Supporting Information

Palladium-Catalyzed Direct Intermolecular Silylation of Remote

Unactivated C(sp³)–H bonds with Disilanes

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1. General information

Solvents: Dichloromethane was distilled from CaH₂, and toluene was distilled from Na before use. Other solvents used in this manuscript were purchased in anhydrous form. **Reagents:** All commercial materials were used as received unless otherwise noted. Pd(OAc)₂ (99.9%, Acros), 1,4-benzoquinone (BQ) (99%, Acros), hexamethyldisilane (HMDS) (98%, Energy), 4Å MS (Energy, activated) were used in the Pd-catalyzed silylation reactions.

Reactions: All reactions were performed in oven-dried glassware under an atmosphere of argon unless otherwise noted. All yields reported were averages of at least two experimental runs.

Chromatography: Thin layer chromatography (TLC) was carried out on silica gel 60 F254 pre-coated glass plates. Visualization was detected by irradiation with UV light (254 nm), or by treatment with a solution of phosphomolybdic acid in ethanol followed by heating. Flash chromatography was carried out on 200 - 300 mesh silica gel, eluting with a mixture of petroleum ether (b.p. 60 - 90 °C) and ethyl acetate.

NMR Spectroscope: ¹H NMR and ¹³C NMR spectra were recorded on a Bruker AVANCE III HD 400 spectrometer, operating at 400 MHz and 100 MHz respectively. Chemical shifts (δ) were given in parts per million (ppm), and referenced relative to residual solvent CHCl₃ (7.26 ppm) in CDCl₃, or tetramethylsilane (0.00 ppm) as an internal standard for ¹H NMR spectra and deuterated solvent CDCl₃ (77.0 ppm) for ¹³C NMR spectra. Coupling constants (*J*) were reported in hertz (Hz). The following abbreviations are used to indicate the multiplicity of the signals: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, and associated combinations, e.g. dd = doublet of doublets.

Mass Spectrometry: High resolution mass spectra (HRMS) were obtained on a Bruker Daltonics SolariX 7.0 Tesla Fourier Transform Ion Cyclotron Resonance (FT-ICR) Mass Spectrometer using the electrospray ionization (ESI) technique.

Melting Points: Melting points (m.p.) were measured on a WRS-1B digital melting point apparatus (Shanghai Precision & Scientific Instrument Co., Ltd) and were uncorrected.

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2. Preparation of substrates

General procedure A:



According to literature procedure ^[1], to a solution of 8-aminoquinoline (10 mmol, 1.0 equiv) in dry DCM at 0 °C was added acyl chloride (11 mmol, 1.1 equiv) and Et₃N (12 mmol, 1.2 equiv). Then the reaction mixture was stirred overnight at room temperature. Saturated NaHCO₃ solution was added and the mixture was extracted with DCM (20 mL × 3). The combined organic layers were washed with water and brine, dried over anhydrous Na₂SO₄, filtered, and concentrated *in vacuo*. The resulting residue was purified by flash column chromatography to give the corresponding amide.

General procedure B:

$$\begin{array}{c} O \\ R^2 \\ OH \end{array} \xrightarrow{1) \text{ SOCI}_2, \text{ DMF, DCM, reflux}} \\ 2) \text{ 8-aminoquinoline, Et}_3N, \\ DCM, 0 \ ^\circ\text{C} \text{ to rt, overnight} \end{array} \xrightarrow{0}_{R^2} \\ \begin{array}{c} O \\ H \\ N \\ N \end{array}$$

According to literature procedure ^[2], a solution of acid (10 mmol, 1.0 equiv), SOCl₂ (11 mmol, 1.1 equiv) and DMF (3 drops) in DCM (20 mL) was heated to reflux for 5 hours, and the reaction mixture was concentrated *in vacuo*. And the residue was dissolved in DCM (20 mL), 8-aminoquinoline (9 mmol, 0.9 equiv) and Et₃N (13 mmol, 1.3 equiv) was added at 0 °C, and then stirred overnight at room temperature. Saturated NaHCO₃ solution was added and the mixture was extracted with DCM (20 mL × 3). The combined organic layers were washed with water and brine, dried over anhydrous Na₂SO₄, filtered, and concentrated *in vacuo*. The resulting residue was purified by flash column chromatography to give the corresponding amide.

General procedure C:



According to literature procedure ^[2], a mixture of amino-acid (10 mmol, 1.0 equiv) and *o*-phthalic anhydride (11 mmol, 1.1 equiv) was heated at 150 °C for 2 h. After cooling to room temperature, the resulting water and the unreacted *o*-phthalic anhydride were washed off by acetone (2 mL) and DCM (2 ml), the resulting solid was then dissolved in dry DCM (20 mL), SOCl₂ (13 mmol, 1.3 eq) was added and stirred overnight at 55 °C. After the reaction, DCM and excess of SOCl₂ were removed *in vacuo*. The crude acyl chloride was dissolved in dry DCM (20 mL), 8-aminoquinoline (9.2 mmol, 0.92 equiv) and Et₃N (13 mmol, 1.3 equiv) were added and stirred overnight at room temperature. Saturated NaHCO₃ solution was added and the mixture was extracted with DCM (20 mL × 3). The combined organic layers were washed with water and brine, dried over anhydrous Na₂SO₄, and concentrated *in vacuo*. The resulting residue was purified by flash column chromatography to give the corresponding amide.

All these amides used are known compounds and were list as follows.

1) Table S1: Amides 1a^[1], 1b^[3], 1c^[1], 1d^[4], 1e^[5], 1f^[6], 1g^[7], 1j^[1], 1l^[7], 1m^[2], 1n^[8], 1o^[2], 1p^[2], 1q^[3] and 1r^[1] were prepared according to the general procedure A.



Table S1 Amides prepared according to the general procedure A 2) Table S2: Amides **1i**^[3], **1k**^[6], **1s**^[9], **1t**^[10], **1w**^[11] were prepared according to the

general procedure B.



