Supporting Information for

**Total Synthesis of (−)-Ambiguine P**

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Materials and Methods

Unless stated otherwise, reactions were performed under a nitrogen atmosphere using oven- or flame-dried glassware and stir bars. Ambient temperature refers to 22-26 °C. Higher than ambient temperatures were maintained using pre-heated oil baths. Lower temperatures were maintained using acetone/CO$_2$(s) (−78 °C), MeCN/CO$_2$(s) (−40 °C) and water/ice (0 °C) baths. Dichloromethane (CH$_2$Cl$_2$ or DCM), tetrahydrofuran (THF), and dimethylformamide (DMF) were dried by passage through an activated alumina column purification system (Innovative Technology Inc. Pure Solv$^\text{TM}$). Anhydrous methanol (MeOH) and pyridine were purchased from Sigma-Aldrich and used as received. Anhydrous 1-propanol was purchased from Alfa Aesar and used as received. Commercially obtained reagents were used as received, unless stated otherwise. N-bromosuccinimide was recrystallized from water.

Thin-layer chromatography (TLC) was performed using EMD Millipore silica gel 60 Å plates with UV fluorescence quenching (254 nm), KMnO$_4$, or Seebach’s stain. Flash column chromatography was performed on SiliCycle SiliaFlash P60 (40-63 μm particle size) using ACS or HPLC grade solvents purchased from Fisher Scientific. $^1$H NMR spectra were recorded on Bruker 500 spectrometers (at 500 MHz) at 294-297 K. $^{13}$C NMR spectra were recorded on Bruker 500 and Bruker 400 spectrometers (at 125 MHz and 100 MHz, respectively) at 294-297 K. $^1$H spectra were calibrated from internal standard TMS (δ 0.0) or solvent resonance (CD$_3$OD: δ 3.31). $^{13}$C spectra were calibrated from solvent resonance (CDCl$_3$: δ 77.16, CD$_3$OD: δ 49.00). NMR data are reported as: chemical shift (δ ppm) (multiplicity, coupling constant (Hz), and integration). $^{13}$C chemical shifts arising from $^{19}$F isotope effects are indicated in parentheses. High-resolution mass spectral analysis was measured on Agilent Technologies 6224 TOF LC/MS (electrospray ionization). Optical rotations were measured on a Jasco DIP-1000 polarimeter using a 100 mm or a 50 mm path-length cell, c = g/100mL. IR spectra were recorded on a Thermo Scientific Nicolet iS50 FT-IR spectrometer and are reported as frequency of absorption (cm$^{-1}$).
Experimental Procedures and Characterization Data

**Alcohol 11.** (Prepared according to literature)\(^1\) A 250 mL round-bottomed flask equipped with a magnetic stir bar was charged with ethyl indole-2-carboxylate (3.09 g, 16.3 mmol, 1 equiv.) and THF (46 mL). The resulting yellow solution was cooled to −78 °C. Methyl lithium solution (1.6 M in THF, 50 mL, 80 mmol, 4.9 equiv.) was added dropwise over 15 min, and precipitate formation was observed. After 2.5 h, the reaction was carefully quenched with 25 mL H\(_2\)O, and allowed to warm to ambient temperature. The reaction mixture was partitioned between 100 mL H\(_2\)O and 50 mL 1:1 Et\(_2\)O:hexanes, and the aqueous layer was extracted with 40 mL 1:1 Et\(_2\)O:hexanes. The combined organic layers were washed with brine, dried over Na\(_2\)SO\(_4\), and concentrated under reduced pressure. The crude material was purified by quick passage through a short silica plug (25:75 EtOAc:hexanes) to afford alcohol 11 (2.82 g, 16.1 mmol, 99% yield) as a white solid.

\(R_f = 0.25\) (25:75 EtOAc:hexanes)

\(^{1}\)H NMR (500 MHz, CDCl\(_3\)): δ 8.42 (s, 1H), 7.56 (d, \(J = 7.9\) Hz, 1H), 7.37 – 7.35 (m, 1H), 7.18 – 7.15 (m, 1H), 7.10 – 7.07 (m, 1H), 6.32 – 6.31 (m, 1H), 1.83 (s, 1H), 1.69 (s, 6H).

\(^{13}\)C NMR (100 MHz, CDCl\(_3\)): δ 145.7, 135.7, 128.5, 121.9, 120.5, 119.9, 111.0, 97.2, 69.8, 30.8.

HRMS (ESI): calcd for C\(_{11}\)H\(_{14}\)NO [M+H]\(^+\): 176.1070, found: 176.1053.

**Tricycle 13.** A 10 mL recovery flask equipped with a magnetic stir bar was charged with diene 12 (prepared according to literature)\(^2\) (238 mg, 1.05 mmol, 2 equiv.), alcohol 11 (92.1 mg, 0.526 mmol, 1
equiv.) and DCM (3.5 mL). The resulting colorless solution was cooled to −78 °C. TMSOTf (distilled over P₂O₅ before use; 0.105 mL, 0.578 mmol, 1.1 equiv.) was added dropwise, and the mixture immediately turned deep red. After 1 h, the reaction was quenched with 2 mL H₂O, and allowed to warm to ambient temperature. The reaction mixture was partitioned between 20 mL saturated aqueous NaHCO₃ and 20 mL DCM, and the aqueous layer was extracted with 20 mL DCM. The combined organic layers were washed with 10% HCl (40 mL × 2), dried over Na₂SO₄, and concentrated under reduced pressure. The crude material was purified by flash chromatography (20:80 → 25:75 EtOAc:hexanes) to afford tricycle 13 (74.0 mg, 0.326 mmol, 62% yield) as an off-white solid.

\[ R_f = 0.36 \text{ (25:75 EtOAc:hexanes)} \]

\(^1\text{H NMR (500 MHz, CDCl₃)}: \delta 7.85 (s, 1H), 7.51 (dd, \( J = 7.8, 0.5 \text{ Hz, 1H})\), 7.33 – 7.31 (m, 1H), 7.19 – 7.15 (m, 1H), 7.14 – 7.11 (m, 1H), 3.12 – 3.10 (m, 2H), 2.94 (s, 2H), 2.74 – 2.72 (m, 2H), 1.43 (s, 6H).

\(^{13}\text{C NMR (100 MHz, CDCl₃)}: \delta 212.3, 140.7, 134.7, 128.2, 122.0, 119.6, 118.0, 110.5, 110.4, 54.9, 44.5, 35.2, 30.4, 19.0.

HRMS (ESI): calcd for C₁₅H₁₈NO [M+H]^+: 228.1383, found: 228.1386.

**Tetracycle 15.** A 25 mL recovery flask equipped with a magnetic stir bar was charged with diene 14 (prepared according to literature\(^3\) (282 mg, 1.01 mmol, 2 equiv.), alcohol 11 (88.1 mg, 0.503 mmol, 1 equiv.) and DCM (5 mL). The resulting light yellow solution was cooled to −78 °C. TMSOTf (distilled over P₂O₅ before use; 0.100 mL, 0.553 mmol, 1.1 equiv.) was added dropwise, and the mixture turned more yellow. After 1 h, the reaction was quenched with 0.5 mL H₂O, and allowed to warm to ambient temperature. After 20 min vigorous stirring, the reaction mixture was partitioned between 30 mL 1:1
saturated aqueous NaHCO₃:H₂O and 15 mL DCM, and the aqueous layer was extracted with 15 mL DCM. The combined organic layers were dried over Na₂SO₄ and concentrated under reduced pressure. The crude material was purified by flash chromatography (15:85 → 20:80 EtOAc:hexanes) to afford tetracycle 15 (123 mg, 0.437 mmol, 87% yield) as a white solid.

R_f = 0.31 (15:85 EtOAc:hexanes)

1H NMR (500 MHz, CDCl₃): δ 7.84 (s, 1H), 7.51 (d, J = 7.7 Hz, 1H), 7.32 (dt, J = 8.0, 0.9 Hz, 1H), 7.20 – 7.16 (m, 1H), 7.14 – 7.11 (m, 1H), 3.40 – 3.35 (m, 1H), 3.10 (d, J = 12.9 Hz, 1H), 2.92 (d, J = 3.3 Hz, 1H), 2.73 (d, J = 12.9 Hz, 1H), 2.47 – 2.45 (m, 1H), 2.03 – 2.00 (m, 1H), 1.87 – 1.83 (m, 1H), 1.64 – 1.59 (m, 1H), 1.57 – 1.44 (m, 4H), 1.41 (s, 3H), 1.41 (s, 3H).

13C NMR (100 MHz, CDCl₃): δ 213.4, 140.7, 134.7, 128.5, 122.1, 119.6, 118.0, 114.8, 110.6, 58.5, 51.4, 36.4, 34.3, 32.3, 31.0, 29.7, 29.4, 27.1, 22.7.

HRMS (ESI): calcd for C₁₉H₂₄NO [M+H]^⁺: 282.1852, found: 282.1856.

