

Supporting Information

Transition Metal-Free Rongalite-Mediated Geminal C(sp³)-H Functionalization of Fluorenes

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1. General information: All chemicals and solvents were purchased from Alfa Aesar, Spectrochem, SRL, Finar and used as received. Fluorene derivatives, rongalite and potassium carbonate is purchased from Sisco Research Laboratories Pvt. Ltd. Some of the fluorene derivatives **1c-d**¹, **1f**², **1o**³, **1q-s**⁴, **1t**⁵ & **1u**⁶ were prepared by following the literature reports. The conformation of the reactions was monitored using analytical on Thin Layer Chromatography (TLC) Merck silica gel G/GF 254 plates and used UV-Cabinet for visualization of compound spots on TLC plate. Purification of compounds using column chromatography was performed with the Rankem silica gel (60-120 mesh. Finding the melting points of solid compounds was determined by open capillaries using Stuart SMP30 melting point apparatus and is uncorrected. NMR (¹H, ¹³C and ¹⁹F) spectra of all the synthesized compounds were recorded on a Bruker AVANCE HD (400 MHz, 100 MHz, and 376 MHz) spectrometer using CDCl₃ and DMSO-*d*₆ as solvents and TMS as an internal standard. The data of the compounds was recorded as chemical shifts (δ ppm) (multiplicity, coupling constant (Hz), integration). Abbreviations for the multiplicity as follows: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet, dd = doublet of doublet. The mass spectrum analysis was recorded in Bruker- micro-TOF MS analyser 2.

2. General Procedures

2.1 General Procedure for synthesis of (9-methyl-9H-fluoren-9-yl) methanol (3a-3u). An oven dried 10 mL reaction flask equipped with a magnetic stirring bar was charged with appropriate fluorene derivatives **1** (0.6 mmol), rongalite **2** (2.1 mmol), K₂CO₃ (1.8 mmol) and DMSO (2 mL). The mixture was stirred at 100 °C for the appropriate time (6 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 (Na₂SO₄) and evaporated to give a residue that was purified on a short pad of silica gel by column chromatography using ethyl acetate and hexanes (10/90) as an eluent.

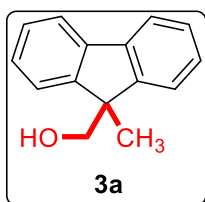
2.2 Procedure for synthesis of (9-methyl-9H-fluoren-9-yl)methyl 2-(4-isobutylphenyl)propanoate (4).⁷ To the oven-dried round bottom flask equipped with a magnetic stir bar were added 9-Methyl-9H-fluoren-9-yl)methanol (100 mg, 0.47 mmol), ibuprofen (147 mg, 0.71 mmol), *N,N*-dimethylaminopyridine (DMAP) (116 mg, 0.95 mmol), dicyclohexylcarbodiimide (DCC) (117 mg, 0.57 mmol, 1.2 equiv.) and 2 mL of CH₂Cl₂. The

resulting reaction mixture was stirred at room temperature. The progress of the reaction was monitored by TLC. After the completion of the reaction (3 h), the reaction mixture was washed with water and extracted with EtOAc. The combined organic layers were washed with brine, dried with anhydrous Na₂SO₄, and concentrated under a vacuum. The crude product was purified by silica gel column chromatography (eluent: petroleum ether/EtOAc = 9.5:0.5, v/v) to afford 4-acetylphenyl 2-(4-isobutylphenyl) propanoate 4 as a white solid 108 mg, 89% yield.

2.3 Procedure for synthesis of 9H-fluorene-9-carbaldehyde (5).⁸ To a solution of (9-methylfluoren-9-yl)methanol (100 mg, 0.47 mmol) in CH₂Cl₂ (2 mL) was added PCC (204 mg, 0.95 mmol) at 0 °C. The reaction progress was monitored by TLC. Upon completion (60 min), the solvent was removed under reduced pressure, the crude residue was purified by column chromatography on silica gel (petroleum ether/ethyl acetate, 95:5) to afford the desired product as a white solid (78 mg, 79% yield).

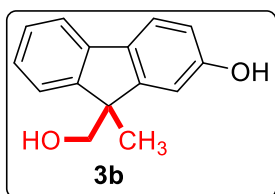
3. Spectral data of Products 3a-3u

9-Methyl-9H-fluoren-9-yl) methanol (3a). White crystalline powder; Yield: 104 mg, 82%;



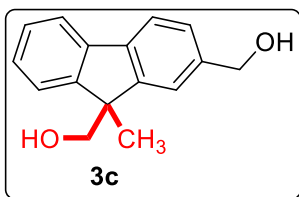
mp: 123–124 °C; The title compound was prepared according to the general procedure described above (EtOAc:hexanes = 10:90, v/v); ¹H NMR (400 MHz, DMSO-*d*₆) δ 7.82 (d, *J* = 7.2 Hz, 2H), 7.56 (d, *J* = 7.2 Hz, 2H), 7.35 (t, *J* = 7.2 Hz, 2H), 7.30 (t, *J* = 7.2 Hz, 2H), 4.99 (s, 1H), 3.54 (s, 2H), 1.45 (s, 3H); ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) δ 150.9, 140.0, 127.5, 127.4, 124.4, 120.3, 68.5, 52.4, 21.8; HRMS (ESI) *m/z*: [M+Na]⁺ calcd for C₁₅H₁₄NaO: 233.0942; found: 233.0947.

9-(Hydroxymethyl)-9-methyl-9H-fluoren-2-ol (3b). White crystalline solid; Yield: 111 mg,



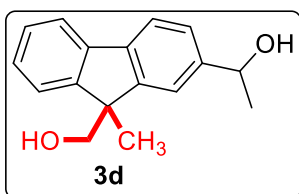
89%; mp: 157–158 °C; The title compound was prepared according to the general procedure described above (EtOAc:hexanes = 20:80); ¹H NMR (400 MHz, DMSO-*d*₆) δ 9.44 (s, 1H), 7.62 (dd, *J* = 19.8, 7.8 Hz, 2H), 7.46 (d, *J* = 7.6 Hz, 1H), 7.28 (t, *J* = 7.4 Hz, 1H), 7.17 (t, *J* = 7.2 Hz, 1H), 6.95 (d, *J* = 2.4 Hz, 1H), 6.75 (dd, *J* = 8.2, 2.4 Hz, 1H), 4.97 (t, *J* = 5.4 Hz, 1H), 3.51 (dd, *J* = 10.4, 5.2 Hz, 1H), 3.43 (dd, *J* = 10.4, 5.2 Hz, 1H), 1.39 (s, 3H); ¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) δ 157.5, 152.8, 150.1, 140.4, 131.1, 127.4, 125.7, 124.1, 121.1, 119.1, 114.6, 111.7, 68.6, 52.2, 22.0; HRMS (ESI) *m/z*: [M+Na]⁺ calcd for C₁₅H₁₄NaO₂: 249.0891; found: 249.0847.

(9-Methyl-9H-fluorene-2,9-diyl)dimethanol (3c). White crystalline solid; Yield: 104 mg,



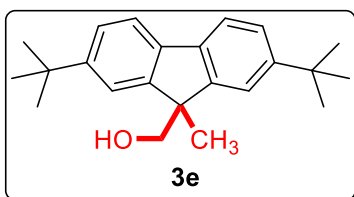
85%; mp: 152–153 °C; The title compound was prepared according to the general procedure described above (EtOAc:hexanes = 20:80); ^1H NMR (400 MHz, DMSO- d_6) δ 7.81 – 7.75 (m, 2H), 7.56 – 7.51 (m, 2H), 7.37 – 7.25 (m, 3H), 5.21 (t, J = 5.6 Hz, 1H), 4.99 (t, J = 5.2 Hz, 1H), 4.57 (d, J = 5.8 Hz, 2H), 3.52 (s, 2H), 1.44 (s, 3H); ^{13}C { ^1H } NMR (100 MHz, DMSO- d_6) δ 155.7, 155.6, 146.7, 144.8, 143.4, 132.3, 131.9, 130.8, 129.2, 127.4, 124.9, 124.7, 73.3, 68.5, 57.1, 26.7; HRMS (ESI) m/z : $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{16}\text{H}_{16}\text{NaO}_2$: 263.1048; found: 263.1055.

1-(9-(Hydroxymethyl)-9-methyl-9H-fluoren-2-yl)ethan-1-ol (3d). White crystalline solid;



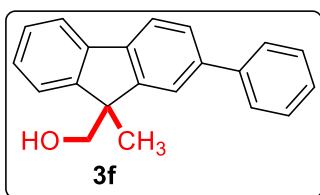
Yield: 105 mg, 87%; mp: 114–115 °C; The title compound was prepared according to the general procedure described above (EtOAc:hexanes = 20:80, v/v); ^1H NMR (400 MHz, DMSO- d_6) δ 7.77 (dd, J = 12, 8.0 Hz, 2H), 7.54 (d, J = 8.0 Hz, 2H), 7.36 – 7.32 (m, 2H), 7.27 (td, J = 7.2, 1.6 Hz, 1H), 5.17 (dd, J = 4.4, 3.2 Hz, 1H), 4.98 (q, J = 5.2 Hz, 1H), 4.82 – 4.75 (m, 1H), 3.56 – 3.48 (m, 2H), 1.44 (d, J = 1.6 Hz, 3H), 1.38 (d, J = 7.2 Hz, 3H); ^{13}C { ^1H } NMR (100 MHz, DMSO- d_6) δ 151.0, 150.7, 146.9, 140.0, 138.5, 127.5, 127.0, 124.9, 124.4, 121.3, 120.1, 119.8, 68.9, 68.6, 52.3, 26.5, 21.9; HRMS (ESI) m/z : $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{17}\text{H}_{18}\text{NaO}_2$: 277.1204; found: 277.1214.

(2,7-Di-tert-butyl-9-methyl-9H-fluoren-9-yl)methanol (3e). Pale yellow colour solid;



Yield: 63 mg, 67%; mp: 143–144 °C; The title compound was prepared according to the general procedure described above (EtOAc:hexanes = 10:90); ^1H NMR (400 MHz, DMSO- d_6) δ 7.65 (d, J = 8 Hz, 2H), 7.56 (d, J = 1.6 Hz, 2H), 7.36 (dd, J = 8, 1.8 Hz, 2H), 4.98 – 4.90 (m, 1H), 3.53 (d, J = 5.2 Hz, 2H), 1.45 (s, 3H), 1.34 (s, 18H); ^{13}C { ^1H } NMR (100 MHz, DMSO- d_6) δ 150.9, 149.6, 137.3, 123.9, 120.9, 119.4, 68.7, 52.4, 35.0, 31.8, 22.1; HRMS (ESI) m/z : $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{23}\text{H}_{30}\text{NaO}$: 345.2194; found: 345.2207.

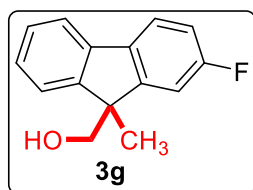
(9-Methyl-2-phenyl-9H-fluoren-9-yl)methanol (3f). White semiliquid; Yield: 94 mg, 80%;



The title compound was prepared according to the general procedure described above (EtOAc:hexanes = 10:90); ^1H NMR (400 MHz, DMSO- d_6) δ 7.91 (dd, J = 8, 0.6 Hz, 1H), 7.87 – 7.84 (m, 2H), 7.75 – 7.72 (m, 2H), 7.66 (dd, J = 8, 1.8 Hz, 1H), 7.58 (dt,

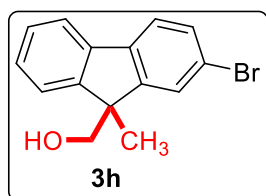
$J = 7.2, 0.8$ Hz, 1H), 7.52 – 7.47 (m, 2H), 7.38 (td, $J = 7.6, 1.2$ Hz, 2H), 7.32 (td, $J = 7.2, 1.2$ Hz, 1H), 5.03 (t, $J = 5.4$ Hz, 1H), 3.65 – 3.57 (m, 2H), 1.51 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 151.7, 151.2, 141.1, 139.7, 139.5, 139.4, 129.3, 127.7, 127.6, 127.5, 127.2, 126.3, 124.4, 122.8, 120.8, 120.5, 68.5, 52.6, 21.8; HRMS (ESI) m/z : $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{21}\text{H}_{18}\text{NaO}$: 309.1255; found: 309.1265.

(2-Fluoro-9-methyl-9H-fluoren-9-yl)methanol (3g). Pale yellow colour semiliquid; Yield:



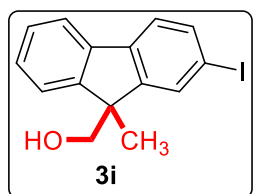
113 mg, 84%; The title compound was prepared according to the general procedure described above (EtOAc:hexanes = 10:90); ^1H NMR (400 MHz, DMSO- d_6) δ 7.87 – 7.79 (m, 2H), 7.56 – 7.52 (m, 1H), 7.41 – 7.26 (m, 3H), 7.18 (ddd, $J = 9.6, 8.2, 2.4$ Hz, 1H), 5.03 (t, $J = 5.2$ Hz, 1H), 3.61 (dd, $J = 10.4, 5.4$ Hz, 1H), 3.51 (dd, $J = 10.2, 5.6$ Hz, 1H), 1.44 (s, 3H). ^{19}F NMR (376 MHz, DMSO- d_6) δ 115.01; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 163.5 (d, $^1J_{\text{C-F}} = 240$ Hz), 153.5, (d, $^3J_{\text{C-F}} = 8$ Hz), 150.7, 139.2, 136.3, 127.7, 127.1, 124.2, 121.7, (d, $^3J_{\text{C-F}} = 8$ Hz), 120.2, 114.6, (d, $^2J_{\text{C-F}} = 23$ Hz), 112.0, (d, $^2J_{\text{C-F}} = 23$ Hz), 68.2, 52.8, 21.6; HRMS (ESI) m/z : $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{15}\text{H}_{13}\text{FNaO}$: 251.0848; found: 251.0859.

(2-Bromo-9-methyl-9H-fluoren-9-yl) methanol (3h). White powder; Yield: 102 mg, 88%;



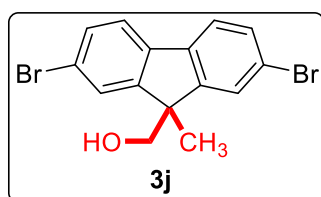
mp: 83–84 °C; The title compound was prepared according to the general procedure described above (EtOAc:hexanes = 10:90); ^1H NMR (400 MHz, DMSO- d_6) δ 7.89 – 7.72 (m, 3H), 7.54 (p, $J = 5.4$ Hz, 2H), 7.34 (h, $J = 7.4$ Hz, 2H), 5.06 (d, $J = 11.1$ Hz, 1H), 3.64 (q, $J = 8.3$ Hz, 1H), 3.53 – 3.45 (m, 1H), 1.43 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 153.4, 150.5, 139.4, 139.0, 130.4, 127.9, 127.8, 127.6, 124.3, 122.2, 120.6, 120.5, 68.1, 52.9, 21.6; HRMS (ESI) m/z : $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{15}\text{H}_{13}\text{BrNaO}$: 311.0047; found: 311.0056.

(2-Iodo-9-methyl-9H-fluoren-9-yl) methanol (3i). Pale yellow colour semiliquid; Yield: 99



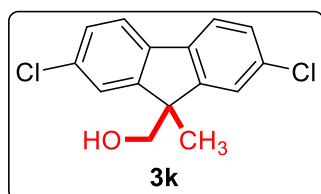
mg, 86%; The title compound was prepared according to the general procedure described above (EtOAc:hexanes = 10:90, v/v); ^1H NMR (400 MHz, DMSO- d_6) δ 7.92 (d, $J = 15.6$ Hz, 1H), 7.82 (q, $J = 6.6$ Hz, 1H), 7.74 – 7.61 (m, 2H), 7.53 (q, $J = 6.7$ Hz, 1H), 7.34 (p, $J = 6.5$ Hz, 2H), 5.06 – 4.98 (m, 1H), 3.61 (dq, $J = 10.4, 5.1$ Hz, 1H), 3.47 (t, $J = 7.2$ Hz, 1H), 1.41 (d, $J = 14.3$ Hz, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 153.4, 150.3, 139.8, 139.1, 136.2, 133.3, 128.0, 127.7, 124.2, 122.5, 120.6, 93.4, 68.1, 52.8, 40.6, 40.4, 40.1, 39.9, 39.7, 39.5, 39.3, 21.6; HRMS (ESI) m/z : $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{15}\text{H}_{13}\text{INaO}$: 358.9909; found: 358.9927.

(2,7-Dibromo-9-methyl-9H-fluoren-9-yl)methanol (3j). White crystalline solid; Yield: 98



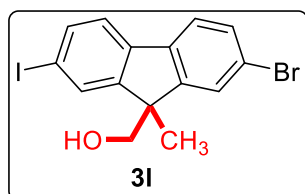
mg, 85%; mp: 171–172 °C; The title compound was prepared according to the general procedure described above (EtOAc:hexanes = 10:90, v/v); ^1H NMR (400 MHz, DMSO- d_6) δ 7.93 (d, J = 1.6 Hz, 1H), 7.79 (d, J = 8.4 Hz, 1H), 7.75 (d, J = 4 Hz, 1H), 7.72 (dd, J = 8.0, 1.6 Hz, 1H), 7.66 (d, J = 8.0 Hz, 1H), 7.54 (dd, J = 8.0, 2.0 Hz, 1H), 5.041 (t, J = 5.6 Hz, 1H), 3.59 (d, J = 5.6 Hz, 2H), 1.42 (s, 3H); ^{13}C { ^1H } NMR (100 MHz, DMSO- d_6) δ 153.1, 138.4, 130.6, 127.5, 122.5, 121.1, 67.8, 53.3, 40.4, 21.4; HRMS (ESI) m/z : $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{15}\text{H}_{12}\text{Br}_2\text{NaO}$: 388.9153; found: 388.9177.

(2,7-Dichloro-9-methyl-9H-fluoren-9-yl)methanol (3k). White crystalline solid; Yield: 108



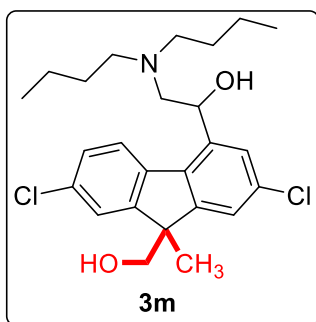
mg, 91%; mp: 163–164 °C. The title compound was prepared according to the general procedure described above (EtOAc:hexanes = 10:90, v/v); ^1H NMR (400 MHz, DMSO- d_6) δ 7.86 (d, J = 8.0 Hz, 2H), 7.63 (d, J = 2.0 Hz, 2H), 7.42 (dd, J = 8.4, 2.4 Hz, 2H), 5.05 (t, J = 5.4 Hz, 1H), 3.61 (d, J = 5.6 Hz, 2H), 1.43 (s, 3H); ^{13}C { ^1H } NMR (100 MHz, DMSO- d_6) δ 152.9, 138.0, 132.4, 127.8, 124.7, 122.1, 67.8, 53.2, 40.6, 40.4, 40.2, 39.9, 39.7, 39.5, 39.3, 21.4; HRMS (ESI) m/z : $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{15}\text{H}_{12}\text{Cl}_2\text{NaO}$: 301.0163; found: 301.0171.

(2-Bromo-7-iodo-9-methyl-9H-fluoren-9-yl)methanol (3l). Pale yellow crystalline solid;



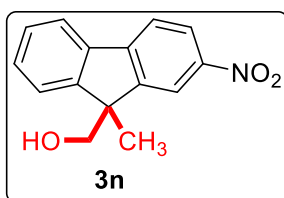
Yield: 97 mg, 86%; mp: 189–190 °C; The title compound was prepared according to the general procedure described above (EtOAc:hexanes = 10:90, v/v); ^1H NMR (400 MHz, DMSO- d_6) δ 7.93 (d, J = 1.6 Hz, 1H), 7.72 (dd, J = 8.0, 1.6 Hz, 1H), 7.66 (d, J = 8.0 Hz, 1H), 7.54 (dd, J = 8, 1.6 Hz, 1H), 5.04 (t, J = 5.2 Hz, 1H), 3.59 (d, J = 5.2 Hz, 2H), 1.42 (s, 3H); ^{13}C { ^1H } NMR (100 MHz, DMSO- d_6) δ 153.1, 138.8, 138.5, 136.5, 133.2, 130.6, 127.4, 122.8, 122.5, 121.1, 94.0, 67.8, 53.2, 21.4; HRMS (ESI) m/z : $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{15}\text{H}_{12}\text{BrINaO}$: 436.9014; found: 436.9038.

2-(Dibutylamino)-1-(2,7-dichloro-9-(hydroxymethyl)-9-methyl-9H-fluoren-4-yl)ethan-1-ol (3m). Pale yellow colour semiliquid; Yield: 72 mg, 69%; The title compound was prepared



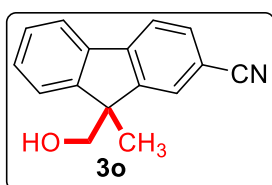
according to the general procedure described above (EtOAc:hexanes = 20:80); ^1H NMR (400 MHz, DMSO- d_6) δ 7.98 (d, J = 8.6 Hz, 1H), 7.65 (d, J = 2.2 Hz, 1H), 7.53 – 7.50 (m, 2H), 7.39 (dd, J = 8.4, 2.2 Hz, 1H), 5.29 – 5.24 (m, 1H), 5.07 (t, J = 5.4 Hz, 1H), 3.57 (dt, J = 19.8, 5.2 Hz, 2H), 2.74 (dd, J = 13.4, 4.5 Hz, 1H), 2.63 – 2.58 (m, 1H), 2.46 – 2.40 (m, 2H), 1.42 (s, 3H), 1.34 – 1.11 (m, 12H), 0.81 (t, J = 7.2 Hz, 6H); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 153.7, 142.5, 137.8, 134.4, 132.8, 132.2, 132.0, 127.4, 125.9, 125.6, 124.6, 122.8, 69.3, 68.0, 60.7, 54.0, 52.4, 29.1, 21.8, 20.4, 14.4; HRMS (ESI) m/z : $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{25}\text{H}_{32}\text{NCl}_2\text{NaO}_2$: 472.1786; found: 472.1806.

(9-Methyl-2-nitro-9H-fluoren-9-yl)methanol (3n). Pale yellow colour semiliquid; Yield: 89



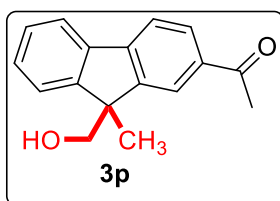
mg, 63%; The title compound was prepared according to the general procedure described above (EtOAc:hexanes = 10:90, v/v); ^1H NMR (400 MHz, DMSO- d_6) δ 7.81 (dt, J = 29.8, 8.5 Hz, 3H), 7.60 – 7.49 (m, 2H), 7.40 – 7.29 (m, 2H), 5.10 – 4.99 (m, 1H), 3.64 (dt, J = 10.6, 5.2 Hz, 1H), 3.49 (dt, J = 10.2, 4.8 Hz, 1H), 1.43 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 153.4, 150.5, 139.4, 139.0, 130.4, 127.9, 127.8, 127.6, 124.3, 122.2, 120.6, 120.5, 68.1, 52.9, 21.6; HRMS (ESI) m/z : $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{15}\text{H}_{12}\text{NNaO}_3$: 278.0793; found: 278.0809.

9-(Hydroxymethyl)-9-methyl-9H-fluorene-2-carbonitrile (3o). Colourless semiliquid;



Yield: 110 mg, 89%; The title compound was prepared according to the general procedure described above (EtOAc:hexanes = 10:90); ^1H NMR (400 MHz, DMSO- d_6) δ 8.06 – 8.03 (m, 2H), 7.99 – 7.95 (m, 1H), 7.84 (dd, J = 7.9, 1.5 Hz, 1H), 7.65 – 7.60 (m, 1H), 7.45 – 7.41 (m, 2H), 5.08 (t, J = 5.4 Hz, 1H), 3.71 (dd, J = 10.4, 5.4 Hz, 1H), 3.58 (dd, J = 10.4, 5.5 Hz, 1H), 1.48 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 151.6, 144.9, 138.4, 132.1, 129.2, 128.2, 128.0, 124.4, 121.7, 121.3, 120.1, 109.3, 67.9, 53.0, 21.3; HRMS (ESI) m/z : $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{16}\text{H}_{13}\text{NNaO}$: 258.0895; found: 258.0918.

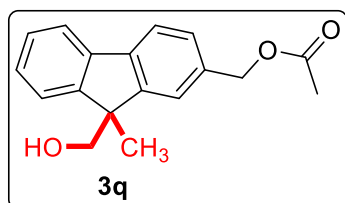
1-(9-(Hydroxymethyl)-9-methyl-9H-fluoren-2-yl) ethan-1-one (3p). White crystalline solid;



Yield: 109 mg, 90%; mp: 93–94 °C; The title compound was prepared according to the general procedure described above (EtOAc:hexanes = 10:90, v/v); ^1H NMR (400 MHz, DMSO- d_6) δ 8.15 (d, J = 1.6 Hz, 1H), 8.01 (dd, J = 7.8, 1.6 Hz, 1H), 7.97 – 7.90 (m, 2H), 7.64 – 7.56

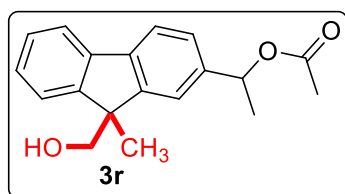
(m, 1H), 7.44 – 7.36 (m, 2H), 5.04 (t, $J = 5.6$ Hz, 1H), 3.67 (dd, $J = 10.4, 5.6$ Hz, 1H), 3.57 (dd, $J = 10.2, 5.4$ Hz, 1H), 2.63 (s, 3H), 1.49 (s, 3H).); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 198.1, 152.1, 151.1, 144.8, 138.9, 136.0, 128.7, 128.6, 127.8, 124.5, 124.0, 121.5, 120.3, 68.3, 52.7, 40.6, 40.4, 40.1, 39.9, 39.7, 39.5, 39.3, 27.3, 21.6; HRMS (ESI) m/z : $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{17}\text{H}_{16}\text{NaO}_2$: 275.1048; found: 275.1054.

(9-(Hydroxymethyl)-9-methyl-9H-fluoren-2-yl)methyl acetate (3q). Colourless



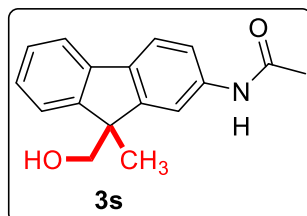
semiliquid; Yield: 100 mg, 83%; The title compound was prepared according to the general procedure described above (EtOAc:hexanes = 10:90); ^1H NMR (400 MHz, DMSO- d_6) δ 7.60 – 7.56 (m, 2H), 7.34 – 7.30 (m, 2H), 7.12 (td, $J = 7.4, 1.6$ Hz, 2H), 7.06 (td, $J = 7.3, 1.5$ Hz, 1H), 4.89 (s, 2H), 4.80 – 4.75 (m, 1H), 3.36 – 3.27 (m, 2H), 1.85 (s, 3H), 1.21 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 170.8, 151.1, 140.0, 139.6, 135.2, 127.8, 127.6, 127.6, 124.4, 124.4, 120.5, 120.3, 68.4, 66.3, 52.5, 21.8, 21.2; HRMS (ESI) m/z : $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{18}\text{H}_{18}\text{NaO}_3$: 305.1154; found: 305.1179.

1-(9-(Hydroxymethyl)-9-methyl-9H-fluoren-2-yl)ethyl acetate (3r). Colourless semiliquid;



Yield: 102 mg, 87%; The title compound was prepared according to the general procedure described above (EtOAc:hexanes = 10:90); ^1H NMR (400 MHz, DMSO- d_6) δ 7.59 – 7.54 (m, 2H), 7.33 – 7.29 (m, 2H), 7.08 (dtd, $J = 22.4, 7.3, 1.2$ Hz, 3H), 5.64 (qd, $J = 6.6, 2.3$ Hz, 1H), 4.77 (td, $J = 5.3, 2.8$ Hz, 1H), 3.34 – 3.26 (m, 2H), 1.82 (d, $J = 2.2$ Hz, 3H), 1.28 (dd, $J = 6.6, 0.9$ Hz, 3H), 1.21 (d, $J = 2.4$ Hz, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 170.2, 151.1, 140.8, 139.7, 139.6, 127.6, 127.4, 125.5, 124.4, 122.1, 122.0, 120.4, 120.2, 72.4, 68.4, 52.5, 22.7, 21.8, 21.5; HRMS (ESI) m/z : $[\text{M}+\text{Na}]^+$ calcd for $\text{C}_{19}\text{H}_{20}\text{NaO}_3$: 319.1310; found: 319.1326.

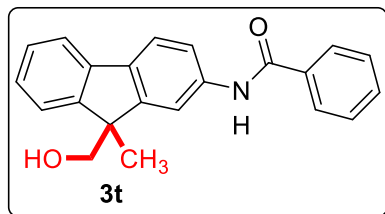
N-(9-(Hydroxymethyl)-9-methyl-9H-fluoren-2-yl)acetamide (3s). White crystalline solid;



Yield: 79 mg, 79%; mp: 182–183 °C; The title compound was prepared according to the general procedure described above (EtOAc:hexanes = 20:80); ^1H NMR (400 MHz, DMSO- d_6) δ 10.01 (s, 1H), 7.77 – 7.71 (m, 3H), 7.64 – 7.60 (m, 1H), 7.51 (d, $J = 7.3$ Hz, 1H), 7.32 (td, $J = 7.5, 1.3$ Hz, 1H), 7.24 (td, $J = 7.5, 1.3$ Hz, 1H), 5.01 (s, 1H), 3.54 (dd, $J = 10.3, 5.2$ Hz, 1H), 3.45 (dd, $J = 10.3, 5.4$ Hz, 1H), 2.07 (s, 3H), 1.42 (s, 3H); $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 168.6, 151.5, 150.5, 140.0, 139.0, 134.9, 127.5,

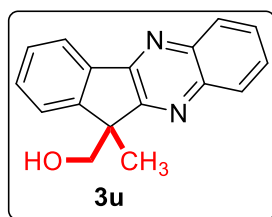
126.6, 124.2, 120.4, 119.7, 118.5, 115.3, 68.6, 52.4, 24.5, 21.9; HRMS (ESI) m/z : $[M+Na]^+$ calcd for $C_{17}H_{17}NNaO_2$: 290.1157; found: 290.1170.

