

Supporting Information for

Expedient access to α -(hetero)aryl- α -keto-1,3-diamines via redox-neutral photocatalyzed reactions of *N*-vinylimides with α -aminoalkyl radicals

Yu Zhao, Yutao Jing, Yan Li, Li Qiu and Yewen Fang

Table of Contents

1. General Information	S3
1.1 Solvents, Reagents, and Starting Materials	S3
1.2 Instruments	S3
1.3 Picture of a Typical Reaction Setup	S3
2. Preparation of <i>N</i> -vinylimides.....	S3
2.1 Know substrates reported in our previous work	S3
2.2 Synthesis of <i>N</i> -vinylimide	S4
3. General procedure of photoredox-catalyzed acyl migration reactions	S8
4. Mechanism study	S24
5. Procedure of Suzuki reaction.....	S25
6. References	S26
7. X-ray crystal data for compounds 3q and 4j.....	S27
8. NMR Spectra of New Compounds	S29

1. General Information

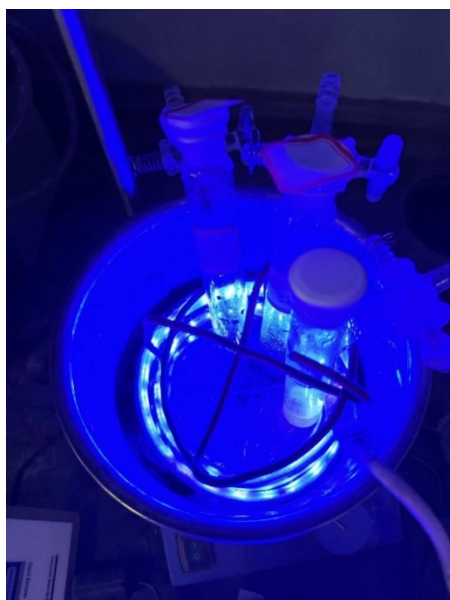
1.1 Solvents, Reagents, and Starting Materials

All reactions were carried out in glassware under inert (nitrogen) atmosphere unless otherwise noted. DMF and CH₂Cl₂ were dried from CaH. The dehydrated solvents DMSO, DMA and acetonitrile were purchased from Energy Chemical Chemicals. *N*-Vinylimides were prepared according to literature procedures and our previous report.¹ All known tertiary amines and α -silylamines are commercially available or prepared *via* reported procedures.² Photocatalysts and all other chemicals were purchased from local vendors and used as supplied unless otherwise stated.

1.2 Instruments

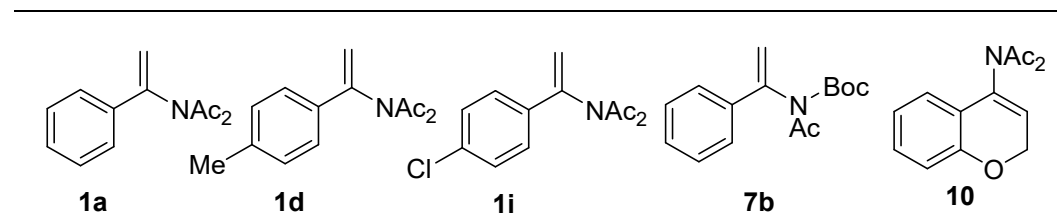
NMR spectra were recorded on a Bruker Avance 500 spectrometer (500 MHz). Chemical shifts were reported in ppm downfield from tetramethylsilane, and calibrated using residue undeuterated solvent (CHCl₃ at 7.26 ppm ¹H NMR, 77.0 ppm ¹³C NMR). Spectra were reported as follows: chemical shift (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz) and integration. High resolution mass spectra (HRMS) were recorded on an ESI-Q-TOF spectrometer Agilent 6210 ESI/TOF. Single Crystal X-ray Diffraction (SC-XRD) recorded on a Bruker D8 Quest. TLC analyses were performed on precoated GF₂₅₄ silica gel plates and were visualized under UV254 nm light or by I₂ staining. Column chromatography was carried out using 300-400 mesh silica gel and eluted with petroleum/ethyl acetate unless otherwise noted.

1.3 Picture of a Typical Reaction Setup

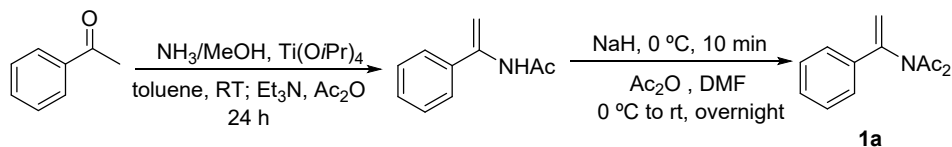


2. Preparation of *N*-vinylimides

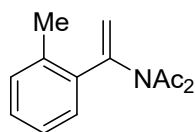
2.1 Known substrates reported in our previous work¹



2.2 Synthesis of *N*-vinylimide^{3,4}



- (a) To a dry 250 mL Schlenk tube was added acetophenone (3.65 g, 30 mmol, 1.0 equiv) and anhydrous toluene (10 mL under nitrogen). The resultant solution was stirred and cooled in an ice/water bath. To the resultant cold stirring solution was added 7N NH₃ in MeOH (6.6 mL, 45 mmol, 1.5 equiv) followed by dropwise addition of Ti(O*i*-Pr)₄ (18 mL, 90 mmol, 3.0 equiv). After 10 min, the ice/water cooling bath was removed, and the solution was stirred at room temperature for 24 h. The reaction mixture was then cooled in -5 °C and added Et₃N (16.7 mL, 120 mmol, 4.0 equiv) followed by Ac₂O (5.7 mL, 60 mmol, 2.0 equiv). The solution was stirred at room temperature for 3 h. The reaction mixture was then added *N,N,N',N'*-tetrakis (2-hydroxyethyl) ethylenediamine (75% W.t., 18 mL, 63 mmol, 2.1 equiv) at room temperature and the solution was then heated at 55 °C for 20 min. The reaction mixture was cooled to room temperature and diluted with NH₄OH (20 mL), water (20 mL), and EtOAc (30 mL). After separation of the organic phase, the resulting aqueous phase was extracted with additional EtOAc (3 × 10 mL). The combined organic layers were dried over anhydrous Na₂SO₄ and evaporated in vacuo. The desired product enamide was obtained after purification by flash chromatography on silica gel with hexane/ethyl acetate as the eluent.
- (b) The *N*-(1-phenylvinyl)acetamide (1.61 g, 10 mmol, 1.0 equiv) was dissolved in dry DMF (10 mL) in a dry round-bottom flask. The solution was cooled to 0 °C and sodium hydride (60% dispersion in mineral oil) (1.0 g, 25 mmol, 2.5 equiv) was added in portions. The resulting suspension was stirred at the same temperature for 10 min. Then Ac₂O (2.81 mL, 30 mmol, 3.0 equiv) was added dropwise and the final solution was continued to stir for overnight at room temperature. The completion of the reaction was confirmed by checking TLC and the excess of sodium hydride was quenched by adding water (20 mL) at 0 °C. The resulting solution was extracted with ethyl acetate (5 × 10 mL). The combined organic layers were dried over anhydrous sodium sulfate, filtered, and evaporated under reduced pressure to give the crude product, which was purified by column chromatography over silica gel to give the pure product **1a**.

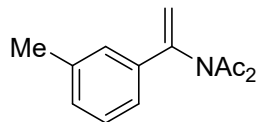


***N*-Acetyl-*N*-(1-(*o*-tolyl)vinyl)acetamide (1b).** Flash column chromatography to afford product as a yellow oil.

¹H NMR (500 MHz, CDCl₃) δ 7.26 – 7.20 (m, 2H), 7.20 – 7.16 (m, 1H), 7.15 – 7.12 (m, 1H), 5.59 (s, 1H), 5.52 (s, 1H), 2.52 (s, 3H), 2.40 (s, 6H).

¹³C NMR (126 MHz, CDCl₃) δ 173.1, 142.7, 136.2, 135.8, 131.7, 128.5, 126.7, 126.1, 119.9, 26.3, 21.1.

HRMS (ESI) [M+Na]⁺: calculated for C₁₃H₁₅NO₂Na: 240.1000, found 240.1003.

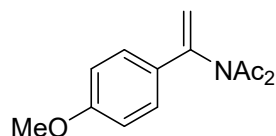


N-Acetyl-N-(1-(m-tolyl)vinyl)acetamide (1c). Flash column chromatography to afford product as a yellow oil.

¹H NMR (500 MHz, CDCl₃) δ 7.26 – 7.12 (m, 4H), 5.99 (s, 1H), 5.27 (s, 1H), 2.39 (s, 6H), 2.39 (s, 6H), 2.34 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 172.7, 144.5, 138.5, 135.0, 129.8, 128.7, 125.4, 122.0, 115.4, 26.1, 21.3.

HRMS (ESI) [M+Na]⁺: calculated for C₁₃H₁₅NO₂Na: 240.1000, found 240.1003.

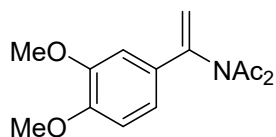


N-Acetyl-N-(1-(4-methoxyphenyl)vinyl)acetamide (1e). Flash column chromatography to afford product as a yellow oil.

¹H NMR (500 MHz, CDCl₃) δ 7.32 (d, *J* = 8.9 Hz, 2H), 6.87 (d, *J* = 8.9 Hz, 2H), 5.87 (s, 1H), 5.18 (s, 1H), 3.79 (s, 3H), 2.39 (s, 6H).

¹³C NMR (126 MHz, CDCl₃) δ 172.8, 160.2, 144.0, 127.7, 126.3, 114.3, 113.5, 55.3, 26.1.

HRMS (ESI) [M+Na]⁺: calculated for C₁₃H₁₅NO₃Na: 256.0950, found 256.0953.

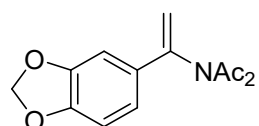


N-Acetyl-N-(1-(3,4-dimethoxyphenyl)vinyl)acetamide (1f). Flash column chromatography to afford product as a yellow solid.

¹H NMR (500 MHz, CDCl₃) δ 6.97 (d, *J* = 2.1 Hz, 1H), 6.89 – 6.85 (m, 1H), 6.82 (d, *J* = 8.4 Hz, 1H), 5.89 (s, 1H), 5.21 (s, 1H), 3.89 (s, 3H), 3.87 (s, 3H), 2.40 (s, 6H).

¹³C NMR (126 MHz, CDCl₃) δ 172.9, 150.1, 149.3, 144.2, 128.1, 117.7, 113.8, 111.1, 108.1, 55.9, 26.2.

HRMS (ESI) [M+Na]⁺: calculated for C₁₄H₁₇NO₄Na: 286.1055, found 286.1059.

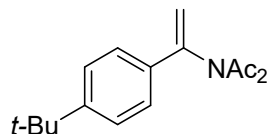


N-Acetyl-N-(1-(benzo[d][1,3]dioxol-5-yl)vinyl)acetamide (1g). Flash column chromatography to afford product as a yellow solid.

¹H NMR (500 MHz, CDCl₃) δ 6.91 (d, *J* = 1.8 Hz, 1H), 6.85 (dd, *J* = 8.2, 1.9 Hz, 1H), 6.77 (d, *J* = 8.2 Hz, 1H), 5.98 (s, 2H), 5.85 (s, 1H), 5.20 (s, 1H), 2.40 (s, 6H).

¹³C NMR (126 MHz, CDCl₃) δ 172.8, 148.5, 148.4, 144.1, 129.6, 119.1, 114.2, 108.5, 105.5, 101.5, 26.2.

HRMS (ESI) [M+Na]⁺: calculated for C₁₃H₁₃NO₄Na: 270.0742, found 270.0746.

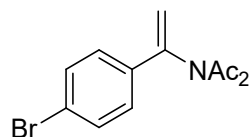


N-Acetyl-N-(1-(4-(tert-butyl)phenyl)vinyl)acetamide (1h). Flash column chromatography to afford product as a yellow solid.

¹H NMR (500 MHz, CDCl₃) δ 7.38 (d, *J* = 8.7 Hz, 2H), 7.33 (d, *J* = 8.5 Hz, 2H), 5.98 (s, 1H), 5.25 (s, 1H), 2.41 (s, 6H), 1.30 (s, 9H).

¹³C NMR (126 MHz, CDCl₃) δ 172.7, 152.3, 144.4, 132.2, 125.9, 124.6, 114.7, 34.6, 31.1, 26.2.

HRMS (ESI) [M+Na]⁺: calculated for C₁₆H₂₁Na: 282.1470, found 282.1471.

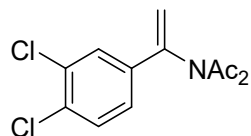


N-Acetyl-N-(1-(4-bromophenyl)vinyl)acetamide (1j). Flash column chromatography to afford product as a yellowoil.

¹H NMR (500 MHz, CDCl₃) δ 7.52 – 7.47 (m, 2H), 7.28 – 7.25 (m, 2H), 6.01 (d, *J* = 1.1 Hz, 1H), 5.33 (d, *J* = 1.1 Hz, 1H), 2.39 (s, 6H).

