Supporting Information

Overhauser Dynamic Nuclear Polarization with Selectively Deuterated BDPA Radicals

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1. General Information about Synthesis

Unless otherwise stated, all reactions were carried out under an atmosphere of nitrogen using the standard Schlenk technique. All solvents and reagents were obtained from commercial sources and were purified following standard procedures before use if necessary.

NMR spectra were measured on Bruker Avance III HD 600 MHz, spectrometer. Proton chemical shifts are expressed in parts per million (ppm, δ scale) and are referenced to the residual proton in the NMR solvents $CDCl_3$ at δ 7.26 or DMSO-d_6 at 2.50 ppm. ^{1}H NMR spectroscopic data are reported as follows: Chemical shift in ppm (multiplicity, coupling constants J (Hz), integration intensity, assigned proton). The multiplicities are abbreviated with s (singlet), d (doublet), t (triplet), m (multiplet). All ^{13}C spectra recorded are proton-decoupled. The carbon chemical shifts are expressed in parts per million (ppm, δ scale) and are referenced to the carbon resonance of the NMR solvents $CDCl_3$ at δ 77.16 or DMSO at 39.52 ppm. ^{13}C NMR spectroscopic data are reported as follows: Chemical shift in ppm (multiplicity, coupling constants J (Hz), assigned carbon). All raw fid files were processed and the spectra analyzed using the program MestReNOVA 14.2 from Mestrelab Supporting Information SI4 Research S. L.

Deuterium incorporation <100 % at specific positions will result in non-deuterated analogs of targeted compounds. These impurities will appear on NMR spectra and therefore need to be distinguished from the desired selectively deuterated compounds. Hence, for ^{1}H NMR, two set of signals are reported, one corresponding to the desired product, labelled Deuterated Product (**DP**), and the other corresponding to the non-deuterated impurity, labelled Non-Deuterated Impurity (**NDI**). Overlap between both products signals occurred, and the reported signals are from the peaks that could be resolved. Deuterium incorporation was calculated from the integrated ratio of well-defined ^{1}H NMR signals that are known to be associated with both compounds. For clarity purposes, ^{13}C NMR signals were only reported for the main product, but the signals from non-deuterated impurities can be observed in spectra with high signal-to-noise ratio (**SNR**). The chemical shifts of the deuterated products and non-deuterated impurities singlet differ due to the ^{1}H/^{2}H isotope effect. Signals from carbon atoms directly attached to deuterium atoms are triplet or multiplet due to deuterium coupling. Due to low SNR or the complexity of the molecules further down the synthetic route, some signals could or should not be assigned as triplets, and were assigned as multiplets.

High-resolution mass spectra were recorded on a JEOL AccuTOF LC-Plus 46 with an ionSense DART system.

2. Synthesis of Selectively Deuterated Fluorene Moieties

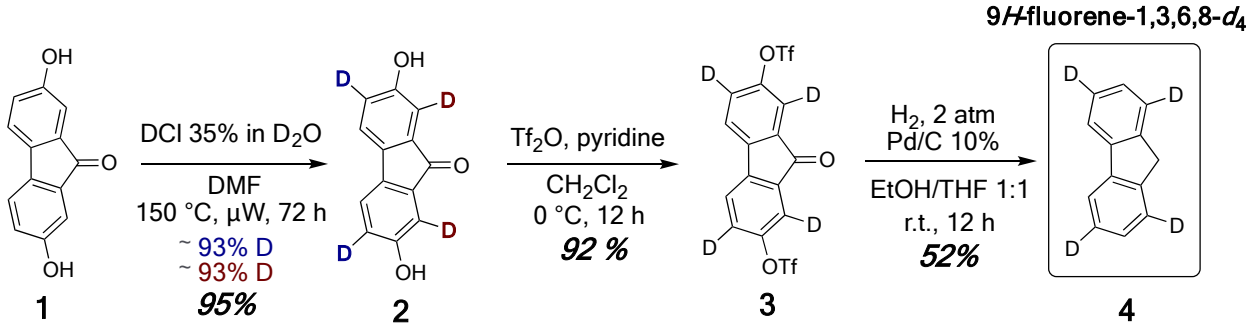


Figure S1. General synthetic route for **9*H*-fluorene-1,3,6,8-d_4**

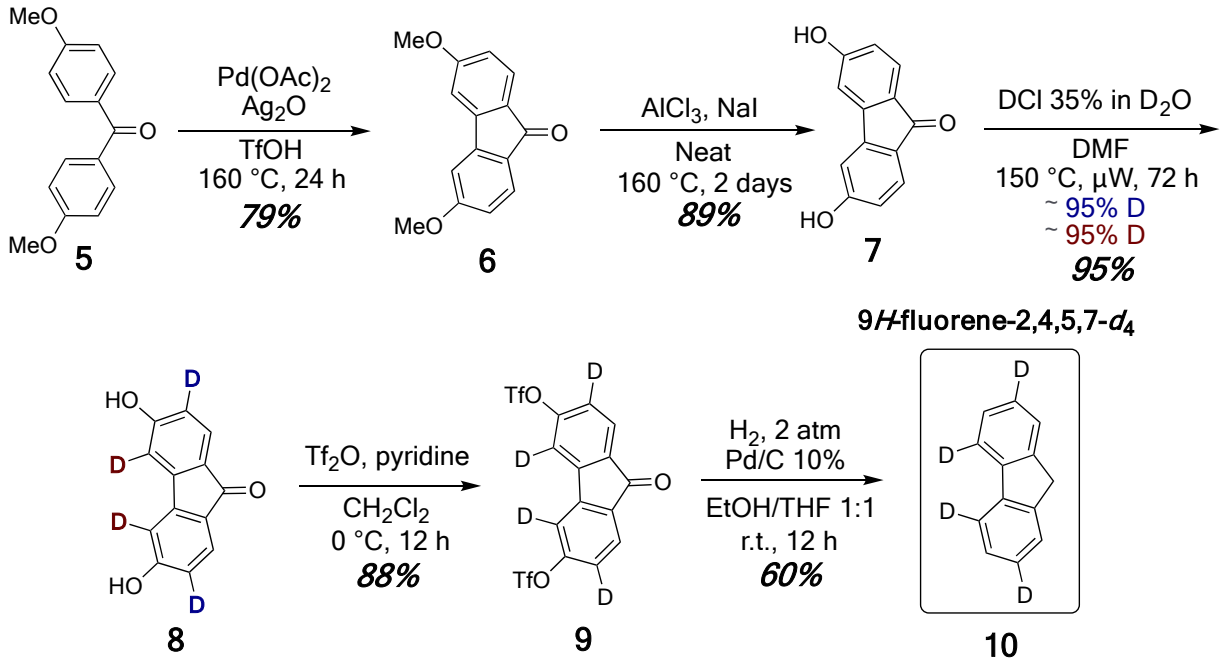
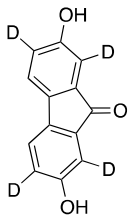


Figure S2. General synthetic route for **9*H*-fluorene-2,4,5,7-d_4**

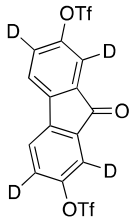
Synthesis of 9*H*-fluorene-1,3,6,8-d_4



2,7-dihydroxy-9*H*-fluoren-9-one-1,3,6,8-d_4 (2)
In a 20 mL microwave reaction vial with a magnetic stirrer bar, 2,7-di(hydroxy-*d*)-9*H*-fluoren-9-one (600 mg, 2.83 mmol) was added, followed by 35 wt.% DCl solution in D_2O (5 mL) and DMF (5 mL) under an argon atmosphere. The vial was sealed and heated in the microwave synthesis apparatus for 24h at 150 °C. The mixture was cooled to room temperature and an additional 3 mL of 35 wt.% DCl solution in D_2O was added. The reaction was suggested again to microwave irradiation at 150°C for 12h to reach a higher deuterium incorporation. Reaction follow-up was done by ^{1}H-NMR. Then, the mixture was cooled to room temperature and was added to of ice-cold H_2O (20 mL), forming a reddish precipitate. The precipitate was filtered on a fritted funnel and, washed with hexanes (3x10 mL), saturated aqueous sodium bicarbonate solution (3x20 mL) and water (3x20 mL). The solid residue was dried over $MgSO_4$ and then dried overnight in a vacuum oven at 100°C to yield 2,7-dihydroxy-9*H*-fluoren-9-one-1,3,6,8-d_4 as a red solid (580 mg, 95%). The total incorporation yield for both positions was determined by ^{1}H NMR spectroscopy relative to the intensity of a nonexchangeable proton in the molecule, and was further confirmed by LC-MS analysis. Deuterium incorporation (Position 1 and 8: ~93%; Position 3 and 6: ~93%).

DP:
^{1}H NMR (600 MHz, DMSO-d_6) δ 9.83 (s, 2H), 7.37 (s, 2H).
^{13}C NMR (151 MHz, DMSO-d_6) δ 193.40, 157.47, 135.68, 135.06, 120.96, 120.70 (m), 110.83 (m).
HRMS (DART/AccuTOF) m/z: $[M]^+$ Calcd for $C_{13}H_4D_4O_3$ 217.0758; Found 217.0769.

NDI:
^{1}H NMR (600 MHz, DMSO-d_6) δ 6.88 (d, J = 2.6 Hz, 0.14H), 6.86 (dd, J = 8.0, 2.3 Hz, 0.14H).

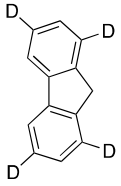


9-oxo-9*H*-fluorene-2,7-diyl-1,3,6,8-d_4 bis(trifluoromethanesulfonate) (3)
To a solution of 2,7-di(hydroxy-*d*)-9*H*-fluoren-9-one-1,3,6,8-d_4 (550 mg, 2.54 mmol) and pyridine (1 mL) in dichloromethane (8 mL) was added a 1M solution of Tf_2O in DCM (6.35 mL, 6.35 mmol) at 0°C. The stirring was continued overnight at room temperature before it was poured into water and the organic phase was extracted with dichloromethane (3x10 mL). The combined organic phases were washed subsequently with 3% aqueous HCl solution (2x10 mL), saturated aqueous sodium bicarbonate solution (1x10 mL) and brine (2x10 mL) and then dried over $MgSO_4$. The reaction mixture was concentrated in vacuo and the purple residue was directly purified by flash chromatography on silica gel (hexanes/ethyl acetate, 90:10) to yield 9-oxo-9*H*-fluorene-2,7-diyl-1,3,6,8-d_4 bis(trifluoromethanesulfonate**)** as a purple solid (1.12 g, 92%).

DP:
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.66 (s, 2H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 189.26, 150.46, 142.72, 136.32, 127.73 (t, *J* = 25.5 Hz,) 122.41, 119.89, 118.36, 118.12 (t, *J* = 25.5 Hz), 117.76, 115.64.
HRMS (DART/AccuTOF) m/z: $[M]^+$ Calcd for $C_{15}H_2D_4F_6O_7S_2$ 480.9744; Found 480.9774.

NDI:
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.61 (d, *J* = 1.8 Hz, 0.13H), 7.46 (dd, *J* = 8.1, 1.9 Hz, 0.13H).

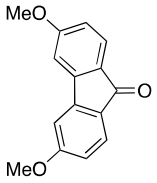


9*H*-fluorene-1,3,6,8-d_4 (4)
To a round-bottom flask under an argon atmosphere, 10 wt.% palladium on carbon (300 mg, 0.28 mmol) was added, then a 1:1 mixture of THF/MeOH (10 mL) was added slowly and the reaction was degassed with argon for 20 min. Then, 9-oxo-9*H*-fluorene-2,7-diyl-1,3,6,8-d_4 bis(trifluoromethanesulfonate) (1.0 g, 2.08 mmol) was added and the reaction was bubbled with 2 atmosphere of hydrogen gas for 20 min. The reaction was sealed and left to stir for 24h or until complete consumption of starting material. The reaction was followed by TLC using fluorene as co-spot. The reaction was then filtered on Celite and the reaction mixture was concentrated in vacuo to afford a beige residue. Further purification by flash chromatography on silica gel (hexanes/ethyl acetate, 95:5) yielded 9*H*-fluorene-1,3,6,8-d_4 as a white solid (184 mg, 52%).

DP:
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.82 (s, 2H), 7.33 (s, 2H), 3.93 (s, 2H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 143.26, 141.84, 126.61, 126.56 (t, 24.5 Hz), 124.86 (t, 24.5 Hz), 119.89, 36.97.
HRMS (DART/AccuTOF) m/z: $[M]^+$ Calcd for $C_{13}H_6D_4$ 171.1067; Found 171.0933.

NDI:
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.57 (dd, *J* = 7.4, 1.0 Hz, 0.14H), 7.40 (t, *J* = 7.5 Hz, 0.14H).

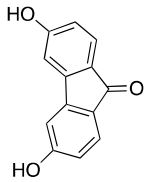
Synthesis of 9*H*-fluorene-2,4,5,7-d_4



3,6-dimethoxy-9*H*-fluoren-9-one (6)
Synthesized following a modified literature procedure[1]. In a microwave reaction vial with a magnetic stirrer bar, 4,4'-dimethoxybenzophenone (5.0 g, 20.6 mmol), palladium(II) acetate (463 mg, 2.06 mmol), and silver(I) oxide (7.05 g, 30.45 mmol) was added, followed by trifluoroacetic acid (15 mL) under an argon atmosphere. The vial was sealed and heated in the microwave

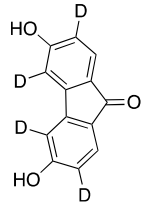
synthesis apparatus for 12h at 160 °C. After completion of the reaction, the reaction mixture was cooled to room temperature, filtered through a short Celite pad and was washed several times with dichloromethane. The combined filtrate was concentrated in vacuo and purified by flash chromatography on silica gel (hexanes/ethyl acetate, 70:30) to yield 3,6-dimethoxy-9*H*-fluoren-9-one as a pale-yellow solid (3.92 g, 79%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.58 (d, J = 8.2 Hz, 2H), 7.00 (d, J = 2.2 Hz, 2H), 6.75 (dd, J = 8.2, 2.2 Hz, 2H), 3.90 (s, 6H).



3,6-dihydroxy-9*H*-fluoren-9-one (7)
Synthesized following a modified literature procedure[2]. In an unsealed 50 mL pressure vial, 3,6-dimethoxy-9*H*-fluoren-9-one (2.0 g, 8.32 mmol), $AlCl_3$ (3.33 g, 24.97 mmol) and NaI (6.24 g, 41.6 mmol) were heated at 160 °C for 12 h under neat conditions. The crude was cooled to room temperature and deionized H_2O (50 mL) was added. The mixture was extracted with 3 x 20 mL of ethyl acetate and the organic phase was dried over $MgSO_4$, concentrated in vacuo and purified by flash chromatography on silica gel (dichloromethane/ethyl acetate, 95:5) to yield 3,6-dihydroxy-9*H*-fluoren-9-one as a white solid (1.57 g, 89%).

^{1}H NMR (600 MHz, Acetone-d_6) δ 9.40 (s, 2H), 7.42 (d, J = 8.1 Hz, 2H), 7.18 (d, J = 2.3 Hz, 2H), 6.81 (dd, J = 8.1, 2.2 Hz, 2H).

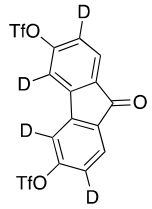


3,6-dihydroxy-9*H*-fluoren-9-one-2,4,5,7-d_4 (8)
In a 20 mL microwave reaction vial with a magnetic stirrer bar, 3,6-di(hydroxy-*d*)-9*H*-fluoren-9-one (600 mg, 2.83 mmol) was added, followed by 35 wt.% DCl solution in D_2O (5 mL) and DMF (5 mL) under an argon atmosphere. The vial was sealed and heated in the microwave synthesis apparatus for 24h at 150 °C. The mixture was cooled to room temperature and was added to of ice-cold H_2O (20 mL), forming a yellow precipitate. The precipitate was filtered on a fritted funnel and, washed with hexanes (3x10 mL), saturated aqueous sodium bicarbonate solution (1x10 mL) and water (3x20 mL). The solid residue was dried overnight in a vacuum oven at 100°C to yield 3,6-dihydroxy-9*H*-fluoren-9-one-2,4,5,7-d_4 as a yellow solid (580 mg, 95%). The total incorporation yield for both positions was determined by ^{1}H NMR spectroscopy relative to the intensity of a nonexchangeable proton in the molecule, and was further confirmed by LC-MS analysis. Deuterium incorporation (Position 2 and 7: ~95%; Position 4 and 5: ~95%)

DP:

^{1}H NMR (600 MHz, DMSO-d_6) δ 10.51 (s, 2H), 7.38 (s, 2H).
^{13}C NMR (151 MHz, DMSO-d_6) δ 190.31, 163.59, 145.70, 126.01, 125.33, 114.72 (m), 108.27 (t, 23.5 Hz)
HRMS (DART/AccuTOF) m/z: $[M]^+$ Calcd for $C_{13}H_4D_4O_3$ 217.0758; Found 217.0742.

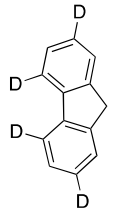
NDI:
^{1}H NMR (600 MHz, DMSO-d_6) δ 7.04 (s, 0.09H), 6.67 (d, J = 8.1 Hz, 0.10H).



9-oxo-9*H*-fluorene-3,6-diyl-2,4,5,7-d_4 bis(trifluoromethanesulfonate) (9)
Prepared according to the general procedure used for compound **2**, 2,7-di(hydroxy-*d*)-9*H*-fluoren-9-one-1,3,6,8-d_4 (500 mg, 2.36 mmol), pyridine (1 mL), dichloromethane (7.5 mL), and 1M solution of Tf_2O in DCM (5.90 mL, 5.90 mmol) were used. Yielding 9-oxo-9*H*-fluorene-3,6-diyl-2,4,5,7-d_4 bis(trifluoromethanesulfonate) as a yellow solid (1.07 g, 88%).

DP:
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.81 (s, 2H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 189.47, 154.02, 145.01, 133.67, 126.57, 122.87 (t, 25.0 Hz), 119.88, 117.76, 114.68 (t, 25.0 Hz).
HRMS (DART/AccuTOF) m/z: $[M]^+$ Calcd for $C_{15}H_2D_4F_6O_7S_2$ 480.9844; Found 480.9842.

NDI:
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.48 (s, 0.08H), 7.30 (d, J = 8.1 Hz, 0.09H).



9H-fluorene-2,4,5,7-d_4 (10)
Prepared according to the general procedure used for compound **3**, 10 wt.% palladium on carbon (240 mg, 0.22 mmol), 1:1 mixture of THF/MeOH (8 mL), 9-oxo-9*H*-fluorene-2,7-diyl-1,3,6,8-d_4 bis(trifluoromethanesulfonate) (800 mg, 1.66 mmol) was used. Yielding 9H-fluorene-2,4,5,7-d_4 as a white solid (170 mg, 60%).

DP:
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.56 (s, 1H), 7.39 (s, 1H), 3.92 (s, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 143.34, 141.75, 126.68, 126.53 (t, 24.5 Hz), 125.05, 119.74 (t, 24.5 Hz), 37.06.
HRMS (DART/AccuTOF) m/z: $[M]^+$ Calcd for $C_{13}H_6D_4$ 171.1067; Found 171.0934.

NDI:
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.81 (d, J = 7.6 Hz, 0.10H), 7.32 (t, J = 7.4 Hz, 0.11H).