N-(9-(Hydroxymethyl)-9-methyl-9H-fluoren-2-yl)benzamide (3t). White crystalline solid;



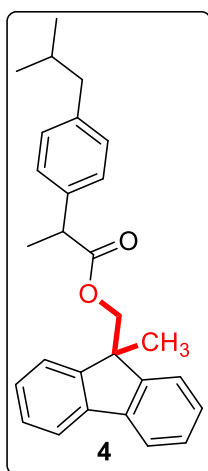
Yield: 93 mg, 81%; mp: 161–162 °C; The title compound was prepared according to the general procedure described above (EtOAc:hexanes = 20:80); 1H NMR (400 MHz, DMSO- d_6) δ 10.35 (s, 1H), 8.03 – 7.99 (m, 3H), 7.83 – 7.76 (m, 3H), 7.61 – 7.53 (m, 4H), 7.34 (td, J = 7.5, 1.2 Hz, 1H), 7.26 (td, J = 7.5, 1.3 Hz, 1H), 5.06 (t, J = 5.3 Hz, 1H), 3.60 – 3.49 (m, 2H), 1.46 (s, 3H); ^{13}C $\{^1H\}$ NMR (100 MHz, DMSO- d_6) δ 165.8, 151.4, 150.7, 139.9, 138.7, 135.6, 135.4, 132.0, 128.8, 128.1, 127.6, 126.8, 124.3, 120.4, 119.9, 116.7, 68.6, 52.5, 21.9; HRMS (ESI) m/z : $[M+Na]^+$ calcd for $C_{22}H_{19}NNaO_2$: 352.1313; found: 352.1329.

(11-Methyl-11H-indeno[1,2-b]quinoxalin-11-yl)methanol (3u). White crystalline solid;



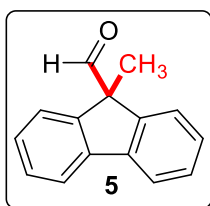
Yield: 97 mg, 81%; mp: 128–129 °C; The title compound was prepared according to the general procedure described above (EtOAc:hexanes = 10:90); 1H NMR (400 MHz, DMSO- d_6) δ 8.17 – 8.10 (m, 3H), 7.84 – 7.77 (m, 3H), 7.64 (td, J = 7.5, 1.3 Hz, 1H), 7.55 (td, J = 7.4, 1.2 Hz, 1H), 4.79 (t, J = 5.4 Hz, 1H), 4.03 (dd, J = 10.2, 5.4 Hz, 1H), 3.96 (dd, J = 10.3, 5.4 Hz, 1H), 1.52 (s, 3H); ^{13}C $\{^1H\}$ NMR (100 MHz, DMSO- d_6) δ 165.5, 154.6, 152.3, 142.2, 141.3, 136.8, 132.1, 129.8, 129.3, 129.3, 129.3, 128.6, 125.0, 122.2, 67.9, 51.1, 20.7; HRMS (ESI) m/z : $[M+Na]^+$ calcd for $C_{17}H_{14}N_2NaO$: 285.1004; found: 285.1023.

(9-methyl-9H-fluoren-9-yl)methyl 2-(4-isobutylphenyl) propanoate (4). White semiliquid;



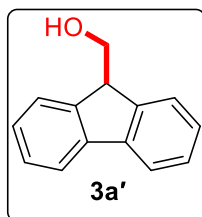
yield: 108 mg, 89%; The title compound was prepared according to the general procedure 4 described above (EtOAc:hexanes = 10:90). 1H NMR (400 MHz, DMSO- d_6) δ 7.79 – 7.69 (m, 2H), 7.34 – 7.23 (m, 4H), 7.21 – 7.17 (m, 1H), 7.15 – 7.10 (m, 1H), 7.05 – 6.96 (m, 4H), 4.21 – 4.15 (m, 1H), 4.04 (dt, J = 10.6, 2.1 Hz, 1H), 3.65 – 3.59 (m, 1H), 2.34 (d, J = 8.3 Hz, 2H), 1.73 (dt, J = 13.7, 6.3 Hz, 1H), 1.29 (d, J = 1.2 Hz, 3H), 1.21 (dt, J = 7.2, 2.1 Hz, 3H), 0.76 (dt, J = 6.7, 2.1 Hz, 6H); ^{13}C $\{^1H\}$ NMR (100 MHz, DMSO- d_6) δ 173.9, 149.1, 149.0, 140.2, 139.9, 138.0, 129.4, 128.1, 128.0, 127.6, 127.6, 124.2, 124.1, 120.5, 69.9, 50.1, 44.7, 44.7, 30.0, 22.5, 21.5, 18.2; HRMS (ESI) m/z : $[M+NH_4]^+$ calcd for $C_{28}H_{34}NO_2$: 416.2590; found: 416.2600.

9H-fluorene-9-carbaldehyde (5). Colourless semiliquid; yield: 86 mg, 79%; The title



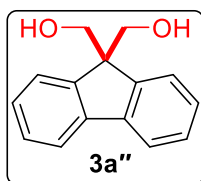
compound was prepared according to the general procedure 5 described above (EtOAc:hexanes = 10:90); ^1H NMR (400 MHz, DMSO- d_6) δ 8.93, 7.98, 7.98, 7.98, 7.97, 7.96, 7.96, 7.96, 7.53, 7.52, 7.52, 7.52, 7.51, 7.50, 7.50, 7.50, 7.50, 7.48, 7.48, 7.48, 1.62; $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 197.9, 144.2, 141.5, 129.2, 128.4, 125.1, 121.1, 62.9, 17.7.

(9H-fluoren-9-yl)methanol (3a') White crystalline powder; mp: 106–108 °C; The title



compound was prepared according to the general procedure described above (EtOAc:hexanes = 10:90, v/v); ^1H NMR (400 MHz, DMSO- d_6) δ 7.86 (dt, J = 7.5, 1.0 Hz, 2H), 7.67 (dq, J = 7.4, 0.9 Hz, 2H), 7.39 (tt, J = 7.5, 1.0 Hz, 2H), 7.31 (td, J = 7.4, 1.2 Hz, 2H), 5.08 (t, J = 5.1 Hz, 1H), 4.01 (t, J = 7.0 Hz, 1H), 3.74 (dd, J = 7.0, 5.1 Hz, 2H). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 145.6, 141.1, 127.6, 127.2, 125.7, 120.3, 64.2, 50.5.

(9H-fluorene-9,9-diyl)dimethanol (3a'') White crystalline powder; mp: 146–148 °C; The title

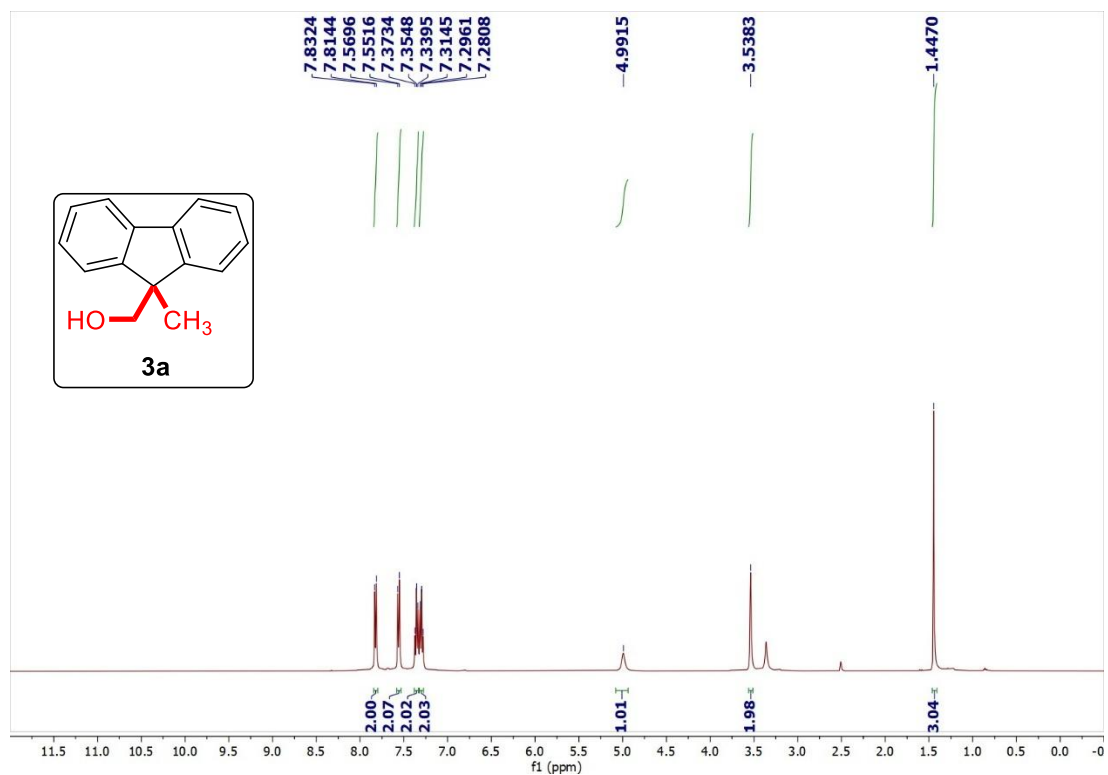


compound was prepared according to the general procedure described above (EtOAc:hexanes = 10:90, v/v); ^1H NMR (400 MHz, DMSO- d_6) δ 7.83 (d, J = 7.5 Hz, 2H), 7.60 (d, J = 7.5 Hz, 2H), 7.37 (td, J = 7.4, 1.2 Hz, 2H), 7.28 (td, J = 7.4, 1.2 Hz, 2H), 4.85 (t, J = 5.3 Hz, 2H), 3.71 (d, J = 5.3 Hz, 4H). $^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, DMSO- d_6) δ 148.03, 140.88, 127.67, 127.09, 125.63, 120.19, 64.24, 57.97.

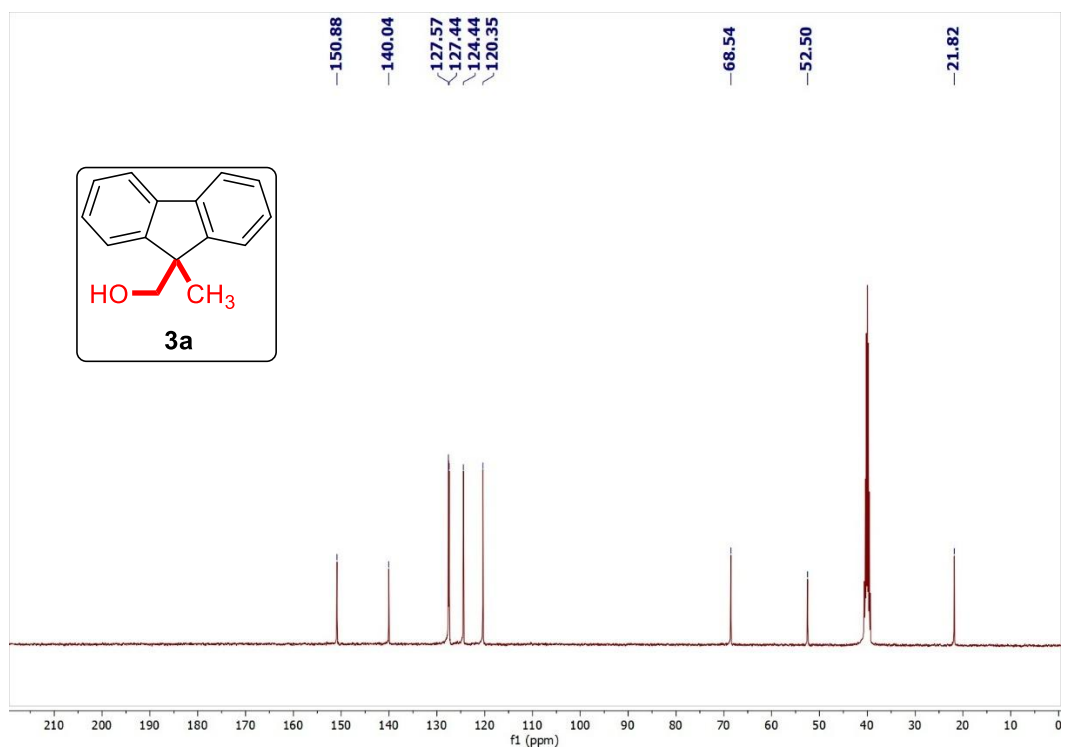
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^1H NMR (400 MHz, $\text{DMSO-}d_6$) spectrum of (9-Methyl-9H-fluoren-9-yl)methanol (3a)

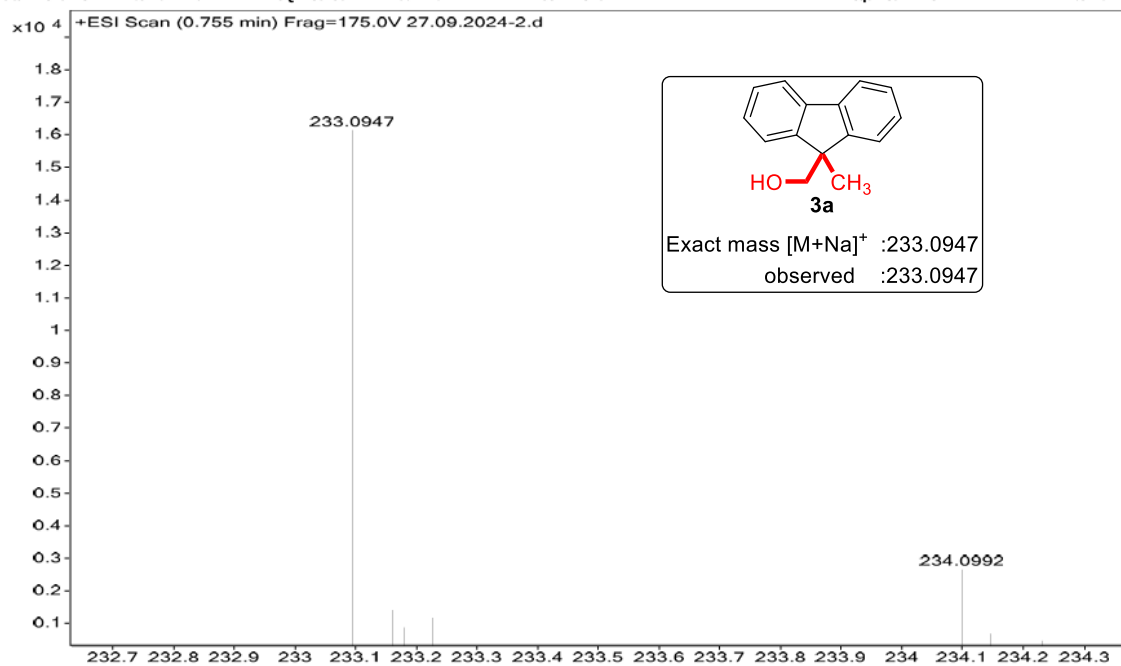


$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, $\text{DMSO-}d_6$) spectrum of (9-Methyl-9H-fluoren-9-yl)methanol (3a)

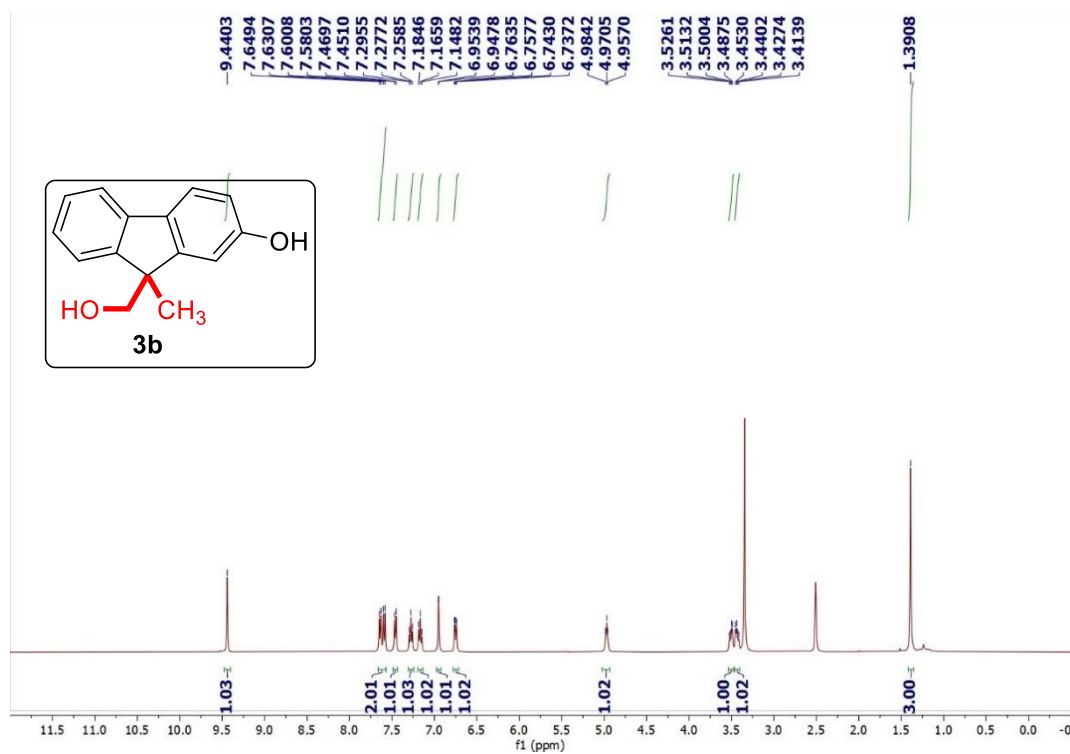


HRMS of (9-Methyl-9H-fluoren-9-yl)methanol (3a)

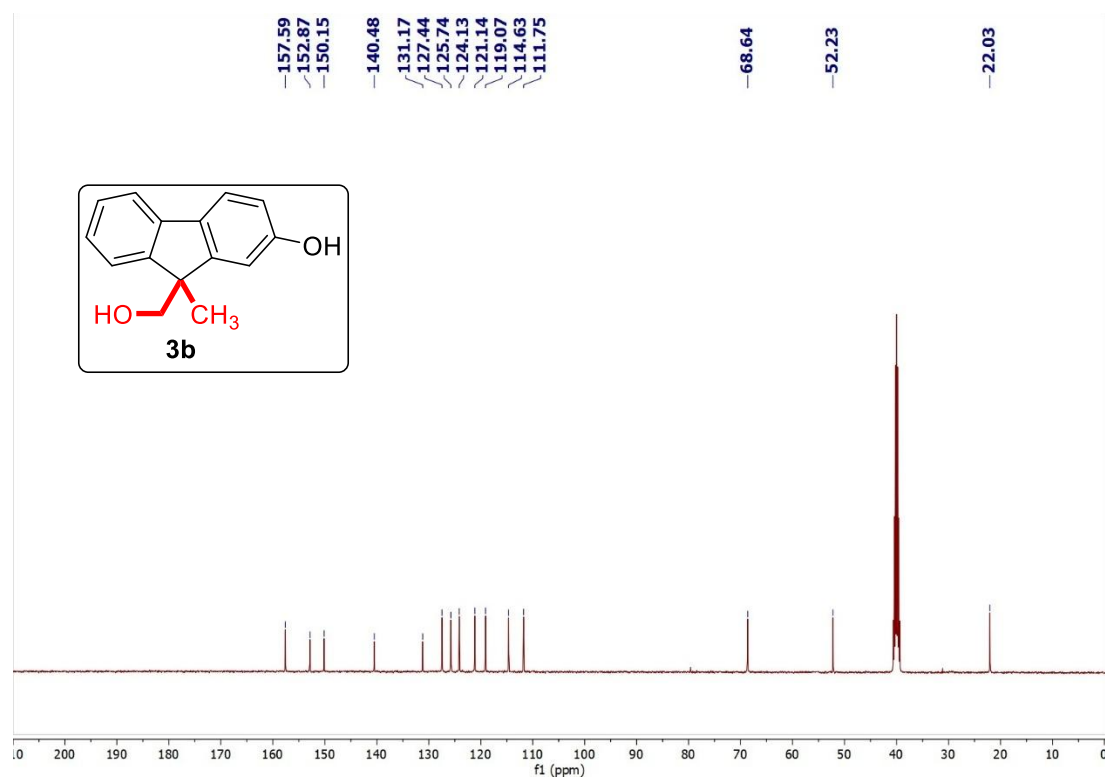
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¹H NMR (400 MHz, DMSO-*d*₆) spectrum of 9-(Hydroxymethyl)-9-methyl-9*H*-fluoren-2-ol (3b)

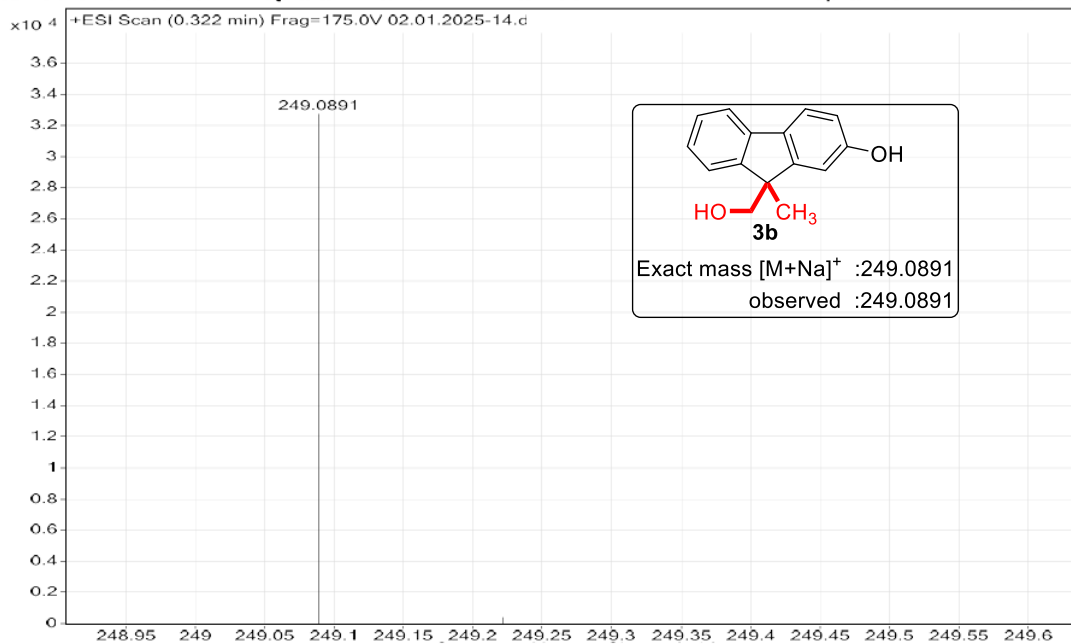


¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) spectrum of 9-(Hydroxymethyl)-9-methyl-9*H*-fluoren-2-ol (3b)

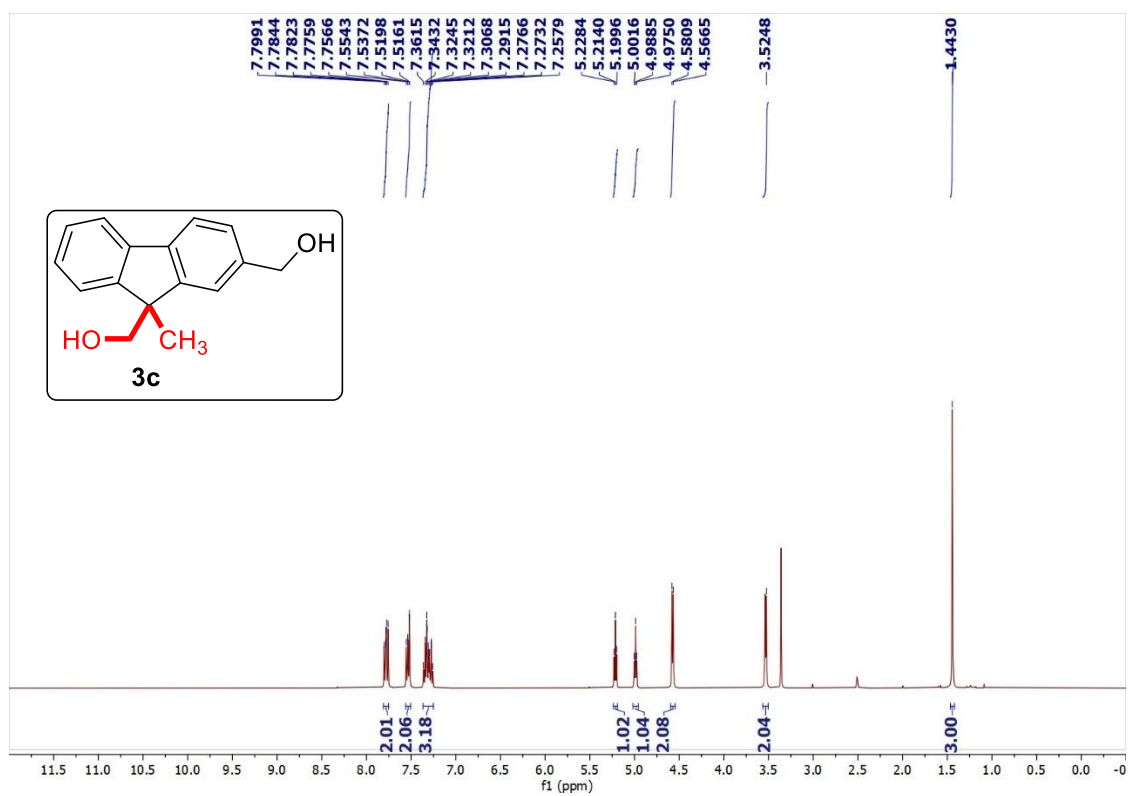


HRMS of 9-(Hydroxymethyl)-9-methyl-9H-fluoren-2-ol (3b)

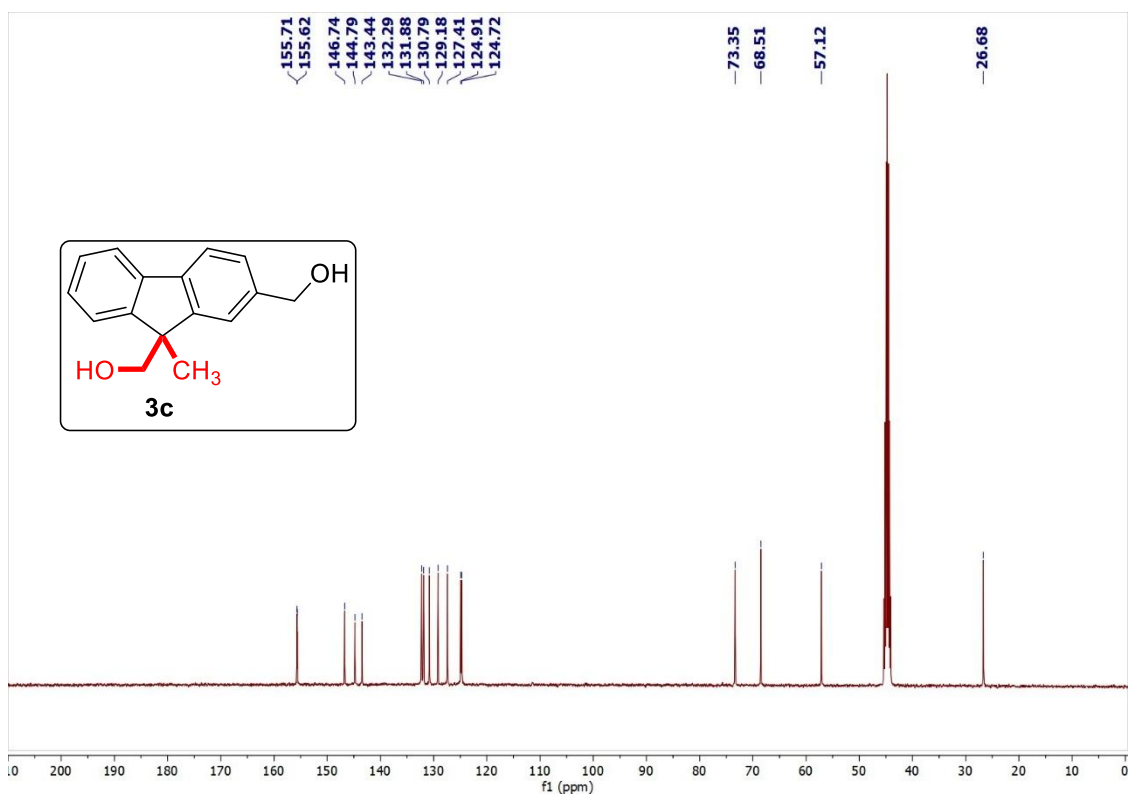
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¹H NMR (400 MHz, DMSO-*d*₆) spectrum of (9-Methyl-9*H*-fluorene-2,9-diyl)dimethanol (3c)

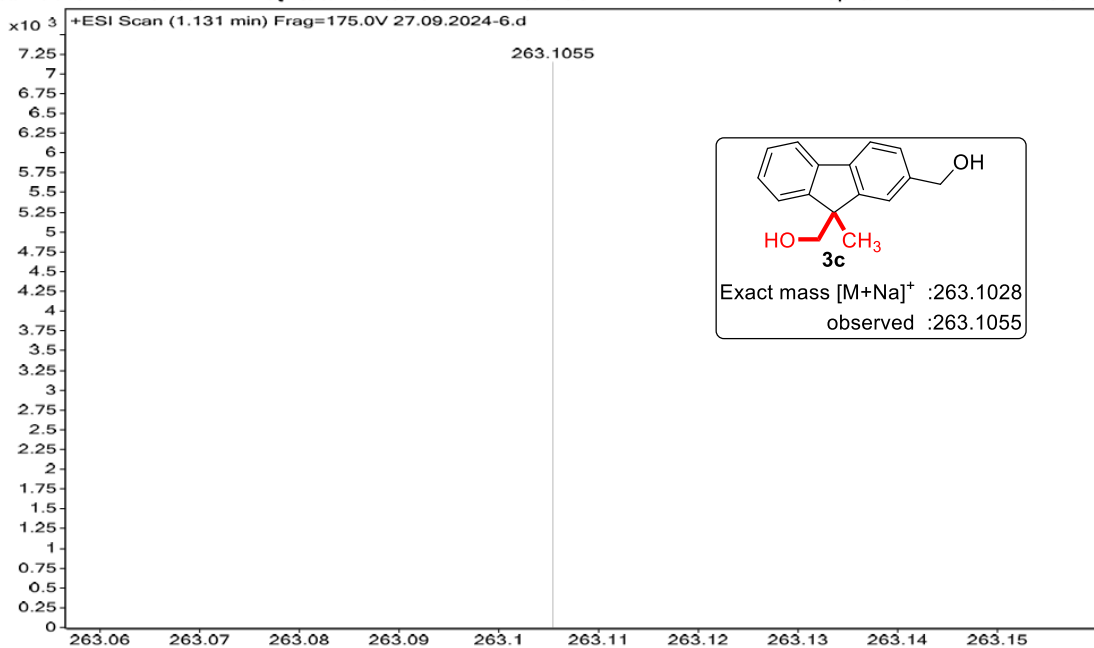


¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) spectrum of (9-Methyl-9*H*-fluorene-2,9-diyl)dimethanol (3c)

