Electronic Supplementary Material (ESI) for Organic & Biomolecular Chemistry.

Transition Metal-Free Functionalization of 2-Oxindoles via Sequential Aldol and Reductive Aldol Reactions Using Rongalite as C1 Reagent

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General Information: All chemicals and solvents were purchased from Alfa Aesar, SRL, Finar and used as received. Thin layer chromatography was performed on 200 µm aluminium-foil backed silica gel plates and the column chromatography was performed using 100-200 mesh silica gel (Merk). ^{1}H NMR spectra were recorded on Bruker Avance 400 MHz spectrometer, $CDCl_3$ and DMSO-d_6 as solvents and TMS as an internal standard. The following abbreviations were used to explain multiplicities: s = singlet, d = doublet, t = triplet, q = quartet and m = multiplet. Coupling constants, *J* were reported in Hertz unit (Hz). ^{13}C NMR spectra were recorded on Bruker Avance 100 MHz spectrometer, and they were fully decoupled by broad band proton decoupling. Chemical shifts were reported in ppm referenced to the center line of a triplet at 77.16 ppm of chloroform-*d* (a multiplet at 39.52 ppm of DMSO-d_6). Melting points were determined with a Stuart SMP30 apparatus and are uncorrected. FT-IR spectra were recorded on a Perkin Elmer spectrometer. HRMS were analyzed with Agilent Q-TOF 6230. Indolin-2-ones **1a-1w** were synthesized according to the previous reports[1] and 3-methyleneindolin-2-one **6** is prepared using previous report.[2]

General Procedures

General Procedure (A) for synthesis of 3-methylindolin-2-ones (3a-3o). An oven dried 10 mL reaction flask equipped with a magnetic stirring bar was charged with appropriate indolin-2-one-derivative **1** (1 mmol), rongalite **2** (2 mmol), K_2CO_3 (2 mmol) and toluene+H_2O (2 mL, 8:2 v/v). The mixture was stirred at 100 °C for the appropriate time (1-10 h). The progress of the reaction was monitored by TLC using hexanes and ethyl acetate as an eluent. After completion of reaction, toluene is evaporated under vacuum and extracted with ethyl acetate (3 x 10 mL). The organic layers were separated, dried (Na_2SO_4) and evaporated to give a residue that was purified

on a short pad of silica gel by column chromatography using hexanes and ethyl acetate as an eluent.

General Procedure (B) for synthesis of 3-(hydroxymethyl)-3-methylindolin-2-ones (4a-4w). An oven dried 10 mL reaction flask equipped with a magnetic stirring bar was charged with appropriate indolin-2-one-derivative **1** (1 mmol), rongalite **2** (3 mmol), K_2CO_3 (2.5 mmol) and DMSO (2 mL). The mixture was stirred at 80 °C for the appropriate time (15 min-8 h). The progress of the reaction was monitored by TLC using hexanes and ethyl acetate as an eluent. After completion of reaction, reaction mixture was extracted with ethyl acetate (3 x 10 mL). The organic layers were separated, dried (Na_2SO_4) and evaporated to give a residue that was purified on a short pad of silica gel by column chromatography using hexanes and ethyl acetate as an eluent.

Illustration of one-pot methylation and hydroxymethylation by ^{1}H NMR spectroscopy

An oven dried 10 mL reaction flask equipped with a magnetic stirring bar was charged with indolin-2-one **1a** (66 mg, 0.5 mmol), rongalite **2** (3 equiv.), K_2CO_3 (2.5 equiv.) and DMSO-d_6 (1 mL) at 80 °C. A 10 µL aliquot of the reaction mixture was transferred to an NMR tube, diluted with DMSO-d_6 (0.5 mL) and recorded ^{1}H NMR. The ^{1}H NMR spectra of all the aliquots are shown in Figure S1.

Characterization data of the identified compounds are as follows. ^{1}H NMR spectrum of indolin-2-one **1a** is shown in panel [a] (Figure S1). When the reaction mixture is recorded at 1 h, peaks at δ 10.42, 10.34, 3.45, 1.32 and 1.15 ppm were observed, which correspond to the mixture of indolin-2-one **1a**, 3-methylindolin-2-one **3a** and 3-(hydroxymethyl)-3-methylindolin-2-one **4a** (Figure S1, panel [b]). The singlet peaks at δ 10.42 ppm represents the NH proton of **3a**, δ 10.34

ppm represents the NH proton of **4a**, δ 3.45 ppm represents the CH_2 protons of **1a** and δ 1.15 represents the CH_3 protons of **4a**. The doublet at δ 1.32 ppm represents the CH_3 protons of **3a**. Notably, decreasing the intensity of peak at δ 10.42 ppm and increasing the intensity of peak at

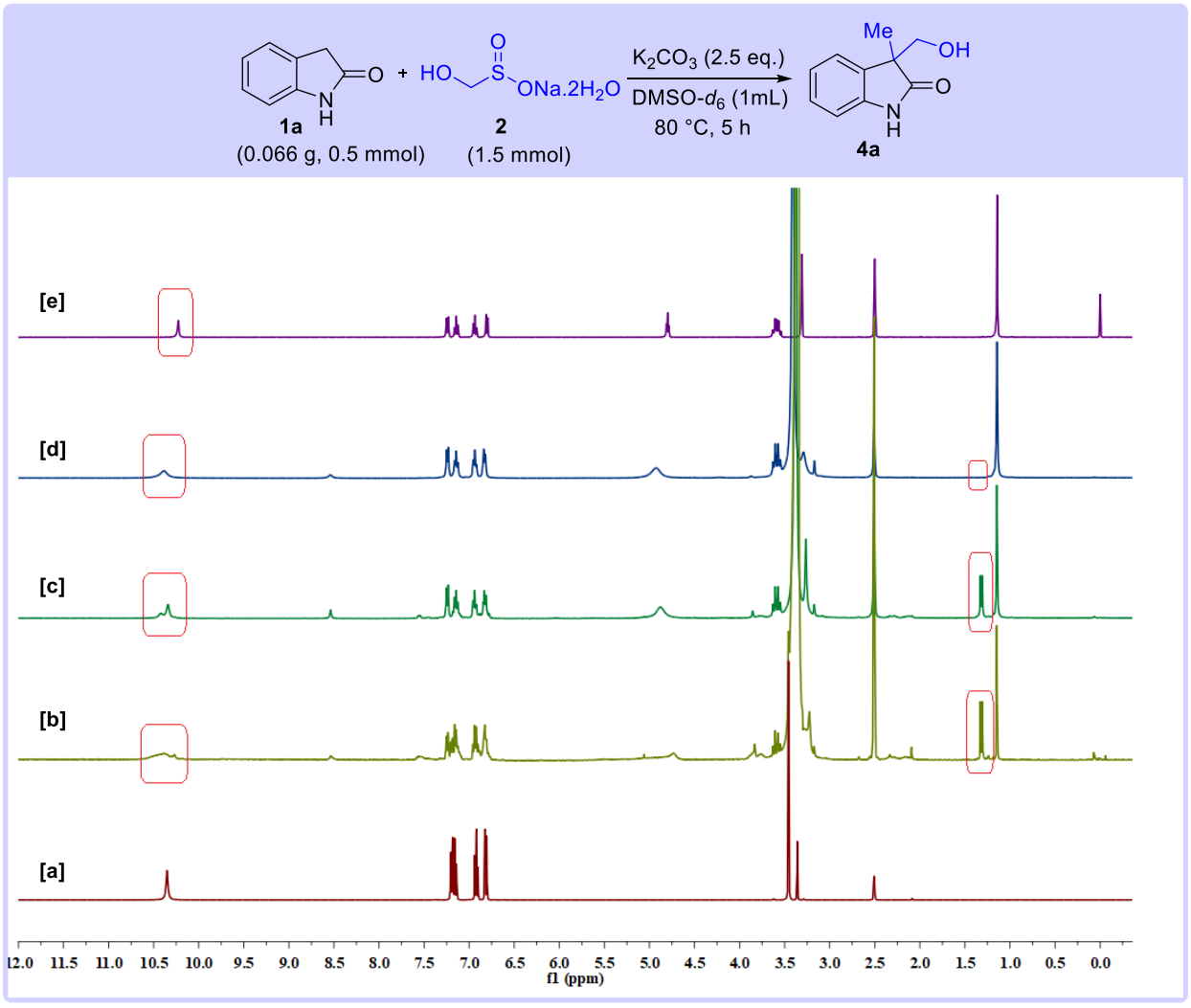


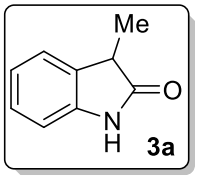
Figure S1 400 MHz ^{1}H NMR spectra of aliquots taken at noted times. All spectra were recorded by diluting an aliquot of the reaction mixture in DMSO-d_6. Panel [a]. 2-oxindole; panel [b]. Aliquot of 2-oxindole **1a**, rongalite **2** and K_2CO_3 after 1 h; panel [c]. after 3 h; panel [d]. After 5 h; panel [e]. Purified compound **4a.**

δ 10.34 ppm was observed and also the intensity of the doublet peak at δ 1.32 ppm is decreased and the peak at δ 1.15 ppm is increased, when aliquot was recorded at 3 h and there is no peak

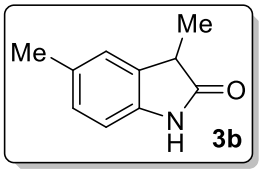
correspond to the indolin-2-one **1a** (Figure S1, panel [c]). Finally, the singlet at δ 10.42 ppm and doublet at δ 1.32 ppm peaks were disappeared after 5 h, which indicates that the intermediate product **3a** is completely converted to final compound **4a** in 5 h using rongalite (Figure S1, panel [d]). The ^{1}H NMR spectrum of aliquot at 5 h is compared with the ^{1}H NMR spectrum of purified compound **4a** (Figure S1, panel [e]).

Characterization data

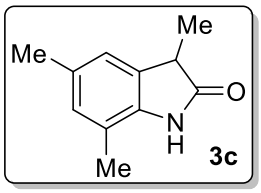
3-methylindolin-2-one (3a). White solid; Yield (138 mg, 94%); mp: 115-116 °C; The title compound is prepared according to the general procedure (A) described as above; FT-IR (KBr, cm^{-1}) 3398, 3016, 2980, 1708, 1641, 881; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 10.30 (s, 1H), 7.24 (d, J = 7.2 Hz, 1H), 7.16 (t, J = 7.6 Hz, 1H), 6.94 (t, J = 7.6 Hz, 1H), 6.81 (d, J = 7.6 Hz, 1H), 3.39 (q, J = 7.6 Hz, 1H), 1.32 (d, J = 7.6 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ (ppm): 181.7, 141.3, 131.3, 127.9, 123.8, 122.4, 109.8, 41.1, 15.2; HRMS (ESI) *m/z*: $[M+H]^+$ calcd for $C_9H_{10}NO$ 148.0762; found 148.0764.



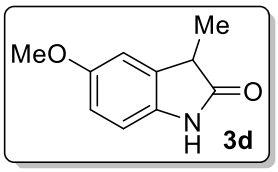
3,5-dimethylindolin-2-one (3b). Off-white crystalline solid; Yield (148 mg, 92%); mp: 148-149 °C; The title compound is prepared according to the general procedure (A) described as above; FT-IR (KBr, cm^{-1}) 3415, 3064, 2982, 1667, 1552, 880; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 9.06 (s, 1H), 7.03 (s, 1H), 7.00 (d, J = 7.6 Hz, 1H), 6.81 (d, J = 8.0 Hz, 1H), 3.43 (q, J = 7.6 Hz, 1H), 2.32 (s, 3H), 1.49 (d, J = 7.6 Hz, 3H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) δ (ppm): 181.7, 138.9, 131.8, 131.4, 128.1, 124.6, 109.5, 41.2, 21.1, 15.3; HRMS (ESI) *m/z*: $[M+H]^+$ calcd for $C_{10}H_{12}NO$ 162.0919; found 162.0917.



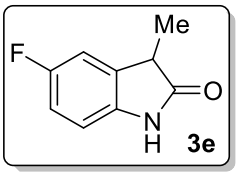
3,5,7-trimethylindolin-2-one (3c). White solid; Yield (159 mg, 91%); mp: 178-179 °C; The title compound is prepared according to the general procedure (A) described as above; FT-IR (KBr, cm^{-1}) 3436, 3077, 2970, 1705, 1625, 741; ^1H NMR (400 MHz, DMSO-d_6) δ (ppm): 10.26 (s, 1H), 6.87 (s, 1H), 6.78 (s, 1H), 3.32 (q, J = 7.6 Hz, 1H), 2.21 (s, 3H), 2.15 (s, 3H), 1.29 (d, J = 7.6 Hz, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, DMSO-d_6) δ (ppm): 180.5, 138.9, 131.5, 130.5, 129.7, 122.2, 118.6, 40.9, 21.1, 16.8, 15.7; HRMS (ESI) *m/z*: $[M+H]^+$ calcd for $C_{11}H_{14}NO$ 176.1075; found 176.1075.



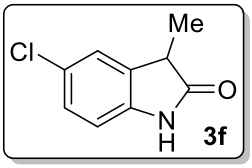
5-methoxy-3-methylindolin-2-one (3d). White solid; Yield (156 mg, 88%); mp: 140-141 °C; The title compound is prepared according to the general procedure (A) described as above; FT-IR (KBr, cm^{-1}) 3456, 3062, 2980, 1697, 1602, 785; ^1H NMR (400 MHz, $CDCl_3$) δ (ppm): 8.97 (s, 1H), 6.77 – 6.73 (m, 2H), 6.66 (dd, J = 8.4, 2.0 Hz, 1H), 3.71 (s, 3H), 3.38 (q, J = 7.6 Hz, 1H), 1.42 (d, J = 7.6 Hz, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ (ppm): 181.5, 155.8, 134.8, 132.7, 112.3, 111.1, 110.1, 55.8, 41.6, 15.3; HRMS (ESI) *m/z*: $[M+H]^+$ calcd for $C_{10}H_{12}NO_2$ 178.0868; found 178.0864.



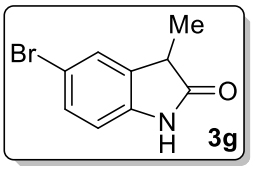
5-fluoro-3-methylindolin-2-one (3e). Off-white solid; Yield (150 mg, 91%); mp: 183-184 °C; The title compound is prepared according to the general procedure (A) described as above; FT-IR (KBr, cm^{-1}) 3437, 3095, 2929, 1713, 1691, 706; ^1H NMR (400 MHz, DMSO-d_6) δ (ppm): 10.38 (s, 1H), 7.23 (dd, J = 8.4, 1.6 Hz, 1H), 7.07 – 7.01 (m, 1H), 6.85 (dd, J = 8.4, 4.4 Hz, 1H), 3.49 (q, J = 7.6 Hz, 1H), 1.38 (d, J = 7.6 Hz, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, DMSO-d_6) δ (ppm): 180.2, 158.5 (d, $^1J_{C\text{-}F}$ = 236.4 Hz), 138.6 (d, $^4J_{C\text{-}F}$ = 1.6 Hz), 133.1 (d, $^3J_{C\text{-}F}$ = 8.0 Hz), 113.8 (d, $^2J_{C\text{-}F}$ = 24.0 Hz), 111.6 (d, $^2J_{C\text{-}F}$ = 24.0 Hz), 110.1



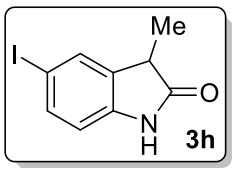
(d, $^{3}J_{C\text{-}F}$ = 8.0 Hz), 41.3 (d, $^{5}J_{C\text{-}F}$ = 1.2 Hz), 15.2; HRMS (ESI) *m/z*: $[M+H]^+$ calcd for C_9H_9FNO 166.0668; found 166.0667.

5-chloro-3-methylindolin-2-one (3f). Off-white solid; Yield (161 mg, 89%); mp: 201-202 °C; 

The title compound is prepared according to the general procedure (A) described as above; FT-IR (KBr, cm^{-1}) 3444, 3061, 2980, 1725, 1670, 817; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 10.45 (s, 1H), 7.34 (s, 1H), 7.21 (dd, *J* = 8.0, 1.6 Hz, 1H), 6.82 (d, *J* = 8.4 Hz, 1H), 3.45 (q, *J* = 7.6 Hz, 1H), 1.33 (d, *J* = 7.6 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, DMSO-d_6) δ (ppm): 179.7, 141.5, 133.5, 127.6, 126.2, 124.1, 110.7, 40.9, 15.2; HRMS (ESI) *m/z*: $[M+H]^+$ calcd for C_9H_9ClNO 182.0373; found 182.0370.

5-bromo-3-methylindolin-2-one (3g). Off-white solid; Yield (203 mg, 90%); mp: 189-190 °C; 

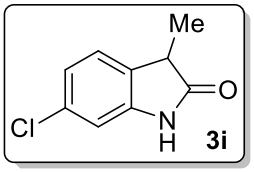
The title compound is prepared according to the general procedure (A) described as above; FT-IR (KBr, cm^{-1}) 3307, 3065, 1716, 1674, 1117, 811; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 8.99 (s, 1H), 7.30 – 7.23 (m, 2H), 6.73 (d, *J* = 8.4 Hz, 1H), 3.40 (q, *J* = 7.6 Hz, 1H), 1.42 (d, *J* = 7.6 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ (ppm): 180.9, 140.3, 133.3, 130.8, 127.1, 115.1, 111.2, 41.2, 15.1; HRMS (ESI) *m/z*: $[M+H]^+$ calcd for C_9H_9BrNO 225.9868; found 225.9862.

5-iodo-3-methylindolin-2-one (3h). White solid; Yield (218 mg, 80%); mp: 173-174 °C; The 

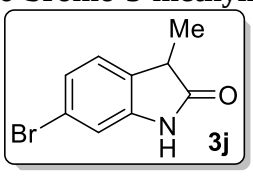
title compound is prepared according to the general procedure (A) described as above; FT-IR (KBr, cm^{-1}) 3302, 3093, 2979, 1703, 1562, 815; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 8.66 (s, 1H), 7.56 – 7.50 (m, 2H), 6.70 (d, *J* = 8.0 Hz, 1H), 3.46 (q,

J = 7.6 Hz, 1H), 1.48 (d, *J* = 7.6 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ (ppm): 180.4, 140.8, 136.8, 133.7, 132.8, 111.7, 84.9, 40.9, 15.1; HRMS (ESI) *m/z*: $[M+H]^+$ calcd for C_9H_9INO 273.9729; found 273.9724.

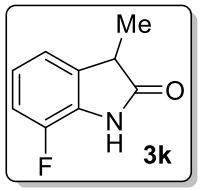
6-chloro-3-methylindolin-2-one (3i). Off-white crystalline solid; Yield (163 mg, 90%); mp: 145-146 °C; The title compound is prepared according to the general procedure (A) described as above; FT-IR (KBr, cm^{-1}) 3444, 3061, 2980, 1725, 1670, 815; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 9.01 (s, 1H), 7.05 (d, *J* = 8.0 Hz, 1H), 6.94 (dd, *J* = 8.0, 1.6 Hz, 1H), 6.86 (d, *J* = 1.6 Hz, 1H), 3.37 (q, *J* = 7.6 Hz, 1H), 1.42 (d, *J* = 7.6 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ (ppm): 181.5, 142.4, 133.6, 129.6, 124.7, 122.3, 110.5, 40.7, 15.2; HRMS (ESI) *m/z*: $[M+H]^+$ calcd for C_9H_9ClNO 182.0373; found 182.0371.



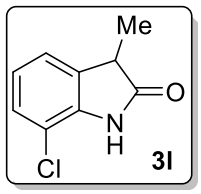
6-bromo-3-methylindolin-2-one (3j). Off-white solid; Yield (201 mg, 89%); mp: 182-183 °C; The title compound is prepared according to the general procedure (A) described as above; FT-IR (KBr, cm^{-1}) 3307, 3065, 1716, 1674, 1117, 809; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 10.46 (s, 1H), 7.21 (d, *J* = 8.0 Hz, 1H), 7.13 (d, *J* = 7.6 Hz, 1H), 6.95 (d, *J* = 1.6 Hz, 1H), 3.39 (q, *J* = 7.6 Hz, 1H), 1.31 (d, *J* = 7.6 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ (ppm): 181.3, 142.5, 130.1, 125.3, 125.1, 121.3, 113.2, 40.8, 15.1; HRMS (ESI) *m/z*: $[M+H]^+$ calcd for C_9H_9BrNO 225.9868; found 225.9862.



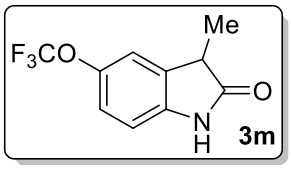
7-fluoro-3-methylindolin-2-one (3k). White crystalline solid; Yield (149 mg, 90%); mp: 147-148 °C; The title compound is prepared according to the general procedure (A) described as above; FT-IR (KBr, cm^{-1}) 3437, 3095, 2929, 1713, 1691, 702; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 10.83 (s, 1H), 7.13 – 7.05 (m, 2H), 6.99 – 6.93 (m, 1H), 3.50 (q, J = 7.6 Hz, 1H), 1.34 (d, J = 7.6 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ (ppm): 180.3, 147.0 (d, $^{1}J_{C\text{-}F}$ = 242.4 Hz), 133.9 (d, $^{3}J_{C\text{-}F}$ = 3.2 Hz), 128.4 (d, $^{2}J_{C\text{-}F}$ = 12.0 Hz), 123.0 (d, $^{3}J_{C\text{-}F}$ = 5.8 Hz), 119.5 (d, $^{4}J_{C\text{-}F}$ = 3.2 Hz), 115.0 (d, $^{2}J_{C\text{-}F}$ = 17.2 Hz), 41.3 (d, $^{5}J_{C\text{-}F}$ = 2.0 Hz), 15.3; HRMS (ESI) *m/z*: $[M+H]^+$ calcd for C_9H_9FNO 166.0668; found 166.0668.



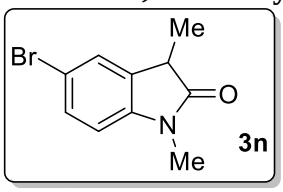
7-chloro-3-methylindolin-2-one (3l). White crystalline solid; Yield (161 mg, 89%); mp: 131-132 °C; The title compound is prepared according to the general procedure (A) described as above; FT-IR (KBr, cm^{-1}) 3399, 3078, 2981, 1719, 1620, 774; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 10.76 (s, 1H), 7.28 – 7.20 (m, 2H), 6.97 (t, J = 7.6 Hz, 1H), 3.54 (q, J = 7.6 Hz, 1H), 1.34 (d, J = 7.6 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ (ppm): 179.8, 138.9, 132.5, 127.9, 123.3, 122.1, 114.9, 41.9, 15.3; HRMS (ESI) *m/z*: $[M+H]^+$ calcd for C_9H_9ClNO 182.0373; found 182.0372.



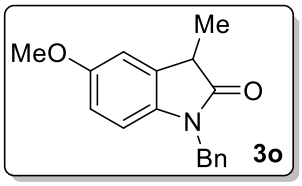
3-methyl-5-(trifluoromethoxy)indolin-2-one (3m). Colorless semi-solid; Yield (173 mg, 75%); The title compound is prepared according to the general procedure (A) described as above; FT-IR (KBr, cm^{-1}) 3435, 3045, 2926, 1716, 1703, 761; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 9.04 (s, 1H), 7.12 – 7.07 (m, 2H), 6.92 – 6.88 (m, 1H), 3.50 (q, J = 7.6 Hz, 1H), 1.52 (d, J = 7.6 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ



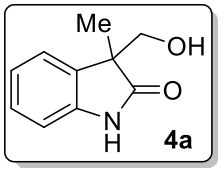
(ppm): 181.3, 144.7, 139.9, 132.6, 121.2, 120.6 (q, $^{1}J_{C\text{-}F}$ = 255 Hz), 117.8, 110.2, 41.4, 15.0; HRMS (ESI) *m/z*: $[M+H]^+$ calcd for $C_{10}H_9F_3NO_2$ 232.0585; found 232.0582.

5-bromo-1,3-dimethylindolin-2-one (3n). White solid; Yield (170 mg, 71%); mp: 80-81 °C; 

The title compound is prepared according to the general procedure (A) described as above; FT-IR (KBr, cm^{-1}) 3091, 2982, 1642, 1550, 1079, 818; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.42 – 7.33 (m, 2H), 6.70 (d, *J* = 8.4 Hz, 1H), 3.43 (q, *J* = 7.6 Hz, 1H), 3.19 (s, 3H), 1.47 (d, *J* = 7.6 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ (ppm): 177.9, 143.1, 132.7, 130.7, 126.8, 115.1, 109.3, 40.6, 26.3, 15.2; HRMS (ESI) *m/z*: $[M+H]^+$ calcd for $C_{10}H_{11}BrNO$ 240.0024; found 240.0026.

5-methoxy-1,3-dimethylindolin-2-one (3o).[3] Colorless semi-solid; Yield (173 mg, 65%); The 

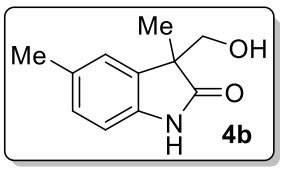
title compound is prepared according to the general procedure (A) described as above; FT-IR (KBr, cm^{-1}) 3444, 3036, 2926, 1707, 1600, 804; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.31 – 7.24 (m, 5H), 6.86 (dd, *J* = 2.4, 0.8 Hz, 1H), 6.69 – 6.65 (m, 1H), 6.59 (d, *J* = 8.4 Hz, 1H), 4.88 (s, 2H), 3.76 (s, 3H), 3.52 (q, *J* = 7.6 Hz, 1H), 1.53 (d, *J* = 7.6 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ (ppm): 178.5, 155.9, 136.5, 136.1, 132.04, 128.8, 127.5, 127.3, 111.9, 111.2, 109.3, 55.8, 43.8, 40.9, 15.7.

3-(hydroxymethyl)-3-methylindolin-2-one (4a). White solid; Yield (161 mg, 91%); mp: 165- 

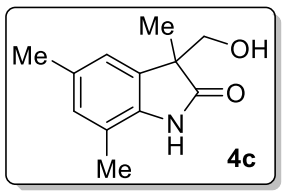
166 °C; The title compound is prepared according to the general procedure (B) described as above; FT-IR (KBr, cm^{-1}) 3351, 3040, 2922, 1707, 1012,

655; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 10.25 (s, 1H), 7.25 (d, J = 7.6 Hz, 1H), 7.15 (td, J = 7.6, 1.6 Hz, 1H), 6.94 (td, J = 7.6, 1.2 Hz, 1H), 6.84 – 6.80 (m, 1H), 4.82 (t, J = 5.6 Hz, 1H), 3.65 – 3.55 (m, 2H), 1.15 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) δ (ppm): 181.1, 142.6, 134.1, 127.9, 123.8, 121.6, 109.5, 66.8, 50.8, 19.6; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_{10}H_{11}NNaO_2$ 200.0687; found 200.0687.

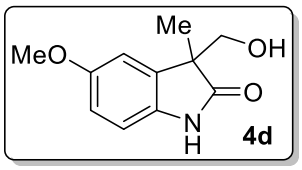
3-(hydroxymethyl)-3,5-dimethylindolin-2-one (4b). White solid; Yield (176 mg, 92%); mp: 160-161 °C; The title compound is prepared according to the general procedure (B) described as above; FT-IR (KBr, cm^{-1}) 3323, 3201, 3024, 2968, 1704, 1490; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 10.14 (s, 1H), 7.07 (d, J = 1.2 Hz, 1H), 6.95 (d, J = 8.0 Hz, 1H), 6.70 (d, J = 8.0 Hz, 1H), 4.80 (t, J = 5.6 Hz, 1H), 3.62 – 3.53 (m, 2H), 2.26 (s, 3H), 1.13 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) δ (ppm): 181.1, 140.1, 134.2, 130.3, 128.0, 124.6, 109.2, 66.8, 50.8, 21.3, 19.6; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_{11}H_{13}NNaO_2$ 214.0844; found 214.0843.



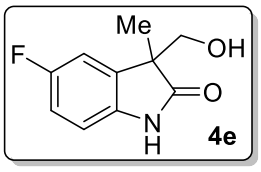
3,5,7-trimethylindolin-2-one (4c). White crystalline solid; Yield (182 mg, 89%); mp: 178-179 °C; The title compound is prepared according to the general procedure (B) described as above; FT-IR (KBr, cm^{-1}) 3501, 3280, 3005, 2971, 1700, 744; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 10.19 (s, 1H), 6.89 (s, 1H), 6.77 (s, 1H), 4.77 (t, J = 5.6 Hz, 1H), 3.61 – 3.54 (m, 2H), 2.23 (s, 3H), 2.16 (s, 3H), 1.12 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) δ (ppm): 181.6, 138.7, 133.9, 130.2, 129.6, 121.8, 118.3, 66.9, 50.9, 21.2, 19.8, 16.9; HRMS (ESI) *m/z*: $[M+H]^+$ calcd for $C_{12}H_{16}NO_2$ 206.1181; found 206.1181.



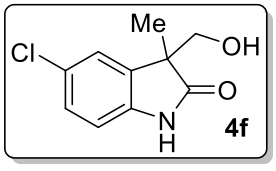
3-(hydroxymethyl)-5-methoxy-3-methylindolin-2-one (4d). White crystalline solid; Yield (180 mg, 87%); mp: 161-162 °C; The title compound is prepared according to the general procedure (B) described as above; FT-IR (KBr, cm^{-1}) 3368, 3049, 2962, 1705, 1654, 617; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 10.06 (s, 1H), 6.91 (s, 1H), 6.72 (d, J = 1.6 Hz, 2H), 4.80 (t, J = 5.6 Hz, 1H), 3.71 (s, 3H), 3.64 – 3.53 (m, 2H), 1.14 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, DMSO-d_6) δ (ppm): 180.9, 155.1, 135.9, 135.5, 112.4, 111.2, 109.6, 66.7, 55.9, 51.3, 19.6; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_{11}H_{13}NNaO_3$ 230.0793; found 230.0793.



5-fluoro-3-(hydroxymethyl)-3-methylindolin-2-one (4e). Off-white crystalline solid; Yield (166 mg, 85%); mp: 219-220 °C; The title compound is prepared according to the general procedure (B) described as above; FT-IR (KBr, cm^{-1}) 3478, 3377, 3065, 2974, 1707, 752; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 10.31 (s, 1H), 7.21 (dd, J = 8.8, 2.8 Hz, 1H), 7.05 – 6.99 (m, 1H), 6.84 (dd, J = 8.4, 4.4 Hz, 1H), 4.93 (t, J = 5.2 Hz, 1H), 3.72 – 3.60 (m, 2H), 1.20 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, DMSO-d_6) δ (ppm): 181.1, 158.4 (d, $^{1}J_{C\text{-}F}$ = 234.4 Hz), 138.8 (d, $^{4}J_{C\text{-}F}$ = 1.0 Hz), 136.1 (d, $^{3}J_{C\text{-}F}$ = 8.4 Hz), 113.9 (d, $^{2}J_{C\text{-}F}$ = 24 Hz), 111.8 (d, $^{2}J_{C\text{-}F}$ = 24.2 Hz), 109.9 (d, $^{3}J_{C\text{-}F}$ = 8.0 Hz), 66.6, 51.5 (d, $^{5}J_{C\text{-}F}$ = 1.2 Hz), 19.3; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_{10}H_{10}FNNaO_2$ 218.0595; found 218.0595.

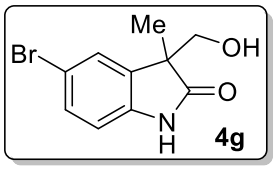


5-chloro-3-(hydroxymethyl)-3-methylindolin-2-one (4f). Off-white solid; Yield (181 mg, 86%); mp: 207-208 °C; The title compound is prepared according to the general procedure (B) described as above; FT-IR (KBr, cm^{-1}) 3393, 3069,

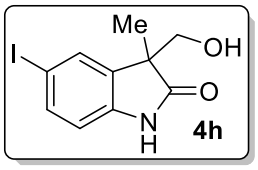


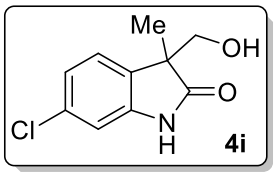
2928, 1719, 1616, 732; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 10.37 (s, 1H), 7.35 (d, J = 2.0 Hz, 1H), 7.20 (dd, J = 8.4, 2.4 Hz, 1H), 6.81 (d, J = 8.4 Hz, 1H), 4.89 (t, J = 5.6 Hz, 1H), 3.68 – 3.55 (m, 2H), 1.16 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) δ (ppm): 180.8, 141.6, 136.3, 127.7, 125.7, 124.2, 110.8, 66.6, 51.4, 19.2; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_{10}H_{10}ClNNaO_2$ 234.0298; found 234.0296.

5-bromo-3-(hydroxymethyl)-3-methylindolin-2-one (4g). White crystalline solid; Yield (218 mg, 85%); mp: 189-190 °C; The title compound is prepared according to the general procedure (B) described as above; FT-IR (KBr, cm^{-1}) 3498, 3022, 2980, 1642, 1554, 816; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 10.12 (s, 1H), 7.06 (s, 1H), 6.94 (d, J = 8.4 Hz, 1H), 6.70 (d, J = 8.0 Hz, 1H), 4.78 (t, J = 5.6 Hz, 1H), 3.63 – 3.52 (m, 2H), 1.14 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) δ (ppm): 181.1, 140.1, 134.2, 130.2, 127.9, 124.5, 109.1, 66.8, 50.8, 19.6; HRMS (ESI) *m/z*: $[M+H]^+$ calcd for $C_{10}H_{11}BrNO_2$ 255.9973; found 255.9971.

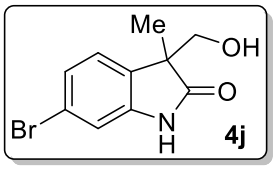


3-(hydroxymethyl)-5-iodo-3-methylindolin-2-one (4h). White solid; Yield (260 mg, 86%); mp: 188-189 °C; The title compound is prepared according to the general procedure (B) described as above; FT-IR (KBr, cm^{-1}) 3456, 3301, 3031, 2929, 1703, 1553, 805; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 10.37 (s, 1H), 7.59 (d, J = 1.6 Hz, 1H), 7.49 (dd, J = 8.0, 1.6 Hz, 1H), 6.67 (d, J = 8.0 Hz, 1H), 4.89 (t, J = 5.2 Hz, 1H), 3.66 – 3.53 (m, 2H), 1.14 (s, 3H); ^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) δ (ppm): 180.5, 142.5, 137.0, 136.4, 132.3, 111.9, 84.6, 66.6, 51.1, 19.3; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_{10}H_{10}INNaO_2$ 325.9654; found 325.9649.

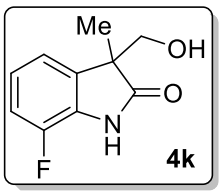


6-chloro-3-(hydroxymethyl)-3-methylindolin-2-one (4i). Off-white solid; Yield (177 mg, 

84%); mp: 175-176 °C; The title compound is prepared according to the general procedure (B) described as above; FT-IR (KBr, cm^{-1}) 3393, 3069, 2928, 1719, 1616, 729; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 10.39 (s, 1H), 7.26 (d, J = 8.0 Hz, 1H), 6.99 (dd, J = 8.0, 2.0 Hz, 1H), 6.82 (d, J = 2.0 Hz, 1H), 4.86 (t, J = 5.6 Hz, 1H), 3.65 – 3.55 (m, 2H), 1.15 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, DMSO-d_6) δ (ppm): 181.0, 144.2, 133.0, 132.2, 125.2, 121.2, 109.5, 66.7, 50.7, 19.2; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_{10}H_{10}ClNNaO_2$ 234.0298; found 234.0297.

6-bromo-3-(hydroxymethyl)-3-methylindolin-2-one (4j). White crystalline solid; Yield (215 

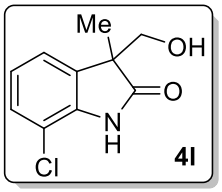
mg, 84%); mp: 157-158 °C; The title compound is prepared according to the general procedure (B) described as above; FT-IR (KBr, cm^{-1}) 3498, 3022, 2980, 1642, 1554, 810; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 10.40 (s, 1H), 7.21 (d, J = 8.0 Hz, 1H), 7.13 (dd, J = 7.6, 2.0 Hz, 1H), 6.94 (d, J = 2.0 Hz, 1H), 4.88 (t, J = 5.6 Hz, 1H), 3.64 – 3.53 (m, 2H), 1.13 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, DMSO-d_6) δ (ppm): 180.9, 144.5, 133.5, 125.7, 124.1, 120.4, 112.3, 66.6, 50.8, 19.1; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_{10}H_{10}BrNNaO_2$ 277.9793; found 277.9792.

7-fluoro-3-(hydroxymethyl)-3-methylindolin-2-one (4k). White solid; Yield (172 mg, 88%); 

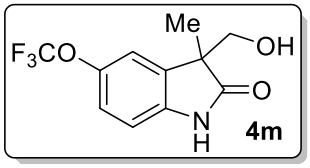
mp: 210-211 °C; The title compound is prepared according to the general procedure (B) described as above; FT-IR (KBr, cm^{-1}) 3478, 3377, 3065, 2978, 1705, 755; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 10.75 (s, 1H), 7.12 (d, J = 7.2 Hz, 1H), 7.09 – 7.04 (m, 1H), 6.99 – 6.93 (m, 1H), 4.89 (t, J = 5.2 Hz, 1H), 3.67 – 3.58 (m, 2H), 1.17 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, DMSO-d_6) δ (ppm): 180.9, 146.7 (d,

$^{1}J_{C\text{-}F}$ = 240 Hz), 137.3 (d, $^{3}J_{C\text{-}F}$ = 3.7 Hz), 129.5 (d, $^{2}J_{C\text{-}F}$ = 11.6 Hz), 122.5 (d, $^{3}J_{C\text{-}F}$ = 5.7 Hz), 119.9 (d, $^{4}J_{C\text{-}F}$ = 2.7 Hz), 115.0 (d, $^{2}J_{C\text{-}F}$ = 17.2 Hz), 66.8, 51.4 (d, $^{5}J_{C\text{-}F}$ = 1.7 Hz), 19.4; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_{10}H_{10}FNNaO_2$ 218.0593; found 218.0595.

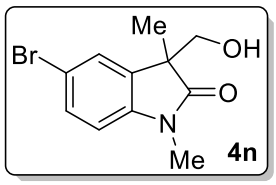
7-chloro-3-(hydroxymethyl)-3-methylindolin-2-one (4l). White crystalline solid; Yield (188 mg, 89%); mp: 196-197 °C; The title compound is prepared according to the general procedure (B) described as above; FT-IR (KBr, cm^{-1}) 3393, 3068, 2929, 1718, 1610, 728; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 10.67 (s, 1H), 7.25 – 7.19 (m, 2H), 6.97 (dd, *J* = 8.4, 7.6 Hz, 1H), 4.89 (t, *J* = 5.6 Hz, 1H), 3.67 – 3.58 (m, 2H), 1.16 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, DMSO-d_6) δ (ppm): 181.0, 140.4, 136.1, 127.9, 122.9, 122.5, 113.8, 66.8, 51.9, 19.4; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_{10}H_{10}ClNNaO_2$ 234.0298; found 234.0293.



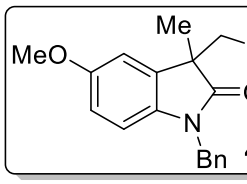
3-(hydroxymethyl)-3-methyl-5-(trifluoromethoxy)indolin-2-one (4m). White solid; Yield (240 mg, 92%); mp: 164-165 °C; The title compound is prepared according to the general procedure (B) described as above; FT-IR (KBr, cm^{-1}) 3407, 3025, 2974, 1709, 1631, 617; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 10.36 (s, 1H), 7.24 (d, *J* = 1.6 Hz, 1H), 7.10 – 7.06 (m, 1H), 6.80 (d, *J* = 8.4 Hz, 1H), 4.83 (t, *J* = 5.2 Hz, 1H), 3.62 – 3.48 (m, 2H), 1.10 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, DMSO-d_6) δ (ppm): 181.1, 143.5, 141.9, 136.0, 121.0, 120.7 (q, $^{1}J_{C\text{-}F}$ = 253.4 Hz), 117.7, 110.1, 66.6, 51.5, 19.2; HRMS (ESI) *m/z*: $[M+H]^+$ calcd for $C_{11}H_{11}F_3NO_3$ 262.0691; found 262.0686.



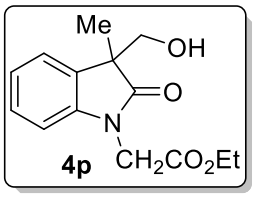
5-bromo-3-(hydroxymethyl)-1,3-dimethylindolin-2-one (4n). White solid; Yield (248 mg, 92%); mp: 185-186 °C; The title compound is prepared according to the general procedure (B) described as above; FT-IR (KBr, cm^{-1}) 3467, 3083, 2979, 1643, 1554, 1075, 818; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 7.53 (d, J = 2.0 Hz, 1H), 7.45 (dd, J = 8.4, 2.4 Hz, 1H), 6.95 (d, J = 8.4 Hz, 1H), 4.90 (t, J = 5.6 Hz, 1H), 3.71 – 3.57 (m, 2H), 3.11 (s, 3H), 1.18 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, DMSO-d_6) δ (ppm): 178.8, 143.6, 135.9, 130.7, 126.5, 114.3, 110.4, 66.6, 51.0, 26.5, 19.2; HRMS (ESI) *m/z*: $[M+H]^+$ calcd for $C_{11}H_{13}BrNO_2$ 270.0130; found 270.0125.