Table S2 Amides prepared according to the general procedure B

3) Table S3: Amide **1h**^[2], **9a**^[12], **9b**^[12], **9c**^[12], **9d**^[12], **9e**^[12] and **9f**^[12] were prepared according to the general procedure C.



Table S3 Amides prepared according to the general procedure C

3. Experimental details and characterization data



Typical procedure for Pd-catalyzed silvlation reaction: A mixture of amide **1a** (42.9 mg, 0.2 mmol), Pd(OAc)₂ (4.5 mg, 0.02 mmol, 10 mol %), BQ (86.5 mg, 0.4 mmol, 2 equiv), 4Å MS (30 mg) and HMDS (400 μ L, 2.0 mmol, 10 equiv) in DMA (1 mL) in 10-mL glass vial (purged with Ar, sealed with PTFE cap) was heated at 110 °C for 12 hours. The reaction mixture was cooled to room temperature, poured into water (20 mL). The aqueous layer was extracted with Et₂O (20 mL × 3), washed with saturated Na₂CO₃ solution (5 mL × 2). And the combined organic layers were dried over anhydrous Na₂SO₄, filtered, and concentrated *in vacuo*. The resulting residue was purified by flash column chromatography to give the corresponding silvlated product **2a**.



2a (colorless oil, 32.1 mg, 56%) was isolated as a colorless oil following the typical procedure starting from **1a**.



2b (colorless oil, 24.0 mg, 44%) was isolated following the typical procedure starting from **1b**.

¹H NMR (400 MHz, CDCl₃) δ 9.86 (s, 1H), 8.81 – 8.78 (m, 2H), 8.14 (dd, *J* = 8.3, 1.6 Hz, 1H), 7.54 – 7.45 (m, 2H), 7.43 (dd, *J* = 8.3, 4.2 Hz,

1H), 2.56 – 2.52 (m, 2H), 1.06 – 1.02 (m, 2H), 0.07 (s, 9H). 13 C NMR (100 MHz, CDCl₃) δ 173.0, 148.1, 138.3, 136.3, 134.6, 127.9, 127.4, 121.5, 121.2, 116.3, 32.7, 12.2, -1.9.

HRMS (ESI) *m/z* calculated for C₁₅H₂₁N₂OSi [M+H⁺] 273.1418, found 273.1417.



2c (colorless oil, 43.0 mg, 75%) was isolated following the typical procedure starting from **1c**.

 $\begin{array}{|c|c|c|c|c|c|c|c|} \hline SiMe_3 & & & ^{1}H \ NMR \ (400 \ MHz, \ CDCl_3) \ \delta \ 9.89 \ (s, \ 1H), \ 8.81 - 8.78 \ (m, \ 2H), \ 8.13 \\ \hline 2c & & & (dd, \ J = 8.3, \ 1.6 \ Hz, \ 1H), \ 7.54 - 7.46 \ (m, \ 2H), \ 7.43 \ (dd, \ J = 8.3, \ 4.2 \\ \hline Hz, \ 1H), \ 2.77 - 2.68 \ (m, \ 1H), \ 1.37 \ (d, \ J = 6.9 \ Hz, \ 3H), \ 1.20 \ (dd, \ J = 14.7, \ 7.0 \ Hz, \ 1H), \ 0.88 \\ \hline (dd, \ J = 14.7, \ 7.7 \ Hz, \ 1H), \ 0.06 \ (s, \ 9H). \ ^{13}C \ NMR \ (100 \ MHz, \ CDCl_3) \ \delta \ 176.2, \ 148.1, \ 138.4, \\ 136.3, \ 134.6, \ 127.9, \ 127.4, \ 121.5, \ 121.2, \ 116.3, \ 39.2, \ 22.3, \ 21.3, \ -1.0. \ HRMS \ (ESI) \ m/z \\ calculated \ for \ C_{16}H_{23}N_2OSi \ [M+H^+] \ 287.1574, \ found \ 287.1574. \end{array}$



2d (colorless oil, 46.9 mg, 78%) was isolated following the typical procedure starting from **1d**.

¹H NMR (400 MHz, CDCl₃) δ 9.86 (s, 1H), 8.82 – 8.80 (m, 2H), 8.14 (dd, J = 8.3, 1.6 Hz, 1H), 7.55 – 7.47 (m, 2H), 7.44 (dd, J =

8.3, 4.2 Hz, 1H), 2.52 – 2.45 (m, 1H), 1.90 – 1.79 (m, 1H), 1.70 – 1.60 (m, 1H), 1.14 (dd, J = 14.7, 8.9 Hz, 1H), 1.00 (t, J = 7.4 Hz, 3H), 0.88 (dd, J = 14.7, 5.9 Hz, 1H), 0.03 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 175.4, 148.1, 138.4, 136.3, 134.5, 127.9, 127.4, 121.5, 121.2, 116.4, 46.7, 29.5, 20.6, 12.0, -1.1. HRMS (ESI) *m/z* calculated for C₁₇H₂₅N₂OSi [M+H⁺] 301.1731, found 301.1730.



2e (colorless oil, 49.7 mg, 79%) was isolated following the typical procedure starting from **1e**.

¹H NMR (400 MHz, CDCl₃) δ 9.87 (s, 1H), 8.82 – 8.80 (m, 2H), 8.13 (dd, *J* = 8.3, 1.6 Hz, 1H), 7.54 – 7.41 (m, 2H), 7.43 (dd, *J*

= 8.3, 4.2 Hz, 1H), 2.61 – 2.54 (m, 1H), 1.87 – 1.78 (m, 1H), 1.60 – 1.51 (m, 1H), 1.46 – 1.37 (m, 2H), 1.14 (dd, J = 14.7, 8.8 Hz, 1H), 0.93 (t, J = 7.3 Hz, 3H), 0.88 (dd, J = 14.8, 6.0 Hz, 1H), 0.04 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 175.5, 148.1, 138.4, 136.2, 134.5, 127.9, 127.4, 121.5, 121.2, 116.4, 44.9, 38.7, 20.9, 20.7, 14.1, -1.1. HRMS (ESI) m/z calculated for C₁₈H₂₇N₂OSi [M+H⁺] 315.1887, found 315.1886.



