Electronic Supporting Information for

Acetic Acid-Promoted Cascade N-Acyliminium ion/aza-Prins Cyclization: Stereoselective Synthesis of Functionalized Fused Tricyclic Piperidines

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General Information

All reagents were purchased at the highest commercial quality and used without further purification. Yields refer to isolated, homogenous and spectroscopically pure material, unless otherwise stated. Crude reaction mixtures were purified by silica gel chromatography (E. Merck silica gel, particle size 0.043–0.063 mm) and thin layer chromatography was carried out using E. Merck silica plates (60F-254) with UV light (254 nm) as the visualization agent. Microwave reactions were carried out in an Initiator single-mode reactor producing controlled radiation at 2450 MHz, and temperature was monitored via the built-in online IR sensor. $^1$H NMR spectra were recorded at 400 MHz and $^{13}$C{$^1$H} NMR spectra at 100 MHz. The chemical shifts for $^1$H NMR and $^{13}$C{$^1$H} NMR spectra were referenced to tetramethylsilane via residual solvent signals ($^1$H, CDCl$_3$ at 7.26 ppm, CD$_3$OD at 3.31 ppm; $^{13}$C, CDCl$_3$ at 77.16 ppm, CD$_3$OD at 49.0 ppm). LC/MS was performed on an instrument equipped with a C18 column (50 × 3.0 mm, particle size 2.6 μm, pore size 100 Å). Accurate mass values were determined using a mass spectrometer equipped with an electrospray ion source and time-of-flight detector. Electrophilic precursors 1a–1m were prepared following the literature procedure.$^{[1,2]}$

General procedure for cascade aza-Prins cyclisation, exemplified by the preparation of (±)-6-Oxo-5,8,9,10,11,11a-hexahydro-6H-pyrido[1,2-c]quinazolin-10-yl acetate (3a)

![Chemical Structure](image)

Chemical Formula: C$_{14}$H$_{18}$N$_2$O$_3$
Exact Mass: 260.1161

A sealed 0.5–2 mL Pyrex process vial charged with aldehyde 1a (40 mg, 223 μmol), amine 2a (20 mg, 281 μmol) and acetic acid (1 mL) was subjected to microwave irradiation at 140 °C for 20 min. The reaction mixture was cooled to room temperature, diluted with 2 mL ethyl acetate. The volatiles were concentrated in vacuo and the residue was purified by silica gel chromatography (55% EtOAc in n-pentane) to yield the title compound as a white solid (49 mg, 189 μmol, 86%, 99/1 cis/trans ratio).

$^1$H NMR (400 MHz; CDCl$_3$) δ 1.58–1.69 (m, 1H), 1.99–2.03 (m, 1H), 2.04 (s, 4H), 2.27 (ddt, J = 12.4, 4.6, 2.3, 2.3 Hz, 1H), 2.73 (ddd, J = 13.8, 12.9, 2.6 Hz, 1H), 4.55 (dd, J = 12.0, 2.6 Hz, 1H), 4.69 (ddd, J = 13.8, 4.6, 2.4 Hz, 1H), 4.90–5.26 (m, 1H), 6.61–6.76 (m, 1H), 6.91–6.98 (m, 1H), 7.00–7.07 (m, 1H), 7.11–7.20 (m, 1H), 7.88 (s, 1H).
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$^{13}$C NMR (100 MHz; CDCl$_3$) $\delta$ 21.4, 30.6, 39.7, 41.8, 56.6, 71.1, 114.0, 120.0, 122.3, 125.6, 128.7, 135.7, 152.8, 170.5.

HRMS (ESI) calc’d for C$_{16}$H$_{20}$N$_3$O$_3$ m/z 302.1505, found m/z 302.1508 (MeCN + H$^+$ adduct).

(±)-6-Oxo-5,8,9,10,11,11a-hexahydro-6H-pyrido[1,2-c]quinazolin-10-yl)formate (3b)

Chemical Formula: C$_{13}$H$_{14}$N$_2$O$_3$
Exact Mass: 246.1004

Prepared following the general procedure, using formic acid (1 mL) instead of AcOH. Starting from aldehyde 1a (40 mg, 223 µmol) and amine 2a (20 mg, 281 µmol), the title compound was obtained after silica gel chromatography (55% EtOAc in n-pentane) as a white solid (20 mg, 83 µmol, 36%, 99/1 cis/trans ratio).

$^1$H NMR (400 MHz; CDCl$_3$) $\delta$ 1.63–1.74 (m, 1H), 2.00 – 2.10 (m, 1H), 2.21–2.35 (m, 1H), 2.74 (td, $J$ = 13.4, 13.4, 2.6 Hz, 1H), 4.56 (dd, $J$ = 11.9, 2.6 Hz, 1H), 4.72 (ddd, $J$ = 13.9, 4.7, 2.4 Hz, 1H), 5.12–5.22 (m, 1H), 6.72 (dd, $J$ = 8.0, 1.1 Hz, 1H), 6.88–7.00 (m, 1H), 7.00–7.07 (m, 1H), 7.11–7.23 (m, 1H), 8.03 (s, 1H), 8.27 (s, 1H).

$^{13}$C NMR (100 MHz; CDCl$_3$) $\delta$ 30.5, 39.5, 41.7, 56.5, 70.9, 114.2, 119.7, 122.3, 125.5, 128.8, 135.8, 153.0, 160.4.

HRMS (ESI) calc’d for C$_{13}$H$_{15}$N$_2$O$_3$ m/z 247.1083, found m/z 247.1092.

