Visible Light-Induced Deaminative Thioesterification of Amino Acid-Derived Katritzky Salts via Electron-Donor Acceptor Complex Formation

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1. General Methods

Unless stated otherwise, all reactions were carried out under an atmosphere of argon using standard Schlenk techniques. All solvents and reagents were obtained from commercial sources and were purified according to standard procedures before use. Organic solutions were concentrated under reduced pressure on a Büchi rotary evaporator. Chromatographic purification of products was accomplished by flash chromatography on silica gel. The product spots on the thin layer chromatography (TLC) was identified/visualized by fluorescence quenching or potassium permanganate stains. $^1$H and $^{13}$C NMR spectra were recorded on a Bruker Avance 400 (400 MHz and 100 MHz) or a Bruker Avance 500 (500 MHz and 126 MHz) instrument, and are internally referenced to solvent residual signals (note: CDCl$_3$ referenced at 7.26 and 77.00 ppm respectively). Data for $^1$H NMR were reported as follows: chemical shift ($\delta$, ppm), multiplicity (s = singlet, d = doublet, t = triplet, m = multiplet or unresolved, coupling constant(s) in Hz, integration). Data for $^{13}$C NMR are reported in ppm with the internal chloroform signal at 77.00 ppm as a standard. High resolution mass spectra were recorded on Waters Micromass GCT Premier (EI) and Exactive Plus LC-MS (ESI) mass spectrometers. GC measurements were performed on a GCMS-QP2010SE from SHIMADZU.
2. Synthesis of Katritzky Salts

Katritzky salts \(1a, 1b, 1d, 1h, 1j, 1o, 1p, 1q, 1r\) were prepared according to previous reported procedures.\(^1\) Unknown Katritzky salts \(1c, 1e, 1f, 1g, 1i, 1k, 1l, 1m, 1n\) were prepared according to literature procedures\(^1\) as described as follows:

The corresponding amino acid ester hydrochloride (1.2 equiv) was weighed into a reaction tube then EtOH (1.0 M) and \(\text{Et}_3\text{N}\) (1.2 equiv) were added successively. The reaction mixture was stirred at room temperature for 30 min and then triphenylpyrylium tetrafluoroborate (1 g, 1.0 equiv) was added and heated to 90 °C. After 4 h cooled to room temperature, diethyl ether was added to the reaction mixture and the resulting suspension was stirred at rt for at least 1 h to complete the precipitation process. The solid was collected by filtration and washed with \(\text{H}_2\text{O}\) (to remove \(\text{Et}_3\text{N}\cdot\text{HX}\) salts) and \(\text{Et}_2\text{O}\). The solid was then dried under reduced pressure to give the pyridinium salts without further purification. If precipitation did not take place, the solvent was removed under reduced pressure. The crude product was then dissolved in \(\text{CH}_2\text{Cl}_2\) (1 M), washed with isopyknic \(\text{H}_2\text{O}\) (3 times) and concentrated under reduced pressure. The crude product was then purified by column chromatography with a gradient of acetone in \(\text{CH}_2\text{Cl}_2\) eluting to afford the pyridinium salt product.

\[\text{1c} \quad 1-\text{(1-(benzyloxy)-1-oxo-3-phenylpropan-2-yl)}-2,4,6\text{-triphenylpyridin-1-iunum tetrafluoroborate (1c), recrystallized from hot EtOH/Et}_2\text{O (v/v 1:4), white solid, 81% yield.}\]

\(^1\text{H NMR (400 MHz, CDCl3):} \ \delta (\text{ppm}): 7.95 \text{ (s, 2H), 7.85} – 7.66 \text{ (m, 4H), 7.58} – 7.37 \text{ (m, 14H), 7.18 (d, } J = 6.8 \text{ Hz, 2H), 7.10} – 7.00 \text{ (m, 3H), 6.72 (d, } J = 7.2 \text{ Hz, 2H), 5.68}\]
- 5.67 (m, 1H), 5.12 (d, \( J = 11.6 \) Hz, 1H), 5.02 (d, \( J = 11.6 \) Hz, 1H), 3.51 – 3.47 (m, 1H), 2.75 (dd, \( J = 14.4, 8.4 \) Hz, 1H).

\[^{13}\text{C NMR (100 MHz, CDCl}_3\): \delta (ppm): 167.4, 156.9, 156.8, 136.0, 133.5, 133.5, 132.4, 132.2, 131.4, 129.6, 129.3, 129.1, 128.9, 128.7, 128.5, 128.4, 127.7, 127.1, 70.4, 69.0, 37.3.\]

\[^{19}\text{F NMR (376 MHz, CDCl}_3\): \delta (ppm): -152.68, -152.73.\]

\[^{1}\text{H NMR (400 MHz, CDCl}_3\): \delta (ppm): 7.94 (s, 2H), 7.84 – 7.78 (m, 4H), 7.59 – 7.48 (m, 11H), 6.57 (d, \( J = 8.0 \) Hz, 1H), 6.41 (s, 1H), 6.34 (d, \( J = 8.0 \) Hz, 1H), 5.64 (dd, \( J = 6.8, 4.0 \) Hz, 1H), 3.76 (s, 3H), 3.72 (s, 3H), 3.67 (s, 3H), 3.35 (dd, \( J = 14.4, 4.0 \) Hz, 1H), 2.86 (dd, \( J = 14.4, 7.2 \) Hz, 1H).\]

\[^{13}\text{C NMR (100 MHz, CDCl}_3\): \delta (ppm): 168.0, 156.8, 148.8, 148.0, 133.6, 132.4, 132.3, 131.5, 129.7, 129.4, 129.1, 128.6, 128.4, 127.8, 121.0, 112.1, 110.9, 70.5, 55.84, 55.76, 53.7, 37.4.\]

\[^{19}\text{F NMR (376 MHz, CDCl}_3\): \delta (ppm): -152.63, -152.68.\]

\[^{1}\text{H NMR (400 MHz, CDCl}_3\): \delta (ppm): 7.94 (s, 2H), 7.84 – 7.78 (m, 4H), 7.59 – 7.48 (m, 11H), 6.57 (d, \( J = 8.0 \) Hz, 1H), 6.41 (s, 1H), 6.34 (d, \( J = 8.0 \) Hz, 1H), 5.64 (dd, \( J = 6.8, 4.0 \) Hz, 1H), 3.76 (s, 3H), 3.72 (s, 3H), 3.67 (s, 3H), 3.35 (dd, \( J = 14.4, 4.0 \) Hz, 1H), 2.86 (dd, \( J = 14.4, 7.2 \) Hz, 1H).\]

\[^{13}\text{C NMR (100 MHz, CDCl}_3\): \delta (ppm): 168.0, 156.8, 148.8, 148.0, 133.6, 132.4, 132.3, 131.5, 129.7, 129.4, 129.1, 128.6, 128.4, 127.8, 121.0, 112.1, 110.9, 70.5, 55.84, 55.76, 53.7, 37.4.\]

\[^{19}\text{F NMR (376 MHz, CDCl}_3\): \delta (ppm): -152.63, -152.68.\]

HRMS (EI): \( m/z \) calculated for \( \text{C}_{39}\text{H}_{32}\text{NO}_2\) (M-BF\(_4\))^+: 546.2428, found 546.2430.

1-(3-(3,4-dimethoxyphenyl)-1-methoxy-1-oxopropan-2-yl)-2,4,6-triphenylpyridin-1-i um tetrafluoroborate (1e), recrystallized from EtOH/Et\(_2\)O (v/v 1:4), white solid, 86% yield.
1-(3-(4-fluorophenyl)-1-methoxy-1-oxopropan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (1f), recrystallized from EtOH/Et₂O (v/v 1:4), white solid, 82% yield.

**¹H NMR (400 MHz, CDCl₃):** δ (ppm): 7.92 (s, 2H), 7.82 – 7.80 (m, 4H), 7.59 – 7.47 (m, 11H), 6.81 – 6.72 (m, 4H), 5.56 (d, J = 7.6 Hz, 1H), 3.69 (s, 3H), 3.47 (d, J = 13.6 Hz, 1H), 2.87 (dd, J = 14.0, 8.4 Hz, 1H).

**¹³C NMR (100 MHz, CDCl₃):** δ (ppm): 167.8, 161.8 (d, J =245.0 Hz, 1C), 157.0, 156.7, 133.7, 132.4, 132.3 (d, J = 3.0 Hz, 1C), 132.2, 131.5, 130.6 (d, J = 8.0 Hz, 1C), 129.6, 129.3, 129.1, 128.5, 127.8, 115.3 (d, J = 21.0 Hz, 1C), 70.0, 53.7, 36.9.