3. Synthesis of Selectively Deuterated and Perdeuterated BDPA Radicals

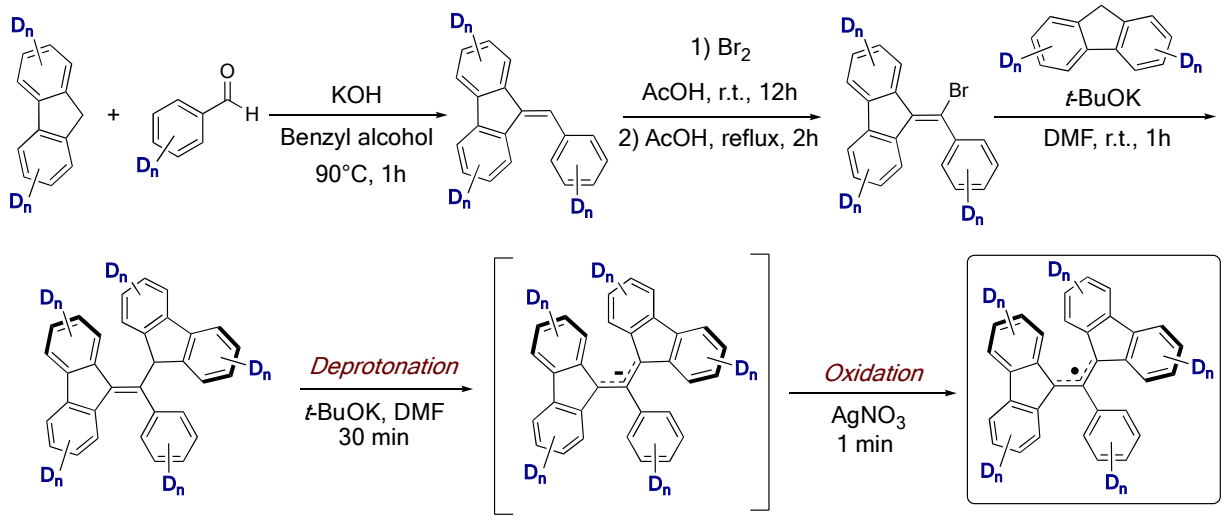
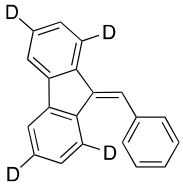


Figure S3. General synthetic route to **1,3-[α,γ-d_8]-BDPA** and **1,3-[β,δ-d_8]-BDPA**

Synthesis of 1,3-[α,γ-d_8]-BDPA radical

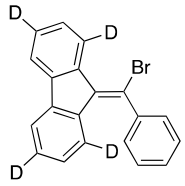


9-benzylidene-9*H*-fluorene-1,3,6,8-d_4 (11)

Synthesized following a modified literature procedure[3]. Benzaldehyde (60 µL, 0.587 mmol) was added dropwise to a solution of 9*H*-fluorene-1,3,6,8-d_4 (100 mg, 0.587 mmol) and $CsOH{\cdot}H_2O$ (40 mg, 0.235 mmol) in EtOH (1.2 mL) and the mixture was stirred at 25 °C for 2h. Complete consumption of the starting materials was observed by TLC and the reaction mixture was concentrated in vacuo. The crude reaction mixture was purified by flash chromatography on silica gel (hexanes/ethyl acetate, 80:20) to yield 9-benzylidene-9*H*-fluorene-1,3,6,8-d_4 as a pale-yellow solid (131 mg, 86%).

DP:
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.77 – 7.69 (m, 3H), 7.60 (d, *J* = 7.2 Hz, 2H), 7.47 (t, *J* = 7.6 Hz, 2H), 7.40 (t, *J* = 7.6 Hz, 1H), 7.34 (s, 1H), 7.06 (s, 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 146.88, 141.39, 139.56, 139.33, 137.04, 136.60, 129.40, 128.67, 128.17, 127.42, 126.91, 126.58, 124.99 – 124.07 (m), 120.39 – 119.79 (m), 119.74, 119.61.
HRMS (DART/AccuTOF) m/z: $[M]^+$ Calcd for $C_{20}H_{10}D_4$ 259.1180; Found 259.1158.

NDI:
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.80 (d, *J* = 7.6 Hz, 0.16H), 7.56 (d, *J* = 7.8 Hz, 0.16H), 7.31 (d, *J* = 7.4 Hz, 0.23H), 7.25 – 7.21 (m, 0.48H).

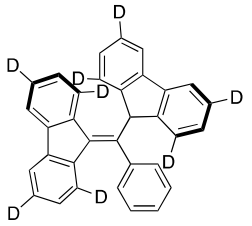


9-(bromo(phenyl)methylene)-9*H*-fluorene-1,3,6,8-d_4 (12)

Synthesized following a modified literature procedure[3]. To a flask covered with aluminum foil was suspended 9-benzylidene-9*H*-fluorene-1,3,6,8-d_4 (70 mg, 0.27 mmol) in acetic acid (1 mL). The reaction was cooled to 0°C, then Br_2 (13 µL, 0.26 mmol) was added dropwise while stirring heavily. The reaction mixture was warmed at room temperature and stir overnight. Then, 50 mL of water was added to precipitate the product. The mixture was extracted with dichloromethane (3 x 5 mL) and the organic phases were washed with saturated aqueous sodium bicarbonate solution (3 x 20 mL) and water (3 x 20 mL). The organic phase was concentrated in vacuo and the intermediate product 9-bromo-9-[bromo(phenyl)methyl]-9*H*-fluorene was dried for 2h before being suspended in acetic acid (2 ml) and heated at reflux for 2 h. The reaction mixture was left to cool at room temperature overnight and the product was collected by filtration and dried under vacuum to give the crude desired product with a 90% purity. The crude mixture was further purified by flash chromatography on silica gel (hexanes/ethyl acetate, 85:15) to yield 9-(bromo(phenyl)methylene)-9*H*-fluorene-1,3,6,8-d_4 as a yellow solid (54 mg, 59%).

DP:
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.74 (s, 1H), 7.65 (s, 1H), 7.56 – 7.44 (m, 5H), 7.40 (s, 1H), 6.85 (s, 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 142.96, 141.35, 139.95, 138.24, 137.99, 136.17, 129.40, 129.38, 129.30 – 128.73 (m), 128.69, 128.55 – 127.76 (m), 127.01, 126.75, 126.20 – 125.67 (m), 124.8 – 124.35 (m), 121.04 – 120.38 (m), 119.56, 119.28.
HRMS (DART/AccuTOF) m/z: $[M]^+$ Calcd for $C_{20}H_9D_4Br$ 337.0412; Found 337.0382.

NDI:
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.88 (d, *J* = 8.0 Hz, 0.07H), 7.70 (s, 0.08H), 7.62 – 7.58 (m, 0.17H), 7.34 (s, 0.07H), 7.23 (t, *J* = 7.5 Hz, 0.08H), 7.06 (s, 0.06H), 6.21 (d, *J* = 8.0 Hz, 0.07H).

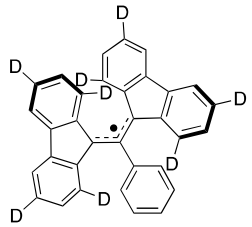


9-((9*H*-fluoren-9-yl-1,3,6,8-d_4)(phenyl)methylene)-9*H*-fluorene-1,3,6,8-d_4 (13)

Synthesized following a modified literature procedure[3]. ^{t}BuOK (27 mg, 0.24 mmol) was added in portions to a stirred solution of 9-(bromo(phenyl)methylene)-9*H*-fluorene-1,3,6,8-d_4 (20 mg, 0.059 mmol) and 9*H*-fluorene-1,3,6,8-d_4 (11 mg, 0.065 mmol) in DMF (0.5 mL) under N_2. The reaction mixture was stirred 3h at room temperature, then hydrochloric acid (1 M, 1 mL was added and the reaction mixture followed by water (2 ml) to give an orange precipitate. The crude mixture was collected by filtration, washed with water (10 ml) and methanol (10 ml) and dried overnight. Purification by flash chromatography on silica gel (hexanes/ethyl acetate, 80:20) gave the 9-((9*H*-fluoren-9-yl-1,3,6,8-d_4)(phenyl)methylene)-9*H*-fluorene-1,3,6,8-d_4 as an orange solid (20 mg, 78%).

DP:
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.89 (s, 1H), 7.75 (s, 1H), 7.66 (s, 2H), 7.36 (s, 1H), 7.26 (s, 2H), 7.07 (t, *J* = 7.4 Hz, 1H), 6.99 (t, *J* = 7.6 Hz, 2H), 6.81 (s, 1H), 6.67 (d, *J* = 7.1 Hz, 2H), 6.48 (s, 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 145.27, 144.22, 142.11, 141.44, 139.89, 139.00, 138.78, 138.71, 136.06, 128.48, 127.86, 127.36, 127.20, 126.83, 126.48, 126.09 – 125.63 (m), 125.21 – 124.69 (m), 119.99, 119.95, 119.08, 52.79.
HRMS (DART/AccuTOF) m/z: $[M]^+$ Calcd for $C_{33}H_{14}D_8$ 427.2257; Found 427.2345.

NDI:
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.44 (d, *J* = 8.0 Hz, 0.07H), 7.60 (d, *J* = 7.6 Hz, 0.14H), 7.47 (t, *J* = 7.5 Hz, 0.08H), 7.34 (t, *J* = 7.6 Hz, 0.14H), 7.22 (t, *J* = 7.3 Hz, 0.15H), 6.49 (s, 0.08H), 5.90 (d, *J* = 8.0 Hz, 0.07H).

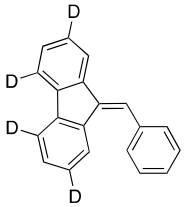


1,3-[α,γ-d_8]-BDPA radical (14)

Synthesized following a modified literature procedure[4]. ^{t}BuOK (24 mg, 0.21 mmol) was added in portions to a stirred solution of compound 9-((9*H*-fluoren-9-yl-1,3,6,8-d_4)(phenyl)methylene)-9*H*-fluorene-1,3,6,8-d_4 (15 mg, 0.035 mmol) in DMF (0.3 ml) under N_2 at room temperature which resulted in a colour change from orange to dark blue. After 30 min, a solution of $AgNO_3$ (35 mg, 0.21 mmol) in DMF (0.1 mL) was added. The solution immediately became red-brown and was

diluted after 30 min of stirring with 2 mL of 0.01 M HCl and extracted with diethyl ether (3x2 mL). The combined organic layers were washed with water (3x10 mL) and dried under vacuum. Purification by flash chromatography on silica gel (hexanes/ethyl acetate, 80:20) gave the 1,3-[α,γ-d_8]-BDPA radical as a deep red glassy solid (10 mg, 66%).

Synthesis of 1,3-[β,δ-d_8]-BDPA radical

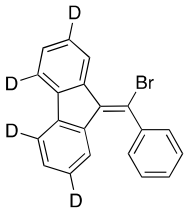


9-benzylidene-9*H*-fluorene-2,4,5,7-d_4 (15)

Prepared according to the general procedure used for compound **11**. Benzaldehyde (60 µL, 0.587 mmol), 9*H*-fluorene-2,4,5,7-d_4 (100 mg, 0.587 mmol) and $CsOH{\cdot}H_2O$ (40 mg, 0.235 mmol), and EtOH (1.2 mL) was used. Yielding 9-benzylidene-9*H*-fluorene-2,4,5,7-d_4 as a pale-yellow solid (136 mg, 90%).

DP:
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.80 (s, 1H), 7.71 (s, 1H), 7.59 (d, J = 8.1 Hz, 2H), 7.56 (s, 1H), 7.47 (t, J = 7.5 Hz, 2H), 7.43 – 7.36 (m, 2H), 7.31 (s, 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 141.29, 139.61, 139.24, 137.04, 136.67, 136.62, 129.40, 128.67, 128.46, 128.16, 128.13, 127.40, 127.14 – 126.30 (m), 124.44, 120.26, 119.90 – 119.25 (m).
HRMS (DART/AccuTOF) m/z: $[M]^+$ Calcd for $C_{20}H_{10}D_4$ 259.1180; Found 259.1168.

NDI:
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.75 – 7.71 (m, 0.06H), 7.35 – 7.32 (m, 0.06H), 7.06 (td, J = 7.6, 1.3 Hz, 0.06H).



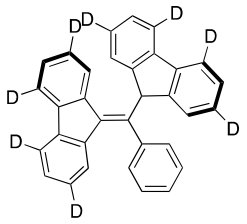
9-(bromo(phenyl)methylene)-9*H*-fluorene-2,4,5,7-d_4 (16)

Prepared according to the general procedure used for compound **12**. 9-Benzylidene-9*H*-fluorene-2,4,5,7-d_4 (70 mg, 0.27 mmol), Br_2 (13 µL, 0.26 mmol) and acetic acid (1 +2 mL) was used. Yielding 9-(bromo(phenyl)methylene)-9*H*-fluorene-2,4,5,7-$\boldsymbol{d_4}$ as a pale-yellow solid (49 mg, 54%).

DP:

^{1}H NMR (600 MHz, $CDCl_3$) δ 8.87 (s, 1H), 7.60 – 7.38 (m, 6H), 7.23 (s, 1H), 6.21 (s, 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 142.98, 141.27, 139.87, 138.32, 138.07, 136.20, 129.40, 129.39, 129.06, 128.70, 128.05, 127.25 –126.46 (m), 126.08, 124.76, 124.65, 124.44, 120.26, 119.71 – 118.91 (m).
HRMS (DART/AccuTOF) m/z: $[M]^+$ Calcd for $C_{20}H_9D_4Br$ 337.0412; Found 337.0399.

NDI:
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.80 (s, 0.12H), 7.74 (d, *J* = 7.5 Hz, 0.06H), 7.71 (s, 0.11H), 7.65 (d, *J* = 7.5 Hz, 0.06H), 7.56 (s, 0.07H), 7.41 (s, 0.06H). 7.31 (s, 0.06H), 6.85 (t, *J* = 7.7 Hz, 0.07H).

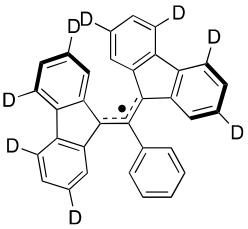


9-((9*H*-fluoren-9-yl-2,4,5,7-d_4)(phenyl)methylene)-9*H*-fluorene-2,4,5,7-d_4 (17)

Prepared according to the general procedure used for compound **13**. ^{t}BuOK (27 mg, 0.24 mmol), 9-(bromo(phenyl)methylene)-9*H*-fluorene-2,4,5,7-d_4 (20 mg, 0.059 mmol), 9*H*-fluorene-1,3,6,8-d_4 (11 mg, 0.065 mmol), and DMF (0.5 ml) was used. Yielding 9-((9*H*-fluoren-9-yl-2,4,5,7-d_4)(phenyl)methylene)-9*H*-fluorene-2,4,5,7- d_4 as an orange solid (17 mg, 68%).

DP:
^{1}H NMR (600 MHz, $CDCl_3$) δ 8.45 (s, 1H), 7.60 (s, 2H), 7.47 (s, 1H), 7.34 (s, 2H), 7.23 (s, 1H), 7.08 (t, *J* = 7.5 Hz, 1H), 7.00 (t, *J* = 7.7 Hz, 2H), 6.67 (d, *J* = 7.5 Hz, 2H), 6.50 (s, 1H), 5.90 (s, 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 145.27, 144.30, 142.03, 141.36, 139.81, 139.07, 138.86, 138.71, 136.11, 128.50, 127.85, 127.37, 127.36, 127.32, 126.20 – 126.92 (m), 126.04, 125.51, 125.14, 119.55 – 120.09 (m), 118.66 – 119.15 (m), 52.84.
HRMS (DART/AccuTOF) m/z: $[M]^+$ Calcd for $C_{33}H_{14}D_8$ 427.2257; Found 427.2242.

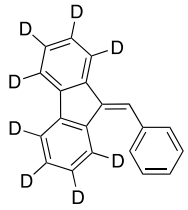
NDI:
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.89 (d, *J* = 7.6 Hz, 0.06H), 7.75 (d, *J* = 7.6 Hz, 0.06H), 7.66 (d, *J* = 7.6 Hz, 0.12H), 7.37 (d, *J* = 7.7 Hz, 0.06H), 6.82 (t, *J* = 7.7 Hz, 0.10H).



1,3-[β,δ-d_8]-BDPA radical (18)

Prepared according to the general procedure used for compound **14**. ^{t}BuOK (24 mg, 0.21 mmol) was added in portions to a stirred solution of compound 9-((9*H*-fluoren-9-yl-2,4,5,7-d_4)(phenyl)methylene)-9*H*-fluorene-2,4,5,7-d_4 (15 mg, 0.035 mmol), $AgNO_3$ (35 mg, 0.21 mmol), DMF (0.3 mL) was used. Yielding 1,3-[β,δ-d_8]-BDPA radical as a deep red glassy solid (11 mg, 73%).

Synthesis of 1,3-[α,β,γ,δ-d_{16}]-BDPA radical

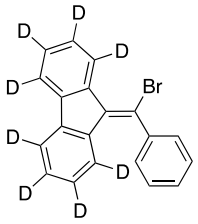


9-benzylidene-9*H*-fluorene-1,2,3,4,5,6,7,8-d_8 (19)

Prepared according to the general procedure used for compound **11**. Benzaldehyde (461 µL, 4.54 mmol), 9*H*-fluorene-d_{10} (800 mg, 4.54 mmol) and $CsOH \cdot H_2O$ (305 mg, 1.81 mmol), and EtOH (9.0 mL) was used. Yielding 9-benzylidene-9*H*-fluorene-1,2,3,4,5,6,7,8-d_8 as a pale-yellow solid (1.05 g, 88%).

DP:
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.73 (s, 1H), 7.63 (d, J = 7.0 Hz, 2H), 7.50 (t, J = 7.4 Hz, 2H), 7.43 (t, J = 7.2 Hz, 1H).
^{13}C NMR (151 MHz, $CDCl_3$) δ 141.31, 139.55, 139.24, 137.04, 136.60, 129.39, 128.66, 128.14, 127.38, 126.89 – 125.98 (m), 124.45 – 123.80 (m), 120.30 – 119.02 (m).

HRMS (DART/AccuTOF) m/z: $[M]^+$ Calcd for $C_{20}H_6D_8$ 262.1598; Found 262.1638.

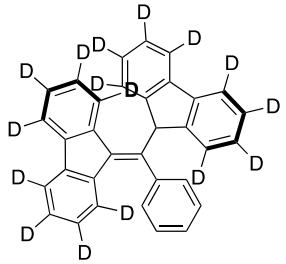


9-(bromo(phenyl)methylene)-9*H*-fluorene-1,2,3,4,5,6,7,8-d_8 (20)

Prepared according to the general procedure used for compound **12**. 9-benzylidene-9*H*-fluorene-1,2,3,4,5,6,7,8-d_8 (1.0 g, 3.73 mmol), Br_2 (187 µL, 3.65 mmol) and acetic acid (15 +15 mL) was used. Yielding 9-(bromo(phenyl)methylene)-9*H*-fluorene-1,2,3,4,5,6,7,8-$d_8$as a pale-yellow solid (690 mg, 51%).
^{1}H NMR (600 MHz, $CDCl_3$) δ 7.56 – 7.44 (m, 5H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 142.97, 141.27, 139.88, 138.24, 137.99, 136.19, 129.39, 129.37, 128.69, 127.03 – 125.24 (m), 124.62, 124.56 – 124.03 (m), 119.60 – 118.70 (m).

