Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.
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Supporting Information for:

Expedient and modular access to 2-azabicyclic architectures by iron-catalyzed dehydrative coupling of alcohol-bearing allylic lactams

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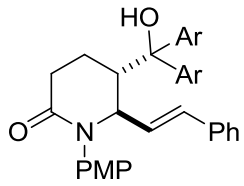
2. Experimental Section

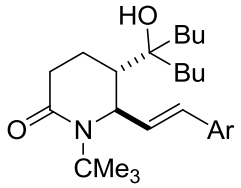
All experiments involving air- and moisture-sensitive reagents were carried out under an inert atmosphere of nitrogen and using freshly distilled solvents. Freshly purchased acetonitrile was stored under 4 A^o molecular sieves for several days prior to use. THF was distilled from sodium benzophenone ketyl. 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) or CAM, *p*-anisaldehyde, or $KMnO_4$ stain. All reported temperatures were internal to the reaction vessel. Unless otherwise indicated, ^1H, ^{13}C, and DEPT-135, COSY 45, and HMQC (or HSQC) spectra were acquired using $CDCl_3$ as solvent at room temperature. Chemical shifts are quoted in parts per million (ppm). HRMS-EI^+ data were obtained using either electronspray 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 alcohol-bearing allylic lactams employed in these studies (see below) were prepared using our previously reported protocol,[1] with Grignard reagents employed in place of organolithiums in some cases (*e.g.*, allylmagnesium bromide in place of allyllithium).

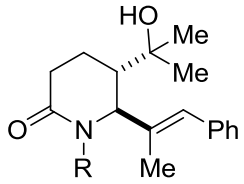
The iron-catalyzed conditions were greatly inspired by insightful reports from the laboratories of Liu and coworkers.[2]



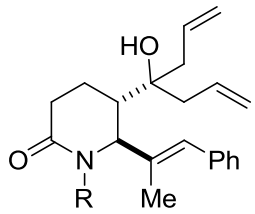
2a, Ar = Ph
2b, Ar = MMP



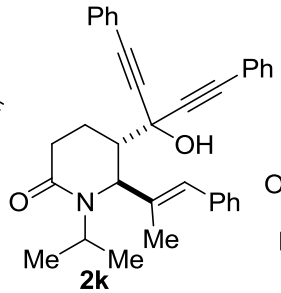
2c, Ar = Ph
2d, Ar = PMP



2e, R = PMP
2f, R = isopropyl
2g, R = cyclopropyl
2h, R = allyl



2i, R = isopropyl
2j, R = cyclopropyl



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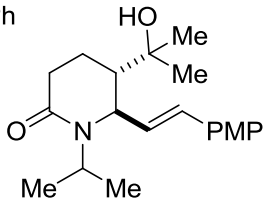

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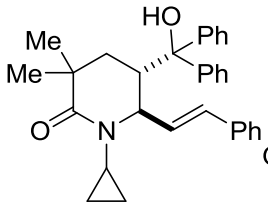

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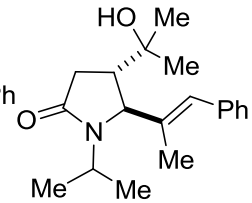

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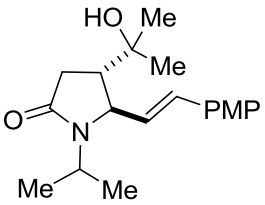

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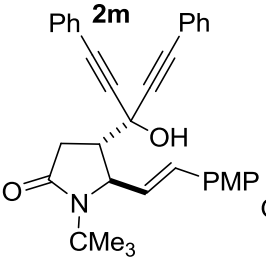

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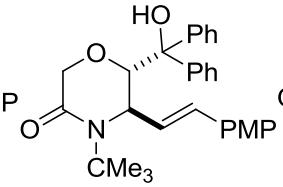

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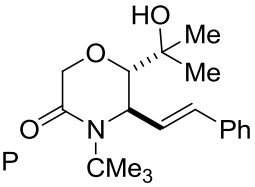

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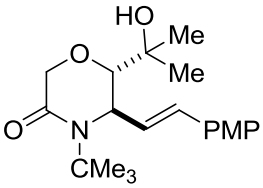

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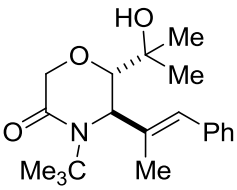

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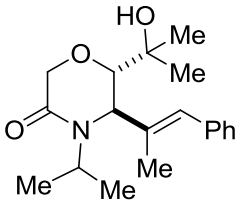

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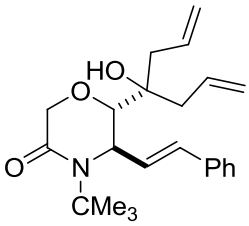

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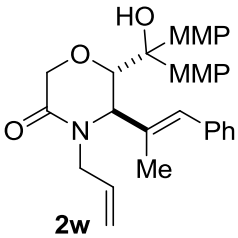

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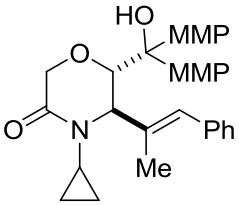
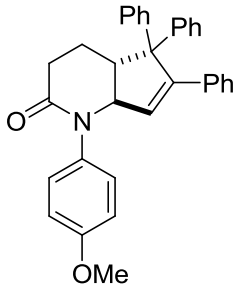


2x

Ph = phenyl, PMP = *para*-methoxyphenyl
MMP = *meta*-methoxyphenyl

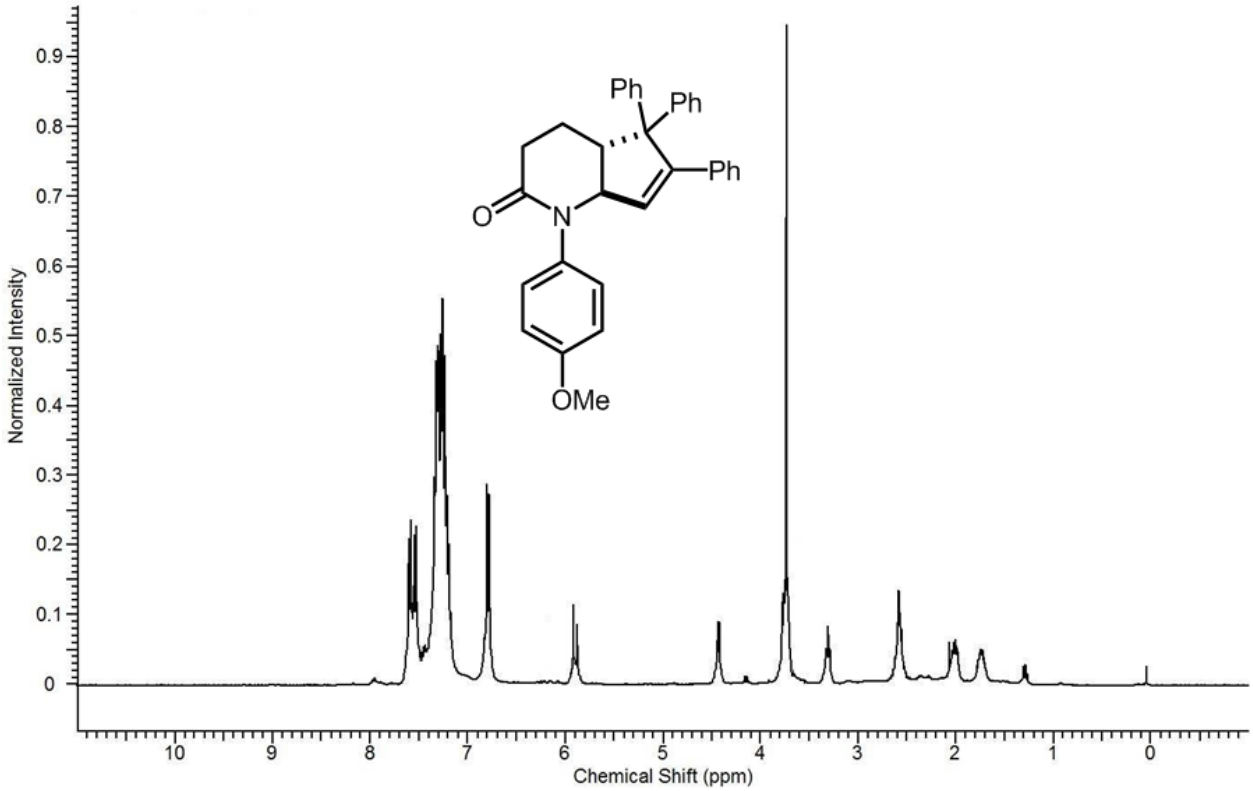
General Procedure A: Conversion of 2 to 1

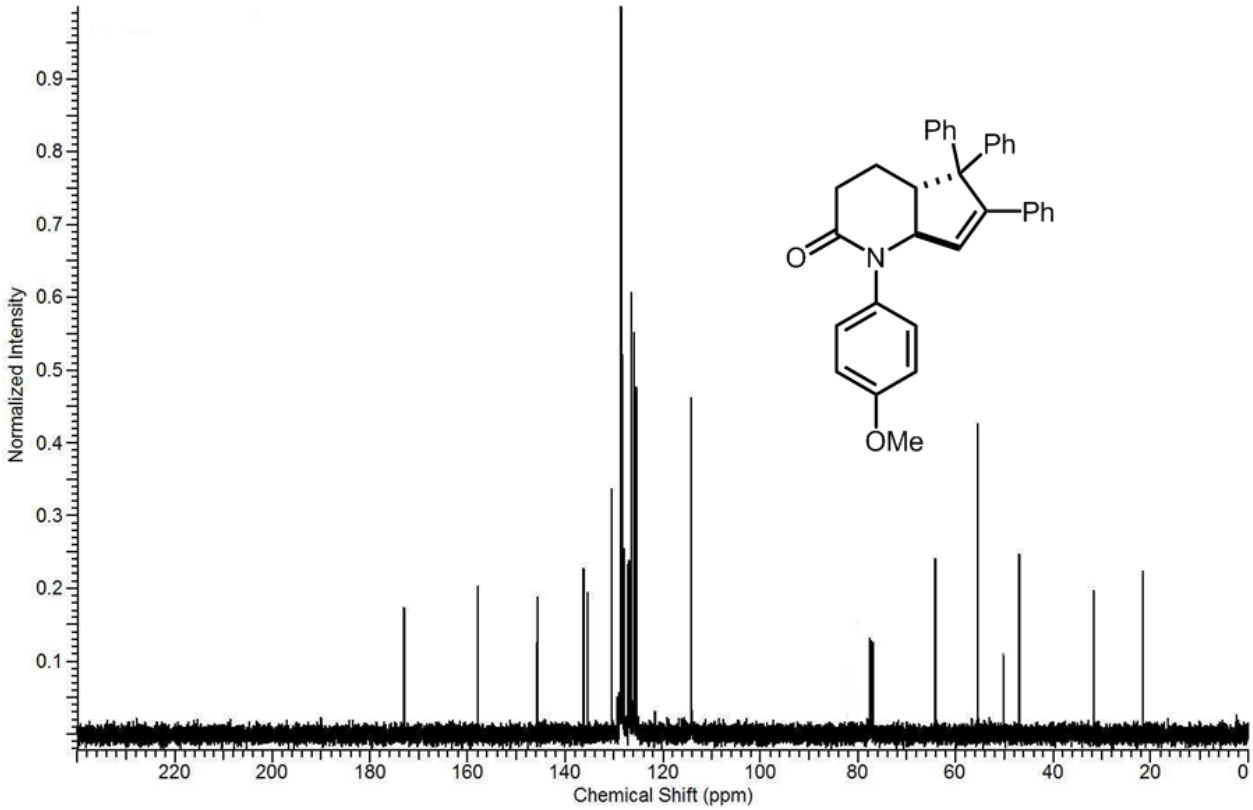
To a solution of alcohol-bearing alkene **2** (1 mmol) in MeCN (5 mL) was added $FeCl_3 \cdot 6H_2O$ (5 mol%) in a vial. The contents were placed in an oil bath thermostatted at 80 ^oC. Heating was continued until complete consumption of **2** (GC-MS and TLC monitoring). Following completion of the reaction, the mixture was evaporated under vacuum and purified by flash column chromatography on silica (pretreated with Et_3N) to afford the desired product (*i.e.*, **1**).



1a; 87%, 95:5 dr

Prepared from alcohol **2a** (122.3 mg, 0.250 mmol) using General Procedure A. Oily substance. Yield = 102.4 mg, 87%. ^1H NMR (400 MHz, $CDCl_3$) δ 7.61 to 7.14 (m, 17H), 6.82 (qd, *J* = 9.2, 7.9, 5.3 Hz, 2H), 5.90 (d, *J* = 8.0 Hz, 1H), 4.38 (d, *J* = 8.3 Hz, 1H), 3.79 (s, 3H), 3.34 to 3.28 (m, 1H), 2.59 to 2.52 (m, 2H), 2.05 to 1.97 (m, 1H), 1.78 to 1.72 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.0, 158.0, 145.8, 136.3, 135.4, 130.5, 130.5, 129.4, 128.8, 128.7, 128.6, 128.5, 128.2, 127.9, 127.2, 127.0, 126.9, 126.5, 126.1, 125.9, 125.5, 114.1, 64.1, 55.4, 50.2, 46.9, 31.6, 21.5. **HRMS-EI$^+$** (*m/z*): calc'd for $C_{33}H_{29}NO_2$ 471.2198; found 471.2203.


Ph
Ph
Ph
O
N
OMe
Normalized Intensity
Chemical Shift (ppm)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O
N
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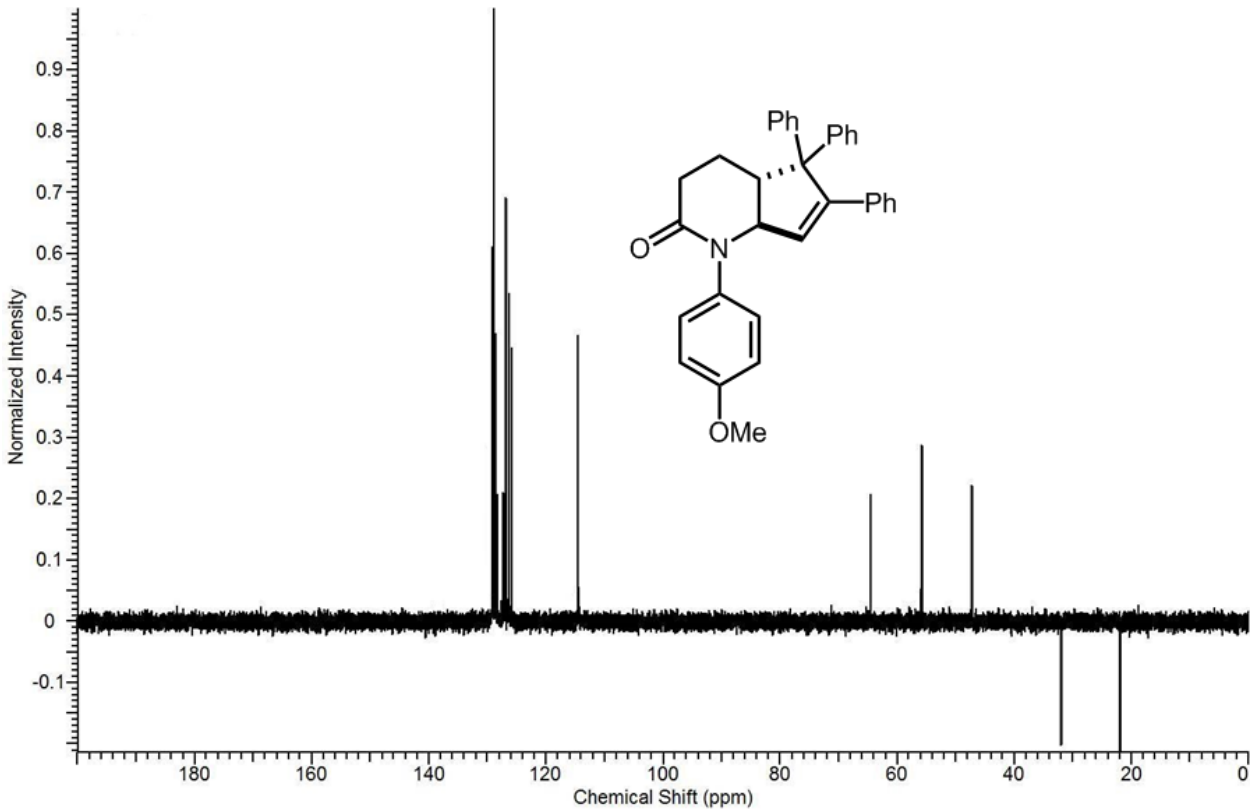
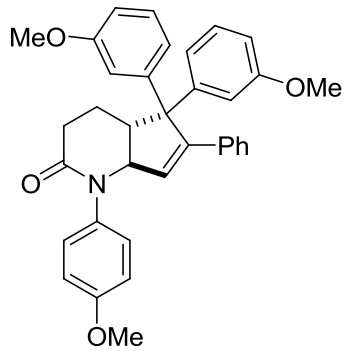
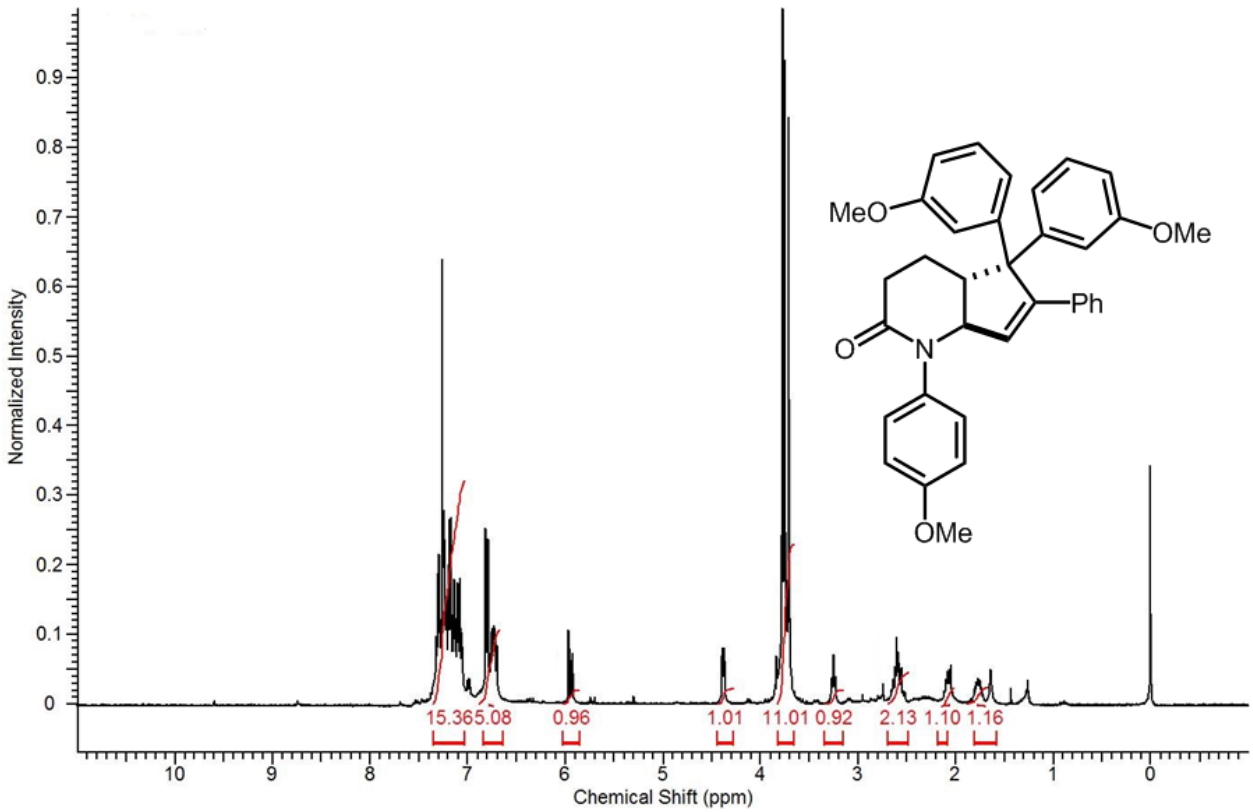
Normalized Intensity
Chemical Shift (ppm)





1b; 91%, 96:4 dr

Prepared from alcohol **2b** (137.3 mg, 0.250 mmol) using General Procedure A. Oily substance Yield = 120.9 mg, 91%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 to 7.08 (m, 12H), 6.82 to 6.70 (m, 5H), 5.94 (d, *J* = 7.8 Hz, 1H), 4.42 (d, *J* = 7.2 Hz, 1H), 3.76 to 3.71 (m, 9H), 3.45 to 3.35 (m, 1H), 2.59 to 2.52 (m, 2H), 2.12 to 2.05 (m, 1H), 1.78 to 1.72 (m, 1H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.7, 159.8, 158.0, 147.1, 147.0, 136.2, 135.3, 130.7, 130.4, 129.6, 129.5, 128.8, 128.7, 128.1, 127.9, 126.7, 126.5, 118.1, 117.7, 114.2, 112.1, 64.0, 55.4, 55.3, 55.2, 52.0, 46.8, 31.6, 21.6. **HRMS-EI^{+}** (*m/z*): calc'd for $C_{35}H_{33}NO_4$ 531.2410; found 531.2417.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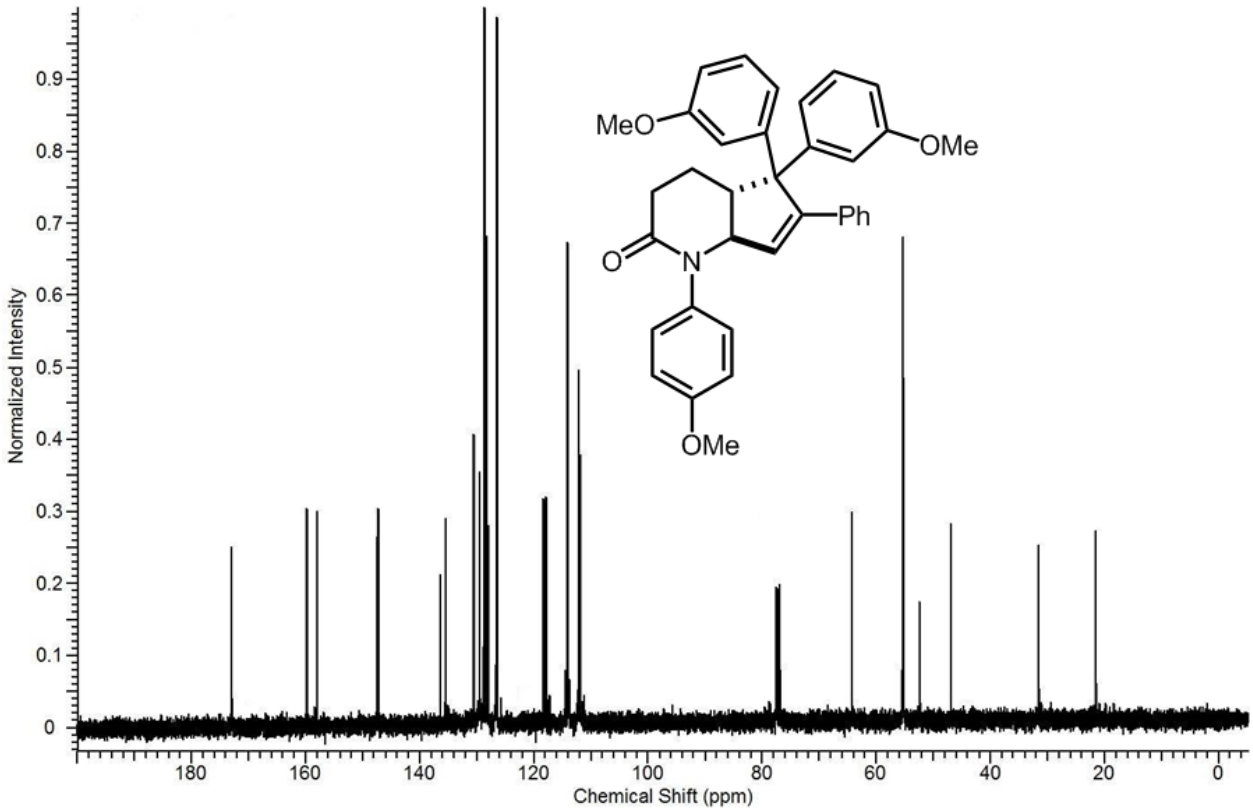
OMe
Ph
O
N
OMe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Chemical Shift (ppm)
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Ph
O
N
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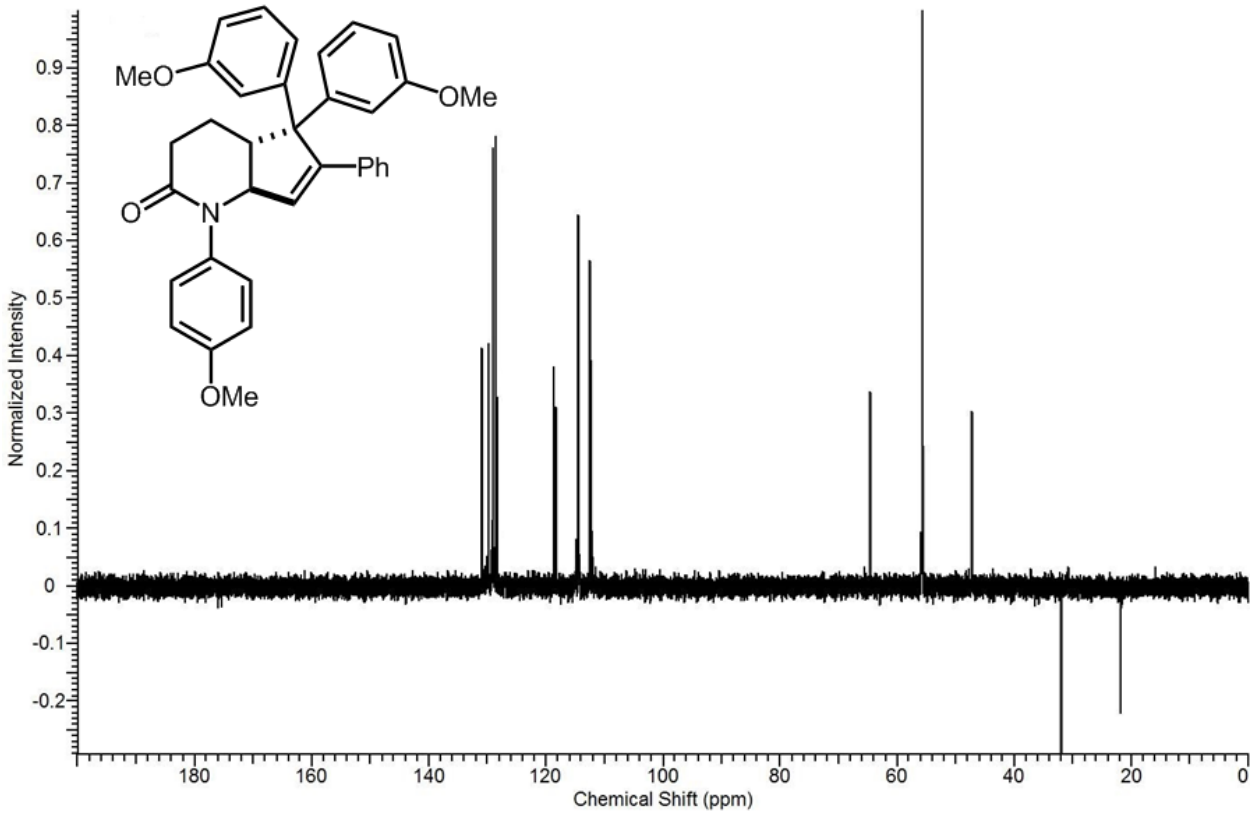
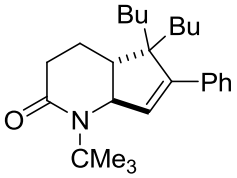
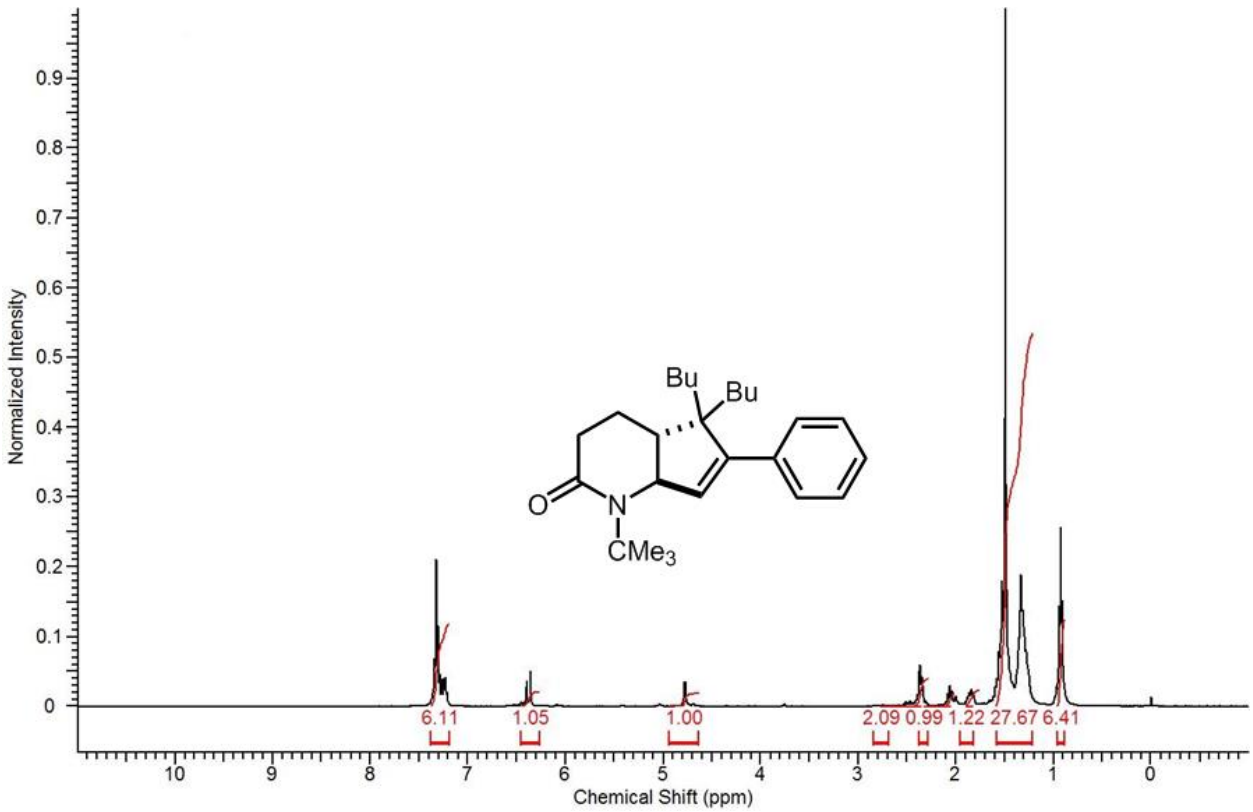




1c; 82%, 97:3 dr

Prepared from alcohol **2c** (100 mg, 0.250 mmol) using General Procedure A. Oily substance Yield = 78.2 mg, 82%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 to 7.20 (m, 5H), 6.42 (d, 1H), 4.77 to 4.74 (dd, 1H), 2.53 to 2.41 (m, 2H), 2.08 to 1.81 (m, 1H), 1.59 to 1.41 (m, 1H), 1.37 to 1.25 (m, 22H), 0.98 to 0.91 (m, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.5, 136.6, 135.0, 128.0, 127.7, 126.4, 126.3, 58.3, 55.3, 52.0, 48.2, 36.9, 32.7, 32.0, 28.9, 26.2, 23.5, 22.4, 16.8. **HRMS-EI^{+}** (*m/z*): calc'd for $C_{26}H_{39}NO$ 381.3032; found 381.3038.