**¹⁹F NMR (376 MHz, CDCl₃):** δ (ppm): -115.18, -152.51, -152.56.

**HRMS (EI):** m/z calculated for C₃₃H₂₇FNO₂ (M-BF₄)⁺: 488.2020, found 488.2021.

1-(1-methoxy-3-(4-nitrophenyl)-1-oxopropan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (1g), recrystallized from EtOH/Et₂O (v/v 1:4), brown solid, 74% yield.
$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm): 7.95 (s, 2H), 7.88 – 7.59 (m, 10H), 7.57 – 7.47 (m, 7H), 7.02 (d, $J = 8.4$ Hz, 2H), 5.59 (d, $J = 8.8$ Hz, 1H), 3.73 – 3.69 (m, 1H), 3.69 (s, 3H), 2.92 (dd, $J = 14.4$, 9.2 Hz, 1H).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm): 167.5, 157.2, 156.5, 146.8, 144.6, 133.7, 132.4, 132.1, 131.7, 130.1, 129.6, 129.2, 128.4, 127.8, 123.5, 69.1, 53.9, 37.4.

$^{19}$F NMR (376 MHz, CDCl$_3$): $\delta$ (ppm): -152.23, -152.28.

HRMS (EI): $m/z$ calculated for C$_{33}$H$_{27}$N$_2$O$_4$ (M-BF$_4$)$^+$: 515.1965, found 515.1969.

![Structure of 1](image)

1-(1,5-diethoxy-1,5-dioxopentan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (II), recrystallized from EtOH/Et$_2$O (v/v 1:4), white solid, 82% yield.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm): 7.95 (s, 2H), 7.85 – 7.74 (m, 4H), 7.67 – 7.49 (m, 11H), 5.61 – 5.60 (m, 1H), 4.22 – 4.10 (m, 2H), 3.91 (q, $J = 7.2$ Hz, 2H), 2.26 – 2.19 (m, 2H), 2.04 – 1.96 (m, 2H), 1.20 (t, $J = 7.2$ Hz, 3H), 1.13 (t, $J = 7.2$ Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm): 171.6, 168.0, 157.0, 156.9, 133.7, 132.4, 131.4, 129.6, 129.2, 128.5, 127.9, 67.8, 63.5, 60.6, 31.0, 26.8, 13.9, 13.7.

$^{19}$F NMR (376 MHz, CDCl$_3$): $\delta$ (ppm): -153.04, -153.09.

HRMS (EI): $m/z$ calculated for C$_{32}$H$_{32}$NO$_4$ (M-BF$_4$)$^+$: 494.2326, found 494.2328.
1-(3-methoxy-3-oxoprop-1-en-2-yl)-2,4,6-triphenylpyridin-1-i um tetrafluoroborate (1k), purified by flash chromatography on silica gel (dichloromethane/acetone = 80:1), yellow solid, 70% yield.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm): 7.99 (s, 2H), 7.84 – 7.83 (m, 2H), 7.56 – 7.45 (m, 13H), 6.64 (s, 1H), 6.45 (s, 1H), 3.60 (s, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm): 161.8, 158.3, 156.4, 136.5, 134.2, 134.0, 132.3, 132.2, 130.8, 129.7, 129.5, 128.8, 128.4, 126.0, 53.4.

$^{19}$F NMR (376 MHz, CDCl$_3$): $\delta$ (ppm): -152.94, -152.99.

HRMS (EI): $m/z$ calculated for C$_{27}$H$_{22}$NO$_2$ (M-BF$_4$)$^+$: 392.1645, found 392.1647.

1-(1-carboxy-3-hydroxypropyl)-2,4,6-triphenylpyridin-1-i um tetrafluoroborate (II), purified by flash chromatography on silica gel (dichloromethane/acetone = 70:1), yellow solid, 77% yield.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm): 8.09 – 8.03 (m, 3H), 7.91 (s, 1H), 7.82 (d, $J = 7.6$ Hz, 2H), 7.71 – 7.49 (m, 11H), 5.66 (dd, $J = 11.2$, 9.2 Hz, 1H), 3.98 (q, $J = 8.4$ Hz, 1H), 3.38 – 3.32 (m, 1H), 3.03 – 2.93 (m, 1H), 2.78 – 2.68 (m, 1H).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm): 172.1, 159.2, 157.1, 156.2, 133.6, 132.8, 132.6, 131.8, 131.6, 131.4, 131.3, 130.3, 130.1, 129.9, 129.7, 129.6, 129.2, 128.8, 128.7, 128.4, 126.8, 66.6, 63.6, 27.3.
$^{19}$F NMR (376 MHz, CDCl3): $\delta$ (ppm): -152.84, -152.89.

HRMS (EI): $m/z$ calculated for C$_{27}$H$_{22}$NO$_2$ (M-BF$_4$)$^+$: 392.1645, found 392.1646.

1-(1-(benzyloxy)-1-oxopropan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (1m), recrystallized from EtOH/Et$_2$O (v/v 1:4), white solid, 80% yield.

$^1$H NMR (400 MHz, CDCl3): $\delta$ (ppm): 7.89 (s, 2H), 7.79 (d, $J = 7.6$ Hz, 2H), 7.67 – 7.35 (m, 16H), 7.22 (d, $J = 6.0$ Hz, 2H), 5.58 (q, $J = 7.2$ Hz, 1H), 5.15 (d, $J = 12.0$ Hz, 1H), 5.06 (d, $J = 12.0$ Hz, 1H), 1.42 (d, $J = 6.8$ Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl3): $\delta$ (ppm): 168.7, 156.9, 156.7, 133.9, 132.7, 132.2, 131.2, 129.6, 129.0, 128.8, 128.7, 128.43, 128.36, 127.7, 127.5, 126.9, 68.7, 64.8, 16.9.

$^{19}$F NMR (376 MHz, CDCl3): $\delta$ (ppm): -153.10, -153.16.

HRMS (EI): $m/z$ calculated for C$_{33}$H$_{28}$NO$_2$ (M-BF$_4$)$^+$: 470.2115, found 470.2116.

1-(1-ethoxy-4-methyl-1-oxopentan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (1n), recrystallized from EtOH/Et$_2$O (v/v 1:4), white solid, 81% yield.
"\^{1}H\ NMR\ (400\ MHz,\ CDCl_{3}):\ \delta\ (ppm):\ 7.93\ (s,\ 2H),\ 7.84\ (d,\ J = 7.2\ Hz,\ 2H),\ 7.73 - 7.49\ (m,\ 13H),\ 5.45\ (d,\ J = 5.2\ Hz,\ 1H),\ 4.18\ (dd,\ J = 12.4,\ 6.4\ Hz,\ 2H),\ 1.78 - 1.76\ (m,\ 1H),\ 1.51 - 1.44\ (m,\ 1H),\ 1.37 - 1.29\ (m,\ 1H),\ 1.22\ (t,\ J = 7.2\ Hz,\ 3H),\ 0.58\ (d,\ J = 6.4\ Hz,\ 3H),\ 0.46\ (d,\ J = 6.4\ Hz,\ 3H)."

"\^{13}C\ NMR\ (100\ MHz,\ CDCl_{3}):\ \delta\ (ppm):\ 168.9,\ 156.8,\ 156.7,\ 133.8,\ 133.8,\ 132.7,\ 132.2,\ 131.4,\ 129.6,\ 129.0,\ 128.5,\ 128.0,\ 67.7,\ 63.3,\ 40.2,\ 26.3,\ 22.3,\ 20.7,\ 13.8."

"\^{19}F\ NMR\ (376\ MHz,\ CDCl_{3}):\ \delta\ (ppm):\ -152.98,\ -153.00,\ -153.03,\ -153.05."

"HRMS\ (EI):\ m/z\ calculated\ for\ C_{31}H_{32}NO_{2}\ (M-BF_{4})^{+}:\ 450.2428,\ found\ 450.2429."
3. General Procedure for the Photoinduced Deaminative Thioesterification

A mixture of amino acid Katritzky salts 1 (0.20 mmol), thiobenzoic acid 2a (0.40 mmol), DIPEA (0.40 mmol) and DCM (2.0 mL) was stirred at room temperature with 6 W Blue LEDs for 24 h. After quenching the reaction, the mixture was purified by flash chromatography on silica gel to give the products 3.