DP:
HRMS (DART/AccuTOF) m/z: $[M]^+$ Calcd for $C_{20}H_9D_4Br$ 340.0703; Found 340.0722.



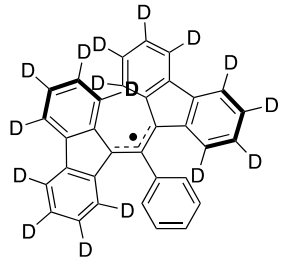
9-((9*H*-fluoren-9-yl-1,2,3,4,5,6,7,8-d_8)(phenyl)methylene)-9*H*-fluorene-1,2,3,4,5,6,7,8-d_8 (21)

Prepared according to the general procedure used for compound **13**. ^{t}BuOK (525 mg, 04.68 mmol), 9-(bromo(phenyl)methylene)-9*H*-fluorene-1,2,3,4,5,6,7,8-d_8 **(**400 mg, 1.17 mmol), 9*H*-fluorene-d_{10} (227 mg, 1.29 mmol), and DMF (7.3 ml) was used. Yielding 9-((9*H*-fluoren-9-yl-1,2,3,4,5,6,7,8-d_8)(phenyl)methylene)-9*H*-fluorene-1,2,3,4,5,6,7,8-d_8 as an orange solid (330 mg, 65%).

^{1}H NMR (600 MHz, $CDCl_3$) δ 7.08 (t, J = 7.4 Hz, 1H), 7.00 (t, J = 7.5 Hz, 2H), 6.68 (d, J = 6.8 Hz, 2H), 6.49 (s, 1H).

^{13}C NMR (151 MHz, $CDCl_3$) δ 145.26, 144.22, 142.02, 141.36, 139.81, 138.99, 138.77, 138.72, 136.07, 128.48, 127.85, 127.35, 127.27 – 124.42 (m), 120.09 – 119.37 (m), 52.79.

DP:
HRMS (DART/AccuTOF) m/z: $[M]^+$ Calcd for $C_{33}H_{14}D_8$ 437.2726; Found 437.2755.



1,3-[α,β,γ,δ-d_{16}]-BDPA radical (22)

Prepared according to the general procedure used for compound **14**. ^{t}BuOK (47 mg, 0.42 mmol) was added in portions to a stirred solution of compound 9-((9*H*-fluoren-9-yl-1,2,3,4,5,6,7,8-d_8)(phenyl)methylene)-9*H*-fluorene-1,2,3,4,5,6,7,8-d_8 (30 mg, 0.07 mmol), $AgNO_3$ (71 mg, 0.42 mmol), DMF (0.35 mL) was used. Yielding 1,3-[d_{16}]-BDPA radical as a deep red glassy solid (22 mg, 72%).

4. NMR Spectra

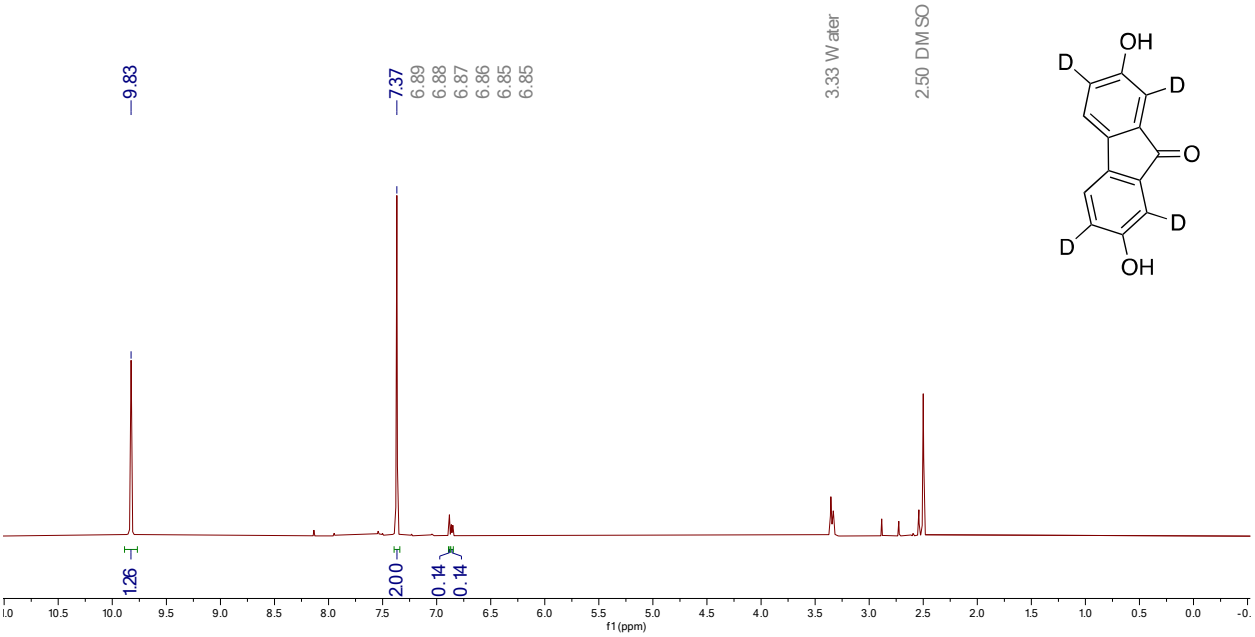


Figure S4 ^{1}H NMR spectrum (600 MHz, DMSO-d_6) of **2,7-dihydroxy-9*H*-fluoren-9-one-1,3,6,8-d_4 (2)**

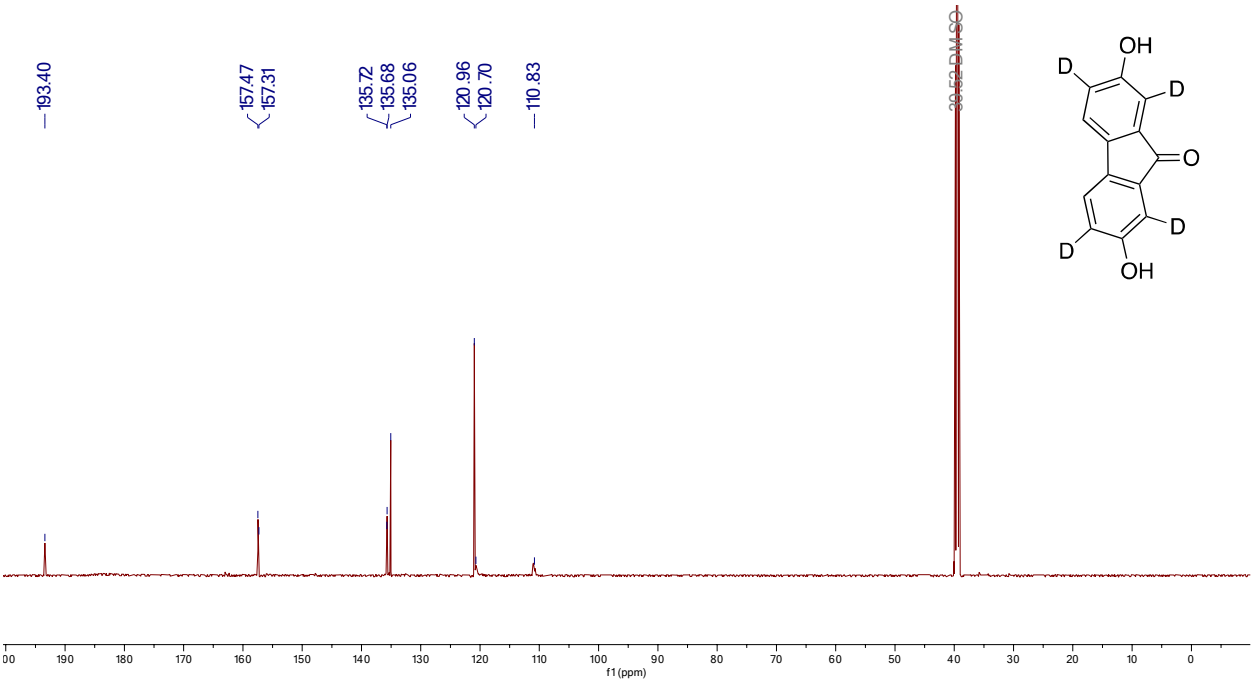


Figure S5 ^{13}C NMR spectrum (151 MHz, DMSO-d$_6$) of **2,7-dihydroxy-9H-fluoren-9-one-1,3,6,8-d$_4$ (2)**

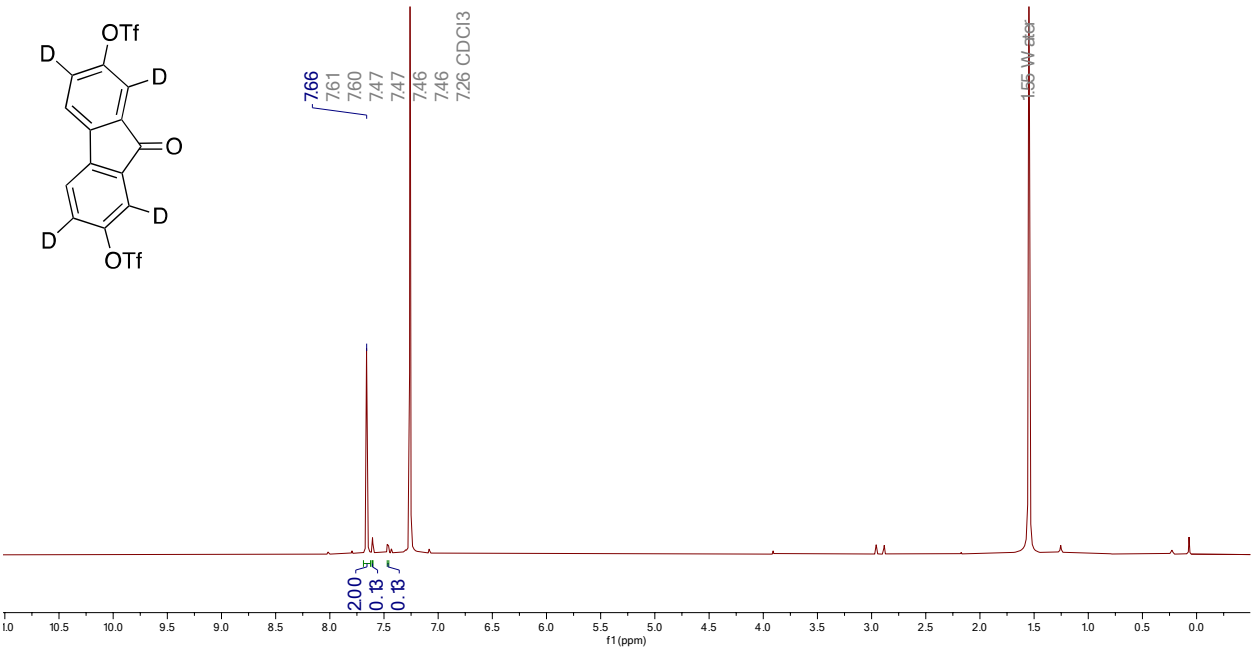


Figure S6 ^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **9-oxo-9*H*-fluorene-2,7-diyl-1,3,6,8-*d*$_4$ bis(trifluoromethanesulfonate) (3)**

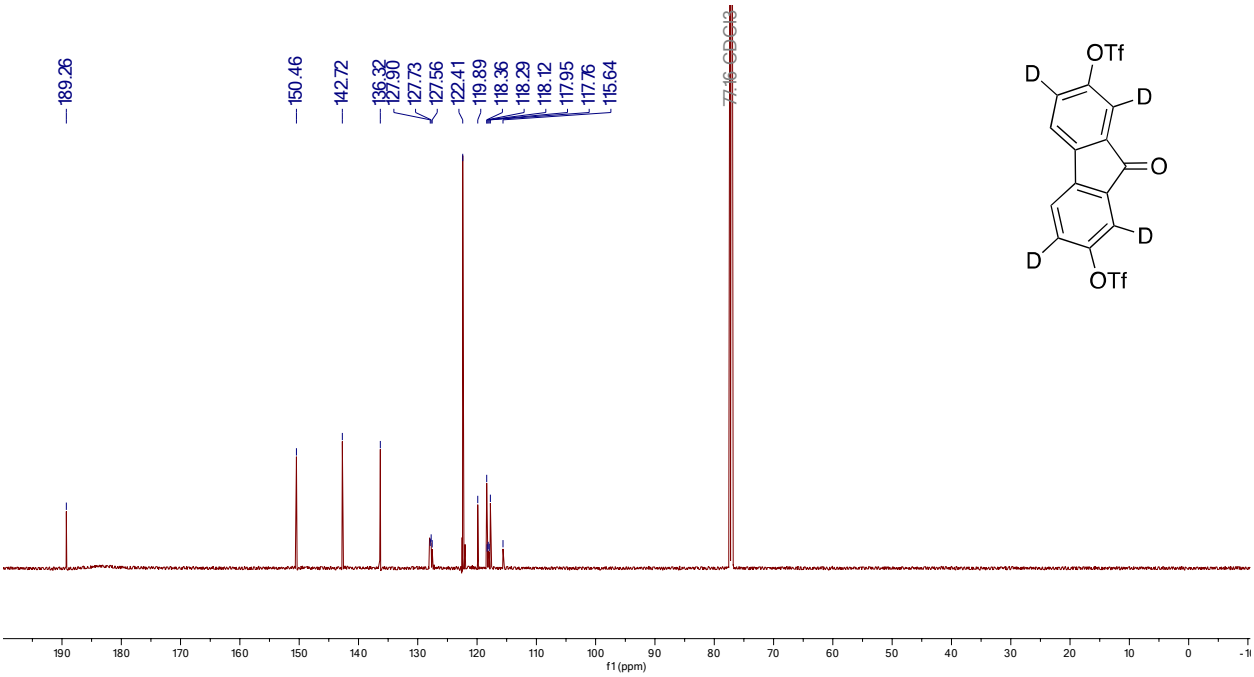


Figure S7 ^{13}C NMR spectrum (151 MHz, $CDCl_3$) of **9-oxo-9*H*-fluorene-2,7-diyl-1,3,6,8-d_4 bis(trifluoromethanesulfonate) (3)**

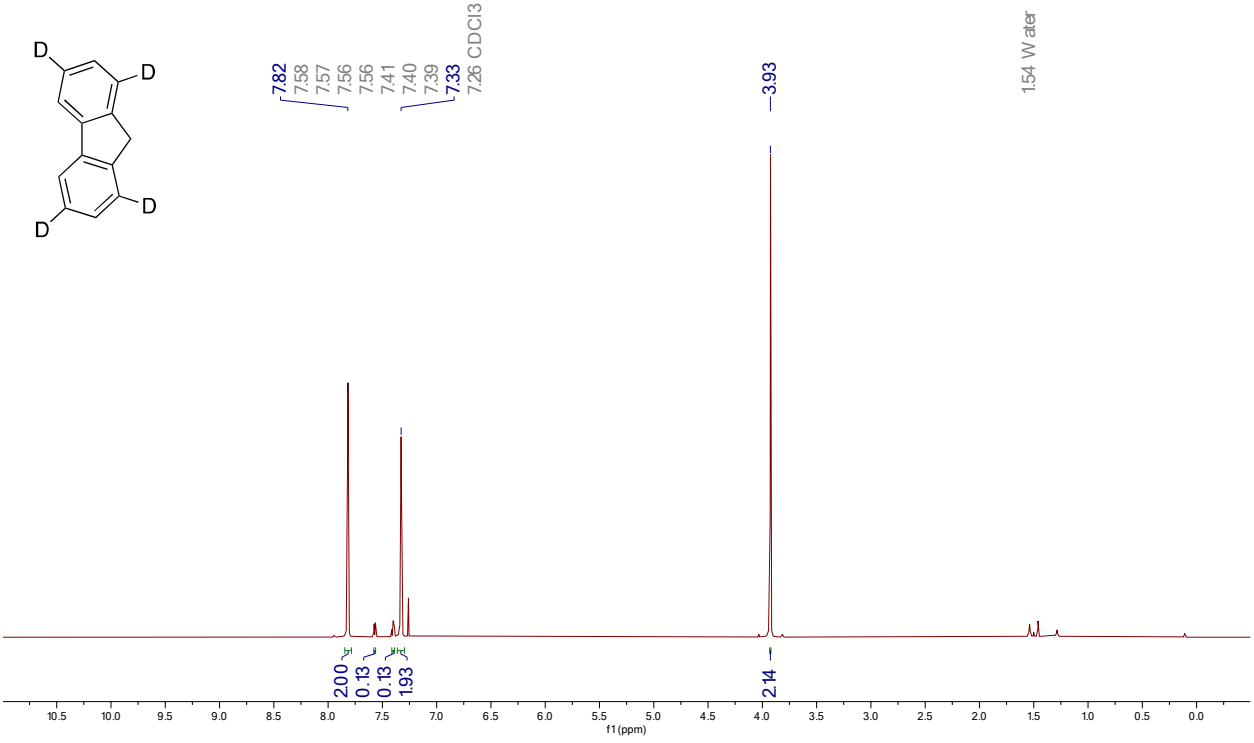


Figure S8 ^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **9*H*-fluorene-1,3,6,8-d_4 (4)**

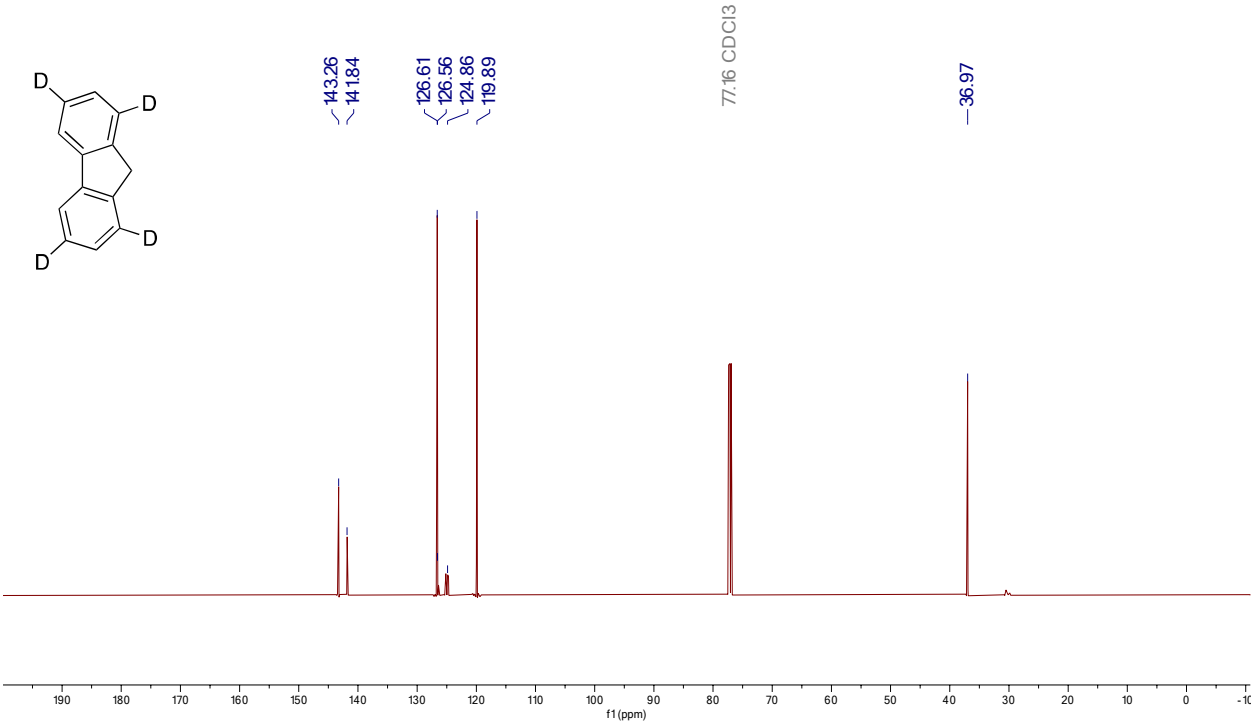


Figure S9 ^{13}C NMR spectrum (151 MHz, $CDCl_3$) of **9*H*-fluorene-1,3,6,8-d_4 (4)**