**Enol triflate 17.** Ketone 16 was prepared according to the procedure reported by Baran and coworkers.⁴ The starting material for Baran’s procedure, (4S,8R/S)-(−)-p-menth-1-en-9-ol (CAS# 937035-21-7), was prepared by hydroboration/oxidation of (S)-(−)-limonene.⁵

A 250 mL round-bottomed flask with a magnetic stir bar was charged with ketone 16 (2.41 g, 13.5 mmol, 1 equiv.) and THF (48 mL). The colorless solution was cooled to −78 °C. KHMDS (1.0 M in THF, 17.6 mL, 17.6 mmol, 1.3 equiv.) was added dropwise, and the resulting yellow mixture was stirred at −78 °C for 1.5 h. Comins’ reagent (8.50 g, 21.7 mmol, 1.6 equiv.) was transferred in using a total of 20 mL THF, and stirring was continued for another 1.5 h. The reaction was quenched with 20 mL saturated aqueous NH₄Cl, and allowed to warm up ambient temperature. The reaction mixture was partitioned
between 200 mL 1:1 brine:H$_2$O and 100 mL 1:1 Et$_2$O:hexanes, and the aqueous layer was extracted with 70 mL 1:1 Et$_2$O:hexanes. The combined organic layers were washed with brine, dried over Na$_2$SO$_4$, and concentrated under reduce pressure. The crude material was purified by flash chromatography (0:100 → 1:99 EtOAc:hexanes) to afford enol triflate 17 (3.74 g, 12.1 mmol, 89% yield) as a pale yellow oil.

$R_f = 0.33$ (3:97 EtOAc:hexanes)

$^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 5.74 (dd, $J = 17.4, 10.6$ Hz, 1H), 5.71 (d, $J = 8.1$ Hz, 1H), 5.17 (dd, $J = 10.6, 0.7$ Hz, 1H), 5.12 (dd, $J = 17.4, 0.6$ Hz, 1H), 4.90 – 4.89 (m, 1H), 4.76 (s, 1H), 2.94 (dd, $J = 9.8, 5.2$ Hz, 1H), 1.83 – 1.76 (m, 1H), 1.77 (s, 3H), 1.73 – 1.68 (m, 1H), 1.65 – 1.61 (m, 1H), 1.58 – 1.52 (m, 1H), 1.27 (s, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 153.4, 146.0, 141.7, 120.5, 118.5 (q, $J_{19F-13C} = 317$ Hz; actual signals at 123.3, 120.1, 116.9, 113.7), 114.9, 112.9, 42.4, 41.7, 34.7, 23.4, 22.6, 21.6.

HRMS (ESI): calcd for C$_{13}$H$_{18}$F$_3$O$_3$S [M+H]$^+$: 311.0923, found: 311.0925.

$[\alpha]^{24}_D = -89.4^\circ$ (c = 0.725, CHCl$_3$).

Enone 18. (Procedure adapted from Stoltz et al.)

A 500 mL round-bottomed flask with a magnetic stir bar was charged with enol triflate 17 (3.66 g, 11.8 mmol, 1 equiv.) and DMF (18 mL). CuI (225 mg, 1.18 mmol, 0.1 equiv.), Pd(dppf)Cl$_2$•DCM (963 mg, 1.18 mmol, 0.1 equiv.), and LiCl (flame-dried before use; 2.65 g, 62.5 mmol, 5.3 equiv.) were added sequentially. Another 100 mL DMF was cannulated in, and tributyl(1-ethoxyvinyl)tin (5.98 mL, 17.7 mmol, 1.5 equiv.) was added. The mixture was frozen in a liquid N$_2$ bath and degassed by three freeze-pump-thaw cycles. The reaction mixture was then placed in a pre-heated 40 $^\circ$C oil bath. After 14 h, the reaction mixture was cooled to ambient temperature and diluted with 200 mL H$_2$O. The mixture was then partitioned between 30 mL H$_2$O and 100 mL 1:1 Et$_2$O:hexanes,
and the aqueous layer was extracted with 1:1 Et$_2$O:hexanes (100 mL × 2). The combined organic layers were washed with 1 N HCl (× 2), H$_2$O (× 2), and brine (× 2), dried over Na$_2$SO$_4$, and concentrated under reduced pressure. The crude ethyl enol ether was dissolved in 150 mL DCM, and 75 mL 2 N HCl was added. The reaction mixture was stirred vigorously at ambient temperature for 1.75 h. The mixture was then partitioned between 50 mL H$_2$O and 60 mL DCM, and the aqueous layer was extracted with 70 mL DCM. The combined organic layer was washed with saturated aqueous NaHCO$_3$, dried over Na$_2$SO$_4$, and concentrated under reduced pressure. The resulting crude material was filtered through a plug of silica to remove residual solids using 25:75 Et$_2$O:hexanes as the eluent, and then concentrated under reduced pressure. The crude mixture was purified by flash chromatography (6:94 → 8:92 Et$_2$O:hexanes) to afford enone 18 (1.95 g, 9.54 mmol, 81% yield) as a yellow oil.

$R_f = 0.40$ (10:90 Et$_2$O:hexanes)

$^{1}$H NMR (500 MHz, CDCl$_3$): $\delta$ 6.74 (d, $J = 3.5$ Hz, 1H), 5.91 (dd, $J = 17.5, 10.6$ Hz, 1H), 4.98 (dd, $J = 10.6, 1.2$ Hz, 1H), 4.87 – 4.86 (m, 1H), 4.86 (dd, $J = 17.5, 1.2$ Hz, 1H), 4.70 – 4.69 (m, 1H), 2.94 – 2.91 (m, 1H), 2.28 (s, 3H), 1.82 – 1.74 (m, 1H), 1.79 (m, 3H), 1.59 – 1.52 (m, 3H), 1.33 (s, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 199.6, 147.2, 146.5, 145.9, 143.6, 112.1, 111.6, 43.9, 39.6, 36.4, 27.8, 25.7, 23.0, 21.6.

HRMS (ESI): calcd for C$_{14}$H$_{21}$O [M+H]$^+$: 205.1587, found: 205.1592.

$[\alpha]^{24.3}_D = -138^\circ$ (c = 0.865, CHCl$_3$).

TBS enol ether 19. A 250 mL round-bottomed flask with a magnetic stir bar was charged with TBSCl (2.30 g, 15.3 mmol, 1.5 equiv.) and THF (36 mL). The resulting colorless solution was cooled to $-78$ °C, and KHMDS (1.0 M in THF, 15.3 mL, 15.3 mmol, 1.5 equiv.) was added dropwise. After 5 min, enone 18
(2.08 g, 10.2 mmol, 1 equiv.) was added slowly over 25 min using a total of 15 mL THF. The reaction mixture was stirred at −78 °C for 1.5 h, and diluted with 150 mL hexanes. The reaction mixture was allowed to warm to ambient temperature, and filtered through a pad of Celite using 20:80 Et₂O:hexanes as the eluent. The mixture was concentrated under reduced pressure to provide the crude TBS enol ether 19 as a yellow oil, which was carried forward without further purification.

**Tricycle 23.** TBS enol ether 19 obtained in the previous step was divided evenly into three portions, and three identical reactions were performed. After all the reactions were quenched, they were combined, and subjected to further work-up and purification.

Each of the three 50 mL recovery flask with a magnetic stir bar was charged with alcohol 11 (307 mg, 1.75 mmol, 1 equiv.) and DCM (5.5 mL). TBS enol ether 19 (3.40 mmol, 1.94 equiv.) was added as a solution in 12 mL DCM. The resulting yellow solution was cooled to −78 °C. TMSOTf (distilled over P₂O₅ before use; 0.332 mL, 1.84 mmol, 1.05 equiv.) was added dropwise, and the mixture turned deep red. After 35 min, the reaction was quenched with 10 mL saturated aqueous NaHCO₃, and allowed to warm to ambient temperature.

The three reaction mixtures were combined and partitioned between 50 mL 1:1 saturated aqueous NaHCO₃:H₂O and 30 mL DCM. The aqueous layer was extracted with 15 mL DCM, and the combined organic layers were dried over Na₂SO₄ and concentrated under reduced pressure. Purification by flash chromatography (6:94 → 7:93 EtOAc:hexanes) provided tricycle 23 (1.075 g, 2.97 mmol, 57% yield) as a yellow gel. Enone 18, which was generated from hydrolysis of unreacted 19, was recovered and reused.

\[ R_f = 0.38 \ (10:90 \text{ EtOAc:hexanes}) \]
$^1$H NMR (500 MHz, CDCl$_3$): δ 9.26 (s, 1H), 7.51 (dd, $J = 7.8$, 1.0 Hz, 1H), 7.34 – 7.31 (m, 1H), 7.12 – 7.09 (m, 1H), 7.05 – 7.02 (m, 1H), 6.54 (d, $J = 3.5$ Hz, 1H), 6.22 (dd, $J = 2.1$, 0.9 Hz, 1H), 5.88 (dd, $J = 17.5$, 10.6 Hz, 1H), 5.00 (dd, $J = 10.6$, 1.1 Hz, 1H), 4.87 (dd, $J = 17.5$, 1.1 Hz, 1H), 4.82 – 4.81 (m, 1H), 4.59 (m, 1H), 3.02 (d, $J = 16.6$ Hz, 1H), 2.94 (d, $J = 16.6$ Hz, 1H), 2.77 – 2.74 (m, 1H), 1.75 – 1.70 (m, 1H), 1.72 – 1.71 (m, 3H), 1.51 – 1.46 (m, 3H), 1.50 (s, 6H), 1.31 (s, 3H).