¹³C NMR (126 MHz, CDCl₃) δ 172.6, 143.7, 134.2, 132.1, 126.5, 123.3, 116.3, 26.2.

HRMS (ESI) [M+Na]⁺: calculated for C₁₂H₁₂NO₂NaBr: 303.9949, found 303.9953.

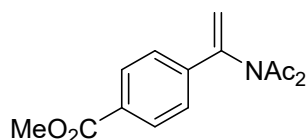


N-Acetyl-N-(1-(3,4-dichlorophenyl)vinyl)acetamide (1k). Flash column chromatography to afford product as a yellow solid.

¹H NMR (500 MHz, CDCl₃) δ 7.42 (d, *J* = 2.0 Hz, 1H), 7.31 – 7.22 (m, 2H), 5.94 (s, 1H), 5.55 (s, 1H), 2.41 (s, 6H).

¹³C NMR (126 MHz, CDCl₃) δ 173.0, 140.7, 135.0, 133.3, 132.1, 131.0, 130.6, 127.6, 122.1, 26.4.

HRMS (ESI) [M+Na]⁺: calculated for C₁₂H₁₁NO₂NaCl₂: 294.0065, found 294.0068.

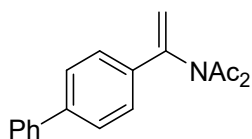


Methyl 4-(1-(N-acetylacetamido)vinyl)benzoate (1l). Flash column chromatography to afford product as a yellow solid.

¹H NMR (500 MHz, CDCl₃) δ 8.03 (d, *J* = 8.6 Hz, 2H), 7.47 (d, *J* = 8.6 Hz, 2H), 6.12 (s, 1H), 5.43 (s, 1H), 3.92 (s, 3H), 2.40 (s, 6H).

¹³C NMR (126 MHz, CDCl₃) δ 172.6, 166.4, 143.9, 139.5, 130.6, 130.3, 124.9, 117.9, 52.3, 26.3.

HRMS (ESI) [M+Na]⁺: calculated for C₁₄H₁₅NO₄Na: 284.0899, found 284.0902.

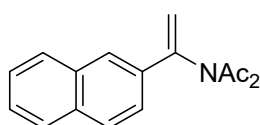


N-(1-([1,1'-Biphenyl]-4-yl)vinyl)-N-acetylacetamide (1m). Flash column chromatography to afford product as a yellow solid.

¹H NMR (500 MHz, CDCl₃) δ 7.59 (t, *J* = 9.0 Hz, 4H), 7.50 – 7.42 (m, 4H), 7.37 (t, *J* = 7.4 Hz, 1H), 6.07 (s, 1H), 5.34 (s, 1H), 2.45 (s, 6H).

¹³C NMR (126 MHz, CDCl₃) δ 172.8, 144.3, 142.0, 140.1, 134.0, 128.9, 127.7, 127.0, 125.4, 115.6, 26.3.

HRMS (ESI) [M+Na]⁺: calculated for C₁₈H₁₇NO₂Na: 302.1157, found 302.1160.

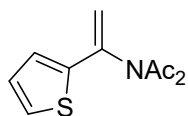


N-Acetyl-N-(1-(naphthalen-2-yl)vinyl)acetamide (1n). Flash column chromatography to afford product as a yellow solid.

¹H NMR (500 MHz, CDCl₃) δ 8.50 (d, *J* = 8.0 Hz, 1H), 7.88 (d, *J* = 9.5 Hz, 1H), 7.82 (d, *J* = 8.1 Hz, 1H), 7.59 – 7.55 (m, 1H), 7.54 – 7.49 (m, 1H), 7.43 (t, *J* = 7.7 Hz, 1H), 7.36 (dd, *J* = 7.3, 1.2 Hz, 1H), 5.87 (s, 1H), 5.74 (s, 1H), 2.46 (s, 6H).

¹³C NMR (126 MHz, CDCl₃) δ 173.3, 141.4, 134.3, 134.2, 130.9, 129.4, 128.7, 126.9, 126.0, 124.9, 124.8, 124.2, 121.1, 26.4.

HRMS (ESI) [M+Na]⁺: calculated for C₁₆H₁₅NO₂Na: 276.1000, found 276.1001.

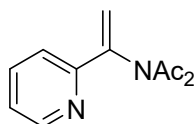


N-Acetyl-N-(1-(thiophen-2-yl)vinyl)acetamide (1o). Flash column chromatography to afford product as a yellow oil.

¹H NMR (500 MHz, CDCl₃) δ 7.24 (d, *J* = 4.7 Hz, 1H), 7.00 – 6.93 (m, 2H), 5.86 (s, 1H), 5.18 (s, 1H), 2.42 (s, 6H).

¹³C NMR (126 MHz, CDCl₃) δ 172.3, 140.3, 139.2, 127.8, 126.3, 125.0, 114.6, 26.0.

HRMS (ESI) [M+Na]⁺: calculated for C₁₀H₁₁NO₂NaS: 232.0408, found 232.0412.

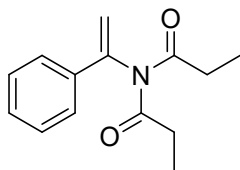


N-Acetyl-N-(1-(pyridin-2-yl)vinyl)acetamide (1p). Flash column chromatography to afford product as a brown oil.

¹H NMR (500 MHz, CDCl₃) δ 8.56 – 8.52 (m, 1H), 7.70 – 7.64 (m, 1H), 7.45 (d, *J* = 8.0 Hz, 1H), 7.22 – 7.18 (m, 1H), 6.38 (s, 1H), 5.51 (s, 1H), 2.38 (s, 6H).

¹³C NMR (126 MHz, CDCl₃) δ 172.9, 152.7, 149.5, 144.5, 136.8, 123.3, 119.7, 118.5, 26.1.

HRMS (ESI) [M+Na]⁺: calculated for C₁₁H₁₂N₂O₂Na: 227.0796, found 227.0800.

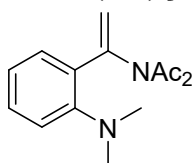


***N*-(1-Phenylvinyl)-*N*-propionylpropionamide (7).** According to the general procedure, propionic anhydride instead of acetic anhydride was used. Flash column chromatography to afford product as a yellow oil.

$^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.47 – 7.21 (m, 5H), 5.98 (s, 1H), 5.25 (s, 1H), 2.73 (q, $J = 6.9$ Hz, 4H), 1.10 (t, $J = 7.3$ Hz, 6H).

$^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 176.3, 144.0, 135.4, 128.82, 128.75, 124.7, 115.4, 31.2, 8.9.

HRMS (ESI) $[\text{M}+\text{Na}]^+$: calculated for $\text{C}_{14}\text{H}_{17}\text{NO}_2\text{Na}$: 254.1157, found 254.1160.



***N*-Acetyl-*N*-(1-(2-(dimethylamino)phenyl)vinyl)acetamide (12).** Flash column chromatography to afford product as a yellow solid.

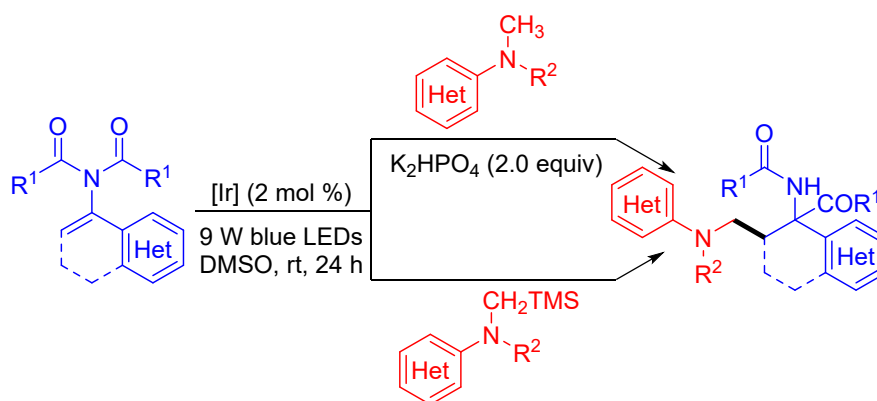
$^1\text{H NMR}$ (500 MHz, CDCl_3) δ 7.28 – 7.20 (m, 1H), 7.20 – 7.15 (m, 1H), 7.13 (d, $J = 8.0$ Hz, 1H), 6.98 (t, $J = 7.5$ Hz, 1H), 6.43 (s, 1H), 5.42 (s, 1H), 2.70 (s, 6H), 2.39 (d, $J = 1.2$ Hz, 6H).

$^{13}\text{C NMR}$ (126 MHz, CDCl_3) δ 172.8, 152.4, 143.0, 129.4, 129.0, 127.7, 122.7, 120.0, 118.7, 43.9, 26.1.

HRMS (ESI) $[\text{M}+\text{H}]^+$: calculated for $\text{C}_{14}\text{H}_{19}\text{N}_2\text{O}_2$: 247.1447, found 247.1446.

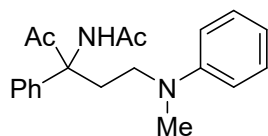
3. General procedure of photoredox-catalyzed acyl migration reactions

reactions



To an oven dried transparent 10 mL Schlenk tube equipped with stirring bar, $\text{Ir}[\text{dF}(\text{CF}_3)\text{ppy}]_2(\text{dtbbpy})\text{PF}_6$ (4.5 mg, 0.004 mmol, 0.02 equiv), *N*-vinylimide (0.2 mmol, 1.0 equiv), and degassed DMSO (4 mL) were added. The tube was evacuated and filled with nitrogen for 3 times. The tube was then charged under nitrogen with α -silylamine (0.4 mmol, 2.0 equiv) or a mixture of tertiary amine (0.4 mmol, 2.0 equiv) and K_2HPO_4 (0.4 mmol, 2.0 equiv). The tube was irradiated with a 9 W blue LEDs strip spiraled within a bowel for 24 h (cooling with a fan). After the reaction was complete, the reaction solution was quenched by the addition of water (5

mL) and extracted with EtOAc (5 x 10 mL). The combined organic layer was washed with brine, dried over Na₂SO₄, filtered, and solvent was evaporated to obtain crude product. Flash chromatography over silica gel afforded the product.

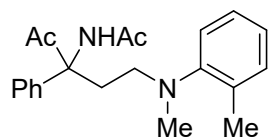


***N*-(1-(Methyl(phenyl)amino)-4-oxo-3-phenylpentan-3-yl)acetamide (3a).** Yellow oil (40.8 mg, 78% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.46 (s, 1H), 7.34-7.33 (m, 4H), 7.30 – 7.22 (m, 3H), 6.73 (t, *J* = 7.3 Hz, 1H), 6.69 (d, *J* = 8.1 Hz, 2H), 3.50 – 3.43 (m, 1H), 3.33 – 3.26 (m, 1H), 3.18 – 3.11 (m, 1H), 2.82 (s, 3H), 2.81 – 2.75 (m, 1H), 2.05 (s, 3H), 1.71 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 204.0, 168.5, 148.9, 139.3, 129.3, 128.9, 128.0, 126.0, 117.0, 112.6, 68.5, 48.5, 40.0, 29.3, 23.8, 23.2.

HRMS (ESI) [M+H]⁺: calculated for C₂₀H₂₅N₂O₂: 325.1911, found 325.1920.

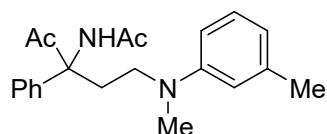


***N*-(1-(Methyl(*o*-tolyl)amino)-4-oxo-3-phenylpentan-3-yl)acetamide (3b).** Yellow oil (52.8 mg, 78% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.37 – 7.30 (m, 5H), 7.29 – 7.24 (m, 1H), 7.16 (t, *J* = 7.6 Hz, 2H), 7.04 (d, *J* = 7.8 Hz, 1H), 6.98 (t, *J* = 7.4 Hz, 1H), 3.33 – 3.25 (m, 1H), 3.00 – 2.93 (m, 1H), 2.72 – 2.64 (m, 1H), 2.67 (s, 3H), 2.54 – 2.47 (m, 1H), 2.27 (s, 3H), 1.99 (s, 3H), 1.80 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 204.9, 168.3, 151.5, 139.3, 133.1, 131.3, 128.8, 127.9, 126.5, 126.0, 123.3, 119.8, 68.8, 51.2, 42.5, 29.0, 23.8, 23.3, 18.2.

HRMS(ESI) [M+H]⁺: calculated for C₂₁H₂₇N₂O₂: 339.2073, found 339.2077.