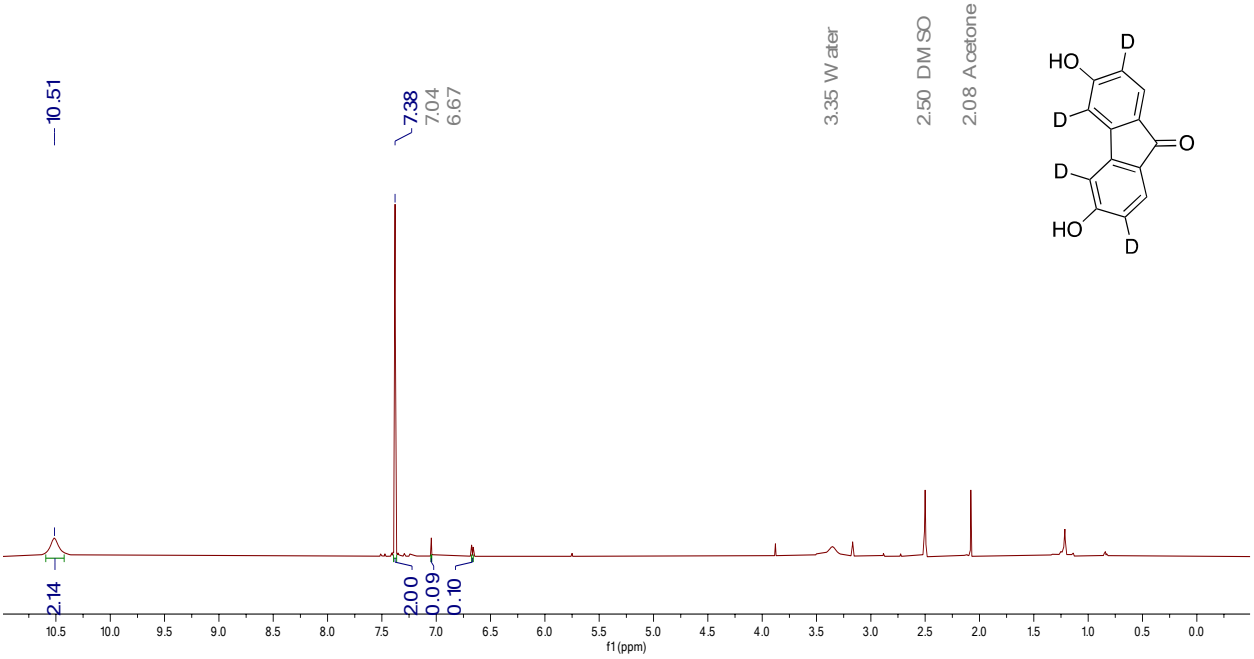


Figure S10 ^{1}H NMR spectrum (600 MHz, DMSO-d_6) of **3,6-dihydroxy-9*H*-fluoren-9-one-2,4,5,7-d_4 (8)**

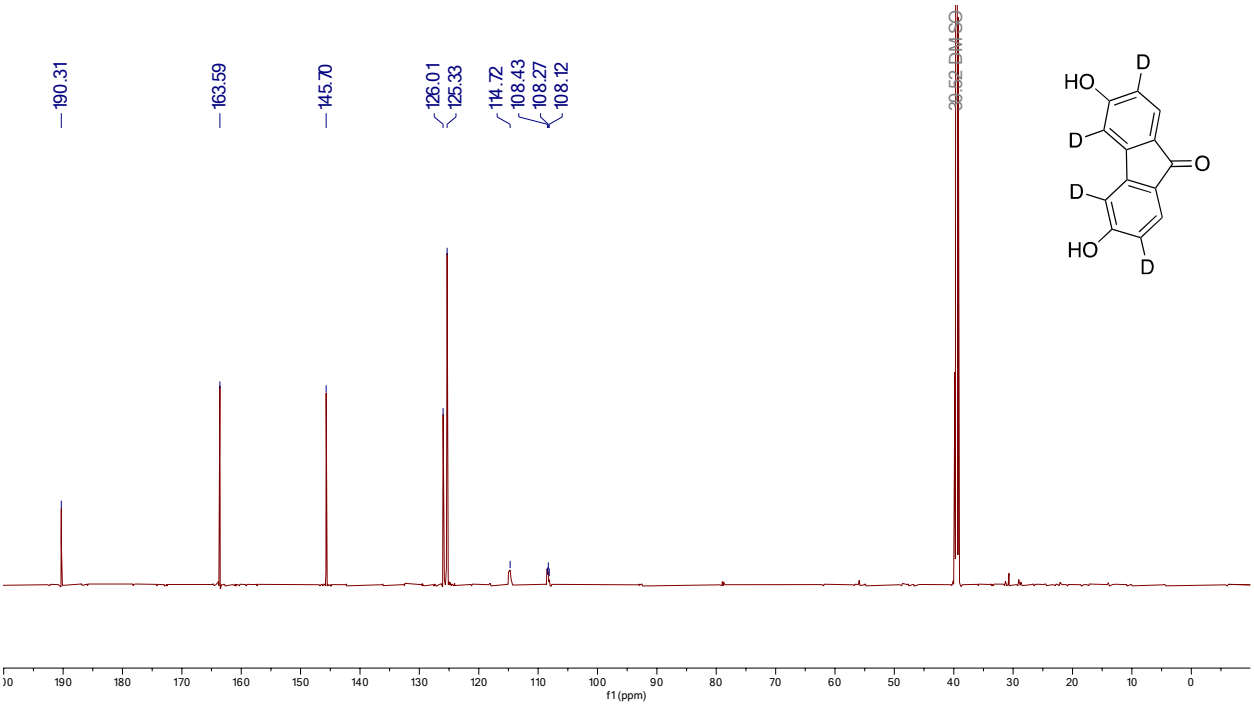


Figure S11 ^{13}C NMR spectrum (151 MHz, DMSO-d_6) of **3,6-dihydroxy-9*H*-fluoren-9-one-2,4,5,7-d_4 (8)**

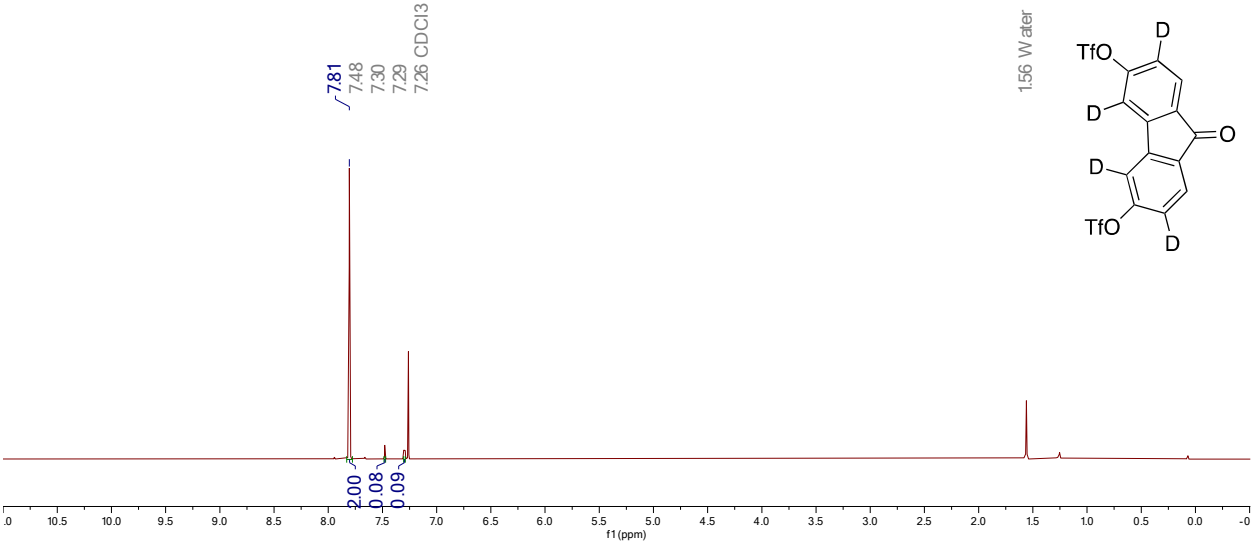


Figure S12 ^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **9-oxo-9*H*-fluorene-3,6-diyl-2,4,5,7-d_4 bis(trifluoromethanesulfonate) (9)**

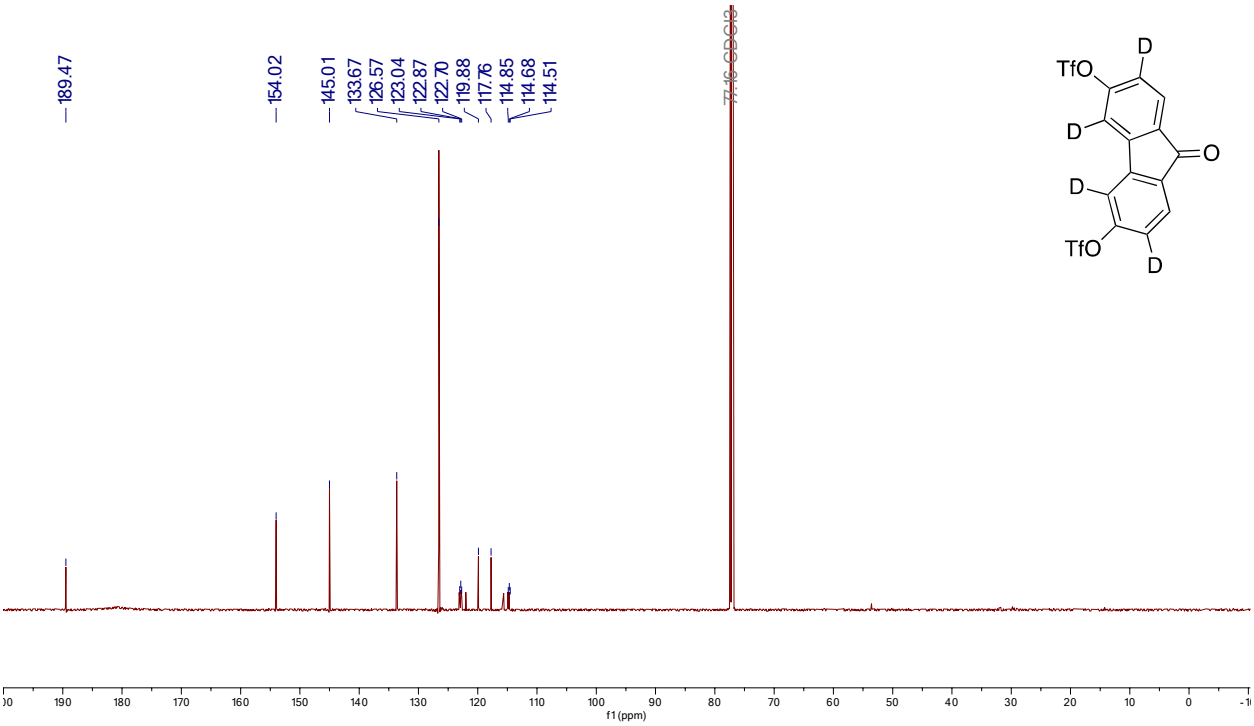


Figure S13 ^{13}C NMR spectrum (151 MHz, $CDCl_3$) of **9-oxo-9*H*-fluorene-3,6-diyl-2,4,5,7-*d*$_4$ bis(trifluoromethanesulfonate) (9)**

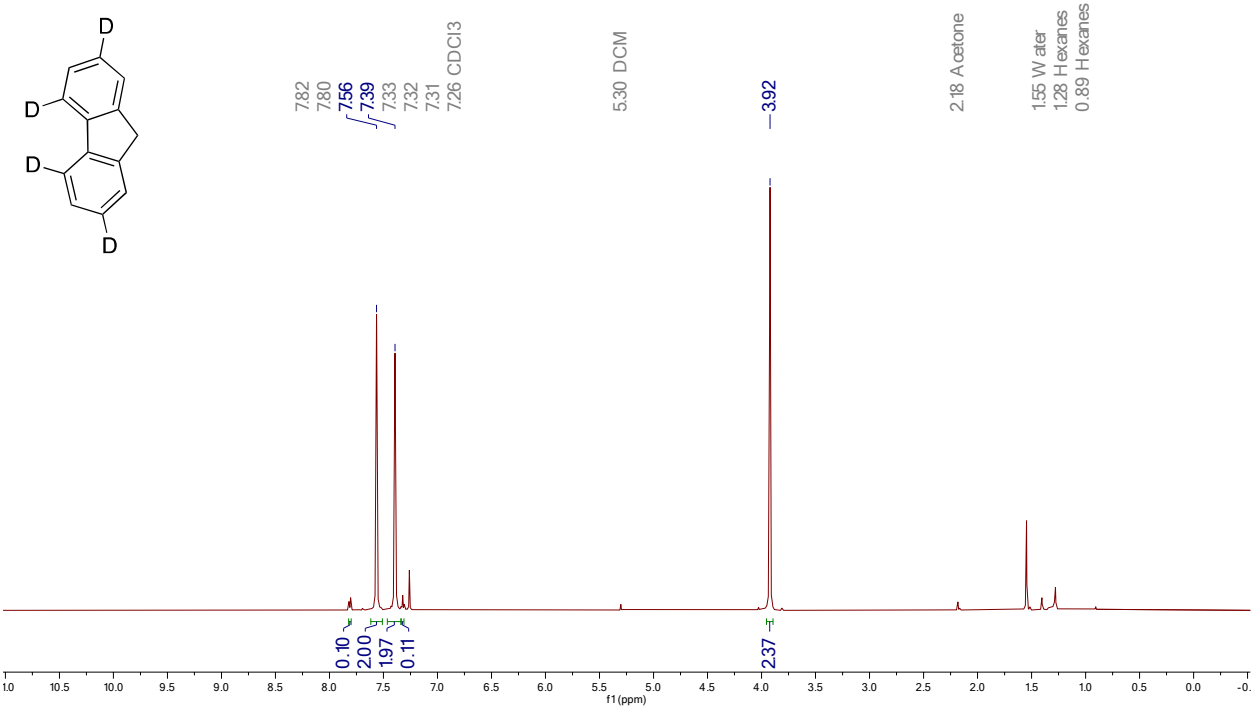


Figure S14 ^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **9H-fluorene-2,4,5,7-*d*$_4$ (10)**

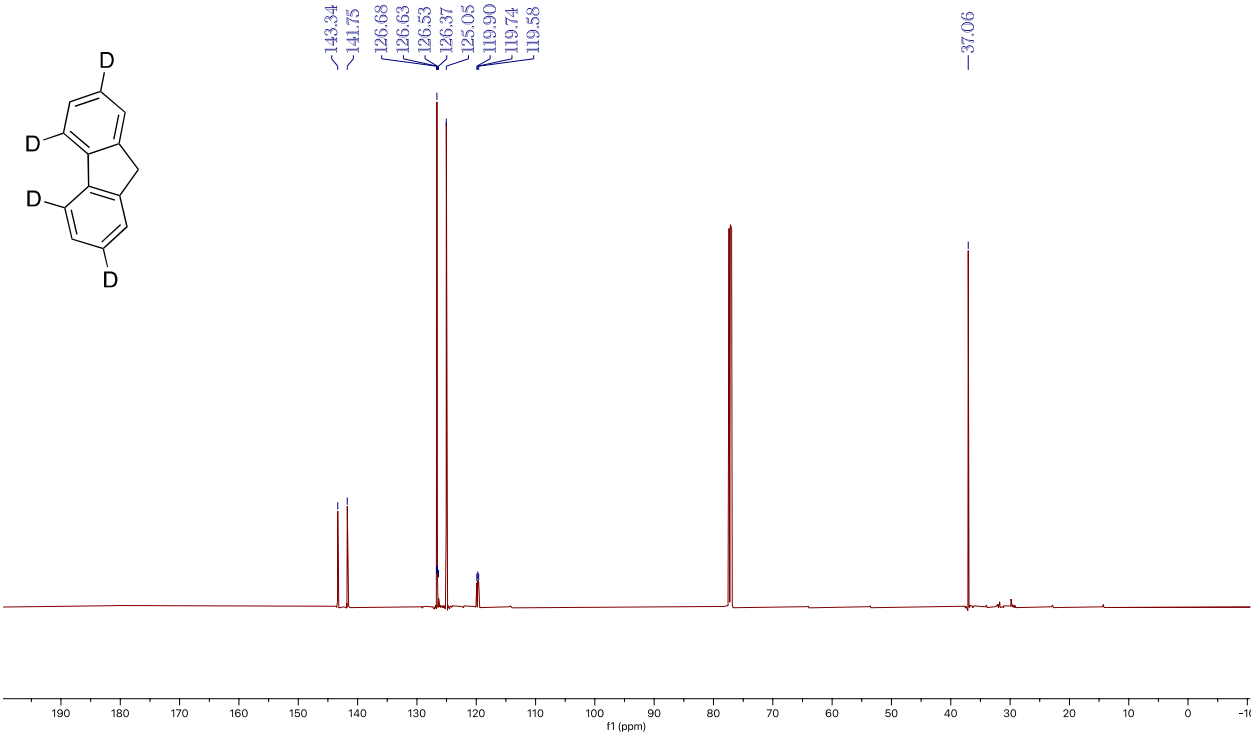


Figure S15 ^{13}C NMR spectrum (151 MHz, $CDCl_3$) of **9H-fluorene-2,4,5,7-*d*$_4$ (10)**