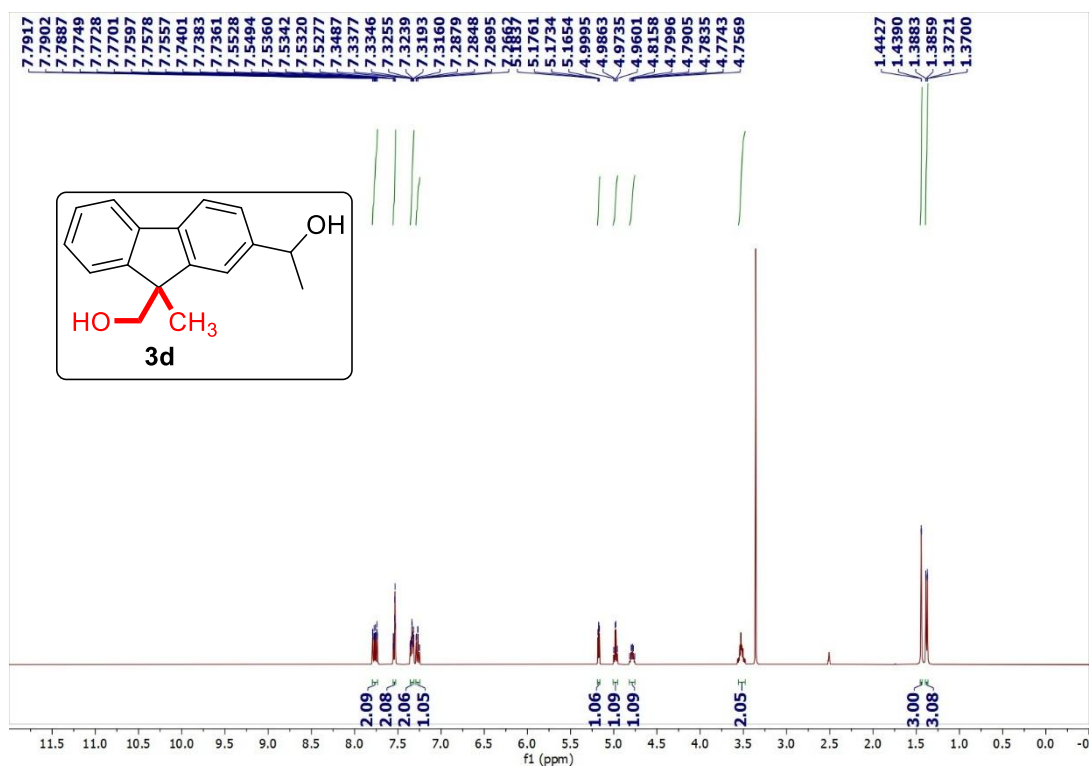


HRMS of (9-Methyl-9H-fluorene-2,9-diyl)dimethanol (3c)

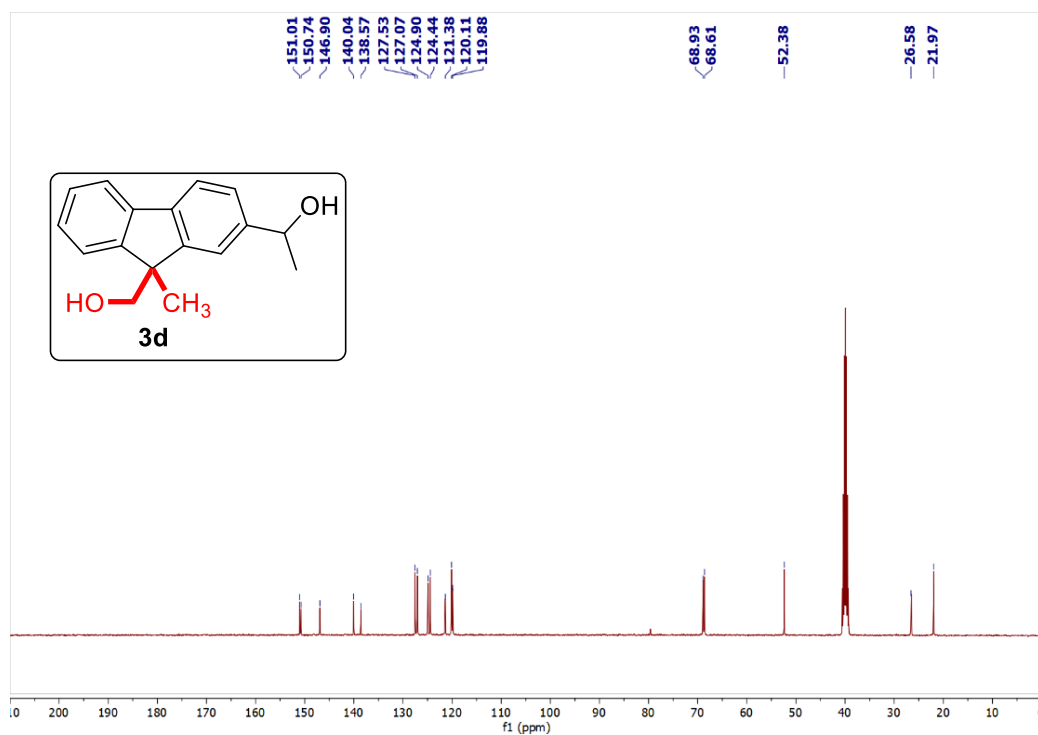
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¹H NMR (400 MHz, DMSO-*d*₆) spectrum of 1-(9-(Hydroxymethyl)-9-methyl-9*H*-fluoren-2-yl)ethan-1-ol (3d)

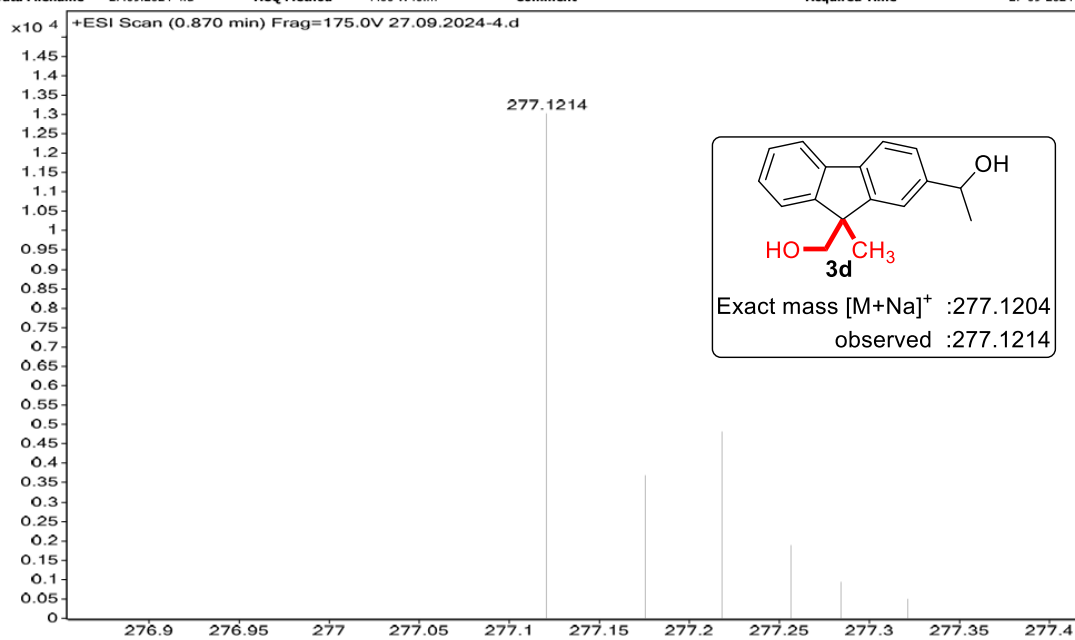


¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) spectrum of 1-(9-(Hydroxymethyl)-9-methyl-9*H*-fluoren-2-yl)ethan-1-ol (3d)

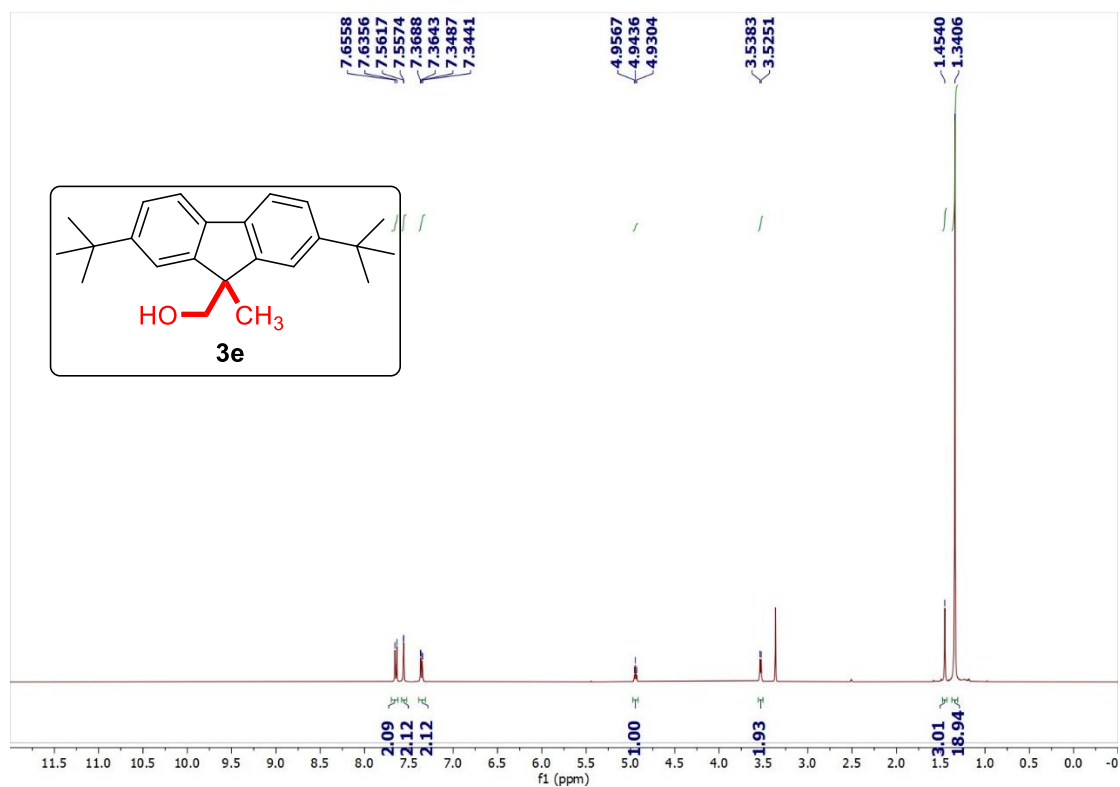


HRMS of 1-(9-(Hydroxymethyl)-9-methyl-9H-fluoren-2-yl)ethan-1-ol (3d)

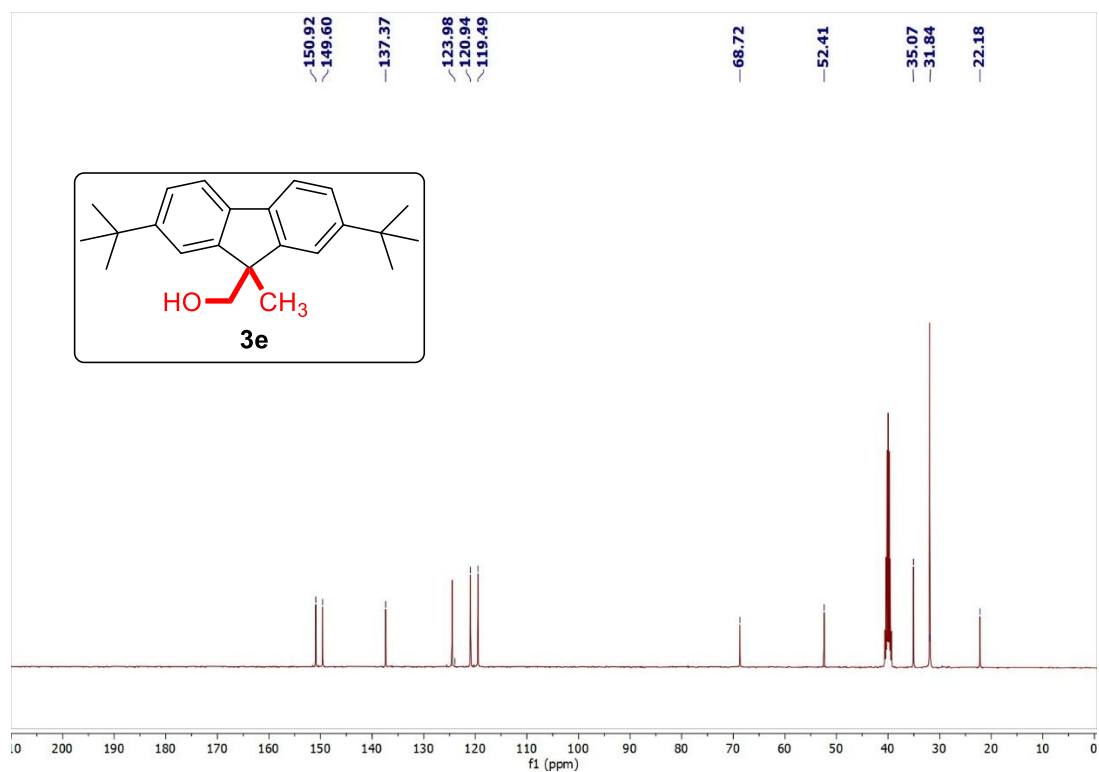
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¹H NMR (400 MHz, DMSO-*d*₆) spectrum of (2,7-Di-*tert*-butyl-9-methyl-9*H*-fluoren-9-yl)methanol (3e)

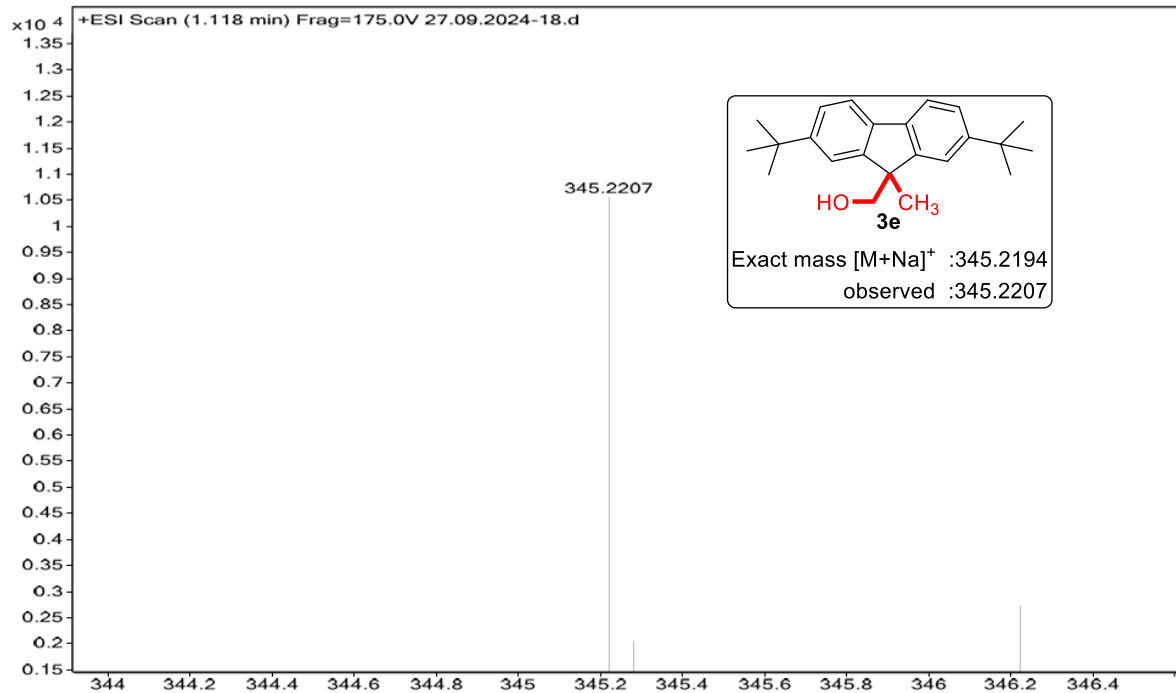


¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) spectrum of (2,7-Di-*tert*-butyl-9-methyl-9*H*-fluoren-9-yl)methanol (3e)

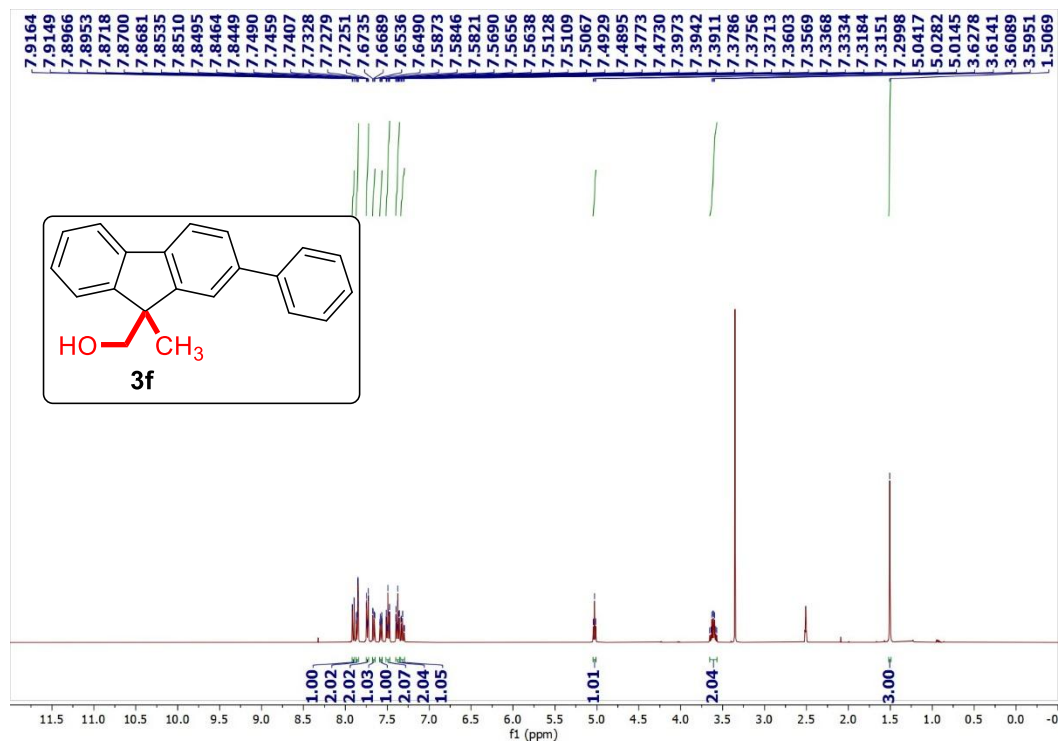


HRMS of (2,7-Di-tert-butyl-9-methyl-9H-fluoren-9-yl)methanol (3e)

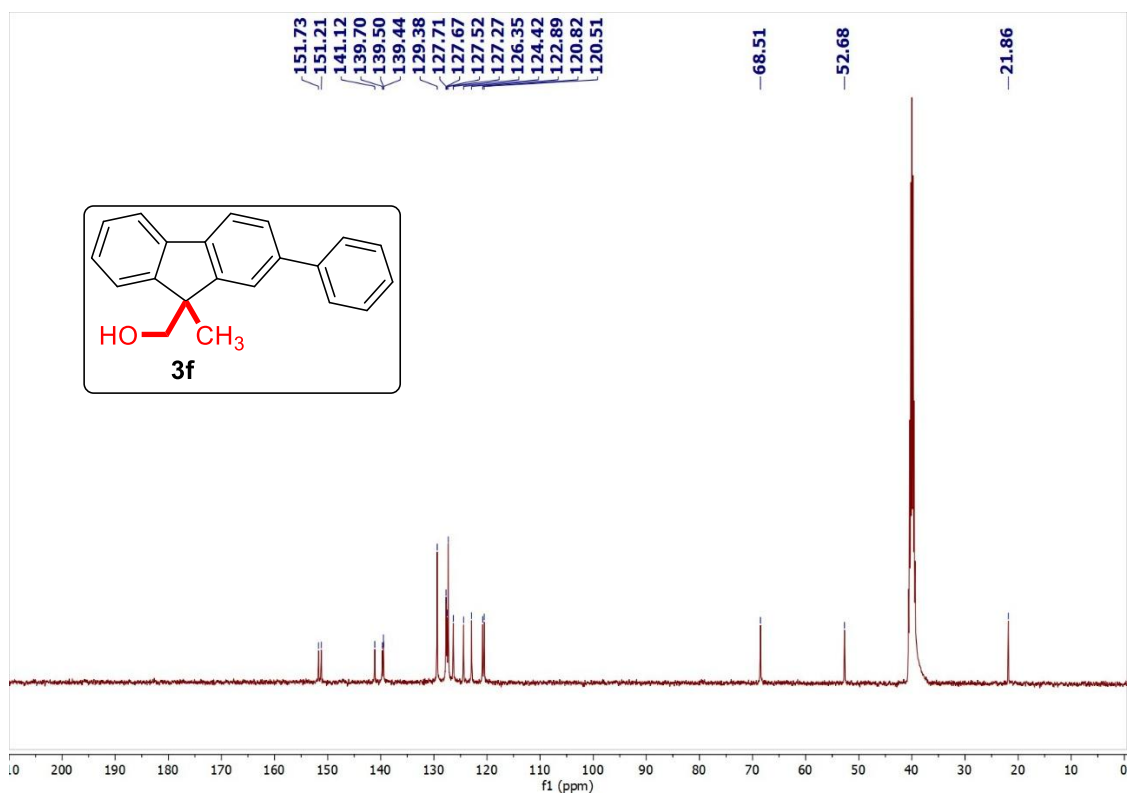
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¹H NMR (400 MHz, DMSO-*d*₆) spectrum of (9-Methyl-2-phenyl-9*H*-fluoren-9-yl)methanol (3f)

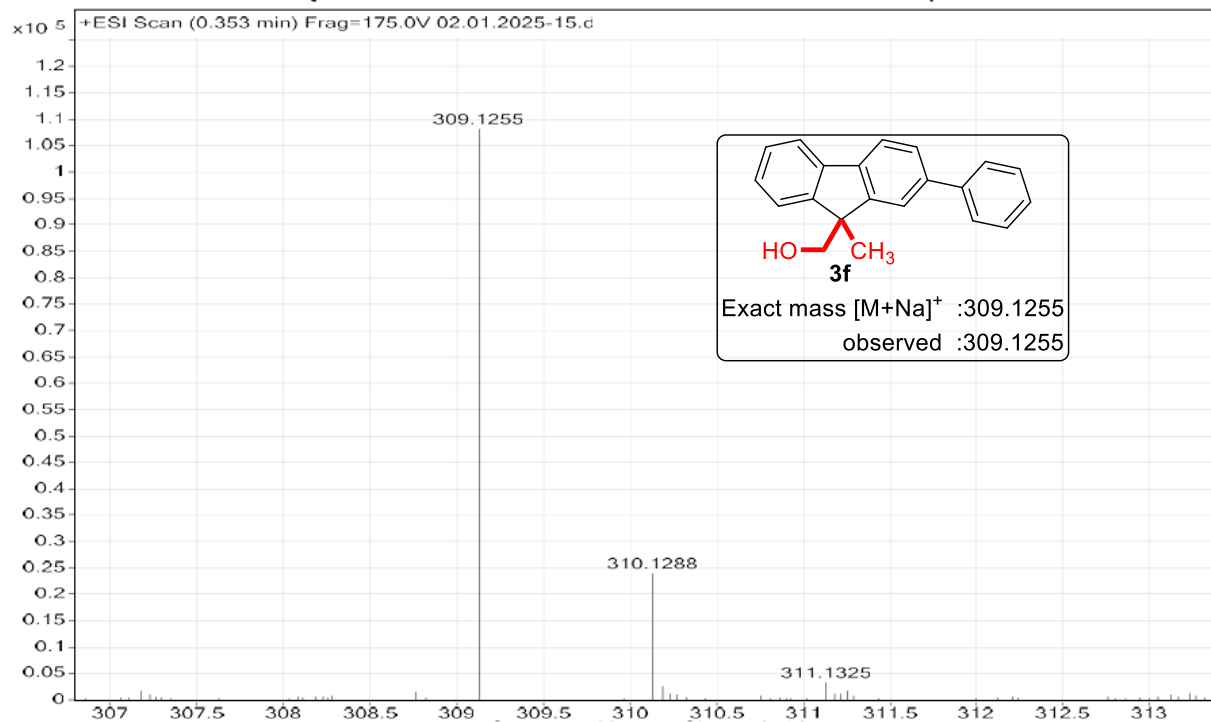


¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) spectrum of (9-Methyl-2-phenyl-9*H*-fluoren-9-yl)methanol (3f)

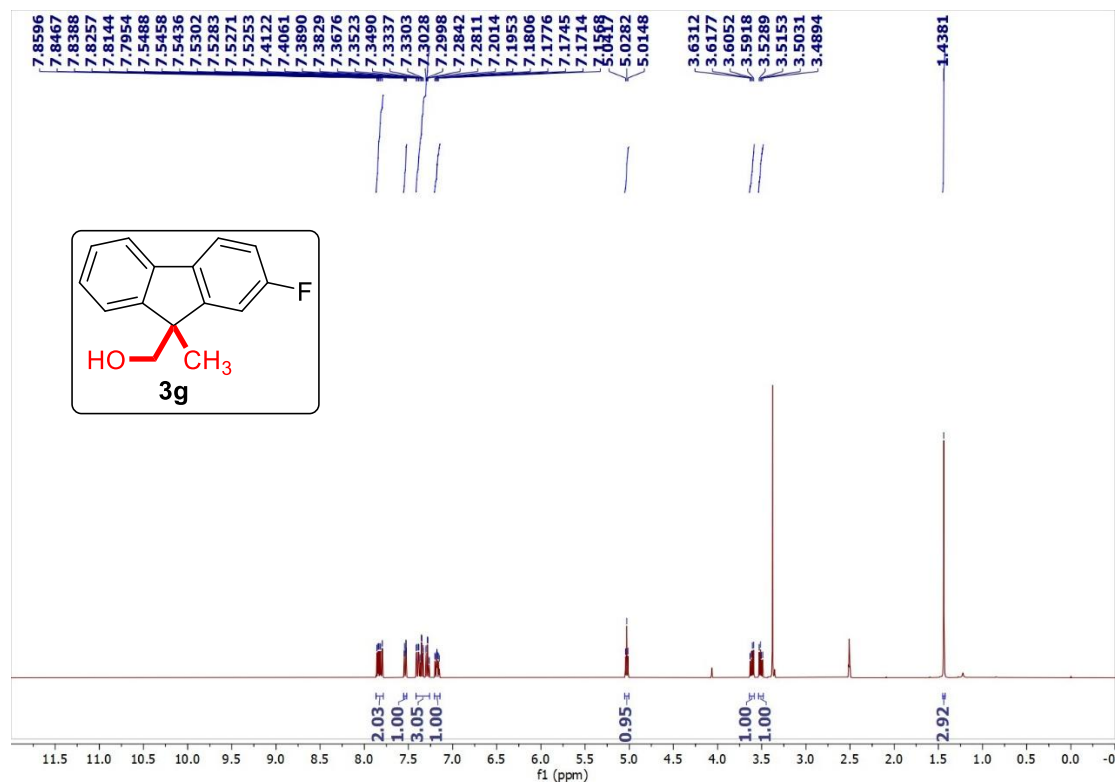


HRMS of (9-Methyl-2-phenyl-9H-fluoren-9-yl)methanol (3f)

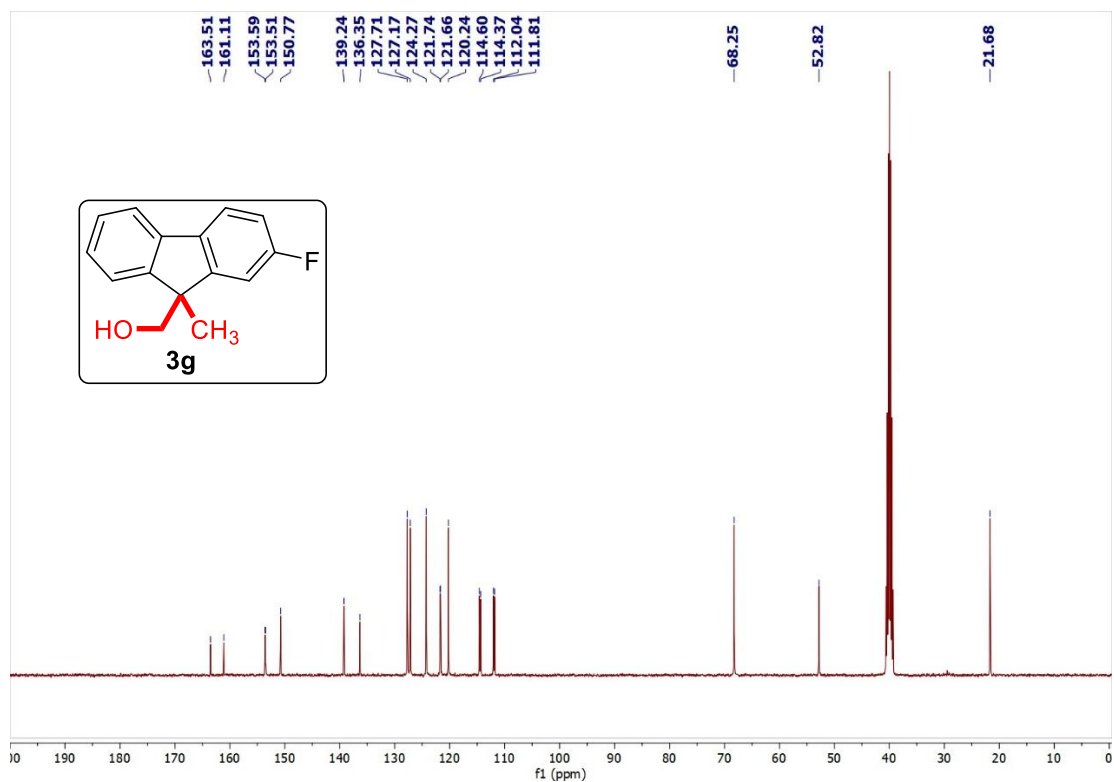
Sample Name	khp-kr-f22	Position	P1-B6	Instrument Name	Instrument 1	User Name	
Inj Vol	1	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	02.01.2025-15.d	ACQ Method	M60 W40.m	Comment		Acquired Time	03-01-2025 14:10:12



