Supporting Information

For

Radical alkylation of isocyanides with amino acid-/peptide-derived Katritzky salts via photoredox catalysis

Ze-Fan Zhu, Miao-Miao Zhang and Feng Liu*

Jiangsu Key Laboratory of Neuropsychiatric Diseases and Department of Medicinal Chemistry, College of Pharmaceutical Sciences, Soochow University, 199 Ren-Ai Road, Suzhou, Jiangsu 215123, People’s Republic of China
E-mail: fliu2@suda.edu.cn

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1. General remarks

$^1$H NMR spectra were recorded on 400 or 600 MHz (100 or 150 MHz for $^{13}$C NMR, 376 or 564 MHz for $^{19}$F NMR) agilent NMR spectrometer with CDCl$_3$ as the solvent and tetramethylsilane (TMS) as the internal standard. Chemical shifts were reported in parts per million (ppm, δ scale) downfield from TMS at 0.00 ppm and referenced to the CDCl$_3$ at 7.26 ppm (for $^1$H NMR) or 77.16 ppm (for $^{13}$C NMR). $^{19}$F NMR chemical shifts were determined relative to CFCl$_3$ at δ 0.00 ppm. Mass spectroscopy data of the products were collected on a GCT PremierTM (CI) and Agilent Technologies 1290 Infinity (ESI). Mass Spectrometer Infrared (FT-IR) spectra were recorded on a Varian 1000FT-IR, $\nu_{\text{max}}$ in cm$^{-1}$. Melting points were measured using SGW, X-4B and values are uncorrected. All commercially available reagents and solvents were used as received unless otherwise specified. The substrates were readily prepared according to known methods. (Angew. Chem. Int. Ed. 2017, 56, 12336; Org. Lett. 2013, 15, 5520; Org. Chem. Front, 2017, 4, 2049).

2. Synthesis of pyridinium salts

![Reaction Scheme]

Synthesis of triphenylpyrylium tetrafluoroborate: Benzaldehyde (1 equiv) and acetophenone (2 equiv) were placed in a closed two-necked flask equipped with a magnetic stirrer, then boron trifluoride etherate (2.5 equiv) was added dropwise under argon treatment. The mixture was reacted at 100 °C for two hours and cooled to ambient temperature. Methyl tert-butyl ether was added to the reaction mixture and the resulting suspension stirred at ambient temperature. The solid was collected by filtration and washed with methyl tert-butyl ether. Recrystallization by acetone and methyl tert-butyl ether to get pure light yellow solid.

A closed flask equipped with a magnetic stirrer bar was charged with triphenylpyrylium tetrafluoroborate (1.0 equiv) and the corresponding primary amine (1.2 equiv). Ethanol (1.0 M) was added to the reaction vessel and the tube sealed. No precautions to protect the reaction mixture from air and moisture were taken. The reaction mixture was heated to 90 °C for 4 h and then cooled to ambient temperature. Methyl tert-butyl ether was added to the reaction mixture and the resulting suspension
stirred at room temperature. The solid was collected by filtration and washed with methyl tert-butyl ether. After the operations required the solids were dried under reduced pressure to obtain the analytically pure pyridinium salts.

Amine hydrochlorides as starting materials: In case amine hydrochlorides were used as feedstocks for the pyridinium salts, the amine hydrochloride (1.2 equiv) was added to a clean and closed flask. Ethanol (1.0 M) and triethyl amine (1.2 equiv) were added. The resulting suspension was stirred for 30 min at ambient temperature. Triphenylpyrylium tetrafluoroborate (1.0 equiv) was added, the tube sealed and stirred for 4 h at 90 °C. Methyl tert-butyl ether was added to the reaction mixture and the resulting suspension stirred at room temperature for at least 1 h to complete the precipitation process. The solid was collected by filtration and washed with methyl tert-butyl ether. After the operations required the solids were dried under reduced pressure to obtain the analytically pure pyridinium salts. To remove water-soluble impurities, the collected solids were washed with water before washing with methyl tert-butyl ether.

3. Typical experimental procedure

To a solution of 1a (27 mg, 0.15 mmol), 2a (167.2 mg, 0.3 mmol), Ru(bpy)_3Cl_2ꞏ6H_2O (5.6 mg, 0.0075 mmol) in CH_3CN (2 ml) was added K_2CO_3 (41.5 mg, 0.3 mmol) at room temperature under N_2 atmosphere. The resulting mixture was stirred for 12 hours upon 22W blue LEDs irradiation. The solvent was then removed under reduced pressure and the residue was purified by flash column chromatography on silica gel (petroleum ether/EtOAc = 100:1 to 30:1) to give 3a as a yellow solid (48 mg, 94% yield).
4. Gram-scale Reactions

To a solution of 1a (1.0 g, 5.6 mmol), 2a (6.0 g, 11.2 mmol), Ru(bpy)₃Cl₂·6H₂O (209 mg, 0.28 mmol) in CH₃CN (35 ml) was added K₂CO₃ (1.55 g, 11.2 mmol) at room temperature under N₂ atmosphere. The resulting mixture was stirred for 12 hours upon 22W blue LEDs irradiation. The solvent was then removed under reduced pressure and the residue was purified by flash column chromatography on silica gel (petroleum ether/EtOAc = 100:1 to 30:1) to give 3a as a yellow solid (1.73 g, 90% yield).

5. Mechanistic studies

5.1 Fluorescence quenching experiment

Emission intensities were recorded using LS55 Luminescence Spectrometer for all experiments. Acetonitrile was degassed with argon for at least 30 minutes by ultrasonic treatment. All Ru(bpy)₃Cl₂·6H₂O solutions were excited at 450 nm and the emission intensity was collected at 580-630 nm. In a typical experiment, the CH₃CN solution of Ru(bpy)₃Cl₂·6H₂O (0.04 μM) was added the appropriate amount of quencher in a screw-top 1.0cm quartz cuvette. After degassing with argon for 10 min, the emission spectra of the samples were collected. The results showed that pyridinium salt 2a quenched the photoexcited Ru(bpy)₃Cl₂·6H₂O effectively but isocyanobiphenyl 1a did not.
**Figure S1.** a) UV-vis absorption spectrum of Ru(bpy)$_3$Cl$_2$·6H$_2$O. b) Ru(bpy)$_3$Cl$_2$·6H$_2$O emission quenched by 2-isocyanobiphenyl 1a. c) Ru(bpy)$_3$Cl$_2$·6H$_2$O emission quenched by Katritzky salt 2a.

### 5.2 TEMPO-radical trapping experiment

When 2.0 equiv of TEMPO was added to the reaction of 1a with 2a under the standard conditions, no desired product (3a) was detected by TLC. A TEMPO-trapped product was determined by HRMS and NMR.

**Methyl 3-phenyl-2-((2,2,6,6-tetramethylpiperidin-1-yl)oxy)propanoate:** colorless oil; $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 7.32 (t, $J = 7.0$ Hz, 2H), 7.27 (d, $J = 6.8$ Hz, 1H), 7.25 – 7.19 (m, 2H), 4.52 (dd, $J = 10.0$, 5.4 Hz, 1H), 3.57 (s, 3H), 3.32 (dd, $J = 13.1$, 5.2 Hz, 1H), 3.11 – 3.01 (m, 1H), 1.57 – 1.45 (m, 4H), 1.42 – 1.32 (m, 2H), 1.30 (s, 3H), 1.19 (s, 6H), 1.09 (s, 3H); $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 172.9, 135.8, 129.2, 128.2, 126.5, 86.4, 60.4, 59.3, 50.9, 40.1, 40.0, 38.4, 33.4, 32.7, 20.1, 19.9, 16.9; HRMS (CI) calcd [M + H]$^+$: 320.2226, found: 320.2234.
5.3 Light on/off experiment

[Graph showing the profile of the reaction with light on/off over time. Yield was determined by $^1$H NMR.]


6 References for known products

<table>
<thead>
<tr>
<th>Entry</th>
<th>References</th>
<th>Compounds</th>
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<tbody>
<tr>
<td>1</td>
<td><em>Angew. Chem. Int. Ed.</em> 2017, 56, 12336–12339.</td>
<td>2a, 2b, 2d, 2f, 2h</td>
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7 Characterization of the substrates and products

**2a: (1-Methoxy-1-oxo-3-phenylpropan-2-yl)-2,4,6-triphenylpyridin-1-iumtetrafluoroborate**

\[
\begin{align*}
\text{H NMR (600 MHz, CDCl}_3) & \delta 7.93 (s, 2H), 7.86 – 7.69 (m, 4H), 7.60 (t, J = 7.2 Hz, 2H), 7.57 – 7.40 (m, 9H), 7.14 – 7.05 (m, 3H), 6.77 (d, J = 7.3 Hz, 2H), 5.64 (dd, J = 7.5, 3.7 Hz, 1H), 3.69 (s, 3H), 3.49 – 3.42 (m, 1H), 2.93 (dd, J = 14.4, 8.0 Hz, 1H); \\
\text{13C NMR (150 MHz, CDCl}_3) & \delta 168.0, 157.1, 157.0, 136.4, 133.8, 132.5, 132.4, 131.7, 129.8, 129.6, 129.2, 129.1, 128.72, 128.66, 128.0, 127.3, 70.3, 53.9, 37.8; \\
\text{19F NMR (564 MHz, CDCl}_3) & \delta -152.80 (d, J = 4.5 Hz), -152.86 (d, J = 3.5 Hz). 
\end{align*}
\]

