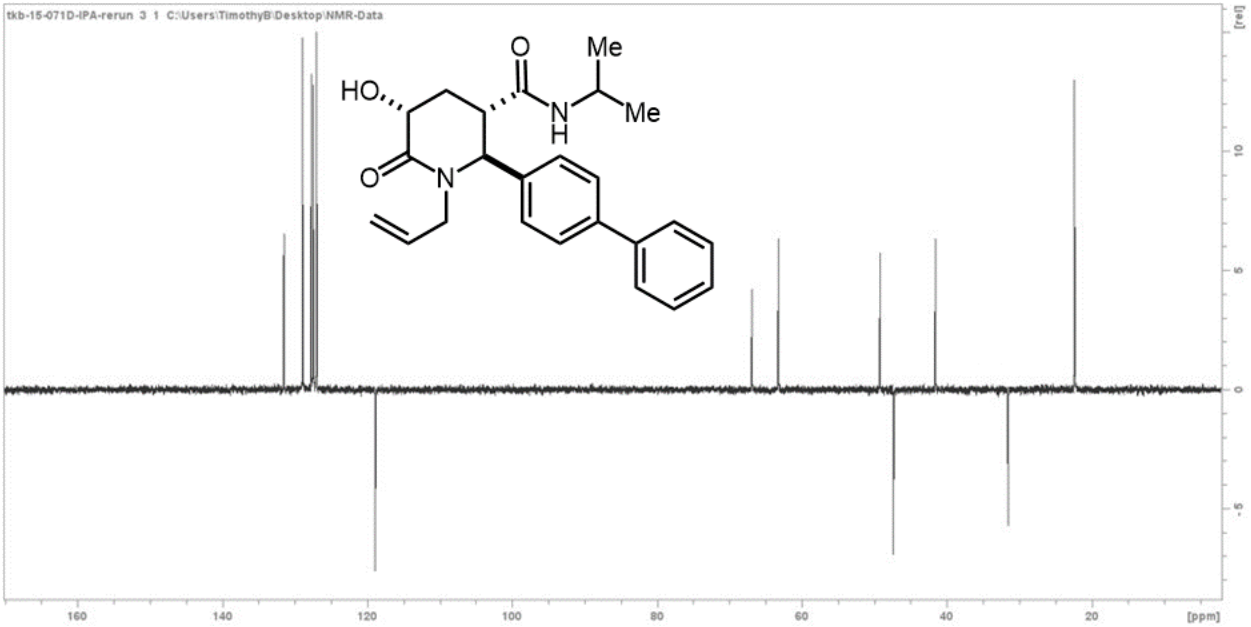
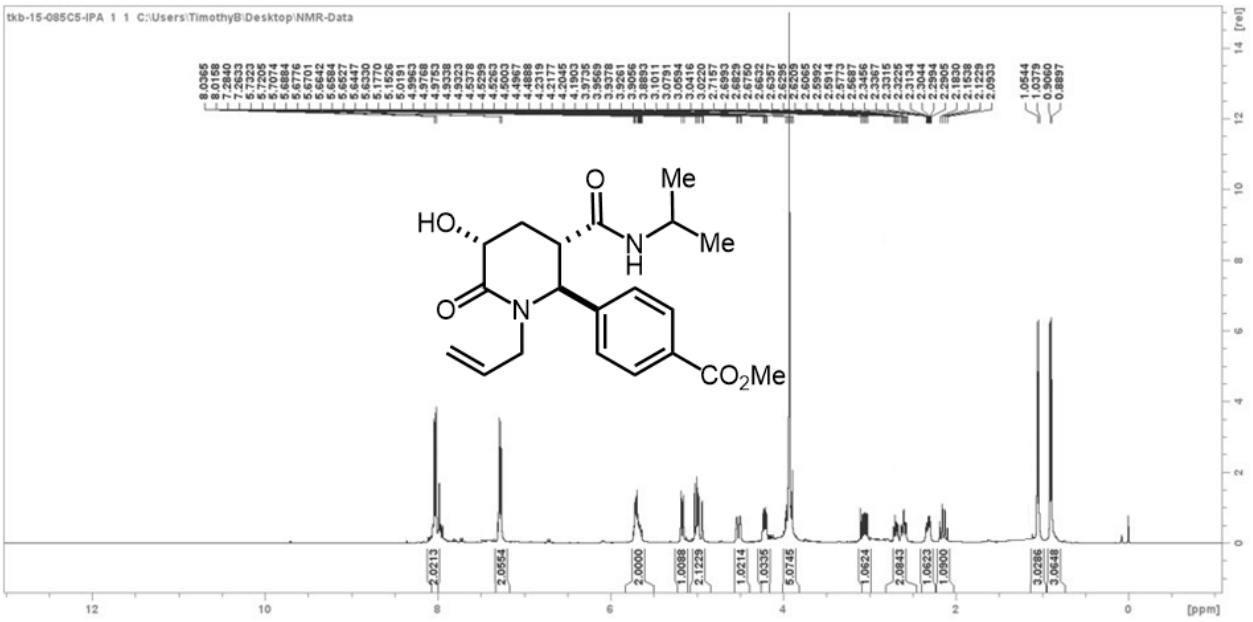


Compound 7j

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 50:50). Yellowish oil. Yield = 359.4 mg, 96%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, *J* = 7.6 Hz, 2H), 7.27 (d, *J* = 7.6 Hz, 2H), 5.75 – 5.67 (m, 2H), 5.16 (dd, *J* = 10.1, 1.4 Hz, 1H), 5.07 – 4.91 (m, 2H), 4.51 (ddt, *J* = 15.0, 4.8, 1.7 Hz, 1H), 4.21 (dd, *J* = 11.0, 5.7 Hz, 1H), 4.01 – 3.87 (m, 5H), 3.12 – 3.00 (m, 1H), 2.74 – 2.55 (m, 2H), 2.32 (ddd, *J* = 12.9, 5.7, 3.5 Hz, 1H), 2.14 (dt, *J* = 13.0, 11.4 Hz, 1H), 1.05 (d, *J* = 6.6 Hz, 3H), 0.90 (d, *J* = 6.5 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.57, 169.33, 166.46, 162.48, 144.83, 131.30, 130.30, 130.24, 127.08, 119.06, 66.82, 63.09, 52.28, 48.75, 47.50, 41.60, 31.77, 22.43, 22.30. **HRMS-EI^{+}** (*m/z*): calc for $C_{20}H_{26}N_2O_5$ $[M]^{+}$ 374.1842, found 374.1847.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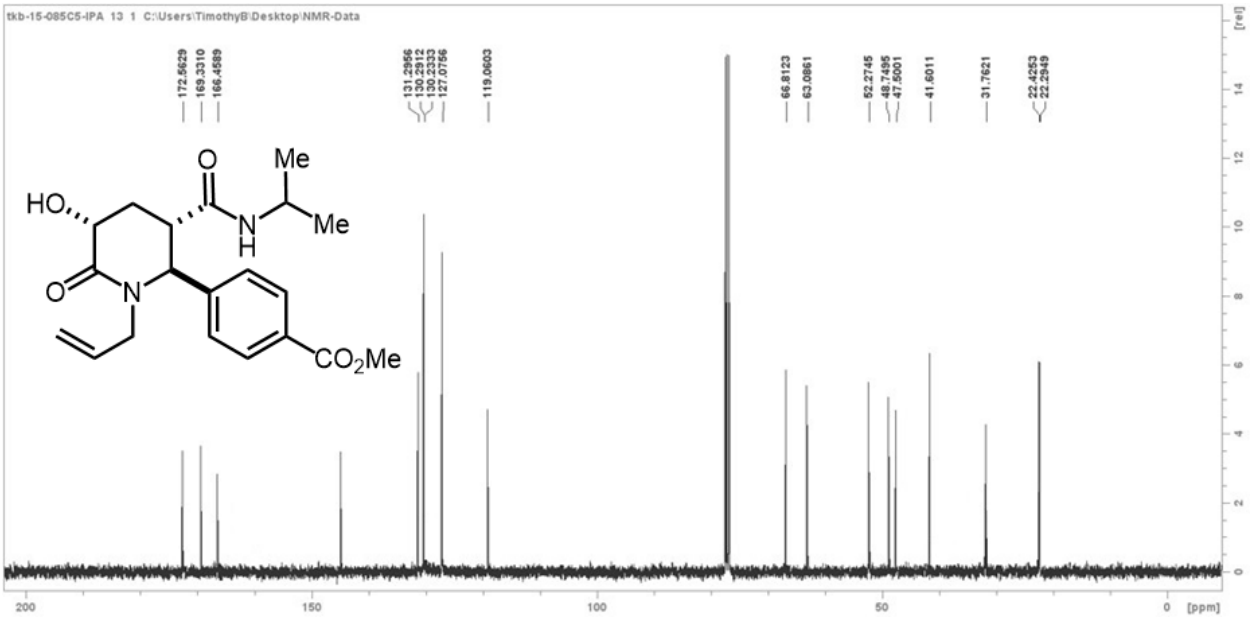
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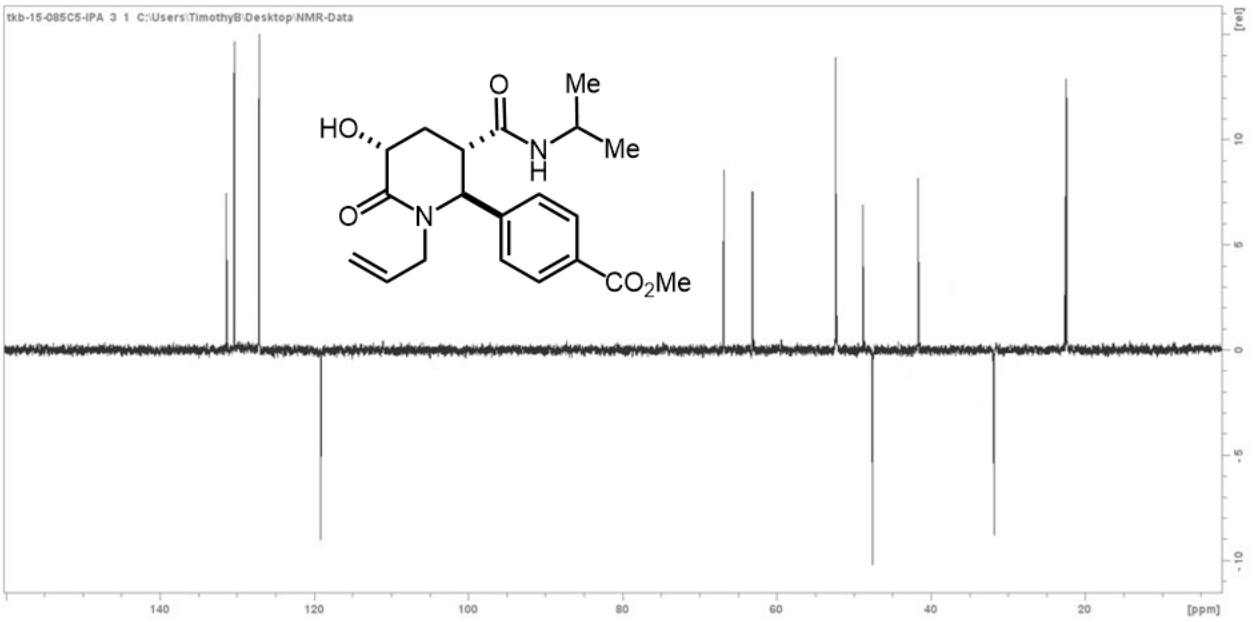
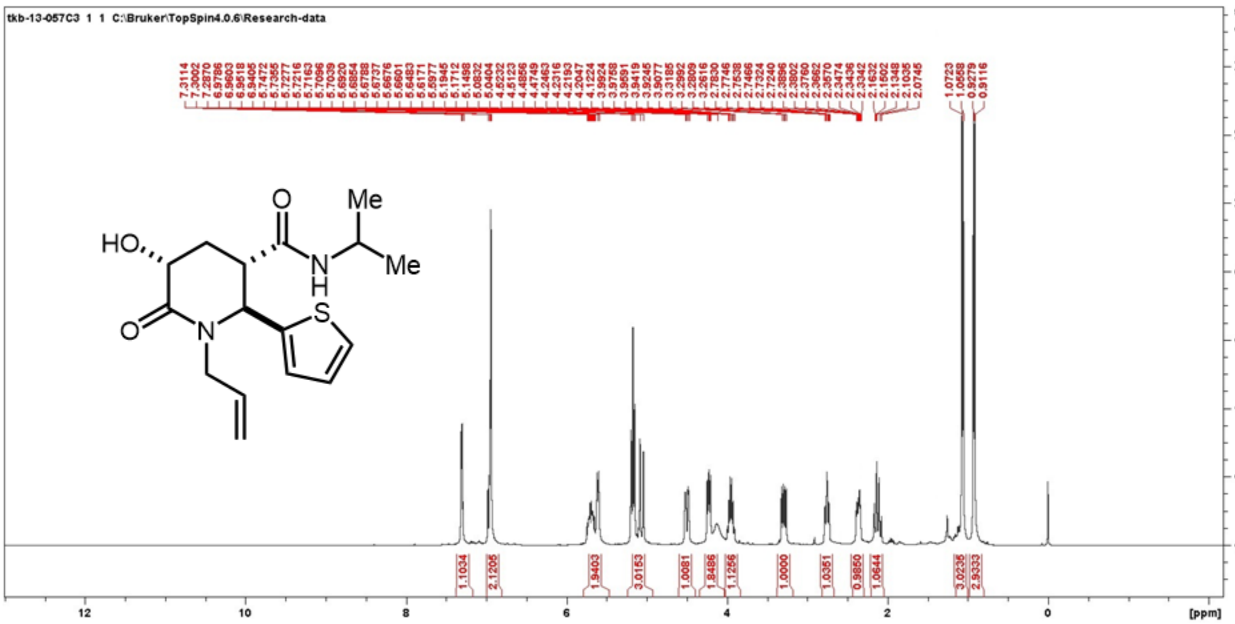


Compound 7k

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 40:60). Amorphous solid. Yield = 296.6 mg, 92%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.30 (dd, *J* = 4.6, 1.7 Hz, 1H), 7.00 – 6.92 (m, 2H), 5.70 (dddd, *J* = 17.5, 10.0, 7.6, 4.6 Hz, 1H), 5.61 (d, *J* = 8.0 Hz, 1H), 5.17 (t, *J* = 8.9 Hz, 2H), 5.06 (dd, *J* = 17.2, 1.8 Hz, 1H), 4.50 (dd, *J* = 15.2, 4.5 Hz, 1H), 4.23 (dd, *J* = 10.9, 5.9 Hz, 1H), 4.12 (s, 1H), 3.94 (dp, *J* = 13.4, 6.7 Hz, 1H), 3.29 (dd, *J* = 15.1, 7.7 Hz, 1H), 2.81 – 2.63 (m, 1H), 2.36 (ddd, *J* = 13.1, 5.9, 3.6 Hz, 1H), 2.12 (dt, *J* = 12.9, 11.2 Hz, 1H), 1.06 (d, *J* = 6.5 Hz, 3H), 0.92 (d, *J* = 6.5 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.08, 169.42, 143.26, 131.57, 127.09, 126.95, 125.90, 125.49, 118.82, 66.72, 59.15, 49.66, 47.26, 41.58, 31.48, 30.32, 22.43, 22.39. **HRMS-EI^{+}** (*m/z*): calc for $C_{16}H_{22}N_2O_3S$ $[M]^{+}$ 322.1351, found 322.1355.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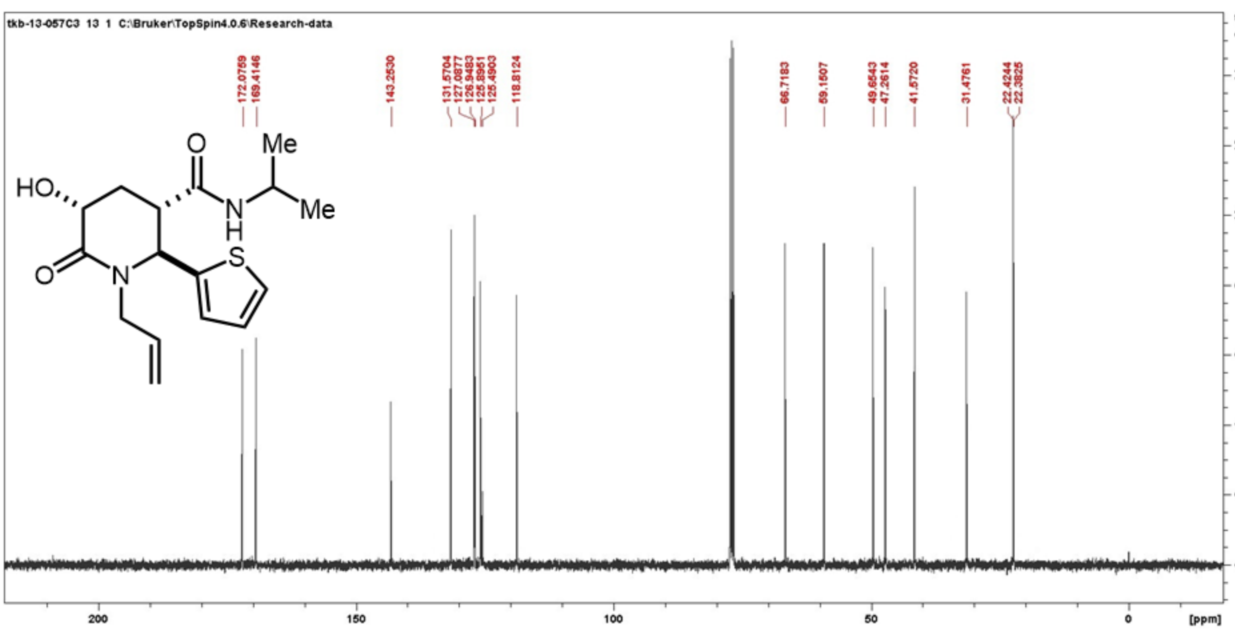
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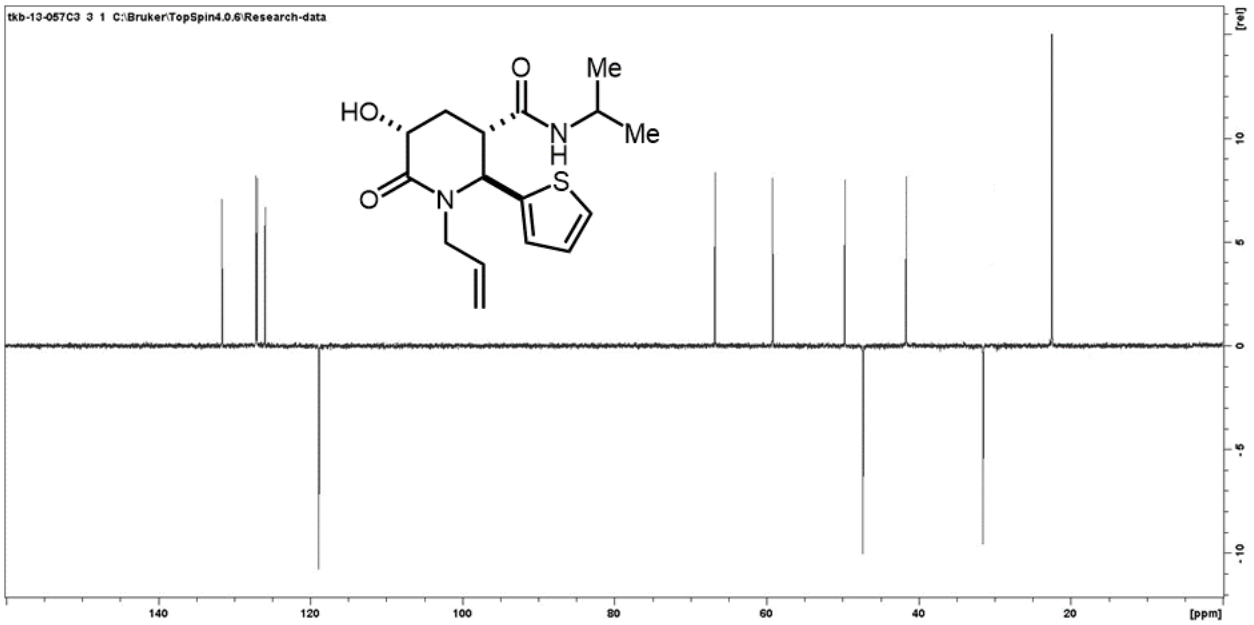
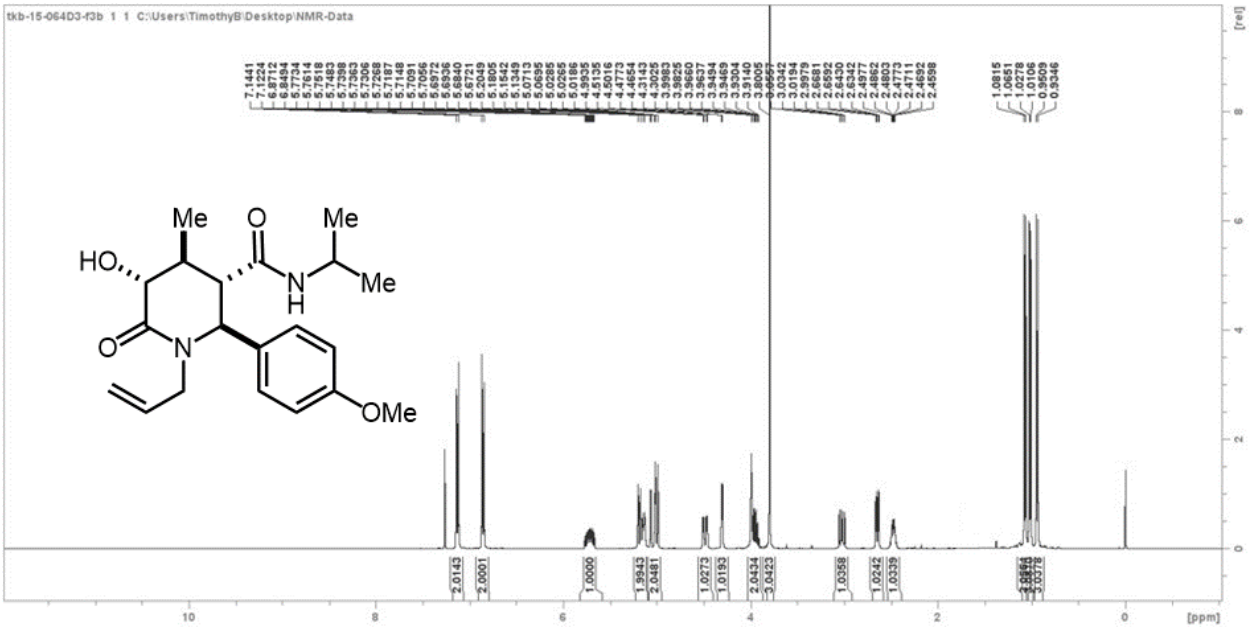


Compound 7l

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 50:50). Greenish-yellow oil. Yield = 317.2 mg, 88%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.13 (d, *J* = 7.5 Hz, 2H), 6.88 (d, *J* = 7.5 Hz, 2H), 5.72 (dddd, *J* = 17.0, 10.0, 8.6, 4.8 Hz, 1H), 5.23 – 5.11 (m, 2H), 5.10 – 5.00 (m, 1H), 5.01 (d, *J* = 10.1 Hz, 1H), 4.49 (ddt, *J* = 14.5, 4.8, 1.5 Hz, 1H), 4.31 (d, *J* = 4.7 Hz, 1H), 4.00 (d, *J* = 1.6 Hz, 1H), 3.95 (dq, *J* = 7.9, 6.6 Hz, 1H), 3.80 (s, 3H), 3.03 (dd, *J* = 14.5, 8.6 Hz, 1H), 2.65 (dd, *J* = 10.0, 3.5 Hz, 1H), 2.54 – 2.42 (m, 1H), 1.07 (d, *J* = 6.5 Hz, 3H), 1.02 (d, *J* = 6.9 Hz, 3H), 0.94 (d, *J* = 6.5 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.35, 168.82, 159.37, 131.87, 131.26, 128.53, 119.76, 114.30, 70.52, 58.80, 55.31, 52.66, 47.17, 41.58, 36.31, 22.52, 22.45, 8.24. **HRMS-EI$^+$** (*m/z*): calc for $C_{20}H_{28}N_2O_4$ $[M]^+$ 360.2049, found 360.2054.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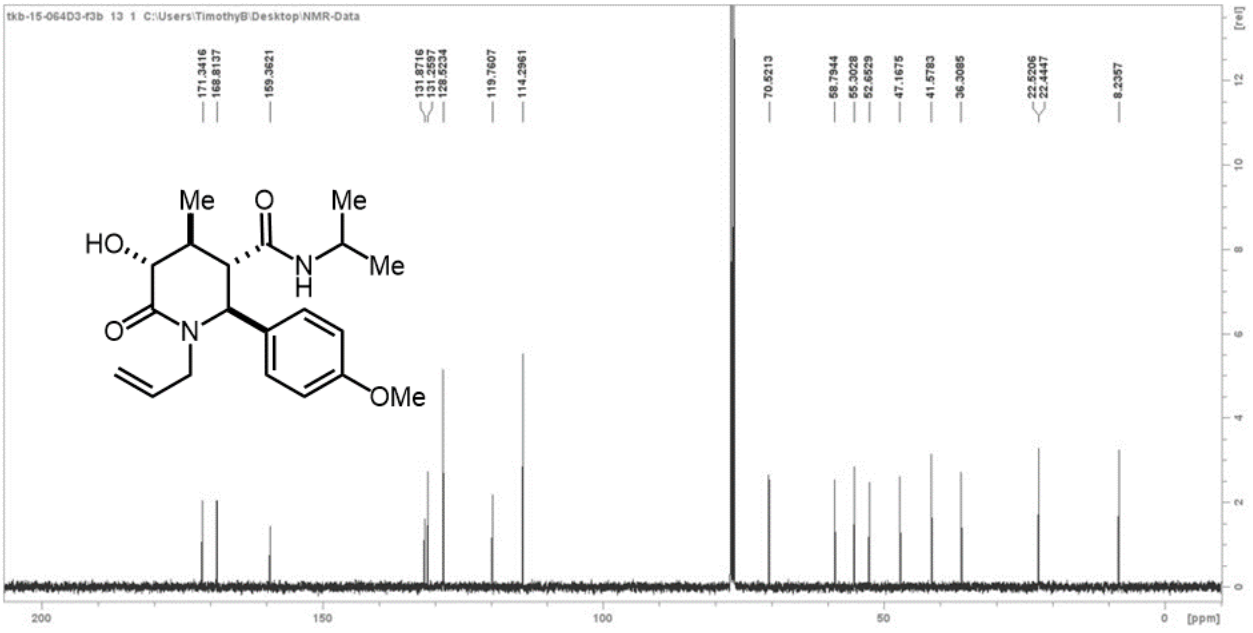
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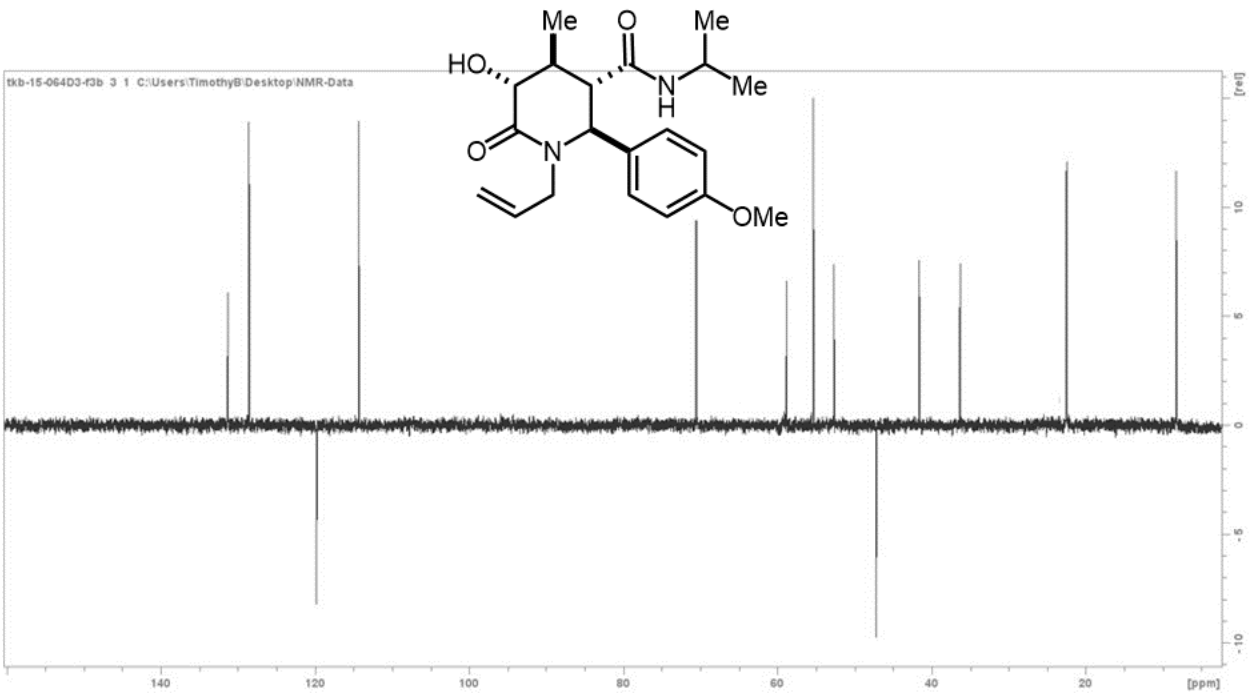
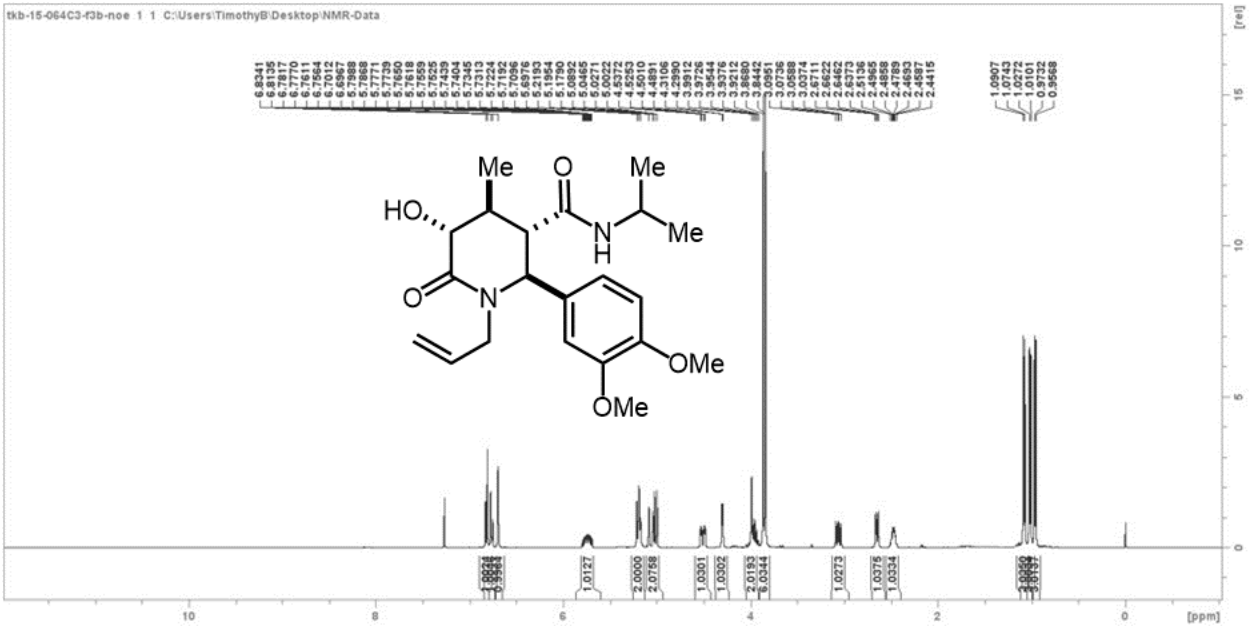
OMe
[ppm]



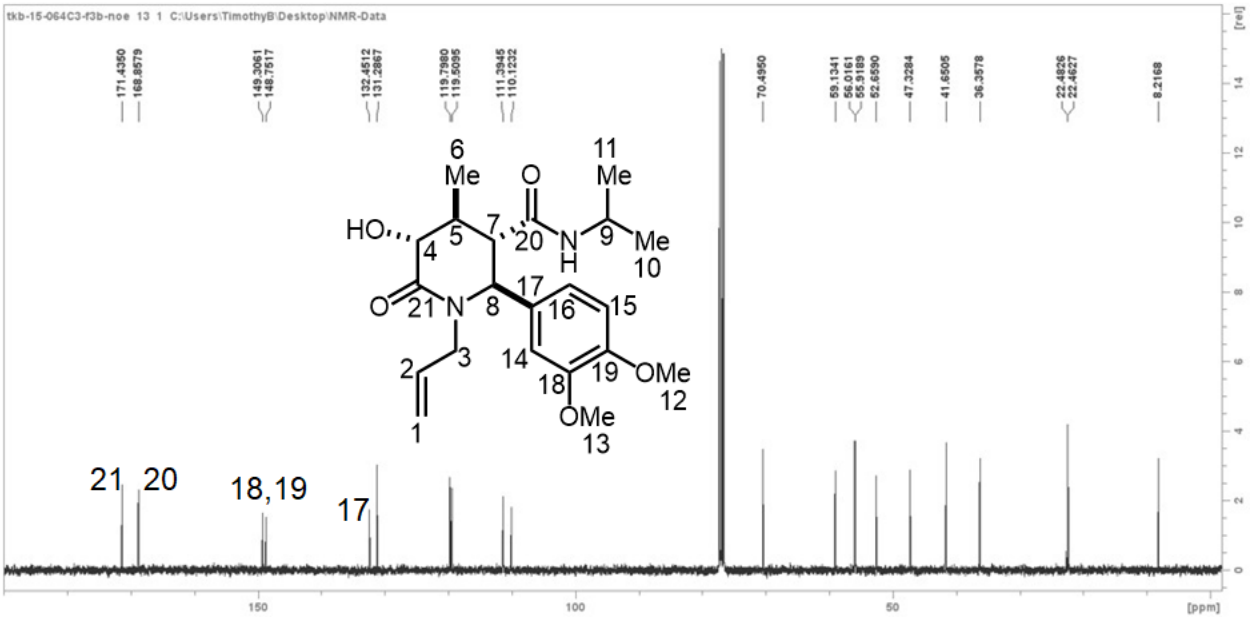
Compound 7m

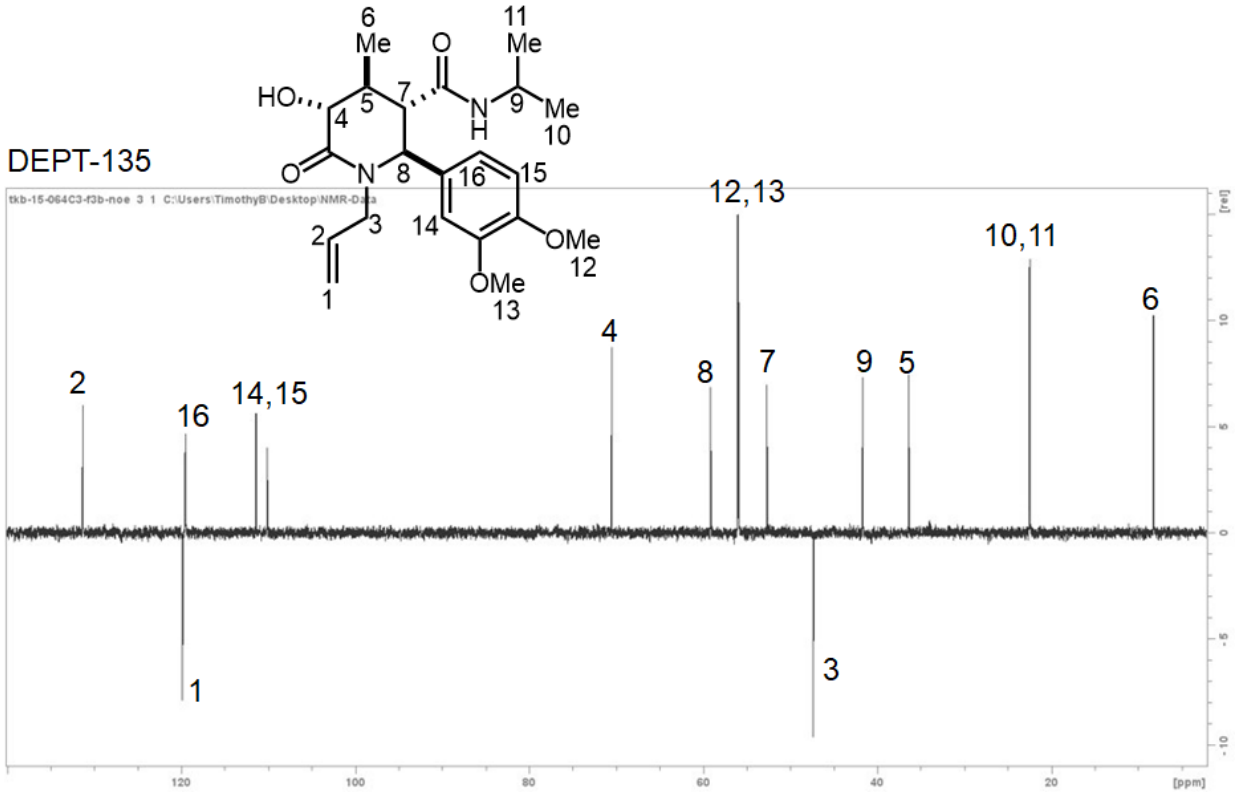
Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 30:70). Yellow oil. Yield = 355.3 mg, 91%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.82 (d, *J* = 8.2 Hz, 1H), 6.77 (dd, *J* = 8.3, 2.0 Hz, 1H), 6.70 (d, *J* = 2.0 Hz, 1H), 5.75 (dddd, *J* = 17.0, 10.0, 8.5, 4.8 Hz, 1H), 5.20 (t, *J* = 8.1 Hz, 2H), 5.12 – 5.02 (m, 1H), 5.01 (d, *J* = 10.0 Hz, 1H), 4.51 (ddt, *J* = 14.5, 4.8, 1.5 Hz, 1H), 4.30 (d, *J* = 4.7 Hz, 1H), 4.03 – 3.89 (m, 2H), 3.86 (s,s, 6H), 3.07 (dd, *J* = 14.5, 8.6 Hz, 1H), 2.65 (dd, *J* = 10.0, 3.6 Hz, 1H), 2.54 – 2.42 (m, 1H), 1.08 (d, *J* = 6.6 Hz, 3H), 0.99 (d,d, *J* = 6.7 Hz, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.44, 168.86, 149.31, 148.76, 132.45, 131.29, 119.80, 119.51, 111.40, 110.13, 70.50, 59.14, 56.02, 55.92, 52.66, 47.33, 41.65, 36.36, 22.49, 22.47, 8.22. **HRMS-EI^{+}** (*m/z*): calc for $C_{21}H_{30}N_2O_5$ $[M]^{+}$ 390.2155, found 390.2161.