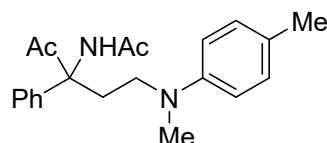


***N*-(1-(Methyl(*m*-tolyl)amino)-4-oxo-3-phenylpentan-3-yl)acetamide (3c).** Yellow oil (57.5 mg, 85% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.47 (s, 1H), 7.35 (d, *J* = 4.3 Hz, 4H), 7.30 – 7.25 (m, 1H), 7.13 (t, *J* = 7.8 Hz, 1H), 6.57 (d, *J* = 7.4 Hz, 1H), 6.52 – 6.48 (m, 2H), 3.47 – 3.41 (m, 1H), 3.31 – 3.25 (m, 1H), 3.17 – 3.11 (m, 1H), 2.82 (s, 3H), 2.80 – 2.74 (m, 1H), 2.32 (s, 3H), 2.04 (s, 3H), 1.74 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 204.0, 168.5, 148.9, 139.2, 138.9, 129.1, 128.8, 127.9, 126.0, 117.9, 113.3, 109.8, 68.5, 48.5, 39.9, 29.2, 23.8, 23.2, 21.8.

HRMS (ESI) [M+H]⁺: calculated for C₂₁H₂₇N₂O₂: 339.2073, found 339.2076.



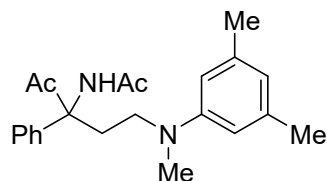
***N*-(1-(Methyl(*p*-tolyl)amino)-4-oxo-3-phenylpentan-3-yl)acetamide (3d).** Yellow oil (49.4 mg,

73% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.48 (s, 1H), 7.34 (d, *J* = 4.3 Hz, 4H), 7.29 – 7.25 (m, 1H), 7.06 (d, *J* = 8.4 Hz, 2H), 6.62 (d, *J* = 8.5 Hz, 2H), 3.45 – 3.39 (m, 1H), 3.30 – 3.24 (m, 1H), 3.15 – 3.07 (m, 1H), 2.81 – 2.74 (m, 4H), 2.25 (s, 3H), 2.04 (s, 3H), 1.71 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 203.9, 168.5, 146.9, 139.4, 129.8, 128.8, 127.9, 126.5, 126.0, 113.1, 68.5, 48.9, 40.3, 29.3, 23.8, 23.2, 20.2.

HRMS (ESI) [M+H]⁺: calculated for C₂₁H₂₇N₂O₂: 339.2073, found 339.2076.



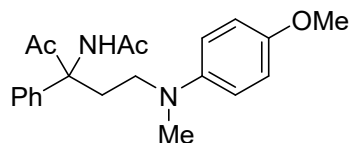
***N*-(1-((3,5-Dimethylphenyl)(methyl)amino)-4-oxo-3-phenylpentan-3-yl)acetamide (3e).**

Yellow oil (61.3 mg, 87% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.43 (s, 1H), 7.34 (d, *J* = 4.3 Hz, 4H), 7.30 – 7.25 (m, 1H), 6.41 (s, 1H), 6.30 (s, 2H), 3.44 – 3.37 (m, 1H), 3.30 – 3.24 (m, 1H), 3.16 – 3.09 (m, 1H), 2.81 (s, 3H), 2.78 – 2.72 (m, 1H), 2.27 (s, 6H), 2.04 (s, 3H), 1.76 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 204.1, 168.5, 149.1, 139.3, 138.8, 128.8, 128.0, 126.0, 119.1, 110.6, 68.6, 48.6, 40.0, 29.2, 23.8, 23.2, 21.7.

HRMS (ESI) [M+H]⁺: calculated for C₂₂H₂₉N₂O₂: 353.2229, found 353.2233.

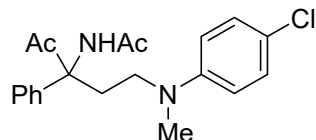


***N*-(1-((4-Methoxyphenyl)(methyl)amino)-4-oxo-3-phenylpentan-3-yl)acetamide (3f).** Yellow oil (50.3 mg, 71% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.48 (s, 1H), 7.34-7.32 (m, 4H), 7.29 – 7.24 (m, 1H), 6.87 – 6.81 (m, 2H), 6.71 – 6.65 (m, 2H), 3.76 (s, 3H), 3.39 – 3.33 (m, 1H), 3.28 – 3.22 (m, 1H), 3.09 – 3.02 (m, 1H), 2.77 – 2.70 (m, 4H), 2.04 (s, 3H), 1.70 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 203.9, 168.5, 152.2, 143.8, 139.4, 128.8, 128.0, 126.1, 114.9, 114.8, 68.6, 55.7, 49.5, 40.9, 29.3, 23.9, 23.3.

HRMS (ESI) [M+Na]⁺: calculated for C₂₁H₂₆N₂O₃Na: 377.1841, found 377.1845.

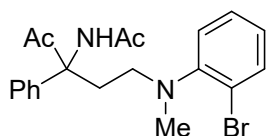


***N*-(1-((4-Chlorophenyl)(methyl)amino)-4-oxo-3-phenylpentan-3-yl)acetamide (3g).** Yellow oil (50.2 mg, 70%).

¹H NMR (500 MHz, CDCl₃) δ 7.42 (s, 1H), 7.37 – 7.27 (m, 5H), 7.17 (d, *J* = 9.0 Hz, 2H), 6.58 (d, *J* = 9.0 Hz, 2H), 3.46 – 3.39 (m, 1H), 3.31 – 3.25 (m, 1H), 3.14 – 3.07 (m, 1H), 2.80 (s, 3H), 2.77 – 2.70 (m, 1H), 2.04 (s, 3H), 1.72 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 203.9, 168.5, 147.5, 139.1, 129.1, 128.9, 128.1, 125.9, 121.9, 113.7, 68.5, 48.7, 40.0, 29.1, 23.8, 23.2.

HRMS (ESI) [M+H]⁺: calculated for C₂₀H₂₄N₂O₂Cl: 359.1526, found 359.1530.

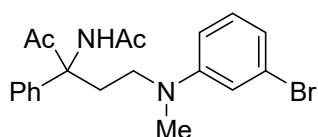


***N*-(1-((2-Bromophenyl)(methyl)amino)-4-oxo-3-phenylpentan-3-yl)acetamide (3h)**. Yellow oil (69.3 mg, 86% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.56 – 7.52 (m, 1H), 7.41 (s, 1H), 7.33 (d, *J* = 4.3 Hz, 4H), 7.28 – 7.23 (m, 2H), 7.09 – 7.04 (m, 1H), 6.93 – 6.87 (m, 1H), 3.26 – 3.19 (m, 1H), 2.98 – 2.91 (m, 1H), 2.84 – 2.76 (m, 1H), 2.72 (s, 3H), 2.59 – 2.52 (m, 1H), 1.99 (s, 3H), 1.90 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 204.6, 168.7, 150.9, 138.7, 133.8, 128.8, 128.2, 127.8, 126.0, 124.6, 121.9, 120.0, 69.0, 51.6, 41.7, 29.0, 23.6(3), 23.6(0).

HRMS (ESI) [M+H]⁺: calculated for C₂₀H₂₄N₂O₂Br: 403.1021, found 403.1024.

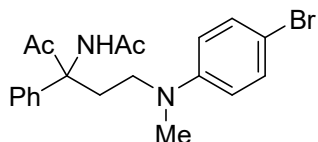


***N*-(1-((3-Bromophenyl)(methyl)amino)-4-oxo-3-phenylpentan-3-yl)acetamide (3i)**. Yellow oil (68.5 mg, 85% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.41 (s, 1H), 7.38 – 7.26 (m, 5H), 7.07 (t, *J* = 8.1 Hz, 1H), 6.84 – 6.81 (m, 1H), 6.76–6.75 (m, 1H), 6.57 (dd, *J* = 8.4, 2.3 Hz, 1H), 3.42 – 3.36 (m, 1H), 3.30 – 3.23 (m, 1H), 3.17 – 3.09 (m, 1H), 2.82 (s, 3H), 2.75 – 2.68 (m, 1H), 2.05 (s, 3H), 1.77 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 204.0, 168.6, 150.0, 139.0, 130.5, 128.9, 128.1, 125.9, 123.5, 119.6, 115.1, 110.9, 68.5, 48.3, 39.6, 28.9, 23.8, 23.2.

HRMS (ESI) [M+H]⁺: calculated for C₂₀H₂₄N₂O₂Br: 403.1021, found 403.1025.

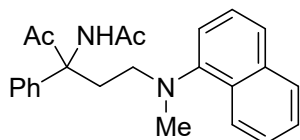


***N*-(1-((4-Bromophenyl)(methyl)amino)-4-oxo-3-phenylpentan-3-yl)acetamide (3j)**. Yellow oil (58.8 mg, 73% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.42 (s, 1H), 7.38 – 7.26 (m, 7H), 6.53 (d, *J* = 9.0 Hz, 2H), 3.45 – 3.38 (m, 1H), 3.31 – 3.23 (m, 1H), 3.15 – 3.07 (m, 1H), 2.80 (s, 3H), 2.77 – 2.68 (m, 1H), 2.04 (s, 3H), 1.72 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 203.9, 168.5, 147.8, 139.1, 132.0, 128.9, 128.1, 125.9, 114.1, 109.0, 68.5, 48.6, 39.9, 29.1, 23.8, 23.2.

HRMS (ESI) [M+H]⁺: calculated for C₂₀H₂₄N₂O₂Br: 403.1021, found 403.1025.



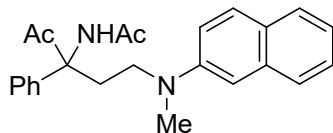
***N*-(1-((Methyl(naphthalen-1-yl)amino)-4-oxo-3-phenylpentan-3-yl)acetamide (3k)**. Yellow oil (61.4 mg, 82% yield).

¹H NMR (500 MHz, CDCl₃) δ 8.14 (d, *J* = 9.0 Hz, 1H), 7.85 – 7.81 (m, 1H), 7.55 (d, *J* = 8.2 Hz, 1H), 7.52 – 7.44 (m, 2H), 7.41 (t, *J* = 7.8 Hz, 1H), 7.37 – 7.30 (m, 5H), 7.29 – 7.24 (m, 1H), 7.12

(d, $J = 7.4$ Hz, 1H), 3.44 – 3.36 (m, 1H), 3.23 – 3.16 (m, 1H), 2.94 – 2.88 (m, 1H), 2.87 (s, 3H), 2.68 – 2.60 (m, 1H), 1.91 (s, 3H), 1.73 (s, 3H).

^{13}C NMR (126 MHz, CDCl_3) δ 204.8, 168.5, 149.3, 139.1, 134.8, 129.2, 128.8, 128.3, 127.9, 126.0, 125.8, 125.6, 125.3, 123.7, 123.5, 115.3, 68.8, 51.6, 43.6, 29.1, 23.6, 23.3.

HRMS (ESI) $[\text{M}+\text{H}]^+$: calculated for $\text{C}_{24}\text{H}_{27}\text{N}_2\text{O}_2$: 375.2073, found 375.2076.

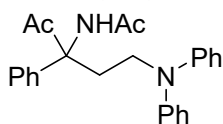


***N*-(1-(Methyl(naphthalen-2-yl)amino)-4-oxo-3-phenylpentan-3-yl)acetamide (3l)**. White solid (64.4 mg, 86% yield).

^1H NMR (500 MHz, CDCl_3) δ 7.72 (d, $J = 9.1$ Hz, 1H), 7.68 (dd, $J = 16.8, 8.2$ Hz, 2H), 7.49 (s, 1H), 7.39 (t, $J = 8.0$ Hz, 1H), 7.35 (d, $J = 4.3$ Hz, 4H), 7.31 – 7.27 (m, 1H), 7.23 (t, $J = 7.8$ Hz, 1H), 7.07 (dd, $J = 9.0, 2.5$ Hz, 1H), 6.92 (d, $J = 2.2$ Hz, 1H), 3.61 – 3.54 (m, 1H), 3.39 – 3.32 (m, 1H), 3.30 – 3.22 (m, 1H), 2.95 (s, 3H), 2.87 – 2.79 (m, 1H), 2.07 (s, 3H), 1.73 (s, 3H).

^{13}C NMR (126 MHz, CDCl_3) δ 204.1, 168.6, 146.7, 139.2, 134.9, 129.1, 128.9, 128.0, 127.4, 126.9, 126.3, 126.2, 126.0, 122.3, 116.0, 106.9, 68.6, 48.7, 40.2, 29.3, 23.8, 23.3.

HRMS (ESI) $[\text{M}+\text{H}]^+$: calculated for $\text{C}_{24}\text{H}_{27}\text{N}_2\text{O}_2$: 375.2073, found 375.2077.

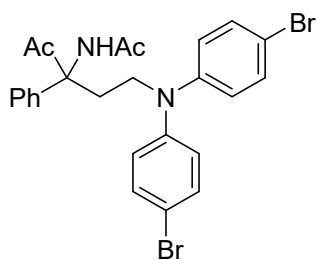


***N*-(1-(Diphenylamino)-4-oxo-3-phenylpentan-3-yl)acetamide (3m)**. Yellow oil (58.7 mg, 76% yield).

^1H NMR (500 MHz, CDCl_3) δ 7.37 – 7.30 (m, 5H), 7.30 – 7.25 (m, 5H), 7.02 – 6.94 (m, 6H), 3.82 – 3.75 (m, 1H), 3.55 – 3.44 (m, 2H), 2.72 – 2.66 (m, 1H), 2.00 (s, 3H), 1.75 (s, 3H).

