Supporting Information

TBHP-Mediated Photochemical Coupling/Cyclization of N-arylacrylamide via Thiyl Radical

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1. General Information

The reactions via general procedure was carried out under an atmosphere of argon unless otherwise noted. Column chromatography was performed using silica gel (200-300 mesh) or thin layer chromatography was performed using silica gel (GF254). ¹H NMR and ¹³C NMR spectra were recorded on Bruker-AVANCE-III-HD (400 and 100 MHz, respectively) and processed using either MestReNova. ¹H NMR chemical shifts are given in ppm with respect to the residual CDCl₃ peak (δ 7.26 ppm), residual DMSO- d_6 (δ 2.50 ppm), or an internal TMS standard (δ 0.00 ppm), ¹³C NMR shifts are given in ppm with respect to $CDCl_3$ (δ 77.00 ppm), DMSO- d_6 (δ 39.52 ppm). Mass spectra were measured on Agilent 5977 GC-MS instrument (EI). High-resolution mass spectra (ESI) were obtained with the Thermo Scientific LTQ Orbitrap XL mass spectrometer. The structures of known compounds were further corroborated by comparing their ¹H NMR, ¹³C NMR data and MS data with those in literature. Melting points were measured with a YUHUA X-5 melting point instrument and were uncorrected. Fluorescence quenching experiments were recorded with PTI-QM40 spectrophotometer. A commercially available blue LED (35W, HIPAR30, luminous flux is not less than 3200 lm) was purchased from Shenzhen Jing Feng Times Lighting Technology Co., Ltd as the reaction light source. All irradiation reactions were carried out in borosilicate glass vessel. The distance from the light source to the irradiation vessel is around 2-3 cm. Unless otherwise noted, all other reagents were obtained from commercial suppliers and used without further purification.

2. General Procedure for Photoinduced Thiolation/Cyclization Reac-

tion



General Procedure: A 20 mL reaction vessel was charged with photocatalyst 4CzIPN (3.2 mg, 0.004 mmol), *N*-arylacrylamide (1, 0.2 mmol, 1.0 equiv.), thiol (2, 0.28 mmol, 1.4 equiv.) and TBHP (70% in water, 42 μ L, 0.3 mmol, 1.5 equiv.) in 2.0 mL CH₂Cl₂ under Ar atmosphere. The resulting mixture was stirred for 24 – 48 h under irradiation with a 35 W blue LEDs at 40 °C. The reaction was monitored by TLC. The crude reaction mixture was quenched with saturated sodium carbonate and extracted with DCM (3×10 mL). The extracts were combined, dried over sodium sulfate, and filtered, and the volatiles were removed under reduced pressure. Column chromatography was performed using silica gel (200-300 mesh) or thin layer chromatography was performed using silica gel (GF254) to give product thionated oxindoles (3 or 4).



5 mmol scale reaction: A 20 mL reaction vessel was charged with photocatalyst 4CzIPN (20 mg, 0.025 mmol), *N*-methyl-*N*-phenylmethacrylamide **1a** (875 mg, 5.0 mmol, 1.0 equiv.), cyclohexanethiol **2a** (900 μ L, 7.0 mmol, 1.4 equiv.) or 2-mercaptoethan-1-ol **2k** (525 μ L, 7.0 mmol, 1.4 equiv.) and TBHP (70% in water, 1.1 mL, 7.5 mmol, 1.5 equiv.) in 50 mL CH₂Cl₂ or DMF under Ar atmosphere. The resulting mixture was stirred for 36 h or 48 h under irradiation with 2 × 35 W blue LEDs at 40 °C. The reaction was monitored by TLC. The crude reaction mixture was quenched with saturated sodium carbonate and extracted with DCM (3×30 mL). The extracts were combined, dried over sodium sulfate, and filtered, and the volatiles were removed under reduced pressure. Column chromatography was performed using silica gel (200-300 mesh) or thin layer chromatography was performed using silica gel (GF254) to give product corresponding oxindoles **3a** (912 mg, 63% yield) or **3k** (565 mg, 45% yield), respectively.

3. Mechanistic Studies

3.1 Stern-Volmer Quenching

Formulation solution: *N*-methyl-*N*-phenylmethacrylamide (**1a**, 87.5 mg) was dissolved in CH_2Cl_2 in a 5 mL volumetric flask to set the concentration to be 0.1 M. cyclohexanethiol (**2a**, 312.0 µL) was dissolved in CH_2Cl_2 in a 25 mL volumetric flask to set the concentration to be 0.1 M. TBHP (341.0 mg) was dissolved in CH_2Cl_2 in a 25 mL volumetric flask to set the concentration to be 0.1 M. Photocatalyst 4CzIPN (2.0 mg) was dissolved in CH_2Cl_2 in a 25 mL volumetric flask to set the concentration to be 0.1 M. Photocatalyst 4CzIPN (2.0 mg) was dissolved in CH_2Cl_2 in a 25 mL volumetric flask to set the concentration to be 0.1 M.

Experimental procedure: The resulting 0.1 mM solution (20 μ L) was added to cuvette to obtain different concentrations of catalyst solution. This solution was then diluted to a volume of 2.0 mL by adding CH₂Cl₂ to prepare a 1.0 μ M solution. The resulting mixture was sparged with argon for 3 minutes and then irradiated at 400 nm. Fluorescence emission spectra were recorded (3 trials per sample). Into this solution, 20.0 μ L of a *N*-methyl-*N*-phenylmethacrylamide **1a** solution was successively added and uniformly stirred, and the resulting mixture was bubbled with argon for 3 minutes and irradiated at 400 nm. Fluorescence emission spectra of 0 μ L, 20.0 μ L, 40.0 μ L, 60.0 μ L, 80.0 μ L 100.0 μ L fluorescence intensity. Follow this method and make changes to the amount to obtain the Stern–Volmer relationship in turn. The results were shown in the following figures.



Figure S1 Emission quenching of 4CzIPN with 1a in CH₂Cl₂



Figure S2 Emission quenching of 4CzIPN with 2a in CH₂Cl₂







Figure S4 Emission quenching of 4CzIPN with 1a + TBHP in CH₂Cl₂



Figure S5 Emission quenching of 4CzIPN with 2a + TBHP in CH₂Cl₂

3.2 Switch Light Experiments

Conducted the relationship of products with light on-off under standard conditions. Subsequent samples (each 20 μ L) taken at regular time intervals and determined by GC. The corresponding experimental results were constructed in Figure S6.



Figure S6 Plot of light on-off experiments

3.3 Radical Trapping Experiments

Radical trapping experiments of visible-light-mediated thiolation/cyclization of N-phenylmethacrylamide **1a** with thiol **2a** were performed under standard conditions. The corresponding experimental results were constructed in Figure S7.





Figure S7

3.4 Control Experiments

(a) The following reaction was carried out under standard conditions: A 20 mL reaction vessel was charged with photocatalyst 4CzIPN (3.2 mg, 0.004 mmol), N-arylacrylamide 1a (35 mg, 0.2 mmol, 1.0 equiv.), cyclohexanethiol 2a (35 µL, 0.28 mmol, 1.4 equiv.) and TBHP (70% in water, 42 µL, 0.3 mmol, 1.5 equiv.) in 2.0 mL CH₂Cl₂ under Ar atmosphere. The resulting mixture was stirred for 12 h under irradiation with a 35 W blue LEDs at 40 °C. After completion, the crude residues were analyzed by GC-MS. Cyclization product 3a and 1,2-dicyclohexyldisulfane 10 was detected by GC-MS.







(b) The following reaction was carried out under standard conditions: A 20 mL reaction vessel was charged with photocatalyst 4CzIPN (3.2 mg, 0.004 mmol), *N*-arylacrylamide **1a** (35 mg, 0.2 mmol, 1.0 equiv.), 1,2-dicyclohexyldisulfane **10** (64.4 mg, 0.28 mmol, 1.4 equiv.) and TBHP (70% in water, 42 μ L, 0.3 mmol, 1.5 equiv.) in 2.0 mL CH₂Cl₂ under Ar atmosphere. The resulting mixture was stirred for 24 h under irradiation with a 35 W blue LEDs at 40 °C. After completion, the crude residues were analyzed by GC-MS. Product **3a** was detected by GC-MS in 29% yield.



Figure S9

(c) The following reaction was carried out under standard conditions: A 20 mL reaction vessel was charged with photocatalyst 4CzIPN (3.2 mg, 0.004 mmol), *N*-arylacrylamide **1a** (35 mg, 0.2 mmol, 1.0 equiv.), 1,2-diphenyldisulfane **2r** (61.0 mg, 0.28 mmol, 1.4 equiv.) and TBHP (70% in water, 42 μ L, 0.3 mmol, 1.5 equiv.) in 2.0 mL CH₂Cl₂ under Ar atmosphere. The resulting mixture was stirred for 48 h under irradiation with a 35 W blue LEDs at 40 °C. After completion, the crude residues were analyzed by GC-MS. Product **3r** was detected by GC-MS in trace yield.



Figure S10

3.5 Checked the Reactivity of Thiophenols with N-arylacrylamide

The following reaction was carried out under standard conditions: A 20 mL reaction vessel was charged with photocatalyst (**PC**, 0.004 mmol), *N*-arylacrylamide (**1a**, 35.0 mg, 0.2 mmol, 1.0 equiv.), 4-chlorobenzenethiol (**2t**, 40.6 mg, 0.28 mmol, 1.4 equiv.) and TBHP (70% in water, 42 μ L, 0.3 mmol, 1.5 equiv.) in 2.0 mL CH₂Cl₂ under Ar atmosphere. The resulting mixture was stirred for 48 h under irradiation with a 35 W light source at 40 °C. The reaction was monitored by TLC. The yield was determined by GC analysis of the crude reaction mixture using dodecane as the internal standard.

Table S1. Investigating the reaction of the thiophenol with N-arylacrylamide

	a + CI + C	PC TBHP CH ₂ Ch ₂ , Ar, 40 °C, 48 h light source	
entry	РС	light source	yield (%)
1	4CzIPN	blue LEDs	23
2	[Ir]PF ₆	blue LEDs	20
3	<i>fac</i> -Ir(ppy) ₃	blue LEDs	trace
4	$Ru(bpy)_3(PF_6)_2$	blue LEDs	11
5	Eosin B	blue LEDs	21
6	Rose Bengal	blue LEDs	trace
7	4CzIPN	purple LEDs	13
8	4CzIPN	green LEDs	19
9	4CzIPN	yellow LEDs	trace

4. Late-Stage Derivation and Application

(a) Under argon atmosphere, to a stirred solution of the oxindole **3a** (57.8 mg, 0.2 mmol, 1.0 equiv.) in dry THF (3 mL) at 0 °C was added LiAlH₄ (32 mg, 0.8 mmol, 4.0 equiv.), and the mixture was stirred at 85 °C for 16 h.¹ The mixture was quenched by sat. aqueous NaHCO₃, and diluted by EtOAc. The organic phase was collected, and the aqueous phase was extracted with EtOAc. The combined organic layers were dried over anhydrous Na₂SO₄, filtered, and concentrated under reduced pressure. The crude product was purified by flash column chromatography (petroleum ether/ethyl acetate 40:1) to afford product **5** (47.3 mg, 86% yield) as colorless liquid.



