Oxidative coupling of 1-(2-methyl-4-phenylquinolin-3-yl)ethanone with ethanol and unexpected deacetyltative synthesis of 3-hydroxy quinoline

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General Experimental Details:

$^1$H NMR and $^{13}$C NMR spectra were recorded on Bruker Avance-400 using CDCl$_3$ and DMSO-d$_6$ as solvent and tetramethylsilane as internal reference. Splitting patterns are described as singlet (s), doublet (d), triplet (t), multiplet (m) and broad (br). IR spectra were recorded on a FT-IR spectrophotometer and melting points were determined on melting point apparatus and are uncorrected. Electrospray ionization mass spectrometry (ESI-MS) was obtained on Thermo LCQ Advantage Max Spectrometer and HRMS was recorded on Agilent 6520 Q-TOF. Reactions were monitored on silica gel TLC plates (coated with TLC grade silica gel, obtained from Merck). All glass apparatus were oven dried prior to use. Column chromatography was performed over silica gel (230-400 Mesh) by using Smart flash EPCLC AI-700X YAMAZEN with minimal amount of solvent. All chemicals and reagents were obtained from Aldrich (USA) and Alfa Aesar (England) and were used without further purification.

General procedure for the synthesis of product 2:

Take solution of 1-(2-methyl-4-phenylquinolin-3-yl)ethanone 1 (1.0 mmol), N-bromosuccinimide (1.5 mmol), tert-butyl hydroperoxide (5 mmol) and potassium carbonate (3.0 mmol) in ethanol (20 mL) was stirred at room temperature for 24 hours. Next, the ethanol was removed under reduced pressure and the crude of reaction was diluted with water (80 mL). The aqueous layer was extracted with ethylacetate (3×20 mL) three times. The organic layer was further dried over sodium sulphate (anhydrous) and removed under reduced pressure to give crude product, which further purified by column chromatography over silica gel afforded the desired product 2a-2r.

General procedure for the synthesis of product 5:

Take solution of 1-(2-methyl-4-phenylquinolin-3-yl)ethanone 1 (1.0 mmol), N-bromosuccinimide (1.5 mmol) and potassium carbonate (3.0 mmol) in methanol (20 mL) was stirred at room temperature for 6 hours. Next, the methanol was removed under reduced pressure and the crude of reaction was diluted with water (80 mL). The aqueous layer was extracted with ethylacetate (3×20 mL) three times. The organic layer was further dried over sodium sulphate (anhydrous) and removed under reduced pressure to give crude product, which further purified by column chromatography over silica gel afforded the desired product 5a-5h.
Compound Characterization Data:

(2-methyl-4-phenylquinolin-3-yl)(3-methyloxiran-2-yl)methanone (2a):

![2a](image)

Yield 78% as white solid: mp 100-102°C; FT-IR (KBr, cm⁻¹) 3019, 1635, 1215, 1069; ¹HNMR (400 MHz, CDCl₃) δ 1.06 (d, J = 5.12 Hz, 3H), 2.70-2.77 (m, 4H), 3.07 (d, J = 1.80 Hz, 1H), 7.32-7.34 (m, 1H), 7.47-7.52 (m, 2H), 7.53-7.59 (m, 3H), 7.65-7.68 (dd, J = 8.4, 0.8 Hz, 1H), 7.75-7.79 (m, 1H), 8.12 (d, J = 8.4 Hz, 1H); ¹³C NMR (100 MHz, DMSO-d₆) δ 17.57 (CH₃), 23.98 (CH₃), 56.89 (CH), 60.21 (CH), 124.81 (C), 126.19 (CH), 127.49 (CH), 128.99 (CH), 129.12 (CH), 129.17 (CH), 129.57 (CH), 130.46 (CH), 130.69 (CH), 131.15 (CH), 131.72 (C), 134.74 (C), 145.69 (C), 147.70 (C), 154.16 (C) 203.26 (C); ESI-MS (m/z) 304 (M+H)⁺; HRMS (ESI) calculated for C₂₀H₁₈NO₂ (M + H)⁺ 304.1338, found 304.1330.

(7-chloro-2-methyl-4-phenylquinolin-3-yl)(3-methyloxiran-2-yl)methanone (2b):

![2b](image)

Yield 63% as white solid: mp 114-116°C; FT-IR (KBr, cm⁻¹) 3019, 1644, 1215, 1069; ¹H NMR (400 MHz, DMSO-d₆) δ 0.97 (d, J = 5.04 Hz, 3H), 2.60-2.65 (m, 4H), 3.41 (d, J = 1.80 Hz, 1H), 7.29-7.32 (m, 1H), 7.42-7.44 (m, 1H), 7.51-7.62 (m, 5H), 8.10 (d, J = 2.04 Hz, 1H); ¹³C NMR (100 MHz, DMSO-d₆) δ 17.55 (CH₃), 24.02 (CH₃), 56.94 (CH), 60.15 (CH), 123.59 (C), 127.72 (CH), 128.09 (CH), 128.31 (CH), 129.09 (CH), 129.26 (CH), 129.78 (CH), 130.42 (CH), 130.64 (CH), 131.97 (C), 134.24 (C), 135.86 (C), 145.85 (C), 148.08 (C), 155.87 (C) 202.96 (C); ESI-MS (m/z) 338 (M+H)⁺; HRMS (ESI) calculated for C₂₀H₁₇ClNO₂ (M + H)⁺ 338.0948, found 338.0933.
(6-chloro-2-methyl-4-phenylquinolin-3-yl)(3-methyloxiran-2-yl)methanone (2c):