2f (colorless oil, 22.9 mg, 38%) was isolated following the typical procedure starting from **1f**.

¹H NMR (400 MHz, CDCl₃) δ 9.84 (s, 1H), 8.81 – 8.79 (m, 2H),

8.13 (dd, J = 8.3, 1.6 Hz, 1H), 7.54 – 7.46 (m, 2H), 7.43 (dd, J =

8.3, 4.2 Hz, 1H), 2.60 (dd, J = 15.1, 5.2 Hz, 1H), 2.47 (dd, J = 15.1, 9.3 Hz, 1H), 1.68 – 1.57 (m, 1H), 1.53 – 1.42 (m, 1H), 1.38 – 1.30 (m, 1H), 1.00 (t, J = 7.4 Hz, 3H), 0.07 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 172.3, 148.0, 138.3, 136.3, 134.5, 127.9, 127.4, 121.5, 121.2, 116.3, 38.5, 24.5, 23.2, 13.7, -2.4. HRMS (ESI) m/z calculated for C₁₇H₂₅N₂OSi [M+H⁺] 301.1731, found 301.1730.



2g (colorless oil, 28.3 mg, 45%) was isolated following the typical procedure starting from **1g**.

¹H NMR (400 MHz, CDCl₃) δ 9.83 (s, 1H), 8.81 – 8.78 (m, 2H),

8.14 (dd, *J* = 8.3, 1.5 Hz, 1H), 7.55 – 7.46 (m, 2H), 7.43 (dd, *J*

= 8.3, 4.2 Hz, 1H), 2.61 (dd, J = 15.1, 4.7 Hz, 1H), 2.45 (dd, J = 15.1, 8.5 Hz, 1H), 1.53 – 1.34 (m, 5H), 0.87 (t, J = 6.7 Hz, 3H), 0.06 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 172.3, 148.0, 138.3, 136.3, 134.6, 127.9, 127.4, 121.5, 121.2, 116.3, 38.9, 32.8, 22.6, 22.2, 14.4, -2.5. HRMS (ESI) m/z calculated for C₁₈H₂₇N₂OSi [M+H⁺] 315.1887, found 315.1886.



2h (white solid, 44.1 mg, 48%) was isolated following the typical procedure starting from **1h**. m.p. = 98.7 - 99.9 °C. ¹H NMR (400 MHz, CDCl₃) δ 9.76 (s, 1H), 8.78 (dd, J = 4.2, 1.7 Hz, 1H), 8.68

(dd, J = 6.6, 2.4 Hz, 1H), 8.11 (dd, J = 8.3, 1.7 Hz, 1H), 7.73 – 7.68 (m, 2H), 7.62 – 7.57 (m, 2H), 7.48 – 7.40 (m, 3H), 3.70 – 3.59 (m, 2H), 2.59 (dd, J = 15.2, 5.1 Hz, 1H), 2.45 (dd, J = 15.2, 8.6 Hz, 1H), 1.86 – 1.71 (m, 2H), 1.64 – 1.56 (m, 1H), 1.52 – 1.37 (m, 2H), 0.04 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 171.7, 168.2, 148.0, 138.2, 136.2, 134.3, 133.6, 131.9, 127.8, 127.3, 122.9, 121.5, 121.2, 116.3, 38.5, 38.0, 27.9, 27.6, 22.1, -2.5. HRMS (ESI) m/z calculated for C₂₆H₃₀N₃O₃Si [M+H⁺] 460.2051, found 460.2046.



2i (colorless oil, 38.4 mg, 40%) was isolated following the typical procedure starting from **1i**. ¹H NMR (400 MHz, CDCl₃) δ 9.83 (s, 1H), 8.82 – 8.79 (m, 2H), 8.15 (dd, *J* = 8.3, 1.6 Hz, 1H), 7.55 – 7.47 (m,

2H), 7.45 (dd, J = 8.3, 4.2 Hz, 1H), 5.33 – 5.24 (m, 2H), 2.62 (dd, J = 15.0, 4.9 Hz, 1H), 2.46 (dd, J = 15.0, 8.6 Hz, 1H), 1.98 – 1.89 (m, 3H), 1.58 – 1.51 (m, 1H), 1.45 – 1.34 (m, 4H), 1.30 – 1.17 (m, 17H), 0.87 (t, J = 6.8 Hz, 3H), 0.06 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 172.3, 148.0, 138.2, 136.3, 134.6, 129.8, 129.7, 127.9, 127.5, 121.5, 121.2, 116.3, 38.9, 31.9, 30.4, 29.7, 29.6, 29.6, 29.5, 29.3, 29.0, 27.2, 22.8, 22.7, 14.1, -2.4. HRMS (ESI) m/z calculated for C₃₀H₄₉N₂OSi [M+H⁺] 481.3609, found 481.3605.



2j (white solid, 36.9 mg, 53%) was isolated following the typical procedure starting from **1j**.

m.p. = 85.6 – 87.0 °C. ¹H NMR (400 MHz, CDCl₃) δ 9.81 (s, 1H), 8.74 (dd, *J* = 4.2, 1.4 Hz, 1H), 8.68 (dd, *J* = 7.1, 1.7 Hz,

1H), 8.09 (dd, J = 8.3, 1.4 Hz, 1H), 7.47 – 7.38 (m, 3H), 7.24 – 7.18 (m, 4H), 7.07 (t, J = 7.1 Hz, 1H), 3.08 (dd, J = 15.7, 11.4 Hz, 1H), 2.97 (dd, J = 15.7, 4.3 Hz, 1H), 2.84 (dd, J = 11.3, 4.3 Hz, 1H), 0.03 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 171.0, 147.9, 142.2, 138.2, 136.2, 134.4, 128.3, 127.8, 127.4, 127.3, 124.8, 121.4, 121.2, 116.3, 38.4, 32.8, -3.0. HRMS (ESI) m/z calculated for C₂₁H₂₅N₂OSi [M+H⁺] 349.1730, found 349.1729.