**2b: (1-Methoxy-1-oxopropan-2-yl)-2,4,6-triphenylpyridin-1-iumtetrafluoroborate**

\[
\begin{align*}
\text{H NMR (400 MHz, CDCl}_3) & \delta 7.87 (s, 2H), 7.81 – 7.70 (m, 4H), 7.47 (t, J = 7.2 Hz, 2H), 5.52 (q, J = 7.0 Hz, 1H), 3.66 (s, 3H), 1.46 (d, J = 6.8 Hz, 3H); \\
\text{13C NMR (150 MHz, CDCl}_3) & \delta 168.9, 157.0, 156.8, 134.0, 132.7, 132.3, 131.5, 129.7, 129.3, 129.2, 129.1, 128.5, 127.9, 64.6, 53.8, 17.3; \\
\text{19F NMR (564 MHz, CDCl}_3) & \delta -153.17 (s), -153.22 (s). 
\end{align*}
\]

**2c: (1-Methoxy-3-methyl-1-oxobutan-2-yl)-2,4,6-triphenylpyridin-1-iumtetrafluoroborate**

\[
\begin{align*}
\text{H NMR (400 MHz, CDCl}_3) & \delta 7.98 (s, 2H), 7.87 (d, J = 7.2 Hz, 2H), 7.75 – 7.54 (m, 9H), 7.53 – 7.46 (m, 4H), 5.14 (d, J = 10.2 Hz, 1H), 3.74 (s, 3H), 2.12 – 2.01 (m, 1H), 0.73 (d, J = 6.3 Hz, 3H), 0.71 (d, J = 7.2 Hz, 3H); \\
\text{13C NMR (150 MHz, CDCl}_3) & \delta -153.17 (s), -153.22 (s). 
\end{align*}
\]
CDCl₃) δ 167.0, 157.2, 157.1, 133.1, 133.0, 132.0, 130.0, 129.6, 129.5, 129.0, 128.8, 127.9, 73.7, 53.9, 30.1, 22.4, 19.3; ¹⁹F NMR (564 MHz, CDCl₃) δ -153.22 (s), -153.28 (s); HRMS (ESI) calcd C₂₉H₂₈NO₂ [M-BF₄]+: 422.2115, found: 422.2114.

(1-Methoxy-4-methyl-1-oxopentan-2-yl)-2,4,6-triphenylpyridin-1-iumtetrafluoroborate (2d): ¹H NMR (400 MHz, CDCl₃) δ 7.89 (s, 2H), 7.80 (d, J = 7.3 Hz, 2H), 7.70 (br, 2H), 7.62 – 7.52 (m, 7H), 7.51 – 7.45 (m, 2H), 5.46 (dd, J = 7.8, 2.8 Hz, 1H), 3.74 (s, 3H), 1.76 – 1.64 (m, 1H), 1.62 – 1.50 (m, 1H), 1.35 – 1.24 (m, 1H), 0.56 (d, J = 6.5 Hz, 3H), 0.41 (d, J = 6.4 Hz, 3H); ¹³C NMR (150 MHz, CDCl₃) δ 168.7, 156.8, 156.7, 133.7, 132.5, 131.6, 129.6, 129.4, 129.1, 128.5, 127.9, 67.4, 53.9, 40.4, 26.1, 22.3, 20.7. ¹⁹F NMR (564 MHz, CDCl₃) δ -153.01 (s), -153.07 (s).

(1-Methoxy-3-methyl-1-oxopentan-2-yl)-2,4,6-triphenylpyridin-1-iumtetrafluoroborate (2e): Two isomers, d.r.: 1:1; ¹H NMR (600 MHz, CDCl₃) δ 8.00 (s, 2H), 7.93 – 7.87 (m, 2H), 7.68 (s, 1H), 7.65 – 7.58 (m, 7H), 7.56 – 7.46 (m, 5H), 5.32 (d, J = 10.2 Hz, 0.5H)/5.18 (d, J = 10.2 Hz, 0.6H), 3.77 (s, 1.6H)/ 3.74 (s, 1.3H), 2.02 – 1.93 (m, 0.6H)/ 1.75 – 1.66 (m, 0.9H), 1.41 – 1.34 (m, 0.6H)/ 1.01 – 0.91 (m, 0.9H), 1.30 – 1.18 (m, 1.6H)/ 0.50 – 0.43 (m, 1.3H), 0.84 (t, 2.3H)/ 0.70 (t, J = 7.6 Hz, 3H), 0.82 – 0.75 (m, 1H); ¹³C NMR (150 MHz, CDCl₃) δ 167.0, 166.8, 157.10, 157.06, 133.1, 133.0, 131.9, 129.9, 129.8, 129.7, 129.4, 129.2 128.9, 128.8, 128.6, 127.8, 73.5, 71.3, 53.93, 53.87, 36.0, 35.6, 27.8, 25.4, 18.5, 15.2, 11.1, 9.5; ¹⁹F NMR (564 MHz, CDCl₃) δ -153.28 (s), -153.34 (s); HRMS (ESI) calcd C₃₀H₃₀NO₂ [M-BF₄]⁺: 436.2271, found: 436.2270.

(1-Methoxy-4-(methylthio)-1-oxobutan-2-yl)-2,4,6-triphenylpyridin-1-iumtetrafluoroborate (2f): ¹H NMR (400 MHz, CDCl₃) δ 7.87 (s, 2H), 7.74 (dd, J = 31.7, 8.1 Hz, 4H), 7.63 – 7.50 (m, 7H), 7.46 (t, J = 7.3 Hz, 2H), 5.92 (dd, J = 7.7 Hz, 1H), 3.73 (s, 3H), 2.37 – 2.16 (m, 3H), 1.95 – 1.85 (m, 1H), 1.84 (s, 3H); ¹³C NMR (150 MHz,
CDCl₃) δ 168.4, 156.9, 133.8, 132.5, 132.2, 131.5, 129.6, 129.1, 128.5, 66.7, 53.9, 31.4, 30.8, 14.7; ¹⁹F NMR (564 MHz, CDCl₃) δ -152.74 (s), -152.79 (s).

1-(1-Methoxy-3-(4-methoxyphenyl)-1-oxopropan-2-yl)-2,4,6-triphenylpyridin-1-i
um tetrafluoroborate (2g): ¹H NMR (600 MHz, CDCl₃) δ 7.90 (s, 2H), 7.84 – 7.68
(m, 4H), 7.59 (t, J = 7.4 Hz, 2H), 7.56 – 7.50 (m, 5H), 7.50 – 7.36 (m, 4H), 6.67 (d, J = 8.2 Hz, 2H), 6.60 (d, J = 7.8 Hz, 2H), 5.58 (dd, J = 7.1, 4.9 Hz, 1H), 3.68 (d, J = 9.9 Hz, 6H), 3.31 (dd, J = 14.5, 4.3 Hz, 1H), 2.89 (dd, J = 14.6, 7.7 Hz, 1H); ¹³C NMR (150 MHz, CDCl₃) δ 167.8, 158.7, 156.9, 133.6, 132.5, 132.2, 131.6, 130.1, 129.7, 129.5, 129.2, 128.6, 128.0, 127.8, 114.0, 70.4, 55.3, 53.8, 36.9; ¹⁹F NMR (564 MHz, CDCl₃) δ -152.75 – -152.79 (m), -152.80 – -152.85 (m); HRMS (ESI) calcd C₃₄H₃₀NO₃ [M-BF₄]⁺: 500.2220, found: 500.2217.

1-(1-Methoxy-1-oxo-4-phenylbutan-2-yl)-2,4,6-triphenylpyridin-1-i
um tetrafluoroborate (2h): ¹H NMR (600 MHz, CDCl₃) δ 7.90 (s, 2H), 7.84 – 7.65
(m, 4H), 7.59 – 7.43 (m, 11H), 7.16 – 7.09 (m, 3H), 6.92 (d, J = 6.5 Hz, 2H), 5.37 (dd, J = 8.5, 2.1 Hz, 1H), 3.71 (s, 3H), 2.48 – 2.36 (m, 3H), 2.03 – 1.96 (m, 1H); ¹³C NMR (150 MHz, CDCl₃) δ 168.7, 157.1, 156.9, 138.7, 134.0, 132.6, 132.4, 131.5, 129.8, 129.2, 128.71, 128.65, 128.56, 128.1, 126.6, 68.1, 53.8, 33.5, 33.2; ¹⁹F NMR (564 MHz, CDCl₃) δ -152.86 (s), -152.92 (s).

1-(1,4-Dimethoxy-1,4-dioxobutan-2-yl)-2,4,6-triphenylpyridin-1-i
um tetrafluoroborate (2i): ¹H NMR (400 MHz, CDCl₃) δ 7.93 (s, 3H), 7.79 (t, J = 11.8 Hz, 3H), 7.73 – 7.37 (m, 11H), 6.22 (d, J = 9.4 Hz, 1H), 3.63 (s, 3H), 3.53 (s, 3H), 3.38 (dd, J = 17.4 Hz, 1H), 2.55 (dd, J = 17.4, 9.7 Hz, 1H); ¹³C NMR (150 MHz, CDCl₃) δ 169.6, 167.74, 167.70, 157.4, 133.9, 132.5, 131.7, 129.8, 129.4, 128.6, 64.1, 54.1, 52.6, 36.0;
$^{19}$F NMR (564 MHz, CDCl$_3$) $\delta$ -152.78 (d, $J = 4.1$ Hz), -152.83 (s); HRMS (ESI) calcd C$_{29}$H$_{26}$NO$_4$ [M-BF$_4$]$^+$: 452.1856, found: 452.1853.