**Reaction Setup:** Reactions were conducted in a 6 W photo-reactor (Model: H106062, GEAO CHEMICAL, Wuhan, China) as shown below. One place/hole with three 1W blue LED beads ($\lambda_{\text{max}} = 458$ nm) on both side, and the distance between the Schlenk tube and LED beads is ca. 2 cm.
**Methyl 2-(benzoylthio)-3-phenylpropanoate (3a)**

![Chemical Structure](image)

Prepared according to the general procedure using 1-(1-methoxy-1-oxo-3-phenylpropan-2-yl)-2,4,6-triphenylpyridin-1-ium-tetrafluoroborate (114.4 mg, 0.20 mmol, 1.0 equiv), thiobenzoic acid (46.8 μl, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 μl, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 15:1 to 10:1) delivered 3a as a liquid in 96% yield (57.6 mg).

Asymmetric induction was investigated with enantiomerically pure amino ester-derived Katritzky salt 1a under the standard conditions. The enantiomeric excess was determined by chiral HPLC analysis on Chiralpak OD-H column (n-hexane/2-propanol 95:5, 1 mL/min), UV 254 nm, t_r = 4.36 min and 4.69 min (1.3% ee).

Reaction was also performed in toluene, and the enantiomeric excess was determined to be 2%.

**1H NMR (400 MHz, CDCl3):** δ (ppm): 7.92 (d, J = 7.6 Hz, 2H), 7.57 (t, J = 7.6 Hz, 1H), 7.43 (t, J = 7.6 Hz, 2H), 7.32 – 7.21 (m, 5H), 4.65 (t, J = 7.6 Hz, 1H), 3.68 (s, 3H), 3.34 (dd, J = 14.0, 8.4 Hz, 1H), 3.14 (dd, J = 14.0, 8.4 Hz, 1H).

**13C NMR (100 MHz, CDCl3):** δ (ppm): 189.7, 171.6, 137.2, 136.1, 133.8, 129.1, 128.7, 128.5, 127.4, 127.1, 52.6, 47.1, 38.1.

**HRMS (EI):** m/z calculated for C_{17}H_{16}O_{3}S+H*: 301.0893, found 301.0887.

**Ethyl 2-(benzoylthio)-3-phenylpropanoate (3b)**

![Chemical Structure](image)
Prepared according to the general procedure using 1-(1-ethoxy-1-oxo-3-phenylpropan-2-yl)-2,4,6-triphenylpyridin-1-ium-tetrafluoroborate (114.2 mg, 0.20 mmol, 1.0 equiv), thiobenzoic acid (46.8 µl, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 µl, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 15:1 to 10:1) delivered 3b as a liquid in 94% yield (59.0 mg).

\[ ^1H \text{ NMR (400 MHz, CDCl}_3 \text{): } \delta \text{ (ppm): 7.91 (d, } J = 7.6 \text{ Hz, } 2H), 7.55 \text{ (t, } J = 7.2 \text{ Hz, } 1H), 7.41 \text{ (t, } J = 7.2 \text{ Hz, } 2H), 7.29 - 7.21 \text{ (m, } 5H), 4.61 \text{ (t, } J = 7.2 \text{ Hz, } 1H), 4.12 \text{ (q, } J = 7.2 \text{ Hz, } 2H), 3.31 \text{ (dd, } J = 13.6, 9.2 \text{ Hz, } 1H), 3.13 \text{ (dd, } J = 13.6, 6.4 \text{ Hz, } 1H), 1.15 \text{ (t, } J = 6.4 \text{ Hz, } 3H). \]

\[ ^{13}\text{C NMR (100 MHz, CDCl}_3 \text{): } \delta \text{ (ppm): 189.8, 171.0, 137.3, 136.3, 133.7, 129.2, 128.7, 128.4, 127.4, 127.0, 61.7, 47.4, 38.2, 14.0.} \]

HRMS (EI): \( m/z \) calculated for \( \text{C}_{18}\text{H}_{18}\text{O}_3\text{S}^+\text{H}^+ \): 315.1049, found 315.1042.

**Benzyl 2-(benzoylthio)-3-phenylpropanoate (3c)**

![Chemical Structure](image)

Prepared according to the general procedure using 1-(1-benzyloxy)-1-oxo-3-phenylpropan-2-yl)-2,4,6-triphenylpyridin-1-ium-tetrafluoroborate (126.6 mg, 0.20 mmol, 1.0 equiv), thiobenzoic acid (46.8 µl, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 µl, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 12:1 to 10:1) delivered 3c as a liquid in 86% yield (64.6 mg).

\[ ^1H \text{ NMR (400 MHz, CDCl}_3 \text{): } \delta \text{ (ppm): 7.91 (d, } J = 7.6 \text{ Hz, } 2H), 7.56 \text{ (t, } J = 7.6 \text{ Hz, } 1H), 7.43 \text{ (t, } J = 7.2 \text{ Hz, } 2H), 7.28 - 7.21 \text{ (m, } 10H), 5.11 \text{ (s, } 2H), 4.69 \text{ (t, } J = 8.0 \text{ Hz, } 1H), 3.33 \text{ (dd, } J = 13.6, 8.8 \text{ Hz, } 1H), 3.16 \text{ (dd, } J = 14.0, 6.8 \text{ Hz, } 1H). \]
\[^{13}\text{C NMR (100 MHz, CDCl}_3\text{): } \delta \text{ (ppm): 189.7, 170.9, 137.1, 136.2, 135.3, 133.7, 129.2, 128.7, 128.5, 128.4, 128.2, 128.1, 127.4, 127.0, 67.3, 47.4, 38.2.}\]

\[^{\text{HRMS (EI): } m/z \text{ calculated for C}}_{23}\text{H}_{20}\text{O}_{3}\text{S+H}^+: 377.1211, \text{found 377.1201.}\]

**Methyl 2-(benzoylthio)-3-(4-hydroxyphenyl)propanoate (3d)**

![Methyl 2-(benzoylthio)-3-(4-hydroxyphenyl)propanoate (3d) Structure](attachment:image)

Prepared according to the general procedure using 1-(3-(4-hydroxyphenyl)-1-methoxy-1-oxopropan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (114.6 mg, 0.20 mmol, 1.0 equiv), thiobenzoic acid (46.8 \(\mu\)l, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 \(\mu\)l, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 8:1 to 4:1) delivered 3d as a liquid in 89% yield (56.2 mg).

\[^{1}\text{H NMR (400 MHz, CDCl}_3\text{): } \delta \text{ (ppm): 7.93 (d, } J = 7.6 \text{ Hz, 2H), 7.58 (t, } J = 7.6 \text{ Hz, 1H), 7.44 (t, } J = 7.6 \text{ Hz, 2H), 7.12 (d, } J = 7.2 \text{ Hz, 2H), 6.76 (d, } J = 7.2 \text{ Hz, 2H), 4.61 (t, } J = 7.6 \text{ Hz, 1H), 3.69 (s, 3H), 3.26 (dd, } J = 14.0, 8.8 \text{ Hz, 1H), 3.08 (dd, } J = 14.0, 6.8 \text{ Hz, 1H).}\]

\[^{13}\text{C NMR (100 MHz, CDCl}_3\text{): } \delta \text{ (ppm): 190.1, 171.9, 154.8, 136.1, 133.8, 130.3, 129.2, 128.7, 127.4, 115.4, 52.7, 47.4, 37.3.}\]

\[^{\text{HRMS (EI): } m/z \text{ calculated for C}}_{17}\text{H}_{16}\text{O}_{4}\text{S+Na}^+: 339.0662, \text{found 339.0654.}\]
Methyl 2-(benzoylthio)-3-(3,4-dimethoxyphenyl)propanoate (3e)

Prepared according to the general procedure using 1-(3-(3,4-dimethoxyphenyl)-1-methoxy-1-oxopropan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (123.4 mg, 0.20 mmol, 1.0 equiv), thiobenzoic acid (46.8 µl, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 µl, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 6:1 to 4:1) delivered 3e as a liquid in 99% yield (71.3 mg).

$^1$H NMR (400 MHz, CDCl$_3$): δ (ppm): 7.92 (d, $J = 7.6$ Hz, 2H), 7.58 (t, $J = 7.6$ Hz, 1H), 7.44 (t, $J = 7.6$ Hz, 2H), 6.82 – 6.77 (m, 3H), 4.63 (t, $J = 8.0$ Hz, 1H), 3.87 (s, 3H), 3.85 (s, 3H), 3.70 (s, 3H), 3.29 (dd, $J = 14.0$, 8.4 Hz, 1H), 3.09 (dd, $J = 14.0$, 6.8 Hz, 1H).