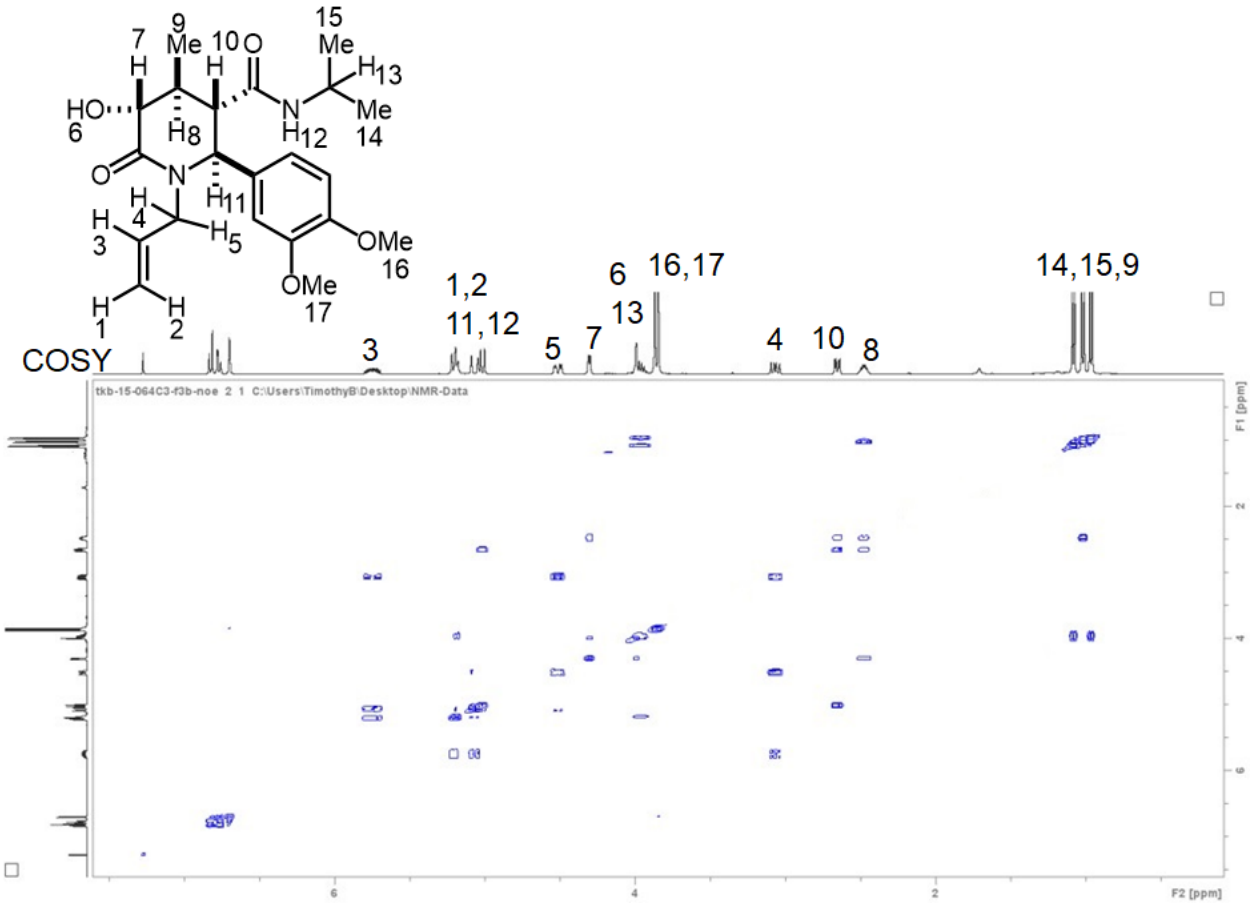


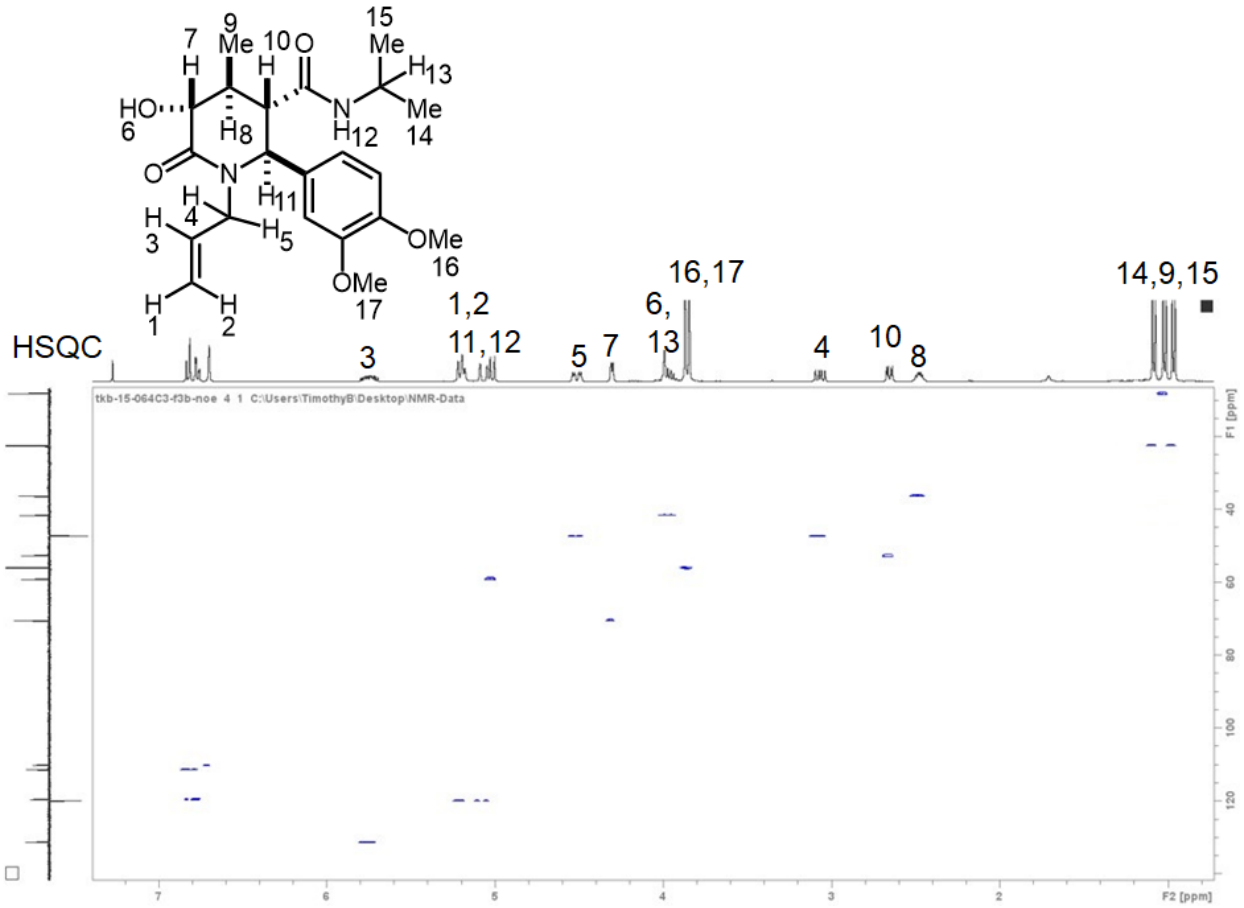
See the DEPT-135 for remaining assignments

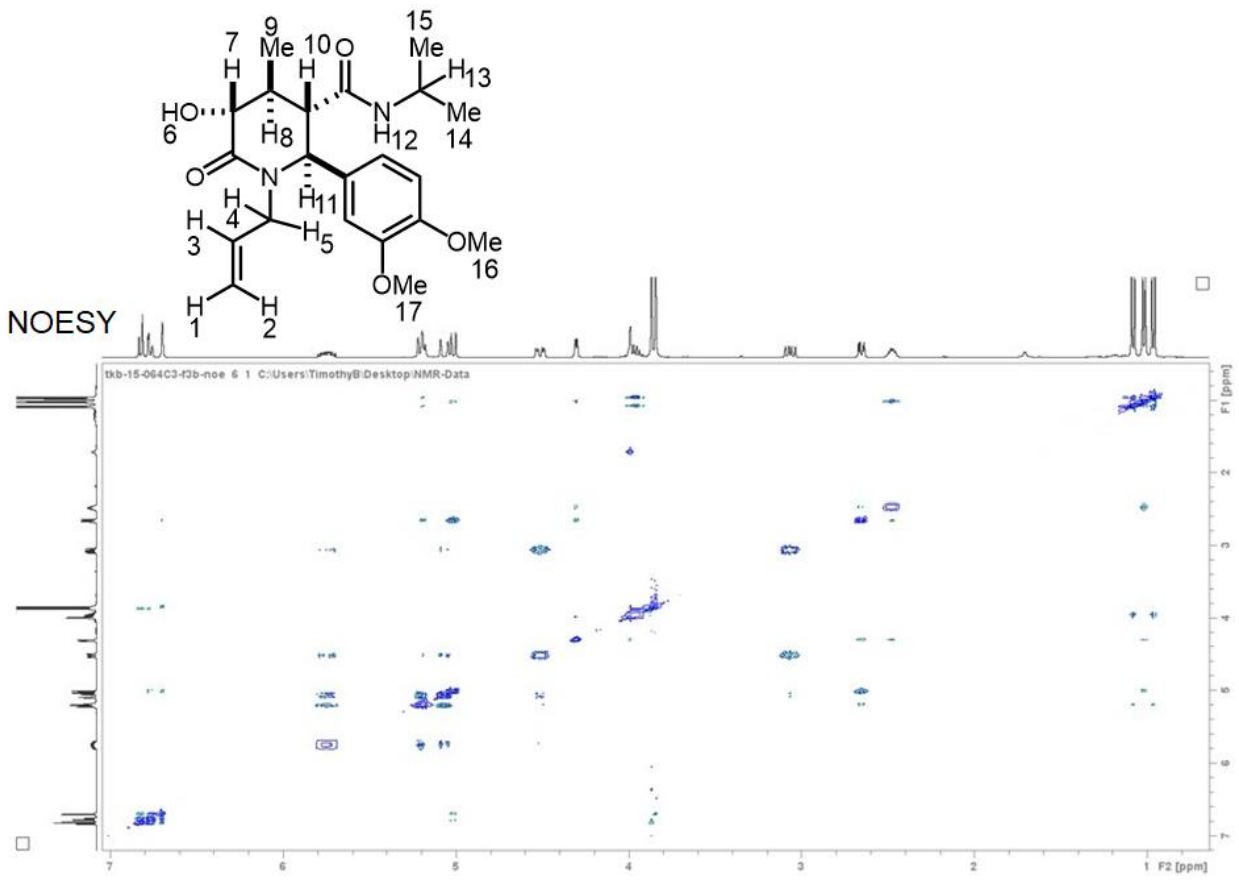
^{13}C NMR

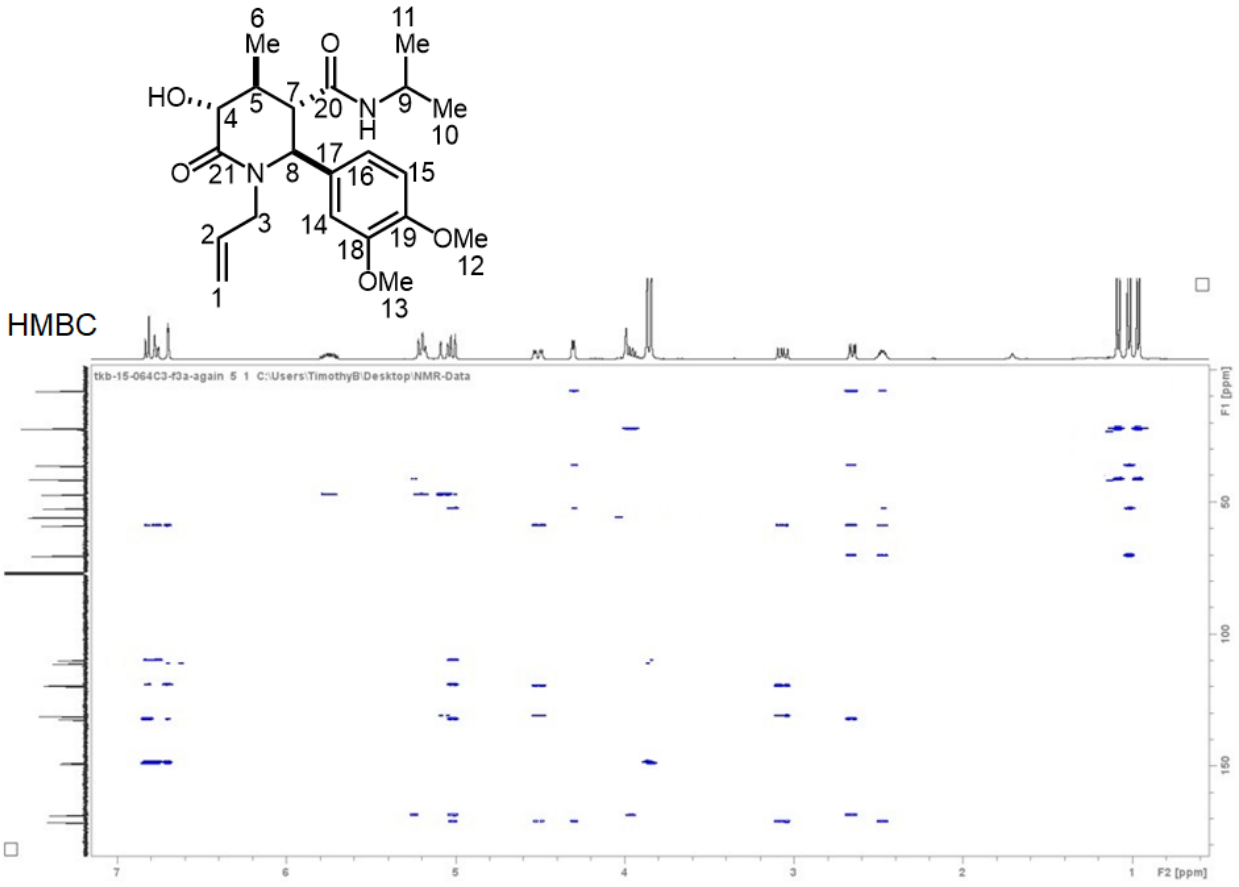



DEPT-135
6
Me
11
Me
HO
O
7
5
4
N
9
Me
10
H
O
N
8
16
15
2
3
14
OMe
12
OMe
13
1
12,13
10,11
6
4
2
16
14,15
8
7
9
5
1
3
120
100
80
60
40
20
[ppm]

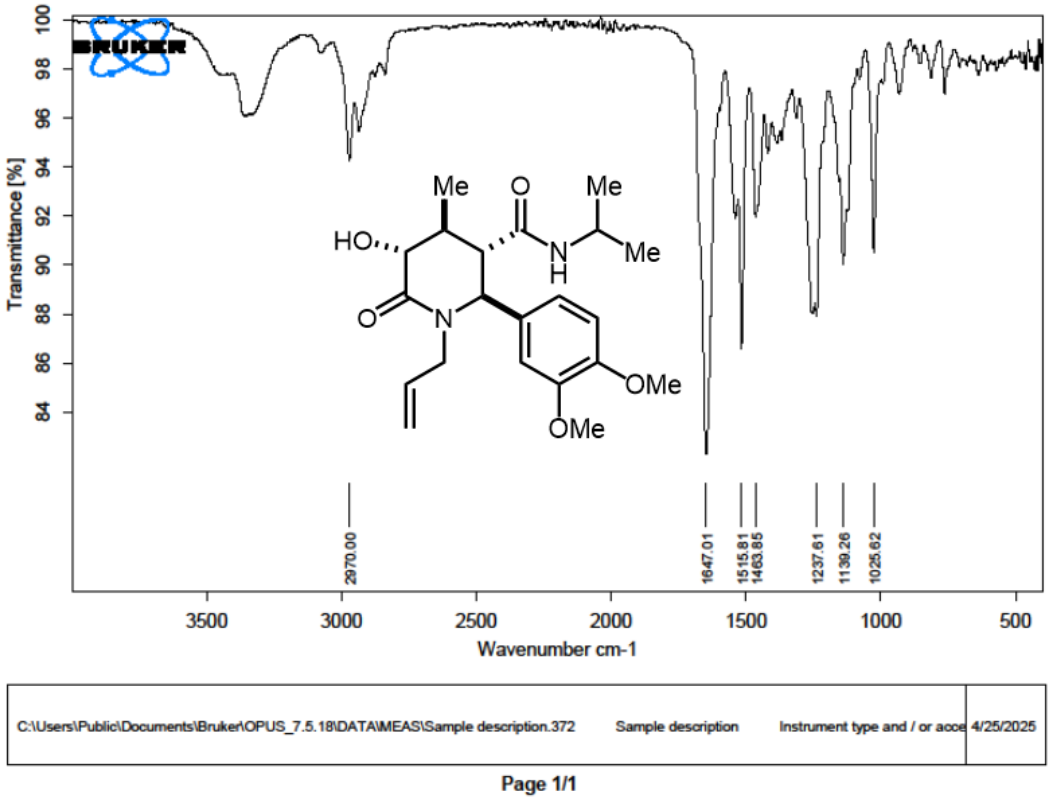

COSY
1,2
11,12
3
5
7
6
13
16,17
4
10
8
14,15,9
OMe
16
OMe
17
tkb-15-064C3-f3b-noe 2 1 C:\Users\TimothyB\Desktop\NMR-Data
F1 [ppm]
F2 [ppm]


HSQC
tkb-15-064C3-f3b-noe 4 1 C:\Users\TimothyB\Desktop\NMR-Data
F1 [ppm]
F2 [ppm]
14,9,15
16,17
6,
13
1,2
11,12
10
8
5
7
4
3
OMe
16
OMe
17


NOESY
7
H
9
Me
10
H
O
15
Me
H13
HO
6
H8
N
H12
Me
14
O
N
H
4
H11
H
3
H5
OMe
16
OMe
17
H
1
H
2
tkb-15-064C3-f3b-noe 6 1 C:\Users\TimothyB\Desktop\NMR-Data
F1 [ppm]
F2 [ppm]

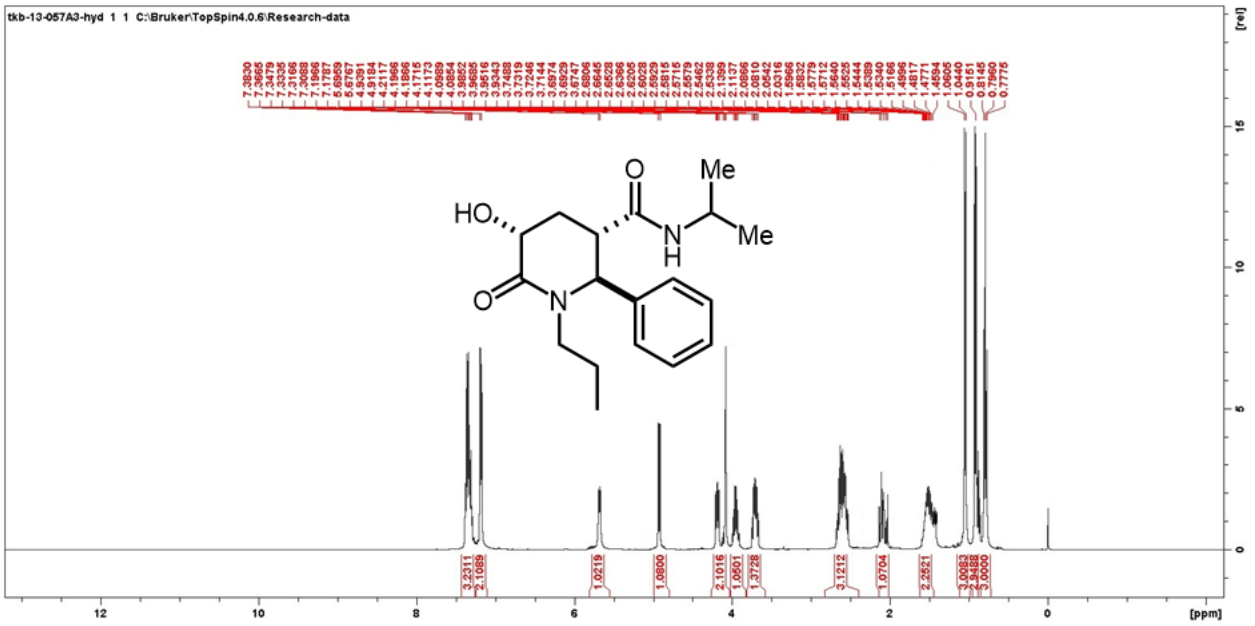

HMBC
tkb-15-064C3-f3a-again 5 1 C:\Users\TimothyB\Desktop\NMR-Data
F1 [ppm]
F2 [ppm]
HO
O
N
H
OMe
OMe

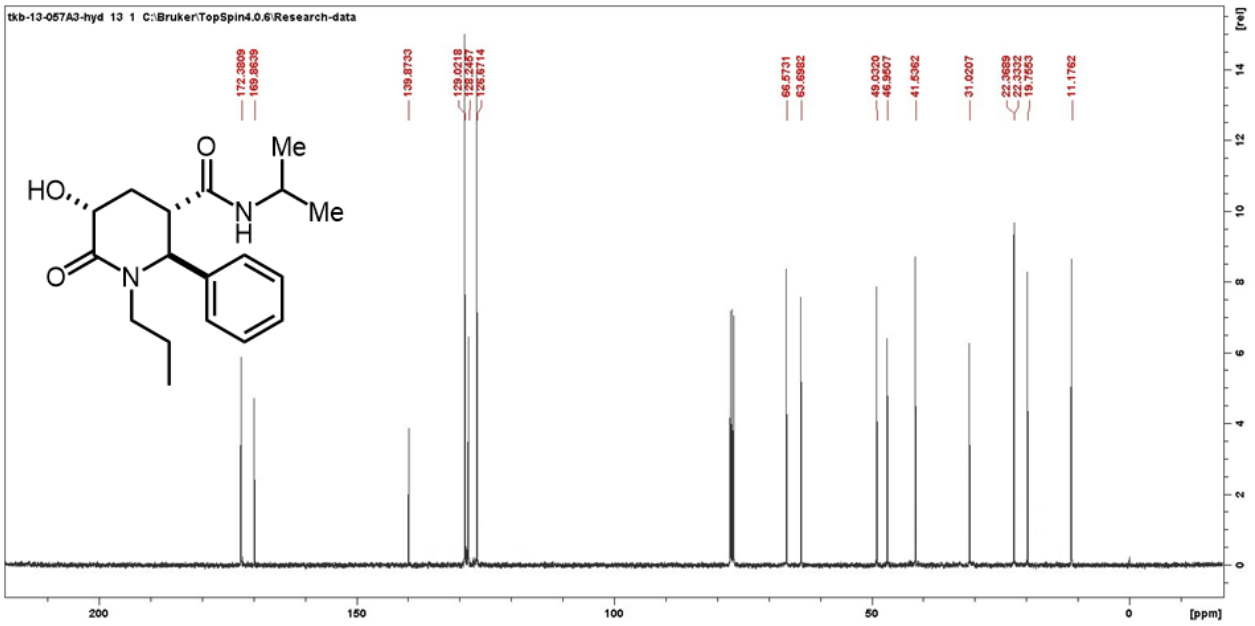
IR Spectrum

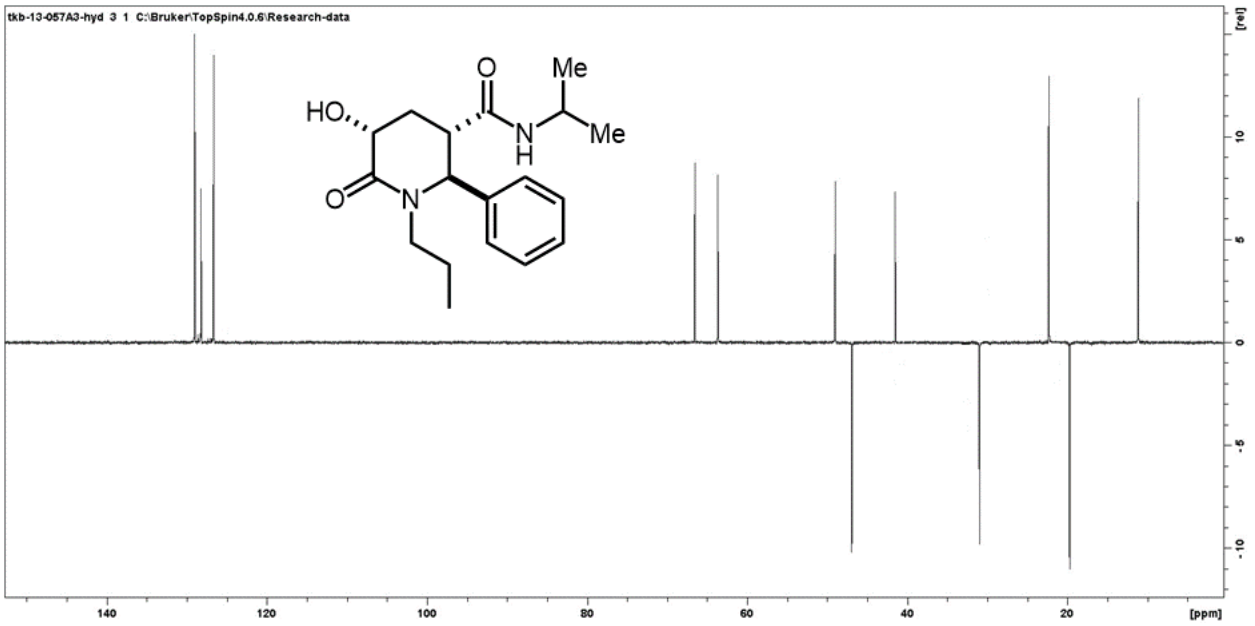


Compound 7n

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 50:50). Yellow oil. Yield = 286.6 mg, 90%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.41 – 7.22 (m, 3H), 7.19 (dd, J = 6.9, 1.7 Hz, 2H), 5.69 (d, J = 7.9 Hz, 1H), 4.93 (d, J = 8.4 Hz, 1H), 4.19 (dd, J = 10.1, 6.0 Hz, 1H), 4.09 (s, 1H), 4.04 – 3.88 (m, 1H), 3.71 (dq, J = 9.4, 6.9 Hz, 1H), 2.72 – 2.51 (m, 3H), 2.28 (ddd, J = 13.3, 6.0, 3.8 Hz, 1H), 2.16 – 2.01 (m, 1H), 2.01 – 1.87 (m, 0H), 1.62 – 1.44 (m, 1H), 1.05 (d, J = 6.6 Hz, 3H), 0.95 – 0.84 (m, 3H), 0.80 (t, J = 7.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.38, 169.87, 139.88, 129.03, 128.25, 126.67, 66.58, 63.70, 49.04, 46.95, 41.54, 31.02, 22.37, 22.34, 19.76, 11.18. **HRMS-EI^{+}** (*m/z*): calc for $C_{18}H_{26}N_2O_3$ $[M]^{+}$ 318.1943, found 318.1948.

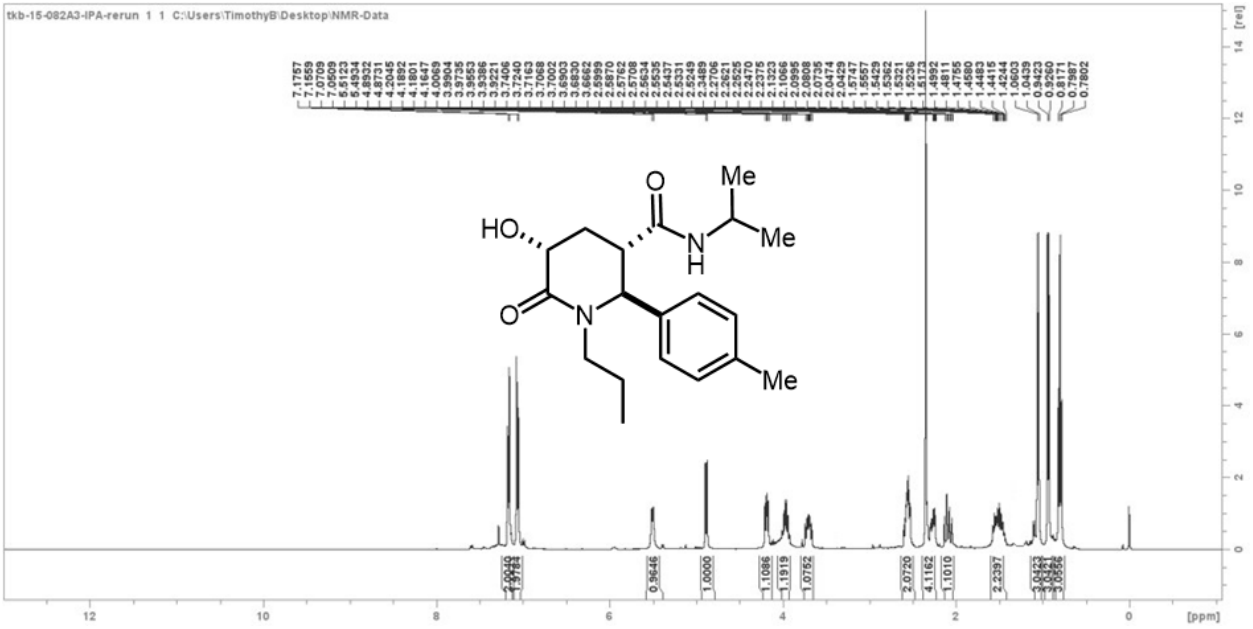

tkb-13-057A3-hyd 1 1 C:\Bruker\TopSpin4.0.6\Research-data
HO
O
N
O
N
H
Me
Me
[ppm]
[rel]

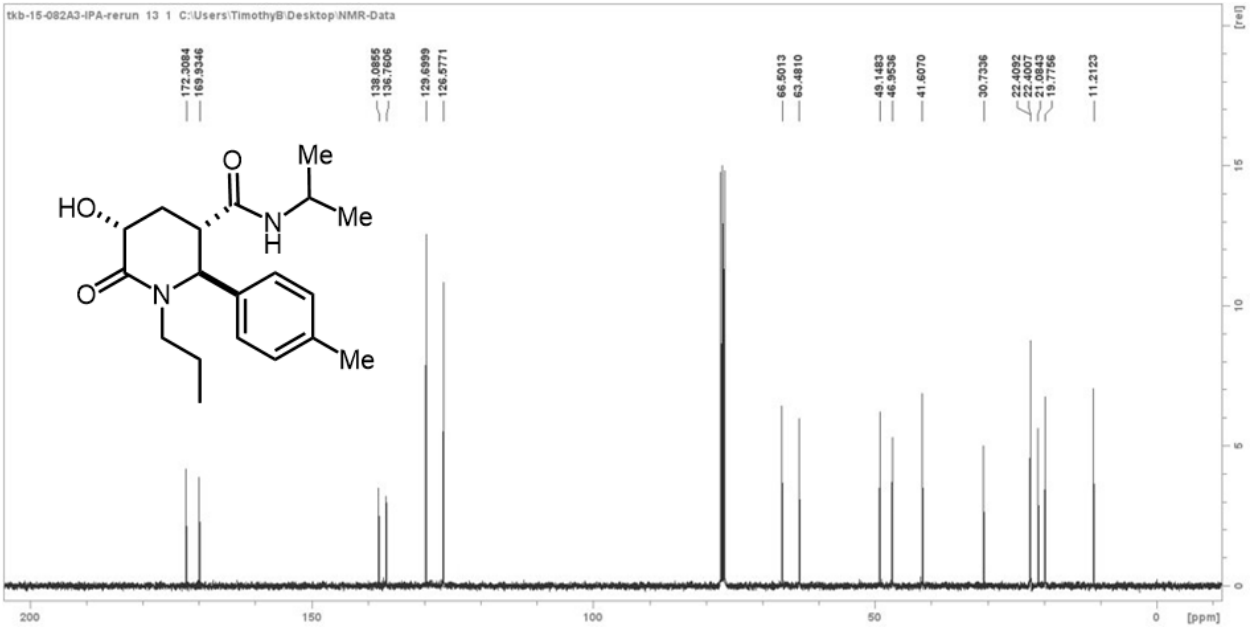

tkb-13-057A3-hyd 13 1 C:\Bruker\TopSpin4.0.6\Research-data
172.3809
169.8639
139.8733
129.0218
128.6714
66.5731
63.6982
49.0320
46.9507
41.5362
31.0207
22.3689
22.3332
19.7553
11.1762
HO
O
N
O
N
H
Me
Me
[ppm]
[rel]

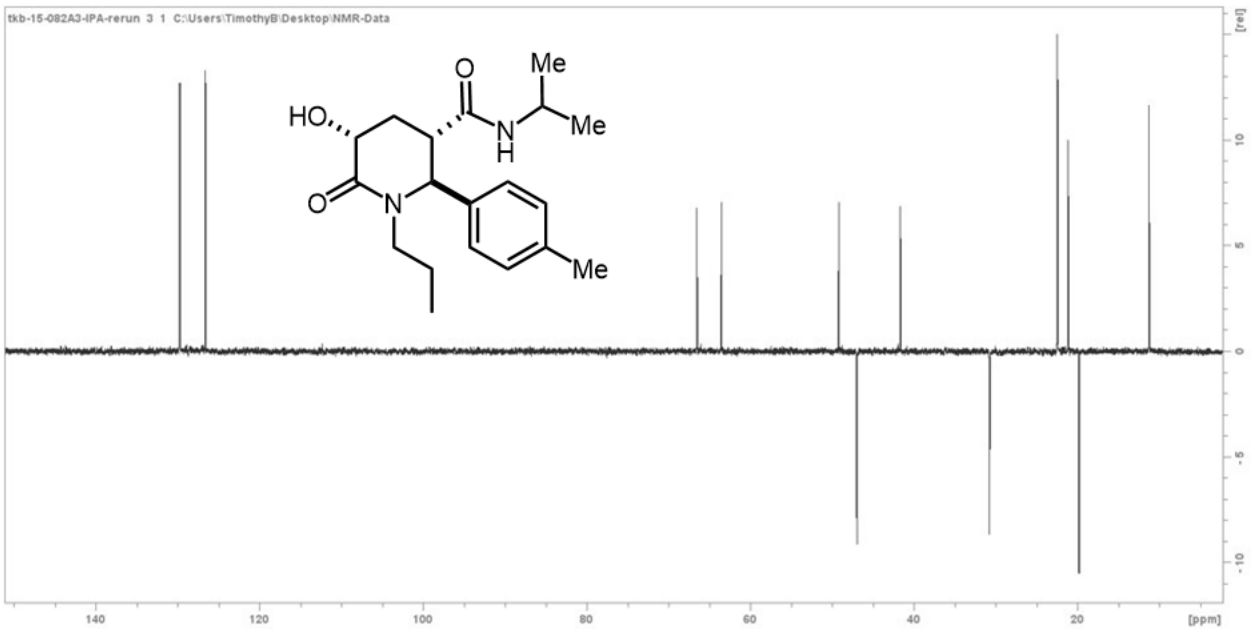


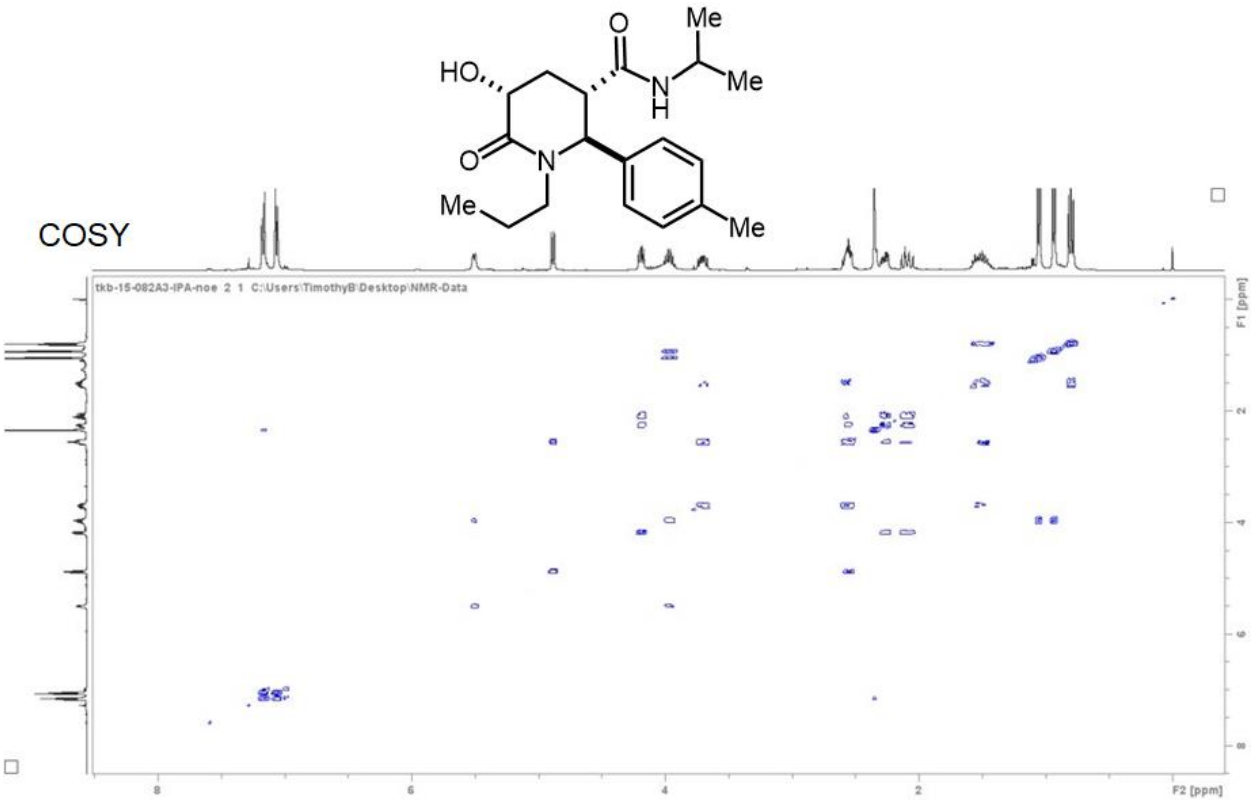
Compound 7o

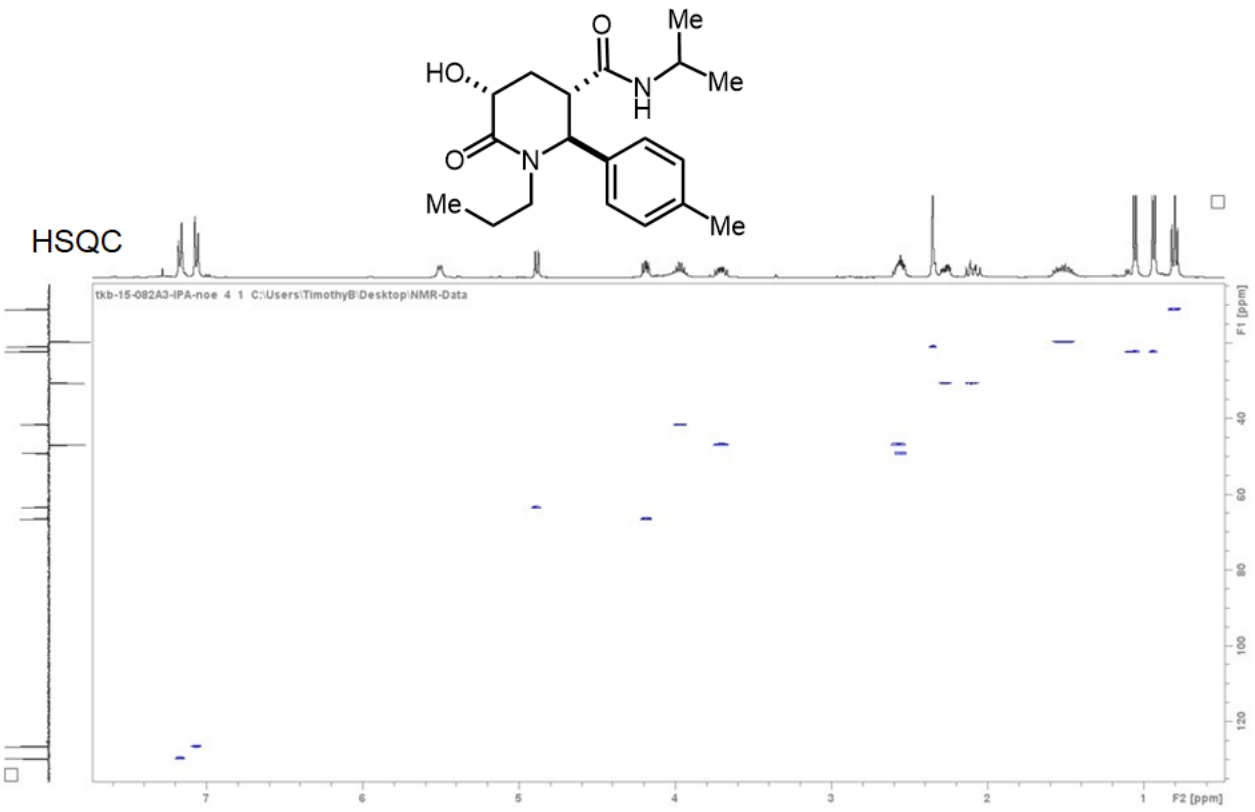
Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 50:50). Pale-yellow oil. Yield = 312.5 mg, 94%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.17 (d, *J* = 7.8 Hz, 2H), 7.06 (d, *J* = 7.8 Hz, 2H), 5.50 (d, *J* = 7.9 Hz, 1H), 4.88 (d, *J* = 8.1 Hz, 1H), 4.18 (dd, *J* = 9.9, 6.1 Hz, 1H), 4.05 – 3.88 (m, 1H), 3.80 – 3.64 (m, 1H), 2.62 – 2.50 (m, 2H), 2.35 (s, 3H), 2.27 (ddd, *J* = 13.4, 6.2, 3.8 Hz, 1H), 2.15 – 2.00 (m, 1H), 1.61 – 1.40 (m, 2H), 1.05 (d, *J* = 6.6 Hz, 3H), 0.93 (d, *J* = 6.5 Hz, 3H), 0.80 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.31, 169.94, 138.09, 136.76, 129.70, 126.58, 66.50, 63.48, 49.15, 46.96, 41.61, 30.74, 22.42, 22.40, 21.09, 19.78, 11.22. **HRMS-EI$^+$** (*m/z*): calc for $C_{19}H_{28}N_2O_3$ $[M]^+$ 332.2100, found 332.2106.

