General Electrochemical Minisci Alkylation of \( N \)-Heteroarenes with Alkyl Halides

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1. General methods and starting materials

Starting materials 1a-h, 1k-1y, 2a-p as well as solvents for the reactions, were acquired from commercial sources (tetrahydrofuran was inhibitor free, water was tap water). Starting materials 1i, 1j and 10 were synthesized following a procedure described in the literature. For thin layer chromatography (TLC), silica gel plates with fluorescence indicator 254 nm were used and compounds were visualized by irradiation with UV light and/or by treatment with a solution of potassium permanganate in water followed by heating. Flash column chromatography was performed using Geduran® Silica Gel 60 (0.040-0.063 nm). Cyclohexane, ethyl acetate, dichloromethane and methanol for flash chromatography were acquired from commercial sources and were used without previous purification. NMR spectra were acquired on a Bruker Avance 300 MHz spectrometer, running at 300 and 75 MHz for $^1$H and $^{13}$C, respectively. $^{19}$F-NMR spectra were acquired on a Bruker Avance 500 MHz spectrometer, running at 471 MHz. Chemical shifts (δ) are reported in ppm relative to residual solvent signals (CDCl$_3$, 7.26 ppm for $^1$H-NMR and 77.2 ppm for $^{13}$C-NMR). $^{13}$C-NMR was acquired on a broad band decoupled mode. The following abbreviations are used to describe peak patterns when appropriate: s (singlet), d (doublet), t (triplet), q (quartet), quint (quintet), m (multiplet), bs (broad singlet), tt (triplet of triplets), td (triplet of doublets). Electrospray ionization has been used for measuring the exact mass (indicated for each case): MS (ESI) (Electrospray ionization mass spectroscopy) was acquired with an Agilent Technologies 6120 Quadrupole LC/MS. In this technique, MassWorks software ver. 4.0.0.0 (Cerno Bioscience) was used for the formula identification. MassWorks is a MS calibration software which calibrates for isotope profile as well as for mass accuracy, allowing highly accurate comparisons between calibrated and theoretical spectra.
2. Optimization tables

Table 1. Alkylation of 4-methylquinoline.

![Chemical structure diagram]

| Entry<sup>a</sup> | Solvent (3 mL) | Current (mA) | Time (min) | H<sup>+</sup> source (x equiv.) | Electrolite (x M) | Solvent (3 mL) | W (+) / C (-) | Conv. (%)<sup>b</sup> |
|-------------------|----------------|--------------|------------|-------------------------------|------------------|----------------|---------------|-----------------|
| 1                 | THF/H<sub>2</sub>O (2:1) | -            | 42         | RVC / Ni foam                 | -                | -              | -             | -               |
| 2                 | THF/H<sub>2</sub>O (2:1) | 10           | 42         | RVC / Ni foam                 | -                | -              | -             | 73              |
| 3<sup>d</sup>     | THF/H<sub>2</sub>O (2:1) | 10           | 42         | RVC / Ni foam                 | -                | -              | -             | 24              |
| 4<sup>e</sup>     | THF/H<sub>2</sub>O (2:1) | 10           | 42         | RVC / Ni foam                 | -                | -              | -             | 38              |
| 5<sup>f</sup>     | THF/H<sub>2</sub>O (2:1) | 10           | 42         | RVC / Ni foam                 | -                | -              | -             | 43              |
| 6                 | THF/H<sub>2</sub>O (2:1) | 30           | 42         | RVC / Ni foam                 | -                | -              | -             | 56              |
| 7                 | THF/H<sub>2</sub>O (2:1) | 3            | 42         | RVC / Ni foam                 | -                | -              | -             | 46              |
| 8                 | MeTHF / H<sub>2</sub>O (2:1) | 10           | 42         | RVC / Ni foam                 | -                | -              | -             | 27              |
| 9                 | MeOH / H<sub>2</sub>O (2:1) | 10           | 42         | RVC / Ni foam                 | -                | -              | -             | 23              |
| 10                | DMF / H<sub>2</sub>O (2:1) | 10           | 42         | RVC / Ni foam                 | -                | -              | -             | 17              |
| 11                | THF              | 10           | 42         | RVC / Ni foam                 | -                | -              | -             | 65              |
| 12                | DMF              | 10           | 42         | RVC / Ni foam                 | -                | -              | -             | 65              |
| 13<sup>g</sup>    | THF/H<sub>2</sub>O (2:1) | 10           | 42         | RVC / Ni foam                 | -                | -              | -             | 30              |
| 14<sup>i</sup>    | THF/H<sub>2</sub>O (2:1) | 10           | 42         | RVC / Ni foam                 | -                | -              | -             | 65              |
| 15                | THF/H<sub>2</sub>O (2:1) | 10           | 42         | RVC / Zn                      | -                | -              | -             | -               |
| 16                | THF/H<sub>2</sub>O (2:1) | 10           | 42         | RVC / Zn                      | -                | -              | -             | -               |
| 17                | THF/H<sub>2</sub>O (2:1) | 10           | 120        | RVC / Ni foam                 | -                | -              | >98 (92)<sup>c</sup> | -               |
| 18<sup>e</sup>    | THF/H<sub>2</sub>O (2:1) | 10           | 120        | RVC / Ni foam                 | -                | -              | 88 (80)<sup>d</sup> | -               |
| 19<sup>j</sup>    | THF/H<sub>2</sub>O (2:1) | 10           | 120        | RVC / Ni foam                 | -                | -              | 87 (75)<sup>e</sup> | -               |

<sup>a</sup>Standard reaction conditions: 0.1 mmol of 1a and 0.5 mmol of 2a in THF:H<sub>2</sub>O (2:1, 3mL) with NH<sub>4</sub>PF<sub>6</sub> (0.5 mmol) and diphenyl phosphate (0.1 mmol), under air atmosphere, were set at constant current for indicated time at room temperature. <sup>b</sup>Conversions were determined by <sup>1</sup>H-NMR. <sup>c</sup>Isolated yield in brackets. <sup>d</sup>No diphenyl phosphate. <sup>e</sup>TFA instead of diphenyl phosphate. <sup>f</sup>pTsOH instead of diphenyl phosphate. <sup>g</sup>TBAPF<sub>6</sub> instead of NH<sub>4</sub>PF<sub>6</sub>. <sup>h</sup>NH<sub>4</sub>BF<sub>4</sub> instead of NH<sub>4</sub>PF<sub>6</sub>. 0.02 mmol of diphenyl phosphate. 0.02 mmol of diphenyl phosphate and 0.2 equiv. of 2a.
Table 2. Alkylation, allylation and benzylation of acridine.

![Chemical Structures]

| Entry | X-R      | Solvent (3 mL)               | Current (mA) | Time (min) | W (+) / C (-) | Conv. (%) |
|-------|----------|------------------------------|--------------|------------|---------------|-----------|
| 1     | I-Bu     | THF/H₂O (2:1)               | 10           | 42         | RVC / Zn     | >98 (80)c |
| 2     | Br-Allyl | THF/H₂O (2:1)               | 10           | 42         | RVC / Zn     | 70        |
| 3d    | Br-Allyl | THF/H₂O (2:1)               | 10           | 42         | RVC / Ni foam| 70        |
| 4de   | Br-Allyl | THF/H₂O (2:1)               | 10           | 42         | RVC / Ni foam| 90 (62)c  |
| 5de   | Br-Allyl | DMF                          | 10           | 42         | RVC / Ni foam| -         |
| 6de.f | Br-Allyl | THF/H₂O (2:1)               | 10           | 42         | RVC / Ni foam| -         |
| 7de   | Br-Allyl | THF/H₂O (2:1)               | 3            | 42         | RVC / Ni foam| -         |
| 8de   | Br-Allyl | THF/H₂O (2:1)               | 15           | 42         | RVC / Ni foam| 55        |
| 9d    | Br-Bn    | THF/H₂O (2:1)               | 10           | 42         | RVC / Ni foam| 66        |
| 10d   | Br-Bn    | THF/H₂O (2:1)               | 10           | 42         | RVC / Zn     | 100 (70)c |
| 11d   | Br-Bn    | MeTHF/H₂O (2:1)             | 10           | 42         | RVC / Zn     | 100 (81)c |

*a*Standard reaction conditions: 0.1 mmol of 1v and 0.5 mmol of 2a in THF:H₂O (2:1, 3mL) with NH₄PF₆ (0.5 mmol) and diphenyl phosphate (0.1 mmol), under air atmosphere, were set at constant current for indicated time at room temperature. *b*Conversions were determined by ¹H-NMR. *c*Isolated yield in brackets. *d*TFA instead of diphenyl phosphate. *e*1 mmol of allyl bromide and NH₄PF₆. *f*TBAPF₆ instead of NH₄PF₆.
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3. General procedure A: Alkylation of heteroaryl compounds

Diphenyl phosphate (25.0 mg, 1 equiv.), ammonium hexafluorophosphate (81.5 mg, 5 equiv.) and a magnetic stirrer were added to a 5 mL ElectraSyn vial. Reagents were dissolved in THF (2 mL) and to the stirred solution were added 1 (0.1 mmol) and 2 (5 equiv.), followed by water (1 mL). The vial was closed, reticulated vitreous carbon was used as working electrode and nickel foam as counter electrode, ElectraSyn 2.0 was set at constant current (10 mA) during 120 min. The crude mixture was then diluted with ethyl acetate, extracted with saturated aqueous solution of NaHCO₃ (2 x 5 mL), washed with brine (3 x 30 mL), dried over anhydrous MgSO₄, filtered and the solvent was evaporated under reduced pressure. The crude mixture was purified by flash column chromatography using silica gel and the eluent indicated in each case.

