1. General

1.1. Reagents
All reagents and solvents were obtained from commercial suppliers and were used without further purification unless otherwise stated. Purification was carried out according to standard laboratory methods.

1.2 Purification of Solvents
i) All solvents used for dry reactions (dichloromethane, tetrahydrofuran, toluene) were obtained from a PureSolv SPS-400-5 solvent purification system. These solvents were transferred to and stored in a septum-sealed oven-dried flask over previously activated 4 Å molecular sieves and purged with and stored under nitrogen.
ii) Dichloromethane, diethyl ether, ethyl acetate, methanol, and petroleum ether 40-60° for purification purposes were used as obtained from suppliers without further purification.

1.3 Purification of Starting Materials
2-Picoline was dried by heating to reflux over calcium chloride and then distilled under reduced pressure, then purged with and stored under nitrogen over 4 Å molecular sieves.

1.4 Organometallic Reagents
n-Butyllithium (1.6 M in hexanes) and isopropylmagnesium chloride (2 M in Et₂O) were used as obtained from commercial suppliers.

1.5 Experimental Details
i) Air-sensitive reactions were carried out using conventional glassware. The glassware was oven-dried and purged with N₂ before use.
ii) Purging refers to a vacuum/nitrogen-refilling procedure.
iii) Reactions were carried out at -78 °C using dry ice/acetone baths.
iv) Reactions were carried out at -20 °C using controlled dry ice/acetone baths.
v) Reactions were carried out at 0 °C using ice/water baths.
vi) Room temperature was generally ca. 18 °C.
vii) Reactions were carried out at elevated temperatures using a temperature-regulated hotplate/stirrer.
1.6 Purification of Products

i) Thin layer chromatography was carried out using Merck silica plates coated with fluorescent indicator UV254. These were analysed under 254 nm UV light or developed using potassium permanganate solution.

ii) Flash chromatography was carried out using ZEOprep 60 HYD 40-63 µm silica gel or IST Isolute Flash silica cartridges.

1.4 Analysis of Products

i) Fourier Transformed Infra-Red (FTIR) spectra were obtained on a Shimadzu IRAffinity-1 machine.

ii) $^1$H and $^{13}$C NMR spectra were obtained on a Bruker AV 400 at 400 MHz and 100 MHz respectively. Chemical shifts are reported in ppm and coupling constants are reported in Hz with CDCl$_3$ referenced at 7.27 (1H) and 77.36 ppm (13C), respectively.

iii) High-resolution mass spectra were obtained on a Thermo Surveyor HPLC coupled to a LTQ Orbitrap mass spectrometer (ThermoFisher) instrument at the Strathclyde Institute for Pharmacy and Biomedical Science, University of Strathclyde, Glasgow. Run conditions 4.6 x 50 mm Gemini C18 column (Phenomenex) flow rate 0.4 ml min$^{-1}$ MeCN/H$_2$O containing 0.1% v/v formic acid (95:5): (i) run time = 3 min; (ii) positive ion ESI mode spray voltage 4.5 kV; (iii) sheath gas 50 psi; (iv) auxiliary gas 15 psi; (v) heated capillary temperature 275 °C; (vi) resolution setting = 30,000.

(iv) Crystal data for 3a (C$_{15}$H$_{11}$NO, MW = 221.25) were measured at 123 K using an Oxford Diffraction Xcalibur E instrument and MoKα radiation, λ = 0.71073 Å. Found monoclinic, space group P21/c, a = 9.0140(5), b = 10.7721(5), c = 11.3234(7) Å, $\beta = 101.481(6)^\circ$, V = 1077.50(10) Å$^3$, Z = 4, $\mu = 0.086$ mm$^{-1}$, 20max = 54°, 4957 reflections, 2347 unique, Rint 0.0276; final refinement to convergence on F2 gave R = 0.0475 (F, 1703 obs. data only) and Rw = 0.1061 (F2, all data), GOF = 1.041. CCDC reference number 915082.

2. Experimental Procedures

2.1 Preparation of Weinreb Amides

The Weinreb amides required for the synthesis of the ketopyridines were prepared either via the corresponding acyl chloride (Method A) or ester (Method B) as per the following chart.
2.2 General Experimental Procedure for the Preparation of Weinreb Amides from Acyl Chlorides and \(N,O\)-Dimethylhydroxylamine Hydrochloride

For example, for Compound 5d. 4-(\(n\)-Butyl)-\(N\)-methoxy-\(N\)-methylbenzamide

An oven-dried flask containing a stir bar and fitted with a septum was purged with \(N_2\). The flask was then charged with \(N,O\)-dimethylhydroxylamine hydrochloride (1.1 equiv, 27.5 mmol, 2.68 g) and dry \(CH_2Cl_2\) (100 mL). The mixture was cooled to 0 °C and stirred for 5 min before addition of \(Et_3N\) (2.5 equiv, 50 mmol, 5.06 g, 6.97 mL) and 4-\(n\)-butylbenzoyl chloride (1 equiv, 25 mmol, 4.92 g). The resulting mixture was stirred for 30 min before warming to room temperature and stirring for 16 h. The mixture was treated with sat. aq. \(NaHCO_3\) solution (30 mL) and stirred vigorously for 10 min. The phases were separated and the aqueous layer was re-extracted with \(CH_2Cl_2\) (3 x 20 mL). The combined organic phases were dried by passing through a hydrophobic frit and concentrated under reduced pressure to afford a residue that was purified by flash column chromatography (1:3 EtOAc/petroleum ether 40-60°) to afford the title compound (5.083 g, 92% yield).

2.3 General Experimental Procedure for the Preparation of Weinreb Amides from Esters and \(N,O\)-Dimethylhydroxylamine Hydrochloride Using Isopropylmagnesium Chloride
For example, for **Compound 5p. N-Methoxy-N-methylfuran-2-carboxamide**

Following the procedure of Williams: An oven-dried flask containing a stir bar and fitted with a septum was purged with N₂. The flask was then charged with N₂-dimethylhydroxylamine hydrochloride (1.55 equiv, 15.5 mmol, 1.51 g), methyl 2-furoate (1 equiv, 10 mmol, 1.26 g, 1.07 mL) and dry THF (20 mL). The mixture was cooled to -20 °C and stirred for 5 min before addition of i-PrMgCl (2 M in Et₂O, 3 equiv, 30 mmol, 15 mL) dropwise over 15 min. The resulting mixture was stirred for a further 10 min before quenching with sat. aq. NaHCO₃ solution (10 mL) and H₂O (10 mL). The mixture was allowed to warm to room temperature and was stirred vigorously for 10 min. EtOAc (10 mL) was added and the phases were separated. The aqueous layer was re-extracted with EtOAc (3 x 20 mL). The combined organic phases were dried (MgSO₄), filtered, and concentrated under reduced pressure to afford a residue that was purified by flash column chromatography (1:1 EtOAc/petroleum ether 40-60°) to afford the title compound (1.32 g, 85% yield).

**2.4 General Experimental Procedure for the Deprotonation of 2-Picoline and Nucleophilic Addition to Weinreb Amides**

For example, for **Compound 1a. 1-Phenyl-2-(pyridin-2-yl)ethanone**

An oven-dried flask equipped with a stirrer bar and fitted with a septum was purged with N₂. The flask was cooled to -78 °C by submerging in a bath of dry ice/acetone. The flask was then charged with dry THF (11 mL) and 2-picoline (2 equiv, 4 mmol, 372.5 mg, 0.395 mL) and stirred. n-Butyllithium (1.6 M in hexanes, 2 equiv, 4 mmol, 2.5 mL) was added to the flask dropwise over 5 minutes. Concurrently, an additional oven dried flask equipped with a stirrer bar and fitted with a septum was purged with N₂. The flask was cooled to -78 °C by submerging in a dry ice/acetone bath. The flask was then charged with dry THF (5 mL) and N-methoxy-N-methylbenzamide (1 equiv, 2 mmol, 330.4 mg, 0.304 mL) and stirred. After 30 min, the deprotonated picoline solution was then added to the Weinreb amide solution by dropwise addition via syringe over 10 minutes. The reaction was allowed to proceed for 3 hours before quenching with water. EtOAc (5 mL) was added and the phases were separated. The aqueous layer was then re-extracted with EtOAc (5 x 5 mL). The combined organic phases were dried (MgSO₄), filtered, and concentrated under reduced pressure to afford a residue that was purified by flash column chromatography (1:2 EtOAc/petroleum ether 40-60°) to afford the title compound (327 mg, 83% yield).