2k (pale yellow oil, 38.8 mg, 53%) was isolated following the typical procedure starting from **1k**.

¹H NMR (400 MHz, CDCl₃) δ 9.77 (s, 1H), 8.73 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.67 (dd, *J* = 7.0, 2.0 Hz, 1H), 8.08 (dd, *J* = 8.3,

1.6 Hz, 1H), 7.48 – 7.38 (m, 3H), 7.16 – 7.11 (m, 2H), 6.96 – 6.90 (m, 2H), 3.04 – 2.92 (m, 2H), 2.81 (dd, J = 11.2, 4.6 Hz, 1H), 0.02 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 170.8, 160.6 (d, J = 241.0 Hz), 147.9, 138.2, 137.8, 136.2, 134.3, 128.6 (d, J = 7.6 Hz), 127.8, 127.2, 121.4, 121.3, 116.3, 115.1 (d, J = 21.0 Hz), 38.5, 32.0, -3.1. HRMS (ESI) m/z calculated for C₂₁H₂₄FN₂OSi [M+H⁺] 367.1636, found 367.1634.



2I (colorless oil, 18.9 mg, 30%) was isolated following the typical procedure starting from **1I**.

¹H NMR (400 MHz, CDCl₃) δ 9.85 (s, 1H), 8.81 (dd, *J* = 4.2, 1.6

21 Hz, 1H), 8.79 (d, J = 7.4, 1.4 Hz, 1H), 8.15 (dd, J = 8.3, 1.6 Hz, 1H), 7.54 – 7.47 (m, 2H), 7.44 (dd, J = 8.3, 4.2 Hz, 1H), 2.63 – 2.51 (m, 2H), 2.10 – 1.98 (m, 1H), 1.49 – 1.44 (m, 1H), 1.03 (d, J = 6.9 Hz, 3H), 0.98 (d, J = 6.9 Hz, 3H), 0.10 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 172.6, 148.1, 138.3, 136.3, 134.6, 127.9, 127.4, 121.5, 121.2, 116.3, 35.7, 30.0, 29.0, 22.4, 21.4, -1.0. HRMS (ESI) *m/z* calculated for C₁₈H₂₇N₂OSi [M+H⁺] 315.1893, found 315.1895.



2m (colorless oil, 22.5 mg, 33%) was isolated following the typical procedure starting from 1m.

¹H NMR (400 MHz, CDCl₃) δ 9.82 (s, 1H), 8.81 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.78 (dd, *J* = 7.5, 1.5 Hz, 1H), 8.14 (dd, *J* = 8.3, 1.6 Hz,

1H), 7.55 – 7.42 (m, 2H), 7.44 (dd, J = 8.3, 4.2 Hz, 1H), 2.61 (dd, J = 6.3, 1.5 Hz, 2H), 2.05 – 1.94 (m, 1H), 1.87 – 1.78 (m, 2H), 1.66 – 1.54 (m, 2H), 1.53 – 1.42 (m, 3H), 1.30 – 1.18 (m, 2H), 0.09 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 172.5, 148.1, 138.3, 136.3, 134.7, 127.9, 127.4, 121.5, 121.1, 116.3, 42.0, 37.4, 32.7, 31.6, 27.1, 25.2, 24.8, -1.3. HRMS (ESI) *m/z* calculated for C₂₀H₂₉N₂OSi [M+H⁺] 341.2044, found 341.2042.



2n (ratio = 1:1, colorless oil, 32.1 mg, 51%) was isolated following the typical procedure starting from 1n.

¹H NMR (400 MHz, CDCl₃) δ 9.85 (s, 1H), 9.83 (s, 1H), 8.86 – 8.81 (m, 4 H), 8.16 – 8.13 (m, 2 H), 7.55 – 7.51 (m, 2 H), 7.50 –

7.46 (m, 2H), 7.46 – 7.42 (m, 2H), 2.47 – 2.42 (m, 1H), 2.40 – 2.35 (m, 1H), 1.94 – 1.83 (m, 2H), 1.75 – 1.68 (m, 1H), 1.67 – 1.58 (m, 1H), 1.28 – 1.14 (m, 2H), 1.09 – 1.05 (m, 6 H), 1.01 – 0.97 (m, 6H), 0.07 (s, 9H), 0.05 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 174.7, 174.5, 148.1, 138.4, 138.4, 136.3, 134.6, 134.4, 127.9, 127.9, 127.4, 121.5, 121.2, 121.1, 116.4, 116.3, 53.7, 52.4, 25.8, 23.9, 23.7, 23.2, 12.9, 12.5, 11.9, -1.9, -2.1. HRMS (ESI) *m/z* calculated for C₁₈H₂₇N₂OSi [M+H⁺] 315.1887, found 315.1886.



20 (white solid, 32.2 mg, 45%) was isolated following the typical procedure starting from **10**.

m.p. = 91.4 – 93.1 °C. ¹H NMR (400 MHz, CDCl₃) δ 9.94 (s,
1H), 8.82 (dd, J = 4.2, 1.6 Hz, 1H), 8.72 (dd, J = 7.5, 1.0 Hz,

1H), 8.15 (dd, J = 8.3, 1.6 Hz, 1H), 7.54 – 7.43 (m, 3H), 2.38 (t, J = 9.2 Hz, 1H), 0.44 (d, J = 9.2 Hz, 2H), 0.16 (s, 18H). ¹³C NMR (100 MHz, CDCl₃) δ 171.0, 148.0, 138.2, 136.3, 134.9, 128.0, 127.5, 121.5, 120.8, 116.2, 25.9, 13.0, 0.8. HRMS (ESI) m/z calculated for C₁₉H₂₉N₂OSi₂ [M+H⁺] 357.1811, found 357.1813.