Normalized Intensity
Chemical Shift (ppm)
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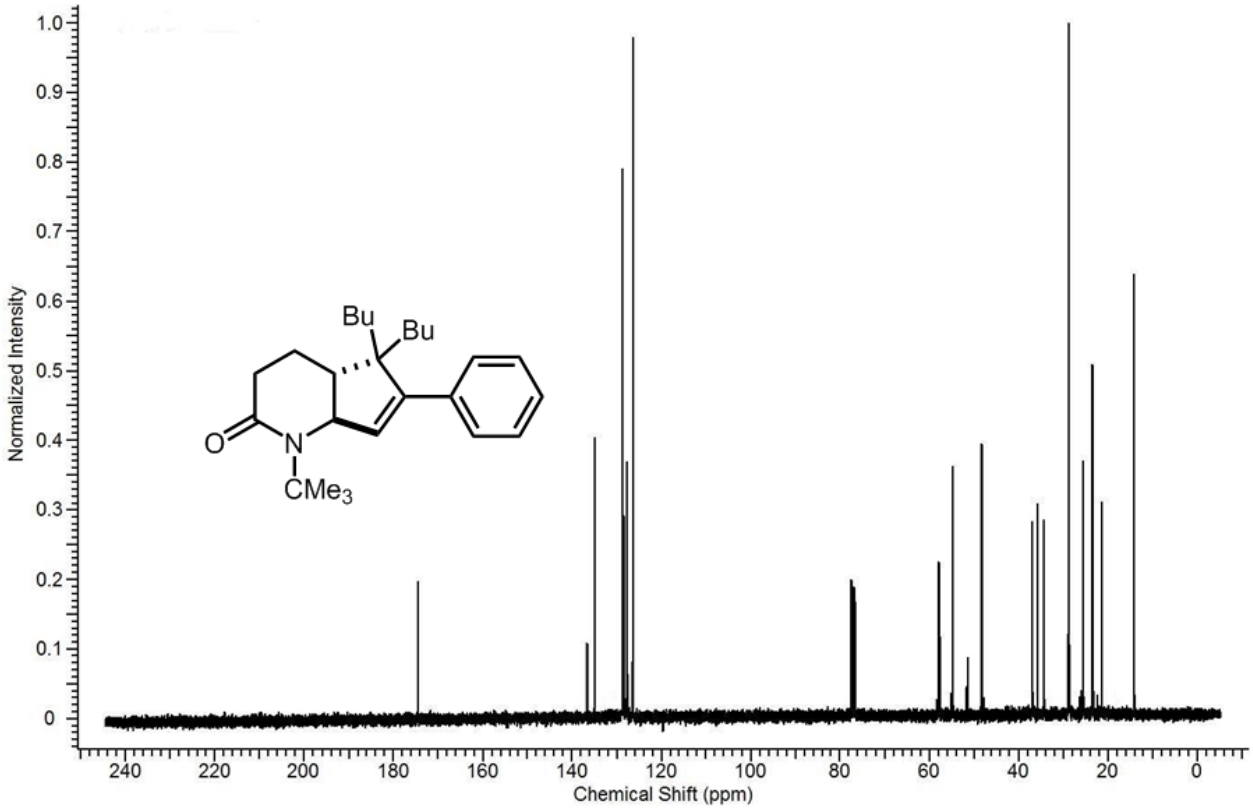
2.09
0.99
1.22
27.67
6.41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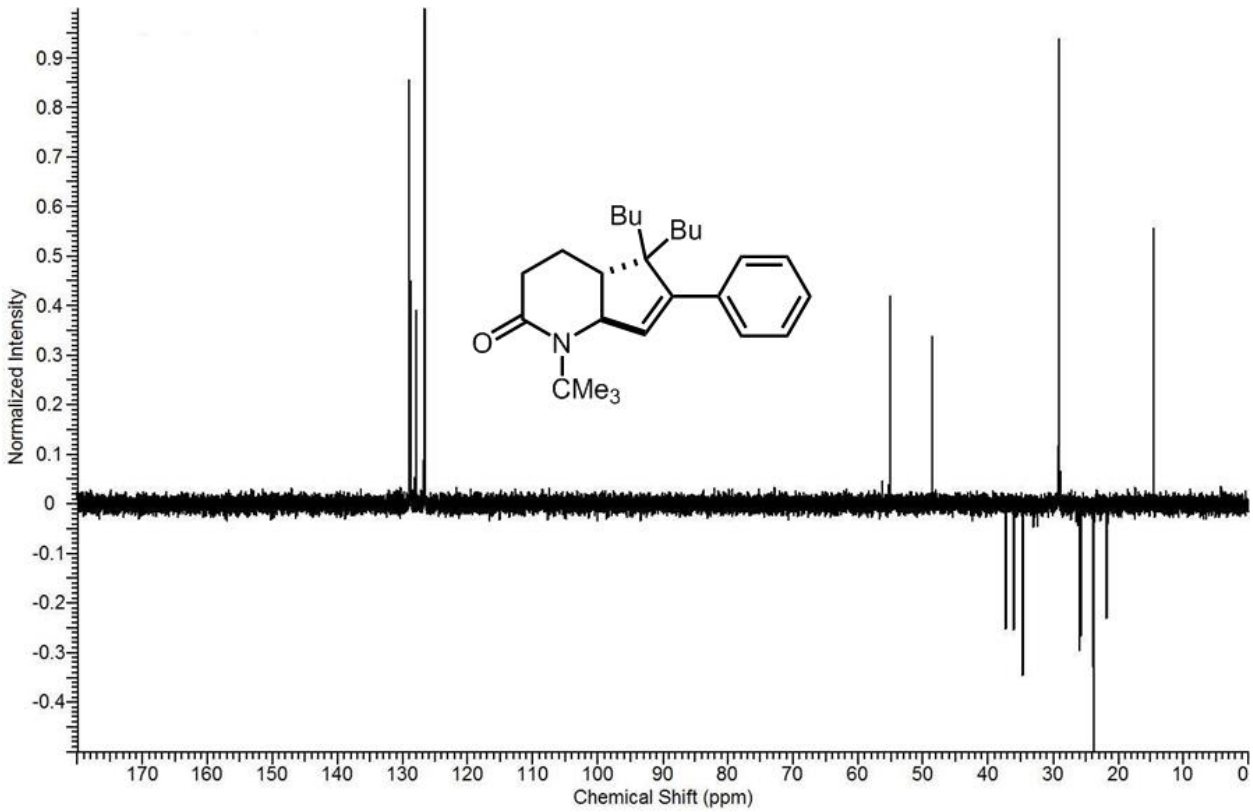
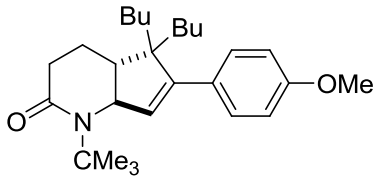
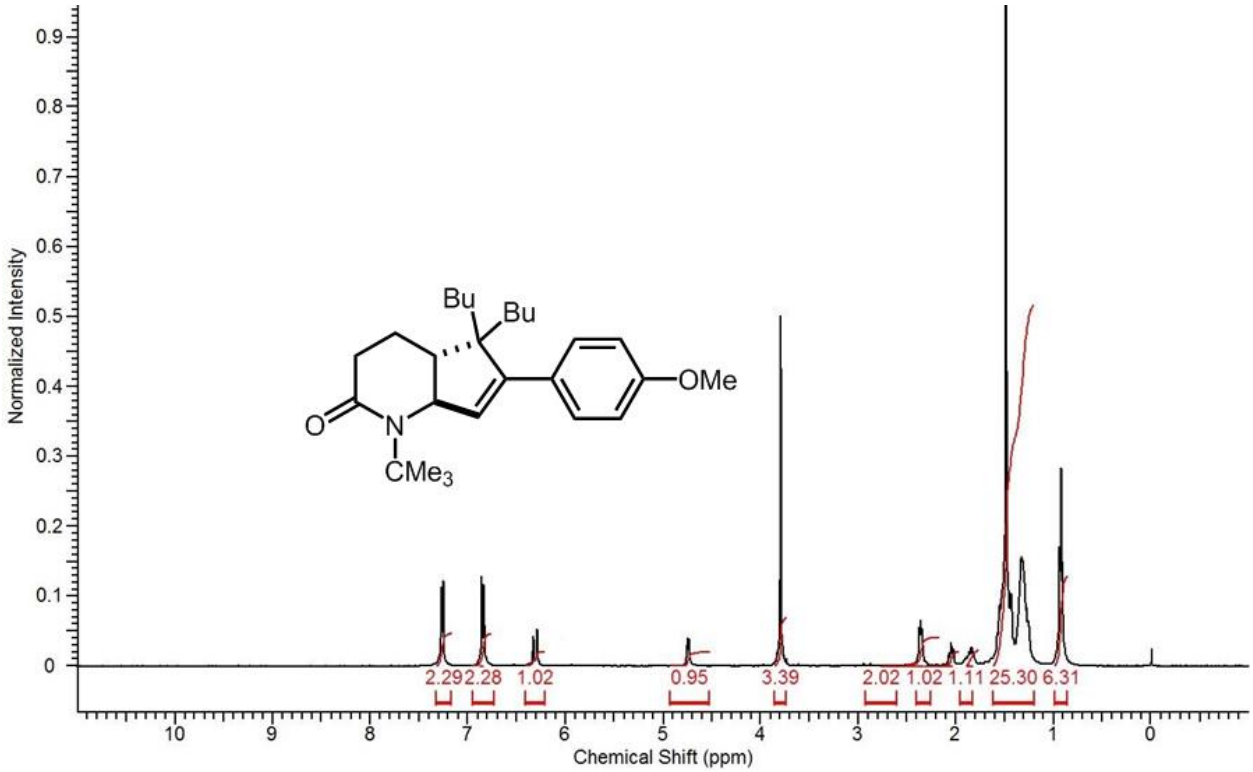
N
CMe3

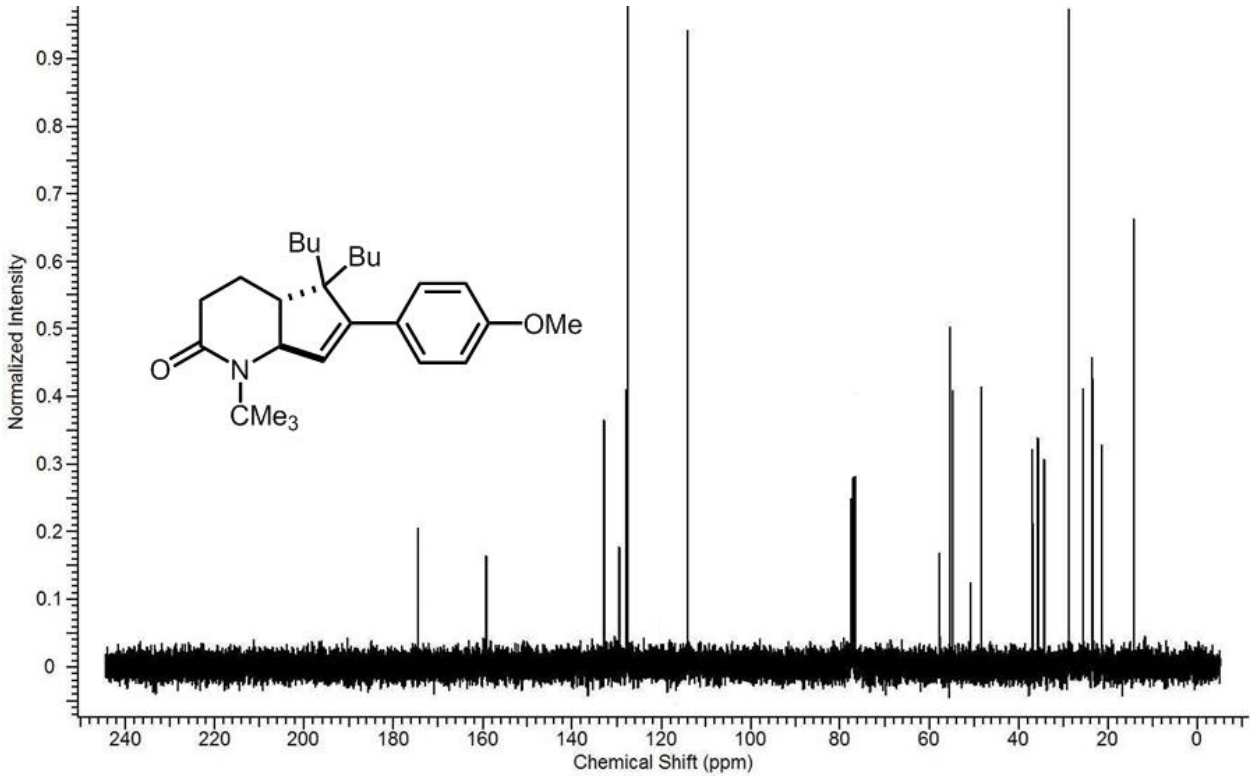




1d; 85%, 98:2 dr

Prepared from alcohol **2d** (107.5 mg, 0.250 mmol) using General Procedure A. Oily substance Yield = 87.5 mg, 85%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.26 (d, *J* = 8.5 Hz, 2H), 6.85 (d, *J* = 8.4 Hz, 2H), 6.31 (d, *J* = 6.9 Hz, 1H), 4.74 (d, *J* = 6.9 Hz, 1H), 3.80 (s, 3H), 2.39 to 2.30 (m, 2H), 2.07 to 2.02 (m, 1H), 1.88 to 1.80 (m, 1H), 1.57 to 1.25 (m, 22H), 0.98 to 0.94 (m, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.6, 159.2, 132.8, 129.3, 127.8, 127.4, 114.1, 57.7, 55.4, 54.7, 51.2, 48.3, 36.9, 35.7, 34.3, 28.7, 25.6, 25.4, 23.5, 23.4, 21.3, 14.2. **HRMS-EI^{+}** (*m/z*): calc'd for $C_{27}H_{41}NO_2$ 411.3137; found 411.3142.


Normalized Intensity
Chemical Shift (ppm)
Bu
Bu
OMe
O
N
CMe3
2.29 2.28 1.02 0.95 3.39 2.02 1.02 1.11 25.30 6.31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Chemical Shift (ppm)
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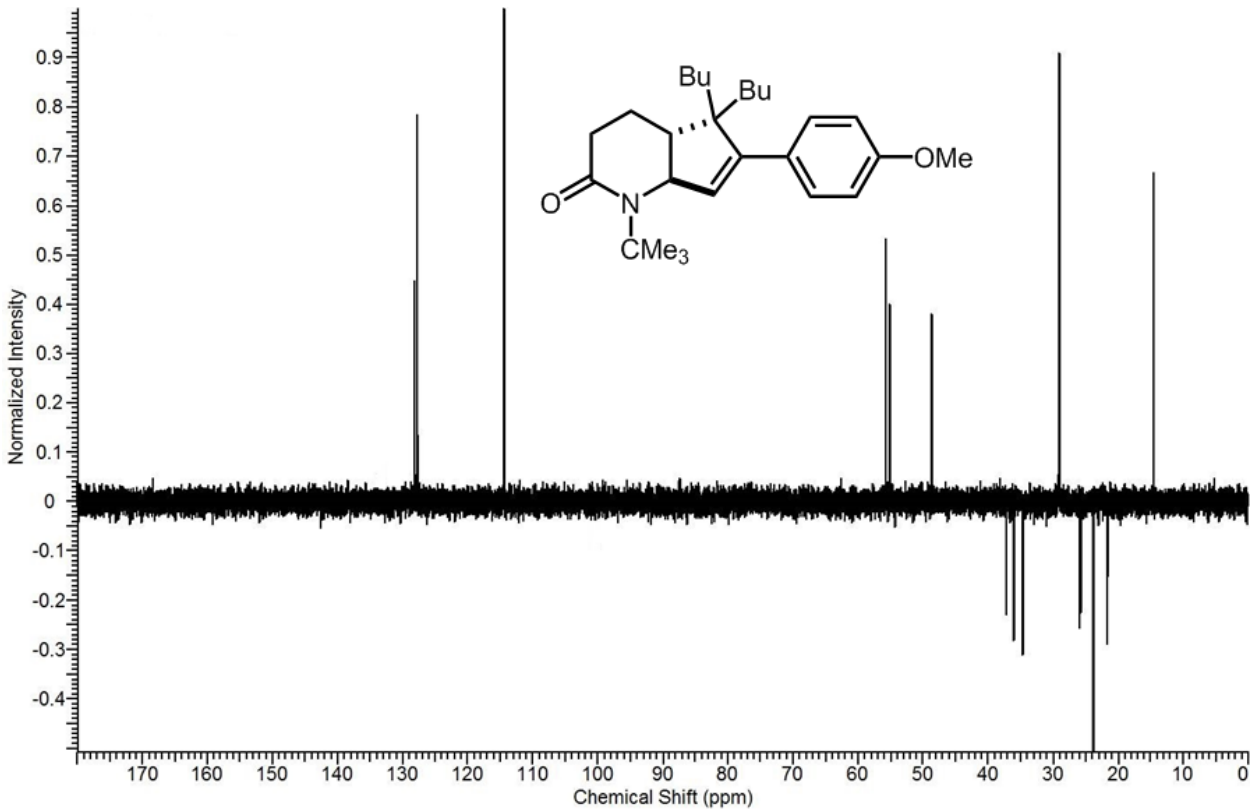
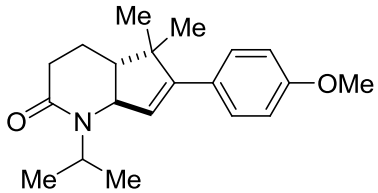
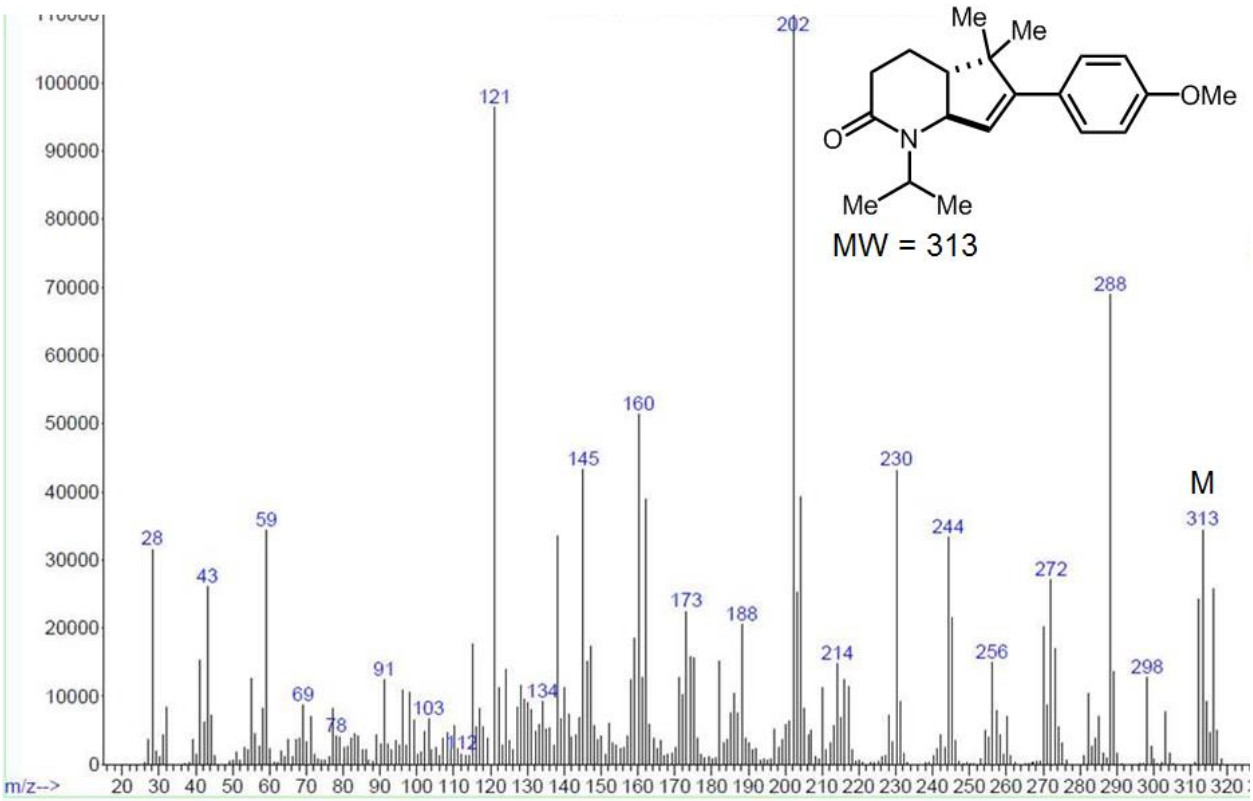




1e; 76%, 93:7 dr

Prepared from alcohol **2e** (83 mg, 0.250 mmol) using General Procedure A. Oily substance Yield = 60 mg, 76%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.22 (d, *J* = 6.4 Hz, 2H), 6.78 (d, *J* = 6.4 Hz, 2H), 6.43 (d, 1H), 4.36 to 4.26 (m, 2H), 3.75 to 3.60 (m, 4H), 2.39 to 2.05 (m, 3H), 1.93 to 1.71 (m, 2H), 1.31 to 1.06 (m, 12H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.8, 159.2, 132.3, 131.9, 129.3, 128.4, 128.1, 127.6, 127.5, 127.4, 113.9, 56.7, 55.3, 51.4, 50.3, 48.2, 31.6, 29.4, 29.3, 29.1, 25.8, 23.6, 20.8, 20.1, 17.8. **HRMS-EI^{+}** (*m/z*): calc'd for $C_{20}H_{27}NO_2$ 313.2042; found 313.2045.


MW = 313
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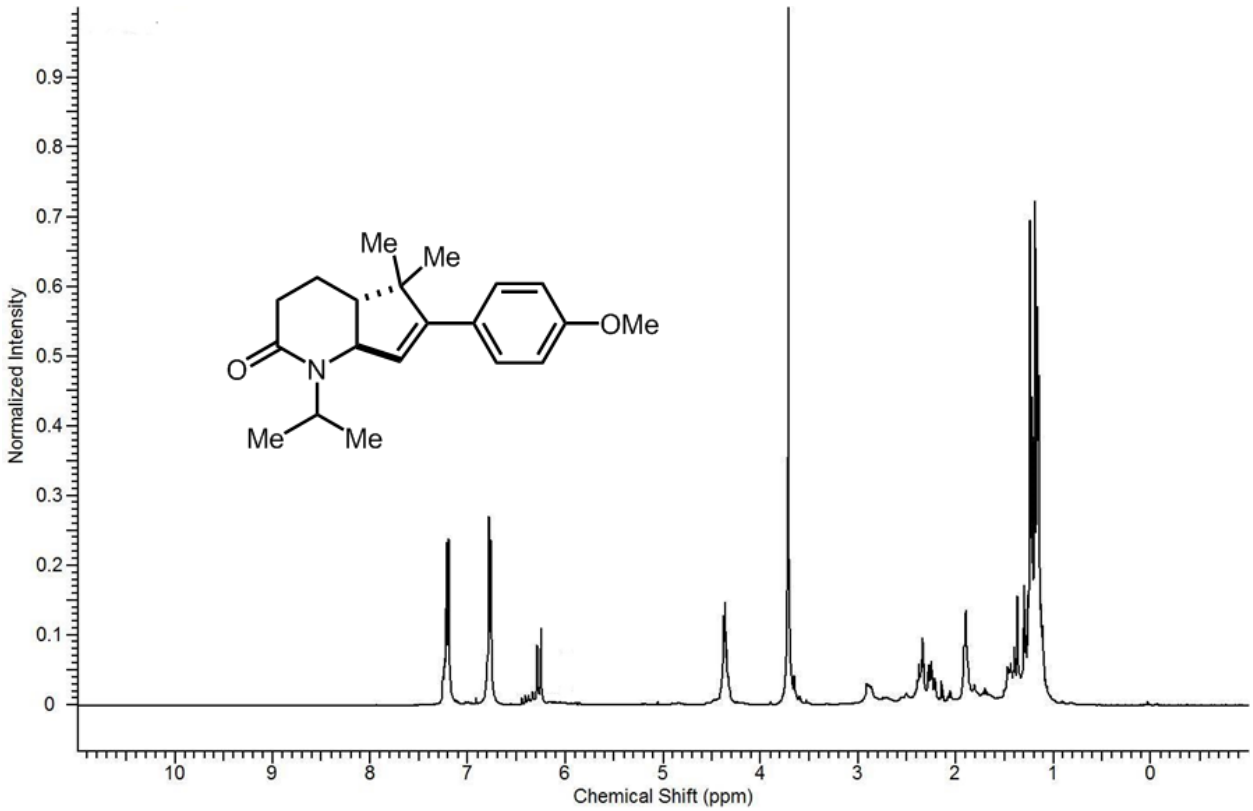
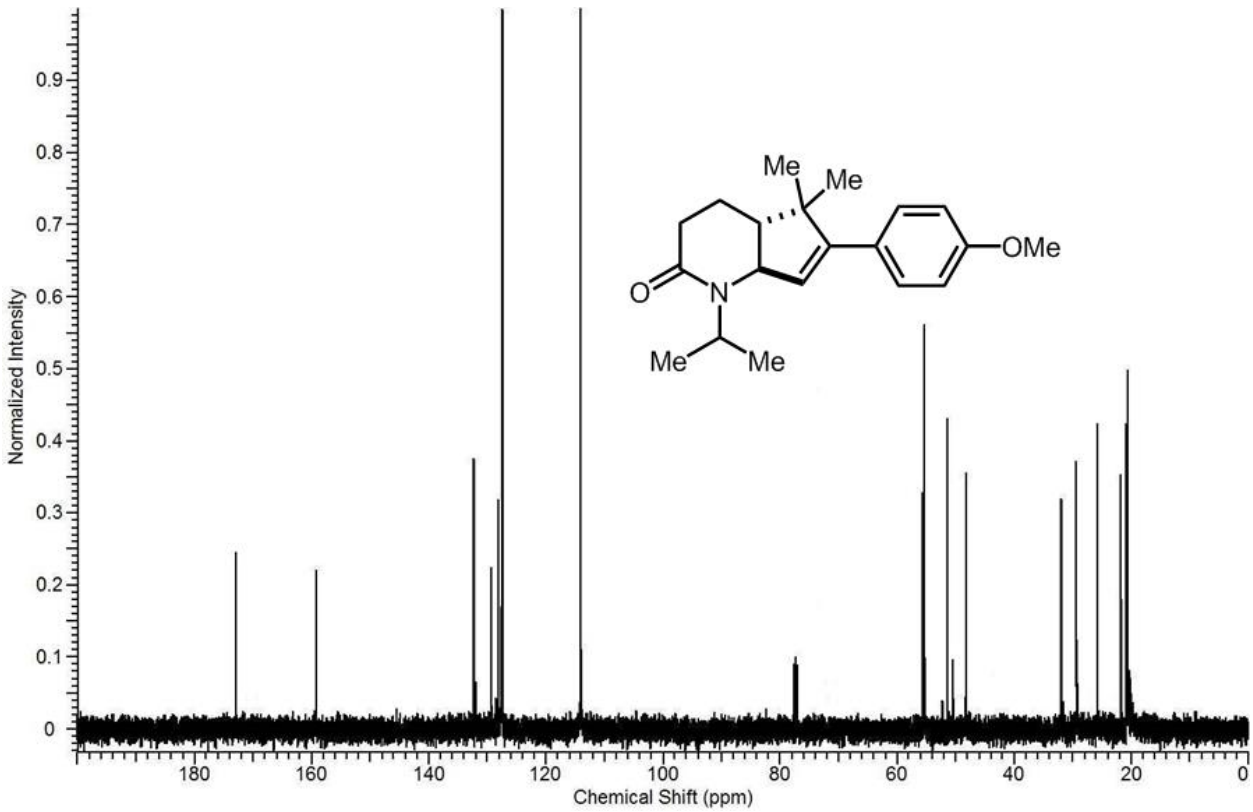
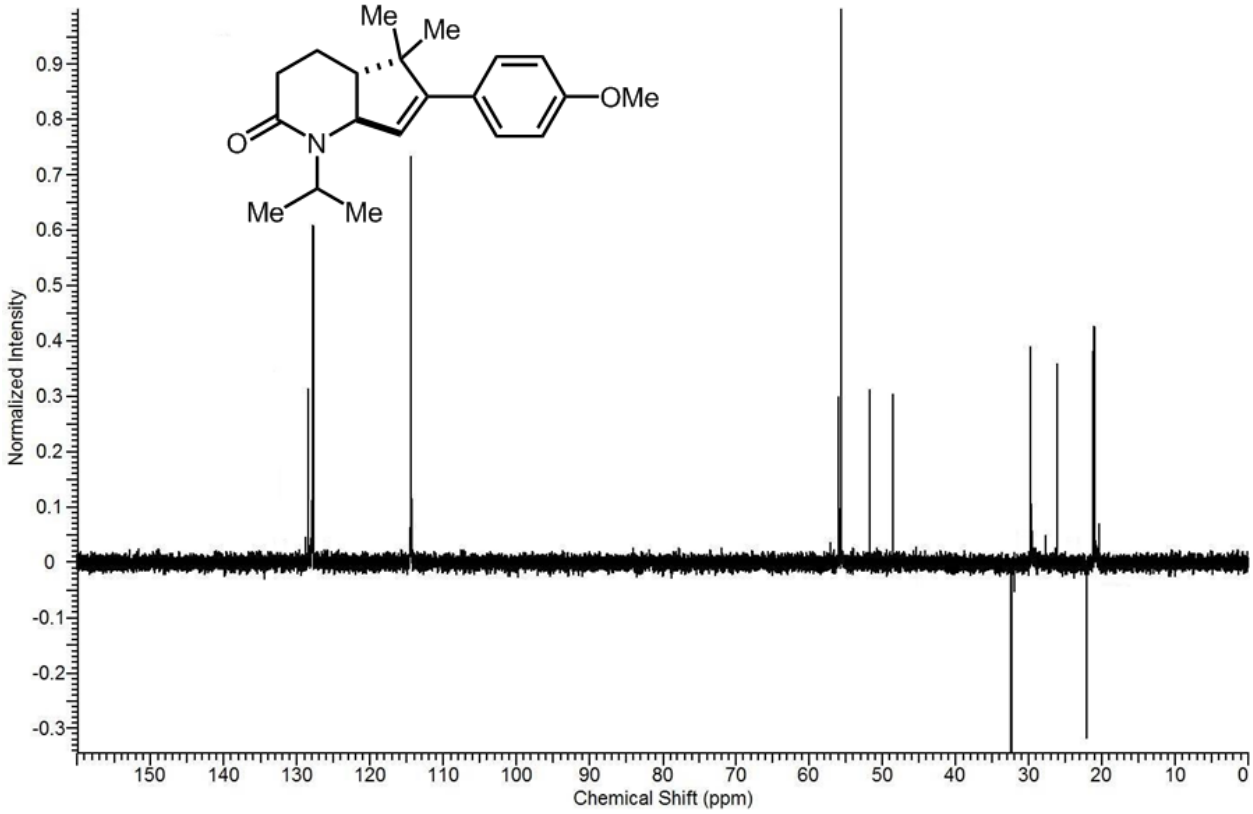
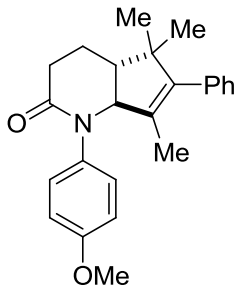
m/z-->
Me
Me
OMe
O
N
Me
Me


Normalized Intensity
Chemical Shift (ppm)
Me
Me
OMe
O
N
Me
Me

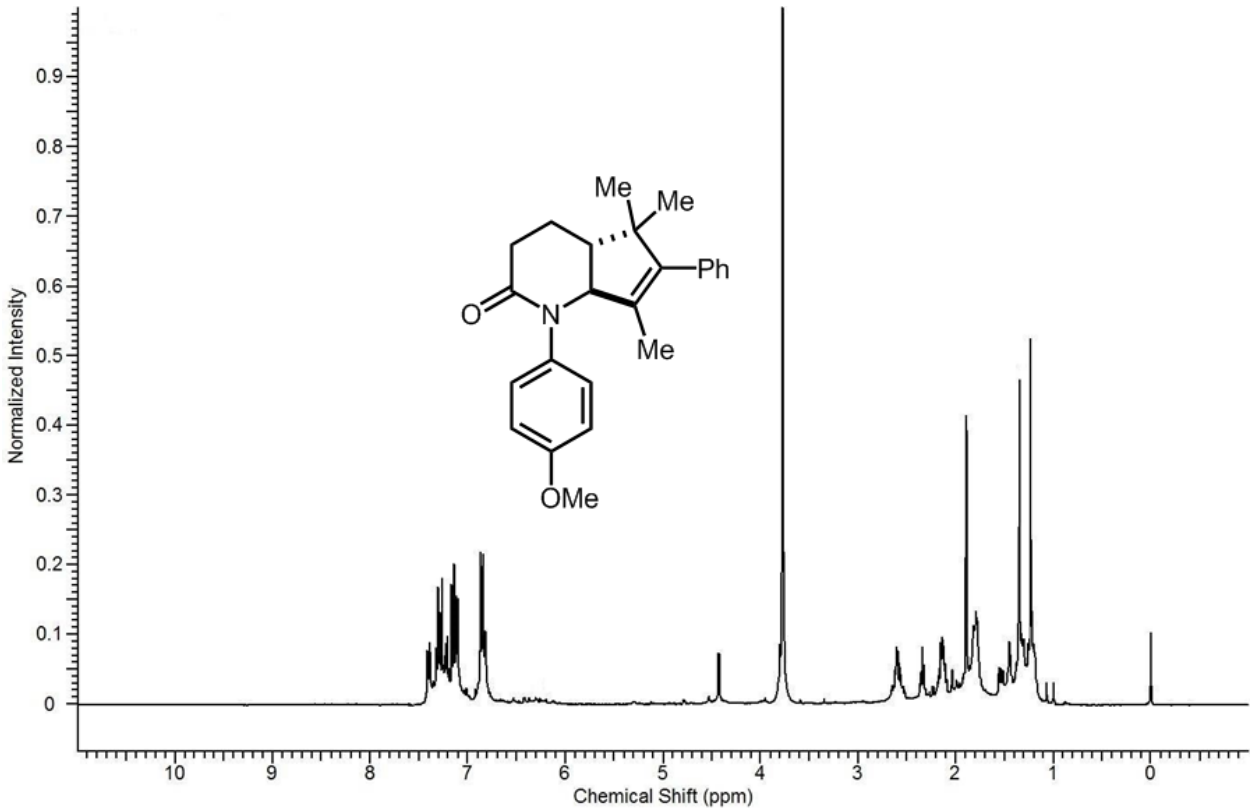

Normalized Intensity
Chemical Shift (ppm)
Me
Me
OMe
O
N
Me
Me

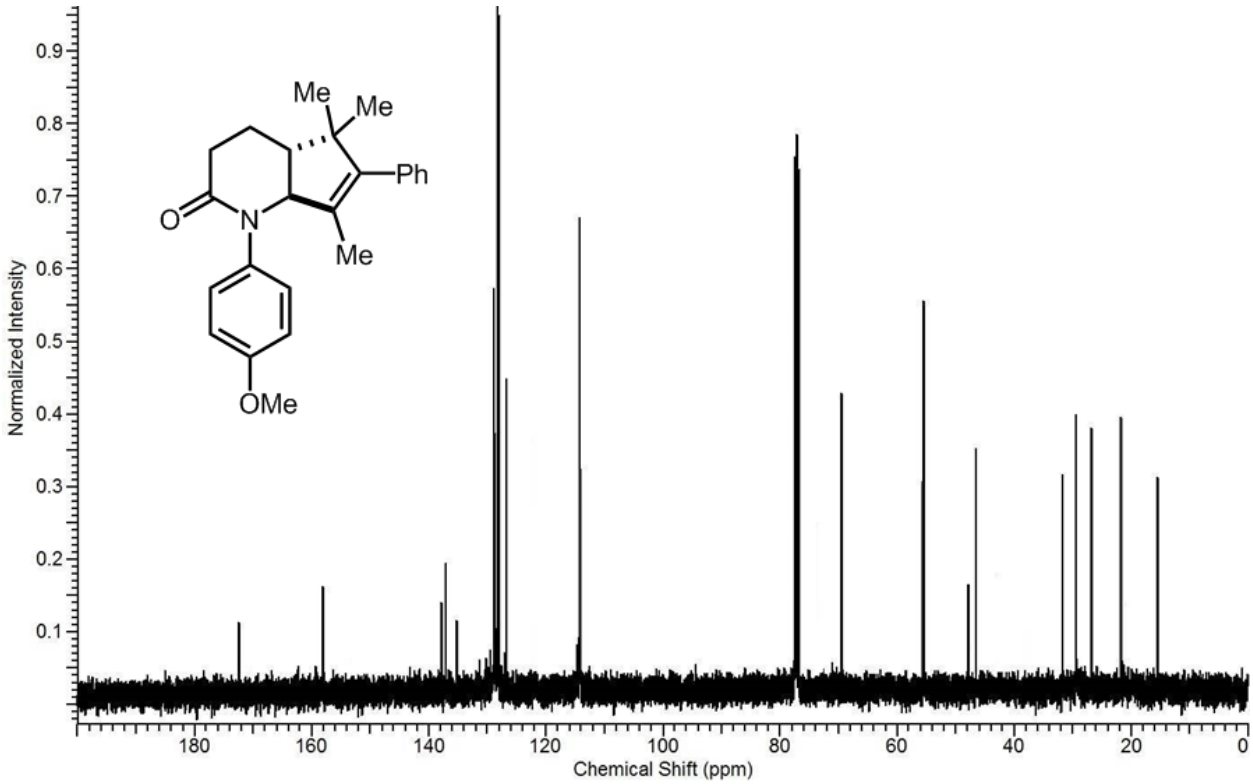

Normalized Intensity
Chemical Shift (ppm)
Me
Me
OMe
O
N
Me
Me

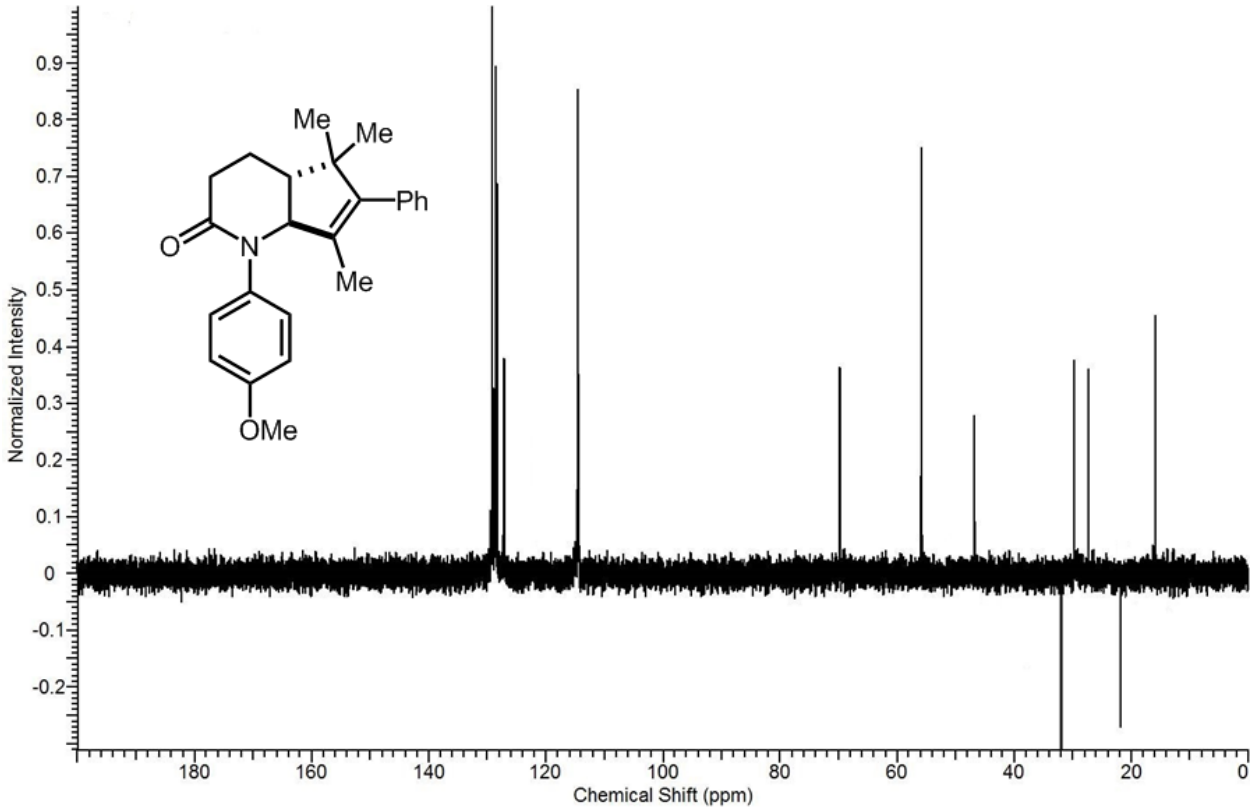


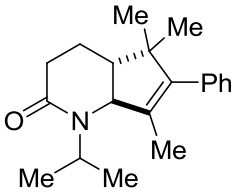
1f; 80%, 96:4 dr

Prepared from alcohol **2f** (95 mg, 0.250 mmol) using General Procedure A. Oily substance. Yield = 72.5 mg, 80%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.42 to 7.07 (m, 7H), 6.82 (d, 2H), 4.43 (d, *J* = 3.9 Hz, 1H), 3.79 (s, 3H), 2.65 to 2.57 (m, 2H), 2.36 to 2.11 (m, 2H), 1.89 to 1.75 (m, 5H), 1.43 to 1.22 (m, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 172.4, 158.0, 137.7, 137.1, 135.1, 131.2, 129.4, 128.7, 128.0, 127.9, 126.8, 121.7, 114.6, 70.0, 55.7, 47.6, 46.6, 31.7, 29.4, 26.8, 21.7, 15.4. **HRMS-EI$^+$** (*m/z*): calc'd for $C_{24}H_{27}NO_2$ 361.2042; found 361.2045.