1-benzyl-3-(hydroxymethyl)-5-methoxy-3-methylindolin-2-one (4o). White solid; Yield (282 mg, 95%); mp: 175-176 °C; The title compound is prepared according to the general procedure (B) described as above; FT-IR (KBr, cm^{-1}) 3361, 3065, 2925, 1686, 1496, 719; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 7.32 – 7.23 (m, 5H), 7.02 (d, J = 2.4 Hz, 1H), 6.72 – 6.65 (m, 2H), 4.97 – 4.92 (m, 2H), 4.80 (d, J = 16.0 Hz, 1H), 3.75 – 3.68 (m, 5H), 1.23 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, DMSO-d_6) δ (ppm): 179.2, 155.8, 136.9, 136.5, 134.8, 128.9, 127.6, 127.4, 112.2, 111.2, 109.4, 66.8, 55.9, 51.1, 42.9, 19.7; HRMS (ESI) *m/z*: $[M+H]^+$ calcd for $C_{18}H_{20}NO_3$ 298.1443; found 298.1440.

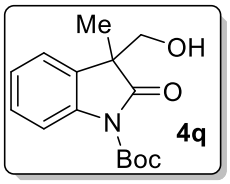


ethyl 2-(3-(hydroxymethyl)-3-methyl-2-oxoindolin-1-yl)acetate (4p). White crystalline solid; Yield (250 mg, 95%); mp: 115-116 °C; The title compound is prepared according to the general procedure (B) described as above; FT-IR (KBr, cm^{-1}) 3427, 3017, 2974, 1642, 1415, 810; ^{1}H NMR (400 MHz, $CDCl_3$) δ



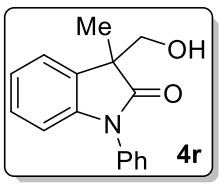
(ppm): 7.23 – 7.16 (m, 2H), 7.04 (t, *J* = 7.6 Hz, 1H), 6.69 (d, *J* = 7.6 Hz, 1H), 4.40 (d, *J* = 2.0 Hz, 2H), 4.13 (q, *J* = 7.2 Hz, 2H), 3.82 (d, *J* = 10.8 Hz, 1H), 3.70 (d, *J* = 10.8 Hz, 1H), 2.07 (s, 1H), 1.36 (s, 3H), 1.17 (d, *J* = 7.1 Hz, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ (ppm): 179.9, 167.6, 142.3, 131.6, 128.3, 123.1, 123.1, 108.2, 67.8, 61.9, 50.2, 41.3, 18.9, 14.1; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_{14}H_{17}NNaO_4$ 286.1055; found 286.1052.

***tert*-butyl 3-(hydroxymethyl)-3-methyl-2-oxoindoline-1-carboxylate (4q).** White solid; Yield



(246 mg, 89%); mp: 90-91 °C; The title compound is prepared according to the general procedure (B) described as above; FT-IR (KBr, cm^{-1}) 3465, 3021, 2980, 1642, 1557, 816; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.80 (d, *J* = 8.4 Hz, 1H), 7.28 – 7.23 (m, 1H), 7.17 – 7.10 (m, 2H), 3.82 (d, *J* = 10.8 Hz, 1H), 3.71 (d, *J* = 10.8 Hz, 1H), 1.85 (s, 1H), 1.58 (s, 9H), 1.35 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ (ppm): 178.6, 149.2, 139.7, 130.6, 128.6, 124.7, 122.6, 115.3, 84.5, 68.2, 50.6, 28.1, 19.9; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_{15}H_{19}NNaO_4$ 300.1212; found 300.1211.

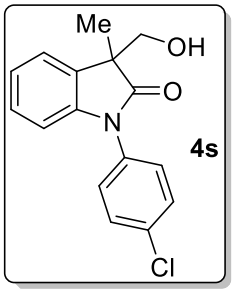
3-(hydroxymethyl)-3-methyl-1-phenylindolin-2-one (4r). White solid; Yield (230 mg, 91%);



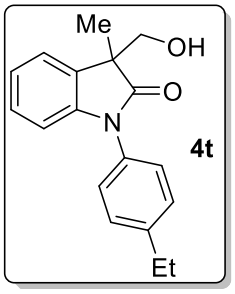
mp: 121-122 °C; The title compound is prepared according to the general procedure (B) described as above; FT-IR (KBr, cm^{-1}) 3415, 3013, 2972, 1689, 1648, 542; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.54 – 7.49 (m, 2H), 7.43 – 7.38 (m, 3H), 7.28 (dd, *J* = 7.2, 0.8 Hz, 1H), 7.22 (td, *J* = 7.8, 1.2 Hz, 1H), 7.12 (td, *J* = 7.6, 1.2 Hz, 1H), 6.85 (d, *J* = 7.8 Hz, 1H), 3.95 – 3.81 (m, 2H), 2.38 (dd, *J* = 8.8, 3.6 Hz, 1H), 1.51 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ (ppm): 179.4, 143.7, 134.4, 131.7, 129.6,

128.3, 128.1, 126.6, 123.2, 123.1, 109.6, 67.9, 50.1, 19.3; HRMS (ESI) *m/z*: $[M+H]^+$ calcd for $C_{16}H_{16}NO_2$ 254.1181; found 254.1179.

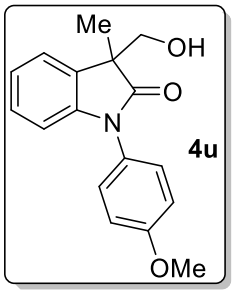
1-(4-chlorophenyl)-3-(hydroxymethyl)-3-methylindolin-2-one (4s). White solid; Yield (258 mg, 90%); mp: 103-104 °C; The title compound is prepared according to the general procedure (B) described as above; FT-IR (KBr, cm^{-1}) 3456, 3083, 2956, 1706, 1610, 757; ^1H NMR (400 MHz, DMSO-d_6) δ (ppm): 7.63 (d, *J* = 8.8 Hz, 2H), 7.46 (d, *J* = 8.8 Hz, 2H), 7.43 (dd, *J* = 7.2, 0.8 Hz, 1H), 7.22 (td, *J* = 7.6, 1.2 Hz, 1H), 7.11 (td, *J* = 7.6, 1.2 Hz, 1H), 6.78 (d, *J* = 7.6 Hz, 1H), 5.01 (t, *J* = 5.2 Hz, 1H), 3.75 (d, *J* = 5.2 Hz, 2H), 1.30 (s, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, DMSO-d_6) δ (ppm): 178.9, 143.5, 133.9, 133.2, 132.5, 130.0, 128.9, 128.1, 124.1, 123.2, 108.9, 67.3, 50.8, 19.6; HRMS (ESI) *m/z*: $[M+H]^+$ calcd for $C_{16}H_{15}ClNO_2$ 288.0791; found 288.0792.



1-(4-ethylphenyl)-3-(hydroxymethyl)-3-methylindolin-2-one (4t). White solid; Yield (258 mg, 90%); mp: 107-108 °C; The title compound is prepared according to the general procedure (B) described as above; FT-IR (KBr, cm^{-1}) 3415, 3013, 2972, 1689, 1648, 541; ^1H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.36 – 7.30 (m, 4H), 7.28 (d, *J* = 7.2 Hz, 1H), 7.22 (td, *J* = 7.6, 1.2 Hz, 1H), 7.11 (td, *J* = 7.6, 1.2 Hz, 1H), 6.85 (d, *J* = 7.6 Hz, 1H), 3.96 – 3.82 (m, 2H), 2.72 (q, *J* = 7.6 Hz, 2H), 2.33 (s, 1H), 1.52 (s, 3H), 1.28 (t, *J* = 7.6 Hz, 3H); $^{13}C\{^1H\}$ NMR (100 MHz, $CDCl_3$) δ (ppm): 179.5, 144.4, 143.9, 131.8, 131.6, 129.1, 128.3, 126.4, 123.1, 122.9, 109.7, 67.9, 49.9, 28.6, 19.3, 15.5; HRMS (ESI) *m/z*: $[M+H]^+$ calcd for $C_{18}H_{20}NO_2$ 282.1494; found 282.1488.

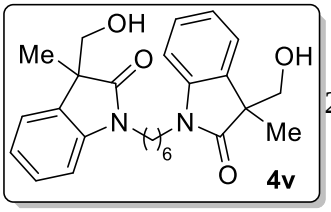


3-(hydroxymethyl)-1-(4-methoxyphenyl)-3-methylindolin-2-one (4u). Colorless semi-solid;



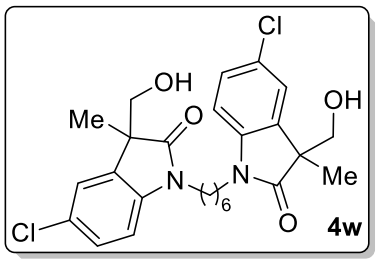
Yield (252 mg, 89%); The title compound is prepared according to the general procedure (B) described as above; FT-IR (KBr, cm^{-1}) 3428, 3073, 2975, 1640, 1414, 508; ^{1}H NMR (400 MHz, $CDCl_3$) δ (ppm): 7.30 (d, J = 9.2 Hz, 2H), 7.28 – 7.25 (m, 1H), 7.21 (td, J = 7.6, 1.2 Hz, 1H), 7.10 (td, J = 7.6, 1.2 Hz, 1H), 7.02 (d, J = 9.2 Hz, 2H), 6.79 (d, J = 7.6 Hz, 1H), 3.92 – 3.80 (m, 5H), 2.48 (s, 1H), 1.49 (s, 3H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$) δ (ppm): 179.7, 159.2, 144.1, 131.6, 128.3, 127.9, 126.9, 123.1, 122.9, 114.9, 109.5, 67.9, 55.6, 50.1, 19.2; HRMS (ESI) *m/z*: $[M+H]^+$ calcd for $C_{17}H_{18}NO_3$ 284.1287; found 284.1279.

1,1'-(hexane-1,6-diyl)bis(3-(hydroxymethyl)-3-methylindolin-2-one) (4v). White solid; Yield (375 mg, 86%); mp: 147-148 °C; The title compound is prepared according to the general



procedure (B) described as above; FT-IR (KBr, cm^{-1}) 3368, 3084, 2924, 1683, 1610, 755; ^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 7.30 (d, J = 7.2 Hz, 2H), 7.22 (t, J = 7.6 Hz, 2H), 7.01 (t, J = 7.6 Hz, 2H), 6.97 (d, J = 7.6 Hz, 2H), 4.82 (t, J = 5.2 Hz, 2H), 3.67 – 3.56 (m, 8H), 1.54 (s, 4H), 1.30 (s, 4H), 1.15 (s, 6H); $^{13}C\{^{1}H\}$ NMR (100 MHz, DMSO-d_6) δ (ppm): 179.2, 143.4, 133.5, 128.0, 123.6, 122.1, 108.6, 66.7, 50.4, 39.4, 27.3, 26.2, 19.6; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_{26}H_{32}N_2NaO_4$ 459.2260; found 459.2252.

1,1'-(hexane-1,6-diyl)bis(5-chloro-3-(hydroxymethyl)-3-methylindolin-2-one) (4w). White



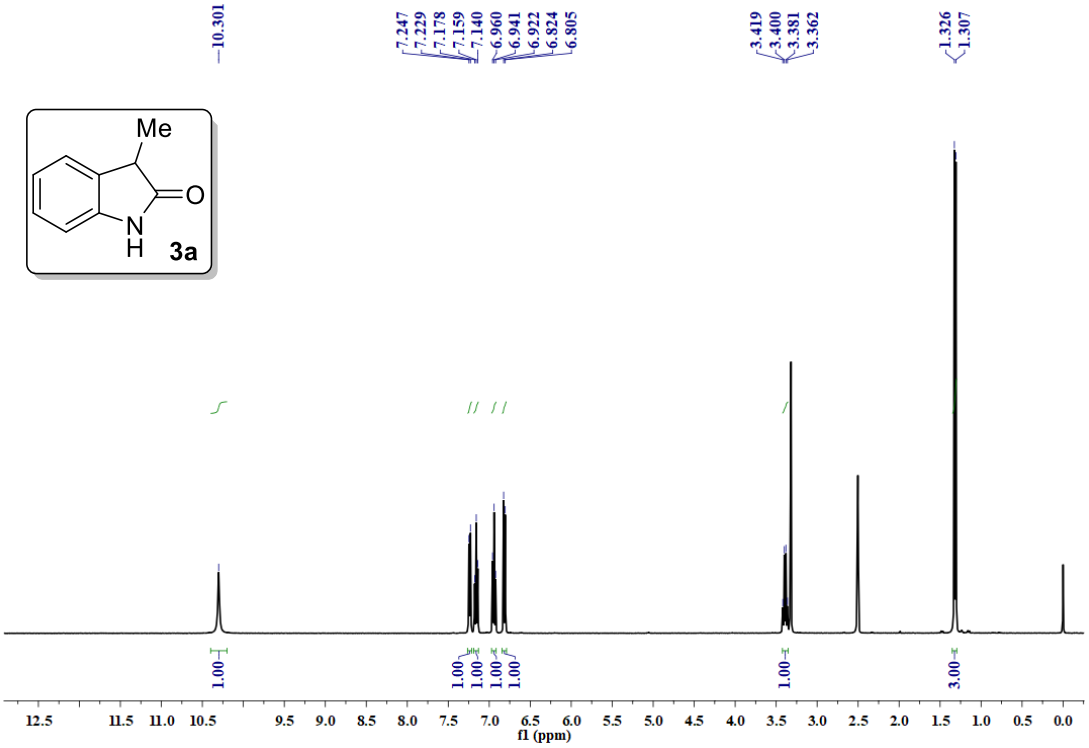
solid; Yield (444 mg, 88%); mp: 192-193 °C; The title compound is prepared according to the general procedure (B) described as above; FT-IR (KBr, cm^{-1}) 3432, 3021, 2977, 1640, 1558, 816;

^{1}H NMR (400 MHz, DMSO-d_6) δ (ppm): 7.41 (d, J = 2.0 Hz, 2H), 7.26 (dd, J = 8.4, 2.0 Hz, 2H), 6.99 (d, J = 8.4 Hz, 2H), 4.88 (t, J = 5.2 Hz, 2H), 3.70 – 3.54 (m, 8H), 1.51 (s, 4H), 1.27 (s, 4H), 1.15 (s, 6H); ^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) δ (ppm): 178.9, 142.4, 135.6, 127.8, 126.3, 124.0, 110.0, 66.6, 50.9, 39.6, 27.2, 26.1, 19.3; HRMS (ESI) *m/z*: $[M+Na]^+$ calcd for $C_{26}H_{30}Cl_2N_2NaO_4$ 527.1480; found 527.1474.

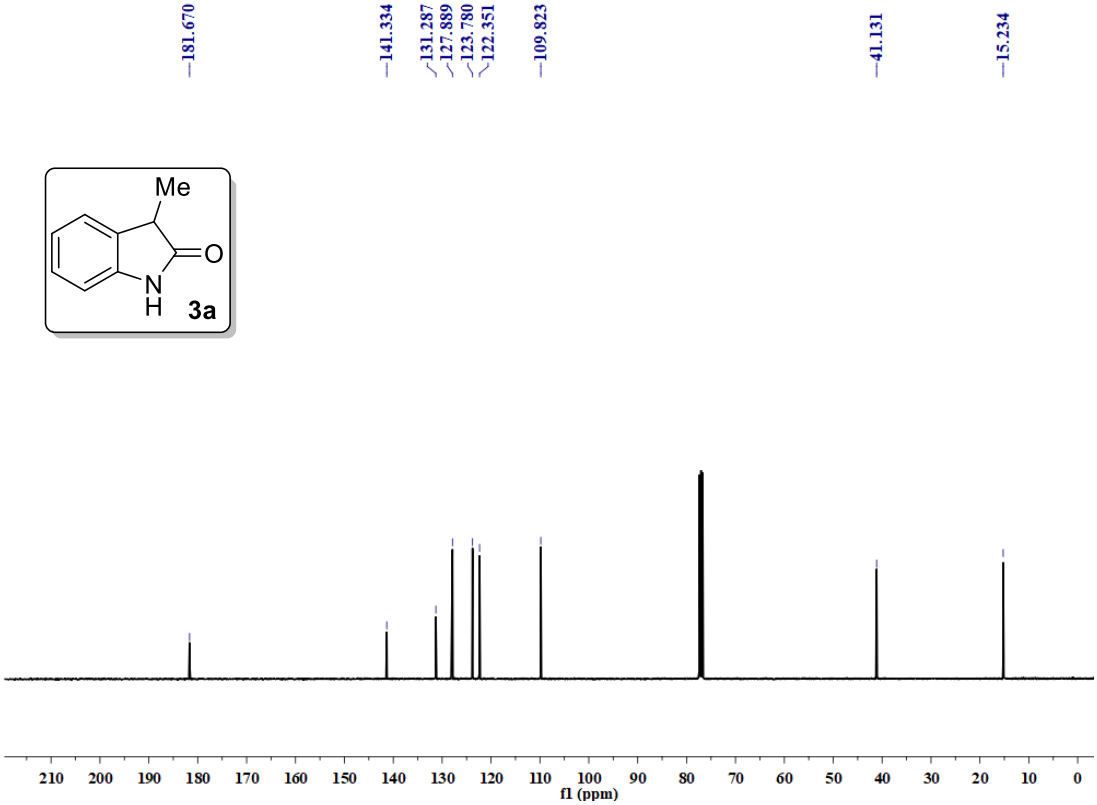
References

1. (a) Y. Lai, L. Ma, W. Huang, X. Yu, Y. Zhang, H. Ji and J. Tian, *Bioorg. Med. Chem. Lett.*, 2010, **20**, 7349; (b) S. Basak, A. Alvarez-Montoya, L. Winfrey, R. L. Melen, L. C. Morrill and A. P. Pulis, *ACS Catal.*, 2020, **10**, 4835.
2. A. Rossettia, A. Sacchettia, M. Bonfantia, G. Rodac, G. Rainoldid and A. Silvani, *Tetrahedron*, 2017, **73**, 4584.
3. G. P. da Silva, A. Ali, R. C. da Silva, H. Jiang and M. W. Paixao, *Chem. Commun.*, 2015, **51**, 15110.

^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 3-methylindolin-2-one (3a)

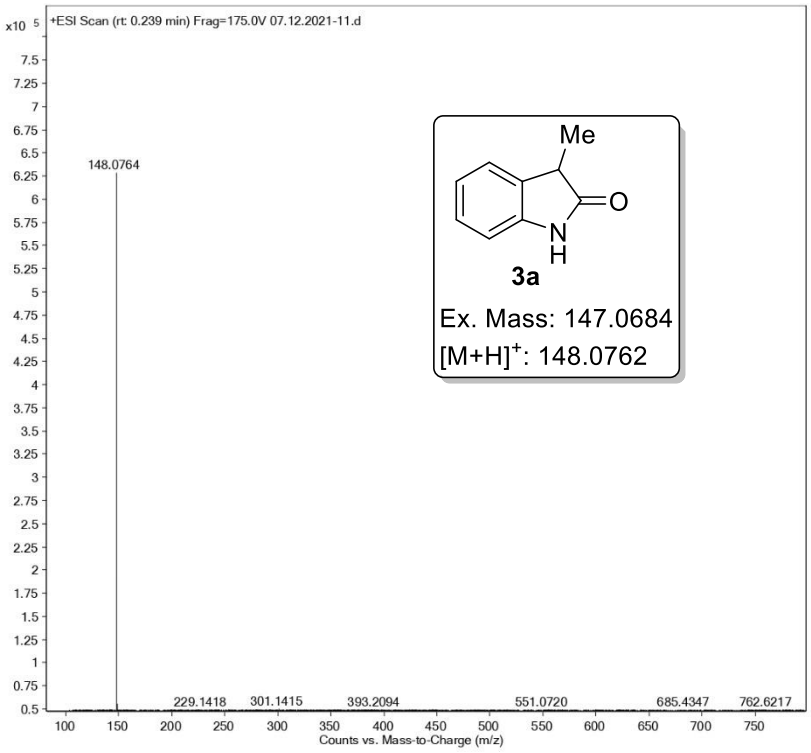


^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of 3-methylindolin-2-one (3a)

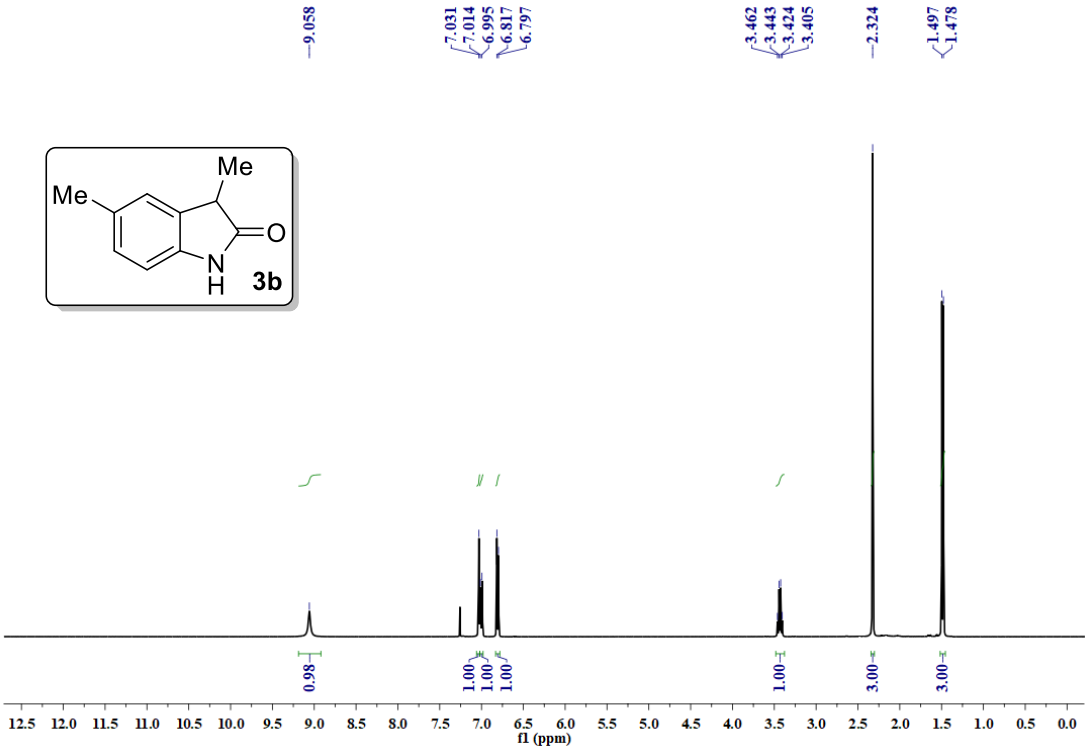


HRMS of 3-methylindolin-2-one (3a)

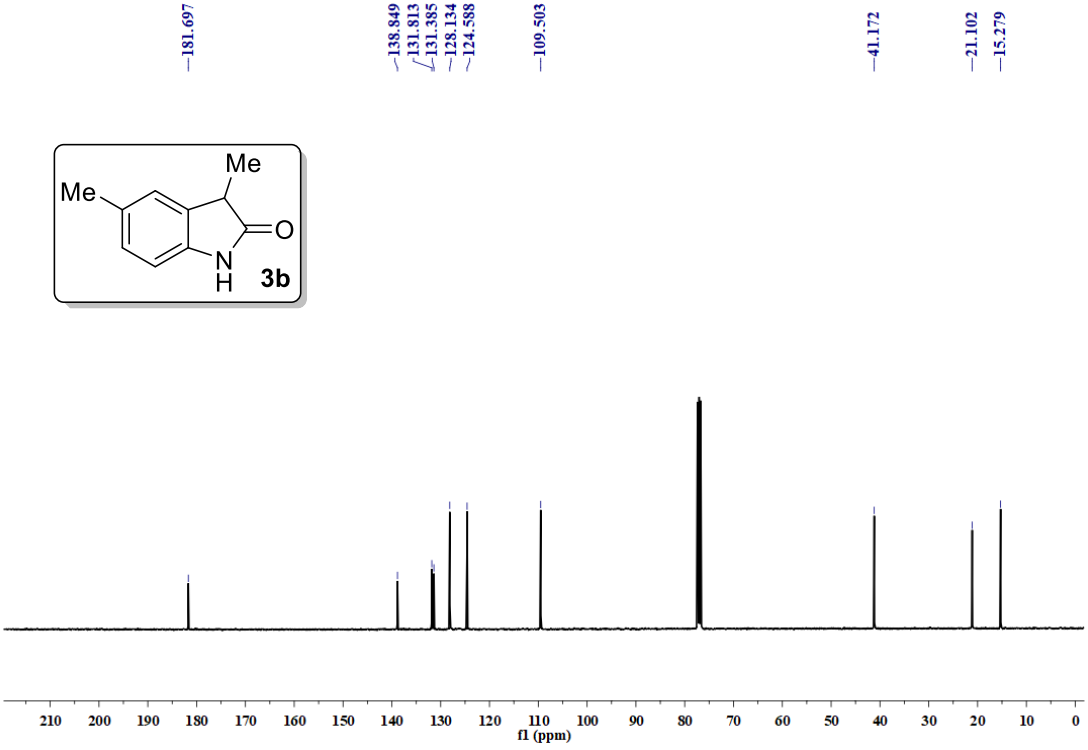
Sample Name	shivaparwathi-1	**Position**	P1-B2	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-11.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 5:33:18 PM



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3,5-dimethylindolin-2-one (3b)

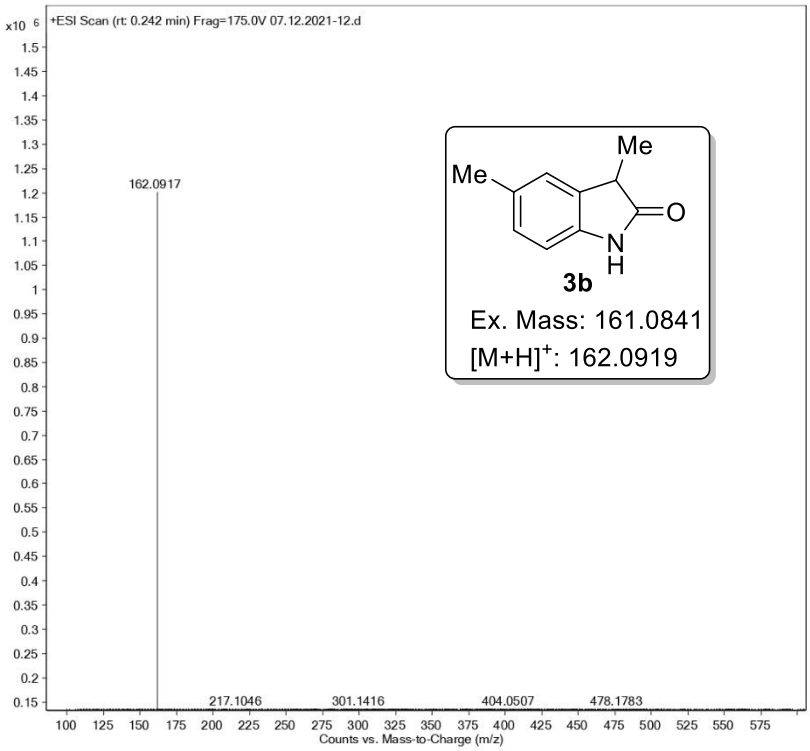


^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of 3,5-dimethylindolin-2-one (3b)

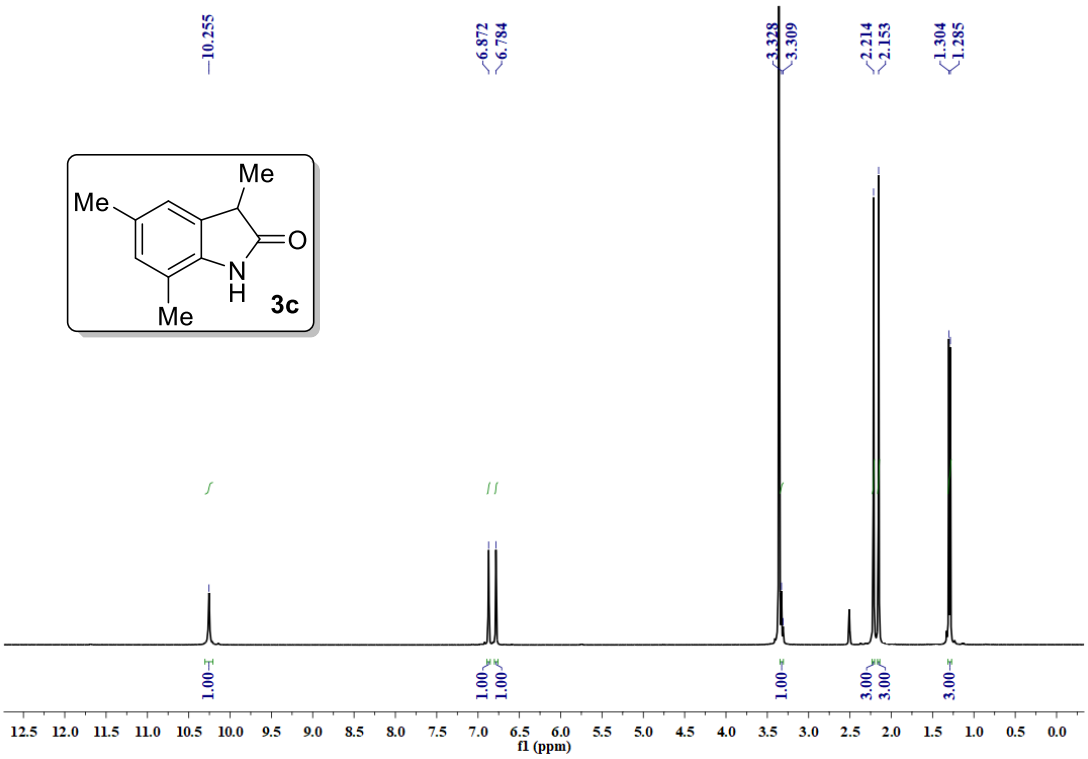


HRMS of 3,5-dimethylindolin-2-one (3b)

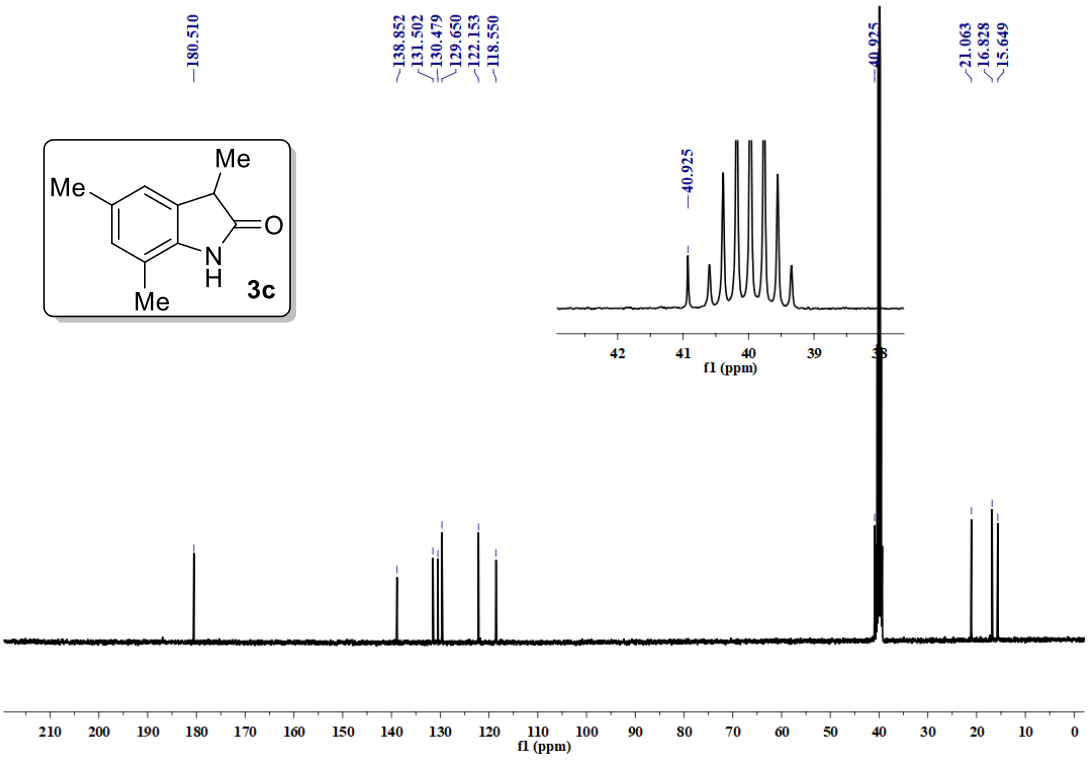
Sample Name	shivaparwathi-2	**Position**	P1-B3	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-12.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 5:37:17 PM



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 3,5,7-trimethylindolin-2-one (3c)

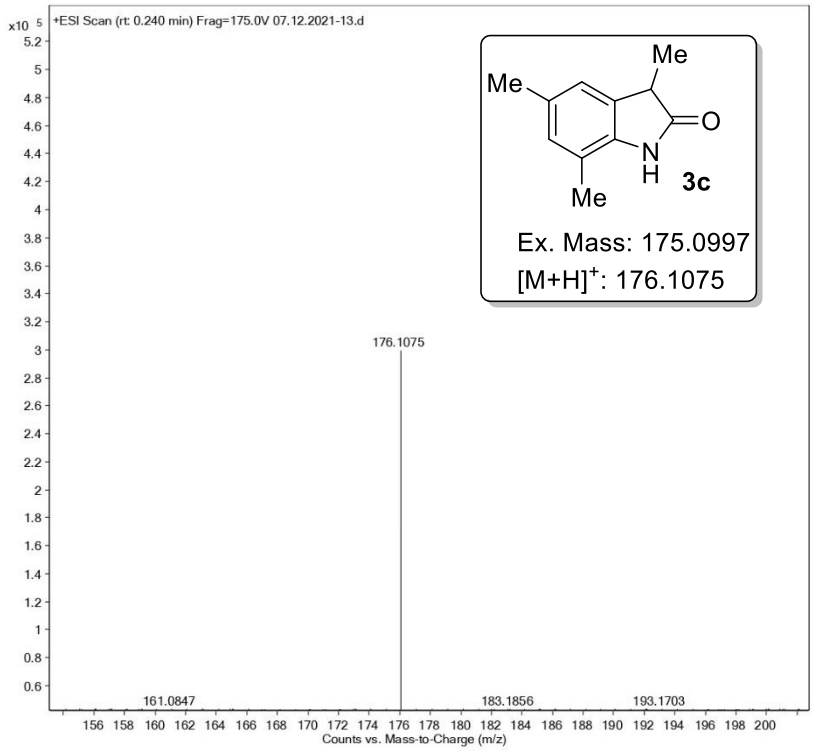


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 3,5,7-trimethylindolin-2-one (3c)

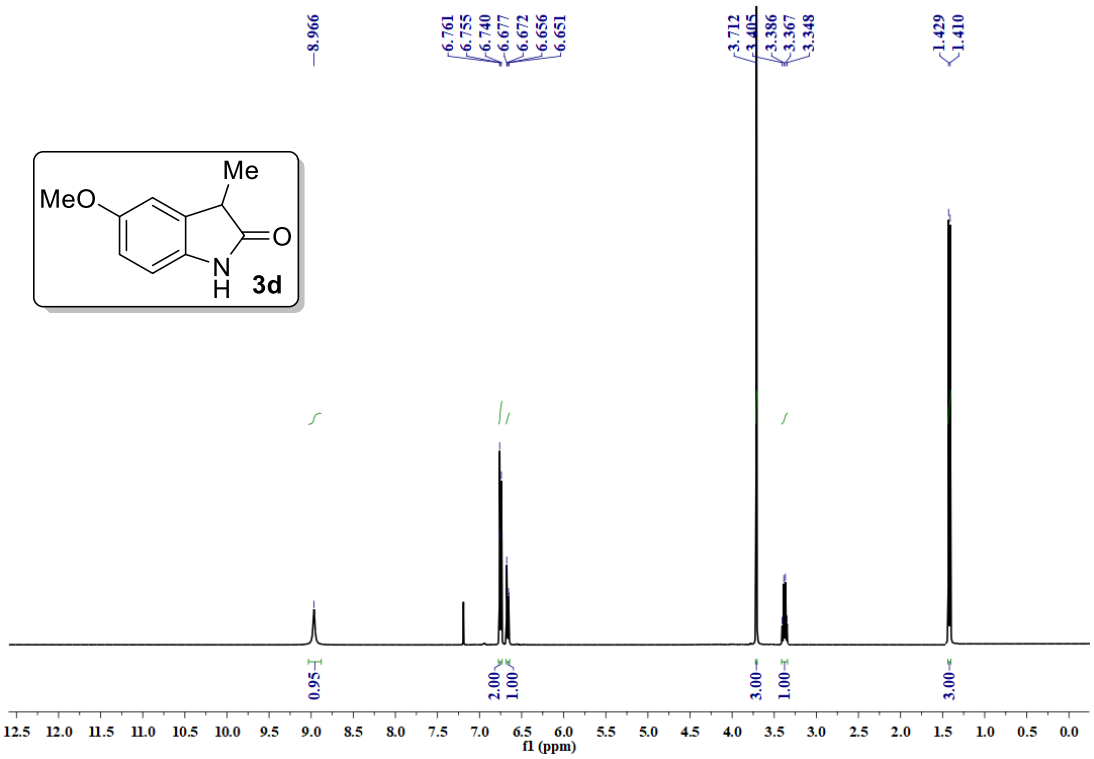


HRMS of 3,5,7-trimethylindolin-2-one (3c)

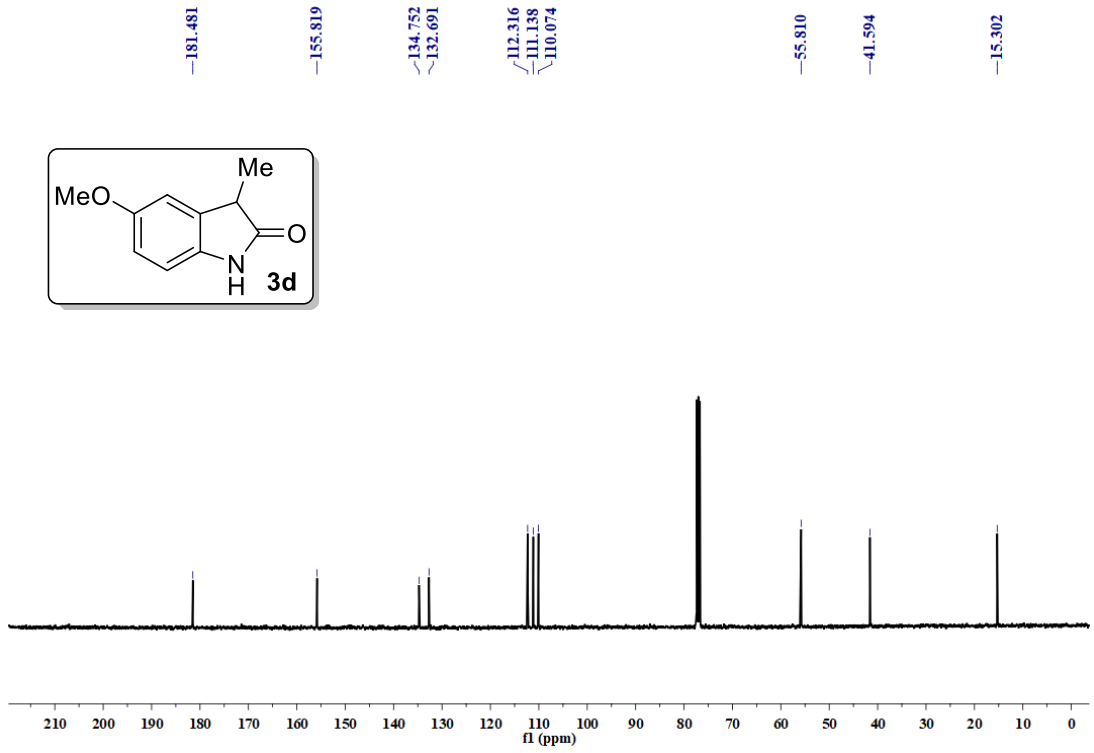
Sample Name	shivaparwathi-3	**Position**	P1-B4	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-13.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 5:41:15 PM



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 5-methoxy-3-methylindolin-2-one (3d)

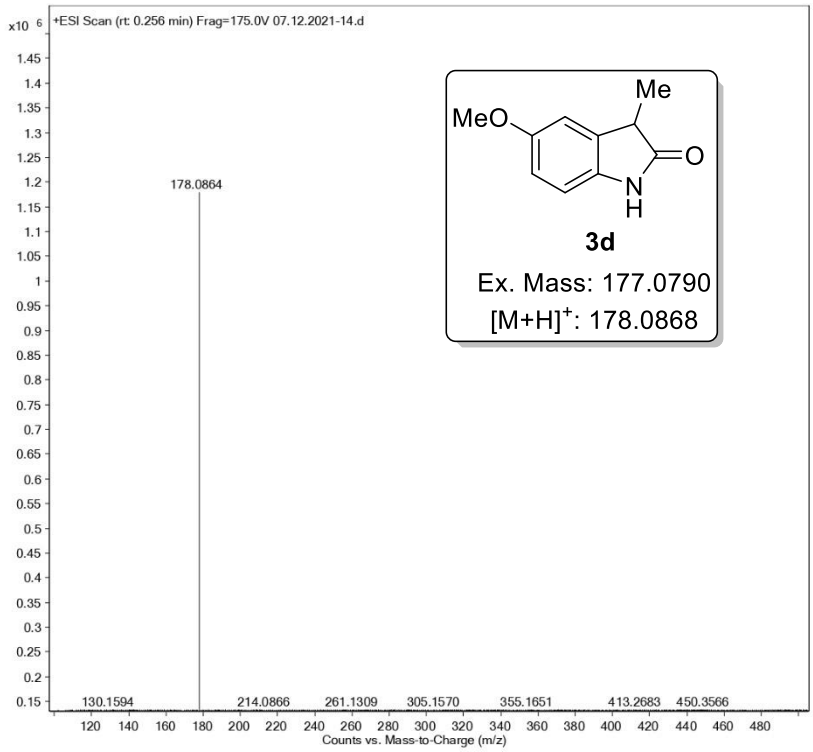


^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of 5-methoxy-3-methylindolin-2-one (3d)