$^{13}$C NMR (125 MHz, CDCl$_3$): δ 202.7, 147.1, 147.0, 146.5, 146.4, 142.5, 136.0, 128.1, 121.2, 120.0, 119.5, 112.1, 111.9, 110.9, 97.5, 51.4, 43.8, 39.9, 36.5, 34.6, 29.0, 28.8, 25.7, 22.9, 21.5.

HRMS (ESI): calcd for C$_{25}$H$_{32}$NO [M+H]$^+$: 362.2478, found: 362.2488.

[α]$^{24.7}_D = −76.6^\circ$ (c = 0.515, CHCl$_3$).

**Tetracycle 21.** A 50 mL recovery flask with a magnetic stir bar was charged with tricycle 23 (566 mg, 1.56 mmol, 1 equiv.) and 1-propanol (5 mL) to afford a yellow solution. A solution of NaAuCl$_4$•2H$_2$O (156 mg, 0.391 mmol, 0.25 equiv.) in 8 mL 1-propanol was prepared. 1 mL of the NaAuCl$_4$•2H$_2$O solution was added quickly into the substrate solution, and the rest of the catalyst solution was added slowly via a syringe pump in the dark over 20 h. The reaction mixture was stirred for an additional 7.5 h in the dark at ambient temperature, and then quenched with 10 mL saturated aqueous NaHCO$_3$. The mixture was partitioned between 50 mL 1:1 saturated aqueous NaHCO$_3$:H$_2$O and 40 mL 1:1 EtOAc:hexanes, and the aqueous layer was extracted with 50 mL 1:1 EtOAc:hexanes. The combined organic layers were washed with brine, dried over Na$_2$SO$_4$, and concentrated under reduced pressure. The crude material was purified via flash chromatography (25:75 → 35:65 Et$_2$O:hexanes) to give tetracycle 21 (315 mg, 0.871 mmol, 56% yield) as a pale yellow solid.
$R_f = 0.38$ (30:70 Et$_2$O:hexanes)

$^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 7.90 (s, 1H), 7.54 (d, $J = 8.0$ Hz, 1H), 7.29 – 7.26 (m, 1H), 7.12 (ddd, $J = 8.1$, 7.0, 1.1 Hz, 1H), 7.03 (ddd, $J = 8.0$, 7.0, 1.0 Hz, 1H), 5.96 (dd, $J = 17.6$, 10.9 Hz, 1H), 4.95 (dd, $J = 17.6$, 1.2 Hz, 1H), 4.93 (dd, $J = 10.9$, 1.2 Hz, 1H), 4.73 (m, 1H), 4.50 – 4.49 (m, 1H), 3.64 (dd, $J = 11.7$, 4.8 Hz, 1H), 2.93 (dd, $J = 4.7$, 1.1 Hz, 1H), 2.71 (d, $J = 10.3$ Hz, 1H), 2.63 – 2.56 (m, 1H), 2.59 (d, $J = 10.3$ Hz, 1H), 2.28 (td, $J = 12.0$, 3.3 Hz, 1H), 2.00 – 1.91 (m, 1H), 1.73 (ddd, $J = 13.6$, 7.6, 3.2 Hz, 1H), 1.52 (s, 3H), 1.51 – 1.47 (m, 1H), 1.38 (s, 3H), 1.27 (s, 3H), 1.19 (s, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 211.6, 148.8, 148.5, 140.3, 133.9, 129.8, 121.7, 120.0, 118.9, 113.5, 111.4, 110.7, 110.2, 62.7, 58.6, 45.4, 39.5, 36.1, 34.6, 31.6, 29.9, 29.8, 28.4, 25.7, 23.3.

HRMS (ESI): calcd for C$_{25}$H$_{32}$NO [M+H]$^+$: 362.2478, found: 362.2489.

$[\alpha]_{D}^{24.6} = +7.9^\circ$ (c = 0.905, CHCl$_3$).

**Pentacycle 24.** A 100 mL recovery flask with a magnetic stir bar was charged with tetracycle 21 (481 mg, 1.33 mmol, 1 equiv.) and DCM (22 mL). The resulting yellow solution was cooled to 0 °C. BF$_3$•OEt$_2$ (0.657 mL, 5.32 mmol, 4 equiv.) was added dropwise, and the mixture turned deep red. After 5 min, MeOH (anhydrous, 0.054 mL, 1.33 mmol, 1 equiv.) was added dropwise. The reaction mixture was stirred at 0 °C for 24 h, and was then quenched with 10 mL saturated aqueous NaHCO$_3$ and warmed to ambient temperature. The mixture was partitioned between 40 mL 1:1 saturated aqueous NaHCO$_3$:H$_2$O and 25 mL DCM, and the aqueous layer was extracted with 25 mL DCM. Then the combined organic layers were washed with brine, dried over Na$_2$SO$_4$, and concentrated under reduced pressure. The crude material was purified by flash chromatography (30:70 → 40:60 Et$_2$O:hexanes) to provide pentacycle 24 (390 mg, 1.08 mmol, 81% yield) as a yellow solid.
$R_f = 0.40$ (40:60 Et$_2$O:hexanes)

$^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 7.72 (s, 1H), 7.10 – 7.05 (m, 2H), 6.97 (dd, $J = 6.3$, 1.6 Hz, 1H), 5.94 (dd, $J = 17.6$, 10.8 Hz, 1H), 5.08 (dd, $J = 17.6$, 1.2 Hz, 1H), 4.97 (dd, $J = 10.8$, 1.2 Hz, 1H), 3.80 (d, $J = 7.4$ Hz, 1H), 3.35 (dd, $J = 10.9$, 7.5 Hz, 1H), 2.76 (d, $J = 14.7$ Hz, 1H), 2.55 (d, $J = 14.7$ Hz, 1H), 2.44 – 2.38 (m, 1H), 1.87 – 1.84 (m, 1H), 1.81 (s, 3H), 1.65 – 1.48 (m, 3H), 1.50 (s, 3H), 1.39 (s, 3H), 1.24 (s, 3H), 1.23 (s, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ 210.5, 148.0, 141.3, 137.7, 132.4, 126.9, 122.4, 113.5, 111.7, 110.2, 107.6, 64.9, 54.9, 45.4, 37.8, 37.6, 33.1, 32.8, 32.7, 31.1, 29.8, 25.5, 25.1, 25.0, 20.5.

HRMS (ESI): calcd for C$_{25}$H$_{32}$NO [M+H]$^+$: 362.2478, found: 362.2485. 

$[\alpha]^{21.3}_D = -106^\circ$ (c = 0.510, CHCl$_3$).

**Enone 26.** A 100 mL recovery flask with a magnetic stir bar was charged with pentacycle 24 (390 mg, 1.08 mmol, 1 equiv.) and THF (18.9 mL) to afford a yellow solution. H$_2$O (2.1 mL) was added, and the yellow solution was cooled to 0 °C. DDQ (1.71 g, 7.54 mmol, 7 equiv.) was added in two portions, and the mixture turned deep red. After 5 min, the ice/water bath was removed, and the deep red solution was stirred at ambient temperature for 2 h. The reaction mixture was quenched with 50 mL 2.5 N NaOH. The mixture was partitioned between 50 mL 1:1 2.5 N NaOH:H$_2$O and 60 mL EtOAc, and the aqueous layer was extracted with EtOAc (40 mL × 2). The combined organic layers were washed with 2.5 N NaOH and brine, dried over Na$_2$SO$_4$, and concentrated under reduced pressure. Purification by flash chromatography (20:80 EtOAc:hexanes) afforded enone 26 (374 mg, 1.04 mmol, 96% yield) as a yellow solid.
$R_f = 0.40$ (20:80 EtOAc:hexanes)

$^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 8.00 (s, 1H), 7.18 – 7.12 (m, 2H), 7.03 (dd, $J = 6.7$, 1.3 Hz, 1H), 5.89 (dd, $J = 17.5$, 10.7 Hz, 1H), 5.04 (dd, $J = 17.5$, 1.2 Hz, 1H), 4.99 (dd, $J = 10.7$, 1.2 Hz, 1H), 3.15 (d, $J = 11.5$ Hz, 1H), 2.70 (dd, $J = 11.1$, 6.5 Hz, 1H), 2.55 (d, $J = 11.5$ Hz, 1H), 2.02 – 1.96 (m, 1H), 1.91 – 1.83 (m, 1H), 1.74 (td, $J = 13.3$, 3.3 Hz, 1H), 1.64 – 1.60 (m, 1H), 1.53 (s, 6H), 1.46 (s, 3H), 1.34 (s, 3H), 1.03 (s, 3H).

$^{13}$C NMR (125 MHz, CDCl$_3$): $\delta$ 204.4, 147.7, 141.8, 141.0, 140.1, 135.6, 132.7, 125.5, 123.1, 113.6, 111.7, 108.3, 107.9, 56.1, 47.7, 40.5, 38.9, 37.8, 35.0, 29.0, 28.1, 25.7, 25.3, 23.7, 20.0.

