Supporting Information

For

I₂-Mediated Oxidative Bicyclization of 4-Pentenamines to Prolinol Carbamates with CO₂ Incorporating Oxyamination of C=C Bond

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General information
MeCN, DMF (N,N-dimethylformamide), DCM (dichloromethane) were dried by 4 Å molecular sieves. THF and Et₂O were purified by solvent purification system. Unless otherwise mentioned, reagents purchased were used without further purification. 1H NMR and 13C NMR spectra were recorded with 600 MHz, 400 MHz and 300 MHz spectrometer. CDCl₃ was selected as the solvent and residual proton resonance of CDCl₃ was referenced using the 7.26 ppm in 1H NMR and 77.16 ppm in 13C NMR. Coupling constants (J) were obtained in Hertz (Hz). The abbreviation of s, d, t and m means singlet, doublet, triplet, and multiplet, respectively. HRMS were recorded with ESI in positive ion mode on IT-TOF instrument. Pent-4-en-1-amine (1q) was prepared according reported literature.

Experiment procedure
Synthesis of substrate 1a, 1f, 1n

Representative procedure
100 mL Schlenk tube with NaH (22 mmol) was evacuated and back-filled with N₂ and 25 mL DMF was added. A solution of 2,2-diphenylacetonitrile (20mmol) in 10 mL DMF was injected via syringe. Mixture was stirred at room temperature for 1 h and turned yellow. Then Schlenk tube was cooled to 0 °C followed by addition of allyl bromide (22 mmol). The reaction was warmed to room temperature and stirred for 12 h, diluted with ice water and extracted with EtOAc for 3 times. Organic layer combined was washed with saturated NaCl solution, dried by Na₂SO₄, evaporated and isolated by silica gel flash chromatography (petroleum ether/ EtOAc).

100mL Schlenk tube containing LiAlH₄ (64 mmol) was evacuated and back-filled with
N₂ followed by addition of 30mL ether. The purified nitrile (16 mmol) in 10 mL ether was added to suspension at 0 °C. Then the reaction was stirred at 30 °C for 12 h followed by addition of 5M NaOH slowly in 0 °C to quench the reaction. After extraction with EtOAc for 3 times, combined organic layer was washed with saturated NaCl solution, dried by Na₂SO₄, evaporated and purified by silica gel flash chromatography (petroleum ether/ EtOAc).

2,2-Diphenylpent-4-en-1-amine(1a)³

¹H NMR (400 MHz, CDCl₃) δ 7.28-7.30 (m, 4H), 7.17-7.21 (m, 6H), 5.36-5.43 (m, 1H), 5.05 (d, J = 17.0 Hz, 1H), 4.97 (d, J = 10.2 Hz, 1H), 3.32 (s, 2H), 2.93 (d, 2H, J = 6.0 Hz), 1.01 (s, 2H); ¹³C NMR (101 MHz, CDCl₃) 146.4, 134.7, 128.4, 128.2, 126.1, 117.9, 51.5, 48.6, 41.3.

2-Phenylpent-4-en-1-amine(1j)³

¹H NMR (400 MHz, CDCl₃) δ 7.36-7.28 (m, 2H), 7.25-7.15 (m, 3H), 5.69 (ddt, J = 17.3, 10.1, 7.0 Hz, 1H), 4.99 (d, J = 17.0 Hz, 1H), 4.94 (d, J = 10.1 Hz, 1H), 2.96 (dd, J = 12.6, 5.2 Hz, 1H), 2.85 (dd, J = 12.7, 8.7 Hz, 1H), 2.75-2.62 (m, 1H), 2.47-2.30 (m, 2H), 1.08-0.95 (m, 1H).

¹³C NMR (76 MHz, CDCl₃) δ 143.2, 136.7, 128.6, 128.1, 126.6, 116.2, 49.5, 47.6, 38.5.

4-Methyl-2,2-diphenylpent-4-en-1-amine(1n)³

¹H NMR (400 MHz, CDCl₃) δ 7.29-7.24 (m, 4H), 7.22-7.14 (m, 6H), 4.84-4.79 (m, 1H), 4.59 (s, 1H), 3.41 (s, 2H), 2.92 (s, 2H), 1.07 (s, 3H), 0.74 (s, 1H).

¹³C NMR (76 MHz, CDCl₃) δ 147.0, 143.0, 128.5, 128.1, 126.1, 115.3, 51.4, 48.0, 44.1, 24.5.
Synthesis of 1b, 1h, 1i, 1k, 1p

Representative procedure²

100 mL Schlenk tube was evacuated and back-filled with N₂ with addition of iPr₂NH (20 mmol) in 20 mL THF. The reaction was cooled to -78 °C followed by addition of nBuLi (1.6 M, 20 mmol) and stirred for 15 min. Then solution of cyclopentanecarbonitrile (20 mmol) in 10 mL THF was injected to LDA solution with stirring at -78 °C for 30 minutes. After dropwise addition of Allyl bromide (22 mmol), the resulting solution was warmed to room temperature and stirred for 16 h. Then the mixture was treated with saturated solution of NH₄Cl and extracted with EtOAc for 3 times. The combined organic layer was washed with saturated NaCl solution, dried by Na₂SO₄, evaporated and purified by silica gel flash chromatography.

100mL Schlenk tube containing LiAlH₄ (60 mmol) was evacuated and back-filled with N₂ followed by addition of 30mL ether. Solution of purified nitrile (15 mmol) in 10 mL ether was added to suspension at 0 °C. Then the reaction was stirred at 30 °C for 12 h followed by addition of 5M NaOH at 0 °C to quench the reaction. After extraction with EtOAc for 3 times, the combined organic layer was washed with saturated NaCl solution, dried by Na₂SO₄, concentrated and purified by silica gel flash chromatography (petroleum ether/ EtOAc).

2,2-Dibenzylpent-4-en-1-amine(1b)
\[ ^1H \text{NMR (400 MHz, CDCl}_3 \] \( \delta \) 7.34-7.22 (m, 10H), 6.10-6.02 (m, 1H), 5.24-5.10 (m, 2H), 2.72 (dd, \( J = 5.5, 2.0 \text{ Hz, 4H} \)), 2.53 (d, \( J = 1.6 \text{ Hz, 2H} \)), 2.11-2.06 (m, 2H), 1.10 (s, 2H).

\[ ^{13}C \text{NMR (101 MHz, CDCl}_3 \] \( \delta \) 138.7, 135.2, 130.8, 128.2, 126.2, 118.1, 47.0, 42.7, 41.7, 38.7.