(b) A mixture of oxindole **3a** (57.8 mg, 0.2 mmol, 1.0 equiv.), H_2O_2 (30% in water, 60 µL, 2.0 mmol, 10 equiv.) and Sc(OTf)₃ (20 mg, 0.04 mmol, 20 mol%) in DCM/EtOH (1.0 mL/1.0 mL)

was stirred at room temperature for 3 h.² The mixture was quenched by water, and diluted by DCM. The organic phase was collected, and the aqueous phase was extracted with DCM. The combined organic layers were dried over anhydrous Na_2SO_4 , filtered, and concentrated under reduced pressure. The crude product was purified by flash column chromatography (petroleum ether/ethyl acetate 2:1) to afford product **6** in 70% yield (42.7 mg, 1:1 *dr*) as colorless liquid.



(c) A mixture of oxindole **3a** (57.8 mg, 0.2 mmol, 1.0 equiv.), H_2O_2 (30% in water, 60 µL, 2.0 mmol, 10 equiv.) and Sc(OTf)₃ (20 mg, 0.04 mmol, 20 mol%) in DCM/EtOH (1.0 mL/1.0 mL) was stirred at room temperature for 24 h.² The mixture was quenched by water, and diluted by DCM. The organic phase was collected, and the aqueous phase was extracted with DCM. The combined organic layers were dried over anhydrous Na₂SO₄, filtered, and concentrated under reduced pressure. The crude product was purified by flash column chromatography (petroleum ether/ethyl acetate 2:1) to afford product **6** in 63% yield (40.4 mg) as light yellow liquid.



(d) To a stirred solution of the alcohol **3k** (47.4 mg, 0.2 mmol, 1.0 equiv.) in dry THF (4 mL) at 0 °C was added LiAlH₄ (32 mg, 0.8 mmol, 4.0 equiv.), and the mixture was stirred at this temperature for 2 h.³ The mixture was quenched by sat. aqueous NaHCO₃, and diluted by EtOAc. The organic phase was collected, and the aqueous phase was extracted with EtOAc. The combined organic layers were dried over anhydrous Na₂SO₄, filtered, and concentrated under reduced pressure. The crude product was purified by flash column chromatography (petroleum ether/ethyl acetate 50:1) to afford cyclization product **8** (38.1 mg, 81% yield) as colorless liquid.





(e) Under argon atmosphere, to a stirred solution of the alcohol **3k** (47.4 mg, 0.2 mmol, 1.0 equiv.) in CH₂Cl₂ (3 mL) at room temperature was added Dess-Martin periodinane (DMP) (127 mg, 0.3 mmol, 1.5 equiv.), and the mixture was stirred for 6 h.³ The mixture was quenched by sat. aqueous Na₂S₂O₃ and sat. aqueous NaHCO₃ successively, and diluted by CH₂Cl₂. The organic phase was collected, and the aqueous phase was extracted with CH₂Cl₂. The combined organic layers were dried over anhydrous Na₂SO₄, filtered, and concentrated under reduced pressure. The crude product was purified by flash column chromatography (petroleum ether/ethyl acetate 5:1) to afford product **9** (36.3 mg, 73% yield) as a colorless liquid.



5. Characterization Data of all Products

3-((cyclohexylthio)methyl)-1,3-dimethylindolin-2-one (3a)



The desired compound **3a** was synthesized according to General Procedure using *N*-methyl-*N*-phenylmethacrylamide (35.0 mg, 0.2 mmol) and cyclohexanethiol (32.5 mg, 0.28 mmol) irradiated with blue LEDs for 24 h. Purification by column chromatography (silica gel, petroleum ether/CH₂Cl₂ 1:4) was performed to give **3a** (41.1 mg) in 71% yield; Colorless liquid. ¹H NMR (400 MHz, CDCl₃-*d*) δ 7.29 – 7.25 (m, 2H), 7.07 – 7.01 (m, 1H), 6.83 (d, *J* = 8.2 Hz, 1H), 3.21 (s, 3H), 3.04 (d, *J* = 12.6 Hz, 1H), 2.88 (d, *J* = 12.6 Hz, 1H), 2.43 – 2.35 (m, 1H), 1.86 – 1.75 (m, 2H), 1.70 – 1.60 (m, 2H), 1.55 – 1.48 (m, 1H), 1.40 (s, 3H), 1.20 – 1.08 (m, 5H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.5, 143.4, 132.9, 128.1, 123.0, 122.3, 107.9, 48.9, 44.8, 38.0, 33.7, 33.5, 26.1, 26.0, 25.6, 22.8; HRMS (ESI) m/z calced for C₁₇H₂₄NOS⁺ (M+H)⁺ 290.1573, found 290.1575.

3-((cyclopentylthio)methyl)-1,3-dimethylindolin-2-one (3b)



The desired compound **3b** was synthesized according to General Procedure using *N*-methyl-*N*-phenylmethacrylamide (35.0 mg, 0.2 mmol) and cyclopentanethiol (28.6 mg, 0.28 mmol) irradiated with blue LEDs for 24 h. Purification by column chromatography (silica gel, petroleum ether/CH₂Cl₂ 1:3) was performed to give **3b** (42.4 mg) in 77% yield; Light yellow liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.30 – 7.26 (m, 2H), 7.06 (t, *J* = 7.4 Hz, 1H), 6.85 (d, *J* = 7.9 Hz, 1H), 3.23 (s, 3H), 3.06 (d, *J* = 12.6 Hz, 1H), 2.93 – 2.86 (m, 2H), 1.89 – 1.83 (m, 2H), 1.68 – 1.58 (m, 2H), 1.49 – 1.45 (m, 2H), 1.42 (s, 3H), 1.40 – 1.32 (m, 2H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.5, 143.3, 132.9, 128.1, 122.9, 122.4, 108.0, 48.9, 45.0, 39.5, 33.8, 33.5, 26.2, 24.6, 22.9; HRMS (ESI) m/z calced for C₁₆H₂₂NOS⁺ (M+H)⁺ 276.1417, found 276.1418.

3-((((3s,5s,7s)-adamantan-1-yl)thio)methyl)-1,3-dimethylindolin-2-one (3c)



The desired compound 3c was synthesized according to General Procedure using

N-methyl-*N*-phenylmethacrylamide (35.0 mg, 0.2 mmol) and (3s,5s,7s)-adamantane-1-thiol (47.1 mg, 0.28 mmol) irradiated with blue LEDs for 36 h. Purification by column chromatography (silica gel, petroleum ether/CH₂Cl₂ 1:2) was performed to give **3c** (51.9 mg) in 76% yield; Colorless liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.33 – 7.24 (m, 2H), 7.05 (t, *J* = 7.5 Hz, 1H), 6.84 (d, *J* = 7.8 Hz, 1H), 3.22 (s, 3H), 2.99 (d, *J* = 11.7 Hz, 1H), 2.89 (d, *J* = 11.7 Hz, 1H), 2.03 – 1.95 (m, 3H), 1.79 – 1.71 (m, 6H), 1.67 – 1.58 (m, 6H), 1.43 (s, 3H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.5, 143.2, 132.9, 128.0, 123.2, 122.4, 107.9, 48.0, 44.3, 43.1, 36.2, 32.9, 29.6, 26.2, 23.0; HRMS (ESI) m/z calced for C₂₁H₂₈NOS⁺ (M+H)⁺ 342.1886, found 342.1887.

1,3-dimethyl-3-((propylthio)methyl)indolin-2-one (3d)



The desired compound **3d** was synthesized according to General Procedure using *N*-methyl-*N*-phenylmethacrylamide (35.0 mg, 0.2 mmol) and propane-1-thiol (21.3 mg, 0.28 mmol) irradiated with blue LEDs for 24 h. Purification by column chromatography (silica gel, petroleum ether/CH₂Cl₂ 1:5) was performed to give **3d** (33.9 mg) in 68% yield; Colorless liquid. ¹H NMR (400 MHz, CDCl₃-*d*) δ 7.32 – 7.26 (m, 2H), 7.07 (t, *J* = 7.5 Hz, 1H), 6.86 (d, *J* = 7.7 Hz, 1H), 3.24 (s, 3H), 3.03 (d, *J* = 12.8 Hz, 1H), 2.91 (d, *J* = 13.1 Hz, 1H), 2.31 (t, *J* = 7.3 Hz, 2H), 1.46 (q, *J* = 7.4 Hz, 2H), 1.42 (s, 3H), 0.86 (t, *J* = 7.4 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.5, 143.5, 132.9, 128.2, 123.0, 122.4, 108.0, 49.2, 40.0, 35.7, 26.2, 22.9, 22.9, 13.2; HRMS (ESI) m/z calced for C₁₄H₂₀NOS⁺ (M+H)⁺ 250.1260, found 250.1262.

1,3-dimethyl-3-((octylthio)methyl)indolin-2-one (3e)



The desired compound **3e** was synthesized according to General Procedure using *N*-methyl-*N*-phenylmethacrylamide (35.0 mg, 0.2 mmol) and octane-1-thiol (41.0 mg, 0.28 mmol) irradiated with blue LEDs for 24 h. Purification by column chromatography (silica gel, petroleum ether/CH₂Cl₂ 1:4) was performed to give **3e** (38.9 mg) in 61% yield; Colorless liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.30 – 7.25 (m, 2H), 7.05 (t, J = 7.5 Hz, 1H), 6.85 (d, J = 7.4 Hz, 1H), 3.22 (s, 3H), 3.01 (d, J = 13.0 Hz, 1H), 2.90 (d, J = 13.0 Hz, 1H), 2.31 (t, J = 7.4 Hz, 2H), 1.42 – 1.39 (m, 5H), 1.28 – 1.20 (m, 10H), 0.85 (t, J = 6.9 Hz, 3H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.5, 143.5, 133.0, 128.2, 123.0, 122.4, 108.0, 49.2, 40.1, 33.8, 31.8, 29.6, 29.1, 29.1, 28.6, 26.2, 22.9, 22.6, 14.1; HRMS (ESI) m/z calced for C₁₉H₃₀NOS⁺ (M+H)⁺ 320.2043, found 320.2044.

1,3-dimethyl-3-(((3-methylbutan-2-yl)thio)methyl)indolin-2-one (3f, 1.38:1 dr)



The desired compound **3f** was synthesized according to General Procedure using *N*-methyl-*N*-phenylmethacrylamide (35.0 mg, 0.2 mmol) and 3-methylbutane-2-thiol (29.2 mg, 0.28 mmol) irradiated with blue LEDs for 36 h. Purification by column chromatography (silica gel, petroleum ether/CH₂Cl₂ 1:3) was performed to give **3f** (36.0 mg) in 65% yield; Colorless liquid. ¹H NMR (400 MHz, CDCl₃-*d*) δ 7.32 – 7.24 (m, 2H), 7.09 – 7.01 (m, 1H), 6.84 (d, *J* = 7.5 Hz, 1H), 3.22 (d, *J* = 3.9 Hz, 3H), 3.05 – 2.97 (m, 1H), 2.92 – 2.85 (m, 1H), 2.50 (qd, *J* = 6.9, 4.6 Hz, 0.42H), 2.40 (qd, *J* = 6.9, 4.6 Hz, 0.58H), 1.71 – 1.41 (m, 1H), 1.41 (s, 3H), 1.06 (t, *J* = 6.6 Hz, 3H), 0.84 – 0.77 (m, 6H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.6, 179.5, 143.5, 143.5, 133.0, 133.0, 128.1, 128.1, 122.9, 122.4, 122.3, 108.0, 107.9, 49.3, 49.2, 48.6, 48.5, 39.0, 39.0, 32.9, 32.7, 26.2, 26.1, 22.9, 22.8, 19.6, 19.6, 18.4, 18.4, 17.9, 17.7; HRMS (ESI) m/z calced for C₁₆H₂₄NOS⁺ (M+H)⁺ 278.1573, found 278.1574.