^{13}C NMR (126 MHz, CDCl_3) δ 204.9, 168.5, 148.0, 139.0, 129.4, 128.9, 128.0, 125.9, 121.7, 121.0, 68.7, 48.1, 28.9, 23.7, 23.2.

HRMS (ESI) $[\text{M}+\text{Na}]^+$: calculated for $\text{C}_{25}\text{H}_{26}\text{N}_2\text{O}_2\text{Na}$: 409.1892, found 409.1895.

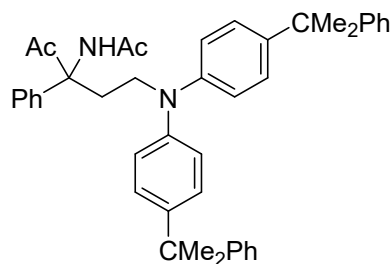


***N*-(1-(Bis(4-bromophenyl)amino)-4-oxo-3-phenylpentan-3-yl)acetamide (3n)**. Yellow solid (63.1 mg, 58% yield).

^1H NMR (500 MHz, CDCl_3) δ 7.38 – 7.32 (m, 6H), 7.29 (t, $J = 6.3$ Hz, 4H), 6.84 (d, $J = 8.8$ Hz, 4H), 3.73 – 3.64 (m, 1H), 3.50 – 3.37 (m, 2H), 2.65 – 2.56 (m, 1H), 2.01 (s, 3H), 1.79 (s, 3H).

^{13}C NMR (126 MHz, CDCl_3) δ 204.7, 168.7, 146.6, 138.7, 132.4, 129.0, 128.2, 125.8, 122.6, 114.6, 68.6, 48.2, 29.0, 23.7, 23.3.

HRMS (ESI) $[\text{M}+\text{Na}]^+$: calculated for $\text{C}_{25}\text{H}_{24}\text{N}_2\text{O}_2\text{NaBr}_2$: 565.0102, found 565.0106.



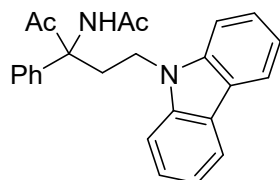
***N*-(1-(Bis(4-(2-phenylpropan-2-yl)phenyl)amino)-4-oxo-3-phenylpentan-3-yl)acetamide (3o).**

Yellow oil (66.0 mg, 53% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.37 – 7.32 (m, 5H), 7.31 – 7.26 (m, 9H), 7.21 – 7.17 (m, 2H), 7.14 (d, *J* = 8.7 Hz, 4H), 6.91 (d, *J* = 8.7 Hz, 4H), 3.83 – 3.76 (m, 1H), 3.59 – 3.51 (m, 1H), 3.45 – 3.38 (m, 1H), 2.70 – 2.62 (m, 1H), 1.99 (s, 3H), 1.72 (s, 3H), 1.69 (s, 12H).

¹³C NMR (126 MHz, CDCl₃) δ 205.0, 168.5, 150.8, 145.5, 143.8, 139.1, 128.9, 128.0, 127.9, 127.7, 126.7, 126.0, 125.5, 120.4, 68.8, 48.2, 42.3, 30.7, 28.7, 23.7, 23.1.

HRMS (ESI) [M+Na]⁺: calculated for C₄₃H₄₆N₂O₂Na: 645.3457, found 645.3459.

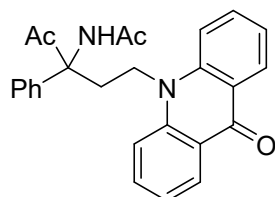


***N*-(1-(9H-Carbazol-9-yl)-4-oxo-3-phenylpentan-3-yl)acetamide (3p).** Colorless oil (66.9 mg, 87% yield).

¹H NMR (500 MHz, CDCl₃) δ 8.09 (d, *J* = 8.0 Hz, 2H), 7.45 (t, *J* = 7.7 Hz, 2H), 7.37 – 7.28 (m, 5H), 7.27 – 7.23 (m, 4H), 7.08 (s, 1H), 4.36 – 4.29 (m, 1H), 4.20 – 4.13 (m, 1H), 3.65 – 3.58 (m, 1H), 2.94 – 2.87 (m, 1H), 1.90 (s, 3H), 1.80 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 204.0, 169.0, 139.9, 138.2, 129.1, 128.3, 125.8(8), 125.8(6), 123.0, 120.4, 119.2, 108.5, 68.7, 38.5, 30.2, 23.4, 23.0.

HRMS (ESI) [M+Na]⁺: calculated for C₂₅H₂₄N₂O₂Na: 407.1735, found 407.1739.

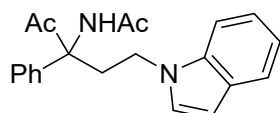


***N*-(4-Oxo-1-(9-oxoacridin-10(9H)-yl)-3-phenylpentan-3-yl)acetamide (3q).** Yellow solid (52.0 mg, 63% yield).

¹H NMR (500 MHz, CDCl₃) δ 8.48 (d, *J* = 8.2 Hz, 2H), 7.59 – 7.53 (m, 2H), 7.49 (s, 1H), 7.40 (d, *J* = 4.2 Hz, 4H), 7.38 – 7.33 (m, 1H), 7.22 – 7.16 (m, 4H), 4.13 – 4.06 (m, 1H), 4.04 – 3.97 (m, 1H), 3.32 – 3.23 (m, 1H), 2.83 – 2.73 (m, 1H), 2.24 (s, 3H), 2.06 (s, 3H), .

¹³C NMR (126 MHz, CDCl₃) δ 204.5, 178.1, 169.9, 141.6, 137.8, 134.1, 129.3, 128.6, 127.8, 125.6, 122.4, 121.3, 114.1, 68.7, 42.1, 30.6, 23.9, 23.6.

HRMS (ESI) [M+Na]⁺: calculated for C₂₆H₂₄N₂O₃Na: 435.1685, found 435.1687.

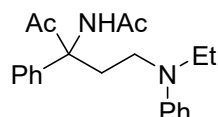


***N*-(1-(1*H*-Indol-1-yl)-4-oxo-3-phenylpentan-3-yl)acetamide (3r).** Yellow oil (21.4 mg, 32% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.62 (d, *J* = 7.9 Hz, 1H), 7.41 – 7.36 (m, 2H), 7.35 – 7.30 (m, 3H), 7.25 – 7.19 (m, 2H), 7.13 – 7.09 (m, 1H), 6.85 (d, *J* = 3.1 Hz, 1H), 6.81 (s, 1H), 6.48 (d, *J* = 3.1 Hz, 1H), 4.28 – 4.22 (m, 1H), 4.01 – 3.94 (m, 1H), 3.56 – 3.49 (m, 1H), 2.97 – 2.89 (m, 1H), 1.77 (s, 3H), 1.70 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 204.0, 169.1, 137.9, 135.6, 129.1, 128.8, 128.3, 128.0, 125.9, 121.9, 121.2, 119.7, 109.3, 101.6, 68.8, 42.0, 31.4, 23.3, 22.9.

HRMS (ESI) [M+Na]⁺: calculated for C₂₁H₂₂N₂O₂Na: 357.1579, found 357.1583.

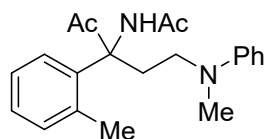


***N*-(1-(Ethyl(phenyl)amino)-4-oxo-3-phenylpentan-3-yl)acetamide (3s).** Brown oil (58.2 mg, 86% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.48 (s, 1H), 7.34 (d, *J* = 4.3 Hz, 4H), 7.30 – 7.21 (m, 3H), 6.75 – 6.68 (m, 3H), 3.46 – 3.34 (m, 2H), 3.33 – 3.24 (m, 1H), 3.11 – 3.01 (m, 2H), 2.83 – 2.74 (m, 1H), 2.04 (s, 3H), 1.72 (s, 3H), 1.08 (t, *J* = 7.0 Hz, 3H), .

¹³C NMR (126 MHz, CDCl₃) δ 204.1, 168.5, 147.2, 139.4, 129.4, 128.8, 127.9, 126.0, 116.8, 113.2, 68.6, 46.2, 45.5, 29.5, 23.8, 23.3, 11.4.

HRMS (ESI) [M+H]⁺: calculated for C₂₁H₂₇N₂O₂: 339.2073, found 339.2075.

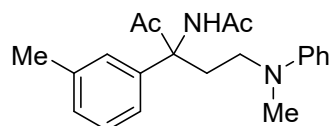


***N*-(1-(Methyl(phenyl)amino)-4-oxo-3-(*o*-tolyl)pentan-3-yl)acetamide (4a).** Yellow oil (52.8 mg, 78%).

¹H NMR (500 MHz, CDCl₃) δ 7.63 (d, *J* = 7.4 Hz, 1H), 7.28 – 7.19 (m, 5H), 7.09 (d, *J* = 7.3 Hz, 1H), 6.73 (t, *J* = 7.3 Hz, 1H), 6.69 (d, *J* = 8.0 Hz, 2H), 3.50 – 3.43 (m, 1H), 3.38 – 3.31 (m, 1H), 3.21 – 3.14 (m, 1H), 2.88 (s, 3H), 2.55 – 2.47 (m, 1H), 2.13 (s, 3H), 2.03 (s, 3H), 1.83 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 205.3, 167.7, 148.9, 136.7, 135.6, 132.5, 129.3, 128.2, 127.6, 126.5, 116.9, 112.6, 68.6, 47.9, 39.2, 29.3, 23.8, 23.5, 20.5.

HRMS (ESI) [M+H]⁺: calculated for: C₂₁H₂₇N₂O₂: 339.2073, found 339.2078.

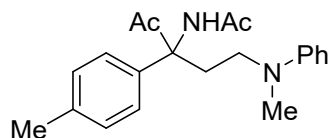


***N*-(1-(Methyl(phenyl)amino)-4-oxo-3-(*m*-tolyl)pentan-3-yl)acetamide (4b).** Yellow oil (52.1 mg, 77% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.44 (s, 1H), 7.29 – 7.22 (m, 3H), 7.17 – 7.08 (m, 3H), 6.75 (t, *J* = 7.3 Hz, 1H), 6.71 (d, *J* = 8.1 Hz, 2H), 3.50 – 3.44 (m, 1H), 3.34 – 3.27 (m, 1H), 3.19 – 3.12 (m, 1H), 2.85 (s, 3H), 2.82 – 2.74 (m, 1H), 2.35 (s, 3H), 2.07 (s, 3H), 1.75 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 204.1, 168.5, 148.9, 139.2, 138.5, 129.3, 128.8, 128.7, 126.6, 123.1, 117.0, 112.6, 68.5, 48.5, 39.9, 29.2, 23.9, 23.2, 21.6.

HRMS (ESI) [M+H]⁺: calculated for C₂₁H₂₇N₂O₂: 339.2073, found 339.2077.

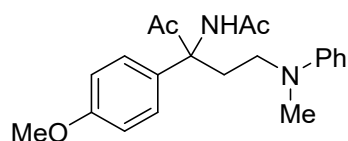


***N*-(1-(Methyl(phenyl)amino)-4-oxo-3-(*p*-tolyl)pentan-3-yl)acetamide (4c).** Yellow oil (58.2 mg, 86% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.45 (s, 1H), 7.27 – 7.21 (m, 4H), 7.15 (d, *J* = 8.1 Hz, 2H), 6.74 (t, *J* = 7.3 Hz, 1H), 6.69 (d, *J* = 8.1 Hz, 2H), 3.49 – 3.42 (m, 1H), 3.30 – 3.24 (m, 1H), 3.18 – 3.11 (m, 1H), 2.83 (s, 3H), 2.80 – 2.73 (m, 1H), 2.32 (s, 3H), 2.04 (s, 3H), 1.73 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 204.1, 168.5, 148.9, 137.7, 136.3, 129.6, 129.3, 125.9, 116.9, 112.6, 68.3, 48.5, 39.9, 29.3, 23.8, 23.1, 20.9.

HRMS (ESI) [M+H]⁺: calculated for C₂₁H₂₇N₂O₂: 339.2073, found 339.2077.

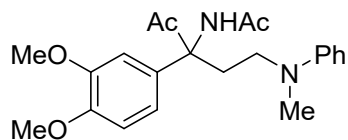


***N*-(3-(4-Methoxyphenyl)-1-(methyl(phenyl)amino)-4-oxopentan-3-yl)acetamide (4d).** Yellow solid (51.7 mg, 73% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.43 (s, 1H), 7.27 – 7.22 (m, 4H), 6.86 (d, *J* = 8.8 Hz, 2H), 6.73 (t, *J* = 7.3 Hz, 1H), 6.69 (d, *J* = 8.2 Hz, 2H), 3.78 (s, 3H), 3.49 – 3.42 (m, 1H), 3.31 – 3.24 (m, 1H), 3.17 – 3.10 (m, 1H), 2.82 (s, 3H), 2.78 – 2.71 (m, 1H), 2.04 (s, 3H), 1.72 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 204.2, 168.5, 159.2, 148.9, 131.2, 129.3, 127.2, 117.0, 114.2, 112.6, 68.0, 55.2, 48.6, 40.0, 29.3, 23.9, 23.1.