\( \text{(1- Allylcyclopentyl)methanamine (1h)} \)

\[ ^1H \text{NMR (400 MHz, CDCl}_3 \] \( \delta \) 5.84-5.71 (m, 1H), 5.10-4.97 (m, 2H), 2.47 (s, 2H), 2.07-2.01 (m, 2H), 1.53-1.59 (m, 4H), 1.33-1.39 (m, 4H), 1.09 (s, 2H).

\[ ^{13}C \text{NMR (101 MHz, CDCl}_3 \] \( \delta \) 135.6, 116.0, 49.2, 46.0, 40.9, 34.1, 24.9.

\( \text{(1- Allylcyclohexyl)methanamine (1i)} \)

\[ ^1H \text{NMR (400 MHz, CDCl}_3 \] \( \delta \) 5.87-5.74 (m, 1H), 5.11-4.98 (m, 2H), 2.58-2.48 (m, 2H), 2.12-2.05 (m, 2H), 1.48-1.25 (m, 11H), 0.97 (s, 2H).

\[ ^{13}C \text{NMR (101 MHz, CDCl}_3 \] \( \delta \) 135.1, 116.9, 48.9, 39.9, 37.1, 33.3, 26.5, 21.6.

\( \text{2-Methyl-2-phenylpent-4-en-1-amine (1k)} \)

\[ ^1H \text{NMR (400 MHz, CDCl}_3 \] \( \delta \) 7.36-7.28 (m, 4H), 7.21-7.17 (m, 1H), 5.61-5.48 (m, 1H), 5.03-4.91 (m, 2H), 2.95 (d, \( J = 13.1 \text{ Hz, 1H} \)), 2.72 (d, \( J = 13.1 \text{ Hz, 1H} \)), 2.50 (dd, \( J = 13.8, 6.4 \text{ Hz, 1H} \)), 2.27 (dd, \( J = 13.8, 8.1 \text{ Hz, 1H} \)), 1.29 (s, 3H), 1.01 (s, 2H).

\[ ^{13}C \text{NMR (101 MHz, CDCl}_3 \] \( \delta \) 145.3, 134.7, 128.3, 126.7, 125.9, 117.3, 53.4, 44.6, 43.0, 22.2.

\( \text{2,2-Diphenylhex-5-en-1-amine(1p)} \)
$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.30 (t, $J = 7.5$ Hz, 4H), 7.23-7.17 (m, 6H), 5.84-5.69 (m, 1H), 5.03-4.87 (m, 2H), 3.34 (s, 2H), 2.25-2.14 (m, 2H), 1.76 (dt, $J = 10.5$, 6.7 Hz, 2H), 0.80 (s, 2H).

$^{13}$C NMR (400 MHz, CDCl$_3$) 23.4, 34.2, 35.9, 49.1, 51.7, 114.6, 125.9, 128.0, 128.2, 138.6, 146.5

**Synthesis of S-1d, S-1e, S-1f**

![Chemical reaction diagram]

**Representative procedure**

250mL sealed tube containing Pd(OAc)$_2$ (0.5 mmol) and SPhos (1.0 mmol) was evacuated and back-filled with N$_2$ for 3 times. After injection of dry dioxane under N$_2$ flows, the resulting solution was stirred at room temperature for 30 min. The tube was added chloroacetonitrile (20 mmol), phenylboronic acid (30 mmol), Na$_2$CO$_3$ (45 mmol) and H$_2$O (6 mL) in 50 mL dioxane via syringe and sealed after addition. Then the reaction was stirred at 60 °C for 12 h and cooled to room temperature. The mixture was filtrated with a pad of silica gel using EtOAc. After concentration under reduced pressure, the crude product was purified by silica gel flash chromatography (petroleum ether/ EtOAc).

100mL sealed tube containing Pd(OAc)$_2$ (0.25 mmol), 2,2'-bis(dicyclohexylphosphino)-1,1'-biphenyl (DCPB) (0.5 mmol) was evacuated and back-filled with N$_2$ for 3 times, followed by addition of 10 mL dry dioxane and stirring for 30 min at room temperature. Then arylacetonitrile (5 mmol), aryl bromide (7.5 mmol), K$_3$PO$_4$ (15 mmol) in dry dioxane (20 mL) were added under N$_2$ flow and the reaction was conducted at 80 °C for 24 h. After cooling to room temperature, the mixture was filtrated with a pad of silica gel with EtOAc. The filtrate was concentrated
and purified by silica gel flash chromatography (petroleum ether/ EtOAc).

**Synthesis of substrate 1d, 1e, 1f**

Procedure follows the synthesis of 1b, 1h, 1i, 1k, 1p

\[ \text{Tol} \quad \text{NH}_2 \]

\[ \text{Tol} \quad \text{=CH} \]

**2,2-Di-p-tolylpent-4-en-1-amine (1d)**

$^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.12-7.04 (m, 8H), 5.46-5.39 (m, 1H), 5.06 (dd, $J$ = 17.1, 1.7 Hz, 1H), 5.01-4.95 (m, 1H), 3.29 (d, $J$ = 1.3 Hz, 2H), 2.90 (d, $J$ = 7.0 Hz, 2H), 2.33 (s, 6H), 0.85 (s, 2H).

$^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 143.4, 135.5, 135.0, 128.9, 128.1, 117.6, 50.8, 48.8, 41.3, 21.0.

\[ \text{F} \quad \text{NH}_2 \]

\[ \text{F} \quad \text{=CH} \]

**2,2-Bis(4-fluorophenyl)pent-4-en-1-amine (1e)**

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.11 (ddd, $J$ = 8.9, 5.4, 2.7 Hz, 4H), 6.96 (t, $J$ = 8.7 Hz, 4H), 5.35 (ddt, $J$ = 17.1, 10.1, 7.1 Hz, 1H), 5.02 (dd, $J$ = 17.1, 1.7 Hz, 1H), 4.97 (d, $J$ = 10.1 Hz, 1H), 3.26 (s, 2H), 2.86 (d, $J$ = 7.0 Hz, 2H), 0.77 (s, 2H).

$^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 161.24 (d, $J$ = 245.4 Hz), 141.87 (d, $J$ = 3.1 Hz), 134.1, 129.7 (d, $J$ = 7.7 Hz), 118.2, 115.0 (d, $J$ = 21.0 Hz), 50.6, 48.8, 41.4.

\[ \text{S} \quad \text{NH}_2 \]

**2,2-Di(thiophen-3-yl)pent-4-en-1-amine (1f)**

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.24-7.18 (m, 2H), 7.03-6.99 (m, 2H), 6.81 (d, $J$ = 5.0 Hz, 2H), 5.48 (ddt, $J$ = 17.1, 10.0, 7.0 Hz, 1H), 5.04 (d, $J$ = 17.1 Hz, 1H), 4.98 (d, $J$ = 10.1 Hz, 1H), 3.20 (s, 2H), 2.83 (d, $J$ = 6.9 Hz, 2H), 1.06 (s, 2H).
$^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 146.8, 134.5, 127.7, 125.4, 121.0, 117.9, 49.5, 48.5, 41.9.