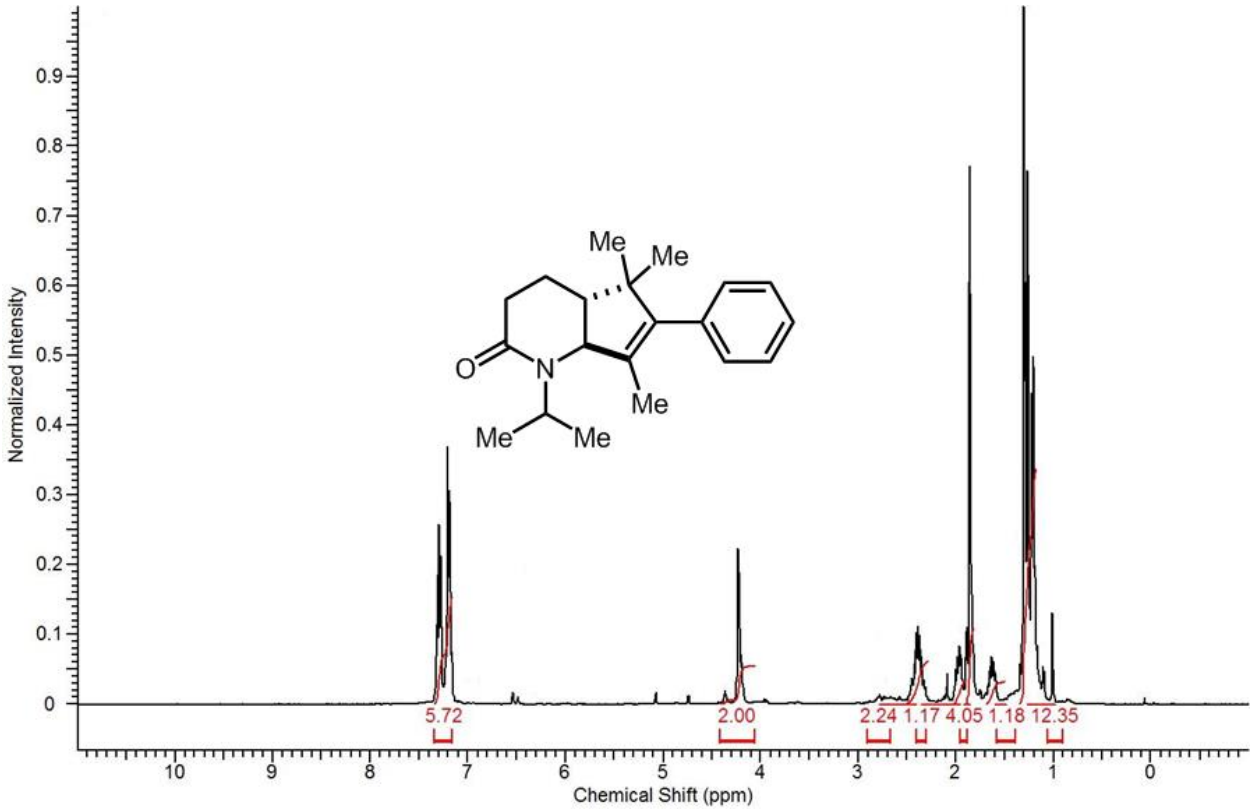

Me
Me
Ph
O
N
Me
OMe
Normalized Intensity
Chemical Shift (ppm)

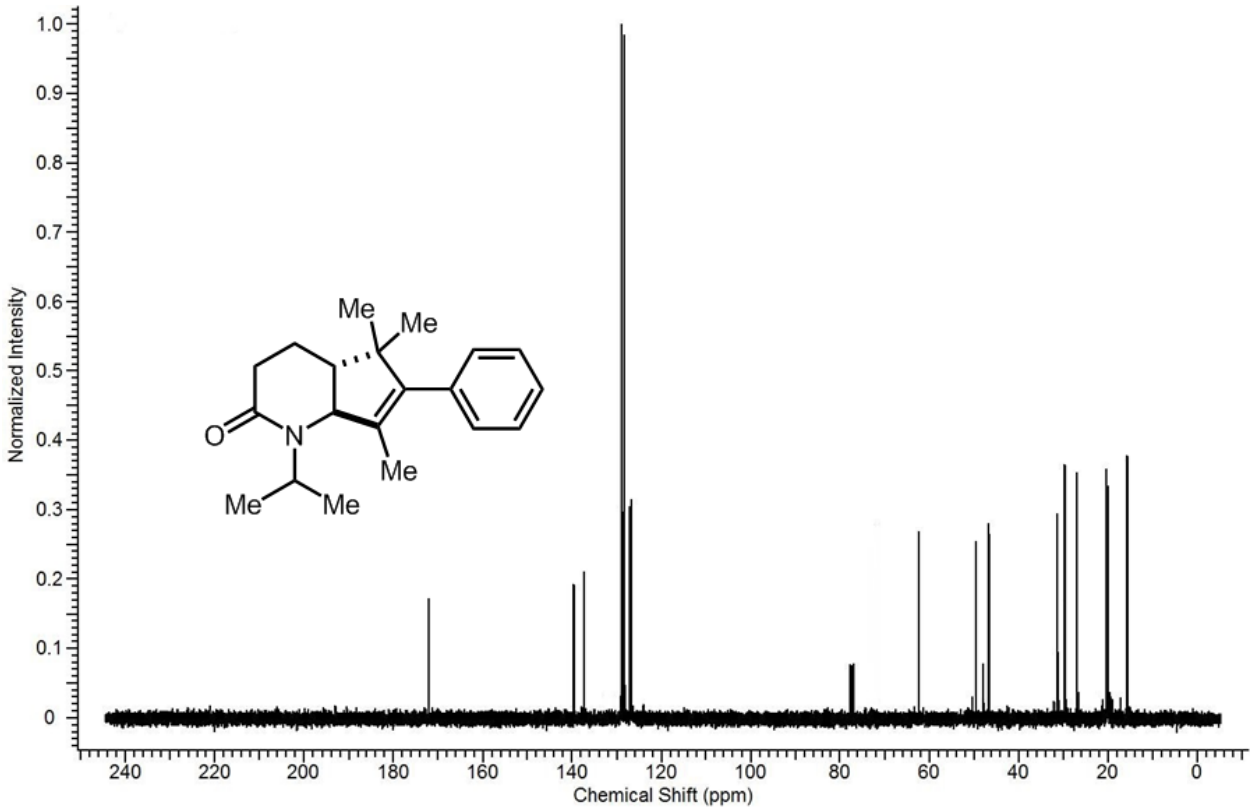

Me
Me
Ph
O
N
Me
OMe
Normalized Intensity
Chemical Shift (ppm)

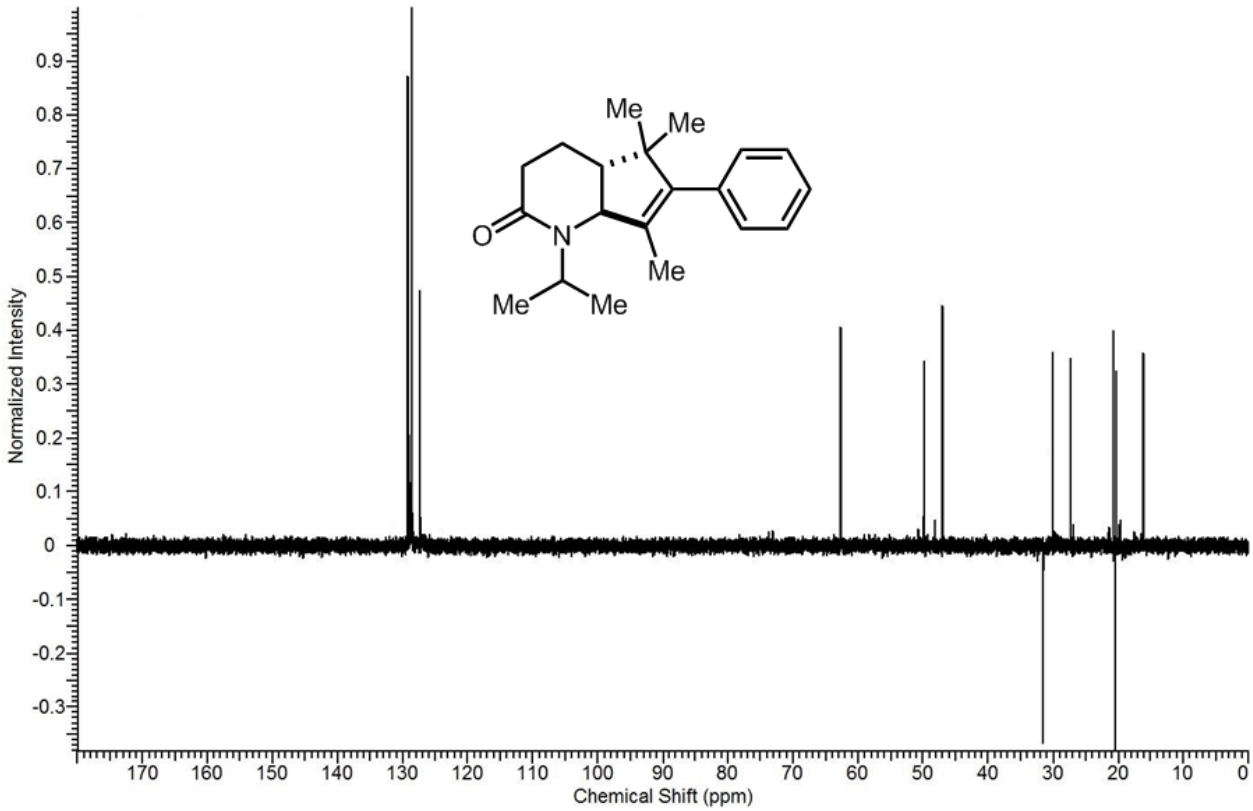


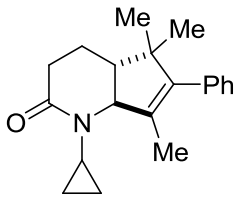
1g; 75%, 92:8 dr

Prepared from alcohol **2g** (158 mg, 0.50 mmol) using General Procedure A. Oily substance. Yield = 113.4 mg, 75%. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7 7.30 (m, 2H), 7.27 – 7.14 (m, 3H), 4.25 to 4.18 (m, 2H), 2.46 to 2.31 (m, 2H), 2.24 to1.75 (m, 5H), 1.43 to 1.40 (m, 1H), 1.26 to 1.09 (m, 12H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.14, 139.66, 137.92, 128.86, 128.62, 126.97, 126.64, 124.07, 63.33, 48.34, 46.64, 44.37, 29.34, 28.44, 22.80, 19.82, 17.24. **HRMS-EI^{+}** (*m/z*): calc'd for $C_{20}H_{27}NO$ 297.2093; found 297.2096.



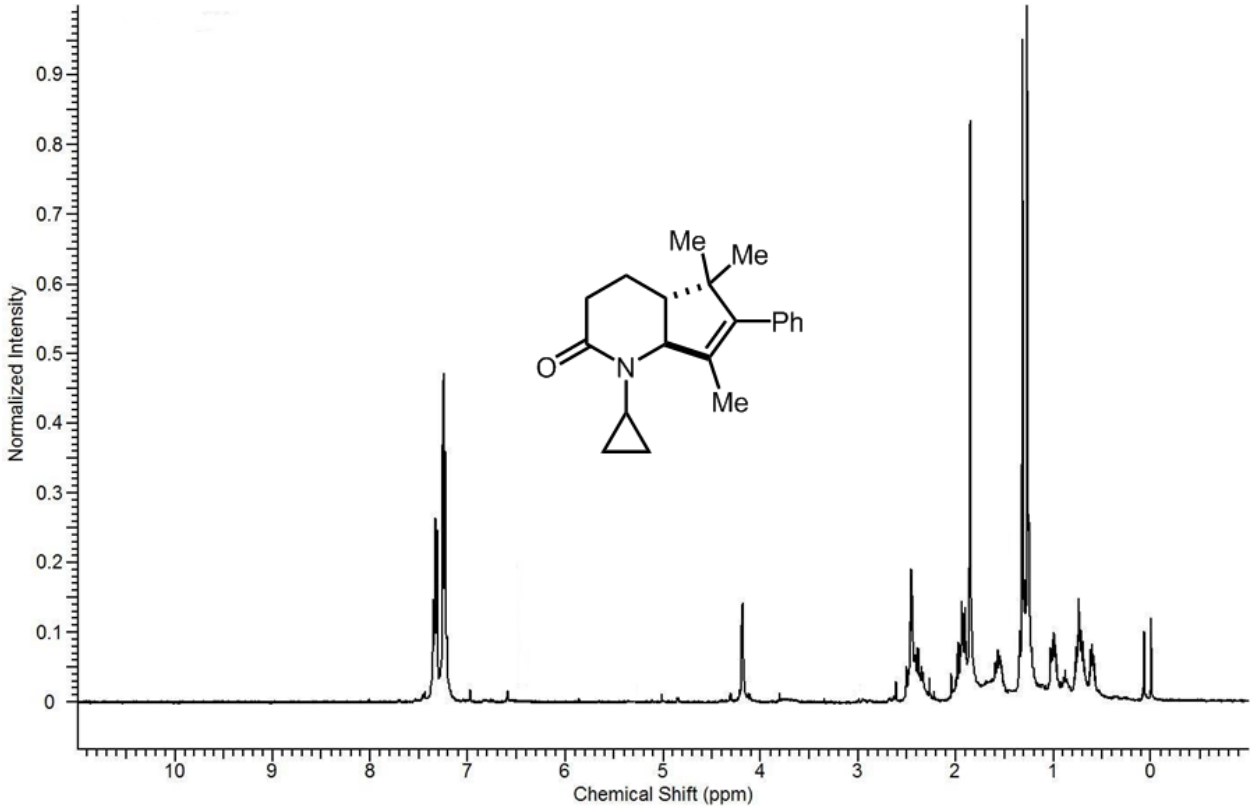

Normalized Intensity
Chemical Shift (ppm)
Me
Me
O
N
Me
Me
Me

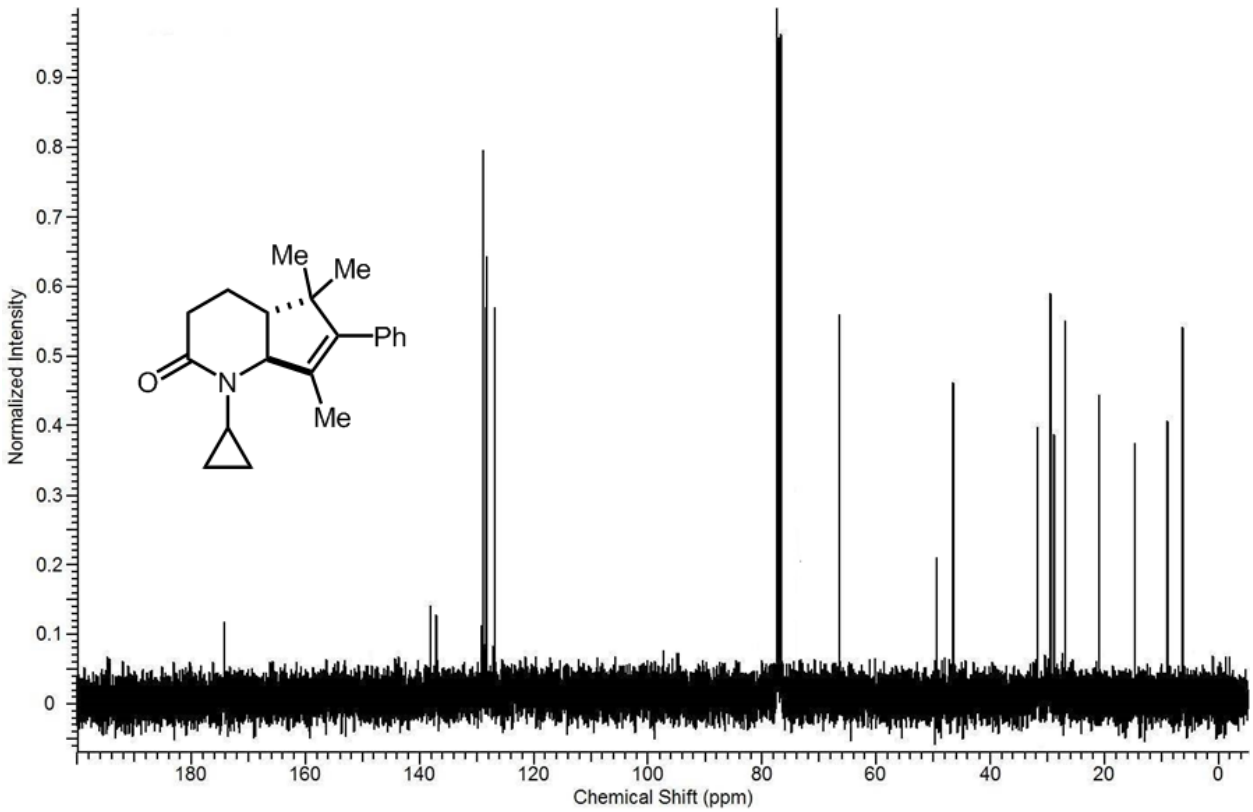

Normalized Intensity
Chemical Shift (ppm)
Me
Me
O
N
Me
Me
Me

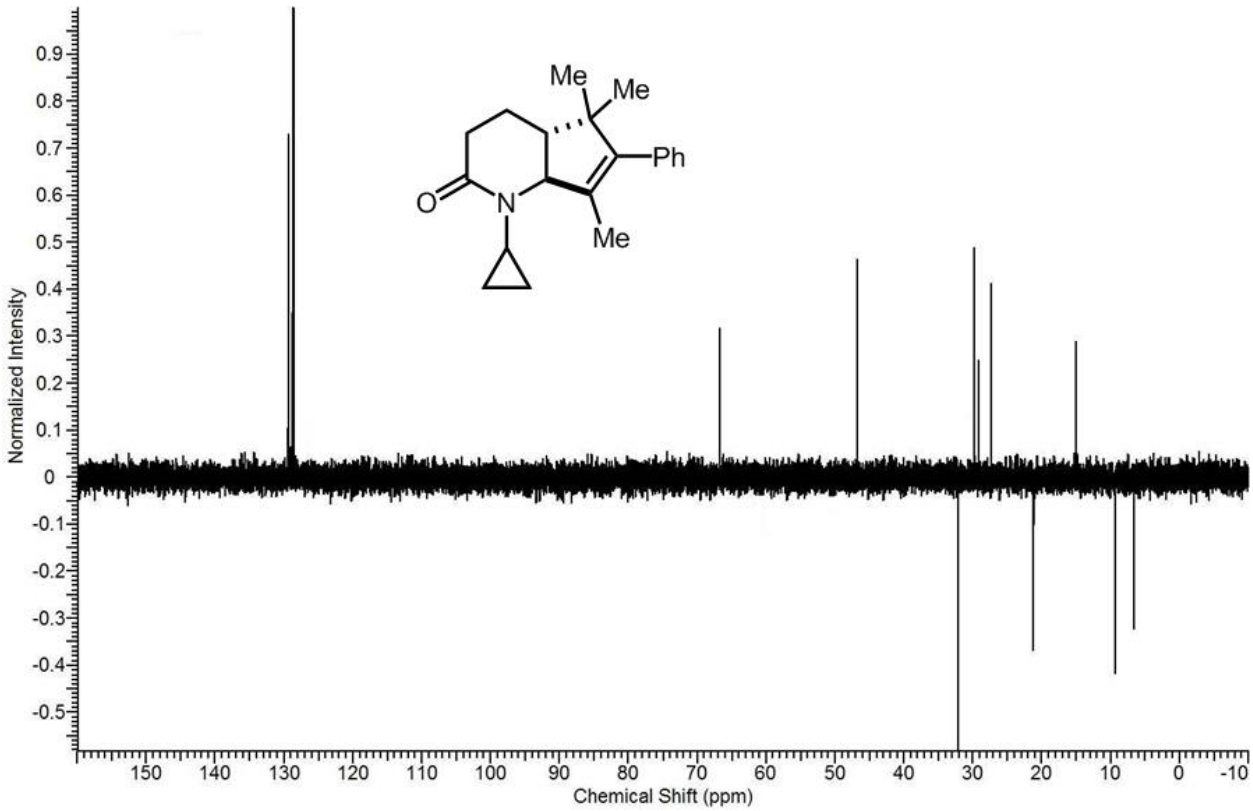


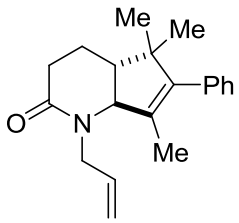
1h; 77%, 95:5 dr

Prepared from alcohol **2h** (157 mg, 0.50 mmol) using General Procedure A. Oily substance. Yield = 113.6 mg, 77%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.46 to 7.21 (m, 5H), 4.21 (d, 1H), 2.49 to 2.22 (m, 2H), 1.98 to 1.85 (m, 5H), 1.60 to 1.52 (m, 1H), 1.35 to 1.22 (overlapping singlets, 6H), 0.98 to 0.86 (m, 1H), 0.79 to 0.56 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.2, 138.1, 137.1, 128.9, 128.7, 128.4, 128.3, 126.8, 66.4, 49.2, 46.5, 31.8, 30.3, 29.4, 28.8, 26.9, 20.9, 14.7, 9.0, 6.3. **HRMS-EI$^+$** (*m/z*): calc'd for $C_{20}H_{25}NO$ 295.1936; found 295.1942.



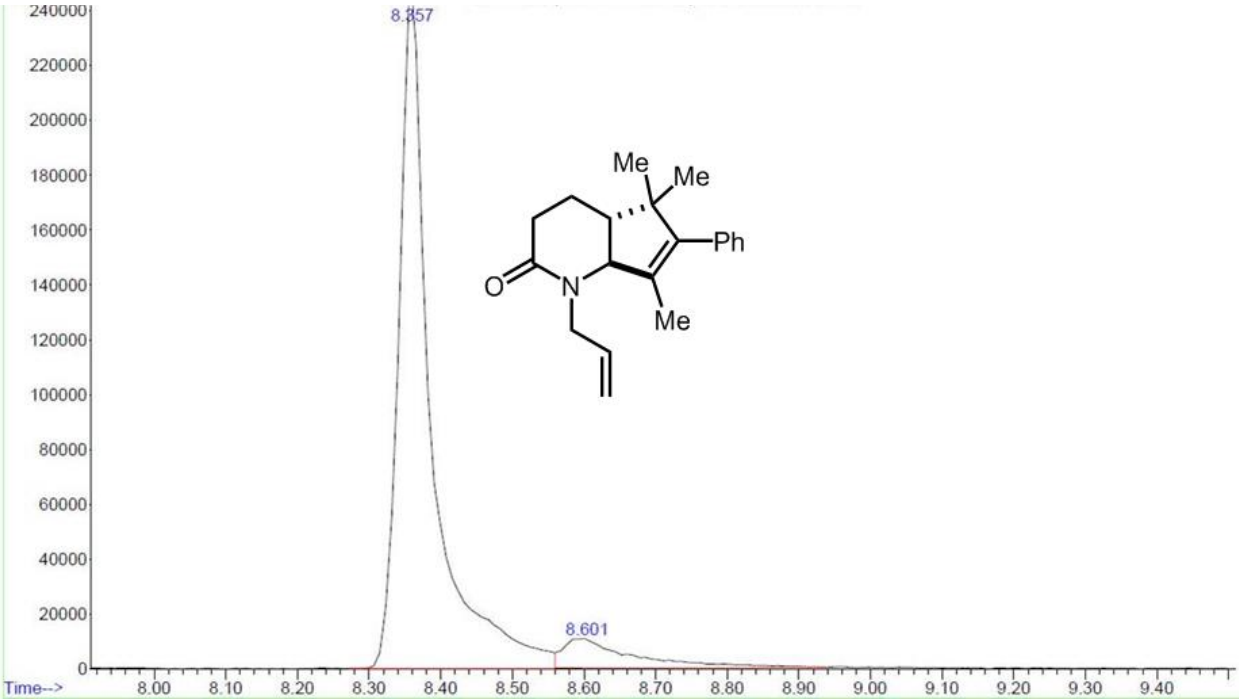

Me
Me
Ph
O
N
Me
Normalized Intensity
Chemical Shift (ppm)

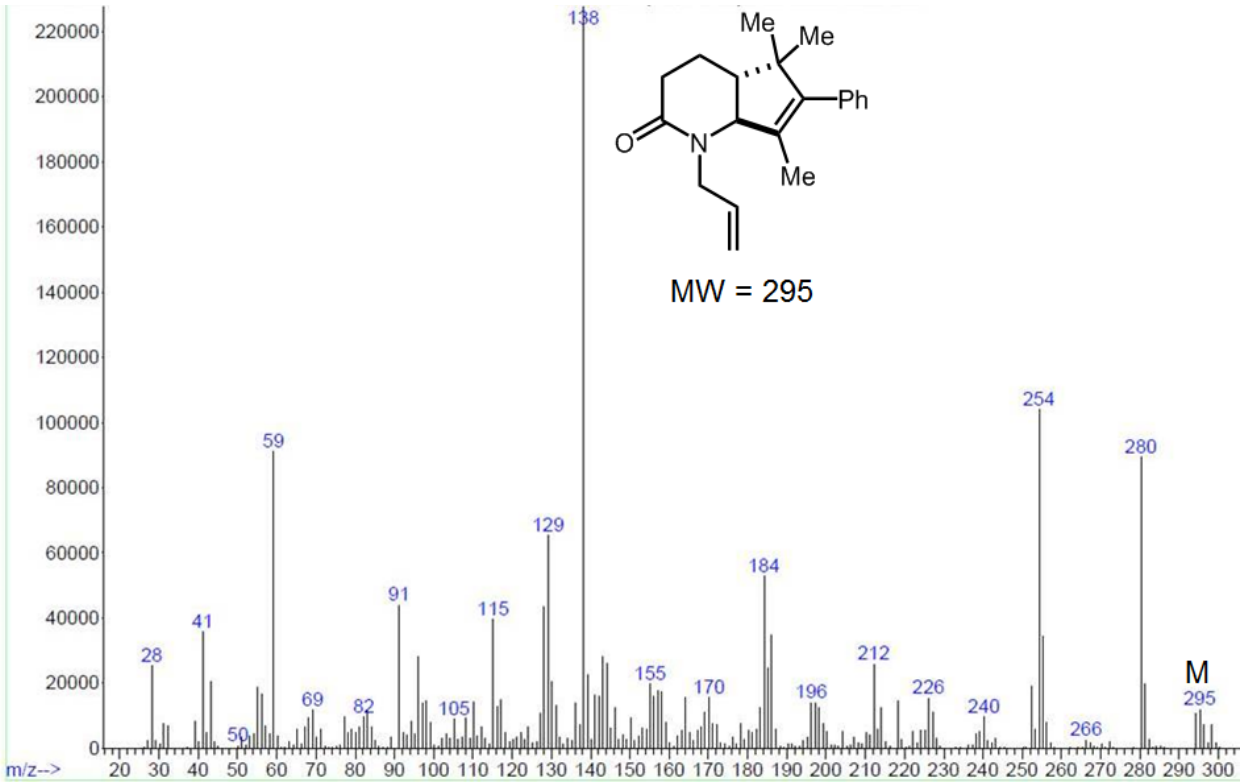

Me
Me
Ph
O
N
Me
Normalized Intensity
Chemical Shift (ppm)

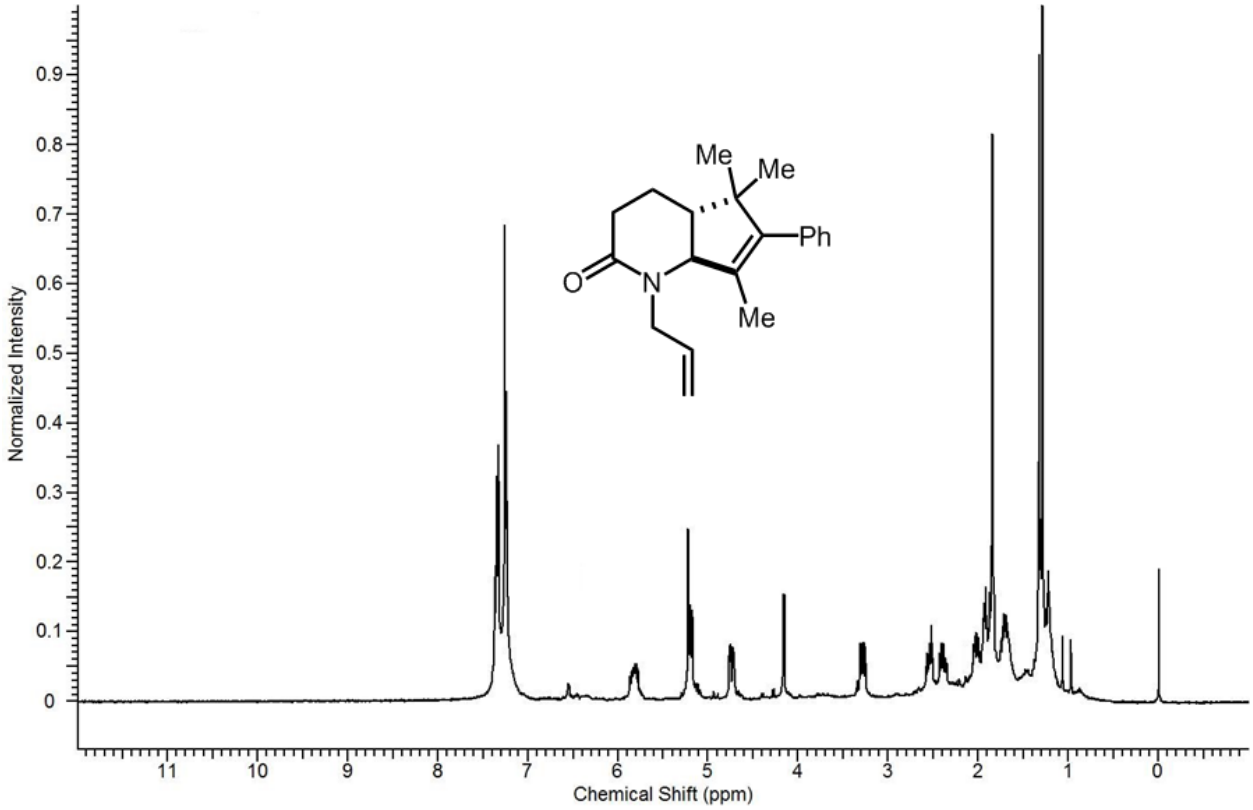


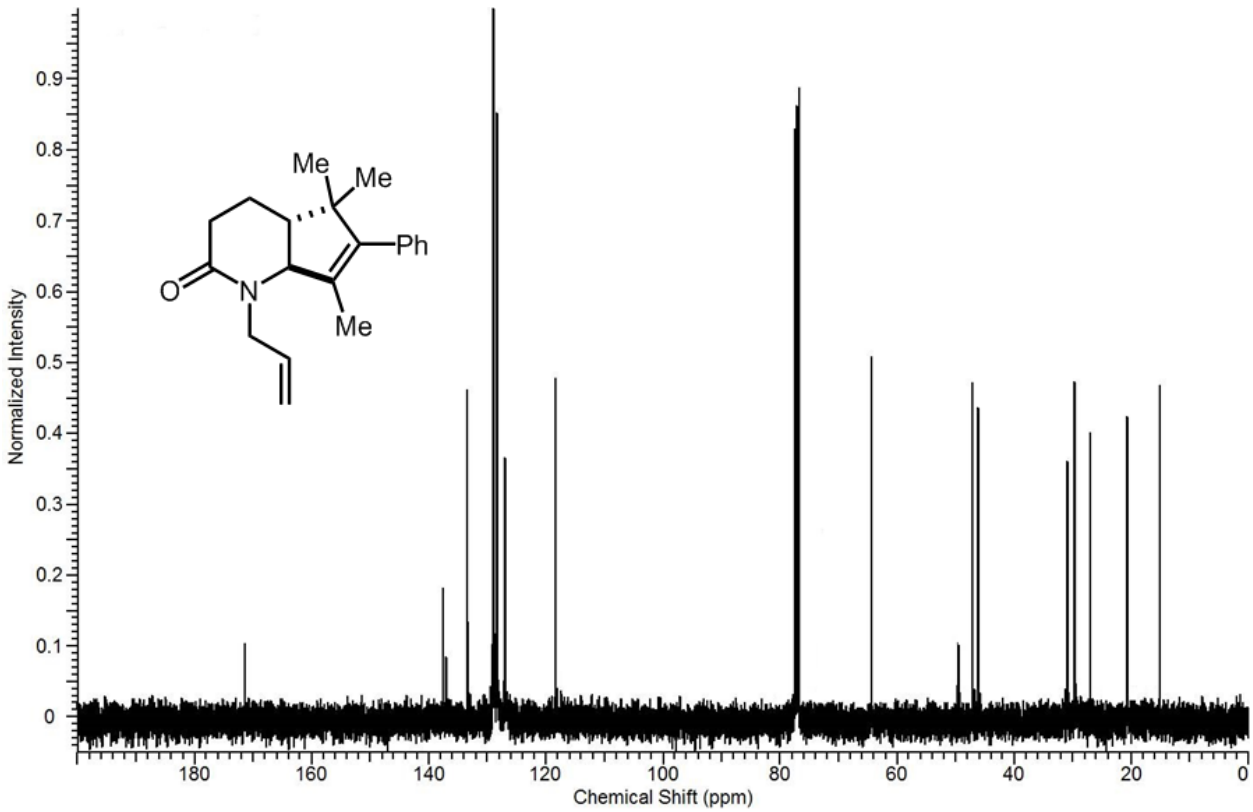
1i; 73%, 95:5 dr

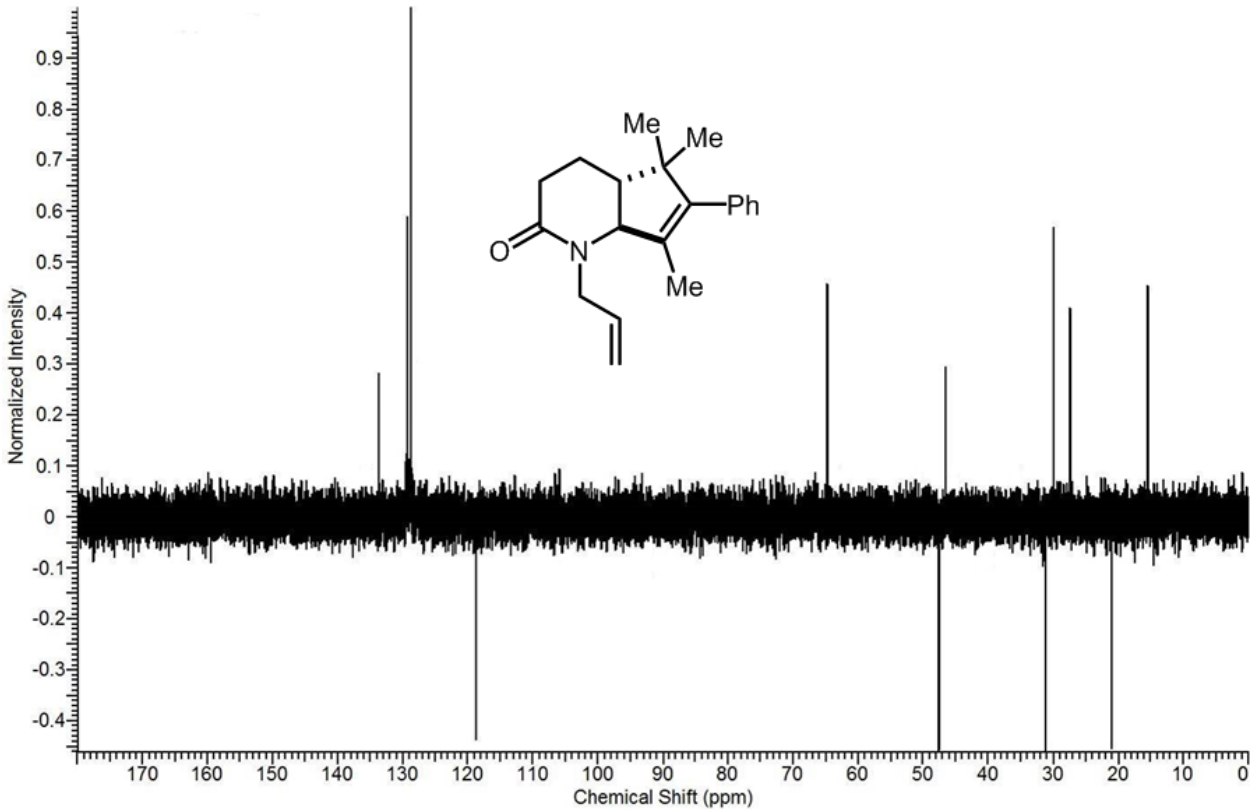
Prepared from alcohol **2i** (157 mg, 0.50 mmol) using General Procedure A. Oily substance. Yield = 108 mg, 73%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 to 7.22 (m, 5H), 5.90 – 5.72 (m, 1H), 5.28 – 5.06 (m, 2H), 4.74 (dd, *J* = 15.1, 4.8 Hz, 1H), 4.16 (d, *J* = 4.4 Hz, 1H), 3.29 to 3.25 (dd, 1H), 2.52 to 2.14 (m, 2H), 1.95 to 1.62 (m, 9H), 1.32 to 1.21 (two overlapping s, 6H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.4, 137.5, 136.9, 133.3, 130.5, 129.0, 128.9, 128.7, 128.6, 128.3, 127.2, 126.9, 118.3, 65.0, 49.1, 46.7, 45.8, 31.2, 29.6, 25.8, 20.7, 15.1. **HRMS-EI^{+}** (*m/z*): calc'd for $C_{20}H_{25}NO$ 295.1936; found 295.1940.