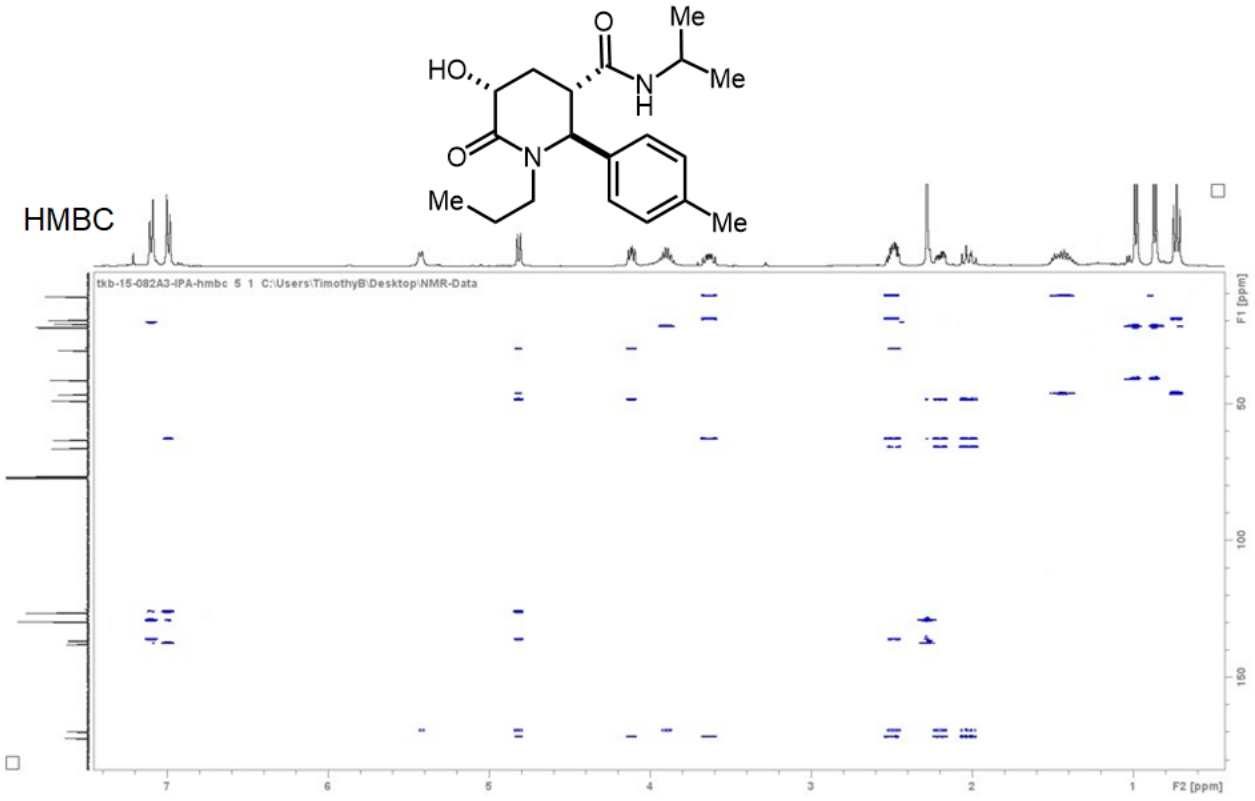

tkb-15-082A3-IPA-rerun 1 1 C:\Users\TimothyB\Desktop\NMR-Data
HO
O
N
H
Me
Me
O
N
Me
[ppm]


tkb-15-082A3-IPA-rerun 13 1 C:\Users\TimothyB\Desktop\NMR-Data
172.3084
169.9346
138.0855
136.7606
129.6999
126.5771
66.5013
63.4810
49.1483
46.9536
41.6070
30.7336
22.4092
21.4027
21.0843
19.7766
11.2123
HO
O
N
H
Me
Me
O
N
Me
[ppm]

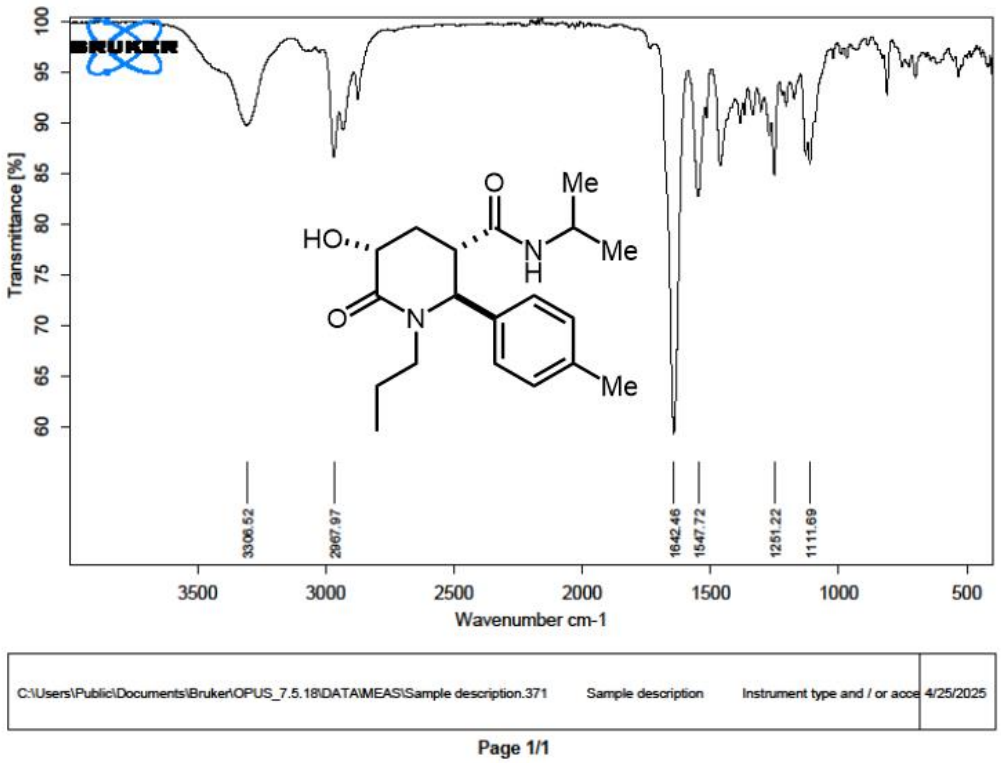

tkb-15-082A3-IPA-rerun 3 1 C:\Users\TimothyB\Desktop\NMR-Data
HO
O
N
H
Me
Me
O
N
Me
140
120
100
80
60
40
20
[ppm]


COSY
HO
O
N
H
Me
Me
O
N
Me
Me
tkb-15-082A3-IPA-noe 2 1 C:\Users\TimothyB\Desktop\NMR-Data
F1 [ppm]
F2 [ppm]


HSQC
tkb-15-082A3-IPA-noe 4 1 C:\Users\TimothyB\Desktop\NMR-Data
F1 [ppm]
F2 [ppm]

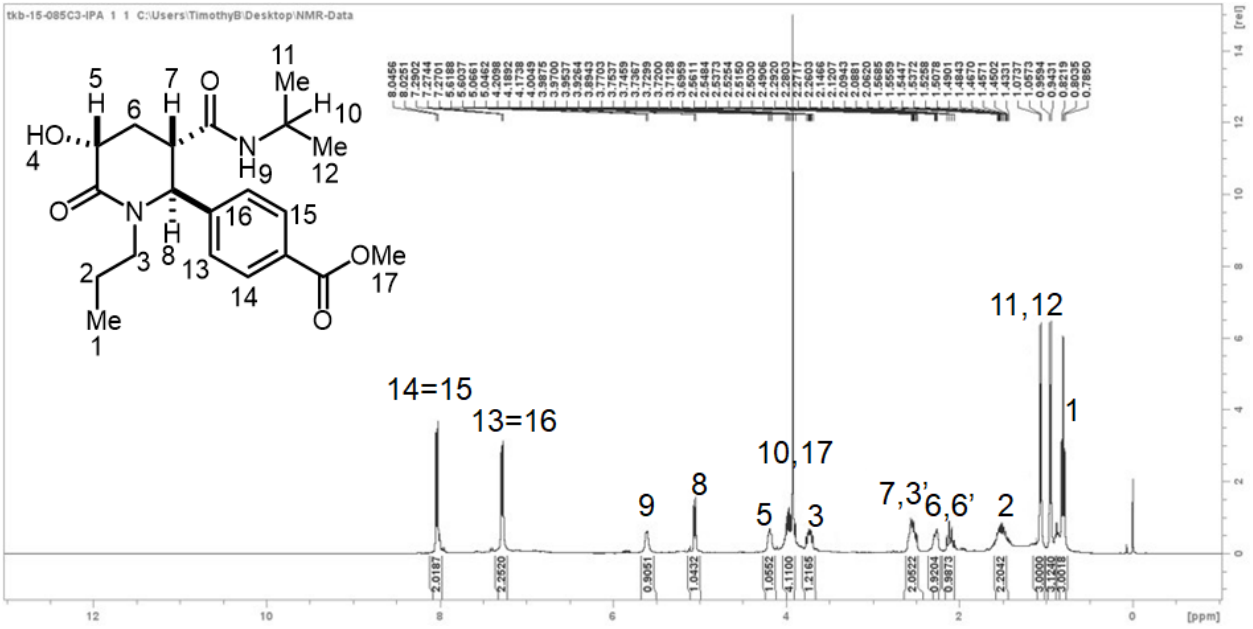

HMBC
tkb-15-082A3-IPA-hmbc 5 1 C:\Users\TimothyB\Desktop\NMR-Data
F1 [ppm]
F2 [ppm]

IR Spectrum



Compound 7p

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 50:50). Greenish-yellow oil. Yield = 331.2 mg, 88%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.02 (d, *J* = 8.0 Hz, 2H), 7.28 (d, *J* = 8.0 Hz, 2H), 5.64 – 5.57 (m, 1H), 5.06 (d, *J* = 8.0 Hz, 1H), 4.19 (dd, *J* = 9.9, 6.1 Hz, 1H), 4.04 – 3.94 (m, 1H), 3.93 (s, 3H), 3.73 (ddd, *J* = 13.5, 9.7, 6.6 Hz, 1H), 2.54 (dtd, *J* = 18.7, 10.0, 4.3 Hz, 2H), 2.36 – 2.22 (m, 1H), 2.10 (dt, *J* = 13.2, 10.3 Hz, 1H), 1.61 – 1.41 (m, 2H), 1.07 (d, *J* = 6.6 Hz, 3H), 0.95 (d, *J* = 6.6 Hz, 3H), 0.79 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.43, 169.48, 166.46, 145.06, 130.37, 126.74, 66.43, 63.33, 52.30, 48.80, 47.28, 41.79, 30.97, 29.70, 22.49, 22.35, 19.78, 11.19. **HRMS-EI$^+$** (*m/z*): calc for $C_{20}H_{28}N_2O_5$ $[M]^+$ 376.1998, found 376.1994.


tkb-15-085C3-IPA 1 1 C:\Users\TimothyB\Desktop\NMR-Data
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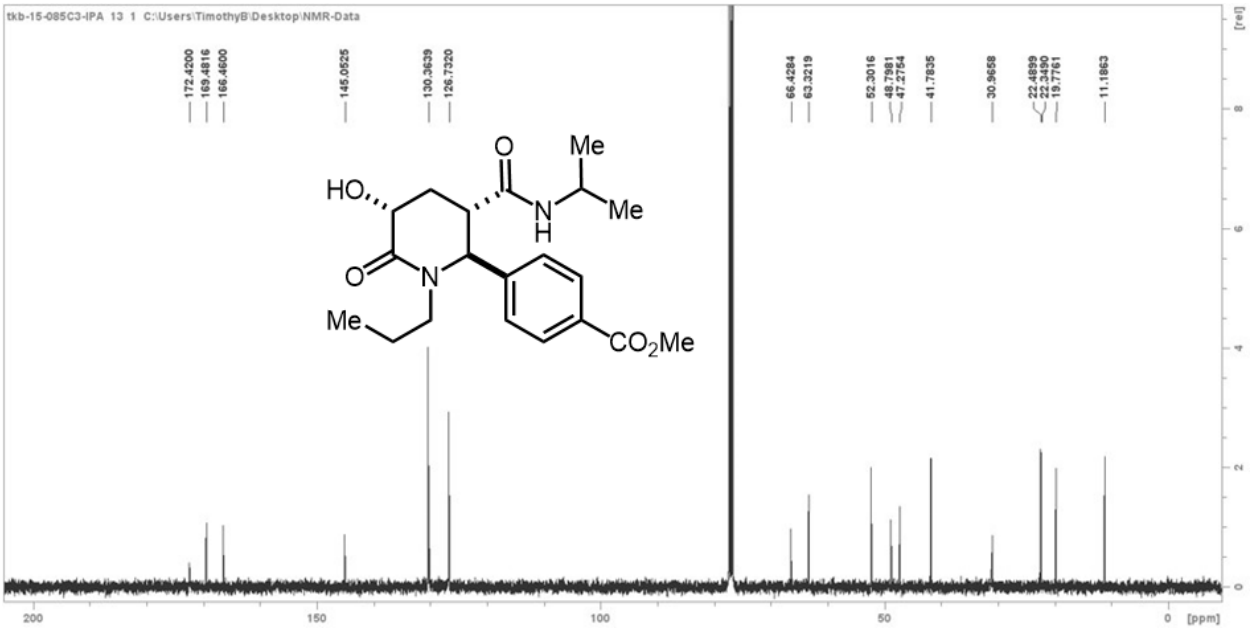
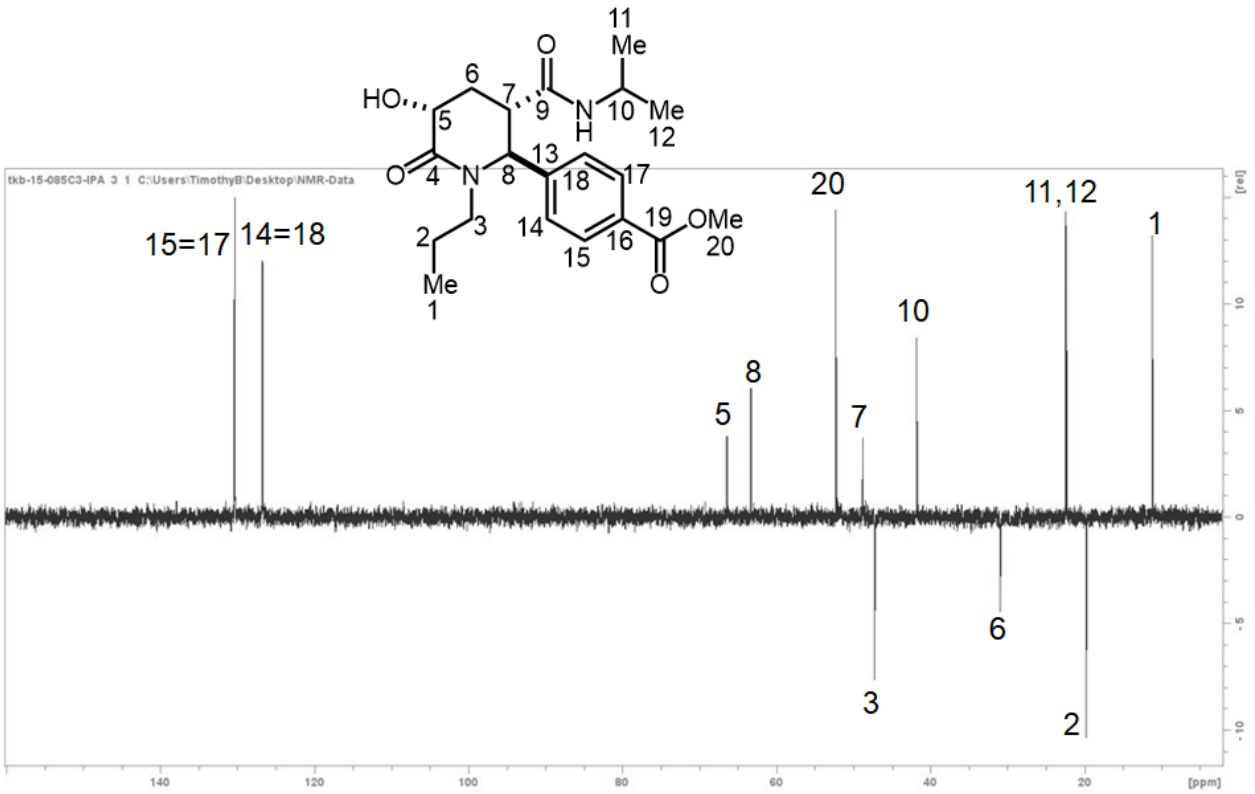
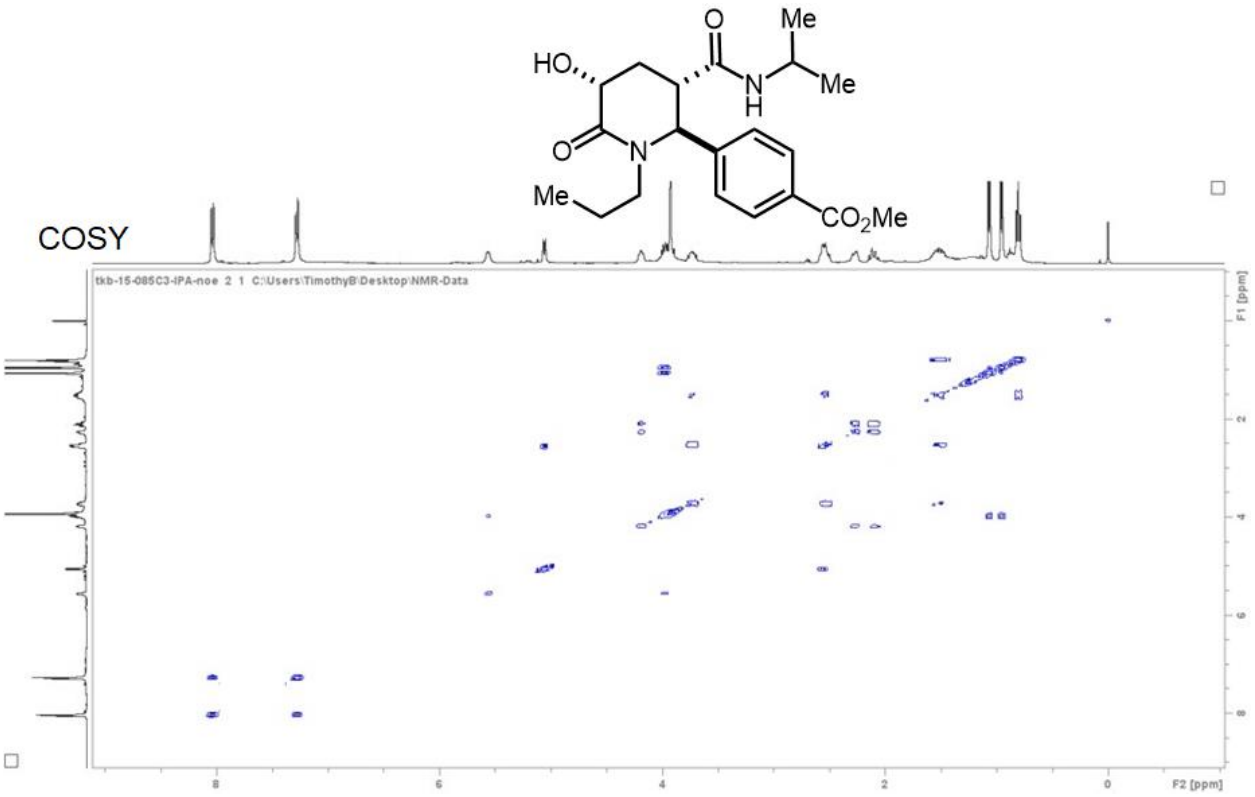
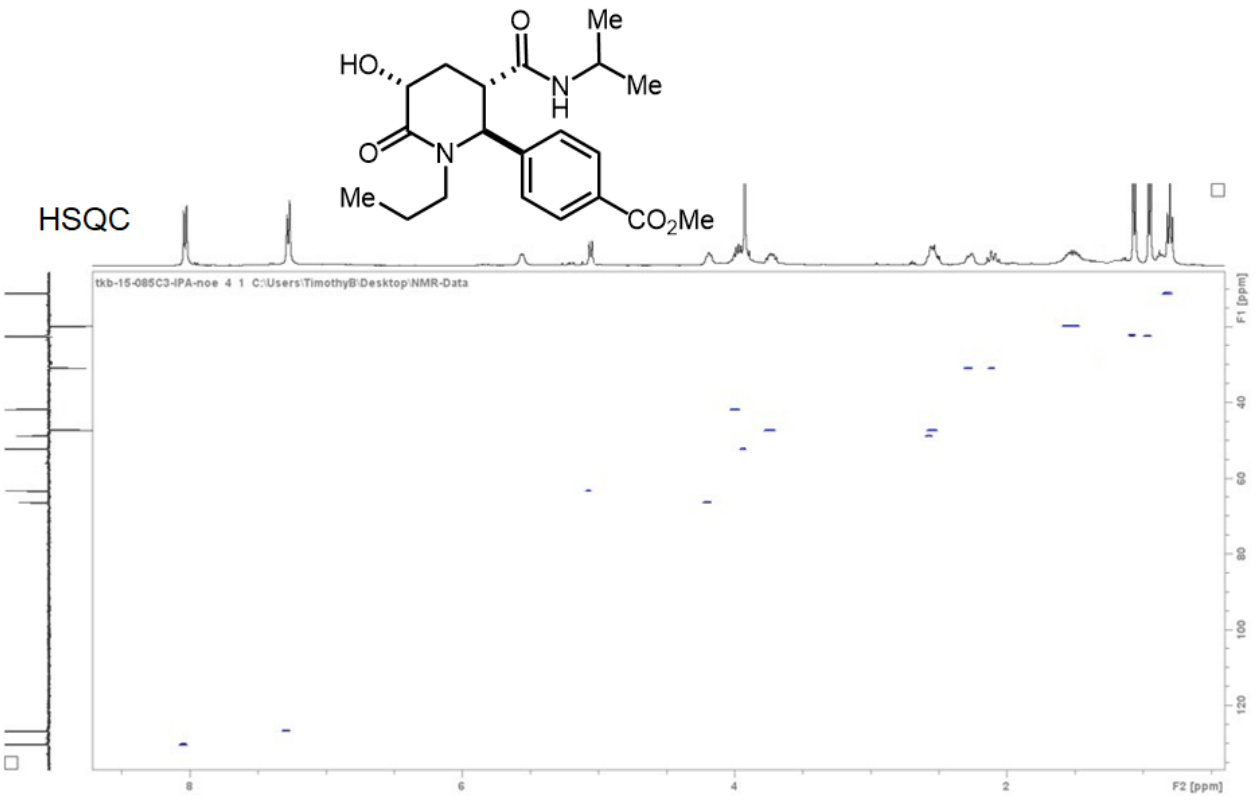
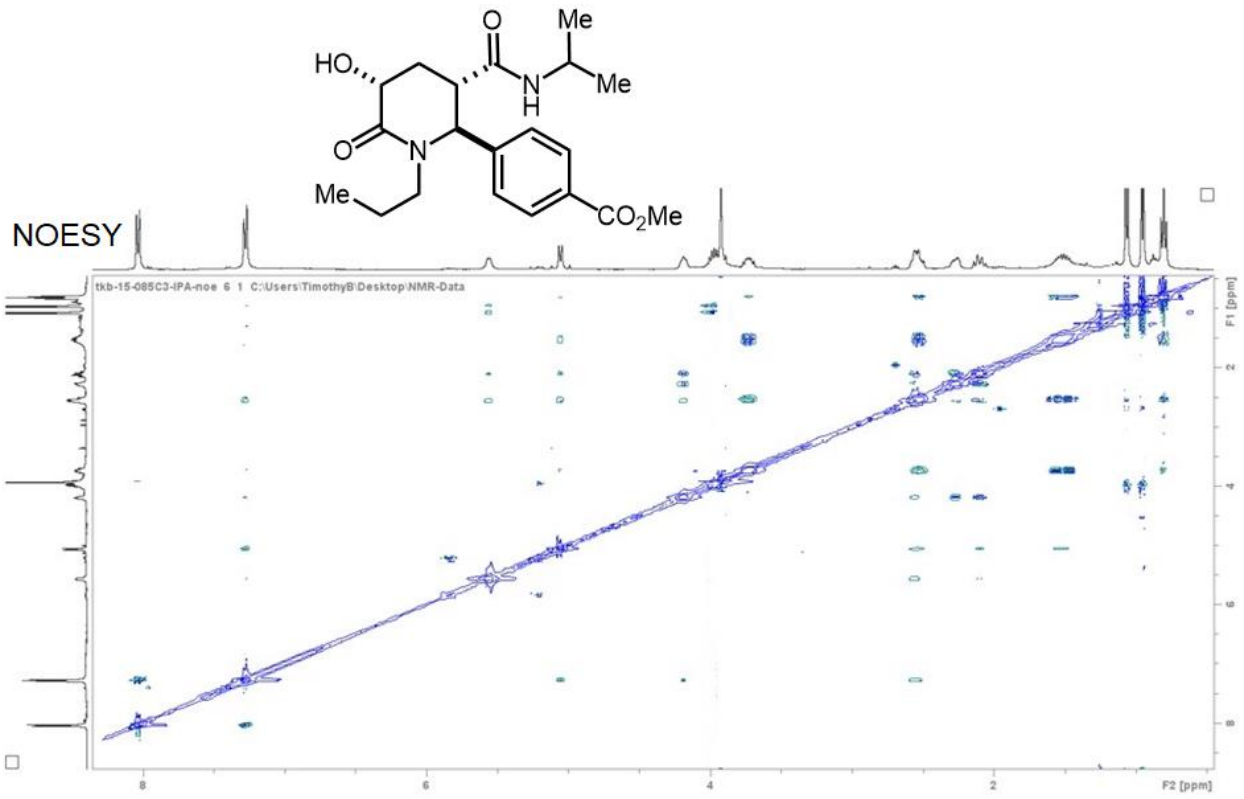
13=16
9
8
10,17
5
3
7,3'
6,6'
2
11,12
1
[ppm]


tkb-15-085C3-IPA 13 1 C:\Users\TimothyB\Desktop\NMR-Data
172.4200
169.4816
166.4600
145.0525
130.3639
126.7320
66.4284
63.3219
52.3016
48.7981
47.2764
41.7835
30.9658
22.4899
22.3490
19.7761
11.1863
[ppm]

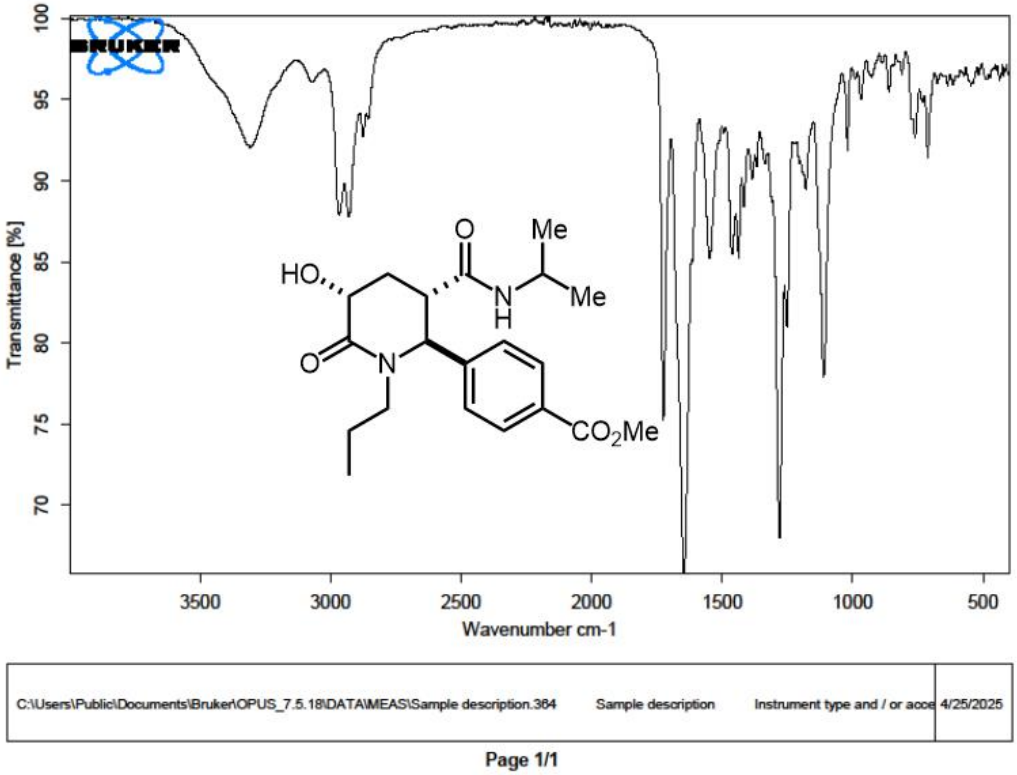

11
Me
O
6
HO
7
5
9
N
10
Me
H
12
O
4
N
8
13
18
17
2
3
14
19
OMe
20
16
15
Me
1
O
tkb-15-085C3-IPA 3 1 C:\Users\TimothyB\Desktop\NMR-Data
15=17
14=18
20
11,12
1
10
8
5
7
6
3
2
[rel]
[ppm]


O
Me
HO
N
H
Me
O
N
Me
CO2Me
COSY
tkb-15-085C3-IPA-noe 2 1 C:\Users\TimothyB\Desktop\NMR-Data
F1 [ppm]
F2 [ppm]


HSQC
HO
O
N
H
Me
Me
O
N
Me
CO2Me
tkb-15-085C3-IPA-noe 4 1 C:\Users\TimothyB\Desktop\NMR-Data
F1 [ppm]
F2 [ppm]

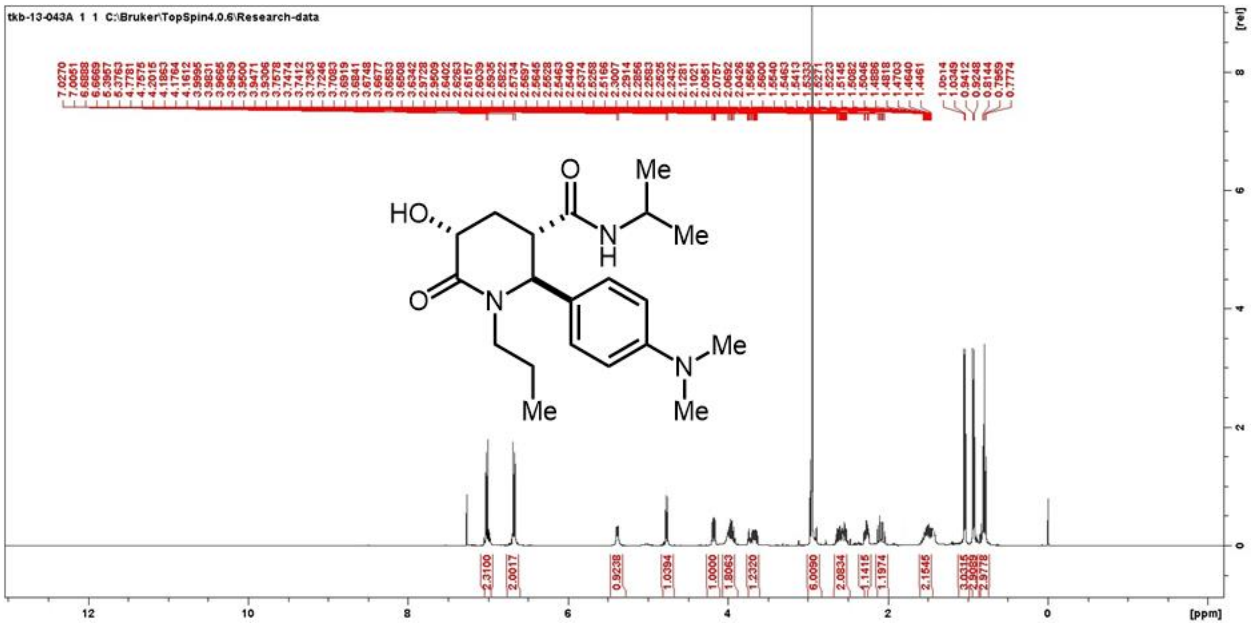

NOESY
HO
O
N
H
Me
Me
O
N
Me
CO2Me
tkb-15-085C3-IPA-noe 6 1 C:\Users\TimothyB\Desktop\NMR-Data
F1 [ppm]
F2 [ppm]

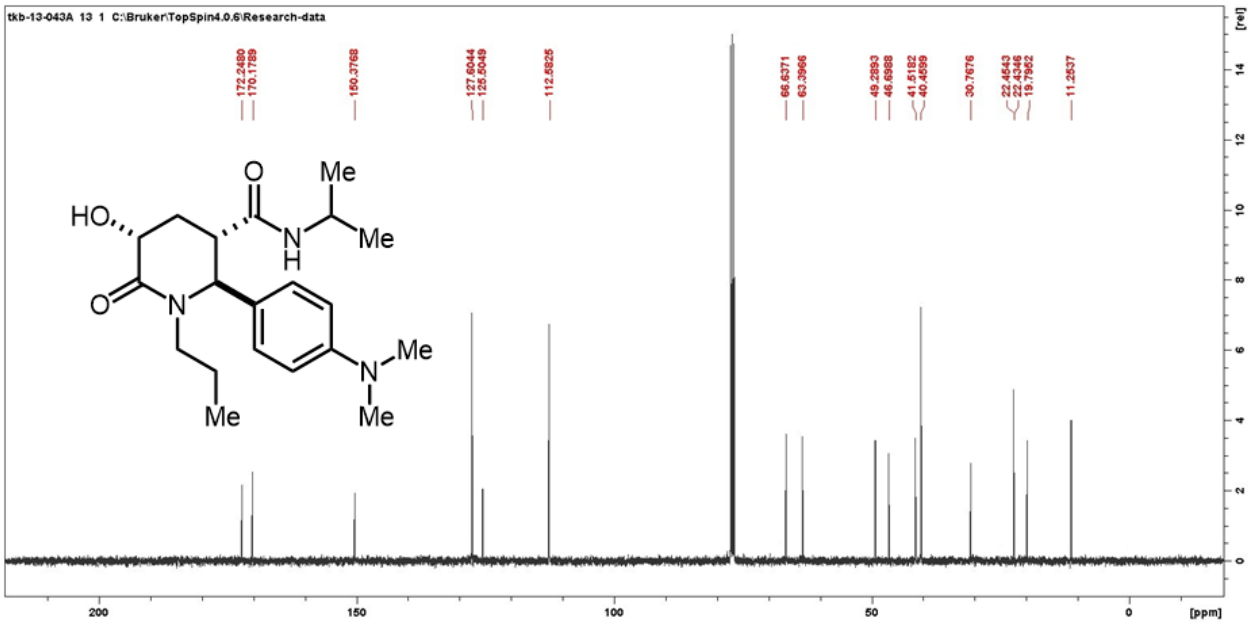
IR Spectrum

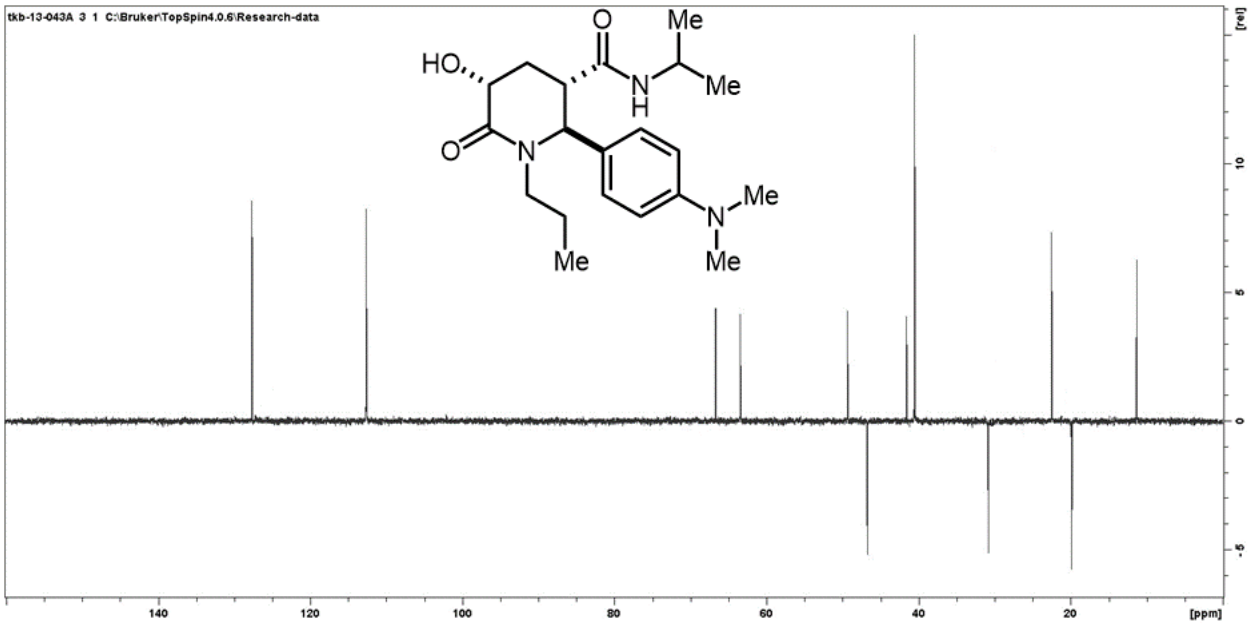


Compound 7q

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 20:80). Pale-yellow oil. Yield = 339.8 mg, 94%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.03 (d, *J* = 7.3 Hz, 2H), 6.69 (d, *J* = 7.3 Hz, 2H), 5.39 (d, *J* = 7.9 Hz, 1H), 4.77 (d, *J* = 8.2 Hz, 1H), 4.18 (dd, *J* = 10.1, 6.1 Hz, 1H), 4.04 – 3.88 (m, 2H), 3.78 – 3.61 (m, 1H), 2.95 (s,s, 6H), 2.68 – 2.45 (m, 2H), 2.34 – 2.22 (m, 1H), 2.27 (s, 1H), 2.09 (dt, *J* = 13.3, 10.5 Hz, 1H), 1.60 – 1.39 (m, 2H), 1.04 (d, *J* = 6.5 Hz, 3H), 0.93 (d, *J* = 6.5 Hz, 3H), 0.80 (q, *J* = 7.0 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.25, 170.18, 150.38, 127.61, 126.92, 125.51, 112.58, 66.64, 63.40, 49.29, 46.70, 41.52, 40.46, 30.33, 22.46, 22.44, 19.80, 11.26. **HRMS-EI$^+$** (*m/z*): calc for $C_{20}H_{31}N_3O_3$ $[M]^+$ 361.2365, found 361.2372.

