Synthetic and Mechanistic Aspects of the Regioselective
Base-Mediated Reaction of Perfluoroalkyl- and
Perfluoroarylsilanes with Heterocyclic N-Oxides

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General Procedures

Materials and methods: Tetrahydrofuran was distilled from sodium benzophenone
ketyl. Isoquinoline-N-oxide was purchased from Alfa Aesar, 4-phenylpyridine N-oxide,
(pentafluorophenyl)trimethylsilane, and (pentafluoroethyl)trimethylsilane were
purchased from TCI. (Trifluoromethyl)trimethylsilane was purchased from Matrix
Scientific, and (difluoromethyl)trimethylsilane was purchased from Oakwood Chemicals.
All other chemicals were used as commercially available (Sigma-Aldrich, Acros, Alfa
Aesar, Combi-Blocks, Strem). All reactions were conducted with continuous magnetic
stirring under an atmosphere of argon in oven-dried glassware. Low-temperature
experiments were conducted using a Neslab Cryotrol CB-80 cryostat. Reactions were
monitored by TLC until deemed complete using silica gel-coated glass plates (Merck
Kieselgel 60 F254). Plates were visualized under ultraviolet light (254 nm).

Purification: Column chromatography was performed using CombiFlash Rf-200
(Teledyne-Isco) automated flash chromatography system with self-packed RediSep
columns.

Characterization: $^1$H, $^{13}$C, $^{19}$F NMR spectra were recorded at 500 and 300 MHz ($^1$H),
125 and 75 MHz ($^{13}$C), and 282 MHz ($^{19}$F) on Varian Mercury VX 300 and Agilent
Inova 500 instruments in CDCl$_3$ solutions. Chemical shifts ($\delta$) are reported in parts per
million (ppm) from the residual solvent peak and coupling constants ($J$) in Hz. Proton
multiplicity is assigned using the following abbreviations: singlet (s), doublet (d), triplet (t), quartet (quart.), quintet (quint.), septet (sept.), multiplet (m), broad (br).

Infrared measurements were carried out neat on a Bruker Vector 22 FT-IR spectrometer fitted with a Specac diamond attenuated total reflectance (ATR) module.

**5-Bromoquinoline 1-oxide** (S1)

To a stirred solution of 5-bromoquinoline (400 mg, 1.94 mmol) in chloroform (10 mL) was added *meta*-chloroperoxybenzoic acid (620 mg, 2.52 mmol, 1.3 equiv., 70 % in H₂O). After 12 h the reaction was diluted with a saturated aqueous solution of sodium thiosulfate/sodium carbonate (20 mL, 1:1). After separating the layers the aqueous layer was extracted with dichloromethane (3 x 10 mL), the organic layers combined, dried over anhydrous sodium sulfate, and concentrated under reduced pressure to yield S1 (332 mg, 77 %) as tan solid. – m.p.: 65–67 °C. – ¹H NMR (500 MHz): 7.41 (1 H, dd, J = 6, 8 Hz), 7.61 (1 H, dd, J = 7.5, 8.5 Hz), 7.94 (1 H, dd, J = 1, 7.5 Hz), 8.12 (1 H, d, J = 8.5 Hz), 8.56 (1 H, dd, J = 0.5, 6 Hz), 8.77 (1 H, d, J = 8.5 Hz) ppm. – ¹³C NMR (125 MHz): 119.5, 121.8, 122.3, 125.9, 129.7, 130.5, 132.7, 136.2, 142.1 ppm. – IR: 1056, 1142, 1195, 1255, 1289, 1394, 1443, 1505, 1663, 2999, 3067, 3107 cm⁻¹.

**4-(tert-Butylthio)-7-chloroquinoline 1-oxide** (S2)

To a stirred solution of S3 (200 mg, 0.930 mmol) in ethanol (5 mL) was added sodium 2-methyl-2-propanethiolate (136 mg, 1.21 mmol, 1.3 equiv.) and the reaction heated to 50 °C for 12 h. The reaction was concentrated on Celite and purified by column chromatography [hexanes/EtOAc/Si₂O] to yield S2 (151 mg, 61 %) as colorless solid. – m.p.: 125 – 127
°C. – \(^1\)H NMR (500 MHz): 1.32 (9 H, s), 7.52 (1 H, d, \(J = 7\) Hz), 7.62 (1 H, dd, \(J = 2.5, 10\) Hz), 8.46 (1 H, d, \(J = 6.5\) Hz), 8.55 (1 H, d, \(J = 10\) Hz), 8.75 (1 H, d, \(J = 2.5\) Hz) ppm. – \(^{13}\)C NMR (125 MHz): 31.2, 49.4, 119.4, 128.2, 129.5, 129.9, 130.4, 130.6, 132.0, 135.2, 137.2, 142.1 ppm. – IR: 1134, 1183, 1242, 1345, 1459, 2900, 2971, 3097 cm\(^{-1}\). – MS (ESI): 267.9, HRMS: 268.0140, calcd: 268.0557 [M+H\(^+\)].