HRMS (ESI) [M+H]⁺: calculated for C₂₁H₂₇N₂O₃: 355.2022, found 355.2022.

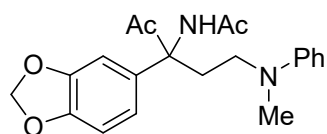


***N*-(3-(3,4-Dimethoxyphenyl)-1-(methyl(phenyl)amino)-4-oxopentan-3-yl)acetamide (4e).** Yellow oil (65.3 mg, 85% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.40 (s, 1H), 7.27 – 7.21 (m, 2H), 6.93 (dd, *J* = 8.4, 2.3 Hz, 1H), 6.82 (d, *J* = 8.5 Hz, 1H), 6.75 – 6.71 (m, 2H), 6.68 (d, *J* = 8.0 Hz, 2H), 3.84 (s, 3H), 3.83 (s, 3H), 3.47 – 3.41 (m, 1H), 3.30 – 3.24 (m, 1H), 3.16 – 3.09 (m, 1H), 2.82 (s, 3H), 2.75 – 2.68 (m, 1H), 2.04 (s, 3H), 1.74 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 204.1, 168.5, 149.2, 148.9, 148.8, 131.7, 129.3, 118.7, 117.0, 112.5, 111.2, 109.2, 68.1, 56.0, 55.8, 48.6, 39.9, 29.2, 23.8, 23.0.

HRMS (ESI) [M+H]⁺: calculated for C₂₂H₂₉N₂O₄: 385.2127, found 385.2131.



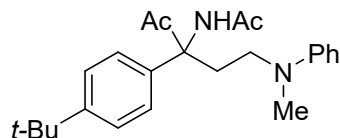
***N*-(3-(Benzo[d][1,3]dioxol-5-yl)-1-(methyl(phenyl)amino)-4-oxopentan-3-yl)acetamide (4f).** White solid (53.0 mg, 72% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.44 (s, 1H), 7.27 – 7.22 (t, *J* = 7.7 Hz, 2H), 6.83 (dd, *J* = 8.2, 1.8

Hz, 1H), 6.79 – 6.71 (m, 3H), 6.68 (d, $J = 8.2$ Hz, 2H), 5.94 (s, 2H), 3.49 – 3.42 (m, 1H), 3.27 – 3.21 (m, 1H), 3.16 – 3.08 (m, 1H), 2.82 (s, 3H), 2.74 – 2.67 (m, 1H), 2.05 (s, 3H), 1.73 (s, 3H).

^{13}C NMR (126 MHz, CDCl_3) δ 203.8, 168.5, 148.8, 148.2, 147.3, 133.3, 129.3, 119.7, 117.1, 112.6, 108.4, 106.6, 101.3, 68.1, 48.6, 40.0, 29.4, 23.8, 23.0.

HRMS (ESI) $[\text{M}+\text{H}]^+$: calculated for $\text{C}_{21}\text{H}_{25}\text{N}_2\text{O}_4$: 369.1814, found 369.1818.



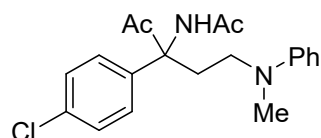
N-(3-(4-*tert*-Butylphenyl)-1-(methyl(phenyl)amino)-4-oxopentan-3-yl)acetamide (4g).

Yellow oil (67.8 mg, 89% yield).

^1H NMR (500 MHz, CDCl_3) δ 7.46 (s, 1H), 7.34 (d, $J = 8.5$ Hz, 2H), 7.27 – 7.22 (m, 4H), 6.73 (t, $J = 7.3$ Hz, 1H), 6.68 (d, $J = 8.2$ Hz, 2H), 3.49 – 3.42 (m, 1H), 3.32 – 3.25 (m, 1H), 3.18 – 3.10 (m, 1H), 2.82 (s, 3H), 2.80 – 2.74 (m, 1H), 2.05 (s, 3H), 1.73 (s, 3H), 1.29 (s, 9H).

^{13}C NMR (126 MHz, CDCl_3) δ 204.1, 168.6, 150.7, 148.9, 135.9, 129.2, 125.7, 125.6, 116.9, 112.5, 68.3, 48.6, 39.9, 34.4, 31.2, 29.3, 23.8, 23.2.

HRMS (ESI) $[\text{M}+\text{H}]^+$: calculated for $\text{C}_{24}\text{H}_{33}\text{N}_2\text{O}_2$: 381.2542, found 381.2545.

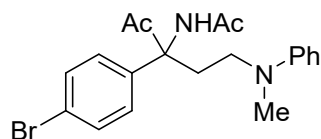


N-(3-(4-Chlorophenyl)-1-(methyl(phenyl)amino)-4-oxopentan-3-yl)acetamide (4h). Yellow oil (62.4 mg, 87% yield).

^1H NMR (500 MHz, CDCl_3) δ 7.47 (s, 1H), 7.33 – 7.23 (m, 6H), 6.74 (t, $J = 7.2$ Hz, 1H), 6.68 (d, $J = 8.2$ Hz, 2H), 3.50 – 3.43 (m, 1H), 3.29 – 3.23 (m, 1H), 3.16 – 3.09 (m, 1H), 2.81 (s, 3H), 2.79 – 2.72 (m, 1H), 2.04 (s, 3H), 1.70 (s, 3H).

^{13}C NMR (126 MHz, CDCl_3) δ 203.4, 168.5, 148.8, 138.1, 134.0, 129.3, 129.0, 127.5, 117.2, 112.7, 68.1, 48.5, 40.2, 29.3, 23.8, 23.1.

HRMS (ESI) $[\text{M}+\text{H}]^+$: calculated for $\text{C}_{20}\text{H}_{24}\text{N}_2\text{O}_2\text{Cl}$: 359.1526, found 359.1528.

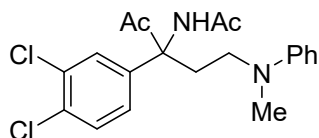


N-(3-(4-Bromophenyl)-1-(methyl(phenyl)amino)-4-oxopentan-3-yl)acetamide (4i). Yellow oil (64.4 mg, 80% yield).

^1H NMR (500 MHz, CDCl_3) δ 7.51 – 7.43 (m, 3H), 7.27 – 7.20 (m, 4H), 6.75 (t, $J = 7.3$ Hz, 1H), 6.69 (d, $J = 8.1$ Hz, 2H), 3.50 – 3.44 (m, 1H), 3.29 – 3.22 (m, 1H), 3.16 – 3.09 (m, 1H), 2.81 (s, 3H), 2.79 – 2.72 (m, 1H), 2.05 (s, 3H), 1.70 (s, 3H).

^{13}C NMR (126 MHz, CDCl_3) δ 203.3, 168.5, 148.7, 138.6, 132.0, 129.3, 127.8, 122.2, 117.2, 112.7, 68.1, 48.5, 40.2, 29.3, 23.8, 23.1.

HRMS (ESI) $[\text{M}+\text{H}]^+$: calculated for $\text{C}_{20}\text{H}_{24}\text{N}_2\text{O}_2\text{Br}$: 403.1021, found 403.1024.

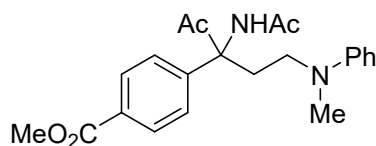


N-(3-(3,4-Dichlorophenyl)-1-(methyl(phenyl)amino)-4-oxopentan-3-yl)acetamide (4j). Yellow solid (59.8 mg, 76% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.66 (d, *J* = 8.7 Hz, 1H), 7.34 (d, *J* = 2.2 Hz, 1H), 7.31 (dd, *J* = 8.6, 2.2 Hz, 1H), 7.29 – 7.21 (m, 3H), 6.73 (t, *J* = 7.3 Hz, 1H), 6.67 (d, *J* = 8.7 Hz, 2H), 3.49 – 3.41 (m, 1H), 3.35 – 3.27 (m, 1H), 3.22 – 3.14 (m, 1H), 2.88 (s, 3H), 2.32 – 2.24 (m, 1H), 2.02 (s, 3H), 1.92 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 203.2, 168.5, 148.6, 135.3, 134.9, 133.2, 130.8, 130.6, 129.3, 127.2, 116.9, 112.5, 67.6, 47.4, 39.1, 28.4, 23.9, 23.4.

HRMS (ESI) [M+H]⁺: calculated for C₂₀H₂₃N₂O₂Cl₂: 393.1137, found 393.1140.

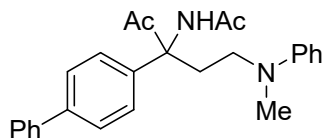


Methyl 4-(3-acetamido-1-(methyl(phenyl)amino)-4-oxopentan-3-yl)benzoate (4k). Yellow solid (61.9 mg, 80% yield).

¹H NMR (500 MHz, CDCl₃) δ 8.00 (d, *J* = 8.6 Hz, 2H), 7.50 (s, 1H), 7.42 (d, *J* = 8.6 Hz, 2H), 7.28 – 7.22 (m, 2H), 6.74 (t, *J* = 7.3 Hz, 1H), 6.69 (d, *J* = 8.1 Hz, 2H), 3.89 (s, 3H), 3.52 – 3.45 (m, 1H), 3.32 – 3.26 (m, 1H), 3.17 – 3.09 (m, 1H), 2.86 – 2.78 (m, 4H), 2.05 (s, 3H), 1.68 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 203.1, 168.5, 166.4, 148.7, 144.5, 130.1, 129.8, 129.4, 126.1, 117.3, 112.7, 68.5, 52.1, 48.5, 40.3, 29.4, 23.7, 23.2.

HRMS (ESI) [M+H]⁺: calculated for C₂₂H₂₇N₂O₄: 383.1971, found 383.1974.

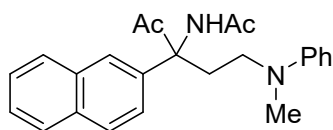


N-(3-([1,1'-Biphenyl]-4-yl)-1-(methyl(phenyl)amino)-4-oxopentan-3-yl)acetamide (4l). White solid (66.4 mg, 83% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.54 – 7.50 (m, 4H), 7.48 (s, 1H), 7.40 – 7.35 (m, 4H), 7.30 (t, *J* = 7.3 Hz, 1H), 7.24 – 7.20 (m, 2H), 6.71 (t, *J* = 7.3 Hz, 1H), 6.67 (d, *J* = 8.2 Hz, 2H), 3.48 – 3.42 (m, 1H), 3.33 – 3.27 (m, 1H), 3.17 – 3.10 (m, 1H), 2.83 – 2.75 (m, 4H), 2.03 (s, 3H), 1.73 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 203.8, 168.6, 148.9, 140.8, 140.2, 138.2, 129.3, 128.7, 127.5, 127.4, 127.0, 126.4, 117.1, 112.6, 68.4, 48.5, 40.0, 29.4, 23.8, 23.2.

HRMS (ESI) [M+H]⁺: calculated for C₂₆H₂₉N₂O₂: 401.2229, found 401.2234.



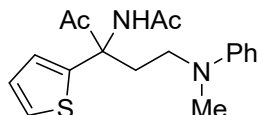
N-(1-(Methyl(phenyl)amino)-3-(naphthalen-2-yl)-4-oxopentan-3-yl)acetamide (4m). White solid (58.5 mg, 78% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.86 – 7.77 (m, 3H), 7.74 (d, *J* = 8.0 Hz, 1H), 7.49 – 7.43 (m, 2H),

7.42 – 7.35 (m, 2H), 7.25 – 7.20 (m, 2H), 6.75 – 6.66 (m, 3H), 3.64 – 3.56 (m, 1H), 3.43 – 3.35 (m, 1H), 3.29 – 3.21 (m, 1H), 2.89 (s, 3H), 2.59 – 2.50 (m, 1H), 1.91 (s, 3H), 1.75 (s, 3H).

^{13}C NMR (126 MHz, CDCl_3) δ 207.1, 168.1, 148.9, 134.5, 134.2, 130.6, 129.6, 129.3, 126.41, 126.37, 125.3, 125.1, 123.2, 116.9, 112.6, 68.7, 47.9, 39.2, 29.5, 23.9, 23.6.

HRMS (ESI) $[\text{M}+\text{Na}]^+$: calculated for $\text{C}_{24}\text{H}_{26}\text{N}_2\text{O}_2\text{Na}$: 397.1892, found 397.1894.

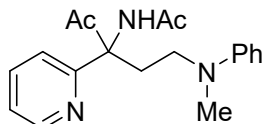


***N*-(1-(Methyl(phenyl)amino)-4-oxo-3-(thiophen-2-yl)pentan-3-yl)acetamide (4n)**. Yellow oil (60.1 mg, 91% yield).

^1H NMR (500 MHz, CDCl_3) δ 7.45 (s, 1H), 7.28 – 7.22 (m, 3H), 7.03 – 7.00 (m, 1H), 6.98 – 6.95 (m, 1H), 6.74 (t, $J = 7.3$ Hz, 1H), 6.69 (d, $J = 8.1$ Hz, 2H), 3.49 – 3.43 (m, 1H), 3.32 – 3.26 (m, 1H), 3.17 – 3.10 (m, 1H), 2.86–2.79 (m, 4H), 2.06 (s, 3H), 1.83 (s, 3H).