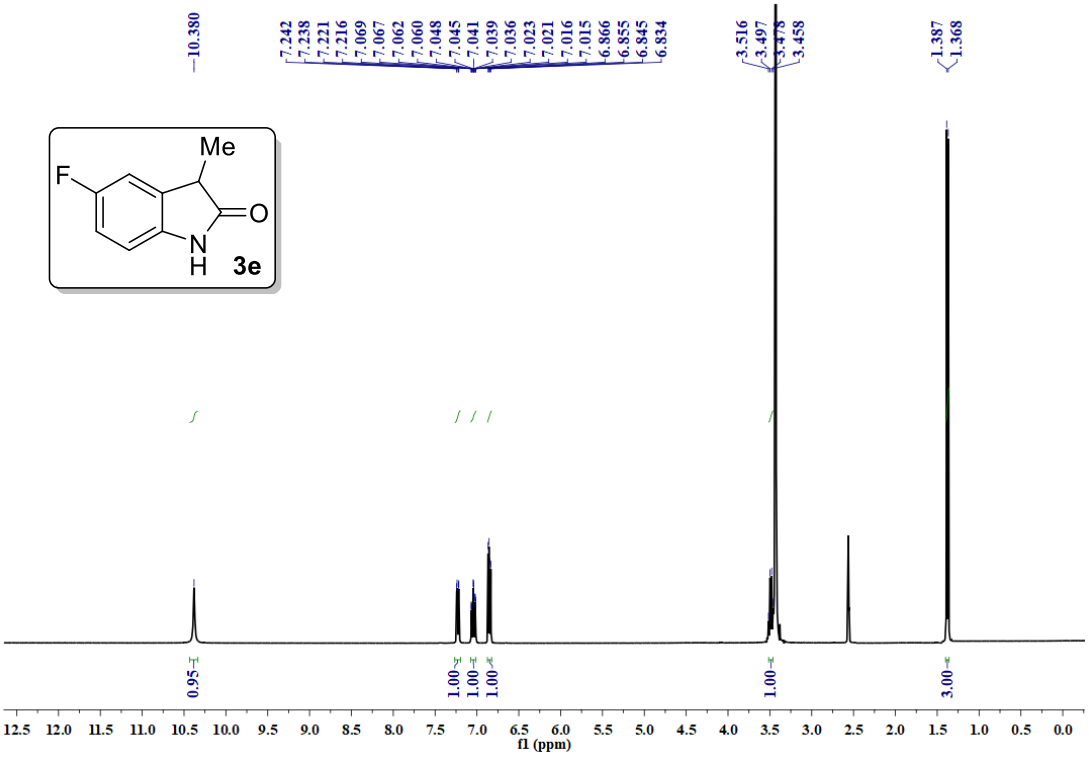


HRMS of 5-methoxy-3-methylindolin-2-one (3d)

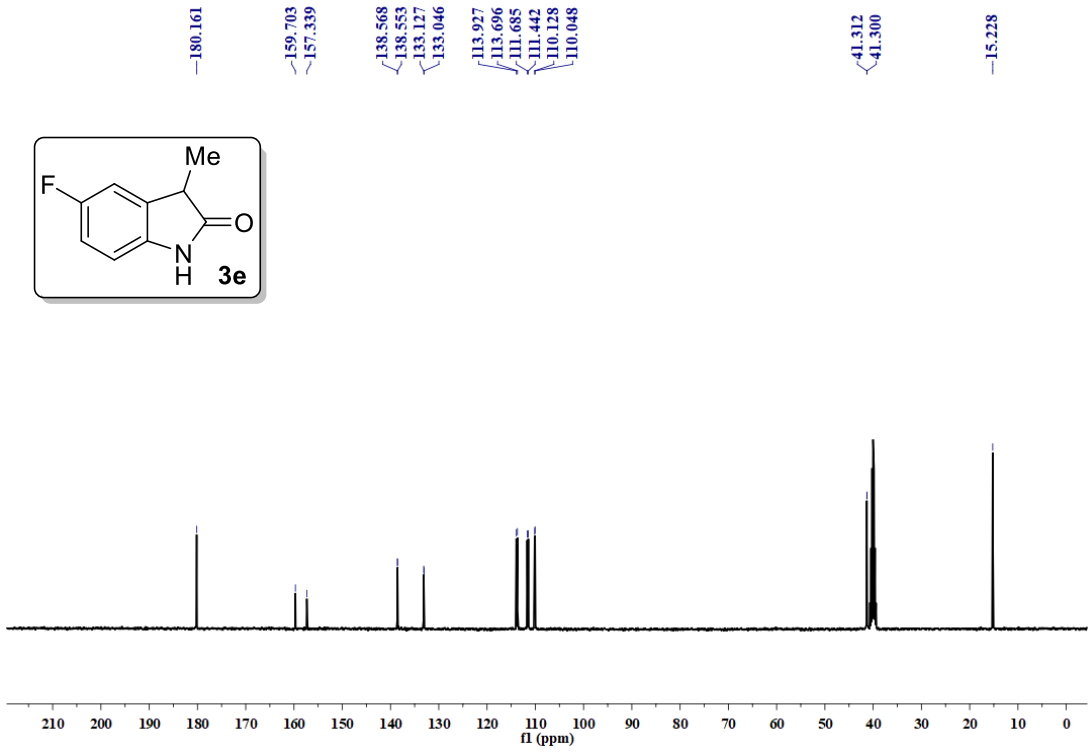
Sample Name	shivaparwathi-4	**Position**	P1-B5	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-14.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 5:45:14 PM



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 5-fluoro-3-methylindolin-2-one (3e)

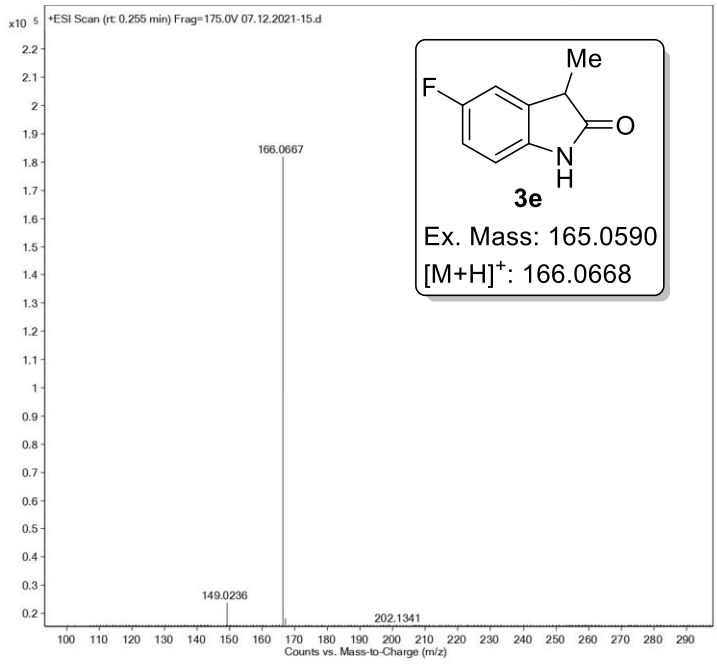


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 5-fluoro-3-methylindolin-2-one (3e)

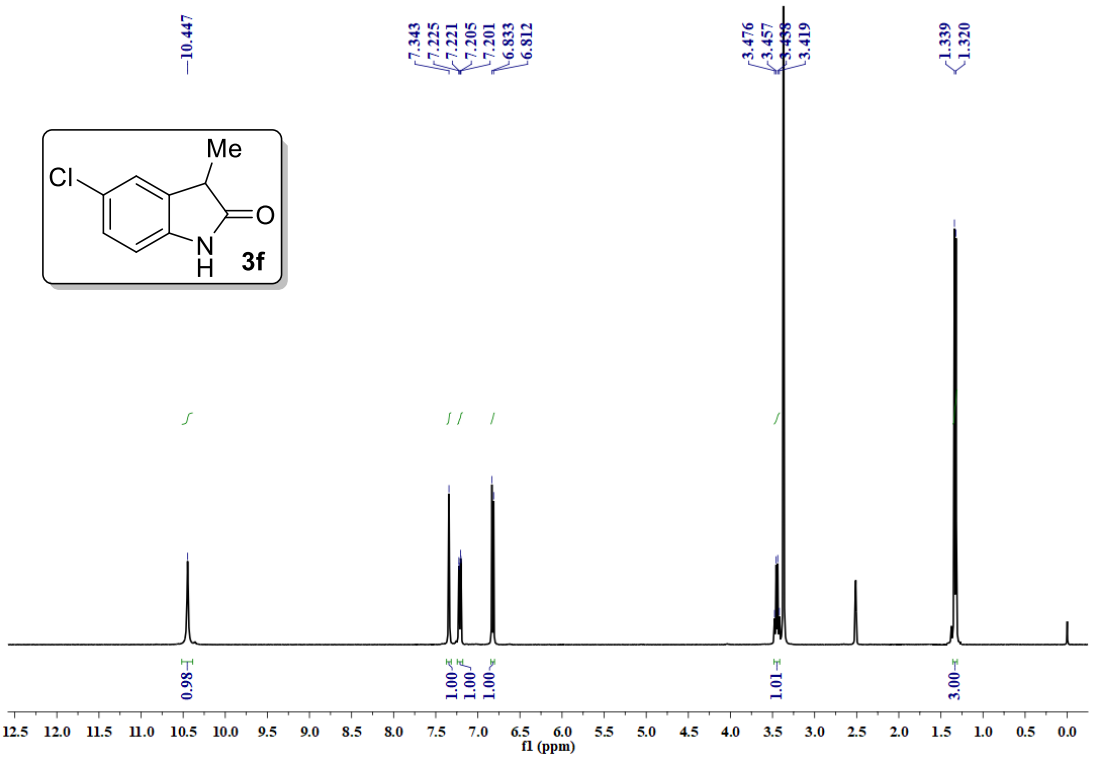


HRMS of 5-fluoro-3-methylindolin-2-one (3e)

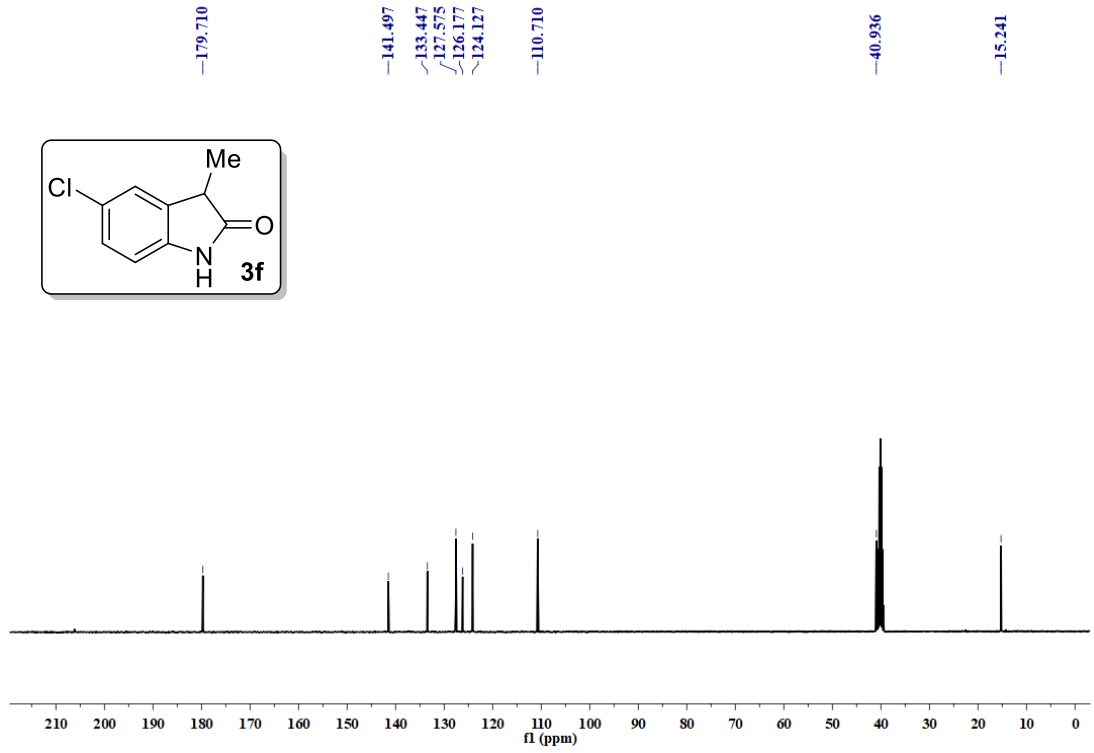
Sample Name	shivaparwathi-5	**Position**	P1-B6	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-15.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 5:49:13 PM



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 5-chloro-3-methylindolin-2-one (3f)

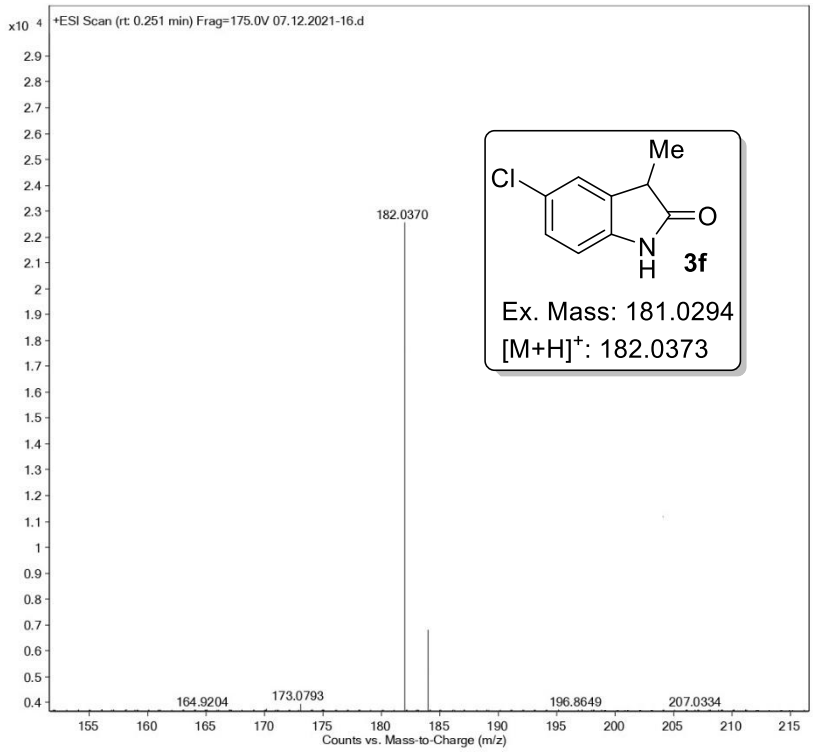


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 5-chloro-3-methylindolin-2-one (3f)

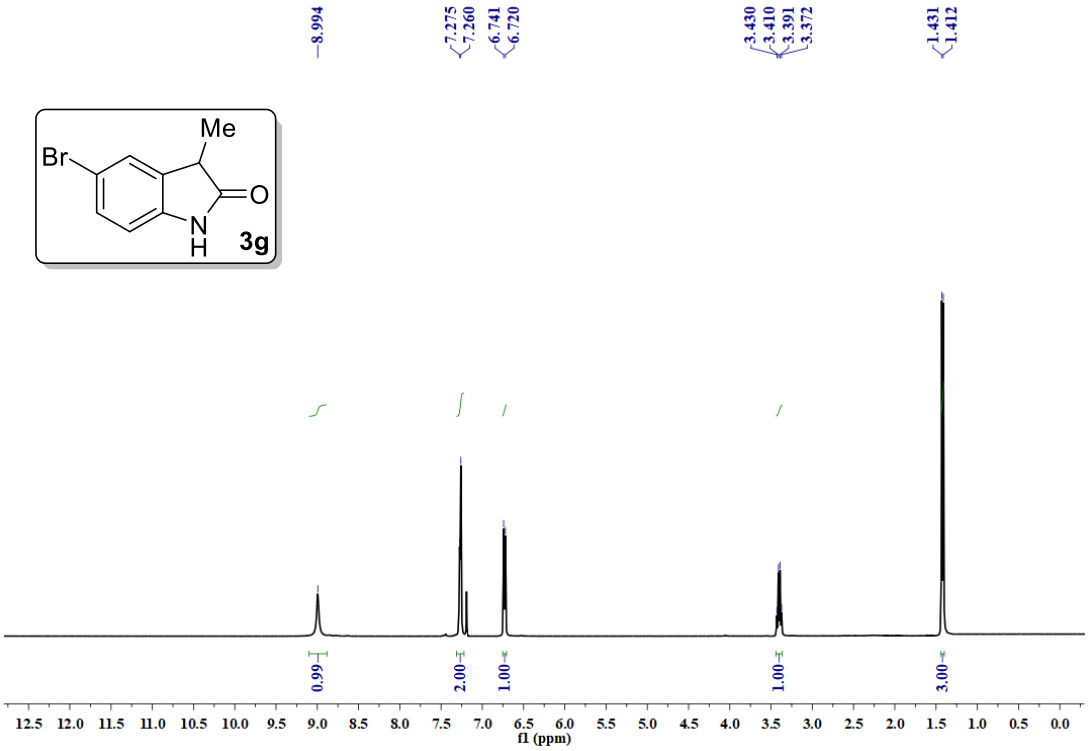


HRMS of 5-chloro-3-methylindolin-2-one (3f)

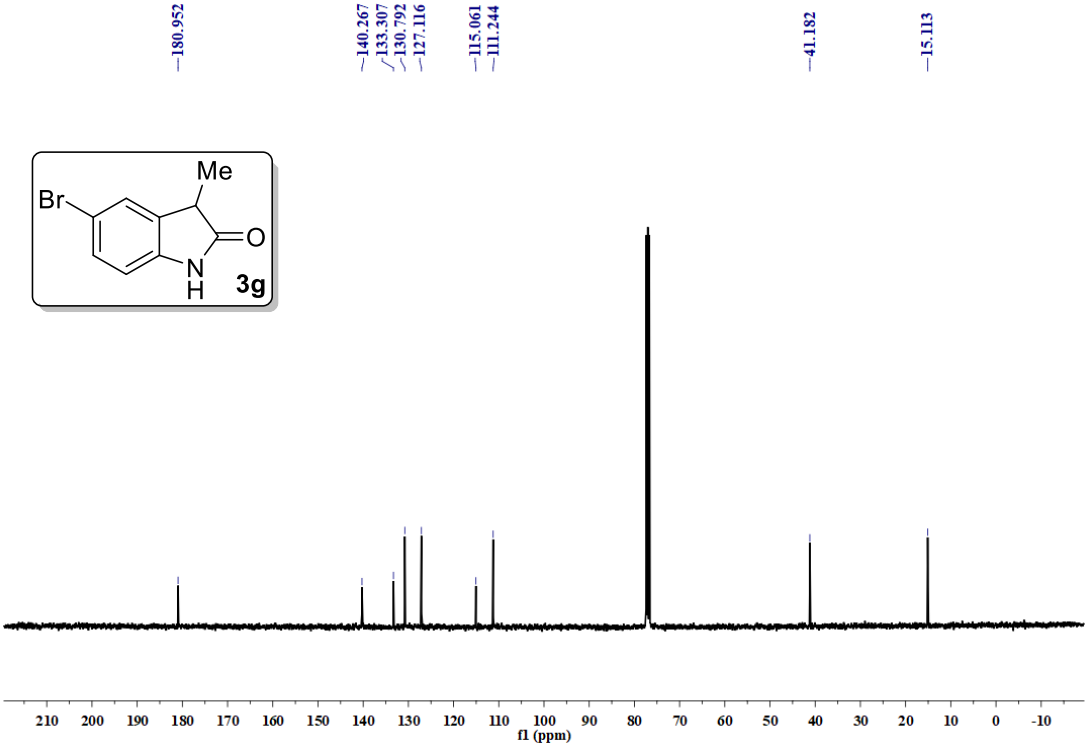
Sample Name	shivaparwathi-6	**Position**	P1-B7	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-16.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 5:53:11 PM



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 5-bromo-3-methylindolin-2-one (3g)

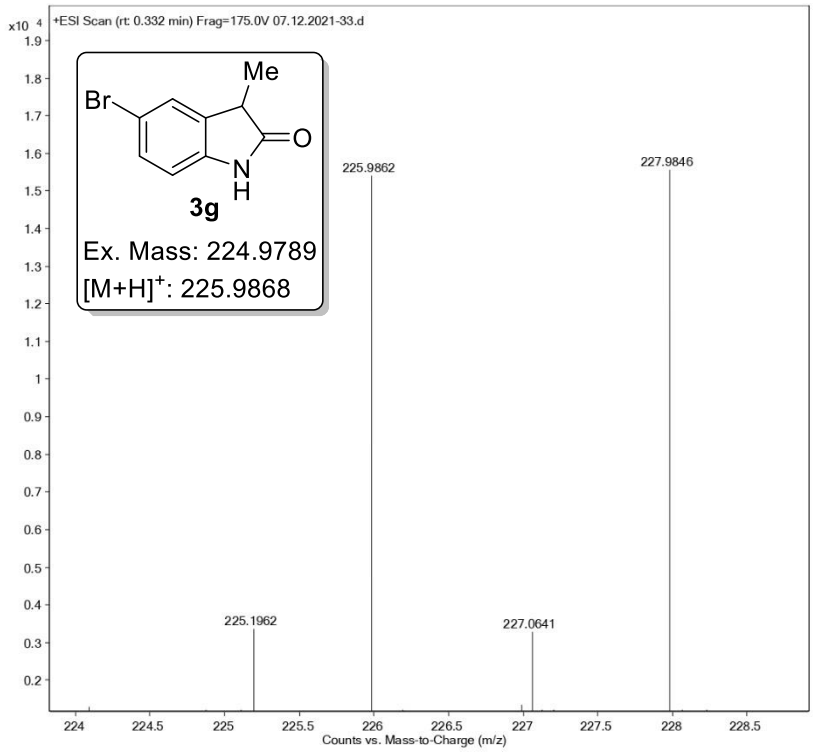


^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of 5-bromo-3-methylindolin-2-one (3g)

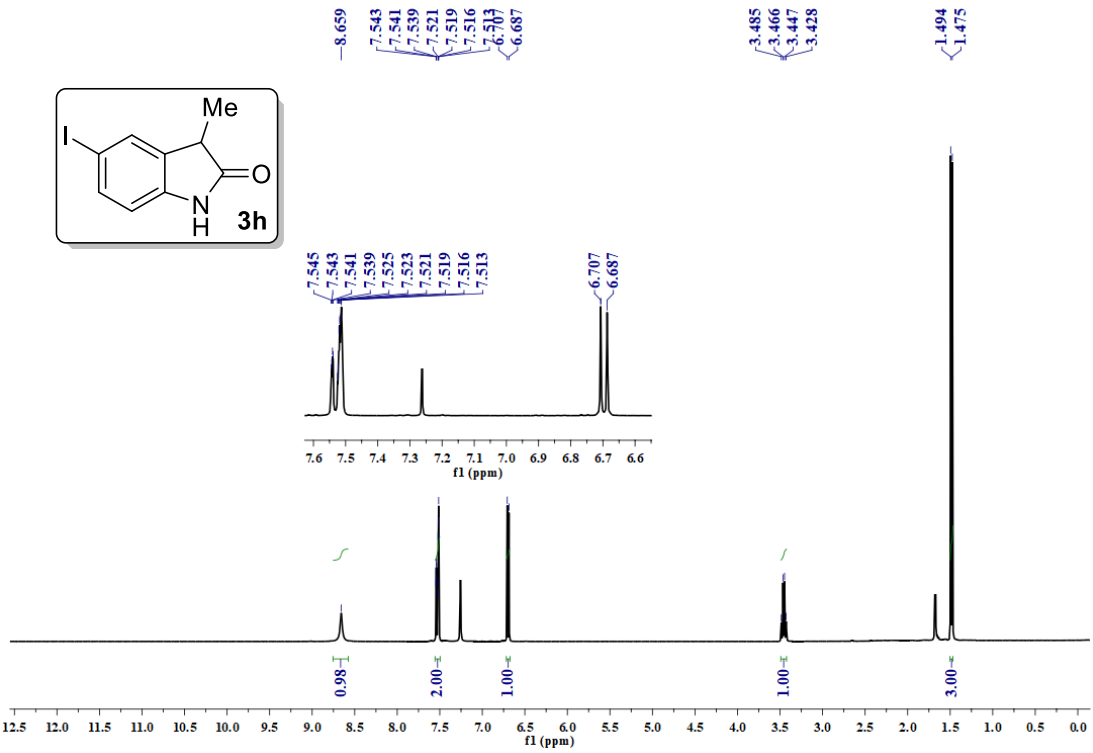


HRMS of 5-bromo-3-methylindolin-2-one (3g)

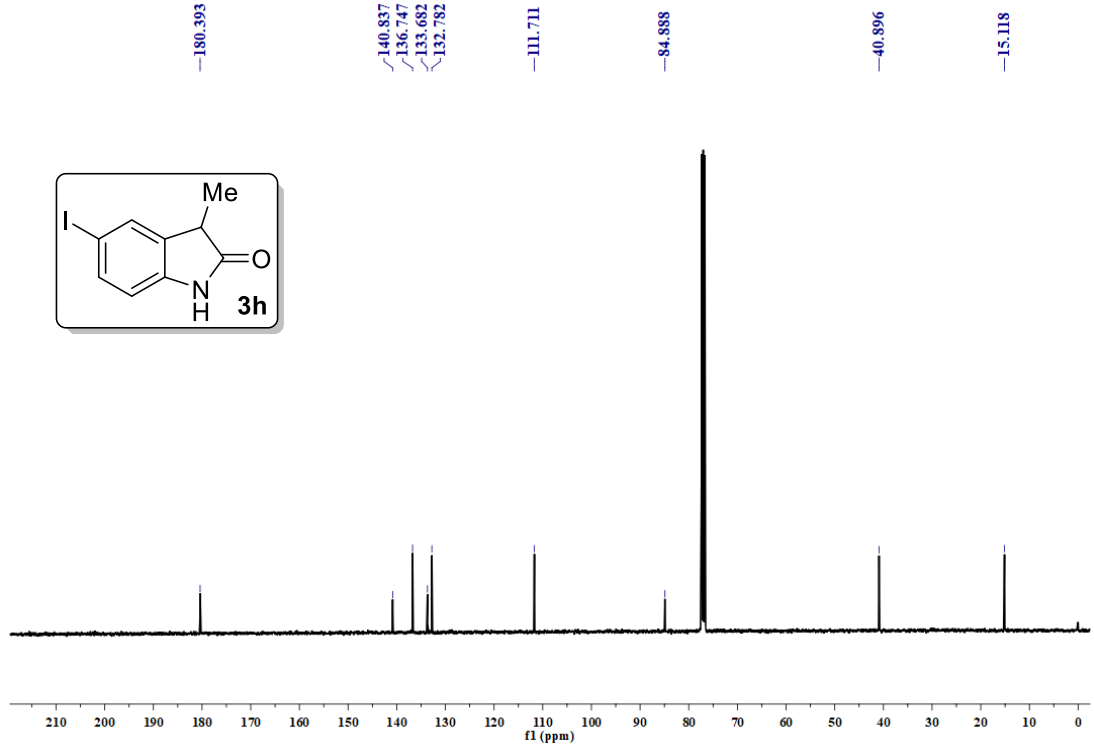
Sample Name	5-broxi-2	**Position**	P1-D6	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-33.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 7:24:20 PM



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 5-iodo-3-methylindolin-2-one (3h)

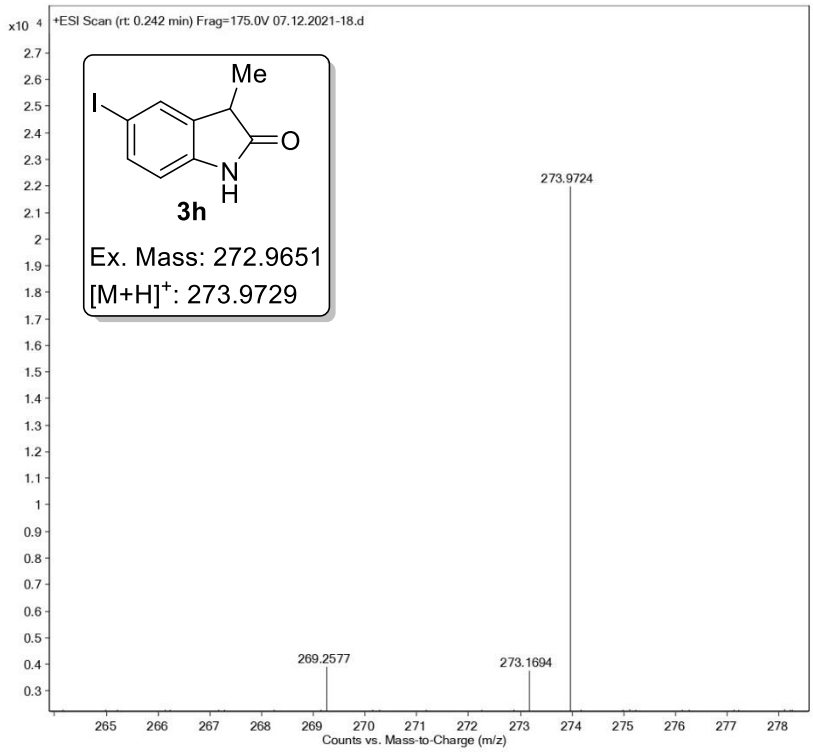


^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of 5-iodo-3-methylindolin-2-one (3h)

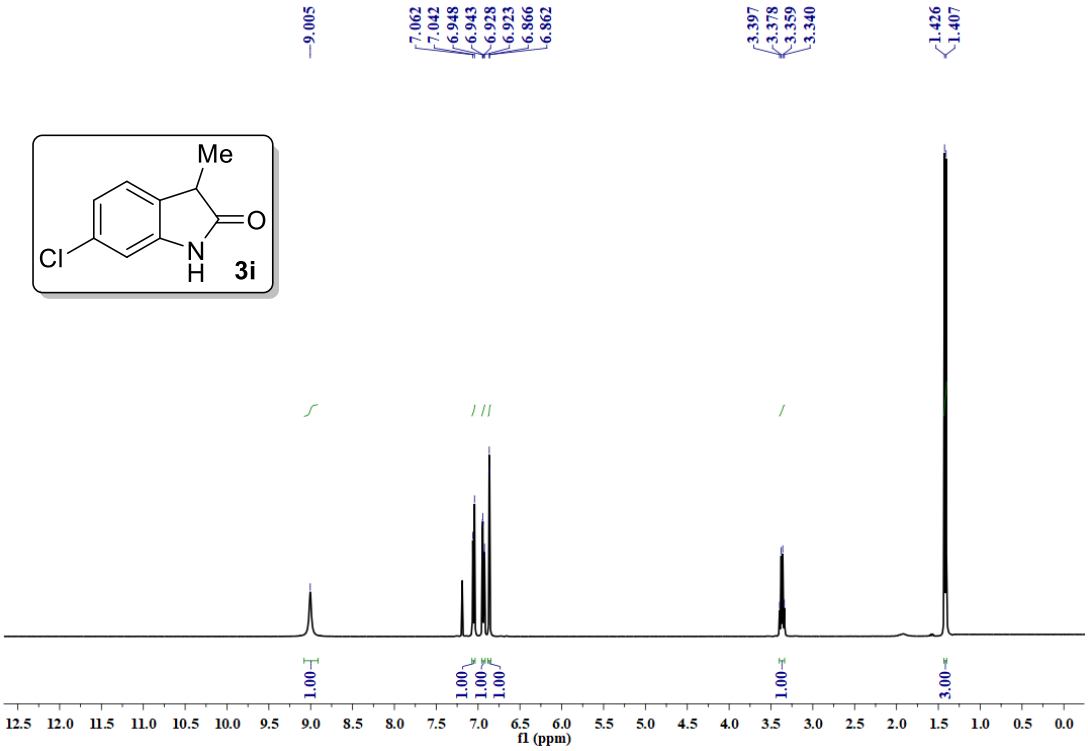


HRMS of 5-iodo-3-methylindolin-2-one (3h)

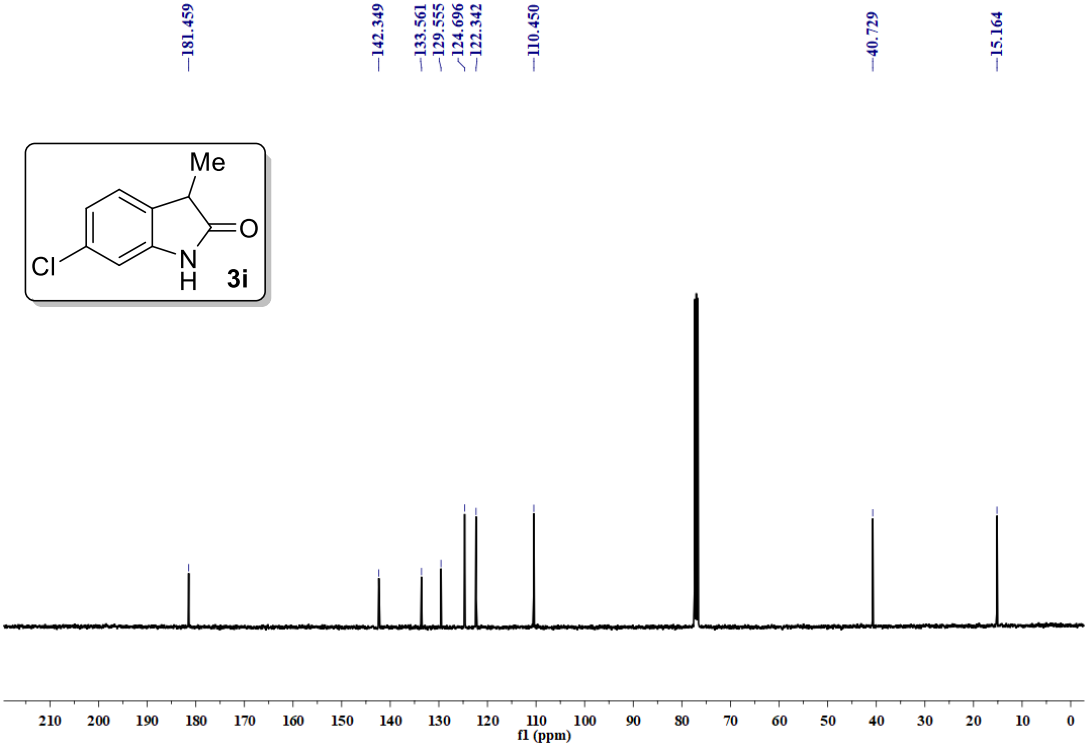
Sample Name	5ioxi-1	**Position**	P1-B9	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-18.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 6:24:41 PM



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 6-chloro-3-methylindolin-2-one (3i)

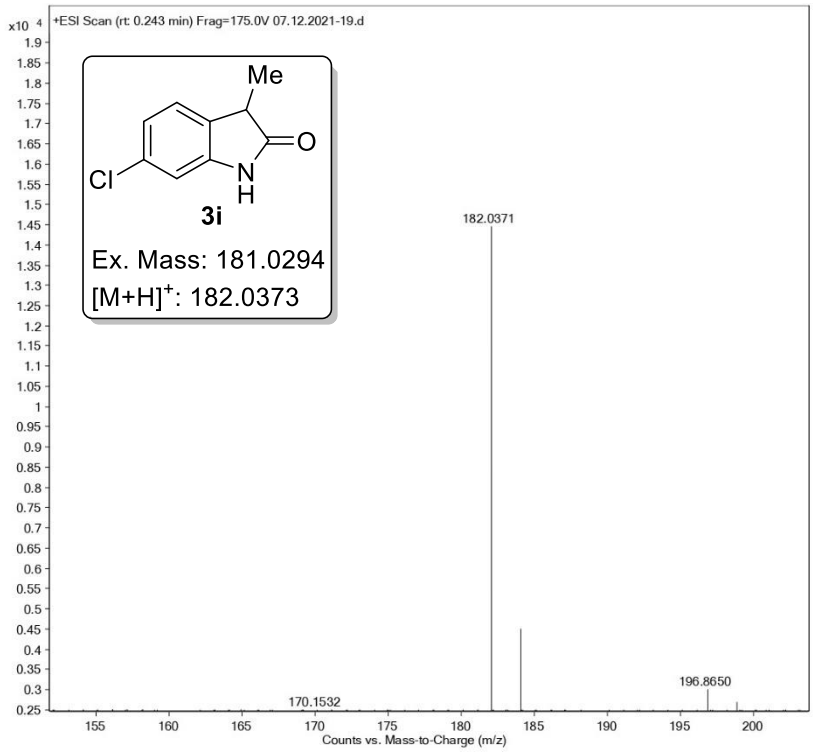


^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of 6-chloro-3-methylindolin-2-one (3i)

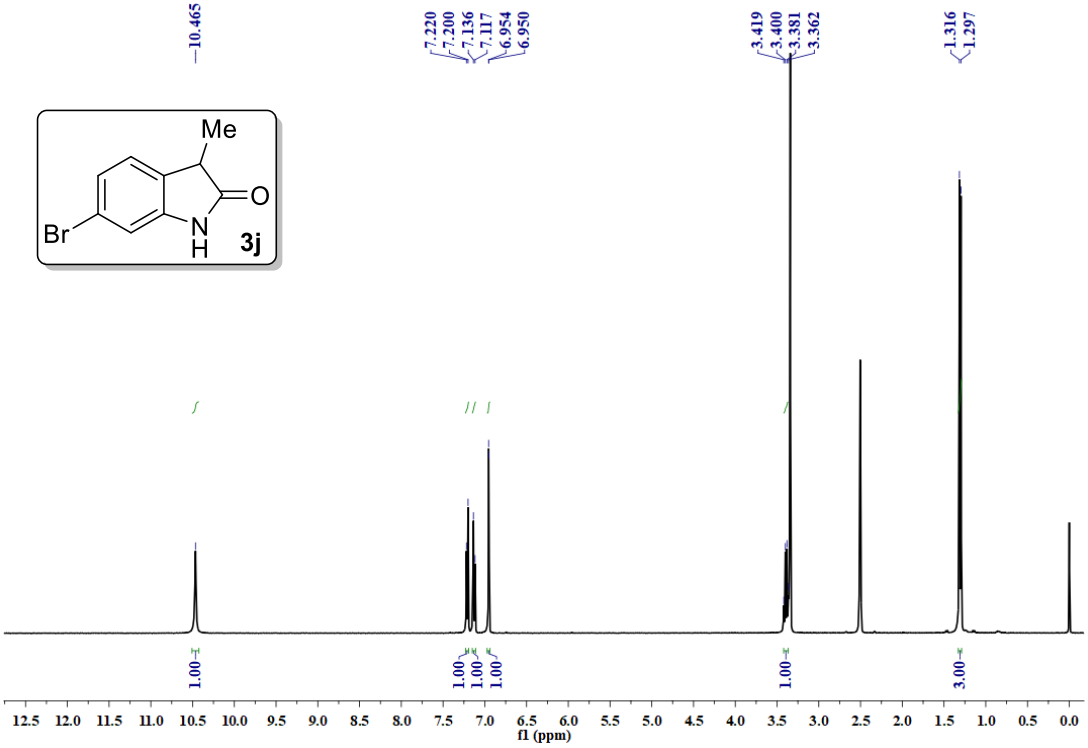


HRMS of 6-chloro-3-methylindolin-2-one (3i)

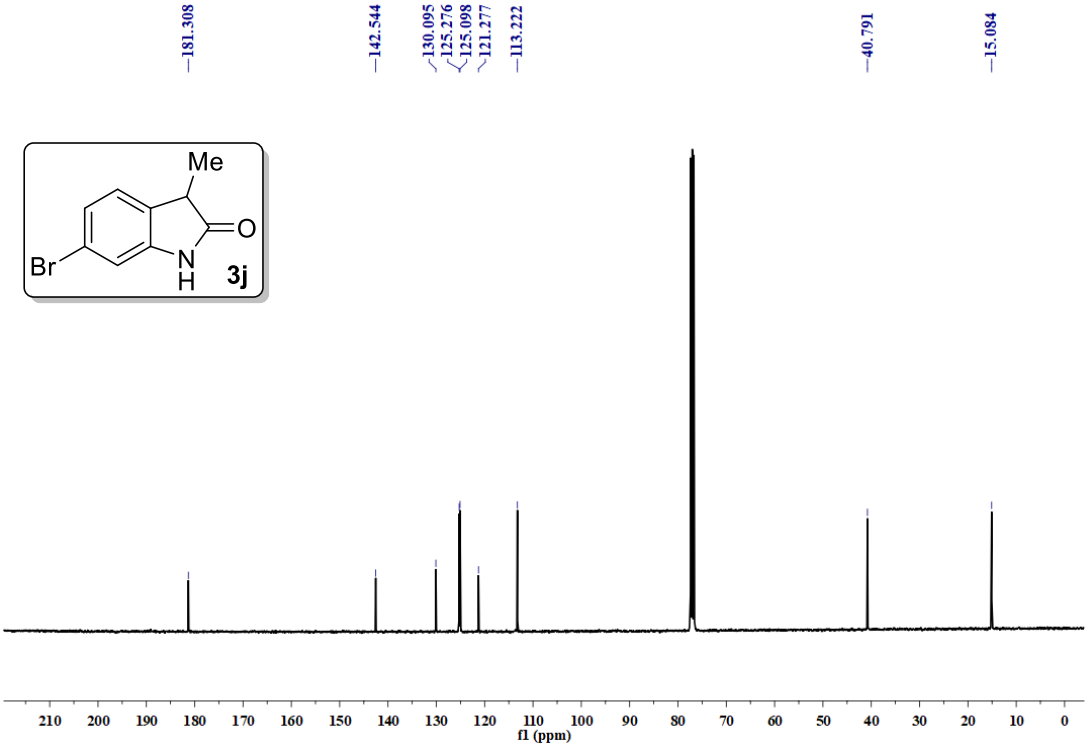
Sample Name	6cloxi-1	**Position**	P1-C1	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-19.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 6:28:40 PM



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 6-bromo-3-methylindolin-2-one (3j)