1,3-dimethyl-3-(((3-oxobutan-2-yl)thio)methyl)indolin-2-one (3g, 1.1:1 dr)



The desired compound 3g was synthesized according to General Procedure using *N*-methyl-*N*-phenylmethacrylamide (35.0 mg, 0.2 mmol) and 3-mercaptobutan-2-one (29.2 mg, 0.28 mmol) irradiated with blue LEDs for 36 h. Purification by column chromatography (silica gel, petroleum ether/ethyl acetate 10:1) was performed to give 3g (38.8 mg) in 70% yield; Light yellow liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.33 – 7.23 (m, 2H), 7.08 (t, *J* = 7.5 Hz, 1H), 6.87 (d, *J* = 7.8 Hz, 1H), 3.23 (s, 3H), 2.97 – 2.92 (m, 1H), 2.88 – 2.85 (m, 1H), 2.12 (s, 1.57H), 2.11 (s, 1.43H), 1.41 – 1.40 (m, 3H), 1.25 – 1.20 (m, 3H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 205.4, 205.3, 178.9, 143.4, 143.4, 132.2, 132.2, 128.4, 128.3, 123.0, 122.9, 122.5, 122.5, 108.1, 108.0, 49.1, 48.6, 48.4, 37.8, 37.1, 26.2, 25.5, 25.5, 23.1, 23.0, 15.9, 15.7; HRMS (ESI) m/z calced for C₁₅H₁₉NNaO₂S⁺ (M+Na)⁺ 300.1029, found 300.1030.

1,3-dimethyl-3-(((2-(4-methyl-2-oxocyclohexyl)propan-2-yl)thio)methyl)indolin-2-one (3h, 4:1 *dr*)



The desired compound **3h** was synthesized according to General Procedure using *N*-methyl-*N*-phenylmethacrylamide (35.0 mg, 0.2 mmol) and 2-(2-mercaptopropan-2-yl)-5-methylcyclohexan-1-one (52.2 mg, 0.28 mmol) irradiated with blue LEDs for 48 h. Purification by column chromatography (silica gel, petroleum ether/ethyl acetate

10:1) was performed to give **3h** (36.6 mg) in 51% yield; Colorless liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.30 – 7.26 (m, 2H), 7.06 (t, J = 7.5 Hz, 1H), 6.85 (d, J = 7.8 Hz, 1H), 3.22 (d, J = 6.3 Hz, 3H), 3.03 (d, J = 11.8 Hz, 0.2H), 2.95 (d, J = 11.6 Hz, 0.8H), 2.87 – 2.81 (m, 1H), 2.56 (dd, J = 12.6, 5.8 Hz, 0.2H), 2.48 (dd, J = 11.6, 5.1 Hz, 0.2H), 2.42 – 2.32 (m, 2H), 2.27 – 2.23 (m, 1H), 2.05 (t, J = 12.5 Hz, 1H), 1.88 – 1.81 (m, 2H), 1.72 – 1.66 (m, 1.6H), 1.44 – 1.40 (m, 6H), 1.26 (s, 2.4H), 1.22 (s, 0.6H), 1.00 (d, J = 6.2 Hz, 2.4H), 0.90 (d, J = 7.1 Hz, 0.6H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 210.6, 179.4, 143.2, 132.8, 128.2, 123.1, 122.4, 108.1, 57.2, 52.3, 48.0, 47.1, 36.7, 35.2, 34.6, 29.5, 27.2, 26.2, 24.2, 23.0, 22.2; HRMS (ESI) m/z calced for C₂₁H₂₉NNaO₂S⁺ (M+Na)⁺ 382.1811, found 282.1812.

methyl 2-(((1,3-dimethyl-2-oxoindolin-3-yl)methyl)thio)acetate (3i)



The desired compound **3i** was synthesized according to General Procedure using *N*-methyl-*N*-phenylmethacrylamide (35.0 mg, 0.2 mmol) and methyl 2-mercaptoacetate (29.7 mg, 0.28 mmol) irradiated with blue LEDs for 36 h. Purification by column chromatography (silica gel, petroleum ether/ethyl acetate 10:1) was performed to give **3i** (45.2 mg) in 81% yield; Colorless liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.31 – 7.24 (m, 2H), 7.06 (t, *J* = 7.5 Hz, 1H), 6.85 (d, *J* = 7.8 Hz, 1H), 3.67 (s, 3H), 3.22 (s, 3H), 3.18 (d, *J* = 13.3 Hz, 1H), 3.07 – 3.02 (m, 2H), 2.92 (d, *J* = 14.9 Hz, 1H), 1.40 (s, 3H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.1, 170.6, 143.4, 132.2, 128.4, 122.9, 122.5, 108.1, 52.3, 49.1, 39.7, 34.2, 26.2, 23.0; HRMS (ESI) m/z calced for C₁₄H₁₇NNaO₃S⁺ (M+Na)⁺ 302.0821, found 302.0822.

ethyl 2-(((1,3-dimethyl-2-oxoindolin-3-yl)methyl)thio)propanoate (3j, 1.38:1 dr)



The desired compound **3j** was synthesized according to General Procedure using *N*-methyl-*N*-phenylmethacrylamide (35.0 mg, 0.2 mmol) and ethyl 2-mercaptopropanoate (37.6 mg, 0.28 mmol) irradiated with blue LEDs for 36 h. Purification by column chromatography (silica gel, petroleum ether/acetone 10:1) was performed to give **3j** (45.5 mg) in 74% yield; Colorless liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.32 – 7.25 (m, 2H), 7.07 (t, *J* = 7.5 Hz, 1H), 6.86 (d, *J* = 7.8 Hz, 1H), 4.20 – 4.11 (m, 2H), 3.36 – 3.32 (m, 1H), 3.23 (d, *J* = 2.3 Hz, 3H), 3.19 – 3.15 (m, 1H), 3.10 – 3.04 (m, 1H), 1.43 (d, *J* = 3.2 Hz, 3H), 1.30 – 1.20 (m, 6H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.1, 179.0, 172.9, 172.7, 143.5, 143.3, 132.3, 132.3, 128.3, 128.3, 123.1, 122.9, 122.5, 122.3, 108.0, 108.0, 61.0, 48.8, 48.6, 42.2, 41.4, 39.1, 38.3, 26.1, 22.8, 22.7, 17.0, 16.9, 14.1, 14.0; HRMS (ESI) m/z calced for C₁₆H₂₁NNaO₃S⁺ (M+Na)⁺ 330.1134, found 330.1136.

3-(((2-hydroxyethyl)thio)methyl)-1,3-dimethylindolin-2-one (3k)



The desired compound **3k** was synthesized according to General Procedure using *N*-methyl-*N*-phenylmethacrylamide (35.0 mg, 0.2 mmol) and 2-mercaptoethan-1-ol (21.8 mg, 0.28 mmol) irradiated with blue LEDs for 48 h. Purification by column chromatography (silica gel, petroleum ether/acetone 10:1) was performed to give **3k** (21.1 mg) in 42% yield; Colorless liquid. ¹H NMR (400 MHz, CDCl₃-*d*) δ 7.32 (t, *J* = 7.7 Hz, 1H), 7.26 (d, *J* = 8.0 Hz, 1H), 7.10 (t, *J* = 7.5 Hz, 1H), 6.89 (d, *J* = 7.8 Hz, 1H), 3.63 (qt, *J* = 11.6, 5.7 Hz, 2H), 3.26 (s, 3H), 3.05 (q, *J* = 12.9 Hz, 2H), 2.55 (t, *J* = 5.7 Hz, 2H), 1.42 (s, 3H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.6, 143.4, 132.5, 128.4, 122.8, 122.7, 108.2, 60.8, 49.4, 39.1, 36.4, 26.3, 23.1; HRMS (ESI) m/z calced for C₁₃H₁₇NNaO₂S⁺ (M+Na)⁺ 274.0872, found 274.0874.

3-((benzylthio)methyl)-1,3-dimethylindolin-2-one (3l)



The desired compound **31** was synthesized according to General Procedure using *N*-methyl-*N*-phenylmethacrylamide (35.0 mg, 0.2 mmol) and phenylmethanethiol (34.8 mg, 0.28 mmol) irradiated with blue LEDs for 24 h. Purification by column chromatography (silica gel, petroleum ether/CH₂Cl₂ 1:2) was performed to give **31** (39.8 mg) in 67% yield; Light yellow liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.33 – 7.19 (m, 7H), 7.08 (t, J = 7.5 Hz, 1H), 6.88 (d, J = 7.8 Hz, 1H), 3.52 (q, J = 13.2 Hz, 2H), 3.25 (s, 3H), 2.88 (s, 2H), 1.39 (s, 3H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.5, 143.5, 138.0, 132.9, 128.9, 128.4, 128.3, 126.9, 123.0, 122.4, 108.1, 49.1, 39.0, 37.4, 26.2, 22.8; HRMS (ESI) m/z calced for C₁₈H₁₉NNaOS⁺ (M+Na)⁺ 320.1080, found 320.1082.

1,3-dimethyl-3-((phenethylthio)methyl)indolin-2-one (3m)



The desired compound **3m** was synthesized according to General Procedure using *N*-methyl-*N*-phenylmethacrylamide (35.0 mg, 0.2 mmol) and 2-phenylethane-1-thiol (38.7 mg, 0.28 mmol) irradiated with blue LEDs for 24 h. Purification by column chromatography (silica gel, petroleum ether/CH₂Cl₂ 1:2) was performed to give **3m** (42.9 mg) in 69% yield; Light yellow liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.33 – 7.24 (m, 4H), 7.19 (t, *J* = 7.3 Hz, 1H), 7.14 – 7.03 (m, 3H), 6.86 (d, *J* = 7.8 Hz, 1H), 3.23 (s, 3H), 3.04 (d, *J* = 13.1 Hz, 1H), 2.96 (d, *J* = 13.0 Hz, 1H), 2.75 – 2.71 (m, 2H), 2.59 (t, *J* = 7.6 Hz, 2H), 1.43 (s, 3H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.4, 143.5, 140.3, 132.8, 128.4, 128.3, 128.2, 126.2, 122.9, 122.4, 108.1, 49.2, 40.1, 36.2, 35.0, 26.2, 22.9; HRMS (ESI) m/z calced for C₁₉H₂₂NOS⁺ (M+H)⁺ 312.1417, found 312.1418.