**Representative Synthesis procedure of substrates 1l, 1m**

![Chemical structure](image)

100mL schlenk tube was evacuated and backfilled with N$_2$ 3 times. Allyl magnesium bromide (30 mmol) was injected and diluted with Et$_2$O. The reaction was added cyclopentene oxide (10 mmol) dropwise at room temperature over 15 min and refluxed for 3h. After cooling to room temperature, the solution was quenched with saturated NH$_4$Cl solution and extracted with EtOAc for 3 times. The combined organic layer was washed with brine, dried by Na$_2$SO$_4$, concentrated for next step without further purification.

Then 250 mL Schlenk tube with phthalimide (12.35 mmol) and PPh$_3$ (12.35 mmol) was evacuated and backfilled with N$_2$ for 3 times followed by addition of alcohol in THF (60 mL) and DEAD (40% solution in toluene, 13.3 mmol). The mixture was stirred at room temperature for 22 h. After removal of solvent in vaccum, the residue was triturated with PE/Et$_2$O (2:1) until complete precipitation of the solid. The solid was filtered off and washed with the same mixture of solvents. The combined organic layers were evaporated in vaccum and purified by silica gel flash chromatography (petroleum ether/ EtOAc).

To a solution of phthalimide (8 mmol) in MeOH (10 mL) was added a solution of hydrazine monohydrate (9.6 mmol) in methanol (10 mL). The resulting solution was refluxed for 3 h. Then the reaction was evaporated followed by addition of CH$_2$Cl$_2$ and white solid appeared. The suspension was filtrated and the separated organic layer was mixed with water and acidified (pH < 2) with 1 M HCl. After separation, the aqueous phase basified with solid KOH (pH > 10) and extracted with EtOAc for 3 times, dried
by Na₂SO₄, evaporated and product was obtained without further purification.

2- Allylcyclopentanamine (II)

¹H NMR (400 MHz, CDCl₃) δ 5.81 (ddt, J = 17.0, 10.1, 6.9 Hz, 1H), 5.09-5.00 (m, 1H), 4.98-4.91 (m, 1H), 3.32-3.24 (m, 1H), 2.18 (dt, J = 14.3, 7.1 Hz, 1H), 2.04 (dt, J = 14.3, 7.2 Hz, 1H), 1.86-1.65 (m, 4H), 1.52 (ddt, J = 9.5, 7.4, 4.5 Hz, 1H), 1.44-1.31 (m, 2H), 1.11 (s, 2H).

¹³C NMR (101 MHz, CDCl₃) δ 138.3, 115.0, 54.2, 44.4, 34.8, 34.1, 28.7, 21.9.

2- Allylcyclohexanamine (1m)

¹H NMR (400 MHz, CDCl₃) δ 5.80-5.67 (m, 1H), 5.02-4.91 (m, 2H), 2.99-2.91 (m, 1H), 2.08-2.00 (m, 1H), 1.98-1.89 (m, 1H), 1.61-1.46 (m, 5H), 1.39-1.17 (m, 6H).

¹³C NMR (101 MHz, CDCl₃) δ 137.7, 115.5, 49.5, 41.0, 36.3, 33.4, 26.4, 24.8, 21.0.

Synthesis procedure of substrate 1g

250 mL sealed tube was evacuated and back-filled with N₂ for 3 times. Dimethyl malonate (20 mmol) in 80mL DMSO was added to sealed tube under N₂ flows. Then K₂CO₃ was added and the reaction was stirred at room temperature for 10 minutes. After completion of basification, allyl bromide (24 mmol) in 20 mL DMSO was added dropwise. Stirred at room temperature for 14 h, the reaction was quenched with water, extracted with EtOAc for 3 times, dried by Na₂SO₄, evaporated and purified by silica gel flash chromatography (petroleum ether/ EtOAc).

100mL Schlenk tube containing NaH (9.9 mmol) was evacuated and back-filled with
N₂ for 3 times followed by addition of 30 mL THF. After addition of dimethyl 2-allylmalonate (9 mmol), the suspension was stirred for 30 min at 0 °C. Then the mixture was added N-chloromethylphthalimide (9 mmol) and stirred at room temperature for 16 h. After quenching by saturated NH₄Cl solution, the mixture was extracted with EtOAc for 3 times and combined organic layer was dried by Na₂SO₄ and evaporated under vacuum. Petroleum ether was added to afford unsolvable white solid, and crude product was obtained after filtration.

Crude dimethyl 2-allyl-2-((1,3-dioxoisoindolin-2-yl)methyl)malonate (8 mmol) was added to a solution of hydrazine monohydrate (9.6 mmol) in methanol (20 mL). And the resulting solution was refluxed for 3 h. Then the reaction was evaporated followed by addition of CH₂Cl₂ and eliminated white solid after filtration. The organic layer was acidified (pH < 2) with 1 M HCl. After separation, the aqueous phase was basified with solid KOH (pH > 10) and extracted with EtOAc for 3 times. The organic layers was combined, washed with saturated NaCl solution, dried by Na₂SO₄ and dimethyl 2-allyl-2-(aminomethyl)malonate was obtained after evaporation.

**Dimethyl 2-allyl-2-(aminomethyl)malonate (1g)**

\[ \text{MeO}_2\text{C} \quad \text{NH}_2 \quad \text{MeO}_2\text{C} \]

\(^1\text{H NMR (600 MHz, CDCl}_3\) \(\delta \) 5.62-5.53 (m, 1H), 5.05-4.96 (m, 2H), 3.63 (s, 6H), 2.99 (s, 2H), 2.56 (d, \(J = 7.4 \text{ Hz, 2H})\), 1.14 (s, 2H).

\(^1\text{C NMR (151 MHz, CDCl}_3\) \(\delta \) 170.9, 132.3, 119.0, 59.9, 52.3, 44.9, 36.5.