$^{13}$C NMR (100 MHz, CDCl$_3$): δ (ppm): 189.8, 171.7, 148.8, 148.0, 136.1, 133.8, 129.7, 128.7, 127.3, 121.2, 112.1, 111.0, 55.8, 55.8, 52.6, 47.2, 37.8.

HRMS (EI): $m/z$ calculated for C$_{19}$H$_{20}$O$_5$S+H$: 361.1100, found 361.1098.
**Methyl 2-(benzoylthio)-3-(4-fluorophenyl)propanoate (3f)**

![Structure of 3f]

Prepared according to the general procedure using 1-(3-(4-fluorophenyl)-1-methoxy-1-oxopropan-2-yl)-2,4,6-triphenylpyridin-1-ium-tetrafluoroborate (115.0 mg, 0.20 mmol, 1.0 equiv), thiobenzoic acid (46.8 μl, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 μl, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 12:1 to 10:1) delivered 3f as a liquid in 76% yield (48.4 mg).

**1H NMR (400 MHz, CDCl3):** \(\delta\) (ppm): 7.92 (d, \(J = 8.0\) Hz, 2H), 7.58 (t, \(J = 7.6\) Hz, 1H), 7.45 (t, \(J = 7.6\) Hz, 2H), 7.25 – 7.22 (m, 2H), 6.98 (t, \(J = 8.8\) Hz, 2H), 4.61 (t, \(J = 7.6\) Hz, 1H), 3.69 (s, 3H), 3.31 (dd, \(J = 14.0, 8.4\) Hz, 1H), 3.11 (dd, \(J = 14.0, 7.2\) Hz, 1H).

**13C NMR (100 MHz, CDCl3):** \(\delta\) (ppm): 189.6, 171.4, 161.9 (d, \(J = 243.7\) Hz, 1C), 136.1, 133.9, 132.9 (d, \(J = 3.2\) Hz, 1C), 130.7 (d, \(J = 8.0\) Hz, 1C), 128.7, 127.4, 115.3 (d, \(J = 21.2\) Hz, 1C), 52.7, 47.0, 37.4.

**HRMS (EI):** m/z calculated for C_{17}H_{15}FO_{3}S+H^+: 319.0799, found 319.0792.

**Methyl 2-(benzoylthio)-3-(4-nitrophenyl)propanoate (3g)**

![Structure of 3g]
Prepared according to the general procedure using 1-(1-methoxy-3-(4-nitrophenyl)-1-oxopropan-2-yl)-2,4,6-triphenylpyridin-1-ium-tetrafluoroborate (120.4 mg, 0.20 mmol, 1.0 equiv), thiobenzoic acid (46.8 μl, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 μl, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 4:1) delivered 3g as a liquid in 47% yield (32.4 mg).

$^1$H NMR (400 MHz, CDCl$_3$): δ (ppm): 8.16 (d, $J = 7.2$ Hz, 2H), 7.91 (d, $J = 7.2$ Hz, 2H), 7.60 (t, $J = 7.2$ Hz, 1H), 7.47 – 7.46 (m, 4H), 4.69 (t, $J = 7.2$ Hz, 1H), 3.71 (s, 3H), 3.46 (dd, $J = 13.6$, 8.8 Hz, 1H), 3.24 (dd, $J = 13.6$, 6.8 Hz, 1H).

$^{13}$C NMR (100 MHz, CDCl$_3$): δ (ppm): 189.1, 171.0, 147.1, 145.0, 135.9, 134.1, 130.2, 128.8, 127.4, 123.7, 52.9, 46.2, 38.0.

HRMS (EI): $m/z$ calculated for C$_{17}$H$_{15}$NO$_5$S$^+Na^+$: 368.0563, found 368.0558.

**Methyl 2-(benzoylthio)-4-phenylbutanoate (3h)**

![Methyl 2-(benzoylthio)-4-phenylbutanoate](image)

Prepared according to the general procedure using 1-(1-methoxy-1-oxo-4-phenylbutan-2-yl)-2,4,6-triphenylpyridin-1-ium-tetrafluoroborate (114.2 mg, 0.20 mmol, 1.0 equiv), thiobenzoic acid (46.8 μl, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 μl, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 10:1) delivered 3h as a liquid in 75% yield (47.0 mg).

$^1$H NMR (400 MHz, CDCl$_3$): δ (ppm): 7.96 (d, $J = 7.6$ Hz, 2H), 7.59 (t, $J = 7.6$ Hz, 1H), 7.46 (t, $J = 7.2$ Hz, 2H), 7.30 – 7.25 (m, 2H), 7.20 – 7.18 (m, 3H), 4.44 (t, $J = 6.9$ Hz, 1H), 3.74 (s, 3H), 2.83 – 2.69 (m, 2H), 2.40 – 2.31 (m, 1H), 2.23 – 2.11 (m, 1H).
$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm): 189.8, 172.0, 140.4, 136.2, 133.8, 128.7, 128.5, 128.4, 127.4, 126.2, 52.7, 45.4, 33.5, 33.3.

HRMS (EI): $m/z$ calculated for C$_{18}$H$_{16}$O$_3$S$^+$: 315.1049, found 315.1041.

Diethyl 2-(benzoylthio)pentanedioate (3i)

```
EtO2C-S
Ph
CO2Et
```

Prepared according to the general procedure using 1-(1,5-diethoxy-1,5-dioxopentan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (116.2 mg, 0.20 mmol, 1.0 equiv), thiobenzoic acid (46.8 $\mu$l, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 $\mu$l, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 8:1) delivered 3i as a liquid in 80% yield (51.8 mg).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm): 7.95 (d, $J = 7.2$ Hz, 2H), 7.59 (t, $J = 7.2$ Hz, 1H), 7.46 (t, $J = 7.2$ Hz, 2H), 4.46 (t, $J = 6.4$ Hz, 1H), 4.22 (q, $J = 6.4$ Hz, 2H), 4.14 (q, $J = 6.4$ Hz, 2H), 2.50 – 2.46 (m, 2H), 2.42 – 2.35 (m, 1H), 2.24 – 2.15 (m, 1H), 1.30 – 1.24 (m, 6H).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm): 189.6, 172.2, 171.1, 136.2, 133.8, 128.7, 127.4, 61.8, 60.6, 45.2, 31.6, 27.2, 14.2, 14.1.

HRMS (EI): $m/z$ calculated for C$_{16}$H$_{20}$O$_5$S$^+$: 325.1100, found 325.1095.
Dimethyl 2-(benzoylthio)succinate (3j)

Prepared according to the general procedure using 1-(1,4-dimethoxy-1,4-dioxobutan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (107.8 mg, 0.20 mmol, 1.0 equiv), thiobenzoic acid (46.8 μl, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 μl, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 8:1) delivered 3j as a liquid in 70% yield (39.5 mg).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm): 7.95 (d, $J = 8.0$ Hz, 2H), 7.60 (t, $J = 7.6$ Hz, 1H), 7.46 (t, $J = 7.6$ Hz, 2H), 4.80 – 4.77 (m, 1H), 3.77 (s, 3H), 3.72 (s, 3H), 3.11 (dd, $J = 16.8$, 7.6 Hz, 1H), 2.98 (dd, $J = 17.2$, 5.6 Hz, 1H).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm): 189.2, 170.9, 170.9, 135.9, 134.0, 128.8, 127.5, 53.1, 52.1, 40.8, 36.7.

HRMS (EI): $m/z$ calculated for C$_{13}$H$_{14}$O$_5$S+H$: 283.0635, found 283.0629.

Methyl 2,3-bis(benzoylthio)propanoate (3k)

Prepared according to the general procedure using 1-(3-methoxy-3-oxoprop-1-en-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (95.8 mg, 0.20 mmol, 1.0 equiv), thiobenzoic acid (93.6 μl, 0.80 mmol, 4.0 equiv) and DIPEA (128.0 μl, 0.80 mmol, 4.0 equiv). After quenching the reaction, purification of
the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 10:1 to 5:1) delivered 3k as a liquid in 58% yield (41.8 mg).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm): 7.97 – 7.96 (m, 4H), 7.62 – 7.56 (m, 2H), 7.48 – 7.43 (m, 4H), 4.72 (t, $J = 7.2$ Hz, 1H), 3.79 (s, 3H), 3.72 (dd, $J = 13.6$, 8.0 Hz, 1H), 3.63 (dd, $J = 13.6$, 6.4 Hz, 1H).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm): 190.4, 188.8, 170.7, 136.5, 135.9, 134.0, 133.6, 128.8, 128.6, 127.6, 127.4, 53.0, 45.2, 30.5.