**2.5 General Experimental Procedure for the Synthesis of Quinolizinones via Tandem Horner-Wadsworth-Emmons Olefination/Intramolecular Cyclisation**

For example, for **Compound 3a. 2-Phenyl-4H-quinolizin-4-one**

An oven-dried flask equipped with stirrer bar was purged with N₂ and cooled to 0 °C by submerging in an ice/water bath. The flask was charged with dry toluene (5 mL), triethyl phosphonoacetate (2 equiv, 2 mmol, 448.4 mg, 0.398 mL), and NaH (60% dispersion in mineral oil, 2 equiv, 2 mmol, 83.0 mg). The mixture was stirred for 15 min before removing from the cooling bath and warming to room temperature. 1-Phenyl-2-(pyridin-2-yl)ethanone (1a, 1 equiv, 1 mmol, 197.1 mg) was added and the reaction mixture was heated to reflux for 20 hours. The reaction was then treated with Et₂O (5 mL), concentrated under reduced pressure to a residue that was purified by flash column chromatography (1:1 EtOAc/petroleum ether 40-60°) to afford the title compound (203.5 mg, 92% yield).
3. Compound Characterisation Data

**Compound 1a. 1-Phenyl-2-(pyridin-2-yl)ethanone**

Appearance: Orange solid

$\nu_{\text{max}}$ (solid): 3051, 2922, 1680, 1595, 1543, 1491, 1465 cm$^{-1}$. Approx. 3:1 keto:enol and approx. 3.8:1 enol olefin stereoisomers based on $^1$H NMR taken in CDCl$_3$ at 298 K.$^2$

For $^1$H and $^{13}$C NMR spectra, see Section 3.5.

HRMS (C$_{13}$H$_{12}$NO) [M+H$^+$] requires 198.0919, found [M+H$^+$] 198.0913.

**Compound 1b. 1-(Pyridin-2-yl)heptan-2-one**

Appearance: Yellow oil.

$\nu_{\text{max}}$ (neat): 2959, 2926, 1709, 1636, 1585, 1464 cm$^{-1}$. Approx. 5.6:1 keto:enol and approx. 1.5:1 enol olefin stereoisomers based on $^1$H NMR taken in CDCl$_3$ at 298 K.$^2$

For $^1$H and $^{13}$C NMR spectra, see Section 3.5.

HRMS (C$_{12}$H$_{18}$NO) [M+H$^+$] requires 192.1388, found [M+H$^+$] 192.1381.

**Compound 1c. 3-Methyl-1-(pyridin-2-yl)butan-2-one**

Appearance: Orange oil.

$\nu_{\text{max}}$ (neat): 2957, 2928, 2870, 1709, 1636, 1585, 1475 cm$^{-1}$. Approx. 12.5:1 keto:enol and approx. 7:1 enol olefin stereoisomers based on $^1$H NMR taken in CDCl$_3$ at 298 K.$^2$

For $^1$H and $^{13}$C NMR spectra, see Section 3.5.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 8.55 (d, 1H, $J = 4.9$ Hz, keto), 8.47 (d, 1H, $J = 5.6$ Hz, enol), 8.38 (d, 1H, $J = 5.8$, enol), 8.21 (d, 1H, $J = 7.5$ Hz, enol).

$^{13}$C NMR (400 MHz, CDCl$_3$): $\delta$

HRMS (C$_{10}$H$_{14}$NO) [M+H$^+$] requires 164.1075, found [M+H$^+$] 164.1070.

**Compound 1d. 1-Cyclobutyl-2-(pyridin-2-yl)ethanone**

Appearance: Yellow oil.

$\nu_{\text{max}}$ (neat): 2982, 2943, 1705, 1643, 1591, 1474, 1435 cm$^{-1}$. Approx. 5:1 keto:enol and approx. 1:1 enol olefin stereoisomers based on $^1$H NMR taken in CDCl$_3$ at 298 K.$^2$

For $^1$H and $^{13}$C NMR spectra, see Section 3.5.

HRMS (C$_{11}$H$_{16}$NO) [M+H$^+$] requires 176.1075, found [M+H$^+$] 176.1069.
Compound 1e. 1-(1-Benzylpiperidin-4-yl)-2-(pyridin-2-yl)ethanone

Appearance: Brown oil.

$\nu_{\text{max}}$ (neat): 2940, 2801, 2758, 1709, 1641, 1595, 1551, 1474 cm$^{-1}$.

Approx. 8.3:1 keto:enol and only one olefin stereoisomer based on $^1$H NMR taken in CDCl$_3$ at 298 K.\(^2\)

For $^1$H and $^{13}$C NMR spectra, see Section 3.5.

HRMS (C$_{19}$H$_{23}$N$_2$O) [M+H$^+$] requires 295.1810, found [M+H$^+$] 295.1805.

Compound 1f. 1-(4-Fluorophenyl)-2-(pyridin-2-yl)ethanone

Appearance: Yellow solid.

$\nu_{\text{max}}$ (solid): 2922, 1632, 1595, 1506, 1456 cm$^{-1}$.

Approx. 2:1 keto:enol and approx. 1.7:1 enol olefin stereoisomers based on $^1$H NMR taken in CDCl$_3$ at 298 K.\(^2\)

For $^1$H and $^{13}$C NMR spectra, see Section 3.5.

HRMS (C$_{13}$H$_{11}$FNO) [M+H$^+$] requires 216.0825, found [M+H$^+$] 216.0816.

Compound 1g. 1-(4-Chlorophenyl)-2-(pyridin-2-yl)ethanone

Appearance: Yellow powder.

$\nu_{\text{max}}$ (solid): 3088, 3026, 1626, 1589, 1549, 1454, 1406 cm$^{-1}$.

Approx. 1.3:1 keto:enol and approx. 7.6:1 enol olefin stereoisomers based on $^1$H NMR taken in CDCl$_3$ at 298 K.\(^2\)

For $^1$H and $^{13}$C NMR spectra, see Section 3.5.

HRMS (C$_{13}$H$_{11}$ClNO) [M+H$^+$] requires 232.0529, found [M+H$^+$] 232.0525.

Compound 1h. 1-(4-Bromophenyl)-2-(pyridin-2-yl)ethanone

Appearance: Orange powder.

$\nu_{\text{max}}$ (solid): 2922, 1626, 1585, 1547, 1450, 1404 cm$^{-1}$.

Approx. 1.2:1 keto:enol and approx. 3.5:1 enol olefin stereoisomers based on $^1$H NMR taken in CDCl$_3$ at 298 K.\(^2\)

For $^1$H and $^{13}$C NMR spectra, see Section 3.5.

HRMS (C$_{13}$H$_{11}$BrNO) [M+H$^+$] requires 276.0024, found [M+H$^+$] 276.0020.
**Compound 1j. 2-(Pyridin-2-yl)-1-(α-tolyl)ethanone**

Appearance: Red solid.

\(\nu_{\text{max}}\text{ (solid): } 3053, 2924, 1665, 1579, 1568, 1468, 1454, 1431 \text{ cm}^{-1}\).

Approx. 2:4:1 keto:enol and only one enol olefin stereoisomer based on \(^1\text{H} \text{ NMR taken in CDCl}_3 \text{ at 298 K}\). For \(^1\text{H} \text{ and } ^{13}\text{C} \text{ NMR spectra, see Section 3.5.}\)

HRMS (C\(_{14}\)H\(_{14}\)NO) [M+H\(^+\)] requires 212.1075, found [M+H\(^+\)] 212.1068.

**Compound 1k. 1-(4-(n-Butyl)phenyl)-2-(pyridin-2-yl)ethanone**

Appearance: Yellow solid.

\(\nu_{\text{max}}\text{ (solid): } 2953, 2928, 2857, 1681, 1628, 1595, 1547, 1464 \text{ cm}^{-1}\).

Approx. 2:1 keto:enol and approx. 2.2:1 enol olefin stereoisomers based on \(^1\text{H} \text{ NMR taken in CDCl}_3 \text{ at 298 K}\). For \(^1\text{H} \text{ and } ^{13}\text{C} \text{ NMR spectra, see Section 3.5.}\)

HRMS (C\(_{17}\)H\(_{20}\)NO) [M+H\(^+\)] requires 254.1545, found [M+H\(^+\)] 254.1538.

**Compound 1l. 1-(4-Methoxyphenyl)-2-(pyridin-2-yl)ethanone**

Appearance: Yellow solid.

\(\nu_{\text{max}}\text{ (solid): } 3011, 1624, 1591, 1547, 1510 \text{ cm}^{-1}\).

Approx. 7:1:1 keto:enol and approx. 16:1 enol olefin stereoisomers based on \(^1\text{H} \text{ NMR taken in CDCl}_3 \text{ at 298 K}\). For \(^1\text{H} \text{ and } ^{13}\text{C} \text{ NMR spectra, see Section 3.5.}\)

HRMS (C\(_{14}\)H\(_{14}\)NO\(_2\)) [M+H\(^+\)] requires 228.1025, found [M+H\(^+\)] 228.1021.