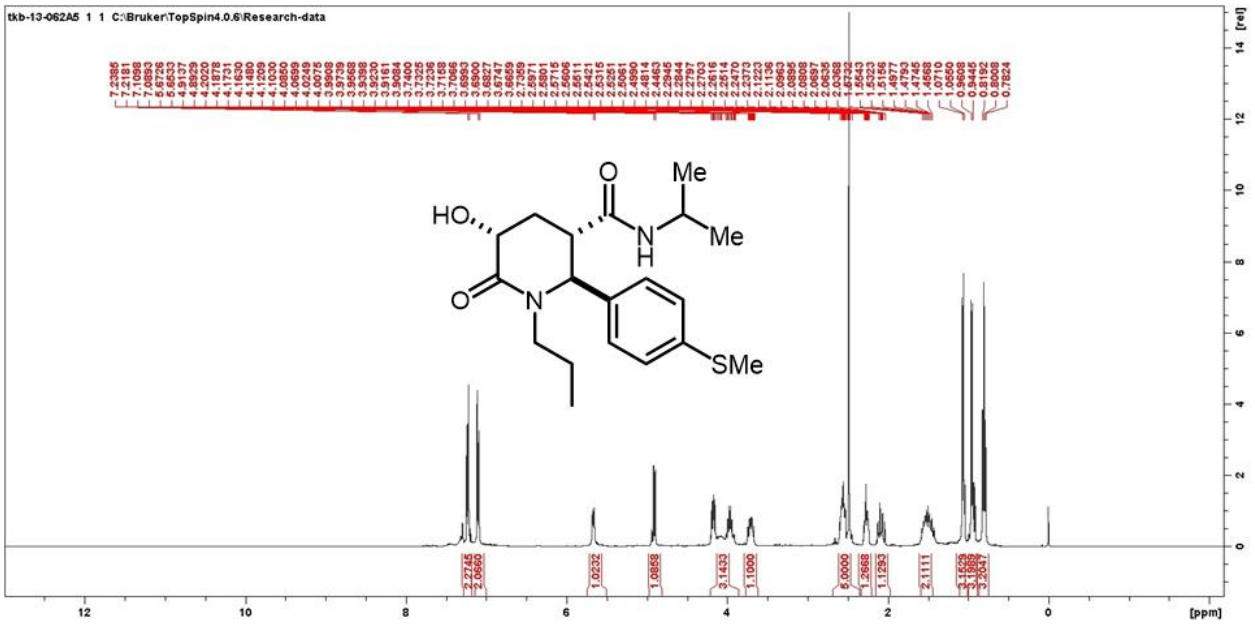

tkb-13-043A 1 1 C:\Bruker\TopSpin4.0.6\Research-data
HO
O
N
H
Me
Me
O
N
Me
N
Me
Me
[rel]
[ppm]


tkb-13-043A 13 1 C:\Bruker\TopSpin4.0.6\Research-data
172.2480
170.1789
150.3768
127.6044
125.5049
112.5825
66.6371
63.3966
49.2893
46.6868
41.5182
40.4599
30.7676
22.4643
22.4346
19.7952
11.2537
HO
O
N
H
Me
Me
O
N
Me
N
Me
Me
[rel]
[ppm]



Compound 7r

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 50:50). Amorphous solid. Yield = 331.7 mg, 91%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.21 (d, *J* = 7.7 Hz, 2H), 7.10 (d, *J* = 7.7 Hz, 2H), 5.66 (d, *J* = 7.9 Hz, 1H), 4.90 (d, *J* = 8.3 Hz, 1H), 4.17 (dd, *J* = 10.1, 5.9 Hz, 1H), 3.96 (dp, *J* = 12.6, 6.4 Hz, 1H), 3.71 (dddd, *J* = 13.3, 9.7, 6.5, 3.3 Hz, 1H), 2.56 (ddt, *J* = 14.3, 6.6, 4.1 Hz, 2H), 2.48 (s, 3H), 2.32 – 2.21 (m, 1H), 2.16 – 2.01 (m, 1H), 1.62 – 1.39 (m, 2H), 1.06 (d, *J* = 6.6 Hz, 3H), 0.94 (dd, *J* = 12.5, 6.6 Hz, 3H), 0.80 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.34, 169.81, 138.87, 136.53, 129.05, 127.16, 126.79, 126.68, 66.53, 63.22, 49.00, 46.96, 41.64, 30.98, 22.44, 22.36, 19.77, 15.57, 11.20. **HRMS-EI$^+$** (*m/z*): calc for $C_{19}H_{28}N_2O_3S$ $[M]^+$ 364.1821, found 364.1825.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O
N
H
Me
Me
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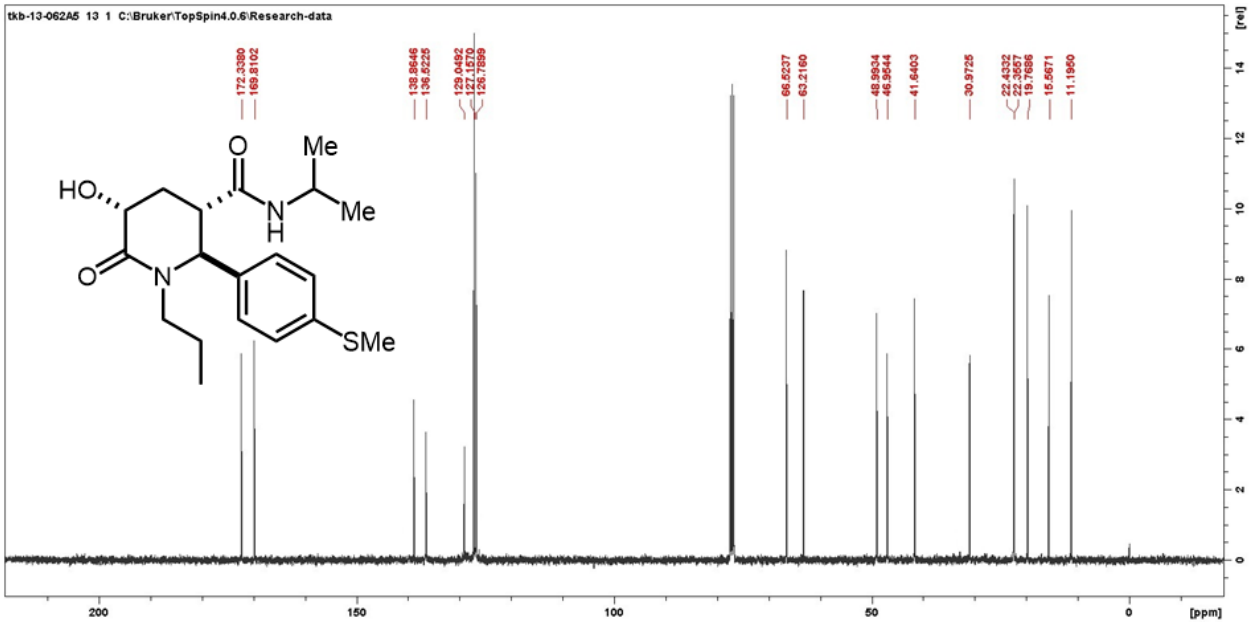
N
SMe
[ppm]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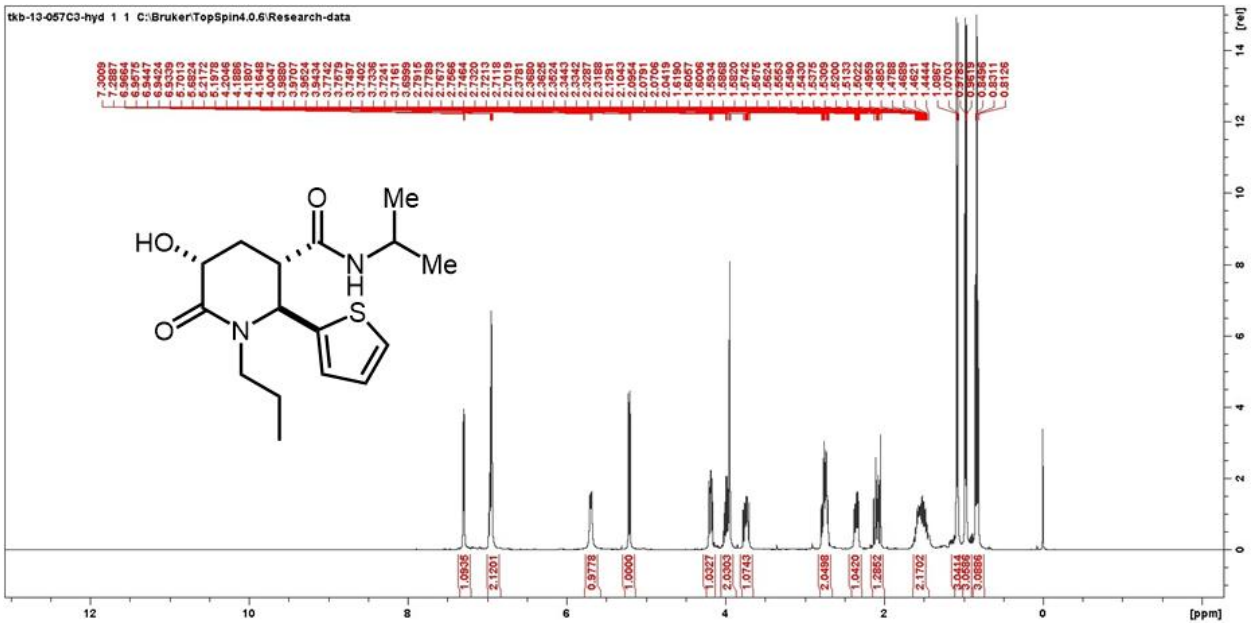
41.6403
30.9725
22.4332
22.3657
19.7686
15.5671
11.1950
HO
O
N
H
Me
Me
O
N
SMe
[ppm]

Compound 7s

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 50:50). Pale-yellow oil. Yield = 292.0 mg, 90%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.29 (dd, *J* = 4.9, 1.4 Hz, 1H), 7.00 – 6.90 (m, 2H), 5.69 (d, *J* = 7.9 Hz, 1H), 5.21 (d, *J* = 7.7 Hz, 1H), 4.23 – 4.09 (m, 1H), 4.06 – 3.93 (m, 1H), 3.94 (s, 1H), 3.74 (ddd, *J* = 13.5, 9.7, 6.5 Hz, 1H), 2.76 (dq, *J* = 7.8, 4.4 Hz, 1H), 2.75 – 2.60 (m, 1H), 2.35 (ddd, *J* = 13.5, 6.3, 4.0 Hz, 1H), 2.15 – 2.02 (m, 1H), 1.64 – 1.42 (m, 2H), 1.08 (d, *J* = 6.6 Hz, 3H), 0.97 (d, *J* = 6.6 Hz, 3H), 0.83 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.87, 169.56, 143.75, 127.02, 126.44, 125.68, 66.30, 59.55, 49.57, 47.15, 41.68, 30.69, 22.46, 22.38, 19.96, 11.22. **HRMS-EI$^+$** (*m/z*): calc for $C_{16}H_{24}N_2O_3S$ $[M]^+$ 324.1508, found 324.1512.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N
H
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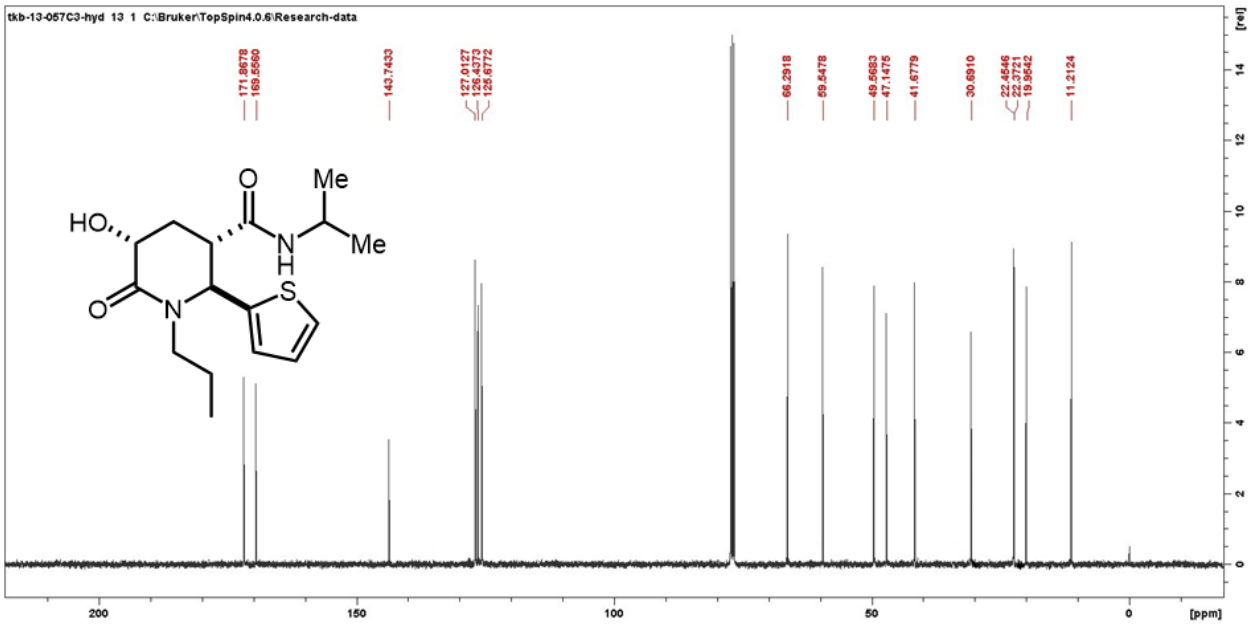
S
[ppm]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127.0127
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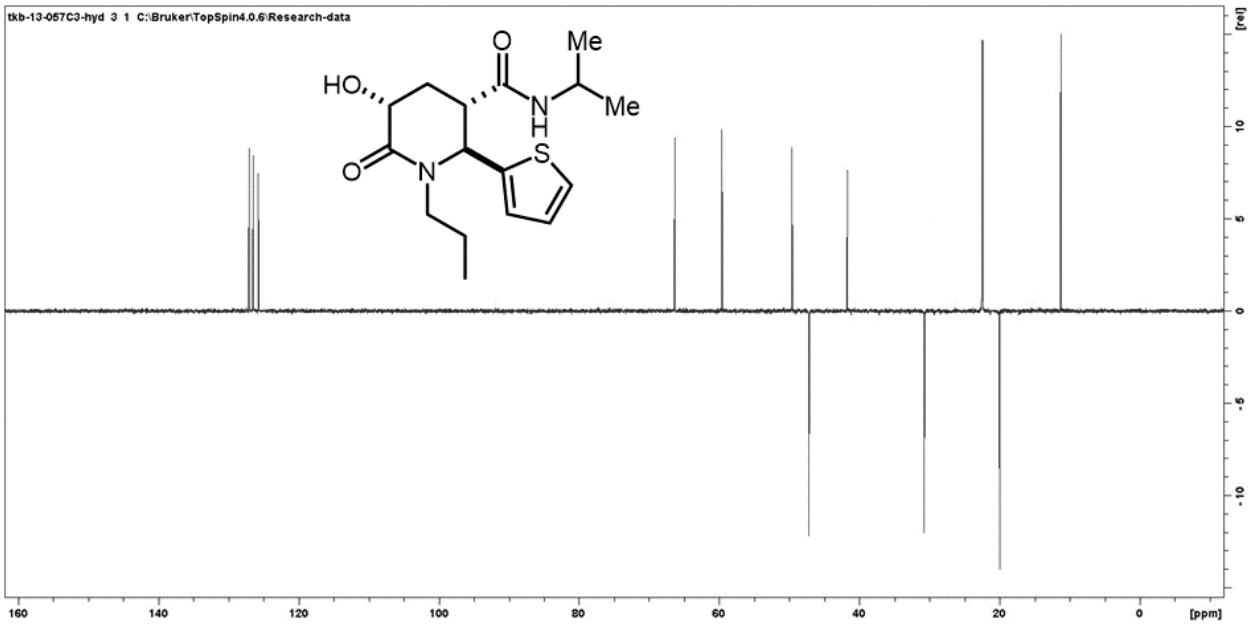
O
N
O
Me
N
H
Me
S
[ppm]


tkb-13-057C3-hyd 3 1 C:\Bruker\TopSpin4.0.6\Research-data
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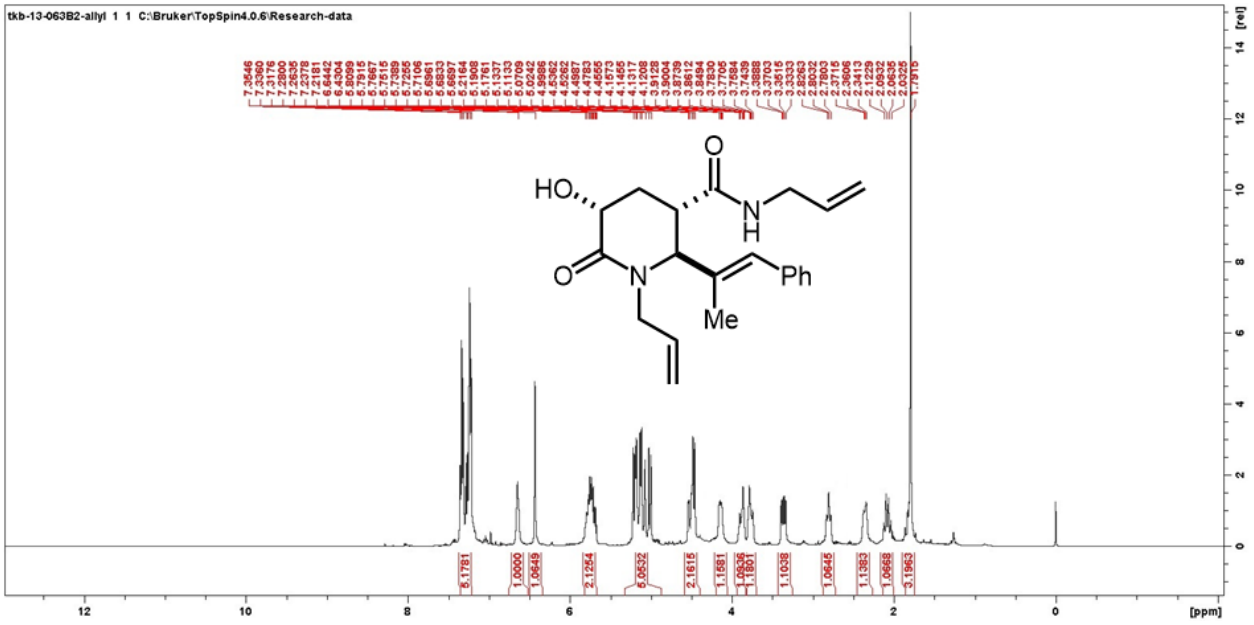
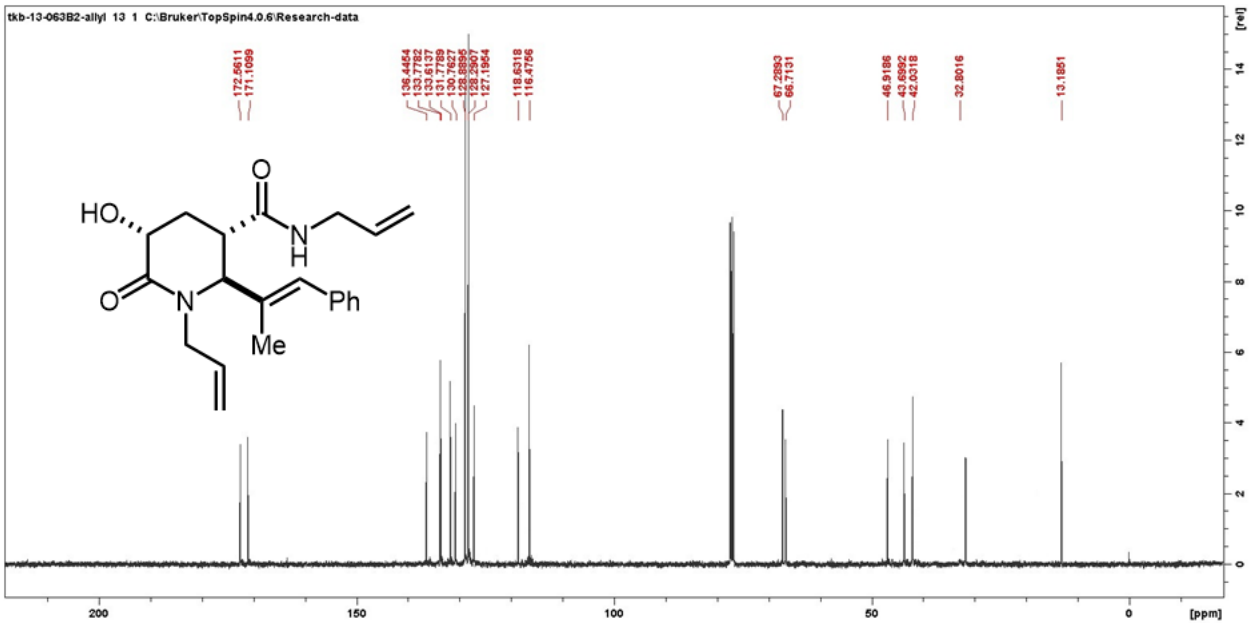
O
N
H
Me
Me
O
N
S
160
140
120
100
80
60
40
20
0
[ppm]

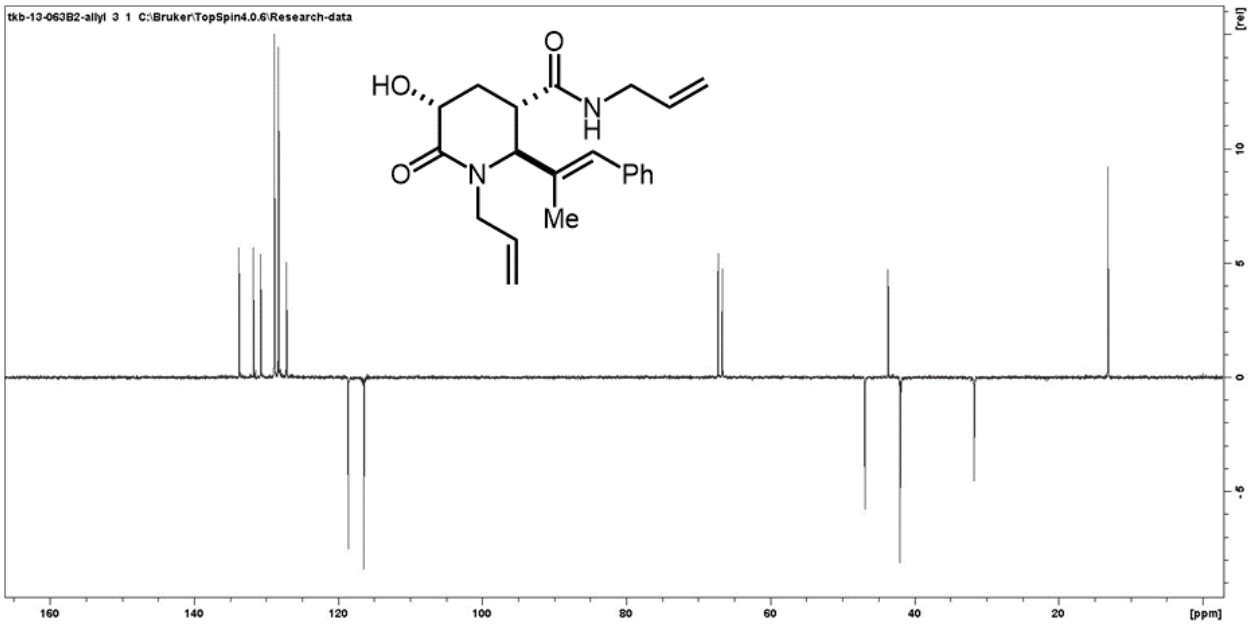
Scope of amines

Compound 7t

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 60:40). Pale-yellow oil. Yield = 329.6 mg, 93%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 – 7.20 (m, 5H), 6.64 (d, *J* = 6.1 Hz, 1H), 6.43 (s, 1H), 5.74 (dqd, *J* = 22.4, 11.0, 4.0 Hz, 2H), 5.24 – 5.05 (m, 4H), 5.01 (d, *J* = 10.2 Hz, 1H), 4.51 (dd, *J* = 14.8, 4.6 Hz, 1H), 4.47 (d, *J* = 9.0 Hz, 1H), 4.14 (dd, *J* = 11.2, 5.4 Hz, 1H), 3.88 (dt, *J* = 16.0, 5.7 Hz, 1H), 3.76 (dt, *J* = 15.9, 5.6 Hz, 1H), 3.36 (dd, *J* = 14.9, 7.4 Hz, 1H), 2.81 (ddt, *J* = 12.5, 9.3, 4.7 Hz, 1H), 2.74 – 2.61 (m, 1H), 2.41 – 2.25 (m, 1H), 2.06 (dt, *J* = 22.4, 11.1 Hz, 1H), 2.02 – 1.99 (m, 1H), 1.79 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.56, 171.11, 136.45, 133.78, 133.62, 131.78, 130.77, 128.89, 128.29, 127.20, 118.64, 116.48, 67.29, 66.72, 46.92, 43.70, 42.03, 32.80, 13.19. **HRMS-EI$^+$** (*m/z*): calc for $C_{21}H_{26}N_2O_3$ $[M]^+$ 354.1943, found 354.1958.



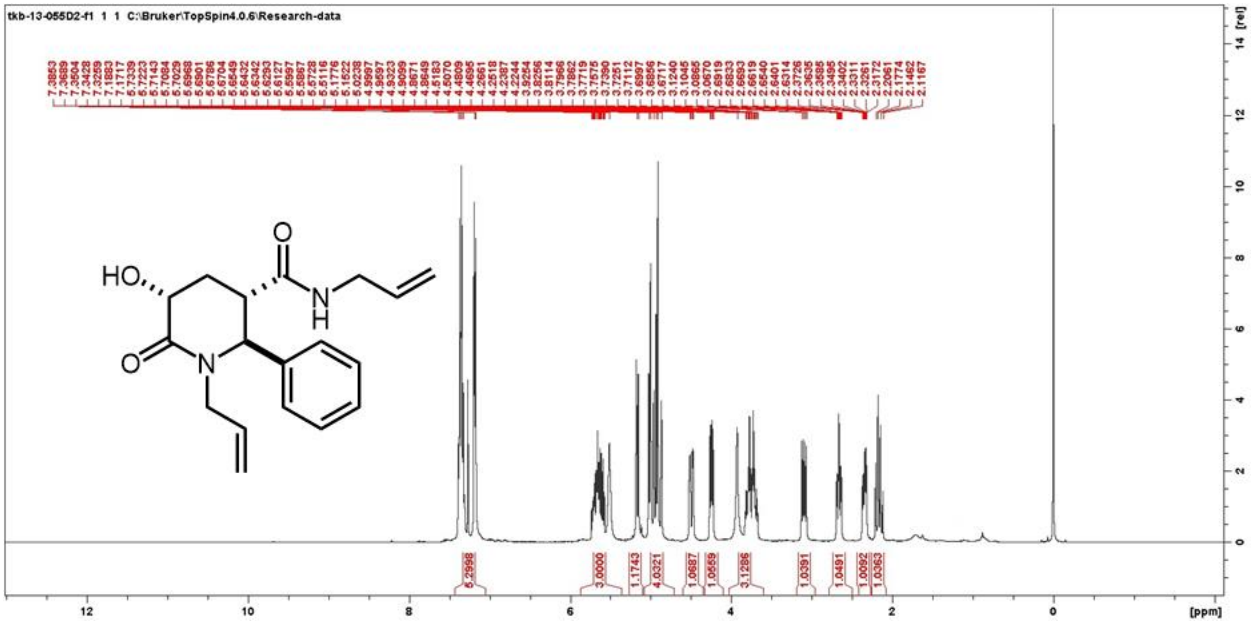


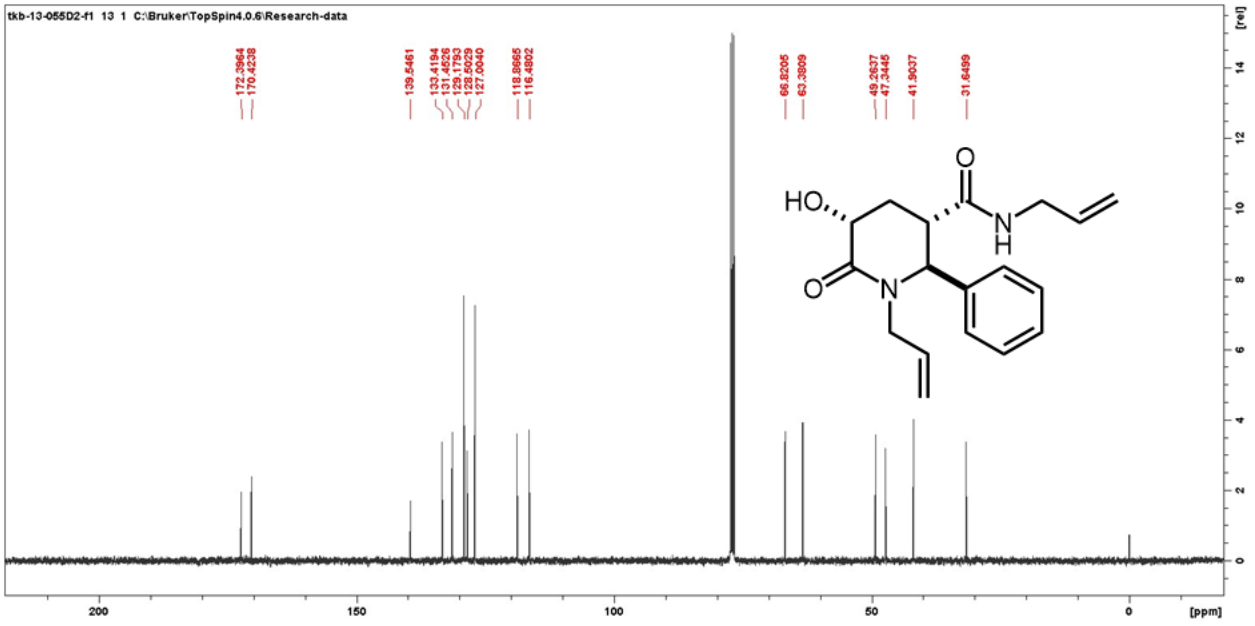


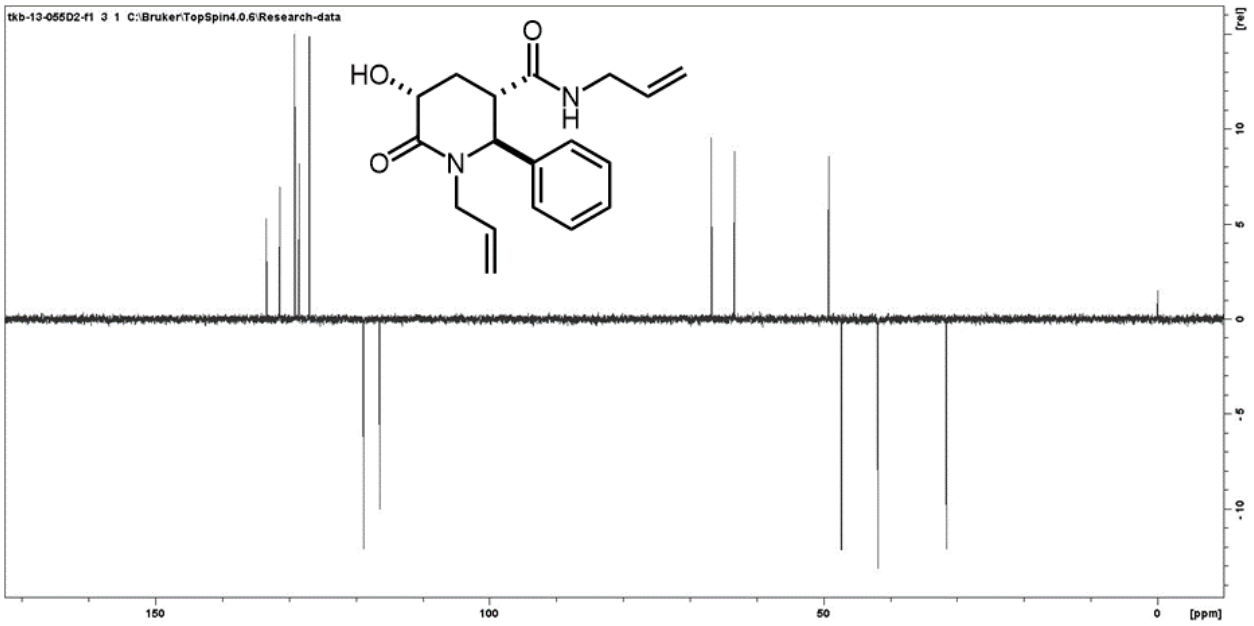
Compound 7u

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 50:50). Pale-yellow oil. Yield = 279.8 mg, 89%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (qd, *J* = 7.7, 3.6 Hz, 3H), 7.22 – 7.14 (m, 2H), 5.76 – 5.55 (m, 2H), 5.50 (d, *J* = 6.1 Hz, 1H), 5.17 (d, *J* = 10.2 Hz, 1H), 5.05 – 4.84 (m, 4H), 4.55 – 4.44 (m, 1H), 4.25 (dd, *J* = 11.0, 5.7 Hz, 1H), 3.95 – 3.90 (m, 1H), 3.85 – 3.65 (m, 2H), 3.10 (dd, *J* = 15.0, 7.8 Hz, 1H), 2.66 (ddd, *J* = 12.2, 9.0, 3.5 Hz, 1H), 2.35 (ddd, *J* = 13.0, 5.8, 3.5 Hz, 1H), 2.16 (dt,

J = 13.0, 11.4 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.40, 170.43, 139.55, 133.42, 131.46, 129.18, 128.51, 127.01, 118.87, 116.48, 66.83, 63.38, 49.27, 47.35, 41.91, 31.65. **HRMS-EI$^+$** (*m/z*): calc for $C_{18}H_{22}N_2O_3$ $[M]^+$ 314.1630, found 314.1636.

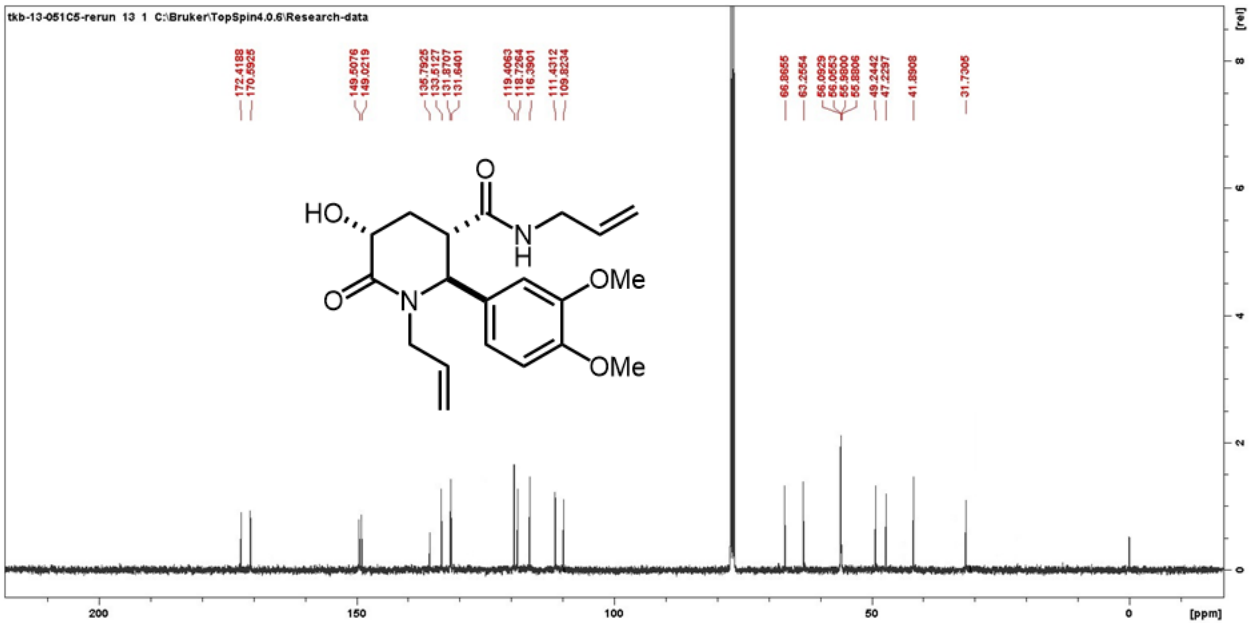


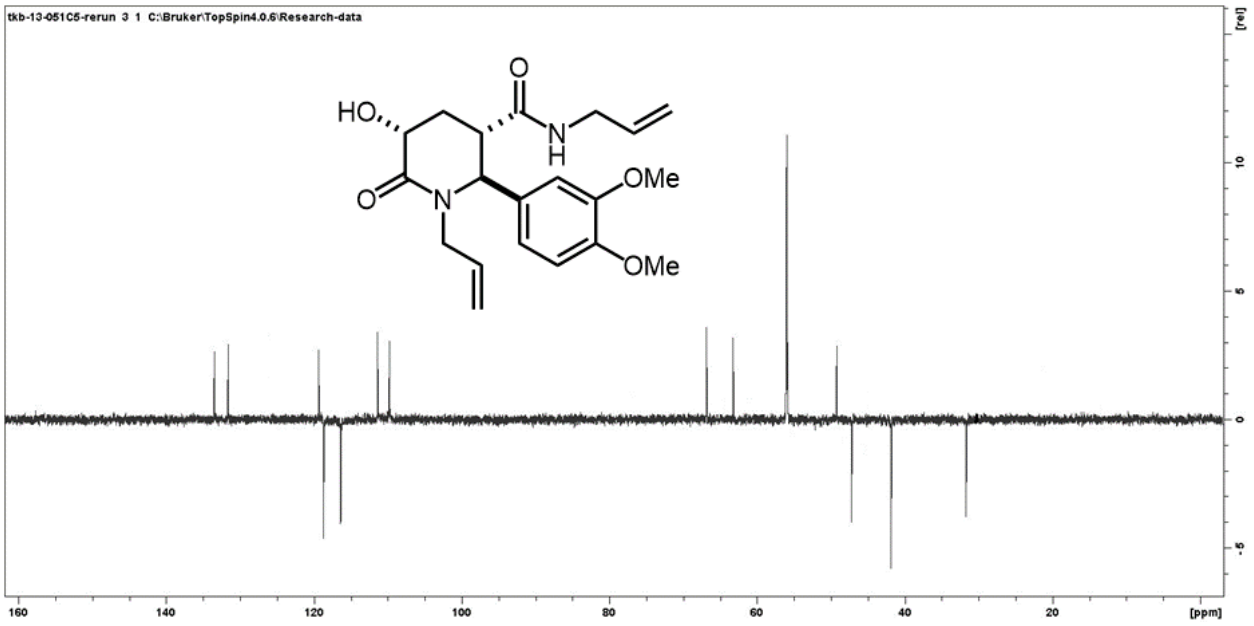




Compound 7v

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 25:75). Yellowish oil. Yield = 359.4 mg, 96%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.98 (s, 1H), 6.85 (dd, *J* = 12.9, 8.3 Hz, 1H), 6.82 – 6.60 (m, 2H), 5.77 – 5.57 (m, 2H), 5.12 (s, 1H), 5.27 – 4.81 (m, 5H), 4.62 – 4.42 (m, 1H), 4.24 (dt, *J* = 14.2, 7.0 Hz, 1H), 4.02 – 3.88 (m, 1H), 3.87 (dd, *J* = 12.1, 3.5 Hz, 7H), 3.84 – 3.64 (m, 1H), 3.71 (s, 1H), 3.14 (dq, *J* = 13.9, 7.9 Hz, 1H), 2.80 – 2.55 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.42, 170.60, 149.51, 149.03, 135.80, 133.52, 131.87, 131.64, 125.52, 119.41, 118.73, 116.39, 111.43, 109.83, 66.87, 63.26, 56.10, 56.06, 55.98, 55.88, 49.25, 47.23, 41.89, 31.73. **HRMS-EI^{+}** (*m/z*): calc for $C_{20}H_{26}N_2O_5$ $[M]^{+}$ 374.1842, found 374.1845.