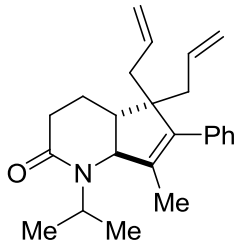



MW = 295
M
295
m/z-->


Normalized Intensity
Chemical Shift (ppm)

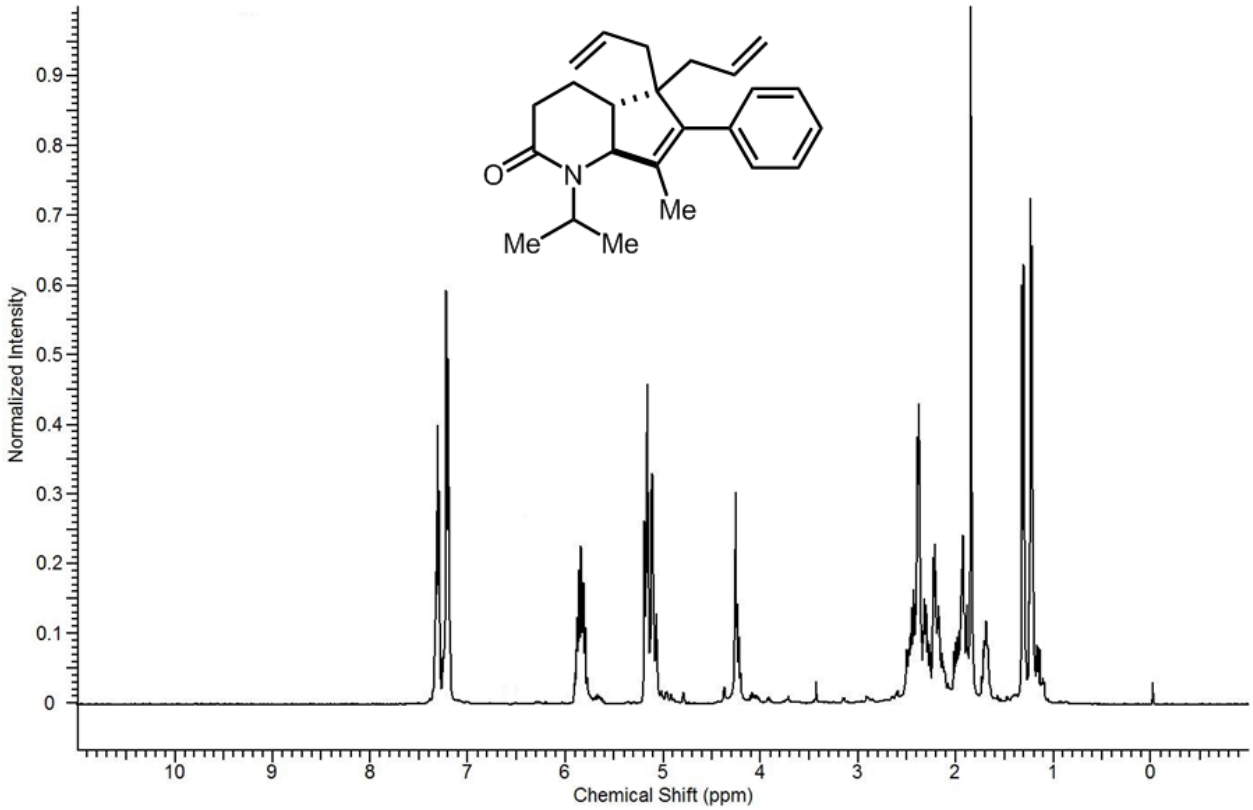

Me
Me
Ph
O
N
Me
Normalized Intensity
Chemical Shift (ppm)

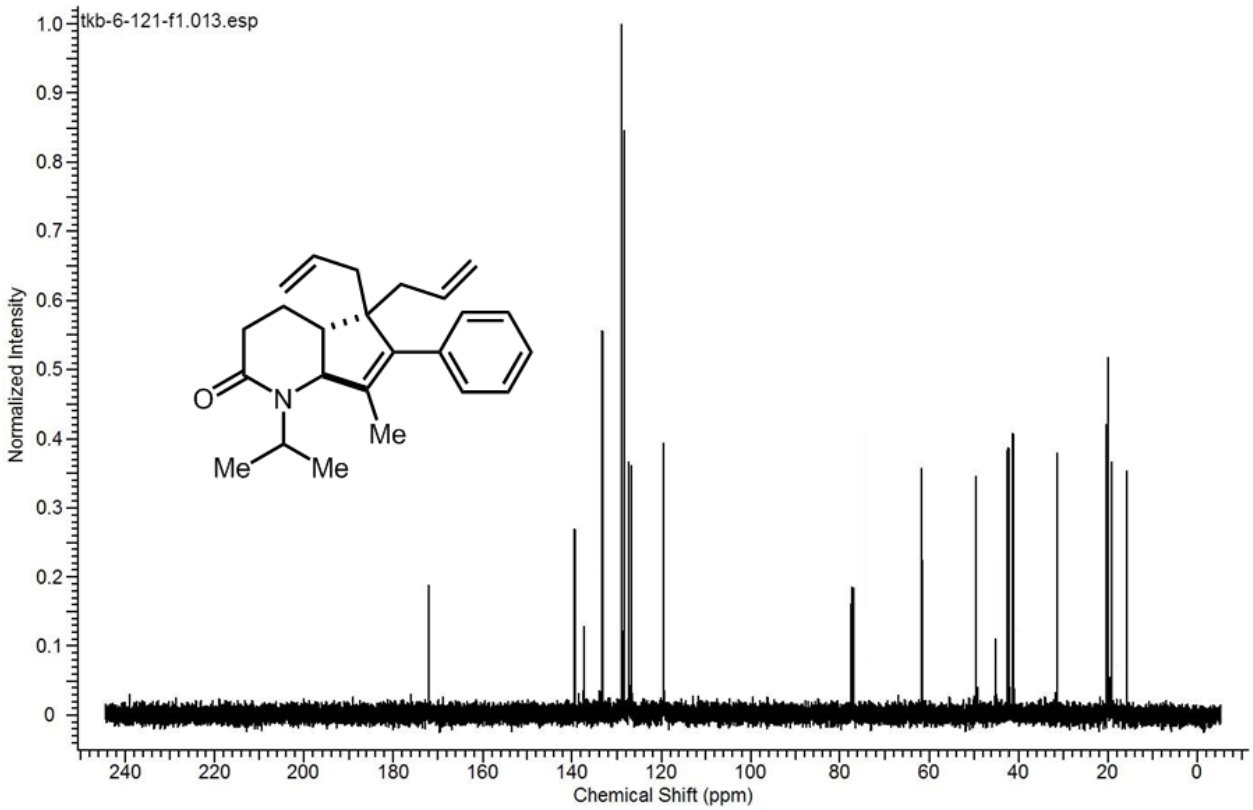

Me
Me
Ph
O
N
Me
Normalized Intensity
Chemical Shift (ppm)



1j; 78%, 92:8 dr

Prepared from alcohol **2j** (92 mg, 0.25 mmol) using General Procedure A. Oily substance. Yield = 68 mg, 78%. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.34 to 7.18 (m, 5H), 5.89 to 5.76 (m, 2H), 5.16 to 4.92 (m, 4H), 4.26 to 4.19 (m, 2H), 2.51 to 1.65 (m, 13H), 1.28 to 1.11 (m, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 172.18, 141.10, 138.54, 136.84, 128.94, 128.53, 127.29, 127.00, 126.69, 125.56, 119.59, 118.74, 61.66, 49.28, 44.29, 41.96, 41.20, 40.17, 31.53, 21.27, 19.71, 19.20, 17.23. **HRMS-EI^{+}** (*m/z*): calc'd for $C_{24}H_{31}NO$ 349.2406; found 349.2411.

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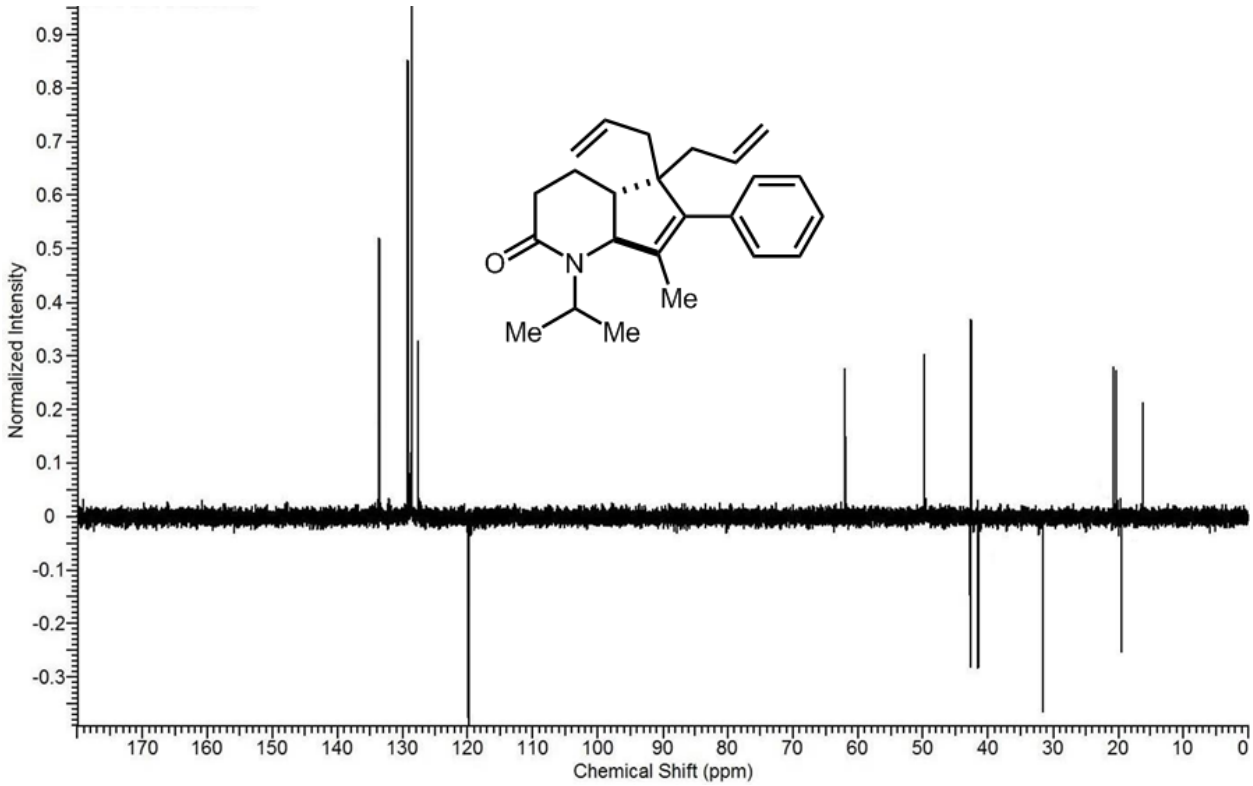
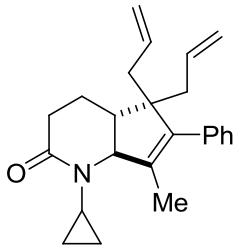
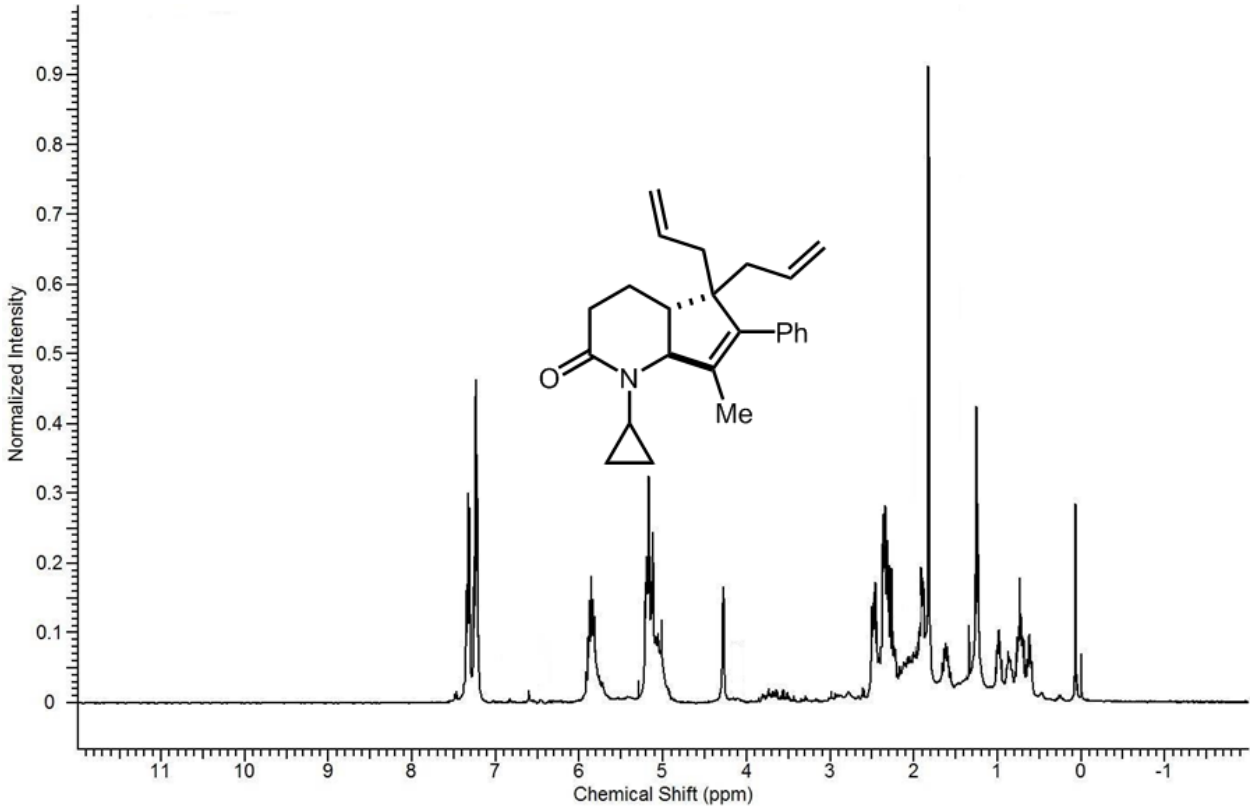
Normalized Intensity
Chemical Shift (ppm)
O
N
Me
Me
Me

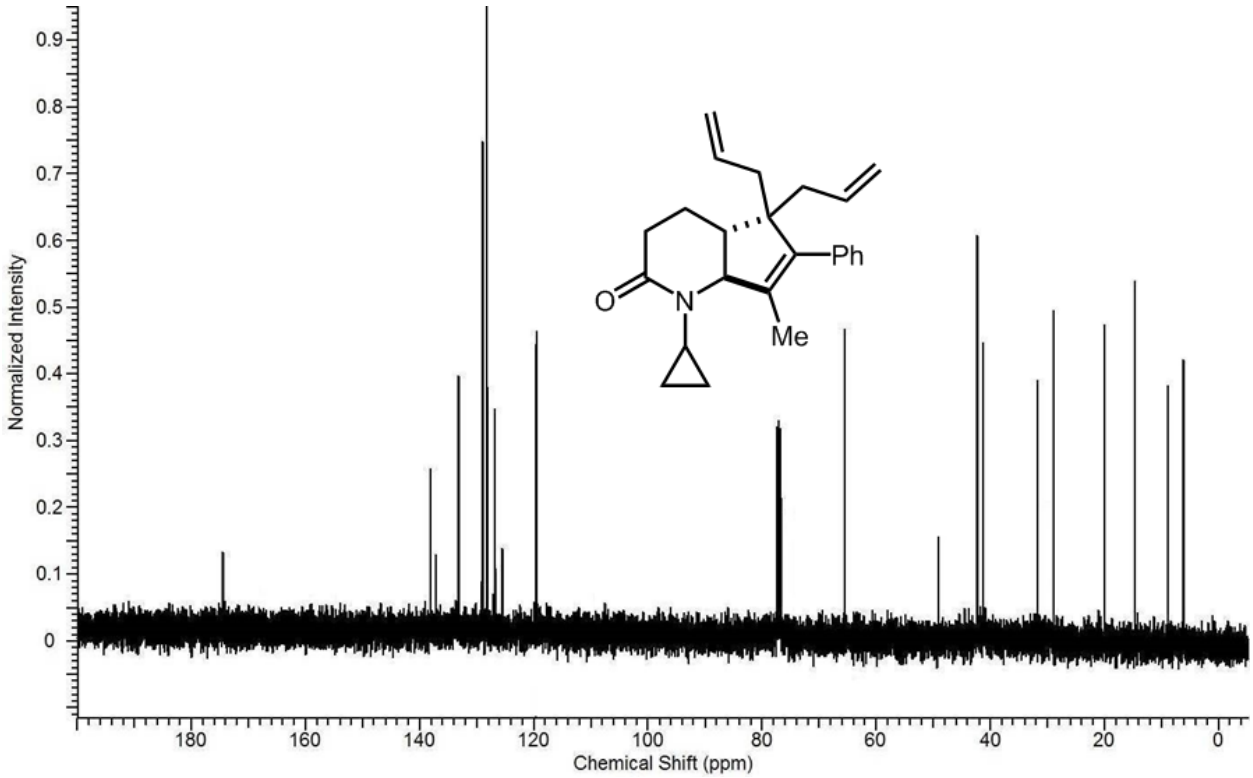

Normalized Intensity
Chemical Shift (ppm)
O
N
Me
Me
Me

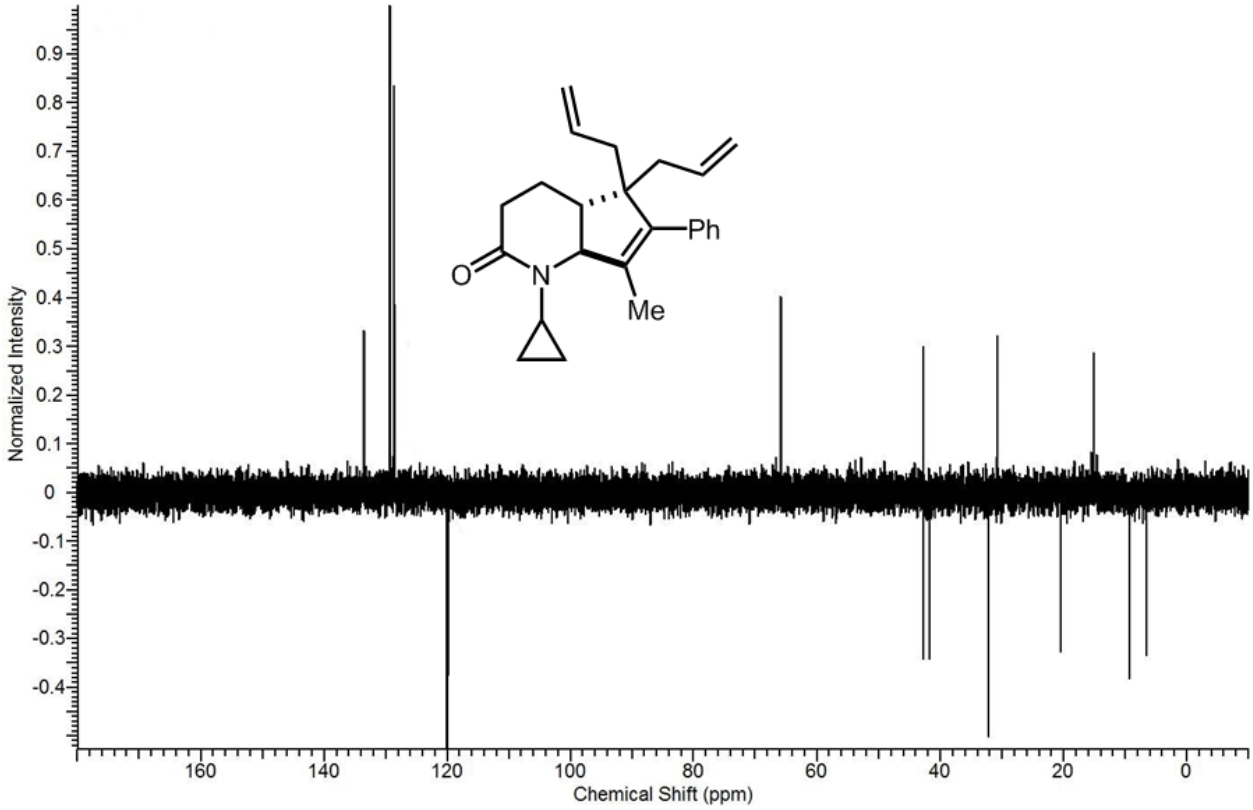


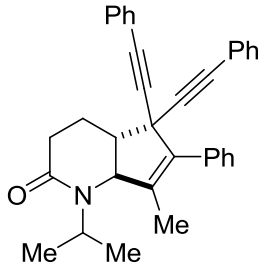
1k; 70%, 95:5 dr

Prepared from alcohol **2k** (183 mg, 0.50 mmol) using General Procedure A. Oily substance. Yield = 121.5 mg, 70%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.49 to 7.18 (m, 5H), 5.92 to 5.72 (m, 2H), 5.30 to 4.92 (m, 4H), 4.30 to 4.27 (br s, 1H), 2.59 to 0.60 (m, 17H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.5, 138.0, 137.1, 133.1, 133.1, 129.0, 128.9, 128.8, 128.2, 128.1, 126.8, 125.5, 119.6, 65.5, 49.2, 41.3, 40.9, 31.8, 30.3, 29.7, 20.0, 14.7, 8.9, 6.1. **HRMS-EI^{+}** (*m/z*): calc'd for $C_{24}H_{29}NO$ 347.2249; found 347.2243.



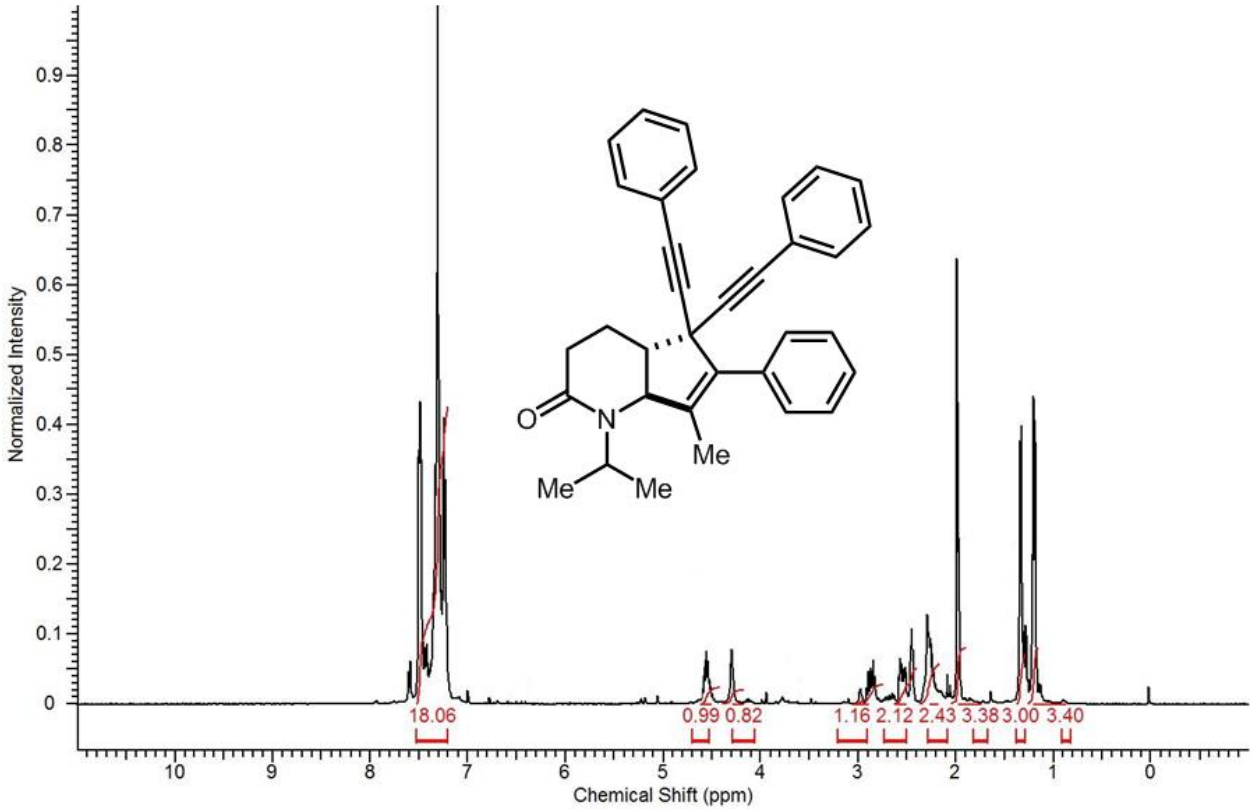

Normalized Intensity
Chemical Shift (ppm)
Ph
Me
O
N

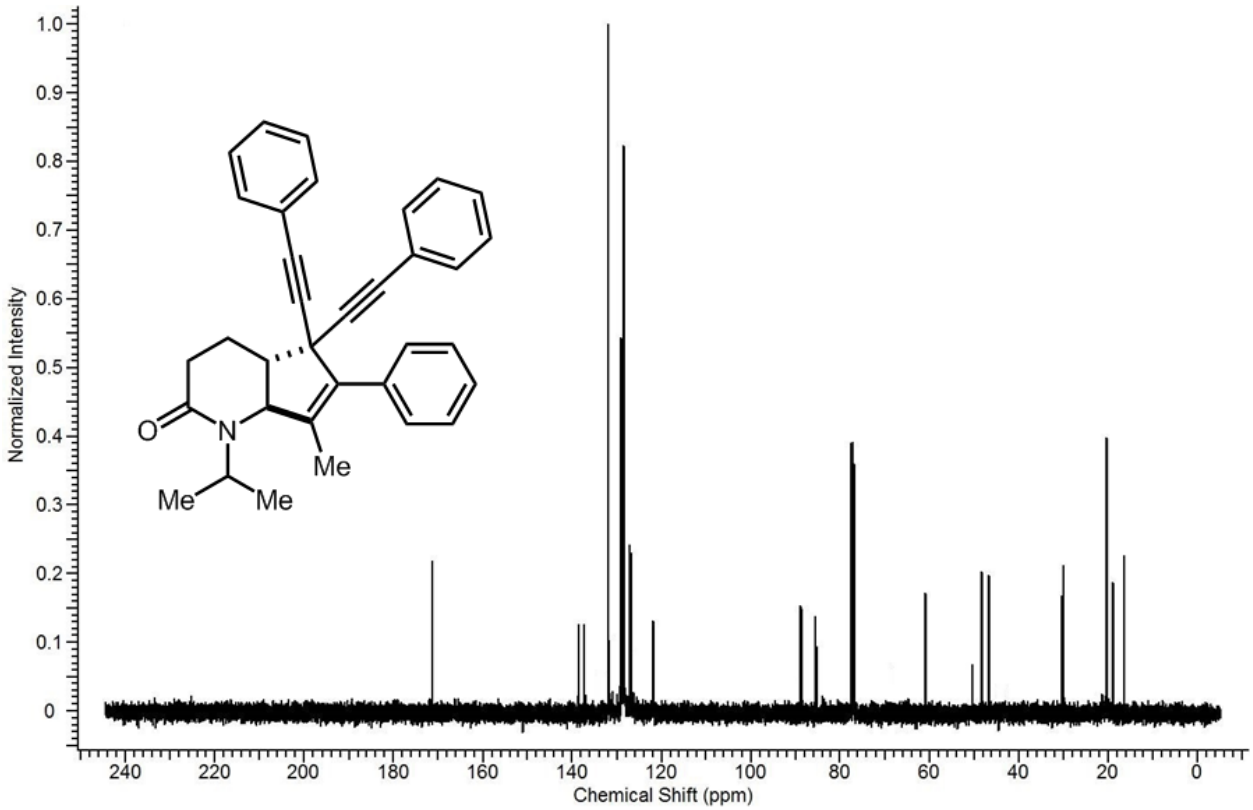

Normalized Intensity
Chemical Shift (ppm)
Ph
Me
O
N

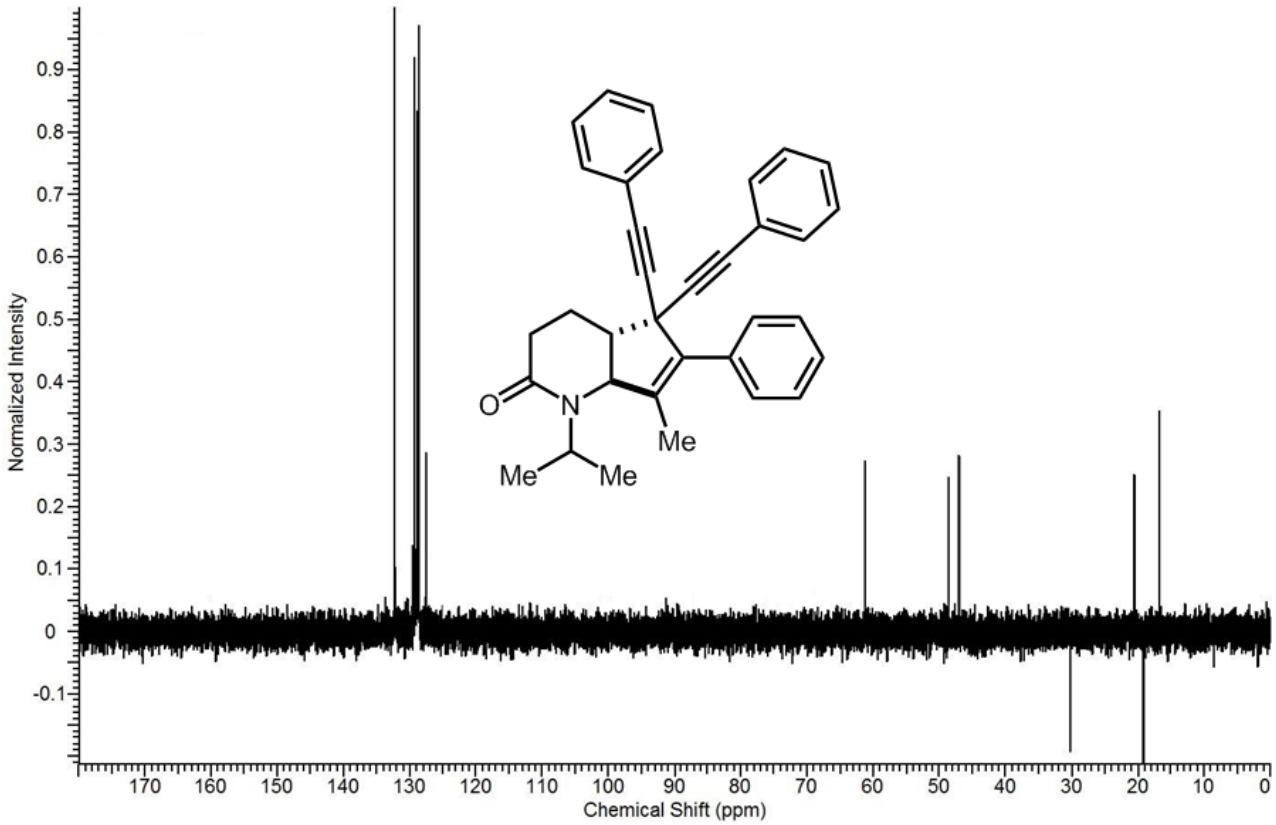


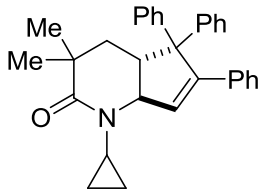
1l; 83%, 92:8 dr

Prepared from alcohol **2l** (122 mg, 0.25 mmol) using General Procedure A. Oily substance. Yield = 97.5 mg, 83%. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.52 to 7.14 (m, 15H), 4.69 to 4.52 (m, 1H), 4.43 to 4.37 (m, 1H), 2.89 to 2.85 (m, 1H), 2.59 to 2.46 (m, 2H), 2.33 to 2.11 (m, 2H), 1.96 (s, 3H), 1.34 to 1.19 (m, 6H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.71, 137.52, 136.83, 130.72, 129.35, 129.29, 128.98, 128.69, 128.30, 127.52, 126.99, 126.79, 121.12, 88.84, 83.64, 60.77, 48.21, 46.84, 43.32, 30.72, 20.63, 19.49, 18.43. **HRMS-EI^{+}** (*m/z*): calc'd for $C_{34}H_{31}NO$ 469.2406; found 469.2401.