(±)-6-Oxo-5,8,9,10,11,11a-hexahydro-6H-pyrido[1,2-c]quinazolin-10-yl propionate (3c)

Chemical Formula: C$_{15}$H$_{18}$N$_2$O$_3$
Exact Mass: 274.1317

S5
Prepared following the general procedure, using propionic acid (1 mL) instead of AcOH. Starting from aldehyde 1a (40 mg, 223 µmol) and amine 2a (21 mg, 295 µmol), the title compound was obtained after silica gel chromatography (30–55% EtOAc in n-pentane) as a white solid (31 mg, 113 µmol, 51%, 97/3 cis/trans ratio).

\[^1\text{H} \text{NMR} (400 \text{ MHz}; \text{CDCl}_3) \delta 1.12 (t, J = 7.6, 7.6 \text{ Hz}, 3\text{H}), 1.55–1.67 (m, 1\text{H}), 1.67–1.75 (m, 1\text{H}), 1.97–2.07 (m, 1\text{H}), 2.23–2.29 (m, 2\text{H}), 2.29–2.34 (m, 1\text{H}), 2.64–2.80 (m, 1\text{H}), 4.54 (dd, J = 12.0, 2.6 \text{ Hz}, 1\text{H}), 4.69 (ddd, J = 13.8, 4.7, 2.4 \text{ Hz}, 1\text{H}), 5.03 (ddd, J = 11.3, 6.7, 4.6 \text{ Hz}, 1\text{H}), 6.70–6.77 (m, 1\text{H}), 6.89–6.95 (m, 1\text{H}), 6.99–7.04 (m, 1\text{H}), 7.13–7.19 (m, 1\text{H}), 8.71 (s, 1\text{H}).

\[^{13}\text{C} \text{NMR} (100 \text{ MHz}; \text{CDCl}_3) \delta 27.8, 30.5, 39.6, 41.6, 56.4, 70.7, 114.0, 119.8, 122.0, 125.3, 128.5, 135.8, 153.1, 173.8.

HRMS (ESI) calc’d for C\(_{17}\)H\(_{22}\)N\(_3\)O\(_3\) m/z 316.1661, found m/z 316.1675 (MeCN + H\(^+\) adduct).

(±)-2-Methoxy-6-oxo-5,8,9,10,11,11a-hexahydro-6H-pyrido[1,2-c]quinazolin-10-yl acetate (4)

Chemical Formula: C\(_{17}\)H\(_{22}\)N\(_3\)O\(_3\)
Exact Mass: 290.1267

Prepared following the general procedure, starting from aldehyde 1b (40 mg, 191 µmol) and amine 2a (21 mg, 295 µmol), the title compound was obtained after silica gel chromatography (70% EtOAc in n-pentane) as a white solid (48 mg, 164 µmol, 86%, 96/4 cis/trans ratio).

\[^1\text{H} \text{NMR} (400 \text{ MHz}; \text{CDCl}_3) \delta 1.59–1.76 (m, 2\text{H}), 1.97–2.04 (m, 1\text{H}), 2.04 (s, 3\text{H}), 2.23–2.34 (m, 1\text{H}), 2.71 (ddd, J = 13.8, 12.9, 2.6 \text{ Hz}, 1\text{H}), 3.75 (s, 3\text{H}), 4.50 (dd, J = 12.0, 2.5 \text{ Hz}, 1\text{H}), 4.68 (ddd, J = 13.8, 4.6, 2.4 \text{ Hz}, 1\text{H}), 5.01 (tt, J = 11.3, 11.3, 4.6, 4.6 \text{ Hz}, 1\text{H}), 6.58 (d, J = 2.7 \text{ Hz}, 1\text{H}), 6.63 (d, J = 8.6 \text{ Hz}, 1\text{H}), 6.73 (dd, J = 8.6, 2.7 \text{ Hz}, 1\text{H}), 7.80 (s, 1\text{H}).

\[^{13}\text{C} \text{NMR} (100 \text{ MHz}; \text{CDCl}_3) \delta 21.4, 30.5, 39.4, 41.8, 55.8, 56.8, 71.1, 110.9, 114.4, 115.0, 120.9, 129.3, 152.9, 155.2, 170.5.

HRMS (ESI) calc’d for C\(_{15}\)H\(_{19}\)N\(_2\)O\(_4\) m/z 291.1345, found m/z 291.1343.
(±)-2-Bromo-6-oxo-5,8,9,10,11,11a-hexahydro-6H-pyrido[1,2-c]quinazolin-10-yl acetate (5)

Prepared following the general procedure, starting from aldehyde 1c (40 mg, 155 µmol) and amine 2a (21 mg, 295 µmol), the title compound was obtained after silica gel chromatography (45% EtOAc in n-pentane) as a white solid (45 mg, 132 µmol, 85%, 95/5 cis/trans ratio).

1H NMR (400 MHz; CDCl3) δ 1.56–1.78 (m, 2H), 2.05 (s, 3H), 2.27 (ddt, J = 12.3, 4.6, 2.3, 2.3 Hz, 1H), 2.72 (ddd, J = 13.8, 13.0, 2.6 Hz, 1H), 4.52 (dd, J = 12.3, 2.6 Hz, 1H), 4.67 (ddd, J = 13.8, 4.6, 2.4 Hz, 1H), 5.00 (tt, J = 11.3, 11.3, 4.6, 4.6 Hz, 1H), 6.61 (d, J = 8.5 Hz, 1H), 7.16 (d, J = 2.1 Hz, 1H), 7.24–7.28 (m, 1H), 8.33 (s, 1H).

13C NMR (100 MHz; CDCl3) δ 21.3, 30.5, 39.6, 41.8, 56.1, 70.8, 114.3, 115.7, 121.9, 128.5, 131.6, 135.0, 152.6, 170.5.

HRMS (ESI) calc’d for C16H15BrN3O3 m/z 380.0160, found m/z 380.0628 (MeCN + H+) adduct.