**Compound 1m. 1-(4-((tert-Butyldimethylsilyl)oxy)phenyl)-2-(pyridin-2-yl)ethanone**

Appearance: Yellow solid.

\(\nu_{\text{max}}\text{ (solid): } 3055, 2934, 1636, 1599, 1574, 1513, 1466 \text{ cm}^{-1}\).

Approx. 8:3:1 keto:enol and only one enol olefin stereoisomer based on \(^1\text{H} \text{ NMR taken in CDCl}_3 \text{ at 298 K}\). For \(^1\text{H} \text{ and } ^{13}\text{C} \text{ NMR spectra, see Section 3.5.}\)

HRMS (C\(_{19}\)H\(_{26}\)NO\(_2\)Si) [M+H\(^+\)] requires 328.1733, found [M+H\(^+\)] 328.1725.
**Compound 1n. 1-(4-(Dibenzylamino)phenyl)-2-(pyridin-2-yl)ethanone**

Appearance: Brown oil.

$\nu_{\text{max}}$ (neat): 3024, 2914, 1661, 1593, 1555, 1522, 1493, 1450 cm$^{-1}$.

Approx. 20:1 keto:enol and approx. 1.5:1 enol olefin stereoisomers based on $^1$H NMR taken in CDCl$_3$ at 298 K.$^2$

For $^1$H and $^{13}$C NMR spectra, see Section 3.5.

HRMS (C$_{27}$H$_{25}$N$_2$O) $[\text{M+H}^+]$ requires 393.1967, found $[\text{M+H}^+]$ 393.1958.

**Compound 1o. 1-(Furan-2-yl)-2-(pyridin-2-yl)ethanone**

Appearance: Brown solid.

$\nu_{\text{max}}$ (solid): 3129, 3049, 3007, 1670, 1589, 1564, 1493, 1433 cm$^{-1}$.

Approx. 4.5:1 keto:enol and only one enol olefin stereoisomer based on $^1$H NMR taken in CDCl$_3$ at 298 K.$^2$

For $^1$H and $^{13}$C NMR spectra, see Section 3.5.

HRMS (C$_{11}$H$_{10}$NO) $[\text{M+H}^+]$ requires 188.0712, found $[\text{M+H}^+]$ 188.0705.

**Compound 3a. 2-Phenyl-4$H$-quinolizin-4-one**

Appearance: Yellow crystalline solid.

$\nu_{\text{max}}$ (solid): 3121, 3057, 1651, 1626, 1543, 1466, 1429, 1408 cm$^{-1}$.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 9.13 (d, 1H, $J = 7.4$ Hz), 7.70-7.72 (m, 2H), 7.44-7.54 (m, 4H), 7.34-7.38 (m, 1H), 6.99-7.03 (m, 1H), 6.91 (s, 2H).

$^{13}$C NMR (400 MHz, CDCl$_3$): $\delta$ 158.7, 150.5, 142.3, 138.4, 129.7, 129.3, 129.0, 127.3, 125.7, 114.9, 106.6, 102.2.

HRMS (C$_{15}$H$_{12}$NO) $[\text{M+H}^+]$ requires 222.0919, found $[\text{M+H}^+]$ 222.0912.

**Compound 3b. 2-Pentyl-4$H$-quinolizin-4-one**

Appearance: Brown oil.

$\nu_{\text{max}}$ (neat): 2965, 2932, 2970, 1709, 1636, 1597, 1553, 1475, 1435 cm$^{-1}$.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 9.04 (d, 1H, $J = 8.0$ Hz), 7.37 (d, 1H, $J = 8.9$ Hz), 7.24-7.28 (m, 1H), 6.90 (ddd, 1H, $J = 7.70, 6.60, 1.4$ Hz), 6.49 (d, 2H, $J = 2.1$ Hz), 2.61 (t, 2H, $J = 8.0$ Hz), 1.64-1.72 (m, 2H), 1.31-1.37 (m, 4H), 0.90 (t, 3H, $J = 7.0$ Hz).

$^{13}$C NMR (400 MHz, CDCl$_3$): $\delta$ 158.9, 154.8, 142.1, 129.4, 127.4, 125.3, 114.5, 109.0, 104.3, 36.2, 31.7, 30.0, 22.8, 14.2.
HRMS (C_{14}H_{18}NO) [M+H^+] requires 216.1388, found [M+H^+] 216.1380.

**Compound 3c. 2-Isopropyl-4H-quinolizin-4-one**

Appearance: Yellow crystalline solid.

$\nu_{\text{max}}$ (solid): 2965, 2870, 1708, 1533, 1476, 1435 cm$^{-1}$.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 9.03 (d, 1H, $J = 7.4$ Hz), 7.39 (d, 1H, $J = 8.9$ Hz), 7.26 (m, 1H), 6.91 (m, 1H), 6.54 (s, 2H), 2.89 (dt, 1H, $J = 13.8, 6.9$ Hz), 1.29 (d, $J = 6.9$ Hz, 6H).

$^{13}$C NMR (400 MHz, CDCl$_3$): $\delta$ 160.2, 158.9, 142.0, 129.0, 127.0, 125.2, 114.2, 106.9, 102.4, 34.3, 22.9.

HRMS (C$_{12}$H$_{14}$NO) [M+H$^+$] requires 188.1075, found [M+H$^+$] 188.1069.

**Compound 3d. 2-Cyclobutyl-4H-quinolizin-4-one**

Appearance: Brown oil.

$\nu_{\text{max}}$ (neat): 3109, 3061, 2960, 2935, 2864, 1659, 1627, 1560, 1547, 1413 cm$^{-1}$.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 9.01 (d, 1H, $J = 7.4$ Hz), 7.37 (d, 1H, $J = 8.9$ Hz), 7.22-7.26 (m, 1H), 6.88 (m, 1H), 6.48 (s, 2H), 3.53 (pent, 1H, $J = 8.7$ Hz), 2.32-2.40 (m, 2H), 1.98-2.08 (m, 1H), 1.84-1.92 (m, 1H).

$^{13}$C NMR (400 MHz, CDCl$_3$): $\delta$ 158.7, 157.5, 141.9, 129.0, 127.0, 125.0, 114.2, 106.4, 101.9, 39.8, 28.7, 18.1.

HRMS (C$_{13}$H$_{14}$NO) [M+H$^+$] requires 200.1075, found [M+H$^+$] 200.1069.

**Compound 3e. 2-(1-Benzylpiperidin-4-yl)-4H-quinolizin-4-one**

Appearance: Brown oil.

$\nu_{\text{max}}$ (neat): 2936, 2797, 2755, 1652, 1629, 1452 cm$^{-1}$.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 9.03 (dd, 1H, $J = 7.3, 0.6$ Hz), 7.24-7.39 (m, 7H), 6.89-6.93 (m, 1H), 6.52-6.55 (m, 2H), 3.56 (s, 2H), 3.04 (d, 2H, $J = 11.8$ Hz), 2.47-2.55 (m, 1H), 2.08-2.14 (m, 2H), 1.77-1.86 (m, 4H).

$^{13}$C NMR (400 MHz, CDCl$_3$): $\delta$ 158.8, 157.7, 142.0, 138.2, 129.2, 129.1, 128.2, 127.0, 125.2, 114.3, 107.4, 102.4, 63.3, 53.8, 42.6, 32.4.

HRMS (C$_{21}$H$_{22}$N$_2$O) [M+H$^+$] requires 319.1810, found [M+H$^+$] 319.1802.

**Compound 3f. 2-(4-Fluorophenyl)-4H-quinolizin-4-one**
Appearance: Yellow crystalline solid.

$\nu_{\text{max}}$ (solid): 3123, 2922, 1653, 1626, 1599, 1562, 1506, 1454 cm$^{-1}$.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 9.12 (d, 1H, $J = 7.3$ Hz), 7.66-7.70 (m, 2H), 7.53 (d, 1H, $J = 8.9$ Hz), 7.35-7.39 (m, 1H), 7.17-7.21 (m, 2H), 6.99-7.03 (m, 1H), 6.85 (d, 2H, $J = 3.5$ Hz).

$^{13}$C NMR (400 MHz, CDCl$_3$): $\delta$ 163.8 (d, $^1J_{C-F} = 249.4$ Hz), 162.6, 158.8, 149.5, 142.6, 134.0 (d, $^4J_{C-F} = 2.9$ Hz), 128.5 (d, $^3J_{C-F} = 8.5$ Hz), 127.5, 125.8, 116.4, 115.5 (d, $^2J_{C-F} = 21.6$ Hz), 106.5, 102.0.