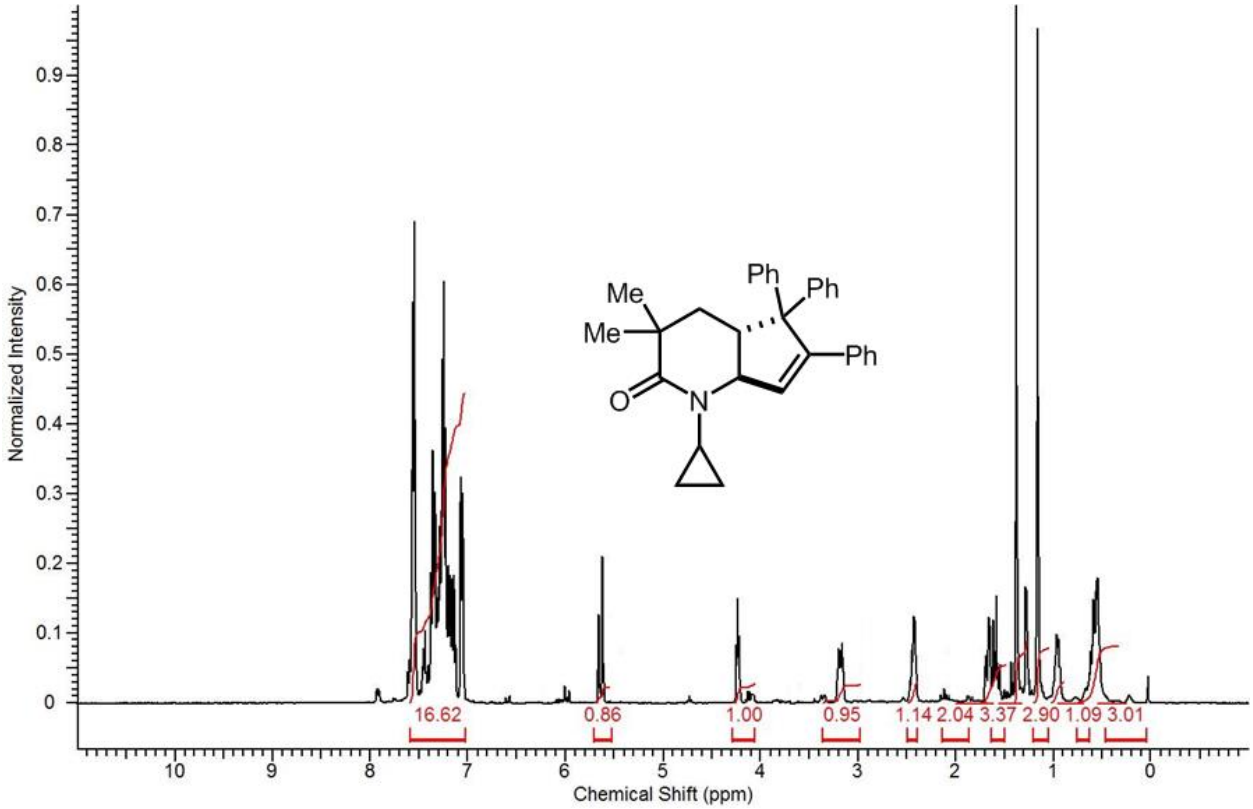

Normalized Intensity
Chemical Shift (ppm)
O
N
Me
Me
Me

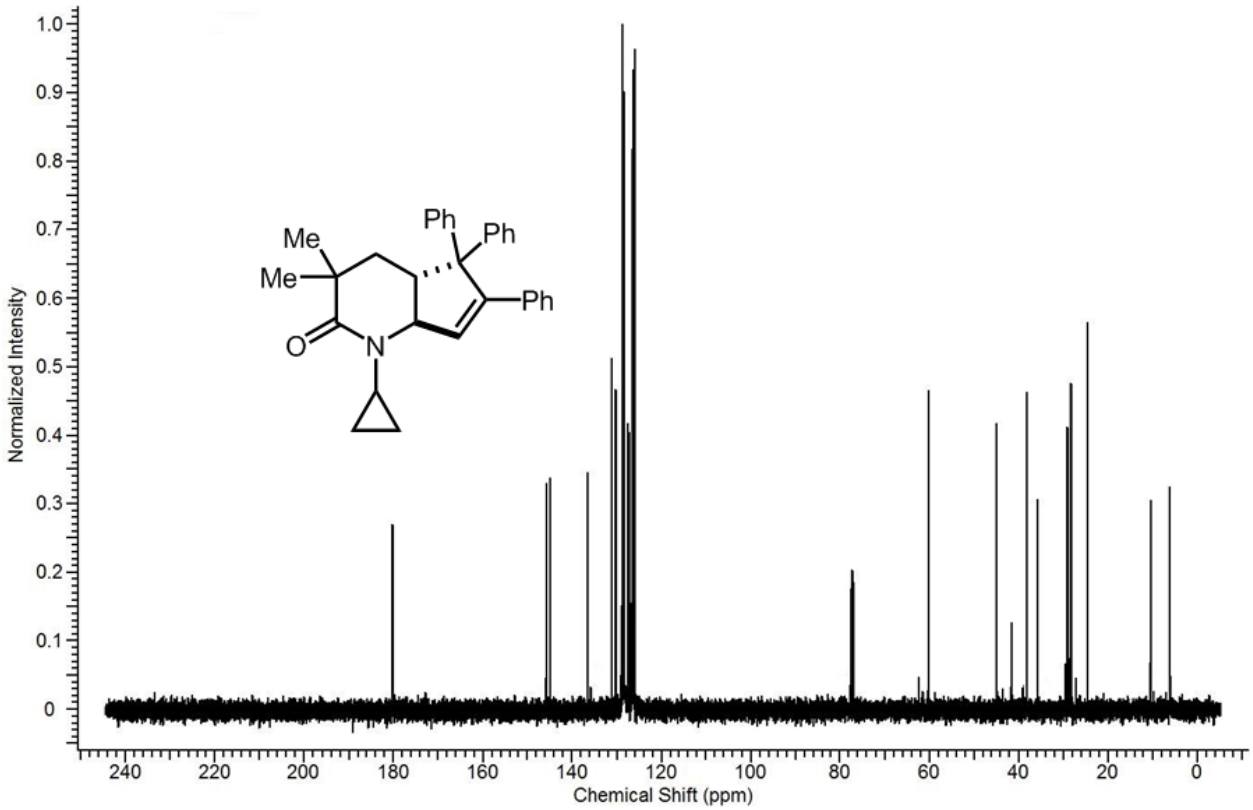

Normalized Intensity
Chemical Shift (ppm)
O
N
Me
Me
Me



1m; 83%, 90:10 dr

Prepared from alcohol **2m** (113 mg, 0.25 mmol) using General Procedure A. Oily substance. Yield = 90 mg, 83%. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.61 to 7.05 (m, 15H), 5.51 to 5.47 (dd, 1H), 4.23 to 4.20 (t, 1H), 3.21 to 3.15 (dd, 1H), 2.45 to 2.40 (m, 1H), 1.68 to 1.53 (m, 2H), 1.38 (s, 3H), 1.28 (s, 3H), 0.97 to 0.91 (m, 1H), 0.67 to 0.49 (m, 3H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 180.28, 145.77, 144.83, 136.63, 129.01, 128.71, 128.61, 128.14, 127.44, 127.12, 126.92, 26.56, 126.42, 126.28, 125.99, 125.02, 124.97, 61.38, 43.50, 40.86, 39.10, 38.13, 29.48, 28.50, 27.23, 24.57, 10.39, 5.96. **HRMS-EI^{+}** (*m/z*): calc'd for $C_{31}H_{31}NO$ 433.2406; found 433.2409.




Normalized Intensity
Chemical Shift (ppm)
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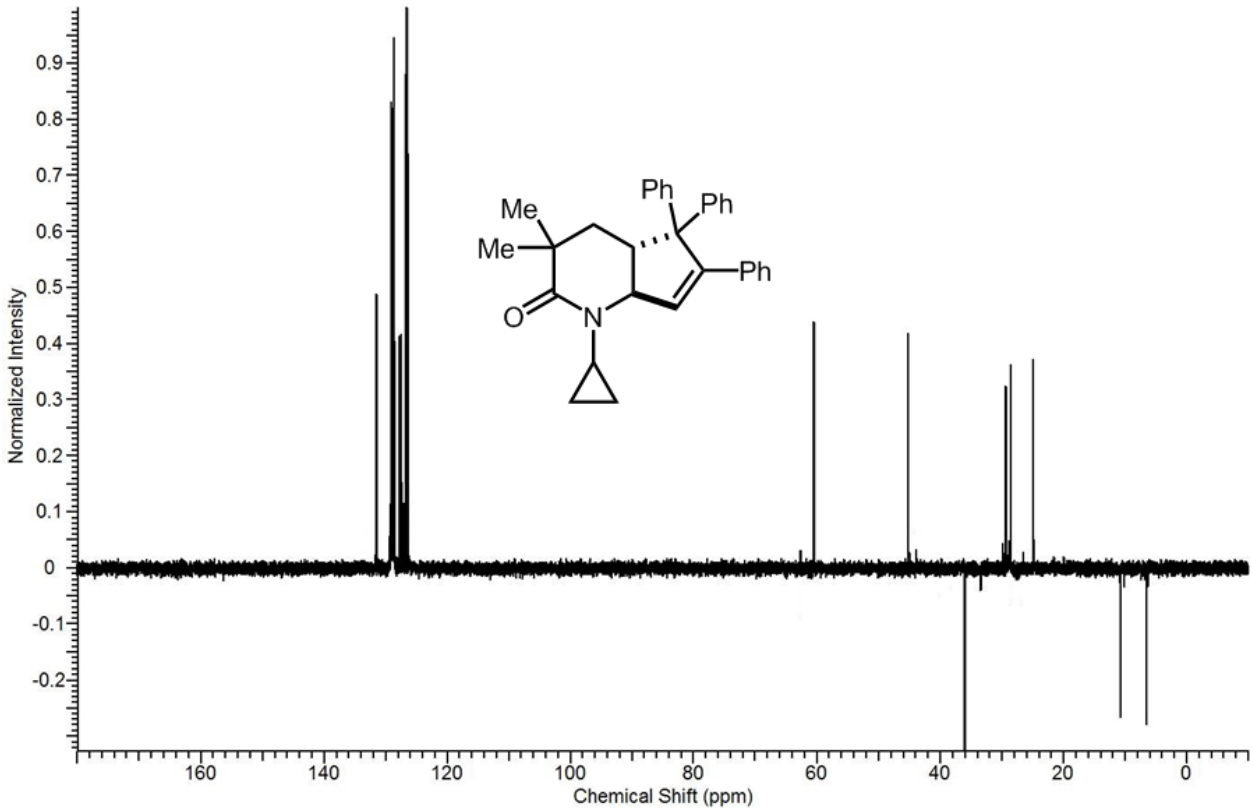
Me
Ph
Ph
Ph
O
N


Normalized Intensity
Chemical Shift (ppm)
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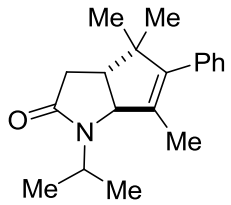
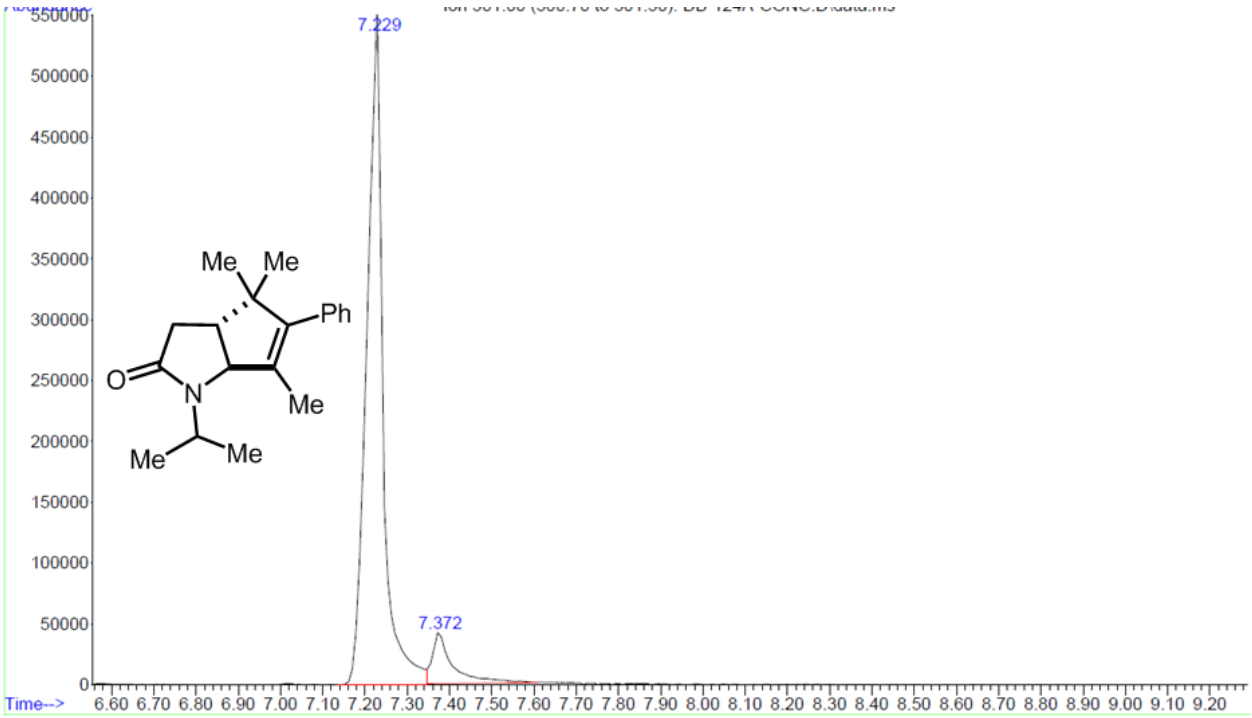
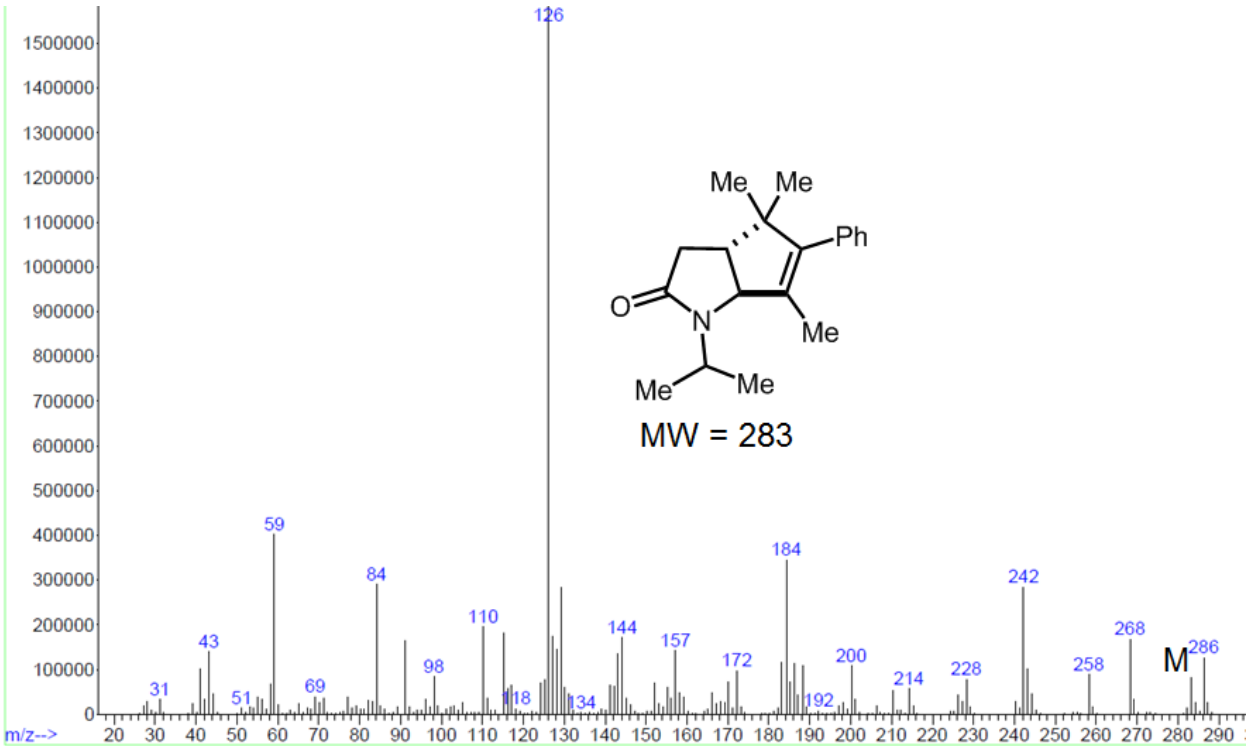
Me
Ph
Ph
Ph
O
N



1n; 80%, 94:6 dr

Prepared from alcohol **2n** (150.6 mg, 0.50 mmol) using General Procedure A. Oily substance. Yield = 113.3 mg, 80%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 to 7.17 (m, 5H), 4.38 to 4.10 (m, 2H), 2.66 to 2.54 (m, 1H), 2.33 to 2.20 (m, 1H), 2.08 to 2.04 (dd, 1H), 1.88 (s, 3H), 1.46 to 0.95 (m, 12H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 174.4, 139.1, 137.1, 129.5, 128.9, 128.9, 128.7, 128.4, 128.4, 128.3, 128.1, 127.7, 127.0, 126.9, 126.8, 125.5, 66.9, 49.1, 47.2, 44.5, 34.7, 27.8, 25.2, 20.3, 18.7, 13.2. **HRMS-EI^{+}** (*m/z*): calc'd for $C_{19}H_{25}NO$ 283.1936; found 283.1941.

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59
84
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43
31
51
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98
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134
144
157
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184
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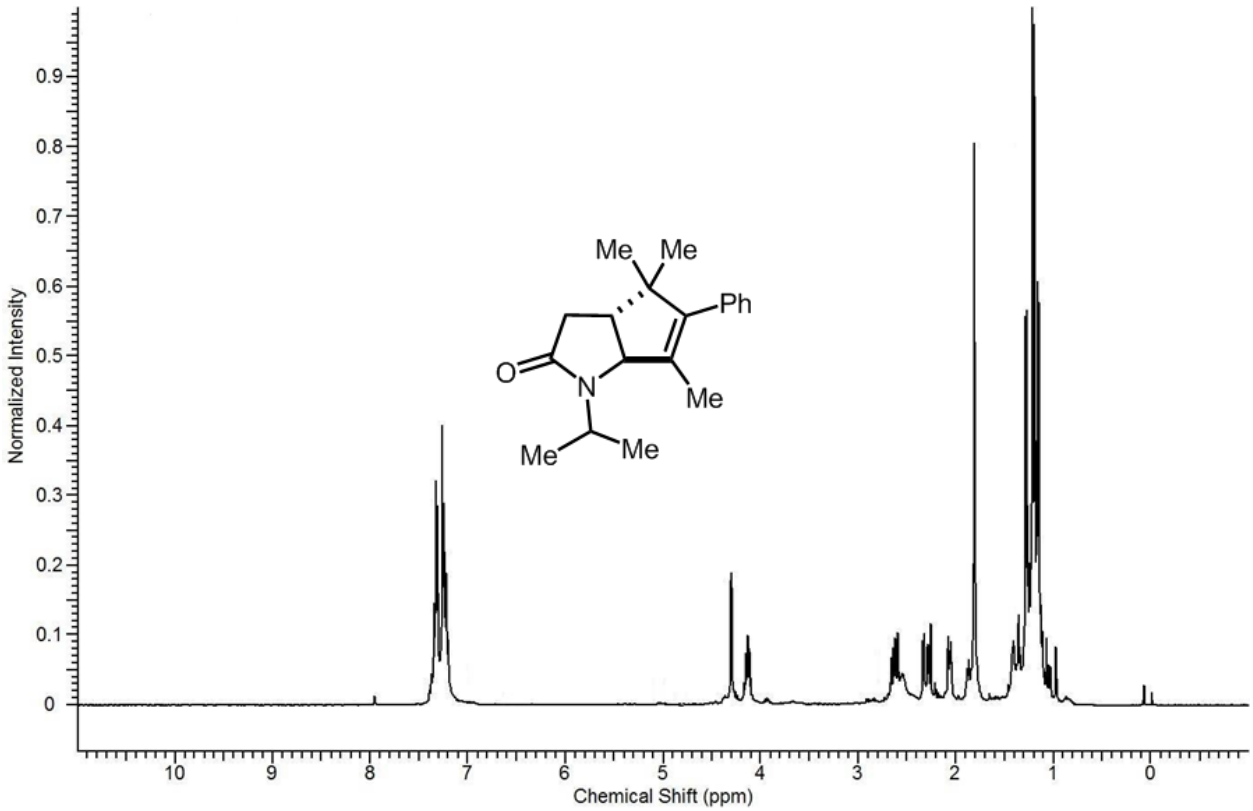
242
258
268
286
M
MW = 283
Me
Me
Ph
O
N
Me
Me
Me
m/z-->


Normalized Intensity
Chemical Shift (ppm)
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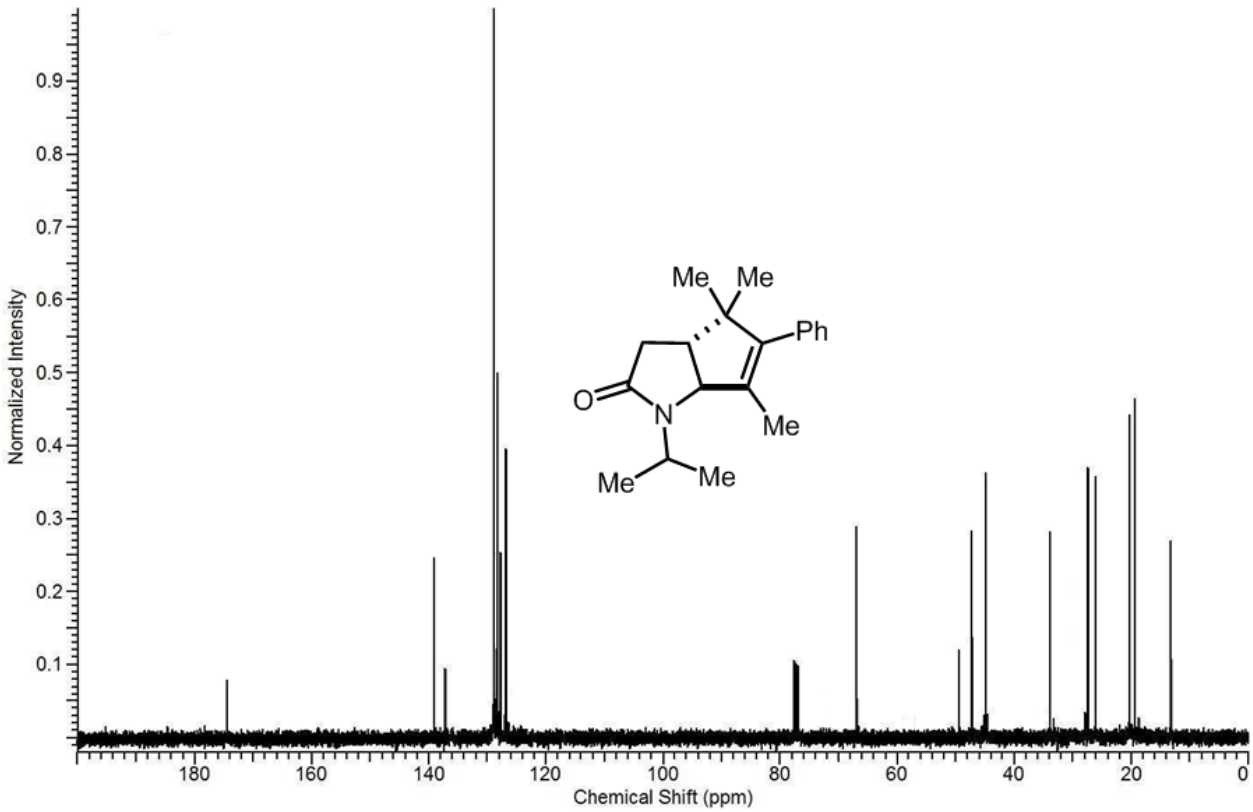
Me
Ph
O
N
Me
Me
Me


Normalized Intensity
Chemical Shift (ppm)
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Ph
O
N
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Me
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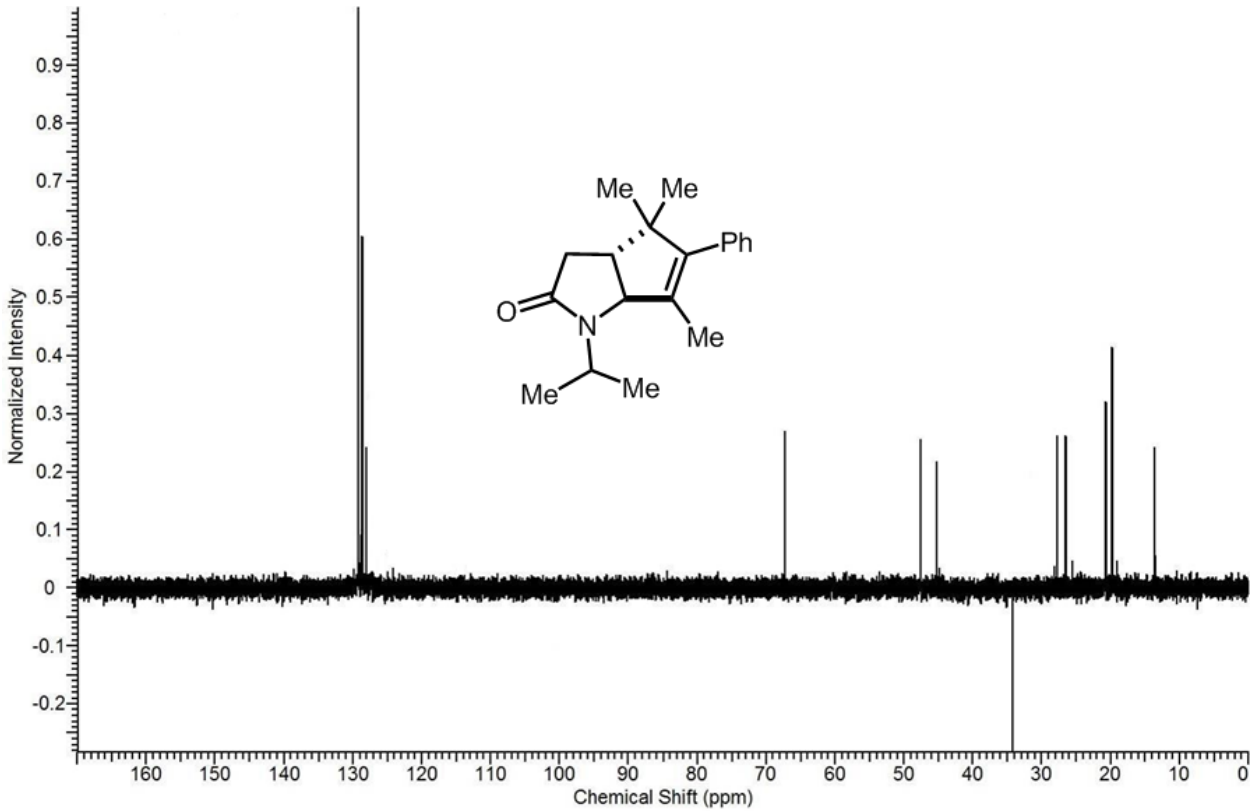
Normalized Intensity
Chemical Shift (ppm)
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Me
Ph
O
N
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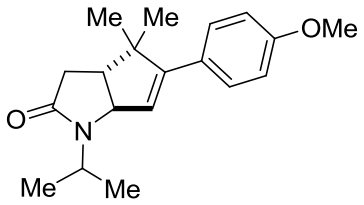
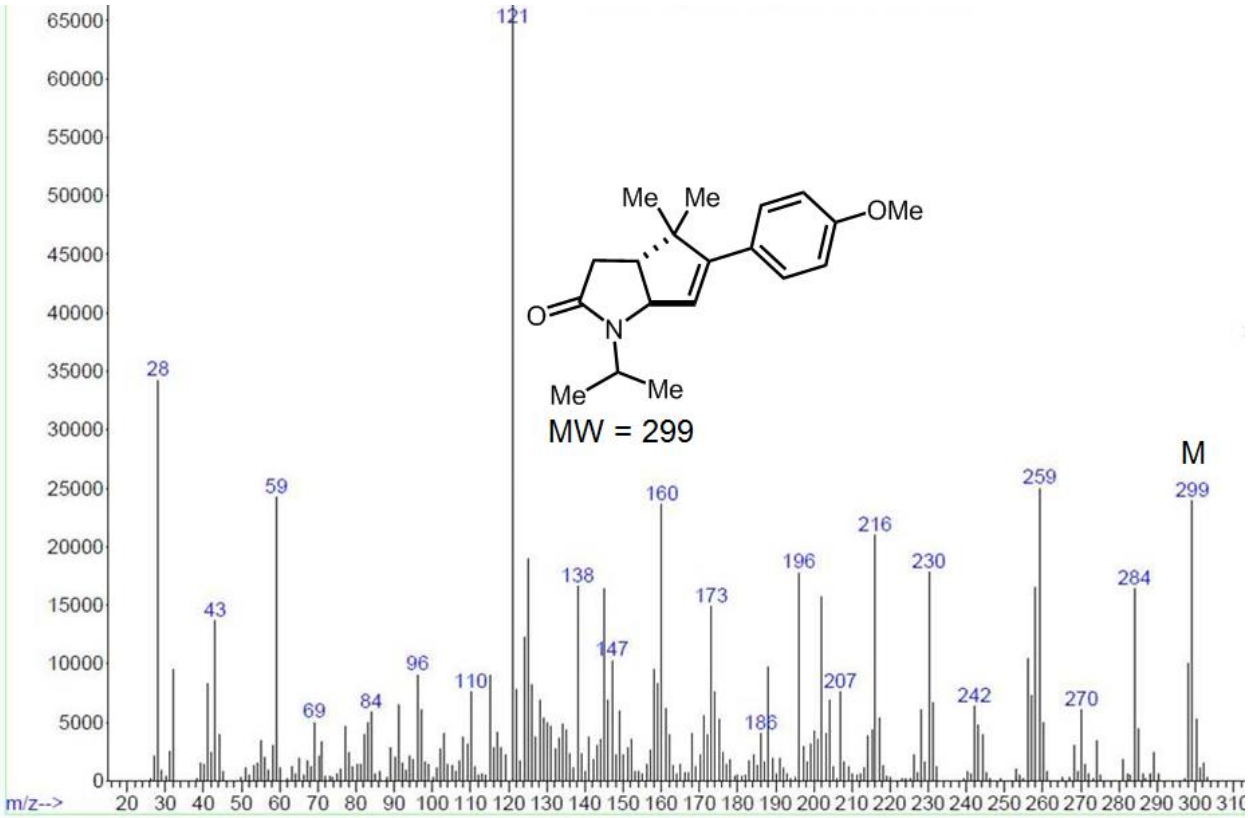
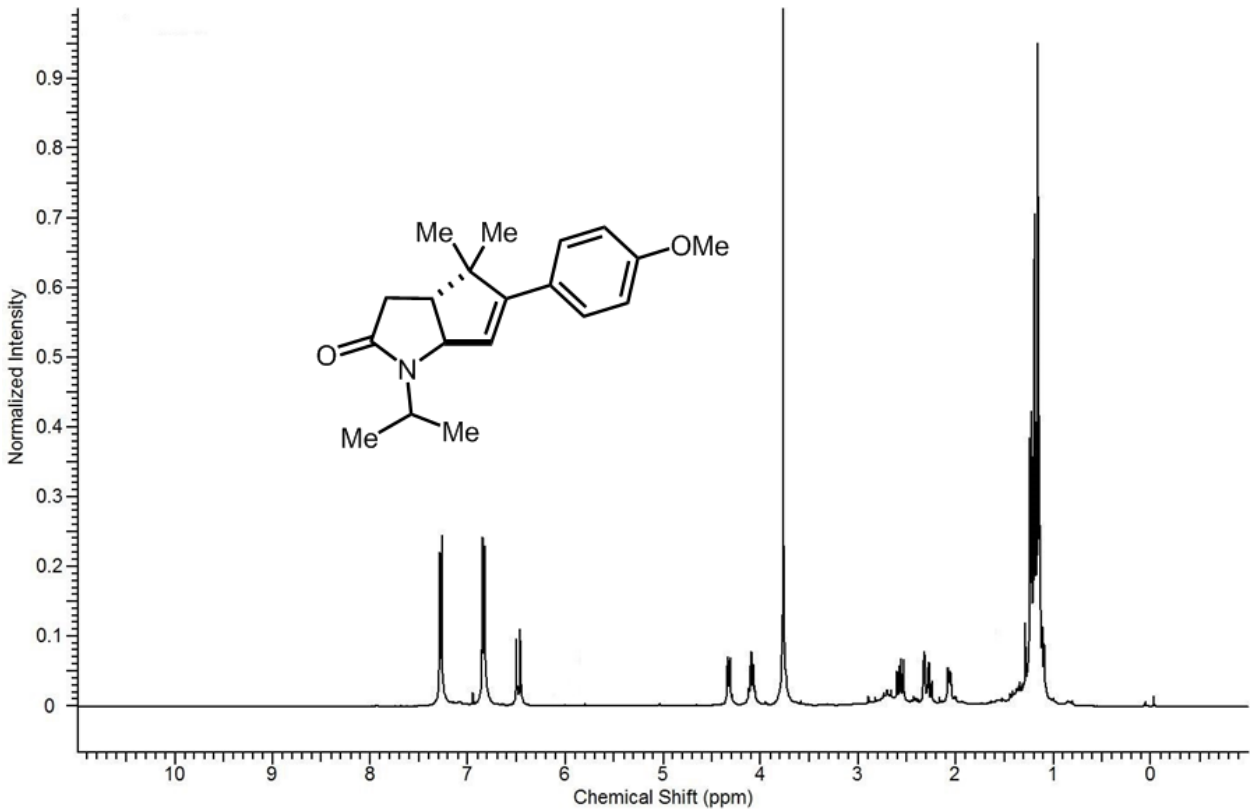
Me
Me



1o; 85%, 92:8 dr

Prepared from alcohol **2o** (158.6 mg, 0.50 mmol) using General Procedure A. Oily substance. Yield = 127 mg, 85%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.24 (d, 2H), 6.85 (d, 2H), 6.48 (d, 1H), 4.33 to 4.28 (dd, 1H), 4.19 to 4.02 (m, 1H), 3.79 (s, 3H), 2.51 to 2.41 (dd, 1H), 2.30 to 2.20 (dd, 1H), 2.04 to 1.91 (m, 1H), 1.35 to 1.04 (m, 12H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 173.7, 159.7, 131.9, 130.7, 130.0, 129.0, 128.7, 127.7, 125.9, 114.1, 61.6, 55.3, 51.4, 49.7, 44.5, 32.2, 27.9, 26.5, 21.6, 19.8. **HRMS-EI$^+$** (*m/z*): calc'd for $C_{19}H_{25}NO_2$ 299.1885; found 299.1891.