HRMS (ESI): calcd for C$_{25}$H$_{30}$NO [M+H]$^+$: 360.2322, found: 360.2330.

$[\alpha]^{22}_D = +59.1^\circ$ (c = 0.390, CHCl$_3$).

**N-Boc indole 27.** A 100 mL recovery flask with a magnetic stir bar was charged with enone 26 (373 mg, 1.04 mmol, 1 equiv.) and DCM (15 mL). DMAP (12.7 mg, 0.104 mmol, 0.1 equiv.), Et$_3$N (0.723 mL, 5.12 mmol, 5 equiv.), and Boc$_2$O (906 mg, 4.15 mmol, 4 equiv.) were added, followed by addition of another 10 mL DCM. After 13 h stirring at ambient temperature, the reaction mixture was concentrated under reduced pressure to give the crude material as a brown oil. Purification via flash chromatography (6:94 → 8:92 EtOAc:hexanes) afforded $N$-Boc indole 27 (456 mg, 0.992 mmol, 95% yield) as a yellow solid.

$R_f = 0.45$ (10:90 EtOAc:hexanes)

$^1$H NMR (500 MHz, CDCl$_3$): $\delta$ 7.52 (dd, $J = 8.3$, 0.6 Hz, 1H), 7.24 (dd, $J = 8.2$, 7.5 Hz, 1H), 7.13 (dd, $J = 7.4$, 0.6 Hz, 1H), 6.05 (dd, $J = 17.5$, 10.7 Hz, 1H), 4.97 (dd, $J = 17.5$, 1.0 Hz, 1H), 4.95 (dd, $J = 10.7$, 0.6 Hz, 1H), 3.15 (d, $J = 11.5$ Hz, 1H), 2.70 (dd, $J = 11.1$, 6.5 Hz, 1H), 2.55 (d, $J = 11.5$ Hz, 1H), 2.02 – 1.96 (m, 1H), 1.91 – 1.83 (m, 1H), 1.74 (td, $J = 13.3$, 3.3 Hz, 1H), 1.64 – 1.60 (m, 1H), 1.53 (s, 6H), 1.46 (s, 3H), 1.34 (s, 3H), 1.03 (s, 3H).
1.0 Hz, 1H), 2.92 (d, \( J = 12.8 \) Hz, 1H), 2.74 (d, \( J = 12.8 \) Hz, 1H), 2.64 (dd, \( J = 10.7, 6.4 \) Hz, 1H), 1.98 – 1.93 (m, 1H), 1.83 – 1.75 (m, 1H), 1.70 (s, 9H), 1.68 – 1.59 (m, 2H), 1.54 (s, 3H), 1.51 (s, 3H), 1.48 (s, 3H), 1.47 (s, 3H), 1.05 (s, 3H).

\(^{13}\)C NMR (100 MHz, CDCl\(_3\)): \( \delta \) 200.5, 151.4, 149.3, 146.2, 142.2, 142.1, 141.2, 134.4, 125.3, 124.8, 116.5, 116.1, 111.1, 109.8, 84.9, 60.7, 49.4, 40.4, 38.5, 38.3, 35.1, 28.2, 28.0, 25.0, 24.8, 23.9, 22.8, 20.2.

HRMS (ESI): calcd for C\(_{30}\)H\(_{38}\)NO\(_3\) [M+H]\(^+\): 460.2846, found: 460.2852.

\([\alpha]\)\(^{23,6}\) = +63.4° (c = 0.222, CHCl\(_3\)).

**Allylic alcohol 28.** A 25 mL recovery flask with a magnetic stir bar was charged with N-Boc indole 27 (105 mg, 0.228 mmol, 1 equiv.) and THF (6.5 mL). The resulting yellow solution was cooled to \(-40 \) °C. LiAl(OMe)\(_3\)H (freshly prepared from 1 M LiAlH\(_4\) in THF and 3 equiv. of MeOH; 1 M in THF, 1.59 mL, 7 equiv.) was added dropwise. The reaction mixture was stirred for 2.5 h, during which time the MeCN/CO\(_2\)(s) bath was allowed to warm to \(-10 \) °C, and then carefully quenched with 0.07 mL H\(_2\)O, 0.14 mL 2 N NaOH and 0.14 mL H\(_2\)O. After warming up to ambient temperature, the reaction was diluted with Et\(_2\)O and dried over MgSO\(_4\). The mixture was then filtered through a pad of Celite, and concentrated under reduced pressure to give the crude material. Purification via flash chromatography (6:94 \( \rightarrow \) 10:90 EtOAc:hexanes) provided allylic alcohol 28 (102 mg, 0.221 mmol, 97% yield) as a while solid.

\( R_f = 0.42 \) (10:90 EtOAc:hexanes)

\(^1\)H NMR (500 MHz, CDCl\(_3\)): \( \delta \) 7.51 (dd, \( J = 8.3, 0.6 \) Hz, 1H), 7.21 (dd, \( J = 8.3, 7.4 \) Hz, 1H), 7.09 (dd, \( J = 7.4, 0.6 \) Hz, 1H), 6.20 (dd, \( J = 17.7, 10.6 \) Hz, 1H), 5.33 (dd, \( J = 17.7, 1.2 \) Hz, 1H), 5.26 (dd, \( J = 10.6, 1.2 \) Hz, 1H), 4.58 (ddd, \( J = 12.0, 7.7, 2.2 \) Hz, 1H), 2.81 (d, \( J = 12.1 \) Hz, 1H, exchangeable with D\(_2\)O), 2.49
(ddd, J = 9.4, 6.9, 2.2 Hz, 1H), 2.18 (ddd, J = 12.8, 7.8, 1.0 Hz, 1H), 1.91 (d, J = 12.9 Hz, 1H), 1.91 – 1.86 (m, 1H), 1.82 – 1.74 (m, 1H), 1.76 (s, 3H), 1.70 (s, 9H), 1.61 (td, J = 13.1, 3.1 Hz, 1H), 1.53 (s, 3H), 1.49 – 1.45 (m, 1H), 1.44 (s, 6H), 0.98 (s, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$): δ 151.7, 149.0, 146.5, 142.9, 142.1, 134.9, 125.4, 124.3, 123.2, 115.6, 115.2, 113.5, 111.7, 84.0, 72.7, 59.2, 46.3, 41.4, 38.8, 38.3, 38.1, 29.6, 28.3, 28.2, 24.8, 23.8, 23.5, 20.4.

HRMS (ESI): calcd for C$_{30}$H$_{40}$NO$_3$ [M+H]$^+$: 462.3003, found: 462.3011.

$[\alpha]^{23.8}_D = +95.2^\circ$ (c = 0.251, CHCl$_3$).

**N-Boc Diene 29.** A 25 mL recovery flask with a magnetic stir bar was charged with allylic alcohol 28 (77.7 mg, 0.168 mmol, 1 equiv.) and DCM (6.7 mL). The resulting colorless solution was cooled to 0°C. Without weighing, Martin sulfurane (ca. 200 mg, 0.29 mmol, 1.7 equiv.) was added, and the mixture turned yellow. After 40 min stirring at 0°C, the reaction mixture was diluted with 6 mL saturated aqueous NaHCO$_3$, and allowed to warm to ambient temperature. Then the mixture was partitioned between 20 mL 1:1 saturated aqueous NaHCO$_3$:H$_2$O and 20 mL DCM, and the aqueous layer was extracted with 15 mL DCM. The combined organic layers were dried over Na$_2$SO$_4$, and concentrated under reduced pressure. The crude material was purified by flash chromatography (3:97 → 4:96 EtOAc:hexanes) to afford N-Boc diene 29 (72.1 mg) as a white solid, containing an impurity that was removed after the next step.

$R_f = 0.46$ (5:95 EtOAc:hexanes)
Diene 7. A 5 mL round-bottomed flask fitted with a condenser containing a magnetic stir bar was charged with N-Boc diene 29 (72.1 mg, containing an impurity, see previous page), and THF (1.8 mL) to afford a colorless solution. Sodium methoxide (25 wt% in MeOH; 0.557 mL, 2.44 mmol, 15 equiv.) was added, followed by additional 0.5 ml THF. The reaction was then placed in a pre-heated oil bath and stirred at reflux. After 3.5 h, additional sodium methoxide (25 wt% in MeOH; 0.186 mL, 0.813 mmol, 5 equiv.) was added, and stirring was continued for another 3 h. The reaction was cooled to ambient temperature, and partitioned between 20 mL 1:1 saturated aqueous NaHCO₃·H₂O and 20 mL DCM. The aqueous layer was extracted with 15 mL DCM, and the combined organic layers were dried over Na₂SO₄ and concentrated under reduced pressure. Purification via flash chromatography (8:92 → 12:88 Et₂O:hexanes) gave diene 7 (50.4 mg, 0.147 mmol, 87% yield over 2 steps) as a light yellow solid.

\[ R_f = 0.43 \text{ (10:90 EtOAc:hexanes)} \]

\(^1\)H NMR (500 MHz, CDCl₃): \( \delta \) 7.80 (s, 1H), 7.16 – 7.12 (m, 2H), 7.02 (dd, \( J = 5.3 \), 2.6 Hz, 1H), 5.92 (dd, \( J = 11.4 \), 0.9 Hz, 1H), 5.85 (dd, \( J = 17.5 \), 10.6 Hz, 1H), 5.33 (dd, \( J = 11.4 \), 0.9 Hz, 1H), 5.09 (dd, \( J = 17.5 \), 1.4 Hz, 1H), 5.02 (dd, \( J = 10.6 \), 1.4 Hz, 1H), 2.88 (dd, \( J = 11.0 \), 7.2 Hz, 1H), 2.00 – 1.95 (m, 1H), 1.95 – 1.87 (m, 1H), 1.71 (td, \( J = 13.0 \), 3.9 Hz, 1H), 1.66 – 1.62 (m, 1H), 1.64 (s, 3H), 1.53 (s, 3H), 1.40 (s, 3H), 1.03 (s, 3H), 0.99 (s, 3H).