(±)-2-Fluoro-6-oxo-5,8,9,10,11,11a-hexahydro-6H-pyrido[1,2-c]quinazolin-10-yl acetate (6)

Prepared following the general procedure, starting from aldehyde 1d (40 mg, 203 µmol) and amine 2a (21 mg, 295 µmol), the title compound was obtained after silica gel chromatography (53% EtOAc in n-pentane) as a white solid (46 mg, 165 µmol, 81%, 96/4 cis/trans ratio).

1H NMR (400 MHz; CDCl3) δ 1.57–1.78 (m, 2H), 2.00–2.04 (m, 1H), 2.05 (s, 3H), 2.26 (ddt, J = 12.4, 4.6, 2.3, 2.3 Hz, 1H), 2.72 (ddd, J = 13.8, 12.9, 2.6 Hz, 1H), 4.52 (dd, J = 11.9, 2.6
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$^1$H NMR (400 MHz; CDCl$_3$) $\delta$ 1.56–1.78 (m, 3H), 2.05 (s, 3H), 2.21–2.31 (m, 1H), 2.68–2.80 (m, 1H), 4.52 (dd, $J = 11.9$, 2.6 Hz, 1H), 4.69 (ddd, $J = 13.7$, 4.7, 2.4 Hz, 1H), 4.96–5.06 (m, 1H), 6.73 (d, $J = 1.9$ Hz, 1H), 6.90 (dd, $J = 8.2$, 1.9 Hz, 1H), 6.95 (d, $J = 8.2$ Hz, 1H), 8.36 (s, 1H).

$^{13}$C NMR (100 MHz; CDCl$_3$) $\delta$ 21.2, 30.4, 39.5, 41.7, 56.0, 70.7, 113.8, 118.3, 122.2, 126.6, 134.1, 136.8, 152.3, 170.3.

HRMS (ESI) calc’d for C$_{16}$H$_{19}$ClN$_3$O$_3$ m/z 336.1115, found m/z 336.1128 (MeCN + H$^+$ adduct).

(±)-3-Chloro-6-oxo-5,8,9,10,11,11a-hexahydro-6H-pyrido[1,2-c]quinazolin-10-yl acetate (7)

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\begin{array}{c}
\text{O} \\
\text{O} \\
\text{N} \\
\text{N}
\end{array}
\]

Chemical Formula: C$_{14}$H$_{15}$ClN$_2$O$_3$

Exact Mass: 294.0771

Prepared following the general procedure, starting from aldehyde 1e (40 mg, 203 µmol) and amine 2a (21 mg, 295 µmol), the title compound was obtained after silica gel chromatography (45% EtOAc in n-pentane) as a white solid (45 mg, 155 µmol, 83%, 94/6 cis/trans ratio).

$^1$H NMR (400 MHz; CDCl$_3$) $\delta$ 1.56–1.78 (m, 3H), 2.05 (s, 3H), 2.21–2.31 (m, 1H), 2.68–2.80 (m, 1H), 4.52 (dd, $J = 11.9$, 2.6 Hz, 1H), 4.69 (ddd, $J = 13.7$, 4.7, 2.4 Hz, 1H), 4.96–5.06 (m, 1H), 6.73 (d, $J = 1.9$ Hz, 1H), 6.90 (dd, $J = 8.2$, 1.9 Hz, 1H), 6.95 (d, $J = 8.2$ Hz, 1H), 8.36 (s, 1H).

$^{13}$C NMR (100 MHz; CDCl$_3$) $\delta$ 21.2, 30.4, 39.5, 41.7, 56.0, 70.7, 113.8, 118.3, 122.2, 126.6, 134.1, 136.8, 152.3, 170.3.

HRMS (ESI) calc’d for C$_{16}$H$_{19}$ClN$_3$O$_3$ m/z 336.1115, found m/z 336.1128 (MeCN + H$^+$ adduct).
(±)-6-Oxo-3-(trifluoromethyl)-5,8,9,10,11,11a-hexahydro-6H-pyrido[1,2-c]quinazolin-10-yl acetate (8)

![Chemical structure of (±)-6-Oxo-3-(trifluoromethyl)-5,8,9,10,11,11a-hexahydro-6H-pyrido[1,2-c]quinazolin-10-yl acetate (8)](image)

Chemical Formula: C17H17F3N3O3
Exact Mass: 328.1035

Prepared following the general procedure, starting from aldehyde 1f (40 mg, 161 µmol) and amine 2a (21 mg, 295 µmol), the title compound was obtained after silica gel chromatography (45% EtOAc in n-pentane) as a white solid (45 mg, 136 µmol, 84%, 93/7 cis/trans ratio).

1H NMR (400 MHz; CDCl3) δ 1.61–1.80 (m, 3H), 2.05 (s, 3H), 2.25–2.35 (m, 1H), 2.68–2.84 (m, 1H), 4.60 (dd, J = 12.1, 2.5 Hz, 1H), 4.71 (ddd, J = 13.9, 4.6, 2.4 Hz, 1H), 5.04 (tt, J = 11.3, 11.3, 4.6, 4.6 Hz, 1H), 6.95–6.98 (m, 1H), 7.12–7.16 (m, 1H), 7.17–7.21 (m, 1H), 8.75 (s, 1H).

13C NMR (100 MHz; CDCl3) δ 21.3, 30.5, 39.5, 41.9, 56.4, 70.8, 111.0 (q, 3J CF = 3.7 Hz), 118.9 (q, 3J CF = 3.9 Hz), 123.4 (q, 4J CF = 1.5 Hz), 123.7 (q, 1J CF = 270.2 Hz), 126.2, 131.3 (q, 2J CF = 32.5 Hz), 136.5, 152.7, 170.5.

HRMS (ESI) calc’d for C17H17F3N3O3 m/z 370.1379, found m/z 370.1378.