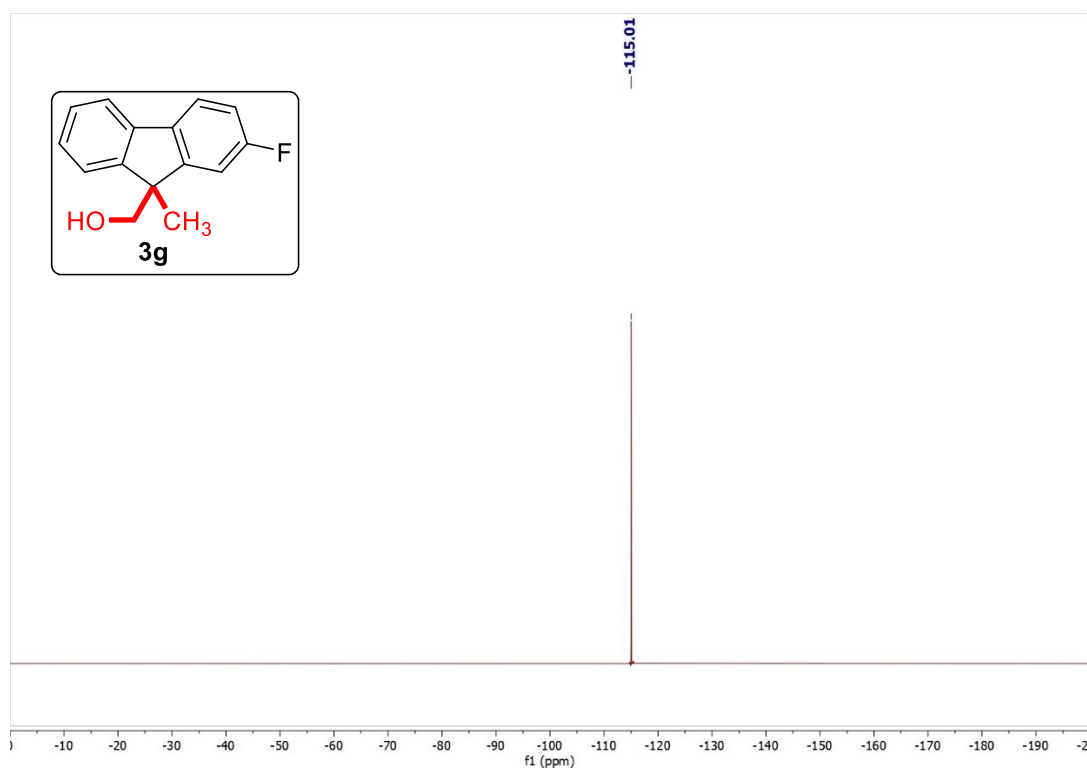
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of (2-Fluoro-9-methyl-9*H*-fluoren-9-yl)methanol (3g)



¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) spectrum of (2-Fluoro-9-methyl-9*H*-fluoren-9-yl)methanol (3g)

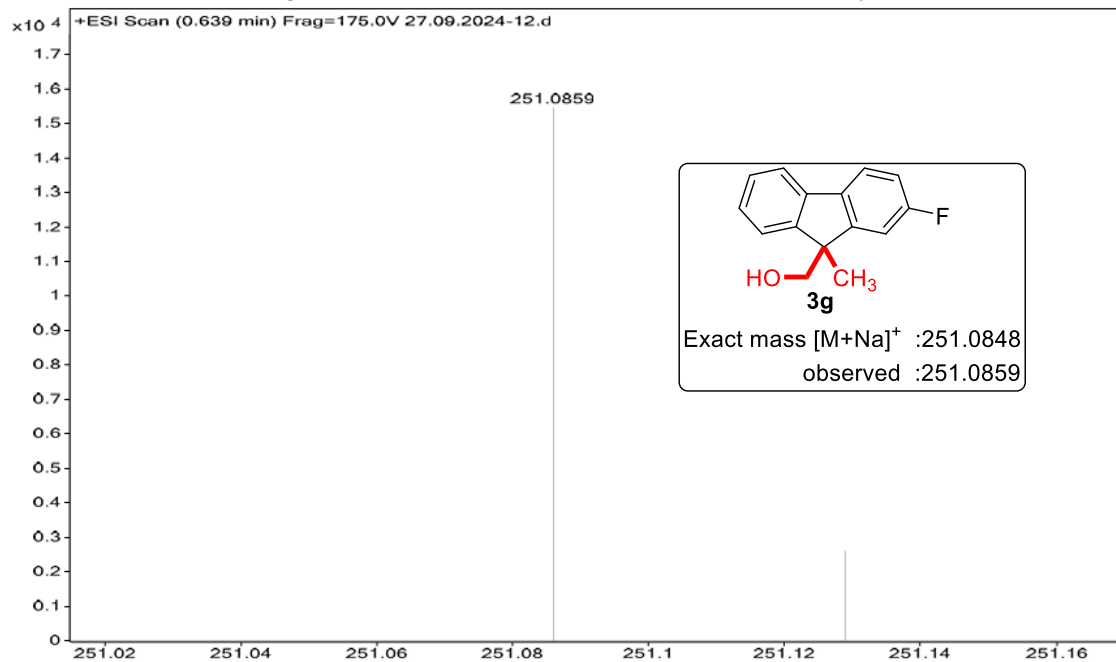


^{19}F spectrum of (2-Fluoro-9-methyl-9H-fluoren-9-yl)methanol (3g)

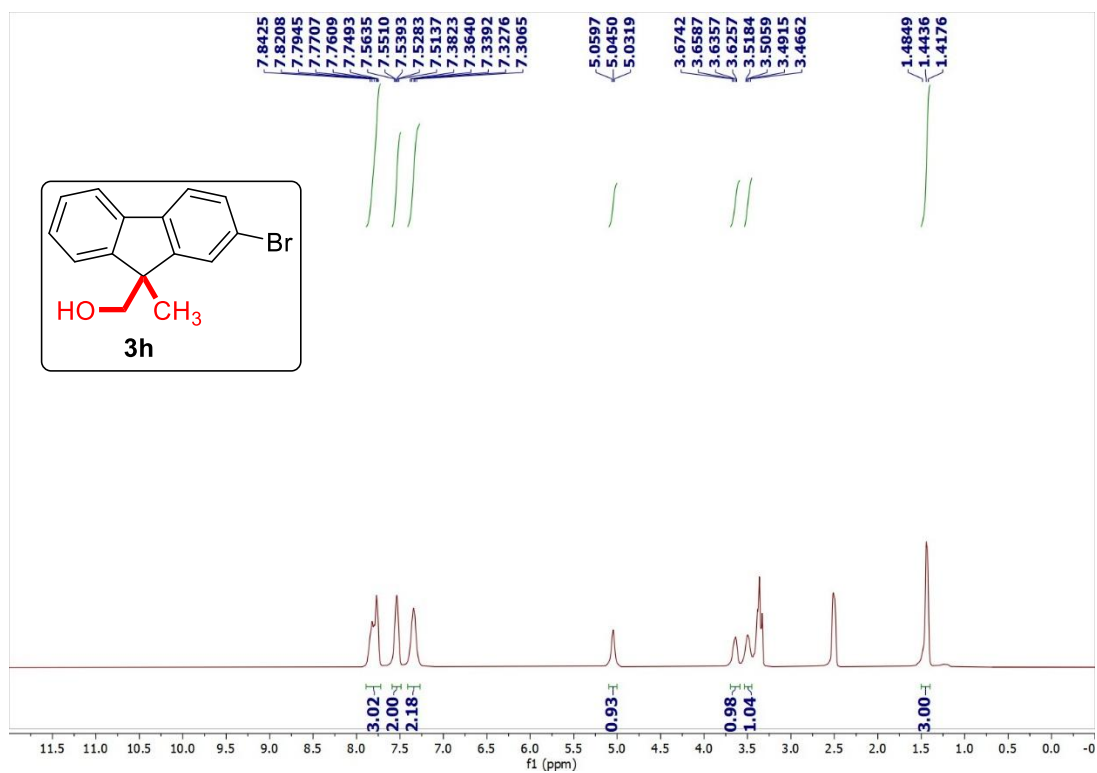


HRMS of (2-Fluoro-9-methyl-9H-fluoren-9-yl)methanol (3g)

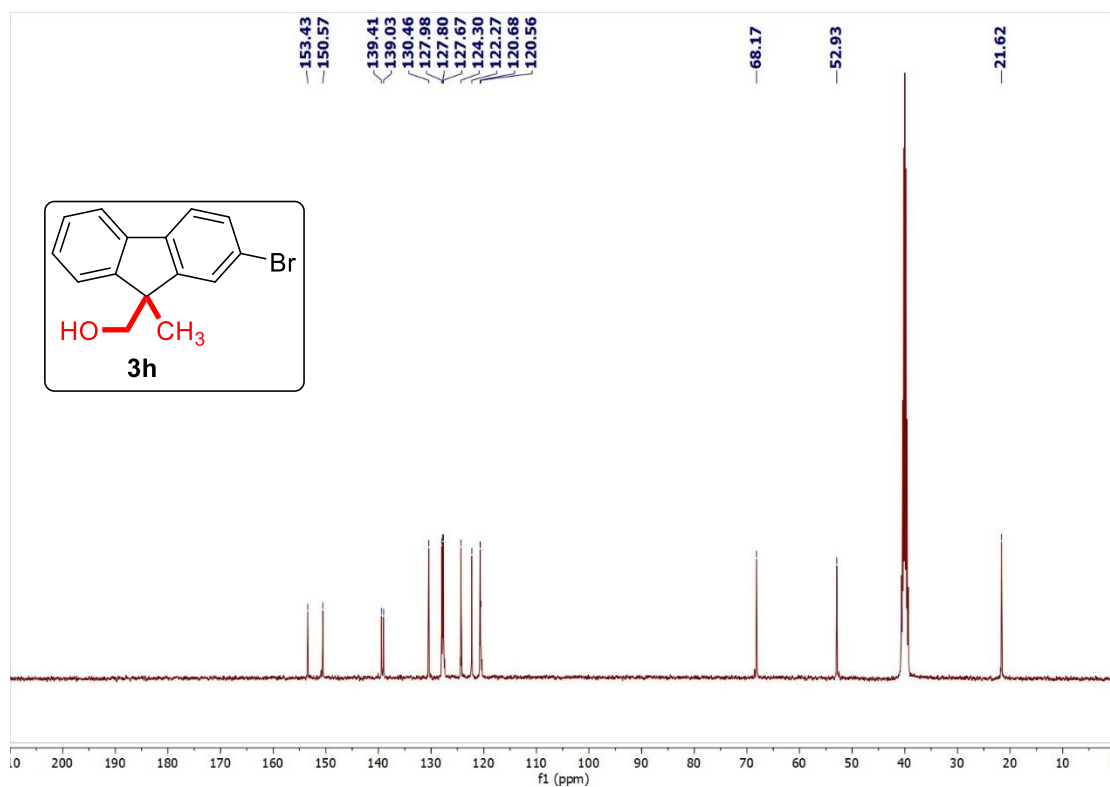
Sample Name	Position	Instrument Name	User Name
khp-kr-f11	P1-B3	Instrument 1	
Inj Vol	InjPosition	SampleType	IRM Calibration Status
2		Sample	Success
Data Filename	ACQ Method	Comment	Acquired Time
27.09.2024-12.d	M60 W40.m		27-09-2024 13:34:51



¹H NMR (400 MHz, DMSO-*d*₆) spectrum of (2-Bromo-9-methyl-9*H*-fluoren-9-yl)methanol (3h)

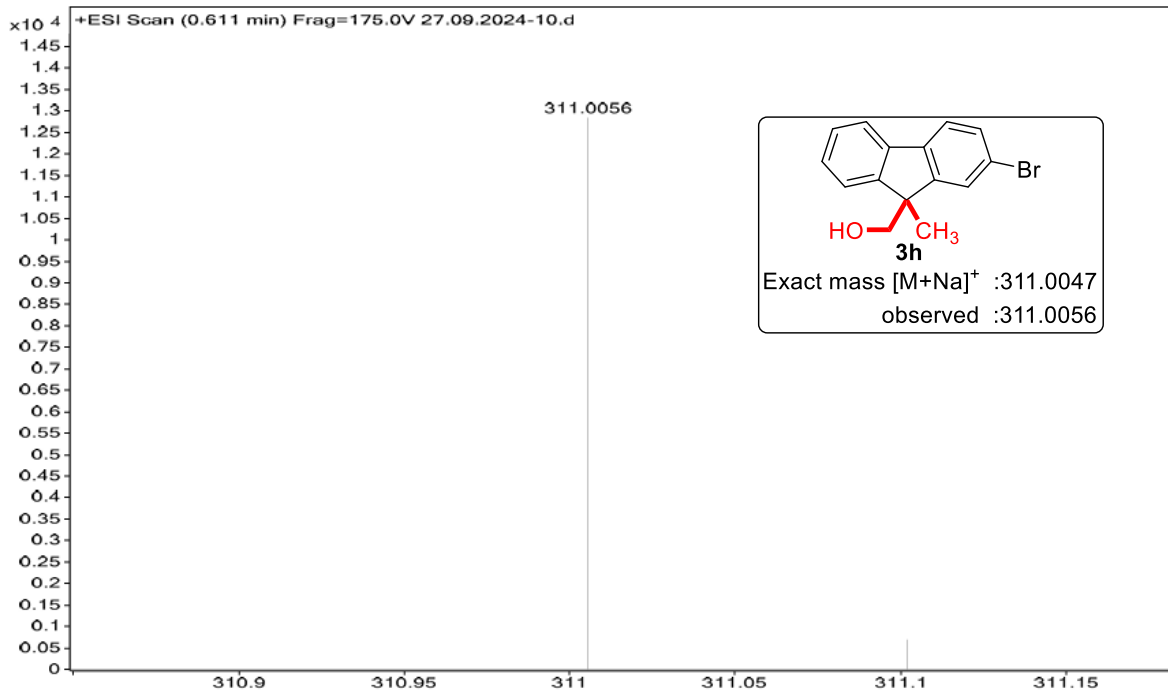


¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) spectrum of (2-Bromo-9-methyl-9*H*-fluoren-9-yl)methanol (3h)

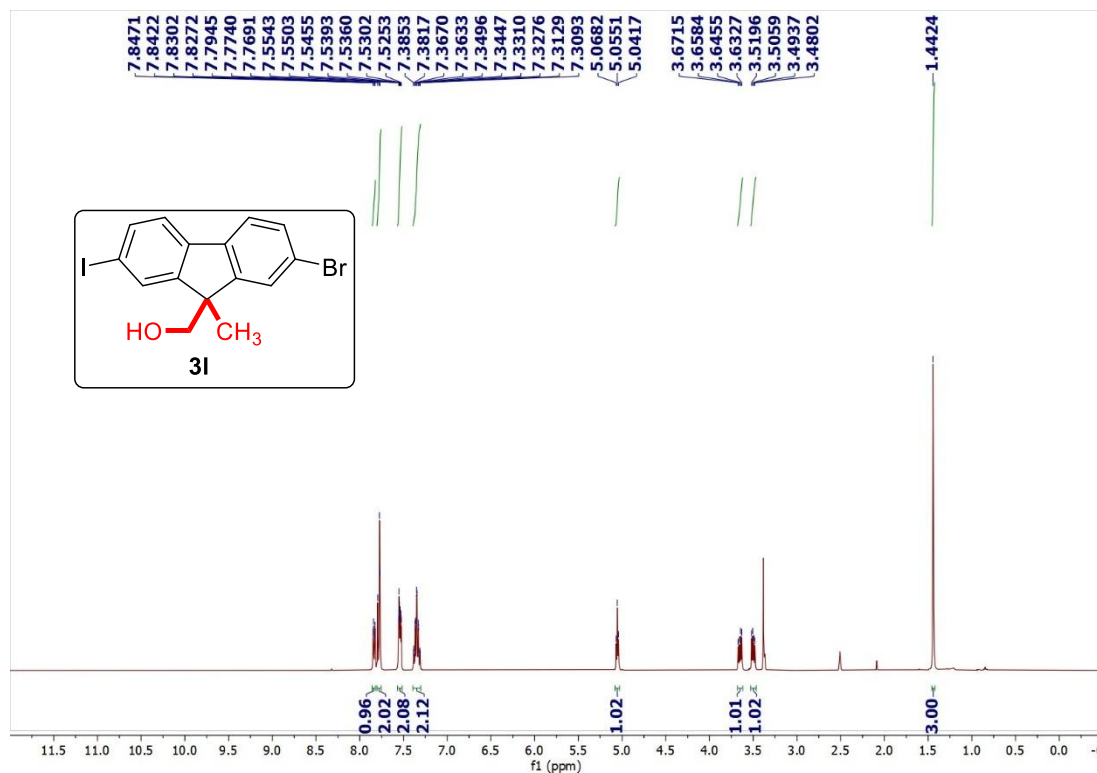


HRMS of (2-Bromo-9-methyl-9H-fluoren-9-yl)methanol (3h)

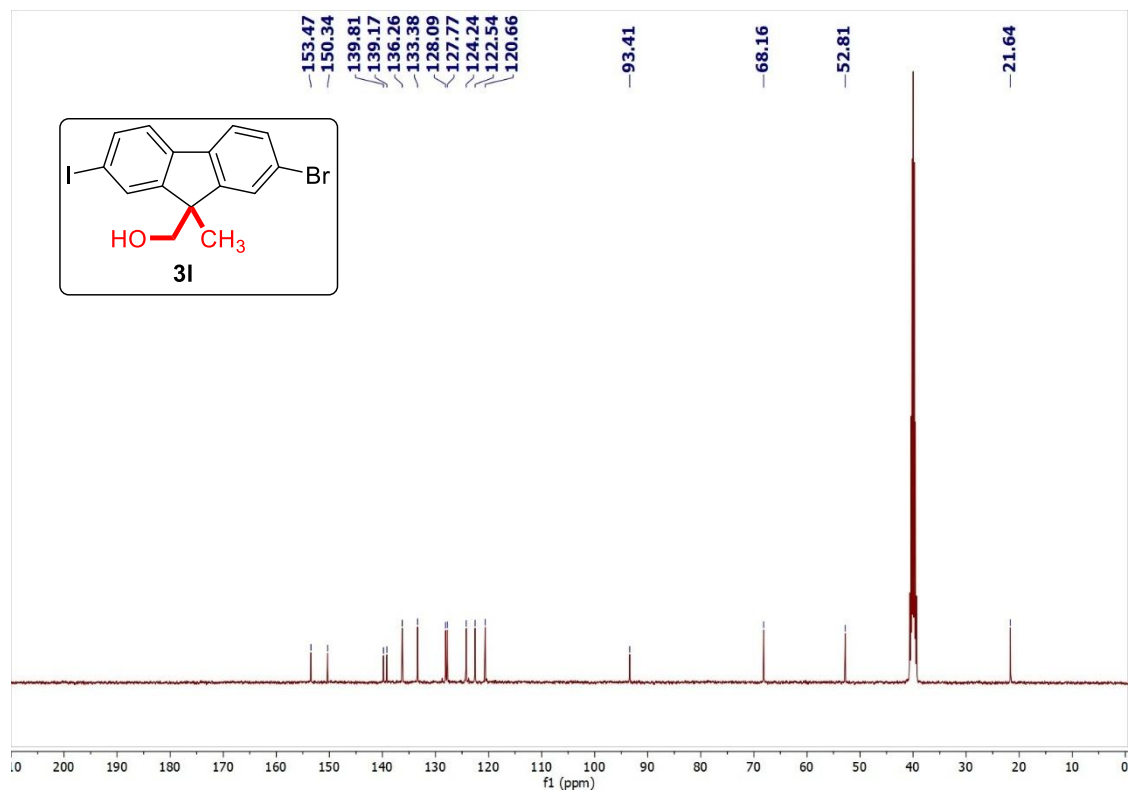
Sample Name	khp-kr-f9	Position	P1-B1	Instrument Name	Instrument 1	User Name	
Inj Vol	2	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	27.09.2024-10.d	ACQ Method	M60 W40.m	Comment		Acquired Time	27-09-2024 13:26:50



¹H NMR (400 MHz, DMSO-*d*₆) spectrum of (2-Iodo-9-methyl-9*H*-fluoren-9-yl)methanol (3i)

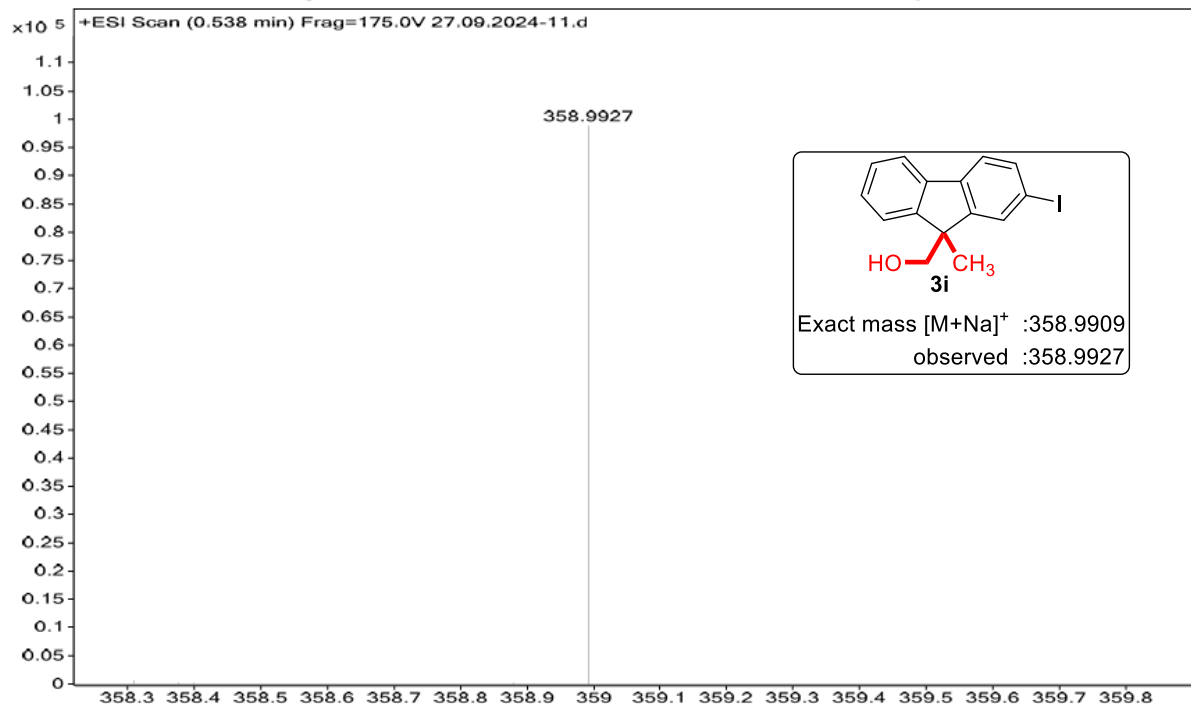


¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) spectrum of (2-Iodo-9-methyl-9*H*-fluoren-9-yl)methanol (3i)

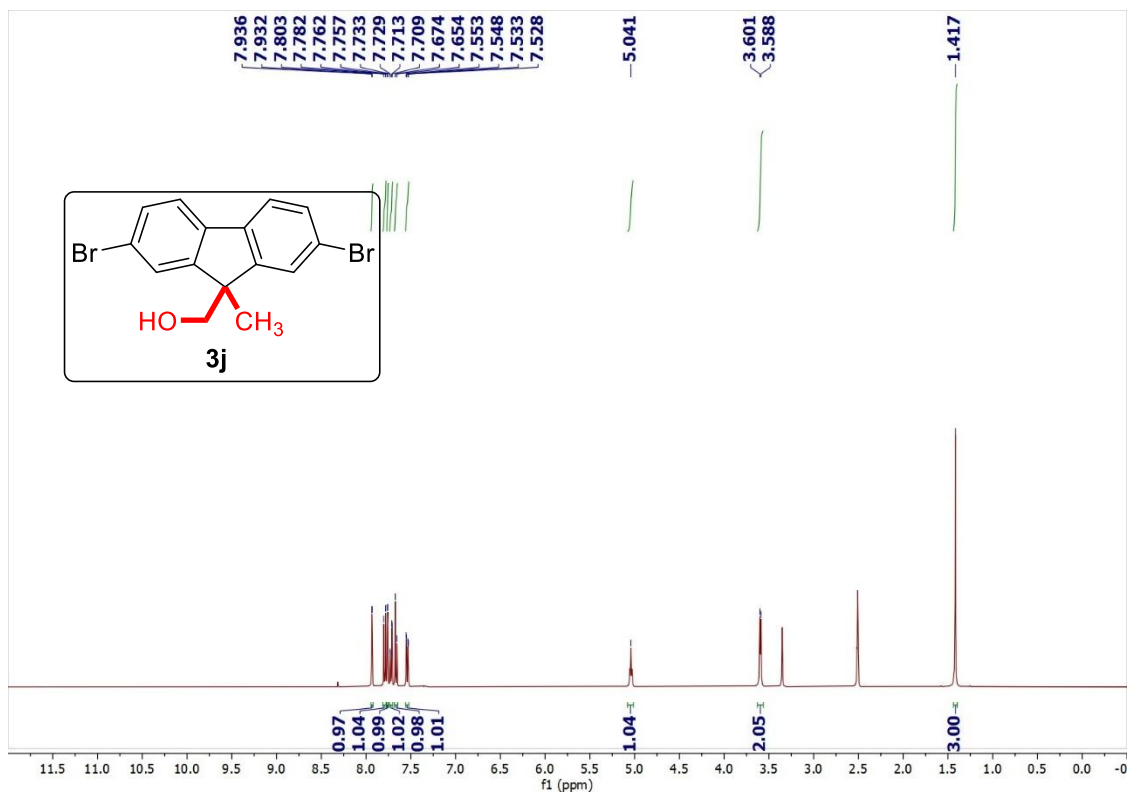


HRMS of (2-Iodo-9-methyl-9H-fluoren-9-yl)methanol (3i)

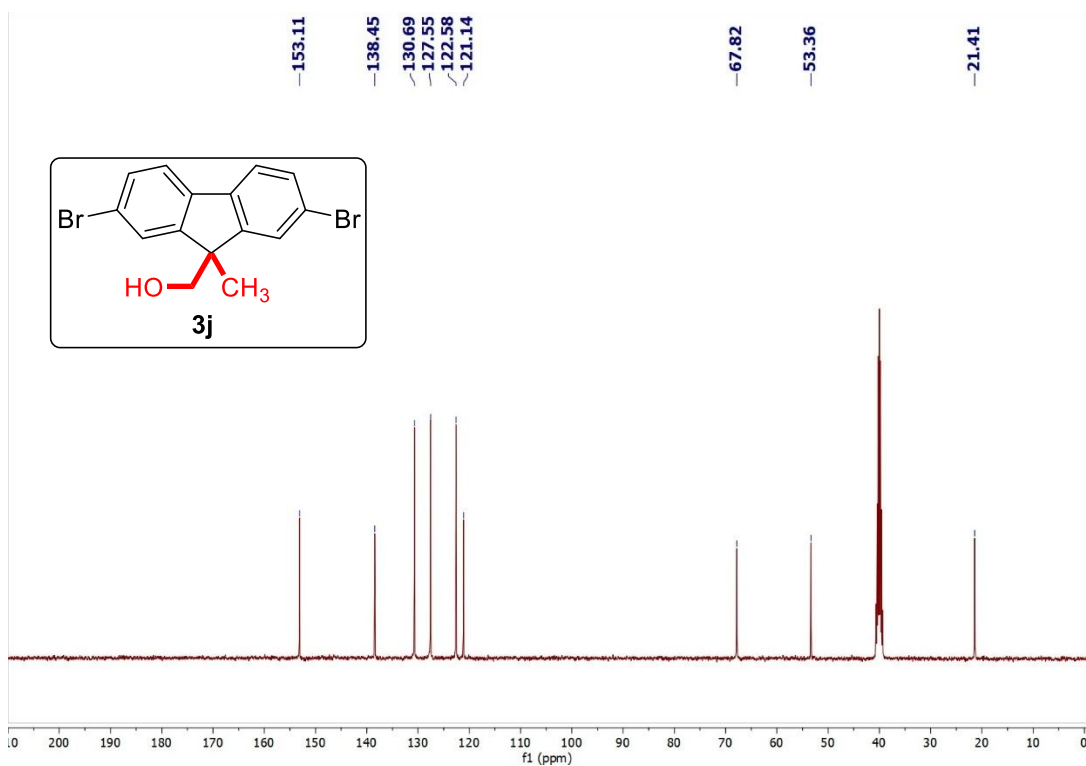
Sample Name	khp-kr-f10	Position	P1-B2	Instrument Name	Instrument 1	User Name	
Inj Vol	2	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	27.09.2024-11.d	ACQ Method	M60 W40.m	Comment		Acquired Time	27-09-2024 13:30:49



¹H NMR (400 MHz, DMSO-*d*₆) spectrum of (2,7-Dibromo-9-methyl-9*H*-fluoren-9-yl)methanol (3j)