HRMS (C$_{15}$H$_{11}$FNO) [M+H$^+$] requires 240.0825, found [M+H$^+$] 240.0818.

**Compound 3g. 2-(4-Chlorophenyl)-4H-quinolizin-4-one**

Appearance: Yellow powder.

$\nu_{\text{max}}$ (solid): 3105, 2928, 1651, 1620, 1558, 1494, 1454, 1435, 1414 cm$^{-1}$.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 9.12 (d, 1H, $J = 7.3$ Hz), 7.62-7.64 (m, 2H), 7.56-7.58 (m, 2H), 7.53 (d, 1H, $J = 8.9$ Hz), 7.46-7.49 (m, 2H), 7.36-7.40 (m, 1H), 7.00-7.04 (m, 1H), 6.85 (d, 2H, $J = 3.8$ Hz).

$^{13}$C NMR (400 MHz, CDCl$_3$): $\delta$ 158.9, 149.5, 142.8, 137.2, 135.8, 130.2, 129.6, 127.6, 126.0, 115.3, 106.7, 102.0.

HRMS (C$_{15}$H$_{11}$ClNO) [M+H$^+$] requires 256.0529, found [M+H$^+$] 256.0524.

**Compound 3h. 2-(4-Bromophenyl)-4H-quinolizin-4-one**

Appearance: Yellow solid.

$\nu_{\text{max}}$ (solid): 3105, 2927, 1651, 1620, 1558, 1495, 1454, 1435, 1414 cm$^{-1}$.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 9.13 (d, 1H, $J = 7.3$ Hz), 7.62-7.64 (m, 2H), 7.56-7.58 (m, 2H), 7.53 (d, 1H, $J = 8.9$ Hz), 7.36-7.40 (m, 1H), 7.00-7.04 (m, 1H), 6.83-6.85 (m, 2H).

$^{13}$C NMR (400 MHz, CDCl$_3$): $\delta$ 159.0, 149.6, 142.9, 137.8, 132.6, 130.3, 129.1, 127.8, 126.0, 124.1, 115.3, 106.7, 102.0.

HRMS (C$_{15}$H$_{11}$BrNO) [M+H$^+$] requires 300.0024, found [M+H$^+$] 300.0020.

**Compound 3j. 2-(o-Tolyl)-4H-quinolizin-4-one**

Appearance: Brown oil.

$\nu_{\text{max}}$ (neat): 3061, 3016, 1655, 1628, 1560, 1547, 1489, 1439, 1406 cm$^{-1}$.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 9.13 (dd, 1H, $J = 7.4$ Hz), 7.46 (d, 1H, $J = 8.9$ Hz), 7.25-7.36 (m, 5H), 7.00 (ddd, 1H, $J =$ 7.7, 6.6, 1.4 Hz), 6.61 (m, 2H), 2.33 (s, 3H).
$^{13}$C NMR (400 MHz, CDCl$_3$): $\delta$ 158.3, 152.2, 141.7, 139.5, 134.9, 130.5, 129.5, 128.8, 128.3, 127.1, 125.9, 125.4, 114.8, 109.4, 104.6, 20.2.

HRMS (C$_{16}$H$_{14}$NO) [M+H$^+$] requires 236.1075, found [M+H$^+$] 236.1068.

**Compound 3k. 2-(4-(n-Butyl)phenyl)-4H-quinolizin-4-one**

Appearance: Brown solid.

$\nu_{\text{max}}$ (solid): 2955, 2928, 2856, 1735, 1681, 1628, 1562, 1545, 1512, 1454 cm$^{-1}$.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 9.04 (d, 1H, $J$ = 7.5 Hz), 8.67 (d, 1H, $J$ = 4.7 Hz), 7.74 (d, 1H, $J$ = 7.7 Hz), 7.38-7.42 (m, H), 7.12 (d, 2H, $J$ = 8.3 Hz), 6.91 (t, 1H, $J$ = 7.2 Hz), 6.60 (s, 1H), 6.50 (s, 1H), 2.57 (m, 2H), 1.28-1.38 (m, 4H), 0.90 (t, 3H, $J$ = 7.3 Hz).

$^{13}$C NMR (400 MHz, CDCl$_3$): $\delta$ 159.1, 150.6, 144.8, 142.6, 135.9, 129.8, 129.4, 127.5, 127.4, 125.9, 121.0, 114.9, 106.6, 102.2, 35.7, 33.8, 22.7, 14.2.

HRMS (C$_{19}$H$_{20}$NO) [M+H$^+$] requires 278.1545, found [M+H$^+$] 278.1540.

**Compound 3l. 2-(4-Methoxyphenyl)-4H-quinolizin-4-one**

Appearance: Yellow solid.

$\nu_{\text{max}}$ (solid): 3115, 2994, 2839, 1651, 1620, 1560, 1545, 1510, 1442, 1418 cm$^{-1}$.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 9.09 (d, 1H, $J$ = 7.4 Hz), 7.55-7.62 (m, 2H), 7.49 (d, 1H, $J$ = 8.9 Hz), 7.30-7.34 (m, 1H), 6.94-6.98 (m, 2H), 6.87 (s, 2H), 3.88 (s, 3H).

$^{13}$C NMR (400 MHz, CDCl$_3$): $\delta$ 161.1, 159.1, 150.6, 144.8, 142.6, 135.9, 129.8, 129.4, 127.5, 127.4, 125.9, 115.0, 114.8, 106.2, 102.1, 55.8.

HRMS (C$_{16}$H$_{14}$NO$_2$) [M+H$^+$] requires 252.1025, found [M+H$^+$] 252.1018.

**Compound 3m. 2-(4-((tert-Butyldimethylsilyl)oxy)phenyl)-4H-quinolizin-4-one**

Appearance: Brown solid.

$\nu_{\text{max}}$ (solid): 2930, 2857, 1736, 1649, 1603, 1552, 1512, 1454 cm$^{-1}$.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 9.09 (d, 1H, $J$ = 7.3 Hz), 7.58-7.62 (m, 2H), 7.49 (d, 1H, $J$ = 8.9 Hz), 7.30-7.34 (m, 1H), 6.94-6.98 (m, 3H), 6.86 (s, 2H), 1.02 (s, 9H), 0.26 (s, 6H).

$^{13}$C NMR (400 MHz, CDCl$_3$): $\delta$ 159.1, 157.5, 150.4, 142.6, 131.5, 129.9, 128.8, 127.6, 125.9, 121.0, 114.9, 106.3, 102.2, 26.0, 18.6, 4.0.
HRMS (C_{16}H_{15}NO_{2}Si) [M-(Me + t-Bu)+2H] requires 281.0872, found [M-(Me + t-Bu)+2H] 281.1515 (fragment shown below).

HRMS fragment of compound 3n.

**Compound 3n. 2-(4-(Dibenzylamino)phenyl)-4H-quinolizin-4-one**

Appearance: Brown/yellow solid.

$\nu_{\text{max}}$(solid): 3061, 2913, 2862, 1655, 1603, 1518, 1450 cm$^{-1}$.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 9.05 (d, 1H, $J = 7.3$ Hz), 7.57-7.60 (m, 2H), 7.45 (d, 1H, $J = 8.9$ Hz), 7.35-7.39 (m, 4H), 7.26-7.32 (m, 7H), 6.82-6.94 (m, 5H), 4.74 (s, 4H).

$^{13}$C NMR (400 MHz, CDCl$_3$): $\delta$ 150.5, 150.3, 142.5, 138.1, 129.7, 129.2, 128.6, 127.5, 126.9, 125.9, 120.0, 114.6, 112.9, 105.2, 54.5.

HRMS (C$_{29}$H$_{25}$N$_2$O) [M+H$^+$] requires 417.1967, found [M+H$^+$] 417.1961.

**Compound 3o. 2-(Furan-2-yl)-4H-quinolizin-4-one**

Appearance: Brown solid.

$\nu_{\text{max}}$(solid): 3102, 2957, 2822, 2853, 1736, 1651, 1618, 1593, 1460 cm$^{-1}$.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 9.08 (d, 1H, $J = 7.4$ Hz), 7.59 (d, 1H, $J = 1.2$ Hz), 7.48 (d, 1H, $J = 8.9$ Hz), 7.30-7.34 (m, 1H), 6.90-6.97 (m, 4H), 6.56 (dd, 1H, $J = 3.4, 1.8$ Hz).

$^{13}$C NMR (400 MHz, CDCl$_3$): $\delta$ 159.0, 151.7, 144.6, 143.0, 139.4, 130.1, 127.8, 125.9, 114.9, 112.6, 110.2, 102.6, 98.9.