^{13}C NMR (126 MHz, CDCl_3) δ 202.4, 168.9, 148.8, 144.3, 129.3, 127.2, 125.5, 125.3, 117.2, 112.7, 66.8, 48.5, 40.1, 31.5, 23.8, 22.6.

HRMS (ESI) $[\text{M}+\text{H}]^+$: calculated for $\text{C}_{18}\text{H}_{23}\text{N}_2\text{O}_2\text{S}$: 331.1480, found 331.1484.

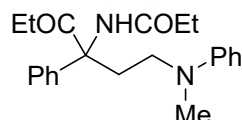


***N*-(1-(Methyl(phenyl)amino)-4-oxo-3-(pyridin-2-yl)pentan-3-yl)acetamide (4o)**. Brown oil (46.2 mg, 71% yield).

^1H NMR (500 MHz, CDCl_3) δ 8.58 (d, $J = 4.8$ Hz, 1H), 8.43 (s, 1H), 7.75 – 7.68 (m, 1H), 7.34 – 7.27 (m, 2H), 7.15 – 7.09 (m, 2H), 6.63 (t, $J = 7.2$ Hz, 1H), 6.42 (d, $J = 8.3$ Hz, 2H), 3.16 – 3.08 (m, 1H), 2.96 – 2.87 (m, 2H), 2.71 (s, 3H), 2.62 – 2.55 (m, 1H), 2.18 (s, 3H), 1.89 (s, 3H).

^{13}C NMR (126 MHz, CDCl_3) δ 204.8, 169.2, 154.8, 148.8, 148.3, 137.5, 129.0, 123.2, 120.8, 116.2, 112.2, 69.6, 47.7, 38.2, 30.6, 24.0, 23.3.

HRMS (ESI) $[\text{M}+\text{H}]^+$: calculated for $\text{C}_{19}\text{H}_{24}\text{N}_3\text{O}_2$: 326.1869, found 326.1872.

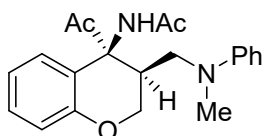


***N*-(1-(Methyl(phenyl)amino)-4-oxo-3-phenylhexan-3-yl)propionamide (8)**. Brown oil (52.8 mg, 75% yield).

^1H NMR (500 MHz, CDCl_3) δ 7.52 (s, 1H), 7.36 – 7.30 (m, 4H), 7.28 – 7.21 (m, 3H), 6.73 (t, $J = 7.3$ Hz, 1H), 6.68 (d, $J = 8.1$ Hz, 2H), 3.45 – 3.38 (m, 1H), 3.34 – 3.28 (m, 1H), 3.16 – 3.08 (m, 1H), 2.83 (s, 3H), 2.80 – 2.73 (m, 1H), 2.35 – 2.24 (m, 2H), 2.14 – 2.01 (m, 2H), 1.17 (t, $J = 7.6$ Hz, 3H), 0.75 (t, $J = 7.3$ Hz, 3H).

^{13}C NMR (126 MHz, CDCl_3) δ 207.6, 172.0, 148.9, 139.7, 129.2, 128.8, 127.8, 125.9, 116.9, 112.5, 68.1, 48.6, 39.7, 30.1, 29.2, 28.5, 9.7, 8.4.

HRMS (ESI) $[\text{M}+\text{H}]^+$: calculated for $\text{C}_{22}\text{H}_{29}\text{N}_2\text{O}_2$: 353.2229, found 353.2233.



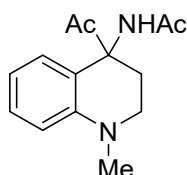
***N*-((3*R**,4*S**)-4-Acetyl-3-((methyl(phenyl)amino)methyl)chroman-4-yl)acetamide ((+/-)-11).**

Brown oil (59.9 mg, 85% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.36 (s, 1H), 7.27 – 7.19 (m, 3H), 6.95 – 6.86 (m, 3H), 6.75 (t, *J* = 7.3 Hz, 1H), 6.64 (d, *J* = 8.0 Hz, 2H), 4.69 (t, *J* = 10.2 Hz, 1H), 4.31 (dd, *J* = 10.5, 4.0 Hz, 1H), 3.29 – 3.26 (m, 2H), 3.13 – 3.07 (m, 1H), 2.89 (s, 3H), 2.02 (s, 3H), 1.80 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 204.1, 169.4, 155.7, 148.6, 129.9, 129.3, 126.6, 121.3, 120.0, 117.8, 117.4, 112.7, 65.5, 64.4, 50.6, 40.4, 38.4, 23.9, 23.7.

HRMS (ESI) [M+H]⁺: calculated for C₂₁H₂₅N₂O₃: 353.1865, found 353.1870.

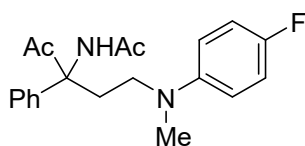


***N*-(4-Acetyl-1-methyl-1,2,3,4-tetrahydroquinolin-4-yl)acetamide (13).** Yellow oil (11.4 mg, 23% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.23 – 7.18 (m, 1H), 6.95 (dd, *J* = 7.8, 1.6 Hz, 1H), 6.68 (d, *J* = 8.3 Hz, 1H), 6.64 (t, *J* = 7.5 Hz, 1H), 6.23 (s, 1H), 3.42 – 3.35 (m, 1H), 3.33 – 3.28 (m, 1H), 2.94 (s, 3H), 2.66 – 2.61 (m, 1H), 2.54 – 2.48 (m, 1H), 2.13 (s, 3H), 2.00 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 204.8, 169.2, 147.2, 129.9, 126.9, 119.2, 116.7, 112.2, 64.3, 47.4, 39.2, 30.1, 25.7, 23.7.

HRMS (ESI) [M+Na]⁺: calculated for C₁₄H₁₈N₂O₂Na: 269.1266, found 269.1269.



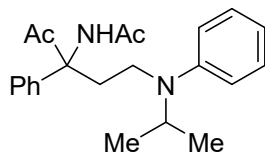
***N*-(1-((4-Fluorophenyl)(methyl)amino)-4-oxo-3-phenylpentan-3-yl)acetamide (6a).** Yellow oil (56.7 mg, 82% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.46 (s, 1H), 7.37 – 7.31 (m, 4H), 7.31 – 7.26 (m, 1H), 6.95 (t, *J* = 8.7 Hz, 2H), 6.64 – 6.58 (m, 2H), 3.43 – 3.36 (m, 1H), 3.30 – 3.23 (m, 1H), 3.12 – 3.04 (m, 1H), 2.78 (s, 3H), 2.77 – 2.69 (m, 1H), 2.04 (s, 3H), 1.71 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 203.9, 168.5, 155.7 (d, *J*_{C-F} = 236.1 Hz), 145.6, 139.2, 128.9, 128.0, 126.0, 115.7 (d, *J*_{C-F} = 21.9 Hz), 114.0 (d, *J*_{C-F} = 7.4 Hz), 68.5, 49.2, 40.5, 29.2, 23.8, 23.2.

¹⁹F NMR (471 MHz, CDCl₃) δ -128.2.

HRMS (ESI) [M+H]⁺: calculated for C₂₀H₂₄N₂O₂F: 343.1816, found 343.1825.

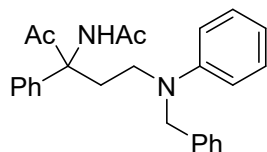


***N*-(1-(*iso*-Propyl(phenyl)amino)-4-oxo-3-phenylpentan-3-yl)acetamide (6b).** Yellow oil (61.4 mg, 87% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.51 (s, 1H), 7.34 – 7.29 (m, 4H), 7.28 – 7.21 (m, 3H), 6.82 (d, *J* = 8.2 Hz, 2H), 6.77 (t, *J* = 7.2 Hz, 1H), 3.98 – 3.90 (m, 1H), 3.23 – 3.16 (m, 2H), 2.99 – 2.91 (m, 1H), 2.65 – 2.56 (m, 1H), 2.04 (s, 3H), 1.79 (s, 3H), 1.17 (d, *J* = 6.7 Hz, 3H), 1.05 (d, *J* = 6.6 Hz, 3H).

^{13}C NMR (126 MHz, CDCl_3) δ 205.0, 168.3, 148.9, 139.5, 129.2, 128.8, 127.9, 126.0, 118.2, 116.2, 68.8, 51.8, 38.5, 29.8, 23.8, 23.5, 20.5, 18.8.

HRM (ESI) $[\text{M}+\text{H}]^+$: calculated for $\text{C}_{22}\text{H}_{29}\text{N}_2\text{O}_2$: 353.2224, found 353.2227.

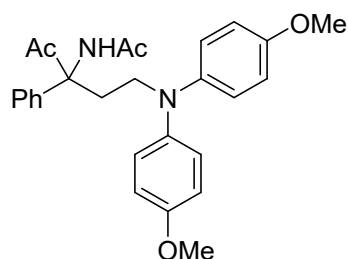


N-(1-(benzyl(phenyl)amino)-4-oxo-3-phenylpentan-3-yl)acetamide (**6c**). Yellow oil (71.3 mg, 89% yield).

^1H NMR (500 MHz, CDCl_3) δ 7.39 – 7.27 (m, 8H), 7.26 – 7.19 (m, 5H), 6.77 – 6.71 (m, 3H), 4.50 – 4.34 (m, 2H), 3.56 – 3.49 (m, 1H), 3.40 – 3.33 (m, 1H), 3.22 – 3.14 (m, 1H), 2.84 – 2.76 (m, 1H), 1.97 (s, 3H), 1.75 (s, 3H).

^{13}C NMR (126 MHz, CDCl_3) δ 204.5, 168.5, 147.9, 139.2, 138.4, 129.4, 128.9, 128.5, 128.0, 126.9 (d, $J = 4.6$ Hz), 125.9, 117.2, 113.1, 68.6, 55.2, 46.2, 28.8, 23.7, 23.2.

HRMS (ESI) $[\text{M}+\text{H}]^+$: calculated for $\text{C}_{26}\text{H}_{29}\text{N}_2\text{O}_2$: 401.2224, found 401.2233.

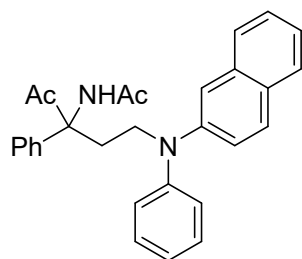


N-(1-(Bis(4-methoxyphenyl)amino)-4-oxo-3-phenylpentan-3-yl)acetamide (**6d**). Yellow oil (78.6 mg, 88% yield).

^1H NMR (500 MHz, CDCl_3) δ 7.43 (s, 1H), 7.35 – 7.29 (m, 4H), 7.29 – 7.24 (m, 1H), 6.92 – 6.79 (m, 8H), 3.77 (s, 6H), 3.74 – 3.66 (m, 1H), 3.46 – 3.38 (m, 1H), 3.37 – 3.28 (m, 1H), 2.72 – 2.64 (m, 1H), 1.98 (s, 3H), 1.74 (s, 3H).

^{13}C NMR (126 MHz, CDCl_3) δ 204.9, 168.5, 154.6, 142.5, 139.1, 128.8, 127.9, 125.9, 122.3, 114.6, 68.7, 55.5, 48.7, 29.0, 23.6, 23.3.

HRM (ESI) $[\text{M}+\text{Na}]^+$: calculated for $\text{C}_{27}\text{H}_{30}\text{N}_2\text{O}_4\text{Na}$: 469.2098, found 469.2107.

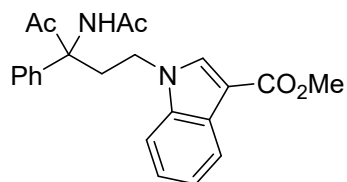


N-(1-(Naphthalen-2-yl(phenyl)amino)-4-oxo-3-phenylpentan-3-yl)acetamide (**6e**). Yellow oil (73.4 mg, 74% yield).

^1H NMR (500 MHz, CDCl_3) δ 7.75 (d, $J = 8.1$ Hz, 1H), 7.70 (d, $J = 8.9$ Hz, 2H), 7.46 – 7.41 (m, 2H), 7.37 – 7.29 (m, 9H), 7.18 (dd, $J = 8.9, 2.3$ Hz, 1H), 7.09 (d, $J = 7.7$ Hz, 2H), 7.03 (t, $J = 7.4$ Hz, 1H), 3.94 – 3.87 (m, 1H), 3.69 – 3.55 (m, 2H), 2.82 – 2.74 (m, 1H), 2.04 (s, 3H), 1.80 (s, 3H).

^{13}C NMR (126 MHz, CDCl_3) δ 204.9, 168.6, 148.1, 145.3, 138.9, 134.5, 129.4, 129.2, 128.9, 128.8, 128.0, 127.4, 126.8, 126.3, 125.9, 123.9, 122.2, 121.7, 115.3, 68.8, 48.4, 29.0, 23.7, 23.3.

HRMS (ESI) [M+Na]⁺: calculated for C₂₉H₂₈N₂O₂Na: 459.2043, found 459.2052.