1-(1,5-Dimethoxy-1,5-dioxopentan-2-yl)-2,4,6-triphenylpyridin-1-iumtetrafluoroborate (2j): $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.93 (s, 2H), 7.83 (d, $J = 7.7$ Hz, 2H), 7.80 – 7.52 (m, 11H), 7.49 (t, $J = 7.5$ Hz, 2H), 5.60 (t, $J = 6.2$ Hz, 1H), 3.71 (s, 3H), 3.46 (s, 3H), 2.26 (tt, $J = 13.5$, 6.9 Hz, 1H), 2.21 – 2.14 (m, 2H), 2.06 (tt, $J = 14.6$, 7.3 Hz, 1H); $^{13}$C NMR (150MHz, CDCl$_3$) $\delta$ 172.1, 168.2, 157.2, 133.7, 132.5, 132.4, 131.7, 129.8, 129.4, 128.6, 128.1, 67.6, 54.0, 51.8, 30.8, 27.0; $^{19}$F NMR (564 MHz, CDCl$_3$) $\delta$ -153.06 (s), -153.11 (s); HRMS (ESI) calcd C$_{30}$H$_{28}$NO$_4$ [M-BF$_4$]$^+$: 466.2013, found: 466.2012.

$^{19}$F NMR (564 MHz, CDCl$_3$) $\delta$ -152.78 (d, $J = 4.1$ Hz), -152.83 (s); HRMS (ESI) calcd C$_{29}$H$_{26}$NO$_4$ [M-BF$_4$]$^+$: 452.1856, found: 452.1853.

1-(3-(4-hydroxyphenyl)-1-methoxy-1-oxopropan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate(2k): $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.92 (s, 2H), 7.86 – 7.83 (m, 2H), 7.79 – 7.46 (m, 13H), 6.62 (d, $J = 8.5$ Hz, 2H), 6.44 (d, $J = 8.4$ Hz, 2H), 5.60 (t, $J = 6.7$ Hz, 1H), 3.70 (s, 3H), 3.15 (dd, $J = 14.8$, 7.1 Hz, 1H), 2.89 (dd, $J = 14.8$, 6.4 Hz, 1H); $^{13}$C NMR (151 MHz, CDCl$_3$) $\delta$ 168.2, 157.1, 156.3, 132.9, 132.2, 131.9, 130.0, 129.8, 129.4, 128.7,116.3, 70.9, 53.9, 36.7; $^{19}$F NMR (564 MHz, CDCl$_3$) $\delta$ -151.85 (s), -151.90 (s); HRMS (ESI) calcd C$_{33}$H$_{28}$NO$_3$ [M-BF$_4$]$^+$: 486.2064, found: 486.2062.

1-(tert-Butoxy)-1-oxo-3-phenylpropan-2-yl)-2,4,6-triphenylpyridin-1-iumtetrafluoroborate (2l): $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.95 (d, $J = 2.9$ Hz, 2H), 7.88 – 7.78 (m, 3H), 7.79 – 7.44 (m, 12H), 7.07 (dd, $J = 9.3$, 5.7 Hz, 3H), 6.83 – 6.76 (m, 2H), 5.55 (dd, $J = 8.3$, 2.8 Hz, 1H), 3.45 – 3.39 (m, 1H), 2.48 – 2.41 (m, 1H), 1.35 (s, 9H); $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 167.84, 167.80, 156.8, 146.0, 137.3, 137.2, 134.0, 133.0, 132.4, 131.5, 129.8, 128.9, 128.6, 128.5, 127.0, 85.6, 72.0, 37.8, 27.8; $^{19}$F
NMR (564 MHz, CDCl₃) δ -152.65 (s), -152.70 (s); HRMS (ESI) calcd C₃₆H₃₄NO₂ [M-BF₄]⁺: 512.2584, found: 512.2586.

1-(1-(Diethylamino)-1-oxo-3-phenylpropan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (2m): ¹H NMR (600 MHz, CDCl₃) δ 8.04 (br, 2H), 7.85 (s, 2H), 7.76 (d, J = 7.7 Hz, 2H), 7.74 – 7.49 (m, 9H), 7.46 (t, J = 7.6 Hz, 2H), 7.16 – 7.07 (m, 3H), 6.87 (d, J = 7.5 Hz, 2H), 5.96 (dd, J = 9.5, 3.4 Hz, 1H), 3.53 (dd, J = 14.3, 3.2 Hz, 1H), 3.23 – 3.04 (m, 2H), 2.96 – 2.85 (m, 1H), 2.26 – 2.10 (m, 2H), 0.98 (t, J = 6.2 Hz, 3H), 0.37 (t, J = 6.3 Hz, 3H); ¹³C NMR (150 MHz, CDCl₃) δ 165.2, 157.7, 155.8, 145.8, 135.8, 133.9, 133.6, 132.4, 131.2, 129.9, 129.7, 129.0, 128.93, 128.87, 128.5, 128.4, 127.4, 70.9, 41.3, 40.6, 39.1, 12.8, 12.2; ¹⁹F NMR (564 MHz, CDCl₃) δ -152.75 (s), -152.80 (s); HRMS (ESI) calcd C₃₆H₃₅N₂O [M-BF₄]⁺: 511.2744, found: 511.2743.

1-(1-Oxo-3-phenyl-1-(piperidin-1-yl)propan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (2n): ¹H NMR (600 MHz, CDCl₃) δ 8.27 (br, 2H), 7.83 (s, 2H), 7.77 (d, J = 7.8 Hz, 2H), 7.65 – 7.43 (m, 11H), 7.14 (t, J = 7.4 Hz, 2H), 7.10 (t, J = 7.2 Hz, 1H), 6.99 (d, J = 7.6 Hz, 2H), 6.18 (dd, J = 10.5, 3.1 Hz, 1H), 3.79 – 3.68 (m, 1H), 3.61 (dd, J = 13.5, 2.7 Hz, 1H), 2.86 – 2.73 (m, 1H), 2.47 (dd, J = 13.0, 11.1 Hz, 1H), 1.98 – 1.88 (m, 1H), 1.62 – 1.51 (m, 1H), 1.47 – 1.38 (m, 1H), 1.34 – 1.08 (m, 3H), 0.67 – 0.53 (m, 1H), 0.28 – 0.13 (m, 1H); ¹³C NMR (150 MHz, CDCl₃) δ 165.9, 157.8, 155.4, 135.7, 134.6, 133.8, 132.3, 131.0, 129.9, 129.7, 129.5, 129.0, 128.4, 128.3, 127.6, 71.2, 46.1, 43.8, 39.6, 24.9, 24.5, 23.5; ¹⁹F NMR (564 MHz, CDCl₃) δ -152.70 (s), -152.75 (s); HRMS (ESI) calcd C₃₇H₃₅N₂O [M-BF₄]⁺: 523.2744, found: 523.2739.

1-(1-((2-methoxy-2-oxoethyl)amino)-1-oxo-3-phenylpropan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (2o): ¹H NMR (600 MHz, CDCl₃) δ 7.89 (s, 2H), 7.79 (d, J = 7.7 Hz, 2H), 7.64 (br, 2H), 7.61 – 7.36 (m, 11H), 7.20 (t, J = 7.3 Hz, 1H), 7.14 (t, J = 7.4 Hz, 2H), 6.69 (d, J = 7.5 Hz, 2H), 5.74 (t, J = 7.0 Hz, 1H), 4.01 – 3.88
(m, 2H), 3.70 (s, 3H), 3.26 – 3.15 (m, 2H); $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 169.3, 167.0, 157.9, 156.6, 135.6, 133.7, 132.63, 132.56, 131.6, 129.9, 129.7, 129.3, 129.2, 128.8, 128.5, 128.0, 127.8, 71.4, 52.4, 41.9, 36.6; $^{19}$F NMR (564 MHz, CDCl$_3$) $\delta$ -152.45 (s), -152.50 (s); HRMS (ESI) calcd C$_{35}$H$_{31}$N$_2$O$_3$ [M-BF$_4$]$^+$: 527.2329, found: 527.2330.

1-(1-((1-methoxy-1-oxopropan-2-yl)amino)-1-oxo-3-phenylpropan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (2p): Two isomers, $d.r.$: 1:3:1; $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 7.94 (s, 2H), 7.83 (d, $J$ = 8.2 Hz, 2H), 7.77 – 7.71 (m, 3H), 7.65 – 7.51 (m, 10H), 7.19 – 7.13 (m, 3H), 6.77 – 6.72 (m, 2H), 5.79 (dd, $J$ = 9.0, 4.3 Hz, 0.64H)/5.74 (dd, $J$ = 8.1, 5.6 Hz, 0.5H), 4.35 – 4.25 (m, 1H), 3.74 (s, 1.3H)/3.63 (s, 1.7H), 3.65 (dd, $J$ = 14.9, 4.3 Hz, 0.81H)/3.48 (dd, $J$ = 15.0, 5.6 Hz, 0.64H), 2.85 (dd, $J$ = 14.9, 8.2 Hz, 0.57H)/2.59 (dd, $J$ = 14.9, 9.1 Hz, 0.75H), 1.33 (d, $J$ = 7.1 Hz, 3H); $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 172.3, 171.9, 167.7, 166.8, 157.4, 157.0, 156.8, 136.0, 136.0, 134.2, 133.2, 133.0, 132.4, 131.5, 129.8, 129.6, 129.5, 129.3, 129.2, 128.7, 128.6, 128.6, 128.2, 128.0, 127.9, 71.7, 71.4, 52.70, 52.65, 49.4, 37.3, 37.2, 29.9, 17.7, 17.2; $^{19}$F NMR (564 MHz, CDCl$_3$) $\delta$ -152.21 (s), -152.26 (s); HRMS (ESI) calcd C$_{36}$H$_{33}$N$_2$O$_3$ [M-BF$_4$]$^+$: 541.2486, found: 541.2484.