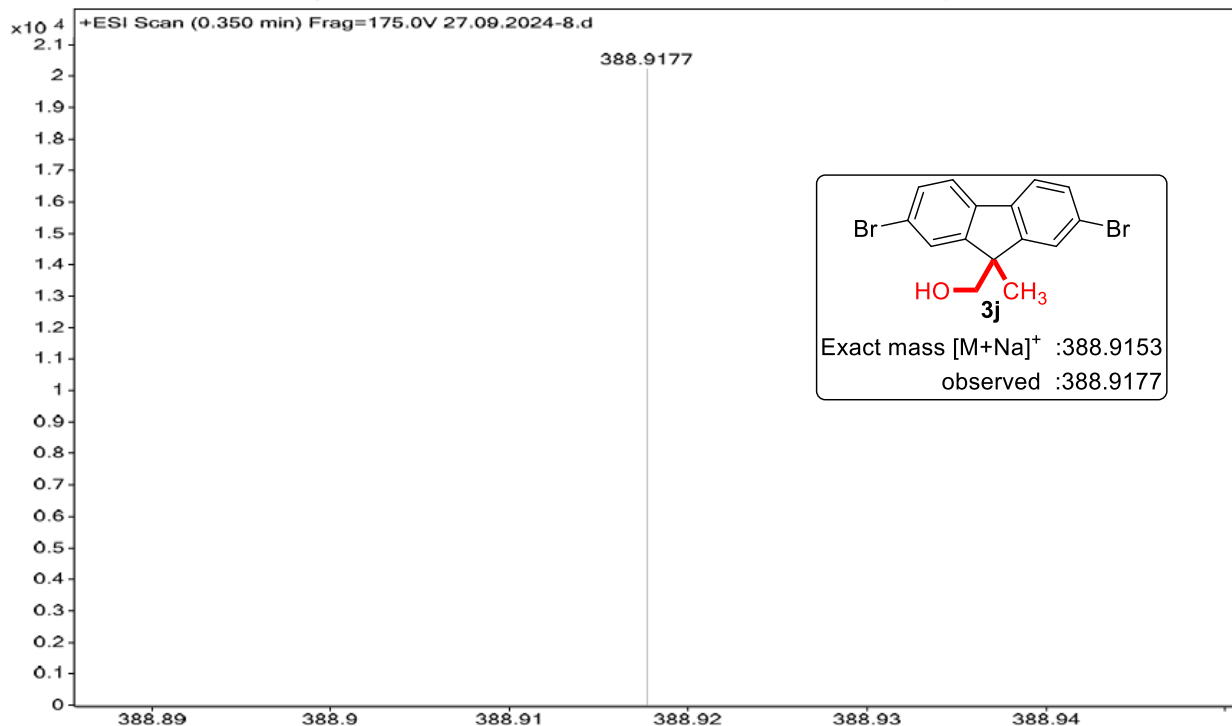


¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) spectrum of (2,7-Dibromo-9-methyl-9*H*-fluoren-9-yl)methanol (3j)

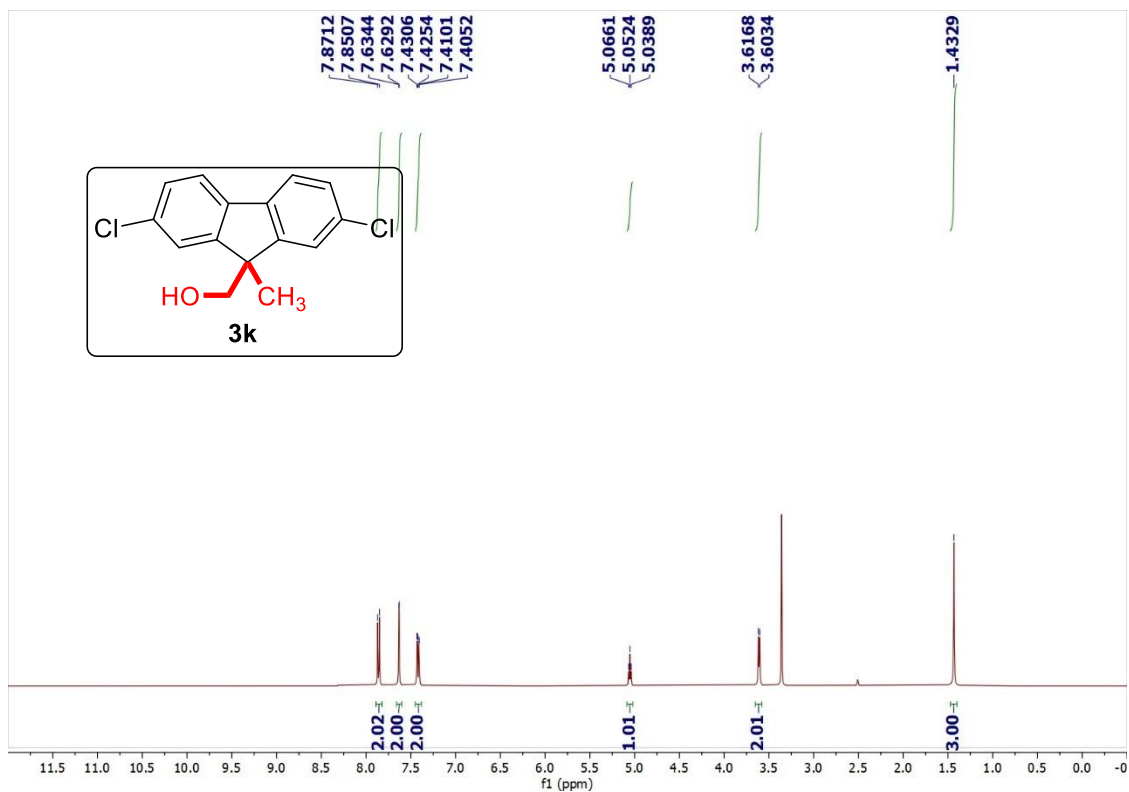


HRMS of (2,7-Dibromo-9-methyl-9H-fluoren-9-yl)methanol (3j)

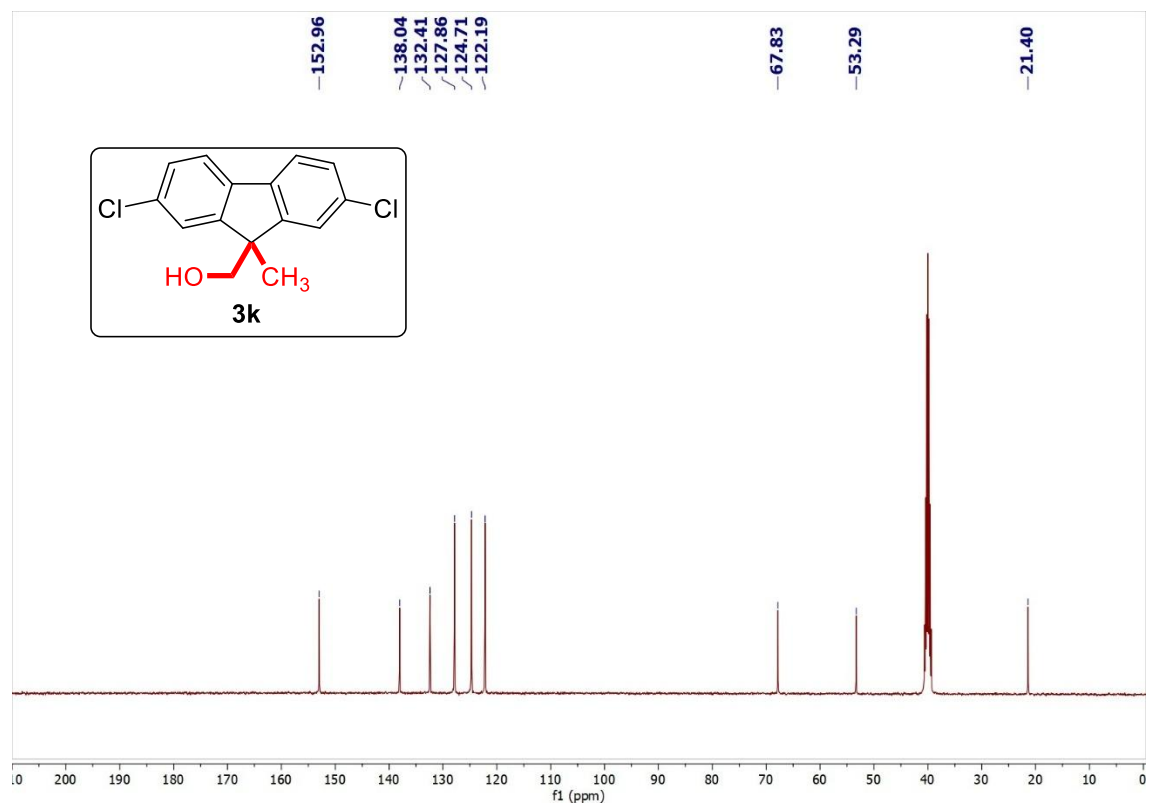
Sample Name	khp-kr-f7	Position	P1-A8	Instrument Name	Instrument 1	User Name	
Inj Vol	2	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	27.09.2024-8.d	ACQ Method	M60 W40.m	Comment		Acquired Time	27-09-2024 13:18:52



¹H NMR (400 MHz, DMSO-*d*₆) spectrum of (2,7-Dichloro-9-methyl-9H-fluoren-9-yl)methanol (3k)

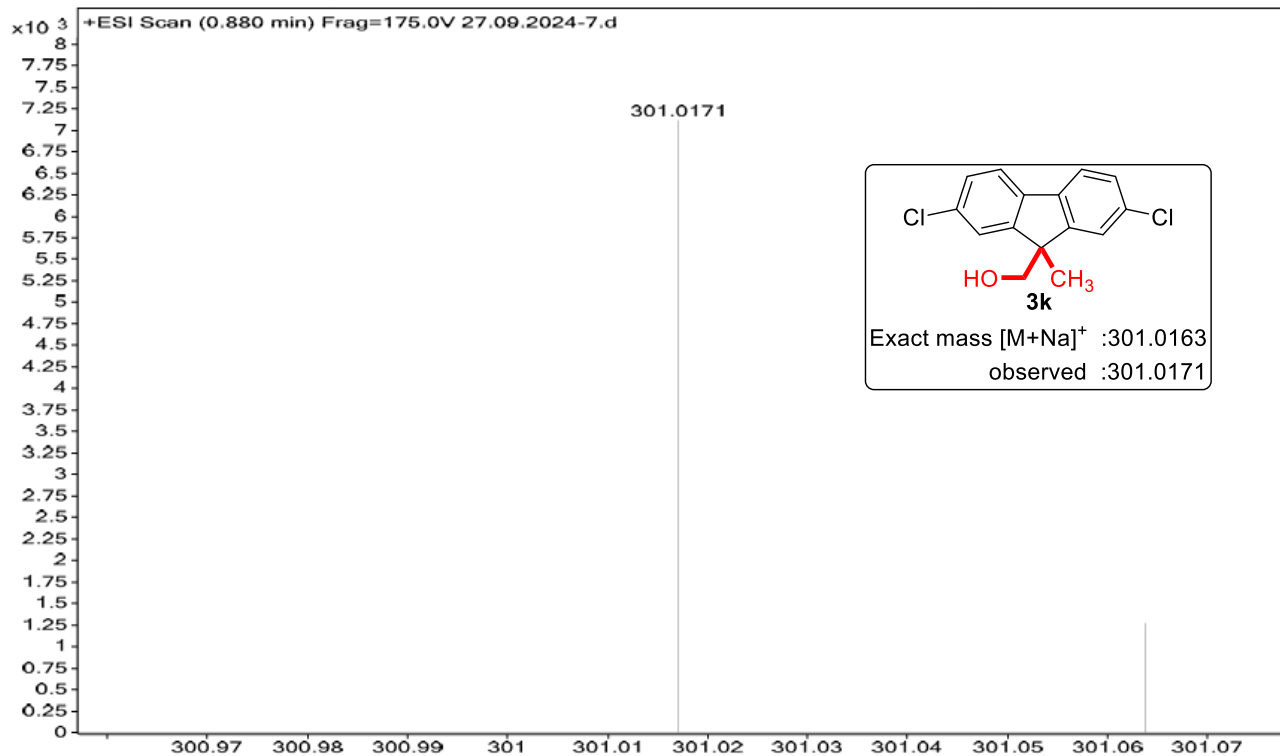


¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) spectrum of (2,7-Dichloro-9-methyl-9H-fluoren-9-yl)methanol (3k)

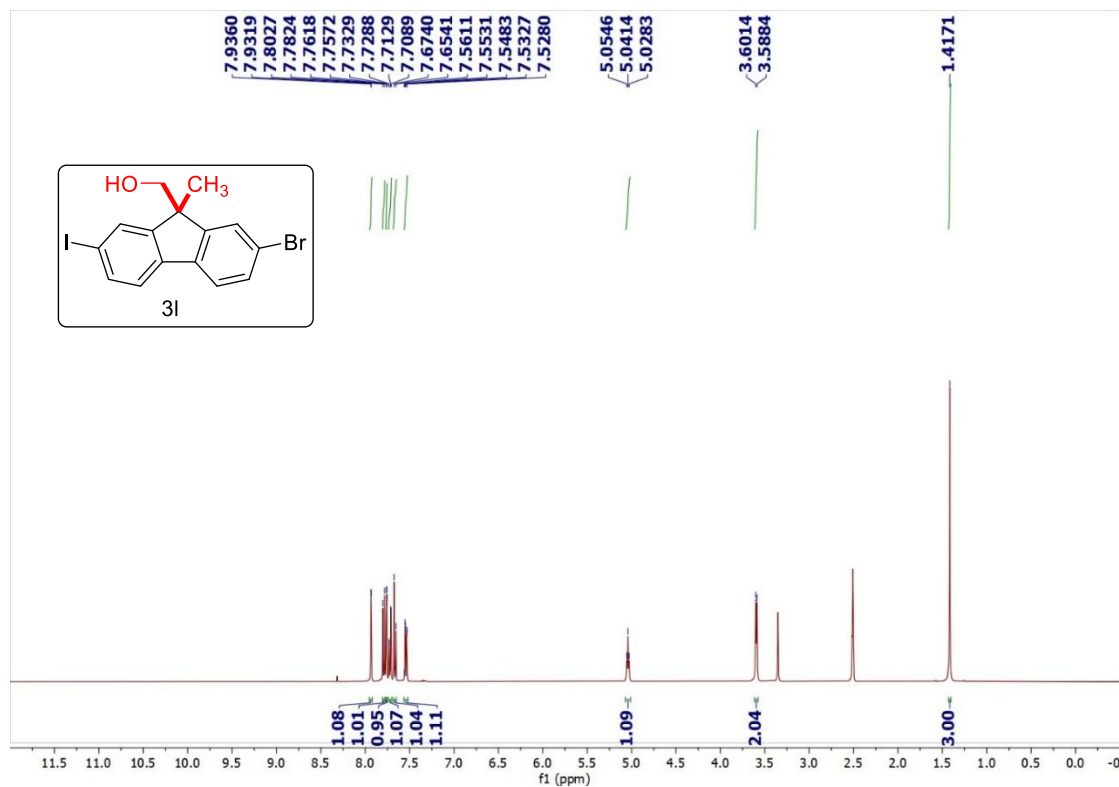


HRMS of (2,7-Dichloro-9-methyl-9H-fluoren-9-yl)methanol (3k)

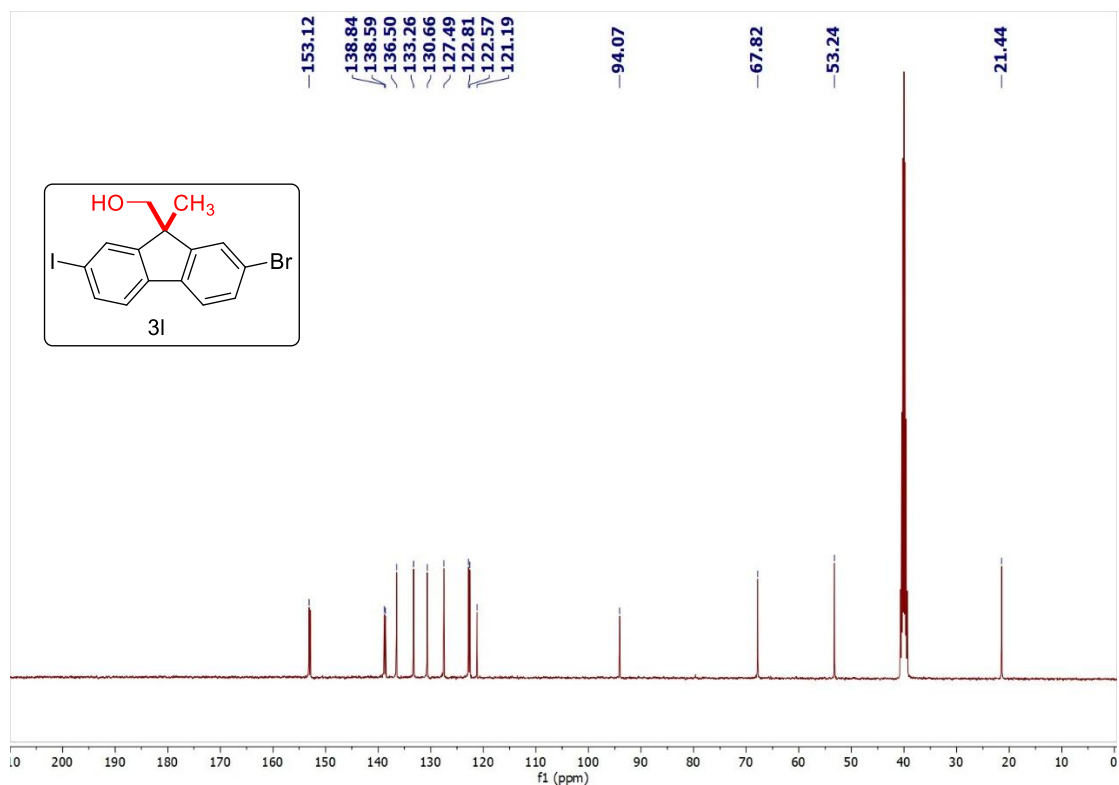
Sample Name	khp-kr-f6	Position	P1-A7	Instrument Name	Instrument 1	User Name	
Inj Vol	2	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	27.09.2024-7.d	ACQ Method	M60 W40.m	Comment		Acquired Time	27-09-2024 13:14:52



¹H NMR (400 MHz, DMSO-*d*₆) spectrum of (2-Bromo-7-iodo-9-methyl-9H-fluoren-9-yl)methanol (3l)

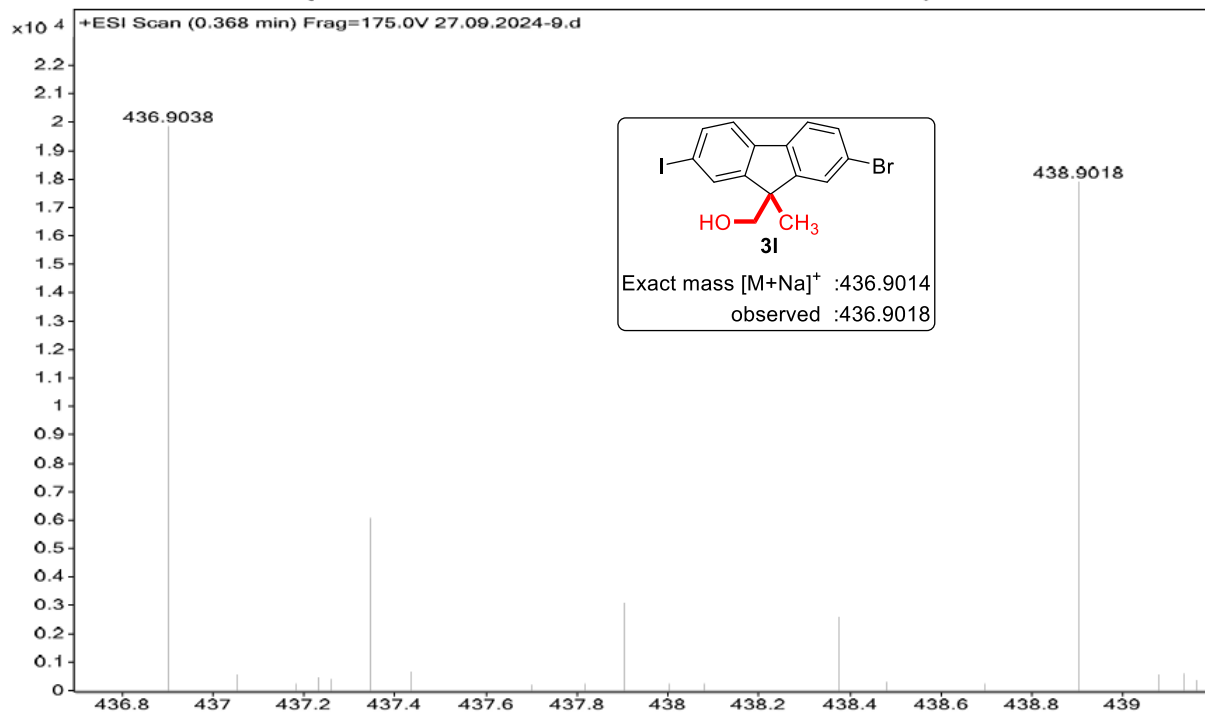


¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) spectrum of (2-Bromo-7-iodo-9-methyl-9H-fluoren-9-yl)methanol (3l)

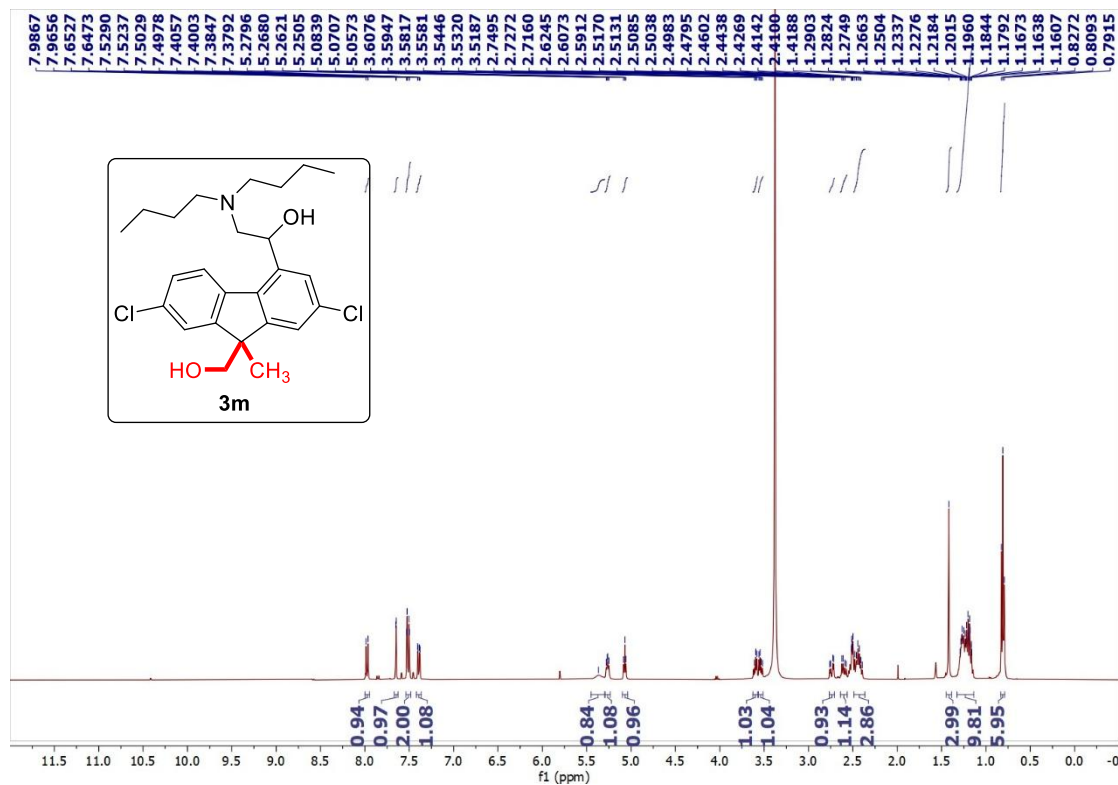


HRMS of (2-Bromo-7-iodo-9-methyl-9H-fluoren-9-yl)methanol (3I)

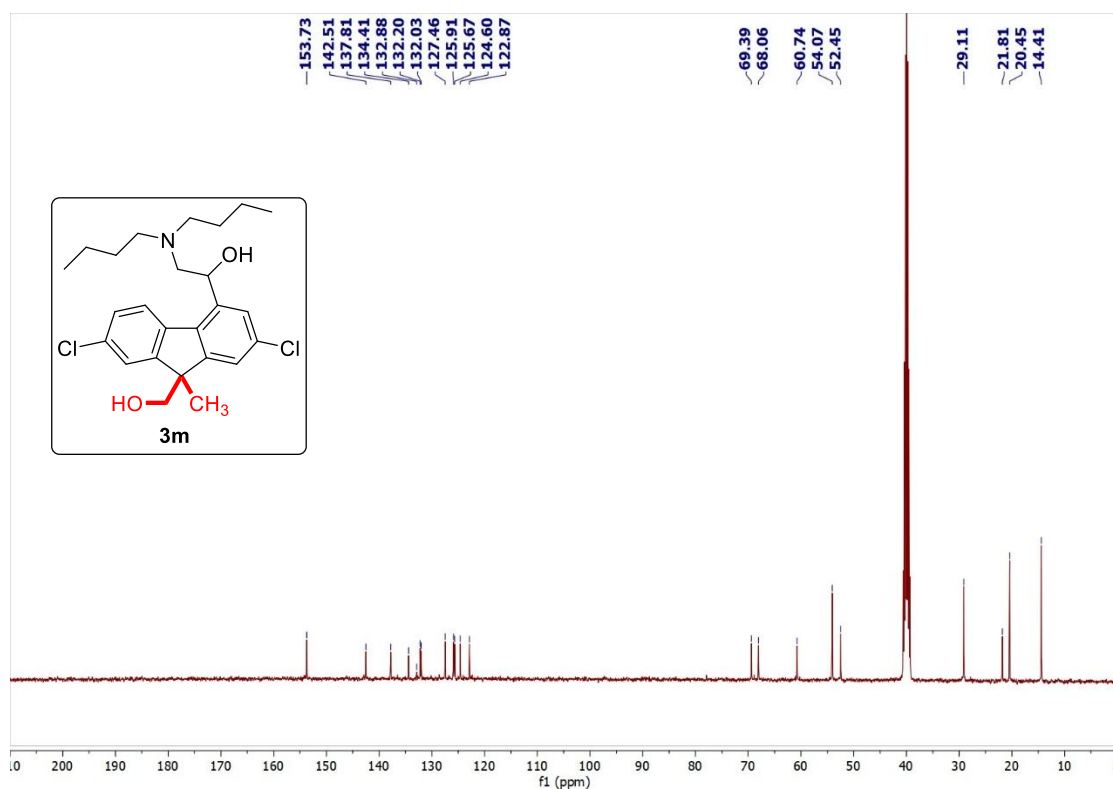
Sample Name	khp-kr-f8	Position	P1-A9	Instrument Name	Instrument 1	User Name	
Inj Vol	2	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	27.09.2024-9.d	ACQ Method	M60 W40.m	Comment		Acquired Time	27-09-2024 13:22:50



^1H NMR (400 MHz, $\text{DMSO-}d_6$) spectrum of 2-(Dibutylamino)-1-(2,7-dichloro-9-(hydroxymethyl)-9-methyl-9H-fluoren-4-yl)ethan-1-ol (3m)

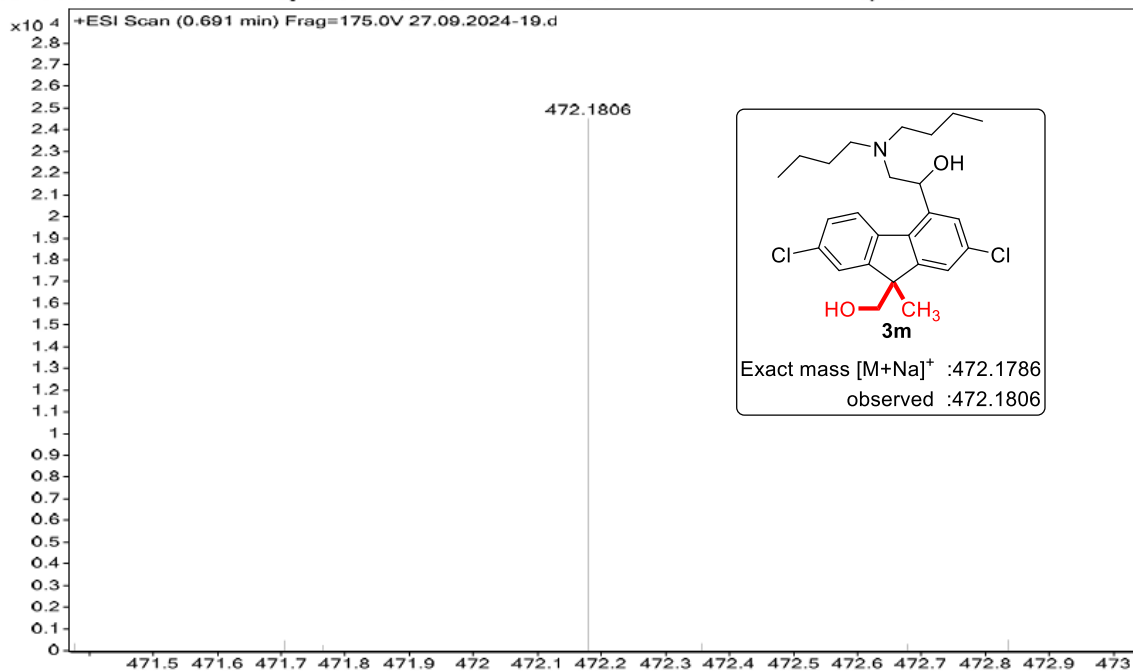


$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, $\text{DMSO-}d_6$) spectrum of 2-(Dibutylamino)-1-(2,7-dichloro-9-(hydroxymethyl)-9-methyl-9H-fluoren-4-yl)ethan-1-ol (3m)

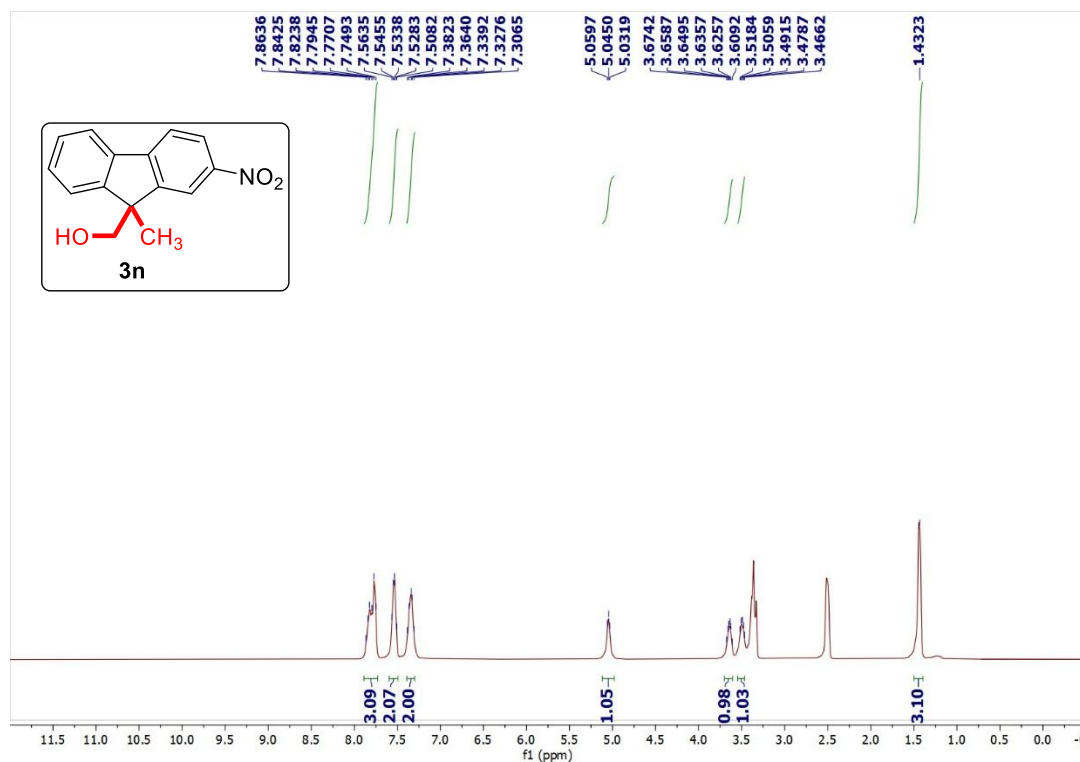


HRMS of 2-(Dibutylamino)-1-(2,7-dichloro-9-(hydroxymethyl)-9-methyl-9H-fluoren-4-yl)ethan-1-ol (3m)