2p (white solid, 38.6 mg, 52%) was isolated following the typical procedure starting from 1p.

m.p. = 114.6 – 116.3 °C. ¹H NMR (400 MHz, CDCl₃) δ 9.58 (s, 1H), 8.85 (dd, *J* = 7.6, 1.3 Hz, 1H), 8.77 (dd, *J* = 4.2, 1.7

Hz, 1H), 8.14 (dd, J = 8.3, 1.6 Hz, 1H), 7.56 – 7.52 (m, 1H), 7.46 (dd, J = 8.2, 1.3 Hz, 1H), 7.42 (dd, J = 8.2, 4.2 Hz, 1H), 3.54 – 3.49 (m, 1H), 2.46 – 2.34 (m, 3H), 2.11 – 2.05 (m, 1H), 0.00 (s, 18H). ¹³C NMR (100 MHz, CDCl₃) δ 173.9, 147.9, 138.3, 136.2, 134.9, 128.0, 127.6, 121.5, 120.8, 116.1, 45.9, 28.8, 23.9, -1.9. HRMS (ESI) *m/z* calculated for C₂₀H₃₁N₂OSi₂ [M+H⁺] 371.1969, found 371.1967.



2q (white solid, 40.8 mg, 53%) was isolated following the typical procedure starting from **1q**.

m.p. = 89.8 – 90.2 °C. ¹H NMR (400 MHz, CDCl₃) δ 9.84 (s, 1H), 8.80 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.76 (dd, *J* = 7.3, 1.6 Hz,

1H), 8.14 (dd, J = 8.3, 1.7 Hz, 1H), 7.53 – 7.42 (m, 3H), 3.11 (t, J = 6.2 Hz, 1H), 2.18 – 2.08 (m, 2H), 2.03 – 1.95 (m, 2H), 1.28 – 1.21 (m, 2H), 0.00 (s, 18H). ¹³C NMR (100 MHz, CDCl₃) δ 174.2, 148.2, 138.5, 136.4, 134.8, 128.1, 127.7, 121.6, 121.2, 116.4, 52.7, 36.3, 27.3, -1.6. HRMS (ESI) m/z calculated for C₂₁H₃₃N₂OSi₂ [M+H⁺] 385.2126, found 385.2124.



2r (white solid, 40.7 mg, 51%) was isolated following the typical procedure starting from **1r**.

m.p. = 107.9 – 110.0 °C. ¹H NMR (400 MHz, CDCl₃) δ 9.94 (s, 1H), 8.81 (dd, *J* = 4.2, 1.6 Hz, 1H), 8.77 (dd, *J* = 7.4, 1.5 Hz,

1H), 8.14 (dd, J = 8.3, 1.6 Hz, 1H), 7.54 – 7.46 (m, 2H), 7.43 (dd, J = 8.3, 4.2 Hz, 1H), 2.83 (t, J = 4.1 Hz, 1H), 2.26 – 2.15 (m, 2H), 2.05 – 2.00 (m, 1H), 1.64 – 1.60 (m, 2H), 1.39 – 1.26 (m, 1H), 0.96 – 0.90 (m, 2H), -0.03 (s, 18H). ¹³C NMR (100 MHz, CDCl₃) δ 173.5, 148.1, 138.4, 136.2, 134.6, 127.9, 127.5, 121.5, 121.0, 116.3, 43.4, 32.1, 29.2, 22.2, -2.7. HRMS (ESI) *m/z* calculated for C₂₂H₃₅N₂OSi₂ [M+H⁺] 399.2282, found 399.2279.



2s (white solid, 43.6 mg, 64%) was isolated following the typical procedure starting from **1s**, except for using BQ (2 equiv) and HMDS (5 equiv).

 $\begin{array}{|c|c|c|c|c|} \hline 2s \\ \hline m.p. = 103.4 - 105.1 \ ^{\circ}C. \ ^{1}H \ NMR \ (400 \ MHz, \ CDCl_3) \ \delta \ 9.87 \ (s, 1H), \ 8.81 - 8.78 \ (m, 2H), \ 8.14 \ (dd, J = 8.3, 1.6 \ Hz, 1H), \ 7.54 - 7.46 \ (m, 2H), \ 7.44 \ (dd, J = 8.3, 4.2 \ Hz, 1H), \ 2.91 \ (dt, J = 9.6, 5.8 \ Hz, 1H), \ 2.20 - 2.13 \ (m, 1H), \ 2.03 - 1.90 \ (m, 3H), \ 1.88 - 1.81 \ (m, 2H), \ 1.74 - 1.68 \ (m, 1H), \ 1.57 - 1.38 \ (m, 2H), \ 1.33 - 1.22 \ (m, 1H), \ 1.08 \ (ddd, J = 9.6, \ 5.6, \ 2.1 \ Hz, \ 1H), \ 0.01 \ (s, \ 9H). \ ^{13}C \ NMR \ (100 \ MHz, \ CDCl_3) \ \delta \ 175.5, \ 148.0, \ 138.4, \ 136.3, \ 134.7, \ 127.9, \ 127.5, \ 121.5, \ 121.1, \ 116.3, \ 47.4, \ 32.7, \ 31.7, \ 30.7, \ 30.5, \ 25.6, \ 25.1, \ -2.0. \ HRMS \ (ESI) \ m/z \ calculated \ for \ C_{20}H_{29}N_2OSi \ [M+H^+] \ 341.2044, \ found \ 341.2042.$



2t (white solid, 9.8 mg, 13%) was isolated following the typical procedure starting from **1t**.

m.p. = 103.7 - 105.2 °C. ¹H NMR (400 MHz, CDCl₃) δ 10.26 (s, 1H), 8.85 - 8.82 (m, 2H), 8.14 (dd, *J* = 8.2, 1.4 Hz, 1H),

7.55 – 7.43 (m, 3H), 2.44 (d, *J* = 11.2 Hz, 1H), 2.46 – 2.42 (m, 1H), 2.33 – 2.29 (m, 1H), 2.22 – 2.19 (m, 2H), 2.17 – 2.14 (m, 1H), 2.05 – 2.00 (m, 1H), 1.95 – 1.81 (m, 4H), 1.79 – 1.64 (m, 3H), 1.61 – 1.60 (m, 1H), 0.00 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 176.1, 148.1, 138.8, 136.2, 134.6, 127.9, 127.5, 121.5, 121.1, 116.3, 44.4, 43.7, 39.7, 38.7, 36.8, 35.7, 34.1, 30.2, 28.6, 28.0, 0.2. HRMS (ESI) *m/z* calculated for C₂₃H₃₁N₂OSi [M+H⁺] 379.2200, found 379.2198.