3-(((furan-2-ylmethyl)thio)methyl)-1,3-dimethylindolin-2-one (3n)



The desired compound **3n** was synthesized according to General Procedure using *N*-methyl-*N*-phenylmethacrylamide (35.0 mg, 0.2 mmol) and furan-2-ylmethanethiol (32.0 mg, 0.28 mmol) irradiated with blue LEDs for 48 h. Purification by column chromatography (silica gel, petroleum ether/CH₂Cl₂ 1:4) was performed to give **3n** (24.7 mg) in 43% yield; Colorless liquid. ¹H NMR (400 MHz, CDCl₃-*d*) δ 7.33 – 7.28 (m, 2H), 7.25 (d, *J* = 5.5 Hz, 1H), 7.07 (t, *J* = 7.2 Hz, 1H), 6.87 (d, *J* = 7.7 Hz, 1H), 6.28 (d, *J* = 3.1 Hz, 1H), 6.14 (d, *J* = 3.2 Hz, 1H), 3.55 (d, *J* = 14.7 Hz, 1H), 3.42 (d, *J* = 14.7 Hz, 1H), 3.25 (s, 3H), 3.01 – 2.89 (m, 2H), 1.40 (s, 3H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.4, 151.3, 143.5, 142.2, 132.8, 128.3, 123.0, 122.5, 110.3, 108.1, 107.8, 49.2, 39.0, 29.4, 26.3, 22.9; HRMS (ESI) m/z calced for C₁₆H₁₈NO₂S⁺ (M+H)⁺ 288.1053, found 288.1054.

3-(((1,3,4-thiadiazol-2-yl)thio)methyl)-1,3-dimethylindolin-2-one (30)



The desired compound **30** was synthesized according to General Procedure using *N*-methyl-*N*-phenylmethacrylamide (35.0 mg, 0.2 mmol) and 1,3,4-thiadiazole-2-thiol (33.1 mg, 0.28 mmol) irradiated with blue LEDs for 36 h. Purification by column chromatography (silica gel, petroleum ether/ethyl acetate 2:1) was performed to give **30** (41.4 mg) in 71% yield; Colorless liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 8.89 (s, 1H), 7.24 (t, *J* = 7.4 Hz, 1H), 7.17 (d, *J* = 7.3 Hz, 1H), 6.93 (t, *J* = 7.5 Hz, 1H), 6.84 (d, *J* = 7.8 Hz, 1H), 4.05 (d, *J* = 13.0 Hz, 1H), 3.82 (d, *J* = 13.1 Hz, 1H), 3.23 (s, 3H), 1.50 (s, 3H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 178.3, 165.3, 151.7, 143.3, 131.2, 128.6, 123.5, 122.5, 108.2, 48.6, 41.2, 26.3, 22.7; HRMS (ESI) m/z calced for C₁₃H₁₄N₃OS₂⁺ (M+H)⁺ 292.0573, found 292.0576.

1,3-dimethyl-3-(((1-methyl-1H-tetrazol-5-yl)thio)methyl)indolin-2-one (3p)



The desired compound **3p** was synthesized according to General Procedure using *N*-methyl-*N*-phenylmethacrylamide (35.0 mg, 0.2 mmol) and 1-methyl-1*H*-tetrazole-5-thiol (32.5 mg, 0.28 mmol) irradiated with blue LEDs for 24 h. Purification by column chromatography (silica gel, petroleum ether/ethyl acetate 3:1) was performed to give **3p** (44.5 mg) in 77% yield; Colorless solid; mp 94-96 °C.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.22 (t, *J* = 7.7 Hz, 1H), 6.97 (d, *J* = 7.3 Hz, 1H), 6.88 (t, *J* = 8.0 Hz, 1H), 6.84 (d, *J* = 7.8 Hz, 1H), 3.90 (d, *J* = 13.3 Hz, 1H), 3.74 (d, *J* = 13.3 Hz, 1H), 3.52 (s, 3H), 3.22 (s, 3H), 1.44 (s, 3H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 178.1, 153.7, 143.2, 130.5, 128.6, 123.0, 122.6, 108.0, 48.8, 40.7, 33.1, 26.3, 22.7; HRMS (ESI) m/z calced for C₁₃H₁₆N₅OS⁺ (M+H)⁺ 290.1070, found 290.1072.

1,3-dimethyl-3-((pyridin-2-ylthio)methyl)indolin-2-one (3q)



The desired compound 3q was synthesized according to General Procedure using *N*-methyl-*N*-phenylmethacrylamide (35.0 mg, 0.2 mmol) and pyridine-2-thiol (31.0 mg, 0.28 mmol) irradiated with blue LEDs for 36 h. Purification by column chromatography (silica gel, petroleum ether/ethyl acetate 10:1) was performed to give 3q (29.0 mg) in 51% yield; Colorless liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 8.37 (d, J = 4.2 Hz, 1H), 7.34 (td, J = 7.7, 1.9 Hz, 1H), 7.23 – 7.19 (m, 2H), 6.97 (d, J = 8.1 Hz, 1H), 6.94 – 6.87 (m, 2H), 6.82 (d, J = 8.2 Hz, 1H), 3.89 (d, J = 13.1 Hz, 1H), 3.64 (d, J = 13.1 Hz, 1H), 3.23 (s, 3H), 1.49 (s, 3H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.2, 157.9, 149.0, 143.2, 135.7, 132.4, 128.1, 123.4, 122.6, 122.2, 119.6, 107.8, 48.5, 36.8, 26.2, 22.7; HRMS (ESI) m/z calced for C₁₆H₁₇N₂OS⁺ (M+H)⁺ 285.1056, found 285.1058.

1,3-dimethyl-3-((phenylthio)methyl)indolin-2-one (3r)⁴



The desired compound 3r was synthesized according to General Procedure using *N*-methyl-*N*-phenylmethacrylamide (35.0 mg, 0.2 mmol) and benzenethiol (30.8 mg, 0.28 mmol) irradiated with blue LEDs for 48 h. Purification by column chromatography (silica gel, petroleum ether/ethyl acetate 10:1) was performed to give 3r (9.6 mg) in 17% yield; Colorless liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.28 (t, *J* = 7.9 Hz, 1H), 7.23 – 7.10 (m, 6H), 6.98 (t, *J* = 7.5 Hz, 1H), 6.86 (d, *J* = 7.8 Hz, 1H), 3.45 – 3.34 (m, 2H), 3.21 (s, 3H), 1.45 (s, 3H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.0, 143.3, 136.1, 132.2, 130.5, 128.6, 128.2, 126.4, 123.2, 122.4, 108.0, 48.9, 42.7, 26.2, 22.9.

1,3-dimethyl-3-((pyridin-2-ylthio)methyl)indolin-2-one (3s)⁴



The desired compound **3s** was synthesized according to General Procedure using *N*-methyl-*N*-phenylmethacrylamide (35.0 mg, 0.2 mmol) and 4-methoxybenzenethiol (39.2 mg, 0.28 mmol) irradiated with blue LEDs for 48 h. Purification by column chromatography (silica gel, petroleum ether/ethyl acetate 10:1) was performed to give **3s** (9.4 mg) in 15% yield; Colorless liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.28 (t, *J* = 7.5 Hz, 1H), 7.13 (t, *J* = 8.5 Hz, 3H), 6.98 (t, *J* = 7.5 Hz, 1H), 6.86 (d, *J* = 7.8 Hz, 1H), 6.71 (d, *J* = 8.8 Hz, 2H), 3.75 (s, 3H), 3.31 (s, 2H), 3.21 (s, 3H), 1.41 (s, 3H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.0, 158.9, 143.4, 133.8, 132.3, 128.1, 126.4, 123.2, 122.4, 114.2, 107.9, 55.2, 49.2, 44.4, 26.2, 23.1.

3-(((4-chlorophenyl)thio)methyl)-1,3-dimethylindolin-2-one (3t)⁴



The desired compound **3t** was synthesized according to General Procedure using *N*-methyl-*N*-phenylmethacrylamide (35.0 mg, 0.2 mmol) and 4-chlorobenzenethiol (40.3 mg, 0.28 mmol) irradiated with blue LEDs for 48 h. Purification by column chromatography (silica gel, petroleum ether/ethyl acetate 10:1) was performed to give **3t** (12.0 mg) in 19% yield; Colorless liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.28 (t, *J* = 7.1 Hz, 1H), 7.14 – 7.08 (m, 5H), 6.98 (t, *J* = 7.5 Hz, 1H), 6.86 (d, *J* = 7.8 Hz, 1H), 3.37 (dd, *J* = 12.8, 2.5 Hz, 2H), 3.21 (s, 3H), 1.43 (s, 3H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 178.8, 143.4, 134.5, 132.5, 132.0, 131.9, 128.7, 128.3, 123.2, 122.5, 108.1, 49.1, 42.9, 26.3, 23.1.

3-(((4-bromophenyl)thio)methyl)-1,3-dimethylindolin-2-one (3u)⁵



The desired compound 3u was synthesized according to General Procedure using *N*-methyl-*N*-phenylmethacrylamide (35.0 mg, 0.2 mmol) and 4-bromobenzenethiol (39.2 mg, 0.28 mmol) irradiated with blue LEDs for 48 h. Purification by column chromatography (silica gel, petroleum ether/ethyl acetate 10:1) was performed to give 3u (14.5 mg) in 20% yield; Colorless liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.32 – 7.24 (m, 3H), 7.13 (d, *J* = 7.7 Hz, 1H), 7.07 – 6.95 (m, 3H), 6.86 (d, *J* = 7.8 Hz, 1H), 3.37 (dd, *J* = 12.8, 3.0 Hz, 2H), 3.21 (s, 3H), 1.44 (s, 3H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 178.8, 143.3, 135.2, 132.0, 132.0, 131.6, 128.3, 123.2, 122.5, 120.4, 108.1, 49.0, 42.7, 26.2, 23.0.

3-((cyclohexylthio)methyl)-1,3,5-trimethylindolin-2-one (4a)



The desired compound **4a** was synthesized according to General Procedure using *N*-methyl-*N*-(*p*-tolyl)methacrylamide (37.8 mg, 0.2 mmol) and cyclohexanethiol (32.5 mg, 0.28 mmol) irradiated with blue LEDs for 24 h. Purification by column chromatography (silica gel, petroleum ether/CH₂Cl₂ 1:4) was performed to give **4a** (50.3 mg) in 83% yield; Colorless liquid. ¹H NMR (400 MHz, CDCl₃-*d*) δ 7.11 – 7.05 (m, 2H), 6.73 (d, *J* = 7.8 Hz, 1H), 3.20 (s, 3H), 3.04 (d, *J* = 12.7 Hz, 1H), 2.87 (d, *J* = 12.7 Hz, 1H), 2.43 – 2.37 (m, 1H), 2.33 (s, 3H), 1.88 – 1.75 (m, 2H), 1.71 – 1.61 (m, 2H), 1.56 – 1.51 (m, 1H), 1.40 (s, 3H), 1.20 – 1.13 (m, 5H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.4, 141.0, 132.9, 131.8, 128.3, 123.8, 107.6, 49.0, 44.8, 38.0, 33.7, 33.4, 26.2, 26.0, 25.6, 22.8, 21.1; HRMS (ESI) m/z calced for C₁₈H₂₆NOS⁺ (M+H)⁺ 304.1730, found 304.1732.