(±)-4-Methoxy-6-oxo-5,8,9,10,11,11a-hexahydro-6H-pyrido[1,2-c]quinazolin-10-yl acetate (9)

![Chemical structure of (±)-4-Methoxy-6-oxo-5,8,9,10,11,11a-hexahydro-6H-pyrido[1,2-c]quinazolin-10-yl acetate (9)](image)

Chemical Formula: C15H18N2O4
Exact Mass: 290.1267

Prepared following the general procedure, starting from aldehyde 1g (40 mg, 191 µmol) and amine 2a (21 mg, 295 µmol), the title compound was obtained after silica gel chromatography (60% EtOAc in n-pentane) as a white solid (38 mg, 130 µmol, 68%, 97/3 cis/trans ratio). A single crystal was prepared for X-ray diffraction studies by recrystallization from THF/pentane. CCDC 1519982 contains the supplementary crystallographic data for this
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ESI

paper. The data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/structures.

$^1$H NMR (400 MHz; CDCl$_3$) $\delta$ 1.57–1.76 (m, 2H), 1.98–2.04 (m, 1H), 2.04 (s, 3H), 2.22–2.32 (m, 1H), 2.71 (ddd, $J$ = 13.8, 12.9, 2.6 Hz, 1H), 3.85 (s, 3H), 4.55 (ddd, $J$ = 12.0, 2.5 Hz, 1H), 4.68 (ddd, $J$ = 13.8, 4.7, 2.4 Hz, 1H), 5.00 (tt, $J$ = 11.3, 11.3, 4.6, 4.6 Hz, 1H), 6.63–6.67 (m, 1H), 6.71–6.78 (m, 1H), 6.87–6.92 (m, 1H), 6.93–6.98 (m, 1H).

$^{13}$C NMR (100 MHz; CDCl$_3$) $\delta$ 21.4, 30.6, 39.5, 41.8, 55.9, 56.7, 71.1, 109.5, 117.3, 120.2, 122.0, 125.3, 145.0, 151.9, 170.5.

HRMS (ESI) calc’d for C$_{15}$H$_{19}$N$_2$O$_4$ m/z 291.1345, found m/z 291.1359.

(±)-4-Methyl-6-oxo-5,8,9,10,11,11a-hexahydro-6H-pyrido[1,2-c]quinazolin-10-yl acetate (10)

![Chemical Structure](image)

Chemical Formula: C$_{15}$H$_{18}$N$_2$O$_3$

Exact Mass: 274.1317

Prepared following the general procedure, starting from aldehyde 1h (40 mg, 207 µmol) and amine 2a (21 mg, 295 µmol), the title compound was obtained after silica gel chromatography (50% EtOAc in n-pentane) as a white solid (43 mg, 155 µmol, 75%, 97/3 cis/trans ratio).

$^1$H NMR (400 MHz; CDCl$_3$) $\delta$ 1.58–1.78 (m, 2H), 1.98–2.02 (m, 1H), 2.04 (s, 3H), 2.19 (s, 3H), 2.21–2.29 (m, 1H), 2.66–2.80 (m, 1H), 4.53 (dd, $J$ = 12.0, 2.6 Hz, 1H), 4.66 (ddd, $J$ = 13.8, 4.7, 2.4 Hz, 1H), 5.02 (ddd, $J$ = 11.3, 6.7, 4.6 Hz, 1H), 6.76–6.92 (m, 3H), 6.98–7.06 (m, 1H).

$^{13}$C NMR (100 MHz; CDCl$_3$) $\delta$ 16.6, 21.2, 30.4, 39.6, 41.7, 56.5, 70.9, 119.6, 121.2, 121.9, 123.3, 129.8, 133.7, 152.3, 170.4.

HRMS (ESI) calc’d for C$_{17}$H$_{22}$N$_3$O$_3$ m/z 316.1661, found m/z 316.1675 (MeCN + H$^+$ adduct).
(±)-6-Oxo-5,8,9,10,11,11a-hexahydro-6H-dipyrdo[1,2-c:3',2'-e]pyrimidin-10-yl acetate (11)

Prepared following the general procedure, starting from aldehyde 1i (40 mg, 222 µmol) and amine 2a (21 mg, 295 µmol), the title compound was obtained after silica gel chromatography (50% EtOAc in n-pentane) as a white solid (36 mg, 137 µmol, 62%, 92/8 cis/trans ratio).

\[ \begin{array}{c}
\text{Chemical Formula: } C_{13}H_{16}N_{3}O_{3} \\
\text{Exact Mass: } 261.1113
\end{array} \]

\[ \begin{array}{c}
1^1H \text{ NMR (400 MHz; CDCl}_3\delta 1.57–1.77 (m, 2H), 2.00–2.03 (m, 1H), 2.05 (s, 3H), 2.27 (ddt, } J = 12.3, 4.6, 2.4, 2.4 \text{ Hz, 1H), 2.73 (ddd, } J = 13.9, 13.0, 2.6 \text{ Hz, 1H), 4.56 (dd, } J = 12.0, 2.7 \text{ Hz, 1H), 4.70 (ddd, } J = 14.0, 4.7, 2.4 \text{ Hz, 1H), 5.01 (tt, } J = 11.3, 11.3, 4.6, 4.6 \text{ Hz, 1H), 6.91 (dd, } J = 7.5, 5.0 \text{ Hz, 1H), 7.37 (ddd, } J = 7.5, 1.7, 0.8 \text{ Hz, 1H), 8.23 (ddd, } J = 5.0, 1.7, 0.6 \text{ Hz, 1H), 8.81 (s, 1H).}
\end{array} \]

\[ \begin{array}{c}
13^1C \text{ NMR (100 MHz; CDCl}_3\delta 21.2, 30.4, 39.4, 41.4, 55.3, 70.6, 115.4, 117.9, 133.8, 147.8, 148.9, 152.0, 170.3.
\end{array} \]

HRMS (ESI) calc’d for C_{13}H_{16}N_{3}O_{3} m/z 262.1192, found m/z 262.1210.