HRMS (C$_{13}$H$_{10}$NO$_2$) [M+H$^+$] requires 212.0712, found [M+H$^+$] 212.0704.

**Compound 5a. N-Methoxy-N-methylbenzamide**

Appearance: Colourless oil.

$\nu_{\text{max}}$(neat): 2970, 1638, 1447, 1412 cm$^{-1}$.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.67-7.70 (m, 2H), 7.41-7.46 (m, 3H), 3.57 (s, 3H), 3.37 (s, 3H).

$^{13}$C NMR (400 MHz, CDCl$_3$): $\delta$ 169.9, 134.1, 130.5, 128.1, 128.0, 61.0, 33.7.
HRMS (C₉H₁₂NO₂) [M+H⁺] requires 166.0868, found [M+H⁺] 166.0863.

**Compound 5b. N-Methoxy-N-methylhexanamide**

Appearance: Colourless oil.

ν<sub>max</sub> (neat): 2955, 2934, 2860, 1637, 1462, 1443, 1412 cm⁻¹.

¹H NMR (400 MHz, CDCl₃): δ 3.69 (s, 3H), 3.18 (s, 3H), 2.41 (t, 2H, J = 7.6 Hz), 1.60-1.68 (m, 2H), 1.29-1.38 (m, 4H), 0.91 (t, 3H, J = 7.0 Hz).

¹³C NMR (100 MHz, CDCl₃): δ 174.5, 60.8, 31.8, 31.5, 31.3, 24.0, 22.1, 13.6.

HRMS (C₈H₁₈NO₂) [M+H⁺] requires 160.1338, found [M+H⁺] 160.1331.

**Compound 5c. N-Methoxy-N-methylisobutyramide**

Appearance: Colourless oil.

ν<sub>max</sub> (neat): 2961, 1732, 1483, 2870, 1647, 1460, 1404 cm⁻¹.

¹H NMR (400 MHz, CDCl₃): δ 3.69 (s, 3H), 3.18 (s, 3H), 2.93-3.00 (m, 1H), 1.13 (d, 6H, J = 6.9 Hz).

¹³C NMR (400 MHz, CDCl₃): δ 178.9, 60.3, 39.1, 33.4, 26.9.

HRMS (C₆H₁₄NO₂) [M+H⁺] requires 132.1025, found [M+H⁺] 132.1018.

**Compound 5d. N-Methoxy-N-methylcyclobutanecarboxamide**

Appearance: Colourless oil.

ν<sub>max</sub> (neat): 2943, 2870, 1674, 1462, 1443, 1414 cm⁻¹.

¹H NMR (400 MHz, CDCl₃): δ 3.58 (s, 3H), 3.41 (br m, 1H), 3.10 (s, 3H), 2.20-2.30 (m, 2H), 2.02-2.10 (m, 2H), 1.85-1.96 (m, 1H), 1.74-1.84 (m, 1H).

¹³C NMR (400 MHz, CDCl₃): δ 175.0, 174.0, 61.1, 35.9, 32.1, 24.7, 18.5.

HRMS (C₇H₁₄NO₂) [M+H⁺] requires 144.1025, found [M+H⁺] 144.1020.

**Compound 5e. 1-Benzyl-N-methoxy-N-methylpiperidine-4-carboxamide**

Appearance: Orange oil.

ν<sub>max</sub> (neat): 2947, 2754, 1651, 1447 cm⁻¹.

¹H NMR (400 MHz, CDCl₃): δ 7.32-7.34 (m, 3H), 7.24-7.30 (m, 2H), 3.70 (s, 3H), 3.10 (s, 3H), 2.20-2.30 (m, 2H), 2.02-2.10 (m, 2H), 1.85-1.96 (m, 1H), 1.74-1.84 (m, 1H).

¹³C NMR (400 MHz, CDCl₃): δ 176.4, 138.4, 129.0, 128.1, 126.9, 63.2, 61.5, 53.1, 38.2, 32.3, 28.3.

HRMS (C₁₅H₂₃N₂O₂) [M+H⁺] requires 263.1760, found [M+H⁺] 263.1751.
Compound 5f. 4-Fluoro-N-methoxy-N-methylbenzamide

Appearance: Colourless oil.

$\nu_{\text{max}}$ (neat): 2970, 2938, 1637, 1603, 1506, 1460, 1441, 1418 cm$^{-1}$.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.73-7.76 (m, 2H), 7.07-7.11 (m, 2H), 3.54 (s, 3H), 3.37 (s, 3H).

$^{13}$C NMR (400 MHz, CDCl$_3$): 164.4 (d, $^1$$J_{C-F} = 250.9$ Hz), 162.8, 131.2 (d, $^3$$J_{C-F} = 8.5$ Hz), 130.2 (d, $^4$$J_{C-F} = 3.0$ Hz), 115.4 (d, $^2$$J_{C-F} = 21.9$ Hz), 61.0, 33.6.

HRMS (C$_9$H$_{11}$FNO$_2$) [M+H$^+$] requires 184.0774, found [M+H$^+$] 184.0767.

Compound 5g. 4-Chloro-N-methoxy-N-methylbenzamide

Appearance: Pale yellow oil.

$\nu_{\text{max}}$ (neat): 2968, 1638, 1593, 1458, 1442, 1414 cm$^{-1}$.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.66 (d, 2H, $J = 8.6$ Hz), 7.38 (d, 2H, $J = 8.6$ Hz), 3.54 (s, 3H), 3.36 (s, 3H).

$^{13}$C NMR (400 MHz, CDCl$_3$): $\delta$ 168.6, 136.7, 132.3, 129.8, 128.2, 61.0, 33.5.

HRMS (C$_9$H$_{11}$ClNO$_2$) [M+H$^+$] requires 200.0478, found [M+H$^+$] 200.0474.

Compound 5h. 4-Bromo-N-methoxy-N-methylbenzamide

Appearance: Pale yellow oil.

$\nu_{\text{max}}$ (neat): 2968, 2934, 1635, 1587, 1458, 1412 cm$^{-1}$.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 7.59 (d, 2H, $J = 8.7$ Hz), 7.55 (d, 2H, $J = 8.7$ Hz), 3.54 (s, 3H), 3.36 (s, 3H).

$^{13}$C NMR (400 MHz, CDCl$_3$): $\delta$ 168.7, 132.8, 131.2, 130.0, 125.1, 61.1, 33.5.

HRMS (C$_9$H$_{11}$BrNO$_2$) [M+H$^+$] requires 243.9973, found [M+H$^+$] 243.9971.

Compound 5i. N-Methoxy-N-methyl-4-nitrobenzamide

Appearance: Colourless oil.

$\nu_{\text{max}}$ (neat): 3115, 2986, 2930, 1634, 1595, 1524, 1423 cm$^{-1}$.

$^1$H NMR (400 MHz, CDCl$_3$): $\delta$ 8.27 (d, 2H, $J = 8.9$ Hz), 7.84 (d, 2H, $J = 8.9$ Hz), 3.53 (s, 3H), 3.40 (s, 3H).

$^{13}$C NMR (400 MHz, CDCl$_3$): $\delta$ 167.7, 148.8, 140.0, 129.2, 123.2, 61.4, 33.1.

HRMS (C$_9$H$_{10}$N$_2$O$_4$) [M+H$^+$] requires 211.0719, found [M+H$^+$] 211.0713.
Compound 5j. \( \text{N-Methoxy-}N,2\text{-dimethylbenzamide} \)

\[
\begin{array}{c}
\text{Me} \\
\text{O} \\
\text{N} \\
\text{OMe}
\end{array}
\]

Appearance: Pale yellow oil.

\( \nu_{\text{max}} \) (neat): 2968, 2934, 1668, 1459, 1410 cm\(^{-1}\).

\( ^1H \) NMR (400 MHz, CDCl\(_3\)): \( \delta \) 7.18-7.31 (m, 4H), 3.52 (br s, 3H), 3.30 (s, 3H), 2.34 (s, 3H).

\( ^13C \) NMR (400 MHz, CDCl\(_3\)): \( \delta \) 170.6, 135.6, 135.0, 130.2, 129.2, 126.4, 125.5, 61.1, 33.5, 19.1.

HRMS (C\(_{10}\)H\(_{14}\)NO\(_2\)) [M+H\(^+\)] requires 180.1025, found [M+H\(^+\)] 180.1018.

Compound 5k. 4-(\(n\)-Butyl)-N-methoxy-\(N\)-methylbenzamide

\[
\begin{array}{c}
\text{n-Bu} \\
\text{O} \\
\text{N} \\
\text{OMe}
\end{array}
\]

Appearance: Colourless oil.