10a (white solid, 73.4 mg, 88%) was isolated following the typical procedure starting from **9a**.

m.p. = 107.1 - 108.7 °C. ¹H NMR (400 MHz, CDCl₃) δ 10.47 (s, 1H), 8.74 - 8.69 (m, 2H), 8.13 (dd, J = 8.3, 1.7

Hz, 1H), 7.92 - 7.86 (m, 2H), 7.76 - 7.72 (m, 2H), 7.52 - 7.48 (m, 2H), 7.42 (dd, J = 8.3, 4.2 Hz, 1H), 5.23 (dd, J = 12.0, 5.0 Hz, 1H), 2.08 (dd, J = 14.5, 12.0 Hz, 1H), 1.66 (dd, J = 14.5, 5.0 Hz, 1H), 0.05 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 168.2, 168.0, 148.3, 138.5, 136.2, 134.2, 133.9, 131.8, 127.8, 127.2, 123.5, 121.8, 121.5, 116.5, 52.2, 17.1, -1.5. HRMS (ESI) *m/z* calculated for C₂₃H₂₄N₃O₃Si [M+H⁺] 418.1581, found 418.1577.



Mixture of **10b** (*major*) and **10b'** (d.r. = 5:1, white solid, 69.9 mg, 81%) were isolated following the typical procedure starting from **9b**.

10b m.p. = 142.6 – 146.6 °C. ¹H NMR (400 MHz, CDCl₃) δ 10.76 (s, 1H), 8.88 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.78 – 8.72 (m, 1H), 8.13 (dd, *J* = 8.3, 1.6 Hz, 1H), 7.90 – 7.85 (m, 2H), 7.74 – 7.69 (m, 2H), 7.52 – 7.47 (m, 2H), 7.44 (dd, *J* = 8.3, 4.2 Hz, 1H), 5.00 (d, *J* = 12.2 Hz, 1H), 2.44 – 2.34 (m, 1H), 0.99 (d, *J* = 7.4 Hz, 3H), 0.08 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 168.3, 167.3, 148.5, 138.7, 136.1, 134.3, 134.2, 131.5, 127.9, 127.2, 123.5, 121.9, 121.5, 117.0, 60.5, 19.8, 12.4, -2.6. HRMS (ESI) *m/z* calculated for C₂₄H₂₆N₃O₃Si [M+H⁺] 432.1738, found 432.1734.



¹H NMR (400 MHz, CDCl₃) δ 10.73 (s, 1H), 8.92 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.78 – 8.72 (m, 1H), 8.13 (dd, *J* = 8.3, 1.6 Hz, 1H), 7.90 – 7.85 (m, 2H), 7.74-7.69 (m, 2H), 7.52 – 7.47 (m, 2H), 7.44 (dd, *J* = 8.3, 4.2 Hz, 1H), 4.96 (d, *J* =

12.2 Hz, 1H), 2.63 – 2.54 (m, 1H), 1.19 (d, J = 7.4 Hz, 3H), -0.02 (s, 9H). ¹³C NMR (100

MHz, CDCl₃) δ 168.0, 167.5, 148.5, 138.8, 136.1, 134.4, 134.2, 131.7, 127.9, 127.2, 123.6, 121.9, 121.5, 116.9, 59.9, 19.5, 13.0, -2.6.



10c (pale yellow oil, 29.5 mg, 32%) isolated following the typical procedure starting from **9c**.

¹H NMR (400 MHz, CDCl₃) δ 10.76 (s, 1H), 8.90 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.75 – 8.71 (m, 1H), 8.12 (dd, *J* = 8.3,

1.6 Hz, 1H), 7.91 – 7.85 (m, 2H), 7.73 – 7.69 (m, 2H), 7.51 – 7.47 (m, 2H), 7.44 (dd, J = 8.3, 4.2 Hz, 1H), 5.25 (d, J = 12.5 Hz, 1H), 2.52 (dd, J = 12.5, 2.1 Hz, 1H), 2.01 – 1.94 (m, 1H), 1.10 (d, J = 7.1 Hz, 3H), 0.96 (d, J = 7.1 Hz, 3H), 0.15 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 168.4, 167.6, 148.5, 138.8, 136.1, 134.4, 134.2, 131.7, 127.9, 127.2, 123.6, 121.9, 121.6, 117.2, 58.5, 32.3, 28.2, 22.0, 21.0, 0.6. HRMS (ESI) m/z calculated for C₂₆H₃₀N₃O₃Si [M+H⁺] 460.2051, found 460.2045.



10d (white solid, 22.9 mg, 23%) isolated following the typical procedure starting from **9d**.

m.p. = 172.1 – 173.2 °C. ¹H NMR (400 MHz, CDCl₃) δ 10d 10.94 (s, 1H), 8.93 (dd, J = 4.2, 1.7 Hz, 1H), 8.82 – 8.78 (m, 1H), 8.14 (dd, J = 8.3, 1.7 Hz, 1H), 7.70 – 7.66 (m, 2H), 7.57 – 7.54 (m, 2H), 7.53 – 7.50 (m, 2H), 7.45 (dd, J = 8.3, 4.2 Hz, 1H), 7.14 – 7.10 (m, 4H), 6.99 – 6.92 (m, 1H), 5.61 (d, J = 13.5 Hz, 1H), 3.90 (d, J = 13.5 Hz, 1H), 0.02 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 168.1, 167.1, 148.6, 139.5, 138.8, 136.1, 134.4, 133.9, 131.2, 128.2, 127.9, 127.2, 125.4, 123.2, 122.1, 121.6, 117.3, 59.1, 36.4, -2.1. HRMS (ESI) *m/z* calculated for C₂₉H₂₈N₃O₃Si [M+H⁺] 494.1894, found 494.1888.