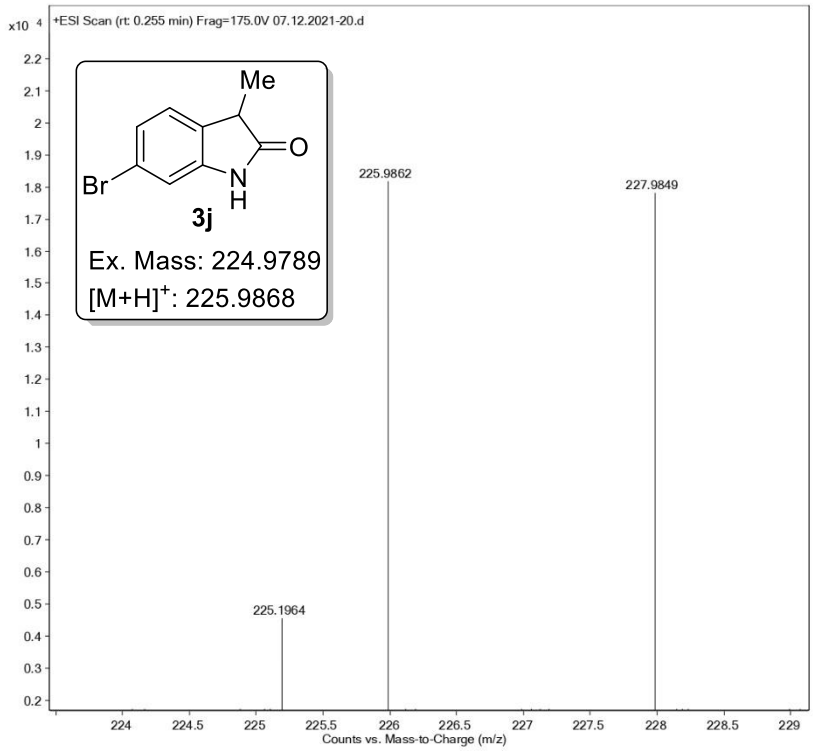


^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of 6-bromo-3-methylindolin-2-one (3j)

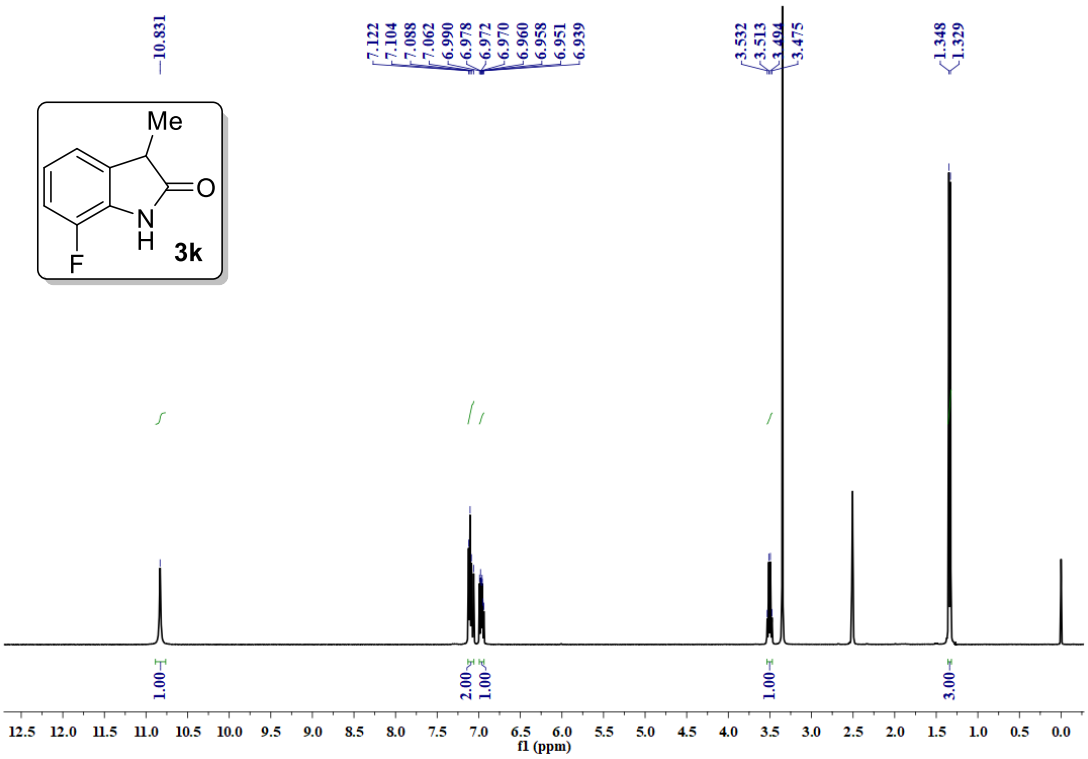


HRMS of 6-bromo-3-methylindolin-2-one (3j)

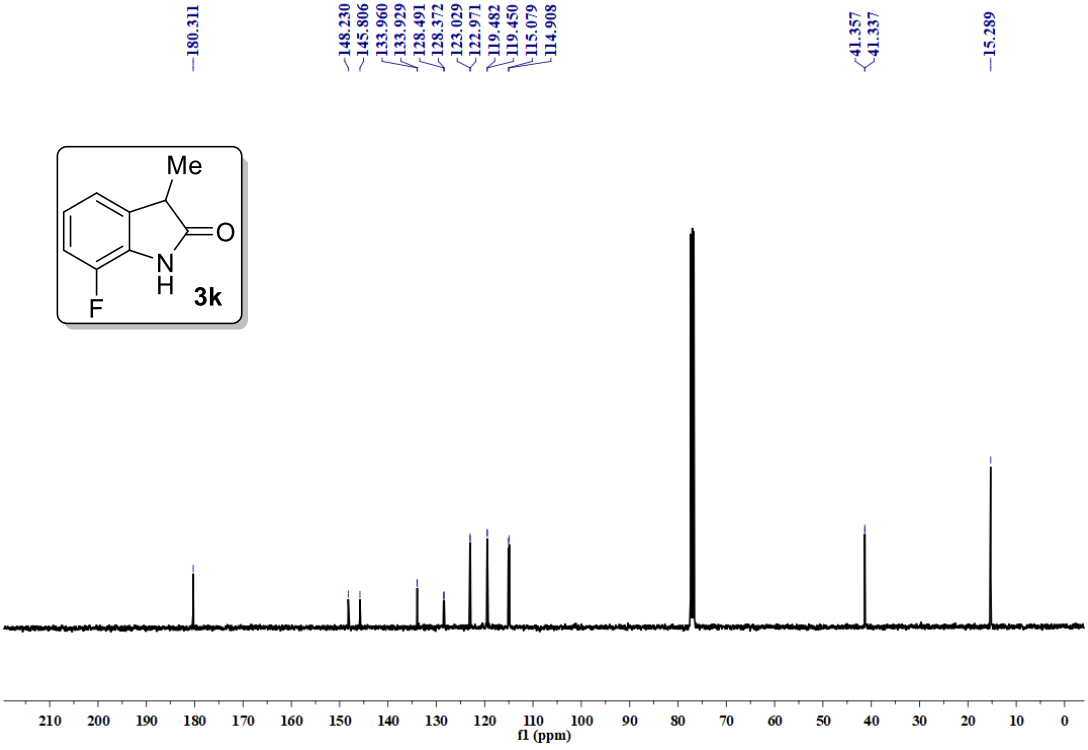
Sample Name	6broxi-1	**Position**	P1-C2	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-20.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 6:32:39 PM



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 7-fluoro-3-methylindolin-2-one (3k)

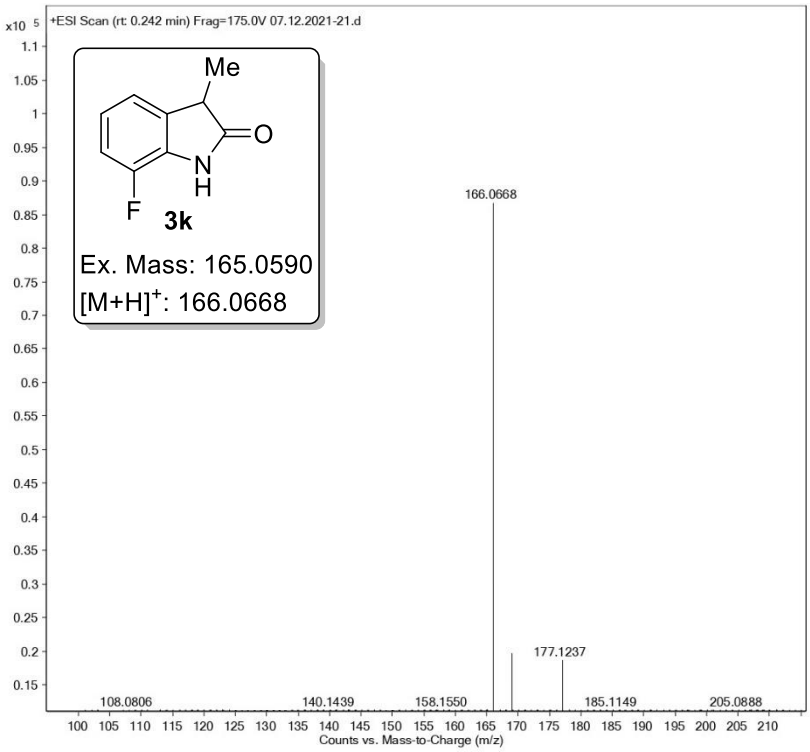


^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of 7-fluoro-3-methylindolin-2-one (3k)

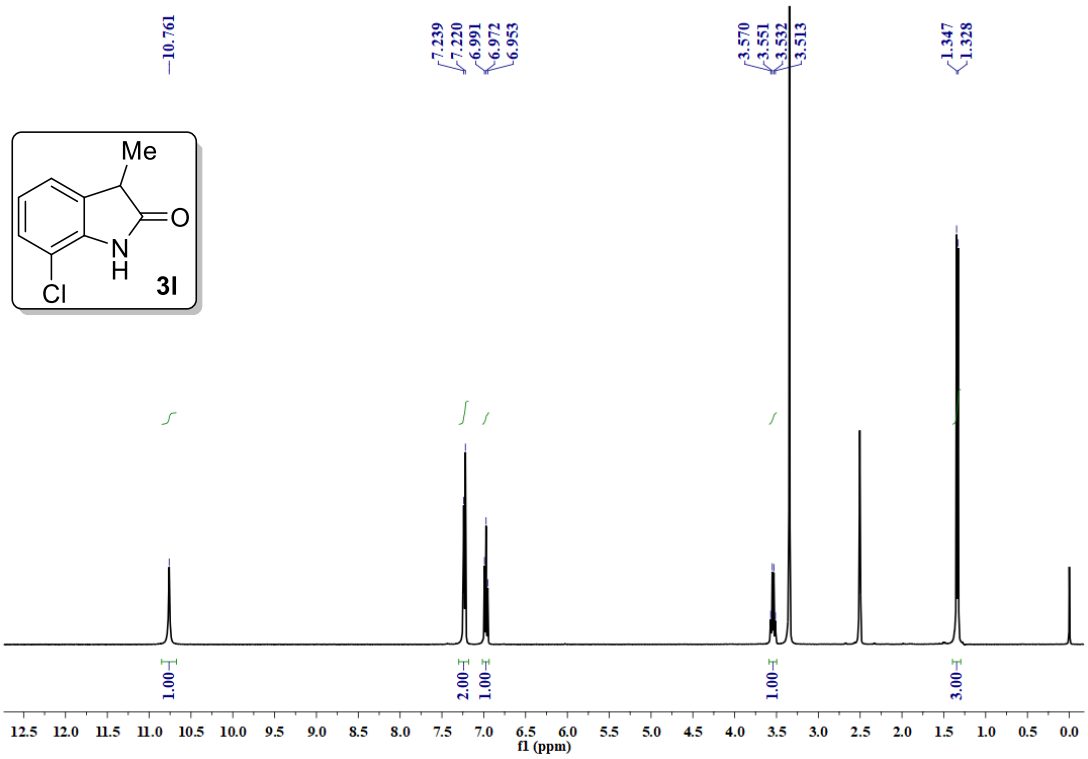


HRMS of 7-fluoro-3-methylindolin-2-one (3k)

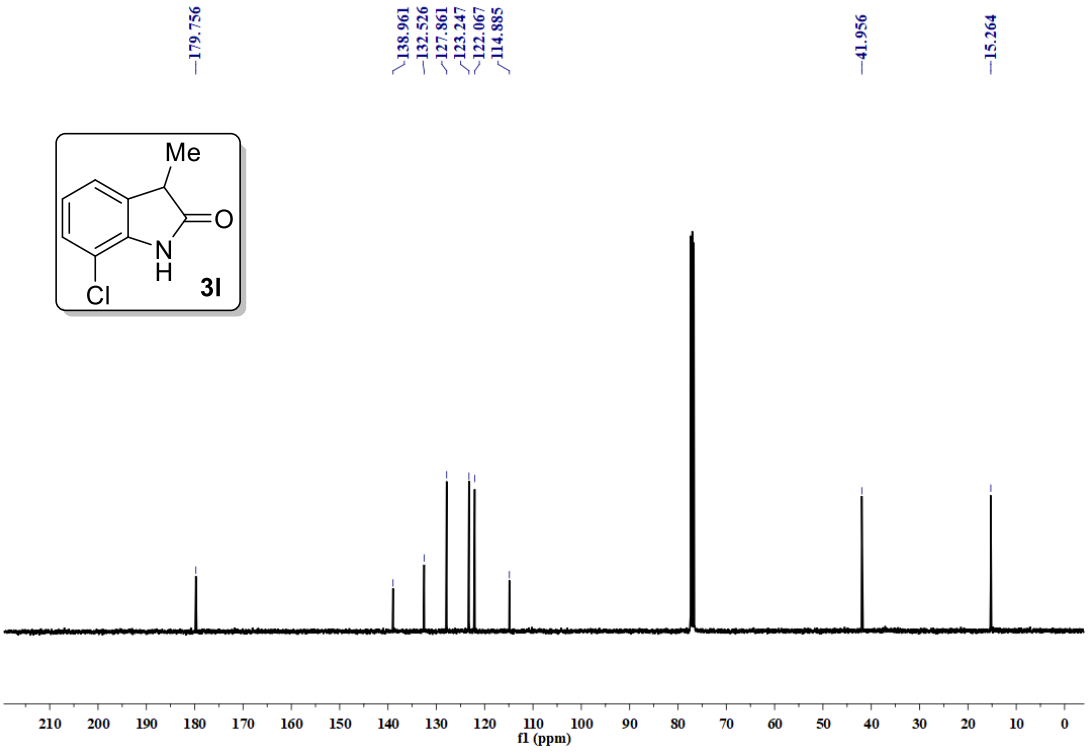
Sample Name	7foxi-1	**Position**	P1-C3	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-21.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 6:36:37 PM



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 7-chloro-3-methylindolin-2-one (3l)

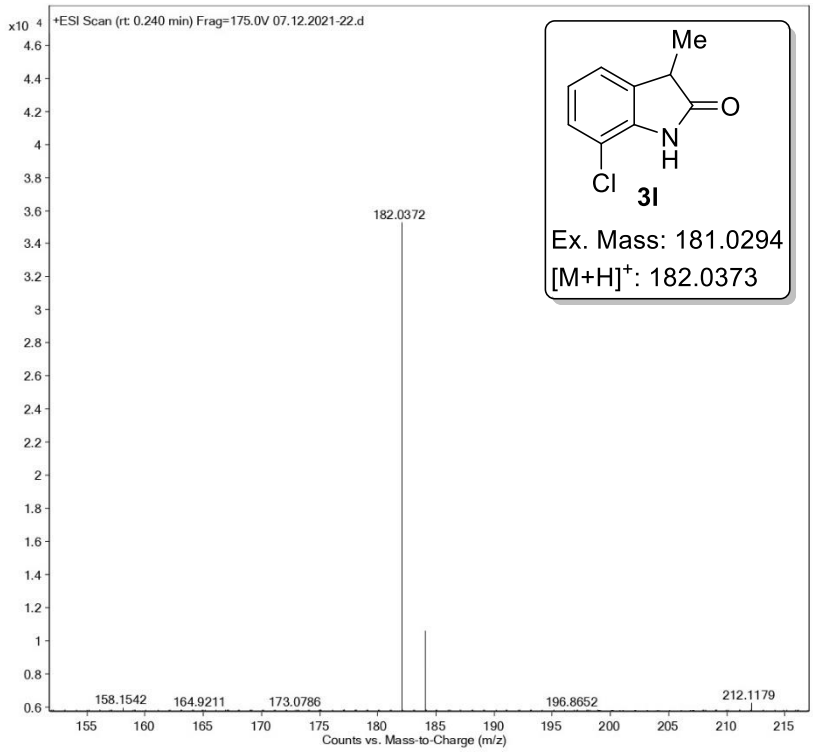


^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of 7-chloro-3-methylindolin-2-one (3l)

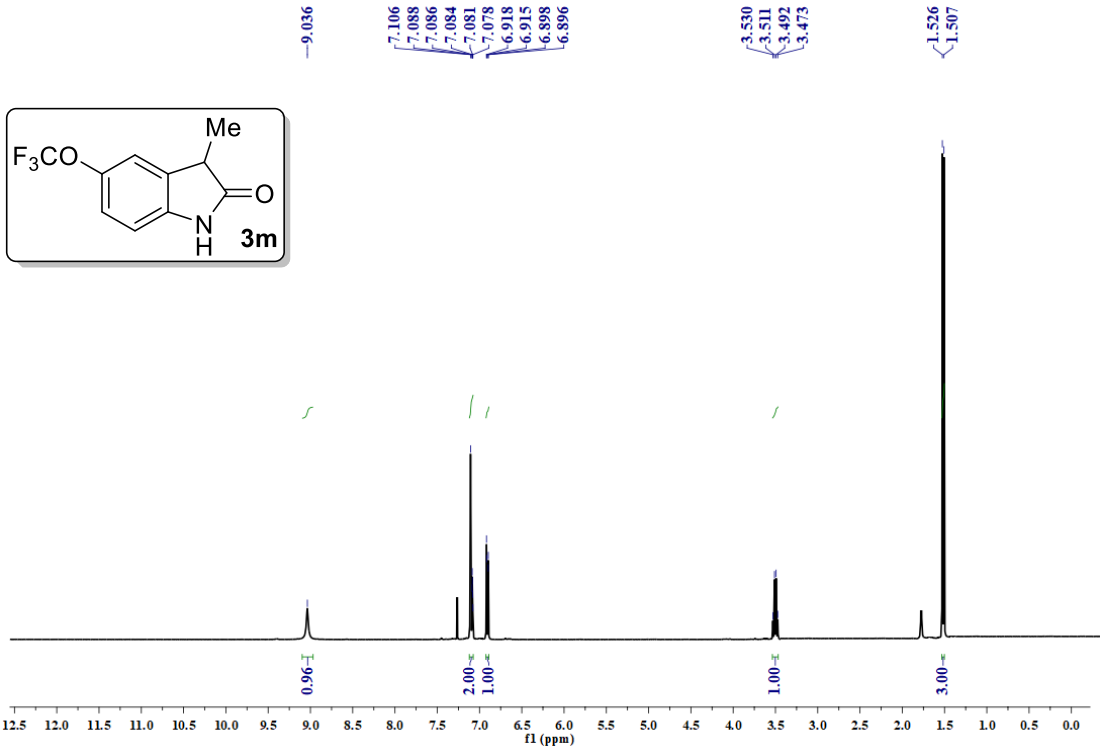


HRMS of 7-chloro-3-methylindolin-2-one (3l)

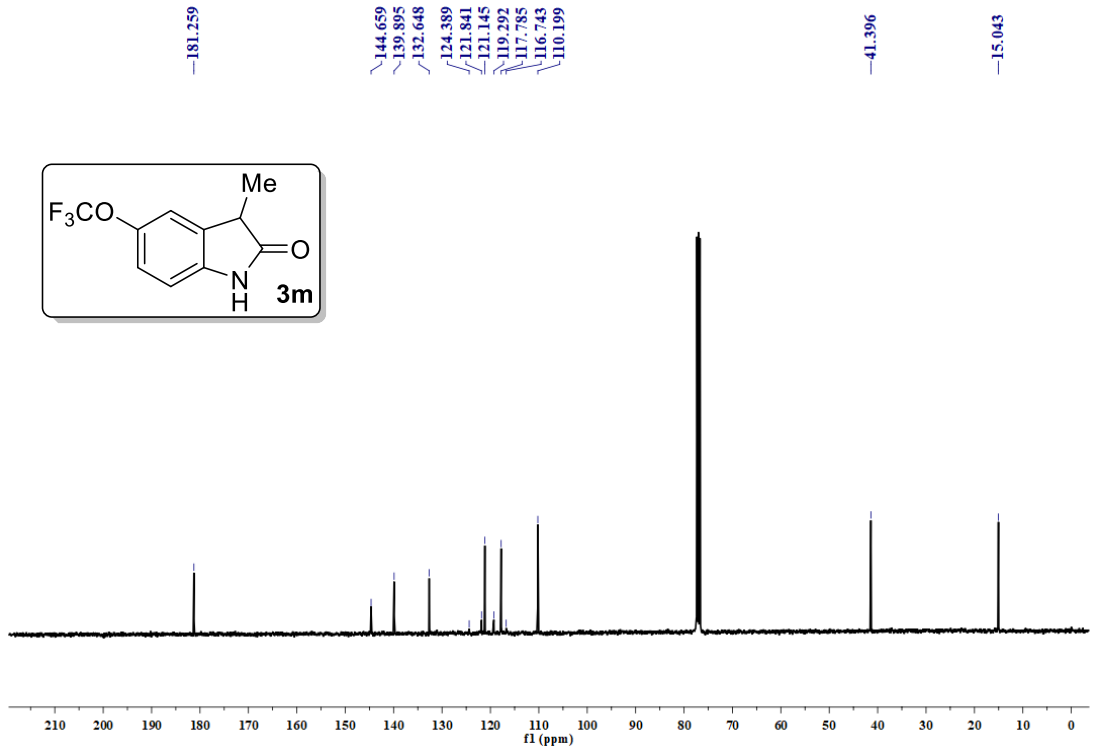
Sample Name	7cloxi-1	**Position**	P1-C4	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-22.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 6:40:36 PM



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3-methyl-5-(trifluoromethoxy)indolin-2-one (3m)

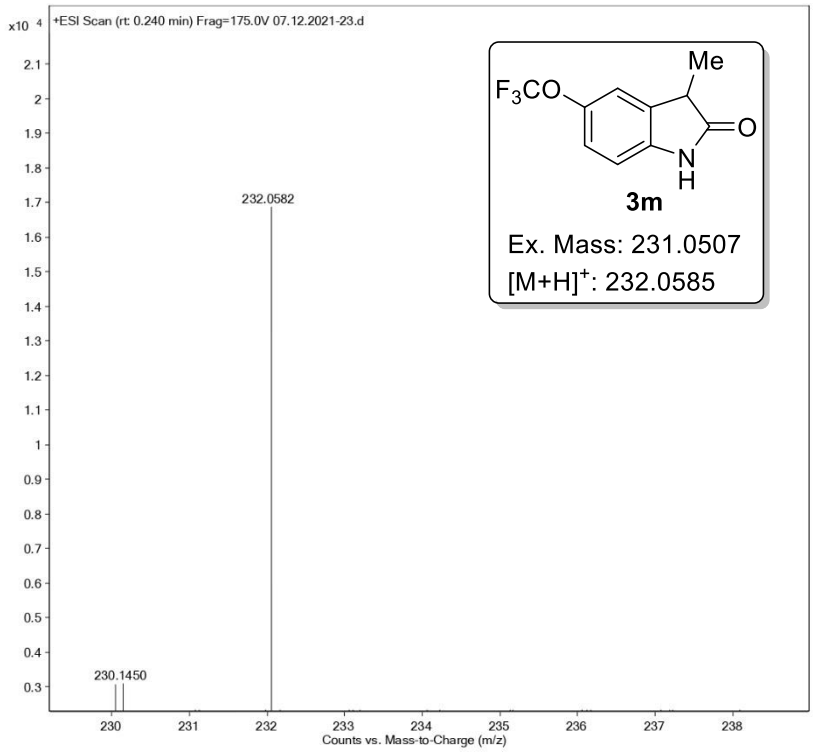


^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of 3-methyl-5-(trifluoromethoxy)indolin-2-one (3m)

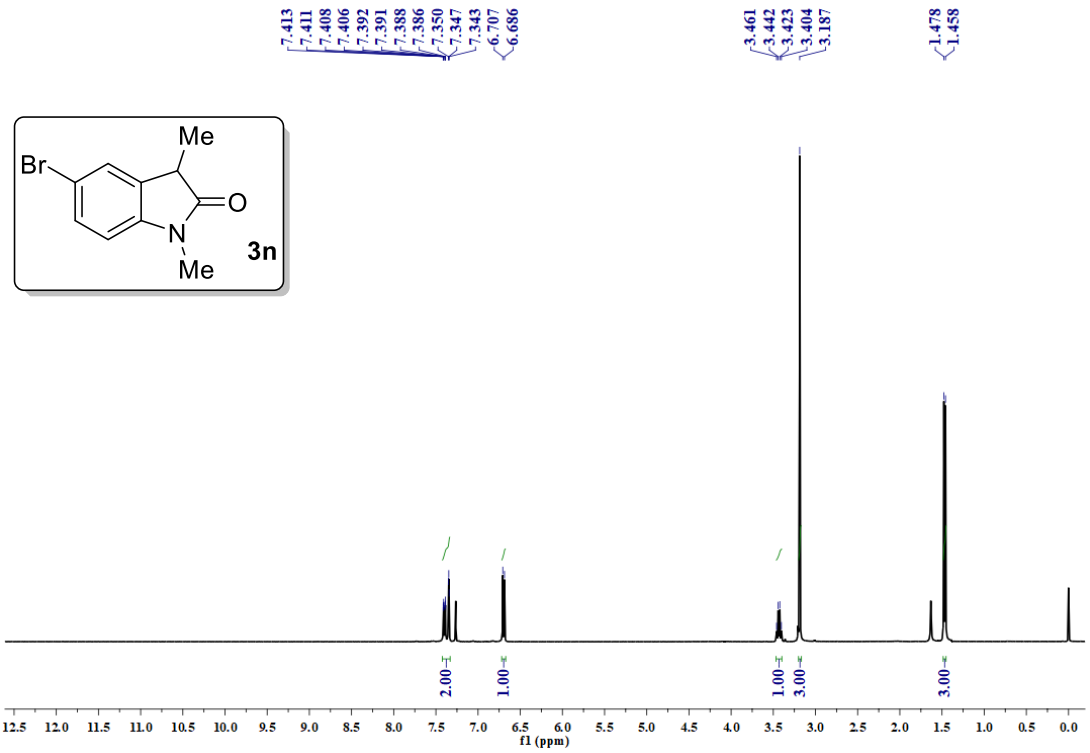


HRMS of 3-methyl-5-(trifluoromethoxy)indolin-2-one (3m)

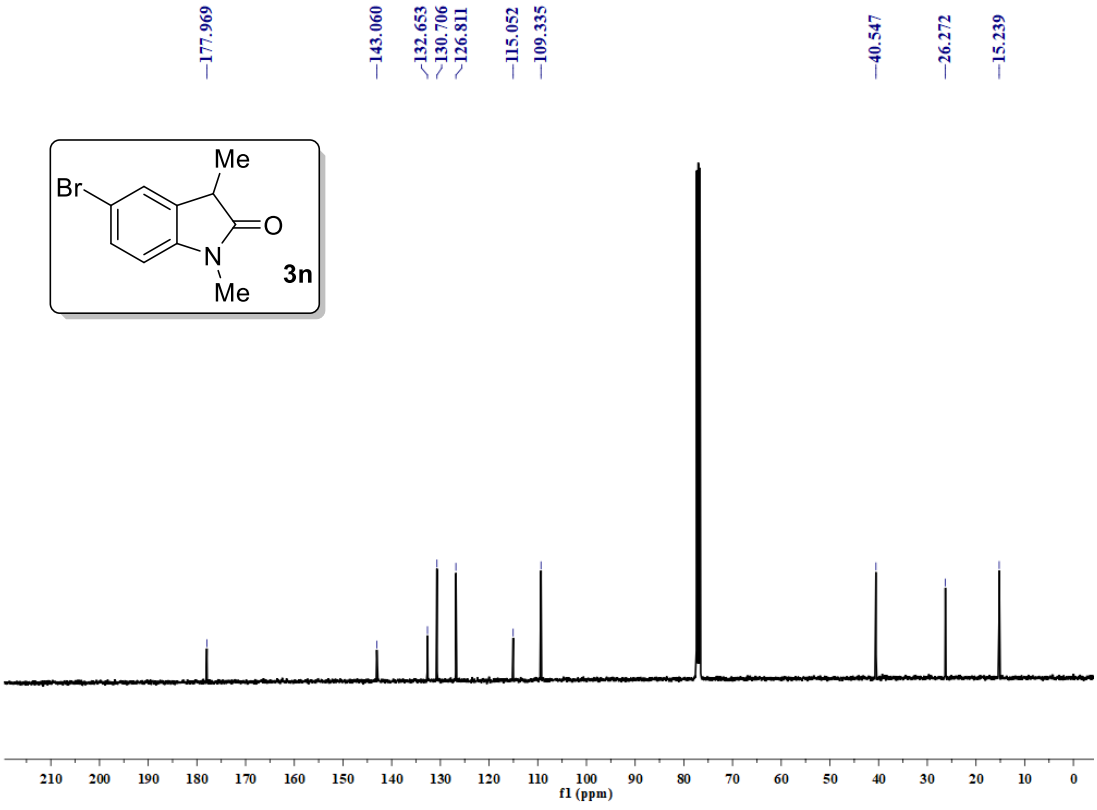
Sample Name	5ocf3oxi-1	**Position**	P1-C5	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-23.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 6:44:34 PM



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 5-bromo-1,3-dimethylindolin-2-one (3n)

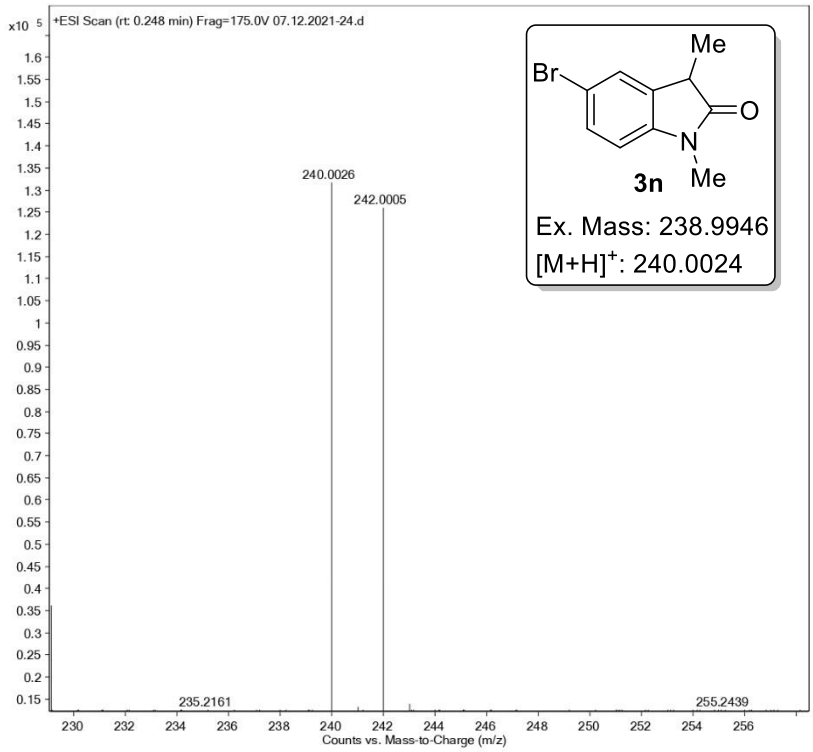


^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of 5-bromo-1,3-dimethylindolin-2-one (3n)

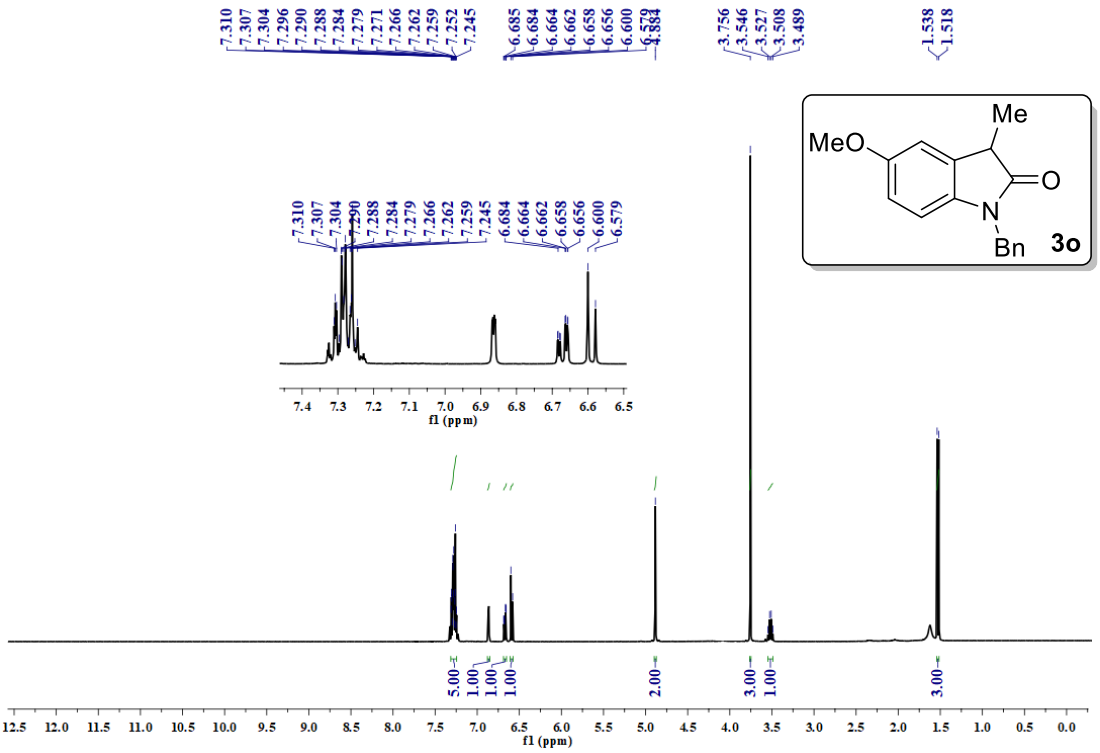


HRMS of 5-bromo-1,3-dimethylindolin-2-one (3n)

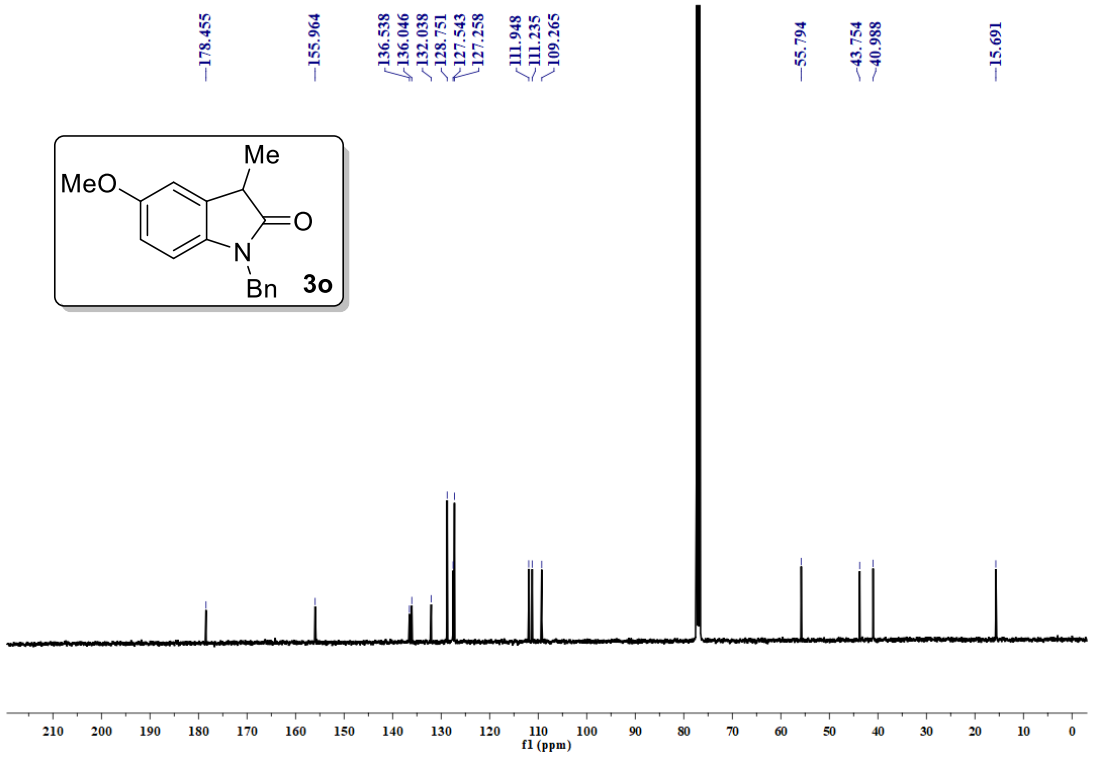
Sample Name	5brnmeoxi-1	**Position**	P1-C6	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-24.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 6:48:34 PM



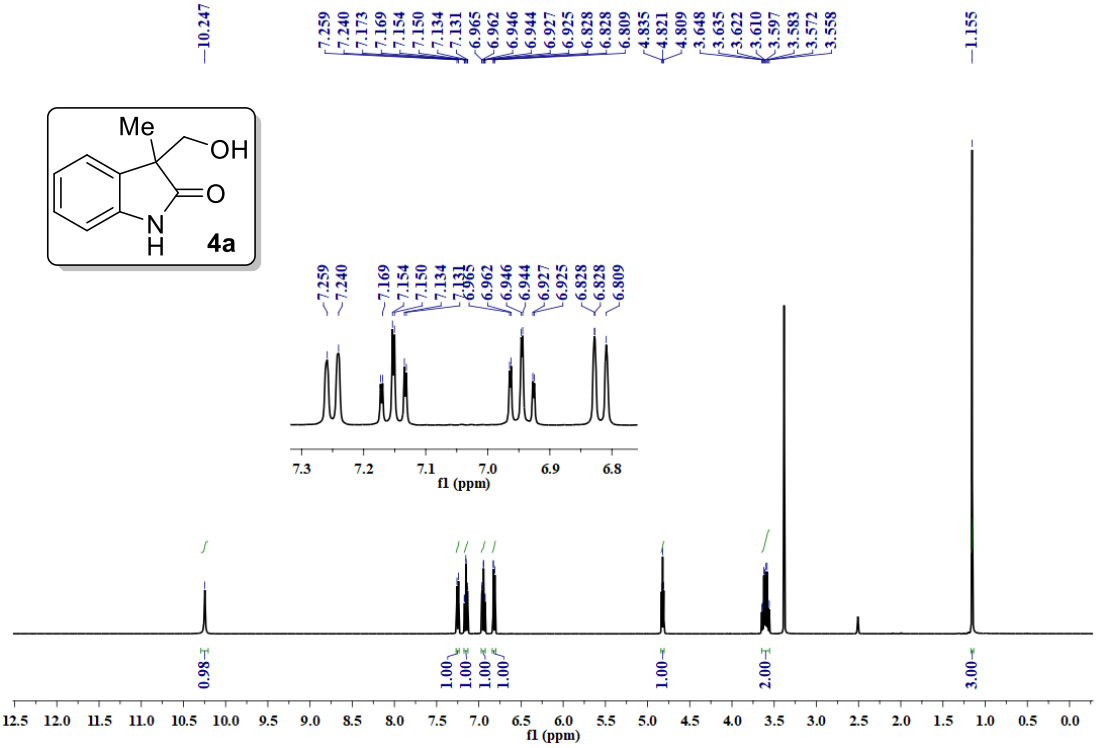
^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 1-benzyl-5-methoxy-3-methylindolin-2-one (3o)



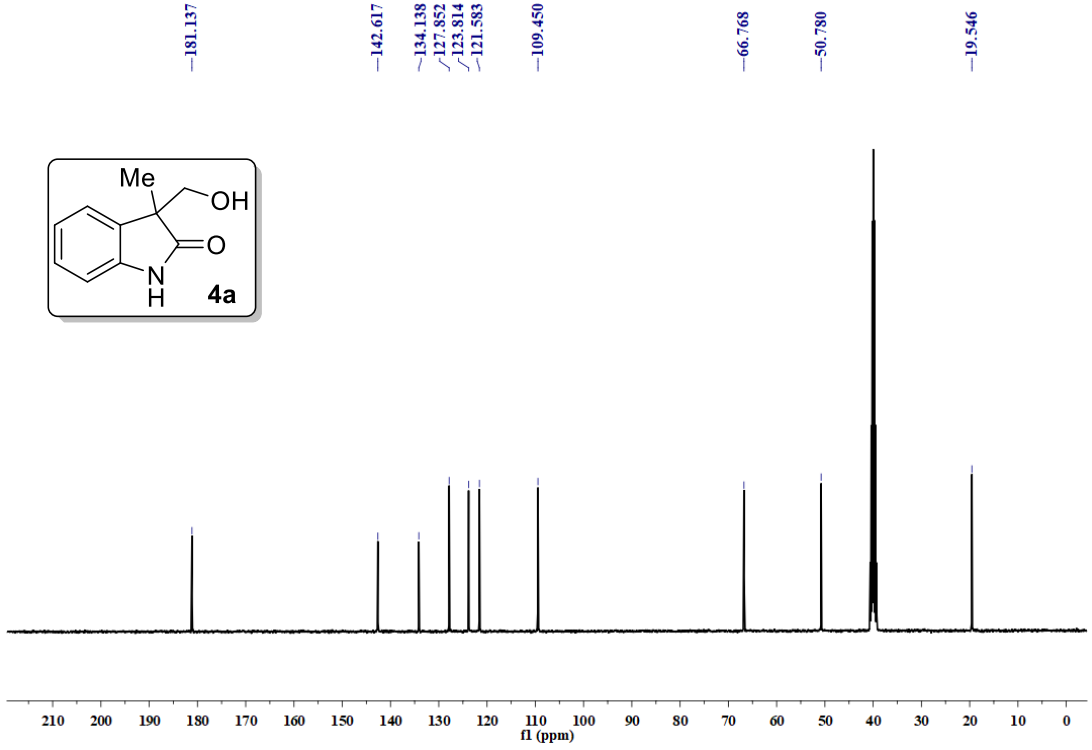
^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of 1-benzyl-5-methoxy-3-methylindolin-2-one (3o)