**4,7-Dichloroquinoline 1-oxide\(^2\) (S3)**

S3 was prepared according to literature procedure. – m.p.: 164–165 °C\(^3\)

– \(^1\)H NMR (500 MHz): 7.36 (1 H, d, \(J = 6.5\) Hz), 7.68 (1 H, d, \(J = 9\) Hz), 8.13 (1 H, d, \(J = 9\) Hz), 8.43 (1 H, d, \(J = 6.5\) Hz), 8.77 (1 H, s) ppm. – \(^{13}\)C NMR (125 MHz): 119.93, 121.22, 121.42, 125.60, 126.52, 126.75, 128.68, 129.85, 130.80, 135.96, 138.22, 142.33, 150.88 ppm. – IR: 829, 1091, 1291, 1367, 1412, 1555, 1609, 3025, 3094 cm\(^{-1}\).

**7-Chloro-4-(isopropylthio)quinoline 1-oxide (S4)**

To a stirred solution of S3 (200 mg, 0.930 mmol) in ethanol (5 mL) was added sodium 2-propanethiolate (118 mg, 1.21 mmol, 1.3 equiv.) and the reaction heated to 50 °C for 12 h. The reaction was concentrated on Celite and purified by column chromatography [hexanes/EtOAc/SiO\(_2\)] to yield S4 (168 mg, 71 %) as yellow solid. – m.p.: 50–53 °C. – \(^1\)H NMR (500 MHz): 1.41 (6 H, d, \(J = 6.5\) Hz), 3.57 (1 H, sept., \(J = 6.5\) Hz), 7.27 (1 H, s), 7.63 (1 H, dd, \(J = 2, 9\) Hz), 8.26 (1 H, d, \(J = 9\) Hz), 8.44 (1 H, d, \(J = 6.5\) Hz), 8.81 (1 H, d, \(J = 2\) Hz) ppm. – \(^{13}\)C NMR (125 MHz): 22.8, 38.0, 119.7, 121.0, 127.0, 128.2, 129.6,
134.8, 135.4, 137.3, 141.3 ppm. – IR: 1158, 1182, 1213, 1345, 1364, 1441, 1573, 2869, 2967, 3099 cm\(^{-1}\). – MS (ESI): 253.9, HRMS: 253.8814, calcd: 254.0401 [M+H\(^+\)].

8-Methoxyquinoline\(^4\) (S5)

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S5 was prepared according to literature procedure.\(^4\) – \(^1\)H NMR (300 MHz): 4.06 (3 H, s), 7.05 (1 H, td, \(J = 1, 7.5\) Hz), 7.36–7.50 (3 H, m), 8.19 (1 H, td, \(J = 1, 7.5\) Hz), 8.96 (1 H, td, \(J = 1, 7.5\) Hz) ppm. – \(^{13}\)H NMR (75 MHz): 56.03, 108.18, 119.52, 121.73, 127.25, 129.34, 137.24, 138.66, 148.45, 154.58 ppm. – IR: 1076, 1219, 1440, 1501, 1615, 2838, 2938, 3054 cm\(^{-1}\).

8-Methoxyquinoline 1-oxide\(^2\) (S6)

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\begin{array}{c}
\text{S6} \\
\includegraphics[width=0.2\textwidth]{s6.png}
\end{array}
\]