(±)-5-Methyl-6-oxo-5,8,9,10,11,11a-hexahydro-6H-pyrido[1,2-c]quinazolin-10-yl acetate (12)

Prepared following the general procedure, starting from aldehyde 1j (40 mg, 207 µmol) and amine 2a (21 mg, 295 µmol), the title compound was obtained after silica gel chromatography (30% EtOAc in n-pentane) as a white solid (44 mg, 160 µmol, 77%, 98/2 cis/trans ratio).

\[ \begin{array}{c}
\text{Chemical Formula: } C_{15}H_{18}N_{3}O_{3} \\
\text{Exact Mass: } 274.1317
\end{array} \]

\[ \begin{array}{c}
1^1H \text{ NMR (400 MHz; CDCl}_3\delta 1.58–1.73 (m, 2H), 1.96–2.02 (m, 1H), 2.03 (s, 3H), 2.21 (ddt, } J = 12.3, 4.5, 2.4, 2.4 \text{ Hz, 1H), 2.74 (ddd, } J = 13.8, 12.9, 2.6 \text{ Hz, 1H), 3.33 (s, 3H), 4.46 (dd, } J
\end{array} \]
Cascade N-Acyliminium Ion/aza-Prins Cyclization

\[ \text{ESI} \]

\[ \delta = 12.0, 2.6 \text{ Hz, 1H}, 4.64 (\text{ddd, } J = 13.8, 4.6, 2.4 \text{ Hz, 1H}), 5.02 (\text{ddd, } J = 11.4, 6.7, 4.7 \text{ Hz, 1H}), 6.83 (\text{dd, } J = 8.2, 1.0 \text{ Hz, 1H}), 6.98 (\text{td, } J = 7.4, 7.4, 1.1 \text{ Hz, 1H}), 7.05 (\text{ddt, } J = 7.5, 1.7, 0.6, 0.6 \text{ Hz, 1H}), 7.22–7.32 (m, 1H). \]

\[^{13}\text{C NMR (100 MHz; CDCl}_3\text{) } \delta 21.2, 30.0, 30.2, 39.2, 43.1, 55.8, 71.0, 112.9, 121.7, 122.0, 125.5, 128.5, 138.1, 153.3, 170.3. \]

HRMS (ESI) calc’d for C\(_{15}\)H\(_{19}\)N\(_2\)O\(_3\) m/z 275.1396, found m/z 275.1400.

(±)-5-Allyl-6-oxo-5,8,9,10,11,11a-hexahydro-6H-pyrido[1,2-c]quinazolin-10-yl acetate (13)

\[
\begin{align*}
\text{Chemical Formula: } & C_{17}H_{20}N_2O_3 \\
\text{Exact Mass: } & 300.1474
\end{align*}
\]

Prepared following the general procedure, starting from aldehyde 1k (40 mg, 182 µmol) and amine 2a (21 mg, 295 µmol), the title compound was obtained after silica gel chromatography (32% EtOAc in n-pentane) as a white solid (45 mg, 150 µmol, 82%, 93/7 cis/trans ratio).

\[^{1}\text{H NMR (400 MHz; CDCl}_3\text{) } \delta 1.58–1.75 (m, 2H), 1.96–2.00 (m, 1H), 2.03 (s, 3H), 2.18–2.29 (m, 1H), 2.74 (\text{ddd, } J = 13.8, 13.0, 2.6 \text{ Hz, 1H}), 4.35–4.51 (m, 2H), 4.54–4.74 (m, 2H), 5.02 (\text{ddd, } J = 11.4, 6.7, 4.7 \text{ Hz, 1H}), 5.13–5.22 (m, 2H), 5.8–5.97 (m, 1H), 6.75–6.84 (m, 1H), 6.93–7.02 (m, 1H), 7.03–7.09 (m, 1H), 7.17–7.24 (m, 1H). \]

\[^{13}\text{C NMR (100 MHz; CDCl}_3\text{) } \delta 21.3, 30.3, 39.5, 43.2, 45.3, 56.0, 71.1, 113.9, 116.2, 121.8, 122.1, 125.7, 128.5, 133.1, 137.3, 153.0, 170.5. \]

HRMS (ESI) calc’d for C\(_{17}\)H\(_{21}\)N\(_2\)O\(_3\) m/z 301.1552, found m/z 301.1549.
(±)-5-Benzyl-6-oxo-5,8,9,10,11,11a-hexahydro-6H-pyrido[1,2-c]quinazolin-10-yl acetate (14)

Prepared following the general procedure, starting from aldehyde 1 (40 mg, 182 µmol) and amine 2a (21 mg, 295 µmol), the title compound was obtained after silica gel chromatography (35% EtOAc in n-pentane) as a white liquid (43 mg, 122 µmol, 82%, 93/7 cis/trans ratio).

^1^H NMR (400 MHz; CDCl\textsubscript{3}) δ 1.64–1.81 (m, 2H), 2.00–2.04 (m, 1H), 2.06 (s, 3H), 2.23–2.32 (m, 1H), 2.65–3.38 (m, 1H), 4.54 (dd, J = 12.0, 2.6 Hz, 1H), 4.71 (ddd, J = 13.8, 4.6, 2.4 Hz, 1H), 4.99–5.14 (m, 2H), 5.17–5.28 (m, 1H), 6.70 (dd, J = 8.2, 1.0 Hz, 1H), 6.94 (td, J = 7.5, 7.5, 1.0 Hz, 1H), 7.02–7.14 (m, 2H), 7.16–7.28 (m, 3H), 7.27–7.38 (m, 2H).