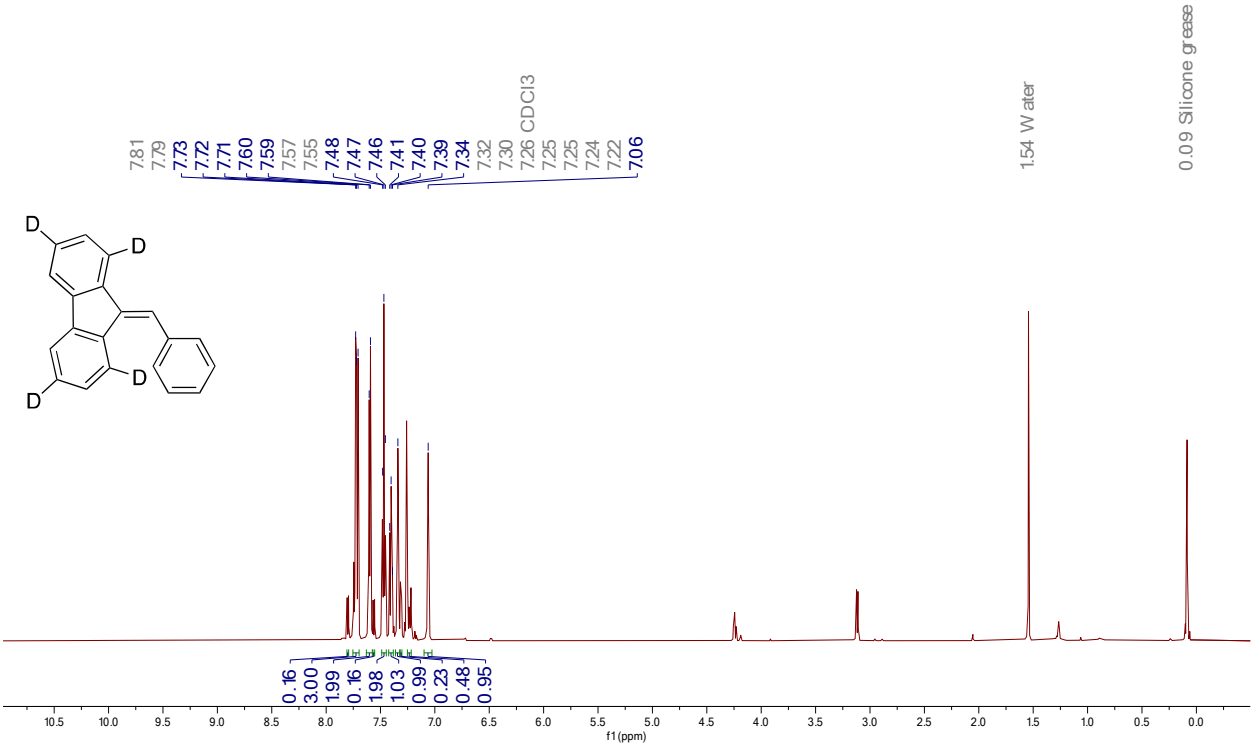


Figure S16 ^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **9-benzylidene-9*H*-fluorene-1,3,6,8-*d*$_4$ (11)**

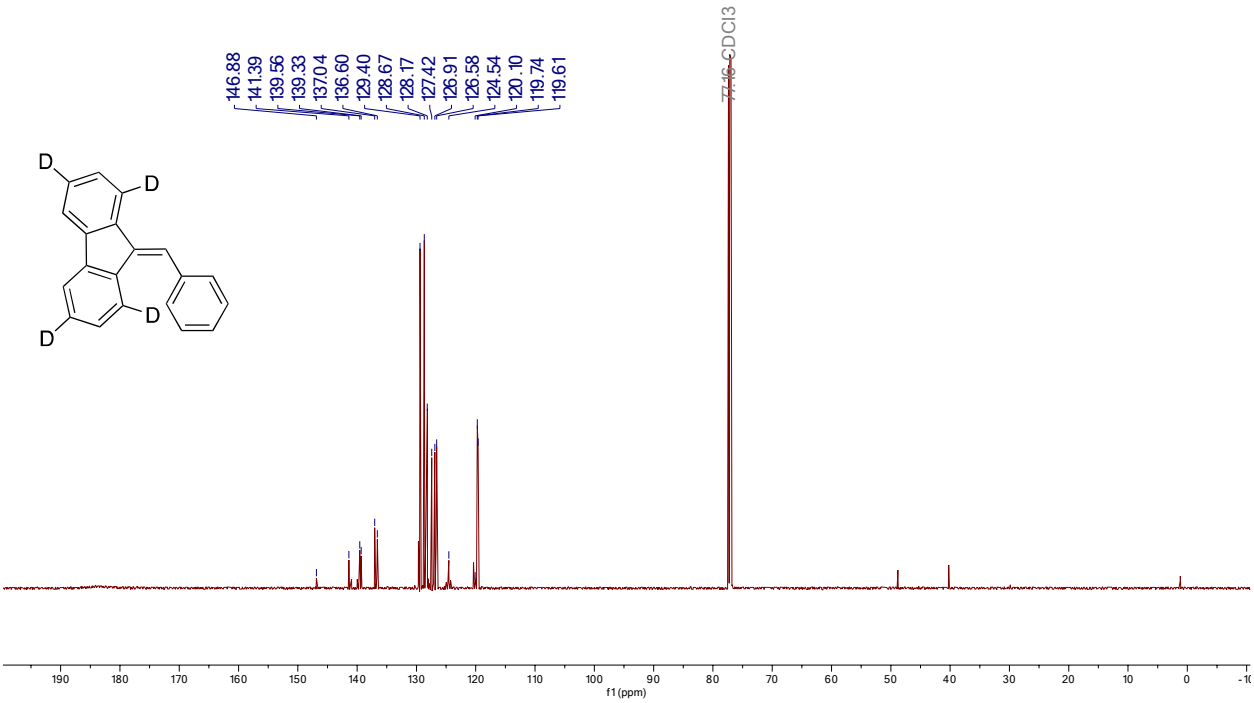


Figure S17 ^{13}C NMR spectrum (151 MHz, $CDCl_3$) of **9-benzylidene-9*H*-fluorene-1,3,6,8-d_4 (11)**

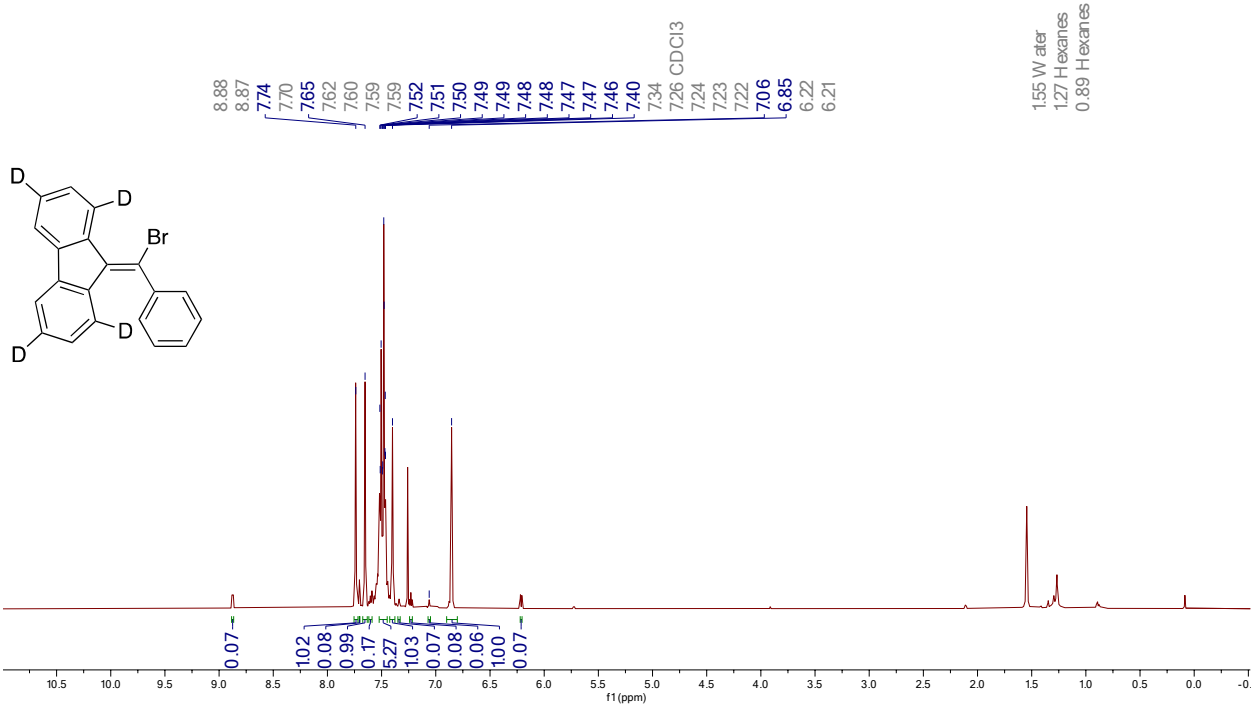


Figure S18 ^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **9-(bromo(phenyl)methylene)-9*H*-fluorene-1,3,6,8-d_4 (12)**

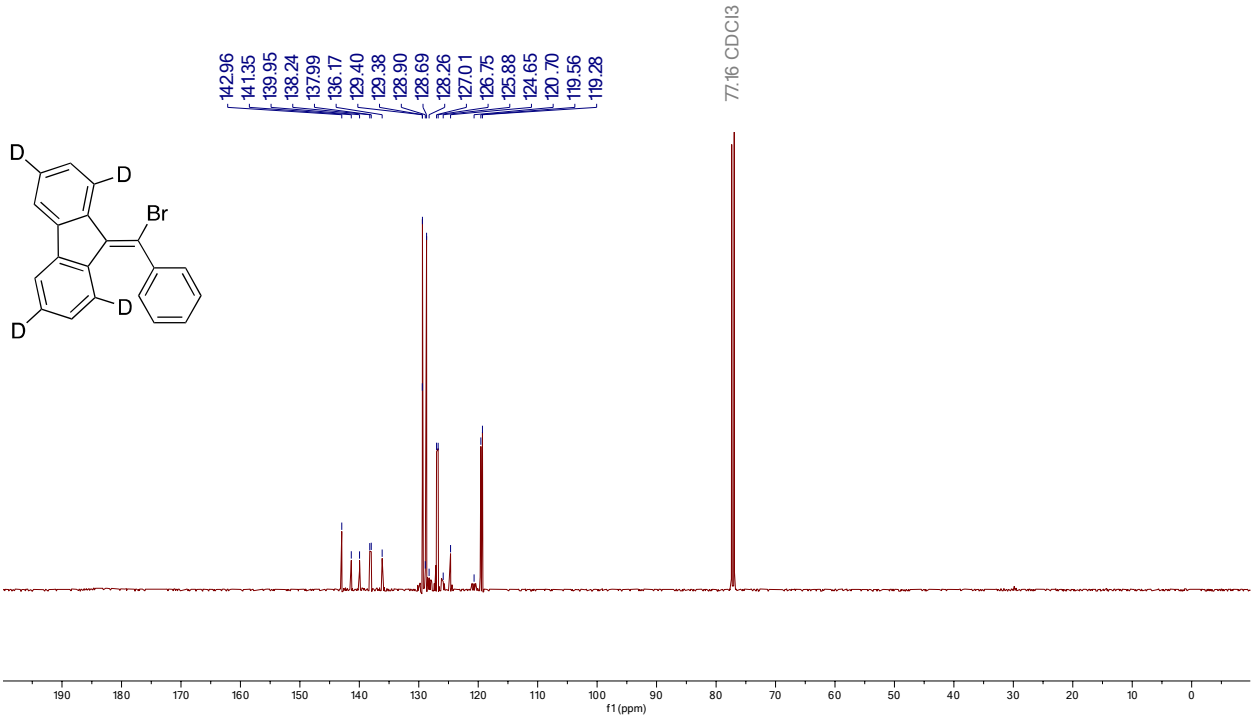


Figure S19 ^{13}C NMR spectrum (151 MHz, $CDCl_3$) of **9-(bromo(phenyl)methylene)-9*H*-fluorene-1,3,6,8-*d*$_4$ (12)**

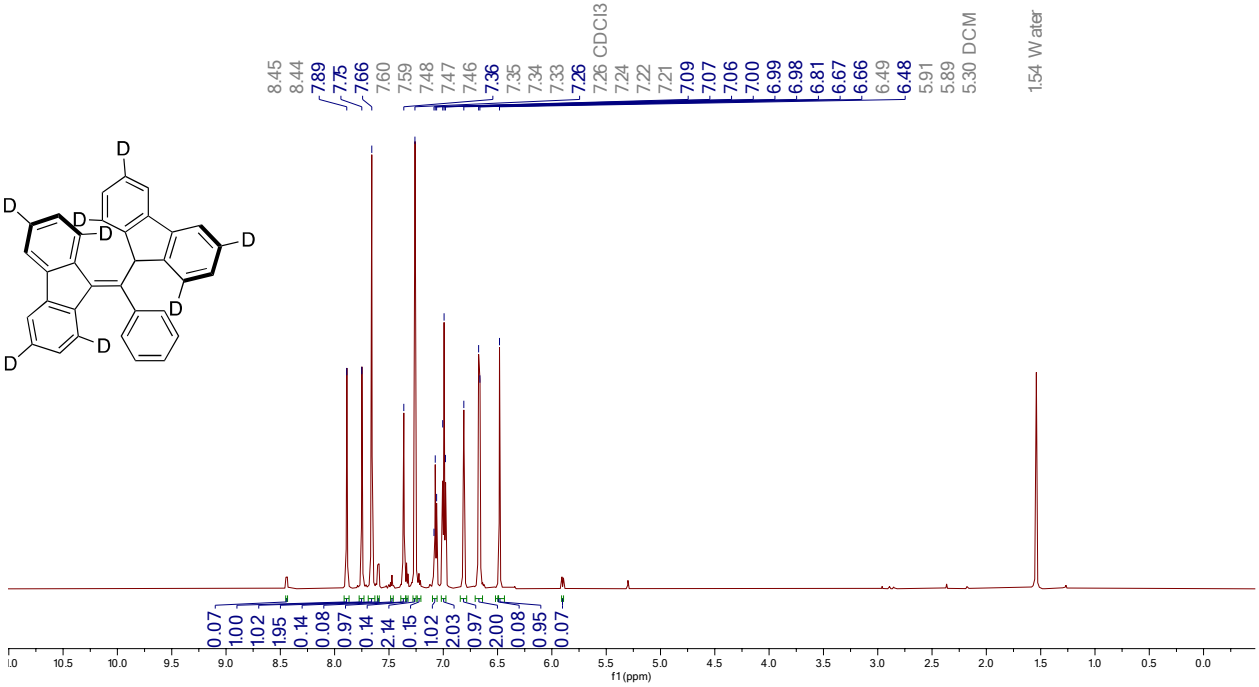


Figure S20 ^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **9-((9*H*-fluoren-9-yl-1,3,6,8-*d*$_4$)(phenyl)methylene)-9*H*-fluorene-1,3,6,8-*d*$_4$ (13)**

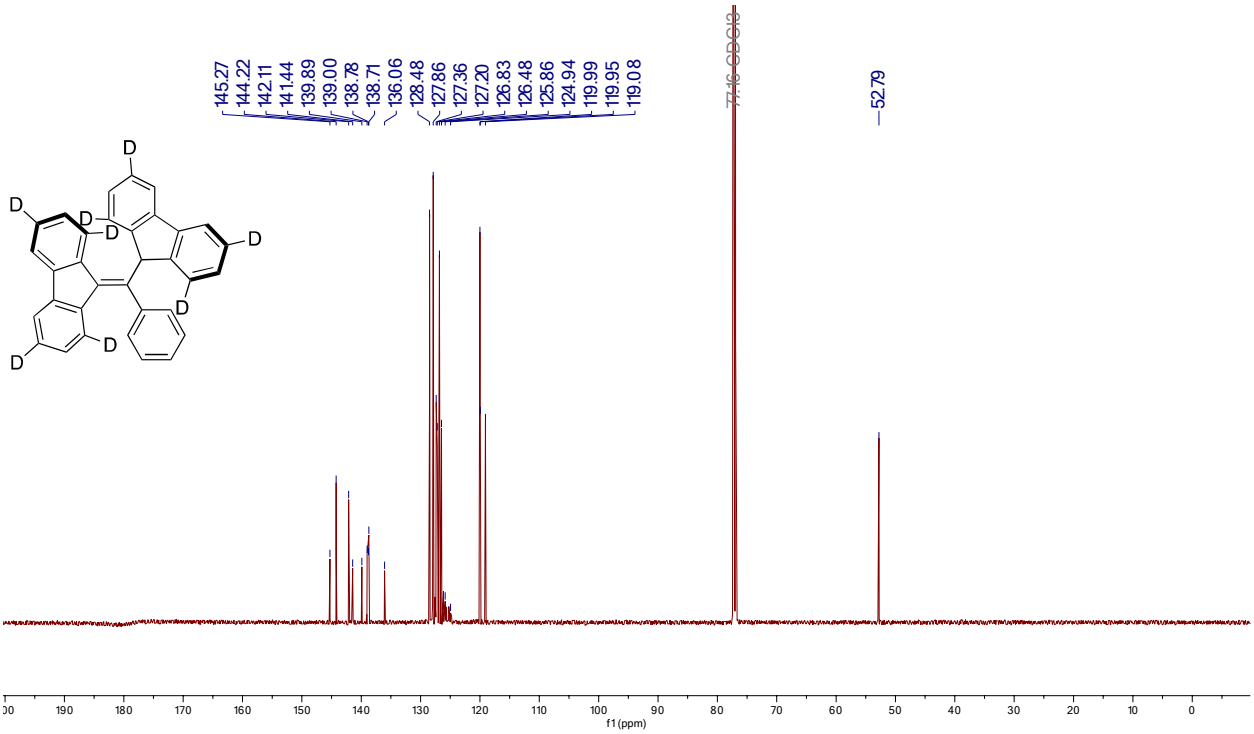


Figure S21 ^{13}C NMR spectrum (151 MHz, $CDCl_3$) of **9-((9*H*-fluoren-9-yl-1,3,6,8-d_4)(phenyl)methylene)-9*H*-fluorene-1,3,6,8-d_4 (13)**

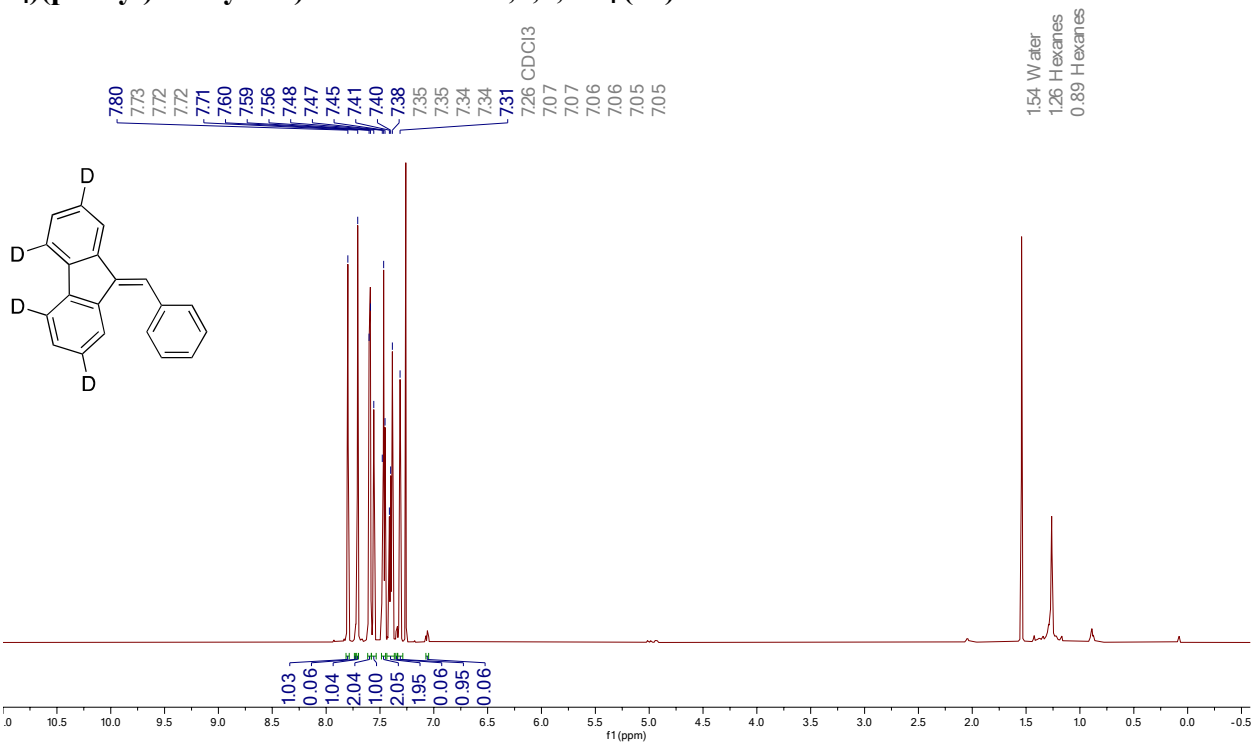


Figure S22 ^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **9-benzylidene-9*H*-fluorene-2,4,5,7-d_4 (15)**