To a stirred solution of S5 (340 mg, 2.12 mmol) in acetonitrile (1 mL) was added hydrogen peroxide (313 \(\mu\)L, 3.18 mmol, 1.5 equiv., 30% in \(\text{H}_2\text{O}\)) and phosphomolybdic acid (197 \(\mu\)L, 0.21 mmol, 1 mol %, 20% in \(\text{EtOH}\)) followed by heating the reaction to 50 °C. After 12 h the reaction was diluted with a saturate aqueous solution of ammonium chloride (2 mL) and the aqueous layer extracted with dichloromethane (3 x 5 mL). The combined organic layers were dried over anhydrous sodium sulfate, concentrated under reduced pressure, and purified by column chromatography to yield S6 (260 mg, 71%) as brown solid. – m.p.: 38–41 °C – \(^1\)H NMR (300 MHz): 3.92 (3 H, s), 6.88 (1 H, dd, \(J = 1.5, 8\) Hz), 7.20–7.40 (3 H, m), 7.96 (1 H, dd, \(J = 1.5, 8\) Hz), 8.78 (1 H, dd, \(J = 1.5, 4\) Hz) ppm. – \(^{13}\)C NMR (75 MHz): 55.84, 107.57, 119.42, 121.57, 126.72, 129.19, 136.07, 139.66, 148.89, 155.02 ppm. – IR: 910, 1090, 1232, 1378, 1467, 1504, 2858, 2954, 3037 cm\(^{-1}\).
7-Chloro-4-phenylquinolinene\(^5\) (S7)

\(\text{S7} \) was prepared according to a literature procedure.\(^5\) – \(^1\text{H NMR} \) (500 MHz): 7.30 (1 H, d, \(J = 4 \) Hz), 7.41 – 7.53 (6 H, m), 7.84 (1 H, d, \(J = 9 \) Hz), 8.19 (1 H, d, \(J = 1 \) Hz), 8.93 (1 H, d, \(J = 4 \) Hz) ppm. – \(^{13}\text{C NMR} \) (125 MHz): 121.4, 125.0, 127.3, 127.4, 128.3, 128.7, 128.8, 129.4, 135.1, 137.4, 148.3, 149.1, 150.9 ppm. – IR: 1071, 1167, 1271, 1304, 1374, 1417, 1488, 1573, 2834, 2877, 3031 \(\text{cm}^{-1}\).

5,7-Dichloro-8-methoxyquinoline\(^6\) (S8)

\(\text{S8} \) was prepared according to a literature procedure.\(^7\) – m.p.: 84 – 86 °C.

– \(^1\text{H NMR} \) (500 MHz): 4.19 (3 H, s), 7.55 (1 H, dd, \(J = 4.5, 9 \) Hz), 7.67 (1 H, s), 8.54 (1 H, dd, \(J = 1, 9 \) Hz), 9.02 (1 H, dd, \(J = 1.5, 4.5 \) Hz) ppm.

– \(^{13}\text{C NMR} \) (125 MHz): 62.2, 121.9, 126.0, 126.1, 126.4, 127.6, 133.1, 143.4, 150.8, 151.4 ppm. – IR: 1112, 1190, 1246, 1353, 1385, 1402, 1488, 2848, 2942, 3068 \(\text{cm}^{-1}\).

5-Chloro-8-methoxyquinoline\(^8\) (S9)

\(\text{S9} \) was prepared according to a literature procedure.\(^7\) – \(^1\text{H NMR} \) (500 MHz): 4.05 (3 H, s), 6.90 (1 H, d, \(J = 8.5 \) Hz), 7.46 (1 H, d, \(J = 8.5 \) Hz), 7.50 (1 H, dd, \(J = 4, 8.5 \) Hz), 8.46 (1 H, dd, \(J = 1, 8.5 \) Hz), 8.95 (1 H, dd, \(J = 1, 4 \) Hz) ppm. – \(^{13}\text{C NMR} \) (125 MHz): 55.6, 106.9, 121.5, 125.9, 126.3, 127.9, 132.2, 140.0, 149.1, 154.1 ppm. – IR: 1100, 1159, 1252, 1269, 1307, 1385, 1440, 1502, 2841, 2956, 3035 \(\text{cm}^{-1}\).
7-Chloro-4-phenylquinoline 1-oxide (S10)