Methyl 2-(phenanthridin-6-yl)-3-phenylpropanoate (3a): Yellow solid; m.p. 126-127 °C; 94% yield (48 mg); $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.63 (d, $J$ = 8.2 Hz, 1H), 8.53 (d, $J$ = 8.1 Hz, 1H), 8.30 – 8.21 (m, 2H), 7.81 (t, $J$ = 7.5 Hz, 1H), 7.74 (t, $J$ = 7.4 Hz, 1H), 7.70 – 7.61 (m, 2H), 7.33 (d, $J$ = 7.2 Hz, 2H), 7.24 (t, $J$ = 7.2 Hz, 2H), 7.17 (t, $J$ = 7.0 Hz, 1H), 5.03 (t, $J$ = 6.1 Hz, 1H), 3.79 (dd, $J$ = 13.8, 8.1 Hz, 1H), 3.68 – 3.58 (m, 4H); $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 172.7, 157.4, 143.5, 139.8, 133.3, 130.5, 129.2, 128.7, 128.4, 127.6, 127.1, 126.4, 125.6, 125.2, 123.8, 122.7, 121.9, 52.36, 52.32, 37.2; FT-IR (thin film, KBr): $\nu$ (cm$^{-1}$) 2918, 1745, 1722, 1161, 751; HRMS (CI) calcd C$_{23}$H$_{20}$NO$_2$ [M + H]$^+$: 342.1494, found: 342.1489.
Methyl 2-(2-chlorophenanthridin-6-yl)-3-phenylpropanoate (3b): Yellowish solid; m.p. 115-116 °C; 88% yield (49 mg); ¹H NMR (400 MHz, CDCl₃) δ 8.51 (d, J = 8.3 Hz, 1H), 8.47 (s, 1H), 8.25 (d, J = 8.3 Hz, 1H), 8.15 (d, J = 8.7 Hz, 1H), 7.81 (t, J = 7.6 Hz, 1H), 7.71 – 7.65 (m, 2H), 7.29 (d, J = 7.6 Hz, 2H), 7.23 (t, J = 7.4 Hz, 2H), 7.16 (t, J = 7.3 Hz, 1H), 4.99 (t, J = 7.3 Hz, 1H), 3.75 (dd, J = 14.0, 8.0 Hz, 1H), 3.66 (s, 3H), 3.60 (dd, J = 14.0, 6.6 Hz, 1H); ¹³C NMR (150 MHz, CDCl₃) δ 172.6, 157.7, 142.0, 139.6, 133.0, 132.3, 131.9, 130.8, 129.3, 129.2, 128.5, 128.3, 126.5, 125.7, 125.4, 124.9, 122.7, 121.7, 52.4, 52.3, 37.1; FT-IR (thin film, KBr): ν ( cm⁻¹ ) 1733, 1537, 1150, 766, 695; HRMS (CI) calcld C₂₃H₁₉₃₅ClNO₂ [M + H]⁺: 376.1104, found: 376.1098.

Methyl 3-phenyl-2-(2-phenylphenanthridin-6-yl)propanoate (3c): Yellow solid; m.p. 147-148 °C; 70% (42 mg); ¹H NMR (400 MHz, CDCl₃) δ 8.76 – 8.66 (m, 2H), 8.31 – 8.21 (m, 2H), 7.96 (d, J = 8.3 Hz, 1H), 7.85 – 7.73 (m, 3H), 7.66 (t, J = 7.6 Hz, 1H), 7.51 (t, J = 7.5 Hz, 2H), 7.41 (t, J = 7.2 Hz, 1H), 7.30 (d, J = 7.3 Hz, 2H), 7.22 (t, J = 7.3 Hz, 2H), 7.14 (t, J = 7.1 Hz, 1H), 5.00 (t, J = 7.2 Hz, 1H), 3.77 (dd, J = 13.9, 8.0 Hz, 1H), 3.64 (s, 3H), 3.62 – 3.57 (m, 1H); ¹³C NMR (150 MHz, CDCl₃) δ 172.7, 157.4, 143.0, 141.1, 140.0, 139.8, 133.4, 130.9, 130.5, 129.2, 129.1, 128.53, 128.47, 128.2, 127.74, 127.69, 126.4, 125.7, 125.4, 124.0, 122.7, 120.3, 52.40, 52.37, 37.2; FT-IR (thin film, KBr): ν (cm⁻¹) 1730, 1251, 1212, 1149, 760; HRMS (CI) calcld C₂₉H₂₄NO₂ [M + H]⁺: 418.1807, found: 418.1806.

Methyl 2-(8-fluorophenanthridin-6-yl)-3-phenylpropanoate (3d): White solid; m.p. 121-122 °C; 85% (46 mg); ¹H NMR (400 MHz, CDCl₃) δ 8.54 (dd, J = 8.9, 5.4 Hz, 1H), 8.40 (d, J = 8.9 Hz, 1H), 8.21 (d, J = 8.1 Hz, 1H), 7.84 (d, J = 9.4 Hz, 1H), 7.70 (t, J = 7.5 Hz, 1H), 7.61 (t, J = 7.6 Hz, 1H), 7.49 (t, J = 8.4 Hz, 1H), 7.26 (d, J = 9.4 Hz, 2H), 7.20 (d, J = 7.4 Hz, 2H), 7.13 (t, J = 7.3 Hz, 1H), 4.85 (t, J = 7.3 Hz, 1H), 3.74 (dd, J = 13.9, 7.7 Hz, 1H), 3.66 – 3.57 (m, 4H); ¹⁳C NMR (150 MHz, CDCl₃) δ 172.4, 161.5 (d, J = 248.5 Hz), 156.4 (d, J₇₃CF = 3.8 Hz), 143.2, 139.5, 130.6, 129.9, 129.2, 128.6, 128.5, 127.3, 126.6 (d, J₇₃CF = 7.6 Hz), 125.2 (d, J₇₃CF = 8.5 Hz), 123.4, 121.7, 119.6 (d, J₇₃CF = 23.8 Hz), 110.3 (d, J = 22.0 Hz), 52.42, 52.35, 37.0; ¹⁹F NMR (564 MHz, CDCl₃) δ -111.40 – -111.47 (m, 1F); FT-IR (thin film, KBr): ν (cm⁻¹) 2923, 2227, 1739, 1209, 757; HRMS (CI) calcld C₂₃H₁₉FNO₂ [M + H]⁺: 360.1400, found: 360.1396.
Methyl 2-(8-methoxyphenanthridin-6-yl)-3-phenylpropanoate (3e): Yellowish solid; m.p. 124-125 °C; 76% (42 mg); \(^1\)H NMR (600 MHz, CDCl\(_3\)) δ 8.48 (d, \(J = 9.0\) Hz, 1H), 8.40 (d, \(J = 8.1\) Hz, 1H), 8.17 (d, \(J = 8.1\) Hz, 1H), 7.64 (t, \(J = 7.5\) Hz, 1H), 7.58 (t, \(J = 7.5\) Hz, 1H), 7.51 (d, \(J = 2.1\) Hz, 1H), 7.39 (dd, \(J = 9.0, 2.4\) Hz, 1H), 7.27 (d, \(J = 7.3\) Hz, 2H), 7.19 (t, \(J = 16.6, 9.0\) Hz, 2H), 7.13 (t, \(J = 7.3\) Hz, 1H), 4.86 (t, \(J = 7.2\) Hz, 1H), 3.88 (s, 3H), 3.75 (dd, \(J = 14.0, 8.0\) Hz, 1H), 3.62 (s, 3H), 3.57 (dd, \(J = 14.0, 6.4\) Hz, 1H); \(^{13}\)C NMR (150 MHz, CDCl\(_3\)) δ 172.8, 158.8, 156.5, 142.8, 139.9, 130.4, 129.2, 128.5, 127.7, 127.6, 127.1, 126.5, 126.4, 124.3, 124.0, 121.5, 121.1, 105.8, 55.6, 52.9, 52.3, 37.1; FT-IR (thin film, KBr): ν (cm\(^{-1}\)) 2950, 1718, 1221, 1162, 703; HRMS (CI) calcd C\(_{24}\)H\(_{22}\)NO\(_3\) [M + H]\(^+\): 372.1600, found: 372.1613.

Methyl 3-phenyl-2-(8-(trifluoromethoxy)phenanthridin-6-yl)propanoate (3f): Yellowish solid; m.p. 98-99 °C; 67% (43 mg); \(^1\)H NMR (400 MHz, CDCl\(_3\)) δ 8.67 (d, \(J = 9.0\) Hz, 1H), 8.50 (d, \(J = 8.0\) Hz, 1H), 8.24 (d, \(J = 8.0\) Hz, 1H), 8.06 (s, 1H), 7.77 (t, \(J = 7.4\) Hz, 1H), 7.68 (t, \(J = 8.8\) Hz, 2H), 7.27 (d, \(J = 7.6\) Hz, 2H), 7.21 (t, \(J = 7.2\) Hz, 2H), 7.14 (t, \(J = 6.8\) Hz, 1H), 4.88 (t, \(J = 7.2\) Hz, 1H), 3.75 (dd, \(J = 13.9, 6.9\) Hz, 1H), 3.67 (s, 3H), 3.57 (dd, \(J = 13.9, 8.0\) Hz, 1H); \(^{13}\)C NMR (150 MHz, CDCl\(_3\), overlapping peaks) δ 172.4, 156.6, 148.0, 143.7, 139.4, 131.8, 130.7, 129.2, 128.5, 127.7, 126.6, 126.1, 125.0, 124.0, 123.1, 122.0, 122.0, 120.7 (q, \(J = 258.3\) Hz), 117.0, 52.5, 37.1; \(^{19}\)F NMR (564 MHz, CDCl\(_3\)) δ -57.77 (s, 3F). FT-IR (thin film, KBr): ν (cm\(^{-1}\)) 3030, 1727, 1191, 1153, 760; HRMS (CI) calcd C\(_{24}\)H\(_{19}\)F\(_3\)NO\(_3\) [M + H]\(^+\): 426.1317, found: 426.1308.