HRMS (EI): $m/z$ calculated for C$_{18}$H$_{16}$O$_4$S$_2$+Na$: 383.0382, found 383.0379.

**Methyl 2-(benzoylthio)-4-hydroxybutanoate (3l)**

Prepared according to the general procedure using 1-(4-hydroxy-1-methoxy-1-oxobutan-2-yl)-2,4,6-triphenylpyridin-1-ium-tetrafluoroborate (102.2 mg, 0.20 mmol, 1.0 equiv), thiobenzoic acid (46.8 $\mu$l, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 $\mu$l, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 10:1 to 5:1) delivered 3l as a liquid in 54% yield (23.8 mg).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm): 7.95 (d, $J = 7.6$ Hz, 2H), 7.62 (t, $J = 7.6$ Hz, 1H), 7.48 (t, $J = 7.6$ Hz, 2H), 4.57 – 4.52 (m, 1H), 4.47 – 4.37 (m, 2H), 2.91 – 2.83 (m, 1H), 2.46 – 2.36 (m, 1H).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm): 189.5, 174.5, 135.7, 134.2, 128.8, 127.5, 66.8, 40.2, 30.5.

HRMS (EI): $m/z$ calculated for C$_{11}$H$_{10}$O$_3$S+Na$: 245.0243, found 245.0241.
**Benzyl 2-(benzoylthio)propanoate (3m)**

Prepared according to the general procedure using 1-(1-(benzyloxy)-1-oxopropan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (101.4 mg, 0.20 mmol, 1.0 equiv), thiobenzoic acid (46.8 μl, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 μl, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 10:1 to 8:1) delivered 3m as a liquid in 74% yield (44.4 mg).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm): 7.94 (d, $J = 7.2$ Hz, 2H), 7.59 (t, $J = 7.2$ Hz, 1H), 7.45 (t, $J = 7.2$ Hz, 2H), 7.35 – 7.33 (m, 5H), 5.21 (s, 2H), 4.50 (q, $J = 6.8$ Hz, 1H), 1.63 (d, $J = 6.4$ Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm): 190.0, 171.9, 136.3, 135.5, 133.7, 128.7, 128.5, 128.2, 128.0, 127.3, 67.3, 41.0, 17.7.

HRMS (EI): $m/z$ calculated for C$_{17}$H$_{16}$O$_3$S+Na$: 323.0712, found 323.0704.

**Ethyl 2-(benzoylthio)-4-methylpentanoate (3n)**

Prepared according to the general procedure using 1-(1-ethoxy-4-methyl-1-oxopentan-2-yl)-2,4,6-triphenylpyridin-1-ium-tetrafluoroborate -te (107.4 mg, 0.20 mmol, 1.0 equiv), thiobenzoic acid (46.8 μl, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 μl, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification
of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 12:1) delivered 3n as a liquid in 82% yield (46.0 mg).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm): 7.96 (d, $J = 7.6$ Hz, 2H), 7.58 (t, $J = 7.6$ Hz, 1H), 7.45 (t, $J = 7.6$ Hz, 2H), 4.43 (t, $J = 7.6$ Hz, 1H), 4.22 (q, $J = 7.2$ Hz, 2H), 1.96 – 1.89 (m, 1H), 1.79 – 1.67 (m, 2H), 1.29 (t, $J = 7.2$ Hz, 3H), 1.00 (d, $J = 6.0$ Hz, 3H), 0.95 (d, $J = 6.0$ Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm): 190.2, 172.1, 136.4, 133.7, 128.7, 127.4, 61.6, 44.5, 40.8, 26.3, 22.3, 22.3, 14.1.

HRMS (EI): $m/z$ calculated for C$_{15}$H$_{20}$O$_3$S+: 281.1206, found 281.1201.

Methyl 2-(benzoylthio)-4-(methylthio)butanoate (3o)

\[
\begin{align*}
\text{O} & \quad \text{Ph} \\
\text{MeO}_2\text{C-S} & \quad \text{3o}
\end{align*}
\]

Prepared according to the general procedure using 1-(1-methoxy-4-(methylthio)-1-oxobutan-2-yl)-2,4,6-triphenylpyridin-1-ium-tetrafluoroborate (108.2 mg, 0.20 mmol, 1.0 equiv), thiobenzoic acid (46.8 $\mu$l, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 $\mu$l, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 8:1) delivered 3o as a liquid in 92% yield (52.2 mg).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm): 7.96 (d, $J = 7.6$ Hz, 2H), 7.59 (t, $J = 7.6$ Hz, 1H), 7.46 (t, $J = 7.6$ Hz, 2H), 4.57 (t, $J = 7.2$ Hz, 1H), 3.77 (s, 3H), 2.68 – 2.56 (m, 2H), 2.39 – 2.29 (m, 1H), 2.20 – 2.14 (m, 1H), 2.11 (s, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm): 189.6, 171.76, 136.1, 133.8, 128.7, 127.4, 52.8, 44.6, 31.3, 31.3, 15.4.

HRMS (EI): $m/z$ calculated for C$_{13}$H$_{16}$O$_3$S$_2$+Na+: 307.0433, found 307.0430.
**Methyl 2-(benzoylthio)-6-(((benzyloxy)carbonyl)amino)hexanoate (3p)**

![Chemical Structure of 3p]

Prepared according to the general procedure using 1-(6-(((benzyloxy)carbonyl)amino)-1-methoxy-1-oxohexan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (134.4 mg, 0.20 mmol, 1.0 equiv), thiobenzoic acid (46.8 μl, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 μl, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 8:1 to 4:1) delivered 3p as a liquid in 76% yield (63.0 mg).

**1H NMR (400 MHz, CDCl3):** δ (ppm): 7.95 (d, J = 7.6 Hz, 2H), 7.58 (t, J = 7.2 Hz, 1H), 7.45 (t, J = 7.6 Hz, 2H), 7.34 – 7.31 (m, 5H), 5.08 (s, 2H), 4.80 (s, 1H), 4.41 (t, J = 6.8 Hz, 1H), 3.75 (s, 3H), 3.20 – 3.19 (m, 2H), 2.08 – 2.01 (m, 1H), 1.92 – 1.87 (m, 1H), 1.58 – 1.54 (m, 1H), 1.47 – 1.46 (m, 2H).

**13C NMR (100 MHz, CDCl3):** δ (ppm): 190.0, 172.0, 156.3, 136.6, 136.2, 133.8, 128.7, 128.5, 128.0, 127.4, 66.6, 52.7, 45.5, 40.7, 31.5, 29.4, 24.2.

**HRMS (EI):** m/z calculated for C_{22}H_{25}NO_{5}S+Na+: 438.1346, found 438.1338.

**Ethyl 2-(benzoylthio)-3-(1H-indol-3-yl)propanoate (3q)**

![Chemical Structure of 3q]
Prepared according to the general procedure using 1-(1-ethoxy-3-(1H-indol-3-yl)-1-oxopropan-2-yl)-2,4,6-triphenylpyridin-1-ium-tetrafluoroborate (122.0 mg, 0.20 mmol, 1.0 equiv), thiobenzoic acid (46.8 µl, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 µl, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 8:1 to 4:1) delivered 3q as a liquid in 58% yield (41.0 mg).

$^1$H NMR (400 MHz, CDCl$_3$): δ (ppm): 8.06 (s, 1H), 7.96 (d, $J = 7.2$ Hz, 2H), 7.77 (d, $J = 7.6$ Hz, 1H), 7.58 (t, $J = 7.2$ Hz, 1H), 7.45 (t, $J = 7.2$ Hz, 2H), 7.35 (d, $J = 7.6$ Hz, 1H), 7.22 – 7.15 (m, 2H), 7.11 (s, 1H), 4.75 (t, $J = 6.8$ Hz, 1H), 4.12 (q, $J = 6.8$ Hz, 2H), 3.51 (dd, $J = 14.4$, 9.2 Hz, 1H), 3.35 (dd, $J = 14.4$, 6.4 Hz, 1H), 1.15 (t, $J = 6.8$ Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$): δ (ppm): 190.2, 171.5, 136.4, 136.0, 133.7, 128.7, 127.4, 127.3, 122.9, 122.1, 119.6, 118.9, 111.9, 111.1, 61.6, 46.9, 28.3, 13.9.