To a stirred solution of S7 (400 mg, 1.67 mmol) in dichloromethane (5 mL) was added *meta*-chloroperoxybenzoic acid (748 mg, 2.17 mmol, 1.3 equiv., 50 % solution in H₂O) at 0 °C. After 12 h the reaction was diluted with a saturated aqueous solution of sodium thiosulfate/sodium carbonate (30 mL, 1:1), and the aqueous layer extracted with dichloromethane (4 x 10 mL). The combined organic layers were dried over anhydrous sodium sulfate, concentrated under reduced pressure, and purified by column chromatography to yield S10 (472 mg, 85 %) as tan solid. – m.p.: 112 – 114 °C. – ¹H NMR (500 MHz): 7.23 (1 H, d, J = 6 Hz), 7.45 – 7.55 (6 H, m), 7.88 (1 H, d, J = 9 Hz), 8.55 (1 H, d, J = 6 Hz), 8.85 (1 H, d, J = 2 Hz) ppm. – ¹³C NMR (125 MHz): 119.4, 121.5, 127.1, 128.2, 128.3, 128.8, 128.9, 129.4, 129.6, 135.6, 136.3, 136.8, 138.4, 141.6 ppm. – IR: 1001, 1083, 1152, 1208, 1301, 1373, 1442, 1551, 2992, 3032, 3103 cm⁻¹. – MS (ESI): 255.9, HRMS: 256.0550, calcd: 256.0524 [M+H⁺].

5-Chloro-8-methoxyquinoline 1-oxide (S11)

To a stirred solution of S9 (500 mg, 2.59 mmol) in dichloromethane (5 mL) was added *meta*-chloroperoxybenzoic acid (1.16 g, 3.36 mmol, 1.3 equiv., 50 % solution in H₂O) at 0 °C. After 12 h the reaction was diluted with a saturated aqueous solution of sodium thiosulfate/sodium carbonate (30 mL, 1:1), and the aqueous layer extracted with dichloromethane (4 x 10 mL). The combined organic layers were dried over anhydrous sodium sulfate, concentrated under reduced pressure, and purified by column chromatography to yield S11 (501 mg, 92 %) as brown
oil. – \(^1\)H NMR (300 MHz): 3.98 (3 H, s), 6.95 (1 H, d, \(J = 5\) Hz), 7.27–7.32 (1 H, m), 7.52 (1 H, d, \(J = 5\) Hz), 8.00 (1 H, d, \(J = 9.5\) Hz), 8.42 (1 H, d, \(J = 7\) Hz) ppm. – \(^{13}\)C NMR (125 MHz): 57.1, 110.5, 122.0, 122.2, 122.9, 128.7, 130.9, 134.8, 138.5, 152.8 ppm. – IR: 1092, 1160, 1264, 1342, 1397, 1464, 2838, 2887, 3015 cm\(^{-1}\). – MS (ESI): 210.0, HRMS: 210.0376, calcd: 210.0316 \([\text{M+H}^+]\).

**Phenanthridine 5-oxide\(^2\) (S12)**

According to GP1, phenanthridine (50 mg, 0.279 mmol) was reacted with hydrogen peroxide (83 \(\mu\)L, 0.837 mmol, 3 equiv., 30% in H\(_2\)O) and phosphomolybdic acid (25 \(\mu\)L, 0.005 mmol, 2 mol%, 20% in EtOH) in acetonitrile (200 \(\mu\)L). The isolated product afforded S12 (41 mg, 76%) as brown oil. – \(^1\)H NMR (500 MHz): 7.25–8.09 (5 H, m), 8.53–8.63 (2 H, m), 8.97 (1 H, d, \(J = 2\) Hz), 9.19 (1 H, d, \(J = 2\) Hz) ppm. – \(^{13}\)C NMR (125 MHz): 120.67, 122.13, 122.77, 127.06, 128.18, 128.99, 129.53, 129.71, 129.91, 130.15, 130.46, 133.83, 169.27 ppm. – IR: 1071, 1191, 1473, 1559, 1647, 3071 cm\(^{-1}\).

**7-Chloro-4-methoxyquinoline\(^9\) (S13)**

To a stirred solution of 4,7-dichloroquinoline (5 g, 25.51 mmol) in methanol (50 mL) was added sodium methoxide (6.88 g, 127.55 mmol, 5 equiv.). The reaction was heated at 95 °C for 12 h, then concentrated under reduced pressure, diluted with EtOAc (30 mL) and washed with H\(_2\)O (2 x 20 mL). The organic layer was dried over anhydrous sodium sulfate and concentrated under reduced pressure to yield S13 (4.78 g, 97 %) as colorless solid. – m.p.: 145–148 °C\(^10\) –
$^1$H NMR (300 MHz): 3.19 (3 H, s), 6.58 (1 H, d, $J = 5.5$ Hz), 7.32 (1 H, dd, $J = 2$, 9 Hz), 7.92 (1 H, d, $J = 2$ Hz), 7.98 (1 H, d, $J = 9$ Hz), 8.62 (1 H, d, $J = 5$ Hz) ppm. – $^{13}$C NMR (75 MHz): 55.71, 100.26, 119.66, 123.36, 126.34, 127.64, 135.54, 149.45, 152.42, 162.16 ppm. – IR: 982, 1070, 1209, 1360, 1425, 1503, 1616, 2984, 3050 cm$^{-1}$.