^1^C NMR (100 MHz; CDCl\textsubscript{3}) δ 21.7, 30.8, 40.1, 43.7, 47.0, 56.5, 71.5, 114.5, 122.1, 122.6, 126.1, 126.7, 127.4, 128.9, 129.1, 137.6, 138.0, 153.8, 170.8.

HRMS (ESI) calc’d for C\textsubscript{21}H\textsubscript{23}N\textsubscript{2}O\textsubscript{3} m/z 351.1709, found m/z 351.1700.

(±)-Ethyl-2-(10-acetoxy-6-oxo-9,10,11,11a-tetrahydro-6H-pyrido[1,2-c]quinazolin-5(8H)-yl)acetate (15)

Prepared following the general procedure, starting from aldehyde 1m (40 mg, 150 µmol) and amine 2a (21 mg, 295 µmol), the title compound was obtained after silica gel chromatography (35% EtOAc in n-pentane) as a yellow liquid (38 mg, 109 µmol, 72%, 96/4 cis/trans ratio).
Cascade N-Acyliminium Ion/aza-Prins Cyclization

ESI

$^1$H NMR (400 MHz; CDCl$_3$) δ 1.26 (t, $J = 7.1$, 7.1 Hz, 3H), 1.60–1.81 (m, 2H), 1.96–2.02 (m, 1H), 2.03 (s, 3H), 2.23 (ddt, $J = 12.4$, 2.2, 2.2 Hz, 1H), 2.77 (ddd, $J = 13.8$, 13.0, 2.6 Hz, 1H), 4.14–4.28 (m, 2H), 4.49 (dd, $J = 12.0$, 2.6 Hz, 1H), 4.57–4.68 (m, 1H), 4.59–4.76 (m, 2H), 5.03 (ddd, $J = 11.3$, 6.7, 4.7 Hz, 1H), 6.61 (dd, $J = 8.2$, 1.0 Hz, 1H), 6.99 (td, $J = 7.5$, 7.4, 1.0 Hz, 1H), 7.07 (dd, $J = 7.6$, 1.6 Hz, 1H), 7.21 (ddd, $J = 8.5$, 7.3, 1.6 Hz, 1H).

NMR (100 MHz; CDCl$_3$) δ 14.2, 21.2, 30.2, 39.3, 43.2, 44.4, 56.0, 61.4, 70.9, 112.6, 121.8, 122.4, 125.9, 128.6, 137.0, 152.9, 169.2, 170.4.

HRMS (ESI) calc’d for C$_{18}$H$_{23}$N$_2$O$_5$ m/z 347.1607, found m/z 347.1606.

(±)-6-Oxo-8-phenyl-5,8,9,10,11,11a-hexahydro-6H-pyrido[1,2-c]quinazolin-10-yl acetate (16)

 Prepared following the general procedure, starting from aldehyde 1a (40 mg, 150 µmol) and amine 2b (150 mg, 1 mmol, prepared following the literature procedure$^{[3]}$), the title compound was obtained after silica gel chromatography (25% EtOAc in n-pentane) as a colorless liquid (40 mg, 119 µmol, 55%, 98/2 cis/trans ratio).

$^1$H NMR (400 MHz; CDCl$_3$) δ 1.85 (td, $J = 12.2$, 12.2, 11.2 Hz, 1H), 1.94–2.05 (m, 1H), 2.07 (s, 3H), 2.14 (ddt, $J = 12.4$, 4.8, 2.3, 2.3 Hz, 1H), 2.83 (ddt, $J = 13.4$, 4.2, 2.0, 2.0 Hz, 1H), 4.47 (dd, $J = 12.2$, 2.6 Hz, 1H), 5.13–5.26 (m, 1H), 6.20 (d, $J = 5.4$ Hz, 1H), 6.68–6.79 (m, 1H), 6.91 (dd, $J = 4.2$, 0.8 Hz, 2H), 7.09–7.22 (m, 1H), 7.26–7.33 (m, 1H), 7.38 (dd, $J = 8.5$, 6.9 Hz, 2H), 7.47 (dt, $J = 8.3$, 1.2, 1.2 Hz, 2H), 8.18 (s, 1H).

$^{13}$C NMR (100 MHz; CDCl$_3$) δ 21.3, 31.3, 40.0, 51.7, 52.1, 67.9, 113.9, 119.8, 122.2, 125.9, 126.7, 127.2, 128.5, 129.0, 135.4, 137.7, 153.4, 170.5.

HRMS (ESI) calc’d for C$_{20}$H$_{21}$N$_2$O$_3$ m/z 337.1552, found m/z 337.1559.

S14
To a stirred solution of 3a (30 mg, 115 µmol) in methanol (1 mL) at ambient temperature was added K$_2$CO$_3$ (34 mg, 246 µmol). The resulting solution was stirred for 17 h, after which it was partitioned between brine (10 mL) and EtOAc (10 mL). The aqueous phase was extracted into EtOAc (2x20 mL), and the combined organics were dried over MgSO$_4$ and concentrated in vacuo. Silica gel chromatography (6% MeOH in DCM) provided the title product as a white solid (22 mg, 100 µmol, 88%). This transformation can also be carried out using NaOH, resulting in a slightly altered diastereomeric cis/trans ratio (87:13 vs. 89:11 using K$_2$CO$_3$).

$^1$H NMR (400 MHz; CDCl$_3$) δ 1.23–1.61 (m, 2H), 1.87–2.03 (m, 1H), 2.12 (dd, J = 12.4, 4.5, 2.3 Hz, 1H), 2.70 (td, J = 13.4, 13.3, 2.6 Hz, 1H), 3.85 (tt, J = 11.0, 11.0, 4.5, 4.5 Hz, 1H), 4.45 (ddd, J = 13.7, 4.5, 2.4 Hz, 1H), 4.52 (dd, J = 11.9, 2.5 Hz, 1H), 6.72 (dd, J = 8.0, 1.1 Hz, 1H), 6.92 (td, J = 7.5, 7.5, 1.2 Hz, 1H), 7.06–7.16 (m, 2H).