3-((cyclohexylthio)methyl)-5-methoxy-1,3-dimethylindolin-2-one (4b)



The desired compound **4b** was synthesized according to General Procedure using N-(4-methoxyphenyl)-N-methylmethacrylamide (41.0 mg, 0.2 mmol) and cyclohexanethiol (32.5 mg, 0.28 mmol) irradiated with blue LEDs for 36 h. Purification by column chromatography (silica gel, petroleum ether/CH₂Cl₂ 1:4) was performed to give **4b** (50.4 mg) in 79% yield; Light yellow liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 6.92 (d, *J* = 2.5 Hz, 1H), 6.81 (d, *J* = 8.5 Hz, 1H), 6.75 (d, *J* = 8.4 Hz, 1H), 3.79 (s, 3H), 3.20 (s, 3H), 3.03 (d, *J* = 12.7 Hz, 1H), 2.88 (d, *J* = 12.7 Hz, 1H), 2.46 – 2.42 (m, 1H), 1.88 – 1.80 (m, 2H), 1.68 – 1.66 (m, 2H), 1.57 – 1.55 (m, 1H), 1.41 (s, 3H), 1.19 – 1.14 (m, 5H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.2, 155.9, 136.9, 134.3, 112.2, 110.7, 108.2, 55.8, 49.3, 44.9, 38.0, 33.8, 33.5, 26.3, 26.0, 25.7, 22.9; HRMS (ESI) m/z calced for C₁₈H₂₆NO₂S⁺ (M+H)⁺ 320.1679, found 320.1680.

5-chloro-3-((cyclohexylthio)methyl)-1,3-dimethylindolin-2-one (4c)



The desired compound **4c** was synthesized according to General Procedure using *N*-(4-chlorophenyl)-*N*-methylmethacrylamide (41.9 mg, 0.2 mmol) and cyclohexanethiol (32.5 mg, 0.28 mmol) irradiated with blue LEDs for 24 h. Purification by column chromatography (silica gel, petroleum ether/CH₂Cl₂ 1:4) was performed to give **4c** (45.3 mg) in 70% yield; Colorless liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.28 – 7.21 (m, 2H), 6.76 (d, *J* = 8.1 Hz, 1H), 3.20 (s, 3H), 3.01 (d, *J* = 12.7 Hz, 1H), 2.88 (s, 1H), 2.45 – 2.36 (m, 1H), 1.85 – 1.76 (m, 2H), 1.71 – 1.63 (m, 2H), 1.56 – 1.50 (m, 1H), 1.40 (s, 3H), 1.22 – 1.09 (m, 5H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.0, 142.0, 134.6, 128.0, 127.7, 123.6, 108.9, 49.2, 45.0, 37.8, 33.7, 33.4, 26.3, 25.9, 25.6, 22.7; HRMS (ESI) m/z calced for C₁₇H₂₃ClNOS⁺ (M+H)⁺ 324.1183, found 324.1187.

5-bromo-3-((cyclohexylthio)methyl)-1,3-dimethylindolin-2-one (4d)



The desired compound **4d** was synthesized according to General Procedure using N-(4-bromophenyl)-N-methylmethacrylamide (50.8 mg, 0.2 mmol) and cyclohexanethiol (32.5 mg, 0.28 mmol) irradiated with blue LEDs for 36 h. Purification by column chromatography (silica gel, petroleum ether/ethyl acetate 10:1) was performed to give **4d** (59.6 mg) in 81% yield; Colorless liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.42 – 7.36 (m, 2H), 6.71 (d, J = 8.7 Hz, 1H), 3.19 (s, 3H), 3.01 (d, J = 12.9 Hz, 1H), 2.86 (d, J = 13.0 Hz, 1H), 2.44 – 2.33 (m, 1H), 1.85 – 1.76 (m, 2H), 1.70 – 1.63 (m, 2H), 1.55 – 1.52 (m, 1H), 1.39 (s, 3H), 1.21 – 1.09 (m, 5H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 178.9, 142.5, 135.0, 130.9, 126.3, 115.0, 109.4, 49.2, 45.1, 37.8, 33.7, 33.5, 26.2, 26.0, 25.6, 22.7; HRMS (ESI) m/z calced for C₁₇H₂₃BrNOS⁺ (M+H)⁺ 368.0678, found 368.0680.

3-((cyclohexylthio)methyl)-1,3-dimethyl-5-(trifluoromethyl)indolin-2-one (4e)



The desired compound **4e** was synthesized according to General Procedure using *N*-methyl-*N*-(4-(trifluoromethyl)phenyl)methacrylamide (48.6 mg, 0.2 mmol) and cyclohexanethiol (32.5 mg, 0.28 mmol) irradiated with blue LEDs for 36 h. Purification by column chromatography (silica gel, petroleum ether/CH₂Cl₂ 1:2) was performed to give **4e** (60.0 mg) in 84% yield; Colorless liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.56 (d, J = 8.1 Hz, 1H), 7.52 (s, 1H), 6.91 (d, J = 8.2 Hz, 1H), 3.25 (s, 3H), 3.05 (d, J = 13.0 Hz, 1H), 2.89 (d, J = 13.0 Hz, 1H), 2.39 – 2.28 (m, 1H), 1.86 – 1.79 (m, 1H), 1.75 – 1.71 (m, 1H), 1.69 – 1.60 (m, 2H), 1.55 – 1.49 (m, 1H), 1.43 (s, 3H), 1.19 – 1.12 (m, 5H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.5, 146.5, 133.5, 125.9 (q, J = 4.0 Hz), 124.5 (q, J = 32.4 Hz), 124.4 (q, J = 271.6 Hz), 120.1 (q, J = 3.7 Hz), 107.7, 49.1, 45.1, 37.8, 33.7, 33.5, 26.3, 25.9, 25.6, 22.6; ¹⁹F NMR (376 MHz, CDCl₃-*d*) δ -61.33; HRMS (ESI) m/z calced for C₁₈H₂₂F₃NNaOS⁺ (M+Na)⁺ 380.1266, found 280.1268.

3-((cyclohexylthio)methyl)-1,3-dimethyl-2-oxoindoline-5-carbonitrile (4f)



The desired compound **4f** was synthesized according to General Procedure using *N*-(4-cyanophenyl)-*N*-methylmethacrylamide (40.0 mg, 0.2 mmol) and cyclohexanethiol (32.5 mg, 0.28 mmol) irradiated with blue LEDs for 36 h. Purification by column chromatography (silica gel, petroleum ether/CH₂Cl₂ 1:2) was performed to give **4f** (39.6 mg) in 63% yield; Light yellow solid; mp 115-117 °C.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.61 (d, J = 8.1 Hz, 1H), 7.54 (s, 1H), 6.91 (d, J = 8.1 Hz, 1H), 3.25 (s, 3H), 3.04 (d, J = 12.8 Hz, 1H), 2.89 (d, J = 12.8 Hz, 1H), 2.47 – 2.37 (m, 1H), 1.86 – 1.76 (m, 2H), 1.70 – 1.66 (m, 2H), 1.54 (d, J = 9.8 Hz, 1H), 1.43 (s, 3H), 1.21 – 1.13 (m, 5H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.2, 147.3, 133.9, 133.5, 126.5, 119.2, 108.4, 105.4, 48.8, 45.2, 37.7, 33.7, 33.4, 26.4, 25.9, 25.6, 22.6; HRMS (ESI) m/z calced for C₁₈H₂₃N₂OS⁺ (M+H)⁺ 315.1526, found 315.1528.

3-((cyclohexylthio)methyl)-1,3-dimethyl-5-nitroindolin-2-one (4g)



The desired compound **4g** was synthesized according to General Procedure using *N*-methyl-*N*-(4-nitrophenyl)methacrylamide (44.0 mg, 0.2 mmol) and cyclohexanethiol (32.5 mg, 0.28 mmol) irradiated with blue LEDs for 48 h. Purification by column chromatography (silica gel, petroleum ether/ethyl acetate 10:1) was performed to give **4g** (34.1 mg) in 51% yield; Yellow liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 8.27 (dd, *J* = 8.6, 2.3 Hz, 1H), 8.17 (s, 1H), 6.93 (d, *J* = 8.6 Hz, 1H), 3.29 (s, 3H), 3.09 (d, *J* = 12.8 Hz, 1H), 2.93 (d, *J* = 12.8 Hz, 1H), 2.47 – 2.42 (m, 1H), 1.86 – 1.75 (m, 2H), 1.71 – 1.65 (m, 2H), 1.55 – 1.52 (m, 1H), 1.47 (s, 3H), 1.21 – 1.11 m, 5H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.6, 149.2, 143.3, 133.7, 125.6, 118.9, 107.6, 49.1, 45.3, 37.7, 33.7, 33.4, 26.6, 25.9, 25.9, 25.5, 22.7; HRMS (ESI) m/z calced for C₁₇H₂₂N₂NaO₃S⁺ (M+Na)⁺ 357.1243, found 357.1246.

4-chloro-3-((cyclohexylthio)methyl)-1,3-dimethylindolin-2-one and 6-chloro-3-((cyclohexylthio)methyl)-1,3-dimethylindolin-2-one (4h, rr = 2.7:1)



The desired compound **4h** was synthesized according to General Procedure using N-(3-chlorophenyl)-N-methylmethacrylamide (41.9 mg, 0.2 mmol) and cyclohexanethiol (32.5 mg,

0.28 mmol) irradiated with blue LEDs for 36 h. Purification by column chromatography (silica gel, petroleum ether/ CH_2Cl_2 1:3) was performed to give **4h** (49.8 mg) in 77% yield; Light yellow liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.23 – 7.17 (m, 1H), 7.01 (dd, *J* = 7.9, 1.9 Hz, 0.27H), 6.97 (d, *J* = 8.2 Hz, 0.73H), 6.83 (d, *J* = 1.9 Hz, 0.27H), 6.74 (d, *J* = 7.8 Hz, 0.73H), 3.53 (d, *J* = 12.7 Hz, 0.73H), 3.20 (d, *J* = 7.1 Hz, 3H), 3.02 (d, *J* = 12.6 Hz, 0.27H), 2.96 (d, *J* = 12.7 Hz, 0.73H), 2.86 (d, *J* = 12.7 Hz, 0.27H), 2.44 – 2.35 (m, 1H), 1.89 – 1.80 (m, 1.4H), 1.71 – 1.60 (m, 2.6H), 1.52 – 1.50 (m, 3.3H), 1.39 (s, 0.82H), 1.18 – 1.06 (m, 5H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.4, 178.8, 145.6, 144.6, 133.8, 131.2, 130.6, 129.4, 128.8, 123.9, 123.3, 122.1, 108.7, 106.5, 51.0, 48.7, 45.0, 44.4, 37.8, 34.9, 33.7, 33.7, 33.4, 26.3, 26.2, 26.0, 25.6, 22.7, 20.8; HRMS (ESI) m/z calced for C₁₇H₂₃CINOS⁺ (M+H)⁺ 324.1183, found 324.1186.