HRMS (EI): m/z calculated for C$_{20}$H$_{19}$NO$_3$S+H$: 354.1158, found 354.1151.

tert-Butyl-2-(3-(benzoylthio)-2-oxo-2,3,4,5-tetrahydro-1H-benzo[b]azepin-1-yl) acetate (3r)

Prepared according to the general procedure using 1-(1-(2-(tert-butoxy)-2-oxoethyl)-2-oxo-2,3,4,5-tetrahydro-1H-benzo[b]azepin-3-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (133.6 mg, 0.20 mmol, 1.0 equiv), thiobenzoic acid (46.8 µl, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 µl, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column
chromatography on silica gel (petroleum ether/ethyl acetate = 8:1) delivered **3r** as a liquid in 49% yield (40.2 mg).

**1H NMR (400 MHz, CDCl₃):** \( \delta \) (ppm): 7.90 (d, \( J = 7.6 \) Hz, 2H), 7.54 (t, \( J = 7.2 \) Hz, 1H), 7.40 (t, \( J = 7.6 \) Hz, 2H), 7.34 (t, \( J = 7.6 \) Hz, 1H), 7.28 – 7.21 (m, 3H), 4.73 (d, \( J = 16.8 \) Hz, 1H), 4.51 – 4.46 (m, 1H), 4.25 (d, \( J = 17.2 \) Hz, 1H), 3.53 – 3.46 (m, 1H), 2.74 – 2.70 (m, 1H), 2.55 – 2.42 (m, 2H), 1.41 (s, 9H).

**13C NMR (100 MHz, CDCl₃):** \( \delta \) (ppm): 190.8, 170.0, 167.6, 141.5, 136.5, 135.3, 133.5, 129.7, 128.6, 128.1, 127.2, 127.1, 122.6, 82.0, 51.4, 45.8, 35.9, 29.7, 28.0.

**HRMS (EI):** \( m/z \) calculated for \( \text{C}_{23}\text{H}_{25}\text{NO}_{4}\text{S}^+ \): 412.1577, found 412.1576.
4. General Procedure for the Photoinduced Deaminative Thiolation

A mixture of asparagine amino acid Katritzky salts 1 (0.20 mmol), thiols 2 (0.40 mmol), DIPEA (0.40 mmol) and DCM (2.0 mL) was stirred at room temperature with 6 W Blue LEDs for 36 h. After quenching the reaction, the mixture was purified by flash chromatography on silica gel to give the products 4.

**Dimethyl 2-(butylthio)succinate (4a)**

Prepared according to the general procedure using 1-(1,4-dimethoxy-1,4-dioxobutan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (107.8 mg, 0.20 mmol, 1.0 equiv), butanethiol (43.2 µl, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 µl, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 12:1) delivered 4a as a liquid in 80% yield (37.4 mg).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm): 3.76 (s, 3H), 3.69 (s, 3H), 3.67 – 3.65 (m, 1H), 3.00 (dd, $J = 17.2$, 10.0 Hz, 1H), 2.70 – 2.59 (m, 3H), 1.59 – 1.50 (m, 2H), 1.43 – 1.34 (m, 2H), 0.90 (t, $J = 7.2$ Hz, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm): 172.3, 171.2, 52.5, 52.0, 41.4, 36.3, 31.2 (2C), 21.9, 13.6.

HRMS (EI): $m/z$ calculated for $\text{C}_{10}\text{H}_{18}\text{O}_4\text{S}^+\text{Na}^+$: 257.0818, found 257.0812.

**Dimethyl 2-(dodecylthio)succinate (4b)**
Prepared according to the general procedure using 1-(1,4-dimethoxy-1,4-dioxobutan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (107.8 mg, 0.20 mmol, 1.0 equiv), dodecy mercaptan (95.6 μl, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 μl, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 12:1) delivered 4b as oil liquid in 85% yield (58.8 mg).

\(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) (ppm): 3.76 (s, 3H), 3.69 (s, 3H), 3.69 – 3.65 (m, 1H), 3.00 (dd, \(J = 17.2, 10.0\) Hz, 1H), 2.71 – 2.58 (m, 3H), 1.58 – 1.50 (m, 2H), 1.35 – 1.25 (m, 18H), 0.87 (t, \(J = 6.4\) Hz, 3H).

\(^13\)C NMR (100 MHz, CDCl\(_3\)): \(\delta\) (ppm): 172.3, 171.2, 52.5, 52.0, 41.4, 36.4, 31.9, 31.5, 29.62, 29.60, 29.56, 29.5, 29.3, 29.17, 29.14, 28.8, 22.7, 14.1.

HRMS (EI): \(m/z\) calculated for C\(_{18}\)H\(_{34}\)O\(_4\)S\(^+\):369.2070, found 369.2071.

**Dimethyl 2-(phenethylthio)succinate (4c)**

Prepared according to the general procedure using 1-(1,4-dimethoxy-1,4-dioxobutan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (107.8 mg, 0.20 mmol, 1.0 equiv), 2-phenylethanethiol (53.4 μl, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 μl, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 9:1) delivered 4c as a liquid in 73% yield (41.2 mg).

\(^1\)H NMR (400 MHz, CDCl\(_3\)): \(\delta\) (ppm): 7.30 (t, \(J = 7.2\) Hz, 2H), 7.24 – 7.19 (m, 3H), 3.76 (s, 3H), 3.72 – 3.69 (m, 1H), 3.69 (s, 3H), 3.01 (dd, \(J = 16.8, 10.0\) Hz, 1H), 2.96
– 2.84 (m, 4H), 2.67 (dd, J = 17.2, 5.6 Hz, 1H).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm): 172.1, 171.1, 139.9, 128.5, 126.5, 52.5, 52.0, 41.5, 36.3, 35.7, 33.0.

HRMS (EI): $m/z$ calculated for C$_{14}$H$_{18}$O$_4$S+: 283.0999, found 283.0994.

**Dimethyl 2-(cyclohexylthio)succinate (4d)**

![4d](image)

Prepared according to the general procedure using 1-(1,4-dimethoxy-1,4-dioxobutan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (107.8 mg, 0.20 mmol, 1.0 equiv), cyclohexyl mercaptan (48.8 $\mu$l, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 $\mu$l, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 10:1) delivered 4d as a liquid in 81% yield (42.0 mg).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm): 3.75 (s, 3H), 3.75 – 3.71 (m, 1H), 3.68 (s, 3H), 2.99 (dd, J =16.8, 10.0 Hz, 1H), 2.93 – 2.81 (m, 1H), 2.66 (dd, J =16.8, 5.6 Hz, 1H), 2.04 – 1.92 (m, 2H), 1.75 – 1.73 (m, 2H), 1.61– 1.58 (m, 1H), 1.37 – 1.21 (m, 5H).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm): 172.7, 171.2, 52.4, 52.0, 44.3, 40.3, 36.9, 33.6, 33.4, 26.0, 25.8, 25.6.

HRMS (EI): $m/z$ calculated for C$_{12}$H$_{20}$O$_4$S+Na$: 283.0975, found 283.0971.

**Dimethyl 2-((3-oxobutan-2-yl)thio)succinate (4e)**

![4e](image)
Prepared according to the general procedure using 1-(1,4-dimethoxy-1,4-dioxobutan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (107.8 mg, 0.20 mmol, 1.0 equiv), 3-mercapto-2-butanone (40.0 μl, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 μl, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 8:1) delivered 4e as a liquid in 58% yield (28.8 mg).

$^1$H NMR (400 MHz, CDCl$_3$): δ (ppm): 3.75 (s, 3H), 3.69 (s, 3H), 3.71 – 3.60 (m, 2H), 3.00 – 2.93 (m, 1H), 2.72 – 2.64 (m, 1H), 2.26 (s, 3H), 1.42 (d, $J$ = 7.2 Hz, 1.5 H), 1.40 (d, $J$ = 7.2 Hz, 1.5 H).

$^{13}$C NMR (100 MHz, CDCl$_3$): 205.5 (205.3), 171.9 (171.8), 170.7 (170.6), 52.6 (52.1), 49.7 (48.8), 45.5, 41.5 (41.0), 36.7 (36.3), 26.1 (25.7), 16.7 (16.4).

HRMS (EI): m/z calculated for C$_{10}$H$_{16}$O$_5$S$^+$/Na$: 271.0611$, found 271.0606.