2-Cyclohexyl-4-methylquinoline (3a)

Following the general procedure A; 4-methylquinoline 1a (13.2 μL, 0.1 mmol) and iodo cyclohexane 2a (64.7 μL, 0.5 mmol) gave product 3a (92% yield) as a colorless oil. Eluent: cyclohexane: ethyl acetate from 100:0 to 80:20.

¹H-NMR: δ 8.05 (d, J = 8.2 Hz, 1H), 7.94 (d, J = 8.3 Hz, 1H), 7.70 – 7.61 (m, 1H), 7.52 – 7.45 (m, 1H), 7.17 (s, 1H), 2.88 (tt, J = 11.9, 3.4 Hz, 1H), 2.68 (s, 3H), 2.07 – 1.96 (m, 2H), 1.95 – 1.84 (m, 2H), 1.83 – 1.74 (m, 1H), 1.72 – 1.55 (m, 2H), 1.54 – 1.37 (m, 2H), 1.37 – 1.24 (m, 1H) ppm.

Spectra data are consistent with those reported in the literature.

The reaction was scaled up to 1.0 mmol. Procedure A was followed using a 10 mL ElectraSyn vial as 6 mL of THF and 3 mL of water were used as solvents. The reaction was carried out at 10 mA for 16 hours. After workup and purification as described above, 3a (167 mg, 75% yield) was obtained as a slightly yellow oil.

2-Isopropyl-4-methylquinoline (3b)

Following the general procedure A; 4-methylquinoline 1a (13.2 μL, 0.1 mmol) and 2-iodopropane 2b (49.1 μL, 0.5 mmol) gave product 3b (55% yield) as a colorless oil. Eluent: cyclohexane: ethyl acetate from 100:0 to 80:20.
$^1$H-NMR: δ 8.08 (d, $J = 8.4$ Hz, 1H), 7.95 (d, $J = 8.3$ Hz, 1H), 7.72 – 7.63 (m, 1H), 7.54 – 7.46 (m, 1H), 7.18 (s, 1H), 3.32 – 3.15 (m, 1H), 2.69 (s, 3H), 1.39 (d, $J = 7.0$ Hz, 6H) ppm.

Spectra data are consistent with those reported in the literature. 3

4-Methyl-2-(tetrahydro-2H-pyran-4-yl)quinoline (3c)

Following the general procedure A; 4-methylquinoline 1a (13.2 μL, 0.1 mmol) and 4-iodotetrahydro-2H-pyran 2c (106 mg, 0.5 mmol) gave product 3c (82% yield) as a colorless oil. Eluent: cyclohexane: ethyl acetate from 100:0 to 80:20.

$^1$H-NMR: δ 8.06 (d, $J = 8.4$ Hz, 1H), 7.96 (d, $J = 8.3$ Hz, 1H), 7.72 – 7.64 (m, 1H), 7.56 – 7.47 (m, 1H), 7.18 (s, 1H), 4.13 (dd, $J = 11.0$, 2.9 Hz, 2H), 3.60 (td, $J = 11.6$, 2.5 Hz, 2H), 3.14 (tt, $J = 11.7$, 4.2 Hz, 1H), 2.70 (s, 3H), 2.11 – 1.86 (m, 4H) ppm.

Spectra data are consistent with those reported in the literature. 3

tert-Butyl 4-(4-methylquinolin-2-yl)piperidine-1-carboxylate (3d)

Following the general procedure A; 4-methylquinoline 1a (13.2 μL, 0.1 mmol) and tert-butyl 4-iodopiperidine-1-carboxylate 2d (155.5 mg, 0.5 mmol) gave product 3d (70% yield) as a colorless oil. Eluent: cyclohexane: ethyl acetate from 100:0 to 80:20.

$^1$H-NMR: δ 8.03 (d, $J = 8.5$ Hz, 1H), 7.95 (d, $J = 8.3$ Hz, 1H), 7.68 (t, $J = 7.6$ Hz, 1H), 7.51 (t, $J = 7.6$ Hz, 1H), 7.14 (s, 1H), 4.38 – 4.19 (m, 2H), 3.01 (tt, $J = 11.9$, 3.9 Hz, 1H), 2.88 (t, $J = 12.2$ Hz, 2H), 2.69 (s, 3H), 2.02 – 1.92 (m, 2H), 1.91 – 1.75 (m, 2H), 1.49 (s, 9H) ppm.

Spectra data are consistent with those reported in the literature. 4

2-Cyclopentyl-4-methylquinoline (3e)

Following the general procedure A; 4-methylquinoline 1a (13.2 μL, 0.1 mmol) and iodocyclopentane 2e (57.8 μL, 0.5 mmol) gave product 3e (65% yield) as a colorless oil. Eluent: cyclohexane: ethyl acetate from 100:0 to 80:20.

$^1$H-NMR: δ 8.05 (d, $J = 8.4$ Hz, 1H), 7.94 (d, $J = 8.3$ Hz, 1H), 7.70 – 7.62 (m, 1H), 7.53 – 7.45 (m, 1H), 7.18 (s, 1H), 3.42 – 3.26 (m, 1H), 2.68 (s, 3H), 2.24 – 2.09 (m, 2H), 1.94 – 1.80 (m, 4H), 1.80 – 1.68 (m, 2H) ppm.

Spectra data are consistent with those reported in the literature. 3

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4-Methyl-2-(tetrahydrofuran-3-yl)quinoline (3f)

Following a slightly modified procedure A; 4-methylquinoline 1a (13.2 μL, 0.1 mmol) and 3-iodotetrahydrofuran 2f (44.0 μL, 0.5 mmol) gave product 3f (54% yield) as a colorless oil when the reaction was carried out at 10 mA for 4 hours. Eluent: cyclohexane: ethyl acetate from 100:0 to 80:20.

$^1$H-NMR: δ 8.03 (d, $J = 8.5$ Hz, 1H), 7.96 (d, $J = 8.2$ Hz, 1H), 7.73 – 7.64 (m, 1H), 7.57 – 7.48 (m, 1H), 7.21 (s, 1H), 4.28 – 4.11 (m, 2H), 4.06 (dd, $J = 8.6$, 6.7 Hz, 1H), 3.95 (q, $J = 7.9$ Hz, 1H), 3.81 – 3.67 (m, 1H), 2.69 (s, 3H), 2.54 – 2.40 (m, 1H), 2.38 – 2.23 (m, 1H) ppm.

Spectra data are consistent with those reported in the literature.

2-(tert-Butyl)-4-methylquinoline (3g)

Following a slightly modified procedure A; 4-methylquinoline 1a (13.2 μL, 0.1 mmol) and 2-iodo-2-methylpropane 2g (59.6 μL, 0.5 mmol) gave product 3g (78% yield) as a colorless oil, when Zn was used as counterelectrode. Eluent: cyclohexane: ethyl acetate from 100:0 to 80:20.

$^1$H-NMR: δ 8.07 (d, $J = 8.3$ Hz, 1H), 7.94 (d, $J = 8.3$ Hz, 1H), 7.71 – 7.62 (m, 1H), 7.54 – 7.45 (m, 1H), 7.36 (s, 1H), 2.69 (s, 3H), 1.46 (s, 9H) ppm.

Spectra data are consistent with those reported in the literature.

2-Ethyl-4-methylquinoline (3h)

Following a slightly modified procedure A; 4-methylquinoline 1a (6.6 μL, 0.05 mmol) and iodoethane 2h (40.2 μL, 0.5 mmol) gave product 3h (63% yield) as a colorless oil when the reaction was carried out at 10 mA for 4 hours in 2 mL of THF and 1 mL of H$_2$O with 1 equivalent of diphenyl phosphate and 5 equivalents of electrolyte. Eluent: cyclohexane: ethyl acetate from 100:0 to 80:20.

$^1$H-NMR: δ 8.11 (d, $J = 8.4$ Hz, 1H), 7.96 (d, $J = 8.3$ Hz, 1H), 7.73 – 7.64 (m, 1H), 7.56 – 7.47 (m, 1H), 7.18 (s, 1H), 2.99 (q, $J = 7.6$ Hz, 2H), 2.70 (s, 3H), 1.39 (t, $J = 7.6$ Hz, 3H) ppm.

Spectra data are consistent with those reported in the literature.

4-Methyl-2-phenethylquinoline (3i)

Following a slightly modified procedure A; 4-methylquinoline 1a (6.6 μL, 0.05 mmol) and (2-iodoethyl)benzene 2i (72.4 μL, 0.5 mmol) gave product 3i (41% yield) as a colorless oil when the reaction was carried
out at 10 mA for 4 hours in 2 mL of THF and 1 mL of H$_2$O with 1 equivalent of diphenyl phosphate and 5 equivalents of electrolyte. Eluent: cyclohexane: ethyl acetate from 100:0 to 80:20.