\(^{13}\)C NMR (100 MHz, CDCl₃): \( \delta \) 149.2, 140.8, 138.1, 134.8, 132.9, 130.1, 129.5, 128.2, 125.5, 122.8, 113.4, 111.0, 110.3, 108.1, 46.7, 40.3, 39.6, 36.8, 35.4, 26.9, 25.7, 25.0, 23.7, 23.6, 19.8.

HRMS (ESI): calcd for C_{25}H_{30}N [M+H]⁺: 344.2373, found: 344.2378.

\([\alpha]^{25.0}_{D} = -25.8^\circ \text{ (c = 0.580, CHCl₃)}\).
(-)-Ambiguine P (5). A 2-dram vial equipped with a magnetic stir bar and a screw cap containing a Teflon-faced silicon septum was charged with diene 7 (6.3 mg, 0.0183 mmol, 1 equiv.). 0.75 mL DCM and 0.75 mL pyridine (both were sparged with Argon for 40 min before use) were added sequentially to afford a light yellow solution, and H₂O (0.040 mL, 2.20 mmol, 120 equiv.) was added. After 5 min of stirring, the light yellow solution was cooled to −40 °C, and NBS (3.3 mg, 0.0183 mmol, 1 equiv.) was added dropwise as a 0.15 mL DCM solution (prepared with 32.6 mg NBS and 1.50 mL DCM). The resulting orange solution was stirred at −40 °C for 25 min, and quenched with a mixture of 1 mL saturated aqueous Na₂S₂O₃ and 1 mL saturated aqueous NaHCO₃. The mixture was allowed to warm to ambient temperature, and partitioned between 20 mL 1:1 saturated aqueous NaHCO₃:H₂O and 10 mL DCM. The aqueous layer was extracted with DCM (10 mL × 2), and the combined organic layers were dried over Na₂SO₄ and concentrated under reduced pressure. Purification by preparative thin layer chromatography (25:75 EtOAc:hexanes) gave ambiguine P (5) as a 2.0:1 mixture of diastereomers at C-15 (4.1 mg, 0.0114 mmol, 62% yield) as a white solid. Further subjection of the diastereomers to HPLC purification (OD-H column, 250 × 4.6 mm, 5:95 1PrOH:hexanes, 1 mL/min, 254 nm, multiple injections) afforded (-)-ambiguine P (5) (2.6 mg, 0.00723 mmol, 39%) as a white solid, and 15-epi-ambiguine P (1.2 mg, 0.00334 mmol, 18%) as a white solid.

(-)-ambiguine P (5):

R₁ = 0.51 (20:80 EtOAc:hexanes)

¹H NMR (500 MHz, CD₃OD): δ 7.16 (d, J = 8.0 Hz, 1H), 7.07 (dd, J = 7.9, 7.2 Hz, 1H), 6.95 (d, J = 7.2 Hz, 1H), 5.93 (d, J = 11.5 Hz, 1H), 5.89 (dd, J = 17.4, 10.5 Hz, 1H), 5.40 (d, J = 11.5 Hz, 1H), 5.11 (dd, J = 10.5, 1.8 Hz, 1H), 4.93 (dd, J = 17.4, 1.8 Hz, 1H), 2.18 – 2.12 (m, 1H), 1.99 – 1.93 (m, 1H), 1.79 (ddd, 1H), 1.28 (dd, J = 10.5, 1.8 Hz, 1H), 1.12 (ddd, J = 10.5, 1.8 Hz, 1H), 0.88 (s, 3H), 0.86 (s, 3H), 0.84 (s, 3H), 0.83 (s, 3H), 0.82 (s, 3H), 0.81 (s, 3H), 0.80 (s, 3H), 0.79 (s, 3H).
$J = 13.6, 3.8, 2.7$ Hz, 1H), $1.68$ (s, 3H), $1.60$ (dt, $J = 13.3, 3.4$ Hz, 1H), $1.53$ (s, 3H), $1.23$ (s, 3H), $1.02$ (s, 3H), $1.00$ (s, 3H).

$^{13}$C NMR (125 MHz, CD$_3$OD): δ 146.9, 142.2, 139.2, 135.0, 133.9, 133.5, 133.3, 128.6, 125.6, 123.5, 114.9, 114.6, 108.8, 77.2, 45.9, 42.5, 36.7, 34.0, 29.2, 28.9, 27.5, 27.1, 26.4, 18.6.

IR (Neat film): 3552, 3306, 2960, 2922, 1559, 1458, 1360, 1320, 1265, 1156, 1089, 1029, 751, 699.

HRMS (ESI): calcd for C$_{25}$H$_{30}$NO [M+H]$^+$: 360.2322, found: 360.2311.

$[\alpha]^{24.6}_{D} = -184^\circ$ (c = 0.059, MeOH).

15-epi-ambiguine P:

$R_f = 0.51$ (20:80 EtOAc:hexanes)

$^1$H NMR (500 MHz, CD$_3$OD): δ 7.17 – 7.15 (m, 1H), 7.09 – 7.06 (m, 1H), 6.96 (d, $J = 6.9$ Hz, 1H), 5.89 (d, $J = 11.5$ Hz, 1H), 5.87 (dd, $J = 17.6, 10.7$ Hz, 1H), 5.34 (d, $J = 11.5$ Hz, 1H), 5.11 (dd, $J = 17.6, 1.4$ Hz, 1H), 5.00 (dd, $J = 10.7, 1.4$ Hz, 1H), 2.31 (td, $J = 14.2, 3.3$ Hz, 1H), 2.08 – 2.02 (m, 1H), 1.91 (dt, $J = 13.9, 3.4$ Hz, 1H), 1.66 (s, 3H), 1.56 (s, 3H), 1.49 (dt, $J = 13.2, 3.5$ Hz, 1H), 1.39 (s, 3H), 1.04 (s, 3H), 1.02 (s, 3H).

$^{13}$C NMR (125 MHz, CD$_3$OD): δ 150.0, 141.9, 139.0, 135.4, 135.0, 133.0, 131.9, 128.6, 125.6, 123.5, 114.9, 111.4, 109.6, 108.7, 77.0, 45.7, 41.5, 36.7, 34.1, 29.0, 27.6, 27.4, 26.5, 23.0, 18.5.

IR (Neat film): 3343, 2966, 2360, 2342, 1559, 1457, 1380, 1317, 1059, 912, 748, 668.

HRMS (ESI): calcd for C$_{25}$H$_{30}$NO [M+H]$^+$: 360.2322, found: 360.2305.

$[\alpha]^{25.8}_{D} = +71.7^\circ$ (c = 0.024, MeOH).
C-12 gem-dimethyl analog 25. 25 was prepared by the same route starting from (5S)-2,2-dimethyl-5-isopropenylcyclohexanone.

In a 20 mL scintillation vial, 1.9 mg pentacycle 25 was dissolved in ca. 5 mL 1:1 EtOAc:hexanes. The solvent was removed via rotary evaporation, and a yellow oil/gel was obtained. The vial was capped and kept in a fridge for 14 h to allow crystals to grow. See X-ray crystallographic data below.

\( R_f = 0.28 \) (15:85 EtOAc:hexanes)
m.p. = 226-230 °C.

\(^1\)H NMR (500 MHz, CDCl\(_3\)): \( \delta 7.72 \) (s, 1H), 7.10 – 7.06 (m, 2H), 6.96 (dd, \( J = 5.9, 2.0 \) Hz, 1H), 3.70 (d, \( J = 7.5 \) Hz, 1H), 3.32 (dd, \( J = 11.0, 7.5 \) Hz, 1H), 2.84 (d, \( J = 14.6 \) Hz, 1H), 2.57 (dd, \( J = 14.6, 1.3 \) Hz, 1H), 2.23 – 2.17 (m, 1H), 1.82 (s, 3H), 1.80 – 1.77 (m, 1H), 1.57 – 1.49 (m, 2H), 1.52 (s, 3H), 1.38 (s, 3H), 1.37 – 1.34 (m, 1H), 1.21 (s, 3H), 1.12 (s, 3H), 0.99 (s, 3H).

\(^1^3\)C NMR (125 MHz, CDCl\(_3\)): \( \delta 211.0, 141.4, 137.6, 132.4, 127.0, 122.4, 113.5, 110.6, 107.5, 64.9, 55.2, 45.6, 37.7, 35.3, 33.2, 32.7, 32.2, 31.2, 29.9, 29.1, 28.3, 25.5, 25.0, 21.1.