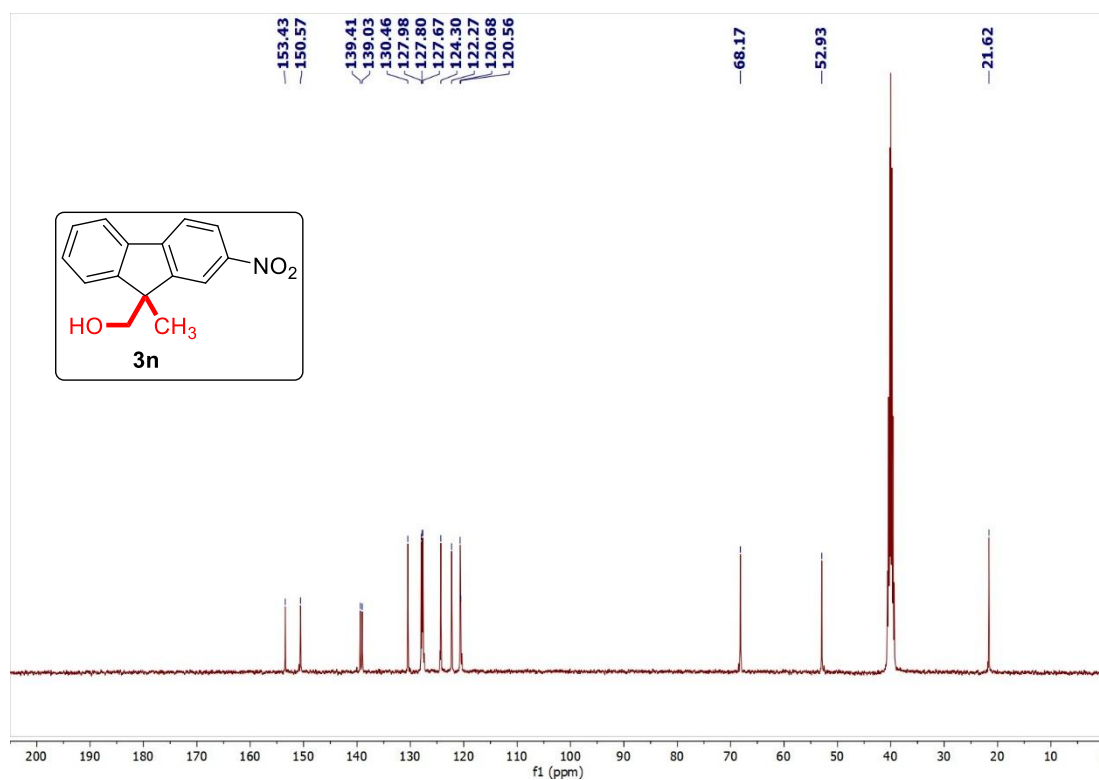
Sample Name	khp-kr-f18	Position	P1-C1	Instrument Name	Instrument 1	User Name	
Inj Vol	2	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	27.09.2024-19.d	ACQ Method	M60 W40.m	Comment		Acquired Time	27-09-2024 14:03:09



¹H NMR (400 MHz, DMSO-*d*₆) spectrum of (9-Methyl-2-nitro-9H-fluoren-9-yl)methanol 3 (3n)

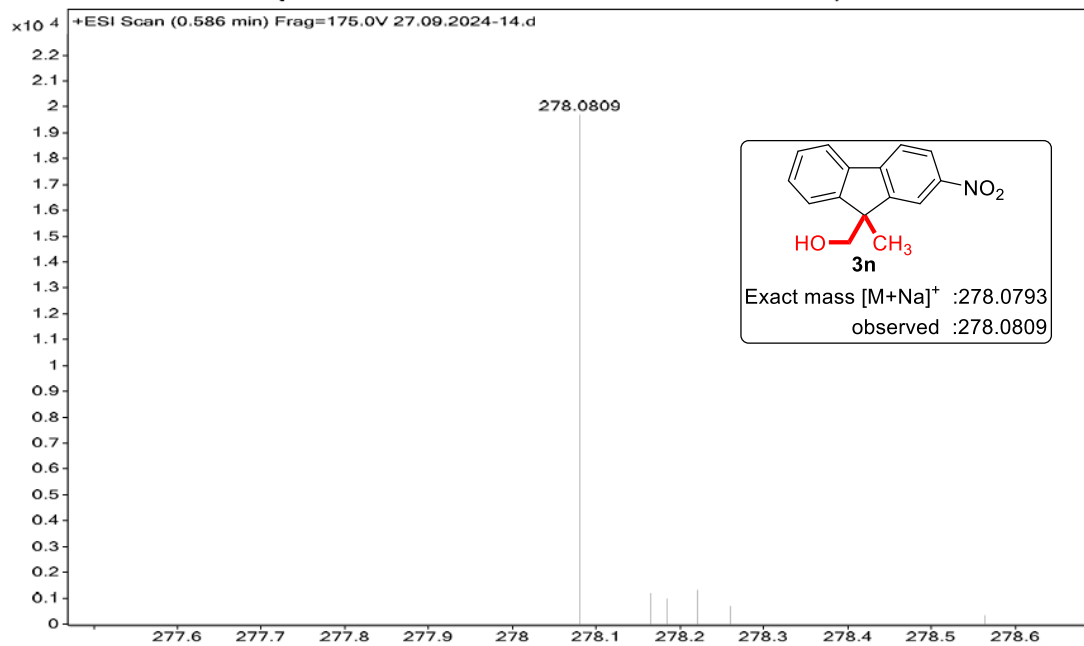


¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) spectrum of (9-Methyl-2-nitro-9H-fluoren-9-yl)methanol 3 (3n)

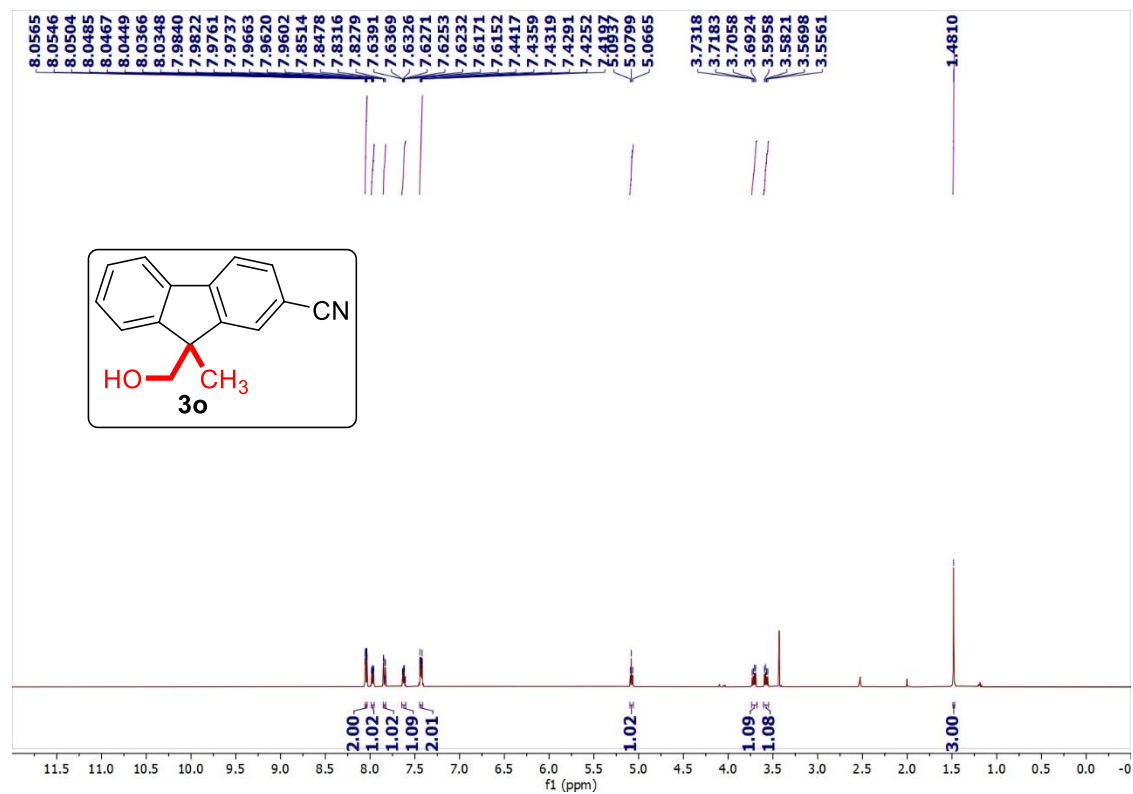


HRMS of (9-Methyl-2-nitro-9H-fluoren-9-yl)methanol 3 (3n)

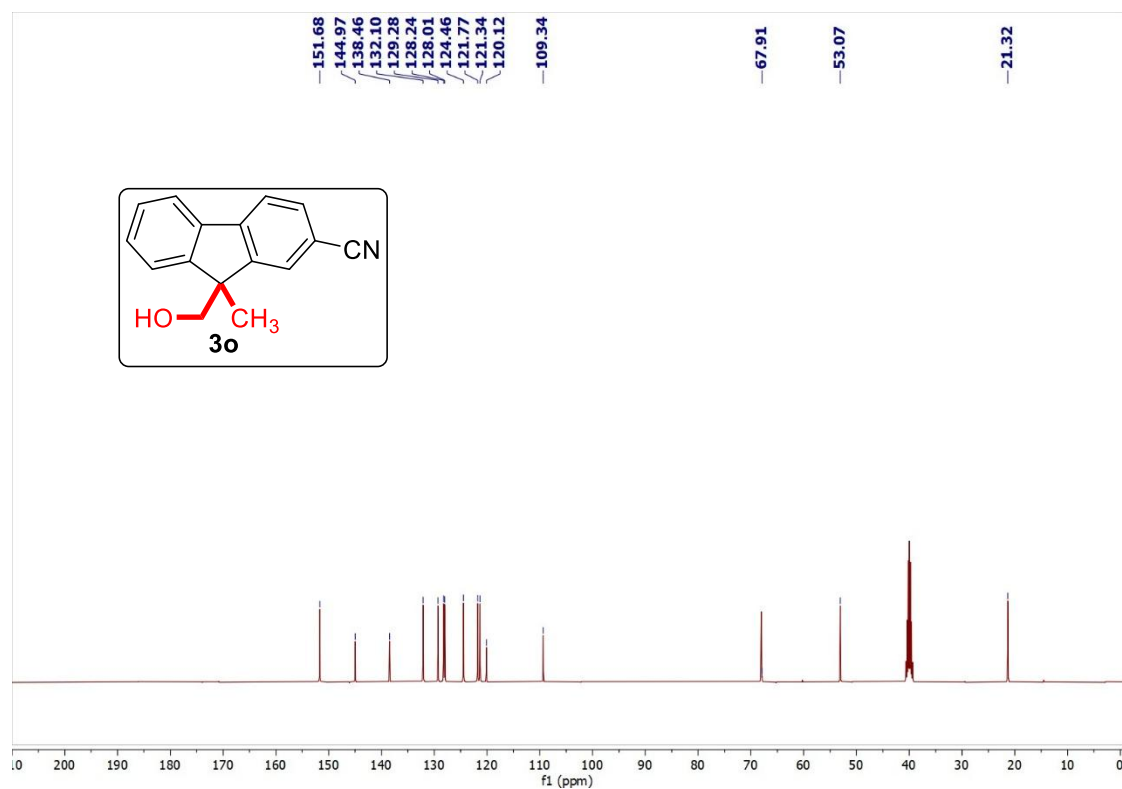
Sample Name	khp-kr-f13	Position	P1-B5	Instrument Name	Instrument 1	User Name	
Inj Vol	2	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	27.09.2024-14.d	ACQ Method	M60 W40.m	Comment		Acquired Time	27-09-2024 13:42:58



¹H NMR (400 MHz, DMSO-*d*₆) spectrum of 9-(Hydroxymethyl)-9-methyl-9*H*-fluorene-2-carbonitrile (3o)

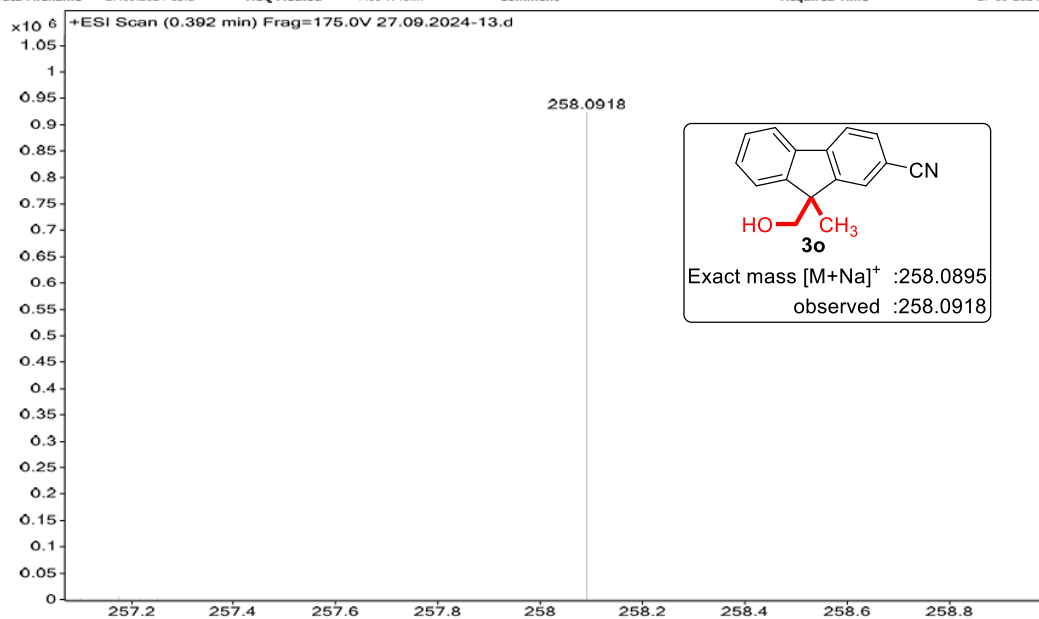


¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) spectrum of 9-(Hydroxymethyl)-9-methyl-9*H*-fluorene-2-carbonitrile (3o)

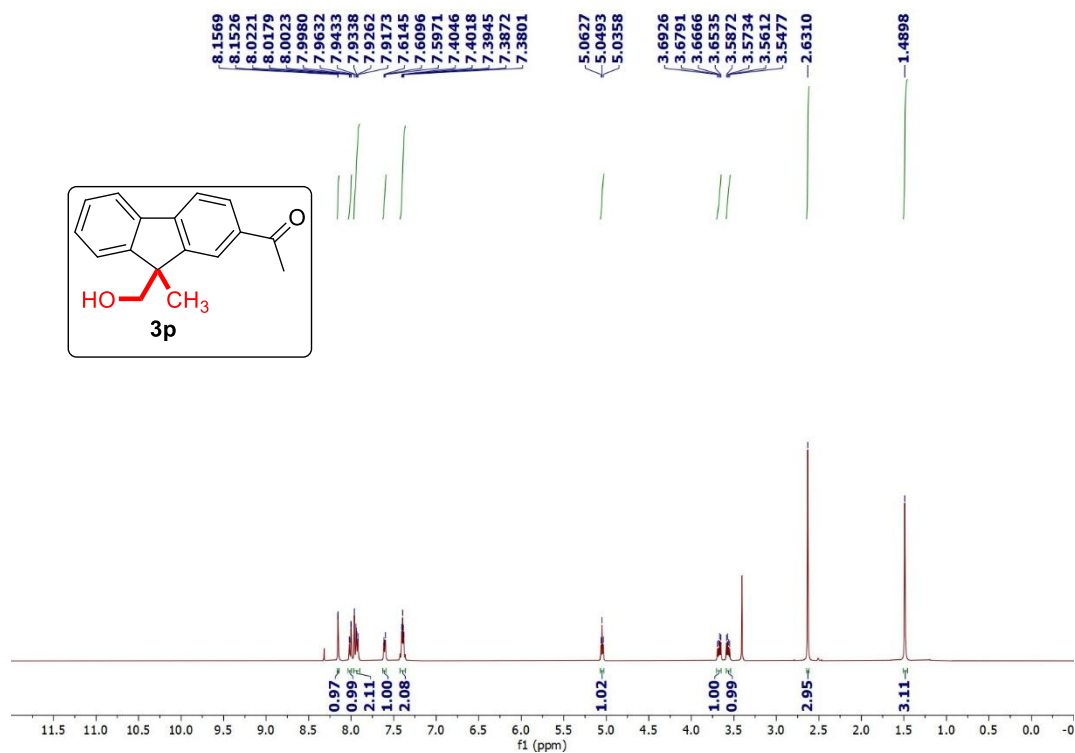


HRMS of 9-(Hydroxymethyl)-9-methyl-9H-fluorene-2-carbonitrile (3o)

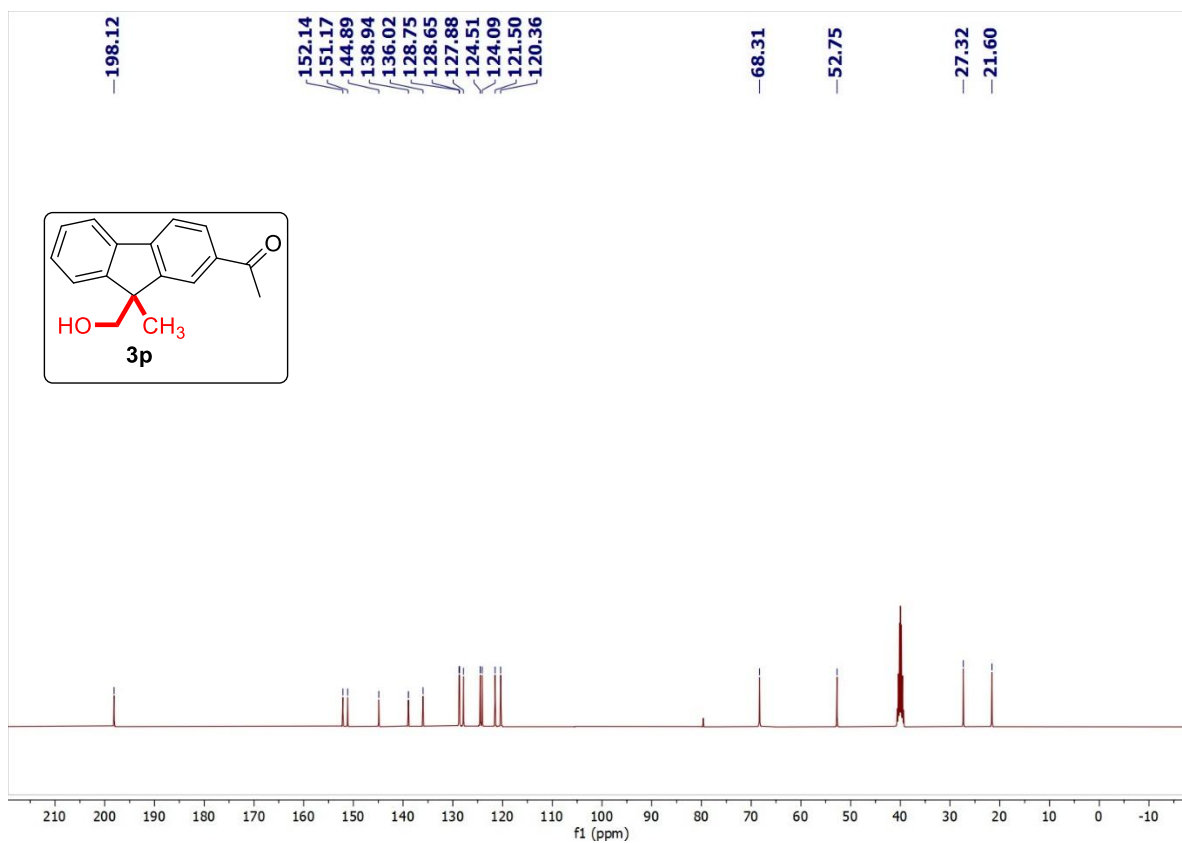
Sample Name	khp-kr-f12	Position	P1-B4	Instrument Name	Instrument 1	User Name	
Inj Vol	2	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	27.09.2024-13.d	ACQ Method	M60 W40.m	Comment		Acquired Time	27-09-2024 13:38:54



¹H NMR (400 MHz, DMSO-*d*₆) spectrum of 1-(9-(Hydroxymethyl)-9-methyl-9*H*-fluoren-2-yl)ethan-1-one (3p)

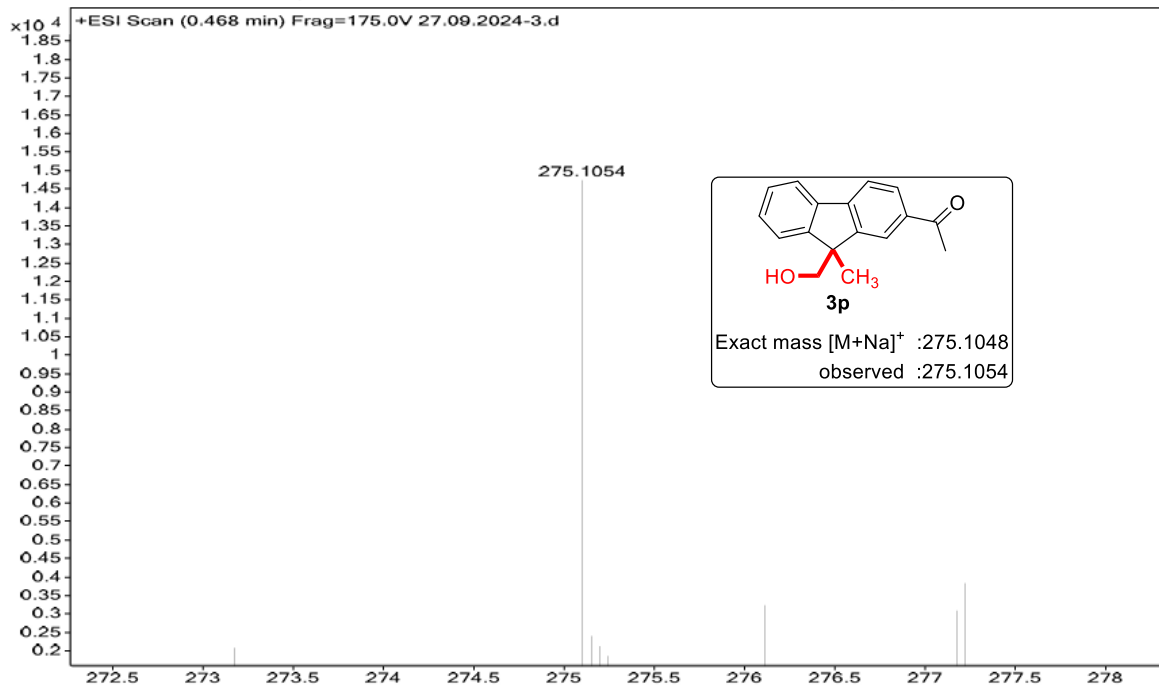


¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) spectrum of 1-(9-(Hydroxymethyl)-9-methyl-9*H*-fluoren-2-yl)ethan-1-one (3p)

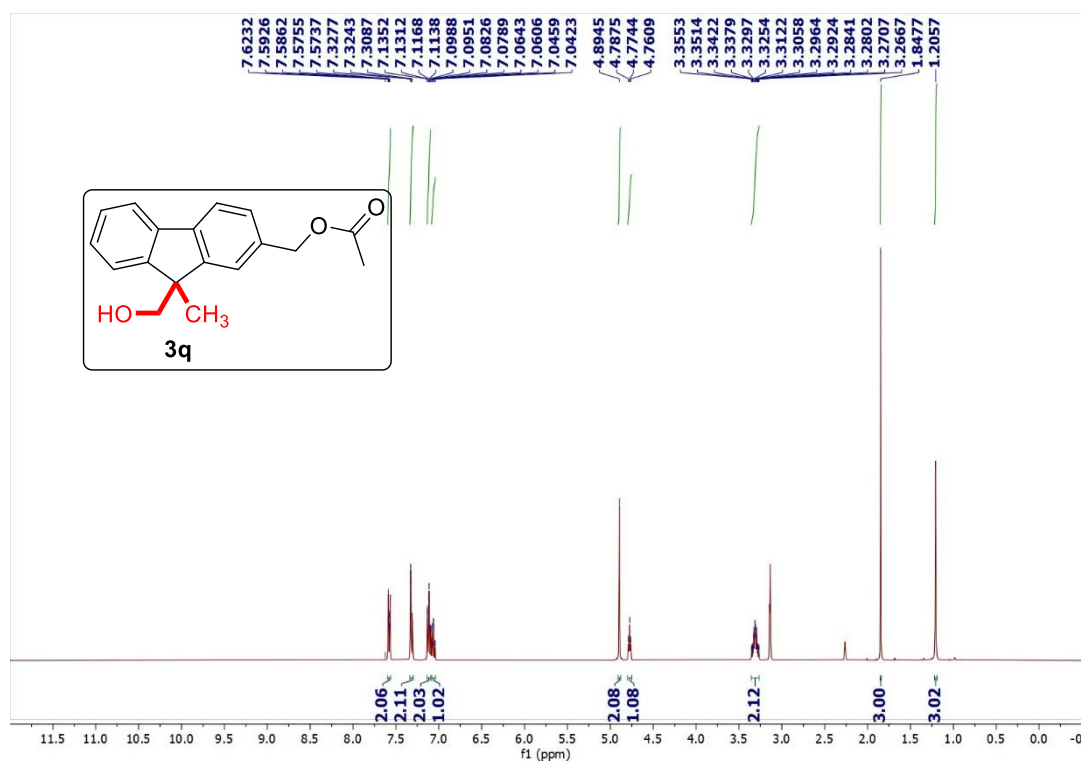


HRMS of 1-(9-(Hydroxymethyl)-9-methyl-9H-fluoren-2-yl)ethan-1-one (3p)

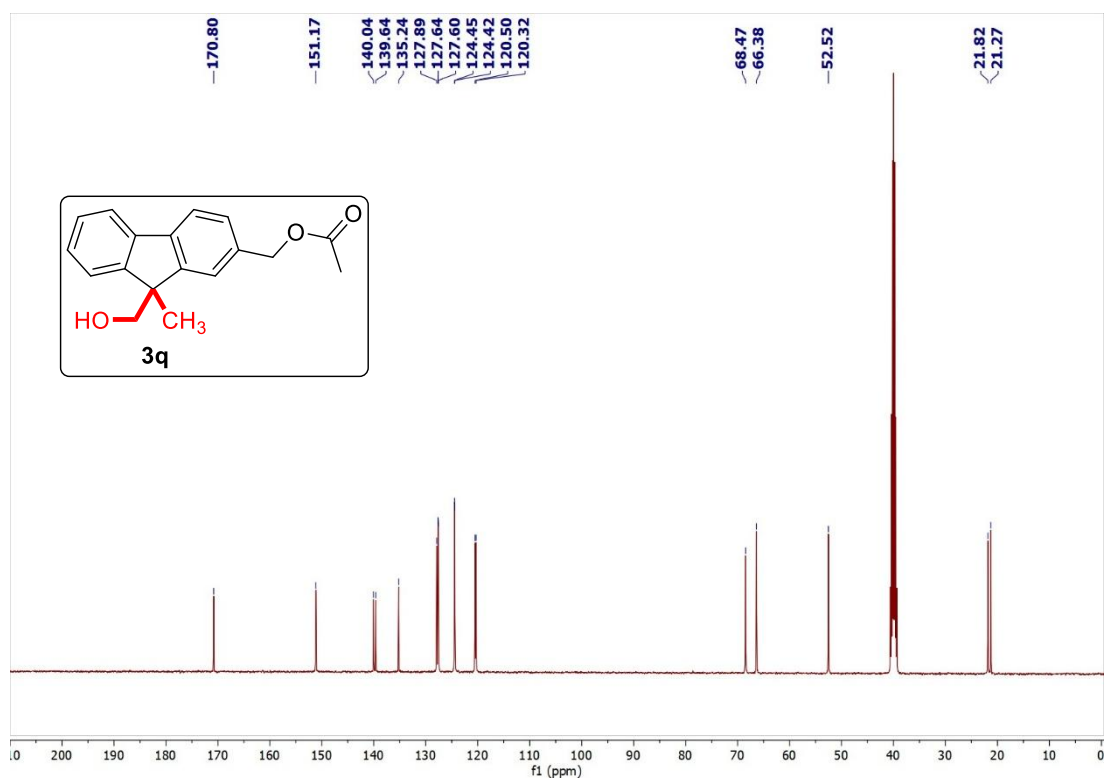
Sample Name	khp-kr-f2	Position	P1-A3	Instrument Name	Instrument 1	User Name	
Inj Vol	2	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	27.09.2024-3.d	ACQ Method	M60 W40.m	Comment		Acquired Time	27-09-2024 12:58:46



¹H NMR (400 MHz, DMSO-*d*₆) spectrum of (9-(Hydroxymethyl)-9-methyl-9*H*-fluoren-2-yl)methyl acetate (3q)