Yield 72% as white solid: mp 142-143°C; FT-IR (KBr, cm\(^{-1}\)) 3019, 1636, 1215, 1068; \(^1\)H NMR (400 MHz, DMSO-\(d_6\)) \(\delta\) 0.99 (d, \(J = 5.04\) Hz, 3H), 2.60 (s, 3H), 2.63-2.66 (m, 1H), 3.43 (d, \(J = 1.84\) Hz, 1H), 7.31-7.33 (m, 1H), 7.42-7.46 (m, 2H), 7.56-7.64 (m, 3H), 7.84-7.87 (dd, \(J = 8.96, 2.4\) Hz, 1H), 8.08 (d, \(J = 8.96\) Hz, 1H); \(^1\)C NMR (100 MHz, DMSO-\(d_6\)) \(\delta\) 17.57 (CH\(_3\)), 23.97 (CH\(_3\)), 56.97 (CH), 60.12 (CH), 124.72 (CH), 125.79 (C), 129.20 (CH), 129.36 (CH), 129.89 (CH), 130.42 (CH), 130.62 (CH), 131.41 (CH), 131.66 (CH), 131.97 (C), 132.49 (C), 134.01 (C), 144.98 (C), 146.14 (C), 154.98 (C) 203.01 (C); ESI-MS (m/z) 338 (M+H); HRMS (ESI) calculated for C\(_{20}\)H\(_{17}\)ClNO\(_2\) (M + H)\(^+\) 338.0948, found 338.0947.

(5-fluoro-2-methyl-4-phenylquinolin-3-yl)(3-methyloxiran-2-yl)methanone (2d):

Yield 70% as white solid: mp 75-78°C; FT-IR (KBr, cm\(^{-1}\)) 3019, 1637, 1215, 1069; \(^1\)H NMR (400 MHz, DMSO-\(d_6\)) \(\delta\) 1.02 (d, \(J = 5.04\) Hz, 3H), 2.54-2.56 (m, 1H), 2.58 (s, 3H), 3.44 (d, \(J = 1.64\) Hz, 1H), 7.28-7.34 (m, 2H), 7.40 (brd, 1H), 7.43-7.49 (m, 3H), 7.79-7.84 (m, 1H), 7.92 (d, \(J = 8.36\) Hz, 1H); \(^1\)C NMR (100 MHz, DMSO-\(d_6\)) \(\delta\) 17.57 (CH\(_3\)), 23.97 (CH\(_3\)), 56.91 (CH), 60.07 (CH), 112.85 (d, \(J = 21.32\) Hz, CH), 115.30 (C), 125.67 (CH), 128.19 (2×CH), 128.98 (CH), 129.43 (CH), 129.68 (CH), 131.32 (d, \(J = 9.62\) Hz, CH), 133.79 (C), 136.85 (C), 142.70 (C), 146.97 (C), 154.85 (C), 159.76 (d, \(J = 256.54\) Hz, C-F), 203.07 (C); ESI-MS (m/z) 322 (M+H); HRMS (ESI) calculated for C\(_{20}\)H\(_{17}\)FNO\(_2\) (M+H)\(^+\) 322.1243, found 322.1239.
(6-chloro-4-(2-chlorophenyl)-2-methylquinolin-3-yl)(3-methyloxiran-2-yl)methanone (2e):

Yield 66% as white solid: mp 162-164°C; FT-IR (KBr, cm⁻¹) 3019, 1637, 1215, 1071; 
¹HNMR (400 MHz, DMSO-d₆) δ 1.12 (d, J = 5.04 Hz, 3H), 2.66 (s, 3H), 2.73-2.77 (m, 1H), 3.62 (d, J = 1.72 Hz, 1H ), 7.16 (d, J = 2.28 Hz, 1H ), 7.49-7.52 (dd, J = 7.52, 1.80 Hz, 1H), 7.57-7.66 (m, 2H), 7.71-7.73 (dd, J = 7.8, 1.08 Hz, 1H), 7.87-7.89 (dd, J = 8.96, 2.32 Hz, 1H), 8.12 (d, J = 8.96 Hz, 1H ); ¹³C NMR (100 MHz, DMSO-d₆) δ 17.59 (CH₃), 24.06 (CH₃), 57.21 (CH), 59.80 (CH), 124.30 (CH), 125.46 (C), 128.05 (CH), 130.35 (CH), 131.54 (CH), 131.95 (CH), 132.06 (CH), 132.33 (C), 132.54 (CH), 132.68 (C), 132.83 (C), 133.21 (C), 142.10 (C), 145.26 (C), 155.25 (C) 201.76 (C); ESI-MS (m/z) 372 (M+H)⁺; HRMS (ESI) calculated for C₂₀H₁₆Cl₂NO₂ (M+H)⁺ 372.0558, found 372.0551.

(8-bromo-6-chloro-2-methyl-4-phenylquinolin-3-yl)(3-methyloxiran-2-yl)methanone (2f):

Yield 64% as light yellow solid: mp 142-144°C; FT-IR (KBr, cm⁻¹) 3019, 1637, 1215, 1069; 
¹HNMR (400 MHz, DMSO-d₆) δ 1.00 (d, J = 5.04 Hz, 3H), 2.63-2.65 (m, 4H), 3.44 (d, J = 1.8 Hz, 1H), 7.31-7.33 (m, 1H), 7.41-7.45 (m, 2H), 7.56-7.64 (m, 3H), 8.33 (d, J = 2.24 Hz, 1H); ¹³C NMR (100 MHz, DMSO-d₆) δ 17.55 (CH₃), 24.25 (CH₃), 56.99 (CH), 60.07 (CH), 124.91 (CH), 125.91 (C), 126.77 (C), 129.20 (CH), 129.35 (CH), 130.04 (CH), 130.43 (CH), 130.67 (CH), 131.68 (C), 133.26 (C), 133.69 (C), 134.32 (CH), 143.08 (C), 145.74 (C), 156.00 (C) 202.66 (C); ESI-MS (m/z) 416 (M+H)⁺; HRMS (ESI) calculated for C₂₀H₁₆BrClNO₂ (M+H)⁺ 416.0053, found 416.0057.
(6,8-dibromo-2-methyl-4-phenylquinolin-3-yl)(3-methylxiran-2-yl)methanone (2g):