**Synthesis procedure of 1c**

250 mL Schlenk tube was evacuated and back-filled with N₂, iPrNH₂ (60 mml, 8.4mL) and THF (45mL) was added. The resulting solution was injected nBuLi (1.6M in hexane, 60 mmol) via syringe at -78°C and stirred for 30min. Then the LDA solution was stored at 0 °C. Another 250 mL Schlenk tube was evacuated and back-filled with N₂ followed by addition of MeCN (20 mmol) and THF (10 mL). The resulting solution cooled to -78 °C followed by addition of a third of prepared LDA solution (30.3 mL) and stirred for 15 min. Allyl bromide (20mmol) was added and the mixture was warmed
to room temperature with stirring for 30 min. The reaction was cooled to -78 °C again with addition of another equivalent LDA solution and stirred for 15 min. Then the reaction was added allyl bromide (20 mmol) again and warmed to room temperature with stirring. The reaction was cooled to -78 °C once more followed by addition remaining LDA solution with 15 min stirring. The mixture was added allyl bromide (20 mmol) and warmed to room with stirring for 12 h and quenched with water. After extraction with EtOAc for 3 times, combined organic layer was washed with brine and dried by Na$_2$SO$_4$ followed by removal of solvent under vacuum and purified by silica gel flash chromatography (petroleum ether/ EtOAc).

100mL Schlenk tube containing LiAlH$_4$ (20 mmol) was evacuated and back-filled with N$_2$ followed by addition of 15mL ether. Solution of purified nitrile (5 mmol) in 5 mL ether was added to suspension at 0 °C. Then the reaction was stirred at 30 °C for 12 h and slowly quenched by 5M NaOH at 0° C. After extraction with EtOAc for 3 times, combined organic layer was washed with saturated NaCl solution and dried by Na$_2$SO$_4$, evaporated and purified by silica gel flash chromatography (petroleum ether/ EtOAc).

![Structure of 2,2-Diallylpent-4-en-1-amine(1c)](image)

**2,2-Diallylpent-4-en-1-amine(1c)**

$^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 5.87-5.76 (m, 3H), 5.09-5.02 (m, 6H), 2.49 (d, $J = 1.4$ Hz, 2H), 2.04-1.98 (m, 6H), 1.06 (s, 2H).

$^{13}$C NMR (101 MHz, CDCl$_3$) $\delta$ 134.6, 117.7, 47.5, 40.7, 39.2.
**General procedure for oxyamination in MeCN**

25 mL Schlenk tube was evacuated and backfilled with CO₂ for 3 times. Amine (0.5 mmol) in MeCN (5 mL) was added under flow of CO₂, followed by addition of DBU (1.0 mmol). After stirring for 10 min, I₂ (0.5 mmol) was added and the reaction was conducted at room temperature for 16 h. Quenched with water, the reaction was extracted with EtOAc for 3 times and washed with saturated Na₂S₂O₃ solution and brine. The combined organic layer was dried by Na₂SO₄, evaporated and isolated by silica gel flash chromatography (petroleum ether/ EtOAc).

**General procedure for oxyamination in EtOH or EtOAc**

25 mL Schlenk tube was evacuated and backfilled with CO₂ for 3 times. Amine (0.5 mmol) in EtOH or EtOAc (5 mL) was added under flow of CO₂, followed by addition of DBU (1.0 mmol). After stirring for 10 min, I₂ (0.5 mmol) was added and the reaction was conducted at room temperature for 16 h. The reaction was monitored by NMR in CDCl₃ with CH₂Br₂ as internal standard.
Spectrum data

6,6-Diphenyltetrahydropyrrolo[1,2-c]oxazol-3(1H)-one (2a): White solid, yield: 84%, 117 mg, Rf = 0.30 (PE : EA = 3:1), mp: 179-180 °C. 1H NMR (400 MHz, CDCl3) δ 7.32-7.28 (m, J = 9.2, 5.0, 1.8 Hz, 4H), 7.24-7.17 (m, 6H), 4.48 (t, J = 8.6 Hz, 1H), 4.21 (d, J = 11.4 Hz, 1H), 4.16 (dd, J = 8.9, 4.8 Hz, 1H), 4.05 (ddt, J = 10.0, 8.6, 4.9 Hz, 1H), 3.91 (d, J = 11.4 Hz, 1H), 2.54 (dd, J = 11.8, 5.1 Hz, 1H), 2.37 (dd, J = 11.7, 10.3 Hz, 1H). 13C NMR (101 MHz, CDCl3) δ 145.7, 145.5, 128.7, 128.6, 127.0, 126.9, 126.8, 126.7, 68.0, 58.1, 57.8, 57.2, 43.7. IR (neat) νmax cm⁻¹ 1754; HRMS (ESI) calcd for C18H17NO2 [M+H]+ 280.1332, found: 280.1334.

6,6-Dibenzyltetrahydropyrrolo[1,2-c]oxazol-3(1H)-one (2b): Colorless oil, yield: 65%, 100 mg, Rf = 0.28 (PE:EA = 3:1). 1H NMR (600 MHz, CDCl3) δ 7.34-7.25 (m, 6H), 7.20 (d, J = 7.5 Hz, 2H), 7.14 (d, J = 7.5 Hz, 2H), 4.31 (t, J = 8.4 Hz, 1H), 3.98 (dd, J = 8.8, 4.3 Hz, 1H), 3.69 (d, J = 11.7 Hz, 1H), 3.51-3.45 (m, 1H), 3.21 (d, J = 11.7 Hz, 1H), 2.86-2.73 (m, 4H), 2.03 (dd, J = 12.7, 6.2 Hz, 1H), 1.54 (dd, J = 12.7, 9.8 Hz, 1H). 13C NMR (151 MHz, CDCl3) δ 137.7, 137.3, 130.7, 130.6, 128.6, 128.5, 126.9, 126.8, 68.5, 58.3, 54.0, 50.0, 46.1, 44.2, 41.1. IR (neat) νmax cm⁻¹ 1747; HRMS (ESI) calcd for C20H21NO2 [M+H]+ 308.1645, found: 308.1643.