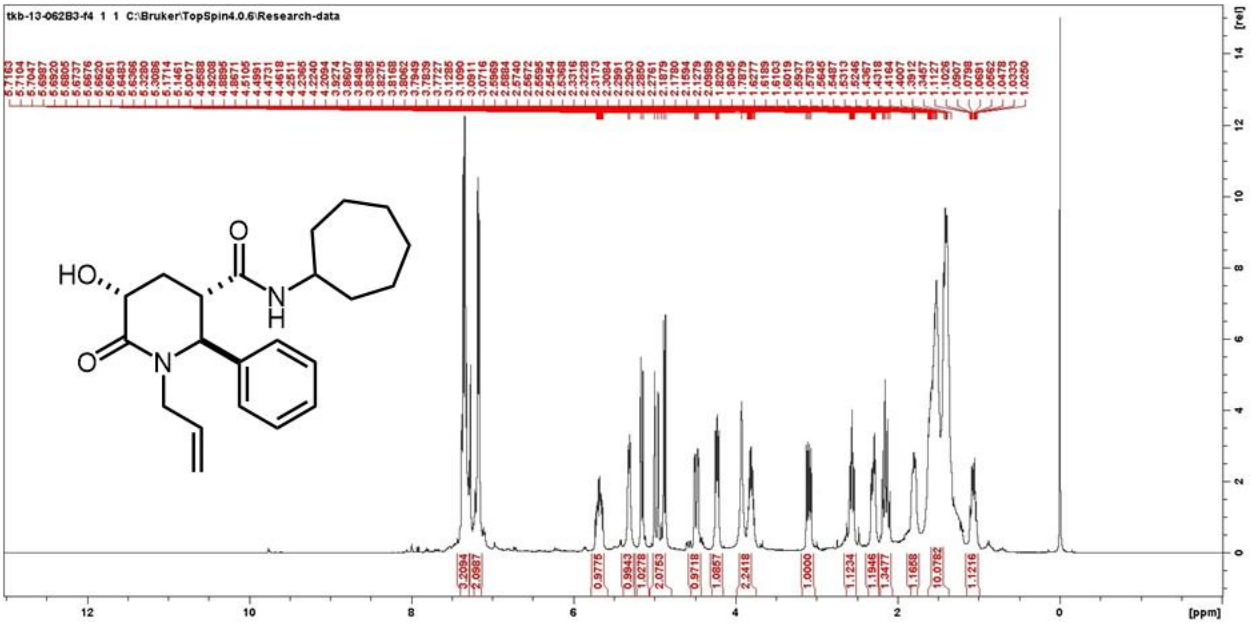


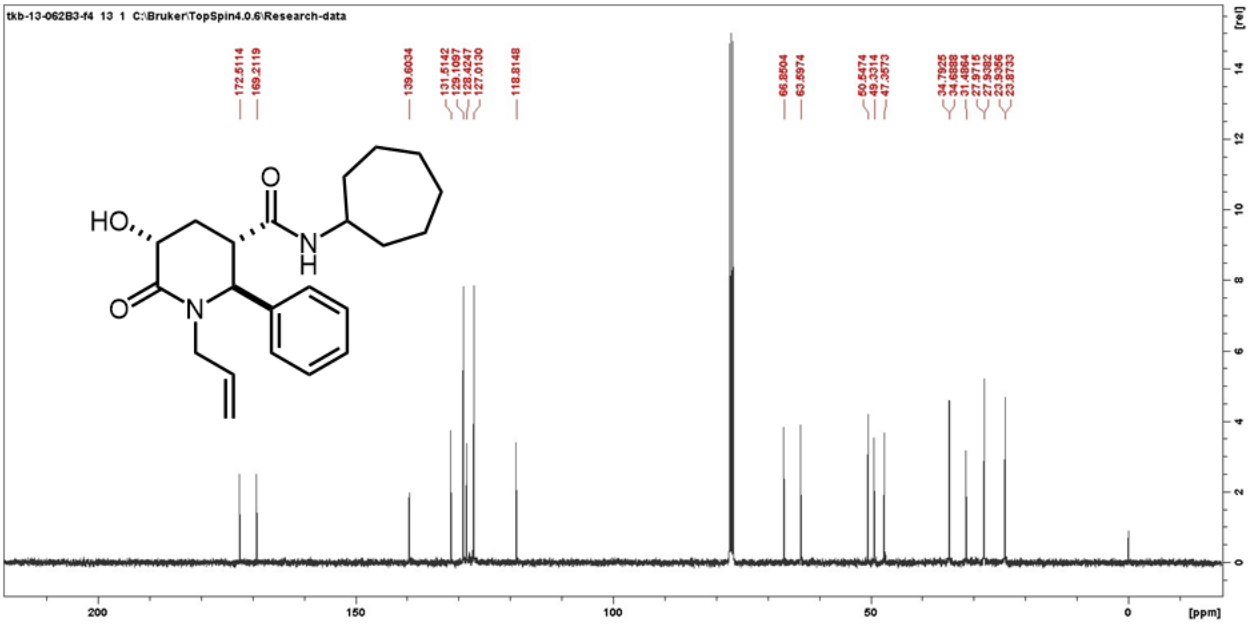


Compound 7w

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 50:50). Yellowish oil. Yield = 314.9 mg, 85%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 – 7.26 (m, 3H), 7.26 – 7.13 (m, 2H), 5.69 (dddd, *J* = 17.6, 10.2, 7.8, 4.6 Hz, 1H), 5.32 (d, *J* = 8.1 Hz, 1H), 5.20 – 5.12 (m, 1H), 4.98 (dd, *J* = 17.3, 1.8 Hz, 1H), 4.88 (d, *J* = 9.0 Hz, 1H), 4.63 – 4.38 (m, 1H), 4.23 (dd, *J* = 10.9, 5.8 Hz, 1H), 3.93 (s, 1H), 3.81 (ddh, *J* = 13.9, 9.5, 4.7 Hz, 1H), 3.10 (dd, *J* = 15.0, 7.8 Hz, 1H), 2.57 (ddd, *J* = 12.2, 8.9, 3.5

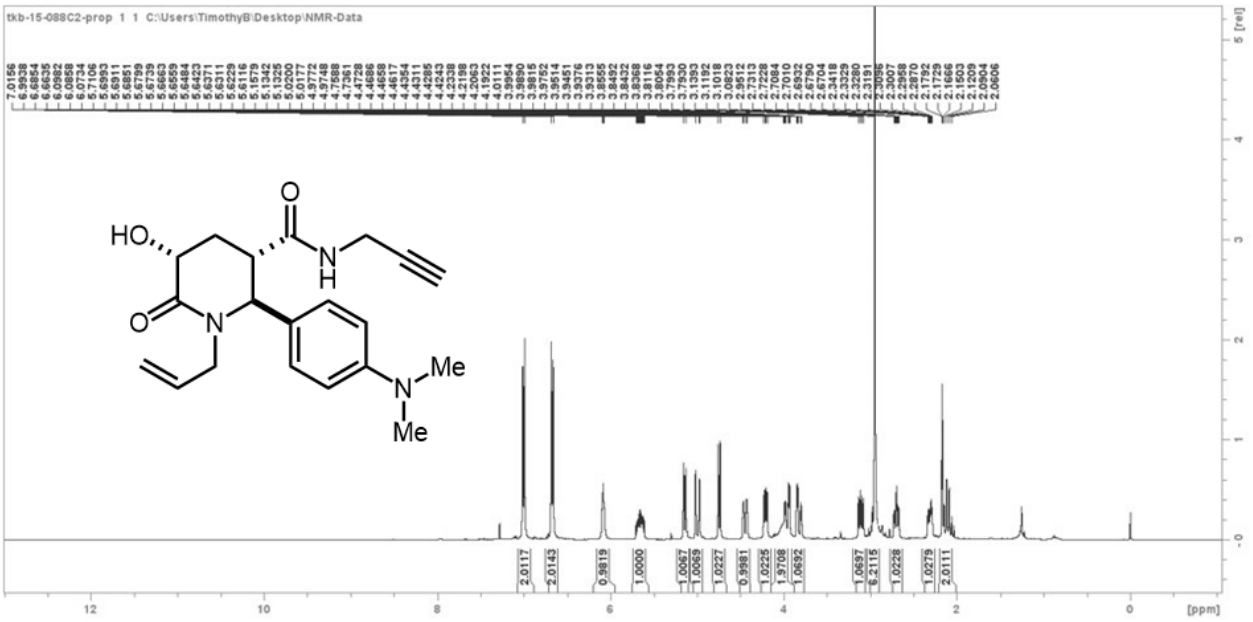
Hz, 1H), 2.30 (ddd, *J* = 13.1, 5.9, 3.5 Hz, 1H), 2.22 – 2.07 (m, 1H), 1.86 – 1.75 (m, 1H), 1.69 – 1.56 (m, 1H), 1.46 – 1.28 (m, 10H), 1.07 (dtd, *J* = 12.8, 8.9, 3.7 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.51, 169.22, 139.61, 131.52, 129.11, 128.43, 127.02, 118.82, 66.86, 63.60, 50.55, 49.34, 47.36, 34.80, 34.69, 31.49, 27.97, 27.94, 23.94, 23.88. **HRMS-EI$^+$** (*m/z*): calc for $C_{22}H_{30}N_2O_3$ $[M]^+$ 370.2256, found 370.2259.





Compound 7x

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 25:75). Yellowish oil. Yield = 330.5 mg, 93%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 6.99 (d, *J* = 7.2 Hz, 2H), 6.67 (d, *J* = 7.2 Hz, 2H), 6.09 (t, *J* = 5.2 Hz, 1H), 5.66 (dddd, *J* = 17.6, 10.2, 7.8, 4.5 Hz, 1H), 5.15 (dq, *J* = 10.2, 1.3 Hz, 1H), 5.00 (dq, *J* = 17.1, 1.5 Hz, 1H), 4.75 (d, *J* = 9.1 Hz, 1H), 4.45 (ddt, *J* = 15.0, 4.6, 1.7 Hz, 1H), 4.21 (dd, *J* = 11.1, 5.6 Hz, 1H), 3.96 (ddd, *J* = 17.6, 5.6, 2.6 Hz, 1H), 3.82 (ddd, *J* = 17.6, 5.0, 2.6 Hz, 1H), 3.11 (dd, *J* = 15.0, 7.8 Hz, 1H), 2.95 (s,s, 6H), 2.70 (ddd, *J* = 12.3, 9.0, 3.5 Hz, 1H), 2.31 (ddd, *J* = 12.8, 5.6, 3.5 Hz, 1H), 2.14 – 2.02 (m, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.25, 170.85, 150.43, 131.69, 127.86, 126.44, 118.55, 112.66, 79.11, 71.69, 66.84, 62.82, 48.70, 46.95, 40.43, 31.41, 29.25. **HRMS-EI^{+}** (*m/z*): calc for $C_{20}H_{25}N_3O_3$ $[M]^{+}$ 355.1896, found 355.1893.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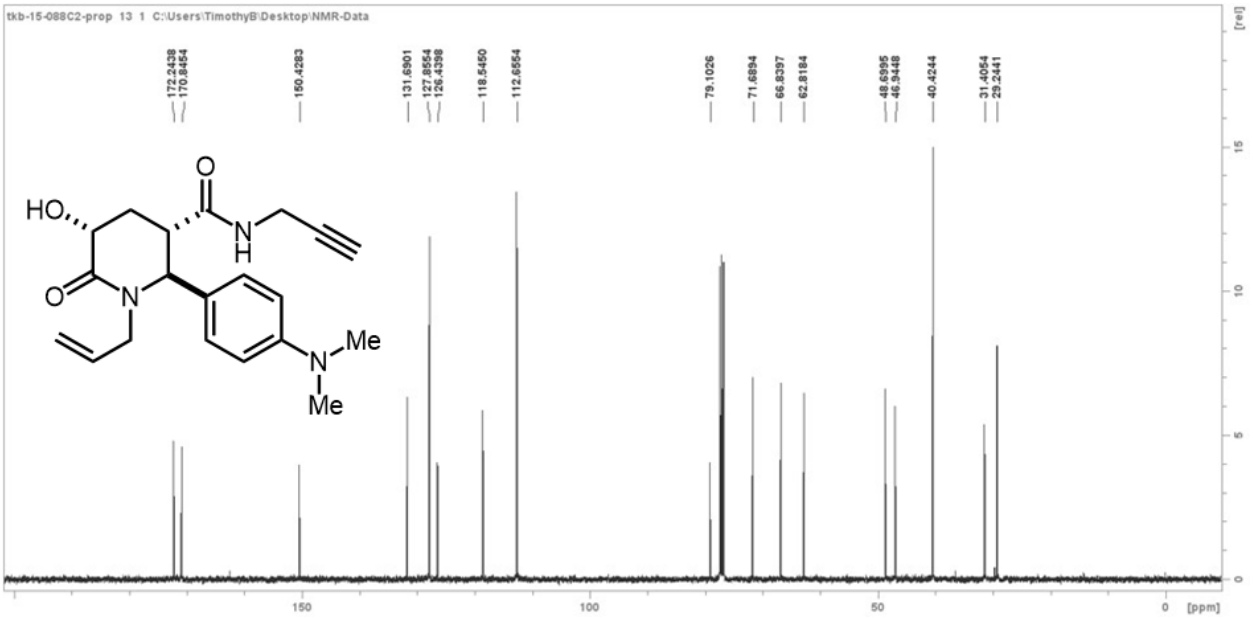
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62.8184
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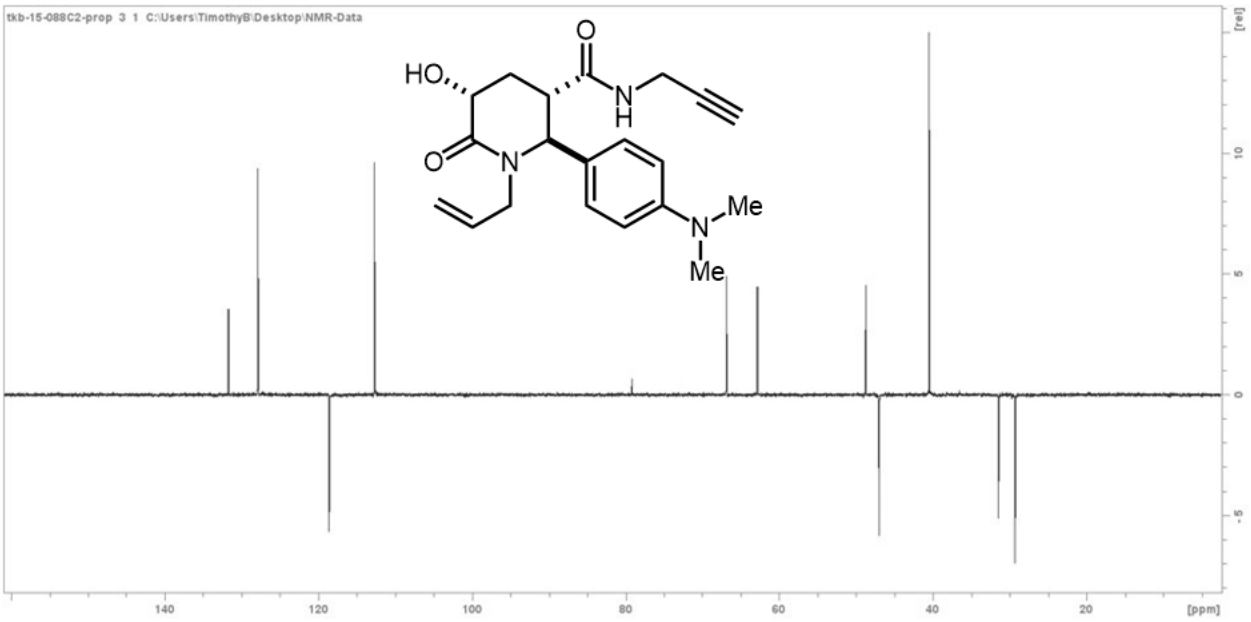
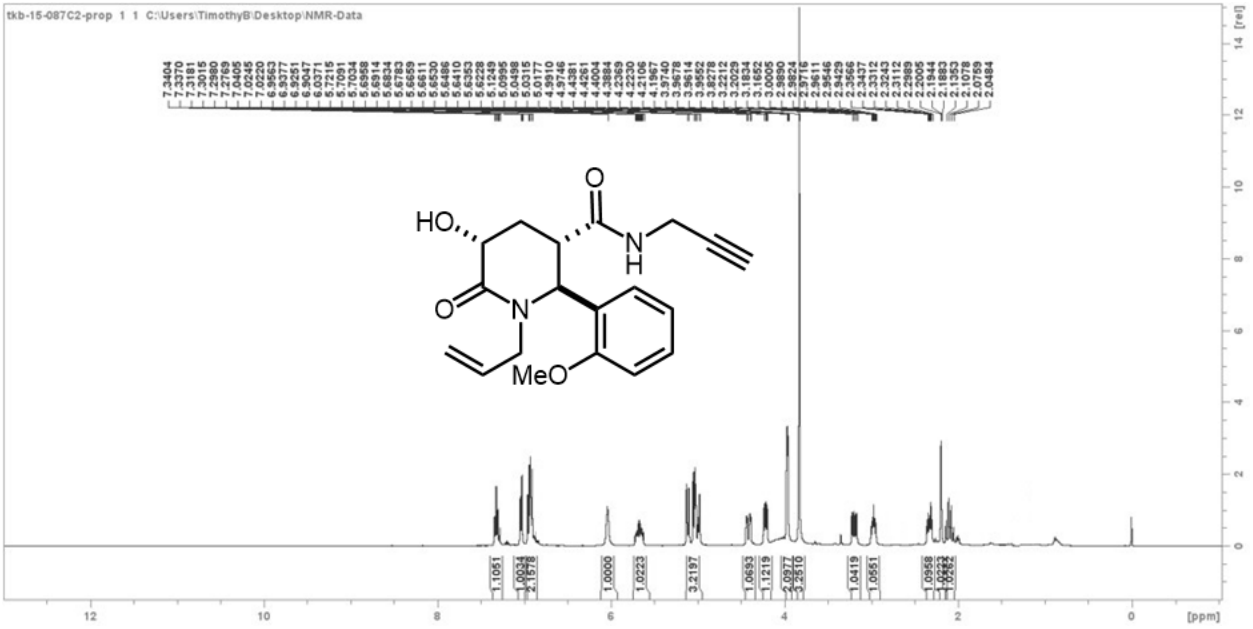
H
O
N
Me
N
Me
[ppm]



Compound 7y

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 50:50). Yellowish oil. Yield = 301.3 mg, 88%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 (td, *J* = 7.8, 1.7 Hz, 1H), 7.03 (dd, *J* = 7.5, 1.8 Hz, 1H), 6.98 – 6.90 (m, 1H), 6.94 – 6.81 (m, 1H), 6.04 (t, *J* = 5.3 Hz, 1H), 5.67 (dddd, *J* = 17.3, 10.3, 7.2, 4.9 Hz, 1H), 5.11 (dt, *J* = 10.2, 1.3 Hz, 1H), 5.07 – 4.95 (m, 2H), 4.41 (ddt, *J* = 15.1, 5.0, 1.6 Hz, 1H), 4.22 (dd, *J* = 10.6, 5.6 Hz, 1H), 3.96 (dd, *J* = 5.2, 2.6 Hz, 2H), 3.83 (s, 3H), 3.19 (dd, *J* = 15.1, 7.3 Hz, 1H), 2.97 (ddd, *J* = 11.6, 7.3, 4.6 Hz, 1H), 2.33 (dt, *J* = 13.1, 5.1 Hz, 1H), 2.19 (s, 1H), 2.15 – 1.96 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.44, 171.54, 156.90, 131.83, 129.83, 128.72, 126.86, 120.94, 118.26, 111.33, 79.14, 71.78, 66.22, 59.74, 55.44, 47.61, 45.13, 31.53, 29.37. **HRMS-EI^{+}** (*m/z*): calc for $C_{19}H_{22}N_2O_4$ $[M]^{+}$ 342.1580, found 342.1585.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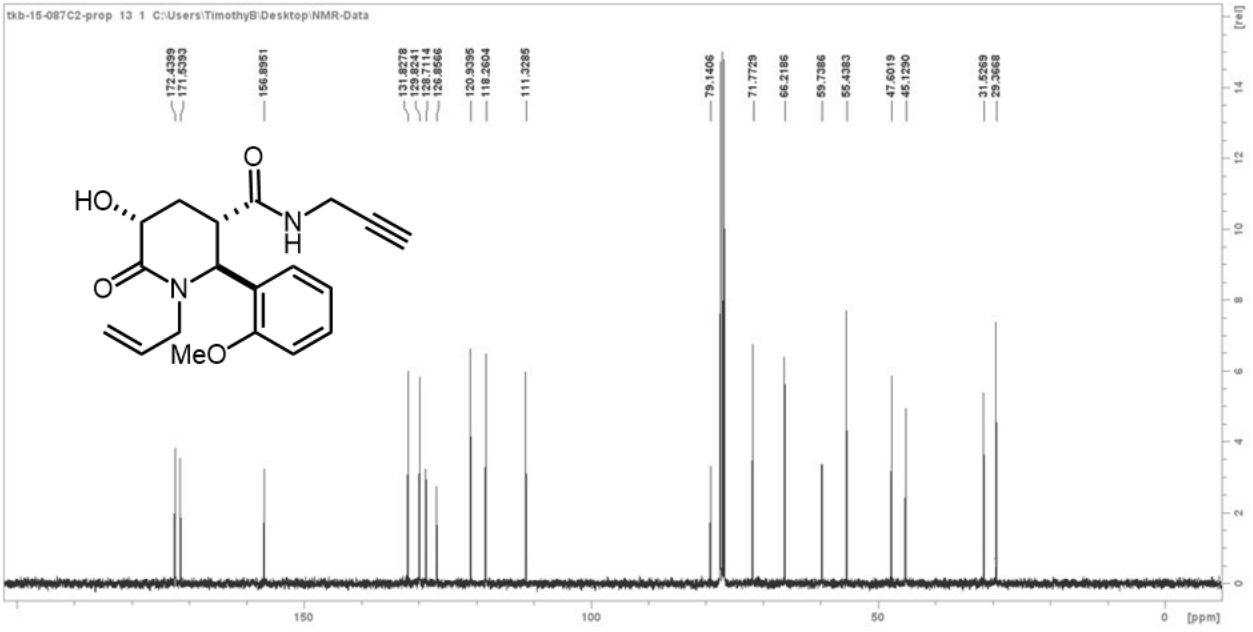
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66.2186
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O
N
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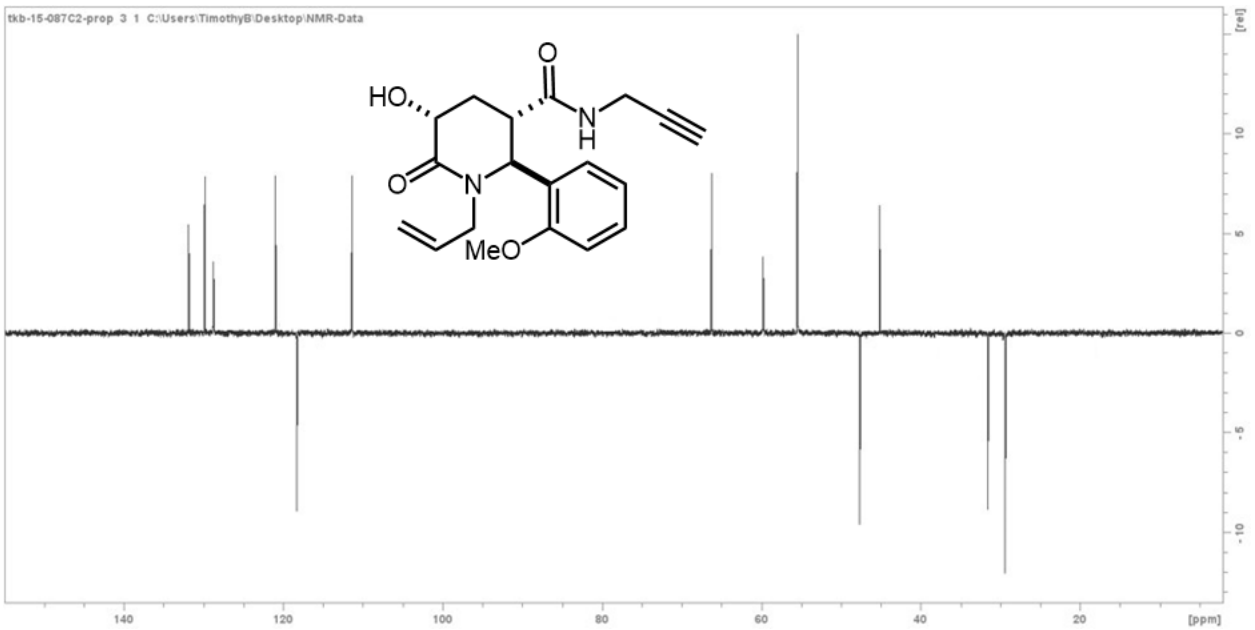
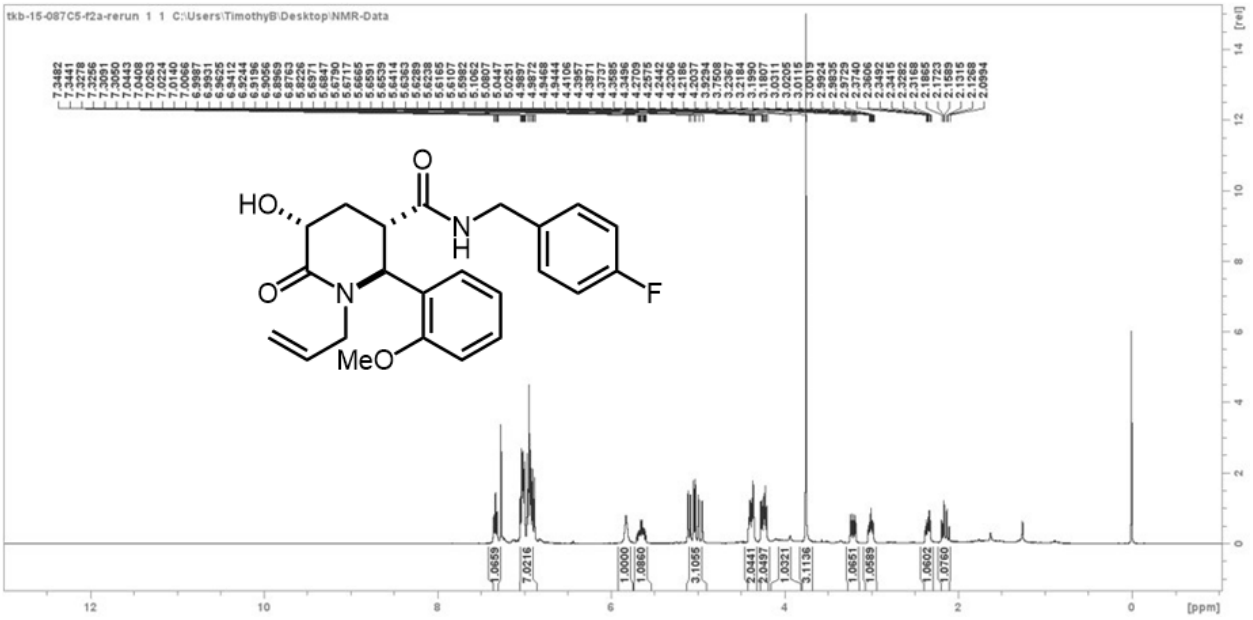


Compound 7z

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 50:50). Greenish-yellow oil. Yield = 301.3 mg, 85%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.33 (td, *J* = 7.8, 1.8 Hz, 1H), 7.15 – 6.85 (m, 7H), 5.82 (s, 1H), 5.65 (dddd, *J* = 17.3, 10.2, 7.3, 5.0 Hz, 1H), 5.09 (dd, *J* = 10.0, 1.6 Hz, 1H), 5.04 (d, *J* = 7.9 Hz, 1H), 4.97 (dq, *J* = 17.0, 1.5 Hz, 1H), 4.38 (ddd, *J* = 15.0, 5.6, 4.1 Hz, 2H), 4.29 – 4.18 (m, 2H), 4.00 – 3.91 (m, 1H), 3.75 (s, 3H), 3.21 (dd, *J* = 15.1, 7.3 Hz, 1H), 3.00 (ddd, *J* = 11.7, 7.8, 4.2 Hz, 1H), 2.35 (ddd, *J* = 13.0, 5.6, 4.2 Hz, 1H), 2.21 – 2.08 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.23, 171.51, 163.38, 160.93, 157.02, 133.60, 133.57, 131.89, 129.90, 129.37, 129.30, 129.22, 126.62, 120.92, 118.24, 115.60, 115.39, 111.41, 66.39, 60.47, 55.42, 47.51, 45.42, 42.95, 31.55. **HRMS-EI$^+$** (*m/z*): calc for $C_{23}H_{25}FN_2O_4$ $[M]^+$ 412.1798, found 412.1793.

^{1}H NMR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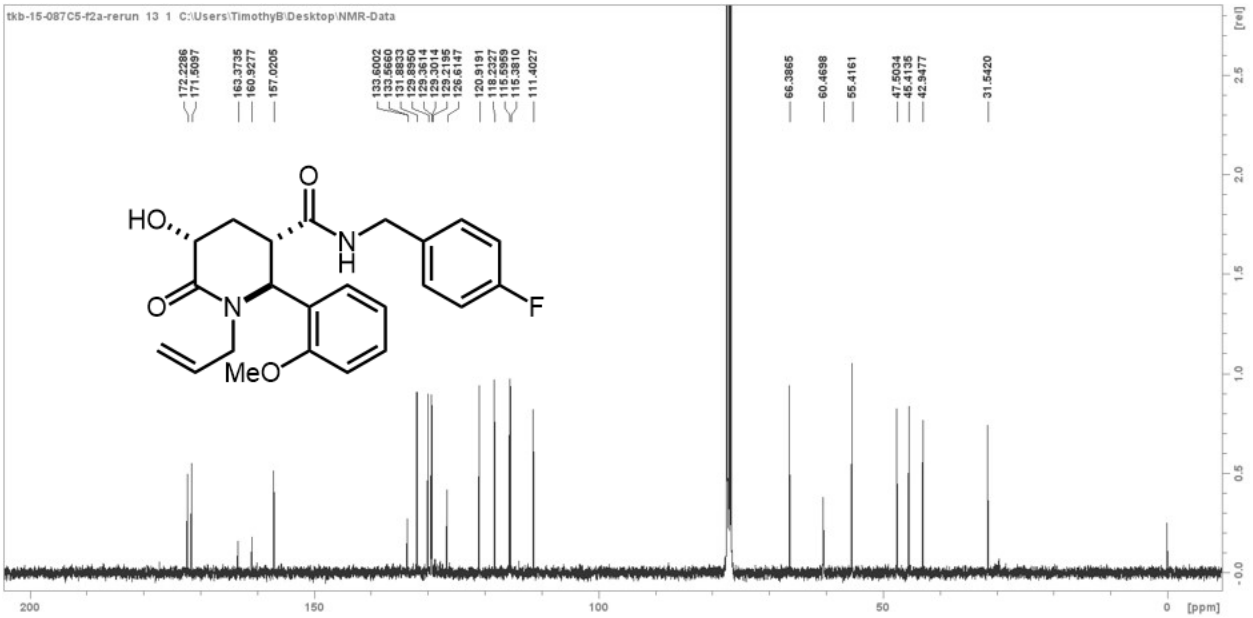
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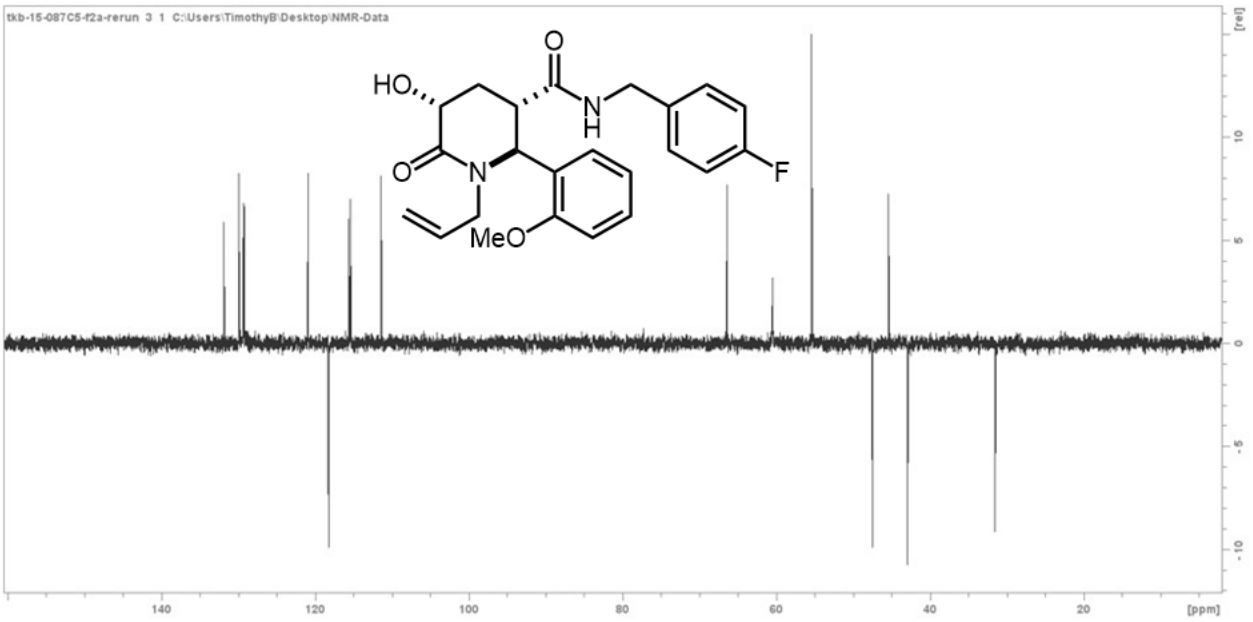
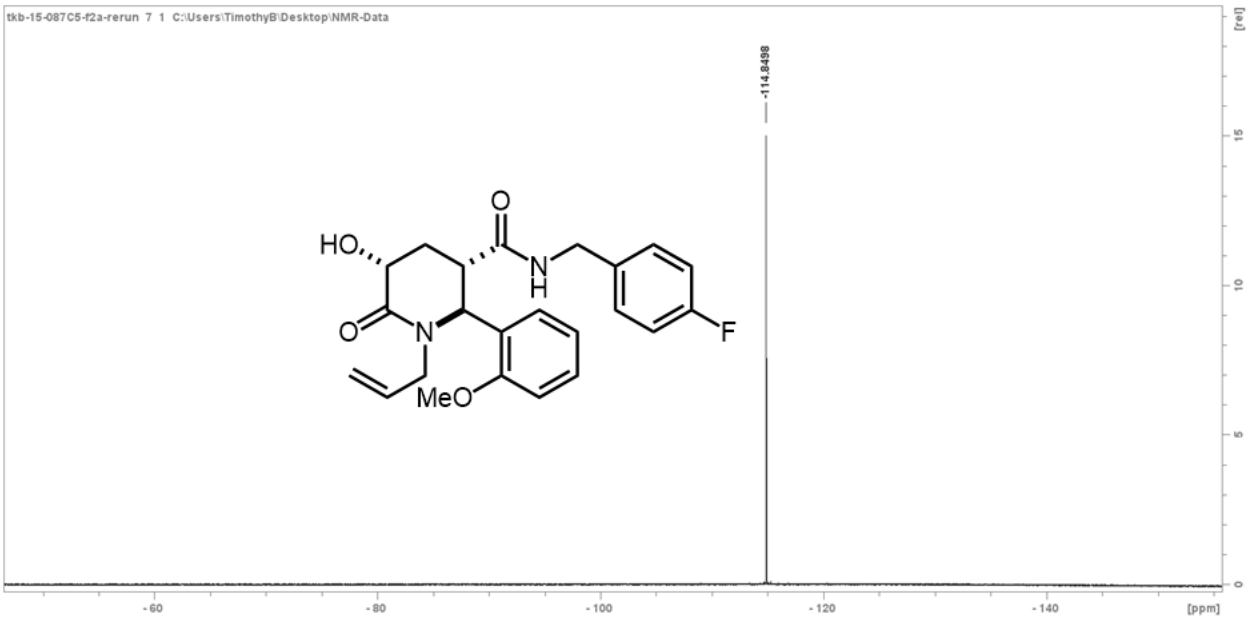


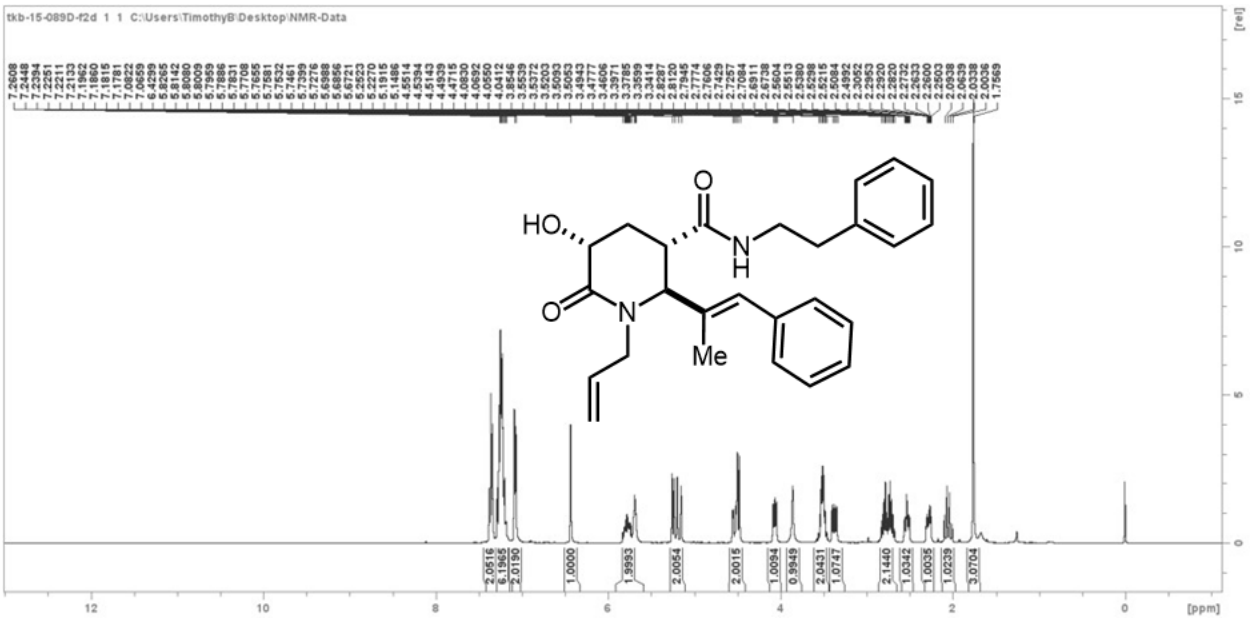
^{19}F NMR

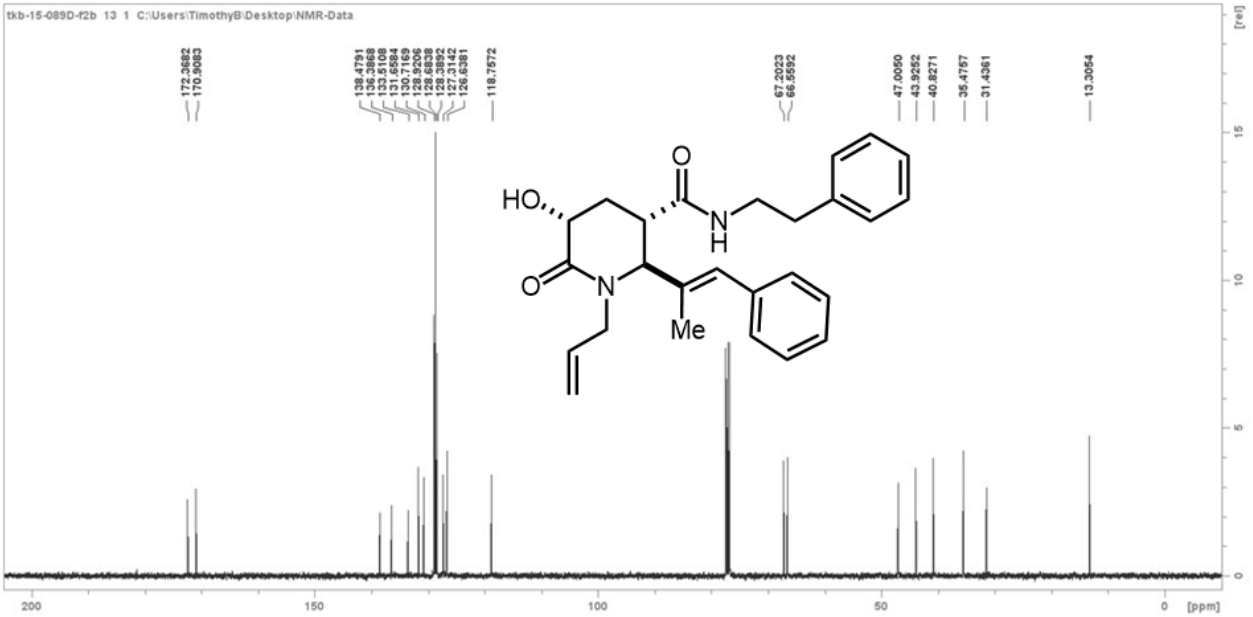


Compound 7z1

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 50:50). Greenish-yellow oil. Yield = 376.7 mg, 90%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 (t, *J* = 7.5 Hz, 2H), 7.32 – 7.14 (m, 6H), 7.11 – 7.04 (m, 2H), 6.43 (s, 1H), 5.78 (dddd, *J* = 17.4, 10.3, 7.4, 4.9 Hz, 1H), 5.69 (t, *J* = 5.9 Hz, 1H), 5.24 (dd, *J* = 10.3, 1.5 Hz, 1H), 5.17 (dd, *J* = 17.1, 1.5 Hz, 1H), 4.58 – 4.48 (m, 1H), 4.48 (d, *J* = 8.9

Hz, 1H), 4.06 (dd, *J* = 11.2, 5.5 Hz, 1H), 3.85 (s, 1H), 3.51 (qd, *J* = 6.7, 4.2 Hz, 2H), 3.37 (dd, *J* = 14.8, 7.4 Hz, 1H), 2.75 (dtd, *J* = 27.7, 13.8, 6.8 Hz, 2H), 2.53 (ddd, *J* = 12.3, 8.9, 3.8 Hz, 1H), 2.28 (ddd, *J* = 12.9, 5.5, 3.8 Hz, 1H), 2.05 (q, *J* = 12.0 Hz, 1H), 1.76 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.37, 170.91, 138.48, 136.39, 133.51, 131.66, 130.72, 128.92, 128.69, 128.39, 127.32, 126.64, 118.76, 67.21, 66.56, 47.01, 43.93, 40.83, 35.48, 31.44, 13.31. **HRMS-EI$^+$** (*m/z*): calc for $C_{26}H_{30}N_2O_3$ $[M]^+$ 418.2256, found 418.2252.