Yield 69% as light yellow solid: mp 141-143°C; FT-IR (KBr, cm\(^{-1}\)) 3019, 1637, 1210, 1069; \(^1\)H NMR (400 MHz, DMSO-\(d_6\)) \(\delta\) 1.01 (d, \(J = 4.88\) Hz, 3H), 2.66 (m, 4H), 3.46 (s, 1H), 7.34 (d, \(J = 5.44\) Hz, 1H), 7.46 (d, \(J = 6.68\) Hz, 1H), 7.58-7.62 (m, 4H), 8.42 (d, \(J = 1.64\) Hz, 1H); \(^13\)C NMR (100 MHz, DMSO-\(d_6\)) \(\delta\) 17.52 (CH\(_3\)), 24.26 (CH\(_3\)), 57.04 (CH), 60.06 (CH), 119.93 (C), 125.92 (C), 127.32 (C), 128.14 (CH), 129.20 (CH), 129.35 (CH), 130.06 (CH), 130.40 (CH), 130.64 (CH), 133.23 (C), 133.65 (C), 136.62 (CH), 143.24 (C), 145.61 (C), 156.08 (C) 202.66 (C); ESI-MS (m/z) 462 (M+H\(^+\)); HRMS (ESI) calculated for C\(_{20}\)H\(_{16}\)Br\(_2\)NO\(_2\) (M+H\(^+\)) 461.9527, found 461.9526.

(8-bromo-6-chloro-4-(2-chlorophenyl)-2-methylquinolin-3-yl)(3-methylxiran-2-yl)methanone (2h):

Yield 65% as light yellow solid: mp 138-140°C; FT-IR (KBr, cm\(^{-1}\)) 3019, 1635, 1215, 1068; \(^1\)H NMR (400 MHz, DMSO-\(d_6\)) \(\delta\) 1.12 (d, \(J = 5.04\) Hz, 3H), 2.69 (m, 3H), 2.73-2.77 (m, 1H), 3.63 (d, \(J = 1.6\) Hz, 1H), 7.17 (d, \(J = 2.16\) Hz, 1H), 7.49-7.51 (dd, \(J = 7.48\), 1.64 Hz, 1H), 7.57-7.66 (m, 2H), 7.70-7.72 (dd, \(J = 7.84\), 0.92 Hz, 1H), 8.35 (d, \(J = 2.16\) Hz, 1H); \(^13\)C NMR (100 MHz, DMSO-\(d_6\)) \(\delta\) 17.57 (CH\(_3\)), 24.35 (CH\(_3\)), 57.25 (CH), 59.75 (CH), 124.42 (CH), 126.05 (C), 126.39 (C), 128.07 (CH), 130.37 (CH), 132.16 (C), 132.23 (CH), 132.34 (C), 132.55 (CH), 132.80 (C), 134.03 (C), 134.66 (CH), 142.83 (C), 143.01 (C), 156.32 (C) 201.45 (C); ESI-MS (m/z) 450 (M+H\(^+\)); HRMS (ESI) calculated for C\(_{20}\)H\(_{15}\)BrCl\(_2\)NO\(_2\) (M+H\(^+\)) 449.9663, found 449.9650.
(4-(4-bromophenyl)-6-chloro-2-methylquinolin-3-yl)(3-methyloxiran-2-yl)methanone (2i): 

Yield 71% as light yellow solid: mp 140-142°C; FT-IR (KBr, cm⁻¹) 3019, 1700, 1215, 1070; 
¹H NMR (400 MHz, DMSO-d₆) δ 1.06 (d, J = 5.04 Hz, 3H), 2.61 (s, 3H), 2.75-2.79 (m, 1H), 
3.47 (d, J = 1.84 Hz, 1H), 7.28-7.31 (dd, J = 8.12, 2.16 Hz, 1H), 7.40-7.44 (m, 2H), 7.78-7.89 
(m, 3H), 8.09 (d, J = 8.92 Hz, 1H); ¹³C NMR (100 MHz, DMSO-d₆) δ 17.44 (CH₃), 23.96 
(CH₃), 56.98 (CH), 60.13 (CH), 123.60 (C), 124.60 (CH), 125.51 (C), 131.41 (CH), 131.80 
(CH), 132.20 (C), 132.26 (C & CH), 132.35 (CH), 132.49 (CH), 132.82 (CH), 132.21 (C), 
143.94 (C), 146.13 (C), 154.99 (C), 203.00 (C); ESI-MS (m/z) 416 (M+H)⁺; HRMS (ESI) 
calculated for C₂₀H₁₆BrClNO₂ (M+H)⁺ 416.0053, found 416.0055.

(4-(4-bromophenyl)-5-fluoro-2-methylquinolin-3-yl)(3-methyloxiran-2-yl)methanone (2j): 

Yield 63% as light yellow solid: mp 102-104°C; FT-IR (KBr, cm⁻¹) 3019, 1619, 1215, 1069; 
¹H NMR (400 MHz, DMSO-d₆) δ 1.09 (d, J = 5.04 Hz, 3H), 2.59 (s, 3H), 2.69-2.73 (m, 1H), 
3.48 (d, J = 1.76 Hz, 1H), 7.27-7.29 (dd, J = 8.04, 2.08 Hz, 1H), 7.32-7.38 (m, 2H), 7.66-7.72 
(m, 2H), 7.81-7.86 (m, 1H), 7.93 (d, J = 7.84 Hz, 1H); ¹³C NMR (100 MHz, DMSO-d₆) δ 17.47 (CH₃), 23.85 (CH₃), 57.02 (CH), 60.08 (CH), 112.96 (d, J = 21.27 Hz, CH), 115.11 (d, 
J = 8.96 Hz, C), 122.64 (CH), 125.76 (CH), 131.19 (2×CH), 131.37 (C), 131.49 (CH), 131.95 
(CH), 133.49 (C), 136.07 (C), 141.59 (C), 148.93 (CH), 154.94 (C), 159.65 (d, J = 255.95
Hz, C-F), 203.09 (C); ESI-MS (m/z) 400 (M+H)+; HRMS (ESI) calculated for C_{20}H_{16}BrFNO_{2} (M + H)+ 400.0348, found 400.0341.