$^1$H-NMR: δ 8.07 (d, $J = 8.5$ Hz, 1H), 7.97 (d, $J = 8.2$ Hz, 1H), 7.69 (t, $J = 7.7$ Hz, 1H), 7.52 (t, $J = 7.6$ Hz, 1H), 7.33 – 7.26 (m, 5H), 7.24 – 7.16 (m, 1H), 7.11 (s, 1H), 3.30 – 3.20 (m, 2H), 3.19 – 3.07 (m, 2H), 2.67 (s, 3H) ppm.

Spectra data are consistent with those reported in the literature.  

**2-Cyclohexyl-7-methoxy-4-methylquinoline (3j)**  

Following the general procedure A; 7-methoxy-4-methylquinoline 1b (20.9 mg, 0.1 mmol) and iodocyclohexane 2a (64.7 μL, 0.5 mmol) gave product 3j (68% yield) as a colorless oil. Eluent: cyclohexane: ethyl acetate from 100:0 to 80:20.

$^1$H-NMR: δ 7.82 (d, $J = 9.1$ Hz, 1H), 7.39 (d, $J = 2.6$ Hz, 1H), 7.14 (dd, $J = 9.1$, 2.6 Hz, 1H), 7.03 (s, 1H), 3.94 (s, $J = 4.8$ Hz, 1H), 2.83 (tt, $J = 11.9$, 3.4 Hz, 1H), 2.64 (s, $J = 0.6$ Hz, 1H), 2.06 – 1.96 (m, 1H), 1.95 – 1.84 (m, 1H), 1.83 – 1.73 (m, 1H), 1.69 – 1.57 (m, 1H), 1.56 – 1.28 (m, 1H) ppm.

$^{13}$C-NMR: δ 167.1, 160.5, 149.6, 144.4, 124.9, 122.2, 118.5, 118.4, 107.8, 55.7, 47.9, 33.1 (2C), 26.8 (2C), 26.3, 19.0 ppm.

HRMS (ESI$^+$): calculated for C$_{17}$H$_{21}$NO $[M-H]^+$: 256.1696; found: 256.1638.

**7-Bromo-2-cyclohexyl-4-methylquinoline (3k)**  

Following the general procedure A; 7-bromo-4-methylquinoline 1c (22.2 mg, 0.1 mmol) and iodocyclohexane 2a (64.7 μL, 0.5 mmol) gave product 3k (64% yield) as a colorless oil. Eluent: cyclohexane: ethyl acetate from 100:0 to 80:20.

$^1$H-NMR: δ 8.23 (d, $J = 2.0$ Hz, 1H), 7.79 (d, $J = 8.9$ Hz, 1H), 7.56 (dd, $J = 8.9$, 2.0 Hz, 1H), 7.16 (d, $J = 0.8$ Hz, 1H), 2.84 (tt, $J = 11.9$, 3.4 Hz, 1H), 2.66 (d, $J = 0.8$ Hz, 3H), 2.04 – 1.94 (m, 2H), 1.94 – 1.84 (m, 2H), 1.84 – 1.74 (m, 1H), 1.71 – 1.54 (m, 3H), 1.54 – 1.35 (m, 2H) ppm.

$^{13}$C-NMR: δ 167.9, 148.7, 144.5, 132.0, 128.9, 125.9, 125.2, 123.2, 121.0, 47.6, 32.9 (2C), 26.7 (2C), 26.3, 18.9 ppm.

HRMS (ESI$^+$): calculated for C$_{16}$H$_{19}$BrN $[M-H]^+$: 304.0695; found: 304.0639.
6-Bromo-2-cyclohexyl-4-methylquinoline (3l)

Following the general procedure A; 6-bromo-4-methylquinoline 1d (22.2 mg, 0.1 mmol) and iodo-cyclohexane 2a (64.7 μL, 0.5 mmol) gave product 3l (73% yield) as a colorless oil. Eluent: cyclohexane: ethyl acetate from 100:0 to 90:10.

$^1$H-NMR: δ 8.08 (d, $J = 2.2$ Hz, 1H), 7.90 (d, $J = 8.9$ Hz, 1H), 7.72 (dd, $J = 8.9, 2.2$ Hz, 1H), 7.17 (s, 1H), 2.84 (tt, $J = 11.8, 3.4$ Hz, 1H), 2.64 (s, 3H), 2.05 – 1.95 (m, 2H), 1.95 – 1.84 (m, 2H), 1.84 – 1.73 (m, 2H), 1.69 – 1.51 (m, 3H), 1.50 – 1.30 (m, 2H) ppm.

$^{13}$C-NMR: δ 167.2, 146.5, 143.6, 132.5, 131.5, 128.5, 126.2, 121.3, 119.4, 47.7, 32.9 (2C), 26.7 (2C), 26.3, 19.0 ppm.

HRMS (ESI$^+$): calculated for C$_{16}$H$_{19}$BrN [M-H]$^-$: 304.0695; found: 304.0670.

2-Cyclohexyl-4-phenylquinoline (3m)

Following the general procedure A; 4-phenylquinoline 1e (20.5 mg, 0.1 mmol) and iodo-cyclohexane 2a (64.7 μL, 0.5 mmol) gave product 3m (54% yield) as a colorless oil. Eluent: cyclohexane: ethyl acetate from 100:0 to 95:5.

$^1$H-NMR: δ 8.13 (d, $J = 8.4$ Hz, 1H), 7.86 (d, $J = 8.4$ Hz, 1H), 7.68 (ddd, $J = 8.4, 6.9, 1.2$ Hz, 1H), 7.55 – 7.47 (m, 5H), 7.43 (ddd, $J = 8.4, 6.9, 1.2$ Hz, 1H), 7.27 (s, 1H), 3.05 – 2.88 (m, 1H), 2.14 – 2.02 (m, 2H), 1.98 – 1.84 (m, 2H), 1.84 – 1.75 (m, 1H), 1.75 – 1.59 (m, 2H), 1.58 – 1.41 (m, 2H), 1.39 – 1.28 (m, 1H) ppm.

Spectra data are consistent with those reported in the literature.$^5$

2-(2-Cyclohexylquinolin-4-yl)-1-(4-(trifluoromethyl)phenyl)ethan-1-one (3n)

Following a slightly modified procedure A; 2-(quinolin-4-yl)-1-(4-(trifluoromethyl)phenyl)ethan-1-one 1f (31.5 mg, 0.1 mmol) and iodo-cyclohexane 2a (64.7 μL, 0.5 mmol) gave product 3n (63% yield) as a yellow oil when it was carried out at 7.5 mA for 4 hours. Eluent: cyclohexane: ethyl acetate from 100:0 to 95:5 to 90:10.

$^1$H-NMR: δ 8.16 (d, $J = 8.6$ Hz, 2H), 8.10 (d, $J = 8.4$ Hz, 1H), 7.82 – 7.74 (m, 3H), 7.68 (ddd, $J = 8.4, 6.9, 1.2$ Hz, 1H), 7.48 (ddd, $J = 8.4, 6.9, 1.2$ Hz, 1H), 7.19 (s, 1H), 4.73 (s, 2H), 2.88 (tt, $J = 11.7, 3.3$ Hz, 1H), 2.07 – 1.95 (m, 2H), 1.94 – 1.83 (m, 2H), 1.83 – 1.73 (m, 1H), 1.69 – 1.53 (m, 2H), 1.53 – 1.24 (m, 3H) ppm.
\[^{13}C\text{-NMR:}\ δ 195.6, 166.6, 148.3, 140.4, 139.2, 135.08 (q, J = 32.8 Hz), 130.1, 129.5, 129.0 (2C),
126.4, 126.3, 126.1 (q, J = 3.7 Hz, 2C), 123.7 (q, J = 272.8 Hz), 123.3, 121.6, 47.6, 42.9, 32.9 (2C), 26.7 (2C), 26.2 ppm.
\]

\[^{19}F\text{-NMR:}\ δ -63.2 ppm.

HRMS (ESI\(^+\)): calculated for C\(_{24}\)H\(_{23}\)F\(_3\)NO [M-H]\(^+\): 398.1726; found: 398.1787.

4-Bromo-2-cyclohexylquinoline (3o)

Following a slightly modified procedure A; 4-bromoquinoline 1g (20.8 mg, 0.1 mmol) and iodocyclohexane 2a (64.7 μL, 0.5 mmol) gave product 3o (44% yield) as a colorless oil when it was carried out at 5 mA for 1 hour. Eluent: cyclohexane: ethyl acetate from 100:0 to 90:10.

\[^{1}H\text{-NMR:}\ δ 8.13 (d, J = 8.4 Hz, 1H), 8.03 (d, J = 8.4 Hz, 1H), 7.77 – 7.68 (m, 1H), 7.63 (s, 1H), 7.61 – 7.53 (m, 1H), 2.88 (tt, J = 11.8, 3.3 Hz, 1H), 2.09 – 1.98 (m, 2H), 1.97 – 1.85 (m, 2H), 1.84 – 1.74 (m, 1H), 1.70 – 1.54 (m, 2H), 1.54 – 1.40 (m, 2H), 1.40 – 1.24 (m, 1H) ppm.