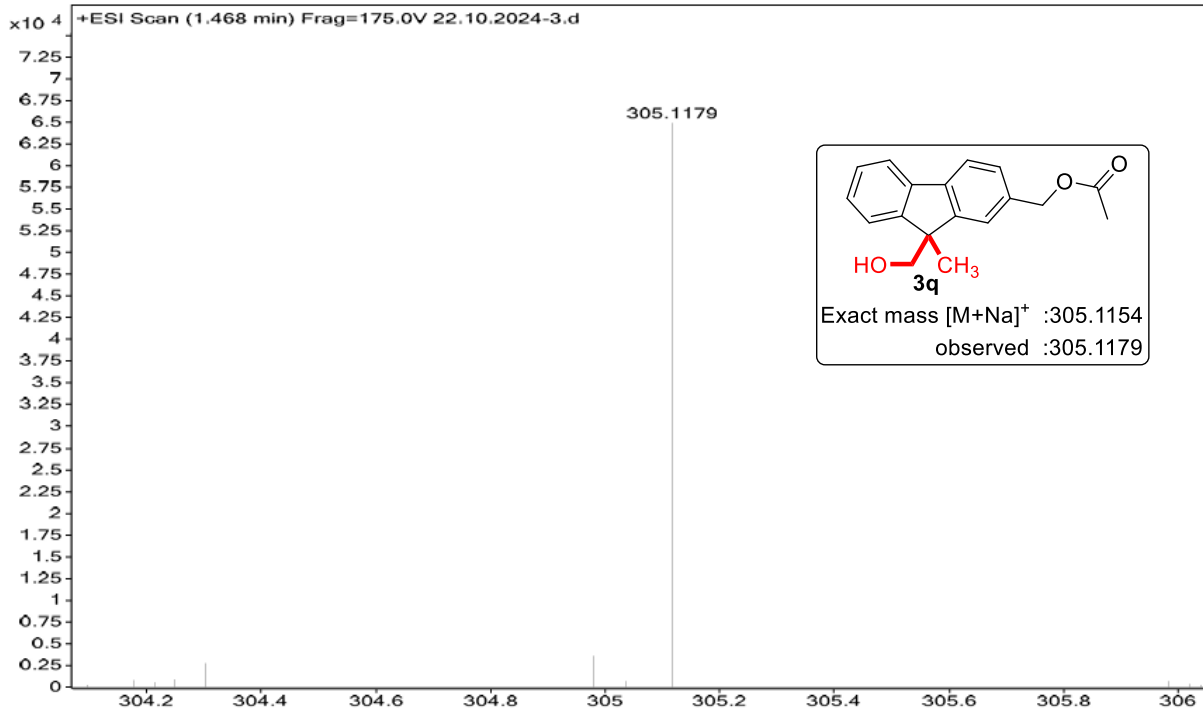


¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) spectrum of (9-(Hydroxymethyl)-9-methyl-9*H*-fluoren-2-yl)methyl acetate (3q)

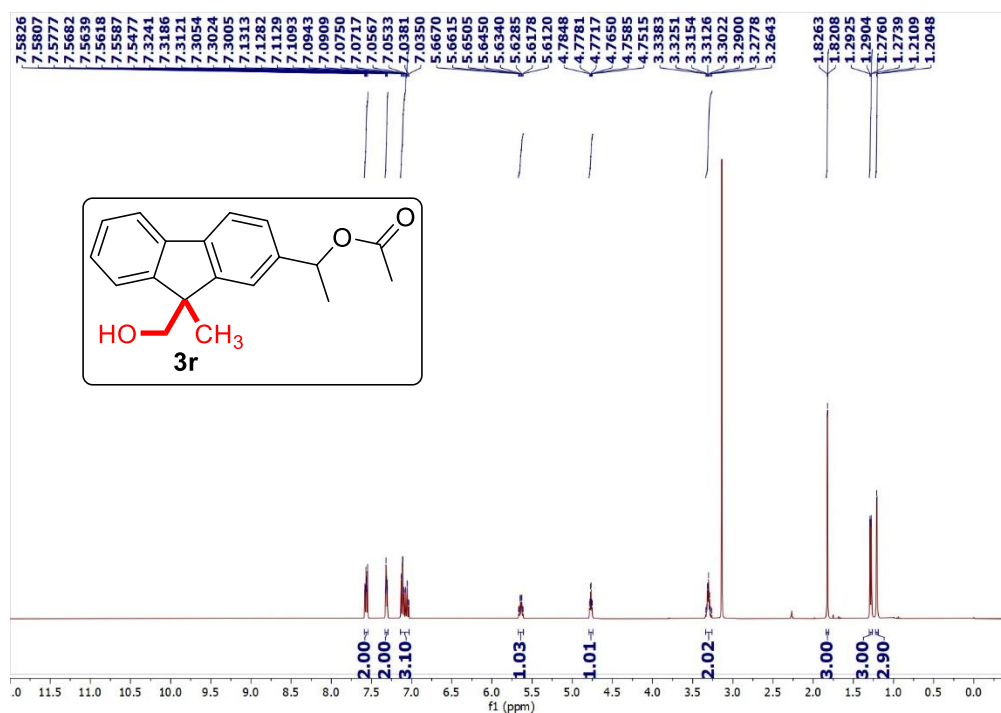


HRMS of (9-(Hydroxymethyl)-9-methyl-9H-fluoren-2-yl)methyl acetate (3q)

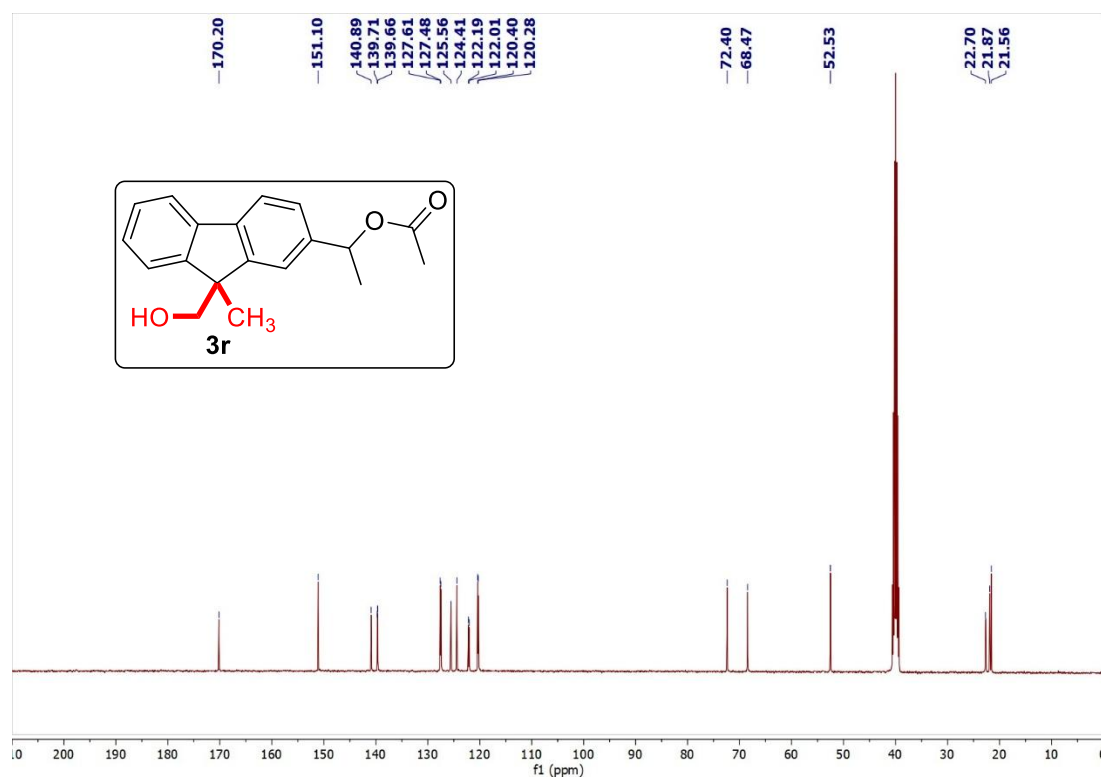
Sample Name	khp-kr-f20	Position	P1-A3	Instrument Name	Instrument 1	User Name	
Inj Vol	2	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	22.10.2024-3.d	ACQ Method	M60 W40.m	Comment		Acquired Time	22-10-2024 13:31:34



¹H NMR (400 MHz, DMSO-*d*₆) spectrum of 1-(9-(Hydroxymethyl)-9-methyl-9*H*-fluoren-2-yl)ethyl acetate (3r)

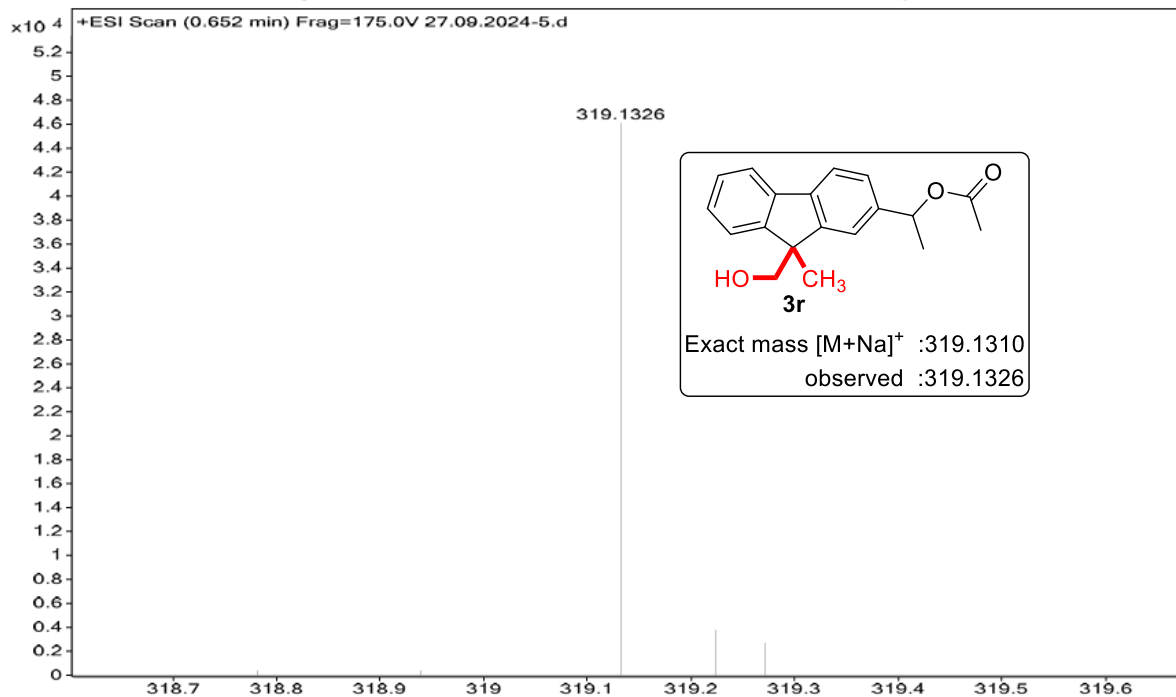


¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) spectrum of 1-(9-(Hydroxymethyl)-9-methyl-9*H*-fluoren-2-yl)ethyl acetate (3r)

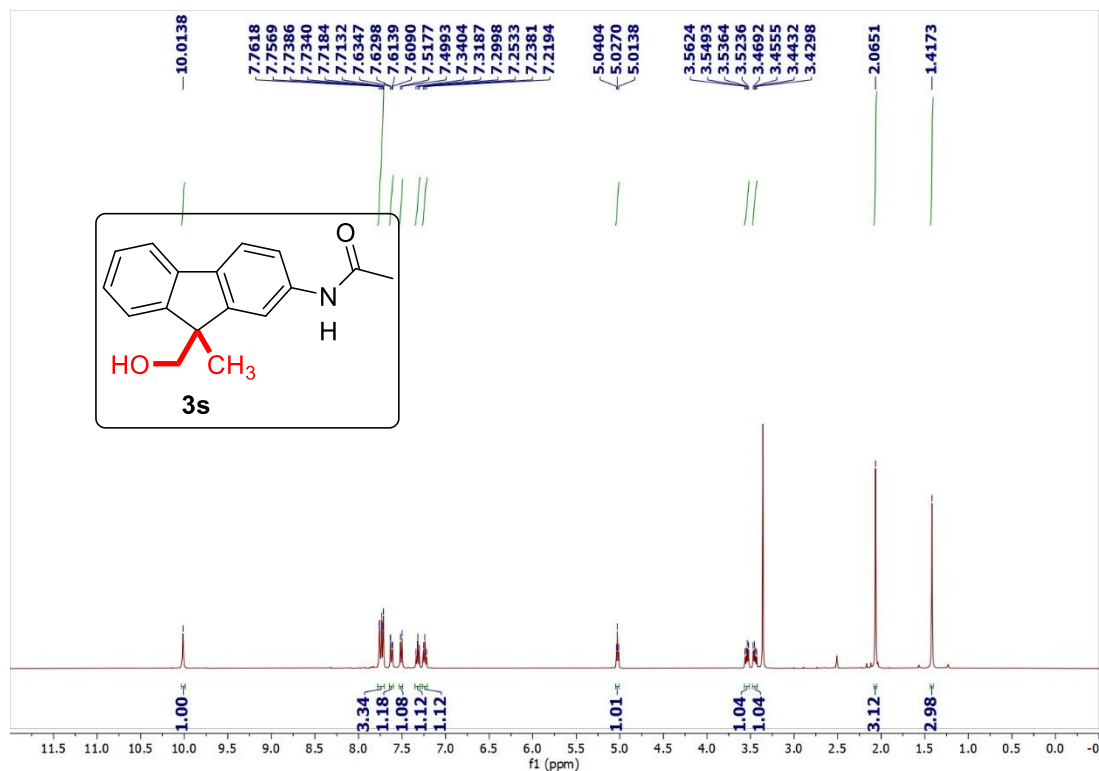


HRMS of 1-(9-(Hydroxymethyl)-9-methyl-9H-fluoren-2-yl)ethyl acetate (3r)

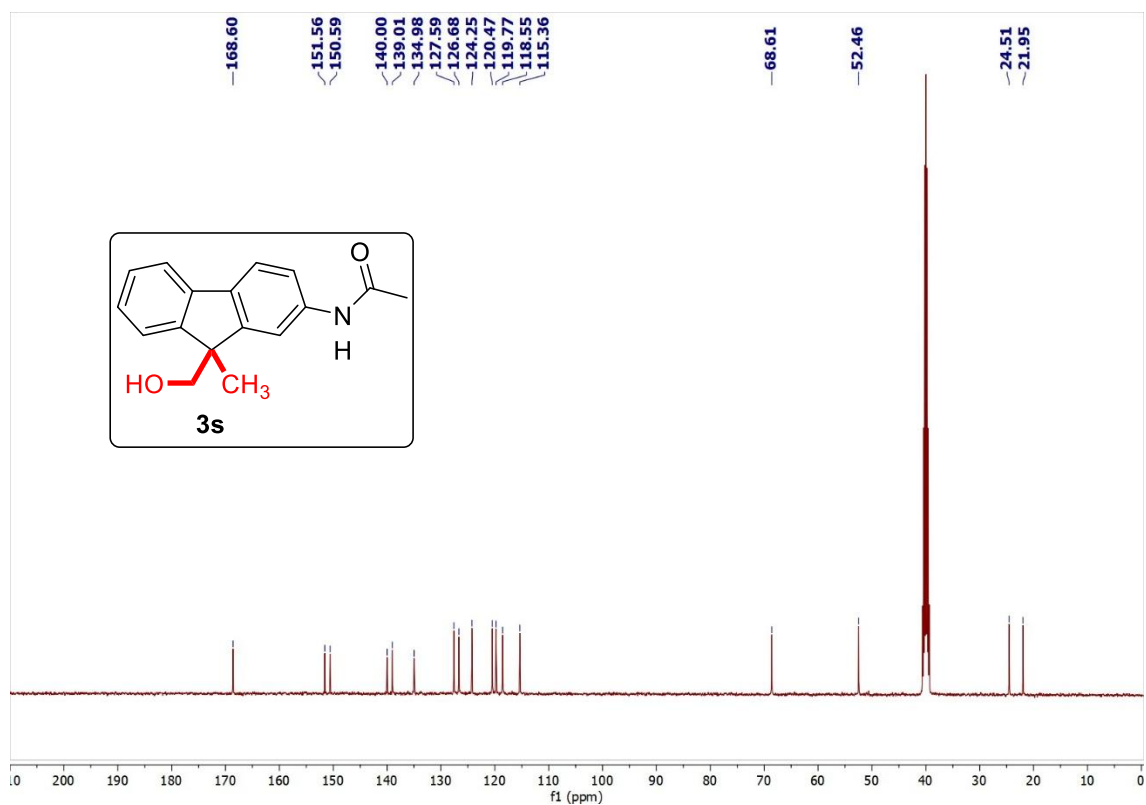
Sample Name	khp-kr-f4	Position	P1-A5	Instrument Name	Instrument 1	User Name	
Inj Vol	2	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	27.09.2024-5.d	ACQ Method	M60 W40.m	Comment		Acquired Time	27-09-2024 13:06:49



¹H NMR (400 MHz, DMSO-*d*₆) spectrum of N-(9-(Hydroxymethyl)-9-methyl-9H-fluoren-2-yl)acetamide (3s)

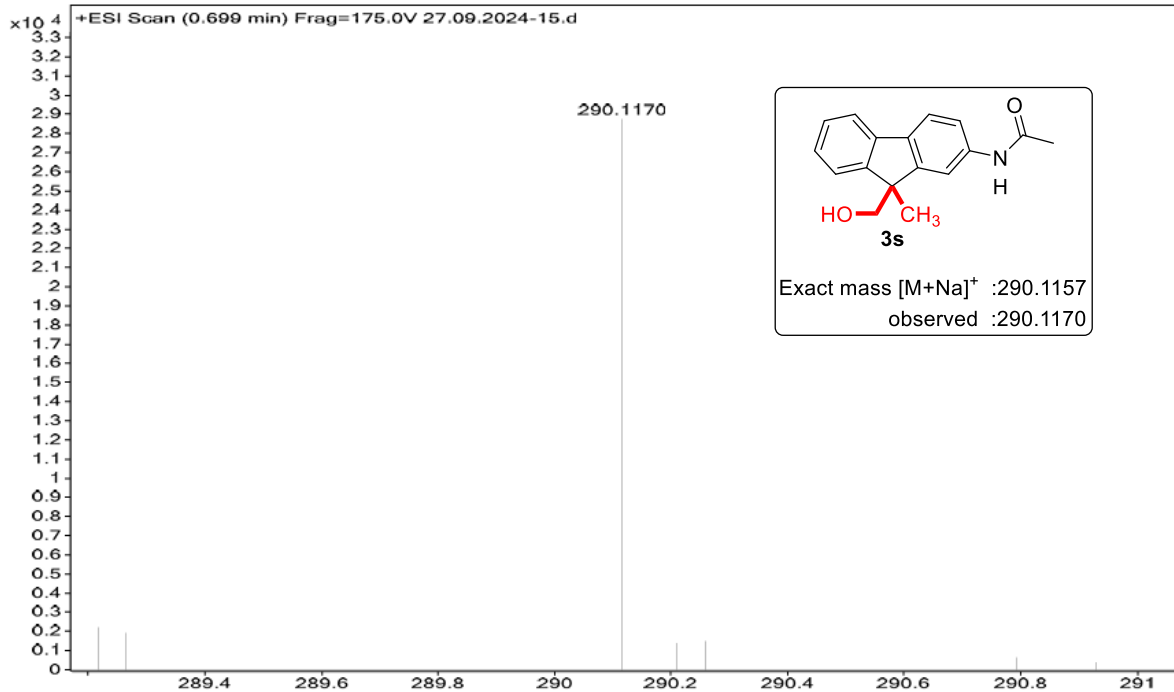


¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) spectrum of N-(9-(Hydroxymethyl)-9-methyl-9H-fluoren-2-yl)acetamide (3s)

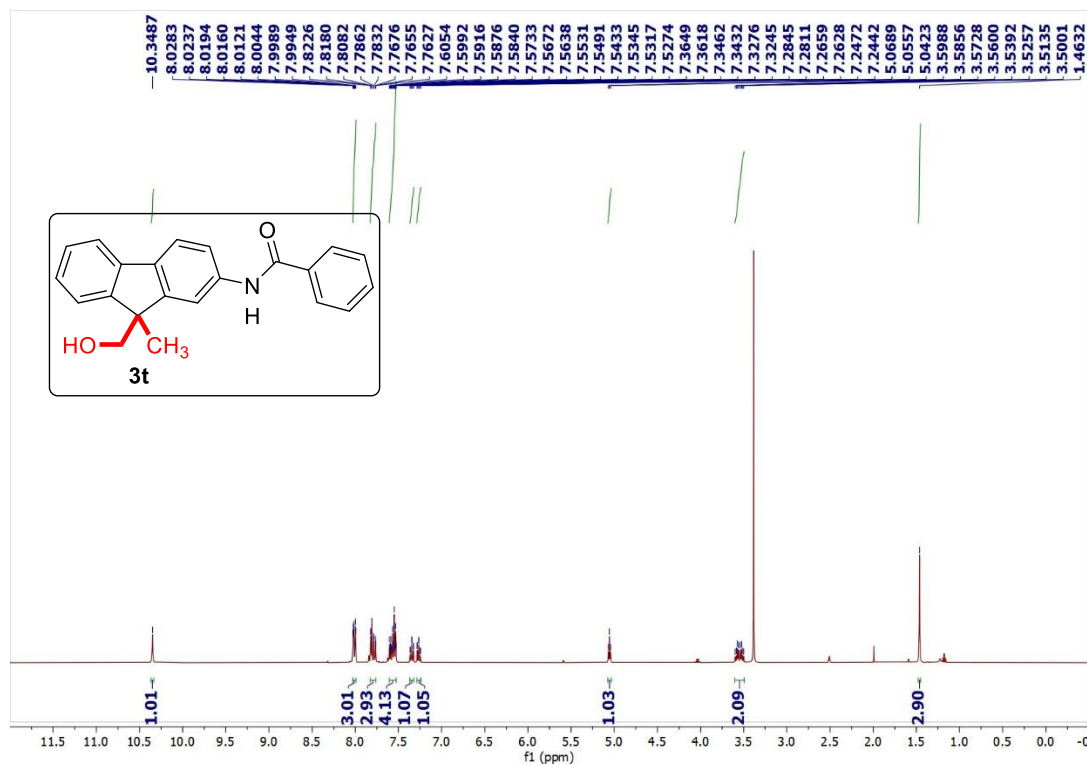


HRMS of spectrum of N-(9-(Hydroxymethyl)-9-methyl-9H-fluoren-2-yl)acetamide (3s)

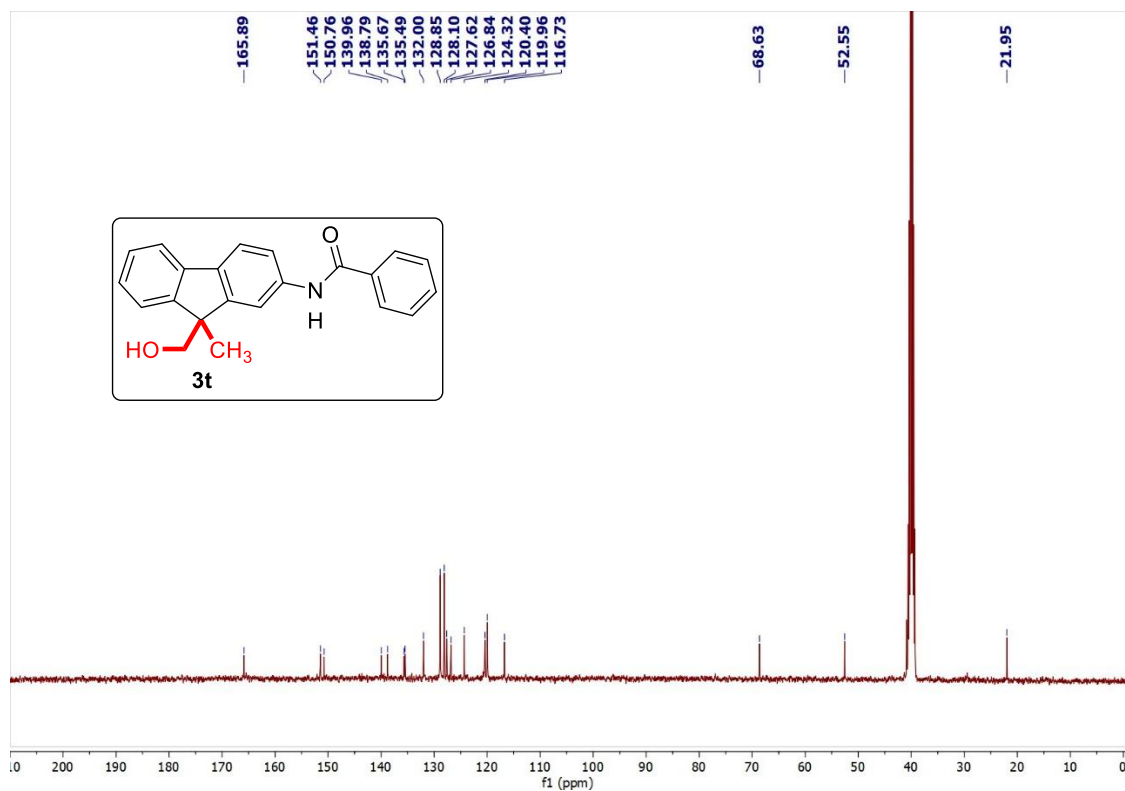
Sample Name	khp-kr-f14	Position	P1-B6	Instrument Name	Instrument 1	User Name	
Inj Vol	2	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	27.09.2024-15.d	ACQ Method	M60 W40.m	Comment		Acquired Time	27-09-2024 13:47:01



¹H NMR (400 MHz, DMSO-*d*₆) spectrum of N-(9-(Hydroxymethyl)-9-methyl-9H-fluoren-2-yl)benzamide (3t)

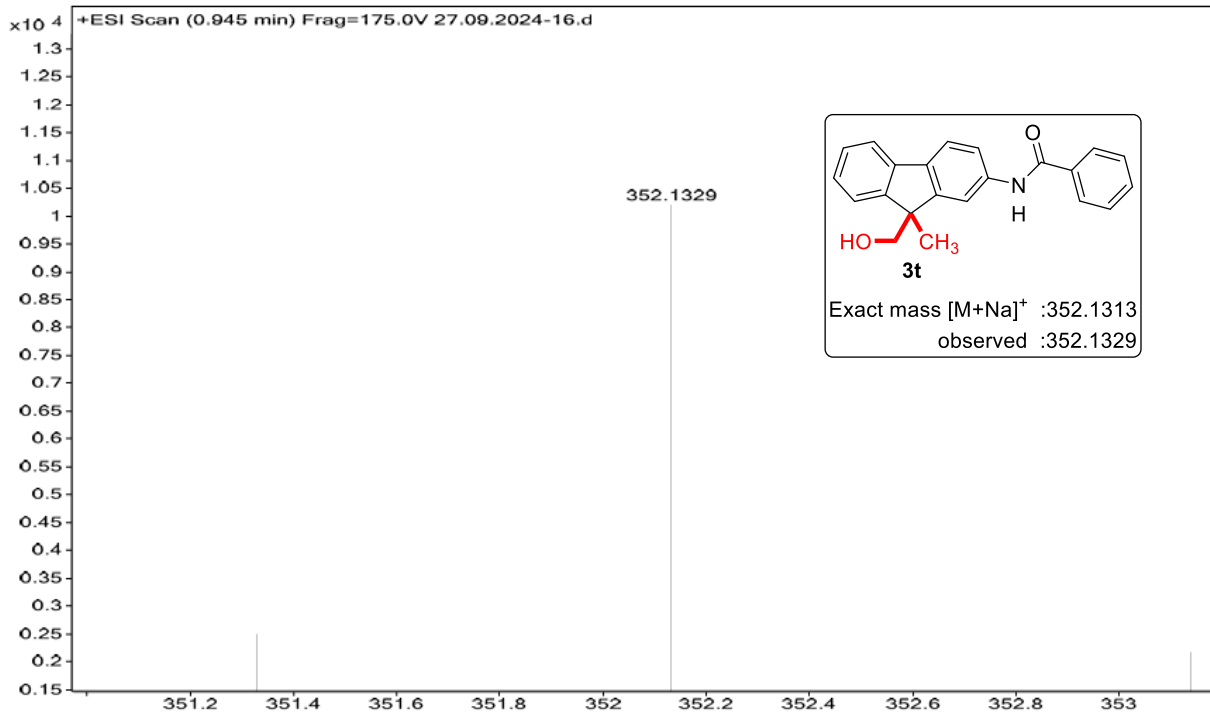


¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) spectrum of N-(9-(Hydroxymethyl)-9-methyl-9H-fluoren-2-yl)benzamide (3t)

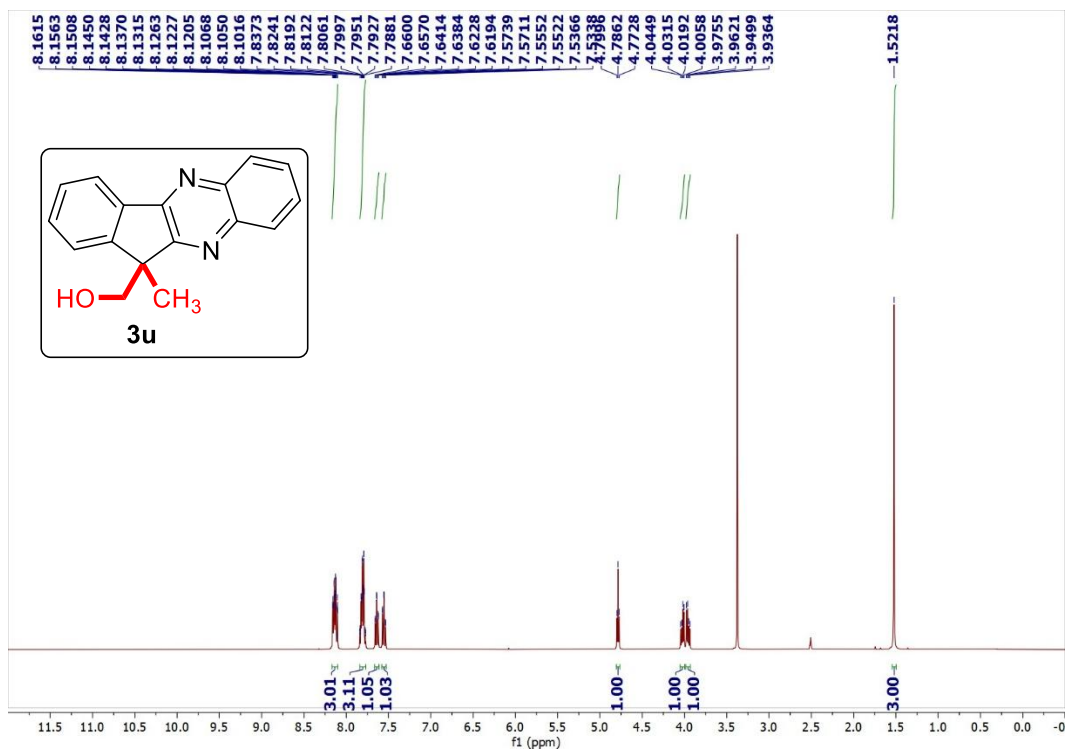


HRMS of N-(9-(Hydroxymethyl)-9-methyl-9H-fluoren-2-yl)benzamide (3t)