10e (white solid, 40.2 mg, 45%) was isolated following the typical procedure starting from **9e**.

m.p. = 113.0 – 114.3 °C. ¹H NMR (400 MHz, CDCl₃) δ 10.63 (s, 1H), 8.83 (dd, J = 4.2, 1.7 Hz, 1H), 8.77 – 8.73

(m, 1H), 8.11 (dd, J = 8.3, 1.7 Hz, 1H), 7.88 – 7.81 (m, 2H), 7.72 – 7.68 (m, 2H), 7.51 –

7.47 (m, 2H), 7.42 (dd, J = 8.3, 4.2 Hz, 1H), 4.72 (d, J = 10.6 Hz, 1H), 3.26 – 3.15 (m, 1H), 1.03 (dd, J = 14.4, 1.1 Hz, 1H), 0.98 (d, J = 6.6 Hz, 3H), 0.58 (dd, J = 14.4, 11.5 Hz, 1H), 0.04 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 168.1, 166.7, 148.4, 138.6, 136.0, 134.2, 134.1, 131.5, 127.8, 127.1, 123.5, 121.9, 121.6, 116.9, 64.6, 28.7, 22.0, 18.8, -0.7. HRMS (ESI) m/z calculated for C₂₅H₂₈N₃O₃Si [M+H⁺] 446.1894, found 446.1889.



10f (white solid, 41.4 mg, 45%) was isolated following the typical procedure starting from **9f**.

m.p. = 135.6 – 137.3 °C. ¹H NMR (400 MHz, CDCl₃) δ 10.67 (s, 1H), 8.87 (dd, *J* = 4.2, 1.7 Hz, 1H), 8.77 – 8.72

(m, 1H), 8.12 (dd, J = 8.3, 1.7 Hz, 1H), 7.88 – 7.83 (m, 2H), 7.72 – 7.68 (m, 2H), 7.50 – 7.46 (m, 2H), 7.44 (dd, J = 8.3, 4.2 Hz, 1H), 4.89 (d, J = 11.2 Hz, 1H), 3.35 – 3.27 (m, 1H), 1.64 – 1.54 (m, 1H), 1.46 – 1.36 (m, 1H), 0.86 – 0.82 (m, 5H), 0.04 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 168.1, 167.1, 148.5, 138.7, 136.1, 134.2, 134.2, 131.5, 127.8, 127.1, 123.5, 121.9, 121.6, 117.0, 61.6, 32.9, 23.6, 17.0, 8.0, -0.8. HRMS (ESI) *m/z* calculated for C₂₆H₃₀N₃O₃Si [M+H⁺] 460.2050, found 460.2045.

4. Gram-scale reaction



A mixture of amide **9a** (1.38 g, 4.0 mmol), Pd(OAc)₂ (89.8 mg, 0.4 mmol, 10 mol %), BQ (864.8 mg, 8.0 mmol, 2 equiv), 4Å MS (600 mg) and HMDS (4.0 mL, 20 mmol, 10 equiv) in DMA (10 mL) in 75-mL glass vial (purged with Ar, sealed with PTFE cap) was heated at 110 °C for 12 hours. The reaction mixture was cooled to RT, and filtered through Celite^{*}. And the filtrate cake was washed with Et₂O (100 mL). The filtrate was poured into water (50 mL). The aqueous layer was extracted with Et₂O (100 mL × 3), washed with saturated Na₂CO₃ solution (30 mL × 3). And the combined organic layers were dried over anhydrous Na₂SO₄, filtered, and concentrated *in vacuo*. The resulting residue was purified by flash column chromatography to give the corresponding silylated product **10a** (pale yellow solid, 1.34 g, 80%).

5. Removal of the auxiliary



A mixture of compound **10a** (83.5 g, 0.2 mmol) and $BF_3 \cdot Et_2O$ (150 µL, 1.2 mmol, 6 equiv) in MeOH (2 mL) in 10-mL glass vial (purged with Ar, sealed with PTFE cap) was heated at 100 °C for 18 hours. The reaction mixture was cooled to RT, diluted with DCM (3 mL), and Et_3N (280 µL, 2.0 mmol, 10 equiv) was added. Then the mixture was concentrated *in vacuo*. The resulting residue was purified by flash column chromatography to give the corresponding carboxylic ester **15** (pale yellow oil, 78.4 mg, 86%) and 8aminoquinoline (brown solid, 40.9 mg, 95%).



¹H NMR (400 MHz, CDCl₃) δ 7.87 – 7. 28 (m, 2H), 7.74 – 7.70 (m, 2H), 4.94 (dd, *J* = 11.7, 4.8 Hz, 1H), 3.69 (s, 3H), 1.70 (dd, *J* = 15.1, 11.7 Hz, 1H), 1.52 (dd, *J* = 15.1, 4.8 Hz, 1H), -0.05 (s, 9H). ¹³C NMR (100 MHz, CDCl₃) δ 171.0, 167.5, 134.1, 131.8, 123.4,

52.7, 49.1, 17.0, -1.7. HRMS (ESI) m/z calculated for $C_{15}H_{20}NO_4Si$ [M+H⁺] 306.1156, found 306.1156.

6. References

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7. X-Ray crystallographic analysis of 2j, 2p and 10f

A colorless block shaped crystal of **2j** ($C_{21}H_{24}N_2OSi$) was used for the X-ray crystallographic analysis. The X-ray intensity data were measured at 173(2) K, on a Bruker D8 VENTURE diffractometer with helios mx multilayer monochrmator Cu-K_{α} radiation ($\lambda = 1.54178$ Å). The X-ray crystallographic files, in CIF format, are available from the Cambridge Crystallographic Data Centre on quoting the deposition numbers CCDC 1508602 for **2j**. Copies of the data can be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge CB2 IEZ, UK (Fax: +44-1223-336033; E-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).