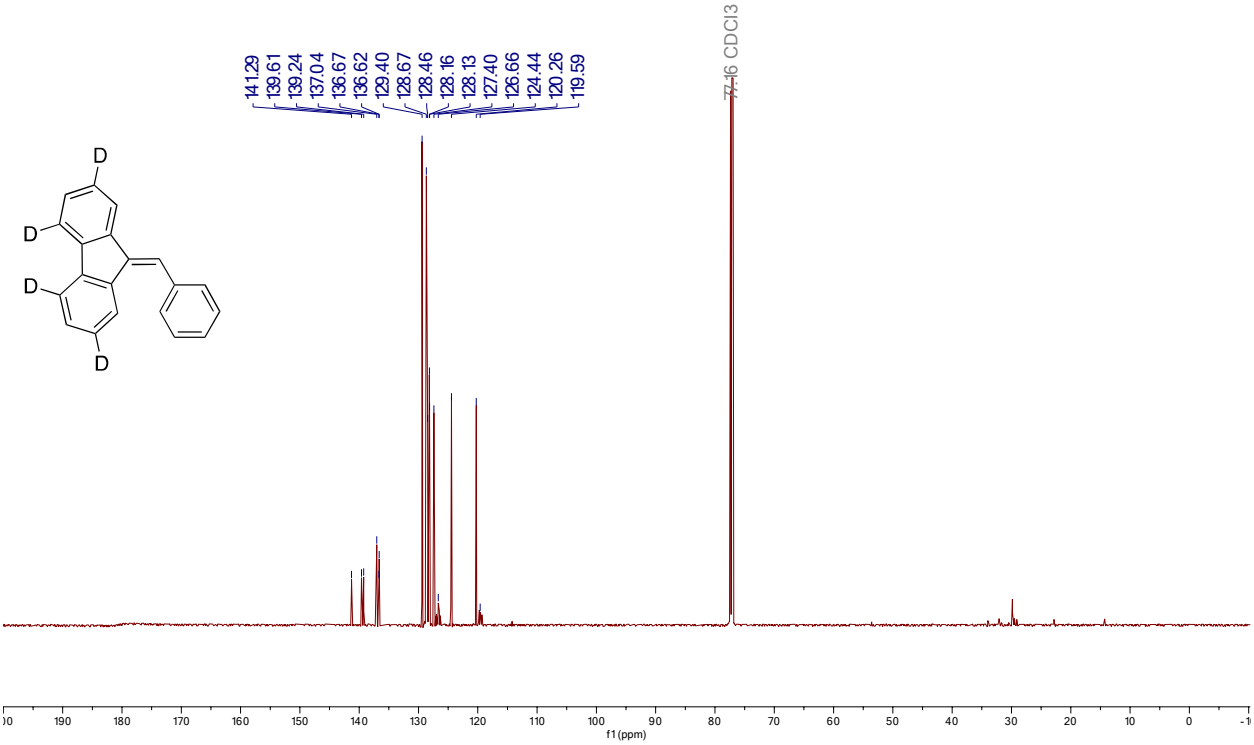


Figure S23 ^{13}C NMR spectrum (151 MHz, $CDCl_3$) of **9-benzylidene-9*H*-fluorene-2,4,5,7-*d*$_4$ (15)**

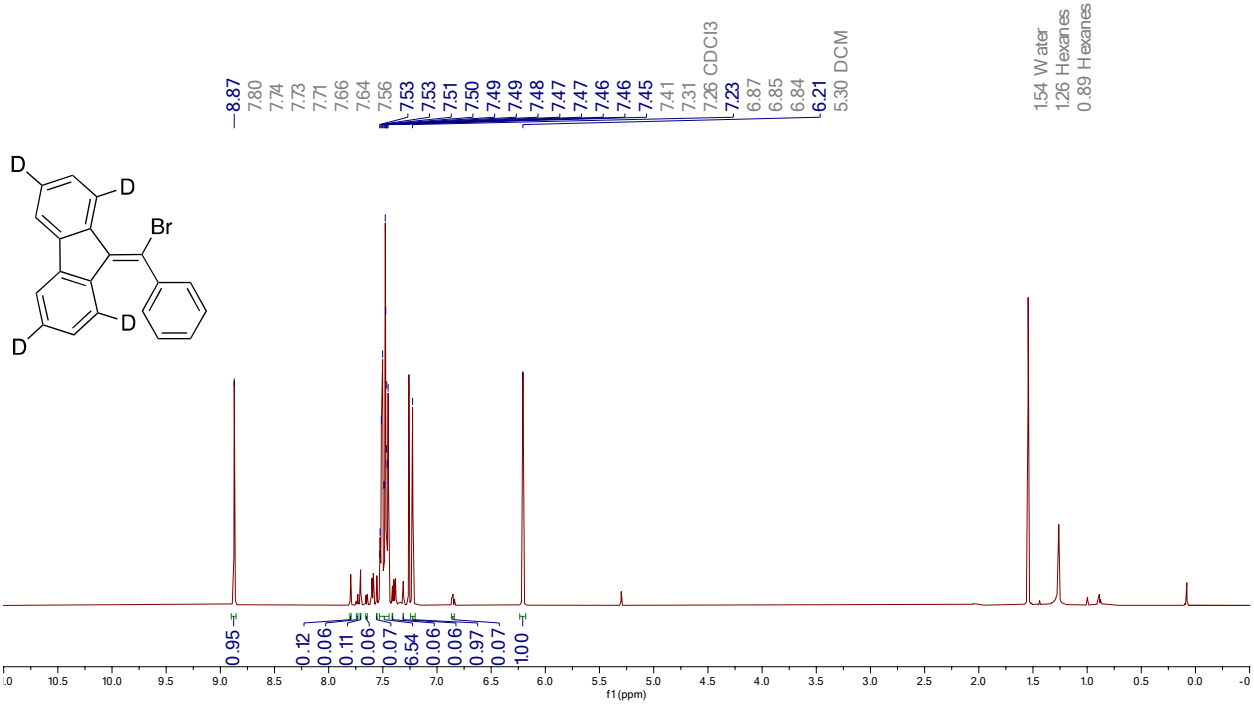


Figure S24 ^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **9-(bromo(phenyl)methylene)-9*H*-fluorene-2,4,5,7-*d*$_4$ (16)**

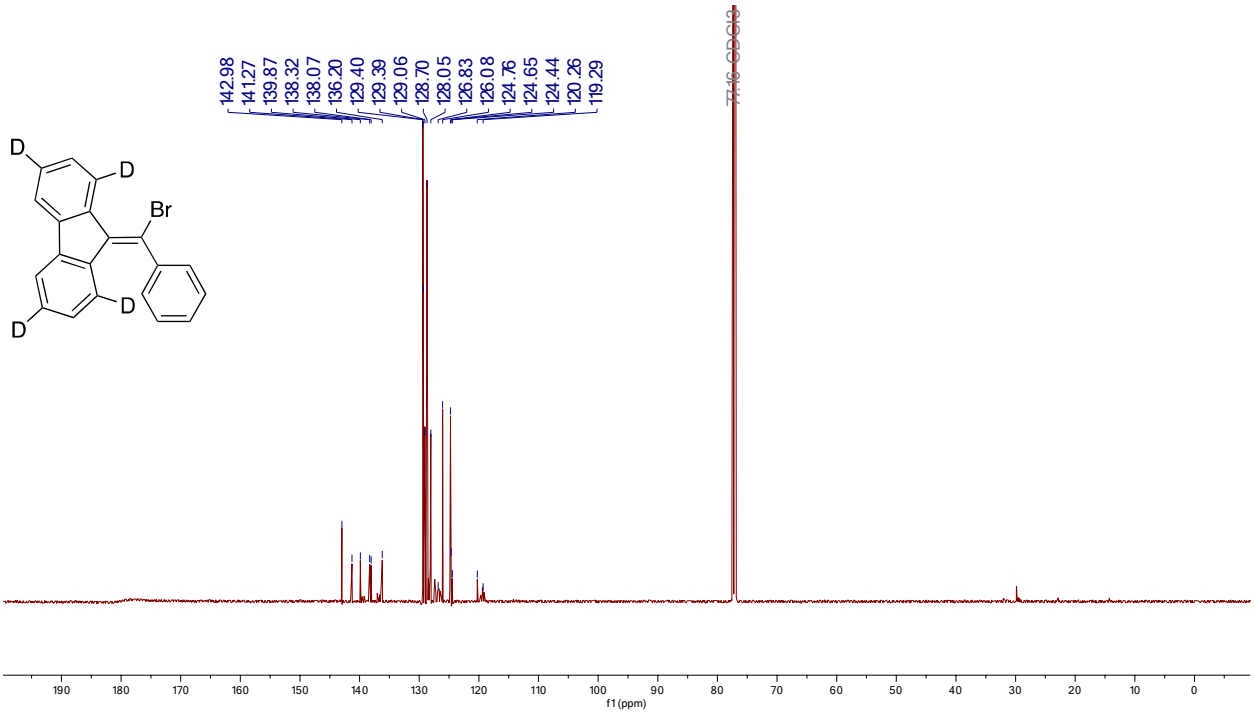


Figure S25 ^{13}C NMR spectrum (151 MHz, $CDCl_3$) of **9-(bromo(phenyl)methylene)-9*H*-fluorene-2,4,5,7-*d*$_4$ (16)**

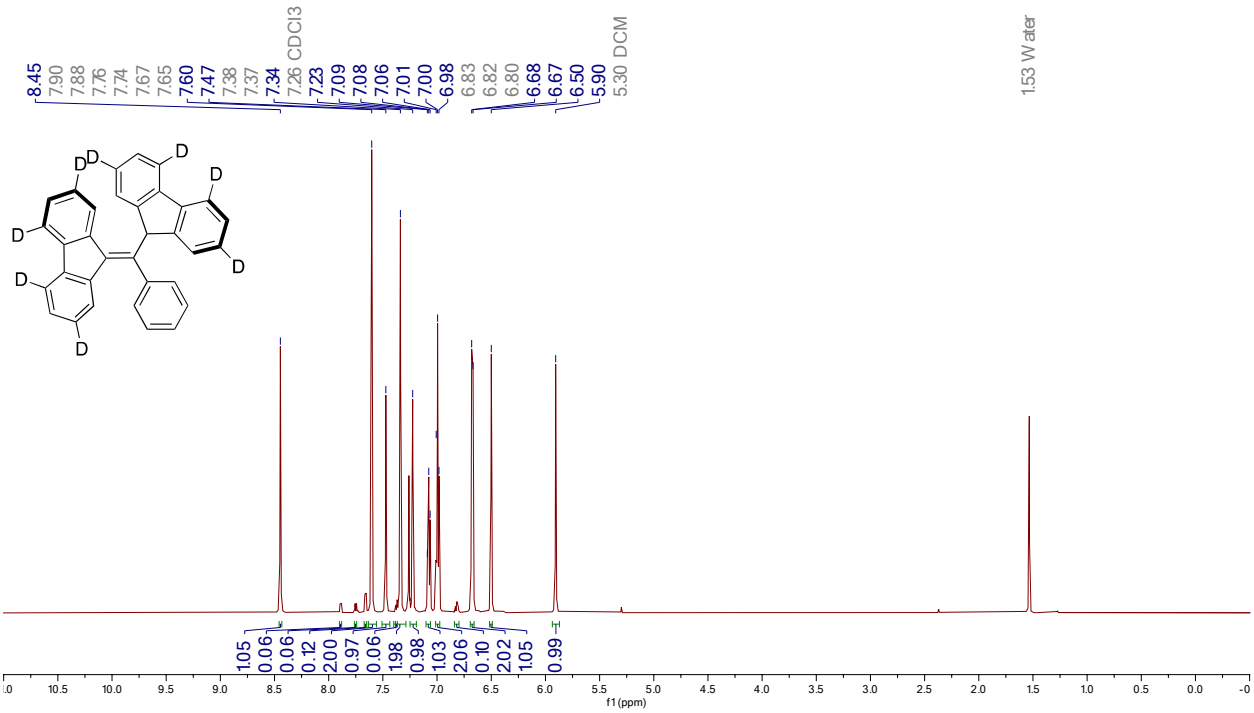


Figure S26 ^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **9-((9*H*-fluoren-9-yl-2,4,5,7-*d*$_4$)(phenyl)methylene)-9*H*-fluorene-2,4,5,7-*d*$_4$ (17)**

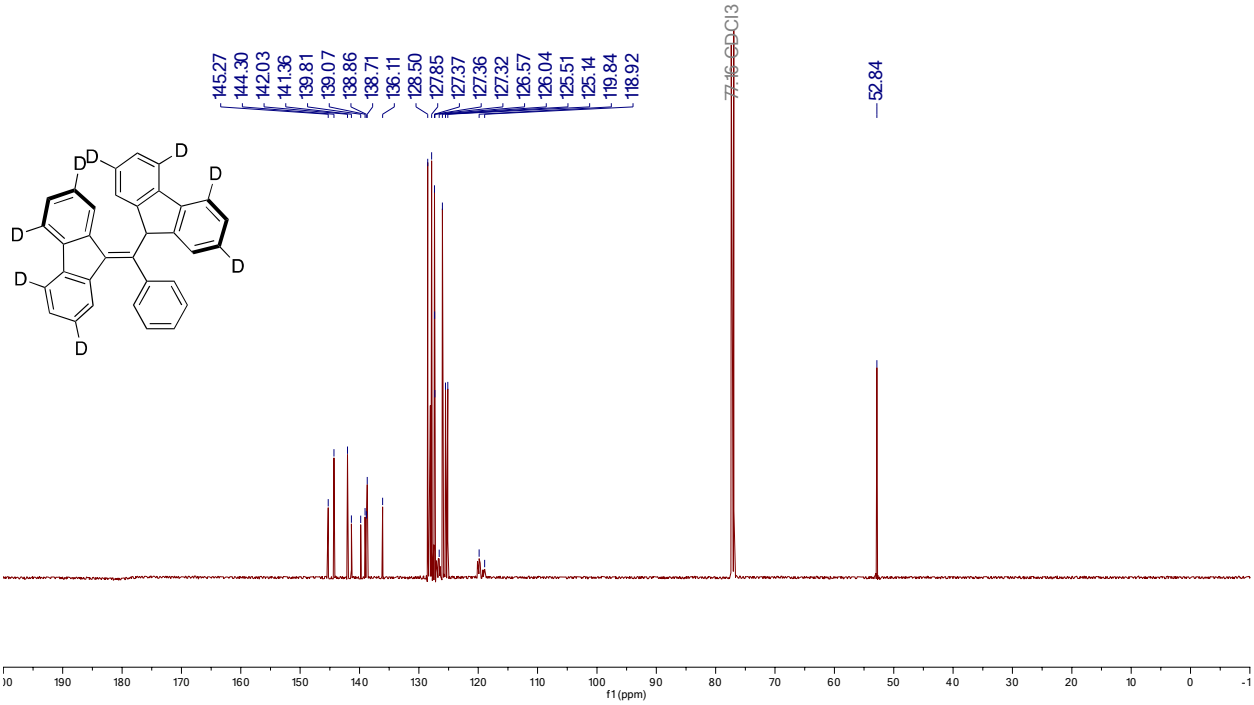


Figure S27 ^{13}C NMR spectrum (151 MHz, $CDCl_3$) of **9-((9*H*-fluoren-9-yl-2,4,5,7-d_4)(phenyl)methylene)-9*H*-fluorene-2,4,5,7-d_4 (17)**

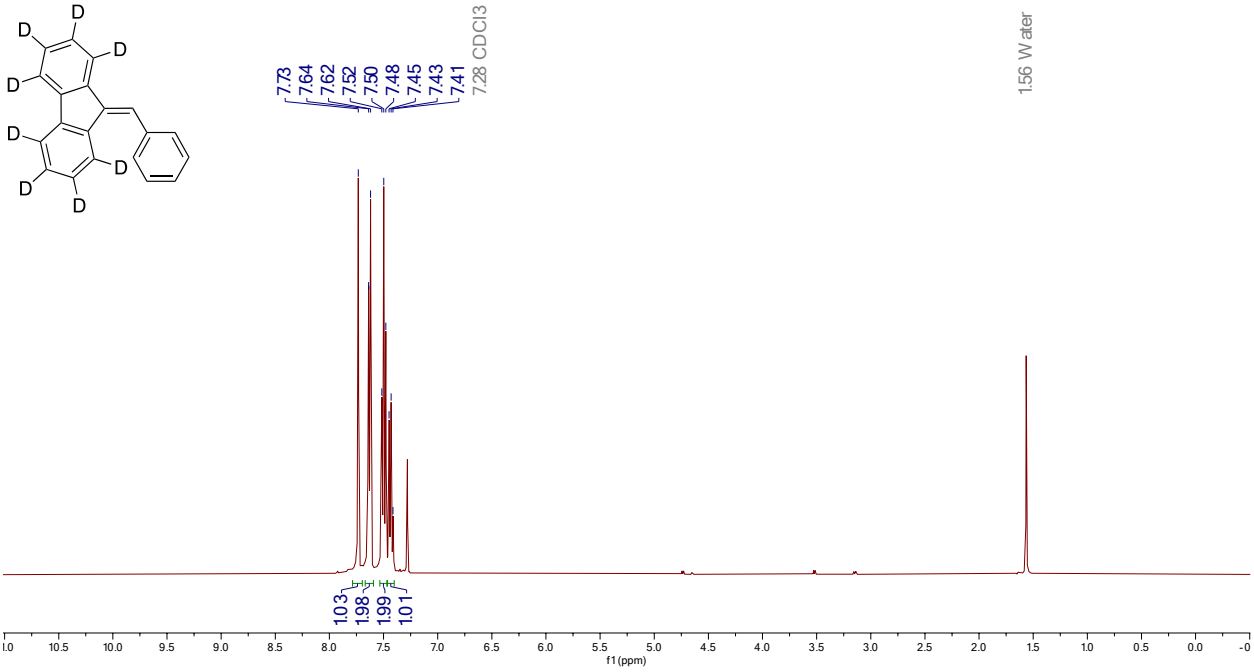


Figure S28 ^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **9-benzylidene-9*H*-fluorene-1,2,3,4,5,6,7,8-d_8 (19)**