Spectra data are consistent with those reported in the literature.\(^5\)

Methyl 6-bromo-2-cyclohexylquinoline-4-carboxylate (3p)

Following a slightly modified procedure A; ethyl 6-bromoquinoline-4-carboxylate 1h (26.6 mg, 0.1 mmol) and iodocyclohexane 2a (64.7 μL, 0.5 mmol) gave product 3p (46% yield) as a colorless oil when the reaction was carried out at 5 mA for 90 minutes. Eluent: cyclohexane: ethyl acetate from 100:0 to 95:5.

\[^{1}H\text{-NMR:}\ δ 8.93 (d, J = 2.1 Hz, 1H), 7.95 (d, J = 9.0 Hz, 1H), 7.87 – 7.68 (m, 1H), 7.63 (s, 1H), 7.61 – 7.53 (m, 1H), 2.93 (tt, J = 11.9, 3.4 Hz, 1H), 2.07 – 1.97 (m, 2H), 1.96 – 1.86 (m, 2H), 1.85 – 1.75 (m, 1H), 1.74 – 1.58 (m, 2H), 1.55 – 1.42 (m, 2H), 1.37 – 1.27 (m, 1H) ppm.

\[^{13}C\text{-NMR:}\ δ 166.9, 166.6, 147.6, 134.2, 133.2, 131.4, 128.0, 125.0, 122.3, 121.8, 53.0, 47.5, 32.7 (2C), 26.6 (2C), 26.2 ppm.

HRMS (ESI\(^+\)): calculated for C\(_{17}\)H\(_{19}\)BrNO\(_2\) [M-H]\(^+\): 348.0594; found: 348.0600.

2-Cyclohexyl-N-methylquinoline-4-carboxamide (3q)

Following a slightly modified procedure A; N-methylquinoline-4-carboxamide 1j (18.6 mg, 0.1 mmol) and iodocyclohexane 2a (64.7 μL, 0.5 mmol) gave product 3q (55% yield) as a colorless oil when the reaction was carried out at 15 mA for 1 hour. Eluent: cyclohexane: ethyl acetate from
100:0 to 80:20.

$^1$H-NMR: $\delta$ 8.10 (d, $J = 8.3$ Hz, 1H), 8.04 (d, $J = 8.5$ Hz, 1H), 7.74 – 7.64 (m, 1H), 7.55 – 7.45 (m, 1H), 7.34 (s, 1H), 6.25 (s, 1H), 3.07 (d, $J = 4.7$, 3H), 2.87 (t, $J = 10.9$ Hz, 1H), 2.03 – 1.73 (m, 6H), 1.68 – 1.21 (m, 4H) ppm.

$^{13}$C-NMR: $\delta$ 168.7, 166.5, 148.4, 142.6, 130.0, 129.5, 126.8, 125.1, 123.3, 117.3, 47.6, 32.9 (2C), 26.9, 26.6 (2C), 26.2 ppm.

HRMS (ESI$^+$): calculated for $C_{17}H_{21}N_2O$ [M-H]$^-$: 269.1648; found: 269.1605.

4-Cyclohexyl-2-methylquinoline (3r)

Following the general procedure A; 2-methylquinoline 1I (13.1 µL, 0.1 mmol) and iodocyclohexane 2a (64.7 µL, 0.5 mmol) gave product 3r (76% yield) as a colorless oil. Eluent: cyclohexane: ethyl acetate from 100:0 to 80:20.

$^1$H-NMR: $\delta$ 8.09 – 7.98 (m, 2H), 7.70 – 7.58 (m, 1H), 7.57 – 7.42 (m, 1H), 7.16 (s, 1H), 3.39 – 3.19 (m, 1H), 2.72 (s, 3H), 2.09 – 1.79 (m, 5H), 1.64 – 1.44 (m, 3H), 1.44 – 1.23 (m, 2H) ppm.

Spectra data are consistent with those reported in the literature.$^3$

6-Chloro-4-cyclohexyl-2-methylquinoline (3s)

Following the general procedure A; 6-chloro-2-methylquinoline 1m (17.8 mg, 0.1 mmol) and iodocyclohexane 2a (64.7 µL, 0.5 mmol) gave product 3s (69% yield) as a colorless oil. Eluent: cyclohexane: ethyl acetate from 100:0 to 90:10.

$^1$H-NMR: $\delta$ 8.00 – 7.93 (m, 2H), 7.58 (dd, $J = 9.0$, 2.3 Hz, 1H), 7.18 (s, 1H), 3.24 – 3.12 (m, 1H), 2.70 (s, 3H), 2.03 – 1.81 (m, 5H), 1.64 – 1.46 (m, 4H), 1.43 – 1.24 (m, 1H) ppm.

$^{13}$C-NMR: $\delta$ 159.4, 152.8, 146.7, 131.3, 131.3, 129.8, 126.1, 122.2, 119.4, 39.0, 33.7 (2C), 27.0 (2C), 26.4, 25.7 ppm.

HRMS (ESI$^+$): calculated for $C_{16}H_{19}ClN$ [M-H]$^-$: 260.1201; found: 260.1190.

Methyl 4-cyclohexyl-2-methylquinoline-6-carboxylate (3t)

Following the general procedure A; ethyl 2-methylquinoline-6-carboxylate 1n (20.1 mg, 0.1 mmol) and iodocyclohexane 2a (64.7 µL, 0.5 mmol) gave product 3t (66% yield) as a colorless oil. Eluent: cyclohexane: ethyl acetate from 100:0 to 90:10.
$^1$H-NMR: δ 8.80 (d, J = 1.6 Hz, 1H), 8.23 (dd, J = 8.8, 1.6 Hz, 1H), 8.04 (d, J = 8.8 Hz, 1H), 7.23 (s, 1H), 4.00 (s, 3H), 3.46 – 3.30 (m, 1H), 2.74 (s, 3H), 2.07 – 1.82 (m, 5H), 1.70 – 1.49 (m, 4H), 1.47 – 1.28 (m, 1H) ppm.

$^{13}$C-NMR: δ 167.3, 161.6, 155.1, 150.4, 130.0, 128.6, 127.0, 126.3, 124.6, 119.4, 52.6, 38.8, 34.0 (2C), 27.0 (2C), 26.4, 25.9 ppm.

HRMS (ESI$^+$): calculated for C$_{18}$H$_{22}$N$_2$O$_2$ [M-H]$^+$: 284.1645; found: 284.1637.

2-Cyclohexylquinoline (3u) and 4-Cyclohexylquinoline (3u')

Following a slightly modified procedure A; quinoline 1w (11.8 μL, 0.1 mmol) and iodosicyclohexane 2a (64.7 μL, 0.5 mmol) gave product 3u (26% yield) and 3u' (24% yield) as a colorless oil when the reaction was carried out at 5 m for 60 minutes. Eluent: cyclohexane: ethyl acetate from 100:0 to 80:20.

$^1$H-NMR: δ 8.13 – 7.99 (m, 2H), 7.82 – 7.73 (m, 1H), 7.73 – 7.58 (m, 1H), 7.47 (dd, J = 8.5, 6.9 Hz, 1H), 7.33 (d, J = 8.5 Hz, 1H), 3.01 – 2.84 (m, 1H), 2.11 – 1.97 (m, 2H), 1.97 – 1.85 (m, 2H), 1.82 – 1.63 (m, 1H), 1.57 – 1.45 (m, 2H), 1.38 – 1.10 (m, 2H), 0.97 – 0.80 (m, 1H) ppm.

Spectra data are consistent with those reported in the literature.$^9$

$^1$H-NMR: δ 8.85 (d, J = 4.7 Hz, 1H), 8.16 – 8.06 (m, 2H), 7.69 (ddd, J = 8.4, 6.8, 1.4 Hz, 1H), 7.56 (ddd, J = 8.4, 6.8, 1.4 Hz, 1H), 7.28 (d, J = 4.7 Hz, 1H), 3.42 – 3.28 (m, 1H), 2.08 – 1.81 (m, 6H), 0.99 – 0.79 (m, 4H) ppm.

Spectra data are consistent with those reported in the literature.$^9$

1-Cyclohexyl-3-methylisoquinolin-4-ol (3v)

Following the general procedure A; 3-methylisoquinoline 1x (14.3 mg, 0.1 mmol) and iodosicyclohexane 2a (64.7 μL, 0.5 mmol) gave product 3v (77% yield) as a slightly yellow solid. Eluent: cyclohexane: ethyl acetate from 100:0 to 80:20.

$^1$H-NMR: δ 8.21 – 8.05 (m, 2H), 7.70 – 7.57 (m, 1H), 7.57 – 7.42 (m, 1H), 3.43 (tt, J = 11.2, 3.3 Hz, 1H), 2.63 (s, 3H), 1.97 – 1.84 (m, 4H), 1.86 – 1.72 (m, 3H), 1.63 – 1.45 (m, 2H), 1.42 – 1.28 (m, 1H) ppm.