Normalized Intensity
Chemical Shift (ppm)
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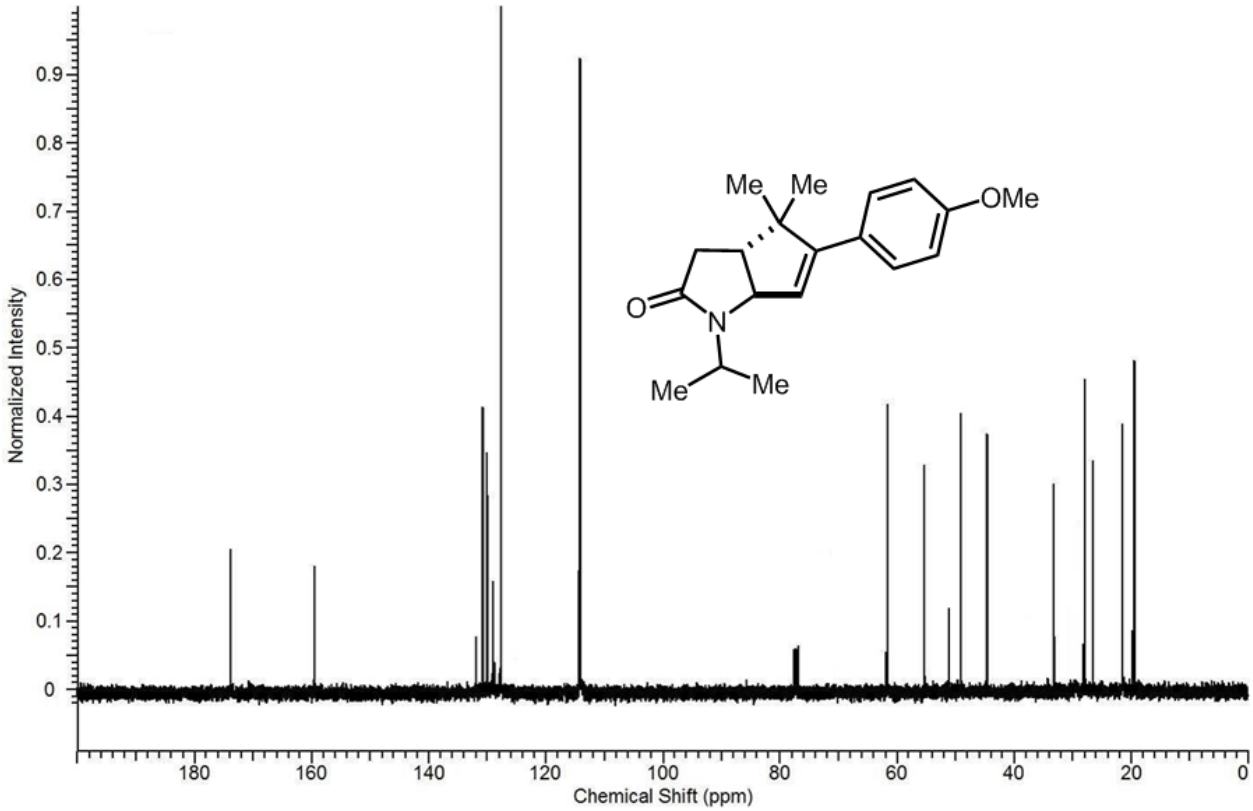
Me
OMe
O
N
Me
Me


Normalized Intensity
Chemical Shift (ppm)
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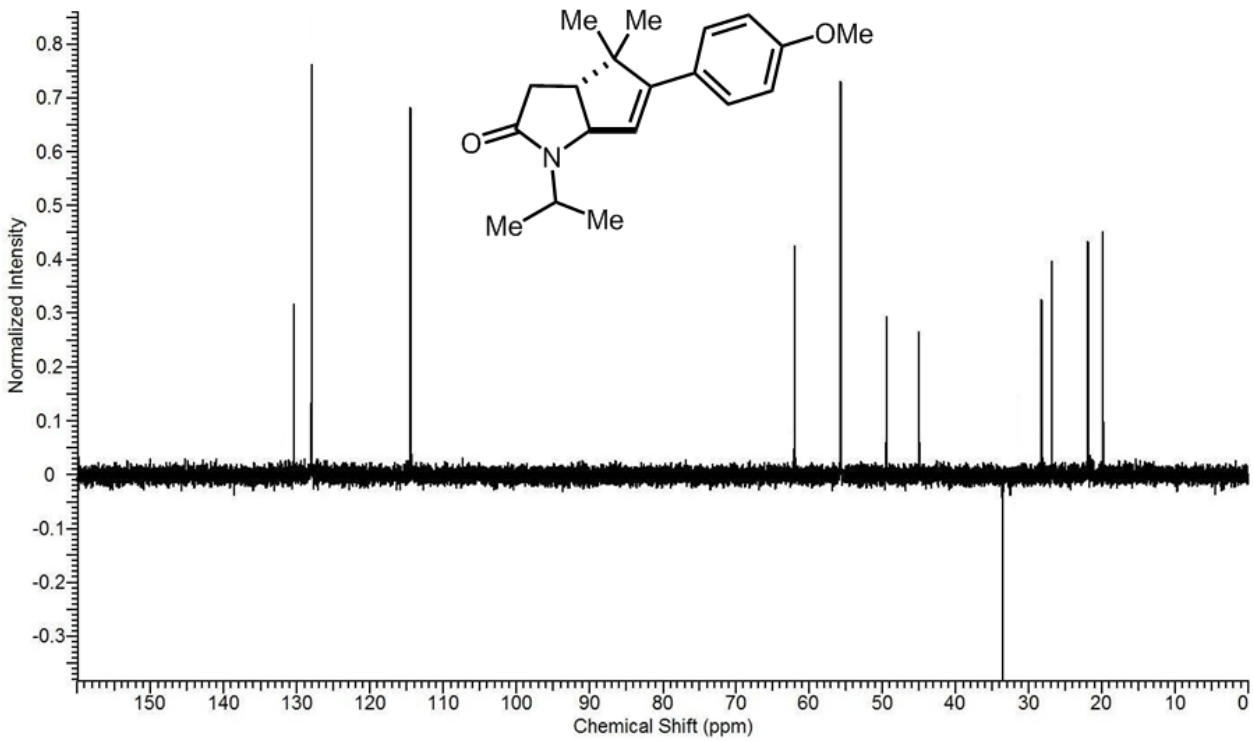
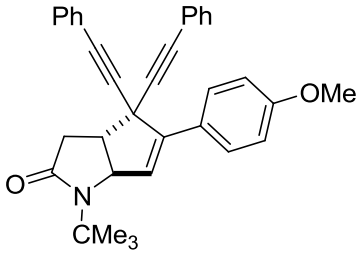
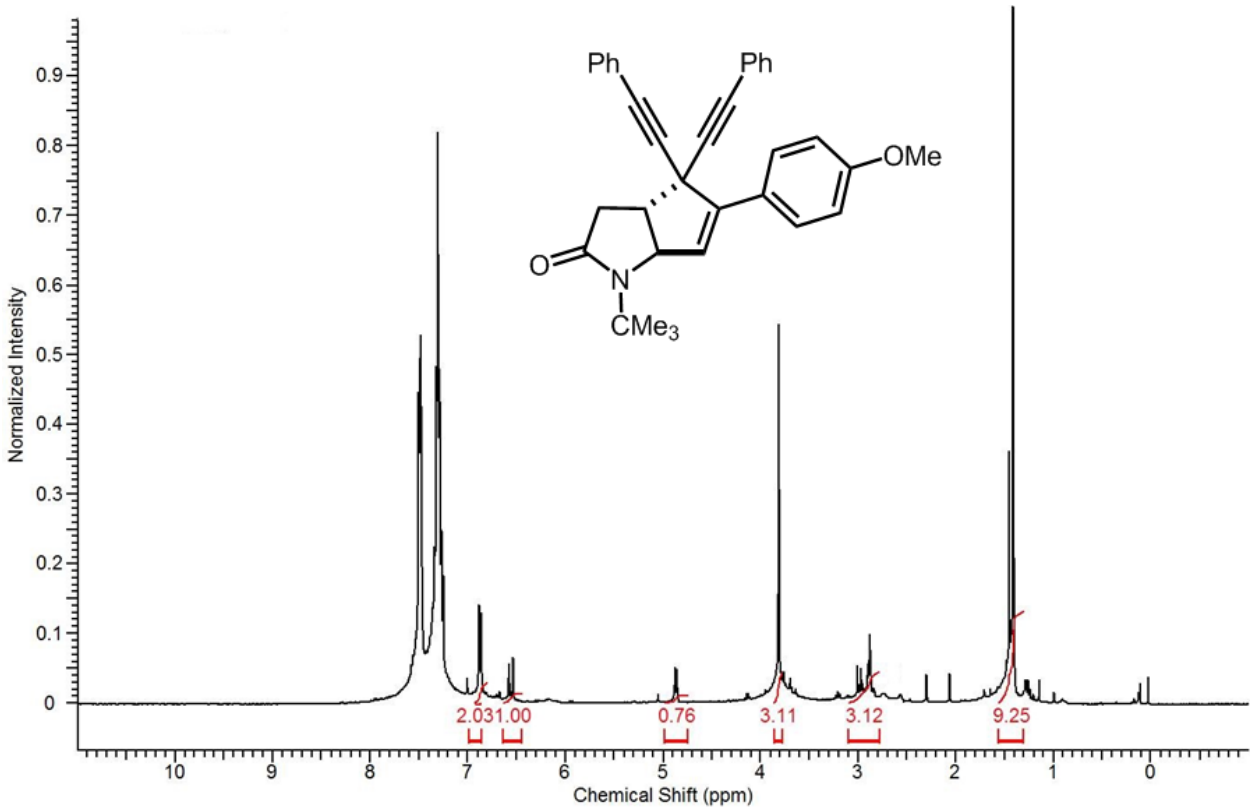
Me
OMe
O
N
Me
Me





1p; 89%, 92:8 dr

Prepared from alcohol **2p** (122.3 mg, 0.25 mmol) using General Procedure A. Oily substance. Yield = 105 mg, 89%. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.58 to 7.21 (m, 12H), 6.88 to 6.86 (d, 2H), 6.58 to 6.56 (d, 1H), 4.89 to 4.86 (t, 1H), 3.77 (s, 3H), 2.91 to 2.83 (m, 3H), 1.36 (s, 9H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 174.36, 159.46, 133.19, 132.20, 132.14, 132.08, 132.03, 130.85, 130.22, 129.97, 129.35, 129.17, 129.00, 128.97, 128.90, 128.75, 128.67, 128.42, 128.33, 127.70, 125.62, 122.24, 122.05, 121.89, 121.82, 114.20, 87.72, 84.13, 66.48, 63.98, 62.80, 55.44, 50.11, 41.36, 39.76, 29.39. **HRMS-EI^{+}** (*m/z*): calc'd for $C_{33}H_{29}NO_2$ 471.2198; found 471.2192.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N
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Normalized Intensity
Chemical Shift (ppm)
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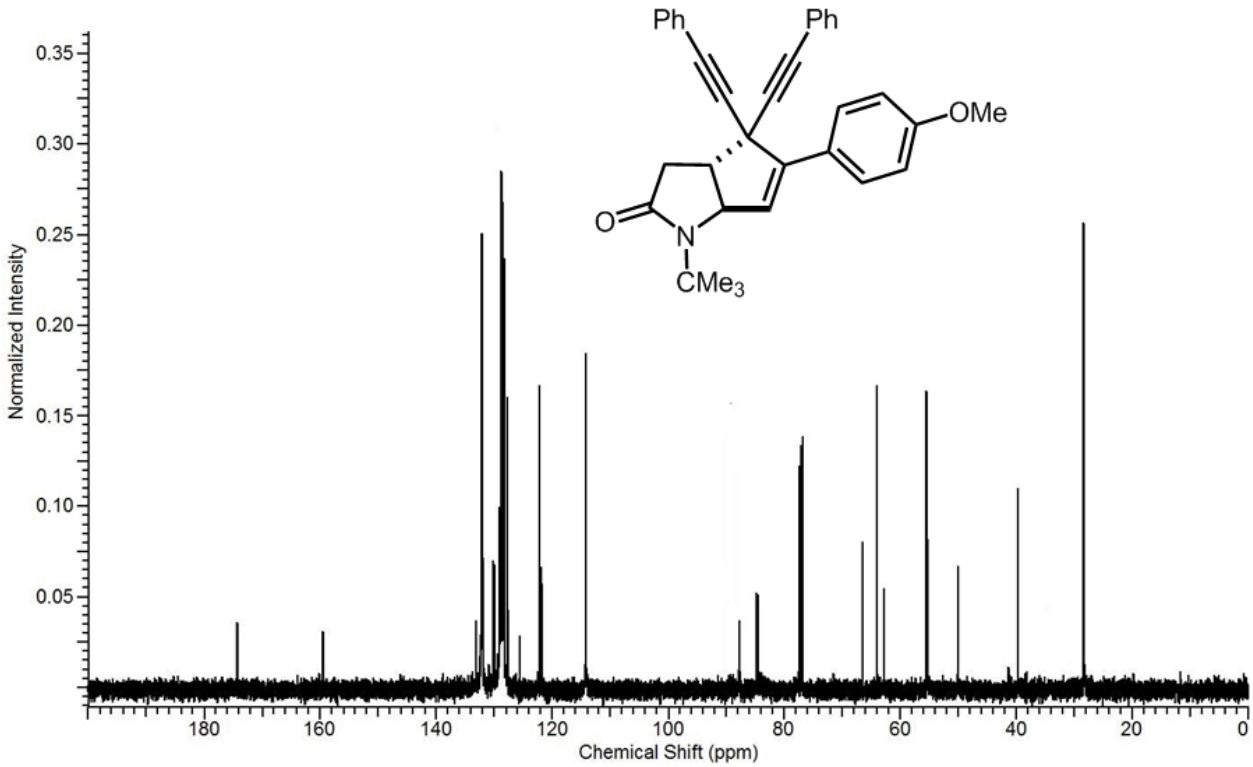
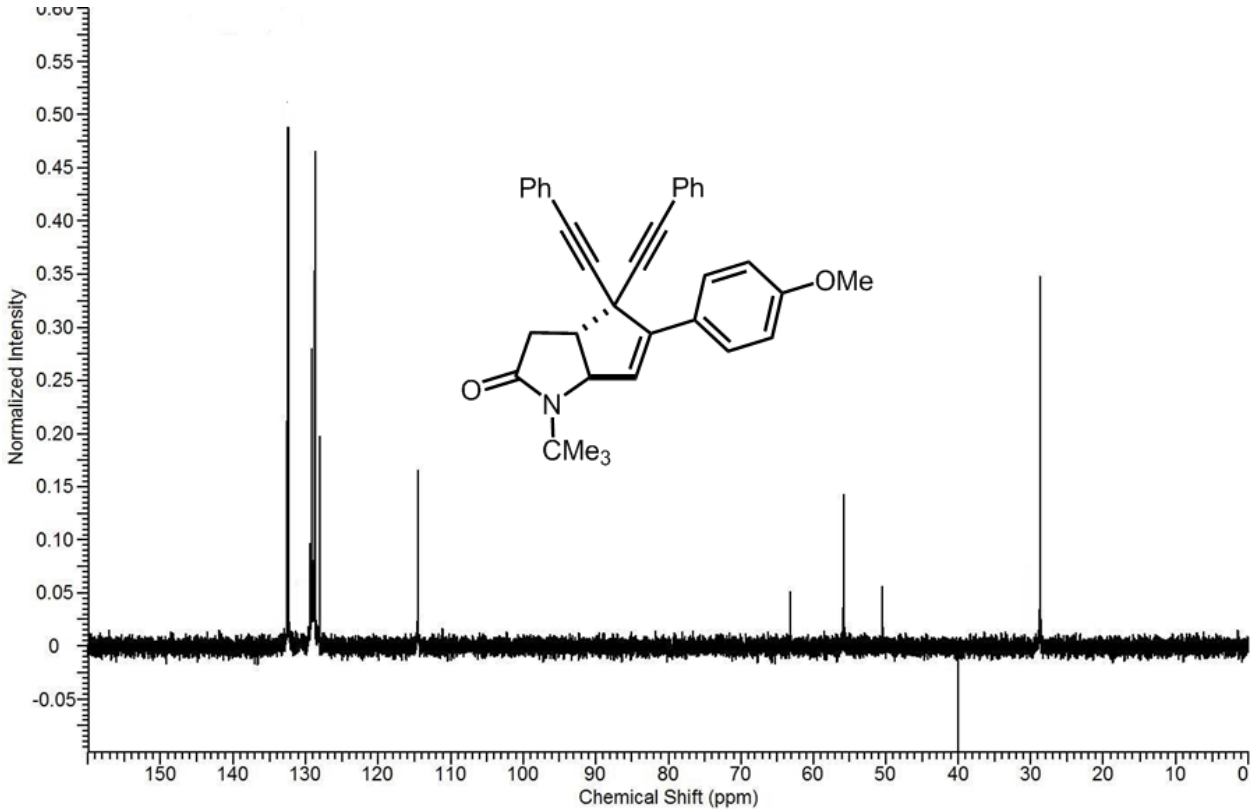
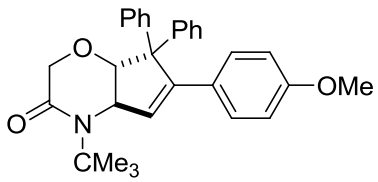
3.12
9.25

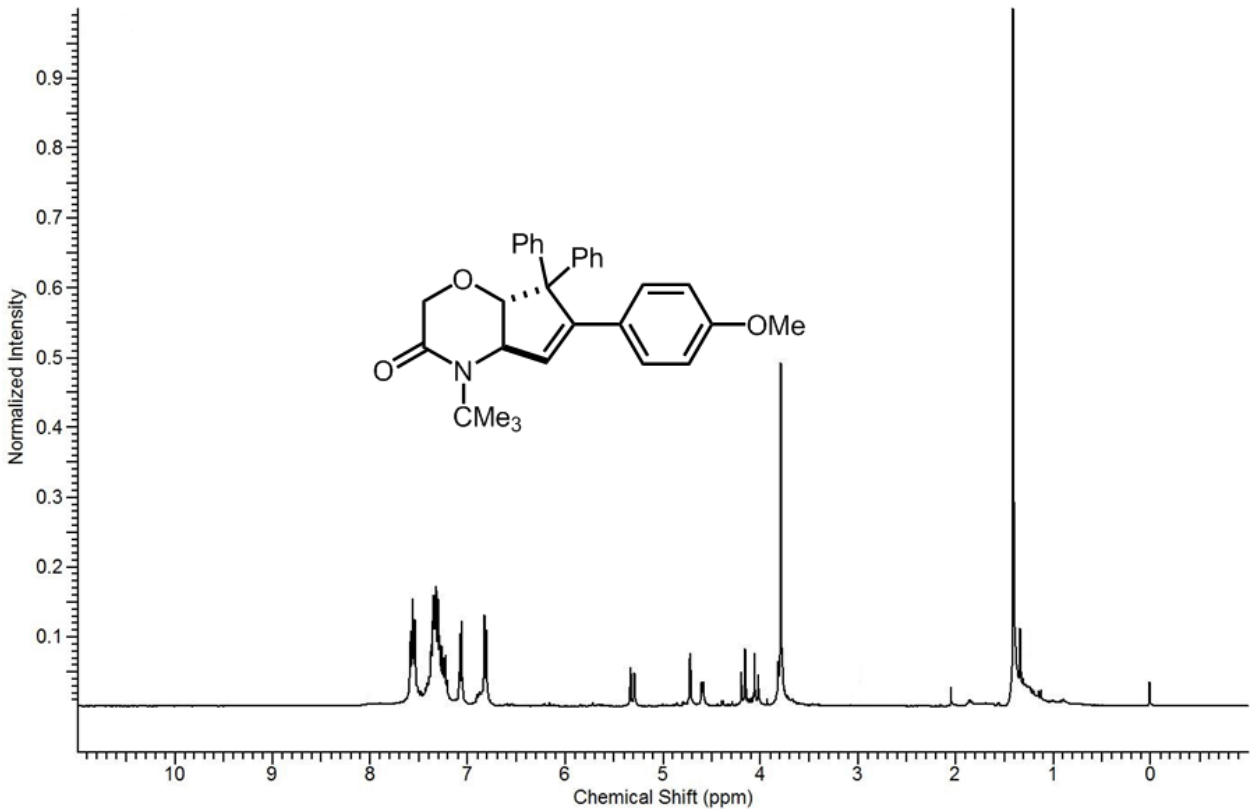

Ph
Ph
OMe
O
N
CMe3
Normalized Intensity
Chemical Shift (ppm)





1q; 90%, 96:4 dr

Prepared from alcohol **2q** (118 mg, 0.25 mmol) using General Procedure A. Oily substance. Yield = 102 mg, 90%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.59 to 7.21 (m, 10H), 7.07 (d, *J* = 8.3 Hz, 2H), 6.84 (d, *J* = 8.3 Hz, 2H), 5.33 (d, 1H), 4.72 (d, *J* = 2.7 Hz, 1H), 4.59 (dd, *J* = 8.4, 2.7 Hz, 1H), 4.18 (d, *J* = 15.2 Hz, 1H), 4.04 (d, *J* = 15.1 Hz, 1H), 3.79 (s, 3H), 1.31 (s, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.6, 159.5, 145.0, 142.8, 131.5, 130.4, 129.5, 128.7, 128.5, 128.4, 128.4, 128.3, 128.2, 128.2, 127.8, 127.6, 127.4, 127.3, 126.5, 126.4, 126.2, 126.0, 126.0, 125.9, 125.3, 124.7, 114.2, 83.2, 68.4, 59.3, 58.0, 57.7, 52.31, 28.7. **HRMS-EI$^+$** (*m/z*): calc'd for $C_{30}H_{31}NO_3$ 453.2304; found 453.2310.


Normalized Intensity
Chemical Shift (ppm)
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Ph
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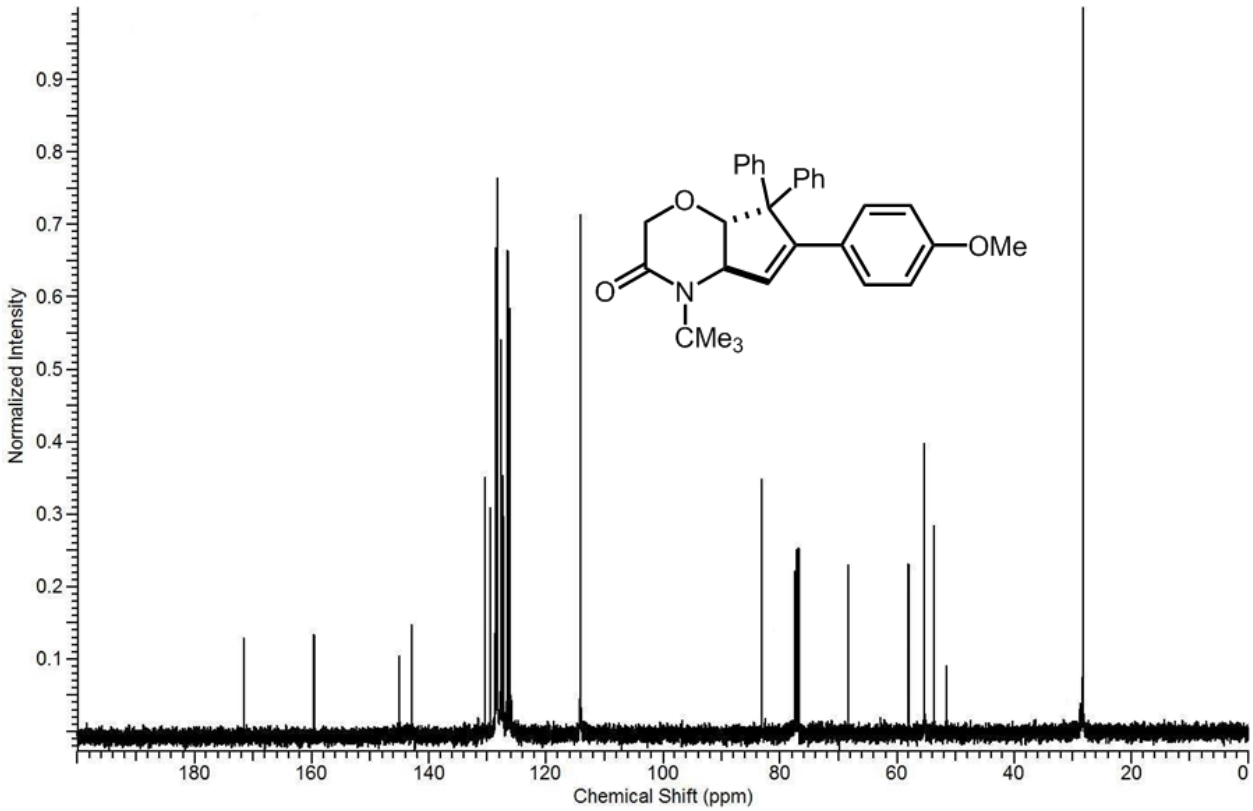
O
N
CMe3
OMe


Normalized Intensity
Chemical Shift (ppm)
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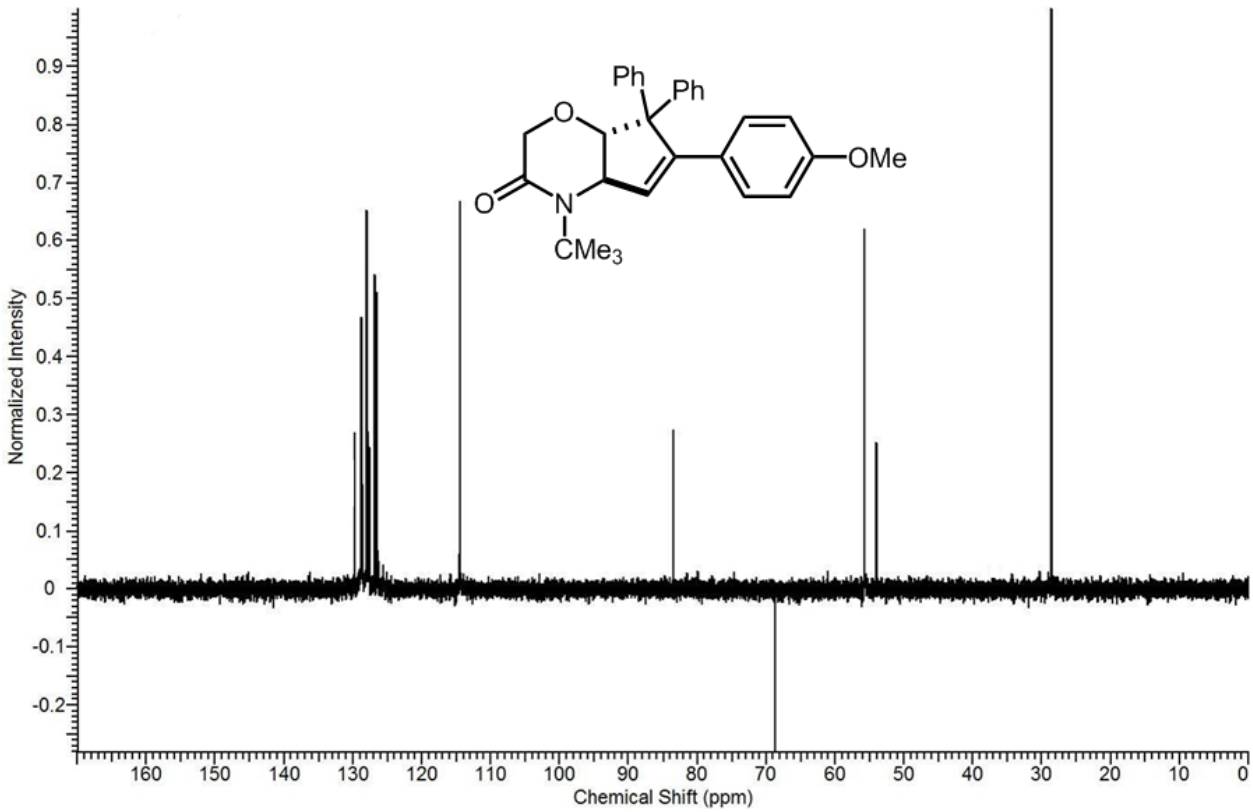
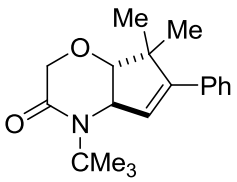
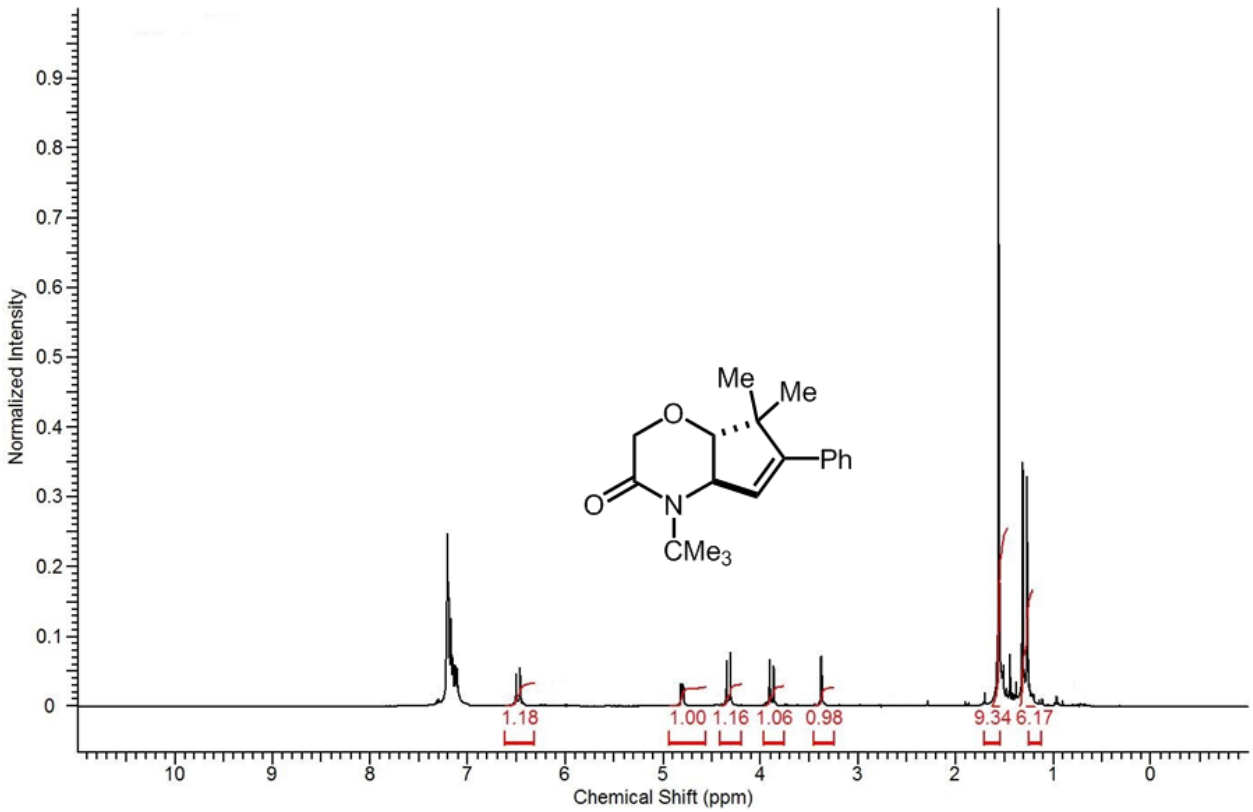
Ph
O
O
N
CMe3
OMe





1r; 81%, 95:5 dr

Prepared from alcohol **2r** (158.6 mg, 0.50 mmol) using General Procedure A. Oily subtance. Yield = 121.2 mg, 81%. ^{1}H NMR (400 MHz, C_6D_6) δ 7.32 to 7.03 (m, 5H), 6.48 (d, *J* = 8.0 Hz, 1H), 4.80 (dd, *J* = 8.0, 2.4 Hz, 1H), 4.33 (d, *J* = 15.2 Hz, 1H), 3.88 (d, *J* = 15.2 Hz, 1H), 3.38 (d, *J* = 2.4 Hz, 1H), 1.61 (s, 9H), 1.38 to 1.31 (two overlapping s, 6H). ^{13}C NMR (101 MHz, C_6D_6) δ 171.0, 136.4, 133.1, 130.8, 128.7, 128.7, 128.5, 128.0, 128.0, 127.9, 127.9, 127.8, 127.6, 127.5, 126.5, 84.9, 67.9, 57.5, 53.6, 48.9, 28.0, 26.9, 23.5. **HRMS-EI^{+}** (*m/z*): calc'd for $C_{19}H_{25}NO_2$ 299.1885; found 299.1889.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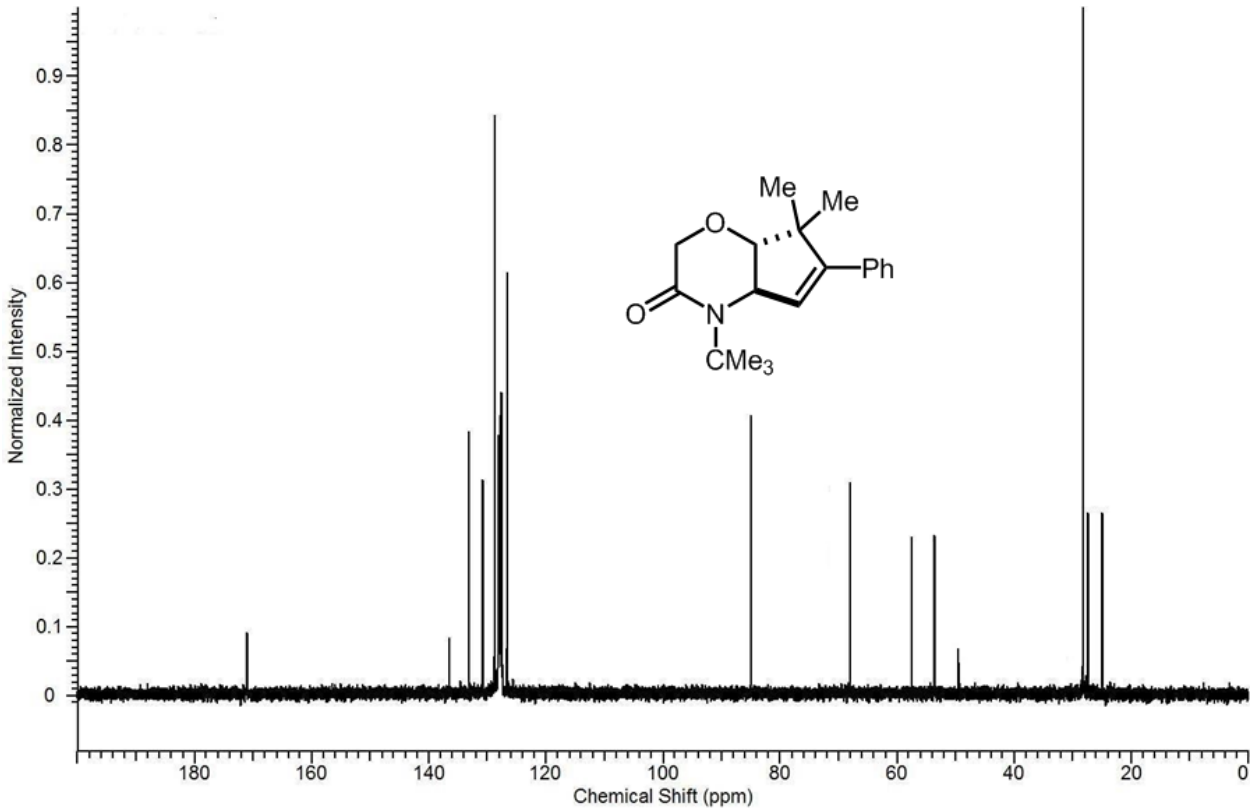
Normalized Intensity
Chemical Shift (ppm)
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Ph
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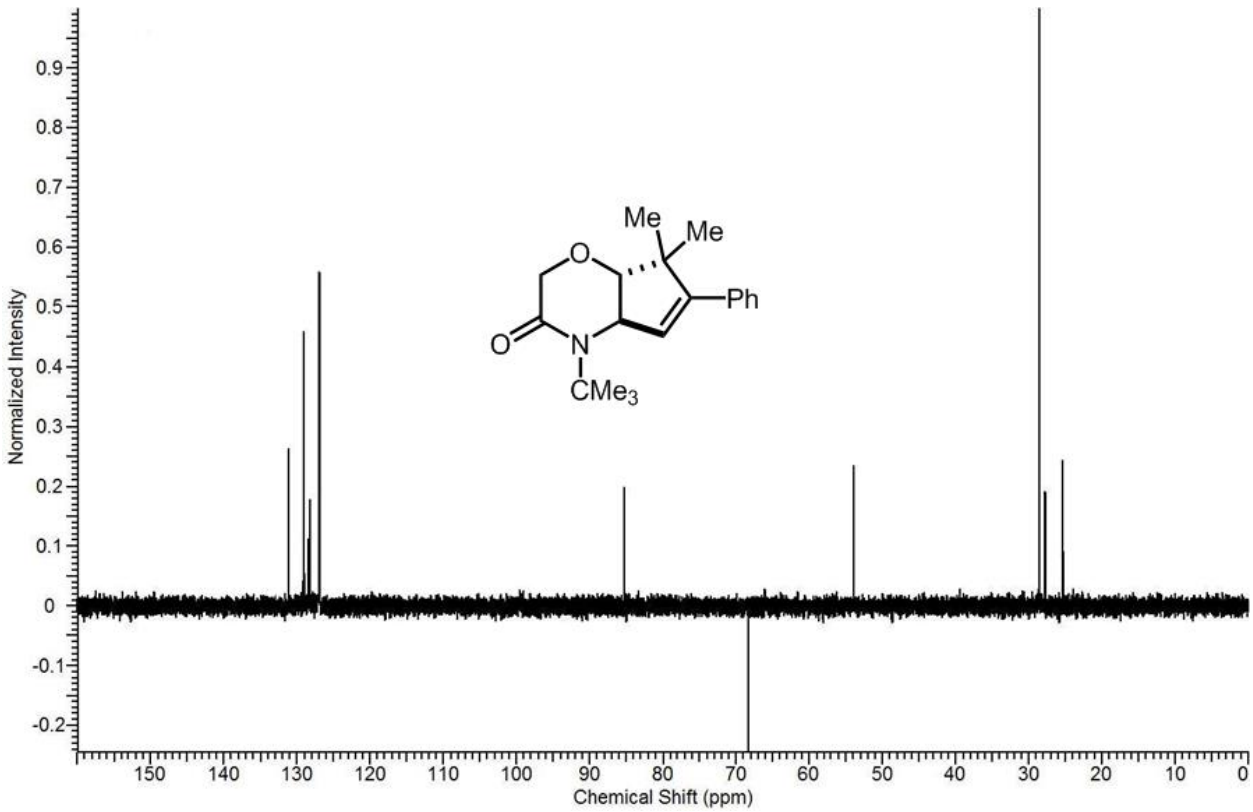
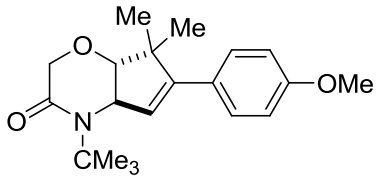
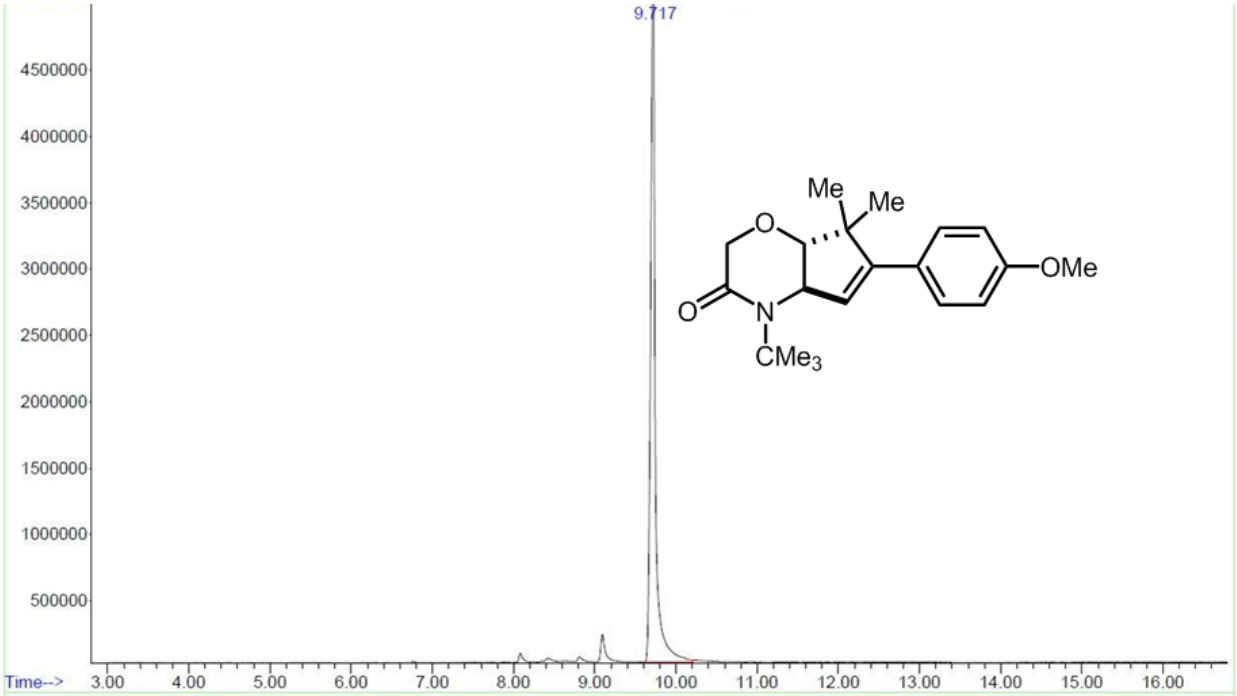