(5-fluoro-4-(4-fluorophenyl)-2-methylquinolin-3-yl)(3-methyloxiran-2-yl)methanone (2k):

Yield 67% as white solid: mp 108-110°C; FT-IR (KBr, cm^{-1}) 3019, 1637, 1216, 1069; ^{1}H NMR (400 MHz, CD_{3}OD) \delta 1.14 (d, J = 5.08 Hz, 3H), 2.67 (s, 3H), 2.72-2.74 (m, 1H), 3.35 (d, J = 1.6 Hz, 1H), 7.21-7.29 (m, 3H), 7.32-7.36 (m, 1H), 7.47-7.51 (m, 1H), 7.79-7.84 (m, 1H), 7.94 (d, J = 8.48 Hz, 1H); ^{13}C NMR (100 MHz, CD_{3}OD) \delta 16.11 (CH_{3}), 22.10 (CH_{3}), 56.61 (CH), 59.97 (CH), 112.23 (d, J = 21.85 Hz, CH ), 114.55 (d, J = 21.87 Hz, CH ), 114.69 (d, J = 21.95 Hz, CH), 115.38 (d, J = 9.3 Hz, C), 124.42 (d, J = 3.89 Hz, CH), 130.78 (d, J = 9.68 Hz, CH), 131.13-131.22 (m, CH), 131.57-131.66 (m, CH), 132.59 (C), 133.51 (C), 142.60 (C), 148.65 (C), 155.02 (C), 159.88 (d, J = 257.23 Hz, C-F), 164.28 (d, J = 245.89 Hz, C-F), 202.97 (C); ESI-MS (m/z) 334 (M+H)^{+}; HRMS (ESI) calculated for C_{20}H_{16}F_{2}NO_{2} (M + H)^{+} 340.1149, found 340.1140.

(2-methyl-6-nitro-4-phenylquinolin-3-yl)(3-methyloxiran-2-yl)methanone (2l):

Yield 52% as white solid: mp 153-155°C; FT-IR (KBr, cm^{-1}) 3019, 1626, 1216, 1069; ^{1}H NMR (400 MHz, DMSO-\textit{d}_{6}) \delta 1.00 (d, J = 5.04 Hz, 3H), 2.68-2.71 (m, 4H), 3.46 (d, J = 1.80 Hz, 1H ), 7.38-7.39 (m, 1H), 7.51-7.53 (m, 1H), 7.61-7.69 (m, 3H), 8.27 (d, J = 9.2 Hz, 1H), 8.36 (d, J = 2.44 Hz, 1H), 8.52-8.55 (dd, J = 9.16, 2.56 Hz, 1H); ^{13}C NMR (100 MHz, DMSO-\textit{d}_{6}) \delta 17.55 (CH_{3}), 23.39 (CH_{3}), 57.05 (CH), 60.12 (CH), 123.02 (CH), 124.17 (C), 124.47 (CH), 129.32 (CH), 129.47 (CH), 130.30 (CH), 130.57 (CH), 130.79 (CH), 131.17
(4-(4-fluorophenyl)-2-methylquinolin-3-yl)(3-methyloxiran-2-yl)methanone (2p):

Yield 64% as white solid; mp 118-120°C; FT-IR (KBr, cm$^{-1}$) 3019, 1619, 1215, 1076; $^1$H NMR (400 MHz, CD$_3$OD) $\delta$ 1.08 (d, $J = 5.08$ Hz, 3H), 2.66 (s, 3H), 2.73-2.77 (m, 1H), 3.25 (d, $J = 1.76$ Hz, 1H), 7.30-7.37 (m, 3H), 7.49-7.63 (m, 3H), 7.79-7.84 (m, 1H), 8.05 (d, $J = 8.44$ Hz, 1H); $^{13}$C NMR (100 MHz, CD$_3$OD) $\delta$ 16.08 (CH$_3$), 22.09 (CH$_3$), 56.55 (CH), 60.09 (CH), 115.49 (d, $J = 21.89$ Hz, CH), 115.53 (d, $J = 21.86$ Hz, CH), 124.95 (C), 125.68 (CH), 126.97 (CH), 127.71 (CH), 130.59 (d, $J = 3.51$ Hz, C), 130.81 (CH), 131.49 (C), 132.29 (d, $J = 8.45$ Hz, CH), 132.65 (d, $J = 8.26$ Hz, CH), 145.69 (C), 147.29 (C), 154.36 (C), 164.56 (d, $J = 246.78$ Hz, C-F), 203.11 (C); ESI-MS (m/z) 322 (M+H)$^+$; HRMS (ESI) calculated for C$_{20}$H$_{17}$FNO$_2$ (M+H)$^+$ 322.1243, found 322.1243.

(4-(4-bromophenyl)-2-methylquinolin-3-yl)(3-methyloxiran-2-yl)methanone (2q):

Yield 71% as yellow solid; mp 124-126°C; FT-IR (KBr, cm$^{-1}$) 3019, 1637, 1215, 1069; $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$ 1.05 (d, $J = 5.08$ Hz, 3H), 2.52 (s, 3H), 2.74-2.78 (m, 1H), 3.46 (d, $J = 1.84$ Hz, 1H), 7.27-7.29 (dd, $J = 8.08$, 2.16 Hz, 1H), 7.39-7.42 (dd, $J = 8.2$, 2.2 Hz, 1H), 7.50-7.53 (dd, $J = 8.36$, 0.88 Hz, 1H), 7.57-7.61 (m, 1H), 7.77-7.87 (m, 3H), 8.06 (d, $J = 7.96$ Hz, 1H); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$ 17.45 (CH$_3$), 23.98 (CH$_3$), 56.93 (CH), 60.20 (CH), 123.27 (C), 124.55 (C), 126.08 (CH), 127.68 (CH), 129.15 (CH), 131.31 (CH), 131.46 (C), 132.00 (CH), 132.17 (CH), 132.53 (CH), 132.89 (CH), 133.94 (C), 144.65
(C), 147.67 (C), 154.21 (C), 203.27 (C); ESI-MS (m/z) 382 (M+H); HRMS (ESI) calculated for C_{20}H_{17}BrNO_{2} (M + H)^{+} 382.0443, found 382.0442.