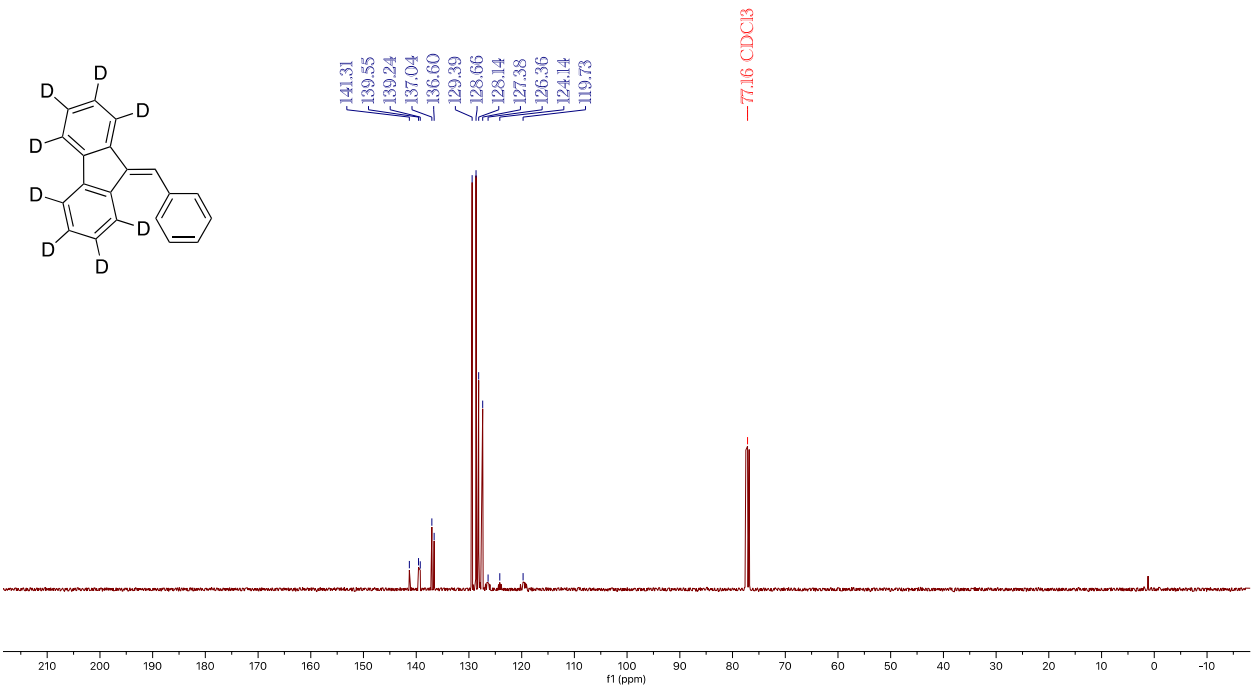


Figure S29 ^{13}C NMR spectrum (151 MHz, $CDCl_3$) of **9-benzylidene-9*H*-fluorene-1,2,3,4,5,6,7,8-d_8 (19)**

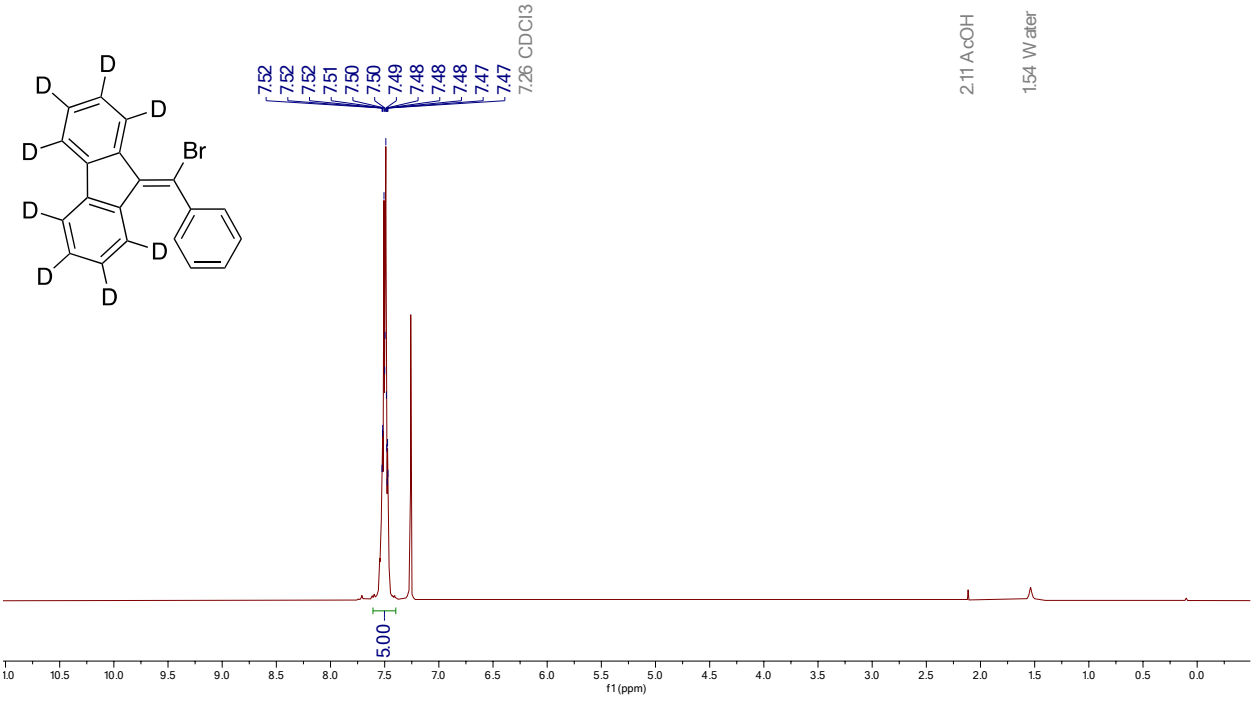


Figure S30 ^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **9-(bromo(phenyl)methylene)-9*H*-fluorene-1,2,3,4,5,6,7,8-d_8 (20)**

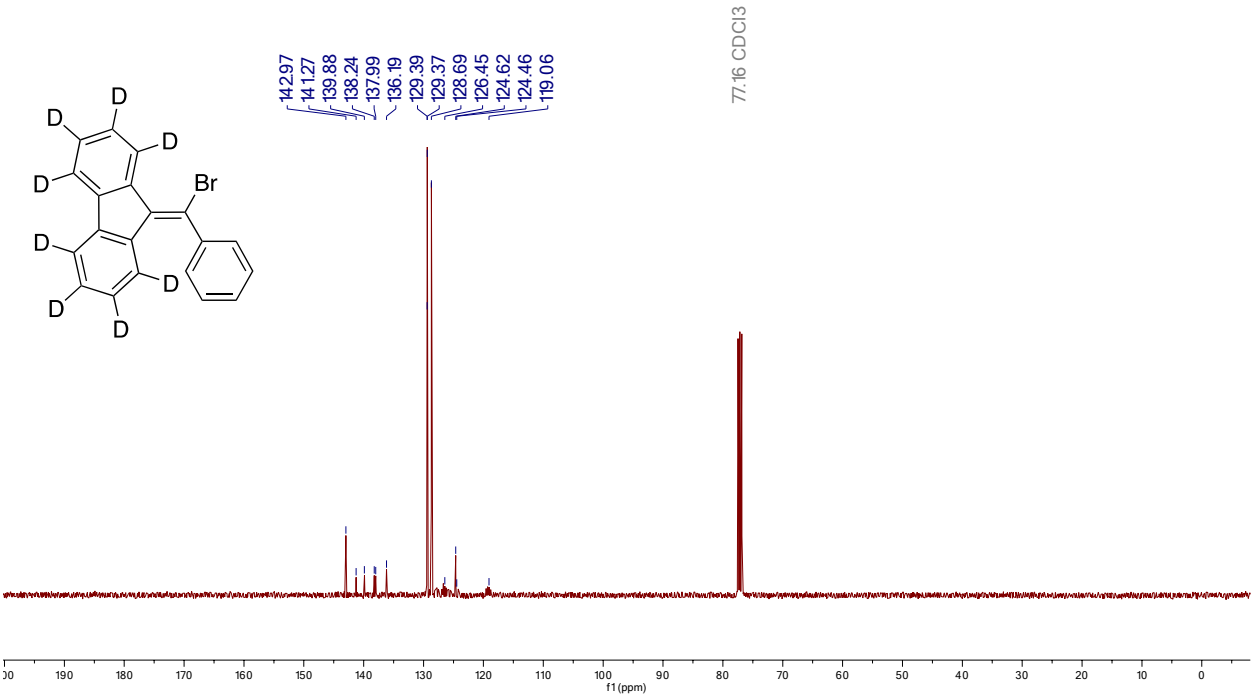


Figure S31 ^{13}C NMR spectrum (151 MHz, $CDCl_3$) of **9-(bromo(phenyl)methylene)-9*H*-fluorene-1,2,3,4,5,6,7,8-d_8 (20)**

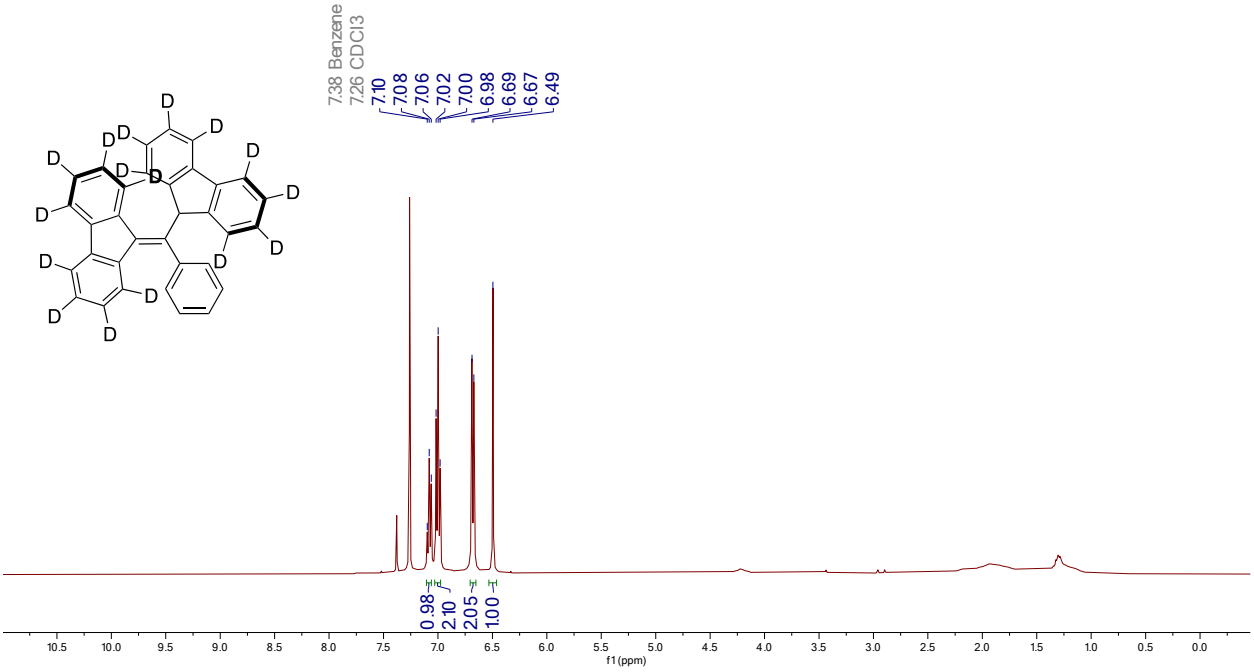


Figure S32 ^{1}H NMR spectrum (600 MHz, $CDCl_3$) of **9-((9*H*-fluoren-9-yl-1,2,3,4,5,6,7,8-d_8)(phenyl)methylene)-9*H*-fluorene-1,2,3,4,5,6,7,8-d_8 (21)**

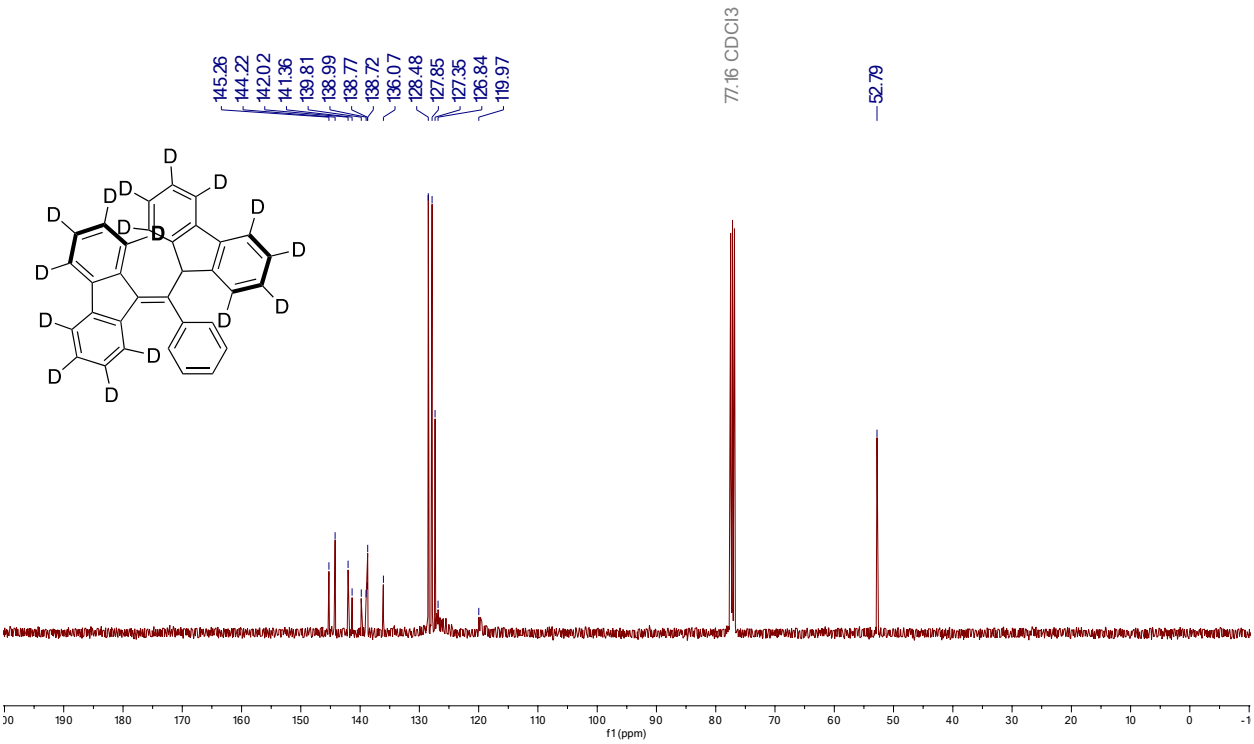


Figure S33 ^{13}C NMR spectrum (151 MHz, $CDCl_3$) of **9-((9*H*-fluoren-9-yl-1,2,3,4,5,6,7,8-d_8)(phenyl)methylene)-9*H*-fluorene-1,2,3,4,5,6,7,8-d_8 (21)**

5. Sample Purity

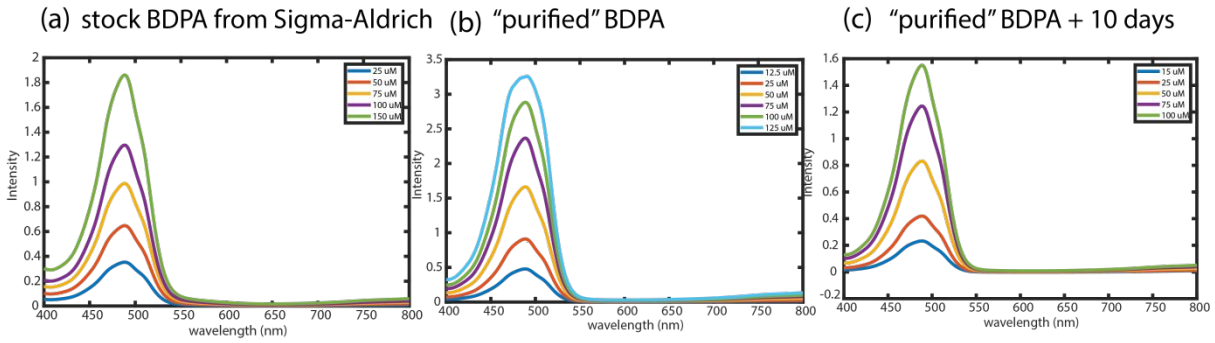


Figure S34. UV-vis measurements at different concentrations for (a) stock h_{21}-BDPA (complex with benzene) as purchased from Sigma-Aldrich and used in the presented work, (b) h_{21}-BDPA purified via silica gel chromatography, and (c) the same as (b) only measured 10 days later.

Sample	Extinction coefficient
Stock h_{21}-BDPA from Sigma-Aldrich	12930 ± 650
"Purified" h_{21}-BDPA	28770 ± 3300
"Purified" h_{21}-BDPA after 10 days	16360 ± 920

Table S1. Calculated extinction coefficients for h_{21}-BDPA based on measurements given in Fig. S28. The stock h_{21}-BDPA used in this work is found to possess only about 50% of active radicals.

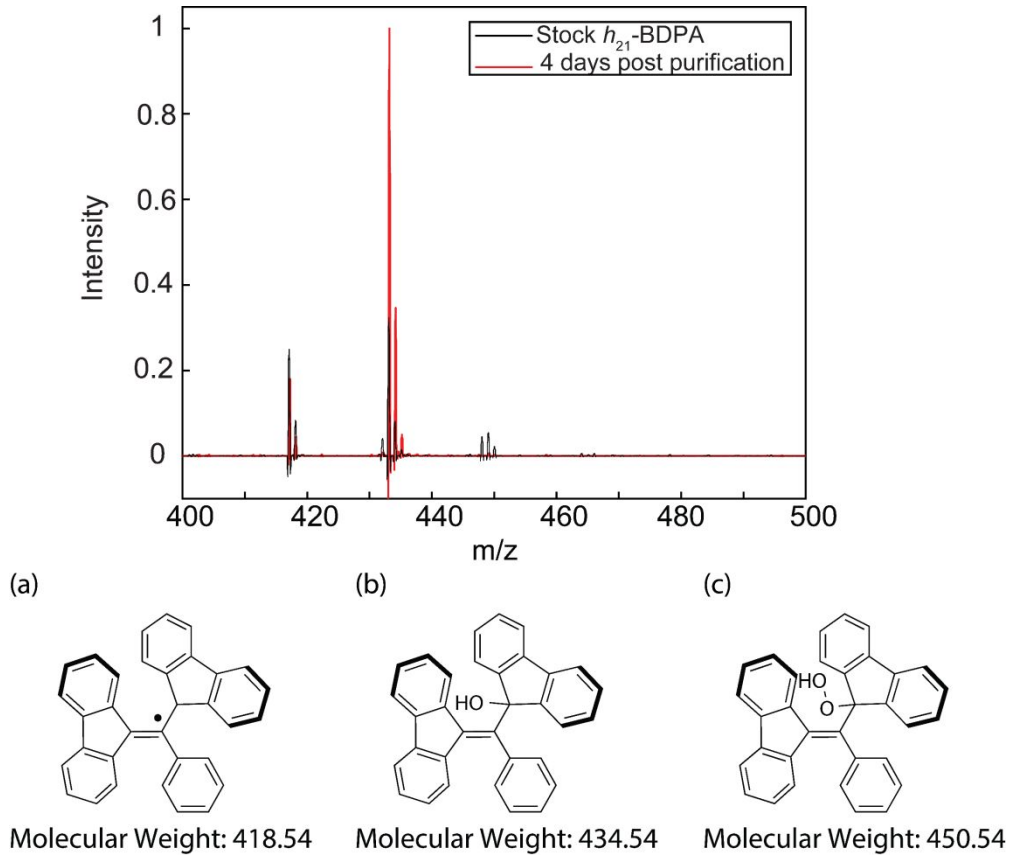


Figure S35. Matrix assisted laser desorption ionization-time of light mass spectrometry (MALDI-TOF-MS) of Sigma Aldrich of h_{21}-BDPA 1:1 in complex with benzene (black) and 4 days after silica gel chromatography purification (red). MALDI-TOF-MS samples were prepared by dissolving h_{21}-BDPA into chloroform and spotting onto sample plate. Major species in the MALDI-TOF-MS spectrum are (a) h_{21}-BDPA radical, (b) hydroxylated BDPA, and (c) hydroperoxylated BDPA. Following purification of the sample and removal of benzene, h_{21}-BDPA is prone to hydroxylation.