$^{13}$C-NMR: δ 157.3, 142.2, 133.0, 128.8, 128.0, 126.0 (2C), 124.8, 121.1, 41.4, 32.8 (2C), 27.1 (2C), 26.4, 18.7 ppm.

HRMS (ESI$^+$): calculated for C$_{16}$H$_{20}$NO [M-H]$^+$: 242.1539; found: 242.1544.
2-Cyclohexyl-4,7-diphenyl-1,10-phenanthroline (3w)

Following a slightly modified procedure A; 4,7-diphenyl-1,10-phenanthroline 1y (33.2 mg, 0.1 mmol) and iodocyclohexane 2a (64.7 μL, 0.5 mmol) gave product 3w (35% yield) as a yellowish oil when the reaction was carried out at 5 mA. Eluent: cyclohexane: ethyl acetate from 100:0 to 50:50.

$^1$H-NMR: δ 9.28 (dd, $J = 4.5$, 0.7 Hz, 1H), 7.79 (dd, $J = 3.0$, 0.7 Hz, 2H), 7.59 – 7.47 (m, 12H), 3.41 (tt, $J = 12.0$, 3.4 Hz, 1H), 2.25 – 2.14 (m, 2H), 1.99 – 1.77 (m, 2H), 1.74 – 1.45 (m, 5H), 1.45 – 1.20 (m, 1H) ppm.

Spectra data are consistent with those reported in the literature.

6-Cyclohexylphenanthridine (3x)

Following the general procedure A; phenanthridine 1o (17.9 mg, 0.1 mmol) and iodocyclohexane 2a (64.7 μL, 0.5 mmol) gave product 3x (70% yield) as a white solid. Eluent: cyclohexane: ethyl acetate from 100:0 to 95:5.

$^1$H-NMR: δ 8.66 (d, $J = 8.2$ Hz, 1H), 8.54 (dd, $J = 8.2$, 1.3 Hz, 1H), 8.32 (d, $J = 8.2$ Hz, 1H), 8.14 (d, $J = 7.6$ Hz, 1H), 7.82 (t, $J = 7.4$ Hz, 1H), 7.75 – 7.66 (m, 2H), 7.64 – 7.57 (m, 1H), 3.62 (t, $J = 11.3$ Hz, 1H), 2.14 – 2.04 (m, 2H), 2.03 – 1.91 (m, 3H), 1.90 – 1.80 (m, 1H), 1.68 – 1.40 (m, 4H) ppm.

Spectra data are consistent with those reported in the literature.

6-Cyclohexylbenzo[d]thiazole (3y)

Following a slightly modified procedure A; benzo[d]thiazole 1p (10.9 μL, 0.1 mmol) and iodocyclohexane 2a (64.7 μL, 0.5 mmol) gave product 3y (60% yield) as a colorless oil when the reaction was carried out during 8 hours. Eluent: cyclohexane: ethyl acetate from 100:0 to 95:5.

$^1$H-NMR: δ 8.00 – 7.93 (m, 1H), 7.89 – 7.80 (m, 1H), 7.48 – 7.40 (m, 1H), 7.38 – 7.29 (m, 1H), 3.11 (tt, $J = 11.6$, 3.6 Hz, 1H), 2.31 – 2.15 (m, 2H), 1.99 – 1.83 (m, 2H), 1.82 – 1.59 (m, 3H), 1.53 – 1.22 (m, 3H) ppm.

Spectra data are consistent with those reported in the literature.

2-Cyclohexyl-1-methyl-1H-benzo[d]imidazole (3z)

Following a slightly modified procedure A; 1-methyl-1H-benzo[d]imidazole 1q (13.2 mg, 0.1 mmol) and iodocyclohexane 2a (64.7 μL, 0.5 mmol) gave product 3z (56% yield) as a colorless oil when the reaction was carried out during 8 hours. Eluent: cyclohexane: ethyl acetate from 100:0 to 90:10.
$^1$H-NMR: $\delta$ 7.80 – 7.70 (m, 1H), 7.33 – 7.18 (m, 3H), 3.75 (s, 3H), 2.85 (tt, $J$ = 11.6, 3.4 Hz, 1H), 2.05 – 1.87 (m, 4H), 1.87 – 1.73 (m, 3H), 1.52 – 1.32 (m, 3H) ppm. Spectra data are consistent with those reported in the literature.$^3$

5,7-Dichloro-2-cyclohexyl-4-(4-fluorophenoxy)quinoline (3aa)

Following a slightly modified procedure A; 5,7-dichloro-4-(4-fluorophenoxy)quinoline 1r (30.8 mg, 0.1 mmol) and iodo cyclohexane 2a (64.7 $\mu$L, 0.5 mmol) gave product 3aa (45% yield) as a white solid when the reaction was carried out at 5 mA. Eluent: cyclohexane: ethyl acetate from 100:0 to 95:5 to 90:10.

$^1$H-NMR: $\delta$ 7.96 (d, $J$ = 2.1 Hz, 1H), 7.51 (d, $J$ = 2.1 Hz, 1H), 7.21 – 7.05 (m, 4H), 6.52 (s, 1H), 2.78 – 2.58 (m, 1H), 1.94 – 1.69 (m, 5H), 1.54 – 1.21 (m, 5H) ppm. Spectra data are consistent with those reported in the literature.$^3$

(R)-{2-Cyclohexyl-6-methoxyquinolin-4-yl}[(1S,2S,4S,5R)-5-vinylquinuclidin-2-yl]methanol (3ab)

Following a slightly modified procedure A; (R)-{6-methoxyquinolin-4-yl}[(1S,2S,4S,5R)-5-vinylquinuclidin-2-yl]methanol 1s (32.4 mg, 0.1 mmol) and iodo cyclohexane 2a (64.7 $\mu$L, 0.5 mmol) gave product 3ab (74% yield) as a white solid when it was carried out during 4 hours. Eluent: dichloromethane: methanol from 97:3 to 93:7.

$^1$H-NMR: $\delta$ 7.96 (d, $J$ = 9.2 Hz, 1H), 7.48 (s, 1H), 7.31 (dd, $J$ = 9.2, 2.6 Hz, 1H), 7.19 (s, 1H), 5.82 – 5.66 (m, 1H), 5.62 – 5.56 (m, 1H), 5.04 – 4.88 (m, 2H), 3.88 (s, 3H), 3.58 – 3.41 (m, 1H), 3.20 – 3.05 (m, 2H), 2.91 – 2.77 (m, 1H), 2.76 – 2.61 (m, 2H), 2.38 – 2.19 (m, 1H), 2.07 – 1.92 (m, 3H), 1.94 – 1.68 (m, 6H), 1.61 – 1.44 (m, 4H), 1.37 – 1.19 (m, 3H) ppm. Spectra data are consistent with those reported in the literature.$^6$

(R)-{2-Cyclohexylquinolin-4-yl}[(1S,2S,4S,5R)-5-vinylquinuclidin-2-yl]methanol (3ac)

Following a slightly modified procedure A; (R)-quinolin-4-yl[(1S,2S,4S,5R)-5-vinylquinuclidin-2-yl]methanol 1t (29.4 mg, 0.1 mmol) and iodo cyclohexane 2a (64.7 $\mu$L, 0.5 mmol) gave product 3ac (71% yield) as a white solid when the reaction was carried out during 4 hours. Eluent: dichloromethane: methanol from 97:3 to 93:7.

$^1$H-NMR: $\delta$ 8.07 (d, $J$ = 8.3 Hz, 1H), 7.94 (d, $J$ = 8.2 Hz, 1H), 7.65 (ddd, $J$ = 8.3, 7.0, 1.2 Hz, 1H), 7.53 (s, 1H), 7.44 (ddd, $J$ = 8.2, 7.0, 1.2 Hz, 1H), 5.82 – 5.63 (m, 2H), 5.03 – 4.86 (m, 2H), 3.54 –
3.39 (m, 1H), 3.18 – 3.04 (m, 2H), 2.90 (tt, \( J = 12.0, 3.4 \) Hz, 1H), 2.75 – 2.60 (m, 2H), 2.32 – 2.20 (m, 1H), 2.04 – 1.95 (m, 2H), 1.94 – 1.83 (m, 2H), 1.83 – 1.59 (m, 7H), 1.59 – 1.38 (m, 5H) ppm.

\(^{13}\text{C-NMR:} \, \delta 166.7, 149.4, 148.0, 124.0, 129.0, 125.9, 124.4, 122.7, 116.7, 114.5, 72.1, 60.4, 47.9, 43.5, 40.1, 33.0, 28.2, 27.1, 26.7 (2C), 26.2, 21.0\) ppm.

HRMS (ESI\(^+\)): calculated for C\(_{25}\)H\(_{33}\)N\(_2\)O \[M - \text{H}\]^+: 377.2587; found: 377.2518.