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 3-(hydroxymethyl)-3-methylindolin-2-one (4a)

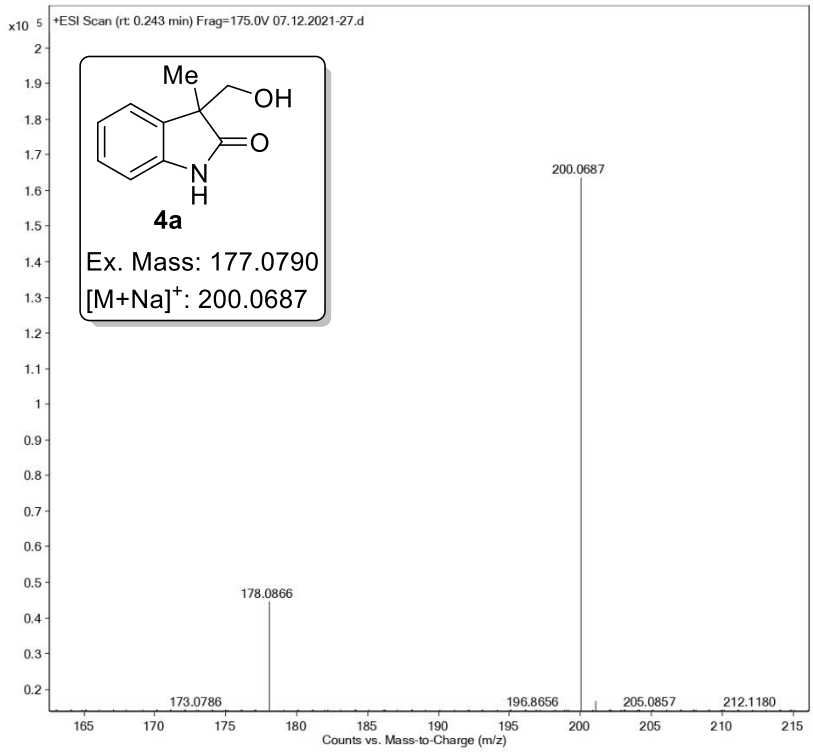


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 3-(hydroxymethyl)-3-methylindolin-2-one (4a)

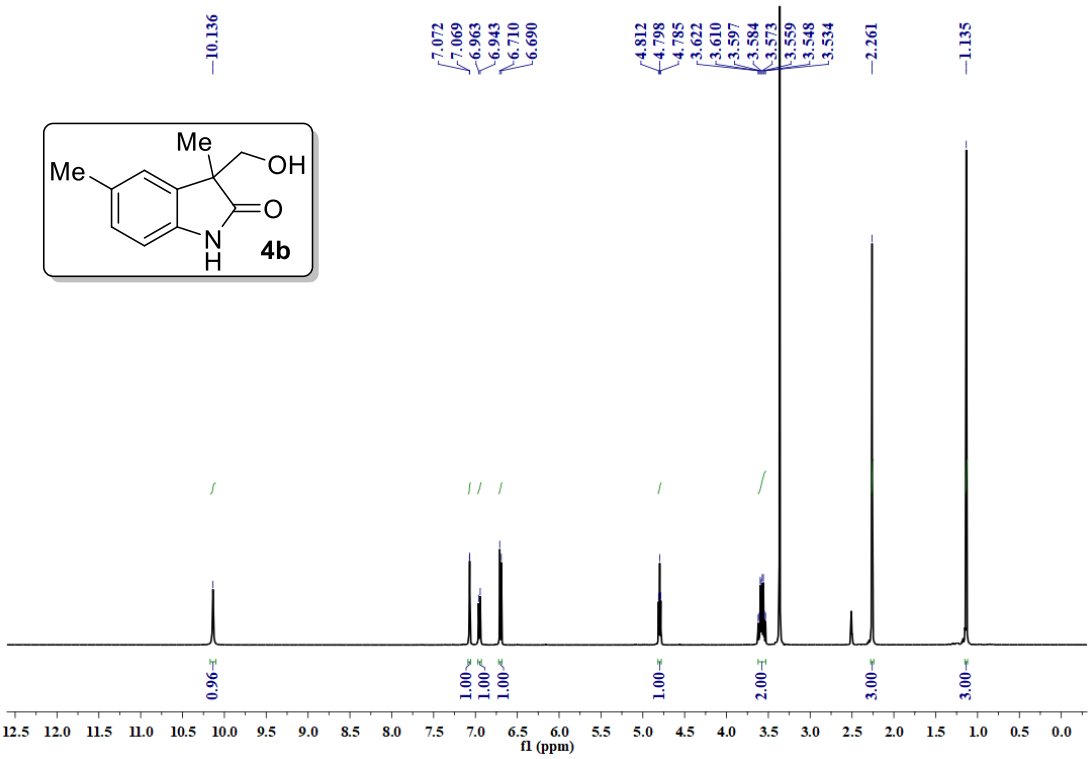


HRMS of 3-(hydroxymethyl)-3-methylindolin-2-one (4a)

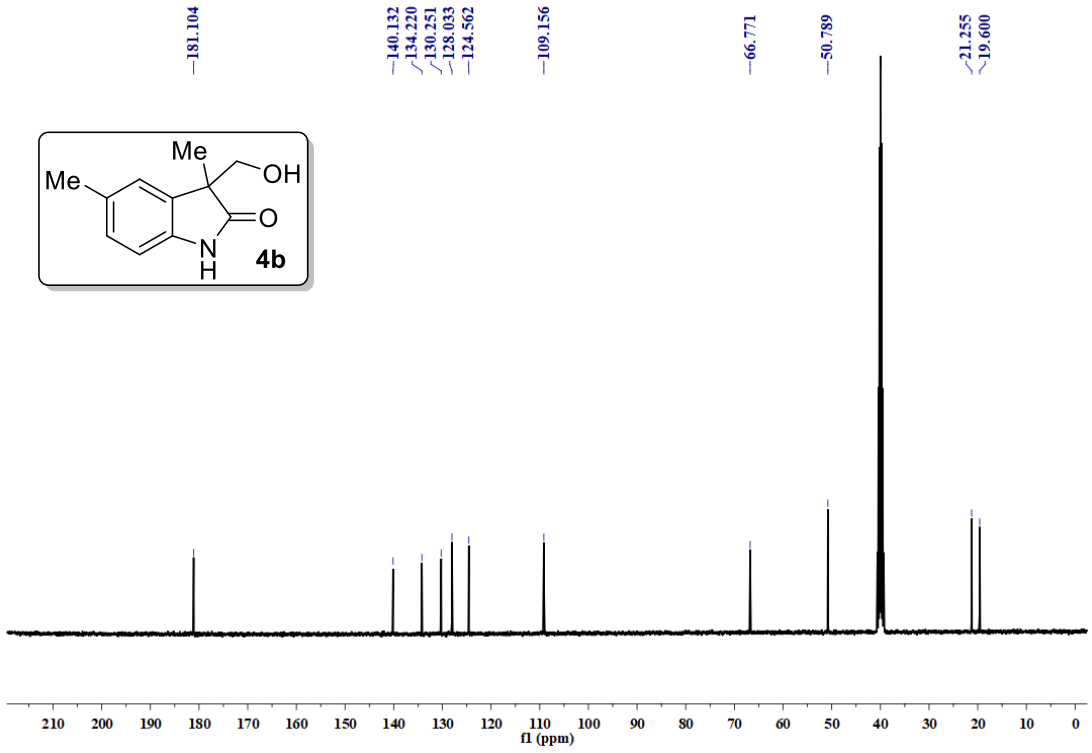
Sample Name	oxis-2	**Position**	P1-C9	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-27.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 7:00:30 PM



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 3-(hydroxymethyl)-3,5-dimethylindolin-2-one (4b)

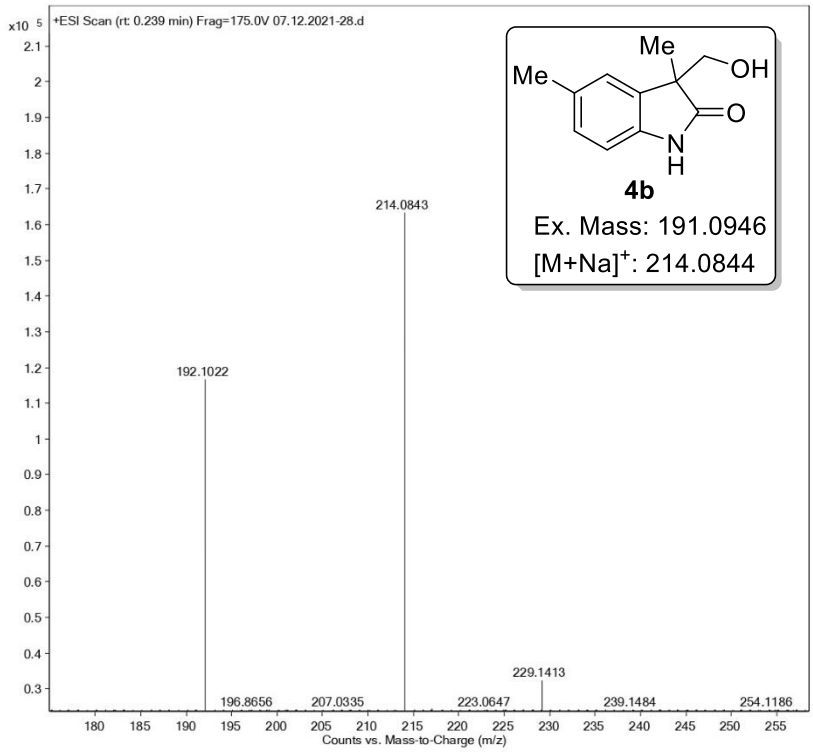


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 3-(hydroxymethyl)-3,5-dimethylindolin-2-one (4b)

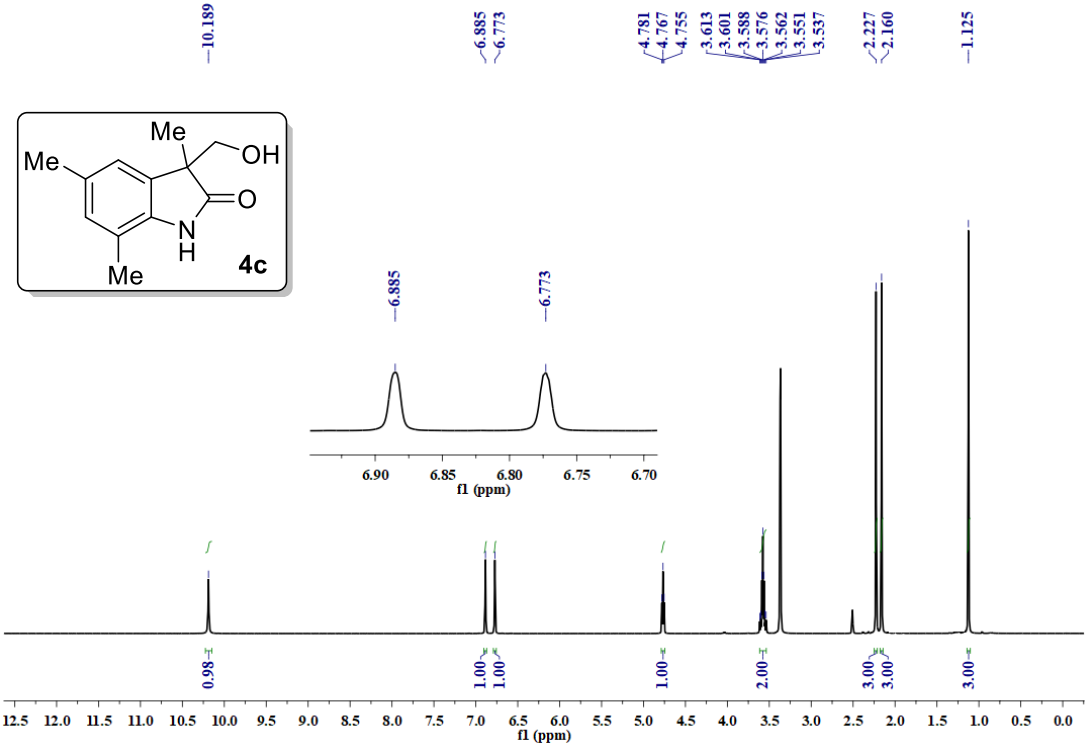


HRMS of 3-(hydroxymethyl)-3,5-dimethylindolin-2-one (4b)

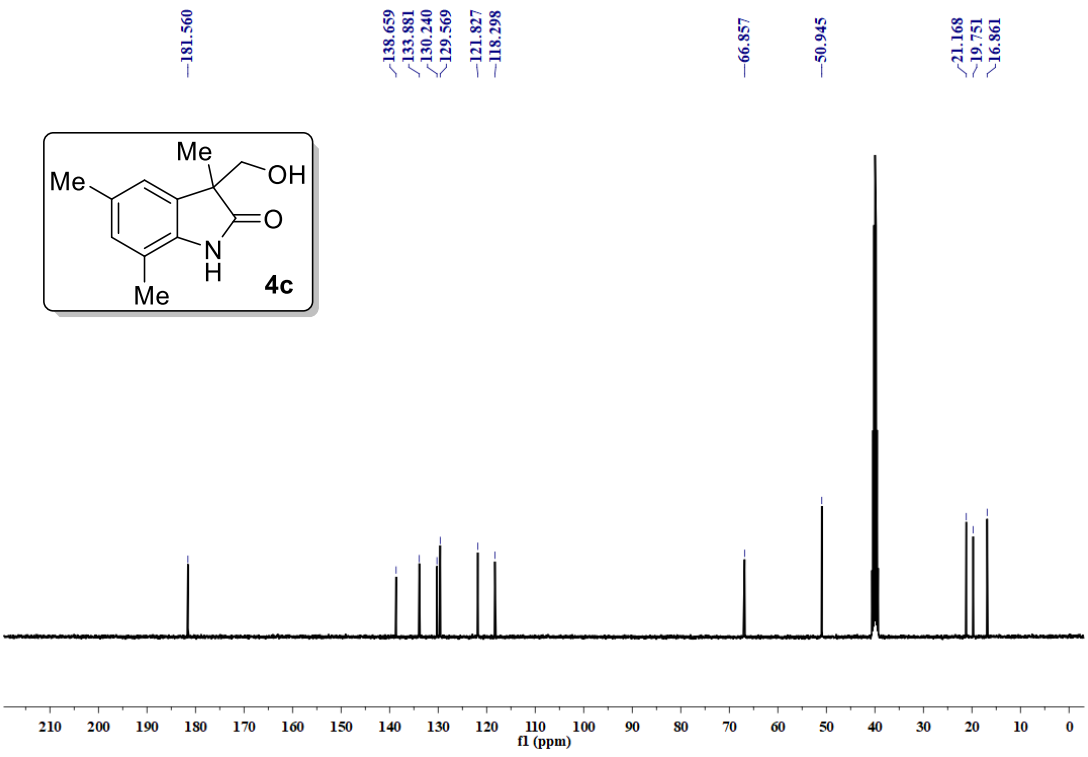
Sample Name	5-meoxi-2	**Position**	P1-D1	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-28.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 7:04:26 PM



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 3-(hydroxymethyl)-3,5,7-trimethylindolin-2-one (4c)

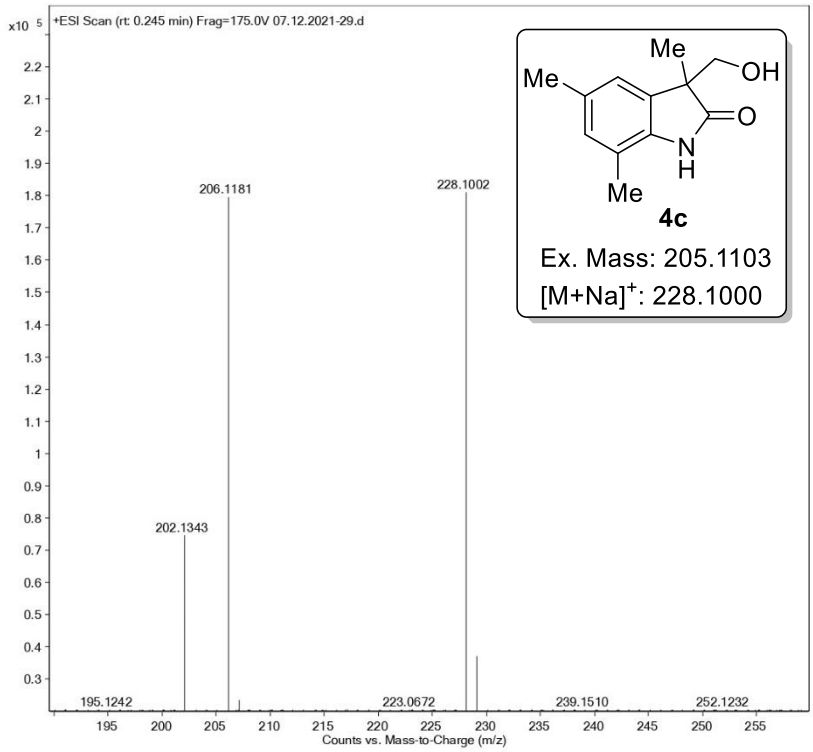


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 3-(hydroxymethyl)-3,5,7-trimethylindolin-2-one (4c)

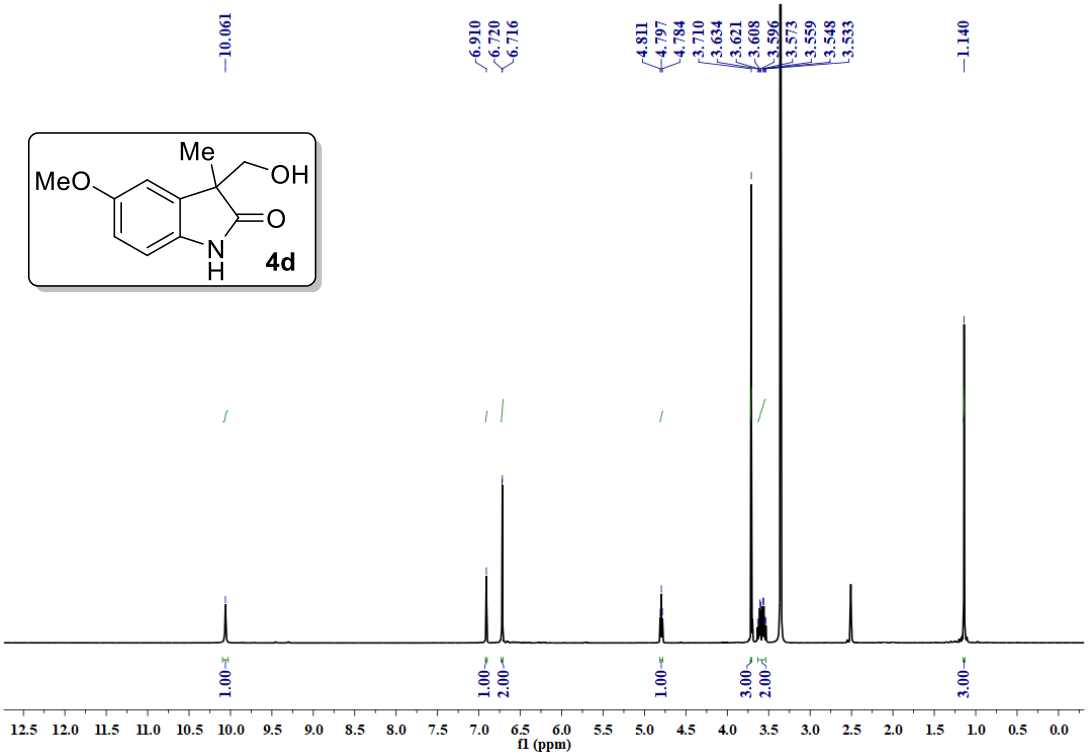


HRMS of 3-(hydroxymethyl)-3,5,7-trimethylindolin-2-one (4c)

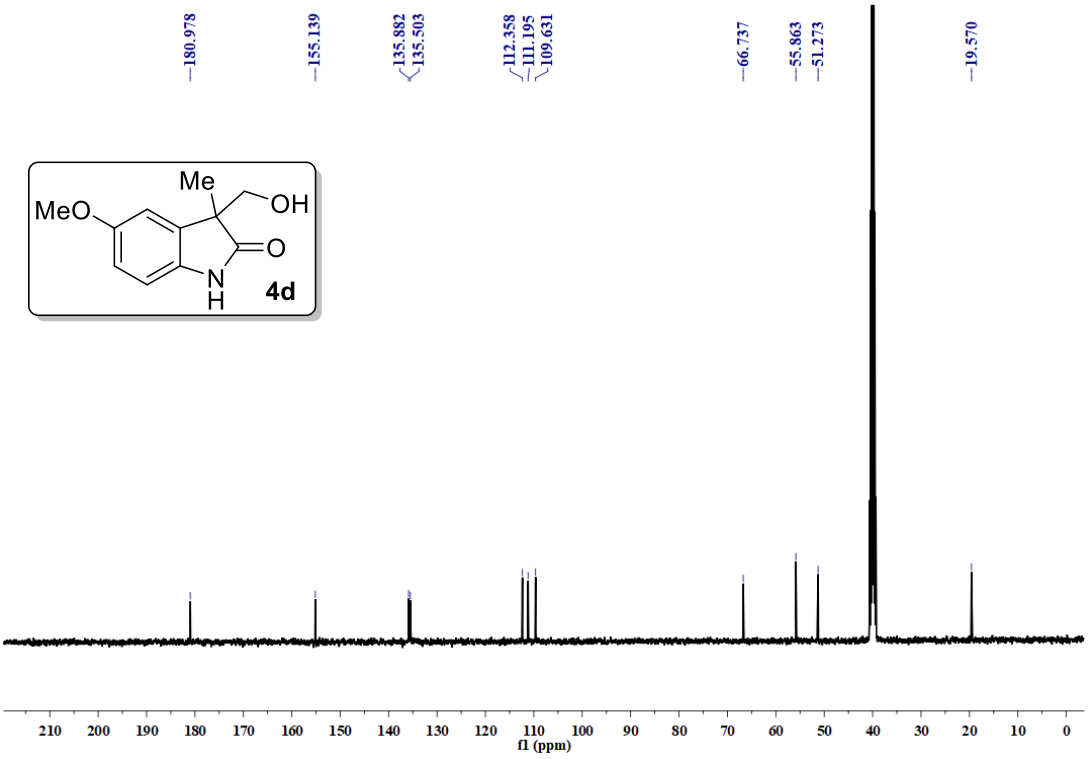
Sample Name	5 7 meoxi-2	**Position**	P1-D2	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-29.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 7:08:26 PM



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 3-(hydroxymethyl)-5-methoxy-3-methylindolin-2-one (4d)

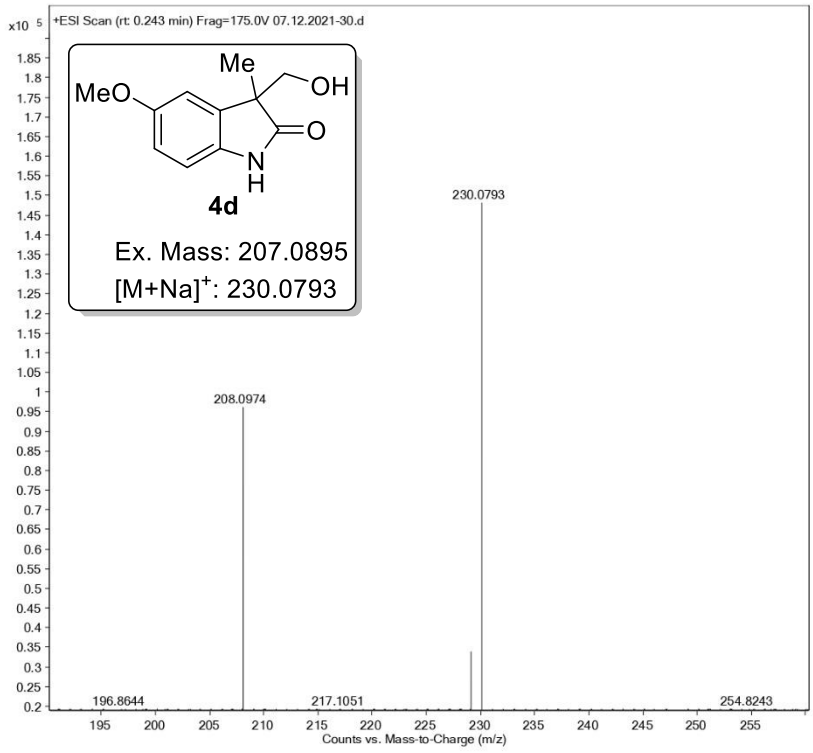


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 3-(hydroxymethyl)-5-methoxy-3-methylindolin-2-one (4d)

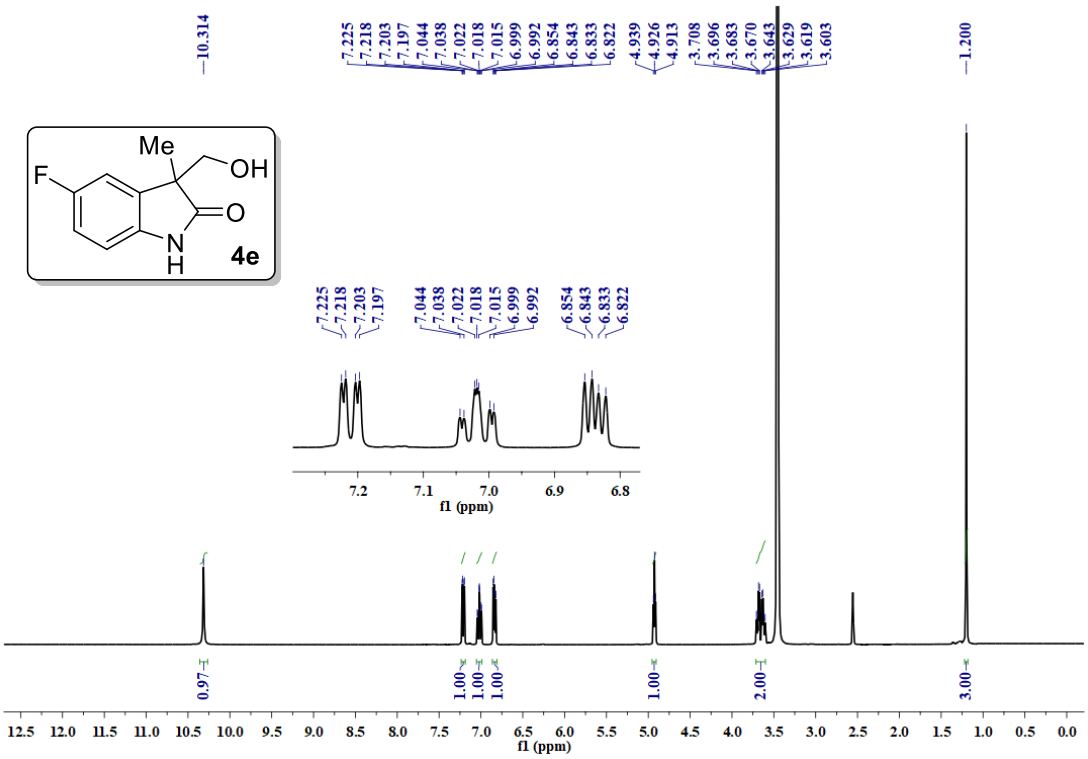


HRMS of 3-(hydroxymethyl)-5-methoxy-3-methylindolin-2-one (4d)

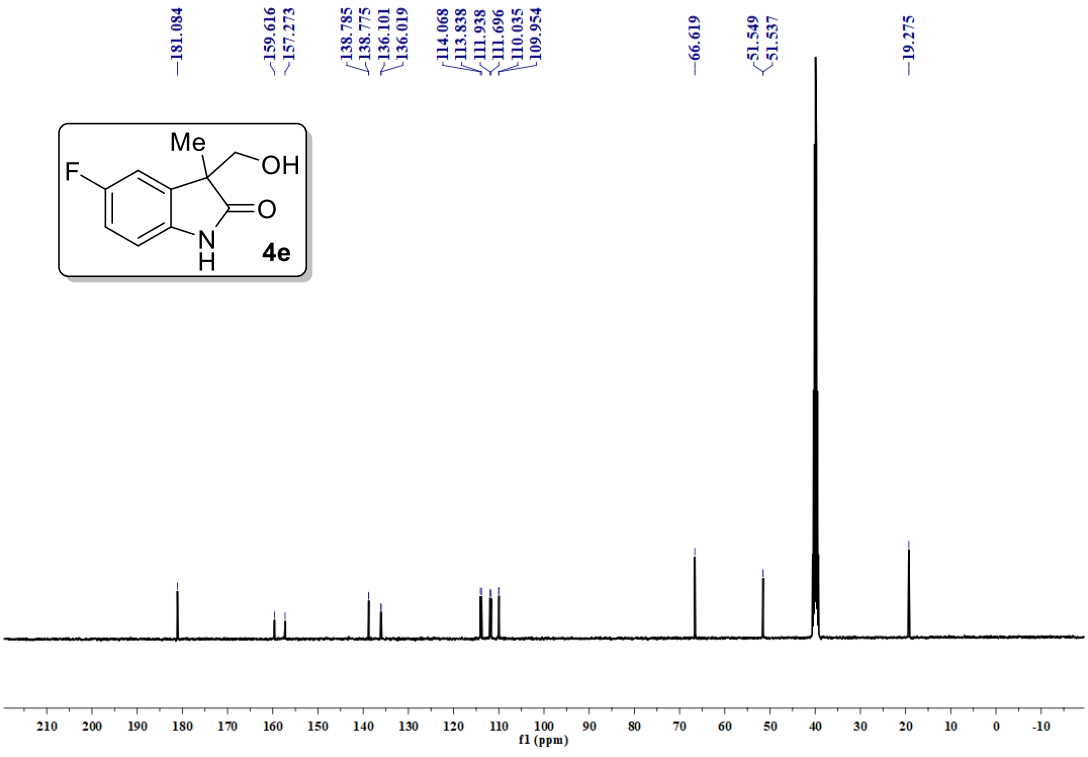
Sample Name	5 omeoxi-2	**Position**	P1-D3	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-30.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 7:12:24 PM



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 5-fluoro-3-(hydroxymethyl)-3-methylindolin-2-one (4e)

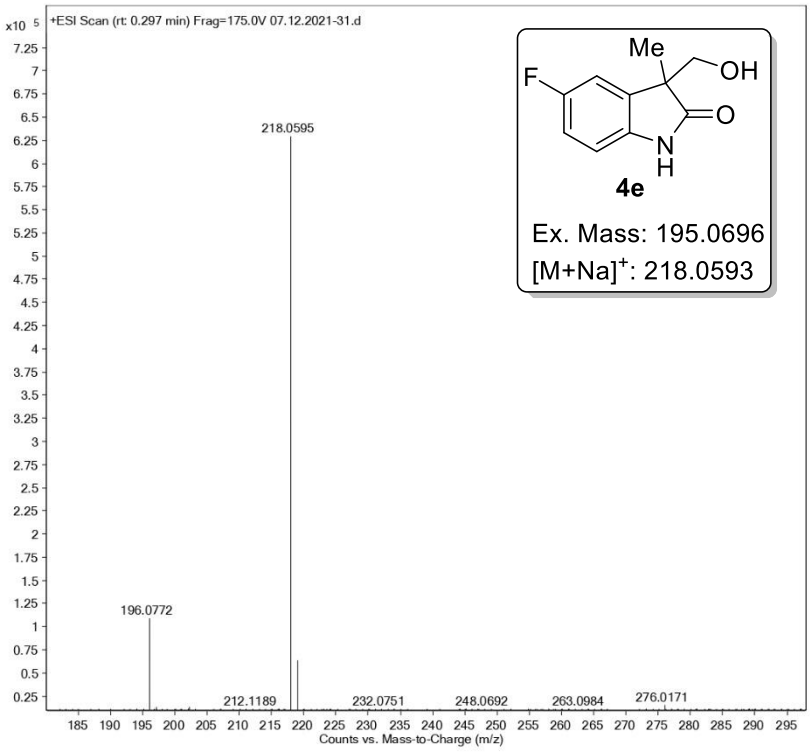


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 5-fluoro-3-(hydroxymethyl)-3-methylindolin-2-one (4e)

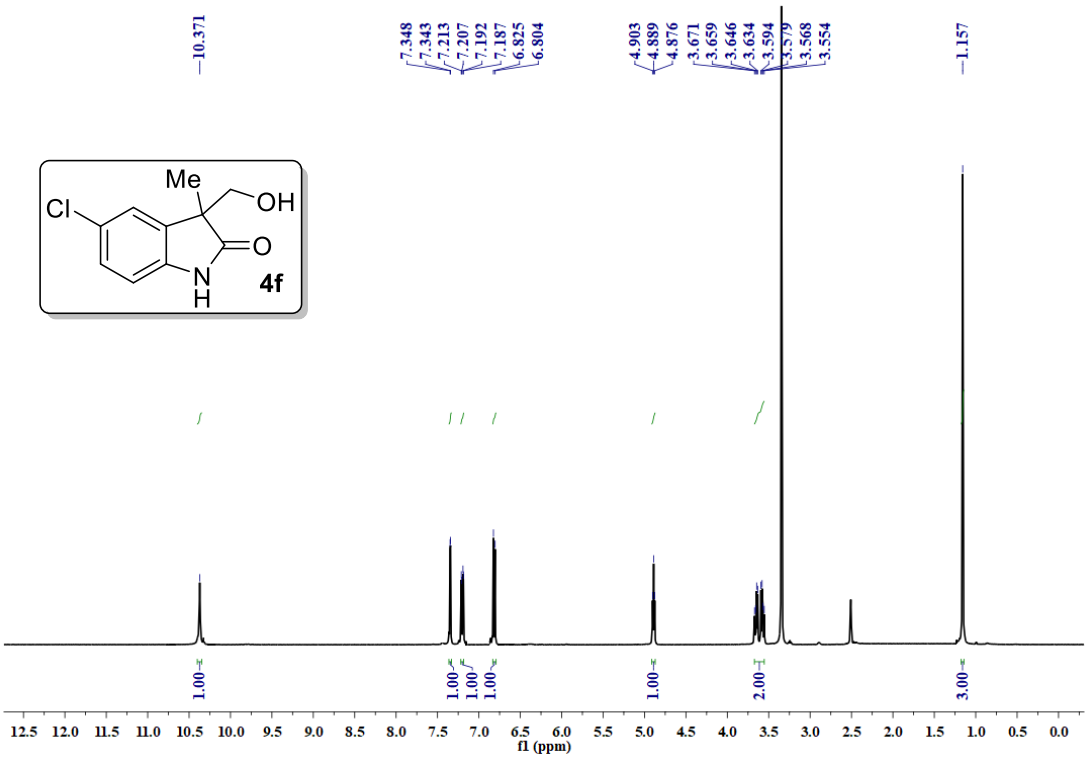


HRMS of 5-fluoro-3-(hydroxymethyl)-3-methylindolin-2-one (4e)

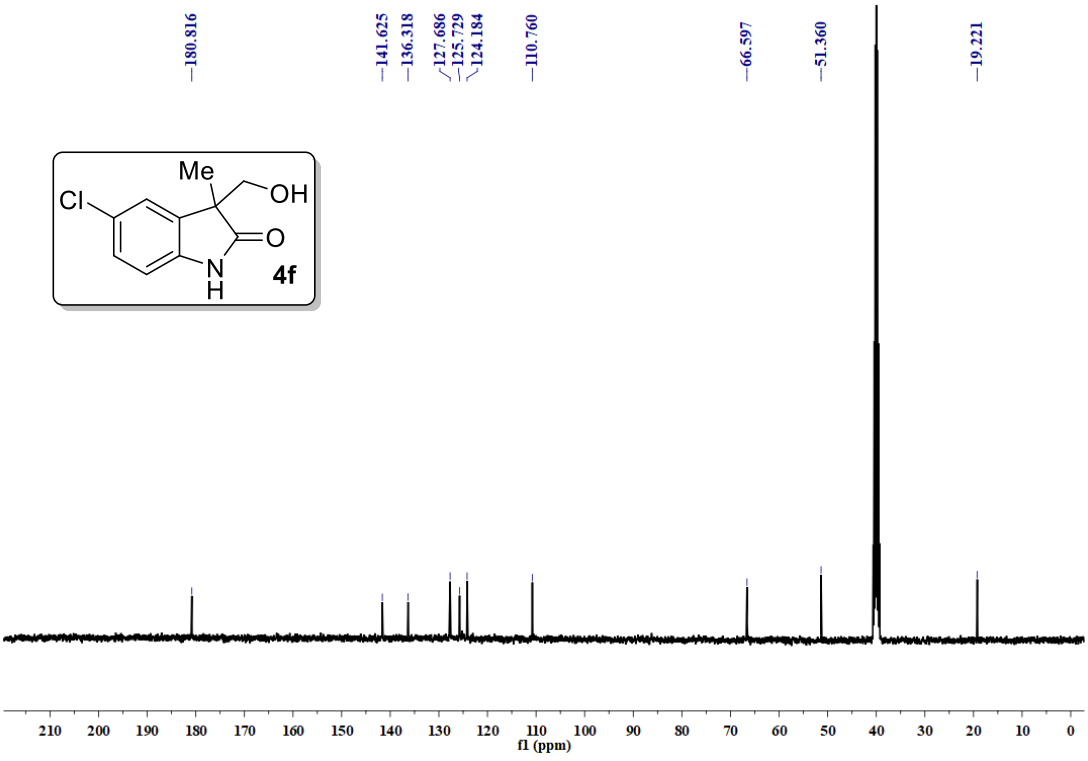
Sample Name	5-foxi-2	**Position**	P1-D4	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-31.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 7:16:22 PM



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 5-chloro-3-(hydroxymethyl)-3-methylindolin-2-one (4f)

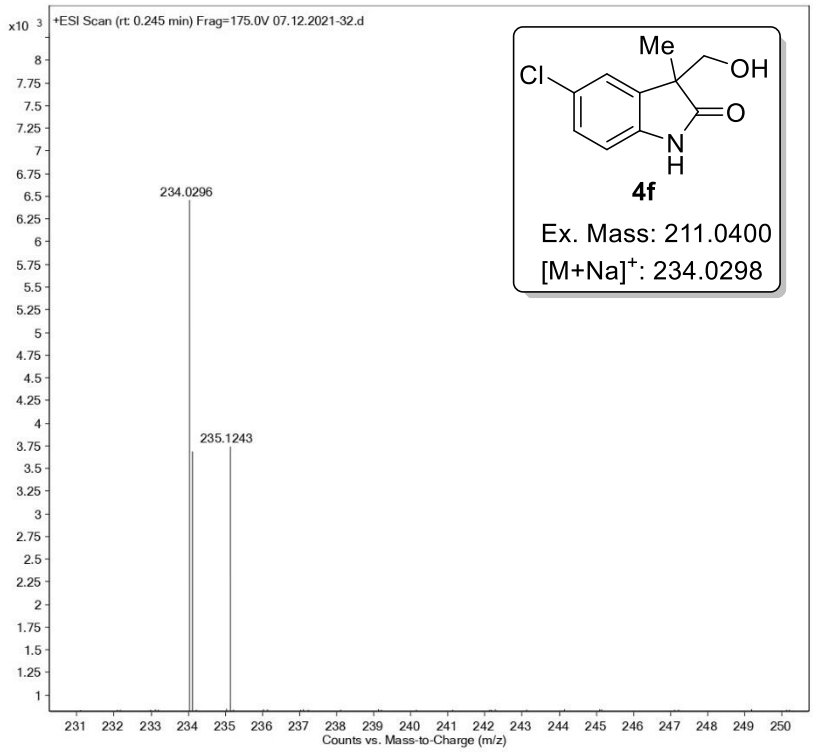


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 5-chloro-3-(hydroxymethyl)-3-methylindolin-2-one (4f)

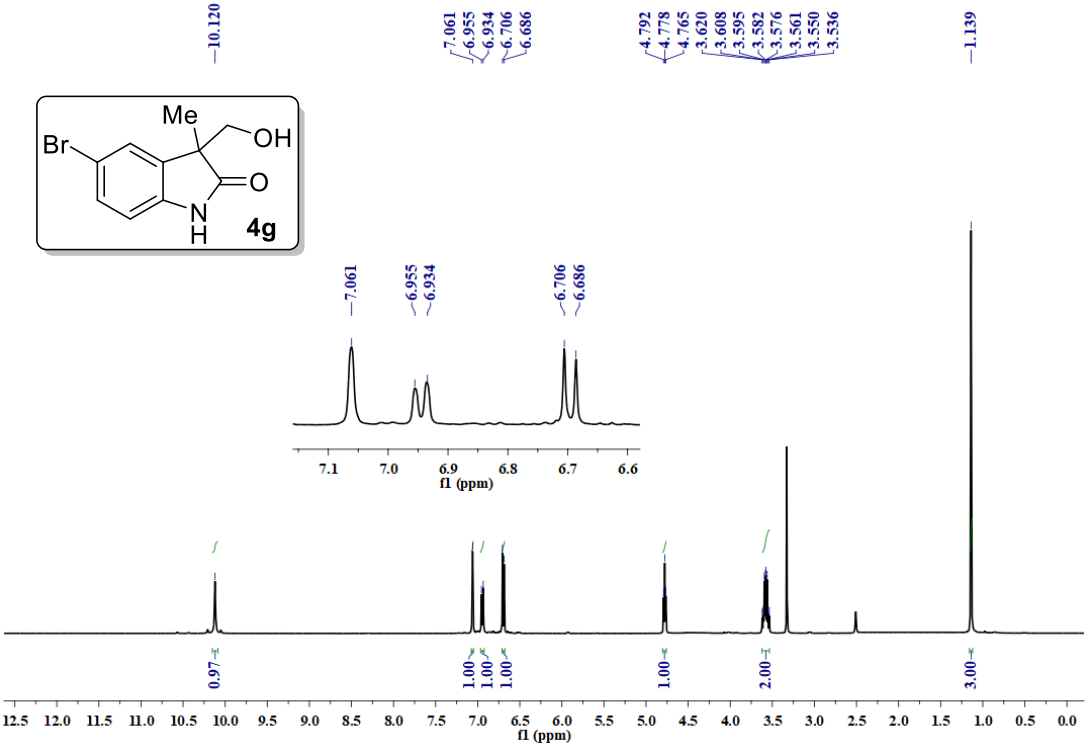


HRMS of 5-chloro-3-(hydroxymethyl)-3-methylindolin-2-one (4f)