Figure 1 X-Ray Structure of 2j

Table 54 Crystal uata and structure refinement for Z	Table S4	Crystal data and	structure	refinement for 2j
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Empirical formula	$C_{21}H_{24}N_2OSi$	
Formula weight	348.51	
Temperature	173(2) K	
Wavelength	1.54178 Å	
Crystal habit	Colorless block	
Crystal system, space group,	Triclinic, P -1	
Unit cell dimensions	a = 10.0800(3) Å	alpha = 88.653(2)°
	b = 11.8813(4) Å	beta = 86.499(2)°

	c = 16.5509(6) Å gamma = 89.381(2)°	
Volume	1977.86(11) ų	
Z, Calculated density	4, 1.170 g/cm ³	
Absorption coefficient	1.117 mm ⁻¹	
F(000)	800	
Crystal size	0.200 x 0.200 x 0.200 mm ³	
Diffractometer	Bruker APEX-II CCD	
Radiation type	CuKα	
Data collection method	phi and omega scans	
Theta range for data collection	4.54 to 68.38°	
Limiting indices	-11 ≤ h ≤ 11, -13 ≤ k ≤ 13, -19 ≤ l ≤ 19	
Reflections collected	9858	
Data/restraints/parameters	6044/0/457	
Goodness-of-fit	1.033	
Final R indices [I > 2 sigma(I)]	R1 = 0.0536, wR2 = 0.1413	
R indices (all data)	R1 = 0.0715, wR2 = 0.1518	

A colorless block shaped crystal of **2p** ($C_{20}H_{30}N_2OSi_2$) was used for the X-ray crystallographic analysis. The X-ray intensity data were measured at 173(2) K, on a Bruker D8 VENTURE diffractometer with helios mx multilayer monochrmator Cu-K_{α} radiation ($\lambda = 1.54178$ Å). The X-ray crystallographic files, in CIF format, are available from the Cambridge Crystallographic Data Centre on quoting the deposition numbers CCDC 1508603 for **2p**. Copies of the data can be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge CB2 IEZ, UK (Fax: +44-1223-336033; E-mail: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).



Figure 2 X-Ray Structure of 2p

Table S5	Crystal data	and structure	refinement for 2p
Table 33	Crystar uata		

Empirical formula	$C_{20}H_{30}N_2OSi_2$	
Formula weight	370.64	
Temperature	173(2) K	
Wavelength	1.54178 Å	
Crystal habit	Colorless block	
Crystal system, space group,	Monoclinic, P 21/c	
Unit cell dimensions	a = 17.5738(8) Å	alpha = 90°

	b = 10.3894(4) Å	beta = 100.934(2)°
	c = 12.0069(5) Å	gamma = 90°
Volume	2152.44(16) Å ³	
Z, Calculated density	4, 1.144 g/cm ³	
Absorption coefficient	1.562 mm ⁻¹	
F(000)	800	
Crystal size	0.250 x 0.220 x 0.200 mm ³	
Diffractometer	Bruker APEX-II CCD	
Radiation type	CuKα	
Data collection method	phi and omega scans	
Theta range for data collection	2.56 to 68.22°	
Limiting indices	-21 ≤ h ≤ 18, -12 ≤ k ≤ 12, -12 ≤ l ≤ 14	
Reflections collected	17400	
Data/restraints/parameters	3834/0/232	
Goodness-of-fit	1.006	
Final R indices [I > 2 sigma(I)]	R1 = 0.0427, wR2 = 0.1225	
R indices (all data)	R1 = 0.0495, wR2 = 0.1337	

A colorless block shaped crystal of **10f** ($C_{26}H_{29}N_3O_3Si$) was used for the X-ray crystallographic analysis. The X-ray intensity data were measured at 173(2) K, on a Bruker D8 VENTURE diffractometer with helios mx multilayer monochrmator Cu-K_{α} radiation ($\lambda = 1.54178$ Å). The X-ray crystallographic files, in CIF format, are available from the Cambridge Crystallographic Data Centre on quoting the deposition numbers CCDC 1508604 for **10f**. Copies of the data can be obtained free of charge from the Director, CCDC, 12 Union Road, Cambridge CB2 IEZ, UK (Fax: +44-1223-336033; E-mail: deposit@ccdc.cam.ac.uk or www: http://www. ccdc.cam.ac.uk).



Figure 3 X-Ray Structure of 10f

Table S6	Crystal data and structure refinement for	or 10f
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Empirical formula	$C_{26}H_{29}N_3O_3Si$	
Formula weight	459.61	
Temperature	173(2) K	
Wavelength	1.54178 Å	
Crystal habit	Colorless block	
Crystal system, space group,	Monoclinic, C 2	
Unit cell dimensions	a = 40.8407(9) Å	alpha = 90°

	b = 11.7200(3) Å	beta = 102.7310(10)°	
	c = 10.6590(2) Å	gamma = 90°	
Volume	4976.53(19) Å ³		
Z, Calculated density	8, 1.227 g/cm ³		
Absorption coefficient	1.086 mm ⁻¹		
F(000)	1952		
Crystal size	0.200 x 0.200 x 0.200 mm ³		
Diffractometer	Bruker APEX-II CCD		
Radiation type	CuKα		
Data collection method	phi and omega scans		
Theta range for data collection	3.93 to 68.28°		
Limiting indices	-46 ≤ h ≤ 49, -14 ≤ k ≤ 14, -11 ≤ l ≤ 12		
Reflections collected	25688		
Data/restraints/parameters	8653/1/ 603		
Goodness-of-fit	1.050		
Final R indices [I > 2 sigma(I)]	R1 = 0.0448, wR2 = 0.1094		
R indices (all data)	R1 = 0.0521, wR2 =	0.1130	

8. Copies of ¹H and ¹³C NMR spectra





77.48 77.16

40.76

_____17.39 _____14.48 -3.33







S29








































S49







































0 Ο н Ń 0 SiMe₃



19.83 19.48 12.98 12.41











0.61








168.05

138.64 136.04 134.18 134.18 134.14 137.11 127.81 127.81 127.81 123.55 116.89 -148.42

77.32 77.00 76.68 64.55

Т

____22.03 ____18.79 -28.74

-0.72