(S)-11-Cyclohexyl-4-ethyl-4-hydroxy-1,12-dihydro-14H-pyrano[3′,4′:6,7]indolizino[1,2-b]quinoline-3,14(4H)-dione (3ad)

Following a slightly modified procedure A; (S)-4-ethyl-4-hydroxy-1,12-dihydro-14H-pyrano[3′,4′:6,7]indolizino[1,2-b]quinoline-3,14(4H)-dione 1u (34.8 mg, 0.1 mmol) and iodocyclohexane 2a (64.7 \( \mu \)L, 0.5 mmol) gave product 3ad (38% yield) as a slightly yellow solid when the reaction was carried out at 5 mA. Eluent: dichloromethane: methanol from 100:0 to 95:5.

\(^1\text{H-NMR:} \, \delta 8.29 – 8.19 (m, 2H), 7.79 (t, \( J = 7.0 \) Hz, 1H), 7.71 – 7.62 (m, 2H), 5.76 (d, \( J = 16.3 \) Hz, 1H), 5.42 (s, 1H), 5.31 (d, \( J = 16.3 \) Hz, 1H), 3.75 (bs, 1H), 3.64 (bs, 1H), 2.06 – 1.96 (m, 4H), 1.96 – 1.78 (m, 4H), 1.72 – 1.53 (m, 4H), 1.04 (t, \( J = 7.4 \) Hz, 3H) ppm.

\(^{13}\text{C-NMR:} \, \delta 167.7, 166.6, 147.6, 134.3, 133.2, 131.4, 126.0, 125.0, 121.9, 121.8, 53.0, 37.3, 22.4 (2C) ppm.

HRMS (ESI\(^+\)): calculated for C\(_{21}\)H\(_{19}\)BrNO\(_2\) [M-H]^+: 308.0281; found: 308.0259.

Methyl 6-bromo-2-isopropylquinoline-4-carboxylate (6)

Following a slightly modified procedure A; ethyl 6-bromoquinoline-4-carboxylate 1h (26.6 mg, 0.1 mmol) and 2-iodopropane 2b (49.1 \( \mu \)L, 0.5 mmol) gave product 6 (39% yield) as a colorless oil when it was carried out at 5 mA for 90 minutes. Eluent: cyclohexane: ethyl acetate from 100:0 to 95:5.

\(^1\text{H-NMR:} \, \delta 8.93 (d, \( J = 2.2 \) Hz, 1H), 7.96 (d, \( J = 9.0 \) Hz, 1H), 7.87 (s, 1H), 7.79 (dd, \( J = 9.0, 2.2 \) Hz, 1H), 4.05 (s, 3H), 3.38 – 3.16 (m, 1H), 1.40 (d, \( J = 6.9 \) Hz, 6H) ppm.

\(^{13}\text{C-NMR:} \, \delta 167.7, 166.6, 147.6, 134.3, 133.2, 131.4, 126.0, 125.0, 121.9, 121.8, 53.0, 37.3, 22.4 (2C) ppm.

HRMS (ESI\(^+\)): calculated for C\(_{14}\)H\(_{15}\)BrNO\(_2\) [M-H]^+: 308.0281; found: 308.0259.

Methyl 2-cyclohexylquinoline-4-carboxylate (8)

Following a slightly modified procedure A; ethyl quinoline-4-carboxylate 1i (18.7 mg, 0.1 mmol) and iodocyclohexane 2a (64.7 \( \mu \)L, 0.5 mmol) gave product 8 (53% yield) as a colorless oil when the reaction was carried out
during 1 hour. Eluent: cyclohexane: ethyl acetate from 100:0 to 95:5.

$^1$H-NMR: δ 8.68 (d, $J = 8.2$ Hz, 1H), 8.10 (d, $J = 8.5$ Hz, 1H), 7.83 (s, 1H), 7.76 – 7.68 (m, 1H), 7.62 – 7.53 (m, 1H), 4.04 (s, 3H), 2.96 (tt, $J = 11.9$, 3.4 Hz, 1H), 2.10 – 1.98 (m, 2H), 1.96 – 1.86 (m, 2H), 1.84 – 1.75 (m, 1H), 1.74 – 1.59 (m, 2H), 1.57 – 1.30 (m, 3H) ppm.

Spectra data are consistent with those reported in the literature.

*2-Cyclohexylquinoline-4-carboxylic acid (9)*

Following a slightly modified procedure A; quinoline-4-carboxylic acid 1k (17.3 mg, 0.1 mmol) and iodo cyclohexane 2a (64.7 μL, 0.5 mmol) gave product 9 (30% yield) as a colorless oil when the reaction was carried out at 5 mA for 1 hour. Eluent: cyclohexane: ethyl acetate from 100:0 to 80:20.

$^1$H-NMR: δ 8.77 (d, $J = 8.3$ Hz, 1H), 8.14 (d, $J = 8.3$ Hz, 1H), 7.94 (s, 1H), 7.58 (t, $J = 7.5$ Hz, 1H), 3.00 (t, $J = 11.9$ Hz, 1H), 2.08 – 2.00 (m, 2H), 1.96 – 1.85 (m, 2H), 1.83 – 1.74 (m, 1H), 1.73 – 1.58 (m, 2H), 1.56 – 1.29 (m, 3H) ppm.

Spectra data are consistent with those reported in the literature.

*3-Cyclohexyl-N,2-diphenylpropanamide (11)*

Following the general procedure A; N,2-diphenylacrylamide 10 (22.3 mg, 0.1 mmol) and iodo cyclohexane 2a (64.7 μL, 0.5 mmol) gave product 11 (70% yield) as a colorless oil. Eluent: cyclohexane: ethyl acetate from 100:0 to 95:5

$^1$H-NMR: δ 7.43 (d, $J = 7.7$ Hz, 2H), 7.36 (d, $J = 4.3$ Hz, 4H), 7.34 – 7.25 (m, 3H), 7.19 (s, 1H), 7.06 (t, $J = 7.3$ Hz, 1H), 3.65 (t, $J = 7.6$ Hz, 1H), 2.21 – 2.02 (m, 1H), 1.83 – 1.59 (m, 6H), 1.24 – 1.07 (m, 4H), 1.04 – 0.85 (m, 2H) ppm.

$^{13}$C-NMR: δ 172.1, 140.1, 138.1, 129.2 (2C), 129.1 (2C), 128.2 (2C), 127.6, 124.4, 119.9 (2C), 51.6, 40.9, 35.3, 33.8, 33.0, 26.7, 26.3, 26.2 ppm.

HRMS (ESI⁺): calculated for C21H26NO [M-H]⁺: 308.2009; found: 308.2040.
4. General procedure B: Alkylation, allylation and benzylation of acridine

Acridine 1 (17.9 mg, 1 equiv.), ammonium hexafluorophosphate (82 mg, 5 equiv.) and a magnetic stirrer were added to a 5 mL ElectraSyn vial. Reagents were dissolved in 2 mL of THF (or Me-THF) and to the stirred solution were added trifluoroacetic acid (7.3 μL, 1 equiv.) and 2 (5 equiv.), followed by water (1 mL). The vial was closed, reticulated vitreous carbon was used as working electrode and zinc as counterelectrode, ElectraSyn 2.0 was set at constant current (10 mA) during 42 min. The crude mixture was then diluted with diethyl ether, extracted with saturated aqueous solution of NaHCO₃ (2 x 5 mL), washed with brine (3 x 30 mL), dried over anhydrous MgSO₄, filtered and the solvent was evaporated under reduced pressure. The crude mixture was purified by flash column chromatography using the eluent indicated in each case.

9-(tert-Butyl)-9,10-dihydroacridine (4a)

Following the general procedure B; acridine 1v (17.9 mg, 0.1 mmol) and 2-iodo-2-methy propane 2g (59.6 μL, 0.5 mmol) gave product 4a (80% yield) as a white solid. Eluent: cyclohexane: ethyl acetate from 100:0 to 95:5.

¹H-NMR: δ 7.18 – 7.10 (m, 4H), 6.94 – 6.87 (m, 2H), 6.79 – 6.73 (m, 2H), 6.00 (s, 1H), 3.63 (s, 1H), 0.81 (s, 9H) ppm.

¹³C-NMR: δ 141.4 (2C), 131.3 (2C), 127.0 (2C), 121.8 (2C), 120.1 (2C), 113.7 (2C), 53.4, 38.5, 27.5 (3C) ppm.

HRMS (ESI⁺): calculated for C₁₇H₂₀N [M-H]⁺: 238.1590; found: 238.1603.

9-Allyl-9,10-dihydroacridine (4b)

Following a modified procedure B; acridine 1v (17.9 mg, 0.1 mmol) and 3-bromoprop-1-ene 2j (86.6 μL, 1.0 mmol) when employing ammonium hexafluorophosphate (164 mg, 10 equiv.) and trifluoroacetic acid (14.5 μL, 2 equiv.) with a Ni foam counterelectrode instead of Zn, gave product 4b (62% yield) as a slightly yellow solid. Eluent: cyclohexane: ethyl acetate from 100:0 to 95:5.
$^1$H-NMR: $\delta$ 7.17 – 7.07 (m, 4H), 6.89 (td, $J$ = 7.4, 1.0 Hz, 2H), 6.73 (d, $J$ = 7.9 Hz, 2H), 6.03 (s, 1H), 5.71 (ddt, $J$ = 17.1, 10.1, 6.9 Hz, 1H), 4.87 (dd, $J$ = 19.1, 17.1 Hz, 1H), 4.03 (t, $J$ = 6.9 Hz, 1H), 2.36 (t, $J$ = 6.9 Hz, 2H) ppm.