Methyl 2-(8-cyanophenanthridin-6-yl)-3-phenylpropanoate (3g): Yellow solid; m.p. 166-167 °C; 65% (36 mg); \(^1\)H NMR (600 MHz, CDCl\(_3\)) δ 8.68 (d, \(J = 8.5\) Hz, 1H), 8.52 (d, \(J = 8.2\) Hz, 1H), 8.47 (s, 1H), 8.27 (d, \(J = 8.1\) Hz, 1H), 7.95 (d, \(J = 8.5\) Hz, 1H), 7.85 (t, \(J = 7.6\) Hz, 1H), 7.73 (t, \(J = 7.5\) Hz, 1H), 7.24 – 7.16 (m, 4H), 7.13 (t, \(J = 6.9\) Hz, 1H), 4.92 (t, \(J = 7.3\) Hz, 1H), 3.75 (dd, \(J = 13.9, 6.9\) Hz, 1H), 3.67 (s, 3H), 3.62 (dd, \(J = 13.9, 7.7\) Hz, 1H); \(^{13}\)C NMR (150 MHz, CDCl\(_3\)) δ 172.1, 156.7, 144.5,
139.1, 135.8, 131.6, 131.2, 130.8, 130.6, 129.2, 128.6, 128.0, 126.7, 124.9, 123.9, 122.5, 118.6, 111.0, 52.6, 51.9, 37.2; FT-IR (thin film, KBr): ν (cm\(^{-1}\)) 2923, 1727, 1144, 760, 697; HRMS (CI) calcd C\(_{24}\)H\(_{19}\)N\(_2\)O\(_2\) [M + H]: 367.1447, found: 367.1438.

**Methyl 2-(7,9-difluorophenanthridin-6-yl)-3-phenylpropanoate (3h):** Light green solid; m.p. 95-96 °C; 88% (49 mg); \(^1\)H NMR (400 MHz, CDCl\(_3\)) δ 8.35 (d, \(J = 7.9\) Hz, 1H), 8.16 (d, \(J = 7.8\) Hz, 1H), 8.07 (d, \(J = 9.5\) Hz, 1H), 7.78 (t, \(J = 7.1\) Hz, 1H), 7.66 (t, \(J = 7.1\) Hz, 1H), 7.35 (d, \(J = 6.5\) Hz, 2H), 7.22 (t, \(J = 6.7\) Hz, 2H), 7.19 - 7.09 (m, 2H), 5.13 (t, \(J = 6.0\) Hz, 1H), 3.70 (s, 3H), 3.57 (d, \(J = 6.0\) Hz, 2H); \(^{13}\)C NMR (150 MHz, CDCl\(_3\)) δ 172.8, 163.1 (dd, \(J = 253.0\), 14.1 Hz), 161.0 (dd, \(J = 256.7\), 13.1 Hz), 154.7 (d, \(J = 6.4\) Hz), 143.5, 139.9, 137.4 (dd, \(J = 10.5\), 4.7 Hz), 130.6, 130.1, 129.5, 128.3, 127.7, 126.3, 122.4, 122.0 (t, \(J = 3.6\) Hz), 112.5 (d, \(J = 11.7\) Hz), 104.4 (dd, \(J = 21.9\), 3.9 Hz), 104.1 (t, \(J = 28.1\) Hz), 55.7, 52.2, 37.3; \(^{19}\)F NMR (564 MHz, CDCl\(_3\)) δ -103.02 (t, \(J = 11.8\) Hz), -104.28 (dd, \(J = 20.3\), 9.3 Hz); FT-IR (thin film, KBr): ν (cm\(^{-1}\)) 2917, 1745, 1150, 754, 700; HRMS (CI) calcd C\(_{23}\)H\(_{18}\)F\(_2\)NO\(_2\) [M + H]: 378.1306, found: 378.1302.

**Methyl 2-(7,9-dimethylphenanthridin-6-yl)-3-phenylpropanoate (3i):** Yellow solid; m.p. 123-125 °C; 64% (36 mg); \(^1\)H NMR (400 MHz, CDCl\(_3\)) δ 8.52 (d, \(J = 8.1\) Hz, 1H), 8.35 (s, 1H), 8.14 (d, \(J = 7.9\) Hz, 1H), 7.70 (t, \(J = 7.3\) Hz, 1H), 7.61 (t, \(J = 7.4\) Hz, 1H), 7.28 (s, 1H), 7.24 - 7.07 (m, 5H), 5.40 (t, \(J = 6.8\) Hz, 1H), 3.74 - 3.60 (m, 4H), 3.51 (dd, \(J = 14.1\), 6.3 Hz, 1H), 2.94 (s, 3H), 2.55 (s, 3H); \(^{13}\)C NMR (150 MHz, CDCl\(_3\)) δ 173.1, 158.3, 143.0, 139.9, 135.2, 135.1, 134.0, 129.9, 129.1, 128.4, 128.3, 126.7, 126.2, 123.7, 123.6, 122.3, 121.0, 53.9, 52.2, 38.5, 26.1, 21.9; FT-IR (thin film, KBr): ν (cm\(^{-1}\)) 1738, 1715, 1171, 757, 703; HRMS (CI) calcd C\(_{23}\)H\(_{24}\)NO\(_2\) [M + H]: 370.1807, found: 370.1793.
Methyl 2-(benzo[c][2,7]naphthyridin-5-yl)-3-phenylpropanoate (3j): Yellowish solid; m.p. 160-161 °C; 91% (47 mg); ¹H NMR (600 MHz, CDCl₃) δ 9.59 (s, 1H), 8.88 (d, J = 5.6 Hz, 1H), 8.49 (d, J = 8.1 Hz, 1H), 8.34 (d, J = 5.6 Hz, 1H), 8.25 (d, J = 8.2 Hz, 1H), 7.84 (t, J = 7.6 Hz, 1H), 7.70 (t, J = 7.6 Hz, 1H), 7.25 (d, J = 7.5 Hz, 2H), 7.19 (t, J = 7.4 Hz, 2H), 7.12 (t, J = 7.3 Hz, 1H), 5.07 (t, J = 7.3 Hz, 1H), 3.76 (dd, J = 13.9, 7.4 Hz, 1H), 3.68 – 3.61 (m, 4H); ¹³C NMR (150 MHz, CDCl₃) δ 172.1, 157.2, 149.6, 148.4, 144.9, 139.2, 138.1, 131.0, 130.6, 129.2, 128.5, 127.7, 126.6, 122.5, 121.7, 120.3, 115.7, 52.5, 51.6, 37.1; FT-IR (thin film, KBr): ν (cm⁻¹) 2920, 1730, 1144, 769, 700; HRMS (CI) calcd C₂₂H₂₀N₂O₂ [M + H]⁺: 343.1447, found: 343.1444.

Methyl 2-(9-methylphenanthridin-6-yl)-3-phenylpropanoate (3k): Yellowish solid; m.p. 88-90 °C; 44% (19 mg); ¹H NMR (600 MHz, CDCl₃) δ 8.54 (d, J = 8.1 Hz, 1H), 8.43 (s, 1H), 8.20 (d, J = 8.1 Hz, 1H), 8.14 (d, J = 8.4 Hz, 1H), 7.72 (t, J = 7.5 Hz, 1H), 7.64 (t, J = 7.6 Hz, 1H), 7.49 (d, J = 8.4 Hz, 1H), 7.29 (d, J = 10.8 Hz, 2H), 7.22 (t, J = 13.9, 6.4 Hz, 2H), 7.15 (t, J = 7.3 Hz, 1H), 4.96 (t, J = 7.2 Hz, 1H), 3.75 (dd, J = 14.0, 8.1 Hz, 1H), 3.64 (s, 3H), 3.58 (dd, J = 14.0, 6.4 Hz, 1H), 2.64 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ 172.8, 157.3, 140.9, 139.9, 133.5, 130.5, 129.3, 129.2, 129.1, 128.6, 128.5, 126.9, 126.4, 125.5, 123.7, 123.4, 122.4, 122.0, 52.5, 52.4, 37.2, 22.3; FT-IR (thin film, KBr): ν (cm⁻¹) 2923, 1742, 1191, 1159, 697; HRMS (CI) calcd C₂₄H₂₂NO₂ [M + H]⁺: 356.1651, found: 356.1649.