6,6-Diallyltetrahydropyrrolo[1,2-c]oxazol-3(1H)-one (2c): White solid, yield: 83%, 86 mg, Rf = 0.29 (PE:EA = 3:1), mp: 29-31 °C. 1H NMR (600 MHz, CDCl3) δ 5.78-5.62 (m, 2H), 5.13-5.01 (m, 4H), 4.42 (t, J = 8.3 Hz, 1H), 4.08-3.99 (m, 2H), 3.45 (d, J = 11.8 Hz, 1H), 2.91 (d, J = 11.8 Hz, 1H), 2.17-2.10 (m, 4H), 1.88 (dd, J = 12.8, 6.5 Hz, 1H), 1.39 (dd, J = 12.8, 9.5 Hz, 1H). 13C NMR (151 MHz, CDCl3) δ 161.3, 133.6, 133.5, 119.0, 118.9, 68.1, 58.2, 55.2, 47.8, 43.0, 42.1, 41.5. IR (neat) νmax cm⁻¹ 1746;
6,6-Di-p-tolyltetrahydropyrrolo[1,2-c]oxazol-3(1H)-one (2d): White solid, yield: 88%, 135 mg, Rf = 0.28 (PE:EA=3:1). 1H NMR (400 MHz, CDCl$_3$) $\delta$ 7.12-7.07 (m, $J = 3.7$, 3.1 Hz, 8H), 4.45 (t, $J = 8.6$ Hz, 1H), 4.19-4.12 (m, 2H), 4.09-4.00 (m, 1H), 3.91 (d, $J = 11.4$ Hz, 1H), 2.53 (dd, $J = 11.8$, 5.2 Hz, 1H), 2.40-2.33 (m, 1H), 2.31 (d, $J = 1.8$ Hz, 6H). 13C NMR (101 MHz, CDCl$_3$) $\delta$ 142.8, 142.7, 136.3, 136.2, 129.2, 129.2, 126.7, 126.4, 68.0, 58.0, 57.3, 57.1, 43.6, 20.9, 20.8. IR (neat) $\nu_{\text{max}}$ cm$^{-1}$ 1755; HRMS (ESI) calcd for C$_{20}$H$_{21}$NO$_2$ [M+H]$^+$ 308.1645, found: 308.1644.

6,6-Bis(4-fluorophenyl)tetrahydropyrrolo[1,2-c]oxazol-3(1H)-one (2e): Light yellow oil, yield: 65%, 102 mg, Rf = 0.32 (PE:EA=3:1). 1H NMR (400 MHz, CDCl$_3$) $\delta$ 7.15 (dt, $J = 8.3$, 5.3 Hz, 4H), 6.99 (td, $J = 8.5$, 5.7 Hz, 4H), 4.52 (t, $J = 8.7$ Hz, 1H), 4.19 (dd, $J = 9.0$, 4.9 Hz, 1H), 4.14 (d, $J = 11.5$ Hz, 1H), 4.05 (tt, $J = 10.0$, 5.0 Hz, 1H), 3.86 (d, $J = 11.5$ Hz, 1H), 2.49 (dd, $J = 11.8$, 5.2 Hz, 1H), 2.40-2.33 (m, 1H). 13C NMR (101 MHz, CDCl$_3$) $\delta$, 161.8, 161.7 (d, $J = 248.5$ Hz), 161.6 (d, $J = 248.5$ Hz), 141.3 (d, $J = 2.9$ Hz), 141.1 (d, $J = 3.1$ Hz), 128.6 (d, $J = 7.9$ Hz), 128.3 (d, $J = 8.0$ Hz), 115.7 (d, $J = 14.1$ Hz), 115.5 (d, $J = 14.2$ Hz), 68.1, 58.1, 57.5, 57.0, 43.9. 19F NMR (565 MHz, CDCl$_3$) $\delta$ -115.18, -115.63. IR (neat) $\nu_{\text{max}}$ cm$^{-1}$ 1754; HRMS (ESI) calcd for C$_{18}$H$_{15}$F$_2$NO$_2$ [M+Na]$^+$ 338.0963, found: 338.0967.

6,6-Di(thiophen-3-yl)tetrahydropyrrolo[1,2-c]oxazol-3(1H)-one (2f): White solid, yield: 68%, 99 mg, Rf = 0.19 (PE:EA=3:1). 1H NMR (400 MHz, CDCl$_3$) $\delta$ 7.31-7.24 (m, 2H), $\delta$ 7.04 (dd, $J = 2.9$, 1.4 Hz, 1H), 7.01 (dd, $J = 2.9$, 1.4 Hz, 1H), 6.91-6.83 (m, 2H), 4.49 (t, $J = 8.1$ Hz, 1H), 4.19-4.02 (m, 3H), 3.91 (d, $J = 11.6$ Hz, 1H), 2.62 (dd, $J
= 12.3, 5.6 Hz, 1H), 2.31 (dd, \( J = 12.3, 8.9 \) Hz, 1H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \( \delta \) 161.56, 146.34, 145.84, 127.11, 126.82, 126.75, 126.60, 120.51, 120.41, 68.06, 58.48, 58.22, 52.71, 44.83. IR (neat) \( \nu_{\text{max}} \) cm\(^{-1}\) 1748; HRMS (ESI) calcd for C\(_{14}\)H\(_{13}\)NO\(_2\)S\(_2\) [M+H]\(^{+}\) 292.0460, found: 292.0463.

Dimethyl 3-oxotetrahydropyrrolo[1,2-c]oxazole-6,6(1H)-dicarboxylate (2g): Light yellow oil, yield: 47 %, 57 mg, \( R_f = 0.33 \) (PE:EA=1:1). \(^{1}\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) 4.53-4.48 (m, 1H), 4.26-4.20 (m, 2H), 4.14-4.06 (m, 1H), 3.77 (s, 3H), 3.75 (s, 3H), 3.66 (d, \( J = 12.8 \) Hz, 1H), 2.59 (dd, \( J = 13.4, 6.7 \) Hz, 1H), 2.21 (dd, \( J = 13.4, 8.9 \) Hz, 1H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \( \delta \) 170.4, 170.3, 161.0, 67.6, 60.8, 58.5, 53.6, 53.5, 52.5, 38.9. IR (neat) \( \nu_{\text{max}} \) cm\(^{-1}\) 1728; HRMS (ESI) calcd for C\(_{10}\)H\(_{13}\)NO\(_2\) [M+H]\(^{+}\) 244.0816, found: 244.0817.

Dihydro-1'H-spirocyclopentane-1,6'-pyrrolo[1,2-c]oxazol]-3'(5'H)-one (2h): Colorless oil, yield: 81%, 73 mg, \( R_f = 0.23 \) (PE:EA=3:1). \(^{1}\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) 4.51-4.44 (m, 1H), 4.11-4.01 (m, 2H), 3.45 (dd, \( J = 11.2, 3.5 \) Hz, 1H), 2.99 (dd, \( J = 11.2, 3.7 \) Hz, 1H), 1.94-1.87 (m, 1H), 1.67-1.47 (m, 9H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \( \delta \) 161.7, 68.4, 58.9, 57.9, 52.5, 43.9, 38.4, 38.3, 24.3, 24.2. IR (neat) \( \nu_{\text{max}} \) cm\(^{-1}\) 1744; HRMS (ESI) calcd for C\(_{10}\)H\(_{13}\)NO\(_2\) [M+H]\(^{+}\) 182.1176 found: 182.1178.