3-((cyclohexylthio)methyl)-1,3,7-trimethylindolin-2-one (4i)



The desired compound **4i** was synthesized according to General Procedure using *N*-methyl-*N*-(*o*-tolyl)methacrylamide (37.8 mg, 0.2 mmol) and cyclohexanethiol (32.5 mg, 0.28 mmol) irradiated with blue LEDs for 36 h. Purification by column chromatography (silica gel, petroleum ether/CH₂Cl₂ 1:4) was performed to give **4i** (37.0 mg) in 61% yield; Colorless liquid. ¹H NMR (400 MHz, CDCl₃-*d*) δ 7.10 (d, *J* = 7.2 Hz, 1H), 7.00 (d, *J* = 7.6 Hz, 1H), 6.94 (t, *J* = 7.5 Hz, 1H), 3.51 (s, 3H), 3.03 (d, *J* = 12.6 Hz, 1H), 2.89 (d, *J* = 12.6 Hz, 1H), 2.58 (s, 3H), 2.45 – 2.39 (m, 1H), 1.88 – 1.78 (m, 2H), 1.69 – 1.66 (m, 2H), 1.55 – 1.53 (m, 1H), 1.39 (s, 3H), 1.21 – 1.13 (m, 5H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 180.3, 141.2, 133.6, 131.9, 122.3, 120.8, 119.6, 48.3, 44.8, 38.3, 33.8, 33.5, 29.5, 26.0, 25.7, 23.3, 19.1; HRMS (ESI) m/z calced for C₁₈H₂₆NOS⁺ (M+H)⁺ 304.1730, found 304.1732.

3-((cyclohexylthio)methyl)-1-ethyl-3-methylindolin-2-one (4j)



The desired compound **4j** was synthesized according to General Procedure using *N*-ethyl-*N*-phenylmethacrylamide (37.8 mg, 0.2 mmol) and cyclohexanethiol (32.5 mg, 0.28 mmol) irradiated with blue LEDs for 36 h. Purification by column chromatography (silica gel, petroleum ether/CH₂Cl₂ 1:4) was performed to give **4j** (40.6 mg) in 67% yield; Colorless liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.30 – 7.23 (m, 2H), 7.04 (t, *J* = 7.5 Hz, 1H), 6.86 (d, *J* = 8.4 Hz, 1H), 3.90 – 3.81 (m, 1H), 3.74 – 3.65 (m, 1H), 3.05 (d, *J* = 12.8 Hz, 1H), 2.90 (d, *J* = 12.7 Hz, 1H), 2.45 – 2.34 (m, 1H), 1.87 – 1.75 (m, 2H), 1.69 – 1.64 (m, 2H), 1.53 – 1.49 (m, 1H), 1.40 (s, 3H), 1.27 (t, *J* = 7.2 Hz, 3H), 1.19 – 1.12 (m, 5H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.1, 142.5, 133.2, 128.0, 123.1, 122.1, 108.1, 48.8, 44.6, 37.9, 34.6, 33.7, 33.4, 25.9, 25.6, 22.8, 12.6; HRMS

(ESI) m/z calced for $C_{18}H_{26}NOS^+$ (M+H)⁺ 304.1730, found 304.1731.

3-((cyclohexylthio)methyl)-1-isopropyl-3-methylindolin-2-one (4k)



The desired compound **4k** was synthesized according to General Procedure using *N*-isopropyl-*N*-phenylmethacrylamide (40.6 mg, 0.2 mmol) and cyclohexanethiol (32.5 mg, 0.28 mmol) irradiated with blue LEDs for 36 h. Purification by column chromatography (silica gel, petroleum ether/CH₂Cl₂ 1:3) was performed to give **4k** (54.6 mg) in 86% yield; Colorless liquid. ¹H NMR (400 MHz, CDCl₃-*d*) δ 7.24 (t, *J* = 7.6 Hz, 2H), 7.02 (t, *J* = 7.3 Hz, 2H), 4.71 – 4.66 (m, 1H), 3.03 (d, *J* = 12.7 Hz, 1H), 2.89 (d, *J* = 12.7 Hz, 1H), 2.40 – 2.33 (m, 1H), 1.87 – 1.72 (m, 2H), 1.68 – 1.64 (m, 2H), 1.56 – 1.50 (m, 1H), 1.48 (dd, *J* = 7.1, 2.1 Hz, 6H), 1.39 (s, 3H), 1.22 – 1.06 (m, 5H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.2, 142.2, 133.4, 127.8, 123.1, 121.7, 109.7, 48.6, 44.5, 43.6, 38.2, 33.7, 33.4, 25.9, 25.6, 22.9, 19.4, 19.4; HRMS (ESI) m/z calced for C₁₉H₂₈NOS⁺ (M+H)⁺ 318.1886, found 318.1891.

1-cyclohexyl-3-((cyclohexylthio)methyl)-3-methylindolin-2-one (41)



The desired compound **41** was synthesized according to General Procedure using *N*-cyclohexyl-*N*-phenylmethacrylamide (48.7 mg, 0.2 mmol) and cyclohexanethiol (32.5 mg, 0.28 mmol) irradiated with blue LEDs for 36 h. Purification by column chromatography (silica gel, petroleum ether/CH₂Cl₂ 1:3) was performed to give **41** (57.2 mg) in 80% yield; Colorless liquid. ¹H NMR (400 MHz, CDCl₃-*d*) δ 7.24 (t, *J* = 7.7 Hz, 2H), 7.09 – 6.98 (m, 2H), 4.24 – 4.15 (m, 1H), 3.03 (d, *J* = 12.7 Hz, 1H), 2.89 (d, *J* = 12.7 Hz, 1H), 2.39 – 2.32 (m, 1H), 2.22 – 2.10 (m, 2H), 1.92 – 1.71 (m, 8H), 1.68 – 1.65 (m, 2H), 1.54 – 1.49 (m, 1H), 1.45 – 1.42 (m, 1H), 1.39 (s, 3H), 1.29 – 1.23 (m, 1H), 1.19 – 1.04 (m, 5H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.3, 142.6, 133.4, 127.7, 123.1, 121.6, 109.9, 52.1, 48.5, 44.5, 38.2, 33.7, 33.4, 29.1, 26.0, 26.0, 25.7, 25.4, 24.5, 23.1; HRMS (ESI) m/z calced for C₂₂H₃₂NOS⁺ (M+H)⁺ 358.2199, found 358.2204.

3-((cyclohexylthio)methyl)-3-methyl-1-phenylindolin-2-one (4m)



The desired compound **4m** was synthesized according to General Procedure using *N*,*N*-diphenylmethacrylamide (47.5 mg, 0.2 mmol) and cyclohexanethiol (32.5 mg, 0.28 mmol) irradiated with blue LEDs for 48 h. Purification by column chromatography (silica gel, petroleum ether/CH₂Cl₂ 1:1) was performed to give **4m** (40.1 mg) in 57% yield; Colorless solid; mp 105-107 $^{\circ}$ C.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.53 (t, *J* = 7.7 Hz, 2H), 7.48 – 7.37 (m, 3H), 7.33 (d, *J* = 7.2 Hz, 1H), 7.21 (t, *J* = 8.4 Hz, 1H), 7.10 (t, *J* = 7.5 Hz, 1H), 6.82 (d, *J* = 7.8 Hz, 1H), 3.16 (d, *J* = 12.8 Hz, 1H), 3.04 (d, *J* = 12.8 Hz, 1H), 2.46 – 2.40 (m, 1H), 1.91 – 1.86 (m, 1H), 1.83 – 1.80 (m, 1H), 1.70 – 1.65 (m, 2H), 1.55 – 1.53 (m, 4H), 1.23 – 1.13 (m, 5H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.0, 143.6, 134.6, 132.6, 129.5, 128.0, 127.9, 126.7, 123.1, 122.8, 109.2, 49.2, 44.7, 38.5, 33.8, 33.5, 25.9, 25.6, 23.1; HRMS (ESI) m/z calced for C₂₂H₂₆NOS⁺ (M+H)⁺ 352.1730, found 352.1734.

3-((cyclohexylthio)methyl)-3-methyl-1-((trimethylsilyl)methyl)indolin-2-one (4n)



The desired compound **4n** was synthesized according to General Procedure using *N*-phenyl-*N*-((trimethylsilyl)methyl)methacrylamide (49.5 mg, 0.2 mmol) and cyclohexanethiol (32.5 mg, 0.28 mmol) irradiated with blue LEDs for 36 h. Purification by column chromatography (silica gel, petroleum ether/CH₂Cl₂ 1:4) was performed to give **4n** (19.5 mg) in 27% yield; Colorless liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.28 – 7.23 (m, 2H), 7.02 (t, *J* = 7.5 Hz, 1H), 6.78 (d, *J* = 7.9 Hz, 1H), 3.26 (d, *J* = 15.4 Hz, 1H), 3.16 (d, *J* = 15.4 Hz, 1H), 3.06 (d, *J* = 12.6 Hz, 1H), 2.89 (d, *J* = 12.7 Hz, 1H), 2.47 – 2.38 (m, 1H), 1.87 – 1.78 (m, 2H), 1.73 – 1.67(m, 2H), 1.54 – 1.51 (m, 1H), 1.39 (s, 3H), 1.17 – 1.12 (m, 5H), 0.12 (s, 9H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 178.9, 143.9, 133.3, 127.9, 122.9, 121.9, 108.4, 48.7, 44.8, 37.8, 33.7, 33.4, 31.4, 26.0, 25.7, 23.3, -1.4; HRMS (ESI) m/z calced for C₂₀H₃₂NOSSi⁺ (M+H)⁺ 362.1968, found 362.1970.

ethyl 2-(3-((cyclohexylthio)methyl)-3-methyl-2-oxoindolin-1-yl)acetate (40)



The desired compound **40** was synthesized according to General Procedure using ethyl *N*-methacryloyl-*N*-phenylglycinate (49.4 mg, 0.2 mmol) and cyclohexanethiol (32.5 mg, 0.28 mmol) irradiated with blue LEDs for 48 h. Purification by column chromatography (silica gel, petroleum ether/ethyl acetate 10:1) was performed to give **40** (34.7 mg) in 48% yield; Colorless liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.34 (d, J = 7.4 Hz, 1H), 7.28 – 7.24 (m, 1H), 7.08 (t, J = 7.5 Hz, 1H), 6.74 (d, J = 7.8 Hz, 1H), 4.58 (d, J = 17.5 Hz, 1H), 4.38 (d, J = 17.5 Hz, 1H), 4.21 (q, J = 6.9 Hz, 2H), 3.09 (d, J = 12.8 Hz, 1H), 2.92 (d, J = 12.7 Hz, 1H), 2.47 – 2.42 (m, 1H), 1.90 – 1.81 (m, 2H), 1.70 – 1.68 (m, 3H), 1.47 (s, 3H), 1.24 (t, J = 7.3 Hz, 3H), 1.18 – 1.12 (m, 5H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.6, 167.6, 142.0, 132.7, 128.1, 123.4, 122.7, 108.0, 61.6, 48.9, 44.9, 41.3, 37.7, 33.7, 33.5, 26.0, 25.7, 23.0, 14.1; HRMS (ESI) m/z calced for C₂₀H₂₈NO₃S⁺ (M+H)⁺ 362.1784, found 362.1786.