(4-(4-chlorophenyl)-2-methylquinolin-3-yl)(3-methyloxiran-2-yl)methanone (2r):

![Structure 2r](image)

Yield 72\% as white solid: mp 105-107°C; FT-IR (KBr, cm\(^{-1}\)) 3019, 1643, 1215, 1069; \(^{1}\)H NMR (400 MHz, DMSO-\(d_6\)) \(\delta\) 1.04 (d, \(J = 5.04\) Hz, 3H), 2.61 (s, 3H), 2.74-2.76 (m, 1H), 3.45 (d, \(J = 1.84\) Hz, 1H), 7.33-7.36 (dd, \(J = 8.12, 2.12\) Hz, 1H), 7.45-7.51 (m, 2H), 7.56-7.69 (m, 3H), 7.82-7.86 (m, 1H), 7.93 (dd, \(J = 8.84, 2.2\) Hz, 1H), 7.95-7.97 (m, 1H), 8.03 (d, \(J = 8.32\) Hz, 1H); \(^{13}\)C NMR (100 MHz, DMSO-\(d_6\)) \(\delta\) 17.44 (CH\(_{3}\)), 23.96 (CH\(_{3}\)), 56.88 (CH), 60.20 (CH), 124.63 (C), 126.06 (CH), 127.66 (CH), 129.08 (CH), 129.14 (CH), 129.23 (CH), 131.29 (CH), 131.54 (C), 132.28 (CH), 132.64 (CH), 133.56 (C), 134.55 (C), 144.61 (C), 147.67 (C), 154.19 (C), 203.27 (C); ESI-MS (m/z) 338 (M+H); HRMS (ESI) calculated for C_{20}H_{17}ClNO_{2} (M + H)^{+} 338.0948, found 338.0944.

(4-(4-methoxyphenyl)-2-methylquinolin-3-yl)(3-methyloxiran-2-yl)methanone (2s):

![Structure 2s](image)

Yield 67\% as white solid: mp 112-114°C; FT-IR (KBr, cm\(^{-1}\)) 3019, 1637, 1215, 1069; \(^{1}\)H NMR (400 MHz, DMSO-\(d_6\)) \(\delta\) 1.04 (d, \(J = 5.04\) Hz, 3H), 2.59 (s, 3H), 3.36 (d, \(J = 1.84\) Hz, 1H), 3.85 (s, 3H), 7.10-7.17 (m, 2H), 7.22-7.25 (dd, \(J = 8.24, 2.0\) Hz, 1H), 7.35-7.37 (dd, \(J = 8.44, 2.2\) Hz, 1H), 7.54-7.61 (m, 2H), 7.79-7.84 (m, 1H), 8.03 (d, \(J = 8.32\) Hz, 1H); \(^{13}\)C NMR (100 MHz, DMSO-\(d_6\)) \(\delta\) 17.58 (CH\(_{3}\)), 23.95 (CH\(_{3}\)), 55.79 (CH\(_{3}\)), 56.75 (CH), 60.23 (CH), 114.58 (CH), 114.63 (CH), 125.13 (C), 126.23 (CH), 123.63 (C), 127.37 (CH), 129.10 (CH), 131.08 (CH), 131.72 (C), 131.98 (CH), 132.16 (CH), 145.70 (C), 147.75 (C), 154.21 (C), 160.27 (C), 203.42 (C); ESI-MS (m/z) 334 (M+H); HRMS (ESI) calculated for C_{21}H_{20}NO_{3} (M + H)^{+} 334.1443, found 334.1430.
(2,4-dimethylquinolin-3-yl)(3-methyloxiran-2-yl)methanone (2t):

Yield 62% as white solid: mp 120-122°C; FT-IR (KBr, cm⁻¹) 3021, 1636, 1216, 1069; ¹H NMR (300 MHz, CDCl₃) δ 1.43 (d, J = 5.13 Hz, 3H), 2.60 (s, 3H), 2.64 (s, 3H ), 3.04-3.09 (m, 1H), 3.56 (d, J = 1.83 Hz, 1H ), 7.53-7.59 (m, 1H), 7.70-7.76 (m, 1H), 7.97-8.04 (m, 2H); ¹³C NMR (75 MHz, CDCl₃) δ 15.79 (CH₃), 17.42 (CH₃), 23.73 (CH₃), 55.67 (CH), 61.45 (CH), 123.73 (CH), 125.71 (C), 126.47 (CH), 129.33 (CH), 130.27 (CH), 130.55 (C), 141.66 (C), 147.31 (C), 153.89 (C), 205.13 (C); ESI-MS (m/z) 242 (M+H)⁺ for C₁₅H₁₆NO₃.

(2-methyl-4-phenylquinolin-3-yl)(oxiran-2-yl)methanone (2u):

Yield 20% as white solid: mp 133-135°C; FT-IR (KBr, cm⁻¹) 3019, 1651, 1215, 1069; ¹H NMR (400 MHz, CDCl₃) δ 2.45-2.47 (dd, J = 6.44, 2.36 Hz, 1H), 2.59-2.62 (q, J = 4.48 Hz, 1H), 2.72 (s, 3H), 3.26-3.27 (q, J = 2.36 Hz, 1H), 7.31-7.33 (m, 1H), 7.45-7.56 (m, 5H), 7.65-7.67 (m, 1H), 7.74-7.78 (m, 1H), 8.10 (d, J = 8.4 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 23.95 (CH₃), 47.54 (CH₂), 53.55 (CH), 124.80 (C), 126.20 (CH), 126.75 (CH), 128.52 (CH), 128.70 (CH), 129.01 (CH), 129.22 (CH), 130.35 (CH), 130.70 (C&CH), 130.76 (CH), 134.84 (C), 146.35 (C), 148.04 (C), 154.40 (C), 203.13 (C); ESI-MS (m/z) 290 (M+H)⁺; HRMS (ESI) calculated for C₁₉H₁₆NO₂ (M + H)⁺ 290.1181, found 290.1179.