$^{13}$C NMR (100 MHz; CDCl$_3$) δ 33.7, 41.5, 43.0, 56.5, 68.1, 113.4, 120.6, 121.9, 125.2, 127.9, 135.6, 153.5.

HRMS (ESI) calc’d for C$_{12}$H$_{15}$N$_2$O$_2$ m/z 219.1134, found m/z 219.1132.

(±)-10-Hydroxy-5,8,9,10,11,11a-hexahydro-6H-pyrido[1,2-c]quinazolin-6-one (17)

To a stirred suspension of 17 (50 mg, 229 µmol) in DCM (5 mL) at ambient temperature was added DMP (97 mg, 229 µmol). The resulting mixture was stirred at ambient temperature overnight, after which sat. NaHCO$_3$ (10 mL) and Na$_2$S$_2$O$_3$ (26 mg) was added. The resulting aqueous phase was extracted into diethyl ether (2x40 mL), after which the combined organics were dried over Na$_2$SO$_4$ and concentrated in vacuo. The residue was purified by silica gel chromatography (30% EtOAc in hexanes) to yield the title compound as a white solid (35 mg, 164 µmol, 72%). Spectral data were in agreement with literature values. [2]
To a stirred solution of 3a (30 mg, 115 µmol) in DMF (1 mL) at ambient temperature was added NaH (5 mg, 60% dispersion in paraffin) and EtI (20 mg, 128 µmol). The resulting mixture was stirred for 40 min, after which it was partitioned between brine (10 mL) and EtOAc (10 mL). The aqueous phase was extracted into EtOAc (2x20 mL), and the combined organics were dried over MgSO₄ and concentrated in vacuo. Silica gel chromatography (30% EtOAc in n-pentane) provided the title product as a yellow solid (25 mg, 87 µmol, 75%, 94/6 cis/trans ratio).

$^1$H NMR (400 MHz; CDCl₃) δ 1.17 (t, J = 7.1 Hz, 3H), 1.42–1.68 (m, 2H), 1.91 (ddd, J = 12.7, 4.8, 2.3 Hz, 1H), 1.96 (s, 3H), 2.12 (ddt, J = 12.4, 4.6, 2.3, 2.3 Hz, 1H), 2.65 (ddd, J = 13.8, 12.9, 2.6 Hz, 1H), 3.80 (dq, J = 14.5, 7.1, 7.1, 7.1 Hz, 1H), 3.94 (dd, J = 14.5, 7.2 Hz, 1H), 4.37 (dd, J = 12.0, 2.6 Hz, 1H), 4.56 (ddd, J = 13.8, 4.6, 2.4 Hz, 1H), 4.94 (tt, J = 11.3, 11.3, 4.7, 4.7 Hz, 1H), 6.78 (dd, J = 8.3, 1.0 Hz, 1H), 6.89 (td, J = 7.5, 7.4, 1.1 Hz, 1H), 6.98 (dd, J = 7.5, 1.5 Hz, 1H), 7.14–7.18 (m, 1H).

$^{13}$C NMR (100 MHz; CDCl₃) δ 12.7, 21.3, 30.2, 37.7, 39.2, 43.0, 55.9, 71.1, 112.9, 121.8, 121.8, 125.8, 128.5, 136.9, 152.8, 170.4.

HRMS (ESI) calc’d for C₁₄H₂₆N₂O₂ m/z 258.1243, found m/z 258.1249 (MeCN + H⁺ adduct).

NOE experiments for the determination of relative stereochemistry of compounds 3a, 10 and 16

The relative stereochemistry of aza-Prins compounds were determined by NOESY, DPGSE-1D-NOESY, COSY, TOCSY and X-ray diffraction studies (for compound 10). For all compounds, a cis relationship exists between the benzylic hydrogen (Hₐ) and the hydrogen adjacent to the acetyl group (Hₐ). The introduction of a benzylic phenyl substituent did not have any effect on this relationship. Typically, NOE mixing times of 500–800 ms were used for selective irradiation experiments.
Cascade N-Acyliminium Ion/aza-Prins Cyclization

Literature references


Cascade N-Acyliminium Ion/aza-Prins Cyclization

$^{1}H$ and $^{13}C$ NMR spectra

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Chemical Formula: C$_{26}$H$_{26}$N$_{2}$O$_{2}$
Exact Mass: 260.1161

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Chemical Formula: C$_{26}$H$_{26}$N$_{2}$O$_{2}$
Exact Mass: 260.1161
Cascade N-Acyliminium Ion/aza-Prins Cyclization

Chemical Formula: C_{14}H_{18}N_{2}O_{3}
Exact Mass: 246.1004

Parameter | Value
--- | ---
1 Solvent | ccd3
2 Temperature | 25.0
3 Spectrometer Frequency | 399.86
4 Nucleus | 1H

Parameter | Value
--- | ---
1 Solvent | ccd3
2 Temperature | 25.0
3 Spectrometer Frequency | 100.55
4 Nucleus | 13C
Cascade N-Acyliminium Ion/aza-Prins Cyclization

Parameter | Value
---|---
1 Solvent | cdc3
2 Temperature | 25.0
3 Spectrometer Frequency | 399.86
4 Nucleus | 1H

Chemical Formula: C_{16}H_{19}N_{2}O_{3}
Exact Mass: 274.1317

Chemical Formula: C_{16}H_{19}N_{2}O_{3}
Exact Mass: 274.1317
Cascade N-Acyliminium Ion/aza-Prins Cyclization