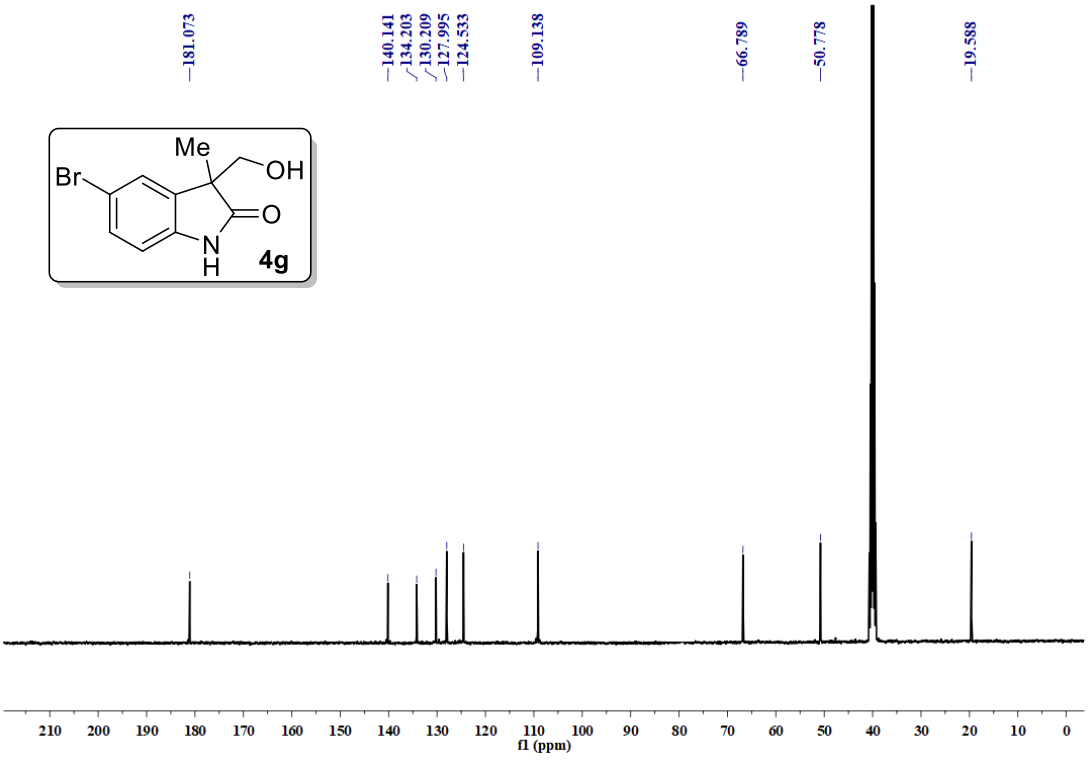
Sample Name	5-cloxi-2	**Position**	P1-D5	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-32.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 7:20:21 PM



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 5-bromo-3-(hydroxymethyl)-3-methylindolin-2-one (4g)

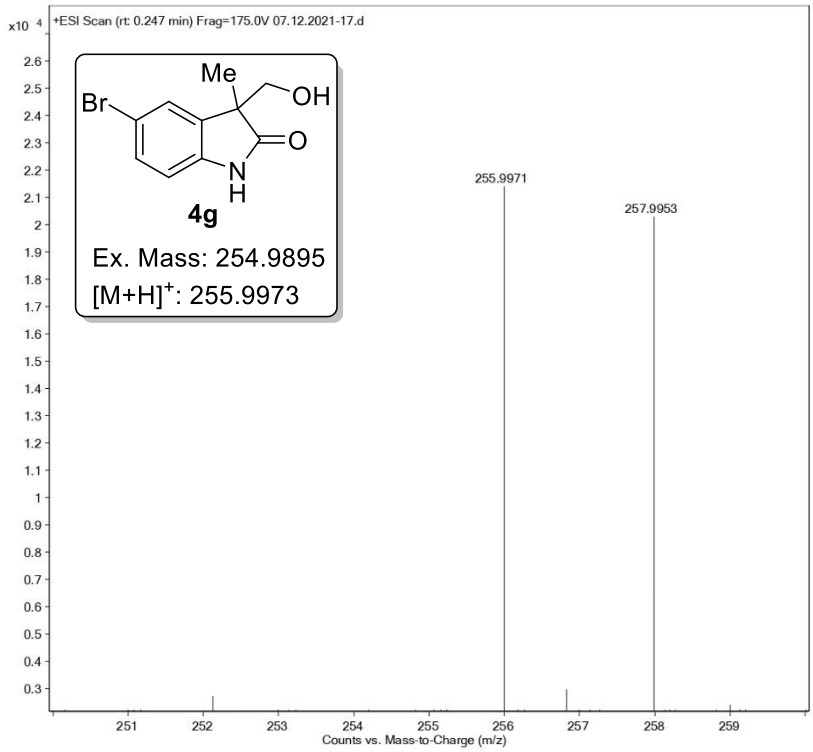


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 5-bromo-3-(hydroxymethyl)-3-methylindolin-2-one (4g)

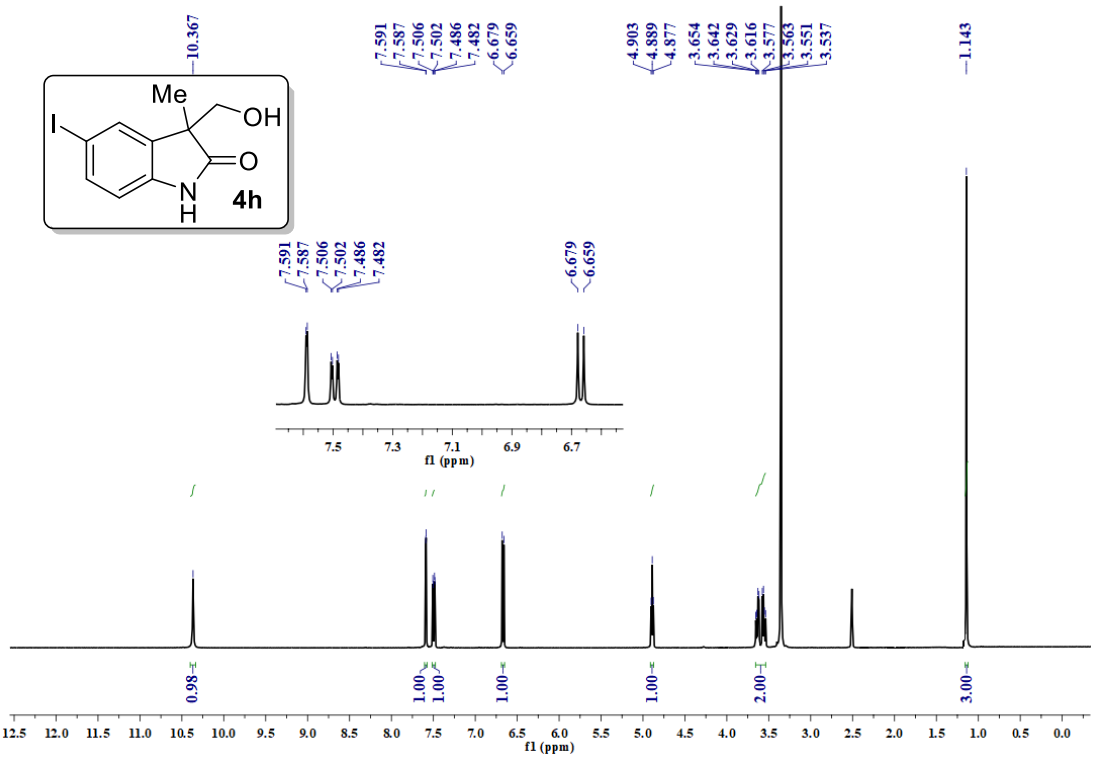


HRMS of 5-bromo-3-(hydroxymethyl)-3-methylindolin-2-one (4g)

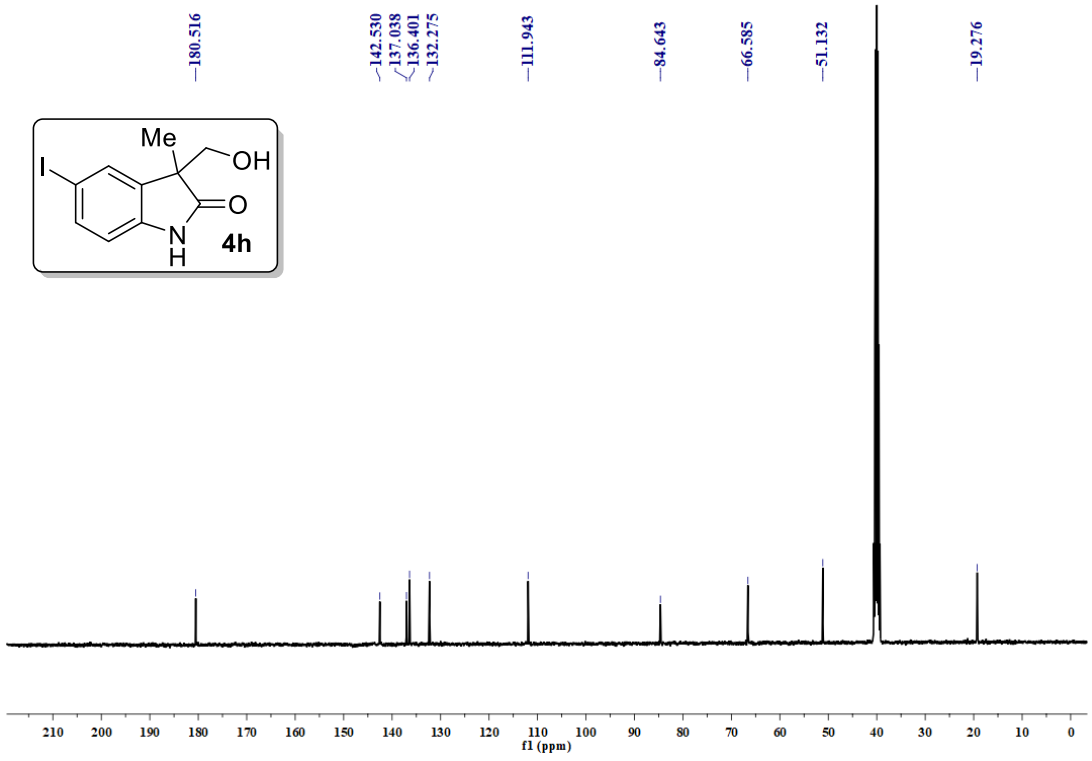
Sample Name	5broxi-1	**Position**	P1-B8	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-17.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 6:20:44 PM



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 3-(hydroxymethyl)-5-iodo-3-methylindolin-2-one (4h)

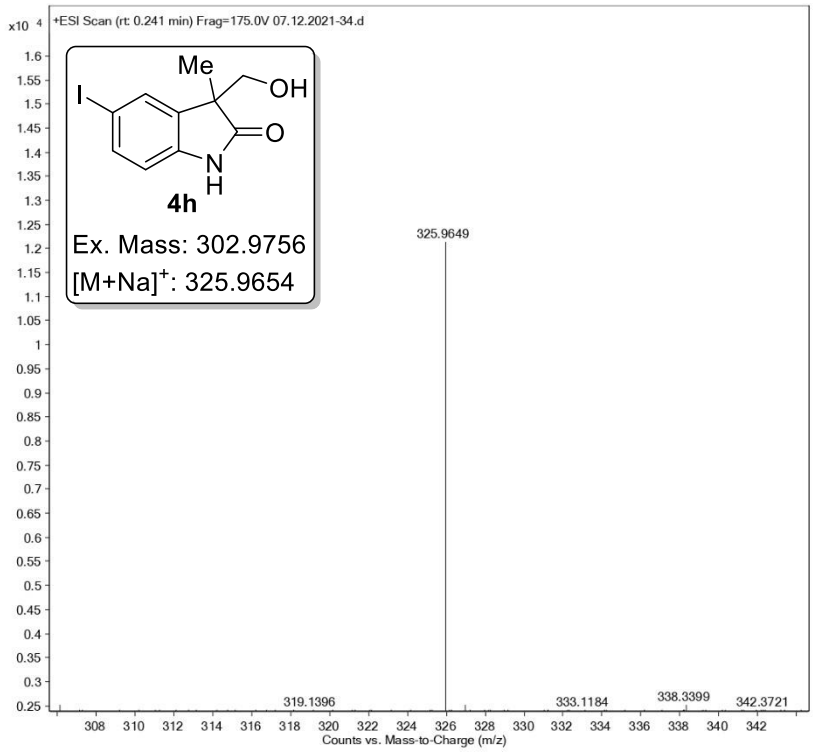


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 3-(hydroxymethyl)-5-iodo-3-methylindolin-2-one (4h)

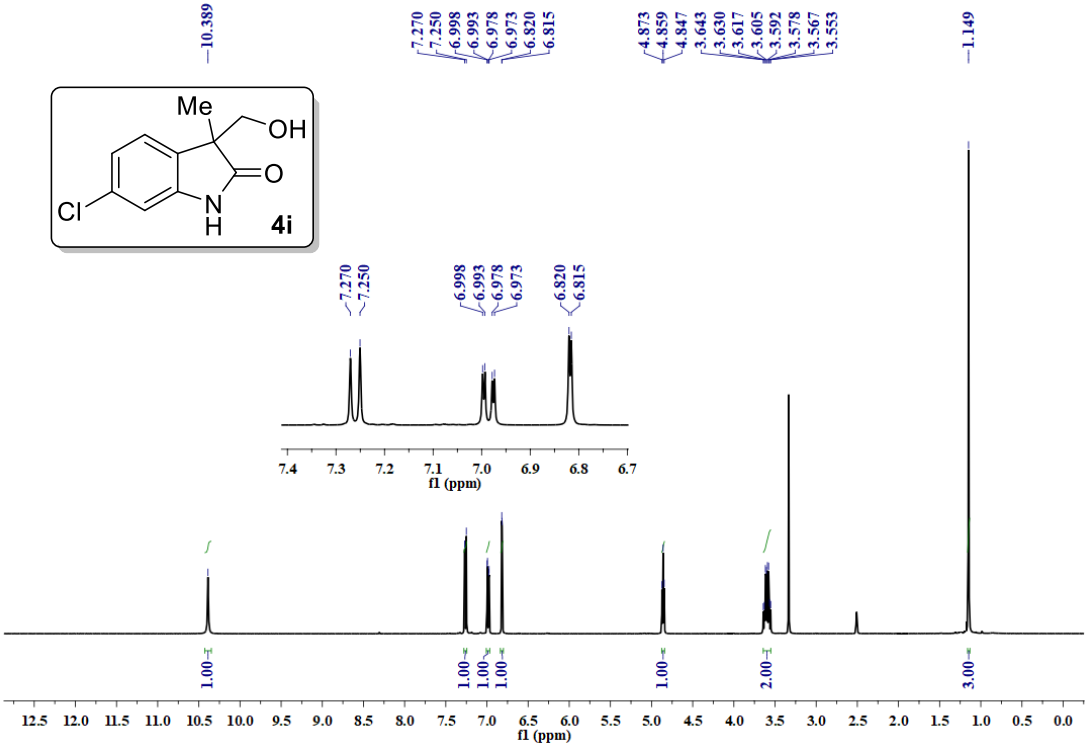


HRMS of 3-(hydroxymethyl)-5-iodo-3-methylindolin-2-one (4h)

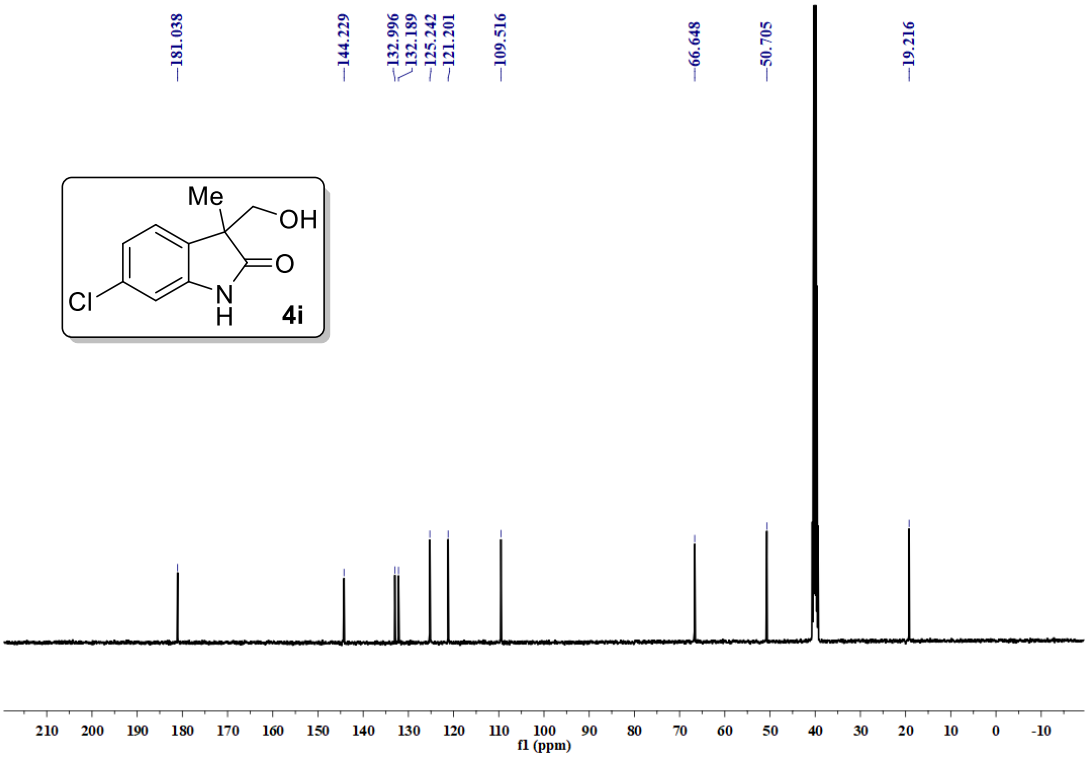
Sample Name	5-ioxi-2	**Position**	P1-D7	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-34.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 7:28:19 PM



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 6-chloro-3-(hydroxymethyl)-3-methylindolin-2-one (4i)

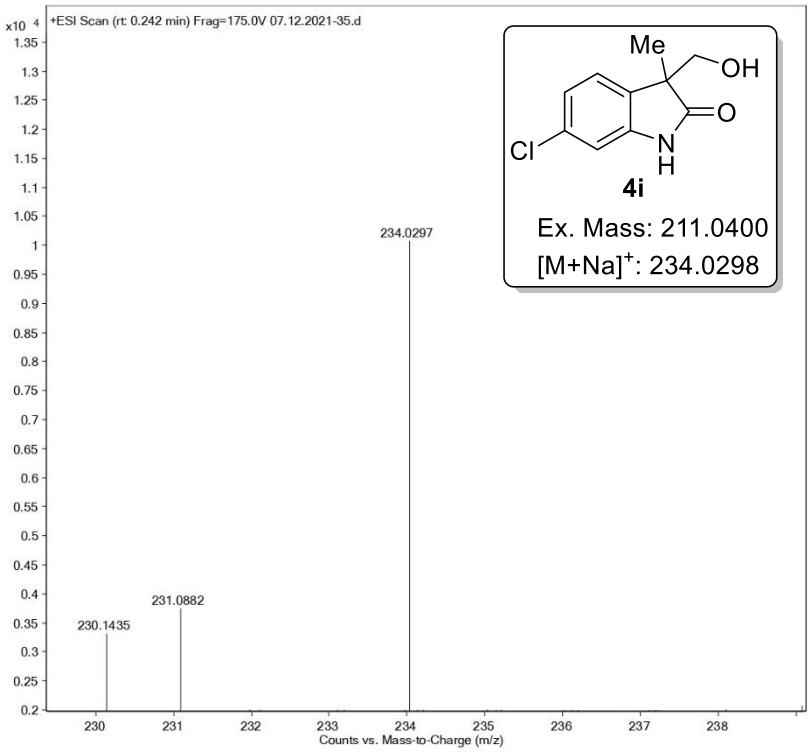


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 6-chloro-3-(hydroxymethyl)-3-methylindolin-2-one (4i)

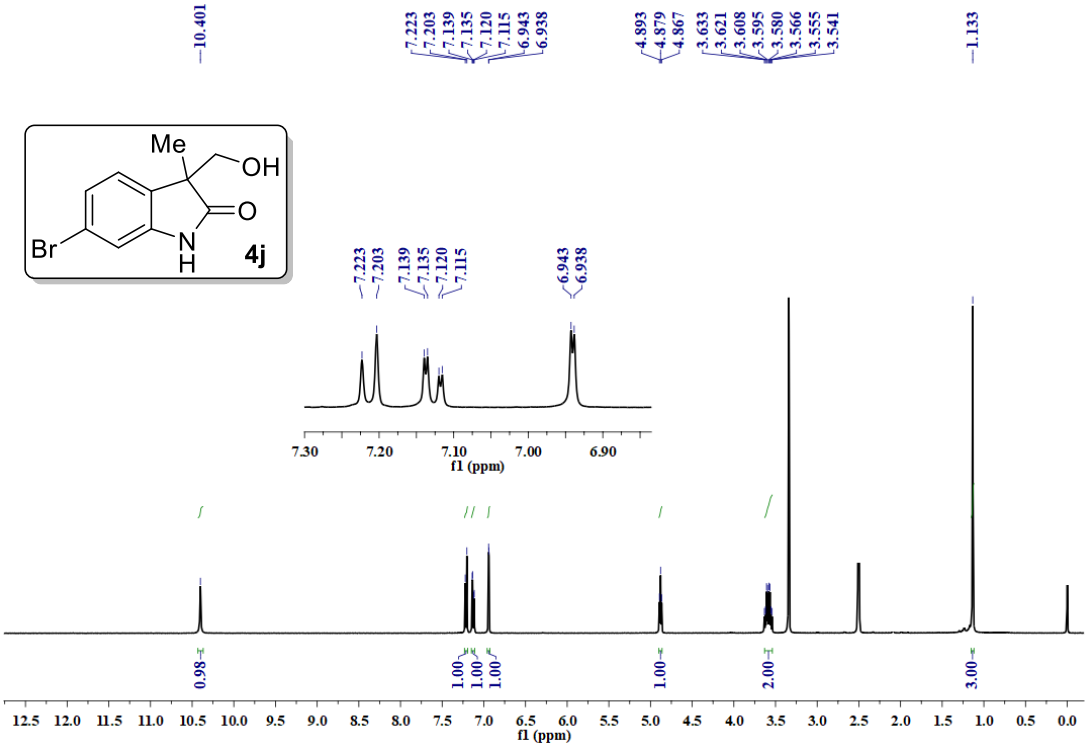


HRMS of 6-chloro-3-(hydroxymethyl)-3-methylindolin-2-one (4i)

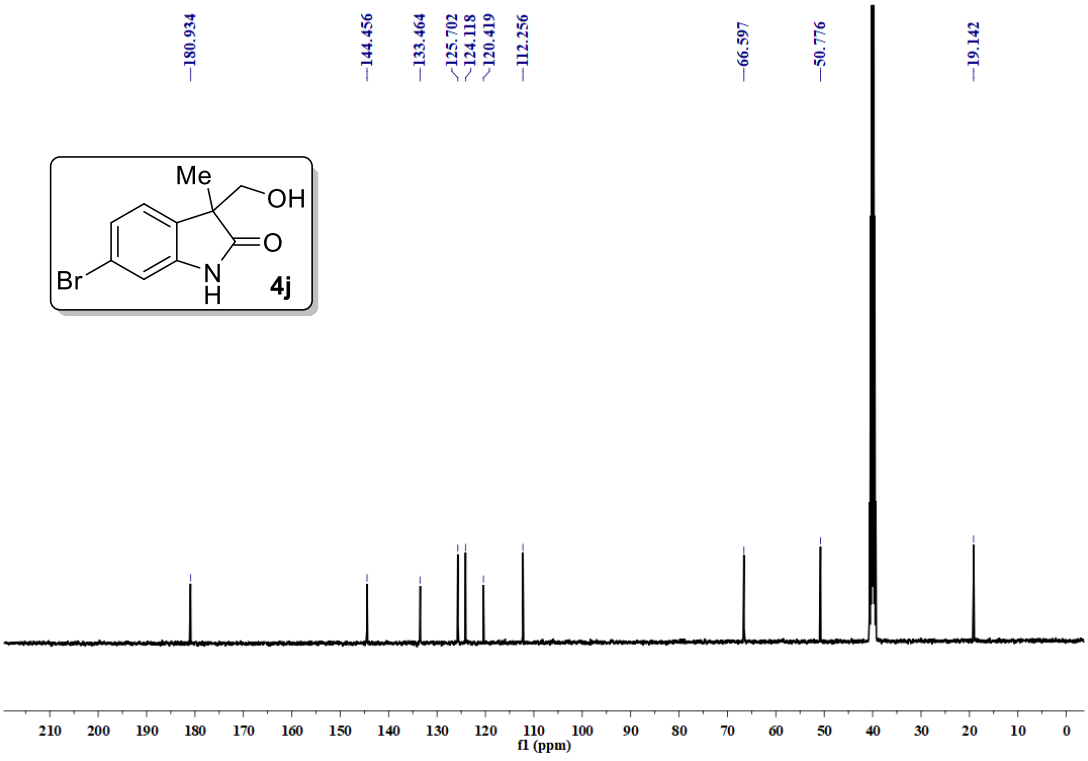
Sample Name	6cl-oxi-2	**Position**	P1-D8	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-35.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 7:32:17 PM



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 6-bromo-3-(hydroxymethyl)-3-methylindolin-2-one (4j)

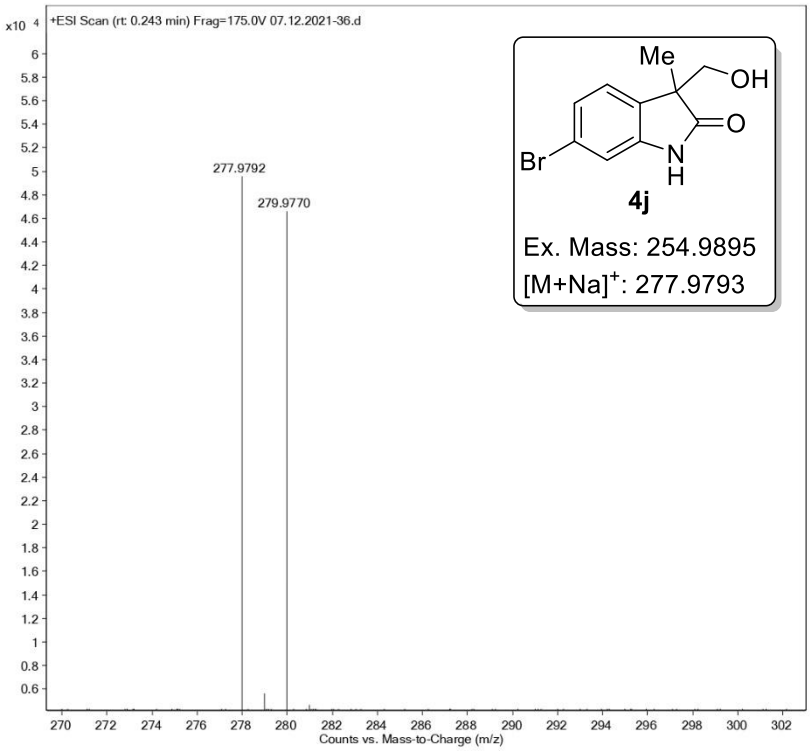


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 6-bromo-3-(hydroxymethyl)-3-methylindolin-2-one (4j)

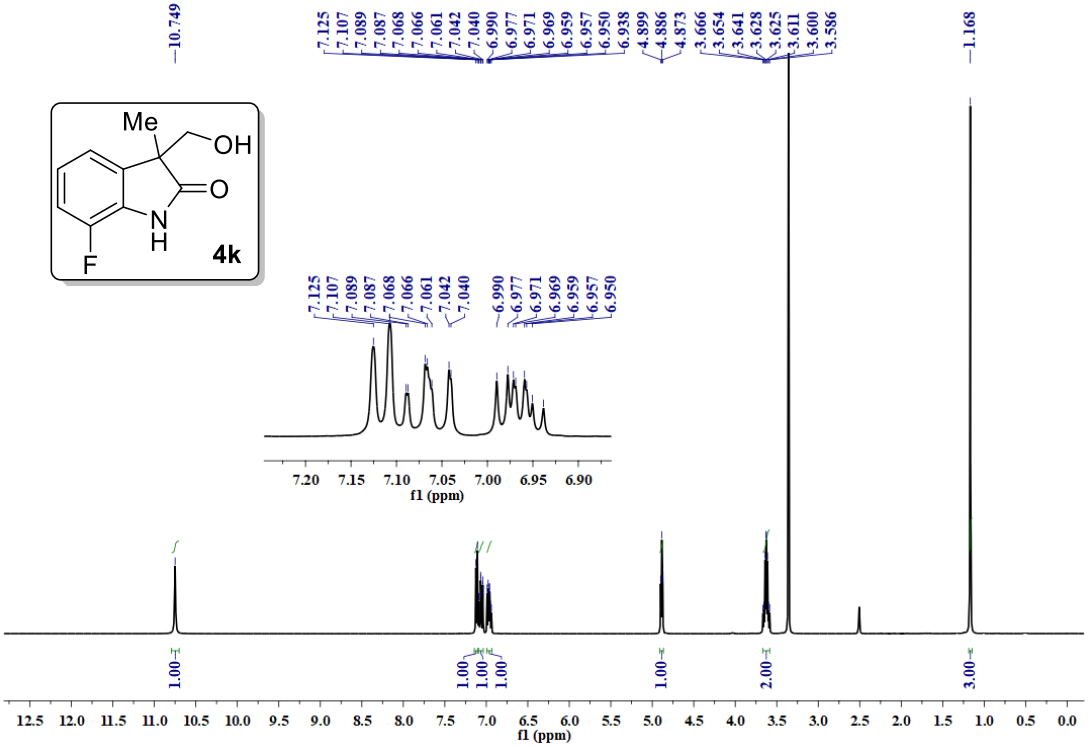


HRMS of 6-bromo-3-(hydroxymethyl)-3-methylindolin-2-one (4j)

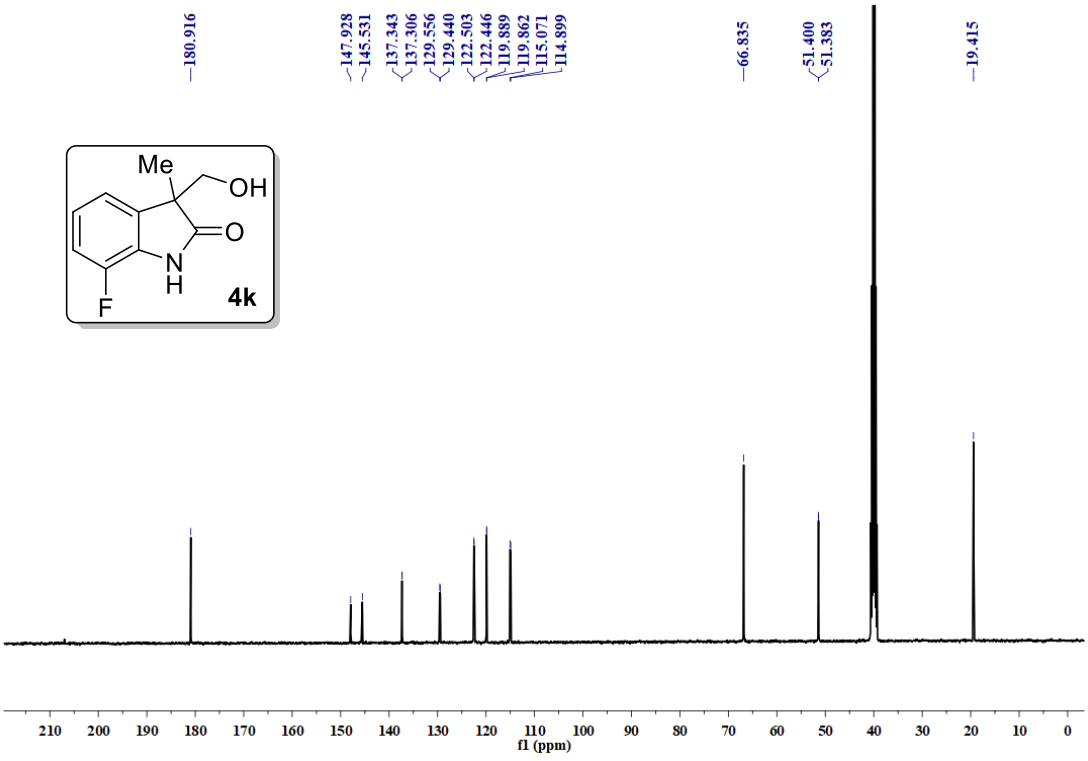
Sample Name	6-broxi-2	**Position**	P1-D9	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-36.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 7:36:16 PM



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 7-fluoro-3-(hydroxymethyl)-3-methylindolin-2-one (4k)

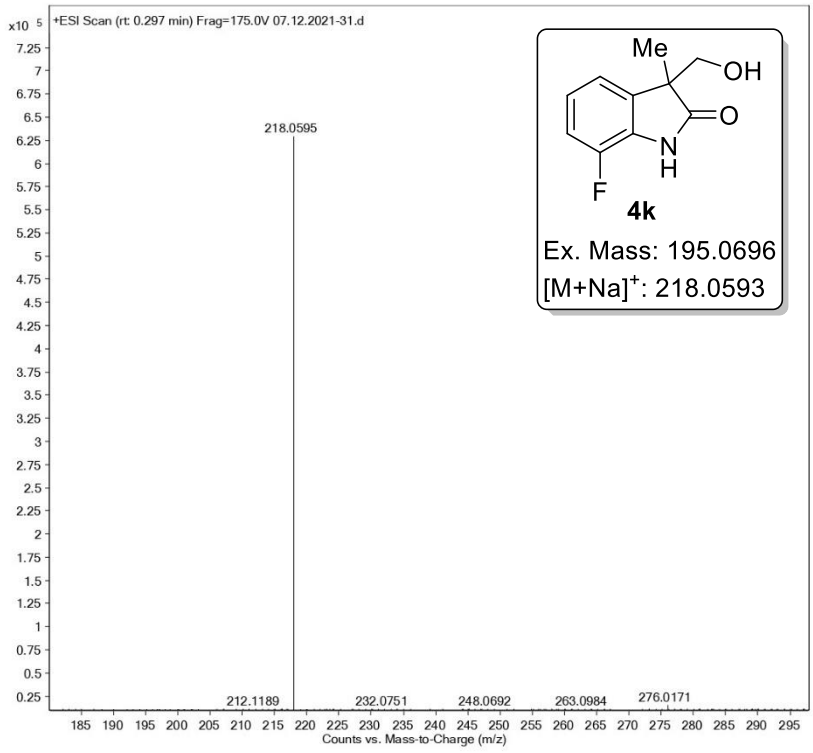


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 7-fluoro-3-(hydroxymethyl)-3-methylindolin-2-one (4k)

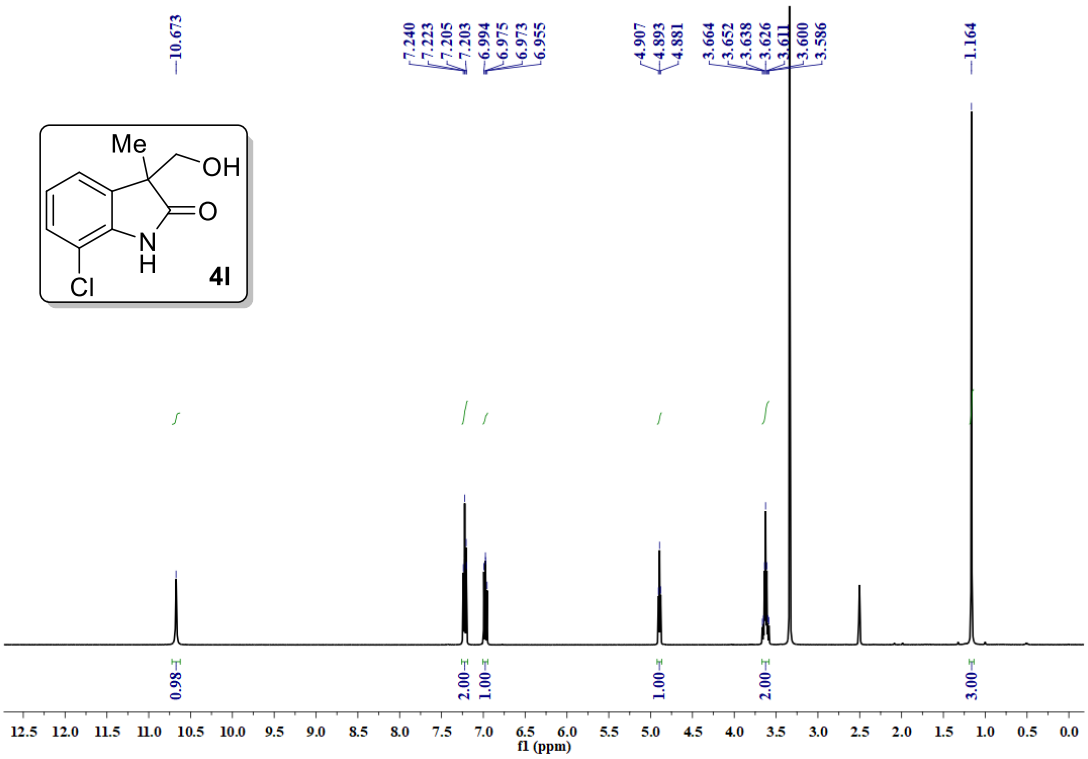


HRMS of 7-fluoro-3-(hydroxymethyl)-3-methylindolin-2-one (4k)

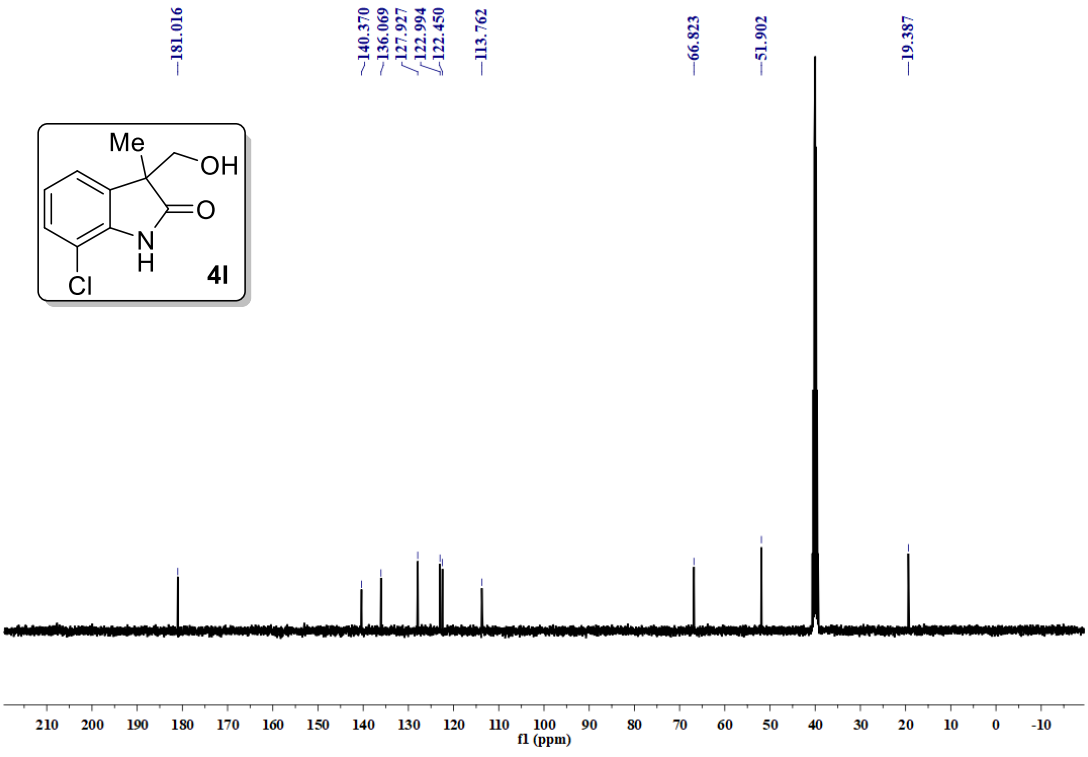
Sample Name	7-foxi-2	**Position**	P1-D6	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-31.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-2017: 18:22 PM



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 7-chloro-3-(hydroxymethyl)-3-methylindolin-2-one (4l)