$^{13}C$-NMR: $\delta$ 139.8 (2C), 135.9, 129.0 (2C), 127.1 (2C), 123.8 (2C), 120.8 (2C), 117.0, 113.6 (2C), 44.4, 43.1 ppm.

HRMS (ESI$^+$): calculated for C$_{16}$H$_{16}$N [M-H]$^+$: 222.1277; found: 222.1237.

9-Benzyl-9,10-dihydroacridine (4c)

Following a slightly modified procedure B; acridine 1v (17.9 mg, 0.1 mmol) and (bromomethyl)benzene 2k (59.5 μL, 0.5 mmol) in Me-THF:H$_2$O (2:1) gave product 4c (81% yield) as a white solid. Eluent: cyclohexane: ethyl acetate from 100:0 to 90:10.

$^1$H-NMR: $\delta$ 7.18 – 7.14 (m, 3H), 7.13 – 7.07 (m, 2H), 6.87 (dd, $J$ = 7.4, 1.2 Hz, 2H), 6.84 – 6.75 (m, 4H), 6.70 (d, $J$ = 7.9 Hz, 2H), 5.99 (s, 1H), 4.18 (t, $J$ = 7.0 Hz, 1H), 2.85 (d, $J$ = 7.0 Hz, 2H) ppm.

$^{13}C$-NMR: $\delta$ 139.9 (2C), 139.2, 130.1 (2C), 129.1 (2C), 127.9 (2C), 127.1 (2C), 126.1, 123.5 (2C), 120.6 (2C), 113.5 (2C), 46.2, 45.4 ppm.

HRMS (ESI$^+$): calculated for C$_{20}$H$_{18}$N [M-H]$^+$: 272.1434; found: 272.1494.

9-(4-Chlorobenzyl)-9,10-dihydroacridine (4d)

Following a slightly modified procedure B; acridine 1v (17.9 mg, 0.1 mmol) and 1-(bromomethyl)-4-chlorobenzene 2l (102.7 mg, 0.5 mmol) in Me-THF:H$_2$O (2:1) gave product 4d (85% yield) as a white solid. Eluent: cyclohexane: ethyl acetate from 100:0 to 95:5.

$^1$H-NMR: $\delta$ 7.15 – 7.00 (m, 4H), 6.91 (dd, $J$ = 7.4, 1.3 Hz, 2H), 6.83 (td, $J$ = 7.4, 1.1 Hz, 2H), 6.75 – 6.60 (m, 4H), 5.94 (s, 1H), 4.19 (t, $J$ = 6.7 Hz, 1H), 2.82 (d, $J$ = 6.7 Hz, 1H) ppm.

$^{13}C$-NMR: $\delta$ 139.9 (2C), 137.5, 132.0, 131.4 (2C), 129.0 (2C), 128.0 (2C), 127.3 (2C), 123.0 (2C), 120.7 (2C), 113.5 (2C), 45.6, 45.1 ppm.

HRMS (ESI$^+$): calculated for C$_{20}$H$_{17}$ClN [M-H]$^+$: 306.1044; found: 306.1045.
9-(2-Iodobenzyl)-9,10-dihydroacridine (4e)

Following a slightly modified procedure B; acridine \( \text{1v} \) (17.9 mg, 0.1 mmol) and 1-(bromomethyl)-2-iodobenzene \( \text{2m} \) (148.5 mg, 0.5 mmol) in MeTHF:H\( \text{2O} \) (2:1) gave product \( \text{4e} \) (65% yield) as a yellowish solid. Eluent: cyclohexane: ethyl acetate from 100:0 to 80:20.

\( ^1\text{H-NMR} \): \( \delta \) 7.84 (dd, \( J = 7.9, 1.2 \text{ Hz} \), 1H), 7.16 – 7.09 (m, 2H), 7.05 (td, \( J = 7.6, 1.7 \text{ Hz} \), 1H), 6.16 (s, 1H), 4.32 (t, \( J = 7.6 \text{ Hz} \), 1H), 2.94 (d, \( J = 7.6 \text{ Hz} \), 1H) ppm.

\( ^{13}\text{C-NMR} \): \( \delta \) 141.7, 140.0 (2C), 139.4, 132.1, 129.2 (2C), 128.1, 127.7, 127.3 (2C), 123.2 (2C), 120.7 (2C), 113.6 (2C), 101.4, 49.4, 43.1 ppm.

HRMS (ESI\(^+\)): calculated for \( \text{C}_{20}\text{H}_{17}\text{IN} [\text{M-H}]^+ \): 398.0400; found: 396.0370.

9-(4-(Trifluoromethyl)benzyl)-9,10-dihydroacridine (4f)

Following a slightly modified procedure B; acridine \( \text{1v} \) (17.9 mg, 0.1 mmol) and 1-(bromomethyl)-4-(trifluoromethyl)benzene \( \text{2n} \) (119.5 mg, 0.5 mmol) in MeTHF:H\( \text{2O} \) (2:1) gave product \( \text{4f} \) (53% yield) as a slightly yellow solid. Eluent: cyclohexane: ethyl acetate from 100:0 to 95:5.

\( ^1\text{H-NMR} \): \( \delta \) 7.37 (d, \( J = 8.0 \text{ Hz} \), 2H), 7.11 (td, \( J = 7.9, 1.7 \text{ Hz} \), 2H), 6.92 – 6.79 (m, 6H), 6.69 (d, \( J = 7.9 \text{ Hz} \), 2H), 5.95 (s, 1H), 4.23 (t, \( J = 6.7 \text{ Hz} \), 1H), 2.89 (d, \( J = 6.7 \text{ Hz} \), 2H) ppm.

\( ^{13}\text{C-NMR} \): \( \delta \) 143.2, 139.9 (2C), 130.3 (2C), 129.0 (2C), 128.6 (d, \( J = 9.0 \text{ Hz} \)), 127.4 (2C), 127.3 (q, \( J = 137.1 \text{ Hz} \)), 124.7 (q, \( J = 3.8 \text{ Hz} \), 2C), 122.8 (2C), 120.8 (2C), 113.6 (2C), 46.0, 45.0 ppm.

\( ^{19}\text{F-NMR} \): \( \delta \) -62.1 ppm.

HRMS (ESI\(^+\)): calculated for \( \text{C}_{21}\text{H}_{17}\text{F}_3\text{N} [\text{M-H}]^+ \): 340.1308; found: 340.1263.

9-(3,5-Dimethoxybenzyl)-9,10-dihydroacridine (4g)

Following a slightly modified procedure B; acridine \( \text{1v} \) (17.9 mg, 0.1 mmol) and 1-(bromomethyl)-3,5-dimethoxybenzene \( \text{2o} \) (115.5 mg, 0.5 mmol) in MeTHF:H\( \text{2O} \) (2:1) gave product \( \text{4g} \) (75% yield) as a white solid. Eluent: cyclohexane: ethyl acetate from 100:0 to 95:5.

\( ^1\text{H-NMR} \): \( \delta \) 7.10 (td, \( J = 7.6, 1.5 \text{ Hz} \), 2H), 6.94 (d, \( J = 7.4 \text{ Hz} \), 2H), 6.83 (td, \( J = 7.4, 1.1 \text{ Hz} \), 2H), 6.69 (d, \( J = 7.9 \text{ Hz} \), 2H), 6.39 – 6.26 (m, 1H), 6.00 (s, 1H), 5.90 (d, \( J = 2.2 \text{ Hz} \), 2H), 4.19 (t, \( J = 6.8 \text{ Hz} \), 1H), 3.62 (s, 6H), 2.78 (d, \( J = 6.8 \text{ Hz} \), 2H) ppm.
$^{13}$C-NMR: δ 160.3 (2C), 141.3, 139.9 (2C), 129.1 (2C), 127.2 (2C), 123.4 (2C), 120.7 (2C), 113.5 (2C), 107.8 (2C), 106.7, 99.0, 55.3 (2C), 46.5, 45.2 ppm.

HRMS (ESI$^+$): calculated for C$_{22}$H$_{22}$N$_2$O$_2$ [M-H]$^+$: 332.1645; found: 332.1673.

9-(Benzo[d][1,3]dioxol-5-ylmethyl)-9,10-dihydroacridine (4h)

Following a slightly modified procedure B; acridine 1v (17.9 mg, 0.1 mmol) and 5-(bromomethyl)benzo[d][1,3]dioxole 2p (107.5 mg, 0.5 mmol) in Me-THF:H$_2$O (2:1) gave product 4h (79% yield) as a white solid. Eluent: cyclohexane: ethyl acetate from 100:0 to 95:5.

$^1$H-NMR: δ 7.10 (t, $J$ = 7.5 Hz, 2H), 6.90 (d, $J$ = 7.5 Hz, 2H), 6.82 (t, $J$ = 7.5 Hz, 2H), 6.70 (d, $J$ = 7.5 Hz, 2H), 6.60 (d, $J$ = 7.8 Hz, 1H), 6.30 (s, 1H), 6.20 (d, $J$ = 7.8 Hz, 1H), 6.00 (s, 1H), 5.90 (s, 2H), 4.13 (t, $J$ = 6.9 Hz, 1H), 2.75 (d, $J$ = 6.9 Hz, 2H) ppm.