Methyl 2-(7-methylphenanthridin-6-yl)-3-phenylpropanoate (3k’): Yellow oil; 40% (17 mg); ¹H NMR (600 MHz, CDCl₃) δ 8.56 (d, J = 8.3 Hz, 1H), 8.53 (d, J = 8.3 Hz, 1H), 8.17 (d, J = 8.1 Hz, 1H), 7.72 (t, J = 7.5 Hz, 1H), 7.67 – 7.60 (m, 2H), 7.44 (d, J = 8.7 Hz, 1H), 7.21 – 7.13 (m, 4H), 7.11 (t, J = 6.8 Hz, 1H), 5.43 (t, J = 7.1 Hz, 1H), 3.69 (s, 3H), 3.68 – 3.64 (m, 1H), 3.51 (dd, J = 14.2, 6.6 Hz, 1H), 2.97 (s, 3H); ¹³C NMR (150 MHz, CDCl₃) δ 173.0, 158.4, 142.8, 139.7, 135.3, 135.0, 132.2, 129.9, 129.8, 129.1, 128.6, 128.3, 127.0, 126.2, 125.5, 123.9, 122.3, 121.3, 53.9, 52.3, 38.5, 26.2; FT-IR (thin film, KBr): ν (cm⁻¹) 2917, 1721, 1173, 748, 698; HRMS (CI) calcd C₂₄H₂₂NO₂ [M + H]⁺: 356.1651, found: 356.1646.
Methyl 2-(phenanthridin-6-yl)propanoate (4a): White solid; m.p. 85-86 °C; 83% (33 mg); \(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta\) 8.65 (d, \(J = 8.3\) Hz, 1H), 8.53 (d, \(J = 9.7\) Hz, 1H), 8.21 (d, \(J = 8.2\) Hz, 1H), 8.16 (d, \(J = 8.1\) Hz, 1H), 7.83 (t, \(J = 7.6\) Hz, 1H), 7.70 (dt, \(J = 14.9, 7.5\) Hz, 2H), 7.64 (t, \(J = 7.5\) Hz, 1H), 4.77 (q, \(J = 7.1\) Hz, 1H), 3.70 (s, 3H); 13C NMR (150 MHz, CDCl\(_3\)) \(\delta\) 174.3, 159.4, 143.6, 133.4, 130.5, 130.3, 128.7, 127.6, 127.0, 125.6, 124.8, 123.8, 122.8, 121.9, 52.3, 45.6, 16.6; FT-IR (thin film, KBr): \(\nu\) (cm\(^{-1}\)) 2950, 1724, 1221, 754, 727; HRMS (CI) calcd C\(_{17}\)H\(_{16}\)NO\(_2\) [M + H]\(^+\): 266.1181, found: 266.1182.

Methyl 3-methyl-2-(phenanthridin-6-yl)butanoate (4b): Yellowish solid; m.p. 86-88 °C; 68% (30 mg); \(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta\) 8.67 (d, \(J = 8.3\) Hz, 1H), 8.56 (d, \(J = 8.1\) Hz, 1H), 8.41 (d, \(J = 8.3\) Hz, 1H), 8.21 (d, \(J = 8.1\) Hz, 1H), 7.84 (t, \(J = 7.6\) Hz, 1H), 7.73 (d, \(J = 7.6\) Hz, 1H), 7.71 (d, \(J = 8.1\) Hz, 1H), 7.64 (t, \(J = 17.6, 9.8\) Hz, 1H), 4.39 (d, \(J = 10.0\) Hz, 1H), 3.63 (s, 3H), 3.16 – 3.06 (m, 1H), 1.23 (d, \(J = 6.6\) Hz, 3H), 0.83 (d, \(J = 6.7\) Hz, 3H); 13C NMR (150 MHz, CDCl\(_3\)) \(\delta\) 172.9, 157.4, 143.8, 133.3, 130.5, 128.7, 127.6, 127.0, 125.9, 123.7, 122.7, 122.0, 58.1, 52.1, 30.1, 21.6, 21.0; FT-IR (thin film, KBr): \(\nu\) (cm\(^{-1}\)) 2917, 1733, 1149, 757, 721; HRMS (CI) calcd C\(_{19}\)H\(_{20}\)NO\(_2\) [M + H]\(^+\): 294.1494, found: 294.1499.

Methyl 4-methyl-2-(phenanthridin-6-yl)pentanoate (4c): Yellow solid; m.p. 89-90 °C; 75% (35 mg); \(^1\)H NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.67 (d, \(J = 8.2\) Hz, 1H), 8.55 (d, \(J = 8.1\) Hz, 1H), 8.28 (d, \(J = 8.2\) Hz, 1H), 8.18 (d, \(J = 8.0\) Hz, 1H), 7.84 (t, \(J = 7.5\) Hz, 1H), 7.72 (t, \(J = 6.5\) Hz, 2H), 7.64 (t, \(J = 7.5\) Hz, 1H), 4.77 (t, \(J = 7.2\) Hz, 1H), 3.68 (s, 3H), 2.38 – 2.28 (m, 1H), 2.17 – 2.07 (m, 1H), 1.76 – 1.66 (m, 1H), 1.01 (d, \(J = 7.3\) Hz, 3H), 0.99 (d, \(J = 7.1\) Hz, 3H); 13C NMR (150 MHz, CDCl\(_3\)) \(\delta\) 173.6, 158.5, 143.7, 133.4, 130.5, 128.7, 127.6, 127.0, 125.1, 123.8, 122.8, 121.9, 52.3, 49.2, 40.2, 26.6, 22.8, 22.7; FT-IR (thin film, KBr): \(\nu\) (cm\(^{-1}\)) 2938, 1718, 1162, 751, 724; HRMS (CI) calcd C\(_{20}\)H\(_{22}\)NO\(_2\) [M + H]\(^+\): 308.1651, found: 308.1646.
Methyl 3-methyl-2-(phenanthridin-6-yl)pentanoate (4d): Yellowish oil; 74% (34 mg); Two isomers, d.r.: 1.3:1; \(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta\) 8.67 (d, \(J = 8.3\) Hz, 1H), 8.56 (d, \(J = 8.1\) Hz, 1H), 8.41 (t, \(J = 7.5\) Hz, 1H), 8.22 (d, \(J = 8.1\) Hz, 1H), 7.84 (t, \(J = 7.6\) Hz, 1H), 7.74 (d, \(J = 7.5\) Hz, 1H), 7.71 (d, \(J = 8.2\) Hz, 1H), 7.65 (t, \(J = 7.5\) Hz, 1H), 4.52 (d, \(J = 10.0\) Hz, 0.44H)/4.50 (d, \(J = 10.2\) Hz, 0.56H), 3.63 (s, 3H), 2.98 – 2.87 (m, 1H), 1.86 – 1.08 (m, 2H), 1.20 (d, \(J = 6.6\) Hz, 1.3H)/0.79 (d, \(J = 6.7\) Hz, 1.7H), 1.06 (t, \(J = 7.4\) Hz, 1.7H)/0.84 (t, \(J = 7.4\) Hz, 1.3H); \(^{13}\)C NMR (150 MHz, CDCl\(_3\)) \(\delta\) 172.9, 172.8, 157.4, 157.2, 143.8, 133.24, 133.22, 130.59, 130.53, 130.46, 128.7, 127.62, 127.59, 127.0, 125.99, 125.95, 125.8, 123.7, 122.7, 121.9, 56.8, 56.5, 52.12, 52.10, 36.1, 36.0, 28.1, 27.3, 17.4, 16.8, 11.5, 11.4; FT-IR (thin film, KBr): \(\nu\) (cm\(^{-1}\)) 2965, 1730, 1165, 754, 721; HRMS (CI) calcd C\(_{20}\)H\(_{22}\)NO\(_2\) [M + H]\(^+\): 308.1651, found: 308.1651.

Methyl 4-(methylthio)-2-(phenanthridin-6-yl)butanoate (4e): Yellow oil; 84% (42 mg); \(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta\) 8.68 (d, \(J = 8.3\) Hz, 1H), 8.56 (d, \(J = 8.1\) Hz, 1H), 8.34 (d, \(J = 8.3\) Hz, 1H), 8.15 (d, \(J = 8.1\) Hz, 1H), 7.86 (t, \(J = 7.6\) Hz, 1H), 7.76 – 7.69 (m, 2H), 7.66 (t, \(J = 7.5\) Hz, 1H), 4.96 (t, \(J = 6.5\) Hz, 1H), 3.68 (s, 3H), 2.69 – 2.64 (m, 2H), 2.64 – 2.54 (m, 2H), 2.13 (s, 3H); \(^{13}\)C NMR (150 MHz, CDCl\(_3\)) \(\delta\) 173.1, 157.8, 143.6, 133.4, 130.6, 130.4, 128.8, 127.8, 127.1, 125.7, 125.2, 123.9, 122.8, 122.0, 52.4, 48.8, 32.6, 30.4, 15.5; FT-IR (thin film, KBr): \(\nu\) (cm\(^{-1}\)) 2923, 1748, 1724, 1127, 760; HRMS (CI) calcd C\(_{19}\)H\(_{20}\)NO\(_2\)S [M + H]\(^+\): 326.1215, found: 326.1211.

Methyl 3-(4-methoxyphenyl)-2-(phenanthridin-6-yl)propanoate (4f): Yellowish oil; 97% (60 mg); \(^1\)H NMR (600 MHz, CDCl\(_3\)) \(\delta\) 8.65 (d, \(J = 6.7\) Hz, 1H), 8.55 (d, \(J = 8.1\) Hz, 1H), 8.25 (d, \(J = 8.2\) Hz, 1H), 8.21 (d, \(J = 6.7\) Hz, 1H), 7.83 (t, \(J = 7.6\) Hz, 1H), 7.75 (t, 1H), 7.70 – 7.64 (m, 2H), 7.22 (d, \(J = 8.5\) Hz, 2H), 6.76 (d, \(J = 8.5\) Hz, 2H), 4.94 (t, \(J = 7.7, 6.8\) Hz, 1H), 3.74 (s, 3H), 3.69 (dd, \(J = 14.1, 8.1\) Hz, 1H), 3.64 (s,
3H), 3.53 (dd, $J = 14.1, 6.4$ Hz, 1H); $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 172.8, 158.2, 157.5, 143.6, 133.3, 131.9, 130.5, 130.2, 128.8, 127.6, 127.1, 125.7, 125.3, 123.9, 122.7, 122.0, 113.9, 55.3, 52.7, 52.4, 36.3; FT-IR (thin film, KBr): $\nu$ (cm$^{-1}$) 1730, 1510, 1245, 754, 721; HRMS (CI) calcd C$_{24}$H$_{22}$NO$_3$ [M + H]$^+$: 372.1600, found: 372.1593.