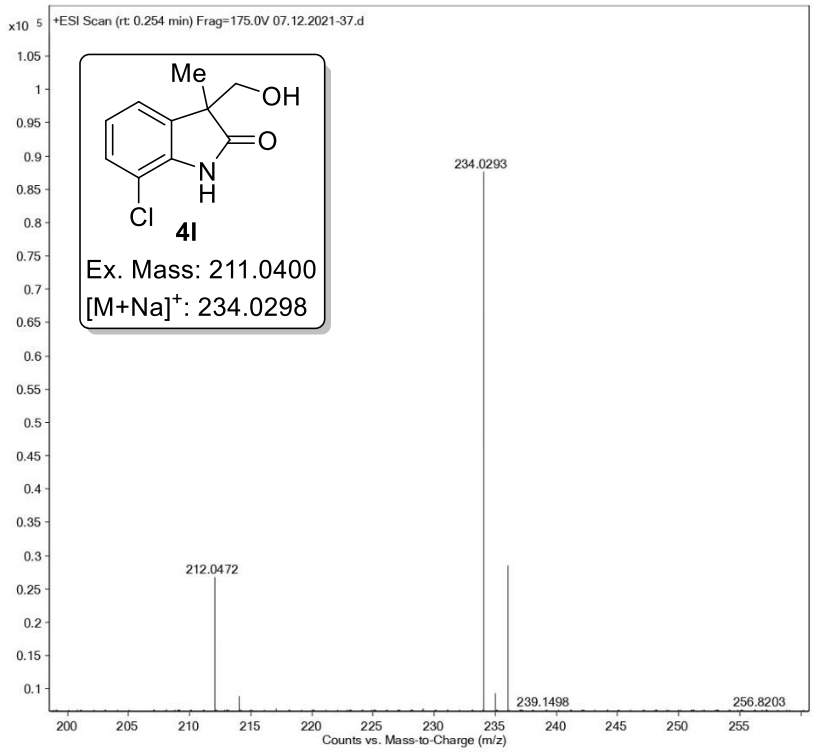


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 7-chloro-3-(hydroxymethyl)-3-methylindolin-2-one (4l)

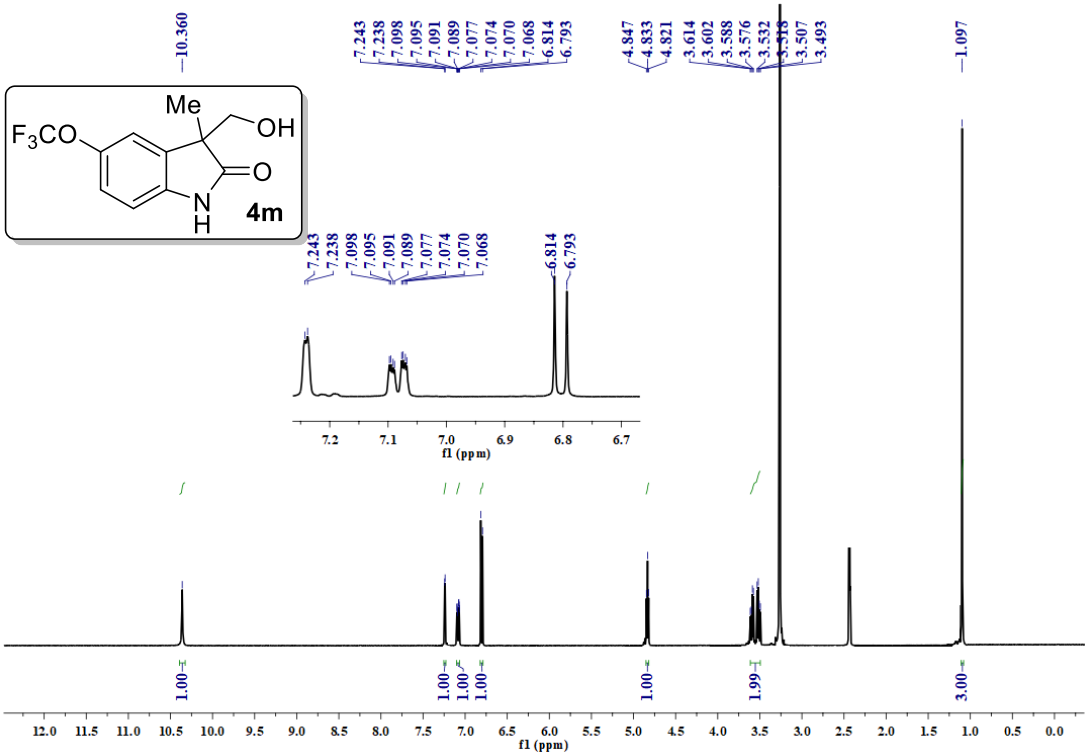


HRMS of 7-chloro-3-(hydroxymethyl)-3-methylindolin-2-one (4l)

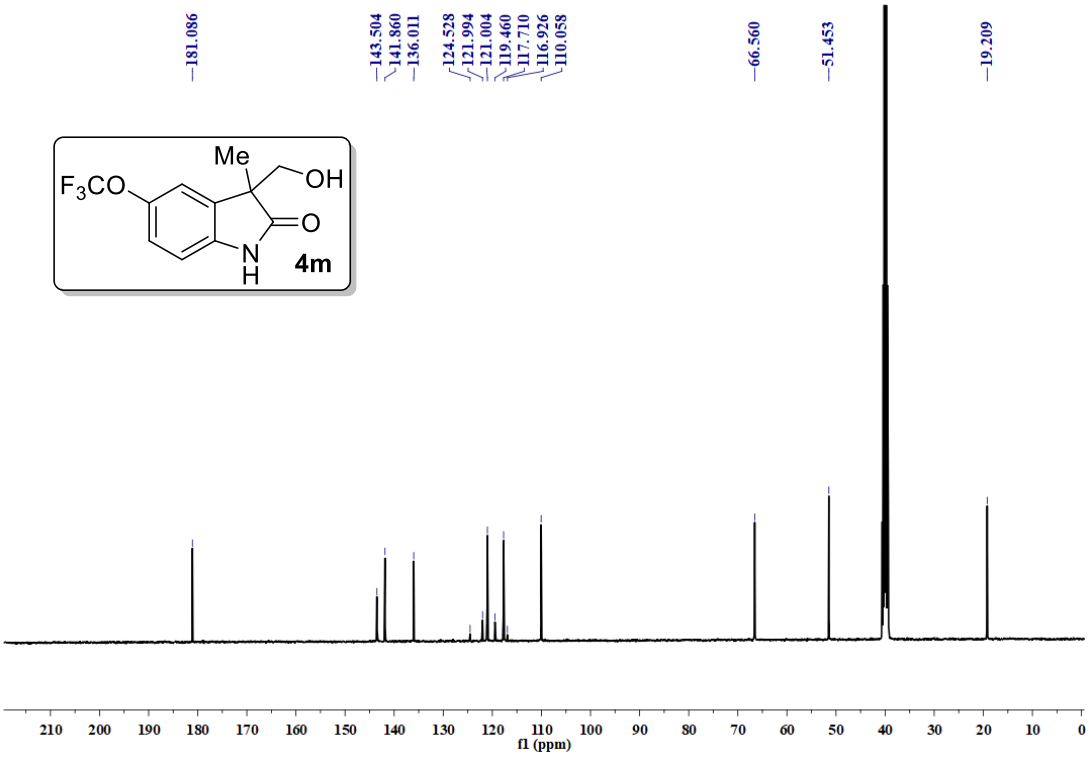
Sample Name	7-cloxi-2	**Position**	P1-E1	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-37.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 7:40:13 PM



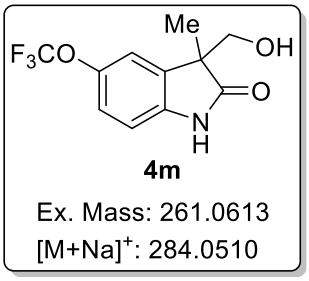
^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 3-(hydroxymethyl)-3-methyl-5-(trifluoromethoxy)indolin-2-one (4m)



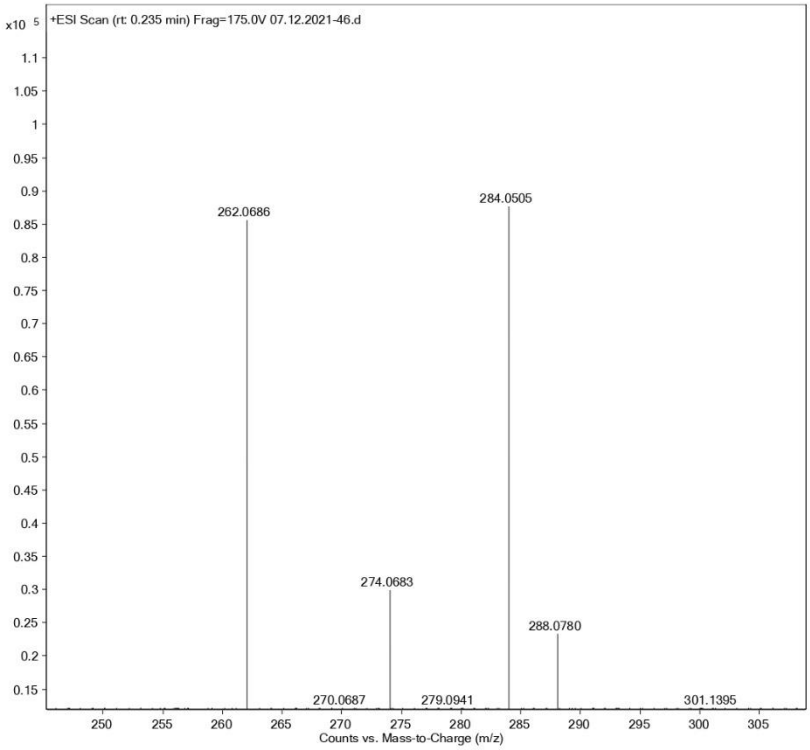
^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 3-(hydroxymethyl)-3-methyl-5-(trifluoromethoxy)indolin-2-one (4m)



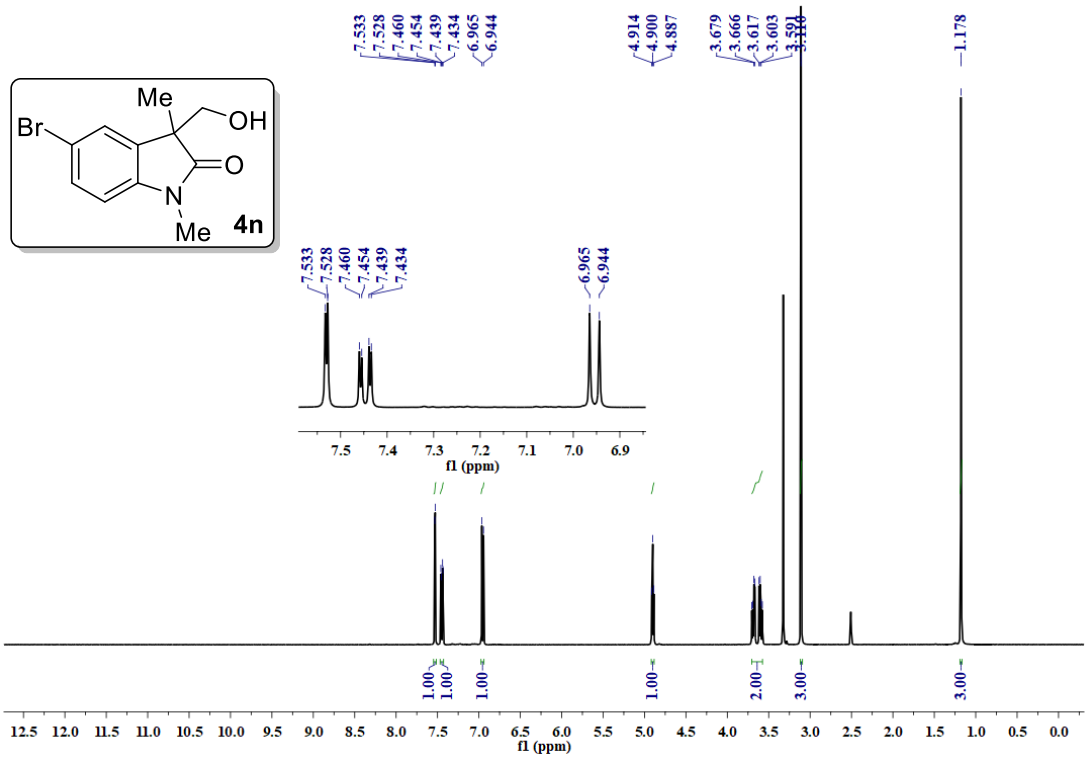
HRMS of 3-(hydroxymethyl)-3-methyl-5-(trifluoromethoxy)indolin-2-one (4m)



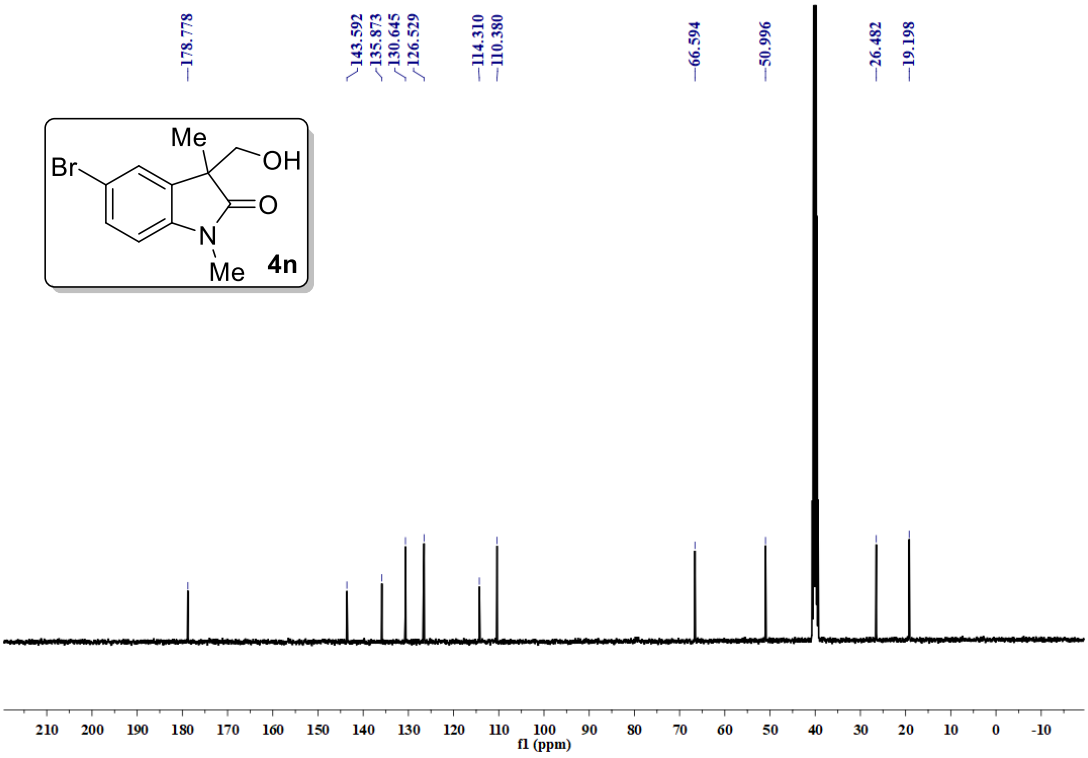
Sample Name	5 ocf3oxi-2	**Position**	P1-F1	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-46.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 8:16:01 PM



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 5-bromo-3-(hydroxymethyl)-1,3-dimethylindolin-2-one (4n)

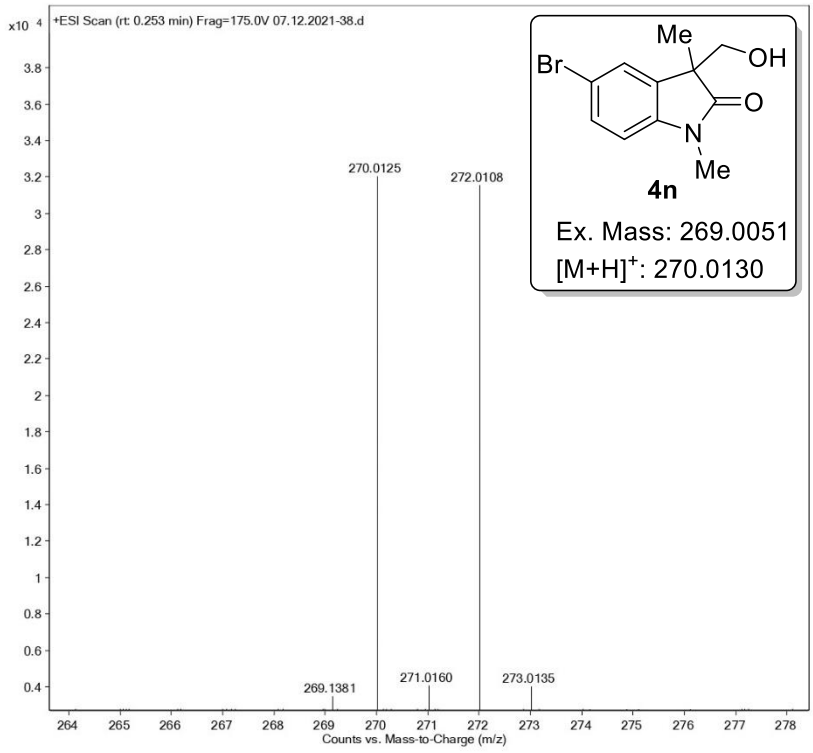


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 5-bromo-3-(hydroxymethyl)-1,3-dimethylindolin-2-one (4n)

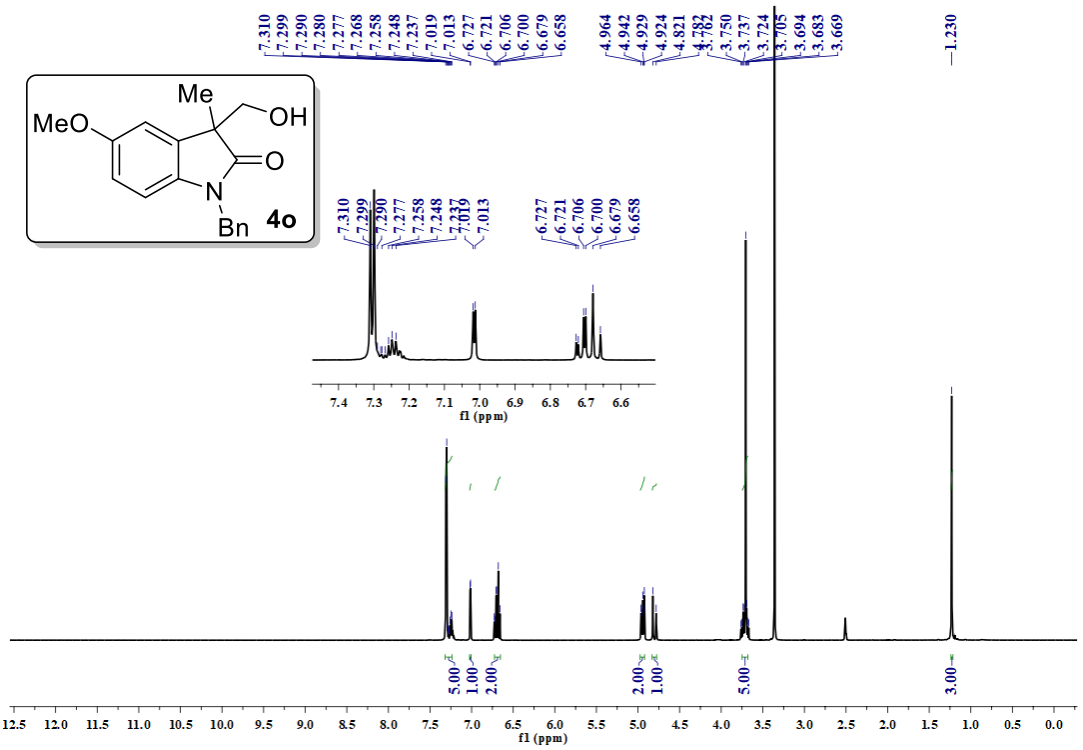


HRMS of 5-bromo-3-(hydroxymethyl)-1,3-dimethylindolin-2-one (4n)

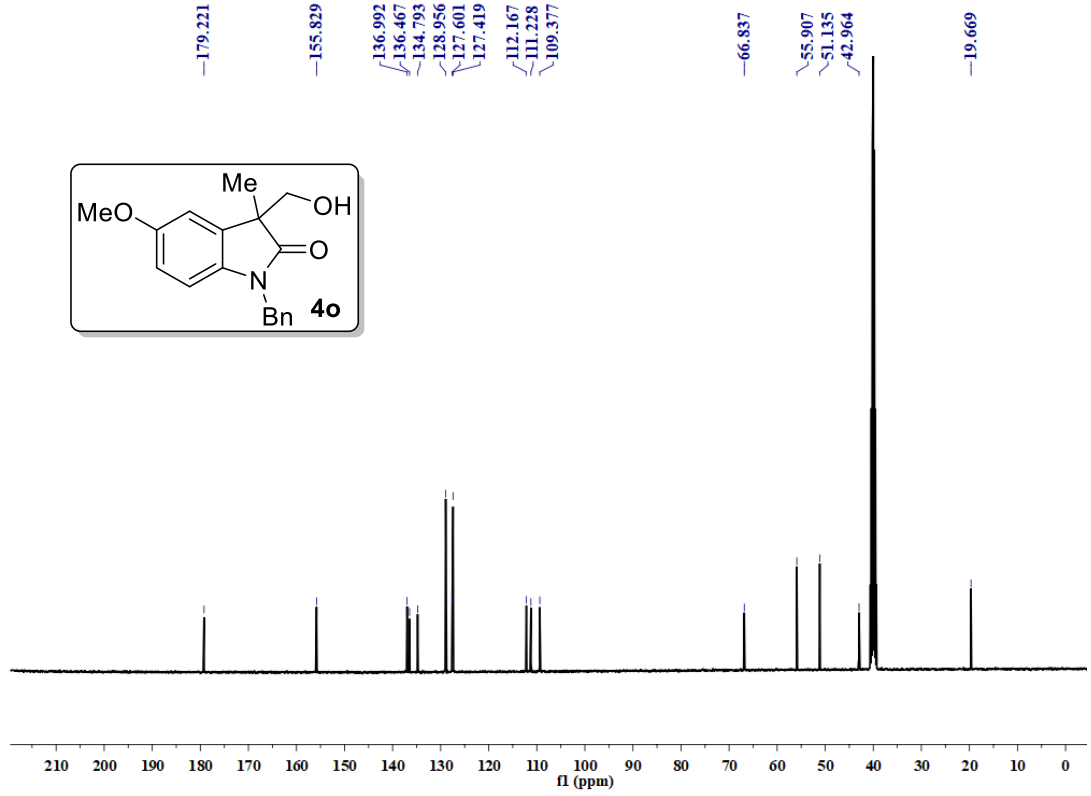
Sample Name	5-br nmeoxi-2	**Position**	P1-E2	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-38.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 7:44:12 PM



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 1-benzyl-3-(hydroxymethyl)-5-methoxy-3-methylindolin-2-one (4o)

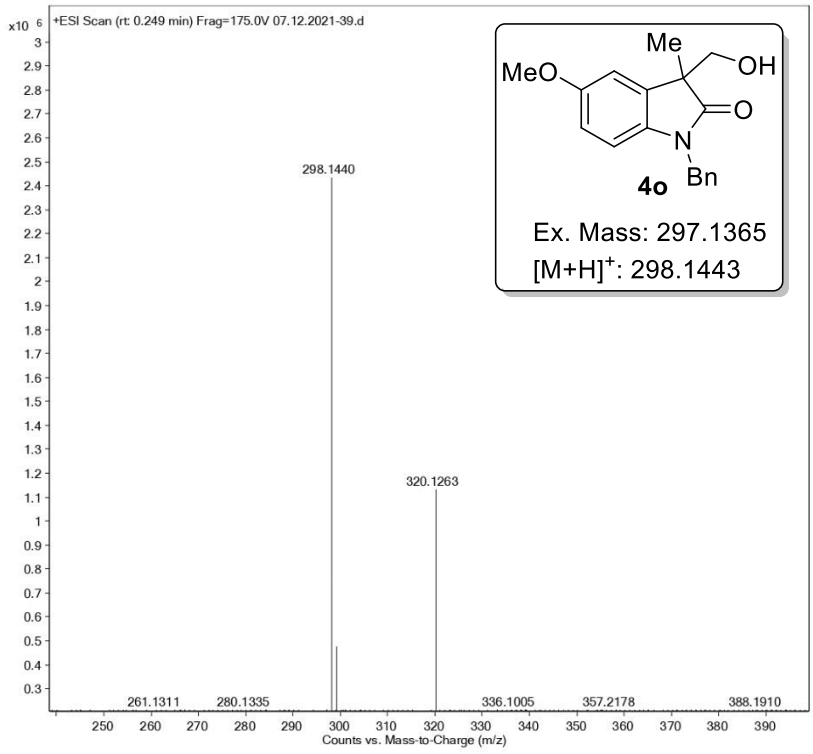


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 1-benzyl-3-(hydroxymethyl)-5-methoxy-3-methylindolin-2-one (4o)

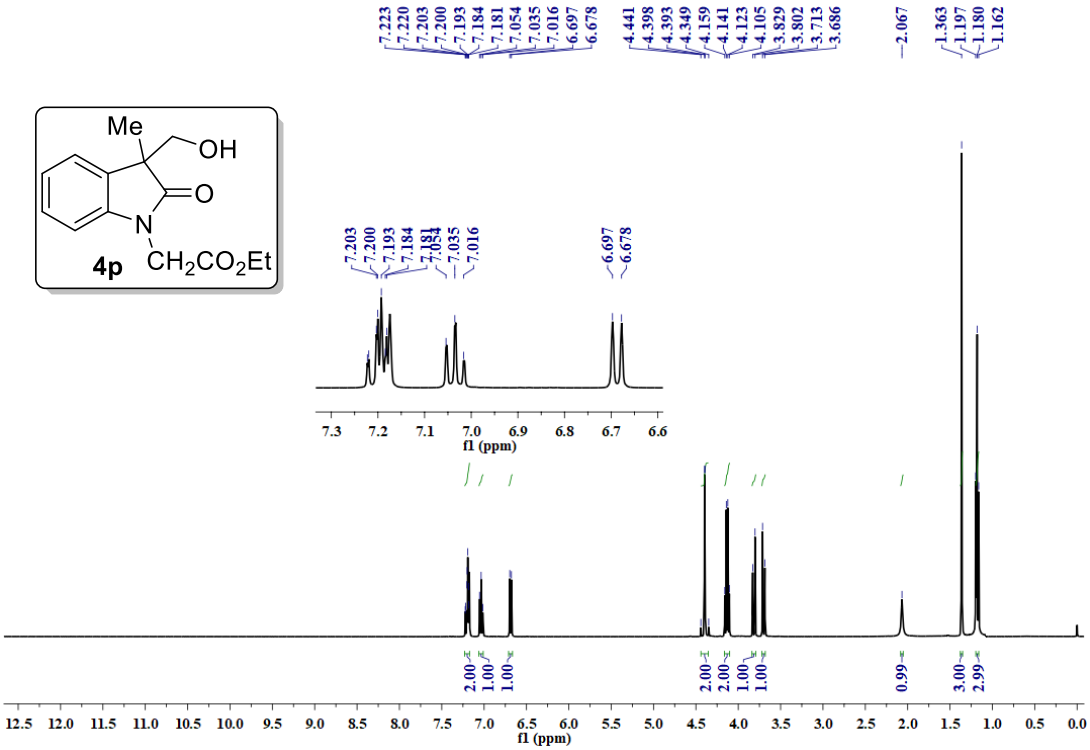


HRMS of 1-benzyl-3-(hydroxymethyl)-5-methoxy-3-methylindolin-2-one (4o)

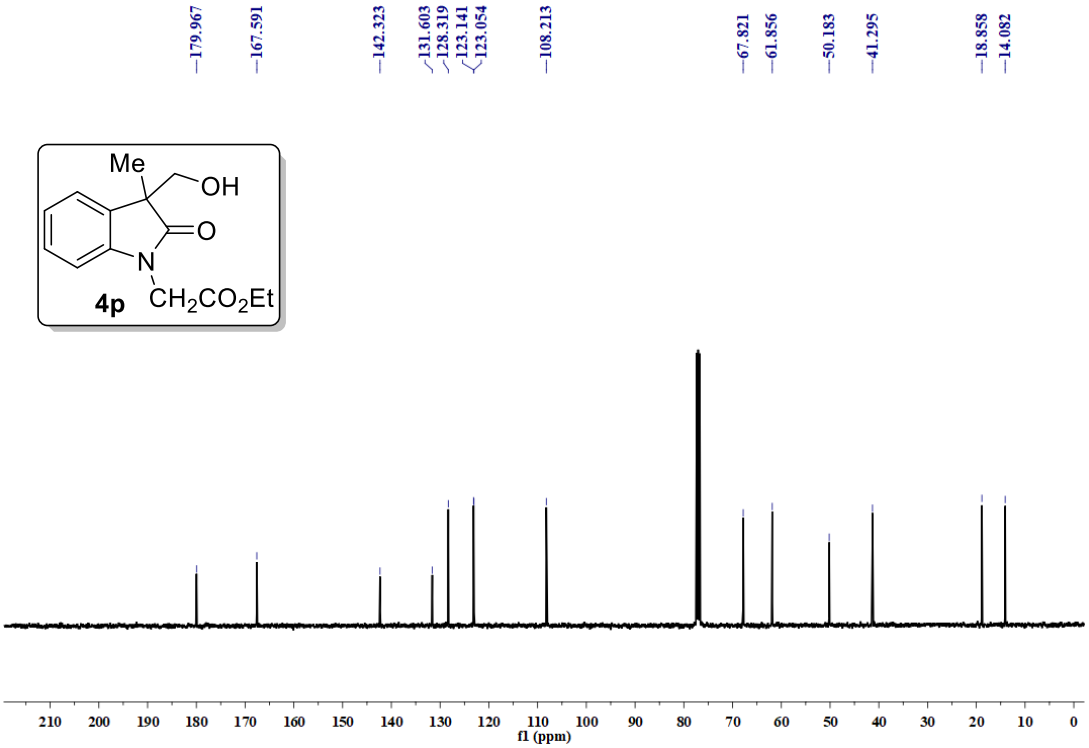
Sample Name	5-omenboxi-2	**Position**	P1-E3	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-39.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 7:48:11 PM



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of ethyl 2-(3-(hydroxymethyl)-3-methyl-2-oxoindolin-1-yl)acetate (4p)

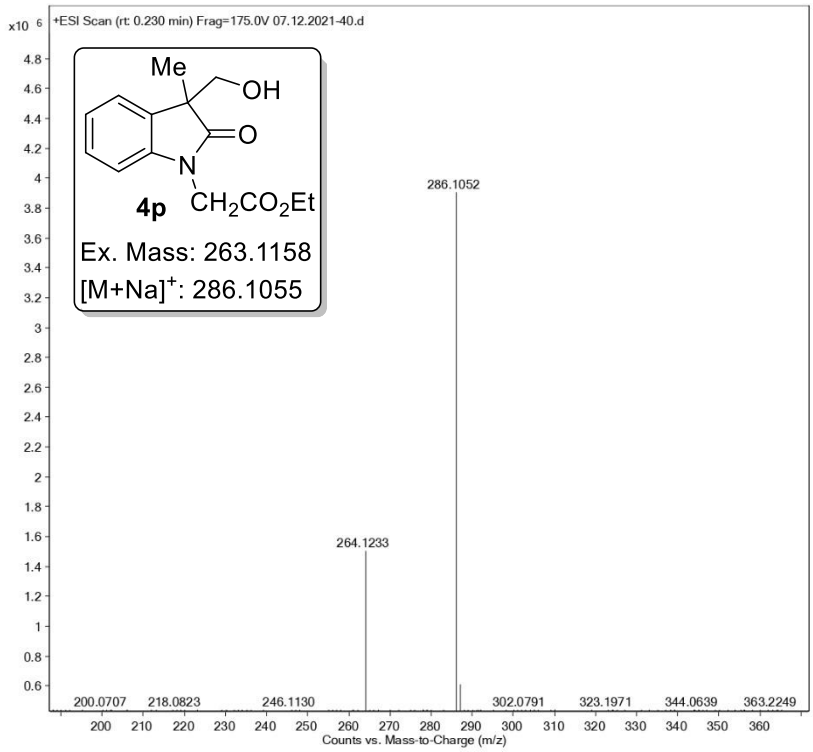


^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of ethyl 2-(3-(hydroxymethyl)-3-methyl-2-oxoindolin-1-yl)acetate (4p)

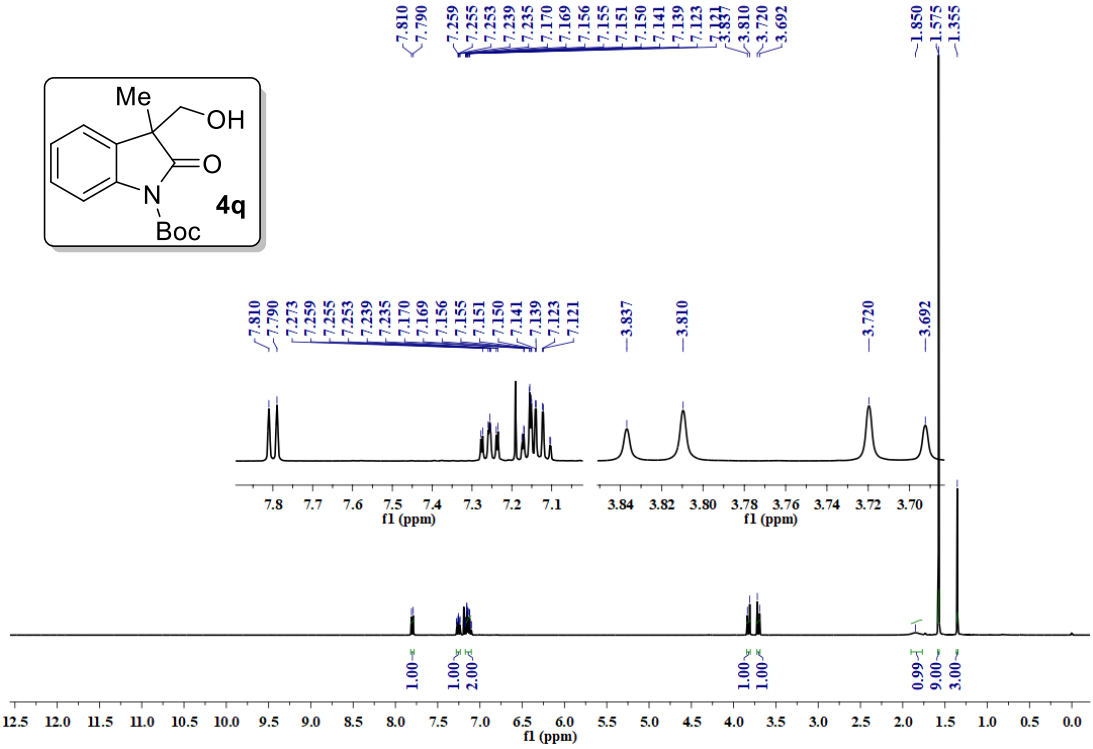


HRMS of ethyl 2-(3-(hydroxymethyl)-3-methyl-2-oxoindolin-1-yl)acetate (4p)

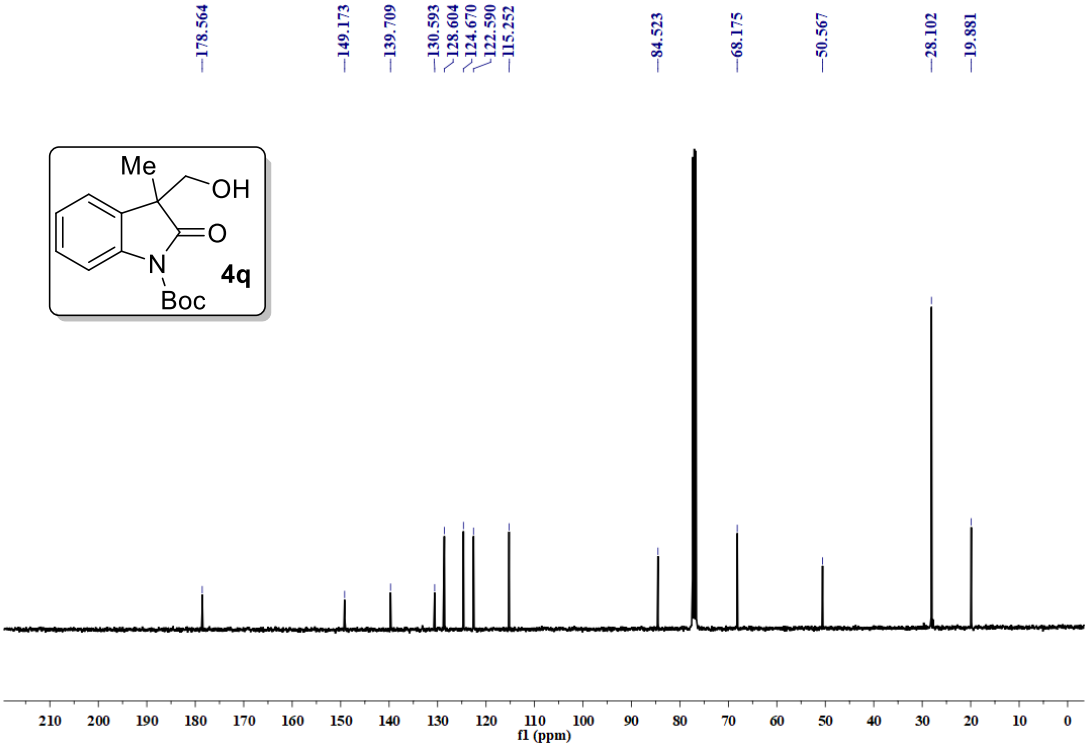
Sample Name	NCH2CO2ETOXI-2	**Position**	P1-E4	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-40.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 7:52:10 PM



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of *tert*-butyl 3-(hydroxymethyl)-3-methyl-2-oxoindoline-1-carboxylate (4q)

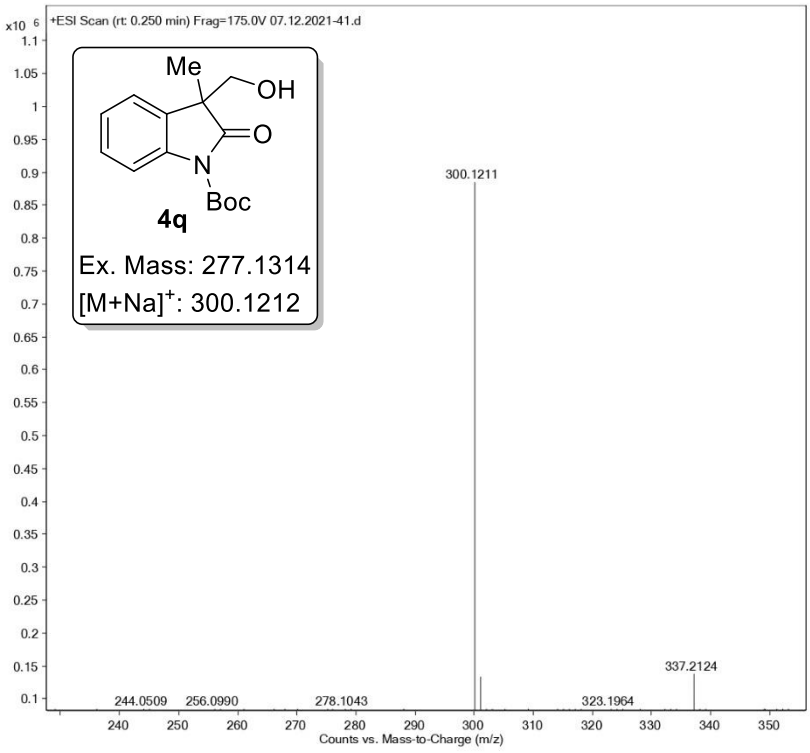


^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of *tert*-butyl 3-(hydroxymethyl)-3-methyl-2-oxoindoline-1-carboxylate (4q)

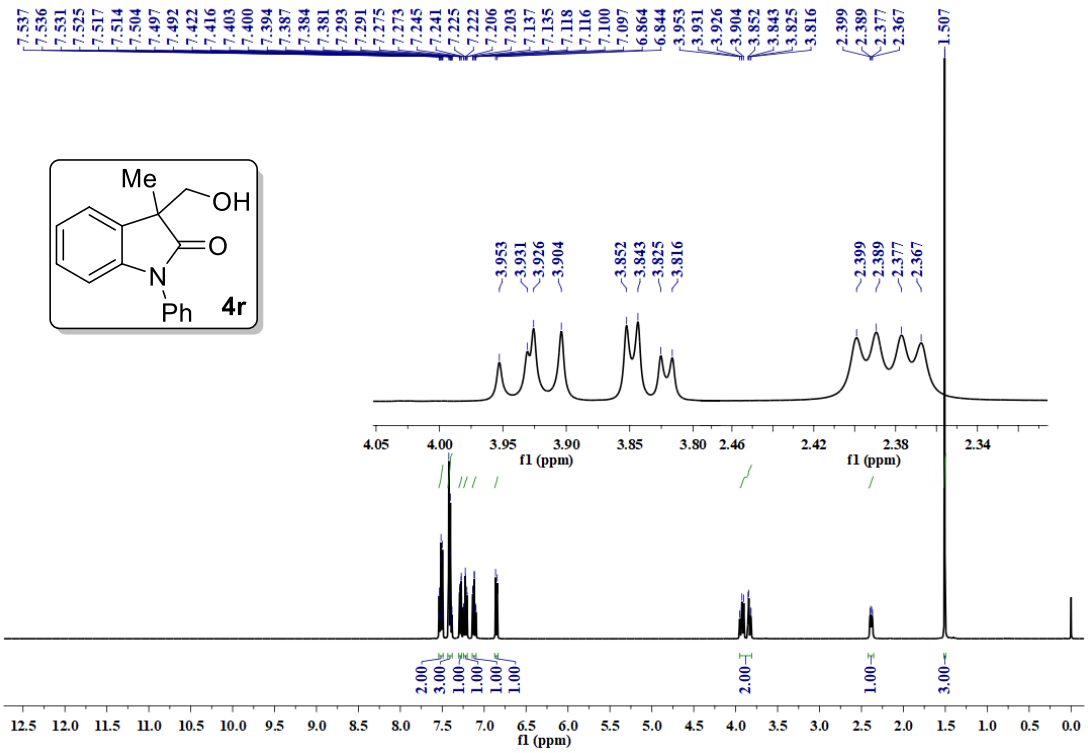


HRMS of *tert*-butyl 3-(hydroxymethyl)-3-methyl-2-oxoindoline-1-carboxylate (4q)