$^{13}$C-NMR: δ 147.2, 145.9, 139.8 (2C), 133.0, 129.1 (2C), 127.2 (2C), 123.4 (2C), 123.0, 120.7 (2C), 113.5 (2C), 110.4, 107.8, 100.8, 45.9, 45.5 ppm.

HRMS (ESI$^+$): calculated for C$_{21}$H$_{18}$N$_2$O$_2$ [M-H]$^+$: 316.1332; found: 316.1269.
5. General procedure C: Derivatizations

6-Bromo-2-isopropylquinoline-4-carboxylic acid (7)

![Chemical Structure](image)

1M lithium hydroxide aqueous solution (0.3 mL) was added to a vial containing methyl 6-bromo-2-isopropylquinoline-4-carboxylate (6, 6 mg, 0.05 mmol) in methanol (0.5 mL). The reaction was stirred at room temperature for two hours. Then 1M HCl aqueous solution was added until pH= 1. The crude was extracted with EtOAc (2 x 5 mL). Organic layer was dried over anhydrous Na$_2$SO$_4$, filtered and concentrated. Crude was purified by flash column chromatography using cyclohexane: ethyl acetate/methanol/acetic acid (3/1/2%) from 100:0 to 80:20. 6-Bromo-2-isopropylquinoline-4-carboxylic acid (7, 80% yield) was obtained as a colorless oil.

$^1$H-NMR (CDCl$_3$ + few drops CD$_3$OD): $\delta$ 9.01 (d, $J = 1.9$ Hz, 1H), 8.05 – 7.90 (m, 2H), 7.79 (dd, $J = 9.0$, 2.0 Hz, 1H), 3.41 – 3.22 (m, 1H), 1.40 (d, $J = 6.9$ Hz, 6H) ppm.

$^{13}$C-NMR (CDCl$_3$ + few drops CD$_3$OD): $\delta$ 167.9 (2C), 147.3, 133.1 (2C), 130.9, 128.2, 125.3, 122.0, 121.6, 37.2, 22.3 (2C) ppm.

HRMS (ESI$^+$): calculated for C$_{13}$H$_{13}$BrN$_2$O$_2$ [M-H]$^-$: 293.0046; found: 293.0090.

9-(Benzo[d][1,3]dioxol-5-ylmethyl)acridine (5)

![Chemical Structure](image)

MnO$_2$ (55 mg, 10 equiv.) was added to a vial containing 9-(Benzo[d][1,3]dioxol-5-ylmethyl)-9,10-dihydroacridine (4h, 20 mg, 0.06 mmol) in tetrahydrofuran (1.0 mL). The reaction was stirred at room temperature 16 h. Crude was purified by flash column chromatography using cyclohexane: ethyl acetate from 100:0 to 95:5 to 90:10. 9-(Benzo[d][1,3]dioxol-5-ylmethyl)acridine (5, 85% yield) was obtained as a white solid.

$^1$H-NMR: $\delta$ 8.31 – 8.18 (m, 4H), 7.84 – 7.71 (m, 2H), 7.59 – 7.48 (m, 2H), 6.69 – 6.63 (m, 1H), 6.62 – 6.54 (m, 2H), 5.86 (s, 2H), 4.91 (s, 2H) ppm.

S22
$^{13}$C-NMR: $\delta$ 149.1 (2C), 148.1, 146.3, 143.6, 133.4, 130.6 (2C), 130.0 (2C), 126.3 (2C), 125.8 (2C), 124.8 (2C), 121.3, 108.8, 108.6, 101.1, 32.9 ppm.

HRMS (ESI$^+$): calculated for C$_{21}$H$_{16}$NO$_2$ [M-H]$^+$: 314.1176; found: 314.1157.
6. Nuclear magnetic resonance spectra

2-Cyclohexyl-4-methylquinoline (3a)

2-Isopropyl-4-methylquinoline (3b)
4-Methyl-2-(tetrahydro-2H-pyran-4-yl)quinoline (3c)

tert-Butyl 4-(4-methylquinolin-2-yl)piperidine-1-carboxylate (3d)
2-Cyclopentyl-4-methylquinoline (3e)

4-Methyl-2-(tetrahydrofuran-3-yl)quinoline (3f)
2-\((\text{tert-Butyl})\)-4-methylquinoline (3g)

2-Ethyl-4-methylquinoline (3h)
4-Methyl-2-phenethylquinoline (3i)
2-Cyclohexyl-7-methoxy-4-methylquinoline (3j)
7-Bromo-2-cyclohexyl-4-methylquinoline (3k)
6-Bromo-2-cyclohexyl-4-methylquinoline (3I)
2-Cyclohexyl-4-phenylquinoline (3m)
2-(2-Cyclohexylquinolin-4-yl)-1-(4-(trifluoromethyl)phenyl)ethan-1-one (3n)
4-Bromo-2-cyclohexylquinoline (3o)
Methyl 6-bromo-2-cyclohexylquinoline-4-carboxylate (3p)
2-Cyclohexyl-N-methylquinoline-4-carboxamide (3q)
4-Cyclohexyl-2-methylquinoline (3r)
6-Chloro-4-cyclohexyl-2-methylquinoline (3s)
Methyl 4-cyclohexyl-2-methylquinoline-6-carboxylate (3t)
2-Cyclohexylquinoline (3u)

4-Cyclohexylquinoline (3u')
1-Cyclohexyl-3-methylisoquinolin-4-ol (3v)
2-Cyclohexyl-4,7-diphenyl-1,10-phenanthroline (3w)

6-Cyclohexylphenanthridine (3x)
2-Cyclohexylbenzodthiazole (3y)

2-Cyclohexyl-1-methyl-1H-benzo[d]imidazole (3z)
5,7-Dichloro-2-cyclohexyl-4-(4-fluorophenoxy)quinoline (3aa)

(R)-(2-Cyclohexyl-6-methoxyquinolin-4-yl)(1S,2S,4S,5R)-5-vinylquinuclidin-2-yl)methanol (3ab)
(R)-(2-Cyclohexylquinolin-4-yl)((15,25,45,5R)-5-vinylquinuclidin-2-yl)methanol (3ac)
(S)-11-Cyclohexyl-4-ethyl-4-hydroxy-1,12-dihydro-14H-pyrano[3',4':6,7]indolizino[1,2-b]quinoline-3,14(4H)-dione (3ad)
Methyl 6-bromo-2-isopropylquinoline-4-carboxylate (6)
Methyl 2-cyclohexylquinoline-4-carboxylate (8)

2-Cyclohexylquinoline-4-carboxylic acid (9)
3-Cyclohexyl-N,2-diphenylpropanamide (11)
9-(tert-Butyl)-9,10-dihydroacridine (4a)
9-Allyl-9,10-dihydroacridine (4b)
9-Benzyl-9,10-dihydroacridine (4c)
9-(4-Chlorobenzyl)-9,10-dihydroacridine (4d)
9-(2-Iodobenzyl)-9,10-dihydroacridine (4e)
9-(4-(Trifluoromethyl)benzyl)-9,10-dihydroacridine (4f)
9-(3,5-Dimethoxybenzyl)-9,10-dihydroacridine (4g)
9-(Benzo[d][1,3]dioxol-5-ylmethyl)-9,10-dihydroacridine (4h)
6-Bromo-2-isopropylquinoline-4-carboxylic acid (7) NMR Solvents: CDCl$_3$ + few drops CD$_2$OD
9-(Benzo[d][1,3]dioxol-5-ylmethyl)acridine (5)
7. Cyclic Voltammetry

CVs were performed under argon atmosphere at room temperature, using 0.25 M tetrabutylammonium hexafluorophosphate (TBAPF₆) solution in acetonitrile (CH₃CN) as electrolyte. Measurements were carried out by using an Ivium CompaqStat potentiostat interfaced with a computer. A standard three-electrode electrochemical cell was used. Potentials were referred to an Ag/AgCl, TBAPF₆ 0.4 M reference electrode in ethylene glycol, and measured potentials were calibrated using an internal Fc/Fc⁺ standard. The working electrode used to perform the experiments was a glassy carbon electrode. The counterelectrode consisted of a Pt electrode immersed in a conductive solution.

Figure S1. Cyclic voltammetry of 4-methylquinoline (1a) (blue) vs 4-methylquinoline (1a) activated with 1 equivalent of diphenyl phosphate (black). They were measured in CH₃CN (0.25 M TBAPF₆) at 100 mV/s using glassy carbon electrode as WE, Ag/AgCl as RE and Pt bar as CE.
Figure S2. Cyclic voltammogram of iodoctohexane (2a) under argon (orange) vs iodoctohexane (2a) with the oxygen from the solvent (purple). They were measured in CH$_3$CN (0.25 M TBAPF$_6$) at 100 mV/s using glassy carbon electrode as WE, Ag/AgCl as RE and Pt bar as CE.
8. References

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