1s; 76%, 97:3 dr

Prepared from alcohol **2s** (173.6 mg, 0.50 mmol) using General Procedure A. Oily substance. Yield = 125 mg, 76%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.27 (d, *J* = 8.5 Hz, 2H), 6.85 (d, *J* = 8.5 Hz, 2H), 6.40 (d, *J* = 8.0 Hz, 1H), 4.68 (dd, *J* = 8.0, 2.7 Hz, 1H), 4.17 (d, *J* = 15.3 Hz, 1H), 3.97 (d, *J* = 15.3 Hz, 1H), 3.79 (s, 3H), 3.51 (d, *J* = 2.7 Hz, 1H), 1.44 to 1.24 (m, 9H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.3, 159.6, 132.0, 130.5, 128.7, 127.8, 127.7, 125.5, 124.9, 114.1, 85.0, 68.7, 58.8, 57.9, 55.7, 50.7, 28.5, 26.2, 25.6. **HRMS-EI$^+$** (*m/z*): calc'd for $C_{20}H_{27}NO_3$ 329.1991; found 329.1996.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OMe
O
N
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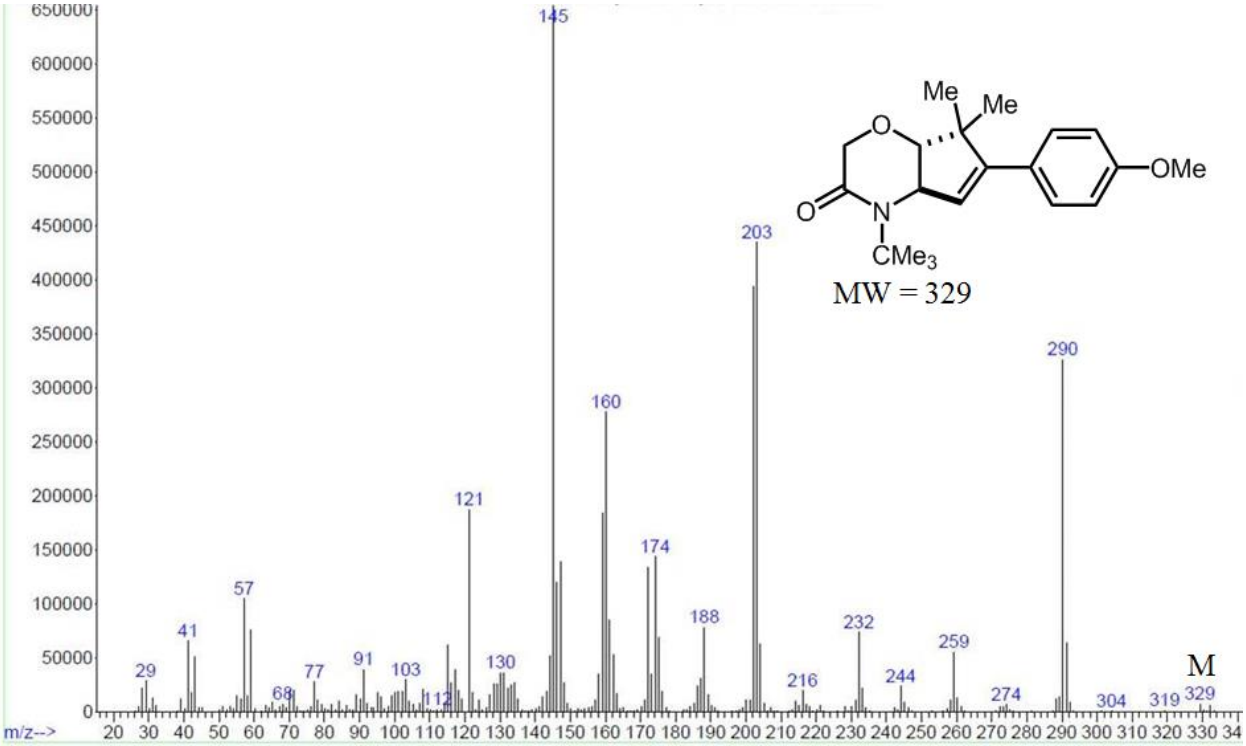
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274
304
319
329
M
Me
Me
O
OMe
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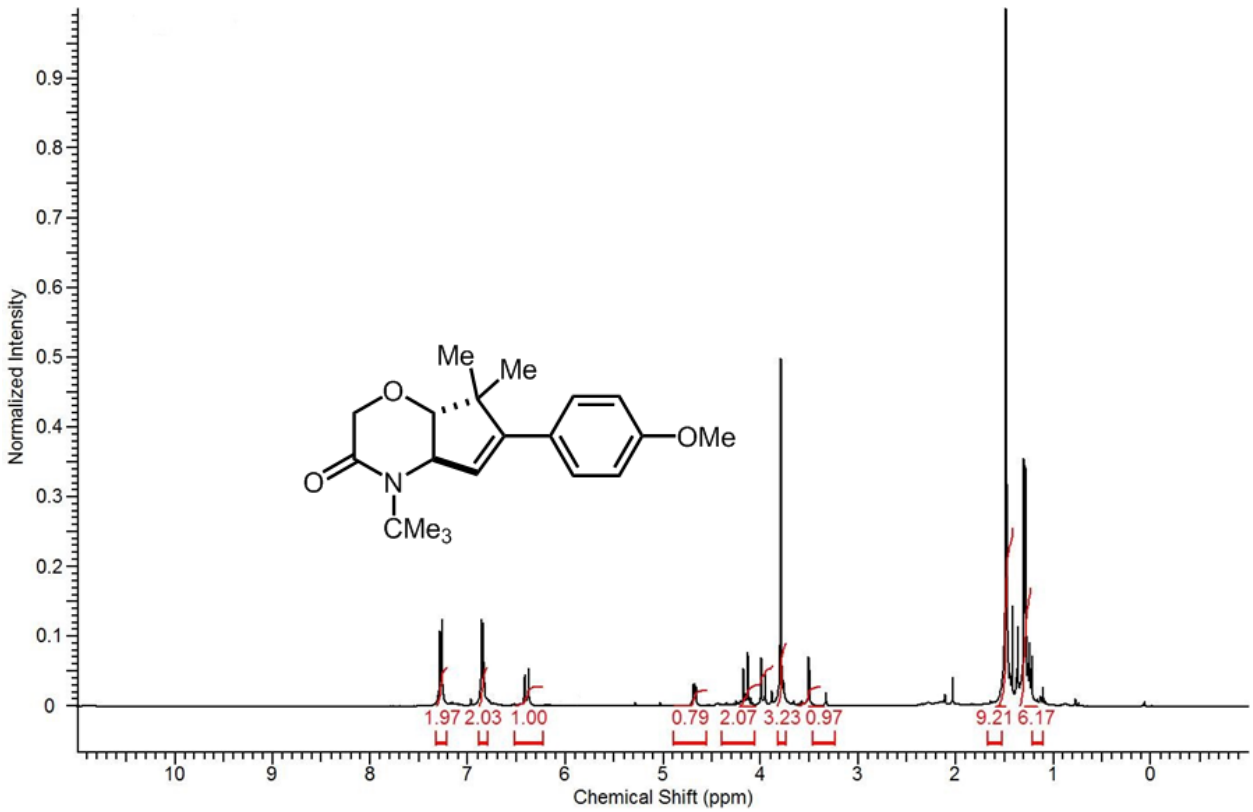
Normalized Intensity
Chemical Shift (ppm)
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OMe
O
N
CMe3
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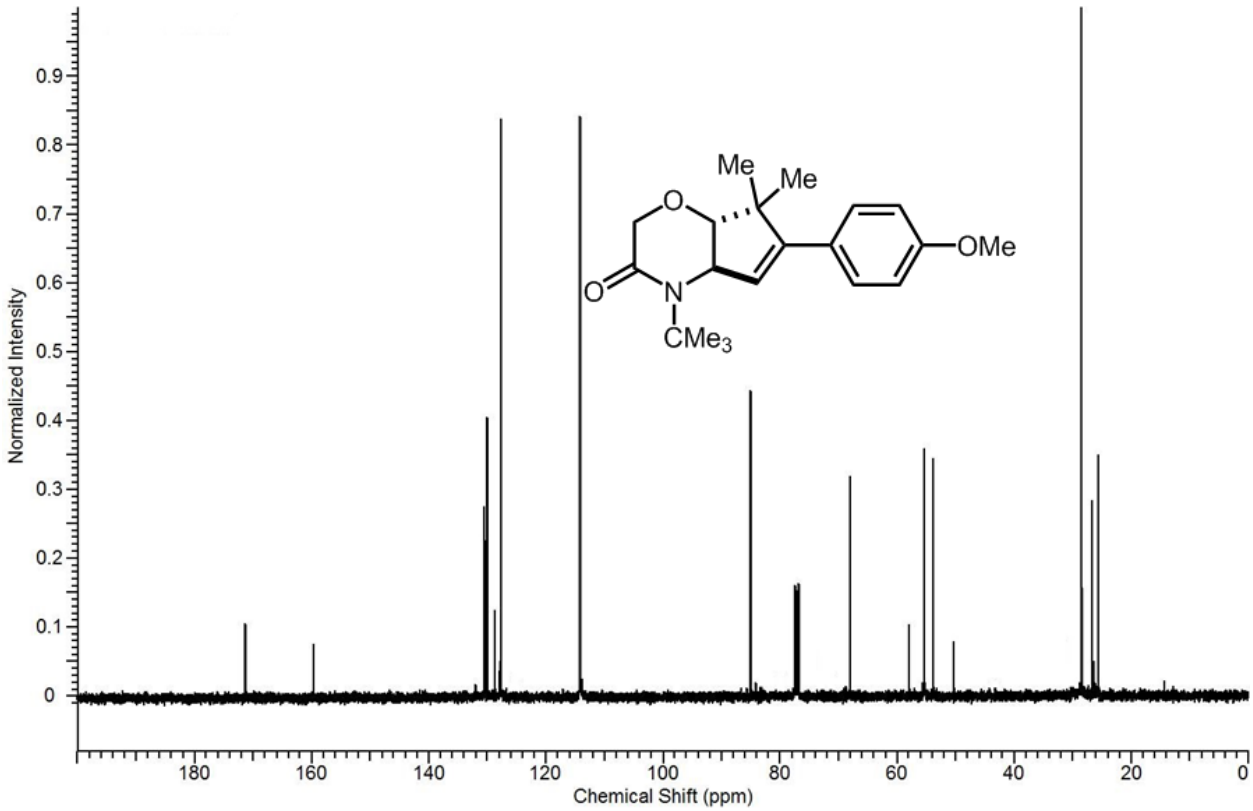
0.79 2.07 3.23 0.97
9.21 6.17


Normalized Intensity
Chemical Shift (ppm)
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OMe
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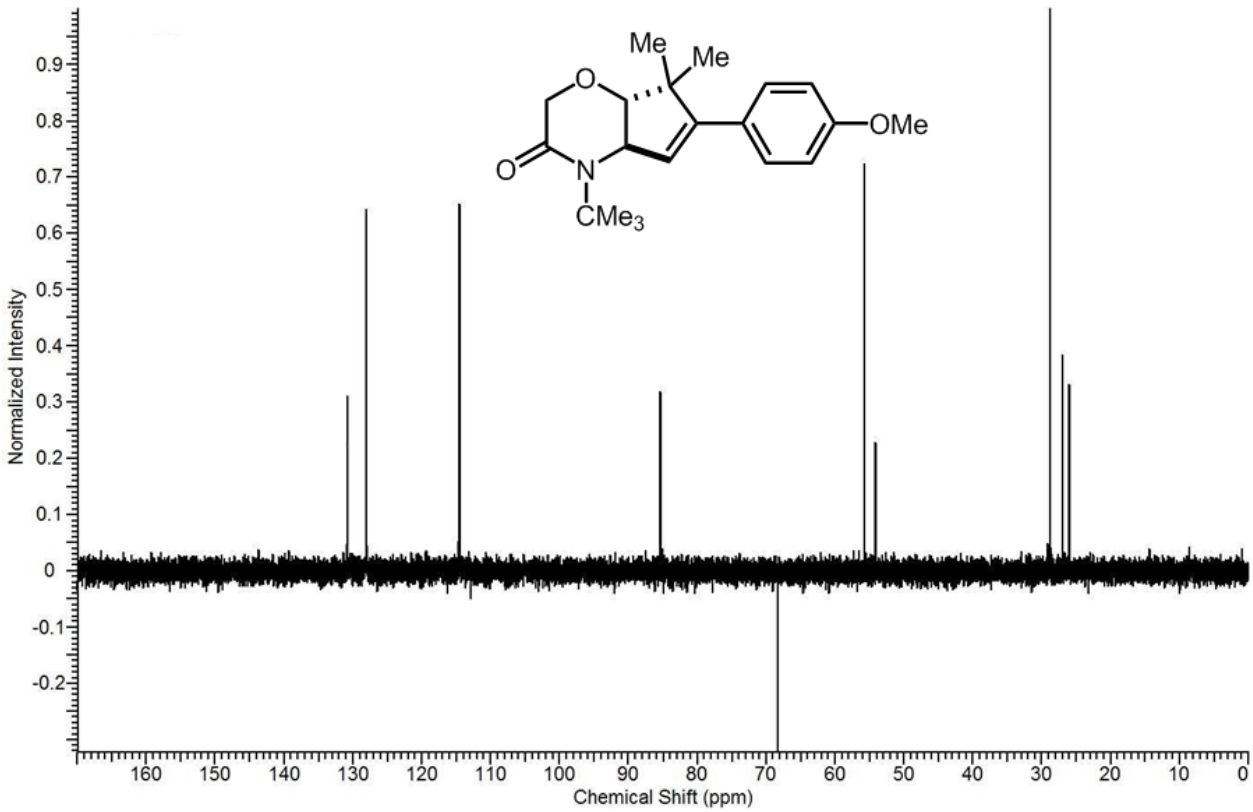
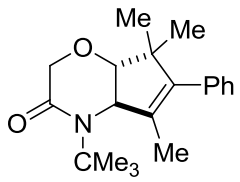
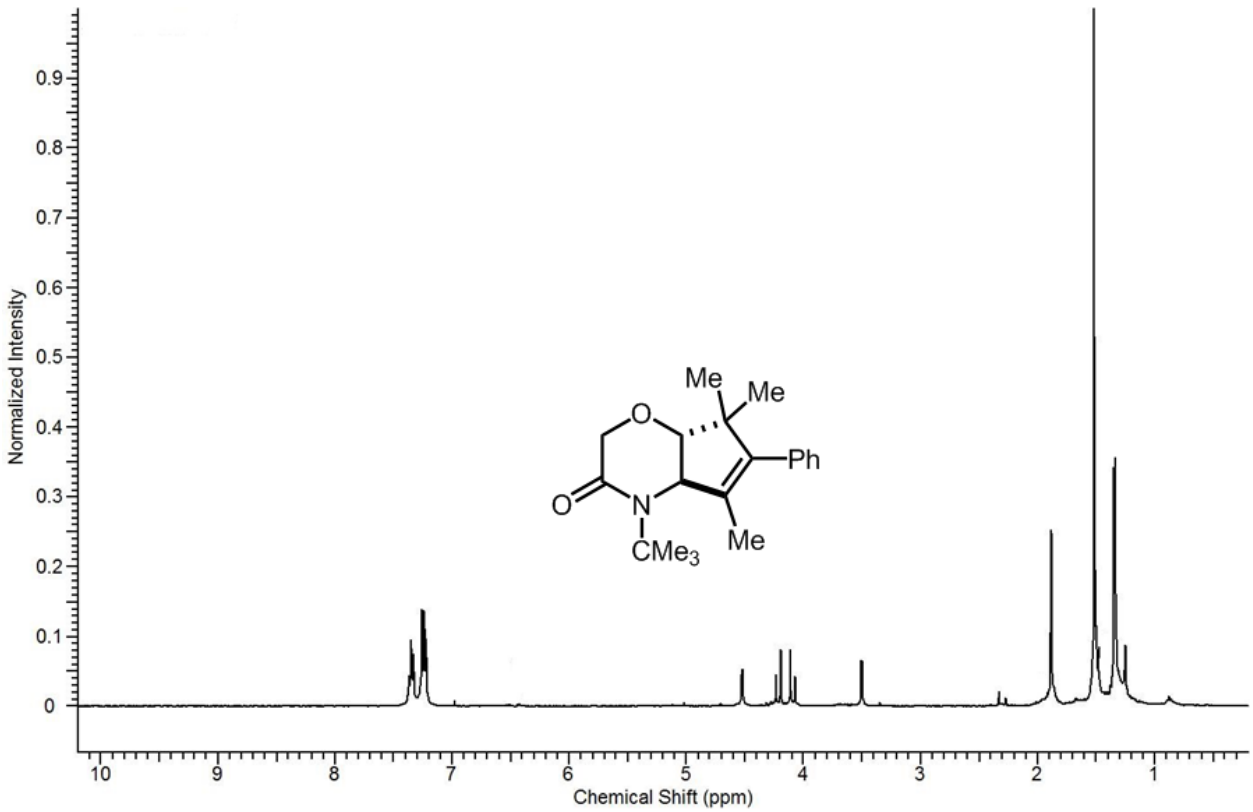
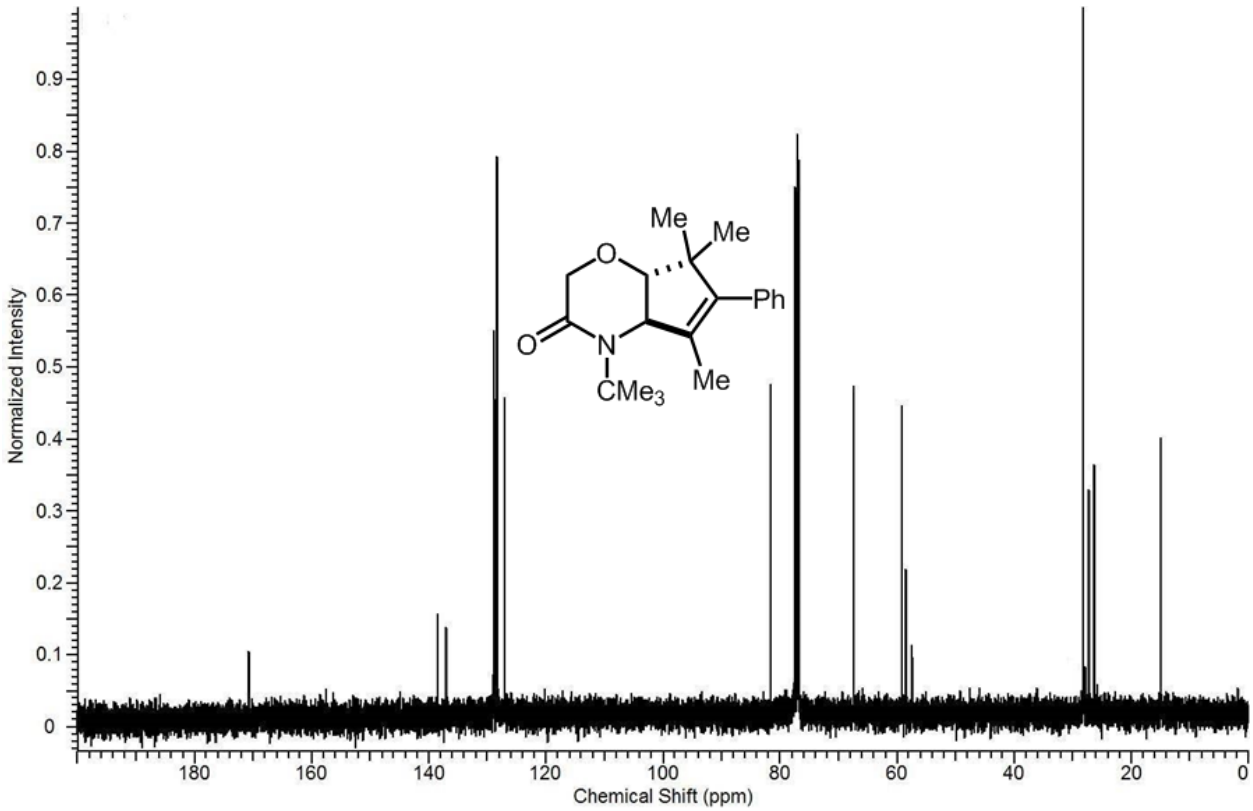


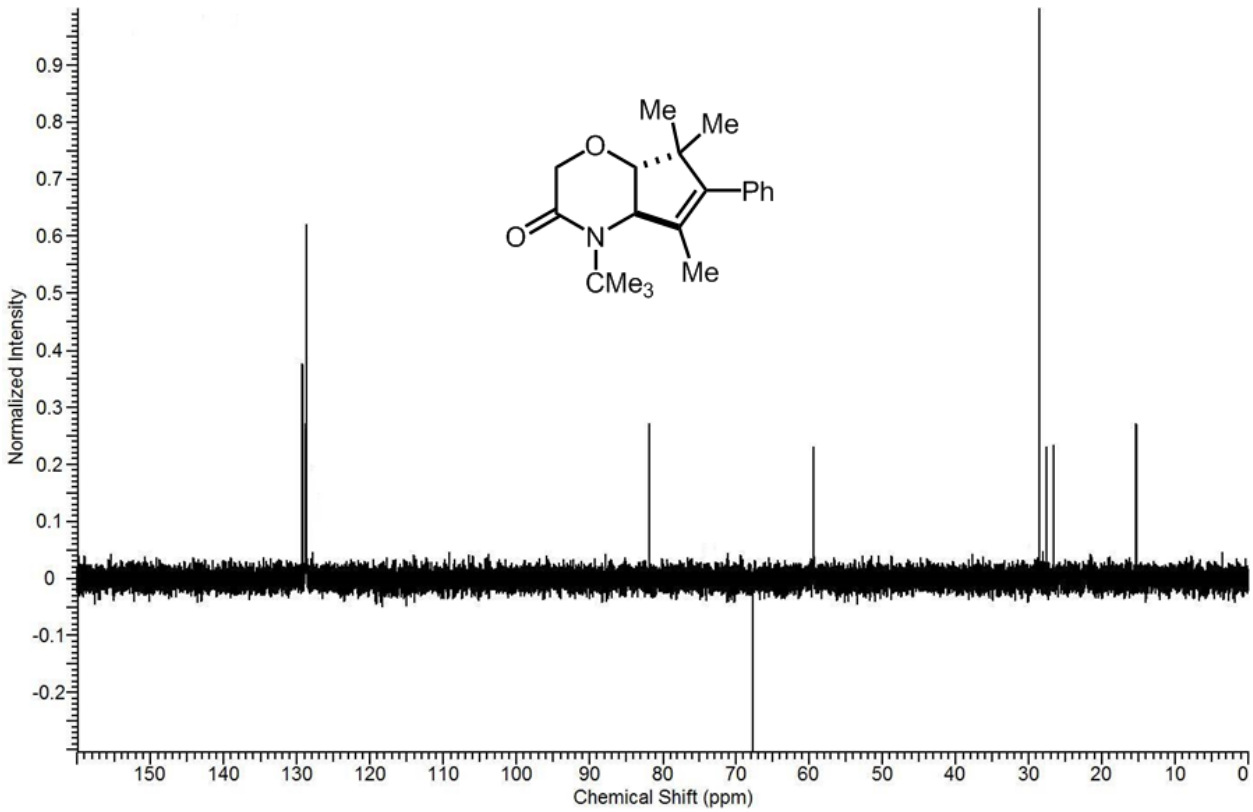


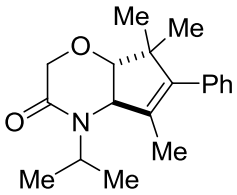
1t; 80%, 96:4 dr

Prepared from alcohol **2t** (165.6 mg, 0.50 mmol) using General Procedure A. Oily substance. Yield = 125.3 mg, 80%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.37 to 7.22 (m, 5H), 4.52 (d, *J* = 2.7 Hz, 1H), 4.21 (d, *J* = 15.8 Hz, 1H), 4.09 (d, *J* = 15.8 Hz, 1H), 3.50 (d, *J* = 2.7 Hz, 1H), 1.88 (s, 3H), 1.54 to 1.30 (m, 15H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 170.7, 138.4, 136.9, 128.9, 128.4, 128.3, 128.3, 126.9, 81.5, 67.4, 59.1, 58.5, 57.3, 28.2, 27.8, 26.3, 15.0. **HRMS-EI$^+$** (*m/z*): calc'd for $C_{20}H_{27}NO_2$ 313.2042; found 313.2046.


Normalized Intensity
Chemical Shift (ppm)
Me
Me
O
Ph
O
N
CMe3
Me

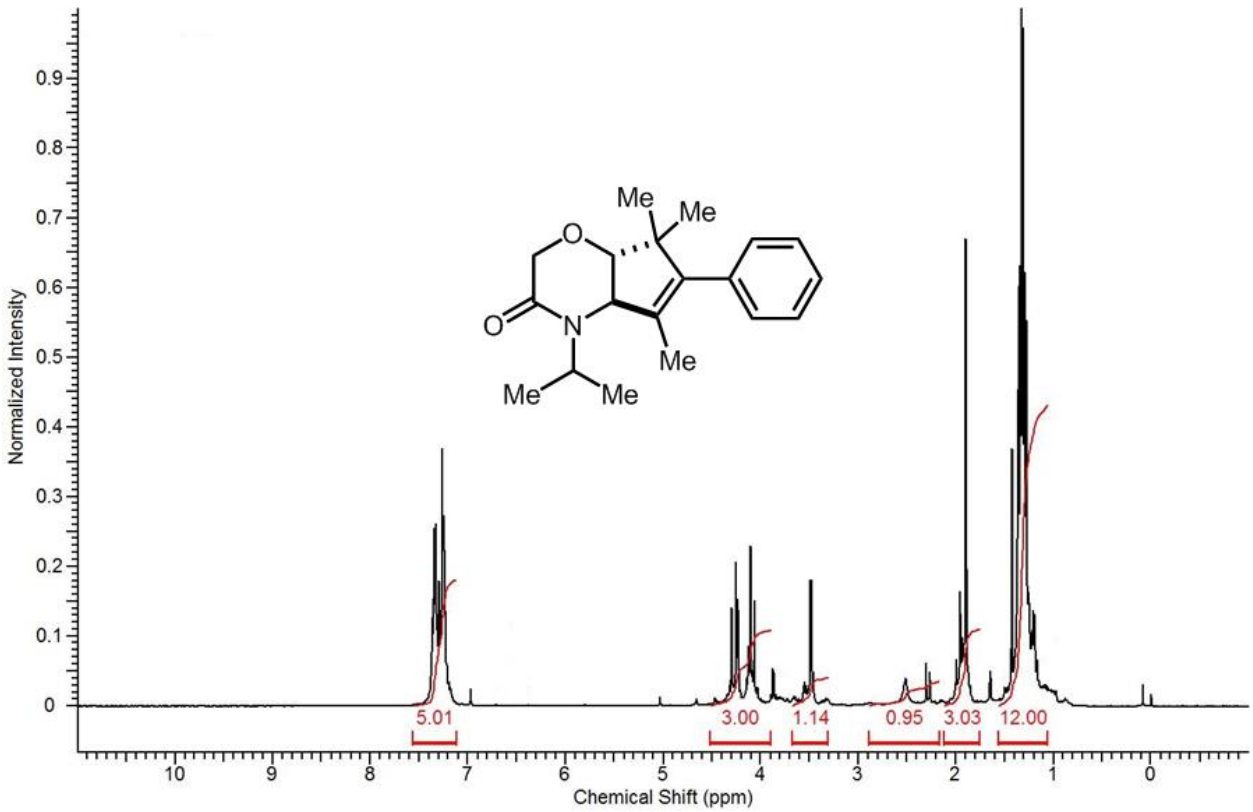

Normalized Intensity
Chemical Shift (ppm)
Me
Me
O
Ph
O
N
CMe3
Me





1u; 74%, 90:10 dr

Prepared from alcohol **2u** (158.6 mg, 0.50 mmol) using General Procedure A. Oily substance. Yield = 111 mg, 74%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.38 to 7.18 (m, 5H), 4.48 to 4.03 (m, 4H), 3.52 to 3.36 (m, 1H), 2.52 (s, 1H), 1.99 (s, 3H), 1.50 to 1.02 (m 12H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.5, 137.0, 136.7, 131.0, 129.0, 129.0, 128.9, 128.7, 128.6, 128.4, 128.3, 128.0, 127.2, 126.1, 126.0, 119.6, 81.2, 67.4, 62.7, 59.9, 55.32, 49.2, 47.8, 30.4, 27.7, 26.7, 26.4, 25.8, 22.4, 20.6, 19.8, 19.7, 19.7, 15.9, 14.3. **HRMS-EI$^+$** (*m/z*): calc'd for $C_{19}H_{25}NO_2$ 299.1885; found 299.1889.