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Chemical Formula: C_{14}H_{34}N_{14}O_{14}
Exact Mass: 290.1287
Cascade N-Acyliminium Ion/aza-Prins Cyclization

Parameter | Value
---|---
1 Solvent | cdCl3
2 Temperature | 25.0
3 Spectrometer Frequency | 399.94
4 Nucleus | 1H

Chemical Formula: C_{18}H_{16}BrN_2O_2
Exact Mass: 338.0306

Parameter | Value
---|---
1 Solvent | cdCl3
2 Temperature | 25.0
3 Spectrometer Frequency | 100.58
4 Nucleus | 13C

Chemical Formula: C_{18}H_{16}BrN_2O_2
Exact Mass: 338.0306
Cascade N-Acyliminium Ion/aza-Prins Cyclization

Parameter | Value
---|---
1 Solvent | cdc3
2 Temperature | 25.0
3 Spectrometer Frequency | 399.86
4 Nucleus | 1H

Chemical Formula: C_{14}H_{16}F2N2O2
Exact Mass: 278.1087

Parameter | Value
---|---
1 Solvent | cdc3
2 Temperature | 25.0
3 Spectrometer Frequency | 100.55
4 Nucleus | 13C

Chemical Formula: C_{14}H_{16}F2N2O2
Exact Mass: 278.1087
Cascade N-Acyliminium Ion/aza-Prins Cyclization

Parameter | Value
--- | ---
1 Solvent | ccd3
2 Temperature | 25.0
3 Spectrometer Frequency | 399.86
4 Nucleus | 1H

Chemical Formula: C<sub>17</sub>HOClN<sub>2</sub>O<sub>2</sub>
Exact Mass: 294.0771

Other diagram with similar information and chemical formula:

Chemical Formula: C<sub>17</sub>HOClN<sub>2</sub>O<sub>2</sub>
Exact Mass: 294.0771
Cascade N-Acyliminium Ion/aza-Prins Cyclization

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Chemical Formula: C_{12}H_{15}F_{3}N_{2}O_{3}
Exact Mass: 328.1035

ESI
Cascade N-Acyliminium Ion/aza-Prins Cyclization

Parameter | Value
--- | ---
1 Solvent | ccd3
2 Temperature | 25.0
3 Spectrometer Frequency | 399.86
4 Nucleus | 1H

Chemical Formula: C_{18}H_{24}N_{2}O_{4}
Exact Mass: 399.1307

Parameter | Value
--- | ---
1 Solvent | ccd3
2 Temperature | 25.0
3 Spectrometer Frequency | 100.55
4 Nucleus | 13C

Chemical Formula: C_{18}H_{24}N_{2}O_{4}
Exact Mass: 290.1267
Cascade N-Acyliminium Ion/aza-Prins Cyclization

Parameter | Value
--- | ---
1 Solvent | cdCl3
2 Temperature | 25.0
3 Spectrometer Frequency | 399.86
4 Nucleus | 1H

Chemical Formula: C_{14}H_{19}N_{2}O_{3}
Exact Mass: 274.1317

Parameter | Value
--- | ---
1 Solvent | cdCl3
2 Temperature | 25.0
3 Spectrometer Frequency | 100.55
4 Nucleus | 13C

Chemical Formula: C_{14}H_{19}N_{2}O_{3}
Exact Mass: 274.1317
Cascade N-Acyliminium I on/aza-Prins Cyclization

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Chemical Formula: C_{6}H_{12}N_{2}O_{3}
Exact Mass: 274.1317

±12

Chemical Formula: C_{6}H_{12}N_{2}O_{3}
Exact Mass: 274.1317

±12

S30
Cascade N-Acyliminium Ion/aza-Prins Cyclization

Chemical Formula: C₃₆H₄₂N₂O₃
Exact Mass: 500.1474

Parameter | Value
--- | ---
1 Solvent | cdCl₃
2 Temperature | 25.0
3 Spectrometer Frequency | 399.86
4 Nucleus | 1H

Chemical Formula: C₃₆H₄₂N₂O₃
Exact Mass: 500.1474

Parameter | Value
--- | ---
1 Solvent | cdCl₃
2 Temperature | 25.0
3 Spectrometer Frequency | 100.55
4 Nucleus | 13C
Cascade N-Acyliminium Ion/aza-Prins Cyclization

Parameter | Value
--- | ---
1 Solvent | ccd3
2 Temperature | 25.0
3 Spectrometer Frequency | 399.94 MHz
4 Nucleus | 1H

Chemical Formula: C_{29}H_{12}N_{2}O_{3}
Exact Mass: 336.1474

Parameter | Value
--- | ---
1 Solvent | ccd3
2 Temperature | 25.0
3 Spectrometer Frequency | 100.57 MHz
4 Nucleus | 13C

Chemical Formula: C_{29}H_{12}N_{2}O_{3}
Exact Mass: 336.1474
Cascade N-Acyliminium Ion/aza-Prins Cyclization

Chemical Formula: C$_7$H$_8$N$_2$O$_3$
Exact Mass: 216.0899

Parameter | Value
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1. Solvent | dioxane
2. Temperature | 25.0
3. Spectrometer frequency | 100.15
4. Nucleus | 13C
Cascade N-Acyliminium Ion/aza-Prins Cyclization

Parameter | Value
---|---
1 Solvent | CDCl₃
2 Temperature | 298.0
3 Spectrometer Frequency | 400.13
4 Nucleus | 1H

Chemical Formula: C₉H₈NO₃
Exact Mass: 280.1474

Parameter | Value
---|---
1 Solvent | CDCl₃
2 Temperature | 298.0
3 Spectrometer Frequency | 100.62
4 Nucleus | 13C

Chemical Formula: C₉H₈NO₃
Exact Mass: 280.1474