2-bromo-1-(2-methyl-4-phenylquinolin-3-yl)ethanone (3):
Yield 90% as white solid: mp 175-177°C; FT-IR (KBr, cm⁻¹) 3021, 1621, 1215, 1069; ¹H NMR (400 MHz, CDCl₃) δ 2.75 (s, 3H), 3.51 (s, 2H), 7.37-7.39 (m, 2H), 7.45-7.49 (m, 1H), 7.53-7.57 (m, 3H), 7.64-7.66 (dd, J = 8.4, 0.8 Hz, 1H), 7.73-7.78 (m, 1H), 8.10 (d, J = 8.44 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 24.01 (CH₃), 35.21 (CH₂), 124.61 (C), 126.25 (CH), 126.79 (CH), 129.05 (CH), 129.15 (2×CH), 129.47 (CH), 129.92 (2×CH), 130.70 (CH), 131.36 (C), 134.96 (C), 144.77 (C), 147.96 (C), 154.93 (C), 198.35 (C); ESI-MS (m/z) 340 (M+H)+ for C₁₈H₁₅BrNO.

2-methyl-4-phenylquinolin-3-yl acetate (4):

![Structure of 4](image)

Yield 50% as light yellow solid: mp 97-99°C; FT-IR (KBr, cm⁻¹) 3019, 1626, 1216, 1069; ¹H NMR (400 MHz, CDCl₃) δ 2.78 (s, 3H), 3.58 (s, 2H), 7.34-7.37 (m, 2H), 7.41-7.51 (m, 4H), 7.57-7.59 (dd, J = 8.4, 0.84 Hz, 1H), 7.69-7.74 (m, 1H), 8.08 (d, J = 8.4 Hz, 1H); ¹³C NMR (100 MHz, CDCl₃) δ 23.80 (CH₃), 52.15 (CH₂), 125.09 (C), 126.46 (CH), 126.54 (CH), 127.28 (CH), 128.28 (2×CH), 128.50 (CH), 128.88 (CH), 129.25 (2×CH), 130.32 (CH), 135.69 (C), 146.41 (C), 147.78 (C), 154.56 (C), 169.01 (C); ESI-MS (m/z) 278 (M+H)+ for C₁₈H₁₆NO₂.

2-methyl-4-phenylquinolin-3-ol (5a):

![Structure of 5a](image)

Yield 62% as light yellow solid: mp 239-241°C; FT-IR (KBr, cm⁻¹) 3400, 3019, 1638, 1215, 1068; ¹H NMR (500 MHz, DMSO-d₆) δ 2.64 (s, 3H), 7.24-7.26 (dd, J = 8.35, 0.8 Hz, 1H ), 7.33-7.37 (m, 3H), 7.47-7.51 (m, 2H), 7.54-7.57 (m, 2H), 7.87-7.89 (m, 3H), 8.90 (s, 1H);
$^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$ 21.72 (CH$_3$), 124.63 (CH), 126.24 (CH), 126.30 (CH), 128.01 (C), 128.35 (CH), 128.70 (CH), 128.99 (2×CH), 129.23 (C), 130.84 (2×CH), 134.29 (C), 142.58 (C), 145.92 (C), 153.02 (C); ESI-MS (m/z) 236 (M+H)$^+$; HRMS (ESI) calculated for C$_{16}$H$_{14}$NO (M + H)$^+$: 236.1075, found 236.1075.

6-chloro-2-methyl-4-phenylquinolin-3-ol (5b):

![Structure of 6-chloro-2-methyl-4-phenylquinolin-3-ol (5b)](image)

Yield 61% as light brown solid: mp 235-237°C; FT-IR (KBr, cm$^{-1}$) 3399, 3019, 1603, 1216, 1071; $^1$H NMR (500 MHz, DMSO-$d_6$) $\delta$ 2.63 (s, 3H), 7.15 (d, $J = 2.2$ Hz, 1H ), 7.35-7.37 (m, 2H), 7.48-7.53 (m, 2H), 7.56-7.59 (m, 2H), 7.90 (d, $J = 8.9$ Hz, 1H ), 9.19 (s, 1H); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$ 21.73 (CH$_3$), 123.09 (CH), 126.66 (CH), 128.24 (C), 128.70 (CH), 129.11 (C), 129.22 (2×CH), 130.78 (2×CH), 130.83 (C), 130.92 (CH), 133.59 (C), 140.89 (C), 146.86 (C), 153.83 (C); ESI-MS (m/z) 270 (M+H)$^+$; HRMS (ESI) calculated for C$_{16}$H$_{13}$CINO (M + H)$^+$: 270.0686, found 270.0676.

6-chloro-4-(2-chlorophenyl)-2-methylquinolin-3-ol (5c):

![Structure of 6-chloro-4-(2-chlorophenyl)-2-methylquinolin-3-ol (5c)](image)

Yield 58% as white solid: mp 221-223°C; FT-IR (KBr, cm$^{-1}$) 3400, 3019, 1638, 1215, 1068; $^1$H NMR (500 MHz, DMSO-$d_6$) $\delta$ 2.64 (s, 3H), 6.90 (d, $J = 2.25$ Hz, 1H ), 7.37-7.39 (dd, $J = 7.5$, 1.8 Hz, 1H ), 7.49-7.58 (m, 3H), 7.68-7.69 (dd, $J = 7.8$, 1.15 Hz, 1H), 7.92 (d, $J = 8.9$ Hz, 1H ), 9.51 (s, 1H); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$ 21.65 (CH$_3$), 122.54 (CH), 125.07 (C), 125.07 (C), 126.73 (CH), 128.12 (CH), 128.71 (C), 130.19 (CH), 130.92 (CH), 130.98 (CH), 131.12 (C), 132.59 (C), 132.85 (CH), 134.04 (C), 140.66 (C), 147.40 (C), 153.86 (C); ESI-MS (m/z) 304 (M+H)$^+$; HRMS (ESI) calculated for C$_{16}$H$_{12}$Cl$_2$NO (M + H)$^+$: 304.0296, found 304.0286.
6-chloro-4-(2-fluorophenyl)-2-methylquinolin-3-ol (5d):