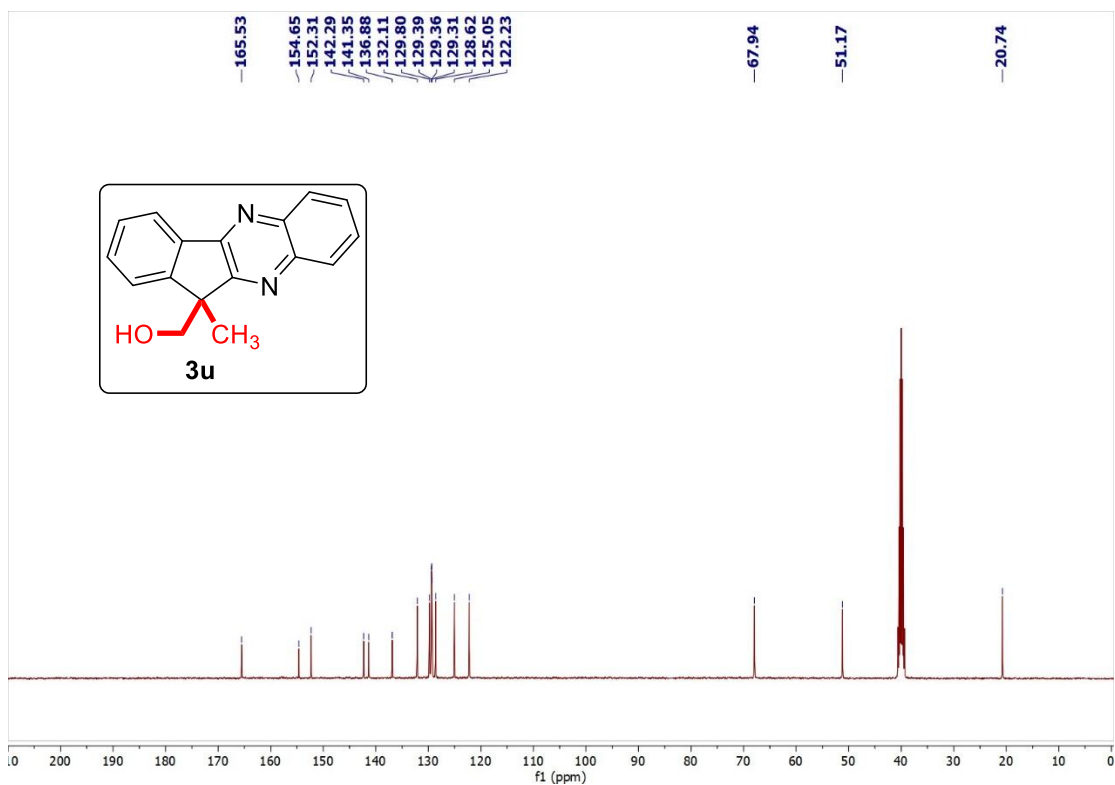
Sample Name	khp-kr-f15	Position	P1-B7	Instrument Name	Instrument 1	User Name	
Inj Vol	2	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	27.09.2024-16.d	ACQ Method	M60 W40.m	Comment		Acquired Time	27-09-2024 13:51:08



¹H NMR (400 MHz, DMSO-*d*₆) spectrum of (11-Methyl-11*H*-indeno[1,2-*b*]quinoxalin-11-yl)methanol (3u)

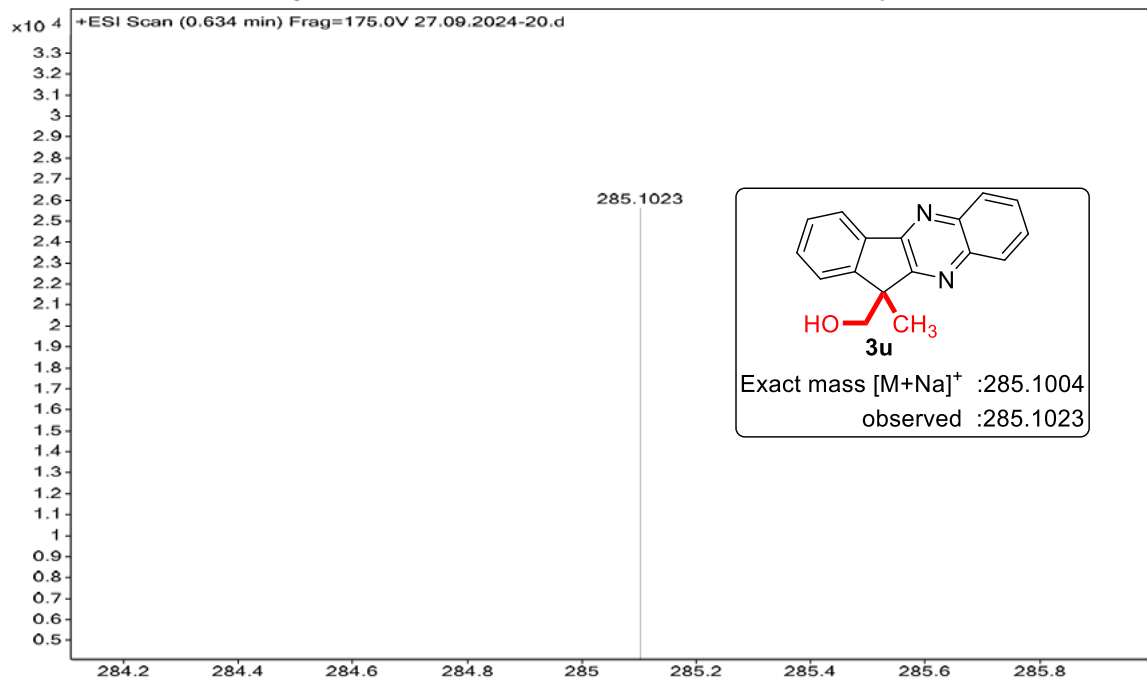


¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) spectrum of (11-Methyl-11*H*-indeno[1,2-*b*]quinoxalin-11-yl)methanol (3u)

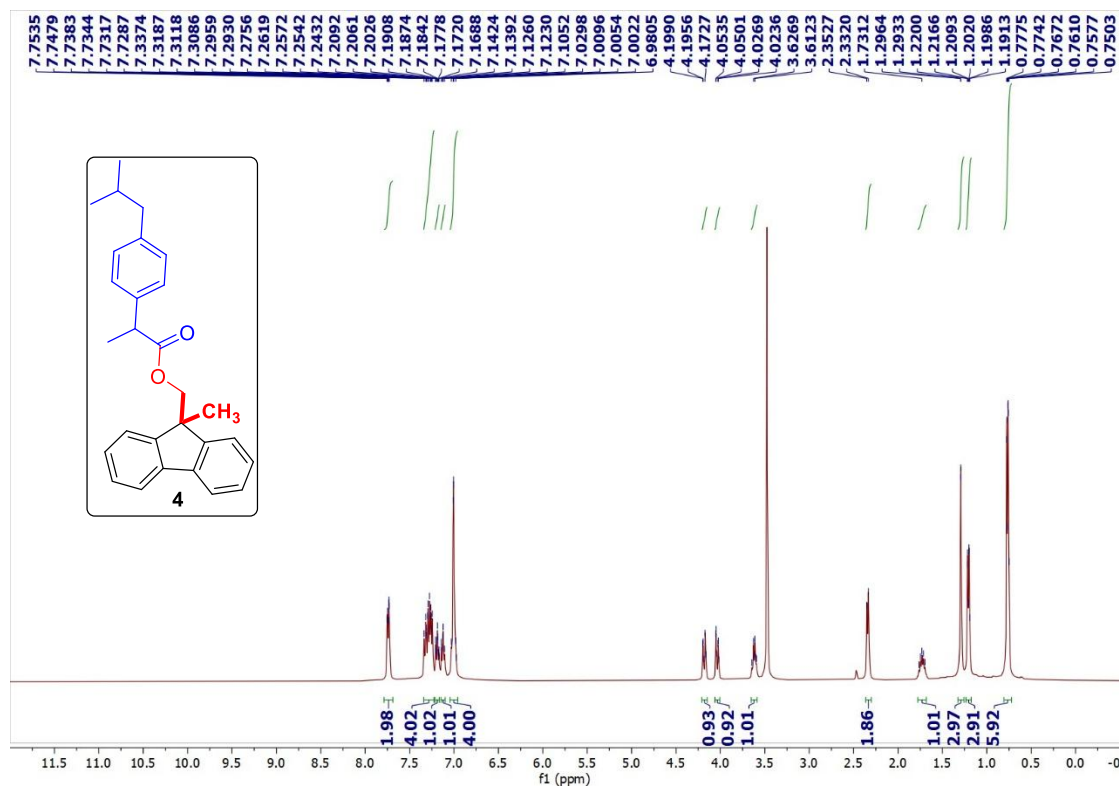


HRMS of (11-Methyl-11H-indeno[1,2-b]quinoxalin-11-yl)methanol (3u)

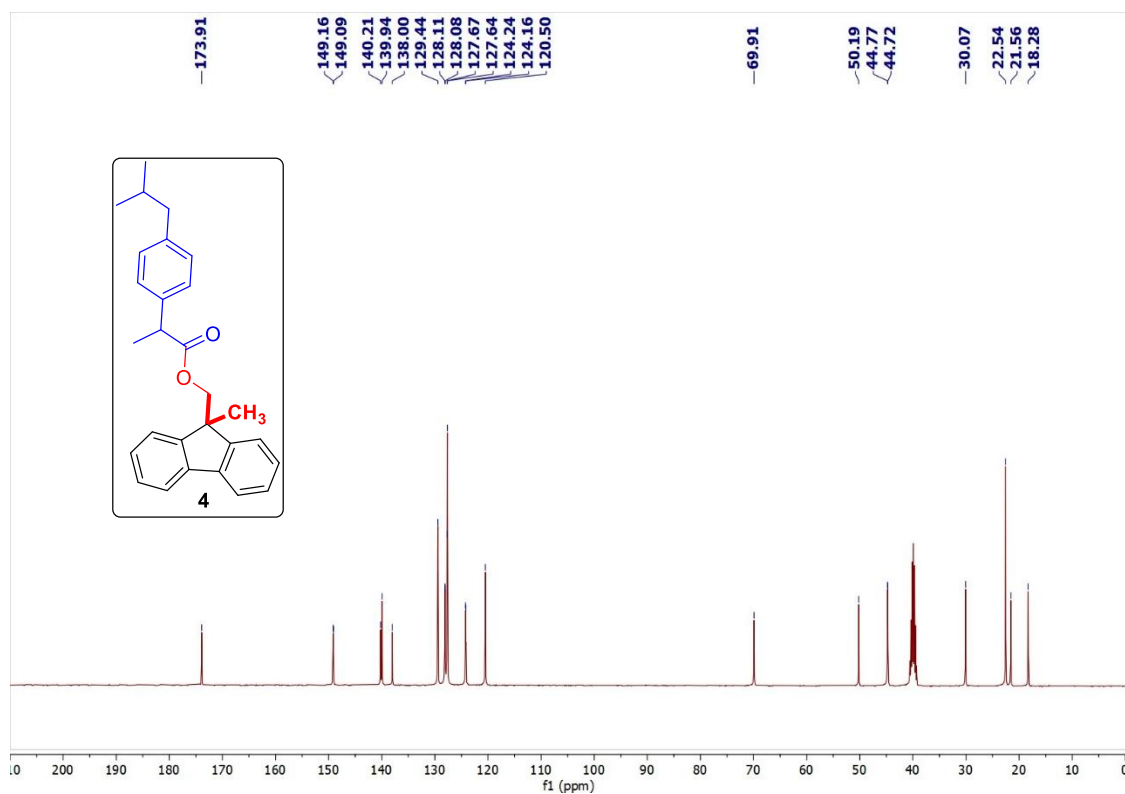
Sample Name	khp-kr-f19	Position	P1-C2	Instrument Name	Instrument 1	User Name	
Inj Vol	2	InjPosition		SampleType	Sample	IRM Calibration Status	Success
Data Filename	27.09.2024-20.d	ACQ Method	M60 W40.m	Comment		Acquired Time	27-09-2024 14:07:08



¹H NMR (400 MHz, DMSO-*d*₆) spectrum of (9-methyl-9*H*-fluoren-9-yl)methyl 2-(4-isobutylphenyl)propanoate (4)

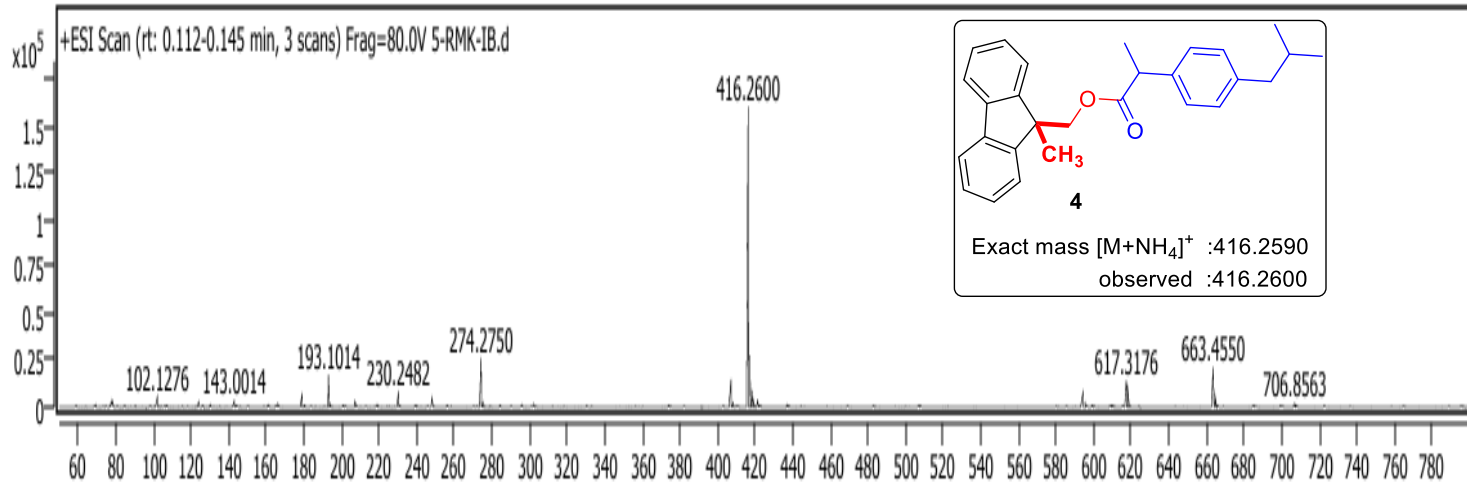


¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) spectrum of (9-methyl-9*H*-fluoren-9-yl)methyl 2-(4-isobutylphenyl)propanoate (4)

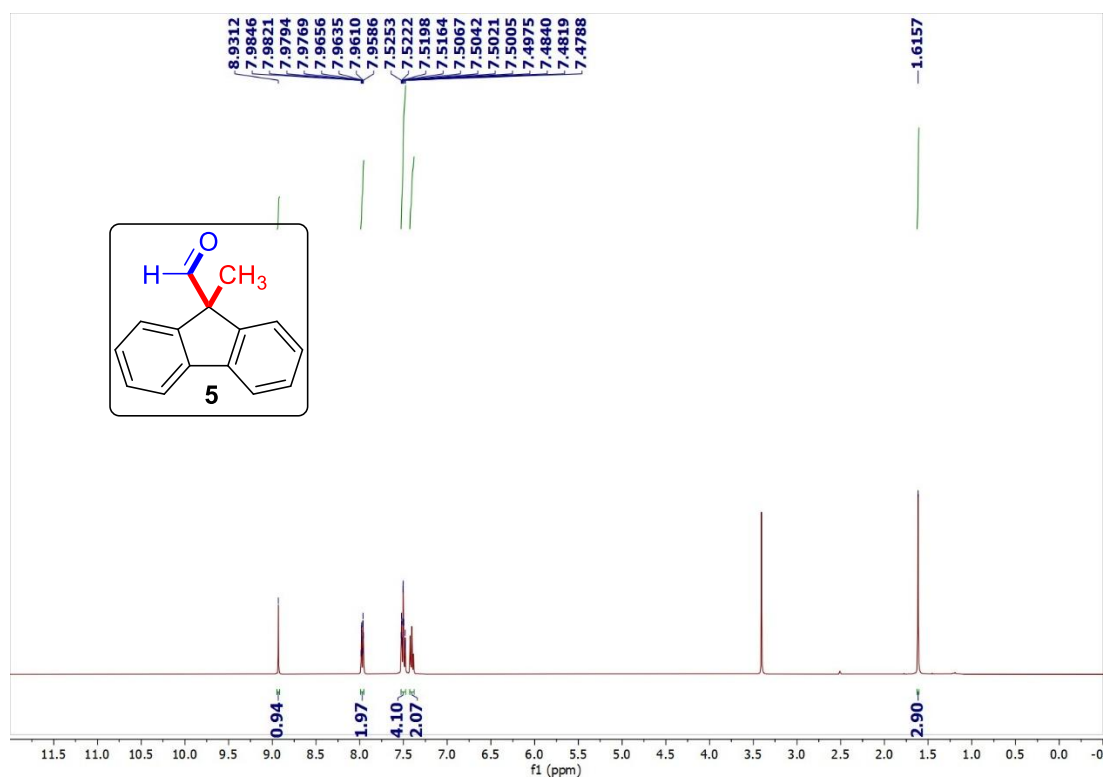


HRMS of (9-methyl-9H-fluoren-9-yl)methyl 2-(4-isobutylphenyl)propanoate (4)

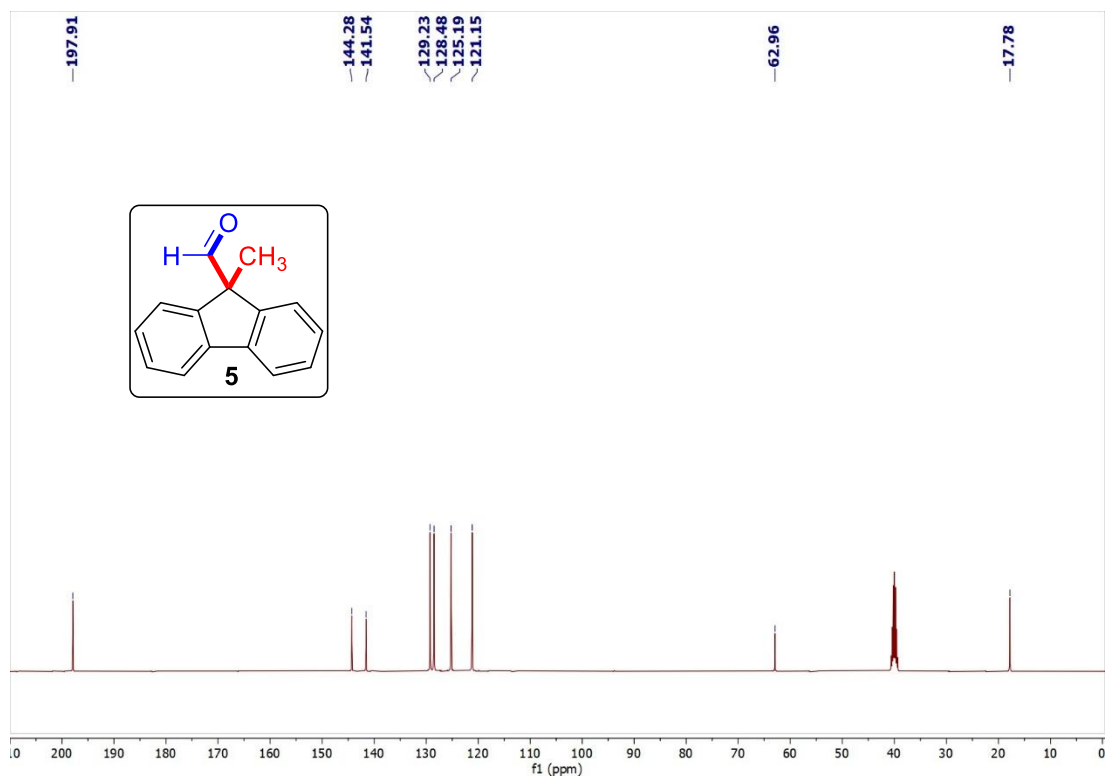
+ Scan (rt: 0.112-0.145 min)



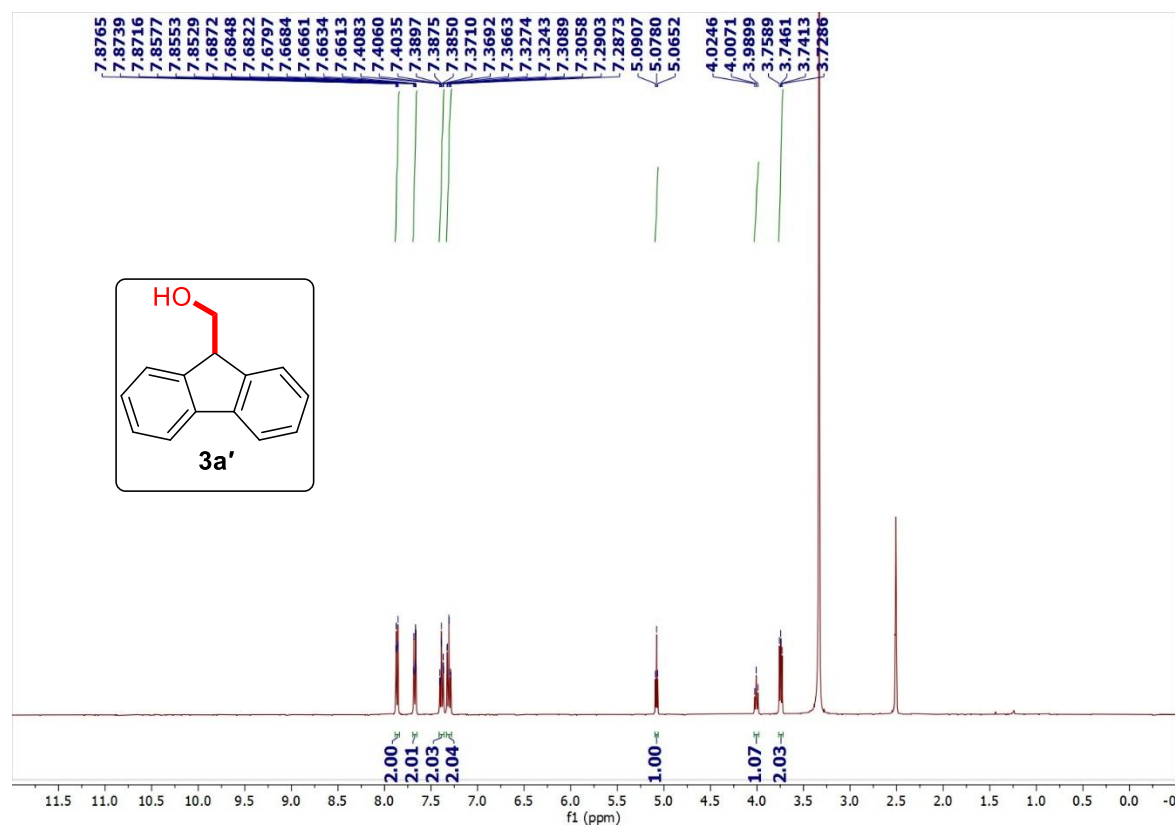
^1H NMR (400 MHz, $\text{DMSO-}d_6$) spectrum of 9H-fluorene-9-carbaldehyde (4b)



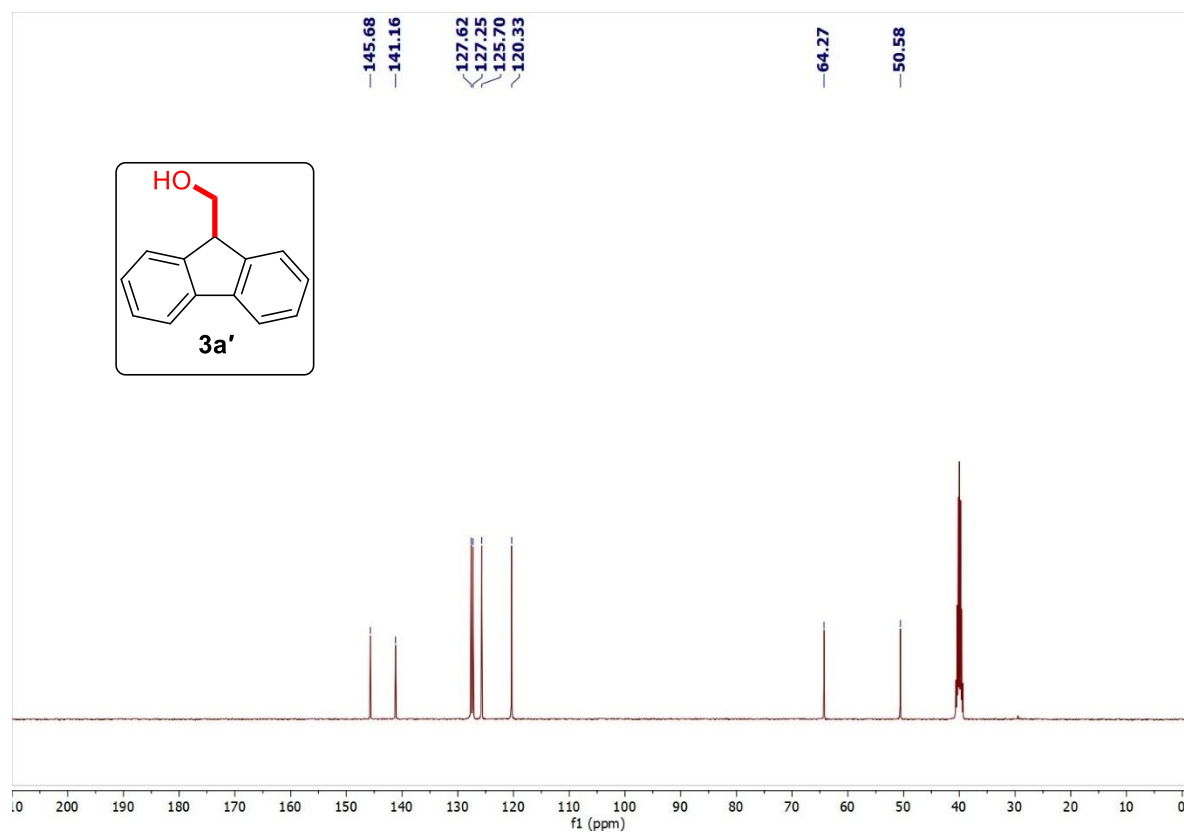
$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, $\text{DMSO-}d_6$) spectrum of 9H-fluorene-9-carbaldehyde (4b)



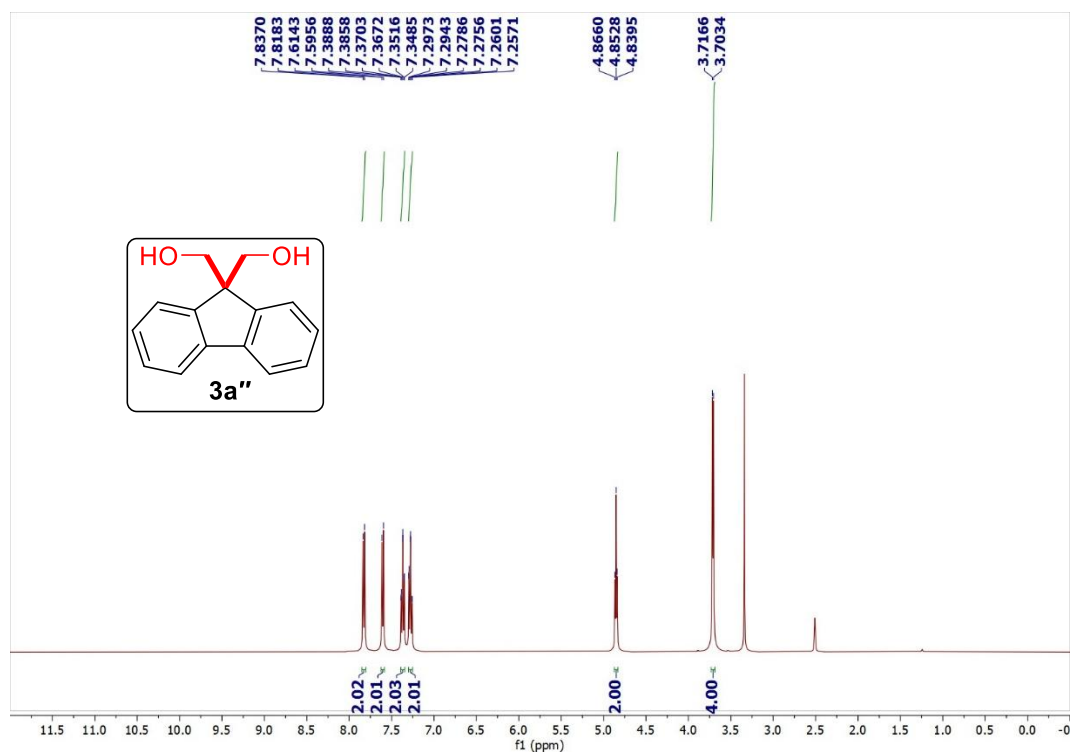
¹H NMR (400 MHz, DMSO-*d*₆) spectrum of (9*H*-fluoren-9-yl)methanol (3a')



¹³C{¹H} NMR (100 MHz, DMSO-*d*₆) spectrum of (9*H*-fluoren-9-yl)methanol (3a')



^1H NMR (400 MHz, $\text{DMSO-}d_6$) spectrum of (9*H*-fluorene-9,9-diyl)dimethanol (3a'')



$^{13}\text{C}\{^1\text{H}\}$ NMR (100 MHz, $\text{DMSO-}d_6$) spectrum of (9*H*-fluorene-9,9-diyl)dimethanol (3a'')