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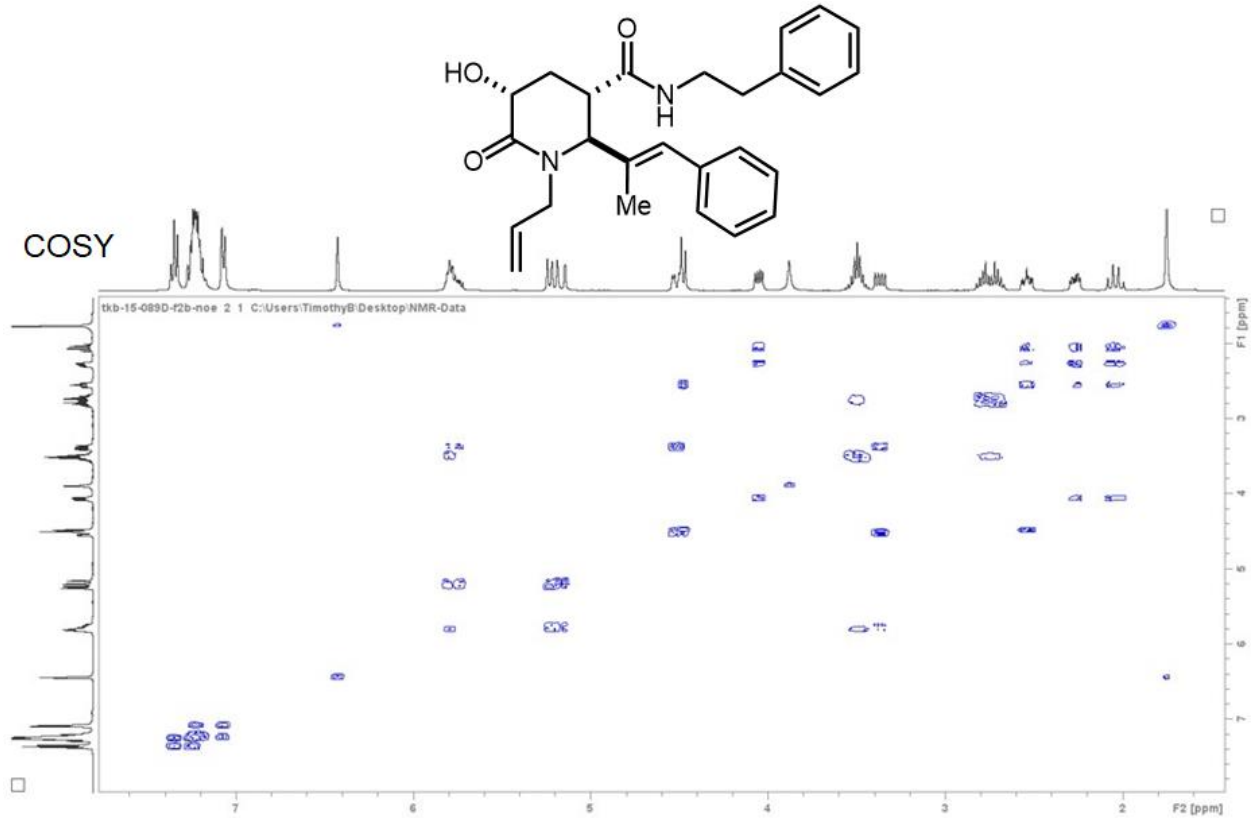
[ppm]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tkb-15-089D-f2b-noe 2 1 C:\Users\TimothyB\Desktop\NMR-Data
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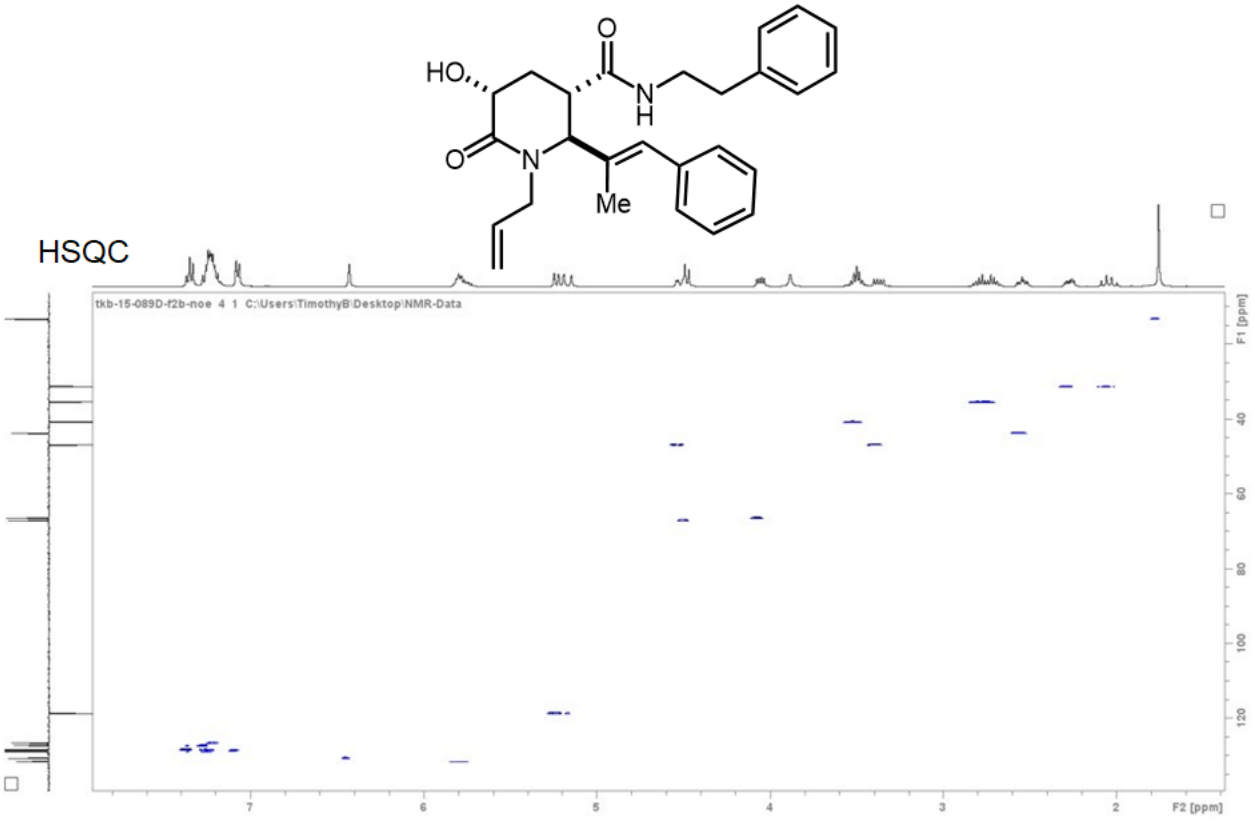
F2 [ppm]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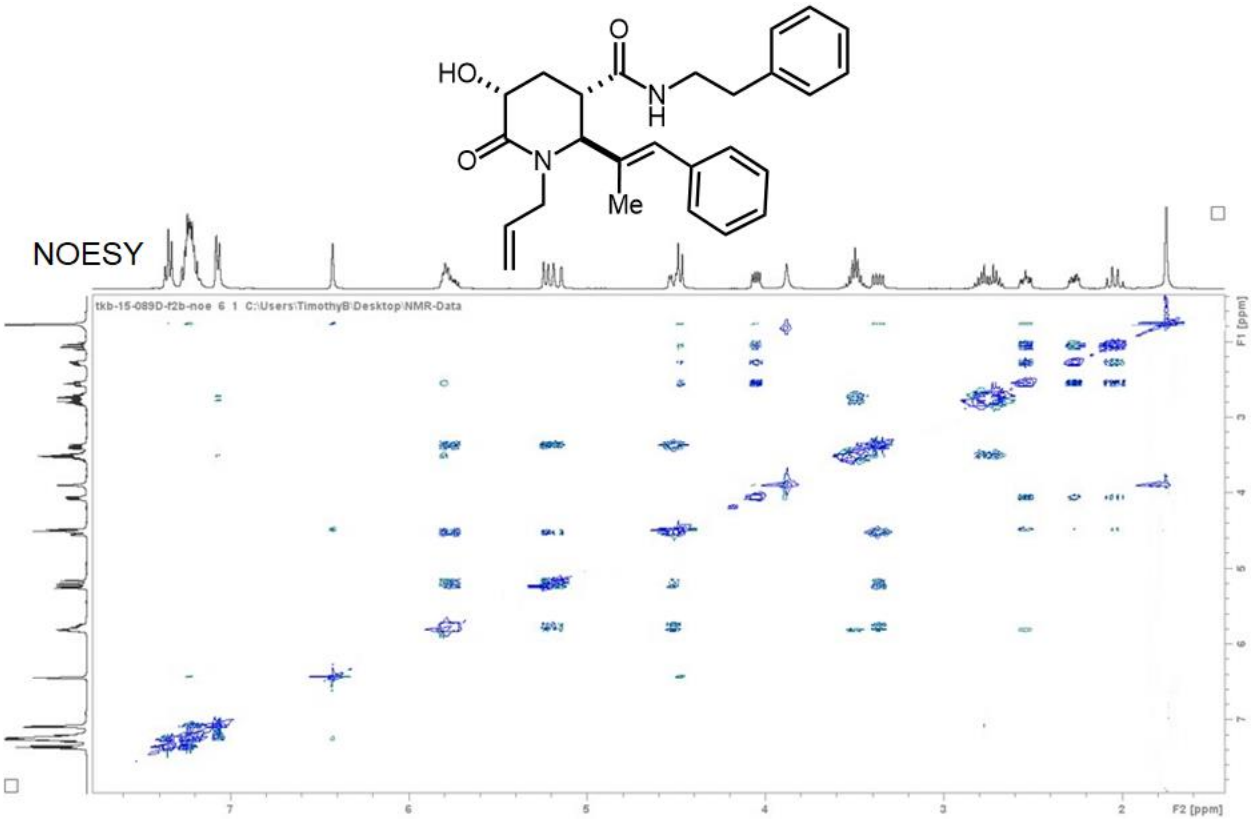
F1 [ppm]
F2 [ppm]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F1 [ppm]
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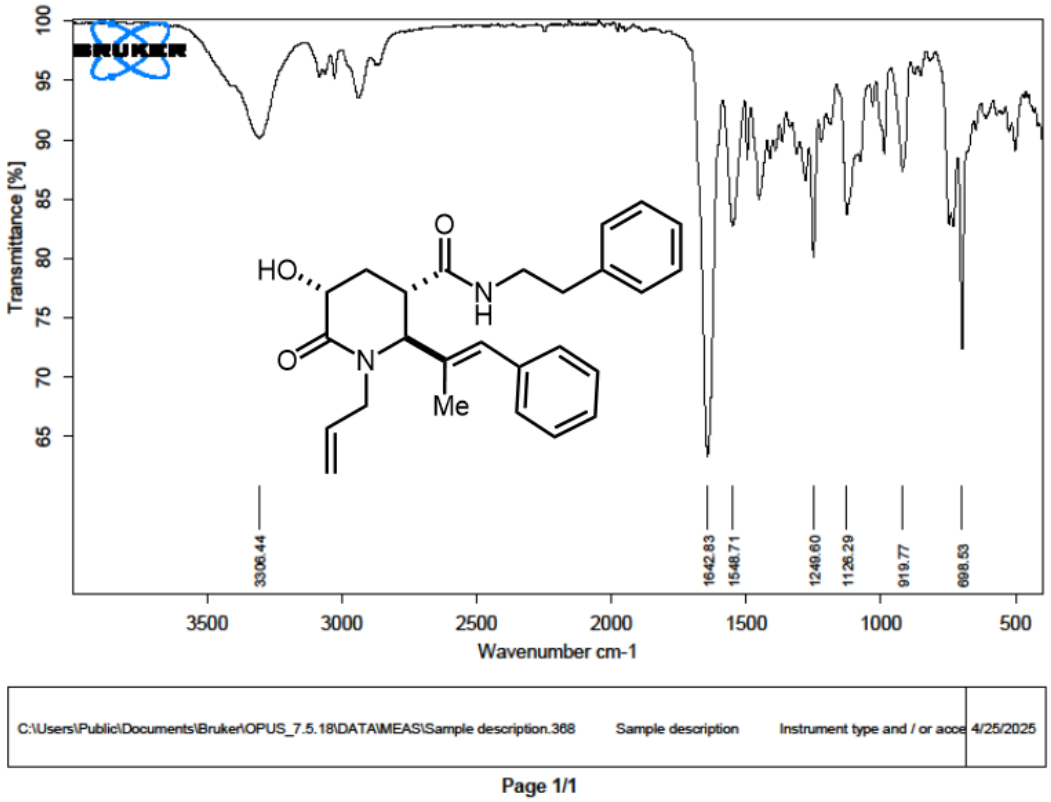
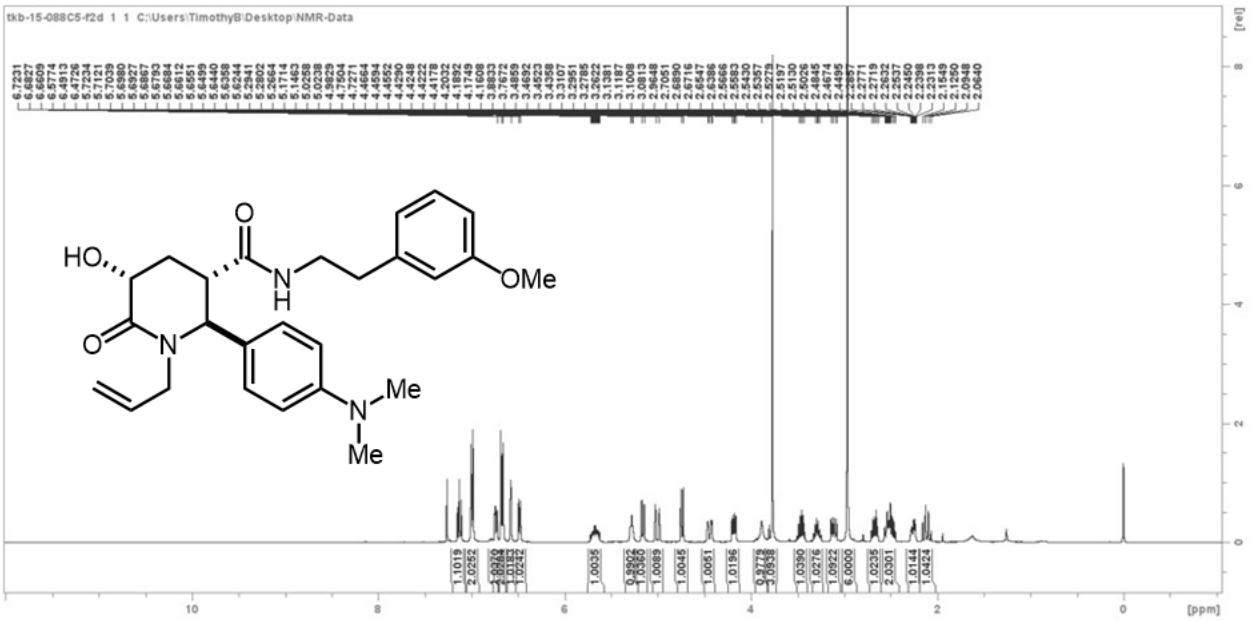
IR Spectrum



Compound 7z2

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 50:50). Greenish-yellow oil. Yield = 397.3 mg, 88%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.13 (t, *J* = 7.9 Hz, 1H), 7.04 – 6.95 (m, 2H), 6.82 – 6.71 (m, 1H), 6.71 – 6.63 (m, 2H), 6.58 (t, *J* = 2.1 Hz, 1H), 6.48 (dt, *J* = 7.5, 1.2 Hz, 1H), 5.67 (dddd, *J* = 17.6, 10.2, 7.8, 4.5 Hz, 1H), 5.28 (t, *J* = 5.8 Hz, 1H), 5.16 (dq, *J* = 10.1, 1.3 Hz, 1H), 5.00 (dq, *J* = 17.2, 1.5 Hz, 1H), 4.74 (d, *J* = 9.3 Hz, 1H), 4.44 (ddt, *J* = 15.0, 4.7, 1.7 Hz, 1H), 4.18 (dd, *J* = 11.4, 5.6 Hz, 1H), 3.88 (s, 1H), 3.77 (s, 3H), 3.46 (dq, *J* = 13.2, 6.6 Hz, 1H), 3.29 (tt, *J* = 13.4, 6.3 Hz, 1H), 3.11 (dd, *J* = 14.9, 7.8 Hz, 1H), 2.96 (s, 6H), 2.67 (dt, *J* = 13.3, 6.5 Hz, 1H), 2.59 – 2.43 (m, 2H), 2.26 (ddd, *J* = 12.8, 5.6, 3.4 Hz, 1H), 2.11 (q, *J* = 12.1 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.29, 170.84, 159.85, 150.44, 140.19, 131.73, 129.57, 127.98, 126.56, 121.04, 118.54, 114.38, 112.54, 111.78, 66.93, 63.06, 55.16, 49.39, 46.88, 40.53, 40.41, 35.54, 31.66. **HRMS-EI^{+}** (*m/z*): calc for $C_{26}H_{33}N_3O_4$ $[M]^{+}$ 451.2427, found 451.2423.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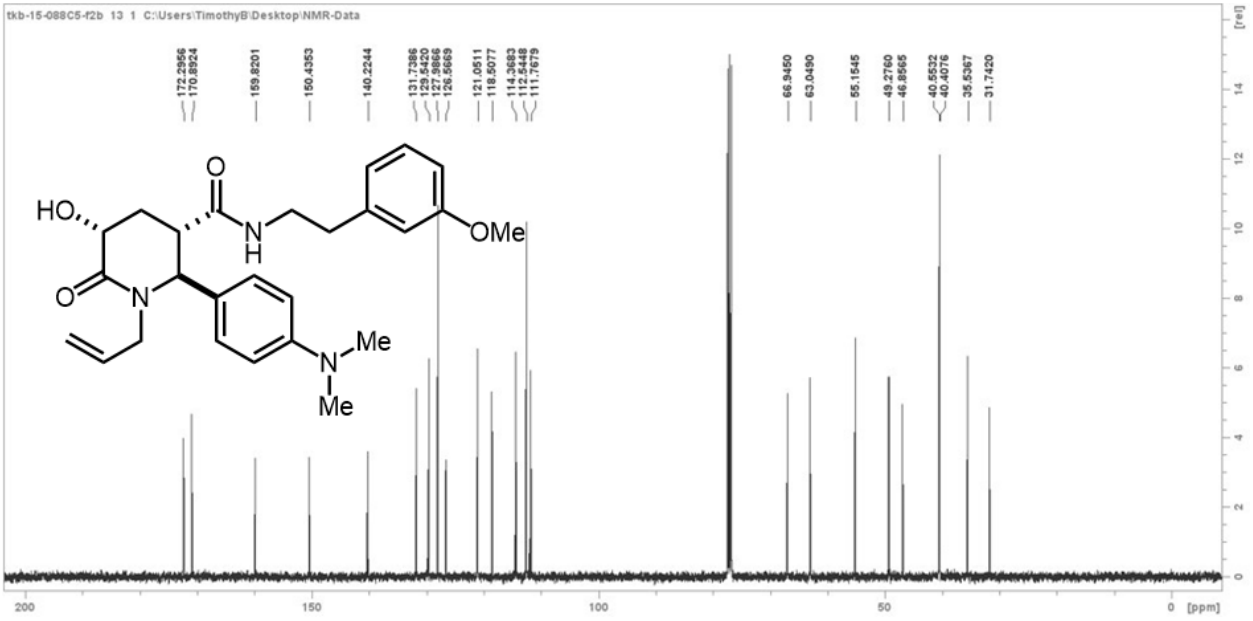
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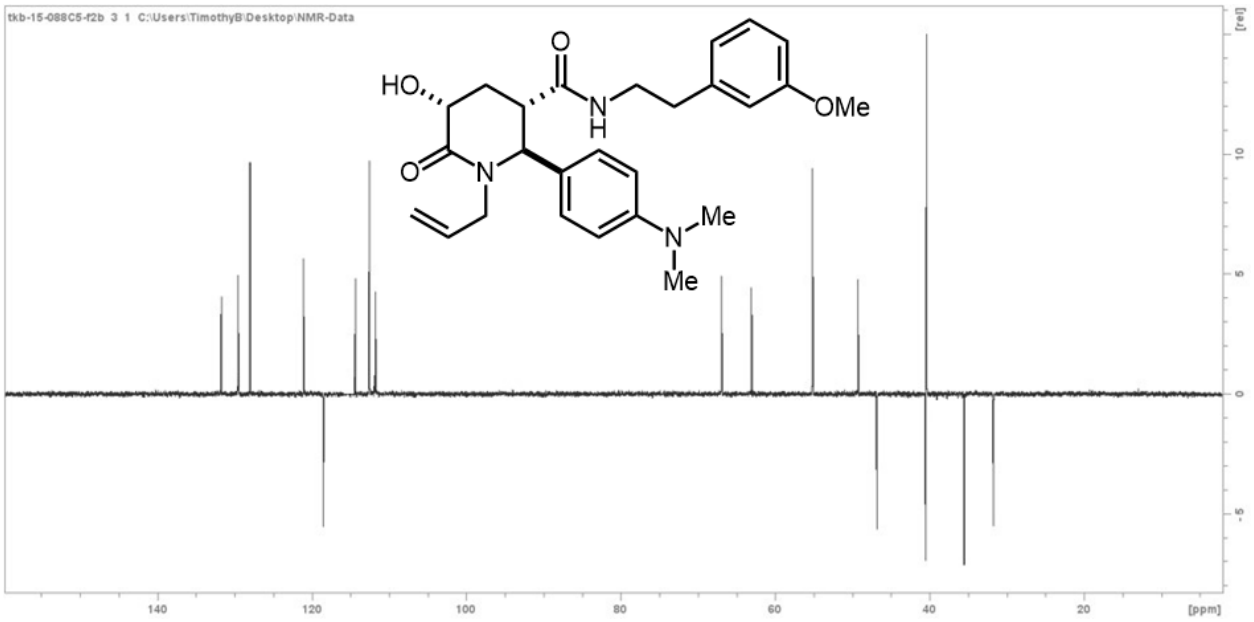
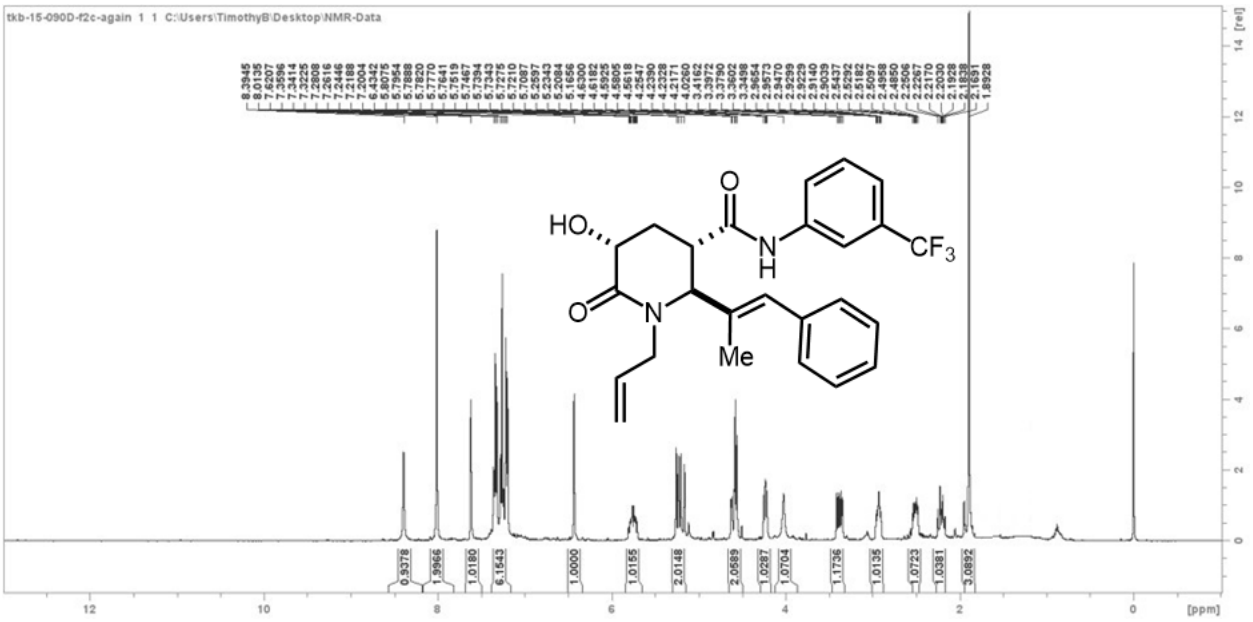


Compound 7z3

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 60:40). Greenish-yellow oil. Yield = 380.5 mg, 83%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 8.39 (s, 1H), 8.01 (s, 2H), 7.62 (s, 1H), 7.39 – 7.18 (m, 6H), 6.43 (s, 1H), 5.76 (dddd, *J* = 17.4, 10.2, 7.5, 4.8 Hz, 1H), 5.25 (d, *J* = 10.1 Hz, 1H), 5.19 (dd, *J* = 17.1, 1.6 Hz, 1H), 4.66 – 4.52 (m, 2H), 4.24 (dd, *J* = 9.0, 6.1 Hz, 1H), 4.03 (s, 1H), 3.44 – 3.33 (m, 1H), 2.99 – 2.88 (m, 1H), 2.59 – 2.45 (m, 1H), 2.21 (dt, *J* = 13.7, 9.5 Hz, 1H), 1.89 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.85, 169.99, 138.86, 135.98, 133.26, 132.65, 132.32, 131.26, 130.41, 128.84, 128.47, 127.53, 124.33, 121.62, 119.76, 119.22, 118.11, 66.76, 66.03, 47.54, 44.49, 29.91, 14.07. **HRMS-EI^{+}** (*m/z*): calc for $C_{25}H_{25}F_3N_2O_3$ $[M]^{+}$ 458.1817, found 458.1823.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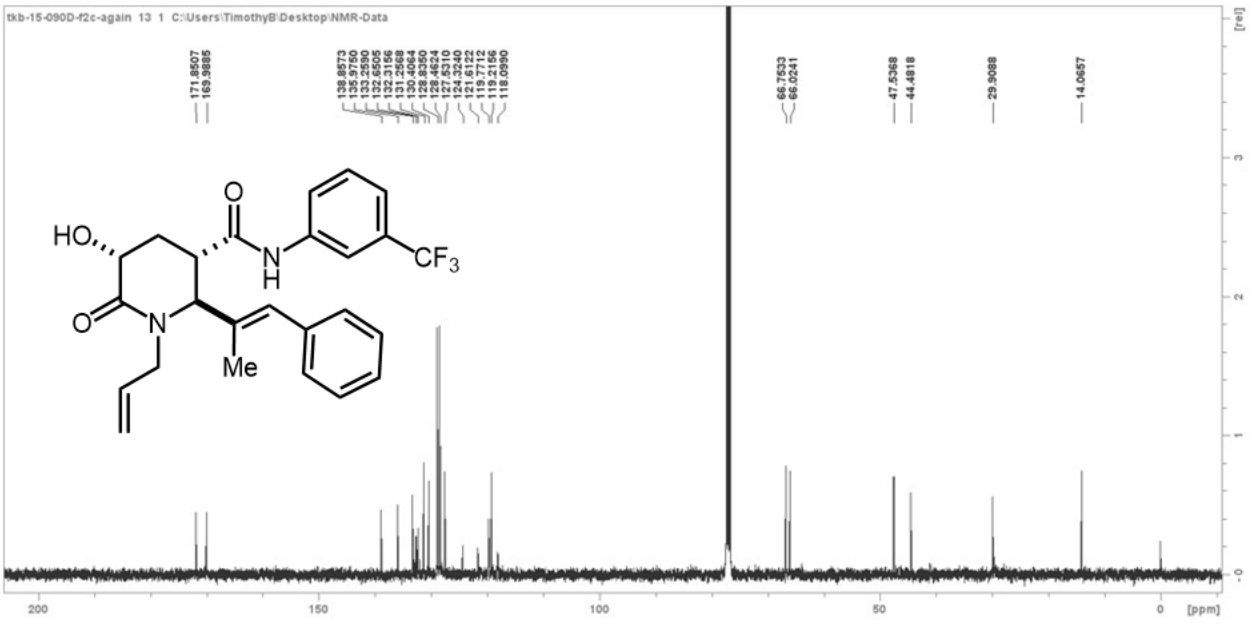
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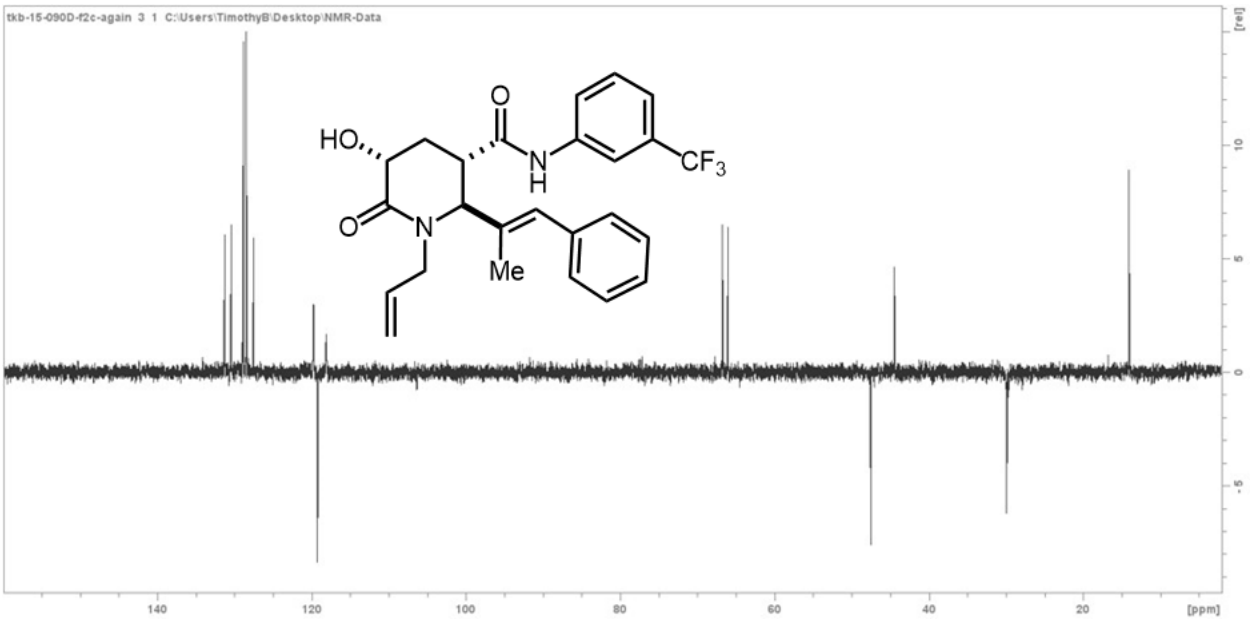
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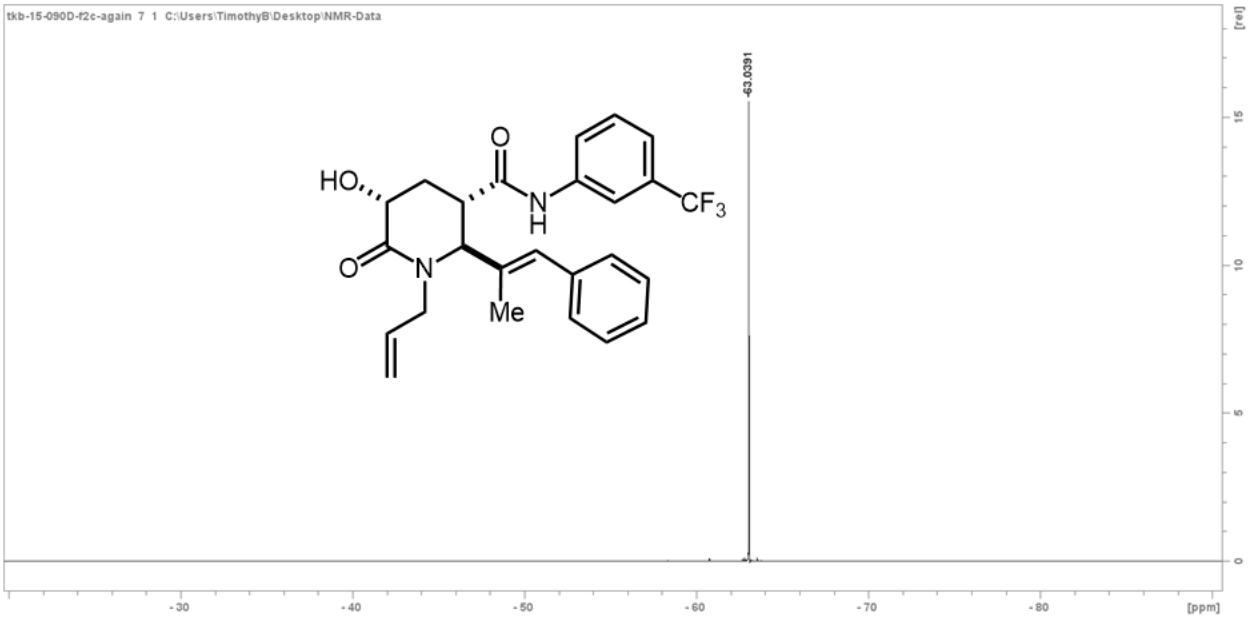
^{19}F NMR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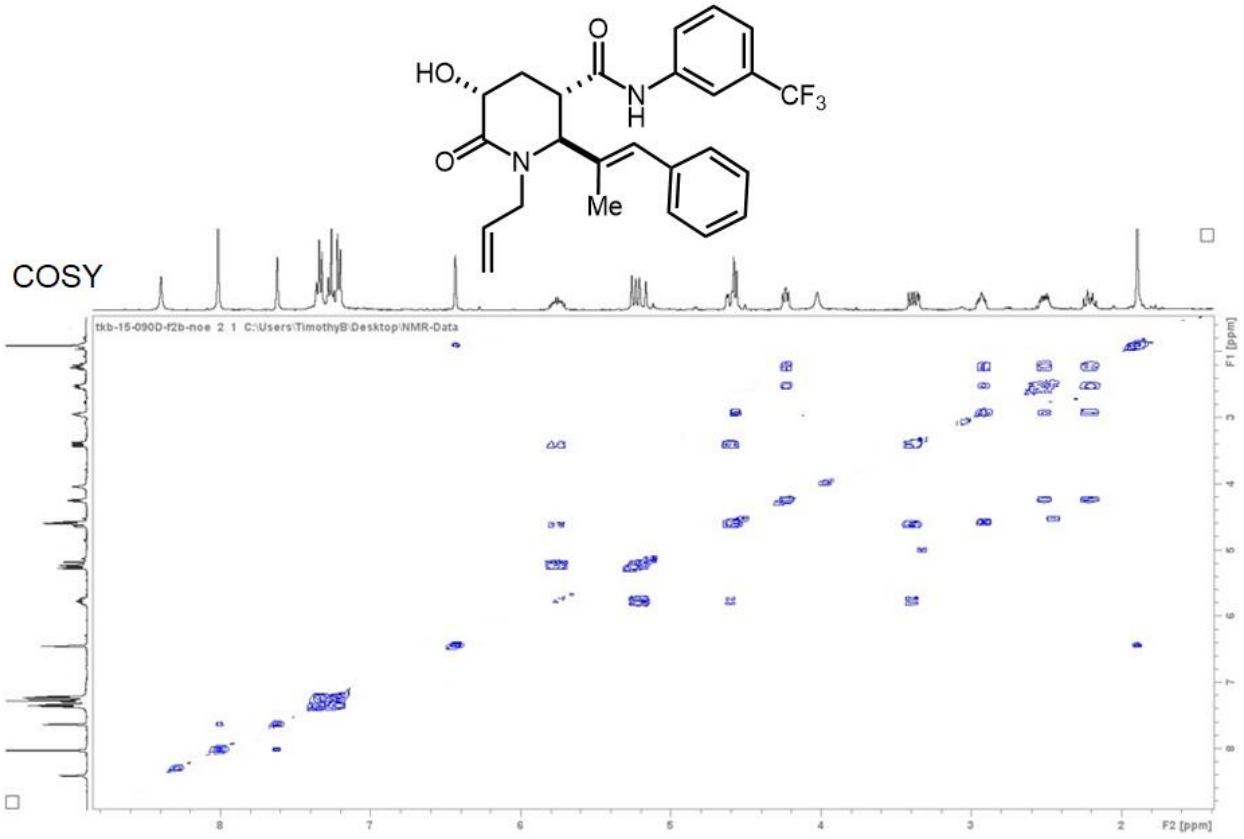
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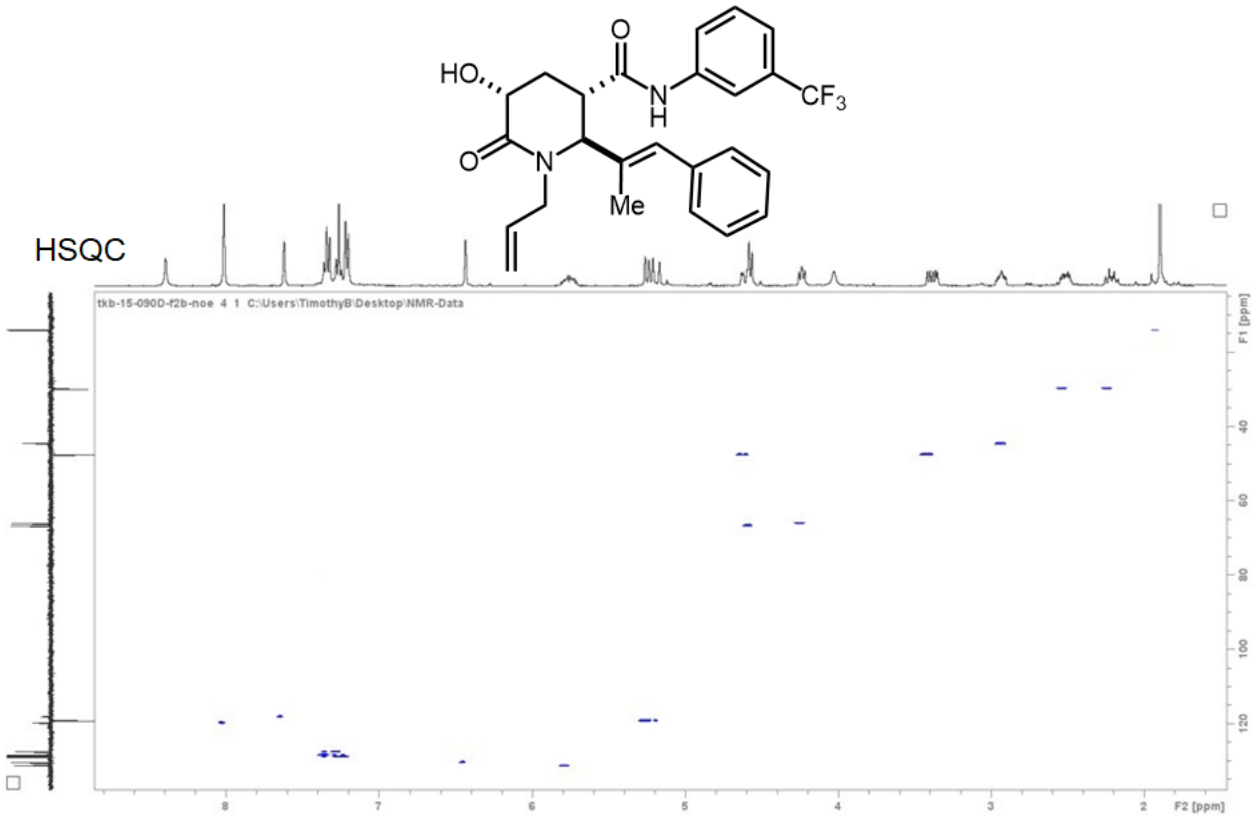
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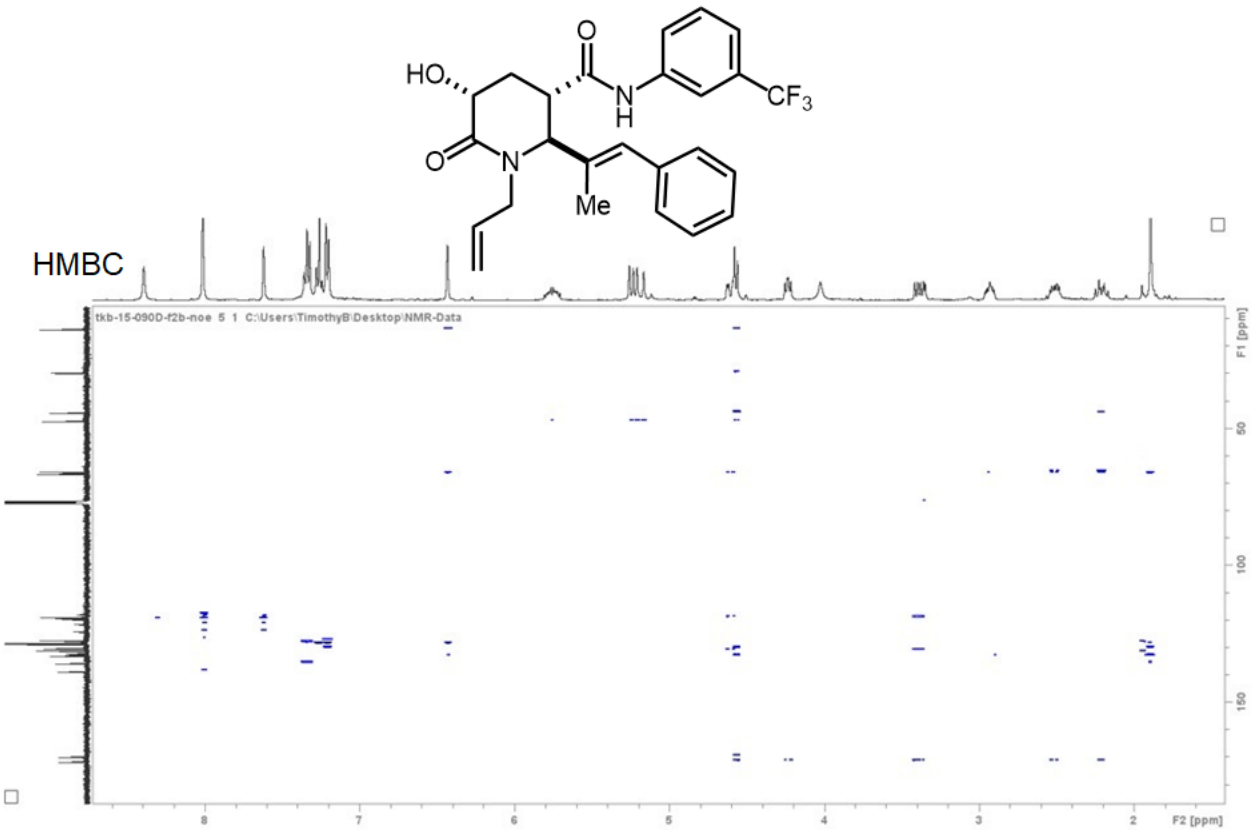
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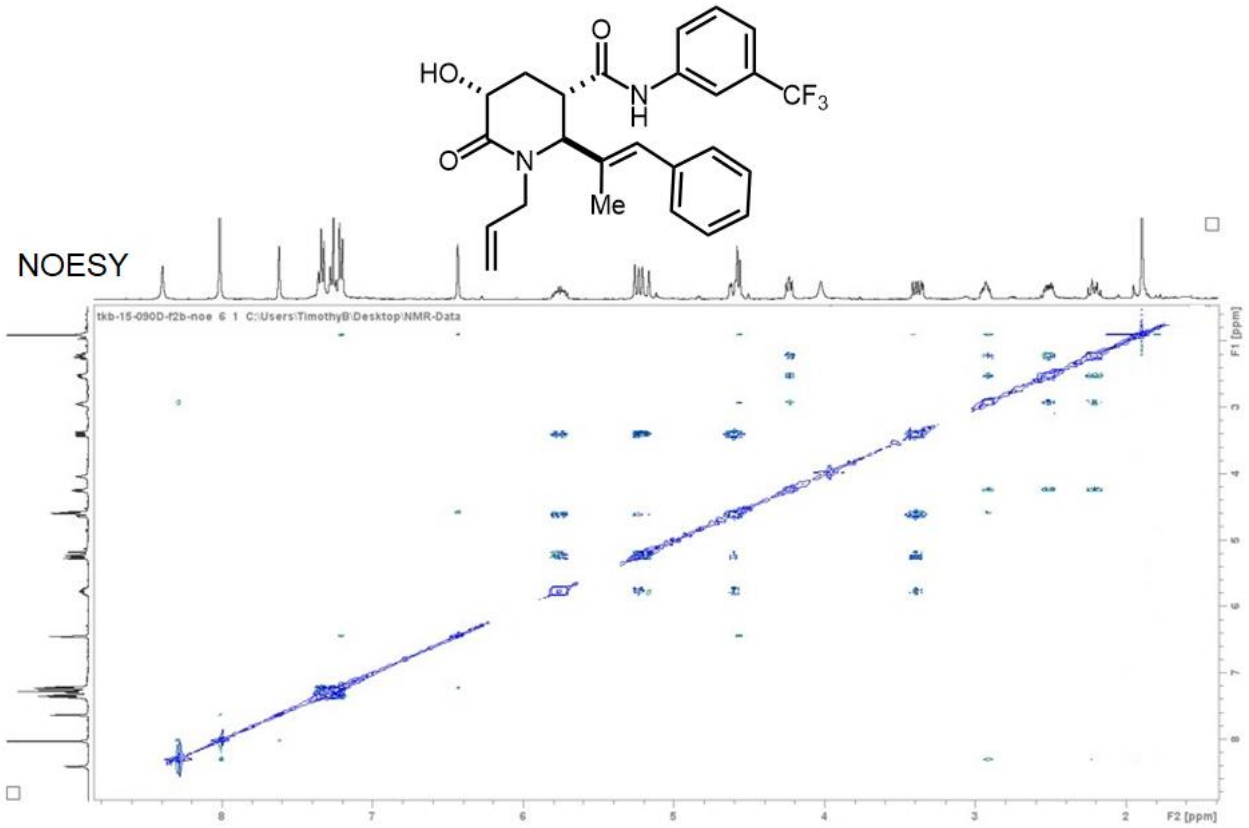
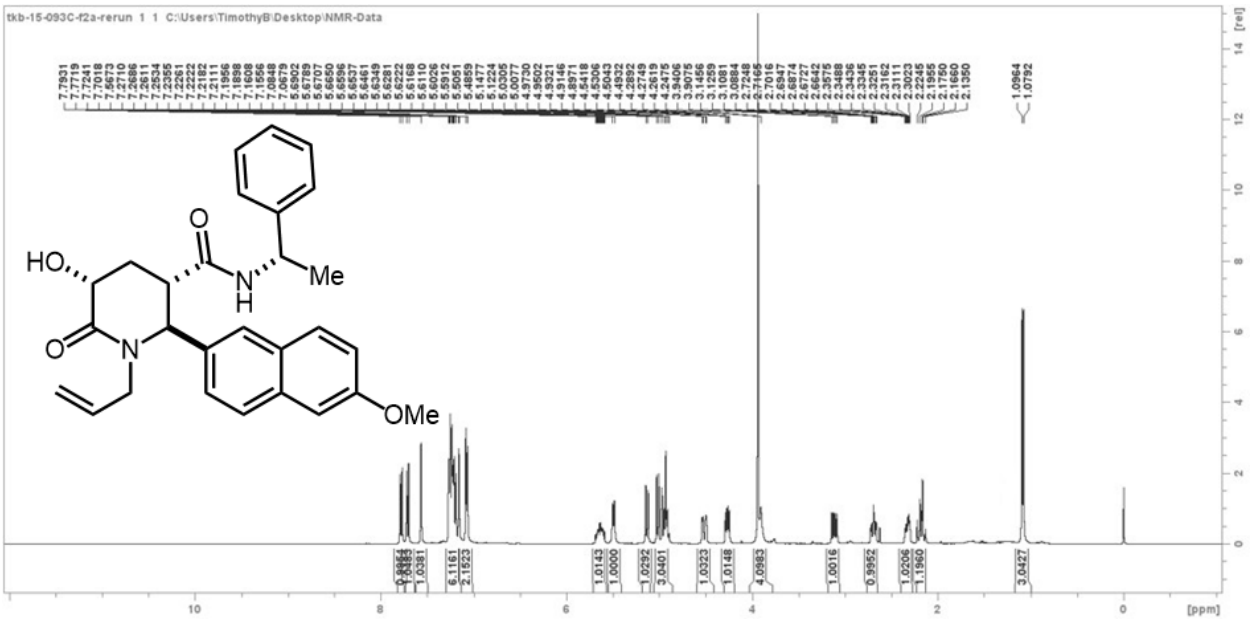