Dihydro-1'H-spirocyclohexane-1,6'-pyrrolo[1,2-c]oxazol]-3'(5'H)-one (2i): Light yellow oil, yield: 70%, 68 mg, \( R_f = 0.30 \) (PE:EA=3:1). \(^{1}\)H NMR (400 MHz, CDCl\(_3\)) \( \delta \) 4.47 (t, \( J = 8.2 \) Hz, 1H), 4.10-4.06 (m, 1H), 4.05-4.00 (m, 1H), 3.44 (d, \( J = 11.5 \) Hz, 1H), 2.94 (d, \( J = 11.5 \) Hz, 1H), 1.92 (dd, \( J = 12.5, 6.3 \) Hz, 1H), 1.52-1.32 (m, 11H). \(^{13}\)C NMR (101 MHz, CDCl\(_3\)) \( \delta \) 161.7, 68.4, 58.1, 57.1, 52.7, 45.6, 44.0, 38.1, 36.4, 25.6, 24.0, 23.3. IR (neat) \( \nu_{\text{max}} \) cm\(^{-1}\) 1755; HRMS (ESI) calcd for C\(_{11}\)H\(_{17}\)NO\(_2\) [M+H]\(^{+}\) 196.1332.
6-Phenyltetrahydro[1,2-c]oxazol-3(1H)-one (2j): Yellow oil, yield: 46%, 47 mg, R_f = 0.45 (PE:EA=1:1). 1H NMR (400 MHz, CDCl_3) δ 7.32 (t, J = 7.6 Hz, 2H), 7.24 (dd, J = 10.3, 7.3 Hz, 3H), 4.54 (t, J = 8.5 Hz, 1H), 4.27 (dd, J = 9.0, 3.2 Hz, 1H), 4.13-4.07 (m, 1H), 3.72-3.62 (m, 2H), 3.61-3.57 (m, 1H), 2.39 (dt, J = 10.8, 5.2 Hz, 1H), 1.70 (q, J = 11.2 Hz, 1H). 13C NMR (101 MHz, CDCl_3) δ 141.0, 128.9, 127.2, 127.1, 67.4, 60.3, 52.9, 46.6, 40.1. IR (neat) ν_max cm⁻¹ 1750; HRMS (ESI) calcd for C_{12}H_{13}NO_2 [M+H]^+ 204.1019, found: 204.1020.

6-Methyl-6-phenyltetrahydro[1,2-c]oxazol-3(1H)-one 4-63 (2k): Light yellow oil, yield: 69%, mg, R_f = 0.35 (PE:EA=3:1), major:minor=5:4. 1H NMR (600 MHz, CDCl_3) major product: δ 7.36-7.30 (m, 2H), 7.27 (d, J = 7.2 Hz, 1H), 7.25-7.20 (t, J = 8.2 Hz, 2H), 4.58-4.54 (m, 1H), 3.29 (ddd, J = 12.7, 10.5, 4.7 Hz, 1H), 4.21-4.12 (m, 1H), 3.93 (d, J = 11.0 Hz, 1H), 3.40 (d, J = 11.0 Hz, 1H), 2.23 (dd, J = 11.9, 5.3 Hz, 1H), 1.95 (t, J = 11.3 Hz, 1H), 1.46 (s, 3H); minor product: δ 7.36-7.30 (m, 2H), 7.27 (d, J = 7.2 Hz, 1H), 7.25-7.20 (t, J = 8.2 Hz, 2H), 4.21-4.12 (m, 2H), 3.78 (d, J = 11.6 Hz, 1H), 3.55 (d, J = 11.6 Hz, 1H), 2.45 (dd, J = 12.9, 7.2 Hz, 1H), 1.86 (dd, J = 12.8, 7.0 Hz, 1H), 1.44 (s, 3H). 13C NMR (151 MHz, CDCl_3) major product: δ 161.6, 128.7, 126.6, 125.6, 68.11, 59.3, 58.7, 49.4, 44.8, 29.8. minor product: δ 162.06, 146.70, 128.8, 126.7, 125.8, 69.5, 58.6, 58.2, 49.1, 45.7, 29.5. IR (neat) ν_max cm⁻¹ 1745.

Hexahydro-1H-cyclopenta[4,5]pyrrolo[1,2-c]oxazol-3(4aH)-one (2l):
**Compound 2l-1**, colorless oil, yield: 50 %, 42 mg, R_f=0.62(PE:EA=1:1). ^1^H NMR (400 MHz, CDCl_3) δ 4.49-4.42 (m, 1H), 4.33 (td, J = 7.4, 3.7 Hz, 1H), 4.09 (dd, J = 8.9, 3.0 Hz, 1H), 3.958-3.91 (m, 1H), 2.83-2.75 (m, 1H), 1.98-1.93 (m, 1H), 1.90-1.81 (m, 2H), 1.68-1.57 (m, 3H), 1.57-1.46 (m, 1H), 1.38-1.30 (m, 1H). ^1^C NMR (101 MHz, CDCl_3) δ 161.0, 67.5, 63.8, 58.6, 44.1, 38.5, 34.4, 32.7, 26.4. IR (neat) ν_{max} cm^{-1} 1744; HRMS (ESI) calcd for C₉H₁₃NO₂ [M+H]^+ 168.1019, found: 168.1018.

**Compound 2l-2**, colorless oil, yield: 25 %, 21 mg, R_f=0.52(PE:EA=1:1). ^1^H NMR (400 MHz, CDCl_3) δ 4.43-4.37 (m, 1H), 4.18-4.09 (m, 1H), 3.98 (t, J = 7.9 Hz, 1H), 3.91 (t, J = 7.2 Hz, 1H), 3.02-2.94 (m, 1H), 2.53 (dt, J = 7.1, 3.0 Hz, 1H), 2.21-2.15 (m, 1H), 1.68-1.53 (m, 5H), 1.23-1.14 (m, 1H). ^1^C NMR (101 MHz, CDCl_3) δ 155.8, 68.3, 61.6, 60.6, 47.7, 38.0, 31.7, 28.3, 24.4. IR (neat) ν_{max} cm^{-1} 1744; HRMS (ESI) calcd for C₉H₁₃NO₂ [M+H]^+ 168.1019, found: 168.1016.

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Octahydrooxazolo[3,4-a]indol-3(1H)-one (2m):

**Compound 2m-1**, Coloreless oil, yield: 38 %, 34 mg, R_f=0.52(PE:EA=1:1). ^1^H NMR (600 MHz, CDCl_3) δ 4.58 (t, J = 8.6 Hz, 1H), 4.29-4.24 (m, 1H), 4.04 (dd, J = 8.6, 6.2 Hz, 1H), 3.85 (dt, J = 8.7, 5.9 Hz, 1H), 2.34 (dt, J = 13.9, 7.0 Hz, 1H), 2.04 -1.99(m, 1H), 1.81-1.75 (m, 1H), 1.71-1.68 (m, 2H), 1.63-1.57 (m, 2H), 1.54-1.50 (m, 1H), 1.43 (m, 2H), 1.35-1.31 (m, 1H). ^1^C NMR (101 MHz, CDCl_3) δ 162.1, 70.3, 59.1, 56.7, 38.5, 35.5, 27.6, 26.3, 22.7, 21.1. IR (neat) ν_{max} cm^{-1} 1744; HRMS (ESI) calcd for C₁₀H₁₅NO₂ [M+H]^+ 182.1176, found: 182.1177.