1-((cyclohexylthio)methyl)-1-methyl-5,6-dihydro-4*H*-pyrrolo[3,2,1-*ij*]quinolin-2(1*H*)-one (4p)



The desired compound **4p** was synthesized according to General Procedure using 1-(3,4-dihydroquinolin-1(2H)-yl)-2-methylprop-2-en-1-one (40.2 mg, 0.2 mmol) and cyclohexanethiol (32.5 mg, 0.28 mmol) irradiated with blue LEDs for 36 h. Purification by column chromatography (silica gel, petroleum ether/ethyl acetate 10:1) was performed to give**4p**(46.0 mg) in 73% yield; Colorless liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.12 (d, *J* = 7.3 Hz, 1H), 7.03 (d, *J* = 7.1 Hz, 1H), 6.94 (t, *J* = 7.5 Hz, 1H), 3.72 (td, *J* = 5.6, 3.6 Hz, 2H), 3.05 (d, *J* = 12.7 Hz, 1H), 2.87 (d, *J* = 12.6 Hz, 1H), 2.81 – 2.77 (m, 2H), 2.47 – 2.41 (m, 1H), 2.00 (p, *J* = 6.2 Hz, 2H), 1.88 – 1.80 (m, 2H), 1.71 – 1.63 (m, 2H), 1.55 – 1.52 (m, 1H), 1.42 (s, 3H), 1.22 – 1.12 (m, 5H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 178.4, 139.1, 131.5, 126.9, 121.8, 120.9, 120.0, 50.2, 44.8, 38.8, 37.9, 33.8, 33.5, 26.0, 25.7, 24.6, 22.5, 21.2; HRMS (ESI) m/z calced for C₁₉H₂₆NOS⁺ (M+H)⁺ 316.1730, found 316.1733.

2-((cyclohexylthio)methyl)-2-methyl-6,7-dihydrobenzo[6,7]azepino[3,2,1-*hi*]indol-1(2*H*)-one (4q)



The desired compound 4q was synthesized according to General Procedure using 1-(10,11-dihydro-5*H*-dibenzo[*b*,*f*]azepin-5-yl)-2-methylprop-2-en-1-one (52.6 mg, 0.2 mmol) and cyclohexanethiol (32.5 mg, 0.28 mmol) irradiated with blue LEDs for 36 h. Purification by column chromatography (silica gel, petroleum ether/ CH₂Cl₂ 1:1) was performed to give 4q (65.7 mg) in 87% yield; Colorless liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.89 (d, *J* = 8.2 Hz, 1H), 7.30 (d, *J* = 6.8 Hz, 1H), 7.24 – 7.14 (m, 3H), 7.06 – 6.99 (m, 2H), 3.17 (d, *J* = 12.6 Hz, 1H), 3.11 – 3.00 (m, 5H), 2.48 – 2.39 (m, 1H), 1.93 – 1.79 (m, 2H), 1.70 – 1.65 (m, 2H), 1.55 – 1.49 (m, 4H), 1.20 – 1.13 (m, 5H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 180.1, 140.6, 136.7, 136.1, 133.1, 130.0, 129.4, 126.4, 126.3, 126.1, 125.0, 122.2, 120.6, 48.8, 44.7, 38.8, 33.8, 33.8, 33.6, 33.4, 25.9, 25.6, 23.8; HRMS (ESI) m/z calced for C₂₄H₂₈NOS⁺ (M+H)⁺ 378.1886, found 378.1888.

3-((cyclohexylthio)methyl)-1,3-dimethyl-1,3-dihydro-2H-pyrrolo[2,3-b]pyridin-2-one (4r)



The desired compound $4\mathbf{r}$ was synthesized according to General Procedure using *N*-methyl-*N*-(pyridin-2-yl)methacrylamide (35.2 mg, 0.2 mmol) and cyclohexanethiol (32.5 mg, 0.28 mmol) irradiated with blue LEDs for 48 h. Purification by column chromatography (silica gel, petroleum ether/ethyl acetate 10:1) was performed to give $4\mathbf{r}$ (40.0 mg) in 69% yield; Colorless liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 8.17 (dd, *J* = 5.3, 1.6 Hz, 1H), 7.53 (dd, *J* = 7.2, 1.6 Hz, 1H), 6.93 (dd, *J* = 7.3, 5.3 Hz, 1H), 3.29 (s, 3H), 3.04 (d, *J* = 12.8 Hz, 1H), 2.86 (d, *J* = 12.8 Hz, 1H), 2.48 – 2.42 (m, 1H), 1.87 – 1.76 (m, 2H), 1.68 – 1.65 (m, 2H), 1.54 – 1.52 (m, 1H), 1.42 (s, 3H), 1.19 – 1.14 (m, 5H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.2, 156.8, 146.9, 130.6, 127.4, 117.9, 48.6, 45.1, 37.5, 33.7, 33.4, 25.9, 25.9, 25.6, 25.3, 22.1; HRMS (ESI) m/z calced for C₁₆H₂₃N₂OS⁺ (M+H)⁺ 291.1526, found 291.1527.

2-(3-((cyclohexylthio)methyl)-3-methyl-2-oxoindolin-1-yl)ethyl (2S)-2-(6-methoxynaphthale n-2-yl)propanoate (4s, 1.08:1 *dr*)



The desired compound **4s** was synthesized according to General Procedure using 2-(*N*-phenylmethacrylamido)ethyl (*S*)-2-(6-methoxynaphthalen-2-yl)propanoate (83.5 mg, 0.2 mmol) and cyclohexanethiol (32.5 mg, 0.28 mmol) irradiated with blue LEDs for 36 h. Purification by column chromatography (silica gel, petroleum ether/ethyl acetate 6:1) was performed to give **4s** (56.3 mg) in 53% yield; Colorless liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.68 – 7.62 (m, 2H), 7.58 – 7.56 (m, 1H), 7.32 – 7.28 (m, 1H), 7.24 (d, *J* = 7.6 Hz, 1H), 7.16 – 7.09 (m, 3H), 7.02 (td, *J* = 7.7, 2.4 Hz, 1H), 6.79 (d, *J* = 7.7 Hz, 1H), 4.42 – 4.34 (m, 1H), 4.30 – 4.24 (m, 1H), 4.03 – 3.94 (m, 1H), 3.91 (s, 3H), 3.90 – 3.84 (m, 1H), 3.81 – 3.70 (m, 1H), 3.03 (dd, *J* = 12.7, 4.1 Hz, 1H), 2.86 (dd, *J* = 12.7, 2.1 Hz, 1H), 2.38 – 2.34 (m, 1H), 1.87 – 1.73 (m, 3H), 1.69 – 1.61 (m, 2H), 1.49 (dd, *J* = 7.2, 2.9 Hz, 3H), 1.37 (s, 1.56H), 1.33 (s, 1.44H), 1.20 – 1.07 (m, 5H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.6, 179.6, 174.5, 174.4, 157.6, 142.7, 142.6, 135.2, 135.2, 133.6, 132.7, 132.7, 129.2, 128.8, 128.0, 128.0, 127.1, 126.1, 126.1, 126.0, 125.9, 123.0, 123.0, 122.3, 118.9, 108.4, 108.3, 105.5, 62.0, 61.8, 55.3, 48.8, 48.8, 45.3, 45.3, 44.8, 38.8, 37.9, 33.7, 33.4, 26.0, 25.6, 22.8, 22.8, 18.3, 18.3; HRMS (ESI) m/z calced for C₃₂H₃₈NO₄S⁺ (M+H)⁺ 532.2516, found 532.2517.

2-(3-((cyclohexylthio)methyl)-3-methyl-2-oxoindolin-1-yl)ethyl 2-(4-isobutylphenyl)propano ate (4t, 1:1 *dr*)



The desired compound **4t** was synthesized according to General Procedure using 2-(N-phenylmethacrylamido) ethyl 2-(4-isobutylphenyl) propanoate (78.7 mg, 0.2 mmol) and cyclohexanethiol (32.5 mg, 0.28 mmol) irradiated with blue LEDs for 36 h. Purification by column chromatography (silica gel, petroleum ether/ethyl acetate 10:1) was performed to give **4r** (67.0 mg) in 66% yield; Colorless liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.29 – 7.20 (m, 2H), 7.12 – 7.03 (m, 5H), 6.86 (d, *J* = 7.8 Hz, 1H), 4.41 – 4.30 (m, 1H), 4.28 – 4.22 (m, 1H), 4.06 – 3.95 (m, 1H), 3.96 – 3.83 (m, 1H), 3.60 (p, *J* = 7.2 Hz, 1H), 3.05 (dd, *J* = 12.7, 3.6 Hz, 1H), 2.88 (d, *J* = 12.7 Hz, 1H), 2.43 (d, *J* = 7.2 Hz, 2H), 2.41 – 2.30 (m, 1H), 1.86 – 1.76 (m, 3H), 1.69 – 1.60 (m, 2H), 1.54 – 1.51 (m, 1H), 1.43 – 1.35 (m, 6H), 1.20 – 1.07 (m, 5H), 0.89 (d, *J* = 6.6 Hz, 6H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 179.6, 174.6, 174.5, 142.8, 142.7, 140.5, 137.4, 137.3, 132.8, 132.8, 129.3, 128.1, 127.1, 123.1, 123.1, 122.4, 108.5, 108.4, 61.8, 61.6, 48.9, 48.8, 45.0, 45.0, 44.8, 38.8, 38.8, 38.0, 37.9, 33.7, 33.5, 30.1, 26.0,

25.7, 22.9, 22.8, 22.4, 18.3, 18.3; HRMS (ESI) m/z calced for $C_{31}H_{42}NO_3S^+$ (M+Na)⁺ 508.2880, found 508.2882.

3-benzyl-3-((cyclohexylthio)methyl)-1-methylindolin-2-one (4u)



The desired compound 4u was synthesized according to General Procedure using 2-benzyl-*N*-methyl-*N*-phenylacrylamide (50.2 mg, 0.2 mmol) and cyclohexanethiol (32.5 mg, 0.28 mmol) irradiated with blue LEDs for 48 h. Purification by column chromatography (silica gel, petroleum ether/ethyl acetate 10:1) was performed to give 4u (46.0 mg) in 63% yield; Colorless liquid.

¹H NMR (400 MHz, CDCl₃-*d*) δ 7.26 (d, *J* = 7.7 Hz, 1H), 7.19 (t, *J* = 7.7 Hz, 1H), 7.06 – 7.00 (m, 4H), 6.84 – 6.84 (m, 2H), 6.59 (d, *J* = 7.8 Hz, 1H), 3.21 (d, *J* = 12.6 Hz, 1H), 3.14 (s, 2H), 3.05 (d, *J* = 12.6 Hz, 1H), 2.97 (s, 3H), 2.51 – 2.43 (m, 1H), 1.91 – 1.80 (m, 2H), 1.73 – 1.64 (m, 2H), 1.57 – 1.52 (m, 1H), 1.20 – 1.15 (m, 5H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 178.0, 143.9, 135.4, 130.0, 129.8, 128.2, 127.4, 126.4, 123.9, 121.9, 107.7, 55.0, 44.9, 43.1, 36.7, 33.7, 33.4, 26.0, 25.8, 25.6; HRMS (ESI) m/z calced for C₂₃H₂₈NOS⁺ (M+H)⁺ 366.1886, found 366.1888.