\( \nu_{\text{max}} \) (neat): 2957, 2930, 1639, 1611, 1458, 1416 cm\(^{-1}\).

\( ^1H \) NMR (400 MHz, CDCl\(_3\)): \( \delta \) 7.61 (d, 2H, \( J = 8.2 \) Hz), 7.21 (d, 2H, \( J = 8.3 \) Hz), 3.57 (s, 3H), 3.35 (s, 3H), 2.64 (t, 2H, \( J = 8.0 \) Hz), 1.58-1.65 (m, 2H), 1.32-1.41 (m, 2H), 0.93 (t, 3H, \( J = 7.3 \) Hz).

\( ^13C \) NMR (400 MHz, CDCl\(_3\)): \( \delta \) 169.9, 145.7, 131.2, 128.2, 127.9, 60.8, 35.4, 33.8, 33.2, 22.2, 13.8.

HRMS (C\(_{13}\)H\(_{20}\)NO\(_2\)) [M+H\(^+\)] requires 222.1494, found [M+H\(^+\)] 222.1488.

Compound 5l. 4-Methoxy-N-methoxy-\(N\)-methylbenzamide

\[
\begin{array}{c}
\text{Me} \\
\text{O} \\
\text{N} \\
\text{OMe}
\end{array}
\]

Appearance: Pale yellow oil.

\( \nu_{\text{max}} \) (neat): 2965, 2934, 1632, 1605, 1510, 1460, 1420 cm\(^{-1}\).

\( ^1H \) NMR (400 MHz, CDCl\(_3\)): \( \delta \) 7.74 (d, 2H, \( J = 8.9 \) Hz), 6.91 (d, 2H, \( J = 8.9 \) Hz), 3.85 (s, 3H), 3.57 (s, 3H), 3.35 (s, 3H).

\( ^13C \) NMR (400 MHz, CDCl\(_3\)): \( \delta \) 169.3, 161.5, 130.5, 126.0, 113.2, 60.8, 55.2, 33.8.

HRMS (C\(_{10}\)H\(_{14}\)NO\(_3\)) [M+H\(^+\)] requires 196.0974, found [M+H\(^+\)] 196.0968.

Compound 5m. 4-((\(\text{tert-Butyldimethylsilyl}\))oxy)-\(N\)-methoxy-\(N\)-methylbenzamide

\[
\begin{array}{c}
\text{TBSO} \\
\text{O} \\
\text{N} \\
\text{OMe}
\end{array}
\]

Appearance: Colourless oil.

\( \nu_{\text{max}} \) (neat): 2955, 2930, 2859, 1639, 1603, 1506, 1462, 1415 cm\(^{-1}\).

\( ^1H \) NMR (400 MHz, CDCl\(_3\)): \( \delta \) 7.65 (d, 2H, \( J = 8.7 \) Hz), 6.85 (d, 2H, \( J = 8.7 \) Hz), 3.56 (s, 3H), 3.35 (s, 3H), 0.99 (s, 9H), 0.22 (s, 6H).

\( ^13C \) NMR (100 MHz, CDCl\(_3\)): \( \delta \) 169.5, 158.0, 130.3, 126.7, 119.4, 60.8, 33.9, 25.6, 18.1, -4.5.

HRMS (C\(_{15}\)H\(_{26}\)NO\(_3\)Si) [M+H\(^+\)] requires 296.1682, found [M+H\(^+\)] 296.1677.
Compound 5n. 4-(Dibenzylamino)-N-methoxy-N-methylbenzamide

Appearance: Colourless oil.

\[ \text{\( \nu_{max} \) (neat): 3030, 2932, 1620, 1522, 1445 cm}^{-1} \].

\( ^1\text{H NMR (400 MHz, CDCl}_3 \)): \( \delta \) 7.66 (d, 2H, \( J = 9.1 \text{ Hz} \)), 7.33-7.37 (m, 4H), 7.28-7.3 (m, 2H), 7.23-7.25 (m, 4H), 6.72 (d, 2H, \( J = 9.0 \text{ Hz} \)), 4.71 (s, 4H), 3.60 (s, 3H), 3.34 (s, 3H).

\( ^{13}\text{C NMR (400 MHz, CDCl}_3 \)): \( \delta \) 169.8, 151.1, 137.7, 130.7, 128.7, 127.1, 126.5, 121.2, 111.1, 60.7, 53.9, 34.2.

HRMS (C\(_{22}\)H\(_{25}\)N\(_2\)O\(_2\)) [M+H\(^+\)] requires 361.1916, found [M+H\(^+\)] 361.1911.

Compound 5o. N-Methoxy-N-methylfuran-2-carboxamide

Appearance: Brown oil.

\[ \text{\( \nu_{max} \) (neat): 3119, 2968, 2938, 1627, 1560, 1407 cm}^{-1} \].

\( ^1\text{H NMR (400 MHz, CDCl}_3 \)): \( \delta \) 7.59 (d, 1H, \( J = 1.5 \text{ Hz} \)), 7.14 (d, 1H, \( J = 3.5 \text{ Hz} \)), 6.51 (dd, 1H, \( J = 3.5, 1.7 \text{ Hz} \)), 3.77 (s, 3H), 3.35 (s, 3H).

\( ^{13}\text{C NMR (400 MHz, CDCl}_3 \)): \( \delta \) 159.1, 145.7, 145.2, 117.3, 111.5, 61.3, 33.1.

HRMS (C\(_7\)H\(_{10}\)NO\(_3\)) [M+H\(^+\)] requires 156.0661, found [M+H\(^+\)] 156.0654.
3.5 IR, $^1$H NMR, $^{13}$C NMR, and HRMS Spectra for Compounds

Compound 1a: 1-Phenyl-2-(pyridin-2-yl)ethanone

$^1$H NMR:

$^{13}$C NMR:
FTIR:

HRMS:

AW027 #63  RT: 1.57  AV: 1  NL: 1.21E8
F: FTMS + c ESI Full ms [75.00-1200.00]
Compound 1b: 1-(Pyridin-2-yl)heptan-2-one

$^1$H NMR:

$^{13}$C NMR:
**FTIR:**

[Image of FTIR spectrum]

**HRMS:**

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<th>RT: 1.44</th>
<th>AV: 1</th>
<th>NL: 1.95E8</th>
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</tr>
</tbody>
</table>
Compound 1c: 3-Methyl-1-(pyridin-2-yl)butan-2-one

$^1$H NMR:

$^{13}$C NMR:
FTIR:

HRMS:

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Compound 1d: 1-Cyclobutyl-2-(pyridin-2-yl)ethanone

$^1$H NMR:

$^{13}$C NMR:
FTIR:

HRMS:

AW013 #60  RT: 1.43  AV: 1  NL: 8.63E7
F: FTMS + c ESI Full ms [75.00-1200.00]
Compound 1e: 1-(1-Benzylpiperidin-4-yl)-2-(pyridin-2-yl)ethanone

$^1$H NMR:

$^{13}$C NMR:
FTIR:

HRMS:

**AW007 #32**  RT: 0.75  AV: 1  SB: 13  0.46-0.58, 0.89-1.04  NL: 9.76E7
F: FTMS + c ESI Full ms [75.00-1200.00]
Compound 1f: 1-(4-Fluorophenyl)-2-(pyridin-2-yl)ethanone

$^1$H NMR:

$^{13}$C NMR:
Compound 1g: 1-(4-Chlorophenyl)-2-(pyridin-2-yl)ethanone

$^1$H NMR:

$^{13}$C NMR:
Compound 1h: 1-(4-Bromophenyl)-2-(pyridin-2-yl)ethanone

$^1$H NMR:

$^{13}$C NMR:
FTIR:

HRMS:

AW033 #79  RT: 1.97  AV: 1  NL: 6.63E7
F: FTMS + c ESI Full ms [75.00-1200.00]
Compound 1: 2-(Pyridin-2-yl)-1-(o-tolyl)ethanone

$^1$H NMR:

$^{13}$C NMR:
FTIR:

HRMS:

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Compound 1k: 1-(4-(n-Butyl)phenyl)-2-(pyridin-2-yl)ethanone

$^1$H NMR:

$^{13}$C NMR:
Compound 1: 1-(4-Methoxyphenyl)-2-(pyridin-2-yl)ethanone

$^1$H NMR:

$^{13}$C NMR:
FTIR:

HRMS:

AW004 #66  RT: 1.55  AV: 1  NL: 1.28E7
F: FTMS + c ESI Full ms [75.00-1200.00]
Compound 1m: 1-((tert-Butyldimethylsilyl)oxy)phenyl)-2-(pyridin-2-yl)ethanone

$^1$H NMR:

$^{13}$C NMR:
FTIR:

HRMS:

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Compound 1n: 1-(4-(Dibenzylamino)phenyl)-2-(pyridin-2-yl)ethanone

$^1$H NMR:

$^{13}$C NMR:
**FTIR:**

**HRMS:**

AW040 #56  RT: 1.42  AV: 1  NL: 1.83E8
F: FTMS + c ESI Full ms [75.00-1200.00]
Compound 10: 1-(Furan-2-yl)-2-(pyridin-2-yl)ethanone

$^1$H NMR:

$^{13}$C NMR:
Compound 3a: 2-Phenyl-4H-quinolizin-4-one

$^1$H NMR:

$^{13}$C NMR:
FTIR:

HRMS:

AW029 #69  RT: 1.73  AV: 1  NL: 9.01E7
F: FTMS + c ESI Full ms [75.00-1200.00]
Compound 3b: 2-Pentyl-4H-quinolizin-4-one

$^1$H NMR:

$^{13}$C NMR:
Compound 3c: 2-Isopropyl-4H-quinolinizin-4-one

$^1$H NMR:

$^{13}$C NMR:
Compound 3d: 2-Cyclobutyl-4H-quinolizin-4-one

$^1$H NMR:

$^{13}$C NMR:
FTIR:

HRMS:
Compound 3e: 2-(1-Benzylpiperidin-4-yl)-4H-quinolin-4-one

$^1$H NMR:

$^{13}$C NMR:
Compound 3f: 2-(4-Fluorophenyl)-4H-quinolin-4-one

$^1$H NMR:

$^{13}$C NMR:
Compound 3g: 2-(4-Chlorophenyl)-4H-quinolinizin-4-one

$^1$H NMR:

$^{13}$C NMR:
Compound 3h: 2-(4-Bromophenyl)-4H-quinolizin-4-one

$^1$H NMR:

$^{13}$C NMR:
**FTIR:**

**HRMS:**

**AW039 #78**  
RT: 1.97  AV: 1  NL: 3.89E7  
F: FTMS + c ESI Full ms [75.00-1200.00]
Compound 3: 2-(o-Tolyl)-4H-quinolin-4-one

$^1$H NMR:

$^{13}$C NMR:
FTIR:

HRMS:

**AW009 #75**  RT: 1.81  AV: 1  NL: 1.49E8
F: FTMS + c ESI Full ms [75.00-1200.00]

236.1068
237.1102
Compound 3k: 2-(4-((n-Butyl)phenyl)-4H-quinolin-4-one

$^1$H NMR:

$^{13}$C NMR:
Compound 3l: 2-(4-Methoxyphenyl)-4H-quinolizin-4-one

$^1$H NMR:

$^{13}$C NMR:
Compound 3m: 2-(4-((tert-Butyldimethylsilyl)oxy)phenyl)-4H-quinolin-4-one

$^1$H NMR:

$^{13}$C NMR:
FTIR:

HRMS:

No expected molecular ion in this sample
Compound 3n: 2-(4-(Dibenzylamino)phenyl)-4H-quinolizin-4-one

$^1$H NMR:

$^{13}$C NMR:
FTIR:

HRMS:

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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Compound 3o: 2-(Furan-2-yl)-4H-quinolizin-4-one

$^1$H NMR:

$^{13}$C NMR:
FTIR:

HRMS:

C:\Xcalibur\data\Dave\Allan Watson\AW023 26/03/2012 15:17:50 27-3 CWM

RT: 0.00 - 2.98
0.0 ... Abundance
212.0704
213.0738
234.0527 275.1020184.0759156.0809
204.1182
89.1714 124.0872

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Compound 5a: N-Methoxy-N-methylbenzamide

$^1$H NMR:

$^{13}$C NMR:
Compound 5b: N-Methoxy-N-methylhexanamide

$^1$H NMR:

$^{13}$C NMR:
Compound 5c: N-Methoxy-N-methylisobutyramide

$^1$H NMR:

$^{13}$C NMR:
FTIR:

HRMS:

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
This journal is © The Royal Society of Chemistry 2013
Compound 5d: N-Methoxy-N-methylcyclobutanecarboxamide

$^1$H NMR:

$^{13}$C NMR:
FTIR:

HRMS:

AW008 #65  RT: 1.56  AV: 1  NL: 3.72E7
F: FTMS + c ESI Full ms [75.00-1200.00]
Compound 5e: 1-Benzyl-N-methoxy-N-methylpiperidine-4-carboxamide

$^1$H NMR:

$^{13}$C NMR:
FTIR:

HRMS:

AW006 #34  RT: 0.80  AV: 1  NL: 3.78E8  
F: FTMS + c ESI Full ms [75.00-1200.00]
Compound 5f: 4-Fluoro-N-methoxy-N-methylbenzamide

$^1$H NMR:

$^{13}$C NMR:
FTIR:

HRMS:

**AW038 #61**  RT: 1.54  AV: 1  NL: 7.76E7
F: FTMS + c ESI Full ms [75.00-1200.00]

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Compound 5g: 4-Chloro-N-methoxy-N-methylbenzamide

$^1$H NMR:

$^{13}$C NMR:
Compound 5h: 4-Bromo-N-methoxy-N-methylbenzamide

$^1$H NMR:

$^{13}$C NMR:
FTIR:

HRMS:

AW030 #66  RT: 1.65  AV: 1  NL: 3.85E7
F: FTMS + c ESI Full ms [75.00-1200.00]
Compound 5i: N-Methoxy-N-methyl-4-nitrobenzamide

$^1$H NMR:

$^{13}$C NMR:
Compound 5j: N-Methoxy-N,2-dimethylbenzamide

$^1$H NMR:

$^{13}$C NMR:
Compound 5k: 4-(n-Butyl)-N-methoxy-N-methylbenzamide

$^1$H NMR:

$^{13}$C NMR:
Compound 5: 4-Methoxy-N-methoxy-N-methylbenzamide

$^1$H NMR:

$^{13}$C NMR:
FTIR:

[Graph of FTIR spectrum]

HRMS:

[Graph of HRMS spectrum]
Compound 5m: 4-((tert-Butyldimethylsilyl)oxy)-N-methoxy-N-methylbenzamide

$^1$H NMR:

$^{13}$C NMR:
FTIR:

HRMS:

AW018 #103  RT: 2.50  AV: 1  NL: 1.23E8
F: FTMS + c ESI Full ms [75.00-1200.00]
Compound 5n: 4-(Dibenzylamino)-N-methoxy-N-methylbenzamide

$^1$H NMR:

$^{13}$C NMR:
FTIR:

HRMS:

AW036 #78  RT: 1.98  AV: 1  NL: 1.38E8
F: FTMS + c ESI Full ms [75.00-1200.00]
Compound 5o: N-Methoxy-N-methylfuran-2-carboxamide

$^1$H NMR:

$^{13}$C NMR:
FTIR:

HRMS:

Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry
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3.6 Crystal Structure Data for 3a

3.6.1 General

Crystal data for 3a (C\(_{15}\)H\(_{11}\)NO, MW = 221.25) were measured at 123 K using an Oxford Diffraction Xcalibur E instrument and MoK\(_\alpha\) radiation, \(\lambda = 0.71073\) Å. Found monoclinic, space group P2\(_1\)/c, a = 9.0140(5), b = 10.7721(5), c = 11.3234(7) Å, \(\beta = 101.481(6)^\circ\), \(V = 1077.50(10)\) Å\(^3\), \(Z = 4\), \(\mu = 0.086\) mm\(^{-1}\), \(2\theta_{\text{max}} = 54^\circ\), 4957 reflections, 2347 unique, \(R_{\text{int}} = 0.0276\); final refinement to convergence on \(F^2\) gave \(R = 0.0475\) (\(F\), 1703 obs. data only) and \(R_w = 0.1061\) (\(F^2\), all data), GOF = 1.041. CCDC reference number 915082.