6. Radical Concentrations

EPR signal intensity of DNP samples in a 4 mm sapphire rotor was determined using a Magnettch ESR5000 spectrometer and double integral (DI) signal intensities were calculated using MATLAB[5]. The quantity of electron spins in h_{21}-BDPA, 1,3-[α,β,γ,δ- d_{16}]-BDPA ,1,3-[α,γ-d_8]-BDPA, and 1,3-[β,δ-d_8]-BDPA were determined relative to a standard sample whose concentration and number of electron spins was determined using absolute spin quantification methods on a EMXnano spectrometer. The number of electron spins, n, in each DNP sample were determined as $n_{DNP} = \frac{DI_{DNP} \times n_{Standard}}{DI_{Standard}}$. In Figure S30, the double integrals for each of the four polarizing agents are shown. The concentration was then calculated by measuring the length of sample in the rotor, the uncertainty in length measurement is reflected in the minimum and maximum concentration values in Figure S31.

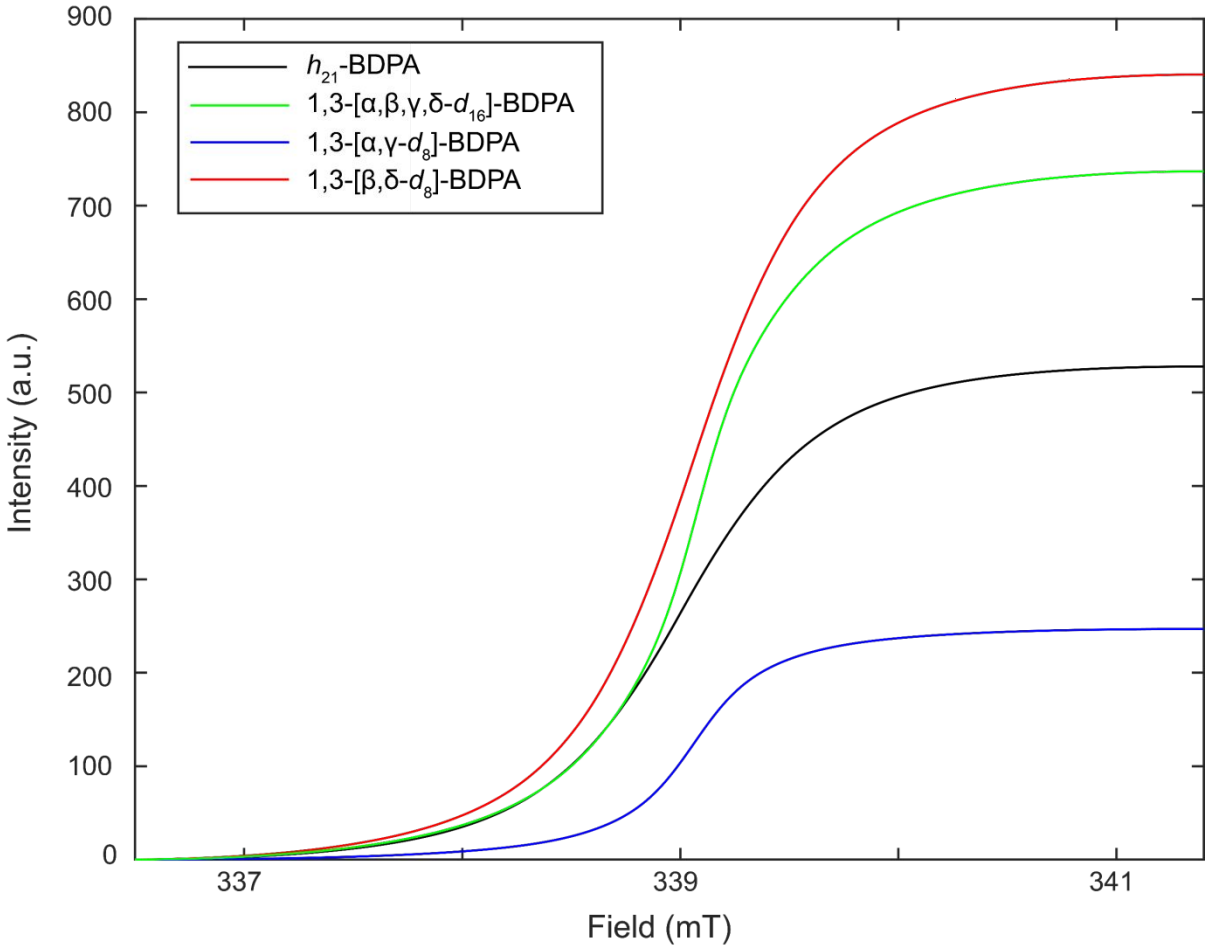


Figure S36. Double Integral of X-Band EPR derivative spectra for h_{21}-BDPA, 1,3-[α,β,γ,δ- d_{16}]-BDPA, 1,3-[α,γ-d_8]-BDPA, and 1,3-[β,δ-d_8]-BDPA.

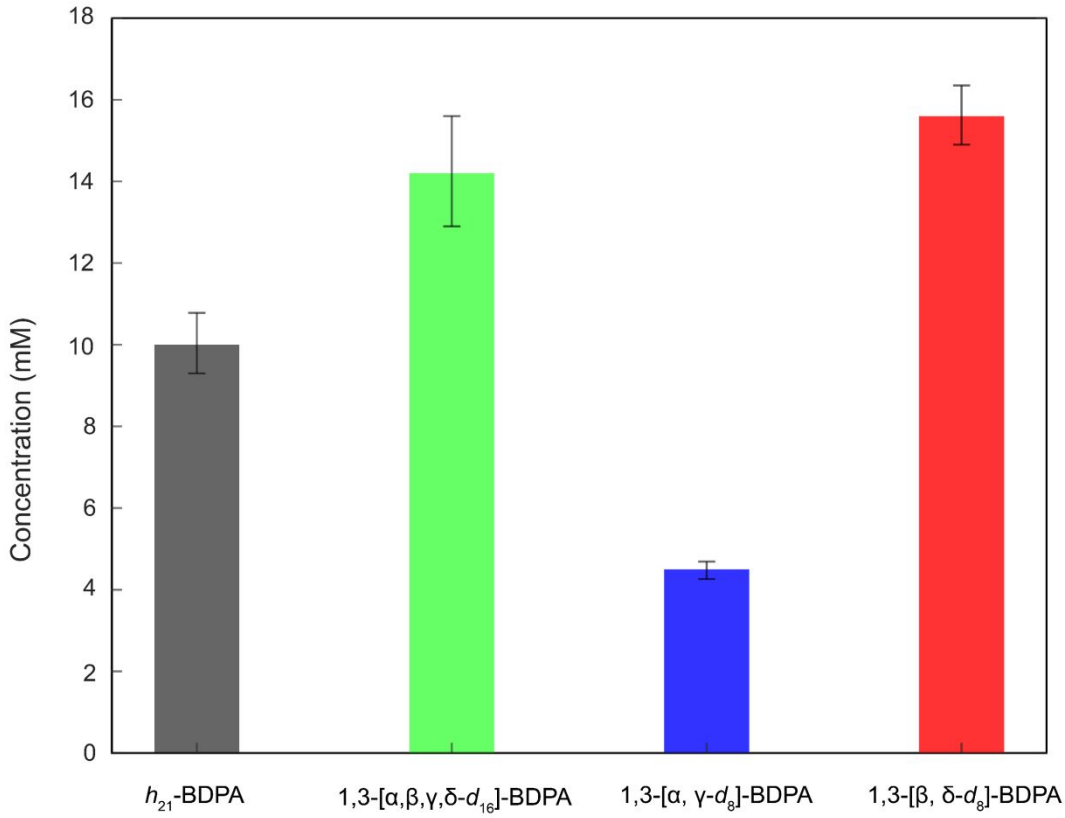


Figure S37. Radical concentrations from X-Band EPR for h_{21}-BDPA, 1,3-[d_{16}]-BDPA, 1,3-[α,γ-d_8]-BDPA, and 1,3-[β,δ-d_8]-BDPA. Determined concentrations by EPR were 10 mM, 14.2 mM, 4.5 mM, and 15.6 mM for h_{21}-BDPA, 1,3-[α,β,γ,δ-d_{16}]-BDPA, 1,3-[α,γ-d_8]-BDPA, and 1,3-[β,δ-d_8]-BDPA, respectively.

7. DNP Supporting Information

All DNP samples were degassed using a freeze-pump-thaw procedure using the homebuilt adapter shown in Figure S38(a). The adapter was 3D printed using a Form3 3D printer using Rigid 4K resin (Formlabs Somerville, MA). The adapter was then epoxied to a 3-way valve, as shown in Figure S38(b), through which vacuum was applied to the rotor directly. The sample was kept immersed in liquid nitrogen while being transferred to a glove bag following 5 freeze-pump-thaw cycles. The rotor was removed from degassing apparatus and endcap inserted in the glove bag and immediately inserted into spectrometer.

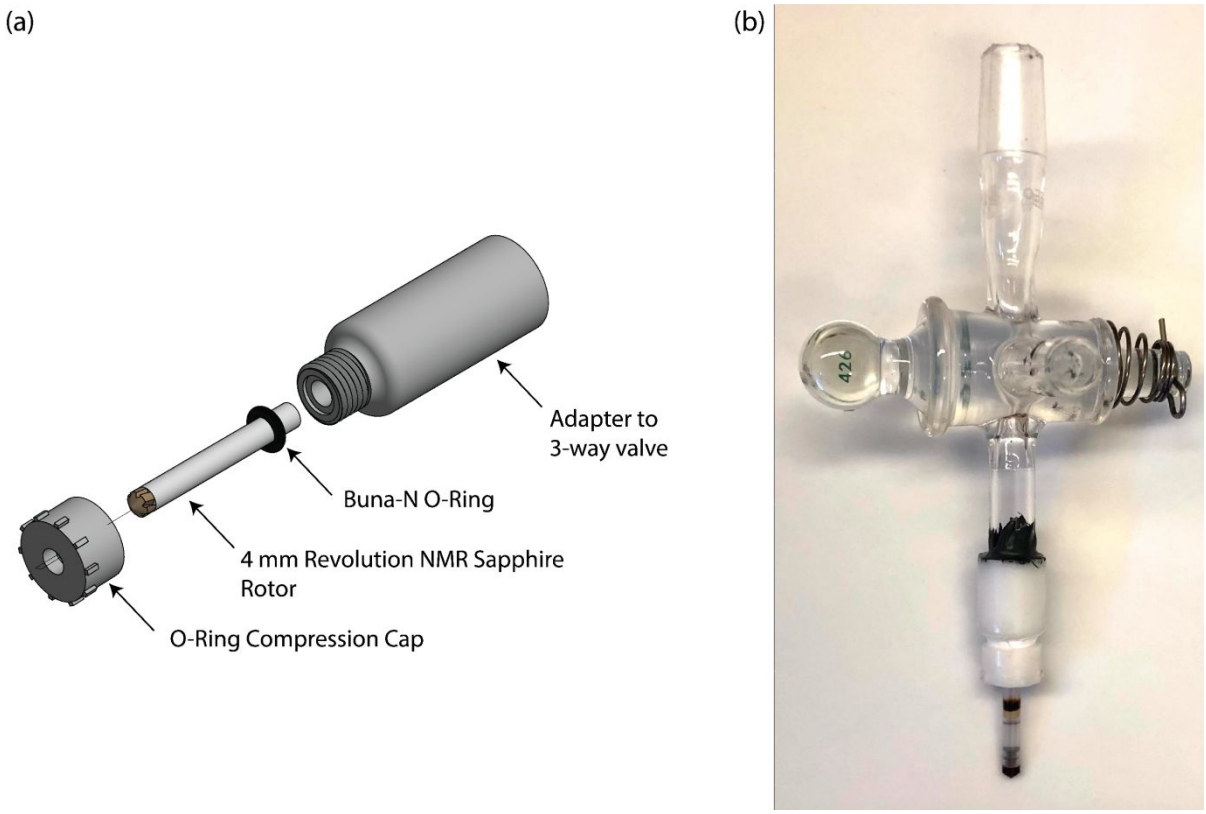


Figure S38. Homebuilt degassing apparatus used for DNP sample preparation. (a) CAD rendering of 3D printed adapter. Sample rotor was sealed against atmosphere using Buna-N O-ring which was observed to maintain sufficient sealing while sample was immersed into liquid nitrogen during freezing and pumping steps. (b) Photograph of assembled adapter epoxied on 3-way valve which enabled connection to vacuum pump in addition to nitrogen gas purge line.

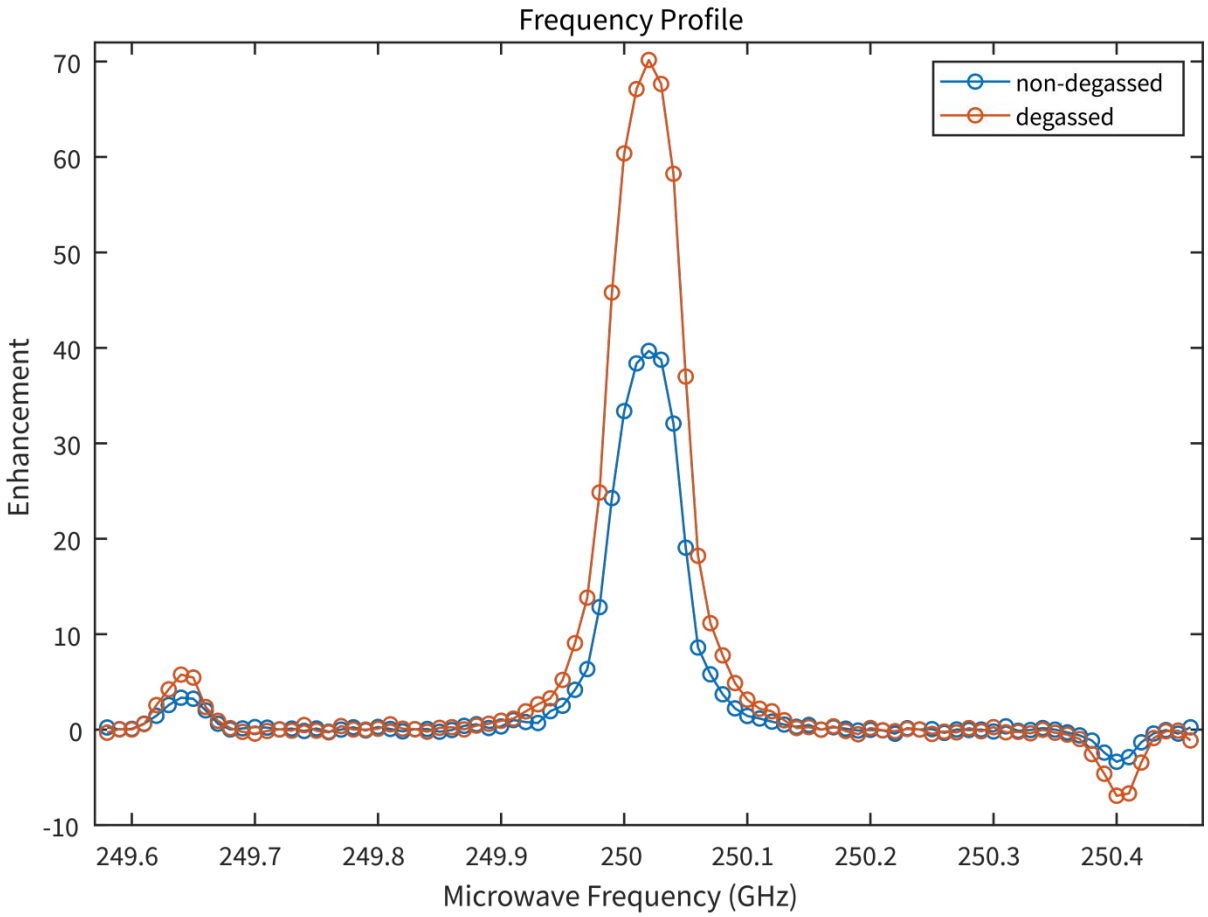


Figure S39. ^{1}H DNP enhancement frequency profiles for 2.5 wt% h_{21}-BDPA in 5 mol% h_{14}- and 95 mol% d_{14}-oTP matrix with the sample subjected to freeze-pump-thaw degassing (red) or not (blue). The NMR magnetic field strength was 8.92 T (380 MHz ^{1}H Larmor frequency), the sample temperature was 90 K, and the MAS frequency was 5 kHz.

Sample	T_1 /s	$T_{B,OE}$ /s	$T_{B,SE}$ /s	Enh. (OE)	Enh. (SE)
Non-degassed	32.1 ± 2.2	37.5 ± 1.5	38.4 ± 1.9	70.2	5.8
Degassed	42.6 ± 3.3	43.6 ± 2.3	42.4 ± 2.1	39.7	3.3

Table S2. Experimentally observed spin-lattice relaxation times (T_1), DNP buildup times with the microwave irradiation at the OE frequency ($T_{B,OE}$) or at the positive SE frequency ($T_{B,SE}$) are expressed in seconds for 2.5 wt% h_{21}-BDPA in 5 mol% h_{14}- and 95 mol% d_{14}-oTP matrix with the sample subjected to freeze-pump-thaw degassing or not. The enhancement values calculated for the OE and the positive SE are also mentioned. The NMR magnetic field strength was 8.92 T (380 MHz ^{1}H Larmor frequency), the sample temperature was 90 K, and the MAS frequency was 5 kHz.

Table S3. Experimentally measured enhancement values with error estimates at the OE microwave frequency (ε_{OE}) and at the +SE frequency (ε_{SE}) and a constant recovery period of 180 s for all the samples.

Radical	$\boldsymbol{\varepsilon_{OE}}$	$\boldsymbol{\varepsilon_{SE}}$
h_{21}-BDPA	71.0 ± 2.7	6.7 ± 0.3
1,3-[α,β,γ,δ-d_{16}]-BDPA	-11.6 ± 0.7	1.5 ± 0.2
1,3-[α,γ-d_8]-BDPA	-1.2 ± 0.2	0.8 ± 0.1
1,3-[β,δ-d_8]-BDPA	31.7 ± 3.6	1.9 ± 0.4

8. References

1. Gandeepan, P., C.-H. Hung, and C.-H. Cheng, *Pd-catalyzed double C–H bond activation of diaryl ketones for the synthesis of fluorenones.* Chemical Communications, 2012. **48**(75): p. 9379-9381.
2. Brunetti, F.G., et al., *Strain and Hückel Aromaticity: Driving Forces for a Promising New Generation of Electron Acceptors in Organic Electronics.* Angewandte Chemie International Edition, 2010. **49**(3): p. 532-536.
3. Plater, M.J., S. Kemp, and E. Lattmann, *Heterocyclic free radicals. Part 1. 4,5-Diazafluorene derivatives of Koelsch's free radical: an EPR and metal-ion complexation study.* Journal of the Chemical Society, Perkin Transactions 1, 2000(6): p. 971-979.
4. Haze, O., et al., *Water-Soluble Narrow-Line Radicals for Dynamic Nuclear Polarization.* Journal of the American Chemical Society, 2012. **134**(35): p. 14287-14290.
5. *MATLAB R2020a*. 2020, The MathWorks, Inc. Natick, Massachusetts.