Methyl 2-(phenanthridin-6-yl)-4-phenylbutanoate (4g): Yellow solid; m.p. 90-91 °C; 92% (48 mg); $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 8.66 (d, $J = 8.2$ Hz, 1H), 8.56 (d, $J = 8.0$ Hz, 1H), 8.18 (d, 1H), 8.04 (d, $J = 8.2$ Hz, 1H), 7.83 (t, 1H), 7.73 (t, 1H), 7.68 – 7.62 (m, 2H), 7.32 – 7.26 (m, 2H), 7.23 – 7.18 (m, 3H), 4.66 (t, $J = 6.7$ Hz, 1H), 3.69 (s, 3H), 2.84 – 2.70 (m, 3H), 2.63 – 2.55 (m, 1H); $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 173.3, 158.0, 143.7, 141.6, 133.4, 130.5, 130.4, 128.8, 128.7, 128.5, 127.6, 127.1, 126.1, 125.7, 125.2, 123.8, 122.8, 122.0, 52.4, 49.9, 34.1, 32.9; FT-IR (thin film, KBr): $\nu$ (cm$^{-1}$) 1730, 1435, 1153, 760, 727; HRMS (CI) calcd C$_{24}$H$_{22}$NO$_2$ [M + H]$^+$: 356.1651, found: 356.1649.

Dimethyl 2-(phenanthridin-6-yl)succinate (4h): Yellowish oil; 98% (48 mg); $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.66 (d, $J = 8.2$ Hz, 1H), 8.55 (d, $J = 8.0$ Hz, 1H), 8.35 (d, $J = 8.2$ Hz, 1H), 8.13 (d, $J = 8.0$ Hz, 1H), 7.86 (t, $J = 7.5$ Hz, 1H), 7.74 (d, $J = 8.2$ Hz, 1H), 7.70 (d, $J = 8.2$ Hz, 1H), 7.65 (t, $J = 7.4$ Hz, 1H), 5.27 (t, 1H), 3.70 (s, 3H), 3.68 (s, 3H), 3.49 (dd, $J = 17.1, 8.1$ Hz, 1H), 3.28 (dd, $J = 17.1, 6.2$ Hz, 1H); $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 172.7, 172.4, 156.7, 143.4, 133.4, 130.7, 130.4, 128.7, 127.7, 127.2, 125.8, 125.0, 123.9, 122.7, 122.0, 52.7, 52.1, 45.9, 35.5; FT-IR (thin film, KBr): $\nu$ (cm$^{-1}$) 2953, 1727, 1153, 757, 727; HRMS (CI) calcd C$_{19}$H$_{18}$NO$_4$ [M + H]$^+$: 324.1236, found: 324.1226.
Dimethyl 2-(phenanthridin-6-yl)pentanedioate (4i): Yellow oil; 88% (44 mg); \(^1H\) NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.66 (d, \(J = 8.2\) Hz, 1H), 8.55 (d, \(J = 8.0\) Hz, 1H), 8.35 (d, \(J = 8.2\) Hz, 1H), 8.15 (d, \(J = 8.0\) Hz, 1H), 7.86 (t, \(J = 19.9, 12.5\) Hz, 1H), 7.77 – 7.69 (m, 2H), 7.65 (t, \(J = 7.4\) Hz, 1H), 4.83 (t, \(J = 6.8\) Hz, 1H), 3.69 (s, 3H), 3.66 (s, 3H), 2.71 – 2.58 (m, 2H), 2.57 – 2.45 (m, 2H); \(^13C\) NMR (150 MHz, CDCl\(_3\)) \(\delta\) 173.8, 173.0, 157.8, 143.5, 133.3, 130.6, 130.4, 128.7, 127.7, 127.1, 125.7, 125.1, 123.8, 122.7, 122.0, 52.4, 51.7, 49.4, 31.8, 26.3; FT-IR (thin film, KBr): \(\nu\) (cm\(^{-1}\)) 2923, 2848, 1724, 1156, 697; HRMS (CI) calcd C\(_{20}\)H\(_{20}\)NO\(_4\) [M+H]\(^+\): 338.1392, found: 338.1388.

Methyl 3-(4-hydroxyphenyl)-2-(phenanthridin-6-yl)propanoate (4j): Yellow oil; 68% (36.1 mg); \(^1H\) NMR (400 MHz, CDCl\(_3\)) \(\delta\) 8.66 (d, \(J = 8.3\) Hz, 1H), 8.58 – 8.54 (m, 1H), 8.25 (d, \(J = 8.2\) Hz, 1H), 8.19 (d, \(J = 8.1, 1.0\) Hz, 1H), 7.87 – 7.81 (m, 1H), 7.75 – 7.63 (m, 3H), 7.05 (d, \(J = 8.4\) Hz, 2H), 6.60 (d, \(J = 8.5\) Hz, 2H), 4.92 (t, \(J = 7.3\) Hz, 1H), 3.69 (dd, \(J = 14.1, 7.7\) Hz, 1H), 3.62 (s, 3H), 3.49 (dd, \(J = 14.1, 6.9\) Hz, 1H); \(^13C\) NMR (101 MHz, CDCl\(_3\)) \(\delta\) 173.1, 157.7, 154.5, 143.3, 133.5, 131.2, 130.7, 130.3, 130.2, 128.9, 127.7, 127.2, 125.8, 125.2, 123.9, 122.8, 122.1, 115.5, 53.3, 52.5, 36.4; HRMS (CI) calcd C\(_{23}\)H\(_{20}\)NO\(_3\) [M+H]\(^+\): 358.1443, found: 358.1435.

tert-Butyl 2-(phenanthridin-6-yl)-3-phenylpropanoate (4k): White solid; m.p. 147-148 °C; 94% (54 mg); \(^1H\) NMR (600 MHz, CDCl\(_3\)) \(\delta\) 8.62 (d, \(J = 8.3\) Hz, 1H), 8.53 (d, \(J = 8.1\) Hz, 1H), 8.25 (d, \(J = 8.3\) Hz, 1H), 8.20 (d, \(J = 8.1\) Hz, 1H), 7.79 (t, \(J = 13.6, 6.2\) Hz, 1H), 7.71 (t, \(J = 7.5\) Hz, 1H), 7.66 – 7.61 (m, 2H), 7.31 (d, \(J = 7.6\) Hz, 2H), 7.22 (t, \(J = 7.5\) Hz, 2H), 7.14 (t, \(J = 7.3\) Hz, 1H), 4.89 – 4.84 (m, 1H), 3.71 (dd, \(J = 14.1, 8.3\) Hz, 1H), 3.54 (dd, \(J = 14.1, 6.2\) Hz, 1H), 1.31 (s, 9H); \(^13C\) NMR (150 MHz, CDCl\(_3\)) \(\delta\) 171.5, 158.1, 143.7, 140.2, 133.3, 130.5, 130.3, 129.3, 128.6, 128.4, 127.3, 126.9, 126.3, 125.8, 125.3, 123.8, 122.6, 122.0, 81.3, 53.2, 37.1, 28.1; FT-IR (thin film, KBr): \(\nu\) (cm\(^{-1}\)) 2971, 1635, 757, 724, 695; HRMS (CI) calcd C\(_{26}\)H\(_{26}\)NO\(_2\) [M+H]\(^+\): 384.1964, found: 384.1970.
**N,N-Diethyl-2-(phenanthridin-6-yl)-3-phenylpropanamide (4l):** Yellow oil; 95% (57 mg); $^1$H NMR (600 MHz, CDCl$_3$) $\delta$ 8.66 (d, $J = 8.3$ Hz, 1H), 8.55 (d, $J = 8.1$ Hz, 1H), 8.40 (d, $J = 8.2$ Hz, 1H), 8.17 (d, $J = 8.0$ Hz, 1H), 7.82 (t, $J = 7.6$ Hz, 1H), 7.71 (t, $J = 7.5$ Hz, 1H), 7.69 – 7.62 (m, 2H), 7.29 (d, $J = 7.5$ Hz, 2H), 7.21 (t, $J = 7.4$ Hz, 2H), 7.14 (t, $J = 7.3$ Hz, 1H), 4.97 – 4.93 (m, 1H), 3.89 (dd, $J = 13.9$, 8.2 Hz, 1H), 3.62 (dq, $J = 13.8$, 6.9 Hz, 1H), 3.35 (dd, $J = 14.0$, 5.4 Hz, 1H), 3.25 – 3.13 (m, 2H), 3.07 (dq, $J = 14.2$, 6.9 Hz, 1H), 1.11 (t, $J = 7.0$ Hz, 3H), 0.69 (t, $J = 7.0$ Hz, 1H); $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 170.4, 159.1, 143.6, 140.7, 133.4, 130.5, 130.4, 129.2, 128.7, 128.3, 127.5, 127.0, 126.2, 125.7, 124.8, 123.6, 122.8, 121.9, 52.8, 41.6, 40.5, 38.2, 13.9, 12.7; FT-IR (thin film, KBr): $\nu$ (cm$^{-1}$) 2938, 1629, 1218, 760, 695; HRMS (CI) calcd C$_{26}$H$_{27}$N$_2$O [M + H]$^+$: 383.2123, found: 383.2140.