Yield 63% as light yellow solid: mp 237-239°C; FT-IR (KBr, cm⁻¹) 3399, 3019, 1619, 1215; ¹H NMR (500 MHz, DMSO-d₆) δ 2.64 (s, 3H), 7.07 (s, 1H), 7.38-7.44 (m, 3H), 7.51-7.53 (dd, J = 8.85, 2.3 Hz, 1H), 7.57-7.62 (m, 1H), 7.92 (d, J = 8.85 Hz, 1H ), 9.54 (s, 1H); ¹³C NMR (100 MHz, DMSO-d₆) δ 21.68 (CH₃), 116.60 (d, J = 21.6 Hz, CH), 121.04 (d, J = 16.82 Hz, C), 121.96 (C), 122.61 (CH), 125.33 (CH), 126.83 (CH), 128.88 (C), 131.02 (CH), 131.20 (C), 131.48 (d, J = 8.1 Hz, CH), 133.0 (CH), 140.73 (C), 147.77 (C), 153.83 (C), 161.56 (d, J = 243.59 Hz, C-F); ESI-MS (m/z) 288 (M+H)⁺; HRMS (ESI) calculated for C₁₆H₁₂ClFNO (M + H)⁺: 288.0591, found 288.0581.

6-chloro-4-(2-fluorophenyl)-2-methylquinolin-3-ol (5e):

Yield 56% as white yellow solid: mp 277-279°C; FT-IR (KBr, cm⁻¹) 3400, 3019, 1609, 1215, 1069; ¹H NMR (400 MHz, DMSO-d₆) δ 2.62 (s, 3H), 7.09-7.15 (m, 1H), 7.27 (d, J = 8.24 Hz, 2H), 7.44-7.49 (m, 1H), 7.61-7.65 (m, 2H), 7.74 (d, J = 7.92 Hz, 1H ), 9.07 (s, 1H); ¹³C NMR (100 MHz, DMSO-d₆) δ 21.62 (CH₃), 111.80 (d, J = 21.22 Hz, CH), 117.82 (d, J = 8.74 Hz, C), 121.36 (C), 124.80 (C), 125.29 (CH), 126.19 (d, J = 9.46 Hz, CH), 131.26 (2×CH), 132.14 (2×CH), 135.64 (C), 143.88 (C), 147.25 (C), 153.96 (C), 158.57 (d, J = 251.96 Hz, C-F); ESI-MS (m/z) 332 (M+H)⁺; HRMS (ESI) calculated for C₁₆H₁₂BrFNO (M+H)⁺: 332.0086, found 332.0088.
4-(4-bromophenyl)-2-methylquinolin-3-ol (5f):

Yield 52% as white solid: mp 288-290°C; FT-IR (KBr, cm$^{-1}$) 3399, 3019, 1618, 1215; $^1$H NMR (400 MHz, DMSO-$_d_6$) $\delta$ 2.63 (s, 3H), 7.24 (d, $J = 8.16$ Hz, 1H ), 7.31-7.38 (m, 3H), 7.48-7.52 (m, 1H), 7.74 (d, $J = 8.32$ Hz, 2H), 7.88 (d, $J = 8.16$ Hz, 2H), 9.03 (s, 1H); $^{13}$C NMR (100 MHz, DMSO-$_d_6$) $\delta$ 21.69 (CH$_3$), 121.87 (C), 124.38 (CH), 126.40 (CH), 126.45 (CH), 127.66 (C), 127.78 (C), 128.76 (CH), 132.04 (2×CH), 133.13 (2×CH), 133.55 (C), 142.51 (C), 145.95 (C), 153.02 (C); ESI-MS (m/z) 314 (M+H)$^+$; HRMS (ESI) calculated for C$_{16}$H$_{13}$BrNO (M + H)$^+$: 314.0181, found 314.0184.

4-(4-chlorophenyl)-2-methylquinolin-3-ol (5g):

Yield 55% as white solid: mp 270-273°C; FT-IR (KBr, cm$^{-1}$) 3399, 3019, 1602, 1215, 1070; $^1$H NMR (500 MHz, DMSO-$_d_6$) $\delta$ 2.64 (s, 3H), 7.25 (d, $J = 8.95$ Hz, 1H ), 7.34-7.40 (m, 3H), 7.48-7.57 (m, 3H), 7.91 (d, $J = 1.9$ Hz, 2H), 9.03 (s, 1H); $^{13}$C NMR (100 MHz, DMSO-$_d_6$) $\delta$ 21.79 (CH$_3$), 126.66 (CH), 126.77 (C & CH), 127.29 (CH), 128.58 (CH), 129.11 (2×CH), 129.27 (C), 130.80 (C & 2×CH), 133.77 (C), 142.83 (C), 146.37 (C), 154.68 (C); ESI-MS (m/z) 270 (M+H)$^+$; HRMS (ESI) calculated for C$_{16}$H$_{13}$ClNO (M + H)$^+$: 270.0686, found 270.0677.
4-(4-methoxyphenyl)-2-methylquinolin-3-ol (5h):