3-((cyclohexylthio)methyl)-1,3-dimethylindoline (5)



¹H NMR (400 MHz, CDCl₃-*d*) δ 7.11 (t, *J* = 7.6 Hz, 1H), 7.06 (d, *J* = 7.5 Hz, 1H), 6.70 (t, *J* = 7.3 Hz, 1H), 6.49 (d, *J* = 7.8 Hz, 1H), 3.44 (d, *J* = 8.9 Hz, 1H), 2.97 (d, *J* = 8.9 Hz, 1H), 2.82 (d, *J* = 12.1 Hz, 1H), 2.76 – 2.73 (m, 4H), 2.53 – 2.47 (m, 1H), 1.99 – 1.89 (m, 2H), 1.77 – 1.70 (m, 2H), 1.60 – 1.56 (m, 2H), 1.38 (s, 3H), 1.31 – 1.18 (m, 7H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 137.0, 128.0, 122.3, 117.8, 107.5, 67.3, 45.1, 44.5, 40.7, 35.8, 33.8, 33.8, 26.1, 25.8, 24.0; HRMS (ESI) m/z calced for C₁₇H₂₆NS⁺ (M+H)⁺ 276.1780, found 276.1781.

3-((cyclohexylsulfinyl)methyl)-1,3-dimethylindolin-2-one (6, 1:1 dr)



6': ¹H NMR (400 MHz, CDCl₃-*d*) δ 7.42 (d, *J* = 7.4 Hz, 1H), 7.32 (t, *J* = 7.1 Hz, 1H), 7.11 (t, *J* = 8.0 Hz, 1H), 6.89 (d, *J* = 7.8 Hz, 1H), 3.24 (s, 3H), 3.17 (d, *J* = 13.5 Hz, 1H), 3.08 (d, *J* = 13.4 Hz, 1H), 2.53 – 2.45 (m, 1H), 2.03 – 2.01 (m, 1H), 1.89 – 1.84 (m, 1H), 1.81 – 1.79 (m, 2H), 1.67 –

1.63 (m, 1H), 1.54 (s, 3H), 1.31 – 1.22 (m, 5H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 178.6, 142.7, 131.2, 128.7, 124.5, 123.0, 108.4, 60.0, 58.3, 46.7, 26.4, 26.3, 25.4, 25.3, 25.0, 24.0, 24.0, and **6'':** ¹H NMR (400 MHz, CDCl₃-*d*) δ 7.36 (d, *J* = 7.4 Hz, 1H), 7.32 (d, *J* = 9.0 Hz, 1H), 7.08 (t, *J* = 7.5 Hz, 1H), 6.87 (d, *J* = 7.7 Hz, 1H), 3.27 – 3.23 (m, 4H), 3.01 (d, *J* = 13.1 Hz, 1H), 2.58 – 2.48 (m, 1H), 2.09 – 2.04 (m, 1H), 1.89 – 1.79 (m, 3H), 1.69 – 1.64 (m, 1H), 1.53 (s, 3H), 1.32 – 1.23 (m, 5H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 178.8, 143.3, 131.3, 128.7, 123.8, 122.5, 108.5, 60.2, 56.7, 45.8, 26.5, 26.4, 25.4, 25.3, 25.0, 24.7, 24.0; HRMS (ESI) m/z calced for C₁₇H₂₄NO₂S⁺ (M+H)⁺ 306.1522, found 306.1524.

3-((cyclohexylsulfonyl)methyl)-1,3-dimethylindolin-2-one (7)



¹H NMR (400 MHz, CDCl₃-*d*) δ 7.39 (d, J = 7.4 Hz, 1H), 7.36 – 7.29 (m, 1H), 7.10 (t, J = 7.5 Hz, 1H), 6.89 (d, J = 7.8 Hz, 1H), 3.61 (d, J = 14.1 Hz, 1H), 3.47 (d, J = 14.0 Hz, 1H), 3.25 (s, 3H), 2.57 – 2.49 (m, 1H), 2.06 – 2.04 (m, 2H), 1.88 – 1.85 (m, 2H), 1.67 – 1.60 (m, 2H), 1.45 – 1.39 (m, 5H), 1.17 – 1.15 (m, 2H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 178.2, 143.3, 130.4, 128.8, 123.8, 122.5, 108.6, 62.9, 55.1, 45.2, 26.6, 25.1, 25.0, 24.9, 24.7; HRMS (ESI) m/z calced for $C_{17}H_{24}NO_3S^+$ (M+H)⁺ 322.1471, found 322.1474.

5a,10-dimethyl-2,3,5,5a,10,10a-hexahydro-[1,4]oxathiepino[7,6-b]indole (8)



¹H NMR (400 MHz, CDCl₃-*d*) δ 7.12 (t, J = 7.0 Hz, 1H), 6.94 (d, J = 7.3 Hz, 1H), 6.70 (t, J = 7.4 Hz, 1H), 6.43 (d, J = 7.8 Hz, 1H), 4.62 (s, 1H), 4.50 (d, J = 12.4 Hz, 1H), 3.71 (t, J = 13.2 Hz, 1H), 3.05 – 2.94 (m, 2H), 2.89 (s, 3H), 2.88 – 2.81 (m, 1H), 2.54 (d, J = 14.3 Hz, 1H), 1.37 (s, 3H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 149.0, 134.9, 128.0, 121.6, 117.6, 108.4, 105.2, 75.0, 53.1, 40.0, 36.1, 30.3, 24.8; HRMS (ESI) m/z calced for C₁₃H₁₈NOS⁺ (M+H)⁺ 236.1104, found 236.1106. **2-(((1,3-dimethyl-2-oxoindolin-3-yl)methyl)thio)acetaldehyde (9)**



¹H NMR (400 MHz, CDCl₃-*d*) δ 9.27 (t, *J* = 3.3 Hz, 1H), 7.30 (t, *J* = 7.7 Hz, 1H), 7.24 (d, *J* = 9.3 Hz, 1H), 7.08 (t, *J* = 7.5 Hz, 1H), 6.87 (d, *J* = 7.8 Hz, 1H), 3.23 (s, 3H), 3.02 (dd, *J* = 14.6, 3.1 Hz, 1H), 2.92 - 2.87 (m, 3H), 1.39 (s, 3H); ¹³C NMR (100 MHz, CDCl₃-*d*) δ 194.0, 178.9, 143.6, 132.0, 128.6, 123.0, 122.7, 108.3, 49.1, 42.2, 39.0, 26.3, 23.1; HRMS (ESI) m/z calced for C₁₃H₁₆NO₂S⁺ (M+H)⁺ 250.0896, found 250.0899.

6. References

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7. Copies of ¹H and ¹³C NMR spectra of all Products



¹H and ¹³C NMR spectra of 3a (CDCl₃)

¹H and ¹³C NMR spectra of 3b (CDCl₃)



¹H and ¹³C NMR spectra of 3c (CDCl₃)



¹H and ¹³C NMR spectra of 3d (CDCl₃)



¹H and ¹³C NMR spectra of 3e (CDCl₃)


¹H and ¹³C NMR spectra of 3f (CDCl₃)





¹H and ¹³C NMR spectra of 3g (CDCl₃)



¹H and ¹³C NMR spectra of 3h (CDCl₃)



¹H and ¹³C NMR spectra of 3i (CDCl₃)



¹H and ¹³C NMR spectra of 3j (CDCl₃)





¹H and ¹³C NMR spectra of 3k (CDCl₃)



¹H and ¹³C NMR spectra of 3l (CDCl₃)







S44

¹H and ¹³C NMR spectra of 3n (CDCl₃)



¹H and ¹³C NMR spectra of 30 (CDCl₃)



S46

¹H and ¹³C NMR spectra of 3p (CDCl₃)



¹H and ¹³C NMR spectra of 3q (CDCl₃)



¹H and ¹³C NMR spectra of 3r (CDCl₃)



¹H and ¹³C NMR spectra of 3s (CDCl₃)



¹H and ¹³C NMR spectra of 3t (CDCl₃)



fl (ppm)

¹H and ¹³C NMR spectra of 3u (CDCl₃)



S52

¹H and ¹³C NMR spectra of 4a (CDCl₃)



¹H and ¹³C NMR spectra of 4b (CDCl₃)



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## ¹H and ¹³C NMR spectra of 4c (CDCl₃)



## ¹H and ¹³C NMR spectra of 4d (CDCl₃)



## ¹H, ¹³C and ¹⁹F NMR spectra of 4e (CDCl₃)



S57



10 0 -10 -20 -30 -40 -50 -60 -70 -80 -90 -100 -110 -120 -130 -140 -150 -160 -170 -180 -190 -200 -210 fl (ppm)

## ¹H and ¹³C NMR spectra of 4f (CDCl₃)



## ¹H and ¹³C NMR spectra of 4g (CDCl₃)



## ¹H and ¹³C NMR spectra of 4h (CDCl₃)

7.7 7.7 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17 7.17Ӊс H,C С \ Сн₃ 'n r.r. = 2.7:1 0.740.270.270.730.730.26Hee.0 1.43₉ 2.61 0.73 = 0.730.26 = 0.733.30 0.82 5.02 0 7.0 6.5 6.0 3.5 9.5 9.0 8.5 8.0 7.5 5.5 5.0 4.5 4.0 3.0 2.5 2.0 1.5 1.0 0.5 0.0 fl (ppm)  $\int_{-\infty}^{\infty} \frac{145.59}{131.24}$   $\int_{-\infty}^{\infty} \frac{144.60}{131.24}$   $\int_{-\infty}^{\infty} \frac{131.24}{129.35}$   $\int_{-\infty}^{\infty} \frac{128.75}{123.27}$   $\sim 108.72$   $\sim 108.72$ 179.41 178.82 50.99 48.69 44.96 44.45 44.45 33.71 33.71 33.71 33.71 33.71 33.71 33.72 33.72 26.24 26.24 26.24 26.24 26.24 26.24 26.24 26.24 26.24 26.24 26.24 26.24 26.24 26.24 27.25 27.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.81 20.810 77.32 77.00 76.68 н₃с Ӊс 0 =0 CI `сњ 'n 00 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30  $\frac{1}{20}$ 10

## ¹H and ¹³C NMR spectra of 4i (CDCl₃)



## ¹H and ¹³C NMR spectra of 4j (CDCl₃)



## ¹H and ¹³C NMR spectra of 4k (CDCl₃)



## ¹H and ¹³C NMR spectra of 4l (CDCl₃)



## ¹H and ¹³C NMR spectra of 4m (CDCl₃)



## ¹H and ¹³C NMR spectra of 4n (CDCl₃)



## ¹H and ¹³C NMR spectra of 40 (CDCl₃)



S68

## ¹H and ¹³C NMR spectra of 4p (CDCl₃)

#### 7.7.26 7.111 7.112 7.111 7.112 7.111 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112 7.112



¹H and ¹³C NMR spectra of 4q (CDCl₃)



¹H and ¹³C NMR spectra of 4r (CDCl₃)



¹H and ¹³C NMR spectra of 4s (CDCl₃)



S72
¹H and ¹³C NMR spectra of 4t (CDCl₃)



S73

## ¹H and ¹³C NMR spectra of 4u (CDCl₃)



## ¹H and ¹³C NMR spectra of 5 (CDCl₃)



¹H and ¹³C NMR spectra of 6 (CDCl₃)







¹H and ¹³C NMR spectra of 7 (CDCl₃)



S78

## ¹H and ¹³C NMR spectra of 8 (CDCl₃)



## ¹H and ¹³C NMR spectra of 9 (CDCl₃)



S80