HRMS (ESI): calcd for C\(_{24}\)H\(_{32}\)NO [M+H]+: 350.2478, found: 350.2478.

\( [\alpha]^{24^\circ}_D = -69.1^\circ \) (c = 0.288, CHCl\(_3\)).
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Experimental Spectra
Supporting Information

Current Data Parameters
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PROCNO  1

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S25
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PROCNO  1

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PLAG:  16.09099980 W
PLWL2:  0.19865000 W

F2 - Processing parameters
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WDW:  EM
SSB:  0
LB:  1.00 Hz
GB:  0
PC:  1.40
BRUKER

Current Data Parameters
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EXPN0: 1
PROCNO: 1

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DW: 19.800 usc
DE: 6.50 usc
TE: 297.2 K
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D11: 0.0300000 sec
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SP02: 400.1516006 MHz
NUC2: 1H
CPDPFG[2] [waltz16
PCPD2: 90.00 usc
PLW2: 16.09009960 W
PLW12: 0.19865000 W

F2 - Processing parameters
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NS 8
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P2 - Processing Parame
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LB 0 Hz
GB 0
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(-)-ambiguine P (5)
(CD$_3$OD, 125 MHz)
X-ray Crystallographic Data for 25

Crystal growth of C$_{24}$H$_{31}$NO: Jiasu Xu (prof. Viresh H. Rawal’s group).

Data collected and structure solved/refined: Andrew McNeece/Alexander S. Filatov, July/2018 (X-ray Laboratory, Searle B013, Department of Chemistry, the University of Chicago, Chicago, Il).

General information: The diffraction data were measured at 100 K on a Bruker D8 VENTURE with PHOTON 100 CMOS detector system equipped with a Mo-target X-ray tube (λ = 0.71073 Å). Data reduction and integration were performed with the Bruker APEX3 software package (Bruker AXS, version 2015.5-2, 2015). Data reduction and integration were performed with the Bruker APEX3 software package (Bruker AXS, version 2015.5-2, 2015). Data were scaled and corrected for absorption effects using the multi-scan procedure as implemented in SADABS (Bruker AXS, version 2014/5, Krause, Herbst-Irmer, Sheldrick & Stalke, J. Appl. Cryst. 2015, 48, 3-10). The structure was solved by SHELXT (Version 2014/5: Sheldrick, G. M. Acta Crystallogr. 2015, A71, 3-8) and refined by a full-matrix least-squares procedure using OLEX2 (O. V. Dolomanov, L. J. Bourhis, R. J. Gildea, J. A. K. Howard and H. Puschmann. J. Appl. Crystallogr. 2009, 42, 339-341) (XL refinement program version 2018/3, Sheldrick, G. M. Acta Crystallogr. 2015, C71, 3-8). Crystallographic data and details of the data collection and structure refinement are listed in Table 1.

Specific details for structure refinement: All atoms were refined with anisotropic thermal parameters. Hydrogen atoms were included in idealized positions for structure factor calculations except those of N-H groups which were located in the difference Fourier map and freely refined without any restraints. All structures are drawn with thermal ellipsoids at 50% probability.
Table 1 Crystal data and structure refinement for 0632.

| Property                        | Value                              |
|--------------------------------|------------------------------------|
| Identification code            | 0632                               |
| Empirical formula              | C_{24}H_{31}NO                      |
| Formula weight                 | 349.50                             |
| Temperature/K                  | 100(2)                             |
| Crystal system                 | monoclinic                         |
| Space group                    | P2_1                               |
| a/Å                            | 10.883(4)                          |
| b/Å                            | 14.071(6)                          |
| c/Å                            | 13.651(6)                          |
| α/°                            | 90                                 |
| β/°                            | 105.587(11)                        |
| γ/°                            | 90                                 |
| Volume/Å^3                     | 2013.7(14)                         |
| Z                              | 4                                  |
| ρ_{calc} g/cm^3                | 1.153                              |
| μ/mm^-1                        | 0.069                              |
| F(000)                         | 760.0                              |
| Crystal size/mm^3              | 0.387 × 0.251 × 0.114              |
| Radiation                      | MoKα (λ = 0.71073)                 |
| 2θ range for data collection/^o| 4.846 to 50.212                    |
| Index ranges                   | -12 ≤ h ≤ 12, -16 ≤ k ≤ 16, -16 ≤ l ≤ 16 |
| Reflections collected          | 31216                              |
| Independent reflections        | 6779 [R_{int} = 0.1216, R_{sigma} = 0.0863] |
| Data/restraints/parameters     | 6779/1/489                         |
| Goodness-of-fit on F^2         | 1.028                              |
| Final R indexes [I>=2σ (I)]    | R_1 = 0.0543, wR_2 = 0.1276        |
| Final R indexes [all data]     | R_1 = 0.1015, wR_2 = 0.1494        |
| Largest diff. peak/hole / e Å^-3| 0.18/-0.23                        |

R_{int} = \Sigma | F_o^2 - <F_o^2> | / \Sigma | F_o^2 |
R1 = \Sigma | | F_o | - | F_c || | F_o | / \Sigma | F_o |
wR2 = [\Sigma [w (F_o^2 - F_c^2)^2] / \Sigma [w (F_o^2]^2]]^{1/2}
Goodness-of-fit = [\Sigma [w (F_o^2 - F_c^2)^2] / (n-p)]^{1/2}

n: number of independent reflections; p: number of refined parameters
Two independent molecules in the crystal
The crystal is made of dimers based on N–H···O intermolecular hydrogen bonds
Table 2 Fractional Atomic Coordinates (×10⁴) and Equivalent Isotropic Displacement Parameters (Å²×10³) for 0632. \(U_{eq}\) is defined as 1/3 of the trace of the orthogonalised \(U_{ij}\) tensor.

| Atom | \(x\)     | \(y\)     | \(z\)     | \(U_{eq}\) |
|------|------------|------------|------------|------------|
| O1   | 6646(3)    | 3809(2)    | 10162(2)   | 45.4(8)    |
| N1   | 3326(4)    | 4510(3)    | 7456(3)    | 38.1(10)   |
| C1   | 2375(4)    | 4245(3)    | 7886(3)    | 36.6(11)   |
| C2   | 1205(4)    | 4649(3)    | 7887(4)    | 42.4(12)   |
| C3   | 538(5)     | 4229(3)    | 8487(4)    | 45.1(13)   |
| C4   | 986(4)     | 3422(4)    | 9088(4)    | 43.0(12)   |
| C5   | 2130(4)    | 3001(3)    | 9080(3)    | 36.2(11)   |
| C6   | 2802(4)    | 3418(3)    | 8449(3)    | 35.0(11)   |
| C7   | 4022(4)    | 3183(3)    | 8337(3)    | 34.1(11)   |
| C8   | 4703(4)    | 2338(3)    | 8919(3)    | 34.1(11)   |
| C9   | 4261(4)    | 2252(3)    | 9905(3)    | 36.5(11)   |
| C10  | 2768(4)    | 2128(3)    | 9680(3)    | 41.1(12)   |
| C11  | 2409(5)    | 2039(4)    | 10693(4)   | 53.2(14)   |
| C12  | 2299(5)    | 1238(4)    | 9029(4)    | 47.4(13)   |
| C13  | 5022(5)    | 1490(4)    | 10620(4)   | 44.5(12)   |
| C14  | 6453(4)    | 1667(4)    | 10854(3)   | 42.9(12)   |
| C15  | 6940(4)    | 1671(3)    | 9909(4)    | 41.0(12)   |
| C16  | 6774(5)    | 688(3)     | 9401(4)    | 50.7(13)   |
| C17  | 8365(4)    | 1912(4)    | 10188(4)   | 51.1(14)   |
| C18  | 6176(4)    | 2404(3)    | 9116(3)    | 35.0(11)   |
| C19  | 6532(4)    | 3437(3)    | 9330(3)    | 37.7(11)   |
| C20  | 6703(4)    | 4031(3)    | 8451(3)    | 41.8(12)   |
| C21  | 5566(4)    | 4016(3)    | 7455(3)    | 36.3(11)   |
| C22  | 4340(4)    | 3876(3)    | 7744(3)    | 35.9(11)   |
| C23  | 5705(4)    | 3236(4)    | 6711(3)    | 44.0(12)   |
| C24  | 5597(5)    | 4974(4)    | 6918(4)    | 47.1(13)   |
| O2   | 2663(3)    | 6184(3)    | 6261(3)    | 54.7(9)    |
| N2   | 2049(4)    | 9070(3)    | 7644(3)    | 40.1(10)   |
| C25  | 806(4)     | 8931(3)    | 7659(3)    | 36.5(11)   |
| C26  | 132(5)     | 9184(3)    | 8351(4)    | 45.7(12)   |
| C27  | -1132(5)   | 8916(4)    | 8110(4)    | 48.7(13)   |
| C28  | -1714(5)   | 8393(4)    | 7229(4)    | 48.7(13)   |
| C29  | -1057(4)   | 8139(3)    | 6544(4)    | 41.5(12)   |
| C30  | 213(4)     | 8440(3)    | 6766(3)    | 35.1(11)   |
Table 3 Anisotropic Displacement Parameters (Å²×10³) for 0632. The Anisotropic displacement factor exponent takes the form: -2π²[h²a²*U₁₁+2hka*b*U₁₂+...].