**Compound 2m-2**, Coloreless oil, yield: 26 %, 24 mg, R_f=0.45(PE:EA=1:1). ^1^H NMR (600 MHz, CDCl_3) δ 4.49 (t, J = 8.3 Hz, 1H), 4.26-4.19 (m, 1H), 4.00 (t, J = 9.0 Hz, 1H), 3.57 (dt, J = 9.6, 6.3 Hz, 1H), 2.53 (dt, J = 11.3, 5.8 Hz, 1H), 2.48-2.42 (m, 1H), 1.86 (dt, J = 11.8, 5.9 Hz, 1H), 1.72-1.65 (m, 4H), 1.50-1.46 (m, 1H), 1.36-1.19 (m, 3H). ^1^C NMR (101 MHz, CDCl_3) δ 157.7, 71.1, 59.7, 55.1, 41.5, 33.4, 27.4, 26.7, 23.1, 21.6. IR (neat) ν_{max} cm^{-1} 1742; HRMS (ESI) calcd for C₁₀H₁₅NO₂ [M+H]^+ 182.1176,
found: 182.1178.

7a-Methyl-6,6-diphenyltetrahydropyrrolo[1,2-c]oxazol-3(1H)-one (2n): White solid, yield: 40%, 59 mg, Rf=0.39 (PE:EA=3:1), mp: 176-177 °C. 1H NMR (400 MHz, CDCl3) δ 7.34-7.26 (m, 6H), 7.21-7.13 (m, 4H), 4.80 (dd, J = 12.7, 1.1 Hz, 1H), 3.98 (d, J = 8.5 Hz, 1H), 3.71 (d, J = 8.4 Hz, 1H), 3.57 (d, J = 12.7 Hz, 1H), 2.88 (dd, J = 13.7, 1.1 Hz, 1H), 2.66 (d, J = 13.7 Hz, 1H), 1.38 (s, 3H). 13C NMR (101 MHz, CDCl3) δ 161.1, 146.1, 144.3, 129.0, 128.8, 126.9, 126.8, 126.7, 76.0, 64.8, 58.6, 55.1, 49.9, 27.9. IR (neat) νmax cm⁻¹ 1746; HRMS (ESI) calcd for C19H19NO2 [M+H]+ 294.1489, found: 294.1486.

6,6-Diphenyltetrahydro-1H-oxazolo[3,4-a]pyridin-3(5H)-one: White solid, yield: 24%, 35 mg, Rf=0.32 (PE:EA=3:1), mp:115-117 °C. 1H NMR (400 MHz, CDCl3) δ 7.31 (d, J = 4.3 Hz, 4H), 7.28-7.24 (m, 2H), 7.21-7.16 (m, 4H), 4.72 (dd, J = 13.9, 2.5 Hz, 1H), 4.45 (t, J = 7.7 Hz, 1H), 3.85-3.70 (m, 2H), 2.99 (d, J = 13.9 Hz, 1H), 2.73 (dq, J = 13.6, 2.9 Hz, 1H), 2.33 (td, J = 13.4, 3.0 Hz, 1H), 1.85 (dq, J = 13.0, 3.4 Hz, 1H), 1.25-1.21 (m, 1H). 13C NMR (101 MHz, CDCl3) δ 156.6, 146.7, 143.5, 128.9, 128.7, 127.7, 126.8, 126.5, 126.4, 68.6, 54.2, 49.3, 45.9, 33.9, 26.8. IR (neat) νmax cm⁻¹ 1744; HRMS (ESI) calcd for C19H19NO2 [M+H]^+ 294.1489, found: 294.1487.
Mechanistic studies

25 mL Schlenk tube with 2,2-diphenylpent-4-en-1-amine (0.1 mmol) was evacuated and backfilled with CO₂. CD₃CN (1mL) was added under flow of CO₂ followed by addition of DBU (1 equiv.). The resulting solution was stirred for 1 h.

\[ \text{HN} - \text{O} - [\text{DBUH}]^+ \]

¹H NMR (400 MHz, Acetonitrile-\(\text{d}_3\)) δ 12.30 (s, 1H), 7.34-7.28 (m, 4H), 7.26-7.20 (m, 6H), 5.62-5.52 (m, 1H), 5.07-5.01 (m, 1H), 4.92 (dd, \(J = 10.2, 2.3 \text{ Hz}\), 1H), 4.49 (s, 1H), 3.85 (d, \(J = 5.9 \text{ Hz}\), 2H), 3.50-3.43 (m, 2H), 3.40 (t, \(J = 5.9 \text{ Hz}\), 2H), 3.22 (t, \(J = 5.7 \text{ Hz}\), 2H), 2.98 (d, \(J = 7.1 \text{ Hz}\), 2H), 2.77-2.69 (m, 2H), 1.95-1.86 (m, 2H), 1.76-1.68 (m, 2H), 1.68-1.57 (m, 4H). ¹³C NMR (101 MHz, Acetonitrile-\(\text{d}_3\)) δ166.5, 162.7, 147.8, 136.1, 129.0, 128.8, 126.8, 118.1, 54.5, 51.2, 49.1, 48.9, 42.1, 38.9, 32.4, 29.7, 27.4, 24.9, 20.4.

calcd for C₉H₁₇N₂⁺ = 153.1386
Radical scavenger experiment

25 mL Schlenk tube with 2,2-diphenylpent-4-en-1-amine (0.5 mmol) was evacuated and backfilled with CO$_2$. CH$_3$CN 5mL was added under flow of CO$_2$, followed by addition of DBU (2 equiv.). The solution was stirred for 10 min with addition of TEMPO and I$_2$ (1 equiv.).
Extension of prolinol carbamates synthesis

Synthesis of 2a in gram scale

The 250 mL Schlenk tube with 1a (5 mmol) was evacuated and backfilled with CO\textsubscript{2} for 3 times followed by addition of MeCN (50 mL). The DBU (5 mmol) was injected via syringe and the resulting solution was stirred for 30 min. Then I\textsubscript{2} was added with stirring for 24 h at room temperature. The reaction was quenched with water, extracted with EtOAc and washed with saturated Na\textsubscript{2}S\textsubscript{2}O\textsubscript{3} and brine. Combined organic layer was dried by Na\textsubscript{2}SO\textsubscript{4}, evaporated and isolated by silica gel flash chromatography (petroleum ether/ EtOAc).