Compound 7z4

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 50:50). Greenish-yellow oil. Yield = 417.2 mg, 91%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.78 (d, *J* = 8.5 Hz, 1H), 7.71 (d, *J* = 8.9 Hz, 1H), 7.57 (d, *J* = 1.9 Hz, 1H), 7.23 (dddd, *J* = 16.2, 11.4, 6.0, 2.1 Hz, 5H), 7.16 (d, *J* = 2.5 Hz, 1H), 7.11 – 7.04 (m, 2H), 5.64 (dddd, *J* = 17.6, 10.2, 7.8, 4.5 Hz, 1H), 5.50 (d, *J* = 7.8 Hz, 1H), 5.17 – 5.10 (m, 1H), 5.02 (d, *J* = 9.1 Hz, 1H), 4.99 – 4.87 (m, 2H), 4.57 – 4.47 (m, 1H), 4.27 (dd, *J* = 11.0, 5.7 Hz, 1H), 3.94 (s, 3H), 3.91 (s, 1H), 3.12 (dd, *J* = 15.0, 7.9 Hz, 1H), 2.75 – 2.61 (m, 1H), 2.33 (ddd, *J* = 13.0, 5.8, 3.5 Hz, 1H), 2.25 – 2.11 (m, 1H), 1.09 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.44, 169.59, 158.29, 142.38, 134.44, 134.29, 131.50, 129.37, 128.70, 128.62, 128.21, 127.53, 126.60, 126.00, 124.35, 119.71, 118.87, 105.79, 66.90, 63.59, 55.40, 49.09, 49.03, 47.27, 31.36, 21.11. **HRMS-EI^{+}** (*m/z*): calc for $C_{28}H_{30}N_2O_4$ $[M]^{+}$ 458.2206, found 458.2211.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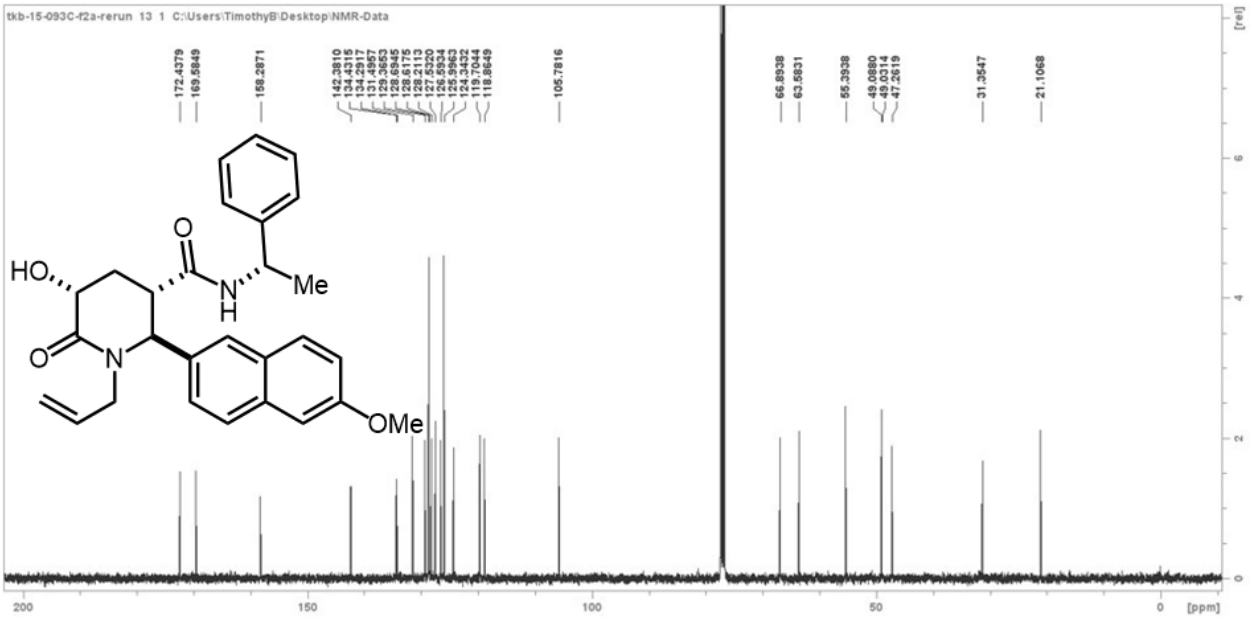
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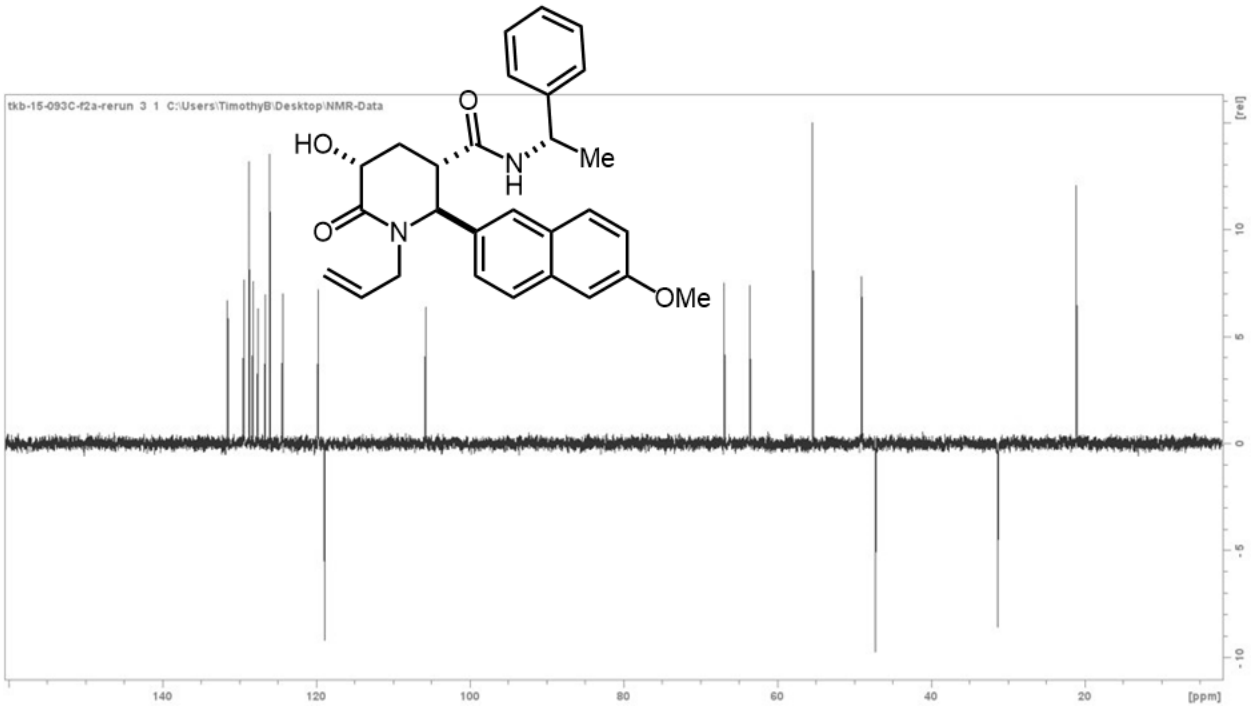
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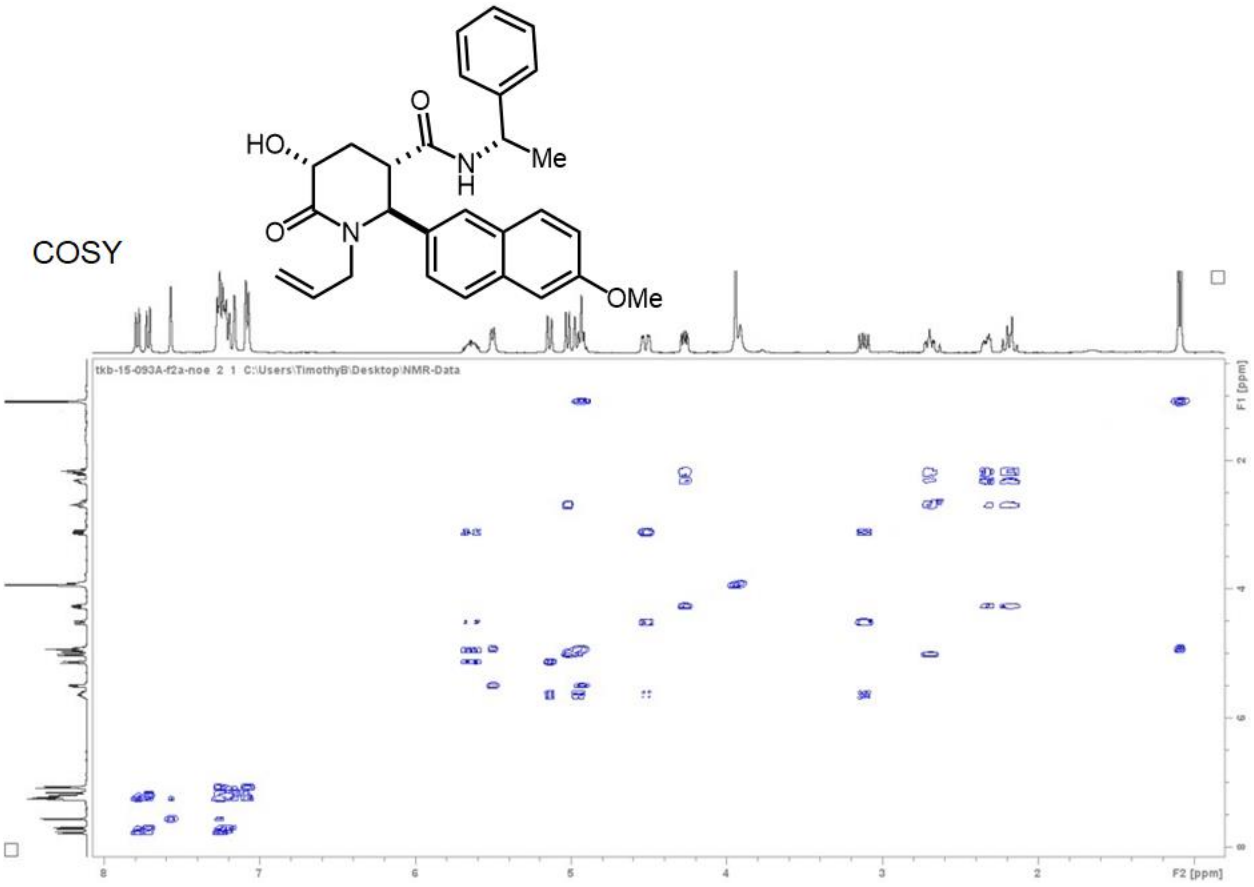
COSY
HO
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N
OMe
tkb-15-093A-f2a-noe 2 1 C:\Users\TimothyB\Desktop\NMR-Data
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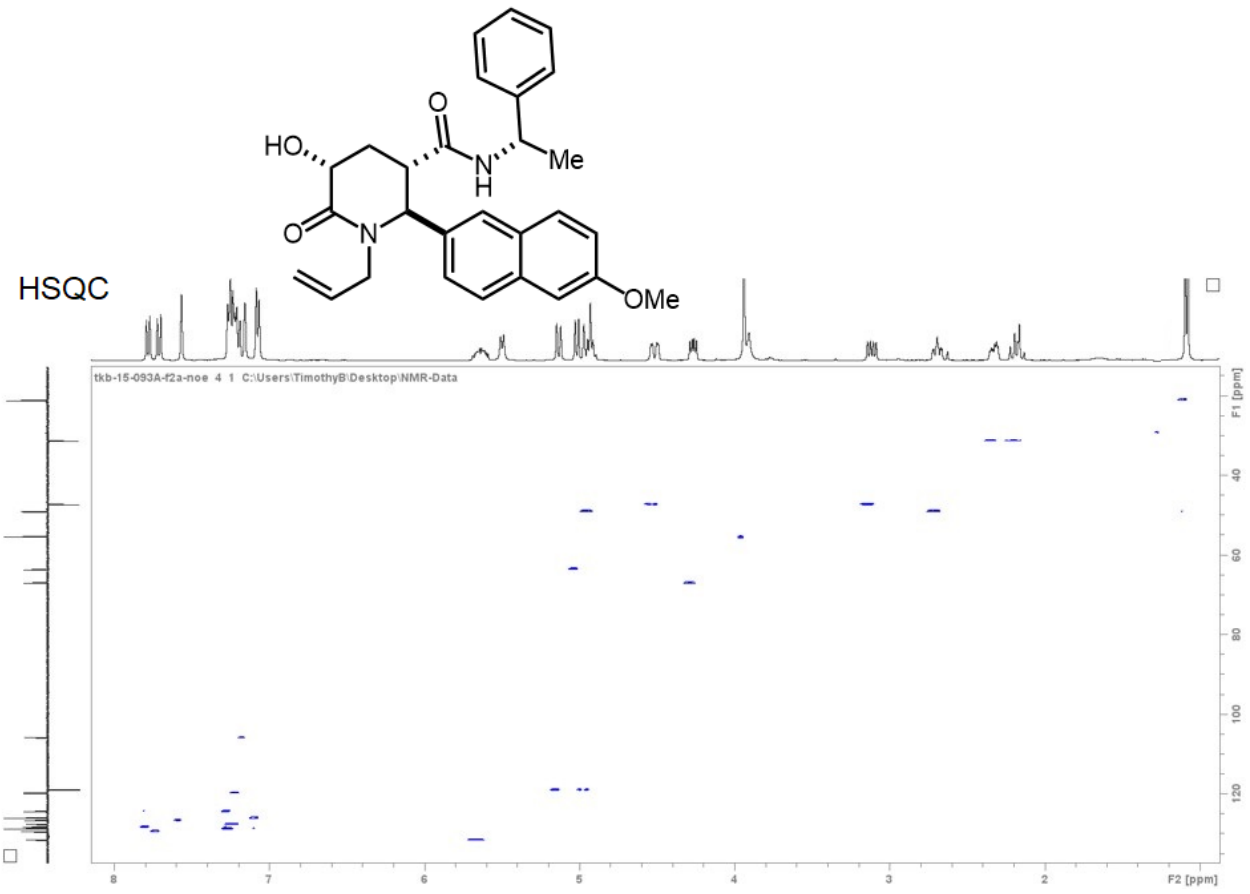
4
6
8
8
7
6
5
4
3
2
F2 [ppm]


HSQC
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H
Me
O
N
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tkb-15-093A-f2a-noe 4 1 C:\Users\TimothyB\Desktop\NMR-Data
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80
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7
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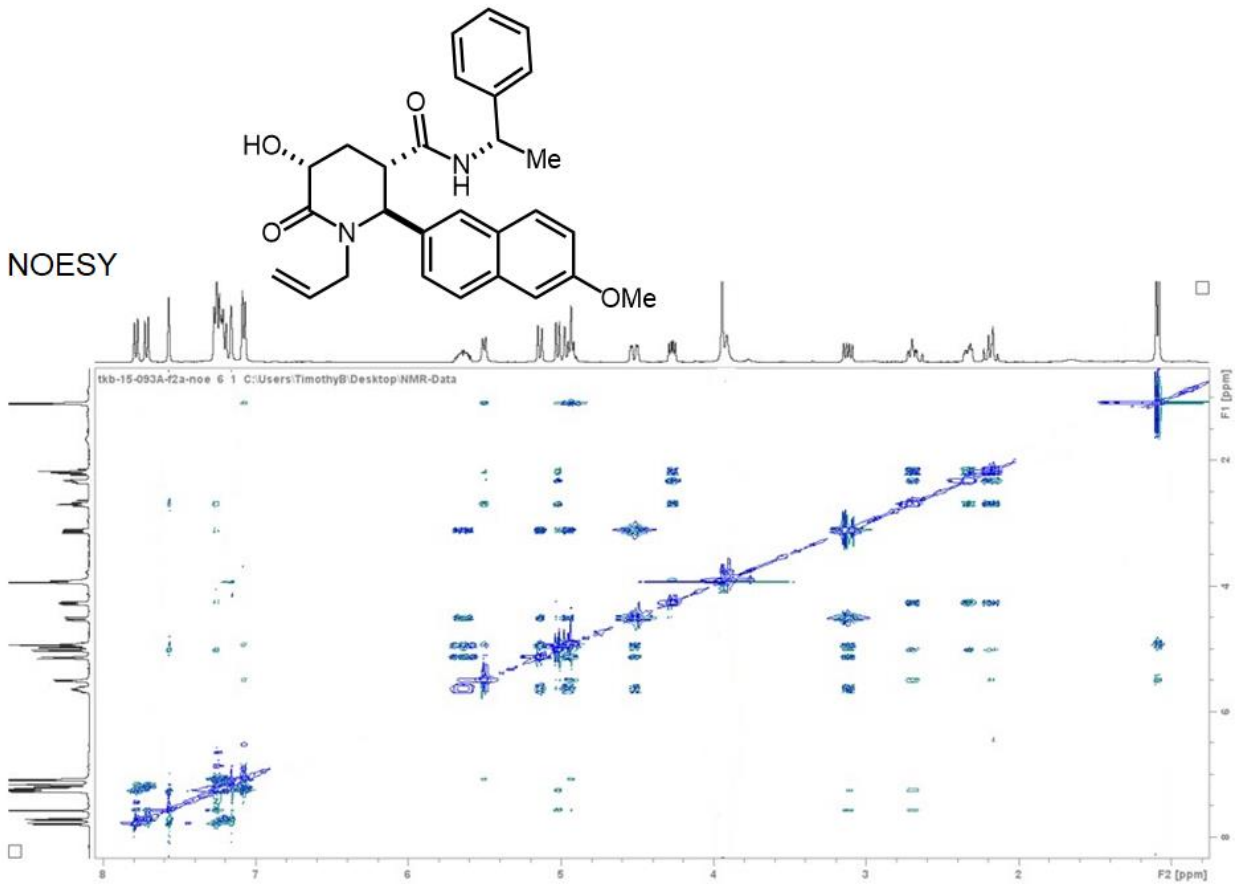
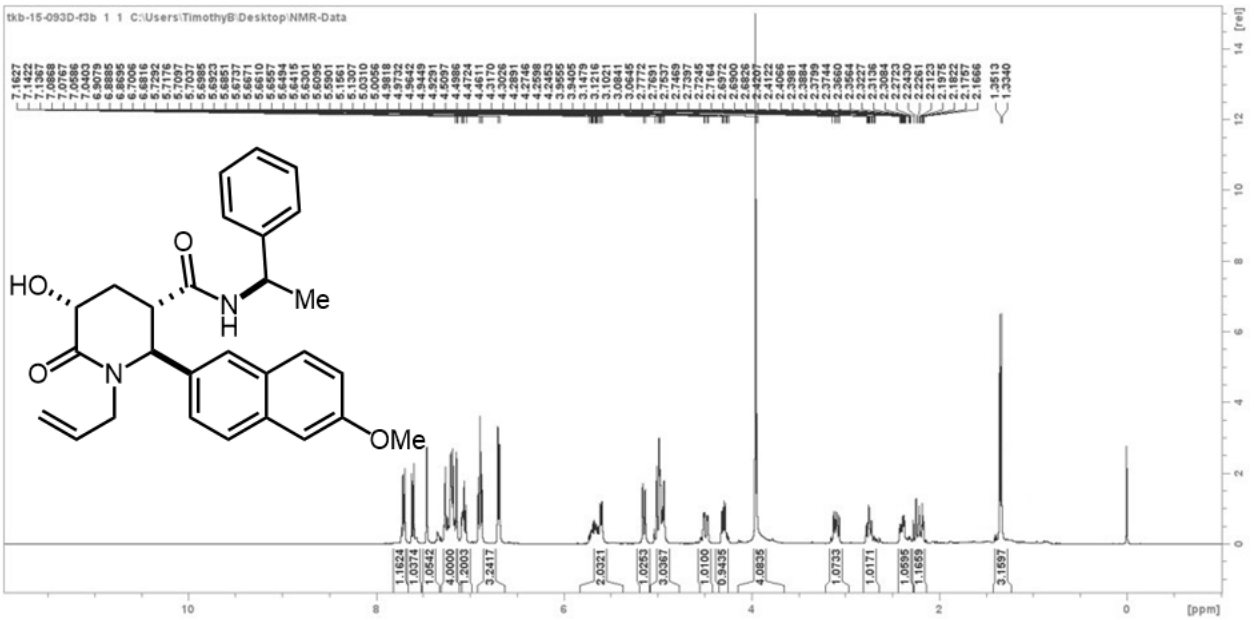
3
2
F2 [ppm]



Compound 7z5

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 50:50). Greenish-yellow oil. Yield = 412.6 mg, 90%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.71 (dd, *J* = 8.8, 5.6 Hz, 1H), 7.64 – 7.54 (m, 1H), 7.46 (d, *J* = 1.8 Hz, 1H), 7.38 – 7.22 (m, 1H), 7.25 – 7.10 (m, 4H), 7.07 (dt, *J* = 12.2, 4.7 Hz, 1H), 6.89 (t, *J* = 7.7 Hz, 1H), 6.69 (d, *J* = 7.6 Hz, 1H), 5.75 – 5.57 (m, 2H), 5.18 – 5.11 (m, 1H), 5.05 – 4.89 (m, 3H), 4.49 (ddt, *J* = 15.0, 3.7, 1.7 Hz, 1H), 4.28 (td, *J* = 11.4, 5.7 Hz, 1H), 3.95– 3.90 (m, 4H), 3.09 (dd, *J* = 15.1, 7.9 Hz, 1H), 2.80 – 2.61 (m, 1H), 2.39 (ddd, *J* = 12.9, 5.8, 3.4 Hz, 1H), 2.33 – 2.14 (m, 1H), 1.34 (d, *J* = 6.9 Hz, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.43, 169.65, 158.29, 142.18, 134.44, 133.98, 131.50, 129.65, 128.64, 128.32, 128.27, 127.08, 126.79, 125.61, 124.26, 119.53, 118.82, 105.76, 66.97, 63.65, 55.44, 49.20, 48.76, 47.19, 31.62, 21.55. **HRMS-EI^{+}** (*m/z*): calc for $C_{28}H_{30}N_2O_4$ $[M]^{+}$ 458.2206, found 458.2211.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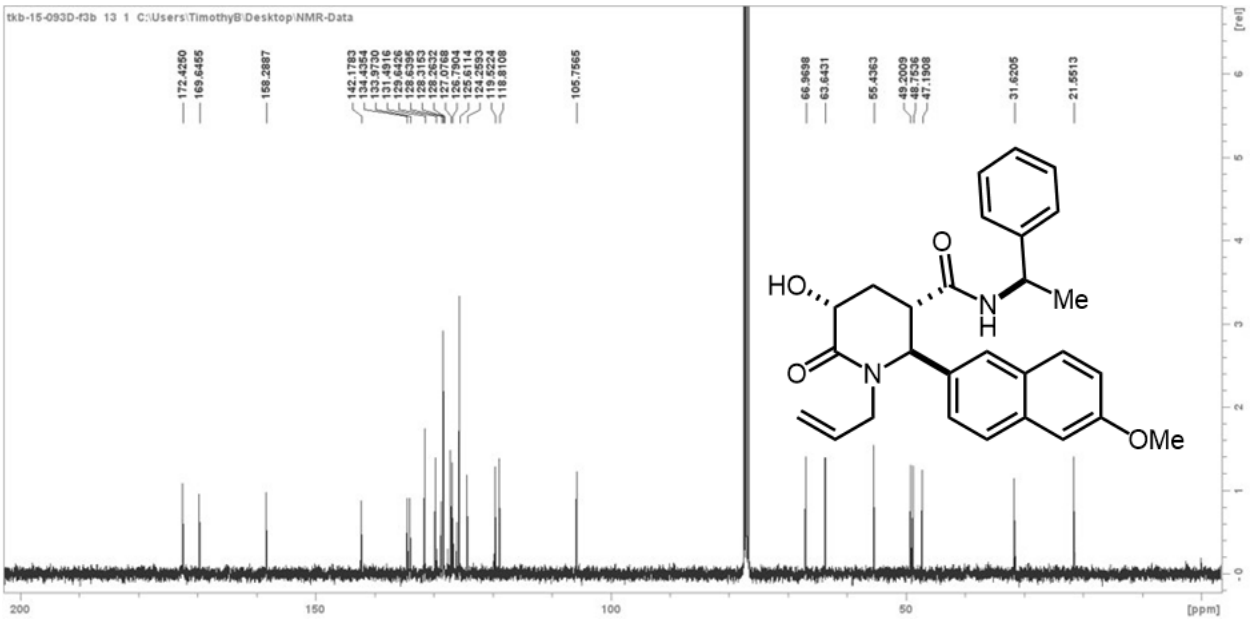
H
Me
O
N
OMe
[ppm]


tkb-15-093D-f3b 13 1 C:\Users\TimothyB\Desktop\NMR-Data
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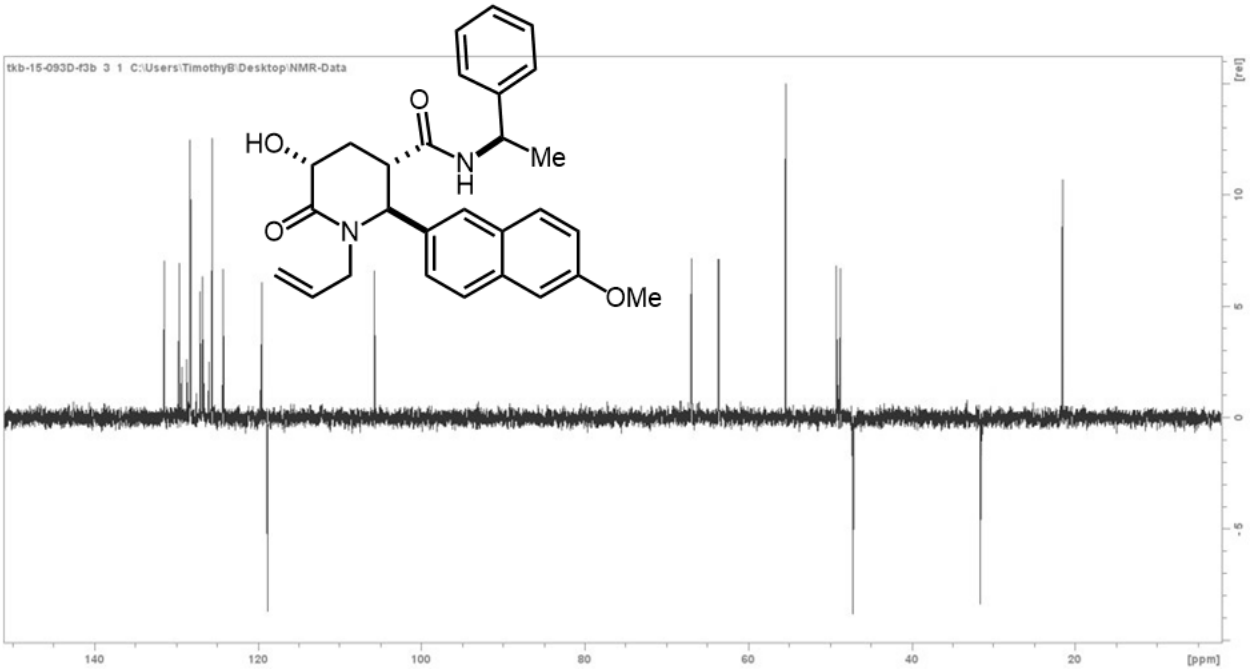
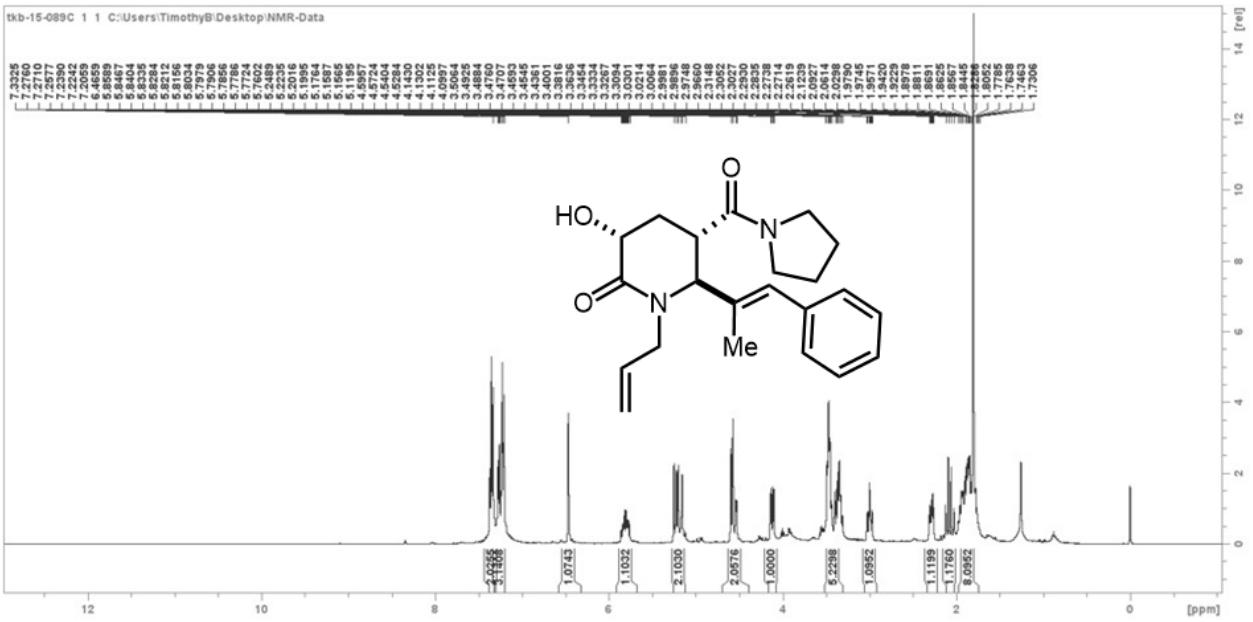
N
H
Me
O
N
OMe
[ppm]



Compound 7z6

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 50:50). Yellowish oil. Yield = 350.1 mg, 95%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.32 – 7.20 (m, 5H), 6.47 (s, 1H), 5.81 (dddd, *J* = 17.3, 10.2, 7.4, 4.8 Hz, 1H), 5.22 – 5.10 (m, 1H), 4.62 – 4.46 (m, 2H), 4.12 (dd, *J* = 12.2, 5.2 Hz, 1H), 3.41 – 3.29 (m, 5H), 3.00 (ddd, *J* = 12.9, 9.4, 3.5 Hz, 1H), 2.29 (ddd, *J* = 12.5, 5.2, 3.6 Hz, 1H), 2.21 – 1.98 (m, 1H), 1.87 – 1.71 (m, 8H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.54, 169.64, 136.54, 133.79, 131.73, 130.54, 128.78, 128.37, 127.20, 118.63, 67.41, 66.92, 46.88, 46.67, 46.17, 40.99, 31.23, 26.09, 24.24, 13.35. **HRMS-EI$^+$** (*m/z*): calc for $C_{22}H_{28}N_2O_3$ $[M]^+$ 368.2100, found 368.2105.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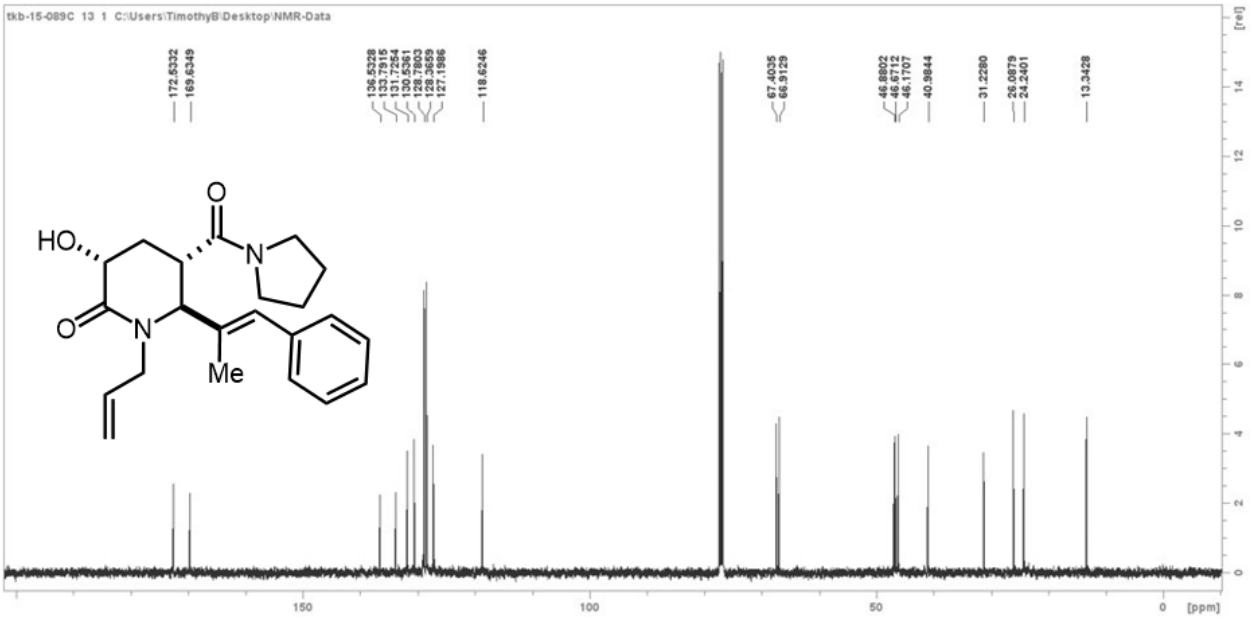
N
Me
[ppm]


tkb-15-089C 13 1 C:\Users\TimothyB\Desktop\NMR-Data
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46.8802
46.6712
46.1707
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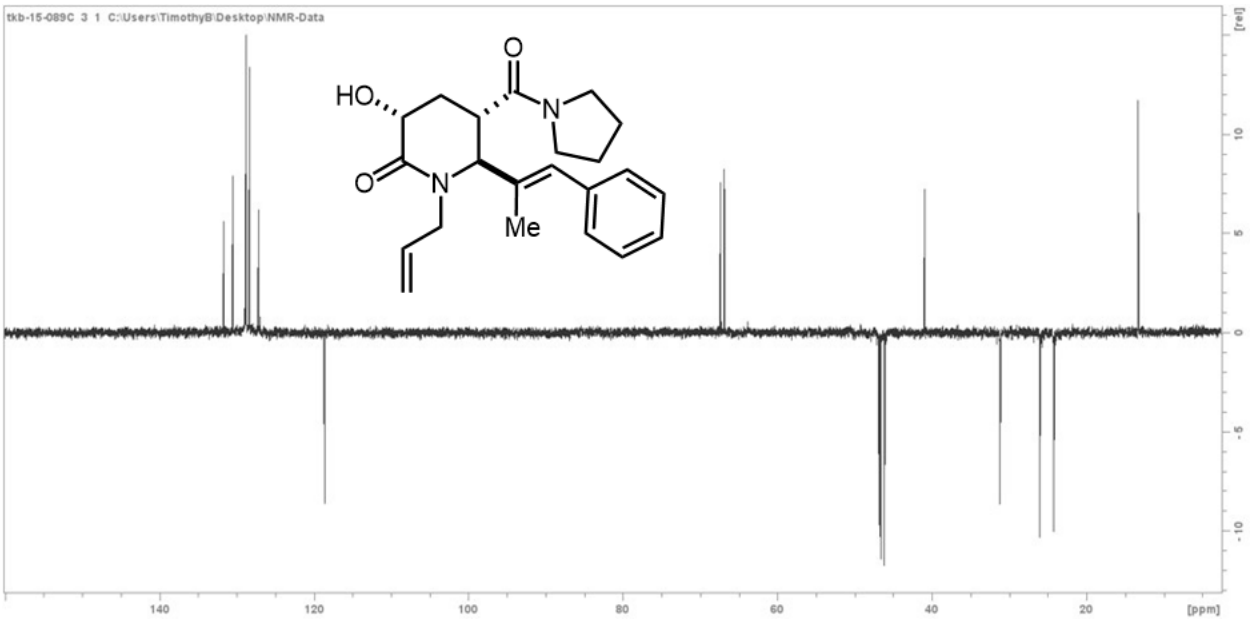
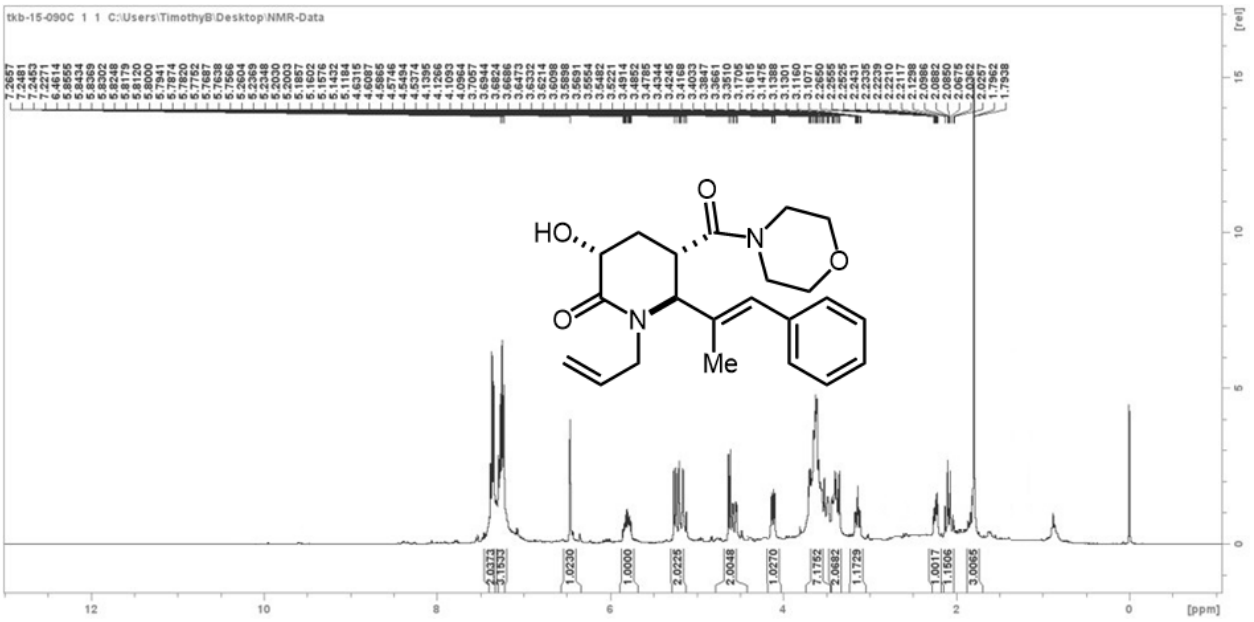
N
O
N
Me
[ppm]