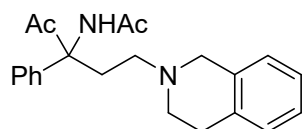


Methyl 1-(3-acetamido-4-oxo-3-phenylpentyl)-1H-indole-3-carboxylate (6f). Yellow oil (52.6 mg, 67% yield).

¹H NMR (500 MHz, CDCl₃) δ 8.18 – 8.14 (m, 1H), 7.63 (s, 1H), 7.39 – 7.34 (m, 2H), 7.33 – 7.25 (m, 6H), 7.06 (s, 1H), 4.23 – 4.15 (m, 1H), 4.01 – 3.93 (m, 1H), 3.90 (s, 3H), 3.56 – 3.48 (m, 1H), 2.93 – 2.85 (m, 1H), 1.88 (s, 3H), 1.78 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 203.8, 169.2, 165.2, 137.6, 136.1, 134.2, 129.2, 128.4, 126.7, 125.6, 123.1, 122.1, 121.9, 109.8, 107.3, 68.6, 50.9, 42.7, 31.7, 23.3, 23.1.

HRMS (ESI) [M+H]⁺: calculated for C₂₃H₂₄N₂O₄Na: 415.1628, found 415.1637.

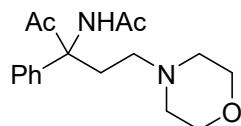


N-(1-(3,4-dihydroisoquinolin-2(1H)-yl)-4-oxo-3-phenylpentan-3-yl)acetamide (6g). Yellow oil (53.3 mg, 76% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.49 (s, 1H), 7.38 – 7.27 (m, 5H), 7.07 (t, *J* = 7.8 Hz, 1H), 6.96 (d, *J* = 7.2 Hz, 1H), 6.64 – 6.57 (m, 2H), 3.38 – 3.26 (m, 2H), 3.23 – 3.17 (m, 1H), 3.12 – 3.04 (m, 2H), 2.87 – 2.65 (m, 3H), 2.04 (s, 3H), 1.98 – 1.83 (m, 2H), 1.81 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 204.3, 168.5, 145.1, 139.4, 129.3, 128.8, 128.0, 127.1, 126.0, 123.4, 116.5, 110.8, 68.6, 50.3, 47.4, 29.0, 27.7, 23.8, 23.3, 21.7.

HRMS (ESI) [M+H]⁺: calculated for C₂₂H₂₇N₂O₂: 351.2067, found 351.2077.

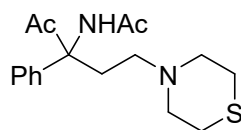


N-(1-(morpholino-4-oxo-3-phenylpentan-3-yl)acetamide (6h). Yellow oil (55.4 mg, 91% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.65 (s, 1H), 7.37 – 7.30 (m, 4H), 7.28 – 7.24 (m, 1H), 3.70 – 3.58 (m, 4H), 3.04 – 2.97 (m, 1H), 2.59 – 2.50 (m, 3H), 2.34 – 2.22 (m, 4H), 1.99 (s, 3H), 1.94 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 204.5, 168.4, 139.2, 128.7, 127.8, 126.1, 69.0, 66.6, 53.8, 29.0, 24.1, 23.7.

HRMS (ESI) [M+H]⁺: calculated for C₁₇H₂₅N₂O₃: 305.1865, found 305.1873.

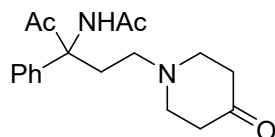


N-(4-oxo-3-phenyl-1-thiomorpholinopentan-3-yl)acetamide (6i). Yellow oil (44.9 mg, 70% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.63 (s, 1H), 7.37 – 7.30 (m, 4H), 7.29 – 7.24 (m, 1H), 3.04 – 2.98 (m, 1H), 2.82 – 2.75 (m, 2H), 2.66 – 2.50 (m, 7H), 2.39 – 2.32 (m, 1H), 2.27 – 2.20 (m, 1H), 2.00 (s, 3H), 1.93 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 204.5, 168.4, 139.4, 128.8, 127.8, 126.2, 68.9, 55.2, 54.0, 29.2, 27.6, 24.2, 23.8.

HRMS (ESI) [M+H]⁺: calculated for C₁₇H₂₅N₂O₂S: 321.1637, found 321.1639.

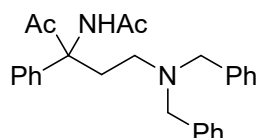


N-(4-Oxo-1-(4-oxopiperidin-1-yl)-3-phenylpentan-3-yl)acetamide (6j). Yellow oil (41.2 mg, 65% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.54 (s, 1H), 7.39 – 7.33 (m, 4H), 7.31 – 7.27 (m, 1H), 3.21 – 3.14 (m, 1H), 2.87 – 2.80 (m, 2H), 2.69 – 2.57 (m, 3H), 2.47 – 2.33 (m, 6H), 2.01 (s, 3H), 1.97 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 208.5, 204.6, 168.5, 139.4, 129.0, 128.1, 126.2, 68.9, 53.5, 52.3, 40.8, 29.8, 24.0, 23.9.

HRMS (ESI) [M+H]⁺: calculated for C₁₈H₂₅N₂O₃: 317.1865, found 317.1869.

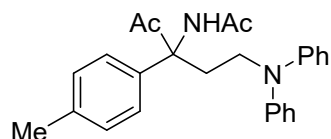


N-(1-(Dibenzylamino)-4-oxo-3-phenylpentan-3-yl)acetamide (6k). Yellow oil (46.5 mg, 56% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.35 – 7.32 (m, 8H), 7.31 – 7.27 (m, 3H), 7.26 – 7.23 (m, 5H), 3.78 (d, *J* = 13.6 Hz, 2H), 3.39 (d, *J* = 13.6 Hz, 2H), 3.14 – 3.06 (m, 1H), 2.59 – 2.51 (m, 1H), 2.42 – 2.31 (m, 2H), 1.77 (s, 3H), 1.75 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 204.8, 168.4, 139.2, 139.0, 128.9, 128.7, 128.3, 127.7, 127.1, 126.1, 68.9, 58.8, 48.3, 28.7, 23.5, 23.4.

HRMS (ESI) [M+H]⁺: calculated for C₂₇H₃₁N₂O₂: 415.2386, found 415.2389.

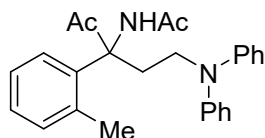


N-(1-(Diphenylamino)-4-oxo-3-(p-tolyl)pentan-3-yl)acetamide (6l). Yellow oil (74.5 mg, 93% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.36 (s, 1H), 7.28 (t, *J* = 7.9 Hz, 4H), 7.22 – 7.13 (m, 4H), 7.05 – 6.92 (m, 6H), 3.84 – 3.73 (m, 1H), 3.55 – 3.44 (m, 2H), 2.73 – 2.62 (m, 1H), 2.32 (s, 3H), 2.00 (s, 3H), 1.77 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 205.0, 168.5, 147.9, 137.8, 135.9, 129.6, 129.3, 125.8, 121.6, 121.0, 68.5, 48.1, 29.0, 23.6, 23.1, 20.9.

HRMS (ESI) [M+Na]⁺: calculated for C₂₆H₂₈N₂O₂Na: 423.2043, found 423.2052.

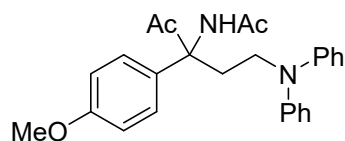


***N*-(1-(Diphenylamino)-4-oxo-3-(*o*-tolyl)pentan-3-yl)acetamide (6m).** Yellow oil (70.5 mg, 88% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.61 (d, *J* = 7.8 Hz, 1H), 7.30 – 7.23 (m, 5H), 7.22 – 7.15 (m, 2H), 7.09 (d, *J* = 7.3 Hz, 1H), 7.01 (d, *J* = 7.8 Hz, 4H), 6.96 (t, *J* = 7.3 Hz, 2H), 3.79 – 3.66 (m, 2H), 3.53 – 3.45 (m, 1H), 2.53 – 2.45 (m, 1H), 2.11 (s, 3H), 2.00 (s, 3H), 1.77 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 205.9, 167.7, 147.8, 136.6, 135.5, 132.5, 129.4, 128.2, 127.6, 126.5, 121.6, 120.9, 68.8, 47.6, 29.7, 23.6, 23.4, 20.4.

HRMS (ESI) [M+Na]⁺: calculated for C₂₆H₂₈N₂O₂Na: 423.2050, found 423.2043.

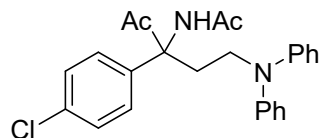


***N*-(1-(Diphenylamino)-3-(4-methoxyphenyl)-4-oxopentan-3-yl)acetamide (6n).** Yellow oil (74.2 mg, 89% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.36 (s, 1H), 7.27 (t, *J* = 7.9 Hz, 4H), 7.22 (d, *J* = 8.8 Hz, 2H), 7.04 – 6.94 (m, 6H), 6.86 (d, *J* = 8.8 Hz, 2H), 3.83 – 3.72 (m, 4H), 3.52 – 3.43 (m, 2H), 2.71 – 2.60 (m, 1H), 1.99 (s, 3H), 1.76 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 205.0, 168.6, 159.2, 147.9, 130.8, 129.3, 127.1, 121.6, 121.0, 114.2, 68.2, 55.1, 48.1, 29.0, 23.6, 23.0.

HRMS (ESI) [M+Na]⁺: calculated for C₂₆H₂₈N₂O₃Na: 439.1992, found 439.2001.

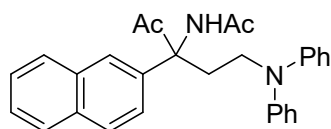


***N*-(3-(4-Chlorophenyl)-1-(diphenylamino)-4-oxopentan-3-yl)acetamide (6o).** Yellow oil (80.0 mg, 95% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.40 (s, 1H), 7.32 – 7.23 (m, 8H), 7.02 – 6.94 (m, 6H), 3.83 – 3.73 (m, 1H), 3.52 – 3.42 (m, 2H), 2.70 – 2.60 (m, 1H), 1.99 (s, 3H), 1.76 (s, 3H).

¹³C NMR (126 MHz, CDCl₃) δ 204.3, 168.5, 147.9, 137.7, 134.0, 129.3, 129.0, 127.4, 121.7, 121.0, 68.3, 48.0, 28.9, 23.6, 23.1.

HRMS (ESI) [M+Na]⁺: calculated for C₂₅H₂₅N₂O₂NaCl: 443.1497, found 443.1503.



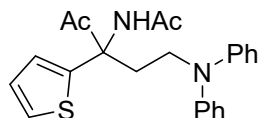
***N*-(1-(Diphenylamino)-3-(naphthalen-2-yl)-4-oxopentan-3-yl)acetamide (6p).** Yellow oil (77.8 mg, 89% yield).

¹H NMR (500 MHz, CDCl₃) δ 7.86 (t, *J* = 7.1 Hz, 2H), 7.82 (d, *J* = 8.2 Hz, 1H), 7.73 (d, *J* = 8.4 Hz, 1H), 7.49 (t, *J* = 7.8 Hz, 1H), 7.46 – 7.38 (m, 3H), 7.32 – 7.26 (m, 4H), 7.06 – 7.01 (m, 4H),

6.97 (t, $J = 7.3$ Hz, 2H), 3.91 – 3.80 (m, 2H), 3.65 – 3.56 (m, 1H), 2.62 – 2.54 (m, 1H), 1.92 (s, 3H), 1.72 (s, 3H).

^{13}C NMR (126 MHz, CDCl_3) δ 207.6, 168.1, 147.7, 134.5, 134.0, 130.5, 129.6, 129.4, 126.5, 126.4, 125.4, 125.2, 123.1, 121.6, 120.8, 68.8, 47.6, 30.0, 29.6, 23.8, 23.6.

HRMS (ESI) $[\text{M}+\text{Na}]^+$: calculated for $\text{C}_{29}\text{H}_{28}\text{N}_2\text{O}_2\text{Na}$: 459.2043, found 459.2052.



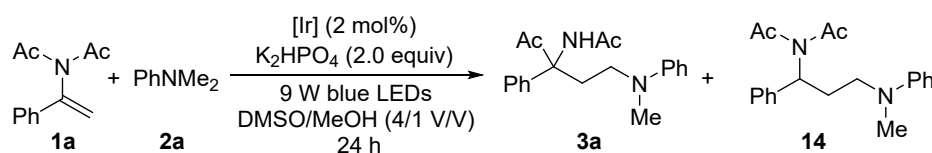
***N*-(1-(Diphenylamino)-4-oxo-3-(thiophen-2-yl)pentan-3-yl)acetamide (6q)**. Yellow oil (63.6 mg, 81% yield).

^1H NMR (500 MHz, CDCl_3) δ 7.30 – 7.22 (m, 6H), 7.02 – 6.93 (m, 8H), 3.82 – 3.73 (m, 1H), 3.51 – 3.43 (m, 2H), 2.77 – 2.68 (m, 1H), 2.00 (s, 3H), 1.87 (s, 3H).