Synthesis of 3

The 25 mL Schlenk tube with 2a (0.4 mmol) was evacuated and back-filled with N\textsubscript{2} followed by addition THF 4 mL. The allyl magnesium bromide (1 M in ether, 0.4 mmol) was injected via syringe at -78 °C. With stirring for 4 h, the reaction was warmed to room temperature and quenched with saturated NH\textsubscript{4}Cl solution and was extracted with EtOAc (3×5 mL), dried by Na\textsubscript{2}SO\textsubscript{4}, evaporated and purified by silica gel flash chromatography (petroleum ether/ EtOAc).

\[ \text{ Piperidin-1-ylbut-3-en-1-one (3) } \]

\[ \text{1H NMR (400 MHz, CDCl\textsubscript{3}) } \delta 7.32-7.25 \text{ (m, 4H)}, 7.19 \text{ (q, } J = 7.3, 5.9 \text{ Hz, 6H}), 5.93 \text{ (ddt, } J = 16.9, 10.3, 6.6 \text{ Hz, 1H}), 5.37-5.32 \text{ (m, 1H)}, 5.23-5.12 \text{ (m, 2H)}, 4.46-4.37 \text{ (m, 1H)}, 4.15-4.05 \text{ (m, 1H)}, 3.88 \text{ (d, } J = 10.9 \text{ Hz, 1H}), 3.76-3.69 \text{ (m, 1H)}, 3.62 \text{ (dd, } J = 10.7, 7.4 \text{ Hz, 1H}), 3.20 \text{ (d, } J = 6.6 \text{ Hz, 2H}), 2.86-2.78 \text{ (m, 1H)}, 2.21 \text{ (dd, } J = 12.7, 10.6 \text{ Hz, 1H}). \]

\[ \text{13C NMR (101 MHz, CDCl\textsubscript{3}) } \delta 171.8, 145.0, 144.2, 130.6, 128.9, 128.8, 127.0, 127.0, 126.6, 126.4, 118.8, 66.9, 61.7, 58.2, 52.5, 40.6, 40.2. \]
Synthesis of 4

25 mL Schlenk tube containing LiAlH₄ (1.6 mmol) was evacuated and back-filled with N₂ followed by addition of THF 4 mL. 2a (0.4 mmol) in THF (4 mL) was added to the suspension at 0 °C. Then the reaction was warmed to 30°C with stirring for 12 h. The mixture was cooled to 0 °C and quenched by 5M NaOH slowly. After extraction with EtOAc for 3 times, combined organic layer was washed with saturated NaCl solution and dried by Na₂SO₄, evaporated and generate pure product without further purification.

![Chemical Structure](image)

(1-methyl-4,4-diphenylpyrrolidin-2-yl)methanol (4)

¹H NMR (400 MHz, CDCl₃) δ 7.35-7.31 (m, 2H), 7.23 (q, J = 7.7, 7.2 Hz, 6H), 7.17-7.07 (m, 2H), 3.90 (d, J = 9.9 Hz, 1H), 3.57 (dd, J = 11.1, 3.7 Hz, 1H), 3.37 (dd, J = 11.0, 2.3 Hz, 1H), 2.99 (d, J = 9.9 Hz, 1H), 2.79 (dd, J = 12.4, 8.6 Hz, 1H), 2.68 (ddt, J = 8.7, 6.4, 3.0 Hz, 1H), 2.59 (dd, J = 12.4, 6.6 Hz, 2H), 2.38 (s, 3H).¹³C NMR (101 MHz, CDCl₃) δ 148.7, 148.3, 128.5, 128.4, 127.2, 127.1, 126.1, 125.9, 68.3, 66.8, 61.6, 53.1, 41.8, 41.0.
Reference


$^1$H NMR, $^{13}$C NMR and $^{19}$F NMR Spectra

$^1$H NMR and $^{13}$C NMR for compound 2a
$^1$H NMR and $^{13}$C NMR for compound 2b
$^1$H NMR and $^{13}$C NMR for compound 2c
$^1$H NMR and $^{13}$C NMR for compound 2d
$^{1}$H NMR, $^{13}$C NMR and $^{19}$F NMR for compound 2e
$^1$H NMR and $^{13}$C NMR for compound 2f
$^{1} \text{H NMR and } ^{13} \text{C NMR for compound } 2g$
$^{1}H$ NMR and $^{13}C$ NMR for compound 2h
$^1$H NMR and $^{13}$C NMR for compound 2i
$^1$H NMR and $^{13}$C NMR for compound 2j
The NOE spectrum of 2j with irradiation at δ 4.08
$^1$H NMR and $^{13}$C NMR for compound 2k
$^1$H NMR and $^{13}$C NMR for compound 2l-1
$^1$H NMR and $^{13}$C NMR for compound 2l-2
$^1$H NMR and $^{13}$C NMR for compound 2m-1
$^1$H NMR and $^{13}$C NMR for compound 2m-2
$^1$H NMR and $^{13}$C NMR for compound 2n
\(^1\)H NMR and \(^13\)C NMR for compound 2p
$^1$H NMR and $^{13}$C NMR for compound 3
$^1$H NMR and $^{13}$C NMR for compound 4
Crystal data and structure refinement for 2a.

Identification code 2a
Empirical formula C_{18}H_{17}NO_{2}
Formula weight 279.33
Temperature/K 173.15
Crystal system triclinic
Space group P-1
a/Å 6.3952(13)
b/Å 9.4580(19)
c/Å 12.085(2)
α/° 102.98(3)
β/° 94.15(3)
γ/° 90.64(3)
Volume/Å³ 710.1(2)
Z 2
ρ_{calc}g/cm³ 1.306
μ/mm⁻¹ 0.085
F(000) 296
Crystal size/mm³ 0.415 × 0.332 × 0.197
Radiation MoKα (λ = 0.710747)
Theta range for data collection/° 3.46 to 54.98
Index ranges -8 ≤ h ≤ 8, -12 ≤ k ≤ 12, -15 ≤ l ≤ 15
Reflections collected 9148
Independent reflections 3206 [R_{int} = 0.0267, R_{sigma} = 0.0214]
Data/restraints/parameters 3206/0/190
Goodness-of-fit on F² 1.065
Final R indexes [I>=2σ (I)] R₁ = 0.0410, wR₂ = 0.1055
Final R indexes [all data] R₁ = 0.0427, wR₂ = 0.1070
Largest diff. peak/hole / e Å$^{-3}$  0.29/-C14:D330.17