**Dimethyl 2-(m-tolylthio)succinate (4f)**

![Chemical structure](image)

Prepared according to the general procedure using 1-(1,4-dimethoxy-1,4-dioxobutan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (107.8 mg, 0.20 mmol, 1.0 equiv), 3-methylbenzenethiol (47.6 μl, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 μl, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 14:1) delivered 4f as a liquid in 79% yield (42.4 mg).

$^1$H NMR (400 MHz, CDCl$_3$): δ (ppm): 7.28 (d, $J$ = 7.2 Hz, 2H), 7.21 (t, $J$ = 7.6 Hz, 1H), 7.14 (d, $J$ = 7.2 Hz, 1H), 4.03 – 3.99 (m, 1H), 3.70 (s, 3H), 3.68 (s, 3H), 2.96 (dd, $J$ = 16.8, 9.6 Hz, 1H), 2.74 (dd, $J$ = 17.2, 5.6 Hz, 1H), 2.34 (s, 3H).

$^{13}$C NMR (100 MHz, CDCl$_3$): 171.6, 171.1, 138.9, 134.7, 131.3, 131.1, 129.6, 128.9, 52.5, 52.0, 45.6, 36.4, 21.2.

HRMS (EI): m/z calculated for C$_{13}$H$_{16}$O$_4$S$^+$/Na$: 291.0662$, found 291.0656.
**Dimethyl 2-((p-tolylthio)succinate (4g)**

![Chemical Structure](image)

Prepared according to the general procedure using 1-(1,4-dimethoxy-1,4-dioxobutan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (107.8 mg, 0.20 mmol, 1.0 equiv), p-toluenethiol (49.6 mg, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 μl, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 14:1) delivered 4g as a liquid in 81% yield (43.4 mg).

**1H NMR (400 MHz, CDCl₃):** δ (ppm): 7.36 (d, J = 7.6 Hz, 2H), 7.14 (d, J = 7.6 Hz, 2H), 3.95 (dd, J = 9.6, 5.6 Hz, 1H), 3.71 (s, 3H), 3.68 (s, 3H), 2.92 (dd, J = 17.2, 9.6 Hz, 1H), 2.72 (dd, J = 16.8, 5.6 Hz, 1H), 2.35 (s, 3H).

**13C NMR (100 MHz, CDCl₃):** δ (ppm): 171.5, 171.1, 139.3, 134.8, 129.9, 127.6, 52.5, 52.0, 45.7, 36.3, 21.2.

**HRMS (EI):** m/z calculated for C₁₃H₁₆O₄S+H⁺: 269.0842, found 269.0838.

**Dimethyl 2-((4-(tert-butyl)phenyl)thio)succinate (4h)**

![Chemical Structure](image)

Prepared according to the general procedure using 1-(1,4-dimethoxy-1,4-dioxobutan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (107.8 mg, 0.20 mmol, 1.0 equiv), 4-tert-butthiophenol (68.8 μl, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 μl, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 14:1) delivered 4h as a liquid in 74% yield (45.8 mg).

**1H NMR (400 MHz, CDCl₃):** δ (ppm): 7.40 (d, J = 7.2 Hz, 2H), 7.34 (d, J = 7.2 Hz, 2H), 7.22 (d, J = 7.2 Hz, 2H), 7.10 (d, J = 7.2 Hz, 2H), 3.92 (dd, J = 9.6, 5.6 Hz, 1H), 3.70 (s, 3H), 3.66 (s, 3H), 2.91 (dd, J = 17.2, 9.6 Hz, 1H), 2.70 (dd, J = 16.8, 5.6 Hz, 1H), 2.34 (s, 3H).
2H), 3.98 - 3.95 (m, 1H), 3.71 (s, 3H), 3.67 (s, 3H), 2.94 (dd, \(J = 16.8, \ 9.6\ Hz, \ 1H\)), 2.73 (dd, \(J = 17.2, \ 5.2\ Hz, \ 1H\)), 1.31 (s, 9H).

**\(^{13}C\) NMR (100 MHz, CDCl\(_3\)):** \(\delta\) (ppm): 171.6, 171.1, 152.3, 134.3, 127.8, 126.2, 52.5, 52.0, 45.7, 36.4, 34.7, 31.2.

**HRMS (EI):** \(m/z\) calculated for C\(_{16}\)H\(_{22}\)O\(_4\)S\(^+\): 333.1131, found 333.1127.

**Dimethyl 2-((4-methoxyphenyl)thio)succinate (4i)**

![Image of 4i](image)

Prepared according to the general procedure using 1-(1,4-dimethoxy-1,4-dioxobutan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (107.8 mg, 0.20 mmol, 1.0 equiv), 4-methoxybenzenethiol (49.2 \(\mu\)l, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 \(\mu\)l, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 10:1) delivered 4i as a liquid in 80% yield (45.4 mg).

**\(^1H\) NMR (400 MHz, CDCl\(_3\)):** \(\delta\) (ppm): 7.40 (d, \(J = 7.6\ Hz, \ 2H\)), 6.85 (d, \(J = 7.6\ Hz, \ 2H\)), 3.90 - 3.86 (m, 1H), 3.80 (s, 3H), 3.70 (s, 3H), 3.67 (s, 3H), 2.89 (dd, \(J = 16.8, \ 9.6\ Hz, \ 1H\)), 2.70 (dd, \(J = 16.8, \ 5.6\ Hz, \ 1H\)).

**\(^{13}C\) NMR (100 MHz, CDCl\(_3\)):** \(\delta\) (ppm): 171.5, 171.1, 160.7, 137.1, 121.4, 114.6, 55.3, 52.4, 52.0, 46.0, 36.2.

**HRMS (EI):** \(m/z\) calculated for C\(_{13}\)H\(_{16}\)O\(_5\)S\(^+\): 307.0611, found 307.0606.

**Dimethyl 2-((3-fluorophenyl)thio)succinate (4j)**

![Image of 4j](image)

Prepared according to the general procedure using
1-(1,4-dimethoxy-1,4-dioxobutan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (107.8 mg, 0.20 mmol, 1.0 equiv), 3-fluorothiophenol (33.8 μl, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 μl, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 14:1) delivered 4j as a liquid in 62% yield (33.8 mg).

\[ ^1H \text{ NMR (400 MHz, CDCl}_3 \]: } \delta (ppm): 7.33 – 7.20 (m, 3H), 7.03 (t, J = 8.0 Hz, 1H), 4.07 – 4.03 (m, 1H), 3.72 (s, 3H), 3.69 (s, 3H), 2.98 (dd, J = 17.2, 9.6 Hz, 1H), 2.76 (dd, J = 17.2, 5.6 Hz, 1H).

\[ ^{13}C \text{ NMR (100 MHz, CDCl}_3 \]: } \delta (ppm): 171.3, 170.8, 162.50 (d, J = 247.9 Hz, 1C), 134.11 (d, J = 7.9 Hz, 1C), 130.33 (d, J = 8.3 Hz, 1C), 129.04 (d, J = 3.0 Hz, 1C), 120.20 (d, J = 22.1 Hz, 1C), 115.71 (d, J = 20.9 Hz, 1C), 52.6, 52.1, 45.5, 36.3.

HRMS (EI): m/z calculated for C\(_{12}\)H\(_{13}\)FO\(_4\)S+Na\(^+\): 295.0416, found 295.0408.

**Dimethyl 2-((4-fluorophenyl)thio)succinate (4k)**

![4k](image)

Prepared according to the general procedure using 1-(1,4-dimethoxy-1,4-dioxobutan-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (107.8 mg, 0.20 mmol, 1.0 equiv), 4-fluorothiophenol (42.6 μl, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 μl, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 14:1) delivered 4k as a liquid in 58% yield (31.6 mg).

\[ ^1H \text{ NMR (400 MHz, CDCl}_3 \]: } \delta (ppm): 7.47 (t, J = 6.0 Hz, 2H), 7.03 (t, J = 8.0 Hz, 2H), 3.95 – 3.92 (m, 1H), 3.70 (s, 3H), 3.69 (s, 3H), 2.92 (dd, J = 16.8, 9.6 Hz, 1H), 2.71 (dd, J = 16.8, 5.6 Hz, 1H).

\[ ^{13}C \text{ NMR (100 MHz, CDCl}_3 \]: } \delta (ppm): 171.3, 170.9, 163.42 (d, J = 248.4 Hz, 1C), 137.01 (d, J = 8.4 Hz, 1C), 126.46 (d, J = 3.4 Hz, 1C), 116.27 (d, J = 21.8 Hz, 1C), 52.5, 52.1, 46.0, 36.2.
HRMS (EI): $m/z$ calculated for C$_{12}$H$_{13}$FO$_4$S+Na$^+$: 295.0416, found 295.0408.