Normalized Intensity
Chemical Shift (ppm)
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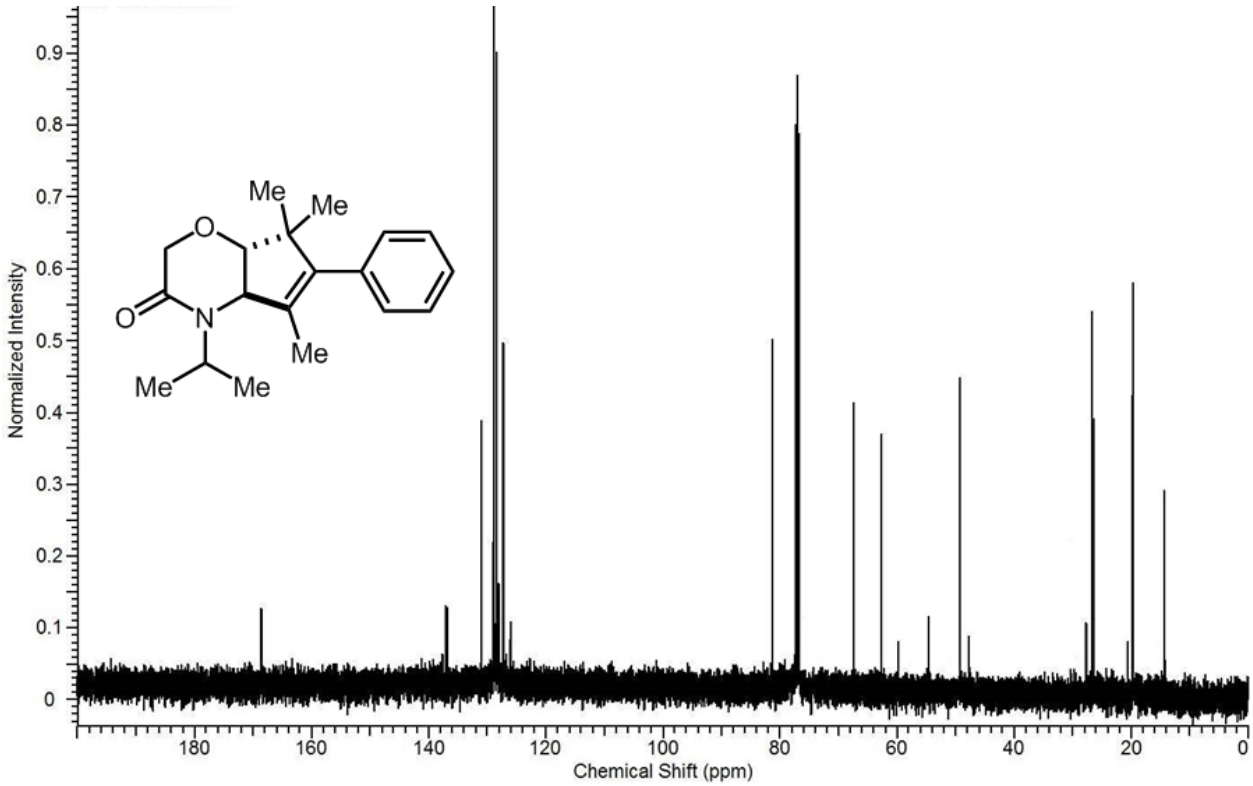
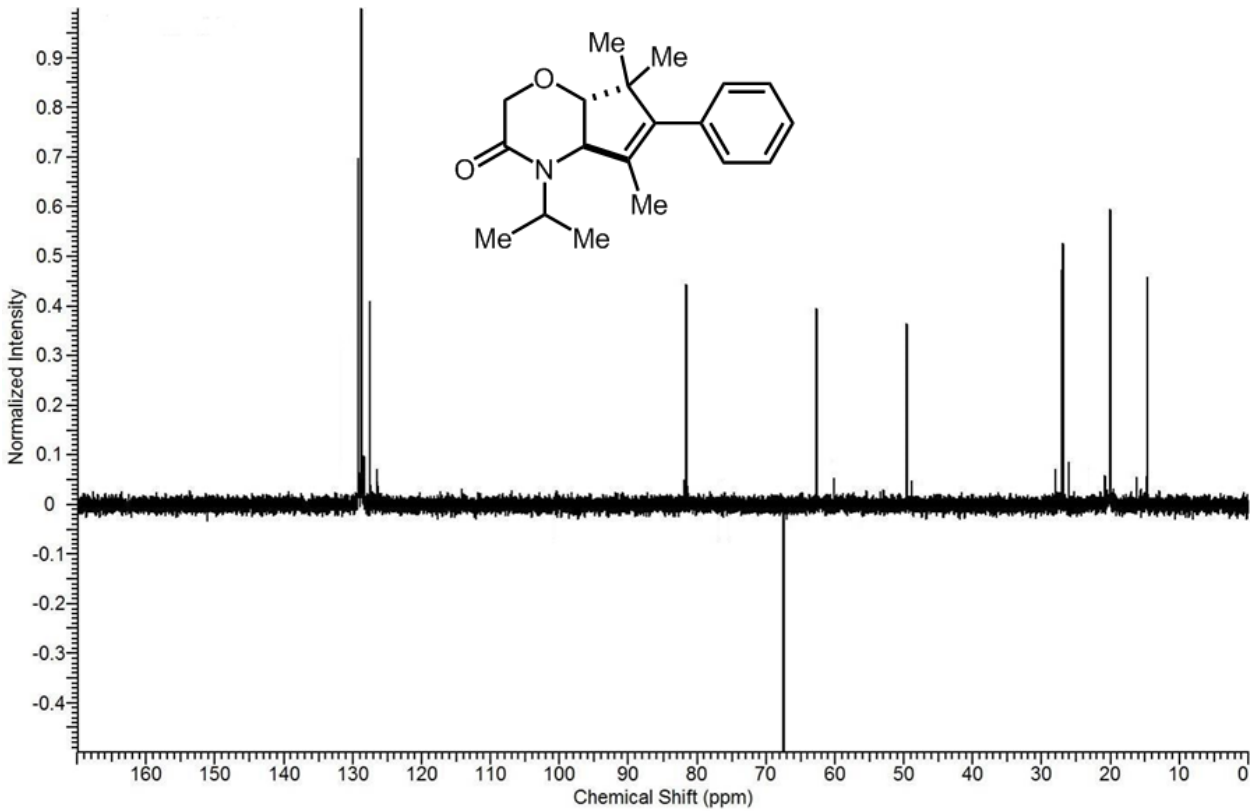
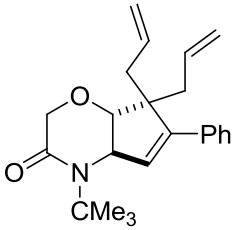
3.00
1.14
0.95
3.03
12.00
Me
Me
O
O
N
Me
Me
Me

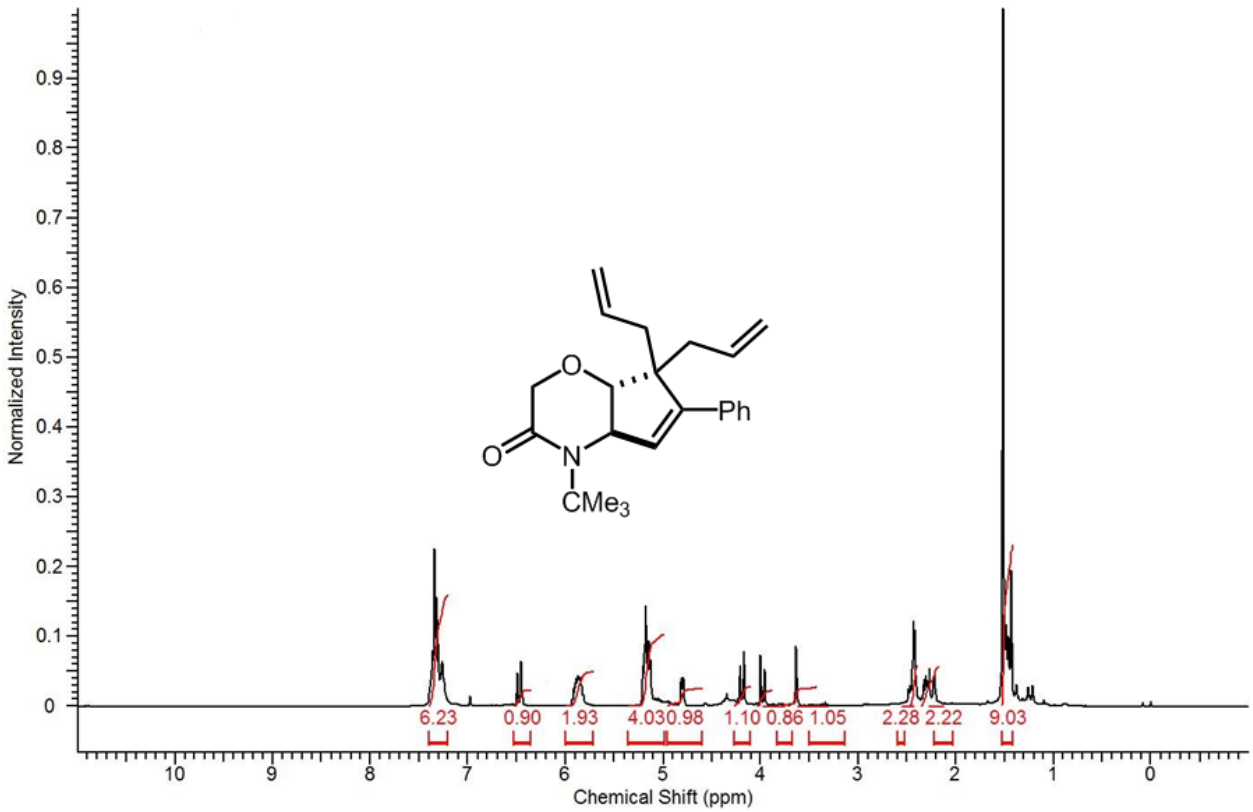

Normalized Intensity
Chemical Shift (ppm)
Me
Me
O
O
N
Me
Me
Me





1v; 78%, 95:5 dr

Prepared from alcohol **2v** (184.6 mg, 0.50 mmol) using General Procedure A. Oily substance. Yield = 137 mg, 78%. ^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.38 to 7.22 (m, 5H), 6.47 (d, 1H), 5.93 to 5.82 (m, 2H), 5.20 to 5.11 (m, 4H), 4.81 to 4.79 (dd, 1H), 4.17 (d, 1H), 3.98 (d, 1H), 3.63 (d, 1H), 2.47 to 2.42 (m, 2H), 2.33 to 2.22 (m, 2H), 1.43 (s, 9H). ^{13}C NMR (101 MHz, Chloroform-*d*) δ 171.15, 136.06, 134.27, 132.83, 131.84, 130.26, 128.62, 127.55, 126.62, 126.53, 119.66, 119.03, 81.56, 71.78, 68.56, 58.13, 54.91, 47.92, 40.79, 40.07, 28.85. **HRMS-EI^{+}** (*m/z*): calc'd for $C_{23}H_{29}NO_2$ 351.2198; found 351.2204.


Normalized Intensity
Chemical Shift (ppm)
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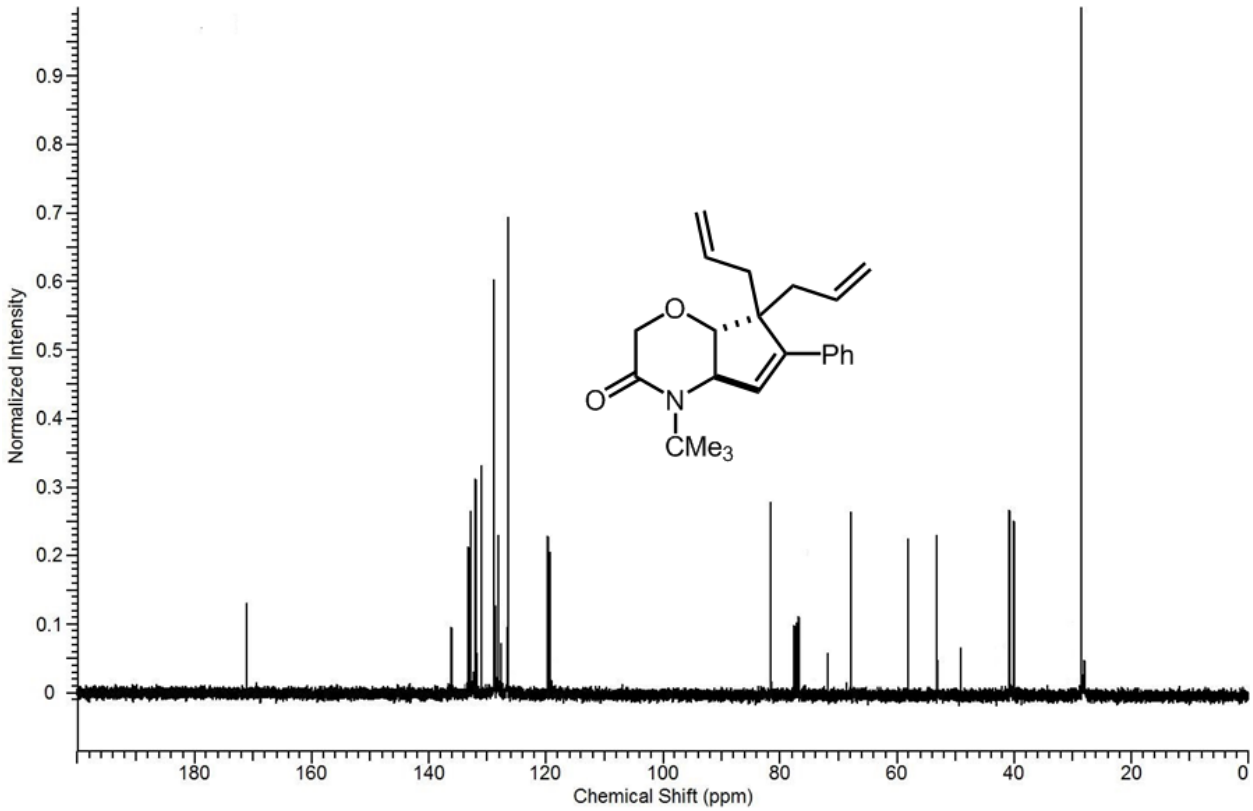
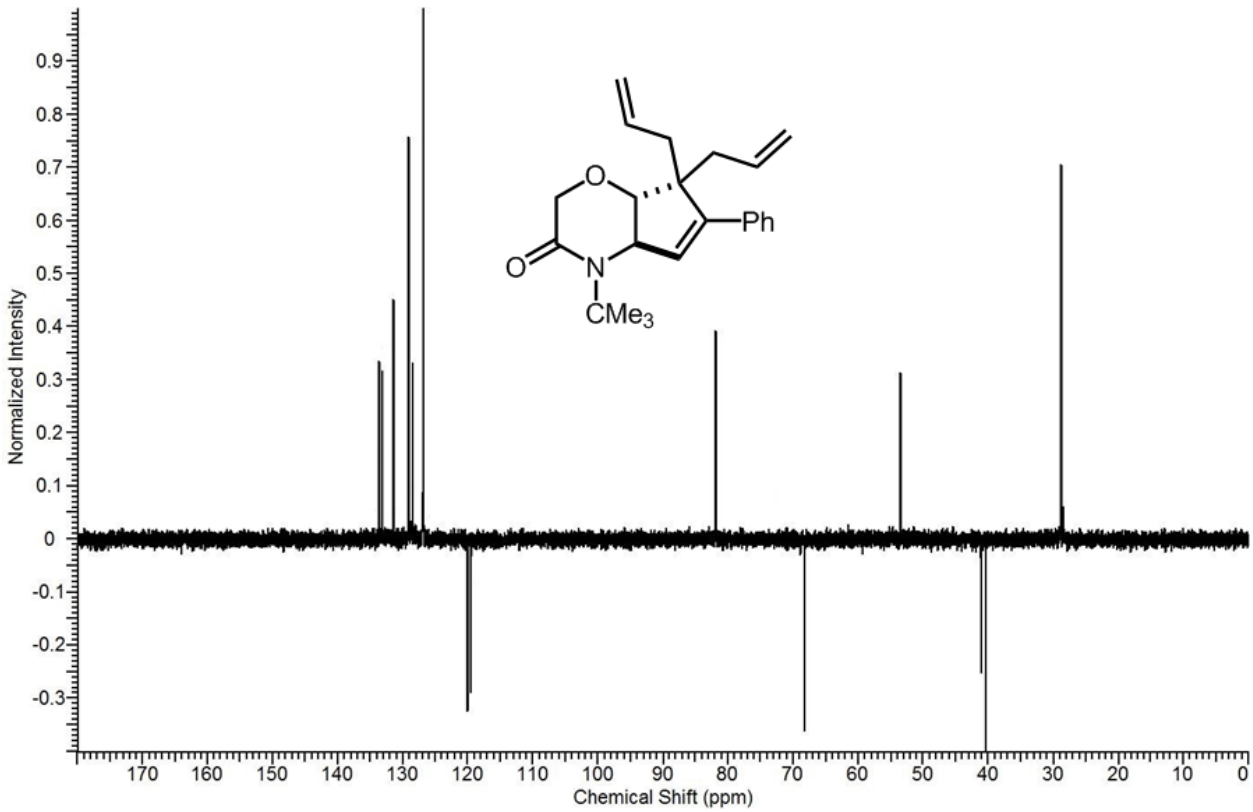
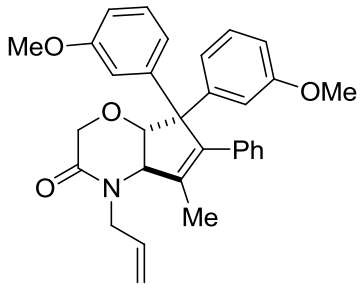
4.03
0.98
1.10
0.86
1.05
2.28
2.22
9.03
Ph
CMe3

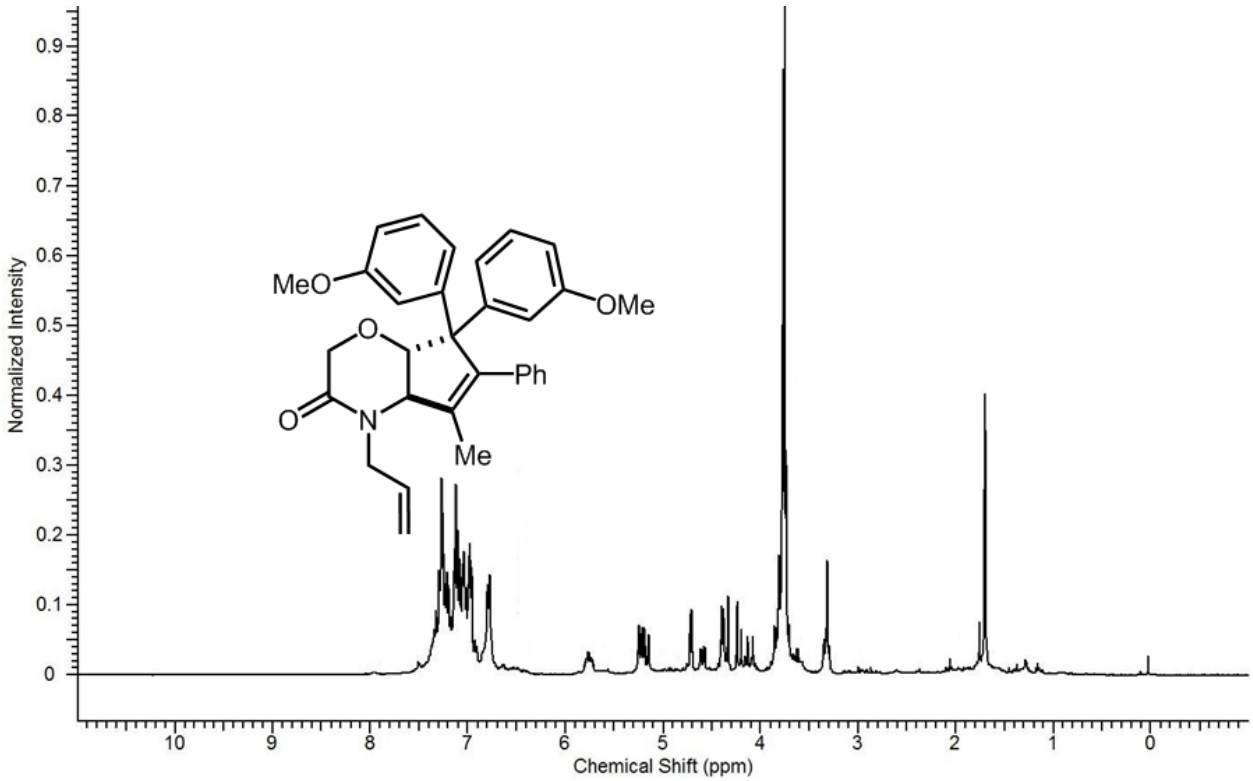

Normalized Intensity
Chemical Shift (ppm)
Ph
CMe3

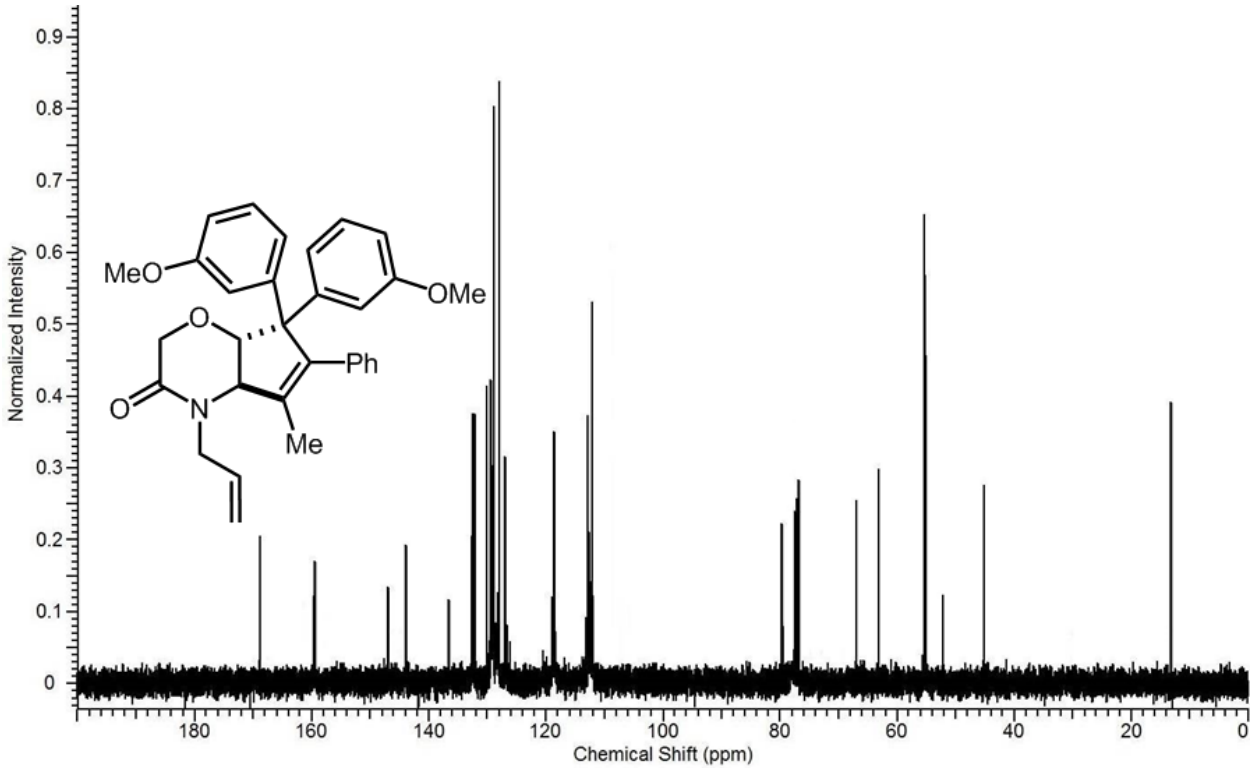


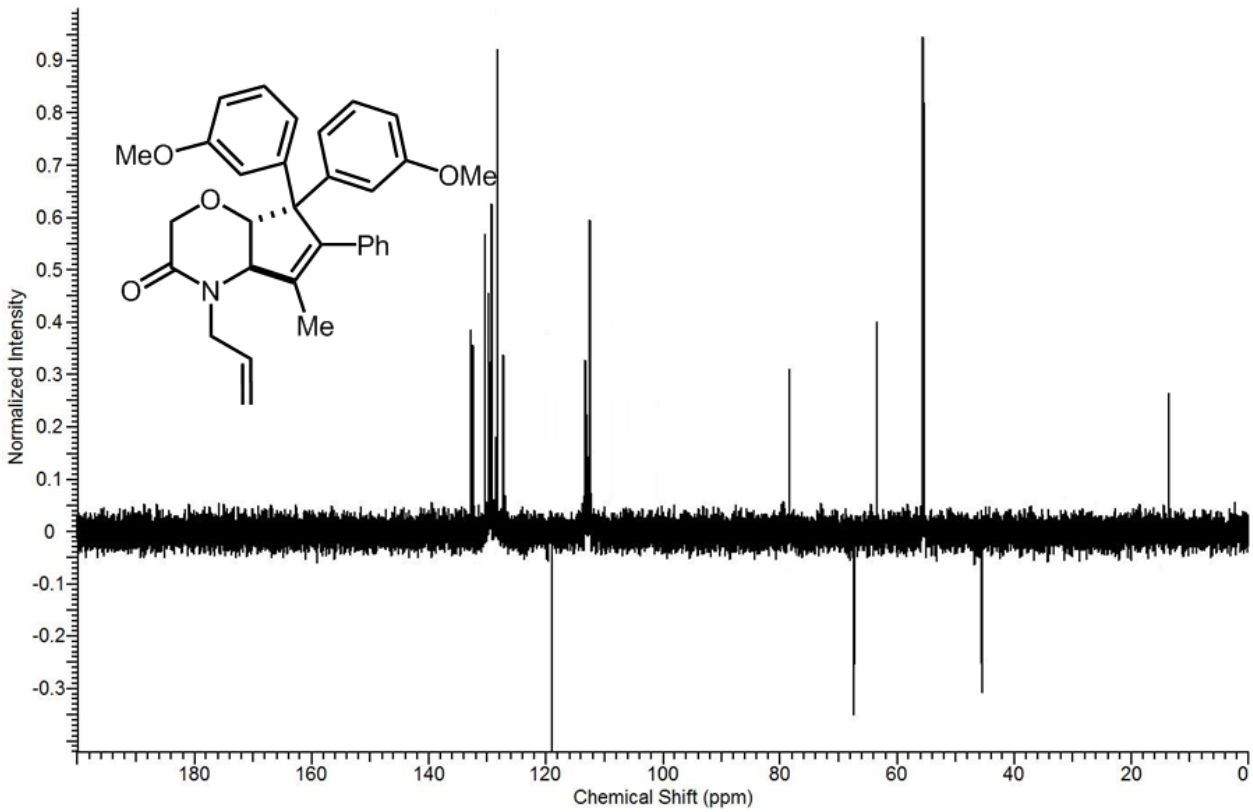


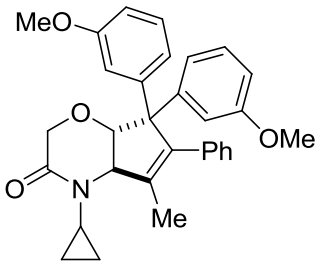
1w; 85%, 94:6 dr

Prepared from alcohol **2w** (125 mg, 0.25 mmol) using General Procedure A. Oily substance. Yield = 102.3 mg, 85%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.36 to 6.81 (m, 13H), 5.78 to 5.71 (m, 1H), 5.26 to 5.15 (dd, 2H), 4.72 (d, 1H), 4.62 to 4.58 (dd, 1H), 4.44 to 4.10 (m, 3H), 3.79 to 3.71 (two overlapping s, 6H), 3.33 to 3.00 (m, 2H), 1.78 (s, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 168.8, 160.9, 159.6, 146.9, 143.9, 136.5, 132.6, 132.4, 130.0, 129.5, 129.5, 129.4, 129.3, 129.3, 129.2, 129.1, 129.0, 128.9, 128.5, 128.4, 128.2, 127.9, 126.9, 126.6, 126.1, 118.9, 118.5, 113.0, 112.0, 79.6, 66.9, 63.1, 55.2, 55.1, 52.1, 45.1, 14.1. **HRMS-EI^{+}** (*m/z*): calc'd for $C_{31}H_{31}NO_4$ 481.2253; found 481.2249.


MeO
OMe
O
Ph
O
N
Me
Normalized Intensity
Chemical Shift (ppm)

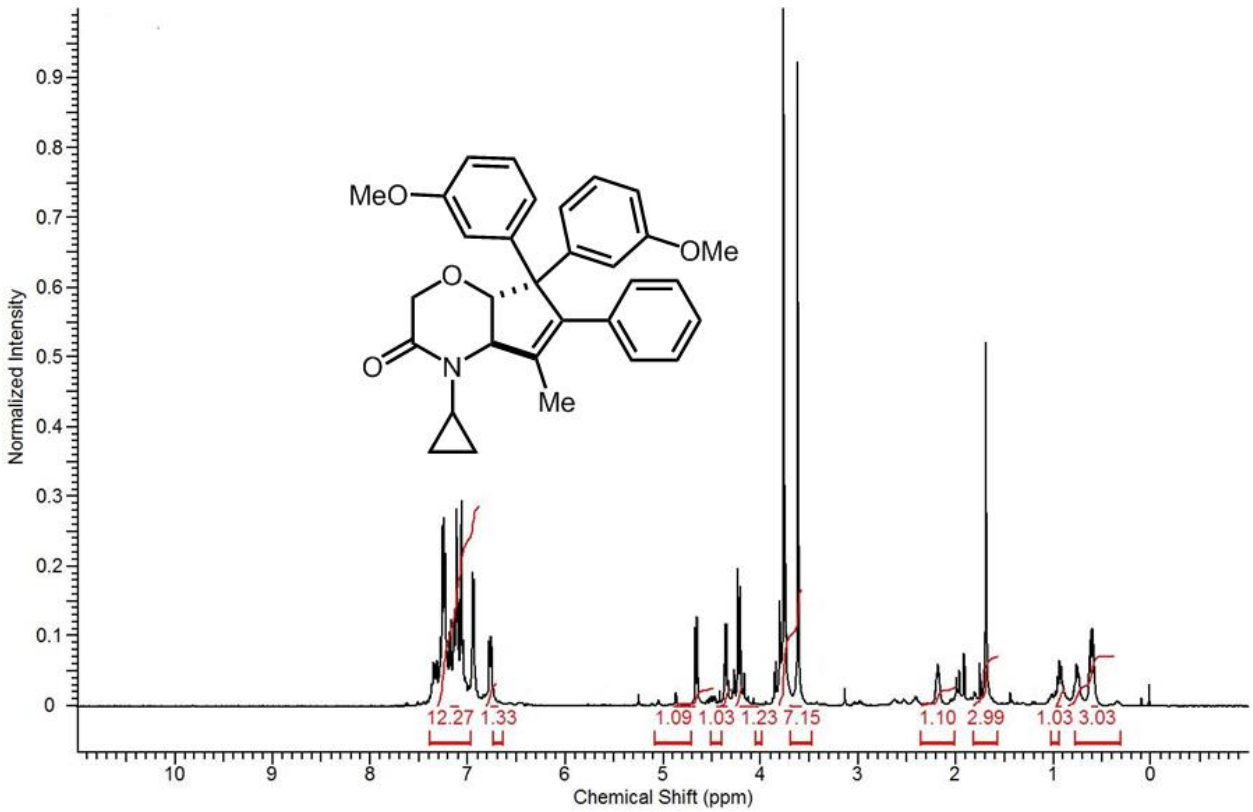

MeO
OMe
O
Ph
O
N
Me
Normalized Intensity
Chemical Shift (ppm)

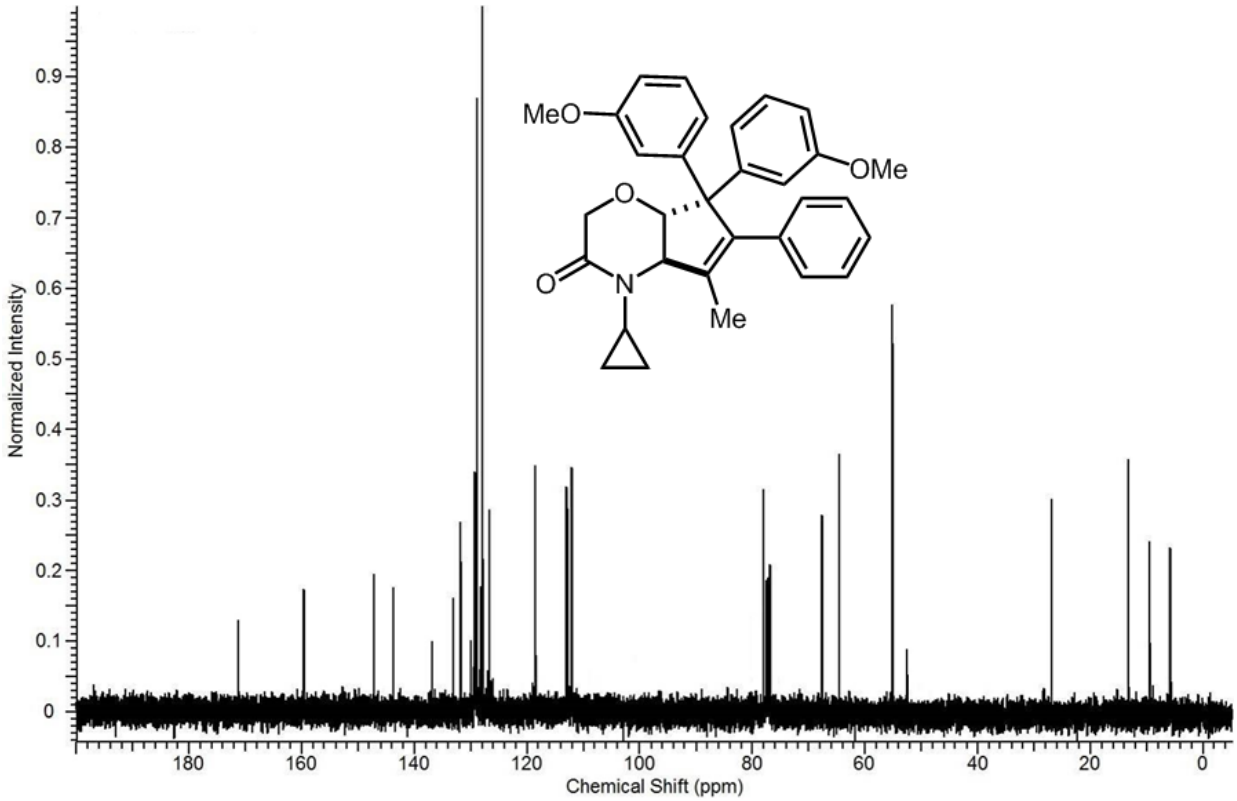


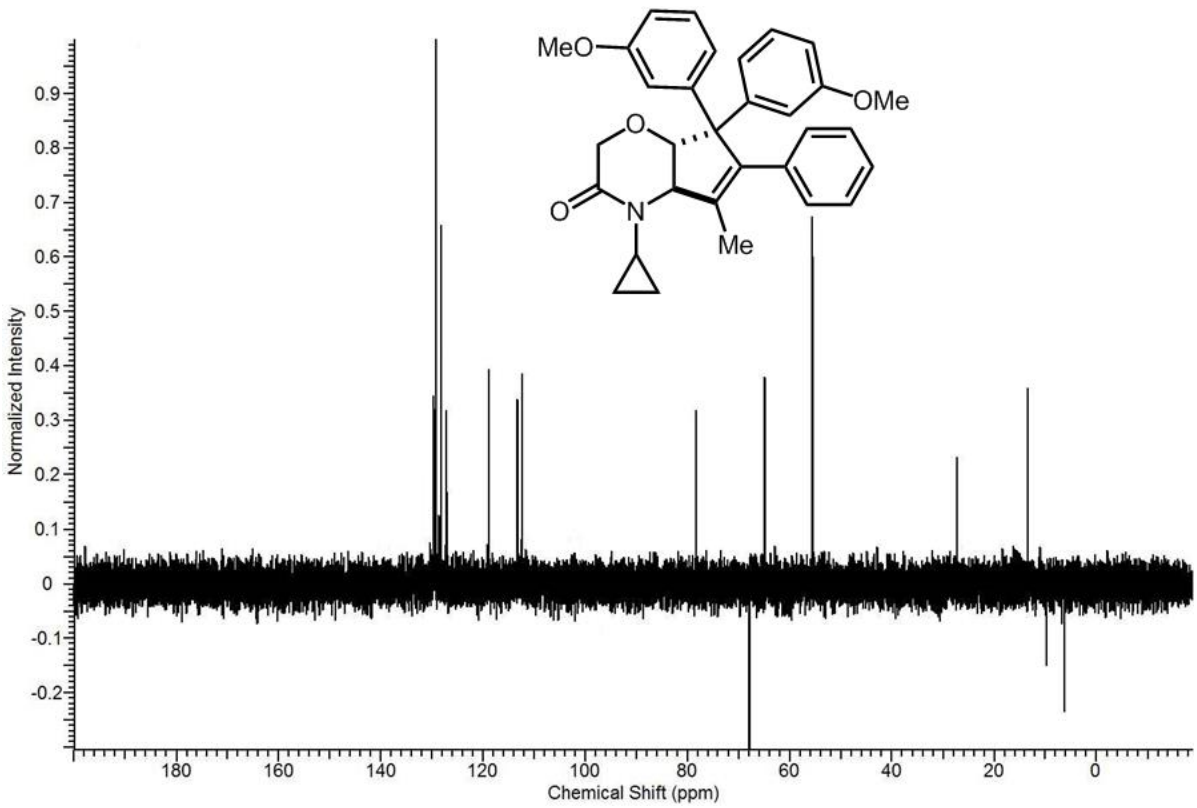


1x; 79%, 91:9 dr

Prepared from alcohol **2x** (125 mg, 0.25 mmol) using General Procedure A. Oily substance. Yield = 95 mg, 79%. ^{1}H NMR (400 MHz, $CDCl_3$) δ 7.39 to 6.93 (m, 12H), 6.82 (d, 1H), 4.87 (d, 1H), 4.42 to 4.18 (m, 2H), 3.86 to 3.59 (m, 7H), 2.20 to 2.16 (m, 1H), 1.76 (s, 3H), 1.04 to 0.94 (m, 1H), 0.86 to 0.54 (m, 3H). ^{13}C NMR (101 MHz, $CDCl_3$) δ 171.3, 159.6, 159.4, 147.1, 143.7, 136.8, 133.1, 131.8, 129.9, 129.4, 129.3, 129.2, 129.1, 128.8, 128.2, 127.9, 126.9, 126.7, 126.5, 118.5, 117.8, 112.9, 112.0, 111.2, 79.2, 67.5, 65.6, 64.6, 55.3, 55.2, 52.0, 26.9, 13.2, 9.4, 5.9. **HRMS-EI^{+}** (*m/z*): calc'd for $C_{31}H_{31}NO_4$ 481.2253; found 481.2259.


MeO
OMe
O
O
N
Me
Normalized Intensity
Chemical Shift (ppm)
12.27 1.33
1.09 1.03 1.23 7.15
1.10 2.99
1.03 3.03


MeO
OMe
O
O
N
Me
Normalized Intensity
Chemical Shift (ppm)


MeO
OMe
O
O
N
Me
Normalized Intensity
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
-0.1
-0.2
180
160
140
120
100
80
60
40
20
0
Chemical Shift (ppm)

References

(1) Braunstein, H.; Langevin, S.; Khim, M.; Adamson, J.; Hovenkotter, K.; Kotlarz, L.; Mansker, B.; Beng, T. K. *Org. Biomol. Chem.* **2016**, *14*, 8864.

(2) Liu, Z.-Q.; Zhang, Y.; Zhao, L.; Li, Z.; Wang, J.; Li, H.; Wu, L.-M. *Org. Lett.* **2011**, *13*, 2208.