Compound 7z7

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 50:50). Yellowish oil. Yield = 349.9 mg, 91%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.35 – 7.22 (m, 5H), 6.46 (s, 1H), 5.80 (dddt, *J* = 18.4, 10.9, 7.8, 5.2 Hz, 1H), 5.29 – 5.09 (m, 2H), 4.65 – 4.51 (m, 2H), 4.12 (dd, *J* = 12.1, 5.2 Hz, 1H), 3.73 – 3.45 (m, 7H), 3.48 – 3.33 (m, 2H), 3.14 (ddd, *J* = 12.7, 9.2, 3.6 Hz, 1H), 2.24 (ddd, *J* = 12.7, 5.2, 3.6 Hz, 1H), 2.08 (q, *J* = 12.5 Hz, 1H), 1.79 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.40, 169.97, 136.29, 133.32, 131.69, 130.99, 128.84, 128.47, 127.39, 118.74, 67.44, 67.03, 66.91, 66.73, 46.92, 46.39, 42.69, 37.81, 31.68, 13.34. **HRMS-EI$^+$** (*m/z*): calc for $C_{22}H_{28}N_2O_4$ $[M]^+$ 384.2049, found 384.2053.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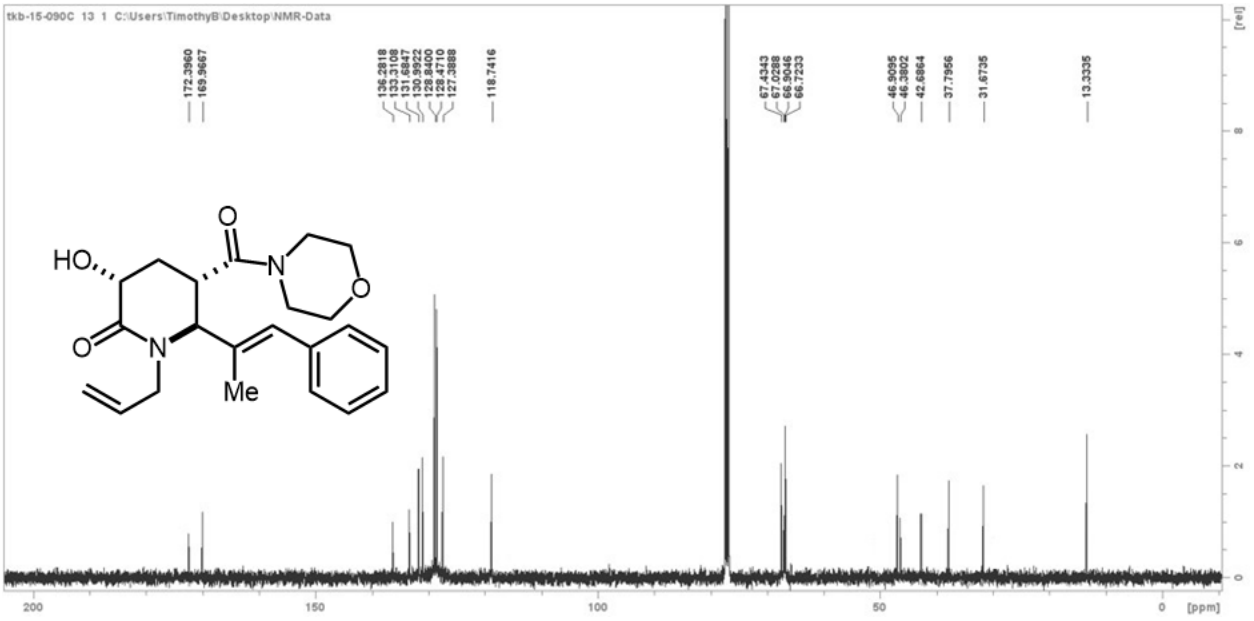
Me
[ppm]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128.4710
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67.0088
66.9046
66.7233
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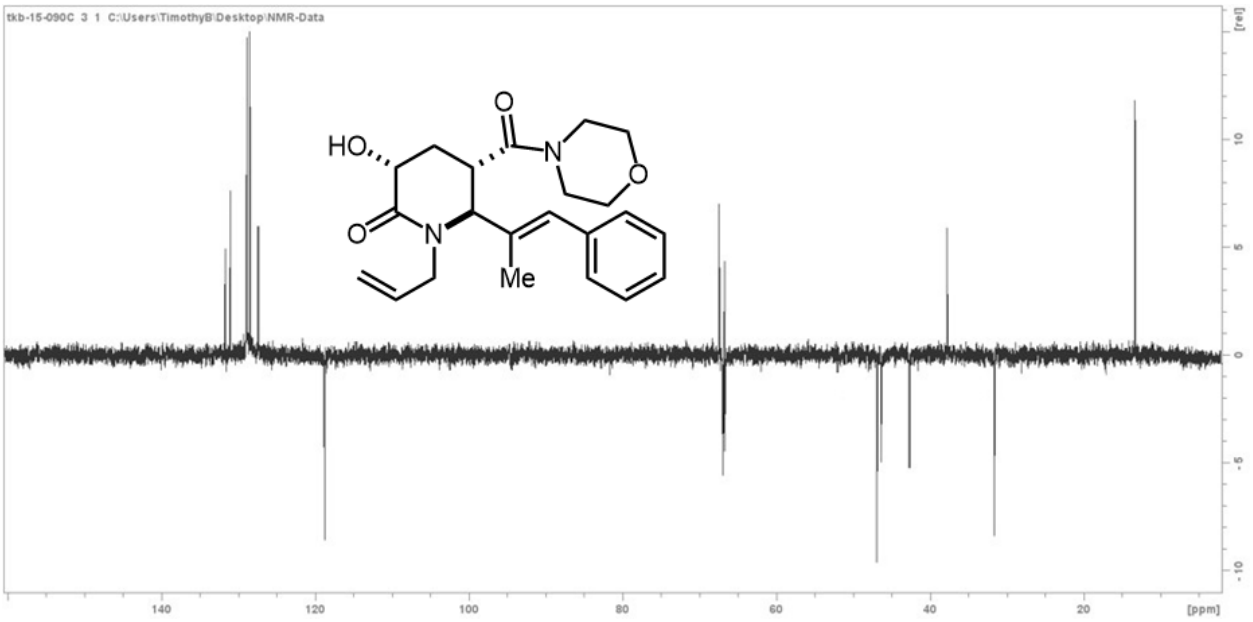
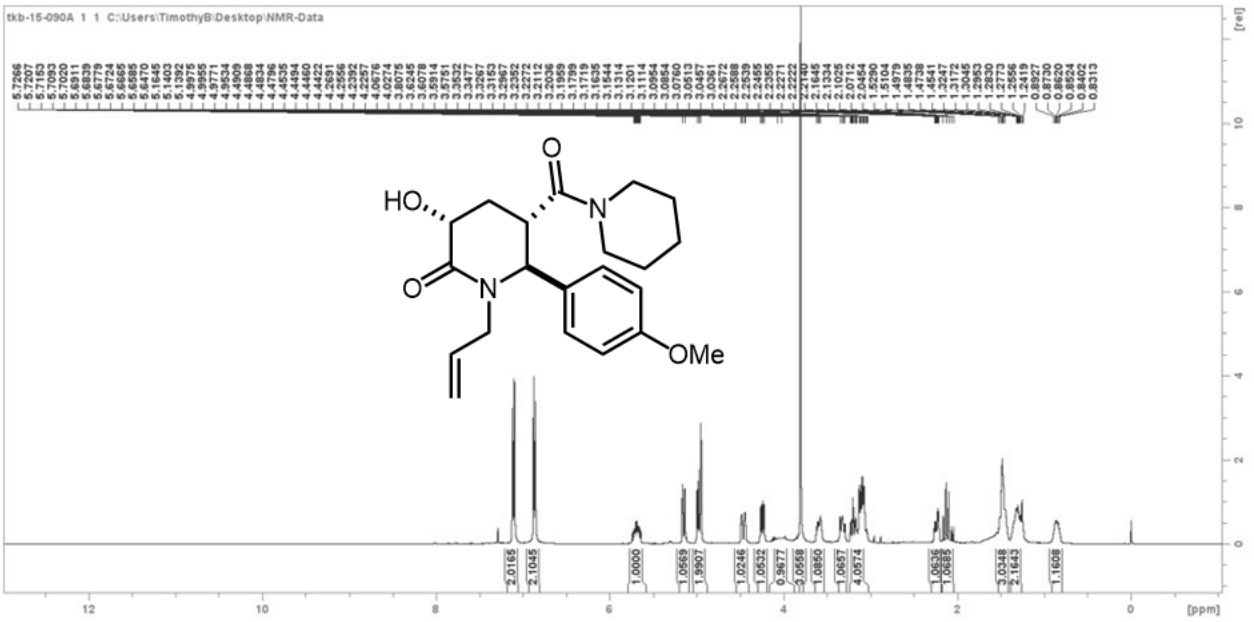
Me
[ppm]



Compound 7z8

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 40:60). Yellowish oil. Yield = 342.7 mg, 92%, >99:1 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.15 (d, *J* = 7.5 Hz, 2H), 6.83 (d, *J* = 7.5 Hz, 2H), 5.70 (dddd, *J* = 17.6, 10.2, 7.8, 4.6 Hz, 1H), 5.15 (dt, *J* = 10.2, 1.3 Hz, 1H), 5.02 – 4.93 (m, 2H), 4.47 (ddt, *J* = 15.0, 4.7, 1.7 Hz, 1H), 4.25 (dd, *J* = 12.0, 5.4 Hz, 1H), 4.03 (s, 1H), 3.81 (s, 3H), 3.59 (ddd, *J* = 13.1, 6.6, 3.9 Hz, 1H), 3.32 (ddd, *J* = 13.0, 8.0, 2.5 Hz, 1H), 3.20 (ddd, *J* = 12.7, 9.5, 3.3 Hz, 1H), 3.19 – 3.06 (m, 2H), 3.10 – 3.01 (m, 1H), 2.24 (ddd, *J* = 12.7, 5.4, 3.3 Hz, 1H), 2.19 – 2.02 (m, 1H), 1.48 (dtd, *J* = 11.0, 7.2, 2.9 Hz, 3H), 1.39 – 1.20 (m, 3H), 0.86 (ddt, *J* = 12.0, 7.6, 4.1 Hz, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.49, 169.07, 159.51, 131.73, 128.45, 118.56, 114.32, 67.19, 63.38, 55.35, 46.97, 46.78, 43.38, 43.11, 32.10, 26.31, 25.53, 24.34. **HRMS-EI$^+$** (*m/z*): calc for $C_{21}H_{28}N_2O_4$ $[M]^+$ 372.2049, found 372.2055.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OMe
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1.9907
1.0246
1.0532
0.9677
3.0558
1.0850
1.0657
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1.0685
3.0348
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10
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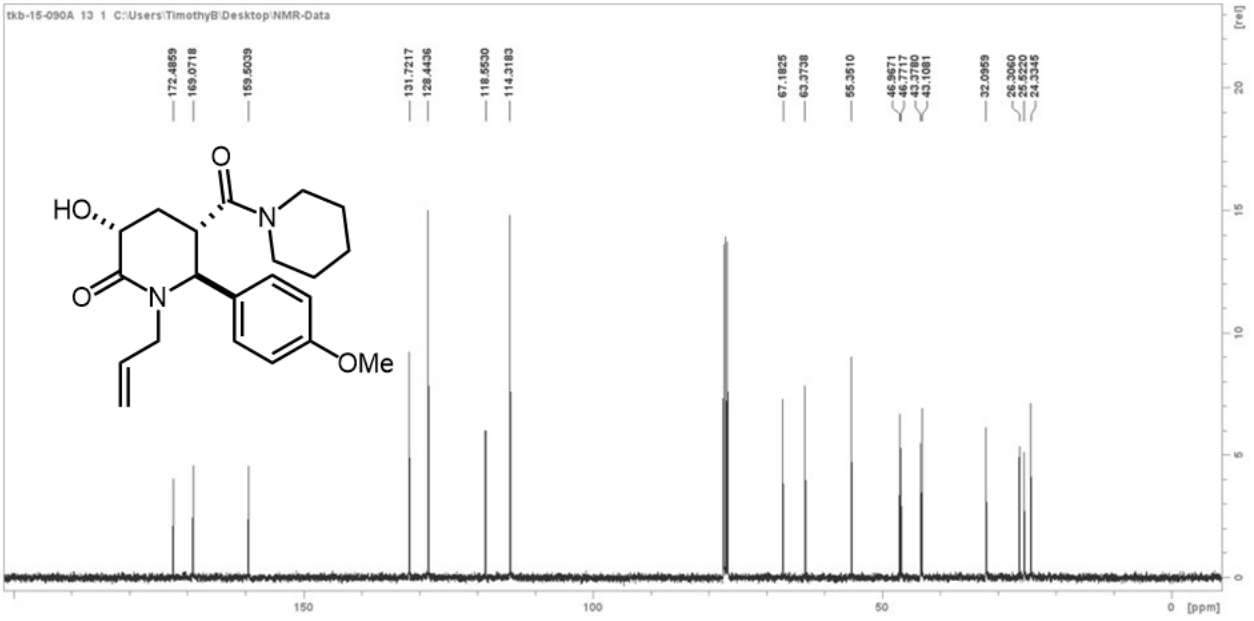
[ppm]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114.3183
67.1825
63.3738
55.3510
46.9671
45.7717
45.3780
43.1081
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25.5220
24.3345
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O
N
OMe
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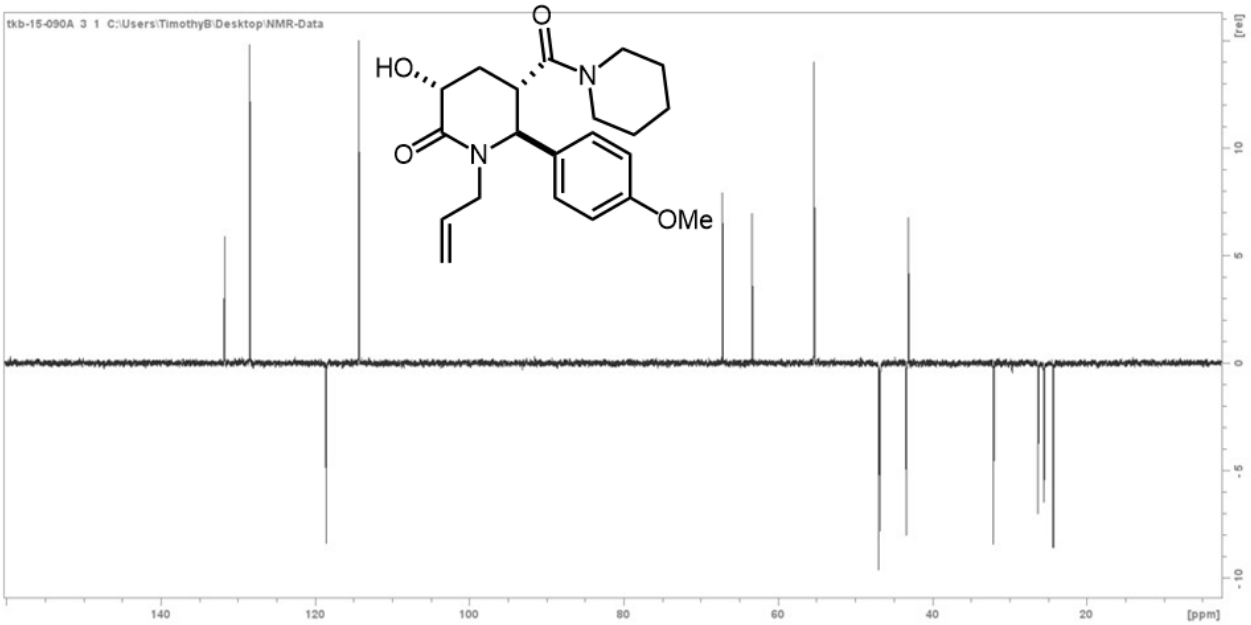
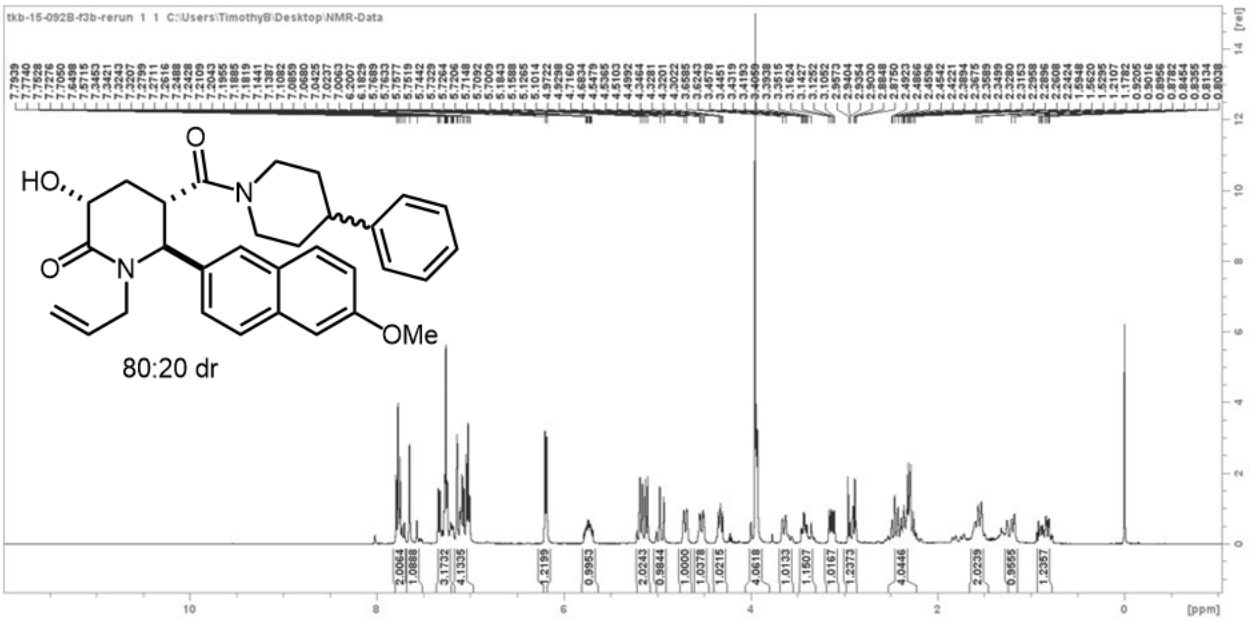
50
0
[ppm]



Compound 7z9

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 40:60). Yellowish oil. Yield = 438.8 mg, 92%, 80:20 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.82 – 7.67 (m, 2H), 7.65 (d, *J* = 1.8 Hz, 1H), 7.37 – 6.98 (m, 7H), 6.19 (dd, *J* = 7.0, 1.8 Hz, 1H), 5.74 (dddd, *J* = 17.6, 10.3, 7.9, 4.6 Hz, 1H), 5.24 – 5.08 (m, 2H), 5.03 – 4.91 (m, 1H), 4.70 (ddt, *J* = 13.2, 4.5, 2.3 Hz, 1H), 4.53 (dt, *J* = 15.0, 3.1 Hz, 1H), 4.32 (dd, *J* = 10.9, 6.9 Hz, 1H), 3.97 – 3.91 (m, 3H), 3.61 (dd, *J* = 26.0, 13.4 Hz, 1H), 3.48 – 3.29 (m, 1H), 3.13 (dd, *J* = 15.0, 7.9 Hz, 1H), 2.98 – 2.85 (m, 1H), 2.58 – 2.15 (m, 4H), 1.55 (dd, *J* = 13.2, 3.7 Hz, 1H), 1.19 (d, *J* = 13.6 Hz, 1H), 0.96 – 0.75 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$, major diastereomer) δ 172.72, 169.03, 158.45, 144.46, 134.58, 134.14, 131.61, 129.48, 128.62, 128.36, 128.19, 127.42, 126.62, 126.34, 126.27, 124.19, 120.02, 118.81, 105.93, 67.47, 64.92, 55.33, 47.07, 46.83, 43.18, 42.80, 42.55, 33.41, 32.68, 31.97. **HRMS-EI^{+}** (*m/z*): calc for $C_{31}H_{34}N_2O_4$ $[M]^{+}$ 498.2519, found 498.2524.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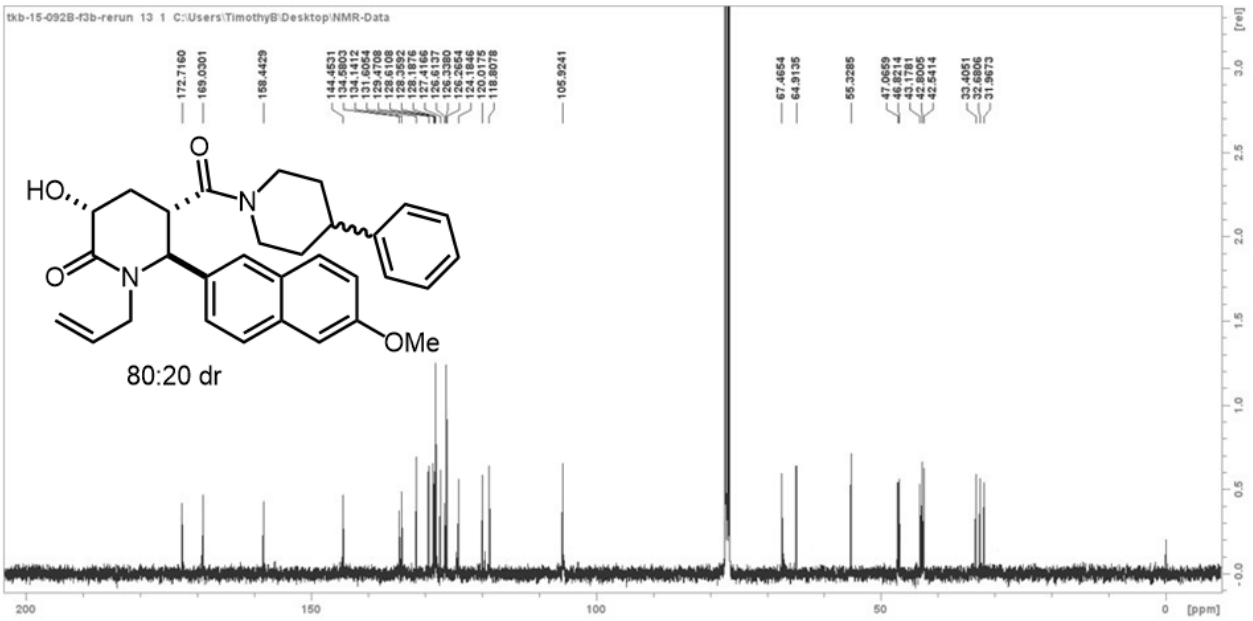
N
O
N
OMe
80:20 dr
[ppm]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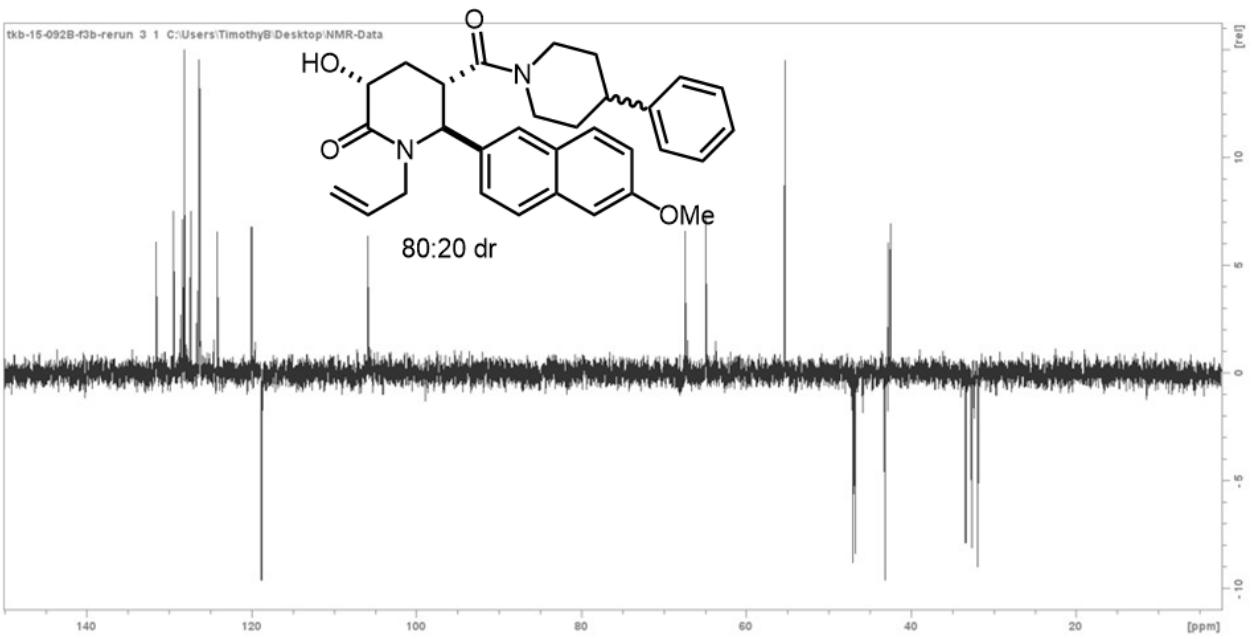
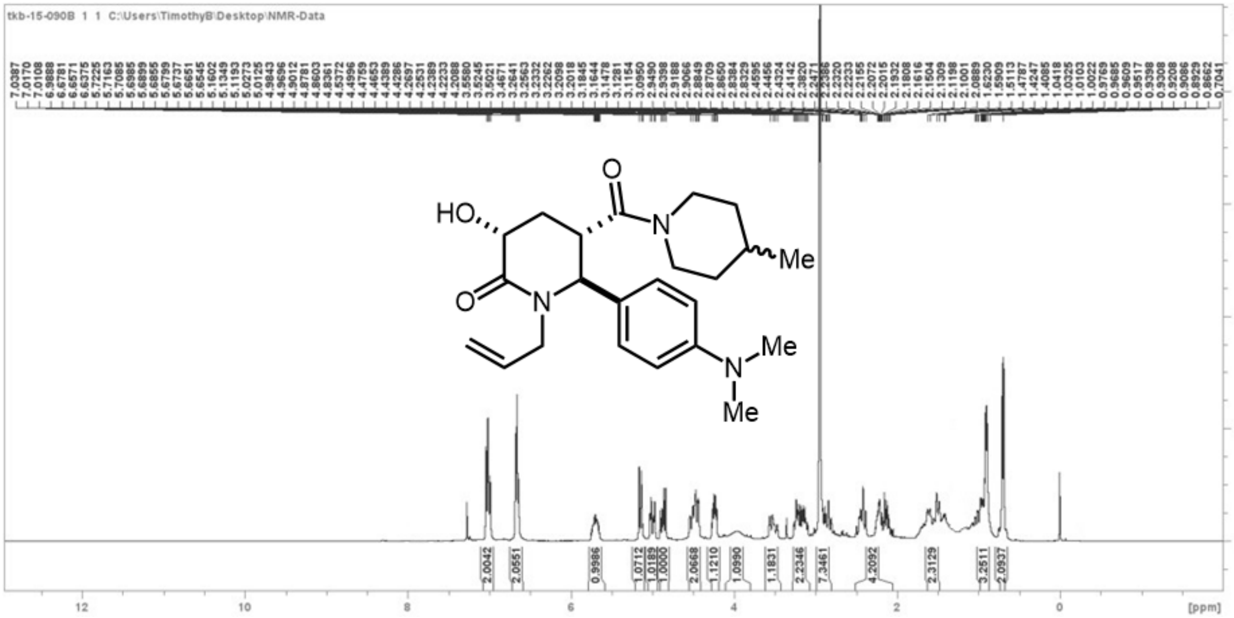
[ppm]



Compound 7z10

Prepared in 1.0 mmol scale using **General Procedure A**. Purification: Flash chromatography on silica eluting with hexane/acetone (90:10 to 50:50). Yellowish oil. Yield = 339.6 mg, 85%, 65:35 dr. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.01 (d, *J* = 11.4, 8.5 Hz, 2H), 6.66 (t, *J* = 8.0 Hz, 2H), 5.70 (dddd, *J* = 17.6, 10.2, 7.8, 4.3 Hz, 1H), 5.15 (d, *J* = 10.2 Hz, 1H), 5.00 (dt, *J* = 17.0, 3.7 Hz, 1H), 4.87 (dd, *J* = 16.4, 9.5 Hz, 1H), 4.57 – 4.40 (m, 2H), 4.24 (dt, *J* = 12.1, 6.1 Hz, 1H), 4.04 (s, 1H), 3.92 (s, 0H), 3.59 – 3.44 (m, 1H), 3.29 – 3.08 (m, 2H), 2.97 – 2.75 (m, 6H), 2.52 – 2.35 (m, 1H), 2.29 – 2.05 (m, 2H), 1.65 – 1.36 (m, 2H), 1.25 (s, 1H), 1.11 – 0.92 (m, 1H), 0.90 (q, *J* = 5.3 Hz, 2H), 0.70 (d, *J* = 6.5 Hz, 2H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.54, 169.43, 150.58, 150.30, 131.87, 128.20, 127.99, 127.04, 126.83, 118.43, 118.35, 112.72, 112.49, 67.34, 67.14, 63.98, 63.29, 46.88, 46.81, 46.58, 45.58, 43.20, 43.03, 42.47, 40.49, 40.45, 34.79, 33.89, 33.74, 33.64, 32.32, 31.97, 30.90, 30.84, 21.52, 21.48. **HRMS-EI^{+}** (*m/z*): calc for $C_{23}H_{33}N_3O_3$ $[M]^{+}$ 399.2522, found 399.2527.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Me
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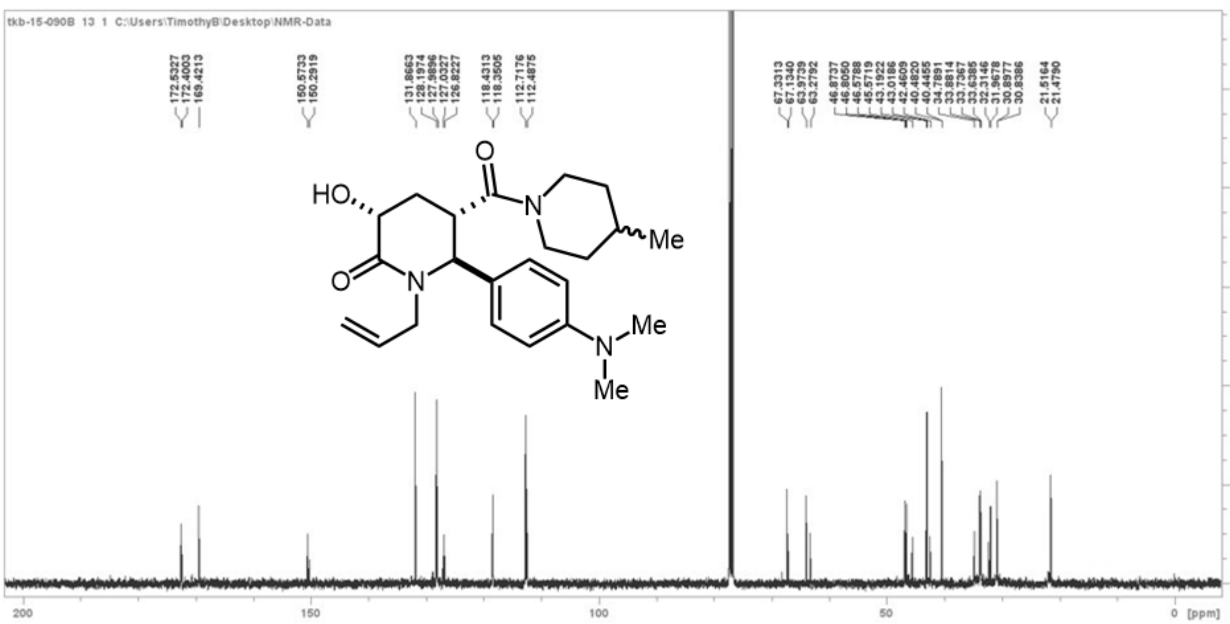
[rel]
[ppm]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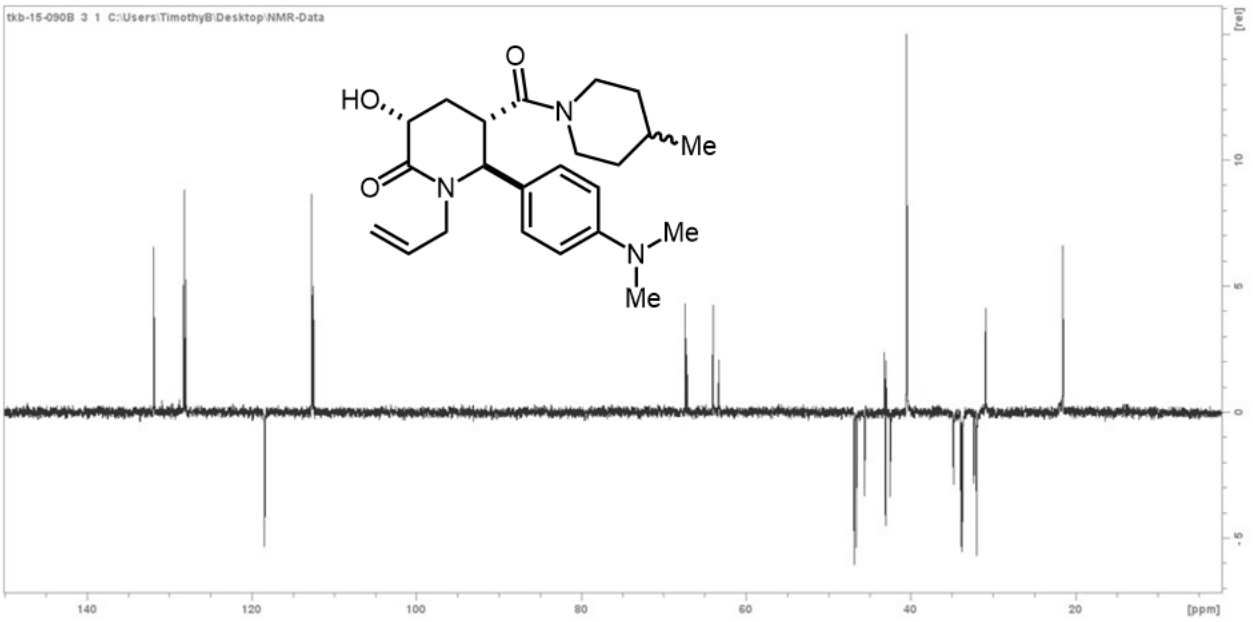
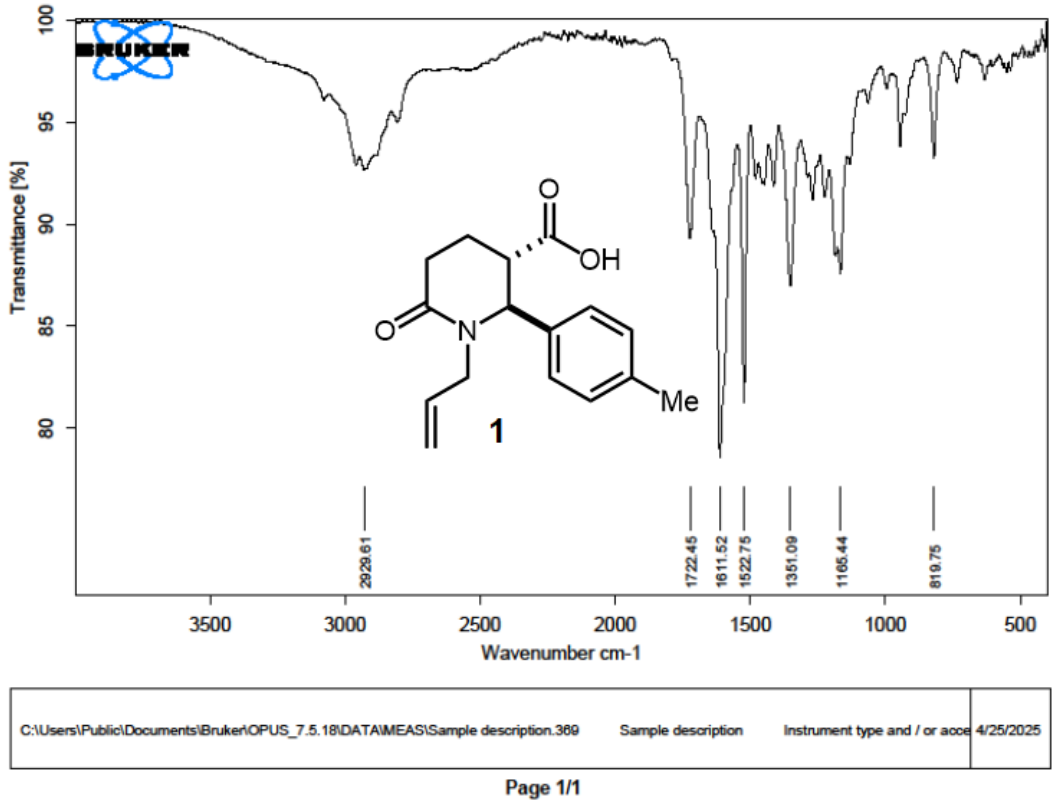
[rel]
[ppm]



IR spectrum (OH stretching frequency goes from ~2700 – 3200 cm^{-1})



References

1 T. K. Beng, V. Shearer, A. O. Farah, *JACS 2025, minor revisions requested*.