| Atom | U₁₁   | U₁₂   | U₁₃   | U₂₂   | U₂₃   | U₁₂   |
|------|-------|-------|-------|-------|-------|-------|
| O₁   | 54 (2) | 40 (2) | 33.1 (18) | -1.6 (16) | -2.9 (15) | -3.2 (16) |
| N₁   | 39 (2) | 34 (2) | 39 (2) | 6 (2) | 5 (2) | 3 (2) |
| C₁   | 37 (3) | 35 (3) | 34 (3) | 3 (2) | 3 (2) | 0 (2) |
| C₂   | 37 (3) | 35 (3) | 50 (3) | 1 (2) | 3 (2) | 3 (2) |
| C₃   | 36 (3) | 45 (3) | 52 (3) | -1 (3) | 7 (2) | 2 (2) |
| C₄   | 37 (3) | 43 (3) | 49 (3) | -3 (3) | 10 (2) | -1 (2) |
| C₅   | 37 (3) | 36 (3) | 33 (3) | -5 (2) | 4 (2) | -3 (2) |
| C₆   | 37 (3) | 34 (3) | 30 (2) | -4 (2) | 1 (2) | -2 (2) |
| C₇   | 37 (3) | 33 (3) | 28 (2) | -4 (2) | 1 (2) | 3 (2) |
| C₈   | 39 (3) | 26 (2) | 34 (2) | 2 (2) | 4 (2) | 2.1 (19) |
| C₉   | 42 (3) | 33 (3) | 32 (2) | 3 (2) | 5 (2) | 0 (2) |
| C₁₀  | 42 (3) | 35 (3) | 44 (3) | -2 (2) | 9 (2) | -4 (2) |
| C₁₁  | 57 (3) | 55 (3) | 52 (3) | 13 (3) | 21 (3) | 8 (3) |
| C₁₂  | 40 (3) | 39 (3) | 58 (3) | 1 (3) | 4 (2) | -7 (2) |
| C13 | 51 (3) | 42 (3) | 37 (3) | 7 (2) | 8 (2) | 2 (2) |
| C14 | 46 (3) | 40 (3) | 37 (3) | 9 (2) | 1 (2) | 5 (2) |
| C15 | 40 (3) | 37 (3) | 41 (3) | 0 (2) | 2 (2) | 2 (2) |
| C16 | 54 (3) | 40 (3) | 51 (3) | -3 (3) | 2 (3) | 7 (2) |
| C17 | 42 (3) | 52 (3) | 52 (3) | 7 (3) | 1 (2) | 7 (2) |
| C18 | 34 (2) | 35 (3) | 33 (2) | 0 (2) | 3 (2) | 4 (2) |
| C19 | 33 (2) | 39 (3) | 35 (3) | 3 (2) | 0 (2) | 3 (2) |
| C20 | 40 (3) | 41 (3) | 41 (3) | 5 (2) | 6 (2) | -5 (2) |
| C21 | 41 (3) | 39 (3) | 27 (2) | 3 (2) | 6 (2) | 3 (2) |
| C22 | 40 (3) | 34 (3) | 31 (2) | 4 (2) | 4 (2) | 3 (2) |
| C23 | 45 (3) | 36 (3) | 54 (3) | 2 (2) | 7 (2) | 7 (2) |
| C24 | 48 (3) | 49 (3) | 46 (3) | 7 (3) | 16 (3) | 3 (3) |
| O2  | 67 (2) | 41 (2) | 53 (2) | 13.3 (18) | 12.0 (18) | 9.8 (18) |
| N2  | 41 (2) | 46 (3) | 31 (2) | -5 (2) | 5 (2) | -2 (2) |
| C25 | 40 (3) | 35 (3) | 35 (2) | 5 (2) | 11 (2) | 5 (2) |
| C26 | 56 (3) | 39 (3) | 41 (3) | 3 (2) | 11 (3) | 3 (2) |
| C27 | 56 (3) | 43 (3) | 53 (3) | 2 (3) | 25 (3) | 8 (3) |
| C28 | 42 (3) | 50 (3) | 57 (3) | 5 (3) | 18 (3) | 4 (2) |
| C29 | 40 (3) | 37 (3) | 47 (3) | 5 (2) | 11 (2) | 4 (2) |
| C30 | 39 (3) | 31 (3) | 33 (2) | 2 (2) | 5 (2) | -1 (2) |
| C31 | 36 (2) | 26 (2) | 30 (2) | -1 (2) | 2 (2) | -2 (2) |
| C32 | 41 (3) | 35 (3) | 29 (2) | 1 (2) | 0 (2) | -4 (2) |
| C33 | 47 (3) | 40 (3) | 40 (3) | 0 (2) | 9 (2) | -8 (2) |
| C34 | 43 (3) | 48 (3) | 48 (3) | -7 (3) | 7 (2) | -5 (2) |
| C35 | 62 (4) | 73 (4) | 97 (5) | -23 (4) | 30 (4) | -30 (3) |
| C36 | 48 (3) | 76 (4) | 51 (3) | -5 (3) | -9 (3) | 10 (3) |
| C37 | 59 (3) | 51 (4) | 59 (3) | -13 (3) | 13 (3) | -20 (3) |
| C38 | 78 (4) | 43 (3) | 58 (3) | -15 (3) | 25 (3) | -18 (3) |
| C39 | 56 (3) | 41 (3) | 42 (3) | -14 (2) | 9 (3) | -11 (2) |
| C40 | 66 (4) | 61 (4) | 34 (3) | -10 (3) | 2 (3) | -14 (3) |
| C41 | 75 (4) | 47 (3) | 58 (3) | -14 (3) | 22 (3) | -3 (3) |
| C42 | 43 (3) | 35 (3) | 34 (3) | 1 (2) | 3 (2) | -1 (2) |
| C43 | 50 (3) | 32 (3) | 38 (3) | 0 (2) | 12 (2) | 6 (2) |
| C44 | 43 (3) | 47 (3) | 38 (3) | 8 (2) | 3 (2) | 8 (2) |
| C45 | 40 (3) | 43 (3) | 32 (2) | -1 (2) | 2 (2) | -3 (2) |
| C46 | 39 (3) | 35 (3) | 32 (2) | 2 (2) | 2 (2) | -1 (2) |
| C47 | 47 (3) | 48 (3) | 47 (3) | 4 (3) | 11 (2) | -6 (2) |
Table 4 Bond Lengths for 0632.

| Atom | Atom | Length/Å | Atom | Atom | Length/Å |
|------|------|----------|------|------|----------|
| O1   | C19  | 1.226(5) | O2   | C43  | 1.225(6) |
| N1   | C1   | 1.371(6) | N2   | C25  | 1.373(6) |
| N1   | C22  | 1.392(6) | N2   | C46  | 1.392(6) |
| C1   | C2   | 1.394(6) | C25  | C26  | 1.389(7) |
| C1   | C6   | 1.404(6) | C25  | C30  | 1.399(6) |
| C2   | C3   | 1.368(7) | C26  | C27  | 1.377(7) |
| C3   | C4   | 1.408(7) | C27  | C28  | 1.407(7) |
| C4   | C5   | 1.383(6) | C28  | C29  | 1.369(6) |
| C5   | C6   | 1.400(6) | C29  | C30  | 1.399(6) |
| C5   | C10  | 1.534(6) | C29  | C34  | 1.535(7) |
| C6   | C7   | 1.416(6) | C30  | C31  | 1.409(6) |
| C7   | C8   | 1.508(6) | C31  | C32  | 1.523(6) |
| C7   | C22  | 1.370(6) | C31  | C46  | 1.377(6) |
| C8   | C9   | 1.551(6) | C32  | C33  | 1.547(6) |
| C8   | C18  | 1.556(6) | C32  | C42  | 1.553(6) |
| C9   | C10  | 1.581(6) | C32  | C34  | 1.558(7) |
| C9   | C13  | 1.534(6) | C33  | C37  | 1.531(7) |
| C10  | C11  | 1.539(6) | C34  | C35  | 1.539(7) |
| C10  | C12  | 1.542(7) | C34  | C36  | 1.544(7) |
| C13  | C14  | 1.524(7) | C37  | C38  | 1.519(8) |
| C14  | C15  | 1.521(6) | C38  | C39  | 1.515(7) |
| C15  | C16  | 1.535(7) | C39  | C40  | 1.548(7) |
| C15  | C17  | 1.532(7) | C39  | C41  | 1.533(7) |
| C15  | C18  | 1.563(6) | C39  | C42  | 1.563(6) |
| C18  | C19  | 1.513(7) | C42  | C43  | 1.505(6) |
| C19  | C20  | 1.514(6) | C43  | C44  | 1.511(7) |
| C20  | C21  | 1.574(6) | C44  | C45  | 1.570(7) |
| C21  | C22  | 1.504(6) | C45  | C46  | 1.501(6) |
| C21  | C23  | 1.530(6) | C45  | C47  | 1.525(6) |
| C21  | C24  | 1.538(7) | C45  | C48  | 1.542(6) |
Table 5 Bond Angles for 0632.