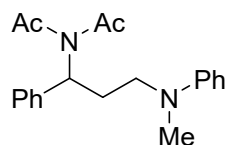
^{13}C NMR (126 MHz, CDCl_3) δ 203.5, 169.0, 148.0, 143.9, 129.4, 127.3, 125.6, 125.4, 121.7, 121.1, 67.0, 48.0, 31.0, 23.7, 22.7.

HRMS (ESI) $[\text{M}+\text{H}]^+$: calculated for $\text{C}_{23}\text{H}_{24}\text{N}_2\text{O}_2\text{NaS}$: 415.1451, found 415.1460.

4. Mechanism study



To an oven dried transparent 10 mL Schlenk tube equipped with stirring bar, $\text{Ir}[\text{dF}(\text{CF}_3)\text{ppy}]_2(\text{dtbbpy})\text{PF}_6$ (4.5 mg, 0.004 mmol, 0.02 equiv), K_2HPO_4 (69.6 mg, 0.4 mmol), *N*-acetyl-*N*-(1-phenylvinyl)acetamide **1a** (40.6 mg, 0.2 mmol), and *N,N*-dimethylaniline **2a** (48.4 mg, 0.4 mmol) were added. The tube was evacuated and filled with nitrogen for 3 times. The degassed DMSO (4 mL) and dried methanol (1 mL) were added via syringe. The tube was irradiated with 9 W blue LEDs strip spiraled within a bowel for 24 h (cooling with a fan). After the reaction was completed, the reaction solution was quenched by the addition of water (5 mL) and extracted with EtOAc (5×10 mL). The combined organic layer was washed with brine, dried over Na_2SO_4 , filtered, and solvent was evaporated to obtain crude product. Flash chromatography over silica gel afforded the product **3a** and **14**.

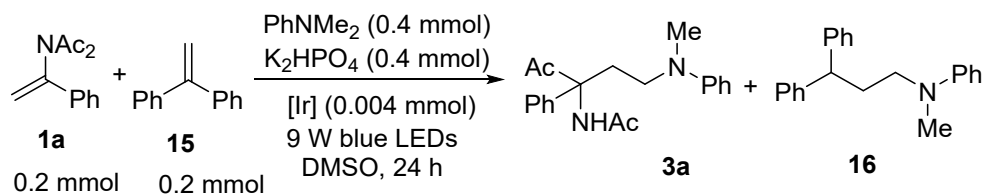


***N*-Acetyl-*N*-(3-(methyl(phenyl)amino)-1-phenylpropyl)acetamide (14)**. Yellow oil (9.8 mg, 15% yield).

^1H NMR (500 MHz, CDCl_3) δ 7.37 – 7.31 (m, 2H), 7.31 – 7.21 (m, 5H), 6.76 – 6.69 (m, 3H), 5.63 – 5.55 (m, 1H), 3.48 – 3.31 (m, 2H), 2.93 (s, 3H), 2.67 – 2.56 (m, 1H), 2.49 – 2.40 (m, 1H), 2.28 (s, 6H).

^{13}C NMR (126 MHz, CDCl_3) δ 174.4, 149.2, 139.6, 129.3, 128.7, 127.5, 126.8, 117.1, 112.9, 56.0, 50.2, 38.7, 28.8, 26.8.

HRMS (ESI) $[\text{M}+\text{H}]^+$: calculated for $\text{C}_{20}\text{H}_{25}\text{N}_2\text{O}_2$: 325.1916, found 325.1917.



To an oven dried transparent 10 mL Schlenk tube equipped with stirring bar, Ir[dF(CF₃)ppy]₂(dtbbpy)PF₆ (4.5 mg, 0.004 mmol), K₂HPO₄ (69.6 mg, 0.4 mmol), *N*-acetyl-*N*-(1-phenylvinyl)acetamide **1a** (40.6 mg, 0.2 mmol), 1,1-diphenylethylene **15** (36 mg, 0.2 mmol), and *N,N*-dimethylaniline **2a** (48.4 mg, 0.4 mmol) were added. The tube was evacuated and filled with nitrogen for 3 times. The degassed DMSO (4 mL) was added via syringe. The tube was irradiated with 9 W blue LEDs strip spiraled within a bowel for 24 h (cooling with a fan). After the reaction was completed, the reaction solution was quenched by the addition of water (5 mL) and extracted with EtOAc (5 × 10 mL). The combined organic layer was washed with brine, dried over Na₂SO₄, filtered, and solvent was evaporated to obtain crude product. Flash chromatography over silica gel afforded the product **3a** (37.5 mg, 58% yield) and **16** (20.5 mg, 34% yield).

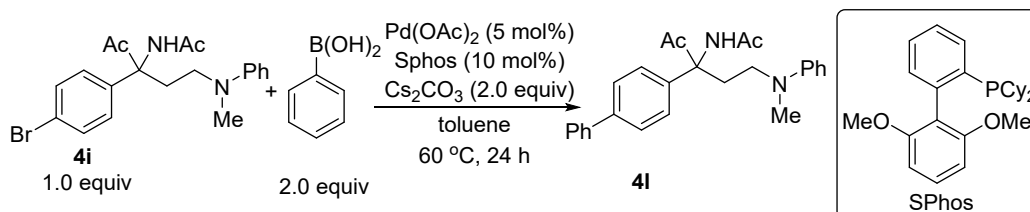
N-(3,3-Diphenylpropyl)-*N*-methylaniline **16**.

¹H NMR (500 MHz, CDCl₃) δ 7.32 – 7.26 (m, 8H), 7.22 – 7.17 (m, 4H), 6.67 (t, J = 7.2 Hz, 1H), 6.58 (d, J = 8.2 Hz, 2H), 3.94 (t, J = 7.8 Hz, 1H), 3.30 – 3.25 (m, 2H), 2.88 (s, 3H), 2.33 (q, J = 7.7 Hz, 2H).

¹³C NMR (126 MHz, CDCl₃) δ 149.1, 144.5, 129.1, 128.5, 127.7, 126.3, 116.0, 112.2, 51.3, 49.0, 38.2, 32.0.

This compound has been reported in the published literature.⁴

5. Procedure of Suzuki reaction



To an oven dried transparent 10 mL Schlenk tube equipped with stirring bar, compound **4i** (77 mg, 0.2 mmol), phenylboronic acid (48.8 mg, 0.4 mmol), Cs₂CO₃ (130 mg, 0.4 mmol), Pd(OAc)₂ (1.7 mg, 5 mol%), and SPhos (8.2 mg, 10 mol%) were added. The tube was evacuated and filled with nitrogen for 3 times. Then anhydrous toluene (2.0 mL) was introduced and the mixture was stirred at 60 °C for 24 h. After completion of the reaction, the mixture was cooled down to room temperature and diluted with EtOAc (15.0 mL). The catalyst and inorganic base were filtered off using a short pad of silica gel. The filtrate was concentrated under reduced pressure. The residue was purified by column chromatography on a silica gel column using petroleum ether/EtOAc as the eluent to give the product **4l** (73.6 mg, 92% yield).

6. References

- [1] (a) L. Huai, L. Zhang, Z. Wang, H. Wu and Y. Fang, *Org. Chem. Front.*, 2023, **10**, 1245; (b) L. Huai, L. Zhang, Z. Wang and Y. Fang, *Org. Chem. Front.*, 2024, **11**, 2344.
- [2] (a) B. Han, Y. Li, Y. Yu and L. Gong, *Nat. Commun.*, 2019, **10**, 3804; (b) Y. Jia, K. Zhang, L.-Q. Lu, Y. Cheng and W.-J. Xiao, *ACS Catal.*, 2024, **14**, 13550; (c) C. Remeur, C. B. Kelly, N. R. Patel and G. A. Molander, *ACS Catal.*, 2017, **7**, 6065; (d) A. Ilic, B. R. Strücker, C. E. Johanson, S. Hainz, R. Lomoth and K. Wärnmark, *Chem. Sci.*, 2024, **15**, 12077; (e) Y. Zhao, L. D. Bruce, J. Jin, B. Xia and P. W. H. Chan, *Green Chem.*, 2020, **22**, 5296; (f) J. Li, L. Carli, S. H. Kyne and P. W. H. Chan, *Adv. Synth. Catal.*, 2023, **365**, 2422.
- [3] J. T. Reeves, Z. Tan, Z. S. Han, G. Li, Y. Zhang, Y. Xu, D. C. Reeves, N. C. Gonnella, S. Ma, H. Lee, B. Z. Lu and C. H. Senanayake, *Angew. Chem. Int. Ed.*, 2012, **51**, 1400.
- [4] N. A. Larionova, J. M. Onozabal, E. G. Smith and X. C. Cambeiro, A Photocatalytic Regioselective Direct Hydroaminoalkylation of Aryl-Substituted Alkenes with Amines, *Org. Lett.*, 2021, **23**, 5383.

7. X-ray crystal data for compounds 3q and 4j

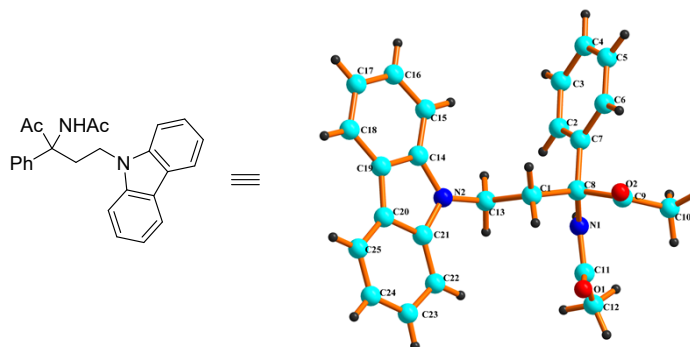


Table S1 Crystal data and structure refinement for 3p

Identification code	CCDC 2415011 (3p)
Empirical formula	C ₂₅ H ₂₄ N ₂ O ₂
Formula weight	384.4790
Temperature/K	273.15
Crystal system	monoclinic
Space group	P2 ₁ /c
a/Å	9.835(3)
b/Å	19.389(6)
c/Å	11.255(4)
α/°	90
β/°	102.090(9)
γ/°	90
Volume/Å ³	2098.8(11)
Z	42
ρ _{calc} /cm ³	1.2167
μ/mm ⁻¹	0.078
F(000)	816.4
Crystal size/mm ³	0.34×0.20×0.14
Radiation	MoKα (λ = 0.71073)
2θ range for data collection/°	4.2 to 50
Index ranges	-11 ≤ h ≤ 11, -23 ≤ k ≤ 23, -13 ≤ l ≤ 13
Reflections collected	46194
Independent reflections	3698 [R _{int} = 0.0801, R _{sigma} = 0.0369]
Data/restraints/parameters	3698/0/264
Goodness-of-fit on F ²	1.088
Final R indexes [I >= 2σ (I)]	R ₁ = 0.0668, wR ₂ = 0.1779
Final R indexes [all data]	R ₁ = 0.1026, wR ₂ = 0.2156
Largest diff. peak/hole / e Å ⁻³	0.46/-0.34

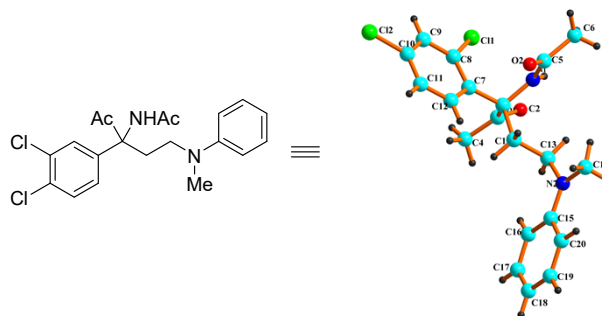


Table S2 Crystal data and structure refinement for 4j	
Identification code	CCDC 2415006 (4j)
Empirical formula	C ₂₀ H ₂₂ Cl ₂ N ₂ O ₂
Formula weight	393.3080
Temperature/K	273.15
Crystal system	monoclinic
Space group	Cc
a/Å	7.1592(8)
b/Å	14.4200(16)
c/Å	19.648(2)
α/°	90
β/°	93.554(3)
γ/°	90
Volume/Å ³	2024.5(4)
Z	1
ρ _{calc} /cm ³	1.2903
μ/mm ⁻¹	0.337
F(000)	825.5
Crystal size/mm ³	0.35×0.18×0.14
Radiation	MoKα (λ = 0.71073)
2θ range for data collection/°	4.16 to 50.84
Index ranges	-8 ≤ h ≤ 7, -17 ≤ k ≤ 17, -23 ≤ l ≤ 23
Reflections collected	16708
Independent reflections	3599 [R _{int} = 0.0497, R _{sigma} = 0.0417]
Data/restraints/parameters	3599/2/238
Goodness-of-fit on F ²	1.060
Final R indexes [I >= 2σ (I)]	R ₁ = 0.0368, wR ₂ = 0.0887
Final R indexes [all data]	R ₁ = 0.0469, wR ₂ = 0.0971
Largest diff. peak/hole / e Å ⁻³	0.15/-0.28

8. NMR Spectra of New Compounds