3.6.2 CIF Check

cHECK CIF/PLATON report (basic structural check)

No syntax errors found. CIF dictionary

Please wait while processing .... Interpreting this report

Datablock: watson1

---

Bond precision: C-C = 0.0021 Å Wavelength=0.71073 Å

Cell: a=9.0140(5) b=10.7721(5) c=11.3234(7)

alpha=90 beta=101.481(6) gamma=90

Temperature:123 K

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<th>Reported</th>
</tr>
</thead>
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<tr>
<td>Space group</td>
<td>P 21/c</td>
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<tr>
<td>Hall group</td>
<td>-P 2ybc</td>
</tr>
<tr>
<td>Moiety formula</td>
<td>C15 H11 N O</td>
</tr>
<tr>
<td>Sum formula</td>
<td>C15 H11 N O</td>
</tr>
<tr>
<td>Mr</td>
<td>221.25</td>
</tr>
<tr>
<td>Dx, g cm(^{-3})</td>
<td>1.364</td>
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<tr>
<td>Z</td>
<td>4</td>
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<tr>
<td>Mu (mm(^{-1}))</td>
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<td>F000</td>
<td>464.0</td>
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<tr>
<td>F000'</td>
<td>464.19</td>
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<td>h,k,lmax</td>
<td>11,13,14</td>
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<td>Tmin'</td>
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Correction method= MULTI-SCAN

Data completeness= 0.999 Theta(max)= 27.000

R(reflections)= 0.0475( 1703) wR2(reflections)= 0.1061( 2347)

S = 1.041 Npar= 154

The following ALERTS were generated. Each ALERT has the format
Click on the hyperlinks for more details of the test.

---

**Alert level G**

PLAT005_ALERT_5_G No _iucr_refine_instructions_details in the CIF

- 0 **ALERT level A** = Most likely a serious problem - resolve or explain
- 0 **ALERT level B** = A potentially serious problem, consider carefully
- 0 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
- 1 **ALERT level G** = General information/check it is not something unexpected

- 0 **ALERT type 1** CIF construction/syntax error, inconsistent or missing data
- 0 **ALERT type 2** Indicator that the structure model may be wrong or deficient
- 0 **ALERT type 3** Indicator that the structure quality may be low
- 0 **ALERT type 4** Improvement, methodology, query or suggestion
- 1 **ALERT type 5** Informative message, check

It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

**Publication of your CIF in IUCr journals**

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (Acta Crystallographica, Journal of Applied Crystallography, Journal of Synchrotron Radiation); however, if you intend to submit to Acta Crystallographica Section C or E, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

**Publication of your CIF in other journals**

Please refer to the Notes for Authors of the relevant journal for any special instructions relating to CIF submission.

### 3.6.3 Crystal Data and Structure Refinement for 3a.

Table 1. Crystal Data and Structure Refinement for 3a.

<table>
<thead>
<tr>
<th>Identification code</th>
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<td>Empirical formula</td>
<td>C15 H11 N O</td>
</tr>
<tr>
<td>Formula weight</td>
<td>221.25</td>
</tr>
<tr>
<td>Temperature</td>
<td>123(2) K</td>
</tr>
<tr>
<td>Wavelength</td>
<td>0.71073 Å</td>
</tr>
</tbody>
</table>
Crystal system: Monoclinic
Space group: P2₁/c
Unit cell dimensions:
  a = 9.0140(5) Å  \( \beta = 90^\circ \)
  b = 10.7721(5) Å  \( \beta = 101.481(6)^\circ \)
  c = 11.3234(7) Å  \( \beta = 90^\circ \)
Volume: 1077.50(10) Å³
Z: 4
Density (calculated): 1.364 Mg/m³
Absorption coefficient: 0.086 mm⁻¹
F(000): 464
Crystal size: 0.30 x 0.20 x 0.20 mm³
Theta range for data collection: 2.98 to 27.00°
Index ranges:
  \(-11 \leq h \leq 11, -13 \leq k \leq 13, -14 \leq l \leq 14\)
Reflections collected: 4957
Independent reflections: 2347 [R(int) = 0.0276]
Completeness to theta = 27.00°: 99.9 %
Absorption correction: Semi-empirical from equivalents
Max. and min. transmission: 1.00000 and 0.95340
Refinement method: Full-matrix least-squares on F²
Data / restraints / parameters: 2347 / 0 / 154
Goodness-of-fit on F²: 1.041
Final R indices [I>2σ(I)]: R1 = 0.0475, wR2 = 0.0937
R indices (all data): R1 = 0.0769, wR2 = 0.1061
Largest diff. peak and hole: 0.235 and -0.234 e.Å⁻³

Table 2. Atomic coordinates (x10⁴) and equivalent isotropic displacement parameters (Å²x10³)
for 3a. U(eq) is defined as one third of the trace of the orthogonalized Uij tensor.

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<th>y</th>
<th>z</th>
<th>U(eq)</th>
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<td>2359(1)</td>
<td>416(1)</td>
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<td>N(1)</td>
<td>1440(2)</td>
<td>4324(1)</td>
<td>1213(1)</td>
<td>18(1)</td>
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<tr>
<td>C(1)</td>
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<td>3433(2)</td>
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Table 3. Bond lengths [Å] and angles [°] for 3a.

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<td>1.403(2)</td>
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C(2)-C(3)-C(10)  121.02(14)
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C(5)-C(4)-H(4)  119.4
C(3)-C(4)-H(4)  119.4
C(4)-C(5)-N(1)  119.25(14)
C(4)-C(5)-C(6)  123.43(15)
N(1)-C(5)-C(6)  117.32(14)
C(7)-C(6)-C(5)  121.73(16)
C(7)-C(6)-H(6)  119.1
C(5)-C(6)-H(6)  119.1
C(6)-C(7)-C(8)  119.36(16)
C(6)-C(7)-H(7)  120.3
C(8)-C(7)-H(7)  120.3
C(9)-C(8)-C(7)  119.56(16)
C(9)-C(8)-H(8)  120.2
C(7)-C(8)-H(8)  120.2
C(8)-C(9)-N(1)  121.70(16)
C(8)-C(9)-H(9)  119.2
N(1)-C(9)-H(9)  119.2
C(15)-C(10)-C(11)  118.34(15)
C(15)-C(10)-C(3)  120.14(15)
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C(10)-C(11)-H(11)  119.7
C(11)-C(12)-C(13)  120.24(17)
C(11)-C(12)-H(12)  119.9
C(13)-C(12)-H(12)  119.9
C(14)-C(13)-C(12)  119.90(16)
C(14)-C(13)-H(13)  120.0
C(12)-C(13)-H(13)  120.0
C(13)-C(14)-C(15)  120.07(17)
C(13)-C(14)-H(14)  120.0
C(15)-C(14)-H(14)  120.0
C(14)-C(15)-C(10)  120.85(16)
C(14)-C(15)-H(15)  119.6
C(10)-C(15)-H(15)  119.6

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters (Å² x 10³) for watson1. The anisotropic displacement factor exponent takes the form: -2π²[ h²a²U₁₁ + ... + 2hk a* b* U₁² ]
Table 5. Hydrogen coordinates (x10^4) and isotropic displacement parameters (Å^2x10^3) for 3a.

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Table 6. Torsion angles [°] for 3a.

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C(9)-N(1)-C(1)-O(1) 2.9(2)
C(5)-N(1)-C(1)-O(1) 179.90(15)
C(9)-N(1)-C(1)-C(2)  -175.44(14)
C(5)-N(1)-C(1)-C(2)  1.5(2)
O(1)-C(1)-C(2)-C(3)  -178.43(17)
N(1)-C(1)-C(2)-C(3)  -0.2(2)
C(1)-C(2)-C(3)-C(4)  -1.6(2)
C(1)-C(2)-C(3)-C(10)  176.44(15)
C(2)-C(3)-C(4)-C(5)  2.3(2)
C(10)-C(3)-C(4)-C(5)  -175.78(15)
C(5)-N(1)-C(1)-C(2)  -0.9(2)
C(9)-N(1)-C(1)-C(2)  -2.9(2)
C(1)-N(1)-C(5)-C(6)  -179.82(14)
O(1)-C(1)-C(2)-C(3)  178.43(17)
C(1)-N(1)-C(5)-C(6)  175.97(15)
C(9)-N(1)-C(5)-C(6)  30.6(2)
C(1)-N(1)-C(5)-C(6)  31.8(2)
C(2)-C(3)-C(10)-C(15)  -147.47(16)
C(3)-C(4)-C(10)-C(15)  30.6(2)
C(2)-C(3)-C(10)-C(11)  31.8(2)
C(4)-C(3)-C(10)-C(11)  -150.15(15)
C(15)-C(10)-C(11)-C(12)  0.3(2)
C(3)-C(10)-C(11)-C(12)  -179.02(15)
C(10)-C(11)-C(12)-C(13)  0.9(3)
C(11)-C(12)-C(13)-C(14)  -0.7(3)
C(12)-C(13)-C(14)-C(15)  0.1(2)
C(13)-C(14)-C(15)-C(10)  179.40(15)

Symmetry transformations used to generate equivalent atoms:

4. References
2. The β-ketopyridines prepared exist as a mixture of enol and keto tautomers in various ratios. In addition, some of the enol tautomers exhibit cis-trans isomerism. As such, the $^1$H and $^{13}$C NMR spectra for these compounds are complicated and we have elected to provide the spectra, which can be found in the appendix.