**2-(Phenanthridin-6-yl)-3-phenyl-1-(piperidin-1-yl)propan-1-one (4m):** Light yellow solid; m.p. 116-117 °C; 98% (58 mg); $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.53 (d, $J = 8.2$ Hz, 1H), 8.41 (d, $J = 8.0$ Hz, 1H), 8.19 (d, $J = 8.2$ Hz, 1H), 8.04 (d, $J = 7.9$ Hz, 1H), 7.69 (t, $J = 7.5$ Hz, 1H), 7.59 (t, $J = 7.4$ Hz, 1H), 7.54 – 7.46 (m, 2H), 7.17 – 7.11 (m, 2H), 7.08 (t, $J = 7.2$ Hz, 2H), 7.04 – 6.98 (m, 1H), 4.86 (t, $J = 6.6$ Hz, 1H), 3.74 (dd, $J = 13.8$, 8.0 Hz, 1H), 3.69 – 3.59 (m, 1H), 3.34 – 3.20 (m, 2H), 3.18 – 2.98 (m, 2H), 1.40 – 1.20 (m, 4H), 0.96 – 0.81 (m, 1H), 0.70 – 0.57 (m, 1H); $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 169.8, 159.0, 143.6, 140.7, 133.4, 130.5, 130.4, 129.4, 128.7, 128.4, 127.6, 127.0, 126.2, 125.6, 124.8, 123.6, 122.8, 121.9, 52.4, 46.7, 43.6, 38.1, 25.8, 25.6, 24.5; FT-IR (thin film, KBr): $\nu$ (cm$^{-1}$) 2935, 1632, 1221, 763, 698; HRMS (CI) calcd C$_{27}$H$_{27}$N$_2$O [M + H]$^+$: 395.2123, found: 395.2140.

**Methyl (2-(phenanthridin-6-yl)-3-phenylpropanoyl)glycinate (4n):** Yellow oil; 72% (39 mg); $^1$H NMR (400 MHz, CDCl$_3$) $\delta$ 8.61 (d, $J = 8.3$ Hz, 1H), 8.56 (d, $J = 8.1$ Hz, 1H), 8.48 (br, 1H), 8.27 (d, $J = 8.0$ Hz, 1H), 8.16 (d, $J = 8.2$ Hz, 1H), 7.79 (t, $J = 7.5$ Hz, 2H), 7.69 (t, $J = 7.5$ Hz, 1H), 7.58 (t, $J = 7.6$ Hz, 1H), 7.16 – 6.98 (m, 5H), 4.98 (t, $J = 8.3$, 6.1 Hz, 1H), 4.13 (dd, $J = 18.5$, 5.2 Hz, 1H), 3.96 (dd, $J = 18.5$, 4.7 Hz, 1H), 3.89 (dd, $J = 14.0$, 8.2 Hz, 1H), 3.62 (dq, $J = 13.8$, 6.9 Hz, 1H), 3.35 (dd, $J = 14.0$, 5.4 Hz, 1H), 3.25 – 3.13 (m, 2H), 3.07 (dq, $J = 14.2$, 6.9 Hz, 1H), 1.11 (t, $J = 7.0$ Hz, 3H), 0.69 (t, $J = 7.0$ Hz, 1H); $^{13}$C NMR (150 MHz, CDCl$_3$) $\delta$ 170.4, 159.1, 143.6, 140.7, 133.4, 130.5, 130.4, 129.2, 128.7, 128.3, 127.5, 127.0, 126.2, 125.7, 124.8, 123.6, 122.8, 121.9, 52.8, 41.6, 40.5, 38.2, 13.9, 12.7; FT-IR (thin film, KBr): $\nu$ (cm$^{-1}$) 2938, 1629, 1218, 760, 695; HRMS (CI) calcd C$_{26}$H$_{27}$N$_2$O [M + H]$^+$: 383.2123, found: 383.2140.
Hz, 1H), 3.76 – 3.67 (m, 4H), 3.63 (dd, J = 13.3, 5.7 Hz, 1H); $^{13}$C NMR (150 MHz, CDCl$_3$) δ 172.1, 170.4, 158.7, 142.9, 139.0, 133.1, 130.9, 129.9, 129.1, 129.0, 128.3, 127.7, 127.3, 126.4, 125.8, 125.6, 123.8, 122.5, 122.2, 52.5, 52.4, 41.7, 41.3; FT-IR (thin film, KBr): ν (cm$^{-1}$) 2923, 1754, 1206, 751, 692; HRMS (ESI) calcd C$_{25}$H$_{23}$N$_2$O$_3$ [M + H]$^+$: 399.1709, found: 399.1703.

Methyl (2-(phenanthridin-6-yl)-3-phenylpropanoyl)alaninate (4o): Yellow solid; m.p. 163-165 ℃; 80% (49 mg); Two isomers, d.r.: 1.2:1; $^1$H NMR (600 MHz, CDCl$_3$) δ 8.75 (d, J = 5.9 Hz, 0.55H)/8.47 (d, J = 6.6 Hz, 0.45H), 8.64 – 8.58 (m, 1H), 8.56 (d, J = 8.1 Hz, 1H), 8.26 (t, J = 8.5 Hz, 1H), 8.22 – 8.12 (m, 1H), 7.79 (t, J = 7.0 Hz, 2H), 7.69 (t, J = 7.5 Hz, 1H), 7.58 (dd, J = 14.4, 7.1 Hz, 1H), 7.15 – 6.96 (m, 5H), 4.93 (dd, J = 14.7, 7.9 Hz, 1H), 4.62 – 4.52 (m, 1H), 3.78 (s, 1.4H)/ 3.57 (s, 1.7H), 3.74 – 3.59 (m, 2H), 1.46 (d, J = 7.1 Hz, 1.7H)/ 1.28 (d, J = 7.3 Hz, 1.3H); $^{13}$C NMR (150 MHz, CDCl$_3$) δ 173.6, 173.3, 171.39, 171.37, 158.8, 158.7, 142.9, 139.1, 138.8, 133.1, 133.0, 130.8, 129.8, 129.21, 129.17, 129.0, 128.3, 127.7, 127.3, 127.2, 126.5, 126.4, 125.7, 125.6, 125.5, 123.7, 122.53, 122.48, 122.2, 122.1, 52.6, 52.5, 52.3, 48.44, 48.38, 41.5, 41.3, 18.7, 18.4; FT-IR (thin film, KBr): ν (cm$^{-1}$) 1715, 1364, 1141, 754, 698; HRMS (Cl) calcd C$_{26}$H$_{25}$N$_2$O$_3$ [M + H]$^+$: 413.1865, found: 413.1872.

8 NMR Spectra for the substrates and products
$^1$H NMR of 2a

$^{13}$C NMR of 2a

$^{19}$F NMR of 2a

S23
$^1$H NMR of 2b

$^{13}$C NMR of 2b
$^{19}$F NMR of 2b

$^1$H NMR of 2c
$^{13}$C NMR of 2c

$^{19}$F NMR of 2c
$^1$H NMR of 2d

$^{13}$C NMR of 2d
$^{19}$F NMR of 2d

$^1$H NMR of 2e
$^{13}$C NMR of 2e

$^{19}$F NMR of 2e
$^1$H NMR of $2f$

$^{13}$C NMR of $2f$
$^{19}$F NMR of 2f

$^1$H NMR of 2g
$^{13}$C NMR of $2g$

$^{19}$F NMR of $2g$
$^1$H NMR of 2h

$^{13}$C NMR of 2h
$^{19}$F NMR of 2h

$^{1}$H NMR of 2i
$^{13}$C NMR of 2i

$^{19}$F NMR of 2i
$^1$H NMR of 2j

$^{13}$C NMR of 2j
$^{19}$F NMR of 2j

$^1$H NMR of 2k
$^{13}$C NMR of 2k

$^{19}$F NMR of 2k
1H NMR of 21

12C NMR of 21
$^{19}$F NMR of 2l

$^1$H NMR of 2m
$^{13}$C NMR of 2m

$^{19}$F NMR of 2m
$^{1}H$ NMR of 2n

$^{13}C$ NMR of 2n
\[1^9\text{F NMR of 2n}\]

\[1^1\text{H NMR of 2o}\]
$^{13}$C NMR of 2o

$^{19}$F NMR of 2o
$^1$H NMR of 2p

$^{13}$C NMR of 2p
$^{19}$F NMR of 2p

$^{1}$H NMR of 3a
$^{13}$C NMR of 3a

$^1$H NMR of 3b


13C NMR of 3b

1H NMR of 3c
$^{13}$C NMR of 3e

$^1$H NMR of 3d
$^{13}$C NMR of 3d

$^{19}$F NMR of 3d
$^1$H NMR of 3e

$^{13}$C NMR of 3e
$^1$H NMR of 3f

$^{13}$C NMR of 3f
$^1$H NMR of 3g

$^{19}$F NMR of 3f
$^{13}$C NMR of $3g$

$^1$H NMR of $3h$
$^{13}$C NMR of 3h

$^{19}$F NMR of 3h
$^1$H NMR of 3i

$^{13}$C NMR of 3i
$^{1}H$ NMR of $3j$

$^{13}C$ NMR of $3j$
$^1$H NMR of 3k

$^{13}$C NMR of 3k
$^1$H NMR of $3k'$

$^{13}$C NMR of $3k'$
$^1$H NMR of 4a

$^{13}$C NMR of 4a
\( ^{1}H \) NMR of 4b

\[ \text{Structural diagram} \]

\( ^{13}C \) NMR of 4b

\[ \text{Structural diagram} \]
$^{1}H$ NMR of 4c

$^{13}C$ NMR of 4c
$^1$H NMR of 4e

$^{13}$C NMR of 4e
$^{1}$H NMR of 4f

$^{13}$C NMR of 4f
$^1$H NMR of 4g

$^{13}$C NMR of 4g
$^1$H NMR of 4h

$^{13}$C NMR of 4h
$^{1}H$ NMR of 4i

$^{13}C$ NMR of 4i
$^1$H NMR of 4j

$^{13}$C NMR of 4j
$^1$H NMR of 4k

$^{13}$C NMR of 4k
$^1$H NMR of 4l

$^{13}$C NMR of 4l
**1H NMR of 4m**

**13C NMR of 4m**
$^{1}H$ NMR of 4n
$^{13}$C NMR of 4n

$^1$H NMR of 4o
$^{13}$C NMR of $4o$

$^{1}$H NMR of TEMPO-trapped product

$^{13}$C NMR of TEMPO-trapped product