**Dimethyl 2-((4-(trifluoromethyl)phenyl)thio)succinate (4I)**

![Dimethyl 2-((4-(trifluoromethyl)phenyl)thio)succinate (4I)](image)

Prepared according to the general procedure using 1-(1,4-dimethoxy-1,4-dioxobutan-2-yl)-2,4,6-triphenylpyridin-1-iium tetrafluoroborate (107.8 mg, 0.20 mmol, 1.0 equiv), 4-(trifluoromethyl) thiophenol (54.8 $\mu$l, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 $\mu$l, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 12:1) delivered 4I as a liquid in 91% yield (58.6 mg).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm): 7.57 (s, 4H), 4.13 (dd, $J = 9.2$, 6.0 Hz, 1H), 3.73 (s, 3H), 3.70 (s, 3H), 3.01 (dd, $J = 17.2$, 9.2 Hz, 1H), 2.77 (dd, $J = 17.2$, 6.0 Hz, 1H).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm): 171.2, 170.7, 132.4, 130.0, 128.8 (q, $J = 270.4$ Hz, 1C), 125.9 (q, $J = 3.7$ Hz, 1C), 52.8, 52.2, 45.0, 36.2.

HRMS (EI): $m/z$ calculated for C$_{13}$H$_{13}$F$_3$O$_4$S+H$^+$: 323.0559, found 323.0554.
5. Scale-up Reaction and Deprotection to Free Thiol

A scaled-up reaction was conducted with 1e (2 mmol, 1.23 g) under the standard conditions. The isolated yield of 3e was 99%. A mixture of the obtained product 3e, aq. NaOH (1.0 M, 3 mL) and MeOH (30 mL) was degassed for three times and then stirred at room temperature under argon for 24 h. The reaction was then acidified with 15 mL of aq. HCl (0.2 M), extracted with degassed DCM, and washed with saturated sodium chloride solution. Concentration and further purification by flash chromatography on silica gel (petroleum ether/ethyl acetate = 8:1 to 4:1) gave the products 5 in 93 % yield (474 mg).

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ (ppm): 6.79 - 6.77 (m, 1H), 6.73 - 6.70 (m, 2H), 3.84 (s, 6H), 3.68 (s, 3H), 3.59 – 3.53 (m, 1H), 3.17 (dd, $J = 14.0$, 8.8 Hz, 1H), 2.95 (dd, $J = 14.0$, 6.8 Hz, 1H), 2.10 (d, $J = 8.8$ Hz, 1H).

$^{13}$C NMR (100 MHz, CDCl$_3$): $\delta$ (ppm): 173.2, 148.8, 148.0, 130.1, 121.1, 112.1, 111.1, 55.8 (2C), 52.4, 42.4, 41.4.

HRMS (EI): $m/z$ calculated for C$_{12}$H$_{16}$O$_4$S+Na$^+$: 279.0667, found 279.0667.
6. Tandem Reaction with Benzenedithiol

Prepared according to the general procedure using 1-(3-methoxy-3-oxoprop-1-en-2-yl)-2,4,6-triphenylpyridin-1-ium tetrafluoroborate (1k, 95.8 mg, 0.20 mmol, 1.0 equiv), 1,2-benzenedithiol (46.0 μl, 0.40 mmol, 2.0 equiv) and DIPEA (64.0 μl, 0.40 mmol, 2.0 equiv). After quenching the reaction, purification of the crude product via column chromatography on silica gel (petroleum ether/ethyl acetate = 12:1) delivered 6 as a solid in 54% yield (24.4 mg).

$^1$H NMR (400 MHz, CDCl$_3$): δ (ppm): 7.33 – 7.26 (m, 2H), 7.09 – 7.07 (m, 2H), 4.33 (t, $J$ = 6.0 Hz, 1H), 3.79 (s, 3H), 3.33 (d, $J$ = 6.0 Hz, 2H).

$^{13}$C NMR (100 MHz, CDCl$_3$): δ (ppm): 170.1, 133.1, 131.7, 129.4, 129.0, 126.2, 125.9, 53.0, 46.8, 31.3.

HRMS (EI): $m/z$ calculated for C$_{10}$H$_{10}$O$_2$S$_2$+Na$: 249.0020$, found 249.0014.
7. References


8. NMR Spectra

$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 1c

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 1c
$^{19}$F NMR (376 MHz, CDCl$_3$) spectrum of product 1c

$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 1e
\( ^{13}\text{C} \text{ NMR (100 MHz, CDCl}_3 \text{) spectrum of product 1e} \)

\( ^{19}\text{F} \text{ NMR (376 MHz, CDCl}_3 \text{) spectrum of product 1e} \)
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 1f

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 1f
\(^{19}\text{F} \text{NMR (376 MHz, CDCl}_3\text{)} \text{ spectrum of product 1f}\)

![\(^{19}\text{F} \text{NMR (376 MHz, CDCl}_3\text{)} \text{ spectrum of product 1f}\)](image)

\(^{1}\text{H} \text{NMR (400 MHz, CDCl}_3\text{)} \text{ spectrum of product 1g}\)

![\(^{1}\text{H} \text{NMR (400 MHz, CDCl}_3\text{)} \text{ spectrum of product 1g}\)](image)
$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 1g

$^{19}$F NMR (376 MHz, CDCl$_3$) spectrum of product 1g
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 1i

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 1i
$^{19}$F NMR (376 MHz, CDCl$_3$) spectrum of product 1i

$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 1k
$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 1k

$^{19}$F NMR (376 MHz, CDCl$_3$) spectrum of product 1k
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 1l

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 1l
$^{19}$F NMR (376 MHz, CDCl$_3$) spectrum of product 1I

$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 1m
$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 1m

![13C NMR spectrum of product 1m](image)

$^{19}$F NMR (376 MHz, CDCl$_3$) spectrum of product 1m

![19F NMR spectrum of product 1m](image)
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 1n

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 1n
$^{19}$F NMR (376 MHz, CDCl$_3$) spectrum of product 1n
\(^{1}\text{H} \text{NMR} (400 \text{ MHz, CDCl}_3)\) spectrum of product 3a

\[^{13}\text{C} \text{NMR} (100 \text{ MHz, CDCl}_3)\) spectrum of product 3a
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 3b

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 3b
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 3c

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 3c
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 3d

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 3d
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 3e

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 3e
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 3f

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 3f
\(^1\)H NMR (400 MHz, CDCl\(_3\)) spectrum of product 3g

\(^{13}\)C NMR (100 MHz, CDCl\(_3\)) spectrum of product 3g
$^1$H NMR (400 MHz, CDCl₃) spectrum of product 3h

$^{13}$C NMR (100 MHz, CDCl₃) spectrum of product 3h
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 3i

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 3i
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 3j

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 3j
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 3k

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 3k
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 3l

13C NMR (100 MHz, CDCl$_3$) spectrum of product 3l
\(^1\)H NMR (400 MHz, CDCl\(_3\)) spectrum of product 3m

\(^{13}\)C NMR (100 MHz, CDCl\(_3\)) spectrum of product 3m
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 3n

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 3n
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 3o

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 3o
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 3p

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 3p
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 3q

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 3q
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 3r

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 3r
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 3s

![NMR Spectrum](image)
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 4a

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 4a
$^{1}H$ NMR (400 MHz, CDCl$_3$) spectrum of product 4a

$^{13}C$ NMR (100 MHz, CDCl$_3$) spectrum of product 4b
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 4c

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 4c
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 4d

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 4d
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 4e

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 4e
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 4f

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 4f
$^{1}H$ NMR (400 MHz, CDCl$_3$) spectrum of product 4g

$^{13}C$ NMR (100 MHz, CDCl$_3$) spectrum of product 4g
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 4h

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 4h
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 4i

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 4i
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 4j

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 4j
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 4k

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 4k
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 4l

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 4l
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 5

$^{13}$C NMR (100 MHz, CDCl$_3$) spectrum of product 6
$^1$H NMR (400 MHz, CDCl$_3$) spectrum of product 6
# 9. HPLC Traces

![HPLC Trace](image1)

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![HPLC Trace](image2)

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