| Atom  | Atom  | Atom  | Angle/°  | Atom  | Atom  | Atom  | Angle/°  |
|-------|-------|-------|----------|-------|-------|-------|----------|
| C1    | N1    | C22   | 109.7(4) | C25   | N2    | C46   | 109.7(4) |
| N1    | C1    | C2    | 133.2(4) | N2    | C25   | C26   | 132.6(4) |
| N1    | C1    | C6    | 106.4(4) | N2    | C25   | C30   | 106.4(4) |
| C2    | C1    | C6    | 120.3(4) | C26   | C25   | C30   | 121.0(4) |
| C3    | C2    | C1    | 117.2(5) | C27   | C26   | C25   | 116.3(5) |
| C2    | C3    | C4    | 122.8(5) | C26   | C27   | C28   | 122.6(4) |
| C5    | C4    | C3    | 120.8(4) | C29   | C28   | C27   | 121.5(5) |
| C4    | C5    | C6    | 116.5(4) | C28   | C29   | C30   | 116.1(5) |
| C4    | C5    | C10   | 128.2(4) | C28   | C29   | C34   | 128.8(4) |
| C6    | C5    | C10   | 115.4(4) | C30   | C29   | C34   | 115.1(4) |
| C1    | C6    | C7    | 108.5(4) | C25   | C30   | C31   | 108.8(4) |
| C5    | C6    | C1    | 122.3(4) | C29   | C30   | C25   | 122.4(4) |
| C5    | C6    | C7    | 128.9(4) | C29   | C30   | C31   | 128.7(4) |
| C6    | C7    | C8    | 118.1(4) | C30   | C31   | C32   | 118.2(4) |
| C22   | C7    | C6    | 106.9(4) | C46   | C31   | C30   | 106.9(4) |
| C22   | C7    | C8    | 134.8(4) | C46   | C31   | C32   | 134.8(4) |
| C7    | C8    | C9    | 107.2(4) | C31   | C32   | C33   | 106.7(3) |
| C7    | C8    | C18   | 112.5(4) | C31   | C32   | C42   | 112.2(3) |
| C9    | C8    | C18   | 113.6(4) | C33   | C32   | C42   | 115.2(4) |
| C8    | C9    | C10   | 112.5(4) | C32   | C33   | C34   | 112.8(4) |
| C13   | C9    | C8    | 111.5(4) | C37   | C33   | C32   | 110.2(4) |
| C13   | C9    | C10   | 113.4(4) | C37   | C33   | C34   | 114.2(4) |
| C5    | C10   | C9    | 108.0(4) | C29   | C34   | C33   | 109.6(4) |
| C5    | C10   | C11   | 111.0(4) | C29   | C34   | C35   | 109.5(4) |
| C5    | C10   | C12   | 108.1(4) | C29   | C34   | C36   | 107.5(4) |
| C11   | C10   | C9    | 109.3(4) | C35   | C34   | C33   | 109.4(4) |
| C11   | C10   | C12   | 109.1(4) | C35   | C34   | C36   | 108.8(5) |
| C12   | C10   | C9    | 111.4(4) | C36   | C34   | C33   | 112.1(4) |
| C14   | C13   | C9    | 111.5(4) | C38   | C37   | C33   | 111.4(4) |
| C15   | C14   | C13   | 113.1(4) | C39   | C38   | C37   | 113.0(4) |
| C14   | C15   | C16   | 110.8(4) | C38   | C39   | C40   | 110.1(4) |
| C14   | C15   | C17   | 110.5(4) | C38   | C39   | C41   | 110.0(5) |
| C14   | C15   | C18   | 110.4(4) | C38   | C39   | C42   | 110.8(4) |
| C16   | C15   | C18   | 107.8(4) | C40   | C39   | C42   | 107.8(4) |
| C17   | C15   | C16   | 107.4(4) | C41   | C39   | C40   | 107.7(4) |
Table 6 Hydrogen Atom Coordinates ($\AA\times10^4$) and Isotropic Displacement Parameters ($\AA^2\times10^3$) for 0632.

| Atom  | $x$      | $y$      | $z$      | U(eq)   |
|-------|----------|----------|----------|---------|
| H1    | 3300(50) | 5020(50) | 7140(40) | 68(19)  |
| H2    | 885.61   | 5193.05  | 7487.06  | 51      |
| H3    | -261.71  | 4491.48  | 8499.59  | 54      |
| H4    | 494.99   | 3165.16  | 9504.87  | 52      |
| H8    | 4407.48  | 1757.59  | 8499.61  | 41      |
| H9    | 4473.29  | 2871.66  | 10269.4  | 44      |
| H11A  | 2777.17  | 2571.73  | 11137.21 | 80      |
| H11B  | 2743.79  | 1439.29  | 11024.45 | 80      |
| H11C  | 1478.73  | 2047.98  | 10561.34 | 80      |
| H12A  | 1382.8   | 1154.28  | 8944.74  | 71      |
| H12B  | 2758.64  | 677.76   | 9368.97  | 71      |
| H12C  | 2459.55  | 1314.23  | 8360.9   | 71      |
| H13A  | 4764.83  | 1489.87  | 11262.36 | 53      |
| H13B  | 4822.17  | 857.04   | 10300.46 | 53      |
| H14A  | 6653.31  | 2286.66  | 11204.06 | 51      |
|   |       |       |       |       |
|---|-------|-------|-------|-------|
| H14B | 6908.24 | 1167.7 | 11324 | 51    |
| H16A | 5863.73 | 540.78 | 9149.74 | 76   |
| H16B | 7192.6  | 207.02 | 9897.64 | 76   |
| H16C | 7161.57 | 690.29 | 8830.07 | 76   |
| H17A | 8647.05 | 1965.1 | 9566.19 | 77   |
| H17B | 8848.09 | 1407.84 | 10619.45 | 77  |
| H17C | 8510.44 | 2516.97 | 10556.57 | 77  |
| H18  | 6363.21 | 2248.57 | 8455.76 | 42   |
| H20A | 7481.75 | 3810.21 | 8276.87 | 50   |
| H20B | 6851.42 | 4697.95 | 8684.24 | 50   |
| H23A | 6546.26 | 3284.86 | 6581.42 | 66   |
| H23B | 5039.97 | 3311.94 | 6070.26 | 66   |
| H23C | 4916.91 | 4988.09 | 6280.3  | 71   |
| H24A | 6425.18 | 5051.51 | 6772.3  | 71   |
| H24B | 5468.66 | 5491.8  | 7360.35 | 71   |
| H2A  | 2630(50) | 9240(40) | 8120(40) | 48(16) |
| H26  | 520.29 | 9522.96 | 8956.31 | 55   |
| H27  | -1629.02 | 9091.47 | 8556.1  | 58   |
| H28  | -2583.37 | 8212.57 | 7106.73 | 58   |
| H32  | 286.6 | 8211.93 | 4666.6  | 44   |
| H33  | 13.27 | 6637.28 | 5916.23 | 52   |
| H35A | -2088.11 | 6350.6 | 6190.96 | 113  |
| H35B | -2881.5 | 6509.79 | 5033.01 | 113  |
| H35C | -3184.25 | 7141.19 | 5915.07 | 113  |
| H36A | -2898.57 | 8574.93 | 4887.57 | 94   |
| H36B | -2498.72 | 7918.83 | 4066.23 | 94   |
| H36C | -1567.72 | 8749.15 | 4627.16 | 94   |
| H37A | -1384.88 | 5922.72 | 4489.51 | 69   |
| H37B | -1136.9 | 6764.65 | 3776.34 | 69   |
| H38A | 788.19 | 5473.92 | 4856.39 | 70   |
| H38B | 124.13 | 5416.26 | 3660.89 | 70   |
| H40A | 192.18 | 7461.32 | 3072.91 | 84   |
| H40B | 583.84 | 6581.54 | 2477.48 | 84   |
| H40C | 1576.34 | 7426.62 | 2876.27 | 84   |
| H41A | 3247.19 | 6367.4  | 3869.22 | 89   |
| H41B | 2332.47 | 5500.91 | 3384.84 | 89   |
|   |        |        |        |   |
|---|--------|--------|--------|---|
| H41C | 2911.69 | 5568.82 | 4591.28 | 89 |
| H42  | 2251.26 | 7798.11 | 4622.36 | 46 |
| H44A | 4455.42 | 7612.47 | 5905.35 | 53 |
| H44B | 4353.05 | 7302.79 | 7004.46 | 53 |
| H47A | 2875.81 | 9149.9  | 5101.29 | 71 |
| H47B | 4389.71 | 9250.33 | 5535.32 | 71 |
| H47C | 3464.57 | 9970.19 | 5901.95 | 71 |
| H48A | 4441.34 | 9650.15 | 7648.57 | 76 |
| H48B | 5424.62 | 8912.75 | 7383.36 | 76 |
| H48C | 4554.02 | 8595.37 | 8102.56 | 76 |