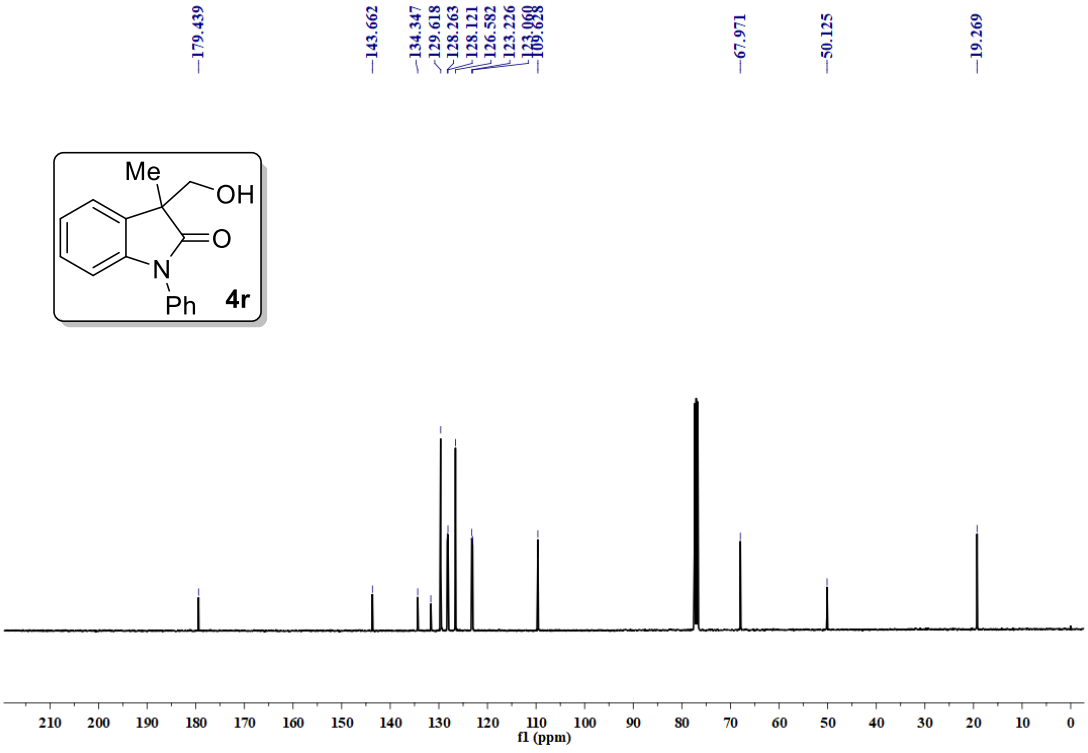
Sample Name	NBOCOXI-2	**Position**	P1-E5	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-41.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 7:56:08 PM



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3-(hydroxymethyl)-3-methyl-1-phenylindolin-2-one (4r)

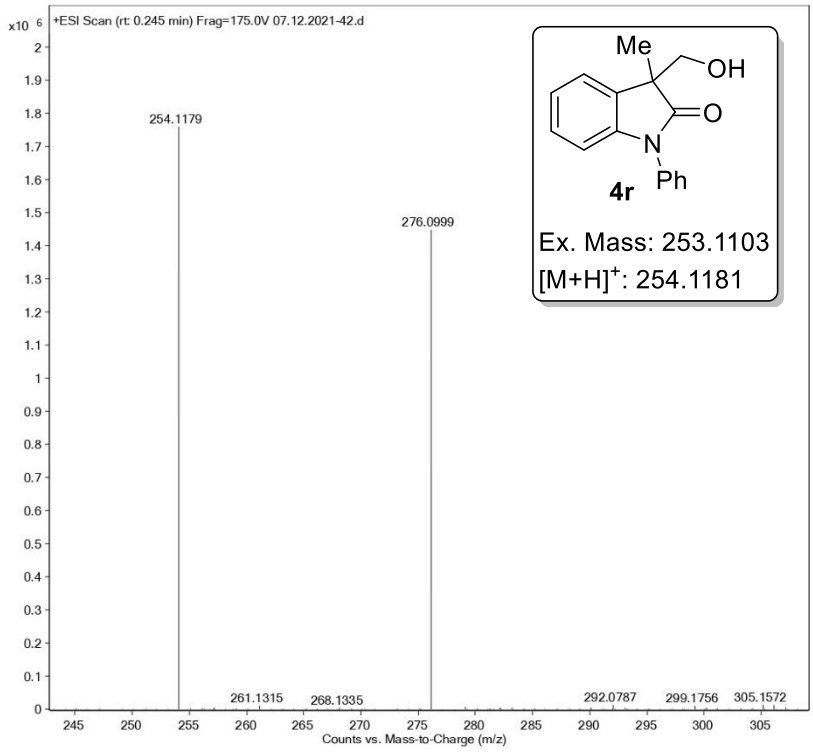


^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of 3-(hydroxymethyl)-3-methyl-1-phenylindolin-2-one (4r)

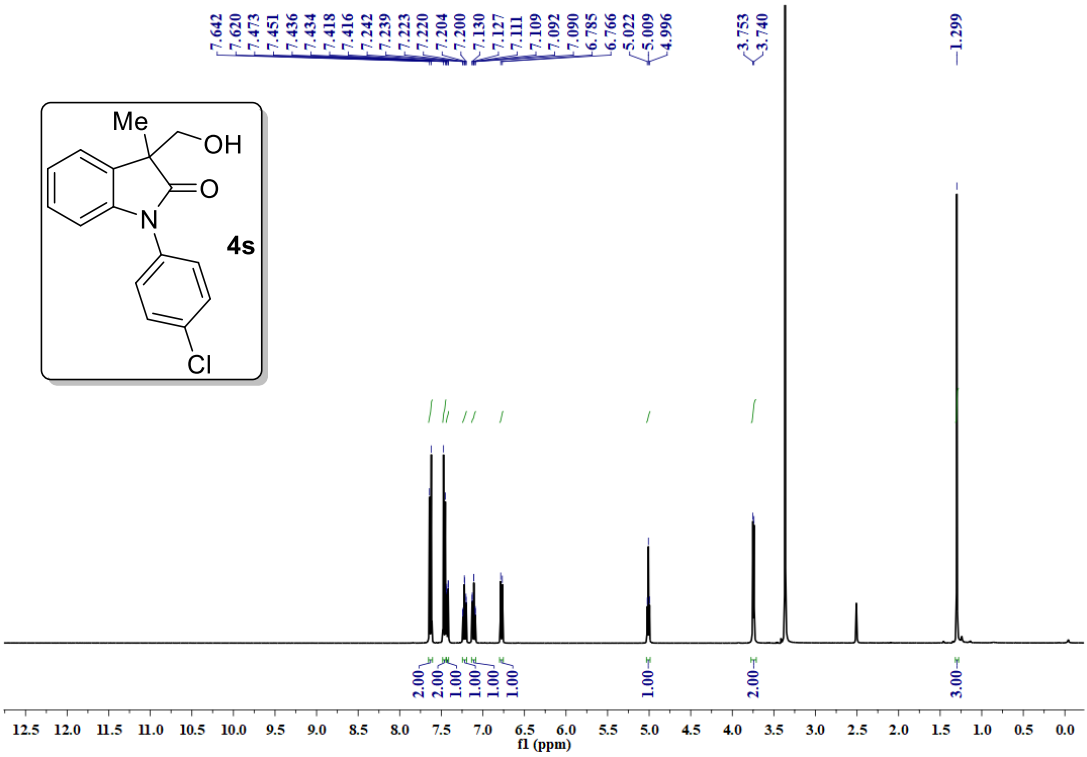


HRMS of 3-(hydroxymethyl)-3-methyl-1-phenylindolin-2-one (4r)

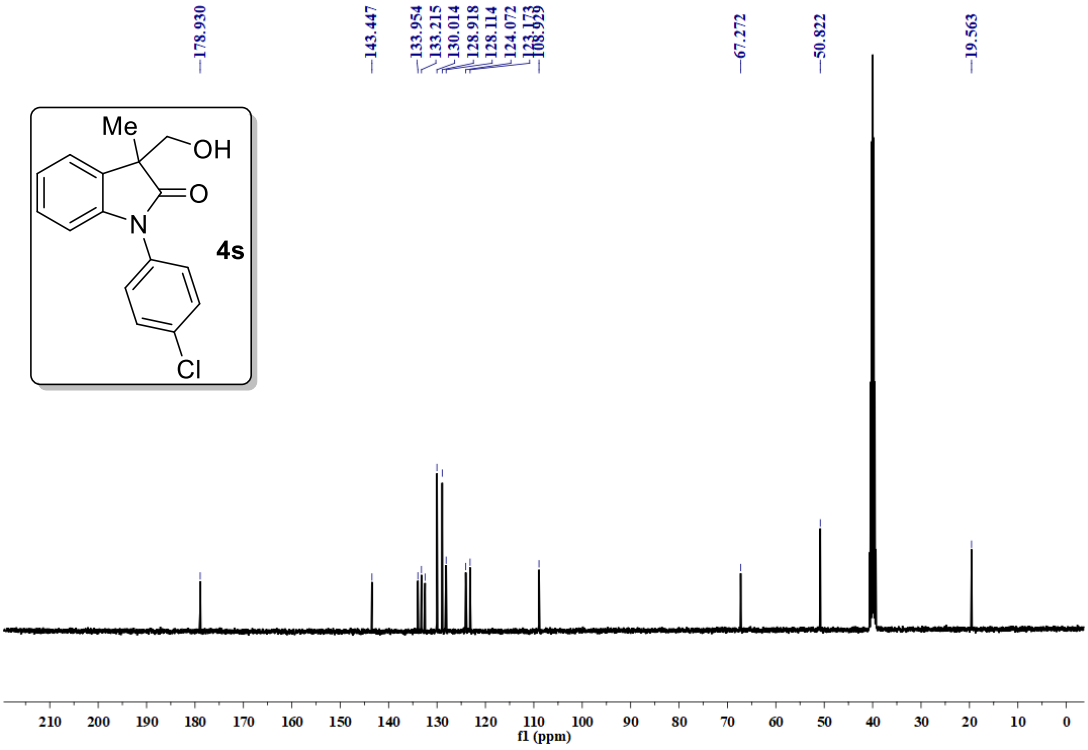
Sample Name	NPHOXI-2	**Position**	P1-E6	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-42.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 8:00:07 PM



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 1-(4-chlorophenyl)-3-(hydroxymethyl)-3-methylindolin-2-one (4s)

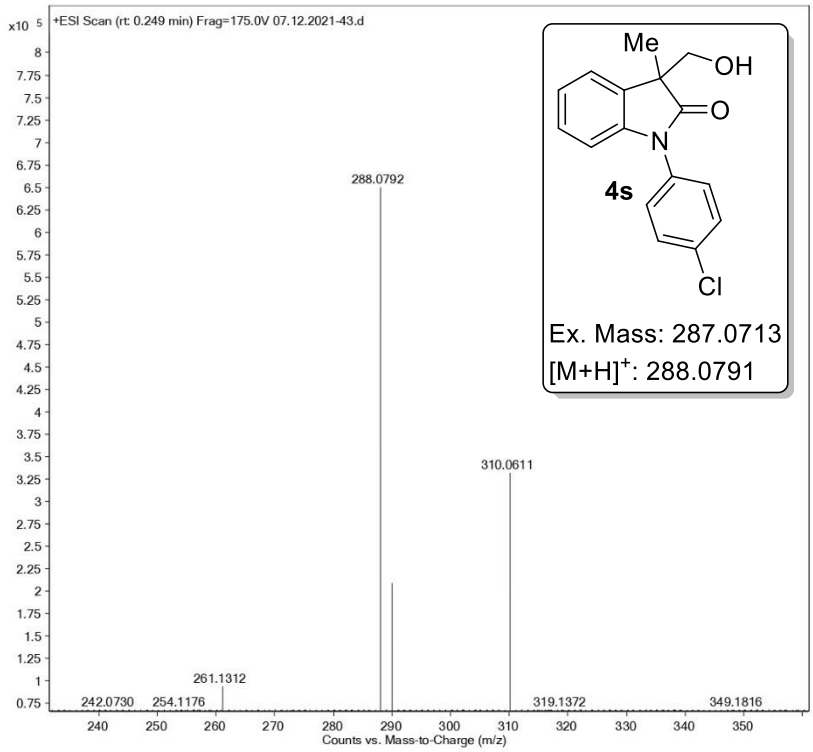


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 1-(4-chlorophenyl)-3-(hydroxymethyl)-3-methylindolin-2-one (4s)

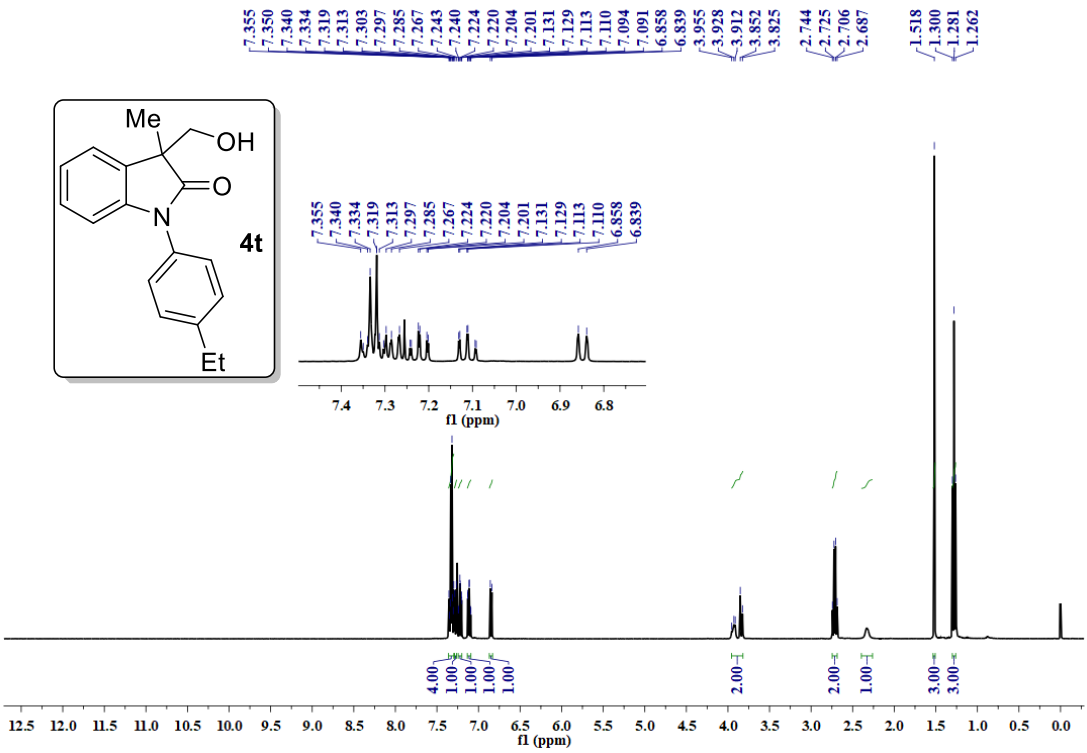


HRMS of 1-(4-chlorophenyl)-3-(hydroxymethyl)-3-methylindolin-2-one (4s)

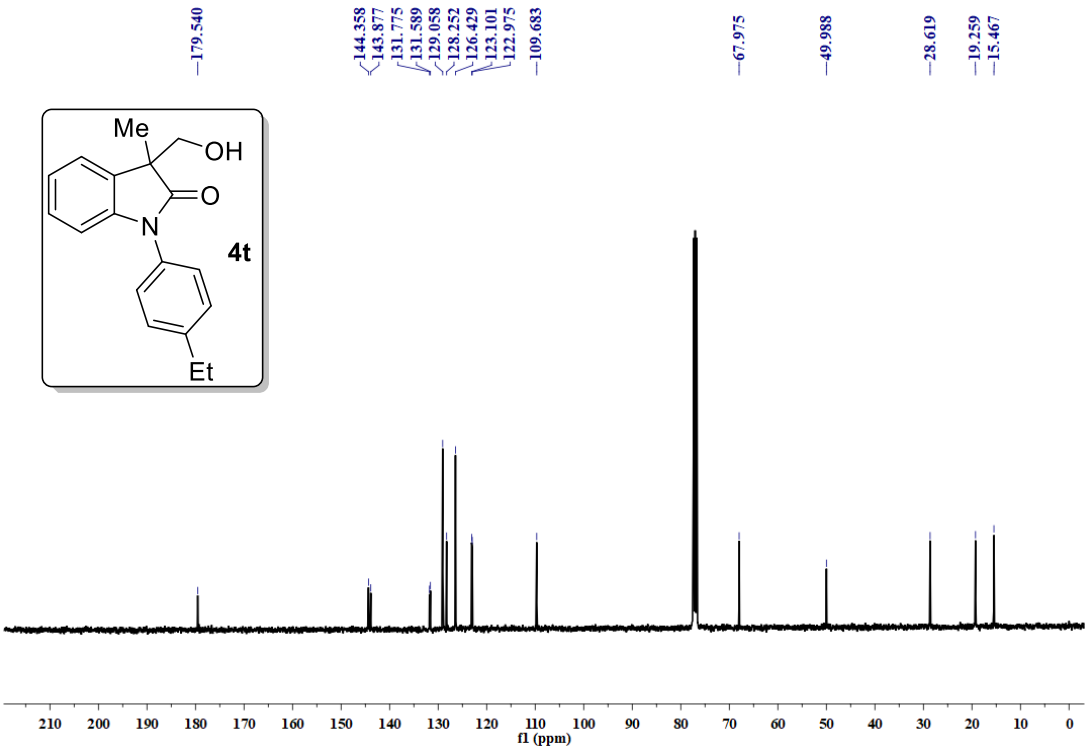
Sample Name	Nuclphoxi-2	**Position**	P1-E7	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-43.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 8:04:05 PM



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 1-(4-ethylphenyl)-3-(hydroxymethyl)-3-methylindolin-2-one (4t)

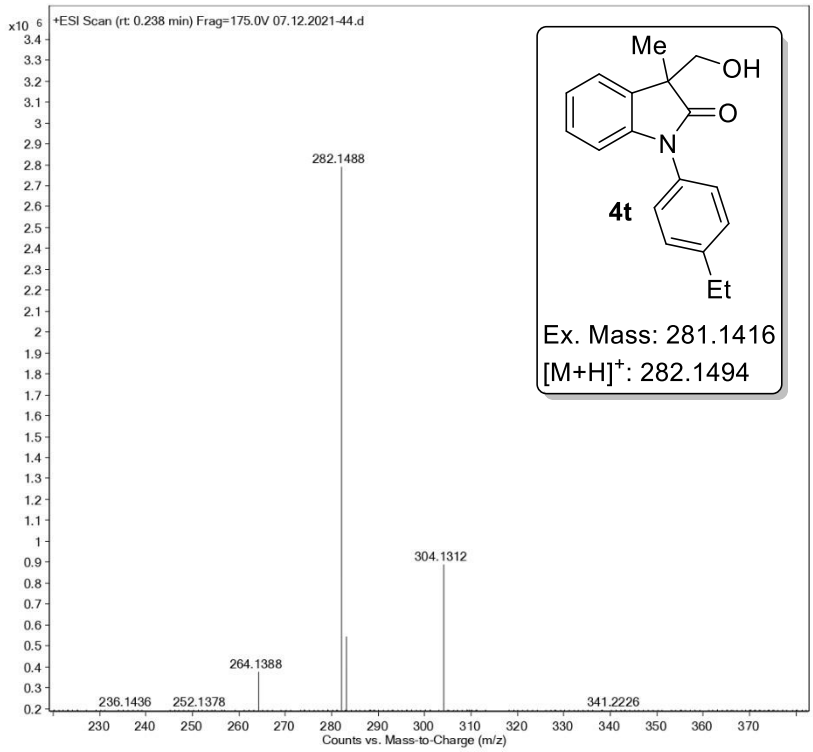


^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of 1-(4-ethylphenyl)-3-(hydroxymethyl)-3-methylindolin-2-one (4t)

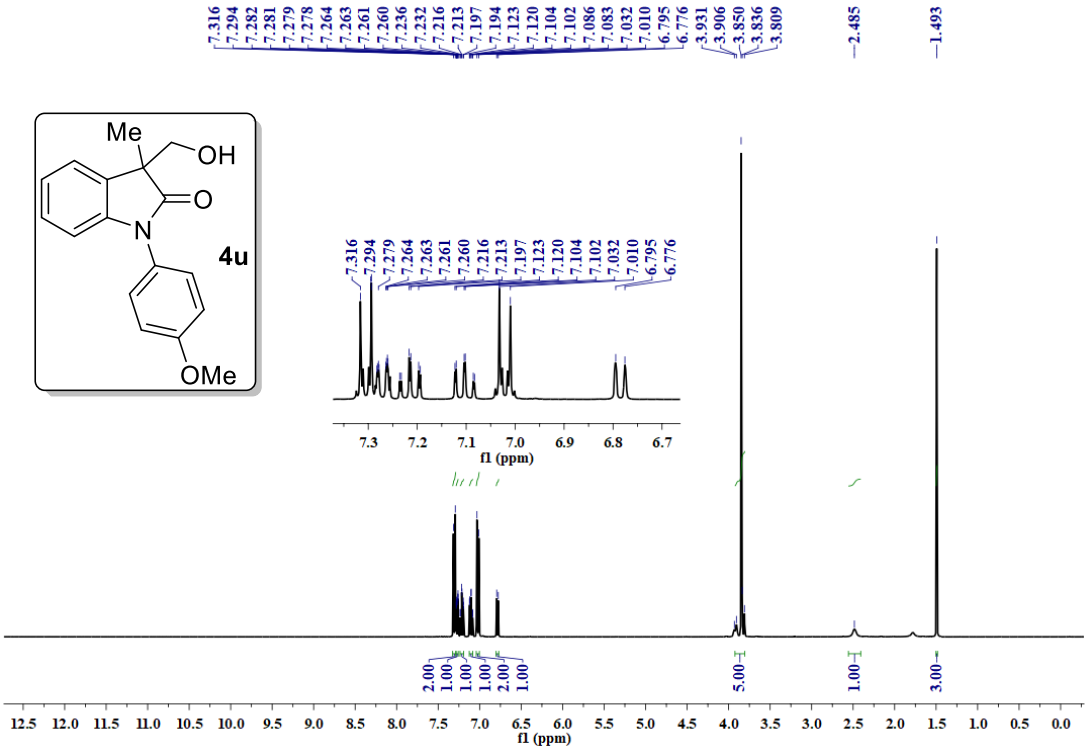


HRMS of 1-(4-ethylphenyl)-3-(hydroxymethyl)-3-methylindolin-2-one (4t)

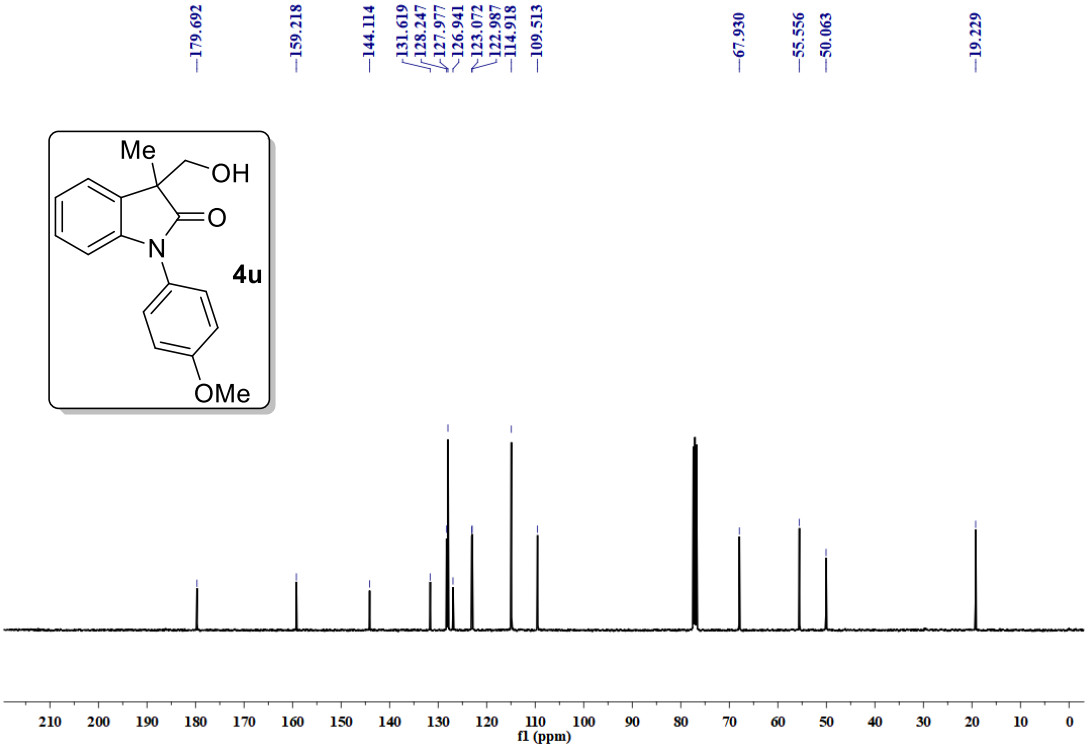
Sample Name	Nuetphoxi-2	**Position**	P1-E8	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-44.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 8:08:04 PM



^{1}H NMR (400 MHz, $CDCl_3$) spectrum of 3-(hydroxymethyl)-1-(4-methoxyphenyl)-3-methylindolin-2-one (4u)

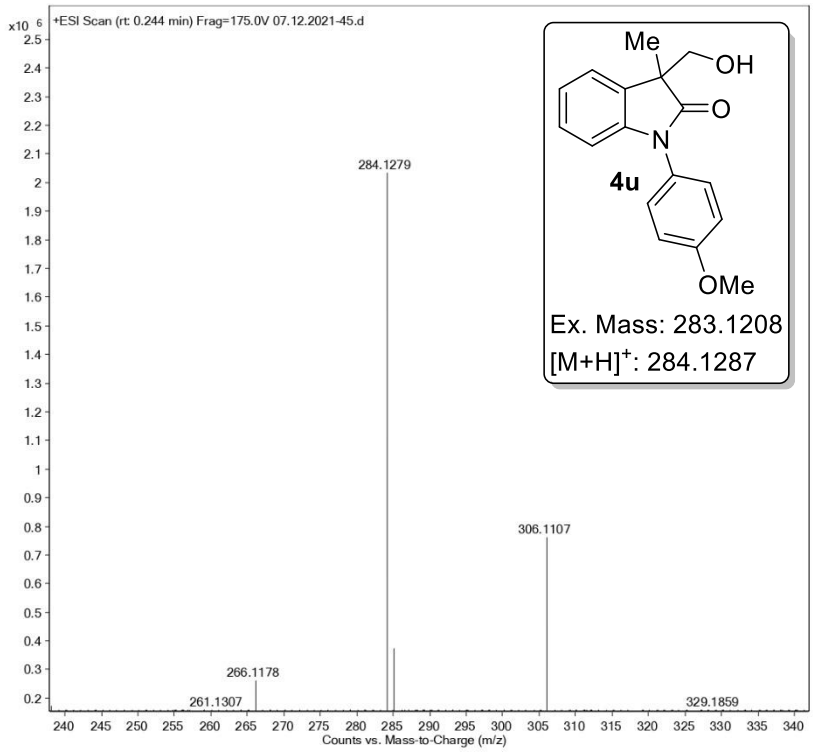


^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$) spectrum of 3-(hydroxymethyl)-1-(4-methoxyphenyl)-3-methylindolin-2-one (4u)

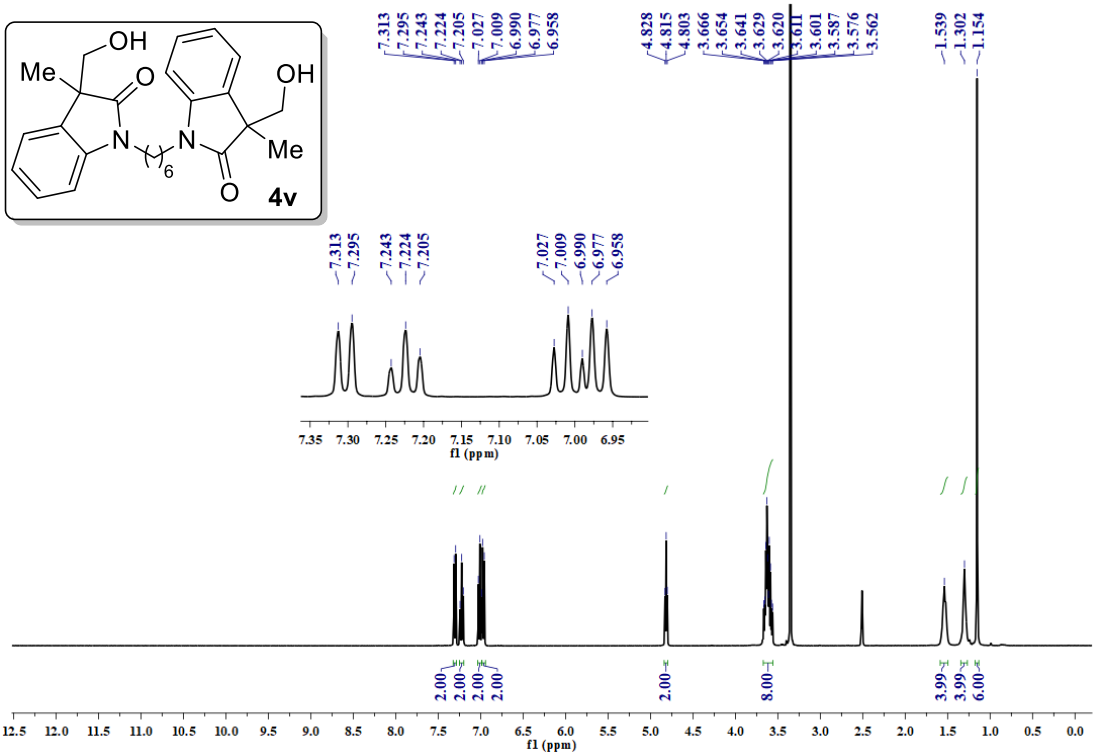


HRMS of 3-(hydroxymethyl)-1-(4-methoxyphenyl)-3-methylindolin-2-one (4u)

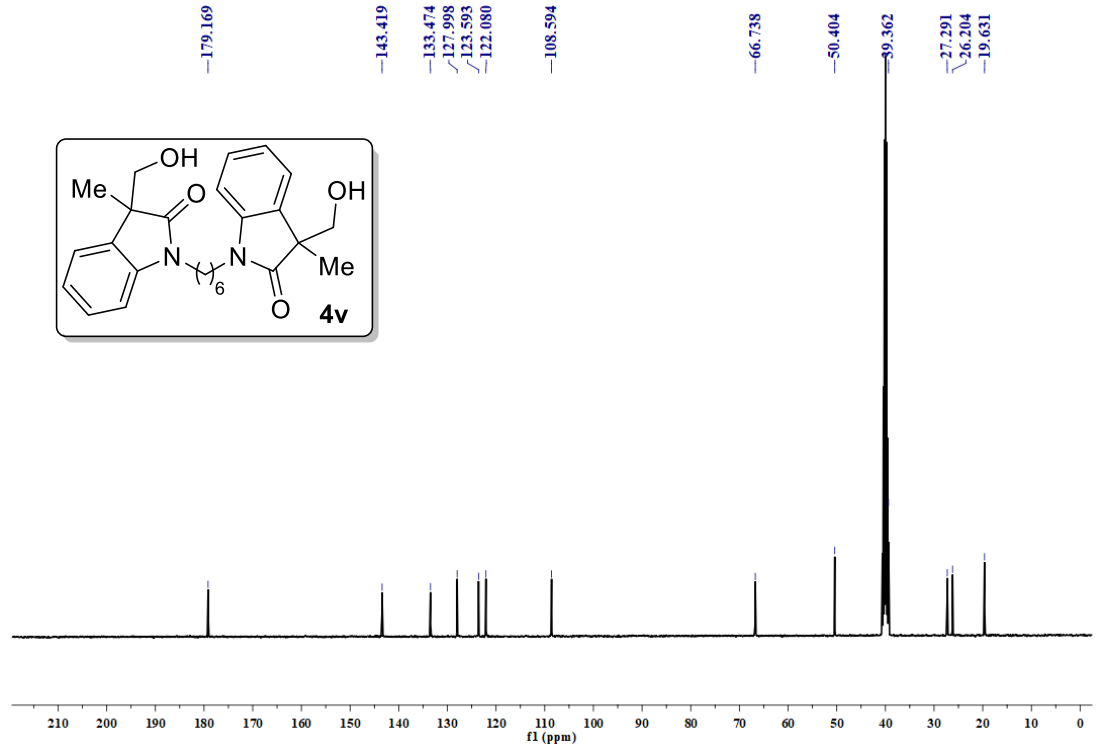
Sample Name	Nuomephoxi-2	**Position**	P1-E9	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-45.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 8:12:02 PM



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 1,1'-(hexane-1,6-diyl)bis(3-(hydroxymethyl)-3-methylindolin-2-one) (4v)

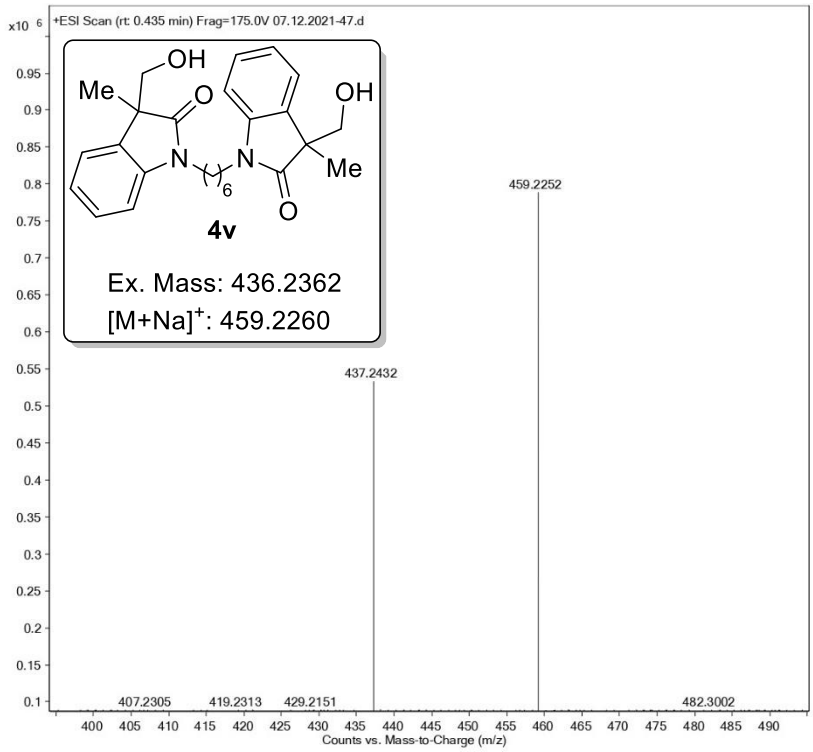


^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 1,1'-(hexane-1,6-diyl)bis(3-(hydroxymethyl)-3-methylindolin-2-one) (4v)

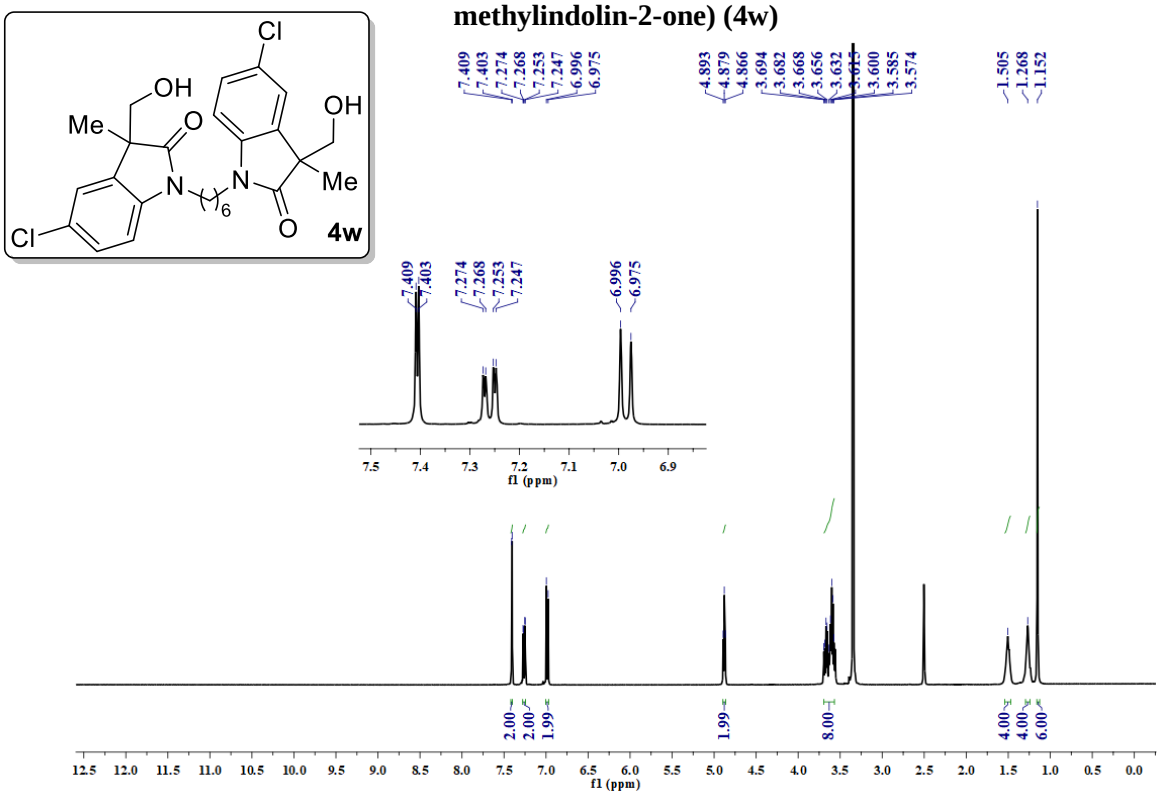


HRMS of 1,1'-(hexane-1,6-diyl)bis(3-(hydroxymethyl)-3-methylindolin-2-one) (4v)

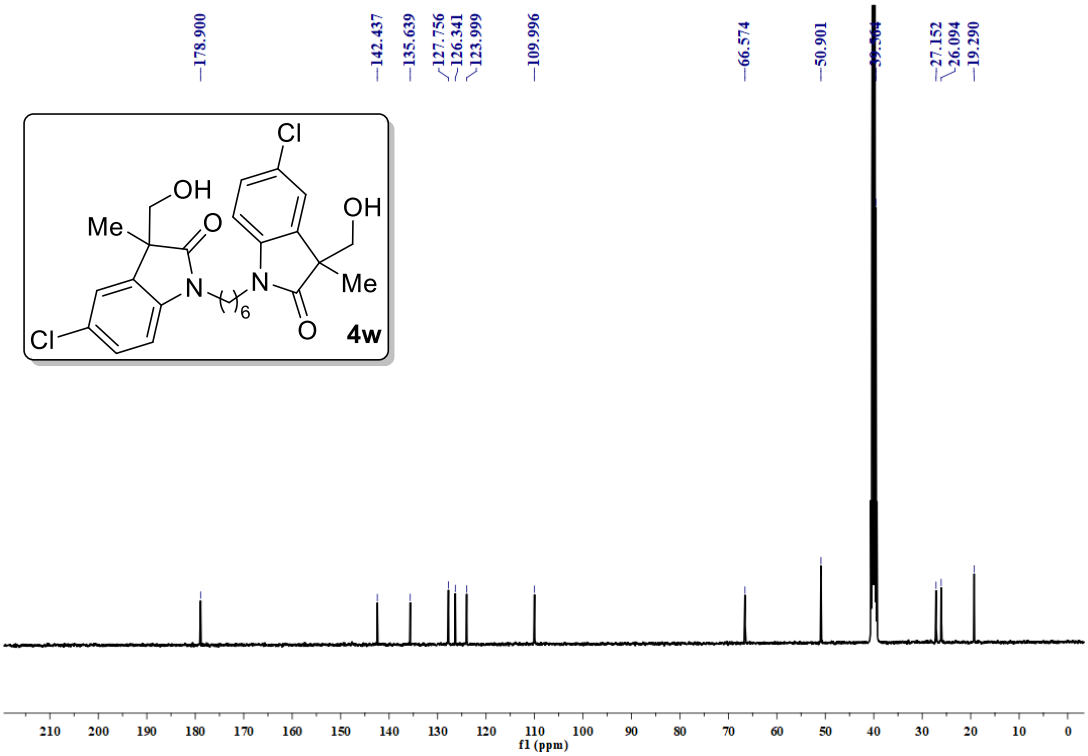
Sample Name	i-6-dimeroxi-2	**Position**	P1-F2	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-47.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 8:19:59 PM



^{1}H NMR (400 MHz, DMSO-d_6) spectrum of 1,1'-(hexane-1,6-diyl)bis(5-chloro-3-(hydroxymethyl)-3-methylindolin-2-one) (4w)



^{13}C{^{1}H} NMR (100 MHz, DMSO-d_6) spectrum of 1,1'-(hexane-1,6-diyl)bis(5-chloro-3-(hydroxymethyl)-3-methylindolin-2-one) (4w)



HRMS of 1,1'-(hexane-1,6-diyl)bis(5-chloro-3-(hydroxymethyl)-3-methylindolin-2-one) (4w)

Sample Name	5-cl -i-6-oxi-2	**Position**	P1-F3	**Instrument Name**	Instrument 1
User Name		**Inj Vol**	2	**InjPosition**	
Sample Type	Sample	**IRM Calibration Status**	Success	**Data Filename**	07.12.2021-48.d
ACQ Method	NITW-W.m	**Comment**		**Acquired Time**	07-Dec-21 8:23:58 PM