Yield 64% as light yellow solid: mp 221-222°C; FT-IR (KBr, cm$^{-1}$) 3399, 3019, 1643, 1215, 1068; $^1$H NMR (400 MHz, DMSO-$d_6$) $\delta$ 2.63 (s, 3H), 3.86 (s, 3H), 7.10-7.14 (m, 2H), 7.27-7.38 (m, 4H), 7.46-7.51 (m, 1H), 7.87-7.89 (m, 1H), 8.82 (s, 1H); $^{13}$C NMR (100 MHz, DMSO-$d_6$) $\delta$ 21.69 (CH$_3$), 55.61 (CH$_3$), 114.54 (2×CH), 124.73 (CH), 126.00 (C), 126.13 (CH), 126.21 (CH), 128.31 (C), 128.67 (CH), 128.84 (C), 132.09 (2×CH), 142.62 (C), 146.14 (C), 152.88 (C), 159.41 (C); ESI-MS (m/z) 266 (M+H)$^+$; HRMS (ESI) calculated for C$_{17}$H$_{16}$NO$_2$ (M + H)$^+$: 266.1181, found 266.1174.
X-Ray Data Collection and Structure Refinement Details:

A good quality single crystal of size 0.20 x 0.20 x 0.20 mm, was selected under a polarizing microscope and was mounted on a glass fiber for data collection. Single crystal X-ray data for compound 2b were collected on the Rigaku Kappa 3 circle diffractometer equipped with the AFC12 goniometer and enhanced sensitivity (HG) Saturn724+ CCD detector in the 4x4 bin mode using the monochromated Mo-Kα radiation generated from the microfocus sealed tube MicroMax-003 X-ray generator equipped with specially designed confocal multilayer optics. Data collection was performed using ω-scans of 0.5° steps at 293(2) K. Cell determination, data collection and data reduction was performed using the Rigaku CrystalClear-SM Expert 2.1 b24¹ software. Structure solution and refinement were performed by using SHELX-97². Refinement of coordinates and anisotropic thermal parameters of non-hydrogen atoms were carried out by the full-matrix least-squares method. The hydrogen atoms attached to carbon atoms were generated with idealized geometries and isotropically refined using a riding model.

ORTEP diagram of compound 2b (CCDC 1492781)


Figure 1 ORTEP diagram drawn with 30% ellipsoid probability for non-H atoms of the asymmetric unit of the crystal structure of compound 2b determined at 293 K.
Table 1 Crystal data and structure refinement details for 2b

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$^1$H NMR Spectra of 2a (400 MHz, CDCl$_3$)

$^{13}$C NMR Spectra of 2a (100 MHz, DMSO-$d_6$)
$^1$H NMR Spectra of 2b (400 MHz, DMSO-$d_6$)

$^{13}$C NMR Spectra of 2b (100 MHz, DMSO-$d_6$)
1H NMR Spectra of 2c (400 MHz, DMSO-$d_6$)

13C NMR Spectra of 2c (100 MHz, DMSO-$d_6$)
H NMR Spectra of 2d (400 MHz, DMSO-\textit{d}_6)

\[ \text{F} \]

\[ \text{N} \]

\[ \text{O} \]

\[ \text{F} \]

\[ \text{N} \]

\[ \text{O} \]

\[ \text{F} \]

1\text{H} NMR Spectra of 2d (400 MHz, DMSO-\textit{d}_6)

\[ \text{F} \]

\[ \text{N} \]

\[ \text{O} \]

\[ \text{F} \]

\[ \text{N} \]

\[ \text{O} \]

\[ \text{F} \]

1\text{C} NMR Spectra of 2d (100 MHz, DMSO-\textit{d}_6)
$^1$H NMR Spectra of 2e (400 MHz, DMSO-$d_6$)

$^{13}$C NMR Spectra of 2e (100 MHz, DMSO-$d_6$)
$^{1}$H NMR Spectra of 2f (400 MHz, DMSO-$d_6$)

$^{13}$C NMR Spectra of 2f (100 MHz, DMSO-$d_6$)
$^1$H NMR Spectra of 2g (400 MHz, DMSO-$d_6$)

$^{13}$C NMR Spectra of 2g (100 MHz, DMSO-$d_6$)
$^1$H NMR Spectra of 2h (400 MHz, DMSO-$d_6$)

$^{13}$C NMR Spectra of 2h (100 MHz, DMSO-$d_6$)
$^{1}$H NMR Spectra of 2i (400 MHz, DMSO-$d_6$)

$^{13}$C NMR Spectra of 2i (100 MHz, DMSO-$d_6$)
$^1$H NMR Spectra of 2j (400 MHz, DMSO-$d_6$)

$^{13}$C NMR Spectra of 2j (100 MHz, DMSO-$d_6$)
$^1$H NMR Spectra of 2k (400 MHz, CD$_3$OD)

$^{13}$C NMR Spectra of 2k (100 MHz, CD$_3$OD)
$^1$H NMR Spectra of 2l (400 MHz, DMSO-$d_6$)

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$^1$H NMR Spectra of 4 (400 MHz, CDCl$_3$)

$^{13}$CNMR Spectra of 4 (100 MHz, CDCl$_3$)
H NMR Spectra of 5a (500 MHz, DMSO-\textit{d}_6)

\[ \text{H NMR Spectra of 5a (500 MHz, DMSO-\textit{d}_6)} \]

\[ \text{13C NMR Spectra of 5a (100 MHz, DMSO-\textit{d}_6)} \]
$^1$H NMR Spectra of 5b (500 MHz, DMSO-$d_6$)

$^{13}$C NMR Spectra of 5b (100 MHz, DMSO-$d_6$)
$^1$H NMR Spectra of 5c (500 MHz, DMSO-$d_6$)

$^{13}$C NMR Spectra of 5c (100 MHz, DMSO-$d_6$)
$^1$H NMR Spectra of 5d (500 MHz, DMSO-$d_6$)

$^{13}$C NMR Spectra of 5d (100 MHz, DMSO-$d_6$)
$^1$H NMR Spectra of 5e (400 MHz, DMSO-$d_6$)

$^{13}$C NMR Spectra of 5e (100 MHz, DMSO-$d_6$)
$^{1}H$ NMR Spectra of 5f (400 MHz, DMSO-$d_6$)

$^{13}C$ NMR Spectra of 5f (100 MHz, DMSO-$d_6$)
$^1$H NMR Spectra of 5g (500 MHz, DMSO-$d_6$)

$^{13}$C NMR Spectra of 5g (100 MHz, DMSO-$d_6$)
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