7-Chloro-4-methoxyquinoline 1-oxide$^{11}$ (S14)

To a stirred solution of S13 (1.6 g, 5.54 mmol) in acetonitrile (2.5 mL) were added hydrogen peroxide (1.7 mL, 16.62 mmol, 3 equiv., 30% in H$_2$O) and phosphomolybdic acid (1 mL, 0.118 mmol, 2 mol%, 20% in EtOH) then heated to 50 °C. After 12 h the reaction was diluted with a saturated aqueous solution of ammonium chloride (10 mL), and the aqueous layer extracted with dichloromethane (4 x 15 mL). The organic layers were combined, dried over anhydrous sodium sulfate, concentrated under reduced pressure, and purified by column chromatography to yield S14 (1.12 g, 97%) as colorless solid. – m.p.: 145–147 °C – $^1$H NMR (500 MHz): 4.06 (3 H, s), 6.64 (1 H, d, $J = 7$ Hz), 7.59 (1 H, d, $J = 2$ Hz), 8.15 (1 H, d, $J = 9$ Hz), 8.46 (1 H, d, $J = 7$ Hz), 8.77 (1 H, d, $J = 2$ Hz) ppm. – $^{13}$C NMR (125 MHz): 56.34, 99.84, 119.57, 120.99, 123.39, 124.28, 129.06, 136.99, 137.88, 154.26 ppm. – IR: 1110, 1243, 1325, 1445, 2988, 3025 cm$^{-1}$.

Quinoline 1-oxide$^{2}$ (2)

According to GP1, quinoline (5 g, 40.65 mmol) was reacted with hydrogen peroxide (6.1 mL, 60.97 mmol, 1.5 equiv., 30% in H$_2$O) and phosphomolybdic acid (3.7 mL, 0.406 mmol, 1 mol%, 20% in EtOH) in acetonitrile (20
mL). The crude product was purified by column chromatography to afford 2 (4.95 g, 88%) as brown solid. – m.p.: 60–62 °C\textsuperscript{12} – \textsuperscript{1}H NMR (300 MHz): 7.29 (1 H, dd, \( J = 6, 8 \) Hz), 7.64 (1 H, dt, \( J = 1, 7 \) Hz), 7.72–7.79 (2 H, m), 7.86 (1 H, dd, \( J = 1.5, 8 \) Hz), 8.52 (1 H, dd, \( J = 1, 6 \) Hz) ppm. – \textsuperscript{13}C NMR: 119.81, 120.96, 125.87, 128.12, 128.76, 130.41, 130.50, 135.60 ppm. – IR: 1157, 1311, 1452, 1554, 2931, 2995, 3025 cm\textsuperscript{−1}.
Crystal Structure of 4-(tert-Butylthio)-7-chloroquinoline 1-oxide (S2)

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References

7-Chloro-4-phenylnoline 1-oxide (S10)
5-Chloro-8-methoxyquinoline 1-oxide (S11)
8-Methoxy-2-(trifluoromethyl)quinoline (5)
7-Chloro-4-methoxy-2-(trifluoromethyl)quinoline (6)
5-Bromo-2-(trifluoromethyl)quinoline (7)
7-Chloro-4-phenyl-2-(trifluoromethyl)quinoline (9)

ppm (1H)
7-Chloro-4-(isopropylthio)-2-(trifluoromethyl)quinoline (12)

7-Chloro-4-(isopropylthio)-2-(trifluoromethyl)quinoline (12)
7-Chloro-4-(isopropylthio)-2-(perfluoropropyl)quinoline (14)
4-(tert-Butylthio)-7-chloro-2-(perfluoropropyl)quinoline (15)
5-Bromo-2-(perfluoroethyl)quinoline (16)
8-Methoxy-2-(perfluoroethyl)quinoline (18)
8-(tert-Butoxy)-5,7-dichloro-2-(trifluoromethyl)quinoline (28)
8-(tert-Butoxy)-5,7-dichloro-2-(perfluoroethyl)quinoline (29)

8-(tert-Butoxy)-5,7-dichloro-2-(perfluoroethyl)quinoline (29)

ppm (H)